INTERNATIONAL STANDARD

ISO 8780-2

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Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics —

Part 2:
Dispersion using an oscillatory shaking machine

Pigments et matières de charge — Méthodes de dispersion pour évaluer la dispersibilité —

Partie 2: Dispersion à l'aide d'une machine à secousses

Reference number
ISO 8780-2:1990(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 8780-2 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

ISO 8780 consists of the following parts, under the general title *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics*:

— *Part 1: Introduction*

— *Part 2: Dispersion using an oscillatory shaking machine*

— *Part 3: Dispersion using a high-speed impeller mill*

— *Part 4: Dispersion using a bead mill*

— *Part 5: Dispersion using an automatic muller*

— *Part 6: Dispersion using a triple-roll mill*

Annex A forms an integral part of this part of ISO 8780.

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INTERNATIONAL STANDARD ISO 8780-2:1990(E)

Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics —

Part 2:
Dispersion using an oscillatory shaking machine

1 Scope

This part of ISO 8780 specifies a method for the dispersion of pigments and extenders using an oscillatory shaking machine. The method is suitable for the preparation of dispersions of several samples in the quality control of pigments. This part is for use in conjunction with the methods of assessment described in ISO 8781, using an agreed binder system of low viscosity. It should be read in conjunction with ISO 8780-1.

NOTE 1 The advantage of the oscillatory shaking method is that several batches of small amounts of mill base can be tested simultaneously in a closed system where solvent loss is minimized.

This method is restricted to low-viscosity mill bases allowing free movement of the grinding spheres (see 7.1). It is not suitable for optimizing mill base formulations for other dispersing techniques.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8780. 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 8780 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 787-10:1981, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 2431:1984, *Paints and varnishes — Determination of flow time by use of flow cups.*

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

ISO 8781-1:1990, *Pigments and extenders — Methods of assessment of dispersion characteristics — Part 1: Assessment from the change in tinting strength of coloured pigments.*

ISO 8781-2:1990, *Pigments and extenders — Methods of assessment of dispersion characteristics — Part 2: Assessment from the change in fineness of grind.*

ISO 8781-3:1990, *Pigments and extenders — Methods of assessment of dispersion characteristics — Part 3: Assessment from the change in gloss.*

3 Required supplementary information

For any particular application, the test method specified in this part of 8780 needs to be completed by supplementary information. The items of supplementary information are given in annex A.

4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

4.1 Paint-conditioning machine, in which the containers are subjected to 680 to 690 reciprocating strokes per minute through a distance of 16 mm and an oscillatory action through an angle of ±15°.

4.2 Holder, for several mill base containers, designed for fastening to the centre axis of the shaking machine in such a way that

— the centre point of each mill base container is 70 mm from the centre axis of the shaking machine;

— the top and bottom of each mill base container are at the same distance from a horizontal plane through the centre axis of the shaking machine;

