

# DR890 光度计

## 分析操作说明书



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te/dk 2/02 Sed

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铝 (0 to 0.80 mg/L)

方法号: 8012

### 试铝灵法



1. 输入检测铝 (A1) 的预定程序编号。

按下: **PRGM**

屏幕上将显示:

**PRGM?**

注: 为了能够得到最精确的结果, 应使用去离子水进行试剂空白校正。(见第 1 章)

2. 按: **1 ENTER**  
屏幕将显示 **mg/L**, **A1** 和 **ZERO** 图标。

注: 总铝的测定在检测前要进行消解处理。

注: 如检测其他形态 (A1203), 按 **CONC**。

3. 往标有刻度的混合量筒中注入 50 毫升的样本。

注: 在使用前, 用 1:1 盐酸和去离子水清洗量筒, 从而避免由于玻璃上所吸附的污染所导致的结果偏差。

2. 为得到精确的结果, 样品的稳定应保持在  $20-25^{\circ}\text{C}$  ( $68-77^{\circ}\text{F}$ )。

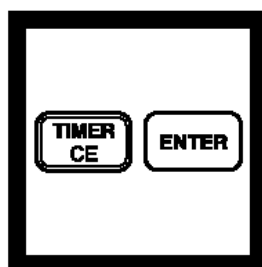
4. 加入一包抗坏血酸粉末, 塞好塞子, 倒转多次使粉末溶解。



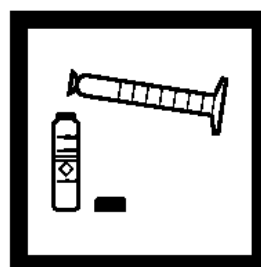
5. 加入一包 AluVer 3 铝试剂粉包, 反复倒转使之溶解。

注: 如果铝的成分存在, 溶液则会呈橙红色。

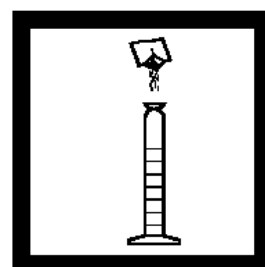
注: 如粉末未完全溶解, 将会产生不正确的结果。



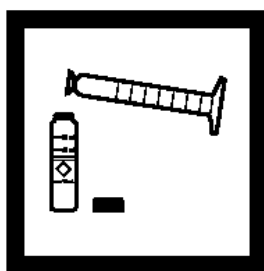
6. 按下: **TIMER ENTER**  
将开始三分钟的反应。反复倒转量筒三分钟。



7. 倒出 25 mL 混合液到容积为 25mL 的比色瓶中 (待测样品)。



8. 加入一包 Bleaching 3 试剂粉包到混合量筒剩余的 25mL 溶液中, 盖好盖子。



9. 屏幕将显示

**00:30 Timer 2**

按下: ENTER

开始进行 30 秒的反应。期间要不断地摇动量筒。

10. 倒出量筒中剩余的25mL混合液到另一个25mL的比色瓶中（空白试样）。

11. 屏幕将显示:

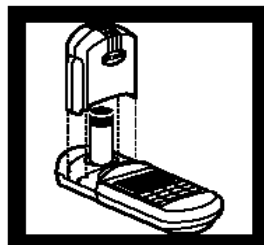
**15:00 TIMER 3**

按下: ENTER

将会开始 15 分钟的反应。

12. 在计时器鸣叫后三分钟内，将空白试样放入样品适配器总，盖紧遮光盖。

*注：通过漂泊溶液颜色将变浅成中度橙色，而不会变成无色。*



13. 按 **ZERO**，指针将向右移，屏幕显示：  
0.000 mg/L Al<sup>3+</sup>。

*注：如果进行试剂空白校正，屏幕将闪烁显示“limit”。*

14. 迅速将装有预先准备好的比色瓶放进样品适配器中，并盖紧遮光盖。

15. 按 **READ** 指针将向右移。屏幕将显示铝的含量，单位是 mg/L。

*注：进行完测试应马上用清洗液和刷子清洗量筒和样品管。*  
*注：用预定的标准溶液进行标准校正。*

## 采样和存放

将收集的样本存放在一个干净的玻璃或者塑料的容器内。为了存放样本，可往样本溶液中加入硝酸（大约 1.5 毫升/升样本溶液），调整溶液的 PH 值到 2 或者以下。在室温的条件下，所存放的样本可以保存 6 个月。在进行样本分析之前，应加入 5.0 N 的氢氧化钠溶液将样本溶液的 PH 值调到 3.5-4.5 的范围。要为所加部分进行检测结果的修正，欲知其具体情况可参考第一部分中的加标测试的内容。

## 精确检测

### 加标测试法

- 量取一定数量浓度为 50 毫克/升的 Aluminum Voluette Ampule 标准铝溶液。
- 使用移液管分别往 3 个样本中滴入 0.1 毫升、0.2 毫升、0.3 毫升的标准铝溶液。轻轻摇晃使之充分混合。同时还应准备一个没有加入任何指示剂的样本。
- 根据上述所示步骤进行分析样本。样本中铝的含量应该随着每增加 0.1 毫升的铝标准溶液就会增加 0.1 毫克/升。
- 如果这些增加没有出现，则参考第一部分的加标测试法的详细内容说明。

### 标准溶液法

将 1.00 毫升的含量为 100 毫克/升的含  $AL^{3+}$  离子标准溶液注入容积为 250 毫升的烧瓶中，用去离子水稀释成 0.4 毫克/升的铝盐标准溶液。该配好的溶液应该马上用作检测。按照上述步骤进行铝测定过程，铝含量的读数应该是 0.4 毫克/升。

或者使用 TenSette 移液管，将 0.8 毫升的含铝量为 50 毫克/升 Voluette Ampule 标准溶液注入一个容积为 100 毫升的烧瓶内，然后用去离子水进行稀释。该配好的溶液应该马上用作检测或作为样本。

## 检测方法的效果

### 精度

在一个单独的实验室里，如果使用 0.4 克/升的铝盐溶液和仪器所附带的两种典型试剂，单个检测员所得的铝含量检测结果的标准误差是  $\pm 0.013$  毫克/升。

### 预计检测极限

程序 #1 的预计检测极限是铝含量为 0.013 毫克/升。欲了解更详细的检测极限方面的情况请参考第一部分的相关内容。

## 干扰

| 干扰物质 | 干扰水平和处理                                                                                                                                                                                                       |
|------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 酸度   | 酸度大于300 mg/L的CaCO <sub>3</sub> 。样品的酸度大于300 mg/L的CaCO <sub>3</sub> ，须按如下步骤处理：<br>a) 在步骤3的样品溶液中加入一滴间硝基酚指示剂溶液。<br>b) 加入一滴5.0 N的氢氧化钠标准溶液，塞好量筒。倒转多次使混合均匀，直到颜色从无色变为黄色。<br>c) 加入一滴5.25 N的硫酸标准溶液，使溶液的颜色从黄色转变成无色，继续测试。 |
| 碱度   | 碱度为1000 mg/L 的CaCO <sub>3</sub> 。更高碱度时的干扰可由如下预处理消除。<br>a) 在步骤3的样品溶液中加入一滴间硝基酚指示剂溶液。呈现黄色表明碱度过高。<br>b) 加入一滴5.25N的硫酸标准溶液，塞好量筒，倒转使混合均匀。如果仍然呈现黄色，继续倒转直到样品变成无色，继续测试。                                                 |
| 氟化物  | 所有水平上均干扰。参看下面的曲线。                                                                                                                                                                                             |
| 铁    | 大于20 mg/L。                                                                                                                                                                                                    |
| 磷酸盐  | 大于50 mg/L。                                                                                                                                                                                                    |
| 多磷酸盐 | 多磷酸盐由于会引起负误差，在所有水平上均干扰，所以不能允许存在。在测试之前，多磷酸盐必须用酸水解成正磷酸盐，方法记载在测磷的程序中。                                                                                                                                            |

氟化物由于与铝结合，在所有水平上均干扰。当已知氟化物浓度时，可使用氟化物干扰曲线图测定铝的实际浓度。氟化物干扰曲线图的使用：

1. 在曲线图的顶端，找到代表步骤12显示的铝含量的垂直线。
2. 找出代表样品内氟化物含量的水平线与垂直线相交的点。
3. 沿着交点两侧中最近的曲线往下推，与横坐标的交点为推测的铝实际浓度。

度。

例如，如果测试结果显示铝含量为0.7 mg/L，并且样品中存在1 mg/L氟化物（F<sup>-</sup>），沿着代表铝含量0.7mg/L的垂直线与代表1 mg/L F<sup>-</sup>的水平线的交点附近的曲线往下推，落在铝曲线的1.2 and 1.3 mg/L之间，这样，铝的实际含量就是1.27 mg/L。

## 氟化物干扰图

Fluoride Interference Graph



## 所需试剂

|                                              | 货号       |
|----------------------------------------------|----------|
| 铝试剂一套 (100 Tests) .....                      | 22420-00 |
| 包括: (1) 14290-99, (1) 14577-99, (1) 14294-49 |          |

| 试剂种类                   | 所需数量<br>每次测试 | 单位           | 货号.      |
|------------------------|--------------|--------------|----------|
| AluVer 3 铝粉末试剂 .....   | 1 包 .....    | 100/pkg..... | 14290-99 |
| 抗坏血酸粉末 .....           | 1 包 .....    | 100/pkg..... | 14577-99 |
| Bleaching 3 粉末试剂 ..... | 1 包 .....    | 100/pkg..... | 14294-49 |

## 所需仪器

|                                |         |            |          |
|--------------------------------|---------|------------|----------|
| 具有50毫升刻度的混合量筒 .....            | 1 ..... | 个 .....    | 1896-41  |
| 样品比色瓶 10-20-25 mL, w/ cap..... | 2 ..... | 6/pkg..... | 24019-06 |

## OPTIONAL REAGENTS

|                                                                           |                 |          |
|---------------------------------------------------------------------------|-----------------|----------|
| Aluminum Standard Solution, 100 mg/L.....                                 | 100 mL.....     | 14174-42 |
| Aluminum Standard Solution, Voluette ampule,<br>50 mg/L as Al, 10 mL..... | 16/pkg.....     | 14792-10 |
| Hydrochloric Acid Solution, 6N (1:1) .....                                | 500 mL.....     | 884-49   |
| m-Nitrophenol Indicator Solution, 10 g/L.....                             | 100 mL.....     | 2476-32  |
| Nitric Acid, ACS.....                                                     | 500 mL.....     | 152-49   |
| Nitric Acid Solution, 1:1.....                                            | 500 mL.....     | 2540-49  |
| Sodium Hydroxide Standard Solution, 5.0 N .....                           | 100 mL MDB..... | 2450-32  |
| Sodium Hydroxide Standard Solution, 5.0 N .....                           | 50 mL SCDB..... | 2450-26  |
| Sulfuric Acid Standard Solution, 5.25 N .....                             | 100 mL MDB..... | 2449-32  |
| Water, deionized.....                                                     | 4 L.....        | 272-56   |

## OPTIONAL APPARATUS

|                                                                        |                  |          |
|------------------------------------------------------------------------|------------------|----------|
| Ampule Breaker Kit.....                                                | each.....        | 21968-00 |
| Brush.....                                                             | each.....        | 690-00   |
| Flask, volumetric, Class A, 100 mL .....                               | each.....        | 14574-42 |
| Flask, volumetric, Class A, 250 mL .....                               | each.....        | 14574-46 |
| Fluoride Combination Electrode.....                                    | each.....        | 50265-00 |
| Fluoride Analysis Package.....                                         | each.....        | 50269-00 |
| pH Indicator Paper, 1 to 11 pH .....                                   | 5 rolls/pkg..... | 391-33   |
| pH/ISE Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> 2, portable..... | each.....        | 51725-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....                                    | each .....       | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....                          | 50/pkg.....      | 21856-96 |
| Pipet, Volumetric, Class A, 1.00 mL .....                              | each.....        | 14515-35 |
| Thermometer, -10 to 110 °C.....                                        | each.....        | 1877-01  |

## For Technical Assistance, Price and Ordering

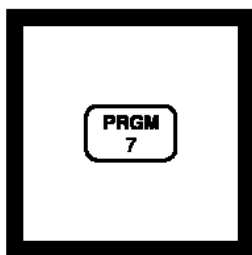
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# 苯并三唑 或 甲苯并三唑 (0—16.0 mg/L)

方法号: 8079

## 紫外光分解法



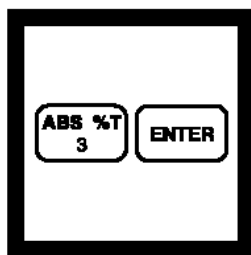
1. 输入检测苯并三唑 (Benzo)或甲苯并三唑 (Toly) 的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为得到最精确的结果, 应用去离子水进行试剂空白校正。



2. 按下: 3 ENTER 进行苯并三唑或甲苯并三唑的检测。

屏幕将显示:

mg/L、BENZO 和 ZERO 图标或者 mg/L、TOLY 和 ZERO 图标。

按下: CONC 键

选择要检测的样品种类。



3. 往比色管中装入 25 mL 样品。

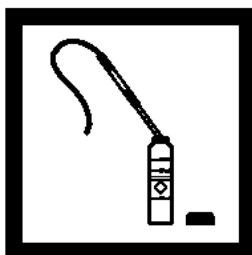
注: 样品的温度应在 20—25 °C (68—77 °F)。

注: 如果样品中含有亚硝酸盐或硼砂 (硼酸钠), 应用 1 N 的硫酸将样品 PH 值调整到 4 至 6。



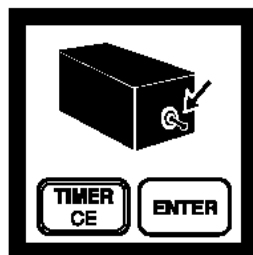
4. 加入一包三唑试剂粉包。旋转混合使之完全溶解。

注: 如果样品中包含 500 毫克/升的硬度物质 (碳酸钙), 应加入 10 滴酒石酸钾钠。



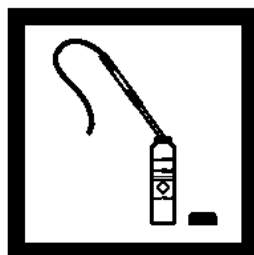
5. 在比色管中嵌入紫外灯。

注: 在紫外灯打开时, 应戴上紫外护目镜。



6. 打开紫外灯, 按下 TIMER 回车 开始5分钟的反应计时。

注: 如有三唑物存在, 溶液将呈黄色。



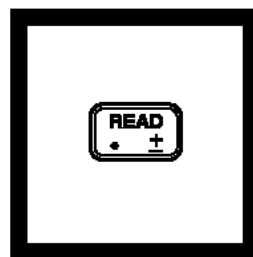
7. 当计时器鸣叫时, 关紫外灯。从比色管中取出紫外灯。旋转样品管充分混合。

注: 如果光分解作用进行时间低于或超过 5 分钟, 将会得到一个较小的结果。

注: 不要触摸紫外灯的石英表面。在使用时要将灯进行清洗和用干净和柔软的薄绵纸擦干。



8. 往另一个比色管中装入 25mL 样品 (空白试样)。



9. 将空白试样放入样品适配器，并用盖紧紧盖上遮光盖。

10. 按 **ZERO**。指针将向右移动。然后屏幕将显示：

0.0 mg/L Benzo 或  
0.0 mg/L Toly。

*注：如果正在进行样本空白校正，则屏幕将闪烁显示“limit”字样。详见第1章。*

11. 将预制试样放入样品适配器，并用盖紧紧盖上遮光盖。

12. 按 **READ**。指针将向右移动。然后屏幕将显示苯并三唑或甲苯并三唑的含量，单位是 mg/L。

*注：要用预制的标准溶液进行标准校正。*

## 取样和存放

样本一经取样应该马上进行分析，这样才能够得到最可靠的结果。

## 精确检测

### 加标测试

- 使用TenSette移液管分别往3个25毫升的样本中注入0.1毫升、0.2毫升、0.3毫升的浓度为500毫克/升的苯并三唑标准溶液。然后根据上述步骤进行检测。

注：该测试是不会区分苯并三唑和甲苯并三唑的。

- 在没有加标准溶液所得的读数的基础上，每增加0.1毫升的标准溶液将会使读数增加2克/升。
- 如果这些增加的情况没有出现，请参考第一部分加标测试的有关详细内容。

## 紫外灯检测

为了验证紫外灯（通常寿命是5000小时）是否正常工作，要进行以下的测试：

- 将10.0毫升的含量为500毫克/升的苯并三唑溶液注入容积为1000毫升的烧瓶中，然后稀释成5.0毫克/升的苯并三唑标准溶液。
- 根据上述步骤进行测试分析，如果所得结果是小于5.0毫克/升的话，就应该更换紫外灯。

## 检测方法的效果

### 精度

在一个单独的实验室，如果在使用9.0毫克/升的标准三唑溶液和仪器所附

带的典型试剂，单个测试人员所得的结果的标准误差是苯并三唑为±0.21毫克/升以及甲苯并三唑为±0.20毫克/升的。

### 预计检测极限

程序3的预计检测极限是苯并三唑和甲苯并三唑含量为0.7毫克/升。欲了解更详细的检测极限方面的情况请参考第一部分的相关内容。

### 干扰

当以下物质的浓度超过以下所列的标准时，将产生干扰。

| 干扰物质                | 干扰水平                             |
|---------------------|----------------------------------|
| 丙烯酸盐（甲基丙烯酸盐）        | 大于50 mg/L。                       |
| 明矾                  | 大于400 mg/L。                      |
| 硼酸盐（四硼酸钠）           | 大于4000 mg/L。                     |
| 氯（Cl <sub>2</sub> ） | 大于20 mg/L。                       |
| 铬（铬酸盐）              | 大于12 mg/L。                       |
| 铜                   | 大于10 mg/L。                       |
| 硬度                  | 大于500 mg/L 的 CaCO <sub>3</sub> 。 |
| 铁                   | 大于20 mg/L。                       |
| 木素磺化盐               | 大于40 mg/L。                       |
| 镁                   | 大于300 mg/L 的 CaCO <sub>3</sub> 。 |
| 钼（钼酸盐）              | 大于200 mg/L。                      |
| 亚硝酸盐                | 大于4000 mg/L。                     |
| 膦酸盐（AMP or HEDP）    | 大于100 mg/L。                      |
| 硫酸盐                 | 大于200 mg/L。                      |
| 锌                   | 大于80 mg/L。                       |
| 强氧化剂或还原剂            | 在所有水平上干扰。                        |

## 所需试剂

| 试剂种类         | 所需数量<br>每次测试 | 单位            | 货号       |
|--------------|--------------|---------------|----------|
| 三唑粉末试剂 ..... | 1 包.....     | 100/pkg ..... | 21412-99 |

## 所需仪器

|                                |         |             |          |
|--------------------------------|---------|-------------|----------|
| UV 安全护目镜 .....                 | 1 ..... | 个 .....     | 21134-00 |
| 样品比色瓶 10-20-25 mL, w/cap ..... | 2 ..... | 6/pkg ..... | 24019-06 |

## 根据所用电压选择以下一种:

|                             |         |         |          |
|-----------------------------|---------|---------|----------|
| 带电源的紫外灯, 115 V, 60 Hz ..... | 1 ..... | 个 ..... | 20828-00 |
| 带电源的紫外灯, 230 V, 50 Hz.....  | 1 ..... | 个 ..... | 20828-02 |

## OPTIONAL REAGENTS

|                                                 |                  |          |
|-------------------------------------------------|------------------|----------|
| Benzotriazole Standard Solution, 500 mg/L ..... | 100 mL .....     | 21413-42 |
| Rochelle Salt Solution.....                     | 29 mL* DB .....  | 1725-33  |
| Sulfuric Acid Standard Solution, 1.00 N.....    | 100 mL MDB ..... | 1270-32  |
| Water, deionized .....                          | 4 L .....        | 272-56   |

## OPTIONAL APPARATUS

|                                                  |                   |          |
|--------------------------------------------------|-------------------|----------|
| Flask, volumetric, Class A, 1000 mL.....         | each .....        | 14574-53 |
| Lamp, UV (lamp only) .....                       | each .....        | 20823-00 |
| pH Paper, 1 to 11 pH .....                       | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>sension™</i> I, portable .....      | each .....        | 51700-00 |
| Pipet Filler, safety bulb.....                   | each .....        | 14651-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....              | each .....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....    | 50/pkg .....      | 21856-96 |
| Pipet Tips, for 19700-01 TenSette Pipet .....    | 1000/pkg .....    | 21856-28 |
| Pipet, volumetric, 10.0 mL, Class A .....        | each .....        | 14515-38 |
| Stopwatch .....                                  | each .....        | 14645-00 |
| Thermometer, mercury-filled, -20 to 110 °C ..... | each .....        | 566-01   |
| Timer, interval, 1 second to 99 hours .....      | each .....        | 23480-00 |

## *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 硼 低量程 (0 to 1.60 mg/L as B)

方法号: 10061

### 甲亚胺-H 法



1. 输入检测硼 (Boron) 的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: 3 ENTER  
屏幕将显示:  
mg/L、B 和 ZERO 图标

注: 如测试另一种形态物质 ( $H_3BO_3$ ), 按下: CONC 键

3. 将 25ml 的纯净水注入一个干净的塑料烧瓶。(空白试样)

注: 为进行精确的测试, 应在每次样品检测时进行空白测试。

4. 将 25ml 的测试样品注入另一个干净的锥形烧瓶。

注: 如果样品颜色很深、混浊或含有干扰杂质, 其处理请参考干扰部分样品预处理环节。



5. 分别向两个烧瓶各加入 10 滴的 1M 乙二胺四乙酸 (EDTA) 溶液。

6. 向装有测试样品的那个烧瓶中加入一包 BoroTrace #2 试剂粉包。

7. 迅速盖上烧瓶, 大力旋转摇晃使粉末完全溶解。

注: 马上进行第 8 和第 9 步。

8. 按下  
TIMER 回车  
开始 10 分钟反应计时。



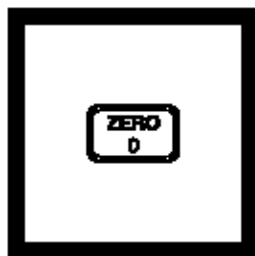
9. 继续剧烈摇晃 30 秒。在计时反应过程中应使烧瓶保持密封。

10. 在反应过程期间，把 另 外 一 包 BoroTrace #2 试剂粉包加入到装有空白试样的烧瓶中。

11. 迅速盖上烧瓶，大力旋转摇晃使粉末完全溶解。

12. 计时器鸣叫后，加入一包BoroTrace #3试剂粉包到每个烧瓶中。盖好瓶盖并摇晃使之溶解。

注：BoroTrace #3试剂的作用是使反应“停止”。



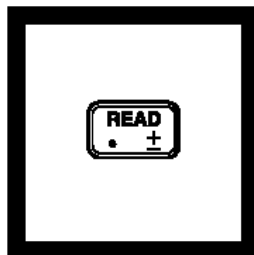
13. 将装空白试样的烧瓶中的溶液转移一支干净的比色瓶中。同时将装有预制试样的烧瓶中的溶液转移到另外一支比色瓶中。最后为两支比色瓶记上适当的标签。

14. 擦去比色瓶上指纹或其它痕迹。  
注：先用湿毛巾擦，后用干毛巾擦。这样可以擦去比色瓶上指纹或其它痕迹。

15. 将装有空白试剂的比色瓶放入样品适配器中。盖紧遮光盖。

16. 按 **ZERO**。指针将向右移动。然后屏幕将显示：

0.0 mg/L B



17. 将装有预制试剂的比色瓶放入样品适配器中。盖紧遮光盖。

18. 按 **READ**。指针将向右移动。然后屏幕将显示硼的含量，单位是 mg/L。

*注：根据样品温度要进行温度校正。如外界温度是22-24° C (72-75 ° F)时，请参考样品温度补偿部分的内容。*

*注：可能要用标准溶液进行标准校正。*

## 采样、保存和存放

收集样本到一个干净的聚乙烯瓶子中。不要用含硼基的清洁剂和肥皂清洗容器或实验室用于该测试的实验器具。所以塑料的器具在使用完之后，应该用大量的去离子水进行清洗，然后风干密封存放。

## 精确检测

### 加标测试法

- 将5.0毫升的含量为1000毫克/升的硼溶液注入容积为100毫升的烧瓶中，然后用去离子水稀释成50毫克/升的硼标准溶液，并且塞住盖好摇晃直到溶液彻底混合为止。
- 使用TenSette移液管分别各自往3个25毫升的水样本中注入0.1毫升、0.2毫升、0.3毫升的硼含量为50毫克/升的标准溶液。
- 根据上述步骤进行测试分析每个样品。
- 硼的浓度应该是每增加0.1毫升硼标准溶液就增加0.20毫克/升。

### 标准溶液法

使用一个塑料移液管，将4.0毫升的浓度为250毫克/升的硼标准溶液注入一个容积为1000毫升的塑料烧瓶内，然后用去离子水进行稀释，塞住盖好直到彻底混合为止。之后在第四步的时候，取该含量为1.0毫克/升的标准液作为样本，根据上述步骤进行分析。

## 样本的温度补偿

该反应的化学性质是取决于样本的温度的。哈西的标定是23° C (73 ° F)。如果样本的温度超出22-24 ° C (72-75 ° F)，就应该在第十八步所得结果乘以相应的放大系数来增大结果。

| Sample Temperature |    | Multiplier |
|--------------------|----|------------|
| °C                 | °F |            |
| 5                  | 41 | 0.70       |
| 7                  | 44 | 0.73       |
| 10                 | 50 | 0.78       |
| 12                 | 53 | 0.81       |
| 14                 | 57 | 0.84       |
| 16                 | 61 | 0.87       |
| 18                 | 64 | 0.91       |
| 20                 | 68 | 0.94       |
| 25                 | 77 | 1.04       |
| 26                 | 79 | 1.06       |
| 27                 | 81 | 1.08       |
| 28                 | 82 | 1.10       |
| 29                 | 84 | 1.12       |
| 30                 | 86 | 1.15       |

## 检测方法的效果

### 精度

在一个单独的实验室，如果在使用1.0毫克/升的标准硼溶液和仪器所带的两种典型试剂，单个测试人员所得结果的标准误差是±0.04毫克/升。欲了解更详细的情况请参考第一部分的相关内容。

### 预计检测极限

程序4的预计检测极限是硼的含量为0.03毫克/升。欲了解更详细的检测极限方面的情况请参考第一部分的相关内容。

## 干扰

以下物质经过测试，结果表明在低于以下所示水平时不会产生干扰 (mg/L):

| 干扰物质                   |                   | 干扰水平                        |
|------------------------|-------------------|-----------------------------|
| 铝( <sup>3+</sup> )     |                   | 10                          |
| 苯并噻唑                   |                   | 20                          |
| 生物灭杀剂                  | 氨基甲酸盐形式           | 120                         |
|                        | Isothiazolin-type | 120                         |
|                        | Quat-type         | 90                          |
|                        | 硫氰酸盐形式            | 60                          |
| 钙                      |                   | 1000 (如CaCO <sub>3</sub> )  |
| 氯化物                    |                   | 2500                        |
| 铜 ( <sup>2+</sup> )    |                   | 20                          |
| 镁                      |                   | 1000 (如 CaCO <sub>3</sub> ) |
| 锰( <sup>7+</sup> )     |                   | 5                           |
| 钼酸盐(Mo <sup>6+</sup> ) |                   | 60                          |
| 膦酸盐, AMP               |                   | 20                          |
| 膦酸盐, HEDP              |                   | 20                          |
| Polyacrylates          |                   | 20 (as Acumer 1000, 1100)   |
| Polymaleic Acid        |                   | 40 (as Belcene 200)         |
| 硅                      |                   | 120                         |
| 硫酸盐                    |                   | 1800                        |
| 亚硫酸盐                   |                   | 40                          |
| Tolyltriazole          |                   | 20                          |
| 锌( <sup>2+</sup> )     |                   | 10                          |

## 干扰物质和建议的处理方法

| 干扰物质, 干扰水平 (正或负干扰)  | 推荐的处理方法                                                                                                                                                                                       |
|---------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 碱度 >500 mg/L (+或 -) | <ol style="list-style-type: none"> <li>1. 用1.0N的硫酸溶液调节样品的pH到5–7之间。</li> <li>2. 继续分析程序中的步骤5。</li> </ol>                                                                                        |
| 颜色 (+)              | <ol style="list-style-type: none"> <li>1. 用高纯水对仪器调零。(0.00 mg/L B)</li> <li>2. 从样品的颜色估量并记录表观浓度, 以mg/L B表示。</li> <li>3. 从测试程序步骤15所得结果减去表观浓度。</li> </ol>                                         |
| 卤素 (溴或氯)所有水平(+)     | <p>样品中的卤素消毒剂在加入BoroTrace #2后会产生红色。可按如下消除干扰:</p> <ol style="list-style-type: none"> <li>1. 分别加入1包Dechlorinating 试剂到 25-mL 高纯水和样品中。</li> <li>2. 盖上盖子并摇晃使溶解。</li> <li>3. 继续测试程序中的步骤5。</li> </ol> |

|                                                              |                                                                                                                                                                                                             |
|--------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 铁 ( $\text{Fe}^{3+}$ or $\text{Fe}^{2+}$ ),<br>超过 8 mg/L (+) | <p>样品中高水平的铁在加入BoroTrace #2试剂后将产生红色。</p> <p>为了补偿,将加入到每个样品管中的EDTA的量从10滴增加到15滴(步骤5),或者用高纯水稀释样品并继续测试程序中的步骤5。用合适的稀释因子修正结果(步骤5)。</p>                                                                              |
| 亚硝酸盐,所有水平(+)                                                 | <ol style="list-style-type: none"> <li>1. 分别加入0.1克硫酸到塑料管中25mL的高纯水和样品中。</li> <li>2. 盖好盖子并摇晃使溶解。</li> <li>3. 打开盖子并等待5分钟。</li> <li>4. 分别加入 5N氢氧化钠溶液到 每个管中,用pH试纸调节pH 5 - 8。</li> <li>5. 继续测试程序中的步骤5。</li> </ol> |
| 浑浊 (+)                                                       | 测试前用3 $\mu\text{m}$ 滤膜过滤样品。不要使用玻璃纤维过滤器。                                                                                                                                                                     |

## 所需试剂

|                                                            | 货号       |
|------------------------------------------------------------|----------|
| BoroTrace 试剂一套 .....                                       | 26669-00 |
| 包括: (1) 26666-69, (1) 26667-46, (1) 22419-26, (1) 25946-49 |          |

| 试剂种类                        | 所需数量<br>每次测试 | 单位         | 货号       |
|-----------------------------|--------------|------------|----------|
| BoroTrace 2 粉末试剂.....       | 2            | 100/pkg    | 26666-69 |
| BoroTrace 3 粉末试剂.....       | 2            | 100/pkg    | 26667-99 |
| 乙二胺四乙酸 (EDTA) 溶液, 1 M ..... | 20 滴         | 50 mL SCDB | 22419-26 |
| 纯净水, 不含乙醛.....              | 25 mL        | 500 mL     | 25946-49 |

## 所需仪器

|                                      |   |       |          |
|--------------------------------------|---|-------|----------|
| 剪刀, for opening powder pillows ..... | 1 | 个     | 968-00   |
| 锥形烧瓶, , 50-mL, w/cap .....           | 2 | 个     | 20898-41 |
| 样品比色瓶, 10-20-25 mL, w/cap .....      | 2 | 6/pkg | 24019-06 |

## OPTIONAL REAGENTS

|                                                                     |            |          |
|---------------------------------------------------------------------|------------|----------|
| Boron Standard Solution, 250 mg/L as B, 10-mL Voluette Ampule ..... | 16/pkg     | 14249-10 |
| Boron Standard Solution, 1000 mg/L as B .....                       | 100 mL     | 1914-42  |
| Dechlorinating Reagent Powder Pillows.....                          | 100/pkg    | 14363-69 |
| Eluant Solution.....                                                | 1 L        | 22256-53 |
| Phosphate Buffer Solution, pH 7.2.....                              | 1 L        | 431-53   |
| Sodium Hydroxide Standard Solution, 5.0 N.....                      | 50 mL DB   | 2450-26  |
| Sulfamic Acid.....                                                  | 113 g      | 2344-14  |
| Sulfuric Acid Standard Solution, 1.000 N.....                       | 100 mL MDB | 1270-32  |
| Water, deionized .....                                              | 4 L        | 272-56   |

## OPTIONAL APPARATUS

|                                                       |        |          |
|-------------------------------------------------------|--------|----------|
| Ampule Breaker Kit (for 10-mL ampules).....           | each   | 21968-00 |
| Filter Holder Assembly .....                          | each   | 2468-00  |
| Flask, volumetric, polypropylene, 100 mL, w/cap.....  | each   | 14060-42 |
| Flask, volumetric, polypropylene, 1000 mL, w/cap..... | each   | 14060-53 |
| Membrane Filters, 3-micron.....                       | 25/pkg | 25940-25 |
| pH Paper, pH 1-11.....                                | each   | 391-33   |
| Pipet, Mohr-type, polypropylene, 5 mL .....           | each   | 2106-37  |
| Pipet, TenSette, 0.1 to 1.0 mL.....                   | each   | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....         | each   | 21856-96 |
| Spoon, measuring, 0.1 g .....                         | each   | 511-00   |
| Syringe, 30-cc.....                                   | each   | 22258-00 |
| Thermometer, -10 to 110 °C.....                       | each   | 1877-01  |

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溴 (0 to 4.50 mg/L)

方法号: 8016

DPD 法

使用试剂粉包



1. 输入检测溴的试剂粉法的预定程序编号。

按下: PRGM

屏幕将显示:

PRGM?



2. 按下: 5 ENTER  
屏幕将显示:  
0.00 mg/L Br<sub>2</sub> 和  
ZERO 图标。



3. 往比色瓶中装入  
10 mL 样品 (空白试  
样)。

注: 样品应马上进行  
分析, 不能保存留待  
以后使用。



4. 将空白试样放入样  
品适配器, 并用盖紧  
遮光盖。



5. 按 ZERO, 屏幕显  
示:  
0.00 mg/L Br<sub>2</sub>。

注: 如果正在进行样  
本空白校正, 则屏幕  
将闪烁显示 "limit"  
字样。



6. 将一包 DPD 总氯试  
剂粉加入到装有样品  
的比色瓶中。大力晃  
动使粉剂溶解。(预制  
试样)

注: 不需要粉剂完全  
溶解。如果溴存在,  
则溶液呈现粉红色。



7. 按下:  
TIMER ENTER  
将开始 3 分钟的反应  
计时。



8. 当计时器鸣响后,  
将预制试样放入样品  
适配器中, 并盖上遮  
光盖。



9. 按下：READ      注：应使用预制的标准溶液进行标准校正。  
指针将右移，屏幕将显示溴的含量，单位为 mg/L，

注：如果溴含量过高，加入试剂后，溶液将临时呈现黄色。此时应稀释样品然后再进行检测。在稀释过程中，少量的溴将会损失，应将结果乘以一定的稀释因子。

## 采样和存放

溴样本一经采集应该立即进行分析。

不要使用塑料容器，否则会产生大量的溴化作用。

应该将所用的玻璃容器进行预处理从而除去可能导致溴化因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水清洗过，那么预处理过程就不需要经常进行。

一个测试溴的时候常见的错误就是没有采集到具有代表性的样本。正确的做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后注入样本到10毫升的刻度。样本采集完之后应该马上进行溴分析。

## 精确检测

### 加标测试法（药剂法）

- 量取一定数量LR Chlorine PourRite® Ampule标准溶液。
- 使用TenSette移液管将0.1毫升的该溶液注入已经充分反应的预制试样（混合样本）中。
- 使用原始样本（空白试样）来使设备读数清零。
- 将预制试样放入样品管槽，按READ，记录读数。
- 计算加入样本的溴的当量浓度（毫克/升）：

$$\text{所需加入溴的当量浓度 (mg/L)} = \frac{0.1(\text{标准溶液}) \times \text{标称值 (mg/L氯气)} \times 2.25}{10.1(\text{样本和标准数})}$$

- f) 预制样本的结果（步骤d）应该等于所分析的样本实际值加上所计算的溴的当量浓度值（步骤e）。
- g) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

#### 加标测试法（安培瓶法）

- a) 量取LR Chlorine PourRite® Ampule标准溶液。
- b) 使用量筒量各取25毫升的样本到两个烧杯中。
- c) 使用移液管往每个烧杯注入0.2毫升的标准溶液然后旋转使之混合。（这就是预制试样）
- d) 在每个烧杯中放入 一个DPD Total 氯试剂安培瓶。
- e) 按照上述步骤分析预制试样和空白试样。
- f) 计算加入到样本中的溴的当量浓度（毫克/升）

$$\text{所需加入溴的当量浓度 (mg/L)} = \frac{0.2(\text{标准溶液}) \times \text{标称值 (mg/L氯气)} \times 2.25}{25.2(\text{样本和标准数})}$$

- g) 预制试样的结果应该等于所要分析样本的实际值加上溴的当量浓度（步骤f）。
- h) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

#### 检测方法的效果

##### 精度

在一个单独的实验室，如果使用2.34毫克/升的标准溴溶液和仪器所附带的试剂，单个测试人员所得结果的标准误差是±0.02毫克/升。

在一个单独的实验室，如果使用2.31毫克/升的标准溴溶液和安培瓶法，单个测试人员所得结果的标准误差是±0.02毫克/升。

##### 预计检测极限

程序5的预计检测极限是溴含量为0.04毫克/升。程序6的预计检测极限是溴含量为0.03毫克/升，欲了解更详细的检测极限方面的情况请参考第一部分的相关内容。

## 干扰

| 干扰物质                                                                         | 干扰水平和处理                                                                                                                                                                                                                                    |
|------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 酸度                                                                           | 大于150 mg/L的CaCO <sub>3</sub> ，不会生成颜色或颜色很快褪去。用1N的氢氧化钠中和到pH6-7。计算加入每个单独样品中的氢氧化钠的量，然后加入相同的量到测试中的样品中。修正体积的增加。                                                                                                                                  |
| 碱度                                                                           | 大于 250 mg/L 的CaCO <sub>3</sub> 。不会生成颜色或颜色很快褪去。用1N的硫酸中和到pH6-7。计算加入每个单独样品中的硫酸的量，然后加入相同的量到测试中的样品中。修正体积的增加。                                                                                                                                    |
| 氯                                                                            | 所有水平上均干扰。                                                                                                                                                                                                                                  |
| 二氧化氯                                                                         | 所有水平上均干扰。                                                                                                                                                                                                                                  |
| 氯胺，有机氯胺                                                                      | 可能干扰                                                                                                                                                                                                                                       |
| 硬度                                                                           | 低于1,000 mg/L的 CaCO <sub>3</sub> 不干扰。                                                                                                                                                                                                       |
| 碘                                                                            | 所有水平上均干扰。                                                                                                                                                                                                                                  |
| 锰，氧化锰(Mn <sup>4+</sup> , Mn <sup>7+</sup> )<br>或<br>铬，氧化铬(Cr <sup>6+</sup> ) | <ol style="list-style-type: none"> <li>1. 调节样品 pH 到 6 - 7。</li> <li>2. 加入3 滴碘化钾(30 g/L)到25mL样品中。</li> <li>3. 混合并等待片刻。</li> <li>4. 加入3亚砷酸钠(5 g/L)并混合。</li> <li>5. 分析10mL程序中所述的处理过的样品。</li> <li>6. 从原始分析结果中减去这次分析得到的结果，即得到正确的溴浓度。</li> </ol> |
| Monochloramine                                                               | 所有水平上均干扰。                                                                                                                                                                                                                                  |
| 臭氧                                                                           | 所有水平上均干扰。                                                                                                                                                                                                                                  |
| 过氧化物                                                                         | 可能干扰。                                                                                                                                                                                                                                      |
| 极端样品pH                                                                       | 调节pH6 - 7。                                                                                                                                                                                                                                 |
| 高缓冲样品                                                                        | 调节pH 6 - 7。                                                                                                                                                                                                                                |

## 方法总结

溴和 DPD 试剂反应所形成的紫红色的深浅是和溴的浓度成正比的。

## Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interference will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

**所需试剂(使用试剂粉包)**

| 试剂种类             | 所需数量<br>每次测试 | 单位           | 货号       |
|------------------|--------------|--------------|----------|
| DPD 总氯粉末试剂 ..... | 1 包 .....    | 100/pkg..... | 21056-69 |

**所用试剂(使用安瓿瓶法)**

|                           |          |             |          |
|---------------------------|----------|-------------|----------|
| DPD 总氯试剂 AccuVac 安瓿瓶..... | 1瓶 ..... | 25/pkg..... | 25030-25 |
|---------------------------|----------|-------------|----------|

**所需仪器(使用试剂粉末)**

|                                  |            |          |
|----------------------------------|------------|----------|
| 样品比色瓶, 10-20-25-mL, w/ cap ..... | 6/pkg..... | 24019-06 |
|----------------------------------|------------|----------|

**所需仪器 (使用安瓿瓶)**

|                 |        |           |        |
|-----------------|--------|-----------|--------|
| 烧瓶 , 50 mL..... | 1..... | each..... | 500-41 |
|-----------------|--------|-----------|--------|

**OPTIONAL REAGENTS**

|                                                                     |                  |          |
|---------------------------------------------------------------------|------------------|----------|
| Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL ..... | 20/pkg.....      | 26300-20 |
| Potassium Iodide Solution, 30 g/L.....                              | 100 mL* MDB..... | 343-32   |
| Sodium Arsenite, 5 g/L.....                                         | 100 mL* MDB..... | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N.....                    | 100 mL* MDB..... | 1045-32  |
| Sulfuric Acid Standard Solution, 1 N .....                          | 100 mL* MDB..... | 1270-32  |
| Water, deionized.....                                               | 4 L.....         | 272-56   |

**OPTIONAL APPARATUS**

|                                                                    |                  |          |
|--------------------------------------------------------------------|------------------|----------|
| AccuVac Snapper Kit.....                                           | each.....        | 24052-00 |
| PourRite Ampule Breaker.....                                       | each.....        | 24846-00 |
| Cylinder, graduated, 25 mL .....                                   | each.....        | 508-40   |
| pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> 1, portable..... | each.....        | 51700-00 |
| pH Indicator Paper, 1 to 11 pH units .....                         | 5 rolls/pkg..... | 391-33   |
| Pipet, TenSette, 0.1 to 1.0 mL.....                                | each.....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....                      | 50/pkg.....      | 21856-96 |

***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

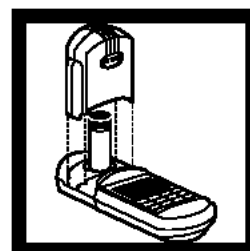
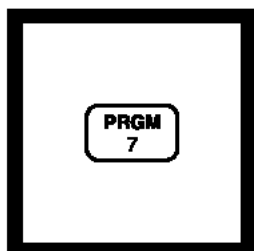
**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

\* Contact Hach for larger sizes

## 二氧化氯 (0 to 5.00 mg/L)

方法号: 10126

### DPD 方法 使用试剂粉包



1. 输入检测二氧化氯试剂粉法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

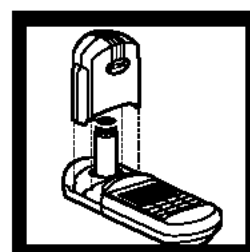
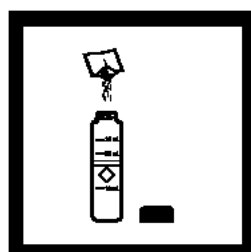
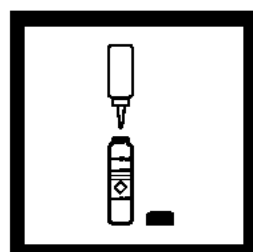
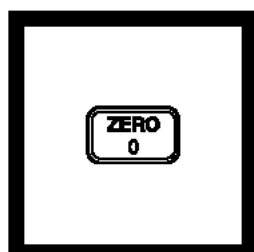
2. 按下: 112 ENTER  
屏幕将显示:

0.00 mg/L ClO<sub>2</sub> 和  
ZERO 图标。

3 往比色瓶中装入 10 mL 样品 (空白试样)。

4. 将空白试样瓶放入样品适配器, 并用盖紧遮光盖。

*注: 为得到最佳结果, 应使用去离子水作为样平品测得试剂的空白校正值。然后从样品的读数减去空白校正值可得最终结果。*



5. 按 ZERO, 屏幕显示:

0.00 mg/L ClO<sub>2</sub>

*注: 如果正在进行样本空白校正, 则屏幕将闪烁显示“limit”字样。*

6. 加入 4 滴氨基乙酸 (Glycine) 试剂到样品中, 晃动使之混合。(预制试样)

7. 加 DPD 自由氯试剂粉包到装有预制试样的比色瓶中。盖上遮光盖, 并旋转使溶液混合。

*注: 如果存在二氧化氯溶液将呈粉红色。  
注: 加入试剂1分钟之内应实施步骤9的过程。*

8. 等待 30 秒使不溶解的粉末沉淀。然后将预制试样瓶放入样品适配器中, 并盖上遮光盖。

*注: 在将样品放入之前, 应擦去比色瓶表面的液体和手印。*



#### 9. 按下: READ

指针将右移, 屏幕将显示二氧化氯的含量, 单位为 mg/L,

*注: 如果样品中二氧化氯的含量超过测试的最大限度, 颜色将呈现黄色。此时应用不含氯的高纯水稀释样品, 然后再重复测试。由于稀释可能引起氯的损失, 因此结果要乘以一定的稀释因子。*

### 使用 AccuVac 安瓿瓶



1. 输入检测二氧化氯 AccuVac 安瓿瓶法程序编号。

按下: PRGM

屏幕将显示:

PRGM?



2. 按下 113 回车 屏幕将显示:

0.00 mg/L ClO<sub>2</sub> 和 ZERO 图标



3. 将至少 10ml 空白试样注入比色瓶。在一个容积为 50ml 的烧杯内注入 40ml 的待测样品。使用正确数量的样品是十分重要的。

*注: 样品必须马上进行分析, 而不能留给以后的分析用。*

*注: 在将比色瓶放入之前, 应擦去表面的液体和手印。*



4. 将空白试样瓶放入样品适配器中, 并盖上遮光盖。

*注: 为得到最佳结果, 应使用去离子水作为样品测得试剂的空白校正值。然后从样品的读数减去空白校正值得最终结果。*



5. 按 **ZERO**，屏幕显示：  
**0.00 mg/L ClO<sub>2</sub>**。

注：如果正在进行样本空白校正，则屏幕将闪烁显示“limit”字样。



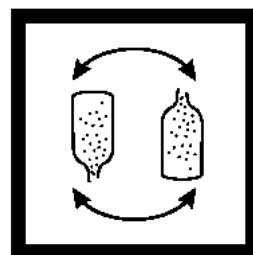
6. 加入 16 滴氨基乙酸（Glycine）试剂到装有样品的烧瓶中，晃动使之混合。（预制试样）



7. 将烧瓶中的待测样本注入一个装有脱氯 DPD 试剂的 AccuVac 安瓿瓶内。

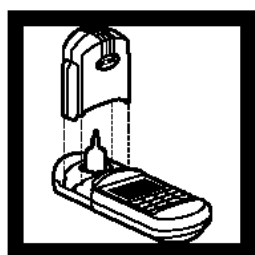
注：在注入时，应将瓶口完全浸没在溶液中。

注：在加入试剂的 1 分钟之内，应进行步骤 10 的过程。

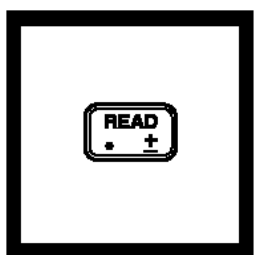


8. 迅速地翻转安瓿瓶多次，使溶液充分混合。擦去瓶表面的液体和手印。

注：如果二氧化氯存在，溶液将呈粉红色。



9. 等待 30 秒使不溶解的粉末沉淀。然后将安瓿瓶放入样品管槽，并盖紧样品瓶盖。



10. 按下：**READ** 指针将右移，屏幕将显示二氧化氯的含量，单位为 mg/L，

注：如果样品中二氧化氯的含量超过测试的最大限度，颜色将呈现黄色。此时应用不含氯的高纯水稀释样品，然后再重复测试。由于稀释可能引起氯的损失，因此结果要乘以一定的稀释因子。

## 采样和存放

二氧化氯的样本一经采样应该马上进行分析。二氧化氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地和大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等都会影响二氧化氯在水中的分解情况。

由于会产生氯化作用，因此不要使用塑料容器。

应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

检测二氧化氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

## 精确检测

因为生产二氧化氯是十分困难且危险，所以用含氯标准样品来检测DPD和甘氨酸试剂。过程如下所述：

1. 准备好含量为1毫克/升的自由氯标准样品。

### 方法1

- a. 获取自由氯标准样品（目录编号 14268-10）
- b. 从随标准样品附带的分析证书上获得标准样品的浓度(范围是50-75毫克/升)。用以下公式计算出所需标准溶液的容量：

所需标准样品的容量（毫升）=  $100 \div \text{标准溶液的浓度}$

- c. 按照所需容量，将标准样品注入容积为100毫升的烧瓶内，用不含氯的高纯水稀释样品，倒转使之混合完全。

### 方法2

- a. 稀释1 滴的含量5%商业氯水漂白剂到1 公升自由氯除去离子水中。并使用其作为标准样本。
2. 使用哈西自由氯方法检测标准样本的浓度，#8021.
  3. 在不增加甘氨酸条件下，对标准样本进行二氧化氯的检测。（第 6 步骤）
  4. 二氧化氯的读数应该是大约氯的检测结果的 2.45倍或以上。 如果结果正确，这证明DPD和仪器工作正常。
  5. 在添加甘氨酸条件下，重复进行对标准样本进行二氧化氯的检测（第 6 步骤） 读数应该少于 0.10毫克 / 升. 这证明了甘氨酸去除了自由氯的干扰。

## 检测方法的效果

### 精度

#### Precision

| <u>Program</u> | <u>Standard</u> | <u>95% Confidence Limits</u>    |
|----------------|-----------------|---------------------------------|
| 112            | 0.24 mg/L       | 0.22–0.26 mg/L ClO <sub>2</sub> |
| 112            | 4.79 mg/L       | 4.67–4.91 mg/L ClO <sub>2</sub> |
| 113            | 0.26 mg/L       | 0.21–0.27 mg/L ClO <sub>2</sub> |
| 113            | 4.83 mg/L       | 4.71–4.97 mg/L ClO <sub>2</sub> |

如果想了解检测精度和测试方法的限度的更多信息请参考操作过程手册的第一章节。

**预计检测极限(EDL)**

| <b><u>Program</u></b> | <b><u>EDL</u></b>          |
|-----------------------|----------------------------|
| 112                   | 0.04 mg/L ClO <sub>2</sub> |
| 113                   | 0.04 mg/L ClO <sub>2</sub> |

如果想了解测试误差和测试极限的更多信息请参考操作过程手册的第一章节。

## 干扰

如果一种物质会影响到结果偏差0.1mg/L或以上的，这就叫做引起干扰。

| 干扰物质                                                                     | 干扰水平和处理                                                                                                                                                                      |
|--------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 酸度                                                                       | 大于150 mg/L的 $\text{CaCO}_3$ 。可能导致生色不充分或颜色很快褪去。用1N的氢氧化钠中和到pH6-7。测定加入到每个单独样品中所用氢氧化钠用量，并在测试的样品中加入相同的量。校正体积的增加（参阅1.2.2部分之“体积增加的校正”）。                                             |
| 碱度                                                                       | 大于250 mg/L 的 $\text{CaCO}_3$ 。可能导致生色不充分或颜色很快褪去。用1N的硫酸中和到pH6-7。测定加入到每个单独样品中所用硫酸的用量，并在测试的样品中加入相同的量。校正体积的增加（参阅1.2.2部分之“体积增加的校正”）。                                               |
| 溴, $\text{Br}_2$                                                         | 所有水平上均干扰。                                                                                                                                                                    |
| 氯, $\text{Cl}_2$                                                         | 大于6mg/L的水平可能干扰。额外的氨基乙酸可补偿干扰。                                                                                                                                                 |
| 氯胺, 有机氯胺                                                                 | 可能干扰。                                                                                                                                                                        |
| 凝絮剂                                                                      | 可允许多数高水平的凝絮剂。如氯存在，容许限度降低。参阅表中关于金属的信息。当存在0.6mg/L $\text{Cl}_2$ ， $\text{Al}_2(\text{SO}_4)_3$ (< 500 mg/L)， $\text{FeCl}_2$ (<200 mg/L)可允许。                                   |
| 硬度                                                                       | 低于1000mg/L的 $\text{CaCO}_3$ 不影响。                                                                                                                                             |
| 碘, $\text{I}_2$                                                          | 所有水平上均干扰。                                                                                                                                                                    |
| 氧化锰 ( $\text{Mn}^{4+}$ , $\text{Mn}^{7+}$ )<br>或氧化铬 ( $\text{Cr}^{6+}$ ) | 氧化锰在全程产生干扰，氧化铬在大于2 mg/L产生干扰，去干扰方法：<br>1. PH调至6-7；<br>2. 加三滴碘化钾 (30 g/L) 到25ml样品中。<br>3. 混合等待一分钟<br>4. 加三滴亚砷酸钠 (5 g/L) 混和<br>5. 取10ml以上样品测试。<br>6. 将原始结果减去以上测试结果可得正确的二氧化氯的结果。 |
| 金属                                                                       | 大量的金属将会和氨基乙酸化合从而产生干扰，而所加入氨基乙酸本来是为了清除氯的干扰用的。除非氯的存在，否则金属的干扰是有限的。当氯的含量达到0.6mg/L时，铜 (>10mg/L) 和镍 (>50 mg/L) 会产生干扰。其它金属也会产生干扰，其主要取决于它阻止氨基乙酸和样品中的氯进行反应的能力。因此要克服干扰应加入更多的氨基乙酸。       |
| 一氯化物                                                                     | 会导致读数偏高。在加入试剂后一分钟内读取读数，3 mg/L的一氯化物会导致二氧化氯的读数增加0.1mg/L。                                                                                                                       |
| 臭氧                                                                       | >1.5mg/L有干扰                                                                                                                                                                  |
| 过氧化物                                                                     | 可能有干扰                                                                                                                                                                        |
| 大量缓冲溶液                                                                   | PH调至6-7                                                                                                                                                                      |
| 极端PH                                                                     | PH调至6-7                                                                                                                                                                      |

## 所需试剂 (使用试剂粉包)

| 试剂种类                                       | 所需数量<br>每次测试 | 单位            | 货号       |
|--------------------------------------------|--------------|---------------|----------|
| 二氧化氯DPD/氨基乙酸试剂一套 (100 tests).....          |              |               | 27709-00 |
| 包括以下其中一种:                                  |              |               |          |
| DPD 自由氯粉末试剂flows, 10 mL .....              | 1 包.....     | 100/pkg ..... | 21055-69 |
| 氨基乙酸试剂 .....                               | 4 滴 .....    | 29 mL .....   | 27621-33 |
| 所用试剂(使用AccuVac® 安瓿瓶)                       |              |               |          |
| 二氧化氯DPD/氨基乙酸AccuVac®安瓿瓶试剂一套(25 tests)..... |              |               | 27710-00 |
| 包括以下其中一种:                                  |              |               |          |
| DPD 自由氯 AccuVac® 安瓿瓶 .....                 | 1 .....      | 25/pkg .....  | 25020-25 |
| 氨基乙酸试剂.....                                | 16 滴 .....   | 29 mL .....   | 27621-33 |

## OPTIONAL REAGENTS

|                                                   |                    |  |          |
|---------------------------------------------------|--------------------|--|----------|
| Chlorine Standard Solution, Voluette™ ampule,     |                    |  |          |
| 50-75 mg/L, 10 mL .....                           | 16/pkg .....       |  | 14268-10 |
| DPD Free Chlorine Reagent w/dispensing cap.....   | 250 tests .....    |  | 21055-29 |
| Potassium Iodide Solution, 30 g/L .....           | 100 mL * MDB ..... |  | 343-32   |
| Sodium Arsenite, 5 g/L .....                      | 100 mL * MDB ..... |  | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N ..... | 100 mL * MDB ..... |  | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N .....    | 100 mL * MDB ..... |  | 1270-32  |
| Water, deionized.....                             | 4 L .....          |  | 272-56   |
| Water, sterile, chlorine dioxide-free.....        | 500 mL .....       |  | 26415-49 |

## OPTIONAL APPARATUS

|                                                        |                   |  |          |
|--------------------------------------------------------|-------------------|--|----------|
| AccuVac® Snapper Kit .....                             | each .....        |  | 24052-00 |
| Cylinder, graduated, 25 mL .....                       | each .....        |  | 508-40   |
| pH Meter, <i>sens<sup>ion</sup></i> ™I, portable ..... | each .....        |  | 51700-10 |
| pH Paper, 1 to 11 pH units.....                        | 5 rolls/pkg ..... |  | 391-33   |
| Pipet, TenSette®, 0.1 to 1.0 mL .....                  | each .....        |  | 19700-01 |
| Pipet Tips, for 19700-01 TenSette® Pipet .....         | 50/pkg .....      |  | 21856-96 |
| PourRite™ Ampule Breaker.....                          | each .....        |  | 24846-00 |

### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

\* Marked Dropper Bottle - contact Hach for larger sizes.

## 二氧化氯 直读法 中量程 (0—50.0 mg/L)

方法号: 8345



1. 输入检测二氧化氯直读法程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

2. 按程序号: 7

再按下: **ENTER**

屏幕将显示:

**0.00 mg/L ClO<sub>2</sub>** 和

**ZERO** 图标

3. 用 10ml 去离子水注入一只比色管。(空白试样)

*注: 样品一经采集应马上进行分析。*

4. 将空白试样瓶放入样品适配器中, 并盖上遮光盖。



5. 按 **ZERO**, 屏幕显示:

**0.00 mg/L ClO<sub>2</sub>**。

6. 将待测样品注入另一支比色瓶中。(预制试样)

7. 将装有预制试样的比色瓶放入样品适配器中。盖上遮光盖, 并旋转使溶液混合。

8. 按下: **READ**  
指针将右移, 屏幕将显示二氧化氯的含量, 单位为 mg/L。

*注: 如果屏幕闪烁显示“limit”, 这是由于样品中二氧化氯的含量超过测试的最大限度。应稀释样品, 然后再重复测试。由于稀释可能引起氯的损失, 因此结果要乘以一定的稀释因子。*

### 采样和存放

将样品采集到干净的塑料或玻璃瓶中。由于二氧化氯是易挥发和不稳定的, 因此样品一经采样应该立即进行检测。

## 精确检测

### 标准溶液法

准备二氧化氯的标准样品是困难的和危险的。另外，这些标准样品是可爆炸的和可挥发的！只有经过训练的化学人员在使用适当的安全仪器和进行预防后才能准备该标准样品。本公司不推荐把二氧化氯标准样品作为单独的标准样品。如果需要准备单独的标准样品，请参考“水和废水检测所用标准样品”指引中“存储二氧化氯溶液”和“标准二氧化氯溶液”部分。

## 检测方法的效果

### 检测精度

在一个单独的实验室，如果使用25.0毫克/升的标准二氧化氯溶液，单个测试人员所得结果的标准误差是±0.3毫克/升。欲了解更详细的信息请查阅第一章。

### 预计检测极限

程序7的预计检测极限是二氧化氯含量为7.3毫克/升。欲了解更详细的信息请参考第一章的相关内容。

## 方法概述

二氧化氯是一种黄色气体，在水溶液中它能够被直接检测。该方法是使用 420 nm 的波长来增加测试的范围

## 所需试剂和仪器

| 试剂种类                            | 所需数量       |            |          |
|---------------------------------|------------|------------|----------|
|                                 | 每次测试       | 单位         | 货号       |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 2.....     | 6/pkg..... | 24019-06 |
| 去离子水.....                       | 10 mL..... | 4 L.....   | 272-56   |

### ***For Technical Assistance, Price and Ordering***

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**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 余氯 高量程 (0 to 5.00 mg/L)

方法号: 10069

### DPD 方法



1. 输入检测高量程余氯的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到更加精确的结果, 应使用去离子水进行试剂空白校正。

2. 按下 **8 ENTER** 屏幕将显示:

**0.00 mg/L**、**Cl<sub>2</sub>** 和 **ZERO** 图标。

3. 用 10ml 去离子水注入两支比色瓶中。

注: 样品一经采集应马上进行分析。不能保存留待以后使用。

4. 将一包余氯试剂粉加入到其中一支比色瓶中。(预制试剂) 盖上瓶盖, 混动使之溶解。

注: 如果存在氯, 则溶液会呈现粉红色。



5. 在加入试剂后一分钟之内, 将去离子水加入到每只比色瓶中, 直至达到 25 毫升的刻度。盖上瓶盖, 反转两次使之混合。

6. 将没有加试剂的那只比色瓶放入样品适配器中, 盖紧遮光盖。

7. 按下: **ZERO** 指针将右移, 屏幕将会显示:

**0.00 mg/L Cl<sub>2</sub>**

8. 将装有预制试样的那只比色瓶放入样品适配器中, 盖紧遮光盖。



#### 9. 按下： READ

指针将右移，屏幕将显示余氯的含量，单位是 mg/L。

*注：应使用预制标准溶液进行标准校正。*

### 采样和存放

自由氯样品一经采集应该立即进行检测。氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等都会影响氯在水中的分解情况。

由于会产生化学作用，因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

不要使用相同的试管装载自由氯和总氯的样本。如果总氯试剂中的少量碘化物渗入自由氯样本中，一氯化物将会产生干扰。最好的方法就是各自使用单独的试管来装载自由氯和总氯的样品。

检测自由氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

### 精确检测

#### 加标检测法

- 量取一定数量的LR Chlorine PourRite® Ampule标准溶液。
- 使用TenSette移液管将0.1毫升的该溶液注入10毫升的预制试样（混合样本）中，轻轻晃动使之混合。
- 从第四步开始检测预制样本。
- 计算加入到样本的氯的浓度（毫克/升）：

$$\text{所需加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- e) 预制样本的结果应该等于所分析样本值加上所计算的所加氯的浓度值（步骤d）。
- f) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

## 检测方法的效果

### 检测精度

在一个单独的实验室，如果使用2.40毫克/升的氯溶液和仪器所附带的试剂，单个测试人员所得结果的标准误差是±0.06毫克/升。

### 预计检测极限

程序8的预计检测极限是氯含量为0.03毫克/升。通常在浓度在2毫克/升以下，可使用方法# 8021来检测自由氯的浓度。如果想了解测试误差和测试极限的更多信息请参考第一章节。

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候，根据Federal RCRA for arsenic (D004)条例，样品将会成为有危险的废物。有关这些废物的处理信息请查看第三章。

## 方法概述

使用DPD方法测试自由氯时，根据样品的数量按比例地加入指示剂可以增加检测的范围。因此可以加入更大块的DPD自由氯试剂粉块到10毫升的样本中。在颜色显影之后，用去离子水将样品容量调整到25毫升。这样将允许在1英寸的试管内直接进行颜色检测，而不需要将其移入小的试管里进行。

稀释已反应的样品可以避免测试前稀释样品所带来的潜在的氯损失。由于准确性和精密度方面的损失可以被预测，因此在浓度低于2个毫克 / 升时，推荐使用方法# 8021来检测自由氯的浓度。

## 干扰

表1 干扰物质和建议的处理方法

| 干扰物质                          | 干扰水平和处理                                                                                                                                                                                                                            |
|-------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 溴                             | 所有水平上均干扰                                                                                                                                                                                                                           |
| 二氧化氯                          | 所有水平上均干扰                                                                                                                                                                                                                           |
| 氯胺，有机氯胺                       | 可能干扰                                                                                                                                                                                                                               |
| 碘                             | 所有水平上均干扰                                                                                                                                                                                                                           |
| 锰，氧化锰(Mn4+, Mn7+)或铬，氧化铬(Cr6+) | <ol style="list-style-type: none"> <li>1. 调节 pH to 6–7.</li> <li>2. 加入3滴碘化钾(30 g/L)到10-mL 样品中.</li> <li>3. 混合并等待1分钟。</li> <li>4. 加入3滴亚砷酸钠(5 g/L)并混合。</li> <li>5. 按程序所示分析处理过的样品。</li> <li>6. 从原始分析结果中减去该测试的结果，得到正确的氯的浓度。</li> </ol> |

|                          |                                                                                                                                                                               |               |         |         |         |
|--------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|---------|---------|---------|
| 一氯胺 (NH <sub>2</sub> Cl) | 对于常规的自由氯消毒（“断裂点”之上），典型的一氯胺的浓度很低。如果样品中存在一氯胺，它对自由氯测试的干扰取决于样品的温度、一氯胺和自由氯的相对浓度以及分析所需要的时间。典型的NH <sub>2</sub> Cl对HR Free Cl <sub>2</sub> 测试的干扰水平(1分钟测试时间，干扰的mg/L Cl <sub>2</sub> ): |               |         |         |         |
|                          |                                                                                                                                                                               | 样品温度° C (° F) |         |         |         |
|                          | NH <sub>2</sub> Cl水平                                                                                                                                                          | 5 (41)        | 10 (50) | 20 (68) | 30 (86) |
|                          | 1.2 mg/L                                                                                                                                                                      | +0.15         | 0.19    | 0.30    | 0.29    |
|                          | 2.5 mg/L                                                                                                                                                                      | +0.35         | 0.38    | 0.55    | 0.61    |
|                          | 3.5 mg/L                                                                                                                                                                      | +0.38         | 0.56    | 0.69    | 0.73    |
|                          | 5.0 mg/L                                                                                                                                                                      | +0.68         | 0.75    | 0.93    | 1.05    |
| 臭氧                       | 所有水平上均干扰                                                                                                                                                                      |               |         |         |         |
| 过氧化物                     | 可能干扰                                                                                                                                                                          |               |         |         |         |
| 极端样品pH值或高缓冲样品            | 用酸或碱调节pH6-7                                                                                                                                                                   |               |         |         |         |

#### 所需试剂

| 试剂种类             | 所需数量<br>每次测试 | 单位      | 货号       |
|------------------|--------------|---------|----------|
| DPD 自由氯粉末试剂..... | 1 包.....     | 100/pkg | 14070-99 |
| 去离子水 .....       | 30 mL.....   | 4 L     | 272-56   |

#### 所需仪器

|                                  |         |       |          |
|----------------------------------|---------|-------|----------|
| 样品比色瓶, 10-20-25 mL, w/ caps..... | 2 ..... | 6/pkg | 24019-06 |
|----------------------------------|---------|-------|----------|

#### OPTIONAL REAGENTS

|                                                                        |                  |          |
|------------------------------------------------------------------------|------------------|----------|
| Chlorine Standard Solution, PourRite ampule,<br>50-75 mg/L, 2 mL ..... | 20/pkg           | 14268-20 |
| Potassium Iodide Solution, 30 g/L .....                                | 100 mL* MDB      | 343-32   |
| Sodium Arsenite, 5 g/L .....                                           | 100 mL* MDB      | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N .....                      | 100 mL* MDB      | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N.....                          | 100 mL* MDB..... | 1270-32  |

#### OPTIONAL APPARATUS

|                                                                     |             |          |
|---------------------------------------------------------------------|-------------|----------|
| Cylinder, graduated, mixing, 25 mL .....                            | each        | 20886-40 |
| pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> 1, portable ..... | each        | 51700-00 |
| pH Paper, 1 to 11 pH units .....                                    | 5 rolls/pkg | 391-33   |
| Pipet, TenSette, 0.1 to 1.0 mL .....                                | each        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....                       | 50/pkg      | 21856-96 |
| PourRite Ampule Breaker .....                                       | each        | 24846-00 |

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\* Marked Dropper Bottle (contact Hach for larger sizes)

## 总氯 高量程 (0 to 5.00 mg/L)

方法号: 10070

### DPD 方法



1. 输入检测高量程总氯的程序编号。

按下: PRGM

屏幕将显示:

PRGM?



2. 按下 8 ENTER  
屏幕将显示:

0.00 mg/L、Cl<sub>2</sub> 和  
ZERO 图标。



3. 用 10ml 去离子水  
注入两支比色瓶中。

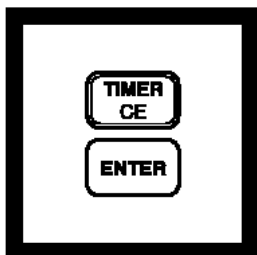
注: 样品一经采集应  
马上进行分析。不能  
保存留待以后使用。



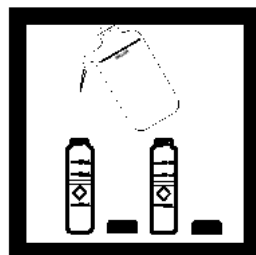
4. 将一包总氯试剂粉  
加入到其中一支比色  
瓶中。(预制试剂) 盖  
上瓶盖, 混动使之溶  
解。

注: 如果存在氯, 则  
溶液会呈现粉红色。

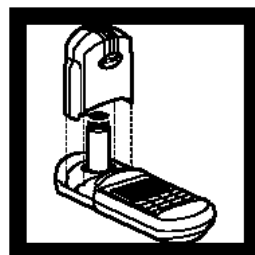
注: 为得到更加精确  
的结果, 应使用去离  
子水进行试剂空白校  
正。



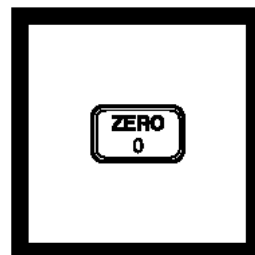
5. 按下: TIMER  
将会开始 2 分钟的反  
应计时。



6. 在计时器鸣响后,  
将去离子水加入到每  
只比色瓶中, 直至达  
到 25 毫升的刻度。盖  
上瓶盖, 反转两次使  
之混合。



7. 将没有加试剂的那  
只比色瓶放入样品适  
配器中, 盖紧遮光盖。



8. 按下: ZERO  
指针将右移, 屏幕将  
会显示:

0.00 mg/L Cl<sub>2</sub>



9. 将预制试样瓶放入样品适配器中，盖上遮光盖。
10. 按下：**READ**  
指针将右移，屏幕将显示总氯的含量，单位是 mg/L。

*注：应使用预制标准溶液进行标准校正。*

### 采样和存放

氯样品一经采集应该立即进行检测。氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地和大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等，都会影响氯在水中的分解情况。

由于会产生化学作用，因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商业用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

不要使用相同的试管装载自由氯和总氯的样本。如果总氯试剂中的少量碘化物渗入自由氯样本中，一氯化物将会产生干扰。最好的方法就是各自使用单独的试管来装载自由氯和总氯的样品。

检测氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

### 精确检测

#### 加标检测法

- 选用LR Chlorine PourRite® Ampule标准溶液。
- 使用TenSette移液管将0.1毫升的该溶液注入10毫升的预制试样（混合样本）中，轻轻晃动使之混合。
- 从第四步开始检测预制样本。
- 计算加入到样本的氯的浓度（毫克/升）：

$$\text{所需加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- e) 预制样本的结果应该等于所分析样本值加上所计算的所加氯的浓度值（步骤d）。
- f) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

## 检测性能

### 检测精度

在一个单独的实验室，如果使用2.40毫克/升的氯溶液和仪器所附带试剂，单个测试人员所得结果的标准误差是±0.06毫克/升。

### 检测极限

程序8的预计检测极限是氯含量为0.03毫克/升。通常在浓度在2毫克/升以下，可使用方法# 8167来检测总氯的浓度。如果了解测试误差和测试极限的更多信息请参考第一章。

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候，根据Federal RCRA for arsenic (D004)条例，样品将会成为危险废物。有关这些废物的处理信息请查看第三章。

## 方法概述

使用DPD方法测试总氯时，根据样品的数量按比例地加入指示剂可以增加检测的范围。因此可以加入更大块的DPD自由氯试剂粉块到10毫升的样本中。在颜色显影之后，用去离子水将样品容量调整到25毫升。这样将允许在1英寸的试管内直接进行颜色检测，而不需要将其移入小的试管里进行。

稀释已反应的样品可以避免测试前稀释样品所带来的潜在的氯损失。由于准确性和精密度方面的损失可以被预测，因此在浓度低于2个毫克 / 升时，推荐使用方法# 8167来检测总氯的浓度。

## 干扰

表1 干扰物质和建议的处理方法

| 干扰物质                                                                 | 干扰水平和处理                                                                                                                                                                                                                            |
|----------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 溴                                                                    | 所有水平上均干扰                                                                                                                                                                                                                           |
| 二氧化氯                                                                 | 所有水平上均干扰                                                                                                                                                                                                                           |
| 氯胺，有机氯胺                                                              | 可能干扰                                                                                                                                                                                                                               |
| 碘                                                                    | 所有水平上均干扰                                                                                                                                                                                                                           |
| 锰，氧化锰(Mn <sup>4+</sup> , Mn <sup>7+</sup> )或铬，氧化铬(Cr <sup>6+</sup> ) | <ol style="list-style-type: none"> <li>1. 调节 pH to 6–7.</li> <li>2. 加入3滴碘化钾(30 g/L)到10-mL 样品中.</li> <li>3. 混合并等待1分钟。</li> <li>4. 加入3滴亚砷酸钠(5 g/L)并混合。</li> <li>5. 按程序所示分析处理过的样品。</li> <li>6. 从原始分析结果中减去该测试的结果，得到正确的氯的浓度。</li> </ol> |
| 臭氧                                                                   | 所有水平上均干扰                                                                                                                                                                                                                           |
| 过氧化物                                                                 | 可能干扰                                                                                                                                                                                                                               |
| 极端样品pH值或高缓冲样品                                                        | 用酸或碱调节pH6-7                                                                                                                                                                                                                        |

## 所需试剂

| 试剂种类            | 所需数量<br>每次测试 | 单位            | 货号       |
|-----------------|--------------|---------------|----------|
| DPD 总氯粉末试剂..... | 1 包.....     | 100/pkg ..... | 14064-99 |
| 去离子水 .....      | 30 mL.....   | 500mL .....   | 272-49   |

## 所需仪器

|                                  |         |             |          |
|----------------------------------|---------|-------------|----------|
| 样品比色瓶, 10-20-25 mL, w/ caps..... | 2 ..... | 6/pkg ..... | 24019-06 |
|----------------------------------|---------|-------------|----------|

## OPTIONAL REAGENTS

| Description                                                            | Unit              | Cat. No. |
|------------------------------------------------------------------------|-------------------|----------|
| Chlorine Standard Solution, PourRite ampule,<br>50-75 mg/L, 2 mL ..... | 20/pkg .....      | 14268-20 |
| Potassium Iodide Solution, 30 g/L .....                                | 100 mL* MDB ..... | 343-32   |
| Sodium Arsenite, 5 g/L .....                                           | 100 mL* MDB ..... | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N .....                      | 100 mL* MDB ..... | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N.....                          | 100 mL* MDB ..... | 1270-32  |

## OPTIONAL APPARATUS

|                                               |                   |          |
|-----------------------------------------------|-------------------|----------|
| Cylinder, graduated, mixing, 25 mL .....      | each .....        | 20886-40 |
| pH Meter, <i>sension™ I</i> , portable .....  | each .....        | 51700-00 |
| pH Paper, 1 to 11 pH units .....              | 5 rolls/pkg ..... | 391-33   |
| Pipet, TenSette, 0.1 to 1.0 mL .....          | each .....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg .....      | 21856-96 |
| PourRite Ampule Breaker .....                 | each .....        | 24846-00 |

## *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 余氯 DPD 方法 (0-2.00 mg/L)

方法号: 8021



1、按“PRGM”键此时萤幕显示 PRGM?

