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Standard Practice for Determining Concentration of Hydrogen Sulfide by Direct Reading, Length of Stain, Visual Chemical Detectors[1]

This standard is issued under the fixed designation D4913; 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 This practice covers the detection of hydrogen sulfide gas by visual chemical detectors. Included under visual chemical detectors are: short-term detector tubes (**1**),[2] long-term detector tubes (**2**), and length-of-stain dosimeters (**3**). Diffusion tubes are not included under this practice because they are not direct reading, and spot tests are not included because of their poor accuracy. The sample results are immediately available by visual observation, thus no analytical equipment is needed.

1.2 This practice reflects the current state-of-the-art for commercially available visual length-of-stain detectors for hydrogen sulfide. Any mention of a specific manufacturer in the text or references does not constitute an endorsement by ASTM.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3686 Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method)

D4490 Practice for Measuring the Concentration of Toxic Gases or Vapors Using Detector Tubes

D4599 Practice for Measuring the Concentration of Toxic Gases or Vapors Using Length-of-Stain Dosimeters

2.2 *Other Documents:*

CFR 1910.1000 Federal Occupational Safety and Health Standard Title 29, Part 1910.1000, Subpart Z, and Part 1926.55, Subpart D[4]

NIOSH Criteria for a Recommended Standard, Occupational Exposure to Hydrogen Sulfide, 1977[5]

Threshold Limit Values for Chemical Substances in the Work Environment Adopted by the American Conference of Governmental Industrial Hygienists, latest issue[6]

2.3 *ANSI Standard:*[7]

ANSI/ISEA 102–1990(R1998) Gas Detector Tube Units-Short-Term Type for Toxic Gases and Vapors in Working Environments

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1356.

4. Summary of Practice

4.1 In general, the length-of-stain visual detectors described in this practice consist of a sealed glass tube filled with a reactive chemical dispersed on a granular material or a paper strip. This reactive material is sensitive to hydrogen sulfide and changes color upon exposure to hydrogen sulfide gas. These detectors are designed so that the length of the color change in the tube is related to the concentration of hydrogen sulfide under conditions of sampling volume or sampling time specified by the manufacturer. These detectors are typically calibrated by the manufacturer on an individual lot basis. Three general types of detectors are in current use.

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard practice.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] Code of Federal Regulations, available from the U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401.

[5] Available from National Technical Information Service (NTIS), 5285 Port Royal Rd., Springfield, VA 22161, http://www.ntis.gov.

[6] Available from American Conference of Governmental Industrial Hygienists, Inc. (ACGIH), 1330 Kemper Meadow Dr., Cincinnati, OH 45240, http://www.acgih.org.

[7] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

4.1.1 *Short-Term Detector Tubes*—A given volume of air (specified by the manufacturer) is pulled through the tube by a pump in a time period on the order of 1 to 10 min. The length-of-stain is related to the amount of air sampled and the hydrogen sulfide concentration during the sample period.

4.1.2 *Long-Term Detector Tubes*—The air sample is pulled through the tube at a slower, constant flow rate (specified by the manufacturer) by an electrical pump over a 1- to 8-h time period. The length of color change and the sampling time are used to estimate the time weighted average (TWA) concentration of hydrogen sulfide.

4.1.3 *Length-of-Stain Dosimeter Tubes*—This detector type samples the atmosphere to be tested by diffusion; no pump is required. Sampling times are on the order of 1 to 8 h and the stain length as a function of sampling time is related to the TWA concentration of hydrogen sulfide.

4.2 Information on the correct use of detector tubes and length-of-stain dosimeters is presented.

5. Significance and Use

5.1 *General*—Hydrogen sulfide is nearly ubiquitous. It occurs naturally in volcanic gases, in sulfur springs and fumaroles, in decaying of plant and animal protein, and in intestines as a result of bacterial action. Hydrogen sulfide is a serious hazard to the health of workers employed in energy production from hydrocarbon or geothermal sources, in the production of fibers and sheets from viscose syrup, in the production of deuterium oxide (heavy water), in tanneries, sewers, sewage treatment and animal waste disposal, in work below ground, on fishing boats, and in chemical operations, including the gas and oil industry.

5.2 In 29 CFR 1910.1000, the Federal Occupational Safety and Health Administration designates that worker exposure to certain gases and vapors must not be exceeded in workplace atmospheres at concentrations above specific values, averaged over a certain time span. Hydrogen sulfide is included in this list. Refer also to NIOSH Criteria for a Recommended Standard, Occupational Exposure to Hydrogen Sulfide.

