SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

SCAN-N 22:96
Revised 1996

Black liquor

Dry matter content and fibre content

0 Introduction

This SCAN-test Standard replaces SCAN-N 22:77 from which it differs in that it also provides a procedure for the determination of fibre content.

The procedure for the determination of dry matter content has at the same time been changed to a new, faster method, because the procedure described in SCAN-N 22:77 was too slow. The results obtained with the new, faster procedure do not differ from the results obtained with the earlier method, see Annex.

1 Scope

This Standard describes the procedures for the determination of dry matter content and fibre content (suspended material) in black liquor.

The Standard is applicable to all kinds of black liquor produced in a pulp mill using the kraft process. It is primarily intended for use in rapid calibration or routine control procedures.

2 References

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3 Definitions

For the purpose of this Standard, the following definitions apply:

3.1 *Dry matter content* – The ratio of the mass after drying to the mass before drying, the drying of the black liquor being performed as specified in this Standard.

Note – The difference in mass is due to evaporation of water and, to a less extent, of other volatile components. Since the loss of mass is dependent on temperature and time, the procedure cannot be taken as an accurate measure of the dry matter content of the sample.

To a small degree, the loss of mass is compensated for by the oxidation of sulphide to thiosulphate:

$$2\ Na_2S + 2½\ O_2 \rightarrow Na_2S_2O_3 + 2\ NaOH$$

The result can be corrected by calculation provided the content of sulphide in the liquor is known.

The dry matter content is normally expressed as a percentage.

3.2 *Fibre content* – The absolute, residual mass after filtering and washing through a well-defined wire cloth, the filtering and washing of the black liquor being performed as specified in this Standard.

Note – The washing procedure may dissolve some of the substances that are difficult to dissolve due to the change in pH and in concentration.

4 Principle

4.1 *Dry matter content* – A black liquor sample is dried on a glass-fibre filter, placed on an aluminium dish, in an oven at a temperature of 105 °C for at least 30 min. After drying, the sample is immediately wrapped in the aluminium dish, and weighed. The dry matter content is calculated.

4.2 *Fibre content (suspended material)* – A known volume of the sample is filtered through a well-defined wire cloth. The residue on the wire cloth is washed with hot water, to remove dissolved solids. The residue is dried in an oven at a temperature of 105 °C for 2 h. The fibre content is calculated.

5 Apparatus

Ordinary laboratory equipment and the items listed below.

5.1 Dry matter content and fibre content:

5.1.1 *Oven*, adjustable to (105 3) C.

5.1.2 *Balance*, with a resolution of 0,1 mg.

5.1.3 *Magnetic stirrer* with magnet.

5.1.4 *Water bath*, thermostated at 90 C.

5.2 Dry matter content:

5.2.1 *Glass-fibre filter*, GF/A, diameter 7 cm, or equivalent.

5.2.2 *Aluminium dishes,* disposable. Alternatively, aluminium foil, 10 cm × 10 cm pieces.

> *Note* – The aluminium used should be free from fat and oil.

5.2.3 *Plastic bottle*, volume 100 ml, with tight, leak-proof closure.

5.2.4 *Balance with IR-drier,* as an alternative to the oven (5.1.1) (see 7.2, Note 2) with a resolution of 1 mg.

5.3 Fibre content:

5.3.1 *Wire cloth*, preferably of nylon, pore size 70 µm, diameter 5,5 cm.

5.3.2 *Vacuum filtration assembly* consisting of a *Büchner funnel* capable of holding the wire cloth (5.3.1) and a *vacuum flask*, capacity 3000 ml, connected to vacuum.

5.3.3 *Balance*, with a resolution of 0,01 g.

6 Sampling and sample preparation

The sampling procedure is not covered by this Standard.

The sample for the determination of dry matter content must be diluted to a dry matter content below 40 % prior to the analysis, as described in 7.1.

All samples for the determination of fibre content have to be diluted. If the dry matter content exceeds 50 %, follow the instructions given in 8.1 and, if the dry matter content is lower than 50 %, follow the instructions given in 8.2.

7 Determination of dry matter content (incl. fibres)

7.1 Dilution

Heat the sample of black liquor in a water bath (5.1.4) at a temperature of 90 C for 20 min. Keep the closure on the sample bottle during the heating period.

Mix the sample carefully with a glass rod, taking care to ensure that any sediment on the bottom of the sample bottle is stirred and homogenized with the rest of the sample.

Place an empty, dry plastic bottle (5.2.3) on the balance (5.1.2). Tare the balance.

> *Note* – It may be convenient to weigh the black liquor, if a lid is used. Tare the balance with the plastic bottle and the lid.

Weigh into the plastic bottle 10 g of the homogenized sample to the nearest 0,01 g (W_C), and then add 50 g of hot, distilled water and weigh to the nearest 0,01 g (W_D).

Put a magnet into the bottle and close the bottle. Tighten the stopper well. Place the bottle on the magnetic stirrer (5.1.3) and stir until the sample is totally homogenized, for approximately 10 min. The sample is now ready to be analysed.

