INTERNATIONAL STANDARD

ISO 6571

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Spices, condiments and herbs — Determination of volatile oil content (hydrodistillation method)

Épices, aromates et herbes — Détermination de la teneur en huiles essentielles (méthode par hydrodistillation)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6571 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 7, *Spices, culinary herbs and condiments*.

This second edition cancels and replaces the first edition (ISO 6571:1984) of which it constitutes a technical revision.

Spices, condiments and herbs — Determination of volatile oil content (hydrodistillation method)

1 Scope

This International Standard specifies a method for the determination of the volatile oil content of spices, condiments and herbs.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 939, *Spices and condiments — Determination of moisture content — Entrainment method*

ISO 2825, *Spices and condiments — Preparation of a ground sample for analysis*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
volatile oil content
all the substances entrained by steam under the conditions specified in this International Standard

NOTE The volatile oil content is expressed in millilitres per 100 g of dry product.

4 Principle

An aqueous suspension of the product is distilled. The distillate is collected in a graduated tube containing a measured volume of xylene to fix the volatile oil. The organic and aqueous phases are then allowed to separate and the total volume of the organic phase read. The volatile oil content is calculated after deducting the volume of xylene.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of at least equivalent purity.

5.1 Xylene.

5.2 Cleaning solutions.

5.2.1 Acetone (for fatty residues).

5.2.2 Liquid detergent (used at the concentration recommended by the manufacturer) or a **solution of sulfuric acid and potassium dichromate** (see the warning) prepared by slowly adding, while stirring continuously, one volume of concentrated sulfuric acid to one volume of saturated potassium dichromate solution and by passing the mixture, after cooling, through a fritted glass filter.

WARNING — Avoid any contact of this solution with the skin and mucous membranes.

6 Apparatus

Usual laboratory equipment, and in particular the following.

6.1 Distillation apparatus, made of strong glass having a low coefficient of thermal expansion[1)].

The apparatus comprises the following components connected by ground glass joints.

6.1.1 Round-bottom flask, with a ground neck, of capacity 500 ml or 1 000 ml, according to the product concerned (see Annex A).

6.1.2 Condenser system, comprising the following components joined together (see Figure 1):

a) a vertical tube (AC), the base of which has a ground joint to fit the flask (6.1.1);

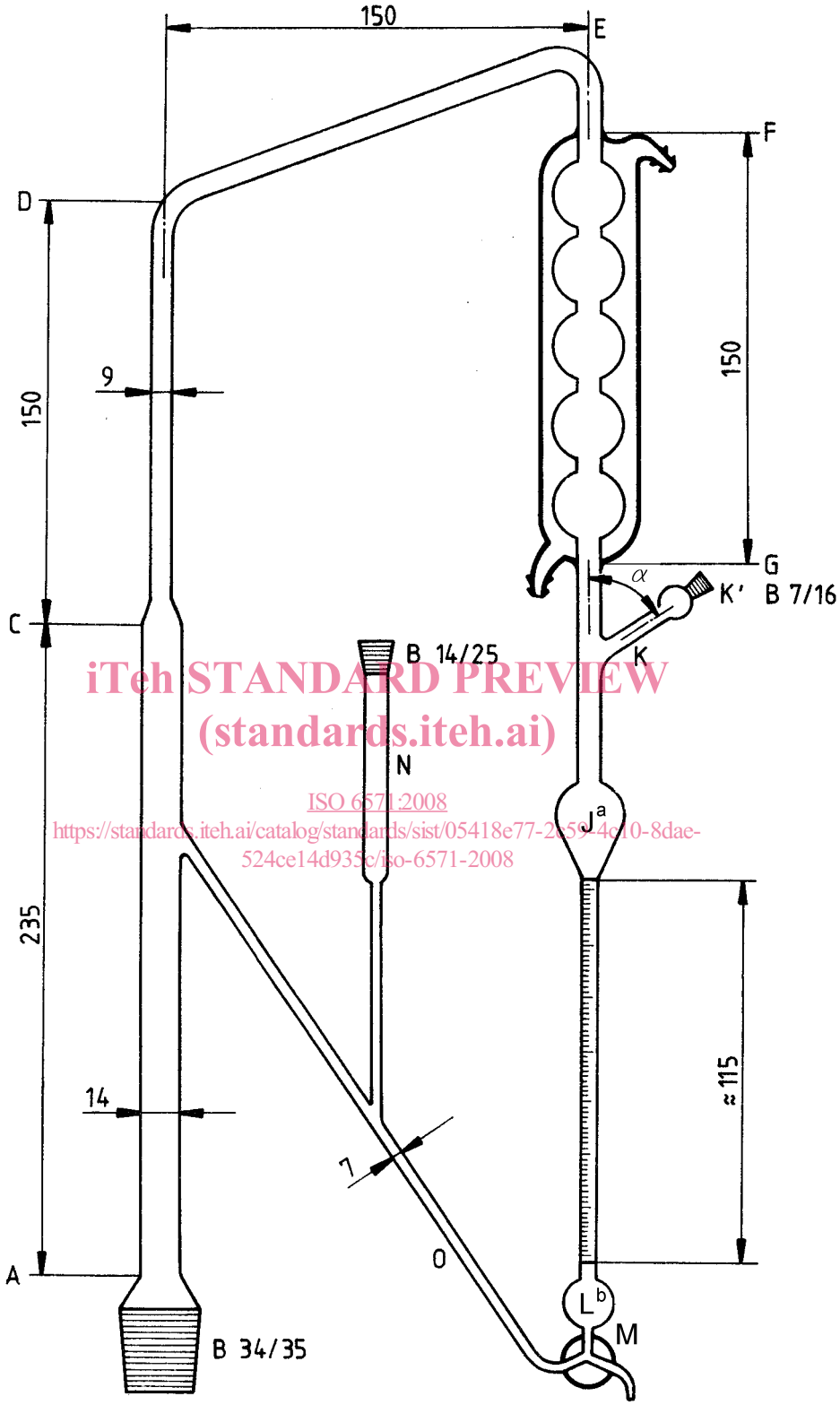
b) a bent tube (CDE);

