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T 625

BALLOT NO. 04 - SARG

DRAFT NO. 03

DATE May 16, 2024

WORKING GROUP
CHAIR Dr. Sharmiza Adnan

SUBJECT
CATEGORY Chemical Properties

RELATED
METHODS See "Additional Information"

CAUTION:

This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. 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 method, the user must 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 Soda and Sulfate Black Liquor
*(Ten-year review of Classical Method T 625 cm-14:
Reconfirmation of T 625)*
(Changes from Draft 2 shown through strikethroughs/underlines)

1. Scope and significance

This method is for the black or spent liquor separated from the pulp cooked by the soda or sulfate process¹. The black liquor sampled for analysis is usually dilute liquor consisting of the combined liquor from the blow pits, diffusers, and washers.

2. Applicable standards

T 650 "Solids content of black liquor."

¹ Corrections have been made in the calculations for active alkali, NaOH, and CaO

3. Apparatus

- 3.1 *Hydrometer*, Baumé, accurately calibrated, or a pycnometer (specific gravity bottle).
- 3.2 *Evaporating dish*, platinum, 100 to 150 mL.
- 3.3 *Stirrer*, magnetic or electric with alkali-resistant glass or other alkali-resistant stirring rod for a 250-mL and an 800-mL beaker.
- 3.4 *Potentiometer* or pH meter, with a millivolt scale.
- 3.5 *Electrodes*: (a) silver-silver sulfide indicator electrode; (b) a "high pH" glass reference electrode which has been preconditioned by storing for at least 2 weeks in 1.0*N* NaOH (it should be kept in that solution); (c) saturated calomel glass electrode.
- 3.6 *Conductance bridge*, with 0.5-in. (12-mm) platinum disk electrode mounted on alkali-resistant glass rod to permit variable spacing.
- 3.7 *Filtering crucible*, porcelain, with 3-5 μm pores, about 30 mL.
- 3.8 *Buret*, 0 to 50 mL.
- 3.9 *Muffle furnace*, electrically heated with temperature controller to 1200°C.
- 3.10 *Other apparatus*: steam bath; 5 to 6 cm diameter Büchner funnel and filtering flask; fine and medium porosity 5 to 6 cm diameter quantitative filter paper; small filter funnel; oven at 105 and 160°C; beakers, 150, 250, 400, 600, and 800 mL; watch glasses for the 250- and 600-mL beakers; pipettes 10, 20, 25, 50, and 100 mL; volumetric flasks, 100, 250, 500, and 1000 mL; graduated cylinder, 100 mL; Erlenmeyer flasks, 250 and 500 mL; hot plate.

NOTE 1: Unless the black liquor to be analyzed is very dilute, the pipettes used for measuring it should be calibrated for the liquor to be tested, using a fixed drainage time before touching the tip to the side of the beaker.

- 3.11 *For total metals and silica* (excluding sodium, for which see TAPPI T 623 "Sodium Determination by the Uranyl Zinc Acetate Method").
 - 3.11.1 *Kjeldahl flask*, 100 mL.
 - 3.11.2 *Platinum crucible*, about 30 mL, with lid.
 - 3.11.3 *Graduated cylinder*, polyethylene, 0-10 mL.

4. Reagents and materials

CAUTION: This method requires the use of chemicals known to be carcinogens; extreme caution is required in their use and disposal.

4.1 Use reagent grade chemicals and dilution water that has been deionized or distilled and recently boiled to expel oxygen and cooled.

4.2 *Hydrochloric acid*, 0.1N, 0.5N, 1:99 and concentrated HCl.

4.3 *Ammonium hydroxide*, 1:1, 1:99 and concentrated NH_4OH .

4.4 *Sodium hydroxide*, 20% solution NaOH.

4.5 *Silver nitrate*, 0.05 to 0.1N AgNO_3 .

4.6 *Silver nitrate-ammonia*, 2.5% solution of AgNO_3 to which dilute NH_3 has been added until the precipitate just clears.

4.7 *Sulfuric acid*, concentrated, 1:2 (one part of concentrated acid to two of water), and approximately 1.0N H_2SO_4 .

4.8 *Zinc carbonate suspension*. Mix 150 mL each of 1M Na_2CO_3 and 1M ZnSO_4 .

4.9 *Mercuric chloride*, 0.05M HgCl_2 . Accurately weigh 13.58 g HgCl_2 and dilute to 1 L in a volumetric flask.

4.10 *Mercury*, about 20 mL.

