
Coating powders —

**Part 15:
Rheology**

*Poudres pour revêtement —
Partie 15: Rhéologie*

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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).

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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*.

A list of all 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.

Introduction

The powder coating process is dependent on several material variables, whose presence within the powder mixture is critical for ensuring smooth operation. A precise understanding of the fluidization behaviour is important for optimal processing parameters in the fluidized bed, during pneumatic conveyance and the spraying process. In addition, understanding the long-term stability during storage or behavioural change during processing can also help to improve the reuse of powder coatings. Furthermore, melt rheology is introduced to understand the melt flow properties of a given powder coating system, to provide information after spraying, on the powder melt viscosity and gel point. The methods in this document can be used for quality-control and detailed studies.

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Coating powders —

Part 15: Rheology

1 Scope

This document specifies methods for the determination of the rheological behaviour of a coating powder both in particulate and molten form.

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 3219-1, *Rheology — Part 1: Vocabulary and symbols for rotational and oscillatory rheometry*

ISO 3219-2, *Rheology — Part 2: General principles of rotational and oscillatory rheometry*

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

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 3219-1, ISO 8130-14 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

apparent viscosity

flow resistance of the fluidized powder bed

4 Principle

4.1 Powder

The different fluidization properties are determined in a rheometer. In addition to rotational speed and torque, the airflow rate through the powder bed can be precisely controlled and the pressure drop determined. This allows complete analysis of all fluidization parameters that influence the powder coating process.

4.2 Melt

In order to characterize the powder in its molten form, a rheometer in accordance with ISO 3219-2 shall be used to determine the melt and polymerization properties.

5 Apparatus

Use ordinary laboratory apparatus, together with the following.

5.1 Rheometer, in accordance with ISO 3219-2, equipped with a cylindrical measurement cell with a minimum diameter of 50 mm and a minimum height of 100 mm, ideally transparent and coated with conductive material to prevent electrostatic charging to maintain visibility. As a measurement system, a rectangular stirrer of minimum of 10 mm × 30 mm measurement surface is recommended unless otherwise specified.

For particulate measurements, a mass flow controller capable of generating an airflow from 0,05 l/min to at least 5 l/min is connected to ensure a uniform fluidization. A pressure transducer is required if pressure drop measurements shall be gathered.

For molten measurements, a disposable plate-plate geometry in a rheometer according to ISO 3219-2 with a heated plate and hood is advised.

5.2 Measurement geometry

For fluidization, deaeration and apparent viscosity measurements, use a profiled cylinder. Alternatively, measure in an undisturbed bed, without any measurement system inserted.

For cohesion strength measurements, a rectangular stirrer with a minimum measurement surface of 10 mm × 30 mm is recommended.

Use of a disposable plate of at least 25 mm diameter is advised in molten measurements.

NOTE A plate of smaller diameter can give spurious results.

6 Sampling

Take a representative sample of the product under test as specified in ISO 15528.

A minimum sample volume for powder particulate rheology measurements of 80 ml is recommended.

For further measurements of the same powder, it is recommended to keep a consistent mass.

For melt rheology measurement, it is advised to fill the plate-plate gap with the excess particulate powder, e.g. × 15 the volume, and then compress it prior to melting.

7 Experimental

7.1 Powder measurements

7.1.1 Fluidization measurements

Carry out the fluidization measurement using the profiled cylinder and perform it in two modes, i.e. unstirred and stirred, e.g. rotational speed 8 min⁻¹. First, decrease the air flow from 5 l/min to 0 l/min continuously, within a minimum of 30 s and then increase again from 0 l/min to 5 l/min.

This measurement is carried out to find the volumetric flow rate i.e. the volume of gas per time necessary for subsequent testing, as well as the volumetric flow rates for incipient and full fluidization of the sample.

NOTE For explanations of transitions such as incipient and full fluidization, see [A.1](#).

The measurements shall be performed first unstirred, then stirred.

A pressure transducer is required for pressure drop measurements by taking a baseline measurement with the same parameters in the empty cell immediately prior to measurement giving absolute results.

7.1.2 Fluidized bed rheology measurements

Carry out fluidized bed rheology measurements with a profiled cylinder system. The rotational speed shall be increased from $0,1 \text{ min}^{-1}$ to $1\,200 \text{ min}^{-1}$, corresponding to an applied shear rate increase from $0,02 \text{ s}^{-1}$ to 320 s^{-1} .

