SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

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Lime and lime sludge

Acid-soluble sodium

1 Scope and field of application

This SCAN-test Method specifies a method for the determination of acid-soluble sodium in lime and in lime sludge, including burnt lime sludge. The Method is primarily intended for use in pulp mills using the sulphate process.

2 Definition

2.1 *Sodium* (in lime and lime sludge): Sodium that dissolves when lime or lime sludge is treated with hydrochloric acid.

3 Principle

The sample is dissolved in hydrochloric acid. The sodium content in the solution 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 *Drying oven*, controlled at a temperature of 105 ± 3 °C.

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

4.3 *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 *Hydrochloric acid*, 6 mol/l. Dilute concentrated hydrochloric acid, HCl, density 1190 kg/m3 with an equal volume of distilled water.

5.2 *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.

5.3 *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.

5.4 *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

Dry wet samples of lime sludge in a drying oven at 105 °C. Grind all samples in a mortar until a fine powder has been obtained.

7 Procedure

7.1 *Preparation of sample solution*. In a 250 ml beaker weigh, to the nearest 0,001 g, a sample of about 2 g. Add 25 ml of distilled water, mix and add with caution 10 to 15 ml of the hydrochloric acid (5.1). Continue the mixing until no more sample dissolves.

Filter through a paper filter and wash the filter with a few small portions of distilled water. Collect the filtrate and the washings in a 1 litre volumetric flask and dilute to the mark with distilled water.

7.2 *Determination of sodium by atomic absorption spectroscopy*. Dilute 10 ml of the standard sodium calibration solution (5.2) 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 (5.3). 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 (5.3). 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 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 (5.2) to 100 ml with distilled water in volumetric flasks. Before filling up to the mark, add 10 ml of the caesium-aluminium solution (5.4).

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 (5.4). 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 for the determination of sodium.

8 Calculation

Calculate the sodium content of the dry sample from the expression

$$X = \frac{100 \cdot c}{(a \cdot w)}$$

where

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

9 Precision

Two samples of lime and two samples of lime sludge were analysed by 10 laboratories. The content of acid-soluble sodium was about 12 g/kg for the lime samples and about 8 g/kg for the lime sludge samples. The results had relative standard deviations in the range 2,3 to 3,0 %.

10 Report

Report the 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.
Distribution: Secretariat, Scandinavian Pulp, Paper and Board Testing Committee, Box 5604,
SE-114 86 Stockholm, Sweden.