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Permanganate number of pulp

Scope

This method provides for the determination of the relative “hardness” or bleachability of chemical pulps (1) having a permanganate number below 35.

This Useful Method covers two variations in a single procedure. One procedure is for low lignin content pulps having a permanganate number less than 20; and a second procedure is for higher lignin content pulps having permanganate numbers of between 20 and 35. For pulps above permanganate number of 35, use TAPPI T 236 “Kappa Number of Pulp.”

TAPPI Standard T 236 “Kappa Number of Pulp,” may be used for all types and grades of chemical and semi-chemical wood pulps, including higher lignin content pulps with yields as high as 70%.

Significance

By definition, the permanganate number is the number of milliliters of tenth normal potassium permanganate solution (0.1N KMnO_4) which is absorbed by 1 g of oven-dry pulp under certain specified and carefully controlled conditions. This permanganate oxidation is a measure of the lignin content of the pulp and consequently a measure of bleach demand. The permanganate number is therefore useful in determining the extent of delignification of chemical pulps after digestion and in the intermediate stages of bleaching. Empirical curves may be found in the literature relating chlorine demand to permanganate number for un-bleached sulfite and Kraft pulps.

Safety precautions

This method requires the use of hazardous chemicals, caution is required in their use and disposal.

Approved by a Working Group of TAPPI in accordance with the Useful Method Guidelines

Apparatus

1. *Mechanical stirrer*, with a propeller-type agitator made of glass or other non-corrosive material. A propeller speed of 500 ± 100 rpm with an agitator 25 mm in diameter should generate sufficient stirring to completely mix the added permanganate in five seconds, yet not possess a vortex great enough to draw air into the solution.
2. *Reaction beakers*, one or more 1000- or 1500-mL white porcelain, or white enamel-lined, reaction beakers. Glass beakers of the same sizes may be used but the white-lined beakers are especially suitable for the titration procedure in this method.
3. *Burettes*, two, preferably of 50-mL capacity and graduated to 0.1 mL.
4. *Volume measuring glassware* such as pipettes and graduated cylinders.
5. *Funnel and filter flask*, a small Büchner funnel and filter flask for slush pulp evaluation.

Reagents

1. *Potassium permanganate*, 0.1N, of exact or known strength, accurate to ± 0.0005 N KMnO_4 .
2. *Sodium thiosulfate*, 0.1N, of exact or known strength, accurate to ± 0.0005 N $\text{Na}_2\text{S}_2\text{O}_3$.
3. *Potassium iodide solution*, approx 1N, produced by mixing 166 g of KI per 1000 mL. The solution should not contain free iodine or iodate. The latter if present will liberate free iodine upon acidification of the solution.
4. *Sulfuric acid*, approx 4N, the solution to be made by adding 112 mL of c.p. concentrated H_2SO_4 (94.5 - 96.5%) to about 500 mL of cold water and diluting to 1000 mL at 20°C.
5. *Starch indicator solution*.
6. *Distilled or demineralized water*.

Test specimen

Obtain duplicate test specimens, representative of the whole flow, or lot, of pulp sampled, each consisting of 1 ± 0.02 g of oven-dry pulp, accurately determined to the nearest 0.005 g.

While any convenient and suitable method may be used to weigh, or to measure out, the portion of the pulp sample used as the test specimen, sufficient accuracy in weight usually requires that the moisture content of the sample be accurately determined on a separate portion and a quantity equivalent to 1.00 g of dry pulp weighed or measured out.

The following examples illustrate suitable methods of obtaining the test specimens.

- If the pulp is sampled as slush, or from wet laps, dilute a portion containing from about 5 to 30 g of dry pulp to about 0.2% consistency with distilled or deionized water. While stirring vigorously, remove a volume equivalent to 1 or 2 g. Accurately measure the volume in a graduated cylinder, or weigh in a tared beaker to ± 0.5 g, then filter in a Büchner funnel, using a filter paper that was previously dried and weighed, and dry in an oven at 100–105°C to determine the actual oven-dry weight of pulp in the volume. Calculate the volume required to contain 1.00 g of oven-dry pulp. Measure out this portion, after vigorous stirring, to constitute the test specimen.
- If the pulp is sampled from dry laps, or from wet laps which have been air-dried, shred the portions and mix together, determine the moisture content on a portion of the mixture, then weigh a quantity equal to 1.00 g of oven-dry pulp and disperse in water until the fibers are completely dispersed.

Procedure

1. Always adjust the reaction mixture at the commencement of the test to 0.0033 ± 0.0001 N in KMnO_4 and 0.133 ± 0.005 N in H_2SO_4 , equal volumes of 0.1N KMnO_4 and 4N H_2SO_4 being diluted with the required amount of water to give this concentration. This is accomplished as follows:

- For pulps containing little lignin and which have a permanganate number not greater than 20, use 25.0 ± 0.1 mL of 0.1N KMnO_4 , 25 ± 1 mL of 4N H_2SO_4 , and 700 mL of water (including any with the pulp specimen) to give a total volume of 750 mL.
- For pulps of higher lignin content and which have a permanganate number greater than 20 (when 25 mL of KMnO_4 is used), use 40 ± 0.1 mL of 0.1N KMnO_4 , 40 ± 1 mL of 4N H_2SO_4 , and 1120 mL of water to give a total volume of 1200 mL.
- If permanganate number is above 35, use T 236 “Kappa Number of Pulp.”