Calculate the powder's apparent viscosity in a manner analogous to that of a non-Newtonian fluid. To this end, the dimensions of the measuring chamber (e.g. 50 mm diameter) and the profiled cylinder (e.g. 17 mm diameter, no cone at the bottom) shall be measured in accordance with ISO 3219-2. Set the data gathered so that each data point is equivalent to at least one full revolution of the cylinder.

7.1.3 Deaeration measurements

Measure bed deaeration by first stirring and fluidizing the powders with an airflow of 5 l/min for 5 min at 8 min^{-1} , then stop the airflow and obtain the pressure data under the powder bed.

7.1.4 Cohesion strength measurements

Cohesion strength describes the internal resistance of the powder to flow, and thereby a measure of powder flowability. It is defined as a measure for the strength of the bonding forces between powder particles.

Carry out cohesion strength measurements as a function of torque with a two-blade stirrer after a steady-state flow is achieved. Precondition the coating powder to remove residual tension by stirring at 8 min^{-1} with an airflow retrieved from full fluidization measurements of the powder.

The cohesion strength can be calibrated using a constant geometry factor, e.g. using a certified reference material (CRM) such as CRM-116 limestone powder at a 3 kPa preconditioning (see [A.2](#) for further details).

The calculation method relies on torque measurements made only when a steady-state flow is achieved.

NOTE The final 20 points of an analysis are usually averaged and used to compare the different samples. Steady-state flow is usually reached within the given time of 100 s.

7.2 Melt rheology measurements

Use a rheometer using a disposable plate-plate geometry with a heated hood and plate.

Set the gap to 0,5 mm, the heating rate to 3,5 K/min with a temperature range between 50 °C and 180 °C, constant oscillatory parameters to 1 Hz and minimum 0,032 5° in amplitude.

8 Expression of results

Apparent viscosity of the fluidized bed is measured over different shear rates according to the characteristics of the powder.

For information on the interpretation of test results, see [Annex A](#).

9 Precision

For fluidized bed rheology measurement, a torque accuracy of 1 nNm and under is recommended.

For cohesion strength measurements in powder coatings, a torque accuracy of 1 μNm and under is recommended.

The powder illustrated in [Figure A.2](#) (fresh) has a repeatability and reproducibility of 1 %.

If the same powder is repeatedly measured and the cohesion strength decreases, this is an indication of segregation. For consistent results, use fresh powder for each measurement.

A reference material is not available for fluidized measurements since the only available certified reference material is too cohesive to be fluidized (see [A.2](#) for details).

10 Test report

The report of results shall contain at least the following information:

- a) all details necessary to identify the powder product tested;
- b) a reference to this document, i.e. ISO 8130-15:2023;
- c) the test method used;
- d) the initial mass of the sample;
- e) the result for the test sieve used, as indicated in [Clause 8](#);
- f) any deviation from the test method specified;
- g) any unusual features (anomalies) observed during the test;
- h) the date of the test.

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Annex A (informative)

Interpretation of results

A.1 Fluidization measurements (duration 15 min)

