



BSI Standards Publication

Lignins — Determination of inorganics content in kraft lignin, soda lignin and hydrolysis lignin

National foreword

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**Lignins — Determination of
inorganics content in kraft lignin,
soda lignin and hydrolysis lignin**

*Lignines — Détermination de la teneur en matières inorganiques
dans la lignine kraft, la lignine soude et la lignine d'hydrolyse*

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Reagents	2
6 Apparatus	3
7 Sampling	4
8 Procedure	4
8.1 Overview	4
8.2 General	4
8.3 Measurement of dry matter content	4
8.4 Incineration	4
9 Determination of inorganics content	5
9.1 General	5
9.2 Dissolution of the residue	5
9.3 Preparation of calibration solutions	6
9.4 Blank solution	6
9.5 Determination	6
10 Expression of results	7
10.1 Inorganics content of kraft lignin (acid form), soda lignin, and hydrolysis lignin	7
10.2 Inorganics content of kraft lignin in the base form	7
11 Precision	8
12 Test report	8
Annex A (informative) Precision	9
Bibliography	12

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

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Introduction

This document describes methods for the determination of the total inorganics content of kraft lignin, soda lignin and lignin obtained from hydrolysis of biomass.

The inorganics content of lignins is of high significance in several applications of lignin as a replacement of fossil-based raw materials, in products such as solid fuels, phenolic resins, polyurethane foams, thermoplastics, carbon fibre and many others. For example, when lignin is used as a fuel in the lime kiln, the ash content must be very low since it can cause bridging and ringing problems in the lime kiln^[5]. Similarly, when lignin is used as a feedstock to make carbon fibres, it is recommended that the ash content be less than a mass fraction of 0,1 %^[6]. In the case of resole-type phenolic resins, the lignin should be in the base form (i.e. have a high inorganics content) since the process of making such resins is conducted at an alkaline pH^[7].

For kraft lignin in the acid form, soda lignin, and hydrolysis lignin, the ash content determined at 525 °C is a good approximation of the total inorganics content of the samples. However, for kraft lignin in the base form, the ash content significantly overestimates - by a factor of 2 to 3 - the inorganics content of lignin. This is largely due to the fact that, upon combustion, sodium and other metals associated with the phenolic and carboxylic acid groups in lignin are converted to sodium/metal sulfate, nitrate and especially carbonate, depending on the relative ratio of sulfur, nitrogen, and carbon in the lignin. In other words, the ashing process leads to the formation of chemical species (e.g. sulfate, nitrate, carbonate) that were not present in the lignin itself, thereby contributing to an overestimation of the inorganics content of the lignin since essentially all organic matter is destroyed at that temperature.

This phenomenon was demonstrated by establishing a sodium mass balance for kraft lignin in the base form. The amount of sodium ions needed to balance the phenolic and carboxylic acid groups was calculated, based on their respective pKa values^[8]. Good agreement was found between the total sodium and the sodium needed to balance the phenolic and carboxylic acid groups. This shows that most of the sodium in kraft lignin in the base form is present as the salt of the phenolic and carboxylic acid groups.

Thus, for kraft lignin in the base form, the total inorganics content is best determined from an analysis of sodium, along with other major inorganic elements, including calcium, potassium, magnesium, iron, manganese, and copper. The level of trace elements such as zinc, cadmium, and chromium is considered to be too low to have a significant impact on the total inorganics content.

For kraft lignin in the acid form, soda lignin, and hydrolysis lignin, the method used for determining ash content is largely based on ISO 1762^[1]. For kraft lignin in the base form, the method used for determining major inorganic elements is based on ISO 12830^[2].