
International Standard



939

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Spices and condiments — Determination of moisture content — Entrainment method

Épices — Détermination de la teneur en eau — Méthode par entraînement

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Descriptors : agricultural products, spices, chemical analysis, water, azeotropic drives method.

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 939 was developed by Technical Committee ISO/TC 34, *Agricultural food products*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 939-1969 which had been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Portugal
Brazil	Greece	Romania
Bulgaria	Hungary	South Africa, Rep. of
Chile	India	Thailand
Colombia	Iran	Turkey
Czechoslovakia	Israel	United Kingdom
Egypt, Arab Rep. of	Korea, Rep. of	USSR
France	Poland	Yugoslavia

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

Netherlands

Spices and condiments — Determination of moisture content — Entrainment method

0 Introduction

This International Standard is applicable to most spices and condiments. In view of the number and variety of such products, however, it may be necessary in particular cases to modify the method or even to choose a more suitable method.

Such modifications or other methods will be indicated in the International Standards giving specifications for the spices and condiments in question.

1 Scope and field of application

This International Standard specifies an entrainment method for the determination of the moisture content of spices and condiments.

2 References

ISO 948, *Spices and condiments — Sampling*.

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

3 Definition

moisture content : The quantity of water, expressed as a percentage by mass, distilled and collected in accordance with the method specified in this International Standard.

4 Principle

Determination of the amount of water entrained by azeotropic distillation, using an organic liquid immiscible with water, and collected in a graduated tube.

5 Reagent

5.1 Toluene.

Saturate the toluene by shaking with a small quantity of water and distil. Use the distillate for the determination of the moisture.

NOTE — Different solvents are used for the determination of moisture. Unless otherwise specified in the International Standard giving specifications for the spice or condiment in question, toluene shall be the solvent used for the determination.

6 Apparatus

6.1 Distillation apparatus (a suitable form of apparatus is described in the annex and shown in the figure), comprising the following components fitted together by means of ground glass joints :

6.1.1 Flask, short-necked, of capacity at least 500 ml.

6.1.2 Reflux condenser.

6.1.3 Receiver, with a graduated tube, interposed between the flask and the condenser.

6.2 Analytical balance.

7 Sampling

Sample the material by the method specified in ISO 948.

8 Procedure

8.1 Preparation of apparatus

Clean the entire apparatus with a potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before use.

8.2 Preparation of test sample

Prepare the test sample by the method specified in ISO 2825.

8.3 Test portion

Weigh, to the nearest 0,01 g, about 40 g of the test sample (8.2), such that the quantity of water collected will not exceed 4,5 ml.

8.4 Determination

Transfer quantitatively the test portion (8.3) to the distillation flask (6.1.1) with the toluene (5.1), add sufficient toluene (about 75 ml in all) to cover the sample completely and swirl to mix. Assemble the apparatus and fill the receiver (6.1.3) with the toluene by pouring it through the condenser (6.1.2) until it begins to overflow into the distillation flask. If necessary, insert a loose cotton plug in the top of the condenser or attach to it a

small calcium chloride tube to prevent condensation of atmospheric moisture within the condenser tube. In order to control refluxing, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to about 200 drops per minute and continue until no more water is collected. Purge the reflux condenser occasionally during the distillation with 5 ml portions of the toluene to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the toluene by occasionally moving a spiral copper wire up and down in the condenser and receiver, thus causing the water to settle at the bottom of the receiver. Reflux until the water level in the receiver remains unchanged for 30 min and then shut off the source of heat.

Flush the condenser with toluene as required, making use of the spiral copper wire to discharge any moisture droplets.

Immerse the receiver in water at room temperature for at least 15 min or until the toluene layer is clear; then read the volume of water.

9 Expression of results

The moisture content, expressed as a percentage by mass, is equal to

$$\frac{100 V}{m}$$

where

V is the volume, in millilitres, of water collected;

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

It is assumed that the density of water is 1 g/ml exactly.

10 Test report

The test report shall show the method used and the result obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

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

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Annex

Example of entrainment distillation apparatus

(This annex does not form part of the standard.)

