INTERNATIONAL STANDARD

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

Part 4:
Dispersion using a bead mill

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

Partie 4: Dispersion à l'aide d'un disperseur à billes

Reference number
ISO 8780-4:1990(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 8780-4 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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Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics —

Part 4:
Dispersion using a bead mill

1 Scope

This part of ISO 8780 specifies a method for the dispersion of pigments and extenders using a bead mill. It 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.

This method is restricted to low- and medium-viscosity mill bases allowing free movement of the grinding spheres (see clause 7). Therefore, it is suitable for mill bases having a viscosity higher than those for which the dispersion method described in ISO 8780-2 is appropriate.

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 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 ISO 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 Bead mill, consisting of the following elements (see also figure 1).

4.1.1 Drive unit.

The power rating of the drive unit shall be sufficient to maintain the effective peripheral speed of the discs (see figure 2) at an agreed value.

The motor of the drive unit shall rotate the impeller shaft at between 1 000 r/min and 5 000 r/min. The drive unit shall be mounted on a stand so that the height of the impeller discs can be adjusted. There shall be a clamping device for the vessel at the foot of the stand, such that the impeller shaft is concentric with the vessel.

4.1.2 Stirrer assembly and vessel.

The stirrer assembly shall consist of a shaft on which solid discs, perforated discs or rings are arranged concentrically or eccentrically. The distance between the revolving stirrer and the wall shall be such that there is adequate clearance between the effective disc periphery and the walls of the vessel (see below).

The degree of dispersion achieved will depend on the type of stirrer assembly used. Figure 1 shows only one possible type.

The geometry of the strirrer assembly and vessel shall be as follows:

Minimum clearance between revolving stirrer asssembly and walls of vessel: 10 mm

Minimum clearance between lowest disc and base of vessel: 10 mm

The minimum effective peripherical speed v, in metres per second, of the disc, shall be adjustable between 5 m/s and 7 m/s, calculated as follows:

$$v = \frac{D \times \pi \times n}{60}$$

where

D is the effective diameter, in metres, of the rotating discs (see figure 2);

n is the rotational frequency, in revolutions per minute, of the shaft;

Dimensions in millimetres

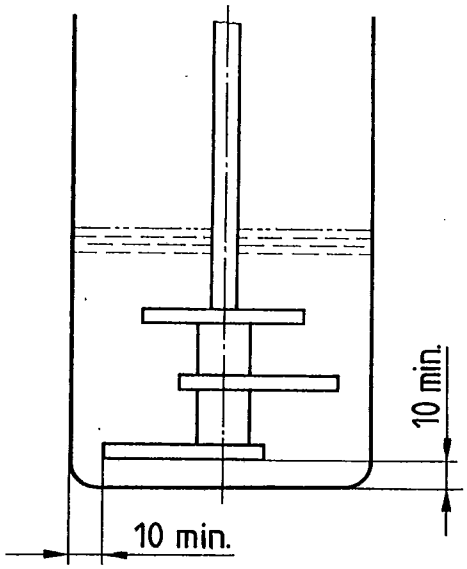

Figure 1 — Example of stirrer assembly

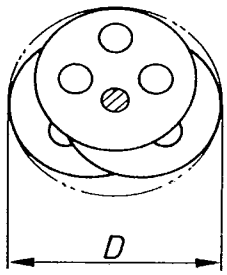

Figure 2 — Effective diameter of discs

The type, number and diameter of the discs, the geometry of the vessel and the effective peripheral speed of the revolving discs shall be agreed on and shall be recorded in the test report.

NOTE 1 A vessel with a double wall to allow circulation of a liquid to control the temperature is recommended, and a lid with a central aperture may also be provided.

NOTE 2 The nominal capacity of the vessel will depend on the volume of the mill base. Examples are given in table 1.

Table 1 — Mill base volume and dimensions of bead mill vessel

Mill base volume ml	Diameter of vessel mm	Height of vessel mm	Nominal capacity of vessel ml
50	50	70	125
100	65	85	250
200	85	110	500

4.2 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 stirring them in a mill base (see clause 7), for example for 60 min, and cleaned.

NOTE 3 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 1 mm to 2 mm and a density of 2,6 g/cm^3 $\pm$ 0,2 g/cm^3 have been found to be suitable. Steel balls with a diameter of 2 mm to 3 mm, a density of about 7,8 g/cm^3 and a hardness on Mohs' scale of 7 to 8 may also be used.

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 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) while the bead mill is running. This can be checked by observation.

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 25 % (*m*/*m*) to 60 % (*m*/*m*);

b) pigments of medium binder demand — pigment concentration 10 % (*m*/*m*) to 25 % (*m*/*m*);

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

8 Volume of grinding spheres

The apparent volume of the grinding spheres (4.2) shall be approximately 40 % 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 Pre-mixing

Weigh the agreed amounts of the binder system and pigment into the vessel (see 4.1.2).

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.

Stir with the appropriate stirrer assembly (4.1.2), without the grinding spheres, until the pigment is wetted. The pre-mixing time shall be agreed on and shall be recorded in the test report.

NOTE 4 The components may also be pre-dispersed with a high-speed impeller mill as described in ISO 8780-3.

9.2 Dispersion

Add the appropriate amount of grinding spheres (see clause 8). If appropriate, bring the mill base to the agreed temperature. Disperse at the agreed effective peripheral speed of the discs. Confirm that the grinding spheres are moving freely (see clause 7). If they are not, adjust the mill base composition (see clause 7) and repeat the procedure from 9.1.

Take test portions from the mill base after each of several (agreed) stirring times as follows:

Stop the stirrer after each of a number of agreed stirring times (for example 4 min, 8 min, 16 min, 32 min) and take a small test portion. Measure the temperature of the mill base and adjust to an agreed temperature before re-starting the stirrer. Remove any grinding spheres in the test portion by screening.

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 portion 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 and complete details of the bead mill (see 4.1).

b) Type, mean diameter, density and total mass of the grinding spheres (see 4.2).

c) Binder system (see clause 5).

d) Composition of the mill base (see clause 7) and its temperature (see 9.2).

e) Pre-mixing time (see 9.1).

f) Stirring times (see 9.2).

g) Stabilization procedure (see 9.3).

h) De-aeration procedure (see 9.4).

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