



BSI Standards Publication

Pulps — Determination of lignin content — Acid hydrolysis method

National foreword

This British Standard is the UK implementation of [ISO 21436:2020](#).

The UK participation in its preparation was entrusted to Technical Committee PAI/11, Methods of test for paper, board and pulps.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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© The British Standards Institution 2020
Published by BSI Standards Limited 2020

ISBN 978 0 580 94703 2

ICS 85.040

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2020.

Amendments/corrigenda issued since publication

Date	Text affected
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INTERNATIONAL STANDARD

ISO
21436

First edition
2020-12-02

Pulps — Determination of lignin content — Acid hydrolysis method

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



Reference number
ISO 21436:2020(E)
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Foreword

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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.