~~Designation:E1746–95 (Reapproved 2001)$^{\varepsilon 1}$~~ Designation: E 1746 – 08

Standard Test Method for Sampling and Analysis of Liquid Chlorine for Gaseous Impurities[1]

This standard is issued under the fixed designation E 1746; 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.

~~ε[1] NOTE—Editorial changes were made in August 2001.~~

1. Scope*

1.1 This test method covers sampling and analysis of liquid chlorine for the determination of oxygen (200 to 400 ~~ppm),~~µg/g), nitrogen (400 to 800 ~~ppm),~~µg/g), and carbon dioxide (800 to 1000 ppm) content at levels normally seen in liquid chlorine. Hydrogen and carbon monoxide concentrations in liquid chlorine are typically at or below the detection limit of this test method.

NOTE 1—The minimum detection limit of hydrogen using a 1 cm^3 gas sample and argon carrier gas is 100 to 200 ~~ppm.~~µg/g.[2] The detection limit for the other components is significantly lower.

~~1.2~~

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

1.3 Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

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 hazards statements are given in Section 7.

~~1.3Review the current Material Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.~~

2. Referenced Documents

2.1 *Code of Federal Regulations:*[3]

49 CFR 173, Code of Federal Regulations Title 49, Transportation: Shippers' General Requirements for Shipments and Packaging, including the following sections:

173.304 Charging of Cylinders with Liquefied Compressed Gas

173.314 Requirements for Compressed Gases in Tank Cars

173.315 Compressed Gases in Cargo Tanks and Portable Tank Containers

2.2 *Other Document:*

Chlorine Institute Pamphlet No. 77 Sampling Liquid Chlorine[4]

3. Summary of Test Method

3.1 A sample of liquid chlorine is trapped in a sampling tube and vaporized into a steel bomb. The vaporized chlorine in the steel bomb is introduced into a gas chromatograph by a gas sampling loop (1 cm^3) using a ten-port gas sampling and switching valve. The separations are made on a Porapak® Q column and on a 5A molecular sieve column whose lengths are such that the peaks do not overlap.

3.2 Any component that co-elutes with the components of interest may interfere with this analysis.

4. Significance and Use

4.1 It is very difficult to exclude sample contamination by ambient air during the process of sampling. The levels of atmospheric contamination caused by poor sampling methods are often equal to or larger than the levels of the gaseous impurities present in

[1] This test method is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and is the direct responsibility of Subcommittee E15.02 on Product Standards.

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[2] Thompson, B., *Fundamentals of Gas Chromatography*, Varian Instruments Division, Sunnyvale, CA, p. 73.

[3] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[4] Available from The Chlorine Institute, Inc., 2001 L Street NW, Washington, DC ~~20036-4919.~~20036-4919.

***A Summary of Changes section appears at the end of this standard.**

the chlorine. This results in markedly elevated levels of detected impurities. As specifications become tighter, it becomes more important to measure the gaseous impurity levels in liquid chlorine correctly.

4.2 Additional problems are experienced in the sampling of liquefied gases for the gaseous impurities. The gaseous impurities reach an equilibrium between the liquid phase and vapor phase in a sample bomb. The quantity of gases measured in any particular sample containing both liquid and vapor will be a function of the amount of vapor space in the sample bomb. This test method avoids the presence of liquid in the sample bomb.

5. Apparatus

5.1 *Gas Chromatograph*—Shimadzu GC-8AIT equipped as shown in Fig. 1, or equivalent, equipped with a thermal conductivity detector.

5.2 *Recorder*, 1 mV, 0.5 s full-scale response.

5.3 *Valve Sequencer and Actuator* , for switching valve control.

5.4 *Switching Valves*.

5.4.1 *Ten-Port Switching and Sampling Valve* (stainless steel is acceptable).

5.4.2 *Four-Port Switching Valve* (stainless steel is acceptable).

