



Standard Test Method for Soluble Chlorides in Asbestos¹

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1. Scope

1.1 This test method covers the leaching out of the soluble chlorides in asbestos and the volumetric determination of chloride ion in the leachate.

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.* See Section 9 for specific safety hazards.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

D 2590 Test Method for Sampling Chrysotile Asbestos Fiber³

D 2946 Terminology for Asbestos and Asbestos-Cement Products³

D 3879 Test Method for Sampling Amphibole Asbestos³

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴

2.2 QAMA Standards:

A-2-72 Definitions of Terms Relating to Asbestos⁵

G-5-74 Soluble Chlorides⁵

2.3 ACS Standards:

Specifications of the Committee on Analytical Reagents⁶

3. Terminology

3.1 Refer to Terminology D 2946 and QAMA Standard A-2-72.

4. Summary of Test Method

4.1 The asbestos is leached with water in a Soxhlet extractor for 3 h.

4.2 Dilute mercuric nitrate solution is added to an acidified specimen of leachate in the presence of mixed diphenyl-carbazone bromphenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenyl-carbazone complex (see Note 5).

4.3 An alternative titration by means of an automatic titrator is presented in Annex A1.

4.4 An alternative simplified titration, suitable for non-referee internal quality control, is presented in Annex A2.

5. Significance and Use

5.1 This test method provides an evaluation of the water-soluble chlorides in asbestos. It is used to determine the suitability of asbestos for use in products, such as gaskets, that may be in contact with metals under hydrothermal conditions that foster chloride ion corrosion.

6. Interferences

6.1 Zinc, lead, nickel, ferrous, and chromous ions affect the solubility of the chloride ion and the end point color, but they do not reduce the accuracy of the titration when present in concentrations up to 100 ppm.

6.2 Copper is tolerable up to 50 ppm.

6.3 Titration in the presence of chromate ion requires an indicator with intensified background color, such as alphasurine, and prior reduction for concentrations above 100 ppm.

6.4 Ferric ion above 10 ppm must be reduced before titration, and sulphite ion must be oxidized.

6.5 A part of any bromide and fluoride ion that is present will also be titrated with the chloride ion.

6.6 Quaternary ammonium salts also interfere, if present in significant concentrations (1 to 2 ppm).

6.7 Deep coloration of the leachate may also interfere.

7. Apparatus

7.1 *Microburet*, 1 or 5-cm³ capacity, with 0.01-cm³ graduation intervals (see Note 5).

7.2 *Soxhlet Extraction Apparatus*, including:

7.2.1 Flask, 500 cm³, with a 24/40 glass joint, flask to tube.

7.2.2 *Extraction Tube*, with a 43-mm diameter by 123-mm length thimble, with a 55/50 glass joint, tube to condenser.

¹ This test method is under the jurisdiction of ASTM Committee C17 on Fiber-Reinforced Cement Products and is the direct responsibility of Subcommittee C17.03 on Asbestos-Cement Sheet Products and Accessories.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 04.05.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Published in the *Chrysotile Asbestos Test Manual*. Available from the Asbestos Institute, 1002 Sherbrooke St. W., Suite 1750, Montreal, QC, Canada H3A 3L6.

⁶ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

- 7.2.3 *Condenser*, with a 55/50 glass joint to tube.
 7.2.4 *Porous Thimble Specimen Holders*.
 7.3 *Desiccator*.
 7.4 *Tweezers*.
 7.5 Refer also to A1.1.

8. Reagents and Materials (see Note 5)

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the American Chemical Society specifications. Other grades may be used, provided it is first ascertained that the reagents are of sufficiently high purity to permit their use without decreasing the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification D 1193.

8.2.1 In addition, reagent water shall be free of chloride ion.

8.3 *Bromophenol Blue*, (3', 3'', 5', 5'' tetrabromophenolsulphonphthalein), powder.⁷

8.4 *Diphenylcarbazone* ($C_6H_5NHNHCON: NC_6H_5 \cdot C_6H_5NHNHCONHNHC_6H_5$), crystalline.⁷

8.5 *Ethanol* (CH_3CH_2OH (ethyl alcohol), 95 %.

