
**Rubber — Determination of metal content
by atomic absorption spectrometry —**

Part 5:

Determination of iron content

*Caoutchouc — Dosage du métal par spectrométrie d'absorption
atomique —*

Partie 5: Dosage du fer

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6101-5 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 6101-5:1990), which has been revised to update the normative references.

ISO 6101 consists of the following parts, under the general title *Rubber — Determination of metal content by atomic absorption spectrometry*:

- *Part 1: Determination of zinc content*
- *Part 2: Determination of lead content*
- *Part 3: Determination of copper content*
- *Part 4: Determination of manganese content*
- *Part 5: Determination of iron content*

Rubber — Determination of metal content by atomic absorption spectrometry —

Part 5: Determination of iron content

1 Scope

This part of ISO 6101 specifies an atomic absorption spectrometric method for the determination of the iron content of rubbers.

It is applicable to raw rubber, rubber products and latex having iron contents of 5,0 mg/kg to 1 000 mg/kg. Higher concentrations may be determined, provided that suitable adjustments are made to the mass of the test portion and/or the concentrations of the solution used.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 123, *Rubber latex — Sampling*

ISO 247:2006, *Rubber — Determination of ash*

ISO 648:1977, *Laboratory glassware — One-mark pipettes*

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

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 Principle

A test portion is ashed at $550\text{ °C} \pm 25\text{ °C}$ in accordance with ISO 247:2006, method A or B. The ash is dissolved in hydrochloric acid and if any silicates are present they are decomposed with a mixture of sulfuric acid and hydrofluoric acid to remove them.

The solution obtained is aspirated into an atomic absorption spectrometer and the absorption is measured at a wavelength of 248,3 nm for concentrations up to 10 mg/kg or 0,001 % (by mass), or 372,0 nm for concentrations of 10 mg/kg to 1 000 mg/kg or 0,001 % (by mass) to 0,1 % (by mass).

NOTE ISO 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*, defines the spectrometric terms used in this part of ISO 6101.

4 Reagents

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

4.1 Sulfuric acid, $\rho = 1,84 \text{ Mg/m}^3$.

4.2 Hydrochloric acid, $\rho = 1,19 \text{ Mg/m}^3$.

4.3 Hydrochloric acid, dilute, 1 + 3 (by volume).

Dilute 1 volume of concentrated hydrochloric acid (4.2) with 3 volumes of water.

4.4 Hydrofluoric acid, $\rho = 1,13 \text{ Mg/m}^3$ [38 % to 40 % (by mass)].

4.5 Nitric acid, $\rho = 1,42 \text{ Mg/m}^3$.

4.6 Iron, standard solution corresponding to 1 g of Fe per cubic decimetre.

Use either commercially available standard iron solutions, or prepare as follows:

Grind metallic iron, purity greater than 99 % (by mass). Weigh 1 g to the nearest 0,01 mg in a 250 cm³ conical flask (5.11) and dissolve it in a mixture of 100 ml of 1 + 3 hydrochloric acid (4.3) and 10 ml of nitric acid (4.5). Transfer it to a 1 000 cm³ one-mark volumetric flask (5.4), dilute to the mark with 1 + 3 hydrochloric acid (4.3) and mix thoroughly.

1 cm³ of this standard solution contains 1 000 µg of Fe.

5 Apparatus

Use ordinary laboratory apparatus and the following, ensuring that all apparatus and laboratory implements are non-ferrous:

5.1 Balance, accurate to 0,01 mg.

5.2 Muffle furnace, capable of being maintained at 550 °C ± 25 °C.

5.3 Beaker, of capacity 250 cm³.

5.4 One-mark volumetric flasks, glass-stoppered, of capacity 50 cm³, 100 cm³, 200 cm³, 500 cm³ and 1 000 cm³, complying with the requirements of ISO 1042:1998, class A.

