WI 220808.06

T 632

BALLOT NO. 3 SARG

DRAFT NO. 02

DATE 06/01/2023

WORKING GROUP
CHAIR Todd Bolton

SUBJECT
CATEGORY Chemical Properties

RELATED
METHODS See "Additional Information"

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 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

(Proposed Reaffirmation of Classical Method T 632 cm-10)

(Underscores, notes, and strikethroughs show changes from Draft 1)

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

Approved by the Standard Specific Interest Group for this Test Method
TAPPI

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. Safety

2.1 Section 7, Silica, uses Hydrofluoric (HF). This is an acutely hazardous substance that requires extreme care when handling. Supplementary laboratory safety training is recommended.

3. Apparatus

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

3.2 *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.

3.3 *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.

3.4 *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.

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

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

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

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

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

3.10 *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.

4. Reagents and materials

4.1 *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.

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

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4.3 *Hydrofluoric acid,* concentrated HF.

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4.4 *Sulfuric acid,* 1:1 H_2SO_4.

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

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

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5. Sampling

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5.1 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.

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5.2 *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.

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5.3 *Tank cars*

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5.3.1 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.

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5.3.2 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.

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5.4 *Solid materials*

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5.4.1 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.

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

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6. Specific gravity Baumé (liquid silicates)

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6.1 *Hydrometer method*

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6.1.1 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.

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

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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).

6.2 *Gravimetric method*

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6.2.1 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.

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

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$$° Bé = 145 - (145/sp\ gr)$$

6.3 *Report*. Report the specific gravity to the nearest 0.1° Bé.

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7. Sodium oxide (alkalinity)

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7.1 Accurately weigh 2 to 3 g of the sample into a tared No. 00 porcelain crucible (or glass-stoppered weighing bottle), using a lid or watch glass as a cover during weighing. Use a fine stream of distilled water from a wash bottle to rinse the entire specimen from the crucible into the evaporating dish, and warm with about 50 mL of water to ensure complete solution.

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7.2 Add 3 to 5 drops of methyl orange or methyl purple indicator and titrate with standard 0.5*N* HCl solution to a reddish orange end point. Reserve the titrated solution for the determination of silica.

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NOTE 2: With most silicates, methyl purple may be used, giving a sharper end point than methyl orange. The color change is from green to purple. Only when the carbonate content of the sample is high is it necessary to use methyl orange.

Calculation: 1 mL 0.5*N* HCl = 0.0155 g Na_2O

7.3 *Report.* Report the percentage of sodium oxide (Na_2O) based on the weight of the specimen to three significant figures.

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8. Silica

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8.1 Add 25 mL of concentrated HCl to the titrated solution reserved from the Na_2O determination, and evaporate to dryness on the steam bath.

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8.2 Take up the residue with 10 mL 1:1 HCl, breaking up any lumps with the stirring rod. Wash down the sides of the dish with about 50 mL of distilled water, allow the mixture to digest on the steam bath for 5 to 10 min to effect solution of soluble salts, and filter through an ashless filter paper into a 400-mL beaker. Wash out the dish with

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hot water to transfer the residue to the filter, using the rubber policeman to dislodge any particles of SiO_2 adhering to the surface of the dish. Wash the residue on filter paper with a little hot water and transfer the combined filtrate and washings from the receiving beaker to the evaporating dish; add 10 mL concentrated HCl, and evaporate to dryness on the steam bath. Transfer the residue to the filter as before and wash on the filter paper with hot water until free from acid, from time to time testing the washings with methyl purple or methyl orange indicator.

8.3 Transfer the paper and the residue to a previously ignited and weighed platinum crucible. Ignite in a muffle until free from carbon, heating slowly at first to char the paper without flaming and gradually increase the temperature to about 1000°C, a bright yellowish red heat. Cover with a platinum lid and heat at this temperature for 10 min. After cooling, moisten the residue in the crucible with 5 or 6 drops of 1:1 H_2SO_4. Evaporate carefully on the hotplate until fumes of H_2SO_4 appear, fume off the H_2SO_4 over a burner, cover the crucible with a platinum lid, and ignite at 1000°C for about 30 min. Cool the crucible and contents in a desiccator and weigh; record the weight of the crucible and SiO_2 plus occluded impurities to the nearest milligram. Moisten the residue in the crucible with about 5 mL of water, add 5 to 6 drops of dilute H_2SO_4 (1:1) cautiously introduce about 10 mL of HF, and evaporate carefully on the hotplate until fumes of SO_3 appear. Add another portion of about 10 mL of HF and evaporate on the hotplate until the SO_3 fumes disappear.

NOTE 2: Alternate techniques exist for the determination of silica using ICP (Induced Coupled Plasma Spectroscopy) that don't require the use of HF.

8.4 Heat the crucible and contents to a bright yellowish red heat (1000°C) for 15 min to reproduce the condition described above for the first ignition. Cool in a desiccator and weigh; record the weight of the crucible and residue. Deduct the weight from that recorded previously to find the difference caused by the volatilization of the SiO_2.

8.5 *Report*. Report the percentage of silica (SiO_2) to three significant figures.

8. Viscosity (liquid silicates)

9.1 *Standardization of viscometer.*

9.1.1 Remove the weight cup from the cord and adjust to a total weight (including the screw cap) of 100 ± 0.1 g, using lead shot; replace the weight cup on the cord. With the water bath platform lowered, place the clean, dry, test cup in position between the fixed prongs of the water bath shell so that the slot in the side of the cup engages with the prong and seats firmly. Attach the cleaned and dried cylindrical rotor to the center shaft of the instrument so that the lock screw engages with the recess in the rotor spindle and fasten securely. Raise the platform along the guide posts until in contact with the stop and fasten it securely in position. The rotor will then extend into the test cup with the top of the cylinder exactly level with the top of the side baffles in the cup. Check that the cylinder rotates freely and is equidistant from the side baffles of the cup.

