SLOVENSKI STANDARD
SIST ISO 624:1996

01-april-1996

Vlaknine - Določanje topnih snovi v diklormetanu

Pulps -- Determination of dichloromethane soluble matter

Pâtes -- Détermination des matières solubles dans le dichlorométhane

Ta slovenski standard je istoveten z: **ISO 624:1974**

ICS:

85.040 Vlaknine Pulps

SIST ISO 624:1996 **en**

INTERNATIONAL STANDARD 624

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Pulps — Determination of dichloromethane soluble matter

Pâtes — Détermination des matières solubles dans le dichlorométhane

First edition — 1974-09-15

To be withdrawn

UDC 676.014 **Ref. No. ISO 624-1974 (E)**

Descriptors : papers, paper pulps, chemical analysis, determination of content, soluble matter, dichloromethane.

Price based on 2 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 6 has reviewed ISO Recommendation R 624 and found it suitable for transformation. International Standard ISO 624 therefore replaces ISO Recommendation R 624-1967.

ISO Recommendation R 624 was approved by the Member Bodies of the following countries :

Argentina	France	Portugal
Australia	Germany	Romania
Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Sweden
Bulgaria	Japan	Switzerland
Canada	Korea, Rep. of	Turkey
Chile	Mexico	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
Egypt, Arab Rep. of	New Zealand	Yugoslavia
Finland	Poland	

The Member Body of the following country has subsequently approved this Recommendation :

Norway

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Italy

No Member Body disapproved the transformation of ISO/R 624 into an International Standard.

Printed in Switzerland

Pulps — Determination of dichloromethane soluble matter

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the separation of pulp components soluble in dichloromethane.

This method may be used for all kinds of chemical and semi-chemical pulp.

NOTE — Dichloromethane extraction according to this method and ethanol extraction give estimates of the solvent extractable content of the pulp. The composition of the extract is not established and varies considerably with the nature of the pulp. Besides resin acids, materials such as fats, fatty acids, sterols, terpenes and waxes as well as oxidation and chlorination products, may be included.

Both dichloromethane and ethanol dissolve these resin components during repeated extraction. Ethanol can also dissolve oxidation products of resin, some lignin and cellulose degradation products, and inorganic salts. Thus in general the ethanol extract is larger than the dichloromethane extract.

2 REFERENCE

ISO/R 638, *Pulps — Determination of dry matter content.*

3 PRINCIPLE

Treatment of the pulp with dichloromethane (see note) in a Soxhlet apparatus. After at least 24 extraction cycles, evaporation of the solvent and drying of the residue at a temperature of 103 ± 2 °C for a period not exceeding 16 h and subsequent weighing.

NOTE — Dichloromethane is preferred to ether because of the fire and explosion risk with the latter solvent.

4 REAGENT

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 and 41 °C and store in a brown glass bottle. The distillate should be neutral (see note, clause 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 shall be 60 to 120 ml.

5.2 Adjustable heater, for example an electric heater, adjustable to at least 200 W per extraction unit.

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.

7 PROCEDURE

Weigh about 10 g of pulp to the nearest 0,01 g. At the same time weigh out a separate test portion for dry matter content determination according to ISO/R 638.

Introduce into the draining tube of the Soxhlet apparatus (5.1) a small wad of surgical cotton previously extracted with the solvent being used and transfer the test portion 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 the dichloromethane (4) corresponding to 1 1/2 times the volume of the extractor. Connect the condenser and start the extraction.

Extract for at least 3 h, 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 shall 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[1] for a period not exceeding 16 h 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.[2]

Carry out at least two determinations.

NOTE — As the solvent is poisonous, provide adequate ventilation.

8 EXPRESSION OF RESULTS

Calculate the dichloromethane extract, in percent, by the formula

$$\frac{m_0}{m_1} \times 100$$

where

m_0 is the mass, in grams, of the dichloromethane extract;

m_1 is the mass, in grams, of pulp, calculated on an oven-dry basis.

Report the result to the second decimal place.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

1) Two consecutive weighings shall not differ by more than 0,5 mg.

2) Some extractable components in wood, for example terpenes and esters of fatty acids, are comparatively volatile and are generally removed during pulping. However, in some unbleached sulphite pulps, e.g. hardwood sulphite, some volatile components remain. Some of these are volatilized during the drying time employed in this method.