

BS EN 16074:2011



BSI Standards Publication

Paints and varnishes — Determination of non-volatile- matter content and spreading rate of coil coating materials

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National foreword

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Paints and varnishes - Determination of non-volatile-matter content and spreading rate of coil coating materials

Peintures et vernis - Détermination de la teneur en matière non volatile et du rendement superficiel spécifique de revêtements pour prélaquage

Beschichtungsstoffe - Bestimmung des Gehaltes an nichtflüchtigen Anteilen und der Ergiebigkeit von Bandbeschichtungsstoffen

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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 16074:2011) has been prepared by Technical Committee CEN/TC 139 "Paints and varnishes", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

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1 Scope

The method specifies the gravimetric procedure for determining the non-volatile-matter content (often referred to as dry solid or weight solids) as a percentage by mass of the majority of thermally cured coil coatings and subsequently for determining the theoretical spreading rate. The method is not suitable for pure epoxy coil coatings.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 13523-0, *Coil coated metals — Test methods — Part 0: General introduction and list of test methods*

EN ISO 2811-1, *Paints and varnishes — Determination of density — Part 1: Pycnometer method (ISO 2811-1:1997)*

EN ISO 2811-2, *Paints and varnishes — Determination of density — Part 2: Immersed body (plummet) method (ISO 2811-2:1997)*

EN ISO 2811-3, *Paints and varnishes — Determination of density — Part 3: Oscillation method (ISO 2811-3:1997)*

EN ISO 2811-4, *Paints and varnishes — Determination of density — Part 4: Pressure cup method (ISO 2811-4:1997)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 13523-0 and the following apply.

3.1 spreading rate
surface area that can be covered by a given quantity of coating material to give a dry film of required thickness

[ISO 4618:2006]

3.2 non-volatile matter
NV
residue by mass obtained by evaporation under specified conditions

[ISO 4618:2006]

4 Principle

The non-volatile-matter content determination is based on the mass of dry residue of a known quantity of wet paint cured in a pan or applied on a metal panel. The spreading rate is subsequently calculated using the liquid paint density and the density of the volatile part.

5 Apparatus and materials

5.1 Analytical balance, capable of weighing to an accuracy of $\pm 0,000$ 1 g.

5.2 Flat-bottomed dish (pan), of metal or glass, inner diameter of base (75 ± 5) mm, height of the rim at least 5 mm.

Dishes having different diameters may be used by agreement between the interested parties. The actual diameter of the dish shall be within ± 5 % of the agreed value.

5.3 Mixing tool (e.g. bare wire, paperclip) to facilitate the spreading of the paint.

5.4 Methyl ethyl ketone (MEK) to facilitate the spreading of the paint.

5.5 Oven capable of reaching 200 °C with an accuracy of ± 5 °C (for the pan method) and 300 °C with an accuracy of ± 5 °C (for the drawdown method).

5.6 200 μ m thickness adhesive tape, which is easily to remove and leaves no residue.

5.7 Drawdown bar.

5.8 5 ml plastics pipette.

6 Paint sample

The sample shall be homogeneous and representative of the paint to be tested.

The sample shall be stored in a closed container until the start of the test.

The sample shall be maintained at room temperature for at least 1 h prior to testing.

7 Test procedure

7.1 Accuracy of weighing

All weighings shall be in grams, to the nearest 0,001 g.

7.2 Procedure for coatings with the exception of PVC plastisols and pure epoxy

7.2.1 Pan method

Precondition the pans and mixing tools in an oven at 110 °C and store in a dessicator prior to use.

For each sample weigh the pan together with the mixing tool. Record this mass, in grams, as P_1 .

Add a quantity as close as possible to 0,5 g or 2 g (± 5 %) as agreed, accurately weighed, of paint to the pan. Weigh the pan with the paint and mixing tool. Record this mass, in grams, as P_2 .

Calculate the exact mass X , in grams, of paint added following Equation (1):

$$X = P_2 - P_1 \quad (1)$$

Add approximately 2 ml of methyl ethyl ketone (MEK) to the paint in the pan and spread the paint across the pan, using the mixing tool. Avoid any skin formation, contamination or waste of paint, contact of the paint with any other object, e.g. gloves.

Evaporate the MEK at room temperature for (30 ± 5) s. Do not remove the mixing tool.

Place the pan with paint and mixing tool in an oven at (165 ± 5) °C for 1 h 30 min (± 10) s).

Remove the pan and allow it to cool to room temperature. Do not water cool.

Weigh again the pan, with dry paint and mixing tool. Record this mass, in grams, as P_3 .

Calculate the non-volatile-matter content Y , in grams, following Equation (2):

$$Y = P_3 - P_1 \quad (2)$$

Calculate the non-volatile-matter content Z , as percentage by mass, following Equation (3):

$$Z = \frac{Y}{X} \times 100 \quad (3)$$

Repeat the procedure twice.

The dry solids content as percentage by mass is the average of three individual measurements (three pans may be put in the oven at the same time).

7.2.2 Drawdown method

Use an aluminium test panel of typical size 100 mm \times 70 mm \times 0,45 mm. The panel shall be flat, uncoated and clean. Degrease the panel with acetone, dry it in the laboratory oven and let it cool to room temperature.

For each sample determine the mass of the uncoated test panel. Record this mass, in grams, as P_1 .

