
International Standard



6632

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Fruits, vegetables and derived products — Determination of volatile acidity

Fruits, légumes et produits dérivés — Détermination de l'acidité volatile

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Foreword

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International Standard ISO 6632 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in May 1980.

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

Australia	Ireland	Portugal
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Canada	Korea, Dem. P. Rep. of	Spain
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

Fruits, vegetables and derived products — Determination of volatile acidity

1 Scope and field of application

This International Standard specifies a method for the determination of volatile acidity in fruits, vegetables and derived products.

The method is applicable to all fresh products and to products preserved without chemical preservatives, as well as to products to which sulphur dioxide has been added with or without one of the following preservatives : sorbic acid, benzoic acid, formic acid.

2 Definition

2.1 volatile acidity : All lower molecular weight fatty acids, such as acetic acid and propionic acid, in free or combined form, with the exception of formic acid.

The volatile acidity, determined by the method specified in this International Standard, is expressed either in milliequivalents per 100 ml or per 100 g of product, or in grams of acetic acid per 100 ml or per 100 g of product.

3 Principle

Acidification of a test portion with tartaric acid, entrainment of the volatile acids by steam distillation, and titration of the distillate with standard volumetric sodium hydroxide solution in the presence of phenolphthalein as indicator. If appropriate, subtraction of the volatile acidity due to added volatile acid antiseptic (preservative) compounds from the volatile acidity thus determined.

4 Reagents

All the reagents shall be of recognized analytical purity. The water used shall be distilled water or water of equivalent purity, free from carbon dioxide.

4.1 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}^{(1)}$.

Prepare just before use and check the concentration by titration immediately before use.

4.2 Phenolphthalein, 10 g/l solution in 95 % (V/V) ethanol.

4.3 Tartaric acid, crystallized.

4.4 Lime water, diluted 1 + 4.

Dilute one volume of the saturated calcium hydroxide solution (4.5) with four volumes of water. Allow the mixture to stand until calcium carbonate is precipitated and decant the limpid solution which shall be alkaline to the phenolphthalein solution (4.2).

This solution is intended for use in the steam generator (5.2.1).

4.5 Calcium hydroxide, limpid saturated solution.

5 Apparatus

Usual laboratory apparatus, and in particular :

5.1 Mechanical mill.

5.2 Apparatus for extrainment by steam distillation (see the figure), comprising the following elements :

5.2.1 Steam generator, suitable for the production of steam free from carbon dioxide, made of heat-resistant glass or metal, and of capacity about 1 500 ml.

5.2.2 Bubbler, comprising a glass tube, of diameter 30 mm and 270 mm long, the lower part of which is sealed and enlarged to form a sphere of diameter 60 mm in which the test portion is placed. The bubbler shall be placed on a metal disc having an orifice of diameter 40 mm, into which the bottom of the bubbler is fitted.

5.2.3 Fractionating column, comprising a glass tube, of diameter 20 mm and 500 mm long, inside which is a spiral-shaped stainless steel net, No. 100, with a lead of 15 mm.

Any other device having the same fractionating efficiency may be used (see the note to 5.2.4).

1) Hitherto expressed as "0,1 N standard volumetric solution".

5.2.4 Condenser (West type), diameter 16 mm and nominally 400 mm long, placed vertically to ensure condensation of the vapour and complete cooling of the distillate.

NOTE — Apparatus other than that described above may be used, provided it complies with the following requirements :

- a) under normal distillation conditions, at least 99,5 % of a known amount of acetic acid added to the sample shall be found in 250 ml of distillate. To check this, use 20 ml of acetic acid solution of concentration, $c(\text{CH}_3\text{COOH}) = 0,1 \text{ mol/l}^{(1)}$;
- b) under the same conditions of distillation, no more than 5 parts per thousand of a known amount of lactic acid added to the sample shall be found in the distillate. To check this, use 20 ml of lactic acid solution of concentration, $c(\text{C}_2\text{H}_5\text{OOCOH}) = 1 \text{ mol/l}^{(2)}$;
- c) the steam produced by the steam generator shall be free from carbon dioxide. Check this by adding two drops of the phenolphthalein solution (4.2) and 0,1 ml of the sodium hydroxide solution (4.1) to 250 ml of distillate, and verifying that a pink colour (stable for at least 10 s) appears.

