

Standard Test Method for Moisture and Residue in Liquid Chlorine¹

This standard is issued under the fixed designation E 410; 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 the determination of moisture and residue in liquid chlorine. Lower limits of detection of 10 ppm (w/w) based on a 150-mL sample are achievable.

1.2 This test method describes operations and special apparatus for sampling liquid chlorine from cylinders. Sampling other commercial facilities such as tank cars, barges, and storage tanks require special techniques of manipulation in filling sample cylinders for testing. If conditions and facilities are favorable, the analysis apparatus may be connected directly to these larger storage and transportation units and on-the-spot tests may be completed.

1.3 The analytical methods are empirical and are gravimetric. They are applicable to the determination of the residue and moisture content of commercial grades of liquid chlorine (condensed gaseous product).

1.4 Residue is defined as being those substances which remain in the sample flask after sample volatilization under the conditions of the test. Moisture is defined as the volatile substances evolved during volatilization and purging of the sample-residue flask and absorbed on the desiccant contained in the absorption tubes under the conditions of the test. Some of the volatile chlorinated hydrocarbons may also be evolved from the sample residue flask during purge and absorbed, thus contributing to the apparent moisture analysis value.

1.5 This test method may be used for determining the moisture content only of gaseous chlorine if suitable and appropriate modifications are made for sample measurement and calculation.

1.6 Review the current material safety data sheet (MSDS) for detailed information concerning toxicity, first aid procedures, and safety precautions.

1.7 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

1.8 *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 ASTM Standards:

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals²

2.2 Federal Standards:³

49 CFR 173 Code of Federal Regulations Title 49 Transportation; Shippers' General Requirements for Shipments and Packagings, including 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

3. Summary of Test Method

3.1 A common liquid sample of chlorine is recommended to provide the best results when determining both residue and moisture simultaneously.

3.2 Liquid product sample is transferred from the same cylinder into a precalibrated, 250-mL sample-residue flask and permitted to volatilize completely under prevailing environmental conditions. The volatilized product (chlorine gas) is passed through anhydrous magnesium perchlorate desiccant for absorption of moisture. It is recommended that the exhaust gas be discharged into a caustic scrubber containing an appropriate amount of 20 % caustic solution to neutralize all the chlorine. This reaction is exothermic and care should be taken to avoid excess heating by choosing a sufficiently large volume of caustic solution to serve as a heat sink.

3.3 After complete volatilization of the sample from the flask, residual chlorine and moisture are expelled from any residue by purging with dry air through the absorption system where moisture is recovered and determined gravimetrically.

3.4 Residue remaining in the flask is determined by careful analytical weighing, the weight gain calculated as residue content of the sample. Moisture is computed from the sum of the gain in weight of the two absorption tubes containing the desiccant. Any gain in weight of the second absorber tube

¹ This test method is under the jurisdiction of ASTM Committee E-15 on Industrial Chemicals and is the direct responsibility of Subcommittee E15.57 on Compressed Gases.

Current edition approved March 10, 1998. Published June 1998. Originally published as E 410 – 70. Last previous edition E 410 – 92.

² *Annual Book of ASTM Standards*, Vol 15.05.

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

should be nominal with respect to that of the first tube in the series.

3.5 The moisture content of a chlorine gas stream is determined gravimetrically as above, by passing a metered sample through the moisture absorption system only.

4. Significance and Use

4.1 This test method provides for the determination of moisture and residue in liquid chlorine. The concentration levels of these impurities are important factors in many commercial uses of liquid chlorine.

5. Apparatus

5.1 The arrangement and assembly of apparatus for collection and measurement of the analytical sample and volatilization is shown in Fig. 1.

5.2 *Sample-Residue Flask (H)*, Erlenmeyer-type, 250-mL capacity, equipped with a 35/20 \mathcal{J} ground-ball joint.⁴ A permanent calibration mark shall be inscribed on the flask at the 150-mL volume level for sample measurement.

