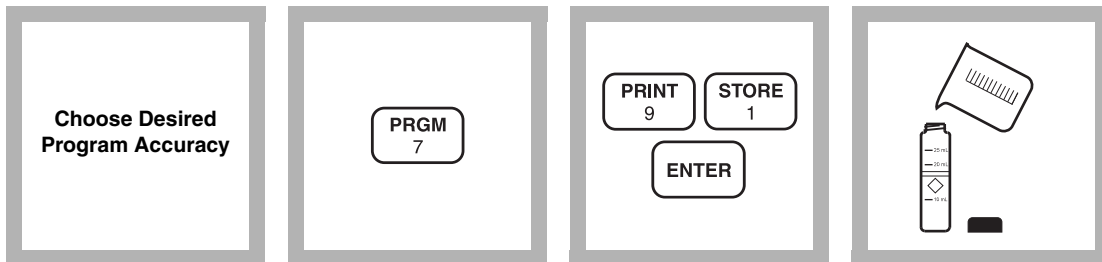


SULFATE (0 to 70 mg/L)

For water, wastewater, and seawater

SulfaVer 4 Method* (Powder Pillows or AccuVac Ampuls); USEPA accepted for reporting wastewater analysis**

Using Powder Pillows

1. A User-Entered Calibration is necessary to obtain the most accurate results. See the *User Calibration* section at the back of this procedure. Program 91 can be used for process control or applications where a high degree of accuracy is not needed.

Note: The nature of turbidimetric tests and reagent lot variation requires user calibration for best results.

2. Enter the stored program number for sulfate (SO_4^-).

Press: **PRGM**

The display will show:
PRGM ?

3. Press: **91 ENTER** or the program number selected for a user-entered calibration.

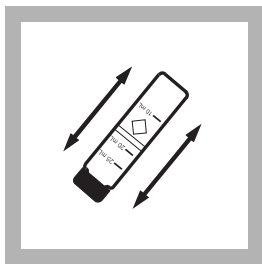
The display will show **mg/L, SO4** and the **ZERO** icon.

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

Note: Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*.

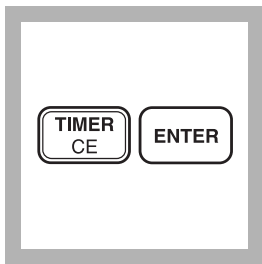
** Procedure is equivalent to USEPA method 375.4 for wastewater.



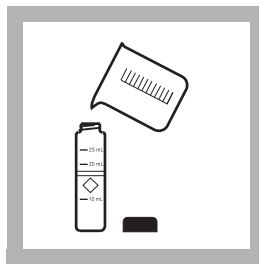
5. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and invert several times to mix.

Note: A white turbidity will develop if sulfate is present in the sample.

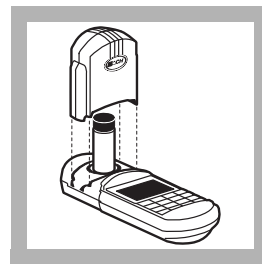
Note: Accuracy is not affected by undissolved powder.



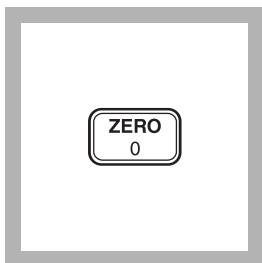
6. Press:
TIMER ENTER
A 5-minute reaction period will begin. Allow the cell to stand undisturbed.



7. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).

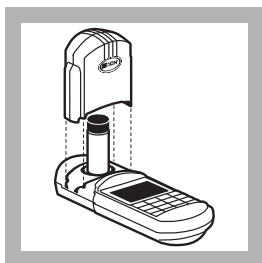


8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

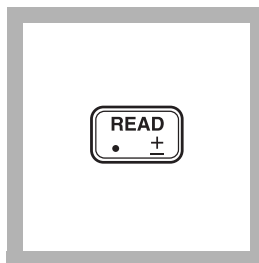


9. Press: **ZERO**
The cursor will move to the right, then the display will show:

0 mg/L SO₄



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

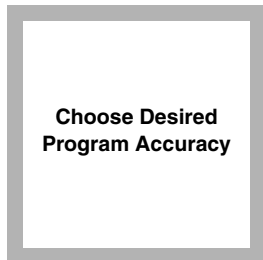


11. Press: **READ**
The cursor will move to the right, then the result in mg/L sulfate will be displayed.

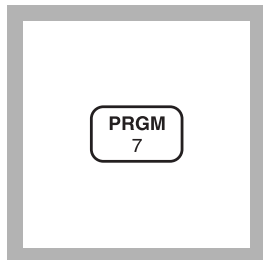
Note: If Program 91 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.

