



Designation: E410 – 08

## Standard Test Method for Moisture and Residue in Liquid Chlorine<sup>1</sup>

This standard is issued under the fixed designation E410; 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 ( $\epsilon$ ) 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  $\mu\text{g/g}$  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. No other units of measurement are included in this standard.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.16 on Industrial and Specialty Product Standards.

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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:<sup>2</sup>

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)<sup>3</sup>

#### 2.2 Federal Standards:<sup>4</sup>

CFR 173 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

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

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

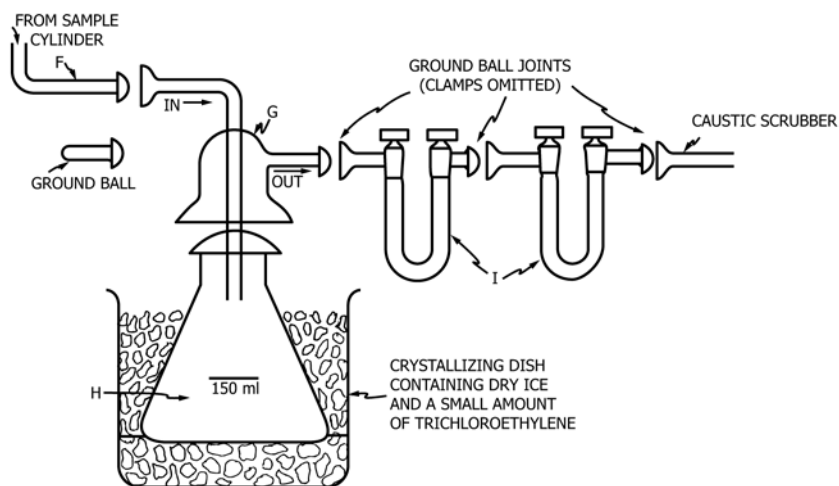


FIG. 1 Apparatus for Collection and Volatilization of Liquid Chlorine

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 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 J ground-ball joint.<sup>5</sup> A permanent calibration mark shall be inscribed on the flask at the 150-mL volume level for sample measurement.

<sup>5</sup> The recommended sample-residue flask, equipped with a 35/20 J ground-ball joint and the adapter equipped with a 35/20 J ground-socket joint and associated 12/5 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 J ground joints.

5.3 *Adapter (G)*, equipped with a 35/20 J ground-socket joint attached and sealed as shown in Fig. 1. In addition, appropriate 12/5 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 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 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 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 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.<sup>6</sup>

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 mass, 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.

<sup>6</sup> *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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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.