INTERNATIONAL STANDARD 1397

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Compounded rubber — Determination of manganese content — Sodium periodate photometric method

Mélanges à base de caoutchoucs — Dosage du manganèse — Méthode photométrique au periodate de sodium

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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 1397 and found it technically suitable for transformation. International Standard ISO 1397 therefore replaces ISO Recommendation R 1397-1970 to which it is technically identical.

ISO Recommendation R 1397 was approved by the Member Bodies of the following countries :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Germany

The Member Body of the following country disapproved the transformation of ISO/R 1397 into an International Standard :

Germany

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Compounded rubber — Determination of manganese content — Sodium 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 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 manganese 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 therefore no alternative to determining the total amount of manganese in the rubber compound.

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 compounds based on the synthetic rubbers is less frequently carried out; nevertheless, the methods specified in this International Standard are applicable to all the commonly used elastomers except those which contain chlorine.

Of the two specified procedures, the first, known as the general method, is believed to be applicable to all compounded rubbers not containing chlorine. In this method, the ash from the rubber is taken through a fusion stage in order to obtain the manganese in a soluble form; it is most suited to those rubber compounds which contain heavy loadings of inert fillers such as clay, or materials which form insoluble phosphates, for example titanium dioxide. The second, known as the restricted method, is shorter and suitable for all other rubber compounds and will probably be more frequently used.

SECTION ONE : DETERMINATION OF MANGANESE — GENERAL METHOD

1 SCOPE AND FIELD OF APPLICATION

Section one of this International Standard specifies a photometric method using sodium periodate for the determination of trace amounts of manganese in compounded or vulcanized rubbers which do not contain chlorine. The method is not intended for compounded rubber latex. It is not affected by heavy loadings of fillers such as synthetic and natural silicates, calcium carbonate in various forms, or by the presence of compounding ingredients which form an insoluble phosphate under the conditions of the test.

2 REFERENCES

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

ISO 1796, *Raw rubber — Sample preparation.*

3 PRINCIPLE

Ashing of the rubber in a platinum crucible. Fusion of the ash with sodium fluoroborate, After treatment with dilute sulphuric and nitric acids, removal of the insoluble matter and oxidation of the manganese to permanganate by boiling with sodium 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 analytical reagent quality suitable for use in trace metal analysis. Distilled water shall be used whenever water is specified, unless otherwise stated.

4.1 Sodium fluoroborate

If analytical grade is not available, this reagent shall be prepared as follows: Dissolve 110 g of technical grade sodium fluoroborate in 100 cm^3 * of water warmed to about 35 °C. After filtering through paper, cool the solution to room temperature and add 100 cm^3 of 96 % ethanol while stirring. Filter the crystalline precipitate onto paper in a Buchner funnel and drain thoroughly under slight vacuum, then transfer to a shallow porcelain or glass dish and dry at about 50 °C under vacuum.

4.2 Sodium periodate.

4.3 Sulphuric acid, concentrated, ρ 1,84 g/cm^3.

4.4 Nitric acid, concentrated, ρ 1,42 g/cm^3.

* The term millilitre (ml) is commonly used as a special name for the cubic centimetre (cm^3), in accordance with a decision of the Twelfth Conférence Générale des Poids et Mesures. The term millilitre is acceptable, in general, for references in International Standards to capacities of volumetric glassware and to liquid volumes.