

45

INTERNATIONAL STANDARD **ISO** 1655



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Raw rubber and rubber latex — Determination of manganese content — Potassium periodate photometric method

Caoutchoucs bruts et latex de caoutchouc — Dosage du manganèse — Méthode photométrique au periodate de potassium

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45 has reviewed ISO Recommendation R 1655 and found it technically suitable for transformation. International Standard ISO 1655 therefore replaces ISO Recommendation R 1655-1971 to which it is technically identical.

ISO Recommendation R 1655 was approved by the Member Bodies of the following countries :

Australia	India	Spain
Austria	Iran	Sri Lanka
Belgium	Israel	Sweden
Brazil	Italy	Switzerland
Canada	Korea, Dem. P. Rep. of	Thailand
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	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 expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1655 into an International Standard.

Raw rubber and rubber latex – Determination of manganese content – Potassium periodate photometric method

0 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 recognized 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, the method specified in this International Standard is applicable to most of the commonly used synthetic elastomers.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a potassium periodate photometric method for the 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 1397, *Compounded rubber – Determination of manganese content – Sodium periodate photometric method*, should be used.

2 REFERENCES

ISO 123, *Rubber latex – Sampling*.

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

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

ISO 1796, *Raw rubber – Sample preparation*.

3 PRINCIPLE

Ashing, in a silica crucible, of the dried latex solids or of the raw rubber. Treatment of the ash with potassium hydrogen sulphate and sulphuric acid to convert the manganese to a soluble form. After dissolution of the ash in

dilute sulphuric acid, complexing of any iron present with orthophosphoric acid and oxidation of the manganese to permanganate by boiling with potassium periodate. Photometric measurement of the absorbance of this solution, which is proportional to the concentration of manganese.

4 REAGENTS

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

4.1 Potassium hydrogen sulphate.

4.2 Potassium periodate.

4.3 Sulphuric acid, ρ 1,84 g/cm³.

4.4 Sulphuric acid, dilute.

Mix 1 volume of concentrated sulphuric acid (4.3) with 19 volumes of water.

4.5 Orthophosphoric acid, 85 to 90 % H₃PO₄.

4.6 Stabilized water.

Dissolve about 0,1 g of potassium permanganate in 1 dm³ 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 cm³ of distillate. Collect the rest of the distillate and store in a glass stoppered bottle.

4.7 Potassium permanganate, approximately 0,001 N solution.

4.8 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 KMnO₄ to a small beaker and acidify with 2 cm³ of sulphuric acid. Add sulphur dioxide-saturated water until the solution is colourless. Boil the solution for 15 min, cool, transfer to a 500 cm³ volumetric flask and dilute to the mark with stabilized water. Pipette 20 cm³ of this stock solution