c) a vertical bulb condenser (FG);

d) an assembly consisting of a tube with a side-arm (K) provided with a ground stopper (K′), a pear-shaped enlargement (J), a tube graduated in divisions of 0,05 ml (JL), a spherical enlargement (L) and a three-way tap (M) connected to the vertical tube (AC) by an inclined tube (O) provided with a safety tube (N), if necessary topped by the steam trap (6.1.3).

1) This apparatus corresponds to the type described in the *European Pharmacopoeia* (V 5.89), 2.8.12.

Dimensions in millimetres



Key

A, C, D, E, F, G, J, K, K′, L, M, N, O	see 6.1.2
B	see Figure 2
a	Capacity 5 ml.
b	Capacity 3 ml.
α	35°

Figure 1 — Condenser system

6.1.3 Steam trap (see Figure 2) which can be connected to the side-arm (K) or to the safety tube (N) (see 6.1.2).

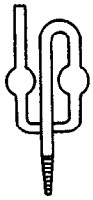


Figure 2 — Steam trap

6.2 Filter paper, of diameter 110 mm.

6.3 Pipette, of capacity 1 ml.

6.4 Heating device.

The method of heating should be such as to avoid overheating of the flask (6.1.1). A device for regulating the temperature is recommended.

6.5 Anti-bumping granules or **glass beads**.

6.6 Measuring cylinder, of capacity 500 ml.

6.7 Analytical balance.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 948[1].

8 Procedure

NOTE It is intended to specify the test parameters in the International Standard specifying requirements for each spice or condiment. In the meantime, these parameters are given in Annex A.

8.1 Preparation of the apparatus

Carefully clean the condenser system (6.1.2). Tightly fix the glass stopper (K′) on the side-arm (K) and the steam trap (6.1.3) on the safety tube (N). Turn the apparatus upside down, fill it with the cleaning solution (5.2) and leave it in this position overnight. Rinse the apparatus very carefully with water after having cleaned it.

8.2 Preparation of the test sample

If the test portion has to be ground (see Annex A), crush a sufficient quantity of the laboratory sample just before adding to the round-bottom flask to a suitable degree of fineness according to the product concerned (see ISO 2825). During the crushing procedure, ensure that the temperature of the test portion does not rise.

The mesh size of the sample shall be stated in each International Standard relating to a given spice.

8.3 Test portion

Weigh, to the nearest 0,01 g, on the filter paper (6.2), the specified quantity of test sample (see Annex A).

8.4 Determination

8.4.1 Determination of the volume of xylene

Using the measuring cylinder (6.6), transfer the specified quantity of water (see Annex A) to the flask (6.1.1) and add the anti-bumping granules or glass beads (6.5). Connect the flask to the condenser system (6.1.2) and fill the tube graduated in divisions of 0,05 ml (JL), the collector bulb (L) and the inclined tube (O) with water through the side-arm (K). Using the pipette (6.3), add 1 ml of the xylene (5.1) through the side-arm. Half fill the steam trap (6.1.3) with water and connect it to the condenser system. Heat the flask and regulate the rate of distillation to 2 ml/min or 3 ml/min unless otherwise specified. Distil for about 30 min and then remove the source of heat (6.4). By means of the three-way tap (M), allow the xylene to flow into the tube (JL) in such a way that the upper level coincides with the zero mark. Allow to cool for at least 10 min and measure the volume of xylene.

8.4.2 Determination of volume of organic phase (volatile oil and xylene)

Transfer the filter paper (6.2) with the test portion (8.3) to the flask (6.1.1), and again connect the flask to the condenser system. Heat the flask and regulate the rate of distillation to 2 ml/min or 3 ml/min unless otherwise specified. Allow the distillation to continue for the specified time (see Annex A). (Record the distillation time for inclusion in the test report.)

Remove the source of heat (6.4) and allow to cool. After 10 min, read the volume of the organic phase (mixture of volatile oil and xylene) collected in the measurement tube.

8.4.3 Determination of moisture content

Determine the moisture content by the method specified in ISO 939.

9 Expression of results

The volatile oil content, w_{VO}, expressed in millilitres per 100 g of dry product, is given by the following equation:

$$w_{VO} = 100 \times \frac{V_1 - V_0}{m} \times \frac{100}{100 - w_{H_2O}}$$

where

V_0 is the volume, in millilitres, of xylene measured in 8.4.1;

V_1 is the total volume, in millilitres, of volatile oil and xylene measured in 8.4.2;

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

w_{H_2O} is the moisture content, expressed as a percentage mass fraction, determined in 8.4.3.

10 Precision

Details of an interlaboratory trial on the precision of the method are summarized in Annex B. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

