INTERNATIONAL STANDARD

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Heat-processed foods in hermetically sealed containers — Determination of pH

Produits alimentaires en conserves — Détermination du pH

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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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Heat-processed foods in hermetically sealed containers — Determination of pH

1 Scope

This International Standard describes a potentiometric method for determining the pH of the aqueous phase of all types of food preserves.

It is particularly intended to be used to check the biological stability of food preserves.

This International Standard is applicable to the following four classes of product.

Class 1: homogeneous products with a liquid or thick texture, or products exhibiting a large liquid or thick phase which imparts a presumed uniformity of pH to the product.

Class 2: homogeneous pastes or heterogeneous products for which homogenization is necessary.

Class 3: heterogeneous products with large solid components.

Class 4: products whose liquid phase mainly consists of oil or a water/oil emulsion.

Measurements may be made at 20 °C or at 25 °C, the temperature chosen being stated in the test report.

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 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 pH of preserves: Potential difference at the measurement temperature between two electrodes immersed in the aqueous phase of the preserves or in the sample(s) of product(s) prepared in accordance with the procedure described in this International Standard. It is expressed in pH units, with an accuracy of 0,1 pH units.

4 Principle

Preparation of the test sample according to the class to which the product to be examined belongs. Measurement of the potential difference between a glass electrode and a reference electrode immersed in the test sample.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Water, complying with grade 1 of ISO 3696, distilled immediately before use so as to avoid the absorption of carbon dioxide.

5.2 Buffer solutions, for calibration of pH-meter.

Use two standard buffer solutions, with pH values known to the second decimal at the measurement temperature, and encompassing the pH of the aqueous phase obtained from the test sample.

NOTE 1 As a general rule, a buffer solution with a pH of about 4 and another one with a pH of about 7 are used.

The following buffer solutions may be used.

5.2.1 Buffer solution of pH 4,00 at 20 °C and 4,01 at 25 °C

In a 1 litre one-mark volumetric flask, dissolve in the water (5.1) 10,120 g of potassium hydrogen phthalate ($KHC_8H_4O_4$), previously dried at 120 °C to constant weight. Make up to the mark with the water at the measurement temperature and mix thoroughly.

Preserve the solution by the addition of about 2 ml of chloroform or carbon tetrachloride.

5.2.2 Buffer solution of pH 6,88 at 20 °C and 6,86 at 25 °C

In a 1 litre one-mark volumetric flask, dissolve in the water (5.1) 3,388 g of potassium dihydrogen phosphate (KH_2PO_4) and 3,533 g of disodium hydrogen phosphate (Na_2HPO_4), both products having been previously dried at 120 °C to constant weight. Make up to the mark with the water at the measurement temperature and mix thoroughly.

Preserve the solution by the addition of about 2 ml of chloroform or carbon tetrachloride.

NOTE 2 Ready-made commercial products may be used, and the manufacturer's recommendations should be followed scrupulously in this case.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 pH-meter, with an accuracy of at least 0,01 pH units, equipped with a glass electrode and a suitable reference electrode, and with temperature compensation.

NOTE 3 A combination electrode (glass electrode and reference electrode) is usually used.

6.2 Thermometer, accurate to within 1 °C.

6.3 Homogenizer, of the adapted rotary or peristaltic-type blender (stomacher) or **chopper**.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. If there is no specific International Standard dealing with sampling of the product concerned, it is recommended that the parties concerned come to an agreement on this subject.

8 Preparation of test sample

8.1 Class 1 products

This class includes homogeneous products of liquid or thick consistency (e.g. soup, dessert cream) or products exhibiting a large liquid or thick phase imparting a presumed uniformity of pH to the products so that the pH measurements can be considered as representative of the whole product (e.g. purées of French beans, peas, corn).

Mix the sample constituted by the whole product thoroughly, using a stirrer or a spatula.

8.2 Class 2 products

This class comprises homogeneous pastes (e.g. mayonnaise) or heterogeneous products (e.g. corned beef, pâté) for which homogenization is necessary each time so that the pH measurements can be considered as representative of the whole product.

Homogenize the sample constituted by the whole product by using a suitable mechanical device or chopper (6.3). Mix thoroughly if a chopper is used.

