International Standard 8053

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Rubber and latex — Determination of copper content — Photometric method

Caoutchouc et latex — Dosage du cuivre — Méthode photométrique

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8053 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

It cancels and replaces International Standard ISO 1396-1975 and ISO Recommendation R 1654-1971, of which it constitutes a technical revision.

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.

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Rubber and latex — Determination of copper content — Photometric method

0 Introduction

Copper in certain forms is known to catalyse the oxidative breakdown of natural rubber although the mechanism by which degradation is brought about is not fully understood. It is recognized also that other forms of copper can be present in the rubber even in relatively large amounts, without degradation taking place, but in these cases there is always the possibility that under the influence of some chemicals, notably the unsatured acids, the copper could assume a more aggressive oxidative catalytic role.

Clearly it would be an advantage to distinguish analytically between catalytically active and inactive forms, but no generally accepted method has yet been put forward for doing so. There is no alternative therefore but to determine the total amount of copper in the rubber. The method described here is a combination of ISO/R 1654-1971 and ISO 1396-1975.

The method specified in this International Standard is applicable to all the commonly used elastomers, including those containing chlorine.

1 Scope and field of application

This International Standard specifies a photometric method for the determination of trace amounts of copper in raw rubber, latices and compounded rubber, both natural and synthetic.

This method may be applied to rubbers containing silica, provided that treatment with hydrofluoric acid is included in the procedure.

The method is sensitive down to 1 mg/kg copper.

2 References

ISO 123, *Rubber latex — Sampling.*

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

ISO 247, *Rubber — Determination of ash.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

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

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

ISO 1796, *Rubber, raw — Sample preparation.*

ISO 4661/2, *Rubber, vulcanized — Preparation of test pieces — Part 2: Chemical tests.*

3 Principle

The rubber sample is subjected to ashing or digestion in a mixture of concentrated sulfuric and nitric acids, followed by removal of excessive amounts of calcium (if present) and complexing of any iron present with ammonium citrate. After making alkaline, the aqueous solution is shaken with a solution of diethyldithiocarbamate in 1,1,1-trichloroethane to form and extract the yellow copper complex. Spectrophotometric measurement of this solution, and comparison of the result with those for standard matching solutions, permit the quantitative determination of copper.

4 Reagents

All precautions and safeguards for carrying out trace metal analysis shall be observed. All recognized health and safety precautions shall be observed when carrying out the procedures specified in this International Standard.

For the analysis, only reagents of recognized analytical grade and only distilled water or water of equivalent purity shall be used.

4.1 Sodium sulfate, anhydrous.

4.2 Sulfuric acid, concentrated, ϱ = 1,84 g/cm^3. [1)]

1) The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the 12th Conférence Générale des Poids et Mesures. Glassware with either designation is acceptable for use in this International Standard.

4.3 Nitric acid, concentrated, ϱ = 1,42 g/cm³.

4.4 Hydrochloric acid/nitric acid mixture, prepared as follows:

Mix together

2 volumes of hydrochloric acid, ϱ = 1,18 g/cm³
1 volume of nitric acid, ϱ = 1,42 g/cm³
3 volumes of water

4.5 Hydrogen peroxide, 30 %.

4.6 Ammonia solution, ϱ = 0,890 g/cm³.

4.7 Hydrochloric acid, c(HCl) = 5 mol/l.

4.8 Hydrofluoric acid, ϱ = 1,13 g/cm³.

4.9 Citric acid solution.

Dissolve 50 g of citric acid in 100 cm³ of water.

4.10 Zinc diethyldithiocarbamate reagent.

Dissolve 1 g of solid zinc diethyldithiocarbamate in 1 000 cm³ of 1,1,1-trichloroethane. If zinc diethyldithiocarbamate is not available, the reagent may be prepared thus: Dissolve 1 g of sodium diethyldithiocarbamate in water and add 2 g of zinc sulfate heptahydrate. Extract the resulting zinc diethyldithiocarbamate by shaking with 100 cm³ of 1,1,1-trichloroethane in a separating flask. Separate the 1,1,1-trichloroethane layer and dilute to 1 000 cm³ with 1,1,1-trichloroethane.

This reagent is stable for at least 6 months when stored in a bottle of non-actinic glass.

4.11 Copper, standard solution, containing 0,01 g of Cu per cubic decimetre.

Weigh 0,393 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) into a small beaker and dissolve in water. Add 3 cm³ of the concentrated sulfuric acid (4.2), transfer the solution to a 1 000 cm³ volumetric flask (5.6) and dilute with water to the mark to form the stock solution. Pipette 10 cm³ of this stock solution into a 100 cm³ volumetric flask (5.6) and dilute with water up to the reference mark.

1 cm³ of this solution contains 0,01 mg of Cu.

Prepare freshly from the stock solution when required.

4.12 Magnesium oxide.

4.13 Litmus paper.

4.14 Filter paper (hardened paper, resistant to acids).

5 Apparatus

Ordinary laboratory apparatus and

5.1 Photometer or **spectrophotometer,** capable of measuring absorbance at approximately 435 nm, with matched cells, usually 10 mm path length.

5.2 Kjeldahl flask, of capacity 100 cm³, of silica or borosilicate glass.

5.3 Crucibles or dishes of porcelain, silica or platinum, of capacity 50 cm³ for small samples, appropriately larger for large samples.

Porcelain or silica ware, especially if etched, should be lined with about 0,1 g of magnesium oxide, distributed over the base and partly up the sides. This minimizes the possibility that copper will be adsorbed on the etched walls and on fillers (if present) and instead the copper will now be preferentially adsorbed on the magnesium oxide. Platinum ware need not be treated with magnesium oxide.

5.4 Pipette, 25 cm³, complying with the requirements of ISO 648.

5.5 Balance, accurate to 0,1 mg.

5.6 One-mark volumetric flasks, 100 cm³ and 1 000 cm³, complying with the requirements of ISO 1042.

5.7 Electric heating plate or **gas burner with sand bath.**

5.8 Platinum rod, as stirrer.

6 Sampling, sample preparation and selection of test portion

6.1 For raw rubber, carry out the sampling in accordance with ISO 1795 and prepare the sample in accordance with ISO 1796.

6.2 For latex, carry out the sampling in accordance with one of the methods specified in ISO 123 and sample preparation in accordance with ISO 124.

6.3 For rubber compounds, carry out the sampling to obtain as representative a sample of the compound as possible. See ISO 4661/2.

6.4 Weigh, to the nearest 0,001 g, a test portion of between 2 and 10 g, selected from 6.1, 6.2 or 6.3.

NOTE — The size of the test portion depends upon the amount of copper present. It should be selected to give an absorbance reading of 0,3 to 0,8 absorbance units, or in the case of very low amounts of copper, at least 10 times the absorbance reading of a blank. Appropriate test portion size will depend largely upon prior experience.