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General methods of test for pigments — Part XX

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 787/XX (originally ISO/DIS 2893) was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes,* and circulated to the Member Bodies in September 1972.

It has been approved by the Member Bodies of the following countries :

Brazil	Israel	Spain
Canada	Italy	Sweden
Czechoslovakia	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	Thailand
France	Poland	Turkey
Germany	Portugal	United Kingdom
India	Romania	U.S.S.R.
Ireland	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Printed in Switzerland

The purpose of this International Standard is to establish a series of general test methods for pigments which are suitable for all or many of the individual pigments for which specifications might be required. In such cases, a cross-reference to the general method should be included in the International Standard relating to that pigment, with a note of any detailed modifications which might be needed in view of the special properties of the pigment in question.

Committee ISO/TC 35 decided that all the general methods should be published as they become available, as parts of a single International Standard, in order to emphasize the relationship of each to the whole series.

The Committee also decided that, where two or more procedures were widely used for determining the same or a similar characteristic of a pigment, there would be no objection to including more than one of them in the ISO series. In such cases it will, however, be essential to state clearly in a specification which method is to be used and, in the test report, which method has been used.

Parts of the series already published are as follows :

Part I : Comparison of colour
Part II : Determination of matter volatile at 105 °C
Part III : Determination of matter soluble in water (Hot extraction method)
Part IV : Determination of acidity or alkalinity of the aqueous extract
Part V : Determination of oil absorption value
Part VI : Determination of residue on sieve (Oil method)
Part VII : Determination of residue on sieve (Water method)
Part VIII : Determination of matter soluble in water (Cold extraction method)
Part IX : Determination of pH value of an aqueous suspension
Part X : Determination of density relative to water at 4 °C
Part XI : Determination of tamped volume
Part XII : Visual comparison of hue of powdered white pigment (Hollow cone method)
Part XIII : Determination of water-soluble sulphates, chlorides and nitrates
Part XIV : Determination of resistivity of aqueous extract
Part XV : Comparison of resistance of coloured pigments of similar types to light from a specified light source
Part XVI : Comparison of relative tinting strength (or equivalent colouring value) and colour on reduction in linseed stand oil using the automatic muller
Part XVII : Comparison of lightening power of white pigments
Part XVIII : Determination of residue on sieve by a mechanical flushing procedure
Part XIX : Determination of water-soluble nitrates (Salicylic acid method)

General methods of test for pigments – Part XX : Comparison of ease of dispersion (Oscillatory shaking method)

0 INTRODUCTION

This document is a part of ISO 787, *General methods of test for pigments.*

1 SCOPE AND FIELD OF APPLICATION

Part XX of this International Standard specifies a general method of test for comparing the ease of dispersion of two similar pigments in a specified medium. The result is expressed in terms of the time of shaking required to obtain a stated fineness-of-grind in a specified apparatus.

This method is most likely to be indicative of the comparative results which will be obtained when the pigments being compared are ground in a resin solution in certain types of mills, for example in a ball mill.

NOTE – When this general method is applicable to a given pigment, a cross-reference to it should simply be included in the International Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment should a special method for comparison of ease of dispersion be specified.

2 REFERENCES

ISO 842, *Raw materials for paints and varnishes – Sampling.*

ISO 1250, *Mineral solvents for paints – White spirits and related hydrocarbon solvents.*

ISO 1524, *Paints and varnishes – Determination of fineness-of-grind.*

3 PRINCIPLE

Portions of the test sample and the agreed sample are each milled with the chosen medium under known conditions on a paint conditioning machine at the same time, and the degree of dispersion of each portion is measured on a fineness-of-grind gauge at known time intervals during the dispersion process. From these results, graphs are constructed, and the times for each pigment to reach a stated fineness-of-grind are taken as indicative of the comparative ease of dispersion.

The progress of dispersion is influenced by a number of factors and for the purpose of a reference method the following have been standardized, although it is realised that other media or other test conditions may be used for control purposes in laboratories or by agreement between the interested parties :

1) capacity and dimensions of container;

2) nature and volume of grinding material;

3) volume of mill base, i.e. pigment plus dispersing medium;

4) nature of dispersing medium;

5) period of milling.

Any of these factors may be modified in special cases by agreement between the interested parties. The remaining important factor is the concentration of pigment in the mill base, which should be chosen in relation to the medium requirement of the pigment (this is not necessarily in proportion to its oil absorption). All pigments can be allocated to one of four groups :

a) pigments of low medium requirement – average milling concentration 60 % of pigment by mass;

b) pigments of intermediate medium requirement – average milling concentration 40 % of pigment by mass;

c) pigments of high medium requirement – average milling concentration 20 % of pigment by mass;

d) pigments of very high medium requirement (for example carbon black) – average milling concentration 10 % of pigment by mass.

