

USEPA¹ SulfaVer 4 Method²

Method 8051

(2 to 70 mg/L)

 Powder Pillows or AccuVac[®] Ampuls

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*.



Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Adapter	Sample cell	Adapter
DR 5000	2495402	A23618	2427606	A23618
DR 2800	2495402	—	2122800	LZV584 (C)
DR/2500	2427606	—	2427606	—
DR/2400	2427606	—	2427606	—

Before starting the test:

Adjust the standard curve for each new lot of reagent (*Standard solution method*).

For best results, calibrate the instrument with each new lot of reagent (see *Calibration*).

For best results, measure 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 enter the value as a reagent blank adjust for automatic subtraction.

Filter highly colored or turbid samples using filter paper and a funnel. Use this sample in step 2 and 5.

The Pour-Thru Cell cannot be used with this procedure.

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. Refer to a current MSDS for safe handling and disposal instructions.

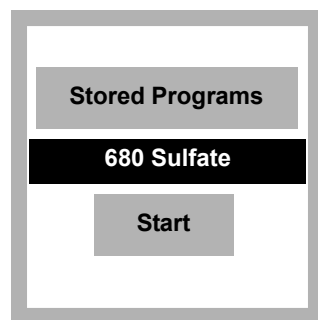
Use a blank AccuVac[®] Ampule in place of the sample cell in Step 5 if necessary.

Collect the following items:

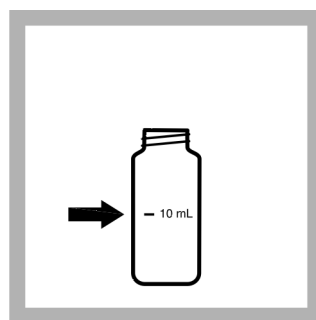
Description	Quantity
Powder Pillow Test:	
SulfaVer® 4 Reagent Powder Pillows	1
Sample Cells (see Instrument-specific information)	2
AccuVac Test:	
SulfaVer® 4 Reagent AccuVac® Ampuls	1
Beaker, 50-mL	1
Sample Cells (see Instrument-specific information)	1
Stopper	1

See [Consumables and replacement items](#) for reorder information.

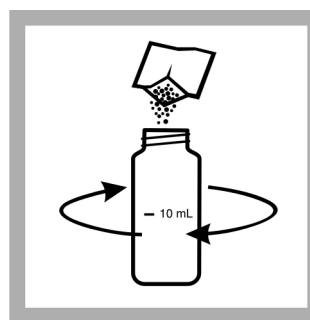
SulfaVer 4 powder pillow procedure



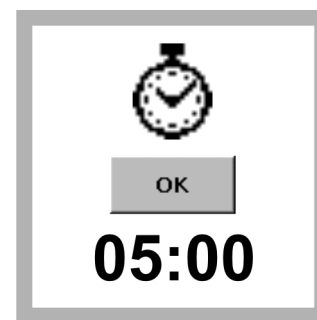
1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)). Refer to the user manual for orientation.



2. Prepared sample: Fill a sample cell with 10 mL of sample.

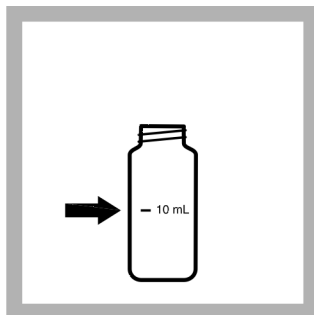


3. Add the contents of one SulfaVer 4 Reagent Powder Pillow to the sample cell. Swirl vigorously to dissolve the powder.
White turbidity will form if sulfate is present.

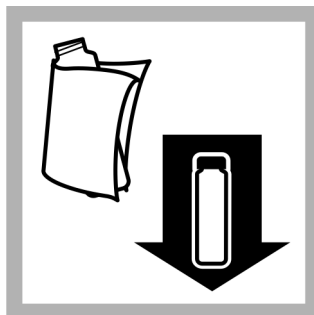


4. Start the instrument timer.
A five-minute reaction time will begin. Do not disturb the cell during this time.
Note: Accuracy is not affected by undissolved powder.

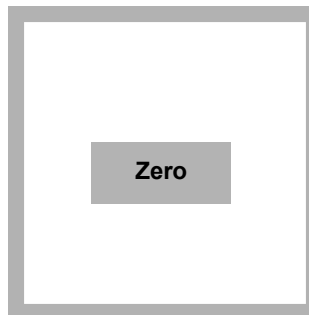
SulfaVer 4 powder pillow procedure (continued)



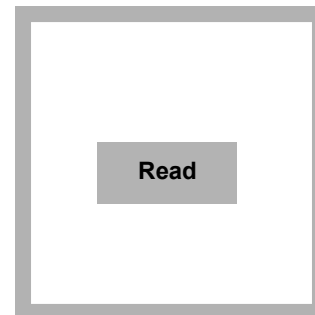
5. Blank preparation:
Fill a second sample cell with 10 mL of sample.



