



Standard Test Method for Determination of Leachable Chloride in Packing and Gasketing Materials by Ion-Selective Electrode Technique¹

This standard is issued under the fixed designation F1277; 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 provides for the measurement of chloride ion leached from flexible graphite, asbestos, or paper-based packing and gasketing materials. Samples containing 7 to 6700 $\mu\text{g Cl}^-/\text{g}$ sample can be analyzed with this procedure. The upper concentration limit can be extended by dilution of the test solution.

1.2 It is the responsibility of the analyst to ensure the validity of this test method for other packing or gasketing materials.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* A specific warning statement is given in 8.3.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D512 Test Methods for Chloride Ion In Water](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory](#)

3. Summary of Test Method

3.1 Chloride ion leached from packing and gasketing materials is measured potentiometrically using a chloride ion-

selective electrode in conjunction with a double junction, sleeve-type reference electrode. Potentials are read using a pH/mV meter having 0.1 mV readability.

3.2 A calibration curve is prepared by plotting the potentials of known chloride solutions versus the corresponding concentrations expressed in micrograms per millilitre. The chloride concentration of the test solution is determined from the calibration curve, and this value is used to calculate the amount of chloride in parts per million that was leached from the packing or gasketing material. The calibration and test solutions must be measured at the same temperature [within $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$)].

3.3 The calibration and test solutions are diluted with ionic strength adjustor which also minimizes possible interferences such as ammonia, bromide, iodide, cyanide, or sulfide (see Test Methods [D512](#)).

4. Significance and Use

4.1 Chloride ion is highly detrimental to the valves, flanges, piping, etc. of stainless steel and copper-bearing alloys employed in certain types of facilities, such as nuclear and chemical plants. Therefore, the amount of chloride ion that can be leached from packing and gasketing materials used in these facilities must be controlled closely for prevention of damage. This test method is suitable for manufacturing control and for verifying that the packing and gasketing material meets specifications.

4.2 It is assumed that the users of this test method will be analysts capable of performing common laboratory procedures skillfully and safely. It is recommended that the work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide [E882](#).

5. Interferences

5.1 There is no interference from up to 500 $\mu\text{g/mL}$ of sulfide, 1000 $\mu\text{g/mL}$ of bromide or iodide, a one hundred-fold excess of cyanide over chloride, or 1000 $\mu\text{g/mL}$ of ammonia. Ferric, cupric, and permanganate ions do not interfere. Mercury must not be present.

¹ This test method is under the jurisdiction of ASTM Committee [F03](#) on Gaskets and is the direct responsibility of Subcommittee [F03.50](#) on Analytical Test Methods. Current edition approved Oct. 1, 2009. Published March 2010. Originally approved in 1990. Last previous edition approved in 2002 as F1277 – 02. DOI: 10.1520/F1277-02R09.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

6. Apparatus

6.1 *Digital pH/mV Meter*, with 0.1 mV readability preferred, but 1 mV readability is acceptable.

6.2 *Chloride Ion-Selective Electrode (Cl⁻)*, having a silver chloride (AgCl) membrane. (**Warning**—Chloride ion-selective electrodes with a silver chloride/silver sulfide membrane are not suitable as the sulfide can be oxidized by the ionic strength adjustor used in this procedure.)

6.3 *Sleeve-Type Double Junction Reference Electrode*.

NOTE 1—Other types of double junction reference electrodes may be suitable, but supporting data included in this test method were obtained with only the recommended type.

6.4 *Oven*.

6.5 *Desiccator*, containing anhydrous calcium sulfate (CaSO₄).

6.6 *Hot Plate*.

6.7 *Magnetic Stirrer*, with polytetrafluorethylene (PTFE)-coated stirring bars.

6.8 *Beakers*, 150 and 250-mL graduated capacities.

6.9 *Volumetric Flasks*, 100, 500, and 1000-mL capacities.

6.10 *Volumetric Pipets*, with 2, 5, 10, 25, and 50-mL capacities.

6.11 *Graduated Cylinder*, 10-mL capacity.

6.12 *Watch Glasses*, with ribs on the bottoms.

6.13 *Glass Stirring Rods*.

6.14 *Glass Funnels*.

6.15 *Filter Paper*, with fast filtering rate.

6.16 *Polyethylene Bottles*, 150, 500, and 1000-mL capacities.

6.17 *Cotton Gloves*.

6.18 *Scissors or Knife*.

7. Preparation of Reference Materials and Samples

7.1 *Preparation of Calibration Solutions:*

7.1.1 Seven solutions shall be prepared in order to calibrate the chloride ion-selective electrode. Each solution shall contain a different concentration of chloride. Prepare the solutions as follows:

7.1.1.1 *500 µg/mL Cl⁻ Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 10 000 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.2 *100 µg/mL Cl⁻ Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 1000 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.3 *50 µg/mL Cl⁻ Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 1000 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.4 *10 µg/mL Cl⁻ Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 100 µg/mL Cl⁻ stock solution into a

100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.5 *5 µg/mL Cl⁻ Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 100 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.6 *2.5 µg/mL Cl⁻ Solution*—With a 25-mL volumetric pipet, pipet 25 mL of 10 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.7 *1 µg/mL Cl⁻ Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 10 µg/mL Cl⁻ stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly.

7.1.1.8 Transfer each solution to a labeled polyethylene bottle and cap tightly.

7.2 *Preparation of Test and Blank Solutions:*

(**Warning**—When preparing packing or gasketing material samples, precautions should be taken to avoid chloride contamination from handling. Clean cotton gloves should be worn.)

7.2.1 Cut, with scissors or knife, or tear the packing or gasketing material into pieces 6 to 12 mm (¼ to ½ in.) square.

7.2.2 Dry the sample in an oven set at approximately 100°C (212°F) for a minimum of 1 h.

NOTE 2—If it can be demonstrated that equivalent precision can be obtained without drying the samples, this step may be eliminated.

7.2.3 Remove the sample from the oven, place in a desiccator, and cool to room temperature. Weigh 15 g of sample material to the nearest 0.01 g, place in a 250-mL beaker, and label.

7.2.4 Add 90 mL of distilled water to the beaker. Using a 10-mL graduated cylinder, add 10 mL of 0.1 M NaOH.

7.2.5 *Preparation of Blank Solution*—Prepare a blank solution by adding 90 mL of distilled water and 10 mL of 0.1 M NaOH to a 250-mL beaker. Treat the blank solution in the same manner as the test solution, but do not stir.

7.2.6 Cover the beaker with a ribbed watch glass and digest the specimen at 90° ± 5°C (195° ± 10°F) for a minimum of 4 h. Stir the specimen occasionally (at least once per hour). (**Warning**—Use glass rods to stir specimens during digestion. Using a magnetic stirrer may cause the sample material to agglomerate.)

7.2.7 Remove the test solution from the hot plate and cool. Place a filter paper in a glass funnel and moisten with distilled water. Filter the test solution into a 100-mL volumetric flask. Wash the material in the beaker with about 15 mL of distilled water and filter washings into flask. Repeat the washing step if possible, but do not let the volume in the flask exceed the 100-mL mark. Bring the solution in the flask up to 100-mL volume with distilled water and mix thoroughly. (**Warning**—Make sure that the sample is washed sufficiently, but the final volume of the test solution must be restricted to 100 mL.)

8. Reagents and Materials

8.1 *Purity and Concentration of Reagents*—The purity and concentration of the chemical reagents used shall be in accordance with the requirements prescribed in the standards.