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**Rubber, raw, vulcanised —  
Determination of metal content by  
ICP-OES**

*Caoutchouc brut, vulcanisé — Dosage de la teneur en métaux par  
ICP-OES*

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ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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## Introduction

The ICP-OES (inductively coupled plasma – optical emission spectrophotometer) instrument is used to determine the concentrations of certain elements in materials of interest. The main advantage of ICP-OES over the AAS (atomic absorption spectroscopy) techniques in general is its multi-element capability, its longer linear dynamic ranges and fewer condensed phase interferences. In addition, besides the refractory compound-forming elements, elements such as iodine, phosphorus and sulfur are detected with more sensitivity by the ICP-OES technique. ICP-OES is also known as ICP-AES (inductively coupled plasma – atomic emission spectrophotometer).

ICP-OES was first introduced as a technique for trace elemental analysis. The technique experiences the least interference of any of the commonly used analytical atomic spectrometry techniques. Chemical interferences are largely eliminated by the high temperature of the plasma. Physical interferences can be compensated for by taking advantage of the ICP's multi-element capability.

In ICP-OES, the light emitted by the excited atoms and ions in the plasma is measured to obtain information about the sample. Because the excited species in the plasma emit light at several different wavelengths, the emission from the plasma is *polychromatic*. This polychromatic radiation has to be separated into individual wavelengths so the emission from each excited species can be identified and its intensity can be measured without interference from emission at other wavelengths.

An important feature of the ICP that is not common to most other emission sources is that since the sample aerosol is introduced through the centre of the ICP, it can be surrounded by the high temperature plasma for a comparatively long time, approximately 2 ms. It is this long residence time of the analyte particles in the centre of the plasma that is largely responsible for the lack of matrix interferences in the ICP.

The determination described in this International Standard is important with respect to product safety and the environment. ICP-OES is a state-of-the art instrument for accurate detection of the trace metals in raw and vulcanized samples of rubber including latex.

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# Rubber, raw, vulcanised — Determination of metal content by ICP-OES

**WARNING 1** — Persons using this International Standard should be familiar with normal laboratory practice. This International 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.

**WARNING 2** — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

## 1 Scope

This International Standard describes the method of determination of both major and trace levels of metal contents in rubber — raw, vulcanized — by ICP-OES.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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* <https://standards.iteh.ai/catalog/standards/sist/90f49a5a-b1c4-4e12-917d-71b14f62715d/iso-123-2015>

ISO 124, *Latex, rubber — Determination of total solids content*

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

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

## 3 Principle

An aqueous sample is converted to an aerosol via a nebulizer. The aerosol is transported to the inductively coupled plasma which has a high temperature zone (8 000 °C to 10 000 °C). The analytes are heated (excited) to different (atomic and/or ionic) states and produce characteristic optical emissions. These emissions are separated based on their respective wavelengths and their intensities are measured (spectrometry). The intensities are proportional to the concentrations of analytes in the aqueous sample. The quantification is carried out via an external multipoint linear standardization obtained by comparing the emission intensity of an unknown sample with that of a standard sample. A process flow of atomization of sample in ICP-OES is shown in [Annex B](#).

## 4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or deionized water or water of equivalent purity.

**4.1 Ultra-pure grade of concentrated nitric acid**, density 1,42 g/ml.

**4.2 Ultra-pure grade of concentrated hydrochloric acid**, density 1,19 g/ml.

- 4.3 **Ultra-pure grade of concentrated sulfuric acid**, density 1,83 g/ml.
- 4.4 **Multi-element primary standard solution**, traceable to NIST (National Institute of Standards and Technology) or any national metrological institute (NMI).
- 4.5 **Single-element primary standard solutions**, traceable to NIST or any NMI.
- 4.6 **Ammonium oxalate saturated solution**.
- 4.7 **Yttrium solution**, 1 000 µg/g.
- 4.8 **Diluted nitric acid**.
- 4.9 **Diluted HCl**.

## 5 Apparatus

Usual laboratory apparatus and glassware and, in particular, the following.

**5.1 ICP-OES system.** A portion of the photons emitted by the ICP is collected with a lens or a concave mirror. This focusing optic forms an image of the ICP on the entrance aperture of a wavelength selection device such as a monochromator. The particular wavelength exiting the monochromator is converted to an electrical signal by a photodetector. The signal is amplified and processed by the detector electronics, then displayed and stored by a personal computer. Different components of the instruments are shown in [Figure 1](#). A description of the different components is given in [5.1.1](#) to [5.1.4](#).

**5.1.1 Test solution introduction**, to produce a steady aerosol of very fine droplets. There are three basic parts to the sample introduction system as listed in [5.1.1.1](#) to [5.1.1.3](#).

**5.1.1.1 Peristaltic pump**, to draw up the test solution and deliver it to the nebulizer.

**5.1.1.2 Nebulizer**, to convert the solution to an aerosol that is sent to the spray chamber.

**5.1.1.3 Spray chamber**, to filter out the large, uneven droplets from the aerosol.

**5.1.2 Energy source**, to get atoms sufficiently energized such that they emit light. There are three basic parts to the energy source as listed in [5.1.2.1](#) to [5.1.2.3](#).

