



DR/4000 PROCEDURE

SULFIDE

✓ Method 8131

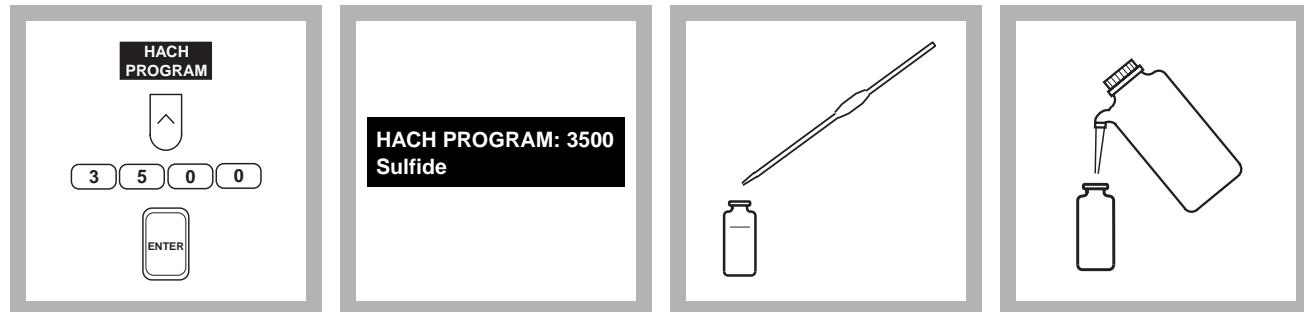
Methylene Blue Method*

(0 to 800 $\mu\text{g/L}$)

Scope and Application: For testing total sulfides, H_2S , HS^- and certain metal sulfides in groundwater, wastewater brines and seawater; USEPA accepted for reporting wastewater analysis**

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

** Procedure is equivalent to USEPA method 376.2 and Standard Method 4500-S²⁻ D for wastewater.



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

Select the stored program number for sulfide (S^{2-}) by pressing **3500** with the numeric keys.

Press: **ENTER**

Note: The Flow Cell and Sipper Modules can be used with this procedure.

Note: Samples must be analyzed immediately and cannot be preserved for later analysis. Avoid excessive agitation of samples.

2. The display will show:

HACH PROGRAM: 3500 Sulfide

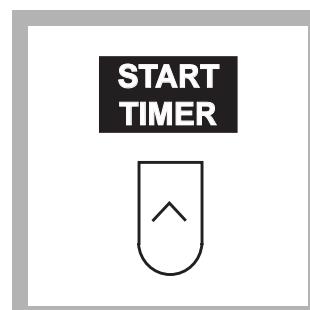
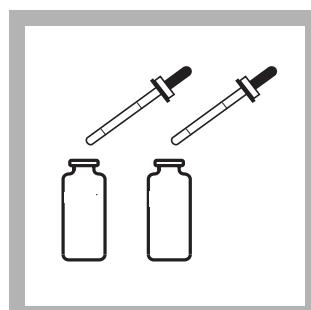
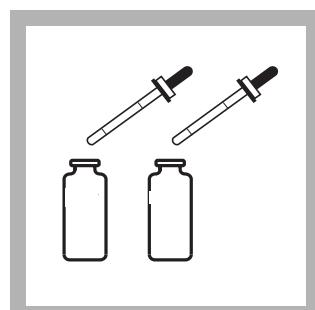
The wavelength (λ), **665 nm**, is automatically selected.

3. Measure 25 mL of sample into a sample cell. This will be the prepared sample.

Note: For turbid samples, see *Interferences* (following these steps) for pretreatment instructions.

Note: Excessive agitation will cause loss of sulfide. Use a pipet to minimize sulfide loss.

4. Measure 25 mL of deionized water into a second sample cell (the blank).



5. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

Note: Use the calibrated 1-mL dropper.

6. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

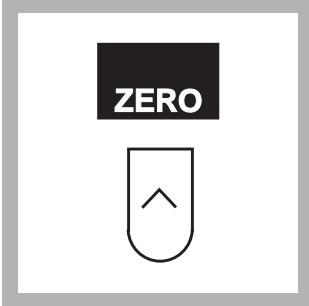
Note: A pink color will develop, then the solution will turn blue if sulfide is present.

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

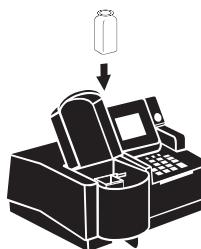
A 5-minute reaction period will begin.

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

SULFIDE, continued



ZERO



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

The display will show:

0 µg/L S²⁻

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. Place the prepared sample in the cell holder. Close the light shield. Results in µg/L sulfide (or chosen units) will be displayed.

Note: Some sulfide loss may occur if dilution is necessary.

Interferences

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interfere by reducing the blue color or preventing its development
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. <ol style="list-style-type: none">1. Measure 25 mL of sample into a 50-mL erlenmeyer flask.2. Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears.3. Add Phenol Solution dropwise until the yellow color just disappears. Use this solution in place of deionized water in step 4.

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Method Performance

Precision

Standard: 400 $\mu\text{g/L}$ S^{2-}

Program	95% Confidence Limits
3500	399–401 $\mu\text{g/L}$ S^{2-}

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

Program	EDL
3500	2 $\mu\text{g/L}$ S^{2-}

For more information on derivation and use of Hach's estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 3500

Portion of Curve	ΔAbs	$\Delta\text{Concentration}$
Entire Range	0.010	4.9 $\mu\text{g/L}$

See Section 1.5.3 *Sensitivity Explained* for more information.

Determining Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine sulfate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration.

High sulfide levels in oil field waters may be determined after proper dilution.

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

Sulfide 2 reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. Please see Section 1 for further information on proper disposal of these materials.

SULFIDE, continued

REQUIRED REAGENTS AND STANDARDS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445-00
Includes: (2) 1816-32, (2) 1817-32	

Description	Quantity Required		Cat. No.
	per test	Unit	
Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32
Water, deionized	25 mL	4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, 25-mL	1	each	508-40
<i>or</i>			
Pipet, volumetric, Class A, 25-mL	1	each	14515-40
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS AND STANDARDS

Bromine Water, 30-g/L	29 mL	2211-20
Phenol Solution, 30-g/L	29 mL	2112-20

OPTIONAL EQUIPMENT AND SUPPLIES

DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm	each	48070-05
DR/4000 Sipper Module Kit, 1-inch	each	48090-03
Dropper, for 1-oz. bottle	each	2258-00
Flask, Erlenmeyer, 50-mL	each	505-41



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