INTERNATIONAL STANDARD

ISO 11162

First edition
2001-03-15

Peppercorns (*Piper nigrum* L.) in brine — Specification and test methods

*Poivre vert (*Piper nigrum *L.) en saumure — Spécifications et méthodes d'essai*

Reference number
ISO 11162:2001(E)

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Printed in Switzerland

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11162 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 7, *Spices and condiments*.

Annexes A, B, C and D form a normative part of this International Standard.

Peppercorns (*Piper nigrum* L.) in brine — Specification and test methods

1 Scope

This International Standard specifies the requirements for peppercorns (*Piper nigrum* L.) in brine.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 927, *Spices and condiments — Determination of extraneous matter content*.

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

ISO 948, *Spices and condiments — Sampling*.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

ISO 5564, *Black pepper and white pepper, whole or ground — Determination of piperine content — Spectrophotometric method*.

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1
peppercorns in brine
product prepared under controlled conditions from fresh green pepper berries of the plant *Piper nigrum* L. which have reached a proper degree of maturity and which satisfy the requirements of this International Standard

3.2
total acidity of brine
acidity produced by all the acids contained in the brine, expressed as a mass fraction of citric acid, in percent

NOTE Citric acid is a triacid with a molar mass equal to 192,13 g.

3.3
piperine content
quantity of pungent components (piperine and similar compounds) contained in the product, determined by the method specified in this International Standard

NOTE It is expressed as a mass fraction in percent.

3.4
chloride content
mass fraction of chloride ions, expressed as sodium chloride, present in the brine of peppercorns when determined in accordance with the method specified in this International Standard

4 Requirements

4.1 Colour and size

The berries shall possess characteristic colour of green pepper of proper maturity varying from pale green to green. The berries are between 3 mm to 6 mm in diameter and shall be more or less of a uniform size within a same batch.

4.2 Odour and flavour

The berries shall have the characteristic odour and flavour of fresh green pepper berries and shall be completely devoid of any objectionable taste or odour.

4.3 Extraneous matter

For the purposes of this International Standard, all materials other than green pepper berries, irrespective of whether they are of vegetable (e.g. stem or leaves) or mineral (e.g. sand) origin, shall be considered as extraneous matter.

NOTE Light berries, pinhead or broken berries are not considered as extraneous matter.

The total percentage of extraneous matter, when determined by the method specified in ISO 927, shall not exceed 1 % (mass fraction).

4.4 Defective berries

The maximum limit for defective berries, including discoloured, dark coloured, light and broken berries and pinheads, shall be 4 % (mass fraction) when determined on a sample of 500 g (drained mass) by physical separation.

4.5 Freedom from moulds, insects, preservatives, colouring matter and flavouring agents

Peppercorns in brine shall be free from moulds and insects. They shall also be free from preservatives, colouring matter and flavouring agents unless permitted under the national legislation of the importing country.

4.6 Piperine content of peppercorns in brine

The piperine content shall be determined following the method given in annex A.

4.7 Characteristics of the brine and processing conditions

4.7.1 The brine shall comply with the requirements given in Table 1.

4.7.2 The peppercorns in brine shall be processed under hygienic conditions.

4.8 Drained mass

The drained mass of the content shall be not less than 50 % (mass fraction) of the net mass when tested in accordance with the method specified in annex D.

Table 1 — Requirements for the brine

Characteristic	Specification	Test method
Appearance	Clear and practically free from sediment	Visual examination
Acetic acid or citric acid, % max.	0,6	Method given in annex B
Chloride content, expressed as sodium chloride, %	Between 12 and 15	Method given in annex C
pH	Between 4,0 and 4,5	pH-meter

5 Sampling

Sampling shall be carried out as specified in ISO 948.

6 Packaging and marking

6.1 Packaging

The product shall be packed in clean and sound containers made of a material which does not affect the product and which protects it from damage or spoilage.

The packaging shall also comply with any national legislation relating to environmental protection.

6.2 Marking

The following particulars shall be marked directly on each container or shall be marked on a label attached to the container:

a) name of the product and tradename, if any;

b) name and address of the processor or packer;

c) brand name, if any;

d) batch or code number;

e) net mass;

f) best before (month and year);

g) any other markings required by the purchaser (such as the year of harvest, the date of packing) or by national regulation (for example, percentage of citric acid in the brine);

h) processing country; and

i) reference of this International Standard.

