INTERNATIONAL STANDARD

ISO 8312

First edition
1999-12-01

Rubber compounding ingredients — Stearic acid — Definition and test methods

Ingrédients de mélange du caoutchouc — Acide stéarique — Définition et méthodes d'essai

Reference number
ISO 8312:1999(E)

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 8312 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

It cancels and replaces ISO 8312-1:1988, which has been technically revised.

Annexes A to K form a normative part of this International Standard. Annex L is for information only.

Rubber compounding ingredients — Stearic acid — Definition and test methods

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

1.1 This International Standard defines stearic acid (including blends of stearic and palmitic acid) for use as a compounding ingredient in the rubber industry and specifies the test methods for describing its properties.

1.2 Classification of stearic acid and stearic acid/palmitic acid blends according to iodine value and typical chemical and physical properties for such materials for use in the rubber industry are given in annex L. This annex is given for information only.

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 660:1996, *Animal and vegetable fats and oils — Determination of acid value and acidity*.

ISO 662:1998, *Animal and vegetable fats and oils — Determination of moisture and volatile matter content*.

ISO 935:1988, *Animal and vegetable fats and oils — Determination of titre*.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3596-1:1988, *Animal and vegetable fats and oils — Determination of unsaponifiable matter — Part 1: Method using diethyl ether extraction (Reference method).*

ISO 3596-2:1988, *Animal and vegetable fats and oils — Determination of unsaponifiable matter — Part 2: Rapid method using hexane extraction*.

ISO 3657:1988, *Animal and vegetable fats and oils — Determination of saponification value*.

ISO 3961:1996, *Animal and vegetable fats and oils — Determination of iodine value*.

ISO 4058:1977, *Magnesium and its alloys — Determination of nickel — Photometric method using dimethyl-glyoxime*.

ISO 5508:1990, *Animal and vegetable fats and oils — Analysis by gas chromatography of methyl esters of fatty acids*.

ISO 5509:—[1], *Animal and vegetable fats and oils — Preparation of methyl esters of fatty acids*.

ISO 5794-1:1994, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*.

ISO 6685:1982, *Chemical products for industrial use — General method for determination of iron content — 1,10-Phenanthroline spectrophotometric method*.

ISO 7780:1998, *Rubber and rubber latices — Determination of manganese content — Sodium periodate photometric methods*.

ISO 8053:1995, *Rubber and latex — Determination of copper content — Photometric method*.

ISO 15528:—[2], *Paints, varnishes and raw materials for paints and varnishes — Sampling*.

3 Term and definition

For the purposes of this International Standard, the following term and definition apply:

3.1
stearic acid (for use in the rubber industry)
a mixture of straight-chain saturated fatty acids composed substantially of stearic acid in the form $C_{17}H_{35}COOH$ and palmitic acid in the form $C_{15}H_{31}COOH$

4 Sampling

Sampling shall be carried out in accordance with ISO 15528, using a stainless-steel sampling device.

5 Physical and chemical properties

The physical and chemical properties shall be determined by the methods of test listed in Table 1.

1) To be published. (Revision of ISO 5509:1978)

2) To be published. (Revision of ISO 842:1984 and ISO 1512:1991)

Table 1 — List of physical and chemical properties of stearic acid and the methods used for their determination

Property	Test method
Acid value, mg KOH/g	ISO 660
Saponification value, mg KOH/g	ISO 3657
Titre value, °C	ISO 935
Fatty acids, C_{16} – C_{18}, including unsaturates, % (*m*/*m*) total	ISO 5508 and ISO 5509
Matter volatile at 105 °C ± 3 °C, % (*m*/*m*)	ISO 662, oven method
Ash at 550 °C ± 25 °C, % (*m*/*m*)	Annex A
Iodine value, g/100 g	ISO 3961
Mineral acidity, cm^3/100 g	Annex F
Copper, mg/kg	Annex B or G[a]
Manganese, mg/kg	Annex C or H[a]
Iron, mg/kg	Annex D or J[a]
Unsaponifiable matter, % (*m*/*m*)	ISO 3596-1 or ISO 3596-2
Nickel, mg/kg	Annex E or K[a]

[a] For speed and simplicity, the methods given in annexes B, C, D and E are recommended.

Where an atomic absorption spectrometer is not available, the molecular absorption spectrometric methods given in annexes G, H, J and K may be used.

