



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.

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.

We possess a range of analytical methods for analysis of:

- Sum parameters (Total and suspended solids, TOC, COD, pH etc.)
- Metals and inorganic anions
- Wood extractives
- Organic components
- Lignin content
- Carbohydrates
- Low molecular weight compounds

Liquors and effluents from the wood biorefinery:

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

A list of analytical services related to aqueous samples is appended.

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SUM PARAMETERS

We offer analysis of the following sum parameters:

- Total solids
- Suspended solids
- TOC, COD, Tot Nitrogen
- pH
- Alkali (total, active, and effective)
- Residual alkali
- Acidic groups content

INORGANIC COMPONENTS

Ash content

Residue on ignition at 525°C according to ISO 1762

Metals/elements content

Several elements may be measured using atomic emission spectroscopy (ICP-AES), for instance Al, Ba, Ca, Cu, Fe, K, Mg, Mn, Na, S and P.

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 ions are determined by ion chromatography

- Bromide, chloride, fluoride and iodine
- Chlorate
- Nitrate and nitrite
- Sulphite, sulphate and thiosulphate
- Oxalic acid

WOOD EXTRACTIVES

Extractives content - MTBE extraction

Used for effluent samples, especially TMP effluents (includes lignans)

Extractives content - Petroleum-ether extraction ("Saltsman-Kuiken")

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 DCM are used for specific analytes

Extractives composition - Group separation with GC/FID

Extracts are silylated and analysed by 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).

Extractives composition - Individual compounds with GC/MS

Extracts are derivatized and analysed by GC/MS.

Typically individual fatty acids, resin acids, sterols, fatty alcohols, hydroxy acids etc. are quantified.

ADDITIONAL ORGANIC COMPONENTS

Carbohydrates

Carbohydrate composition is determined according to standard SCAN-CM 71 acid hydrolysis and ion chromatography.

Lignin

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

Aliphatic, phenolic and carboxylic hydroxyl groups

May be determined in an isolated lignin sample, by NMR (nuclear magnetic resonance spectroscopy).

Hydroxyl groups (phenols) content

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

Low molecular acids

Formic acid, acetic acid, lactic acid, glycolic acid, oxalic acid are analysed using ion chromatography.

Sugar acids, e.g. α - and β -isosaccharinic acid, xylo-isosaccharinic acid and dihydroxypentanoic acid, are quantitatively determined using pyrolysis-GC/MS.

Other compounds

- Ethanol and methanol
- Furfural and hydroxymethyl furfural
- Chlorophenols
- Unchlorinated phenols

CHEMICAL "FINGERPRINT"

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.