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FRUIT AND VEGETABLE PRODUCTS
DETERMINATION OF TOTAL SOLIDS

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BRIEF HISTORY

The ISO Recommendation R 1026, *Fruit and vegetable products – Determination of total solids,* was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question led, in 1966, to the adoption of a Draft ISO Recommendation.

In April 1967, this Draft ISO Recommendation (No. 1212) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina
Australia
Brazil
Bulgaria
Colombia
Czechoslovakia
France
Greece
Hungary
India
Iran
Ireland
Israel
Korea, Rep. of
New Zealand
Norway
Poland
Portugal
Romania
South Africa, Rep. of
Thailand
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

One Member Body opposed the approval of the Draft :

Netherlands

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in March 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1026 March 1969

FRUIT AND VEGETABLE PRODUCTS

DETERMINATION OF TOTAL SOLIDS

1. SCOPE

1.1 This ISO Recommendation describes two methods for the determination of total solids in products derived from fruits and vegetables :

the drying method;

the water-recovery method.

1.2 Field of application

The drying method, which consists in weighing the product before and after drying under defined conditions, is applicable to all fruit and vegetable products, with the exception of products for which drying would be too protracted, or would modify the state of the components, and of certain products having a water content below 10 %.

In these last-mentioned cases the water-recovery method, which consists in measuring volumetrically the water recovered, should be used; entrainment by a volatile solvent affords a means of avoiding the inclusion of matter soluble in the solvent, in the determination, as would occur in a classical extraction by washing. This method, however, is delicate and calls for great care; it can be used within the limits of applicability mentioned above, but certain restrictions should be mentioned, for example when the product is rich in fructose or in certain volatile substances.

2. DRYING METHOD

2.1 Definition

By *total solids* is meant the whole of the substances which do not volatilize under the physical conditions of drying defined in this ISO Recommendation. These physical conditions have been chosen so that the substances constituting the total solids will undergo as little change as possible.

2.2 Principle

Heating to constant mass, after thorough mixing, of liquid products spread over an absorbent surface, or of pasty products mixed with an inert powder, at a moderate temperature under reduced pressure.

The products thus lose not only their "free" water, but their volatile matter. If the result obtained is indeed the total solids, the loss in mass is greater than the initial water content, especially in fermentation products.

2.3 Apparatus

2.3.1 *Oven,* allowing drying to be carried out at 70 °C under a pressure of 26 to 33 mbar (20 to 25 mmHg), while a slow current of dry air (see clause 2.7.3) enters at the rate of about 40 litres per hour, measured at atmospheric pressure before entry into the oven; for example, air introduced by means of a tap is dried by passing it, one bubble at a time, into a wash bottle containing sulphuric acid in front of the tap, ensuring expansion of the air. The temperature should be uniform at all points in the oven.

2.3.2 *Desiccator,* provided with an efficient desiccant.

2.3.3 *Analytical balance.*

2.3.4 *Dishes,* of corrosion-resistant metal (aluminium, nickel, or, preferably, thin stainless steel), cylindrical, with flat bottom (for example, diameter about 60 mm; height 25 mm), provided with airtight but easily removable lids.

2.3.5 *Glass rod,* of length suitable for the size of the dish.

2.4 Support materials

2.4.1 *Paper*

2.4.1.1 PAPER IN STRIPS, FOR LIQUID PRODUCTS. Use ashless filter paper. Filter paper previously washed in hydrochloric acid (2 g of HCl per litre) for 8 hours, rinsed five times with distilled water and dried in air, may also be used. This paper is cut into strips 20 mm wide. It may be crimped or folded in accordion fashion with close pleats, or more simply by winding round a triangular mandrel with 1 cm sides. This strip partially uncoils itself, giving a polygonal spiral. From 4 to 4.5 g of paper should be put in each dish, either 3 m of paper strip if the paper weighs 60 to 70 g/m^2, or 1 m if a thick paper weighing 180 to 200 g/m^2 is used.

2.4.1.2 PAPER IN DISKS, FOR SEMI-LIQUID PRODUCTS. Use disks of strong ashless filter paper, crimped, cut to a diameter slightly less than that of the dishes. If ashless filter paper is not available, the filter paper used should be treated in the same way as described above.

2.4.2 *Sand,* pure, washed with 5 % hydrochloric acid and rinsed until free from hydrochloric acid, sieved so that the grains are of sizes within the range 100 to 400 μm, and calcined.