5.5 *Chromatographic Columns*, ~~-in. (3.2-mm)~~3.2-mm outside diameter, 316 stainless, as follows:

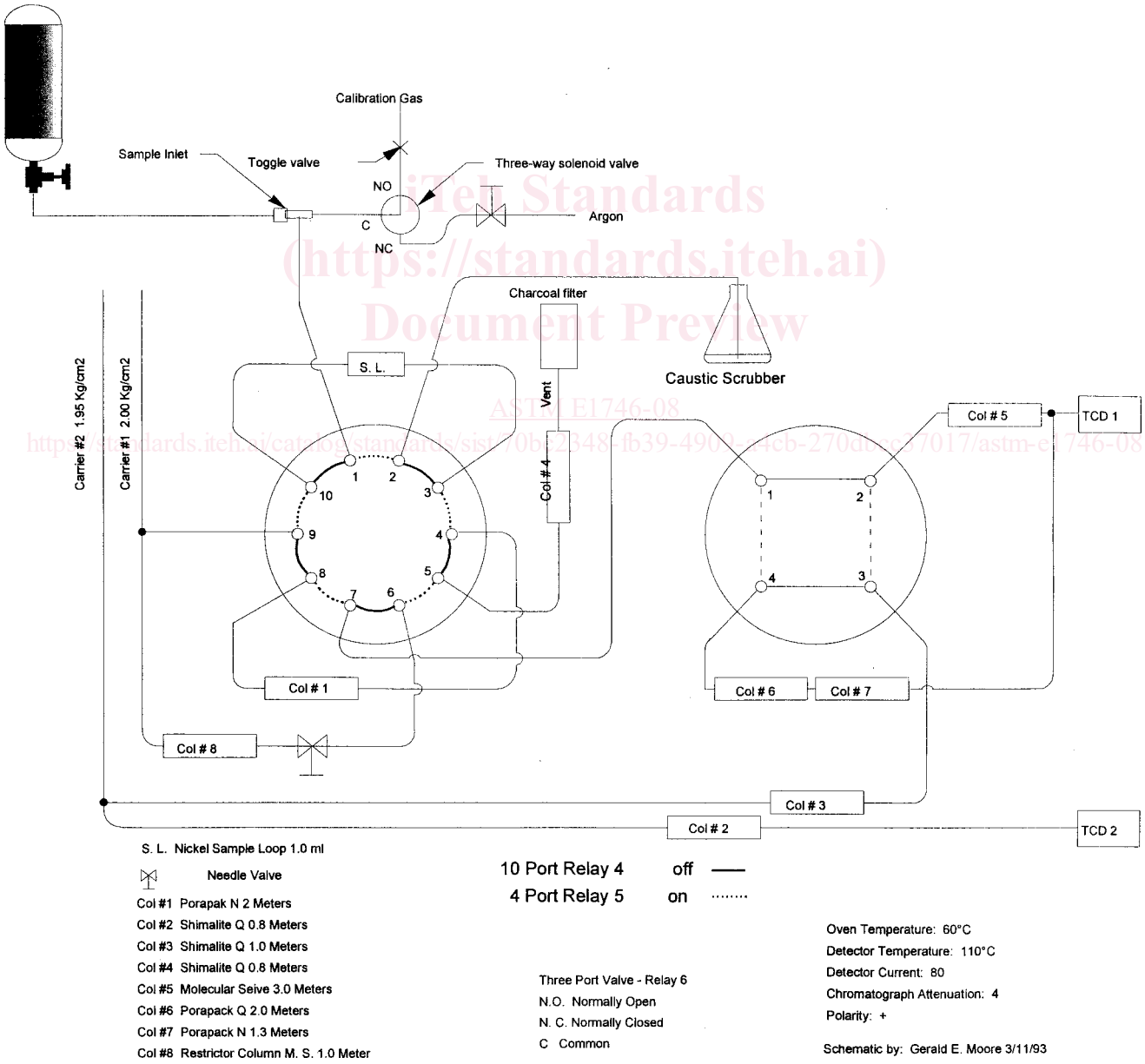

FIG. 1 Chlorine Impurity Analysis System Flow Diagram

5.5.1 2 m of 80/100 mesh Porapak® N,[5]
5.5.2 0.8 m of 80/100 mesh Shimalite® Q,[6]
5.5.3 1 m of 80/100 mesh Shimalite® Q,[6]
5.5.4 0.8 m of 80/100 mesh Shimalite® Q,[6]
5.5.5 3 m of 45/60 mesh molecular sieve 5A,
5.5.6 2 m of 80/100 mesh Porapak® Q,[5]
5.5.7 2 m of 80/100 mesh Porapak® N,[5]and
5.5.8 1 m of 45/60 mesh molecular sieve 5A.

5.6 *Tantalum Tubing*, ~~1/16-in. (1.6-mm) outside diameter, 0.0225-in. (0.57-mm) inside diameter.~~ , 1.6-mm outside diameter, 0.57-mm inside diameter.

NOTE 2—Nickel tubing may be substituted for tantalum.

5.7 *Monel® Sampling Tube*, ~~⅜ by 5.5-in. (9.5 by 140-mm) long (volume 5.4 cm~~ 9.5 by 140-mm long (volume 5.4 cm^3).[7]

5.8 *Electronic Integrator*, or computer integration package.

5.9 *TFE-Fluorocarbon Lined Flex Tubing*, ~~¼ in. (6.35 mm).~~ , 6.35 mm.

5.10 *TFE-Fluorocarbon Tubing*, ~~¼ in. by 10 ft (6.35 mm by 3.05 m).~~ , 6.35 mm by 3.05 m.

5.11 *Cajon® VCR Fitting*. [8]

5.12 *Two-Valves*, ~~⅜ in. (9.5 mm), Monel®.[10]~~ , 9.5 mm, Monel®.[7]

5.13 *Four-Valves*, ~~¼-in. (6.35-mm) tubing to ¼-in. pipe, Monel®.[10]~~ , 6.35-mm tubing to 6.35-mm pipe, Monel®.[7]

5.14 *Hoke® Sample Cylinder*, 1000 cm^3, Monel, nickel, tantalum, or stainless steel.[9]

5.15 *Pressure Gage*, ~~200 lb (91 kg), Monel®.[10]~~ , 91 kg, Monel®.[7]

