International Standard 6636/1

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Fruits, vegetables and derived products — Determination of zinc content — Part 1 : Polarographic method

Fruits, légumes et produits dérivés — Détermination de la teneur en zinc — Partie 1 : Méthode polarographique

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Foreword

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Fruits, vegetables and derived products — Determination of zinc content — Part 1 : Polarographic method

1 Scope and field of application

This part of ISO 6636 specifies a polarographic method for the determination of the zinc content of fruits, vegetables and derived products.

NOTE — ISO 6636/2 specifies an atomic absorption spectrometric method, and ISO 6636/3 a dithizone spectrometric method.

Copper, tin, lead and cadmium do not interfere with the determination.

2 Reference

ISO 874, *Fresh fruits and vegetables — Sampling.*

3 Principle

Ashing of a test portion in a muffle furnace at 525 ± 25 °C. Treatment of the ash obtained with hydrochloric acid. Neutralization with 25 % (*m*/*m*) ammonia solution and determination of the zinc content by polarography using an ammonia/ammonium chloride electrolyte.

4 Reagents

All reagents shall be of recognized analytical grade and the water used shall be distilled water or water of at least equivalent purity.

4.1 Nitric acid (ϱ_{20} = 1,38 g/ml).

4.2 Hydrochloric acid, diluted 1 + 1.

Dilute 1 part by volume of hydrochloric acid (ϱ_{20} = 1,19 g/ml) with 1 part by volume of water.

4.3 Ammonia, 25 % (*m*/*m*) solution.

4.4 Electrolyte solution.

Dissolve 53,5 g of ammonium chloride in water in a 1 000 ml one-mark volumetric flask, add 155 ml of the ammonia solution (4.3), make up to the mark with water and mix.

4.5 Sodium sulfite (Na_2SO_3), 1 mol/l solution.

4.6 Zinc, standard solution corresponding to 0,01 to 0,04 mg of Zn per millilitre.

4.6.1 Stock solution

Dissolve 1 g of metallic zinc [purity at least 99 % (*m*/*m*)] in 10 ml of the hydrochloric acid (3.2) in a conical flask. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix.

4.6.2 Preparation

Dilute 1 to 4 ml of the zinc stock solution (4.6.1) to the mark with water in a 100 ml one-mark volumetric flask and mix.

5 Apparatus

Usual laboratory equipment and in particular

5.1 Polarograph, suitable for determining zinc contents greater than 0,05 mg/kg, provided with a mercury-dropping electrode as the cathode and an electrolyser with mercury at the bottom as the anode.

5.2 Drying oven, capable of being maintained at 100 to 150 °C within a tolerance not exceeding ± 5 °C.

5.3 Muffle furnace, capable of being maintained at 100 to 700 °C within a tolerance not exceeding ± 25 °C.

5.4 Porcelain dishes, of diameter 9 to 11 cm.

5.5 One-mark volumetric flask, of capacity 50 ml.

5.6 Graduated pipettes, of capacities 1 and 10 ml.

5.7 Conical flask, of capacity 25 ml.

5.8 Analytical balance.

5.9 Boiling water-bath.

6 Sampling

For the sampling of fresh fruits and vegetables, see ISO 874.

Homogenize the laboratory sample thoroughly before taking the test portion. Allow frozen and deep-frozen products to thaw in a closed vessel and mix the liquid formed during this process with the product during homogenization.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, 10 to 25 g of the test sample, according to the expected zinc content, into one of the porcelain dishes (5.4).

7.2 Decomposition

7.2.1 Place the dish containing the test portion (7.1) in the oven (5.2) and dry at a temperature of 110 to 120 °C. Then transfer to the muffle furnace (5.3) maintained at 250 °C. Slowly raise the temperature to 350 °C and maintain this temperature until there is no more foam. Raise the temperature gradually to 525 °C (the test portion shall not ignite) and ash for 6 h. Remove the dish from the muffle furnace and allow it to cool. If the ash contains a large amount of carbon particles, proceed as follows :

Wet the ash with 0,5 ml of water and then with 0,5 ml of nitric acid (4.1)

Evaporate to dryness on the boiling water-bath (5.9). Transfer to the muffle furnace, maintained at 250 °C, increase the temperature to 525 °C and continue heating for 1 to 2 h. Repeat this procedure if necessary, until the ash is free from carbon particles.

