Designation: C323 – 56 (Reapproved 2016)

Standard Test Methods for Chemical Analysis of Ceramic Whiteware Clays[1]

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 ($K_2Cr_2O_7$) 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 Lundell[2] and to similar publications. Particularly in the 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:

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[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 C21.04 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.

[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_2SO_4)	1.84 sp gr
Hydrofluoric acid (HF)	40 %
Perchloric acid ($HClO_4$)	60 to 70 %, cp[A]
Sulfurous acid (H_2SO_3)	6 % solution[B]
Ammonium hydroxide (NH_4OH)	0.90 sp gr

[A] Lower purity varieties may contain aluminum oxide, (Al_2O_3), 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, H_2SO_4 (1 + 9) contains 10 volume % of H_2SO_4 (sp gr 1.84). The following will be required:

	Volume %
HCl	50
	20
H_2SO_4	50
	10
	5
HNO_3	30
	3
NH_4OH	50

3.1.3 *Ammonium Chloride* (2 %) —Dissolve 2 g of NH_4Cl in 100 mL of water.

3.1.4 *Ammonium Oxalate Solution (Saturated)*—Dissolve 4 g of $(NH_4)_2C_2O$ in 100 mL of water.

3.1.5 *Chloroplatinic Acid Solution* (10 %).

3.1.6 *Diammonium Phosphate Solution*—Dissolve 10 g of $(NH_4)_2HPO_4$ 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 %) (H_2O_2).

3.1.10 *Manganese Sulfate Solution*—Dissolve 70 g of crystalline $MnSO_4$ in 500 mL of water. Add 140 mL of phosphoric acid (H_3PO_4, sp gr 1.7), and 130 mL of sulfuric acid (H_2SO_4, sp gr 1.84). Dilute to 1 L.

3.1.11 *Mercuric Chloride Solution (Saturated)*—Prepare a saturated solution of $HgCl_2$.

3.1.12 *Potassium Permanganate, Standard Solution* (0.1*N*)—Dissolve 3.25 g of $KMnO_4$ 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. 40*c* of sodium oxalate.

3.1.13 *Potassium Permanganate, Standard Solution* (0.04*N*)—Dissolve 2.5 g of $KMnO_4$ 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. 40*c* of sodium oxalate.

3.1.14 *Sodium Arsenite, Standard Solution*— Dissolve 0.908 g of arsenious oxide, (As_2O_3), in a small amount of hot sodium carbonate (Na_2CO_3) 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 $SnCl_2$ in 100 mL of HCl and dilute to 1000 mL. Keep a few pieces of metallic tin in the bottle.

3.1.16 *Titania, Standard Solution*—Weigh out 0.05 g of calcined titanium dioxide (TiO_2). Fuse with 10 g of $K_2S_2O_7$ in a clean platinum crucible, keeping the temperature as low as possible to maintain fluidity. Cool, and dissolve in about 300 mL of H_2SO_4 (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 NH_4OH. 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 NH_4OH. 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 (SiO_2) blank, approximately 0.25 g of Al_2O_3 should be added as aluminum chloride.

7. Moisture

7.1 Weigh 1.00 g of the sample and heat to constant weight at a temperature not under 105 nor over 110°C. Record the loss in weight as moisture.

8. Loss on Ignition

8.1 Weigh 1.000 g of the moisture-free (105 to 110°C) sample and heat to constant weight over a blast lamp, or in an electric muffle furnace, at 900 to 1000°C. Record the loss in weight as the ignition loss.

