



Standard Test Method for Soluble Sulfate in Ceramic Whiteware Clays (Photometric Method)¹

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1. Scope

1.1 This test method covers the determination of soluble sulfate ions present in water or a filtrate by means of a photometer measuring the turbidity of precipitated barium sulfate. A method of standardizing the photometer for this test method is also given.

1.2 Soluble sulfate ions may be removed from clays or clay-water slurries by leaching with water during mixing and subsequent filter pressing. To remove all the sulfate ions would require an impractical number of washings; therefore, this test method should be considered a control test and not a quantitative analysis for SO₄ ions.

1.3 *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:

C 324 Test Method for Free Moisture in Ceramic Whiteware Clays²

D 1193 Specification for Reagent Water³

2.2 *American Chemical Society Document:*
Specification for Reagent Chemicals⁴

3. Apparatus

3.1 *Balance*, accurate to 0.0001 g.

3.2 *High-Speed Mixer*⁵.

3.3 *Filter Press*, capable of operating at 690 kPa (100 psi).⁶

3.4 *Glass Beakers*, Erlenmeyer Flasks.

3.5 *Transfer Pipets*.

3.6 *Spectrophotometer* and accessories.⁷

3.7 *Measuring Spoon*, to hold approximately 0.2 g of BaCl₂ or THQ Cup.

3.8 Other usual laboratory equipment, including timers, etc.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.⁸ 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type IV Grade reagent water as defined in Specification D 1193.

4.3 Barium Chloride (BaCl₂·H₂O) crystals, 20 to 30 mesh.⁹

4.4 *Salt-Acid Reagent*—may be obtained from the Betz laboratories.¹⁰ However, it can be prepared from: 23.6 ml HCl, 253.0 gm NaCl, 976.4 ml distilled water. This solution should be stored in an automatic pipettor for accurate measurement.

4.5 *Glycerine Reagent*—a mixture of equal volumes of c. p. glycerine and reagent grade water or it may be purchased from a laboratory supply house. This solution should be stored in an automatic pipettor for accurate measurement.

4.6 *Standard Sulfate Solution*—a solution of c. p. sodium sulfate in reagent grade water at a concentration which contains exactly 100 ppm of sulfate ion. This solution may be prepared as required or obtained from the Betz corporation.¹⁰

5. Standardization of Apparatus

5.1 It should be noted that the instrument requires about 30

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² *Annual Book of ASTM Standards*, Vol 15.02.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ Available from American Chemical Society, 1155 16th Street, NW, Washington, DC 20036.

⁵ The Hamilton Beach Mixer, Waring Blender, or similar apparatus have been found satisfactory for this purpose.

⁶ The Baroid No. 300, available from NL Industries, Inc., Houston, TX, has been found satisfactory for this purpose.

⁷ The Bausch and Lomb Spec 20 Photometer, available from Milton-Roy Co., 820 Linden Ave., Rochester, NY 14625; Leitz-Rouy Photometer, available from E. Leitz Inc., Instrument Div., 24 Link Dr., Rockleigh, NJ 07647; Betz Photometer, available from Betz Laboratories, Somerton Rd., Trevose, PA 19047; and Hach Photometer, available from Hach Co., P.O. Box 389, Loveland, CO 80539, have been found satisfactory for this purpose.

⁸ *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.

⁹ Fisher Scientific Catalog No. B-36, available from Fischer Scientific Co., 585 Alpha Drive, Pittsburgh, PA 15238, has been found satisfactory for this purpose.

¹⁰ Betz Laboratories, Somerton Road, Trevose, PA 19047.

min to warm up. Keep a blank cell in the instrument when a sample is not in the holder. For SO_4 determination, the filter wavelength should be set at 550 nm.

5.2 Using the appropriate pipettes, measure 25 ml of the standard 100 ppm solution into a 125 ml Erlenmeyer flask. Add 5 ml of glycerine reagent and 2.5 ml of salt-acid reagent and stir thoroughly.

5.3 Fill a clean transmission cell with the prepared solution. Be sure to wipe off any droplets of water or solution on the outside of the cell since this may interfere with the readings.

5.4 Insert the cell into the cell holder. Put the 550 nm filter in place and adjust the transmission to read 100.0 percent.

