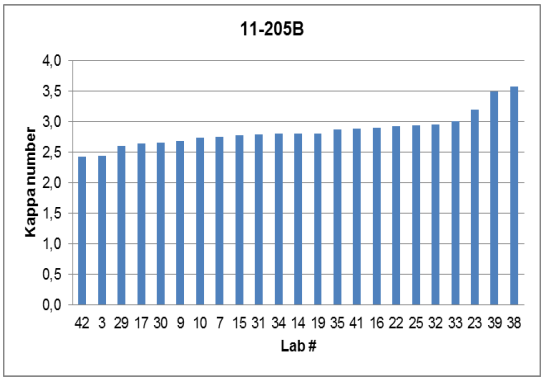
# RISE Biorefinery Proficiency Test 2024
# Chemical analysis of pulps, process liquids and lignin

**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.




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
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
For more information, contact coordinator
Anders Reimann at anders.reimann@ri.se
Phone: +46 768 767 445

RISE
Box 5604, SE-114 86 Stockholm, Sweden
www.ri.se

# 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

## 2024 – ROUND 1 – PULP SAMPLES

The deadline to register for this round is on 21 July, 2024. These pulp and paper samples will be distributed on 8 September, 2024, and the deadline for reporting the results is 27 October, 2024. 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 2024-1A – Kraft pulp I (unbleached), SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-1A-01 | Acetone-soluble matter | +300 |
| 24-1A-02 | Acidic groups | +300 |
| 24-1A-06 | Ash at 525 °C | +300 |
| 24-1A-07 | Carbohydrates | +300 |
| 24-1A-09 | COD and TOC | +300 |
| 24-1A-10 | Kappa number | +300 |
| 24-1A-11 | Lignin | +300 |
| 24-1A-12 | Metals | +300 |
| 24-1A-13 | pH | +300 |
| 24-1A-14 | Sodium and potassium | +300 |
| 24-1A-15 | Sulphur | +300 |

### Sample 2024-1B – Kraft pulp II (bleached), SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-1B-02 | Acidic groups | +300 |
| 24-1B-06 | Ash at 525 °C | +300 |
| 24-1B-08 | Chlorine | +300 |
| 24-1B-09 | COD and TOC | +300 |
| 24-1B-10 | Kappa number | +300 |
| 24-1B-13 | pH | +300 |
| 24-1B-16 | Viscosity | +300 |

### Sample 2024-1C – Sulphite pulp, SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-1C-01 | Acetone-soluble matter | +300 |
| 24-1C-03 | Acid-insoluble ash | +300 |
| 24-1C-04 | Alkali resistance | +300 |
| 24-1C-05 | Alkali solubility | +300 |
| 24-1C-07 | Carbohydrates | +300 |
| 24-1C-11 | Lignin | +300 |
| 24-1C-12 | Metals | +300 |
| 24-1C-14 | Sodium and potassium | +300 |
| 24-1C-15 | Sulphur | +300 |
| 24-1C-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.

## 2024 – ROUND 2 – LIQUORS AND EFFLUENTS

The deadline to register for this round is on July 21, 2024. These liquors and effluent will be distributed on 15 September, 2024, and the deadline for reporting the results is 3 November, 2024. 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 2024-2A – White liquor, SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-2A-18 | Hydrogen sulphide | +300 |
| 24-2A-23 | Sulphur | +300 |
| 24-2A-27 | Total, active and effective alkali | +300 |

### Sample 2024-2B – Black liquor, SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-2B-18 | Hydrogen sulphide | +300 |
| 24-2B-19 | Metals | +300 |
| 24-2B-20 | pH | +300 |
| 24-2B-21 | Residual alkali | +300 |
| 24-2B-22 | Sodium and potassium | +300 |
| 24-2B-23 | Sulphur | +300 |
| 24-2B-24 | TOC | +300 |
| 24-2B-28 | Conductivity | +300 |
| 24-2B-30 | Dry matter content | +300 |

### Sample 2024-2C – Bleaching effluent, SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-2C-17 | Chlorates | +300 |
| 24-2C-19 | Metals | +300 |
| 24-2C-20 | pH | +300 |
| 24-2C-22 | Sodium and potassium | +300 |
| 24-2C-24 | TOC | +300 |
| 24-2C-25 | COD | +300 |
| 24-2C-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 | |
| **Conductivity** | Electrical conductivity | Method of choice | Ω·m | |

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.

## 2024 – ROUND 3 – LIGNIN

The deadline to register for this round is on July 21, 2024. The lignin will be distributed on 15 September, 2024, and the deadline for reporting the results is 3 November, 2024. 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 (*i.e.*. the price for participating in one test is SEK 3550).

### Sample 2024-3A – Softwood Kraft Lignin, SEK 3250

| Test ID | Test | Price for test (SEK) |
|---|---|---|
| 24-3A-01 | Lignin content | +300 |
| 24-3A-02 | Carbohydrate content | +300 |
| 24-3A-03 | Ash content | +300 |
| 24-3A-04 | Metals | +300 |
| 24-3A-05 | Glass transition temperature (Tg) | +300 |
| 24-3A-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.