

T 624 cm-11

TENTATIVE STANDARD – 1942
OFFICIAL STANDARD – 1944
TENTATIVE STANDARD – 1960
OFFICIAL STANDARD – 1968
CLASSICAL METHOD – 1985
REVISED – 2000
CORRECTED – 2004
REVISED – 2011
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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 soda and sulfate white and green liquors

1. Scope

This method is for an accurate analysis of all the main components of soda or sulfate white or green liquors and may be used as a reference method for establishing quicker or more convenient tests suited to routine control.

2. Apparatus

- 2.1 *Hydrometer*, 1.0 to 1.1 sp gr, or a pycnometer (specific gravity bottle).
- 2.2 *Filtering crucible*, 30 mL, porcelain with 3 to 5 μm pores.
- 2.3 *Kjeldahl flask*, 300 mL (long-necked, round-bottom flask).
- 2.4 *pH meter*, fitted with platinum and saturated calomel electrodes, plus glass electrode and buffer solutions.
- 2.5 *Magnetic stirrer*, with 19-mm (3/4-in.) Teflon-covered stirrer bar.
- 2.6 *Muffle furnace*, electrically heated, with temperature controlled to 1200°C.
- 2.7 *Other apparatus*: steam bath; Büchner funnel and suction flask, each 250 mL; 100-, 250-, and 500-mL volumetric flasks; 50-mL burets; 1-, 5-, 10-, 25-, and 50-mL pipets; 250- and 500-mL Erlenmeyer flasks; 150-, 250-, and 600-mL beakers; watch glass cover and stirring rod for 250-mL beaker; filter funnel and 5 to 6-cm diameter fine and medium porosity, quantitative filter paper.
- 2.8 *For ascarite determination of carbonate*:
- 2.8.1 *CO₂ absorption apparatus*, see Figs. 1 and 2.

Approved by the SSIG for this Test Method



Documents

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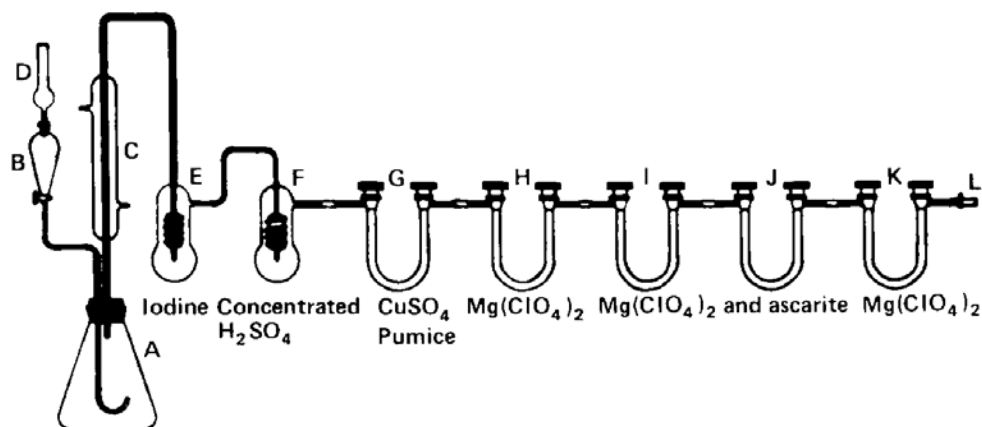


Fig.1. Apparatus for direct determination of carbon dioxide.

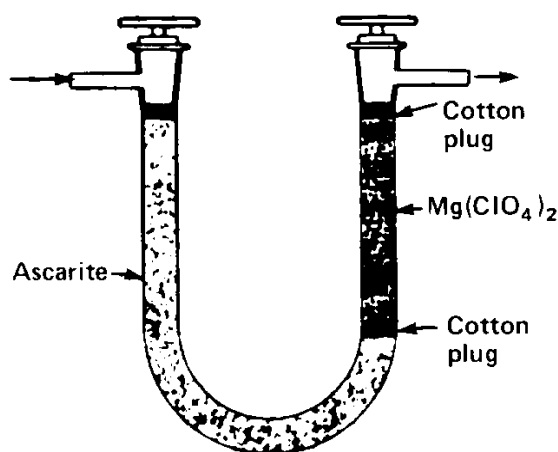


Fig. 2. Absorption tube for carbon dioxide tubes I and J in Fig. 1.

- 2.9 For total sulfur, silica, and metals other than sodium:
- 2.9.1 Platinum evaporating dish, about 100-mL size or larger.
- 2.9.2 Platinum crucible and cover, about 35-mL size or larger.
- 2.9.3 Oven, at 105 and $160 \pm 3^\circ\text{C}$.

