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# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION



# ISO RECOMMENDATION

# R 779

## PULPS

## DETERMINATION OF IRON

1st EDITION

July 1968



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## BRIEF HISTORY

The ISO Recommendation R 779, *Pulps – Determination of iron,* 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. 1070) 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 | Romania |
| Australia | Iran | Spain |
| Belgium | Israel | Sweden |
| Brazil | Italy | Switzerland |
| Bulgaria | Japan | Turkey |
| Canada | Korea, Rep. of | U.A.R. |
| Czechoslovakia | Mexico | United Kingdom |
| Denmark | Netherlands | U.S.A. |
| Finland | New Zealand | U.S.S.R. |
| France | Norway | Yugoslavia |
| Germany | Portugal | |

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 779 July 1968

# PULPS

# DETERMINATION OF IRON



## 1. SCOPE

The ISO Recommendation describes a method for the determination of the iron 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 hydrochloric acid. The concentration of iron is determined colorimetrically with ortho-phenanthroline by measuring the optical density at 510 nm.

## 4. REAGENTS

All reagents should be of analytical grade.

4.1 *Hydrochloric acid*, about 6 M. Dilute 500 ml of hydrochloric acid ($d$ = 1.19) with 500 ml of distilled water.

4.2 *Sodium acetate solution*, 4 M. Dissolve 540 g of sodium acetate ($Na\ COOCH_3\ .\ 3\ H_2O$) in distilled water and dilute to 1 litre.

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

4.4 *Ortho-phenanthroline hydrochloride solution.* Dissolve 1 g of ortho-phenanthroline hydrochloride ($C_{12}H_8N_2\ .\ HCl\ .\ H_2O$) in 100 ml of distilled water. Keep the solution in the dark. Use only a colourless solution.

4.5 *Standard iron solution*, 0.1 mg of iron per millilitre. Dissolve 0.100 g of pure iron wire in the smallest possible quantity of hydrochloric acid ($d$ = 1.19) in a 1 litre volumetric flask and dilute to the mark with distilled water.

## 5. APPARATUS

5.1 *Dishes of platinum, of porcelain or of quartz.*

5.2 *Spectrophotometer or filter colorimeter.*

## 6. CALIBRATION

Dilute the standard iron solution (4.5) ten times so that 1 ml corresponds to 0.01 mg of Fe. Pipette aliquots of 5.0, 10.0, 15.0 and 20.0 ml of the diluted solution into 50 ml volumetric flasks. Use a fifth flask for the preparation pf a reference solution. Add to each flask 1 ml of hydroxylamine hydrochloride solution (4.3) and 1 ml of orthophenanthroline hydrochloride solution (4.4) and dilute to the mark with distilled water. After 15 minutes measure the optical density at 510 nm with the iron free solution as a reference.

NOTE. – Avoid exposure of the coloured solution to direct sunlight.

The iron concentrations of the coloured solutions are 1.0, 2.0, 3.0 and 4.0 mg of Fe per litre respectively. Plot on a diagram the optical density values divided by the length of the cell, against the iron concentrations and check that the points lie on a straight line going through the origin.

## 7. PREPARATION OF SAMPLE

Tear the air-dry sample into pieces of a suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

## 8. PROCEDURE

### 8.1 Preparation of test piece

Weigh about 10 g of pulp (or 5 g for iron contents above 20 mg/kg) to the nearest 0.01 g. 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.*

### 8.2 Determination

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

NOTE. – Cleaning of dishes : wash the dish thoroughly; remove any spots in platinum dishes by cleaning with fine sand. Boil the dish four times with 6 M hydrochloric acid (4.1) and avoid any possible contact with iron. Check in the following way that the dish is free from iron : heat about 2 ml of the hydrochloric acid 6 M, diluted with about 10 ml of distilled water in the dish. Allow to cool and add 1 ml of hydroxylamine hydrochloride solution, 1 ml of ortho-phenanthroline hydrochloride solution and 10 ml of sodium acetate solution. No red colour should appear.

In order to obtain a reagent blank, take another clean dish of the same type as used for the ashing of the test piece and treat as described below.

To each dish add 5 ml of 6 M hydrochloric acid (4.1) and evaporate to dryness on a steam bath. Repeat this once and then treat the residue with another portion of 2.5 ml of hydrochloric acid (4.1) and heat for 5 minutes on the steam bath.

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

The temperature foreseen 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.