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WI 210802.04

T 223

BALLOT NO. 03 SARG

DRAFT NO. 02

DATE 6/1/2023

WORKING GROUP CHAIR To be determined

SUBJECT CATEGORY Chemical Properties

RELATED METHODS See "Additional Information"

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 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.

Pentosans in wood and pulp
(Proposed Reaffirmation of Classical Method T 223 cm-10)

1. Scope

This method for determination of pentosan content can be applied to both wood and to unbleached or bleached pulps.

2. Summary

Pentosans are transformed in boiling 3.85*N* hydrochloric acid to furfural, which is collected in the distillate and determined colorimetrically with orcinol-ferric chloride reagent.

NOTE 1: Hydroxymethyl-furfural formed from hexoses does not interfere in this determination (*1*).

3. Significance

3.1 Wood contains a certain amount of noncellulosic carbohydrates called hemicellulose. Softwood hemicellulose consists of both pentosans and hexosans; hardwood hemicellulose consists mainly of pentosans. Pentosan content in softwoods is about 7 to 10%, and in hardwoods about 19 to 25%.

3.2 Pentosan content in pulp indicates the retention or loss of hemicellulose in general during pulping and bleaching processes, and since hemicellulose contributes to the strength of paper pulps, high pentosan content is esirable. In dissolving pulps, particularly acetate pulps, pentosan content should be kept low.

4. Apparatus

4.1 *Distillation apparatus*, as illustrated in Fig. 1, consisting of a boiling flask, a graduated separatory funnel, Graham-type condenser, a volumetric flask (with marks added at 25-mL intervals), and two- and three-way connecting tubes. All connections of the apparatus are 24/40 ground glass joints.

4.2 *Heater*, electric, with temperature control, for use with boiling flask.

4.3 *Ice bath*, a beaker or dish with crushed ice into which volumetric receiving flask is immersed.

4.4 *Spectrophotometer or filter colorimeter*, for measuring light absorbance at 630 nm, with matched glass cells or cuvettes.

4.5 *Constant temperature bath*, to maintain a temperature of 25 ± 1°C.

4.6 *Timer*, or stopwatch.

4.7 *Other glassware*: volumetric flasks, 50-mL; pipets, 5-mL and 25-mL.

5. Reagents and materials

5.1 *Hydrochloric acid*, 3.85 ± 0.05N HC1 (13.15%). Dilute 315 mL of concentrated HCl (sp gr 1.18- 1.19) with water to 1000 mL. Determine the normality of the HCl by titration with 1.0*N* standardized sodium hydroxide solution whose normality is known to ±0.01*N*.

5.2 *Orcinol reagent.* Dissolve 0.400 g of orcinol, $CH_3C_6H_3(OH)_2$, and 0.500 g of ferric chloride, $FeCl_3 \cdot 6H_2O$, in 1000 mL of 11*N* hydrochloric acid (prepared by diluting 915 mL of concentrated HCl to 1000 mL). Store the reagent in a refrigerator and discard it if more than two weeks old.

5.3 *Sodium chloride*, NaCl crystals.

5.4 Ethanol, 95%, C_2H_5OH, aldehyde free. Ethanol denatured with methanol (Formula 30) may be used.

5.5 *Xylose*, $C_5H_{10}O_5$, reagent grade.

5.6 *Crushed ice*.

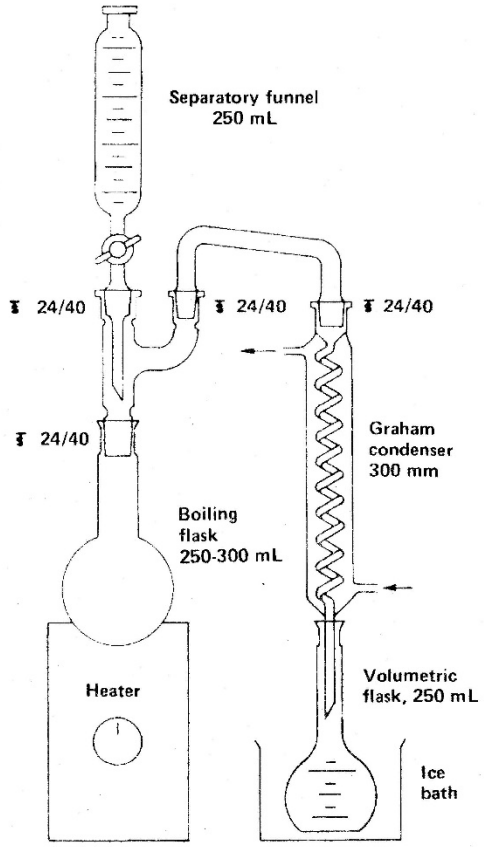

Fig. 1. Distillation apparatus

6. Sampling

6.1 For wood, obtain a sample and prepare 3 to 5 g of extractive-free wood in accordance with TAPPI T 257 “Sampling and Preparing Wood for Analysis” and TAPPI T 263 “Identification of Wood and Fibers from Conifers.”