5.3 *Adapter (G)*, equipped with a 35/20 \mathcal{J} ground-socket joint attached and sealed as shown in Fig. 1. In addition, appropriate 12/5 \mathcal{J} joints shall be sealed to the adapter. A ball joint, sealed and closed off at the end, shall be provided for attachment to the inlet tube after collection of liquid sample in the sample-residue flask. This shall be held in position with an appropriate clamp during volatilization, then removed and replaced with appropriate connections from the metered dry-air supply at the time of aeration and purge of the sample-residue flask.

5.4 A second assembly consisting of a sample-residue flask and adapter shall be used to facilitate collection and disposal of

⁴ The recommended sample-residue flask, equipped with a 35/20 \mathcal{J} ground-ball joint and the adapter equipped with a 35/20 \mathcal{J} ground-socket joint and associated 12/5 \mathcal{J} ground joints shown in Fig. 1, will in all likelihood require fabrication by a glass blower. Alternatively, stock apparatus from Ace Glass Company, Inc., Vineland, NJ, modified slightly and possessing reversed ground neck and adapter joints, has been found satisfactory. Sample-residue flask: Ace stock No. 6975. Adapter: Ace stock No. 5196, modified by reversing the 90° bend of the top inlet tube and attaching 12/5 \mathcal{J} ground joints.

sample purge from the cylinder sample line prior to withdrawal of the analytical sample for test.

5.5 *Regulator*—An appropriate needle valve type shall be attached to the cylinder outlet to control and regulate the flow of liquid chlorine from the sample cylinder. Connection (F) between the outlet from this valve and the adapter (G) shall be by a short length of appropriately sized TFE-fluorocarbon tubing equipped with a 12/5 \mathcal{J} ground-ball joint.

5.6 *Rubber Tubing*, length and size sufficient to exhaust chlorine vapors to the caustic soda absorber or hood system during sample collection and volatilization. A ground-glass 12/5 \mathcal{J} socket joint shall be fitted to one end of the rubber tubing to make appropriate connection to the adapter during sample collection and transferred to the exhaust connection from the second moisture absorption U-tube for gas discharge to disposal during volatilization of the sample.

5.7 *Watch Glass*, small size, used to close the sample-residue flask during analytical weighing before and after sample volatilization to prevent loss of residue or absorption of moisture from the atmosphere.

5.8 *Moisture Absorption Tubes (I)* (Fig. 1)—Schwartz U-tube design, 100 mm length top of arms to center, modified and equipped with ground-glass 12/5 \mathcal{J} socket and ball joints sealed to the inlets and outlets. Two such absorber tubes shall be connected in a series arrangement.

5.9 *Flow Meter*, capable of metering air at 4.5 L/min and equipped with appropriate ground glass connection.

5.10 *Caustic Scrubber*, for chlorine disposal.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

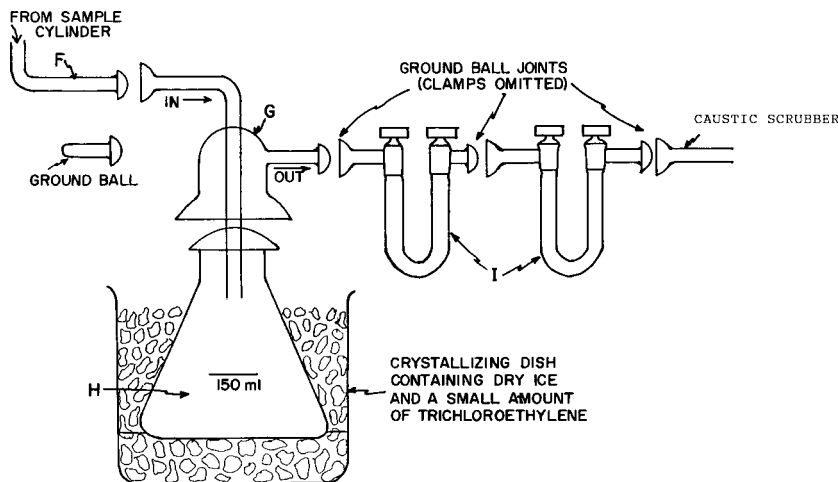


FIG. 1 Apparatus for Collection and Volatilization of Liquid Chlorine

6.2 *Pulverized Dry Ice and a Small Amount of Trichloroethylene*, contained in a glass crystallizing dish (190 by 100 mm), for use in cooling the sample-residue flask during sample purge, collection and measurement.

