International Standard 6638/2

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Fruit and vegetable products — Determination of formic acid content — Part 2 : Titrimetric method

Produits dérivés des fruits et légumes — Détermination de la teneur en acide formique — Partie 2 : Méthode titrimétrique

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Foreword

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Fruit and vegetable products — Determination of formic acid content — Part 2 : Titrimetric method

1 Scope and field of application

This part of ISO 6638 specifies a titrimetric method for the determination of the formic acid content of fruit and vegetable products.

A gravimetric method is specified in ISO 6638/1.

2 Principle

Quantitative entrainment by steam of the formic acid present in a test portion. Determination of the amount of iodine equivalent to the amount of bromine consumed by the acid in the distillate, by titration with sodium thiosulfate standard volumetric solution.

3 Reagents

All reagents shall be of recognized analytical grade, and in particular shall be free from lead. The water used shall be distilled water or water of at least equivalent purity.

3.1 Tartaric acid, crystalline.

3.2 Sodium acetate, 10 % (*m*/*m*) solution.

3.3 Potassium iodide, 10 % (*m*/*m*) solution.

3.4 Bromoacetic acid.

Dilute 8 g of bromine with glacial acetic acid to the mark in a 1 000 ml one-mark volumetric flask.

3.5 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3)$ = 0,1 mol/l.[1)]

3.6 Starch indicator, 1 % (*m*/*m*) solution.

4 Apparatus

Usual laboratory equipment and in particular

4.1 Analytical balance.

4.2 Distillation apparatus as shown in the figure, or equivalent apparatus, comprising

4.2.1 Steam generator, made of metal or glass, of capacity 5 litres.

4.2.2 Condenser, of length 50 cm.

4.2.3 Flask, of capacity 500 ml.

4.2.4 Volumetric flask, of capacity 1 000 ml.

4.3 Conical flask, of capacity 500 ml, with a ground glass stopper.

4.4 Burette.

4.5 Mixer and/or **mechanical grinder.**

5 Procedure

5.1 Preparation of the test sample

Thoroughly mix the laboratory sample. If necessary, first remove stones and hard seed-cavity walls and pass through a mechanical grinder.

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.

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

5.2 Test portion

5.2.1 Liquid products

Take, by means of a pipette, 50 ml of the test sample (5.1) and transfer to the flask (4.2.3).

NOTE — The test portion may also be taken by mass, by weighing, to the nearest 0,01 g, approximately 50 g of the test sample.

5.2.2 Pasty or solid products

Weigh, to the nearest 0,01 g, 50 g of the test sample (5.1) and transfer to the flask (4.2.3) with the minimum volume of water necessary to entrain the whole of the test portion and to make the mixture sufficiently fluid (50 ml of water is usually sufficient).

NOTE — In certain cases, it is necessary to leave the test portion to soak in the water for 1 to 2 h.

5.3 Distillation

Add approximately 0,5 g of the tartaric acid (3.1) to the contents of the flask (4.2.3). Connect the flask to the steam generator (4.2.1) and the condenser (4.2.2) and heat the flask and the steam generator simultaneously. Carry out the distillation ensuring that the volume of the contents of the flask (4.2.3) remains constant to within 5 ml. Collect almost 1 000 ml of distillate in the 1 000 ml volumetric flask (4.2.4), then dilute to the mark with water and mix thoroughly.

5.4 Determination

Take 200 ml of the distillate and transfer to the conical flask (4.3). Add 5 ml of the sodium acetate solution (3.2) and 10 to 25 ml of the bromoacetic acid (3.4). Stopper the flask tightly and allow to stand for 30 min, then add 5 ml of the potassium iodide solution (3.3). Titrate the liberated iodine with the sodium thiosulfate standard volumetric solution (3.5) until the yellow colour due to iodine has almost disappeared, then add 1 ml of the starch indicator solution (3.6) and continue the titration until the blue colour just disappears after very vigorous shaking.

5.5 Blank test

Carry out a blank test in parallel with the determination, replacing the 200 ml of distillate by 200 ml of water.

5.6 Number of determinations

Carry out two determinations on the same test sample (5.1).

6 Expression of results

6.1 Method of calculation and formulae

The formic acid content, expressed in grams per 100 ml or 100 g of sample, is equal to

a) in the case of test portions taken by volume

$$\frac{(V_2 - V_1) \times c \times 0{,}002\,3 \times 5 \times 100}{V_0}$$

b) in the case of test portions taken by mass

$$\frac{(V_2 - V_1) \times c \times 0{,}002\,3 \times 5 \times 100}{m}$$

where

V_0 is the volume, in millilitres, of the test portion;

V_1 is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution (3.5) used for the determination;

V_2 is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution (3.5) used for the blank test;

c is the exact concentration, in moles per litre, of the sodium thiosulfate standard volumetric solution used;

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

Take as the result the arithmetic mean of the values obtained in the two determinations (5.6), provided that the requirement for repeatability (see 6.2) is satisfied.

6.2 Repeatability

The difference between the values obtained in the two determinations (5.6), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 2 % of the mean.

7 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating details not specified in this part of ISO 6638, 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.

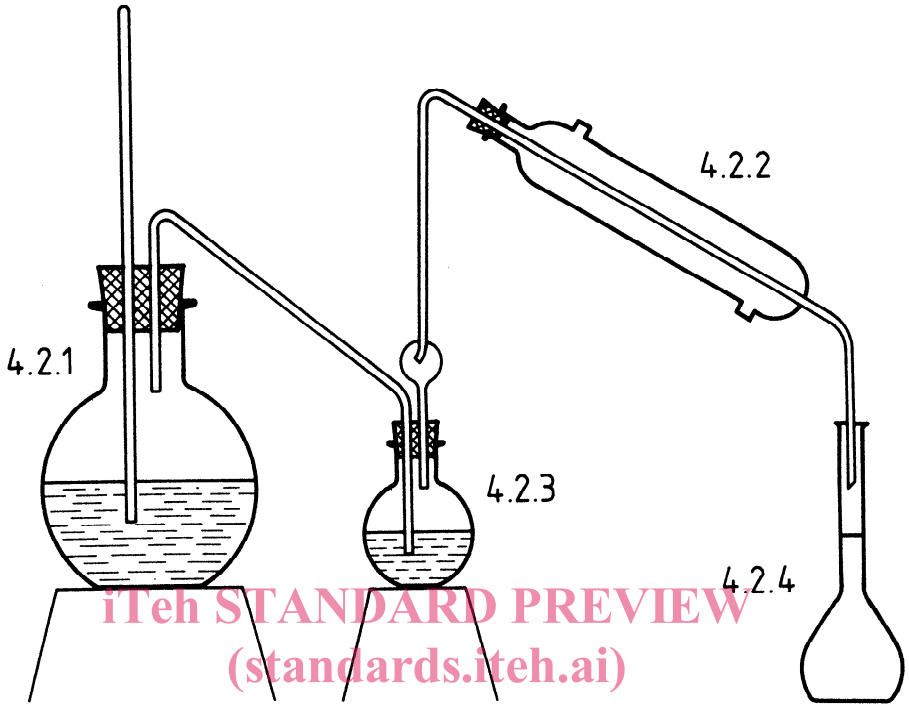

Figure — Distillation apparatus (4.2)

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