

USEPA<sup>1</sup> SulfaVer 4 Method<sup>2</sup>2 to 70 mg/L SO<sub>4</sub><sup>2-</sup>

Method 8051

Powder Pillows

**Scope and application:** For water, wastewater and seawater.<sup>1</sup> USEPA accepted for reporting wastewater analyses. Procedure is equivalent to USEPA method 375.4 for wastewater.<sup>2</sup> Adapted from Standard Methods for the Examination of Water and Wastewater.**Test preparation****Before starting**

Filter samples that are turbid with filter paper and a funnel.

The reagents that are used in this test contain barium chloride. Collect the reacted samples for safe disposal.

Clean the sample cells and caps with a non-abrasive detergent or a solvent (e.g., isopropyl rubbing alcohol) after each test to prevent a build-up of a white film on the sample cells. Rinse with clean water. Dry with a soft cloth.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results.

For the best results, use the Standard solution method and, if necessary, the Standard Calibration Adjust option to adjust the instrument calibration for each reagent lot. Refer to the Standard solution method in Accuracy check on page 3.

Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.

Make sure that the sample cells are clean and there are no scratches where the light passes through them.

Rinse the sample cell and cap with the sample three times before the sample cell is filled.

Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.

Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.

Install the instrument cap over the cell holder before ZERO or READ is pushed.

After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

**Items to collect**

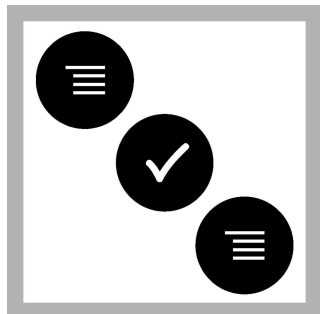
Description	Quantity
SulfaVer® 4 Reagent Powder Pillows, 10-mL	2
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 4 for order information.

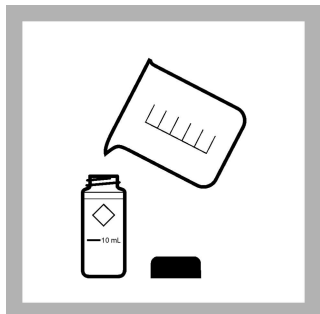
## Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 28 days.
- Let the sample temperature increase to room temperature before analysis.

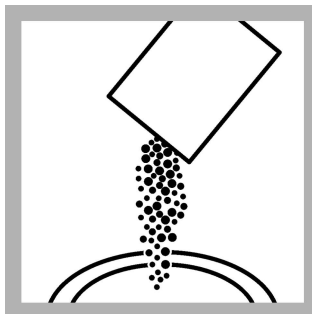
## Powder pillow procedure



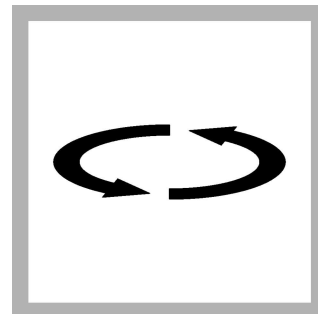
1. Set the instrument to channel 1. Refer to the instrument documentation.



2. **Prepare the sample:** Fill a sample cell to the 10-mL mark with sample.



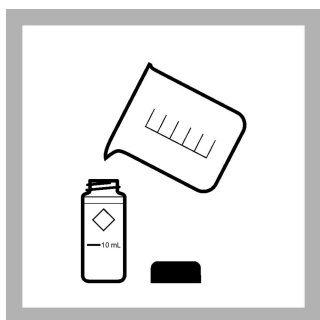
3. Add the contents of one SulfaVer 4 Powder Pillow to the sample cell.



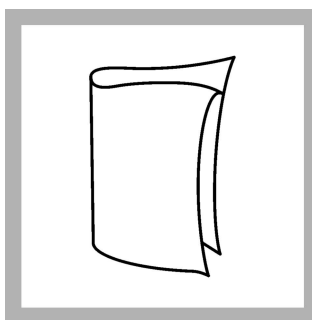
4. Swirl to mix. White turbidity will form if sulfate is in the sample.



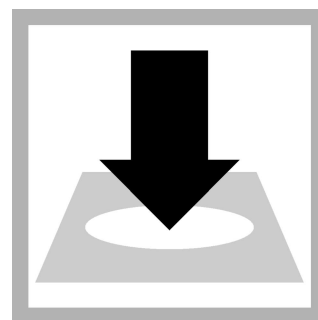
5. Set and start a timer for 5 minutes. A 5-minute reaction time starts. Do not move the sample cell during the reaction time.



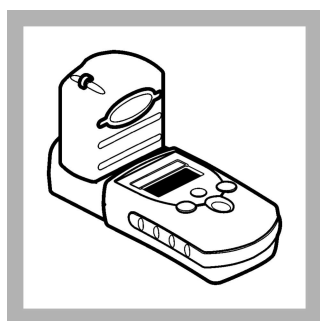
6. **Prepare the blank:** Fill a sample cell to the 10-mL mark with sample. Close the sample cell.



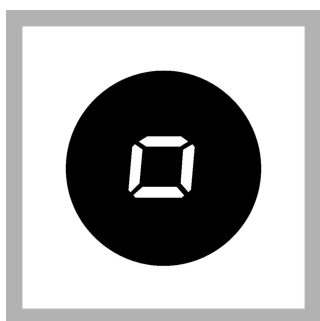
7. When the timer expires, clean the blank sample cell.