6.3 *Granular Form of Anhydrous Magnesium Perchlorate*—Reagent Grade.

6.4 *Caustic Solution*—20 % by weight, sodium hydroxide in water. Dissolve 200 g of sodium hydroxide (NaOH) in water and dilute to 1 L. Store in a tight polyethylene container.

7. Hazards

7.1 Chlorine is a corrosive and toxic material. A well ventilated fume hood should be used to house the test equipment when this product is analyzed in the laboratory.

7.2 The analysis should be attempted only by persons 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 if such clothing is next to the skin, will produce irritations and burns.

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

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

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

8. Sampling Tank Cars, Barges, Large Cylinders, and Storage Facilities

8.1 Sampling from tank cars, barges, storage tanks, and large cylinders present unique problems. Each facility, however, must be capable of delivering liquid-sample (not gas) for test.

8.2 Since location of these larger facilities may not be at the immediate site of analysis, sample collection in a suitable secondary container is recommended to facilitate its safe transport to the laboratory for tests.

8.3 It is recommended that samples be collected from these facilities in small-size sample cylinders with cylinders and valves fabricated of tantalum or nickel and capable of being negotiated in the laboratory fume hood. Technical literature available from producers and distributors of liquid chlorine should be consulted for instructions in filling small size cylinders properly from bulk storage.

8.4 Do not allow the sample cylinder to become liquid full. 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 in accordance with 49 CFR 173.

9. Preparation of Apparatus

9.1 Thoroughly clean the absorption tubes, sample-residue

flasks, and adapters free of grease and lubricants, particularly at the ground surfaces of the joints. After thorough cleansing, dry the apparatus thermally in an oven at 105°C. When cooled, charge the absorption tubes with desiccant.

9.2 Charge each tube within about 20 mm of the top in each leg with a granular form of anhydrous magnesium perchlorate, minimizing the use of dusty product. Place loosely packed glass wool on the surfaces of the desiccant in both legs of the absorber tube to avoid carry-over of chemical dust during sample volatilization and air purge operations. Place the unlubricated stoppers firmly in the ground necks of the tubes and adjust to the “off” position preparatory to conditioning with chlorine gas, air purge, and weighing.

9.3 Connect the moisture absorption tubes in a series by means of appropriately sized spring clamps in an upright operating position supported on a ring stand. Attach the ground joint and rubber tubing to the outlet of the second absorber for exhausting chlorine gas to the caustic scrubber.

9.4 Attach a source of chlorine gas to the inlet of the first absorber tube, open the ground stoppers on both tubes to permit the free flow of gas through them and purge for 1 h at a rate of 2 to 3 bubbles of chlorine gas/s when the exhaust end of the rubber tube is held momentarily beneath the surface of water.

9.5 After purging with chlorine (see Section 10), attach the dry air supply to the first absorber in the series and aerate for exactly 5 min at a rate of 4.5 L/min.

9.6 After aeration, carefully close the ground stoppers, dismantle the tubes from the assembly, and condition in the environment of the balance for 10 min prior to analytical weighing of each tube. After weighing, the tubes may be reassembled for the next analysis.

9.7 Conditioning of the absorber tubes with chlorine is required after an initial fresh charge of desiccant only. Aeration is required following volatilization of the sample for each test. If the second tube contains 25 % of the total weight gain, the tubes need to be refilled.

10. Drying Tower and Aeration

10.1 The arrangement and assembly of apparatus used in aerating the equipment after volatilization includes a drying tower, flow meter capable of metering air at 4.5 L/min, and appropriate S ground connection.

10.2 Construct the drying tower for conditioning air used in aeration of sample residue flask and moisture absorption tubes from laboratory materials. Construct the tower of glass tubing 480 mm in length and 50 mm in outside diameter. Close the ends of the tube with tight-fitting rubber stoppers, the bottom stopper having a hole that supports a short, bent connecting glass tube which supplies compressed air to the tower.