5.5 Filter funnel.

5.6 Crucible, of silica or porcelain, of capacity 150 cm³.

5.7 Crucible, of platinum, of capacity 50 cm³ to 150 cm³.

5.8 Platinum rod, as stirrer.

5.9 Filter paper, ashless, of diameter 150 mm.

5.10 Electrical heating plate or gas burner with sand bath.

5.11 Conical flask, of capacity 250 cm³.

5.12 Atomic absorption spectrometer, fitted with a hollow-cathode lamp capable of emitting radiation of the required wavelengths and a burner fed with acetylene and compressed air. A high-brightness lamp is advisable. The instrument shall be operated in accordance with the manufacturer's instructions for optimum performance.

Alternatively, an electrothermal atomization device (graphite furnace) may be used. It shall be operated by a competent person in accordance with the manufacturer's instructions for optimum performance.

5.13 Volumetric pipettes, of capacity 5 cm³, 10 cm³, 20 cm³, 50 cm³ and 100 cm³, complying with the requirements of ISO 648:1977, class A.

5.14 Watch glasses, for covering the crucibles (5.6 and 5.7).

5.15 Heated water bath.

6 Sampling

Carry out sampling as follows:

- raw rubber, in accordance with ISO 1795;
- latex, in accordance with ISO 123;
- products, to be representative of the whole sample.

7 Procedure

WARNING — All recognized health and safety precautions shall be observed when carrying out the procedures specified in this part of ISO 6101.

7.1 Preparation of test portion

7.1.1 Wash the test portion with 1 + 3 hydrochloric acid (4.3) to remove surface contamination, wash with water and allow to dry in a desiccator.

7.1.2 Weigh, to the nearest 0,01 mg, 1,0 g of milled or cut rubber into a crucible (5.6 or 5.7), using a non-ferrous cutting implement. If the iron content is about 5 mg/kg, it may be necessary to increase the mass of the test portion to 5 g maximum to obtain more reliable absorbances.

7.1.3 If the rubber contains silicates, proceed in accordance with 7.1.2 but use the platinum crucible (5.7).

7.2 Preparation of test solution

7.2.1 Destruction of organic matter

Ash in accordance with method A or B of ISO 247:2006 in the muffle furnace (5.2) maintained at 550 °C ± 25 °C. If the ash is black, caused by small amounts of carbon black, stir carefully with the platinum rod (5.8) and continue heating.

7.2.2 Dissolution of inorganic residue

After ashing, allow the crucible and its contents to cool to ambient temperature. Add carefully 20 cm³ of 1 + 3 hydrochloric acid (4.3) to the ash and heat for 30 min on a water bath (5.15). Cover with a watch glass (5.14) to avoid losses, for example due to carbonates, which cause splashing and foaming.

Transfer the solution and the residue to a beaker (5.3) with 50 cm³ of 1 + 3 hydrochloric acid (4.3) and heat for 30 min.

If the residue dissolves completely, transfer to a 100 cm³ volumetric flask (5.4), dilute to the mark with 1 + 3 hydrochloric acid (4.3) and proceed in accordance with 7.4.

If the ash is not totally dissolved, i.e. silicates are present, ash a new portion in accordance with 7.1.3 and 7.2.1. Add a few drops of sulfuric acid (4.1) and heat to fuming. Cool and add a further three drops of sulfuric acid and 5 cm³ of hydrofluoric acid (4.4). Heat on the electric heating plate or sand bath (5.10) in a fume cupboard and evaporate to dryness, while stirring with the platinum rod (5.8). Repeat this procedure twice.

Cool, and add 50 cm³ of 1 + 3 hydrochloric acid (4.3). Cover with a watch glass (5.14) and heat for 10 min. Filter, washing the filter with 1 + 3 hydrochloric acid (4.3), and combine the filtrate and washings in a 100 cm³ volumetric flask (5.4). Dilute to the mark with 1 + 3 hydrochloric acid (4.3) and mix thoroughly. Proceed in accordance with 7.4.

7.3 Preparation of the calibration curves

7.3.1 Preparation of standard calibration solutions

7.3.1.1 Primary calibration solution

Pipette carefully 10 cm³ of the standard iron solution (4.6) into a 1 dm³ one-mark volumetric flask (5.4) and dilute to the mark with 1 + 3 hydrochloric acid (4.3).

