SCANDINAVIAN PULP, PAPER AND BOARD
TESTING COMMITTEE

SCAN-P 54:84
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Starch

Chloride content

1 Scope

This SCAN-test Method specifies a procedure for the determination of the chloride content in samples of starch used in the manufacture of papers and boards. It is applicable both to native starches and starch derivatives and is primarily intended for quality control purposes. The chloride content is important for corrosion in the paper mill and for the quality of the manufactured paper.

The lower limit of chloride content that can be determined is 0,02 %.

2 Reference

SCAN-P 52 Starch – Dry matter content

3 Principle

The starch sample is dispersed in water and dissolved chloride is determined by potentiometric titration with silver nitrate solution.

4 Apparatus

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

4.2 *Electrodes*, a silver electrode and a mercury/ mercury (I) sulphate reference electrode, for use with 4.1.

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

4.4 *Magnetic stirrer*.

5 Reagents

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

5.1 *Silver nitrate solution*, 50 mmol of $AgNO_3$ per litre; the concentration being known to an accuracy of 0,1 mmo1/l.

5.2 *Acetone*, CH_3COCH_3.

5.3 *Nitric acid*, approx. 3 mol/l. Add with caution 200 ml of concentrated nitric acid (HNO_3, density approx. 1400 kg/m^3) to about 500 ml of distilled water. Dilute to 1 litre.

5.4 *Ethanol*, C_2H_5OH, 95 %.

6 Procedure

Determine the dry matter content on a separate sample as described in SCAN-P 52.

Carry out the following procedure in duplicate. Weigh, to an accuracy of 10 mg, 2 g to 3 g of the starch in a 250 ml beaker. Add 40 ml of distilled water and stir with the magnetic stirrer for 10 min.

> *Note 1* – Some starches soluble in cold water may form lumps when dispersed in water by the above procedure. To prevent this, disperse the dry sample in 20 ml of ethanol (5.4) before adding the water.

Add 70 ml of acetone (5.2) and acidify the suspension by adding a few drops of the nitric acid (5.3).

> *Note 2* – The presence of acetone improves the titration curve. However, it causes water-soluble starches to form lumps and must therefore be replaced by an equal volume of water when such starches are analysed.

Connect the electrodes (4.2) to the potentiometer (4.1) and immerse them in the suspension. Use the magnetic stirrer (4.4) for mixing.

Titrate by adding small portions of the silver nitrate solution (5.1) 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. Small volumes should be added in the vicinity of the equivalence point. Take the inflexion point of the curve as the end-point of the titration. Record the volume of silver nitrate used to reach the end-point.

> *Note 3* – When analysing unknown types of starch it is advisable to make a preliminary test in order to find out whether ethanol should be used and whether the acetone should be omitted.

> *Note 4* – If the sample has a very low content of chloride, add to the suspension a known amount of chloride, for example 5 ml of a solution containing 1 g of sodium chloride per litre. Make a blank titration with the same addition of chloride.

7 Calculation and report

Calculate the chloride content from the expression

$$X = \frac{0{,}355\ a\ b}{k\ w} \qquad [1]$$

where

X = the chloride content of the sample, as a percentage;
a = the volume of the silver nitrate solution used, in millilitres;
b = the concentration of the silver nitrate solution, millimoles per litre;
k = the dry matter content of the sample, as a percenttage;
w = the weight of the sample as taken, in grams.

Express the result to the second decimal as the mean of the two determinations.

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

(a) date and place for testing;
(b) identification mark of the sample;
(c) the results;
(d) any departure from the procedure described in this Method or any other circumstances that may have affected the results.

8 Precision

Seven laboratories analysed samples of the same starch. For oxidized starch a mean chloride content of 0,59 % was obtained, the maximum and minimum results were 0,63 % and 0,53 % respectively. For a cold-water soluble starch a mean of 0,02 % was found, the maximum and minimum results were 0,02 % and 0,01 %, respectively.

9 Additional information

The principle used in this Method is the same as that used in ISO 5810 part 2, *Starch and derived products – Determination of chlorid content – Potentiometric method*. In the ISO Standard, however, a silver/silver chloride electrode is used as the measuring electrode and neither acetone nor ethanol are added.

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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