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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 763

FRUIT AND VEGETABLE PRODUCTS

DETERMINATION OF ASH INSOLUBLE

IN HYDROCHLORIC ACID

1st EDITION June 1968

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BRIEF HISTORY

The ISO Recommendation R 763, Fruit and vegetable products – Determination of ash insoluble in hydrochloric acid, 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 by the Technical Committee began in 1960 and led, in 1964, to the adoption of a Draft ISO Recommendation.

In October 1966, this Draft ISO Recommendation (No. 1023) 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 :

Australia Brazil Bulgaria Chile Colombia Czechoslovakia France Germany Hungary India Iran Ireland Israel Korea, Rep. of Poland Portugal Romania South Africa, Rep. of Thailand Turkey 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 June 1968, to accept it as an ISO RECOMMENDATION.

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FRUIT AND VEGETABLE PRODUCTS

DETERMINATION OF ASH INSOLUBLE

IN HYDROCHLORIC ACID

1. SCOPE

1.1 This ISO Recommendation describes the procedure for the determination of ash insoluble in hydrochloric acid, in fruit and vegetable products.

1.2 Field of application

This method serves for the determination of siliceous impurities, together with silica endogenous to the plant.

The determination of heavy impurities, generally originating from the soil, is carried out by the method described in ISO Recommendation R 762, Fruit and vegetable products – Determination of mineral impurities.

2. PRINCIPLE

Incineration of the product at 500 to 600 $^{\circ}$ C and separation of the mineral matter insoluble in a 10 $^{\circ}$ / $_{\circ}$ solution of hydrochloric acid.

3. REAGENT

Hydrochloric acid solution. Dilute 1 volume of hydrochloric acid ($\rho_{20} = 1.19 \text{ g/ml}$) with 9 volumes of water.

4. APPARATUS

- 4.1 Muffle furnace, regulated at 500 to 600 °C.
- 4.2 Analytical balance.
- 4.3 Water bath.
- 4.4 Drying oven.
- 4.5 Desiccator, containing an efficient desiccant.
- 4.6 Dish, of quartz or platinum.
- 4.7 Filter paper, ashless.

5. PROCEDURE

5.1 Preparation of sample

Before drawing the test portion, thoroughly mix the laboratory sample*, using a blender if necessary. Frozen products should first be thawed.

5.2 Test portion

Heat the empty dish (4.6), allow it to cool in the desiccator (4.5) and weigh it to the nearest 0.0002 g.

Weigh into the previously tared dish, to the nearest 0.01 g, 4 to 25 g of the prepared laboratory sample (see clause 5.1) according to the water content of the product.

5.3 Determination

Evaporate the water present in the product by heating on the water bath (4.3) and dry in the oven (4.4) together with the dish (4.6). This drying is not necessary for dried products.

After this evaporation, if required, calcine and then incinerate the product in the muffle furnace (4.1) at 500 to 600 $^{\circ}$ C.

Allow to cool in the desiccator (4.5). After cooling, add 10 to 25 ml of hydrochloric acid solution (3), cover with a watch-glass and heat on the boiling-water bath (4.3) during 15 minutes.

Transfer the residue to the ashless filter paper (4.7) in a funnel. Rinse the dish with hot distilled water and transfer the contents of the dish to the filter paper. Repeat this operation until there is no trace of chloride ions in the liquid flowing from the funnel (test with silver nitrate solution).

Replace the filter paper and residue in the dish and incinerate for 30 minutes in the muffle furnace at 500 to 600 $^{\circ}$ C.

Cool again in the desiccator and weigh to the nearest 0.0002 g.

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

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.

6. EXPRESSION OF RESULTS

6.1 Method of calculation and formula

The ash insoluble in hydrochloric acid, expressed as a percentage by mass of the product, 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 dish,

 M_2 is the mass, in grammes, of the dish and ash.

Take as the result the arithmetic mean of the two determinations, if the requirement concerning repeatability is satisfied.

Report the result to two decimal places.

6.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0.01 g of ash insoluble in hydrochloric acid, per 100 g of sample.

7. TEST REPORT

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

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