8.6 *Hydrogen Peroxide* (H_2O_2)—30 % solution.

8.7 *Hydroquinone* ($C_6H_6O_2$)—10 g/dm³.

8.7.1 Dissolve 1 g of purified hydroquinone in water and dilute to 100 cm³.

8.8 *Mercuric Nitrate Solution*—($Hg(NO_3)_2 \cdot H_2O$), 0.025 N.

8.8.1 Acidify 50 cm³ of water with 0.5 cm³ of nitric acid, HNO_3 , sp gr 1.42.

8.8.2 Dissolve 4.2830 g of $Hg(NO_3)_2 \cdot H_2O$ in the acidified water.

8.8.3 Dilute to 1 dm³.

8.8.4 Filter, if necessary.

8.8.5 Standardize against the standard NaCl solution, using the procedure in 12.13.

NOTE 1—The end point, while sharp, can be improved somewhat for certain types of leachates by adding to the titration specimen several drops of a 0.05-g/cm³ solution of xylene cyanole FF or alphasaurine blue-green dye (color index 714). These chemicals can be mixed with the indicator in the same proportions.

8.9 *Mercuric Nitrate Solution*—($Hg(NO_3)_2 \cdot H_2O$), 0.0141 N.

8.9.1 Acidify 25 cm³ of water with 0.25 cm³ of nitric acid, HNO_3 sp gr 1.42.

8.9.2 Weigh out 2.5200 g of $Hg(NO_3)_2 \cdot H_2O$.

8.9.3 Dissolve this $Hg(NO_3)_2 \cdot H_2O$ in the acidified water (8.8.1) and dilute to 1 dm³.

8.9.4 Filter, if necessary.

8.9.5 Standardize against the standard NaCl solution, using the procedure in 12.13 (see Note 2).

8.10 *Mixed Indicator*.

8.10.1 Dissolve 0.5 g of crystalline diphenylcarbazone and 0.05 g of bromophenol blue powder in 75 cm³ of ethanol 95 % (Note 2), and dilute to 100 cm³ with the ethanol.

NOTE 2—Denatured alcohol is not suitable. Methanol or isopropanol

may be used if the 95 % ethanol is not available.

8.10.2 Store the mixed indicator in a brown bottle and discard after six months (Note 3).

NOTE 3—The indicator solution generally deteriorates to the point that it yields no end point color after 12 to 18 months. Temperatures above 38°C (100°F) and exposure to bright light may shorten its useful life. A dry powder mixture of the two indicator ingredients is stable for much longer periods. Both the powder mixture (capsule form) and the liquid indicator are available commercially.

8.11 *Nitric Acid* (HNO_3), sp gr 1.42.

8.12 *Nitric Acid Solution* (HNO_3), (3 + 997).

8.12.1 Dissolve 3 cm³ of HNO_3 sp gr 1.42 in water and dilute to 1 dm³.

8.13 *pH Indicating Paper*, long-range type,⁸ covering a pH range from 1 to 11.

8.14 *Sodium Chloride Standard Solution* (NaCl) (0.025 N).

8.14.1 Dry 1.5 g of NaCl for 1 h at 600°C.

8.14.2 Cool in a desiccator.

8.14.3 Weigh out 1.4613 ± 0.0002 g of the dried NaCl.

8.14.4 Dissolve in water and dilute with water at 20°C to 1 dm³ in a volumetric flask.

NOTE 4—Drying for 2 h at 105°C is adequate for practically all analytical requirements. If ultimate accuracy of standardization is desired, fuse the NaCl prior to cooling it in a desiccator.

8.15 *Sodium Hydroxide Solution* (NaOH), 2 g of NaOH per dm³.

8.15.1 Dissolve 2 g of NaOH in water and dilute to 1 dm³.

8.16 *Xylene Cyanole FF*—($OSO_2(NaOSO_2)(HO)C_6H_2C [C_6H_3(CH_3)NHC_2H_5]$, CAS)—No. 2650-17-1, or alphasaurine blue green dye (color index 714).