4.11 *Formaldehyde*, 40% solution, HCHO.

4.12 *Barium chloride*, 0.5 and 1M BaCl_2 .

4.13 *Acetic acid*, dilute 2:5 CH_3COOH (2 parts of concentrated acid to 5 of water).

4.14 *Nitrogen gas* (cylinder).

4.15 *Glycerin*.

4.16 *Indicators*, methyl orange, phenolphthalein, and methyl red (see TAPPI T 610 "Preparation of Indicators and Standard Solutions").

4.17 For total metals, silica, and sulfur (excluding sodium, for which see T 623).

4.17.1 *Nitric acid*, concentrated HNO_3

4.17.2 *Perchloric acid*, concentrated (70 to 72% by weight) HClO_4 .

4.17.3 *Hydrofluoric acid*, concentrated HF.

4.17.4 *Ammonium chloride*, crystals and 0.1% solution NH_4Cl .

4.17.5 *Ammonium oxalate*, crystals and 1% solution $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$.

4.17.6 *o-Cresolphthalein indicator*, 0.2% dissolved in alcohol.

4.17.7 *8-Hydroxyquinoline* (quinolinol), 5% solution in 2M CH_3COOH .

4.17.8 *Sodium tauroglycocholate surfactant*, optional.

5. Safety

5.1 Caution: this method requires the use of chemicals known to be carcinogens; extreme caution is required in their use and disposal.

5.2 Caution: sections of this standard involve the use of perchloric acid; this chemical can be explosive under certain conditions. Follow the procedures covered. A ducted fume hood designed for perchloric acid is highly recommended.

5.3 Caution: this method involves analysis of black liquor which is discharged at elevated temperature (above 100°C). Proper Personnel Protective Equipment (PPE) such as safety goggles, heat resistant mittens and lab coat are recommended when transferring hot black liquor. Ensure that pressure vessel has been sufficiently depressurized before removing the cap or lid. A mask is recommended for laboratory personnel with respiratory health problems due to unpleasant odor released by the black liquor.

6. Sampling

Take a representative sample and store in a completely filled, alkali-resistant bottle. Black liquor is readily oxidized by atmospheric oxygen and will change composition even while stored in a completely filled and tightly sealed container. Accordingly, perform the analyses as soon as possible after sampling and do not allow the sample

or specimens to be exposed to the air more than necessary. State if more than 24 h have elapsed between sampling and analysis.

7. Procedures

7.1 Density or specific gravity

7.1.1 For maximum accuracy, determine the specific gravity at 20°C by means of a calibrated pycnometer, weighing the bottle empty, also filled with water and with the liquor, both at the same temperature.

7.1.2 For ordinary purposes, determine the Baumé gravity of the sample at 60°F with the hydrometer. Convert to specific gravity, if desired, by the formula:

$$\text{Sp gr} = 145 / (145 - \text{Baumé})$$

7.2 *Total solids.* The evaluation of the solids content of black liquor requires the use of Ottawa or other spherical-high silica sand to prevent the black liquor from skimming over during the drying procedure and thus never truly reaching dryness. For the evaluation of total solids of black liquor, substitute the procedure specified in T 650 "Solids Content of Black Liquor."

7.3 *Sulfated ash and organic matter.* Starting with a cold muffle furnace, or a very low flame, heat the residue from the total solids determination until the organic material is mostly, but not completely, destroyed. Do not heat excessively. Cool, moisten the residue with water, add a drop of methyl orange, and add concentrated sulfuric acid dropwise until the indicator color turns pink. Evaporate to dryness on a steam bath or on a radiator, then heat at a higher temperature to fume off the excess sulfuric acid. Finally, heat at a dull red or slightly higher heat to completely destroy all organic material. Cool to room temperature in a desiccator and weigh. Moisten with water and add a drop of methyl orange. If a pink color develops, ignite again, repeat until the acid is destroyed, cool, and weigh as before. The residue is arbitrarily assumed to be pure sodium sulfate.