5.16 *Four-Pipe Tee*, ~~¼ in. (6.35 mm), Monel®.[10]~~ , 6.35 mm, Monel®.[7]

5.17 *Vacuum Source*, suitable for chlorine disposal.

6. Reagents

6.1 *Gas Standard*, 500 ~~ppm~~µg/g H_2, 400 ~~ppm~~µg/g O_2, 800 ~~ppm~~µg/g N_2, 50 ~~ppm~~µg/g CO, and 1000 ~~ppm~~µg/g CO_2 in argon.[10]

6.2 *Argon Carrier Gas*, chromatographic grade.

7. Hazards

7.1 *Safety Precautions*:

7.1.1 Chlorine is a corrosive and toxic material. A well-ventilated fume hood should be used to house all sample handling and to vent the test equipment when this product is analyzed in the laboratory.

7.1.2 The analysis should be attempted only by individuals who are thoroughly familiar with the handling of chlorine, and even an experienced person should not work alone. The operator must be provided with adequate eye protection and respirator. Splashes of liquid chlorine destroy clothing and will produce irritations and burns if such clothing is next to the skin.

7.1.3 Do not allow the sample cylinder to become liquid full if liquid samples are to be taken in cylinders. In accordance with 49 CFR 173.304, 173.314, and 173.315, a good rule is that the weight of the chlorine in the cylinder should not be more than 125 % of the weight of the water that the cylinder could contain.

7.1.4 When sampling and working with chlorine out of doors, people downwind from such an operation should be warned of the possible release of chlorine vapors.

7.1.5 In the event that chlorine is inhaled, first aid should be summoned immediately and oxygen administered without delay.

