International Standard ISO 5523

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Liquid fruit and vegetable products — Determination of sulphur dioxide content (Routine method)

Produits liquides dérivés des fruits et légumes — Détermination de la teneur en dioxyde de soufre (Méthode pratique)

First edition — 1981-08-01

UDC 634/635 : 543.845

Descriptors : *fruit and vegetable products, beverages, chemical analysis, determination of content, sulphur dioxide.*

Ref. No. ISO 5523-1981 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 5523 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in July 1979.

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

Australia	Germany, F. R.	Netherlands
Austria	Hungary	Poland
Brazil	India	Portugal
Bulgaria	Indonesia	Romania
Canada	Israel	South Africa, Rep. of
Chile	Italy	Spain
Cyprus	Kenya	Thailand
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Libyan Arab Jamahiriya	Yugoslavia
Ethiopia	Malaysia	
France	Mexico	

The member body of the following country expressed disapproval of the document on technical grounds :

New Zealand

Printed in Switzerland

Liquid fruit and vegetable products — Determination of sulphur dioxide content (Routine method)

0 Introduction

This method, which was developed for the determination of the sulphur dioxide content of wines, has been found to be suitable for liquid fruit and vegetable products, and can be used as a rapid method.

In the case of more precise analyses, or in cases of dispute, the method specified in ISO 5522 shall be used.

1 Scope and field of application

This International Standard specifies a routine method for the determination of the sulphur dioxide content of liquid fruit and vegetable products.

2 References

ISO/R 385, *Burettes.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1773, *Laboratory glassware — Boiling flasks (narrow-necked).*

ISO 5522, *Fruits, vegetables and derived products — Determination of total sulphur dioxide content.*

3 Definitions

3.1 free sulphur dioxide : Sulphur dioxide as such or as inorganic combinations of H_2SO_3, HSO_3^- and SO_3^{2-}.

3.2 bound sulphur dioxide content : The difference between *the total sulphur dioxide content and the free sulphur* dioxide content.

4 Principle

4.1 Free sulphur dioxide

Direct iodometric titration of the product at a pH between 0,7 and 1, followed by a blank titration of the same product, freed *from sulphur dioxide by boiling under reflux or by binding its* free sulphur dioxide content with an excess of acetaldehyde (ethanal) or propionaldehyde (propanal).

4.2 Bound sulphur dioxide

After titration of the free sulphur dioxide, rendering of the product alkaline and titration of the sulphur dioxide liberated by this hydrolysis with iodine in an acid medium.

A second iodometric titration after a second alkali hydrolysis *permits titration of the sulphur dioxide which may have recombined*, after the first hydrolysis, with any acetaldehyde present in the product.

5 Reagents

All reagents shall be of recognized analytical quality and in particular shall not contain impurities which could be determined as sulphur dioxide. The water used shall be distilled water or *water of at least equivalent purity, recently boiled.*

5.1 Sodium hydroxide, approximately 4 mol/l solution.[1)]

Dissolve 160 g of sodium hydroxide in water and dilute to 1 000 ml.

5.2 Sulphuric acid, 10 % (*V*/*V*) solution (180 g of H_2SO_4 per litre).

5.3 Starch, 5 g/l solution containing 200 g of sodium chloride (as preservative) per litre.

*Maintain the solution at boiling point for 10 min during prepara*tion.

1) *Hitherto expressed as "approximately 4 N solution".*

5.4 Iodine, standard volumetric solution, $c(1/2\ I_2) = 0{,}05$ mol/l.[1)]

5.5 Sodium thiosulphate, standard volumetric solution, $c(Na_2S_2O_3.5H_2O) = 0{,}005$ mol/l.[2)]

5.6 Acetaldehyde, 7 g/l solution; or

5.7 Propionaldehyde, 10 g/l solution.

6 Apparatus

Usual laboratory apparatus, not otherwise specified, and in particular :

6.1 Conical boiling flasks, of capacity 500 ml, complying with the requirements of ISO 1773.

6.2 One-mark pipettes, of capacity 5 — 10 — 20 and 50 ml, complying with the requirements of ISO 648.

6.3 Burettes, of capacity 50 ml, complying with the requirements of ISO/R 385, class A.

6.4 Device for illuminating the bottom of the boiling flask with a vertical, yellow light beam obtained from a sodium vapour lamp, or from an ordinary lamp, the white light of which is filtered through a potassium chromate solution.

6.5 Distillation apparatus, consisting of a 500 ml boiling flask and a reflux condenser.

7 Procedure

7.1 Preparation of test sample

Render the sample homogeneous.

7.2 Test portion

Transfer, by means of a pipette (6.2), 50 ml of the test sample to a 500 ml boiling flask (6.1).

