

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 939

SICES AND CONDIMENTS

DETERMINATION OF MOISTURE CONTENT
(ENTRAINMENT METHOD)

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BRIEF HISTORY

The ISO Recommendation R 939, *Spices and condiments – Determination of moisture content (Entrainment method)*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question by the Technical Committee led, in 1966, to the adoption of a Draft ISO Recommendation.

In January 1967, this Draft ISO Recommendation (No. 1200) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

Netherlands

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided in January 1969, to accept it as an ISO RECOMMENDATION.

SPICES AND CONDIMENTS

DETERMINATION OF MOISTURE CONTENT
(ENTRAINMENT METHOD)

INTRODUCTION

This ISO Recommendation 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 ISO Recommendations giving specifications for the spices and condiments in question.

NOTE. — The method described in this ISO Recommendation is under review, and a new method may be proposed later.

1. SCOPE

This ISO Recommendation describes a method for the determination of the moisture content of spices and condiments.

2. DEFINITION

By *moisture content* is meant the quantity of water, expressed as a percentage by mass, distilled and collected in accordance with the method described.

3. PRINCIPLE

Determination of the amount of water carried over by distillation, with the aid of an organic liquid not miscible with water, and collected in a graduated tube.

4. REAGENT

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

NOTE. — Different solvents are used for the moisture determination. Unless otherwise specified in an individual material specification, toluene should be the solvent used for this determination.

5. APPARATUS

5.1 *Distillation apparatus*, comprising the following components fitted together by means of ground glass joints :

5.1.1 *Flask*, short-necked, at least 500 ml.

5.1.2 *Reflux condenser*.

5.1.3 *Receiver*, with graduated tube, interposed between the flask and the condenser.

A suitable form of apparatus is described in the Appendix and shown in the Figure, page 7.

5.2 *Analytical balance*.

6. SAMPLING

Sample the material by the method described in ISO Recommendation R 948, *Spices and condiments – Sampling*.

7. PROCEDURE

7.1 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 using.

7.2 Preparation of sample

Prepare the sample as described in ISO Recommendation R . . . ,* *Spices and condiments – Preparation of sample for test*.

7.3 Test portion

Weigh, to the nearest 0.01 g, about 40 g of the prepared sample, such that the quantity of water measured will not exceed 4.5 ml.

7.4 Determination

Transfer quantitatively the test portion to the distillation flask (5.1.1) with toluene (4), add sufficient toluene (about 75 ml in all) to cover the sample completely and swirl to mix. Assemble the apparatus and fill the receiver (5.1.3) with the solvent (4) by pouring it through the condenser (5.1.2) 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 that the refluxing may be under control, 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 toluene (4) 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 minutes and then shut off the source of heat.

* In preparation.

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

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

8. EXPRESSION OF RESULTS

The moisture content, 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 grammes, of the test portion.

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

9. TEST REPORT

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

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

APPENDIX

EXAMPLE OF ENTRAINMENT DISTILLATION
APPARATUS

The apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the flask should be interchangeable ground glass joints. The trap 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, page 7, and the various components are described below.

- (a) **Flask.** A 500 ml flask of the shape shown in the Figure made of high resistance glass, well annealed and as free as possible from striae and similar defects.
- (b) **Reflux condenser.** Water cooled, glass reflux condenser, having a jacket approximately 400 ml long and an inner tube of 9.5 to 12.5 mm outside diameter. The tip of the condenser to be inserted in the trap may be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser is 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established.
- (c) **Receiver,** otherwise called the **trap.** Receiver of 5 ml capacity, made of high resistance 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 should not exceed 0.05 ml.
- (d) **Heat source.** The source of heat may be 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.** This should be long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet can be moved up and down.