
International Standard



1208

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Spices and condiments — Determination of filth

Épices — Détermination des impuretés

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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 1208 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in September 1981.

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

Australia	Israel	Portugal
Czechoslovakia	Italy	Romania
Egypt, Arab Rep. of	Kenya	South Africa, Rep. of
Ethopia	Malaysia	Sri Lanka
France	Netherlands	Tanzania
Germany, F.R.	New Zealand	Turkey
Hungary	Peru	United Kingdom
India	Philippines	Yugoslavia
Iraq	Poland	

The member bodies of the following countries expressed disapproval of the document on technical grounds:

Canada
USA

Spices and condiments — Determination of filth

0 Introduction

International Standards specifying requirements for spices and condiments prescribe, *inter alia*, that spices and condiments shall be practically free from dead insects, insect fragments and rodent contamination. For testing compliance with this requirement, hand lens examination is adequate only in the case of whole spices and condiments. For ground spices and condiments, the method to be used is that specified in this International Standard, especially in cases of dispute.

The method 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 and other methods will be indicated in the International Standards appropriate to the spices and condiments concerned.

1 Scope and field of application

This International Standard specifies a method for the quantitative determination of filth in spices and condiments. As no limits have been prescribed for filth in International Standards on spices and condiments, this method should be used for collecting more data and for settling international disputes.

2 Reference

ISO 948, *Spices and condiments — Sampling*.

3 Definition

For the purpose of this International Standard, the following definition applies.

filth: Mineral matter (sand, soil) and matter of animal origin (insect fragments, rodent hairs and excreta) separated from the product under the conditions specified.

4 Principle

Washing the product with chloroform (after, if necessary, preliminary extraction with light petroleum) and examination of

the washings for heavy filth and sand. Washing the product with water, with or without treatment with pancreatin enzyme, and agitation with light petroleum, the light filth collecting at the interface between the liquids after separation. Transference of the light filth to a filter paper and microscopical examination for contaminants such as insect fragments and rodent hairs.

5 Reagents

The water used shall be distilled water or water of at least equivalent purity.

5.1 Chloroform and, if required (see 8.3), **chloroform/carbon tetrachloride mixtures**.

5.2 Pancreatin solution.

Use pancreatin complying with the requirements of the annex, and which has been kept at about 10 °C. Use a solution which has been freshly prepared as follows.

Mix 10 g of pancreatin with 100 ml of warm water (temperature not exceeding 40 °C). Stir mechanically for 10 min or allow to stand for 30 min with intermittent stirring. Pour the solution through a loosely packed pad of cotton wool, 100 mm thick, in a 60° funnel of diameter 100 to 125 mm. Repeat the filtration through the same pad. If filtration is slow in either case, filter with suction through a fast filter paper using a Buchner funnel. If the filtration is still slow, pour the solution through a slightly compressed cotton wool plug in the 60° funnel. Repeat if necessary, until the solution filters rapidly through paper. (Soluble pancreatin may be filtered directly through paper with suction.) Dilute the filtrate to 100 ml for each 10 g portion.

5.3 Trisodium orthophosphate, 50 g/l solution.

5.4 Formaldehyde solution.

5.5 Light petroleum, boiling range 40 to 60 °C.

5.6 Light petroleum, boiling range 100 to 120 °C.

6 Apparatus

Usual laboratory apparatus, and

6.1 Trap flask (Wildman), consisting of a 1 000 ml conical flask into which is inserted a close-fitting rubber stopper carried on a rigid metal rod, 5 mm in diameter and approximately 100 mm longer than the height of the flask. (A rod of a greater diameter is not desirable because of its greater displacement of liquid.) The rod is threaded at its lower end and furnished with nuts and washers to hold it in place on the stopper. The lower nut and washer are countersunk in the rubber to prevent them from striking the flask. See the figure.

Alternatively, a separating funnel, of capacity 1 000 ml, may be used.

6.2 Beakers, of capacity 600 ml.

6.3 Buchner funnel, of diameter 15 cm with a filter paper.

6.4 Buchner funnel, of diameter 7 cm with a filter paper ruled with parallel lines 5 mm apart.

6.5 Filter paper, ashless.

6.6 Crucible, tared.

6.7 Oven, capable of being controlled at 80 °C.

6.8 Oven, capable of being controlled at 103 °C.

6.9 Petri-dish, of diameter 80 mm.

6.10 Suitable magnifying device (microscope, binocular magnifier, etc. or preferably a wide-field stereoscopic microscope).