5.3 This practice will provide means for the determination of airborne concentrations of hydrogen sulfide.

5.4 This practice provides means for either personal or area sampling and for short-term or time-weighted average (TWA) measurements. Refer to Threshold Limit Values for Chemical Substances in the Work Environment.

6. Detector Tubes, Short-Term (1, 4-9)

6.1 *General*—Short-term detector tubes are used for grab-sampling. They allow determination of hydrogen sulfide concentrations in approximately five min.

6.2 *Apparatus:*

6.2.1 Detector tubes for the detection of hydrogen sulfide and an appropriate pump are required. Because manufacturers have different tubes for different measurement ranges of hydrogen sulfide, an estimate of the expected hydrogen sulfide concentration is helpful. Detector tubes made by one manufacturer must not be used with pumps made by a different manufacturer (10).

6.2.2 The Safety Equipment Institute (SEI) has a voluntary certification program for short-term detector tubes for hydrogen sulfide. This program consists of independent laboratory verification of sampling pump precision and of detector tube performance. The pump and tube units certified under this program are listed in the SEI Approved Equipment List. The certification test protocol is similar to that used in the now discontinued NIOSH Certification Program (11-13).

6.2.3 In some sampling situations, particularly when testing for hazardous hydrogen sulfide concentrations in confined spaces, a remote sampling line and adapter can be used. This allows placement of the detector tube in the potentially hazardous area to be tested while the operator is in a safer area.

6.3 *Procedure:*

6.3.1 Carefully follow the instruction sheet of the manufacturer for the proper use of hydrogen sulfide short-term detector tubes (see Practice D4490).

6.3.1.1 Check the pump for leaks, total volume, and flow rate in accordance with the instruction manual for the pump. Also check the sampling line for leaks (if used).

6.3.1.2 Remove one detector tube from the box and break off both tips.

6.3.1.3 Insert the detector tube into the tube holder of the pump or onto the sample line, making sure that it is properly oriented.

6.3.1.4 Face the mounted detector tube into the atmosphere to be tested. Sample an appropriate volume of air by pulling the pump handle out the required number of strokes, if using a piston pump, or squeezing the pump the proper number of times, if using a bellows-type or bulb-type pump.

6.3.1.5 If hydrogen sulfide is present, the indicator chemical in the tube will change color from white to brown or gray. The length of stain, correlated with the volume of air sampled, will indicate the concentration. Most detector tubes now have direct reading concentration scales printed on the tube.

6.3.1.6 For the most reliable estimate of the hydrogen sulfide concentration, the maximum number of pump strokes (as specified by the manufacturer) should be taken so that the stained length is between 20 and 80 % of the total indicating chemical length. At very low or very high concentrations, this is not always possible.

6.4 *Interferences*—When using lead salts as the impregnant, sulfur dioxide, nitrogen dioxide, and mercaptans can interfere with accurate measurements, particularly at low hydrogen sulfide concentrations. Sulfur dioxide, if present at comparable concentrations to hydrogen sulfide, will give a positive interference, that is, will increase the length of stain; however, sulfur dioxide by itself will cause no stain. Mercaptans at or near their TLV levels will not interfere, but at high concentrations (more than 100 ppm) they can give a positive interference. Nitrogen dioxide, at concentrations near its TLV, gives a slight negative error. The effect of these interferences is minimal when silver cyanide, copper, or mercury compounds are used as the impregnant.

6.5 The accuracy of detector tubes from two manufacturers was tested over a temperature range of 4°C [40°F] to 49°C [120°F] and over a relative humidity range of 20 % to 90 %

(14). The accuracy of these hydrogen sulfide detector tubes was reduced only at high temperatures (one manufacturer).

6.6 *Measurement Range*—Hydrogen sulfide tubes are available to measure concentrations from 0.5 to 70 000 ppm.

6.7 *Accuracy*—SEI certified detector tubes must have an accuracy (precision plus bias) of ±25 % of the actual concentration or better for hydrogen sulfide concentrations from 10 to 50 ppm. At 5 ppm the accuracy must be better than ±35 %. At concentrations higher than 50 ppm, a minimum accuracy is not specified in the SEI requirements, but is typically better than ±25 %. Manufacturers of non-SEI certified hydrogen sulfide detector tubes typically claim accuracies of better than 25 to 35 %.