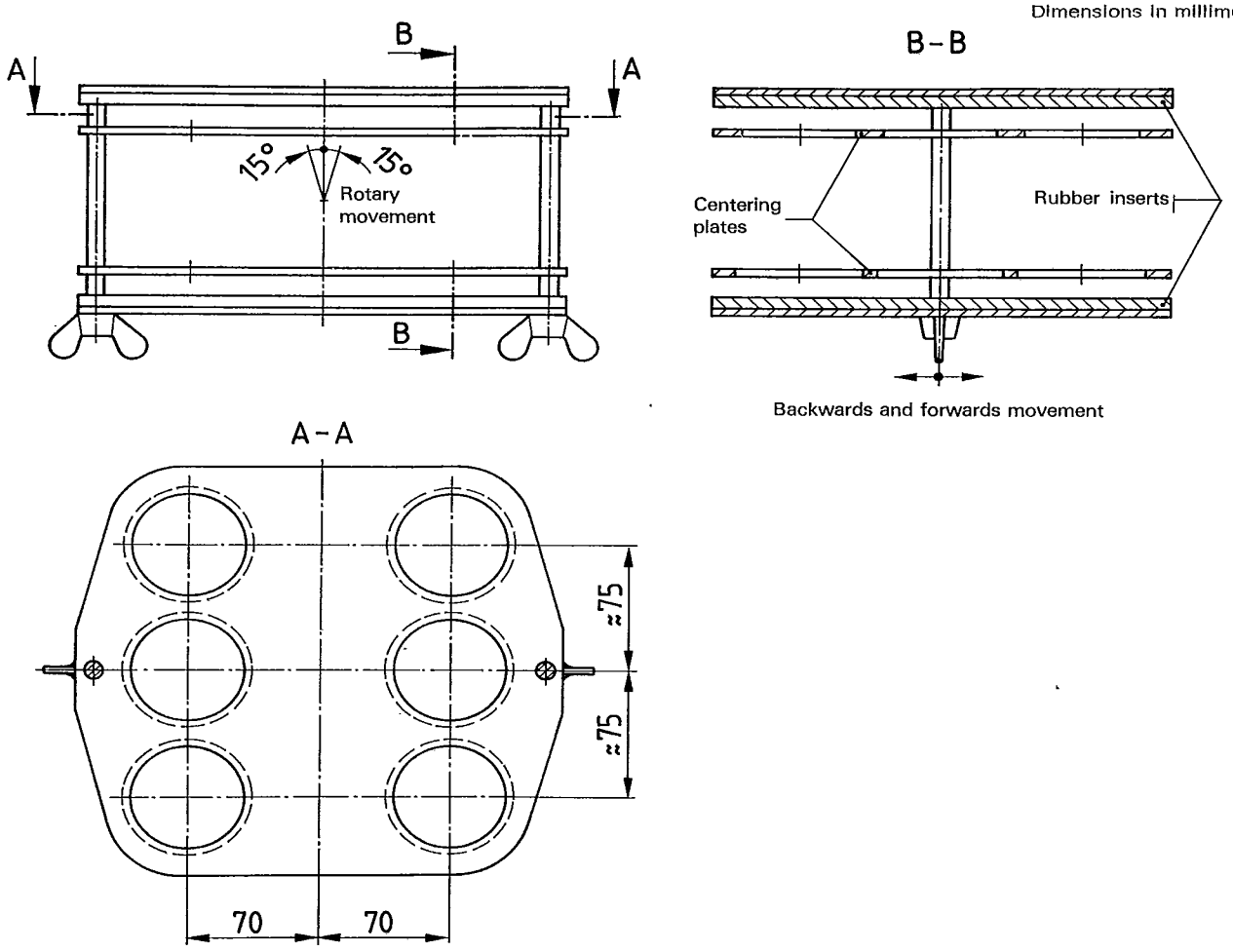
— the mean time-weighted position of the containers is vertical.

The design shown in figure 1 meets these requirements.

4.3 Containers, of a suitable type.

Containers for the mill bases shall be of the same size and type as those used for the agreed reference pigments.

Dimensions in millimetres



Details not given are to be selected to suit the purpose

Figure 1 — Holder

Examples of suitable types are 250 ml glass or polyethylene bottles with screw caps lined with polyethylene inserts.

The size and type of container shall be agreed on and shall be recorded in the test report.

4.4 **Grinding spheres**, of a suitable type.

Spheres of the same size and type shall be used for all the mill bases under simultaneous test. The type, mean diameter and density shall be agreed on and shall be recorded in the test report.

If the spheres have never been used, they shall be conditioned by shaking them in a mill base (see 7.1), for example for 60 min, and cleaned.

NOTE 2 The diameter, density and total mass of the grinding spheres have a significant effect on the dispersion obtained. Glass spheres of a mean diameter of 3 mm $\pm$ 0,5 mm and a density of 2,6 g/cm^3 $\pm$ 0,2 g/cm^3 have been found to be suitable.

5 Binder system

The binder system shall be agreed on between the interested parties. The test report shall state the binder, the solvent and the concentration of the binder in the solvent, as well as giving information on the rheological properties (for example, viscosity or flow time) of the binder system.

The same batch of binder system shall be used for all tests in the same series.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

7 Mill base

7.1 Composition

The viscosity of a mill base depends on the binder demand of the pigment and its concentration in a given binder system. Preliminary experiments shall therefore be carried out to ascertain a suitable mill base composition. The viscosity of the mill base during dispersion shall be such that the grinding spheres can move freely (see clause 1). This can be checked by shaking the containers manually.