Annex A
(normative)

Determination of piperine content

A.1 Introduction

Studies have determined that the test method described in ISO 5564 for the determination of piperine, on a dry basis, does not give consistent results when applied to peppercorns in brine. Studies have shown that this is due to the presence of sodium chloride and therefore it is necessary to take this into account in the calculation of the piperine content of peppercorns in brine. This is the reason why a method specific to peppercorns in brine has been established.

A.2 Principle

The piquant compounds are extracted with ethanol and determined spectrometrically at a wavelength of 343 nm. Then their piperine content is calculated.

A.3 Reagents

Use only reagents of recognized analytical grade.

A.3.1 Ethanol, 96 % (volume fraction).

A.4 Apparatus

Use the apparatus described in ISO 5564, together with the following.

A.4.1 Plastic cuvette, of diameter greater than 10 cm.

A.4.2 Oven, capable of being maintained at 50 °C ± 5 °C.

A.4.3 Analytical balance, capable of weighing to the nearest 0,01 g.

A.5 Preparation of test sample

Drain the brine from the green pepper berries.

Weigh, to the nearest 0,01 g, between 50 g and 60 g of the drained berries in the plastic cuvette (A.4.1). Spread them uniformly over the surface then place them in the oven (A.4.2) set at 50 °C for a minimum of 24 h.

A.6 Procedure

A.6.1 Determination of sodium chloride content

Determine the sodium chloride content using the method specified in annex C.

A.6.2 Determination of moisture content

Determine the moisture content of the drained, dried (see A.5) then crushed berries in accordance with the method specified in ISO 939.

A.6.3 Determination of piperine content

Determine the piperine content on a dry basis of the drained, dried (see A.5) then crushed berries in accordance with the method given in ISO 5564 but using a test sample of 0,7 g rather than 0,5 g.

A.7 Expression of results

The piperine content, on a dry basis, is calculated from the following:

$$P \Big/ \left[\left(1 - \frac{H}{100} \right) \left(1 + \frac{S}{100} \right) - \frac{S}{100} \times \frac{(m_1 - m_0)}{(m_2 - m_0)} \right]$$

where

P is the piperine content of the drained, dried and crushed berries, expressed as a mass fraction in percent, as obtained in A.6.3;

H is the water content of the drained, dried and crushed berries, expressed as a mass fraction in percent, as obtained in A.6.2;

S is the sodium chloride content of the brine, expressed as a mass fraction in percent, as obtained in A.6.1;

m_0 is the mass, in grams, of the empty plastic cuvette;

m_1 is the mass, in grams, of the empty plastic cuvette and drained berries;

m_2 is the mass, in grams, of the empty plastic cuvette and the drained dried berries.

Annex B
(normative)

Determination of total acidity, expressed as citric acid

B.1 Principle

The total acidity of brine is determined by titration with sodium hydroxide, using phenolphthalein as indicator.

B.2 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

B.2.1 **Ethanol**, 95 % to 96 % (volume fraction).

B.2.2 **Phenolphthalein solution**

Dissolve approximately 2 g of powdered phenolphthalein in 1 litre of ethanol (B.2.1).

Take 10 ml of this solution, using a pipette (B.3.2), then dilute to 1 litre with water.

B.2.3 **Sodium hydroxide solution**, 0,1 mol/l.

B.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

B.3.1 **Beaker**, graduated at least at 50 ml and 100 ml, class A.

B.3.2 **Pipette**, class A, to deliver 10 ml.

B.3.3 **Burette**, of capacity 25 ml.

B.3.4 **Magnetic stirrer**.

B.3.5 **Analytical balance**, capable of weighing to the nearest 0,001 g.

B.4 Procedure

B.4.1 Test portion

To ensure that the delivery from the burette is between 5 ml and 20 ml, the test portion, weighed to the nearest 0,001 g in the 100 ml beaker (B.3.1), shall be in accordance with the following inequality:

$$\frac{5}{a} \leqslant m \leqslant \frac{10}{a}$$

where

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

a is the supposed total acidity, expressed as a mass fraction of citric acid, in percent.

B.4.2 Determination of citric acid content

Fill the beaker containing the test portion up to the 50 ml mark with phenolphthalein solution (B.2.2).

Carry out two titrations, with stirring, with the sodium hydroxide solution (B.2.3) until the phenolphthalein turns to pink at the endpoint.