6 Test report

The test report shall include the following information:

a) all details necessary for complete identification of the product tested;

b) a reference to this International Standard (ISO 8312);

c) the results obtained:

   1) percentage ash w_A, from A.4,

   2) copper content w_{Cu}, from B.6 or from annex G (state the method used),

   3) manganese content w_{Mn}, from C.6 or from annex H (state the method used),

   4) iron content w_{Fe}, from D.6 or from annex J (state the method used),

   5) nickel content, w_{Ni}, from E.6 or from annex K (state the method used),

   6) mineral acidity, N_{ma}, from F.5,

   7) the results of other tests which may have been performed (see Table 1);

d) any unusual features noted during the determinations;

e) any operations not included in this International Standard, or in the other International Standards cited, which might have affected the results;

f) the dates of the tests.

Annex A
(normative)

Determination of ash at 550 °C ± 25 °C

A.1 Principle

A weighed test portion is carefully volatilized without ignition, and the residue is ashed in a furnace at 550 °C ± 25 °C. The mass of ash is determined as a percentage of the mass of the original test portion.

A.2 Apparatus

Ordinary laboratory apparatus, plus the following:

A.2.1 Silica crucible.

A.2.2 Heat-resistant non-conducting (insulating) material in plate form, approximately 150 mm × 150 mm.

A.2.3 Analytical balance, accurate to 0,1 mg.

A.2.4 Muffle furnace, capable of being maintained at a temperature of 550 °C ± 25 °C.

A.3 Procedure

A.3.1 Heat the clean silica crucible (A.2.1) to 600 °C, allow to cool in a desiccator and weigh empty to 0,1 mg. Place about 10 g of sample in this crucible and re-weigh to 0,1 mg. Place in a hole in the sheet of heat-resistant material (A.2.2).

A.3.2 Heat the crucible and contents gently in order to volatilize the test portion, taking care to ensure that the vapour does not ignite and that hot gases from the burner do not enter the crucible.

A.3.3 When all volatile material has been removed, place the crucible in the muffle furnace (A.2.4), maintained at 550 °C ± 25 °C, and ignite the contents for 30 min.

A.3.4 Place the crucible in a desiccator and allow to cool.

A.3.5 Re-weigh the crucible to the nearest 0,1 mg.

A.3.6 Repeat the operations specified in A.3.3, A.3.4 and A.3.5 until successive mass determinations differ by less than 2 mg.

A.3.7 Retain the ash obtained in A.3.6 if subsequent use can be made in another test.

A.4 Expression of results

Calculate the percentage ash in accordance with the equation

$$w_A = \frac{m_2 - m_1}{m_0} \times 100$$

where

w_A is the percentage ash;

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

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible and ash.

Annex B
(normative)

Determination of copper content — Atomic absorption spectrometric method

B.1 Principle

Ash made in accordance with annex A is dissolved in hydrochloric acid and the solution made up to standard volume. The absorbance is measured at 324,7 nm in an atomic absorption spectrometer. The copper content is determined by reference to a calibration graph prepared by measuring the absorbance of standard copper solutions.

B.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

B.2.1 Hydrochloric acid, 10 % (*m*/*m*) solution.

B.2.2 Copper, standard solution corresponding to 10 mg of Cu per dm^3.

B.3 Apparatus

Ordinary laboratory apparatus, plus the following:

B.3.1 Atomic absorption spectrometer, fitted with a copper hollow-cathode lamp.

B.3.2 One-mark volumetric flasks, two of capacity 10 cm^3 and six of capacity 50 cm^3, complying with the requirements of ISO 1042, class A.

B.4 Procedure

B.4.1 Obtain a sample of ash by conducting the determination specified in annex A.

B.4.2 Dissolve the ash so obtained in 5 cm^3 of dilute hydrochloric acid (B.2.1). Transfer the solution quantitatively to a 10 cm^3 one-mark volumetric flask (see B.3.2).

B.4.3 Dilute the digested ash to exactly 10 cm^3 in the one-mark volumetric flask by adding water.

B.4.4 Set the wavelength of the spectrometer (B.3.1) to 324,7 nm and aspirate the test solution into the flame, followed immediately by water, and then blank solution made up from the same reagents and using the same procedure but omitting the test portion.

B.4.5 Repeat this procedure and record the mean values of absorbance of the test solution and the blank test solution.

B.5 Preparation of the calibration graph

B.5.1 Preparation of solutions

Into a series of six 50 cm^3 one-mark volumetric flasks (see B.3.2), transfer the volumes of the standard copper solution (B.2.2) indicated in Table B.1, dilute to the mark with water and mix.

Table B.1 — Standard calibration solutions for determination of copper

Volume of standard copper solution (B.2.2) cm^3	Copper content $\mu g/cm^3$
0, 5	0,1
2,5	0,5
5,0	1,0
10,0	2,0
15,0	3,0
25,0	5,0

B.5.2 Spectrometric measurements

Aspirate each of the standard calibration solutions in turn into the flame of the atomic absorption spectrometer (B.3.1) and record their absorbances at a wavelength of 324,7 nm, following the instructions of the instrument manufacturer.