7.4 Sodium sulfide (1, 2)

7.4.1 To an 800-mL beaker, add 100 mL of 20% NaOH solution, 35 mL of 1:99 ammonia, and an aliquot of black liquor containing at least 0.2 g of Na₂S. Adjust the beaker contents to 500 mL with water. Titrate immediately with standard AgNO₃, the concentration of which should be 0.1N or less, corresponding to the relative sulfide concentration in the liquor sample. Use sufficient mechanical or magnetic stirring to cause just the beginning of a vortex. Follow the course of the titration potentiometrically, using a silver-silver sulfide indicator electrode and the high pH glass reference electrode. Add the titrant in fixed increments of volume and record the indicated steady emf after each addition. As the end point approaches, there will be an increase in $\Delta E/\Delta V$, that is, there will be an increase in the change of observed emf per increment of titrant. In the immediate vicinity of the end point, a large and sudden change in the observed emf will occur, to the extent of 100 mV or more. When this happens, add two more increments of the titrant and stop the titration.

7.4.2 Plot the data on linear coordinates with milliequivalents of AgNO₃ as the abscissa and emf as the ordinates to obtain the usual S-shaped titration curve. Determine the exact amount of AgNO₃ used at the end point by projecting the inflection point in the curve to the abscissa.

NOTE 2: An alternate, less time-consuming method to locate the end point is to calculate the zero value of $\Delta E^2/\Delta V^2$, fully described elsewhere (3).

7.5 Sodium sulfite and sodium thiosulfate (3)

7.5.1 *Precipitation of sulfide.* Pipette 50 mL of glycerin and 100 mL of the black liquor into a nitrogen-filled, 1000-mL volumetric flask. Allow the pipette to drain for its calibration period for black liquor. Add 300 mL of freshly prepared zinc carbonate suspension. Mix, dilute to the mark, and mix again. Allow the mixture to settle until the bulk of the solids is in the lower part of the flask, then filter the cloudy supernatant liquid by suction through a quantitative filter paper. Discard the first 100 mL of filtrate. Use the remaining filtrate immediately as described in 5.5.2 and 5.5.3 after checking for the complete precipitation with ammoniacal AgNO₃.

7.5.2 *Sulfite plus thiosulfate.* Place about 20 mL (¼ in.) deep of mercury in a 250-mL beaker. With a pipette, add 100 mL of the filtrate and adjust the pH with 2:5 acetic acid to a value between 7.0 and 7.5. Titrate immediately with 0.05M HgCl₂ while stirring the liquor continuously with the electric stirrer. Follow the course of the titration with the potentiometer connected to a saturated calomel reference electrode immersed in the liquor, but not touching the mercury pool itself, and to a platinum wire or electrode immersed in the mercury. Add the titrant in fixed increments of volume, and record the steady emf after each addition. As the end point approaches, there will be an

increase in $\Delta E/\Delta V$. In the immediate vicinity of the end point, a large and sudden change of emf will occur. When this happens, add two or more increments of the titrant and stop the titration. Plot the data on linear coordinates with millimols of HgCl_2 as abscissa and emf as ordinate. Determine the exact amount of HgCl_2 used at the end point by projecting the inflection point in the titration curve to intersect the abscissa. Denote the millimols of HgCl_2 used as A. As an alternative, calculate the zero value of $\Delta E^2/\Delta V^2$ as described by Lingane (3).

7.5.3 Thiosulfate. Proceed with this determination exactly the same as for determination of the sum of sulfite and thiosulfate in 5.5.2 with the following exception: Immediately after adjusting the pH, add 5 mL of 40% formaldehyde, stir, and wait 5 min before proceeding with the titration. Denote the millimols of HgCl_2 used as B.

NOTE 3: The mercury pool need not be renewed for each titration. After each titration decant the aqueous liquid, leaving the mercury in the beaker. Several washings with distilled water will serve to clean the vessel and the mercury, even though the latter retains a yellowish film on its surface. Before beginning a new titration, remove any available aqueous liquid with a pipette.

7.6 *Sodium sulfate.* Pipette 25 mL of black liquor into a 250-mL beaker containing 100 mL of water and sufficient concentrated HCl to provide 5 mL in excess of that required for neutralization of the liquor. Let the pipette drain for its calibration period. Stir the mixture and warm it to about 95°C on a hot plate, stirring occasionally. Cool to about 30°C, and filter into a 400-mL beaker. Wash the solids on the filter with hot water until the 400-mL beaker is about two-thirds full. Add a drop of methyl orange to the filtrate, neutralize with concentrated NH_4OH , and then add 1 mL of concentrated HCl. Heat to boiling, and, while stirring, add quickly 15 mL of hot 1.0M BaCl_2 . Continue stirring for a minute or two, let the mixture stand and cool for about an hour, and then filter with suction through a tared porcelain filtering crucible of fine porosity. Wash the precipitate and the inner wall of the crucible with at least 250 mL of warm water, dry the crucible in an oven at 105°C, and then place it in a furnace at 800°C for one half-hour. Cool the crucible in a desiccator to room temperature and weigh. Reheat for one half-hour, reweigh and repeat until the weight is constant to obtain the weight of barium sulfate.

