International Standard 6636/2

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Fruits, vegetables and derived products — Determination of zinc content — Part 2 : Atomic absorption spectrometric method

Fruits, légumes et produits dérivés — Détermination de la teneur en zinc — Partie 2 : Méthode par spectrométrie d'absorption atomique

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Foreword

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

Australia	Hungary	Portugal
Austria	India	Romania
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Bulgaria	Israel	Spain
Canada	Italy	Thailand
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No member body expressed disapproval of the document.

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Fruits, vegetables and derived products — Determination of zinc content — Part 2 : Atomic absorption spectrometric method

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1 Scope and field of application

This part of ISO 6636 specifies an atomic absorption spectrometric method for the determination of the zinc content of fruits, vegetables and derived products.

2 References

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

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

3 Principle

Decomposition of organic matter by the dry or wet method and determination of the Zn^{2+} cation by atomic absorption spectrometry.

NOTE — In the case of decomposition by the dry method, dissolution of the ash in hydrochloric acid allows transformation of all mineral salts into easily dissociable chlorides.

For some liquid samples (such as wines, clear fruit juices free from pulp), the determination may be carried out directly, without previous decomposition.

4 Reagents

All reagents shall be of recognized analytical purity, and shall be especially free of zinc. The water used shall have been distilled twice in an apparatus of borosilicate glass, or shall be water of at least equivalent purity.

4.1 Nitric acid, ϱ_{20} = 1,38 g/ml.

4.2 Sulphuric acid, ϱ_{20} = 1,84 g/ml.

4.3 Hydrochloric acid, 1 + 1 (by volume) solution.

Mix one volume of concentrated hydrochloric acid (ϱ_{20} = 1,19 g/ml) with one volume of water.

4.4 Hydrochloric acid, approximately 3,7 g/l solution.

In a 1 000 ml one-mark volumetric flask, dilute 8,3 ml of concentrated hydrochloric acid (ϱ_{20} = 1,19 g/ml) to the mark with water and mix.

4.5 Zinc, standard solution corresponding to 1 g of zinc per litre.

In a conical flask, dissolve 1 g of pure zinc in 10 ml of the hydrochloric acid solution (4.3). Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark with water, and mix.

Store the solution in a bottle of borosilicate glass fitted with a ground glass stopper.

5 Apparatus

Usual laboratory equipment not otherwise specified, and the following :

5.1 Mechanical grinder, the inside and blades of which are coated with polyethylene.

5.2 Platinum or quartz dishes, of diameter 70 mm, or **Kjeldahl flasks,** of capacity 250 ml.

5.3 One-mark volumetric flasks, of capacity 50 ml.

5.4 Volumetric pipettes, of appropriate capacities.

5.5 Centrifuge.

5.6 Boiling water bath.

5.7 Heating device.

5.8 Electrically heated muffle furnace, capable of being controlled at 525 ± 25 °C.

5.9 Electrically heated oven, capable of being controlled at less than 100 °C and at 525 ± 25 °C, preferably programmable between 20 and 550 °C.

5.10 Atomic absorption spectrometer, fitted with a burner for an air-acetylene mixture fed at a pre-determined flow rate (in general 4 ml/min) corresponding to the specified optimum aspiration rate, suitable for measurements at a wavelength of 213,8 nm.

5.11 Analytical balance.

6 Procedure

6.1 Preparation of the test sample

Mix the laboratory sample well. If necessary, first remove seeds and hard seed-cavity walls and pass through the mechanical grinder (5.1).

Allow frozen or deep-frozen products to thaw in a closed vessel, and add the liquid formed during this process to the product before homogenization.

6.2 Test portion

6.2.1 Liquid products

Take 10 ml of the test sample (6.1) by means of a pipette (5.4). In the case of viscous liquids or liquids containing suspended solid particles, weigh the test portion (see 6.2.2).

6.2.2 Pasty, solid or dried products

Weigh, to the nearest 0,01 g, 5 to 10 g of the test sample (6.1), expressed as fresh product, according to the nature of the product.

6.3 Decomposition

Decomposition may be carried out by the dry or wet method.

6.3.1 Decomposition by the dry method

Introduce the test portion (6.2) into one of the dishes (5.2) and place it on the boiling water bath (5.6), regulating the temperature of the bath so as to minimize the risk of loss of material by spattering. Evaporate to dryness. Continue the decomposition in the muffle furnace (5.8), controlled at 525 ± 25 °C.

NOTE — If possible, it is preferable to avoid evaporation on a boiling water bath and to place the dish directly in the electric oven (5.9) which is temperature programmable from 20 to 525 ± 25 °C, in progressive steps, to avoid spattering of the test portion during drying.

Dissolve the ashes in a few drops of the nitric acid (4.1), evaporate on the boiling water bath (5.6), then transfer to the oven (5.9) controlled at 525 ± 25 °C (or controlled at first at a temperature less than 100 °C and then at 525 ± 25 °C). Transfer to the muffle furnace (5.8), controlled at 525 ± 25 °C, and leave until white ashes are obtained. Dissolve the ashes in 1 to 2 ml of the hydrochloric acid solution (4.3). Transfer the contents of the dish quantitatively to a centrifuge tube (5.5), rinsing the dish with about 20 ml of the hydrochloric acid solution (4.4), centrifuge, and transfer the supernatant liquid to a 50 ml volumetric flask (5.3). Add a further 10 ml of the hydrochloric acid solution (4.4) to the contents of the centrifuge tube, centrifuge, and transfer the supernatant liquid to the same flask. Repeat this procedure using 10 ml of water and make up the volume in the volumetric flask to the mark with water. Mix the solution.