8.17 See also A1.2 and A2.1.

9. Hazards

9.1 Most of the reagents in Section 8 are toxic and some are corrosive. Wear protective clothing, gloves, and goggles.

9.2 When handling asbestos avoid creating dust or wear a respiratory protector. Frequent prolonged respiration of excessive concentrations of airborne asbestos may cause serious bodily harm.

10. Sampling, Test Specimens, and Test Units

10.1 *Sampling*:

10.1.1 Sample chrysotile asbestos in accordance with Test Method D 2590.

10.1.2 Sample amphibole asbestos in accordance with Test Method D 3879.

10.2 *Test Specimens*:

10.2.1 The 25-g test specimens shall be derived from the laboratory test sample. Wear gloves to prevent contact of the asbestos with sweat on the skin.

10.2.2 Quarter this sample down to approximately 0.5 kg (1 lb).

10.2.3 Divide the 0.5-kg (1-lb) sample into ten approximately equal portions.

⁷ Eastman Kodak No. 44595 diphenyl-carbazone and No. 752 tetrabromophenolsulonephthalein (bromophenol blue), or their equivalent, have been found satisfactory for this purpose.

⁸ pH Hydrion paper A-B, or its equivalent, has been found satisfactory for this purpose.

- 10.2.4 Subdivide these portions into halves.
- 10.2.5 Combine a half of each of the ten portions and mix thoroughly.
- 10.2.6 Repeat the operation (10.2.4 and 10.2.5) until the sample mass is reduced to approximately 150 g.
- 10.2.7 On the smooth clean surface, spread the 150-g sample into a thin layer over an area of approximately 500 by 500 mm in such a manner that homogeneous pinches may be taken from all parts.
- 10.2.8 Use tweezers to extract twenty bundles of the longer fibers free from rock, grit, dust, or contaminants, each weighing about 2.5 g, and spread these into a thin layer.
- 10.2.9 Use tweezers to extract at least five bundles of the longer fiber from different areas of the thin layer, of such size that when combined and dried to constant mass at 110°C, the test specimen will have a mass of 25 ± 0.2 g.

11. Conditioning

- 11.1 Place each test specimen in a tared porcelain crucible and dry to constant mass at 110°C.
- 11.2 Cool in a desiccator.
- 11.3 Weigh each specimen and crucible to 0.0001 g. This mass minus the tare mass is the specimen mass, S .

12. Procedure

- 12.1 Place the conditioned specimen in the porous extraction thimble.
- 12.2 Place the thimble in the extraction tube.
- 12.3 Add 300 cm³ of water to the extraction flask.
- 12.4 Assemble the Soxhlet extraction apparatus.
- 12.5 Heat the extraction flask to boiling, and continue boiling for 3 h.
- 12.6 Allow the extraction flask to cool.
- 12.7 Pour the leachate into a 500-cm³ volumetric flask.
- 12.8 Rinse out the extraction flask and pour the rinsings into the volumetric flask.
- 12.9 Add water to bring the volume of leachate to the 500-cm³ mark.
- 12.10 Mix thoroughly.
- 12.11 Take an aliquot portion from the 500 cm³ of leachate of such volume that it will not contain more than 20 mg of chloride ion, diluting this aliquot with water to 50 cm³, if necessary.
- 12.11.1 If the volume of the aliquot taken differs from 50 cm³, measure and note this volume, V_3 .
- 12.11.2 If the 500-cm³ volume of leachate contains less than 2.5 mg of chloride ion, make the final titration as described in 12.12, with 0.0141 *N* Hg(NO₃)₂ solution, using a 1 or 5-cm³ microburet (Note 5).
- 12.11.2.1 In this latter case, determine an indicator blank on 50 cm³ of water, applying the same procedure followed for the test specimen.
- 12.11.2.2 If the specimen contains less than 0.1 ppm of chloride ion, concentrate this to an appropriate volume of 50 cm³.

NOTE 5—An automatic titration apparatus may be used to advantage provided that a double junction reference electrode is used. For this alternative procedure, the microburet and reagents required for the end point indicator are not required.

