Sulfate

DOC316.53.01135

USEPA[1] SulfaVer 4 Method[2]

Method 8051

2 to 70 mg/L SO_4^{2-}

Powder Pillows

Scope and application: For water, wastewater and seawater.

[1] USEPA accepted for reporting wastewater analyses. Procedure is equivalent to USEPA method 375.4 for wastewater.

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater*, SM4500-SO_4^{2-}-E.

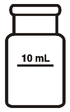
Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

For turbidimetric methods, install the instrument cap or cover on all instruments before ZERO or READ is pushed.
Use the Standard Adjust option with each new lot of reagent for the best results. Refer to the Standard solution method in Accuracy check on page 3.
For best results, calibrate the instrument with each new lot of reagent. Refer to Calibration on page 4.
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 automatically with the reagent blank adjust option.
Filter samples that are turbid with filter paper and a funnel.
Do not use the Pour-Thru Cell or sipper module (for applicable instruments) with this test.
The reagents that are used in this test contain barium chloride. Collect the reacted samples for safe disposal.

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

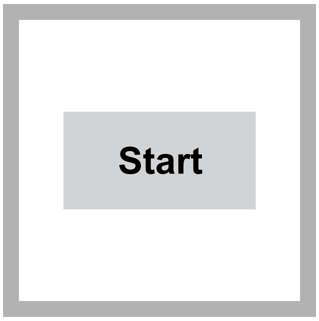
Description	Quantity
SulfaVer® 4 Reagent Powder Pillows, 10-mL	1
Sample Cells (Refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 5 for order information.

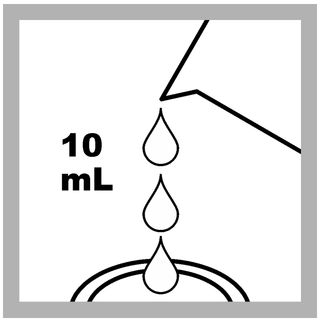
Sample collection and storage

- 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.

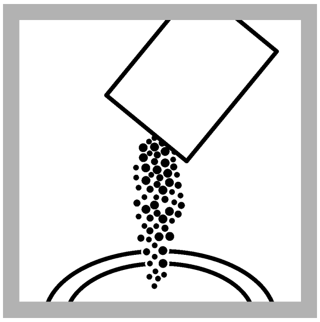
Powder pillow procedure



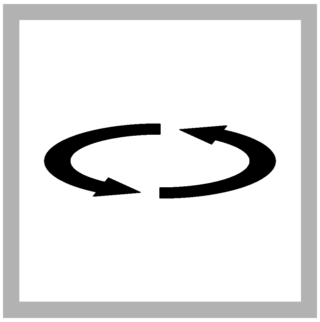
1. Start program **680 Sulfate**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



2. Prepare the sample: Fill a sample cell with 10 mL of sample.



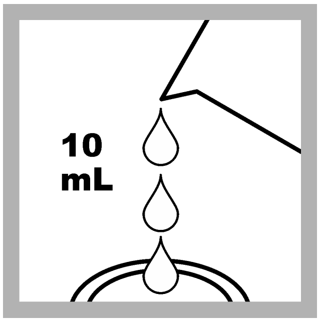
3. Add the contents of one SulfaVer 4 powder pillow to the sample cell.



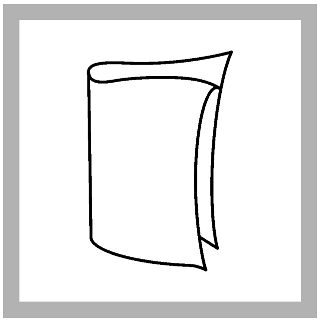
4. Swirl the sample cell to mix. Undissolved powder will not affect accuracy. White turbidity will form if sulfate is present.

5. Start the instrument timer. A 5-minute reaction time starts.

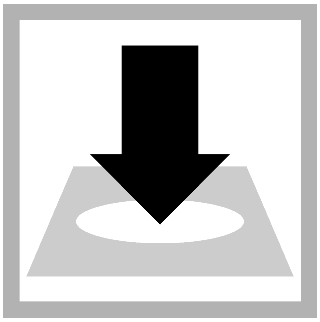
Do not disturb the cell during this time.



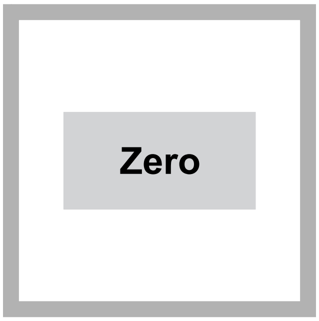
6. Prepare the blank: Fill a second sample cell with 10 mL of sample.



