INTERNATIONAL STANDARD

ISO 8781-1

First edition
1990-11-15

Pigments and extenders — Methods of assessment of dispersion characteristics —

Part 1:
Assessment from the change in tinting strength of coloured pigments

Pigments et matières de charge — Méthodes d'évaluation de la dispersibilité —

Partie 1: Évaluation à partir de la variation de la force colorante

Reference number
ISO 8781-1:1990(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8781-1 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

ISO 8781 consists of the following parts, under the general title *Pigments and extenders — Methods of assessment of dispersion characteristics*:

— *Part 1: Assessment from the change in tinting strength of coloured pigments*

— *Part 2: Assessment from the change in fineness of grind*

— *Part 3: Assessment from the change in gloss*

Annex A forms an integral part of this part of ISO 8781.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

The degree of development of tinting strength of a coloured pigment in a dispersion is dependent on the amount of work done in the preparation of the dispersion of the pigment in a binder system. The ease with which the ultimate tinting strength is achieved may therefore be used to assess the ease of dispersion of that pigment. Thus, if the ultimate tinting strength can be attained easily, the pigment is considered to be readily dispersible.

The method described consists essentially of three steps:

a) preparation in stages of a range of dispersions of the coloured pigment in accordance with an appropriate part of ISO 8780;

b) preparation of reduced pastes from each of these dispersions;

c) determination of the rate of development of the tinting strength as a function of the amount of work done (this latter is related to the incremental stages of dispersion).

The rate of development of tinting strength is assessed either by calculating the percentage increase between two stages of dispersion or graphically.

INTERNATIONAL STANDARD ISO 8781-1:1990(E)

Pigments and extenders — Methods of assessment of dispersion characteristics —

Part 1:
Assessment from the change in tinting strength of coloured pigments

1 Scope

This part of ISO 8781 specifies a method for assessing, on the basis of tinting strength, the dispersion characteristics of coloured pigments which have been dispersed by one of the methods described in ISO 8780. It should be read in conjunction with ISO 8780-1.

The method is of general use for comparing similar pigments, for example a test pigment against an agreed reference pigment.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8781. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8781 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 105-A01:1989, *Textiles — Tests for colour fastness — Part A01: General principles of testing.*

ISO 591:1977, *Titanium dioxide pigments for paints.*

ISO 787-24:1985, *General methods of test for pigments and extenders — Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods.*

ISO 8780-1:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 1: Introduction.*

ISO 8780-2:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 2: Dispersion using an oscillatory shaking machine.*

ISO 8780-3:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 3: Dispersion using a high-speed impeller mill.*

ISO 8780-4:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 4: Dispersion using a bead mill.*

ISO 8780-5:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 5: Dispersion using an automatic muller.*

ISO 8780-6:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 6: Dispersion using a triple-roll mill.*

3 Definitions

For the purposes of this part of ISO 8781, the following definitions apply, plus the definitions given in ISO 8780-1.

3.1 coloured pigment paste: A dispersion of a coloured pigment in a binder system.

3.2 white pigment paste: A dispersion of a white pigment in a binder system.

3.3 reduced paste (reduction paste): A paste resulting from mixing a coloured pigment paste (3.1) with a white pigment paste (3.2).

4 Principle

The pigment under test and, if provided, the agreed reference pigment are each dispersed in stages under specified conditions in an agreed binder system. After each incremental dispersion stage, a portion of the mill base is taken and mixed separately with a white pigment paste to form a reduced paste.

The tinting strength (K/S value) of each reduced paste is determined as described in ISO 787-24. The percentage increase in tinting strength between two dispersion stages is calculated in order to assess the ease of dispersion of the pigment. Alternatively, a graph of the development of the tinting strength may be prepared.

5 Required supplementary information

For any particular application, the test method specified in this part of ISO 8781 needs to be completed by supplementary information. The items of supplementary information are given in annex A.

6 Materials

6.1 White pigment

The type of white pigment used shall be agreed between the interested parties, and shall be compatible with the binder system used in the preparation of the coloured pigment paste.

Unless otherwise agreed, titanium dioxide, grade R2, complying with the requirements of ISO 591 shall be used.

6.2 White pigment paste

Prepare the paste by dispersing the white pigment (6.1) in the binder system agreed for the dispersion of the test pigment. The pigment concentration shall be agreed.

