INTERNATIONAL STANDARD

ISO 10716

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Paper and board — Determination of alkali reserve

Papier et carton — Détermination de la réserve alcaline

Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10716 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

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Introduction

Papers produced to be stable for long time periods normally contain some alkaline filler, such as calcium carbonate, as an alkali reserve to prevent attack from acid substances in ambient air or formed by deterioration of substances in the paper. Specifications for paper permeance may require a minimum alkali reserve. This ISO Standard is intended for checking the amount of alkali reserve present.

Normally, the required alkali reserve is obtained by adding some form of calcium carbonate to the paper furnish, but other substances can also be used for the purpose. By expressing the test result in moles per kilogram of alkaline substances and not as a calcium carbonate content, no confusion arises when alkaline substances other than calcium carbonate are used.

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Paper and board — Determination of alkali reserve

1 Scope

This International Standard specifies a method for the determination of the alkali reserve of papers and boards. It is intended for products that contain alkaline pigments or other alkaline material, added in order to improve their resistance to acid attack (degradation).

This International Standard is not applicable to laminated, printed or otherwise processed grades that will not disintegrate completely by the procedure described.

The result obtained will include alkaline pigments contained in the coating of a coated paper.

NOTE 1 Such alkaline coating will protect the core of the paper from acid substances in ambient air, but its effect on acid substances generated in the base paper itself is uncertain.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10716. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10716 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 186:—[1], *Paper and board — Sampling to determine average quality.*

ISO 287:1985, *Paper and board — Determination of moisture content — Oven drying method.*

3 Definitions

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

3.1 alkali reserve (in paper and board): Compound, such as calcium carbonate, that neutralizes acid that might be generated as a result of natural ageing or from atmospheric pollution, determined as specified in this International Standard.

4 Principle

Digestion of the sample in water containing a measured amount of hydrochloric acid. Heating of the slurry to boiling and titration of unreacted hydrochloric acid with sodium hydroxide solution.

5 Reagents

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

5.1 Hydrochloric acid, standard reference solution, c(HCl) = 0,10 mol/l ± 0,001 mol/l.

5.2 Sodium hydroxide solution, titrant, c(NaOH) = 0,1 mol/l.

5.3 Methyl red, indicator solution for acidometric titration.

Dissolve 0,2 g of methyl red {2-[4-(dimethylamino)-phenylazo]benzoic acid} in 100 ml of ethanol.

6 Apparatus

Ordinary laboratory equipment.

7 Sampling and preparation of sample

Ensure that the sample is representative of the lot to be tested. Where applicable, follow the instructions given in ISO 186.

Select from the sample enough test pieces to provide for the testing to be done, ensuring that these pieces are representative of the whole sample. Tear the

1) To be published. (Revision of ISO 186:1985)

pieces into smaller pieces, about 15 mm × 15 mm, and split thick board pieces. Wear protective gloves when handling the sample.

8 Test procedure

Carry out this procedure in duplicate.

Weigh about 1 g of sample to the nearest 0,001 g. At the same time weigh a separate sample portion for dry matter content determination in accordance with ISO 287.

Transfer the test portion to a clean 250 ml or 300 ml conical flask. Add about 100 ml of distilled water (or more if required to soak the sample portion) and boil the mixture gently for 5 min. Allow the mixture to cool somewhat. With a pipette, add 20,0 ml of hydrochloric acid (5.1).

NOTE 2 The amount of acid, 20 ml or 2 mmol, is sufficient to neutralize an alkali reserve of up to 2 mol/kg [10 % (*m*/*m*) $CaCO_3$]. If the paper contains more than 1,5 mol/kg alkali reserve [7,5 % (*m*/*m*) $CaCO_3$], use a smaller test sample or a larger volume of hydrochloric acid.

Bring the mixture to boiling again and allow it to cool for at least 15 min. Titrate with the sodium hydroxide solution (5.2) to the first lemon-yellow, using three drops of the methyl red solution (5.3) as the indicator.

If less than 5 ml of the sodium hydroxide solution are required to reach the end-point, repeat the procedure with a smaller test sample or with a larger volume of hydrochloric acid.

If a trace of pink indicator remains adsorbed in the surface of the fibres, boil the slurry briefly to desorb the pink colour. Usually a further drop of sodium hydroxide solution will restore the lemon-yellow colour.

Carry out a blank determination by the same procedure, but omit the sample.

NOTE 3 If the sample is dyed so that the end-point cannot readily be detected, an electrometric titration may be used. However, glass electrodes are sensitive to the presence of suspended matter. If interference from suspended matter is observed, the suspension should be filtered before titration. If such a modification to the test procedure has been carried out, this shall be stated in the test report.

9 Calculation

Calculate the alkali reserve, X, expressed in moles per kilogram, from the expression

$$X = \frac{V_0 - V_1}{V_0} \times \frac{V_2 \cdot c(\mathrm{HCl})}{m}$$

where

V_0 is the volume, in millilitres, of sodium hydroxide solution used in the blank titration;

V_1 is the volume, in millilitres, of sodium hydroxide solution used in the sample titration;

V_2 is the volume, in millilitres, of hydrochloric acid used (normally 20 ml);

$c(\mathrm{HCl})$ is the concentration of the hydrochloric acid solution (5.1), in moles per litre;

m is the mass, in grams, of the oven-dry sample.

NOTE 4 The expression above is obtained by combining the equation for the blank titration

$$V_0 \cdot c(\mathrm{NaOH}) = V_2 \cdot c(\mathrm{HCl})$$

with that for the sample titration

$$V_1 \cdot c(\mathrm{NaOH}) = V_2 \cdot c(\mathrm{HCl}) - Xm$$

and solving for X.

Duplicate determinations should agree within 0,07 mol/kg. If this is not the case, repeat the procedure with two more sample portions.

Calculate the mean result and round it off to the first decimal place.

NOTE 5 The alkali reserve, as defined by this International Standard, represents moles per kilogram of an alkali in which the cation is monovalent. One mole of acid is equivalent to 0,5 mol of calcium carbonate, or 50 g of $CACO_3$. One per cent of calcium carbonate thus gives an alkali reserve of 0,2 mol/kg.

10 Precision

In an interlaboratory study conducted within ISO TC 46/SC 10/WG 1, the alkali reserve of a range of printing and writing papers was tested by laboratories in different countries. The procedure used was similar to that described in this International Standard. Some of the results (in moles per kilogram) are quoted in table 1. The data were obtained under reproducibility conditions.

Table 1 — Precision data

Sample No.	Number of participating laboratories	Mean of the results	Standard deviation of repro-ducibility
1	12	3,48	0,54
2	12	3,18	0,18
3	12	2,81	0,17
4	12	1,85	0,07
5	12	0,50	0,06
6	12	0,27	0,06
7	11	0,36	0,06
8	9	0,08	0,02
9	9	0,04	0,03

11 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) date and place of test;

c) all information necessary for complete identification of the sample tested;

d) the mean alkali reserve, expressed in moles per kilogram to the nearest 0,1 mol/kg;

e) any departure from the procedure described in this International Standard, or any other circumstances that may have affected the test results.

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