

RISE Biorefinery Proficiency Test 2023 Chemical analysis of pulps, process liquids and lignin

What is the quality of your routine analyses? Are your procedures fit for purpose? Participating in proficiency tests is a convenient way to find out! RISE annually organizes tests for a number of chemical analyses used in pulp, paper and packaging applications. Also, this year, we are broadening the service by adding a third round for lignin samples.

Proficiency tests are interlaboratory comparisons that give you regular and objective determination of the accuracy of your analyses. It also assesses the quality of your routine work, stimulates technical improvement, and offers comparative overviews of the methods you are using. For accredited laboratories, partitioning in proficiency tests is an essential part of the quality management system.

Since several years, RISE annually organizes comparative testing for a number of chemical analyses used in pulp and paper production and control. Up to 50 laboratories take part in the testing of different properties. In order to arrange a statistically relevant comparison at least five laboratories are required. The results are compiled in a report, having the identity of the participating laboratories coded. At least three rounds are offered annually. In Round 1, the tests include analyses for pulp, in Round 2 liquid samples like white liquor, black liquor and effluents are distributed and Round 3 will focus on analysis of kraft lignin.

General terms

Information about registration, fees, sample distribution, reporting of results, statistical treatment and handling, confidentiality, *etc.* is appended.

Testing properties

A complete list of testing properties and suggested methods is appended.

Reporting

Please register at the website:

<https://comm.ri.se/b/v?survey=484&ucrc=7A5F34E223>

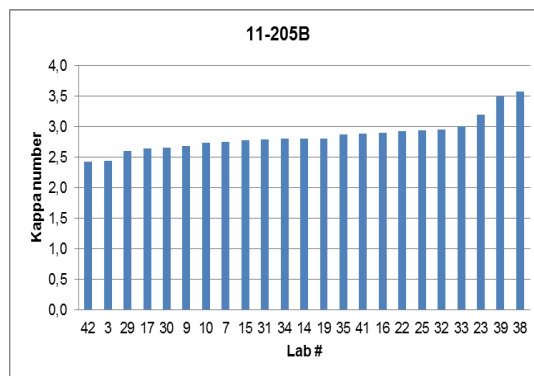
The website for reporting your results will be given when the samples are dispatched. Please note that the ability of your laboratory to report the results on time and using the specified units are considered as a part of the proficiency test, and hence the reporting website will close at the date of the deadline.

Contact

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An example of results. All results are also given in tables together with statistical information, like the assigned value (robust mean), uncertainty of the assigned value (the robust standard deviation), and individual performance scores for all participants (z-scores).



Sample preparation



GENERAL TERMS

Registration and fees

To register for one or several parameters, please visit our website. Upon registration, you will be assigned with a laboratory participation code (*i.e.* a unique lab ID) that should be used when reporting the results. The fees are stated in the information given for each round. For participants that order all samples in a round, a 10% discount will be given. Please note that the invoices will be sent out the same month as the sample dispatch.

Sample distribution

The samples will be dispatched in accordance with the information given for each round. Please note that overseas shipping may delay the delivery.

Sample handling and treatment

The received samples should be handled and treated according to the normal routines on your laboratory. Avoid giving the samples extra attention (extra analyses, etc.) since this test then will not assess your routines.

Suggested methods

In order to assess the quality of your routine work, you should use the procedures that are normally used in your laboratory. We do however always recommend procedures based on standard methods, and in order to assess differences in reported values, we encourage you to specify any deviations between your procedure and the standard method.

Reporting results

Only one value should be reported per tested property (for each sample). For instance, if your procedure states that the reported value should be the average of duplicate measurement, only the average should be reported. The values should be reported in the units given in the information for each test, and the reporting basis (*i.e.* “per dry mass” or “as received”) should be the same as in the suggested method.

Please also note that the ability of your laboratory to report the results no later than the deadline and in the specified units are considered as a part of the proficiency test, and hence the reporting website will close at the date of the deadline.

Statistical treatment and performance evaluation

For each property, an assigned value (robust average) will be calculated using robust statistics (Algorithm A in ISO 13528). By using this method, outliers do not need to be removed since all values are scaled based on their deviation from the median value. By using the same algorithm, the uncertainty of the assigned value (robust standard deviation) will be determined.

