# INTERNATIONAL STANDARD 1657

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# Raw rubber and rubber latex — Determination of iron content — 1,10-Phenanthroline photometric method

*Élastomères bruts et latex d'élastomère — Dosage du fer — Méthode photométrique à la phénanthroline-1,10*

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## FOREWORD

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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.

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No Member Body expressed disapproval of the document.

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# Raw rubber and rubber latex — Determination of iron content — 1,10-Phenanthroline photometric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of small amounts of iron in uncompounded natural rubber, uncompounded synthetic rubbers which do not contain chlorine, and the corresponding uncompounded latices.

## 2 REFERENCES

ISO 123, *Rubber latex — Sampling.*

ISO 124, *Rubber latices — Determination of total solids content.*

ISO 1795, *Raw rubber in bales — Sampling.*

ISO 1796, *Raw rubber — Sample preparation.*

## 3 PRINCIPLE

Ashing of the raw rubber or the dried latex solids in a crucible. Extraction of the ash with hydrochloric acid and making up of the solution to standard volume. After adjustment of the pH by the addition of buffer solution, treatment of an aliquot part of the solution with hydroxylammonium chloride and 1,10-phenanthroline with which the iron forms a coloured complex. Photometric measurement of the absorbance of this solution, which is proportional to the concentration of iron.

## 4 REAGENTS

All reagents shall be of recognized high purity analytical reagent quality suitable for use in trace metal analysis. Distilled water shall be used whenever water is specified.

### 4.1 **Hydrochloric acid,** $\rho$ 1,19 g/cm$^3$ *.

### 4.2 **1,10-Phenanthroline** solution.

Dissolve 0,5 g of 1,10-phenanthroline monohydrate in hot water and dilute the solution to 500 cm$^3$.

### 4.3 **Hydroxylammonium chloride** solution.

Dissolve 10 g of hydroxylammonium chloride in 100 cm$^3$ of water.

### 4.4 Buffer solution

Dissolve 164 g of anhydrous sodium acetate in approximately 250 cm$^3$ of water and to the solution add 28,5 cm$^3$ of glacial acetic acid, $\rho$ 1,05 g/cm$^3$. Dilute this mixture to 500 cm$^3$ and filter immediately before use if it is cloudy. If this buffer solution gives highly coloured reference solutions in the preparation of the calibration curve, an alternative buffer solution may be prepared by dissolving 80 g of sodium hydroxide or 106 g of anhydrous sodium carbonate in 200 cm$^3$ of water, adding 142,5 cm$^3$ of glacial acetic acid, $\rho$ 1,05 g/cm$^3$, and diluting the solution to 500 cm$^3$.

### 4.5 **Standard iron solution** (1 cm$^3$ contains 0,1 mg of Fe).

Dissolve 0,702 1 g of ammonium iron(II) sulphate hexahydrate $[(NH_4)_2Fe(SO_4)_2.6H_2O]$ in water in a 1 000 cm$^3$ volumetric flask. Add 3 cm$^3$ of concentrated hydrochloric acid, $\rho$ 1,19 g/cm$^3$, and dilute to the mark with water. This solution should remain stable for at least 1 month.

### 4.6 **Standard iron solution for calibration** (1 cm$^3$ contains 0,01 mg of Fe).

Pipette 10 cm$^3$ of the standard iron solution (4.5) into a 100 cm$^3$ volumetric flask and dilute to the mark with water. This solution shall be freshly prepared from the stock solution (4.5) when required.

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Electrophotometer** or **spectrophotometer,** capable of measuring absorbance at approximately 510 nm, and **matched cells.**

**5.2 Silica or porcelain crucible,** nominal capacity 50 to 80 cm$^3$.

**5.3 Muffle furnace,** capable of maintaining a temperature of 525 ± 25 °C.

**5.4 Asbestos board** approximately 100 mm square and 6 mm thick, with a hole in the centre to support the crucible so that approximately two-thirds projects below the board.

**5.5 Volumetric flasks,** 50, 100, 1 000 cm$^3$.

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* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm$^3$), in accordance with a decision of the Twelfth Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes.

## 6 SAMPLING

For raw rubber, carry out the sampling in accordance with ISO 1795.

