
**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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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).

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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*.

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.

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*

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.