Subsequently, each participant will be assigned a performance score (z-score) for each tested property based on the deviation from the assigned value and the uncertainty of the assigned value.

Confidentiality

All information supplied by participants is considered confidential. The identity of your laboratory will be confidential and known only to the persons involved in organizing the proficiency test.

Proficiency testing reports

One report will be issued for each round. The reports will be published within one month from the deadline of reporting results and will contain the laboratory participation codes and all results, including assigned values and performance scores.

Reference documents

- ILAC-G13:08/2007 – ILAC guidelines for the requirements for the competence of providers of proficiency testing schemes
- ISO 13528:2022 – Statistical methods for use in proficiency testing by interlaboratory comparisons



2023 – ROUND 1 – PULP SAMPLES

The deadline to register for this round is on 21 July, 2023. These pulp and paper samples will be distributed on 8 September, 2023, and the deadline for reporting the results is 27 October, 2023. Approximately 100 g will be sent of each pulp and paper. At least five participating laboratories are required for each test.

The price for participating in this round is SEK 3250/sample plus SEK 300 for the individual tests (*ie.* the price for participating in one test in one sample is SEK 3550). For participants that order all three samples, a 10% discount will be given.

Sample 2023-1A – Kraft pulp I (unbleached), SEK 3250

Test ID	Test	Price for test (SEK)
23-1A-01	Acetone-soluble matter	+300
23-1A-02	Acidic groups	+300
23-1A-06	Ash at 525 °C	+300
23-1A-07	Carbohydrates	+300
23-1A-09	COD and TOC	+300
23-1A-10	Kappa number	+300
23-1A-11	Lignin	+300
23-1A-12	Metals	+300
23-1A-13	pH	+300
23-1A-14	Sodium and potassium	+300
23-1A-15	Sulphur	+300

Sample 2023-1B – Kraft pulp II (bleached), SEK 3250

Test ID	Test	Price for test (SEK)
23-1B-02	Acidic groups	+300
23-1B-06	Ash at 525 °C	+300
23-1B-08	Chlorine	+300
23-1B-09	COD and TOC	+300
23-1B-10	Kappa number	+300
23-1B-13	pH	+300
23-1B-16	Viscosity	+300

Sample 2023-1C – Sulphite pulp, SEK 3250

Test ID	Test	Price for test (SEK)
23-1C-01	Acetone-soluble matter	+300
23-1C-03	Acid-insoluble ash	+300
23-1C-04	Alkali resistance	+300
23-1C-05	Alkali solubility	+300
23-1C-07	Carbohydrates	+300
23-1C-11	Lignin	+300
23-1C-12	Metals	+300
23-1C-14	Sodium and potassium	+300
23-1C-15	Sulphur	+300
23-1C-16	Viscosity	+300

Properties in the tests included in Round 1 – Pulp samples

Test	Properties	Suggested methods	Unit	Notes
Acetone soluble matter	Acetone-soluble matter	SCAN-CM 49 or ISO 14453	%	
Acidic groups	Total acidic group content	SCAN-CM 65	µmol/g	
Acid-insoluble ash	Acid-insoluble ash	ISO 776	mg/kg	
Alkali resistance	Alkali resistance (R18)	ISO 699	%	
Alkali solubility	Alkali solubility (S18)	ISO 692	%	
Ash	Residue (ash) on ignition at 525 °C	ISO 1762	%	
Ash	Residue (ash) on ignition at 900 °C	ISO 2144	%	
Carbohydrates	Arabinose (Ara)	SCAN-CM 71	mg/g	1
	Galactose (Gal)	SCAN-CM 71	mg/g	1
	Glucose (Glc)	SCAN-CM 71	mg/g	1
	Mannose (Man)	SCAN-CM 71	mg/g	1
	Xylose (Xyl)	SCAN-CM 71	mg/g	1
Chlorine	Total chlorine	SCAN-CM 51 or ISO 11480	mg/kg	
	Organically bound chlorine	SCAN-CM 52 or ISO 11480	mg/kg	
COD and TOC	Chemical oxygen demand (COD) removable by washing	SCAN-CM 44	kg/ton	
	Total organic carbon (TOC) removable by washing	SCAN-CM 44	kg/ton	
Glyoxal	Glyoxal in cold water extract	EN 645 and DIN 54603	mg/kg	
Kappa number	Kappa number	ISO 302	–	
Lignin	Acid-insoluble lignin (Klason)	TAPPI T 222 om	mg/g	2
	Acid-soluble lignin	TAPPI UM 250	mg/g	2
Metals	Acid-soluble magnesium (Mg)	SCAN-CM 38 or ISO 12830	mg/kg	
	Acid-soluble calcium (Ca)	SCAN-CM 38 or ISO 12830	mg/kg	
	Acid-soluble manganese (Mn)	SCAN-CM 38 or ISO 12830	mg/kg	
	Acid-soluble iron (Fe)	SCAN-CM 38 or ISO 12830	mg/kg	
	Acid-soluble copper (Cu)	SCAN-CM 38 or ISO 12830	mg/kg	
	Acid-soluble silicon (Si)	Method of choice	mg/kg	
	Acid-soluble aluminium (Al)	Method of choice	mg/kg	
pH	pH of aqueous extract – cold extraction	ISO 6588-1	–	
	pH of salted water extract	ISO 29681	–	
Sodium and potassium	Acid-soluble sodium (Na)	SCAN-CM 63 or ISO 12830	mg/kg	
	Acid-soluble potassium (K)	SCAN-CM 63 or ISO 12830	mg/kg	
Sulphur	Total sulphur	SCAN-CM 57	mg/kg	
Viscosity	Limiting viscosity number in cupri-ethylenediamine (CED) solution	ISO 5351	mL/g	

