Chemical analysis of liquors and effluents

The composition of liquor samples and filtrates from the pulp and paper mills are of interest in many different aspects. Analysis of the composition is used both to control and to understand the different process stages. Innventia has long experience and advanced equipment for analysis of both inorganic and organic components in all types of sample from black liquors to effluents.

In order to control the process and to prevent problems, such as e.g. formation of scaling, it is important to be able to measure the concentration of both organic and inorganic compounds in the liquors and filtrates from different positions.

What can we do for you?

We have methods for analysis of all different sample types such as e.g.:

- White liquor and green liquor
- Black liquor and sulphite liquor,
- Condensates
- Bleaching filtrates
- Paper machine white water
- Effluents

For all sample types we possess a range of analytical methods for analysis of:

- Sum parameters
- Metals and elements
- Inorganic anions
- Wood extractives
- Organic components
  - Lignin content
  - Carbohydrates
  - Low molecular weight compounds

A list of analytical services related to aqueous samples is appended. Please contact us for a discussion about how we may help you!

Contact

For more information, please contact Anna Jacobs (anna.jacobs@innventia.com, +46 8 676 7152) or Anders Reimann (anders.reimann@innventia.com, +46 8 676 7445). We look forward to your inquiries!
## ANALYSIS OF LIQUORS AND EFFLUENTS

### Sum parameters

We offer determination of several sum parameters, including:

- Total solids
- Suspended solids
- TOC, COD, Total nitrogen
- pH
- Alkali (total, active, and effective)
- Residual alkali
- Acidic groups content (The sum of carboxylic acids is measured using conductometric titration)

### Extractives

**Extraction methods:**

- **MTBE extraction** is used for effluent samples, especially TMP effluents (includes lignans).
- **Petroleum-ether extraction “Saltsman-Kuiken”** is used for determination of tall oil components in black liquor and also for extractive content in other water samples containing lignin (lignin and lignans are excluded).
- **Solvent of choice** – Several different solvents e.g. diethyl ether or dichloromethane are used for specific analytes.

**Characterization of extracts:**

The lipophilic extract composition may be determined using two different methods.

- **Group separation.** Extracts are silylated and analysed by gas chromatography (GC-FID). The peaks in the GC-chromatogram are quantified in six groups: Fatty acids, resin acids, lignans, sterols, steryl esters and triglycerides, as described in NSP report No 3 (2008).
- **Individual resin compounds.** Extracts are derivatized and analysed by gas chromatography–mass spectrometry (GC-MS). Typically, individual fatty acids, resin acids, sterols, fatty alcohols, hydroxy acids etc. are quantified.

### Carbohydrates, lignin and polyphenols

**Carbohydrates**

Carbohydrate composition is determined according to standard SCAN-CM 71 acid hydrolysis and ion chromatography. The content of glucose, mannose, arabinose, xylose, and galactose is reported. The analysis may be performed before and after hydrolysis in order to distinguish between monomeric and polymeric carbohydrates. Total carbohydrates may also be determined using the Orcinol method.

**Lignin**

Klason lignin and acid soluble lignin is determined using acid hydrolysis and UV-spectroscopy respectively.

**Aliphatic, phenolic and carboxylic hydroxyl**

Aliphatic, phenolic and carboxylic hydroxyl groups may be determined in an isolated lignin sample, by nuclear magnetic resonance spectroscopy (NMR).

**Hydroxyl groups (phenols) content**

The total phenolic content may be determined using the Folin-Ciocalteu method with spectrophotometric detection.

### Other organic compounds

**Low molecular acids**

Formic acid, acetic acid, lactic acid, glycolic acid and oxalic acid (more acids may be added) are analysed using ion chromatography. Oxalic acid and Ca-oxalate may be determined separately.

**Other compounds**

- Ethanol and methanol
- Furfural and hydroxymethyl furfural
- Chlorophenols
- Unchlorinated phenols
Chemical “fingerprint”

Analytical pyrolysis
A chemical “fingerprint” of the organic compounds in liquors and effluents may be acquired using pyrolysis–gas chromatography–mass spectrometry. The technique is very well suited for the analysis of polymeric and monomeric substances in demanding matrixes. The technique is fast and has the advantage of giving a complete overview of the organic constituents with just one single run.

Ash and elements composition

Ash content
Residue (i.e. ash) on ignition at 525°C according to standard ISO 1762.

Metals/elements content
A variety of different metals and elements may be measured using atomic emission spectroscopy (ICP-AES), for instance Al, Ba, Ca, Cu, Fe, K, Mg, Mn, Na, S and P. Metals may be analysed before and after filtration in order to determine total content or dissolved content (< 40 µm).

Other inorganic compounds

Carbonate
Carbonate is determined as total inorganic carbon (TIC) by acidification and infrared spectroscopy.

Total sulphur or total chlorine
Total sulphur or total chlorine is determined according to SCAN-CM 57 and SCAN-CM 51 respectively using Schöniger combustion. Formed sulphate is determined according to ISO 9198 and formed chloride according to ISO 9197 with ion chromatography.

Sulphide and hydrogen sulphide
Sulphide is determined by titration according to SCAN-N 31.

Inorganic anions
Several inorganic anions may be determined using ion chromatography:
- Bromide, chloride, fluoride and iodine
- Chlorate
- Nitrate and nitrite
- Sulphite, sulphate and thiosulphate
- Oxalic acid (oxalic acid and Ca-oxalate may be determined separately)

INNVENTIA AB is a world leader in research and development relating to pulp, paper, graphic media, packaging and biorefining. Our unique ability to translate research into innovative products and processes generates enhanced value for our industry partners. We call our approach boosting business with science. Innventia is based in Stockholm, Bäckhammar and in Norway and the U.K. through our subsidiaries PFI and Edge respectively.