

✓Method 8051

SulfaVer 4 Method*

Powder Pillows or AccuVac® Ampuls

(2 to 70 mg/L)

Scope and Application: For water, wastewater, and seawater; USEPA accepted for reporting wastewater analyses

* Adapted from *Standard Methods for the Examination of Water and Wastewater*. Procedure is equivalent to USEPA method 375.4 for wastewater.



Tips and Techniques

- You must adjust the standard curve for each new lot of reagent. See *Standard Solutions* following these steps.
- For best results, perform a new calibration for each lot of reagent. See *Calibration Standard Preparation* following these steps.
- For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the instrument manual for more information on *Running a Reagent Blank*.
- Filter highly colored or turbid samples using filter paper (Cat. No. 1894-57) and a funnel (Cat. No. 1083-67). Use this sample in *step 5*.
- After adding reagent to the sample, a white turbidity will form if sulfate is present.
- Undissolved powder that has settled does not affect accuracy.
- SulfaVer® 4 contains barium chloride. The final solution will contain barium chloride (D005) at a concentration regulated as a hazardous waste by the Federal RCRA. See *Section 5* for more information on proper disposal of these materials.



Powder Pillows

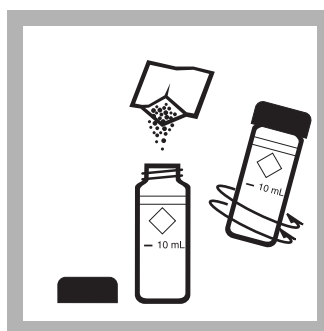
Method 8051



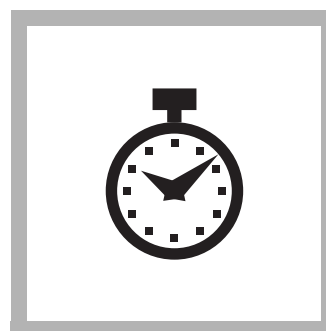
1. Touch **Hach Programs**.
Select program **680 Sulfate**.
Touch **Start**.



2. Fill a clean sample cell with 10 mL of sample.



3. Add the contents of one SulfaVer 4 Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.



4. Touch the timer icon. Touch **OK**.
A five-minute reaction period will begin. Do not disturb the cell during this time.

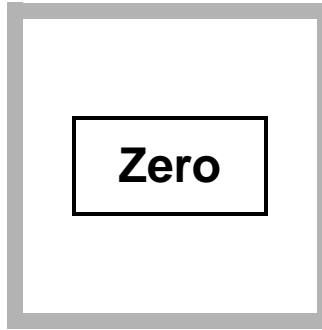
Sulfate



5. Fill a second sample cell with 10 mL of sample (the blank).



6. When the timer beeps, place the blank into the cell holder.



7. Touch **Zero**.
The display will show:
0 mg/L SO₄²⁻



8. Within five minutes after the timer beeps, place the prepared sample into the cell holder.

Results will appear in mg/L SO₄²⁻.



9. Clean the sample cells with soap and a brush.

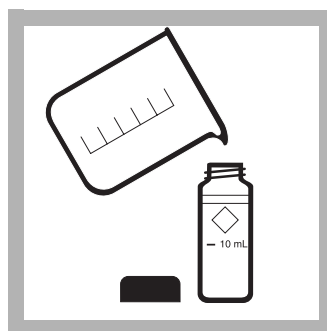


AccuVac Ampul

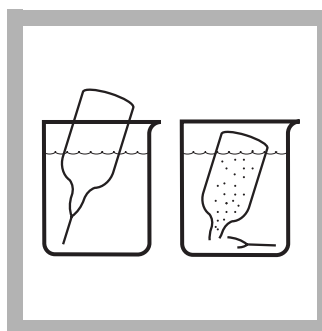
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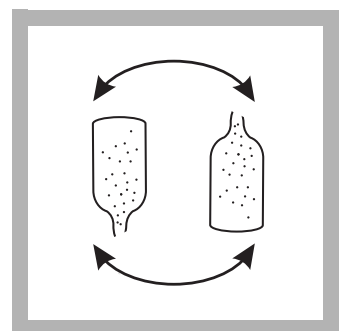
1. Touch **Hach Programs**.
Select program
685 Sulfate AV.
Touch **Start**.



2. Fill a clean sample cell with 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



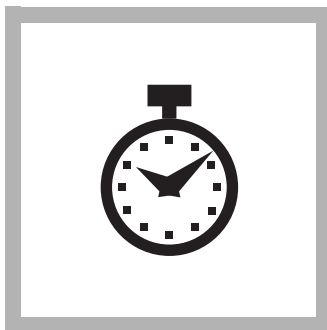
3. Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample. Keep the tip immersed until the ampule fills completely.



4. Quickly invert the ampule several times to mix.



5. Wipe off any liquid or fingerprints from the blank and the ampule.

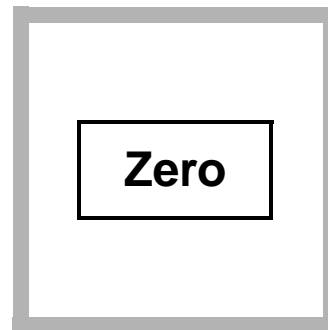


6. Touch the timer icon. Touch **OK**.

A five-minute reaction period will begin. Do not disturb the cell during this time.



