Designation: D4810 – 06

Standard Test Method for Hydrogen Sulfide in Natural Gas Using Length-of-Stain Detector Tubes[1]

1. Scope

1.1 This test method covers a procedure for a rapid and simple field determination of hydrogen sulfide in natural gas pipelines. Available detector tubes provide a total measuring range of 0.5 ppm by volume up to 40 % by volume, although the majority of applications will be on the lower end of this range (that is, under 120 ppm).

1.2 Typically, sulfur dioxide and mercaptans may cause positive interferences. In some cases, nitrogen dioxide can cause a negative interference. Most detector tubes will have a "precleanse" layer designed to remove certain interferences up to some maximum interferent level. Consult manufacturers' instructions for specific interference information.

1.3 The values stated in SI units are to be regarded as the 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 *Gas Processors Association Standard:*

No. 2377-86 Test for Hydrogen Sulfide in Natural Gas Using Length of Stain Tubes[2]

3. Summary of Test Method

3.1 The sample is drawn through a detector tube filled with a specially prepared chemical. Any hydrogen sulfide present in the sampling reacts with the chemical to produce a color change or stain. The length of the stain produced in the detector tube, when exposed to a measured volume of sample, is directly proportional to the amount of hydrogen sulfide present in the sample. A hand-operated piston or bellows-type pump is used to draw a measured volume of sample through the tube at a controlled rate of flow. The length of stain produced is converted to ppm (by volume) hydrogen sulfide (H_2S), by comparison to a calibration scale supplied by the manufacturer for each box of detection tubes (higher range tubes have units of percent by volume). The system is direct reading, easily portable, and completely suited to making rapid spot checks for hydrogen sulfide under field conditions.

4. Significance and Use

4.1 The measurement of hydrogen sulfide in natural gas is important because of the gas quality specifications, the corrosive nature of H_2S on pipeline materials, and the effects of H_2S on utilization equipment.

4.2 This test method provides inexpensive field screening of hydrogen sulfide. The system design is such that it may be used by nontechnical personnel with a minimum of proper training.

5. Apparatus

5.1 *Length-of-Stain Detector Tube and Calibration Scale*—A sealed glass tube with breakoff tips sized to fit the tube holder of the pump. The reagent layer inside the tube, typically a silica gel substrate coated with the active chemicals, must be specific for hydrogen sulfide and must produce a distinct color change when exposed to a sample of gas containing hydrogen sulfide. Any substances known to interfere must be listed in the instructions accompanying the tubes. A calibration scale should be marked directly on the tube or other markings which provide for easy interpretation of hydrogen sulfide content from a separate calibration scale supplied with the tubes. The calibration scale shall correlate hydrogen sulfide concentration to the length of the color stain. Shelf life of the detector tubes must be a minimum of two years from date of manufacture when stored according to manufacturers' recommendations.

5.2 *Detector Tube Pump*—A hand-operated pump of a piston or bellows type. It must be capable of drawing 100 cm^3 per stroke of sample through the detector tube with a volume tolerance of ±5 cm^3.[3] It must be specifically designed for use with detector tubes.

NOTE 1—A detector tube and pump together form a unit and must be

---

[1] This test method is under the jurisdiction of ASTM Committee D03 on Gaseous Fuels and is the direct responsibility of Subcommittee D03.07 on Analysis of Chemical Composition of Gaseous Fuels.

Current edition approved June 1, 2006. Published June 2006. Originally approved in 1988. Last previous edition approved in 1999 as D4810 - 98 (1999). DOI: 10.1520/D4810-06.

[2] Available from Gas Processors Association, 1812 First National Bank Bldg., Tulsa, OK 74103.

[3] *Direct Reading Colorimetric Indicator Tubes Manual* , 1st ed., American Industrial Hygiene Association, Akron, OH 44311, 1976.

used as such. Each manufacturer calibrates detector tubes to match the flow characteristics of their specific pump. Crossing brands of pumps and tubes is not permitted, as considerable loss of system accuracy is likely to occur.[3] (Note that at least one manufacturer allows extended samples up to 100 pumpstrokes to obtain lower detection levels. This may be automated for screening purposes by drawing the sample from an inert collapsable container by vacuum displacement. The sample flow rate should be maintained within ±5 % of the manufacturer's specified flow rate. Accuracy losses are apt to occur in such special applications, and such a system is recommended only for screening purposes. Consult manufacturers regarding limitations.)

