

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1742

GLUCOSE SYRUP

DETERMINATION OF DRY MATTER OR MOISTURE CONTENT
(VACUUM OVEN METHOD)

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1742, *Glucose syrup -- Determination of dry matter or moisture content (Vacuum oven method)*, was drawn up by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, the Secretariat of which is held by the Deutscher Normenausschuss (DNA).

Work on this question led to the adoption of Draft ISO Recommendation No. 1742 which was circulated to all the ISO Member Bodies for enquiry in November 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Iran	Romania
Brazil	Ireland	South Africa, Rep. of
Chile	Israel	Spain
Colombia	Korea, Rep. of	Thailand
Germany	Netherlands	Turkey
Greece	New Zealand	U.A.R.
Hungary	Peru	United Kingdom
India	Poland	U.S.A.

The following Member Body opposed the approval of the Draft :

France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

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GLUCOSE SYRUP

DETERMINATION OF DRY MATTER OR MOISTURE CONTENT

(VACUUM OVEN METHOD)

1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes a vacuum oven method for the determination of dry matter (or moisture content) in glucose syrup obtained by acid hydrolysis.

The method is also applicable to dried glucose syrup and solid glucose (starch sugar).

2. PRINCIPLE

Drying of the test portion, diluted with water and mixed with kieselguhr (to provide a large surface for drying) in a vacuum drying oven at 100 °C, at a pressure not exceeding 34 mbar*.

3. REAGENTS

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

Kieselguhr. Repeatedly wash a large quantity of kieselguhr with water containing hydrochloric acid (1 ml of concentrated acid ($\rho_{20} = 1.19$ g/ml) per litre of water) by percolation on a Büchner funnel. Wash until the effluent gives a red colour to litmus paper. Repeat the washing but with water until the pH value of the effluent is 4 or above. Allow the washed kieselguhr to dry in air. Before using it, dry it overnight in an air oven at 105 °C, at atmospheric pressure, and keep it in a closed container.

4. APPARATUS

- 4.1 *Beaker*, 100 ml.
- 4.2 *Weighing dish*, of metal (unaffected by glucose syrup under the test conditions) or glass (for example 75 mm deep and 90 mm in diameter), with a closely fitting cover.
- 4.3 *Glass stirring rod*, of length appropriate to the dish (for example about 55 mm).
- 4.4 *Electrically heated vacuum drying oven*, with accurate automatic temperature control between 99 and 101 °C, equipped with calibrated thermometer and vacuum gauge. The drying oven should provide uniform heat distribution and hold the reduced pressure for several hours after the vacuum pump is turned off. The oven shelves should be so constructed and fitted as to ensure good heat transfer to the weighing dishes.
- 4.5 *Vacuum pump*, suitable for reducing the pressure in the oven to 34 mbar or less.

* About 25 mmHg.

- 4.6 *Drying train*, consisting of a drying tower filled with dried silica gel; the tower is connected in series to a gas scrubber (containing concentrated sulphuric acid), which is connected with the air inlet of the drying oven.
- 4.7 *Desiccator*, containing an effective drying agent, for example concentrated sulphuric acid or freshly activated silica gel with moisture indicator.
- 4.8 *Analytical balance*.

5. PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.001 g, 8 to 10 g of the sample into the dry beaker (4.1).

5.2 Determination

Weigh about 30 g of the dried kieselguhr into the weighing dish (4.2). Dry the open dish, together with its cover and the stirring rod (4.3), for 5 hours in the vacuum oven (4.4) at a pressure not exceeding 34 mbar. At the end of the 5 hours' drying, restore atmospheric pressure in the oven by allowing air to enter slowly through the drying train (4.6). Before removing the dish from the oven, fit the cover and place the stirring rod on it. Place the covered weighing dish and the stirring rod together in the desiccator (4.7), allow to cool undisturbed for 1 hour, and weigh.

To the test portion in the beaker add about 10 ml of warm water, constantly stirring with the glass rod (4.3). Transfer the diluted test portion quantitatively to the weighing dish (4.2) containing the kieselguhr, using three 5 ml portions of warm water. Stir until a homogeneous mixture is obtained. Place the open weighing dish with the stirring rod and its cover in the vacuum oven (4.4) and heat for 5 hours at 100 °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 (4.6).

After 5 hours, shut off the vacuum pump (4.5) and allow air to enter the oven slowly through the drying train, until atmospheric pressure is reached. Remove the weighing dish from the oven and finely crush the kieselguhr with the stirring rod (4.3). Place the rod wholly within the dish and return the weighing dish to the oven and heat for a further 10 hours at 100 °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 weighing dish from the oven, fit the cover. Place the covered weighing dish in the desiccator (4.7), allow to cool for 1 hour, and weigh. Heat again in the vacuum oven for 5 hours, cool in the desiccator and weigh to confirm that constant mass has been reached (see section 7).

Carry out two determinations on the same prepared sample.

6. EXPRESSION OF RESULTS

6.1 Dry matter content

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 grammes, of the test portion;

m_1 is the mass, in grammes, of the weighing dish, cover, stirring rod and kieselguhr;

m_2 is the mass, in grammes, of the weighing dish, cover, stirring rod, kieselguhr and residue of the test portion after drying.