International Standard 6571

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

Spices, condiments and herbs — Determination of volatile oil content

Épices, aromates et herbes — Détermination de la teneur en huiles essentielles

First edition — 1984-11-15

UDC 664.5 : 543.813 **Ref. No. ISO 6571-1984 (E)**

Descriptors : agricultural products, seasonings, spices, chemical analysis, determination of content, essential oils.

Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 6571 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

Printed in Switzerland

Spices, condiments and herbs — Determination of volatile oil content

1 Scope and field of application

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

2 References

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

ISO 948, *Spices and condiments — Sampling.*

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

3 Definition

volatile oil content: All the substances entrained by steam under the conditions specified in this International Standard, and expressed in millilitres per 100 g of dry product.

4 Principle

Distillation of an aqueous suspension of the product, collection of the distillate in a graduated tube containing a measured volume of xylene to fix the volatile oil, allowing the organic and aqueous phases to separate and reading the total volume of the organic phase. Calculation of the volatile oil content after deducting the volume of xylene.

5 Reagents

All reagents shall be of recognized analytical grade and the water used shall be 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:

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 or 1 000 ml, according to the product concerned (see the annex).

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 ball-shaped 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).

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).

6.2 Filter paper, of diameter 11 cm.

1) This apparatus corresponds to the type described in chapter V.4.5.8 of the European Pharmacopoeia (1980).

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

See ISO 948.

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 the annex.

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 for one night. 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 the annex), crush a sufficient quantity of the laboratory sample to a suitable degree of fineness according to the product concerned.

The mesh size of the sample shall be stated in each 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 the annex).

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 the annex) 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 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, 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 or 3 ml/min unless otherwise specified. Allow the distillation to continue for the specified time (see the annex). (The distillation time shall be mentioned 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, expressed in millilitres per 100 g of dry product, is equal to

$$100 \times \frac{V_1 - V_0}{m} \times \frac{100}{100 - w(\%)}$$

where

V_0 is the volume of xylene, in millilitres, 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;

$w(\%)$ is the moisture content, expressed as a percentage by mass, determined in 8.4.3.

10 Test report

The test report shall show the method used, the distillation time and the results obtained. It shall also mention any operating details not specified in this International Standard, 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.