5.3 *Gas Sampling Chamber*—Any container that provides for access of the detector tube into a uniform flow of sample gas at atmospheric pressure and isolates the sample from the surrounding atmosphere. A stainless steel needle valve (or pressure regulator) is placed between the source valve and the sampling chamber for the purpose of throttling the sample flow. Flow rate should approximate one to two volume changes per minute or, at minimum, provide positive exit gas flow throughout the detector tube sampling period.

NOTE 2—A suitable sampling chamber may be devised from a polyethylene wash bottle of nominal 500-mL (16-oz) or 1-L (32-oz) size. The wash bottle's internal delivery tube provides for delivery of the sample gas to the bottom of the bottle. A 12.5-mm (½-in.) hole cut in the bottle's cap provides access for the detector tube and vent for the purge gas (Fig. 1). (An alternate flow-through sampler may be fashioned using a 1-gal Ziploc-type food storage bag. The flexible line enters one corner of the bag's open end and extends to the bottom of the bag. The opposite corner of the open end is used for tube access and sample vent. The remainder of the bag's top is sealed shut. The basic procedure for the sampler in Fig. 1 applies.)

NOTE 3—An alternate sampling container is a collection bag made of a material suitable for the collection of natural gas (for example, Mylar). The sampling bag should have a minimum capacity of 2 L.

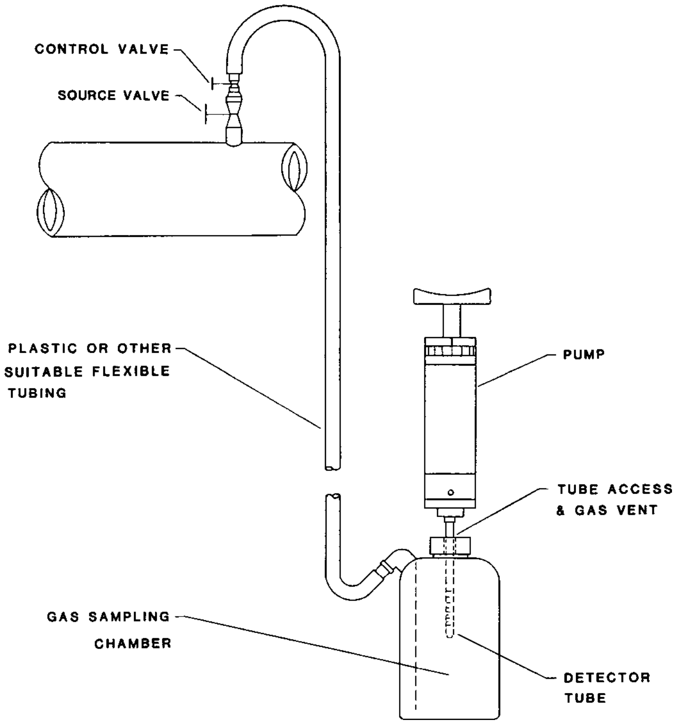

FIG. 1 Apparatus Schematic

6. Procedure

6.1 Select a sampling point that will provide access to a representative sample of the gas to be tested (for example, source valve on the main line). The sample point should be on top of the pipeline and equipped with a stainless steel sample probe extending into the middle third of the pipeline. Open the source valve momentarily to clear the valve and connecting nipple of foreign materials.

6.2 Install needle valve (or pressure regulator) at the source valve outlet. Connect sampling chamber using the shortest length of flexible tubing possible (Fig. 1). Avoid using tubing that reacts with or absorbs H_2S, such as copper or natural rubber. Use materials such as TFE-fluorocarbon, vinyl, polyethylene, or stainless steel.

6.3 Open source valve. Open needle valve enough to obtain positive flow of gas through chamber, in accordance with 5.3. Purge the container for at least 3 min (Fig. 1).

NOTE 4—If a collection bag is used instead of a sampling chamber, follow 6.1 and 6.2, substituting the bag for the chamber. Follow 6.3, disconnecting the bag when filled. Deflate the bag to provide a purge, and fill a second time to provide a sample. The bag must be flattened completely before each filling (Note 3).

6.4 Before each series of measurements, test the pump for leaks by operating it with an unbroken tube in place. Consult the manufacturer's instructions for leak check procedure details and for maintenance instruction, if leaks are detected. The leak check typically takes 1 min.

