INTERNATIONAL STANDARD

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Fruits, vegetables and derived products — Determination of iron content by flame atomic absorption spectrometry

Fruits, légumes et produits dérivés — Détermination de la teneur en fer par spectrométrie d'absorption atomique avec flamme

Reference number
ISO 9526:1990(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Fruits, vegetables and derived products — Determination of iron content by flame atomic absorption spectrometry

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the iron content of fruits, vegetables and derived products.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5515:1979, *Fruits, vegetables and derived products — Decomposition of organic matter prior to analysis — Wet method.*

3 Principle

Decomposition of organic matter by either a dry or a wet method and determination of the Fe^{2+} cation content by flame atomic absorption spectrometry.

4 Reagents

All reagents used shall be of recognized analytical grade and, in particular, shall be free from iron. The water used shall have been distilled twice in borosilicate glass apparatus, or shall be water of at least equivalent purity.

4.1 Sulfuric acid, concentrated (ρ_{20} = 1,84 g/ml).

4.2 Nitric acid, concentrated (ρ_{20} = 1,38 g/ml).

4.3 Hydrochloric acid, diluted 1 + 1 (V/V).

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

4.4 Hydrochloric acid, approximately 0,1 mol/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 Iron, standard solution corresponding to 1 g of iron per litre.

In a 1 000 ml one-mark volumetric flask, dissolve 7,022 g of ammonium iron(II) sulfate hexahydrate [$(NH_4)_2Fe(SO_4)_2.6H_2O$] in water and dilute to the mark with water.

Store this solution in a borosilicate glass flask fitted with a ground glass stopper.

1 ml of this standard solution contains 1 mg of Fe.

5 Apparatus

Prior to use, wash the dishes and all glassware with warm (70 °C to 80 °C) concentrated nitric acid and rinse using twice-distilled water.

Usual laboratory apparatus and, in particular, the following.

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

5.2 Round-bottom flask, of 1 000 ml capacity.

5.3 Dishes, made of platinum or quartz, of 70 mm diameter.

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

5.5 Pipettes, for preparation of the calibration solutions.

5.6 Filter papers, ash-free.

5.7 Boiling water-bath.

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

5.9 Atomic absorption spectrometer, fitted with an air-acetylene burner, suitable for measurements at a wavelength of 248,3 nm.

5.10 Infrared lamp or, in the absence of this, **Bunsen burner.**

5.11 Analytical balance.

6 Preparation of the test sample

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

Frozen or deep-frozen products shall be previously thawed in a closed vessel, and the liquid formed during this process shall be added to the product before mixing.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, 5 g to 10 g of the test sample (clause 6), according to the nature of the product.

7.2 Decomposition

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

7.2.1 Decomposition using the dry method

Put the test portion (7.1) into one of the dishes (5.3) and place it on the boiling water-bath (5.7). Evaporate to dryness. Begin the combustion of the organic matter using an infrared lamp (5.10) or, in the absence of this, using a Bunsen burner, and continue the decomposition in the electrically heated muffle furnace (5.8), controlled at 525 °C ± 25 °C, until the resulting ash has turned white. If carbonized particles persist, add several drops of nitric acid (4.2) to the ashes, evaporate to dryness on the boiling water-bath and then put the dish and its contents back into the muffle furnace. Dissolve the ashes in approximately 1 ml to 2 ml of the hydrochloric acid solution (4.3), add approximately 20 ml of distilled water and leave the dish on the boiling water-bath until evaporation begins. Add 20 ml of the hydrochloric acid solution (4.4) and heat on the water-bath for approximately 5 min.

Filter through an ash-free filter paper (5.6) and collect the filtrate in a one-mark 50 ml volumetric flask (5.4). Rinse the dish and filter paper several times using 5 ml to 10 ml volumes of the hydrochloric acid solution (4.4), and collect the rinsings in the one-mark volumetric flask. Fill to the mark with the hydrochloric acid solution (4.4) and mix.

