Sulfate

DOC316.53.01312

SulfaVer 4 Method[1]

Method 10248

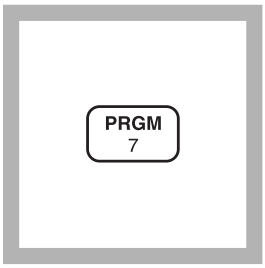
(0–70, 0–700 and 0–7000 mg/L)

Powder Pillows

Scope and Application: For oil and gas field waters.

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

Test procedure

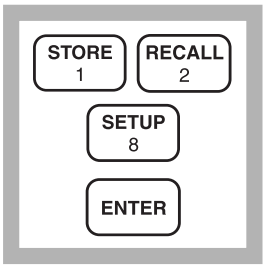


1. Push **PRGM.**

The display shows

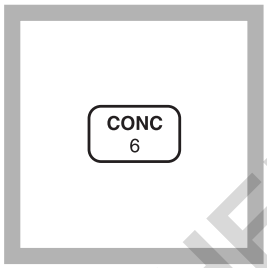
PRGM ?

Initial setup: go to "Instrument Setup" on page 3 to add the program to the instrument.



2. Push **128 ENTER**.

The display shows **mg/L, SO4 L, SO4 M or SO4 H** and the **ZERO** icon**.**



3. Push **CONC** to select the test range:

SO4 L: 0–70 mg/L

SO4 M: 0–700 mg/L

SO4 H: 0–7000 mg/L



4. Add the specified sample volume to a clean sample cell:

SO4 L: 10 mL

SO4 M: 1.0 mL

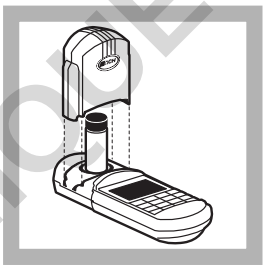
SO4 H: 0.1 mL

Note:*Use a TenSette or glass pipet to measure 0.1 mL or 1.0 mL.*

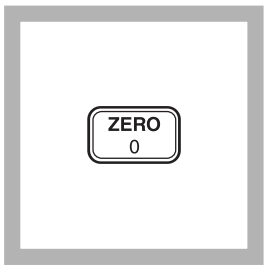


5. If the sample volume is less than 10 mL, add deionized water to the 10-mL line. Tighten the cap on the sample cell and invert to mix.

Note:*A 10-mL graduated mixing cylinder can be used in steps 4 and 5.*



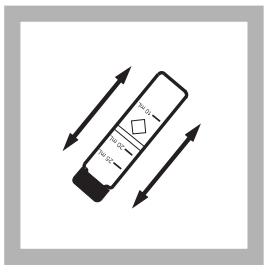
6. Put the sample cell in the cell holder. Put the instrument cap over the sample cell.



7. Push **ZERO.**

The cursor moves to the right, then the display shows:

0 mg/L SO4 and L, M or H

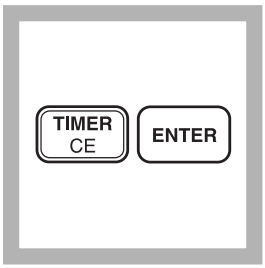


8. Remove the cap and add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell. Tighten the cap and invert to mix.

Note:*The sample will become cloudy if sulfate is in the sample.*

Note:*Accuracy is not affected by undissolved powder.*

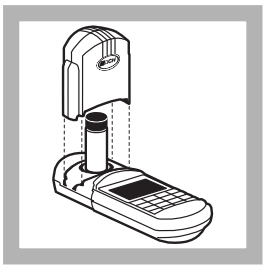
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9. Push
TIMER ENTER

A 5-minute reaction period starts.

Do not move the sample cell during the reaction period.



10. Within five minutes after the timer beeps, put the prepared sample in the instrument. Put the instrument cap over the sample cell.



11. Push **READ.**

The cursor moves to the right, then the result in mg/L sulfate is shown.

Notice! Do not push the **CONC** key at the end of the test to change the range. The result is applicable only to the test range that was selected in step 3.

Clean the sample cells with soap and a brush.

Note:*For best results use the Standard Adjust option. Refer to "Standard Adjust" on page 3.*

Sampling and Storage

Collect the sample in a clean plastic or glass bottle. Samples can be used for up to 28 days if they are kept at or less than 4 °C (39 °F). Let the sample temperature increase to room temperature before analysis.

Interferences

Known interferences are shown in *Table 1*. The interference levels are applicable to an undiluted 10-mL sample. The interference levels increase proportionally as the sample is diluted.

Table 1 Interferences

Substance	Interference Level
Barium	Interferes at all levels. The greater the barium concentration compared to the sulfate concentration, the greater the error. Samples with high barium concentrations will generally give a result that is 20% lower than the actual sulfate concentration.
Calcium	20,000 mg/L as $CaCO_3$
Chloride	40,000 mg/L as Cl^-
Magnesium	10,000 mg/L as $CaCO_3$
Silica	500 mg/L as $CaCO_3$
Turbidity	Filter samples that have a high level of turbidity

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.