6. When the timer expires, wipe the blank and insert it in the cell holder (fill lines face right.)



7. ZERO the instrument.
The display will show:
0 mg/L SO_4^{2-}

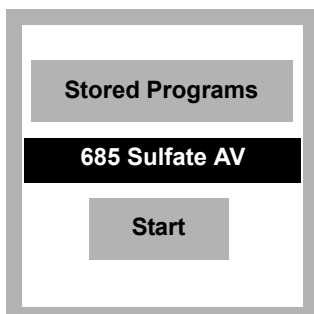


8. Within five minutes after the timer expires, wipe the cell and insert the prepared sample in the cell holder.

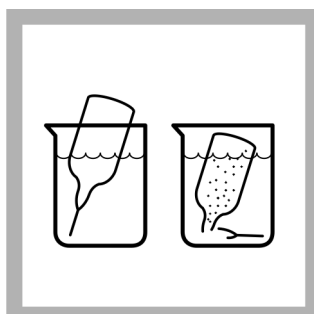
READ the results in mg/L SO_4^{2-} .

Clean sample cells with soap and a brush.

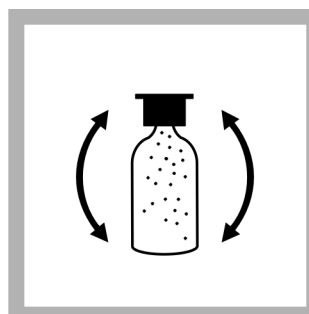
SulfaVer 4 AccuVac® Ampuls procedure



1. Select the test.
Insert an adapter if required (see [Instrument-specific information](#)). Refer to the user manual for orientation.



2. Prepared sample:
Collect at least 40 mL of sample in a 50-mL beaker. Fill a SulfaVer 4 Reagent AccuVac® Ampul with sample from the beaker. Keep the tip immersed while the Ampul fills completely.



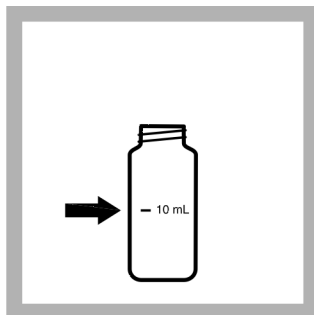
3. Cap or stopper the Ampul and quickly invert several times to mix. White turbidity will form if sulfate is present.



4. Start the instrument timer. A five-minute reaction time will begin. Do not disturb the cell during this time.

Note: Accuracy is not affected by undissolved powder.

SulfaVer 4 AccuVac® Ampuls procedure (continued)



5. Blank Preparation:
Fill a clean sample cell with 10 mL of sample.



6. When the timer expires, wipe the blank and insert it in the cell holder.

ZERO the instrument.
The display will show:
0 mg/L SO_4^{2-}



7. Within five minutes after the timer expires, wipe the Ampul and insert it in the cell holder.

READ the results in mg/L SO_4^{2-} .

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
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, preservation and storage

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)

Required for accuracy check:

- Sulfate Ampule Standard Solution, 2500 mg/L sulfate
- Ampule breaker
- TenSette Pipet and pipet tips
- Mixing cylinder, 25 mL and 50 mL
- Beaker, 50 mL

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select standard additions from the instrument menu:
OPTIONS>(MORE)>STANDARD ADDITIONS.

3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution ampule.
5. Fill three mixing cylinders 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 mixing cylinder and mix thoroughly. Transfer 10 mL of each sample spike to a clean sample cell.

Note: For AccuVac® Ampuls, fill three mixing cylinders 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.

6. Follow the [SulfaVer 4 powder pillow procedure](#) for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.

Note: For AccuVac Ampuls, follow the [SulfaVer 4 AccuVac® Ampuls procedure](#) for each of the spiked samples, starting with the 0.2 mL sample spike. Measure each of the spiked samples in the instrument.

