BS EN 12802:2011

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Road marking materials — Laboratory methods for identification

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National foreword

This British Standard is the UK implementation of EN 12802:2011. It supersedes BS EN 12802:2000 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/509/2, Horizontal road markings and road studs.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Road marking materials - Laboratory methods for identification

Produits de marquage routier - Méthodes de laboratoire pour identification

Straßenmarkierungsmaterialien - Laborverfahren für die Identifikation

This European Standard was approved by CEN on 22 April 2011.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document (EN 12802:2011) has been prepared by Technical Committee CEN/TC 226 “Road equipment”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by December 2011, and conflicting national standards shall be withdrawn at the latest by December 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 12802:2000.

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The Annexes A to H of this European Standard are normative.

This European Standard is one of a package of inter-related European Standards with a common date of withdrawal (dow) fixed on December 2011 *(including the request of an extension fo the co-existence period)*:

- EN 1790, *Road marking materials — Preformed road markings*,
- EN 1824, *Road marking materials — Road trials*,
- EN 1871, *Road marking materials — Paint, thermoplastic and cold plastic materials — Specifications*,
- EN 12802, *Road marking materials — Laboratory methods for identification*,
- EN 13197, *Road marking materials — Wear simulator Turntable,*
- EN 13212, *Road marking materials — Requirements for factory production control*,
- EN 13459, *Road marking materials — Sampling and testing*.

1 Scope

This document specifies laboratory methods for the identification of road marking materials used in horizontal signalization. It is not necessary, unless required, to perform all of the tests described.

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 1423, *Road marking materials — Drop on materials — Glass beads, antiskid aggregates and mixtures of the two*

EN 1424, *Road marking materials — Premix glass beads*

EN 1790, *Road marking materials — Preformed road markings*

EN 13459, *Road marking materials — Sampling and testing*

EN ISO 11890-2, *Paints and varnishes — Determination of volatile organic compound (VOC) content — Part 2: Gas-chromatographic method (ISO 11890-2:2006)*

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling (ISO 15528:2000)*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
paints

liquid product containing binders, pigments, extenders, solvents and additives

NOTE It can be supplied in single or multi-component systems. When applied it produces a cohesive film by the process of solvent evaporation, or solvent evaporation and a chemical reaction.

3.2
cold plastics

viscous products supplied in two or multi-component forms (at least one main component and a hardener system) and free from solvents

NOTE The cohesive film is formed after mixing of all components only by a chemical reaction. Following the reaction, the liquid becomes a solid.

3.3
thermoplastics

solvent-free marking substance supplied in block, granular or powder forms

NOTE It is heated to a molten state and then applied. It forms a cohesive film by cooling.

4 Sampling

Samples representative of each component of the material shall be taken from storage, or prior to application, in accordance with EN 13459. Smaller representative samples, of sufficient quantity to carry out all the tests required, shall be taken from the larger samples. For paints and cold plastics approximately 1 l of the basic component shall be taken.

In the case of thermoplastic in powder form, sufficient quantity shall be taken in accordance with EN 13459 so that it can be melted in a metal container and mixed to a homogeneous mass. After cooling and casting into solid sheets or blocks, representative samples of approximately 1 kg of homogeneous solid material shall be taken for testing.

5 Test methods

5.1 General

The standard test methods are listed in 5.2 to 5.4.

Alternative quantitative analytical test methods may be used providing that:

- the resulting values are comparable to those obtained using the standard methods; and,
- the repeatability of the alternative methods, determined in accordance with ISO 5725-2, can be shown to be not less than that of the methods given in this standard.

5.2 Paint

5.2.1 Density

The density of the paint shall be determined using, either the method laid down in EN ISO 2811-1, or an alternative method complying with 5.1.

5.2.2 Solids content

The solids content of the paint, expressed as a percentage, shall be determined using, either the method described in Annex A, or an alternative method complying with 5.1.

5.2.3 Organic content and identification

The type of organic materials, and the content expressed as a percentage, of the paint shall be determined using, either the method described in Annex B, or an alternative method complying with 5.1.

5.2.4 Inorganic content and identification

The type of inorganic materials, and the content expressed as a percentage, of the paint shall be determined using, either the method described in Annex C, or an alternative method complying with 5.1.

5.2.5 Titanium dioxide content

The titanium dioxide content of the inorganic compound of the paint, expressed as a percentage, shall be determined, either by the method described in Annex D, or an alternative method complying with 5.1.

5.2.6 Glass bead content

The glass bead content of the paint, expressed as a percentage, shall be determined, either as described in Annex E, or an alternative method complying with 5.1.

5.2.7 Solvent content and identification

The type of solvent, and the content expressed as a percentage, of the paint shall be determined using, either the method described in Annex F, or an alternative method complying with 5.1.

5.2.8 Viscosity

The viscosity of the paint shall be determined using, either the method described in Annex G, or an alternative method complying with 5.1.