6.11 Balance.

7 Sampling

Sample the material by the method specified in ISO 948.

8 Procedure

NOTE — To achieve a satisfactory separation of the light filth from spice tissue, it may be necessary to remove most of the volatile oil and fat, or to treat the test portion with pancreatin enzyme to digest starch and protein, or both.

If removal of volatile oil or fat is required, proceed as described in 8.2; otherwise, proceed directly as described in 8.3.

8.1 Test portion

Ensure that the test portion is representative of the laboratory sample (final lot sample).

8.1.1 Whole and broken spices

Break the material into small pieces, weigh, to the nearest 0,1 g, 25 g of the sample and transfer it to a breaker (6.2).

8.1.2 Ground spices

Weigh, to the nearest 0,1 g, 25 g of the sample and transfer it to a beaker (6.2).

8.2 Preliminary removal of volatile oil and fat

Add 200 ml of the light petroleum (5.5) to the test portion (8.1) in the beaker. Boil gently on a warm water bath for 15 min. Remove the light petroleum by decanting, taking care not to lose any of the material under test.

8.3 Separation of heavy filth and sand

Add 400 ml of the chloroform (5.1) to the test portion (8.1) or to the residue from the operation described in 8.2. Allow the beaker to stand for at least 1 h with occasional stirring. Transfer the material and the solvent to the Buchner funnel (6.3), leaving the heavy residue of sand and soil in the beaker. Drain. If appreciable spice tissue remains on the bottom of the beaker, add successive portions of chloroform mixed with carbon tetrachloride to give increasingly higher relative densities until practically all spice tissue has been removed by flotation. Transfer the heavy residue from the beaker to the ashless filter paper (6.5) and wash it with water to remove any sodium chloride present in the spice. Examine this residue. If there is an appreciable residue, place the filter paper in the tared crucible (6.6), ignite, and weigh the sand and soil.

8.4 Treatment of residue retained on the Buchner funnel

Dry the material retained on the Buchner funnel (see 8.3) for 1 h in the oven (6.7), controlled at 80 °C.

8.4.1 Procedure without enzyme treatment

Transfer the residue to the trap flask (6.1). Add about 150 ml of water, heat to boiling and simmer for 15 min with stirring. Wash down the inside of the flask with water and cool to below 20 °C, diluting to about 600 ml with water.

8.4.2 Procedure with enzyme treatment

Transfer the dry residue to a beaker (6.2). Add 300 ml of water and stir until homogeneous. Add 50 ml of the pancreatin solution (5.2) and mix. Adjust to pH 8 with the trisodium orthophosphate solution (5.3). Readjust the pH after about 15 min, and again after about 45 min. Add 5 drops of the formaldehyde solution (5.4) and allow to digest overnight at 37 to 40 °C. Cool and transfer to the trap flask (6.1), making up to about 600 ml with water.

8.5 Separation of light filth

8.5.1 Add, down the stirring rod of the flask, 25 ml of the light petroleum (5.6). Tilt the trap flask at about 45° to the ver-

tical and mix for 1 min at the rate of 4 strokes per second with a brisk rotary motion so that the liquid is brought to a roll. Avoid splashing through the surface of the liquid in the trap flask with the rubber stopper.

Allow to stand for 5 min. Then fill the flask with water and allow to stand for 30 min. Stir every 5 min.

8.5.2 Spin the stopper to remove sediment and raise the stopper as far as possible into the neck of the flask, making sure that the light petroleum layer and at least 1 cm of the liquid below the interface are above the stopper. Holding the stopper in place, transfer the trapped liquid to the Buchner funnel (6.4). Filter.

8.5.3 Add 15 ml of the light petroleum (5.6) to the contents of the trap flask and mix thoroughly. After 15 min, repeat the operations described in 8.5.2. If this second extraction yields an appreciable quantity of filth, decant most of the liquid from the flask, add another 15 ml of the light petroleum (5.6) and carry out a third extraction.

8.6 Microscopical examination of light filth

Remove the filter paper from the Buchner funnel and place it on the petri-dish (6.9). Place the petri-dish in the oven (6.8), controlled at 103 °C, for 30 min. The dried filter paper should be tightly stuck to the petri-dish.

Examine the entire area of the filter paper, using the magnifying device (6.10) in reflected light, scraping and probing with a mounted needle.

Carry out the examination from left to right, from top to bottom for the first interval, from bottom to top for the second interval, from top to bottom for the third interval, etc.

9 Expression of results

9.1 Heavy filth (see 8.4)

Note the presence of mineral matter.

