

INTERNATIONAL STANDARD

ISO
8130-8

First edition
1994-08-15

Coating powders —

Part 8:

Assessment of the storage stability of
thermosetting powders

Poudres pour revêtement —

*Partie 8: Estimation de la stabilité au stockage des poudres
thermodurcissables*



Reference number
ISO 8130-8:1994(E)

Foreword

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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 8130-8 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 pyknometer (referee method)*
- Part 3: *Determination of density by liquid displacement pyknometer*
- 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*

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

- *Part 9: Sampling*
- *Part 10: Determination of deposition efficiency*
- *Part 11: Inclined plane flow test*
- *Part 12: Compatibility*
- *Part 13: Terms and definitions*

Annexes A and B form an integral part of this part of ISO 8130.

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Introduction

Coating powders are subject to two distinct ageing mechanisms: one involving the physical state of the powder and the other its chemical reactivity. Changes in the coating powder may lead to deterioration in the physical and chemical properties of the final coating.

This part of ISO 8130 describes the procedures to be adopted in assessing the tendency of a thermosetting coating powder to maintain its physical and chemical integrity after being subjected to defined storage conditions.

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Coating powders —

Part 8:

Assessment of the storage stability of thermosetting powders

1 Scope

This part of ISO 8130 deals with the estimation of the storage stability of thermosetting coating powders. It specifies the procedures for determining the changes both in the physical state of a thermosetting coating powder and in the chemical reactivity of the powder, together with its capacity to form a satisfactory final coating. A correlation between changes in different properties is not to be expected. Similarly, there may be no correlation between the results obtained under different storage conditions.

The results of the procedures specified in this part of ISO 8130 give an indication of the ability of the coating powder to withstand the effects of storage prior to application.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8130. 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 8130 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 1514:1993, *Paints and varnishes — Standard panels for testing.*

ISO 2808:1991, *Paints and varnishes — Determination of film thickness.*

ISO 2813:1994, *Paints and varnishes — Determination of specular gloss of non-metallic paint films at 20 degrees, 60 degrees and 85 degrees.*

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

ISO 6272:1993, *Paints and varnishes — Falling-weight test.*

ISO 8130-6:1992, *Coating powders — Part 6: Determination of gel time of thermosetting coating powders at a given temperature.*

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

3 Principle

The thermosetting coating powder is subjected to artificial storage conditions for a specified period of time at a defined temperature. Subsequently, any change in the ability of the powder to flow freely and its tendency to agglomerate or to cake according to a given rating scheme is noted. Any change in the ability of the powder to react chemically and to form a satisfactory final coating is then assessed.

Conditions at the bottom of the container may be simulated by placing a weightpiece on the test portion.

NOTE 1 The demonstration that one property of the material has deteriorated to an extent that it is unsatisfactory may make further tests unnecessary.

4 Required supplementary information

For any particular application, the test method specified in this part of ISO 8130 needs to be completed by supplementary information. The items of supplementary information are given in annex A.

5 Apparatus

5.1 Air-circulating oven, capable of being maintained at $(30 \pm 0,5) ^\circ\text{C}$ or $(40 \pm 0,5) ^\circ\text{C}$.

A water bath may also be used, but the samples shall be carefully sealed to protect against the ingress of water.

5.2 Test tubes, of glass, nominally 200 mm long and 40 mm external diameter.

5.3 Test-tube stoppers.

5.4 Test-tube stand that does not impede air (or water) circulation.

5.5 Standard loads, of mass (100 ± 1) g.

NOTE 2 A length of steel rod of diameter sufficient to fit closely within the test tubes (5.2) but without touching the sides is suitable.

5.6 Aluminium-foil discs, of diameter sufficient to fit closely within the test tubes (5.2) but without touching the sides.

5.7 Balance, capable of weighing 100 g to within 0,1 g.

5.8 Test panels, as described in annex B.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 8130-9.

7 Procedure

7.1 Preliminary examination

7.1.1 Determination of initial chemical reactivity of the powder

Determine the gel time of the sample in accordance with ISO 8130-6 at the temperature specified for the product under test and record the results.

7.1.2 Determination of initial physical and chemical properties of the coating

Prepare and coat at least three test panels (5.8), as described in annex B. Discard any test panels where the coating shows signs of pinholing, cratering or cracking. Retain one of the coated test panels for use when comparing the coating made (see 7.3.3) after the artificial-storage treatment of the product (see 7.2). Determine the specular gloss of the coating on two of the remaining panels at an angle of 20° , 60° or 85° depending on the gloss level of the coating, by the procedure described in ISO 2813. Then determine the resistance of the coating to deformation by falling weight using two of the remaining panels and an agreed procedure from ISO 6272.

If agreed between the interested parties, other tests may be specified to establish the initial properties of the product under test.

7.2 Artificial-storage treatment

7.2.1 Unless otherwise agreed, the temperature and respective times at which the product is maintained prior to assessment of its storage stability shall be as follows:

- a) $(30 \pm 1) ^\circ\text{C}$ for 7 days, 28 days and 2 months or
- b) $(40 \pm 1) ^\circ\text{C}$ for 24 h, 7 days and 28 days.