B.4.3 Expression of results

That total acidity, a, expressed as a mass fraction of anhydrous citric acid, in percent, is

$$a = \frac{M \times C_{\mathrm{s}} \times V}{3 \times m}$$

where

M is the molar mass of citric acid, i.e. 192,13 g;

C_{s} is the exact concentration of the sodium hydroxide used in the titration, in moles per litre;

V is the volume, in litres, of sodium hydroxide (titrant) required to reach the endpoint;

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

NOTE Since citric acid is a triacid, the equivalent volume for a given number of moles is three times greater than that for the same number of moles of acetic acid (a monoacid). Because the ratio of molecular masses of citric acid and acetic acid is $192{,}13/60{,}04 = 3{,}2$, the acidity expressed as citric acid (a) will give a value which is very close to that expressed as acetic acid (b), the relation can be expressed by the equation

$$a/b = 1{,}07$$

Annex C
(normative)

Determination of chloride content, expressed as sodium chloride

C.1 Introduction

This annex specifies a method for the determination of the chloride ions present in the brine of peppercorns.

The chloride ions are precipitated by titration with silver nitrate. The endpoint is determined potentiometrically using an automatic titration apparatus.

C.2 Reagents

Use only reagents of recognized analytical grade.

C.2.1 Water, distilled or demineralized or of equivalent quality, with a conductivity of less than 20 μS/cm.

C.2.2 Silver nitrate solution, of concentration 0,1 mol/l.

C.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

C.3.1 Potentiometric titration apparatus, with a silver electrode and a glass reference electrode, and suitable titration flasks.

C.3.2 Analytical balance, capable of weighing to the nearest 0,000 1 g.

C.4 Preparation of test sample

Use the brine collected after draining the peppercorns in brine for the determination of net drained mass (see annex D).

C.5 Procedure

C.5.1 Test solution

Weigh, to the nearest 0,000 1 g, a test portion of about 0,5 g in a titration flask. Dilute to about 50 ml with water (C.2.1).

C.5.2 Determination

Titrate automatically the chloride ions in the test solution (C.5.1) with silver nitrate solution (C.2.2), bearing in mind that 1 ml of this solution is equivalent to 5,844 mg of sodium chloride.

C.6 Expression of results

The chloride content, w, expressed as sodium chloride as a mass fraction in percent, is obtained from the following equation:

$$w = 0{,}584\,4 \times \frac{V_{\text{eq}}}{m}$$

where

V_{eq} is the volume of silver nitrate, in millilitres, needed to reach the endpoint;

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

Annex D
(normative)

Determination of net mass and of drained net mass

D.1 Introduction

This annex describes a method for the determination of the net mass and of the drained net mass of the peppercorns in brine.

D.2 Apparatus

Usual laboratory apparatus and, in particular, the following.

D.2.1 Flat sieve, having a square mesh size of 2,5 mm (thickness of the wire 0,85 mm) of 20 cm or 30 cm in diameter, conforming to ISO 3310-1.

D.2.2 Timer, **watch** or **chronometer**.

D.2.3 Analytical balance, capable of weighing to the nearest 0,1 g.

D.3 Procedure

Weigh, to the nearest 0,1 g, the container with the peppercorns in brine before opening (mass m). Open the container. Check the temperature of the product, which shall be 20 °C $\pm$ 5 °C.

Carry out the following operations at ambient temperature.

Use the 20 cm diameter sieve (D.2.1) for containers having a capacity less than or equal to 850 ml, or the 30 cm diameter sieve for containers having a capacity greater than 850 ml, and weigh to the nearest 1 g (mass m_1)

Put the sieve on the appropriate container. Pour the content of the container on the sieve. Start the timer (D.2.2). Incline the sieve to about 20° to the horizontal to facilitate draining. Count 2 min from the moment when the product has been poured onto the sieve.

Immediately after that, weigh to the nearest 1 g the sieve and its content (mass m_2).

Rince the empty container and its lid, dry them, then weigh them to the nearest 0,1 g (mass m_3).

Keep the brine for the determination of the sodium chloride content in accordance with annex C.

D.4 Expression of results

D.4.1 Determination of the net mass

Calculate the net mass, m_N using the equation:

$$m_N = m - m_3$$

D.4.2 Determination of the drained net mass

Determine the drained net mass, m_E, using the equation:

$$m_E = m_2 - m_1$$

ICS 67.220.10

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