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9.1.2 Fill the water bath to within about 6 mm (0.25 in.) of the top of the test cup with water at a temperature within about 0.1°C (0.2°F) of that specified for the standard viscosity of oil L. Pour the oil L, having approximately the specified temperature, into the test cup until the top of the cylinder is covered to a depth of about 5 mm (3/16 in.). Pour it in a steady stream to avoid forming air bubbles. Check the temperature of the oil by inserting the thermometer into the oil being tested, holding it there long enough to read the temperature accurately. Make such adjustments as may be necessary to keep the temperature of the oil within ± 0.1°C (± 0.2°F).

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9.1.3 Rotate the drum mounted on the top of the main gear casing so that the weight cord winds without overlapping and until the weight cup is drawn up to within about 50 mm (2 in.) of the pulley cover which the cord passes. Release the brake knob and let the weight fall freely over the edge of the bench or table.

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9.1.4 Observe the indicator hand on the revolution counter and when 10 to 20 revs have been made, starting with a major scale division, time the period required for 100 revs. Record the time to the nearest 0.2 s. Lock the mechanism with the brake and rewind the cord on the drum to raise the weight. Repeat the operations as described until four successive timings check to within 1.0% of the average value. Check the temperature immediately before and after each test with a clean thermometer and adjust if necessary.

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NOTE 3: The standard oils usually show certified viscosities for three different temperatures usually 20, 25°C and 100°F. Thus up to three calibration points may be obtained with the same oil in the cup merely by adjusting the temperature to the specified value.

9.1.5 Detach the rotor from the shaft of the instrument, allowing the cylinder to remain in the test cup. Lower the water bath platform, remove the cup and contents, wipe the outside of the cup dry, lift out the cylinder, and drain the oil into a clean, dry, glass-stoppered bottle, appropriately labeled. This oil may be used for subsequent routine check standardizations between periods of ultimate standardizations. However, an ultimate standardization should have been made with fresh portions of the standard-viscosity oils, not longer than two weeks prior to any test.

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9.1.6 Rinse the test cup and cylinder with perchloroethylene to remove the residual oil completely, flush with hot water, rinse with denatured alcohol (or acetone), and dry the cup and rotor with a blast of compressed air. Wipe the thermometer clean. Assemble the apparatus as directed above and, using the standard-viscosity oils M and N respectively, repeat the described operations. Compute the average time in seconds, to the nearest 0.1 s, from the four timings for each of the three standard-viscosity oils.

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9.1.7 Prepare a calibration curve for the instrument to be used for the subsequent conversion of instrument readings in seconds to viscosity in absolute units. On regular coordinate graph paper, plot the average times obtained for the three standard-viscosity oils as one coordinate against the corresponding certified viscosities of these oils in centipoise as the other coordinate. Draw two straight lines on the graph, one connecting the points obtained for the low and medium viscosity oils, and the other connecting the points obtained for the medium and high viscosity oils. The graph should be of such size and the coordinate scales so divided, that the viscosity coordinates can be read to the nearest centipoise, and the time coordinate to the nearest 0.2 s.

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NOTE 4: The calibration curve, prepared as described above, is independent of temperature, since it is the relationship between absolute viscosity and the time for 100 revs of the viscometer.

9.1.8 Instead of preparing a calibration curve, use the calibration data to compute the equation for the two straight lines expressed as:

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$$V = aT - b \qquad (1)$$

where

V = viscosity, cp
T = time required for 100 revolutions of the cylinder, s
a, b = instrument constants under specified test conditions

8.1.9 Substitute the values for the viscosity and time for 100 revs for the low and medium viscosity oils for V and T, respectively, to obtain two equations which are solved simultaneously for the constants a and b. Use the equation obtained by substituting these values for a and b in Eq. 1 to convert instrument readings in seconds to centipoise within the range covered by the two standard oils used. Obtain the equation for the relationship in the region between the medium and high viscosity oils in a similar manner.

9.2 *Determination of viscosity in absolute units*

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9.2.1 Adjust the temperature of a representative specimen to approximately 20°C (68°F) and immerse the container in the water bath at 20 ± 0.1°C (68 ± 0.2°F). Proceed through the manipulation exactly as described in 8.1, using the specimen in place of standard-viscosity oil. Repeat the operations until three successive timings check to within 1.0% of the average. Remove any silicate scum from the surface of the solution around the rotor spindle before each determination.

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9.2.2 Following the determination, discard the silicate solution, flush the cup and cylinder with hot water until free from silicate, rinse with denatured alcohol (or acetone) and dry the cup and rotor by blowing with compressed air. Wash the thermometer with warm water and wipe dry.

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9.2.3 Compute the average time in seconds, to the nearest 0.1 s, from the three recorded values. Convert this average time to centipoise by means of the calibration curves or the equations.

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9.3 *Report*. Report the viscosity at 20°C (68°F) in centipoise, to the nearest unit.

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10. Keywords

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Sodium silicate, Density, Sodium oxide, Alkalinity, Silica, Viscosity

11 Additional information

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11.1 Effective date of issue: To be assigned.

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11.2 This method, formerly T 632 ts-63, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable.

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11.3 In this 2011 method update, the National Bureau of Standards (NBS) has been deleted as a source of viscosity standards, as they no longer provide them. Viscosity standards used, however, must have calibration values traceable to National Institute of Science and Technology (NIST) primary reference standards.

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Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■