T 236 om-99

TENTATIVE STANDARD - 1960
OFFICIAL STANDARD - 1976
CLASSICAL METHOD - 1985
CORRECTION - 1993
OFFICIAL METHOD -1999

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.

Kappa number of pulp

1. Scope and significance

This method applies to the determination of the relative hardness, bleachability, or degree of delignification of pulp. It may be used for all types and grades of chemical and semichemical, unbleached and semibleached pulps obtained in yields under 60%. This method may also be used for pulps obtained in yields up to 70%, provided the pulp has been well screened. See also Additional Information 10.5.

2. Definition

The *kappa number* is the volume (in milliliters) of 0.1*N* potassium permanganate solution consumed by one gram of moisture-free pulp under the conditions specified in this method. The results are corrected to 50% consumption of the permanganate added.

3. Apparatus

3.1 *Agitator*, of propeller type, made of glass or other noncorrosive material (a plastic or glass-covered magnetic stirrer may be used instead).

3.2 *Disintegration apparatus*, of 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.

3.3 *Constant temperature bath*, capable of maintaining a constant temperature of 25.0 " 0.2°C in the reaction vessel.

3.4 *Reaction beaker*, 2000-mL, glass or porcelain.

3.5 *Pipets*, two 100-mL automatic pipets are especially convenient when a large number of determinations are to be made.

3.6 *Buret*, 50-mL, graduated to 0.1 mL. A 52-mL buret will be found more convenient for titrating the reaction mixture in the blank test.

3.7 *Other apparatus*: a Büchner funnel and filter flask to dewater three to four grams of pulp; stopwatch or clock; 1000-mL and a 25- or 50-mL graduated cylinder; 250-mL beaker.

Approved by the Pulp Properties Committee of the Process and Product Quality Division
TAPPI

4. Reagents

4.1 *Potassium permanganate solution*, standardized 0.1000 " 0.0005*N* $KMnO_4$.

4.2 *Sodium thiosulfate solution*, approximately 0.2*N* $Na_2S_2O_3$. Normality known with an accuracy of " 0.0005*N*.

4.3 *Potassium iodide solution*, 1.0*N* KI.

4.4 *Sulfuric acid*, 4.0*N* H_2SO_4.

4.5 Starch indicator solution, 0.2%.

5. Preparation of sample

5.1 *Air-dried pulp sheets*. Tear small pieces from the sample sheets to weigh a total of three to four grams.

5.2 *Screened slush sheets.* Mix and make three to four grams (dry weight) into a pad by filtering on a Büchner funnel; avoid any loss of fibers. Air-dry the pad and tear it into small pieces.

5.3 *Unscreened pulps.* If the pulp sample is taken from unscreened pulp which is normally screened before bleaching and other processing, then remove the shives and knots from the sample by screening. State method of screening along with the test results and choose the method which would give results similar to those obtained by the industrial screening of the pulp. Proceed as in 5.2.

6. Procedure

6.1 Prior to weighing the test samples, condition them for at least 20 min in the atmosphere near the balance.

6.2 Weigh out to the nearest 0.001 g that amount of pulp specimen which will consume approximately 50% of the potassium permanganate solution. The permanganate consumption must be between 30 and 70%. At the same time weigh out a second specimen and determine its moisture content in accordance with TAPPl T 550 "Determination of equilibrium moisture in pulp, paper and paperboard for chemical analysis."

6.3 Disintegrate the test specimen in 500 mL or less of distilled water until free of fiber clots and undispersed fiber bundles. Avoid methods of disintegration which involve extensive cutting of the fibers.

6.4 Transfer the disintegrated test specimen to a 2000-mL reaction beaker and rinse out the apparatus with enough distilled water to bring the total volume to 795 mL. The distilled water should be at least 25.0 " 0.2°C.

6.5 Place the beaker in a constant temperature bath adjusted so that the reaction temperature stays at 25.0 " 0.2°C during the entire reaction. Continuously stir the suspension so as to produce a vortex about 25 mm deep but not fast enough to introduce air into the mixture (see 10.1).

6.6 Pipet 100.0 " 0.1 mL of potassium permanganate solution and 100 mL of the sulfuric acid solution into a 250-mL beaker. Bring this mixture to 25°C quickly and add it immediately to the disintegrated test specimen, simultaneously starting a stopwatch. Rinse out the beaker, using not more than 5 mL of distilled water, and add the washings to the reaction mixture. The total volume should be 1000 " 5 mL.

6.7 At the end of exactly 10.0 min, stop the reaction by adding 20 mL of the potassium iodide solution from a graduated cylinder.

6.8 Immediately after mixing, but without filtering out the fibers, titrate the free iodine with the sodium thiosulfate solution, adding a few drops of the starch indicator toward the end of the reaction (see 10.3).

