



## ✓ Method 8051

## SulfaVer 4 Method\*

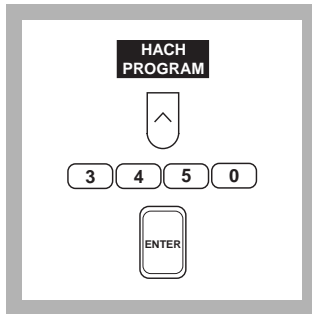
### Powder Pillows or AccuVac® Ampuls

(0 to 70.0 mg/L)

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

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

## Using Powder Pillows



**1.** Press the soft key under **HACH PROGRAM**.

Select the stored program for sulfate ( $\text{SO}_4^{2-}$ ) by pressing **3450** with the numeric keys.

Press: **ENTER**

**Note:** If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

**Note:** The Flow Cell and Sipper Cell Modules cannot be used with this procedure.

**Note:** For best results, perform a new calibration for each lot of reagent. See *Calibration Standard Preparation* following these steps.

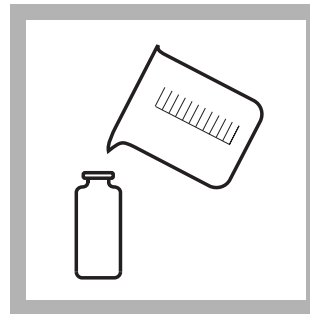


**2.** The display will show: **HACH PROGRAM: 3450 Sulfate**

The wavelength ( $\lambda$ ), **450 nm**, is automatically selected.

**Note:** For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 10, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under **OPTIONS, (MORE)**, and then **BLANK:OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.

**Note:** You must adjust the standard curve for each new lot of reagent. See *Standard Curve Adjustment* following these steps.



**3.** Fill a clean sample cell with 25 mL of sample.

**Note:** Filter highly turbid or colored samples. Use filtered sample in this step and in Step 6. Use labware listed under **OPTIONAL EQUIPMENT AND SUPPLIES**.

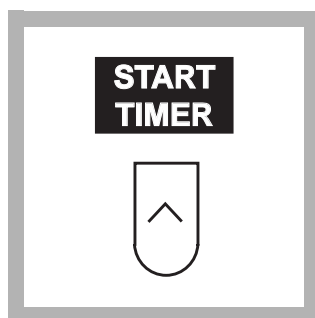
**Note:** For proof of accuracy, use a 70.0 mg/L sulfate standard solution (see *Standard Curve Adjustment*) in place of the sample.



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

**Note:** A white turbidity will develop if sulfate is present.

**Note:** Accuracy is not affected by undissolved powder which has settled.



**5.** Press the soft key under **START TIMER**.

A 5-minute reaction period will begin.

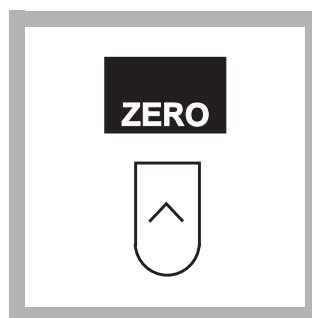
**Note:** Allow cell to stand undisturbed.



**6.** Fill a second sample cell with 25 mL of sample (the blank).



**7.** When the timer beeps, place the blank in the cell holder. Close the light shield.



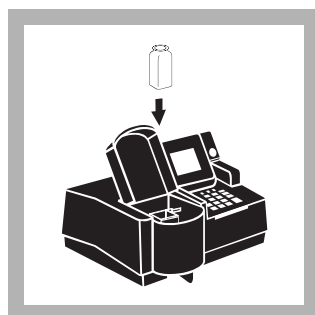
**8.** Press the soft key under **ZERO**.

The display will show:

**0.0 mg/L SO<sub>4</sub><sup>2-</sup>**

**Note:** If you are using a reagent blank correction, the display will show the correction.

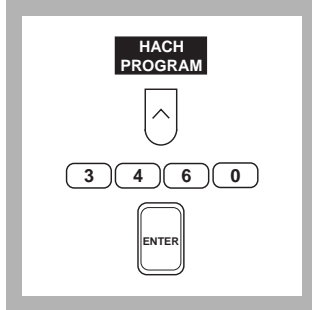
**Note:** For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



**9.** Within five minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield. Results in mg/L sulfate (or chosen units) will be displayed.

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

## Using AccuVac Ampuls



- 1.** Press the soft key under **HACH PROGRAM**.

Select the stored program for sulfate ( $\text{SO}_4^{2-}$ ) by pressing **3460** with the numeric keys.

Press: **ENTER**

**Note:** If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

**Note:** The Flow Cell and Sipper Cell Modules cannot be used with this procedure.

**Note:** For best results, perform a new calibration for each lot of reagent. See *Calibration Standard Preparation* following these steps.



- 2.** The display will show: **HACH PROGRAM: 3460 Sulfate, AV**

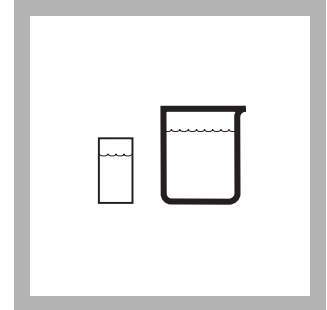
The wavelength ( $\lambda$ ), **450 nm**, is automatically selected.

**Note:** You must determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 10, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under **OPTIONS, (MORE)**, and then **BLANK:OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.

**Note:** You must adjust the standard curve for each new lot of reagent. See *Standard Curve Adjustment* following these steps.