**5.1.2.1 Radio frequency generator**, to generate an oscillating electro-magnetic field at a frequency of 27,12 MHz. This radiation is directed to the load coil. Equipment are also available with electro-magnetic field at a frequency of 40,68 MHz.

**5.1.2.2 Load coil**, to deliver the radiation to the torch.

**5.1.2.3 Torch**, having argon flowing through it which will form a plasma in the RF field.

**5.1.3 Spectrometer**, to diffract the white light from the plasma into wavelengths. There are several types of spectrometers used for ICP. Regardless of type, all of them use a diffraction grating.

**5.1.4 Detector**, to measure the intensity of the wavelengths. The detector is a silicon chip that is composed of many individual photo-active sections called "picture elements". These picture elements, or pixels, will build up charge as photons impinge on them. Individual pixels are of a size such that they can be used to measure individual wavelengths.



- 5.2 Analytical balance, capable of weighing to the nearest  $\pm 0,1$  mg.
- 5.3 Micro syringe, calibrated.
- 5.4 Hot plate.
- 5.5 Pipettes, of different capacities i.e. 10 ml, 20 ml.
- 5.6 Micro-Kjeldahl digestion flask.
- 5.7 Muffle furnace.
- 5.8 TFM (tetrafluoroethylene modified) vessel.
- 5.9 Microwave digestion system.
- 5.10 Sample rack of autosampler.
- 5.11 Filter paper, Whatman No. 44.
- 5.12 Silica or platinum dish.

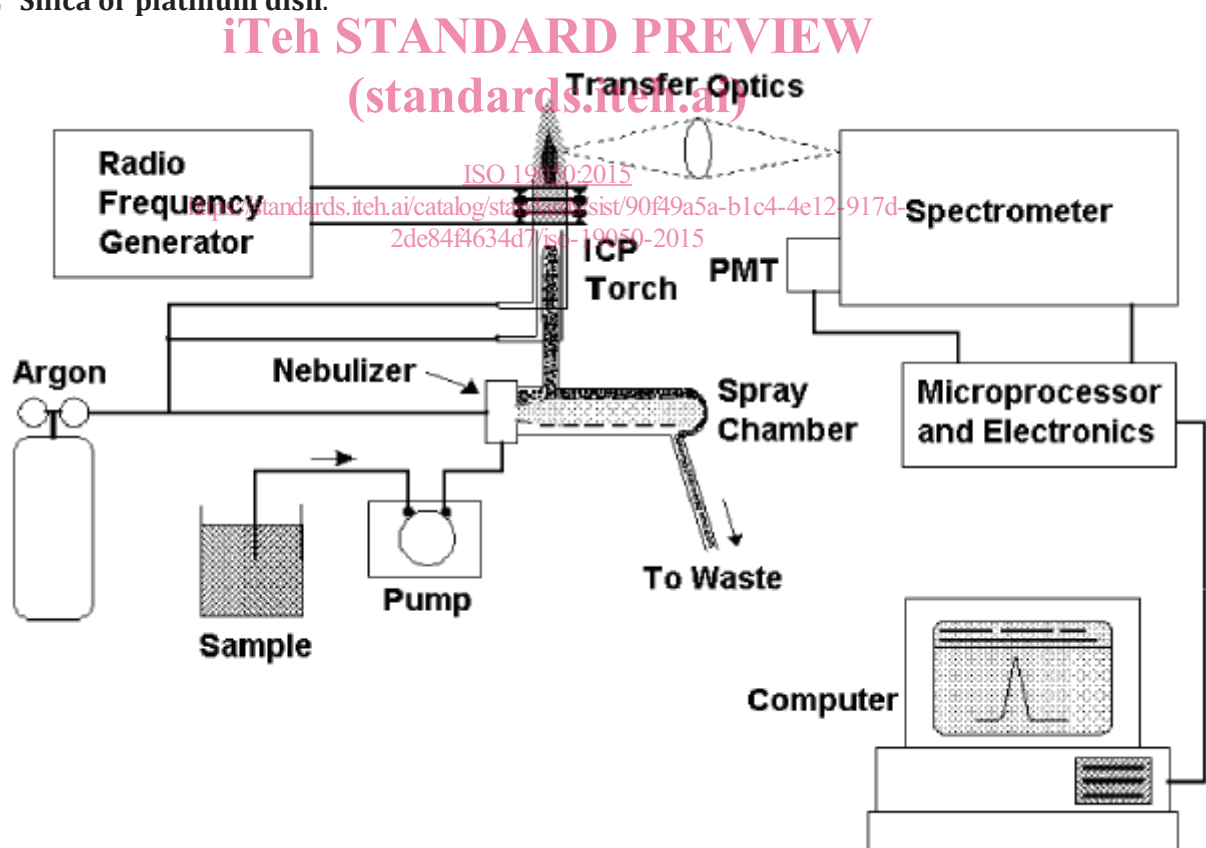


Figure 1 — Different components of a typical ICP-OES instrument

## 6 Calibration

The instrument shall be calibrated in accordance with the schedule given in [Annex A](#).

