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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 624

PULPS

EXTRACTION OF MATERIALS SOLUBLE IN DICHLOROMETHANE

1st EDITION

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 624, *Pulps—Extraction of materials soluble in dichloromethane*, 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 1963 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In January 1966, this Draft ISO Recommendation (No. 891) 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	Germany	Romania
Australia	India	South Africa,
Austria	Iran	Rep. of
Belgium	Israel	Spain
Brazil	Japan	Sweden
Bulgaria	Korea, Rep. of	Switzerland
Canada	Mexico	Turkey
Chile	Netherlands	U.A.R.
Czechoslovakia	New Zealand	United Kingdom
Finland	Poland	U.S.A.
France	Portugal	Yugoslavia

Two Member Bodies opposed the approval of the Draft:

Italy
Norway

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in October 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 624 October 1967

PULPS

EXTRACTION OF MATERIALS SOLUBLE IN DICHLOROMETHANE

1. SCOPE

This ISO Recommendation describes a method for the separation of pulp components soluble in dichloromethane.

2. FIELD OF APPLICATION

This method may be used for all kinds of chemical and semi-chemical pulp (see Appendix X).

3. PRINCIPLE OF THE METHOD

The pulp is treated with dichloromethane (see Appendix Y) in a Soxhlet apparatus. After at least 24 extraction cycles the solvent is evaporated and the residue is dried at a temperature of 103 ± 2 °C for a period not exceeding 16 hours and subsequently weighed.

4. REAGENT

4.1 *Dichloromethane*, CH_2Cl_2, 98 to 100 %, and with a dry matter content of less than 5 mg per litre. The commercial product as a rule should be re-distilled. Collect the fraction distilling over between 38 to 41 °C and store in a brown-glass bottle. The distillate should be neutral (see Note, section 7).

5. APPARATUS

5.1 *Extraction apparatus* of Soxhlet type entirely made of glass with ground-in condenser, extractor and flask. The capacity of the extractor should be 60 to 120 ml.

5.2 *Adjustable heater.**

5.3 *Drying oven*, ventilated, capable of maintaining an air temperature of 103 ± 2 °C.

5.4 *Balance* accurate to 0.5 mg.

6. PREPARATION OF SAMPLE

Cut or tear the air-dry pulp into pieces of about 1.5 cm × 1.5 cm, in sufficient quantity for carrying out at least two determinations.

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* e.g. electric heater, adjustable to at least 200 W per extraction unit.

7. PROCEDURE

Weigh about 10 g of pulp to the nearest 0.01 g. At the same time weigh out a separate sample for dry matter content determination according to ISO Recommendation R 638, *Pulps—Determination of dry matter content.*

Introduce into the draining tube of the Soxhlet apparatus a small wad of surgical cotton previously extracted with the solvent being used and transfer the test sample to the extractor. Connect to the extractor a flask which has been heated to 103 ± 2 °C, cooled and subsequently weighed to the nearest 0.5 mg. Add to the flask a quantity of dichloromethane corresponding to 1½ of the volume of the extractor. Connect the condenser and start the extraction.

Extract for at least 3 hours, adjusting the boiling rate so that the extractor is drained 8 times per hour. If the draining is slower, extract for a correspondingly longer time. The total number of extraction cycles should be at least 24. At the end of the extraction, the extract solution should be clean and free of fibres. Distil off the solvent. Evaporate finally on a steam bath, and dry the flask to constant mass * for a period not exceeding 16 hours in the drying oven at 103 ± 2 °C. Cool the flask in a desiccator for 45 min and weigh to the nearest 0.5 mg (see Appendix Z).

NOTE. — As the solvent is poisonous, adequate ventilation should be provided.

8. EXPRESSION OF RESULTS

Carry out at least two determinations and calculate as follows:

$$X = \frac{a}{m} 100$$

where

a = mass of the dichloromethane extract, in grammes,

m = mass of pulp, calculated on an oven-dry basis, in grammes,

X = dichloromethane extract, per cent.

Report the result to the second decimal place.

9. TEST REPORT

The test report should state the results obtained and indicate all the conditions of the test and any details of procedure regarded as optional or not laid down in the ISO Recommendation and any incidents that may have affected the results.

* Two consecutive weighings should not differ by more than 0.5 mg.