5.3 Conical flask, of capacity 500 ml.

5.4 Graduated pipettes, of capacity 20 ml, complying with the requirements of ISO/R 835.

5.5 Burette, of capacity 25 ml, graduated at 0,1 ml intervals, complying with the requirements of ISO/R 385, class B.

5.6 Analytical balance.

6 Procedure (see clause 8 for the particular case of products containing volatile acid preservatives)

6.1 Preparation of test sample

6.1.1 Liquid products and those in which the liquid can be easily separated (juices, syrups, liquid from stewed fruit compot, brines, etc.)

Thoroughly mix the sample. If the sample contains solid particles in suspension, separate the liquid by filtration through a fluted filter paper. If the sample is in the course of fermentation, or if it contains carbon dioxide, transfer, by means of a pipette, 50 to 60 ml of the product into a 500 ml flask. Remove the carbon dioxide by shaking under reduced pressure for 2 to 3 min. To avoid formation of foam, a small amount of antifoaming agent may be added, for example about 0,2 g of tannic acid, directly to the weighed sample.

6.1.2 Viscous or solid products (marmalade, jams, jellies, concentrated juices, dried fruit and vegetables, etc.)

Remove the seeds and hard seed-cavity walls if necessary, and homogenize the sample in the mechanical mill (5.1).

6.1.3 Frozen or deep-frozen products

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

6.2 Test portion

6.2.1 Liquid products

Transfer, by means of a pipette (5.4), 20 ml of the test sample (6.1.1) to the bubbler (5.2.2). In the case of samples which have strong volatile acidity, a smaller amount may be used and the total volume can be made up to 20 ml by addition of the required amount of water.

6.2.2 Viscous or solid or frozen products

In a beaker, weigh, to the nearest 0,01 g, about 10 g of the test sample (6.1.2 or 6.1.3). Transfer the test portion to the bubbler (5.2.2) with the minimum quantity of water necessary for entrainment of the whole test portion and to render the mixture sufficiently fluid.

6.3 Entrainment of volatile acids

Fill the steam generator (5.2.1) to two-thirds of its volume with the lime water (4.4). Add 0,5 g of the tartaric acid (4.3) to the test portion (6.2) contained in the bubbler (5.2.2). Connect the latter to the steam generator (5.2.1) and to the fractionating column (5.2.3) and condenser (5.2.4). Heat the steam generator and the bubbler simultaneously on burners. If the initial volume of the contents of the bubbler is more than 20 ml, regulate the heating so as to reduce the volume to about 20 ml while the steam is slowly introduced.³⁾ Reduce the rate of heating of the bubbler to maintain the volume of the contents constant (about 20 ml) for the duration of the distillation during which the output of steam from the generator (5.2.1) is at its maximum. The distillation shall last for 10 to 15 min.

Collect the distillate in the conical flask (5.3), continuing the distillation until about 250 ml of distillate has been collected. The volume of distillate shall be at least 12 times that of the test portion (6.2).

6.4 Titration of volatile acids

Add two drops of the phenolphthalein solution (4.2) to the distillate and titrate with the sodium hydroxide solution (4.1) until a light pink colour, persisting for at least 15 s, appears.

6.5 Number of determinations

Carry out two determinations on the same test sample (6.2).

1) Hitherto expressed as "0,1 N solution".

2) Hitherto expressed as "1 N solution".

3) Using 20 ml of liquid test portion, the volume is not reduced, as the steam is introduced into the flask at the maximum rate at the start of the distillation.