5.2.9 Ash content

The ash content of the paint shall be determined using, either the method described in Annex H, or an alternative method complying with 5.1.

5.3 Thermoplastics

5.3.1 Density

The density of the thermoplastics shall be determined using, either the method laid down in EN ISO 2811-2, or an alternative method complying with 5.1.

5.3.2 Organic content and identification

The type of organic materials, and the content expressed as a percentage, of the thermoplastics shall be determined using, either the method described in Annex B, or an alternative method complying with 5.1.

5.3.3 Inorganic content and identification

The type of inorganic materials, and the content expressed as a percentage, of the thermoplastics shall be determined using, either the method described in Annex C, or an alternative method complying with 5.1.

5.3.4 Titanium dioxide content

The titanium dioxide content of the inorganic compound of the thermoplastics, expressed as a percentage, shall be determined, either by the method described in Annex D, or an alternative method complying with 5.1.

5.3.5 Glass bead content

The glass bead content of the thermoplastics, expressed as a percentage, shall be determined, either as described in Annex E, or an alternative method complying with 5.1.

5.3.6 Ash content

The ash content of the thermoplastics shall be determined using, either the method described in Annex H, or an alternative method complying with 5.1.

5.4 Cold plastics

5.4.1 Density

The density of the cold plastics shall be determined using, either the method laid down in EN ISO 2811-2, or an alternative method complying with 5.1.

5.4.2 Organic content and identification

The type of organic materials, and the content expressed as a percentage, of the cold plastics shall be determined using, either the method described in Annex B, or an alternative method complying with 5.1.

5.4.3 Inorganic content and identification

The type of inorganic materials, and the content expressed as a percentage, of the cold plastics shall be determined using, either the method described in Annex C, or an alternative method complying with 5.1.

5.4.4 Titanium dioxide content

The titanium dioxide content of the inorganic compound of the cold plastics, expressed as a percentage, shall be determined, either by the method described in Annex D, or an alternative method complying with 5.1.

5.4.5 Glass bead content

The glass bead content of the cold plastics, expressed as a percentage, shall be determined, either as described in Annex E, or an alternative method complying with 5.1.

5.4.6 Solvent content and identification

The type of solvent, and the content expressed as a percentage, of the cold plastics shall be determined using, either the method described in Annex F, or an alternative method complying with 5.1.

5.4.7 Viscosity

The viscosity of the cold plastics shall be determined using, either the method laid down in EN ISO 2555 with a Type A viscosimeter, or an alternative method complying with 5.1.

5.4.8 Ash content

The ash content of the cold plastics shall be determined using, either the method described in Annex H, or an alternative method complying with 5.1.

5.5 Preformed road markings

The identification methods for preformed road markings are laid down in EN 1790.

5.6 Premix glass beads

5.6.1 Granulometry

The granulometry of the glass beads shall be determined using the method laid down in EN 1424.

5.6.2 Refractive index

The refractive index class of the glass beads shall be determined using the method laid down in EN 1424.

5.6.3 Resistance to water, hydrochloric acid, calcium chloride and sodium sulphide

The glass beads shall not develop any surface haze or dulling when in contact with any of the following: water, hydrochloric acid, calcium chloride and sodium sulphide, using the method laid down in EN 1423.

5.6.4 Defective beads

The percentage of defective glass beads shall be determined using the method laid down in EN 1424.

5.6.5 Surface Treatment

The surface treatment of the glass beads shall be determined using the method laid down in EN 1424.

5.7 Drop on materials

5.7.1 Drop on glass beads

5.7.1.1 Granulometry

The granulometry of the glass beads shall be determined using the method laid down in EN 1423.

5.7.1.2 Refractive index

The refractive index class of the glass beads shall be determined using the method laid down in EN 1423.

5.7.1.3 Resistance to water, hydrochloric acid, calcium chloride and sodium sulphide

The glass beads shall not develop any surface haze or dulling when in contact with any of the following: water, hydrochloric acid, calcium chloride and sodium sulphide, using the method laid down in EN 1423.

5.7.1.4 Defective beads

The percentage of defective glass beads shall be determined using the method laid down in EN 1423.

5.7.1.5 Surface Treatment

The surface treatment of the glass beads shall be determined using the methods laid down in EN 1423.

5.7.2 Drop on antiskid aggregates

5.7.2.1 Friability index

The friability index of the antiskid aggregates shall be determined using the method laid down in EN 1423.

5.7.2.2 Granulometry

The granulometry of the antiskid aggregates shall be determined using the method laid down in EN 1423.

5.7.2.3 Colour co-ordinates and luminance factor

If the antiskid aggregate is not transparent, the chromaticity co-ordinates and the luminance factor shall be determined using the method laid down in EN 1423.

