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Rubber compounding ingredients — Stearic acid —

Part 1:

Definition and test methods

Ingrédients de mélange du caoutchouc — Acide stéarique —

Partie 1: Définition et méthodes d'essai

Reference number
ISO 8312-1 : 1988 (E)

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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International Standard ISO 8312-1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

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ISO 8312 consists of the following parts, under the general title *Rubber compounding ingredients — Stearic acid*:

Part 1: Definition and test methods

Part 2: Specification

Annexes A to K form an integral part of this part of ISO 8312.

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Rubber compounding ingredients — Stearic acid —

Part 1:
Definition and test methods

WARNING — All recognized health and safety precautions shall be observed when carrying out the procedures specified in this part of ISO 8312 and in the International Standards listed in table 1.

1 Scope

This part of ISO 8312 defines stearic acid for use as a compounding ingredient in the rubber industry and specifies the test methods for determining its properties.

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2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8312. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8312 are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

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ISO 660 : 1983, *Animal and vegetable fats and oils — Determination of acid value and of acidity.*

ISO 662 : 1980, *Animal and vegetable fats and oils — Determination of moisture and volatile matter content.*

ISO 842 : 1984, *Raw materials for paints and varnishes — Sampling.*

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

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks.*

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

ISO 3596-2 : —[1], *Animal and vegetable fats and oils — Determination of unsaponifiable matter — Part 2: Rapid method using hexane extraction.*

ISO 3657 : 1977, *Animal and vegetable oils and fats — Determination of saponification value.*

ISO 3961 : 1979, *Animal and vegetable oils and fats — Determination of iodine value.*

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

ISO 5508 : 1978, *Animal and vegetable fats and oils — Analysis by gas-liquid chromatography of methyl esters of fatty acids.*

ISO 5509 : 1978, *Animal and vegetable fats and oils — Analysis by gas-liquid chromatography of methyl esters of fatty acids.*

ISO 5794-1 : 1984, *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 : 1987, *Rubber and rubber latices — Determination of manganese content — Sodium periodate photometric methods.*

ISO 8053 : 1986, *Rubber and latex — Determination of copper content — Photometric method.*

3 Definition

For the purposes of this part of ISO 8312, the following definition applies.

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 $C_{15}H_{31}COOH$.

1) To be published.

4 Sampling

Sampling shall be carried out in accordance with the procedure in ISO 842 for dry powders, using a stainless steel sampling device.

5 Physical and chemical properties

The physical and chemical properties shall be determined according to the methods of test listed in table 1.

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

Property	Test method
Acid value, mgKOH/g	ISO 660
Saponification value, mgKOH/g	ISO 3657
Titre value, °C	ISO 935
Fatty acids, C_{16} to 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[1)]
Manganese, mg/kg	Annex C or H[1)]
Iron, mg/kg	Annex D or J[1)]
Unsaponifiable matter, % (m/m)	ISO 3596-1 or ISO 3596-2
Nickel, mg/kg	Annex E or K[1)]

1) 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.

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Annex A
(normative)

Determination of ash at 550 °C ± 25 °C

A.1 Principle

A weighed sample 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 sample.

A.2 Apparatus

Ordinary laboratory apparatus and

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 1 mg.

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

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A.3 Procedure

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A.3.1 Heat the clean silica crucible (A.2.1) to 600 °C, allow to cool in a desiccator and weigh empty to 1 mg. Place about 10 g of sample in this crucible and re-weigh to 1 mg. Place into 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 for 30 min.

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

A.3.5 Reweigh the crucible to the nearest 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.

NOTE — 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 formula

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

where

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

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

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

A.5 Test report

The test report shall include the following information:

a) identification of the product tested;

b) a reference to this part of ISO 8312;

c) the results obtained and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operations not included in this part of ISO 8312 which might have affected the results.

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 cubic decimetre.

B.3 Apparatus

Ordinary laboratory apparatus and

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 test 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 (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 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 Into a series of six 50 cm^3 one-mark volumetric flasks (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

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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 cubic centimetre 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 formula

$$\frac{10\,(m_3 - m_4)}{m_0}$$

where

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

m_3 is the copper content, in micrograms per cubic centimetre, of the test solution;

m_4 is the copper content, in micrograms per cubic centimetre, of the blank test solution.

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

B.7 Test report

The test report shall include the following information:

a) identification of the product tested;

b) a reference to this part of ISO 8312;

c) the results obtained and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operations not included in this part of ISO 8312 which might have affected the results.

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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 cubic decimetre.

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C.3 Apparatus

Ordinary laboratory apparatus and

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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

Conduct the procedure given in clause B.4 of annex B, but set the wavelength specified in B.4.4 to 279,5 nm in lieu 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 in lieu 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 formula

$$\frac{10\,(m_5 - m_6)}{m_0}$$

where

m_0 is the mass in grams of the test portion;

m_5 is the manganese content, in micrograms per cubic centimetre, of the test solution;

m_6 is the manganese content, in micrograms per cubic centimetre, of the blank test solution.

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

C.7 Test report

The test report shall include the following information:

a) identification of the product tested;

b) a reference to this part of ISO 8312;

c) the results obtained and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operations not included in this part of ISO 8312 which might have affected the results.

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 cubic decimetre.

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D.3 Apparatus

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Ordinary laboratory apparatus and

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

Conduct the procedure given in clause B.4 of annex B, but set the wavelength specified in B.4.4 to 248,3 nm in lieu 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 in lieu 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 formula

$$\frac{10\,(m_7 - m_8)}{m_0}$$

where

m_0 is the mass in grams of the test portion;

m_7 is the iron content, in micrograms per cubic centimetre, of the test solution

m_8 is the iron content, in micrograms per cubic centimetre, of the blank test solution.

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

D.7 Test report

The test report shall include the following information:

a) identification of the product tested;

b) a reference to this part of ISO 8312;

c) the results obtained and the units in which they are expressed;

d) any unusual features noted during the determination;

e) any operations not included in this part of ISO 8312 which might have affected the results.