Designation: D4984 − 06 (Reapproved 2015)

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

This standard is issued under the fixed designation D4984; 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 test method covers a rapid and simple field determination of carbon dioxide in natural gas pipelines. Available detector tubes provide a total measuring range of 100 ppm (parts per million) up to 60 % by volume, although the majority of applications will be on the lower end of this range (that is, under 5 %). At least one manufacturer provides a special kit for measurements from 10 to 100 % CO_2, but the normal 100-cc hand pump is not used. See Note 1.

NOTE 1—High-range carbon dioxide detector tubes will have measuring ranges in percent (%) CO_2, and low-range tubes will be in parts per million (ppm). To convert percent to ppm, multiply by 10 000 (1 % = 10 000 ppm).

1.2 The values stated in SI units are regarded as standard. The inch-pound units in parentheses are for information only.

1.3 *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:*

2337 Test for Hydrogen Sulfide and Carbon Dioxide in Natural Gas Using Length-of-Stain Tubes[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

3.1.1.1 *Discussion*—A detector tube and pump together form a unit and must be 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]

3.1.2 *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 1 to 2 volume changes per minute or, at minimum, provide exit gas flow throughout the detector tube-sampling period.

3.1.2.1 *Discussion*—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 sample gas to the bottom of the bottle. A 14.7-mm (½-in.) hole cut in the bottle's cap provides access for the detector tube and vent for the purge gas (see Fig. 1). (An alternate flow-through sampler may be fashioned using a 1-gal (3.8-L) "zipper"-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 bag's top is sealed shut. The basic procedure for the sampler in Fig. 1 applies.)

3.1.2.2 *Discussion*—An alternate sampling container is a collection bag made of a material suitable for the collection of natural gas (for example, polyester film). The sampling bag should have a minimum capacity of 2 L.

3.1.3 *length-of-stain detector tube*—a sealed glass tube with break-off tips sized to fit the tube holder of the pump. The reagent layer inside the tube, typically a silica gel substance coated with the active chemicals, must be specific for carbon dioxide and produce a distinct color change when exposed to a sample of gas containing carbon dioxide. Any substances known to interfere must be listed in the instructions accompanying the tubes. A calibration scale should be marked directly on the tube; however, other markings that provide for easy interpretation of carbon dioxide content from a separate calibration scale supplied with the tubes shall be acceptable.

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[1] This test method is issued 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 Nov. 1, 2015. Published December 2015. Originally approved in 1989. Last previous edition approved in 2011 as D4984–06 (2011). DOI: 10.1520/D4984-06R15.

[2] Available from Gas Processors Association, 6526 East 60th St., Tulsa, OK 74145.

[3] \, First ed., American Industrial Hygiene Association, Akron, OH 44311.

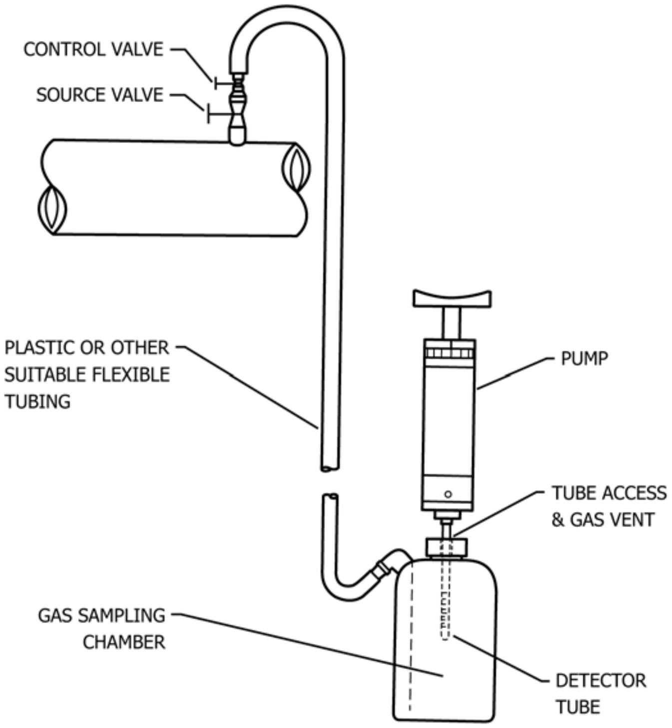

FIG. 1 Apparatus Schematic

The calibration scale shall correlate carbon dioxide concentration to the length of the color stain. Shelf life of the detector tubes must be a minimum of two years from the date of manufacture when stored according to manufacturers' recommendations.

