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Paper and board — Determination of alkali reserve

Papier et carton — Détermination de la réserve alcaline

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 6 *Paper, board and pulps*.

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This second edition cancels and replaces the first edition (ISO 10716:1994), of which it constitutes a minor revision. The main changes are as follows:

— Clause 2 has been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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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 permanence can require a minimum alkali reserve.

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 this purpose. By expressing the test results 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 document specifies a method for the determination of the alkali reserve of papers and boards. lt is intended for products that contain alkaline pigments or other alkaline material, added to improve their resistance to acid attack (degradation).

This document 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 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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ISO 186, *Paper and board — Sampling to determine average quality*

ISO 287, *Paper and board — Determination of moisture content of a lot — Oven-drying method*

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ISO 638-1, *Paper, board, pulps and cellulosic nanomaterials – Determination of dry matter content by oven-drying method – Part 1: Materials in solid form*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
alkali reserve
compound, such as calcium carbonate, that neutralizes acid that can be generated as a result of natural aging or from atmospheric pollution

4 Principle

The sample is digested by boiling in water containing a measured amount of hydrochloric acid. The unreacted hydrochloric acid is titrated with a sodium hydroxide solution.

5 Reagents

Use only reagents of recognized analytical grade, and distilled water or water of equivalent purity.

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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]lbenzoic acid) in 100 ml of ethanol.

6 Apparatus

The usual laboratory apparatus shall be used.

7 Sampling and preparation of test pieces

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

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8 Procedure

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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 test portion for dry matter content determination in accordance with ISO 287 or ISO 638-1, as applicable.

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 test 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).

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 portion or with a larger volume of hydrochloric acid.

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

NOTE 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 portion or a larger volume of hydrochloric acid.

If the sample is dyed so that the end-point cannot readily be detected, an electrometric (potentiometric) or automatic titrator 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.

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9 Calculation

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

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

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(HCl) is the concentration of the hydrochloric acid solution (5.1) in moles per liter;

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

NOTE The expression above is obtained by combining the Formula for the blank titration:

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

with that for the sample titration

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

and solving for X.

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

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

NOTE In this document, the alkali reserve 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

The results of an interlaboratory study to determine the precision of this method are presented in Annex A.

11 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 10716:—;

b) date and place of test;

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

d) the mean alkali reserve, calculated according to Clause 9, expressed in moles per kilogram to the nearest 0,1 mol/kg;

e) any unusual features observed during the test;

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

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Annex A

Precision

In an interlaboratory study conducted within ISO TC 46/SC 10, 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 document. Some of the results (in moles per kilogram) are quoted in Table A.1. The data were obtained under reproducibility conditions.

Table A.1 — Precision data

Sample No.	Number of participating laboratories	Mean of the results moles/kilogram	Standard deviation of reproducibility
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

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