Note: Clean the sample cells with soap and a brush.

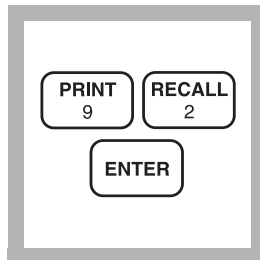
Using AccuVac Ampuls



1. A User-Entered Calibration is necessary to obtain the most accurate results. See User Calibration Section at the back of this procedure. Program 92 can be used for process control or applications where a high degree of accuracy is not needed.



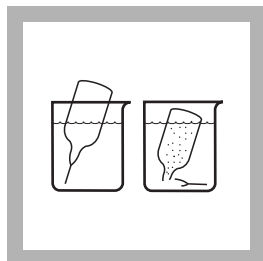
2. Enter the stored program number for sulfate (SO_4^-)-AccuVac Ampuls. Press: **PRGM**
The display will show:
PRGM ?



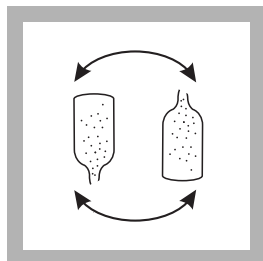
3. Press: **92 ENTER**
The display will show **mg/L, SO4** and the **ZERO** icon.



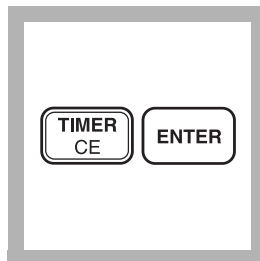
4. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.
Note: Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.



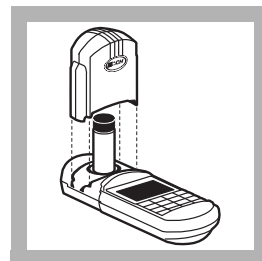
5. Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample.
Note: Keep tip immersed until the ampul fills completely.



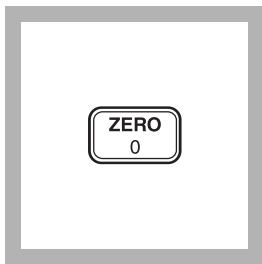
6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.
Note: A white turbidity will develop if sulfate is present.
Note: Accuracy is not affected by undissolved powder.



7. Press:
TIMER ENTER
A 5-minute reaction period will begin.
Note: Allow the ampul to stand undisturbed.



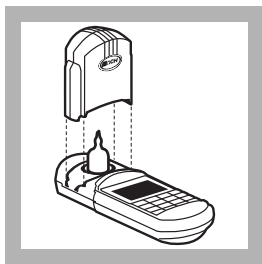
8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



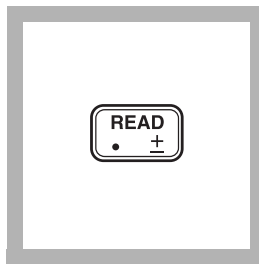
9. Press: ZERO

The cursor will move to the right, then the display will show:

0 mg/L SO₄



10. Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: READ

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

Note: If Program 92 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.

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 SO_4^{2-} .
- 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 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 Additions Method- AccuVac Ampuls

- a) Snap the neck off a Sulfate Standard PourRite Ampule, 2500 mg/L SO_4^{2-} .
- 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 ± 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 ± 3 mg/L sulfate.

Estimated Detection Limit (EDL)

The EDL for program 91 is 4.9 mg/L SO_4 and the EDL for program 92 is 3 mg/L SO_4 . For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following interfere at levels above those concentrations listed:

Calcium: 20,000 mg/L as CaCO_3	Magnesium: 10,000 mg/L as CaCO_3
Chloride: 40,000 mg/L as Cl^-	Silica: 500 mg/L as CaCO_3

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.

SULFATE, continued

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required		Units	Cat. No.
	Per Test			
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow.....	100/pkg	21067-69	
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06	

REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls	1 ampul.....	25/pkg	25090-25
Beaker, 50-mL.....	1	each	500-41H

OPTIONAL REAGENTS

Standard, Drinking Water Inorganics, F ⁻ , NO ₃ ^{-N} , PO ₄ ⁻³ , SO ₄ ⁻²	500 mL	28330-49
Standard, Wastewater Effluent Inorganics, NH ₃ ^{-N} , NO ₃ ^{-N} , PO ₄ ⁻³ , COD, SO ₄ ⁻² , TOC.....	500 mL	28332-49
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, 10 mL	16/pkg	14252-10
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.