- 3.** Insert the 1-inch Cell Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.



- 4.** Fill a Zeroing Vial 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 in Step 5. Use labware listed under **OPTIONAL EQUIPMENT AND SUPPLIES**.

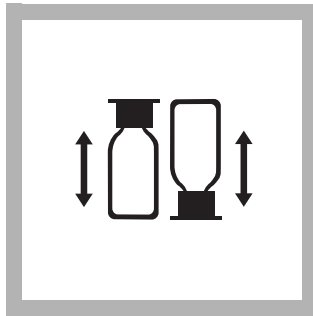
**Note:** For proof of accuracy, use a 70.0 mg/L sulfate standard solution (see *Standard Curve Adjustment*) in place of the sample.

## SULFATE, continued



**5.** Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample (the prepared sample).

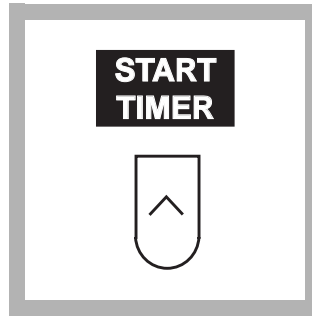
**Note:** Keep tip immersed until the ampul fills completely.



**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 which has settled.



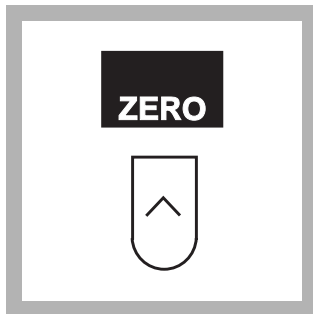
**7.** Press the soft key under **START TIMER**. A 5-minute reaction period will begin.

**Note:** Allow cell to stand undisturbed.

**Note:** Read samples within five minutes after the timer beeps.



**8.** When the timer beeps, place the blank in the cell holder. Close the light shield.



**9.** Press the soft key under **ZERO**.

The display will show:

**0.0 mg/L SO<sub>4</sub><sup>2-</sup>**

**Note:** If you are using a reagent blank correction, the display will show the correction.

**Note:** For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



**10.** Within five minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield. Results in mg/L sulfate (or chosen units) will be displayed.

## Interferences

**Table 1 Interfering Substances and Suggested Treatments**

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

## 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

- a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under **OPTIONS, (MORE)** and then **STD ADD**.
- b. Press **ENTER** to accept the default sample volume (mL), 25.
- c. Press **ENTER** to accept the default standard concentration (mg/L), 1000.
- d. Press the soft key under **ENTRY DONE**.
- e. Snap the neck off a Sulfate 2-mL Ampule Standard, 1000-mg/L sulfate.
- f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 25-mL samples and mix each thoroughly (for AccuVac Ampuls, use 50-mL beakers).
- g. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under **READ** each time. Each addition should reflect approximately 100% recovery.
- h. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.
- i. See Section 1.4.1 *Standard Additions* for more information.

## Standard Curve Adjustment

Using Class A glassware, prepare a 70-mg/L sulfate standard solution by pipetting 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.

To adjust the calibration curve using the reading obtained with the 70-mg/L standard solution, press the soft keys under **OPTIONS, MORE** then **STD: OFF**. Press **ENTER** to accept the displayed concentration, the value of which depends on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section 1.5.5 *Adjusting the Standard Curve* 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  $\text{SO}_4^{2-}$  as follows:

- a. 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 using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the SulfaVer method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

## 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.

## Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to *Section 1*.

## Pollution Prevention and Waste Management

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 1* for more information on proper disposal of these materials.

**REQUIRED REAGENTS AND STANDARDS**

Description	Quantity Required per test	Unit	Cat. No.
SulfaVer 4 Reagent Powder Pillows.....	1	100/pkg.....	12065-99
<i>or</i>			
SulfaVer 4 Sulfate Reagent AccuVac Ampuls .....	1	25/pkg.....	25090-25

**REQUIRED EQUIPMENT AND SUPPLIES**

DR/4000 1-inch Cell Adapter .....	1	each.....	48190-00
DR/4000 AccuVac Ampul Adapter.....	1	each.....	48187-00
Sample Cell, 10-mL with cap (zeroing vial).....	1	each.....	21228-00

**OPTIONAL REAGENTS AND STANDARDS**

Sulfate Standard Solution, 50-mg/L.....	500 mL.....	2578-49
Sulfate Standard Solution, 1000-mg/L.....	500 mL.....	21757-49
Sulfate Standard Solution, 2-mL PourRite Ampul, 1000-mg/L.....	20/pkg.....	21757-20
Water, deionized .....	4 liters.....	272-56

**OPTIONAL EQUIPMENT AND SUPPLIES**

AccuVac Snapper Kit .....	each.....	24052-00
Beaker, 50-mL.....	each.....	500-41
Brush, test tube .....	each.....	690-00
DR/4000 Carousel Module.....	each.....	48070-02
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, volumetric, Class A, 1.00-mL.....	each.....	14515-35
Pipet, volumetric, Class A, 2.00-mL.....	each.....	14515-36
Pipet, volumetric, Class A, 3.00-mL.....	each.....	14515-03
Pipet, volumetric, Class A, 4.00-mL.....	each.....	14515-04
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 6.00-mL.....	each.....	14515-06
Pipet, volumetric, Class A, 7.00-mL.....	each.....	14515-07
Pipet Filler, safety bulb.....	each.....	14651-00
Pipet, TenSette, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....	50/pkg.....	21856-96
PourRite Ampule Breaker Kit .....	each.....	24846-00



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