

TAPPI/ANSI T 236 om-13

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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 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 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.

Kappa number of pulp

1. Introduction

Kappa number is a key test method for determining the level of lignin remaining in a sample of finished or in-process pulp. It is thus a measure of the completeness of the pulping process for many kinds of chemical and semi-chemical pulps, both bleached and semi-bleached. Kappa number is based upon the reaction of a strong oxidizing chemical, potassium permanganate, with lignin as well as small levels of certain other organic impurities remaining in the pulp at various stages of its processing. Kappa number gives the maker of the pulp, as well as the papermaking user of the pulp, valuable information about the properties of the pulp as well as the paper made from it, particularly with regard to the level of residual lignin present.

2. Scope

2.1 This kappa number standard applies to many kinds of chemical, semi-chemical, unbleached and semi-bleached pulps within the kappa number range 1 to 100. Above a kappa number of 100, precision of the test may decrease, and the relationship between kappa number and lignin content may decrease, depending mainly upon the wood species from which the pulp is made. There is no general and unambiguous relationship between the kappa number and the content of lignin of other organic impurities in a particular pulp. The relationship varies according to the wood species and the pulping and delignification procedures used during the pulping process for a specific pulp. Kappa number is essentially a straight line relationship with both klason lignin and chlorine number for pulps below 70% total pulp yields (1). The percentage of klason lignin in a pulp sample whose kappa number is determined by the procedure in this standard test method may be approximated using the following equation

$$\text{Lignin level (\%)} = \text{Kappa number} \times 0.13$$

If the kappa number is to be used to determine a precise numerical value regarding the amount of lignin present in a specific pulp of interest, a more precise relationship can be established by testing the specific pulp of interest.

2.2 As written, this standard is intended for use in the laboratory testing of pulps. It is recognized, however, that kappa number is widely used as an in-process test in the pulp and paper mill, in some cases with modifications.

Section 16 of this standard includes informative information regarding the unintended or unexpected impact that certain deviations from the standard can have on data accuracy, precision, or both.

2.3 Likewise, this standard does describe the use of automated instruments for measuring kappa number. The user of such equipment shall verify the applicability of such equipment for any intended use, and do such testing as may be required to determine the agreement of results from any automated testing equipment used with results obtained using the procedure in this test method.

3. Applicable documents

TAPPI T 550 "Determination of equilibrium moisture in pulp, paper and paperboard for chemical analysis"

TAPPI T 610 "Preparation of indicators and standard solutions"

ISO 302 "Pulps – Determination of kappa number."

PAPTAC Standard G.18 "Kappa number of Pulp"

SCAN-C 1.00 "Kappa number" (withdrawn 2007)

4. Summary of the kappa number procedure

4.1 A sample of air-dried pulp of known weight is disintegrated in a known volume of distilled water, and the lignin and minor levels of other non-cellulose impurities in the sample are chemically oxidized in acidic solution by reaction with a known amount of potassium permanganate (KMnO_4) in a reaction vessel under atmospheric pressure for 10 minutes at 25° Celsius. A separate test specimen will be used for moisture correction determination in order to calculate the equivalent oven-dry specimen weight. See section 16.7 regarding wet processed pulp samples.

4.2 After the 10-minute reaction period, a known amount of potassium iodide solution containing iodide in excess of the expected amount of permanganate remaining unreacted is added to the reaction vessel. The potassium iodide reacts instantaneously with all the remaining unreacted potassium permanganate, stopping or "quenching" any further oxidation of any remaining lignin and other oxidizable organic compounds that were present in the sample. An amount of iodine chemically equivalent to the amount of residual permanganate which remained in the reaction vessel at the time of the iodide addition is formed.

4.3 The resulting iodine formed is titrated with a standardized solution of sodium thiosulfate. From the amount of thiosulfate consumed, the amount of unreacted at the moment the oxidation reaction with the sample was quenched by the iodide addition can be calculated. From that information, the amount of permanganate that reacted with the original sample in the ten-minute reaction time is determined and the kappa number calculated.

4.4 The size of the pulp sample used for the test is chosen such that about 50% of the total oxidation capacity of the amount of added permanganate, as measured by the procedures in 4.2 and 4.3, is consumed by reaction with lignin and other impurities in the sample by the end of the 10-minute reaction time. If the amount of permanganate consumed is greater than or less than specified limits, the sample size shall be adjusted and the test shall be repeated to assure accuracy of the results.

