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R 1830

PULPS

DETERMINATION OF MANGANESE

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November 1970

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BRIEF HISTORY

The ISO Recommendation R 1830, *Pulps – Determination of manganese,* 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 led to the adoption of Draft ISO Recommendation No. 1830, which was circulated to all the ISO Member Bodies for enquiry in April 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Canada	Korea, Rep. of	Sweden
Czechoslovakia	Netherlands	Switzerland
Finland	New Zealand	Turkey
France	Norway	U.A.R.
Germany	Peru	United Kingdom
Greece	Portugal	U.S.A.
India	Romania	U.S.S.R.

The following Member Body opposed the approval of the Draft :

Bulgaria

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

ISO Recommendation | R 1830 | November 1970

PULPS

DETERMINATION OF MANGANESE

1. SCOPE

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

2. FIELD OF APPLICATION

This method applies to all kinds of pulp.

3. PRINCIPLE

Ashing of the pulp and dissolution of the ash in nitric acid. Oxidation of the manganese to permanganate with sodium periodate followed by colorimetric determination by measuring the optical density at 525 nm.

4. REAGENTS

All reagents used should be of recognized analytical reagent quality. Distilled water or water of equivalent purity should be used in the test.

4.1 *Sodium sulphite solution,* (Na_2SO_3) 50 g/l.

4.2 *Nitric acid,* 1.5 M, 100 ml of nitric acid (ρ 1.4 g/ml) per litre.

4.3 *Periodate-phosphoric acid solution,* 50 g of sodium periodate ($NaIO_4$) and 200 ml of 85 % phosphoric acid (H_3PO_4) (ρ 1.70 g/ml) per litre.

4.4 *Standard manganese solution,* 0.1 mg of manganese per millilitre.

Weigh 0.2749 g of manganese sulphate ($MnSO_4$), dried at 300 °C, into a 1 litre volumetric flask. Dissolve with water and dilute to the mark.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Dishes of platinum, porcelain or quartz.*

5.2 *Spectrophotometer* or *filter colorimeter.*

5.3 *Cells,* for use in the spectrophotometer or filter colorimeter.

6. CALIBRATION

Dilute the standard manganese solution (4.4) ten times so that 1 ml corresponds to 0.01 mg of manganese. Pipette aliquots of 1, 2, 5 and 10 ml of the diluted solution into 25 ml volumetric flasks. Without any further dilution heat the solutions by placing the flasks in a steam bath and add 1 ml of the periodate-phosphoric acid solution (4.3) to each flask. Keep the flasks in the steam bath for 5 minutes after the addition and then dilute to the mark. Cool to 20 ± 2 °C and adjust the volume. The temperatures of the solutions in the series should not differ by more than 3 °C. Measure the optical density at 525 nm with a solution containing 1 ml of periodate-phosphoric acid solution and 24 ml of water as a reference. Divide the reading by the length of the cell.

The manganese concentrations of the coloured solutions are 0.4, 0.8, 2.0 and 4.0 mg of manganese per litre respectively. Plot the optical density values divided by the length of the cell against the manganese concentrations, and check that the points lie on a straight line going through the origin.

7. PREPARATION OF THE SAMPLE

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

8. PROCEDURE

8.1 Preparation of test piece

Weigh to the nearest 0.01 g about 20 g of pulp (or 10 g for manganese contents above 5 mg/kg) into a dish. At the same time weigh out a separate test 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 1762, *Pulps – Determination of ash**. Add three drops of sodium sulphite solution (4.1) to the ash and dissolve in a maximum of 5 ml of nitric acid (4.2). Place the dish on a steam bath and evaporate to dryness.

Add a few drops of nitric acid and transfer the contents of the dish with the aid of water to a 25 ml volumetric flask. Heat the contents by placing the flask in a steam bath. Add 1 ml of the periodate-phosphoric acid solution (4.3) and let the flask remain in the steam bath for 5 minutes after the addition. Dilute with water to the mark, cool to 20 ± 2 °C and adjust the volume exactly. The temperature should not differ by more than 3 °C from that of the manganese solutions (see section 6) used for the calibration. If necessary, centrifuge the solution (see note). Measure the optical density at 525 nm with a solution containing 1 ml of periodate-phosphoric acid solution and 24 ml of water as a reference. Divide the reading by the length of the cell.

NOTE. – If the solution is turbid, the turbidity can be removed by centrifuging. Do not filter the solution.

* The temperature foreseen in ISO Recommendation R 1762 concerning the determination of ash in pulp is 575 ± 25 °C; ignition of the pulp is achieved in a muffle furnace adjusted to maintain the temperature within the specified range, previous ignition being obtained over a low flame of a gas burner.