



Designation: E 1746 – 95

Standard Test Method for Sampling and Analysis of Liquid Chlorine for Gaseous Impurities¹

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.

1. Scope

1.1 This test method covers sampling and analysis of liquid chlorine for the determination of oxygen (200 to 400 ppm), nitrogen (400 to 800 ppm), 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 cm³ gas sample and argon carrier gas is 100 to 200 ppm.² The detection limit for the other components is significantly lower.

1.2 *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.*

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 Chlorine⁴

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 cm³) 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 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

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

5.3 *Valve Sequencer and Actuator*, for switching valve control.⁶

5.4 *Switching Valves*.

5.4.1 *Ten-Port Switching and Sampling Valve* (stainless steel is acceptable).⁷

5.4.2 *Four-Port Switching Valve* (stainless steel is acceptable).⁶

5.5 *Chromatographic Columns*, 1/8-in. (3.2-mm) outside diameter, 316 stainless, as follows:

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.

Current edition approved Sept. 10, 1995. Published November 1995.

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.

⁵ Shimadzu Scientific Instruments, Inc., Columbia, MD.

⁶ Carle Instruments, Inc., Fullerton, CA, or equivalent.

⁷ Valco Instruments Co., or equivalent.

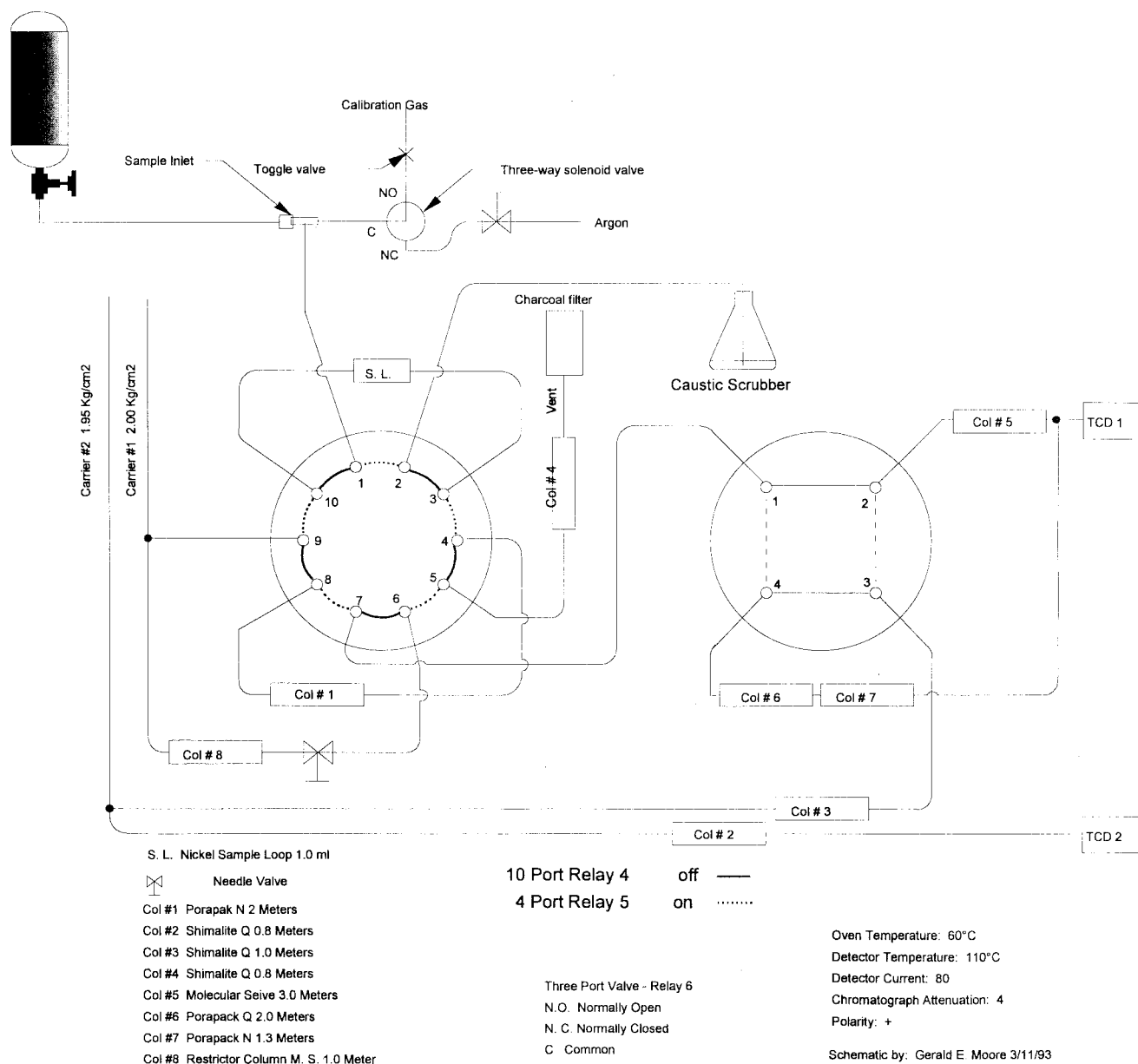


FIG. 1 Chlorine Impurity Analysis System Flow Diagram

5.5.1 2 m of 80/100 mesh Porapak® N,⁸
 5.5.2 0.8 m of 80/100 mesh Shimalite® Q,⁹
 5.5.3 1 m of 80/100 mesh Shimalite® Q,⁹
 5.5.4 0.8 m of 80/100 mesh Shimalite® Q,⁹
 5.5.5 3 m of 45/60 mesh molecular sieve 5A,
 5.5.6 2 m of 80/100 mesh Porapak® Q,⁸
 5.5.7 2 m of 80/100 mesh Porapak® N,⁸ 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.

NOTE 2—Nickel tubing may be substituted for tantalum.

⁸ Porapak® materials, or their equivalent, have been found satisfactory for this purpose.

⁹ Shimalite® materials, or their equivalent, have been found satisfactory for this purpose.

5.7 Monel® Sampling Tube, 3/8 by 5.5-in. (9.5 by 140-mm) long (volume 5.4 cm³).¹⁰

5.8 Electronic Integrator, or computer integration package.

5.9 TFE-Fluorocarbon Lined Flex Tubing, 1/4 in. (6.35 mm).

5.10 TFE-Fluorocarbon Tubing, 1/4 in. by 10 ft (6.35 mm by 3.05 m).

5.11 Cajon® VCR Fitting.¹¹

5.12 Two-Valves, 3/8 in. (9.5 mm), Monel®.¹⁰

5.13 Four-Valves, 1/4-in. (6.35-mm) tubing to 1/4-in. pipe, Monel®.¹⁰

5.14 Hoke® Sample Cylinder, 1000 cm³, stainless steel,

¹⁰ Monel® material, or its equivalent, has been found satisfactory for this purpose.

¹¹ Cajon® fittings, or their equivalent, have been found satisfactory for this purpose.