SLOVENSKI STANDARD
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Sadje, zelenjava in sadni in zelenjavni proizvodi - Razklop organskih snovi - Mokri postopek

Fruits, vegetables and derived products -- Decomposition of organic matter prior to analysis -- Wet method

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Fruits, légumes et produits dérivés -- Décomposition des matières organiques en vue de l'analyse -- Méthode par voie humide

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INTERNATIONAL STANDARD 5515

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

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Internationai Standard ISO 5515 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in November 1976.

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

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France	Netherlands	United Kingdom
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Hungary	Peru	

No member body expressed disapproval of the document.

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

0 INTRODUCTION

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

a) wet decomposition method, described in this International Standard;

b) ashing method (see ISO 5516).

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.

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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 wet digestion, prior to the analysis of their mineral (metal) content.

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2 REFERENCES

ISO 2173, *Fruit and vegetable products — Determination of soluble solids content — Refractometric method.*

ISO 5516, *Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Ashing method.*

3 PRINCIPLE

Heating of the test portion until all organic matter is decomposed and a clear solution is obtained, either

— with sulphuric acid and nitric acid and in certain cases with hydrogen peroxide, or

— with sulphuric acid and nitric acid with the addition of perchloric 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 Nitric acid, ρ_{20} 1,42 g/ml.

4.2 Sulphuric acid, ρ_{20} 1,84 g/ml.

4.3 Hydrogen peroxide, 30 % (*m*/*m*) solution, if necessary (see 6.3.2).

4.4 Perchloric acid, ρ_{20} 1,67 g/ml, if necessary (see 6.4).

4.5 Hydrochloric acid, 5 N solution, if necessary (see 6.4).

Dilute 382,5 ml of concentrated hydrochloric acid, ρ_{20} 1,19 g/ml, to 1 000 ml with water.

4.6 Ammonium oxalate [$(COONH_4)_2$], concentrated solution, approximately 5 g in 100 ml, in the case of determination of tin (see 6.4).

5 APPARATUS

Usual laboratory apparatus, and in particular :

5.1 Kjeldahl flask, 300 ml, made from borosilicate glass or silica.

5.2 Digestion rack with controllable heating.

5.3 Hood.

5.4 Beads made from borosilicate glass.

5.5 One-mark volumetric flasks, 50 ml or 100 ml, complying with ISO 1042.

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

NOTE — Before use, wash all glassware with hot nitric acid (4.1), and rinse thoroughly with water, distilled as described at the beginning of clause 4.

5.7 Analytical balance.

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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 10 g, weighed to the nearest 0,001 g, or 5 to 10 ml, taken with a pipette (5.6), of the test sample in the Kjeldahl flask (5.1).

6.2.2 *Other products*

Place 5 to 10 g, weighed to the nearest 0,001 g, of the test sample (except in specific cases — see 7.1) in the Kjeldahl flask (5.1).

6.3 Decomposition without use of perchloric acid

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6.3.1 *Products having soluble solids contents* (see ISO 2173) *below* 40 % (*m*/*m*)

Add to the test portion a mixture of 30 ml of the nitric acid (4.1) and at least 10 ml of the sulphuric acid (4.2), together with some glass beads (5.4).

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In some cases it may be useful to effect a preliminary digestion, by leaving the mixture in contact in the flask for a period (overnight for example).

Place the flask containing the mixture on the digestion rack (5.2) and heat cautiously, to avoid excessive frothing.

If necessary, interrupt heating and begin again only when vigorous frothing has ceased.

As soon as possible, bring the liquid to the boil and continue boiling until it begins to turn brown. Then begin to add 1 to 2 ml portions of the nitric acid (4.1), drop by drop.

Bring again to the boil after every addition, but avoid vigorous heating. A small amount of nitric acid shall always remain in the mixture, as indicated by the presence of nitrous vapours.

Cease addition of portions of nitric acid when the solution no longer turns brown on addition of the acid. Continue heating until white fumes appear, indicating a high concentration of sulphuric acid and a reduction in nitric acid. If the solution turns brown again, continue the addition of nitric acid and repeat the operations described above until browning ceases.

Allow the solution to cool. The absence of colour or the presence of a light green or yellow colour indicates that the digestion is complete.

Carefully add 15 ml of water to the cold solution, and boil until white fumes appear. Repeat this operation twice more.

Cool and dilute the solution carefully with 5 to 20 ml of water.

Use all of this solution for the analysis of mineral content. Otherwise, transfer the solution quantitatively into a 50 ml or 100 ml volumetric flask (5.5) and dilute to the mark with water.

6.3.2 *Products having soluble solids contents* (see ISO 2173) *higher than* 40 % (*m*/*m*)

Proceed according to 6.3.1.

If the digestion is not complete after the gradual addition of a total of 10 ml of the nitric acid (4.1) and a final period of heating, cool the brown solution and add, drop by drop, 1 to 2 ml of the hydrogen peroxide solution (4.3) and a few drops of the nitric acid.

Boil gently at first, then more vigorously, until brown fumes appear.

Repeat these operations until the solution remains colourless. Boil until nitric acid is eliminated and white fumes appear.