注: 欲得到最正确的结果, 可使用去离子水当作空白溶液。

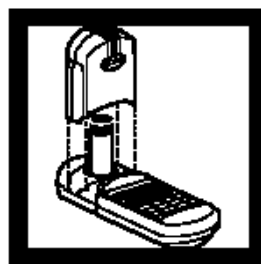


2、输入内程式代号“9”然后按下“ENTER”键, 此时萤幕会出现“mg/1, Cl<sub>2</sub>”及“ZERO icon”

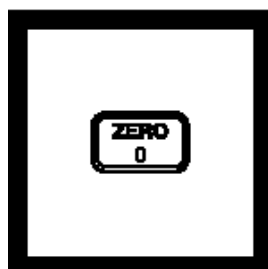


3. 取一支比色瓶加水样至 10ml 标线处, 当作空白溶液。

注: 此空白溶液必须立即分析。



4、将空白溶液瓶放入比色槽中, 并将比色计盖上盖子。



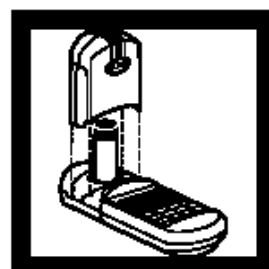
5、按“ZERO”键归零, 萤幕会显示 0.00mg/1 Cl<sub>2</sub>



6、取另一支比色瓶, 加入 10ml 水样。



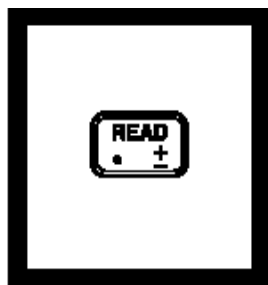
7、加入试剂至比色瓶中, 盖好瓶盖, 摇动使完全溶解, 当作待测溶液。



8、立即放待测溶液至比色槽中, 将比色计盖上盖子。

注: 加药过程以不超过一分钟为限。

注: 假如水样中有余氯存在, 此时会呈粉红色。



9、按 READ 键, 所欲测浓度将会显示出来, 即 mg/1 Cl<sub>2</sub>。

注: 假如加药后, 水样瞬间呈黄色, 或仪器出现“limit”闪烁表示水样中含余氯量过高, 此时可将水样稀释重测, 或使用高浓度的余氯试剂测试。

## 采样和存放

氯样品一经采集应该立即进行检测。自由氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地和大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等，都会影响氯在水中的分解情况。

由于产生化学作用，因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商业用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

不要使用相同的试管装载自由氯和总氯的样本。如果总氯试剂中的少量碘化物渗入自由氯样本中，一氯化物将会产生干扰。最好的方法就是各自使用单独的试管来装载自由氯和总氯的样品。

检测氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

## 精确检测

### 加标检测法（药剂法）

- 量取一定数量的LR Chlorine PourRite® Ampule标准溶液。
- 使用TenSette移液管将0.1毫升的该溶液注入已经充分反应的预制试样（混合样本）中。晃动使之混合。
- 使用原始样本（空白试样）来使仪器设备读数清零。
- 将预制试样放入样品管槽，按READ，记录读数。
- 计算加入样本的氯的浓度（毫克/升）

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- 预制样本的结果（步骤d）应该等于所分析的样本的值加上所计算的溴的浓度值（步骤e）。
- 如果结果不正确，请参考第一部分加标测试的有关详细内容。

### 加标测试（安培瓶法）

- 量取一定的LR Chlorine PourRite® Ampule标准溶液。
- 使用量筒各量取25毫升的样本到两个烧杯中。
- 使用移液管往每个烧杯注入0.2毫升的标准溶液然后旋转使之混合。（这就是预制试样）
- 在每个烧杯中浸入一个DPD 自由氯试剂安培瓶。
- 按照上述步骤分析预制试样和空白试样。
- 计算加入到样本中的氯的当量浓度（毫克/升）

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.2(\text{标准溶液}) \times \text{标称值 (mg/L氯)}}{25.2(\text{样本和标准数})}$$

- g) 预制试样的结果应该等于所要分析样本的值加上氯的浓度值（步骤f）。
- h) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

## 检测方法的效果

### 检测精度

在一个单独的实验室，如果使用1.00毫克/升的氯溶液和仪器所附带的试剂，单个测试人员所得结果的标准误差是±0.01毫克/升。

在一个单独的实验室，如果使用1.00毫克/升的氯溶液和安培瓶法，单个测试人员所得结果的标准误差是±0.01毫克/升。

### 检测极限

程序9和11的预计检测极限是氯含量为0.02毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章。

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候，根据Federal RCRA for arsenic (D004)规定，样品将会成为危险废物。有关这些废物的处理信息请查看第三章。

## 方法概述

以次氯酸或次氯酸盐离子形式存在于样品中的氯会迅速和DPD指示剂反应，生成紫红色，该颜色的深浅和氯的浓度成正比。

## 干扰

| 干扰物质                  | 干扰物允许水平及对策                                                                                                                  |
|-----------------------|-----------------------------------------------------------------------------------------------------------------------------|
| 酸度                    | 如果水样中的碳酸钙的含量大禹 150 毫克/升，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的氢氧化钠将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。              |
| 碱度                    | 如果水样中的碱度超过 250 mg/L CaCO <sub>3</sub> ，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的硫酸将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。 |
| 溴，Br                  | 只要存在就会有干扰                                                                                                                   |
| 二氧化氯，ClO <sub>2</sub> | 只要存在就会有干扰                                                                                                                   |
| 氯胺，有机态                | 可能会产生干扰                                                                                                                     |
| 硬度                    | 在含量不超过 1000 mg/L CaCO <sub>3</sub> 时没有干扰                                                                                    |
| 碘，I <sub>2</sub>      | 只要存在就会有干扰                                                                                                                   |
| 锰，氧化态<br>或            | 1. 将水样的 pH 值调节到 6-7<br>2. 在 25mL 的水样中加入 3 滴 30 g/L 的碘化钾 (Hach                                                               |

|                    |                                                                                                                                                      |
|--------------------|------------------------------------------------------------------------------------------------------------------------------------------------------|
| 六价铬                | #343-32)<br>3. 混合均匀, 反应 1 分钟<br>4. 加入 3 滴 5 g/L 的砷化钠溶液 (Hach #1047-32), 混合均匀<br>5. 取 10mL 处理过的水样, 按照前面的步骤进行分析测量<br>6. 从原来测量的结果中减去此次测量的结果才是水样中总氯的真是含量 |
| 臭氧, O <sub>3</sub> | 只要存在就会产生干扰                                                                                                                                           |
| 过氧化物               | 可能会产生干扰                                                                                                                                              |
| pH 值过高或过低          | 使用 1.00N 的硫酸 (Hach #1270-32) 或者 1.00N 的氢氧化钠 (Hach #1045-32) 将水样的 pH 值调整到 6-7                                                                         |

#### 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                              | 所需数量<br>每次测试 | 单位      | 货号       |
|-----------------------------------|--------------|---------|----------|
| DPD 自由氯粉末试剂, 10 mL.....           | 1 包          | 100/pkg | 21055-69 |
| 样品比色瓶, 10, 20, 25 mL, w/ cap..... | 2            | 6/pkg   | 24019-06 |

#### 所需试剂和仪器 (使用安瓿瓶)

| 试剂种类                        | 所需数量<br>每次测试 | 单位     | 货号       |
|-----------------------------|--------------|--------|----------|
| DPD 自由氯试剂 AccuVac 安瓿瓶 ..... | 1 瓶          | 25/pkg | 25020-25 |
| 烧瓶, 50 mL .....             | 1            | each   | 500-41   |

#### OPTIONAL REAGENTS

| Description                                                         | Unit        | Cat. No. |
|---------------------------------------------------------------------|-------------|----------|
| Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL ..... | 20/pkg      | 26300-20 |
| DPD Free Chlorine Reagent w/dispensing cap.....                     | 250 tests   | 21055-29 |
| Potassium Iodide Solution, 30 g/L .....                             | 100 mL* MDB | 343-32   |
| Sodium Arsenite, 5 g/L .....                                        | 100 mL* MDB | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N .....                   | 100 mL* MDB | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N .....                      | 100 mL* MDB | 1270-32  |
| Water, deionized.....                                               | 4 L         | 272-56   |

#### OPTIONAL APPARATUS

|                                               |             |          |
|-----------------------------------------------|-------------|----------|
| AccuVac Snapper Kit.....                      | each        | 24052-00 |
| Cylinder, graduated, 25 mL .....              | each        | 508-40   |
| pH Meter, <i>sensio™</i> 1, portable .....    | each        | 51700-00 |
| pH Paper, 1 to 11 pH units.....               | 5 rolls/pkg | 391-33   |
| Pipet, TenSette, 0.1 to 1.0 mL .....          | each        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg      | 21856-96 |

PourRite Ampule Breaker..... each..... 24846-00

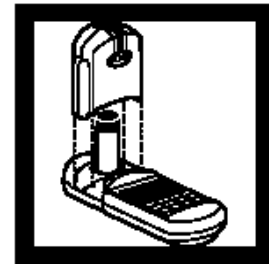
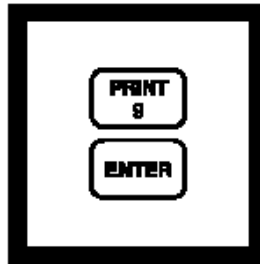
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**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

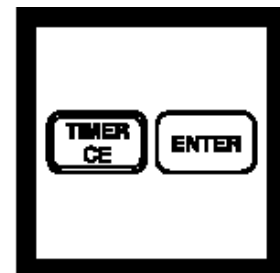
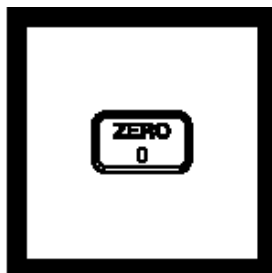
## 总氯 DPD 方法 (0 to 2.00 mg/L)

方法号: 8167



- 1、按“PRGM”键此时萤幕将显示 PRGM?
- 1、输入内程式代号“9”然后按下“ENTER”键，此时萤幕会出现“mg/l, Cl<sub>2</sub>”及“ZERO icon”
- 3、取一支比色瓶加水至 10ml 标线处当空白溶液。
- 4、放空白溶液至比色槽中，并将比色计盖上盖子。

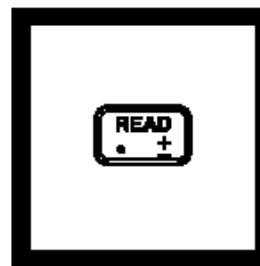
注：此时水样必须立即分析。



- 5、按“ZERO”键归零，萤幕公显示 0.00mg/l Cl<sub>2</sub>
- 6、取另一支比色瓶，加入 10ml 水样
- 7、加入一粒#21056-69 至比色瓶中，盖好瓶盖，将进行 3 分钟反应计时。
- 8、同时按“TIMER”及“ENTER”键，将进行 3 分钟反应计时。

溶液。

注：此处若有试剂残留 注：假如水样中含氯此时会呈粉红色。



- 9、当计时完毕听到哔声，放待测溶液至比色槽中，并将比色计盖上盖子。
- 10、按“READ”键，所欲测浓度将会显示出来，即 mg/l total chlorine。

注：假如加药后，水样瞬间呈黄色，或仪器出现“limit”闪烁表示水样中含总氯量过高，此时可将水样稀释重测，或使用高浓度的总氯试剂测试。

## 采样和存放

氯样品一经采集应该立即进行检测。氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等，都会影响氯在水中的分解情况。

由于会产生化学作用，因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商业用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

不要使用相同的试管装载自由氯和总氯的样本。如果总氯试剂中的少量碘化物渗入自由氯样本中，一氯化物将会产生干扰。最好的方法就是各自使用单独的试管来装载自由氯和总氯的样品。

检测氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

## 精确检测

### 加标检测法（药剂法）

- 量取一定的LR Chlorine PourRite® Ampule标准溶液。
- 使用TenSette移液管将0.1毫升的该溶液注入已经充分反应的预制试样（混合样本）中。晃动使之混合。
- 使用原始样本（空白试样）来使仪器设备读数清零。
- 将预制试样放入样品管槽，按READ，记录读数。
- 计算加入样本的氯的浓度（毫克/升）

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- 预制样本的结果（步骤d）应该等于所分析的样本的值加上所计算的氯的浓度值（步骤e）。
- 如果结果不正确，请参考第一部分加标测试的有关详细内容。

### 加标测试（安培瓶法）

- 量取一定的LR Chlorine PourRite® Ampule标准溶液。
- 使用量筒各量取25毫升的样本到两个烧杯中。
- 使用移液管往每个烧杯注入0.2毫升的标准溶液然后旋转使之混合。（这就是预制试样）
- 在每个烧杯中浸入一个DPD 自由氯试剂安培瓶。
- 按照上述步骤分析预制试样和空白试样。
- 计算加入到样本中的氯的当量浓度（毫克/升）

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.2(\text{标准溶液}) \times \text{标称值 (mg/L氯)}}{25.2(\text{样本和标准数})}$$

- g) 预制试样的结果应该等于所要分析样本的值加上氯的浓度值（步骤f）。
- h) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

## 检测方法的效果

### 检测精度

在一个单独的实验室，如果使用1.00毫克/升的氯溶液和试剂法，单个测试人员所得结果的标准误差是±0.01毫克/升。

在一个单独的实验室，如果使用1.00毫克/升的氯溶液和安培瓶法，单个测试人员所得结果的标准误差是±0.01毫克/升。

### 检测极限

程序9和11的预计检测极限是氯含量为0.02毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章节。

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候，根据Federal RCRA for arsenic (D004)规定，样品将会成为危险废物。有关这些废物的处理信息请查看第三章。

## 干扰

| 干扰物质                  | 干扰物允许水平及对策                                                                                                                  |
|-----------------------|-----------------------------------------------------------------------------------------------------------------------------|
| 酸度                    | 如果水样中的碳酸钙的含量大于 150 毫克/升，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的氢氧化钠将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。              |
| 碱度                    | 如果水样中的碱度超过 250 mg/L CaCO <sub>3</sub> ，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的硫酸将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。 |
| 溴，Br                  | 只要存在就会有干扰                                                                                                                   |
| 二氧化氯，ClO <sub>2</sub> | 只要存在就会有干扰                                                                                                                   |
| 氯胺，有机态                | 可能会产生干扰                                                                                                                     |
| 硬度                    | 在含量不超过 1000 mg/L CaCO <sub>3</sub> 时没有干扰                                                                                    |
| 碘，I <sub>2</sub>      | 只要存在就会有干扰                                                                                                                   |

|                    |                                                                                                                                                                                                                    |
|--------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 锰, 氧化态<br>或<br>六价铬 | 1. 将水样的 pH 值调节到 6-7<br>2. 在 25mL 的水样中加入 3 滴 30 g/L 的碘化钾 (Hach #343-32)<br>3. 混合均匀, 反应 1 分钟<br>4. 加入 3 滴 5 g/L 的砷化钠溶液 (Hach #1047-32), 混合均匀<br>5. 取 10mL 处理过的水样, 按照前面的步骤进行分析测量<br>6. 从原来测量的结果中减去此次测量的结果才是水样中总氯的真是含量 |
| 臭氧, O <sub>3</sub> | 只要存在就会产生干扰                                                                                                                                                                                                         |
| 过氧化物               | 可能会产生干扰                                                                                                                                                                                                            |
| pH 值过高或过低          | 使用 1.00N 的硫酸 (Hach #1270-32) 或者 1.00N 的氢氧化钠 (Hach #1045-32) 将水样的 pH 值调整到 6-7                                                                                                                                       |

#### 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                              | 所需数量<br>每次测试 | 单位      | 货号       |
|-----------------------------------|--------------|---------|----------|
| DPD 总氯粉末试剂, 10 mL.....            | 1 包          | 100/pkg | 21056-69 |
| 样品比色瓶, 10, 20, 25 mL, w/ cap..... | 2            | 6/pkg   | 24019-06 |

#### 所需试剂和仪器 (使用安瓿瓶)

| 试剂种类                       | 所需数量<br>每次测试 | 单位     | 货号       |
|----------------------------|--------------|--------|----------|
| DPD 总氯试剂 AccuVac 安瓿瓶 ..... | 1 瓶          | 25/pkg | 25030-25 |
| 烧瓶, 50 mL .....            | 1            | each   | 500-41   |

#### OPTIONAL REAGENTS

| Description                                                                   | Unit        | Cat. No. |
|-------------------------------------------------------------------------------|-------------|----------|
| Chlorine Standard Solution, PourRite ampule, 25-30 mg/L Cl <sub>2</sub> ..... | 20/pkg      | 26300-20 |
| DPD Total Chlorine Reagent w/dispensing cap .....                             | 250 tests   | 21056-29 |
| Potassium Iodide Solution, 30 g/L.....                                        | 100 mL* MDB | 343-32   |
| Sodium Arsenite, 5 g/L.....                                                   | 100 mL* MDB | 1047-32  |
| Sodium Hydroxide Standard Solution, 1 N .....                                 | 100 mL* MDB | 1045-32  |
| Sulfuric Acid Standard Solution, 1 N .....                                    | 100 mL* MDB | 1270-32  |
| Water, deionized.....                                                         | 4 L         | 272-56   |

#### OPTIONAL APPARATUS

|                                                        |             |          |
|--------------------------------------------------------|-------------|----------|
| AccuVac Snapper Kit.....                               | each        | 24052-00 |
| PourRite Ampule Breaker.....                           | each        | 24846-00 |
| Cylinder, graduated, 25 mL .....                       | each        | 508-40   |
| pH Indicator Paper, 1 to 11 pH units .....             | 5 rolls/pkg | 391-33   |
| pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable..... | each        | 51700-00 |

|                                               |             |          |
|-----------------------------------------------|-------------|----------|
| Pipet, TenSette, 0.1 to 1.0 mL.....           | each.....   | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg..... | 21856-96 |

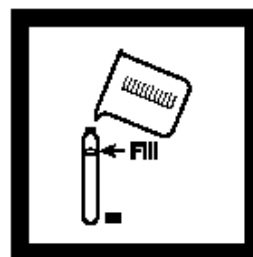
***For Technical Assistance, Price and Ordering***

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## 余氯 DPD Test' N Tube 法 (0 to 5.00 mg/L)

方法号: 10102



1. 输入检测余氯 'N 试管法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为得到更加精确的结果, 应使用去离子水进行试剂空白校正。

2. 按下 10 ENTER 屏幕将显示:

0.00 mg/L、Cl<sub>2</sub> 和 ZERO 图标。

3. 旋转COD/TNT适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。

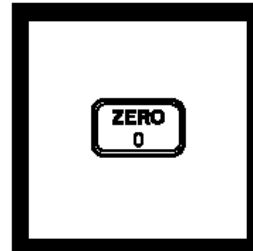
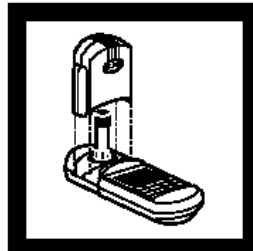
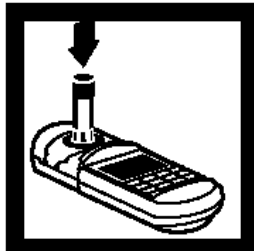
用 10ml 去离子水注入两支比色瓶中。

注: 样品一经采集应马上进行分析。不能保存留待以后使用。

4. 将空白样品注入一支 Test'N Tube™ 试剂瓶中。(空白试样)

注: 应将样品溶液加到顶部的哈西“OVAL”标志。

注: 样品今马上进行测试, 不得留待以后使用。



5. 用湿毛巾将瓶外壁擦干净, 再用干毛巾将瓶外壁的指印及其他印记擦掉。

6. 将装空白试样瓶的试剂瓶放入样品适配器中。

7. 盖上遮光盖。

8. 按下: ZERO 指针将右移, 屏幕将显示:

0.00 mg/L Cl<sub>2</sub>



9. 打开余氯 DPD TNT 试管的瓶盖，然后注入 10 毫升的样品。
10. 盖上瓶盖。反转至少 10 次，使得试剂粉末溶解。这就是预制试样。
11. 用毛巾擦干样品瓶，在混合后 30 秒钟内将其放入样品适配器中。
12. 盖上遮光盖。按下： **READ** 指针将右移，屏幕将显示余氯的含量，单位是 mg/L 。

## 采样和存放

氯样品一经采集应该立即进行检测。自由氯是一种氧化能力很强的氧化剂，在自然的水溶液中是不稳定的。它会迅速地大量无机化合物反应，而且和有机化合物也会慢慢地氧化。许多因素，包括反应物的浓度、日光、PH值、温度、盐度等，都会影响氯在水中的分解情况。

由于产生化学作用，因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液（1毫升的商业用漂泊剂加入1升的去离子水）中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗，那么预处理过程就只需要偶尔进行既可。

检测氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是：如果从水龙头采样的话，应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次，然后再盖上容器，这样的话在样本之上就不会有顶部空间（空气）。如果用样品管采样的话，应该用样品多次清洗该样品管，然后小心地注入10毫升的样本。之后应该马上进行样品分析。

## 精确检测

### 加标检测法

- a) 量取一定的LR Chlorine PourRite® Ampule标准溶液。
- b) 使用TenSette移液管将0.1毫升的该溶液注入已经充分反应的预制试样（混合样本）中。晃动使之混合。
- c) 从步骤8开始检测预制样本。
- d) 计算加入样本的氯的浓度（毫克/升）

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- e) 预制样本的结果（步骤d）应该等于所分析样本的值加上所计算的氯的浓度值（步骤e）。
- f) 如果结果不正确，请参考第一部分加标测试的有关详细内容。

## 检测方法的效果

### 检测精度

在一个单独的实验室，如果使用2.53毫克/升的标准氯溶液和所附带的典型试剂，单个测试人员所得结果的标准误差是±0.14毫克/升。

### 检测极限

程序10的预计检测极限是氯含量为0.03毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候，根据Federal RCRA for arsenic (D004)规定，样品将会成为危险废物。有关这些废物的处理信息请查看第三章  
干扰

| 干扰物质                  | 干扰物允许水平及对策                                                                                                                                                                                                      |
|-----------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 酸度                    | 如果水样中的酸度过高，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的氢氧化钠将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。                                                                                                               |
| 碱度                    | 如果水样中的碱度超过 300 mg/L CaCO <sub>3</sub> ，则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的硫酸将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液，对测量结果也要进行体积校正。                                                                                     |
| 溴，Br                  | 只要存在就会有干扰                                                                                                                                                                                                       |
| 二氧化氯，ClO <sub>2</sub> | 只要存在就会有干扰                                                                                                                                                                                                       |
| 氯胺，有机态                | 可能会产生干扰                                                                                                                                                                                                         |
| 硬度                    | 在含量不超过 1000 mg/L CaCO <sub>3</sub> 时没有干扰                                                                                                                                                                        |
| 碘，I <sub>2</sub>      | 只要存在就会有干扰                                                                                                                                                                                                       |
| 锰，氧化态<br>或<br>六价铬     | 1. 将水样的 pH 值调节到 6-7<br>2. 在 25mL 的水样中加入 3 滴 30 g/L 的碘化钾 (Hach #343-32)<br>3. 混合均匀，反应 1 分钟<br>4. 加入 3 滴 5 g/L 的砷化钠溶液 (Hach #1047-32)，混合均匀<br>5. 取 10mL 处理过的水样，按照前面的步骤进行分析测量<br>6. 从原来测量的结果中减去此次测量的结果才是水样中总氯的真是含量 |
| 臭氧，O <sub>3</sub>     | 只要存在就会产生干扰                                                                                                                                                                                                      |
| 过氧化物                  | 可能会产生干扰                                                                                                                                                                                                         |
| pH 值过高或过低             | 使用 1.00N 的硫酸 (Hach #1270-32) 或者 1.00N 的氢氧化钠 (Hach #1045-32) 将水样的 pH 值调整到 6-7                                                                                                                                    |

## 所需试剂

| 试剂种类                        | 所需数量<br>每次测试 | 单位     | 货号       |
|-----------------------------|--------------|--------|----------|
| Test 'N Tube DPD 自由氯试剂..... | 1 瓶          | 50/pkg | 21055-45 |
| Test 'N Tube 试剂瓶.....       | 1 瓶          | 6/pkg  | 22758-06 |

## 所需仪器

|                   |   |      |          |
|-------------------|---|------|----------|
| COD/TNT 适配器 ..... | 1 | each | 48464-00 |
|-------------------|---|------|----------|

## OPTIONAL REAGENTS

|                                                                     |             |          |
|---------------------------------------------------------------------|-------------|----------|
| Chlorine Standard Solution, PourRite ampule, 50-75 mg/L, 2 mL ..... | 20/pkg      | 14268-20 |
| Potassium Iodide Solution, 30 g/L .....                             | 100 mL* MDB | 343-32   |
| Sodium Arsenite, 5 g/L .....                                        | 100 mL* MDB | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.000 N .....                   | 100 mL* MDB | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N .....                      | 100 mL* MDB | 1270-32  |

## OPTIONAL APPARATUS

|                                                                     |             |          |
|---------------------------------------------------------------------|-------------|----------|
| Beaker, 50 mL.....                                                  | each        | 500-41   |
| pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> 1, portable ..... | each        | 51700-00 |
| pH Paper, pH 1 to 11 pH.....                                        | 5 rolls/pkg | 391-33   |
| Pipet, TenSette, 0.1 to 1.0 mL .....                                | each        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....                       | 50/pkg      | 21856-96 |
| PourRite Ampule Breaker.....                                        | each        | 24846-00 |
| Test Tube Rack.....                                                 | each        | 18641-00 |

# 总氯 DPD Test' N Tube法 (0 to 5.00 mg/L)

方法号: 10101



1. 输入检测余氯 ‘N 试管法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为得到更加精确的结果, 应使用去离子水进行试剂空白校正。

2. 按下 10 ENTER

屏幕将显示:

0.00 mg/L 、Cl<sub>2</sub> 和 ZERO 图标。

3. 旋转COD/TNT适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。

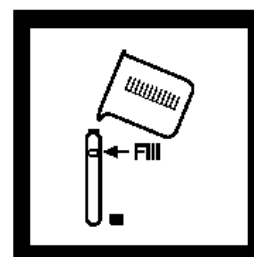
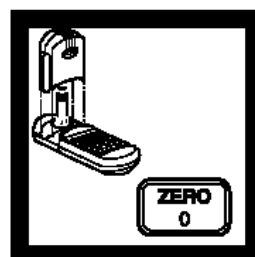
用 10ml 去离子水注入两支比色瓶中。

注: 样品一经采集应马上进行分析。不能保存留待以后使用。

4. 将空白样品注入一支 Test'N Tube™ 试剂瓶中。(空白试样)

注: 应将样品溶液加到顶部的哈西“OVAL”标志。

注: 样品今马上进行测试, 不得留待以后使用。



5. 用湿毛巾将瓶外壁擦干净, 再用干毛巾将瓶外壁的指印及其他印记擦掉。

6. 将装空白试样的试剂瓶放入样品适配器中。

7. 盖上遮光盖。

按下: ZERO

指针将右移, 屏幕将显示:

0.00 mg/L Cl<sub>2</sub>

8. 打开总氯DPD TNT 试管的瓶盖, 然后注入10毫升的样品。

注: 应将样品溶液加到顶部的哈西“OVAL”标志。

注: 如果存在氯, 溶液将呈现粉红色。



9. 盖上瓶盖。反转至少 10 次,使得试剂粉末溶解。这就是预制试样。
10. 按下: **TIMER CE** 将开始 3 分钟的反应计时。
11. 当计时器鸣响后,用毛巾擦干样品瓶,在混合后 30 秒钟内将其放入样品适配器中。
12. 盖上遮光盖。按下: **READ** 指针将右移,屏幕将显示总氯的含量,单位是 mg/L。

### 采样和存放

氯样品一经采集应该立即进行检测。自由氯和化合氯是一种氧化能力很强的氧化剂,在自然的水溶液中是不稳定的。它会迅速地和大量无机化合物反应,而且和有机化合物也会慢慢地氧化。许多因素,包括反应物的浓度、日光、PH值、温度、盐度等,都会影响氯在水中的分解情况。

由于产生化学作用,因此不要使用塑料容器。应该将所用的玻璃容器进行预处理从而除去可能导致氯氧化的因素。该预处理的方法是将玻璃容器浸泡在稀释了的漂泊溶液(1毫升的商业用漂泊剂加入1升的去离子水)中至少一个小时。然后用去离子水或者蒸馏水彻底清洗干净。如果每次使用完后容器都用去离子水或者蒸馏水进行彻底清洗,那么预处理过程就只需要偶尔进行既可。

检测氯的时候常见的错误就是没有采集到具有代表性的样本。正确做法是:如果从水龙头采样的话,应该让水至少流动5分钟从而确保能采到一个有代表性的样本。应该让样本在容量里溢出多次,然后再盖上容器,这样的话在样本之上就不会有顶部空间(空气)。如果用样品管采样的话,应该用样品多次清洗该样品管,然后小心地注入10毫升的样本。之后应该马上进行样品分析。

### 精确检测

#### 加标检测法

- a) 量取一定的LR Chlorine PourRite® Ampule标准溶液。
- b) 使用TenSette移液管将0.1毫升的该溶液注入已经充分反应的预制试样(混合样本)中。晃动使之混合。
- c) 从步骤8开始检测预制样本。
- d) 计算加入样本的氯的浓度(毫克/升)

$$\text{所加入氯的浓度 (mg/L)} = \frac{0.1(\text{标准溶液的量}) \times \text{标称值 (mg/L氯气)}}{10.1(\text{样本和标准液的量})}$$

- e) 预制样本的结果(步骤d)应该等于所分析样本的值加上所计算的氯的浓度值(步骤e)。
- f) 如果结果不正确,请参考第一部分加标测试的有关详细内容。

## 检测方法的效果

### 检测精度

在一个单独的实验室, 如果使用2.53毫克/升的标准氯溶液和试剂法, 单个测试人员所得结果的标准误差是±0.14毫克/升。

### 检测极限

程序10的预计检测极限是氯含量为0.03毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章

## 污染防制和废物管理

当加入亚砷酸钠消除锰或铬引起的干扰时候, 根据Federal RCRA for arsenic (D004)规定, 样品将会成为危险废物。有关这些废物的处理信息请查看第三章。

### 干扰

| 干扰物质                   | 干扰物允许水平及对策                                                                                                                                                                                                         |
|------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 酸度                     | 如果水样中的酸度过高, 则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的氢氧化钠将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液, 对测量结果也要进行体积校正。                                                                                                                |
| 碱度                     | 如果水样中的碱度超过 300 mg/L CaCO <sub>3</sub> , 则会导致发色不完全或者产生的颜色迅速退去。使用 1N 的硫酸将水样的 pH 值调节到 6-7。所有待测的水样应该加入同样体积的氢氧化钠溶液, 对测量结果也要进行体积校正。                                                                                      |
| 溴, Br                  | 只要存在就会有干扰                                                                                                                                                                                                          |
| 二氧化氯, ClO <sub>2</sub> | 只要存在就会有干扰                                                                                                                                                                                                          |
| 氯胺, 有机态                | 可能会产生干扰                                                                                                                                                                                                            |
| 硬度                     | 在含量不超过 1000 mg/L CaCO <sub>3</sub> 时没有干扰                                                                                                                                                                           |
| 碘, I <sub>2</sub>      | 只要存在就会有干扰                                                                                                                                                                                                          |
| 锰, 氧化态<br>或<br>六价铬     | 1. 将水样的 pH 值调节到 6-7<br>2. 在 25mL 的水样中加入 3 滴 30 g/L 的碘化钾 (Hach #343-32)<br>3. 混合均匀, 反应 1 分钟<br>4. 加入 3 滴 5 g/L 的砷化钠溶液 (Hach #1047-32), 混合均匀<br>5. 取 10mL 处理过的水样, 按照前面的步骤进行分析测量<br>6. 从原来测量的结果中减去此次测量的结果才是水样中总氯的真是含量 |
| 臭氧, O <sub>3</sub>     | 只要存在就会产生干扰                                                                                                                                                                                                         |
| 过氧化物                   | 可能会产生干扰                                                                                                                                                                                                            |
| pH 值过高或过低              | 使用 1.00N 的硫酸 (Hach #1270-32) 或者 1.00N 的氢氧化钠 (Hach #1045-32) 将水样的 pH 值调整到 6-7                                                                                                                                       |

## 所需试剂

| 试剂种类                       | 所需数量<br>每次测试 | 单位          | 货号       |
|----------------------------|--------------|-------------|----------|
| Test 'N Tube DPD 总氯试剂..... | 1 瓶 .....    | 50/pkg..... | 21056-45 |
| Test 'N Tube 试剂瓶.....      | 1 瓶 .....    | 6/pkg.....  | 22758-06 |

## 所需仪器

|                   |        |         |          |
|-------------------|--------|---------|----------|
| COD/TNT 适配器 ..... | 1..... | 个 ..... | 48464-00 |
|-------------------|--------|---------|----------|

## OPTIONAL REAGENTS

|                                                                   |                  |          |
|-------------------------------------------------------------------|------------------|----------|
| Chlorine Standard Solution, 2-mL PourRite ampule, 50-75 mg/L..... | 20/pkg.....      | 14268-20 |
| Potassium Iodide Solution, 30 g/L.....                            | 100 mL* MDB..... | 343-32   |
| Sodium Arsenite Solution, 5 g/L .....                             | 100 mL* MDB..... | 1047-32  |
| Sodium Hydroxide Standard Solution, 1.00 N .....                  | 100 mL* MDB..... | 1045-32  |
| Sulfuric Acid Standard Solution, 1.000 N .....                    | 100 mL* MDB..... | 1270-32  |

## OPTIONAL APPARATUS

|                                                                   |                  |          |
|-------------------------------------------------------------------|------------------|----------|
| Beaker, 50 mL.....                                                | each.....        | 500-41   |
| PourRite Ampule Breaker.....                                      | each.....        | 24846-00 |
| pH Indicator Paper, pH 1 to 11 .....                              | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>sens<sup>ion</sup></i> <sup>™</sup> 1, portable..... | each.....        | 51700-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....                               | each.....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....                     | 50/pkg.....      | 21856-96 |
| Test Tube Rack.....                                               | each.....        | 18641-00 |

## 六价铬 (0 to 0.60 mg/L Cr6+)

方法号: 8023

### 1,5-联苯卡巴肼方法



1、按“PRGM”键，萤幕会显示 PRGM?



2、输入内设程代号“13”然后按下“ENTER”键萤幕会出现“mg/L, Cr6”及“ZERO icon”

注：按“CONC”键可转换成测 Cr04, Cr207 之浓度。



3、取一支比色瓶加水样至 10ml 标线处。



4. 加入一 ChromaVer 3 试剂至比色瓶中，盖好瓶盖，摇动数次使充分混合，当待测溶液。

注：假如水样中含有 Cr<sub>6</sub>，此时会呈紫色。



5、同时按“TIMER”及“ENTER”键，将进行 5 分钟的反应计时。



6、取另一支比色瓶加入 10ml 水样，盖好瓶装置，当作空白溶液。

注：假如水样混污，可加入一粒#2126-66 至空白溶液中。



7、当计时完毕，听到哔哔声，立即将空白液瓶放入比色计中测试，并将比色计盖子盖好



8、按“ZERO”键归零萤幕会显示：0.00mg/L Cr6



- 9、放待测溶液瓶至比色计中，并将比色计盖子盖好。
- 10、按“READ”键，所欲测浓度将会显示出来即 mg/L 的  $\text{Cr}^{+6}$

### 采样和存放

采集水样于干净的塑料或玻璃瓶中，可以在4℃ (39 ° F) 或以下保存水样长达24小时。必须在24小时内进行测量。

### 精确检测

#### 加标检测法(药剂法)

- 量取六价铬PourRite® 安培瓶标准溶液，铬离子浓度是5毫克/升。
- 使用TenSette移液管分别各自往3个10毫升的样本中注入0.1毫升、0.2毫升、0.3毫升的标准溶液。晃动使之混合。
- 如上述步骤检测每个样本。铬的浓度应该是每增加0.1毫升的标准溶液相应增加0.05毫克/升。
- 如果结果不正确，请参考第一部分加标测试的有关详细内容。

#### 加标检测法（安培瓶法）

- 量取六价铬Voluette安培瓶标准溶液，铬离子浓度是12.5毫克/升。
- 使用TenSette移液管分别各自往3个装有25毫升的样本的烧杯中注入0.1毫升、0.2毫升、0.3毫升的标准溶液。轻轻晃动使之混合。
- 如上述步骤检测每个样本。铬的浓度应该是每增加0.1毫升的标准溶液相应增加0.05毫克/升。
- 如果结果不正确，请参考第一部分加标测试的有关详细内容。

### 标准溶液法

通过将10毫升的浓度为50毫克/升的六价铬溶液注入容积为1000毫升的烧瓶内，然后用去离子水稀释成浓度为0.5毫克/升的六价铬离子溶液。倒置使之混合。应该当天配置该标准溶液。按照上述步骤，使用该溶液代替样品来检测铬浓度。

### 检测方法的效果

#### 检测精度

在一个单独的实验室，如果使用0.6毫克/升的六价铬溶液和仪器所附带的试剂，单个测试人员所得结果的标准误差是 $\pm 0.008$ 毫克/升。

在一个单独的实验室，如果使用0.6毫克/升的六价铬溶液和安培瓶法，单个测试人员所得结果的标准误差是 $\pm 0.005$ 毫克/升。

### 计检测极限

程序13的预计检测极限是六价铬含量为0.01毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章内容。

### 干扰

|                    |                                          |
|--------------------|------------------------------------------|
| 汞离子                | 会产生轻微干扰                                  |
| 铁                  | 含量超过 1 mg/L 就会产生干扰                       |
| 钒                  | 含量超过 1 mg/L 就会产生干扰；将反应时间延长至 10 分钟可以消除干扰  |
| 高缓冲溶液或极高或极低 PH 值溶液 | 如果水样的 pH 值过高，则可能会超过试剂的缓冲作用，此时需要对水样进行预处理。 |

### 所需试剂和仪器（使用试剂粉包）

| 试剂种类                             | 所需数量<br>每次测试 | 单位      | 货号       |
|----------------------------------|--------------|---------|----------|
| ChromaVer3 铬粉末试剂.....            | 1 包          | 100/pkg | 12710-99 |
| 样品比色瓶, 10-20-25 mL, w/ cap ..... | 2            | 6/pkg   | 24019-06 |

### 所需试剂和仪器（使用AccuVac 安培瓶）

|                               |     |        |          |
|-------------------------------|-----|--------|----------|
| ChromaVer 3 AccuVac 安培瓶 ..... | 1 瓶 | 25/pkg | 25050-25 |
| 烧瓶, 50 mL .....               | 1   | each   | 500-41   |

### OPTIONAL REAGENTS

| Description                                                                                           | Unit    | Cat. No  |
|-------------------------------------------------------------------------------------------------------|---------|----------|
| Acid Reagent Powder Pillows .....                                                                     | 100/pkg | 2126-99  |
| Chromium, Hexavalent, Standard Solution, 50 mg/L Cr <sub>6+</sub> .....                               | 100 mL  | 810-42   |
| Chromium, Hexavalent, Standard Solution,<br>Voluette Ampule, 12.5 mg/L Cr <sub>6+</sub> , 10 mL ..... | 16/pkg  | 14256-10 |
| Chromium, Hexavalent, Standard Solution,<br>PourRite Ampule, 5 mg/L Cr <sub>6+</sub> , 2 mL .....     | 20/pkg  | 26056-20 |
| Water, deionized.....                                                                                 | 4 L     | 272-56   |

### OPTIONAL APPARATUS

| Description                                   | Unit        | Cat. No. |
|-----------------------------------------------|-------------|----------|
| AccuVac Snapper Kit.....                      | each        | 24052-00 |
| Ampule Breaker Kit.....                       | each        | 21968-00 |
| Flask, volumetric, Class A, 1000 mL .....     | each        | 14574-53 |
| pH Paper, 1 to 11 pH units.....               | 5 rolls/pkg | 391-33   |
| pH Meter, EC10, portable .....                | each        | 50050-00 |
| Pipet, TenSette, 0.1 to 1.0 mL .....          | each        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg      | 21856-96 |
| Pipet, volumetric, 5.00 mL, Class A .....     | each        | 14515-37 |
| Pipet Filler, safety bulb .....               | each        | 14651-00 |
| PourRite Ampule Breaker, 2 mL .....           | each        | 24846-00 |

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# 总铬 次溴酸铝法 (0 to 0.60 mg/L)

方法号: 8024



1、按“PRGM”键，此时萤幕将显示 PRGM?



2、输入内设程式代号“15”然后按下“ENTER”键，此时萤幕会出现“mg/L, Cr”及“ZERO icon”



3、取一支比色瓶加水样至 25ml 标线处



4、加入一粒#2043、99 至比色瓶中，盖好瓶盖，摇动数次，使充分混合之后，移去瓶盖当待测溶液



5、将待测溶液放入装沸水的容器中，隔水加热



6、同时按“TIMER”及“ENTER”键，将进行 5 分钟的反应



7、当计时完毕听到哔声，取出待测溶液瓶盖，并将比色瓶拿至水龙头下冲水到 25°C



8、加入一粒#2044-99 至比色瓶中，盖好瓶盖，摇动使充分混合后，移去瓶盖



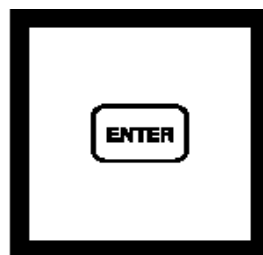
9、加入一酸试剂至比色瓶中，盖好瓶盖，摇动使充分混合后，移去瓶盖



10、加入一ChromaVer 3 试剂至比色瓶中，盖好瓶盖摇动使充分混合后，移去瓶盖。

注：假如水样中含 Cr 此时会呈紫色

注：若有残留未溶解的粉末并不会影响测试结果



11、此时萤幕会显示“05: 00 TIMER 2”，按“ENTER”键，将进行 5 分钟反应计时



12、当计时完毕，听到哔哔声，取另一比色瓶加入 25ml 水样，盖好瓶盖当空白溶液，并放入比色槽中，将比色计盖好盖子。

注：如果水样混污时，如处理待测溶液一样，加入各种试剂至空白溶液中，但是不要加入ChromaVer试剂。



13、按“ZERO”键归零，萤幕会显示 0.00mg/L Cr。



14、放待测溶液至比色槽中，将比色计盖上盖子。



15、按“READ”键所欲测浓度将会显示出来即 mg/L Cr。

注：假如此空白溶液已加入试剂校正此时萤幕会出现“limit”

## 样本采集和存放

采集水样于用酸清洗过的塑料或玻璃瓶中。为保存样本，应该用硝酸将样本PH值调到2或以下（大约每升溶液加2毫升硝酸）。在室温下可以最多存放6个月。在分析检测之前，应用5.0N的氢氧化钠将PH值调到4左右。同时还要对加入的试剂进行结果校正。

## 精确检测

### 加标检测法

- 分别将25毫升的样品溶液注入三只样品试管中。
- 量取三价铬标准安培瓶溶液，其三价铬浓度为12.5毫克/升。
- 使用TenSette移液管分别各自往3只样本试管中注入0.1毫升、0.2毫升、0.3毫升的标准溶液。晃动使之混合。
- 如上述步骤检测每个样本。铬的浓度应该是每增加0.1毫升的标准溶液相应增加0.05毫克/升。
- 如果结果不正确，请参考第一部分加标测试的有关详细内容。

### 标准溶液法

将1毫升的浓度为50毫克/升的三价铬溶液注入容积为100毫升的容器内，然后用去离子水稀释成浓度为0.5毫克/升的三价铬离子溶液。使之彻底混合。按照上述步骤来检测铬浓度。该铬的读数应该是0.5毫克/升。

## 检测性能

### 检测精度

在一个单独的实验室，如果使用0.4毫克/升的三价铬溶液和试剂法，单个测试人员所得结果的标准误差是 $\pm 0.004$ 毫克/升。

### 检测极限

程序15的预计检测极限是铬含量为0.01毫克/升。如果想了解测试误差和测试极限的更多信息请参考第一章内容。

## 干扰

表1 干扰物质和建议的处理方法

| 干扰物质         | 干扰水平和处理                                  |
|--------------|------------------------------------------|
| 高缓冲样品或极端样品pH | 可能超过试剂的缓冲能力，需要样品预处理，参阅第一章。               |
| 有机物质（大量）     | 可能阻止三价铬的完全氧化。如果存在高水平有机物质，参阅第2章部分的样品消化说明。 |

## 所需试剂

|                                                         | 货号       |
|---------------------------------------------------------|----------|
| 总铬试剂一套 (100 Tests) .....                                | 22425-00 |
| 包括: (1) 2126-99, (1) 12066-99, (1) 2043-99, (1) 2044-99 |          |

| 试剂种类                    | 所需数量<br>每次测试 | 单位      | 货号       |
|-------------------------|--------------|---------|----------|
| 酸粉末试剂 .....             | 1 包          | 100/pkg | 2126-99  |
| ChromaVer 3 铬粉末试剂 ..... | 1 包          | 100/pkg | 12066-99 |
| Chromium 1 粉末试剂 .....   | 1 包          | 100/pkg | 2043-99  |
| Chromium 2 粉末试剂 .....   | 1 包          | 100/pkg | 2044-99  |

## 所需仪器

|                                  |   |       |          |
|----------------------------------|---|-------|----------|
| 加热炉, 4" 半径, 120 V .....          | 1 | 个     | 12067-01 |
| 或者                               |   |       |          |
| 加热炉, 4" 半径, 240 V .....          | 1 | 个     | 12067-02 |
| 样品比色瓶, 10-20-25 mL, w/ cap ..... | 2 | 6/pkg | 24019-06 |
| 水盆和支架 .....                      | 1 | 个     | 1955-55  |

## OPTIONAL REAGENTS

|                                                                                                     |           |          |
|-----------------------------------------------------------------------------------------------------|-----------|----------|
| Chromium, trivalent, Standard Solution, 50 mg/L Cr <sup>3+</sup> .....                              | 100 mL    | 14151-42 |
| Chromium, trivalent, Standard Solution, PourRite ampule,<br>12.5 mg/L Cr <sup>3+</sup> , 2 mL ..... | 20/pkg    | 14257-20 |
| Nitric Acid, ACS .....                                                                              | 500 mL    | 152-49   |
| Nitric Acid Solution 1:1 .....                                                                      | 500 mL    | 2540-49  |
| Sodium Hydroxide Standard Solution 5.0 N .....                                                      | 50 mL* DB | 2450-26  |
| Water, deionized .....                                                                              | 4 L       | 272-56   |

## OPTIONAL APPARATUS

|                                                 |             |          |
|-------------------------------------------------|-------------|----------|
| Cylinder, graduated, polypropylene, 25 mL ..... | each        | 1081-40  |
| Finger Cots .....                               | 2/pkg       | 14647-02 |
| pH Paper, 1 to 11 pH units .....                | 5 rolls/pkg | 391-33   |
| pH Meter, EC10, portable .....                  | each        | 50050-00 |
| Pipet, serological, 2 mL .....                  | each        | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL .....            | each        | 19700-01 |
| Pipet Tips for 19700-01 TenSette Pipet .....    | 50/pkg      | 21856-96 |
| Pipet, volumetric, Class A, 1.00 mL .....       | each        | 14515-35 |
| Pipet Filler, safety bulb .....                 | each        | 14651-00 |
| PourRite Ampule Breaker .....                   | each        | 24846-00 |

## *For Technical Assistance, Price and Ordering*

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Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 真色度和表观色度 (0— 500 单位)

方法号: 8025

APHA Pt-Co 标准法



1. 装备好过滤装置 (薄膜滤器、滤器支架、过滤瓶、吸气瓶)
2. 用 50 毫升的去离子水清洗滤器, 然后倒去清洗液。
3. 将另外50ml的去离子水通过滤后注入, 以待步骤4使用。
4. 往一支比色瓶 (空白试样) 中注入 10mL 已过滤的去离子水, 舍弃过量的部分。

注: 检测透明色料不需要滤器, 从第 4 步开始, 跳过第 7 步。

注: 如检测透明色料, 不需过滤直接用去离子水即可。



5. 输入检测色度的程序编号。  
按下: PRGM  
屏幕将显示:  
PRGM?
6. 按下 19 ENTER  
屏幕将显示:  
PtCo 和 ZERO 图标
7. 倒出约 50 mL 样品过滤。
8. 往另一支比色瓶中装入 10mL 过滤过的样品 (预制试样)



9. 将空白试样放入样品适配器中。盖紧遮光盖。

10. 按 **ZERO**, 指针将右移, 屏幕显示: 0 mg/L Pt-Co。

11. 将预制试样放入样品适配器中。盖紧遮光盖。

12. 按 **READ** 指针将右移, 屏幕将显示 Pt-Co 的结果读数。

*注: 要用预制的标准溶液进行标准校正。*

## 样本采集和存放

采集样本到干净的塑料或玻璃瓶中。为得到最准确的结果, 应该样本一经采集就进行分析检测。如果马上不能进行检测, 应该将瓶子装满和盖紧。应该避免激烈的搅动和长时接触空气。通过冷冻在4 ° C (39 ° F)的条件下, 样本可以存放48个小时。在进行检测前, 要将样本温度恢复到室温。

## 精确检测

### 标准溶液法

要检测准确, 首先需要具备500个Platinum-Cobalt单位的色度标准溶液。然后将50毫升的500个Platinum-Cobalt单位的标准溶液注入容积为100毫升烧瓶内, 接着用去离子水稀释成250单位的标准溶液。

## 检测方法的效果

### 检测精度

在一个单独的实验室, 如果使用250个Pt-Co色度单位的标准溶液和典型试剂, 单个测试人员所得结果的标准误差是±10Pt-Co色度单位。如果想了解精度方面更多信息请参考第一章内容。

### 计检测极限

程序19的预计检测极限是色度为25个Pt-Co色度单位。如果想了解测试极限的更多信息请参考第一章内容。

## 所需试剂

| 试剂种类       | 所需数量<br>每次测试 | 单位        | 货号     |
|------------|--------------|-----------|--------|
| 去离子水 ..... | 50 mL.....   | 4 L ..... | 272-56 |

## 所需仪器

|                                 |         |               |          |
|---------------------------------|---------|---------------|----------|
| 抽吸器, 真空器 .....                  | 1 ..... | each .....    | 2131-00  |
| 过滤容器, 47 mm, 刻度300 mL .....     | 1 ..... | each .....    | 13529-00 |
| 过滤薄膜, 47 mm, 0.45 微米 .....      | 1 ..... | 100/pkg ..... | 13530-00 |
| 过滤烧瓶, 500 mL .....              | 1 ..... | each .....    | 546-49   |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 ..... | 6/pkg .....   | 24019-06 |
| 塞子, No. 7, one hole .....       | 1 ..... | 6/pkg .....   | 2119-07  |

## OPTIONAL REAGENTS

|                                                          |           |         |
|----------------------------------------------------------|-----------|---------|
| Color Standard Solution, 500 platinum-cobalt units ..... | 1 L ..... | 1414-53 |
|----------------------------------------------------------|-----------|---------|

## OPTIONAL APPARATUS

|                                          |            |          |
|------------------------------------------|------------|----------|
| Cylinder, graduated, 50-mL, glass .....  | each ..... | 508-41   |
| Flask, volumetric, Class A, 100 mL ..... | each ..... | 14574-42 |
| Pipet, volumetric, Class A, 50 mL .....  | each ..... | 14515-41 |
| Thermometer, -10 to 110 °C.....          | each ..... | 1877-01  |

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# 铜 双金鸡纳酸法 (0 to 5.00 mg/L)

方法号: 8506



1、按“PRGM”键此时萤幕将显示 PRGM?

注:欲得最正确的测试结果,可使用去离子水当空白溶液。

注: 欲测总铜离子含量,水样必须先儿消化处理。

2、输入内设程式代号“20”然后按下“ENTER”键,此时萤幕会出现“mg/L, CU”及“ZERO icon”

3、取一支比色瓶加水样至10ml样线处当空白溶液。

注: 在分析前,须使用 8N, KOH 调整水样,使 PH 值在 4-6 之间, PH 值不可超过 6, 且不可产生铜沉淀。

4、将空白溶液瓶放入样品适配器中,并盖上盖子。



5、按“ZERO”键归零萤幕会显示:  
0.00mg/L Cu

6、取另一支比色瓶加入 10ml 水样。

7、加入一粒CuVer 1 试剂至比色瓶中,盖好瓶盖摇动使充分混合,当待测溶液。

8、同时按“TIMER”及“ENTER”键,将进行 2 分钟,反应计时。

注: 假如水样中含有铜离子,则会呈紫色。

注: 若有试剂残留在比色瓶底部,并不会影响测试结果。



9、当计时完毕，听到 10、按“**READ**”键，  
 哔哔声后的30分钟内， 所欲测浓度将会显示  
 放待测溶液至比色槽 出来，即 mg/L。  
 中，并将比色计盖上盖  
 子。

## 干扰

| 干扰物质                 | 干扰物允许水平及对策                                                                                                                   |
|----------------------|------------------------------------------------------------------------------------------------------------------------------|
| 酸                    | 如果水样的酸性过高(pH<2)，则会产生沉淀。可以滴加 8N 的氢氧化钾溶液，同时摇晃样品瓶以使沉淀物质溶解，而后可以继续操作步骤 3。                                                         |
| 铝，Al <sup>3+</sup>   | 如果有铝的干扰，则操作步骤同上面提到的一样，只不过需要在第 4 步时使用 <b>CuVer 2</b> 试剂 (Hach #21882-99) 代替 <b>CuVer 1</b> 试剂，得到的测量结果是总溶解铜，同时要使用 25mL 的样品池。   |
| 氰化物，CN <sup>-</sup>  | 水样中的氰化物会阻止正常颜色的产生。在加入 <b>CuVer 1</b> 试剂之前，在 10mL 水样中加入 0.2mL 的甲醛 (Hach #2059-32) 溶液，反应时间延长为 4 分钟。所得的测量结果要除以 1.02 以消除体积变化的影响。 |
| 硬度，                  | 如果有硬度的干扰，则操作步骤同上面提到的一样，只不过需要在第 4 步时使用 <b>CuVer 2</b> 试剂 (Hach #21882-99) 代替 <b>CuVer 1</b> 试剂，得到的测量结果是总溶解铜，同时要使用 25mL 的样品池。  |
| 铁离子，Fe <sup>3+</sup> | 如果有铁的干扰，则操作步骤同上面提到的一样，只不过需要在第 4 步时使用 <b>CuVer 2</b> 试剂 (Hach #21882-99) 代替 <b>CuVer 1</b> 试剂，得到的测量结果是总溶解铜，同时要使用 25mL 的样品池。   |
| 银离子，Ag <sup>+</sup>  | 如果水样中一直有浊度，并且逐渐变黑，可以初步判断有银的干扰。在 75mL 水样中加入 10 滴饱和的氯化钾溶液 (Hach #765-42)，使用细的滤纸过滤，而后使用过滤后的水样进行测量。                              |

如果希望能够测量出游离状态的铜离子，而不包括与其它化合物（如EDTA）呈络合状态的铜，则不能使用**CuVer 1**试剂，而要在第4步使用只测量游离状态的铜离子的试剂，在水样中同时加入硫化氢试剂粉枕，而后重新读数。该结果就是包括游离状态的铜和络合状态的铜的总铜含量。与**CuVer 1**不同的是，**CuVer 2**试剂包和安培瓶装的试剂可以直接测量水样中络合状态的铜的含量。

## Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4 to 6 with 8 N potassium hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved copper is to be determined, filter the sample before acid addition using the labware listed under *Optional Apparatus*.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Copper Voluette Ampule Standard, 75 mg/L as Cu.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to the mixing cylinders. Stopper and mix thoroughly.
- d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50-mL beakers to fill the ampules. For analysis with powder pillows, transfer only 10 mL of the solution to 10-mL sample cells.
- e) Analyze each sample as described in the procedure. The copper concentration should increase about 0.3 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 1.00 mg/L copper standard by pipetting 1.00 mL of Copper Standard Solution, 100 mg/L as Cu, into 100-mL volumetric flask. Dilute to volume with deionized water and mix well. Prepare this solution daily. Using this solution as the sample, perform the copper procedure as described above.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 2.25 mg/L Cu and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Cu. In a single laboratory, using a standard solution of 2.25 mg/L Cu and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Cu.

## 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                           | 所需数量<br>每次测试 | 单位           | 货号       |
|--------------------------------|--------------|--------------|----------|
| CuVer 1 铜粉末试剂 .....            | 1 包 .....    | 100/pkg..... | 21058-69 |
| 样品比色瓶, 10-20-25 mL, w/cap..... | 2.....       | 6/pkg.....   | 24019-06 |

## 所需试剂和仪器 (使用安瓿瓶)

|                              |           |             |          |
|------------------------------|-----------|-------------|----------|
| CuVer 2 铜试剂 AccuVac安瓿瓶 ..... | 1 瓶 ..... | 25/pkg..... | 25040-25 |
| 烧瓶, 50 mL .....              | 1.....    | each.....   | 500-41   |

## OPTIONAL REAGENTS

|                                                                    |                   |          |
|--------------------------------------------------------------------|-------------------|----------|
| Copper Standard Solution, 100 mg/L.....                            | 100 mL .....      | 128-42   |
| Copper Standard Solution, Voluette Ampule, 75 mg/L Cu, 10 mL ..... | 16/pkg .....      | 14247-10 |
| CuVer 2 Reagent Powder Pillows .....                               | 100/pkg .....     | 21882-99 |
| Formaldehyde, 37%, ACS.....                                        | 100 mL* MDB ..... | 2059-32  |
| Free Copper Reagent Powder Pillows.....                            | 100/pkg .....     | 21186-69 |
| Hydrochloric Acid Solution, 6.0 N .....                            | 500 mL .....      | 884-49   |
| Hydrosulfite Reagent Powder Pillows.....                           | 100/pkg .....     | 21188-69 |
| Nitric Acid, ACS .....                                             | 500 mL .....      | 152-49   |
| Nitric Acid Solution, 1:1 .....                                    | 500 mL .....      | 2540-49  |
| Potassium Chloride Solution, saturated .....                       | 50 mL SCDB .....  | 765-26   |
| Potassium Hydroxide Standard Solution, 8.0 N.....                  | 100 mL* MDB ..... | 282-32   |
| Sodium Hydroxide Standard Solution, 5.0 N.....                     | 100 mL* MDB ..... | 2450-32  |
| Water, deionized .....                                             | 4 L .....         | 272-56   |

## OPTIONAL APPARATUS

| Description                                    | Unit              | Cat. No. |
|------------------------------------------------|-------------------|----------|
| AccuVac Snapper Kit .....                      | each .....        | 24052-00 |
| Ampule Breaker Kit .....                       | each .....        | 21968-00 |
| Cylinder, graduated, mixing, 25 mL .....       | each .....        | 20886-40 |
| Cylinder, graduated, polypropylene, 25 mL..... | each .....        | 1081-40  |
| Cylinder, graduated, 100 mL.....               | each .....        | 508-42   |
| Filter Paper, folded, 12.5 cm .....            | 100/pkg .....     | 1894-57  |
| Filter Pump.....                               | each .....        | 2131-00  |
| Flask, volumetric, 100 mL .....                | each .....        | 547-42   |
| Funnel, polypropylene, 65 mm .....             | each .....        | 1083-67  |
| Hot Plate, 4" diameter, 120 V .....            | each .....        | 12067-01 |
| Hot Plate, 4" diameter, 240 V .....            | each .....        | 12067-02 |
| pH Indicator Paper, 1 to 11 pH.....            | 5 rolls/pkg ..... | 391-33   |
| pH Meter, EC10, portable .....                 | each .....        | 50050-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....            | each .....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....  | 50/pkg .....      | 21856-96 |
| Pipet, volumetric, Class A, 1.00 mL .....      | each .....        | 14515-35 |
| Pipet Filler, safety bulb .....                | each .....        | 14651-00 |

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# 铜 卟啉法

(0 to 210.0 µg/L)

方法号: 8143



1. 输入检测铜卟啉法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?



2. 按下 22 ENTER 屏幕将显示:

µg/L, Cu和ZERO 图标。



3. 将 10 毫升的样品分别注入两支比色瓶中。

注: 应用清洁剂清洗玻璃器皿。后用自来水清洗。然后用 1: 1 的硝酸溶液清洗。最后用不含铜的去离子水清洗。



4. 将一包 Copper Masking 试剂粉加入到其中一个比色瓶中 (空白试样)。混合使溶解。

注: 另一个比色管为待测样品。



5. 将一包卟啉 1 试剂粉加入到每个比色管中。混合使溶解。



6. 将一包 卟啉2 试剂粉到每个比色管中。混合使溶解。

注: 黄色会立即变成蓝色。如存在铜, 样品会重新变回黄色。



7. 按下: TIMER ENTER 将开始3分钟反应计时。



8. 当计时器鸣叫时, 将空白试样瓶放入样品适配器中, 盖紧遮光盖。



9. 按下: ZERO  
指针将右移, 屏幕会显示:

**0.0 µg/L Cu**

10. 将预制试剂放入样品适配器中, 盖紧遮光盖。

*注: 如果所分析的样品含有高含量的金属, 在管壁上会出现少量的金属沉淀物或黄色的聚集物。*

*注: 可以用硝酸溶液冲洗。稀释新的样品, 重复测试, 结果应乘以稀释因子。*

11. 按下: READ  
指针将右移, 屏幕将显示铜的含量, 单位是µg/L。

*注: 使用预制的标准溶液进行标准校正。*

## 干扰

当以下物质的含量超过所列范围时会产生干扰。

| 干扰物质     | 干扰水平和处理     |
|----------|-------------|
| 铝        | 60 mg/L     |
| 镉        | 10 mg/L     |
| 钙        | 15000 mg/L  |
| 氯化物      | 90,000 mg/L |
| 铬 (Cr6+) | 110 mg/L    |
| 钴        | 100 mg/L    |
| 氟化物      | 30000 mg/L  |
| 铁 (Fe2+) | 6 mg/L      |
| 铅        | 3mg/L       |
| 镁        | 10000 mg/L  |
| 锰        | 140 mg/L    |
| 汞        | 3 mg/L      |
| 钼        | 11mg/L      |
| 镍        | 60mg/L      |
| 钾        | 60000 mg/L  |
| 钠        | 90000 mg/L  |
| 锌        | 9mg/L       |

螯合剂, 例如EDTA, 会在所有水平都产生干扰, 除非先进行消解处理。

高缓冲样品或PH超高或者超低的样品可能会超过试剂的缓冲能力而需要样品预处理。

## Sampling and Storage

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature. Before testing, adjust the pH of the sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

## Accuracy Check

### Standard Additions Method

- a) Fill six (3 pairs) 25-mL graduated mixing cylinders with 25 mL of sample. Properly mark each pair of cylinders as “sample” and “blank”.
- b) Using a TenSette Pipet, add 0.1 mL of Copper Standard Solution, 10.0 mg/L Cu, to two of the cylinders. Add 0.2 mL of standard to two more of the cylinders. Add 0.3 mL of standard to the other two cylinders, making a total of six samples (2 for each volume of standard).
- c) Analyze the samples as described above. The copper concentration reading should increase by 40 µg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To assure the accuracy of the test, prepare a 100 µg/L copper standard:

- a) Pipet 1.00 mL of Copper Standard Solution, 10.0 mg/L Cu, into a 100-mL volumetric flask.
- b) Dilute to volume with copper-free, reagent-grade water.
- c) Use this standard in place of the sample in the procedure. The reading should be 100 µg/L Cu.
- d) Prepare this solution daily.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 100 µg/L copper and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of 3.4 µg/L copper.

### Estimated Detection Limit

The estimated detection limit for program 22 is 5.4 µg/L Cu. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

The porphyrin method is very sensitive to trace amounts of free copper. Due to the sensitivity of the method, a masking agent is used to prepare a “blank” for each sample. The method is free from most interferences and does not require any sample extraction or preconcentration. Interferences from other metals are eliminated by the copper

masking reagent. The porphyrin indicator forms an intense, yellow-colored complex proportional to any free copper present in the sample. Total copper may be determined if a digestion is performed prior to analysis.

### 所需试剂

铜试剂一套, 10-mL 样品 (100 tests) .....26033-00

包括: (1) 26034-49, (2) 26035-49, (2) 26036-49, (1) 129-02

| 试剂种类                     | 所需数量 |   | 单位      | 货号       |
|--------------------------|------|---|---------|----------|
|                          | 每次测试 |   |         |          |
| Copper Masking粉末试剂 ..... | 1    | 包 | 100/pkg | 26034-49 |
| 卟啉 1 粉末试剂 .....          | 2    | 包 | 100/pkg | 26035-49 |
| 卟啉 2 粉末试剂 .....          | 2    | 包 | 100/pkg | 26036-49 |

### 所需仪器

样品比色瓶1, 10-20-25 mL, w/ caps.....2 .....6/pkg .....24019-06

### OPTIONAL REAGENTS

|                                              |            |         |
|----------------------------------------------|------------|---------|
| Copper Standard Solution, 10 mg/L Cu.....    | 100 mL MDB | 129-32  |
| Hydrochloric Acid Solution, 1:1 (6 N).....   | 500 mL     | 884-49  |
| Nitric Acid, ACS .....                       | 500 mL     | 152-49  |
| Nitric Acid Solution, 1:1 .....              | 500 mL     | 2540-49 |
| Sodium Hydroxide Standard Solution, 5 N..... | 1 L        | 2450-53 |
| Water, deionized .....                       | 4 L        | 272-56  |

### OPTIONAL APPARATUS

|                                                         |             |          |
|---------------------------------------------------------|-------------|----------|
| Beaker, 100 mL .....                                    | each        | 500-42   |
| Cylinder, mixing, graduated, 25 mL .....                | each        | 20886-40 |
| Flask, volumetric, Class A, 100 mL.....                 | each        | 14574-42 |
| Hot Plate, 7 x 7 inches, 120 V.....                     | each        | 23441-00 |
| Hot Plate, 7 x 7 inches, 240 V.....                     | each        | 23441-02 |
| pH Paper, 1 to 11 pH units .....                        | 5 rolls/pkg | 391-33   |
| pH Meter, <i>sensio</i> <sup>TM</sup> 1, portable ..... | each        | 51700-00 |
| Pipet, Mohr, 5 mL .....                                 | each        | 20934-37 |
| Pipet, TenSette, 0.1 to 1.0 mL.....                     | each        | 19700-01 |
| Pipet Tips, for 19700-01.....                           | 50/pkg      | 21856-96 |
| Pipet, volumetric, 1.0 mL, Class A .....                | each        | 14515-35 |
| Pipet Filler, safety bulb.....                          | each        | 14651-00 |
| Watch Glass, Pyrex®, 100 mL.....                        | each        | 578-70   |

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

氰化物 (0 to 0.240 mg/L)

方法号: 8027

吡啶-吡唑啉铜法



1、按“PRGM”键，  
萤幕会显示 PRGM?



2、输入内设程式代号  
“ 23 ” 然 后 按 下  
“ENTER” 键，萤幕会  
出现“mg1,CN”及“ZERO  
icon”



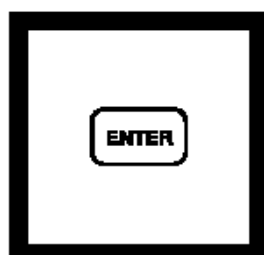
3、取一支比色瓶加水  
样至 10ml 标线处  
注：测试水样温度必  
须保持在 23-25°C



4、加入一CyaniVer  
3 试剂至比色瓶中，  
盖好瓶盖



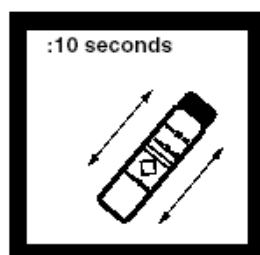
5、同时按“TIMER”  
及“ENTER”键，将  
进行 30 秒反应计时，  
并摇动比色瓶 30 秒。



6、在第一次计时完毕  
听到哔哔声后萤幕将  
显示 0:30 TIMER2, 按  
“ENTER”，将再进行  
30 秒反应计时，此时  
将比色瓶静置 30 秒



7、当计时完毕，听到  
哔哔声，加入一  
CyaniVer4试剂  
至比色瓶中，盖好瓶  
盖。



8、摇动比色瓶 10 秒后  
立即进行步骤 9 之操  
作。

注：此时未溶解的试剂  
并不会影响测试结果。



9、加入一CyaniVer 5 试剂至比色瓶中，盖好瓶盖。



10、快速摇动比色瓶使完全溶解，当待测溶液。



11、此时萤幕将显示 30: 00 TIMER 3，按“ENTER”键，将再进行 30 分钟反应计时。



12、取另一支比色瓶，加入 10ml 水样，盖好瓶盖当空白溶液。

注：假如水样中含氰化物会呈粉红色，数分钟后将转变成蓝色。



13、当计时完毕，听到哔哔声，立即将空白溶液放入比色计中测试，并将比色计盖子盖好。



14、按“ZERO”键归零，萤幕将显示：0.000mg/1 CN



15、放待测溶液至比色计中，并将比色计盖子盖好



16、按“READ”键，所欲测浓度将会显示出来，即 mg/1 CN

## 干扰

| 干扰物质 | 干扰水平和处理方法                                                                                                                                                                                                                                                        |
|------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 氯    | 样品内大量的氯会在加入CyaniVer 5试剂后产生乳白色沉淀。如果已知有氯或其他氧化剂存在,测试前预处理样品,使用本表格中对氧化剂的处理方法。                                                                                                                                                                                         |
| 浑浊   | 浑浊度高会导致读数偏高。在步骤3和12前过滤高浑浊的样品,使用“可选的装置和仪器”所列的仪器。所得的结果就为可溶的氰化物。                                                                                                                                                                                                    |
| 氧化剂  | <p>a)用2.5N的盐酸标准溶液调节 25mL 碱性样品到 pH 7–9, 数出加入多少滴酸。</p> <p>b) 在样品中加入两滴碘化钾溶液和两滴淀粉指示剂溶液。混合。如果存在氧化剂,样品会变蓝。</p> <p>c) 滴加亚砷酸钠溶液直到样品变为无色。每加一滴都充分摇晃溶液。数出滴下多少滴溶液。</p> <p>d) 取另一份25mL样品,并加入与步骤a中所加入相同滴数的盐酸标准溶液。</p> <p>e) 从步骤c中所滴加的亚砷酸钠的总滴数中减去一滴,加入样品中并充分混合。继续氰化物程序的步骤4。</p> |
| 还原剂  | <p>a)用2.5N的盐酸标准溶液调节 25mL 碱性样品到 pH 7–9, 数出加入多少滴酸。</p> <p>b) 在样品中加入四滴碘化钾溶液和四滴淀粉指示剂溶液。混合。样品应无色。</p> <p>c) 滴加溴水溶液直到样品出现蓝色。每加一滴都充分摇晃溶液。数出滴下多少滴溶液。</p> <p>d) 取另一份25mL样品,并加入与步骤a中所加入相同滴数的盐酸标准溶液。</p> <p>e)加入与步骤c中所滴加总滴数一样的亚砷酸钠到样品中并充分混合。</p> <p>f) 继续氰化物程序的步骤4。</p>     |
| 金属   | 不超过1mg/L的镍或钴不干扰。在步骤4 加入CyaniVer 3氰化物试剂粉包前往样品中加入一包HexaVer螯合试剂粉包并在混合除去20 mg/L以内的铜和5 mg/L以内的铁的干扰。在步骤14准备去离子水试剂空白和试剂对仪器调零。                                                                                                                                           |

## Sampling and Storage

Collect samples in glass or plastic bottles and analyze as soon as possible. The presence of oxidizing agents, sulfides and fatty acids can cause cyanide loss during sample storage. Samples containing these substances must be pretreated as described in the following procedures before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

Preserve the sample by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH. Four mL of sodium hydroxide are usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N sodium hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N sodium hydroxide or samples that are highly alkaline due to chlorination treatment processes or distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard Solution. If significant amounts of preservative are used, correct for the volume added; see *Correction for Volume Additions* in Section 1 for more information.

### **Oxidizing Agents**

Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and eliminate their effect, pretreat the sample as follows:

- a) Take a 25-mL portion of the sample and add one drop of m-Nitrophenol Indicator Solution, 10 g/L. Swirl to mix.
- b) Add 2.5 N Hydrochloric Acid Standard Solution drop-wise until the color changes from yellow to colorless. Swirl the sample thoroughly after the addition of each drop.
- c) Add two drops of Potassium Iodide Solution, 30 g/L, and two drops of Starch Indicator Solution, to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.
- d) If Step c suggests the presence of oxidizing agents, add two level 1-g measuring spoonfuls of ascorbic acid per liter of sample.
- e) Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat Steps a to c. If the sample turns blue, repeat Steps d and e.
- f) If the 25-mL sample remains colorless, adjust the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution (usually 4 mL/L).
- g) Perform the procedure given under Interferences, Reducing Agents, to eliminate the effect of excess ascorbic acid, before following the cyanide procedure.

### **Sulfides**

Sulfides quickly convert cyanide to thiocyanate (SCN). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

- a) Place a drop of sample on a disc of hydrogen sulfide test paper that has been wetted with pH 4 Buffer Solution.
- b) If the test paper darkens, add a 1-g measuring spoon of lead acetate to the sample. Repeat Step a.
- c) If the test paper continues to turn dark, keep adding lead acetate until the sample tests negative for sulfide.
- d) Filter the black lead sulfide precipitate using the apparatus listed under Optional Apparatus. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution or neutralize to a pH of 7 for analysis.

### **Fatty Acids**

**Caution**—perform this operation in a hood as quickly as possible.

When distilled, fatty acids will pass over with cyanide and form soaps under the alkaline conditions of the absorber. If the presence of fatty acid is suspected, do not preserve samples with sodium hydroxide until the following pretreatment is performed. The effect of fatty acids can be minimized as follows:

- a) Acidify 500 mL of sample to pH 6 or 7 with Acetic Acid Solution.

- b) Pour the sample into a 1000-mL separatory funnel and add 50 mL of hexane.
- c) Stopper the funnel and shake for one minute. Allow the layers to separate.
- d) Drain off the sample (lower) layer into a 600-mL beaker. If the sample is to be stored, add 5 N Sodium Hydroxide Standard Solution to raise the pH to above 12.

## Accuracy Check

### Standard Solution Method

*Caution—Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.*

Prepare a 100 mg/L cyanide stock solution weekly by dissolving 0.1884 grams of sodium cyanide in deionized water and diluting to 1000 mL. Immediately before use, prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of the 100 mg/L stock solution to 1000 mL using deionized water. Use this prepared standard in place of sample in Step 3. Results should be 0.10 mg/L CN<sup>-</sup>.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 0.19 mg/L CN<sup>-</sup> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L CN<sup>-</sup>.

### Estimated Detection Limit (EDL)

The estimated detection limit for program 23 is 0.008 mg/L CN<sup>-</sup>. For more information on the estimated detection limit, see *Section I*.

## Acid Distillation

For USEPA reporting purposes, samples must be distilled.

All samples should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The “rotten egg” smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L thiocyanate. The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate treatment procedure.

- a) Place a drop of the distillate (already diluted to 250 mL) on a disc of hydrogen sulfide test paper that has been wetted with pH 4.0 Buffer Solution.
- b) If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution drop-wise to the distillate until a neutral pH is obtained.
- c) Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat Step a.
- d) If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide.
- e) Filter the black lead sulfide precipitate through filter paper and funnel. This sample should now be neutralized to pH 7 and analyzed for cyanide without delay.

### Distillation Procedures

A detailed procedure for the distillation of cyanide samples is included with the Hach Distillation Apparatus. Three detailed procedures, Free Cyanides, Cyanides Amenable to Chlorination, and Total Cyanides, are included with the four- and ten-position Midi-Dist Distillation System. See the Optional Apparatus listing.

### Summary of Method

The pyridine-pyrazolone method gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes.