A.1.1 General

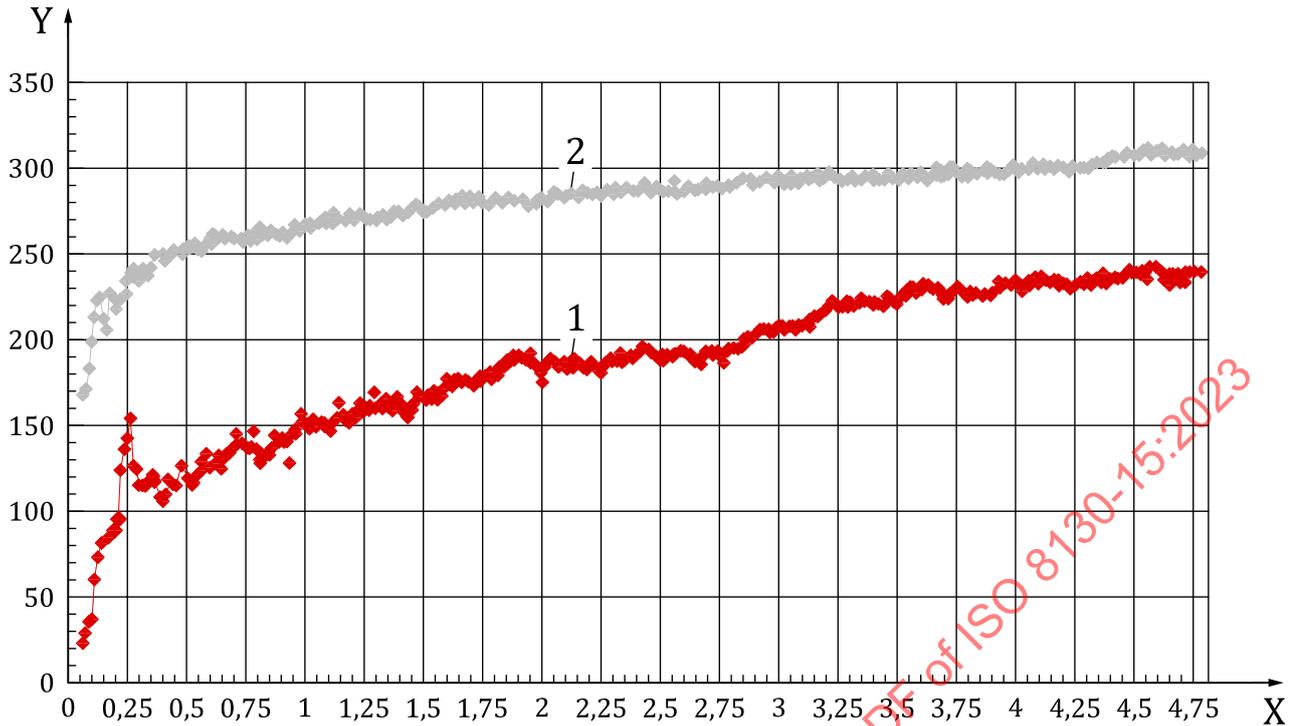
[Figure A.1](#) shows an example of fluidization from 0 l/min to 5 l/min of a powder coating as is (curve 1). This is the same sample containing 1 per mille of fumed silica (curve 2).

A.1.2 Incipient fluidization

The pressure inside the measuring cell rises with increasing volumetric flow, due to the powder particles creating and increasing counter-pressure against the fluidization air. The powder starts to fluidize, once a certain volumetric flow is reached depending on the particle characteristics. An overshoot can be detected to indicate the “incipient fluidization” as depicted in [Figure A.1](#) by curve 1 at 0,26 l/min and curve 2 at 0,16 l/min.

A.1.3 Full fluidization

At this volumetric flow the powder is fully fluidized. This can be recognized by the air flow from which a linear pressure signal is observed as shown in [Figure A.1](#) by curve 1 at 4 l/min and curve 2 at 1,16 l/min. If a sample fluidization behaviour is known, this measurement is not necessary.



Key

- X volumetric flow rate Q , in litres per minute
- Y pressure p , in pascals
- 1 powder coating as is
- 2 same sample with 1 permille of fumed silica added

