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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 777

PULPS

DETERMINATION OF CALCIUM CONTENT

1st EDITION
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BRIEF HISTORY

The ISO Recommendation R 777, *Pulps – Determination of calcium content,* was drawn up by Technical Committee ISO/TC 6, *Paper, board and pulps,* the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1965 and led, the same year, to the adoption of a Draft ISO Recommendation.

In November 1966, this Draft ISO Recommendation (No. 1068) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	India	Portugal
Australia	Iran	Romania
Belgium	Israel	Spain
Brazil	Italy	Sweden
Bulgaria	Japan	Switzerland
Canada	Korea, Rep. of	Turkey
Czechoslovakia	Korea, D.P.R. of	U.A.R.
Denmark	Mexico	United Kingdom
Finland	Netherlands	U.S.A.
France	New Zealand	U.S.S.R.
Germany	Norway	Yugoslavia

One Member Body opposed the approval of the Draft :

Cuba

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 777 July 1968

PULPS

DETERMINATION OF CALCIUM CONTENT

1. SCOPE

This ISO Recommendation describes a method for the determination of the calcium content of pulp.

2. FIELD OF APPLICATION

This method applies to all kinds of pulp.

3. PRINCIPLE OF THE METHOD

The pulp is ashed and the ash is dissolved in nitric acid. Calcium is determined volumetrically with solution of the disodium salt of ethylenediaminetetraacetic acid at a pH above 12. Other metal ions are masked with triethanolamine.

4. REAGENTS

All reagents should be of analytical grade and the distilled water used should not contain copper.

4.1 *Nitric acid*, about 5 M. Dilute 325 ml of nitric acid (d = 1.4) to 1 litre with distilled water.

4.2 *Hydrochloric acid*, about 6 M. Dilute 500 ml of hydrochloric acid (d = 1.19) to 1 litre with distilled water.

4.3 *Hydrochloric acid*, 1 M. Dilute 82.5 ml of hydrochloric acid (d = 1.19) to 1 litre with distilled water.

4.4 *Potassium hydroxide solution*, about 8 M, 449 g of potassium hydroxide per litre.

4.5 *Triethanolamine solution.* Dissolve 100 g of triethanolamine $[(C_2H_5O)_3\ N]$ in distilled water and dilute to 1 litre.

4.6 *Hydroxylamine hydrochloric solution.* Dissolve 2 g of hydroxylamine hydrochloride ($NH_2OH.HCl$) in 100 ml of distilled water.

4.7 *Standard calcium solution*, 0.01 M. Weigh 1 g of chemically pure calcium carbonate, dried at a temperature not exceeding 200 °C, to the nearest 0.001 g into a 500 ml beaker. Add 100 ml of distilled water and add drop by drop from a burette the smallest amount (about 20 ml) of 1 M hydrochloric acid, necessary to dissolve the carbonate. Boil out the carbon dioxide, cool, and transfer quantitatively by rinsing with distilled water into a 1 litre volumetric flask and dilute to the mark. Calculate M_{Ca} = g $CaCO_3$/100.1 as the molarity of the standard solution.

4.8 *Solution of disodium salt of ethylenediaminetetraacetic acid* (EDTA), 0.01 M. Dissolve 3.80 g of the disodium salt of ethylenediaminetetraacetic acid, ($C_{10}H_{14}O_8N_2Na_2 . 2H_2O$), in distilled water in a 1 litre volumetric flask and dilute to the mark.

Keep the solution in a polyethylene bottle, standardize the solution by adding to a conical flask 20.00 ml of 0.01 M standard calcium solution, 40 ml of distilled water, 5 ml of potassium hydroxide solution, 2 ml of hydroxylamine hydrochloride solution and about 100 mg of cal-red indicator. Titrate with the EDTA solution to a colour change from wine-red to pure blue. Titrate also a blank solution containing all the reagents except the standard calcium solution, which is replaced by an equal volume of distilled water.

Calculate the molarity of the EDTA solution from the following equation :

$$M_{EDTA} = \frac{a \times M_{Ca}}{b - c}$$

where

a is the volume of the standard calcium solution, expressed in millilitres,

b is the volume of the EDTA solution, consumed by the standard calcium solution, expressed in millilitres,

c is the volume of the EDTA solution, consumed in the blank determination, expressed in millilitres,

M_{Ca} is the molarity of the standard calcium solution.

The solutions mentioned under clauses 4.4, 4.7 and 4.8 should preferably be stored in polyethylene bottles.

4.9 *Cal-red indicator.* Rub 100 mg of 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthyl-azo)-3-naphthoic acid in a mortar with 10 g of sodium sulphate, Na_2SO_4, to form a homogeneous mixture. Store in a dark bottle.

NOTE. – When the sample contains more than 0.03 mg of copper the indicator is blocked. In this case use calcein as the indicator or mask the copper present by addition of 5 ml of a 1 % solution of potassium cyanide.

5. APPARATUS

Dishes of quartz, platinum or porcelain.

6. PROCEDURE

6.1 Preparation of test piece

Weigh about 10 g of air dry pulp, previously torn to pieces of suitable size, to the nearest 0.01 g.

NOTE. – For pulps containing more than 500 mg of calcium per kilogramme, weigh an amount of pulp corresponding to a maximum of 5 mg of calcium or use a standardized 0.05 M EDTA solution for the titration.

At the same time weigh out a separate sample for dry matter determination in accordance with ISO Recommendation R 638, **Pulps – Determination of dry matter content.*

6.2 Determination

Ash the test piece in a dish as described in ISO Recommendation R . . ., **Pulps – Determination of ash.*

NOTE. – For cleaning of dishes, wash carefully; remove any spots in the dishes by rubbing with fine sand. Boil the dish four times with 6 M hydrochloric acid.

* At present, at the stage of a draft proposal, (6N 392)

The temperature recommended in this draft proposal concerning the determination of ash in pulp is 575 ± 25 °C; ignition of pulp is achieved in a muffle furnace adjusted for maintaining temperature within the specified range, previous ignition being obtained by means of the low flame of a gas burner.