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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

**ISO RECOMMENDATION
R 1396**

**DETERMINATION OF COPPER IN COMPOUNDED RUBBER
(VULCANIZED AND UNVULCANIZED)**

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1396, *Determination of copper in compounded rubber (vulcanized and unvulcanized)*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1396 which was circulated to all the ISO Member Bodies for enquiry in December 1967. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Ireland	Sweden
Austria	Israel	Switzerland
Czechoslovakia	Italy	U.A.R.
France	Japan	United Kingdom
Greece	Netherlands	U.S.A.
Hungary	New Zealand	Yugoslavia
India	Poland	
Iran	Spain	

The following Member Body opposed the approval of the Draft :

Germany

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

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DETERMINATION OF COPPER IN COMPOUNDED RUBBER (VULCANIZED AND UNVULCANIZED)

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 compound even in relatively large amounts, without degradation taking place, but in these cases there is always the possibility that under the influence of some constituents of the compound, notably the unsaturated acids, the copper could assume a more aggressive 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 to determining the total amount of copper in the rubber compound.

Little is known concerning the influence of copper on the catalytic oxidation of synthetic rubbers, although it is widely accepted that its effect is less severe than is the case with natural rubber. Possibly for this reason the determination of copper in compounds based on the synthetic rubbers is less frequently carried out; nevertheless the method recommended in this ISO Recommendation is applicable to all the commonly used elastomers. Furthermore, since the method is insensitive to chlorine, it may be applied to raw polymers which contain chlorine.

1. SCOPE

This ISO Recommendation describes a procedure for the determination of trace amounts of copper in compounded rubber, both natural and synthetic. It may also be used for the determination of copper in raw polymers and in latices which contain chlorine, but is not applicable to rubber having a saturated polymer chain or butyl rubber.

2. PRINCIPLE OF METHOD

Two grammes of the rubber are digested with concentrated sulphuric and nitric acids. Excessive amounts of calcium are removed as sulphate and any iron present is complexed with ammonium citrate. After being made alkaline, the aqueous solution is shaken with a measured volume of a chloroform solution of zinc diethyl-dithiocarbamate and the yellow copper complex formed is extracted. The colour of this solution is measured photometrically and is proportional to the concentration of copper.

3. REAGENTS

All reagents should conform to recognized analytical reagent quality suitable for use in trace metal analysis. Distilled water should be used whenever water is specified, unless otherwise stated.

- 3.1 *Sodium sulphate*, anhydrous.
- 3.2 *Sulphuric acid*, concentrated, ρ 1.84 (g/ml).
- 3.3 *Nitric acid*, concentrated, ρ 1.42 (g/ml).
- 3.4 *Hydrogen peroxide*, 300 g/l.
- 3.5 *Ammonia solution*, ρ 0.890 (g/ml).
- 3.6 *Hydrochloric acid*, 5 N.
- 3.7 *Citric acid solution*. Dissolve 50 g of citric acid in 100 ml of water.
- 3.8 *Zinc diethyldithiocarbamate*. Dissolve 1 g of solid zinc diethyldithiocarbamate in 1000 ml of chloroform. If zinc diethyldithiocarbamate is not available the reagent may be prepared thus: 1 g of sodium diethyldithiocarbamate is dissolved in water to which 2 g of zinc sulphate are then added. The resulting zinc diethyldithiocarbamate is extracted by shaking with 100 ml of chloroform and the chloroform solution is separated and diluted to 1000 ml.
Stored in an amber coloured bottle, this reagent is stable for at least six months.
- 3.9 *Standard copper solution*. Weigh 0.393 g of copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) into a small beaker and dissolve in water. Add 3 ml of concentrated sulphuric acid. Transfer the solution to a 1000 ml volumetric flask and dilute with water to the mark to form the stock solution. Pipette 10 ml of this stock solution into a 100 ml volumetric flask and dilute with water to the mark.
This solution contains the equivalent of 0.01 mg of copper per millilitre and should be freshly prepared from the stock solution when required.

4. APPARATUS

- 4.1 *Electrophotometer, absorptiometer, or spectrophotometer* capable of measuring optical density at approximately 435 nm.
- 4.2 *Kjeldahl flask*, 100 ml capacity, silica or borosilicate glass.

5. PREPARATION OF TEST PORTION

At least 2 g of rubber should be cut from the sample, if necessary from more than one place, so that proper representation of the whole sample is achieved. Homogenize the piece or pieces comprising the test portion by passing a few times between the cold rolls of a laboratory mill to produce a thin sheet or rough crumb. Alternatively the test portion may be prepared by cutting the rubber into smaller portions each weighing approximately 0.1 g. For the determination of copper in latex prepare a specimen of the total solids weighing at least 2 g from the latex. Digestion of this specimen can be facilitated by passing it six times between the cold rolls of a laboratory mill, rolling the rubber into a cylinder after each pass and presenting the cylinder end-on to the rolls for the next pass.

At all stages of sample preparation care should be taken to avoid contamination.