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CAUTION:

This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this test method, the user should determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Analysis of sodium silicate

1. Scope

1.1 The sodium silicates in general use in the paper industry consist of chemical combinations of SiO_2 and Na_2O in ratios varying from about 1:1 to about 4:1.

1.2 They are available as aqueous solutions, anhydrous solids, and crystalline hydrates depending upon their chemical composition and the use for which they are intended. These uses will influence the tests which are required. When the utility is based on their chemical nature, the essential determinations are the Na_2O and SiO_2 content; when used as an adhesive, their viscosity and specific gravity become important.

2. Apparatus

2.1 *Stormer viscometer*, with special silicate test cup without central baffle plate and thermometer well.

2.2 *Thermometer*, ASTM Stormer 49c, range 20 to 70°C, graduated in 0.2° divisions, with immersion distance of 65 mm, or other thermometer with similar range and a scale error of less than 0.1°C.

2.3 *Baumé hydrometers* (unless gravimetric method is used), a set of three, 30.5 cm (12 in.) long, graduated in 0.1° Bé from 29.0 to 41.0, from 39.0 to 51.0, and from 49.0 to 61.0 and calibrated against National Bureau of Standards certified instruments.

2.4 *Hydrometer jar* (unless gravimetric method is used), with lip, minimum inside dimensions 5.1 × 30.5 cm (2 × 12 in.), and of such height that during use, the bottom of the hydrometer is not less than 5.1 cm (2 in.) from the bottom of the jar.

2.5 *Volumetric flask* (for gravimetric test), 250 mL.

2.6 *Evaporating dish*, 15.2 cm (6 in.) diameter, porcelain, with smooth unbroken interior glaze. A dish made of Teflon may be preferred.

2.7 *Crucible*, No. 00 porcelain with cover.

2.8 *Platinum crucible*, 25 mL or larger, and lid.

2.9 *Sample jar*, 1.9-L (2-qt) glass, with cover.

2.10 *Other apparatus*: steam bath; stirring rod with flattened end; 400-mL beaker; and filter paper, ash-free, double acid-washed, having extremely fine retention properties for small particulate matter, of a type recommended for use with the finest precipitates; rubber policeman; muffle furnace 925 to 1000°C; stopwatch or stopclock; hotplate.

3. Reagents and materials

3.1 *Standard viscosity mineral oil.* Use standards with calibration values and certificates of standardization traceable to the National Institute of Science and Technology (NIST). Use a standard having a value as close as possible to the sample being tested.

3.2 *Hydrochloric acid,* concentrated and 0.5N HCl.

3.3 *Hydrofluoric acid,* concentrated HF.

3.4 *Sulfuric acid,* 1:1 H₂SO₄.

3.5 *Indicator,* methyl orange (see TAPPI T 610 "Preparation of Indicators and Standard Solutions") or methyl purple, pH 4.8-5.4.

3.6 *Solvents:* perchloroethylene and denatured alcohol or acetone, commercial grade.

4. Sampling

4.1 Any air bubbles present in the sample will cause errors in the specific gravity and viscosity determinations and require removal either by allowing the sample to stand or by centrifuging.

4.2 *Drums:* Agitate thoroughly and withdraw approximately equal portions from 5 to 10% of the drums of each batch number present. Thoroughly mix the samples and fill a moisture-tight 1.9-L (2-qt.) glass jar with the composite.

4.3 *Tank cars*

4.3.1 Take samples at any desired level by lowering through the dome of the car a rod, to which is attached a wide-mouthed bottle or other suitable container closed with a stopper to which a wire is attached. Collect the samples at four different levels: 30.5 cm (1 ft) below the surface, 30.5 cm (1 ft) above the bottom, and at two equally spaced levels representing the second and third fourths of the car. Thoroughly mix the samples and fill a moisture-tight 1.9-L (2-qt) jar with the composite.

4.3.2 Alternatively, sample tank cars during unloading by collecting from the discharge line four samples which are representative of each quarter of the car's contents.

4.4 *Solid materials*

4.4.1 Select 10 bags or packages from different locations in the shipment. Open the package, push aside the first 15 cm (6 in.) of the silicate, and collect a 0.24-L (0.5-pt) sample from each bag at this level, equidistant from the side walls. Place all the samples in a clean, dry, air-tight container of 3.8-L (1-gal) capacity, such as a friction-top can.

4.4.2 For less than carload shipments, sample 5 to 10 bags as directed above.

5. Specific gravity Baumé (liquid silicates)

5.1 *Hydrometer method*

5.1.1 Adjust the temperature of a representative specimen to 20°C (68°F) by placing in a cold water bath and stirring gently with a thermometer. Fill a hydrometer jar with the specimen to within about 2.5 cm (1 in.) of the top, and very slowly lower into the center of it a clean, dry Baumé hydrometer of suitable range and let it come to rest. Do not push the hydrometer down into the silicate so that it rises when released, as such a procedure would probably cause an error in the reading. Read the bottom of the meniscus to the nearest 0.05° Bé.

5.1.2 Wash the hydrometer thoroughly with warm water, rinse with distilled water, and dry with a soft towel.

NOTE 1: If the determination is made at a temperature above 20°C but not higher than 38°C, an approximate value, which is satisfactory for routine determinations, may be obtained by adding 0.036° Bé for each degree Celsius above 20 (0.02° Bé for each degree Fahrenheit above 68).

5.2 *Gravimetric method*

5.2.1 Weigh, to the nearest 0.1 g, 250 mL of the solution in a weighed volumetric flask at 20 ± 0.5°C (68 ± 1°F). Divide the weight of the liquid by the weight of an equal volume of water at the same temperature to obtain the specific gravity.

5.2.2 Convert the calculated specific gravity at 20/20°C (68/68°F) to degrees Baumé.

$$^{\circ} \text{Bé} = 145 - (145/\text{sp gr})$$