7.2.2 Add to the ash 10 ml of hydrochloric acid (4.2) and place on the boiling water-bath to aid dissolution. Allow to cool.

7.2.3 Quantitatively transfer the solution to a 50 ml volumetric flask (5.5), and wash the sides of the dish with 5 to 10 ml of water, collecting the washings in the same volumetric flask.

7.2.4 Add ammonia solution (4.3) to the solution until the smell of ammonia appears (pH 8). Then add an excess until the pH is 10. Make up to the mark with water, mix and filter.

7.3 Blank test

Carry out a blank test in parallel with the determination, by the same procedure, using the same quantities of all the reagents, but replacing the test portion with an equal mass of water.

7.4 Determination

7.4.1 Transfer, by means of a pipette, two 8 ml aliquot portions of the filtered solution (7.2.4) to two conical flasks (5.7).

7.4.2 Add 1 ml of the sodium sulfite solution (4.5) and 1 ml of water to the contents of one of the conical flasks and make up to 25 ml with the electrolyte solution (4.4). Mix thoroughly. Transfer the solution to the electrolyser of the polarograph, rinsing this beforehand with a small amount of the test solution.

7.4.3 Carry out the polarographic measurement by scanning between −1,0 and −1,6 V, following the manufacturer's instructions. Set the sensitivity according to the expected zinc content. The setting will vary from instrument to instrument. The half-wave potential, $E_{1/2}$, for zinc is approximately −1,2 V. The mercury dropping rate shall be 10 drops per 25 to 30 s.

7.4.4 After recording the first polarogram, empty the electrolyser and before using it again, wash it with small amounts of the next test solution.

7.4.5 To the contents of the second conical flask add 1 ml of the sodium sulfite solution (4.5) and a known volume of the zinc standard solution (4.6), not exceeding 1 ml. Make up to 25 ml with the electrolyte solution (4.4). Proceed as specified in 7.4.2. Repeat this procedure twice more, with similar additions of the zinc standard solution, and obtain the zinc content by extrapolation. Correct this value for the result of the blank test.

7.5 Number of determinations

Carry out two determinations on test portions taken from the same sample.

8 Expression of results

8.1 Calculation

The zinc content, expressed in milligrams per kilogram of product, is equal to

$$\frac{\varrho_{Zn} V_4 h_1 V_1 V_0 \times 1\,000}{(h_2 V_2 - h_1 V_1)\, m V_3}$$

where

h_1 is the height, in millimetres, of the polarographic wave obtained in the first measurement (see 7.4.3);

h_2 is the height, in millimetres, of the polarographic wave obtained in the second measurement (see 7.4.5);

m is the mass, in grams, of the test portion (7.1);

V_0 is the total volume, in millilitres, of solution prepared from the decomposed test portion (see 7.2.4);

V_1 is the volume, in millilitres, of solution prepared for the first measurement (see 7.4.2);

V_2 is the volume, in millilitres, of solution prepared for the second measurement (see 7.4.5);

V_3 is the volume, in millilitres, of the aliquot portion taken for the preparation of the solution for analysis (see 7.4.1);

V_4 is the volume, in millilitres, of the zinc standard solution added (see 7.4.5);

ϱ_{Zn} is the concentration, in milligrams per millilitre, of the zinc standard solution (4.6).

Take as the result the arithmetic mean of the values obtained in the two determinations (7.5), provided that the requirement for repeatability (see 8.2) is satisfied.

Report the result to one decimal place.

8.2 Repeatability

The difference between the results of the two determinations (7.5), carried out simultaneously or in rapid succession under the same conditions (same operator, same apparatus, same laboratory), shall not exceed 5 % of the mean.

9 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

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

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