9. Silica

9.1 Weigh 0.5000 g of the moisture-free (105 to 110°C) sample into a platinum crucible containing about 5 g of powdered anhydrous Na_2CO_3 and mix well with a platinum wire. Cover the mixture with a little more Na_2CO_3. Heat gradually to the full heat of a good burner (1000 to 1100°C) maintained for about 1 h until complete solution is obtained. Place the crucible cover on a triangle, and when the melt has partially cooled, pour it on the lid (Note 3). When cool, place the crucible and lid in a 150-mL beaker, placing the button on a watch glass above the beaker. Add 30 mL of HCl (1 + 1). When solution is complete wash off the crucible and lid with HCl (1 + 4), taking care to remove all SiO_2. Place the button in the solution. Transfer the contents of the beaker to an evaporating dish and evaporate to dryness on a steam bath. Bake for 1 h at 110°C. Add 20 to 30 mL of HCl (1 + 1) and 50 mL of hot water. When all salts have been dissolved, allow to settle for several minutes and then filter through a general-purpose grade acid-washed medium-retention filter paper. Wash the SiO_2 three times by decantation using 20- to 30-mL portions of first hot water, then HCl (1 + 1), then hot water again. Transfer the precipitate to the filter paper, removing all SiO_2 from the dish with a policeman. Wash the paper and precipitate with hot water until free from salt. To recover the small amount of SiO_2 remaining in the filtrate, evaporate to dryness, using the same procedure for baking and filtering as before. Combine the two precipitates, place in a platinum crucible, and burn off the paper carefully to prevent any loss of SiO_2. Ignite the sample to constant weight at 1100 to 1200°C (15 to 20 min is usually sufficient), cool in a desiccator, and weigh. Moisten the residue with several millilitres of water, add 10 mL of HF and three or four drops of H_2SO_4. Evaporate the solution to dryness, ignite carefully to prevent decrepitation, and blast for several minutes at 1100°C. Cool the crucible in a desiccator, weigh, and repeat blasting to constant weight. The loss in weight from the original silica residue represents the SiO_2 content, except for that part of the SiO_2 which is later recovered from alumina, and so forth.

NOTE 3—Another scheme to aid in subsequent solution of the fused melt is to rotate the crucible as it cools, spreading the mass up the side walls.

10. Iron, Aluminum, and Titanium Oxides

10.1 Fuse the residue with 1 g of fused potassium pyrosulfate ($K_2S_2O_7$) or sodium pyrophosphate ($Na_2S_2O_7$), dissolve in a small amount of water, and add to the filtrate from the silica determination (Section 9). Add 5 g of NH_4Cl and three drops of 0.1 % methyl red solution. Heat the solution almost to boiling, and add slowly NH_4OH (1 + 1) until the indicator has changed to a yellow color. Boil for several minutes to remove the excess ammonia. Allow to settle for 30 min and decant through an open, rapid-filtering acid-washed filter paper, transferring the precipitate to the paper and washing the beaker and paper several times with a warm 2 % NH_4Cl solution. Reserve the filtrate, "A," for the determination of calcium oxide (CaO) and magnesium oxide (MgO) (Section 14). Return the precipitate and paper to the original beaker, add 50 mL of hot water and 10 mL of HCl (sp gr 1.19). Stir until the precipitate is dissolved and the paper is well macerated. Dilute to about 200 mL with hot water, precipitate and filter as before. Combine this filtrate "B" with filtrate "A." Wash the paper and precipitate with a warm 2 % NH_4Cl solution. Place the precipitate in a weighed platinum crucible and ignite. Continue the ignition at 1200°C to constant weight (15 to 20 min is usually sufficient). Cool in a desiccator, and weigh with the crucible covered with the lid. The R_2O_3 consists of the aluminum oxide (Al_2O_3), TiO_2, and Fe_2O_3 present in the sample. In addition, there may be small amounts of phosphoric anhydride (P_2O_5), zirconium oxide (ZrO_2), vanadium pentoxide (V_2O_5), and chromic oxide (Cr_2O_3).