6.8 *Reagents*—The reagent system used to detect the presence of hydrogen sulfide is either lead acetate, a mercury complex, a copper salt, or silver cyanide. These salts are impregnated on either granular alumina or silica gel.

7. Detector Tubes, Long-Term (2, 15, 16)

7.1 *General*—Long-term sampling equipment detects and measures hydrogen sulfide over an entire work shift (or fraction thereof) without the need for time consuming laboratory analysis. This system measures a time-weighted-average (TWA) concentration.

7.2 *Apparatus*—Detector tubes for detecting hydrogen sulfide and an electrical pump for pulling the sample through the tube at a low rate, such as 5 to 20 mL/min.

7.3 *Procedure:*

7.3.1 Carefully follow the instruction sheet of the manufacturer for the proper use of hydrogen sulfide long-term tubes.

7.3.1.1 Check the pump for proper flow rate or volume per pump stroke in accordance with the instruction manual for the pump.

7.3.1.2 Remove one detector tube from the box and break off both tips.

7.3.1.3 Insert the tube into the tube holder, making sure it is properly oriented.

7.3.1.4 If necessary, connect the tube holder to the pump. With some systems, the tube is connected directly to the pump without the use of a tube holder.

7.3.1.5 Set the pump to the proper flow rate and turn it on. Record the starting time. For accurate work, the flow rate of the pump should be calibrated under actual use conditions. Refer to appropriate Annex of Practice D3686.

7.3.1.6 Continue sampling for the length of time desired, within the ppm-h capability of the tube. Record the time at the end of sampling.

7.3.1.7 If hydrogen sulfide is present, a brownish stain develops in the detector chemical. To determine the concentration, either read the ppm-h directly from the tube and divide by the sampling time (h) or refer to a calibration graph, finding the point of intersection of the stain length and the sampling time and read from the graph the corresponding concentration.

7.4 *Interferences*—The interferences are the same as for short-term hydrogen sulfide tubes (see 6.4).

7.5 Temperature and relative humidity have little effect as in the case of short-term detector tubes.

7.6 Measurement range 0.5 to 100 ppm.

7.7 *Accuracy*—Typical accuracy (precision plus bias) for long-term detector tubes is better than ±25 % of the actual concentration (16).

7.8 Reagents same as short-term tubes (see 6.8).

8. Length-of-Stain Dosimeters (3, 17, 18)

8.1 *General*—Hydrogen sulfide length-of-stain dosimeters measure TWA concentrations. They operate by diffusion and, therefore, require no auxiliary equipment. They detect and measure hydrogen sulfide over periods from 0.5 to 8.0 h (or longer) without the need for laboratory analysis.

8.2 *Apparatus*—All that is required is a hydrogen sulfide length-of-stain dosimeter and the dosimeter holder supplied by the manufacturer. Dosimeters and holders supplied by different manufacturers should not be interchanged.

8.3 *Procedure:*

8.3.1 Carefully follow the instruction sheet of the manufacturer for the proper use of hydrogen sulfide length-of-stain dosimeter tubes (see Practice D4599).

8.3.1.1 The dosimeter is opened at one end to allow diffusion of the gas into the tube.

8.3.1.2 The tube is mounted into a tube holder, which can be clipped onto a collar or pocket for personal monitoring, or onto a stationary object for area monitoring.

8.3.1.3 The chemically impregnated paper or gel within the tube changes color from white to brownish if hydrogen sulfide is present.

8.3.1.4 The length of color stain and the time of exposure are used to determine the TWA concentration of hydrogen sulfide to which the tube had been exposed.

8.4 *Interferences*—The interferences are the same as for short-term hydrogen sulfide detector tubes (see 6.4), however a certain minimum air movement or face velocity is required for accurate results.

8.5 A 10°C change in temperature causes about a 2 % change in the concentration, higher temperatures giving longer stains. High relative humidity (over 70 %) cause a shortening of the length of stain and a correction factor should be used, which, if required, is given in the manufacturers' instruction sheets.

8.6 Measurement range 0.5 to 150 ppm (typical).

8.7 *Accuracy*—Typical accuracy (precision plus bias) for hydrogen sulfide dosimeters is better than ±25 % of the actual concentration (3, 18).

8.8 *Reagents*—Same as for short-term detector tubes (see 6.8), impregnated on either filter paper, silica gel, or granular alumina.

9. Keywords

9.1 air monitoring; detector tubes; hydrogen sulfide; length-of-stain dosimeters; sampling and analysis; workplace atmospheres

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