7.2.2 Check that the product under test is not agglomerated and, if necessary, pass it through a sieve of appropriate aperture to disperse the sample into its constituent particles.

7.2.3 For each artificial-storage condition (7.2.1) carry out the procedure in triplicate. Weigh (100 ± 1) g of the sample into a test tube (5.2). Holding the test tube vertically, gently tap the bottom on a firm surface to ensure that the powder is not loosely packed. The simulation of conditions at the bottom of a container, if agreed, shall be achieved by application of a standard load (5.5) on an aluminium-foil disc (5.6). If it is agreed not to carry out this pro-

cedure, a note to this effect shall be included in the test report. If appropriate, place the aluminium-foil disc carefully on the surface of the test portion and then gently place one of the standard loads on the disc. Stopper the test tube securely and place the tube in the test-tube stand (5.4).

Transfer the stand and tubes to the oven (5.1), previously set to the specified test temperature. Leave for the specified period of time. Remove the stand and tubes and allow to cool to $(23 \pm 2) ^\circ\text{C}$ for at least two hours.

7.3 Final examination

7.3.1 Change in physical appearance of the powder

Turn the first test tube into a horizontal position, remove the stopper, gently tap the end containing the coating powder and note whether the coating powder flows freely.

Tip the coating powder on to a clean surface and note any evidence of compaction or agglomeration. Record the findings in accordance with table 1.

Repeat for the other two test tubes. Combine the three test portions and mix thoroughly.

7.3.2 Determination of chemical reactivity of the powder

Determine, in duplicate, the gel time on a test portion of the combined test sample, following the procedure described in 7.1.1. Record the results.

7.3.3 Determination of physical and chemical properties of the coating

Prepare and coat test panels (5.8) as described in annex B and carry out the tests as described in 7.1.2.

Compare the appearance of the coatings made from the treated coating powder with that of the retained coated test panel (see 7.1.2).

If additional tests have been carried out on the initial sample (see 7.1.2, last paragraph), carry out equivalent tests on the artificially stored samples.

Table 1 — Assessment of the coating powder

| Rating | Extent of compaction or agglomeration |
|-----------------|--|
| 0 | No change. |
| 1 | Slight evidence of compaction. Agglomerates of powder can easily be broken down. |
| 2 | Significant compaction such that some effort is required to disperse the coating powder. Agglomerates are capable of being broken down by application of hand pressure. |
| 3 ¹⁾ | Considerable compaction such that it is difficult or impossible to disperse the coating powder. Agglomerates are very stable such as to require the use of mechanical energy to break them down. |

1) If a product is recorded at rating 3, the need for further testing should be assessed as the coating powder is unlikely to be satisfactory in use.

8 Expression of results

8.1 Check that the degree of compaction and agglomeration (see 7.3.1) of the three test portions agrees to within one rating unit.

If the results differ by more than this limit, repeat the tests on a fresh test portion.

Report the arithmetic mean of the results to within half a rating unit (i.e. 0,5; 1,5; 2,5).

8.2 Calculate the percentage change in reactivity (i.e. gel time), c , using the equation:

$$c = \frac{t_0 - t_1}{t_0} \times 100$$

where

t_0 is the mean initial gel time, in seconds, of the sample;

t_1 is the mean gel time, in seconds, of the sample after the artificial-storage treatment.

Report the results to the nearest integer.

8.3 Report any difference in the appearance of the coating produced from the treated coating powder (see 7.3.3) when compared with the coating produced from the untreated coating powder (see 7.1.2).

8.4 Report any difference in mean specular gloss and resistance to deformation by falling weight of the coating produced from the treated coating powder (7.3.3) when compared with that from the untreated coating powder (7.1.2).

8.5 Report any difference in the mean results of any other tests of the coating produced from the treated coating powder (see 7.3.3, last paragraph) when compared with that from the untreated coating powder (see 7.1.2, last paragraph).

9 Precision

No precision data are currently available.

10 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 8130 (ISO 8130-8);
- c) the items of supplementary information referred to in annex A;
- d) a reference to the international or national standard, product specification or other document supplying the information referred to in c) above;
- e) whether any pretreatment of the sample (i.e. sieving) was used;
- f) the results of the tests as indicated in clause 8;
- g) any deviation from the test methods specified;
- h) the date of each test.

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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) Material (including thickness) and surface preparation of the substrate (see annex B).
- b) Method of application of the test coating to the substrate (see annex B).
- c) Duration and temperature of stoving of the test coating before testing (see annex B).
- d) Thickness, in micrometres, of the stoved coating, and method of measurement in accordance with ISO 2808.
- e) Time elapsed between end of treatment (7.2.3) and final examination (7.3).
- f) Temperature and humidity for the tests, if different from those specified in annex B (see ISO 3270).
- g) Conditions of artificial storage (see 7.2.1).
- h) Whether pressure is to be applied to the coating powder under test (see 7.2.3).
- i) Temperature at which the gel time is to be determined (see 7.1.1).
- j) Angle at which the specular gloss is to be measured (see 7.1.2).
- k) Procedure for determining resistance to deformation by falling weight (see 7.1.2).
- l) Any additional tests to be carried out on the final coating (see 7.1.2, last paragraph).