6.9 Carry out a blank determination using exactly the same method as above but omitting the pulp (see 10.3)

7. Calculations

7.1 Calculate kappa number as follows:

$$K = \frac{p \times f}{w}$$

and

$$p = \frac{(b - a)\, N}{0.1}$$

where:

K = kappa number
f = factor for correction to a 50% permanganate consumption, dependent on the value of *p* (see Table 1)
w = weight of moisture-free pulp in the specimen, g
p = amount of 0.1*N* permanganate actually consumed by the test specimen, mL
b = amount of the thiosulfate consumed in the blank determination, mL
a = amount of the thiosulfate consumed by the test specimen, mL
N = normality of the thiosulfate

7.2 Factors in Table 1 are based on the equation: $\log K = \log p/w + 0.00093\ (p\text{-}50)$.

Table 1. Factors *f* to correct for different percentages of permanganate used

p	+	*0*	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*
30		0.958	0.960	0.962	0.964	0.966	0.968	0.970	0.973	0.975	0.977
40		0.979	0.981	0.983	0.985	0.987	0.989	0.991	0.994	0.996	0.998
50		1.000	1.002	1.004	1.006	1.009	1.011	1.013	1.015	1.017	1.019
60		1.022	1.024	1.026	1.028	1.030	1.033	1.035	1.037	1.039	1.042
70		1.044									

8. Report

8.1 Report the kappa number as follows:
8.1.1 Under 100, to the nearest 0.1.
8.1.2 Over 100, to the nearest whole number.

9. Precision

9.1 The following estimates of repeatability and reproducibility are based on data from a limited interlaboratory trial involving 6 laboratories and five different pulps. The trial was conducted in November 1997 using the "cm-88" revision of this method. Testing is based on one determination per test result and three results per lab, per material.

Kappa Measurements

Material	Grand Mean	Repeatability r		Reproducibility R		Labs Included
Pulp A	5.76	0.30	5.2%	1.20	20.8%	5
Pulp B	12.33	0.27	2.2%	0.28	2.3%	5
Pulp C	24.48	0.55	2.2%	1.23	5.0%	5
Pulp D	44.52	3.01	6.8%	11.67	26.2%	6
Pulp E	115.45	8.37	7.3%	15.07	13.0%	5

9.2 Repeatability and reproducibility are estimates of the maximum difference (at 95%) which should be expected when comparing test results for materials similar to those described above under similar test conditions. These estimates may not be valid for different materials or testing conditions.

10. Additional information

10.1 Effective date of issue: October 19, 1999.

10.2 This revision differs from the 1960 edition as follows: (a) a precision statement has been added; (b) the use of smaller quantities of specimen and reagents has been eliminated; and (c) the conversion table from 40 mL kappa numbers (T 214) to kappa numbers has been deleted.

10.3 Iodine volatization has been found to be an important variable in the kappa number determination. The timing between the reaction and subsequent titration completion should be as short as possible. Blank determinations should duplicate the testing of the specimen as nearly as possible (omitting the pulp, of course).

10.4 *Correction for reaction temperature*. When a constant temperature bath is not available, determine the temperature after the reaction has been taking place for 5 min and assume this to be the average reaction temperature throughout the test. If this temperature is not higher than 30°C nor lower than 20°C, correct the kappa number as follows:

$$K = \frac{pf}{w} \left[1 + 0.013 \left(25 - t\right) \right]$$

where t = actual reaction temperature in degrees Celsius. If temperature is outside this range, redo the determination maintaining temperature in proper range.

10.5 *Relationship with lignin.* The kappa number gives 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 approximately equals *K* H 0.13.

10.6 *Aging.* Freshly made pulp has a slightly higher permanganate consumption than pulp which has stood several days or months. The change is rather rapid immediately after the pulp is made but reaches a relatively stable stage after two or three days.

10.7 Related methods: Australian Standard AS 1301. P 201m , "Kappa Number of Pulp," Technical Association of the Australian and New Zealand Pulp and Paper Industry, Parkville, Australia; CPPA G.18, "Kappa Number of Pulp," Canadian Pulp and Paper Association, Montreal, Canada; ISO R 302, "Determination of the Kappa Number of Pulp (Degree of Delignification)," International Organization for Standardization, Geneva, Switzerland; SCAN C1, "Kappa Number of Pulp" (essentially identical), Scandinavian Pulp, Paper and Board Testing Committee, Stockholm, Sweden, ISO Standard ISO 302.

10.8 This method, formerly T 236 os-76, has been reclassified as a Classical Method by the Test Methods Management Committee of the TAPPI Board of Directors. This reclassification was made necessary since the last revision was over five years ago and no revision or reaffirmation has been accomplished by the responsible committee. This method was upgraded to an Official Method in 1999.

11. Keywords

Pulp, Lignin, Kappa number

Literature cited

1. Tasman, J. E., and Berzins, V., "The Permanganate Consumption of Pulp Materials," *Tappi* **40** (9): 691 (1957); *Pulp Paper Mag. Canada* **58**(10): 145 (1957).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Operations Manager. •