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ISO RECOMMENDATION

R 787

GENERAL METHODS OF TEST FOR PIGMENTS

FIRST SERIES

I to VII

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BRIEF HISTORY

relating to the First Series

The ISO Recommendation R 787, *General methods of test for pigments,* was drawn up by Technical Committee ISO/TC 35, *Paints, varnishes and related products and their raw materials,* the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In October 1965, this Draft ISO Recommendation (No. 832) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Czechoslovakia	Spain
Australia	India	Switzerland
Austria	Iran	U.A.R.
Belgium	Ireland	United Kingdom
Brazil	Israel	U.S.S.R.
Canada	Japan	Yugoslavia
Chile	Netherlands	
Colombia	Portugal	

In addition, the Member Body of Denmark disapproved Part 2, the Member Body of France disapproved Part 1, the Member Body of Italy disapproved Parts 6 and 7 and the Member Body of Sweden disapproved Part 4.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in July 1968, to accept it as an ISO RECOMMENDATION.

FOREWORD

The purpose of this ISO Recommendation 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 ISO Recommendation 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 in convenient groups as they become available, as parts of a single ISO Recommendation, 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.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART I

COMPARISON OF COLOUR

INTRODUCTION

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

1. SCOPE

1.1 Part I of this ISO Recommendation describes a general method of test for comparing the colour of a coloured pigment with that of an agreed sample of the pigment.

1.2 Either of the procedures described in section 5 is acceptable, but whenever an automatic muller is available, its use is recommended.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification 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 should a special method for comparison of colour be specified.

2. REAGENT

2.1 *Refined linseed oil,* complying with ISO Recommendation R 150, *Raw, refined and boiled linseed oil,* and having an acid value of 5.0 to 7.0 mg of KOH per gramme.

3. APPARATUS

3.1 *Palette knife,* steel knife with a tapered blade of the approximate dimensions 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

3.2 *Glass slide,* clear and colourless, 150 mm × 50 mm or other suitable size.

3.3 *Burette,* with a delivery such that 35 drops equal 1 ml of refined linseed oil.

3.4 *Automatic muller,* plates with a diameter of about 20 cm, speed of rotation about 120 rev/min. Alternatively, there may be used either a hand muller with a diameter of 70 to 75 mm or a palette knife complying with clause 3.1.

3.5 *Plate,* of ground-glass or marble, for use when an automatic muller is not available.

4. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

5. PROCEDURE

5.1 Test portion

The quantity of pigment taken for the test should be that indicated in the Table below, the oil absorption value of the pigment having been determined in accordance with Part V of this ISO Recommendation.

TABLE

Oil absorption value of pigment ml/100g	Mass of the test portion g
25 or less	1.00
26 to 65	0.30
66 upward	0.10

5.2 Procedure using automatic muller

Weigh, to the nearest milligramme, the amount indicated in the Table above of the pigment under test, and transfer it to the clean lower plate of the automatic muller (3.4). Add by means of the palette knife blade (3.1) the number of drops of refined linseed oil sufficient to obtain a satisfactory dispersion so that, when mixed, a smooth pigment-oil paste is obtained. When the pigment has been wetted, spread the paste in a circle of approximately 50 mm in diameter around the centre of the lower plate and clean the palette knife by drawing it across the top plate. Close the muller plates, apply a pressure of 68 kgf and grind the paste in four stages of fifty revolutions for each stage, picking up the paste with the same palette knife and transferring it to the centre of the plate after each stage.

When the grinding has been completed, add a further number of drops of the oil sufficient to obtain a suitable painting consistency, close the muller plates and grind the paste for a further twenty-five revolutions, then remove the paint from the plate and store it on a palette.

Prepare a paint immediately from the agreed sample of the pigment, using the same procedure. Compare the colour of the test portion with that of the agreed sample by spreading the two mixtures so prepared in the same direction on the glass slide (3.2) in opaque strips not less than 25 mm wide with touching edges not less than 40 mm long. Make the colour comparison by examining the strips in diffused daylight through the glass, and on the surface, immediately after application. Where good daylight is not available make the comparison in artifical daylight. By agreement between purchaser and vendor, a suitable colorimeter may also be used for making the comparison.

5.3 Procedure using hand muller or palette knife

Weigh, to the nearest milligramme, the amount indicated in the Table of the pigment under test, and transfer it to the glass or marble plate (3.5). Collect, on the end of the palette knife (3.1), the number of drops of oil sufficient to obtain a suitable consistency for spreading out with the palette knife, and mix it with the pigment on the plate (3.5).