4. Summary of Test Method

4.1 The sample is passed through a detector tube filled with a specially prepared chemical. Any carbon dioxide present in the sample 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 carbon dioxide 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 parts per million (ppm) or percent (%) carbon dioxide by comparison to a calibration scale supplied by the manufacturer for each box of detection tubes. The system is direct reading, easily portable, and completely suited to making rapid spot checks for carbon dioxide under field conditions. (See Note 1.)

5. Significance and Use

5.1 The measurement of carbon dioxide in natural gas is important, because of the gas quality specifications, the corrosive nature of carbon dioxide on pipeline materials, and the affects of carbon dioxide on utilization equipment.

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

6. Interferences

6.1 Detector tubes are usually subject to interferences from gases and vapors other than the target substance. Such interferences may vary among brands as a result of the use of different detection methods. Some detector tubes will have a "precleanse" layer designed to remove interferences up to some maximum interferent level. Consult manufacturer's instructions for specific interference information.

7. Procedure

7.1 Select a sampling point that provides access to a representative sample of the gas being tested (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.

7.2 Install needle valve (or pressure regulator) at the source valve outlet. Connect sampling chamber using the shortest length of flexible tubing possible (see Fig. 1). Most flexible tubing material will be suitable for carbon dioxide sampling; however, if the sampler is also used for other constituents such as hydrogen sulfide, then choose tubing materials carefully. Avoid using tubing that reacts with or absorbs hydrogen sulfide, such as copper or natural rubber. Use materials such as TFE-fluorocarbon, vinyl, polyethylene, or stainless steel.

7.3 Open source valve. Open needle valve enough to obtain positive flow of gas through chamber, in accordance with 3.1.2. Purge the container for at least 3 min (see Fig. 1).

NOTE 2—If a collection bag is used instead of a sampling chamber, follow 7.1 and 7.2, substituting the bag for the chamber. Follow 7.3, 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 (see second Discussion in 3.1.2).

7.4 Before each series of measurements, test the pump for leaks by operating it with an unbroken tube in place. Consult manufacturers' instructions for leak check procedure details and for maintenance instruction if leaks are detected. The leak check typically takes 1 min. A leaking pump used in field testing will bias sample results low.

7.5 Select the tube range that best encompasses carbon dioxide concentration. Reading accuracy is improved when the stain length extends into the upper half of the calibration scale. Consult manufacturer guidelines for using multiple strokes to achieve a lower range on a given tube.

7.6 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, such that the tube inlet is near the chamber center (see Fig. 1).

NOTE 3—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.

7.7 Operate the pump to draw the measured sample volume through the detector tube. Observe tube instructions when applying multiple strokes. Ensure 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 sample is completed. Many detector tube pumps have stroke finish indicators that eliminate the need to time the sample.

NOTE 4—If a collection bag is used, draw the sample 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.

NOTE 5—It is very important to ensure that ambient air is not being drawn into the sample. Intrusion of ambient air into the sample will tend to bias the carbon dioxide readings low.

7.8 Remove the tube from the pump and immediately read carbon dioxide 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. Consult tube instructions for any special information in the event of multicolored stains.

NOTE 6—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.

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

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

7.10 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 follows:

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

NOTE 7—Even though 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 treated to a neutral pH before its disposal.

8. Quality Assurance

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

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

9. Precision and Bias

9.1 The accuracy of detector tube systems is generally considered to be ±25 %. This is based mainly 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. (For example, CO_2, with a TLV of 5000 ppm, was tested at levels 2500, 5000, 10 000, and 25 000 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.

9.2 The Gas Processor's Association reports an observed error of ±8 % or better in tests of two brands of carbon dioxide detector tubes (see 2.1, 2337).

10. Keywords

10.1 gaseous fuels; natural gas

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[4] "NIOSH Certification Requirements for Gas Detector Tube Units," NIOSH-TC-012, 1978.