SLOVENSKI STANDARD
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Suhi česen - Določanje hlapnih organskih žveplovih spojin

Dehydrated garlic -- Determination of volatile organic sulphur compounds

Ail déshydraté -- Détermination des composés sulfurés organiques volatils

Ta slovenski standard je istoveten z: **ISO 5567:1982**

ICS:

67.220.10 Začimbe Spices and condiments

SIST ISO 5567:1997 **en**

International Standard 5567

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Dehydrated garlic — Determination of volatile organic sulphur compounds

Ail déshydraté — Détermination des composés sulfurés organiques volatils

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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 5567 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in April 1981.

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

Brazil	Ireland	Romania
Canada	Israel	South Africa, Rep. of
Czechoslovakia	Kenya	Spain
Egypt, Arab. Rep. of	Korea, Rep. of	Sri Lanka
Ethiopia	Mexico	Tanzania
France	Netherlands	Thailand
Germany, F.R.	New Zealand	Turkey
Hungary	Peru	United Kingdom
India	Philippines	Yugoslavia
Indonesia	Poland	
Iran	Portugal	

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

USA

Printed in Switzerland

Dehydrated garlic — Determination of volatile organic sulphur compounds

1 Scope and field of application

This International Standard specifies a method for the determination of volatile organic sulphur compounds in dehydrated garlic.

2 Principle

After maceration of a test portion in aqueous medium, addition of ethanol, distillation of the volatile organic sulphur compounds, and argentimetric titration of the distillate in nitric acid medium.

3 Reagents

The reagents shall be of recognized analytical purity and the water used shall be distilled water or water of at least equivalent purity.

3.1 Ethanol, 95 % (V/V).

3.2 Liquid paraffin.

3.3 Ammonium hydroxide, 10 % solution.

3.4 Silver nitrate, 0,1 mol/l solution.

3.5 Nitric acid, $\varrho_{20} \approx 1{,}40$ g/ml.

3.6 Nitric acid, 10 % (V/V) solution.

3.7 Iron ammonium alum, cold saturated solution.

3.8 Ammonium thiocyanate, standard volumetric solution, $c(NH_4SCN) = 0{,}1$ mol/l.

4 Apparatus

NOTE — During the analysis, avoid all contact with copper or rubber, especially in the distillation apparatus. The apparatus should have ground glass joints.

Usual laboratory apparatus, and in particular

4.1 Distillation apparatus (see the figure), consisting of a 250 ml flask with a ground-neck which can be fitted with a stopper, and a straight condenser.

4.2 Conical flask, of capacity 250 ml with a ground-neck which can be fitted with a reflux condenser.

4.3 One-mark pipette, of capacity 20 ml.

4.4 Burette with tap, of capacity 25 ml, accurate to 0,05 ml.

4.5 Thermostatically controlled bath, capable of being controlled at 37 ± 1 °C.

4.6 Balance.

4.7 Fritted glass filter, of porosity 4 to 16 µm, and a **filter flask.**

4.8 Vacuum pump.

5 Procedure

5.1 Preparation of the test sample

Homogenize the laboratory sample of dehydrated garlic and, if necessary, reduce it to fine particles.

5.2 Test portion

Weigh, to the nearest 0,01 g, about 10 g of the test sample.

5.3 Determination

5.3.1 Maceration

Introduce the test portion into the flask of the distillation apparatus (4.1), add 100 ml of water at 40 °C, and allow to macerate for 2 h in the thermostatically controlled bath (4.5) at 37 ± 1 °C, keeping the flask closed by means of a ground stopper.

5.3.2 Distillation

Add 20 ml of the ethanol (3.1) and 2 ml of the liquid paraffin (3.2) to avoiding frothing.

Connect the flask quickly to the condenser, with the tip of the latter dipping into the conical flask (4.2) containing approximately 10 ml of the ammonium hydroxide solution (3.3). Ensure that the tip of the condenser is below the surface of the ammonium hydroxide solution.

Regulate the heating in such a way as to avoid entraining any froth that might be formed despite the presence of the liquid paraffin.

Heat the flask (see 4.1) so as to a obtain a brisk rate of distillation and continue the distillation until about 60 ml of distillate has been collected.

Rinse the condenser with water, collecting the rinsings in the conical flask.

5.3.3 Titration

Neutralize the ammoniacal distillate in the flask (4.2), adjusting the pH to 7 ± 0,1 by addition of the nitric acid solution (3.6).

By means of a pipette (4.3), add 20 ml of the silver nitrate solution (3.4) and boil under reflux for 1 h.

Cool the flask and filter the distillate on the fritted glass filter (4.7) placed on the filter flask, operating under suction applied by means of the vacuum pump (4.8). Wash the precipitate four times with hot water and collect the filtrate and washings quantitatively.