11. Iron Oxide

11.1 *Procedure A: Fe_2O_3 Determined on R_2O_3 Sample*—Heat the R_2O_3 precipitate (Note 4) obtained in the determination of iron, aluminum, and titanium oxides (Section 10), with fused $K_2S_2O_7$ or $Na_2S_2O_7$ until solution is complete. Dissolve the fusion in 50 mL of H_2SO_4 (1 + 9) and evaporate to fumes. Cool, dilute with water, and filter off the SiO_2, washing with hot water. Reserve the filtrate for the determination of Fe_2O_3 and TiO_2. Ignite the SiO_2 in a platinum crucible and weigh. Treat the precipitate with 5 mL of HF and two or three drops of H_2SO_4. Evaporate to dryness, ignite, and weigh. The loss in weight represents extra SiO_2 which should be added to that determined previously and also deducted from the weight of the R_2O_3 precipitate. Evaporate the filtrate obtained in correcting the R_2O_3 precipitate for SiO_2 to about 75 mL. Cool, and dilute to 100 mL in a volumetric flask. Reserve 25 mL for the determination of TiO_2 (Section 12). To the remainder, add 25 mL of HCl (1 + 1) and heat to boiling. Reduce the iron by adding $SnCl_2$ solution drop by drop from a pipet with constant swirling of the beaker until the solution is colorless. Then add one drop in excess. Cool quickly in running water, then add at one stroke 15 mL of saturated $HgCl_2$ solution. Allow to stand for 3 min, then transfer with washing to a 1000-mL beaker containing 300 mL of cold distilled water and 25 mL of $MnSO_4$ solution. Titrate with standard 0.04*N* $KMnO_4$ solution, added very slowly while stirring constantly, until a permanent pink end point is obtained.

NOTE 4—Instead of fusing directly in the platinum crucible in which the R_2O_3 was ignited the precipitate may be brushed into a porcelain crucible and then fused with $K_2S_2O_7$ or $Na_2S_2O_7$. This avoids loss of platinum by the action of the pyrosulfate, and no platinum is present in the filtrate to interfere with the iron determination.

11.2 *Procedure B: Fe_2O_3 Determined on a Separate Sample*—Weigh 1.00 g of the finely ground, moisture-free (105 to 110°C) sample into a platinum crucible, add ten drops perchloric acid ($HClO_4$) and 20 mL of HF, and heat almost to dryness on a hot plate. Add 5 to 10 mL of $HClO_4$ and heat until residue has dissolved (Note 5). Cool, place crucible in a 400-mL beaker, add 100 mL of water, and heat to boiling. Any residue present, other than SiO_2, should be filtered off and fused with $K_2S_2O_7$ or $Na_2S_2O_7$ in a porcelain crucible and added to the main solution.

NOTE 5—Decomposition of ceramic whiteware clays and some fired whiteware materials can be effected equally as well by substituting an equal volume of H_2SO_4 (1 + 1) for the $HClO_4$. In this case, heat to fumes once, cool, dilute, filter, and fuse any residue remaining undissolved.

11.3 Determine iron, using one of the approved methods referred to in Note 1 of Section 1.

12. Titania

12.1 Determine TiO_2 colorimetrically by use of a photometer, as follows: Transfer the 25-mL portion reserved for the determination of TiO_2 (Section 11) to a 100-mL volumetric flask. Add 5 mL of H_2SO_4 (1 + 1). Cool to room temperature and dilute to the mark. Transfer exactly half of the solution to another 100-mL volumetric flask. Dilute one of the flasks to the mark with H_2SO_4 (1 + 19). To the other, add 5 mL of 3 % H_2O_2, then dilute nearly to the mark with H_2SO_4 (1 + 19), adjust temperature to 25 ± 1°C, then adjust volume exactly and let stand at least 5 min. Transfer a portion from the first flask to a cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solution to H_2SO_4 (1 + 19), develop color with 5 mL of 3 % H_2O_2, let stand 5 min and read the absorption, using H_2SO_4 (1 + 19) for the zero setting of the potentiometer scale.

NOTE 6—If a spectrophotometer is used, measure the absorption at mean transmission of 420 nm. If a filter photometer is used, use a glass with a maximum transmission in the region of 420 nm.

NOTE 7—As an alternative method, TiO_2 can be determined in the 25-mL portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard TiO_2 solution with several drops of a 30 % H_2O_2 solution. Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a 5 % H_2SO_4 solution for diluting purposes in matching the colors.