For latex, carry out the sampling in accordance with one of the methods specified in ISO 123.

## 7 PROCEDURE

### 7.1 Preparation of test portion

For the determination of iron in raw rubber, prepare a 10 g test portion in accordance with ISO 1796, to produce a thin sheet.

Alternatively, cut the 10 g test portion into small fragments each weighing approximately 0,1 g.

For the determination of iron in latex, take a portion of thoroughly mixed latex containing at least 10 g of total solids and dry to constant mass as specified in ISO 124.

*At all stages of sample preparation, take care to avoid* contamination with iron, particularly from rusty knives, scissors, mills, and other equipment.

### 7.2 Preparation of calibration curve

Make up a series of standard matching solutions in 50 cm$^3$ volumetric flasks, each containing 1 cm$^3$ of concentrated hydrochloric acid (4.1), 10 cm$^3$ of buffer solution (4.4), 1 cm$^3$ of hydroxylammonium chloride solution (4.3) and 10 cm$^3$ of 1,10-phenanthroline solution (4.2). To these solutions, add portions of the standard iron solution for calibration (4.6), ranging from 0 to 20 cm$^3$ (for example 0 – 0,5 – 5 – 10 – 15 – 20 cm$^3$) and dilute the solutions with water to the mark. Allow to stand for 15 min, then decant each solution into the cell of the electrophotometer or spectrophotometer (5.1) and measure its absorbance at the absorption maximum (about 510 nm). Correct the reading by subtracting the absorbance of the solution containing no added iron. If the absorbance is measured on a double-beam instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of each standard matching solution against that of the solution containing no added iron. Plot the reading thus obtained for each solution against the appropriate concentration of iron to give the calibration curve, which shall be checked periodically according to local conditions and the type of instrument used.

### 7.3 Determination[1]

Cut a 10 g test portion of the raw rubber or of the dried latex into small pieces. Transfer to the unetched crucible (5.2) and weigh to the nearest 0,01 g. Support the crucible in the hole cut in the asbestos board (5.4). Heat gently with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to the muffle furnace (5.3) maintained at a temperature of 525 ± 25 °C.

Alternatively, wrap the weighed sample in a piece of ashless filter paper about 150 mm in diameter and transfer to the crucible. Place the crucible and its contents into the furnace at 525 ± 25 °C and close the furnace door.

The furnace door must not be opened during the first hour because of the risk of igniting inflammable gases.

When all the carbon has been oxidized remove the crucible and allow to cool.

In parallel with the determination and following the same procedure, carry out a blank determination using similar filter paper and crucible and using the same quantities of all the reagents as used for the determination.

Add 5 cm$^3$ of hydrochloric acid (4.1) and 5 cm$^3$ of water to the crucible and digest the mixture on a steam bath for 30 to 60 min. If the solution has a deep yellow colour, indicating the presence of much iron, add a further 5 cm$^3$ of the hydrochloric acid and digest for a further 30 min. Filter the solution through a sintered glass crucible (porosity grade 2 or 3), collect the filtrate in a 50 cm$^3$ volumetric flask (5.5) and dilute to the mark.

Transfer an aliquot part of this solution containing not more than 2 cm$^3$ of hydrochloric acid (4.1) to a 50 cm$^3$ volumetric flask. Add 10 cm$^3$ of the buffer solution (4.4), then 1 cm$^3$ of hydroxylammonium chloride solution (4.3) and 10 cm$^3$ of 1,10-phenanthroline solution (4.2). Dilute to the mark with water and allow to stand at room temperature for 15 min.

Measure the absorbance of the solution at the wavelength used for preparing the calibration curve. Correct the reading for the test solution by subtracting the value for the absorbance of the blank solutions. If the absorbance is measured on a double-beam instrument, place the cell containing the blank solution in the reference beam and measure the absorbance of the test solution against the blank solution.

## 8 EXPRESSION OF RESULTS

By means of the calibration curve, determine the concentration of iron corresponding to the corrected reading, and from this calculate the iron content of the test portion.

Express the result as parts per million (ppm) of iron (Fe) calculated by mass.

## 9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

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[1] All precautions and safeguards required for the carrying out of trace metal analysis must be observed.