NOTE 1 For low-viscosity pastes, a suitable dispersion may contain about 20 % of titanium dioxide. For high-viscosity pastes, a suitable paste may contain about 40 % of titanium dioxide (see, for example, the paste specified in sub-clause 5.1 of ISO 787-24:1985).

7 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

7.1 Balance, capable of weighing to 0,1 mg.

7.2 Film applicator, to give films of adequate thickness from the reduced paste (see 8.2).

7.3 Substrates, for application of the reduced paste.

Coated cards or glass plates have been found to be suitable.

7.4 Black and white contrast charts, for checking that an adequate film thickness of the reduced paste has been applied (see 8.2).

7.5 Automatic muller, for preparation of the reduced paste when using a high-viscosity binder system, or **beaker**, of glass or polyethylene, for preparation of the reduced paste when using a low-viscosity binder system.

7.6 Spectrometer, capable of measuring at wavelengths between 400 nm and 700 nm, or **photometer**, with filters appropriate to the pigment under test, or **tristimulus colorimeter**.

7.7 Cardboard template, of approximately 0,5 mm to 0,8 mm thickness, with a circular hole of the same diameter as the photometer (see 7.6) port, for use in measurements on wet films.

8 Procedure

8.1 Preparation of the reduced paste

8.1.1 Select an appropriate procedure from ISO 8780 to prepare the pigment dispersion. Take test portions of the coloured pigment paste at the chosen dispersion stages (see 10.1).

8.1.2 Take suitable amounts of the coloured pigment paste and white pigment paste (6.2), weigh to an accuracy of 0,5 % or better, and mix to provide a reduced paste of suitable depth of shade (see note 2). Mix in accordance with the appropriate procedure described in 8.1.3.

NOTE 2 A suitable reference depth of shade would be between 1/3 and 1/25 of the reference depths for textiles (see ISO 105-A01:1989, clause 12) (e.g. reflectance or reflectance factor between 15 % and 50 %).

8.1.3 Use one of the following procedures, as appropriate.

a) With a low-viscosity paste, mix the coloured pigment paste and the white pigment paste in the beaker (see 7.5) by stirring with a glass rod or a spatula until the mixture is homogeneous. Ensure that the mixing is thorough and carried out in a uniform manner, avoiding excessive shear

and periodically returning material adhering to the stirrer to the bulk of the mixture.

b) With a high-viscosity paste, mix the coloured pigment paste and the white pigment paste using the automatic muller (see 7.5) without application of a load on the upper plate. Place the weighed amounts of the coloured pigment paste and white pigment paste on to the lower plate of the automatic muller (see note 3). Mix together using the minimum effort, with the aid of the spatula. Distribute the paste at several points at a distance of about 35 mm from the centre of the lower plate or spread it in the form of a ring with an internal diameter of 40 mm and an external diameter of 100 mm (see note 4). After the top plate has been fitted, with no load applied, mix in four stages of 25 revolutions each. After each stage, homogenize the mixture with the spatula and spread it as before.

Do not mix pastes of high viscosity with pastes of low viscosity.

NOTE 3 It is recommended that pigment pastes be weighed on a transparent plastics sheet. The bulk of the paste is then transferred with a spatula to the lower plate and the remainder can be scraped off the sheet on to the upper plate.

NOTE 4 It is advisable to lay a paper ring of the requisite shape as a pattern beneath the glass plate.

8.1.4 Prepare a reduced paste with the agreed reference pigment following the same procedure.

8.2 Assessment of the reduced paste

Carry out an initial test by applying reduced paste to a contrast chart (7.4) to determine visually the minimum thickness required to give complete hiding. Then, using the film applicator (7.2), apply at least the minimum thickness of the reduced pastes of the test sample and the agreed reference pigment in quick succession on separate substrates (7.3) to give wet films of uniform thickness. A wet-film thickness of more than about 100 µm incurs the risk of floating and flooding. Thus, to minimize separation, if an opaque film is not obtained with a wet-film thickness of 100 µm, a second and, if necessary, a third film shall be applied after the first has dried.

When the film starts to get tacky, carry out the following rub-out test (see note 5). Lightly rub part of each film with a finger. Compare visually the difference in depth of shade between the rubbed and the non-rubbed surfaces (see note 6).

If specified, allow the film to dry in accordance with the agreed or specified conditions. Otherwise continue as described in clause 9. Carry out the photometric measurements (see clause 9) on reduced pastes that have been prepared consecutively within the same test series (i.e. those having approximately the same age).

