INTERNATIONAL STANDARD

ISO 3260

Third edition
2015-04-01

Pulps — Determination of chlorine consumption (Degree of delignification)

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

Reference number
ISO 3260:2015(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 3260:1982), of which it constitutes a minor revision.

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.

Pulps — Determination of chlorine consumption (Degree of delignification)

WARNING — The method specified in this International Standard involves the use of hazardous chemicals. Appropriate precautions must be taken to ensure the proper use and disposal of these chemicals.

1 Scope

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 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
chlorine consumption of a pulp
amount of active chlorine consumed by a pulp under the conditions specified in this International Standard

Note 1 to entry: The chlorine consumption of a pulp is expressed as a percentage by mass.

4 Principle

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

Determination of the residual chlorine, which shall 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

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 thiosulfate, 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 Figure 1, consisting of the following.

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 ml 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 °C ± 1 °C for at least 20 min and provided with a support for the flask.

6.5 Vacuum-pump.

6.6 Stop-watch.

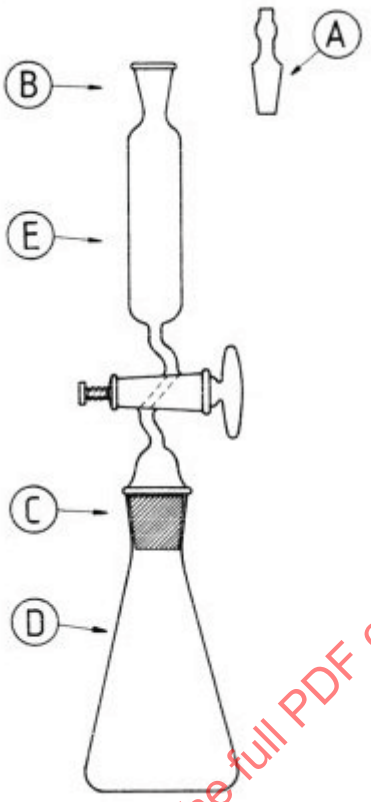

Figure 1 — Apparatus for determination of the chlorine consumption of pulp

7 Preparation of sample

7.1 Air-dried pulp sheets.

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

7.2 Screened slush pulps.

Make a 3 g 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 mg ± 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 °C 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 (6.6). 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 thiosulfate 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.

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 thiosulfate solution of lower concentration than that stated in 5.4.

Carry out two determinations.

9 Expression of results

9.1 Calculate the fraction r of added chlorine not consumed in the determination by means of the Formula (1):

$$r = \frac{V_1}{V_2} \tag{1}$$

where

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

V_2 is the volume, in millilitres, of standard volumetric sodium thiosulfate 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 Table 1.

Table 1 — 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

Table 1 *(continued)*

r	0,00	0,01	0,02	0,03	0,04	0,05	0,06	0,07	0,08	0,09
0,9	1,027	1,024	1,021	1,018	1,016	1,013	1,010	1,008	1,005	1,003

9.2 The chlorine consumption X, expressed as a percentage by mass, is given by the Formula (2):

$$X = \frac{3{,}546\, f\,(V_2 - V_1)\, c}{m} \tag{2}$$

where

c is the concentration, in moles per litre, of the standard volumetric sodium thiosulfate 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, i.e. ISO 3260;

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 the International Standard, or regarded as optional, which might have affected the results.