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Fruit and vegetable products -- Determination of zinc content -- Part 3: Dithizone spectrometric method

Produits dérivés des fruits et légumes -- Détermination de la teneur en zinc -- Partie 3: Méthode spectrométrique à la dithizone

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International Standard 6636/3

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Fruit and vegetable products — Determination of zinc content — Part 3 : Dithizone spectrometric method

Produits dérivés des fruits et légumes — Détermination de la teneur en zinc — Partie 3 : Méthode spectrométrique à la dithizone

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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 6636/3 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in April 1982.

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

Austria	Kenya	Romania
Canada	Korea, Dem. P. Rep. of	South Africa, Rep. of
Czechoslovakia	Malaysia	Spain
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India	Philippines	Turkey
Iran	Poland	USSR
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Netherlands

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Fruit and vegetable products — Determination of zinc content — Part 3 : Dithizone spectrometric method

1 Scope and field of application

This part of ISO 6636 specifies a dithizone spectrometric method for the determination of the zinc content of fruit and vegetable products.

2 Reference

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

3 Principle

Decomposition of organic matter, neutralization of the solution obtained and addition of dithizone (1,5-diphenylthiocarbazone) solution. Extraction of the zinc complex thus formed with chloroform and spectrometric measurement of the absorbance of the extract at a wavelength of 538 nm.

4 Reagents

All reagents shall be of recognized analytical quality and, with the exception of the standard zinc solutions (4.8 and 4.9), shall, in particular, be free from zinc. The water used shall be double-distilled water or water of at least equivalent purity.

4.1 Sulfuric acid, 25 % (V/V) solution.

4.2 Ammonia, 25 % (V/V) solution.

4.3 Phenol red, indicator solution.

Dissolve, in a mortar, 0,1 g of phenolsulfonephthalein in 2,85 ml of 0,1 mol/l sodium hydroxide solution and dilute to 100 ml with water.

4.4 Sodium acetate trihydrate, 100 g/l solution.

4.5 Sodium thiosulfate, 250 g/l solution.

4.6 Hydrochloric acid, concentrated, ϱ_{20} = 1,19 g/ml.

4.7 Chloroform.

4.8 Zinc, standard solution corresponding to 500 µg of zinc per millilitre.

In a 1 000 ml volumetric flask, dissolve 0,500 g of pure granulated zinc in 20 ml of the concentrated hydrochloric acid (4.6), and dilute to the mark with water.

4.9 Zinc, standard solution corresponding to 5 µg of zinc per millilitre.

In a 1 000 ml volumetric flask, dilute 10 ml of the standard zinc solution (4.8) to the mark with the 0,04 mol/l hydrochloric acid solution (4.10).

Prepare this solution at the time of use.

4.10 Hydrochloric acid, 0,04 mol/l solution.

4.11 1,5-diphenylthiocarbazone (dithizone), solution.

Dissolve 0,2 g of dithizone in 100 ml of the chloroform (4.7).

4.12 1,5-diphenylthiocarbazone (dithizone), extraction solution.

Dilute 1,0 ml of the dithizone solution (4.11) to 100 ml with the chloroform (4.7).

5 Apparatus

NOTE — Contamination may lead to high values in the blank test and the apparatus used should, therefore, be rinsed with the dithizone solution.

Usual laboratory apparatus, and

5.1 One-mark volumetric flasks, of capacities 15, 50, 100, 500 and 1 000 ml.

5.2 Separating funnels, of capacity 250 ml.

5.3 Spectrometer, suitable for making measurements at a wavelength of 538 nm, equipped with cells of optical path length 1 cm.

6 Procedure

6.1 Preparation of the test sample

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.

6.2 Test portion

Weigh, to the nearest 0,01 g, 5 to 10 g of the test sample (6.1), according to the expected zinc content.

6.3 Destruction of organic matter

Use the procedure specified in ISO 5515.

NOTE — Milling and sieving of the sample, if necessary, should be carried out in equipment free from zinc and/or copper alloys before the destruction of organic matter.

6.4 Determination

According to the expected zinc content, dilute the solution obtained (see 6.3) to 50 or 100 ml in a volumetric flask. Place 1 to 10 ml of this solution in one of the separating funnels (5.2) and dilute to 20 ml with water.