NOTE 5 The rub-out test determines whether any pigment separation (for example floating, flooding or flocculation) has occurred.

NOTE 6 Application of the reduced paste by a high-shear technique (for example, a spray gun or hypodermic syringe) can be expected to give a film that displays a lower rub-out effect than that obtained with a film applied using a bar applicator.

9 Photometric measurement

Measure the reflectivity or the reflectance factor of the non-rubbed surfaces of the films by the procedure described in sub-clause 8.1.4 of ISO 787-24:1985.

If a spectrometer is used, vary the wavelength of the light between 400 nm and 700 nm until a minimum value of ρ_∞ or R_∞ is obtained and measure at this wavelength.

If a filter photometer or tristimulus photometer is used, select a filter that restricts the measurements to wavelengths close to that of maximum absorption.

Within one set of comparative tests, use the same wavelength or filter, selected on the basis of the maximum level of dispersion of the agreed reference pigment reduction paste. Record the values of ρ_∞ or R_∞. From the measured values, obtain the corresponding values of K/S as given in ISO 787-24:1985, annex B.

NOTE 7 In certain cases, for example for information purposes, it could be of interest to make photometric measurements of the rubbed surface. This should be agreed between the interested parties.

10 Expression of results

In this clause, t_i is the quantity which represents the work done in the dispersion up to the end of stage i. It may be expressed in terms of time, the number of revolutions of a dispersion device, the number of passes through a triple-roll mill or simply the number of stages.

10.1 Increase in tinting strength

Choose two agreed dispersion stages 1 and 2, with stage 2 near the ultimate tinting strength. Calculate the increase in tinting strength between these two stages to the nearest whole number, using the equation

$$IS = \left[\frac{(K/S)_2}{(K/S)_1} - 1 \right] \times 100$$

where

IS is the increase in tinting strength, expressed as a percentage;

$(K/S)_1$ is the K/S value at the end of stage 1;

$(K/S)_2$ is the K/S value at the end of stage 2.

When comparing the increase in tinting strength of different pigments, the same stages 1 and 2 should be used. These stages shall be stated in the test report.

10.2 Tinting strength development graph

Plot the K/S values obtained in accordance with clause 9 as a function of t_i.

NOTE 8 A tinting strength development graph is useful for detailed assessment of ease of dispersion, particularly if a pigment has not yet been tested in this respect. For example, if the tinting strength development graph passes through a maximum (or the plot of the reciprocals of K/S and t_i passes through a minimum), overgrinding, flocculation or recrystallization has occurred. A plot of the reciprocals is preferable as it yields an approximately straight line. Extrapolation of the line to $1/t_i = 0$ ($t_i = \infty$) allows the ultimate tinting strength to be determined.

Easy dispersion is indicated by a shallow slope, difficult dispersion by a steep slope (i.e. a large increase in tinting strength).

11 Significance of results

If the increase in tinting strength IS is between 0 and 20, the difference in dispersibility between the test sample and the agreed reference pigment cannot be considered to be significant unless it is greater than 7.

If the range of IS values is between 50 and 100, a difference of greater than 12 shall be regarded as significant.

12 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this part of ISO 8781 and the relevant part of ISO 8780;

c) the items of supplementary information referred to in annex A;

d) whether wet or dry films were measured (see 8.2 and clause 9);

e) the increase in tinting strength IS and identification of the stages corresponding to $(K/S)_1$ and $(K/S)_2$ (see 10.1), together with a statement whether the observed differences in IS are significant or lie within the limits of experimental error (see clause 11);

f) the time between application of the reduced pastes and the rub-out test, and whether in the rub-out test floating, flocculation or flooding was observed;

g) any deviation from the test procedure specified;

h) the date(s) of the test.

Annex A
(normative)

Required supplementary information

The items of supplementary information listed in this annex shall be supplied as appropriate to enable the method to be carried out.

The information required should preferably be agreed between the interested parties and may be derived, in part or totally, from an international or national standard or other document related to the product under test.

a) Method of dispersion (see ISO 8780).

b) Type of white pigment (see 6.1) and composition of the white pigment paste (see 6.2).

c) Reduction ratio (see 8.1).

d) Method of film application (see 8.2).

e) Method of presenting the results (numerical or graphical).

f) Reference pigment (if any).

UDC 667.622:541.18.051:535.668.2

Descriptors: paints, pigments, extenders, tests, determination, dispersibility.

Price based on 5 pages