5.7.3 Mixture of glass beads and antiskid aggregates

In a mixture of glass beads and antiskid aggregates the glass beads shall conform to EN 1423 and the antiskid aggregates shall conform to EN 1423. The tests on the glass beads and the antiskid aggregates to be incorporated in mixtures shall be conducted separately before mixing.

5.8 Tolerances

Tolerances are as shown in Table 1.

For the initial testing of a material, tolerances apply to the manufacturers declared values for the properties tested and the initial test results shall be within the tolerances in Table 1.

When the values fall inside the tolerances then the values initially declared by the manufacturer are considered to be verified.

When values fall outside of the tolerances there are four possibilities:

- the manufacturer can decide that the test be terminated;
- by agreement between the participants the test can be repeated with the same samples;
- by agreement between the participants the test can be repeated with new samples;
- by agreement between the participants the manufacturer can submit revised declared values;

For re-identification of a material, it may not be necessary to test all of the values. Reference values for the properties tested shall be the values declared by the manufacturer.

Table 1 — Tolerances

Parameter	Maximum relative deviation	Maximum absolute deviation
Solids content	-	± 2
Density	- - -	Paint: ± 0,04 g/cm³ Cold plastic: ± 0,06 g/cm³ Thermoplastic: ± 0,1 g/cm³
Organic constituents	10 %	± 2
Calcium Carbonate		± 3
Inorganic constituents	-	± 3
Titanium dioxide	For TiO_2 concentrations ≥ 10 % Tolerance = 10 %	For TiO_2 concentrations ≤ 10 % Tolerance = ± 1
Glass beads*	20 %	± 5
Solvent content		± 3
Viscosity	Cold plastic: 20 %	Paint: ± 10 units
Ash Content	-	± 3
* Experimental values which are only applicable if the glass bead content is ≥ 10%. It is not possible to set tolerances if the percentage of glass beads is less than 10 %.		

NOTE When assessing the identity of two infrared spectra it has to be checked if all absorptions-/transmission peaks are present or there are additional occurrences which are significantly different from the baseline to stand out. The relative height levels between the peaks must not change significantly.

6 Test Report

At the end of the tests, the test report shall be made available. The test report shall include at least: a reference to this standard and to the test method (Annex A to Annex H); the critical testing conditions; and the expression of the results (as specified in the corresponding test method) and the related uncertainty (if applicable).

Annex A
(normative)

Paint – Test method for the determination of the solids content

A.1 Principle

The volatile constituents of the paint are evaporated at 105 °C. The residual solids are weighed and the solids content calculated.

A.2 Apparatus

a) Cooling equipment, refrigerator or water bath with thermostat at approximately + 10 °C;

b) porcelain dishes with a diameter of approximately 40 mm an a high of 20 mm to 30 mm;

c) analytical balance, with an accuracy of 0,001 g, with zero point correction;

d) warming cupboard, with forced fresh air ventilation and flame-proof interior capable of being heated to 105 °C ± 2 °C;

e) desiccator, with drying agent, e.g. silica gel.

A.3 Reagents

Thinner consisting of a suitable solvent / thinner as recommended by the manufacturer.

A.4 Procedure

A.4.1 Carry out two determinations.

A.4.2 Mark porcelain plates with numbers. Record the mass of the empty plates (L) to the nearest 0,001 g. Place approximately 2 g of the paint, pre-cooled to 10 °C and homogenized, in the plate with a spoon. Weigh to the nearest 0,001 g (mass M_1).

A.4.3 Place the plates containing the sample in a warming cupboard at a temperature of 105 °C ± 2 °C and store there for at least 3 h or until constant mass is reached. Constant mass is reached when, after the sample has been in the warming cupboard for a further 1,5 h, the mass loss is less than 0,2 % of the initial mass. Cool the plate to room temperature in the desiccator, and weigh to the nearest 0,001 g.

A.5 Test result

The percentage solids content of the paint by mass, *S,* shall be calculated according to the following equation:

$$S = \frac{100(M_2 - L_S)}{M_1 - L_S} \tag{A.1}$$

where

*M*1 is the initial mass of paint together with the empty plate, in grams;

*M*2 is the final mass of solid together with the plate, in grams;

L is the mass of the empty plate, in grams.

If the results of the individual determinations differ by more than 0,5 % by mass the procedure shall be repeated. The mean of the two individual results shall be calculated and the solids content given rounded to the nearest 0,1 % by mass.

Annex B
(normative)

Paint, thermoplastics and cold plastics – Test method for the determination and identification of organic constituents

B.1 Principle

Separation of the binder, insoluble organic and inorganic constituents from the specimen by solvent extraction, centrifuging and combustion. The binder type of paints and thermoplastics is identified by IR spectra using a potassium bromide (KBr) pellet or window.

The binder type of cold plastics is identified using EN ISO 11890-2, *Paints and varnishes — Determination of volatile organic compound (VOC) — Part 2: Gaschromatographic method (ISO 11890-2:2006)*.

