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Pulps — Determination of lignin content — Acid hydrolysis method

Pâtes — Détermination de la teneur en lignine — Méthode d'hydrolyse par voie acide

ICS: 85.040

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The main objective of measuring lignin in pulp is to assess the effect of a particular pulping or bleaching process on the degree of delignification. In chemical pulping, the goal is to remove lignin from wood with minimum degradation of the carbohydrates. The higher the level of residual lignin in any type of unbleached pulp, the greater the amount of bleaching chemicals that must be applied in order to achieve a target brightness.

Comprehensive textbooks and reviews have been written on methods of lignin determination.[1-3] This Standard specifies one such method, commonly-used for the determination of the total lignin content of pulp. In this method, a pulp sample is treated with sulfuric acid, in a two-step (primary and secondary) hydrolysis process, to solubilize the carbohydrates. Most of the lignin remains insoluble at the end of the treatment and is filtered off, dried and weighed. This acid-insoluble lignin is also referred to as “Klason lignin”.

A small portion of lignin is dissolved during acid hydrolysis. This so-called acid-soluble lignin is determined spectrophotometrically, from the UV absorbance at 205 nm of the filtrate from the acid-insoluble lignin determination.[4-6] The total lignin content is determined as the sum of the acid-insoluble and acid-soluble lignin.

Two hydrolysis procedures are described in this Standard. In procedure A,[7-9] the primary hydrolysis is performed with 72% sulfuric acid at 30°C for one hour, followed by dilution with water to 4% sulfuric acid, and secondary hydrolysis in an autoclave at 120°C for one hour. In procedure B,[10,11] the primary hydrolysis is done at 15-20°C for two hours, followed by secondary hydrolysis at 3% sulfuric acid in a water bath at 100°C for four hours.

Both procedures have been shown to give the same results; thus either one can be used for determining acid-insoluble lignin. However, procedure A is considerably more rapid, and the use of an autoclave allows multi-samples to be hydrolysed simultaneously with minimum supervision. As such, it is now more commonly-used in laboratories equipped with an autoclave. It is therefore the preferred method, particularly when analysis of carbohydrates is required, in addition to the determination of lignin.

Pulps — Determination of lignin content — Acid hydrolysis method

WARNING — This method involves the use of hazardous chemicals. Care should be taken to ensure that the relevant precautions are taken.

1 Scope

The method is applicable to unbleached, bleached and semi-bleached pulp with a lignin content above 1%. It is not generally intended for fully bleached chemical pulp, because the lignin content in these pulps is too low to be determined accurately.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 1762, *Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 525 °C*

ISO 7213, *Pulps — Sampling for testing*

ISO 14453, *Pulps — Determination of acetone-soluble matter*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
Lignins
A class of complex, aromatic macromolecules that play a key role in the formation of cell walls in wood and bark, conferring mechanical strength and rigidity to the cell walls and to plants as a whole. Lignin is the main non-carbohydrate constitutent of wood.

3.2
Acid-insoluble (Klason) lignin
Residue after treating wood or pulp with sulfuric acid in a two-step hydrolysis procedure to solubilize the carbohydrates into monosaccharides.

3.3
Acid-soluble lignin
Portion of lignin which is soluble during the acid-insoluble lignin determination

3.4
Acetone-soluble matter
The amount of material that can be extracted with acetone from a sample of pulp by the method specified in ISO 14453

4 Principle

A pulp sample is treated with sulfuric acid in a two-step (primary and secondary) hydrolysis process to dissolve the carbohydrates. The residue after hydrolysis is filtered off, dried, and weighed, and referred to as acid-insoluble or Klason lignin. A small amount of lignin is dissolved during acid hydrolysis. This so-called acid-soluble lignin is determined by measuring the absorbance at 205 nm of the filtrate from the acid-insoluble lignin determination. The total lignin content is determined as the sum of the acid-insoluble and acid-soluble lignin.

5 Apparatus

5.1 Filtration equipment: filtering flask; filtering crucible, fritted glass, medium or fine porosity, 30 ml; adapter for the filtering crucible, siphon tube (optional).