Notes

- 1) Please report as anhydrous monosaccharide (*ie.* corrected with an anhydrous factor), but do not correct for possible monosaccharide degradation (*ie.* corrected with recovery/hydrolysis factor).
- 2) Other unit than in the standard.

Please also note that SCAN-test methods are available for free! Please contact Christine Allansson (christine.allansson@sis.se) for more information.



2023 – ROUND 2 – LIQUORS AND EFFLUENTS

The deadline to register for this round is on July 21, 2023. These liquors and effluent will be distributed on 15 September, 2023, and the deadline for reporting the results is 3 November, 2023. Approximately 250 mL will be sent of each liquid.

The price for participating in this round is SEK 3250/sample plus SEK 300 for the individual tests (*ie.* the price for participating in one test in one sample is SEK 3550). For participants that order all three samples, a 10% discount will be given.

Sample 2023-2A – White liquor, SEK 3250

Test ID	Test	Price for test (SEK)
23-2A-18	Hydrogen sulphide	+300
23-2A-23	Sulphur	+300
23-2A-27	Total, active and effective alkali	+300

Sample 2023-2B – Black liquor, SEK 3250

Test ID	Test	Price for test (SEK)
23-2B-18	Hydrogen sulphide	+300
23-2B-19	Metals	+300
23-2B-21	Residual alkali	+300
23-2B-22	Sodium and potassium	+300
23-2B-23	Sulphur	+300
23-2B-24	TOC	+300
23-2B-30	Dry matter content	+300

Sample 2023-2C – Bleaching effluent, SEK 3250

Test ID	Test	Price for test (SEK)
23-2C-17	Chlorates	+300
23-2C-19	Metals	+300
23-2C-20	pH	+300
23-2C-22	Sodium and potassium	+300
23-2C-24	TOC	+300
23-2C-25	COD	+300
23-2C-26	Oxalic acid	+300

Properties in the tests included in Round 2 – Liquors and effluents

Test	Properties	Suggested methods	Unit	Notes
Chlorates	Chlorates	SCAN-W 10	mg/L	
Dry matter content	Dry matter content	SCAN-N 22	%	
Hydrogen sulphide	Hydrogen sulphide ion concentration	SCAN-N 31	mol/L	
Metals	Acid-soluble calcium (Ca)	SCAN-N 38	mg/L	1
	Acid-soluble magnesium (Mg)	SCAN-N 38	mg/L	1
	Acid-soluble iron (Fe)	SCAN-N 38	mg/L	1
	Acid-soluble manganese (Mn)	SCAN-N 38	mg/L	1
	Acid-soluble aluminium (Al)	SCAN-N 38	mg/L	1
	Acid-soluble silica (Si)	SCAN-N 38	mg/L	1
	Acid-soluble phosphorus (P)	SCAN-N 38	mg/L	1
pH	pH in liquid	Method of choice	–	5
Residual alkali	Residual alkali (Hydroxide ion concentration)	SCAN-N 33	mol/L	
Sodium and potassium	Sodium (Na)	SCAN-N 37	mg/L	1
	Potassium (K)	SCAN-N 37	mg/L	1
Sulphur	Total sulphur	SCAN-N 5	g/L	2
	Total sulphur	SCAN-N 35	g/kg	3
Total, active and effective alkali	Total alkali	SCAN-N 30	mol/L	
	Active alkali	SCAN-N 30	mol/L	
	Effective alkali	SCAN N 30	mol/L	
TOC	Total organic carbon (TOC)	EN 1484	mg/L	4
COD	Chemical oxygen demand (COD)		mg/l	
Oxalic acid	Oxalic acid		mg/l	

