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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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Foreword

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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].

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

1 Scope

This method describes procedures for the determination of inorganics content in kraft, soda, and hydrolysis lignin. The method is applicable to lignin isolated from a kraft pulping process, a soda pulping process, or lignin obtained by hydrolysis of biomass.

For kraft lignin in the acid form, soda lignin, and hydrolysis lignin, the inorganics content is determined from the ash content of the sample. For kraft lignin in the base form, the inorganics content is determined from the sum of the contents of calcium, magnesium, manganese, iron, copper, sodium and potassium.

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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6350, *Lignins – Determination of dry matter content – Oven-drying and freeze-drying methods*

3 Terms and definitions

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

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

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

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

3.1
lignins
class of complex organic macromolecules, containing aromatic sub-units, that plays 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

Note 1 to entry: Lignin is the main non-carbohydrate constituent of wood.

3.2
kraft lignin
depolymerized and chemically modified lignin isolated from a kraft pulping process, such as that originating from kraft black liquor

3.3
soda lignin
depolymerized and chemically modified lignin isolated from a soda pulping process, such as that originating from soda liquor

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3.4
hydrolysis lignin
lignin produced by conversion of biomass, through enzymatic or acid hydrolysis, into sugars and lignin streams, followed by separation of the lignin fraction

3.5
biomass
biological material derived from living, or previously living organisms, such as wood, agricultural crops and other plant-based biodegradable material

3.6
residue on ignition
ratio of the mass of the residue remaining after a test specimen of lignin is ignited at 525 °C ± 25 °C to the oven-dry mass of the test specimen before ignition

3.7
inorganics content
content of chemical elements or compounds that lack carbon–hydrogen bonds

4 Principle

For kraft lignin in the acid form, soda lignin, and hydrolysis lignin, the test specimen is weighed in a heat-resistant crucible and ignited in a muffle furnace at 525 °C ± 25 °C. The dry matter content of a separate test specimen is also measured. The percentage ash is then determined, on a dry (moisture-free) basis, from the mass of residue after ignition and the dry matter content of the sample. The ash content represents the total inorganics content of the sample.

For kraft lignin in the base form, the test specimen is ashed at 525 °C ± 25 °C, as described for kraft lignin in the acid form. The residue after ashing is then dissolved in 6 mol/l hydrochloric acid, and the sodium, calcium, potassium, magnesium, iron, manganese, and copper in the resulting solution are determined by Inductively Coupled Plasma Emission Spectrometry (ICP/ES). The inorganics content is estimated from the sum of the content of these seven elements. Trace elements, such as zinc, cadmium and chromium, are not included, since their levels are considered to be too low to have a significant impact on the total inorganics content.

NOTE 1 Other techniques or instrumentation other than ICP/ES, such as ICP-mass spectrometry (ICP/MS) or atomic absorption spectrometry (AAS), can also be used provided that they have been properly validated.

NOTE 2 Only the acid-soluble form of metals is determined by this method. If acid-insoluble metals are also present, as would be the case if combined with silicates, they can be determined after digestion or fusion as described in ISO 17812[3]. However, in most cases, the level of silicates is considered to be too low to have a significant impact on the total inorganics content.

NOTE 3 For some applications of kraft lignin in the acid form, soda lignin, or hydrolysis lignin, analysis of individual elements can also be required. In such cases the ash is dissolved in hydrochloric or nitric acid and the concentration of each element in the resulting solution is determined as specified for kraft lignin in the base form.

5 Reagents

5.1 General

All chemicals shall be of reagent grade or better unless otherwise indicated. Water shall be distilled or deionized, of grade 2 or better in accordance with ISO 3696.

5.2 Hydrochloric acid (HCl), about 6 mol/l, trace metal grade. Dilute 500 ml of concentrated hydrochloric acid (specific gravity 1,19 g/ml) to 1 000 ml with water.

5.3 Nitric acid (HNO_3), concentrated (specific gravity 1,4 g/ml), trace metal grade.

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5.4 Standard stock solutions of each element, commercially available certified atomic emission standard solutions can be used. Standard stock solutions can also be prepared as follows:

5.4.1 Magnesium, 1 000 mg/l standard solution. Dissolve 1,000 g of magnesium metal ribbon in 100 ml of a volume fraction of 25 % nitric acid (5.3) and dilute to 1 000 ml with water.