7.2 Analysis, using an oven (or an IR-drier)

Run the whole procedure in duplicate.

Place a glass-fibre filter (5.2.1) in an aluminium dish (5.2.2). Place the filter and the dish on the balance (5.1.2) and weigh to the nearest 0,1 mg (W_T). Tare the balance.

Shake the sample thoroughly until the sample is totally homogenized.

With the aid of a Pasteur pipette, drip between 1 g and 2 g of the sample onto the glass-fibre filter and weigh it quickly to the nearest 0,1 mg (W_A).

Place the aluminium dish with the glass-fibre filter and sample in the oven (5.1.1) and dry at a temperature of (105 3) C for 30 min.

After 30 min, quickly fold the dish walls over the glass-fibre filter and the sample, and weigh immediately to the nearest 0,1 mg (W_B). Check that the sample is dried to constant mass (weight).

Note 1 – It is not necessary to dry the aluminium dish or the glass-fibre filter before use. None of the materials are hygroscopic.

Note 2 – The use of a balance with an IR-drier gives the same result as the drying with an oven, but with poorer precision data, see Clause 11.2. The low number of participant laboratories in this investigation may have influenced the result.

If an IR-drier is used, follow the recommendations given by the IR-drier supplier.

8 Determination of fibre content

8.1 Dilution, dry matter content > 50 %

Heat the sample in a water bath (5.1.4) at 90 C for 20 min.

Mix the sample carefully with a glass rod taking care to ensure that any sediment on the bottom of the sample bottle is stirred and homogenized with the rest of the sample.

Place an empty glass beaker (volume 3 litre) on the balance (5.3.3) and tare. Add 100 g of the sample and weigh to the nearest 0,1 g (M), and then add 1900 g of distilled water and weigh to the nearest 0,1 g.

Put a magnet in to the glass beaker, place the beaker on the magnetic stirrer (5.1.3) and stir until the sample is totally dissolved and homogenized, for approximately 10 min.

The sample is now ready to be analysed as described in 8.3.

8.2 Dilution, dry matter content < 50 %

Place an empty glass beaker (volume 3 litre) on the balance (5.3.3) and tare.

Shake the sample thoroughly until it is totally homogenized. Add 500 g of the sample to the beaker and weigh to the nearest 0,1 g (M), and then add 1500 g of distilled water and weigh to the nearest 0,1 g.

Put a magnet into the beaker, place the beaker on the magnetic stirrer (5.1.3) and stir until the sample is totally dissolved and homogenized, for approximately 10 min.

The sample is now ready to be analysed as described in 8.3.

8.3 Filtration

Place an Erlenmeyer flask containing 2 litre of distilled water on the hot plate.

Weigh on the balance (5.1.2) the wire cloth (5.3.1) to the nearest 0,1 mg (W_w).

Place the wire cloth in the Büchner funnel (5.3.2) and attach the filtering equipment to the vacuum flask. Filter the whole sample through the wire cloth.

Wash the inside of the beaker with hot, distilled water so that no visible residue of suspended material is left. Filter the rinsing through the wire cloth.

Wash the residue on the wire cloth with 4 portions of 500 ml of distilled water (80 C), taken from the Erlenmeyer flask using a measuring cylinder.

If the rinsing is not clear, continue the washing procedure until the rinsing is clear.

Dry the wire cloth with the sample residue in the oven (5.1.1) at 105 C for 2 h. Cool the wire cloth and the dry residue in a desiccator for 30 min.

Weigh the wire cloth with the sample residue to the nearest 0,1 mg (W_s).

Note – Investigations have shown that it is not necessary to dry wire cloth made of nylon before use, since the material is non-hygroscopic. If however any other material is used, the hygroscopic behaviour must be investigated before use.

If the residue on the wire cloth contains material other than fibres, this should be reported.

9 Calculation

9.1 Dry matter content

Calculate the dry matter content using the expression:

$$X = \frac{100\left(W_B - W_T\right)\cdot W_D}{W_A \cdot W_C} \qquad [1]$$

where

X is the dry matter content, in per cent;
W_B is the mass of the dried aluminium dish, glass-fibre filter and sample, in grams;
W_T is the mass of aluminium dish and glass-fibre filter, in grams;
W_D is the mass of black liquor and distilled water after dilution, in grams;
W_A is the mass of the sample, in grams;
W_C is the mass of black liquor before dilution, in grams,.

Calculate the mean of the parallel determinations and report the result to one decimal place. The results of the parallel determinations should not deviate by more than 1 % in dry matter content from their mean.

9.2 Fibre content

9.2.1 Calculate the fibre content (suspended material) using the expression:

$$Y = 10^6 \frac{\left(W_s - W_w\right)}{M} \qquad [2]$$

where
Y is the fibre content in the original black liquor, in milligrams per kilogram;
W_s is the mass of the wire cloth including the sample residue, in grams;
W_w is the mass of the wire cloth, in grams;
M is the original mass of black liquor, before dilution and filtration, in grams.