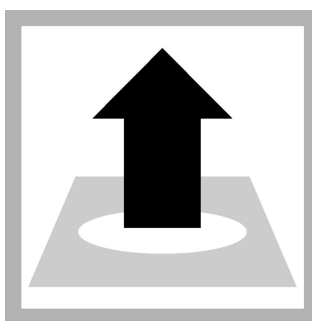
8. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



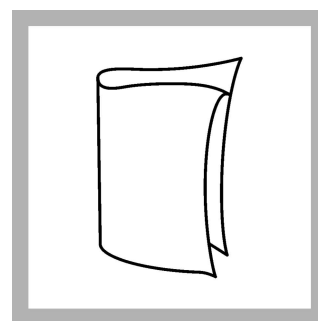
9. Install the instrument cap over the cell holder.



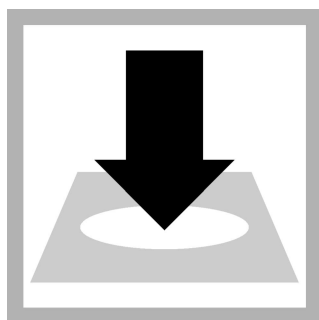
10. Push **ZERO**. The display shows "0".



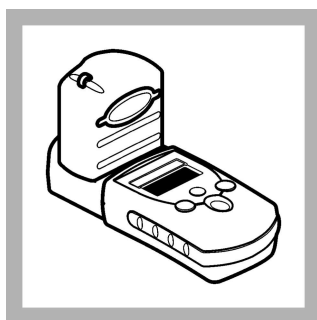
11. Remove the sample cell from the cell holder.



12. Clean the prepared sample cell.



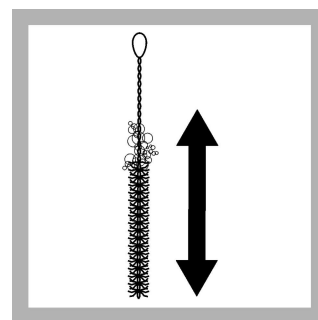
**13.** Within 5 minutes after the timer expires, insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



**14.** Install the instrument cap over the cell holder.



**15.** Push **READ**. Results show in mg/L sulfate ( $\text{SO}_4^{2-}$ ).



**16.** Clean the sample cells with soap and a brush.

## Interferences

Interfering substance	Interference level
Barium	Interferes at all levels. The higher the relative barium concentration when compared to the sulfate concentration, the higher the error. Samples with high barium concentrations will generally give a result that is 20% lower than the actual sulfate concentration.
Calcium	More than 20,000 mg/L as $\text{CaCO}_3$
Chloride	More than 40,000 mg/L as $\text{Cl}^-$
Magnesium	More than 10,000 mg/L as $\text{CaCO}_3$
Silica	More than 500 mg/L $\text{SiO}_2$

## Pollution prevention and waste management

Reacted samples contain barium and must be disposed of as a hazardous waste. Dispose of reacted solutions according to local, state and federal regulations.

## Accuracy check

### Standard additions method

Use the standard additions method to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Sulfate Ampule Standard Solution, 2500 mg/L sulfate
  - Ampule breaker
  - Pipet, TenSette®, 0.1–1.0 mL and tips
  - Mixing cylinders (3), 25-mL
1. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 25-mL portions of fresh sample. Mix well.
  2. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
  3. Compare the expected result to the actual result. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

## Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Sulfate Standard Solution, 50-mg/L
1. Use the test procedure to measure the concentration of the standard solution.
  2. Compare the expected result to the actual result.

**Note:** The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are small variations in the reagents or instruments.

## Method performance

The method performance data that follows was derived from laboratory tests that were measured on a Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
$50 \pm 3 \text{ mg/L SO}_4^{2-}$

## Summary of method

Sulfate ions in the sample react with barium in the SulfaVer 4 Reagent and form a precipitate of barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration.

## Consumables and replacement items

### Required reagents

Description	Quantity/test	Unit	Item no.
SulfaVer <sup>®</sup> 4 Reagent Powder Pillow <sup>1</sup> , 10-mL	1	100/pkg	2106769

### Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

### Recommended standards

Description	Unit	Item no.
Sulfate Standard Solution, 50-mg/L SO <sub>4</sub>	500 mL	257849
Sulfate Standard Solution, 2500-mg/L SO <sub>4</sub>	500 mL	1425249
Sulfate Standard Solution, 2500-mg/L, 10-mL ampules as SO <sub>4</sub>	16/pkg	1425210
Drinking Water Standard, Mixed Parameter, Inorganic for F <sup>-</sup> , NO <sub>3</sub> -N, PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup>	500 mL	2833049
Wastewater Influent Standard Solution, Mixed Parameter, for NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> , COD, SO <sub>4</sub> , TOC	500 mL	2833149
Wastewater Effluent Standard Solution, Mixed Parameter, for NH <sub>3</sub> -N, NO <sub>3</sub> -N, PO <sub>4</sub> <sup>3-</sup> , COD, SO <sub>4</sub> <sup>2-</sup> , TOC	500 mL	2833249

<sup>1</sup> SulfaVer is a registered trademark of Hach Company.

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**Optional reagents and apparatus**

Description	Unit	Item no.
Mixing cylinder, graduated, 25 mL	each	189640
Mixing cylinder, graduated, 50 mL	each	189641
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, TenSette®, 1.0–10.0 mL	each	1970010
Pipet tips for TenSette® Pipet, 1.0–10.0 mL	50/pkg	2199796
Liqui-Nox Phosphate-free detergent	946 mL	2088153
Test tube brush	each	69000



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WORLD HEADQUARTERS

Telephone: (970) 669-3050

FAX: (970) 669-2932