



Designation: C323 – 56 (Reapproved 2006)

Standard Test Methods for Chemical Analysis of Ceramic Whiteware Clays¹

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1. Scope

1.1 These test methods cover the chemical analysis of clays used in the manufacture of ceramic whitewares.

1.2 The analytical procedures appear in the following order:

	Section
Moisture	7
Loss on Ignition	8
Silica	9
Iron, Aluminum, and Titanium Oxides	10
Iron Oxide	11
Titania	12
Alumina	13
Lime	14
Magnesia	15
Alkalies	16

NOTE 1—These test methods have been compiled as standard procedures for use in referee analyses. These test methods, however, when the determination of iron oxide as Fe_2O_3 is involved, are not intended to preclude the use of other procedures that give results within the permissible variations. For the sake of uniformity the classical Zimmerman-Reinhardt procedure is specified for the determination of iron oxide. It is recognized that numerous other procedures are equally accurate and often more convenient. The other procedures commonly in use include reduction of an oxidized solution with zinc or other metal, and titration with standard potassium permanganate (KMnO_4) or potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) solution, as well as titration with a standard solution of titanous chloride in an oxidized solution. These procedures shall be considered acceptable, provided the analyst has obtained results by his special procedure that check with the Zimmerman-Reinhardt procedure within the limits specified in Section 17. It is suggested that National Institute of Standards and Technology standard samples be used for checking the accuracy of procedures.

It will be understood that the making of a complete analysis of a ceramic whiteware clay is a difficult procedure requiring a wide knowledge of the chemistry involved in the operations and a thorough training in carrying out the work. A skilled analyst of good training is therefore required to do the work. The descriptions here given cover the vital points of procedure, but frequent reference in regard to the details of the various manipulations should be made to "Applied Inorganic Analysis" by Hillebrand and Lundell² and to similar publications. Particularly in the

¹ These test methods are under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and are the direct responsibility of Subcommittee C 21.04 on Raw Materials.

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² Hillebrand, W. F., and Lundell, G. E. F., *Applied Inorganic Analysis*, Wiley and Son, New York, 1929.

determination of alumina, reference should be made to *Scientific Paper No. 286* of the National Bureau of Standards.³

1.3 The values stated in acceptable metric 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.*

2. Referenced Documents

2.1 *ASTM Standards*:⁴

C322 Practice for Sampling Ceramic Whiteware Clays

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Reagents

3.1 Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Unless otherwise indicated, references to water shall be understood to mean distilled water. Paragraphs 3.1.1-3.1.16 include those reagents common to two or more of the analytical procedures. Other reagents will be found listed with the particular test method in which they are prescribed.

3.1.1 *Concentrated Acids and Ammonium Hydroxide*—Concentrated acids and ammonium hydroxide of approximately the following specific gravities or concentrations will be required:

³ Blum, W., "Determination of Alumina as Oxide," National Bureau of Standards, *Scientific Paper No. 286*.

⁴ 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.

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 Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



Hydrochloric acid (HCl)	1.19 sp gr
Nitric acid (HNO ₃)	1.42 sp gr
Sulfuric acid (H ₂ SO ₄)	1.84 sp gr
Hydrofluoric acid (HF)	40 %
Perchloric acid (HClO ₄)	60 to 70 %, cp ^A
Sulfurous acid (H ₂ SO ₃)	6 % solution ^B
Ammonium hydroxide (NH ₄ OH)	0.90 sp gr

^A Lower purity varieties may contain aluminum oxide, (Al₂O₃), as an impurity.

^B As supplied by reagent manufacturers.

3.1.2 *Diluted Acids and Ammonium Hydroxide*—The diluted acids and ammonium hydroxide referred to are of varying percentages by volume. They shall be made up by mixing proportional volumes of the concentrated reagent and water. The diluted sulfuric acid mixtures shall be made up by slowly stirring the acid into the water. These diluted acids and ammonium hydroxide are designated in the methods as (1 + 4), (1 + 9), and so forth, except very diluted solutions which are referred to by the percent of reagent added. The designation in parentheses indicates the ratio of the volume of the concentrated reagent to the volume of water; for example, H₂SO₄ (1 + 9) contains 10 volume % of H₂SO₄ (sp gr 1.84). The following will be required:

	Volume %
HCl	50
	20
H ₂ SO ₄	50
	10
	5
HNO ₃	30
	3
NH ₄ OH	50

3.1.3 *Ammonium Chloride (2 %)*—Dissolve 2 g of NH₄Cl in 100 mL of water.

3.1.4 *Ammonium Oxalate Solution (Saturated)*—Dissolve 4 g of (NH₄)₂C₂O in 100 mL of water.