Aspirate water into the flame after each measurement.

B.5.3 Plotting the calibration graph

Plot a graph having the masses, in micrograms, of copper per cm^3 of the calibration solutions as abscissae and the corresponding values of absorbance as ordinates.

B.6 Expression of results

By reference to the calibration graph prepared as described in B.5.3, determine the copper content corresponding to the absorbances of the test solution and the blank test solution.

The concentration of copper to be determined shall fall within the linear part of the calibration curve.

The total copper content of the sample, expressed in milligrams per kilogram, is given by the equation

$$w_{Cu} = \frac{10(m_3 - m_4)}{m_0}$$

where

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

m_3 is the copper content, in micrograms per cm^3, of the test solution;

m_4 is the copper content, in micrograms per cm^3, of the blank test solution.

Express the result to the nearest 0,1 mg/kg.

Annex C
(normative)

Determination of manganese content — Atomic absorption spectrometric method

C.1 Principle

Ash made in accordance with annex A is dissolved in hydrochloric acid and the solution made up to standard volume. The absorbance is measured at 279,5 nm in an atomic absorption spectrometer. The manganese content is determined by reference to a calibration graph prepared by measuring the absorbance of standard manganese solutions.

C.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

C.2.1 Hydrochloric acid, 10 % (m/m) solution.

C.2.2 Manganese, standard solution corresponding to 10 mg of Mn per dm^3.

C.3 Apparatus

Ordinary laboratory apparatus, plus the following:

C.3.1 Atomic absorption spectrometer, fitted with a manganese hollow-cathode lamp.

C.3.2 One-mark volumetric flasks, two of capacity 10 cm^3 and six of capacity 50 cm^3, complying with the requirements of ISO 1042, class A.

C.4 Procedure

Carry out the procedure given in clause B.4 of annex B, but set the wavelength specified in B.4.4 to 279,5 nm instead of 324,7 nm.

C.5 Preparation of the calibration graph

Prepare a calibration graph for manganese following the instructions given in clause B.5 of annex B, but using the standard manganese solution (C.2.2) and recording the absorbances at 279,5 nm instead of 324,7 nm.

C.6 Expression of results

Determine the manganese content corresponding to the absorbances of the test solution and the blank test solution by reference to the calibration graph prepared as described in clause C.5.

The concentration of manganese to be determined shall fall within the linear part of the calibration curve.

The total manganese content of the sample, expressed in milligrams per kilogram, is given by the equation

$$w_{Mn} = \frac{10(m_5 - m_6)}{m_0}$$

where

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

m_5 is the manganese content, in micrograms per cm^3, of the test solution;

m_6 is the manganese content, in micrograms per cm^3, of the blank test solution.

Express the result to the nearest 0,1 mg/kg.

Annex D
(normative)

Determination of iron content — Atomic absorption spectrometric method

D.1 Principle

Ash made in accordance with annex A is dissolved in hydrochloric acid and the solution made up to standard volume. The absorbance is measured at 248,3 nm in an atomic absorption spectrometer. The iron content is determined by reference to a calibration graph prepared by measuring the absorbance of standard iron solutions.

D.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

D.2.1 Hydrochloric acid, 10 % (m/m) solution.

D.2.2 Iron, standard solution corresponding to 10 mg of Fe per dm^3.

D.3 Apparatus

Ordinary laboratory apparatus, plus the following:

D.3.1 Atomic absorption spectrometer, fitted with an iron hollow-cathode lamp.

D.3.2 One-mark volumetric flasks, two of capacity 10 cm^3 and six of capacity 50 cm^3, complying with the requirements of ISO 1042, class A.

D.4 Procedure

Carry out the procedure given in clause B.4 of annex B, but set the wavelength specified in B.4.4 to 248,3 nm instead of 324,7 nm.

D.5 Preparation of the calibration graph

Prepare a calibration graph for iron following the instructions given in clause B.5 of annex B, but using the standard iron solution (D.2.2) and recording the absorbances at 248,3 nm instead of 324,7 nm.

D.6 Expression of results

Determine the iron content corresponding to the absorbances of the test solution and the blank test solution by reference to the calibration graph prepared as described in clause D.5.

The concentration of iron to be determined shall fall within the linear part of the calibration curve.

The total iron content of the sample, expressed in milligrams per kilogram, is given by the equation

$$w_{Fe} = \frac{10(m_7 - m_8)}{m_0}$$

where

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

m_7 is the iron content, in micrograms per cm^3, of the test solution;

m_8 is the iron content, in micrograms per cm^3, of the blank test solution.