7.7 *Total alkali.*

7.7.1 This is essentially the conductometric method of McElhinney et al. (4, 5). Dilute 10 mL of black liquor to 100 mL in a volumetric flask. Pipette 20 mL of the diluted liquor into a 250-mL beaker containing 150 mL of water. Immerse the electrodes of the conductance bridge in the solution, stir continuously with a mechanical or a magnetic stirrer and obtain the initial bridge reading. For most black liquors a 1-cm spacing of the electrode disks will be suitable. Add 0.1N HCl in fixed increments of volume and take a reading after each addition, keeping the temperature constant. Continue the titration until the change in the bridge reading remains constant for five or six additions of titrant. Plot the data on linear coordinates with reciprocal ohms as ordinate and milliequivalents of acid as abscissa. The curve obtained will usually resemble a V with the apex flattened or broadly rounded. To locate the end point, extrapolate the arms of the V in a straight line until the extrapolated segments intersect. The point of intersection projected on the abscissa will give the milliequivalents of acid corresponding to the end point.

7.7.2 The titration measures the NaOH, Na_2CO_3 , organic sodium salts, silicates, and Na_2SO_3 (to NaHSO_3). Na_2SO_4 and $\text{Na}_2\text{S}_2\text{O}_3$ are not measured. A value for the total recoverable sodium content of the liquor is obtained from the sum of the total alkali, Na_2SO_4 , $\text{Na}_2\text{S}_2\text{O}_3$, and one-half the Na_2SO_3 .

7.8 *Active alkali (NaOH + Na_2S)* (6, 7). Pipette 100 mL of black liquor into a 500-mL volumetric flask containing about 100 mL of water and allow the pipette to drain for its calibration period. Add 100 mL of 0.5M BaCl_2 , stir, and allow to settle until partially clear. By means of a glass stirring rod, transfer a drop of the clear supernatant liquid to a small test tube containing about 1 mL of 1.0N H_2SO_4 . If no white precipitate is observed, add an additional 20 mL of BaCl_2 to the flask. When there is an excess of BaCl_2 , dilute the contents of the flask to the mark, mix well, and allow to settle until about 200 mL of clear liquid stands above the precipitate. Pipette 100 mL of the supernatant liquid into a 250-mL beaker equipped with a mechanical or magnetic stirrer, add 5 mL of 40% formaldehyde, and titrate immediately with 0.5N HCl to a pH of about 3.5. Follow the course of the titration potentiometrically on the pH range of a pH meter, using the saturated calomel-general purpose glass electrode pair. Add the titrant in increments of fixed volume, record the pH value after each addition, and plot the data on linear coordinates with pH as the ordinate and milliequivalents of HCl as the abscissa. Connect the plotted points in a smooth curve. Obtain the milliequivalents of acid corresponding to the end point by projecting the inflection point near pH 8.3 to the abscissa. From this value subtract the acid equivalent, if any, of the 5 mL of formaldehyde as determined in a separate test, using phenolphthalein indicator. The difference is the milliequivalents of acid consumed by the liquor.

7.9 *Silica, iron, aluminum, sulfur, calcium, magnesium, and sodium* (8)

7.9.1 The following procedure, or a modification described by Bethge (9), may be applied to dried solid or liquid spent liquor. The essence of the method is the destruction and removal of all organic matter and the oxidation of all inorganic material, including sulfur, to its highest valence by digesting it with HNO_3 and HClO_4 . The result of the digestion is a clear solution containing all the inorganic constituents of the original liquor with the exception of silica, which is dehydrated and precipitated.

7.9.2 Pipette 10 mL of the black liquor into a 100-mL Kjeldahl flask, allowing the pipette to drain for its calibration period for the black liquor. Rinse down the neck of the flask with 10 to 15 mL of distilled water. Next add 15 mL of concentrated HNO_3 and 5 mL of concentrated (70%) HClO_4 in that order. **Warning: To prevent an explosion be sure to have the nitric acid present before adding the perchloric acid.**

7.9.3 Place the flask on a steam bath situated in a hood. If the solution being digested suddenly darkens when the main part of the excess HNO_3 has boiled off, stop the digestion immediately and add more HNO_3 (5 mL) before continuing the digestion.