For binder systems with flow times of 20 s to 40 s, determined with flow cup ISO 2431 No. 6, the following pigment concentrations have been found to be suitable as starting points:

a) pigments of low binder demand — pigment concentration higher than 40 % (m/m);

b) pigments of medium binder demand — pigment concentration 10 % (m/m) to 40 % (m/m);

c) pigments of high binder demand — pigment concentration lower than 10 % (m/m).

7.2 Volume

The mill base shall occupy approximately 30 % of the volume of the container (4.3). The mass of the pigment and of the binder system may be determined by using the following equations:

Mass, m_p, of pigment, in grams:

$$m_p = \frac{0{,}3 \times V}{\frac{1}{\rho_p} + \frac{100 - PC}{\rho_M \times PC}}$$

Mass, m_M, of binder system, in grams:

$$m_M = \frac{m_p \times (100 - PC)}{PC}$$

where

PC is the pigment concentration in the mill base, expressed as a percentage by mass;

V is the volume, in millilitres, of the container (4.3);

ρ_M is the density, in grams per cubic centimetre, of the binder system;

ρ_p is the density, in grams per cubic centimetre, of the pigment, determined by one of the procedures described in ISO 787-10.

8 Volume of grinding spheres

The apparent volume of the grinding spheres (see note 2) shall be approximately 50 % of the total volume of the container. The same volume of grinding spheres shall be used in all tests in the same series.

9 Procedure

9.1 Filling the containers

Unless otherwise agreed or found advisable (see note 3), weigh the grinding spheres (4.4) into the container (4.3). Add the required mass of binder system (m_M), calculated in accordance with 7.2. Wet the grinding spheres with the binder system by tilting the container. Then add the required mass of pigment (m_p), calculated in accordance with 7.2. Close the container. Wet the pigment by carefully tilting the container.

NOTE 3 If it is difficult to wet the pigment, it may be advisable to select a different sequence of preparing the containers and, if necessary, the mill base may be placed in the container first and stirred with a spatula prior to the addition of the grinding spheres.

Complete the preparation of the full set of mill bases as quickly as possible in order to avoid differences in stirring or wetting time between the samples.

If the criterion for assessing the dispersion characteristics is to be the evaluation of the development of tinting strength (see ISO 8781-1), the masses of the pigment and of the binder system shall be determined to within 0,5 %. For other methods of assessment (for example fineness of grind, see ISO 8781-2, and change of gloss, see ISO 8781-3), wider tolerance ranges may be agreed on.

9.2 Dispersion

Immediately after the last mill base has been prepared, place the containers in the holder (4.2) and clamp the assembly in the paint-conditioning machine (4.1).

NOTE 4 The intensity of the shaking process depends on the loading of the holder. If repeatable dispersion results are to be obtained, particularly in the case of two-armed shaking machines, the holders should be equally loaded.

Take test portions of the dispersion after each of several (agreed) shaking times. At least four shaking times shall be selected from the following:

— pigments with poor ease of dispersion — 5 min, 10 min, 20 min, 40 min, 80 min, 160 min;

— pigments with high ease of dispersion — 1 min, 2 min, 4 min, 8 min, 16 min, 32 min.

The total mass of these test portions shall not exceed 15 % of the initial mass of the mill base. Otherwise make separate dispersions for each time interval.

9.3 Stabilization

If necessary, for example if the mill base is not stable enough, stabilize each test portion after its removal from the mill base by adding, for example, more binder and/or special additives. The procedure shall be agreed on between the interested parties.

9.4 De-aeration

If necessary, allow any air bubbles within the test portions to escape before proceeding to assess the dispersion. The means by which this is achieved, for example by allowing to stand for a few minutes, shall be agreed on between the interested parties.

10 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 8780;

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

d) any deviation from the procedure specified;

e) 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) Type of machine (see 4.1).

b) Type of holder (see 4.2).

c) Type and size of container (see 4.3).

d) Type, mean diameter, density and total mass of the grinding spheres (see 4.4).

e) Binder system (see clause 5).

f) Composition of the mill base (see 7.1).

g) Shaking times (see 9.2).

h) Stabilization procedure (see 9.3).

i) De-aeration procedure (see 9.4).

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