6.3.2 Decomposition by the wet method

Introduce the test portion (6.2) into a Kjeldahl flask (5.2). If the test portion (6.2.1) contains ethanol, eliminate it beforehand by boiling, and allow to cool. Add 10 ml of the nitric acid (4.1), heat, then carefully add 5 ml of the sulphuric acid (4.2).

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 heating device (5.7) 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 add, drop by drop, 1 to 2 ml portions of the nitric acid (4.1).

Bring 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.

When decomposition is terminated, dilute the sulphuric solution with a few millilitres of water. Transfer the contents of flask quantitatively to a centrifuge tube (5.5), rinsing the flask with about 10 ml of water and collecting the rinsing water in the centrifuge tube. Centrifuge and transfer the supernatant liquid to a 50 ml volumetric flask (5.3). Add a further 10 ml of water to the contents of the centrifuge tube, centrifuge and transfer the supernatant liquid to the same flask. Repeat this procedure with another 10 ml of water and make up the volume in the volumetric flask to the mark with water. Mix the solution.

6.3.3 Blank test

Carry out a blank test, using the same conditions for decomposition (6.3.1 or 6.3.2 as appropriate), but replacing the test portion (6.2) by 10 ml of water.

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6.4 Determination[1)]

6.4.1 Sample decomposed by the dry method

6.4.1.1 Preparation of the calibration graph

Dilute the standard zinc solution (4.5) with the hydrochloric acid solution (4.4) to obtain four solutions containing 0,25 — 0,5 — 1 and 1,5 mg of zinc per litre.

Aspirate each of these solutions, in turn, into the flame of the spectrometer (5.10), at the rate such that the maximum absorbance is obtained for the solution having a zinc content of 1,5 mg/l. Record the corresponding values of absorbance and draw the calibration graph.

6.4.1.2 Spectrometric measurements

Aspirate the test solution obtained (6.3.1) and the blank test solution (6.3.3) into the flame of the spectrometer (5.10) at the same rate as in 6.4.1.1. Record the corresponding absorbances.[2)]

The absorbance of the blank test solution shall be less than or equal to 0,002.

6.4.2 Sample decomposed by the wet method

6.4.2.1 Preparation of the calibration graph

Dilute the standard zinc solution (4.5) with water to obtain four solutions containing 2,5 — 5 — 10 and 15 mg of zinc per litre.

Into a series of four 50 ml volumetric flasks (5.3), place 5 ml of each of these solutions. Add 30 to 35 ml of water, and then 5 ml of the sulphuric acid (4.2). Mix, allow to cool and dilute to the mark with water. Mix. These solutions contain respectively 0,25 — 0,5 — 1 and 1,5 mg of zinc per litre.

Aspirate each of these solutions, in turn, into the flame of the spectrometer (5.10), at the rate such that the maximum absorbance is obtained for the solution having a zinc content of 1,5 mg/l. Record the corresponding values of absorbance and draw the calibration graph.

6.4.2.2 Spectrometric measurements

Aspirate the test solution (6.3.2) and the blank test solution (6.3.3) into the flame of the spectrometer (5.10) at the same rate as in 6.4.2.1. Record the corresponding absorbances.[3)]

The absorbance of the blank test solution shall be less than or equal to 0,002.

7 Expression of results

7.1 Method of calculation and formulae

7.1.1 Liquid products

The zinc content, expressed in milligrams per litre of product, is given by the formula

$$(c_1 - c_2) \times 5$$

where

c_1 is the zinc content of the sample, in milligrams per litre, read from the calibration graph;[4)]

c_2 is the zinc content of the blank test solution, in milligrams per litre, read from the calibration graph.

7.1.2 Viscous or inhomogeneous liquids, pasty, solid or dried products

The zinc content, expressed in milligrams per kilogram of product, is given by the formula

$$\frac{(c_1 - c_2) \times 50}{m}$$

where

c_1 is the zinc content of the sample, in milligrams per litre, read from the calibration graph;[4)]

c_2 is the zinc content of the blank test solution, in milligrams per litre, read from the calibration graph;

m is the mass, in grams, of the test portion.

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1) It is also possible to use solutions obtained using the procedures specified in ISO 5515 or ISO 5516, provided that precautions are taken to avoid the presence of zinc in the reagents.

2) If the absorbance of the test solution exceeds that of the most concentrated calibration solution, measure the absorbance of the test solution suitably diluted with the hydrochloric acid solution (4.4).

3) If the absorbance of the test solution exceeds that of most concentrated calibration solution, measure the absorbance of the test solution suitably diluted with 10 % (*V*/*V*) sulphuric acid solution.

4) If the test solution was diluted, use the appropriate dilution factor in the calculation.

If it is desired to express the zinc content relative to the dry product, take the moisture content of the sample into account in the calculation.

7.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst, on the same sample, shall not exceed 10 % (relative).

8 Test report

The test report shall show the method used and the result obtained, indicating clearly the method of expression used. It shall also mention all operating details not specified in this International Standard or regarded as optional, as well as any incidents likely to have affected the results.

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

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