13. Alumina

13.1 Subtract the calculated weight of Fe_2O_3 (see 11.2), TiO_2 (Section 12), and SiO_2 (Section 9) from the weight of R_2O_3 (Section 10). The remainder is the weight of Al_2O_3 plus small amounts of the oxides which may include those previously mentioned in Section 10. These are generally considered as Al_2O_3 in reporting the analysis of ceramic whiteware clays.

14. Lime

14.1 Evaporate the combined filtrates reserved (Section 10) for the determination of CaO and MgO to about 200 mL, add 10 to 15 mL of the saturated ammonium oxalate solution and 2 to 3 mL of NH_4OH. Heat for 1 to 2 h, by which time the volume should be about 75 to 100 mL. Allow the precipitated calcium oxalate to settle. Decant through a dense filter paper, taking care to retain the precipitate in the beaker, wash several times with warm water by decantation, and then wash the paper until free from soluble salts. Reserve the filtrate for the MgO determination (Section 15). Return the paper to the beaker containing the precipitate, add 100 mL of H_2SO_4 solution (5 %), warm, and titrate to a faint pink end point with standard 0.04*N* $KMnO_4$ solution. A blank should be previously determined for the effect of the paper.

NOTE 8—For greater accuracy, a double precipitation should be made, in which case, after precipitating the calcium oxalate as described above, decant the liquid and wash the beaker and paper several times with warm water. Dissolve the precipitate on the paper with warm HCl (1 + 4), allowing it to run into the beaker containing the major portion of the calcium oxalate. Wash the paper with hot water. To the solution (about 75 to 100 mL in volume) add several millilitres of saturated $(NH_4)_2C_2O_4$ solution and NH_4OH in slight excess. Heat for 2 h, filter, wash, and titrate as described above.

15. Magnesia

15.1 Evaporate the filtrate from the CaO determination (Section 14) to about 150 to 200 mL and add 2 to 3 g of diammonium phosphate ($(NH_4)_2HPO_4$), stir until dissolved, and then add NH_4OH until alkaline and then 20 mL in excess. Allow the solution to stand overnight. Filter and wash with 5 % NH_4OH. Dissolve the precipitate on the paper with hot HCl (1 + 4), allowing it to run into the beaker containing the precipitate. Wash the paper with hot water. To the solution, which should be not more than 100 mL in volume, add 0.1 to 0.2 g of $(NH_4)_2HPO_4$. Make ammoniacal, and then add a slight excess while stirring constantly until the precipitate is well formed. Then add 10 mL more of NH_4OH and allow to stand overnight or at least for 4 h. Filter through a dense filter paper. Transfer the precipitate to the paper and wash well with 5 % NH_4OH. Place the paper in a weighed platinum or porcelain crucible, burn off the paper at a low temperature (below 900°C), and ignite to constant weight at 1050 to 1100°C (15 to 30 min is sufficient).

16. Alkalies[6]

16.1 Weigh 1.00 g of the moisture-free (105 to 110°C) sample (ground to an impalpable powder) and 1.0 g of NH_4Cl into an agate mortar and mix well. Add 7 to 8 g of calcium carbonate ($CaCO_3$) (Note 9) and again mix intimately. Place a 3.2–mm (⅛ -in.) layer of $CaCO_3$ in the bottom of a platinum crucible, and then add the above mixture, tapping the crucible occasionally to obtain a dense mass. Place a 3.2–mm layer of $CaCO_3$ on the top. Heat the crucible over a low flame until ammonia fumes are no longer given off, then increase the heat so that the bottom half of the crucible is a dull red and maintain this temperature for about 1 h. Cool, fill the crucible three-fourths full of water, and heat until the contents can be taken out and crushed in an agate mortar. Transfer to a platinum or porcelain dish by means of a jet of water. Evaporate to a low volume, decant through a dense paper, and wash the material in the dish several times by decantation with warm water. Transfer to the paper and wash several times with hot water. Acidify with several millilitres of HCl and evaporate to a