Figure A.1 — Example of the upward fluidization

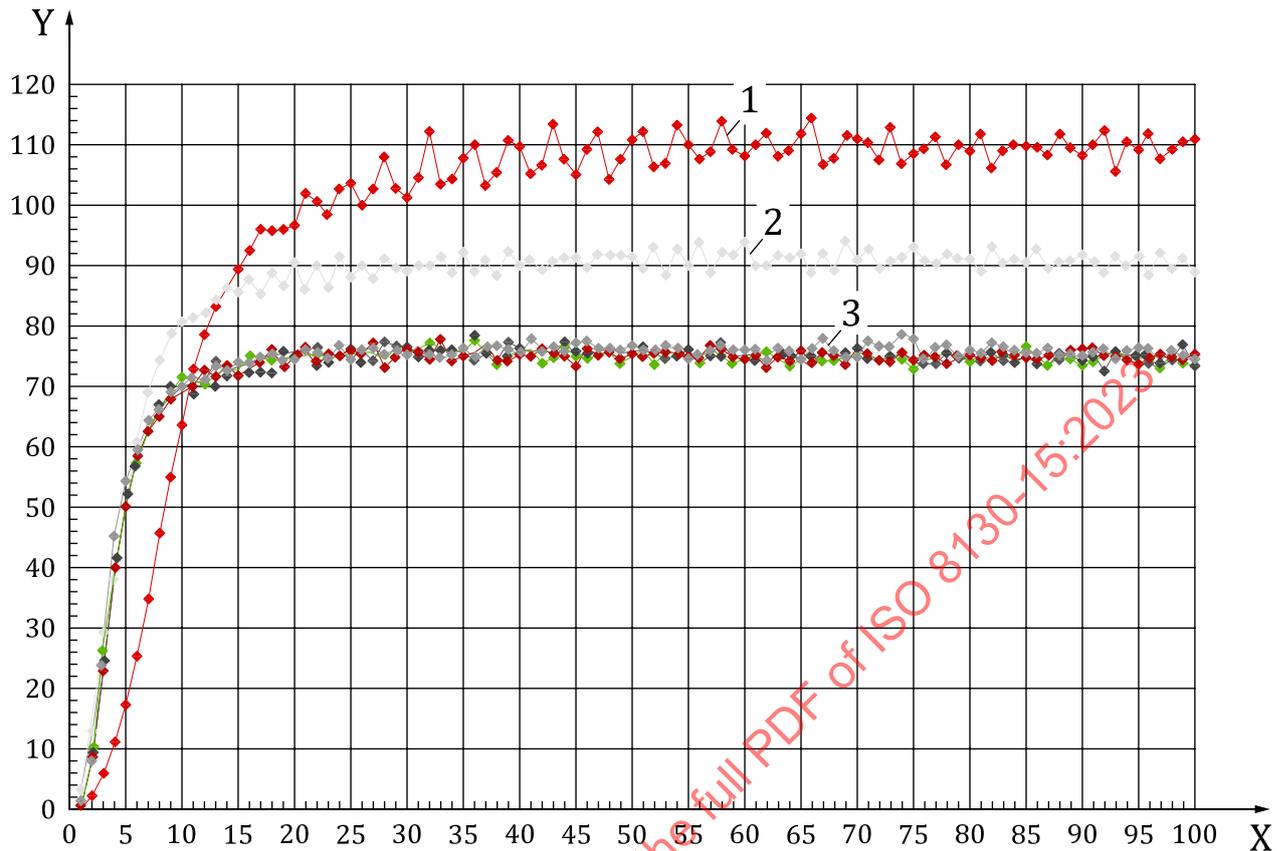
A.2 Cohesion strength measurements (5 min)

Figure A.2 shows cohesion strength measurements of degraded and fresh powders.

Cohesion strength can be used to establish a range of values that are usable within a given setup. It is essentially a constant velocity torque measurement with the cohesion strength being a constant value calibrated by comparison with Jenike shear cell data, according to ASTM D6128. While not an absolute measurement like shear cells, it is highly sensitive, quick and very repeatable, making it a useful continuous monitoring tool.

Cohesion strength is analysed by the slope at the beginning of the measurement (interval time 0 s to 20 s in Figure A.2). This cohesion strength is influenced by factors such as the powder’s aeration behaviour, air holding capacity and packing behaviour. A lower starting point indicates a higher air-holding capacity. A flatter slope increase can stem from a higher air-holding capacity, but also from the powder’s aeration behaviour from stirring. After a certain measurement time, easy- and free-flowing powders should display a constant torque and thereby constant cohesion strength. In contrast, cohesive and very cohesive powders do not necessarily show the same behaviour and it is possible that the torque does not become constant during the standard measurement time. In this case, an increase in the measurement time will help.

Cohesion strength is a relative measure and is calculated by the measured torque value and a geometry-specific factor [(for controlled shear stress (CSS)]. The torque value is determined by using a linear regression over the last 20 data points (see Figure A.2), whereas the CSS factor is given by the intercept. It is a calibration measurement with calcium carbonate (CRM-116 limestone powder), given by applying a stress of 3 kPa and no strain during measurement.



Key

- X interval time t_{intv} in seconds
- Y cohesion strength S , in pascals
- 1 degraded powder
- 2 degraded powder with 0,5 % fumed silica as a flow aid
- 3 five measurements of undegraded powder, illustrating reproducibility

