International Standard 3260

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Pulps — Determination of chlorine consumption (Degree of delignification)

Pâtes — Détermination de la consommation en chlore (Degré de délignification)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3260 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 3260-1975), which had been approved by the member bodies of the following countries :

Austria	Germany, F. R.	Poland
Belgium	Hungary	Romania
Bulgaria	India	South Africa, Rep. of
Canada	Iran	Spain
Czechoslovakia	Israel	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
Finland	New Zealand	Turkey
France	Norway	USSR

No member body expressed disapproval of the document.

Pulps — Determination of chlorine consumption (Degree of delignification)

0 Introduction

The method specified in this International Standard for determining the degree of delignification of pulp by measuring its chlorine consumption under specified conditions is related to that for determining the degree of delignification of pulp by measuring its potassium permanganate consumption under specified conditions, given in ISO 302, *Pulps — Determination of Kappa number*. Unlike that method, the method for the determination of chlorine consumption has the merit of not being restricted to pulps obtained in yields under 60 %.

It has been shown experimentally that there is a linear relationship between the chlorine consumption and the total lignin content of pulp. This relationship is independent of the method used in the manufacture of the pulp.

1 Scope and field of application

This International Standard specifies a method for the determination of the degree of delignification of pulp by measuring its chlorine consumption.

This method is applicable to all kinds of pulp.

2 Reference

ISO 638, *Pulps — Determination of dry matter content.*

3 Definition

For the purposes of this International Standard, the following definition applies :

chlorine consumption of a pulp : The amount of active chlorine consumed by a pulp under the conditions specified in this International Standard.

It is expressed as a percentage by mass.

4 Principle

Treatment of a test portion of pulp for 15 min, at a temperature of 25 ±1 °C, with chlorine generated by acidification of a sodium hypochlorite solution.

Determination of the residual chlorine, which must be more than 50 % of the amount added (see the note in clause 9), by iodometric titration. Correction of the chlorine consumption so obtained to the consumption at constant concentration of available chlorine.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sodium hypochlorite (NaClO), solution containing about 20 g of active chlorine per litre and of total alkalinity corresponding to a pH of 12,0 ±0,5, measured with a glass electrode.

5.2 Hydrochloric acid, 4 mol/l solution, obtained by adding 100 ml of hydrochloric acid (HCl), ϱ = 1,19 g/ml, to 200 ml of water.

5.3 Potassium iodide, 1 mol/l solution, containing 166 g of potassium iodide (KI) per litre.

5.4 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3)$ = 0,2 mol/l.

The concentration shall be known to ± 0,000 4 mol/l.

5.5 Starch, 2 g/l indicator solution.

6 Apparatus

Ordinary laboratory apparatus, and

6.1 High-speed wet disintegration apparatus, for example a kitchen mixer or similar apparatus which disintegrates the pulp completely with a minimum of damage to the fibres.

6.2 Apparatus for the determination of chlorine consumption, as shown in the figure, consisting of

6.2.1 Thick-walled conical flask (D), of capacity 750 ml, with a standard ground joint (C).

6.2.2 Separating funnel (E), of capacity 50 to 100 ml, with standard ground joints (B and C) and a glass stopper (A).

6.3 Motor-driven coated magnetic stirrer, providing efficient stirring when the magnet and the motor table are approximately 40 mm apart.

6.4 Water bath, capable of maintaining a temperature of 25 ± 1 °C for at least 20 min and provided with a support for the flask.

6.5 Vacuum-pump.

6.6 Stop-watch.

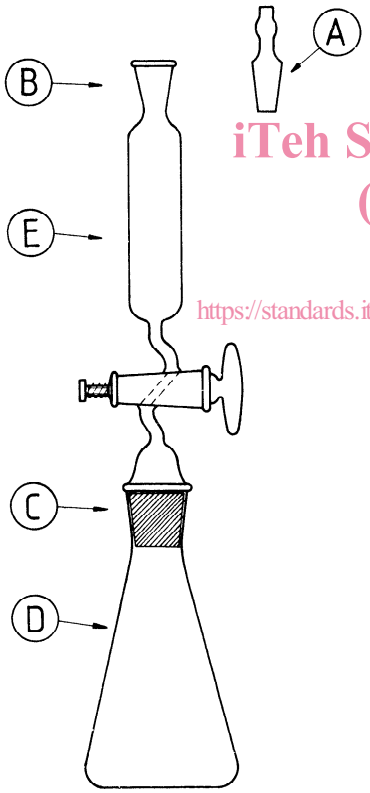

Figure — Apparatus for determination of the chlorine consumption of pulp

7 Preparation of sample

7.1 Air-dried pulp sheets

Tear 3 to 10 g of the pulp into small pieces.

7.2 Screened slush pulps

Make a 3 to 10 g air-dry pad by filtering on a Büchner funnel, avoiding any loss of fibres. Air-dry the pad and tear it into small pieces.

