
**Fruit and vegetable products —
Determination of ash insoluble in
hydrochloric acid**

*Produits dérivés des fruits et légumes — Détermination des cendres
insolubles dans l'acide chlorhydrique*

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 763 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 3, *Fruit and vegetable products*.

This second edition cancels and replaces the first edition (ISO 763:1982), which has been technically revised.

Fruit and vegetable products — Determination of ash insoluble in hydrochloric acid

1 Scope

This International Standard specifies a method for the determination of the hydrochloric acid-insoluble ash yielded by fruit and vegetable products.

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

NOTE A method for the determination of mineral impurities generally originating from the soil is specified in ISO 762¹⁾.

2 Principle

A test portion is incinerated at approximately 525 °C and the mineral matter insoluble in dilute hydrochloric acid is separated.

3 Reagents

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

3.1 Hydrochloric acid, 10 % (by mass) solution.

3.2 Silver nitrate, approximately 17 g/l solution.

4 Apparatus

Usual laboratory apparatus and, in particular, the following.

4.1 Blender.

4.2 Muffle furnace, capable of being maintained at 525 °C ± 25 °C.

4.3 Boiling water bath.

4.4 Drying oven, capable of being maintained at 103 °C ± 2 °C.

4.5 Desiccator, containing an efficient desiccant.

4.6 Incineration dishes, made of quartz or platinum.

1) ISO 762, *Fruit and vegetable products — Determination of mineral impurities content*.

4.7 Filter paper, ashless.

4.8 Analytical balance, capable of weighing to the nearest 0,000 2 g.

5 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

6 Preparation of test sample

Allow frozen or deep-frozen products to thaw in a closed vessel and add the liquid formed during this process to the product before mixing. Before taking the test portion, thoroughly mix the laboratory sample using, if necessary, the blender (4.1).

7 Procedure

7.1 Preparation of first dish

Heat an empty dish (4.6) in the furnace (4.2) set at the incineration temperature. Allow it to cool in the desiccator (4.5) then weigh to the nearest 0,000 2 g. Repeat until a constant mass is achieved.

7.2 Test portion

Weigh, to the nearest 0,01 g, in the previously prepared dish (7.1), 4 g to 25 g of the test sample (Clause 6) according to the water content of the product. For liquid products, the test portion may be taken by volume (see 8.2).

7.3 Determination

7.3.1 Drying

Place the dish and its contents in the boiling water bath (4.3) and evaporate the water present in the product. Dry in the oven (4.4) set at 103 °C. This drying is not necessary for dry products.

7.3.2 Incineration

After drying (if appropriate), carbonize and then completely incinerate the product in the furnace (4.2) set at 525 °C; the ash may still be grey after incineration.

Pre-incineration at a distinctly lower temperature before placing in the furnace is sometimes necessary for products with a high sugar content in order to avoid foaming and subsequent loss of foam. For that purpose, slowly heat the dried sample on the hot plate until carbonization of the most of the organic matter.

It is recommended to decrease the salt content of products containing more than 2 % of sodium chloride by the following method. Pre-incinerate the sample then wash the carbonaceous residue several times with small amounts of hot distilled water.

7.3.3 Treatment with hydrochloric acid

Allow the sample to cool in the desiccator (4.5). After cooling, add 10 ml to 25 ml of the hydrochloric acid solution (3.1). Cover with a watch-glass and heat in the boiling water bath (4.3) for 15 min ± 2 min.

Transfer the residue to the ashless filter paper (4.7) placed in a funnel. Rinse the dish with hot water and transfer the contents of the dish to the filter paper. Wash the filter paper and its contents until there is no trace of chloride ions in the liquid flowing from the funnel [test with the silver nitrate solution (3.2)].

7.3.4 Preparation of the second dish

Prepare a new dish (4.6) as specified in 7.1 or clean the first dish. Heat it in the muffle furnace (4.2) to the incineration temperature. Allow it to cool in the desiccator then weigh to the nearest 0,000 2 g. Repeat until a constant mass is achieved.

7.3.5 Drying and incineration

Place the filter paper and residue in the dish. Dry in the oven (4.4) set at 103 °C, then incinerate in the muffle furnace (4.2), set at 525 °C, for 30 min ± 2 min.

Cool in the desiccator (4.5), then weigh to the nearest 0,000 2 g. Repeat until a constant mass is achieved.

8 Expression of results

8.1 Method of calculation

The ash insoluble in hydrochloric acid, w , expressed as a percentage by mass, is given by the formula

$$w = \frac{m_2 - m_3}{m_0 - m_1} \times 100 \%$$

where

m_0 is the mass, in grams, of the dish and test portion (7.2);

m_1 is the mass, in grams, of the empty dish (7.1);

m_2 is the mass, in grams, of the dish and acid-insoluble ash (7.3.5);

m_3 is the mass, in grams, of the second empty dish (7.3.4).

Report the results to two decimal places.

8.2 Other method of expression

For liquid products, it is possible to express the result in grams per 100 ml of product, by taking the test portion (7.2) by volume and by replacing the denominator ($m_0 - m_1$) in the equation (see 8.1) by V , the volume of the test portion.

9 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,01 g of ash insoluble in hydrochloric acid per 100 g of sample.

10 Test report

The test report shall specify:

- a) all information necessary for the complete identification of the sample;
- b) the sampling method used, if known;
- c) the test method used, with reference to this International Standard;
- d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- e) the test result(s) obtained or, if the repeatability has been checked, the final quoted result obtained.

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