The inorganic constituents are kept for further IR examination (Annex C), determination of titanium dioxide (Annex D) and the glass bead content (Annex E).

B.2 Apparatus

a) centrifuge, high-speed, min 10 000*g*1);

b) centrifuge bowls, steel bowls or glass tubes, each with a capacity of 80 cm^3;

c) spatula;

d) warming cupboard, with forced ventilation, capable of being heated to 105 °C ± 10 °C;

e) rotary evaporator;

f) glass flask, with a capacity of 500 ml, with ground glass neck;

g) infrared spectrometer, automatically recording FTIR spectrometer, range 4000 cm^{-1} to 400 cm^{-1} with an optical resolving power better than 5 cm^{-1} throughout the spectral range using an ordinate with a scale of 0 % to 100 % transmission.

h) analytical balance, with an accuracy of 0,001 g, with zero point correction;

i) two erlenmeyer flasks of heat resistant glass, 250 ml or 300 ml capacity;

j) electric muffle furnace, adjustable to 450 °C ± 25 °C;

k) desiccator, with drying agent, e.g. silica gel;

l) ultrasonic dissolver or similar device.

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1) *g* equals 9,81 m x s^{-2}.

B.3 Solvents being used

The following solvents shall be used unless otherwise stipulated by the supplier:

- for paints containing organic solvents: mixture of three parts toluene p.a. to one part acetone p.a. by volume;
- for waterborne paints: tetrahydrofurane;
- for thermoplastics: mixture of three parts toluene p.a. to one part acetone p.a. by volume or methylethylketone;
- for cold plastics: ethyl acetate;
- acetone.

B.4 Procedure

B.4.1 Separation of binder

Weigh the centrifuge bowls to the nearest 0,01 g (W_1). Weigh a portion of approximately at least 10 g to the nearest 0,01 g together with the bowl (W_2).

Place a portion of the marking material, carefully homogenized, in a centrifuge tube. Add approximately 25 ml of an appropriate solvent (see B.3). Seal the tube with a stopper and, only for thermoplastic, allow to stand overnight. After standing, stir the contents of the tube thoroughly with a spatula and centrifuge the mixture at 10 000*g* to 15 000*g* for 20 min. Decant the liquid phase into the glass flask. Thin the solid phase with a further 25 ml of solvent, stir again with the spatula, rinse and centrifuge again. Repeat the procedure three times or until the liquid supernatant is of clear colour. Use a highly volatile solvent the third time (such as acetone). Expose the centrifuge bowls containing the solid phase to the air for 5 min and dry in the warming cupboard for approximately 2 h.

Cool down the tubes in a desiccator and reweigh them to the nearest 0,01 g (W_3).

For dispersions do the extraction with tetrahydrofurane and centrifuge at a low speed of about 1000*g*, as high speeds can throw out high molecular components of the binder.

B.4.2 Preparation of spectra

Concentrate the solutions obtained in B.4.1 down to about 20 ml in the 500 ml glass flask in a rotary evaporator.

For recording the IR spectrum of the binder of a road marking material, use the binder solutions obtained in B.4.2. Coat the cuvette window or the potassium bromide (KBr) pellet (both referred to as 'pellet' below) evenly with a specimen of the solution.

Dip a previously prepared pellet into the liquid obtained from the appropriate extraction and coat it evenly with a specimen of the solution. As the solvent evaporates, examine the pellet to make sure that the film formed on it is uniform and homogeneous. There shall be no inhomogeneity or separation. In order to remove the solvent completely, store the coated pellet in a warming cupboard at 60 °C ± 2 °C until no bands of solvent are identifiable.

Place the pellet in the sample holder of the IR spectrometer and record the IR spectrum. Keep the tracing as a reference.

If the intensity of the spectrum is too weak, dip the pellet into the solution a second time, dry it and test again. Repeat the procedure until a spectrum is produced in which the strongest bands have an intensity of 5 % to 15 % transmittance. If the intensity is to strong, thin with an appropriate solvent.

Record the spectrum in such a way that the strongest absorptions have a transparency between 5 % and 15 %. The film thickness suitable for evaluation of the spectra shall be determined empirically.

B.4.3 Processing of the solids

The solids from the separation may contain insoluble organic compounds. They are ashed at 450 °C ± 25 °C in a muffle furnace and the loss in mass determined. The ash residue is used for the determination of the titanium dioxide in Annex D (initial mass M_4) and inorganic constituents in Annex C.

Carry out two determinations.

Weight a empty flask to the nearest 0.001g (W_4), Take the residue of the centrifuge tube an place it in the flask directly in the muffle furnace afterwards and raise the temperature gradually to 450 °C ± 25 °C. Keep the flask in the furnace for at least 2 h or until constant mass is obtained. Remove the flask containing the residues, cool in a desiccator and reweigh (W_5).