7. When the timer beeps, place the blank into the cell holder.



8. Touch **Zero**. The display will show:

0 mg/L SO₄²⁻



9. Within five minutes after the timer beeps, place the ampule into the cell holder.

Results will appear in mg/L SO₄²⁻.

Interferences

| Interfering Substance | Interference Levels and Treatments |
|-----------------------|---|
| Calcium | Greater than 20,000 mg/L as CaCO ₃ |
| Chloride | Greater than 40,000 mg/L as Cl |
| Magnesium | Greater than 10,000 mg/L as CaCO ₃ |
| Silica | Greater than 500 mg/L as SiO ₂ |

Sample Collection, Storage, and Preservation

Collect samples in clean plastic or glass bottles. Samples may be stored up to 7 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Touch **Options**. Touch **Standard Additions**. A summary of the standard additions procedure will appear.
3. Touch **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Touch **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See *Standard Additions* in the instrument manual for more information.
4. Snap the neck off a Sulfate 2-mL Ampule Standard, 1000-mg/L sulfate.
5. Prepare three sample spikes. Fill three mixing cylinders (Cat. No. 1896-40) with 25 mL of sample. Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Transfer 10 mL of each sample spike to a clean sample cell and analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by touching **Read**. Each addition should reflect approximately 100% recovery.

Note: For AccuVac Ampuls, fill three Mixing Cylinders (Cat. No. 1896-41) with 50 mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL Beakers (Cat. No. 500-41H). Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by touching **Read**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, touch **Graph** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Touch **View: Fit**, then select **Ideal Line** and touch **OK** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

See *Section 3.2.2 Standard Additions* on page 26 for more information.

Standard Solutions

Prepare a 70-mg/L sulfate standard solution as follows:

1. Using Class A glassware, Pipet 7 mL of Sulfate Standard Solution, 1000-mg/L, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the SulfaVer procedure as described above.
2. To adjust the calibration curve using the reading obtained with the 70-mg/L standard solution, touch **Options** on the current program menu. Touch **Standard Adjust**.
3. Touch **On**. Touch **OK** to accept the displayed concentration. If an alternate concentration is used, touch **Adjust** and then enter the actual concentration. Touch **OK**.

See *Section 3.2.4 Adjusting the Standard Curve* on page 29 for more information.

Calibration Standard Preparation

To perform a sulfate calibration using the SulfaVer method, use Class A glassware to prepare calibration standards containing 10, 20, 30, 40, 50, 60 and 70 mg/L SO_4^{2-} as follows:

1. Into seven different 100-mL Class A volumetric flasks, pipet 1, 2, 3, 4, 5, 6, and 7 mL of the 1000-mg/L Sulfate Standard Solution.
2. Dilute to the mark with deionized water. Mix thoroughly.
3. Using the SulfaVer method and the calibration procedure described in the User-Entered Programs section of the spectrophotometer *Instrument Manual*, generate a calibration curve from the calibration standards prepared above.

Method Performance

Precision

Standard: 30 mg/L SO_4^{2-}

| Program | 95% Confidence Limits of Distribution |
|---------|---------------------------------------|
| 680 | 27–33 mg/L SO_4^{2-} |
| 685 | 18–43 mg/L SO_4^{2-} |

See *Section 3.4.3 Precision* on page 33 for more information, or if the standard concentration did not fall within the specified range.

Sensitivity

| Program | ΔAbs | $\Delta\text{Concentration}$ |
|---------|--------------------|------------------------------|
| 680 | 0.010 | 1 mg/L SO_4^{2-} |
| 685 | 0.010 | 2 mg/L SO_4^{2-} |

See *Section 3.4.5 Sensitivity* on page 34 for more information.

Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 and form a precipitate of 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. Test results are measured at 450 nm.

Required Reagents

| Description | Quantity Required | | Unit | Cat. No. |
|---|-------------------|--|--------------|----------|
| | per test | | | |
| SulfaVer [®] 4 Reagent Powder Pillows | 1 | | 100/pkg..... | 21067-69 |
| <i>or</i> | | | | |
| SulfaVer [®] 4 Sulfate Reagent AccuVac Ampuls..... | 1 | | 25/pkg..... | 25090-25 |

Required Apparatus

| | | | | |
|----------------------------------|---|--|------------|----------|
| Beaker, 50-mL | 1 | | each..... | 500-41H |
| Sample cells, 10-mL, w/cap | 2 | | 6/pkg..... | 24276-06 |

Required Standards

| | | | | |
|---|--|--|----------------|----------|
| Sulfate Standard Solution, 1000-mg/L | | | 500 mL..... | 21757-49 |
| Sulfate Standard Solution, 1000-mg/L, 2-mL Ampules | | | 20/pkg..... | 21757-20 |
| Wastewater Standard, Effluent Inorganics, Mixed Parameter | | | 500 mL..... | 28332-49 |
| Water, deionized | | | 4 liters | 272-56 |



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