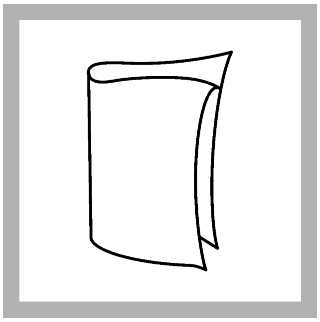
7. When the timer expires, clean the blank sample cell.



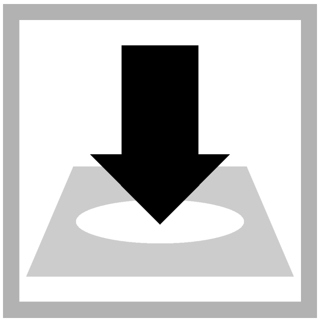
8. Insert the blank into the cell holder.



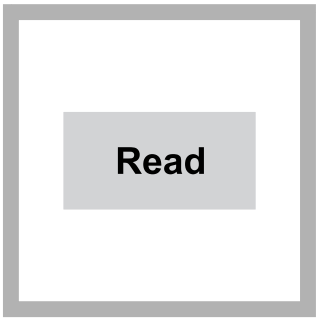
9. Push **ZERO**. The display shows 0 mg/L SO_4^{2-}.



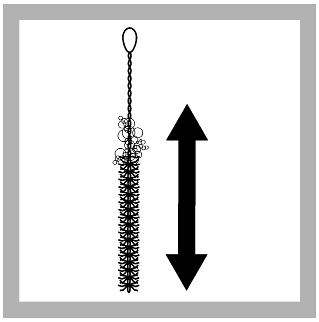
10. Clean the prepared sample cell.



11. Within 5 minutes after the timer expires, insert the prepared sample into the cell holder.



12. Push **READ**. Results show in mg/L SO_4^{2-}.



13. 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 $CaCO_3$
Chloride	More than 40,000 mg/L as Cl^-
Magnesium	More than 10,000 mg/L as $CaCO_3$
Silica	More than 500 mg/L SiO_2

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) 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 (3x), 25-mL

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.

4. Open the standard solution.
5. 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.
6. 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.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

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

Items to collect:

- Sulfate standard solution, 1000-mg/L
- 100-mL volumetric flask, Class A
- 7-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 70-mg/L sulfate standard solution as follows:
   a. Use a pipet to add 7.0 mL of 1000-mg/L sulfate standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. 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.*

Calibration

A calibration is recommended for the SulfaVer 4 method for the best accuracy. Complete the steps that follow to enter a new calibration curve in the instrument. Make a new calibration curve for each new lot of reagent.

Items to collect:

- Sulfate standard solution, 1000 mg/L
- 100-mL volumetric flasks (7), Class A
- 1–10 mL TenSette pipet and tips
- Deionized water

1. Prepare seven calibration standard solutions (10, 20, 30, 40, 50, 60 and 70 mg/L SO_4^{2-}) as follows:
   a. Use a pipet to add 1, 2, 3, 4, 5, 6 and 7 mL of the 1000-mg/L sulfate standard solution into seven different 100-mL volumetric flasks.
   b. Dilute each flask to the mark with deionized water. Mix well.
2. Use the test procedure to measure the concentration of each standard solution.
3. Refer to the user manual for the instrument to enter the calibration into the instrument as a user program.

Method performance

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

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
680	40 mg/L SO_4^{2-}	30–50 mg/L SO_4^{2-}	0.4 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. The measurement wavelength is 450 nm for spectrophotometers or 520 nm for colorimeters.

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.

Consumables and replacement items

Required reagents

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

Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-20-25-mL, with cap	2	6/pkg	2401906
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402

Recommended standards

Description	Unit	Item no.
Sulfate Standard Solution, 1000-mg/L as SO_4^{2-}	500 mL	2175749
Sulfate Standard Solution, 2500-mg/L, 10-mL ampules as SO_4^{2-}	16/pkg	1425210
Drinking Water Standard, Mixed Parameter, Inorganic for F^-, NO_3–N, PO_4^{3-}, SO_4^{2-}	500 mL	2833049

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
Flask, volumetric, Class A, 100 mL, glass	each	1457442

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[1] SulfaVer is a registered trademark of Hach Company.

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