SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

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Lime sludge

Water-soluble sodium

1 Scope and field of application

This SCAN-test Method specifies a method for the determination of the water-soluble fraction of the sodium content in lime sludge. The Method is primarily intended for use in pulp mills using the sulphate process.

2 Definition

2.1 *Water-soluble sodium* (in lime sludge): Sodium that is dissolved and washed out from lime sludge when this is treated with water by a specified standardized procedure.

3 Principle

A sample of lime sludge, analysed as taken, is mixed with water at room temperature for 1 h. Undissolved material is removed by filtration. The sodium is determined by atomic absorption spectroscopy or by flame photometry, using a caesium or caesium-aluminium solution respectively to minimize interferences.

4 Apparatus

4.1 *Atomic absorption spectrophotometer*, equipped for the determination of sodium at 589,0 nm, or

4.2 *Flame photometer*, equipped for the determination of sodium.

5 Reagents

All reagents shall be of analytical grade (pro analysi). Distilled or deionized water, free from sodium and having a conductivity of less than 300 µS/m, shall be used in the preparation of solutions and for diluting samples.

5.1 *Standard sodium solution*, 100 mg/l. Ignite a portion of anhydrous sodium sulphate, Na_2SO_4 at 550 °C in a crucible of platinum or porcelain. Allow to cool to room temperature in a desiccator. Weigh 308,9 mg, using an analytical balance, and dissolve in distilled water. Dilute to 1 litre in a volumetric flask. Store in a polyethene bottle. This solution contains 0,1 mg of sodium per millilitre.

Commercially available calibration solutions may be used.

5.2 *Caesium solution*, 50 g/l. (This solution is used only when sodium is determined by atomic absorption spectroscopy.) Dissolve 63,5 g of caesium chloride, CsCl, in distilled water and dilute to 1 litre.

5.3 *Caesium-aluminium solution*, 50 mg CsCl and 250 g $Al(NO_3)_3$ per litre. (This solution is used only when sodium is determined by flame photometry.) Dissolve 50 mg of caesium chloride, CsCl, and

250 g of aluminium nitrate, $Al(NO_3)_3$, in distilled water and dilute to 1 litre.

6 Preparation of sample

Use samples as taken in the mill. Determine the dry matter content of the sludge on a separate sample as described in SCAN-N 23.

7 Procedure

7.1 *Preparation of sample solution*. Weigh rapidly to the nearest 0,1 g a sample of about 15 g of the lime sludge in a 1 litre conical flask. From a volumetric flask add 500 ml of distilled water. Close the conical flask and stir vigourously with a magnetic stirrer for 60 min.

Filter the mixture through a glass fiber filter (Whatman GF/A) and collect the filtrate in another flask. Do not wash the sludge and do not use any rinsing water.

7.2 *Determination of sodium by atomic absorption spectroscopy*. Dilute 10 ml of the standard sodium calibration solution (5.1) to 100 ml in a volumetric flask. This solution contains 0,01 mg of sodium per millilitre.

Prepare a series of 5 calibration solutions by diluting v ml of the diluted standard sodium solution to 100 ml with distilled water in volumetric flasks. Before filling up to the mark, add 2 ml of the caesium solution (5.2). Select the volumes v so that the working range of the atomic absorption spectrophotometer is covered; this range is normally 0,1 to 1,0 mg/l. Prepare also a blank solution (v = 0). Prepare fresh solutions each day determinations are made.

Dilute a ml of the sample solution (7.1) to 100 ml in a volumetric flask. Before filling up to the mark, add 2 ml of the caesium solution (5.2). Select a so that the sodium content of the final solution is within the range covered by the calibration solutions.

Following the instructions for the spectrophotometer, measure the absorbance at 589,0 nm in an air-acetylene flame. Measure the absorbance of the calibration solutions before and after that of the sample solution. Determine the sodium content of the sample solution from a calibration graph obtained by plotting the absorbance against the sodium content for the series of calibration solutions.

7.3 *Determination of sodium by flame photometry*. Prepare a series of 5 calibration solutions by diluting v ml of the standard sodium solution (5.1) to 100 ml with distilled water in volumetric flasks. Before filling up to the mark, add 10 ml of the caesium-aluminium solution (5.3). Select the volumes v so that the working range of the flame photometer is covered; this range is normally 1 to 10 mg/l. Prepare also a blank solution (v = 0).

Dilute a ml of the sample solution (7.1) to 100 ml in a volumetric flask. Before filling up to the mark, add 10 ml of the caesium-aluminium solution (5.3). Select a so that the sodium content of the final solution is within the range covered by the calibration solutions.

Following the instructions for the flame photometer, measure the emission at 589,0 nm. Measure the emission from the calibration solutions before and after that of the sample solution Determine the sodium content of the sample solution from a calibration graph obtained by plotting the emission against the sodium content for the series of calibration solutions.

Note – The calibration graph is theoretically a curve passing through a maximum. Only the almost linear part near the origin should be used when determining sodium.

8 Calculation

Calculate the water-soluble sodium content of the sample (oven dry basis) from the expression

$$X = \frac{5000 \cdot c}{a \cdot w \cdot d}$$

where

X is the content of water-soluble sodium in grams per kilogram,
c is the concentration of sodium obtained from the calibration graph, in milligrams per litre,
a is the volume of sample solution taken, in millilitres,
w is the mass of sample taken, in grams,
d is the dry matter content of the sample, in per cent.

The numerical factor 5000 contains the factors 0,1 (volume of the final solution), 0,5 (volume of the added water) and 100*1000 to bring the result into grams per kilogram.

9 Precision

Five samples of lime sludge, having contents of water-soluble sodium from 1,2 to 5,2 g/kg, were analysed by 10 laboratories. The relative standard deviation was in the range 4,2 to 7,8 per cent.

10 Report

Report the water-soluble sodium content in grams per kilogram with two significant figures. The report shall include reference to this SCAN-test Method and the following particulars:

a) date and place of testing,
b) identification mark of the sample tested,
c) the results,
d) any departure from this Method or other circumstances that may have affected the test results.

SCAN-test Methods are issued and recommended by KCL, PFI and STFI-Packforsk for the pulp, paper and board industries in Finland, Norway and Sweden.
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