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Coating powders —

Part 13: Particle size analysis by laser diffraction

Poudres pour revêtement —

Partie 13: Analyse granulométrique par diffraction laser

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 8130-13:2001), which has been technically revised.

The main changes compared to the previous edition are as follows:

— clauses on precautions, accuracy, reference materials and error sources have been added;

— the requirements concerning the analyser (5.1) are more detailed and description of the testing (8.3) has been extended;

— the test report has been amended to be more detailed;

— the text has been editorially revised and the normative references have been updated.

A list of all the parts in the ISO 8130 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Coating powders —

Part 13: Particle size analysis by laser diffraction

1 Scope

This document specifies a method for the determination of the equivalent-sphere particle size distribution of coating powders by laser diffraction, for particles of the size range from 1 µm to 300 µm.

NOTE There is a possibility that particle sizes >300 µm need the use of a different optical model.

This document is specific for the measurement of coating powders and also draws attention to ISO 13320, which provides guidance on instrument qualification and particle size distribution.

Laser diffraction is not suitable for determining oversize material, which can be verified by sieve analysis as described in ISO 8130-1 or by dynamic image analysis as described in ISO 13322-2.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 8130-14, *Coating powders — Part 14: Vocabulary*

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

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO 9276-4, *Representation of results of particle size analysis — Part 4: Characterization of a classification process*

ISO 13320, *Particle size analysis — Laser diffraction methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8130-14, ISO 13320 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
obscuration
percentage or fraction of incident light that is attenuated due to extinction (scattering and/or absorption) by the particles during a laser diffraction measurement

4 Principle

A coating powder sample is analysed by dynamic light scattering. The powder particles scatter light and their intensities are measured at various angles by a multi array detector to yield a scattering pattern. The pattern is then transformed to numerical values, using an appropriate optical model and mathematical procedure, to yield the proportion of the total volume of particles for a discrete number of size classes forming a volumetric particle size distribution (PSD).

5 Apparatus

Ordinary laboratory apparatus, together with the following:

5.1 Laser diffraction particle size analyser, as specified in ISO 13320, with a size range of 1 µm to 300 µm.

The instrument should be located in a clean draft free environment to avoid particulate contamination and be vibration free such that signal to noise ratio is not compromised. More specific guidance is provided in ISO 13320.

The apparatus shall have a feeder suitable for coating powders of different particle shape and a dispersing unit ensuring a constant and agglomerate free flow of the coating powder.

For coating powders, compressed air or vacuum is used to disperse the powder particles, and it is essential that the compressed air is free from contamination which can influence the measurement.

5.2 Computer to store the optical model for analysis, usually as a model matrix containing light scattering vectors per unit of volume per size class, scaled to the detector's geometry and sensitivity and for calculation of the PSD.

5.3 Spatula or **spoon,** to dispense the coating powder into the feeder unit.

6 Sampling

Take a representative sample of the product under test as specified in ISO 15528. A quantity between 3 g and 5 g is normally sufficient for each determination and should follow the advice of the instrument supplier.

7 Test conditions

Measurements should be made under standard atmosphere 23/50 class 2 as specified in ISO 291 or by agreement between the interested parties.

8 Procedure

8.1 General

The operator should be familiar with the procedures specified in ISO 13320 as well as the operating instructions of the instrument manufacturer.

8.2 Precautions

Follow all the necessary safety instructions provided by the equipment manufacturer, paying particular attention to earthing to prevent ignition of dust caused by electrostatic discharges.

Allow time for the laser to warm up and ensure alignment of the laser diffraction system components, as per the provided instructions.

Verify that the dispersion unit generates a steady mass flow of powder particles by the obscuration values.

8.3 Testing

8.3.1 Sample preparation

Prepare and disperse a sample according to Clause 6. The sample shall be representative for the batch of product within a stated confidence interval, as specified in ISO 15528. The amount of test sample should correspond to at least the minimum required for precision. The dispersion conditions should lead to complete deagglomeration without comminution.

Using the spatula or spoon (5.3), add the powder to the powder feeder and adjust the powder flow as recommended by the instrument supplier.

8.3.2 Measurement

Start the test in accordance with the instrument instructions. The level of obscuration should lie between 1 % and 10 %.

8.3.3 Instrument performance

Qualify the instrument performance with respect to both precision and accuracy at regular time intervals by measuring a reference material of known particle size distribution and record the date and results. In the absence of a reference material, use a known sample from a previous run.

Clean the instrument thoroughly after the determination.

8.3.4 Selection of an appropriate optical model

Either the Fraunhofer approximation or the Mie theory is used for calculation of a scattering matrix, which represents the signal at each detector element per unit volume of particles in a given size class.

The choice depends upon the size range of the coating powder particles to be measured, their optical properties and shape.