7.1.6 Store pressurized samples where involuntary release would not cause excessive risk to people or property.

7.1.7 It is recommended that means be available for the disposal of excess chlorine in an environmentally safe and acceptable manner. A chlorine absorption system should be provided if the chlorine cannot be disposed of in a chlorine consuming process. When the analysis and sampling regimen requires an initial purging of chlorine from a container, the purged chlorine should be handled similarly. Purging to the atmosphere should be avoided.

8. Sampling

8.1 Assemble the sampling apparatus as shown in Fig. 2, and purge the system with argon before going into the field to sample.

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[5] ~~Shimadzu Scientific Instruments, Inc., Columbia, MD.~~
[5] Porapak® materials, or their equivalent, have been found satisfactory for this purpose.
[6] ~~Carle Instruments, Inc., Fullerton, CA, or equivalent.~~
[6] Shimalite® materials, or their equivalent, have been found satisfactory for this purpose.
[7] ~~Valco Instruments Co., or equivalent.~~
[7] Monel® material, or its equivalent, has been found satisfactory for this purpose.
[8] ~~Porapak® materials,~~Cajon® fittings, or their equivalent, have been found satisfactory for this purpose.
[9] ~~Shimalite® materials,~~Hoke® sample cylinders, or their equivalent, have been found satisfactory for this purpose.
[10] ~~Monel® material, or its equivalent, has been found satisfactory for this purpose.~~
[10] This reagent is used for calibration only.