Note The choice of fritted glass porosity depends on the rate of filtration of the particular type of sample. For slow filtering samples, the use of medium (M) porosity is preferable. In low-yield sulfite pulps especially, lignin forms a fine dispersion and clogs the pores of the filter. Filtration can be facilitated by using a medium porosity crucible with a disc of fine porosity glass-fibre filter paper fitted over the sintered glass in the crucible.

Other types of filtering crucibles, such as alundum or porous porcelain crucibles lined with a mat of glass fibres can also be used.

5.2 Constant temperature water bath, capable of maintaining a temperature of (30 ± 1)°C (Procedure A); or (20 ± 1)°C (Procedure B).

5.3 Flask, Erlenmeyer, 1000 ml, or smaller depending on the size of the pulp specimen.

5.4 Procedure A only: Autoclave, capable of maintaining a temperature of (120 ± 3)°C

5.5 Drying oven, convection type, maintained at (105 ± 2)°C.

5.6 Analytical balance, accurate to 0.1 mg.

5.7 Spectrophotomer, UV-visible, diode array or simple wavelength, with high purity quartz cuvettes of pathlength 1 cm.

6 Reagents

6.1 Water, distilled or deionized

6.2 Sulfuric acid, 72% w/w (specific gravity 1,6338 at 20°C). 72% sulfuric acid is available commercially. It can also be prepared from concentrated sulfuric acid as follows:

Add 300 ml of water to a 1000 ml volumetric flask. Add slowly 670 ml of concentrated sulphuric acid (H_2SO_4 sp gr 1.84) while cooling under a cold water tap. When the temperature has reached equilibrium with the ambient temperature, dilute to the mark and mix.

6.3 Acetone, only if extraction of the sample is required prior to hydrolysis with sulfuric acid.

7 Sampling

If the analysis is being made to evaluate a lot of a consignment of pulp, the sample shall be taken in accordance with ISO 7213. If the analysis is made on another type of sample, report the origin of the sample, and if possible the sampling procedure.

Obtain a representative sample of pulp equivalent to about 10 g moisture-free pulp. Air dry the pulp and disintegrate in a household blender, or grind in a Wiley mill to pass a No. 20 (0,85 mm) screen. Groundwood and high yield pulps containing a significant amount of resins shall be extracted with acetone according to ISO 14453 before testing.

NOTE Resins, if not extracted from the pulp prior to analysis, would remain insoluble in acid and be weighed as lignin.

NOTE Acetone is considered an effective solvent for extracting resin from pulp. Dichloromethane and ethanol/benzene (1:2), as specified in other methods, are not recommended due to health hazards. In particular, benzene is a confirmed carcinogen.

Determine the moisture content of the pulp according to ISO 638 by drying a 2-3 g specimen in an oven at 105 ± 3°C to constant weight. If the pulp has to be pre-extracted, the moisture content must be determined on the extracted pulp.

8 Test Specimens

Weigh a test specimen, equivalent to 300 mg to 1,0 g of oven-dried mass of pulp to the nearest 0.1 mg (see NOTE) and transfer to a 200 ml beaker. Make sure that the test specimens taken are representative of the sample received.

NOTE The mass of the test specimen should be such as to provide a minimum of 20 mg of lignin, in the residue remaining after acid hydrolysis, for accurate weighing. The following can be used as a guide to the amount of pulp that should be selected for analysis, based on the lignin content:

> 10% lignin: 300 mg pulp

5-10% lignin: 500 mg pulp

< 5% lignin: 1,0 g pulp

9 Procedure

Run the entire procedure in duplicate.

9.1 Hydrolysis

9.1.1 General

Two hydrolysis procedures are described in this Standard. In procedure A,[7-9] the primary hydrolysis is performed at 30°C for 1 h, followed by secondary hydrolysis in an autoclave at 120°C for 1 h. In procedure B,[10,11] the primary hydrolysis is done at 15-20°C for 2 h ours, followed by secondary hydrolysis in a water bath at 100°C for 4 h .

Both procedures have been shown to give the same results[7]; thus either one can be used for determining acid-insoluble lignin. However, procedure A is considerably more rapid, and the use of an autoclave allows multi-samples to be hydrolysed simultaneously with minimum supervision. As such, it is now more commonly-used in laboratories equipped with an autoclave. It is therefore the preferred method, particularly when analysis of carbohydrates is required, in addition to the determination of lignin.