NOTE 4: A sudden darkening almost always indicates insufficient nitric acid and until the worker is familiar with the procedure, an excess of the recommended quantity of nitric acid should be used.

7.9.4 When the oxidation reaction has subsided, as indicated by the absence of red-brown nitrogen dioxide fumes, remove the flask from the steam bath and heat over a burner with low to moderate heat until the oxidation is complete, as evidenced by a colorless solution and dense white perchloric acid fumes appear. Allow the solution to cool to room temperature and add about 50 mL of warm distilled water to dissolve all the salts. If it is desired to determine the silica, it is better to carry out the digestion in a platinum vessel.

7.9.5 Filter off the silica on a small filter paper of medium porosity and wash with 1:99 HCl. Collect the filtrate and washings in a platinum evaporating dish. Evaporate the filtrate and washings to near dryness on a steam bath and add 2 to 3 mL of concentrated HCl. Add enough hot distilled water to dissolve all the salts and again filter off the silica on the filter used previously. Wash the residue thoroughly with cold 1:99 HCl and catch the filtrate and washings in a 250-mL beaker. Remove any silica adhering to the evaporating dish by wiping with a small piece of filter paper, using the stirring rod, and place the bits of paper in the filter. Reserve the filtrate and washings for the determination of iron and aluminum.

7.9.5.1 Place the filter with its bits of paper in a tared platinum crucible with cover. Dry the filter paper and smoke it off at low heat with the cover placed at an angle on the crucible. When the paper has been charred and smoking almost ceases, increase the heat to at least 1000°C and heat at this temperature for one half-hour or more. Place in a desiccator with the crucible lid in place and cool to room temperature. Weigh, remembering that dehydrated silica is hygroscopic, reheat, and weigh again to make sure that it is at constant weight.

7.9.5.2 Carefully moisten the contents of the crucible with about 2 mL of water, adding it from a pipette inserted beneath the cover. Add 4 or 5 drops of 1:2 H_2SO_4 and then add carefully about 5 mL of concentrated HF measured from a polyethylene graduated cylinder. Place the crucible in a hood and heat gently to evaporate off the HF without boiling the liquid. Increase the heat to fume off the H_2SO_4 . When the contents of the crucible are nearly dry, gradually increase the heat to at least 1000°C and bring the crucible to constant weight as before. The loss in weight represents the grams of SiO_2 in the 10-mL specimen.

7.9.6 *Aluminum and iron.* Adjust the volume of the filtrate and washings from the silica determination to about 125 mL and heat to about the boiling point. Add a few drops of methyl red indicator solution, then add concentrated NH_4OH dropwise, with stirring, until the indicator changes from red to yellow. Add 1 drop of NH_4OH in excess and heat the beaker in a steam bath until the precipitate flocks. Filter on a small filter paper of medium porosity and wash the precipitate with 1% NH_4Cl . Collect the filtrate and washings in a 250-mL volumetric flask. Ignite the paper and precipitate in a tared platinum crucible under good oxidizing conditions at over 110°C to constant weight. The final net weight of the residue equals the R_2O_3 content of the 10-mL specimen of liquor ($\text{R}_2\text{O}_3 = \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$).

7.9.7 *Sulfur.* Dilute the filtrate and washings from the R_2O_3 determination to the mark on the 250-mL volumetric flask and mix thoroughly. Pipette a 50-mL aliquot of this solution into a 150-mL beaker. Add concentrated HCl dropwise until the methyl red indicator turns pink, then add 1 mL of the acid in excess. Heat to boiling and rapidly pour 15 mL of hot 1M BaCl_2 into the hot solution while stirring. Continue stirring for a few minutes and let the beaker stand in a warm place for about an hour. Filter off the BaSO_4 in a tared porcelain filter crucible of fine porosity. Wash the precipitate until a test with silver nitrate indicates the absence of chloride ion in the filtrate. Ignite to a final temperature of 800°C, cool and weigh.

7.9.8 *Magnesium (10).* Add 75 mL of concentrated HNO_3 to the combined filtrates and washings from the calcium determination in the 600-mL beaker while partially covered with a watch glass and evaporate to dryness to destroy excess ammonium salts. Add 2 mL of concentrated HCl to the dry salts and enough water to dissolve the salts.