Inappropriate choice of the optical model or of the values of the refractive index may result in significant bias of the resulting PSD. This bias often manifests itself as inappropriate quantities of material being ascribed to the size classes at the lower end of the size distribution.

Generally, the Fraunhofer approximation is used for coating powders, for which the details on refractive indices are not needed because the model assumes that the material absorbs all light and there is no transmission nor refraction.

Whereas the Mie theory, requires the refractive index of the powder particles specifically at the lower particle size range where light may be transmitted and also refracted. Mie model gives a better approximation of size at the micron and sub-micron range.

Instrument conditions for the measurement of the PSD shall be chosen taking into account Clause 8 or by agreement between interested parties.

9 Analysis

9.1 General

Laser diffraction systems are based on first principles and the analysis is performed on idealized spherical particles to give the size. Particle shape or different orientations of non-spherical particles will lead to different scattering patterns and thus variable particle size results. ISO 13320 provides further information.

9.2 Reference materials

Particle size measurement greatly depends on the calibration of the instrument for which certified and suitable standards are required. Powder calibration standards can be used to check the performance of the instrument and to verify coating powders with similar PSD.

For the determination of particle size during process control of similar coating powders; it may be permissable to use a coating powder sample previously measured for the same PSD and under the same analytical conditions.

9.3 Accuracy

Accuracy of coating powders can be difficult to assess and depends on the sampling of the coating powder specimen as well as the instrument conditions. After sampling, a major influence on accuracy is the dispersing mechanism of the instrument which uses compressed air or vacuum to create a semi homogeneous dispersion of coating powder particles for measurement by the laser. During dispersion, the coating powder particles may be reduced by mechanical and/or physical stresses which occur due to the applied air pressure, it should be noted that incomplete dispersion will also influence the accuracy. In an ideal situation, a homogeneous dispersion of spherical particles of similar particle size can be expected. However, not all coating powders are manufactured in the same manner leading to inhomogeneous powder particles and PSD.

The effects of physical stress caused by air pressure to disperse the coating powder should be determined to find a suitable dispersion pressure. A measuring sequence can be performed in 0,5 bar increments over the complete pressure or vacuum range appropriate for the coating powder. The dispersion pressure/vacuum yielding the lowest change in the fine particle fraction, of less than 10 µm, shall be selected for the measurements and subsequent analysis.

9.4 Precision

9.4.1 Repeatability

With suitable dispersing conditions (see 9.2) as well as a consistent measurement procedure, the repeatability for the same coating powder should have the following relative standard deviation (RSD) with respect to the cumulative powder particle volume. The RSD below may be used or as agreed between the interested parties for a particular coating powder. ISO 13320 provides further information.

For median particle size x_{10}, representing 10 % particle volume, the RSD is ≤3 %.

For median particle size x_{50}, representing 50 % particle volume, the RSD is ≤1 %.

For median particle size x_{90}, representing 90 % particle volume, the RSD is ≤3 %.

RSD values ≤3 % signify an instrument under control, consistent measurement and a good sampling procedure.

9.4.2 Reproducibility

The reproducibility of similar coating powders is influenced by the manufacturing process, sampling and the dispersion mechanism. Due to the variability described in Clause 9, the reproducibility can be low if no special precautions are taken. For improvement of the reproducibility it is essential to have consistent sampling and to align the dispersing conditions between the coating powder samples. The acceptance of measured results obtained under reproducibility conditions should be agreed between the interested parties.

10 Error sources

A detailed error evaluation is specified in ISO 13320.

Some of the errors relevant to coating powders have been described in Clause 8 and Clause 9. The most important is the sampling of the coating powder and similar coating powders which are produced via different manufacturing methods.

11 Expression of results

Express the results as specified in ISO 9276-1 and ISO 9276-2. Calculate the values x_{10}, x_{50}, x_{90}, and other percentile size values of interest in the range x_{10} to x_{90} by agreement. Report all details of the operation and results.

12 Test report

Report results as specified in ISO 9276-1, ISO 9276-2, and ISO 9276-4 such that measurements can be repeated by different operators in different laboratories. Further information may be added from ISO 13320 or as agreed between the interested parties.

Characteristic size values below x_5 and above x_{95} are likely to be vulnerable to uncertainty due to the limitations of laser diffraction.

Quoting the x_{100} value by laser diffraction shall not make reference to this document.

The report shall contain at least the following information:

a) a reference to this document, i.e. ISO 8130-13:2019;

b) sample:

   1) complete sample identification, such as coating powder type, batch number, date and time of sampling, etc.,

   2) sampling procedure and deviation from ISO 15528,

   3) sample pre-treatment (optional), e.g. pre-sieving, type and conditions,

   4) sampling during production or after *x* hours,

   5) amount of sample,

c) dispersion:

   1) details of the dispersing device, e.g. diameter of delivery tube, primary pressure,

   2) type of dosing/feeding device,

   3) dosing rate,