6.2 For pulp, obtain a sample, equivalent to 5 to 10 g oven-dry, in accordance with a predetermined sampling procedure.

7. Test specimens

7.1 Allow the sample to come to moisture equilibrium in the atmosphere near the balance.

7.2 Using Table 1 as a guide, weigh out two test specimens to the nearest 0.1 mg and at the same time weigh another specimen for moisture determination.

7.3 Table 1 indicates the approximate size of a test specimen to provide furfural concentration in a suitable range (see Note 2).

NOTE 2: For an accurate measurement, the absorbance of the solution should be between about 0.1 and 0.8. If the reading is out of this range, either repeat the entire determination with a smaller or larger test specimen, or take less or more (from 2 to 10 mL, instead of 5 mL) of the distillate for development of color, or use cuvettes with a shorter or longer light-path. However, the same volume of distillate and the same type of cuvettes must be used both for the preparation of the calibration graph and for the test specimens.

Table 1. Test specimen sizes (approximate)

	Hardwood	*Softwood*
Wood	0.2-0.3 g	0.5-0.7 g
Unbleached pulp	0.5-1 g	l-2 g
Bleached pulp	2-3 g	3-5 g

8. Procedure

8.1 Place the test specimen in a boiling flask and add 20 g NaCl, 100 mL of 3.85*N* HCl and a few boiling stones. Connect the flask to the distillation apparatus and mark the acid level in the flask. Add 250 mL of 3.85*N* HCl to the separatory funnel.

8.2 Apply heat and distill the acid at a uniform rate of about 2.5 mL per min. Collect the distillate in a 250-mL volumetric flask immersed in an ice bath.

8.3 During distillation, maintain a constant volume of 100 mL in the boiling flask by adding HCl dropwise from the separatory funnel, or in 25-mL increments every 10 min. Continue the distillation for 90 ± 5 min, in which time 225 ± 10 mL of distillate should be collected.

8.4 Bring the temperature of the distillate to about 20°C, add 3.85*N* HCl to the 250-mL mark and mix thoroughly. Pipet 5.0 mL of the distillate into a 50-mL volumetric flask. Add 25.0 mL of orcinol reagent, mix, and place the flask in a water bath at 25 ± 1°C.

8.5 After 60 ± 5 min, add ethanol up to the 50-mL mark, mix, and return to the waterbath; then after another 60 ± 5 min, measure the absorbence of the solution with a spectrophotometer at 630 nm. To avoid corrosion of the instrument by HCl fumes, the cells or cuvettes should be covered.

8.6 Read the number of milligrams of xylan in the test specimen from a previously prepared calibration graph (see Section 12).

9. Calculation

Calculate the pentosan content in the test specimen:

$$\text{Pentosans, \%} = A / 10W$$

where

A = xylan in test specimen, mg
W = oven-dry weight of test specimen, g

10. Report

Report the percentage pentosan content as an average of two determinations, to one decimal if above 1%, or to 2 decimals if below 1%.

11. Precision

11.1 Repeatability (within a laboratory) = 6.6%; comparability (between materials) = not known; reproducibility (between laboratories) = 16.9%; in accordance with the definition of these terms in TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibilitiy."

11.2 These values are based on an earlier interlaboratory study (*2*) conducted by 7 laboratories on 10 pulps with pentosan content from about 0.4% to 16% using a colorimetric method essentially the same as this method.

12. Preparation of calibration graph

12.1 Dry pure xylose in a vacuum oven at about 60°C for 2-4 h and weigh, to the nearest 0.1 mg, several portions in a range from 10 to 100 mg (for example: 10, 20, 40, 60, 80, 100 mg) and place in boiling flasks; then proceed with distillation and development of the color exactly as previously outlined in Section 8.

12.2 Measure the absorbance of the distillates at 630 nm, using a blank containing 5 mL of 3.85*N* HCl instead

of distillate as the reference solution.

12.3 Calculate milligrams of xylan (anhydroxylose) in the specimen:

$$\text{Xylan, mg} = \text{xylose, mg} \times 0.88$$

12.4 Plot on graph paper absorbence against milligrams of xylan.

12.5 Check a few points on the calibration graph with xylose when new reagents are prepared. Make a new calibration graph if the checkpoints differ from the previous graph.

14. Additional information

14.1 Effective date of issue: To be assigned.

14.2 This method, formerly T 223 os-78, was reclassified as a Classical Method in 1984. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable. In 2010, reference to the related ASTM method was deleted, as the method has been withdrawn from ASTM. A suggested approach to standardization of the 3.85*N* HCl was added.

14.3 The determination of furfural by bromination (formerly a part of this method) is available as Useful Method 236.

14.4 Related methods: Canadian PAPTAC G.12, Scandinavian SCAN-C4.

Keywords

Wood, Pulp, Pentosans, Hemicellulose

References

1. Johansson, A., "The Determination of Pentosan," *Svensk Papperstid*. **55** (21): 820 (1952).
2. Wilson, W.K., and Mandel, J., "Determination of Pentosans," *Tappi* **43** (12): 998 (1960).

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■