Add about 5 ml of the nitric acid (3.5), a few drops of the iron ammonium alum solution (3.7) and titrate with the ammonium thiocyanate solution (3.8) until a persistent pink coloration is obtained.

5.4 Number of determinations

Carry out two determinations on the same prepared sample.

6 Expression of results

6.1 Method of calculation and formula

The volatile organic sulphur compounds content, expressed as allyl sulphide as a percentage by mass, is equal to

$$0{,}005\,7\,(20 - V) \times \frac{100}{m}$$

where

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

V is the volume, in millilitres, of the ammonium thiocyanate solution used.

Take as the result the arithmetic mean of the values obtained in the two determinations, provided that the requirements for repeatability (see 6.2) are satisfied. Otherwise, repeat the analysis.

6.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 5 % of the mean.

7 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 incidents likely to have influenced the results.

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

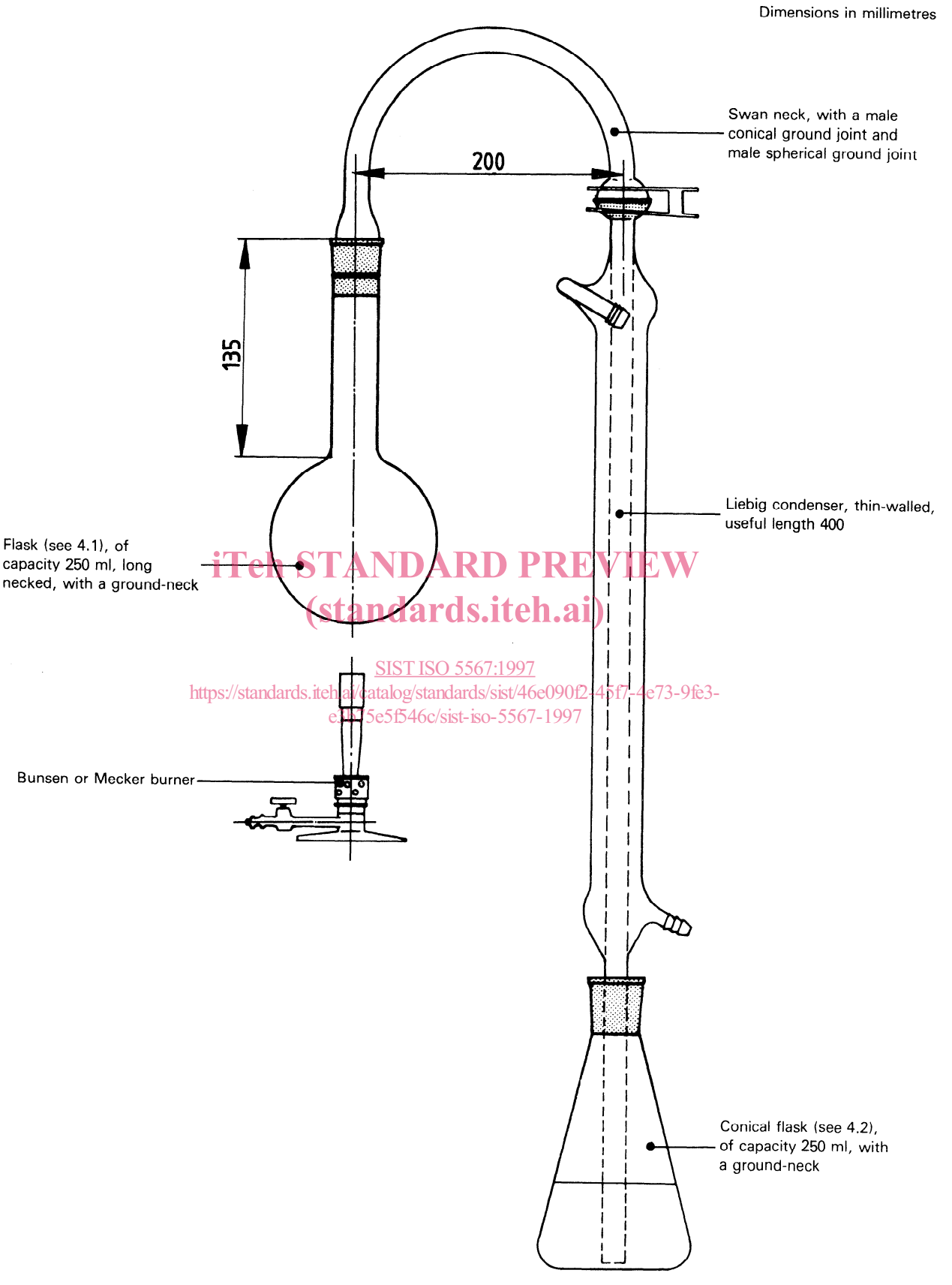

Figure — Allyl sulphide distillation apparatus (recommended)