Notes

- 1) The scope of the suggested method is black liquors, but please use the suggested method also for effluents.
- 2) Use this suggested method for white liquor.
- 3) Use this suggested method for black liquor (per dry substance).
- 4) The scope of the suggested method is effluents, but please use the suggested method also for black liquors after dilution.
- 5) Please report pH value with at least two digits after the decimal point.

Please also note that SCAN-test methods are available for free! Please contact Christine Allansson (christine.allansson@sis.se) for more information.

**2023 – ROUND 3 – LIGNIN**

The deadline to register for this round is on July 21, 2023. The lignin will be distributed on 15 September, 2023, and the deadline for reporting the results is 3 November, 2023. Approximately 20 g will be sent of the lignin. The price for participating in this round is SEK 3250/sample plus SEK 300 for the individual tests (*ie.* the price for participating in one test is SEK 3550).

Sample 2023-3A – Softwood Kraft Lignin, SEK 3250

Test ID	Test	Price for test (SEK)
23-3A-01	Lignin content	+300
23-3A-02	Carbohydrate content	+300
23-3A-03	Ash content	+300
23-3A-04	Metals	+300
23-3A-05	Glass transition temperature (Tg)	+300
23-3A-06	Phenolic hydroxyl groups	+300

Properties in the tests included in Round 3 – Lignin

Test	Properties	Suggested methods	Unit	Notes
Lignin content	Acid-insoluble lignin (Klason)	Method of choice	mg/g	
Lignin content	Acid-soluble lignin	Method of choice	mg/g	
Carbohydrates	Arabinose (Ara)	Method of choice	mg/g	1
	Galactose (Gal)	Method of choice	mg/g	1
	Glucose (Glc)	Method of choice	mg/g	1
	Mannose (Man)	Method of choice	mg/g	1
	Xylose (Xyl)	Method of choice	mg/g	1
Ash content	Residue (ash) on ignition at 525 °C	Method of choice	%	
Elements	Acid-soluble aluminium (Al)	Method of choice	mg/kg	
	Acid-soluble barium (Ba)	Method of choice	mg/kg	
	Acid-soluble calcium (Ca)	Method of choice	mg/kg	
	Acid-soluble copper (Cu)	Method of choice	mg/kg	
	Acid-soluble iron (Fe)	Method of choice	mg/kg	
	Acid-soluble potassium (K)	Method of choice	mg/kg	
	Acid-soluble magnesium (Mg)	Method of choice	mg/kg	
	Acid-soluble manganese (Mn)	Method of choice	mg/kg	
	Acid-soluble sodium (Na)	Method of choice	mg/kg	
	Acid-soluble phosphorus (P)	Method of choice	mg/kg	
	Acid-soluble sulphur (S)	Method of choice	mg/kg	
	Acid-soluble silica (Si)	Method of choice	mg/kg	
	Acid-soluble zinc (Zn)	Method of choice	mg/kg	
Glass transition temp.	Glass transition temp.	Method of choice	(°C)	
Phenolic hydroxyl groups	Amount of phenolic hydroxyls	Method of choice	mmol/g	

Notes

1) Please report as anhydrous monosaccharide (*ie.* corrected with an anhydrous factor), but do not correct for possible monosaccharide degradation (*ie.* corrected with recovery/hydrolysis factor).

Please note also that no standard methods are available for lignin samples. However, Lignin Test Methods recommended by RISE are available for free! Please contact for more information Anders Reimann at anders.reimann@ri.se.