NOTE 4 In certain cases, to obtain greater fluidity, it may be necessary to add at the most 10 ml to 20 ml of distilled water to 100 g of product. The small amount of distilled water added will not change the pH of most products, but special attention should be paid to products with a low buffer capacity.

8.3 Class 3 products

This class comprises products with large solid components (e.g. cassoulet, sauerkraut).

Separate each category of major components (e.g. vegetables, meat) from the sample constituted by the whole product.

If necessary, homogenize each category separately, as described in 8.2.

8.4 Class 4 products

This class comprises products whose liquid phase consists essentially of an oil or a water/oil emulsion (e.g. sardines).

After opening the container, transfer the liquid phase of the laboratory sample to a separating funnel. Collect the aqueous phase and eliminate the oil phase.

In applicable cases, combine the aqueous phase with the solid part of the laboratory sample. Proceed as described in 8.2 after adding, if necessary, at most 10 ml to 20 ml of water (5.1) to 100 g of solid part.

9 Procedure

9.1 Calibration of pH-meter

Adjust the temperature of the buffer solution (5.2) to the chosen measurement temperature (20 °C or 25 °C) and calibrate the pH-meter according to the manufacturer's instructions.

NOTES

5 For tests in series, verify the calibration of the pH-meter with one or two buffer solutions at least every 30 min.

6 In certain cases it is necessary to check the electrodes more frequently using the reference solutions (e.g. measurement of very fat products, some pickling brines). As soon as any irregularity is detected (deviation of at least 0,1 pH units caused by clogging), clean the electrodes (see 9.4).

9.2 Test portion

9.2.1 Class 1 products

Take measurements directly on the test sample considered as homogeneous, in its container.

In the case of large containers (more than 5 litres), carry out the measurements on a representative test sample of at least 200 g.

9.2.2 Class 2 products

Take measurements on the homogenized test sample.

9.2.3 Class 3 products

Carry out measurements on each category of major component of the test sample or, for large containers, on a quantity of the test sample representative of each major component, after homogenization, if required.

For each component and according to its own properties, make a separate determination

— in the aqueous or liquid phase, and

— on the solid components (e.g. pieces of meat, sausages).

9.2.4 Class 4 products

Take measurements

— either in the aqueous phase, or

— in the homogenate (aqueous phase and solid product).

9.3 Determination

One determination is sufficient for class 1 products and for the aqueous (or liquid) phase of class 3 and 4 products.

Make three determinations on the same test portion for class 2 and 4 homogenized products, and on each solid component for class 3 products.

Insert the thermometer (6.2) into the test portion (class 1 and 2 products) or into the aqueous phase (class 3 and 4 products). Read the measurement temperature, then insert the electrodes into the test portion.

Make the determination using the appropriate procedure for the pH-meter employed. When the reading becomes constant, read the pH directly to the nearest 0,1 pH unit on the scale of the instrument.

9.4 Cleaning the electrodes

Clean the electrodes by rinsing them successively with acetone at ambient temperature and with water at 30 °C to 35 °C. Wipe them thoroughly with clean paper.

To take account of clogging and ageing of the electrodes, clean them more completely at regular intervals, according to the manufacturer's instructions.

NOTE 7 The electrodes may also be cleaned, if necessary, by using a suitable enzymatic solution. In the case of bacterial contamination, the electrodes may also be cleaned with 70 % ethanol.

10 Expression of results

10.1 Class 1 products and aqueous liquid phases of class 3 and 4 products

Record the pH value, to the nearest 0,1 pH unit, and the measurement temperature.

10.2 Homogenized class 2 and 4 products and solid components of class 3 products

Take the arithmetic mean of the three values obtained for each test sample or for each solid component (class 3 products) if the repeatability conditions are satisfied. If not, repeat the test.

Express the mean pH for each component to the nearest 0,1 pH unit.

11 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a

short interval of time, should not exceed 0,15 pH units.

12 Test report

The test report shall specify

— the method in accordance with which sampling was carried out, if known,

— the method used,

— the test result(s) obtained, and

— if the repeatability has been checked, the final quoted result obtained.

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 test result(s).

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

For heterogeneous products with large solid components, the different components and the point where the pH value was measured shall be stated.