10.3 The stopper at the top of the tube shall support a floating-ball flow meter of suitable size and capacity to meter air at a rate of 4.5 L/min. Provide the outlet from the flow meter with a minimum length of rubber tubing and a $12/5$ S ground-ball joint capable of connection to either the adapter of the sample-residue flask or the moisture absorption tubes.

NOTE 1—Use of minimum lengths of connecting rubber tubing reduces

the likelihood of extracting significant amounts of moisture from the tubing itself.

10.4 Introduce compressed air at the bottom of the dryer through a system of proper regulating valves and devices to provide flow at appropriate pressures through the assembly during aeration.

10.5 With the bottom stopper in place and secured, support the drying tower in a vertical position and charge by placing a 40-mm layer of loosely packed glass wool at the bottom, then adding a 190-mm layer of anhydrous magnesium perchlorate desiccant on the top of the glass wool. Place a 20-mm separating layer of glass wool on the first charge of desiccant, then a second 190-mm layer of desiccant and final glass wool packing at the top of the tower.

10.6 Make appropriate register of the flow meter float which delivers 4.5 L of air/min experimentally by the use of a wet-test gas meter connected to the exhaust of the drying tower.

11. Procedure

11.1 Since sampling, volatilization, and aeration are quite closely related, a description of these operations is included in the following procedure.

11.2 Remove the sample-residue flasks and watch glass from the drying oven, cover the neck of one of the flasks (sample) with the watch glass, and permit to cool to room temperature. The second flask need not be covered or weighed since it is used to collect purge and discard liquid chlorine.

11.3 Accurately weigh the sample-residue flask plus the watch glass cover and the two moisture absorption tubes to 0.1 mg. Record the respective weights.

11.4 Attach the sample regulating valve to the sample cylinder and invert the cylinder or situate in a fashion enabling the discharge of liquid chlorine sample (Fig. 1).

11.5 Attach the adapter to the sample-residue flask (purge) and secure the ground joints with a spring clamp. Place the flask in the crystallizing dish and attach the adapter to the liquid discharge from the sample cylinder. Attach the vent tubing to the outlet from the adapter, permitting the exhaust of vaporized chlorine to go to the caustic scrubber.

11.6 Place pulverized dry ice around the sample-residue flask and add about 100 mL of trichloroethylene to reduce the temperature and minimize flashing or volatilization, or both, of the sample during collection. Do not use acetone for accelerated cooling. Permit the assembly to cool a few minutes.

11.7 With the regulator valve on the cylinder closed, cautiously partially open the main control valve on the cylinder. Conduct a final check of the apparatus at this point.

11.8 Cautiously and slowly partially open the regulator needle valve until liquid chlorine flows in a very slow stream into the sample-residue flask. Collect 30 to 50 mL of liquid sample as purge.

11.9 Close the regulator valve that stops the flow of liquid chlorine, and allow the connecting tube between the cylinder and adapter to defrost. Carefully dry this tube with absorbant tissue to prevent entry of any moisture into the delivery tube at the time of disconnection at the ground joint.

11.10 Disconnect the ground joint and very carefully remove the sample-residue flask from the dry-ice packing, disconnect the exhaust tube, and vent the excess chlorine into

the caustic scrubber for disposal.

11.11 Immediately attach the similar sample-residue flask assembly that has been accurately weighed to the sample line, pack in the dry-ice mixture, and collect 150 mL of liquid sample in the manner described for the purge collection. At the conclusion of sample collection, close the main valve at the cylinder; then close the needle regulator valve and permit all liquid to drain into the sample flask. Disconnect the sample line from the adapter at the ground joint, and immediately place and secure the ground glass plug at the adapter joint.

11.12 After sample collection and disconnection of the flask and adapter from the cylinder, open the stopcocks on the absorption tubes to permit free flow of gas. Attach the absorber tubes to the adapter at the outlet port and secure in position by the use of spring clamps for the ground joints.