2.5 Procedure

2.5.1 *Preparation of sample*

2.5.1.1 LIQUID OR SEMI-LIQUID PRODUCTS (juices, concentrates, thick juices, etc.). Mix the sample until it is uniform.

2.5.1.2 THICK, PULPY OR HETEROGENEOUS PRODUCTS (fruits in syrup, jams, vegetable conserves, etc.). After mixing the laboratory sample*, draw from it a quantity sufficient for at least two determinations and homogenize by means of a blender.

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* Pending the completion of an ISO Recommendation on the sampling of fruit and vegetable products, the term "laboratory sample" is used in the English text to denote the sample as delivered to the laboratory.

2.5.2 *Test portion*

2.5.2.1 LIQUID AND SEMI-LIQUID PRODUCTS. Dry in the oven (2.3.1) at 70 °C, under the conditions described in clause 2.3.1, a metal dish (2.3.4) in which have been placed the paper strip or two disks of crimped paper, as the case may be (see clause 2.4.1), the lid being removed. After drying for 1 hour in the oven, weigh the dish to the nearest 0.0002 g after cooling in the desiccator (2.3.2), the lid being placed on the dish before the latter is taken out of the oven.

By means of a pipette, withdraw from the sample, prepared as described in clause 2.5.1, 10 ml (in the case of liquid products) or a few millilitres (in the case of semi-liquid products), and soak completely the paper strips or disks, as the case may be (see clause 2.4.1) in the dish (2.3.4), avoiding the accumulation of an excess of liquid against the metal. Carry out the operations as quickly as possible in order to avoid the action of atmospheric moisture. Close the dish and weigh to the nearest 0.0002 g. For semi-liquid products, the soaking can be facilitated by slightly moistening the paper disks with distilled water after weighing the product added.

2.5.2.2 THICK, PULPY OR HETEROGENEOUS PRODUCTS. Dry in the oven (2.3.1) at 70 °C, under the conditions described in clause 2.3.1, a metal dish (2.3.4) in which have been put 10 to 20 g of sand (2.4.2) and the glass rod (2.3.5). After drying for 1 hour in the oven, weigh the dish to the nearest 0.0002 g after cooling in the desiccator (2.3.2), the lid being placed on the dish before the latter is taken out of the oven.

Transfer to the dish 2 to 5 g of the sample, prepared as described in clause 2.5.1, and weigh. Mix it intimately with the sand by means of the rod, taking great care to avoid any loss of product or sand to the outside of the dish. If mixing is difficult, a little distilled water may be added, provided that this is done after weighing the product in the dish.

2.5.3 *Determination*

2.5.3.1 LIQUID AND SEMI-LIQUID PRODUCTS. Put the dish containing the paper and the test portion (2.5.2.1) into the oven (2.3.1) at 70 °C, taking off the lid. Heat under reduced pressure while passing a current of dry air, under the conditions described in clause 2.3.1 (see clause 2.7.2).

Dry for 3 hours and continue the drying until two weighings carried out at 1 hour intervals do not differ by more than 0.001 g. Carry out the weighings to the nearest 0.0002 g, after cooling in the desiccator, putting the lid on the dish before the latter is taken out of the oven.

2.5.3.2 THICK, PULPY OR HETEROGENEOUS PRODUCTS. Put the dish containing the sand, the rod and the test portion, with the lid removed, in the oven (2.3.1) regulated at 70 °C and provided with its means of sweeping with dry air. Continue as for liquid products (see clause 2.5.3.1) except that the 3 hour period of drying is increased to 4 hours (see clause 2.7.2).

2.5.3.3 Carry out at least two determinations on the same prepared sample.

2.6 Expression of results

2.6.1 *Method of calculation and formula.* The percentage, by mass, of total solids, is equal to

$$(M_2 - M_0) \times \frac{100}{M_1 - M_0}$$

where

M_0 is the mass, in grammes, of the dish and accessories (paper or sand and rod, lid);

M_1 is the mass, in grammes, of the same dish with the moist product, before drying;

M_2 is the mass, in grammes, of the same dish with the dry residue.

Take as the result the arithmetic mean of the two determinations, if the appropriate requirement of clause 2.6.2 is satisfied. Give the result to one decimal place.

NOTE. – In the case of products containing a small amount of water, the result may be expressed as a percentage, by mass, of water.

2.6.2 *Repeatability.* The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not be greater than

1 % of the mean value for total solids contents greater than 10 g per 100 g of sample;

2 % of the mean value for total solids contents less than or equal to 10 g per 100 g of sample.

2.7 Notes on procedure

2.7.1 The apparatus and technique, when a filter-paper support is used, can be checked by carrying out the following two simple tests :

(*a*) *Check on the airtightness of the dishes.* A dish containing paper which has been dried, when closed by its lid and left exposed to the air of the laboratory after cooling in the desiccator over sulphuric acid, should not increase in mass by more than 0.001 g per hour.

