INTERNATIONAL STANDARD 5516

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Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Ashing method

Fruits, légumes et produits dérivés — Décomposition des matières organiques en vue de l'analyse — Méthode par incinération

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FOREWORD

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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 5516 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in November 1976.

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

Australia	India	Portugal
Austria	Iran	Romania
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Canada	Korea, Rep. of	Spain
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Ireland
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Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Ashing method

0 INTRODUCTION

There are two methods for decomposition of the organic matter present in fruits, vegetables and derived products :

a) ashing method, described in this International Standard;

b) wet decomposition method (see ISO 5515).

The specific International Standards on analysis of the products will, if necessary, identify which method to use and any modifications to be made to the method.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the decomposition of the organic matter in fruits, vegetables or derived products by ashing, prior to the analysis of their mineral (metal) content.

2 REFERENCE

ISO 5515, *Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Wet method.*

3 PRINCIPLE

Incineration of a test portion at 525 ± 25 °C after addition, in certain cases, of aluminium chloride or magnesium acetate solution to facilitate ashing. Dissolution of the ash obtained in sulphuric acid or hydrochloric acid.

4 REAGENTS

All reagents shall be of recognized analytical quality.

For the preparation of solutions, for rinsing glassware, and in the procedure itself, use only water distilled in a borosilicate glass or silica apparatus and stored in a borosilicate glass or silica bottle.

4.1 Aluminium chloride solution, if necessary (see 6.3).

Dissolve 7,0 g of aluminium chloride hexahydrate ($AlCl_3.6H_2O$) in water and dilute to 100 ml.

or

4.2 Magnesium acetate solution, if necessary (see 6.3).

Dissolve 15,0 g of magnesium acetate [$(CH_3COO)_2Mg$] in water and dilute to 100 ml.

4.3 Sulphuric acid, ρ_{20} 1,84 g/ml.

or

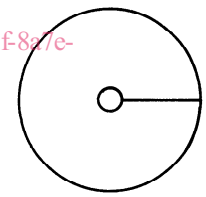
4.4 Hydrochloric acid, ρ_{20} 1,19 g/ml.

5 APPARATUS

Usual laboratory apparatus, and in particular :

5.1 Dish, of platinum or any other material not attacked under the conditions of the test, flat-bottomed, diameter about 60 mm, height about 35 mm.

5.2 Circle of ashless filter paper, fitting accurately into the dish (5.1). Cut out a round hole, of 2 to 3 mm diameter, from the centre and cut the paper along a radius (see the diagram).



5.3 Ashless filter paper, if necessary (see 6.3).

5.4 Electric muffle furnace, capable of being controlled at 525 ± 25 °C.

5.5 Suitable equipment for drying (for example, boiling water bath and oven capable of being maintained at 120 ± 5 °C) **and pre-incineration** (for example, gas burner or hot-plate).

It is also possible to use an overhead radiant heater with an infra-red lamp.

5.6 Volumetric flask, 50 ml or 100 ml, complying with ISO 1042.

5.7 Pipettes, of appropriate capacity, either one-mark, complying with ISO 648, or graduated, complying with ISO/R 835.

NOTE — Before use, wash the volumetric flask and the pipettes with hot nitric acid, and rinse thoroughly with water, distilled as described at the beginning of clause 4.

5.8 Analytical balance.

6 PROCEDURE

6.1 Preparation of test sample

Remove seeds and carpellary tissue, if necessary, and blend the sample carefully.

Frozen products shall be previously thawed in a closed container, and the liquid formed during thawing shall be added to the product before blending.

6.2 Test portion

6.2.1 *Liquid products*

Place 5 to 20 g, weighed to the nearest 0,001 g, or 5 to 20 ml, taken with a pipette (5.7), of the test sample in the dish (5.1).

6.2.2 *Other products*

Place 5 to 20 g, weighed to the nearest 0,001 g, of the test sample in the dish (5.1).

6.3 Pre-treatment of the test portion

In the case of products difficult to ash (for example, products of high sugar content), add to the test portion (6.2), with a pipette (5.7), 1,5 ml of the aluminium chloride solution (4.1) or magnesium acetate solution (4.2), mix thoroughly with a glass rod, clean the glass rod with a small piece of ashless filter paper (5.3) and place this piece of filter paper in the dish.

NOTES

1 Do not use aluminium chloride if the determination of aluminium content is to be carried out.

2 Do not use magnesium acetate if the determination of magnesium content is to be carried out.

6.4 Drying and pre-incineration

Place the prepared circle of filter paper (5.2) in the dish to cover the test portion completely, and proceed according to a) or b) using the appropriate equipment (see 5.5).

a) Place the dish on the boiling water bath, and evaporate off the greater part of the water; transfer the dish to the drying oven and leave it for 15 min. Finally, char the contents of the dish carefully over a gas burner or hot-plate. Do not allow the material to ignite.

b) Place the dish under the overhead radiant heater at a distance of about 11 to 12 cm, until the contents of the dish are dried and charred.

6.5 Incineration

Place the dish containing the charred material in the muffle furnace (5.4), controlled at 525 ± 25 °C. Incinerate until no more carbon particles can be seen in the ash.

If carbon particles remain after 60 min of incineration, cool, and moisten the ash with water. Evaporate off the water either on the boiling water bath followed by a brief period in the oven at 120 ± 5 °C, or under the overhead radiant heater. Replace the dish in the muffle furnace. The incineration is completed when no more carbon particles can be seen.

6.6 Preparation of test solution

Moisten the ash with 1 ml of the sulphuric acid (4.3) or hydrochloric acid (4.4) and **carefully** add 10 ml of water. Heat on the boiling water bath for a few minutes until the ash is dissolved. Transfer the cooled solution quantitatively into a 50 ml or 100 ml volumetric flask (5.7) and dilute to the mark with water.

7 TEST REPORT

The test report shall indicate the method used. In particular, it shall specify the mass of the test portion, whether aluminium chloride or magnesium acetate was added (see 6.3), and whether the solution was made up to 50 ml or 100 ml. It shall also mention any operating details not described in this International Standard or regarded as optional, as well as any circumstances that may have affected the result.

The test report shall give all the information necessary for complete identification of the sample.