11.13 Attach the rubber tubing leading to the hood exhaust system to the outlet from the second absorber tube by the ground-joint connection.

11.14 Permit complete volatilization of the sample to proceed at prevailing environmental conditions. This requires 2.5 to 3 h.

11.15 After volatilization, attach the dry air purge to the adapter. Regulate the flow of air to 4.5 L/min and permit to aerate for 5 min.

11.16 Carefully disconnect the absorbers, close the stopcocks, wipe carefully with a lintless tissue, and place near the balance for 10 min prior to accurate weighing. After weighing, these absorbers are ready for future tests as needed.

11.17 Carefully disassemble the sample-residue flask, place the small watch glass on the neck of the flask, wipe clean and dry with a lintless tissue, and condition near the balance for 10 min prior to accurate weighing.

11.18 Weigh and record the gain in weight of the sample-residue flask plus the cover glass. Weigh and record the gain in weight of the moisture absorber tubes.

11.19 Clean the sample-residue flask thoroughly with acetone, water, and scouring powder to remove any waxes. Rinse the flask thoroughly with water and dry thermally in the oven at 105°C to make ready for reuse.

12. Calculation

12.1 Calculate the residue content as follows:

$$\% \text{ residue} = \frac{(A - B) \times 100}{V \times 1.68} \quad (1)$$

where:

- A = weight of flask plus residue plus watch glass cover, g,
- B = tare weight of flask plus watch glass cover, g,
- V = volume of liquid chlorine sample, mL, and
- 1.68 = weight of 1 mL of liquid chlorine at -80°C, g.

12.2 Calculate the moisture content as follows:

$$\% \text{ moisture} = \frac{(N_1 - N_2) + (N_1' - N_2') \times 100}{V \times 1.68} \quad (2)$$

where:

- N_1 and N_1' = weight of moisture absorption U-tubes, 1 and 2, respectively, after sample exposure, g,
- N_2 and N_2' = tare weight of moisture absorption U-tubes, 1 and 2, respectively, before sample exposure, g,
- V = volume of liquid chlorine sample, mL, and
- 1.68 = weight of 1 mL of liquid chlorine at -80°C , g.

13. Report

13.1 Report the percentage of moisture and residue to the nearest 0.001 %.

14. Precision and Bias

14.1 The following criteria should be used for judging the acceptability of the results (see Note 2):

14.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be the percent absolute value in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is the percent absolute value in Table 1.

14.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), Formerly Called Repeatability*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the percent absolute value in Table 1 at the

indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute value in Table 1.

14.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be the percent absolute value in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute value in Table 1.

NOTE 2—These precision estimates are based on an interlaboratory study of analyses performed in 1967-68 on two samples of liquid chlorine contained and circulated in a 50-lb size nickel cylinder. The average moisture and residue content of the first sample was 0.0017 % and 0.0025 %, respectively. The average moisture and residue content of the second sample was 0.0022 % and 0.0061 %, respectively. One analyst in each of five laboratories performed duplicate determinations and repeated one day later.⁶ The cylinder was circulated to each collaborator. At the conclusion of test on the first sample, the cylinder was emptied, cleaned, dried and refilled with liquid chlorine from another source. A similar plan of transmittal and testing was used for the second sample. Practice E 180 was used in developing these precision estimates.

14.2 *Bias*—The bias of this test method has not been determined due to the lack of acceptable reference material.

15. Keywords

15.1 analysis; chlorine; gravimetric; moisture; residue; water

⁶ Supporting data are available from ASTM Headquarters. Request RR: E15-1005.

TABLE 1 Precision for Moisture and Residue, %

	Repeatability			Laboratory Precision			Reproducibility		
	Standard Deviation	Degrees of Freedom	95 % Range	Standard Deviation	Degrees of Freedom	95 % Range	Standard Deviation	Degrees of Freedom	95 % Range
Moisture:	0.000552	20	0.002	0.000413	10	0.001	0.000522	4	0.002
Residue:	0.000715	20	0.002	0.000376	10	0.001	0.000976	4	0.003

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