7.3 Determination

7.3.1 Add 3 ml of the sulphuric acid solution (5.2) and 5 ml of the starch solution (5.3) to the contents of the boiling flask. Immediately titrate with the iodine solution (5.4) (see 9.1) until the mixture turns blue (see 9.2). Decolourize with the minimum volume necessary of the sodium thiosulphate solution (5.5). Subtract one tenth of the volume of this solution from the volume of the iodine solution used.

7.3.2 Add 8 ml of the sodium hydroxide solution (5.1). Swirl once only and allow to stand for 5 min. With a single movement and whilst swirling the flask vigorously, pour in 10 ml of the sulphuric acid solution (5.2) from a beaker. Immediately titrate with the iodine solution (5.4) (see 9.1) until the mixture turns blue (see 9.2). Decolourize with the minimum volume necessary of the sodium thiosulphate solution (5.5). Subtract one tenth of the volume of this solution from the volume of the iodine solution used.

7.3.3 Add 20 ml of the sodium hydroxide solution (5.1). Swirl once only and allow to stand for 5 min.

Dilute with 200 ml of water at a temperature close to 0 °C.

With a single movement and whilst swirling the flask vigorously, pour in 30 ml of the sulphuric acid solution (5.2) from a beaker. Immediately titrate the sulphur dioxide thus liberated with the iodine solution (5.4) (see 9.1) until the mixture turns blue (see 9.2). Decolourize with the minimum volume necessary of the sodium thiosulphate solution (5.5). Subtract one tenth of the volume of this solution from the volume of the iodine solution used.

7.3.4 Blank titration

Some substances (especially ascorbic acid), present in a natural state or added to a product, are oxidized by iodine in acid medium. This leads to a false value for the iodometric titration and, in such cases, the determinations shall be carried out on a test sample desulphurized by one of the following procedures.

7.3.4.1 Desulphurization by boiling under reflux

Place 100 ml of the product to be analysed in the 500 ml boiling flask of the distillation apparatus (6.5) fitted with the reflux condenser, and boil vigorously for at least 30 min.

The condenser shall be sufficiently short and wide to enable the sulphur dioxide to escape by diffusion at a sufficient rate. Allow to cool before removing the condenser. Transfer 50 ml of the liquid thus freed from sulphur dioxide into a 500 ml boiling flask (6.1).

7.3.4.2 Desulphurization by combination with an aldehyde

Place 50 ml of the product to be analysed in a 500 ml boiling flask (6.1) and add 5 ml of the acetaldehyde solution (5.6) or 5 ml of the propionaldehyde solution (5.7). Stopper the flask, and allow to stand for at least 30 min.

7.3.4.3 Titration

Proceed as specified in 7.3.1 using 50 ml of the desulphurized product (7.3.4.1 or 7.3.4.2).

1) Hitherto expressed as "0,05 N standard volumetric solution".

2) Hitherto expressed as "0,005 N standard volumetric solution".

7.4 Number of determinations

Carry out two determinations on the same test sample (7.1).

8 Expression of results

8.1 Method of calculation and formulae

The free sulphur dioxide content, expressed in milligrams per litre of product, is given by the formula[1)]

$$1{,}6 \times \frac{1\,000}{50}(V_0 - V_3) = 32\,(V_0 - V_3)$$

The total sulphur dioxide content, expressed in milligrams per litre of product, is given by the formula[1)]

$$32\,(V_0 + V_1 + V_2 - V_3)$$

The bound sulphur dioxide content, expressed in milligrams per litre of product, is given by the formula[1)]

$$32\,(V_1 + V_2)$$

where

V_0 is the corrected volume, in millilitres, of iodine solution used in 7.3.1;

V_1 is the corrected volume, in millilitres, of iodine solution used in 7.3.2;

V_2 is the corrected volume, in millilitres, of iodine solution used in 7.3.3;

V_3 is the corrected volume, in millilitres, of iodine solution used in 7.3.4.3;

1,6 is the mass, in milligrams, of sulphur dioxide corresponding to 1 ml of 0,05 mol/l iodine solution.

NOTE — If the product contains acetaldehyde, V_2 represents, in general, 5 to 15 % of V_1.

8.2 Repeatability

To be added later.

9 Notes on procedure

9.1 For products having low sulphur dioxide contents, it is preferable to use a more dilute iodine solution, for example a solution of concentration $c(1/2\ I_2) = 0{,}02$ mol/l.

9.2 For highly coloured products, it is more advantageous to use the device producing a yellow light beam (6.4) for illuminating the bottom of the flask containing the test portion.

Operate in a dark room and observe the transparency of the product, which becomes opaque when the starch changes colour.

10 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 International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

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

1) If iodine solution of concentration $c(1/2\ I_2) = 0{,}02$ mol/l was used, replace the coefficient 32 by 12,8.