5.5 Pour the contents of the cell back into the flask and add 1 THQ cupful of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ crystals. Stir thoroughly, making sure that all crystals are dissolved. Let stand undisturbed for 5 min.

5.6 After the standing time, quickly fill a clean transmission cell with the solution and insert it into the cell holder. Again, be sure that no water or solution droplets are adhering to the outside of the cell. Read percent transmission to the nearest 1 %.

5.7 The standard 100 ppm solution is then made into a series of lower concentration solutions in accordance with the following dilution chart:

ml 100 ppm Sulfate	Solution ml H_2O	ppm Sulfate ion
90	10	90
80	20	80
70	30	70
60	40	60
50	50	50
40	60	40
30	70	30
20	80	20
10	90	10
0	100	0

5.8 Each solution is to be tested as described above and percent transmission recorded. The percent transmission is to be plotted against the ppm sulfate ion concentration and a smooth curve drawn through the points. The resulting chart is to be used in determining the sulfate concentrations of unknown samples of water or filtrate.

6. Procedure

6.1 The clay sample, if in crude lump form, is to be dried at 105°C for 24 h, crushed with the mortar and pestle and pulverized with a micro-pulverizer. If air-floated, continue with 6.2. If slurry continue with 6.4.

6.2 A 150-g portion of the ground dried clay is then weighed to the nearest 0.1 g.

6.3 Blunge the clay in 350 ml of reagent grade water for 30 min at high speed using the Hamilton Beach drink mixer.

6.4 Place a fresh piece of Baroid filter paper in the cylinder of a Baroid Filter Press. Pour the slurry into the cylinder and

tighten the screw on the lid of the cylinder.

6.5 Pressure is applied gradually until 100 psi is obtained. Filtrate is caught in an Erlenmeyer flask. To eliminate errors from initial turbidity, the first 5 ml of filtrate is discarded.

6.6 Prepare the filtrate for testing by carefully transferring the desired quantity of filtrate from the Erlenmeyer flask to a clean 30 ml beaker using the appropriate pipette. A 25 ml total sample is used for testing; this sample may be comprised of between 1 ml and 25 ml of filtrate depending on the sulfate ion concentration in the filtrate. If the selected quantities yield readings beyond the calibration line, further dilution is required. For example, if one initially prepares a sample of 15 ml of filtrate and 10 ml of $\text{DI H}_2\text{O}$ (25 ml total) and obtains a percent transmission reading below the calibration chart, then one should probably use 5 ml of filtrate and 20 ml of $\text{DI H}_2\text{O}$ for the next test. One should use very accurate measuring pipettes when transferring any filtrate or water in this test.

6.7 Add 5 ml of glycerine solution and 2, 5 ml of salt-acid reagent to the solution in the 30 ml beaker.

6.8 Stir thoroughly with a clean glass rod.

6.9 Pour the solution into a transmission cell taking care not to create any air bubbles or spill solution on the side of the cell. (Do not touch the unfrosted side of the cell).

6.10 Adjust the meter to the 100 % transmission mark on the scale by using the calibration knob.

6.11 Pour the contents of the cell back into the beaker and thoroughly rinse and dry the cell. (Do not scratch the cell).

6.12 Add 1 THQ cupful of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ crystals to the solution.

6.13 Stir carefully and make sure all the crystals are dissolved.

6.14 Allow to stand for five min.

6.15 Pour the solution into the transmission cell and insert the cell into the holder. Record the percent transmission.

7. Report

7.1 From the percent transmission readings obtained, use the following method to calculate soluble sulfate ion concentration in ppm.

7.2 Percent transmission is converted to ppm SO_4 of the dilute solution by using the prepared calibration chart.

7.3 The dilution factor is obtained by dividing 17.5 ml by the number of ml of filtrate used.

7.4 The water/solids factor is obtained by dividing the % water by the % solids. For 150-g clay and 350-g H_2O , the factor is 2.33.

7.5 Use the following formula to calculate concentration of soluble sulfate:

$$\text{ppm SO}_4 \text{ clay (dwb)} = (\text{ppm SO}_4 \text{ dilute solution} \times \text{dilution factor}) \times \text{water/solids factor.} \quad (1)$$

C 867

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