1. Fill three sample cells with sample as specified in steps 4 and 5 of the test procedure.
2. Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of a 1000 mg/L Sulfate Standard Solution to the sample cells. Mix fully.
3. Complete the test procedure for each sample.

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4. Review the results. The sulfate concentration should increase by 10 mg/L for SO4 L, 100 mg/L for SO4 M or 1000 mg/L for SO4 H for each 0.1 mL of standard that is added.

5. If the concentration does not increase by the correct amount, refer to Standard Additions in Section 1 of the procedures manual.

Standard Solution Method
Use a 50 mg/L Sulfate Standard Solution to validate the test procedure, reagents and the instrument. Select the SO4 L test range in step 3 and use 10 mL of the standard solution instead of the sample in step 4. To adjust the result, refer to *Standard Adjust*.

To prepare this standard solution, add 5.0 mL of a 1000 mg/L Sulfate Standard Solution to a 100-mL volumetric flask. Dilute to the mark with deionized water and mix fully.

Standard Adjust
The standard adjust option is recommended when program 128 is used.

6. Measure the concentration of a 50 mg/L Sulfate Standard Solution. Select the SO4 L range and use 10 mL of the standard solution. Keep the sample cell in the instrument.

7. Push the **SETUP** key and use the arrow keys to scroll to the STD option.

8. Push **ENTER**.

9. Push the numbers **50** to make the instrument read the value of the standard solution concentration.

10. Push **ENTER** to complete the adjustment.

Note: *The MR and HR calibration curves are adjusted proportionally when the SO4 L calibration curve is adjusted. Refer to Section 1, Standard Curve Adjustment of the procedures manual.*

Method Performance

Precision
In a single laboratory, with a 50 mg/L sulfate standard solution, two representative lots of powder pillows and the instrument, a single operator got a standard deviation of ± 0.5 mg/L sulfate.

Estimated Detection Limit (EDL)
The EDL for program 128 SO4 L is 4.9 mg/L SO_4. For more information on derivation and use of the estimated detection limit, refer to Section 1 of the procedures manual.

Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 Sulfate Reagent to make an insoluble barium sulfate precipitate. The amount of precipitate is proportional to the sulfate concentration. The SulfaVer 4 also contains a stabilizing agent to hold the precipitate in suspension.

Instrument Setup

This procedure adds program 128 to a DR/820, DR/850 or DR/890 instrument.

1. Push the **ON** key to turn on the instrument.

2. Push the **SETUP** key.

3. Push the down arrow key until the prompt line shows USER.

4. Push the **ENTER** key.

5. Push the numbers **8138**, then push **ENTER**.

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6. Refer to *Table 2*. Find the number from the Enter column that corresponds to Line Number 1 on the display. Push these numbers on the keypad, then push **ENTER**. Continue to add the numbers that correspond to each line number on the display.

Note: *Use the arrow keys to scroll and review or change numbers at any time.*

Table 2 Instrument setup

Line Number	Enter	Line Number	Enter
1	128	29	77
2	24	30	83
3	72	31	79
4	0	32	52
5	0	33	32
6	0	34	72
7	0	35	65
8	65	36	32
9	198	37	0
10	169	38	0
11	251	39	66
12	66	40	200
13	25	41	0
14	10	42	0
15	61	43	0
16	0	44	80
17	0	45	0
18	0	46	3
19	0	47	30
20	83	48	1
21	48	49	44
22	52	50	0
23	32	51	0
24	76	52	0
25	83	53	0
26	79	54	37
27	52	55	0
28	32	56	255

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REQUIRED REAGENTS AND APPARATUS

Description	Quantity Per Test	Units	Item No.
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow	100/pkg	2106769
Sample Cell, 10-20-25 mL, with cap	1	6/pkg	2401906

OPTIONAL REAGENTS

Description	Units	Item No.
Sulfate Standard Solution, 50 mg/L	500 mL	257849
Sulfate Standard Solution, 1000 mg/L	500 mL	2175749
Water, deionized	4 L	27256

OPTIONAL APPARATUS

Description	Units	Item No.
Cylinder, graduated mixing, 10 mL	1	2088638
Filter Paper, folded, 12.5 cm	100/pkg	189457
Flask, volumetric, 100 mL, Class A	1	1457442
Funnel, poly, 65 mm	1	108367
Pipet, TenSette, 0.1 to 1.0 mL	1	1970001
Pipet Tips, for 19700-01 Pipet	50/pkg	2185696
Pipet, TenSette, 1.0 to 10.0 mL	1	1970010
Pipet Tips, for 19700-10 Pipet	50/pkg	2199796
Pipet, volumetric, 5.00 mL, Class A	1	1451537
Pipet Filler, safety bulb	1	1465100

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HACH COMPANY
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Telephone: (970) 669-3050
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