The apparatus consists of a glass flask heated by a suitable means and provided with a reflux condenser discharging into a receiver connected to the flask. The connections between the receiver and the condenser and the flask are interchangeable ground glass joints. The receiver serves to collect and measure the condensed water, and to return the solvent to the flask. The assembly of the apparatus is shown in the figure and the various components are described below.

a) **Flask**, of capacity 500 ml, of the shape shown in the figure and made of heat-resistant glass, well annealed and as free as possible from striae and similar defects.

b) **Reflux condenser**, water cooled, made of glass, having a jacket approximately 400 mm long and an inner tube of diameter 9,5 to 12,5 mm. The tip of the condenser to be inserted in the receiver may be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the receiver, the tip of the condenser is 6 to 7 mm above the surface of the liquid in the receiver after distillation conditions have been established.

c) **Receiver**, of capacity 5 ml, made of heat-resistant glass, well annealed and as free as possible from striae and similar defects, provided with ground glass joints, with the shape, dimensions and tolerances given in the figure, and consisting essentially of the upper chamber, together with the tube and ground joint leading to the flask, and the graduated tube. The graduated portion has a capacity of 5 ml when filled to the highest graduation mark.

The scale covers the range of 0 to 5 ml and is graduated at intervals of 0,1 ml. The graduation marks corresponding to each millilitre are numbered and carried completely round the tube. The graduation marks midway between the numbered marks are carried three-quarter-way, and the remaining marks are carried half-way, around the tube. The error at any indicated capacity does not exceed 0,05 ml.

d) **Heat source**, either an oil bath or an electric heater provided with a sliding rheostat or other means of heat control.

The temperature of the oil in the bath should not be very much higher than the boiling point of toluene.

e) **Copper wire**, long enough to extend through the condenser and with one end twisted into a spiral. The diameter of the spiral is such that it fits snugly within the graduated portion of the receiver and yet can be moved up and down.

Dimensions in millimetres

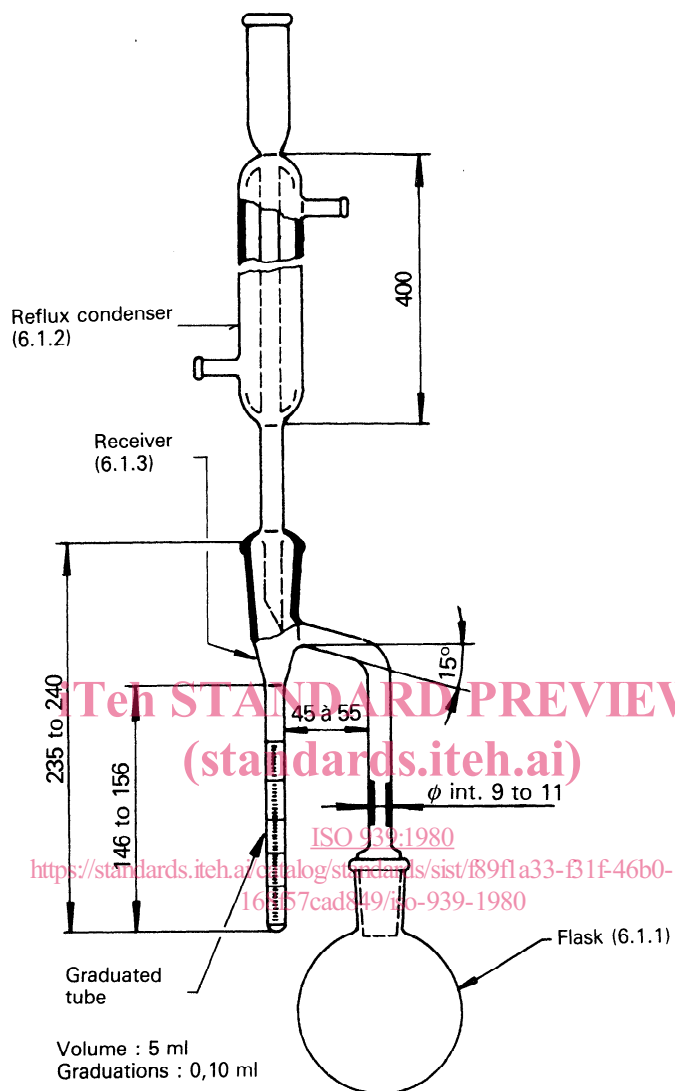


Figure — Distillation apparatus

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