INTERNATIONAL STANDARD

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

Part 3:
Dispersion using a high-speed impeller mill

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

Partie 3: Dispersion à l'aide d'une turbine disperseuse à grande vitesse

Reference number
ISO 8780-3:1990(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 8780-3 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 3:
Dispersion using a high-speed impeller mill

1 Scope

This part of ISO 8780 specifies a method for the dispersion of pigments and extenders using a high-speed impeller mill. It is for use in conjunction with the methods of assessment described in ISO 8781, using an agreed binder system. It should be read in conjunction with ISO 8780-1.

NOTE 1 A high-speed impeller mill may be used either to disperse a pigment fully or partially as a preliminary to further dispersion using other equipment, for example sand or bead mills or attritors.

This method is restricted to mill bases of moderately high viscosity due to either high binder concentration and/or high pigment concentration which produces high shear forces. It is not intended to provide a means of formulating full-scale mill base compositions (scaling up the process from laboratory equipment to factory mills is not simple).

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 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 High-speed impeller mill, consisting of a cylindrical vessel and a horizontal disc stirrer blade driven by a motor. A disc with a serrated edge is commonly used.

4.1.1 Drive unit.

The power rating of the drive unit shall be sufficient to maintain the peripheral speed of the disc at an agreed value.

A reduced rotational frequency shall be available for pre-mixing in accordance with 8.2.

The motor of the drive unit shall be mounted on a stand together with the impeller shaft so that its height 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 Disc and vessel.

The diameters of the vessel and the disc shall be such that there is adequate clearance between the disc periphery and the walls of the vessel (see below). If the disc is serrated, it shall be mounted so that the direction of movement of the serrations is such that the mill base flows in the directions shown in figure 1.

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

The geometry of the vessel/disc assembly shall be related to the diameter D of the disc as follows:

Diameter of vessel: $1{,}3D$ to $3D$

Clearance between plane of disc and base of vessel: $0{,}25D$ to $0{,}5D$

Depth of mill base: $0{,}5D$ to $2D$

The peripheral speed v, in metres per second, of the disc, shall be adjustable between 5 m/s and 20 m/s, calculated as follows:

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

where

D is the diameter, in metres, of the disc;

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

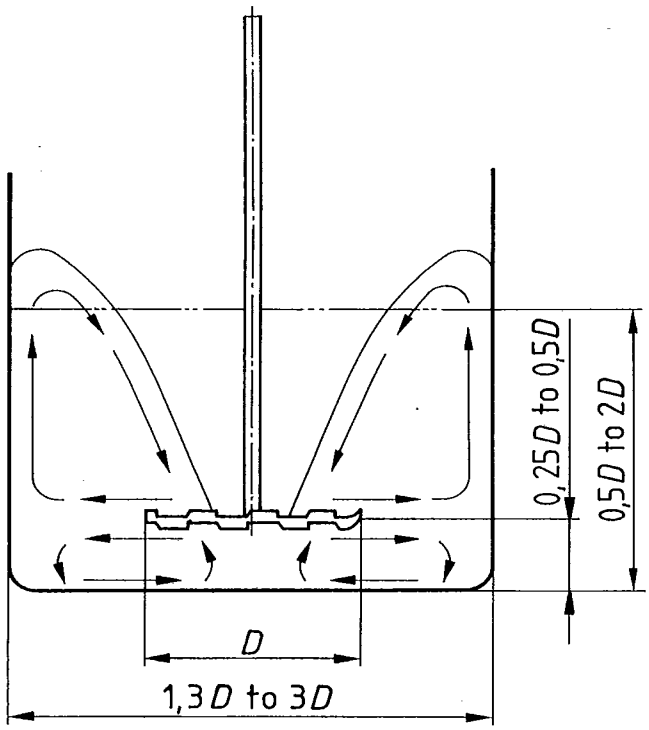

Figure 1 — Schematic section of a serrated-disc impeller mill

The type and diameter of the disc, the geometry of the vessel and the speed of the disc shall be agreed on and recorded in the test report.

NOTE 2 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 3 The diameter of a vessel used in an industrial-scale mill is normally two to three times the diameter of the impeller, or sometimes even greater. The vessel/disc geometry and the disc speed specified in this part of ISO 8780 make allowance for the small diameter of a laboratory disc.

4.2 Spatula.

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

7.1 General requirements

It is essential that the mill base assumes a flow pattern in the form of a toroid (or doughnut). The concentrations of pigment and binder system required to give this pattern shall be determined by prior experiment.

In order to develop high shear in the mill, a high-viscosity mill base is desirable. Thus, high pigment loadings and/or high binder-solids are usually recommended. The best combination depends on the wetting properties of the binder system for the pigment being tested.

NOTE 4 It may be preferable to operate the laboratory mill under non-ideal conditions so that differences between pigments are exaggerated. It has been found in practice that, with well-formulated mill bases, differences between easily dispersible pigments are minimized so that this test method is insensitive.

7.2 Determination of mill base composition

Add sufficient binder system to cover the impeller. Start the motor at the lowest speed. Make small additions of pigment and gradually increase the speed to that agreed. Observe the flow pattern throughout the whole operation and note how much pigment has been added when a toroid is first formed. Continue to add pigment until the toroid starts to collapse and again note the pigment mass added. Select a pigment concentration between these two points.

8 Procedure

8.1 Preliminaries

Weigh the pre-determined amount (see 7.2) of the binder system into the vessel. Weigh the pre-determined amount (see 7.2) of the pigment into a separate container.

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.

8.2 Pre-mixing

If appropriate, bring the vessel and the binder system to the agreed temperature (see 8.3). Immerse the impeller to the agreed depth (see 4.1.2).

With the impeller running at a low speed, gradually incorporate the pigment within a period of 5 min. Add the pigment at such a rate that a small amount of unwetted pigment always remains visible on the surface. Stop the motor, raise the impeller and, using a spatula (4.2), scrape off any pigment adhering to the impeller shaft and walls of the vessel into the mill base.

8.3 Dispersion

Lower the impeller into the vessel to the agreed depth. Adjust the speed of rotation to that agreed. Confirm, from the flow pattern, that the mill base composition is satisfactory (see 7.2). If the flow pattern is unsatisfactory, adjust the amount of pigment or binder system in the vessel until the flow pattern is correct and then, using the revised proportions, repeat the procedure from 8.1.

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

Stop the impeller 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 impeller.

NOTE 5 The method described can also be adopted as a pre-mix procedure for mill bases that have to be further dispersed by a laboratory bead mill in accordance with ISO 8780-4.

8.4 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.

8.5 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.

9 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 impeller mill (see 4.1).

b) Binder system (see clause 5).

c) Composition of the mill base (see 7.1) and its temperature (see 8.3).

d) Stirring times (see 8.3).

e) Stabilization procedure (see 8.4).

f) De-aeration procedure (see 8.5).

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