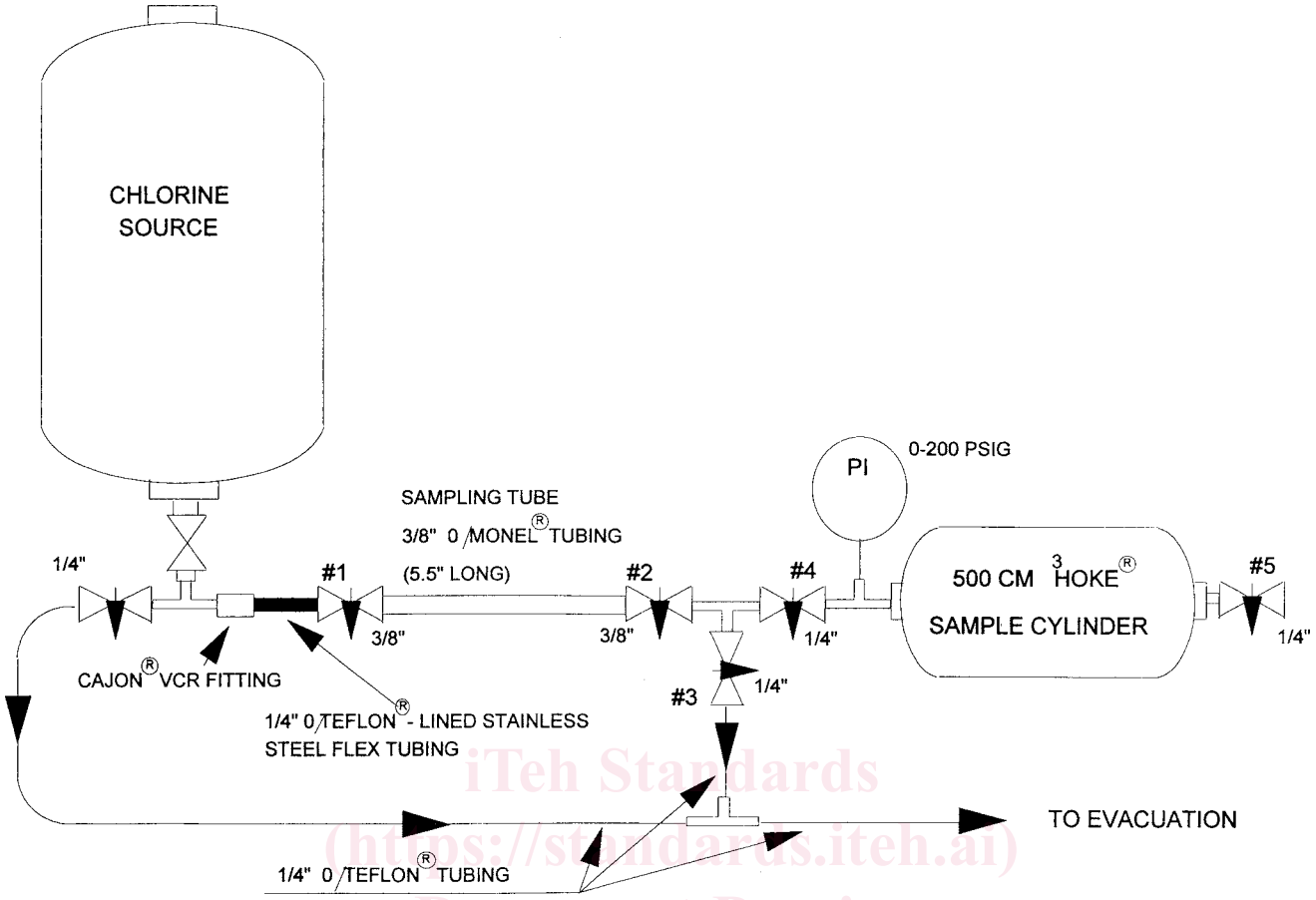

FIG. 2 Chlorine Sampling Apparatus

8.2 Attach the sampling apparatus to the source of liquid chlorine to be sampled and the vacuum source.

8.3 Open all valves on the sample apparatus except Valve No. 5 on the sample bomb end opposite the gage. Evacuate the system using the vacuum source.

8.4 Close all of the valves in the system. Leave the apparatus attached to the vacuum system with the vacuum system on.

8.5 Open the valve on the source of liquid chlorine.

8.6 The following describes the cleanout of the sampling tube made from the ~~-in. (9.5-mm)~~9.5-mm Monel® tubing:

8.6.1 Open Valve No. 3 from the sample bomb to the vacuum source and leave open.

8.6.2 Open Valve No. 1 on the end of the sampling tube connected to the chlorine source for approximately 15 s.

8.6.3 Close Valve No. 1.

8.6.4 Slowly open Valve No. 2 on the end of the sampling tube that is connected to the sample bomb, and vent the chlorine trapped in the sampling tube into the vacuum system.

8.6.5 Close Valve No. 2.

8.7 Repeat 8.6-8.10 two more times so that the sampling tube has been filled and emptied a total of three times.

8.8 Close Valve No. 3 between the vacuum source and sample bomb, and open Valve No. 4 on the gage end of the sample bomb.

8.9 Open Valve No. 1 on the end of the sampling tube connected to the chlorine source for approximately 15 s.

8.10 Close Valve No. 1 and open Valve No. 2 slowly.

8.11 Slowly open Valve No. 3 between the sample cylinder and the vacuum source.

8.12 Close Valves No. 2 and No. 3.

8.13 Repeat 8.11-8.15 three more times. On the fourth time purging the sample cylinder, do not open Valve No. 3, which connects the sample bomb connections to the vacuum source, but close Valve No. 4 on the gage end of the sample bomb.

8.14 Close the valve on the source of the liquid chlorine.

8.15 Evacuate all lines that might contain liquid chlorine by opening all valves except those on the sample bomb and liquid chlorine source. Check the pressure on the sample bomb to ensure that it is below the vapor pressure of liquid chlorine at room temperature. This ensures that only vapor chlorine is present in the sample bomb.

8.16 Disconnect the sample bomb from the sampling apparatus and the sampling apparatus from the source of the chlorine. The pressure in the sample bomb should be below ~~120 lb (54 kg)~~54 kg to contain only vapor in the bomb.

8.17 This chlorine sample is now ready for analysis by the following method.