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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

**ISO RECOMMENDATION
R 1655**

RAW RUBBER AND RUBBER LATEX

DETERMINATION OF MANGANESE

1st EDITION

May 1971

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

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(standards.iteh.ai)

ISO/R 1655:1971

<https://standards.iteh.ai/catalog/standards/sist/71803966-0922-4d62-a62d-dd0deee728f1/iso-r-1655-1971>

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.

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 $550 \pm 25 \text{ }^\circ\text{C}$.

5. PREPARATION OF TEST PORTION

For the determination of manganese in rubber, cut at least 10 g from the sample in such a way that proper representation of the whole is achieved. Treat the piece or pieces comprising the test portion in accordance with ISO Recommendation R 1796, *Raw rubber – Preparation of samples**.

For the determination of manganese in latex, take a portion of thoroughly mixed latex containing about 5 g of total solids and dry to constant mass as described in ISO Recommendation R 124, *Determination of total solids of latex***.

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

6. PREPARATION OF CALIBRATION CURVE

NOTE. – All precautions and safeguards required for the carrying out of trace metal analysis must be observed.

Prepare a series of standard solutions, each containing 20 ml of dilute sulphuric acid (3.5), 3 ml of orthophosphoric acid (3.4), and potassium hydrogen sulphate (3.1) equal in amount to that used in ashing the test portion. To these solutions, add portions of the standard manganese solution ranging from 0 to 15 ml, followed in each case by 0.3 g of potassium periodate (3.2). Bring the solutions to the boil, maintain at boiling point for 10 minutes, and hold at 90 °C or above for a further 10 minutes to ensure full development of the permanganate colour. Cool each solution, transfer to a 50 ml volumetric flask and dilute to the mark with stabilized water (3.7).

Rinse the cell of the electrophotometer, absorptiometer or spectrophotometer first with approximately 0.001 N potassium permanganate solution (3.6), then with stabilized water (3.7), and finally with the appropriate standard solution. Fill the cell with the standard solution and measure the optical density at the absorption maximum (about 525 nm). Correct the reading by subtracting the optical density of the solution containing no added manganese. If the optical density is measured on a double beam or null point instrument, the cell containing the blank solution should be placed in the reference beam and the optical density of each standard solution measured against that of the solution containing no added manganese. Plot the reading thus obtained for each solution against the appropriate concentration of manganese to give the calibration curve, which should be checked periodically according to local conditions and the type of instrument used.

7. PROCEDURE

NOTE. – All precautions and safeguards required for the carrying out of trace metal analysis must be observed.

Weigh, to the nearest 10 mg, a 10 g portion of the raw rubber or about 5 g of the dried latex solids, cut into small pieces, and place in a crucible (4.2) which is supported in a hole cut in an asbestos board so that about two-thirds of the crucible project below the asbestos. Commence a blank determination at the same time using a similar crucible and give identical treatment throughout to the test and blank determinations. Heat the crucible and contents with a small gas flame until a dry carbonaceous residue remains and then transfer the crucible to a muffle furnace (4.3) at a temperature of 550 ± 25 °C and heat until all carbon has been oxidized. Remove the crucible and allow to cool. Add concentrated sulphuric acid (3.3), delivered drop by drop from a pipette with a fine jet, round the sides of the crucible in amount just sufficient to moisten the ash. Fume off the excess acid by gentle heating and replace the crucible in the muffle furnace at 550 ± 25 °C to remove the last traces of carbon.

After cooling, give the ash a further treatment with sulphuric acid, adding the acid as before from a pipette, but heating only until fuming ceases in order to retain the ash as far as possible in the form of sulphate. Cool the crucible. Add 2 to 3 g of potassium hydrogen sulphate (3.1) and, supporting the crucible on the asbestos board, apply strong heat from a burner until a clear melt is obtained. Cool the crucible and contents.

* At present at the stage of Draft ISO Recommendation.

** 2nd edition, 1966.