- 12.12 Add 5 to 10 drops of mixed indicator, and shake or swirl the flask. If a blue-violet or red color develops, add HNO₃ (3 + 997) dropwise until the color changes to yellow.
- 12.12.1 Add 1 cm³ excess acid.
- 12.12.2 If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (2 g/dm³) dropwise until the color changes to blue-violet.
- 12.12.2.1 Add NHO₃ (3 + 997) dropwise until the color changes to yellow.
- 12.12.2.2 Add 1 cm³ excess acid (Note 6).

NOTE 6—The prescribed acidification provides a satisfactory pH range from 3.0 to 3.5. Acidified specimens on which electrometric pH measurements have been made shall not be used for chloride determinations because the use of the calomel reference electrode may introduce error due to chloride contamination. Instrumental pH measurements may be made on an aliquot of the specimen, the chloride content determined on the balance of the specimen being corrected accordingly.

- 12.13 Titrate the solution with 0.025 *N* Hg(NO₃)₂ until a blue-violet color, as viewed in transmitted light, persists throughout the solution (Note 7).

- 12.13.1 Record the volume of Hg(NO₃)₂ titrated.
- 12.14 If chromate ion is present in the absence of iron and in concentration less than 100 ppm, use the alphazurine modified mixed indicator (Note 2) and acidify the specimen as described in 12.12 to 12.12.2.2, but to pH 3 as indicated by pH indicating paper.

- 12.14.1 Titrate the leachate as described in 12.13 and 12.13.1, but to an olive-purple end point.

NOTE 7—The use of indicator modifications and the presence of heavy metal ions can change solution colors without affecting accuracy of the determination. For example, solutions containing alphazurine may be bright blue when neutral, greyish purple when basic, blue-green when acidic, and blue-violet at the chloride end-point. When applying this test method to samples that contain colored ions or that require modified indicator, it is recommended that the operator familiarize himself with the specific color changes involved by experimenting with solutions prepared as standards for comparison of color effects.

- 12.15 If chromate ion is present in the absence of iron and in a concentration greater than 100 ppm add two cm³ of fresh hydroquinone solution and proceed as described in 12.13.

- 12.16 If ferric ion is present in the absence or presence of chromate ion, use a sample of such volume as to contain no more than 2.5 mg of ferric ion or of ferric ion plus chromate ion. Add 2 cm³ of fresh hydroquinone solution, and proceed as described in 12.11 to 12.13.

- 12.17 If sulphite ion is present, add 0.5 cm³ of H₂O₂ to 50 cm³ of the sample in the Erlenmeyer flask and mix for 1 min. Then proceed as described in 12.11 to 12.13.

- 12.18 For titration with an automatic titrator, refer to A1.3 and A1.4.

13. Calculation

- 13.1 Calculate the chloride ion concentration, in parts per million (ppm), in the original test specimen as follows:

$$\text{Chloride, ppm} = 35500 N V_3 (V_1 - V_2) / S V_4 \quad (1)$$

where:

- V_1 = volume of standard $\text{Hg}(\text{NO}_3)_2$ solution consumed by the titration, cm^3 ,
 V_2 = volume of standard $\text{Hg}(\text{NO}_3)_2$ solution consumed by the blank titration, cm^3 ,
 V_3 = volume of aliquot in accordance with 12.11.1, cm^3 ,
 V_4 = total volume of leachate, cm^3 ,
 N = normality of the standard $\text{Hg}(\text{NO}_3)_2$ solution, and
 S = mass of specimen, g.

14. Precision and Bias

14.1 Precision:

14.1.1 Repeatability:

14.1.1.1 The single-sample, multiple-operator intralaboratory repeatability, ($2S$) as defined in Practice E 177, is ± 0.1 ppm or $\pm 2\%$ of the chloride ion content, whichever is greater.

14.1.2 Reproducibility:

14.1.2.1 Based upon the QAMA Standard G-5-74, from which this test method was derived, the following statement on interlaboratory reproducibility is quoted directly: "The precision of this method is 0.1 ppm or two percent of the chloride ion content, whichever is greater. The accuracy is approximately equal to precision in the absence of interferences."

