

# DRAFT INTERNATIONAL STANDARD

## ISO/DIS 21437

ISO/TC 6

Secretariat: SCC

Voting begins on:  
2019-12-20

Voting terminates on:  
2020-03-13

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## Pulps — Determination of carbohydrate composition

*Pâtes — Détermination de la composition en carbohydrate*

ICS: 85.040

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Reference number  
ISO/DIS 21437:2019(E)

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Published in Switzerland

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 6, Paper, board and pulps.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The carbohydrate composition - the contents of the five principal, neutral monosaccharides; arabinose, galactose, glucose, xylose and mannose - provides chemical information about the main polysaccharides in wood pulps. The most commonly used methods are based on acid hydrolysis of the polysaccharides using sulphuric acid, followed by subsequent chromatographic determination of the monosaccharides.

This document describes a method for the determination of the contents of the five principal, neutral monosaccharides; arabinose, galactose, glucose, xylose and mannose, as they appear in wood pulps. The procedure is based on the sulphuric acid hydrolysis of the samples. The monosaccharides are determined either by using high performance anion exchange chromatography with a pulsed amperometric detector (HPAEC-PAD) – subsequently referred to as ion chromatography (IC), or by using gas chromatography with a flame ionization detector (GC-FID) – subsequently referred to as gas chromatography (GC).

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# Pulps — Determination of carbohydrate composition

## 1 Scope

This method describes the determination of the carbohydrate composition in wood pulp samples. This method makes it possible to determine concentrations of individual anhydrous monosaccharides down to 1 mg/g oven-dry pulp.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 7213, *Pulps — Sampling for testing*

ISO 14453, *Pulps — Determination of acetone-soluble matter*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1

#### Carbohydrate composition

Amounts of the five principal, neutral monosaccharides; arabinose, galactose, glucose, mannose and xylose, in a sample, in milligrams per gram.

## 4 Principle

The pulp samples are hydrolysed with sulphuric acid using a two-step technique. The amounts of the different monosaccharides are determined using either ion chromatography (IC) or gas chromatography (GC) in the presence of an internal standard to validate the results. If GC is used, the hydrolysed sample is reduced and acetylated, and the resulting alditol acetates of the monosaccharides are then separated and determined by GC.

## 5 Apparatus

5.1 **Grinder with a 40 mesh** screen or equivalent equipment.

5.2 **Water bath** at a temperature of  $(30 \pm 1)$  °C.

5.3 **Autoclave** at a temperature of  $(120 \pm 3)$  °C

**5.4 Drying oven**, (105 ± 2) °C for determining dry matter content in accordance with ISO 638

The apparatus in 5.5 is used for IC determination only:

**5.5 Ion-chromatograph (IC)** with an anion-exchange column for monosaccharide determination and pulsed amperometric detector (PAD).

Apparatuses in 5.6-5.7 are used for GC-determinations only:

**5.6 Water bath** at a temperature of (40 ± 0,5) °C

**5.7 Gas chromatograph (GC)** with a suitable column for monosaccharide determination and flame ionization detector (FID).

## 6 Reagents

All chemicals shall be of reagent grade or better, unless otherwise indicated

**6.1 Water of high purity**, distilled or deionized

**6.2 Monosaccharide standards**

Monosaccharide standards, for calibration: arabinose, galactose, glucose, mannose and xylose. Prepare standard solutions of appropriate concentrations, each standard solution containing all five monosaccharides.

**6.3 Sulphuric acid, 72%** Prepare as follows: Add 300 ml of water to 1000 ml volumetric flask. Add slowly 670 ml of concentrated sulphuric acid (H<sub>2</sub>SO<sub>4</sub> sp-gr 1,84) while cooling under a cold water tap. When the temperature has reached equilibrium with the ambient temperature, dilute to the mark and mix.

72% H<sub>2</sub>SO<sub>4</sub> is also available commercially.

**6.4 Eluent solution** (for IC determination)

The composition of this solution depends on the type of IC column to be used. Therefore, follow the recommendations given by the IC column supplier.

The following reagents are required only for GC determinations:

**6.5 Ammonia**, NH<sub>3</sub> conc. 25 %

**6.5.1 Ammonia 12 M**

Mix 9 parts ammonia (25 %) with 1 part of water.

**6.6 Potassium hydroxide, KOH,**

**6.6.1 Potassium hydroxide 7.5 M**

Weigh 123 g KOH pellets into a 250 ml beaker. Add approx. 150 ml water while stirring. Transfer the solution into a 250 ml volumetric flask, using an additional 20-30 ml of water to rinse the beaker in order to complete the transfer.

The reaction is exothermic, heat is evolved. Allow the solution to cool to ambient temperature and dilute to the mark with water.



**6.7 Potassium borohydride,  $\text{KBH}_4$** **6.7.1 Potassium borohydride solution**

Dissolve 150 mg  $\text{KBH}_4$  in 250  $\mu\text{l}$  12 M  $\text{NH}_3$  and 750  $\mu\text{l}$  distilled water in a septum vial (4 ml).

This solution shall be freshly prepared every day before use.

**6.8 Acetic acid,  $\text{CH}_3\text{COOH}$ , conc.****6.9 Acetic acid anhydride, conc.****6.10 1-methylimidazole****6.11 Ethanol, 95-99 %****6.12 Sodium sulphate,  $\text{Na}_2\text{SO}_4$ , water-free****6.13 2-Deoxy-galactose****6.13.1 Internal standard solution, 2-deoxy-galactose 20 mg/ml**

Weigh 1,00 g 2-deoxy-galactose to the nearest 0.1 mg, transfer it quantitatively to a 50 ml volumetric flask, and dilute to the mark with distilled water.

Note to entry: Other internal standards than 2-deoxy-galactose, such as fucose or myo-inositol, can also be used. However, this must be specified in the report.

**7 Sampling**

If the analysis is being made to evaluate a lot of a consignment of pulp, the sample shall be taken in accordance with ISO 7213. If the analysis is made on another type of sample, report the origin of the sample, and if possible the sampling procedure. Pulp samples shall be ground before analysis using an appropriate grinder (5.1). Groundwood and high yield pulps containing a significant amount of resins shall be extracted with acetone according to ISO 14453 before testing.

Note 1 to entry Resins, if not extracted from the pulp prior to analysis, would remain insoluble in acid and may interfere with the determination of carbohydrate composition.

Note 2 to entry: Acetone is considered an effective solvent for extracting resin from pulp. Dichloromethane and ethanol/benzene (1:2), as specified in other methods, are not recommended due to health hazards. In particular, benzene is a confirmed carcinogen.

**8 Test Specimens**

Make sure that the test portions taken are representative of the sample received.

**9 Procedure**

Carry out the preparation and testing in duplicate.