7.2.2 Decomposition using the wet method

Put the test portion (7.1) into a round-bottom flask (5.2). If the test portion contains ethanol, remove the ethanol by evaporation. Add 5 ml of the nitric acid (4.2), heat, then carefully add 5 ml of the sulfuric acid (4.1)[1]. Then proceed as specified in ISO 5515:1979, subclause 6.3.1, second to eighth paragraphs.

When the decomposition is complete, dilute the sulfuric solution with a few millilitres of water, filter through ash-free filter paper (5.6) and collect the filtrate in a one-mark 50 ml volumetric flask (5.4); rinse the round-bottom flask and the filter paper with several millilitres of water and collect the rinsings in the one-mark volumetric flask. Mix, leave to cool and fill to the mark with water. Mix the solution.

7.2.3 Blank test

Carry out a blank test, using the same decomposition procedure (7.2.1 or 7.2.2), but replacing the test portion (7.1) by 10 ml of water.

7.3 Determination

7.3.1 Test portion decomposed using the dry method

7.3.1.1 Preparation of the calibration graph

Dilute the standard iron solution (4.5) with the hydrochloric acid solution (4.4) to obtain four solutions with an iron content of 0,4 mg/l, 0,8 mg/l, 1,2 mg/l and 1,6 mg/l respectively.

Aspirate each of these solutions, in turn, into the flame of the spectrometer (5.9), at a rate such that the maximum absorbance value is obtained for the solution having an iron content of 1,6 mg/l.

1) For certain products, 10 ml of sulfuric acid may be used, in which case the concentrations of sulfuric acid used in the preparation of the calibration curve (7.3.2.1) should be modified accordingly.

Take care to keep the aspiration rate constant throughout the preparation of the calibration graph. Spray water through the burner after each measurement.

Record the corresponding values of absorbance and plot the calibration graph.

7.3.1.2 Aspiration

Aspirate into the flame of the spectrometer (5.9), at the same aspiration rate as used in 7.3.1.1, the test solution obtained in 7.2.1, and the blank test solution obtained in 7.2.3. Record the corresponding absorbances.

If the absorbance of the test solution is greater than that of the most concentrated solution used to prepare the calibration graph, dilute the test solution with the hydrochloric acid solution (4.4) and measure the absorbance.

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

7.3.2 Test portion decomposed using the wet method

7.3.2.1 Preparation of the calibration graph

Dilute the standard iron solution (4.5) with water to obtain four solutions with an iron content of 4 mg/l, 8 mg/l, 12 mg/l and 16 mg/l respectively.

Into a series of four 50 ml one-mark volumetric flasks (5.4), place 5 ml of each of these solutions (one dilution per volumetric flask). Add approximately 35 ml of water, and then 5 ml of the sulfuric acid (4.1). Mix, allow to cool and dilute to the mark with water. Mix. The iron content of these solutions is 0,4 mg/l, 0,8 mg/l, 1,2 mg/l and 1,6 mg/l respectively.

Aspirate each of these solutions, in turn, into the flame of the spectrometer (5.9), at a rate such that the maximum absorbance value is obtained for the solution having an iron content of 1,6 mg/l.

Take care to keep the aspiration rate constant throughout the preparation of the calibration graph. Spray water through the burner after each measurement.

Record the corresponding values of absorbance and plot the calibration graph.

7.3.2.2 Aspiration

Aspirate into the flame of the spectrometer (5.9), at the same aspiration rate as used in 7.3.2.1, the test solution obtained in 7.2.2 and the blank test solution obtained in 7.2.3. Record the corresponding absorbances.

If the absorbance of the test solution is greater than that of the most concentrated solution used to prepare the calibration graph, dilute the test solution with a 10 % (V/V) sulfuric acid solution and measure the absorbance.

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

8 Expression of results

8.1 Method of calculation

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

$$\frac{c_1 - c_2}{m_0} \times 50$$

where

c_1 is the iron content of the test solution, expressed in milligrams per litre, read from the calibration graph;

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

m_0 is the mass, in grams, of the test portion.

If the test solution was diluted, take the dilution factor into account in the calculation.

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

8.2 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) of the mean value.

9 Test report

The test report shall specify the method used and the results 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, together with details of any incidents which may have influenced the results.

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

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