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[6] This procedure for the determination of alkali is the J. Lawrence Smith Method.

volume of 150 to 200 mL. Add several millilitres of NH_4OH and sufficient $(NH_4)_2CO_3$ to precipitate the lime, keeping the dish covered with a watch glass. Warm until the precipitate settles out. Filter and wash with warm water. Evaporate the solution to a low volume, then add a small lump of $(NH_4)_2CO_3$ to determine whether practically all calcium has been precipitated. If no precipitate forms, evaporate to dryness, otherwise precipitate and filter as before. Drive off the ammonium salts by heating just short of a dull red. Dissolve the residue in water and add a few millilitres of a saturated solution $(NH_4)_2C_2O_4$ and 1 to 2 mL of NH_4OH to precipitate the last trace of calcium. Heat for 30 to 45 min, filter, and wash with water containing $(NH_4)_2C_2O_4$ (0.1 %). Catch the filtrate in a weighed platinum dish. Add several drops of HCl and evaporate to dryness. Ignite gently as before and weigh as sodium chloride (NaCl) and potassium chloride (KCl).

NOTE 9—Calcium carbonate of the ACS grade "low in alkalies and heavy metals" shall be used.

16.2 The separation of the potassium and sodium must be carried out in an atmosphere free from ammonia fumes. Add to the solution of the combined chlorides in a small porcelain dish, sufficient chloroplatinic acid solution to react with all of the sodium and potassium. The necessary amount to use is readily calculated from the known strength of the platinum solution and the weight of mixed chlorides counted as NaCl. The dilution of the resulting solution should be such that when heated on the steam bath any precipitate that may have formed entirely dissolves. This is to prevent inclusion of mother liquor in a mass of crystals suddenly formed. Evaporate until the solution is just syrupy enough to solidify on cooling. Do not evaporate to dryness, as this will dehydrate the sodium salt and render it less soluble in alcohol. Drench the residue with alcohol (80 %), filter by decantation through a small paper, wash by decantation with more of the alcohol, crushing the crystals with a small pestle or a widened and rounded end of a short glass rod. Reserve the filtrate and washings if sodium is to be determined directly. The residue should be golden yellow. An orange-red color indicates incomplete removal of the sodium salt. It is unnecessary to bring the mass of the precipitate upon the filter. Dry the dish and paper for a few moments to remove adhering alcohol. Dissolve the precipitate on the filter with hot water, catching the solution in a weighed crucible or small dish of platinum. Evaporate to dryness and add the salt that is still in the porcelain dish. If the salt is at all lumpy, redissolve it in water and again evaporate to dryness. Heat for 1 h at 130°C in an air oven (100°C suffices for very small amounts of fine-grained precipitate). It is necessary to cover the receptacle at first because the precipitate is prone to decrepitate. When dry, cool and weigh as K_2PtC_6. Calculate the oxides, as follows:

$$\mathrm{KCl} = \text{wt of } \mathrm{K_2PtCl_6} \times 0.3068 \tag{1}$$

$$\mathrm{K_2O} = \text{wt of } \mathrm{K_2PtCl_6} \times 0.1941$$

$$\mathrm{NaCl} = \text{total chlorides} - \mathrm{KCl}$$

$$\mathrm{Na_2O} = \mathrm{NaCl} \times 0.5303$$

17. Precision and Bias

17.1 *Precision*—In all cases, check determinations shall be made, and the results shall be redetermined if satisfactory checks are not obtained. Results shall be considered satisfactory if the differences between check determinations do not exceed the following limits:

	Permissible Variations Between Check Determinations, max, %
For silica or other constituent amounting to 30 %^{A} or over	0.3
For alumina or other constituent amounting to 10 to 30 %^{A}	0.2
For any other constituent amounting to under 10 %^{A}	0.1

[A] These figures are stated in terms of the whole sample as 100 %.

17.2 *Bias*—The bias of these test methods have not yet been determined.

18. Keywords

18.1 ceramic whiteware clays; chemical analysis