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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1655

RAW RUBBER AND RUBBER LATEX

DETERMINATION OF MANGANESE

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1655, *Raw rubber and rubber latex – Determination of manganese*, 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. 1655, which was circulated to all the ISO Member Bodies for enquiry in December 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

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.

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RAW RUBBER AND RUBBER LATEX

DETERMINATION OF MANGANESE

INTRODUCTION

Manganese 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 recognised also that other forms of manganese 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 manganese in the rubber.

Little is known concerning the influence of manganese 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 manganese 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 gives a method suitable for the quantitative determination of small amounts of manganese in raw natural rubber, raw synthetic elastomers which do not contain chlorine, and the corresponding uncompounded latices.

For compounded rubber the methods given in ISO Recommendation R 1397, *Determination of manganese in compounded rubber*, should be used.

2. PRINCIPLE

10 g of the dried latex solids or of the raw rubber are ashed in a silica crucible, and the ash is treated with potassium hydrogen sulphate and sulphuric acid to convert the manganese to a soluble form. After dissolving the ash in dilute sulphuric acid, any iron present is complexed with orthophosphoric acid and the manganese oxidized to permanganate by boiling with potassium periodate. The absorbance of the solution is measured photometrically and is proportional to the concentration of manganese.

3. REAGENTS

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

3.1 *Potassium hydrogen sulphate.*

3.2 *Potassium periodate.*

3.3 *Sulphuric acid, $\rho = 1.84 \text{ Mg/m}^3$.*

3.4 *Orthophosphoric acid, 85 to 90 % H_3PO_4 .*

3.5 *Sulphuric acid, dilute.*

Mix 1 volume of concentrated sulphuric acid (3.3) with 19 volumes of water.

3.6 *Potassium permanganate, approximately 0.001 N solution.*

3.7 *Stabilized water.*

Dissolve about 0.1 g of potassium permanganate in 1 litre of water to which a few drops of sulphuric acid have been added. Distil the water through an effective spray trap, discarding the first and last 50 ml of distillate. Collect the rest of the distillate and store in a glass-stoppered bottle.

3.8 *Standard manganese solution.* Either of the following solutions may be used :

- (a) Prepare an approximately 0.1 N solution of potassium permanganate and standardize against sodium oxalate. Transfer the calculated amount of this standardized solution to contain 0.720 g of KMnO_4 to a small beaker and acidify with 2 ml of sulphuric acid. Add sulphur dioxide-saturated water until the solution is colourless. Boil the solution for 15 minutes, cool, transfer to a 500 ml volumetric flask and dilute to the mark with stabilized water. Pipette 20 ml of this stock solution into a second 500 ml volumetric flask and again dilute to the mark with stabilized water. This dilute solution contains the equivalent of 0.02 mg of manganese per millilitre and should be freshly prepared from the stock solution when required.
- (b) Weigh 0.770 g of manganese sulphate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) into a small beaker and dissolve in water containing 2 ml of sulphuric acid. Transfer the solution to a 500 ml volumetric flask and dilute to the mark. This solution should be stable for at least a month. Pipette 20 ml of this stock solution into a second 500 ml volumetric flask and again dilute to the mark. This dilute solution contains the equivalent of 0.02 mg of manganese 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 525 nm.

4.2 *Silica crucibles*, nominal capacity 80 ml.

4.3 *Muffle furnace*, capable of maintaining a temperature of $550 \pm 25 \text{ }^\circ\text{C}$.