INTERNATIONAL STANDARD

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

Part 6:
Dispersion using a triple-roll mill

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

Partie 6: Dispersion à l'aide d'une broyeuse tricylindre

Reference number
ISO 8780-6:1990(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 8780-6 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 6:
Dispersion using a triple-roll mill

1 Scope

This part of ISO 8780 specifies a method for the dispersion of pigments and extenders using a triple-roll mill. The results permit conclusions to be drawn as to the behaviour of the products tested when dispersed using corresponding production machinery. This part of ISO 8780 is for use in conjunction with the methods of assessment described in ISO 8781, using an agreed binder system of high viscosity free from volatile materials. It should be read in conjuction with ISO 8780-1.

This method is restricted to high-viscosity mill bases. To improve the precision of the method, it is recommended that the dispersion procedures for the test pigment and agreed reference pigment are carried out at the same temperature.

The results obtained are not suitable for making comparisons with dispersibility results obtained using other methods of dispersion.

NOTE 1 The advantages of the triple-roll mill described in this part of ISO 8780 lie in the ease of handling of the equipment, the possibility of preparing adequate quantities of mill base for extensive evaluation procedures, the ease of cleaning and the possibility of correlating the results with those from production machinery. Unlike dispersing equipment for low-viscosity mill base formulations, a triple-roll mill of the kind described in this part of ISO 8780 permits the processing of mill base formulations within a wide range of high viscosities. Owing to this greater flexibility, precise specification of suitable mill base formulations is unnecessary.

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 787-24:1985, *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 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 1524:1983, *Paints and varnishes — Determination of fineness of grind.*

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 Triple-roll mill, with temperature control unit and mixing unit and complying with the following requirements:

a) Roll diameter: about 150 mm (if rolls of other dimensions are used, this shall be agreed and shall be recorded in the test report).

NOTE 2 Results obtained with roll diameters of less than 150 mm will not correlate with those from production machinery.

b) Roll length: 200 mm to 300 mm (if rolls of other dimensions are used, this shall be agreed and shall be recorded in the test report).

c) Transmission ratio ("friction") of the rolls: 1 : (2,5 to 3) : (5 to 6).

d) Speed of the take-off roll: 150 r/min to 200 r/min.

e) Roll contact force: capable of being set reproducibly up to a line pressure of 80 N per millimetre roll length (the corresponding pressure gauge reading in newtons per square millimetre is dependent on the design and shall be obtained from the mill manufacturer).

Roll contact force shall be expressed as line pressure, to allow comparison with other types of triple-roll mill. The line pressure is the contact force per millimetre of roll length. In most cases, this parameter cannot be read directly from the pressure gauges but has to be calculated from the gauge readings.

Since dispersion is also influenced by parameters other than the line pressure, correlation between different types of triple-roll mill shall be determined experimentally using information obtained from the mill manufacturer.

f) Temperature control of the heating or cooling fluid: to ± 1 °C, within the range from 20 °C to 60 °C.

g) Any gap-setting device shall not be active during the operation of this test.

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) 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 flow characteristics of a mill base depend on the binder demand of the pigment, its concentration in the mill base and the rheological properties of the binder system. Preliminary experiments shall therefore be carried out to ascertain a suitable mill base composition and this composition shall be agreed on between the interested parties. The mill base shall have a high viscosity and a sticky nature.

NOTE 3 The white pigment paste specified in ISO 787-24 has a viscosity considered to be the minimum value for this method.

Typical pigment concentrations are:

a) pigments of low binder demand — pigment concentration 65 % (m/m);

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

c) pigments of high binder demand — pigment concentration 25 % (m/m).

8 Procedure

8.1 Test portion

For the apparatus specified in 4.1, the mass of the mill base shall be not less than 50 g.

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

Thoroughly mix the agreed amounts of pigment and binder system with the spatula (4.2). Pre-heat the rolls of the triple-roll mill (4.1) to an agreed temperature and set the first and second roll at the lowest pressure setting, leaving the third roll clear. Load the mill base on to the first roll, switch on the triple-roll mill and mix until a homogeneous dispersion has been obtained.

NOTE 4 The pre-mixing time will depend on the type and quantity of mill base. For 50 g of mill base, 2 min is generally sufficient.

Alternatively, by agreement between the interested parties, the pre-mixing can be carried out using a high-speed impeller mill (see ISO 8780-3) or other suitable equipment. In this case, load the pre-mixed mill base on to the first roll of the triple-roll mill as in the above pre-mixing procedure, ensuring that its temperature is slightly below that of the pre-heated rolls.

8.3 Dispersion

With the rolls still rotating following pre-mixing, set them to produce friction, i.e. set the roll contact forces at levels appropriate to the viscosity of the mill base (so that the second and third rolls have a thin, uniform coating of mill base on them). Contact between rolls one and two shall be adjusted first and the third roll adjusted last (so that at no time is there metal-to-metal roll contact).

A pass is deemed to have been completed when the first roll is almost dry, just before the last of the mill base has passed the middle roll. Any material which runs off the sides of rolls shall be discarded as it may be either contaminated or not properly dispersed.

The appropriate roll contact force shall be determined by preliminary experiments and shall be agreed on between the interested parties.

If two or more pigments are to be compared, adjust the temperature of the prepared mill bases containing the individual pigments to within 2 °C.

Collect the mill base from the apron of the machine in a suitable container and mix thoroughly with the spatula. The ease of dispersion may be evaluated either by measuring, in accordance with ISO 1524, the fineness of grind of the material after one pass through the mill or by making one or more additional passes, taking portions of the dispersion after each pass and assessing their dispersion characteristics by one of the methods specified in ISO 8781.

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.

In contrast to low-viscosity mill bases (for which an oscillatory shaking machine, a bead mill or a high-speed impeller mill, for instance, may be used) stabilization is of minor importance.

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) the solvent (if any);

e) the method and time of pre-mixing (see 8.2);

f) the roll contact force, temperature of the thermostatic fluid and number of passes (see 8.3);

g) any deviation from the 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) Type and complete details (see 4.1) of the triple-roll mill.

b) Binder system (see clause 5).

c) Composition of the mill base (see clause 7) and its temperature (see 8.3).

d) Conditions of pre-mixing (see 8.2).

e) Conditions of dispersion (see 8.3).

f) Stabilization procedure (see 8.4).

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