NOTE – In general, inorganic pigments belong to the groups a) or b), organic pigments to group c), and carbon black to group d).

Because the concentration of pigment in the mill base affects the rate of milling, it is advisable to carry out three simultaneous millings with different concentrations of pigment, unless the suitable conditions for the pigment in question are already known.

The total volume of mill base, i.e. pigment and media, should be kept constant and the table gives the amounts of the two components for pigments of different relative densities and different milling concentrations.

TABLE – **Masses of pigment and medium for stated milling concentrations**

Select the vertical column to which the relative density of the pigment used most nearly corresponds. Then select in this column the figures in the horizontal rows for the milling concentration considered to be most suitable.

Milling concentration, % (*m/m*)	Mass[1] g	Relative density of pigment							
		1,25	1,5	2,0	2,5	3,0	4,0	5,0	6,0
4	pigment	1,0	1,0	1,0	1,0	1,0	1,0	1,0	1,0
	medium	24,3	24,4	24,6	24,7	24,7	24,8	24,8	24,9
6	pigment	1,5	1,5	1,6	1,6	1,6	1,6	1,6	1,6
	medium	24,0	24,2	24,4	24,5	24,6	24,7	24,7	24,8
8	pigment	2,1	2,1	2,1	2,1	2,1	2,1	2,1	2,1
	medium	23,7	23,9	24,1	24,3	24,4	24,6	24,6	24,7
10	pigment	2,6	2,6	2,7	2,7	2,7	2,7	2,7	2,7
	medium	23,3	23,6	23,9	24,1	24,3	24,4	24,5	24,6
12	pigment	3,1	3,2	3,2	3,3	3,3	3,3	3,3	3,3
	medium	22,9	23,3	23,7	23,9	24,1	24,3	24,5	24,5
15	pigment	4,0	4,0	4,1	4,2	4,2	4,3	4,3	4,3
	medium	22,4	22,8	23,3	23,6	23,8	24,1	24,3	24,4
20	pigment	5,4	5,5	5,7	5,8	5,9	5,9	6,0	6,0
	medium	21,5	22,0	22,7	23,1	23,4	23,8	24,0	24,2
25	pigment	6,8	7,0	7,3	7,5	7,6	7,8	7,9	8,0
	medium	20,5	21,1	22,0	22,5	22,9	23,4	23,7	24,0
30	pigment	8,4	8,7	9,1	9,4	9,6	9,8	10,0	10,1
	medium	19,5	20,3	21,3	21,9	22,4	23,0	23,4	23,6
40	pigment	11,6	12,2	13,1	13,7	14,1	14,7	15,0	15,3
	medium	17,4	18,3	19,6	20,5	21,1	22,0	22,5	23,0
50	pigment	15,1	16,2	17,7	18,8	19,6	20,7	21,5	22,0
	medium	15,1	16,2	17,7	18,8	19,6	20,7	21,5	22,0
60	pigment	18,9	20,6	23,2	25,1	26,6	28,7	30,1	31,1
	medium	12,6	13,7	15,5	16,8	17,7	19,1	20,1	20,7
70	pigment	23,1	25,6	29,8	33,0	35,6	39,5	42,2	44,2
	medium	9,9	11,0	12,8	14,2	15,3	16,9	18,1	18,9

1) If it is found to be more convenient, the resin solution may be measured by volume rather than by mass, the relative density being accurately known.

4 REAGENTS

4.1 Alkyd resin (see note), 75 % (*m/m*) in Type A solvent complying with ISO 1250. The resin solution should be filtered before use.

NOTE – The alkyd resin shall have the following characteristics :

Long-oil linseed oil/pentaerythritol alkyd resin containing approximately 68 % (*m/m*) of fatty acid and 20 % (*m/m*) of phthalic anhydride and having a viscosity, as a 75 % (*m/m*) solution in mineral solvent, of 6 to 8 N·s/m^2 (60 to 80 P) at 20 °C.

4.2 Alkyd resin, 20 % (*m/m*) solution in mineral solvent, freshly prepared by mixing 20 parts of the 75 % resin solution (4.1) with 55 parts by mass of the same mineral solvent and filtered before use.

5 APPARATUS

5.1 Paint conditioning machine, in which the glass bottles are subjected to 680 to 690 reciprocating strokes per minute through a distance of 16 mm, and an oscillatory action through an angle of 30°.

NOTE – A suitable machine is the "Red Devil" manufactured by Red Devil Tools Inc., 2400 Vauxhall Road, Union (New Jersey) 07083 U.S.A.

5.2 Glass bottles, of approximately 125 ml capacity, external dimensions approximately 70 mm high and 60 mm in diameter, with screw caps. The cardboard inserts in the caps shall be coated with polyethylene, or if not so coated,

oversize liners of a film of suitable plastics material of thickness approximately 0,01 mm shall be used to separate the contents from the insert.