5.4.2 Calcium, 1 000 mg/l standard solution. Dissolve 2,497 g of primary standard calcium carbonate ($CaCO_3$) in a minimum volume fraction of 25 % nitric acid (5.3) and dilute to 1 000 ml with water.

5.4.3 Manganese, 1 000 mg/l standard solution. Dissolve 1,000 g of manganese metal strip or wire in a minimum volume fraction of 100 % nitric acid (5.3) and dilute to 1 000 ml with water.

5.4.4 Iron, 1 000 mg/l standard solution. Dissolve 1,000 g or iron metal strip or wire in 20 ml of hydrochloric acid (5.2) and dilute to 1 000 ml with water.

5.4.5 Copper, 1 000 mg/l standard solution. Dissolve 1,000 g of copper metal strip or wire in a minimum volume fraction of 100 % nitric acid (5.3) and dilute to 1 000 ml with water.

5.4.6 Sodium, 1 000 mg/l standard solution. Ignite a portion of anhydrous sodium sulfate (Na_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 3,089 g of dried sodium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

5.4.7 Potassium, 1 000 mg/l standard solution. Ignite a portion of anhydrous potassium sulfate (K_2SO_4) at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Dissolve 2,228 g of dried potassium sulfate in water and dilute to 1 000 ml with water. Store in a polyethylene bottle.

5.5 Carrier gas, appropriate for the inductively coupled plasma spectrometer. Argon is usually recommended as a carrier gas.

6 Apparatus

6.1 Drying oven, capable of maintaining the air temperature at 105 °C ± 2 °C, and suitably ventilated.

6.2 Heat-resistant crucibles, made of platinum, porcelain or silica, with a capacity of 50 ml to 100 ml.

Larger-capacity crucibles may also be used for low-density materials to accommodate sufficient sample.

A lid of an appropriate material, placed slightly ajar to allow entry of air for combustion, may also be used with the crucible to help prevent low-density or flyaway material from escaping during the ash ignition process.

Platinum crucibles are recommended if a small amount of residue is expected.

6.3 Muffle furnace, capable of maintaining a temperature of 525 °C ± 25 °C. It is recommended that the furnace be placed in a hood or that means be provided for evacuating smoke and fumes.

6.4 Analytical balance, with a scale division (readability) of 0,1 mg or better in order to obtain a measurement precision of 0,01 % or better.

6.5 Desiccator, using Drierite™ [1] or equivalent desiccant.

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1) Drierite™ is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

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6.6 Inductively coupled plasma/emission spectrometer (ICP/ES), for determination of inorganics content in kraft lignin in the base form only and if determination of individual elements is performed on kraft lignin in the acid form, soda lignin, or hydrolysis lignin

6.7 Freeze-dryer, (only if freeze-drying is used for the determination of moisture content) typically available with condenser refrigeration of −80 °C, and capable of maintaining a pressure (vacuum) of about 3,33 Pa (25 mtorr).

7 Sampling

Obtain a representative sample of lignin equivalent to about 2 g to 3 g on an air-dry basis. Report the origin of the sample and the sampling procedure. For example, in the case of kraft lignin samples, it shall be reported whether they were collected in their base form, or after acid-washing; or as they come out of the press, partly dried, flash dried, or otherwise.

If the sample is not analysed immediately after collection, it shall be stored in an airtight container or sealable polyethylene bag. If it is necessary to store the samples for longer than 2 days to 3 days, they shall be kept in a refrigerator or cold room at 5 °C ± 1 °C prior to use. Samples shall be brought back to room temperature before opening the container or bag.

NOTE Larger amounts of sample are recommended if the analysis of minor elements, such as manganese, iron, or copper, is required.

8 Procedure

8.1 Overview

8.2 to 8.4 apply to all types of lignin. For kraft lignin in the acid form, soda lignin, and hydrolysis lignin, the inorganics content is determined from the measurement of the residue on ignition, as described in 8.4.2 and 10.1. However, for kraft lignin in the base form, the inorganics content is determined from the analysis of individual elements, as described in 9.5 and 10.2.

8.2 General

Carry out the procedure in at least duplicate. Allow wet test specimens and dry matter specimens to air-dry, and condition other test specimens, under dust-free conditions in the ambient laboratory air until they reach equilibrium moisture.