When the pigment has become uniformly wetted with oil, start rubbing with the palette knife (3.1) or hand muller (3.4), using a backwards and forwards motion. Rubbing should spread the mixture over an area approximately 200 mm × 75 mm. After one hundred rubs (one rub consists of one forward plus one backward stroke of the knife), scrape the pigment-oil mixture into a heap at the centre of the plate, making sure that any unground pigment is removed from the knife blade outside the area which has been in contact with the plate.

Repeat the rubbing-out operation using a further hundred rubs; after which add a further number of drops of oil sufficient to obtain a suitable painting consistency. Mix well until the mixture is homogeneous, then transfer the mixture to one corner of the plate and clean the rest of the plate thoroughly.

Rub out the agreed sample of the pigment in the same way, using the same rubbing consistency as that used in treating the test portion, even though this may involve adding a larger or smaller number of drops of oil than used with the test portion to bring the agreed sample to the required consistency.

NOTE. – In each case, a total of between ten and forty-five drops of oil will be required to produce a consistency which allows the mixture to be painted out.

Compare, as described in clause 5.2, the colour of the test portion with that of the agreed sample of the pigment.

6. TEST REPORT

The test report should include the following information :

(*a*) A reference to ISO Recommendation R 787, Part I, or to a corresponding national standard.

(*b*) Type and identification of the pigment under test.

(*c*) Any deviation, by agreement or otherwise, from the test procedures described above.

(*d*) The procedure used (automatic muller, hand muller or palette knife).

(*e*) The result of the test (equal to, or better or worse than, the agreed sample of pigment).

(*f*) The date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART II

DETERMINATION OF MATTER VOLATILE AT 105° C

INTRODUCTION

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

1. SCOPE

Part II of this ISO Recommendation describes a general method of test for determining the percentage by mass of matter volatile at a temperature of 105 °C, in a sample of pigment.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification 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 should a special method for determination of volatile matter be specified.

2. APPARATUS

2.1 *Weighing bottle,* short, wide-mouthed, with ground-glass stopper.

2.2 *Oven,* capable of being maintained at a temperature of 105 ± 2 °C.

2.3 *Balance,* sensitivity 1 mg.

2.4 *Desiccator.*

3. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

4. PROCEDURE

4.1 Test portion

Spread 10 ± 1 g of the pigment under test in a uniform layer on the bottom of the tared weighing bottle (2.1), insert the stopper and weigh to the nearest milligramme.

4.2 Determination

Heat the weighing bottle (2.1) and contents, with the stopper removed, in an oven at a temperature of 105 ± 2 °C. Cool the bottle in the desiccator to room temperature, insert the stopper and weigh to the nearest milligramme.

Repeat the heating and cooling until constant mass is attained, i.e. until the results of two successive weighings, at an interval including at least 30 minutes heating, do not differ by more than 5 mg.

NOTE. – If the pigment under test is unstable at a temperature of 105 °C, the testing conditions should be agreed between purchaser and vendor and indicated in the test report.

5. EXPRESSION OF RESULTS

Calculate the matter volatile at a temperature of 105 °C, in percentage loss by mass, i.e.

$$\frac{100\,(m - m_1)}{m}$$

where

m is the original mass, in grammes, of the test portion,

m_1 is the final mass, in grammes, of the test portion after heating.

Report the result to the nearest 0.1 %.

6. TEST REPORT

The test report should include the following information :

(*a*) A reference to ISO Recommendation R 787, Part II, or to a corresponding national standard.

(*b*) Type and identification of the pigment under test.

(*c*) Any deviation, by agreement or otherwise, from the test procedures described above.

(*d*) The result of the test as indicated in section 5.

(*e*) The date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART III

DETERMINATION OF MATTER SOLUBLE IN WATER

(HOT EXTRACTION METHOD)

INTRODUCTION

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

1. SCOPE

1.1 Part III of this ISO Recommendation describes a general method of test for determining the percentage by mass of matter soluble in boiling water, in a sample of pigment.

1.2 Part VIII of this ISO Recommendation* describes a method for determining the percentage of matter soluble in cold water. For most pigments, these two test methods will give different results, and it is therefore essential to state clearly in a specification, which method is to be used, and in a test report, which method has been used.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when neither of these general methods is applicable to a particular pigment should a special method for determination of water-soluble matter be specified.

2. APPARATUS

2.1 *One-mark volumetric flask,* of 250 ml.

2.2 *Filter paper,* fine-textured, or *diaphragm,* or *colloid filter.*

2.3 *Evaporation dish,* flat bottomed, of glass, platinum, glazed porcelain or silica.

2.4 *Oven,* capable of being maintained at a temperature of 105 ± 2 °C.

2.5 *Balance,* sensitivity 1 mg.

2.6 *Desiccator.*

3. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

* At present, Draft ISO Recommendation No. 1251.

4. PROCEDURE

4.1 Test portion

Weigh 2 to 20 g of the sample, to the nearest 0.01 g, into a beaker.

