INTERNATIONAL STANDARD ISO 787/XIX

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General methods of test for pigments – Part XIX

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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.

International Standard ISO 787/XIX (originally ISO/DIS 2807) was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and circulated to the Member Bodies in April 1972.

It has been approved by the Member Bodies of the following countries :

Austria	Israel	Sweden
Brazil	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Romania	U.S.A.
India	South Africa, Rep. of	U.S.S.R.
Ireland	Spain	

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

Canada

Printed in Switzerland

The purpose of this International Standard is to establish a series of general test methods for pigments which are suitable for all or many of the individual pigments for which specifications might be required. In such cases, a cross-reference to the general method should be included in the International Standard relating to that pigment, with a note of any detailed modifications which might be needed in view of the special properties of the pigment in question.

Committee ISO/TC 35 decided that all the general methods should be published as they become available, as parts of a single International Standard, in order to emphasize the relationship of each to the whole series.

The Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment, there would be no objection to including more than one of them in the ISO series. In such cases it will, however, be essential to state clearly in a specification which method is to be used and, in the test report, which method has been used.

Parts of the series already published are as follows :

Part I : Comparison of colour
Part II : Determination of matter volatile at 105 °C
Part III : Determination of matter soluble in water (Hot extraction method)
Part IV : Determination of acidity or alkalinity of the aqueous extract
Part V : Determination of oil absorption value
Part VI : Determination of residue on sieve (Oil method)
Part VII : Determination of residue on sieve (Water method)
Part VIII : Determination of matter soluble in water (Cold extraction method)
Part IX : Determination of pH value of an aqueous suspension
Part X : Determination of density relative to water at 4 °C
Part XI : Determination of tamped volume
Part XII : Visual comparison of hue of powered white pigment (Hollow cone method)
Part XIII : Determination of water-soluble sulphates, chlorides and nitrates
Part XIV : Determination of resistivity of aqueous extract
Part XV : Comparison of resistance of coloured pigments of similar types to light from a specified light source
Part XVI : Comparison of relative tinting strength (or equivalent colouring value) and colour on reduction in linseed stand oil using the automatic muller
Part XVII : Comparison of lightening power of white pigments
Part XVIII : Determination of residue on sieve by a mechanical flushing procedure

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General methods of test for pigments – Part XIX : Determination of water-soluble nitrates (Salicylic acid method)

0 INTRODUCTION

This document is a part of ISO 787, *General methods of test for pigments.*

1 SCOPE AND FIELD OF APPLICATION

Part XIX of this International Standard specifies a general method of test for determining the water-soluble nitrates in a sample of pigment by a spectrophotometric method using salicylic acid.

Part XIII specifies a method for determining the water-soluble nitrates in a sample of pigment using Nessler's method.

NOTES

1 When this general method is applicable to a given pigment, a cross-reference to it will be included in the International Standard relating to that pigment, with a note of any detailed modifications which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment will a special method for determination of nitrates be specified.

2 It should be noted that the two methods may not necessarily give the same result, and work is being carried out to compare the two methods.

2 REFERENCE

ISO 842, *Raw materials for paints and varnishes – Sampling.*

3 PRINCIPLE

The nitrate present in the extract of the pigment sample is used to nitrate salicylic acid in sulphuric acid medium. The nitro-compound formed is of an intense yellow colour in alkaline solution and the colour is measured spectrophotometrically at a wavelength of 410 nm.

4 REAGENTS

All reagents shall be of recognized analytical reagent quality. Distilled water or water of equivalent purity shall be used.

4.1 Sulphuric acid, ρ 1,84 g/ml.

4.2 Sulphuric acid, 5 N.

4.3 Ethanol, 95 % (*V/V*).

4.4 Sodium salicylate, 5 g/l solution, freshly prepared.

4.5 Sodium hydroxide, 300 g/l solution.

4.6 Sodium hydroxide, 4 N solution.

4.7 Potassium nitrate, dried at 120 °C and cooled in a desiccator.

5 APPARATUS

5.1 Spectrophotometer, suitable for measurements at a wavelength of 410 nm.

5.2 10 mm cells for use with the spectrophotometer.