### 所需试剂

|                                              | 货号       |
|----------------------------------------------|----------|
| 氰化物试剂一套 (100 Tests), 10 mL 样品 .....          | 24302-00 |
| 包括: (1) 21068-69, (1) 21069-69, (1) 21070-69 |          |

| 试剂种类                     | 所需数量 |        | 单位            | 货号       |
|--------------------------|------|--------|---------------|----------|
|                          | 每次测试 |        |               |          |
| CyaniVer 3 氰化物粉末试剂 ..... | 1    | 包..... | 100/pkg ..... | 21068-69 |
| CyaniVer 4 氰化物粉末试剂 ..... | 1    | 包..... | 100/pkg ..... | 21069-69 |
| CyaniVer 5 氰化物粉末试剂 ..... | 1    | 包..... | 100/pkg ..... | 21070-69 |

### 所需仪器

|                             |   |       |       |          |
|-----------------------------|---|-------|-------|----------|
| 样品比色瓶, 10-20-25, w/cap..... | 2 | ..... | 6/pkg | 24019-06 |
|-----------------------------|---|-------|-------|----------|

### OPTIONAL REAGENTS

#### Description Unit Cat. No.

|                                                  |            |          |
|--------------------------------------------------|------------|----------|
| Acetic Acid Solution, 10%.....                   | 500 mL     | 14816-49 |
| Ascorbic Acid.....                               | 100 g      | 6138-26  |
| Bromine Water .....                              | 25 mL      | 2211-20  |
| Buffer Solution, pH 4.0 .....                    | 500 mL     | 12223-49 |
| Hexanes, ACS.....                                | 4 L        | 14478-17 |
| HexaVer Chelating Reagent Powder Pillows .....   | 100/pkg    | 243-99   |
| Hydrochloric Acid Standard Solution, 2.5 N ..... | 100 mL MDB | 1418-32  |
| Lead Acetate, trihydrate, ACS .....              | 500 g      | 7071-34  |

|                                                 |                  |          |
|-------------------------------------------------|------------------|----------|
| Magnesium Chloride Solution .....               | 1 L .....        | 14762-53 |
| m-Nitrophenol Indicator.....                    | 100 mL MDB ..... | 2476-32  |
| Potassium Iodide Solution, 30 g/L.....          | 100 mL MDB ..... | 343-32   |
| Sodium Arsenite Solution, APHA .....            | 100 mL MDB ..... | 1047-32  |
| Sodium Cyanide, ACS .....                       | 28 g .....       | 184-20   |
| Sodium Hydroxide Standard Solution, 0.25 N..... | 1 L .....        | 14763-53 |
| Sodium Hydroxide Standard Solution, 5.0 N.....  | 1 L .....        | 2450-53  |
| Starch Indicator Solution.....                  | 10 mL MDB .....  | 349-32   |
| Sulfuric Acid Standard Solution, 19.2 N.....    | 500 mL .....     | 2038-49  |
| Water, deionized .....                          | 4 L .....        | 272-56   |

#### **OPTIONAL APPARATUS**

| <b>Description</b>                                        | <b>Unit</b> | <b>Cat. No.</b> |
|-----------------------------------------------------------|-------------|-----------------|
| Beaker, glass, 600 mL .....                               | each .....  | 500-52          |
| Bottle, wash, 500 mL .....                                | each .....  | 620-11          |
| Cylinder, graduated, 50 mL.....                           | each .....  | 508-41          |
| Cylinder, graduated, 250 mL.....                          | each .....  | 508-46          |
| Distillation Apparatus, cyanide accessories .....         | each .....  | 22658-00        |
| Distillation Apparatus, general purpose accessories ..... | each .....  | 22653-00        |

#### **OPTIONAL APPARATUS, continued**

| <b>Description</b>                                                       | <b>Unit</b>  | <b>Cat. No.</b> |
|--------------------------------------------------------------------------|--------------|-----------------|
| Distillation Apparatus Heater and Support Apparatus, 115 Vac, 60 Hz..... | each.....    | 22744-00        |
| Distillation Apparatus Heater and Support Apparatus, 230 Vac, 50 Hz..... | each.....    | 22744-02        |
| Dropper, plastic.....                                                    | each.....    | 6080-00         |
| Filter Paper, folded, 12.5 cm.....                                       | 100/pkg..... | 1894-57         |
| Flask, volumetric, Class A, 1000 mL .....                                | each.....    | 14574-53        |
| Flask, volumetric, Class A, 250 mL .....                                 | each.....    | 14574-46        |
| Funnel, poly, 65 mm .....                                                | each.....    | 1083-67         |
| Funnel, separatory, 500 mL .....                                         | each.....    | 520-49          |
| Hydrogen Sulfide Test Papers.....                                        | 100/pkg..... | 25377-33        |
| Midi-Dist Distillation System, 4-position.....                           | each.....    | 26384-00        |
| Midi-Dist Distillation System, 10-position.....                          | each.....    | 26385-00        |
| pH Meter, <i>sensio<sup>TM</sup>1</i> , portable.....                    | each.....    | 51700-00        |
| Pipet, volumetric, Class A, 1.00 mL.....                                 | each.....    | 14515-35        |
| Pipet Filler, safety bulb .....                                          | each.....    | 14651-00        |
| Scoop, double ended .....                                                | each.....    | 12257-00        |
| Spoon, measuring, 1.0 g.....                                             | each.....    | 510-00          |
| Support Ring, 4 inch .....                                               | each.....    | 580-01          |
| Support Stand.....                                                       | each.....    | 563-00          |
| Thermometer, -10 to 110 °C.....                                          | each.....    | 1877-01         |

#### ***For Technical Assistance, Price and Ordering***

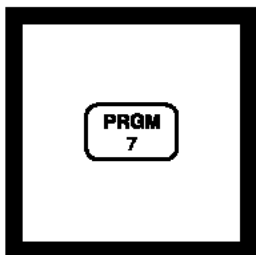
**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 氰尿酸 (cyanuric acid) (7—55 mg/L)

方法号: 8139

### 浊度法

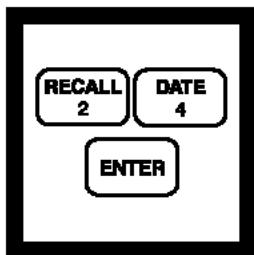


1. 输入检测氰尿酸的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

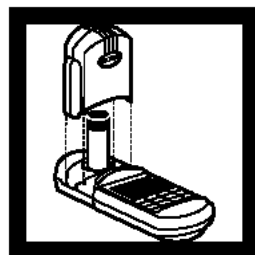


2. 按下 24 ENTER  
屏幕将显示:

mg/L、CYACD 和  
ZERO 图标



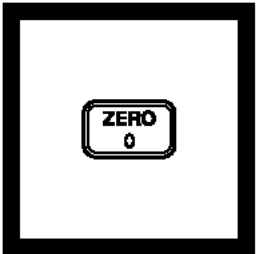
3. 将 25ml 的空白试样注入一支比色瓶中。(空白试样)



4 将空白试样瓶放入样品管槽。盖紧上样品管盖。

注: 为得到最精确的结果, 应用去离子水进行试剂空白校正。

注: 高混浊的样品需要先过滤。

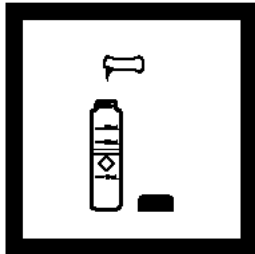


5. 按 ZERO, 指针将右移, 屏幕显示:

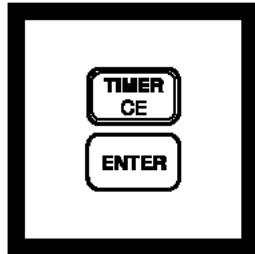
0 mg/L CYACD。



6. 在另外一支比色瓶中加入 25ml 待测样品。



7. 加入氰尿酸 2 试剂, 晃动使之混合。(预制样品)



8. 按 Timer 回车开始三分钟的反应。

注: 如果正在进行样本空白校正, 则屏幕将闪烁显示 “limit” 字样。空白校正的详情见第 1 章。

注: 如果存在氰尿酸, 溶液将生成白色混浊物。

注: 未溶解的粉末不会影响测试的准确性。



9. 将预制试样瓶放入样品适配器中，并盖紧遮光盖。
10. 按 READ  
指针将右移，屏幕会显示氰尿酸浓度，单位是 mg/L。

*注：要用预制的标准溶液进行标准校正。*

*注：每次测试完毕后，要用清洗液、清水、刷子清洗样品管，以免生产白色薄膜。*

## 干扰

混浊会产生干扰，因此分析前应过滤混浊待测样品。

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

## Accuracy Check

### Standard Solution Method

- a) Dissolve 1.000 gram of cyanuric acid in 1000 mL of deionized water to make a 1000 mg/L solution. It takes several hours for the cyanuric acid to dissolve. This solution is stable for several weeks.
- b) Dilute 2.00 mL of the 1000 mg/L solution to 100 mL with deionized water to make a 20 mg/L solution. Prepare fresh daily.
- c) Testing the 20 mg/L solution should give test results of about 20 mg/L cyanuric acid.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 25.0 mg/L cyanuric acid and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 1.2$  mg/L cyanuric acid.

### Estimated Detection Limit

The estimated detection limit for program 24 is 7.0 mg/L cyanuric acid. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The test for cyanuric acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any cyanuric acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present. Due to the nature of the precipitation reaction, low levels of cyanuric acid (less than 7 mg/L) are not detected by this method.

### 所需试剂和仪器

| 试剂种类                             | 所需数量<br>每次测试 | 单位           | 货号       |
|----------------------------------|--------------|--------------|----------|
| 氰尿酸 2粉末试剂 .....                  | 1 包.....     | 50/pkg ..... | 2460-66  |
| 样品比色瓶 , 10-20-25 mL, w/cap ..... | 2 .....      | 6/pkg .....  | 24019-06 |

### OPTIONAL REAGENTS

|                        |            |         |
|------------------------|------------|---------|
| Cyanuric Acid .....    | 25 g ..... | 7129-24 |
| Water, deionized ..... | 4 L .....  | 272-56  |

### OPTIONAL APPARATUS

|                                                          |               |          |
|----------------------------------------------------------|---------------|----------|
| Balance, Analytical, 115 V, Scientech, Model SA 120..... | each .....    | 26103-00 |
| Balance, Analytical, 230 V, Scientech, Model SA 120..... | each .....    | 26103-02 |
| Filter Paper, folded 12.5 cm .....                       | 100/pkg ..... | 1894-57  |
| Flask, volumetric, Class A, 100 mL.....                  | each .....    | 14574-42 |
| Flask, volumetric, Class A, 1000 mL.....                 | each .....    | 14574-53 |
| Funnel, poly, 65 mm.....                                 | each .....    | 1083-67  |
| Pipet, volumetric, Class A, 2.00 mL .....                | each .....    | 14515-36 |

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 二乙基羟胺 (0—500 $\mu$ g/L)

方法号: 8140

### 铁还原法



1. 输入检测二乙基羟胺 (DEHA) 的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 如检测其他去氧剂, 其结果应乘以一个适当的系数。



2. 按下 25 ENTER 屏幕将显示:  
 $\mu$ g/L、DEHA和ZERO图标。

注: 为避免铁质沉淀物的污染, 应用 1: 1 的盐酸溶液清洗样品容器和样品试管。然后再用去离子水多次清洗。

注: 样品必须马上进行检测。



3. 往一支比色瓶中注入 25ml 的样品 (预制试样)。

注: 样品的温度应保持在  $25 \pm 3^\circ \text{C}$ 。

注: 在检测那些在室温下会和氧气迅速反应的化合物时, 在第5—11步应盖住装有样品的容器。



4. 将 25ml 的去离子水注入另外一支比色瓶中。(空白试样)



5. 分别将各一包二乙基羟胺粉末试剂 1 分别注入两支比色瓶中。盖上遮光盖, 搅拌使之混合。



6. 往两支比色瓶中注入刚好二乙基羟胺试剂 2。盖上遮光盖, 搅拌使之混合。然后将它们放在黑暗的地方。

注: 如果存在二乙基羟胺 (DEHA), 则样品将慢慢变成紫色。



7. 迅速按下:  
TIMER 回车  
将会开始十分钟的反应。如果测试对苯二酚, 那么反应只需要2分钟。

注: 在整个测试时间内, 两支试管都存放在黑暗的地方。

注: 温度和反应时间将影响结果。



8. 当定时器一鸣响就将空白试样瓶放入样品适配器中, 并盖紧遮光盖。



9. 按 ZERO, 指针将右移, 屏幕显示:  
0  $\mu\text{g/L}$  DEHA。

10. 迅速将预制试样瓶放入样品适配器中, 并盖紧遮光盖。

11. 按 READ 指针将右移, 屏幕会显示 DEHA 浓度, 单位是  $\mu\text{g/L}$ 。

注: 如果屏幕闪烁显示 “limit”, 这是由于样品中 DEHA 的含量超过测试的最大限度。应使用脱氧的去离子水稀释样品, 然后再重复测试。测试结果要乘以一定的稀释因子。

## 亚铁物质检测校正

注: 在检测样品中亚铁物质时, 重复以上步骤, 但是不要加入二乙基羟 (DEHA) 试剂 2。然后按下 “setup”, 滚动到 “Blank” 位置, 再按下回车键。屏幕将显示 “Blank”, 输入所记录的值。按下回车, 此时该值就成为空白试样值, 每次测试结果将会减去该值。

## 干扰

能使铁还原的物质将产生干扰。同时, 大量含有复合铁的物质也会产生干扰。随着颜色的显现, 光也会产生干扰。当含量超过以下所列的浓度时, 以下物质也会产生干扰。

|                                           |            |     |           |
|-------------------------------------------|------------|-----|-----------|
| 硼酸盐 ( $\text{Na}_2\text{B}_4\text{O}_7$ ) | 500 mg/L   | 钼   | 80 mg/L   |
| 钴                                         | 0.025 mg/L | 镍   | 0.8 mg/L  |
| 铜                                         | 8.0 mg/L   | 磷酸盐 | 10 mg/L   |
| 硬度                                        | 1000 mg/L  | 膦酸盐 | 10 mg/L   |
| 木素磺化盐                                     | 0.05 mg/L  | 硫酸盐 | 1000 mg/L |
| 锰                                         | 0.8 mg/L   | 锌   | 50 mg/L   |

## Sampling and Storage

Most oxygen scavengers will react quickly with atmospheric oxygen. Collect samples in acid-rinsed plastic or glass containers, allowing the sample to overflow. Cap the container so there is no head space above the sample. Rinse each sample cell several times with sample, then carefully fill to the fill mark. Analyze the sample immediately.

## Other Oxygen Scavengers

To determine other oxygen scavengers, perform the test as directed above, then multiply the DEHA result by the appropriate factor below:

| Oxygen Scavenger                       | Factor |
|----------------------------------------|--------|
| Erythorbic Acid<br>(Iso-ascorbic acid) | 3.5    |
| Hydroquinone                           | 2.5    |
| Methylethylketoxime (MEKO)             | 4.1    |
| Carbohydrazide                         | 1.3    |

## Method Performance

### Precision

In a single laboratory, using a standard solution of 242 µg/L DEHA and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±6.2 µg/L DEHA.

### Estimated Detection Limit

The estimated detection limit for program 25 is 9 µg/L DEHA. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Diethylhydroxylamine (DEHA) or other oxygen scavengers present in the sample react with ferric iron in DEHA Reagent 2 Solution to produce ferrous iron in an amount equivalent to the DEHA concentration. This solution then reacts with DEHA 1 Reagent, which forms a purple color with ferrous iron. The color is proportional to the concentration of oxygen scavenger in the sample. Using this procedure other oxygen scavengers can be determined by multiplying the DEHA results by the appropriate multiplier.

### 所需试剂

二乙基羟胺粉末试剂一套(100 tests).....24466-00  
包括: (2) 21679-69, (1) 21680-49

| 试剂种类             | 所需数量<br>每次测试 | 单位           | 货号       |
|------------------|--------------|--------------|----------|
| 二乙基羟胺粉末试剂1 ..... | 2 包 .....    | 100/pkg..... | 21679-69 |
| 二乙基羟胺溶液试剂2 ..... | 1 mL.....    | 500 mL.....  | 21680-49 |
| 去离子水 .....       | 25 mL .....  | 4 L.....     | 272-56   |

### 所需仪器

点滴器, 压榨器, 0.5 and 1.0-mL 刻度 ..... 1..... | 20/pkg..... | 21247-20 || 样品比色瓶, 10-20-25 mL, w/ cap..... | 2..... | 6/pkg..... | 24019-06 |

## OPTIONAL REAGENTS

Hydrochloric Acid, 1:1 (6 N) .....500 mL..... 884-49

## OPTIONAL APPARATUS

Bottle, wash, 250-mL..... each.....620-31  
Cylinder, graduated, polypropylene, 25 mL ..... each.....1081-40  
pH Paper, 1 to 11 pH units..... 5 rolls/pkg.....391-33  
Thermometer, -10 to 110 °C ..... each.....1877-01

### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

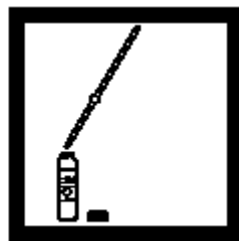
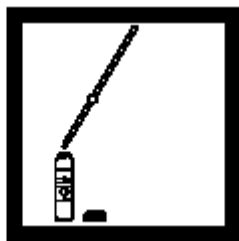
Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 氟化物 (0 to 2.00 mg/L)

方法号: 8029

### SPADNS Method

#### 试剂溶液法



1. 输入检测氟化物 (F) 试剂溶液法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: 27 ENTER  
屏幕将显示:  
0.00 mg/L F 和 ZERO  
图标。

3. 往一个比色瓶中装入 10 mL 样品 (待测样品)。

4. 量取 10ml 的去离子水注入到另外一个比色瓶中。(空白试剂)。

注: 待测样品和空白样品的温度应该基本相同 ( $\pm 1^{\circ}\text{C}$ )。加入试剂的前后可能要要进行温度校正。



5. 往两个比色瓶中分别注入 SPANDS 试剂。晃动使之混合。

6. 按下:  
TIMER ENTER  
将会开始一分钟的反应。

7. 当定时器一鸣响就将空白试样瓶放入样品适配器中, 并盖紧遮光盖。

8. 按 ZERO, 指针将右移, 屏幕显示:  
0  $\mu\text{g/L}$  F。

注: 由于 SPANDS 试剂是有毒和有腐蚀性, 因此检测是应小心注意。应使用移液管进行倾注溶液。

注: SPANDS 试剂应该准确地量取。



9. 迅速将预制试样瓶放入样品适配器中，并盖紧遮光盖。
10. 按 **READ** 指针将右移，屏幕会显示氟化物浓度，单位是 mg/L。

*注：强烈推荐根据每种试剂地特性，使用相应的标准校正。详情见以下精确检测部分。*

## 干扰

该测试对少量干扰比较敏感。玻璃仪器必须很干净。推荐在测试时应该使用相同的玻璃仪器从而确保结果的准确性。

以下物质浓度到达以下所列的水平时会产生干扰。

| 干扰物质                     | 浓度        | 偏差          |
|--------------------------|-----------|-------------|
| 碱度 (CaCO <sub>3</sub> )  | 5000 mg/L | -0.1 mg/L F |
| 铝                        | 0.1 mg/L  | -0.1 mg/L F |
| 氯化物                      | 7000 mg/L | +0.1 mg/L F |
| 铁，三价铁                    | 10 mg/L   | -0.1 mg/L F |
| 磷酸盐，正磷酸盐                 | 16 mg/L   | +0.1 mg/L F |
| Sodium Hexametaphosphate | 1.0 mg/L  | +0.1 mg/L F |
| 硫酸盐                      | 200 mg/L  | +0.1 mg/L F |

SPADNS 试剂含有足够的亚砷酸盐用以清除浓度最高至 5mg/L 氯导致的干扰。如果含有更高浓度的氯导致的干扰，可在每 25 毫升的样品中加入一滴亚砷酸钠溶液。如要检测铝导致的干扰，加入试剂后一分钟读取一次浓度读数，过 15 分钟再读取一次。如果浓度产生明显的增加，则说明铝产生干扰。如果等待 2 小时在读取最终数据，这样可以清除浓度高至 3.0mg/L 的铝产生的干扰。

如果在酸性溶液中蒸馏样品可以清除大多数的干扰，具体如下：

- a) 安装好常用的蒸馏仪器设备。请参考哈西蒸馏仪器手册。使水煮开，使之流过

冷凝器。

- b) 量取 100 毫升的样品注入蒸馏烧瓶中，放入一支有磁性的搅拌棒，打开加热电源。
- c) 小心地量取 150 毫升的 StillVer 蒸馏溶液（2：1 硫酸）注入烧瓶中。如果存在大量的氯化物，应按每毫克/升氯化物的基准加入 5 克硫酸银。
- d) 将加热控制到 10 位置，同时将温度计放在适合位置。在加热时候，黄色的指示灯将亮开。
- e) 当温度达到 180° C（大约 1 小时），停止蒸馏。
- f) 如果有必要，将所收集的蒸馏物稀释到 100 毫升，用上述的方法分析蒸馏物。

## Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

## Accuracy Check

### Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above 1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

### Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the “STD” setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

## Method Performance

### Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.035$  mg/L fluoride. In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS AccuVac Reagent with the instrument, a single operator obtained standard deviations of  $\pm 0.040$  mg/L fluoride.

### Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F-. For more information on derivation and use of Hach’s estimated detection limit, see *Section 1*.

## Summary of Method

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form

a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

### Pollution Prevention and Waste Management

SPADNS Reagent contains sodium arsenite. Final solutions will contain sodium arsenite (D004) in sufficient concentration to be regulated as hazardous waste for Federal RCRA. See *Section 3* for more information on disposal of these materials.

#### 所需试剂(使用溶液)

| 试剂种类              | 所需数量<br>每次测试 | 单位     | 货号     |
|-------------------|--------------|--------|--------|
| SPADNS 试剂 (检测氟化物) | 4 mL         | 500 mL | 444-49 |
| 去离子水              | 10 mL        | 4 L    | 272-56 |

#### 所需仪器(使用溶液)

|                           |   |       |          |
|---------------------------|---|-------|----------|
| 移液管过滤安全球管                 | 1 | 个     | 14651-00 |
| 移液管, Class A, 10.00 mL    | 1 | 个     | 14515-38 |
| 移液管, Class A, 2.00 mL     | 1 | 个     | 14515-36 |
| 样品比色瓶, 10-20-25 mL w/ cap | 2 | 6/pkg | 24019-06 |
| 温度计, -10 to 110°C         | 1 | 个     | 1877-01  |

#### 所需试剂 (使用 ACCUVAC 安瓿瓶)

|                |      |        |          |
|----------------|------|--------|----------|
| SPADNS氟化物试剂安瓿瓶 | 2安瓿瓶 | 25/pkg | 25060-25 |
| 去离子水           | 瓶    | 4 L    | 272-56   |

#### 所需仪器(使用 ACCUVAC 安瓿瓶)

|           |   |   |        |
|-----------|---|---|--------|
| 烧杯, 50 mL | 2 | 个 | 500-41 |
|-----------|---|---|--------|

### OPTIONAL REAGENTS

|                                         |            |         |
|-----------------------------------------|------------|---------|
| Fluoride Standard Solution, 0.2 mg/L F- | 500 mL     | 405-02  |
| Fluoride Standard Solution, 0.4 mg/L F- | 500 mL     | 405-04  |
| Fluoride Standard Solution, 0.5 mg/L F- | 500 mL     | 405-05  |
| Fluoride Standard Solution, 0.6 mg/L F- | 500 mL     | 405-06  |
| Fluoride Standard Solution, 0.8 mg/L F- | 500 mL     | 405-08  |
| Fluoride Standard Solution, 1.0 mg/L F- | 1000 mL    | 291-53  |
| Fluoride Standard Solution, 1.0 mg/L F- | 500 mL     | 291-49  |
| Fluoride Standard Solution, 1.2 mg/L F- | 500 mL     | 405-12  |
| Fluoride Standard Solution, 1.4 mg/L F- | 500 mL     | 405-14  |
| Fluoride Standard Solution, 1.5 mg/L F- | 500 mL     | 405-15  |
| Fluoride Standard Solution, 1.6 mg/L F- | 500 mL     | 405-16  |
| Fluoride Standard Solution, 1.8 mg/L F- | 500 mL     | 405-18  |
| Fluoride Standard Solution, 2.0 mg/L F- | 500 mL     | 405-20  |
| Silver Sulfate, ACS                     | 113 g      | 334-14  |
| Sodium Arsenite Solution                | 100 mL MDB | 1047-32 |
| StillVer Distillation Solution          | 500 mL     | 446-49  |

## OPTIONAL APPARATUS

|                                                                     |             |          |
|---------------------------------------------------------------------|-------------|----------|
| AccuVac Snapper Kit.....                                            | each.....   | 24052-00 |
| Cylinder, graduated, 100 mL .....                                   | each.....   | 508-42   |
| Cylinder, graduated, 250 mL .....                                   | each.....   | 508-46   |
| Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz..... | each.....   | 22744-00 |
| Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz..... | each.....   | 22744-02 |
| Distillation Apparatus General Purpose Accessories .....            | each.....   | 22653-00 |
| pH Meter, <i>sens<sup>ion</sup>™1</i> , portable .....              | each.....   | 51700-00 |
| Pipet, TenSette, 1.0 to 10.0 mL.....                                | each.....   | 19700-10 |
| Pipet Tips, for 19700-10 TenSette Pipet .....                       | 50/pkg..... | 21997-96 |
| Stopper .....                                                       | 6/pkg.....  | 1731-06  |

### ***For Technical Assistance, Price and Ordering***

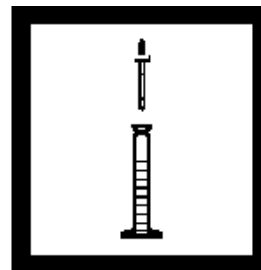
**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

硬度 (0 to 4.00 mg/L Ca and Mg as CaCO<sub>3</sub>)

方法号: 8030

钙和镁; CLG, 比色法



1、按“PRGM”键，萤幕会显示 PRGM?

注:欲得最正确的测试结果,可使用去离子水当作空白溶液。

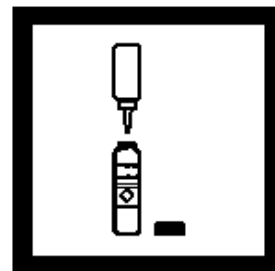
2、输入内设程式代号“30”，然后按下“ENTER”键，萤幕会出现，“mg/1, CaCO<sub>3</sub>”及“ZERO icon”

3、加 100ml 水样至 100ml 量筒中。

注:此时水样温度需维持在 21~29°C。

4、加入 1.0ml 指示剂至样品中，摇动使充分混合。

注: Mg 及 MgCO<sub>3</sub>之间的浓度换算可按“conc”键。



5、再用 1ml 滴管加入 1.0ml 碱液至样品中，摇动使充分混合。

6、各倒入 10ml 加药处理的样品至三个比色瓶中。

7、加入一滴 1M EDTA 至第一个比色瓶中，摇动混合，当作空白溶液

8、加入一滴 EGTA 至第二个比色瓶中，摇动混合，当作待测溶液。



9、放空白溶液至比色槽中，并将比色计盖上盖子。



10、按“ZERO”键，归零萤幕会显示：0.00/1 CaCO<sub>3</sub>。



11、放待测溶液至比色槽中，并将比色计盖上盖子。



12、按“READ”键所欲浓度将显示出来，即 mg/L，magnesium hardness as CaCO<sub>3</sub>



13、不需移去比色瓶，按“PRGM”“29”及“ENTER”键，萤幕会出现“PRGM?”



12、按“ZERO”键归零萤幕会显示 0.00mg/1 CaCO<sub>3</sub>



15、放第三个比色瓶至比色槽中，并将比色计盖上盖子。



16、按“READ”键，所欲测浓度将显示出来，即 mg/1 Calcium hardness as CaCO<sub>3</sub>

注：Ca 及 CaCO<sub>3</sub>之间的浓度换算，可按“CONC”键

注：mg/1 total hardness=mg/1 Ca as CaCO<sub>3</sub>+mg/1 Mg as CaCO<sub>3</sub>

## 干扰

为了得到最准确的钙测试结果，如果钙超过1.0，镁超过0.25 mg/L 的 CaCO<sub>3</sub>，用稀释过的样品重新测试。如果低于这些浓度就没有必要重新测试。

| 干扰物质        | 干扰水平和处理                             |
|-------------|-------------------------------------|
| 铬(3+)       | 大于0.25 mg/L                         |
| 铜(2+)       | 大于0.75 mg/L                         |
| EDTA,螯合的    | 大于0.2 mg/L 的 CaCO <sub>3</sub>      |
| EDTA 或 EGTA | 残留在上次测试的比色管中的痕量物质将使结果出错。使用前彻底清洗比色管。 |
| 铁(2+)       | 大于1.4 mg/L                          |
| 铁(3+)       | 大于2.0 mg/L                          |
| 锰(2+)       | 大于0.20 mg/L                         |
| 锌(2+)       | 大于0.050 mg/L                        |

### **Sampling and Storage**

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months. Adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Correct the test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

### **Accuracy Check**

Using a 2.00 mg/L (as CaCO<sub>3</sub>) standard solution as sample, perform the hardness procedure described above. The results should be 2.00 mg/L calcium (as CaCO<sub>3</sub>).

### **Method Performance**

#### **Precision**

In a single laboratory using a standard solution of 2.00 mg/L Mg as CaCO<sub>3</sub> and 1.88 mg/L Ca as CaCO<sub>3</sub> with the instrument, a single operator obtained a standard deviation of  $\pm 0.09$  mg/L Mg as CaCO<sub>3</sub> and  $\pm 0.08$  mg/L Ca as CaCO<sub>3</sub>.

#### **Estimated Detection Limit**

The estimated detection limit for program 30 is 0.13 mg/L magnesium hardness and 0.08 mg/L calcium hardness. For more information on the estimated detection limit, see *Section 1*.

### **Summary of Method**

The colorimetric method for measuring hardness supplements the conventional titrimetric method because it can measure very low levels of calcium and magnesium. Also some interfering metals (those listed above) in the titrimetric method are inconsequential in the colorimetric method when diluting the sample to bring it within the range of this test. The indicator dye, calmagite, forms a purplish-blue color in a strongly alkaline solution and changes to red when it reacts with free calcium or magnesium. Calcium is chelated with EGTA to destroy any red color due to calcium and then the sample is chelated with EDTA to destroy the red color due to both calcium and magnesium. Measuring the red color in the different stages of chelation gives results as the calcium and magnesium hardness concentrations.

## 所需试剂

|                                                            | 货号       |
|------------------------------------------------------------|----------|
| 硬度试剂一套 (100 Tests) .....                                   | 23199-00 |
| 包括: (1) 22417-32, (1) 22418-32, (1) 22419-26, (1) 22297-26 |          |

| 试剂种类               | 所需数量<br>每次测试 | 单位               | 货号       |
|--------------------|--------------|------------------|----------|
| 检测钙和镁所需的碱性溶液 ..... | 1 mL .....   | 100 mL MDB ..... | 22417-32 |
| 钙和镁指示剂溶液 .....     | 1 mL .....   | 100 mL MDB ..... | 22418-32 |
| EDTA 溶液, 1 M ..... | 1 滴 .....    | 50 mL .....      | 22419-26 |
| EGTA 溶液 .....      | 1 滴 .....    | 50 mL .....      | 22297-26 |

## 所需仪器

|                                 |         |              |          |
|---------------------------------|---------|--------------|----------|
| 混合量筒, 100-mL .....              | 1 ..... | 个 .....      | 1896-42  |
| 点滴器, 0.5 and 1.0 mL .....       | 2 ..... | 20/pkg ..... | 21247-20 |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 3 ..... | 6/pkg .....  | 24019-06 |

## OPTIONAL REAGENTS

|                                                                |                  |          |
|----------------------------------------------------------------|------------------|----------|
| Calcium Standard Solution, 2.0 mg/L as CaCO <sub>3</sub> ..... | 946 mL .....     | 20581-16 |
| Nitric Acid, ACS .....                                         | 500 mL .....     | 152-49   |
| Nitric Acid Solution, 1:1 .....                                | 500 mL .....     | 2540-49  |
| Sodium Hydroxide Standard Solution 5.0 N .....                 | 100 mL MDB ..... | 2450-32  |

## OPTIONAL APPARATUS

|                                                       |            |          |
|-------------------------------------------------------|------------|----------|
| pH Meter, <i>sens<sup>ion</sup>I</i> , portable ..... | each ..... | 51700-00 |
| Thermometer, -10 to 110 °C .....                      | each ..... | 1877-01  |

## For Technical Assistance, Price and Ordering

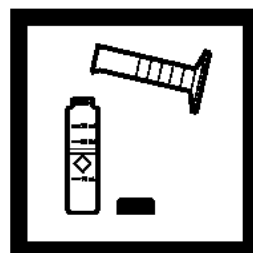
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 联氨 (0 to 500 $\mu\text{g/L}$ )

方法号: 8141

### p-二甲氨基苯甲醛法 试剂溶液法



1. 输入检测联氨 ( $\text{N}_2\text{H}_4$ ) 的试剂溶液法的程序编号。

按下: **PRGM**  
屏幕将显示:

**PRGM?**

2. 按下: 27  
再按: **ENTER**  
屏幕将显示:

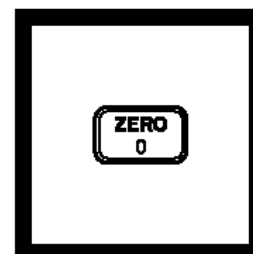
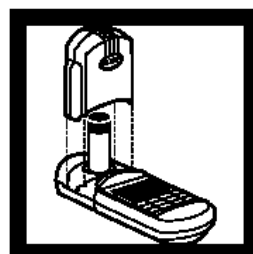
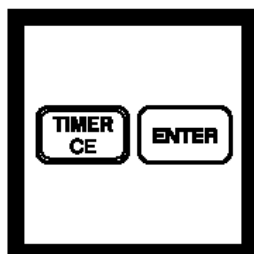
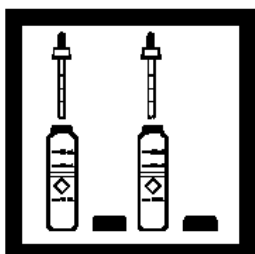
0.00 mg/L、F 和 ZERO  
图标。

3. 往一个比色瓶中装入 10 mL 样品 (待测样品)。

4. 量取 10ml 的去离子水注入到另外一个比色瓶中。(空白试剂)。

*注: 样品应马上进行检测, 不能留待以后使用。*

*注: 待测样品和空白样品的温度应该基本相同 ( $\pm 1^\circ\text{C}$ )。加入试剂的前后可能要温度校正。*



5. 往两个比色瓶中分别注入 0.5ml 的 HydraVer 2 的联氨试剂。盖紧遮光盖, 反转晃动使之混合。

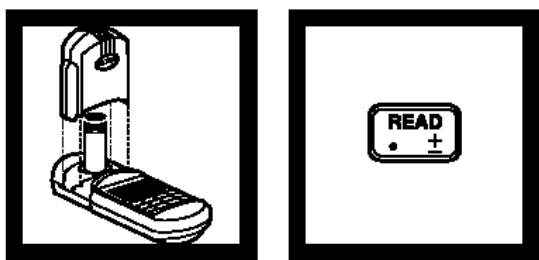
6. 按下: **TIMER** 回车  
将会开始 12 分钟的反应。

7. 当定时器鸣响后, 迅速就将空白试样放入样品适配器中, 并盖紧遮光盖。

8. 按 **ZERO**, 指针将迅速就将空白试样放右移, 屏幕显示:  
**0  $\mu\text{g/L}$   $\text{N}_2\text{H}_4$**

*注: 步骤 7—9 要在 3 分钟内完成。*

*注: 如果存在联氨, 溶液会呈现黄色。如果是空白试剂, 由于 HydraVer 2 的联氨试剂的原因, 溶液将呈淡黄色。*



9. 将预制试样放入样品适配器中，并盖紧遮光盖。
10. 按 READ  
指针将右移，屏幕会显示联氨的浓度，单位是 $\mu\text{g/L}$ 。

### 干扰

当检测深色或浑浊的样品时，将一部分样品中的联氨用1:1的去离子水和普通漂白剂的混合物氧化制成空白溶液。加入2滴该混合液到装有40mL样品含刻度的量筒中，倒转并混合。在步骤3中使用该溶液代替去离子水来作为空白试剂。

在浓度不大于10 mg/L时，氨不会产生影响。当浓度到达20mg/L时，氨的负面干扰就会产生。

浓度在10 mg/L以内，吗啉不干扰。

### Sampling and Storage

Collect samples in glass or plastic containers. Fill the containers completely and cap them tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

### Accuracy Check

#### Standard Solution Method

To assure the accuracy of the test, prepare the following solutions:

- a) Prepare a 25 mg/L hydrazine stock solution by dissolving 0.1016 g of hydrazine sulfate in 1000 mL of oxygen-free deionized water. Use Class A glassware. Prepare this stock solution daily.
- b) Prepare a 100  $\mu\text{g/L}$  hydrazine working solution by diluting 4.00 mL of the 25 mg/L stock solution to 1000 mL with deionized oxygen-free water. Prepare just before analysis.
- c) Use the working solution in place of the sample in Step 4. The result should be 100  $\mu\text{g/L}$  hydrazine.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 250  $\mu\text{g/L}$  hydrazine ( $\text{N}_2\text{H}_4$ ) and two representative lots of reagent with the instrument, a single operator obtained a standard

deviation of  $\pm 9$   $\mu\text{g/L}$  hydrazine.

In a single laboratory using a standard solution of 250  $\mu\text{g/L}$  hydrazine ( $\text{N}_2\text{H}_4$ ) and two lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 3$   $\mu\text{g/L}$  hydrazine.

#### Estimated Detection Limit

The estimated detection limit for program 31 is 16  $\mu\text{g/L}$   $\text{N}_2\text{H}_4$ , and the estimated detection limit for program 32 is 10  $\mu\text{g/L}$   $\text{N}_2\text{H}_4$ . For more information on the estimated detection limit, see *Section 1*.

#### Summary of Method

Hydrazine reacts with the p-dimethylaminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration.

#### 所需试剂 (使用试剂溶液)

| 试剂种类                 | 所需数量<br>每次测试 | 单位                | 货号      |
|----------------------|--------------|-------------------|---------|
| HydraVer 2联氨试剂 ..... | 1 mL.....    | 100 mL* MDB ..... | 1790-32 |
| 去离子水 .....           | 10 mL.....   | 4 L .....         | 272-56  |

#### 所需仪器(使用试剂溶液)

|                                        |         |             |          |
|----------------------------------------|---------|-------------|----------|
| 量筒 25 mL .....                         | 1 ..... | 个.....      | 508-40   |
| 样品比色瓶 10-, 20- and 25 mL, w/ caps..... | 2 ..... | 6/pkg ..... | 24019-06 |

#### 所需试剂 (使用 AccuVac安瓿瓶)

|                       |            |              |          |
|-----------------------|------------|--------------|----------|
| 联氨试剂AccuVac 安瓿瓶 ..... | 2 .....    | 25/pkg ..... | 25240-25 |
| 去离子水 .....            | 10 mL..... | 4 L .....    | 272-56   |

#### 所需仪器 (使用 AccuVac安瓿瓶)

|                |         |        |        |
|----------------|---------|--------|--------|
| 烧杯, 50 L ..... | 2 ..... | 个..... | 500-41 |
|----------------|---------|--------|--------|

#### OPTIONAL REAGENTS

|                              |             |        |
|------------------------------|-------------|--------|
| Hydrazine Sulfate, ACS ..... | 100 g ..... | 742-26 |
|------------------------------|-------------|--------|

#### OPTIONAL APPARATUS

|                                               |               |          |
|-----------------------------------------------|---------------|----------|
| AccuVac Snapper Kit .....                     | each .....    | 24052-00 |
| Balance, AccuLab PocketPro, portable.....     | each .....    | 25568-00 |
| Cylinder, graduated, mixing, 25 mL .....      | each .....    | 1896-40  |
| Flask, volumetric, 100 mL, Class A.....       | each .....    | 14574-42 |
| Flask, volumetric, 1000 mL, Class A.....      | each .....    | 14574-53 |
| Pipet, serological, 1 mL.....                 | each .....    | 532-35   |
| Pipet, TenSette, 0.1 to 1.0 mL .....          | each .....    | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg .....  | 21856-96 |
| Pipet, volumetric, Class A, 1.00 mL .....     | each .....    | 14515-35 |
| Pipet, volumetric, Class A, 4.00 mL .....     | each .....    | 14515-04 |
| Pipet Filler, safety bulb.....                | each .....    | 14651-00 |
| Thermometer, -10 to 110 °C .....              | each .....    | 1877-01  |
| Weighing Boat, 67/46 mm, 8.9 cm sq. ....      | 500/pkg ..... | 21790-00 |

#### For Technical Assistance, Price and Ordering

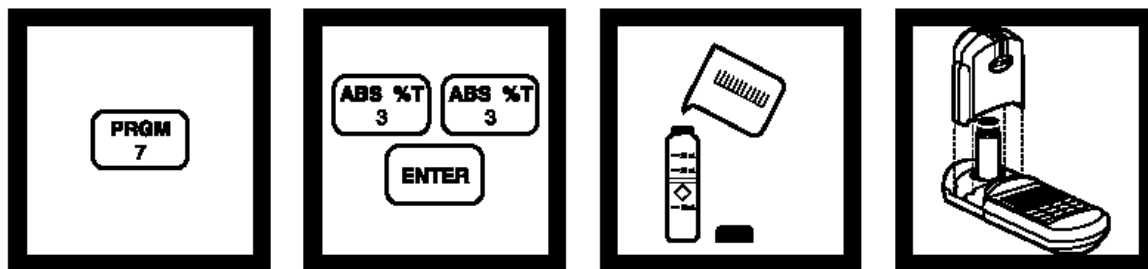
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 亚铁 1,10-菲绕啉法 (0 to 3.00 mg/L)

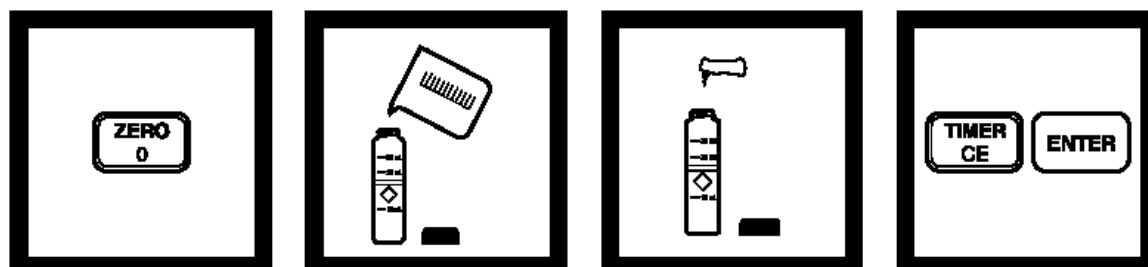
方法号: 8146

使用试剂粉包



1. 输入检测亚铁的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**
2. 按下: **33**  
再按下: **ENTER**  
屏幕将显示:  
**0.00 mg/L、Fe** 和 **ZERO**  
图标。
3. 往一个比色瓶中装入 25 mL 样品 (空白试样)。
4. 将空白试剂放入样品适配器中, 盖紧瓶盖。

注: 在样品分析时候, 应尽快进行, 防止亚铁氧化成铁。



5. 按下 **ZERO**  
指针将右移, 屏幕显示:  
**0 mg/L Fe**
6. 往另外一支比色瓶中注入 25 毫升的样品。
7. 将一包亚铁试剂粉加入到后一支比色瓶中 (预制试样), 盖紧瓶盖, 倒转使之混合。
8. 按下:  
**TIMER ENTER**  
将会开始3分钟的反应。

注: 未溶解的粉末不会影响测试结果。

注: 如果存在亚铁, 溶液会呈现橙色。



9. 将预制试样放入样品适配器中，盖紧瓶盖。



10. 按下：READ  
指针将右移，屏幕将显示亚铁的含量，单位是：mg/L。

*注：应使用预制标准溶液进行标准校正。*

## Sampling and Storage

Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

## Accuracy Check

### Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L  $\text{Fe}_{2+}$ ) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.00 mg/L standard solution. Prepare immediately before use. Run the test using the 1.00 mg/L  $\text{Fe}_{2+}$  Standard Solution by following either the powder pillow or AccuVac procedure. Results should be between 0.90 mg/L and 1.10 mg/L  $\text{Fe}_{2+}$ .

## Method Performance

### Precision

In a single laboratory using an iron standard solution of 2.00 mg/L  $\text{Fe}_{2+}$  and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L  $\text{Fe}_{2+}$ . In a single laboratory using a standard solution of 2.00 mg/L  $\text{Fe}_{2+}$  and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L  $\text{Fe}_{2+}$ .

### Estimated Detection Limit

The estimated detection limit for program 33 (powder pillows and AccuVac Ampuls) is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron ( $\text{Fe}_{3+}$ ) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

### 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                             | 所需数量<br>每次测试 | 单位      | 货号       |
|----------------------------------|--------------|---------|----------|
| 亚铁试剂粉末.....                      | 1 包.....     | 100/pkg | 1037-69  |
| 样品比色瓶, 10-20-25 mL, w/ cap ..... | 2 .....      | 6/pkg   | 24019-06 |

### 所需试剂和仪器(使用 ACCUVAC 安瓿瓶)

|                     |          |        |          |
|---------------------|----------|--------|----------|
| 亚铁 AccuVac 安瓿瓶..... | 1 瓶..... | 25/pkg | 25140-25 |
| 烧杯 , 50 mL .....    | 1 .....  | 个..... | 500-41   |

### OPTIONAL REAGENTS

|                                                 |             |          |
|-------------------------------------------------|-------------|----------|
| Ferrous Ammonium Sulfate, hexahydrate, ACS..... | 113 g ..... | 11256-14 |
| Water, deionized .....                          | 4 L .....   | 272-56   |

### OPTIONAL APPARATUS

|                                              |               |          |
|----------------------------------------------|---------------|----------|
| AccuVac Snapper Kit .....                    | each .....    | 24052-00 |
| Balance, analytical, 115 V.....              | each .....    | 26103-00 |
| Balance, analytical, 230 V.....              | each .....    | 26103-02 |
| Clippers, for opening powder pillows .....   | each .....    | 968-00   |
| Flask, volumetric, 100 mL, Class A.....      | each .....    | 14574-42 |
| Flask, volumetric, 1000 mL, Class A.....     | each .....    | 14574-53 |
| Pipet, volumetric, Class A, 1.00 mL .....    | each .....    | 14515-35 |
| Pipet Filler, safety bulb.....               | each .....    | 14651-00 |
| Weighing Boat, 67/46 mm, 8.9 cm square ..... | 500/pkg ..... | 21790-00 |

### *For Technical Assistance, Price and Ordering*

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 总铁 (0 to 3.00 mg/L)

方法号: 8008



1、按“PRGM”键，萤幕会显示 PRGM?

注：测试总铁需要消解。分析前要先调节 PH 值。



2、输入内充程式代号“33”然后按下“ENTER”键



3、取一支干净的比色瓶加入水样至 10ml 标线处，当作空白溶液。

注：若水样混污此时可加入一匙 0.1g RoVer Rust Remover 摇动使充分混合（若取 25ml 水样则加 0.2g



4、将比色瓶盖上盖子，并放入比色槽中，盖上比色计盖子



5、按“ZERO”键归零，此时萤幕将显示 0.00mg/L Fe



6、取另一比色瓶加入水样至 10ml 标线处。



7、加入一FerroVer Iron试剂至水样中，盖上瓶盖摇动使溶解当作待测溶液。

注：水样中若有试剂未溶解，并不会影响测试结果。



8、按“TIMER/ENTER”键，将进行 3 分钟，的倒数计时。

注：1、水样中含有铁存在，此时会呈现橘色

2、假如水样中含有铁锈，请至少加长反应时间至 5 分钟



- 9、将比色瓶盖上盖子，并放入比色槽中，盖上比色计盖子。
- 10、按“READ”键，所欲测浓度将显示出即 mg/L Fe。

## 干扰

| 干扰物质                    | 干扰水平和处理                                                                                                                                                                                                                                   |
|-------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 钙, $\text{Ca}^{2+}$     | 少于10,000 mg/L没有干扰。                                                                                                                                                                                                                        |
| 氯化物,                    | 少于185,000 mg/L没有干扰。                                                                                                                                                                                                                       |
| 铜                       | 不干扰。FerroVer试剂中含有掩蔽剂。                                                                                                                                                                                                                     |
| 高水平铁                    | 抑制颜色生成。稀释样品重新测试校正结果。                                                                                                                                                                                                                      |
| 氧化铁                     | 需要中、强或Digesdahl消化。消化后，用氢氧化钠调节pH3-5，然后分析。                                                                                                                                                                                                  |
| 镁                       | 少于10,000 mg/L没有干扰。                                                                                                                                                                                                                        |
| 钼酸盐、钼                   | 少于50 mg/L的钼没有干扰。                                                                                                                                                                                                                          |
| 高水平硫化物, $\text{S}^{2-}$ | <ol style="list-style-type: none"> <li>1. 在通风橱等通风良好的地方进行。在250mL锥形瓶中加入5 mL HCl 到100 mL 样品中。煮沸20分钟。</li> <li>2. 冷却，用氢氧化钠调节 pH 到 3 - 5 。用去离子水调节溶液体积为100 mL。</li> <li>3. 分析。</li> </ol>                                                       |
| 浑浊                      | <ol style="list-style-type: none"> <li>1. 加入 一勺0.1 g 量的RoVer Rust Remover 到步骤3的空白试样中，混合。</li> <li>2. 用该空白试样对仪器调零。</li> <li>3. 如样品仍然浑浊，加入3勺 0.2 g量的 RoVer 到75-mL样品中，静置5分钟。</li> <li>4. 用玻璃漏斗过滤或离心。</li> <li>5. 在步骤3和6中使用过滤过的样品。</li> </ol> |
| 极端样品pH/高缓冲样品            | 调节pH到3-5。                                                                                                                                                                                                                                 |

## Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information. If only dissolved iron is to be determined, filter the sample before adding the acid.

## Accuracy Check

### Standard Additions Method

- a) Snap the neck off a 50 mg/L Iron PourRite Ampule Standard Solution.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples and mix thoroughly.
- c) For analysis using AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers to facilitate filling of the ampuls. For analysis with powder pillows, transfer only 10 mL of solution to the 10-mL sample cells.
- d) Analyze each standard addition sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

### Standard Solution Method

Prepare a 1.0-mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, dilute 1.00 mL of an Iron PourRite Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily. Run the test following the procedure for powder pillows or AccuVac Ampuls. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L.

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L Fe.

### Estimated Detection Limit (EDL)

The EDL for program 33 is 0.03 mg/L Fe. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. This reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

## 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                                    | 所需数量<br>每次测试 | 单位           | 货号       |
|-----------------------------------------|--------------|--------------|----------|
| FerroVer 铁粉末试剂 .....                    | 1 包 .....    | 100/pkg..... | 21057-69 |
| 样品比色瓶, 10-20-25 mL, with screw cap..... | 1 .....      | 6/pkg.....   | 24019-06 |

## REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

|                               |             |             |          |
|-------------------------------|-------------|-------------|----------|
| FerroVer 铁试剂 AccuVac安瓿瓶 ..... | 1 安瓿瓶 ..... | 25/pkg..... | 25070-25 |
| 烧杯, 50 mL.....                | 1 .....     | 个 .....     | 500-41   |

## OPTIONAL REAGENTS

| Description                                     | Unit            | Cat. No. |
|-------------------------------------------------|-----------------|----------|
| Ammonium Hydroxide, ACS .....                   | 500 mL.....     | 106-49   |
| Hydrochloric Acid Standard Solution, 6 N.....   | 500 mL.....     | 884-49   |
| Hydrochloric Acid, ACS.....                     | 500 mL.....     | 134-49   |
| Iron Standard Solution, 100 mg/L .....          | 100 mL.....     | 14175-42 |
| Iron PourRite Ampule Standard, 50 mg/L .....    | 20/pkg.....     | 14254-20 |
| Nitric Acid, ACS.....                           | 500 mL.....     | 152-49   |
| Nitric Acid Solution, 1:1.....                  | 500 mL.....     | 2540-49  |
| RoVer Rust Remover .....                        | 454 g .....     | 300-01   |
| Sodium Hydroxide Standard Solution, 5.0 N ..... | 100 mL MDB..... | 2450-32  |
| Water, deionized.....                           | 4 L.....        | 272-56   |

## OPTIONAL APPARATUS

|                                            |              |          |
|--------------------------------------------|--------------|----------|
| AccuVac Snapper Kit.....                   | each.....    | 24052-00 |
| Ampule Breaker, PourRite Ampules .....     | each.....    | 24826-00 |
| Clippers, Shears 7 1/4 " .....             | each.....    | 23694-00 |
| Cylinder, graduated, poly, 25 mL .....     | each.....    | 1081-40  |
| Cylinder, graduated, poly, 100 mL .....    | each.....    | 1081-42  |
| Digesdahl Digestion Apparatus, 115 V ..... | each.....    | 23130-20 |
| Digesdahl Digestion Apparatus, 230 V ..... | each.....    | 23130-21 |
| Filter Discs, glass, 47 mm.....            | 100/pkg..... | 2530-00  |
| Filter Holder, membrane.....               | each.....    | 2340-00  |
| Filter Pump .....                          | each.....    | 2131-00  |
| Flask, Erlenmeyer, 250 mL.....             | each.....    | 505-46   |
| Flask, filtering, 500 mL .....             | each.....    | 546-49   |

## OPTIONAL APPARATUS (continued)

| Description                                | Unit       | Cat. No. |
|--------------------------------------------|------------|----------|
| Flask, volumetric, Class A, 50 mL.....     | each ..... | 14574-41 |
| Flask, volumetric, Class A, 100 mL.....    | each ..... | 14574-42 |
| Hot Plate, 4" diameter, 120 VAC.....       | each ..... | 12067-01 |
| Hot Plate, 4" diameter, 240 VAC.....       | each ..... | 12067-02 |
| pH Meter, <i>sensio™</i> 1, portable ..... | each ..... | 51700-00 |
| pH Indicator Paper, 1 to 11 pH.....        | each ..... | 391-33   |
| Pipet Filler, safety bulb.....             | each ..... | 14651-00 |
| Pipet, serological, 2 mL.....              | each ..... | 532-36   |
| Pipet, serological, 5 mL.....              | each ..... | 532-37   |

|                                               |              |          |
|-----------------------------------------------|--------------|----------|
| Pipet, TenSette, 0.1 to 1.0 mL.....           | each .....   | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg ..... | 21856-96 |
| Pipet, volumetric, Class A, 1.00 mL .....     | each .....   | 14515-35 |
| Spoon, measuring, 0.1 g .....                 | each .....   | 511-00   |

***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 铁 FerroZine 法 (0 to 1.300 mg/L)

方法号: 8147



1. 输入检测铁的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 应在样品分析前调整所存样品的 PH 值。



2. 按下: 37 ENTER  
屏幕将显示:

0.00 mg/L、Fe 和 ZERO 图标。

注: 总铁的检测要预先进行消解处理。



3. 往一个比色瓶中装入 25 mL 样品 (空白试样)。

注: 用 1: 1 的盐酸溶液清洗玻璃仪器, 然后用去离子水清洗。这样做可除去可能引起结果偏差的沉淀物。



4. 将空白试剂瓶放入样品适配器中, 盖紧遮光盖。



5. 按下 ZERO

指针将右移, 屏幕显示:

0 mg/L Fe



6. 往另外一支比色瓶中注入 25 毫升的样品。

注: 如果样品中含有铁锈, 请参考以下干扰部分的内容。



7. 将一包 FerroZine 铁试剂粉加入到后一支比色瓶中 (预制试样), 盖紧瓶盖, 倒转使之混合。



8. 按下:

TIMER 回车  
将会开始 5 分钟的反应。

注: 如果存在铁, 溶液会呈现紫罗兰色。



9. 将预制试样瓶放入样品适配器中，并盖紧遮光盖。

10. 按 READ  
指针将右移，屏幕会显示铁的含量，单位是mg/L。

注：使用预制的标准溶液进行标准校正。

## 干扰

| 干扰物质           | 干扰水平和处理                                                                                                                                                                                                                                                                                                                                                                                                                                     |
|----------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 强的螯合掩蔽剂(EDTA)  | 所有水平上均干扰。对这些样品采用FerroVer 或 TPTZ方法。低含量铁用TPTZ法。                                                                                                                                                                                                                                                                                                                                                                                               |
| 钴              | 可能引起读数稍偏高。                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 铜              | 可能引起读数稍偏高。                                                                                                                                                                                                                                                                                                                                                                                                                                  |
| 氢氧化物           | 在步骤4加入FerroZine铁试剂到样品中，水浴煮沸一分钟。冷却到24 °C (75 °F)后继续步骤5。用去离子水补足样品体积为25mL。或采用第2部分“样品预处理”中的消化方法。                                                                                                                                                                                                                                                                                                                                                |
| 磁铁矿（四氧化三铁）或铁酸盐 | <ol style="list-style-type: none"> <li>1) 往25-mL量筒中装入25 mL样品。</li> <li>2) 将该样品转移到 125-mL的锥形瓶中。</li> <li>3) 加入一包FerroZine铁试剂溶液粉包，混合。</li> <li>4) 将锥形瓶放入加热板上加热至沸。</li> <li>5) 继续煮沸 20到30分钟。</li> </ol> <p>注：不要煮干。</p> <p>注：如存在铁将呈现紫色。</p> <ol style="list-style-type: none"> <li>6) 将已煮沸的样品倒入25-mL量筒中。用少量去离子水冲洗一下锥形瓶，将冲洗液也倒入量筒。</li> <li>7) 用去离子水使样品体积达到25mL刻度线。</li> <li>8) 将溶液倒入比色管，混合。</li> <li>9) 继续执行步骤9或采用第2部分“样品预处理”中的消化方法。</li> </ol> |
| 铁锈             | 在步骤4加入FerroZine铁试剂到样品中，水浴煮沸一分钟。冷却到24 °C (75 °F)后继续步骤5。用去离子水补足样品体积为25mL。或采用第2部分“样品预处理”中的消化方法。                                                                                                                                                                                                                                                                                                                                                |

## Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection and before the addition of nitric acid. Before testing, adjust the sample pH to 3–5 with ammonium hydroxide, ACS. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more detailed information.

## Accuracy Check

### Standard Additions Method

- a) Snap the neck off an Iron Voluette Ampule Standard, 25 mg/L Fe.
- b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10.
- c) Swirl to mix and allow another five-minute reaction period, then measure the iron concentration as in Step 10.
- d) Add two additional 0.1-mL standard increments, taking a concentration reading after allowing the five-minute reaction period for each increment.
- e) Each 0.1 mL of standard added should cause a 0.1 mg/L increase in the concentration reading.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. This solution should be prepared daily. Analyze the working solution according to the above procedure.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 0.80 mg/L iron and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.004$  mg/L iron.

### Estimated Detection Limit

The estimated detection limit for program 37 is 0.011 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The FerroZine Iron Reagent forms a purple colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and can be used to analyze samples containing magnetite (black iron oxide) or ferrites after treatment as described in Interferences.

## 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                        | 所需数量<br>每次测试 | 单位     | 货号       |
|-----------------------------|--------------|--------|----------|
| FerroZine 铁粉末试剂.....        | 1 包.....     | 50/pkg | 2301-66  |
| 剪刀.....                     | 1 .....      | 个      | 968-00   |
| 样品比色瓶, 10-20-25, w/cap..... | 2 .....      | 6/pkg  | 24019-06 |

## OPTIONAL REAGENTS

|                                                                 |         |          |
|-----------------------------------------------------------------|---------|----------|
| Ammonium Hydroxide, ACS.....                                    | 500 mL  | 106-49   |
| Hydrochloric Acid Solution, 1:1 (6N).....                       | 500 mL  | 884-49   |
| FerroZine Iron Reagent Solution.....                            | 1000 mL | 2301-53  |
| Iron Standard Solution, 100 mg/L Fe.....                        | 100 mL  | 14175-42 |
| Iron Standard Solution, Voluette Ampule, 25 mg/L Fe, 10 mL..... | 16/pkg  | 14253-10 |
| Nitric Acid, ACS .....                                          | 500 mL  | 152-49   |
| Nitric Acid Solution, 1:1 .....                                 | 500 mL  | 2540-49  |
| Water, deionized .....                                          | 4 L     | 272-56   |

## OPTIONAL APPARATUS

|                                                |             |          |
|------------------------------------------------|-------------|----------|
| Ampule Breaker Kit .....                       | each        | 21968-00 |
| Clippers, shears, 7¼-inch .....                | each        | 20658-00 |
| Cylinder, graduated, 25 mL.....                | each        | 508-40   |
| Dropper, calibrated, 0.5-mL & 1.0-mL mark..... | 6/pkg       | 23185-06 |
| Flask, erlenmeyer, 125 mL.....                 | each        | 505-43   |
| Flask, volumetric, 250 mL, Class A.....        | each        | 14574-46 |
| Hot plate, 3 ½" diameter, 120 V.....           | each        | 12067-01 |
| Hot plate, 3 ½" diameter, 240 V.....           | each        | 12067-02 |
| pH Indicator Paper, 1 to 11 pH.....            | 5 rolls/pkg | 391-33   |
| Pipet, serological, 2 mL.....                  | each        | 532-36   |
| pH Meter, EC10, portable .....                 | each        | 50050-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....            | each        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....  | 50/pkg      | 21856-96 |
| Pipet, volumetric, 1.00 mL, Class A .....      | each        | 14515-35 |
| Thermometer, -10 to 110 °C.....                | each        | 1877-01  |
| Water Bath, with sample cell rack.....         | each        | 1955-55  |

## *For Technical Assistance, Price and Ordering*

In the U.S.A.:"Call 800-227-4224

Outside the U.S.A.:"Contact the Hach office or distributor serving you.

## 总铁 FerroMo 法 (0 to 1.80 mg/L)

方法号: 8365



1. 输入检测总铁的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到更加精确的结果, 应使用去离子水进行试剂的空白校正。

注: 应在样品分析前调整所存样品的 PH 值。



2. 按下: **38 ENTER**  
屏幕将显示:

0.00 mg/L、Fe 和 ZERO 图标。

注: 总铁的检测要预先进行消解处理。



3. 往一个 50-mL 量筒中注入 50mL 样品。

注: 样品的 PH 值在检测中是十分重要的, 详情见干扰部分的内容。

注: 用 1: 1 的盐酸溶液清洗玻璃仪器, 然后用去离子水清洗。这样做可除去可能引起结果偏差的沉淀物。



4. 将一包 FerroMo 铁试剂 1 粉包加入到量筒中。盖好盖子, 倒转多次使粉剂溶解。此为待测样品。



5. 往一个比色瓶中注入 25 毫升的待测样品。



6. 往比色瓶中加入一包 FerroMo 铁试剂 2 粉包, 盖上瓶盖摇晃 30 秒, 使试剂溶解。此为预制试样。



7. 按下 **Timer** 开始 3 分钟的反应计时。

注: 如果存在铁, 溶液将呈现蓝色。



8. 将量筒中剩余的 25 毫升的待测样品注入另外一支比色瓶中。(空白试样)



9. 将空白试样放入样品适配器中，并盖紧遮光盖。



10. 按 **ZERO** 指针将右移，屏幕会显示：

**0.00 mg/L Fe**



11. 定时器鸣响之后，将预制试样放入样品适配器中，盖上遮光盖。



12. 按下：**READ** 指针将右移，屏幕会显示总铁的含量，单位是 mg/L。

注：如果样品含有高含量的钼酸盐（不低于100 mg/L），应在空白调零后立即对样品读数。

注：应使用预制标准溶液进行标准校正。

## 干扰

加入试剂后样品的pH值如果少于3或大于4时，将会影响颜色的形成，可能会导致所形成的颜色退色或产生浑浊。因此在加入试剂前，应往量筒中逐滴加入1.0N的硫酸标准溶液或1.0N的氢氧化钠标准溶液，从而将样品的pH值调节为3到5之间。如果存在大量的酸或碱基，应进行容积校正。

## Sampling and Storage

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with hydrochloric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. If reporting only dissolved iron, filter the sample immediately after collection and before adding the acid. Before analysis, adjust the sample pH to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume; see *Correction for Volume Additions* in Section 1.

## Accuracy Check

### Standard Additions Method

- Snap the top off an Iron PourRite Ampule Standard Solution, 25 mg/L Fe.
- Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 50-mL samples. Swirl gently to mix.
- Analyze each sample as described above. The iron concentration should increase by 0.1 mg/L for each 0.2 mL of standard added.
- If these increases do not occur, see *Standard Additions* in Section 1 for more Information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. Prepare this solution daily. Analyze this working solution according to the above procedure. Results should be between 0.36 and 0.44 mg/L Fe.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.00 mg/L Fe and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.006$  mg/L Fe.

#### Estimated Detection Limit

The estimated detection limit for program 38 is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron (rust) to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering agent. The indicator reacts with the ferrous iron in the sample, buffered between pH 3-4, resulting in a deep blue-purple color.

### 所需试剂

|                                | 货号       |
|--------------------------------|----------|
| FerroMo试剂一套 (100 tests) .....  | 25448-00 |
| 包括: (4) 25437-68, (2) 25438-66 |          |

| 试剂种类                  | 所需数量<br>每次测试 | 单位           | 货号       |
|-----------------------|--------------|--------------|----------|
| FerroMo铁试剂 1 粉包 ..... | 1 包 .....    | 25/pkg ..... | 25437-68 |
| FerroMo 铁试剂2 粉包 ..... | 1 包 .....    | 50/pkg ..... | 25438-66 |

### 所需仪器

|                                 |         |             |          |
|---------------------------------|---------|-------------|----------|
| 剪刀 .....                        | 1 ..... | 个 .....     | 968-00   |
| 混合量筒, 50 mL .....               | 1 ..... | 个 .....     | 1896-41  |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 ..... | 6/pkg ..... | 24019-06 |

### OPTIONAL REAGENTS

|                                               |              |          |
|-----------------------------------------------|--------------|----------|
| Hydrochloric Acid Solution, 6.0 N (1:1) ..... | 500 mL ..... | 884-49   |
| Hydrochloric Acid, ACS .....                  | 500 mL ..... | 134-49   |
| Iron Standard Solution, 100 mg/L Fe .....     | 100 mL ..... | 14175-42 |
| Iron Standard Solution, PourRite Ampule,      |              |          |

|                                                 |                 |          |
|-------------------------------------------------|-----------------|----------|
| 25 mg/L Fe, 2 mL .....                          | 20/pkg.....     | 24629-20 |
| Sodium Hydroxide Standard Solution, 1.0 N ..... | 100 mL MDB..... | 1045-32  |
| Sodium Hydroxide Standard Solution, 5.0 N ..... | 100 mL MDB..... | 2450-32  |
| Sulfuric Acid Standard Solution, 1.0 N .....    | 100 mL MDB..... | 1270-32  |
| Water, deionized.....                           | 4 L.....        | 272-56   |

#### **OPTIONAL APPARATUS**

|                                            |                   |          |
|--------------------------------------------|-------------------|----------|
| Ampule Breaker Kit.....                    | each.....         | 24846-00 |
| Flask, volumetric, Class A, 250 mL .....   | each.....         | 14574-46 |
| pH Indicator Paper, 1 to 11 pH .....       | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>Sension.1</i> , portable..... | each.....         | 51700-10 |
| Pipet Filler, safety bulb .....            | each.....         | 14651-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....        | each.....         | 19700-01 |
| Pipet Tips, for 19700-01 Pipet .....       | 50/pkg.....       | 21856-96 |
| Pipet, volumetric, Class A, 1.00 mL.....   | each.....         | 14515-35 |

#### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.:**\*Call 800-227-4224

**Outside the U.S.A.:**\*Contact the Hach office or distributor serving you.

# 总铁 TPTZ 法 (0 to 1.80 mg/L)

方法号: 8112

## 使用试剂粉包



1. 输入检测总铁的程  
序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到更加精确  
的结果, 应使用去离  
子水进行试剂的空白  
校正。

注: 应在样品分析前  
调整所存样品的 *PH*  
值。

2. 按下: **39 ENTER**

屏幕将显示:

0.00 mg/L、Fe 和 ZERO

图标。

注: 总铁的检测要预  
先进行消解处理。

3. 往一支比色瓶中注  
入 10mL 样品。

注: 样品的 *PH* 值在检  
测中是十分重要的,

详情见干扰部分的内  
容。

注: 用 1: 1 的盐酸溶  
液清洗玻璃仪器, 然  
后用去离子水清洗。

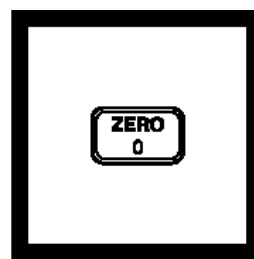
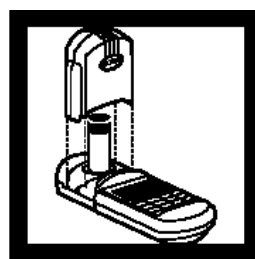
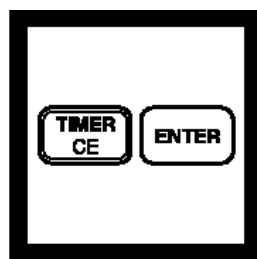
这样做可除去可能引  
起结果偏差的沉淀  
物。

4. 将一包 TPTZ

铁试剂粉包加入到比  
色瓶中。(预制试样)

盖好盖子, 摇晃 30  
秒钟使之溶解。

注: 如果存在铁, 溶  
液将呈现蓝色。



5. 按下 **Timer**  
开始 3 分钟的反应计  
时。

注: 在计时期间, 继  
续进行步骤 6-8 过  
程。

6. 往另外一支比色瓶  
中加入 10 毫升样品。  
(空白试样)

7. 将空白试样放入样  
品适配器中, 盖紧遮  
光盖。

注: 在计时期间, 按  
*Exit* 来读数清零。

8. 按下: **ZERO**  
指针将右移, 屏幕会  
显示:

**0.00 mg/L Fe**



9. 将预制试样放入样品适配器中，并盖紧遮光盖。
10. 按下：**READ**  
指针将右移，屏幕会显示总铁的含量，单位是 mg/L。

*注：应使用预制标准溶液进行标准校正。*

## 干扰

| 干扰物质   | 干扰水平和处理                                                                                                                                       |
|--------|-----------------------------------------------------------------------------------------------------------------------------------------------|
| 镉      | 大于4.0 mg/L。                                                                                                                                   |
| 铬(3+)  | 大于0.25 mg/L。                                                                                                                                  |
| 铬(6+)  | 大于1.2 mg/L。                                                                                                                                   |
| 钴      | 大于0.05 mg/L。                                                                                                                                  |
| 颜色或浑浊  | 在粉包程序中，如果样品不加入TPTZ铁试剂粉包呈现的颜色或出现的浑浊比步骤7中的空白试样（去离子水加TPTZ铁试剂）更重的话，用该样品作为空白试样。                                                                    |
| 铜      | 大于0.6 mg/L。                                                                                                                                   |
| 氰化物    | 大于2.8 mg/L。                                                                                                                                   |
| 锰      | 大于50.0 mg/L。                                                                                                                                  |
| 汞      | 大于0.4 mg/L。                                                                                                                                   |
| 钼      | 大于4.0 mg/L。                                                                                                                                   |
| 镍      | 大于1.0 mg/L。                                                                                                                                   |
| 亚硝酸盐离子 | 大于0.8 mg/L。                                                                                                                                   |
| pH     | 加入试剂后pH少于3或大于4的样品，会影响颜色的形成，引起已形成的颜色很快褪去或出现浑浊。在加入试剂前，在量筒中逐滴用不含铁的酸或碱，如1.0N的硫酸标准溶液或1.0N的氢氧化钠标准溶液将样品的pH调节为3到8之间，使用pH计或pH试纸。如果加入酸或碱的体积影响大，需要作体积校正。 |

## Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Store samples preserved in this manner up to six months at room temperature. If reporting only dissolved iron, filter sample immediately after collection and before addition of nitric acid.

Before testing, adjust the pH of the stored sample to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1*.

## Accuracy Check

### Standard Additions Method (Powder Pillows)

- a) Snap the neck off a PourRite Iron Ampule Standard, 25 mg/L Fe.
- b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10. Swirl to mix.
- c) Measure the iron concentration as in Step 10. The measurement does not require the three-minute waiting period.
- d) Add two additional 0.1-mL aliquots of standard, measuring the concentration after each addition. The iron concentration should increase by 0.25 mg/L for each 0.1-mL addition of standard.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Additions Method (AccuVac Ampuls)

- a) Use a graduated cylinder to measure 25.0 mL of sample into each of three 50-mL beakers.
- b) Snap the neck off an Iron Ampule Standard, 25 mg/L Fe.
- c) Using a TenSette Pipet, add 0.1, 0.2 and 0.3 mL of standard, respectively, to the 50-mL beakers. Swirl to mix.
- d) Fill a TPTZ AccuVac Ampul from each beaker.
- e) Measure the concentration of each ampul according to the procedure. The iron concentration should increase by 0.1 mg/L for each 0.1 mL addition of standard.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

- a) Using Class A glassware, pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.
- b) Dilute to volume with deionized water. Stopper and invert repeatedly to mix. Prepare this solution daily.

## Method Performance

### Precision

In a single laboratory using a standard solution of 1.00 mg/L Fe and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.017$  mg/L Fe.

In a single laboratory using a standard solution of 1.00 mg/L Fe and one representative lot of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.022$  mg/L Fe.

### Estimated Detection Limit

The estimated detection limit for program 39 is 0.04 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron.

### 所需试剂和仪器 (使用试剂粉包)

| 试剂种类                           | 所需数量 |         |          |
|--------------------------------|------|---------|----------|
|                                | 每次测试 | 单位      | 货号       |
| TPTZ 铁试剂粉包 .....               | 1 包  | 100/pkg | 26087-99 |
| 样品比色瓶, 10-20-25 mL, w/cap..... | 1    | 6/pkg   | 24019-06 |
| 所需试剂和仪器(使用 ACCUVAC 安瓿瓶)        |      |         |          |
| TPTZ 铁试剂AccuVac安瓿瓶.....        | 1 瓶  | 25/pkg  | 25100-25 |
| 烧杯, 50 mL.....                 | 1    | 个       | 500-41   |
| 样品比色瓶, 10-20-25 mL, w/cap..... | 1    | 6/pkg   | 24019-06 |

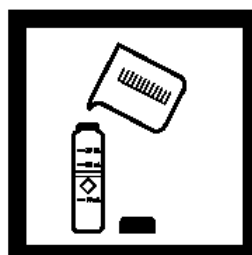
## OPTIONAL REAGENTS

|                                                                |            |          |
|----------------------------------------------------------------|------------|----------|
| Hydrochloric Acid Solution, 1:1, 6.0 N.....                    | 500 mL     | 884-49   |
| Iron Standard Solution, 100 mg/L Fe .....                      | 100 mL     | 14175-42 |
| Iron Standard Solution, PourRite Ampule, 25 mg/L Fe, 2 mL..... | 20/pkg     | 24629-20 |
| Nitric Acid, ACS.....                                          | 500 mL     | 152-49   |
| Nitric Acid Solution, 1:1.....                                 | 500 mL     | 2540-49  |
| RoVer Rust Remover .....                                       | 454 g      | 300-01   |
| Sodium Hydroxide Standard Solution, 1.0 N .....                | 100 mL MDB | 1045-32  |
| Sodium Hydroxide Standard Solution, 5.0 N .....                | 100 mL MDB | 2450-32  |
| Sulfuric Acid Standard Solution .....                          | 100 mL MDB | 1270-32  |
| Water, deionized.....                                          | 4 L        | 272-56   |

# 锰 高碘酸盐氧化法 高量程 (0 to 20.0 mg/L)

方法号: 8034

(需要预先进行消解处理)



1. 输入检测锰的高碘酸盐氧化法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为检测准确, 应用去离子水进行试剂空白校正。

2. 按下: 41 ENTER  
屏幕将显示:

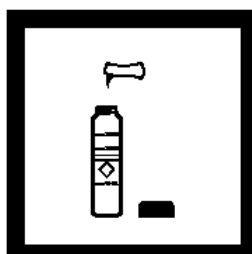
0.00 mg/L Mn 和 ZERO  
图标。

3. 往一个比色瓶中装入 10 mL 空白样品(空白试样)。

注: 为检测锰, 应测试开始时先进行消解处理。

注: 在检测前, 应调整所存样品的 PH 值。

4. 将空白试样放入样品适配器中, 并盖紧遮光盖。



5. 按 ZERO, 指针将右移, 屏幕显示:

0 mg/L Mn

注: 如果正在进行试剂空白校正, 屏幕将闪烁显示 "Limit"。

6. 将比色瓶从样品适配器中拿出, 加入柠檬酸盐类的缓冲药剂。盖上盖子倒转摇晃直到粉末完全溶解。再打开盖子。

7. 加入过高碘酸钠到比色瓶中(预制试样), 盖上盖, 倒转摇晃十秒钟。

8. 按下:  
TIMER 回车  
将会开始 2 分钟的反应。

注: 如果有锰存在, 溶液会呈现紫色。



9. 将预制试样放入样品适配器中，并盖紧遮光盖。

10. 按 **READ**  
指针将右移，屏幕会显示锰的浓度，单位是mg/L。

注：可用标准溶液进行标准校正。

## 干扰

当浓度超过以下所列情况，下列物质将产生干扰。

|     |              |
|-----|--------------|
| 钙   | 700 mg/L     |
| 氯化物 | 70,000 mg/L  |
| 铁   | 5 mg/L       |
| 镁   | 100,000 mg/L |

高缓冲样品或 PH 值高的样品可能会超过试剂的缓冲能力，此时需要进行样品的预处理。

## Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved Mn is to be determined, filter before acid addition.

## Accuracy Check

### Standard Additions Method

- Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.
- Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.

- c) Transfer only 10 mL of each solution to the 10-mL sample cells.
- d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

### Standard Solution Method

Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or Class A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.18$  mg/L Mn.

#### Estimated Detection Limit

The estimated detection limit for program 41 is 0.12 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.