NOTE The determination should be carried out in a properly functioning fume hood.

B.4.4 Processing of the liquid

Weight an empty capsule to the nearest 0.001 g (W_6).

V_1 is the total volume of the liquid part obtained in B 4.1.

Take a small part of the different liquid phase collect in part B 4.1 (V_2) In order to have at the end between 1 and 3 grams of binder and put in the capsule on a hotplate at 60°C under ventilation. Continue the evaporation until constant mass is obtained (W_7). Be careful to cool the capsule before every weighting.

B.5 Test results

The binder content by mass (B), shall be determined using the following equation for thermoplastics cold plastic and paints.

$$B = \frac{W_7 - W_6}{W_8} * 100 \tag{B.1}$$

where

$$W_8 = \frac{(W_2 - W_1) \times V_2}{V_1} \tag{B.2}$$

The percentage *binder + solvent content* by mass, *B+SC,* could be determined (for information only) using the following equation for thermoplastics and reactive materials:

$$B + SC = \frac{100(W_2 - W_3)}{W_2 - W_1} \tag{B.3}$$

where

W_1 is the mass of the empty centrifuge bowl in grams;

W_2 is the mass of the bowl together with the sample in grams;

W_3 is the mass of the bowl together with the residues in grams.

The percentage of *inorganic compounds* content by mass, *I,* shall be determined using the following equation:

$$I = \frac{100(W_5 - W_4)}{W_2 - W_1} \tag{B.4}$$

The percentage *binder content* by mass, *B,* could be also determined using the following equation for paints and cold plastics:

$$B = S - (I + IC) \tag{B.5}$$

"S" is the solid content obtained in Annex A

If the results of the individual determinations of the binder or inorganic content differ by more than 0,5 % by mass from the mean, repeat the procedure. The mean of the two individual results shall be calculated and the binder content given rounded to the nearest 0,1 % by mass.

The percentage of *insoluble organic compounds* (*IC*) by mass is given by the difference:

$$IC = 100 - (I + B + SC) \tag{B.6}$$

Annex C
(normative)

Paint, thermoplastic and cold plastic – Test method for the determination and identification of inorganic constituents

C.1 Principle

The inorganic constituents obtained in B.4.3 are used for the determination.

In the case of containing glass beads and antiskid materials the pigments and fillers are separated with a standard sieve, mesh size 0,090 mm.

The pigments and fillers are determined by IR analysis using a KBr pellet.

Furthermore they are used for the determination of titanium dioxide described in Annex D.

For the determination of the carbonates – expressed as calcium carbonate – a part of the residue obtained in B.4.3 is heated in a furnace up to 1 000 ^{o}C. The loss of weight is used to calculate the carbonate content.

C.2 Apparatus

a) Analytical balance, with an accuracy of 0,001 g, with zero point correction;

b) standard sieve and bottom, mesh size 0,090 mm;

c) agate mortar and pestle;

d) desiccator, with drying agent e.g. silica gel;

e) infrared spectrometer, automatically recording FTIR spectrometer, range 4 000 cm^{-1} to 400 cm^{-1};

f) press;

g) mould, for making pellets;

h) electric muffle furnace, capable of being raised to a temperature of 1000 °C ± 20 °C;

i) porcelain basins.

C.3 Reagent

Potassium bromide in chromatographic quality.

C.4 Procedure

Carry out two determinations.

C.4.1 In the case of containing glass beads and antiskid materials separate the pigments and fillers with a standard sieve and bottom, mesh size 0,090 mm.

Pulverize the pigments and fillers in the agate mortar. It shall be a homogeneous mixture that is representative of the composition of the inorganic constituents of the sample materials.

Take a representative sample of the necessary amount and mix this sample in the agate mortar with approximately 20 mg of KBr to make a pellet from the mixture. Record an IR spectrum of the pellet. Keep the recorded spectrum for reference.

If the pellets are not to be used immediately for recording the IR spectrum, keep them over a drying agent in a desiccator.

Record the spectrum in such a way that the strongest absorption bands have a transmission between 5 % and 15 %. If the absorption intensities do not lie within this range, determine the amount of sample suitable for evaluation empirically and record a new spectrum.

C.4.1 Determination of the carbonates

Weigh the porcelain basin (M_0), add 3-6 g of the residue obtained in B.4.3 and weigh again (M_1). Heat the basin in the electric muffle furnace for 2 h at 1000 °C ± 20°C, then remove it and let it cool down in the desiccator, reweigh it (M_2).