4.5 For further background on the development of the kappa number test, see Sections 16.4 and 16.5.

5. Significance

The level of lignin and other impurities in pulp is very important in pulp production, as it is directly related to the amount and type of bleaching chemical required to produce a finished pulp of specified end use properties, particularly pulp brightness. Kappa number may be used as a specification for pulp used in various end use paper products such as bag papers, printing and writing papers and others.

6. Definition

Kappa number: the volume (in milliliters) of 0.1N potassium permanganate solution consumed by the equivalent of one gram of oven dried pulp under the conditions specified in this method. The results are corrected to the grams of sample which would react with 50% of the potassium permanganate added. Kappa number is expressed as a unitless number.

7. Interferences

The test procedure is based upon oxidation of lignin by permanganate and reduction of excess permanganate by thiosulfate, hence any chemically oxidizable or reducible contaminant not typical of the pulp itself may cause errors in the measured result. In general, such interferences come from samples which contain residues of pulping and bleaching operations that have not been properly or completely washed prior to sampling or sample preparation. This is rarely a problem in the case of air-dried sheet pulps, but should be considered when slush pulps or any in-process pulp samples are being tested.

8. Apparatus

- 8.1 *Agitator*, propeller type, made of glass or other noncorrosive material (a plastic or glass-covered magnetic stirrer may be used instead).
- 8.2 *Disintegration apparatus*, wet, high-speed type, which disintegrates the pulp completely with a minimum of damage to the fibers. Avoid disintegrators that may contaminate the sample with grease.
- 8.3 *Water bath or constant temperature bath*, capable of maintaining a constant temperature of $25.0 \pm 0.2^\circ\text{C}$ in the reaction vessel. It is convenient if the bath is of sufficient size to hold not only the reaction vessel but also the sulfuric acid and potassium permanganate solutions (9.1, 9.4).
- 8.4 *Beaker* 2000-mL.
- 8.5 *Pipets*, two 100-mL automatic pipets are especially convenient when a large number of determinations are to be made.
- 8.6 *Buret*, 50-mL, graduated to 0.1 mL. A 50-mL buret will be found more convenient for titration of the reaction mixture in the blank test.
- 8.7 *Analytical balance*, accurate to ± 0.0002 g.
- 8.8 *Büchner funnel and filter flask* to dewater three to four grams of pulp.
- 8.9 *Filter paper*, coarse pore size, 150-mm diameter.
- 8.10 *Stopwatch*, accurate to the nearest second.
- 8.11 *Graduated cylinders*, 1000, 25 and 50 mL.
- 8.12 *Beaker*, 250 mL.

9. Reagents

- 9.1 *Potassium permanganate solution*, analytical reagent grade, standardized with a normality of $0.1000 \pm 0.0005N$ KMnO_4 . Normality shall be as stated, that is, between $0.0995N$ and $0.1005N$. Care shall be taken in the preparation and standardization of the potassium permanganate solution. In cases where the potassium permanganate solution is used infrequently (less than twice per week) or where the amount prepared lasts for more than a week, standardization of the solution shall be checked weekly and a new batch of the standard solution prepared when the normality deviates from the limits stated above. Refer to TAPPI T 610 "Preparation of indicators and standard solutions." The "aging" of potassium permanganate solution is a recognized problem which must be guarded against if precision and accuracy of test results are to be achieved.
- 9.2 *Sodium thiosulfate solution*, $0.2000 \pm 0.0005N$ $\text{Na}_2\text{S}_2\text{O}_3$.
- 9.3 *Potassium iodide solution*, $1.0N$ KI .
- 9.4 *Sulfuric acid*, $4.0N$ H_2SO_4 .
- 9.5 *Starch indicator solution*, 0.2%.
- 9.6 *Water*, distilled.

10. Sampling

- 10.1 If the test is being made to evaluate a pulp lot, the sample shall be selected in accordance with TAPPI T 400 "Sampling and accepting a single lot of paper, paperboard, containerboard, or related product."
- 10.2 If the test is made on another type of sample, report the source of the sample and the sampling plan or procedure used. Make sure the test portions taken are representative of the pulp.
- 10.3 When testing in-process samples, it is important to remember that the presence of small amounts of spent cooking liquor may cause errors in the measured kappa number. Ensure that any sample which may be contaminated with