Carry out the following steps in subdued light.

Add 2 drops of the phenol red indicator (4.3) and make the solution alkaline by addition of the ammonia solution (4.2) until the colour turns from yellow to red (pH 7,8 to 8,3). Cool the warm solution under a stream of water, loosening and replacing the stopper of the separating funnel from time to time.

Add to the cooled solution, 1 ml of the sodium thiosulfate solution (4.5) and 15 ml of the sodium acetate solution (4.4). Add, drop by drop, 3 ml of the dithizone extraction solution (4.12). Shake vigorously for 3 min after the addition of the dithizone.

Allow the phases to separate. If the organic phase is white, the extraction is complete. If the organic phase is red, continue adding, drop by drop, the dithizone extraction solution until the organic phase becomes white. Shake vigorously for 3 min. Allow the phases to separate.

NOTE — If a large amount of the dithizone extraction solution is added at one time the organic phase will become green and the sample will be unusable.

Collect the organic phase in a dried 15 ml one-mark volumetric flask, dilute to the mark with the chloroform (4.7) and mix thoroughly.

Measure the absorbance of the solution using the spectrometer (5.3) set at a wavelength of 538 nm, using attenuated light and pure chloroform as the reference liquid.

NOTE — If the digested sample solution contains less than 5 µg of zinc, the dilution described in the first paragraph of 6.4 may be omitted. This fact should be taken into account, however, when preparing the calibration graph.

6.5 Blank test

Simultaneously with each set of determinations, carry out a blank test by replacing the digested sample solution with 10 ml of the sulfuric acid solution (4.1) and proceeding as described in 6.4.

6.6 Preparation of the calibration graph

Into a series of four separating funnels, introduce 1 — 2 — 4 and 5 ml of the standard zinc solution (4.9) and dilute to 10 ml with the sulfuric acid solution (4.1). Then add the reagents and carry out the dilution with the chloroform as specified in 6.4.

These solutions contain 5 — 10 — 20 and 25 µg of zinc, respectively.

Measure the absorbances of these calibration solutions using the spectrometer following the procedure specified in 6.4.

Check the calibration graph regularly each day, as the specific absorbance values may vary according to the actual concentration of the dithizone solution.

NOTE — If the digested sample solution is not diluted (see the note to 6.4), place 0,2 — 0,4 — 0,6 and 0,8 ml of the standard zinc solution (4.9) in the separating funnels, and then add the reagents specified in 6.4. These solutions contain 1 — 2 — 3 and 4 µg of zinc, respectively. Measure the absorbances of these calibration solutions using the spectrometer following the procedure specified in 6.4.

6.7 Number of determinations

Carry out two determinations on the same test sample taken for the destruction of organic matter (see 6.1).

7 Expression of results

7.1 Method of calculation and formulae

7.1.1 Calculation of specific absorbance

From the absorbances of the calibration solutions (see 6.6), calculate the individual specific absorbances from the formulae

$$A_1 = \frac{E_1 - E_0}{5}$$

$$A_2 = \frac{E_2 - E_0}{10}$$

$$A_3 = \frac{E_3 - E_0}{20}$$

$$A_4 = \frac{E_4 - E_0}{25}$$

where

A_1, A_2, A_3 and A_4 are the individual specific absorbances;

E_1, E_2, E_3 and E_4 are the absorbances determined as described in 6.6;

E_0 is the absorbance of the blank test solution.

Calculate the overall specific absorbance, A, from the formula

$$A = \frac{A_1 + A_2 + A_3 + A_4}{4}$$

7.1.2 Calculation of zinc content

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

$$\frac{E \times V_0}{A \times m \times V_1}$$

where

E is the absorbance of the test solution;

A is the overall specific absorbance;

V_0 is the total volume, in millilitres, of the diluted digested sample solution (50 or 100 ml) (see 6.4);

V_1 is the volume, in millilitres, of the aliquot portion of digested sample solution transferred to the separating funnel;

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

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 test sample shall not exceed 5 % of the mean value.

8 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 part of ISO 6636, or in the International Standard to which reference is made, or regarded as optional, as well as any circumstances which may have influenced the result.

The test report shall give all the details required for the complete identification of the sample.