
International Standard



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Citrus fruits and derived products — Determination of essential oils content (Reference method)

Agrumes et produits dérivés — Détermination de la teneur en huiles essentielles (Méthode de référence)

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Foreword

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International Standard ISO 1955 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in August 1980.

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

Australia	India	Romania
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Canada	Kenya	Spain
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The member body of the following country expressed disapproval of the document on technical grounds :

USA

Citrus fruits and derived products — Determination of essential oils content (Reference method)

1 Scope and field of application

This International Standard specifies the reference method for the determination of the total essential oils content of citrus fruits and their derived products (whole fruits, fruits cut into small pieces, skins, juices, concentrates, beverage bases, sweetened products etc.).

2 Principle

Boiling of a test portion, diluted or undiluted, and steam entrainment of the essential oils which are collected in a graduated tube; after cooling, direct reading from this tube of the volume of essential oils separated from the distillate.

3 Apparatus

3.1 Apparatus¹⁾, for entrainment and recovery of essential oils, comprising a condenser and a trap terminating in a tube, of diameter 8 to 10 mm, graduated in 0,05 ml divisions, having a capacity of 4 ml.

3.2 Flask, of capacity 3 litres, suitable for connection to the apparatus (3.1).

3.3 Graduated measuring cylinders, of capacity 2 litres.

3.4 Boiling aid.

4 Procedure

4.1 Preparation of the test sample

NOTE — Sufficient test sample is required to provide test portions of 2 litres.

4.1.1 Products having low essential oils content (less than 0,1 ml per 100 ml or per 100 g of product)

Citrus fruit juices, for example, shall be used after simply stirring; thick, pulpy or syrupy products shall be thoroughly mixed with an equal mass of water.

4.1.2 Products rich in essential oils²⁾

Suitably dilute the test sample to obtain an essential oils content of less than 0,1 ml per 100 ml or per 100 g of product.

4.1.3 Products very rich in essential oils (used as bases for carbonated drinks)²⁾

Mix thoroughly by diluting with water in the proportion indicated in 4.1.2, operating in a high-speed mechanical mill in order to avoid separation.

4.1.4 Whole fruits, fruits cut into small pieces, and skins

Grind the sample finely and proceed as specified in 4.1.3.

4.2 Preparation of apparatus (3.1)

Start the circulation of water through the condenser and, if possible, moisten the inside of the condenser with a wetting agent, for example sodium secondary alkylsulphate.

Fill the trap with distilled water. If possible, immerse the trap itself in a large beaker of cold water during the test.

4.3 Test portion²⁾

Transfer 2 litres of the test sample (4.1), measured by means of the graduated measuring cylinder (3.3) and corresponding to a volume V_0 or a mass m of the product before dilution, to the flask (3.2). If necessary, add the boiling aid (3.4).

4.4 Determination

Connect the flask to the apparatus (3.1) and start heating. When the liquid begins to boil, reduce the rate of heating so that only about one drop of distillate falls from the condenser per second.

1) An apparatus of the modified Clevenger type is illustrated, for information only, in the annex.

2) For products which are rich, or very rich, in essential oils (4.1.2 and 4.1.3), it is possible to reduce the volume of the test portion to 1 litre, modifying the capacity of the apparatus and the degree of dilution accordingly.

Allow to boil for 1 to 3 h; the essential oils accumulate in the trap. When the volume of essential oils has not increased for 15 to 30 min, stop heating and allow the trap to cool.

If possible, rinse the condenser with water from a wash-bottle, then bring the lower boundary of the essential oils phase to the "0" mark of the graduated tube, by running out water through the tap. Allow the trap to drain for at least 5 min, then read the volume, to the nearest half-division, indicated by the upper meniscus.

4.5 Number of determinations

Carry out two determinations on the same test sample.

5 Expression of results

5.1 Method of calculation and formula

5.1.1 Samples taken by volume

The essential oils content, expressed as a percentage by volume of product, is given by the formula

$$\frac{V_1}{V_0} \times 100$$

where

V_0 is the volume, in millilitres, of product present in the test portion (4.3);

V_1 is the volume, in millilitres, of essential oils, measured in 4.4.

5.1.2 Samples taken by mass

The essential oils content, expressed in millilitres per 100 g of product, is given by the formula

$$\frac{V_1}{m} \times 100$$

where

m is the mass, in grams, of product present in the test portion (4.3);

V_1 has the same meaning as in 5.1.1.

