

Standard Test Methods for Screening of Reactive Sulfides in Waste¹

This standard is issued under the fixed designation D 4978; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods are applicable to the screening of reactive sulfides in wastes, liquids, sludges, semisolids, and solids by using the following methods:

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Test Method A—Lead Acetate Paper	9-14
Test Method B—Gas Detector Tube	15-19

1.2 These test methods are not applicable in determining the type and concentration of reactive sulfides.

1.3 These test methods are designed and intended as a preliminary test to complement the more sophisticated quantitative analytical techniques that may be used to determine sulfide concentration. These test methods offer, to the user, the option and the ability to screen waste for potentially hazardous levels of reactive sulfide when the sophisticated techniques are not available and the total waste composition is unknown.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard information is given in Section 7 and 11.3.

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *reactive sulfide*—a compound containing sulfide that readily forms hydrogen sulfide gas upon reaction with acid.

3.1.2 *screening analysis*—a preliminary qualitative or semi-quantitative test developed from classical qualitative and quantitative techniques that is designed to efficiently give the user parameters about a waste that will aid in determining waste identification, process compatibility, and safety in handling.

4. Summary of Test Methods

4.1 *Method A: Lead Acetate Paper*—A sample of waste material is acidified. If sulfides are present in the waste, hydrogen sulfide (H_2S) is evolved. In the presence of hydrogen sulfide, lead acetate paper changes color to silvery brown or black.

4.2 *Method B: Gas Detector Tube*—A portion of the sample is acidified in a beaker to release sulfide as hydrogen sulfide gas. The gas is funneled through a detector tube creating a color stain in the tube proportionate to the concentration of sulfide gas in the vapor. A definite color change in the detector tube indicates a positive presence of sulfide.

5. Significance and Use

5.1 These test methods are intended for use by waste treatment, storage, disposal, and remedial facilities, in order to show the presence of potentially hazardous sulfide.

5.2 *Method B: Gas Detector Tube*—This proposed test method was designed to measure gases migrating into an air space at a height of 10 units (for example, 10 ft) from a receiving or mixing pit of 10 units square (for example, 100 ft²). In essence, the total volume of the atmosphere is approximately 10 times the surface area of the sample and test solution.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

¹ These test methods are under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.05 on Screening Methods.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

7. Hazards

- 7.1 These tests should be conducted under a fume hood.
 7.2 Avoid inhalation and skin or eye contact, or both, with all hazardous material.

8. Sampling

- 8.1 Collect a representative sample of the waste in a sealed container.
 8.2 Store the sample in an operating fume hood.
 8.3 The sample should be analyzed as soon as possible.

TEST METHOD A—LEAD ACETATE PAPER

9. Interferences

- 9.1 Compounds evolving gases or vapors upon acidification that react with lead may interfere causing false positives.

NOTE 1—All positives should be followed up by a more detailed method, that is, distillation for total sulfides.

10. Reagents and Materials

- 10.1 *Beakers or Test Tubes.*
 10.2 *Stirring Rod.*
 10.3 *Hydrochloric Acid (1 + 1)*—Add 1 volume of hydrochloric acid (HCl, sp gr 1.19) to 1 volume water.
 10.4 *Lead Acetate Paper.*
 10.5 *Sodium Sulfide, Standard Solution (1 mL = 1 mg Na₂S)*—Dissolve 1.00 g sodium sulfide (Na₂S) in water and dilute to 1 L. Make this solution fresh daily.
 10.6 *Quality Control Check Standard*—Dilute an appropriate volume of sodium sulfide standard solution with water to produce a solution with the desired action level. Make this solution fresh daily.

11. Procedure

- 11.1 Place 5 to 10 g of sample into the beaker (slurry with approximately 15 mL water if the material is solid).
 11.2 Wet a strip of lead acetate paper and let it adhere to the side of beaker above the sample.
 11.3 Slowly and carefully adjust the pH to less than 2.0 with HCl solution 1 + 1.

NOTE 2—**Warning:** The addition of HCl to waste materials may cause violent reaction or highly toxic vapors, or both.

- 11.4 Stir the solution.
 11.5 A silvery brown to black color on the lead acetate paper indicates the presence of sulfides and shall be recorded as positive. Compare the color observed to a blank and standard solution.
 11.6 No color change to the lead acetate paper will be reported as negative.

12. Report

- 12.1 Report the following information:
 12.1.1 Sample identification,
 12.1.2 Date of test,
 12.1.3 Test procedure performed, and
 12.1.4 Sample classification: positive or negative.

13. Quality Control

- 13.1 A quality control check sample shall be tested with

each batch of samples. The concentration of this sample should reflect the action level required by the laboratory. Method or reagent blanks, duplications, and fortification (spikes), should be performed at an action level specified by the laboratory at an appropriate frequency.

13.2 Method detection limits should be determined by each laboratory using the standard and at the appropriate action level.

14. Precision and Bias

14.1 No statement is made about either the precision or bias of Test Method A for measuring sulfides in wastes since the result merely states whether there is conformance to the criteria for success specified in the procedure.

