International Standard 1742

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

Glucose syrups — Determination of dry matter — Vacuum oven method

Sirops de glucose — Détermination de la matière sèche — Méthode par étuvage sous pression réduite

First edition — 1980-08-01

UDC 664.162 : 543.81 **Ref. No. ISO 1742-1980 (E)**

Descriptors : carbohydrates, glucose, tests, determination of content, dry matter, dehydration analysis, stoves (chemistry), low pressure tests.

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.

International Standard ISO 1742 was developed by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, and was circulated to the member bodies in May 1979.

It has been approved by the member bodies of the following countries :

Australia	Libyan Arab Jamahiriya	Spain
Chile	Netherlands	United Kingdom
Germany, F. R.	Poland	USA
Israel	Romania	USSR
Kenya	South Africa, Rep. of	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

France

This International Standard cancels and replaces ISO Recommendation R 1742-1970, of which it constitutes a technical revision.

Printed in Switzerland

Glucose syrups — Determination of dry matter — Vacuum oven method

1 Scope and field of application

This International Standard specifies a vacuum oven method for the determination of the dry matter in glucose syrups, irrespective of their method of production.

The method is also applicable to dried glucose syrup, solid glucose (starch sugar), glucose syrup containing fructose (including *iso*glucose as defined by the European Community[1]).

2 Principle

Drying of a test portion, diluted with water and mixed with kieselguhr to provide a large surface for drying, in a vacuum oven at 70 °C, at a pressure not exceeding 34 mbar*.

3 Reagent

The reagent shall be of recognized analytical quality. Distilled water or water of at least equivalent purity shall be used.

3.1 Diatomaceous earth filter aid (Kieselguhr), prepared as follows.

Wash a large quantity of kieselguhr several times with water acidified with hydrochloric acid [1 ml of concentrated acid (ϱ_{20} = 1,19 g/ml) per litre of water] by filtration on a Büchner funnel until the washings turn litmus paper red. Repeat the washing, but with water, until the pH value of the washings is equal to or slightly more than 4. Allow the washed kieselguhr to dry in air. Before use, dry it overnight in an oven at 105 °C at atmospheric pressure and store it in a closed container.

4 Apparatus

4.1 Analytical balance

4.2 Beaker, of capacity 100 ml.

4.3 Dish, of metal (inert under the test conditions) or of glass, 75 mm deep and 90 mm in diameter, provided with a closely fitting lid.

4.4 Glass stirring rod, of length appropriate to the diameter of the dish.

4.5 Electrically heated vacuum oven, capable of being maintained at 70 ± 1 °C, equipped with a calibrated thermometer and an absolute pressure gauge.

The drying oven shall provide uniform heat distribution and shall maintain the reduced pressure for several hours after the vacuum pump is turned off. The oven shelves shall be so constructed and fitted as to ensure good heat transfer to the dishes.

4.6 Vacuum pump, suitable for reducing the pressure in the oven to 34 mbar or less.

4.7 Drying train, consisting of a drying column filled with dried silica gel, the column is connected in series to a gas scrubber containing concentrated sulphuric acid, which is in turn connected to the air inlet of the drying oven.

4.8 Desiccator, containing an efficient desiccant.

5 Procedure

5.1 Preparation of the test sample

Mix the laboratory sample well.

5.2 Preparation of the dish

Weigh about 30 g of the dried kieselguhr (3.1) into the dish (4.3). Dry the open dish, together with its lid and the stirring rod (4.4), in the oven (4.5) for 5 h at 70 ± 1 °C at a pressure not exceeding 34 mbar. At the end of this period, restore atmospheric pressure in the oven by allowing air to slowly enter through the drying train (4.7). Before removing the dish from the oven, fit the lid and place the stirring rod on it. Place the covered weighing dish and the stirring rod together in the desiccator (4.8), allow to cool for 1 h, and weigh to the nearest 0,001 g.

* 1 mbar = 0,1 kPa

1) JOCE of 28.05.1977, Regulation 1111/77.

5.3 Test portion

Weigh, to the nearest 0,001 g, 8 to 10 g of the test sample (5.1) into the beaker (4.2).

5.4 Determination

To the test portion (5.3) in the beaker add about 10 ml of warm water, constantly stirring with the glass rod (4.4). Transfer quantitatively the diluted test portion to the dish (4.3) containing the kieselguhr, using three 5 ml portions of warm water. Stir until a homogeneous mixture is obtained. Place the open dish with the stirring rod and its cover in the oven (4.5) and heat for 5 h at 70 ± 1 °C, at a pressure not exceeding 34 mbar*. While the samples are drying, draw a slow stream of air into the oven through the drying train (4.7).

After 5 h, shut off the vacuum pump (4.6) and allow air to slowly enter the oven through the drying train, until atmospheric pressure is restored. Remove the dish from the oven and finely crush the kieselguhr with the stirring rod (4.4). Place the rod wholly within the dish, return the dish to the oven and heat for a further 10 h at 70 ± 1 °C at a pressure not exceeding 34 mbar. Again shut off the vacuum pump and restore atmospheric pressure in the oven as before.

Before withdrawing the dish from the oven, fit the cover. Place the covered dish in the desiccator (4.8), allow to cool for 1 h, and weigh to the nearest 0,001 g. Again heat the covered dish in the oven for 5 h at 70 ± 1 °C at a pressure not exceeding 34 mbar, cool in the desiccator and weigh to confirm that constant mass has been reached (see clause 7).

Carry out two determinations on the same test sample (5.1).

6 Expression of results

The dry matter content, expressed as a percentage by mass of the product as received, is equal to

$$(m_2 - m_1) \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (5.3);

m_1 is the mass, in grams, of the dish, cover, stirring rod and kieselguhr (see 5.2);

m_2 is the mass, in grams, of the dish, cover, stirring rod, kieselguhr and residue of the test portion after drying (see 5.4).

7 Note on procedure

The accuracy of the determination largely depends on the thoroughness with which the test portion is mixed with the kieselguhr. Consequently, sufficient time should be spent on this.

Constant mass is attained, for practical purposes, when the loss in mass after the second 5 h drying period is not more than 0,02 % of the mass of the test portion.

If constant mass is not reached after a total drying time of 20 h, this should be attributed to inadequate mixing of the test portion with the kieselguhr, and the test should be repeated.

8 Test report

The test report shall indicate the method used and the result obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for complete identification of the sample.

---

* 34 mbar = 3,4 kPa