6.5 Select a detector tube with the range that best encompasses the expected H_2S concentration. Reading accuracy is improved when the stain length extends into the upper half of the calibration scale. Consult manufacturer's guidelines for using multiple strokes to achieve a lower range on a given tube.

6.6 Break off the tube tips and insert the tube into the pump, observing the flow direction indication on the tube. Place the detector tube into the sampling chamber through the access hole, so that the tube inlet is near the chamber center (Fig. 1).

NOTE 5—Detector tubes have temperature limits of 0 to 40°C (32 to 104°F), and sample gases must remain in that range throughout the test. Cooling probes are available for sample temperatures exceeding 40°C.

6.7 Operate the pump to draw the measured sample volume through the detector tube. Observe tube instructions when applying multiple strokes. Assure that a positive flow is maintained throughout the sample duration at the sampling chamber gas exit vent. Observe tube instructions for proper sampling time per pump stroke. The tube inlet must remain in position inside the sampling chamber until the sample is completed. Many detector tube pumps will have stroke finish indicators that eliminate the need to time the sample.

NOTE 6—If a collection bag is used, the sample is drawn from the bag via a flexible tubing connection. Do not squeeze the bag during sampling. Allow the bag to collapse under pump vacuum, so that the pump's flow characteristics are not altered.

6.8 Remove the tube from the pump and immediately read the H_2S concentration from the tube's calibration scale or from the charts provided in the box of tubes. Read the tube at the maximum point of the stain. If "channeling" has occurred (nonuniform stain length), read the maximum and minimum stain lengths and average the two readings.

NOTE 7—If the calibration scale is not printed directly on the detector tube, be sure that any separate calibration chart is the proper match for the tube in use.

6.9 If the number of strokes used differs from the number of strokes specified for the calibration scale, correct the reading, as below:

$$\text{ppm (corrected)} = \text{ppm (reading)} \times \frac{\text{specified strokes}}{\text{actual strokes}} \tag{1}$$

6.10 Record the reading immediately, along with the gas temperature and the barometric pressure. Observe any temperature corrections supplied in the tube instructions. Altitude corrections become significant at elevations above 2000 ft. Correct for barometric pressure, as below:

$$\text{ppm (corrected)} = \text{ppm (reading)} \times \frac{760\text{ mm Hg}}{\text{barometric pressure in mm Hg}} \tag{2}$$

NOTE 8—Although the amount of chemicals contained in detector tubes is very small, the tubes should not be disposed of carelessly. A general disposal method includes soaking the opened tubes in water before tube disposal. The water should be pH neutralized before its disposal.

7. Quality Assurance

7.1 Detector tubes from each batch or lot of tubes should be tested to conform the published accuracy, (generally ± 25 %).

7.2 The tubes should continue to meet the published accuracy until the expiration date, if the tubes are shipped and stored per manufacturer instructions.

8. Precision and Bias

8.1 The accuracy of detector tube systems is generally considered to be ±25 %. This value is based on programs conducted by the National Institute of Occupational Safety and Health (NIOSH) in certifying detector tubes for low-level contaminants in air, adapted to worker exposure monitoring.[4] NIOSH tested tubes at ½ , 1, 2, and 5 times the threshold limit value (TLV) requiring ±25 % accuracy at the three higher levels and ±35 % at the ½ TLV level.[5] (For example, H_2S with a TLV of 10 ppm was tested at levels of 5, 10, 20, and 50 ppm.) The higher tolerance allowed at the low level was due to the loss of accuracy for shorter stain lengths.[3] NIOSH discontinued this program in 1983, and it was picked up by the Safety Equipment Institute (SEI) in 1986.

8.1.1 The Gas Processors Association Standard No. 2377 for natural gas testing via H_2S detector tubes summarizes detector tube accuracy testing in natural gas in which all reported results are within ±23 %.

8.2 *Repeatability*—Duplicate results by the same operator, under the same test conditions, should produce results within ±10 % between 3- and 120-ppm H_2S and ±5 % between 0.05 and 5 % H_2S (see GPA No. 2377). Repeatability is optimized when all tests using a single brand are conducted with detector tubes of the same lot number.

9. Keywords

9.1 gaseous fuels; natural gas

---

[4] Septon, J. C. and Wilczek, T., "Evaluation of Hydrogen Sulfide Detector Tubes," *App. Ind. Hyg.*, Vol. 1, No. 4, 11/86.

[5] "NIOSH Certification Requirements for Gas Detector Tube Units," NIOSH /TC/A-012, 7/78.