
Coating powders —

Part 13:

Particle size analysis by laser diffraction

Poudres pour revêtement —

Partie 13: Analyse granulométrique par diffraction laser

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Printed in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 8130 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 8130-13 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 8130 consists of the following parts, under the general title *Coating powders*:

- *Part 1: Determination of particle size distribution by sieving*
- *Part 2: Determination of density by gas comparison pycnometer (referee method)*
- *Part 3: Determination of density by liquid displacement pycnometer*
- *Part 4: Calculation of lower explosion limit*
- *Part 5: Determination of flow properties of a powder/air mixture*
- *Part 6: Determination of gel time of thermosetting coating powders at a given temperature*
- *Part 7: Determination of loss of mass on stoving*
- *Part 8: Assessment of the storage stability of thermosetting powders*
- *Part 9: Sampling*
- *Part 10: Determination of deposition efficiency*
- *Part 11: Inclined-plane flow test*
- *Part 12: Determination of compatibility*
- *Part 13: Particle size analysis by laser diffraction*

Coating powders —

Part 13:

Particle size analysis by laser diffraction

1 Scope

This part of ISO 8130 is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a method for the determination of the equivalent-sphere particle size distribution of coating powders by laser diffraction and is suitable for discriminating between particles of the size range from 1 μm to 300 μm .

This method is applicable only for dry powders.

NOTE Most instruments will be capable of discriminating between particles of sizes beyond these limits.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 8130. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 8130 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3270:1984, *Paints and varnishes and their raw materials — Temperatures and humidities for conditioning and testing*

ISO 8130-9:1992, *Coating powders — Part 9: Sampling*

ISO 9276-1:1998, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 13320-1:1999, *Particle size analysis — Laser diffraction methods — Part 1: General principles*

3 Term and definition

For the purposes of this part of ISO 8130, the following term and definition apply.

3.1

obscuration

percentage or fraction of incident light that is attenuated due to extinction (scattering and/or absorption) by the particles of a coating powder

NOTE A full list of definitions is given in ISO 13320-1.

4 Principle

A representative sample of the coating powder, dispersed at an adequate concentration in air, is passed through the beam of a monochromatic laser source. The light scattered by the particles at various angles is measured by a multi-element detector and numerical values relating to the scattering pattern are recorded for subsequent analysis. These numerical scattering values are then transformed using an appropriate optical model and mathematical procedure to yield the proportion by volume of the sample in each size class forming a sphere-equivalent volumetric particle size distribution.

5 Apparatus

NOTE A description of laser analyser apparatus is given in ISO 13320-1.

5.1 Laser diffraction particle size analyser, with a minimum size range of 1 μm to 300 μm .

5.2 Dry-powder feeder, to be connected to the laser apparatus. This shall incorporate suitable means of delivering a constant stream of powder, free from agglomerates, into the analyser.

5.3 Compressed-air supply, delivering sufficient pressure as recommended by the instrument supplier. The air shall be dry, free from oil and filtered to 4 μm .

5.4 Air extraction system, capable of extracting air at the rate required by the instrument manufacturer.

Certain domestic vacuum cleaners may be found to be suitable. The vacuum unit shall be located such that the output of hot air does not reach the measurement zone.

Ensure that all particle-transporting devices, such as the dry-powder feeder, vacuum inlets and hoses, are electrically earthed to prevent electrostatic discharges.

5.5 Computer and software, suitable for analysing the data from the measurement device. The computer may be connected to a suitable printer. Any special requirements for the specifications of computer hardware will normally be advised by the instrument manufacturer.

5.6 Spatula or spoon.

6 Sampling

Prepare a representative sample of the powder to be tested, as described in ISO 8130-9. A quantity between 3 g and 5 g is normally sufficient for each determination.

7 Conditions

Measurements shall be made under standard conditions as specified in ISO 3270. The period of conditioning shall be not less than 30 min.

8 Procedure

NOTE 1 It is recommended that the operator be familiar with the procedures detailed in ISO 13320-1.

WARNING — The radiation from instruments equipped with a low-power laser can cause permanent eye damage. Never look in the direct path of the laser beam or its reflections. Avoid cutting the laser beam with reflecting surfaces. Observe the local laser radiation safety regulations.

Carry out the measurements in duplicate.

Install and adjust the apparatus in accordance with the manufacturer's instructions for powder of the expected particle size range.

When the system is switched on at the start of a series of determinations, allow at least 30 min to elapse to enable the equipment to stabilize, referring to the manufacturer's instructions as necessary. Check that the laser beam is centrally aligned on the detector, and adjust if necessary.

NOTE 2 With most instruments alignment will take place automatically, but it may be necessary for it to be carried out by a service engineer.

Using the spatula or spoon (5.6), add the powder to the powder feeder (5.2) and adjust the air flow as recommended by the instrument supplier.

Make a background measurement (i.e. in the absence of powder) to determine the combined effect of electrical "noise" and contamination in the system. This measurement needs to be subtracted from the sample measurement.

NOTE 3 This procedure is normally undertaken automatically by the instrument's computer.

Start the powder feeder in accordance with the instructions such that the level of obscuration (see 3.1) lies between 1 % and 15 %, preferably between 7 % and 10 %.

Allow the powder sample to pass through the air cell, using a brush to remove any fines remaining in the feed tray.

After the determination, the data are displayed on the computer visual display unit (VDU).

Ensure the instrument is thoroughly cleaned between determinations.

At the end of a measuring session, the computer software may be closed down and the device turned off.

9 Calculation of results

As noted above, the computer software, associated with the particle size analyser, will undertake the calculations and allow the results to be displayed (and, if required, printed) in both tabular and graphical form.

NOTE In general, the Fraunhofer theory will be applied (see ISO 13320-1) and details of refractive indices will not be required. However, in the case of unpigmented coating powders it may be necessary to use Mie theory, in which case refractive indices will be required.

If the results of the two determinations for any size class differ by not more than 1 %, calculate the arithmetic mean. Otherwise, undertake a third determination and report the arithmetic mean of all three measurements, noting this in the test report.

10 Expression of results

Express the results in accordance with ISO 9276-1.

11 Precision

Experience shows that repeatability within 1 μm in the range up to 100 μm particle diameter may be expected.

12 Test report

The test report shall include at least the following information:

- a) all details necessary to identify the product tested;
- b) a reference to this part of ISO 8130 (ISO 8130-13);
- c) a full description of the equipment used, including:
 - 1) a description of the powder feeder, including the name of the manufacturer and type of equipment,
 - 2) the feeding rate,
 - 3) the wavelength of the laser beam,
 - 4) the number of detector elements,
 - 5) the focal length of the lens,
 - 6) the scattering-angle range,
 - 7) the obscuration value,
 - 8) the date of the most recent alignment and calibration date;
- d) the results of the determination in accordance with ISO 9276-1 and clause 9;
- e) any deviations from the test method;
- f) the date of the test.

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