3. Reagents

- 3.1 Use reagent grade chemicals and use dilution water which has been distilled and recently boiled and cooled.
- 3.1.1 Sodium thiosulfate, 0.1N $\text{Na}_2\text{S}_2\text{O}_3$, containing $0.1\text{--}0.2$ g/L Na_2CO_3 (see TAPPI T 610 "Preparation of Indicators and Standard Solutions").
- 3.1.2 Iodine, $0.15\text{--}0.2\text{N}$. Dissolve 20 g I_2 in concentrated solution of 60 g KI in water, and dilute to 1 L. Check daily, or before every analysis, by titration with standardized $\text{Na}_2\text{S}_2\text{O}_3$.
- 3.1.3 Sulfuric acid, 20% by volume, also 0.5N H_2SO_4 (see T 610).
- 3.1.4 Hydrochloric acid, dilute HCl $1:20$ and $1:100$.
- 3.1.5 Zinc sulfate, 1M , $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (287 g/L).
- 3.1.6 Sodium carbonate, 1M Na_2CO_3 (106 g/L).
- 3.1.7 Sodium hydroxide, approximately 1N and 12N NaOH .
- 3.1.8 Sodium sulfite, Na_2SO_3 (25 g/L).
- 3.1.9 Barium chloride, BaCl_2 (10%).
- 3.1.10 Ammonium hydroxide, concentrated and approximately 6N NH_4OH .

- 3.1.11 Silver nitrate-ammonia, 2.5% solution AgNO_3 to which NH_4OH has been added until brown precipitate just clears.
- 3.1.12 Methyl orange indicator (see T 610).
- 3.1.13 Starch indicator (see T 610) or thyodene.
- 3.1.14 Uranyl zinc acetate reagent (see TAPPI T 623 "Sodium Determination by the Uranyl Zinc Acetate Method").
- 3.1.15 Alcohol reagent (see T 623).
- 3.1.16 Ether.
- 3.2 For CO_2 absorption apparatus (for accurate determination of carbonate):
 - 3.2.1 Iodine, 5% solution, dissolved in 1M KI.
 - 3.2.2 Anhydrous magnesium perchlorate, $\text{Mg}(\text{ClO}_4)_2$.
 - 3.2.3 Ascarite, sodium hydrate asbestos absorbent.
 - 3.2.4 Copper-sulfate pumice: soak 4 to 8 mesh pumice stone in saturated CuSO_4 and dry at 150 to 180°C. Keep in tightly sealed container.
- 3.3 For total sulfur, silica and metals other than sodium:
 - 3.3.1 Nitric acid, concentrated HNO_3 nitrite free (white).
 - 3.3.2 Ammonium oxalate, crystals $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ and 1% solution.
 - 3.3.3 Hydrogen peroxide, 30% H_2O_2 .
 - 3.3.4 Hydrofluoric acid, concentrated HF.
 - 3.3.5 Silver nitrate, 0.1N AgNO_3 (see T 610).
 - 3.3.6 Ferric ammonium sulfate, saturated solution $\text{Fe}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$.
 - 3.3.7 Potassium thiocyanate, 0.1N KSCN (see T 610).
 - 3.3.8 8-Hydroxyquinoline, 5% solution in 2N acetic acid (60 g CH_3COOH per L).
 - 3.3.9 Methyl red indicator (see T 610).
 - 3.3.10 O-Cresolphthalein indicator, 0.20% dissolved in alcohol.
 - 3.3.11 Sodium tauroglycocholate, surfactant (optional).

4. Sampling

The composition of sulfate liquors tends to change on standing, especially if access to air is permitted. Take a representative sample and store in a completely filled and stoppered alkali-resistant bottle. Perform the analysis as quickly as possible after sampling; state if more than 24 h have elapsed.

5. Procedures

- 5.1 Density
 - 5.1.1 For maximum accuracy, determine the density at 20°C by means of a calibrated pycnometer, weighing the bottle empty, filled with water, and filled with the liquor at the same temperature.
 - 5.1.2 For ordinary purposes use a hydrometer with a range of 1.0 to 1.1 sp gr.
- 5.2 Sodium sulfate. Pipet 25 mL of liquor into a 250-mL beaker and dilute with 50 mL of water. Cover the beaker with a watch glass and add slowly through the beaker's pouring lip 20 mL of concentrated HCl. Boil gently and evaporate on a steam bath to a moist slush. Dissolve the salts in about 50 mL of hot water. Filter the solution through medium porosity quantitative filter paper and wash the residue with about 75 mL of distilled water. Collect the filtrate and washings in a 250-mL beaker and discard the filter paper and the collected solids. Add two or three drops of methyl orange and add concentrated NaOH dropwise until the solution turns from red to yellow. Add 5 mL of concentrated HCl and heat the solution until it just begins to boil. At this point add rather rapidly and with stirring a boiling mixture of 30 mL of 10% BaCl_2 and 20 mL of water. Continue stirring for a minute or two, let the precipitate settle for about an hour, and then filter with suction through the tared porcelain filter crucible. Wash the precipitate and the inner wall of the crucible with at least 250 mL of warm water, dry the crucible for a few hours in an oven at 105°C, and then place it in a muffle furnace at 800°C for half-hour intervals until constant weight is attained. Cool the crucible in a desiccator before weighing to obtain the weight of barium sulfate. Convert to sodium sulfate, in grams per liter, by the formula:

$$\text{Na}_2\text{SO}_4 = \frac{\text{grams BaSO}_4 \times 1000 \times 142.1}{25 \times 233.4}$$