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ISO RECOMMENDATION

R 1654

RAW RUBBER AND RUBBER LATEX

DETERMINATION OF COPPER

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BRIEF HISTORY

The ISO Recommendation R 1654, *Raw rubber and rubber latex – Determination of copper*, 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. 1645, which was circulated to all the ISO Member Bodies for enquiry in July 1968. It has been approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Spain
Austria	India	Sweden
Brazil	Iran	Switzerland
Canada	Israel	Thailand
Ceylon	Italy	Turkey
Colombia	Japan	U.A.R.
Czechoslovakia	Korea, Rep. of	United Kingdom
France	Netherlands	U.S.A.
Germany	New Zealand	U.S.S.R.
Greece	Poland	

No Member Body opposed the approval of the Draft.

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

ISO Recommendation R 1654 August 1971

RAW RUBBER AND RUBBER LATEX

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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 without degradation taking place, but no generally accepted method is available for distinguishing between the active and inactive forms. At present, therefore, there is no alternative to determining the total amount of copper in the rubber.

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 synthetic rubbers is less frequently carried out; nevertheless this ISO Recommendation is applicable to most of the commonly used synthetic elastomers.

1. SCOPE

This ISO Recommendation describes a method suitable for the quantitative determination of small amounts of copper in raw natural rubber, raw synthetic elastomers which do not contain chlorine, and the corresponding uncompounded latices.

For compounded rubber and for rubbers and latices which contain chlorine, the methods given in ISO Recommendation R 1396, *Determination of copper in compounded rubber (vulcanized and unvulcanized)*, should be used.

2. PRINCIPLE

5 g of the dried latex solids or of the raw rubber are ashed in a silica crucible. The ash is extracted with a hydrochloric/nitric acid mixture and the solution made alkaline with ammonium hydroxide. Any iron present is complexed with ammonium citrate. The aqueous solution is then shaken with a solution in chloroform of zinc diethyldithiocarbamate to form and extract the yellow copper complex. The optical density of this solution is measured photometrically and is proportional to the concentration of copper.

3. REAGENTS

All reagents should be of recognized high purity analytical reagent quality suitable for use in trace metal analysis.

Distilled water should be used whenever water is specified.

3.1 *Light magnesium oxide.*

3.2 *Sodium sulphate*, anhydrous.

3.3 *Hydrochloric acid/nitric acid* mixture, prepared as follows

Mix together :

2 volumes of hydrochloric acid, ρ 1.18 g/ml;
1 volume of nitric acid, ρ 1.42 g/ml;
3 volumes of water.

3.4 *Citric acid solution.*

Dissolve 50 g of citric acid (solid) in 100 ml of water.

3.5 *Ammonia solution*, ρ 0.890 g/ml.

3.6 *Litmus paper.*

3.7 *Zinc diethyldithiocarbamate reagent.*

Dissolve 1 g of solid zinc diethyldithiocarbamate in 1 litre of chloroform.

If zinc diethyldithiocarbamate is not available the reagent may be prepared as follows :

Dissolve 1 g of sodium diethyldithiocarbamate in water and add 2 g of zinc sulphate. Extract the resulting zinc diethyldithiocarbamate by shaking with 100 ml of chloroform and separate the chloroform solution. Dilute to 1 litre. Store in an amber coloured bottle; this reagent is stable for at least six months.

3.8 *Copper standard solution.*

Weigh 0.393 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) into a small beaker and dissolve in water. Add 3 ml of concentrated sulphuric acid. Transfer the solution to a 1000 ml one-mark volumetric flask and dilute to the mark with water to form the stock solution. Pipette 10 ml of this stock solution into a 100 ml one-mark volumetric flask and dilute to the mark with water. This solution contains the equivalent of 0.01 mg of Cu 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 *Matched absorption cells*, 10 to 50 mm in path length.

4.3 *Silica crucibles*, nominal capacity 50 or 80 ml.

4.4 *Muffle furnace*, capable of maintaining a temperature of 550 ± 25 °C.

5. PREPARATION OF TEST PORTION

For the determination of copper in rubber, cut at least 5 g from the sample in such a way 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. Alternatively the test portion may be prepared by cutting the constituent pieces into smaller portions each weighing approximately 0.1 g.