8.3 Measurement of dry matter content

Determine the dry matter content of test specimens (air-dry) using the procedure described in ISO 6350. Although freeze-drying is preferred over oven-drying for determination of the moisture content in order to preserve the integrity of the sample, oven-drying may be used for all types of lignin. Do not use the dry matter content test specimen for incineration. Weigh the crucible containing the dry matter content test specimen at the same time as the crucible containing the test specimen (air-dry) used for incineration.

8.4 Incineration

8.4.1 If a lid is used to prevent material from escaping during the ash ignition process, it shall be considered as an integral part of the crucible and weighed together with the crucible. The lid shall also be closed before weighing.

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8.4.2 Heat the empty crucible (6.2) for 30 min to 60 min in the muffle furnace (6.3) at 525 °C ± 25 °C. Cool it to room temperature in a desiccator (6.5).

Weigh the empty crucible to the nearest 0,1 mg with an analytical balance (6.4). Add approximately 1 g of the test specimen and immediately weigh again to the nearest 0,1 mg. Place the crucible containing the test specimen in the furnace at room temperature and gradually raise the temperature to 525 °C (about 200 °C/h) in order to burn the sample without it bursting into flames, and to ensure that no material is lost in the form of flying particles.

NOTE Larger amounts of test specimen are recommended if minor elements, such as manganese, iron or copper, are included in the analysis

Maintain the ignition temperature of 525 °C for at least 2 h. If black particles remain, repeat the ashing process for another hour. The specimen shall be completely charred as indicated by the absence of black particles.

If a considerable amount of black particles remains after ashing for 2 h, as is likely to be the case for kraft lignin in the base form, add a few drops of water to the residue, shake gently to expose more surface and place back in the furnace at 525 °C for another 1 h to 2 h (or longer if necessary) until the test specimen is completely ashed, as indicated by the absence of black particles.

8.4.3 Measurement of residue (ash) mass

Remove the crucible from the furnace and allow it to attain room temperature in a desiccator (6.5). Weigh the crucible to the nearest 0,1 mg.

The inorganics content in kraft lignin in the acid form, soda lignin, and hydrolysis lignin is determined from the percentage ash in the sample as described in 10.1.

In the case of kraft lignin in the base form, the inorganics content is determined as described in 9.5 and 10.2.

9 Determination of inorganics content

9.1 General

In the case of kraft lignin in the base form, the ash content significantly overestimates - by a factor of 2 to 3 - the inorganics content of lignin. Therefore, 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.

NOTE The procedure described in this section can also be applied to kraft lignin in the acid form, soda lignin, and hydrolysis lignin if the analysis of individual elements is required for these types of lignins.

9.2 Dissolution of the residue

Carefully moisten the residue of incineration from 8.4.2 with water and add cautiously, in a fume hood, 5 ml of hydrochloric acid (5.2) or nitric acid (5.3) to the crucible. Evaporate to dryness on a boiling-water bath, hot plate or infrared lamp. Repeat this procedure to facilitate dissolution of the residue.

Add another 10 ml of hydrochloric acid (5.2) to the residue. Transfer quantitatively with water to a 100 ml volumetric flask. Dilute to 100 ml.

Nitric acid should be used if complete dissolution is not achieved with HCl.

NOTE Microwave digestion with nitric acid[9][10] can also be used to replace the incineration step and for complete dissolution of the sample, provided that the results have been validated.

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9.3 Preparation of calibration solutions

Prepare at least three different concentrations of calibration solution for each element in 100 ml volumetric flasks. Add 10 ml of hydrochloric acid (5.2) or 10 ml of nitric acid (5.3), using the same acid as that used to dissolve the residue after ashing, to the calibration solutions before diluting the corresponding standard stock solution to 100 ml with water.

9.4 Blank solution

A blank solution shall be prepared, omitting the test element and containing the same amount of hydrochloric acid (5.2) or nitric acid (5.3) as in the calibration solution, according to the acid used to dissolve the residue after ashing in 9.2.

9.5 Determination

For each element to be determined, optimize the conditions of the ICP/ES spectrometer and operate the instrument as recommended by the manufacturer.