Dimensions in millimetres

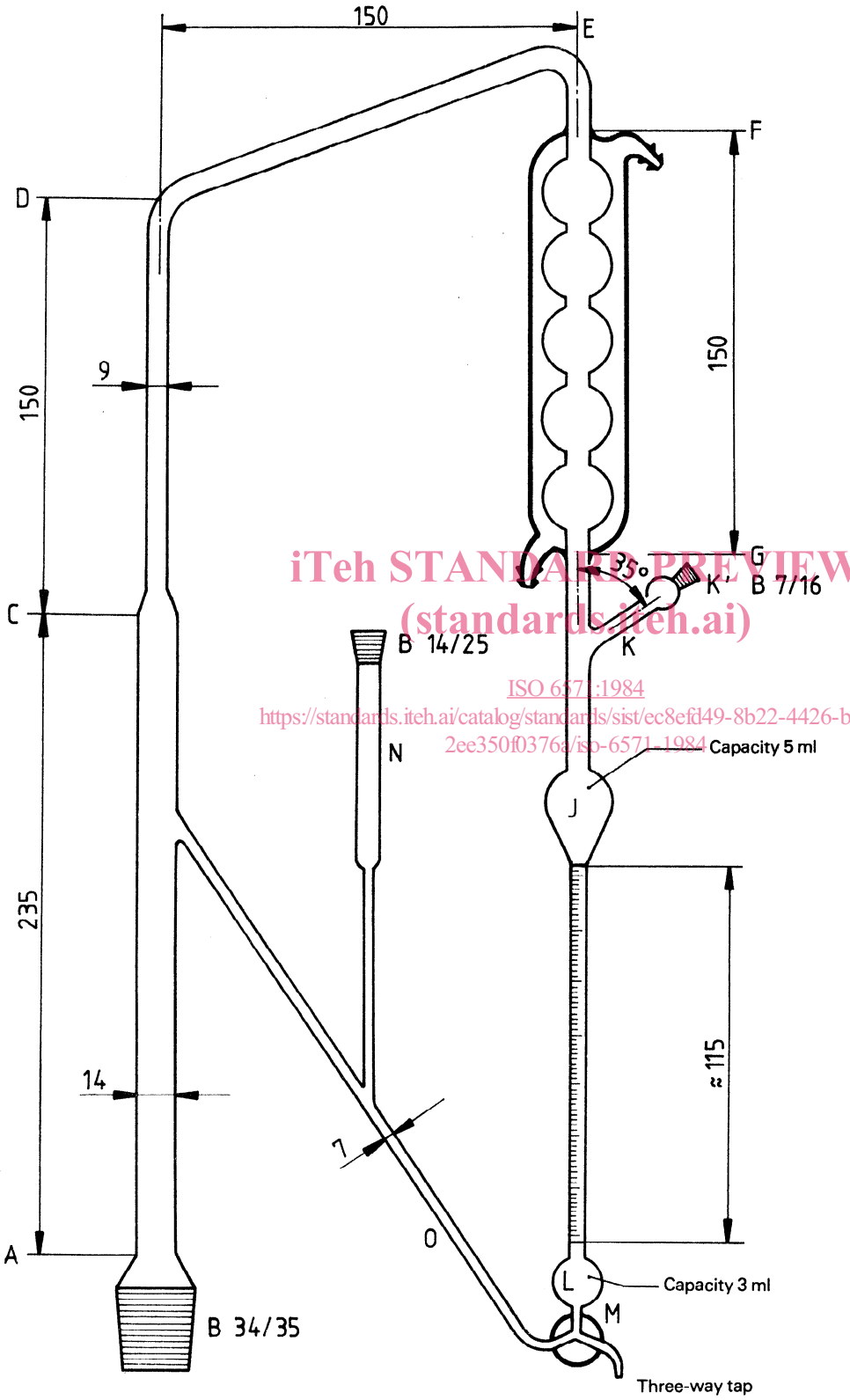

Figure 1 — Condenser system

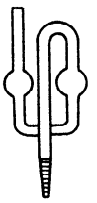

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Figure 2 — Steam trap (6.1.3)

Annex

Test parameters for different spices, condiments and herbs

Spice	Mass of test portion g	Form for distillation	Volume of water ml	Distillation time h
Aniseed	25	ground	500	4
Basil, sweet	50	whole/leaf	500	5
Camomile (Roman)	30	whole/leaf	300	3
Camomile (vulgaris)	50	whole/leaf	500 (0,5 mol/l HCl)	4
Caraway	20	whole	300	4
Cardamom	20	whole	400	5
Cassia	40	ground	400	5
Chervil	40	whole/leaf	600	5
Cinnamon	40	ground	400	5
Clove	4	ground	400	4
Coriander	40	ground	400	4
Cumin seed	25	ground	500	4
Curry powder	25	ground	500	4
Dill	25	ground	500	4
Fennel	25	ground	300	4
Garlic	25	ground	500	4
Ginger	30	ground	500	4
Juniper	25	ground	500	5
Mace	15	ground	400	4
Marjoram, sweet	40	whole/leaf	600	4
Marjoram, wild	40	whole/leaf	600	5
Mint	40	whole/leaf	600	4
Mixed herbs	40	whole/leaf	600	4
Mixed spices	40	ground	600	5
Nutmeg	15	ground	400	4
Parsley	40	whole/leaf	600	5
Penny royal	40	whole/leaf	600	5
Pepper	40	ground	400	4
Peppermint	50	whole/leaf	500	2
Pickling spice	25	ground	500	4
Pimento	30	ground	500	5
Rosemary	40	whole/leaf	600	5
Sage	40	whole/leaf	600	5
Savory	40	whole/leaf	600	5
Tarragon	40	whole/leaf	600	5
Thyme	40	whole/leaf	600	5
Turmeric	40	ground	400	5

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