### 所需试剂

|                                  |          |
|----------------------------------|----------|
|                                  | 货号       |
| 高量程锰试剂一套 (100 tests) 10 mL ..... | 24300-00 |
| 包括: (1) 21076-69, (1) 21077-69   |          |
| 所需试剂和仪器 (使用试剂粉包)                 |          |

| 试剂种类                           | 所需数量<br>每次测试 | 单位           | 货号       |
|--------------------------------|--------------|--------------|----------|
| 柠檬酸盐类的缓冲粉剂包.....               | 1 包.....     | 100/pkg..... | 21076-69 |
| 高碘酸钠粉末试剂包 .....                | 1 包.....     | 100/pkg..... | 21077-69 |
| 所需仪器                           |              |              |          |
| 样品比色瓶, 10-20-25 mL, w/cap..... | 2 .....      | 6/pkg.....   | 24019-06 |

## OPTIONAL REAGENTS

|                                                                                       |                 |          |
|---------------------------------------------------------------------------------------|-----------------|----------|
| Hydrochloric Acid, 6 N .....                                                          | 500 mL.....     | 884-49   |
| Manganese Standard Solution, 1000 mg/L Mn .....                                       | 100 mL.....     | 12791-42 |
| Manganese Standard Solution, Voluette ampule,<br>High Range, 250 mg/L Mn, 10 mL ..... | 16/pkg.....     | 14258-10 |
| Nitric Acid, ACS.....                                                                 | 500 mL.....     | 152-49   |
| Nitric Acid Solution 1:1.....                                                         | 500 mL.....     | 2540-49  |
| Sodium Hydroxide Solution, 5.0 N .....                                                | 100 mL MDB..... | 2450-32  |
| Water, deionized.....                                                                 | 4 L.....        | 272-56   |

## OPTIONAL APPARATUS

|                                                      |                  |          |
|------------------------------------------------------|------------------|----------|
| Ampule Breaker Kit.....                              | each.....        | 21968-00 |
| Flask, Erlenmeyer, 250 mL.....                       | each.....        | 505-46   |
| Flask, volumetric, Class A, 50 mL .....              | each.....        | 14574-41 |
| Flask, volumetric, Class A, 100 mL .....             | each.....        | 14574-42 |
| Flask, volumetric, Class A, 1000 mL .....            | each.....        | 14574-53 |
| pH Indicator Paper, 1 to 11 pH .....                 | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>sens<sup>ion</sup>1</i> , portable..... | each.....        | 51700-10 |
| Pipet, serological, 5 mL .....                       | each.....        | 532-37   |
| Pipet, TenSette, 0.1 to 1.0 mL.....                  | each.....        | 19700-01 |
| Pipet, TenSette, 1.0 to 10.0 mL.....                 | each.....        | 19700-10 |
| Pipet Tips, for 19700-01 TenSette Pipet .....        | 50/pkg.....      | 21856-96 |
| Pipet Tips, for 19700-10 TenSette Pipet .....        | 50/pkg.....      | 21997-96 |
| Pipet, volumetric, Class A, 5.00 mL.....             | each.....        | 14515-37 |
| Pipet, volumetric, Class A, 1.00 mL.....             | each.....        | 14515-35 |
| Pipet Filler, safety bulb .....                      | each.....        | 14651-00 |

### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.:** Call 800-227-4224

**Outside the U.S.A.:** Contact the Hach office or distributor serving you.

## 锰 PAN 方法

低量程

(0 to 0.700 mg/L)

方法号: 8149



1. 输入检测锰的PAN  
法的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**



2. 按下: **43 ENTER**

屏幕将显示:

0.00 mg/L、Mn 和 ZERO  
图标。

注: 如果测试其他形  
态的锰 (MnO<sub>4</sub>或着  
MnO<sub>2</sub>) 时, 按下:

**CONC 键**

注: 为检测总锰, 应  
测试开始时先进行消  
解处理。



3. 往一个比色瓶中装  
入 10 mL 去离子水(空  
白试样)。

注: 先用 1: 1 的硝酸  
溶液清洗所有的玻璃  
器具。然后再用去离  
子清洗。



4. 往另外一个比色瓶  
中装入 10 mL 带测样  
品 (预制试样)。



5. 分别往两个比色瓶  
中样品中加入抗败血  
酸粉末, 盖上瓶盖,  
倒转晃动使之混合。



6. 分别往两个比色瓶  
中样品中注入 15 滴  
碱性氰化物试剂溶  
液。晃动使之混合。

注: 加入碱性氰化物  
试剂溶液后有些样品  
会出现浑浊, 该浑浊  
将会在步骤8后消失。



7. 往每个样品中分别  
加入21滴 PAN 指示  
剂溶液, 0.1%, 到每  
个样品管中, 混合。

注: 如果样品存在锰,  
样品将呈现橙色。



8. 按下:  
**TIMER** 回车  
将会开始 2 分钟的  
反应。



9. 将空白试样放入样品适配器中，并盖紧遮光盖。

10. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L Mn**

11. 将预制试样放入样品适配器中，并盖紧遮光盖。

12. 按 READ 指针将右移，屏幕会显示锰的浓度，单位是mg/L。

注：可用标准溶液进行标准校正。

## 干扰

以下物质在所列的浓度范围内不产生干扰：

| 干扰物质 | 干扰水平和建议处理方式                                                     |
|------|-----------------------------------------------------------------|
| 铝    | 20mg/L                                                          |
| 钙    | 10mg/L                                                          |
| 钴    | 20mg/L                                                          |
| 铜    | 50mg/L                                                          |
| 硬度物质 | 300mg/L                                                         |
| 铁    | 如果样品中铁的含量超过5mg/L，则需要10分钟才能实现颜色完全显现。此时不执行步骤8，先设置定时器为10分钟，然后在按开始。 |
| 铅    | 0.5mg/L                                                         |
| 镁    | 如果样品中硬度物质超过300mg/L的CaCO <sub>3</sub> 。在加入抗坏血酸粉剂之前应注入4滴酒石酸钾溶液。   |
| 镍    | 40mg/L                                                          |
| 锌    | 15mg/L                                                          |

## Sampling and Storage

Collect samples in a clean glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 4.0-5.0 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correction for Volume Additions* in Section 1.

## Accuracy Check

### Standard Additions Method

**Note:** Volume accuracy is very important when performing standard additions with 10-mL volumes. The fill mark on the 10-mL sample cell is not intended to measure standard addition volumes.

- a) Fill three 10-mL graduated mixing cylinders with 10.0 mL of sample.
- b) Snap the neck off a Manganese Voluette Ampule Standard, 10 mg/L Mn.
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and mix each thoroughly.
- d) Analyze each sample as described in the procedure. The manganese concentration should increase 0.1 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

**Note:** An alternative to the above procedure is to pipet 10.0 mL of sample into dry sample cells before performing standard additions. A volumetric pipet or a TenSette Pipet can be used to deliver the sample volume.

### Standard Solution Method

Prepare a 0.5 mg/L manganese standard solution as follows:

- a) Pipet 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask.
- b) Dilute to the mark with deionized water. Prepare this solution daily.
- c) Pipet 10.00 mL of the solution from Step b into a 100-mL volumetric flask.
- d) Dilute to the mark with deionized water. This second dilution is equivalent to 0.5 mg/L Mn.

## Method Performance

### Precision

In a single laboratory using a standard solution of 0.5 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.013$  mg/L Mn.

### Estimated Detection Limit

The estimated detection limit for program 43 is 0.007 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

## Waste Management

The alkaline cyanide solution contains cyanide. Cyanide solutions should be collected for disposal as reactive (D003) waste. Store all cyanide solutions in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. In case of a spill, clean up the area as outlined below:

1. Use a fume hood or self-contained breathing apparatus.
2. While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
3. Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
4. Flush the solution down the drain with a large excess of water.

## Summary of Method

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to  $Mn^{2+}$ . An alkaline-cyanide reagent is added to mask any potential interferences. PAN Indicator is then added to combine with the  $Mn^{2+}$  to form an orange-colored complex.

## 所需试剂

高量程锰试剂一套 (100 tests) 10 mL ..... 24333-00  
货号  
包括: (2) 21223-32, (2) 14577-99, (2) 21224-32, (1) 21128-02

| 试剂种类              | 所需数量<br>每次测试 | 单位               | 货号       |
|-------------------|--------------|------------------|----------|
| 碱性氰化物试剂 .....     | 30 滴.....    | 50 mL SCDB ..... | 21223-26 |
| 抗坏血酸粉末试剂.....     | 2 包 .....    | 100/pkg .....    | 14577-99 |
| PAN 指示剂 0.1%..... | .42 滴 .....  | 50 mL SCDB ..... | 21224-26 |
| 去离子水 .....        | 10 mL.....   | 4 L .....        | 272-56   |

## REQUIRED APPARATUS

量筒, 25 mL ..... 1 ..... 个 ..... 508-40  
样品比色瓶, 10-20-25 mL, w/cap ..... 2 ..... 6/pkg ..... 24019-06

## OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1 (6 N)..... 500 mL ..... 884-49  
Manganese Standard Solution, 1000 mg/L Mn..... 100 mL ..... 12791-42  
Manganese Standard Sol<sup>n</sup>, PourRite Ampule, 20 mg/L Mn, 2 mL..... 20/pkg. .... 21128-20  
Nitric Acid Solution, 1:1 ..... 500 mL ..... 2540-49  
Rochelle Salt Solution..... 29 mL DB ..... 1725-33  
Sodium Hydroxide Solution, 50% ..... 500 mL ..... 2180-49  
Nitric Acid, ACS ..... 500 mL ..... 152-49

## OPTIONAL APPARATUS

Ampule Breaker, PourRite Ampule ..... each ..... 24846-00  
Beaker, glass, 1000 mL ..... each ..... 500-53  
Cylinder, graduated, mixing, 10 mL ..... each ..... 20886-38  
Dropper, plastic, calibrated, 1.0 mL..... 20/pkg ..... 21247-20  
Flask, volumetric, Class A, 1000 mL..... each ..... 14574-53  
Flask, volumetric, Class A, 100 mL..... each ..... 14574-42  
Pipet, TenSette, 0.1 to 1.0 mL..... each ..... 19700-01  
Pipet Tips, for 19700-01 TenSette Pipet ..... 50/pkg ..... 21856-96  
Pipet, volumetric, Class A, 10.0 mL ..... each ..... 14515-38  
Pipet, volumetric, Class A, 5.0 mL ..... each ..... 14515-37  
Pipet Filler, safety bulb..... each ..... 14651-00

## For Technical Assistance, Price and Ordering

In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 钼，钼酸盐 硫醋酸法 (0 — 40 mg/L) 高量程

方法号: 8036

使用试剂药粉



1. 输入检测高量程的钼的药剂法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?



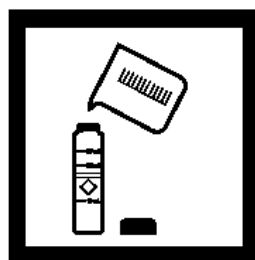
2. 按下: 44 ENTER  
屏幕将显示:

0.00 mg/L、Mo6

和 ZERO 图标。

注: 如果测试其他形态 (MnO4) 时, 按下:

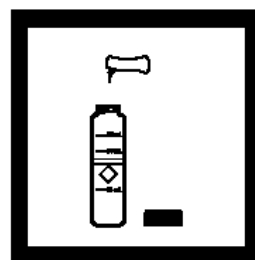
CONC 键



3. 往一个比色瓶中装入 10 mL 样品。

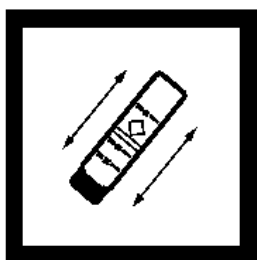
注: 过滤浑浊的样品。

注: 检测前将所存样品进行 PH 值调整。

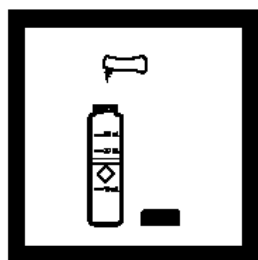


4. 加入一包 MolyVer 1 试剂粉包, 混合。

盖上瓶盖倒转多次使溶液混合。

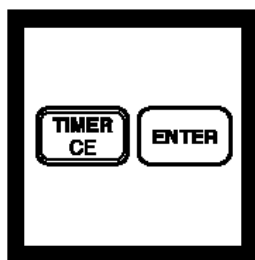


5. 加入一包 MolyVer 2 试剂粉包, 盖上瓶盖倒转多次使溶液混合。



6. 加入一包 MolyVer 3 试剂粉包, 盖上瓶盖倒转多次使溶液混合, 此为待测试样。

注: 不溶解的药剂不会影响检测的准确度。



7. 按下  
TIMER ENTER  
将开始进行 5 分钟反应。

注: 钼的存在会引起溶液呈现黄色。



8. 在计时器鸣响后, 将空白试样注入另外一支比色瓶中。(空白试样)



9. 将空白试样放入样品适配器中，并盖紧遮光盖。

10. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L Mo6**

注：如果正在进行试剂空白校正，屏幕将闪烁显示“Limit”。

11. 将预制试样瓶放入样品适配器中，并盖紧遮光盖。

12. 按：READ  
指针将右移，屏幕会显示的钼浓度，单位是mg/L。

注：可用标准溶液进行标准校正。

## 干扰

| 干扰物质         | 干扰水平和处理方法                                                            |
|--------------|----------------------------------------------------------------------|
| 铝            | 大于 50 mg/L                                                           |
| 铬            | 大于 1000mg/L                                                          |
| 铜            | 如果样品含有10 mg/L或以上的铜将会引起渐增的正干扰。应在5分钟反应时间结束之后尽快读数。                      |
| 铁            | 大于 50 mg/L                                                           |
| 镍            | 大于 50 mg/L                                                           |
| 亚硝酸盐         | 从大于 2000 mg/L的NO <sub>2</sub> <sup>-</sup> 开始干扰，可通过加入氨基磺酸粉包到样品中消除干扰。 |
| 高缓冲样品或极端pH样品 | 可能超过试剂的缓冲能力而需要样品预处理。                                                 |

## Sampling and Storage

Collect samples in clean plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo<sub>6</sub><sup>+</sup>.

- c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.
- d) For analysis with AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.
- e) Analyze each standard addition sample as described in the procedure. The molybdenum concentration reading should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

### Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo<sub>6+</sub>. Follow the procedure for powder pillows or AccuVac Ampuls. Results should be between 9.0 and 11.0 mg/L Mo<sub>6+</sub>.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory using a standard solution of 20.0 mg/L Mo<sub>6+</sub> and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of  $\pm 0.3$  mg/L Mo<sub>6+</sub>.

In a single laboratory using a standard solution of 20.0 mg/L Mo<sub>6+</sub> and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.1$  mg/L Mo<sub>6+</sub>.

### Estimated Detection Limit

The estimated detection limit for program 44 is 0.2 mg/L Mo<sub>6+</sub>. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

### Powder Pillows

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 1 contains a buffer to control the pH in addition to a chelating agent to mask interferences. MolyVer 3 provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

### AccuVac Ampuls

The CDTA Solution masks metal interferences. The MolyVer 6 reagent provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

## 所需试剂 (使用试剂粉包)

|                                                            | 货号       |
|------------------------------------------------------------|----------|
| 钼试剂一套, 10 mL (100 tests) .....                             | 26041-00 |
| 包括: (1) 26042-99, (1) 26043-99, (1) 26044-99, (1) 14265-02 |          |

| 试剂种类                 | 所需数量<br>每次测试 | 单位           | 货号       |
|----------------------|--------------|--------------|----------|
| MolyVer 1 试剂粉包 ..... | 1 包 .....    | 100/pkg..... | 26042-99 |
| MolyVer 2 试剂粉包 ..... | 1 包 .....    | 100/pkg..... | 26043-99 |
| MolyVer 3 试剂粉包 ..... | 1 包 .....    | 100/pkg..... | 26044-99 |

## 所需试剂 (使用 ACCUVAC 安瓿瓶)

|                                        |          |
|----------------------------------------|----------|
| MolyVer 6钼试剂AccuVac一套 (25 tests) ..... | 25220-98 |
| 包括: (1) 25220-25, (1) 26154-36         |          |

|                               |            |                 |          |
|-------------------------------|------------|-----------------|----------|
| CDTA 溶液 0.4M.....             | 4 滴.....   | 15 mL SCDB..... | 26154-36 |
| MolyVer 6 试剂AccuVac 安瓿瓶 ..... | 1 安瓿瓶..... | 25/pkg.....     | 25220-25 |

## 所需仪器 (使用试剂粉包)

|                                 |        |            |          |
|---------------------------------|--------|------------|----------|
| 剪刀 .....                        | 个..... | 个 .....    | 968-00   |
| 样品比色瓶 , 10-20-25 mL, w/cap..... | 2..... | 6/pkg..... | 24019-06 |

## 所需仪器(使用 ACCUVAC 安瓿瓶)

|                                |        |            |          |
|--------------------------------|--------|------------|----------|
| 烧杯, 50 mL.....                 | 2..... | 个 .....    | 500-41   |
| 样品比色瓶, 10-20-25 mL, w/cap..... | 1..... | 6/pkg..... | 24019-06 |

## OPTIONAL REAGENTS

|                                                                                          |                 |          |
|------------------------------------------------------------------------------------------|-----------------|----------|
| Molybdenum Standard Solution, 10 mg/L Mo <sub>6+</sub> .....                             | 100 mL.....     | 14187-42 |
| Molybdenum Standard Solution, Voluette Ampule,<br>500 mg/L Mo <sub>6+</sub> , 10 mL..... | 16/pkg.....     | 14265-10 |
| Nitric Acid, ACS.....                                                                    | 500 mL.....     | 152-49   |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                          | 100 mL MDB..... | 2450-32  |
| Sulfamic Acid Powder Pillows .....                                                       | 100/pkg.....    | 1055-99  |
| Water, deionized.....                                                                    | 4 L.....        | 272-56   |

## OPTIONAL APPARATUS

|                                               |              |          |
|-----------------------------------------------|--------------|----------|
| AccuVac Snapper Kit.....                      | each.....    | 24052-00 |
| Ampule Breaker Kit.....                       | each.....    | 21968-00 |
| Cylinder, graduated, mixing, 25 mL.....       | each.....    | 20886-40 |
| Filter Paper, folded, 12.5 cm.....            | 100/pkg..... | 1894-57  |
| Flask, Erlenmeyer, 250 mL.....                | each.....    | 505-46   |
| Funnel, poly, 65 mm .....                     | each.....    | 1083-67  |
| Pipet, serological, 5 mL .....                | each.....    | 532-37   |
| Pipet, TenSette, 0.1 to 1.0 mL.....           | each.....    | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg.....  | 21856-96 |

## For Technical Assistance, Price and Ordering

In the U.S.A.:"Call 800-227-4224

Outside the U.S.A.:"Contact the Hach office or distributor serving you.

# 钼，钼酸盐 三元复合法 (0 — 3 mg/L) 低含量

方法号: 8169



1. 输入检测低浓度的钼的三元复合法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: 47 ENTER

屏幕将显示:

0.00 mg/L、Mo6

和 ZERO 图标。

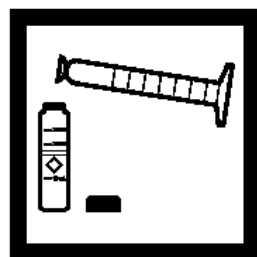
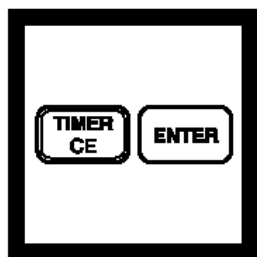
注: 如果测试其他形态 (MnO4) 时, 按下:

CONC 键

3. 将 20ml 的样品注入刻度为 25 mL 的混合量筒内。

注: 过滤浑浊的样品。

4. 将一包钼 1 试剂粉包加入混合量筒。盖上瓶盖倒转多次使之溶解。



5. 将量筒中 10 毫升溶液注入一支比色瓶中。

6. 再往比色瓶中加入 0.5ml 的钼 2 试剂。晃动使之混合。这就是预制试样。

7. 按下:

TIMER ENTER

将开始进行 2 分钟反应。

8. 将量筒中的 10ml 剩余溶液注入另外一支比色瓶中。(空白试样)

注: 钼的存在会导致溶液呈绿色。



9. 将空白试样放入样品适配器中，并盖紧遮光盖。



10. 按 **ZERO**，指针将右移，屏幕显示：  
**0 mg/L Mo6**

注：如果正在进行试剂空白校正，屏幕将闪烁显示“Limit”。



11. 将预制试样放入样品适配器中，并盖紧遮光盖。



12. 按：**READ**  
指针将右移，屏幕会显示的钼浓度，单位是mg/L。

注：可用标准溶液进行标准校正。

## 干扰

可以用钼的标准溶液（2 mg/L Mo<sup>6+</sup>）和可能的干扰离子进行干扰测试。在特定的离子浓度下，当离子对钼标准浓度的影响超过±5%就被认为有干扰。下表是该干扰测试的细节。

表1 负干扰

| 干扰物质         | 大于所列水平产生水平 |
|--------------|------------|
| 铁            | 200 mg/L   |
| 铜            | 98 mg/L    |
| 铬 (Cr6+)     | 4.5 mg/L   |
| 氯化物          | 1,400 mg/L |
| AMP（膦酸盐）     | 15 mg/L    |
| 磷脂羟基乙酸, 乙酸   | 32 mg/L    |
| 硫酸氢盐         | 3,300 mg/L |
| 铝            | 2 mg/L     |
| 丙烯酸盐         | 790mg/L    |
| 明矾           | 7 mg/L     |
| 磺酸盐          | 105 mg/L   |
| 正磷酸盐         | 4500 mg/L  |
| 重碳酸盐         | 5,650 mg/L |
| 乙烯乙二醇        | 2%(体积)     |
| 亚硫酸盐         | 6,500 mg/L |
| 盐酸乙醇二硫代氨基甲酸盐 | 32 mg/L    |
| 正干扰          |            |
| 碳酸盐          | 1,325 mg/L |
| 硅            | 大于600 mg/L |
| 苯并三唑         | 大于210 mg/L |
| 吗啉HEDP       | 大于6 mg/L   |

- 2分钟计时结束后应该立即读出钼的浓度。

表2 不会产生干扰情况

| 干扰物质       | 所测试的最高浓度 (mg/L) |
|------------|-----------------|
| 锌          | 400             |
| 钙          | 720             |
| 镁          | 8000            |
| 锰          | 1600            |
| 氯          | 7.5             |
| PBTC (磷酸盐) | 500             |
| 硫酸盐        | 12800           |
| 亚硫酸盐       | 9,600           |
| 镍          | 250             |

磷酸盐HEDP在浓度不大于30g/L时,将会使钼的读数增加10% (正干扰)。在这些情况下,应将步骤9所得的结果乘以0.9从而得到钼正确的含量。当HEDP的浓度增加到30mg/L以上时,钼含量的读数将偏小。(负干扰)

高缓冲样品或极端PH值的样品可能超过试剂的缓冲能力而需要样品预处理。逐滴加入适当的酸或碱,如1.0N的硫酸标准溶液或1.0N的氢氧化钠标准溶液将样品的pH调节为3到5之间,使用pH计或pH试纸。如果加入酸或碱的体积影响大,需要作体积校正,将总体积划分为(样品+酸+碱),应乘以校正因子来校正测试结果。

在冷却塔里,一些生物杀灭剂会产生较大的干扰。哈西公司建议对这些含有特种生物杀灭剂的样品进行三重复杂的检测步骤,从而判断这些方法对其有没有起作用。

许多样品经过检测后,样品将呈现淡蓝色。器具应用1:1的盐酸溶液清洗,从而去除内积物。

## Sampling and Storage

Collect samples in glass or plastic bottles.

## Accuracy Check

### Standard Addition Method

- Add 25 mL of sample to three 25-mL mixing cylinders.
- Snap the neck off a Molybdenum PourRite Ampule Standard Solution, 75 mg/L Mo6+.
- Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix thoroughly.
- Analyze 20 mL of each spiked sample as described in the procedure. The molybdenum concentration reading should increase by 0.3 mg/L for each 0.1 mL addition of standard.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 2.0-mg/L molybdenum standard solution by pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-mL graduated mixing cylinder. Dilute to the mark with deionized water and mix thoroughly. Analyze 20 mL of this solution according to the procedure.

## Method Performance

### Precision

In a single laboratory using standard solutions of 2.00 mg/L Mo<sup>6+</sup> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.009$  mg/L Mo<sup>6+</sup>.

### Estimated Detection Limit

The estimated detection limit for program 47 is 0.07 mg/L Mo<sup>6+</sup>. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex.

### 所需试剂

钼试剂t, 20 mL 样品 (100 tests) .....24494-00  
包括: (1) 23524-49, (1) 23525-12, (1) 25575-02

| 试剂种类           | 所需数量<br>每次测试 | 单位                   | 货号                    |
|----------------|--------------|----------------------|-----------------------|
| 钼 1 粉末试剂 ..... | 1            | 包                    | 100/pkg .....23524-49 |
| 钼 2 试剂溶液.....  | 0.5 mL       | ..... 50 mL MDB..... | 23525-12              |

### 所需仪器

混合量筒, 25 mL ..... 1.....个 .....1896-40  
样品比色瓶, 10-20-25 mL, w/cap ..... 2.....6/pkg .....24019-06

## OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1, 6.0 N ..... 500 mL .....884-49  
Molybdenum Standard Solution, PourRite Ampule  
75 mg/L Mo<sup>6+</sup>, 2 mL .....20/pkg .....25575-20  
Molybdenum Standard Solution, 10 mg/L Mo<sup>6+</sup>..... 100 mL .....14187-42  
Sodium Hydroxide Standard Solution, 1.0 N..... 100 mL MDB.....1045-32  
Water, deionized ..... 4 L .....272-56

## OPTIONAL APPARATUS

Cylinder, mixing, graduated, 50 mL .....each .....1896-41  
Filter Paper, folded, 12.5 cm .....100/pkg .....1894-57  
Funnel, poly, 65 mm.....each .....1083-67  
pH Paper, 1-11 pH units .....5 rolls/pkg .....391-33  
Pipet, TenSette, 0.1 to 1.0 mL.....each .....19700-01  
Pipet Tips, for 19700-01 TenSette Pipet .....50/pkg .....21856-96  
Pipet, volumetric, 10.00 mL, Class A .....each .....14515-38  
Pipet Filler, safety bulb.....each .....12189-00  
PourRite Ampule Breaker .....each .....24846-00

### For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A. Contact the Hach office or distributor serving you.

## 镍 PAN 法 (0 to 1.000 mg/L)

方法号 8150



1、按“PRGM”键，萤幕会显示 PRGM?



2、输入内设程式代号“48”然后按下“ENTER”键，萤幕会出现“mg/L Ni”及“ZERO Icon”



3、取一支比色瓶加水样至 25ml 标线处当待溶液。



4、取另一支比色瓶加水样，盖好瓶盖当空白溶液。

*注：假如水样温度低于室温，在分析前将水样加热至室温。*



5、各加入一邻苯二甲酸盐-磷酸盐试剂至两支比色瓶中盖上瓶盖摇动数次使充分混合。

*注：假如水样含  $Fe^{+3}$ ，在进行步骤6之前必须使试剂完全溶解。*



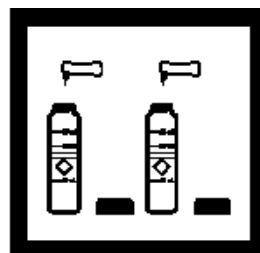
6、各加入 1.0ml 的 0.3% PAN 指示剂至两支比色瓶中，盖好瓶盖，摇动数次使充分混合。

*注：以塑胶吸管取用试剂。*



7、同时按“TIMER”及“ENTER”键，将进行 15 分钟的反应计时

*注：此时水样颜色将从黄橘色转成暗红色，空白溶液将呈黄色。*



8、当计时完毕，听到哔哔声，各加入一 EDTA 试剂至两支比色瓶中，盖好瓶盖，摇动数次使完全溶解。



- 9、放空白溶液至比色计中测试，并将比色计盖子盖好。
- 10、按“ZERO”键归零 萤幕会显示 0.000mg/l Ni。
- 11、放待测溶液至比色计中，并将比色计盖子盖好。
- 12、按“READ”键，所欲测浓度会显示出来即 mg/l Ni。

## 干扰

以下物质超过表中所列的浓度时会干扰

| 表干扰物质            | 干扰水平和处理                              |
|------------------|--------------------------------------|
| Al <sup>3+</sup> | 32 mg/L                              |
| Ca <sup>2+</sup> | 1000 mg/L (CaCO <sub>3</sub> )       |
| Cd <sup>2+</sup> | 20 mg/L                              |
| Cl <sup>-</sup>  | 8000 mg/L                            |
| 螯合剂              | 所有水平上均干扰。用Digesdahl或强烈消化消除干扰（参阅第2部分） |
| Cr <sup>3+</sup> | 20 mg/L                              |
| Cr <sup>6+</sup> | 40 mg/L                              |
| Cu <sup>2+</sup> | 15 mg/L                              |
| F <sup>-</sup>   | 20 mg/L                              |
| Fe <sup>3+</sup> | 10 mg/L                              |
| Fe <sup>2+</sup> | 直接干扰，不能存在。                           |
| K <sup>+</sup>   | 500 mg/L                             |
| Mg <sup>2+</sup> | 400 mg/L                             |
| Mn <sup>2+</sup> | 25 mg/L                              |
| Mo <sup>6+</sup> | 60 mg/L                              |
| Na <sup>+</sup>  | 5000 mg/L                            |
| Pb <sup>2+</sup> | 20 mg/L                              |
| Zn <sup>2+</sup> | 30 mg/L                              |
| 高缓冲样品或极端样品pH     | 可能超过试剂的缓冲能力而需要样品预处理。                 |

## Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions, see *Correcting for Volume Additions*, (Section I) for more information.

## Accuracy Check

### Standard Solution Method

Prepare a 0.5 mg/L nickel standard solution by diluting 10.0 mL of a 5 mg/L working stock solution to 100 mL in a 100-mL volumetric flask. The working stock solution should be prepared daily by diluting 5.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with deionized water. Or, using the TenSette Pipet, add 0.2 mL of a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni, into a 100-mL volumetric flask. Dilute to volume with deionized water. This is a 0.6 mg/L standard solution.

## Method Performance

### Precision

In a single laboratory using a standard solution of 0.50 mg/L nickel and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.008$  mg/L nickel.

### Estimated Detection Limit

The estimated detection limit for program 48 is 0.013 mg/L Ni. For more information on the estimated detection limit, see *Section I*.

## Summary of Method

After buffering the sample and masking any Fe<sup>3+</sup> with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator. The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt.

## 所需试剂

|                                             | 货号       |
|---------------------------------------------|----------|
| 镍试剂一套, 25 mL 样品 (100 tests) .....           | 22426-00 |
| 包括: (2) 7005-99, (4) 21501-66, (2) 21502-32 |          |

| 试剂种类                          | 所需数量<br>每次测试 | 单位              | 货号       |
|-------------------------------|--------------|-----------------|----------|
| EDTA 粉末试剂包 .....              | 2 包 .....    | 100/pkg.....    | 7005-99  |
| 邻苯二甲酸盐-磷酸盐试剂粉包 .....          | 2 包 .....    | 50/pkg.....     | 21501-66 |
| P.A.N. 指示剂, 0.3%.....         | 2 mL.....    | 100 mL MDB..... | 21502-32 |
| 样品比色瓶, 10-20-25, w/caps ..... | 2.....       | 6/pkg.....      | 24019-06 |
| 去离子水 .....                    | 10 mL.....   | 4 L.....        | 272-56   |

## 所需仪器

|                  |        |        |          |
|------------------|--------|--------|----------|
| 剪刀, .....        | 1..... | 个..... | 968-00   |
| 混合量筒, 25 mL..... | 1..... | 个..... | 20886-40 |

## OPTIONAL REAGENTS

|                                                                    |                 |          |
|--------------------------------------------------------------------|-----------------|----------|
| Nickel Standard Solution, 1000 mg/L Ni .....                       | 100 mL.....     | 14176-42 |
| Nickel Standard Solution, Voluette Ampule, 300 mg/L Ni, 10 mL..... | 16/pkg.....     | 14266-10 |
| Nitric Acid, ACS.....                                              | 500 mL.....     | 152-49   |
| Nitric Acid Solution, 1:1.....                                     | 500 mL.....     | 2540-49  |
| Sodium Hydroxide Standard Solution, 5.0 N .....                    | 100 mL MDB..... | 2450-32  |

## OPTIONAL APPARATUS

|                                               |                  |          |
|-----------------------------------------------|------------------|----------|
| Ampule Breaker Kit.....                       | each.....        | 21968-00 |
| Flask, volumetric, Class A, 100 mL .....      | each.....        | 14574-42 |
| Flask, volumetric, Class A, 1000 mL .....     | each.....        | 14574-53 |
| pH Paper, 1 to 11 pH units.....               | 5 rolls/pkg..... | 391-33   |
| pH Meter, EC10, portable.....                 | each.....        | 50050-00 |
| Pipet, serological, 1 mL .....                | each.....        | 532-35   |
| Pipet, serological, 5 mL .....                | each.....        | 532-37   |
| Pipet, TenSette, 0.1 to 1.0 mL.....           | each.....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg.....      | 21856-96 |
| Pipet, volumetric, Class A, 5.0 mL.....       | each.....        | 14515-37 |
| Pipet, volumetric, Class A, 10.0 mL.....      | each.....        | 14515-38 |
| Pipet Filler, safety bulb .....               | each.....        | 14651-00 |
| Thermometer, -10 to 110 °C.....               | each.....        | 1877-01  |

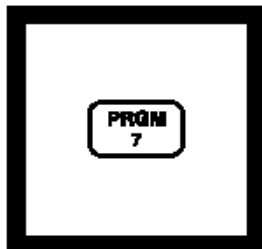
## *For Technical Assistance, Price and Ordering*

In the U.S.A.:<sup>®</sup>Call 800-227-4224

Outside the U.S.A.:<sup>®</sup>Contact the Hach office or distributor serving you.

## 硝酸盐 镉还原法 高量程 (0 to 30.0 mg/L NO<sub>3</sub>-N)

方法号: 8039



1、按“PRGM”键此时萤幕显示 PRGM?

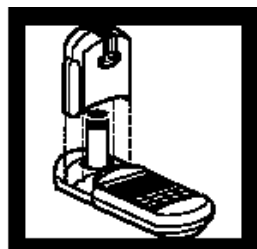
注: 欲得最正确的测试结果, 可使用去离子水当空白溶液。

2、输入内设程式代号“51”然后按下“ENTER”键, 此时萤幕出现“mg/l, NO<sub>3</sub>-N”及“ZERO icon”

3、取一支比色瓶加水样至 25ml 标线处。

4、加入一 NitraVer5 试剂至比色瓶盖, 摇动使充分混合, 当待测溶液。

注: 在此须特别注意将铝箔包中的试剂, 完全倒出, 不可残留。



5、同时按“TIMER”及“ENTER”键, 将进行一分钟的反应计时, 快速摇动比色瓶, 直到计时完毕。

6、计时完毕, 听到哔哔声后, 萤幕将显示“5: 00 TIMER 2”按“ENTER”键, 将再进行 5 分钟, 反应计时。

注: 假如水样含有硝酸亚氮, 则会呈琥珀色。

7、另一支比色瓶, 加入 10ml 水样, 当空白溶液。

注: 若有沉淀出现在比色瓶底部, 并不会影响测试结果。

8、放空白溶液至比色槽中, 并将比色计盖上盖子。



9、当计时完毕，听到哔哔声后，按“ZERO”键归零，萤幕会显示：  
0.00mg/1 NO<sub>3</sub>-N

10、取出空白溶液，放待测溶液至比色槽中，并将比色计盖上盖子。

11、按“READ”键，所欲测浓度将会显示出来，即 mg/1 NO<sub>3</sub>-N

*注：测试完毕，必须立刻冲洗比色瓶以除去所有含镉的颗粒，并适当处理此具危险性的废液，不可随意丢弃。*

## 干扰物质

| 干扰物质      | 干扰物允许水平及对策                                                                                                                                                           |
|-----------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 氯离子       | 如果水样中的氯离子浓度超过 100 mg/L，则会导致测量结果偏低。虽然如此，本方法仍然可以用于测量高含量氯化物水样（比如海水）中的硝酸盐，只不过所用的标准溶液也要有相同的氯离子浓度。                                                                         |
| 铁离子       | 只要存在就会产生干扰。                                                                                                                                                          |
| 亚硝酸盐      | 只要存在就会产生干扰，可以采取以下方法补偿亚硝酸离子的干扰：<br>1. 在步骤 3 之前，逐滴地向水样中加入 30g/L 的溴水（Hach #2211-20）直至水样的颜色变黄；<br>2. 加入一滴 30g/L 的酚溶液（Hach #2112-20）以消除产生的黄颜色；<br>继续作步骤 3，测量结果包括硝酸盐和亚硝酸盐。 |
| pH        | 如果水样的酸度过高，超过了本方法所采用的试剂所能调节的程度，那么就必须对水样进行前处理。                                                                                                                         |
| 强氧化剂和强还原剂 | 只要存在就会产生干扰                                                                                                                                                           |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard, 500 mg/L nitrate nitrogen.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen Standard Solution to the three samples. Stopper and mix thoroughly.
- d) For AccuVac analysis, transfer the solutions to clean, dry 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to clean, dry sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen ( $\text{NO}_3^-$ -N) concentration should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Use a Hach Nitrate-Nitrogen Standard Solution, 10.0 mg/L  $\text{NO}_3^-$ -N, listed under Optional Reagents as the sample and perform the procedure as described above.

## Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. See *Section 1, Standard Curve Adjustment* for more information. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust value is entered.

## Method Performance

### Precision

In a single laboratory using standard solutions of 25.0 mg/L nitrate nitrogen ( $\text{NO}_3^-$ -N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of +0.3 mg/L nitrate nitrogen for program #50 and  $\pm 1.7$  mg/L nitrate nitrogen for program # 51.

### Estimated Detection Limit

The estimated detection limit for program 50 is 0.5 mg/L NO<sub>3</sub> -N and 0.8 mg/L NO<sub>3</sub> -N for program 51. For more information on the estimated detection limit, see *Section 1*.

### Summary Of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

### Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

#### 所需试剂和仪器 (使用试剂粉包)

| 种类                              | 所需数量<br>每次测试 | 单位            | 货号       |
|---------------------------------|--------------|---------------|----------|
| NitraVer 5硝酸盐试剂粉包 .....         | 1 包 .....    | 100/pkg ..... | 21061-69 |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 .....      | 6/pkg .....   | 24019-06 |

#### 所需试剂和仪器(使用 ACCUVAC 安瓿瓶)

|                         |          |              |          |
|-------------------------|----------|--------------|----------|
| NitraVer 5硝酸盐试剂安瓿瓶..... | 1 瓶..... | 25/pkg ..... | 25110-25 |
| 烧杯 , 50 mL .....        | 1 .....  | each .....   | 500-41   |
| 塞子 .....                | 1 .....  | 6/pkg .....  | 1731-06  |

### OPTIONAL REAGENTS

|                                                                                                     |              |       |          |
|-----------------------------------------------------------------------------------------------------|--------------|-------|----------|
| Bromine Water 30 g/L.....                                                                           | 29 mL *      | ..... | 2211-20  |
| Nitrate Nitrogen Standard Solution, 10.0 mg/L as (NO <sub>3</sub> --N) .....                        | 500 mL ..... | ..... | 307-49   |
| Nitrate Nitrogen Standard Solution, 1000 mg/L as (NO <sub>3</sub> --N) .....                        | 500 mL ..... | ..... | 12792-49 |
| Nitrate Nitrogen Standard Solution, PourRite ampule,<br>500 mg/L as NO <sub>3</sub> --N, 2 mL ..... | 20/pkg ..... | ..... | 14260-20 |
| Phenol Solution .....                                                                               | 29 mL .....  | ..... | 2112-20  |
| Sodium Hydroxide Standard Solution, 5.0 N.....                                                      | 50 mL *      | ..... | 2450-26  |
| Sulfuric Acid, ACS.....                                                                             | 500 mL *     | ..... | 979-49   |
| Water, deionized .....                                                                              | 4 L .....    | ..... | 272-56   |

### OPTIONAL APPARATUS

|                                                                    |                  |          |
|--------------------------------------------------------------------|------------------|----------|
| AccuVac Snapper Kit.....                                           | each.....        | 24052-00 |
| Cylinder, graduated, mixing, 25 mL.....                            | each.....        | 1896-40  |
| Dropper, for 29-mL bottle.....                                     | each.....        | 2258-00  |
| pH Indicator Paper, 1 to 11 pH .....                               | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>sens<sup>ion</sup></i> <sup>TM</sup> I, portable..... | each.....        | 51700-10 |
| Pipet Filler, safety bulb .....                                    | each.....        | 14651-00 |
| Pipet, serological, 2 mL .....                                     | each.....        | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL.....                                | each.....        | 19700-01 |

|                                               |             |          |
|-----------------------------------------------|-------------|----------|
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg..... | 21856-96 |
| PourRite Ampule Breaker.....                  | each.....   | 24846-00 |
| Thermometer, -10 to 110 °C .....              | each .....  | 1877-01  |

***For Technical Assistance, Price and Ordering***

**In the U.S.A. call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 硝酸盐 镉还原法 中量程 (0 to 5.0 mg/L NO<sub>3</sub>-N)

方法号: 8171

使用试剂粉包



1. 输入检测中量程硝酸盐的试剂法的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到更加精确的结果, 应用去离子水进行试剂空白校正。

2. 按下: **54 ENTER**

屏幕将显示:

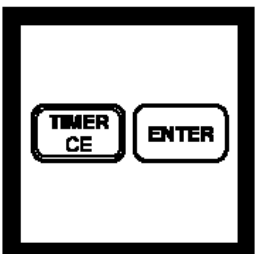
0.00 mg/L、**NO<sub>3</sub>-N** 和 **ZERO** 图标。

注: 如果测试其他形态(**NO<sub>3</sub>**)时, 按下:

**CONC 键**

3. 分别往两支比色瓶中各装入 10 mL 样品。其中一支作为预制试样, 另外一支作为空白试样。

4. 往预制试样瓶中加入一份 NitraVer 5 硝酸盐试剂粉末(预制试样), 盖好瓶盖。



5. 按下:

**TIMER ENTER**

将开始进行 1 分钟反应。大力摇晃比色瓶直到计时器鸣响为止。

注: 摇晃时间和方式将会影响颜色显现。如果摇晃不够力度, 将会得到偏低结果。

6. 在计时器鸣响后, 屏幕将显示:

**5:00 TIMER 2**

再按下 **ENTER**

将开始 5 分钟的计时。

注: 当所加入 NitraVer 5 硝酸盐试剂粉溶解后将产生镉的沉淀物, 但是不会影响结果。

注: 如果氮存在, 溶液将呈现琥珀色。

7. 计时器鸣响后, 擦干净瓶壁的液体和手印。

8. 将空白试样放入样品适配器中, 盖紧遮光盖。



9. 按 ZERO, 指针将右移, 屏幕显示:  
**0 mg/L NO3-N**

注: 如果正在进行试剂空白校正, 屏幕将闪烁显示 “Limit”。

10. 将预制试样放入样品适配器中, 并盖紧遮光盖。

11. 按 READ 指针将右移, 屏幕会显示的NO<sub>3</sub>-N (or NO<sub>3</sub>)浓度, 单位是mg/L。

注: 建议对每种试剂进行标准校正。

。

## 干扰

| 干扰物质      | 干扰物允许水平及对策                                                                                                                                                                     |
|-----------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 氯化物       | 如果水样中的氯离子浓度超过 100 mg/L, 则会导致测量结果偏低。虽然如此, 本方法仍然可以用于测量高含量氯化物水样 (比如海水) 中的硝酸盐, 只不过所用的标准溶液也要有相同的氯离子浓度。                                                                              |
| 铁离子       | 只要存在就会产生干扰。                                                                                                                                                                    |
| 亚硝酸盐      | 只要存在就会产生干扰, 可以采取以下方法补偿亚硝酸离子的干扰:<br>1. 在步骤 3 之前, 逐滴地向水样中加入 30g/L 的溴水 (Hach #2211-20) 直至水样的颜色变黄;<br>2. 加入一滴 30g/L 的酚溶液 (Hach #2112-20) 以消除产生的黄颜色;<br>3. 继续作步骤 3, 测量结果包括硝酸盐和亚硝酸盐。 |
| pH        | 如果水样的酸度过高, 超过了本方法所采用的试剂所能调节的程度, 那么就必须对水样进行前处理。                                                                                                                                 |
| 强氧化剂和强还原剂 | 只要存在就会产生干扰                                                                                                                                                                     |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions*, (Section 1) for more information.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 100 mg/L NO<sub>3</sub>-N.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50 mL beakers. For analysis with powder pillows, transfer only 10 mL of the solution to dry, clean sample cells.
- e) Analyze each sample as described above. The nitrate nitrogen (NO<sub>3</sub>-N) concentration should increase 0.4 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

### Standard Solution Method

A 1.0 mg/L Nitrate Nitrogen Standard Solution is available from Hach. Use this standard in place of sample in the above procedure.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See Section 1, *Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory using a standard solution of 3.0 mg/L nitrate nitrogen (NO<sub>3</sub>-N) and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of +0.2 mg/L nitrate nitrogen.

In a single laboratory using a standard solution of 3.0 mg/L NO<sub>3</sub>-N and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of +0.1 mg/L nitrate nitrogen.

### Estimated Detection Limit

The estimated detection limit for programs 53 and 54 is 0.2 mg/L NO<sub>3</sub>-N. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

### Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

#### 所需试剂和仪器 (使用试剂粉包)

| 种类                              | 所需数量<br>每次测试 | 单位            | 货号       |
|---------------------------------|--------------|---------------|----------|
| NitraVer 5硝酸盐试剂粉包 .....         | 1 包 .....    | 100/pkg ..... | 21061-69 |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 .....      | 6/pkg .....   | 24019-06 |

#### 所需试剂和仪器(使用 ACCUVAC 安瓿瓶)

|                         |          |              |          |
|-------------------------|----------|--------------|----------|
| NitraVer 5硝酸盐试剂安瓿瓶..... | 1 瓶..... | 25/pkg ..... | 25110-25 |
| 烧杯 , 50 mL .....        | 1 .....  | each .....   | 500-41   |
| 塞子 .....                | 1 .....  | 6/pkg .....  | 1731-06  |

### OPTIONAL REAGENTS

|                                                                                                   |                  |         |
|---------------------------------------------------------------------------------------------------|------------------|---------|
| Bromine Water 30 g/L .....                                                                        | 29 mL* .....     | 2211-20 |
| Nitrate Nitrogen Standard Solution, 1.0 mg/L as NO <sub>3</sub> -N.....                           | 500 mL.....      | 2046-49 |
| Nitrate Nitrogen Standard Solution, 100 mg/L as NO <sub>3</sub> -N.....                           | 500 mL.....      | 1947-49 |
| Nitrate Nitrogen Standard Solution, PourRite Ampule,<br>100 mg/L as NO <sub>3</sub> -N, 2 mL..... | 20/pkg.....      | 1947-20 |
| Phenol Solution, 30 g/L .....                                                                     | 29 mL.....       | 2112-20 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                   | 50 mL SCDB*..... | 2450-26 |
| Sulfuric Acid, ACS .....                                                                          | 500 mL* .....    | 979-49  |
| Water, deionized.....                                                                             | 4 L.....         | 272-56  |

### OPTIONAL APPARATUS

|                                            |                  |          |
|--------------------------------------------|------------------|----------|
| AccuVac Snapper Kit.....                   | each.....        | 24052-00 |
| Cylinder, graduated, mixing, 25 mL.....    | each.....        | 20886-40 |
| Dropper, for 1-oz bottle .....             | each.....        | 2258-00  |
| pH Paper, 1 to 11 pH units.....            | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>sension™</i> I, portable..... | each.....        | 51700-10 |
| Pipet Filler, safety bulb .....            | each.....        | 14651-00 |
| Pipet, serological, 2 mL .....             | each.....        | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL.....        | each.....        | 19700-01 |

|                                               |             |          |
|-----------------------------------------------|-------------|----------|
| Pipet Tips, for 19700-01 TenSette Pipet ..... | 50/pkg..... | 21856-96 |
| PourRite Ampule Breaker.....                  | each.....   | 24846-00 |

***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

# 硝酸盐 镉还原法 低量程 (0 to 0.50 mg/L NO<sub>3</sub>-N)

方法号: 8192



1. 输入检测低量程硝酸盐的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为得到更加精确的结果, 应用去离子水进行试剂空白校正。



2. 按下: 55 ENTER  
屏幕将显示:

0.00 mg/L、NO<sub>3</sub>-N 和 ZERO 图标。

注: 如果测试其他形态(NO<sub>3</sub>)时, 按下: CONC 键



3. 往容积为 25ml 量筒中加入 15mL 的待测样品。

注: 检测前调节溶液的 PH 值。



4. 加一份 NitraVer 6 硝酸盐试剂粉末到量筒中, 盖好瓶盖。



5. 按下:

TIMER ENTER

将开始进行 3 分钟反应。在反应期间, 大力摇晃量筒。

注: 摇晃时间和方式将会影响颜色显现。如果摇晃不够力度, 将会得到偏低结果。



6. 当计时器鸣响后, 屏幕将显示:

2:00 TIMER 2

按下: ENTER

将开始 2 分钟的反应计时。

注: 粉剂溶解后将产生沉淀, 但是不会影响检测结果。



7. 在计时器鸣响后, 从量筒中倒出10ml注入到一支比色瓶中。



8. 往比色瓶中加入一份 NitraVer 3 硝酸盐试剂粉末 (预制试样)。盖上瓶盖轻轻摇晃30秒。

注: 如果硝酸盐存在, 溶液将呈现粉红色。



9. 屏幕将显示:

**15:00 TIMER 3**

按下: **ENTER**

将开始 15 分钟的反应计时。往另外一支比色瓶中注入 10 毫升样品。(空白试样)

10. 将空白试样瓶放入样品适配器中, 并盖紧遮光盖。

11. 按 **ZERO**, 指针将右移, 屏幕显示:

**0 mg/L NO<sub>3</sub>-N**

注: 如果正在进行试剂空白校正, 屏幕将闪烁显示 "Limit"。

12. 将预制试样瓶放入样品适配器中, 并盖紧遮光盖。



13. 按 **READ**

指针将右移, 屏幕会显示的NO<sub>3</sub>-N (or NO<sub>3</sub>)浓度, 单位是 mg/L。

注: 建议对每种试剂进行标准校正。

## 干扰

| 干扰物质      | 干扰物允许水平及对策                                                                                                                                                              |
|-----------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 钙         | 100mg/L                                                                                                                                                                 |
| 氯离子       | 如果水样中的氯离子浓度超过 100 mg/L，则会导致测量结果偏低。虽然如此，本方法仍然可以用于测量高含量氯化物水样（比如海水）中的硝酸盐，只不过所用的标准溶液也要有相同的氯离子浓度。                                                                            |
| 铁离子       | 只要存在就会产生干扰。                                                                                                                                                             |
| 亚硝酸盐      | 只要存在就会产生干扰，可以采取以下方法补偿亚硝酸离子的干扰：<br>1. 在步骤 3 之前，逐滴地向水样中加入 30g/L 的溴水（Hach #2211-20）直至水样的颜色变黄；<br>2. 加入一滴 30g/L 的酚溶液（Hach #2112-20）以消除产生的黄颜色；<br>3. 继续作步骤 3，测量结果包括硝酸盐和亚硝酸盐。 |
| pH        | 如果水样的酸度过高，超过了本方法所采用的试剂所能调节的程度，那么就必须对水样进行前处理。                                                                                                                            |
| 强氧化剂和强还原剂 | 只要存在就会产生干扰                                                                                                                                                              |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct the test result for volume additions; see *Correction for Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard additions Method

- Fill three 25-mL graduated mixing cylinders with 15 mL of sample.
- Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 12.0 mg/L NO<sub>3</sub>-N.
- Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.
- Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.08 mg/L for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of a 10.0 mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water. Use this standard in place of sample in Step 3.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 0.20-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **0.20** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment.

If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust feature is entered. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory using a standard solution of 0.25 mg/L nitrate nitrogen (NO<sub>3</sub>-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of +0.03 mg/L nitrate nitrogen.

### Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO<sub>3</sub>-N. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to chromotropic acid to form a pink-colored product.

## Pollution Prevention and Waste Management

NitaVer 6 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

### 所需试剂

低量程硝酸盐试剂一套 (100 tests)..... 24298-00  
包括: (1) 21071-69, (1) 21072-49

| 试剂种类                    | 所需数量<br>每次测试 | 单位      | 货号       |
|-------------------------|--------------|---------|----------|
| NitriVer 3 硝酸盐试剂粉包..... | 1 包          | 100/pkg | 21071-69 |
| NitraVer 6 硝酸盐试剂粉包..... | 1 包          | 100/pkg | 21072-49 |

### 所需仪器

|                                 |   |       |          |
|---------------------------------|---|-------|----------|
| 混合量筒, 25 mL.....                | 1 | 个     | 1896-40  |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 2 | 6/pkg | 24019-06 |

## OPTIONAL REAGENTS

### Description Unit Cat. No.

|                                                                                                    |                  |          |
|----------------------------------------------------------------------------------------------------|------------------|----------|
| Bromine Water, 30 g/L.....                                                                         | 29 mL*           | 2211-20  |
| Nitrate Nitrogen Standard Solution, 10.0 mg/L as NO <sub>3</sub> --N.....                          | 500 mL.....      | 307-49   |
| Nitrate Nitrogen Standard Solution, Voluette ampule,<br>12 mg/L as NO <sub>3</sub> --N, 2 mL ..... | 20/pkg.....      | 25587-20 |
| Phenol Solution, 30 g/L .....                                                                      | 29 mL.....       | 2112-20  |
| Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20 .....                                         | each.....        | 2268-00  |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                    | 50 mL* SCDB..... | 2450-26  |
| Sulfuric Acid, ACS .....                                                                           | 500 mL* .....    | 979-49   |
| Water, deionized.....                                                                              | 4 L.....         | 272-56   |

## OPTIONAL APPARATUS

|                                                        |                 |          |
|--------------------------------------------------------|-----------------|----------|
| Ampule Breaker, PourRite Ampules .....                 | each.....       | 24846-00 |
| Dropper, for 29-mL bottle.....                         | each.....       | 2258-00  |
| Flask, volumetric, Class A, 100 mL .....               | each.....       | 14574-42 |
| pH Indicator Paper, 1 to 11 pH .....                   | 5-roll/pkg..... | 391-33   |
| pH Meter, <i>sens<sup>ion</sup>™ I</i> , portable..... | each.....       | 51700-10 |
| Pipet, serological, 2 mL .....                         | each.....       | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL.....                    | each.....       | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....          | 50/pkg.....     | 21856-96 |
| Pipet, volumetric, Class A, 2.00 mL.....               | each.....       | 14515-36 |
| Pipet Filler, safety bulb .....                        | each.....       | 14651-00 |
| Thermometer, -10 to 110 °C .....                       | each .....      | 1877-01  |

Nitrate at these levels can be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 50235-00).

### *For Technical Assistance, Price and Ordering*

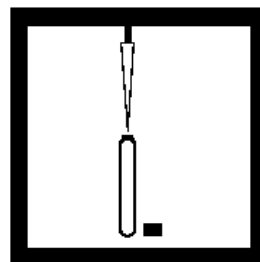
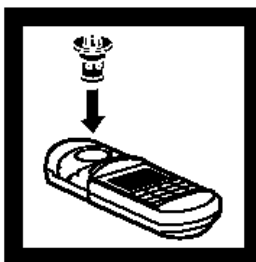
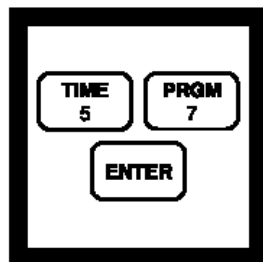
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

# 硝酸盐 铬变酸法 高量程 (0 to 0.50 mg/L NO<sub>3</sub>-N)

方法号: 10020

Test 'N Tube



1. 输入检测高量程总  
硝酸盐Test 'N Tube  
的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

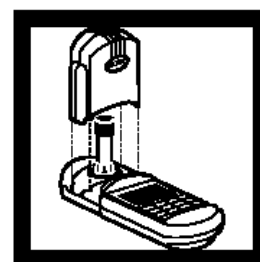
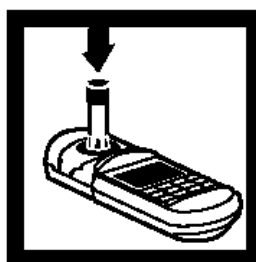
2. 按下: 57 ENTER  
屏幕将显示:

0.00 mg/L、NO<sub>3</sub>-N  
和ZERO图标。

3. 旋转 COD/TNT 适配  
器, 将其嵌入瓶管架  
上适当的位置, 然后  
下按使之完全嵌入。

4. 打开装有硝酸盐预  
处理溶液瓶, 往里面  
加入 1.00 mL 样品(此  
为样品空白)

注: 为得到精确结果,  
应使用去离子水进行  
试剂空白校正。



5. 盖上瓶盖, 反转十  
次使溶液混合。

6. 用毛巾擦干净瓶的  
外表。

7. 将空白试样放入样  
品适配器中。

8. 盖紧瓶盖。

注: 该检测在技术上是灵敏的。如果不按照指引进行操作, 将可能导致结果偏低。应该将瓶保持垂直放置。反转试样瓶使瓶盖朝下, 等待一阵后, 再反转试样瓶, 直到溶液流到瓶底。该过程为一次反转。重复十次。



9. 按下: **ZERO**

指针将右移, 屏幕将显示:

**0.0 mg/L NO<sub>3</sub>-N**

10. 把试样瓶拿出来, 摘去瓶盖。

11. 用漏斗将一包 NitraVer X试剂B加到试样瓶子中。反转瓶十次使溶液混合。(预制试样)。

12. 按下: **TIMER**

再按下: **ENTER**

将开始 5 分钟的反应计时。此时不要反转试样瓶。

注: 如果硝酸盐存在, 溶液将呈现黄色。

注: 在计时器鸣响后 5 分钟内, 完成步骤 13—16 步。



13. 在计时器鸣响后, 用湿毛巾擦干净瓶的外表, 再用干毛巾擦去手印和其他印记。

14. 将预制试样放入样品适配器中。

15. 盖紧瓶盖。

16. 按下: **READ**

指针将右移, 屏幕将显示NO<sub>3</sub>-N的含量, 单位是mg/L。

注: 应用预制标准溶液进行标准校正。

## 干扰

| 干扰物质 | 干扰水平和处理                                                                |
|------|------------------------------------------------------------------------|
| 钡    | 浓度大于1 mg/L产生负干扰。                                                       |
| 氯化物  | 低于1000 mg/L不干扰。                                                        |
| 硬度   | 不干扰。                                                                   |
| 亚硝酸盐 | 浓度大于12 mg/L产生负干扰。去除100mg/L以内的亚硝酸盐干扰可加入400mg尿素到10mL样品中。混合溶解。继续常规的硝酸盐测试。 |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a fresh High Range Nitrate Nitrogen Voluette Ampule Standard, 500 mg/L NO<sub>3</sub>-N.
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to the three mixing cylinders, respectively. Mix each thoroughly.
- d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the spiked sample in each test. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

To test accuracy, prepare a 20.0 mg/L nitrate nitrogen standard solution by pipetting 2.00 mL of a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L NO<sub>3</sub>-N, into a 50 mL Class A volumetric flask. Dilute to the line with deionized water. Substitute this standard for the sample and perform the test as described in the procedure.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 25.0 mg/L nitrate nitrogen (NO<sub>3</sub>-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L NO<sub>3</sub>-N.

### Estimated Detection Limit

The estimated detection limit for program 57 is 0.3 mg/L NO<sub>3</sub>-N. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

## 所需试剂

NitraVer 硝酸盐 Test i®N Tube 试剂一套 (50 tests) ..... 26053-45

包括: (1) 26055-46, (1) 272-42, \*(50) 硝酸盐预处理溶液瓶

| 试剂种类                   | 所需数量<br>每次测试 | 单位          | 货号       |
|------------------------|--------------|-------------|----------|
| Nitrate硝酸盐预处理溶液瓶.....  | 1.....       | 50/pkg..... | *        |
| NitraVer·试剂 B 粉包 ..... | 1.....       | 50/pkg..... | 26055-46 |

## 所需仪器

|                                               |              |             |          |
|-----------------------------------------------|--------------|-------------|----------|
| COD 适配器 .....                                 | 1.....       | 个.....      | 48464-00 |
| 漏斗.....                                       | 1.....       | 个.....      | 25843-35 |
| TenSette移液管, 0.1 to 1.0 mL.....               | 1.....       | 个.....      | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet ..... | varies ..... | 50/pkg..... | 21856-96 |
| 试管架.....                                      | 1-3 .....    | 个.....      | 18641-00 |

## OPTIONAL REAGENTS

Nitrate-Nitrogen Standard Solution, Voluette

|                                                 |             |          |
|-------------------------------------------------|-------------|----------|
| Ampules, 500 mg/L N .....                       | 16/pkg..... | 14260-10 |
| Sodium Hydroxide Standard Solution, 5.0 N ..... | 50 mL ..... | 2450-26  |
| Sulfuric Acid, ACS, concentrated.....           | 500 mL..... | 979-49   |
| Urea, ACS.....                                  | 100 g.....  | 11237-26 |
| Water, deionized.....                           | 4 L.....    | 272-56   |

## OPTIONAL APPARATUS

|                                                      |                  |          |
|------------------------------------------------------|------------------|----------|
| Ampule Breaker Kit.....                              | each.....        | 21968-00 |
| Cylinder, graduated, mixing, 25-mL (3 required)..... | each.....        | 26363-40 |
| Flask, volumetric, Class A, 50 mL .....              | each.....        | 14574-41 |
| pH Paper, 1 to 11 pH units.....                      | 5 rolls/pkg..... | 391-33   |
| Pipet, volumetric, Class A, 2 mL.....                | each.....        | 14515-36 |
| Spoon, measuring, 0.5 g.....                         | each.....        | 907-00   |

## For Technical Assistance, Price and Ordering

In the U.S.A.:<sup>†</sup>Call 800-227-4224

Outside the U.S.A.:<sup>†</sup>Contact the Hach office or distributor serving you.

# 亚硝酸盐 硫酸亚铁法 高量程 (0 to 150 mg/L NO<sub>2</sub>-)

方法号: 8153



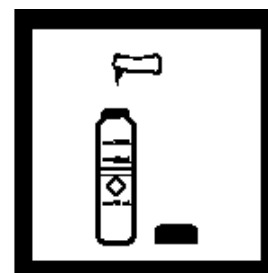
1、按“PRGM”键，此时萤幕会显示 PRGM?



2、输入内设程式代号“59”，然后按下“ENTER”键，此时萤幕会出现“mg/l, NO<sub>2</sub>”及“ZERO lcon”



3、取一支比色瓶加水样至 10ml 标线处。  
注：若有沉淀出现在比色瓶底部，并不会影响测试结果。



4、加一 Nitri Ver2 试剂至比色瓶中，盖好瓶盖，摇动 5~7 次使充分混合，当待测溶液。

注：若水样中含有亚硝酸氮，会呈棕绿色。

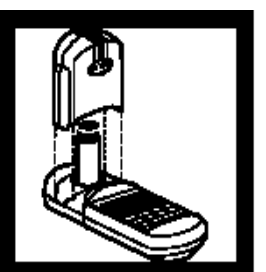


5、同时按“TIMER”及“ENTER”键，将进行 10 分钟的反应计时，在此期间，请勿摇动比色瓶。

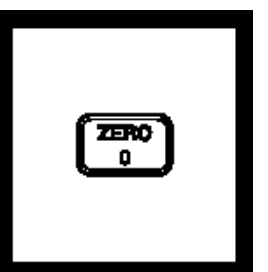


6、取另一支比色瓶，加入 10ml 水样当空白溶液。

注：使用干净的毛巾擦拭比色瓶外部，除去指印或其他痕迹。



7、放空白溶液至比色槽中，并将比色计盖上盖子。



8、按“ZERO”键归零，萤幕会显示 0mg/l NO<sub>2</sub>



- 9、当计时完毕，听到哔哔声后，来回摇动待测溶液比色瓶 2 次后，放待测溶液至比色槽中，并将比色计盖上盖子
- 10、按“READ”键，所测浓度将会显示出来，即 mg/L NO<sub>2</sub>

### Sampling and Storage

Collect samples in clean plastic or glass bottles. If prompt analysis is impossible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 48 hours. Warm to room temperature before running the test. Do not use acid preservatives. Remove suspended solids by filtration.

### Accuracy Check

#### Standard Solution Method

Dissolve 0.150 grams of fresh sodium nitrite and dilute to 1000 mL with deionized water to prepare a 100 mg/L nitrite standard solution. Prepare this solution daily. Alternatively, make a dilution of a fresh Hach Nitrite Standard Solution, 821 mg/L NO<sub>2</sub> (250 mg/L NO<sub>2</sub>-N) using Class A glassware. Dilute 10mL of this standard to 100 mL with deionized water to give an 82 mg/L nitrite standard. Prepare this solution just before use. Using this solution as the sample, perform the nitrite procedure as described above.

### Method Performance

#### Precision

In a single laboratory using a standard solution of 123 mg/L nitrite and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of +1 mg/L nitrite.

#### Estimated Detection Limit

The estimated detection limit for program 59 is 2 mg/L NO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

## Interferences

This test does not measure nitrates nor is it applicable to glycol based samples. Dilute glycol based samples and follow the Low Range Nitrite Procedure.

## Summary of Method

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present.

## 所需试剂和仪器

| 试剂种类                         | 所需数量<br>每次测试 | 单位            | 货号       |
|------------------------------|--------------|---------------|----------|
| NitriVer 2 硝酸盐试剂粉末 .....     | 1 包.....     | 100/pkg ..... | 21075-69 |
| 样品比色瓶, 10-20-25, w/ cap..... | 2 .....      | 6/pkg .....   | 24019-06 |

## OPTIONAL REAGENTS

|                                                                                        |              |          |
|----------------------------------------------------------------------------------------|--------------|----------|
| Nitrite Standard Solution, 821 mg/L NO <sub>2</sub> (250 mg/L NO <sub>2</sub> -N)..... | 500 mL ..... | 23402-49 |
| Sodium Nitrite, ACS .....                                                              | 454 g .....  | 2452-01  |
| Water, deionized .....                                                                 | 4 L .....    | 272-56   |

## OPTIONAL APPARATUS

|                                            |            |          |
|--------------------------------------------|------------|----------|
| Balance, analytical, 110 V.....            | each ..... | 26103-00 |
| Balance, analytical, 220 V.....            | each ..... | 26103-02 |
| Flask, volumetric, 1000 mL .....           | each ..... | 547-53   |
| Flask, volumetric, 100 mL, Class A.....    | each ..... | 14574-42 |
| Pipet, volumetric, 10.00 mL, Class A ..... | each ..... | 14515-38 |
| Pipet Filler, safety bulb.....             | each ..... | 14651-00 |

## *For Technical Assistance, Price and Ordering*

In the U.S.A.:<sup>a</sup>Call 800-227-4224

Outside the U.S.A.:<sup>a</sup>Contact the Hach office or distributor serving you.

## 亚硝酸盐 重氮化作用法 低量程 (0 to 0.350 mg/L NO<sub>2</sub>-N) 方法号: 8507



1、“PRGM”键，此时萤幕显示 PRGM?

注：欲得最正确的测试结果，可使用去离子水当空白溶液。



2、输入内设程式代号“60”，然后按下“ENTER”键，此时萤幕出现“mg/l, NO<sub>2</sub>-N”及“ZERO icon”

注：NO<sub>2</sub>及NaNO<sub>2</sub>之间的浓度换算请按“CONC”键。



3、取一支比色瓶加水样至 10ml 标线处。



4. 加一NitriVer 2试剂至比色瓶中，盖好瓶盖，摇动使充分混合。

注：若有试剂未完全溶解，并不影响测试结果。



5、同时按“TIMER”及“ENTER”键，将进行 15 分钟的反应计时。

注：假如水样中含有亚硝酸盐氮，会呈粉红色。



6、当计时完毕，听到哔哔声，加 10ml 水样至另一比色瓶中，当作空白溶液。



7、以干净的毛巾擦拭比色瓶外部，放空白溶液至比色槽中，并将比色计盖上盖子



8、按“ZERO”键归零，萤幕会显示 0.000mg/L NO<sub>2</sub>-N



9、放待测溶液至比色槽中, 并将比色计盖上盖子

10、按 ” READ” 键, 所欲测浓度将会显示出来, 即 mg/L

## 干扰

| 干扰物质名称    | 干扰物质最大允许含量及消除干扰的办法                                                                             |
|-----------|------------------------------------------------------------------------------------------------|
| 铋离子       | 会产生沉淀                                                                                          |
| 金离子       | 会产生沉淀                                                                                          |
| 铋离子       | 会产生沉淀                                                                                          |
| 氯铂酸盐离子    | 会产生沉淀                                                                                          |
| 铜离子       | 会导致测量结果偏低                                                                                      |
| 亚铁离子      | 会导致测量结果偏低                                                                                      |
| 铁离子       | 会产生沉淀                                                                                          |
| 铅离子       | 会产生沉淀                                                                                          |
| 汞离子       | 会产生沉淀                                                                                          |
| 偏钒酸盐离子    | 会产生沉淀                                                                                          |
| 硝酸根离子     | 水样中如果存在很高含量的硝酸根(>100 mg/L), 其中的一部分会被还原为亚硝酸根, 这种变化可能是自然发生的, 也可能在测量过程中发生, 这样, 水样中就一定会存在一定量的亚硝酸根。 |
| 银离子       | 会产生沉淀                                                                                          |
| 强氧化、还原性物质 | 只要存在就会产生干扰                                                                                     |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives. Remove the suspended solids by filtration.

## Accuracy Check

### Standard Solution Method

Pipet 5.00 mL of a fresh 250 mg/L NO<sub>2</sub>-N standard into a 250.0 mL volumetric flask. Dilute to the mark with deionized water. This makes a 5.00-mg/L intermediate standard. To prepare a 0.100-mg/L NO<sub>2</sub>-N standard solution, dilute 10.00 mL of the 5.00-mg/L intermediate standard to 500 mL in a volumetric flask. Prepare this solution immediately before use. Run the test using the 0.100 mg/L NO<sub>2</sub>-N standard in place of

the sample. Results should be between 0.090 and 0.110 mg/L NO<sub>2</sub>-N.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.001$  mg/L NO<sub>2</sub>-N for the powder pillow method and  $\pm 0.003$  mg/L NO<sub>2</sub>-N for the AccuVac method.

### Estimated Detection Limit

The estimated detection limit for programs 60 and 62 is 0.005 mg/L NO<sub>2</sub>-N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

## 所需试剂

| 试剂种类                              | 所需数量<br>每次测试 | 单位           | 货号       |
|-----------------------------------|--------------|--------------|----------|
| NitriVer 3 硝酸盐试剂粉包.....           | 1 包.....     | 100/pkg..... | 21071-69 |
| 或                                 |              |              |          |
| NitriVer 3 硝酸盐试剂 AccuVac 安瓿瓶..... | 1 安瓿瓶.....   | 25/pkg.....  | 25120-25 |

## 所需仪器

|                                                    |         |            |          |
|----------------------------------------------------|---------|------------|----------|
| 烧杯, 50 mL (for AccuVac procedure) .....            | 1 ..... | 个.....     | 500-41   |
| or                                                 |         |            |          |
| 样品比色瓶, 10-20-25 mL (powder pillow procedure) ..... | 2 ..... | 6/pkg..... | 24019-06 |

## OPTIONAL REAGENTS

|                                                                 |              |          |
|-----------------------------------------------------------------|--------------|----------|
| Nitrite Standard Solution, 250 mg/L as NO <sub>2</sub> -N ..... | 500 mL ..... | 23402-49 |
| Water, deionized.....                                           | 4 L.....     | 272-56   |

## OPTIONAL APPARATUS

| Description                                  | Unit        | Cat. No. |
|----------------------------------------------|-------------|----------|
| AccuVac Snapper Kit.....                     | each.....   | 24052-00 |
| Flask, volumetric, 250 mL.....               | each.....   | 14574-46 |
| Flask, volumetric, 500 mL.....               | each.....   | 14574-49 |
| Pipet, serological, 10 mL .....              | each.....   | 532-38   |
| Pipet, TenSette, 1 to 10 mL.....             | each.....   | 19700-01 |
| Pipet Tips for 19700-01 TenSette Pipet ..... | 50/pkg..... | 21856-96 |
| Pipet, volumetric, Class A, 5.00 mL.....     | each.....   | 14515-37 |
| Pipet, volumetric, Class A, 10.00 mL.....    | each.....   | 14515-38 |
| Pipet Filler, safety bulb .....              | each.....   | 14651-00 |
| Thermometer, -10 to 110 °C.....              | each.....   | 1877-01  |

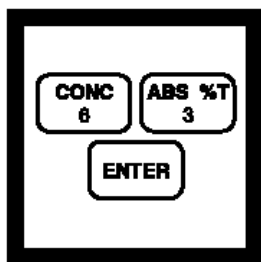
## For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 亚硝酸盐 重氮化作用法 低量程 (0 - 0.500 mg/L NO<sub>2</sub>--N) 方法号: 8507

Test 'N Tube



1. 输入检测低量程亚硝酸盐Test 'N Tube的程序编号。

按下: PRGM

屏幕将显示:

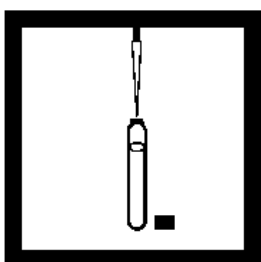
PRGM?

2. 按下: 63 ENTER  
屏幕将显示:

0.00 mg/L、NO<sub>3</sub>-N  
和ZERO图标。

3. 旋转 COD/TNT 适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。

4. 往Test 'N Tube 样品瓶中加入一份 NitriVer3 亚硝酸试剂, 盖紧摇晃至完全溶解, 这就是预制试样。



5. 按下:

TIMER ENTER

将开始 20 分钟的反应计时。

6. 当计时器鸣响后, 往另外一支Test 'N Tube样品瓶注入5毫升的样品。(空白试样)

7. 用毛巾擦干净瓶的外壁。

8. 将空白试样放入样品适配器中。盖紧瓶盖。

注: 如果亚硝酸盐存在, 溶液将呈现粉红色。



9. 盖紧瓶盖。

10. 按下: **ZERO**  
指针将右移, 屏幕将  
显示:  
**0.0 mg/L NO3-N**

11. 将预制试样反应  
样品适配器中。

12. 盖紧瓶盖。  
按下: **READ**  
指针将右移, 屏幕将  
显示NO3-N的含量, 单  
位是mg/L。

*注: 应用预制标准溶  
液进行标准校正*

## 干扰

| 干扰物质名称    | 干扰物质最大允许含量及消除干扰的办法                                                                             |
|-----------|------------------------------------------------------------------------------------------------|
| 铈离子       | 会产生沉淀                                                                                          |
| 金离子       | 会产生沉淀                                                                                          |
| 铋离子       | 会产生沉淀                                                                                          |
| 氯铂酸盐离子    | 会产生沉淀                                                                                          |
| 铜离子       | 会导致测量结果偏低                                                                                      |
| 亚铁离子      | 会导致测量结果偏低                                                                                      |
| 铁离子       | 会产生沉淀                                                                                          |
| 铅离子       | 会产生沉淀                                                                                          |
| 汞离子       | 会产生沉淀                                                                                          |
| 偏钒酸盐离子    | 会产生沉淀                                                                                          |
| 硝酸根离子     | 水样中如果存在很高含量的硝酸根(>100 mg/L), 其中的一部分会被还原为亚硝酸根, 这种变化可能是自然发生的, 也可能在测量过程中发生, 这样, 水样中就一定会存在一定量的亚硝酸根。 |
| 银离子       | 会产生沉淀                                                                                          |
| 强氧化、还原性物质 | 只要存在就会产生干扰                                                                                     |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives. Remove suspended solids by filtration.

## Accuracy Check

### Standard Solution Method

Pipet 5.00 mL of a fresh Hach standard, 250 mg/L as NO<sub>2</sub> -N into a Class A 250-mL volumetric flask. Dilute to the line with deionized water to make a 5.00-mg/L intermediate standard. Pipet 10.00 mL of the 5.0-mg/L intermediate standard into a Class A 500-mL volumetric flask. Dilute to the line with deionized water to make a 0.100 mg/L NO<sub>2</sub>-N standard solution. Prepare immediately before use.

Run the test using the 0.100 mg/L NO<sub>2</sub>-N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO<sub>2</sub> -N.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L NO<sub>2</sub>-N.

### Estimated Detection Limit

The estimated detection limit for program 63 is 0.006 mg/L NO<sub>2</sub>-N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink-colored complex directly proportional to the amount of nitrite present.