14.1.2.2 Refer also to Note 8.

NOTE 8—The repeatability obtained by this test method on concrete

specimens has been reported⁹ as follows for determinations on 69 specimens with an average chloride content of 0.0568 %: "An average absolute error of $\pm 0.0008\%$ characterizes the accuracy level of the proposed test method. The precision, as expressed by the spread (difference between the minimum and maximum readings), for the 69 runs equals 0.013 % chloride. A better measure of the precision, the standard deviation s for the 69 runs amounts to 0.0026 % chloride. A stem-leaf plot and a histogram for the data show a nearly normal distribution of the measurements (a perfect normal distribution is expected only for an infinite number of measurements). According to the laws of statistics for normal distributions, virtually all (99.7 %) of the readings fall at the mean $\pm 3s$, or $0.0568\% \pm 3 \times 0.0026$, that is, between 0.0490 and 0.646 % chloride. All the individual data fall within these limits.

14.2 Bias:

14.2.1 Based upon multiple-sample, multiple-operator intralaboratory trials using asbestos samples spiked with known additions of chloride ion, zero bias was detected by this test method, in the absence of interferences.

15. Keywords

15.1 asbestos; chloride; chloride ion; soluble; soluble chloride ion; test

⁹ Bishava, S. W., "Title No. 88-M32" *American Chemical Institute Materials Journal*, Vol 88, No. 3, May-June 1991, pp. 265-270.

ANNEXES

(Mandatory Information)

A1. ALTERNATIVE TITRATION USING AN AUTOMATIC TITRATOR

A1.1 Apparatus:

A1.1.1 Double Junction Reference Electrode.

A1.1.1.1 The inner unit shall be a single junction reference electrode containing an Ag/AgCl reference element in a glass body with a porous ceramic junction and a fill of 4 M KCl saturated with AgCl.

A1.1.1.2 The glass outer body shall be fitted with a porous ceramic sleeve and cracked-bead junctions. Alternatively, a rugged polymer body with a porous ceramic junction may be used.

A1.2 Reagents and Materials:

A1.2.1 Potassium Chloride Solution (KCl), 4 M, saturated with silver chloride, AgCl.

A1.2.2 Potassium Nitrate Solution (KNO_3), 1.0 M.

A1.2.2.1 Add 10.11 g KNO_3 into a 100- cm^3 volumetric flask and fill to the mark with water.

A1.2.3 Silver Chloride (AgCl), crystals.

A1.3 Preparation of Apparatus:

A1.3.1 The complete electrode unit comes assembled, but dry. The two bodies must be separately filled with the appropriate solutions.

A1.3.2 A snug-fitting O-ring seal in the cap assembly holds the bodies together. To separate them, grasp the lower section

of the cap with one hand and, with the other hand, pull outward on the upper lip. Fill the inner body as follows:

A1.3.2.1 Prepare the filling solution bottle (4 M KCl, saturated with AgCl) by replacing the cap with the supplied dispenser spout. (**Warning**—Never use saturated KCl solution to fill the inner body.)

A1.3.2.2 With the dispensing spout inserted loosely into the fill hole, fill the inner body with the solution to a level of about 6 mm below the fill hole.

A1.3.2.3 With the dispensing spout inserted snugly into the fill hole, tilt the bottle back and squeeze it momentarily to apply air pressure within the inner body. This helps initiate electrolyte flow at the junction.

A1.3.2.4 Reassemble the two bodies, positioning the inner body fill hole 180° away from the outer body fill hole.

A1.3.3 Fill the outer body as follows:

A1.3.3.1 Prepare a filling solution bottle containing the outer body electrolyte (for example, 1.0 M KNO_3) by replacing the cap with a dispenser spout.

A1.3.3.2 With the dispensing spout inserted loosely into the fill hole, fill the outer body with the prepared electrolyte to a level of about 6 mm below the fill hole.

A1.3.3.3 Depending on junction type, initiate electrolyte flow as follows:

(a) *Ceramic or Cracked-Bead Junction*—With dispensing spout inserted snugly into the fill hole, tilt bottle back and squeeze it momentarily to apply air pressure within the outer body.