If the solution continues to turn brown, continue the addition of nitric acid and hydrogen peroxide and proceed as above until browning ceases. Allow the solution to cool. Decomposition is complete when the solution remains colourless after cooling.

Then add 15 ml of water and boil until white fumes appear. Repeat this operation twice more.

Allow to cool and dilute the solution carefully with 5 to 20 ml of water.

Use all of this solution for the analysis of mineral content. Otherwise, transfer the solution quantitatively into a 50 ml or 100 ml volumetric flask (5.5) and dilute to the mark with water.

6.4 Decomposition using perchloric acid

6.4.1 *Precautions*

Contact with oxidizable or combustible substances or with dehydrating or reducing agents may result in a fire or an explosion.

Persons handling perchloric acid shall therefore be informed of these risks. Safety instructions shall include the following :

a) Remove immediately any spilt perchloric acid by washing with large quantities of water.

b) Fume cupboards, ducting and other equipment intended for evacuation of perchloric acid vapour shall be of chemically inert material and designed to facilitate washing with water. Evacuation systems shall discharge into a safe place, and the ventilators shall be accessible for cleaning.

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c) Avoid handling organic compounds in fume cupboards used for perchloric acid decomposition.

d) Use goggles, protection screens and other equipment necessary for the protection of personnel.

e) Only add perchloric acid to dilute solutions containing nitric acid. Do not evaporate to dryness.

f) Contact of perchloric acid solutions with powerful dehydrating agents such as phosphorus(V) chloride or concentrated sulphuric acid can result in explosion of anhydrous perchloric acid.

Perchloric acid is extremely sensitive to mechanical shocks and to heat when its concentration exceeds 72 %.

6.4.2 *Products having a soluble solids content* (see ISO 2173) *below* 15 % (*m/m*) *and not containing a significant quantity of starch*

Add to the test portion 6 to 8 ml of the nitric acid (4.1) and bring to the boil.

Allow to cool, and add 6 to 8 ml of the nitric acid (4.1), 4 ml of the sulphuric acid (4.2) and 3 ml of the perchloric acid (4.4). Boil slowly at first, then more vigorously, until the solution becomes colourless (or green or yellow) and white fumes appear.

If the solution has not lost its colour after 20 to 25 min, cool, add a few millilitres of the nitric acid (4.1) and 1 to 2 ml of the perchloric acid (4.4) and boil for 4 to 5 min. Cool. The digestion period is generally between 25 and 35 min.

6.4.3 *Products having a soluble solids content* (see ISO 2173) *between* 15 *and* 25 % (*m/m*) *or below* 15 % (*m/m*) *and containing a significant quantity of starch*

Add to the test portion 6 to 8 ml of the nitric acid (4.1) and bring to the boil. The solution should still contain some nitric acid after boiling. Also it is preferable, after cooling, to add 6 to 8 ml more of nitric acid and bring to the boil again. Repeat this procedure once more.

Allow to cool, add 6 to 8 ml of the nitric acid (4.1), 4 ml of the sulphuric acid (4.2) and 3 to 5 ml of the perchloric acid (4.4) and boil until the solution becomes colourless. Cool. The digestion period is generally between 35 and 45 min.

6.4.4 *Products having a soluble solids content (see ISO 2173) over* 25 % (*m/m*)

Add to the test portion 6 to 8 ml of the nitric acid (4.1), leave in contact overnight and bring to the boil.

After cooling, add a further portion of 6 to 8 ml of the nitric acid and bring to the boil. Repeat this procedure once or twice.

After cooling, add 6 to 8 ml of the nitric acid and 4 ml of the sulphuric acid (4.2). Decomposition commences violently without heating. After the violent reaction stops, cool, add 4 to 6 ml of the nitric acid and 3 to 5 ml of the perchloric acid and boil until the solution becomes colourless. Cool. The digestion period is generally between 120 and 150 min.

6.4.5 *All products*

In all cases above (6.4.2, 6.4.3 and 6.4.4), remove the remaining traces of nitric acid and perchloric acid. Add 10 ml of water to the cold solution and boil until white fumes appear. Cool, add 15 ml of the hydrochloric acid (4.5) and boil again until white fumes appear.

NOTE — For the determination of arsenic, boil with water instead of with hydrochloric acid. For the determination of tin, add the concentrated solution of ammonium oxalate (4.6).

Use the whole of the solution for the analysis of mineral content. Otherwise, transfer the solution quantitatively into a 50 ml or 100 ml volumetric flask (5.5) and dilute to the mark with water.

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7 SPECIAL CASES

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7.1 Dehydrated products

Use the procedure described in 6.3 or in 6.4, according to the soluble solids content of the product, but using a test portion (6.2.2) of 2 to 5 g with 30 ml of water.

7.2 Products containing ethanol

Add to the test portion (6.2) 100 ml of water. Boil until most of the water has evaporated.

Then use the procedure described in 6.3 or in 6.4, according to the soluble solids content of the product.

8 TEST REPORT

The test report shall indicate the method used. In particular, it shall specify the mass of the test portion, whether hydrogen peroxide was added or if the decomposition was carried out with perchloric acid, 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 information necessary for complete identification of the sample.