## 所需试剂

NitriVer. 3 硝酸盐 Test i®N Tube 试剂 Set (50 tests) .....26083-45

包括:

(50) NitriVer. 3 硝酸盐 Test i®N 试管瓶 .....\*

试管瓶, 6 x 10 mm, 6/pkg .....22758-06

瓶盖, for 22758-06 vials, 6/pkg.....22411-06

去离子水, 100-mL.....272-42

## 所需仪器

| 种类                                           | 所需数量<br>每次测试 | 单位          | 货号       |
|----------------------------------------------|--------------|-------------|----------|
| COD/TNT 适配器.....                             | 1            | 个.....      | 48464-00 |
| 试管架.....                                     | 1-3.....     | 个.....      | 18641-00 |
| 移液管, TenSette, 1 to 10 mL.....               | 1            | 个.....      | 19700-10 |
| Pipet Tips for 19700-10 TenSette Pipet ..... | 1            | 50/pkg..... | 21997-96 |

**OPTIONAL REAGENTS**

|                                                                 |              |          |
|-----------------------------------------------------------------|--------------|----------|
| Nitrite Standard Solution, 250 mg/L as NO <sub>2</sub> -N ..... | 500 mL ..... | 23402-49 |
| Water, deionized.....                                           | 4 L.....     | 272-56   |

**OPTIONAL APPARATUS**

|                                           |           |          |
|-------------------------------------------|-----------|----------|
| Flask, volumetric, 250 mL.....            | each..... | 14574-46 |
| Flask, volumetric, 500 mL.....            | each..... | 14574-49 |
| Pipet, volumetric, Class A, 10.00 mL..... | each..... | 14515-38 |

***For Technical Assistance, Price and Ordering***

**In the U.S.A. call 800-227-4224**

**Outside the U.S.A. Contact the Hach office or distributor serving you.**

# 氨氮 水杨酸盐法 (0 to 0.50 mg/L NH<sub>3</sub>-N)

方法号: 8155



1、按“PRGM”键，萤幕会显示 PRGM?



2、输入内设程式代号“64”然后按下“ENTER”键，萤幕会出现“mg/l”Nha-N及“ZERO lcon”



3、取一支比色瓶加入去离子至 10ml 标线处当空白溶液。



4、取另一支比色瓶加水样，当待测溶液



5、各加入一Ammonia Salicylate试剂至两支比色瓶中，盖好瓶盖摇动使完完全溶解。



6、同时按“TIMER”及“ENTER”键，将进行3分钟的反应计时。



7. 当计时完毕，听到哗哗声，各加入 Ammonia Cyanurate至两支比色瓶中，盖好瓶盖，使完全溶解



8、三分钟计时完毕，萤幕将显示：15: 00 TIMER2，按“ENTER”键，将再进行 15 分钟反应计时

注：假如水样含氨氮，将呈绿色



- 9、当计时完毕，听到哔哔声，立即将空白溶液放入比色计中测试，将比色计盖子盖好
- 10、按“ZERO”键归零，萤幕会显示 0.000 mg/l NH<sub>3</sub>-N
- 11、放待测溶液至比色计中，并将比色计盖子盖好
- 12、按“READ”键，所欲测浓度会显示出来，即 mg/l NH<sub>3</sub>-N

## 干扰

| 干扰物质名称  | 干扰物质最大允许含量及消除干扰的办法                                                                                                                                                                                                                |
|---------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 钙       | 最大允许含量 1000 mg/L，以 CaCO <sub>3</sub> 计                                                                                                                                                                                            |
| 氨基乙酸、联胺 | 会导致被测水样的颜色加深                                                                                                                                                                                                                      |
| 铁       | 各个水平均产生干扰，可以按照以下步骤扣除铁的干扰： <ol style="list-style-type: none"> <li>1. 测量水样中总铁的含量</li> <li>2. 准备反应相同铁离子浓度的去离子水，作为原始样品。将该溶液进行检测。用待测样品实际读数减去刚才所得的结果就可得校正的结果。</li> </ol>                                                                |
| 镁       | 最大允许含量 6,000 mg/L，以 CaCO <sub>3</sub> 计                                                                                                                                                                                           |
| 硝酸盐     | 最大允许含量 100 mg/L，以 NO <sub>3</sub> -N 计                                                                                                                                                                                            |
| 亚硝酸盐    | 最大允许含量 12 mg/L，以 NO <sub>2</sub> -N 计                                                                                                                                                                                             |
| 磷酸盐     | 最大允许含量 100 mg/L，以 PO <sub>4</sub> -P 计                                                                                                                                                                                            |
| 硫酸盐     | 最大允许含量 300 mg/L，以 SO <sub>4</sub> 计                                                                                                                                                                                               |
| 硫化物     | 硫化物会导致产生过深的颜色，可以按照以下步骤扣除硫化物的干扰： <ol style="list-style-type: none"> <li>1. 在 500mL 厄氏容量瓶中，加入 350mL 待测样品；</li> <li>2. 加入一份硫化物抑制试剂 (Hach #2418-99)，摇匀</li> <li>3. 用滤纸 (Hach #692-57) 过滤待测样品。</li> <li>4. 在步骤 3 时使用过滤后的样品。</li> </ol> |
| 浊度、颜色   | 会导致测量结果偏高。如果干扰过大，建议对水样先进行蒸馏，可以采用 HACH 公司的通用蒸馏用装置进行。                                                                                                                                                                               |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection. If chlorine is known to be present, the sample must be treated immediately with sodium thiosulfate. Add one drop of Sodium Thiosulfate Standard Solution, 0.1 N, for each 0.3 mg of chlorine present in a one liter sample.

To preserve the sample, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see *Correction for Volume Additions*, in *Section 1* for more detailed information.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL mixing cylinders with 20 mL of sample.
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Ammonium Nitrogen Standard, 10 mg/L as  $\text{NH}_3\text{-N}$  to the three samples. Stopper the cylinders and mix well.
- c) Analyze a 10-mL portion of sample as described above. The ammonia nitrogen concentration should increase 0.05 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Prepare a 0.40 mg/L ammonia nitrogen standard by diluting 4.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with deionized water. Or, using the TenSette Pipet, prepare a 0.40 mg/L ammonia nitrogen standard by diluting 0.8 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as  $\text{NH}_3\text{-N}$ , to 100 mL with deionized water.

## Method Performance

### Precision

In a single laboratory using a standard solution of 0.40 mg/L ammonia nitrogen ( $\text{NH}_3\text{-N}$ ) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of +0.02 mg/L ammonia nitrogen.

### Estimated Detection Limit

The estimated detection limit for program 64 is 0.02 mg/L  $\text{NH}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final greencolored solution.

**所需试剂和仪器 (使用试剂粉包)**

氨氮试剂 for 10-mL 样品 (100 tests) .....26680-00  
 包括: (2) 26531-99, (2) 26532-99

| 试剂种类                             | 所需数量<br>每次测试 | 单位            | 货号       |
|----------------------------------|--------------|---------------|----------|
| Ammonia Cyanurate 试剂粉包 .....     | 2 包 .....    | 100/pkg ..... | 26531-99 |
| Ammonia Salicylate 试剂粉包 .....    | 2 包 .....    | 100/pkg ..... | 26532-99 |
| 样品比色瓶, 10-20-25 mL, w/ cap ..... | 2 .....      | 6/pkg .....   | 24019-06 |

**OPTIONAL REAGENTS**

|                                                                              |                  |          |
|------------------------------------------------------------------------------|------------------|----------|
| Ammonia Nitrogen Standard Solution, 10 mg/L as NH <sub>3</sub> -N .....      | 500 mL .....     | 153-49   |
| Ammonia Nitrogen, PourRite Ampules, 50 mg/L as NH <sub>3</sub> -N, 2 mL..... | 20/pkg .....     | 14791-20 |
| Cylinder, graduated, mixing, 25 mL .....                                     | each .....       | 20886-40 |
| Sodium Hydroxide Standard Solution, 1.0 N.....                               | 100 mL MDB ..... | 1045-32  |
| Sodium Hydroxide Standard Solution, 5.0 N.....                               | 50 mL SCDB ..... | 2450-26  |
| Sodium Thiosulfate Standard Solution, 0.1 N.....                             | 100 mL MDB ..... | 323-32   |
| Sulfide Inhibitor Reagent Powder Pillows .....                               | 100/pkg .....    | 2418-99  |
| Sulfuric Acid, concentrated, ACS .....                                       | 500 mL .....     | 979-49   |
| Sulfuric Acid Standard Solution, 1.0 N.....                                  | 100 mL MDB ..... | 1270-32  |
| Water, deionized .....                                                       | 4 L .....        | 272-56   |

**OPTIONAL APPARATUS**

|                                                        |              |          |
|--------------------------------------------------------|--------------|----------|
| Cylinder, graduated, polypropylene, 500 mL.....        | each .....   | 1081-49  |
| Distillation Heater and Support Apparatus, 115 V ..... | each .....   | 22744-00 |
| Distillation Heater and Support Apparatus, 230 V ..... | each .....   | 22744-02 |
| Distillation Set, General Purpose .....                | each .....   | 22653-00 |
| Filter Paper, folded, 12.5 cm .....                    | 100 .....    | 1894-57  |
| Flask, Erlenmeyer, polypropylene, 500 mL .....         | each .....   | 1082-49  |
| Flask, volumetric, Class A, 100 mL.....                | each .....   | 14574-42 |
| Funnel, poly, 65 mm.....                               | each .....   | 1083-67  |
| pH Meter, <i>sensio</i> <b>1</b> , portable .....      | each .....   | 51700-10 |
| Pipet Filler, safety bulb.....                         | each .....   | 14651-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....                    | each .....   | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....          | 50/pkg ..... | 21856-96 |
| Pipet, volumetric, Class A, 2.0 mL .....               | each .....   | 14515-36 |
| PourRite Ampule Breaker Kit .....                      | each .....   | 24846-00 |
| Thermometer, -10 to 110 °C.....                        | each .....   | 1877-01  |

**For Technical Assistance, Price and Ordering**

In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 总氮 凯氏法 (0 to 150 mg/L)

方法号: 8075

Nessler 法 (需要消解处理)



1. 为得到最精确的结果，应具有用户可选的输入校正。详情见用户校正部分的内容。
2. 消解所需量的样品，同时消解相同数量的去离子水作为空白试样。
3. 输入测试总氮 凯氏法的程序编号。按下: PRGM  
屏幕将显示: PRGM?
4. 按下:  
85 ENTER  
屏幕将显示:  
mg/L, TKN和ZERO  
图标



5. 根据表一选择适量的消解后的样品，分别移取适量的样品和空白样品到两个的容积25mL的混合量筒中。
6. 分别将一滴 TKN 指示剂加入到每个量筒中。
7. 将 8.0 N KOH 加入到每个量筒中，每次一滴。直到第一次闪现蓝色。
8. 往两个量筒中都装入去离子水直至20mL刻度为止。然后分别加入3滴矿物质稳定剂到每个量筒中。反转多次使之混合。最后再分别加入3滴乙烯聚合物酒精分散剂到每个量筒中，反转多次使之混合。



9. 往两个量筒中都装入去离子水至 25mL 刻度。盖好盖子，混合。



10. 分别移取 1.00 mL Nessler's 试剂到量筒中，盖好盖子，混合。溶液不应浑浊。



11. 按下：  
**TIMER** **ENTER**  
将开始 2 分钟的反应计时。



12. 当计时器鸣响后，分别将试样注入到标有相应标签的 2 支比色瓶中。

*注：浑浊会引起错误结果。*



13. 将空白试样放入样品适配器中，盖紧遮光盖。



14. 按 **ZERO**，指针将右移，屏幕显示：  
**0 mg/L TKN**



15. 将预制试样放入样品适配器中。盖紧瓶盖。



16. 按下：**READ**  
指针将右移，屏幕会显示总凯式氮的读数，单位是 mg/L。

*注：应使用预制的氨水标准溶液进行标准校正。*

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

17. 根据公式计算氮的最终含量。 注：水样品的ppm TKN=mg/L TKN

A = mg/L 显示值

B = g (or mL水) 消解前样品

C = mL 消解后的样品分析体积

**Table 1 Analysis Volumes Based on Concentration**

| <b>AQUEOUS SAMPLES</b> (Solutions of suspensions in water- less than 1% solids) |                             |
|---------------------------------------------------------------------------------|-----------------------------|
| <b>Expected Nitrogen Concentration (mg/L)</b>                                   | <b>Analysis Volume (mL)</b> |
| 0.5-28                                                                          | 10.00                       |
| 2-112                                                                           | 5.00                        |
| 11-560                                                                          | 2.00                        |
| 45-2250                                                                         | 1.00                        |
| 425-22500                                                                       | 0.50                        |
| <b>DRY SAMPLES</b>                                                              |                             |
| <b>Expected Nitrogen Concentration (mg/L)</b>                                   | <b>Analysis Volume (mL)</b> |
| 42-2200                                                                         | 10.0                        |
| 106-5600                                                                        | 5.00                        |
| 350-18000                                                                       | 2.00                        |
| 1000-56000                                                                      | 1.00                        |
| 4200-220000                                                                     | 0.50                        |
| <b>OILS AND FATS</b>                                                            |                             |
| <b>Expected Nitrogen Concentration (mg/L)</b>                                   | <b>Analysis Volume (mL)</b> |
| 85-4500                                                                         | 10.0                        |
| 210-11000                                                                       | 5.00                        |
| 2100-11000                                                                      | 1.00                        |

## Sampling and Storage

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter) and cool to 4 °C. Preserved samples can be stored up to 28 days.

## Accuracy Check

### Kjeldahl Nitrogen Standard Method

This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the Accuracy Check section following the procedures in the Digesdahl Digestion Apparatus Instruction Manual. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within about ±3% of the value of the prepared Kjeldahl standard.

### Standard Solution Method (to check calibration accuracy only)

Add one drop of TKN Indicator to each of two 25-mL graduated mixing cylinders. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0 mg/L Ammonia Nitrogen Solution. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Perform the TKN procedure as described in Steps 9 to 16. This display should show 26-27 mg/L TKN.

### User Calibration

For most accurate results, use a user-calibrated program. The Standard Adjust feature should not be used with a user-entered calibration; it will hinder performance.

A one-time setup of a program for TKN is recommended for each new lot of reagents. A new calibration may be performed for each lot of Nessler Reagent by following these instructions:

### Standard Preparation

Use the following standards to make a calibration curve. See *Preparing a User-Entered Calibration Curve* on page 49, for more information and instructions. Prepare standards representing concentrations of 20, 60, 80, 100, 140 and 160 mg/L NH<sub>3</sub>-N as follows:

- a) Using volumetric pipets, transfer 5.0, 15.0, 20.0, 25.0, 35.0, and 40.0 mL of 100 mg/L NH<sub>3</sub>-N standard solution into six separate 100-mL volumetric flasks. Dilute to volume with deionized water, stopper, and invert to mix.
- b) Begin at step 4 of the procedure using a 3-mL aliquot for the sample volume. Also prepare a blank solution by substituting a 3 mL aliquot of deionized water for sample in Step 4.

**Note:** Standard solutions are prepared as if a 25-mL volume was used for the digestion. Actual concentrations prepared in Step 1 are 5, 15, 20, 25, 35, and 40 mg/L NH<sub>3</sub>-N. These represent original concentrations of 20, 60, 80, 100, 140, and 160 mg/L NH<sub>3</sub>-N, based on the 25 to 100 mL dilution in the digestion.

### User Entered Calibration Settings For TKN

Program # = 101 to 105

Wavelength = 420 nm

Resolution = 0 mg/L

## Method Performance

### Precision

In a single laboratory using a standard solution of 64 mg/L TKN and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 1.0$  mg/L TKN.

### Estimated Detection Limit

The estimated detection limit for program 65 is 2 mg/L TKN. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Total Kjeldahl Nitrogen (also called crude protein) refers to the combination of ammonia and organic nitrogen. Organically-bound nitrogen in the trinegative state, it is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified nessler method test. The Mineral Stabilizer complexes calcium and magnesium. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color forms, proportional to the ammonia concentration.

## Pollution Prevention And Waste Management

Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section 3 for more information on proper disposal of these materials.

## 所需试剂

凯氏总氮试剂一套 ..... 24953-00  
包括: (1) 21196-49, (1) 23766-26, (1) 21194-49, (1) 23765-26, (1) 282-32, (1) 23144-26, (1) 979-49, (1) 22519-26

| 试剂种类                 | 所需数量<br>每次测试 | 单位              | 货号       |
|----------------------|--------------|-----------------|----------|
| 过氧化氢, 50%.....       | 20 mL.....   | 490 mL.....     | 21196-49 |
| 矿物质稳定剂 .....         | 6 滴.....     | 50 mL SCDB..... | 23766-26 |
| Nessler's 试剂 t.....  | 2 mL.....    | 500 mL.....     | 21194-49 |
| Polyvinyl 酒精分散剂..... | 6 滴.....     | 50 mL SCDB..... | 23765-26 |
| 氢氧化钾标准溶液, 8.0 N..... | 不定 .....     | 100 mL MDB..... | 282-32   |
| 氢氧化钾标准溶液, 1.0 N..... | 不定 .....     | 50 mL SCDB..... | 23144-26 |
| 硫酸, ACS .....        | 6 mL.....    | 500 mL.....     | 979-49   |
| TKN 指示剂 .....        | 2 滴.....     | 50 mL SCDB..... | 22519-26 |
| 去离子水.....            | 不定 .....     | 4 L.....        | 272-56   |

## 所需仪器

|                                   |           |             |          |
|-----------------------------------|-----------|-------------|----------|
| Boiling Chips, 碳化硅.....           | 2-3 ..... | 500 g.....  | 20557-34 |
| 混合量筒, 25 mL.....                  | 2 .....   | 个 .....     | 21190-40 |
| 移液管, TenSette, 0.1 to 1.0 mL..... | 1 .....   | 个 .....     | 19700-01 |
| 移液管嘴, 19700-01 TenSette 移液管用..... | 2 .....   | 50/pkg..... | 21856-96 |

|                                 |        |            |          |
|---------------------------------|--------|------------|----------|
| 安全挡板 .....                      | 1..... | 个.....     | 20974-00 |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 2..... | 6/pkg..... | 24019-06 |
| <b>根据所用电压选择其一:</b>              |        |            |          |
| 消解处理装置, 115 V .....             | 1..... | 个.....     | 23130-20 |
| 消解处理装置, 230 V .....             | 1..... | 个.....     | 23130-21 |

## OPTIONAL REAGENTS

|                                                                                                  |             |          |
|--------------------------------------------------------------------------------------------------|-------------|----------|
| Ammonia Nitrogen Standard Solution, 1 mg/L NH <sub>3</sub> -N.....                               | 500 mL..... | 1891-49  |
| Ammonia Nitrogen Standard Solution, Voluette Ampule,<br>150 mg/L NH <sub>3</sub> -N, 10 mL ..... | 16/pkg..... | 21284-10 |
| Ammonia Nitrogen Standard Solution, 100 mg/L NH <sub>3</sub> -N.....                             | 500 mL..... | 24065-49 |

## OPTIONAL APPARATUS

| Description                                | Unit              | Cat. No. |
|--------------------------------------------|-------------------|----------|
| Ampule Breaker Kit .....                   | each .....        | 21968-00 |
| Balance, AccuLab Pocket Pro.....           | each .....        | 25568-00 |
| Bottle, glass dispenser, 118 mL.....       | each .....        | 591-00   |
| Bottle, plastic wash, 1000 mL.....         | each .....        | 620-16   |
| Cylinder, graduated, 50 mL.....            | each .....        | 508-41   |
| Flask, volumetric, 100 mL, Class A.....    | each .....        | 14574-42 |
| Mini Grinder, 120 V.....                   | each .....        | 20991-00 |
| pH Paper, 1 to 11 pH units .....           | 5 rolls/pkg ..... | 391-33   |
| Pipet, volumetric, Class A, 0.50 mL .....  | each .....        | 14515-34 |
| Pipet, volumetric, Class A, 1.00 mL .....  | each .....        | 14515-35 |
| Pipet, volumetric, Class A, 2.00 mL .....  | each .....        | 14515-36 |
| Pipet, volumetric, Class A, 5.00 mL .....  | each .....        | 14515-37 |
| Pipet, volumetric, Class A, 10.00 mL ..... | each .....        | 14515-38 |
| Pipet, volumetric, Class A, 15.00 mL ..... | each .....        | 14515-39 |
| Pipet, volumetric, Class A, 20.00 mL ..... | each .....        | 14515-20 |
| Pipet, volumetric, Class A, 25.00 mL ..... | each .....        | 14515-40 |
| Safety Glasses .....                       | each .....        | 18421-00 |

### *For Technical Assistance, Price and Ordering*

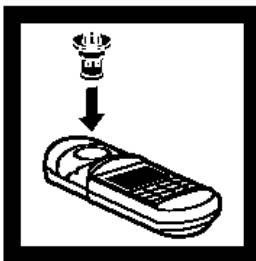
In the U.S.A.:<sup>a</sup>Call 800-227-4224

Outside the U.S.A.:<sup>a</sup>Contact the Hach office or distributor serving you.

# 氨氮 水杨酸法 低量程 (0 to 2.50 mg/L NH3-N)

方法号: 10023

Test 'N Tube



1. 输入检测低量程氨氮Test 'N Tube的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

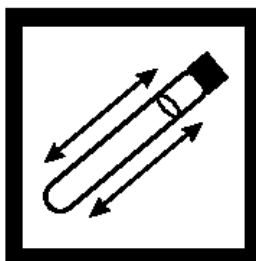
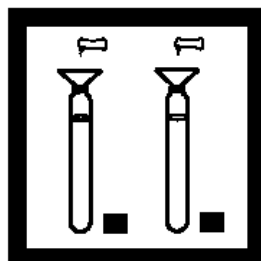
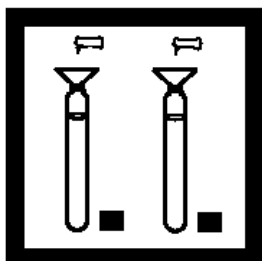
2. 按下: 66 ENTER  
屏幕将显示:

0.00 mg/L、NO3-N  
和ZERO图标。

3. 旋转 COD/TNT 适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。

4. 打开两支 AmVer 稀释液瓶的瓶盖。往其中一支加入2毫升的样品。(预制试样) 往另外一支加入2毫升的去离子水。(空白试样)

注: 分析前应调节所存样品的 PH 值。



5. 使用漏斗往每个瓶中分别加入氨水杨酸试剂粉。

6. 使用漏斗往每个瓶中分别加入氨氰尿酸试剂粉

7. 盖紧瓶盖。大力摇晃是粉末完全溶解。

8. 按下:

TIMER ENTER

将开始 20 分钟的反应计时。

注: 如果氨存在, 溶液将呈现绿色。



9. 用布擦干瓶外壁。 10. 按 **ZERO**, 指针将  
计时器鸣响后, 将空 右移, 屏幕显示:  
白试样放入样品适配 **0.00 mg/L NH<sub>3</sub>-N**  
器中, 盖紧遮光盖。 11. 将预制试样瓶放  
入样品适配器中。盖  
紧瓶盖。 12. 按下: **READ**  
指针将右移, 屏幕会  
显示总氨氮的读数,  
单位是 mg/L。

注: 应使用预制的标  
准溶液进行标准校  
正。

## 干扰

| 干扰物质名称 | 干扰物质最大允许含量及消除干扰的办法                                                                                                                                                   |
|--------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 钙      | 2500 mg/L                                                                                                                                                            |
| 铁      | 1. 测量水样中总铁的含量<br>2. 在步骤 4 加入相同铁离子浓度到去离子水中。铁离子干<br>扰就会完全的清除。                                                                                                          |
| 镁      | 5000 mg/L                                                                                                                                                            |
| 硝酸盐    | 250 mg/L, 以 NO <sub>3</sub> -N 计                                                                                                                                     |
| 亚硝酸盐   | 30 mg/L, 以 NO <sub>2</sub> -N 计                                                                                                                                      |
| 磷酸盐    | 2500 mg/L, 以 PO <sub>4</sub> -P 计                                                                                                                                    |
| PH     | 应将酸性或碱性的样品的 PH 值调到 7 左右。使用 1N 氢氧<br>化钠标准溶液调节酸性样品, 使用 1N 盐酸标准溶液调节碱<br>性样品。                                                                                            |
| 硫酸盐    | 300 mg/L, 以 SO <sub>4</sub> 计                                                                                                                                        |
| 硫化物    | 硫化物会导致产生过深的颜色, 可以按照以下步骤扣除硫<br>化物的干扰:<br>3. 在 500mL 厄氏容量瓶中, 加入 350mL 待测样品;<br>4. 加入一份硫化物抑制试剂(Hach #2418-99), 摇匀<br>3. 用滤纸(Hach #692-57)过滤待测样品。<br>4. 在步骤 3 时使用过滤后的样品。 |
| 其他     | 一些不常见的干扰物质, 如联氨或氨基乙酸, 将会导致预<br>制溶液的颜色加深。较大的浊度和色度将导致结果偏高。<br>含有大量干扰物质的样品应先使用蒸馏处理。                                                                                     |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L  $\text{Cl}_2$  in a one liter sample.

Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions. See *Correcting for Volume Additions on page 22* for more information.

## Accuracy Check

### Standard Additions Method

- a) Snap the neck off a Nitrogen, Ammonia Ampule Standard Solution, 50 mg/L  $\text{NH}_3\text{-N}$ .
- b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25 mL samples. Mix thoroughly.
- c) Analyze each sample as described above. The nitrogen concentration should increase 0.20 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions, Section 1*, for more information.

### Standard Solution Method

To check accuracy, use a 1.0 mg/L Nitrogen, Ammonia Standard Solution listed under Optional Reagents. Or, dilute 1 mL of solution from a 50 mg/L Ampule Standard for Nitrogen, Ammonia to 50 mL with deionized water using a 50-mL volumetric flask.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 1.0 mg/L ammonia nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of +0.02 mg/L  $\text{NH}_3\text{-N}$ .

### Estimated Detection Limit

The estimated detection limit for program 66 is 0.08 mg/L  $\text{NH}_3\text{-N}$ . For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

## Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferrocyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

## 所需试剂

AmVer 氨氮试剂一套 (25 tests).....26045-45

包括: (1) 23952-66, (1) 23954-66, (1) 272-42, \*(50) AmVer Low Range Vials

| 试剂种类                                               | 所需数量<br>每次测试 | 单位          | 货号       |
|----------------------------------------------------|--------------|-------------|----------|
| AmVer 稀释溶液, Test i®N Tube .....                    | 2 瓶.....     | 50/pkg..... | *        |
| 水杨酸盐试剂粉包, 5 mL sample.....                         | 2 包.....     | 50/pkg..... | 23952-66 |
| Cyanurate Reagent Powder Pillows, 5 mL sample .... | 2 包.....     | 50/pkg..... | 23954-66 |

## 所需仪器

|                             |           |             |          |
|-----------------------------|-----------|-------------|----------|
| COD适配器.....                 | 1.....    | 个.....      | 48464-00 |
| 试管架.....                    | 1-3 ..... | 个.....      | 18641-00 |
| TenSette 移液管, 0-10 mL ..... | 1.....    | 个.....      | 19700-10 |
| 移液管嘴 .....                  | 2.....    | 50/pkg..... | 21997-96 |
| 漏斗.....                     | 1.....    | 个.....      | 25843-35 |

## OPTIONAL REAGENTS

Nitrogen, Ammonia Standard Solution, 1.0 mg/L NH<sub>3</sub>-N ..... 500 mL .....1891-49

Nitrogen, Ammonia Standard Solution, 10 mL

Voluette ampules, 50 mg/L NH<sub>3</sub>-N.....16/pkg .....14791-10

Nitrogen, Ammonia Standard Solution, 2 mL

PourRite ampules, 50 mg/L NH<sub>3</sub>-N.....20/pkg .....14791-20

Hydrochloric Acid, ACS ..... 500 mL .....134-49

Sodium Hydroxide Standard Solution, 5.0 N.....50 mL SCDB ..... 2450-26

Sodium Hydroxide, 1.000 N ..... 100 mL MDB .....1045-32

Sodium Thiosulfate Standard Solution, 0.1 N..... 100 mL MDB .....323-32

Sulfide Inhibitor Reagent Powder Pillows .....100/pkg .....2418-99

Sulfuric Acid, 1.00 N ..... 100 mL MDB .....1270-32

Water, deionized ..... 4 L .....272-56

## OPTIONAL APPARATUS

Ampule Breaker Kit .....each .....21968-00

Cylinder, graduated, mixing, 25 mL, Class A.....each .....508-40

Distillation Apparatus Set .....each .....22653-00

Heater and Support Apparatus (for distillation), 115 Vac.....each .....22744-00

Heater and Support Apparatus (for distillation), 230 Vac.....each .....22744-02

Filter Paper, folded .....100/box .....1894-57

Flask, Erlenmeyer, 500 mL .....each .....505-49

Flask, volumetric, 50 mL, Class A.....each .....14547-41

Funnel, analytical (for filtering) .....each .....1083-68

Jack, laboratory (use with distillation apparatus).....each .....22743-00

pH Indicator Paper, 1 to 11 pH.....5 rolls/pkg .....391-33

Ampule Breaker Kit, PourRite .....each .....24846-00

Thermometer, -20 to 110 i°C.....each .....566-01

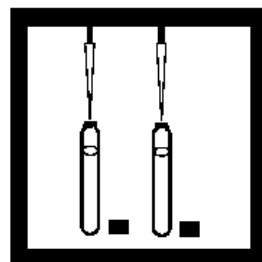
Thermometer, -10 to 260 i°C.....each .....26357-01

**For Technical Assistance, Price and Ordering**

# 氨氮 水杨酸法 低量程 (0 to 50 mg/L NH<sub>3</sub>-N)

方法号: 10031

Test 'N Tube



1. 输入检测高量程氨氮Test 'N Tube的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: 67 ENTER

屏幕将显示:

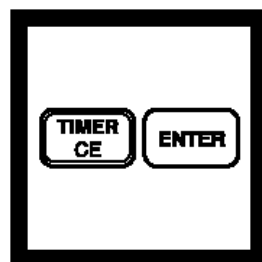
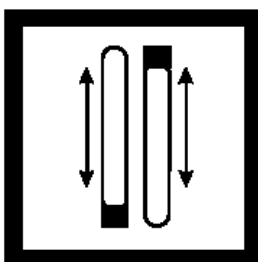
0.00 mg/L、NO<sub>3</sub>-N 和ZERO图标。

注: 如检测其他形态的氮(NH<sub>3</sub>),

按下: CONC

3. 旋转 COD/TNT 适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。

4. 打开两支 AmVer 稀释液瓶的瓶盖。往其中一支加入0.1毫升的样品。(预制试样) 往另外一支加入0.1毫升的去离子水。(空白试样)



5. 往每个瓶中分别加入氨水杨酸试剂粉。

6. 往每个瓶中分别加入氨氰尿酸试剂粉

7. 盖紧瓶盖。大力摇晃是粉末完全溶解。

8. 按下: TIMER ENTER  
将开始 20 分钟的反应计时。

注: 如果氮存在, 溶液将呈现绿色。



9. 用布擦干瓶外壁。  
计时器鸣响后，将空白试样放入样品适配器中，盖紧遮光盖。

10. 按 **ZERO**，指针将右移，屏幕显示：  
**0.00 mg/L NH<sub>3</sub>-N**

11. 将预制试样瓶放入样品适配器中。盖紧瓶盖。

12. 盖紧瓶盖。



13. 按下：**READ**  
指针将右移，屏幕会显示总氨氮的读数，单位是 mg/L。

*注：应使用预制的标准溶液进行标准校正。*

## 干扰

| 干扰物质名称  | 干扰物质最大允许含量及消除干扰的办法                                                                                                              |
|---------|---------------------------------------------------------------------------------------------------------------------------------|
| 酸或者碱性样品 | 将水样的 pH 值调节至中性：对于酸性水样，加入 1N 的氢氧化钠；对于碱性水样，加入 1N 的盐酸                                                                              |
| 钙       | 最大允许含量 50,000 mg/L，以 $\text{CaCO}_3$ 计                                                                                          |
| 氨基乙酸、联胺 | 会导致被测水样的颜色加深                                                                                                                    |
| 镁       | 最大允许含量 300,000 mg/L，以 $\text{CaCO}_3$ 计                                                                                         |
| 铁       | 可以按照以下步骤扣除铁的干扰：<br>3. 测量水样中总铁的含量<br>4. 在第 4 步操作之前，在空白溶液中加入同样浓度的铁                                                                |
| 亚硝酸盐    | 最大允许含量 600 mg/L，以 $\text{NO}_2\text{-N}$ 计                                                                                      |
| 硝酸盐     | 最大允许含量 5,000 mg/L，以 $\text{NO}_3\text{-N}$ 计                                                                                    |
| 正磷      | 最大允许含量 5,000 mg/L，以 $\text{PO}_4\text{-P}$ 计                                                                                    |
| 硫酸盐     | 最大允许含量 5,000 mg/L，以 $\text{SO}_4$ 计                                                                                             |
| 硫化物     | 硫化物会导致产生过深的颜色，可以按照以下步骤扣除硫化物的干扰：<br>5. 在 500mL 厄氏容量瓶中，加入 350mL 待测水样；<br>6. 加入一份硫化物抑制试剂(Hach #2418-99)，摇匀用滤纸(Hach #692-57)过滤待测水样， |
| 浊度、颜色   | 会导致测量结果偏高。如果干扰过大，建议对水样先进行蒸馏，可以采用 HACH 公司的通用蒸馏用装置。                                                                               |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L  $\text{Cl}_2$  in a one liter sample.

Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions.

## Accuracy Check

### Standard Additions Method

- Snap the top off an Ammonia PourRite Ampule Standard, 150 mg/L  $\text{NH}_3\text{-N}$ .
- Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 25-mL samples. Swirl to mix.
- Analyze each sample as described above. The ammonia concentration should increase approximately 1.2 mg/L  $\text{NH}_3\text{-N}$  for each 0.2 mL of standard added.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To check accuracy, use a 10 or 50 mg/L Nitrogen, Ammonia Standard Solution or use a Nitrogen, Ammonia Voluette Ampule Standard, 50 mg/L.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 50 mg/L ammonia nitrogen (NH<sub>3</sub>-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of +5 mg/L NH<sub>3</sub>-N.

### Estimated Detection Limit

The estimated detection limit for program 67 is 1 mg/L NH<sub>3</sub>-N. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a green-colored solution.

## Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheets* for information specific to the reagents used. For additional information, refer to *Section 3*.

## Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferrocyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

## 所需试剂

AmVer 氨氮试剂一套, TNT (25 tests) .....26069-45

包括: (1) 23952-66, (1) 23954-66, (1) 272-42, \*(50) AmVer HR Vials

| 试剂种类                               | 所需数量<br>每次测试 | 单位           | 货号       |
|------------------------------------|--------------|--------------|----------|
| AmVer. HR Test i®N Tube. 试剂瓶 ..... | 2 瓶 .....    | 50/pkg ..... | *        |
| 氨水水杨酸盐粉末试剂 .....                   | 2 包 .....    | 50/pkg ..... | 23952-66 |
| 氨水氰尿酸盐粉末试剂 .....                   | 2 包 .....    | 50/pkg ..... | 23954-66 |

## 所需仪器

|                             |              |             |          |
|-----------------------------|--------------|-------------|----------|
| COD/TNT 适配器.....            | 1 .....      | 个 .....     | 48464-00 |
| TenSette. 移液管, 0-1 mL ..... | 1 .....      | 个 .....     | 19700-01 |
| 移液管嘴.....                   | varies ..... | 50/pkg..... | 21856-96 |
| 试管架 .....                   | 1-3 .....    | 个 .....     | 18641-00 |
| 漏斗 .....                    | 1 .....      | 个 .....     | 25843-35 |

## OPTIONAL REAGENTS

|                                                                                         |              |          |
|-----------------------------------------------------------------------------------------|--------------|----------|
| Nitrogen, Ammonia Standard Solution, 50 mg/L NH <sub>3</sub> -N .....                   | 500 mL ..... | 14791-50 |
| Nitrogen, Ammonia Standard Solution, 10 mg/L NH <sub>3</sub> -N .....                   | 500 mL ..... | 153-49   |
| Ammonia Standard Solution, PourRite ampules,<br>150 mg/L NH <sub>3</sub> -N, 2 mL ..... | 20/pkg ..... | 21284-20 |
| Hydrochloric Acid, ACS .....                                                            | 500 mL ..... | 134-49   |
| Sodium Hydroxide Standard Solution, 5.0 N.....                                          | 50 mL .....  | 2450-26  |
| Sodium Hydroxide Standard Solution, 1.0 N.....                                          | 100 mL ..... | 1045-32  |
| Sodium Thiosulfate Standard Solution, 0.1 N.....                                        | 100 mL ..... | 323-32   |

## OPTIONAL REAGENTS (continued)

### Quantity Required

| Description                            | Per Test | Unit            | Cat. No. |
|----------------------------------------|----------|-----------------|----------|
| Sulfide Inhibitor Powder Pillows ..... |          | 100/pkg.....    | 2418-99  |
| Sulfuric Acid, 1.00 N.....             |          | 100 mL MDB..... | 1270-32  |
| Water, deionized.....                  |          | 4 L .....       | 272-56   |

## OPTIONAL APPARATUS

|                                                               |                  |          |
|---------------------------------------------------------------|------------------|----------|
| Cylinder, 25 mL, graduated, mixing.....                       | each.....        | 20886-40 |
| Distillation Apparatus Set, general purpose .....             | each.....        | 22653-00 |
| Heater and Support Apparatus (for distillation), 115 VAC..... | each.....        | 22744-00 |
| Heater and Support Apparatus (for distillation), 230 VAC..... | each.....        | 22744-02 |
| Filter Paper, folded.....                                     | 100/pkg.....     | 1894-57  |
| Flask, Erlenmeyer, 500 mL.....                                | each.....        | 505-49   |
| Funnel, analytical (for filtering).....                       | each.....        | 1083-68  |
| Jack, laboratory (use with distillation apparatus) .....      | each.....        | 22743-00 |
| pH Indicator Paper, 1 to 11 pH .....                          | 5 rolls/pkg..... | 391-33   |
| PourRite. Ampule Breaker.....                                 | each.....        | 24846-00 |
| Sample Cell, 10-20-25 mL, w/cap.....                          | 6/pkg.....       | 24019-06 |

### *For Technical Assistance, Price and Ordering*

In the U.S.A.;"Call 800-227-4224

Outside the U.S.A.;"Contact the Hach office or distributor serving you.

# 总无机氮 三氯化钛还原法 (0 to 25.0 mg/L N) 方法号: 10021

Test 'N Tube™ 需要离心



1. 输入检测总无机氮  
Test 'N Tube的程  
序编号。  
按下: PRGM  
屏幕将显示:  
PRGM?
2. 按下: 68 ENTER  
屏幕将显示:  
0.00 mg/L、N  
和ZERO图标。
3. 旋转 COD/TNT 适配  
器, 将其嵌入瓶管架  
上适当的位置, 然后  
下按使之完全嵌入。
4. 分别移取1 mL 总  
无机氮预处理碱式浓  
缩液到两个总无机氮  
预处理稀释瓶中。



5. 移取 1 mL 样品到  
一个 TIN 稀释瓶中  
(样品试剂), 移取  
1mL 去离子水到另一  
瓶中(空白试样)。  
盖好瓶盖, 摇晃 30  
秒使之混合。
6. 将两个总无机氮还  
原剂安培瓶中溶液分  
别注入到样品试样和  
空白试样的TIN稀释  
瓶中。  
*注: 为了安全, 打开  
安培瓶时戴上手套。  
注: 立即会形成黑色  
沉淀。*
7. 盖上瓶盖, 轻轻摇  
晃30秒使试剂混合。  
让瓶静置至少一分  
钟。  
*注: 摇晃后沉淀应依  
然是黑色, 过分摇晃  
会生成白色沉淀, 结  
果偏低。*
8. 把试剂瓶放入离心  
机内处理3分钟, 直至  
固体沉淀到瓶底。  
离心处理后马上按  
下:  
TIMER ENTER  
*注: 如不使用离心机,  
沉淀物也会下沉, 但  
是要费时 30 分钟。*



9. 打开两支 AmVer 稀释液瓶的瓶盖。使用移液管，往其中一支加入2毫升的离心处理过的样品。（预制试样）往另外一支加入2毫升的去离子水。（空白试样）

*注：移液时应小心，不要搅动了沉淀物*



10. 使用漏斗往每个瓶中分别加入氨水杨酸试剂粉。



11. 使用漏斗往每个瓶中分别加入氨氰尿酸试剂粉。



12. 盖紧瓶盖。大力摇晃是粉末完全溶解。

*注：如果氨存在，溶液将呈现绿色。*



13. 屏幕将显示：  
**15:00 TIMER 2**  
按下：**ENTER**  
将开始 15 分钟的反应计时。



14. 计时器鸣响后，用布擦干瓶壁。将空白试样放入样品适配器中。



15. 盖紧瓶盖。



16. 按下：**ZERO**  
指针将右移，屏幕将显示：  
**0.0 mg/L N**



17. 将预制试样放入  
样品适配器中。

18. 盖紧瓶盖。

19. 按下：READ  
指针将右移，屏幕将  
显示总无机氮的含  
量，单位是 mg/L。

*注：应使用预制试样  
进行试剂空白校正。*

## 干扰

当含量超高下列情况时，可能产生干扰。

| 种类  | 水平        | 效果 |
|-----|-----------|----|
| 钙   | 1000mg/L  | 正  |
| 锰   | 3 mg/L    | 负  |
| 镁   | 1000 mg/L | 正  |
| 硫化物 | 3 mg/L    | 负  |
| 硫酸盐 | 250 mg/L  | 负  |

当含量低于下列情况，不产生干扰。

| Species                     | Level   |
|-----------------------------|---------|
| $\text{Al}^{3+}$            | 8 mg/L  |
| $\text{Ba}^{2+}$            | 40 mg/L |
| $\text{Cu}^{2+}$            | 40 mg/L |
| $\text{Fe}^{3+}$            | 8 mg/L  |
| $\text{Zn}^{2+}$            | 80 mg/L |
| $\text{F}^-$                | 40 mg/L |
| $\text{PO}_4^{3-}\text{-P}$ | 8 mg/L  |
| $\text{SiO}_2$              | 80 mg/L |
| EDTA                        | 80 mg/L |

## Sampling And Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add 1 drop of 0.1 N sodium thiosulfate for each 0.3 mg/L  $\text{Cl}_2$  in a 1 liter sample.

Preserve the sample by reducing the pH to 2 or less with concentrated hydrochloric acid (at least 2 mL). Store at 4 ° C (39 ° F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a fresh High Range Nitrate Nitrogen PourRite Ampule Standard, 500 mg/L  $\text{NO}_3\text{-N}$ .
- c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to 3 25-mL mixing cylinders. Mix thoroughly.
- d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the prepared sample in Step 5. The nitrogen concentration should increase about 1.8 to 1.9 mg/L for each 0.1 mL of standard added.
- e) If these increases do not occur, see *Standard Additions* in Section 1 for more information.

### Standard Solution Method

To check accuracy, use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional Reagents. Alternatively, a 20.0 mg/L nitrate nitrogen standard can be prepared by diluting 2 mL of solution from a PourRite Ampule Standard for High Range Nitrate Nitrogen, 500 mg/L  $\text{NO}_3\text{-N}$ , to 50 mL with deionized water. Substitute this standard for the sample and perform the test as described. The recovery of the standards should be about 90-95%.

## Method Performance

### Precision/Accuracy

The total inorganic nitrogen test provides an estimate of the total nitrite, nitrate, and ammonia nitrogen load in water or wastewater samples. This test is most applicable for monitoring an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test exhibits different recoveries of each of the three nitrogen species, as summarized below. This test is not recommended for quantifying only one of the three species. In that case, use a specific procedure for each particular analyte.

### Ammonia Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L  $\text{NH}_3\text{-N}$  and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 21.3 mg/L with a standard deviation of  $\pm 0.77$  mg/L N (replicate number = 7 per reagent lot).

### Nitrate Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO<sub>3</sub>-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 18.9 mg/L with a standard deviation of  $\pm 0.55$  mg/L N (replicate number = 7 per reagent lot).

### Nitrite Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO<sub>2</sub>-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 14.6 mg/L with a standard deviation of  $\pm 0.77$  mg/L N (replicate number = 7 per reagent lot).

### Estimated Detection Limit

The estimated detection limit for program 68 is 0.7 mg/L N. For more information on the estimated detection limit, see Section 1.

### Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

### REQUIRED REAGENTS

总无机氮预处理试剂一套 (TiCl<sub>3</sub> 还原作用) (25 tests) ..... 26049-45

包括: (1) 26051-50, (1) 2040-59, \*(50) TIN Pretreatment Diluent Vials

AmVer. 氨氮试剂 (25 tests) 26045-45

包括: (1) 23952-66, (1) 23954-66, (1) 272-42, \*(50) AmVer. Diluent LR Vials

| 试剂种类        | 所需数量 |     | 单位     | 货号       | 总 |
|-------------|------|-----|--------|----------|---|
|             | 每次测试 |     |        |          |   |
| 无机氮预处理稀释瓶   | 2    | 瓶   | 50/pkg |          | * |
| 无机氮还原剂安瓿瓶   | 2    | 安瓿瓶 | 50/pkg | 26051-50 |   |
| 无机氮预处理浓缩液   | 2    | mL  | 50 mL  | 2040-59  |   |
| AmVer. 稀释试剂 | 2    | 瓶   | 50/pkg |          | * |
| 氨水水杨酸盐粉末试剂  | 2    | 包   | 50/pkg | 23952-66 |   |
| 氨水氰尿酸盐粉末试剂  | 2    | 包   | 50/pkg | 23954-66 |   |

### REQUIRED APPARATUS

|                               |   |        |       |          |
|-------------------------------|---|--------|-------|----------|
| 离心分离机, 115V .....             | 1 | 个      | ..... | 26765-00 |
| 离心分离机, 230V .....             | 1 | 个      | ..... | 26765-02 |
| COD/TNT 适配器 .....             | 1 | 个      | ..... | 48464-00 |
| 漏斗 .....                      | 1 | 个      | ..... | 25843-35 |
| TenSette移液管, 0.1 to 1.0 ..... | 1 | 个      | ..... | 19700-01 |
| 移液管嘴 .....                    | 2 | 50/pkg | ..... | 21856-96 |
| 试管架 .....                     | 1 | 个      | ..... | 18641-00 |

## OPTIONAL REAGENTS

|                                                                                                  |                 |          |
|--------------------------------------------------------------------------------------------------|-----------------|----------|
| Hydrochloric Acid, ACS.....                                                                      | 500 mL.....     | 134-49   |
| Nitrate Nitrogen Standard Solution, 10 mg/L NO <sub>3</sub><br>-N.....                           | 500 mL.....     | 307-49   |
| Nitrate Nitrogen Standard Solution, PourRite Ampules,<br>500 mg/L NO <sub>3</sub> -N, 2 mL ..... | 20/pkg.....     | 14260-20 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                  | 50 mL SCDB..... | 2450-26  |
| Sodium Thiosulfate Standard Solution, 0.1 N.....                                                 | 100 mL MDB..... | 323-32   |
| Water, deionized.....                                                                            | 4 L.....        | 272-56   |

## OPTIONAL APPARATUS

|                                           |                  |          |
|-------------------------------------------|------------------|----------|
| Cylinder, graduated, mixing, 25 mL.....   | each.....        | 20886-40 |
| Flask, volumetric, Class A, 50.0 mL ..... | each.....        | 14574-41 |
| pH Indicator Paper, 1 to 11 pH .....      | 5 rolls/pkg..... | 391-33   |
| Pipet, volumetric, Class A, 2.0 mL.....   | each.....        | 14515-36 |
| PourRite Ampule Breaker .....             | each.....        | 24846-00 |

### ***For Technical Assistance, Price and Ordering***

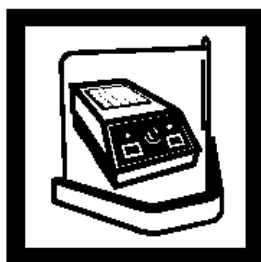
**In the U.S.A.;**\*Call 800-227-4224

**Outside the U.S.A.;**\*Contact the Hach office or distributor serving you.

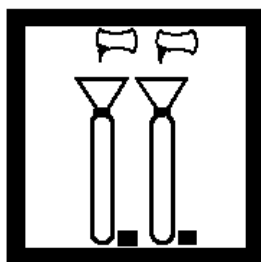
# 总氮 TNT过硫酸盐消解法 (0.0 to 25.0 mg/L N)

方法号: 10071

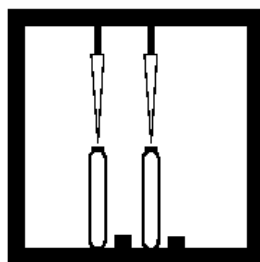
Test 'N Tube



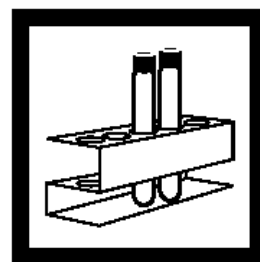
1. 打开COD反应器。  
加热到 103-106 °C。  
(最佳温度 105 度)



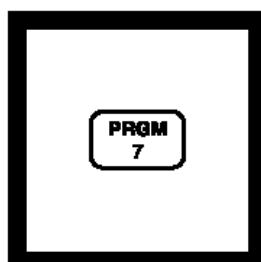
2. 使用漏斗, 将总氮  
过硫酸盐试剂粉加入  
到两支总氮氢氧化物  
试剂瓶中。



3. 往一支瓶中注入 2  
毫升样品。再往另外  
一支加入 2 毫升不含  
有机物的水。盖上瓶  
盖。大力摇晃。(约  
30 秒) 将瓶放在反应  
器上加热 30 分钟。



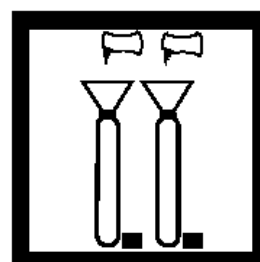
4. 将试剂瓶从反应器  
拿下, 冷却到室温。  
*注: 在加热30分钟拿  
开试剂瓶是十分重要  
的。*



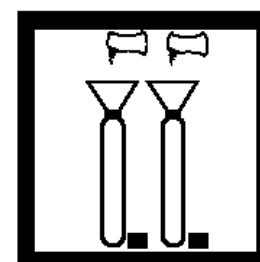
5. 输入检测总无机氮  
**Test 'N Tube**的程  
序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**



6. 按下: **58 ENTER**  
屏幕将显示:  
0.00 mg/L、**N**  
和**ZERO**图标。



7. 打开消解瓶盖。往  
两支瓶中加入 TA 试  
剂 A 粉剂。盖上瓶盖  
摇晃 15 秒钟。  
按下: **TIMER ENTER**  
将开始 3 分钟的反应  
计时。



8. 计时器鸣响后, 打  
开瓶盖往两支瓶中加  
入TA试剂B粉剂。盖  
上瓶盖摇晃15秒钟。  
屏幕将显示:  
**02:00 Timer 2**  
按下: **ENTER**  
将开始两分钟的反应  
计时。

*注: 试剂粉不会完全  
溶解。溶液将变成黄  
色。*



9. 反应器鸣响后，打开两支 TN 试剂 C 瓶，加入 2 毫升消解处理过的样品到其中一瓶中。将 2 毫升消解处理过的空白试样加到另外一瓶中。



10. 盖上瓶盖，反转十次使之混合。反转动作要缓慢振动。试剂瓶将会变暖。



11. 屏幕将显示：  
**05:00 Timer 3**  
按：**ENTER**  
将开始 5 分钟的反应计时。

注：黄色将会加深。



12. 在反应期间，将 COD/TNT 适配器放入。



13. 计时器鸣响后，擦干空白试剂瓶，将其放入样品适配器中。



14. 盖紧瓶盖。按下：**ZERO**  
指针将右移，屏幕显示：**0.0 mg/L N**



15. 擦干净瓶壁。



16. 按下：**READ**  
指针将右移，屏幕将显示总氮的含量。单位是 mg/L。

注：使用预制标准溶液进行标准校正。

注：如果屏幕闪烁显示“Limit”，稀释样品，重复消解处理，完成比色测试。如果要得到精确结果，应重复进行消解处理。稀释和比色测试不会影响最终结果。最终结果应是读数乘上稀释系数。

## 干扰

将会使所测含量改变10%的干扰物质

| 物质           | 水平和影响 |
|--------------|-------|
| 溴化物>60ppm    | 正干扰   |
| 氯化物>1000 ppm | 正干扰   |

下表中的物质经过测试，发现在所示水平范围内不干扰（mg/L）：

| 物质    | 测试最大的水平（mg/L） |
|-------|---------------|
| 钡     | 2.6           |
| 钙     | 300           |
| 铬(3+) | 0.5           |
| 铁     | 2             |
| 铅     | 6.6 ppb       |
| 镁     | 500           |
| 有机碳   | 150           |
| pH    | 13 pH单位       |
| 磷     | 100           |
| 硅     | 150           |
| 银     | 0.9           |
| 锡     | 1.5           |

Hach chemists tested this chemistry on standard nitrogen solutions prepared from the following compounds and obtained  $\geq 95\%$  recovery:

- . Ammonium chloride
- . Ammonium sulfate
- . Ammonium acetate
- . Urea
- . Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in  $\geq 95\%$  recovery. Large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

### Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

### Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

a) Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

b) Weigh 0.4416 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

c) Weigh 0.5274 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above.

Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \times 100$$

The percent recovery should be:

| Compound       | Lowest Expected % Recovery |
|----------------|----------------------------|
| Ammonia-PTSA   | 95%                        |
| Glycine-PTSA   | 95%                        |
| Nicotinic-PTSA | 95%                        |

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

### Standard Solution Method

Substitute 2 mL of a 20 mg/L ammonia nitrogen standard solution for the sample. To prepare a 20-mg/L standard, use a 20-mL Class A pipet to transfer 20 mL of a 100-mg/L Ammonia Nitrogen Standard (see *Optional Reagents*) to a 100-mL Class A volumetric flask. Dilute to the line with organic-free water. A single analyst should obtain less than 5% variation on replicates. Comparison of the user-obtained value with the standard concentration is an indication of test performance for this user.

### Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off an Ammonia Nitrogen Voluette Ampule Standard Solution, 160 mg/L as  $\text{NH}_3\text{-N}$ .

c) Use the TenSette Pipet to add 0.3 mL, 0.6 mL, and 0.9 mL of standard, respectively, to the three mixing cylinders.

d) Stopper each cylinder and mix thoroughly.

e) Add 2 mL of each prepared solution, respectively, to three TN Hydroxide Reagent Sample Digestion Vials.

- f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 2 mg/L for each 0.3 mL of standard added.
- g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

#### Blanks for Colorimetric Measurement

The reagent blank may be used up to 7 days for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18-25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

### Method Performance

#### Precision

A Hach chemist analyzed two independent nutrient standards. The lowest average percent recovery was 95% with a standard deviation of  $\pm 2\%$ .

In a single laboratory, using a standard solution of 15.0 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than  $\pm 0.5$  mg/L N. For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for program 58 is 2 mg/L N. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

### REQUIRED REAGENTS

| Description                                         | Cat. No. |
|-----------------------------------------------------|----------|
| Test $\text{NH}_4^+$ N Tube 总氮试剂一套 (100 vials)..... | 26722-45 |
| 包括:                                                 |          |
| TN Reagent C 瓶, 酸溶液*, 25/pkg.....                   | 26721-25 |
| TN氢氧化物试剂样品消解瓶*, 25/pkg.....                         | 26717-25 |

| 试剂种类                         | 所需数量 |         | 单位           | 货号TN     |
|------------------------------|------|---------|--------------|----------|
|                              | 每次测试 |         |              |          |
| 过(二)硫酸盐粉末试剂.....             | 2    | 包.....  | 100/pkg..... | 26718-49 |
| TN Reagent A, 二硫化物粉末试剂 ..... | 2    | 包 ..... | 100/pkg..... | 26719-49 |
| TN Reagent B, 指示剂粉末试剂.....   | 2    | 包 ..... | 100/pkg..... | 26720-49 |

#### 所需仪器

|                                              |           |        |          |
|----------------------------------------------|-----------|--------|----------|
| COD 反应器, 115/230 V, North American Plug..... | 1.....    | 个..... | 45600-00 |
| COD 反应器, 230 V, European Plug.....           | 1.....    | 个..... | 45600-02 |
| COD/TNT 适配器 .....                            | 1.....    | 个..... | 48464-00 |
| 漏斗.....                                      | 1.....    | 个..... | 25843-35 |
| 试管架.....                                     | 1-3 ..... | 个..... | 18641-00 |
| TenSette 移液管, 0-10 mL .....                  | 1.....    | 个..... | 19700-10 |

|            |           |             |          |
|------------|-----------|-------------|----------|
| 移液管嘴 ..... | 2.....    | 50/pkg..... | 21997-96 |
| 漏斗.....    | 1.....    | 个.....      | 25843-35 |
| 安全挡板.....  | 1.....    | 个.....      | 23810-00 |
| 试管冷却架..... | 1-3 ..... | 个.....      | 18641-00 |

#### OPTIONAL REAGENTS

|                                                                                   |                |          |
|-----------------------------------------------------------------------------------|----------------|----------|
| Nitrogen, Ammonia, 100 mg/L $\text{NH}_3\text{-N}$ .....                          | 500 mL.....    | 24065-49 |
| Nitrogen, Ammonia, Voluette Ampule, 160 mg/L $\text{NH}_3\text{-N}$ , 10 mL ..... | 16/pkg.....    | 21091-10 |
| Sulfuric Acid, ACS .....                                                          | 500 mL.....    | 979-49   |
| Primary Standards for Kjeldahl Nitrogen .....                                     | set of 3.....  | 22778-00 |
| Ammonium p-Toluenesulfonate .....                                                 | 25 g.....      | 22779-24 |
| Glycine p-Toluenesulfonate.....                                                   | 25 g.....      | 22780-24 |
| Nicotinic Acid p-Toluenesulfonate.....                                            | 25 g.....      | 22781-24 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                   | 50 mL MDB..... | 2450-26  |
| Water, organic-free.....                                                          | 500 mL.....    | 26415-49 |

#### OPTIONAL APPARATUS

|                                                       |                   |          |
|-------------------------------------------------------|-------------------|----------|
| Ampule Breaker Kit .....                              | each .....        | 21968-00 |
| Balance, analytical, 115 VAC.....                     | each .....        | 26103-00 |
| Balance, analytical, 230 VAC.....                     | each .....        | 26103-02 |
| Cots, finger .....                                    | 2/pkg .....       | 14647-02 |
| Cylinder, graduated, mixing, 25 mL (3 required) ..... | each .....        | 26363-40 |
| Flask, volumetric, Class A, 1000 mL (3 required)..... | each .....        | 14574-53 |
| Flask, volumetric, Class A, 100 mL.....               | each .....        | 14574-42 |
| Pipet, volumetric, Class A, 20 mL .....               | each .....        | 14515-20 |
| pH Paper, 1 to 11 pH units .....                      | 5 rolls/pkg ..... | 391-33   |

## 总氮 TNT 过硫酸盐消解法 高量程 (10.0 to 150.0 mg/L N) 方法号: 10072

Test 'N Tube



1. 打开COD反应器。  
加热到 103-106 °C。  
(最佳温度 105 度)

注: 防护设备应该到  
位, 以防检测人员受  
到伤害。

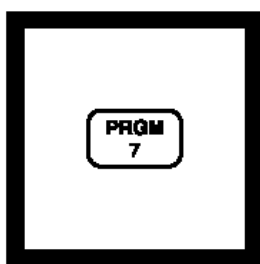
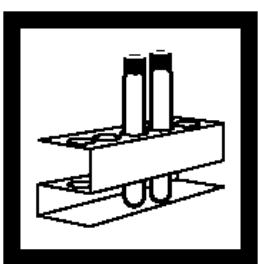
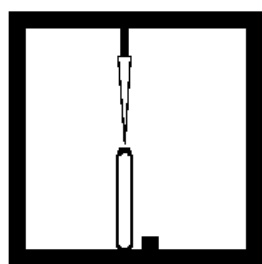
2. 准备试剂空白试  
剂: 用漏斗将总氮过  
硫酸盐试剂粉加入到  
一支HR总氮氢氧化物  
消解试瓶。

3. 往瓶中注入 0.5  
毫升不含有机的  
水。盖上瓶盖。大力  
摇晃。(约 30 秒) 将  
该试剂空白溶液和样  
品一样进行消解和色  
度处理。留于步骤 6  
使用。

4. 准备样品: 用漏斗  
将总氮过硫酸盐试剂  
粉加入到另外一支HR  
总氮氢氧化物消解试  
瓶。

注: 摇晃后过硫酸盐  
试剂粉不会完全溶  
解。

注: 如果在黑暗环境  
中, 空白试剂可以保  
持稳定长达 7 天。



5. 将 5 毫升样品注入  
试瓶中。盖紧瓶盖摇  
晃 30 秒钟。

6. 将两只试瓶放在  
COD反应器上。加热30  
分钟。

7. 将试瓶拿出反应  
器, 冷却到室温。

8. 输入检测总氮的程  
序编号。

按下: PRGM

屏幕显示:

PRGM?



9. 按下: 69 ENTER  
屏幕将显示:  
**mg/L, N和ZERO图标**



10. 分别往装有消解过的空白试剂和样品的试剂瓶中加入总氮试剂A粉末。盖上瓶盖，摇晃15秒。

按下: **TIMER ENTER**  
将开始3分钟的反应计时。



11. 计时器鸣响后，往试剂瓶值中加入总氮试剂 B 粉末。盖紧瓶盖。摇晃 15 秒钟。  
屏幕将显示:

**02:00 Timer 2**

按下: **ENTER**  
将开始两分钟的反应计时。



12. 计时器鸣响后，分别打开总氮试剂C瓶，往其中注入消解过的样品（或空白试剂）。试剂瓶将变暖。



13. 盖上瓶盖，缓慢反转瓶十次。试剂瓶将变暖。



14. 屏幕将显示:  
**05:00 Timer 3**  
按下: **ENTER**  
五分钟反应计时开始。不要再反转试剂瓶。



15. 将 COD/TNT 适配器放入试瓶槽中。



16. 计时器鸣响后，将装有空白试剂的瓶壁擦干净，然后放入样品适配器中。盖紧瓶盖。



17. 按下: ZERO  
屏幕将显示:  
**0 mg/L N**



18. 将装有样品的总氮试剂C瓶壁擦干净。



19. 将样品瓶放入样品适配器中。盖紧瓶盖。



20. 按下: READ  
指针将右移, 屏幕将显示总氮的含量, 单位是mg/L。

*注: 应使用预制的标准试剂进行标准校正。*

*注: 如果屏幕闪烁显示“Limit”, 稀释样品, 重复消解处理, 完成比色测试。如果要得到精确结果, 应重复进行消解处理。稀释和比色测试不会影响最终结果。最终结果应是读数乘上稀释系数。*

## 干扰

将会使所测含量改变10%的干扰物质

| 物质  | 水平和影响        |
|-----|--------------|
| 溴化物 | >240ppm正干扰   |
| 氯化物 | >3000 ppm正干扰 |

下表中的物质经过测试，发现在所示水平范围内不干扰（mg/L）：

| 物质    | 测试最大的水平（mg/L） |
|-------|---------------|
| 钡     | 10.4          |
| 钙     | 1200          |
| 铬(3+) | 2             |
| 铁     | 4             |
| 铅     | 26.4 ppb      |
| 镁     | 2000          |
| 有机碳   | 600           |
| pH    | 13 pH单位       |
| 磷     | 400           |
| 硅     | 650           |
| 银     | 3.69          |
| 锡     | 6             |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL/L). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in Section 1.

## Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

**1.** Prepare one or more of the following three solutions. Each preparation is for an equivalent 120 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

**a)** Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

**b)** Weigh 2.1179 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark. **c)** Weigh 2.5295 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark .

2. Analyze each of these solutions using the test procedure above.

Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{120} \times 100$$

The percent recovery should be:

| Compound       | Lowest Expected % Recovery |
|----------------|----------------------------|
| Ammonia-PTSA   | 95%                        |
| Glycine-PTSA   | 95%                        |
| Nicotinic-PTSA | 95%                        |

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

#### Standard Solution Method

For proof of accuracy, substitute 0.5 mL of a 125 mg/L ammonia nitrogen standard solution for the sample in the procedure. To prepare a 125-mg/L standard, use a 25-mL Class A pipet to transfer 25.00 mL of a 1000-mg/L Ammonia Nitrogen Standard (see *OPTIONAL REAGENTS* on page 370) to a 200-mL Class A volumetric flask. Dilute to the line with organic-free water.

#### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off an Ammonia Nitrogen Voluette. Ampule Standard Solution, 1000 mg/L as  $\text{NH}_3\text{-N}$ .
- Use the TenSette. Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders.
- Stopper each cylinder and mix thoroughly.
- Add 0.5 mL of each prepared solution, respectively, to three HR Total Nitrogen Hydroxide Digestion Vials.
- Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 4 mg/L N for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

#### Blanks for Colorimetric Measurement

The reagent blank may be used repeatedly for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18°C–25 °C) for a maximum of seven days. If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 125 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 3 mg/L N. For more information on Hach's precision statement, see *Section 1*.

### Estimated Detection Limit

The estimated detection limit for program 69 is 7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

### REQUIRED REAGENTS

Test  $\text{j}^-$ N Tube HR总氮试剂一套 (50 vials) .....27141-00

包括: (1) 26718-46, (1) 26719-46, (1) 26720-46, \*(50) Hydroxide Digestion Vials,

\*(50) Acid Solution Vials

| 试剂种类                    | 所需数量<br>每次测试 | 单位     | 货号       |
|-------------------------|--------------|--------|----------|
| HR 总氮氢氧化物消解瓶.....       | 1 瓶          | 50/pkg | *        |
| 总氮过(二)硫酸盐粉末试剂 .....     | 1 包          | 50/pkg | 26718-46 |
| 总氮试剂 A, 重亚硫酸盐粉末试剂 ..... | 1 包          | 50/pkg | 26719-46 |
| 总氮试剂B, 粉末指示剂.....       | 1 包          | 50/pkg | 26720-46 |
| 总氮试剂C 瓶, 酸溶液.....       | 1 瓶          | 50/pkg | *        |

### 所需仪器

|                                              |           |        |          |
|----------------------------------------------|-----------|--------|----------|
| COD 反应器, 115/230 V, North American Plug..... | 1.....    | 个      | 45600-00 |
| COD 反应器, 230 V, European Plug.....           | 1.....    | 个      | 45600-02 |
| COD适配器.....                                  | 1.....    | 个      | 48464-00 |
| 试管架.....                                     | 1-3 ..... | 个      | 18641-00 |
| TenSette 移液管, 0-10 mL .....                  | 1.....    | 个      | 19700-10 |
| 移液管嘴 .....                                   | 2.....    | 50/pkg | 21997-96 |
| 漏斗.....                                      | 1.....    | 个      | 25843-35 |
| 安全挡板.....                                    | 1.....    | 个      | 23810-00 |
| 试管冷却架.....                                   | 1-3 ..... | 个      | 18641-00 |

### OPTIONAL REAGENTS

|                                                                                       |                 |          |
|---------------------------------------------------------------------------------------|-----------------|----------|
| Nitrogen, Ammonia, 1000 mg/L $\text{NH}_3\text{-N}$ .....                             | 1 L .....       | 23541-53 |
| Nitrogen, Ammonia, Voluette Ampule,<br>1000 mg/L $\text{NH}_3\text{-N}$ , 10 mL ..... | 16/pkg .....    | 23541-10 |
| Sulfuric Acid, ACS .....                                                              | 500 mL .....    | 979-49   |
| Primary Standards for Kjeldahl Nitrogen .....                                         | set of 3 .....  | 22778-00 |
| Ammonium p-Toluenesulfonate .....                                                     | 25 g .....      | 22779-24 |
| Glycine p-Toluenesulfonate .....                                                      | 25 g .....      | 22780-24 |
| Nicotinic Acid p-Toluenesulfonate .....                                               | 25 g .....      | 22781-24 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                       | 50 mL MDB ..... | 2450-26  |
| Water, organic-free.....                                                              | 500 mL .....    | 26415-49 |

## OPTIONAL APPARATUS

| Description                                      | Unit             | Cat. No. |
|--------------------------------------------------|------------------|----------|
| Ampule Breaker Kit .....                         | each.....        | 21968-00 |
| Balance, analytical, 115 Vac .....               | each.....        | 26103-00 |
| Balance, analytical, 230 Vac .....               | each.....        | 26103-02 |
| Cots, finger .....                               | 2/pkg.....       | 14647-02 |
| Cylinder, graduated, mixing, 25 mL ..... 3 ..... | each.....        | 26363-40 |
| Flask, volumetric, Class A, 1000 mL .....3 ..... | each.....        | 14574-53 |
| Flask, volumetric, Class A, 200 mL.....          | each.....        | 14574-45 |
| Pipet, volumetric, Class A, 25 mL .....2 .....   | each.....        | 14515-40 |
| pH Paper, 1 to 11 pH units .....                 | 5 rolls/pkg..... | 391-33   |

### ***For Technical Assistance, Price and Ordering***

In the U.S.A.;<sup>a</sup>Call 800-227-4224. Out side the U.S.A;<sup>a</sup> Contact the Hach office or distributor serving you.

Outside the U.S.A.;<sup>a</sup>Contact the Hach office or distributor serving you.

## 总有机碳 直读法 低量程 (0- 20.0 mg/L C)

方法号: 10129

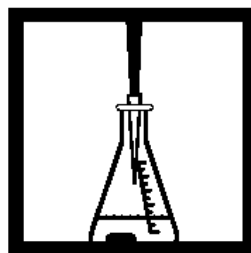


1. 开启COD反应器，加热到103-105 °C。将塑料罩放在反应器前面。

注：确保安全装置放在适当位置，从而保护测试人员免受泄漏物的影响。



2. 用具刻度量筒量取 10mL 样品加入到容积为 50-mL 的带搅拌棒的锥形烧瓶中。



3. 加入 0.4 mL 的 pH 值为2.0的缓冲溶液。

注：用pH试纸检验，从而确保样品pH为2。

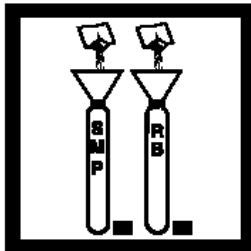


4. 将锥形瓶放在搅拌台上，用中速搅拌 10 分钟。

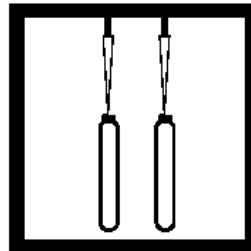


5. 标记两个低含量酸消化瓶，分别为“样品”和“试剂空白”。

注：对每种样品都需要进行试剂空白校正。



6. 使用漏斗分别各加入一包 TOC 过(二)硫酸盐粉包到两个酸消化瓶中（无色液体）。



7. 用 TenSette 移液管分别将 3.0 mL 的不含有机物的水注入到试剂空白的瓶中，将 3.0mL 待测试样注入到样品瓶中。晃动使之混合。



8. 用去离子水冲洗两个蓝色的指示剂安瓿瓶，然后用不含麻的软布拭擦干净。

注：拭擦后不要碰到安瓿瓶的外壁边，应从顶部拿取。



9. 将未开封的安培瓶放入酸消化瓶中，当安培瓶的刻度与酸消化瓶顶部平齐时，折断安培瓶的顶部，让其落入酸消化瓶。



10. 把该组合瓶盖严实后，将它们放入COD反应器2小时，反应的温度为103-105 ° C。



11. 小心地将组合瓶从反应器中取出，将它们放上试管架。为得到准确结果，让瓶子冷却1小时。



12. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。

*注：将安培瓶插入后，不要反转或倾斜组合瓶，以防止指示剂与酸消解瓶中的试剂混合。*



13. 输入检测低量程总有机碳的程序编号。  
按下：PRGM  
屏幕将显示：  
PRGM?



14. 按下：  
116 ENTER  
屏幕将显示：  
0.00 mg/L和ZERO图标。



15. 先用湿毛巾擦试剂空白瓶，然后干毛巾擦，擦去指纹或其它印痕。

*注：试剂空白瓶中的液体为深蓝色。*



16. 将试剂空白瓶放入样品适配器中。直接从瓶的顶部下按直到其牢固地嵌入适配器中。



17. 盖紧遮光盖。



18. 按 **ZERO**, 指针将右移, 屏幕显示:  
**0 mg/L C**



19. 先用湿毛巾擦试剂样品瓶, 然后干毛巾擦, 擦去指纹或其它印痕。



20. 将试剂样品瓶放入样品适配器中。直接从瓶的顶部下按直到其牢固地嵌入适配器中。



21. 盖紧遮光盖。



22. 按下 **READ**  
指针将右移, 屏幕将显示碳 C 的含量, 单位为 mg/L。

## 干扰

经过对以下物质进行过干扰测试后发现当它们的含量不大于以下所列最大水平的情况时，不会产生干扰。

表一 非干扰物质

| 物质     | 最大含量                                          |
|--------|-----------------------------------------------|
| 铝      | 10 mg/L                                       |
| 氨氮     | 1000 mg/L as N                                |
| ASTM废水 | 没影响                                           |
| 溴化物    | 500 mg/L Br <sup>-</sup>                      |
| 溴      | 25 mg/L Br <sub>2</sub>                       |
| 钙      | 2000 mg/L as CaCO <sub>3</sub>                |
| 氯化物    | 500 mg/L                                      |
| 氯      | 10 mg/L Cl <sub>2</sub>                       |
| 二氧化氯   | 6 mg/L ClO <sub>2</sub>                       |
| 铜      | 10 mg/L                                       |
| 氰化物    | 10 mg/L                                       |
| 铁（二价）  | 10 mg/L                                       |
| 铁（三价）  | 10 mg/L                                       |
| 镁      |                                               |
| 锰      | 1 mg/L                                        |
| 一氯化物   | 14 mg/L NH <sub>2</sub> Cl as Cl <sub>2</sub> |
| 亚硝酸盐   | 500 mg/L NO <sub>2</sub> <sup>-</sup>         |
| 臭氧     | 2 mg/L O <sub>3</sub>                         |
| 磷酸盐    | 3390 mg/L                                     |
| 硅石     | 100 mg/L SiO <sub>2</sub>                     |
| 硫酸盐    | 5000 mg/L                                     |
| 硫化物    | 20 mg/L                                       |
| 亚硫酸盐   | 50 mg/L                                       |
| 锌      | 5 mg/L                                        |

如果样品碱性碳酸钙含量超过600mg/L时，在检测过程中加入硫酸之前，将样品的PH值降低到7以下。

样品中大多数的浑浊物将会在消解过程中溶解或者在冷却过程中沉淀。经过测试50NTU单位以下的浑浊物将不产生干扰。

## Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples

## Accuracy Check

### Standard Solutions Method

**a.** Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

**b.** Prepare a 10.0 mg/L C standard by transferring 10.00 mL of the stock standard to a 1000-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

### Standard Additions Method

**a.** Prepare a 150 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with organic-free water. Mix.

**b.** Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 150 mg/L C standard to each of three Acid Digestion vials.

**c.** Add the contents of one TOC Persulfate powder pillow to each vial.

**d.** Add 3.0 mL of sample to each vial. Swirl to mix.

**e.** Proceed with the procedure starting at *step 8*.

**f.** The mg/L C concentration should increase by 5.0 mg/L for each 0.1 mL increment.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 9.0 mg/L C and one lot of reagents, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L C.

### Estimated Detection Limit

The estimated detection limit for Method 10129 is 0.3 mg/L C.

### Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 0.2 mg/L C.

## Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

## 所需试剂

| 试剂种类 | 所需数量<br>每次测试 | 单位 | 货号 |
|------|--------------|----|----|
|------|--------------|----|----|

总有机氮直读法

|                              |          |  |          |
|------------------------------|----------|--|----------|
| Test $\text{j}^-$ N Tube试剂一套 | 50 vials |  | 27603-45 |
|------------------------------|----------|--|----------|

包括:

|         |   |        |   |
|---------|---|--------|---|
| 酸性消解溶液瓶 | 1 | 50/pkg | * |
|---------|---|--------|---|

|         |        |       |        |
|---------|--------|-------|--------|
| 硫酸盐缓冲溶液 | 0.4 mL | 25 mL | 452-33 |
|---------|--------|-------|--------|

|    |   |   |          |
|----|---|---|----------|
| 漏斗 | 1 | 个 | 25843-35 |
|----|---|---|----------|

|                      |   |        |   |
|----------------------|---|--------|---|
| 指示剂安瓿, Low Range TOC | 1 | 10/pkg | * |
|----------------------|---|--------|---|

|                |   |        |   |
|----------------|---|--------|---|
| TOC过(二)硫酸盐粉末试剂 | 1 | 50/pkg | * |
|----------------|---|--------|---|

|          |        |        |          |
|----------|--------|--------|----------|
| 不含有机物水** | 3.0 mL | 500 mL | 26415-49 |
|----------|--------|--------|----------|

## 所需仪器

|                                           |   |   |          |
|-------------------------------------------|---|---|----------|
| COD 反应器, 115/230 V ac (U.S.A. and Canada) | 1 | 个 | 45600-00 |
|-------------------------------------------|---|---|----------|

|                                |   |   |          |
|--------------------------------|---|---|----------|
| COD 反应器, 115/230 V ac (Europe) | 1 | 个 | 45600-02 |
|--------------------------------|---|---|----------|

|           |   |   |        |
|-----------|---|---|--------|
| 量筒, 10-mL | 1 | 个 | 508-38 |
|-----------|---|---|--------|

|             |   |   |        |
|-------------|---|---|--------|
| 锥形烧瓶, 50-mL | 1 | 个 | 505-41 |
|-------------|---|---|--------|

|       |   |   |          |
|-------|---|---|----------|
| 磁力搅拌器 | 1 | 个 | 23436-00 |
|-------|---|---|----------|

|      |   |   |          |
|------|---|---|----------|
| 安全挡板 | 1 | 个 | 50030-00 |
|------|---|---|----------|

|     |     |   |          |
|-----|-----|---|----------|
| 试管架 | 1-3 | 个 | 18641-00 |
|-----|-----|---|----------|

|                            |   |   |          |
|----------------------------|---|---|----------|
| TenSette移液管, 0.1 to 1.0 mL | 1 | 个 | 19700-01 |
|----------------------------|---|---|----------|

|                             |   |   |          |
|-----------------------------|---|---|----------|
| TenSette移液管, 1.0 to 10.0 mL | 1 | 个 | 19700-10 |
|-----------------------------|---|---|----------|

|                                    |   |        |          |
|------------------------------------|---|--------|----------|
| 移液管嘴, for 19700-01 TenSette. Pipet | 2 | 50/pkg | 21856-96 |
|------------------------------------|---|--------|----------|

|                                    |   |        |          |
|------------------------------------|---|--------|----------|
| 移液管嘴, for 19700-10 TenSette. Pipet | 2 | 50/pkg | 21997-96 |
|------------------------------------|---|--------|----------|

|       |   |   |          |
|-------|---|---|----------|
| 磁力搅拌棒 | 1 | 个 | 45315-00 |
|-------|---|---|----------|

|                             |   |         |          |
|-----------------------------|---|---------|----------|
| Wipes, Disposable, Kimwipes | 1 | 280/pkg | 20970-00 |
|-----------------------------|---|---------|----------|

## OPTIONAL REAGENTS

|                                                   |       |  |          |
|---------------------------------------------------|-------|--|----------|
| TOC Standard Solution (KHP Standard, 1000 mg/L C) | 5/pkg |  | 27915-05 |
|---------------------------------------------------|-------|--|----------|

|                          |       |  |        |
|--------------------------|-------|--|--------|
| Potassium Acid Phthalate | 500 g |  | 315-34 |
|--------------------------|-------|--|--------|

|                                        |        |     |         |
|----------------------------------------|--------|-----|---------|
| Sulfuric Acid Reagent Solution, 5.25 N | 100 mL | MDB | 2449-32 |
|----------------------------------------|--------|-----|---------|

## OPTIONAL APPARATUS

|                    |      |  |          |
|--------------------|------|--|----------|
| Analytical Balance | each |  | 26103-00 |
|--------------------|------|--|----------|

|                            |      |  |          |
|----------------------------|------|--|----------|
| Flask, volumetric, 1000-mL | each |  | 14574-53 |
|----------------------------|------|--|----------|

|                           |      |  |          |
|---------------------------|------|--|----------|
| Flask, volumetric, 100-mL | each |  | 14574-42 |
|---------------------------|------|--|----------|

|                          |      |  |          |
|--------------------------|------|--|----------|
| Pipet, Class A, 10.00-mL | each |  | 14515-38 |
|--------------------------|------|--|----------|

|                          |      |  |          |
|--------------------------|------|--|----------|
| Pipet, Class A, 15.00-mL | each |  | 14515-39 |
|--------------------------|------|--|----------|

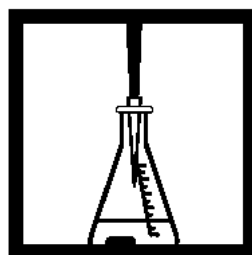


1. 开启COD反应器，加热到103-105 °C。将塑料罩放在反应器前面。

*注：确保安全装置放在适当位置，从而保护测试人员免受泄漏物的影响。*

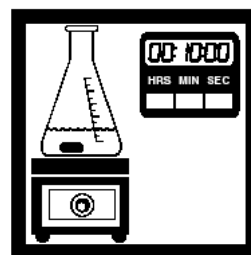


2. 用具刻度量筒量取 10mL 样品加入到容积为 50-mL 的带搅拌棒的锥形烧瓶中。



3. 加入 0.4 mL 的 pH 值为2.0的缓冲溶液。

*注：用pH试纸检验，从而确保样品pH为2。*

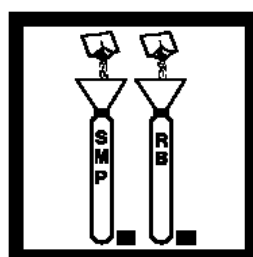


4. 将锥形瓶放在搅拌台上，用中速搅拌 10 分钟。

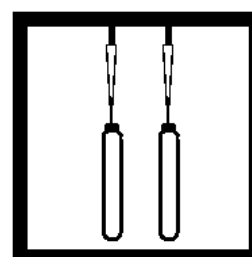


5. 标记两个低含量酸消化瓶，分别为“样品”和“空白试剂”。

*注：对每种样品都需要进行试剂空白校正。*



6. 使用漏斗分别各加入一包 TOC 过硫酸盐粉包到两个酸消化瓶中（无色液体）。



7. 用 TenSette 移液管分别将 3.0 mL 的不含有机物的水注入到试剂空白的瓶中，将 3.0mL 待测试样注入到样品瓶中。晃动使之混合。



8. 用去离子水冲洗两个蓝色的指示剂安瓿瓶，然后用不含麻的软布拭擦干净。

*注：拭擦后不要碰到安瓿瓶的外壁边，应从顶部拿取。*



9. 将未开封的安培瓶放入酸消化瓶中，当安培瓶的刻度与酸消化瓶顶部平齐时，折断安培瓶的顶部，让其落入酸消化瓶。



10. 把该组合瓶盖严实后，将它们放入COD反应器2小时，反应的温度为103-105 °C。



11. 小心地将组合瓶从反应器中取出，将它们放上试管架。为得到准确结果，让瓶子冷却1小时。

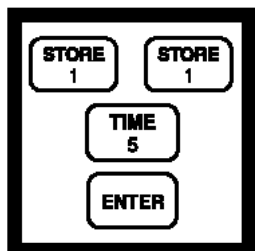


12. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。

*注：将安培瓶插入后，不要反转或倾斜组合瓶，以防止指示剂与酸消解瓶中的试剂混合。*



13. 输入检测低量程总有机碳的程序编号。  
按下：PRGM  
屏幕将显示：  
PRGM?



14. 按下：  
115 ENTER  
屏幕将显示：  
0.00 mg/L和ZERO图标。



15. 先用湿毛巾擦试剂空白瓶，然后干毛巾擦，擦去指纹或其它印痕。

*注：试剂空白瓶中的液体为深蓝色。*



16. 将试剂空白瓶放入样品适配器中。直接从瓶的顶部下按直到其牢固地嵌入适配器中。



17. 盖紧遮光盖。



18. 按 ZERO, 指针将右移, 屏幕显示:  
**0 mg/L C**



19. 先用湿毛巾擦试剂样品瓶, 然后干毛巾擦, 擦去指纹或其它印痕。



20. 将试剂样品瓶放入样品适配器中。直接从瓶的顶部下按直到其牢固地嵌入适配器中。



21. 盖紧遮光盖。



22. 按下 READ  
指针将右移, 屏幕将显示碳 C 的含量, 单位为 mg/L。

## 干扰

经过对以下物质进行过干扰测试后发现当它们的含量不大于以下所列最大水平的情况时，不会产生干扰。

表一 非干扰物质

| 物质     | 最大含量                                          |
|--------|-----------------------------------------------|
| 铝      | 10 mg/L                                       |
| 氨氮     | 1000 mg/L as N                                |
| ASTM废水 | 没影响                                           |
| 溴化物    | 500 mg/L Br <sup>-</sup>                      |
| 溴      | 25 mg/L Br <sub>2</sub>                       |
| 钙      | 2000 mg/L as CaCO <sub>3</sub>                |
| 氯化物    | 500 mg/L                                      |
| 氯      | 10 mg/L Cl <sub>2</sub>                       |
| 二氧化氯   | 6 mg/L ClO <sub>2</sub>                       |
| 铜      | 10 mg/L                                       |
| 氰化物    | 10 mg/L                                       |
| 铁（二价）  | 10 mg/L                                       |
| 铁（三价）  | 10 mg/L                                       |
| 镁      |                                               |
| 锰      | 1 mg/L                                        |
| 一氯化物   | 14 mg/L NH <sub>2</sub> Cl as Cl <sub>2</sub> |
| 亚硝酸盐   | 500 mg/L NO <sub>2</sub> <sup>-</sup>         |
| 臭氧     | 2 mg/L O <sub>3</sub>                         |
| 磷酸盐    | 3390 mg/L                                     |
| 硅石     | 100 mg/L SiO <sub>2</sub>                     |
| 硫酸盐    | 5000 mg/L                                     |
| 硫化物    | 20 mg/L                                       |
| 亚硫酸盐   | 50 mg/L                                       |
| 锌      | 5 mg/L                                        |

如果样品碱性碳酸钙含量超过600mg/L时，在检测过程中加入硫酸之前，将样品的PH值降低到7以下。

样品中大多数的浑浊物将会在消解过程中溶解或者在冷却过程中沉淀。经过测试50NTU单位以下的浑浊物将不产生干扰。

## Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

## Accuracy Check

### Standard Solutions Method

**a.** Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27945-05).

**b.** Prepare a 300 mg/L C standard by transferring 15.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

### Standard Additions Method

**a.** Prepare a 300 mg/L C standard by transferring 18.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

**b.** Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.

**c.** Add the contents of one TOC Persulfate powder pillow to each vial.

**d.** Add 0.3 mL of sample to each vial. Swirl to mix.

**e.** Proceed with the procedure starting at *step 8*.

**f.** The mg/L C concentration should increase by 100 mg/L for each 0.1 mL increment.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 360 mg/L C and one lot of reagents, a single operator obtained a standard deviation of  $\pm 8$  mg/L C.

### Estimated Detection Limit

Use Method Number 10129 to test TOC levels below 20 mg/L C.

### Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 6 mg/L C.

## Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

## 所需试剂

| 试剂种类                         | 所需数量<br>每次测试 | 单位     | 货号       |
|------------------------------|--------------|--------|----------|
| 总有机氮直读法高量程                   |              |        |          |
| Test $\text{j}^-$ N Tube试剂一套 | 50 vials     |        | 27604-45 |
| 包括:                          |              |        |          |
| 酸性消解溶液瓶                      | 1            | 50/pkg | *        |
| 硫酸盐缓冲溶液                      | 0.4 mL       | 25 mL  | 452-33   |
| 漏斗                           | 1            | 个      | 25843-35 |
| 指示剂安瓿, Low Range TOC         | 1            | 10/pkg | *        |
| TOC过(二)硫酸盐粉末试剂               | 1            | 50/pkg | *        |
| 不含有机物水**                     | 3.0 mL       | 500 mL | 26415-49 |

## 所需仪器

|                                           |     |         |          |
|-------------------------------------------|-----|---------|----------|
| COD 反应器, 115/230 V ac (U.S.A. and Canada) | 1   | 个       | 45600-00 |
| COD 反应器, 115/230 V ac (Europe)            | 1   | 个       | 45600-02 |
| 量筒, 10-mL                                 | 1   | 个       | 508-38   |
| 锥形烧瓶, 50-mL                               | 1   | 个       | 505-41   |
| 磁力搅拌器                                     | 1   | 个       | 23436-00 |
| 安全挡板                                      | 1   | 个       | 50030-00 |
| 试管架                                       | 1-3 | 个       | 18641-00 |
| TenSette移液管, 0.1 to 1.0 mL                | 1   | 个       | 19700-01 |
| TenSette移液管, 1.0 to 10.0 mL               | 1   | 个       | 19700-10 |
| 移液管嘴, for 19700-01 TenSette. Pipet        | 2   | 50/pkg  | 21856-96 |
| 移液管嘴, for 19700-10 TenSette. Pipet        | 2   | 50/pkg  | 21997-96 |
| 磁力搅拌棒                                     | 1   | 个       | 45315-00 |
| Wipes, Disposable, Kimwipes               | 1   | 280/pkg | 20970-00 |

## OPTIONAL REAGENTS

|                                                   |        |     |          |
|---------------------------------------------------|--------|-----|----------|
| TOC Standard Solution (KHP Standard, 1000 mg/L C) | 5/pkg  |     | 27915-05 |
| Potassium Acid Phthalate                          | 500 g  |     | 315-34   |
| Sulfuric Acid Reagent Solution, 5.25 N            | 100 mL | MDB | 2449-32  |

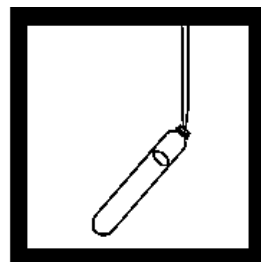
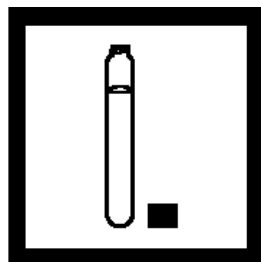
## OPTIONAL APPARATUS

|                            |      |  |          |
|----------------------------|------|--|----------|
| Analytical Balance         | each |  | 26103-00 |
| Flask, volumetric, 1000-mL | each |  | 14574-53 |
| Flask, volumetric, 100-mL  | each |  | 14574-42 |
| Pipet, Class A, 10.00-mL   | each |  | 14515-38 |
| Pipet, Class A, 15.00-mL   | each |  | 14515-39 |

## 化学需氧量 反应器消解法

方法号：8000

### 消解处理



1. 将 500ml 试样用搅拌机均匀搅拌 2 分钟。  
2. 开启 COD 分解炉，预热至 150℃。

*注意：加温时务必加上防护罩以免试管破裂喷溅。*

3. 根据样品的 CONC 的范围选择适当的 COD 消解试剂瓶，打开瓶盖。

| Sample Conc. Range (mg/L) | COD Digestion Reagent Vial Type |
|---------------------------|---------------------------------|
| 0 to 150                  | Low Range                       |
| 0 to 1500                 | High Range                      |
| 0 to 15,000               | High Range Plus                 |

4. 将试剂斜放45度，加入2ml试样（测试0 ~ 15,000 mg/L时加 0.2mL试样）。

*注：试剂泄漏会影响精确度，且对皮肤造成伤害，一旦皮肤沾上试剂，以流动的清水冲洗。*

*注：试剂对光敏感，应贮存在包装盒中，最好能放在冰箱。操作过程所接受的光量不会影响试剂品质。*

**小心：**有些化学物质和在该过程中使用的仪器如果使用不当或者错误使用，将对使用者的健康和安全造成危险。请参考相关部分的内容。应穿带适当的眼睛保护装置和衣服。如果接触到危险物品，应用水冲洗该部位。应小心遵循所有的操作指导。



5. 将试剂瓶放回原位。擦干净瓶外壁。



6. 抓紧试剂瓶盖。反转瓶多次，使溶液混合。将试剂瓶放上预热的 COD 反应器上。



7. 按照步骤 3—6 准备空白试样。用去离子水 2ml 去离子水（测试 0 ~ 15,000 mg/L 时加 0.2mL 水）。代替样品。



8. 将所有试剂瓶加热 2 小时。



9. 关掉反应器。等待大约 20 分钟，等到试瓶温度低于 120 度或以下。



10. 趁热时反转试瓶多次。将瓶放在架上。等到其温度降至室温。

注：当绿色显现时，检测 COD。如有必要，重复检测稀释的样品。



11. 使用以下的技术去测量 COD。

- 比色法  
0-150 mg/L COD
- 比色法  
0-1,500 mg/L COD
- 比色法  
0-15,000 mg/L COD

## 比色检测 0 to 150 mg/L COD



1. 输入检测需氧量法的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 将COD反应器预热至150 °C以待以后步骤使用。



2. 按下: **16 ENTER**

屏幕将显示:

**mg/L, COD和ZERO** 图标。

注: 如果检测其他形态 ( $O_2$ ), 按下:

**CONC键**



3. 旋转COD/TNT适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。



4. 用布擦干净装有空白试样的试瓶。



5. 旋转 COD/TNT 适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。



6. 盖紧瓶盖。



7. 按 **ZERO**, 指针将右移, 屏幕显示:

**0 mg/L COD**



8. 先用湿毛巾擦试剂样品瓶, 然后干毛巾擦, 擦去指纹或其它印痕。



9. 将药品试剂放入样品适配器中。



10. 盖紧瓶盖。



11. 按下：READ  
指针将右移，屏幕将显示 COD 的含量，单位是 mg/L。

比色检测 0 to 1,500 and 0 to 15,000 mg/L COD



1. 输入检测需氧量法的程序编号。  
按下：PRGM  
屏幕将显示：  
PRGM?



2. 按下：17 ENTER  
屏幕将显示：  
mg/L, COD和ZERO  
图标。



3. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



4. 用布擦干净装有空白试样的试瓶。

注：将COD反应器预热至150 ° C以待以后步骤使用。

注：如果检测其他形态 (O<sub>2</sub>)，按下：  
CONC键



5. 旋转 COD/TNT 适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



6. 盖紧瓶盖。



7. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L COD**



8. 先用湿毛巾擦试剂样品瓶，然后干毛巾擦，擦去指纹或其它印痕。



9. 将药品试剂放入样品适配器中。



10. 盖紧瓶盖。



11. 按下：READ  
指针将右移，屏幕将显示 COD 的含量，单位是 mg/L。

*注：当使用高量程的 COD 消解试剂瓶时，读数应该乘以十倍。*

*注：为得到精确的结果，含量范围在1,500 or 15,000 mg/L COD 的样品应使用稀释溶液重复进行检测。*

## 干扰

当测定COD浓度时，氯化物是最主要的干扰。每个 COD瓶 含有可除去表一第一列水平氯化物干扰的硫化汞。含较高氯化物含量的样品应稀释。稀释样品使其能足够减低栏2所列水平的氯化物浓度。如果样品稀释后引起COD浓度太低无法正确测量，在加入样品之前，加入0.50g 硫酸汞到每个COD瓶中。额外的硫酸汞将使氯化物浓度满足第3列水平。

表一。

| 使用的小瓶形式 | 样品中最大Cl <sup>-</sup> 浓度(mg/L) | 稀释样品的建议Cl <sup>-</sup> 浓度 (mg/L) | 样品中最大 Cl <sup>-</sup> 浓度 (加入 0.5g HgSO <sub>4</sub> ) (mg/L) |
|---------|-------------------------------|----------------------------------|--------------------------------------------------------------|
| 低含量     | 2000                          | 1000                             | 8000                                                         |
| 高含量     | 2000                          | 1000                             | 4000                                                         |
| 超高含量    | 20,000                        | 10,000                           | 40,000                                                       |

## Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see *Correction for Volume Additions* (Section 1) for more information.

## Accuracy Check

### Standard Solution Method

Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2.0 mL as the sample volume. The expected result will be 100 mg/L COD. As an alternative, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to make a 100-mg/L standard. Check the accuracy of the 0 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Alternatively, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water. Use 2.0 mL of one of these solutions as the sample volume. Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

## Method Performance

### Precision

Program #16: In a single laboratory, using a standard solution of 100 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 2$  mg/L COD.

Program #17: In a single laboratory, using a standard solution of 1000 mg/L COD and

two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 16$  mg/L COD.

For more information on Hachj<sup>-</sup>'s precision statement, see *Section 1*.

#### Estimated Detection Limit (EDL)

The EDL for program 16 is 4 mg/L COD. The EDL for program 17 is 30 mg/L COD.

For more information on derivation and use of Hachj<sup>-</sup>'s estimated detection limit, see *Section 1*.

### Blanks for Colorimetric Determination

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (420 or 610 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of deionized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

### Summary of Method

The mg/L COD results are defined as the mg of O<sub>2</sub> consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion (Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup>) to green chromic ion (Cr<sub>3</sub><sup>+</sup>). When the 0-150 mg/L colorimetric method is used, the amount of Cr<sub>6</sub><sup>+</sup> remaining is determined. When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr<sub>3</sub><sup>+</sup> produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex the chloride interference.

### Pollution Prevention and Waste Management

Final samples will contain mercury (D009), silver (D011), and chromium (D007) at concentration levels regulated by the Federal RCRA. Please see *Section 3* for further information on proper disposal of these materials.

### 所需试剂

| 试剂种类                                | 所需数量<br>每次测试 | 单位     | 货号       |
|-------------------------------------|--------------|--------|----------|
| 选择适当的 COD 消解试剂瓶:                    |              |        |          |
| 低量程, 0 to 150 mg/L COD.....         | 1 to 2 瓶     | 25/pkg | 21258-25 |
| 高量程, 0 to 1,500 mg/L COD.....       | 1 to 2 瓶     | 25/pkg | 21259-25 |
| 高量程 Plus, 0 to 15,000 mg/L COD..... | 1 to 2 瓶     | 25/pkg | 24159-25 |
| 去离子水.....                           | 不定           | 4 L    | 272-56   |

### REQUIRED APPARATUS

|                                           |   |   |          |
|-------------------------------------------|---|---|----------|
| 振荡搅拌器, 120 V, 14 speed.....               | 1 | 个 | 26747-00 |
| COD 反应器, 115/230 V.....                   | 1 | 个 | 45600-00 |
| COD 反应器, 230 V, European-style plug ..... | 1 | 个 | 45600-02 |
| COD/TNT 适配器 .....                         | 1 | 个 | 48464-00 |

|                                          |                    |             |          |
|------------------------------------------|--------------------|-------------|----------|
| TenSette移液管, 0.1 to 1.0 mL.....          | 1.....             | 个.....      | 19700-01 |
| 移液管嘴, for 19700-01 TenSette. Pipet ..... | 1.....             | 50/pkg..... | 21856-96 |
| Pipet, volumetric, Class A, 2.00 mL..... | 1 .....            | 个 .....     | 14515-36 |
| Pipet Filler, safety bulb .....          | 1 .....            | 个 .....     | 14651-00 |
| 试管架.....                                 | 1 to 2 racks ..... | 个 .....     | 18641-00 |

## OPTIONAL REAGENTS

### Description Unit Cat. No.

|                                                        |                 |          |
|--------------------------------------------------------|-----------------|----------|
| COD Digestion Reagent Vials, 0 to 150 mg/L COD .....   | 150/pkg.....    | 21258-15 |
| COD Digestion Reagent Vials, 0 to 1,500 mg/L COD ..... | 150/pkg.....    | 21259-15 |
| COD Standard Solution, 300 mg/L.....                   | 200 mL.....     | 12186-29 |
| COD Standard Solution, 1000 mg/L .....                 | 200 mL.....     | 22539-29 |
| Mercuric Sulfate.....                                  | 28.3 grams..... | 1915-20  |
| Potassium Acid Phthalate, ACS .....                    | 500 g.....      | 315-34   |
| Potassium Dichromate Standard Solution, 0.25 N.....    | 1000 mL* .....  | 1809-53  |
| Sulfuric Acid, ACS.....                                | 500 mL* .....   | 979-49   |

## OPTIONAL APPARATUS

|                                                           |                  |          |
|-----------------------------------------------------------|------------------|----------|
| Balance, analytical, 115 V.....                           | each.....        | 26103-00 |
| Balance, analytical, 230 V.....                           | each.....        | 26103-02 |
| Beaker, 250 mL .....                                      | each.....        | 500-46   |
| Cylinder, graduated, 5 mL.....                            | each.....        | 508-37   |
| Electromagnetic Stirrer, 120 V, with electrode stand..... | each.....        | 45300-01 |
| Electromagnetic Stirrer, 230 V, with electrode stand..... | each.....        | 45300-02 |
| Flask, volumetric, Class A, 1000 mL.....                  | each.....        | 14574-53 |
| Flask, volumetric, Class A, 100 mL.....                   | each.....        | 14574-42 |
| pH Paper, 1 to 11 pH units .....                          | 5 rolls/pkg..... | 391-33   |
| Pipet, serological, 5 mL.....                             | each.....        | 532-37   |
| Pipet Tips, for 19700-01 TenSette Pipet .....             | 50/pkg*.....     | 21856-96 |
| Pipet, volumetric, Class A, 10 mL .....                   | each.....        | 14515-38 |
| Safety shield, for laboratory bench .....                 | each.....        | 23810-00 |
| Spoon, measuring, 0.5 g .....                             | each.....        | 907-00   |
| Stir Bar, 22.2 x 4.76 mm (7/8" x 3/16") .....             | each.....        | 45315-00 |
| Stir Bar Retriever.....                                   | each.....        | 15232-00 |
| Timer .....                                               | each.....        | 26304-00 |

### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.:** Call 800-227-4224

**Outside the U.S.A.:** Contact the Hach office or distributor serving you.

# 化学需氧量 三价锰消解法 (20 to 1,000 mg/L)

方法号: 10067

(不需清除氯化物)



1. 输入检测需氧量锰消解法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 将COD反应器预热至150 ° C以待以后步骤使用。



2. 按下: 18 ENTER 屏幕将显示:

mg/L, COD和ZERO 图标。

注: 如果检测其他形态 (O<sub>2</sub>) , 按下:

CONC键



3. 将 100mL 试样均匀搅拌后, 倒入 250mL 烧杯



4. 如果不存在大量的氯化物, 将0.5毫升的均匀后的样品注入锰 III COD 试瓶。盖紧瓶盖, 反转多次使之混合。

注: 如果样品的COD值20—100mg/L之间。稀释样品使之达到该范围之内。最终结果是读数乘以稀释因子。



5. 用 0.5 毫升的去离子水代替样品作为空白试样。留待步骤 10 使用。



6. 将所有试瓶放上已经预热到150度的COD反应器上, 消解处理1小时。

注: 在消解过程中, 如果瓶中试剂沸腾表明试瓶密封不好, 此时检测的结果是不正确的。



7. 将试瓶移到冷却架上二分钟, 然后在凉水中冷却至室温。这个过程一般花费 3 分钟。

注: 试瓶溶液将形成无色上层和紫色下层。反转试瓶多次使之混合。



8. 将试瓶从水中拿出, 用干布擦干。

注: 颠倒试瓶多次使溶液混合。

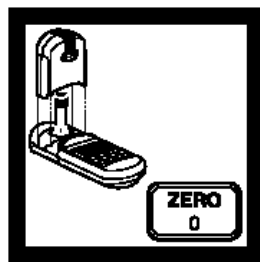
**小心：**有些化学物质和在该过程中使用的仪器如果使用不当或者错误使用，将对使用者的健康和安全造成危险。请参考相关部分的内容。应穿戴适当的眼睛保护装置和衣服。如果接触到危险物品，应用水冲洗该部位。应小心遵循所有的操作指导。



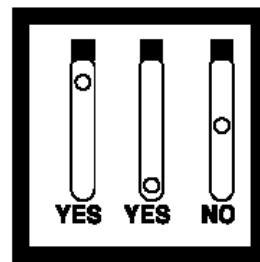
9. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



10. 将空白试样放入样品适配器中，



11. 盖紧瓶盖。  
按下：**ZERO**  
屏幕将显示：  
**0 mg/L COD**



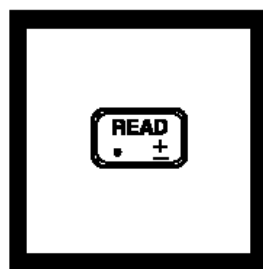
12. 如果氯化物已经清除，则滤片将不会处于试瓶中部。它会产生干扰影响结果。轻轻摇晃或往桌面轻轻敲击试瓶，使之移动。



13. 将预制试样放入样品适配器中。



14. 盖紧瓶盖。



15. 按下：**READ**  
指针将右移，屏幕将显示 COD 的含量，单位是 mg/L。

注：应使用预制的标准溶液进行标准校正。

## 干扰

当数量大的时候，无机物可能被三价锰氧化，从而产生正干扰。氯化物是最常见的干扰物质，应在在样品预处理时将其清除。如果氯化物的数量不多可以不用预处理。一个检测氯是否影响检测结果的简单方法就是分别使用或不使用除氯方法，比较两者结果。其他无机干扰物（例如：亚硝酸盐、三价铁、硫化物）并不会常产生大的干扰。如果有必要，这些干扰物可以在检测后用单独方法对最终结果进行相应的校正。当氯化物存在时，氨氮将产生干扰。

## Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be 800 ± 26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

### Method Performance (for Manganic III COD without the chloride removal procedure)

#### Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

#### Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Summary of Method

Chemical oxygen demand (COD) is defined as ... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, noncarcinogenic

chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical

Oxygen Demand (BOD) values and hexavalent chromium COD tests.

None of the oxygen demand tests provide 100% oxidation of all organic compounds. A calibration is provided which is based on the oxidation of Potassium

Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

## 所需试剂

| 试剂种类                            | 所需数量<br>每次测试 | 单位          | 货号       |
|---------------------------------|--------------|-------------|----------|
| 三价锰 COD 试剂瓶, 20-1000 mg/L ..... | 1.....       | 25/pkg..... | 26234-25 |
| 浓缩硫酸 .....                      | 1 mL.....    | 4 Kg.....   | 979-09   |
| 去离子水.....                       | 不定 .....     | 4 L.....    | 272-56   |

## 所需仪器

|                                                     |              |             |          |
|-----------------------------------------------------|--------------|-------------|----------|
| COD/TNT适配器 .....                                    | 1.....       | 个.....      | 48464-00 |
| 振荡搅拌器, 120 Vac, 14-speed .....                      | 1.....       | 个.....      | 26747-00 |
| 搅拌器容器, 118 mL.....                                  | 1.....       | 2/pkg.....  | 26748-00 |
| 瓶盖, with inert Teflon liner, for mixing bottle..... | varies ..... | 12/pkg..... | 24018-12 |
| COD 反应器, 115 V.....                                 | 1.....       | 个.....      | 45600-00 |
| COD 反应器, 230 V, European-style plug .....           | 1.....       | 个.....      | 45600-02 |
| 镊子, extra fine point .....                          | 1.....       | 个.....      | 26696-00 |
| 玻璃混合瓶, for sample + acid.....                       | 1.....       | 个.....      | 24276-06 |
| TenSette移液管, 0.1 to 1.0 mL.....                     | 1.....       | 个.....      | 19700-01 |
| TenSette移液管, 1.0 to 10.0 mL.....                    | 1.....       | 个.....      | 19700-10 |
| 移液管嘴, for 19700-01 TenSette. Pipet .....            | 2.....       | 50/pkg..... | 21856-96 |
| 移液管嘴, for 19700-10 TenSette. Pipet .....            | 2.....       | 50/pkg..... | 21997-96 |
| 安全挡板 .....                                          | 1.....       | 个.....      | 23810-00 |
| 不锈钢试管架.....                                         | 1.....       | 个.....      | 18641-00 |

## OPTIONAL REAGENTS AND APPARATUS

|                                          |             |          |
|------------------------------------------|-------------|----------|
| COD Standard Solution, 800 mg/L COD..... | 200 mL..... | 26726-29 |
| Potassium Acid Phthalate .....           | 500 g.....  | 315-34   |
| Dispenser for sulfuric acid.....         | each.....   | 25631-37 |

### *For Technical Assistance, Price and Ordering*

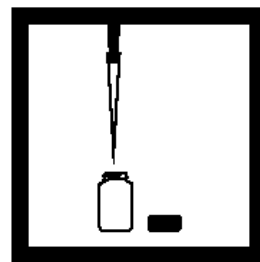
In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

## 化学需氧量 三价锰消解法 (20 to 1,000 mg/L)

方法号: 10067

(需清除氯化物)



1. 输入检测需氧量锰消解法的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 将COD反应器预热至150 ° C以待以后步骤使用。

2. 按下: 18 ENTER 屏幕将显示:

mg/L, COD和ZERO

图标。

注: 如果检测其他形态 ( $O_2$ ), 按下:

CONC键

3. 将 100mL 试样均匀搅拌 30 秒。

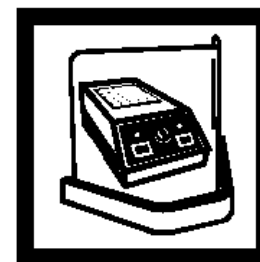
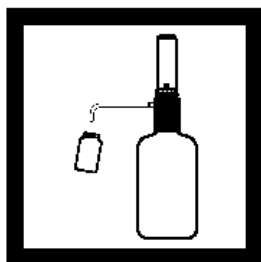
注: 混合能促使固体颗粒散开, 从而提供检测的精确性和重复能力。

除去氯化物的过程:

4. 使用移液管和安全球管将 9 毫升均匀后的样品注入一个玻璃混合瓶内。如果样品的 COD 超过 100mg/L, 根据表一将样品稀释。

注: 如果悬浮物依然存在, 在移液期间仍然不断搅拌样品。

**小心:** 有些化学物质和在该过程中使用的仪器如果使用不当或者错误使用, 将对使用者的健康和安全造成危险。请参考相关部分的内容。应穿戴适当的眼睛保护装置和衣服。如果接触到危险物品, 应用水冲洗该部位。应小心遵循所有的操作指导。



5. 使用自动分配器或 TenSette 移液管, 将 1.0 毫升的浓缩的硫磺酸注入混合瓶中。

注: 浓缩的硫磺酸和水容量并不是简单相加的。增加 1 毫升浓缩的硫磺酸到 9 毫升水中, 最终体积不是等于 10 毫升。

6. 盖上瓶盖, 反转多次。溶液将变热。使用前冷却到室温。

注: 在 4°C 冷冻环境下, 酸化的样品可以保持几个月的稳定。

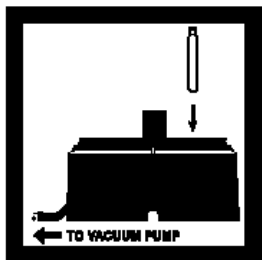
7. 重复步骤 4—6 准备空白试样, 只是用去离子水代替样品。

8. 打开 COD 反应器, 加热至 150 度。

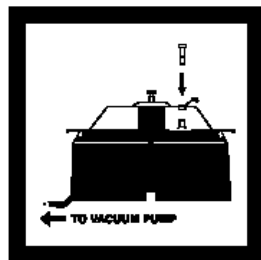
表一 稀释表（只适合除去氯的过程）

| 样品 (mL) | 去离子水 (mL) | 范围 (mg/L COD) | 乘法因子 |
|---------|-----------|---------------|------|
| 6.0     | 3.0       | 30—1500       | 1.5  |
| 3.0     | 6.0       | 60—3000       | 3    |
| 1.0     | 8.0       | 180—9000      | 9    |
| 0.5     | 8.5       | 360—18000     | 18   |

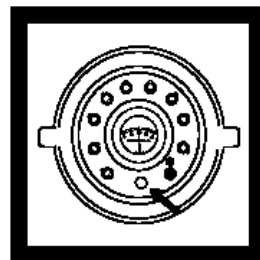
所有稀释液应保持水和硫磺酸比例为9: 1，对于表一中没有列出的稀释液可以简单地将（样品量+去离子水量）/样品量得到乘法因子。



9. 将每个三价锰 COD 试剂瓶注上标签，将其一个个放上真空预处理装置上，

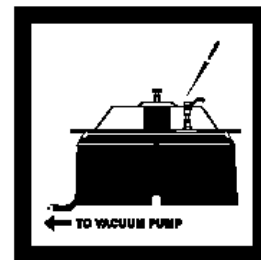


10. 直接在每个试剂瓶上插入一个新的除氯盒（CRC），用所提供地塞子将瓶插入机坐的空座上。



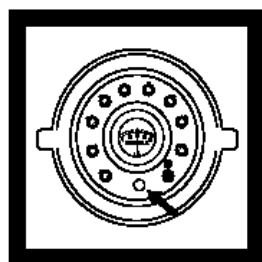
11. 打开真空泵，调节真空阀。直到内置标记读数 20 英寸高水位。

注：最佳的设置是让样品流过 CRC 时间为 30—45 秒。



12. 将 0.6 毫升的酸化样品注入除氯盒（CRC）。  
将 0.6 毫升的酸化空白试样注入另外一个除氯盒。

注：如果样品不流过 CRC，加大真空值直到液体流动。



13. 完全关闭真空调节阀，从而得到完全真空。完全真空一分钟后，打开阀门，除去真空。

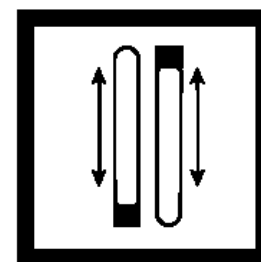


14. 关闭真空泵。移开底座。



15. 使用镊子将每个 CRC 顶部的过滤器移去。将每个过滤器放入相应的三价锰 COD 试剂瓶内。

注：如果样品中不含悬浮固体，不需过滤器。



16. 将试剂瓶从真空架上拿下，盖上瓶盖。反转多次使溶液混合。



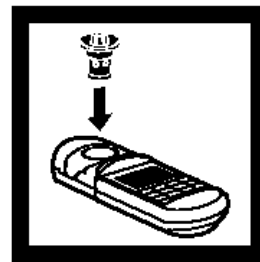
17. 将所有试瓶放上已经预热到150度的COD反应器上,消解处理1小时。



18. 将试瓶移到冷却架,然后在凉水中冷却至室温。这过程一般花费 2 分钟。

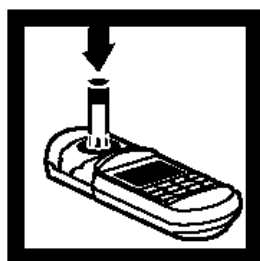


19. 将试瓶从水中拿出,用干布擦干。

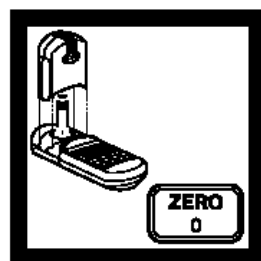


20. 旋转 COD/TNT 适配器,将其嵌入瓶管架上适当的位置,然后下按使之完全嵌入。

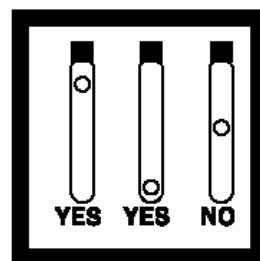
*注: 在消解过程中,如果瓶中试剂沸腾表明试瓶密封不好,此时检测的结果是不正确的。*



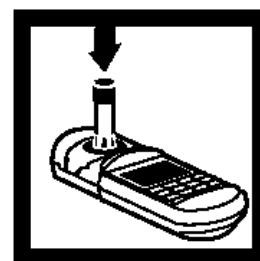
21. 将空白试样放入样品适配器中。



22. 盖紧瓶盖。  
按下: **ZERO**  
屏幕将显示:  
**0 mg/L COD**



23. 如果氯化物已经清除,则滤片将不会处于试瓶中部。它会产生干扰影响结果。轻轻摇晃或往桌面轻轻敲击试瓶,使之移动。



24. 将预制试样放入样品适配器中。



25. 盖紧瓶盖。

26. 按下: **READ**

指针将右移, 屏幕将显示 COD 的含量, 单位是 mg/L。

*注: 应使用预制的标准溶液进行标准校正。*

## 干扰

当数量大的时候, 无机物可能被三价锰氧化, 从而产生正干扰。氯化物是最常见的干扰物质, 应在在样品预处理时将其清除。如果氯化物的数量不多可以不用预处理。一个检测氯是否影响检测结果的简单方法就是分别使用或不使用除氯方法, 比较两者结果。其他无机干扰物 (例如: 亚硝酸盐、三价铁、硫化物) 并不会常产生大的干扰。如果有必要, 这些干扰物可以在检测后用单独方法对最终结果进行相应的校正。当氯化物存在时, 氨氮将产生干扰。

## Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions (Section 1)* for more information.

## Accuracy Check

### Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be 800 ± 26 mg/L COD. An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

## Method Performance (for Manganic III COD without the chloride removal procedure)

### Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 23$  mg/L COD.

### Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Summary of Method

Chemical oxygen demand (COD) is defined as  $\text{mg O}_2/\text{g sample}$ ... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, noncarcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

### 所需试剂

#### 所需试剂

| 试剂种类                          | 所需数量<br>每次测试 | 单位           | 货号       |
|-------------------------------|--------------|--------------|----------|
| 除氯盒 (CRC) .....               | 1 .....      | 25/pkg ..... | 26618-25 |
| 三价锰COD 试剂瓶, 20-1000 mg/L..... | 1 .....      | 25/pkg ..... | 26234-25 |
| 浓缩硫酸.....                     | 1 mL.....    | 4 Kg .....   | 979-09   |
| 去离子水 .....                    | 不定 .....     | 4 L .....    | 272-56   |

### 所需仪器

|                                                     |              |             |          |
|-----------------------------------------------------|--------------|-------------|----------|
| COD/TNT适配器 .....                                    | 1 .....      | 个.....      | 48464-00 |
| 振荡搅拌器, 120 Vac, 14-speed .....                      | 1 .....      | 个.....      | 26747-00 |
| 搅拌器容器, 118 mL.....                                  | 1 .....      | 2/pkg.....  | 26748-00 |
| 瓶盖, with inert Teflon liner, for mixing bottle..... | varies ..... | 12/pkg..... | 24018-12 |
| COD 反应器, 115 V.....                                 | 1 .....      | 个.....      | 45600-00 |
| COD 反应器, 230 V, European-style plug .....           | 1 .....      | 个.....      | 45600-02 |

|                                          |         |             |          |
|------------------------------------------|---------|-------------|----------|
| 镊子, extra fine point .....               | 1.....  | 个.....      | 26696-00 |
| 玻璃混合瓶, for sample + acid.....            | 1.....  | 个.....      | 24276-06 |
| TenSette移液管, 0.1 to 1.0 mL.....          | 1.....  | 个.....      | 19700-01 |
| TenSette移液管, 1.0 to 10.0 mL.....         | 1.....  | 个.....      | 19700-10 |
| 移液管嘴, for 19700-01 TenSette. Pipet ..... | 2.....  | 50/pkg..... | 21856-96 |
| 移液管嘴, for 19700-10 TenSette. Pipet ..... | 2.....  | 50/pkg..... | 21997-96 |
| 安全挡板 .....                               | 1.....  | 个.....      | 23810-00 |
| 不锈钢试管架.....                              | 1.....  | 个.....      | 18641-   |
| 真空预处理装置 (VPD) .....                      | 1 ..... | 个 .....     | 49000-00 |
| 真空泵, 115 V.....                          | 1 ..... | 个 .....     | 14697-00 |
| 真空泵, 230V.....                           | 1 ..... | 个 .....     | 14697-02 |

#### **OPTIONAL REAGENTS AND APPARATUS**

|                                          |              |          |
|------------------------------------------|--------------|----------|
| COD Standard Solution, 800 mg/L COD..... | 200 mL ..... | 26726-29 |
| Potassium Acid Phthalate.....            | 500 g .....  | 315-34   |
| Dispenser for sulfuric acid .....        | each .....   | 25631-37 |

#### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.:**<sup>a</sup>Call 800-227-4224

**Outside the U.S.A.:**<sup>a</sup>Contact the Hach office or distributor serving you.

# 溶解氧 HRDO法 高量程 (0 to 15.0 mg/L O<sub>2</sub>)

方法号: 8166



1. 输入检测高量程的溶解氧程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**



2. 按下: **70 ENTER** 屏幕将显示:

0.00 mg/L、O<sub>2</sub>

和ZERO图标。



3. 将至少 10 毫升的样品注入一支比色瓶中。(空白试样)

往一个蓝色安培盖装入样品。将 40 毫升样品注入一个容积为 50 毫升的烧瓶内。



4. 往一个高含量溶解氧安瓿瓶中装入样品。

*注: 当安培瓶完全充满后, 保持尖端被浸泡。*



5. 不要反转安瓿瓶, 将装有样品的瓶盖稳放在安培瓶的口上。



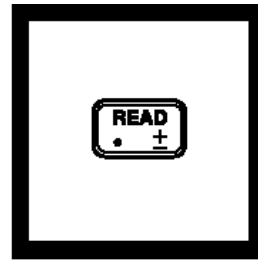
6. 按下: **TIMER ENTER** 将开始2分钟的反应计时。



7. 当反应器鸣响后, 摇晃试剂瓶 30 秒。



8. 将空白试样放入样品适配器中。盖紧瓶盖。



9. 按下: ZERO

指针将右移, 屏幕将显示:

0.0 mg/L O<sub>2</sub>

10. 将安瓿瓶放入样品适配器中。盖紧瓶盖。等待 30 秒, 等气泡完全消散。

11. 按下: READ

指针将右移, 屏幕将显示溶解氧的含量, 单位是 mg/L。

注: 应使用预制标准溶液进行标准校正。

## 干扰

| 干扰物质             | 干扰水平和处理办法                                                                                |
|------------------|------------------------------------------------------------------------------------------|
| Cr <sup>3+</sup> | 大于10mg/L                                                                                 |
| Cu <sup>2+</sup> | 大于10mg/L                                                                                 |
| Fe <sup>2+</sup> | 大于10mg/L                                                                                 |
| Mg <sup>2+</sup> | 镁通常存在海水中, 将会产生负干扰。如果样品存在超过50%的海水, 用该方法所得氧的读数比实际值要低25%。如果样品存在低于50%的海水, 用该方法所得氧的读数比实际值要低5% |
| Mn <sup>2+</sup> | 大于10mg/L                                                                                 |
| Ni <sup>2+</sup> | 大于10mg/L                                                                                 |
| NO <sub>2</sub>  | 大于10mg/L                                                                                 |

## Sampling and Storage

The main consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, it should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards. Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate over-all condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested

immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

### Accuracy Check

The results of this procedure may be compared with the results of a titrimetric procedure (request lit. code number 8042) or dissolved oxygen meter (Cat. No. 50175-00).

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 8.0 mg/L O<sub>2</sub> determined by the Winkler method and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.41$  mg/L O<sub>2</sub>.

#### Estimated Detection Limit

The estimated detection limit for program 70 is 0.10 mg/L O<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, a yellow color forms, which turns purple as the oxygen reacts with the reagent. The color developed is proportional to the concentration of dissolved oxygen.

### 所需试剂

### 所需试剂

| 试剂种类                    | 所需数量<br>每次测试 | 单位          | 货号       |
|-------------------------|--------------|-------------|----------|
| 高量程溶解氧 AccuVac安瓿瓶 ..... | 1 瓶 .....    | 25/pkg..... | 25150-25 |

### 所需仪器

|                                 |          |             |          |
|---------------------------------|----------|-------------|----------|
| 烧杯, 50 mL.....                  | 1.....   | 个.....      | 500-41   |
| 安瓿瓶盖.....                       | 不定 ..... | 25/pkg..... | 1731-25  |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 1.....   | 6/pkg.....  | 24019-06 |

### OPTIONAL REAGENTS AND APPARATUS

|                                                              |                |          |
|--------------------------------------------------------------|----------------|----------|
| AccuVac Dissolved Oxygen Sampler .....                       | each.....      | 24051-00 |
| AccuVac Snapper Kit.....                                     | each.....      | 24052-00 |
| AccuVac Drainer.....                                         | each.....      | 41036-00 |
| BOD bottle and stopper, 300 mL.....                          | each.....      | 621-00   |
| Dissolved Oxygen Meter, <i>ension. 6</i> , Portable .....    | each.....      | 50175-00 |
| Dissolved Oxygen Reagent Set (Buret Method).....             | 100 tests..... | 23514-00 |
| Dissolved Oxygen Reagent Set (Digital Titrator Method) ..... | 50 tests.....  | 22722-00 |

Dissolved oxygen may also be determined by titrimetric methods.

Request Publication 8042 for additional information.

### For Technical Assistance, Price and Ordering

In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 溶解氧 靛蓝胭脂红法 低量程 (0 to 1000 µg/L O<sub>2</sub>)

方法号: 8316



1. 输入检测低量程的溶解氧程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

2. 按下: **71 ENTER**

屏幕将显示:

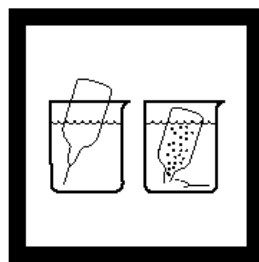
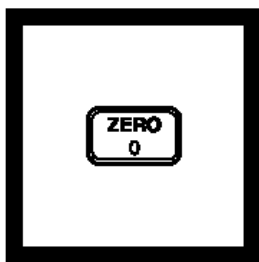
0.00 mg/L、O<sub>2</sub>

和ZERO图标。

3. 将至少 10 毫升的样品注入一支比色瓶中。(空白试样)

4. 将空白试样放入样品适配器中。盖紧瓶盖。

注: 样品应立即分析, 不能存放给以后使用。



5. 按下: **ZERO**

指针将右移, 屏幕将显示:

**0 µg/L O<sub>2</sub>**

10. 往一个容积为50毫升的烧瓶中注入至少四十毫升样品。再往一个低含量溶解氧安瓿瓶中装入样品。

注: 当安瓿瓶完全充满后, 保持尖端被浸泡。

11. 立即将安瓿瓶放入样品适配器中, 盖紧瓶盖。

注: 安瓿瓶中应放有一小块金属丝来维持试剂的质量。溶液显现黄色。

12. 按下: **READ**

指针将右移, 屏幕将显示溶解氧的含量, 单位是µg/L。

注: 使用最初的读数。读数将稳定 30 秒, 之后安瓿瓶溶液将吸收空气中的氧。

## 干扰

| 干扰物质   | 干扰水平和处理办法                  |
|--------|----------------------------|
| 联氨     | 超过100000fold时将会减少指示液的氧化形态。 |
| 连二亚硫酸钠 | 减少指示液的氧化形态, 产生大干扰。         |

过量的巯基乙酸钠、抗坏血酸钠、抗坏血酸钠+亚硫酸钠、抗坏血酸钠+硫酸亚铜、亚硝酸钠、亚硫酸钠、硫代硫酸钠、对苯二酚不会产生大的干扰。

## Sampling and Storage

The main consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

## Accuracy Check

The reagent blank for this test can be checked by following these steps:

- Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.
- Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample and break the tip. Keep the tip immersed while the ampul fills completely.
- Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0  $\pm$  1  $\mu$ g/L.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 500  $\mu$ g/L O<sub>2</sub> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm$  2  $\mu$ g/L O<sub>2</sub>. For more information on Hach's precision statement, see *Section 1*.

### Estimated Detection Limit

The estimated detection limit for program #71 is 10  $\mu$ g/L O<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

When the vacuum-sealed AccuVac ampul is broken open in a sample containing dissolved oxygen, the yellow reagent solution turns blue. The blue color is proportional to the dissolved oxygen concentration.

## 所需试剂和仪器

| 试剂种类                            | 所需数量<br>每次测试 | 单位           | 货号       |
|---------------------------------|--------------|--------------|----------|
| 低量程溶解氧AccuVac安瓿瓶.....           | 1 瓶.....     | 25/pkg ..... | 25010-25 |
| 烧杯, 50 mL.....                  | 1 .....      | 个 .....      | 500-41   |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 1 .....      | 6/pkg .....  | 24019-06 |

## OPTIONAL REAGENTS AND APPARATUS

|                                            |             |          |
|--------------------------------------------|-------------|----------|
| AccuVac Snapper Kit .....                  | each .....  | 24052-00 |
| Sodium Hydrosulfite, technical grade ..... | 500 g ..... | 294-34   |

## For Technical Assistance, Price and Ordering

In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

## 臭氧 靛青法

方法号: 8311

(0 to 0.25 mg/L O<sub>3</sub>, 0 to 0.75 mg/L O<sub>3</sub> or 0 to 1.50 mg/L O<sub>3</sub>)



1. 按“PRGM”键，萤幕会显示 PRGM?



2. 输入内程式代号如下:

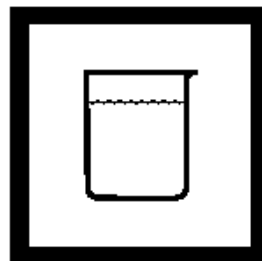
(1) 低浓度范围:  
0~0.25mg/1 时, 请输入“72”, 然后按下“ENTER”键

(2) 中浓度范围:  
0-0.75mg/1 时, 请输入“73”, 然后按下“ENTER”键

(3) 高浓度范围:  
0~1.5mg /1 时, 请输入“74”, 然后按下会出现“mg/1 , O<sub>3</sub>”及“ZERO icon”



3. 慢慢地加入 40ml 水样至一个 50ml 的烧杯中当待测溶液  
\* 水样必须立刻分析, 不可放置太久。



4. 加 40ml 去离子水至另一个 50ml 的烧杯中, 当空白溶液



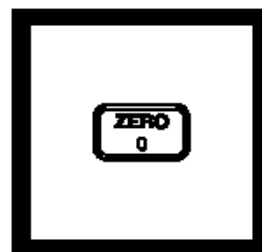
4、取二支真空瓶试药, 如图所示, 分别放入待测及空白溶液。



5 快速地上下颠倒数次, 擦去表面液体或指纹



6、放待测溶液真空瓶至比色计中, 并将比色计盖子盖好。



8、按“ZERO”键归零, 萤幕会显示: 0.00mg/1 O<sub>3</sub>

注: 假如溶液中含臭氧会呈蓝色, 但待测溶液颜色会比空白溶液浅。

注: 该过程的标准方是故意颠倒空白试样和测试试样的顺序。



- 9、放空白溶液真空瓶至至比色计中，并将比色计盖子盖好
- 10、按“READ”键，所欲测浓度将会显示出来即 mg/1 O<sub>3</sub>

### Sampling

The chief consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample or disturbing the sample by stirring or shaking will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

### Stability of Indigo Reagent

Indigo is light-sensitive. Therefore, the AccuVac Ampuls should be kept in the dark at all times. However, the indigo solution decomposes slowly under room light after filling with sample. The blank ampul can be used for multiple measurements during the same day.

### Method Performance

#### Precision

In a single laboratory, using standard solutions of 0.15, 0.28 and 0.96 mg/L ozone for the low, mid and high range, respectively, and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.01$ ,  $\pm 0.02$  and  $\pm 0.02$  mg/L O<sub>3</sub> for the low, mid and high range tests, respectively. For more information on Hach's precision statement, see *Section 1*.

#### Estimated Detection Limit

The estimated detection limit for the programs #72, #73, and #74 is 0.02 mg/L O<sub>3</sub>. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The reagent formulation adjusts the sample pH to 2.5 after the ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone. The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent chlorine interference. No transfer of sample is needed in the procedure. Therefore, ozone loss due to sampling is eliminated.

## 所需试剂

| 试剂种类             | 所需数量<br>每次测试 | 单位           | 货号       |
|------------------|--------------|--------------|----------|
| 臭氧AccuVac安瓿瓶     |              |              |          |
| 选择其中之一：：         |              |              |          |
| 0-0.25 mg/L..... | 2 瓶 .....    | 25/pkg ..... | 25160-25 |
| 0-0.75 mg/L..... | 2 瓶 .....    | 25/pkg ..... | 25170-25 |
| 0-1.50 mg/L..... | 2 瓶 .....    | 25/pkg ..... | 25180-25 |
| 去离子水 .....       | 不定 .....     | 4 L .....    | 272-56   |

## 所需仪器

|                 |         |         |        |
|-----------------|---------|---------|--------|
| 烧杯, 50 mL ..... | 2 ..... | 个 ..... | 500-41 |
|-----------------|---------|---------|--------|

## OPTIONAL APPARATUS

|                              |            |          |
|------------------------------|------------|----------|
| AccuVac Snapper Kit .....    | each ..... | 24052-00 |
| AccuVac Ampule sampler ..... | each ..... | 24051-00 |

## *For Technical Assistance, Price and Ordering*

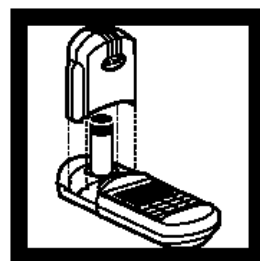
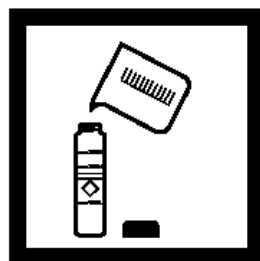
In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

pH (6.5 to 8.5 pH units)

方法号: 10076

使用苯酚检测比色法检测PH值



1. 输入检测PH的程序编号。

按下: PRGM

屏幕将显示:

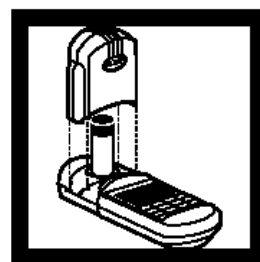
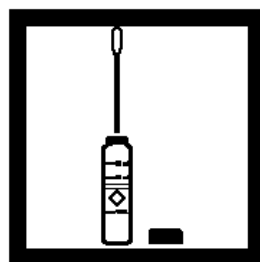
PRGM?

2. 按下: 75 回车  
屏幕将显示:

0.00 mg/L、PH  
和ZERO图标。

3. 往一支比色瓶中装入 10 mL 样品。(空白试样)

4. 将空白试样放入样品适配器中。盖紧瓶盖。



5. 按 ZERO, 指针将右移, 屏幕显示:

0 mg/L PH

6. 往另外一支比色瓶注入 10 毫升样品。(预制试样)

7. 使用点滴器将 1 毫升的酚红指示剂注入预制试样的比色瓶中。

8. 将预制试样放入样品适配器中, 盖紧遮光盖。

注: 盖紧瓶盖, 反转两次使溶液混合。



#### 9. 按下 READ

指针将右移，屏幕将显示 PH 值。

*注：建议对每种新的试剂进行标准校正。*

*注：任何 PH 读数低于 6.5 的都是错误的。*

#### 干扰

当氯含量为6mg/L或以下时，氯不会产生干扰。  
盐水（海水）会产生干扰，不能用该方法检测。

### Sampling and Storage

Analyze samples immediately for best results.

### Accuracy Check

#### Standard Solution Method

Using a clear pH 7.0 buffer solution as the sample, perform the pH procedure as described above.

### Method Performance

#### Precision

In a single laboratory using a standard solution of pH 7.0 and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 0.1 pH units.

#### Estimated Detection Limit

The estimated detection limit for program 75 is a pH of 6.5.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 7.0 buffer solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **7.0** to edit the standard concentration to match that of the standard used. See *Section 1, Standard Curve Adjustment* for more information. Press **ENTER** to complete the curve adjustment.

## Summary of Method

This method uses a sulfonphthalein indicator (Phenol Red) to determine pH colorimetrically. Phenol Red has a working range of pH 6.8 (yellow) to 8.2 (red).

## 所需试剂和仪器

| 试剂种类                             | 所需数量<br>每次测试 | 单位          | 货号       |
|----------------------------------|--------------|-------------|----------|
| 点滴器, 0.5 & 1.0 mL marks .....    | 1.....       | 20/pkg..... | 21247-20 |
| Phenol Red 指示剂, spec grade ..... | 1.0 mL.....  | 50 mL.....  | 26575-12 |
| 样品比色瓶, 10-20-25 mL, w/ cap.....  | 2.....       | 6/pkg.....  | 24019-06 |

## OPTIONAL REAGENTS

pH 7.0 Buffer Solution .....500 mL.....12222-49

## OPTIONAL APPARATUS

### Description Units Cat. No.

Thermometer, -10 to 110 °C..... each.....1877-01

## *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A. Contact the Hach office or distributor serving you.

# 磷酸盐 过硫酸盐紫外光氧化法 (0-2.5 to 1.0-125 mg/L) 方法号: 8007



1. 输入检测磷酸盐的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**
2. 按下: 80 回车  
屏幕将显示:  
0.00 mg/L、**PO<sub>4</sub>**  
和ZERO图标。
3. 按照下表 1 选择适当的样品数量。按所选数量将样品注入 50 毫升刻度的混合量筒中, 用去离子水稀释样品至 50 毫升。  
往一支比色瓶中装入 10 mL 样品。(空白试样)
4. 往一支比色瓶中注入 10 毫升的稀释后样品, 标上标签。(空白试样)。  
往另外一支比色瓶中注入 25 毫升的样品, 注上标签。(预制试样)

注: 为得到更精确的结果, 应用去离子水进行试剂的空白校正。

表1

| 预期范围 (mg/L) 磷酸盐 | 样品数量 |
|-----------------|------|
| 0—2.5           | 50   |
| 0—5             | 25   |
| 0—12.5          | 10   |
| 0—25            | 5    |
| 0—125           | 1    |



5. 往预制试样的瓶中加入一包检测磷酸盐的过硫酸钾试剂粉。摇晃使之混合。



6. 将紫外灯放入装有预制试样的瓶中。

注：灯亮着时，要戴上护目镜。

注：不要接触灯的表面，指纹会腐蚀玻璃。用干净柔软的纸巾擦干净灯才进行下一样品的测定，不能用磷酸盐洗涤剂清洗玻璃仪器。

注：使用特制的适配器 (Cat. No. 19485-00) 可用一个电源同时进行两个样品的消化，需要多加一支紫外灯。



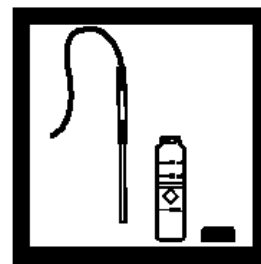
7. 打开紫外灯，消解处理预制试样。

按下：TIMER

将开始十分钟的反应计时。

注：在该过程中，磷酸盐就变成了正磷酸盐。

注：受污染样品或灯太弱会使磷酸盐转化不完全。加长消化时间看读数是否增加可检查转化率。



8. 当计时器鸣响后，拿出紫外灯，打开瓶盖。



9. 将预制试样瓶中的 10 毫升预制试样倒入另外一支干净的比色瓶中。这就是最终预制试样。



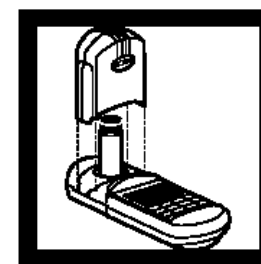
10. 分别将PhosVer 3 磷酸盐试剂粉加入到各个瓶中。迅速搅拌使之混合。

注：如果磷酸盐存在，溶液将显现蓝色。样品和空白试样都会显现颜色。



11. 屏幕将显示：  
**2:00 TIMER 2**  
将开始两分钟的反应计时。

注：如果样品被冷却到 15 度以下，将反应 4 分钟。



12. 计时器鸣响后，将空白试样放入样品适配器中，盖紧瓶盖。

注：在计时器鸣响后三分钟之内要完成步骤 12—15 过程。



13. 按下: **ZERO**  
指针将右移, 屏幕会显示:

**0.0 mg/L PO<sub>4</sub>**

14. 将预制试样放入样品适配器中, 盖紧瓶盖。

15. 按下: **READ**  
指针将右移, 屏幕将显示磷酸盐的读数。从表 2 选取适当的乘法因子, 读数乘以该因子得到实际结果。

16. 结果可根据表3中适当的转换因子将结果表示为实际的活性磷酸盐的浓度。

表2

| 样品数量 (ml) (步骤3所选) | 乘法因子 |
|-------------------|------|
| 50                | 0.1  |
| 25                | 0.2  |
| 10                | 0.5  |
| 5                 | 1.0  |
| 1                 | 5.0  |
| 磷酸盐浓度 = 读数 × 乘法因子 |      |

表3

| 磷酸盐类型                             | 转换系数   |
|-----------------------------------|--------|
| PBTC                              | 2.84   |
| NTP                               | 1.050  |
| HEDPA                             | 1.085  |
| EDTNPA                            | 1.148  |
| HMDTPA                            | 1.295  |
| DETPMPA                           | 1.2087 |
| HPA                               | 1.49   |
| 活性磷酸盐 (mg/L) = 步骤15所得磷酸盐浓度 × 转换因子 |        |

## 干扰

当测试 5mL样品时，以下物质超过表中所列浓度时引起干扰：如样品增大，干扰水平降低。例如，对于5.00mL样品，100 mg/L或低于100 mg/L的铜不干扰；当样品体积增大到10mL，铜在50mg/L以上开始干扰。

| 干扰物质                                 | 干扰水平和处理                |
|--------------------------------------|------------------------|
| 铝，溴化物, CDTA, 铬酸盐, 铜, EDTA, 硅酸盐, 亚硫酸盐 | 100 mg/L               |
| 苯并三唑                                 | 10mg/L                 |
| 砷酸盐                                  | 所有水平上干扰                |
| 重碳酸盐                                 | 1000mg/L               |
| 钙, 氯化物                               | 5000 mg/L              |
| 氰化物                                  | 100 mg/L。紫外消化增加到30分钟。  |
| Diethanoldithiocarbamate             | 50 mg/L                |
| 铁, 硝酸盐                               | 200 mg/L               |
| NTA                                  | 250 mg/L               |
| 正磷酸盐                                 | 15 mg/L                |
| 亚磷酸盐和有机磷化合物                          | 多磷酸盐不干扰。               |
| 高缓冲样品或极端样品pH                         | 可能超过试剂的缓冲能力，需要进行样品预处理。 |
| 硅                                    | 500 mg/L               |
| 硫化物                                  | 所有水平上干扰。               |
| 硫脲                                   | 10 mg/L                |

## Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent. If prompt analysis is impossible, adjust the pH to 2 or less with about 2 mL of sulfuric acid, ACS, per liter of sample. Store at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See *Section 1* for more information on dilution factors, cleaning instructions, etc.

## Accuracy Check

Ideally, a solution containing a known amount of the phosphonate product being used should be prepared. This will check the UV conversion of phosphonate to orthophosphate.

## Summary of Method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV catalyzed oxidation of phosphonate to orthophosphate. Range may be as low as 0 to 2.5 mg/L or as high as 0 to 125 mg/L.

Phosphonate is converted to orthophosphate during the UV digestion. Both the sample and the blank will develop color if orthophosphate is present in the sample. The increase in color in the sample is proportional to the phosphate produced in the digestion.

## 所需试剂

磷酸盐试剂一套 (100 tests)..... 24297-00  
包括: (2) 21060-69, (1) 20847-69

| 试剂种类                    | 所需数量<br>每次测试 | 单位            | 货号       |
|-------------------------|--------------|---------------|----------|
| PhosVer 3 磷酸盐粉末试剂 ..... | 2 包 .....    | 100/pkg ..... | 21060-69 |
| 过(二)硫酸钾粉末试剂.....        | 1 包 .....    | 100/pkg ..... | 20847-69 |
| 去离子水.....               | 不定 .....     | 4 L .....     | 272-56   |

## 所需仪器

|                                 |         |             |          |
|---------------------------------|---------|-------------|----------|
| 混合量筒, 50 mL.....                | 1 ..... | 个 .....     | 1896-41  |
| 护目镜, UV safety.....             | 1 ..... | 个 .....     | 21134-00 |
| 移液管, 25 mL .....                | 1 ..... | 个 .....     | 2066-40  |
| Pipet Filler, safety bulb ..... | 1 ..... | 个 .....     | 14651-00 |
| 样品比色瓶, 10-20-25 mL, w/cap.....  | 2 ..... | 6/pkg ..... | 24019-06 |
| 带电源紫外灯, 115 V.....              | 1 ..... | 个 .....     | 20828-00 |
| 或者                              |         |             |          |
| 带电源紫外灯, 230 V.....              | 1 ..... | 个 .....     | 20828-02 |

## OPTIONAL REAGENTS

|                                     |              |        |
|-------------------------------------|--------------|--------|
| Hydrochloric Acid, 6.0 N (1:1)..... | 500 mL ..... | 884-49 |
| Sulfuric Acid, ACS .....            | 500 mL ..... | 979-49 |

## OPTIONAL APPARATUS

|                                              |                   |          |
|----------------------------------------------|-------------------|----------|
| Cord Adapter, single to dual UV lamp .....   | each .....        | 19485-00 |
| pH Paper, 1 to 11 pH units.....              | 5 rolls/pkg ..... | 391-33   |
| Pipet, serological, 2 mL .....               | each .....        | 532-36   |
| Pipet, TenSette, 1-10 mL .....               | each .....        | 19700-10 |
| Pipet Tips, for 19700-10 Tensette Pipet..... | 50/pkg .....      | 21997-96 |
| Pipet Tips, for 19700-10 Tensette Pipet..... | 1000/pkg .....    | 21997-28 |
| Thermometer, -10 to 110 °C.....              | each .....        | 1877-01  |
| UV Lamp, without power supply .....          | each .....        | 20823-00 |

## *For Technical Assistance, Price and Ordering*

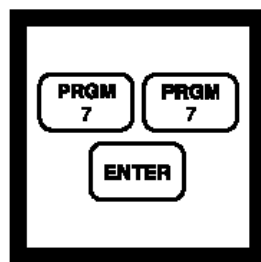
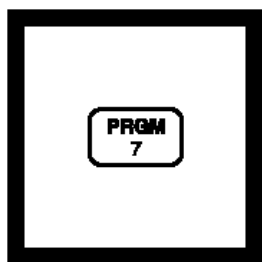
In the U.S.A. call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 活性磷 Molybdovanadate 法 (0 to 45.0 mg/L)

方法号: 8114

## 使用试剂溶液



1. 输入检测高量程磷酸盐的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: 77 ENTER  
屏幕将显示:

PO<sub>4</sub>和ZERO图标。

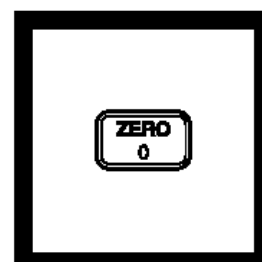
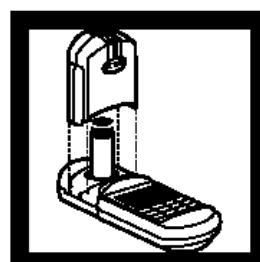
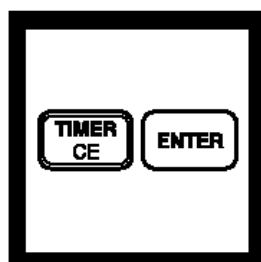
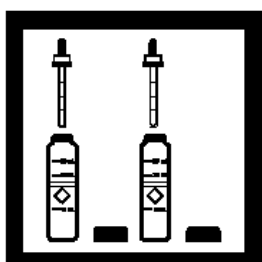
3. 往一支比色瓶中装入 25 mL 去离子水。

(空白试样)

4. 往另外一支比色瓶中注入 25 毫升样品。

(预制试样)

注: 为得最佳结果, 样品的温度范围应在 20-25° C。



5. 加入 1.0 mL Molybdovanadate 试剂到每个比色瓶中。盖上瓶盖, 反转使之混合。

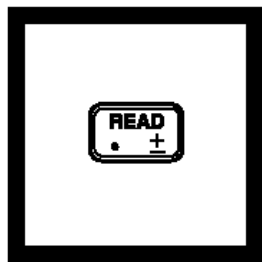
6. 按下: TIMER ENTER  
将开始 5 分钟的反应计时。

7. 将空白试剂放入样品适配器中, 盖紧瓶盖。

8. 按 ZERO, 指针将右移, 屏幕显示:

0.0 mg/L PO<sub>4</sub>

注: 如果存在磷酸盐, 预制试样溶液将显现黄色。由于试剂的原因, 空白试样也会呈现少量的黄色。



9. 将预制试样放入样品适配器中，盖紧瓶盖。
10. 按下：**READ**  
指针将右移，屏幕会显示磷酸盐的含量，单位是 mg/L。

*注：建议对每种新的试剂都进行标准校正。*

## 干扰

干扰物质和建议处理方法

| 干扰物质                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 干扰水平和处理方法                                                                                              |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|
| 砷酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 只有样品被加热时产生干扰。                                                                                          |
| 铁离子                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 如果铁离子浓度小于100g/L，由于铁产生的蓝色物是不会产生干扰。                                                                      |
| 钼酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 大于1000g/L时，产生负干扰。                                                                                      |
| 硅                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | 只有样品被加热时产生干扰。                                                                                          |
| 硫化物                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 产生负干扰。如下方法去除干扰：<br>1. 量取50毫升样品到锥形烧瓶。<br>2. 逐滴加入溴水，同时不停搅拌直到溶液呈现稳定的黄色。<br>3. 逐滴加入苯酚溶液，直到黄色消失。然后再继续步骤4过程。 |
| 过高或过低PH值和高缓冲样品                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 也许是超过了试剂的缓冲能力。样品需要进行预处理，请看预处理部分内容。理想的样品PH值应为7左右。                                                       |
| 氟化物、钍、铋、硫代硫酸盐、硫氰酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 产生负干扰。                                                                                                 |
| <p>如果含量不高于1000mg/L时，下列物质不会产生干扰。</p> <p>焦磷酸盐、四硼酸盐、 硒酸盐、 安息香酸盐、柠檬酸盐、草酸盐、乳酸盐、酒石酸盐、甲酸盐、水杨酸盐、铝离子、三价铁离子、镁离子、钙离子、 Ba<sup>2+</sup>、 Sr<sup>2+</sup>、 Li<sup>+</sup>、 Na<sup>+</sup>、 K<sup>+</sup>、 NH<sub>4</sub><sup>+</sup>、 Cd<sup>2+</sup>、 Mn<sup>2+</sup>、 NO<sub>3</sub><sup>-</sup>、 NO<sub>2</sub><sup>-</sup>、 SO<sub>4</sub><sup>2-</sup>、 SO<sub>3</sub><sup>2-</sup>、 Pb<sup>2+</sup>、 Hg<sup>+</sup>、 Hg<sup>2+</sup>、 Sn<sup>2+</sup>、 Cu<sup>2+</sup>、 Ni<sup>2+</sup>、 Ag<sup>+</sup>、 U<sup>4+</sup>、 Zr<sup>4+</sup>、 AsO<sub>3</sub><sup>-</sup>、 Br<sup>-</sup>、 CO<sub>3</sub><sup>2-</sup>、 ClO<sub>4</sub><sup>-</sup>、 CN<sup>-</sup>、 IO<sub>3</sub><sup>-</sup>、 SiO<sub>4</sub><sup>-</sup>。</p> |                                                                                                        |

## Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is impossible, preserve samples by filtering immediately and storing at 4 °C for up to 48 hours.

## Accuracy Check

### Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as PO<sub>4</sub>.
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and invert to mix well.
- d) For analysis with AccuVac Ampuls, transfer the spiked samples to clean, dry 50-mL beakers to facilitate filling of the ampuls. For analysis with reagent solution, transfer the spiked samples to 25-mL sample cells.
- e) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L PO<sub>4</sub><sup>3-</sup>.
- f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

### Standard Solution Method

Obtain a Hach Phosphate Standard Solution, 10.0 mg/L as phosphate. Using this solution as the sample, perform the phosphate procedure as described above.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* for more information.

## Method Performance

### Precision

In a single laboratory using a standard solution of 30.0 mg/L PO<sub>4</sub><sup>3-</sup>, two lots of reagent, and the instrument, a single operator obtained a standard deviation of ±0.1 mg/L PO<sub>4</sub><sup>3-</sup> for the reagent solution method and a standard deviation of ±0.2 for the AccuVac Ampul method.