5.3 Holder to accommodate six glass bottles and ensuring that the centres of all bottles are 70 mm from the centre axis BC (see the figure).

NOTE — The geometry of the holder is important and information is given in the annex and figure.

5.4 Glass spheres of 3 mm diameter. Since several qualities are available differing from each other in relative density, it is important that the same quality be used in all the bottles in a set.

NOTE — The glass spheres shall have been previously used, before carrying out the tests.

5.5 Spatula.

5.6 Fineness-of-grind gauge with scraper, covering the range 0 to 50 μm.

6 SAMPLING

The sample of pigment used for the test shall be taken in accordance with ISO 842.

7 PROCEDURE

7.1 Weigh, into a bottle (5.2), 100 g of the glass spheres (5.4). The glass spheres shall not more than half fill the bottle. Weigh the appropriate amounts from the table of the 20 % resin solution (4.2) and the reference sample of the pigment. First add the resin solution to the bottle and swirl the bottle to wet the glass spheres with the resin solution before adding the pigment. Add the pigment and carefully stir the contents of the bottle with the spatula to wet the pigment. Clean the spatula by drawing it flat across the neck of the bottle and screw on the cap (see 5.2).

Prepare two additional bottles as above using the quantities of pigment and 20 % resin solution given in the table for the milling concentrations immediately above and immediately below that used for the first bottle. These three bottles will, therefore, contain quantities of pigment and resin solution corresponding to three adjacent concentrations in the table, the middle one being that considered most likely to be suitable.

7.2 Repeat the preparation of three more bottles, using the same quantities of pigment and resin solutions as before, but in this case using the sample of pigment under test.

NOTE — If suitable test conditions for the pigment are already known, it is sufficient to carry out the comparison at a milling concentration known to be satisfactory.

7.3 Place the bottles in the holder (5.3), clamp the latter in the paint conditioner (5.1) so that the centre line of the holder is in line with the driving shaft of the machine, and run it for 5 min. Remove a small representative portion of the pigment dispersion of one of the bottles, test it twice on the 0 to 50 μm fineness-of-grind gauge (5.6) by the method specified in ISO 1524, record the mean of the results, and replace the liner and cap of the bottle. Test the pigment dispersion of each of the bottles in the same way and after replacing them in the holder and the paint conditioner, run the machine for another 5 min, repeating the testing of fineness-of-grind after each period of 5 min, until a reading of less than 5 μm is obtained. (This is unlikely to require more than a total of 30 min milling time.)

NOTE — An "off-gauge" reading of less than 5 μm should be taken as equal to 3 μm for that time of milling.

7.4 Stabilize the mill base by making consecutive additions of 2 ml, 4 ml and 8 ml of the 75 % solution of alkyd resin (4.1), stirring by hand for 0,5 min after each addition, and then replace the cap. Treat the contents of each bottle in turn in this way. Then carry out the fineness-of-grind test on the pigment dispersion from each bottle in the same order as that in which stabilization was carried out and record the results.

NOTE — A decrease in fineness, i.e. an increase in the fineness-of-grind reading, indicates that shock-seeding, i.e. the rapid formation of pigment agglomerates, has occurred; the concentration used for that milling should be recorded as unsuitable. When this information is known for a pigment, it is not necessary to carry out the full examination with three adjacent milling concentrations. The test may then be restricted to the highest milling concentration which does not suffer shock-seeding.

8 EXPRESSION OF RESULTS

Plot the fineness-of-grind reading, in micrometres, against the time of milling, in minutes, but ignore the results of any mill base which indicates "shock-seeding". Join adjacent points by straight lines.

Read off the times of milling, for each mill base, required to reach fineness-of-grind readings of 10 μm and 5 μm.

NOTE — By agreement between the interested parties, the times required to reach readings other than 10 μm and 5 μm may be used.

Compare these times for similar mill bases (i.e. same pigment concentration of the test sample and reference sample) and take these times as an indication of the comparative ease of dispersibility of the samples at a fineness of 10 μm and 5 μm.

9 TEST REPORT

The test report shall include the following information :

a) a reference to this International Standard or to an equivalent national standard;

b) type and identification of the pigment under test and of the reference pigment;

c) milling concentration used;

d) the curves showing the times of milling, in minutes, against fineness-of-grind readings for both the test sample and the reference sample, and the result of the test;

e) any deviation, by agreement or otherwise, from the test procedure described above;

f) date of the test.