1 cm³ of this primary calibration solution contains 10 µg of Fe.

7.3.1.2 Standard calibration solutions

Into a series of eight 100 cm³ one-mark volumetric flasks (5.4), place the volumes of the primary calibration solution (7.3.1.1) as indicated in Table 1, add 1 + 3 hydrochloric acid (4.3) to the mark and mix thoroughly.

Table 1 — Volume of primary calibration solution with corresponding concentration of iron

Volume of primary calibration solution (7.3.1.1) cm ³	Corresponding concentration of iron µg/100 cm ³
100	1 000
50	500
20	200
10	100
5	50
2 ^a	20 ^a
1 ^a	10 ^a
0 ^b	0

^a Only if the sensitivity of the spectrometer is high enough for accurate results.
^b Calibration blank solution.

7.3.2 Spectrometric measurements

Switch on the spectrometer (5.12) sufficiently in advance to ensure stabilization. Adjust the wavelength to 248,3 nm or 372,0 nm, and the sensitivity and the slit aperture according to the characteristics of the instrument.

Adjust the pressure of the air and of the acetylene according to the characteristics of the aspirator/burner in order to obtain a clear, reducing flame.

Adjust the flow rate according to the characteristics of the instrument.

Aspirate the series of standard calibration solutions (7.3.1.2) in succession into the flame, and measure the absorbance of each solution twice, averaging the readings. Take care to ensure that the rate of aspiration is constant throughout this process. It should be ensured that at least one standard is at or below the level corresponding to the rubber being tested.

It is important that water be aspirated through the burner after each measurement.

7.3.3 Plotting the calibration curves

Plot two curves (one for each of the concentration ranges given below) having, for example, the masses, in micrograms, of iron contained in 100 cm³ of the standard calibration solutions as abscissae from

- a) 10 µg/100 cm³ to 100 µg/100 cm³ for 248,2 nm,
- b) 100 µg/100 cm³ to 1 000 µg/100 cm³ for 372,0 nm,

and the corresponding values of absorbance, corrected for the absorbance of the calibration blank solution (see Table 1), as ordinates.

Represent the points on the graph by the best straight line as judged visually, or calculated by the least-square fit method.

7.4 Determination

7.4.1 Spectrometric measurements

Carry out duplicate spectrometric measurements at a wavelength of 248,3 nm or 372,0 nm on the test solution (7.2.2), following the procedures specified in 7.3.2.

7.4.2 Dilution

If the instrument response for the test solution is greater than that found for the standard calibration solution having the highest iron content (see Table 1), dilute as appropriate with 1 + 3 hydrochloric acid (4.3) in accordance with the following procedure.

Pipette carefully a volume V of the test solution (7.2.2) into a 100 cm³ one-mark volumetric flask so that the iron concentration lies within the range covered by the standard calibration solutions. Dilute to the mark with 1 + 3 hydrochloric acid (4.3). Repeat the measurement.

7.4.3 Blank determination

Carry out a blank determination in parallel with the determination, using 1 + 3 hydrochloric acid (4.3), but omitting the test portion.

If sulfuric acid and hydrofluoric acid were used for the test portion preparation, the same quantities of these acids shall be used in the preparation of the blank determination solution.

8 Expression of results

8.1 Read the iron content of the test solution directly from the appropriate calibration graph (see 7.3.3).

The iron content of the test portion, expressed in parts per million by mass, is given by the formula:

$$\frac{\rho_{\text{Fe,t}} - \rho_{\text{Fe,b}}}{100m} \times f \times 10^4$$

where

$\rho_{\text{Fe,t}}$ is the iron concentration, in micrograms per 100 cm³, of the test solution (7.2.2), read from the calibration graph;

$\rho_{\text{Fe,b}}$ is the iron concentration, in micrograms per 100 cm³, of the blank determination solution (7.4.3), read from the calibration graph;