NOTE. – The mass of pigment used should be chosen according to the amount of water-soluble matter in the pigment. This is particularly important for pigments that contain large amounts of matter soluble in water.

4.2 Determination

Wet the pigment in the beaker with a few millilitres of water or other suitable liquid.

NOTE. – If the pigment does not disperse easily in water, a wetting agent may be used; in the case of pigments not soluble in ethanol, 5 ml of ethanol may be used; in the case of pigments soluble in ethanol, a non-ionic wetting agent such as, for example, 10 ml of a 0.01 % solution of a polyethylene oxide condensate should be used. If the wetting agent is non-volatile under the conditions of test an appropriate correction should be made to the final water-soluble matter figure.

Add 200 ml of freshly distilled water, stir, and boil for 5 minutes unless another period is specified. A coagulating agent can be used if the semi-colloidal nature of the pigment makes this desirable, provided the agent selected is not such as to affect the subsequent determination of the acidity or alkalinity of the aqueous extract,* and provided only the minimum quantity is used.

Cool rapidly to room temperature, transfer to the volumetric flask (2.1) and dilute to the mark with neutral distilled water. Mix thoroughly by shaking and inversion, and filter through the filter paper (2.2) or, in the case of pigments which are difficult to filter, through a diaphragm or colloid filter (2.2) returning the filtrate to the filter until it runs clear. If these methods of obtaining a clear filtrate prove unsatisfactory, use a decantation, centrifugal or other method which will do so. Evaporate 100 ml of the perfectly clear filtrate to dryness in the evaporation dish (2.3) on a water bath.

Dry the residue in the oven at 105 ± 2 °C, cool in a desiccator and weigh. Repeat the heating and cooling until the results of the two last weighings, at an interval including at least 30 minutes heating, do not differ by more than 10 % of the final figure obtained for the water-soluble matter.

5. EXPRESSION OF RESULTS

Calculate the water-soluble matter (hot extraction method), in percentage residue by mass, i.e.

$$\frac{250\, m_1}{m}$$

where

m is the mass, in grammes, of the test portion,

m_1 is the mass, in grammes, of residue.

6. TEST REPORT

The test report should include the following information :

(*a*) A reference to ISO Recommendation R 787, Part III or to a corresponding national standard.

(*b*) Type and identification of the pigment under test.

(*c*) Any deviation, by agreement or otherwise, from the test procedures described above.

(*d*) The result of the test as indicated in section 5.

(*e*) The date of the test.

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* See ISO/R 787, Part IV, *Determination of the acidity or alkalinity of the aqueous extract.*

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GENERAL METHODS OF TEST FOR PIGMENTS

PART IV

DETERMINATION OF ACIDITY OR ALKALINITY OF THE AQUEOUS EXTRACT

INTRODUCTION

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

1. SCOPE

Part IV of this ISO Recommendation describes a general method of test for determining the acidity or alkalinity of the aqueous extract of a sample of pigment.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification 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 should a special method for determination of acidity or alkalinity be specified.

2. REAGENTS

2.1 *Hydrochloric* or *sulphuric acid,* 0.05 N standard solution.

2.2 *Sodium* or *potassium hydroxide,* 0.05 N standard solution.

2.3 *Methyl red indicator,* 0.1 g per 100 ml of ethanol 60 % (v/v).

3. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw materials for paints and varnishes.*

4. PROCEDURE

4.1 Following the procedure described in Part III of this ISO Recommendation, *Determination of matter soluble in water (Hot extraction method),* to the stage of obtaining a perfectly clear filtrate.

4.2 To 100 ml of this clear filtrate add five drops of the methyl red indicator (2.3).

4.3 If the solution is orange, it is considered to be neutral.

4.4 If the solution is yellow (alkaline), titrate it with the hydrochloric or sulphuric acid solution (2.1) to an orange end point.

4.5 If the solution is red (acid), titrate it with the sodium or potassium hydroxide solution (2.2) to an orange end point.

5. EXPRESSION OF RESULTS

Report the condition of the aqueous extract in one of the following ways :

(*a*) Neutral.

(*b*) Acid; the acidity should be expressed in terms of the number of millilitres of 0.1 N alkali required to neutralize the aqueous extract from 100 g of pigment, taking account of the mass of the test portion and the strength of the alkaline solution used.

(*c*) Alkaline; the alkalinity should be expressed in terms of the number of millilitres of 0.1 N acid required to neutralize the aqueous extract from 100 g of pigment, taking account of the mass of the test portion and the strength of the acid solution used.

6. TEST REPORT

The test report should include the following information :

(*a*) A reference to ISO Recommendation R 787, Part IV or to a corresponding national standard.

(*b*) Type and identification of the pigment under test.

(*c*) Any deviation, by agreement or otherwise, from the test procedures described above.