(*b*) *Check on degree of drying.* A solution of pure sucrose (100 g per litre) should leave a residue of 100 ± 1 g per litre.

A solution of pure lactic acid (10 g per litre) should leave at least 9.5 g of total solids per litre.

NOTE.– The solution of lactic acid should be prepared as follows :
Dilute 10 ml of pure lactic acid with about 100 ml of water. Heat this solution, in a dish, on a boiling water bath for 4 hours, adding distilled water if the volume falls below about 50 ml. Dilute the contents of the dish to 1 litre and titrate the lactic acid in 10 ml of this solution with a 0.1 N solution of alkali. Adjust the concentration to 10 g per litre.

Increase or reduce the time in the oven, the rate of dry air flow, or the internal pressure to satisfy these conditions.

2.7.2 In certain cases, it is recommended that for the first hour the drying should be carried out at 70 °C and 132 mbar (100 mmHg) and the pressure should then be reduced to 33 mbar (25 mmHg).

2.7.3 If there is a risk of oxidation when air is used, use an inert gas for sweeping through the apparatus.

3. WATER-RECOVERY METHOD

3.1 Definition

By *water* is meant the aqueous distillate entrained and collected by the method described.

The water content is expressed as a percentage, by mass.

3.2 Principle

Entrainment of the water in the form of vapour by a volatile solvent immiscible with water, condensation and separation in a reflux trap, recovery and measurement of the volume of water in a graduated tube.

3.3 Reagent

Benzene

3.4 Apparatus

3.4.1 *Distillation apparatus,* comprising the following items connected together by means of ground joints :

3.4.1.1 *Conical flask,* with short neck, of at least 500 ml capacity.

3.4.1.2 *Reflux condenser.*

3.4.1.3 *Receiver,* as shown in the Figure, or equivalent apparatus, interposed between the flask and the condenser.

NOTE. – In order to eliminate all traces of grease from the graduated tube and the interior of the condenser, clean the apparatus, for example with a chromic-sulphuric acid mixture, and wash it successively with distilled water and acetone. Then dry the apparatus in a current of air, without heating.

3.4.2 *Electric heating apparatus,* with control system, or *water bath.*

3.4.3 *Balance.*

3.5 Procedure

3.5.1 *Preparation of sample.* Make the sample uniform by means of a mill or mortar, producing a fine paste. Add, if necessary, a known amount of water, provided that this is taken into account in the calculation.

For products which are too thick (for example, dried banana) add an equal mass of sand and take this into account in calculating the result.

3.5.2 *Test portion.* Weigh out, to the nearest 0.1 g, 50 to 100 g of the sample prepared as described in clause 3.5.1.

3.5.3 *Determination.* Transfer the test portion to the conical flask (3.4.1.1) with an amount of benzene approximately the same as the mass of the test portion used. For thick products add a boiling regulator, e.g. a few pieces of pumice.

Fit the receiver (3.4.1.3) to the flask and condenser, then connect the tubes supplying the condenser.

Cautiously heat the flask on the electric heating apparatus or the water bath (3.4.2) (see clause 3.7.2), and maintain gentle boiling until the solvent distilling over becomes clear and no more water separates (about 3 hours). The distillate should fall drop by drop from the bottom of the condenser, at the rate of about 2 drops per second.

The water collects in the graduated tube.

At the end of the operation, cease heating, then shake the condenser so as to dislodge drops of water and benzene remaining attached to the wall.

Read the volume of water collected in the graduated tube, after allowing sufficient time for the water to coalesce completely so that there is no emulsified zone.

Carry out two determinations on the same prepared sample.

3.6 Expression of results

The total solids, by definition, are expressed as the difference

$$100 - P$$

where P is the water content of the product, expressed in grammes per 100 g of the material used.

Take account of dilutions and admixtures made during the preparation of the sample.

Take as the result the arithmetic mean of the two determinations.

3.7 Notes on procedure

3.7.1 If the graduation of the lower part of the graduated tube is in doubt, one can, before transferring the test portion into the conical flask, pour benzene into the flask, add a small quantity of water, depending on the volume of the lower part of the tube, and distil until the volume of water in the graduated tube is constant. The meniscus should then always be in the accurate part of the receiver. The determination can then be carried out.

3.7.2 In the case of pasty products, it is recommended that the water bath should be used for heating the conical flask.

4. TEST REPORT

The test report should show the method used and the result obtained. It should also mention all operating details not specified in this ISO Recommendation, or regarded as optional, as well as any circumstances that may have influenced the results.

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