### Estimated Detection Limit

The estimated detection limit for program 77 is 0.3 mg/L PO<sub>4</sub><sup>3-</sup> and 0.4 mg/L PO<sub>4</sub><sup>3-</sup> for program 78. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

## 所需试剂和仪器 (使用试剂溶液)

| 试剂种类                            | 所需数量<br>每次测试 | 单位               | 货号       |
|---------------------------------|--------------|------------------|----------|
| Molybdovanadate 试剂.....         | 2.0 mL       | 100 mL* MDB..... | 20760-32 |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 2            | 6/pkg.....       | 24019-06 |
| 去离子水.....                       | 25 mL        | 4 L              | 272-56   |

## 所需试剂和仪器 (使用安瓿瓶)

|                            |       |             |          |
|----------------------------|-------|-------------|----------|
| Molybdovanadate试剂安瓿瓶 ..... | 2     | 25/pkg..... | 25250-25 |
| 烧杯, 50 mL.....             | 2     | 个.....      | 500-41   |
| 去离子水.....                  | 25 mL | 4 L         | 272-56   |

## OPTIONAL REAGENTS

| Description                                                                                              | Units            | Cat. No. |
|----------------------------------------------------------------------------------------------------------|------------------|----------|
| Bromine Water, 30 g/L.....                                                                               | 29 mL*           | 2211-20  |
| Hydrochloric Acid Solution, 1:1 (6.0 N) .....                                                            | 500 mL.....      | 884-49   |
| Phenol Solution, 30 g/L .....                                                                            | 29 mL.....       | 2112-20  |
| Phosphate Standard Solution, 10.0 mg/L as PO <sub>4</sub> <sup>3-</sup> .....                            | 946 mL.....      | 14204-16 |
| Phosphate Standard Solution, Voluette Ampule,<br>500 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL ..... | 16/pkg.....      | 14242-10 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                          | 100 mL* MDB..... | 2450-32  |
| Sulfuric Acid, ACS .....                                                                                 | 500 mL*.....     | 979-49   |

## OPTIONAL APPARATUS

|                                                  |                   |          |
|--------------------------------------------------|-------------------|----------|
| AccuVac Snapper Kit.....                         | each.....         | 24052-00 |
| Ampule Breaker Kit.....                          | each.....         | 21968-00 |
| Cylinder, graduated, 25 mL .....                 | each .....        | 508-40   |
| Cylinder, graduated, mixing, 25-mL.....          | each.....         | 20886-40 |
| Dispenser, fixed volume, 1.0 mL Repipet Jr. .... | each.....         | 21113-02 |
| Flask, erlenmeyer, 50 mL .....                   | each.....         | 505-41   |
| Flask, volumetric, Class A, 50 mL .....          | each.....         | 14574-41 |
| pH Paper, 1 to 11 pH units.....                  | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>Sension.1</i> , portable.....       | each.....         | 51700-00 |
| Pipet, serological, 2.0 mL .....                 | each.....         | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL.....              | each.....         | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....    | 50/pkg.....       | 21856-96 |
| Thermometer, -10 to 110 °C.....                  | each.....         | 1877-01  |

## For Technical Assistance, Price and Ordering

In the U.S.A.: Call 800-227-4224

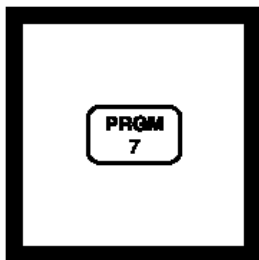
Outside the U.S.A.: Contact the Hach office or distributor serving you.

## 活性磷(正磷酸盐) PhosVer3 抗坏血酸法

方法号: 8048

(0. ~2. 50 mg/L  $\text{PO}_4^{3-}$ ) (试剂粉包或安培瓶)

试剂粉包



1. 输入检测活性磷抗坏血酸法的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

2. 按下: **79 ENTER**  
屏幕将显示:

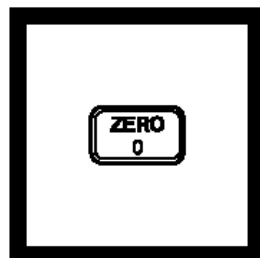
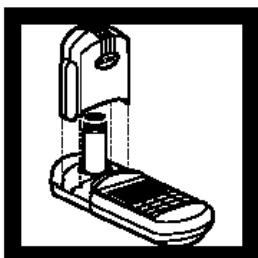
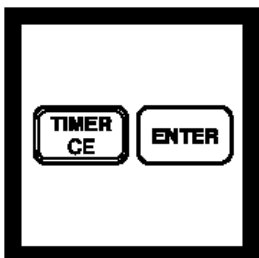
**PO4**和**ZERO**图标。

3. 往一支比色瓶中装入 10 mL 样品。

注: 对于过高或过低 PH 值的样品, 请参考干扰部分的内容。

4. 将一包PhosVer 3 磷酸盐试剂粉加入到比色瓶所装的样品中。  
(预制试样)摇晃十五秒钟。

注: 如果存在磷酸盐, 溶液将显现蓝色。



5. 按下:

**TIMER ENTER**

将开始 2 分钟的反应计时。在此期间, 应执行步骤 6—8 的过程

6. 往另外一支比色瓶中注入 10 毫升的未加试剂的原始样品。  
(空白试样)

7. 将空白试剂放入样品适配器中, 盖紧瓶盖。

8. 按 **ZERO**, 指针将右移, 屏幕显示:

**0.0 mg/L PO4**



9. 将预制试样放入样品适配器中，盖紧瓶盖。
10. 按下：READ  
指针将右移，屏幕会显示磷酸盐的含量，单位是 mg/L。

注：应试样磷酸盐标准溶液进行标准校正。

## 干扰

如果浓度超过下表所列的情况，下列物质将产生干扰。

| 干扰物质         | 干扰水平和处理                                                                                                    |
|--------------|------------------------------------------------------------------------------------------------------------|
| 铝            | 大于200 mg/L                                                                                                 |
| 砷酸盐          | 所有水平上干扰                                                                                                    |
| 铬，铁          | 大于100mg/L                                                                                                  |
| 铜，硅酸盐        | 大于10mg/L                                                                                                   |
| 镍            | 大于300 mg/L                                                                                                 |
| 硅            | 大于50mg/L                                                                                                   |
| 硫化物          | 大于6 mg/L。按下法除去硫化物干扰：<br>1. 量取25mL样品到50mL大口中。<br>2. 一边摇晃一边逐滴加入溴水直到出现持久的黄色。<br>3. 一边摇晃一边逐滴加入苯酚溶液直黄色消失。继续步骤1。 |
| 浊度或色度        | 由于粉包中的酸会溶解部分悬浮粒子并且正磷酸盐对粒子不定的解吸附作用，高浊度会引起结果不一致。                                                             |
| 锌            | 大于80 mg/L                                                                                                  |
| 高缓冲样品或极端样品pH | 可能超过试剂的缓冲能力，需要进行样品预处理。                                                                                     |

## Sampling and Storage

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing samples at 4 °C. Warm to room temperature before testing.

## Accuracy Check

### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off a Phosphate PourRite Ampule Standard Solution, 50 mg/L as  $\text{PO}_4$
- Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.
- For analysis with AccuVacs, transfer solutions to dry, clean 50 mL beakers to fill the AccuVac ampules. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.
- Analyze each standard addition sample as described in the procedure. The phosphate concentration should increase 0.2 mg/L  $\text{PO}_4$  for each 0.1 mL of standard added. **f)** If these increases do not occur, see *Standard Additions in Section 1*.

### Standard Solution Method

Prepare a 2.0 mg/L  $\text{PO}_4$  standard solution by pipetting 4.0 mL of Phosphate Standard Solution, 50 mg/L as  $\text{PO}_4$ , into an acid-washed Class A 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix. Use this solution in place of the sample in the procedure to insure the accuracy of the test. The mg/L  $\text{PO}_4$  reading should be 2.00 mg/L.

## Method Performance

### Precision

In a single laboratory using a standard solution of 1.00 mg/L  $\text{PO}_4$  and two lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.05$  mg/L  $\text{PO}_4$ . In a single laboratory using a standard solution of 1.00 mg/L  $\text{PO}_4$  and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 0.03$  mg/L  $\text{PO}_4$ .

### Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L  $\text{PO}_4$ . For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

### 所需试剂和仪器 (使用试剂溶液)

| 试剂种类                            | 所需数量<br>每次测试 | 单位           | 货号       |
|---------------------------------|--------------|--------------|----------|
| PhosVer 3 磷酸盐粉末试剂               |              |              |          |
| 10 mL sample size .....         | 1 包 .....    | 100/pkg..... | 21060-69 |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 .....      | 6/pkg.....   | 24019-06 |
| 所需试剂和仪器 (使用安瓿瓶)                 |              |              |          |
| PhosVer 3 磷酸盐AccuVac 安瓿瓶.....   | 1 瓶 .....    | 25/pkg.....  | 25080-25 |
| 烧杯, 50 mL .....                 | 1 .....      | 个 .....      | 500-41   |

|                                 |         |             |          |
|---------------------------------|---------|-------------|----------|
| 瓶盖 .....                        | 1 ..... | 25/pkg..... | 1731-25  |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 1.....  | 6/pkg.....  | 24019-06 |

### OPTIONAL REAGENTS

|                                                                                                        |                  |          |
|--------------------------------------------------------------------------------------------------------|------------------|----------|
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                                 | 500 mL.....      | 884-49   |
| Phosphate Pretreatment Powder Pillows .....                                                            | 50/pkg.....      | 14501-66 |
| Phosphate Standard Solution, PourRite ampule,<br>50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL ..... | 20/pkg.....      | 171-20   |
| Phosphate Standard Solution, Voluette Ampul, 50 mg/L, 10 mL.....                                       | 16/pkg.....      | 171-10   |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                        | 100 mL* MDB..... | 2450-32  |
| Water, deionized .....                                                                                 | 4 L.....         | 272-56   |

### OPTIONAL APPARATUS

|                                                             |                   |          |
|-------------------------------------------------------------|-------------------|----------|
| AccuVac Snapper Kit .....                                   | each .....        | 24052-00 |
| Ampule Breaker Kit for 10-ml ampules.....                   | each.....         | 21968-00 |
| Aspirator, vacuum .....                                     | each.....         | 2131-00  |
| Cylinder, graduated, mixing, 25 mL, tall (3 required) ..... | each.....         | 20886-40 |
| Filter Holder, 47 mm, 300 mL, graduated.....                | each.....         | 13529-00 |
| Filter, membrane, 47 mm, 0.45 microns .....                 | 100/pkg.....      | 13530-00 |
| Flask, filtering, 500 mL.....                               | each.....         | 546-49   |
| Flask, volumetric, Class A, 100 mL.....                     | each.....         | 14574-42 |
| pH Indicator Paper, 1 to 11 pH .....                        | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>Sens<sup>ion</sup>.1</i> , portable .....      | each.....         | 51700-00 |
| Pipet, 2 mL serological .....                               | each.....         | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL TenSette Pipet .....         | each.....         | 19700-01 |
| Pipet Tips, for 19700-01 .....                              | 50/pkg.....       | 21856-96 |
| Pipet Filler, safety bulb .....                             | each.....         | 14651-00 |
| Pipet, volumetric, Class A, 4.00 mL .....                   | each.....         | 14515-04 |
| PourRite Ampule Breaker Kit .....                           | each.....         | 24846-00 |

### ***For Technical Assistance, Prices and Ordering***

In the U.S.A. call 800-227-4224

Outside the U.S.A. Contact the Hach office or distributor serving you.

## 总磷酸盐 酸消解法

方法号：8190



1、加入 25ml 水样至 50ml 锥形瓶。



2、加一高锰酸钾试剂摇动使充分混合。



3、再加入 2ml 5.25N 硫酸至水样中。



8、将水样加热，缓慢沸腾 30 分钟。

注：使用浓度 1: 1 的盐酸清洗所有玻璃器皿，再以去离子水冲洗，切不可使用含磷的清洗剂清洗。

注：加热过程中，水样若少于 20ml，则加入去离子水，使水样保持在 20ml。



5、将水样冷却至室温。



6、加入 2ml 5N 氢氧化钠至水样中，摇动使充分混合。



3、将处理好的水样倒入量筒中，可加入去离子水，使水样维持有 25ml。

注：\*此反应所测得之磷酸盐浓度值=有机磷酸盐值+正磷酸盐值+酸解浓缩磷酸盐值。

\*此消化步骤，即将磷酸盐氧化成正磷酸盐，之后再依欲测正磷酸盐浓度范围选择试剂，测正磷酸盐。

## Sampling and Storage

Collect samples in plastic or glass bottles that have been acid-washed with 1:1 HCl and rinsed with deionized water. Do not use detergents containing phosphates for cleaning glassware used in this test. Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm to room temperature before testing. Correct results for volume additions; see *Volume Additions* (Section 1) for more information.

## Interferences

For turbid samples, use 50 mL of sample and double the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to add additional acid in Step 3 to drop the pH of the solution below 1.

## Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus(orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA approved for NPDES reporting.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

## 所需试剂

| 试剂种类               | 所需数量<br>每次测试 | 单位                | 货号      |
|--------------------|--------------|-------------------|---------|
| 过硫酸钾粉末试剂 .....     | 1 包.....     | 100/pkg .....     | 2451-99 |
| 氢氧化钠溶液, 5.0 N..... | 2 mL . ....  | 100 mL* MDB ..... | 2450-32 |
| 硫磺酸溶液, 5.25 N..... | 2 mL .....   | 100 mL* MDB ..... | 2449-32 |

## 所需仪器

|                                 |         |             |          |
|---------------------------------|---------|-------------|----------|
| 量筒, 25 mL.....                  | 2 ..... | 个 .....     | 508-40   |
| 锥形烧瓶, 50 mL.....                | 1 ..... | 个 .....     | 505-41   |
| 样品比色瓶, 10-20-25 mL, w/caps..... | 2 ..... | 6/pkg ..... | 24019-06 |

## OPTIONAL REAGENTS

|                             |              |        |
|-----------------------------|--------------|--------|
| Hydrochloric Acid, 6 N..... | 500 mL ..... | 884-49 |
|-----------------------------|--------------|--------|

|                                       |              |         |
|---------------------------------------|--------------|---------|
| Sodium Hydroxide Solution, 5.0 N..... | 1 L .....    | 2450-53 |
| Sulfuric Acid .....                   | 500 mL ..... | 979-49  |
| Water, deionized .....                | 4 L .....    | 272-56  |

#### **OPTIONAL APPARATUS**

| <b>Description</b>                         | <b>Unit</b>       | <b>Cat. No.</b> |
|--------------------------------------------|-------------------|-----------------|
| Cylinder, graduated, 50 mL .....           | each .....        | 508-41          |
| Flask, erlenmeyer, 125 mL .....            | each .....        | 505-43          |
| Hot Plate, 4" diameter, 120 Vac .....      | each .....        | 12067-01        |
| Hot Plate, 4" diameter, 240 Vac .....      | each .....        | 12067-02        |
| Pads, cooling, 4 x 4" .....                | each .....        | 18376-00        |
| pH Indicator Paper, 1 to 11 pH .....       | 5 rolls/pkg ..... | 391-33          |
| pH Meter, <i>Sension.1</i> , portable..... | each .....        | 51700-00        |

#### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.;**<sup>a</sup>Call 800-227-4224

**Outside the U.S.A.;**<sup>a</sup>Contact the Hach office or distributor serving you.

## 磷 酸水解

## 正磷酸盐水解法

方法号: 8180



1. 使用有刻度的量筒, 量取 25 毫升的样品到容积为 50 毫升的锥形瓶中。



2. 加入2毫升的5.25N的硫磺酸。



3. 将烧瓶放在加热盘上。温火加热 30 分钟。



4. 冷却加热的烧瓶到室温。

*注: 使用 6N 的盐酸清洗所有玻璃器皿。再用去离子水清洗。不使用含有磷酸盐的清洁剂去清洗。*



5. 往预制试样中加入 2 毫升的 5.0N 氢氧化钠溶液。



6. 将预制试样倒入一个有刻度的量筒中。加入清洗过烧瓶的去离子水到量筒中, 使样品重新达到25毫升的数量。然后选择适当的活性磷的检测方法进行检测。

*注: \*此反应所测得之磷酸盐浓度值=正磷酸盐值+酸解浓缩磷酸盐值。浓缩磷酸盐值等于这个结果减去用未经预处理的活性磷样品进行检测所得的值。*

### 干扰

如果样品浑浊, 使用 50 毫升的样品和 2 倍的试剂。在检测活性磷的过程中, 使用水解样品调零仪器。这样就可使浑浊物溶解从而补偿其带来的干扰。

## Sampling and Storage

Analyze samples immediately after collection for best results. If prompt analysis is not possible, samples may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

## Summary of Method

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to reactive orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

### 所需试剂

| 试剂种类                | 所需数量<br>每次测试 | 单位                | 货号      |
|---------------------|--------------|-------------------|---------|
| 氢氧化钠溶液, 5.0 N ..... | 2 mL .....   | 100 mL* MDB.....  | 2450-32 |
| 硫酸溶液, 5.25 N .....  | 2 mL .....   | 100 mL* MDB ..... | 2449-32 |

### 所需仪器

|                   |         |         |        |
|-------------------|---------|---------|--------|
| 量筒, 25 mL .....   | 2 ..... | 个 ..... | 508-40 |
| 锥形烧瓶, 50 mL ..... | 1 ..... | 个 ..... | 505-41 |

## OPTIONAL REAGENTS

|                              |              |        |
|------------------------------|--------------|--------|
| Hydrochloric Acid, 6 N ..... | 500 mL ..... | 884-49 |
| Water, deionized .....       | 4 L .....    | 272-56 |

## OPTIONAL APPARATUS

|                                            |                   |          |
|--------------------------------------------|-------------------|----------|
| Cylinder, graduated, 50 mL .....           | each .....        | 508-41   |
| Flask, erlenmeyer, 125 mL .....            | each .....        | 505-43   |
| Hot Plate, 4" diameter, 120 Vac .....      | each .....        | 12067-01 |
| Hot Plate, 4" diameter, 240 Vac .....      | each .....        | 12067-02 |
| Pad, cooling, 4" x 4" .....                | each .....        | 18376-00 |
| pH indicator Paper, 1 to 11 pH .....       | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>senion.1</i> , portable ..... | each .....        | 51700-00 |
| Thermometer, -10 to 110 °C.....            | each.....         | 1877-01  |

### *For Technical Assistance, Price and Ordering*

In the U.S.A.: Call 800-227-4224

Outside the U.S.A.: Contact the Hach office or distributor serving you.

# 活性磷 PhosVer 3 法 (0.00 to 5.00 mg/L PO<sub>4</sub><sup>3-</sup>)

方法号: 8048

## Test 'N Tube 过程



1. 输入检测活性磷的  
**Test 'N tube**的程序  
编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到根据精确  
的结果, 应使用去离  
子水进行试剂空白校  
正。

2. 按下: **82 ENTER**  
屏幕将显示:

**mg/L, PO4D和ZERO**图  
标。

注: 如果检测其他形  
态, 按下:

**CONC**键

3. 旋转COD/TNT适配  
器, 将其嵌入瓶管架  
上适当的位置, 然后  
下按使之完全嵌入。

4. 使用一支TenSette  
移液管将5毫升的样品  
注入活性磷Test 'N Tube  
稀释瓶中。盖上瓶盖使  
溶液混合。



5. 用布擦干净试瓶的  
外壁。

6. 将样瓶放入样品适  
配器中。

7. 盖紧瓶盖。

8. 按下: **ZERO**  
指针将右移, 屏幕会显  
示:

**0.00 mg/L PO4**

注: 如检测多个样品,  
只需在第一次调零即  
可。



9. 使用漏斗，将一包 PhosVer 3 磷酸盐试剂粉加入到试剂瓶中。



10. 盖上瓶盖，摇晃 10—15 秒钟。



11. 按下：  
**TIMER CE** **ENTER**  
将开始 2 分钟的反应计时。



12. 在计时器鸣响后，马上将试验瓶放入样品适配器中。

*注：在加入 PhosVer 3 试剂后 2—8 分钟内读数。*

*注：如果存在硝酸盐，溶液将呈现蓝色。*



13. 盖紧瓶盖。



14. 按下：**READ**  
指针将右移，屏幕会显示磷酸盐的浓度，单位是 mg/L。

*注：使用预制标准溶液进行标准校正。*

## 干扰

如果浓度超过下表所列的情况，下列物质将产生干扰。

| 干扰物质         | 干扰水平和处理                                                                                                     |
|--------------|-------------------------------------------------------------------------------------------------------------|
| 铝            | 大于200 mg/L                                                                                                  |
| 砷酸盐          | 所有水平上干扰                                                                                                     |
| 铬，铁          | 大于100mg/L                                                                                                   |
| 铜，硅酸盐        | 大于10mg/L                                                                                                    |
| 镍            | 大于300 mg/L                                                                                                  |
| 硅            | 大于50mg/L                                                                                                    |
| 硫化物          | 大于6 mg/L。按下法除去硫化物干扰：<br>4. 量取25mL样品到50mL大口杯中。<br>5. 一边摇晃一边逐滴加入溴水直到出现持久的黄色。<br>6. 一边摇晃一边逐滴加入苯酚溶液直黄色消失。继续步骤1。 |
| 浊度或色度        | 由于粉包中的酸会溶解部分悬浮粒子并且正磷酸盐对粒子不定的解吸附作用，高浊度会引起结果不一致。                                                              |
| 锌            | 大于80 mg/L                                                                                                   |
| 高缓冲样品或极端样品pH | 可能超过试剂的缓冲能力，需要进行样品预处理。                                                                                      |

## Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing at 4 °C. Warm to room temperature before analyzing the sample.

## Accuracy Check

**Note:** Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.
- Analyze each sample as described in the procedure; use 5.0 mL of the prepared standard additions for each test. The concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, prepare a 1.0-mg/L  $\text{PO}_4^{3-}$  standard by pipetting 2 mL of solution from a Phosphate Voluette Ampule Standard for Phosphate, 50 mg/L as  $\text{PO}_4^{3-}$ , into an

acid-washed, Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 5.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.08$  mg/L  $\text{PO}_4^{3-}$ .

### Estimated Detection Limit (EDL)

The EDL for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

## Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

## Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

## 所需试剂

活性磷 Test  $\text{PO}_4^{3-}$  N Tube 试剂一套.....50 tests ..... 27425-45  
包括: (1) 21060-46, (50) 正磷酸盐稀释瓶\*

| 试剂种类                                           | 所需数量<br>每次测试 | 单位     | 货号       |
|------------------------------------------------|--------------|--------|----------|
| PhosVer 3 磷酸盐粉末试剂 .....                        | 1            | 50/pkg | 21060-46 |
| 50正磷酸盐Test $\text{PO}_4^{3-}$ N Tube 稀释瓶 ..... | 1            | 50/pkg | *        |

## 所需仪器

|                                         |     |        |          |
|-----------------------------------------|-----|--------|----------|
| COD/TNT 适配器 .....                       | 1   | 个      | 48464-00 |
| 漏斗.....                                 | 1   | 个      | 25843-35 |
| TenSette移液管, 1 to 10 mL.....            | 1   | 个      | 19700-10 |
| 移液管嘴, for 19700-10 TenSette Pipet ..... | 1   | 50/pkg | 21997-96 |
| 试管架.....                                | 1-3 | 个      | 18641-00 |

## OPTIONAL REAGENTS

|                                                                                             |        |         |
|---------------------------------------------------------------------------------------------|--------|---------|
| Bromine Water, 30 g/L.....                                                                  | 29 mL  | 2211-20 |
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                      | 500 mL | 884-49  |
| Phenol Solution, 30 g/L .....                                                               | 29 mL  | 2112-20 |
| Phosphate Standard Solution, 1 mg/L as $\text{PO}_4^{3-}$ .....                             | 500 mL | 2569-49 |
| Phosphate Standard Solution, Voluette ampule,<br>50 mg/L as $\text{PO}_4^{3-}$ , 10mL ..... | 16/pkg | 171-10  |
| Phosphate Standard Solution, PourRite ampule,<br>50 mg/L as $\text{PO}_4^{3-}$ , 2 mL ..... | 20/pkg | 171-20  |

Water, deionized..... 4 L ..... 272-56

# **OPTIONAL APPARATUS**

Ampule Breaker, Pour Rite (2-mL ampule) ..... each.....24846-00  
 Ampule Breaker Kit ..... each.....21968-00  
 Aspirator, vacuum ..... each.....2131-00  
 Cylinder, graduated, mixing, 25 mL (3 required) ..... each.....20886-40  
 Dispenser, Repipet Jr., 2 mL ..... each.....22307-01  
 Filter Holder, 47 mm, 300 mL, graduated..... each.....13529-00  
 Filter, membrane, 47 mm, 0.45 microns ..... 100/pkg.....13530-00  
 Flask, filtering, 500 mL..... each.....546-49  
 Flask, volumetric, Class A, 100 mL..... each.....14574-42  
 pH Indicator Paper, 1 to 11 pH units..... 5 rolls/pkg.....391-33  
 pH Meter, *sens***ion.1**, portable..... each.....51700-00  
 Pipet, TenSette, 0.1 to 1.0 mL..... each.....19700-01  
 Pipet Tips, for 19700-01..... 50 pkg.....21856-96  
 Pipet Filler, Safety Bulb ..... each.....14651-00  
 Pipet, volumetric, Class A, 5.00 mL ..... each.....14515-37  
 Pipet, volumetric, Class A, 2.00 mL ..... each.....14515-36

## ***For Technical Assistance, Price and Ordering***

In the U.S.A. call 800-227-4224

Outside the U.S.A. Contact the Hach office or distributor serving you.

## 总磷 PhosVer3 过硫酸消解法 (0.00 to 3.50 mg/L $\text{PO}_4^{3-}$ ) 方法号: 8190

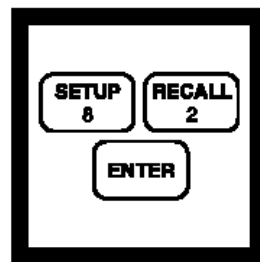
### Test 'N Tube 过程



1. 开启 COD 反应炉，加热至 103–106 °C，将塑料隔板置于反应炉前。



2. 输入检测总磷的预程序编号。  
按下: **PRGM**  
屏幕详细: **PRGM?**



3. 按下: **82 ENTER**  
屏幕将会显示:  
**mg/L, PO4**和**ZERO**图标



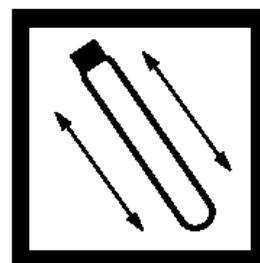
4. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



5. 使用移液管将 5.0mL 试样装入试管中总磷酸性水解检测试瓶。



6. 使用漏斗将一包过硫酸钾试剂粉加入试瓶中。



7. 盖紧瓶盖。晃动使粉剂溶解。



8. 将试样放入 COD 反应器上。加热 30 分钟。



9. 小心的将试瓶从反应器拿出，然后放在试瓶架上，冷却到室温。



10. 使用移液管将2毫升的1.45N的氢氧化钠溶液注入试瓶。



11. 用毛巾擦干净试瓶外壁。



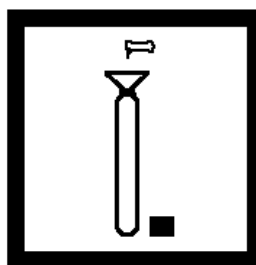
12. 将样品放入样品适配器中。盖紧瓶盖。



13. 用遮光盖盖紧瓶盖。



14. 按下：ZERO  
指针将右移，屏幕会显示：  
**0.00 mg/L PO4**



15. 打开瓶盖。使用漏斗将一包PhosVer 3 磷酸盐试剂粉放入试瓶。



16. 盖紧瓶盖，摇晃 10—15 秒钟。

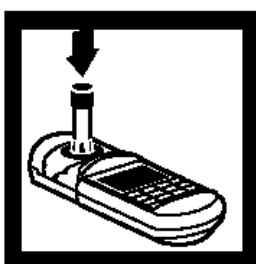
*注：粉剂将不会完全溶解。*



17. 按下：  
**TIMER ENTER**  
将开始两分钟等待时间。



18. 计时器鸣响后，用毛巾擦干净试瓶外壁。



19. 将预制试样瓶放入样品适配器中。



20. 盖紧瓶盖。

*注：在加入PhosVer 3 试剂后 2 至 8 分钟内读数。*

*注：如果磷酸盐存在，溶液将显现蓝色。*



#### 21. 按下：READ

指针将右移，屏幕会显示磷酸盐的浓度，单位是 mg/L。

注：应使用预制标准溶液进行标准校正。

#### 重要注释：

该测试的总磷的浓度范围是0到3.5mg/L。超过3.5mg/L的是用作估计稀释比例，但不是为读数所用。如果超过3.5mg/L，稀释样品，重复消解处理和比色检测。

### 干扰

在干冷环境下保存PhosVer 3磷试剂粉包。

| 干扰物质      | 干扰水平和处理                                        |
|-----------|------------------------------------------------|
| 铝         | 大于200 mg/L                                     |
| 砷酸盐       | 所有水平上干扰                                        |
| 铬         | 大于100 mg/L                                     |
| 铜         | 大于10 mg/L                                      |
| 铁         | 大于100 mg/L                                     |
| 镍         | 大于300 mg/L                                     |
| pH,过度缓冲   | 高缓冲样品或极端样品pH可能超过试剂的缓冲能力，需要进行样品预处理              |
| 硅         | 大于50 mg/L                                      |
| 硅酸盐       | 大于10 mg/L                                      |
| 硫化物       | 大于90 mg/L                                      |
| 浑浊（大量）或颜色 | 由于粉包中的酸会溶解某些悬浮微粒，并且由于正磷酸盐对微粒有可变的吸收，所以会引起读数不稳定。 |
| 锌         | 大于80 mg/L                                      |

### Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with

concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Neutralize and warm the sample to room temperature before analysis. Correct test results for volume additions; see *Volume Additions* in *Section 1*.

### Accuracy Check

**Note:** Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

#### Standard Additions Method

- a) Fill three 25 mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.
- d) Analyze each sample as described in the procedure using 5.0 mL of the prepared standard additions for each test. The concentration should increase 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- e) If these increases do not occur, see *Standard Additions* (Section 1).

#### Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution (see Optional Reagents). Or, prepare a standard by pipetting 2 mL of solution a Voluette Ampule Standard for Phosphate Standard, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid-cleaned Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described. The mg/L  $\text{PO}_4^{3-}$  reading should be 1.0 mg/L. OR Prepare a 2.5 mg/L standard solution by pipetting 5 mL of a 50-mg/L Phosphate Voluette Ampule Standard into an acid-washed 100-mL Class A volumetric flask. Dilute to the mark with deionized water.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 3.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.06$  mg/L  $\text{PO}_4^{3-}$ .

#### Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1*.

### Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

### Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

## 所需试剂

总磷 Test j<sup>-</sup>N Tube 试剂一套.....50 tests ..... 27426-45  
包括: (1) 272-56, (1) 20847-66, (1) 21060-46, (1) 27430-42, (50) 酸稀释瓶 \*

| 试剂种类                                  | 所需数量<br>每次测试 | 单位             | 货号       |
|---------------------------------------|--------------|----------------|----------|
| PhosVer 3 磷酸盐粉末试剂 .....               | 1 .....      | 50/pkg .....   | 21060-46 |
| 过硫酸钾粉末试剂.....                         | 1 .....      | 50/pkg .....   | 20847-66 |
| 氢氧化钠溶液, 1.54 N .....                  | 2 mL .....   | 100 mL .....   | 27430-42 |
| Test j <sup>-</sup> N Tube 酸稀释瓶 ..... | 1 .....      | 50/pkg .....   | *        |
| 去离子水.....                             | 100 mL ..... | 4 liters ..... | 272-56   |

## 所需仪器

|                                                |           |              |          |
|------------------------------------------------|-----------|--------------|----------|
| COD 反应器, 115/230 Vac (U.S.A. and Canada) ..... | 1 .....   | 个 .....      | 45600-00 |
| COD 反应器, 115/230 Vac (Europe) .....            | 1 .....   | 个 .....      | 45600-02 |
| COD/TNT 适配器 .....                              | 1 .....   | 个 .....      | 48464-00 |
| 漏斗.....                                        | 1 .....   | 个 .....      | 25843-35 |
| 安全挡板 .....                                     | 1 .....   | 个 .....      | 23810-00 |
| 试管架.....                                       | 1-3 ..... | 个 .....      | 18641-00 |
| TenSette移液管, 1 to 10 mL.....                   | 1 .....   | 个 .....      | 19700-10 |
| 移液管嘴, for 19700-10 TenSette Pipet .....        | 不定 .....  | 50/pkg ..... | 21997-96 |

## OPTIONAL REAGENTS

| Description                                                                                             | Unit         | Cat. No. |
|---------------------------------------------------------------------------------------------------------|--------------|----------|
| Total and Acid Hydrolyzable Test j <sup>-</sup> N Tube Reagent Set .....                                | each.....    | 27427-45 |
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                                  | 500 mL ..... | 884-49   |
| Phosphate Standard Solution, 1 mg/L as PO <sub>4</sub> <sup>3-</sup> .....                              | 500 mL ..... | 2569-49  |
| Phosphate Standard Solution, PourRite ampule,<br>50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....  | 20/pkg ..... | 171-20   |
| Phosphate Standard Solution, Voluette ampule,<br>50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL ..... | 16/pkg ..... | 171-10   |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                         | 1 L.....     | 2450-53  |
| Water, deionized .....                                                                                  | 4 L .....    | 272-56   |

## OPTIONAL APPARATUS

|                                                       |                  |          |
|-------------------------------------------------------|------------------|----------|
| Ampule Breaker Kit .....                              | each.....        | 21968-00 |
| Ampule Breaker, PourRite ampules .....                | each.....        | 24846-00 |
| Cylinder, graduated, mixing, 25 mL (3 required) ..... | each.....        | 20886-40 |
| pH Indicator Paper, 1 to 11 pH units.....             | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>Sension.1</i> , portable .....           | each.....        | 51700-00 |
| Pipet Filler, safety bulb.....                        | each.....        | 14651-00 |
| Pipet, volumetric, Class A, 5.00 mL .....             | each.....        | 14515-37 |
| Pipet, volumetric, Class A, 2.00 mL .....             | each.....        | 14515-36 |
| Pipet, TenSette, 0.1-1.0 mL.....                      | each.....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....         | 50/pkg.....      | 21856-96 |

## For Technical Assistance, Price and Ordering

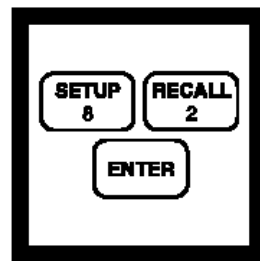
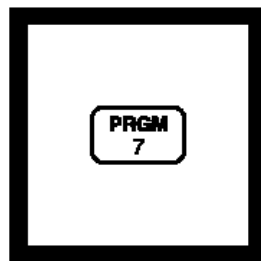
In the U.S.A. call 800-227-4224

Outside the U.S.A.:\*Contact the Hach office or distributor serving

磷 酸水解法 (0.00 to 5.00 mg/L  $\text{PO}_4^{3-}$ )

方法号: 8180

PhosVer 3水解法  
Test 'N Tube

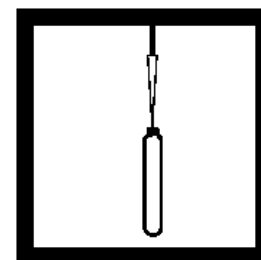
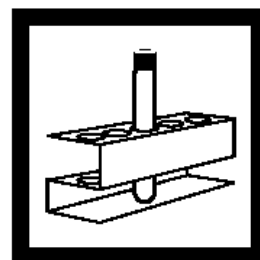


1. 开启 COD 反应炉，加热至 103-106℃，将塑料隔板置于反应炉前。

2. 输入检测磷酸水解法的预制程序编号。  
按下: **PRGM**  
屏幕详细: **PRGM?**

3. 按下: **82 ENTER**  
屏幕将会显示:  
**mg/L, PO4和ZERO**图  
标

4. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



5. 使用移液管将 5.0mL 试样装入试管中总磷酸性水解检测试瓶。

6. 试瓶放在反应器上加热30分钟。

7. 将试瓶移出，放置在试管架上，冷却到室温。

8. 打开瓶盖，滴入 2 毫升的 1N 氢氧化钠溶液。盖上瓶盖混合溶液。



9. 用布擦干净瓶外壁。



10. 将样品放入适配器中。



11. 盖紧瓶盖。  
按下: **ZERO**  
指针将右移, 屏幕会显示:

**0.00 mg/L PO<sub>4</sub>**



12. 移去瓶盖。往试瓶中加入 *PhosVer 3* 试剂粉。

*注: 如检测多个样品, 应在第一次调零, 加入 *PhosVer 3* 试剂后读取数据, 每次的读数减去试剂空白值。*



13. 盖上瓶盖摇晃 10 到 15 秒钟。



14. 按下:  
**TIMER ENTER**  
将开始两分钟等待时间。



15. 计时器鸣响后, 用毛巾擦干净试瓶外壁。



16. 将预制试样瓶放入样品适配器中

*注: 在加入 *PhosVer 3* 试剂后 2 至 8 分钟内读数。*

*注: 如果磷酸盐存在, 溶液将显现蓝色。*



17. 盖紧瓶盖。



18. 按下：READ  
指针将右移，屏幕会  
显示磷酸盐的浓度，  
单位是 mg/L。

*注：应使用预制标准  
溶液进行标准校正。*

## 干扰

| 干扰物质      | 干扰水平和处理                                        |
|-----------|------------------------------------------------|
| 铝         | 大于200 mg/L                                     |
| 砷酸盐       | 所有水平上干扰                                        |
| 铬         | 大于100 mg/L                                     |
| 铜         | 大于10 mg/L                                      |
| 铁         | 大于100 mg/L                                     |
| 镍         | 大于300 mg/L                                     |
| pH,过度缓冲   | 高缓冲样品或极端样品pH可能超过试剂的缓冲能力，需要进行样品预处理              |
| 硅         | 大于50 mg/L                                      |
| 硅酸盐       | 大于10 mg/L                                      |
| 硫化物       | 大于90 mg/L                                      |
| 浑浊（大量）或颜色 | 由于粉包中的酸会溶解某些悬浮微粒，并且由于正磷酸盐对微粒有可变的吸收，所以会引起读数不稳定。 |
| 锌         | 大于80 mg/L                                      |

在干冷环境下保存PhosVer 3磷试剂粉包。

## Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use commercial detergents containing phosphate for cleaning glassware used in this test. Analyze samples immediately after collection for best results. If prompt analysis is impossible, the sample may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

## Accuracy Check

**Note:** Clean glassware with 1:1 hydrochloric acid solution. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.

### Standard Additions Method

- Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as  $\text{PO}_4^{3-}$ .
- Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.
- Analyze each sample as described in the procedure. Use 5.0 mL of the prepared standard additions for each test; the concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L  $\text{PO}_4^{3-}$ , respectively.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Obtain a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, this can be prepared by pipetting 2 mL of a Voluette Ampule Standard for Phosphate, 50 mg/L as  $\text{PO}_4^{3-}$ , into an acid washed Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 3.00 mg/L  $\text{PO}_4^{3-}$  and two lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.06$  mg/L  $\text{PO}_4^{3-}$ .

### Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L  $\text{PO}_4^{3-}$ . For more information on the estimated detection limit, see *Section 1*.

## Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

## Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of

the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

## REQUIRED REAGENTS

### 所需试剂

总磷可酸化 Test i<sup>-</sup>N Tube 试剂1套.....50tests .....27427-45  
包括: (1) 272-56, (1) 1045-42, (1) 20847-66, (1) 21060-46, (50) 总磷可酸化瓶

| 试剂种类                                  | 所需数量  |        |  | 货号       |
|---------------------------------------|-------|--------|--|----------|
|                                       | 每次测试  | 单位     |  |          |
| PhosVer 3 磷酸盐粉末试剂 .....               | 1     | 50/pkg |  | 21060-46 |
| 过硫酸钾粉末试剂.....                         | 1     | 50/pkg |  | 20847-66 |
| 氢氧化钠溶液, 1.54 N .....                  | 2 mL  | 100 mL |  | 27430-42 |
| Test i <sup>-</sup> N Tube 酸稀释瓶 ..... | 1     | 50/pkg |  | *        |
| 去离子水.....                             | 100mL | 4升     |  | 272-56   |

## REQUIRED APPARATUS

### 所需仪器

|                                                |     |        |  |          |
|------------------------------------------------|-----|--------|--|----------|
| COD 反应器, 115/230 Vac (U.S.A. and Canada) ..... | 1   | 个      |  | 45600-00 |
| COD 反应器, 115/230 Vac (Europe) .....            | 1   | 个      |  | 45600-02 |
| COD/TNT 适配器 .....                              | 1   | 个      |  | 48464-00 |
| 漏斗.....                                        | 1   | 个      |  | 25843-35 |
| 安全挡板 .....                                     | 1   | 个      |  | 23810-00 |
| 试管架.....                                       | 1-3 | 个      |  | 18641-00 |
| TenSette移液管, 1 to 10 mL.....                   | 1   | 个      |  | 19700-10 |
| 移液管嘴, for 19700-10 TenSette Pipet .....        | 不定  | 50/pkg |  | 21997-96 |

## OPTIONAL REAGENTS

|                                                                                                         |         |  |  |         |
|---------------------------------------------------------------------------------------------------------|---------|--|--|---------|
| Bromine Water, 30 g/L.....                                                                              | 29 mL   |  |  | 2211-20 |
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                                  | 500 mL  |  |  | 884-49  |
| Phenol Solution, 30 g/L .....                                                                           | 29 mL   |  |  | 2112-20 |
| Phosphate Standard Solution, 1 mg/L as PO <sub>4</sub> <sup>3-</sup> .....                              | 500 mL  |  |  | 2569-49 |
| Phosphate Standard Solution, PourRite ampule,<br>50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 2 mL .....  | 20/pkg  |  |  | 171-20  |
| Phosphate Standard Solution, Voluette ampule,<br>50 mg/L as PO <sub>4</sub> <sup>3-</sup> , 10 mL ..... | 16/pkg  |  |  | 171-10  |
| Sodium Hydroxide Standard Solution, 5.000 N .....                                                       | 1000 mL |  |  | 2450-53 |
| Sulfuric Acid Standard Solution, 1.000 N .....                                                          | 1 L     |  |  | 1270-53 |
| Water, deionized.....                                                                                   | 4 L     |  |  | 272-56  |

## OPTIONAL APPARATUS

| Description                                            | Units             | Cat. No. |
|--------------------------------------------------------|-------------------|----------|
| Ampule Breaker Kit, Voluette.....                      | each .....        | 21968-00 |
| Ampule Breaker, PourRite.....                          | each .....        | 24846-00 |
| Cylinder, graduated, mixing, 25 mL (3 required) .....  | each .....        | 20886-40 |
| Flask, volumetric, Class A, 100 mL .....               | each .....        | 14574-42 |
| pH Indicator Paper, 1 to 11 pH units .....             | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>sens<sup>ion</sup>.1</i> , portable ..... | each .....        | 51700-00 |
| Pipet, TenSette, 0.1-1.0 mL .....                      | each .....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....          | 50/pkg .....      | 21856-96 |
| Pipet, volumetric, Class A, 5.00 mL.....               | each .....        | 14515-37 |
| Pipet, volumetric, Class A, 2.00 mL.....               | each .....        | 14515-36 |
| Pipet Filler, safety bulb .....                        | each .....        | 14651-00 |

### ***For Technical Assistance, Price and Ordering***

**In the U.S.A. call 800-227-4224**

**Outside the U.S.A. Contact the Hach office or distributor serving you.**

# 活性磷 氨基酸法 (0 to 30.00 mg/L $\text{PO}_4^{3-}$ )

方法号: 8178



1. 输入检测活性磷的氨基酸法的预设程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 为得到根据精确的结果, 应使用去离子水进行试剂空白校正。



2. 按下: 85 ENTER  
屏幕将显示:

mg/L,  $\text{PO}_4$ 和ZERO图

注: 如果检测其他形态, 按下:

CONC键



3. 往比色瓶中注入 25 毫升的样品。



4. 使用一毫升的校准点滴器加入1毫升钼酸盐试剂。



5. 加入 1 毫升的氨基酸试剂。盖上瓶盖, 反转多次使溶液混合。(预制试样)



6. 按下:  
TIMER ENTER  
将开始10分钟的反应计时。

注: 在计时期间, 进行步骤7过程。



7. 往另外一支比色瓶中注入 25 毫升的样品 (空白试样)。



8. 当计时器鸣响后, 屏幕会显示:  
**mg/L  $\text{PO}_4$**   
将空白试样放入样品适配器中, 盖紧瓶盖。



9. 按下: ZERO

指针将右移, 屏幕会显示:

**0.00 mg/L PO<sub>4</sub>**

10. 将预制试样瓶放入样品适配器中, 盖紧瓶盖。

11. 按下: READ

指针将右移, 屏幕会显示活性磷的浓度, 单位是 mg/L。

*注: 使用预制标准溶液进行标准校正。*

### 干扰

| 干扰物质         | 干扰水平和处理                                                                                                                              |
|--------------|--------------------------------------------------------------------------------------------------------------------------------------|
| 钙            | 大于10,000 mg/L 的 CaCO <sub>3</sub>                                                                                                    |
| 氯化物          | 大于150,000 mg/L的 Cl <sup>-</sup>                                                                                                      |
| 有颜色样品        | 加入1 mL 10 N硫酸标准溶液到另一个25mL样品中, 用此代替作为空白的未处理样品对仪器调零。用移液管和pipet filler 量取硫酸标准溶液。                                                        |
| 高盐水平         | 引起结果偏低。要除去干扰, 稀释样品直到连续两次稀释产生几乎相同的结果。                                                                                                 |
| 镁            | 大于 40,000 mg/L 的 CaCO <sub>3</sub> 。                                                                                                 |
| 亚硝酸盐         | 使蓝色褪去。除去亚硝酸盐干扰可加入0.50g sulfamic酸到样品中, 混合, 继续步骤4。                                                                                     |
| 高水平磷酸盐       | 磷酸盐浓度增加, 颜色从蓝色变成黄色最终至棕色。棕色表明磷酸盐浓度高达100,000 mg/L。如果形成的不是蓝色, 稀释样品, 重新测试。                                                               |
| 硫化物          | 干扰。对于5mg/L以下的硫化物浓度的干扰可按下法溴水氧化除去:<br>1. 量取25mL样品到比色瓶中。<br>2. 一边摇晃一边逐滴加入溴水直到出现持久的黄色。<br>3. 一边摇晃一边逐滴加入苯酚溶液直黄色消失。<br>4. 继续步骤4。           |
| 温度           | 为得到最佳结果, 样品温度应为21±3° C。                                                                                                              |
| 浊度           | 由于粉包中的酸会溶解部分悬浮粒子并且正磷酸盐对粒子不定的解吸附作用, 高浊度会引起结果不一致。对高浊度样品, 加入1mL 10 N 硫酸标准溶液到另一个25mL样品中, 用此代替作为空白的未处理样品对仪器调零。用移液管和pipet filler 量取硫酸标准溶液。 |
| 高缓冲样品或极端样品pH | 可能超过试剂的缓冲能力, 需要进行样品预处理。                                                                                                              |

## Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) for up to 48 hours.

## Accuracy Check

### Standard Additions Method

- Snap the neck off a Phosphate PourRite Ampule Standard Solution, 500 mg/L as PO<sub>4</sub><sup>3-</sup>.
- Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples. Mix well.
- Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L orthophosphate (PO<sub>4</sub><sup>3-</sup>).
- If these increases do not occur, see *Standard Additions (Section 1)* for more information.

### Standard Solution Method

Prepare a 10.0-mg/L phosphate standard by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L as PO<sub>4</sub><sup>3-</sup> into a 50-mL volumetric flask. Dilute to volume with deionized water. Or, prepare a 10.0-mg/L PO<sub>4</sub><sup>3-</sup> standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate PourRite Ampule Standard, 500 mg/L as PO<sub>4</sub><sup>3-</sup>, into a 50-mL volumetric flask. Dilute to volume with deionized water. Substitute this standard for the sample and perform the test as described. The mg/L PO<sub>4</sub><sup>3-</sup> reading should be 10 mg/L.

## Method Performance

### Precision

In a single laboratory using a standard solution of 15.0 mg/L PO<sub>4</sub><sup>3-</sup> and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.12 mg/L PO<sub>4</sub><sup>3-</sup>.

### Estimated Detection Limit

The estimated detection limit for program 85 is 0.14 mg/L PO<sub>4</sub><sup>3-</sup>. For more information on the estimated detection limit, see *Section 1*.

## 所需试剂

高量程活性磷试剂一套 (100 Test).....22441-00  
包括: (1) 1934-32, (1) 2236-32

| 试剂种类        | 所需数量<br>每次测试 | 单位          | 货号      |
|-------------|--------------|-------------|---------|
| 氨基酸试剂.....  | 1 mL ...     | 100 mL MDB* | 1934-32 |
| 钼酸盐试剂 ..... | 1 mL ...     | 100 mL MDB* | 2236-32 |

## 所需仪器

样品比色瓶, 10-20-25 mL, w/ cap .....2 .....6/pkg .....24019-06

## OPTIONAL REAGENTS

| Description                                                                                          | Units      | Cat. No. |
|------------------------------------------------------------------------------------------------------|------------|----------|
| Amino Acid Reagent Powder Pillow .....                                                               | 100/pkg    | 804-99   |
| Bromine Water, 30 g/L .....                                                                          | 29 mL      | 2211-20  |
| Hydrochloric Acid Solution, 1:1 (6 N) .....                                                          | 500 mL     | 884-49   |
| Phenol Solution, 30 g/L .....                                                                        | 29 mL      | 2112-20  |
| Phosphate Standard Solution, 50 mg/L PO <sub>4</sub> <sup>3-</sup> .....                             | 500 mL     | 171-49   |
| Phosphate Standard Solution, PourRite ampule,<br>500 mg/L PO <sub>4</sub> <sup>3-</sup> , 2 mL ..... | 20/pkg     | 14242-20 |
| Sodium Hydroxide Standard Solution, 5.0 N .....                                                      | 100 mL MDB | 2450-32  |
| Sulfamic Acid .....                                                                                  | 113 g      | 2344-14  |
| Sulfuric Acid Standard Solution, 10 N .....                                                          | 1 L        | 931-53   |
| Water, deionized .....                                                                               | 4 L        | 272-56   |

## OPTIONAL APPARATUS

| Description                                            | Unit              | Cat. No. |
|--------------------------------------------------------|-------------------|----------|
| Ampule Breaker Kit, PourRite.....                      | each.....         | 24846-00 |
| Aspirator, vacuum.....                                 | each.....         | 2131-00  |
| Cylinder, graduated, 50 mL .....                       | each.....         | 508-41   |
| Cylinder, graduated, mixing, 25 mL.....                | each.....         | 20886-40 |
| Filter Holder, 47 mm, 300 mL, graduated .....          | each.....         | 13529-00 |
| Filter, membrane, 47 mm, 0.45 microns.....             | 100/pkg.....      | 13530-00 |
| Flask, filtering, 500 mL .....                         | each.....         | 546-49   |
| Flask, erlenmeyer, 125 mL .....                        | each .....        | 505-43   |
| Flask, volumetric, Class A, 50 mL .....                | each .....        | 14574-41 |
| pH Indicator Paper, 1 to 11 pH .....                   | 5 rolls/pkg ..... | 391-33   |
| pH Meter, <i>sensio<sup>n</sup>.1</i> , portable ..... | each .....        | 51700-00 |
| Pipet, serological, 2.0 mL .....                       | each .....        | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL .....                   | each .....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....          | 50/pkg .....      | 21856-96 |
| Pipet, volumetric, Class A, 10.00 mL .....             | each .....        | 14515-38 |
| Pipet Filler, safety bulb .....                        | each .....        | 12189-00 |
| Spoon, measuring, 0.05 g .....                         | each .....        | 492-00   |
| Thermometer, -10 to 110 j°C.....                       | each.....         | 1877-01  |

### *For Technical Assistance, Price and Ordering*

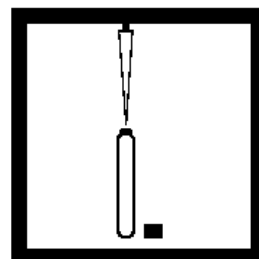
In the U.S.A.;<sup>a</sup>Call 800-227-4224

Outside the U.S.A.;<sup>a</sup>Contact the Hach office or distributor serving you.

# 活性磷 Molybdovanadate 法 高量程

方法号: 8114

Test 'N Tube 法 (0 to 100.0 mg/L  $\text{PO}_4^{3-}$ )



1. 输入检测活性磷的高量程的预设程序编号。

按下: **PRGM**  
屏幕将显示:

**PRGM?**

注: 为得到根据精确的结果, 应使用去离子水进行试剂空白校正。

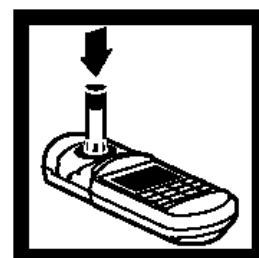
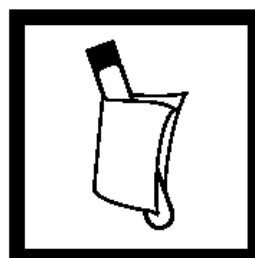
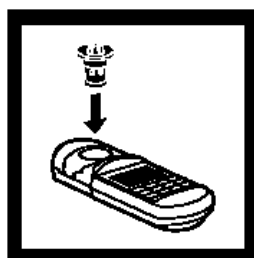
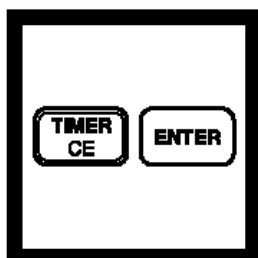
2. 按下: **85 ENTER**  
屏幕将显示:  
**mg/L, PO4D**和**ZERO**图标。

注: 如果检测其他形态, 按下:

**CONC**键

3. 使用 TenSette 移液管, 往一支活性磷 Test 'N 试瓶注入 5 毫升的去离子水。(空白试样)

4. 使用 TenSette 移液管, 往另外一支活性磷 Test 'N 试瓶注入 5 毫升的样品。(预制试样)



5. 按下:

**TIMER ENTER**

将开始 7 分钟的反应计时。

注: 该反应时间是对样品温度为 23°C。

如果样品温度为 13°C, 反应时间为 15 分钟。如果样品温度为 33°C, 反应时间为 2 分钟。

6. 按下:

**TIMER ENTER**

将开始 10 分钟的反应计时。

注: 在计时期间, 进行步骤 7 过程。

7. 用毛巾擦干净每个试瓶的外壁。。

8. 当计时器鸣响后, 将空白试样瓶放入样品适配器中。



9. 盖紧遮光盖。



10. 按下：ZERO  
指针将右移，屏幕会  
显示：  
**0.00 mg/L PO<sub>4</sub>**



11. 将预制试样瓶放  
入样品适配器中，盖  
紧瓶盖。。



13. 按下：READ  
指针将右移，屏幕会  
显示活性磷的浓度，  
单位是 mg/L。

*注：使用预制标准溶  
液进行标准校正。*

## 干扰

| 干扰物质                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 干扰水平和处理                                                                                               |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------|
| 砷酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 如果样品被加热，将会产生正干扰。                                                                                      |
| 铁，亚铁                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 小于100mg/L，不产生干扰。L                                                                                     |
| 钼酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 大于100mg/L时，产生负干扰。                                                                                     |
| 硅                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | 如果样品被加热，会产生正干扰。                                                                                       |
| 硫化物                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 产生负干扰。按下法除去硫化物干扰：<br>5. 量取50mL样品到锥形烧瓶中。<br>6. 一边摇晃一边逐滴加入溴水直到出现持久的黄色。<br>7. 一边摇晃一边逐滴加入苯酚溶液直黄色消失。继续步骤1。 |
| 高缓冲样品或极端样品pH                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 可能超过试剂的缓冲能力，需要进行样品预处理。                                                                                |
| 氟化物、钍、铋、硫代硫酸盐、硫氰酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 产生负干扰。                                                                                                |
| 温度，冷（低于20 °C）                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | 产生负干扰。                                                                                                |
| 温度，热（高于25 °C）                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             | 产生正干扰。                                                                                                |
| 下列物质含量不大于1000mg/L时，不产生干扰。<br>焦磷酸盐、四硼酸盐、 硒酸盐、 安息香酸盐、柠檬酸盐、草酸盐、乳酸盐、酒石酸盐、甲酸盐、水杨酸盐、铝离子、三价铁离子、镁离子、钙离子、 Ba <sup>2+</sup> 、 Sr <sup>2+</sup> 、 Li <sup>+</sup> 、 Na <sup>+</sup> 、 K <sup>+</sup> 、 NH <sub>4</sub> <sup>+</sup> 、 Cd <sup>2+</sup> 、 Mn <sup>2+</sup> 、 NO <sub>3</sub> <sup>-</sup> 、 NO <sub>2</sub> <sup>-</sup> 、 SO <sub>4</sub> <sup>2-</sup> 、 SO <sub>3</sub> <sup>2-</sup> 、 Pb <sup>2+</sup> 、 Hg <sup>+</sup> 、 Hg <sup>2+</sup> 、 Sn <sup>2+</sup> 、 Cu <sup>2+</sup> 、 Ni <sup>2+</sup> 、 Ag <sup>+</sup> 、 U <sup>4+</sup> 、 Zr <sup>4+</sup> 、 AsO <sub>3</sub> <sup>-</sup> 、 Br <sup>-</sup> 、 CO <sub>3</sub> <sup>2-</sup> 、 ClO <sub>4</sub> <sup>-</sup> 、 CN <sup>-</sup> 、 IO <sub>3</sub> <sup>-</sup> 、 SiO <sub>4</sub> <sup>-</sup> 。 |                                                                                                       |

## Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning the glassware used in this test.

For best results, analyze the samples immediately after collection. If prompt analysis is impossible, preserve the samples for up to 748 hours by filtering immediately and storing at 4 °C. The sample should have a neutral (6–8) pH and be at room temperature before analysis.

## Accuracy Check

**Note:** Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

### Standard Additions Method

- Fill three 10-mL graduated mixing cylinders with 10 mL of sample.
- Snap the neck off a Voluette™ Ampule of Phosphate Standard Solution, 500 mg/L as PO<sub>4</sub> (Cat. No. 14242-10).
- Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of sample prepared in *step a*. Mix well.
- Analyze each sample from *step c* as described in the procedure; use 5.0 mL of the prepared sample for each test. The concentration should increase as follows: 5 mg/L, 10 mg/L, and 15 mg/L PO<sub>4</sub>, respectively.

e. If these increases do not occur, see *Standard Additions* in *Section 1* of the *DR/890 Procedures Manual* for more information.

#### **Standard Solution Method**

To check accuracy, prepare an 80 mg/L PO<sub>4</sub> standard by pipetting 8.0 mL of solution from a 10-mL Voluette Ampule of Phosphate Standard Solution, 500 mg/L as PO<sub>4</sub>, into an acid-cleaned 50-mL Class A volumetric flask. Fill to the line with deionized water. Substitute this standard for the sample and perform the procedure as described.

#### **Standard Adjust**

To adjust the calibration curve using the reading obtained with the 80 mg/mL PO<sub>4</sub> standard solution, press the **SETUP** key and scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

## **Method Performance**

#### **Precision**

In a single laboratory, using a standard solution of 80.0 mg/L PO<sub>4</sub> and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO<sub>4</sub>.

#### **Estimated Detection Limit (EDL)**

The EDL for program 86 is 7.0 mg/L PO<sub>4</sub>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

## **Sample Disposal Information**

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

## **Safety**

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

## **Summary of Method**

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

## **Installing this Program on the DR/800**

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.

2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

| Line Number | Entry | Line Number | Entry |
|-------------|-------|-------------|-------|
| 1           | 86    | 29          | 0     |
| 2           | 4     | 30          | 80    |
| 3           | 73    | 31          | 50    |
| 4           | 0     | 32          | 79    |
| 5           | 0     | 33          | 53    |
| 6           | 0     | 34          | 0     |
| 7           | 0     | 35          | 62    |
| 8           | 65    | 36          | 166   |
| 9           | 56    | 37          | 246   |
| 10          | 217   | 38          | 148   |
| 11          | 21    | 39          | 63    |
| 12          | 66    | 40          | 63    |
| 13          | 157   | 41          | 78    |
| 14          | 197   | 42          | 252   |
| 15          | 30    | 43          | 4     |
| 16          | 0     | 44          | 76    |
| 17          | 0     | 45          | 128   |
| 18          | 0     | 46          | 0     |
| 19          | 0     | 47          | 15    |
| 20          | 80    | 48          | 1     |
| 21          | 79    | 49          | 164   |
| 22          | 52    | 50          | 0     |
| 23          | 0     | 51          | 0     |
| 24          | 0     | 52          | 0     |
| 25          | 80    | 53          | 0     |
| 26          | 0     | 54          | 80    |
| 27          | 0     | 55          | 0     |
| 28          | 0     | 56          | 255   |

## 所需试剂

高量程活性磷 Test 'N Tube™ 试剂一套.....50 vials.....27673-45  
包括: (50)高量程活性磷 Test 'N Tube™试剂瓶\*, (2) 272-42

| 试剂种类                          | 所需数量<br>每次测试 | 单位          | 货号     |
|-------------------------------|--------------|-------------|--------|
| 高量程活性磷 Test 'N Tube™ 试剂瓶..... | 1.....       | 50/pkg..... | *      |
| 去离子水.....                     |              | 100 mL..... | 272-42 |

## 所需仪器

|                                           |          |             |          |
|-------------------------------------------|----------|-------------|----------|
| COD/TNT 适配器.....                          | 1.....   | 个.....      | 48464-00 |
| TenSette®移液管, 1 to 10 mL.....             | 1.....   | 个.....      | 19700-10 |
| 移液管嘴s, for 19700-10 TenSette® Pipet ..... | 1.....   | 50/pkg..... | 21997-96 |
| 试管架.....                                  | 1-3..... | 个.....      | 18641-00 |

## OPTIONAL REAGENTS

|                                                                                           |              |          |
|-------------------------------------------------------------------------------------------|--------------|----------|
| Bromine Water, 30 g/L.....                                                                | 29 mL**..... | 2211-20  |
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                    | 500 mL.....  | 884-49   |
| Phenol Solution, 30 g/L.....                                                              | 29 mL.....   | 2112-20  |
| Phosphate Standard Solution, PourRite ampule,<br>500 mg/L as PO <sub>4</sub> , 2 mL.....  | 20/pkg.....  | 14242-20 |
| Phosphate Standard Solution, Voluette ampule,<br>500 mg/L as PO <sub>4</sub> , 10 mL..... | 16/pkg.....  | 14242-10 |

## OPTIONAL APPARATUS

|                                                      |                  |          |
|------------------------------------------------------|------------------|----------|
| Ampule Breaker Kit .....                             | each.....        | 21968-00 |
| Aspirator, vacuum .....                              | each.....        | 2131-00  |
| Cylinder, graduated, mixing, 10 mL, 3 required ..... | each.....        | 20886-38 |
| Filter Holder, 47 mm, 300 mL, graduated.....         | each.....        | 13529-00 |
| Filter, membrane, 47 mm, 0.45 microns .....          | 200/pkg.....     | 13530-00 |
| Flask, filtering, 500 mL.....                        | each.....        | 546-49   |
| Flask, volumetric, Class A, 50-mL .....              | each.....        | 14574-41 |
| pH Indicator Paper, 1 to 11 pH units.....            | 5 rolls/pkg..... | 391-33   |
| pH Meter, <i>sension</i> ™ <b>I</b> , portable.....  | each.....        | 51700-10 |
| Pipet, TenSette®, 0.1 to 1.0 mL.....                 | each.....        | 19700-01 |
| Pipet Tips, for 19700-01 TenSette® Pipet .....       | 50/pkg.....      | 21856-96 |
| Pipet Tips, for 19700-10 TenSette® Pipet .....       | 250/pkg.....     | 21997-25 |
| Pipet, volumetric, Class A, 5.00-mL.....             | each.....        | 14515-37 |
| Pipet, volumetric, Class A, 8.00-mL.....             | each.....        | 14515-08 |
| PourRite™ Ampule Breaker .....                       | each.....        | 24846-00 |

## 总磷 Molybdovanadate 法 高量程

方法号: 10127

(0.0 to 100.0 mg/L  $\text{PO}_4^{3-}$ ) (使用过硫酸消解处理)

Test 'N Tube™



1. 开启COD反应器。加热到150度。将塑料挡板放于反应器之前。

2. 输入检测总磷的高量程的预设程序编号。

按下: **PRGM**  
屏幕将显示:

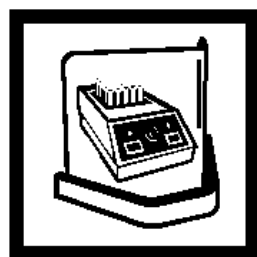
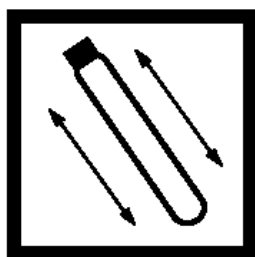
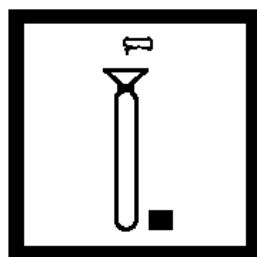
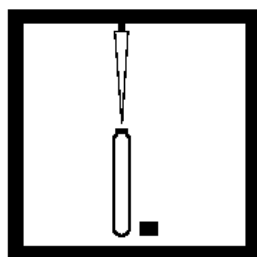
**PRGM?**

3. 按下: **87 ENTER**  
屏幕将显示:  
**mg/L, PO4D和ZERO**图

标。  
注: 如果检测其他形态, 按下:

**CONC**键

4. 使用TenSette移液管, 往一支总磷Test 'N试瓶注入5毫升的去离子水。(空白试样)



5. 使用TenSette移液管, 往另外一支总磷Test 'N试瓶注入5毫升的样品。

6. 使用漏斗将过硫酸钾试剂粉末加入到每个试瓶中。

7. 盖紧瓶盖, 摇晃试瓶使粉末溶解。

8. 将试瓶放在COD反应器上, 加热30分钟。

按下: **TIMER ENTER**  
将开始加热计时。



9. 小心拿下试瓶。将试瓶放在试管架上。让其冷却到室温。



10. 使用移液管将2.0毫升的1.54N氢氧化钠溶液注入每个试瓶内。



11. 使用聚乙烯滴液管将0.5毫升的Molybdovanadate试剂注入每个试瓶中。



12. 按下：  
**TIMER ENTER**  
将开始7分钟反应计时。



13. 旋转COD/TNT适配器，将其嵌入瓶管架上适当的位置，然后下按使之完全嵌入。



14. 用布擦干净试瓶外壁。



15. 当计时器鸣响时，将空白试样瓶放入样品适配器中。



16. 按下：**ZERO**  
指针将右移，屏幕会显示：  
**0.0 mg/L PO<sub>4</sub>**



17. 将预制试样瓶放入样品适配器中。



18. 盖紧瓶盖。



19. 按下：**READ**  
指针将右移，屏幕会显示总磷的含量，单位是 mg/L。

## 干扰

| 干扰物质                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 干扰水平和处理                                                                                                |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|
| 砷酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 如果样品被加热，将会产生正干扰。                                                                                       |
| 铁，亚铁                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   | 小于100mg/L，不产生干扰。L                                                                                      |
| 钼酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 大于100mg/L时，产生负干扰。                                                                                      |
| 硅                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 如果样品被加热，会产生正干扰。                                                                                        |
| 硫化物                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 产生负干扰。按下法除去硫化物干扰：<br>8. 量取50mL样品到锥形烧瓶中。<br>9. 一边摇晃一边逐滴加入溴水直到出现持久的黄色。<br>10. 一边摇晃一边逐滴加入苯酚溶液直黄色消失。继续步骤1。 |
| 高缓冲样品或极端样品pH                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 可能超过试剂的缓冲能力，需要进行样品预处理。                                                                                 |
| 氟化物、钍、铋、硫代硫酸盐、硫氰酸盐                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | 产生负干扰。                                                                                                 |
| 温度，冷（低于20 °C）                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | 产生负干扰。                                                                                                 |
| 温度，热（高于25 °C）                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | 产生正干扰。                                                                                                 |
| 下列物质含量不大于1000mg/L时，不产生干扰。<br>焦磷酸盐、四硼酸盐、 硒酸盐、 安息香酸盐、柠檬酸盐、草酸盐、乳酸盐、酒石酸盐、甲酸盐、水杨酸盐、铝离子、三价铁离子、镁离子、钙离子、 Ba <sup>2+</sup> 、 Sr <sup>2+</sup> 、 Li <sup>+</sup> 、 Na <sup>+</sup> 、 K <sup>+</sup> 、 NH <sub>4</sub> <sup>+</sup> 、 Cd <sup>2+</sup> 、 Mn <sup>2+</sup> 、 NO <sub>3</sub> <sup>-</sup> 、 NO <sub>2</sub> <sup>-</sup> 、 SO <sub>4</sub> <sup>2-</sup> 、 SO <sub>3</sub> <sup>2-</sup> 、 Pb <sup>2+</sup> 、 Hg <sup>+</sup> 、 Hg <sub>2</sub> <sup>2+</sup> 、 Sn <sup>2+</sup> 、 Cu <sup>2+</sup> 、 Ni <sup>2+</sup> 、 Ag <sup>+</sup> 、 U <sup>4+</sup> 、 Zr <sup>4+</sup> 、 AsO <sub>3</sub> <sup>-</sup> 、 Br <sup>-</sup> 、 CO <sub>3</sub> <sup>2-</sup> 、 ClO <sub>4</sub> <sup>-</sup> 、 CN <sup>-</sup> 、 IO <sub>3</sub> <sup>-</sup> 、 SiO <sub>4</sub> <sup>-</sup> 。 |                                                                                                        |

## Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning the glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated H<sub>2</sub>SO<sub>4</sub> (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N NaOH before analysis.

Correct test results for volume additions; see *Volume Additions* in *Section 1* of the *DR/890 Procedures Manual*.

## Accuracy Check

**Note:** Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.

### Standard Additions Method

- Fill each of three 10-mL graduated mixing cylinders with 10 mL of sample.
- Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO<sub>4</sub> (Cat. No. 14242-10).

- c. Use a TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of the water sample prepared in *step a*. Mix well.
- d. Analyze samples from *step c* as described in the procedure. Use 5.0 mL of the prepared sample for each test. The concentration should increase: 5 mg/L, 10 mg/L, and 15 mg/L PO<sub>4</sub>, respectively.
- e. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

#### **Standard Solution Method**

To check accuracy, prepare an 80 mg/L standard by pipetting 8.0 mL of solution from a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO<sub>4</sub> into an acid-cleaned, Class A, 50-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

#### **Standard Adjust**

To adjust the calibration curve using the reading obtained with the 80 mg/L PO<sub>4</sub> standard solution, press the **SETUP** key and scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1 of the Procedures Manual* for more information.

## **Method Performance**

### **Precision**

In a single laboratory, using a standard solution of 80.0 mg/L PO<sub>4</sub> and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO<sub>4</sub>

### **Estimated Detection Limit**

The estimated detection limit for program 87 is 7.0 mg/L PO<sub>4</sub>. For more information on the estimated detection limit, see *Section 1 of the DR/890 Procedures Manual*.

## **Safety**

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

## **Sample Disposal Information**

The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

## **Summary of Method**

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of

the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

## Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

| Line Number | Entry | Line Number | Entry |
|-------------|-------|-------------|-------|
| 1           | 87    | 18          | 0     |
| 2           | 4     | 19          | 0     |
| 3           | 73    | 20          | 80    |
| 4           | 0     | 21          | 79    |
| 5           | 0     | 22          | 52    |
| 6           | 0     | 23          | 0     |
| 7           | 0     | 24          | 0     |
| 8           | 0     | 25          | 80    |
| 9           | 0     | 26          | 0     |
| 10          | 0     | 27          | 0     |
| 11          | 0     | 28          | 0     |
| 12          | 66    | 29          | 0     |
| 13          | 175   | 30          | 80    |
| 14          | 48    | 31          | 50    |
| 15          | 32    | 32          | 79    |
| 16          | 0     | 33          | 53    |
| 17          | 0     | 34          | 0     |
| 35          | 62    | 46          | 0     |

| Line Number | Entry | Line Number | Entry |
|-------------|-------|-------------|-------|
| 36          | 166   | 47          | 15    |
| 37          | 246   | 48          | 7     |
| 38          | 148   | 49          | 8     |
| 39          | 63    | 50          | 1     |
| 40          | 63    | 51          | 164   |
| 41          | 78    | 52          | 0     |
| 42          | 252   | 53          | 0     |
| 43          | 4     | 54          | 40    |
| 44          | 76    | 55          | 0     |
| 45          | 128   | 56          | 255   |

## REQUIRED REAGENTS

高量程Test 'N Tube™ 磷试剂一套..... 50 vials ..... 27672-45  
包括: (50) 总磷 Test 'N Tube™ 试剂瓶\*, (2) 272-42, (1) 20847-66 (1) , 20760-26, (1) , 7430-42

| 试剂种类                     | 所需数量<br>每次测试 | 单位     | 货号       |
|--------------------------|--------------|--------|----------|
| Molybdovanadate 试剂 ..... | 0.5 mL       | 25 mL  | 20760-26 |
| 过硫酸钾粉末试剂 .....           | 1            | 50/pkg | 20847-66 |
| 氢氧化钠溶液, 1.54 N .....     | 2 mL         | 100 mL | 27430-42 |
| 总磷Test 'N Tube™ 试剂瓶..... | 1            | 50/pkg | *        |
| 去离子水.....                |              | 100 mL | 272-42   |

## 所需仪器

|                                                |     |        |          |
|------------------------------------------------|-----|--------|----------|
| COD 反应器, 115/230 Vac (U.S.A. and Canada) ..... | 1   | 个      | 45600-00 |
| COD 反应器, 115/230 Vac (Europe) .....            | 1   | 个      | 45600-02 |
| COD/TNT 适配器, DR/800 series.....                | 1   | 个      | 48464-00 |
| 点滴器, LDPE, 0.5 to 1.0 mL.....                  | 1   | 20/pkg | 21247-20 |
| TenSette®移液管 1 to 10 mL .....                  | 1   | 个      | 19700-10 |
| 移液管嘴, for 19700-10 TenSette® Pipet.....        | 不定  | 50/pkg | 21997-96 |
| 安全挡板 .....                                     | 1   | 个      | 50030-00 |
| 试管架.....                                       | 1-3 | each   | 18641-00 |

## OPTIONAL REAGENTS

| Description                                                                                | Unit   | Cat. No. |
|--------------------------------------------------------------------------------------------|--------|----------|
| Hydrochloric Acid Standard Solution, 6.0 N (1:1) .....                                     | 500 mL | 884-49   |
| Phosphate Standard Solution, PourRite™ ampule,<br>500 mg/L as PO <sub>4</sub> , 2-mL.....  | 20/pkg | 14242-20 |
| Phosphate Standard Solution, Voluette™ ampule,<br>500 mg/L as PO <sub>4</sub> , 10-mL..... | 16/pkg | 14242-10 |
| Sodium Hydroxide Standard Solution, 5.0 N.....                                             | 1 L    | 2450-53  |
| Sulfuric Acid, ACS, concentrated .....                                                     | 500 mL | 979-491  |

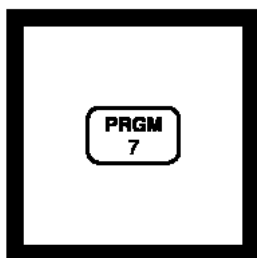
## OPTIONAL APPARATUS

|                                                       |             |          |
|-------------------------------------------------------|-------------|----------|
| Ampule Breaker Kit .....                              | each        | 21968-00 |
| Aspirator, vacuum .....                               | each        | 2131-00  |
| Cylinder, graduated, mixing, 10 mL (3 required) ..... | each        | 20886-38 |
| Filter Holder, 47 mm, 300 mL, graduated.....          | each        | 13529-00 |
| Filter, membrane, 47 mm, 0.45 microns .....           | 200/pkg     | 13530-01 |
| Flask, filtering, 500-mL .....                        | each        | 546-49   |
| Flask, volumetric, Class A, 50-mL .....               | each        | 14574-41 |
| pH Indicator Paper, 1 to 11 pH units.....             | 5 rolls/pkg | 391-33   |
| pH Meter, <i>sensio</i> ™ <b>I</b> , portable.....    | each        | 51700-10 |
| Pipet Filler, Safety Bulb .....                       | each        | 14651-00 |
| Pipet, TenSette®, 0.- to 1.0-mL.....                  | each        | 19700-01 |
| Pipet Tips, for 19700-01.....                         | 50 pkg      | 21856-96 |

|                                          |               |          |
|------------------------------------------|---------------|----------|
| Pipet, volumetric, Class A, 8.00-mL..... | each.....     | 14515-08 |
| Stopper, No. 7 one hole .....            | 6/pkg.....    | 2119-07  |
| Tubing, rubber .....                     | 12 feet ..... | 560-19   |

## 硅 Heteropoly Blue 方法 低量程 (0 to 1.60 mg/L)

方法号: 8186



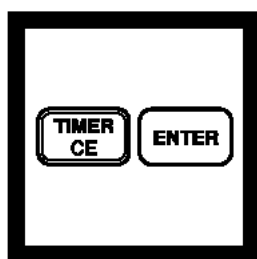
1. 输入检测低量程的硅( $\text{SiO}_2$ )的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**

2. 按下: **90 ENTER**  
屏幕将显示:  
**0.00 mg/L、SiO<sub>2</sub>**  
和ZERO图标。

3. 分别往两个比色瓶中装入 10 mL 样品。

4. 分别向两瓶中注入 15 滴钼酸盐 3 试剂。晃动使之混合。

*注: 如要得到最精确的结果, 应垂直握住滴液瓶。*



5. 按 Timer  
将开始 4 分钟的反应。

*注: 所给的反映时间是对应于样品温度 20 ° C (68 ° F)。如果样品温度为 10 ° C 时, 要反应 8 分钟。如果样品温度为 30 ° C, 应反应 2 分钟。*

6. 定时器鸣响后, 分别将各一包柠檬酸试剂粉末加入两种样品中。晃动使之混合。

7. 屏幕将显示:  
**1:00 TIMER 2**  
按下: 回车  
将开始一分钟的反应。在此期间, 磷酸盐的干扰将会被消除。

*注: 所给的反映时间是对应于样品温度 20 ° C (68 ° F)。如果样品温度为 10 ° C 时, 要反应 2 分钟。如果样品温度为 30 ° C, 应反应 30 秒钟。*

8. 定时器鸣响后, 将一包氨基酸试剂粉末加入到其中一支比色瓶中。反转使之混合。

*注: 没有加入氨基酸试剂的样品就是空白试样。*



9. 屏幕将显示:

**2:00 TIMER 3**

按下: 回车

将开始 2 分钟的反  
应。

10. 在定时器鸣响后,  
将空白试样 (没有加  
氨基酸) 放入样品适  
配器中, 盖紧遮光盖。

11. 按 **ZERO**, 指针将  
右移, 屏幕显示:

**0 mg/L SiO<sub>2</sub>**

12. 将预制试样放入  
样品适配器中, 盖紧  
遮光盖。

注: 如果存在硅, 溶  
液将显现蓝色。



13. 按下 **READ**

指针将右移, 屏幕将  
显示 SiO<sub>2</sub> 的含量, 单  
位为 mg/L。

注: 建议对每种新的  
试剂进行标准校正。

## 干扰

| 干扰物质 | 干扰水平和处理                                                                                                                                           |
|------|---------------------------------------------------------------------------------------------------------------------------------------------------|
| 颜色   | 用原始样品调零除去。                                                                                                                                        |
| 磷酸盐  | 低于50 mg/L的 PO <sub>4</sub> ，不干扰。60 mg/L的PO <sub>4</sub> ，-2%的干扰，75 mg/L的 PO <sub>4</sub> ，-11%的干扰。                                                |
| 铁    | 大量时干扰                                                                                                                                             |
| 钝化硅  | 通常样品含有与钼酸盐缓慢反应的硅，对这些“不反应形式的钼酸盐”的本质还不是很清楚。用重碳酸钠预处理，然后用硫酸处理，可使这些形式的硅与钼酸盐反应。预处理方法在重碳酸钠的硅消化下的“水和废水的标准检查方法”中给出。需要长一些的反应时间，钼酸盐和酸试剂（加入柠檬酸之前）有助于重碳酸盐的预处理。 |
| 硫化物  | 所有水平上干扰                                                                                                                                           |
| 浑浊   | 用原始样品调零除去。                                                                                                                                        |

## Sampling and Storage

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

## Accuracy Check

### Standard Additions Method

- Open a Low Range Silica Standard Solution Pillow, 50 mg/L SiO<sub>2</sub>.
- Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 10-mL samples. Mix thoroughly.
- Analyze each sample as described above. The silica concentration should increase 0.5 mg/L for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L Standard Solution (see *Optional Reagents*), press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.00** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a instrument, a single operator obtained a standard deviation of  $\pm 0.025$  mg/L silica.