## 7 Sample preparation

### 7.1 General

There are four major steps involved in the analysis of metal contents.

- a) obtaining a representative laboratory sample from the bulk received for testing;
- b) decomposition of organic matter;
- c) separation and concentration of the element of interest;
- d) determination.

### 7.2 Sampling

For solid rubber, take the laboratory sample and prepare the test piece in accordance with the method specified in ISO 1795.

For latex, take the laboratory sample in accordance with ISO 123 and prepare the dry latex test piece as specified in ISO 124.

### 7.3 Decomposition of organic matter

The commonly used methods of destruction of organic matter are:

- a) wet oxidation (see [7.3.1](#));
- b) dry ashing (see [7.3.2](#));
- c) microwave digestion (see [7.3.3](#)).

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#### 7.3.1 Wet oxidation

**7.3.1.1** Transfer 0,5 g to 1,0 g of the test piece into a micro-Kjeldahl digestion flask.

**7.3.1.2** Add 20 ml concentrated nitric acid and up to 20 ml water. Boil the contents of the flask to reduce the volume to 20 ml. Cool the solution, add 10 ml of concentrated sulfuric acid and boil again. Add further small quantities of nitric acid whenever the contents begin to blacken. When the addition of nitric acid is no longer necessary (i.e. when the liquid no longer blackens) continue heating till white fumes are evolved.

**7.3.1.3** At this stage, cool the solution and add 10 ml of saturated ammonium oxalate solution and again boil until white fumes are again produced. The oxalate treatment assists in removing yellow coloration due to nitro compounds, so that the final solution is colourless. Make the final volume of 50ml in a polypropylene bottle using deionized water.

**7.3.1.4** Blanks shall be prepared with the same quantities of the reagents as are used in the test but omitting the test portion.

Precaution should be taken during the wet oxidation procedure in case of titanium and tin.

#### 7.3.2 Dry ashing

**7.3.2.1** Weigh accurately 0,5 g to 1,0 g of the test piece in a tared silica or platinum dish.

**7.3.2.2** Heat the dish firstly by means of a soft flame or hot plate to volatilise as much organic matter as possible, then transfer it to a temperature controlled muffle furnace held at about 300 °C.

**7.3.2.3** Once the material has dried and charred, increase the temperature to 550 °C and ash at this temperature till no carbon remains. If it is suspected that all carbon has not been removed, cool the ash, add about 1 ml to 2 ml of concentrated nitric acid, evaporate to dryness and heat again in the muffle furnace.

**7.3.2.4** After ashing is complete, remove the dish from the muffle furnace, cool, cover the dish with a watch glass, and gently add 40 ml to 50 ml of hydrochloric acid (1+1). Rinse down the watch glass with water and heat on a steam bath for 30 min, remove the cover and rinse. Continue heating for another 30 min.

**7.3.2.5** Add 10 ml of hydrochloric acid (1+1) and water to dissolve the salts.

**7.3.2.6** Filter into a 100 ml volumetric flask using Whatman No. 44 filter paper. Wash the residue twice using dilute HCl. Make up to volume with deionized water using a microsyringe.

It is necessary to use reagents and distilled water of suitably low metal content taking into consideration that the concentrated mineral acids are generally used in amounts several times more than the sample. Even when suitable reagents are used, reagent blank determination is still necessary.

NOTE In expressions like (1+2), (1+3) etc. used with the name of a reagent, the first numeral indicates (volume/mass) of (liquid/solid) reagent and the second numeral indicates volume of water. For example HCl (1+2) means reagent prepared by mixing one volume of HCl with two volumes of water.

**7.3.2.7** Blanks shall be prepared with the same quantities of the reagents as are used in the test but omitting the test portion.

**7.3.2.8** Alternatively, dissolution of the inorganic residue using nitric acid can be carried out as described in ISO 6101.

### 7.3.3 Microwave digestion

**7.3.3.1** Place a TFM vessel on the balance plate, tare and weigh the test piece of 0,5 g to 1,0 g.

**7.3.3.2** Add 5 ml of concentrated HNO<sub>3</sub> to each vessel.

**7.3.3.3** Assemble the vessel according to the manufacturer's instructions.

**7.3.3.4** Place assembled vessels into the microwave oven according to the manufacturer's instructions.

**7.3.3.5** Program the oven with parameters that are known to digest the material safely and effectively (producing a clear digest when diluted). Recommended parameters are listed in [Table 1](#). It may be necessary to adjust these parameters to accommodate variations between individual instruments.

**Table 1 — Typical parameters for microwave digestion**

Power	1 200 W <sup>a</sup>
Ramp time	10 min
Final temperature	180 °C
Temperature hold time	10 min
Cool down time	10 min
<sup>a</sup> If there are less than eight vessels in the microwave the wattage can be lowered.	