ANNEX

DESCRIPTION OF BOTTLE HOLDER

The holder described here has been developed as a means not only of clamping a number of bottles or milling containers into the Red Devil machine, but also of positioning them in a repeatable manner in relation to the axis of motion of the machine. The holder takes the form of two separate aluminium plates. The top one has a 10 mm thick layer of flexible foam stuck to the underside to act as a cushion for the tops of the bottles when the sheet is laid over the bottles; the bottom plate has cemented to it a 50 mm thick layer of rigid foam out of which are cut six holes into which the bottles are put. The position and size of these holes are shown in the drawing. The bottom of each hole has a 10 mm thick layer of flexible foam to serve as a cushion for the bottoms of the bottles. Fastened to the underside of the bottom plate are two locating pegs or lugs which fit behind the back of the lower clamping plate of the machine itself; these are set at an angle of approximately 45° to the sides of the bottom plate, the positions being shown in the drawing, and are towards the back of the holder, so that it is convenient to mark the opposite short side as "Front". The holder with the bottles in it is placed horizontally on the lower clamping plate of the machine, so that the locating lugs are behind the back, and the centre line of the holder is in line with the driving shaft of the machine. The top plate is placed on top of the bottles and the clamp screwed to fasten the assembly firmly in the machine.

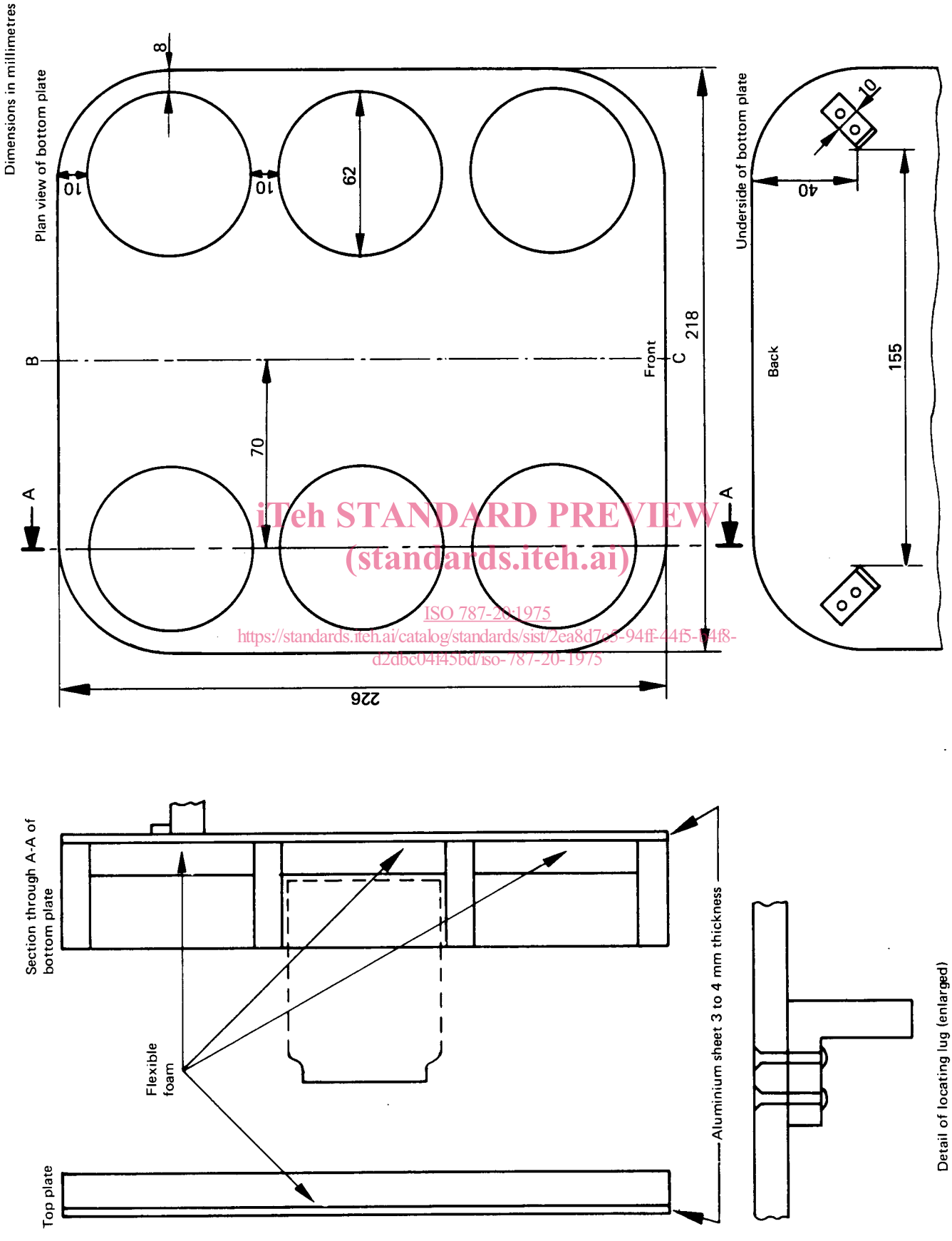

FIGURE — **Bottle holder**