Figure A.2 — Cohesion strength measurements

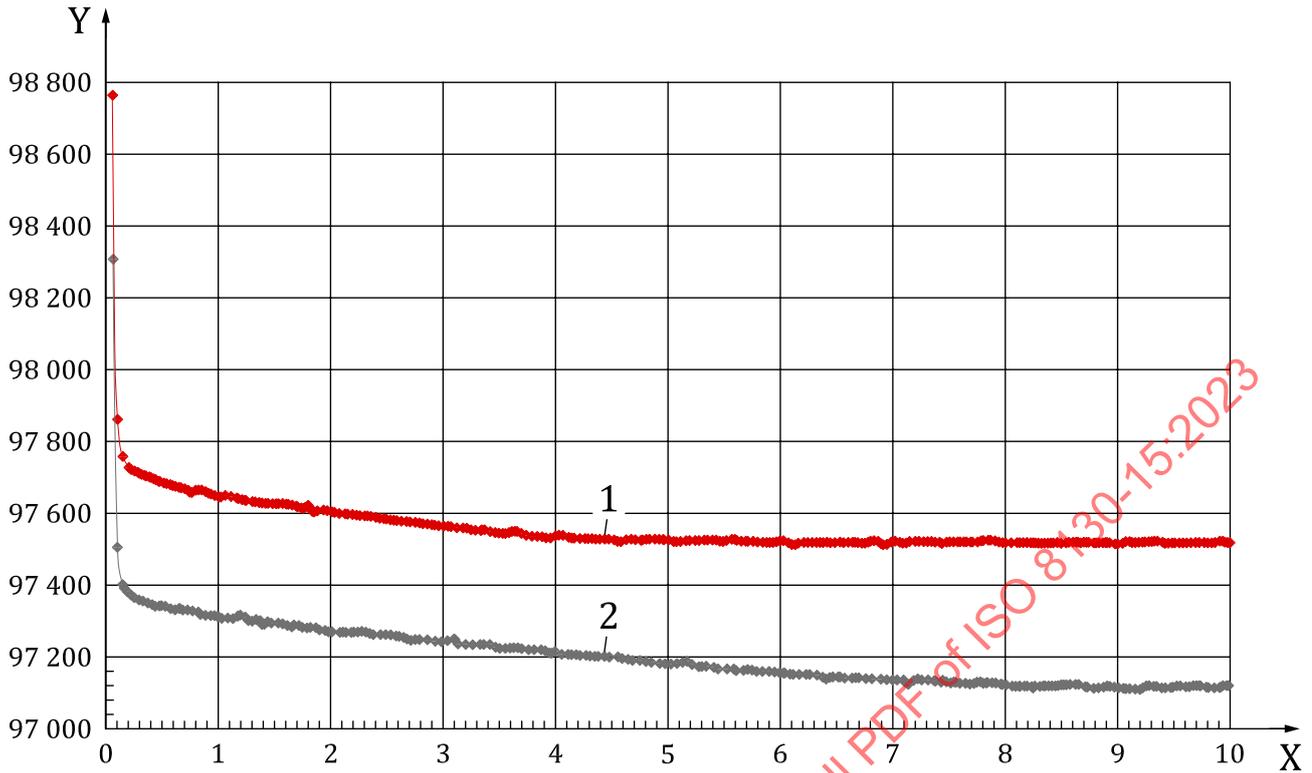
A.3 Deaeration measurements (3 min)

These measurements are performed to understand the air retention capacity of powders. In general, to achieve dense phase flow, it is necessary to have a substantial air retention capability in the powder system since this is the preferred mode of transport. The air-retention describes the duration of the powder bed to settle after fluidization and is analysed by reading the pressure signal.

Figure A.3 shows a comparison of defluidization behaviour of a non-heat treated system that shows a dramatic increase in air retention capability with minimal addition of fumed silica (curve 2). At the point where the pressure becomes constant, the powder is defluidized e.g. 4 s for curve 1 and 9 s for curve 2.

Combined with the fluidization experiments, both compositions are expected to exhibit dense phase fluidized flow and can be considered suitable for pneumatic transport. The compositions therefore exhibit suitability for usage within a powder coating process.

A powder which is transported in dense phase fluidized flow should have an air retention time of at least 3 s.



- Key**
- X interval time t_{int} in seconds
 - Y pressure p , in pascals
 - 1 defluidization point 4 s
 - 2 defluidization point 9 s

Figure A.3 — Comparison of defluidization behaviour of a non-heat-treated system

A.4 Fluidized bed rheology measurements (45 min to 90 min)

The fluidized bed rheology gives the apparent viscosity of a fluidized bed. In essence, any fluidized bed can show at least one order of magnitude difference between high and low shear (1 s^{-1} to 300 s^{-1}). This means that where the shear rate is dramatically increased, for example, through narrow gaps or kinks, then either care and/or modification is necessary to ensure the capability to spray or convey the bulk powder.

Figure A.4 shows the shear rate increase of unmodified (curves 1, 3 and 6) and a powder coating with fumed silica added (curves 2, 4 and 5) at 0 l/min, 0,1 l/min and 2,5 l/min (top to bottom respectively).