7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Required for accuracy check:

- Sulfate standard solution, 1000 mg/L
 - 100 mL Class A volumetric flask
 - Tensette pipet, 1–10 mL and pipet tips
1. Prepare a 70 mg/L sulfate standard solution as follows: use a pipet to add 7.0 mL of sulfate standard solution, 1000 mg/L as SO_4^{2-} , to a 100-mL volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
 2. Follow the [SulfaVer 4 powder pillow procedure](#) or [SulfaVer 4 AccuVac® Ampuls procedure](#) and use the 70-mg/L SO_4^{2-} standard solution in place of the sample.
 3. To adjust the calibration curve using the reading obtained with the standard solution, set standard adjust to on (**OPTIONS>(MORE)>STANDARD ADJUST**) and accept the concentration.

Calibration

A calibration is recommended for the SulfaVer 4 method for the best accuracy. Complete the following steps to enter a new calibration curve in the instrument. Perform this procedure for each new set of reagent.

Required items:

- Sulfate standard solution, 1000 mg/L
 - Seven 100 mL Class A volumetric flasks
 - 1–10 mL TenSette pipet
1. Prepare seven calibration standards (10, 20, 30, 40, 50, 60 and 70 mg/L SO_4^{2-}) as follows. Use the Tensette pipet to add 1, 2, 3, 4, 5, 6 and 7 mL of the 1000-mg/L sulfate standard solution to seven different 100-mL Class A volumetric flasks.
 2. Dilute each flask to the mark with deionized water. Mix thoroughly.
 3. Use each standard solution in place of the sample and follow the [SulfaVer 4 powder pillow procedure](#) or [SulfaVer 4 AccuVac® Ampuls procedure](#).

4. Refer to the user manual (user programs section) to enter the calibration in the instrument as a user program.

Method performance

Program	Instrument	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
680	DR 5000	40 mg/L SO ₄ ²⁻	30–50 mg/L SO ₄ ²⁻	0.4 mg/L SO ₄ ²⁻
	DR 2800	40 mg/L SO ₄ ²⁻	30–50 mg/L SO ₄ ²⁻	0.4 mg/L SO ₄ ²⁻
	DR 2700	40 mg/L SO ₄ ²⁻	30–50 mg/L SO ₄ ²⁻	0.4 mg/L SO ₄ ²⁻
	DR/2500	30 mg/L SO ₄ ²⁻	27–33 mg/L SO ₄ ²⁻	1 mg/L SO ₄ ²⁻
	DR/2400	30 mg/L SO ₄ ²⁻	27–33 mg/L SO ₄ ²⁻	1 mg/L SO ₄ ²⁻
685	DR 5000	40 mg/L SO ₄ ²⁻	32–48 mg/L SO ₄ ²⁻	0.7 mg/L SO ₄ ²⁻
	DR 2800	40 mg/L SO ₄ ²⁻	32–48 mg/L SO ₄ ²⁻	0.7 mg/L SO ₄ ²⁻
	DR 2700	40 mg/L SO ₄ ²⁻	32–48 mg/L SO ₄ ²⁻	0.7 mg/L SO ₄ ²⁻
	DR/2500	30 mg/L SO ₄ ²⁻	18–43 mg/L SO ₄ ²⁻	2 mg/L SO ₄ ²⁻
	DR/2400	30 mg/L SO ₄ ²⁻	18–43 mg/L SO ₄ ²⁻	2 mg/L SO ₄ ²⁻

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. Test results are measured at 450 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
SulfaVer® 4 Reagent Powder Pillows	1	100/pkg	2106769
OR			
SulfaVer® 4 Sulfate Reagent AccuVac® Ampuls	1	25/pkg	2509025

Required apparatus (AccuVac)

Description	Quantity/Test	Unit	Catalog number
Beaker, 50-mL	1	each	50041H
Stopper	1	6/pkg	173106

Recommended standards

Description	Unit	Catalog number
Sulfate Standard Solution, 1000-mg/L	500 mL	2175749
Sulfate Standard Solution, 2500-mg/L, 10-mL Ampules	16/pkg	1425210
Mixed Parameter Standard for sulfate, fluoride, nitrate and phosphate	500 mL	2833049

Optional reagents and apparatus

Description	Unit	Catalog number
Cylinder, mixing, 25-mL	each	189640
Cylinder, mixing, 50-mL	each	189641
Blank AccuVac Ampules	25/pkg	2677825
Ampule Breaker	each	2196800
AccuVac Snapper	each	2405200
Tensette Pipet 0.1–1.0 mL	each	1970001
Tips for Tensette Pipet 1–10	50/pkg	2185696
Tensette Pipet 1–10	each	1970010
Tips for tensette Pipet 1–10	50/pkg	2199796
Flask, volumetric, Class A, 100-mL	each	1457442
Pipet Filler, Safety Bulb	each	1465100



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