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If a slight cloudiness is detectable in the solution, filter it. Adjust the volume of the filtrate or the solution to about 100 mL and add 2 g of NH_4Cl , 0.5 mL 0.2% in alcohol *o*-cresolphthalein indicator and enough 6N NaOH so that 2-3 mL is present in excess of that required to give a violet color (pH 9.5) to the solution. Heat the solution to about 80°C and add dropwise while stirring the 5% solution of 8-hydroxyquinoline acid until a small excess is present as shown by a yellow color in the supernatant liquid. Digest for about half an hour in the steam bath and filter the hot solution through a tared porcelain filter crucible of fine porosity. Add a trace of sodium tauroglycocholate to the solution to reduce the tendency of the precipitate to stick to the beater. Wash with about 50 mL of warm water and dry the precipitate at 160°C to constant weight, first for 1 h and then for ½-h periods. The precipitate dried at this temperature is anhydrous and has the composition $\text{Mg}(\text{C}_9\text{H}_6\text{ON})_2$.

8. Calculations

Except for the percentages, the following calculations give each constituent in grams per liter:

$$\text{Percentage of total solids} = (\text{residue weight} / \text{sample weight}) \times 100$$

$$\text{Sulfated ash, as NaOH (as percentage of total solids} = [(\text{sulfate ash residue} \times 0.563) / \text{total solids residue}] \times 100$$

$$\text{Percentage of organic matter} = 100 - (\text{sulfated ash as NaOH, \% of total solids})$$

$$\text{Na}_2\text{S} = (\text{meq of AgNO}_3 / \text{mL of liquor sample}) \times 39$$

$$\text{Na}_2\text{SO}_3 = 2 (A - B) (126 / 10)$$

$$\text{Na}_2\text{S}_2\text{O}_3 = (2B \times 158) / 10$$

$$\text{Na}_2\text{SO}_4 = [(\text{g of BaSO}_4 \times 1000) / (233.4 \times 25)] \times 142.1$$

$$\text{Total alkali as Na}_2\text{O} = \text{milliequivalents of acid} \times 15.5$$

$$\text{Active alkali as Na}_2\text{O} = \text{milliequivalents of acid} \times 1.55$$

$$\text{NaOH} = (2 \times \text{meq of acid in active alkali determination}) - (\text{Na}_2\text{S} \times 1.025)$$

$$\text{SiO}_2 = \text{g of SiO}_2 \times 100$$

$$\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = \text{g of R}_2\text{O}_3 \times 100$$

$$\text{Sulfur} = [(\text{g of BaSO}_4 \times 1000) / (233.4 \times 2)] \times 32.1$$

Sodium: Each 2-mL aliquot removed from the 250-mL volumetric flask is equivalent to 0.080 mL of the original black liquor. On this basis, calculate the results according to T 623.

$$\text{Calcium as CaO} = [(\text{g of CaO} \times 1000) / (100.1 \times 7.92)] \times 56.1$$

$$\text{Magnesium as MgO} = [(\text{g of Mg}(\text{C}_9\text{H}_6\text{ON})_2 \times 1000) / (312.6 \times 7.92)] \times 40.3$$

9. Report

Report the various constituents in grams per liter to the nearest third significant figure.

10. Precision

The test procedures outlined in this standard are for use within a pulping operation for the analysis of black

liquor and not designed for interlaboratory comparison of cooking liquor conditions. It is up to the users of the procedures described in this standard to develop their own repeatability information.

11. Keywords

Black liquor, Soda pulping, Kraft pulping, Density, Sodium sulfide, Sodium sulfite, Sodium sulfate, Sodium thiosulfate, Total alkali, Active alkali, Silica, Iron, Aluminum, Sulfur, Calcium, Magnesium, Sodium

12. Additional information

12.1 Effective date of issue: To be Assigned.

12.2 This method, formerly T 625 ts-64, was reclassified as a Classical Method in 1985 by the former Test Methods Management Committee of the TAPPI Board of Directors. This reclassification was made necessary since the last revision was over five years ago and no revision or reaffirmation had been accomplished by the responsible committee. It was withdrawn by the Quality and Standards Management Committee in 1999 due to lack of progress in its review.

12.3 This method was returned to active status as a classical method in 2014 by requests from users that use the "ABC" method for black liquor analysis.

12.4 The total solids procedure (7.2) was replaced in the 2014 edition by a reference to T 650 "Solids content of black liquor."

12.5 This test method is similar to PAPTAC Standard J.15P "Analysis of Sulphate Process Black Liquors."

12.6 This method has been reconfirmed with amendments in 2023 by requests from users particularly on safety concerns (5.2 and 5.3).

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