5.3 pH meter.

5.4 One-mark volumetric flasks, of capacity 50 ml, 100 ml 250 ml and 500 ml, complying with ISO/R 1042.

5.5 Pipettes, capacity 10 ml, complying with ISO/R 648 and ISO/R 835.

6 SAMPLING

The sample of pigment used for the test shall be taken in accordance with the provisions of ISO 842.

7 PREPARATION OF CALIBRATION GRAPH

7.1 Standard solution I

Weigh 163 ± 0,1 mg of the potassium nitrate (4.7), dissolve it in water in the 100 ml one-mark volumetric flask, make up to the mark and mix well.

7.2 Standard solution II

Pipette 10 ml of standard solution I into a 500 ml one-mark volumetric flask, make up to the mark and mix well.

7.3 Construction of graph

Pipette 2, 4, 6, 8 and 10 ml of standard solution II (corresponding to 0,04, 0,08, 0,12, 0,16 and 0,2 mg of NO_3 respectively) into separate 100 ml beakers.

To each beaker add 1 ml of sodium salicylate solution (4.4), evaporate to dryness on a water-bath and allow to cool in a desiccator. Moisten each dried residue with 1 ml of the sulphuric acid (4.1) and allow to stand in the desiccator for 10 min. Afterwards, wash the contents into separate 50 ml one-mark volumetric flasks with water, add 10 ml of the sodium hydroxide solution (4.5) to each and cool to room temperature.

Make up to the mark with water and mix well. Determine and record the absorbance of each of the solutions at 410 nm in 10 mm cells against a solution prepared in the same way as the previous solutions but omitting the nitrate solution.

Construct a graph of absorbance against the mass of NO_3, in milligrams.

8 PROCEDURE

8.1 Pipette into a 250 ml one-mark volumetric flask 50 ml of the clear aqueous extract obtained, as appropriate for the pigment under test, by the hot extraction method[1], or the cold extraction method[2]. Make up to the mark with water and mix.

NOTE – If the aqueous extract contains chromate, proceed as follows :

Place 50 ml of the clear aqueous extract into a 250 ml glass beaker and add 5 ml of sulphuric acid (4.2) and 2 ml of ethanol (4.3). Heat the solution until any chromate present is reduced as indicated by the blue-green colour of the solution and the absence of aldehyde odour; take care to avoid losses by splashing. Cool and add sodium hydroxide solution (4.6) until just alkaline. Cool again and adjust the pH to 8,0 ± 0,5 measured by the pH meter. Filter through filter paper and wash with hot water, collecting the filtrate and washings in a 250 ml one-mark volumetric flask. Cool, make up the mark and mix.

8.2 Pipette 10 ml of this solution to a 100 ml glass beaker.

NOTE – If the nitrate content is found to be greater than 0,1 %, carry out a second determination, using 5 ml of solution.

8.3 Add to the beaker 1 ml of sodium salicylate solution (4.4) and proceed as specified in clause 7, including the determination of the absorbance at 410 nm.

8.4 From the known absorbance of the test solution, determine from the calibration graph the corresponding mass of nitrate in milligrams.

9 EXPRESSION OF RESULTS

Calculate the water-soluble nitrate content expressed as NO_3, as a percentage by mass, by the formula :

$$\frac{25a}{2m}$$

where

a is the mass, in milligrams, of NO_3 corresponding to the absorbance of the test solution;

m is the mass, in grams, of the pigment from which the clear aqueous extract was obtained.

NOTE – If 5 ml of the extract was taken because the nitrate content was greater than 0,1 %, the formula becomes $\frac{25a}{m}$.

10 TEST REPORT

The test report shall include the following particulars :

a) a reference to this International Standard or to a corresponding national standard;

b) type and identification of the product under test;

c) any deviation, agreed or otherwise, from the test procedure specified;

d) the result of the test, and whether the hot or cold extraction method was used;

e) date of the test.

1) See Part III.

2) See Part VIII.

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