The commonly recommended emission lines[11] are as follows:

— magnesium: 279,55 nm;

— calcium: 315,887 nm;

— manganese: 257,61 nm;

— iron: 259,94 nm;

— copper: 324,75 nm;

— sodium: 589,00 nm;

— potassium: 766,50 nm.

NOTE 1 For these recommended emission lines, the following interferants have been reported[9]: cerium for magnesium; cobalt, molybdenum, and cerium for calcium; cerium for manganese; none for iron; molybdenum and titanium for copper; none for sodium; and none for potassium. All of these potential interferants would be either absent or present at trace levels in lignin samples, and thus would not affect the accuracy of the measurements.

NOTE 2 Other emission lines than those listed have also been reported as being suitable for the analysis of these elements. Such emission lines can be used, provided that they are of sufficient sensitivity for the elements of interest and are free from interferants.

Carry out the measurement of the calibration solutions, the test solution and the blank solution. If the reading of the test solution is outside the range of the calibration curve, corrected for the blank, dilute with water and the appropriate amount of acid to bring it within this range. All final dilutions of the test solution shall contain the same acid concentration [10 ml/100 ml hydrochloric acid (5.2) or 10 ml/100 ml nitric acid (5.3)] as the corresponding calibration solution. Record the dilution factor.

If the test solution is used without dilution, then further addition of hydrochloric (5.2) or nitric acid (5.3) is not necessary since the dilution already contains acid added after the ashing step.

Once the test solution is within the calibration range, determine the concentration of the element in the solution by referring to the appropriate calibration curve.

In microprocessor-controlled spectrometers, the concentration is determined automatically and plotting of calibration curves is thus not required.

The inorganics content is determined as described in 10.2.

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10 Expression of results

10.1 Inorganics content of kraft lignin (acid form), soda lignin, and hydrolysis lignin

For each crucible from 8.4.3, calculate the percentage ash content on ignition using Formula (1):

$$X = \frac{100\ m_r}{m_s} \quad (1)$$

where

X is the ash content, as a percentage of the mass of the test specimen on an oven-dry or freeze-dried basis;

m_r is the mass of the residue (the mass of the crucible with the residue, minus that of the empty crucible) in grams (g);

m_s is the mass of the test specimen, on an oven-dry or freeze-dried basis, in grams (g). This is determined from the average of the replicate dry matter content determinations.

Check that there is reasonable agreement between the replicate measurements. For test specimens with ash contents above 0,1 %, reasonable agreement exists if the deviation of results from the mean of parallel determinations does not exceed 10 % of the mean value. If this is not the case, repeat the entire procedure with new test specimens, preferably of larger mass.

Calculate the mean ash content. Express the mean to the nearest 0,1 % for samples with ash content above 1 %, and to the nearest 0,01 % or smaller for samples with ash content below 1 %. If the mean ash content is below 0,1 %, it may be reported as "below 0,1 %", or the individual results of replicate determinations may be reported.

The ash content represents the inorganics content of the sample.

10.2 Inorganics content of kraft lignin in the base form

Calculate the mass fraction of each element in the test specimen using Formula (2):

$$m_e = d \cdot c_e \cdot V / m \quad (2)$$

where

m_e is the mass fraction of the particular element, in the sample, in mg/kg;

d is the dilution factor from 9.5, if any;

c_e is the concentration of the particular element in the test solution, corrected for the blank, as obtained from the calibration plot in 9.5 in mg/l;

V is the volume of solution from 9.2, in ml;

m is the mass of the test specimen used for ignition from 8.4.2, on an oven-dry or freeze-dried basis, in grams (g).

The sum of calcium, magnesium, manganese, iron, copper, sodium and potassium, in percent of the test specimen, represents the inorganics content of the sample.

Calculate and report the ash content of the sample as described in 10.1, along with the inorganics content.

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11 Precision

The precision of the method was determined by conducting a round robin study with several types of lignin samples. A description of the samples used in this study, and the repeatability and reproducibility results are presented in Annex A

12 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 9795:2023;

b) date and place of testing;

c) complete identification of the sample tested, as indicated in Clause 7;

d) type of acid used for dissolution of the residue;

e) the result, expressed as indicated in Clause 10;

f) any departure from the procedure described in this document or any other circumstances which could have affected the result.

