SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

SCAN-N 4:78
Revised
1978

White and green liquors

Chloride content

0 Introduction

This SCAN-test Method is a revised version of SCAN-N 4:63 from which it differs in the, method of titration. Instead of the Mohr titration prescribed in the earlier version, the Method now includes a potentiometric titration. The results are expressed in millimoles per litre.

1 Scope and field of application

This SCAN-test Method specifies a method for the determination of the chloride content of white and green cooking liquors used in the sulphate pulping process.

2 Principle

The liquor is oxidized with hydrogen peroxide and acidified with nitric acid. After addition of acetone the chloride is determined by potentiometric titration with silver nitrate solution.

3 Apparatus

3.1 *Potentiometer or pH-meter*, readable to 1 mV or equivalent. Automatic titration devices having a strip-chart recorder may be used.

3.2 *A silver electrode and a mercury/mercury (I) sulphate electrode*, for use with 3.1.

3.3 *Burette*, maximum 25 ml, with 0,1 ml divisions.

4 Reagents

4.1 *Hydrogen peroxide solution*, 300 g of H_2O_2 per kilogram.

4.2 *Silver nitrate solution*, 10 mmol of $AgNO_3$ per litre; the concentration known to an accuracy of 0,05 mmol/l.

4.3 *Nitric acid*, approximately 8 mol/l. Dilute concentrated nitric acid, HNO_3, density ca 1300 kg/m^3, with an equal volume of distilled water.

4.4 *Acetone*, CH_3COCH_3.

All reagents should be of analytical grade (*pro analysi*) .

5 Procedure

Transfer 10,0 ml of the liquor to a 100 ml beaker, using a pipette and add about 10 ml of distilled water. Add several 1 ml portions of the hydrogen peroxide while heating until the solution becomes colourless. After the final addition of hydrogen peroxide, boil the solution for 5 min. Allow the sample to cool to room temperature and transfer it quantitatively to a 100 ml volumetric flask. Dilute to the mark with distilled water.

Using a pipette, transfer a 20,0 ml aliquot of the oxidized solution to a 150 ml beaker. Add 70 ml of acetone and acidify with 10 to 12 drops

of the 8 mol/1 nitric acid. Check that the solution is acid with a strip of indicator paper.

Connect the electrodes to the potentiometer and immerse them in the sample solution. Use a magnetic stirrer for mixing.

Titrate by adding small portions of the silver nitrate solution. Record the potentiometer reading after each addition. Construct the titration curve as the titration proceeds by plotting each reading against the corresponding total volume of silver nitrate solution added. The volumes of portions should be selected so that a smooth S-shaped curve is obtained. Smaller volumes should be taken in the vicinity of the equivalent point. Use the inflection point of the curve as the end-point of the titration.

> *Note* – When new equipment is being used or when samples of a new kind are being analysed, it is advisable to carry out a pre–liminary titration in order to establish the general shape of the curve and the size of the additions.

The titration may be performed with the aid of an automatic titrator if so desired.

6 Calculation

Calculate the chloride concentration from the expression

$$X = 10 \cdot (a - b) \cdot m / v$$

where

- a is the volume of silver nitrate solution consumed at the end-point in the titration of the sample, in milliliters,
- b is the volume of silver nitrate solution consumed at the end-point in the blank titration, in milliliters,
- m is the concentration of the silver nitrate solution, in millimoles per litre,
- v is the volume of oxidized solution taken for titration, in milliliters (normally 20 ml),
- X is the the chloride concentration of the sample, in millimoles per litre.

7 Report

Report the result, in millimoles per litre, to the nearest integer. If several parallel determinations have been carried out, report each result separately.

The test report should include a reference to this Method and the following particulars:

a) date and place for testing
b) identification of the liquor tested
c) the results
d) any departure from the Method or any other circumstances that may have affected the 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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