

Methods of test for

Coffee and coffee products

**Part 15. Instant coffee: determination of
free and total carbohydrate contents by
high performance anion-exchange
chromatography**

Committees responsible for this British Standard

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- Association of Public Analysts
- British Soluble Coffee Manufacturers' Association
- Coffee Trade Federation Ltd.
- Food and Drink Federation
- Institute of Trading Standards Administration
- Ministry of Agriculture, Fisheries and Food
- Natural Resources Institute

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Foreword

This Part of BS 5752 has been prepared by Technical Committee AW/15. It is technically equivalent to ISO 11292 : 1995 *Instant coffee — Determination of free and total carbohydrate contents — Method using high-performance anion-exchange chromatography*, published by the International Organization for Standardization (ISO) and in the preparation of which the United Kingdom played a full part. This Part of BS 5752 differs from ISO 11292 principally in the presentation of the precision data in clause 10 and table 3. The methodology is identical. ISO is considering amending ISO 11292 : 1995 to align it with this Part of BS 5752. This standard embodies an agreement, to which the UK was a party, reached in Subcommittee 15, Coffee, of Technical Committee 34, Agricultural food products, of ISO.

Cross-references

International standard Normative	Corresponding British Standard
ISO 1042 : 1983	BS 1792 : 1982 <i>Specification for one-mark volumetric flasks</i> (Identical)
ISO 3509 : 1989	BS 5456 : 1989 <i>Glossary of terms relating to coffee and its products</i> (Identical)
ISO 3726 : 1983	BS 5752 <i>Methods of test for coffee and coffee products</i> Part 6 : 1983 <i>Instant coffee: determination of loss in mass at 70° C under reduced pressure</i> (Identical)

Informative

ISO 5725 : 1986, to which informative reference is made in the text, has been superseded by ISO 5725-1 : 1994, ISO 5725-2 : 1994, ISO 5725-3 : 1994, ISO 5725-4 : 1994 and ISO 5725-6 : 1994 which are identical with BS ISO 5725 *Accuracy (trueness and precision) of measurement methods and results*, BS ISO 5725-1 : 1994 *General principles and definitions*, BS ISO 5725-2 : 1994 *Basic method for the determination of repeatability and reproducibility of a standard measurement method*, BS ISO 5725-3 : 1994 *Intermediate measures of the precision of a standard measurement method*, BS ISO 5725-4 : 1994 *Basic method for the determination of the trueness of a standard measurement method*, and BS ISO 5725-6 : 1994 *Use in practice of accuracy values*, respectively.

NOTE. For ease of reproduction the amended text of the International Standard has been used.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Instant coffee — Determination of free and total carbohydrate contents — Method using high-performance anion-exchange chromatography

1 Scope

This International Standard specifies a method for the determination of free and total carbohydrate contents in instant coffee using high-performance anion-exchange chromatography. In particular, it determines the content of individual monosaccharides, sucrose and mannitol.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3509:1989, *Coffee and its products — Vocabulary*.

ISO 3726:1983, *Instant coffee — Determination of loss in mass at 70 °C under reduced pressure*.

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 3509 and the following definitions apply.

3.1 free carbohydrate content: Content of each individual monosaccharide (arabinose, fructose, galactose, glucose, mannose), and the sucrose and mannitol contents, determined under the conditions described (method A). Content is expressed as a percentage by mass on a dry basis.

3.2 total carbohydrate content: Content of each individual monosaccharide (arabinose, galactose, glucose, mannose, xylose) and the mannitol content, determined under the conditions described, which includes a strong hydrolysis step (method B). Content is expressed as a percentage by mass on a dry basis.

4 Principle

4.1 Method A

Dissolution of a test portion in water. Separation of the carbohydrates present in the filtered extract by ion chromatography on a high-performance anion-exchange column (HPAEC) using pure water as eluent. Electrochemical detection of the eluted compounds by means of a pulsed amperometric detector (PAD) and quantification by comparison with peak areas given by standard solutions.

4.2 Method B

Hydrolysis of a test portion with aqueous hydrochloric acid. Analysis of the carbohydrates present in the filtered hydrolysed solution as described in method A.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

5.1 Sodium hydroxide (NaOH), 50 % (m/m) aqueous solution.

The reagent should contain the minimum amount of sodium carbonate and mercury. Do not shake or stir the solution before use.

5.2 Hydrochloric acid (HCl), 1,00 mol/l standard volumetric solution.

5.3 Eluent 1 (S1), demineralized water (18 MΩ·cm).

Filter the demineralized water through 0,2 µm membrane filters. Degas by sparging with helium for between 20 min and 30 min.

5.4 Eluent 2 (S2), sodium hydroxide (NaOH), 300 mmol/l solution.

To 985 ml of degassed water (5.3), pipette 15,6 ml of the sodium hydroxide solution (5.1).

CAUTION — It is extremely important to remove dissolved carbon dioxide from the eluents prior to use. Carbonate will act as a strong “pusher” on the column, resulting in a drastic reduction in resolution and efficiency. Prepare the solution the day before the analysis.

5.5 Carbohydrate standard solutions.

Prepare fresh solutions of arabinose, fructose, galactose, glucose, mannose, sucrose and mannitol.

1) Sep-Pack C18 (Waters) and Supelclean LC-18 (Supelco) are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

* See national foreword for details of textual error.

Weigh, to the nearest 0,1 mg, approximately 100 mg of each carbohydrate into separate 100 ml volumetric flasks (6.2) and dilute to the mark with water (stock standard solutions of 1 000 mg/l).

Mixed standard solutions can also be prepared from separate stock solutions once the retention time of each carbohydrate is known under the prevailing chromatographic conditions.

Further dilute the standard solutions to reach carbohydrate concentrations similar to those found in the non-hydrolysed or hydrolysed instant coffee sample solutions.

The resolution of rhamnose from arabinose is difficult to achieve. If these two monosaccharides coelute, do not add rhamnose in a mixed standard solution.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Analytical balance, capable of weighing to an accuracy of ± 0,1 mg. *

6.2 One-mark volumetric flasks, of capacity 100 ml (in accordance with class A of ISO 1042).

6.3 Graduated cylinders, of capacities 1 000 ml and 50 ml, tall form.

6.4 Vacuum filtration system.

6.5 Folded filter papers, medium fast, qualitative.

6.6 Disposable C18 filter cartridges¹⁾, to be used according to the manufacturer's recommendations.

6.7 Disposable membrane filters, 0,2 µm pore size.

6.8 Water bath, capable of being maintained at 100 °C ± 5 °C.