C.5 Calculation and expression of results (carbonate content as part of the inorganic constituents)

Express the result by the arithmetic mean of two measurements, calculated as a percentage of the initial mass $M_1 - M_0$ of the test portion, using the expression:

$$\frac{M1 - M2}{M_1 - M_0} \times 100 \times 2{,}274 \qquad \text{(C.1)}$$

2,274 = stochiometric factor

Annex D
(normative)

Paint, thermoplastics and cold plastics – Test method for the determination of the titanium (IV) dioxide content in the inorganic compound

D.1 Principle

The inorganic constituents (obtained using Annex B) are dissolved in concentrated sulfuric acid and ammonium sulfate. The Ti(IV) is then reduced with aluminium, after Rahm[1] , and determined by titration with an Fe(III) solution, potassium thiocyanate being used as an indicator.

D.2 Apparatus

a) Analytical balance, with an accuracy of 0,001 g, with zero point correction;

b) desiccator, with drying agent, e.g. silica gel;

c) two erlenmeyer flasks of heat resistant glass with ground neck and stopper (45/40) and of 250 ml to 300 ml capacity;

d) sand bath, adjustable up to 300 °C;

e) water bath, for cooling the reaction mixture;

f) burette, with polytetrafluorethylene stopper, zero point setting and storage bottle of 50 ml capacity and with 0,1 ml graduations;

g) contat Göckel valve, standard ground 45/40;

h) spray bottle, with distilled water.

D.3 Reagents

a) sulfuric acid, concentrated p.a. (H_2SO_4) of density 1,84 g/ml;

b) ammonium sulfate [$(NH_4)_2\ SO_4$];

c) hydrochloric acid p.a. (HCl 25 %);

d) aluminium strip p.a. (Al);

e) ammonium iron(III) sulfate {[$(NH_4)\ Fe(S0_4)_2$] 12 H_2O} in H_2SO_4;

f) potassium thiocyanate (potassium rhodanide) p.a. (KSCN);

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[1] J.A. Rahm – Anal. Chem – 24, 1832 (1952)

g) sodium hydrogen carbonate ($NaHCO_3$);

h) titanium(**IV**) dioxide p.a. (TiO_2) as primary standard for setting the factor of the ammonium iron(**III**) sulfate solution;

i) glass boiling beads.

D.4 Preparation of the solutions

D.4.1 Fusion acid: dissolve 500 g of ammonium sulfate p.a. under heat in 740 ml of concentrated sulfuric acid p.a.

D.4.2 Ammonium iron(**III**) sulfate solution: dissolve 30,7 g of $[(NH_4)Fe(S0_4)_2]$12H2O in distilled water; add 25 ml of concentrated sulfuric acid p.a. to the solution and top up with distilled water to 1 000 ml. The molarity of the solution shall be 0,064.

The factor of the solution shall be set with TiO_2 as the primary standard (1 ml of ammonium iron(**III**) sulfate solution shall be equivalent to 5,087 mg of TiO_2). For this, dissolve 0,15 g of ignited TiO_2 p.a in approximately 15 ml of fusion acid. Continue the method as described in D.5.2 and D.5.3.

D.4.3 Potassium thiocyanate solution: a 10 % solution of potassium thiocyanate in distilled water (10 g of KSCN + 90 g of distilled water).

D.4.4 Sodium hydrogen carbonate solution: make a saturated sodium hydrogen carbonate solution by adding 100 g of $NaHCO_3$ p.a. to 1 l of distilled water. After the $NaHCO_3$ has dissolved filter the solution so that the gas pipe in the Göckel valve cannot become clogged due to excess of salt.

D.5 Procedure

D.5.1 General

Carry out two determinations.

D.5.2 Preparation of the sample and reduction of the TiO_2.

For determining the proportion by mass of titanium dioxide (TiO_2) in the inorganic constituents of a single component (or the basic component of a multi-component system) use the ashing residue of the sample, obtained in accordance with Annex B and stored in a moisture-proof place.

If reflecting materials or skid resistance materials are present it is recommended to grind these to improve homogeneity.

Weigh the amount of ashing residue (M_4) obtained in B.4.3 and put it into suspension with 10 ml distilled water and evaporate the water off on a sand bath. (This pre-wetting is intended to aid the digestion of the inorganic constituents.) Add 25 ml of fusion acid. Fuse the ashing residue for at least 10 min by boiling it in the sand bath using glass boiling beads. After cooling, first add 50 ml of dilute hydrochloric acid (HCl) and then 2 g of aluminium (Al) strip to the mixture. Close the flask immediately with a Göckel valve filled with the saturated sodium hydrogen carbonate solution ($NaHCO_3$).

NOTE 1 There should be no solid phase in the Göckel valve so that the gas pipe in the valve cannot become blocked, or the apparatus could explode due to the gas generated.

NOTE 2 The acids in the flask dissolve the aluminium with the release of hydrogen. There may be no naked flames near the flask.