7.3 Unscreened pulps

If the pulp sample is taken from unscreened pulp which is normally screened before bleaching or other processing, then the shives and knots shall be removed from the sample by screening. The method of screening shall be stated in the test report and shall be chosen to give results similar to those obtained by the industrial screening of the pulp. Complete the preparation of the screened pulps as described in 7.2.

8 Procedure

8.1 Test portion

Before weighing the test portions, condition the samples for not less than 20 min in the atmosphere near the balance.

Weigh 500 ± 5 mg of the pulp. At the same time, weigh a separate test portion for the determination of the dry matter content in accordance with ISO 638.

8.2 Determination

Disintegrate the test portion in the disintegrator (6.1) in 250 ml of water at 25 to 26 °C until free from fibre clots and large bundles. Transfer the disintegrated test portion to the reaction flask (6.2.1) using 135 ml of water to rinse the disintegrator. Place the flask on the support in the water bath (6.4) and start the stirrer (6.3).

Connect the separating funnel (6.2.2) and evacuate the flask by means of the vacuum-pump (6.5). Close the stop-cock of the funnel, remove the stopper and place 10 ml of the hydrochloric acid solution (5.2) in the funnel.

Suck down the acid without admitting air and simultaneously start the stop-watch (6.5). Rinse the funnel with 10 ml of water and suck it down. Pipette 15,0 ml of the sodium hypochlorite solution (5.1) into the funnel and suck it down after exactly 2 min. Do not stop the watch at this stage. Rinse the funnel with 5 ml of water and suck it down.

Add 20 ml of the potassium iodide solution (5.3) to the funnel and suck it down exactly 17 min after adding the hydrochloric acid solution. Rinse the funnel with 50 ml of water, suck it down, and shake the flask to dissolve gaseous chlorine. Add 50 ml of water to the funnel and suck it down; leave the stop-cock open and remove the funnel. Titrate with the sodium thiosulphate solution (5.4), using the starch solution (5.5) as indicator. Record the consumption as V_1 ml.

Perform a blank test using the same procedure and record the consumption as V_2 ml.

NOTE — For pulps with very low chlorine consumptions, use a smaller volume of sodium hypochlorite solution (5.1) and increase the volume of water in proportion. Carry out the blank test with the same volumes of sodium hypochlorite and water. For titrations, use a standard volumetric sodium thiosulphate solution of lower concentration than that stated in 5.4.

Carry out two determinations.

9 Expession of results

9.1 Calculate the fraction r of added chlorine not consumed in the determination by means of the formula

$$r = \frac{V_1}{V_2}$$

where

V_1 is the volume, in millilitres, of standard volumetric sodium thiosulphate solution (5.4) consumed in the titration of the test portion;

V_2 is the volume, in millilitres, of standard volumetric sodium thiosulphate solution (5.4) consumed in the titration in the blank test.

If r is less than 0,5, repeat the determination with a smaller test portion. If r is greater than 0,5, obtain the correction factor f from the table.

9.2 The chlorine consumption X, expressed as a percentage by mass, is given by the formula :

$$X = \frac{3{,}546\, f\,(V_2 - V_1)\, c}{m}$$

c is the concentration, in moles per litre, of the standard volumetric sodium thiosulphate solution;

m is the mass, in grams, of the test portion, calculated on an oven-dry basis.

NOTE — For calculating the chlorine consumption X, the expression

$$f = \frac{1}{2}\left(1 + \frac{V_2}{V_2 - V_1} \ln \frac{V_2}{V_1}\right)$$

has been used. It has been derived on the basis of certain assumptions that are generally accepted in the theory of pulp chlorination. It has been proved experimentally and is valid only if r is greater than 0,5.

Report the result as the mean of the two determinations to three significant figures.

10 Test report

The test report shall include the following particulars :

a) all the information necessary for the complete identification of the sample;

b) a reference to this International Standard;

c) the results and the form in which they are expressed;

d) in the case of unscreened pulp (7.3), the method of screening;

e) any unusual features noted during the determination;

f) any operations not specified in this International Standard, or regarded as optional, which might have affected the results.

Table — Factor f for correction of the chlorine consumption

r	0,00	0,01	0,02	0,03	0,04	0,05	0,06	0,07	0,08	0,09
0,5	1,193	1,187	1,181	1,175	1,170	1,164	1,159	1,154	1,148	1,143
0,6	1,139	1,134	1,129	1,124	1,120	1,115	1,111	1,107	1,103	1,098
0,7	1,094	1,091	1,087	1,083	1,079	1,075	1,072	1,068	1,065	1,061
0,8	1,058	1,055	1,051	1,048	1,045	1,042	1,039	1,036	1,033	1,030
0,9	1,027	1,024	1,021	1,018	1,016	1,013	1,010	1,008	1,005	1,003

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[2] KYRKLUND, B., ET STRANDELL, G., *Paperi ja Puu — Papper och Trä* (Paper and Timber) **51** (1969) : 4a,299.

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