Apply one drop of wet paint from a pipette to the panel and draw down according to the paint specification and weigh immediately the panel. Record this mass, in grams, as P_2 .

Cure the panel using the laboratory oven, according to the paint specification. Allow the test panel to cool to room temperature and weigh the panel. Record this mass, in grams, as P_3 . Do not water cool.

Calculate the mass X , in grams, of paint following Equation (4):

$$X = P_2 - P_1 \quad (4)$$

Calculate the non-volatile-matter content Y , in grams, following Equation (5):

$$Y = P_3 - P_1 \quad (5)$$

Calculate the non-volatile-matter content Z , as percentage by mass, following Equation (6):

$$Z = \frac{Y}{X} \times 100 \quad (6)$$

Repeat the procedure twice.

The non-volatile-matter content as percentage by mass shall be expressed as the average of three individual measurements (three panels may be put in the oven at the same time).

7.3 Procedure for PVC plastisol

7.3.1 Pan method

Precondition the pans and mixing tools in an oven at 110 °C and store in a dessicator prior to use.

For each test sample weigh the pan together with the mixing tool. Record this mass, in grams, as P_1 .

Add a quantity as close as possible to 0,5 g or 2 g ($\pm 5\%$) as agreed, accurately weighed, of paint to the pan. Weigh the pan with the paint and mixing tool. Record this mass, in grams, as P_2 .

Calculate the exact mass X , in grams, of paint added following Equation (7):

$$X = P_2 - P_1 \quad (7)$$

Spread the paint across the pan, using the mixing tool. Avoid any skin formation, contamination or waste of paint, contact of the paint with any other object, e.g. gloves.

Place the pan with paint and mixing tool in an oven at (105 ± 5) °C for 1 h (± 10 s).

Remove the pan and allow it to cool to room temperature. Do not water cool.

Weigh again the pan, with dry paint and mixing tool. Record this mass, in grams, as P_3 .

Calculate the non-volatile-matter content Y , in grams, following Equation (8):

$$Y = P_3 - P_1 \quad (8)$$

Calculate the non-volatile-matter content Z , as percentage by mass, following Equation (9):

$$Z = \frac{Y}{X} \times 100 \quad (9)$$

Repeat the procedure twice.

The dry solids content as percentage by mass is the average of three individual measurements (three pans may be put in the oven at the same time).

7.3.2 Drawdown method

Use a panel, typically hot dip galvanized steel, size 100 mm \times 70 mm \times 0,65 mm. The panel shall be flat, suitably primed and clean.

Determine the mass of the primed test panel. Record this mass, in grams, as P_1 .

Place a strip of the adhesive tape along each side of the panel.

Protect the backing coat side with the tape.

Apply the wet paint from a pipette sufficient to cover the coating area to the panel and draw down according to the paint specification. Remove the tapes and weigh immediately the panel. Record this mass, in grams, as P_2 .

Cure the panel using the laboratory oven, according to the paint specification. Allow the test panel to cool to room temperature and weigh the panel. Record this mass, in grams, as P_3 . Do not water cool.

Calculate the mass X , in grams, of paint following Equation (10):

$$X = P_2 - P_1 \quad (10)$$

Calculate the non-volatile-matter content Y , in grams, following Equation (11):

$$Y = P_3 - P_1 \quad (11)$$

Calculate the non-volatile-matter content Z , as percentage by mass, following Equation (12):

$$Z = \frac{Y}{X} \times 100 \quad (12)$$

Repeat the procedure twice.

The dry solid content as percentage by mass shall be expressed as the average of three individual measurements (three test panels may be dried at the same time in the oven).

8 Calculation of spreading rate of the paint

Calculate the spreading rate R of the paint, in square metres per kilogram, to obtain 1 μm of dry paint film using Equation (13):

$$R = \left(\frac{X}{D} - \frac{X - Y}{S} \right) \times \frac{1000}{X} \quad (13)$$

where

X, Y are calculated in Clause 7;

D is the density (in grams per cubic centimetre) of the liquid paint, determined in accordance with EN ISO 2811-1 to EN ISO 2811-4;

S is the density (in grams per cubic centimetre) of the volatile part, also called the average specific solvent density, typically provided by the paint formulator.

9 Precision

9.1 Repeatability

Two results obtained by the same operator on different days should not differ by more than $\pm 1,5 \%$.

9.2 Reproducibility

Results obtained by different laboratories for the same batch sample should not differ by more than $\pm 3 \%$.

10 Test report

The report shall contain at least the following information:

- a) all details necessary to identify the product tested;
- b) a reference to this European Standard (i.e. EN 16074);
- c) the method used to measure density;
- d) the test method, the paint quantity and the curing conditions;
- e) the average non-volatile-matter content as a percentage by mass;
- f) the spreading rate, the density of the liquid paint and the density of the volatile part;
- g) any deviation from the method specified;
- h) any unusual features (anomalies) observed during the test;
- i) the date of the test.

Bibliography

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EN 10169, *Continuously organic coated (coil coated) steel flat products — Technical delivery conditions*

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DIN 53219, *Paints and varnishes — Determination of percentage volume of non-volatile matter*

ASTM D2369, *Standard Test Method for Volatile Content of Coatings*

ASTM D2697, *Standard Test Method for Volume Non-volatile Matter in Clear or Pigmented Coatings*

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