International Standard 1657

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, raw and rubber latex — Determination of iron content — 1,10-Phenanthroline photometric method

Caoutchouc brut et latex de caoutchouc — Dosage du fer — Méthode photométrique à la phénanthroline-1,10

Second edition — 1986-10-15

UDC 678.031/.032 : 543.42/.43 : 546.72 **Ref. No. ISO 1657-1986 (E)**

Descriptors : rubber, crude rubber, latex, chemical analysis, determination of content, iron, photometric analysis.

Price based on 3 pages

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.

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.

International Standard ISO 1657 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

This second edition cancels and replaces the first edition (ISO 1657-1975), clause 5 and sub-clause 7.3 of which have been technically revised.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Rubber, raw 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 5 to 1 000 mg/kg of iron in uncompounded natural rubber, uncompounded synthetic rubbers which do not contain chlorine, and in the corresponding uncompounded latices.

2 References

ISO 123, *Rubber latex — Sampling.*

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

ISO 247, *Rubber — Ash content — Determination.*

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

ISO 1796, *Raw rubber — Sample preparation.*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

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 to reduce any iron(III) present to iron(II) and 1,10-phenanthroline with which the iron(II) forms an orange-red complex. Photometric measurement of the absorbance of this solution, which is proportional to the concentration of iron.

NOTE — The method used is closely similar to that given in ISO 6685, *Chemical products for industrial use — General method for determination of iron content — 1,10-Phenanthroline spectrophotometric method.*

4 Reagents

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

4.1 Hydrochloric acid, ϱ 1,19 g/cm³*.

4.2 1,10-Phenanthroline, solution.

Dissolve 0,5 g of 1,10-phenanthroline monohydrate in hot water and, after cooling, provided that this does not cause precipitation, dilute the solution to 500 cm³.

Store the solution away from the light and use only colourless solutions.

4.3 Hydroxylammonium chloride, solution.

Dissolve 10 g of hydroxylammonium chloride in 100 cm³ of water.

4.4 Buffer solution.

Dissolve 164 g of anhydrous sodium acetate in approximately 250 cm³ of water and to the solution add 28,5 cm³ of glacial acetic acid, ϱ 1,05 g/cm³. Dilute this mixture to 500 cm³ 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 cm³ of water, adding 142,5 cm³ of glacial acetic acid, ϱ 1,05 g/cm³, and diluting the solution to 500 cm³.

4.5 Iron, standard solution corresponding to 0,1 g of Fe per cubic decimetre.

Dissolve 0,702 g, weighed to the nearest 0,000 5 g, of ammonium iron(II) sulfate hexahydrate [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$] in water in a 1 000 cm³ one-mark volumetric flask. Add 3 cm³ of concentrated hydrochloric acid, ϱ 1,19 g/cm³, and dilute to the mark with water.

This solution will generally remain stable for at least 1 month.

1 cm³ of this standard solution contains 0,1 mg of Fe.

* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm³), 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.

4.6 Iron, standard solution for calibration corresponding to 0,01 g of Fe per cubic decimetre.

Pipette 10 cm^3 of the standard iron solution (4.5) into a 100 cm^3 one-mark volumetric flask and dilute to the mark with water.

1 cm^3 of this standard solution contains 0,01 mg of Fe.

This solution shall be freshly prepared from the stock solution (4.5) at the time of use.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Colorimeter or **spectrophotometer**, capable of measuring absorbance at approximately 510 nm, with matched 5 cm 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 Heat-resistant board, iron-free, approximately 100 mm square and 6 mm thick, with a hole in the centre, or **silica triangle**. The hole in the heat-resistant board or the size of the silica triangle shall be such that approximately two-thirds of the crucible projects below the board.

5.5 One-mark volumetric flasks, of capacity 50 cm^3.

5.6 Graduated precision pipettes or **burettes**.

5.7 Sintered glass crucible, of porosity P 40 or P 100 (see ISO 4793).

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 homogenized test piece

For the determination of iron in raw rubber, prepare a homogenized sheet in accordance with ISO 1796.

For the determination of iron in latex, prepare a homogeneous sheet dried to constant mass, as specified in ISO 124, using a portion of thoroughly mixed latex containing at least 10 g of total solids.

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

7.2 Preparation of calibration curve

7.2.1 Preparation of standard colorimetric solutions

To a series of 50 cm^3 one-mark volumetric flasks (5.5), add portions of the standard iron solution (4.6) ranging from 0 to 20 cm^3 (for example 0; 0,5; 5; 10; 15; 20 cm^3). These portions contain 0 to 200 µg of iron.

7.2.2 Colour development

Add to each of the flasks in 7.2.1, 1 cm^3 of the concentrated hydrochloric acid (4.1), 10 cm^3 of the buffer solution (4.4), 1 cm^3 of the hydroxylammonium chloride solution (4.3) and 10 cm^3 of the 1,10-phenanthroline solution (4.2). Dilute the solutions with water to the mark and mix thoroughly. Allow to stand at room temperature for 15 min.

7.2.3 Photometric measurements

Measure the absorbance of the solutions using the colorimeter or spectrophotometer (5.1) at the absorption maximum (about 510 nm) using the matched 5 cm cells.

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.

7.2.4 Plotting the graph

Plot the reading thus obtained for each solution of 7.2.1 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

WARNING — All precautions and safeguards required for the carrying out of trace metals analysis must be observed.

7.3.1 Preparation of test solution

Cut into small pieces, each weighing approximately 0,1 g, a 10 g test portion of the homogenized test piece (7.1), prepared from the raw rubber or the dried latex. Transfer to the unetched crucible (5.2) and weigh to the nearest 0,01 g. Support the crucible in the hole cut in the heat-resistant board (5.4). Heat gently with a small[1] gas flame until a dry carbonaceous residue remains and then transfer the crucible to the muffle furnace (5.3), maintained at a temperature of 550 ± 25 °C.

1) Overheating may cause loss of iron when silica crucibles are used.

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 in the furnace at 550 ± 25 °C and close the furnace door.

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

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

Add 5 cm^3 of the 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 (5.7), transfer the filtrate to a 50 cm^3 one-mark volumetric flask (5.5) and, after cooling, dilute to the mark.

7.3.2 Colour development

Transfer an aliquot part of the test solution (7.3.1) containing not more than 2 cm^3 of hydrochloric acid (4.1), or 400 µg of iron to a 50 cm^3 one-mark volumetric flask. Add 10 cm^3 of the buffer solution (4.4), then 1 cm^3 of the hydroxylammonium chloride solution (4.3) and 10 cm^3 of the 1,10-phenanthroline solution (4.2). Dilute the solution with water to the mark and mix thoroughly. Allow to stand at room temperature for 15 min.

7.3.3 Blank test

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

7.3.4 Photometric measurements

Carry out the photometric measurements on the test solution (7.3.1), and on the blank test solution (7.3.3) after colour development, measuring 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) reference to this International Standard;

b) identification of the test sample;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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