### Estimated Detection Limit (EDL)

The estimated detection limit for program 90 is 0.020 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*. If testing for very low levels of silica, use the ultra-low range silica method on the Hach DR/2010 or DR/4000 Spectrophotometers.

## Reagent Preparation

To prepare Amino Acid F Reagent Solution, dissolve 11.4 grams of Amino Acid F Reagent Powder in 100 mL of 1.0 N Sodium Hydroxide Solution. The solution is stable for at least one month if stored in a plastic bottle.

## Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Acid reduces the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

### 所需试剂

低量程硅试剂一套, 10 mL sample (100 tests) .....24593-00  
包括: (1) 22540-69, (1) 21062-69 (2) 1995-26

| 试剂种类                 | 所需数量<br>每次测试 | 单位         | 货号       |
|----------------------|--------------|------------|----------|
| 氨基酸 F 粉末试剂.....      | 1 包.....     | 100/pkg    | 22540-69 |
| 柠檬酸粉末粉末 .....        | 2 包 .....    | 100/pkg    | 21062-69 |
| Molybdate 3 试剂 ..... | 28 滴 .....   | 50 mL SCDB | 1995-26  |

### REQUIRED APPARATUS

样品比色瓶, 10-20-25 mL, w/ cap .....2 .....6/pkg .....24019-06

### OPTIONAL REAGENTS

Silica Standard Solution, 1.00 mg/L SiO<sub>2</sub> ..... 500 mL .....1106-49  
Silica Standard Solution Pillows, 20 mg/L as SiO<sub>2</sub>, 10 mL.....16/pkg .....14245-10  
Sodium Bicarbonate, ACS .....454 g .....776-01  
Sodium Hydroxide Standard Solution, 1.000 N..... 900 mL .....1045-53  
Sulfuric Acid Standard Solution, 1.0 N ..... 1000 mL .....1270-53

### OPTIONAL APPARATUS

Bottle, 118 mL, polyethylene, oblong.....6/pkg .....23184-06  
Dropper, 0.5- & 1.0-mL marks.....6/pkg .....23185-06  
Pipet, serologic, 2 mL, poly .....each .....2106-36  
Pipet, TenSette, 0.1 to 1.0 mL.....each .....19700-01

|                                                                           |              |          |
|---------------------------------------------------------------------------|--------------|----------|
| Pipet Tips, for 19700-01 Pipet .....                                      | 50/pkg ..... | 21856-96 |
| <i>Standard Methods for the Examination of Water and Wastewater</i> ..... | each .....   | 22708-00 |
| Thermometer, - 10 to 110 °C.....                                          | each .....   | 1877-01  |

***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 硅 钼酸硅法 高量程 (0 to 75.0 mg/L)

方法号: 8185



1. 输入检测高量程的硅( $\text{SiO}_2$ )的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**



2. 按下: **89 ENTER**  
屏幕将显示:  
0.00 mg/L、**SiO<sub>2</sub>**  
和ZERO图标。



3. 分别往两个比色瓶中装入 10 mL 样品。  
将其中一只放在一旁作为空白试样。



4. 往另外一只比色瓶中加入钼酸盐试剂粉, (预制试样)。盖上瓶盖倒转使之混合。

注: 为得到更加精确的结果, 应用去离子水进行试剂空白校正。



5. 将一包用于测试高量程硅的酸性试剂粉加入样品中, 盖上瓶盖, 倒转使之混合。

注: 如存在硅或磷酸盐, 溶液将显现黄色。



6. 按: **TIMER**  
将开始 10 分钟的反映。



7. 定时器鸣响后, 将一包柠檬酸试剂粉末加入到预制试剂中。盖上瓶盖, 反转使之混合。

注: 由于磷酸盐的存在而显现的黄色将消失。



8. 屏幕将显示:  
**2:00 Timer 2**  
将开始2分钟的反应。  
注: 应在定时器鸣响后3分钟内, 执行第9—12步。



9. 在定时器鸣响后，将空白试样（没有加氨基酸）放入样品适配器中，盖紧遮光盖。

10. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L SiO<sub>2</sub>**。

*注：如果在进行试剂空白校正，屏幕将闪烁显示“limit”。*

11. 将预制试样放入样品适配器中，盖紧遮光盖。

12. 按下 READ 指针将右移，屏幕将显示 SiO<sub>2</sub> 的含量，单位为 mg/L。

*注：建议对每种新的试剂进行标准校正。*

## 干扰

| 干扰物质 | 干扰水平和处理                                                                                            |
|------|----------------------------------------------------------------------------------------------------|
| 颜色   | 用原始样品调零除去。                                                                                         |
| 铁    | 大量时干扰                                                                                              |
| 磷酸盐  | 低于50 mg/L的 PO <sub>4</sub> ，不干扰。60 mg/L的PO <sub>4</sub> ，-2%的干扰，75 mg/L的 PO <sub>4</sub> ，-11%的干扰。 |
| 硫化物  | 大量时干扰                                                                                              |
| 浑浊   | 用原始样品调零后可以除去干扰。                                                                                    |

在某些情况下，样品中含有的硅会和钼酸盐反应得十分缓慢，现在还不清楚钼酸盐这种不活跃性的原因。此时，应先用重碳酸钾进行预处理，然后加硫磺酸使这种形态的物质和钼酸盐反应。在加入重碳酸钾的环境下，样品、钼酸盐和酸性试剂的长时间的反应将会有所改善。

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

## Accuracy Check

### Standard Additions Method

- a) Open a High Range Silica Standard Solution, 1000 mg/L SiO<sub>2</sub>.
- b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.
- c) Analyze each sample as described above. The silica concentration should increase 10.0 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To check the accuracy of the method, use the Silica Standard Solutions, 25 and 50 mg/L as SiO<sub>2</sub>, listed under Optional Reagents. Analyze according to the above procedure using deionized water as the blank.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 50.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 50.0 mg/L SiO<sub>2</sub> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L silica.

### Estimated Detection Limit

The estimated detection limit for program 89 is 1.00 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

## 所需试剂

高量程硅试剂一套, 10 mL sample (100 tests) .....24296-00  
包括: (1) 21074-69, (1) 21062-69, (1) 21073-69

| 试剂种类                 | 所需数量<br>每次测试 | 单位         | 货号       |
|----------------------|--------------|------------|----------|
| 酸粉末试剂.....           | 1 包.....     | 100/pkg    | 22540-69 |
| 柠檬酸粉末粉末 .....        | 2 包 .....    | 100/pkg    | 21062-69 |
| Molybdate 粉末试剂 ..... | 28 滴 .....   | 50 mL SCDB | 21073-69 |

## 所需仪器

样品比色瓶, 10-20-25 mL, w/ cap .....2 .....6/pkg .....24019-06

## OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L.....500 mL.....1403-49  
Silica Standard Solution, 25 mg/L.....236 mL.....21225-31  
Silica Standard Solution, 50 mg/L.....200 mL.....1117-29  
Silica Standard Solution, 1000 mg/L.....500 mL.....194-49  
Sodium Bicarbonate, ACS.....454 g.....776-01  
Sulfuric Acid Standard Solution, 1.000 N .....100 mL MDB.....1270-32  
Water, deionized.....4 L.....272-56

## OPTIONAL APPARATUS

Pipet, TenSette, 0.1 to 1.0 mL..... each.....19700-01  
Pipet Tips, for 19700-01 Pipet ..... 50/pkg.....21856-96  
*Standard Methods for the Examination of Water and Wastewater* ..... each.....22708-00  
Thermometer, -10 to 110 °C..... each.....1877-01

## For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 硅 钼酸硅法 超高量程 (0 to 75.0 mg/L)

方法号: 10098



1. 输入检测超高量程的硅( $\text{SiO}_2$ )的程序编号。

按下: **PRGM**

屏幕将显示:

**PRGM?**

注: 为得到更加精确的结果, 应用去离子水进行试剂空白校正。



2. 按下: **88 ENTER**

屏幕将显示:

0.00 mg/L、**SiO<sub>2</sub>**

和ZERO图标。

注: 如果测试其他形态的硅 ( $\text{Si}$ ), 按下:

**CONC** 键



3. 分别往两个比色瓶中装入 10 mL 样品。

注: 样品的温度应为  $15 \text{ to } 25^\circ \text{C}$  ( $59 \text{ to } 77^\circ \text{F}$ )。



4. 分别将 25ml 的去离子水注入两个样品比色瓶中。取其中一支作为空白试样。



5. 将一包用于测试超高量程硅的钼酸盐试剂粉剂加入另外一只样品比色瓶中 (预制试样), 盖上瓶盖, 倒转使之混合。

注: 如存在硅或磷酸盐, 溶液将显现黄色。



6. 加一包测试硅用的酸性试剂粉剂到预制试样中, 盖上瓶盖, 倒转使之混合。

注: 如存在硅或磷酸盐, 溶液将显现黄色。



7. 按下:

**TIMER ENTER**

将开始10分钟的反应。



8. 定时器鸣响后, 将一包柠檬酸试剂粉末加入到预制试剂中。盖上瓶盖, 反转使之混合。

注: 由于磷酸盐的存在而显现的黄色将消失。



9. 屏幕将显示：  
**2:00 Timer 2**  
将开始2分钟的反应。

注：应在定时器鸣响后 3 分钟内，执行第 9—12 步。



10. 在定时器鸣响后，将空白试样（没有加氨基酸）放入样品适配器中，盖紧遮光盖。



11. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L SiO<sub>2</sub>**。

注：如果在进行试剂空白校正，屏幕将闪烁显示“limit”。



12. 将预制试样放入样品适配器中，盖紧遮光盖。



13. 按下 READ  
指针将右移，屏幕将显示 SiO<sub>2</sub> 的含量，单位为 mg/L。

注：建议对每种新的试剂进行标准校正。

### 干扰

| 干扰物质 | 干扰水平和处理                                                                                            |
|------|----------------------------------------------------------------------------------------------------|
| 颜色   | 用原始样品调零除去。                                                                                         |
| 铁    | 大量时干扰                                                                                              |
| 磷酸盐  | 低于50 mg/L的 PO <sub>4</sub> ，不干扰。60 mg/L的PO <sub>4</sub> ，-2%的干扰，75 mg/L的 PO <sub>4</sub> ，-11%的干扰。 |
| 硫化物  | 大量时干扰                                                                                              |
| 浑浊   | 用原始样品调零后可以除去干扰。                                                                                    |

在某些情况下，样品中含有的硅会和钼酸盐反应得十分缓慢，现在还不清楚钼酸盐这种不活跃性的原因。此时，应先用重碳酸钾进行预处理，然后加硫磺酸使这种形态的物质和钼酸盐反应。在加入重碳酸钾的环境下，样品、钼酸盐和酸性试剂的长时间的反应将会有所改善。

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

## Accuracy Check

### Standard Additions Method

- a) Open a High Range Silica Standard Solution, 1000 mg/L SiO<sub>2</sub>.
- b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.
- c) Analyze each sample as described above. The silica concentration should increase 4 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

To prepare a 160-mg/L silica standard, pipet 40.0 mL of a 1000-mg/L Silica Standard Solution into a 250-mL volumetric flask. Dilute to the line with deionized water. Analyze according to the above procedure using deionized water as the blank.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 160-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **160.** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 100.0 mg/L SiO<sub>2</sub> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2.0 mg/L silica.

### Estimated Detection Limit

The estimated detection limit for program 88 is 3.0 mg/L SiO<sub>2</sub>. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

## 所需试剂

|                                            | 货号       |
|--------------------------------------------|----------|
| 高量程硅试剂一套, 25-mL sample (100 tests) .....   | 22443-00 |
| 包括: (1) 1042-99, (1) 14548-99, (2) 1041-66 |          |

| 试剂种类             | 所需数量<br>每次测试 | 单位      | 货号       |
|------------------|--------------|---------|----------|
| 测试硅用的酸性粉末试剂..... | 1            | 100/pkg | 1042-99  |
| 柠檬酸粉末试剂 .....    | 1            | 100/pkg | 14548-99 |
| 钼酸盐粉末试剂 .....    | 1            | 100/pkg | 1041-99  |
| 去离子水 .....       | 30 mL        | 4 L     | 272-56   |

## 所需仪器

|                          |   |       |          |
|--------------------------|---|-------|----------|
| 样品比色瓶 10-20-15 mL, ..... | 2 | 6/pkg | 24019-06 |
|--------------------------|---|-------|----------|

## OPTIONAL REAGENTS

|                                               |            |          |
|-----------------------------------------------|------------|----------|
| Silica Standard Solution, 10 mg/L .....       | 500 mL     | 1403-49  |
| Silica Standard Solution, 25 mg/L .....       | 236 mL     | 21225-31 |
| Silica Standard Solution, 50 mg/L .....       | 200 mL     | 1117-29  |
| Silica Standard Solution, 1000 mg/L .....     | 500 mL     | 194-49   |
| Sodium Bicarbonate, ACS .....                 | 454 g      | 776-01   |
| Sulfuric Acid Standard Solution, 1.000 N..... | 100 mL MDB | 1270-32  |

## OPTIONAL APPARATUS

|                                                                           |        |          |
|---------------------------------------------------------------------------|--------|----------|
| Flask, volumetric, 250 mL, Class A.....                                   | each   | 14574-46 |
| Pipet, TenSette, 0.1 to 1.0 mL.....                                       | each   | 19700-01 |
| Pipet Tips, for 19700-01 Pipet .....                                      | 50/pkg | 21856-96 |
| Pipet, volumetric, Class A, 100 mL .....                                  | each   | 14515-42 |
| Pipet Filler, safety bulb.....                                            | each   | 14651-00 |
| <i>Standard Methods for the Examination of Water and Wastewater</i> ..... | each   | 22708-00 |
| Thermometer, -10 to 110 °C.....                                           | each   | 1877-01  |

## ***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 硫酸盐 SulfaVer 4 法 (0 to 70 mg/L)

方法号: 8051



1、在此项测试中，内设程式代号“91”已足够分析应用，但如果使用者需求，具高度准确性之结果，则请参考原文操作之自订校正曲线说明，自行建立校正曲线。

2、按“PRGM”键此时萤幕将显示 PRGM?

3、输入内设程式代号“91”然后按下“ENTER”键，此时萤幕会出现“mg/l SO<sub>4</sub>”及“ZERO icon”

4、取一支比色瓶加水样至 10ml 标线处，当空溶液。

注：太混浊及有颜色之水样，必须先过滤处理。



5、加入一 Sulfa Ver4 试剂至比色瓶中，盖好瓶盖，摇动使充分混合，当待测溶液。

6、同时按“TIMER”及“ENTER”键，将进行 5 分钟的反应计时。

7、当计时完毕，听到哔哔声，取另一比色瓶，加入 10ml 水样当空白溶液。

8、放空白溶液至比色槽中，并将比色计盖上盖子

注：假如水样中含硫酸根，此时会出现白色混浊。

注：未完全溶解的试剂并不会影响结果。



- 9、按“ZERO”键归零，萤幕会显示 0 mg/l SO<sub>4</sub>
- 10、在计时完毕的 5 分钟内，放待测溶液至比色槽中，将比色计盖上盖子。
- 11、按“READ”键，所欲测浓度将会显示出来，即 mg/l Sulfate。

## 干扰

下列物质超过以下所列含量时，会产生干扰。

|     |                                  |
|-----|----------------------------------|
| 钙   | 20000mg/L，以 CaCO <sub>3</sub> 形式 |
| 镁   | 10000mg/L                        |
| 氯化物 | 40000mg/L                        |
| 硅   | 500mg/L                          |

## User- Entered Calibration

There are various programs to determine sulfate, each with a different level of accuracy. Best results are obtained by performing a user-entered calibration with each new lot of reagent. Programs 91 and 92 can be run when a high degree of accuracy is not needed. Use of the Standard Adjust feature will improve performance when using programs 91 and 92. It should NOT be used with a user calibration, as it will hinder performance.

Using Class A glassware, prepare standards of 10, 20, 30, 40, 50, 60, and 70 mg/L sulfate by pipetting 1, 2, 3, 4, 5, 6, and 7 mL of a 1000-mg/L sulfate standard into 100-mL volumetric flasks. Dilute to the mark with deionized water and mix well.

Zero the instrument with water. The user-entered settings for sulfate are: Program number: #101 to 105

Wavelength: 520 nm

Resolution: 0 mg/L

See *Creating User-Entered Program* in the instrument manual for specific instructions on entering a user-entered program.

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples may be stored up to 28 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

## Accuracy Check

### Standard Additions Method- Powder Pillows

- a) Snap the neck off a Sulfate Standard PourRite Ampule, 1000 mg/L  $\text{SO}_4$ .
- b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three 10-mL samples. Mix thoroughly.
- c) Analyze each sample as described above. The sulfate concentrations should increase 10 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Additions Method- AccuVac Ampuls

- a) Snap the neck off a Sulfate Standard PourRite Ampule, 2500 mg/L  $\text{SO}_4$ .
- b) Fill three 25- mL graduated cylinders with 25 mL of sample. Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three cylinders. Mix thoroughly. For AccuVac Ampuls, transfer to a 50-mL beaker.
- c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.
- d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Check the accuracy of the test by using the Sulfate Standard Solution, 50 mg/L, listed under Optional Reagents. Or, prepare this solution by pipetting 1.0 mL of a PourRite Ampule Standard for Sulfate (2500 mg/L) into a 50-mL volumetric flask. Dilute to volume with deionized water. The final concentration is 50 mg/L sulfate. Substitute this standard for the sample and proceed with the test as described in the procedure.

### Standard Adjust

Standard adjust is recommended when using stored programs 91 or 92. It **should not** be used with a user-entered calibration. To adjust the calibration curve using the reading obtained with the 50-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of  $\pm 0.5$  mg/L sulfate.

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of  $\pm 3$  mg/L sulfate.

### Estimated Detection Limit (EDL)

The EDL for program 91 is 4.9 mg/L  $\text{SO}_4$  and the EDL for program 92 is 3 mg/L  $\text{SO}_4$ .

For more information on derivation and use of Hach's estimated detection limit, see *Section I*.

## Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 Sulfate Reagent to form insoluble barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. The SulfaVer 4 also contains a stabilizing agent to hold the precipitate in suspension.

### 所需试剂和仪器 (使用粉末试剂)

| 试剂种类                             | 所需数量<br>每次测试 | 单位            | 货号       |
|----------------------------------|--------------|---------------|----------|
| SulfaVer 4硫酸盐粉末试剂 .....          | 1 包.....     | 100/pkg ..... | 21067-69 |
| 样品比色瓶, 10-20-25 mL, w/ cap ..... | 2 .....      | 6/pkg .....   | 24019-06 |

### 所需试剂和仪器 (使用安瓿瓶)

|                                |          |              |          |
|--------------------------------|----------|--------------|----------|
| SulfaVer 4 硫酸盐 AccuVac 安瓿..... | 1 瓶..... | 25/pkg ..... | 25090-25 |
| 烧杯, 50-mL.....                 | 1 .....  | 个 .....      | 500-41   |

## OPTIONAL REAGENTS

|                                                                   |              |          |
|-------------------------------------------------------------------|--------------|----------|
| Sulfate Standard Solution, 50 mg/L .....                          | 500 mL ..... | 2578-49  |
| Sulfate Standard Solution, 1000 mg/L .....                        | 500 mL ..... | 21757-49 |
| Sulfate Standard Solution, PourRite Ampule, 2500 mg/L, 2 mL ..... | 20/pkg ..... | 14252-20 |
| Sulfate Standard Solution, PourRite Ampule, 1000 mg/L, 2 mL ..... | 20/pkg ..... | 21757-20 |
| Water, deionized .....                                            | 4 L .....    | 272-56   |

## OPTIONAL APPARATUS

|                                           |               |          |
|-------------------------------------------|---------------|----------|
| AccuVac Snapper Kit .....                 | each .....    | 24052-00 |
| Cylinder, graduated mixing, 25 mL .....   | each .....    | 20886-40 |
| Filter Paper, folded, 12.5 cm .....       | 100/pkg ..... | 1894-57  |
| Flask, volumetric, 50 mL, Class A.....    | each .....    | 14574-41 |
| Funnel, poly, 65 mm.....                  | each .....    | 1083-67  |
| Pipet, TenSette, 0.1 to 1.0 mL.....       | each .....    | 19700-01 |
| Pipet Tips, for 19700-01 Pipet .....      | 50/pkg .....  | 21856-96 |
| Pipet, volumetric, 1.00 mL, Class A ..... | each .....    | 14515-35 |
| Pipet, volumetric, 2.00 mL, Class A ..... | each .....    | 14515-36 |
| Pipet, volumetric, 3.00 mL, Class A ..... | each .....    | 14515-03 |
| Pipet, volumetric, 4.00 mL, Class A ..... | each .....    | 14515-04 |
| Pipet, volumetric, 5.00 mL, Class A ..... | each .....    | 14515-37 |
| Pipet, volumetric, 6.00 mL, Class A ..... | each .....    | 14515-06 |
| Pipet, volumetric, 7.00 mL, Class A ..... | each .....    | 14515-07 |
| Pipet Filler, safety bulb.....            | each .....    | 14651-00 |
| PourRite Ampule Breaker .....             | each .....    | 24846-00 |

### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.



1、按“PRGM”键，此时萤幕将显示 PRGM?



2、输入内设程式代号“93”然后按下“ENTER”键，此时萤幕会出现“mg/l S”及“ZERO icon”



3、取一支比色瓶加水样至25ml 标线处当待测溶液。

注：水样无法保存，必须立即分析且使用移液管取水样，避免水样过度摇动。



4、取另一比色瓶，加入 25ml 去离子水，当空白溶液。

注：野外测试时，必须先以 25ml 量筒取水样。



5、各加入 1ml Sulfite1 试剂至两支比色瓶中，摇动使充分混合。



6、各加入 1ml Sulfite 2 试剂至两支比色瓶中，摇动使充分混合。

注：此时水样会呈现粉红色，假如水样中含硫化物，将会转变为蓝色。



7、同时按“TIMER”及“ENTER”键，将进行 5 分钟的反应计时



8、放空白溶液至比色槽中，并将比色计盖上盖子



- 9、按“ZERO”键归零，萤幕会显示 0.00mg/1 S
- 10、当计时完毕，听到哔哔声，放待测溶液至比色槽中，并将比色计盖上盖子。
- 11、按“READ”键，所欲测浓度将会显示出来，即 mg/1 S

*注：假如水样必须稀释，将会造成所含硫化物量的损失*

## 干扰

| 干扰物质                    | 干扰水平和处理                                                                                                                                                                            |
|-------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 强还原性物质（亚硫酸盐，硫代硫酸盐和次硫酸盐） | 由于还原蓝色或阻碍蓝色的生成                                                                                                                                                                     |
| 高水平硫化物                  | 高浓度硫化物会抑制颜色的完全生成，需要稀释样品。稀释会有硫化物的损失。                                                                                                                                                |
| 浑浊                      | 对于浑浊的样品，准备不含硫化物的空白，用它代替去离子水： <ol style="list-style-type: none"> <li>1、量取25mL样品，放入50mL锥形瓶中。</li> <li>2、边摇匀边滴加溴水，直到刚出现黄色而且并不褪去。</li> <li>3、滴加苯酚溶液直到黄色消失，在步骤4中使用该溶液代替去离子水。</li> </ol> |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

## Method Performance

### Precision

In a single laboratory, using standard solutions of 0.73 mg/L sulfide and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L sulfide.

### Estimated Detection Limit (EDL)

The EDL for program 93 is 0.01 mg/L S<sub>2</sub><sup>-</sup>. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

### Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N, N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after dilution.

### Pollution Prevention and Waste Management

Sulfide 2 Reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. See *Section 3* for more information on proper disposal of these materials.

#### 所需试剂

硫化物试剂一套 (100 tests) ..... 22445-00  
包括: (2) 1816-42, (2) 1817-42

| 试剂种类          | 所需数量<br>每次测试 | 单位              | 货号      |
|---------------|--------------|-----------------|---------|
| 硫化物试剂 1.....  | 2 mL.....    | 100 mL MDB..... | 1816-32 |
| 硫化物试剂 2 ..... | 2 mL.....    | 100 mL MDB..... | 1817-32 |
| 去离子水.....     | 25 mL.....   | 4 L .....       | 272-56  |

#### 所需仪器

|                                 |        |            |          |
|---------------------------------|--------|------------|----------|
| 刻度量筒, 25 mL .....               | 1..... | 个.....     | 508-40   |
| 移液管, Class A, 25.00 mL.....     | 1..... | 个.....     | 14515-40 |
| Pipet Filler, safety bulb ..... | 1..... | 个.....     | 14651-00 |
| 样品比色瓶, 10-20-25 mL, w/ cap..... | 2..... | 6/pkg..... | 24019-06 |

### OPTIONAL REAGENTS

#### Description Units Cat. No.

|                               |            |         |
|-------------------------------|------------|---------|
| Bromine Water, 30 g/L.....    | 29 mL..... | 2211-20 |
| Phenol Solution, 30 g/L ..... | 29 mL..... | 2112-20 |
| Sodium Sulfide, hydrate ..... | 114 g..... | 785-14  |

### OPTIONAL APPARATUS

|                                                                           |           |          |
|---------------------------------------------------------------------------|-----------|----------|
| Bottle, Wash, 250 mL .....                                                | each..... | 620-31   |
| Dropper, for 1 oz. bottle.....                                            | each..... | 2258-00  |
| Flask, erlenmeyer, 50 mL .....                                            | each..... | 505-41   |
| <i>Standard Methods for the Examination of Water and Wastewater</i> ..... | each..... | 22708-00 |

### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 阴离子表面活性剂 (0 to 0.300 mg/L)

方法号: 8028

(也称去垢剂) 紫晶体法



1. 输入检测阴离子表面活性剂的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

2. 按下: **26 ENTER**  
屏幕将显示:

0.00 mg/L、LAS

和ZERO图标。

3. 将200ml的样品注入一个干净的容积500mL刻度的量筒中。然后将样品倒注入一个干净的500mL分液漏斗中。

4. 加入 10 mL 硫酸盐缓冲溶液, 盖好漏斗, 摇晃 5 秒。



5. 加入一包洗涤剂粉末试剂到漏斗中, 盖好漏斗, 并摇晃使粉末溶解

6. 将30 mL苯加到漏斗中, 盖好漏斗。轻轻摇晃一分钟。

*注: 溢出的试剂会影响结果的精确性, 并对皮肤等有害。*

*注: 应在通风良好的地方使用苯。*

7. 将分液漏斗放到支架上。

8. 按下:

**TIMER ENTER**

开始30分钟反应的计时。

*注: 振摇过度会形成乳状液, 将需要长时间分层。对于这样的样品, 移走大部分的水层, 然后在漏斗中加入惰性物, 使用涂上聚四氟乙烯的磁搅拌棒轻轻搅动。*



9. 当计时器鸣叫时，取走塞子并分出下层水层，弃去该水层。

*注：苯溶液是一种受管制的废料，不能直接倒入排水渠。*

10. 将上层的苯溶液注入一个干净的25mL比色瓶中（待测样品）。

*注：在颜色检测之前，苯层溶液不能被过滤。如过滤将会使蓝色消失。*

11. 往另一个比色瓶中装入25mL纯苯。（空白试样）。

12. 将空白试样瓶放入样品适配器中。盖紧样品遮光盖。



13. 按 ZERO，指针将右移，屏幕显示：  
**0 mg/L LAS。**

14. 将预制试样瓶放入样品适配器中。盖紧样品遮光盖。

15. 按下 READ 指针将右移，屏幕将显示阴离子表面活性剂的含量，单位为 mg/L。

*注：建议对每种新的试剂进行标准校正。*

*注：可用丙酮清洗装苯的玻璃器皿。*

## 干扰

高氯酸盐和高碘酸盐离子会产生干扰。高含量的氯化物，如盐水和海水所含氯化物，会导致结果偏低。

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible, but they may be stored at least 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

## Accuracy Check

### Standard Additions Method

- Snap the neck off a Detergent Voluette Ampule Standard Solution, 60 mg/L as LAS.
- Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 300-mL samples. Mix thoroughly.
- Analyze each as described above. The anionic surfactants reading should increase 0.02 mg/L for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* (Section 1) for more information.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 0.150 mg/L LAS, two lots of reagent, and the instrument, a single operator obtained a standard deviation of  $\pm 0.010$  mg/L LAS as anionic surfactant.

### Estimated Detection Limit

The estimated detection limit for program 26 is 0.020 mg/L LAS. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene.

## Pollution Prevention and Waste Management

Benzene (D018) solutions are regulated as hazardous waste by Federal RCRA. Do not pour these materials down the drain. Collect water saturated with benzene solutions for disposal with laboratory solvent wastes. See *Section 3* for more information on proper disposal of these materials.

## 所需试剂

| 试剂种类         | 所需数量<br>每次测试 | 单位          | 货号       |
|--------------|--------------|-------------|----------|
| 苯, ACS.....  | 55 mL.....   | 500 mL..... | 14440-49 |
| 硫酸盐缓冲溶液..... | 10 mL.....   | 500 mL..... | 452-49   |
| 洗涤剂粉末试剂..... | 1 包 .....    | 25/pkg..... | 1008-68  |

## 所需仪器

|                 |        |        |        |
|-----------------|--------|--------|--------|
| 剪刀.....         | 1..... | 个..... | 968-00 |
| 量筒, 25 mL ..... | 1..... | 个..... | 508-40 |

|                                            |        |            |          |
|--------------------------------------------|--------|------------|----------|
| 量筒, 50 mL .....                            | 1..... | 个.....     | 508-41   |
| 量筒 500 mL .....                            | 1..... | 个.....     | 508-49   |
| 分离漏斗, 500 mL .....                         | 1..... | 个.....     | 520-49   |
| Ring, support, 4 inch.....                 | 1..... | 个.....     | 580-01   |
| 样品比色瓶, 10-20-25 mL, w/ cap.....            | 2..... | 6/pkg..... | 24019-06 |
| Stand, support, 127 x 203 mm (5 x 8")..... | 1..... | 个.....     | 563-00   |

#### **OPTIONAL REAGENTS**

|                                                                              |             |          |
|------------------------------------------------------------------------------|-------------|----------|
| Acetone, ACS .....                                                           | 500 mL..... | 14429-49 |
| Detergent Standard Solution, Voluette ampule,<br>60 mg/L as LAS, 10 mL ..... | 16/pkg..... | 14271-10 |

#### **OPTIONAL APPARATUS**

|                                      |             |          |
|--------------------------------------|-------------|----------|
| Ampule Breaker Kit.....              | each.....   | 21968-00 |
| Pipet, TenSette, 0.1 to 1.0 mL.....  | each.....   | 19700-01 |
| Pipet Tips, for 19700-01 Pipet ..... | 50/pkg..... | 21856-96 |
| Thermometer, -10 to 110 °C.....      | each.....   | 1877-01  |

#### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 悬浮固体 (0 — 750 mg/L)

方法号: 8006

### 光度测定法



1. 输入检测悬浮固体的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**
2. 按下: **94 ENTER**  
屏幕将显示:  
0.00 mg/L、SuSld  
和ZERO图标。
3. 量取500 mL样品到  
搅拌器中高速搅拌2  
分钟。
4. 把样品倒入一个容  
积为 600ml 烧杯中。



5. 将 25ml 的自来水  
或去离子水注入一支  
比色瓶中。(空白试  
样)
6. 将空白试样瓶放入  
样品适配器中, 并盖  
上遮光盖。
7. 按下: **ZERO**  
屏幕会显示:  
0 mg/L、SuSld 。
8. 完全地搅拌样品溶  
液。然后迅速将已经  
混合好的样品注入另  
外一支比色瓶中。(预  
制试样)

*注: 通过搅拌或者在  
桌面敲击瓶底来去掉  
水中气泡。*



9. 旋转比色瓶，去除气泡和残渣。



10. 将预制样品瓶放入样品适配器中，盖紧遮光盖。



11. 按下 READ 指针将右移，屏幕将显示悬浮固体的含量，单位为mg/L

## 干扰

该检测法的校正是根据类似样品的处理方法，例如用于都市污水处理厂的比重测定法。对应大多数样品，该校正都可以得到满意的效果。当需要更加精确的结果时，可对相同样品采用并行的光度测定和比重测定法。特殊样品可采取该种以比重测定为基础的校正方法。

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. The sample may be stored seven days by cooling to 4 °C (39 °F).

## Method Performance

### Precision

In a single laboratory, using a standard solution of 847.4 mg/L Suspended Solids with the instrument, a single operator obtained a standard deviation of  $\pm 18.2$  mg/L Suspended Solids. For more information on Hach's precision statement, see *Section 1*.

### Estimated Detection Limit

The estimated detection limit for program 94 is 22.1 mg/L Suspended Solids. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

This method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition and weighing steps that gravimetric procedures do. The USEPA specifies the gravimetric method for solids determinations, while this method is often used for checking in-plant processes.

## 所需仪器

| 种类                          | 要求数量<br>每次测量用量 | 单位      | 货号       |
|-----------------------------|----------------|---------|----------|
| 烧瓶, 600 mL, low form .....  | 1 .....        | 个 ..... | 1080-52  |
| 搅拌器, 1.2 L, 120 V .....     | 1 .....        | 个 ..... | 26161-00 |
| 搅拌器, 1.2 L, 240 V .....     | 1 .....        | 个 ..... | 26161-02 |
| 量筒, 刻度500 mL, .....         | 1 .....        | 个 ..... | 1081-49  |
| 移液管, serologic, 25 mL ..... | 1 .....        | 个 ..... | 2066-40  |
| 吸液球 .....                   | 1 .....        | 个 ..... | 14651-00 |

## OPTIONAL APPARATUS

|                           |             |         |
|---------------------------|-------------|---------|
| Stirring Rod, glass ..... | 3/pkg ..... | 1770-01 |
| Wash Bottle, 250 mL.....  | each .....  | 620-31  |

### *For Technical Assistance, Price and Ordering*

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

# 丹宁酸和木质素 (0 to 9.0 mg/L)

方法号: 8193

## 酪氨酸



1. 输入检测丹宁酸和木质素的程序编号。  
按下: **PRGM**  
屏幕将显示:  
**PRGM?**

2. 按下: **98 ENTER**  
屏幕将显示:  
0.00 mg/L、**tanic**  
和ZERO图标。

3. 往一个干净的比色瓶中装入去离子水到25mL刻度 (空白试样)。

4. 往另一个干净的比色瓶中装入样品到25mL刻度 (待测试样)。

注: 过滤浑浊样品, 结果表示为可溶的丹宁酸, 单位为mg/L。



5. 分别将 0.5 mL TanniVer 3 丹宁酸-木质素试剂注入到两个比色瓶中, 晃动使之混合。

6. 分别将5.0 mL 碳酸钠溶液到两个管中, 晃动使之混合。  
注: 如果存在丹宁和或木质素, 溶液将呈现蓝色。

7. 按下:  
**TIMER ENTER**  
将开始25分钟反应计时。

8. 将空白试样放入样品适配器中。盖紧遮光盖。



9. 按: ZERO  
指针将右移, 屏幕显示:  
0 mg/L tannic

10. 将预制试样瓶放入样品适配器中。盖紧遮光盖。

11. 按下 READ  
指针将右移, 屏幕将显示丹宁酸-木质素的含量, 单位为mg/L。

*注: 在使用预制试样之前应进行标准校正。*

## 干扰

| 干扰物质 | 干扰水平和处理                                                                             |
|------|-------------------------------------------------------------------------------------|
| 三价铁  | 引起正干扰。2mg/L铁离子可导致相当于1 mg/L单宁酸的导致生产的颜色。为除去20mg/L以内的三价铁离子的干扰, 测试前加入一勺0.2-g量的焦磷酸钠到样品中。 |
| 亚硫酸盐 | 可在测试前加入1mL甲醛到样品中除去干扰。                                                               |

## Sampling and Storage

Collect samples in clean plastic or glass bottles.

## Accuracy Check

### Standard Solution Method

Prepare a 200-mg/L tannic acid standard solution by dissolving 0.200 grams of tannic acid in deionized water and diluting to 1000 mL. Prepare this solution monthly. A 2.0 mg/L tannic acid standard is prepared by diluting 10.00 mL of the stock solution to 1000 mL with deionized water. Prepare this standard daily.

## Method Performance

### Precision

In a single laboratory, using standard solutions of 4.0 mg/L tannic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.1$  mg/L tannic acid.

### Estimated Detection Limit

The estimated detection limit for program 98 is 0.1 mg/L tannin and lignin. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

This test measures all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. Report results as total tannin and lignin expressed as mg/L tannic acid.

## 所需试剂

丹宁酸和木质素试剂一套(up to 100 tests) .....22446-00  
包括: (1) 675-49, (1) 2560-42

| 种类                         | 要求数量       |              | 单位      | 货号 |
|----------------------------|------------|--------------|---------|----|
|                            | 每次测量用量     |              |         |    |
| 碳酸钠溶液 .....                | 10 mL..... | 500 mL ..... | 675-49  |    |
| TanniVer 3 丹宁酸和木质素试剂 ..... | 1 mL.....  | 100 mL ..... | 2560-42 |    |
| 去离子水 .....                 | 25 mL..... | 4 L .....    | 272-56  |    |

## 所需仪器

|                                        |         |             |          |
|----------------------------------------|---------|-------------|----------|
| 吸液球 .....                              | 1 ..... | 个 .....     | 14651-00 |
| 移液管, volumetric, Class A, 5.0 mL ..... | 1 ..... | 个 .....     | 14515-37 |
| 移液管, volumetric, Class A, 0.5 mL ..... | 1 ..... | 个 .....     | 14515-34 |
| 样品比色瓶, 10-20-25-mL, w/ cap .....       | 2 ..... | 6/pkg ..... | 24019-06 |

## OPTIONAL REAGENTS

|                                |              |          |
|--------------------------------|--------------|----------|
| Formaldehyde.....              | 100 mL ..... | 2059-32  |
| Sodium Pyrophosphate, ACS..... | 50 g .....   | 14295-25 |
| Tannic Acid .....              | 113 g .....  | 791-14   |

## OPTIONAL APPARATUS

### Description Unit Cat. No

|                                           |              |          |
|-------------------------------------------|--------------|----------|
| Balance, analytical, 115 V .....          | each.....    | 26103-00 |
| Balance, analytical, 230 V .....          | each.....    | 26103-02 |
| Cylinder, graduated, 25 mL .....          | each.....    | 508-40   |
| Filter Paper, folded, 12.5 cm.....        | 100/pkg..... | 1894-57  |
| Flask, volumetric, 1000 mL.....           | each.....    | 547-53   |
| Funnel, poly, 65 mm .....                 | each.....    | 1083-67  |
| Pipet, TenSette, 0.1 to 1.0 mL.....       | each .....   | 19700-01 |
| Pipet Tips, for 19700-01 Pipet .....      | 50/pkg.....  | 21856-96 |
| Pipet, volumetric, Class A, 10.00 mL..... | each.....    | 14515-38 |
| Pipet, Filler, safety bulb .....          | each.....    | 14651-00 |
| Spoon, measuring, 0.2 g.....              | each.....    | 638-00   |
| Weighing Boat, 67/47 mm .....             | 500/pkg..... | 21790-00 |

## For Technical Assistance, Price and Ordering

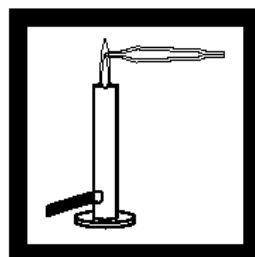
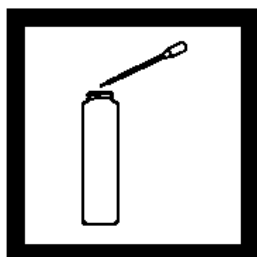
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

## 毒性检测 色度测试法

方法号: 10017

### 疫苗培养



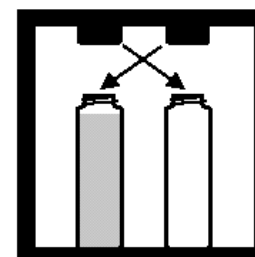
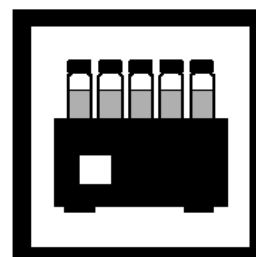
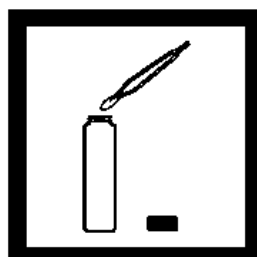
#### 采用本土的生物体

1. 用所提供的其中一支滴定管将 1.0mL 原液加入到胰蛋白酶大豆培养试管内

2. 在37 °C条件下保持培养,直到可以用肉眼清晰看到溶液变为浑浊为止(浑浊表明细菌正在生长);

使用Bactrol疫苗片  
1. 将镊子浸入酒精然后在酒精灯下用火焰消毒

2. 打开 Bactrol 疫苗瓶的瓶盖,用消毒过的镊子夹出一个 Bactrol 疫苗片。



3. 打开月桂醇胰蛋白培养基试管盖,将 Bactrol疫苗片放入试管中,然后晃动使之溶解。

4. 把管放入培养箱中培养至管内产生可见的浑浊。当培养箱为35度而不是在室温下,反应会更快。在35度下一般培养10小时已足够。

5. 通过倒置完成步骤4的试管,为一新的月桂醇胰蛋白培养基试管瓶注入疫苗。然后将两个样品瓶的盖互换。用此新瓶进行以下测试。

注: 如果检测连续进行多天,在培养箱内或室温下可存放疫苗多天。

注: 通过这种方法,一些中间瓶也可以被注入疫苗。

## 比色反应



1. 输入检测毒性的程序编号。

按下: **PRGM**

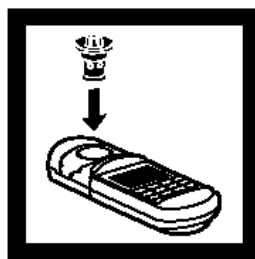
屏幕将显示:

**PRGM?**

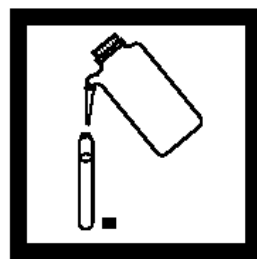


2. 按下: **61 ENTER**  
屏幕将显示:

**ABS 610 nm**和**ZERO**图  
标



3. 旋转COD/TNT适配器, 将其嵌入瓶管架上适当的位置, 然后下按使之完全嵌入。



4. 将去离子水加入到1个测试 ‘N’ 样品瓶内。为该瓶注上 “空白” 标签。擦拭样品管外表面, 不要残留指纹和其它污迹。

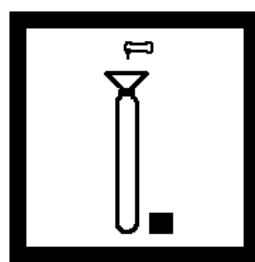


5. 将空白试样放入样品适配器中, 并盖紧遮光盖。

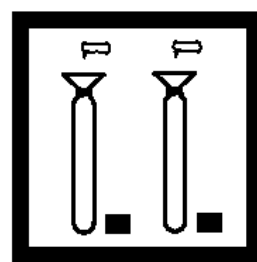


6. 按下 **ZERO**  
指针将右移, 屏幕将显示:

**0.000 ABS**



7. 为另外一支样品瓶注上 “控制” 标签。打开一包ToxTrak试剂粉包, 将粉末加入到空的反应瓶中。



8. 为每个样本瓶清楚地注上标签(s1、s2), 该样品试瓶中是用于装样品或稀释液。然后往样品瓶内加入ToxTrak试剂粉末。



9. 将 5 毫升的去离子水加入到控制瓶中。



10. 向每个样品试瓶中加入5.0mL制备的样品液（或稀释液）。



11. 为每个试瓶加入2滴催化剂,封盖并摇至混合均匀。



12. 为每个试瓶加入0.5mL 疫苗培养液(前面所制备的),封盖和倒置混合。

注：为了测试毒性的  
大约的开始点,可以  
用去离子水稀释1mL  
样品液至10mL,然后  
进行测试,观察是否  
指示为0%。如果不是  
的话，就对该稀释液  
按1/10进行稀释,直  
到达到如第18步所示  
的0%的抑制度的要  
求。



13. 将控制瓶放入样品适配器中，盖紧遮光盖。



14. 按下：READ  
指针将右移，屏幕将  
显示ABS的结果。记录  
下该控制瓶中样品的  
吸收率。



15. 重复步骤13—14  
步，对每个样品和稀  
释液进行测试，并记  
录相应的吸收率。.



16. 让试管瓶中的溶  
液反应，直到控制瓶  
中溶液的吸光率减少  
至  $0.60 \pm 0.10$ 。该过  
程大概要花 45—75  
分钟。



17. 当控制瓶中溶液的吸光率减少至 0.60 ±0.10 吸收单元时，将空白试样放入样品适配器中，盖紧遮光盖。

18. 按下 **ZERO** 指针将右移，屏幕将显示：  
**0.000 ABS**

19. 将控制瓶放入样品适配器中，盖紧遮光盖。并记录其相应的吸收率。

20. 将每个样品和稀释液放入样品适配器中进行测试，盖紧遮光盖。记录它们相应的吸收率。



21. 用一下公式计算抑制度%：

$$\%I = \left[ 1 - \left( \frac{\Delta \text{Abs sample}}{\Delta \text{Abs control}} \right) \right] \times 100$$

其中：ΔA表示初始的吸收率减去最终的吸收率的值。详细情况见下例。

*注：在以呼吸作用为基础的毒性测试中，某些毒素会增加呼吸使抑制率为负数。重复实验，计算结果比-10%更小则视为有毒。*

例：如控制试管中溶液初始吸收率为1.6Abs，最后下降到1.0Abs。样品试管溶液初始吸收率为1.7Abs，最后下降到1.3Abs。

$$\Delta \text{Abs. Sample} = 1.7 - 1.3 = 0.4 \quad \Delta \text{Abs. Control} = 1.6 - 1.0 = 0.6$$

$$\%I = \left( 1 - \left( \frac{0.4}{0.6} \right) \right) \times 100$$

$$\%I = 33.3$$

## Disposal of Test Cultures

Dispose of active bacterial cultures by using one of these methods:

1. Autoclave used test containers at 121 °C for 15 minutes at 15 pounds of pressure. Once the containers are sterile, pour the contents down the drain with running water. The reaction tubes may be washed and re-used.
2. Sterilize test containers by using a 1:10 dilution of commercial laundry bleach. Pour the test container contents and test containers into the bleach solution. Allow 10-15 minutes of contact time with the bleach solution. Then pour the liquid down the drain and wash the reaction tubes for re-use.

## Summary of Method

Resazurin is a redox-active dye, which changes from pink to blue when it is reduced. Bacterial respiration occurring in the sample reduces resazurin. If toxic substances are present, they inhibit the rate of resazurin reduction. The sample color is compared to a toxin-free control tube to determine how toxic the sample is to an indigenous culture or a culture of *E. coli*. A chemical accelerant reduces the reaction time of the procedure.

### 所需试剂

| 种类                                                                       | 每次测量用量 | 单位 | 货号       |
|--------------------------------------------------------------------------|--------|----|----------|
| ToxTrak 试剂一套 (25 tests)                                                  |        |    | 25972-00 |
| 包括: (1) 25607-66, (1) 25608-36, (1) 22336-15, (2) 21247-20, (2) 20962-08 |        |    |          |

### 要求数量

| 种类           | 每次测量用量 | 单位         | 货号       |
|--------------|--------|------------|----------|
| ToxTrak 粉末试剂 | 1 包    | 50/pkg     | 25607-66 |
| ToxTrak促进剂溶液 | 2 滴    | 15 mL SCDB | 25608-36 |
| 胰蛋白酶大豆孵化试管   | 1      | 15/pkg     | 22336-15 |
| 带盖培养管        | 1      | 10/pkg     | 20962-08 |
| 去离子水         | 不定     | 200 mL     | 272-29   |

### 所需仪器

|                                   |    |        |          |
|-----------------------------------|----|--------|----------|
| 大剪刀                               | 1  | 个      | 936-00   |
| COD/TNT 适配器                       | 1  | 个      | 48464-00 |
| 下滴移液管, 1 mL                       | 不定 | 10/pkg | 21247-20 |
| 镊子                                | 1  | 个      | 14537-00 |
| 移液管, volumetric, 5.00 mL, Class A | 1  | 个      | 14515-37 |
| 吸液球                               | 1  | 个      | 14651-00 |
| Test 'N' 试样瓶                      | 1  | 25/pkg | 25831-25 |

## OPTIONAL REAGENTS

Culture Set:

|                                                       |             |          |
|-------------------------------------------------------|-------------|----------|
| (incl. Bactrol Discs and Lauryl Tryptose Broth Tubes) | 25 cultures | 25978-00 |
| Bactrol Discs, <i>E. coli</i>                         | 25/bottle   | 25809-25 |
| Isopropanol                                           | 500 mL      | 14459-49 |
| Lauryl Tryptose Broth Tubes                           | 15/pkg      | 21623-15 |

## OPTIONAL APPARATUS

|                                                                        |             |          |
|------------------------------------------------------------------------|-------------|----------|
| Burner, Alcohol, 60 mL.....                                            | each.....   | 20877-42 |
| Burner, Bunsen .....                                                   | each.....   | 21627-00 |
| Germicidal Cloth.....                                                  | 50/pkg..... | 24632-00 |
| Incubator, Dri Bath, 25 well, 120/230 V .....                          | each.....   | 45900-00 |
| Incubator, Dri Bath, 25 well, 120/230 V, with European power cord..... | each.....   | 45900-02 |
| Test Tube Rack.....                                                    | each.....   | 24979-00 |
| Timer.....                                                             | each.....   | 26305-00 |

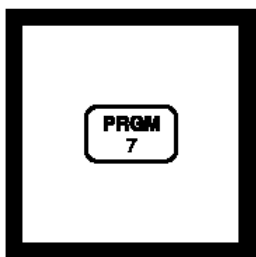
### ***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 浊度 吸收测定法 (0 to 1000 FAU)

方法号: 8237



1. 输入检测浊度的程序编号。

按下: PRGM

屏幕将显示:

PRGM?

注: 当检测formazin的时候:

1 FAU=1 NTU=1 FTU

当检测其他类型的标准液和样品时, 该等式不成立。

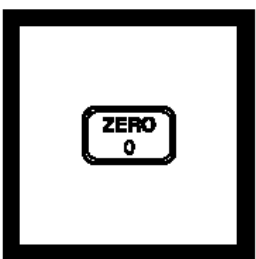
2. 按下: 95 ENTER  
屏幕将显示:

FAU和ZERO图标

3. 将10毫升的去离子水注入一支比色瓶中。(空白试样)

注: 对于高色度的样品使用过滤和的部分样品代替去离子水。

4. 将空白试样瓶放入样品适配器中。盖紧遮光盖。



5. 按下 ZERO  
指针将右移, 屏幕会显示: 0 FAU

6. 将10毫升的样品注入另外一支比色瓶中

7. 将预制试样瓶放入样品适配器中。盖紧遮光盖。

8. 按下 READ  
指针将右移, 屏幕会显示Formazin衰减值 (FAU)。

注: 在使用预制标准试样时将应进行标准校正。

## 干扰

| 干扰物质 | 干扰水平和处理方法                                       |
|------|-------------------------------------------------|
| 气泡   | 在所有水平都会产生干扰。使用排气工具或者超声波容器容器 (ultrasonic bath) 。 |
| 颜色   | 如果颜色的吸光率到达520nm时产生干扰。                           |
| 温度极限 | 改变样品的浊度可能产生干扰。样品一经采集应立即进行检测。在原始样品相同的温度条件下进行检测。  |

## Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible. Store samples up to 48 hours by cooling to 4 °C (39 °F). Analyze the sample at the same temperature as it was collected.

## Accuracy Check

### Standard Solution Method

The stored program has been calibrated using formazin, the primary standard for turbidity. A 200 FAU formazin solution for checking the accuracy of the test can be prepared using the following procedure.

1. Pipet 5.00 mL of a 4000 NTU Formazin stock solution into a 100-mL volumetric flask.
2. Dilute to the mark with deionized water. Prepare this daily. Convenient stabilized turbidity stock solution (200 NTU StablCal™ Standard) is available from Hach.

### Standard Adjust

To adjust the calibration curve using the reading obtained with the 200 FAU formazin standard, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **200** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

## Method Precision

### Precision

In a single laboratory, using a turbidity standard solution of 200 FAU with the instrument, a single operator obtained a standard deviation of  $\pm 2$  FAU.

### Estimated Detection Limit

The estimated detection limit for program 95 is 21 FAU. For more information on the estimated detection limit, see *Section 1*.

## Summary of Method

This turbidity test measures an optical property of the sample which results from scattering and absorption of light by particles in the sample. The amount of turbidity measured depends on variables such as the size, shape, color, and refractive properties of the particles. This procedure is calibrated using formazin turbidity standards and the

readings are in terms of Formazin Attenuation Units (FAU). This test cannot be used for USEPA reporting purposes, but it may be used for daily in-plant monitoring. One FAU is equivalent to one Nephelometric Turbidity Unit (NTU) of Formazin. However, the optical method of measurement for FAU is very different than the NTU method (1 NTU = 1 FTU = 1 FAU when traced to formazin primary standards.)

#### 所需仪器

| 种类                              | 要求数量<br>每次测量用量 | 单位          | 货号       |
|---------------------------------|----------------|-------------|----------|
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2 .....        | 6/pkg ..... | 24019-06 |

#### 所需试剂

| 种类                                                   | 每次测量用量         | 单位 | 货号       |
|------------------------------------------------------|----------------|----|----------|
| Formazin 溶液, 4000 NTU .....                          | 500 mL .....   |    | 2461-49  |
| 硅树脂油 .....                                           | 15 mL DB ..... |    | 1269-36  |
| StablCal Stabilized Turbidity Standard, 200 NTU..... | 500 mL .....   |    | 26604-49 |
| 去离子水 .....                                           | 4 L .....      |    | 272-56   |

#### OPTIONAL APPARATUS

| Description                                   | Units        | Cat. No. |
|-----------------------------------------------|--------------|----------|
| Bath, ultrasonic .....                        | each.....    | 24895-00 |
| Bottle, wash, 250 mL.....                     | each.....    | 620-31   |
| Flask, volumetric, Class A, 100 mL .....      | each.....    | 14574-42 |
| Flask, filter, 500 mL.....                    | each.....    | 546-49   |
| Filter Holder.....                            | each.....    | 13529-00 |
| Filter Pump, aspirator .....                  | each.....    | 2131-00  |
| Oiling cloth, for applying silicone oil ..... | each.....    | 26873-00 |
| Pipet Filler, safety bulb .....               | each.....    | 14651-00 |
| Pipet, volumetric, Class A, 5.0 mL.....       | each.....    | 14515-37 |
| Sample Degassing Kit.....                     | each.....    | 43975-00 |
| Stopper, rubber, one-hole, No. 7 .....        | 6/pkg.....   | 2119-07  |
| Tubing, rubber, 5/16" I.D.....                | 12 feet..... | 560-19   |
| Tweezers, plastic .....                       | each.....    | 14282-00 |

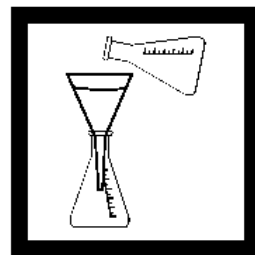
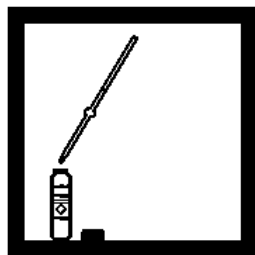
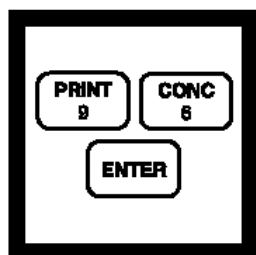
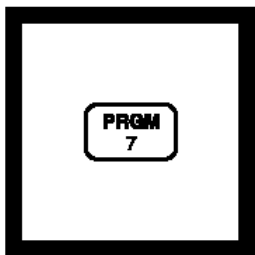
#### *For Technical Assistance, Price and Ordering*

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# 挥发性酸 酯化法 (0 - 2800 mg/L HOAc)

方法号: 8196



1. 输入挥发性酸（醋酸）的程序编号。  
按下: PRGM  
屏幕将显示:  
PRGM?

注: 为得到更加精确的结果, 应使用去离子水进行试剂空白校正。

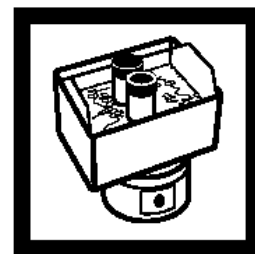
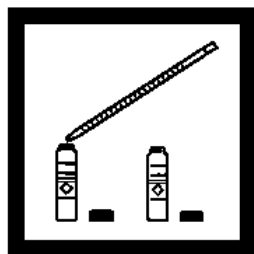
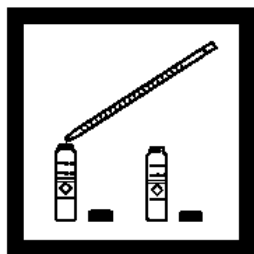
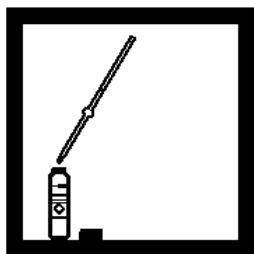
2. 按下: 96 ENTER  
屏幕将显示:  
mg/L, HOAc和ZERO图标

注: 如果酸中存在大量的已溶解的固体物质和矿物质, 应先采用蒸馏的方法处理。

3. 将0.5毫升的去离子水加入到一个25mL的干燥的比色瓶（空白试样）

注: 使用A型或TenSette移液管。  
注: 在分析前, 调整所存样品的PH值。

4. 过滤或者用离心机分离25毫升的样品。  
注: 离心处理比渗透作用样快。



5. 取出0.5mL过滤液或上清液到另一个25mL的干燥比色瓶中（待测试样）。

6. 将1.5mL 乙烯乙二醇注入到每个比色瓶中。旋转混合。

7. 将0.2mL的19.2N的硫酸标准溶液注入到每个比色瓶中。旋转混合。

8. 将两个比色瓶放入沸水浴中。  
注: 可以在600mL的大口杯中加热比色瓶。



9. 按下：  
**TIMER ENTER**  
开始3分钟的反应计时。



10. 当计时器鸣叫时，用流动的自来水将冷却溶液到25 ° C（直到比色瓶冷却到该温度）。用软布



11. 使用移液管，吸取0.5 mL的羟胺氢氧化物溶液到每个比色瓶中。旋转使之混合。



12. 使用移液管，量取 2.0 mL 的 4.5N 的氢氧化钠溶液到每个管。旋转使之混合



13. 加入10mL的硫酸氯化铁溶液到每个管。旋转混合



14. 加入10mL的去离子水到每个管。旋转混合。



15. 屏幕将显示：  
**3:00 TIMER 2**  
按下：**ENTER**  
开始3分钟的反应计时。



16. 当计时器鸣响后，迅速将空白试样瓶放入样品适配器中，盖紧遮光盖。

*注：在3分钟的反应完成后，迅速完成步骤15-16。*



17. 按下: **ZERO**  
指针将右移, 屏幕显示: **0 mg/L HOAc**
18. 将预制试样瓶放入样品适配器中, 盖紧遮光盖。
19. 按下: **READ**  
指针将右移, 屏幕会显示挥发性酸(醋酸)的含量, 单位是mg/L。

## Sampling and Storage

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before testing.

## Accuracy Check

### Standard Additions Method

- Snap the neck off a Volatile Acids PourRite Ampule Standard Solution, 62,500 mg/L as acetic acid.
- Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL graduated mixing cylinders, each containing 25 mL of filtered sample. Stopper. Shake well to mix.
- Remove a 0.5 mL aliquot of sample from each cylinder; add to three dry sample cells. Analyze all three samples along with the original test sample beginning with Step 5 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* in *Section 1*.

### Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids PourRite Ampule Standard Solution (62,500 mg/L as acetic acid) to a 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix.

## Method Performance

### Precision

In a single laboratory, using a standard solution of 500 mg/L volatile acids as acetic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 8$  mg/L.

### Estimated Detection Limit

The estimated detection limit for program 96 is 17 mg/L HOAc. For more information on the estimated detection limit, see *Section 1*.

### Summary of Method

The volatile acids test is designed specifically for the determination of volatile acids in digester sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

### 所需试剂

|                                                                    | 货号       |
|--------------------------------------------------------------------|----------|
| 挥发性酸试剂一套 (90 tests) .....                                          | 22447-00 |
| 包括: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32 |          |

| 种类                     | 要求数量<br>每次测量用量 | 单位           | 货号      |
|------------------------|----------------|--------------|---------|
| 乙烯乙二醇 .....            | 3 mL.....      | 1000 mL..... | 2039-53 |
| 氯化铁-硫磺酸溶液 .....        | 20 mL.....     | 1000 mL..... | 2042-53 |
| 羟胺氢氧化物溶液, 100 g/L..... | 1 mL.....      | 100 mL.....  | 818-42  |
| 氢氧化钠标准溶液, 4.5 N .....  | 4 mL.....      | 1000 mL..... | 2040-53 |
| 硫磺酸标准溶液, 19.2 N .....  | 0.4 mL .....   | 100 mL.....  | 2038-32 |
| 去离子水 .....             | 20.5 mL .....  | 4 L.....     | 272-56  |

### 所需仪器

| 种类                                      | 要求数量<br>每次测量用量 | 单位            | 货号       |
|-----------------------------------------|----------------|---------------|----------|
| 指套.....                                 | 2 .....        | 2/pkg .....   | 14647-02 |
| 具有刻度的量筒, 10 mL.....                     | 1 .....        | 个 .....       | 508-38   |
| 可折叠滤纸, 12.5 cm .....                    | 1 .....        | 100/pkg ..... | 1894-57  |
| 锥形烧瓶, 50 mL.....                        | 1 .....        | 个.....        | 505-41   |
| 聚乙烯漏斗, 65 mm.....                       | 1 .....        | 个.....        | 1083-67  |
| 圆形加热炉, 3.5-inch 直径.....                 | 1 .....        | 个.....        | 12067-01 |
| 吸液球 .....                               | 1 .....        | 个.....        | 14651-00 |
| 移液管t, serological, 2 mL.....            | 2 .....        | 个.....        | 532-36   |
| 移液管, volumetric, Class A, 0.5mL .....   | 3 .....        | 个.....        | 14515-34 |
| 移液管, volumetric, Class A, 10.00mL ..... | 3 .....        | 个.....        | 14515-38 |
| 样品比色瓶l, 10-20-25 mL, w/cap .....        | 2 .....        | 6/pkg .....   | 24019-06 |
| 水盆和支架.....                              | 1 .....        | 个.....        | 955-55   |

### OPTIONAL REAGENTS

|                                                                                              |              |          |
|----------------------------------------------------------------------------------------------|--------------|----------|
| Volatile Acids Standard Solution, PourRite ampule,<br>62,500 mg/L as acetic acid, 2 mL ..... | 16/pkg ..... | 14270-20 |
|----------------------------------------------------------------------------------------------|--------------|----------|

### OPTIONAL APPARATUS

|                                |            |          |
|--------------------------------|------------|----------|
| Ampule Breaker, PourRite ..... | each ..... | 24846-00 |
|--------------------------------|------------|----------|

|                                                 |              |          |
|-------------------------------------------------|--------------|----------|
| Beaker, 600 mL .....                            | each .....   | 500-52   |
| Bottle, wash, 500 mL .....                      | each .....   | 620-11   |
| Centrifuge, laboratory, 115 Vac.....            | each .....   | 26765-00 |
| Centrifuge, laboratory, 230 Vac.....            | each .....   | 26765-02 |
| Centrifuge Tubes, 15 mL.....                    | 10/pkg ..... | 22787-39 |
| Centrifuge Tube Caps.....                       | 20/pkg ..... | 25852-20 |
| Cylinder, graduated, mixing, 25 mL .....        | each .....   | 1896-40  |
| Cylinder, graduated, plastic, 250 mL .....      | each .....   | 1081-46  |
| Distillation Apparatus .....                    | each .....   | 26353-00 |
| Distillation Heater and Support Apparatus ..... | each .....   | 22744-00 |
| Flask, volumetric, Class A, 100 mL.....         | each .....   | 14574-42 |
| Pipet, TenSette, 0.1 to 1.0 mL.....             | each .....   | 19700-01 |
| Pipet Tips, for 19700-01 TenSette Pipet .....   | 50/pkg ..... | 21856-96 |
| Pipet, TenSette, 1.0 to 10.0 mL.....            | each .....   | 19700-10 |
| Pipet Tips, for 19700-10.....                   | 50/pkg ..... | 21997-96 |

***For Technical Assistance, Price and Ordering***

**In the U.S.A.—Call 800-227-4224**

**Outside the U.S.A.—Contact the Hach office or distributor serving you.**

## 锌 (0-3.00mg/L Zn)

方法号: 8009



1、按“PRGM”键，  
萤幕会显示 PRGM?



2、输入内程式代号  
“ 97 ” 然后 按下  
“ENTER” 键，萤幕会  
出现 “mg/l, Zn” 及  
“ZERO icon”



3、取一支 25ml 比色瓶  
加水样至 20ml 标线处  
  
注：在使用前，取浓度  
1: 1 的 HCL，清洗玻璃  
器皿，再以去离子水冲  
洗



4 、 加 入 一  
ZincoVer5 试剂至  
比色瓶中，盖好瓶  
盖摇动数次使充分  
溶解。

注：试剂必须完全  
溶解否则会得到不  
稳定的结果

注：所测得结果水  
样必须为橘红色，  
假如最后水样呈棕  
色或蓝色，请将水  
样稀释后重复测试  
(此结果通常在水  
样所含锌浓度太高  
或有干扰存在时出  
现)



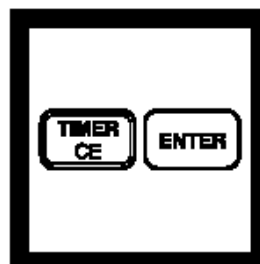
5、自步骤 4 中取 10ml  
呈橘红色水样，加入另  
一支比色瓶中，当作空  
白溶液。



6、加入0.5ml  
cyclohexanone  
试剂至步骤 4,橘红色  
水样中，当作待测溶  
液。  
  
注：以塑胶吸管取用  
试剂橡胶吸球会污染  
试剂。



7、将待测溶液比色  
瓶，盖上瓶盖，剧烈  
摇动比色瓶约 30 秒  
  
注：此时水样视所含  
锌离子浓度不同，可  
能呈红橘色，棕色或  
蓝色。



8、同时按“TIMER”  
及“ENTER”键，将  
进行 3 分钟的反应  
计时  
  
注：当计时完毕，  
听到哔哔声后进行  
步骤 9-11 之操作  
(必须在 10 分钟内  
进行完毕)。



- 9、放空白溶液至比色计中测试，并将比色计盖子盖上
- 10、按“ZERO”键归零 萤幕会显示：0.00mg/l Zn
- 11、放待测溶液至比色计中，并将比色计盖子盖好
- 12、按“READ”键，所欲测浓度将会显示出来，即 mg/l Zn

## 干扰

下列物质当浓度超过以下所列情况时，会产生干扰。

| 干扰物质           | 干扰水平和处理办法                         |
|----------------|-----------------------------------|
| 铝              | 6 mg/L                            |
| 铬              | 0.5 mg/L                          |
| 铜              | 5 mg/L                            |
| 铁              | 7 mg/L                            |
| 锰              | 5 mg/L                            |
| 镍              | 5 mg/L                            |
| 有机物            | 大量时产生干扰。要进行温和消解处理。                |
| 高缓冲溶液和过高或过低PH值 | 可能超出了试剂的缓冲能力，需要进行样品预处理。调整PH值到4—5。 |

## Sampling and Storage

Collect samples in acid-washed plastic bottles. For storage, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). The preserved samples can be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as zinc may be lost as a precipitate. Correct the test result for volume additions; see *Sampling and Storage, Volume Additions*, in *Section 1* for more information. If only dissolved zinc is to be determined, filter the sample before the acid addition.

## Accuracy Check

### Standard Additions Method

- Snap the neck off a Zinc PourRite Ampule Standard, 25 mg/L Zn.
- Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.
- Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.1 mL of standard added.
- If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

### Standard Solution Method

Prepare a 0.50 mg/L zinc standard solution by diluting 5.00 mL of Zinc Standard Solution, 100 mg/L as Zn, to 1000 mL with deionized water in a Class A 1000-mL volumetric flask. Prepare this solution daily. Use this solution as the sample and perform the zinc procedure as described above.

### Method Performance

#### Precision

In a single laboratory, using a standard solution of 1.50 mg/L Zn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L Zn.

#### Estimated Detection Limit (EDL)

The EDL for program 97 is 0.02 mg/L Zn. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

### Pollution Prevention and Waste Management

ZincoVer 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent the release of hydrogen cyanide gas.

In the event of a spill or release, clean up the area by following these steps:

- a) Use a fume hood or supplied-air or self-contained breathing apparatus.
- b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
- c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
- d) Neutralize and flush the solution down the drain with a large excess of water.

### Summary of Method

Zinc and other metals in the sample complex with cyanide.

Adding cyclohexanone selectively releases zinc. The zinc then reacts with the 2-carboxy-2'-hydroxy-5'-sulfoformazyl benzene (zincon) indicator and forms a blue color that is proportional to the zinc concentration.

### 所需试剂

锌试剂一套, 20 mL size (100 tests) .....24293-00  
包括: (1) 14033-32, (1) 21066-69

| 种类                              | 要求数量   |     | 单位     | 货号       |
|---------------------------------|--------|-----|--------|----------|
|                                 | 每次测量用量 |     |        |          |
| 环己酮 .....                       | 0.5 mL | 100 | mL MDB | 14033-32 |
| ZincoVer 5 粉末试剂 .....           | 1 包    | 100 | /pkg   | 21066-69 |
| 所需仪器                            |        |     |        |          |
| 移液管, 10 mL .....                | 1      |     | each   | 532-38   |
| 吸液球 .....                       | 1      |     | each   | 14651-00 |
| 样品比色瓶, 10-20-25 mL, w/cap ..... | 2      |     | 6/pkg  | 24019-06 |
| 压榨器, 塑料点滴器 .....                | 1      |     | 20/pkg | 21247-20 |

## OPTIONAL REAGENTS

|                                                                   |                  |             |
|-------------------------------------------------------------------|------------------|-------------|
| Bleach, household .....                                           | 1 gal .....      | buy locally |
| Cylinder, graduated, mixing, 25mL .....                           | each .....       | 20886-40    |
| Hydrochloric Acid Standard Solution, 6 N .....                    | 500 mL .....     | 884-49      |
| Nitric Acid, ACS .....                                            | 500 mL .....     | 152-49      |
| Nitric Acid 1:1 .....                                             | 500 mL .....     | 2540-49     |
| Sodium Hydroxide Standard Solution, 5.0 N .....                   | 50 mL SCDB ..... | 2450-26     |
| Water, deionized .....                                            | 4 L .....        | 272-56      |
| Zinc Standard Solution, 100 mg/L Zn .....                         | 100 mL .....     | 2378-42     |
| Zinc Standard Solution, PourRite ampule, 25 mg/L as Zn, 2mL ..... | 20/pkg .....     | 14246-20    |

## OPTIONAL APPARATUS

|                                           |               |          |
|-------------------------------------------|---------------|----------|
| Ampule Breaker, PourRite ampules .....    | each .....    | 24846-00 |
| Aspirator, vacuum .....                   | each .....    | 2131-00  |
| Beaker, glass, 1000 mL .....              | each .....    | 500-53   |
| Cylinder, graduated, 100 mL .....         | each .....    | 508-42   |
| Cylinder, graduated, mixing, 250 mL ..... | each .....    | 26362-46 |
| Filter discs, glass, 47 mm .....          | 100/pkg ..... | 2530-00  |
| Filter holder, 47 mm .....                | each .....    | 2340-00  |
| Flask, erlenmeyer, 250 mL .....           | each .....    | 505-46   |
| Flask, filtering, 500 mL .....            | each .....    | 546-19   |

## OPTIONAL APPARATUS (continued)

| Description                                  | Units             | Cat. No. |
|----------------------------------------------|-------------------|----------|
| Flask, volumetric, Class A, 100 mL .....     | each .....        | 14574-42 |
| Flask, volumetric, Class A, 1000 mL .....    | each .....        | 14574-53 |
| Hot plate, micro 115 V .....                 | each .....        | 12067-01 |
| Hot plate, micro 230 V .....                 | each .....        | 12067-02 |
| pH paper, 1 to 11 pH .....                   | 5 rolls/pkg ..... | 391-33   |
| pH meter, <i>Sension™ I</i> , portable ..... | each .....        | 51700-00 |
| Pipet filler, safety bulb .....              | each .....        | 14651-00 |
| Pipet, serological, 2 mL .....               | each .....        | 532-36   |
| Pipet, TenSette, 0.1 to 1.0 mL .....         | each .....        | 19700-01 |
| Pipet, TenSette, tips for 19700-01 .....     | 50/pkg .....      | 21856-96 |
| Pipet, TenSette, 1.0 to 10.0 mL .....        | each .....        | 19700-10 |
| Pipet, TenSette, tips for 19700-10 .....     | 50/pkg .....      | 21997-96 |
| Pipet, TenSette, tips for 19700-10 .....     | 250/pkg .....     | 21997-25 |
| Pipet, volumetric, Class A, 5.00 mL .....    | each .....        | 14515-37 |
| Pipet, volumetric, Class A, 0.5 mL .....     | each .....        | 14515-34 |

## For Technical Assistance, Price and Ordering

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