Express the result to the nearest 0,1 mg/kg.

Annex E
(normative)

Determination of nickel content — Atomic absorption spectrometric method

E.1 Principle

Ash made in accordance with annex A is dissolved in hydrochloric acid and the solution made up to standard volume. The absorbance is measured at 232,0 nm in an atomic absorption spectrometer. The nickel content is determined by reference to a calibration graph prepared by measuring the absorbance of standard nickel solutions.

E.2 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

E.2.1 Hydrochloric acid, 10 % (*m*/*m*) solution.

E.2.2 Nickel, standard solution corresponding to 10 mg of Ni per dm^3.

E.3 Apparatus

Ordinary laboratory apparatus, plus the following:

E.3.1 Atomic absorption spectrometer, fitted with a nickel hollow-cathode lamp.

E.3.2 One-mark volumetric flasks, two of capacity 10 cm^3 and six of capacity 50 cm^3, complying with the requirements of ISO 1042, class A.

E.4 Procedure

Carry out the procedure given in clause B.4 of annex B, but set the wavelength specified in B.4.4 to 232,0 nm instead of 324,7 nm.

E.5 Preparation of the calibration graph

Prepare a calibration graph for nickel following the instructions given in clause B.5 of annex B, but using the standard nickel solution (E.2.2) and recording the absorbances at 232,0 nm instead of 324,7 nm.

E.6 Expression of results

Determine the nickel content corresponding to the absorbances of the test solution and the blank test solution by reference to the calibration graph prepared as described in clause E.5.

The concentration of nickel to be determined shall fall within the linear part of the calibration curve.

The total nickel content of the sample, expressed in milligrams per kilogram, is given by the equation

$$w_{Ni} = \frac{10(m_9 - m_{10})}{m_0}$$

where

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

m_9 is the nickel content, in micrograms per cm^3, of the test solution;

m_{10} is the nickel content, in micrograms per cm^3, of the blank test solution.

Express the result to the nearest 0,1 mg/kg.

Annex F
(normative)

Determination of mineral acidity — Titrimetric method

F.1 Principle

A test portion is dissolved in light petroleum, extracted with water and the aqueous phase titrated with standard volumetric potassium hydroxide solution or standard volumetric sodium hydroxide solution.

F.2 Reagents and materials

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

F.2.1 Light petroleum distillate, boiling point 60 °C to 80 °C.

F.2.2 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,01 mol/dm^3.

F.2.3 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,01 mol/dm^3.

F.2.4 Filtered methyl orange, indicator solution.

F.3 Apparatus

Ordinary laboratory apparatus, plus the following:

F.3.1 Conical flasks, of capacity 250 cm^3 and 500 cm^3.

F.3.2 Separating funnels, of capacity 500 cm^3.

F.4 Procedure

F.4.1 Weigh about 50 g of sample into a 500 cm^3 conical flask (see F.3.1). Add 100 cm^3 of light petroleum distillate (F.2.1).

F.4.2 Warm the mixture in a fume cupboard, taking care to avoid flames.

F.4.3 Transfer the contents of the conical flask to a 500 cm^3 separating funnel (F.3.2).

F.4.4 Rinse the 500 cm^3 conical flask with 10 cm^3 of warm water at 40 °C and add to the separating funnel.

F.4.5 Immediately add a further 40 cm^3 of water to the separating funnel.

F.4.6 Swirl, but do not shake vigorously, to avoid the risk of forming an emulsion, and allow to settle.

F.4.7 Run off the lower, aqueous, layer from the separating funnel into a 250 cm^3 conical flask (see F.3.1).

F.4.8 Wash the light petroleum distillate layer remaining in the separating funnel twice with 50 cm^3 of water, each time running off the lower layer into the 250 cm^3 conical flask, the total contents of which should now measure 150 cm^3.

F.4.9 Titrate the aqueous solution in the conical flask with either potassium hydroxide solution (F.2.2) or sodium hydroxide solution (F.2.3), using filtered methyl orange indicator solution (F.2.4).

F.5 Expression of results

Calculate the mineral acidity, expressed as cm^3 of hydrochloric acid solution, c(HCl) = 0,01 mol/dm^3, per 100 g of sample, using the equation

$$N_{ma} = \frac{100\ V}{m}$$

where

V is the volume, in cm^3, of potassium hydroxide solution (F.2.2) or sodium hydroxide solution (F.2.3) used for the titration;

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

Annex G
(normative)

Determination of copper content — Molecular absorption spectrometric method

Treat the ash obtained from determinations made in accordance with annex A by the procedure given in ISO 8053 and report the results in accordance with that International Standard.