If the amount of sand and soil is such as to require calcination and weighing of these impurities, the mineral impurities content of the product, expressed as a percentage by mass, is equal to

$$m_1 \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue obtained (8.3).

9.2 Light filth (see 8.6)

Note the presence of matter of animal origin (see 8.6).

If required, record separately the numbers of insect fragments, rodent hairs and other items of animal origin in the test portion, i.e. the numbers per 25 g of sample.

10 Test report

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

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

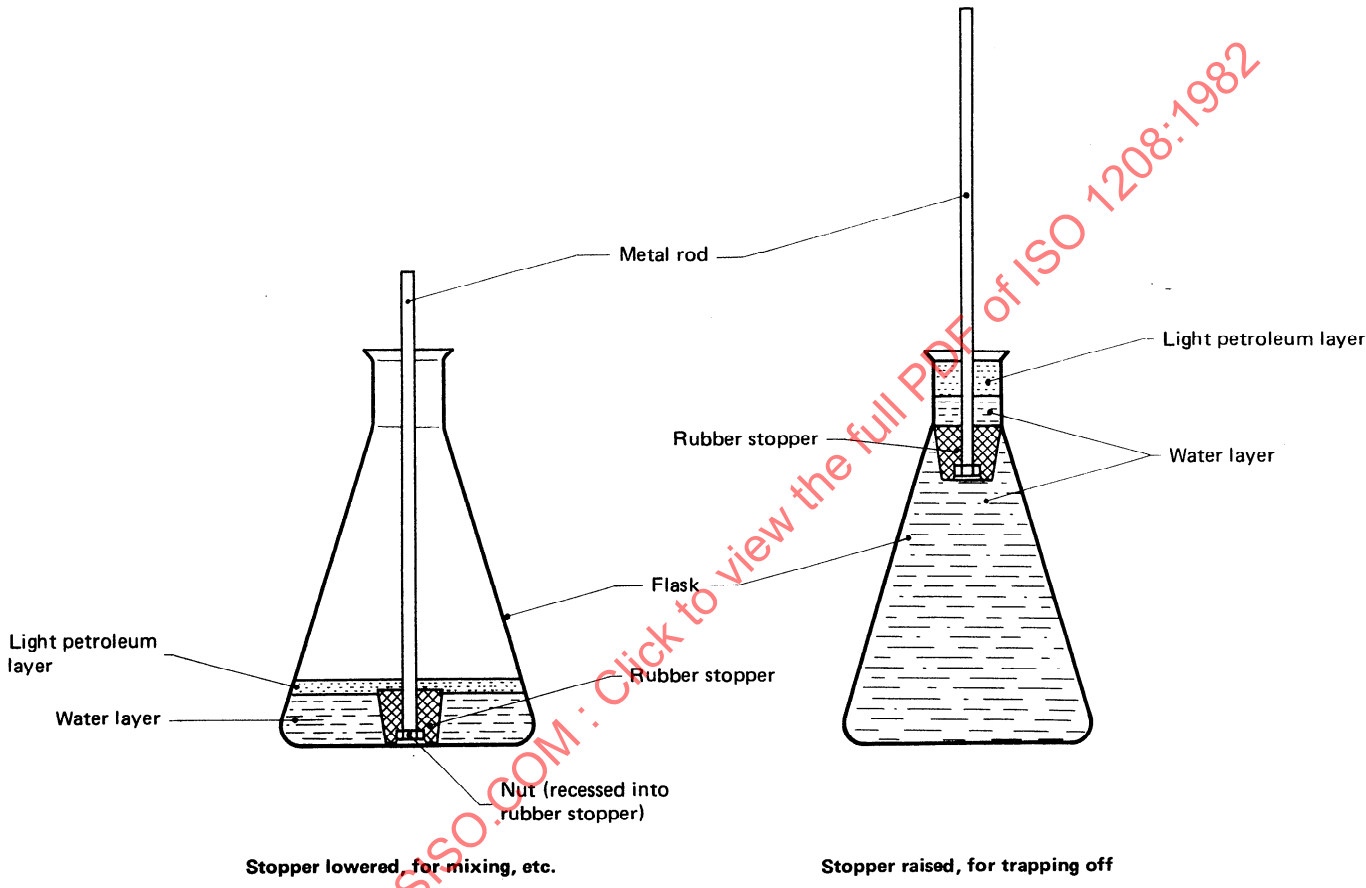


Figure — Wildman trap flask (6.1), showing method of use

NOTE — The upper nut and washer have been omitted.