(b) *Sleeve Junction*—Loosen sleeve by turning, and then firmly seat the sleeve.

A1.4 Procedure:

A1.4.1 Mount the prepared electrode in a suitable holder, and insert the cable pin plug into the pH meter reference or

automatic titrator jack. Observing the following guidelines will produce optimum results with the electrode.

A1.4.1.1 Maintain inner body electrolyte level so that the reference element is always covered. To prevent backflow, inner body electrolyte level should always be several millimetres higher than outer body electrolyte level.

A1.4.1.2 Maintain outer body electrolyte level several millimetres higher than sample solution level to prevent backflow.

A2. ALTERNATIVE TITRATION USING A SIMPLIFIED METHOD (FOR NON-REFEREE TESTING)

A2.1 Reagents:

A2.1.1 *Bromophenol Blue* (3', 3'', 5', 5'' tetrabromophenol-sulphonphthalein), powder,⁸ as 0.2 % solution in ethanol.

A2.1.2 *Diphenylcarbazone* (C₆H₅NHNHCON: NC₆H₅·C₆H₅NHNHCONHNHC₆H₅), crystalline⁸ as 0.2 % solution in ethanol.

A2.1.3 *Mercuric Nitrate*—(Hg(NO₃)₂·H₂O)₃—0.0400 *N* solution.

A2.1.3.1 Dissolve 3.4262 g of Hg(NO₃)₂·H₂O in 500 cm³ of 0.005 *N* HNO₃.

A2.1.3.2 To a 500-cm³ conical flask, add the following:

(a) Add 10 cm³ of the 0.0300 *N* NaCl.

(b) Add 90 cm³ of water.

(c) Add 2.0 cm³ of 0.1 *N* HNO₃.

(d) Add five drops of diphenylcarbazone solution.

A2.1.3.3 Titrate as described in A2.2.

A2.1.4 *Nitric Acid* (HNO₃)—0.05 *N* solution.

A2.1.5 *Nitric Acid* (HNO₃)—0.1 *N* solution.

A2.1.6 *Sodium Chloride* (NaCl)—0.0300 *N* solution.

A2.2 Procedure:

A2.2.1 To the 500 cm³ of cooled leachate, add five drops of bromophenol blue.

A2.2.2 Acidify using 5 cm³ of 0.1 *N* HNO₃.

A2.2.3 Carefully add 0.05 *N* HNO₃ until the yellow color of bromophenol blue appears.

A2.2.4 Add 10 cm³ more of the 0.05 *N* HNO₃ to bring the pH to about 3.0.

A2.2.5 Add five drops of diphenylcarbazone solution.

A2.2.6 Titrate slowly against Hg(NO₃)₂, 0.0400 *N* solution at a rate of five to eight drops per minute. The endpoint is characterized by a color change from very faint yellow to the first appearance of pink; the latter should last more than 60 s.

A2.2.7 In cases where the soluble chloride concentration of the specimen is low, the possibility of not attaining the desired accuracy, because of the small volume of Hg(NO₃)₂ solution consumed by the titration, may be excluded by adding a precisely known aliquot (such as 2 cm³) of the standard NaCl solution to the analyte prior to titration.

A2.2.8 Conduct a blank titration under identical conditions. The blank may be expected to consume 0.5 cm³ of 0.04 *N* Hg(NO₃)₂ solution.

A2.3 Calculation:

A2.3.1 Mass of Soluble Chloride:

$$g = (N_1V_1 - N_2V_2)35.453/1000 \quad (\text{A2.1})$$

where:

N_1 = normality of the Hg(NO₃)₂ solution,

V_1 = titer of the Hg(NO₃)₂ solution, cm³,

N_2 = normality of the NaCl solution, and

V_2 = volume of NaCl solution added, cm³.

A2.3.2 Chloride Ion:

$$\% = (N_1V_1 - N_2V_2)3.5453/W \quad (\text{A2.2})$$

where:

W = mass of specimen, g.

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