3.1.5 *Chloroplatinic Acid Solution (10 %)*.

3.1.6 *Diammonium Phosphate Solution*—Dissolve 10 g of (NH₄)₂HPO₄ in 100 mL of water.

3.1.7 *Ethyl Alcohol (80 %)*—Prepare a solution containing 80 volume % of ethyl alcohol in water.

3.1.8 *Ethyl Alcohol (Absolute)*—Certain commercial brands of denatured absolute alcohol are satisfactory as well as being considerably less expensive than the reagent grade absolute alcohol.

3.1.9 *Hydrogen Peroxide (30 %) (H₂O₂)*.

3.1.10 *Manganese Sulfate Solution*—Dissolve 70 g of crystalline MnSO₄ in 500 mL of water. Add 140 mL of phosphoric acid (H₃PO₄, sp gr 1.7), and 130 mL of sulfuric acid (H₂SO₄, sp gr 1.84). Dilute to 1 L.

3.1.11 *Mercuric Chloride Solution (Saturated)*—Prepare a saturated solution of HgCl₂.

3.1.12 *Potassium Permanganate, Standard Solution (0.1N)*—Dissolve 3.25 g of KMnO₄ in 1000 mL of water. Allow to stand for one week, filter through an asbestos mat, porous glass, or porcelain filter, and keep in a dark place. Standardize against the National Institute of Standards and Technology standard Sample No. 40c of sodium oxalate.

3.1.13 *Potassium Permanganate, Standard Solution (0.04N)*—Dissolve 2.5 g of KMnO₄ in water and make up to 2 L. Allow to stand for one week, filter through an

asbestos mat, porous glass, or porcelain filter, and keep in a dark place. Standardize against the National Institute of Standards and Technology standard Sample No. 40c of sodium oxalate.

3.1.14 *Sodium Arsenite, Standard Solution*—Dissolve 0.908 g of arsenious oxide, (As₂O₃), in a small amount of hot sodium carbonate (Na₂CO₃) solution, cool, filter, and dilute to 1 L. Standardize against a steel of known manganese content.

3.1.15 *Stannous Chloride Solution (50 g/L)*—Dissolve 50 g of SnCl₂ in 100 mL of HCl and dilute to 1000 mL. Keep a few pieces of metallic tin in the bottle.

3.1.16 *Titanium, Standard Solution*—Weigh out 0.05 g of calcined titanium dioxide (TiO₂). Fuse with 10 g of K₂S₂O₇ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity. Cool, and dissolve in about 300 mL of H₂SO₄ (1 + 5). Cool, transfer to a 500-mL volumetric flask, dilute to the mark with water, and mix thoroughly. To standardize the solution, take two 50-mL portions in 400-mL beakers, dilute, boil, and precipitate with NH₄OH. Filter, and wash with hot water. Place the papers in the original beakers, add 15 mL of HCl, stir to macerate the paper, dilute, and precipitate again with NH₄OH. Filter, and wash with hot water until free of alkali salts. Ignite carefully, blast, and weigh. From the weight determined, calculate the strength of the solution.

4. Sampling

4.1 *Selection of Sample*—Obtain the sample in accordance with Practice C322.

4.2 Crush the sample in a small jaw or roll-type crusher with hardened tool-steel faces to pass a 2.36-mm (No. 8) sieve (Note 2). Crush the sample to pass a 850-μm (No. 20) sieve, mix, and quarter to about 50 g. Grind this 50-g sample so that it will all pass a 150-μm (No. 100) sieve, unless otherwise specified, mix thoroughly, and place in a container that will ensure freedom from contamination. Do fine grinding in a suitable mortar (agate, mullite, alumina, or boron carbide) to prevent the introduction of impurities. Take precautions to prevent contamination of the sample by steel particles from the sampling equipment during crushing or grinding.

NOTE 2—Detailed requirements for these sieves are given in Specification E11.

5. Method of Analysis

5.1 Determine moisture on the sample in its ordinary air-dried condition. Determine all other percentage compositions on moisture-free samples and report accordingly on a moisture-free basis. The drying temperature recommended for all moisture determinations is 105 to 110°C. Whenever a sample is weighed out for any determination other than moisture, it shall be moisture-free. If preferred, the sample may be dried in a weighing bottle from which the required samples shall be weighed out.

6. Blank Determinations

6.1 Make blank determinations on the reagents for each constituent in the whiteware clay and deduct this blank in each case. For the determination of the silica (SiO₂) blank, approximately 0.25 g of Al₂O₃ should be added as aluminum chloride.