When the aluminium reacts with the acid, titanium(IV) is reduced to titanium(III). If no gas is generated, carefully warm the reduction mixture. As soon as gas is generated, slow down and control the reaction by cooling the flask in a water bath. When the flask is cooling, sodium hydrogen carbonate may be sucked into it from the Göckel valve, with subsequent release of carbon dioxide that serves as a protective gas. Titanium(III) is sensitive to oxidation and is also oxidized by atmospheric oxygen. In order to prevent uncontrolled oxidation of the titanium(III), no oxygen shall be allowed into the flask. The Göckel valve shall, therefore, always be kept filled with sodium hydrogen carbonate solution. After the aluminium has largely dissolved, carefully bring the mixture to the boil and keep boiling until all of the aluminium has dissolved. Carefully cool the flask in a water bath, during which sodium hydrogen carbonate solution is sucked into the flask which ensures that it is filled with protective carbon dioxide (CO_2).

NOTE 3 Both the fusion of the residues in the flask with concentrated sulfuric acid and the reduction procedure should be carried out in a properly functioning fume hood.

D.5.3 Titration

After the reduction mixture has cooled, take the Göckel valve off the flask and rinse the ground surfaces of the valve and flask with a little distilled water.

NOTE During this and the subsequent titration it is advisable to pass a protection gas (CO_2, N_2, or a rare gas) into the flask.

Add immediately approximately 5 ml of potassium thiocyanate solution to the solution, which is violet due to reduction. Carry out the titration straight away with the ammonium iron(III) sulfate solution. Towards the end of titration the reaction takes place more slowly, therefore add the titrating solution drop by drop. The end point is reached when the weak red-brown colouring has stayed for at least 1 min. Deduct the final amount of ammonium iron(III) sulfate solution that was needed to indicate the end point (e.g. 0,05 ml) from the amount of solution consumed. Record the volume of the titrating solution used (V) in millilitres.

D.5.4 Test result

The percentage by mass of TiO_2, T, shall be calculated from the consumption of ammonium iron(III) sulfate solution according to the following equation:

$$T = \frac{100(V \times N \times F)}{M_4} \tag{D.1}$$

where

V is the volume of ammonium iron(III)sulfate solution in millilitres;

M_4 is the initial mass of the material;

N is the TiO_2 equivalent of the ammonium iron(III) sulfate solution;

F is the the factor, if any, of the ammonium iron(III) sulfate solution.

If the results of the individual determinations differ from one another by more than 0,5 % by mass the procedure shall be repeated. The mean of the two individual results shall be calculated and the titanium(IV) dioxide content given rounded to the nearest 0,1 % by mass.

NOTE Photometric, potentiometric and infra red methods have been shown to give good correlation with this test method.

Annex E
(normative)

Paint, thermoplastics and cold plastics – Test method for the determination of the glass bead content

E.1 Principle

From the ash residue (at 400°C) the glass bead content is determined by a separation process by density with chlorform/bromoform mixtures.

E.2 Apparatus

a) Analytical balance, with an accuracy of 0,001 g, with zero point correction;

b) electric muffle furnace, capable of being raised to a temperature of 400°C ± 20°C;

c) porcelain basins.

E.3 Reagent

a) chloroform (Trichloromethane);

b) bromoform (Tribromomethan);

c) hyrochloric acid or nitric acid;

d) washing up liquid.

E.4 Procedure

1) Weight about 20 g of the material (M_1) in to a cup and burn the solvent and binder.

2) Heat it in a muffle furnace at 400 °C until the carbon is removed (about 3 h).

3) Transfer the residue to a 400 ml beaker glass and dissolve the parts of the calciumcarbonate in 4 m nitric or hydrochloric acid (if no calciumcarbonate is present drop this step).

4) Transfer the whole solution into a 2000 ml (!) beaker, dilute with water to 1800 ml, add 1 or 2 drops of a washing up liquid (this will remove parts of the pigments that are sometimes hold on the beats) and stirr with a glass rod. Wait for about 2 min to allow the glass beads to settle. (the pigments are still dispersed).

5) Decant about 1500 ml of the suspension carefully to avoid the loss of beads and add 1500 ml water once more.

6) Repeat step 5) (now without an washing up liquid) until the supernatant liquid is clear.

7) Dry the residue.

8) Now it is possible to remove (a part) of the beads by using a watch glass.

9) Transfer the rest into a separatory funnel and add chloroform. Now add portions of bromoform till the separation of the anti skid aggregates from the glass beads appear. Depending on the density either the glass beads or the anti skid aggregates go up (at least it is possible to get an enrichment of the beads). Be careful: If an excess of bromoform is added all components goes up. It is recommended to control the separation under a microscope or magnifying glass to estimate the quality of the separation.

10) Dry the glass bead fraction and weigh (M_2).

E.5 Calculation and expression of results

Express the result by the arithmetic mean of two measurements, calculated as a percentage of the initial mass M_1 of the test portion, using the expression:

$$\frac{M_2}{M_1} \times 100 \tag{E.1}$$

NOTE If the two components cannot be separated the mass of both components can be expressed.

