

T 619 cm-10

TENTATIVE STANDARD – 1933
OFFICIAL STANDARD – 1935
CORRECTED – 1944
CORRECTED – 1953
CLASSICAL METHOD – 1984
REAFFIRMED – 2001
REAFFIRMED – 2010
©2010 TAPPI

The information and data contained in this document were prepared by a technical committee of the Association. The committee and the Association assume no liability or responsibility in connection with the use of such information or data, including but not limited to any liability under patent, copyright, or trade secret laws. The user is responsible for determining that this document is the most recent edition published.

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 salt cake

1. Scope and significance

1.1 The principal use of salt cake in the pulp and paper industry is in the manufacture of sulfate pulp. Salt cake is an anhydrous sodium sulfate which contains varying amounts of impurities. The principal impurities usually are sulfuric acid, common salt, and sulfates of Fe, Al, Ca, and Mg. "Chrome salt cake," obtained as a by-product in making chromic acid, has a yellow color and contains $\text{Na}_2\text{Cr}_2\text{O}_7$ (and/or Na_2CrO_4).

1.2 A complete analysis of salt cake is somewhat complicated and involves a considerable number of determinations, as outlined below. Since some mills are mainly interested in the acidity and the Na_2SO_4 content, directions for a "short analysis" are also given.

1.3 This method is based on wet chemical analyses of the parameters of interest. If available, other techniques such as AA, ICP or ion chromatography may be employed.

2. Preparation of sample

Mix the sample thoroughly, breaking down or grinding any lumps; quarter down to about 100 g; grind the quartered portion, if necessary, to about 60-mesh or finer; place in a separate stoppered bottle.

3. Moisture

Weigh 10 g of the prepared sample in a flat-bottomed dish of porcelain, glass, or platinum and dry to constant weight at $105 \pm 3^\circ\text{C}$. (Four hours are usually sufficient.) Let the dish and contents cool in a desiccator before weighing and make the final weighing rapidly. Calculate the loss in weight to percentage and report as moisture.

Approved by the Standard Specific Interest Group for this Test Method



Documents
Document Sharing Hub

4. Insoluble matter

4.1 Weigh 50 g of the prepared sample into a 400- or 500-mL beaker. Add about 250 mL of boiling water and stir well for 5 to 10 min. Filter at once through a filter paper which has been previously dried at $105 \pm 3^\circ\text{C}$, cooled, and weighed in a weighing bottle; collect the filtrate in a liter volumetric flask. Wash the residue and the filter paper thoroughly with hot water until a few drops of the washings give no test for sulfates. Place the filter paper and contents in the weighing bottle and dry to constant weight at 100 to 105°C . Calculate the percentage of insoluble matter.

4.2 Cool the filtrate and washings in the liter flask to room temperature, dilute to the mark at 20°C and mix thoroughly. Call this Solution A.

5. Acidity

Pipet 100 mL of Solution A (equivalent to 5 g of the sample) into a beaker or flask, dilute with an equal volume of water, add about 1 mL of phenolphthalein indicator, and titrate to a pink color with 0.1N NaOH. Calculate the titration to percentage of H_2SO_4 and report as acidity, calculated as H_2SO_4 .

Calculation: 1 mL 0.1N NaOH = 0.0049 g H_2SO_4 .

NOTE 1: This titration gives the total acidity or alkali-consuming power and includes not only the free H_2SO_4 present in the material but also the acidity due to sulfates of Fe and Al and of any bichromate. The actual free H_2SO_4 may be closely approximated by calculating the amount of H_2SO_4 due to the various impurities and subtracting from the percentage of acidity as H_2SO_4 .

Factors: H_2SO_4 equivalent = $\text{Al}_2\text{O}_3 \times 2.89$
 = $\text{FeO} \times 1.36$
 = $\text{Fe}_2\text{O}_3 \times 1.84$
 = $\text{Na}_2\text{Cr}_2\text{O}_7 \times 0.375$

6. Chloride

To the solution which has been titrated for acidity add a drop or two of methyl orange or methyl red indicator, make very slightly acid with a few drops of dilute H_2SO_4 , and bring back to neutrality by adding about 1 g of powdered CaCO_3 and stirring. Set the flask on a white surface or transfer the contents to a porcelain dish; add 10 drops of K_2CrO_4 indicator solution (10%) and titrate with 0.1N AgNO_3 until the brick-red coloration due to Ag_2CrO_4 remains just visible after stirring. Calculate the titration to percentages of Cl and NaCl (and also to the equivalent of Na_2O for use in the final calculations).

Calculations: 1 mL 0.1N AgNO_3 = 0.00355 g Cl
 = 0.00585 g NaCl
 = 0.00310 g Na_2O

7. Ferrous iron

To 5 g of the original prepared sample in an Erlenmeyer flask add 100 mL of warm water and 5 mL of concentrated H_2SO_4 . Warm on the steam bath a few minutes, just long enough to dissolve all soluble matter. Cool under a water tap and titrate at once to a pink color with 0.1N KMnO_4 . Calculate the titration to percentages of FeO and FeSO_4 (and also the equivalent of Fe_2O_3).

Calculations: 1 mL 0.1N KMnO_4 = 0.0072 g FeO
 = 0.0152 g FeSO_4
 = 0.0080 g Fe_2SO_3