



# Standard Test Method for Determination of Leachable Chloride in Packing and Gasketing Materials by Ion-Selective Electrode Technique<sup>1</sup>

This standard is issued under the fixed designation F 1277; 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 provides for the measurement of chloride ion leached from flexible graphite, asbestos, or paper-based packing and gasketing materials. Samples containing 7 to 6700  $\mu\text{g Cl/g}$  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:

D 512 Test Methods for Chloride Ion in Water<sup>2</sup>

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials<sup>3</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>4</sup>

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory<sup>3</sup>

## 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 D 512).

## 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 E 882.

## 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.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

## 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<sup>-</sup>)*, 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 (CaSO<sub>4</sub>).

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<sup>-</sup> Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 10 000 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 1000 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 1000 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 100 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 5-mL volumetric pipet, pipet 5 mL of 100 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 25-mL volumetric pipet, pipet 25 mL of 10 µg/mL Cl<sup>-</sup> 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<sup>-</sup> Solution*—With a 10-mL volumetric pipet, pipet 10 mL of 10 µg/mL Cl<sup>-</sup> 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 Practices E 50.

8.2 *Solutions for Filling Double Junction Reference Electrode:*

8.2.1 *Inner-Chamber Filling Solution*, available from the electrode manufacturer.

8.2.2 *Outer-Chamber Filling Solution*—Use the solution prepared in 8.3.

8.3 *Chloride Ionic Strength Adjustor (CISA)*—Dissolve 15.1 g of sodium bromate ( $\text{NaBrO}_3$ ) in 800 mL of distilled water. Add 75 mL of concentrated nitric acid ( $\text{HNO}_3$ , specific gravity 1.42) and stir well. Dilute with distilled water to 1 L. Store CISA in a polyethylene or glass bottle. (**Warning**—For low level chloride measurements (less than 5 mg/L), the nitric acid used must not contain more than 0.005 % chloride and the sodium bromate must not contain more than 0.003 % chloride.) (**Warning**—Sodium bromate is a strong oxidant and should be handled with care. The preparation and dilution of CISA should be made in a well-ventilated area, preferably in a hood.)

8.4 *10 000  $\mu\text{g/mL}$   $\text{Cl}^-$  Stock Solution*—Place about 20 g of sodium chloride ( $\text{NaCl}$ ) in a weighing bottle and dry in an oven at  $110^\circ\text{C}$  ( $230^\circ\text{F}$ ) for 1 h. Remove weighing bottle from oven, place in a desiccator, and cool to room temperature. As quickly as possible, weigh 16.485 g of the dried  $\text{NaCl}$  and place in a 500-mL beaker. Dissolve the  $\text{NaCl}$  with distilled water. Using a glass funnel, transfer the  $\text{NaCl}$  solution to a 1-L volumetric flask. Wash the beaker with a small quantity (40 to 50 mL) of distilled water and transfer washings to flask. Repeat washing step five times. Bring solution in flask up to the mark with distilled water and mix thoroughly. Store in a labeled 1000-mL polyethylene bottle.

8.5 *1000  $\mu\text{g/mL}$   $\text{Cl}^-$  Stock Solution*—With a 10-mL volumetric pipet, pipet 10 mL of the 10 000  $\mu\text{g/mL}$   $\text{Cl}^-$  stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly. Store in a labeled 150-mL polyethylene bottle.

8.6 *100  $\mu\text{g/mL}$   $\text{Cl}^-$  Stock Solution*—With a 10-mL volumetric pipet, pipet 10 mL of the 1000  $\mu\text{g/mL}$  stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly. Store in a labeled 150-mL polyethylene bottle.

8.7 *10  $\mu\text{g/mL}$   $\text{Cl}^-$  Stock Solution*—With a 10-mL volumetric pipet, pipet 10 mL of the 100  $\mu\text{g/mL}$  stock solution into a 100-mL volumetric flask. Bring the solution in the flask up to the mark with distilled water and mix thoroughly. Store in a labeled 150-mL polyethylene bottle.