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Annex A
(informative)

Precision

A.1 General

In January 2022 an international round robin study was performed in which twelve laboratories from eight countries - Brazil; Belgium; Canada (3 laboratories); Finland; France; Japan; Sweden (2 laboratories) and the U.S. (2 laboratories) - participated.

A total of four samples representing different types of lignins were included in the study, including a kraft softwood lignin, acid form, air-dried; a kraft hardwood lignin, base form, air-dried; a hydrolysis hardwood lignin, flash-dried; and a soda lignin, acid form, flash-dried. The samples were submitted to the participating laboratories for testing according to this document.

All samples were shipped in sealed vials to prevent moisture uptake. If the samples could not be analysed upon receipt, participants were requested to store them in a cold room or refrigerator until the day of analysis.

Participants were also instructed to obtain test specimens directly from the test vials, and test them as-is, without air-drying or further conditioning. Sample vials had to be resealed immediately after collecting the test specimens to prevent moisture uptake.

The concentration of individual elements was determined only on the hardwood kraft lignin in the base form. Four laboratories used microwave digestion instead of dry ashing to solubilize the sample prior to elements analysis.

Repeatability and reproducibility data for ash content are shown in Tables A.1 and A.3, respectively, and for Na, Ca, K, Mg, Fe, Mn, Cu, and total inorganics in Tables A.2 and A.4, respectively. The calculations were made in accordance with ISO/TS 24498[4].

The repeatability and reproducibility limits reported are estimates of the maximum difference which would be expected in 19 instances of 20 instances, when comparing two test results for materials similar to those described under similar test conditions. These estimates might not be valid for different materials or different test conditions.

NOTE 1 Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where 2,77 = 1,96 √2.

NOTE 2 In a number of cases, the number of laboratories listed in Tables A.1 to A.4 is smaller than 12. This can be due to the fact that ashing was incomplete, the reported results were considered as outliers and were therefore not included in the average, or results were not reported for that test.

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A.2 Repeatability

Table A.1 — Estimation of the repeatability of the test for ash content

Type of lignin	Number of laboratories	Ash mean %	Standard deviation S_r %	Coefficient of variation C_V %	Repeatability limit r %
Kraft softwood	12	0,75	0,074	9,9	0,20
Kraft hardwood	8[a]	19,5	0,12	0,62	0,33
Hydrolysis hardwood	11[b]	0,80	0,034	4,2	0,094
Soda	11[c]	1,49	0,057	3,8	0,16

[a] 2 labs were outliers; 2 labs reported incomplete ashing.

[b] 1 lab was an outlier.

[c] 1 lab was an outlier.

Table A.2 — Estimation of the repeatability of elements analysis for the kraft hardwood sample

Element	Number of laboratories	Mean mg/kg	Standard deviation S_r mg/kg	Coefficient of variation C_V %	Repeatability limit r mg/kg
Sodium	8[a]	67 220	856	1,3	2 371
Calcium	9[b]	440	13,7	3,1	37,9
Potassium	7[c]	9 647	118	1,2	327
Magnesium	8[a]	399	3,34	0,84	9,25
Iron	8[a]	96,9	2,35	2,4	6,51
Manganese	8[a]	33,5	0,28	0,84	0,78
Copper	7[d]	4,96	0,18	3,6	0,50
Total	8[a]	77 980	830	1,1	2 299

[a] 3 labs did not report elements analysis; 1 lab was an outlier.

[b] 3 labs did not report elements analysis.

[c] 3 labs did not report elements analysis; 2 labs were outliers.

[d] 3 labs did not report elements analysis; 1 lab reported not detected; 1 lab was an outlier.

A.3 Reproducibility

Table A.3 — Estimation of the reproducibility of ash content

Type of lignin	Number of laboratories	Mean mg/kg	Standard deviation S_r mg/kg	Coefficient of variation C_V %	Repeatability limit r mg/kg
Kraft softwood	12	0,75	0,099	13,2	0,27
Kraft hardwood	9[a]	19,4	0,68	3,5	1,88
Hydrolysis hardwood	9[b]	0,76	0,030	3,9	0,083
Soda	11[c]	1,49	0,074	5,0	0,20

[a] 1 lab was an outlier; 2 labs reported incomplete ashing.

[b] 3 labs were outliers.

[c] 1 lab was an outlier.

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