

T 618 cm-10

TENTATIVE STANDARD – 1932
OFFICIAL STANDARD – 1934
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CLASSICAL METHOD – 1984
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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 limestone

1. Scope

- 1.1 This method is for the analysis of limestone as used in current sulfite pulping practice.
- 1.2 Limestone may be in the calcite form (high in calcium) or the dolomite form (approaching the double carbonate of calcium and magnesium). In pieces of various sizes, it is used in the tower system of making bisulfite cooking liquor. As a rule, the combined total of other oxides including silicon, iron, and aluminum should be less than 3%.

2. Apparatus

- 2.1 *Hammer*, mason's or other type, for sampling large pieces.
- 2.2 *Jaw crusher*, or other equipment for reducing 2 to 3 cm (1-in.) sample lumps to 60-mesh.
- 2.3 *Testing sieves*, 60 and 100 mesh, and pan.
- 2.4 *Mechanical grinder*, or agate mortar and pestle for reducing 60-mesh material to pass a 100-mesh sieve.
- 2.5 *Glass bottles*, about 175 mL (6 oz) rubber-stoppered, wide mouthed, to contain the sample and protect it from the air.
- 2.6 *Platinum crucible*, 20 to 25 mL, with cover.
- 2.7 *Electric muffle furnace*, for 1000°C and lower, with temperature control and indicator.

NOTE 1: If an electric muffle is not available, a grid-top burner may be used, with less assurance of standard results.

- 2.8 *Casserole*, porcelain, 300-mL.
- 2.9 *Hot plate*, with sand bath, for heating the casserole to 200°C. (A steam bath and an oven for temperatures to 200°C may be substituted.)
- 2.10 *Jones-Reductor*, for KMnO_4 titration of R_2O_3 residue.

NOTE 2: See Scott's *Standard Methods of Chemical Analysis*, 6th Edn., Vol. 1, p. 540 (D. Van Nostrand, 1962).

- 2.11 *Filter paper*, 11-cm diameter.

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2.12 *Colorimeter* (for colorimetric measurement of Fe_2O_3), with 520 to 580 nm filter, or spectrophotometer capable of accurate measurement of light absorption in the 520-580 nm range. Nessler tubes and visual comparison may be used if a colorimeter or spectrophotometer is not available.

2.13 *Other apparatus*: beakers, 250, 400, 600, and 800-mL; volumetric flasks, 100 and 250-mL; burets, 50-mL; graduated cylinder, 25-mL; pipets, 2, 5, and 10-mL; funnels, long-stem, 58 or 60°, 65 mm ID at top; stirring rods.

3. Reagents

- 3.1 *Hydrochloric acid*, 1:1, 1:10, 5% and concentrated HCl.
- 3.2 *Ammonium hydroxide*, 1:3, 5% and concentrated NH_4OH .
- 3.3 *Sulfuric acid*, 1:5, 1:1, and concentrated H_2SO_4 .
- 3.4 *Hydrofluoric acid*, 48% HF (for special SiO_2 determination).
- 3.5 *Oxalic acid*, 10% solution $\text{H}_2\text{C}_2\text{O}_4$.
- 3.6 *Ammonium oxalate*, saturated solution $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$.
- 3.7 *Diammonium phosphate*, 10% solution $(\text{NH}_4)_2\text{HPO}_4$ (prepared fresh as needed).
- 3.8 *Bromine water* (saturated), or concentrated HNO_3 .
- 3.9 *Ammonium chloride*, 2% solution NH_4Cl .
- 3.10 *Ammonium oxalate-oxalic acid solution*, containing 2% of the former and 1% of the latter.
- 3.11 *Potassium permanganate*, 0.05N and 0.2N solutions KMnO_4 (see TAPPI T 610 "Preparation of Indicators and Standard Solutions").
- 3.12 *Methyl red indicator*.
- 3.13 *Sodium carbonate*, anhydrous Na_2CO_3 .
- 3.14 *Potassium pyrosulfate* ($\text{K}_2\text{S}_4\text{O}_7$) or potassium bisulfate (KHSO_4).
- 3.15 *Sodium hydroxide*, 10% solution NaOH .
- 3.16 *Hydroxylamine hydrochloride*, 10% solution, $\text{NH}_2\text{OH} \cdot \text{HCl}$.
- 3.17 *Ammonium acetate*, 20% solution $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2$.
- 3.18 *1, 10 - Phenanthroline* (orthophenanthroline), 0.1% solution.
- 3.19 *Congo red indicator paper*.
- 3.20 *Standard iron wire* (A.C.S.) for preparation of standard iron solution.

4. Sampling

4.1 Select material so that it will represent an average of all parts of the pile or car and does not contain a disproportionate share of the top or bottom layers. In view of the usual size of the lumps, it is impractical to take samples with a shovel. It is recommended that samples be taken by breaking small portions with a hammer from approximately 100 lumps situated in various parts of the car or pile. Each piece needs to consist of but an ounce or two, broken so as to pass a 2.5-cm (1-in.) diameter hole.

4.2 Crush the sample as received at the laboratory with a jaw crusher or other means until all of it passes a 60-mesh sieve. Reduce the quantity by quartering (see TAPPI T 605 "Reducing a Gross Sample of Granular or Aggregate Material to Testing Size") to give a minimum of 75 g. Grind this portion, either by a mechanical device or by hand in an agate mortar, until it all passes a 100-mesh sieve. Then thoroughly mix it and place in the widemouth stoppered bottle.

5. Procedures

5.1 *Loss on ignition*. Weigh about a 0.5-g specimen of the prepared sample to the nearest 0.001 g into a clean, previously ignited and weighed, covered platinum crucible. Heat slowly at first to avoid loss by decrepitation, finally to constant weight at 1000°C , in the muffle furnace. (Usually 1 h is sufficient for complete ignition.) Cool in a desiccator and report the loss in weight as the loss on ignition. Save the residue.

5.2 *Silica and insoluble matter*

5.2.1 Transfer the previously-ignited residue quantitatively to a 300-mL porcelain casserole. Carefully slake with about 10 mL of water, and mix to a slurry with a flat-end glass stirring rod. Add 5 to 10 mL of concentrated HCl and digest with gentle heat until solution is complete, carefully breaking up any lumps. Evaporate to dryness and bake the residue on a sand bath or in an oven for 1 h. If the MgO content is high (10% or over on the nonvolatile basis), bake at 120°C ; if below 10% MgO, bake at 200°C . (The high dehydration temperature is efficient for low MgO, but with high MgO, silica redissolves at temperatures over 120°C .) Cool to 40°C or lower, drench with concentrated HCl and allow to