8.8 *0.1 M Sodium Hydroxide Stock Solution ( $\text{NaOH}$ )*—Weigh  $4.0 \text{ g} \pm 0.1 \text{ g}$  of  $\text{NaOH}$  and place in a 250-mL beaker. Add 50 mL of distilled water to the beaker slowly while stirring constantly with a glass stirring rod. Stir until  $\text{NaOH}$  is dissolved completely. Using a glass funnel, transfer the  $\text{NaOH}$  solution to a 1-L volumetric flask. Wash beaker with a small quantity (40 to 50 mL) of distilled water and transfer washings to flask. Bring solution in flask up to the mark with distilled water and mix thoroughly. Store in a 1000-mL polyethylene bottle.

## 9. Preparation of Apparatus

9.1 Prepare and operate the pH/mV meter in accordance with the manufacturer's instructions.

NOTE 3—It is not within the scope of an ASTM test method to prescribe minute details relative to the preparation of the apparatus. For a description and specific details concerning the operation of a particular piece of equipment, refer to the manufacturer's manual.

9.2 Use and care for the chloride ion-selective electrode and the reference electrode in accordance with the manufacturer's

instructions (see Note 3). (**Warning**—It is important to keep the flat end of the electrode well-polished, but care should be taken not to damage it.)

9.2.1 Fill the inner and outer chambers of the reference electrode with the recommended solutions (see 8.2.1 and 8.2.2).

## 10. Calibration

10.1 Label seven clean and dry 150-mL beakers with the chloride concentrations of the seven calibration solutions. Add a stirring bar to each beaker, and pipet 25 mL of the appropriate calibration solution into the variously labeled beakers.

10.2 Pipet 25 mL of CISA into each beaker.

10.3 Place the chloride ion-selective electrode and the reference electrode in the calibration solution of lowest chloride concentration (that is, 1  $\mu\text{g/mL}$ ) and turn on the magnetic stirrer. With the pH/mV meter in the mV mode, wait 1 to 2 min and then record the potential observed.

10.4 Remove the electrodes from the solution, rinse with distilled water using a wash bottle, and gently wipe them dry with a soft paper wiper.

10.5 Measure the potentials of the other calibration solutions as described in 10.3 and 10.4. Begin with the 2.5  $\mu\text{g/mL}$   $\text{Cl}^-$  solution and proceed stepwise to the next solution of higher concentration.

NOTE 4—A stable potential reading may be obtained in less than 1 min for calibration solutions with higher chloride concentrations.

10.6 The calibration curve should be linear at chloride concentrations greater than 5  $\mu\text{g/mL}$ , with a slope of  $58 \pm 3$  mV. Estimate the slope of the linear portion of the curve by taking the difference in potential between the 10 and 100  $\mu\text{g/mL}$  calibration solutions. If the slope is not  $58 \pm 3$  mV, this indicates that either the pH/mV meter or one, or both, of the electrodes may be malfunctioning and requires servicing, or that one of the calibration solutions was prepared improperly. If the potential does differ by  $58 \pm 3$  mV, proceed to prepare the calibration curve in accordance with 10.7.

10.7 *Preparation of Calibration Curve*—Using semilogarithmic graph paper, construct a calibration curve by plotting the potential observed for each of the calibration solutions on the linear scale versus the corresponding chloride concentration in micrograms per millilitre ( $\mu\text{g/mL}$ ) on the logarithmic scale. (**Warning**—This is an extremely sensitive test method; therefore, each time samples are analyzed, a set of calibration solutions must be measured and a new calibration curve must be prepared with the values obtained. Since chloride will be oxidized upon prolonged standing, calibration solutions that have been mixed with CISA should be discarded once the analysis has been completed. Fresh mixtures of the calibration solutions and CISA must be prepared each time samples are analyzed.)