(*d*) The result of the test as indicated in section 5.

(*e*) The date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART V

DETERMINATION OF OIL ABSORPTION VALUE

INTRODUCTION

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

1. SCOPE

Part V of this ISO Recommendation describes a general method of test for determining the quantity of refined linseed oil that is absorbed under standard conditions by a sample of pigment. The oil absorption value may be expressed either on a volume/mass basis or on a mass/mass basis, and is usually required to be compared with the value determined at the same time on an agreed sample of the pigment.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation relating to that pigment, with a note of any detailed modification 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 should a special method for determination of oil absorption value be specified.

2. REAGENT

2.1 *Refined linseed oil,* complying with ISO Recommendation R 150, *Raw, refined and boiled linseed oil,* and having an acid value of 5.0 to 7.0 mg of KOH per gramme.

3. APPARATUS

3.1 *Ground-glass* or *marble plate,* at least 30 cm × 40 cm.

3.2 *Palette knife,* steel knife with a tapered blade of the approximate dimensions 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

3.3 *Burette* of 10 ml, graduated in 0.1 ml divisions.

4. SAMPLING

The sample of pigment used for the test should be taken in accordance with the provisions of ISO Recommendation R 842, *Sampling raw material for paints and varnishes.*

5. PROCEDURE

5.1 Test portion

Weigh the appropriate quantity of the pigment under test in accordance with the expected oil absorption, following the Table below.

TABLE

Expected oil absorption value of pigment ml/100 g	Mass of the test portion g
less than 10	20
10 to 30	10
30 to 50	5
50 to 80	2
over 80	1

5.2 Determination

Place the test portion (5.1) on the plate (3.1). Add the linseed oil slowly, four to five drops at a time, from the burette (3.3). After each addition rub the oil into the pigment with the palette knife (3.2), and continue the addition of oil at this rate until conglomerates of oil and pigment are formed. From this point, add the oil one drop at a time and follow each addition of oil by thoroughly rubbing with the palette knife. Cease the addition of oil when a paste of smooth consistency has been formed. This paste should just spread without cracking or crumbling.

Read the burette and note the quantity of oil used. The time taken for the complete operation should be between 20 and 25 minutes and during this time, the whole pigment mass should be manipulated with maximum effort by the operator.

Where a comparison is required with the oil absorption value of an agreed sample of pigment, repeat the test in exactly the same way using the agreed sample.

6. EXPRESSION OF RESULTS

Calculate the oil absorption value, either as the volume or the mass of oil required per 100 g of pigment, i.e.

$$\text{on the volume/mass basis, oil absorption value} = \frac{100\,V}{m}$$

or

$$\text{on the mass/mass basis, oil absorption value} = \frac{93\,V}{m}$$

where

V is the volume, in millilitres, of oil required,

m is the mass, in grammes, of the test portion.

Report the result to the nearest millilitre or gramme.

7. TEST REPORT

The test report should include the following information :

(*a*) A reference to ISO Recommendation R 787, Part V or to a corresponding national standard.

(*b*) Type and identification of the pigment under test.

(*c*) Any deviation, by agreement or otherwise, from the test procedures described above.

(*d*) The result of the test as indicated in section 6.

(*e*) The date of the test.

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GENERAL METHODS OF TEST FOR PIGMENTS

PART VI

DETERMINATION OF RESIDUE ON SIEVE

(OIL METHOD)

INTRODUCTION

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

1. SCOPE

1.1 Part VI of this ISO Recommendation describes a general method of test for determining the residue, on a sieve of nominal mesh aperture of 63 μm, from a sample of pigment ground into a paste with linseed oil.

1.2 Part VII of this ISO Recommendation describes a method for determining the residue, on a sieve of nominal mesh aperture of 63 μm, from a sample of pigment dispersed with water. For some pigments, these two test methods will give different results, and it is therefore essential to state clearly in a specification which method is to be used, and in the test report, which method has been used.

NOTE. – When this general method is applicable to a given pigment, only a cross-reference to it need be included in the ISO Recommendation 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 neither of these general methods is applicable to a particular pigment should a special method for determination of residue on sieve be specified.

2. REAGENTS

2.1 *Refined linseed oil,* complying with ISO Recommendation R 150, *Raw, refined and boiled linseed oil,* and having an acid value of 5.0 to 7.0 mg of KOH per gramme.

2.2 *White spirit,* complying with ISO/R . . ., **Mineral solvents for paints (white spirits and related hydrocarbon solvents).*

3. APPARATUS

3.1 *Ground-glass* or *marble plate.*

3.2 *Palette knife,* steel knife with a tapered blade of the approximate dimensions 140 to 150 mm long, 20 to 25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

* At present Draft ISO Recommendation No. 1250.