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**Phthalic anhydride for industrial use — Methods of test —
Part X : Determination of 1,4-naphthaquinone content —
Colorimetric method**

*Anhydride phtalique à usage industriel — Méthodes d'essai —
Partie X : Dosage de la naphtaquinone-1,4 — Méthode colorimétrique*

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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 in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1389-1970 and found it technically suitable for transformation. The technical committee, however, divided the recommendation into eleven parts (ISO 1389, parts I to XI), which therefore replace ISO Recommendation R 1389-1970, to which they are technically identical.

ISO Recommendation R 1389 had been approved by the member bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Ireland	Sweden
Cuba	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Portugal	
Hungary	Romania	

No member body had expressed disapproval of the Recommendation.

The member bodies of the following countries disapproved the transformation of the Recommendation into an International Standard :

France
Netherlands

Phthalic anhydride for industrial use — Methods of test — Part X : Determination of 1,4-naphthaquinone content — Colorimetric method

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1389 specifies a colorimetric method for the determination of the 1,4-naphthaquinone content of phthalic anhydride for industrial use.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Development of a pink colour by heating a test portion containing 1,4-naphthaquinone with tin(II) chloride, and comparison of the colour produced with a series of prepared reference colours.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

3.1 Tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$).

3.2 Phthalic anhydride [$\text{C}_6\text{H}_4(\text{CO})_2\text{O}$] free from naphthaquinones.

3.3 1,4-Naphthaquinone ($\text{C}_{10}\text{H}_6\text{O}_2$).

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Series of boiling tubes, of length 160 mm and of diameter 16 mm.

4.2 Electrically heated bath, of capacity about 2 l, containing a mixture of 53 % (*m/m*) potassium nitrate (KNO_3), 40 % (*m/m*) sodium nitrite (NaNO_2) and 7 % (*m/m*) sodium nitrate (NaNO_3), and capable of being controlled at 210 to 215 °C.

5 PREPARATION OF REFERENCE COLOURS

Prepare two mixtures of the phthalic anhydride (3.2) and the 1,4-naphthaquinone (3.3) containing 0,1 and 0,001 % (*m/m*) of naphthaquinone respectively. From these, prepare, by addition of the phthalic anhydride, mixtures containing 0,000 01 — 0,000 05 — 0,000 1 — 0,000 5 — 0,001 — 0,005 and 0,01 % (*m/m*) of 1,4-naphthaquinone.

Transfer respectively an 8 g portion of the phthalic anhydride (3.2) and 8 g portions of each of the seven prepared mixtures to eight of the boiling tubes (4.1), add 1 g of the tin(II) chloride (3.1) to each and heat in the same way as the test portion (see clause 6).

6 PROCEDURE

Weigh 8 g of the test sample into one of the boiling tubes (4.1) and add 1 g of the tin(II) chloride (3.1). Place the tube and its contents in the electrically heated bath (4.2), controlled at 210 to 215 °C, for exactly 2 min, making sure that the contents of the tube are below the level of the liquid in the bath.

NOTE — The water of crystallization of the tin(II) chloride is evolved after about 45 s heating and may cause spluttering. This may be prevented by removing the tube from the bath immediately the contents have melted and shaking the tube until the evolution of water vapour has subsided. The tube should then be replaced in the bath for the remainder of the 2 min heating period.

Remove the tube from the bath and allow the contents to crystallize by cooling in air. Rinse the outside of the tube with water and dry it. Compare the colour of the contents with the series of reference colours prepared in accordance with clause 5.

7 EXPRESSION OF RESULTS

Report the 1,4-naphthaquinone content as equal to that of the reference colour that corresponds most closely to that of the test mixture.

ANNEX

ISO PUBLICATIONS RELATING TO PHTHALIC ANHYDRIDE FOR INDUSTRIAL USE

ISO 1389/I – General.

ISO 1389/II – Measurement of colour of molten material.

ISO 1389/III – Measurement of colour stability.

ISO 1389/IV – Measurement of colour after treatment with sulphuric acid.

ISO 1389/V – Determination of free acidity – Potentiometric method.

ISO 1389/VI – Determination of phthalic anhydride content – Titrimetric method.

ISO 1389/VII – Determination of maleic anhydride content – Polarographic method.

ISO 1389/VIII – Determination of ash.

ISO 1389/IX – Determination of impurities oxidizable in the cold by potassium permanganate – Iodometric method.

ISO 1389/X – Determination of 1,4-naphthaquinone content – Colorimetric method.

ISO 1389/XI – Determination of iron content – 2,2'-Bipyridyl photometric method.