## 11. Procedure

11.1 Label a clean and dry 150-mL beaker with the proper sample identification and add a stirring bar. Pipet 25 mL of the test solution into the beaker and, using a 25-mL volumetric pipet, add 25-mL of CISA to the beaker. Treat all the other test solutions and the blank solution in the same manner. Wait 10

**TABLE 1 Precision Data for Determination of Leachable Chloride in Packing and Gasketing Materials<sup>A</sup>**

Material Type	Leachable Chloride Values		Repeatability Data <sup>B</sup> (Within-Laboratory Data)		Reproducibility Data <sup>C</sup> (Between-Laboratory Data)	
	Range, ppm	Average, ppm	$s_r$ , ppm	$r$ , ppm	$s_R$ , ppm	$R$ , ppm
Type 1, Class 2	165.0–286.7	212.98	15.81	44.27	31.36	87.80
Type 5, Class 1	12.3–41.0	22.952	5.501	15.40	8.321	23.30
Type 5, Class 1	9.0–24.7	16.514	4.246	11.89	4.890	13.69
Type 7, Class 2	53.8–118.0	82.981	18.13	50.76	18.13	50.76

<sup>A</sup> These data were obtained in accordance with the 1987 version of Practice E 691.

<sup>B</sup>  $s_r$  = repeatability standard deviation, and  $r$  = repeatability limit, based on a 95 % confidence level (least significant difference between two individual results) =  $2.8 s_r$ .

<sup>C</sup>  $s_R$  = reproducibility standard deviation, and  $R$  = reproducibility limit, based on a 95 % confidence level (least significant difference between two individual results) =  $2.8 s_R$ .

min before proceeding in order to allow the CISA to react sufficiently with the sample material.

11.2 Place the chloride ion-selective electrode and the reference electrode in the blank solution and turn on the magnetic stirrer.

11.3 With the pH/mV meter in the *mV* mode, measure the potential of the blank solution after 1 min. Remove the electrodes from the solution and rinse with distilled water. Dry the electrodes with a soft wiper, and measure the potential of the first test solution. *Measure the blank and test solutions immediately after measuring the calibration solutions.* The electrode potentials are affected by changes in temperature; therefore, the calibration, blank, and test solutions must be within  $\pm 1^\circ\text{C}$  ( $\pm 2^\circ\text{F}$ ) of each other.

## 12. Calculation of Results

12.1 Refer the potential measurements of the blank and test solutions to the chloride calibration curve to obtain the concentrations in micrograms per millilitre ( $\mu\text{g}/\text{mL}$ ).

NOTE 5—Volume corrections are incorporated in the calibration procedure; therefore, test solutions analyzed in accordance with Section 11 can be read directly in micrograms per litre of solution.

12.1.1 If the potential of the blank solution is greater than that of the least concentrated calibration solution, the chloride concentration of the blank will correspond to less than  $1 \mu\text{g Cl}^-/\text{g}$  sample and can be ignored.

12.2 Subtract the chloride concentration obtained for the blank solution from the chloride concentration found for the

test solution. Then calculate the amount of chloride in the original sample material as follows:

$$\frac{\mu\text{g Cl}^-/\text{mL} \times 100 \text{ mL}}{\text{g of sample}} = \frac{\mu\text{g}}{\text{g}} = \text{ppm Cl}^- \quad (1)$$

## 13. Precision and Bias

13.1 *Precision*—The inherent variability for this test method was established by means of an interlaboratory test study conducted in accordance with Practice E 691. Seven laboratories analyzed each of four gasket materials once on three different days. Three different types of gasket material were analyzed: one Type 1, two Type 5, and one Type 7. The precision results obtained are shown in Table 1.<sup>5</sup>

13.2 *Bias*—A statement concerning the bias of this test method cannot be made due to the lack of suitable reference materials and the difficulty of obtaining multiple chemical determinations to establish the "true" chloride values for the gasket materials analyzed in the interlaboratory test study.

## 14. Keywords

14.1 ion-selective electrode technique; leachable chloride; packing and gasketing materials

<sup>5</sup> Supporting data are available from ASTM Headquarters: Request RR F03-1014.

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