5.1.3 Result

Take as the result the arithmetic mean of the values obtained in the two determinations (4.5), provided that the requirement for repeatability (see 5.2) is fulfilled.

5.2 Repeatability

The difference between the values obtained in the two determinations (4.5), carried out simultaneously or in rapid succession by the same analyst, shall not exceed 5 % (relative) of the mean.

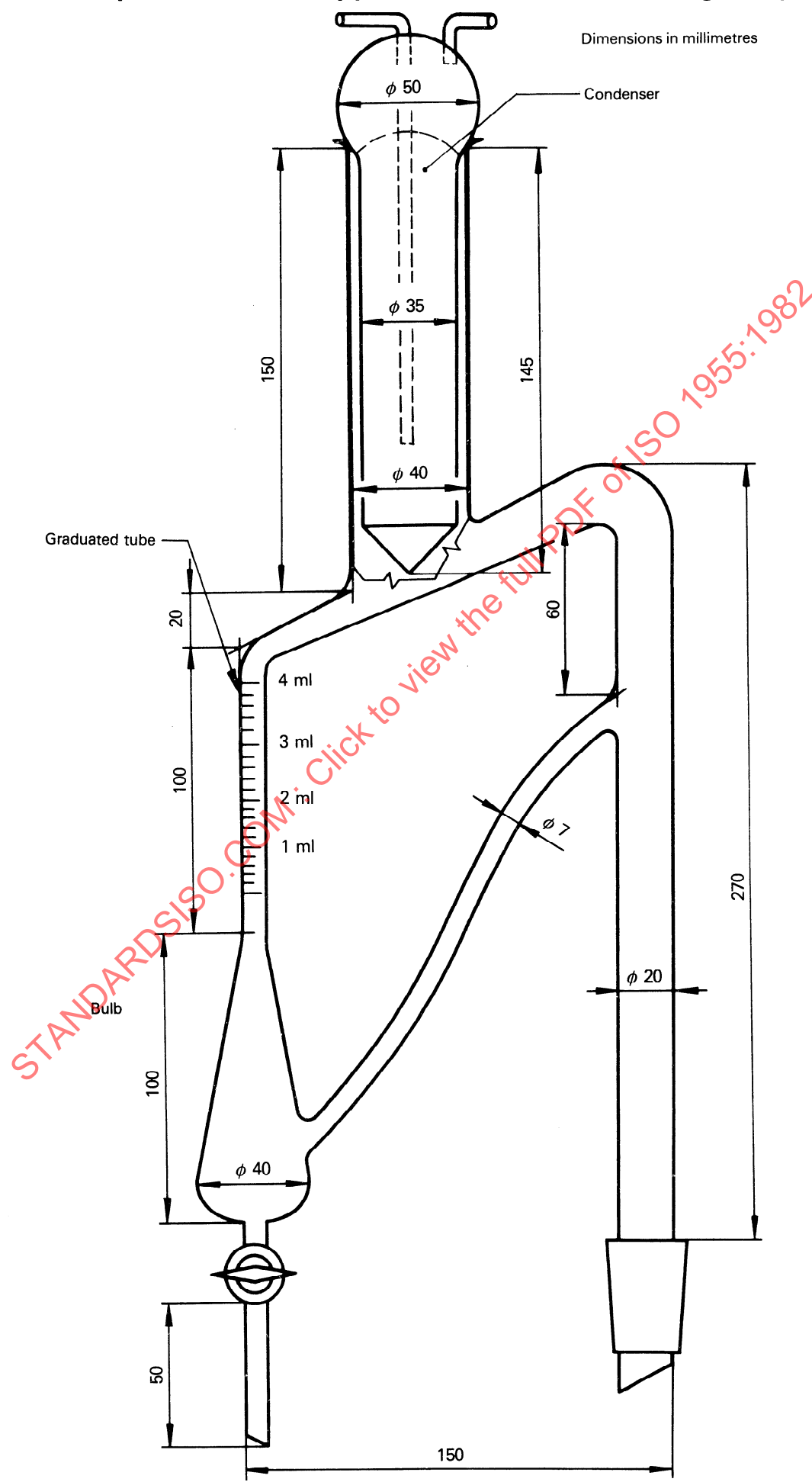
6 Test report

The test report shall show the method used and the result 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 result.

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

Annex

Example of suitable apparatus (modified Clevenger type)



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