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Pulps — Determination of lignin content — Acid hydrolysis method

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

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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 is applied in order to achieve a target brightness.

Comprehensive textbooks and reviews have been written on methods of lignin determination[1]-[3]. This document 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 of the pulp. 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 document. 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. In procedure A, the use of 4 % sulfuric acid, instead of 3 %, for secondary hydrolysis has no impact on the lignin analysis and is accepted when both lignin and carbohydrates need to be analysed in the same sample.

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 multiple 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 and should be used 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 wood 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
class of complex, organic macromolecules, containing aromatic sub-units, that play a key role in the formation of cell walls in wood and bark, conferring mechanical strength and rigidity to cell walls and to plants as a whole

Note 1 to entry: 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* (3.1) which is soluble during the acid-insoluble lignin determination