TC 6

INTERNATIONAL STANDARD ISO 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

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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 drawn up by Technical Committee ISO/TC 6, *Paper, board and pulps*, and circulated to the Member Bodies in April 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Germany	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	U.S.S.R.

No Member Body expressed disapproval of the document.

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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/R 302, *Determination of the Kappa number of pulp (Degree of delignification).* 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/R 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 the pulp under the conditions specified in this International Standard. The chlorine consumption 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 shall be more than 50 % of the amount added, 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 reagent 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 a total alkalinity corresponding to a pH of 12,0 ± 0,5, measured with a glass electrode.

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

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

5.4 Sodium thiosulphate ($Na_2S_2O_3$), 0,2 N standard volumetric solution. The normality shall be known to ± 0,000 4 N.

5.5 Starch indicator solution, 2 g/l.

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) with a standard ground joint (C), volume 750 ml.

6.2.2 Separating funnel (E), volume 50 to 100 ml, with standard ground joints (B and C) and a glass socket (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.

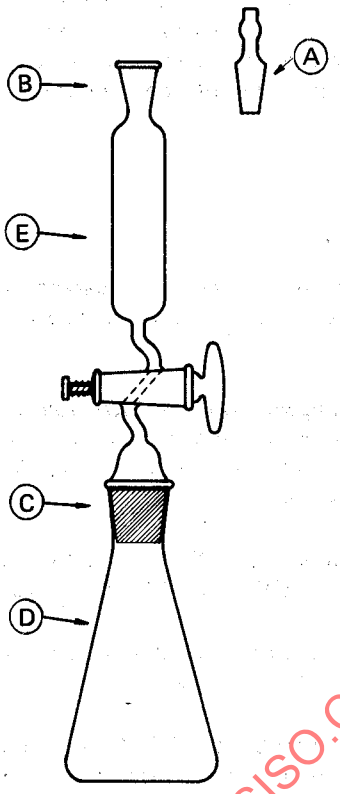

FIGURE — **Apparatus for determination of the chlorine consumption of pulp**

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 in 7.2.

8 PROCEDURE

8.1 Preparation of test portion

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

Weigh out 500 ± 5 mg of the pulp. At the same time, weigh out a separate portion for the determination of dry matter content in accordance with ISO/R 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 socket and add 10 ml of the hydrochloric acid (5.2) to the funnel.

Suck down the acid without admitting air and start the stop-watch (6.5) simultaneously. 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. 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 starch (5.5) as the indicator. Record the consumption as V_1 ml.

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

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

Carry out two determinations.