TEST METHOD B—GAS DETECTOR TUBE

15. Reagents and Materials

- 15.1 *Buffer Solution*—Dissolve 740 g trisodium phosphate (Na₃PO₄) in 3.5 L of water. Add carefully 500 mL of phosphoric acid (H₃PO₄, 85 %) and mix. Adjust the pH to 2 with H₃PO₄ if necessary.
 15.2 *pH Indicator Paper*, range: 0 to 14.
 15.3 *Disposable Beaker*, 400-mL.
 15.4 *Plastic Funnel*, 8-cm.
 15.5 *Jack Stand.*
 15.6 *Ring Stand and Clamps.*
 15.7 *Gas Detector Pump* with a volume range of 0 to 100 cc.
 15.8 *Gas Detector Tube* for hydrogen sulfide.⁴
 15.9 *Magnetic Stirrer.*
 15.10 *Magnetic Stirring Bar.*
 15.11 See Fig. 1 for suggested equipment setup.

16. Procedure

- 16.1 Place approximately 20 g of the material to be tested into a beaker with a magnetic stirring bar. Solid samples shall be crushed into small particles <9.5 mm in size (3/8-in. mesh screen).
 16.2 Add approximately 50 mL of the buffer solution to the sample.
 16.3 With a lab jack, raise the beaker and magnetic stirrer to a point at which the funnel fits down inside the beaker opening.
 16.4 The gas detector tube with the pump attached should be directly positioned through the small opening of the funnel, with the tip of the tube about midway in the funnel area.
 16.5 Begin stirring the sample.
 16.6 Activate the pump by pulling out the handle to draw in 100 cm³ of atmosphere from the apparatus.
 16.7 A positive sulfide is determined by a color stain in the detector tube.
 16.8 Allow at least a 1-min reaction time before reading results.

17. Report

- 17.1 Report the following information:

⁴ Precision gas detector tubes available from Matheson Safety Products, East Rutherford, NJ 07073, have been found to be satisfactory for this purpose.

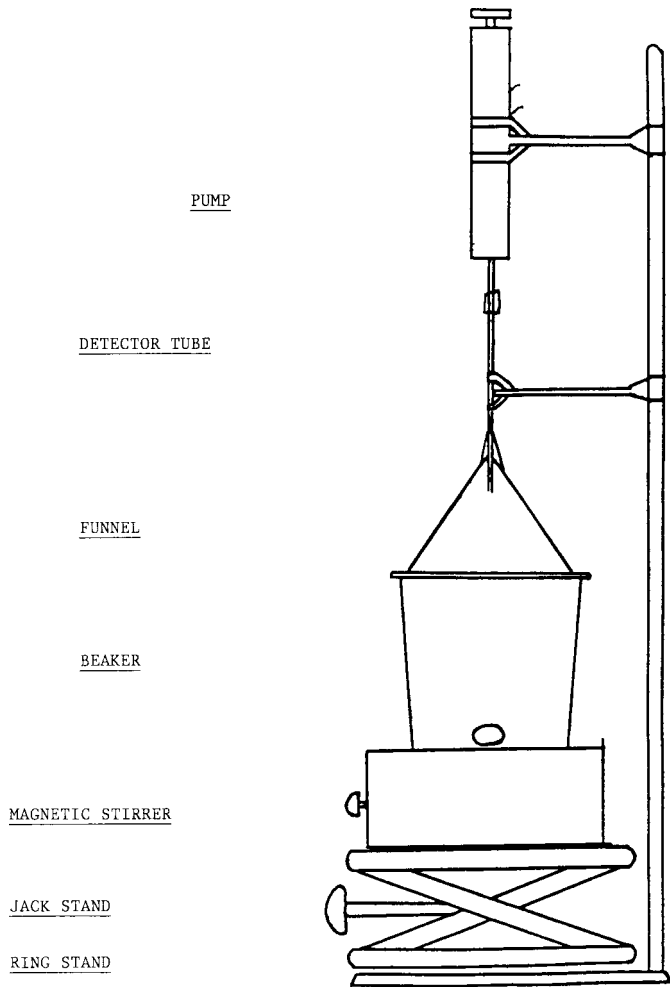


FIG. 1 Method B, Sulfide Test Apparatus

- 17.1.1 Sample identification,
- 17.1.2 Date of test,

- 17.1.3 Test procedure performed, and
- 17.1.4 Sample classification: positive or negative.

18. Quality Control

18.1 A quality control check sample shall be tested with each batch of samples. The concentration of this sample should reflect the action level required by the laboratory. Method or reagent blanks, duplications, and fortification (spikes), should be performed at an action level specified by the laboratory at an appropriate frequency.

18.2 Method detection limits should be determined by each laboratory using the standard and at the appropriate action level.

19. Precision and Bias

19.1 No statement is made about either the precision or bias of Method B for measuring sulfides in wastes since the result merely states whether there is conformance to the criteria for success specified in the procedure.

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