9.2.2 If the fibre content is to be reported per kilogram of dry substance, calculate the fibre content (suspended material) according to the expression:

$$Y_1 = \frac{100\ Y}{X} \qquad [3]$$

where
Y_1 is the fibre content in the original black liquor, in milligrams per kilogram dry substance;
Y is the fibre content in the original black liquor, in milligrams per kilogram;
X is the dry matter content of the original black liquor, in per cent.

Calculate the mean of the parallel determinations and report the result to one decimal place. The result of the parallel determinations should not deviate by more than 5 % in fibre content from their mean.

10 Report

The test report shall include reference to this SCAN-test Standard and the following particulars:

(a) date and place of testing;
(b) identification mark of the sample tested;
(c) information as to whether the sample was diluted before analysis;
(d) the results;
(e) whether an oven or a balance with IR-drier has been used (dry matter content);
(f) any departure from the standard procedure and any other circumstances that may have affected the results.

11 Precision

11.1 Repeatability

One laboratory analysed the dry matter content of one black liquor sample ten times by using an oven. The results were as follows:

Sample	Mean dry matter content, %	Coeff. of variation, %
Black liquor	17,2	0,87

One laboratory analysed the fibre content of one black liquor sample ten times. The results were as follows:

Sample	Mean fibre content, mg/kg	Coeff. of variation, %
Black liquor	52,3	1,13

11.2 Reproducibility

Eight laboratories analysed the dry matter content in two samples, one thick black liquor (> 50 % dry matter content) and one thin black liquor (< 50 % dry matter content), by using an oven. The results were as follows:

Sample	Mean dry matter content, %	Coeff. of variation, %
Thick black liquor	70,7	0,42
Thin black liquor	15,5	0,43

Three laboratories analysed the dry matter content of two samples, one thick black liquor (> 50 % dry matter content) and one thin black liquor (< 50 % dry matter content) by using a balance with IR-drier. The results were as follows:

Sample	Mean dry matter content, %	Coeff. of variation, %
Thick black liquor	68,4	2,15
Thin black liquor	15,1	1,53

Five laboratories analysed the fibre content of one black liquor sample. The results were as follows:

Sample	Mean fibre content, mg/kg	Coeff. of variation, %
Black liquor	32,7	14,4

12 Literature

12.1 Söderhjelm L, Sågfors P-E, Väänänen V. Black liquor dry solids content. Paperi ja Puu *76* (1994):5, 330-332.

Annex –
Comparison of the earlier method and the new, fast and modified method

The dry matter contents in four black liquors were determined according to both the earlier method (SCAN-N 22:77) and the new, fast method (SCAN-N 22:96). The dry matter contents for the two methods were then plotted against each other, as shown in *Figure 1*. The linear coefficient of correlation for the data is 0,993. The line drawn in the figure is the 1:1 line showing the agreement between the two methods.

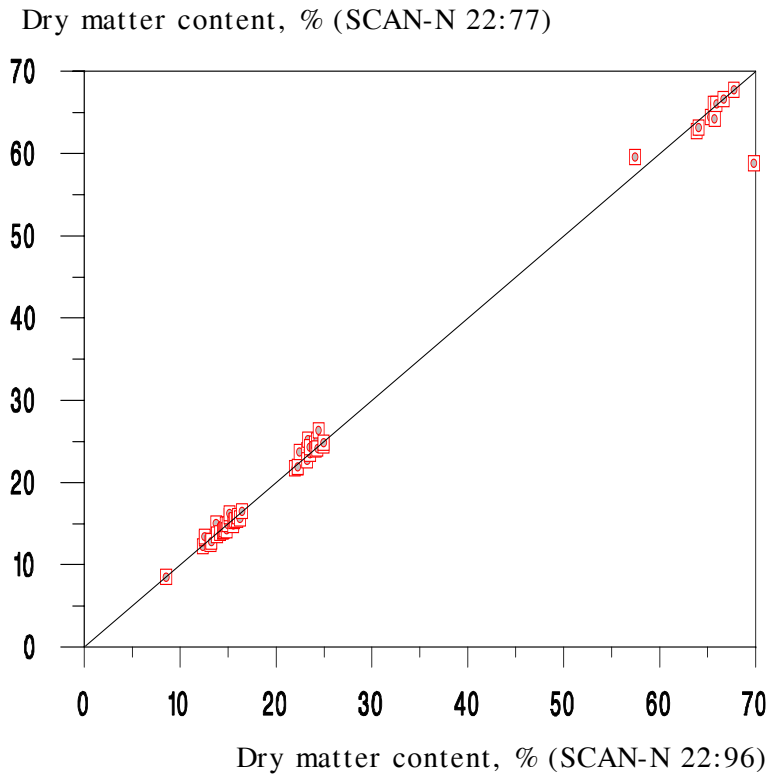

Figure 1. The two different procedures for the determination of the dry matter content give the same result.

SCAN-test Standards are issued and recommended by the central laboratories of the pulp, paper and board industries in Denmark, Finland, Norway and Sweden.
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