Annex F
(normative)

Paint and cold plastics – Test method for the determination and identification of solvents

F.1 Principle

F.1.1 Total Solvent Content

The volatile constituents of the material are evaporated at 105 °C using the method described in Annex A.

F.1.2 Solvent Identification

The solvents, others than water, are identified by gas chromatography after extraction with n-hexane.

F.2 Apparatus

F.2.1 Total Solvent Content

Refer to Annex A, A.2.

F.2.2 Solvent identification

a) ultrasonic bath;

b) analytical balance, with an accuracy of 0,001 g, with zero point correction;

c) gas chromatographic system:

    1) capillary gas chromatograph (autosampler recommended),

    2) injection with syringe: 1µl,

    3) split ratio 1:20,

    4) recommended column: 30m capillary column with a coating of 6 % cyanopropylphenyl / 94 % dimethylpolysiloxan crosslinked and chemically bonded,

    5) detector: flame ionisation;

d) glass vials 5ml and 1ml.

F.3 Reagents

n-Hexane (analytical grade).

Reference substances (depends on the product).

F.4 Procedure

F.4.1 Total Solvent Content

Refer to Annex A, A.4.

F.4.2 Solvent Identification

Weight one 1 g of the product in a glass vial, add 5 ml n-hexane and suspend it in an ultrasonic bath for about 30 min.

After sedimentation (pigment, fillers, glass beads,….) inject 1 µl of the supernatant blank hexane-extract.

The following temperature programme on the gas chromatographic system is recommended:

45 °C isotherm 10 min

increase of temperature 10 °C.min-1

200 °C isotherm 5 min

Run reference substances under the same conditions.

F.5 Test results

F.5.1 Total Solvent Content

The percentage solvent content of the material, H, shall be calculated according to the following equation:

The residual solids are weighed and the solvent content calculated.

$$H = 100 - S \tag{F.1}$$

If the results of the individual determinations differ by more than 0,5 % by mass the procedure shall be repeated. The mean of the two individual results shall be calculated and the solids content given rounded to the nearest 0,1 % by mass.

F.5.2 Solvent Identification

F.5.2.1 Qualitative identification

Compare the retention times of the solvents of the material with the retention times of the reference substances.

Report the result in a table, for example:

Reference substance	**Identified / not identified**
acetone	identified
butanone	identified
toluene	not identified
…….	…….

F.5.2.2 Quantitative identification

After calibration, compare the peak areas of the identified components.

Report the result in a table, for example:

Substance	Amount [Mass %]
acetone	10
butanone	90
…….	…….

Annex G
(normative)

Paint – Test method for the determination of viscosity (Krebs-Stormer method)

G.1 Principle

This method gives a value of apparent viscosity or consistency that refers the load in grams required to produce the specified rate of shear with a specified spindle to a traffic paint.

G.2 Apparatus

G.2.1 Viscosimeter, Stormer, with the paddle-type rotor as illustrated in Figure 1 and Figure 2.

Intercalibration of two viscosimeter may be carried out by determining the load in grams required to produce a shearing rate of 200 rpm with a standard oil having a viscosity of 1 to 1.5 Pa.s at 25 ºC.

NOTE There are actually automatic Stormer viscosimeters where it's no need of weights, or timer as there is a direct display of the Krebs viscosity.

G.2.2 Container

Can currently used for paints with a diameter of 85 mm and a volume of 500 ml, approximately.

G.2.3 Thermometer with an accuracy of 0,2 ºC having a range between 20 °C to 70 ºC.

G.2.4 Stopwatch with an accuracy of 0,2 s.

G.2.5 Set of weights so as to reproduce the values that appear on the table with 500, 200 (two), 100, 50, 25, 10 (two), 5, 2 (two), 1g.

G.2.6 Thermostatic bath able to withstand a temperature of 25 °C ± 0,2 ºC

G.3 Procedure

G.3.1 Preparation of the test

Mix the sample of paint thoroughly and strain it into the container. Fill to within 20 mm of the top of the container, and close hermetically. Bring the temperature of the sample to 25 °C ± 0,2 ºC and maintain it at that temperature during the test. Keep the sample for at least 1 h in the thermostatic bath to elapse between the time the paint is strained and the test is begun.

G.3.2 Determination of viscosity

Stir the paint carefully, to avoid entrapping air, and place the container on the platform of the viscosimeter and raise it until the paddle-type rotor is centrally immersed in the sample just to the mark on the shaft of the rotor. Before starting the test, turn the rotor through approximately 100 revolutions in 25 s to 35 s. Then, using different weights determine the time required for 100 revolutions of the rotor. Select weights that will give at least two readings within a range of 27 s to 33 s. Make these determinations from a running start, permitting the rotor to make at least 10 revolutions before starting the count of a test.

After each determination the driving weight holder must be lifted again with the winding drum fixing it with the brake control knob.

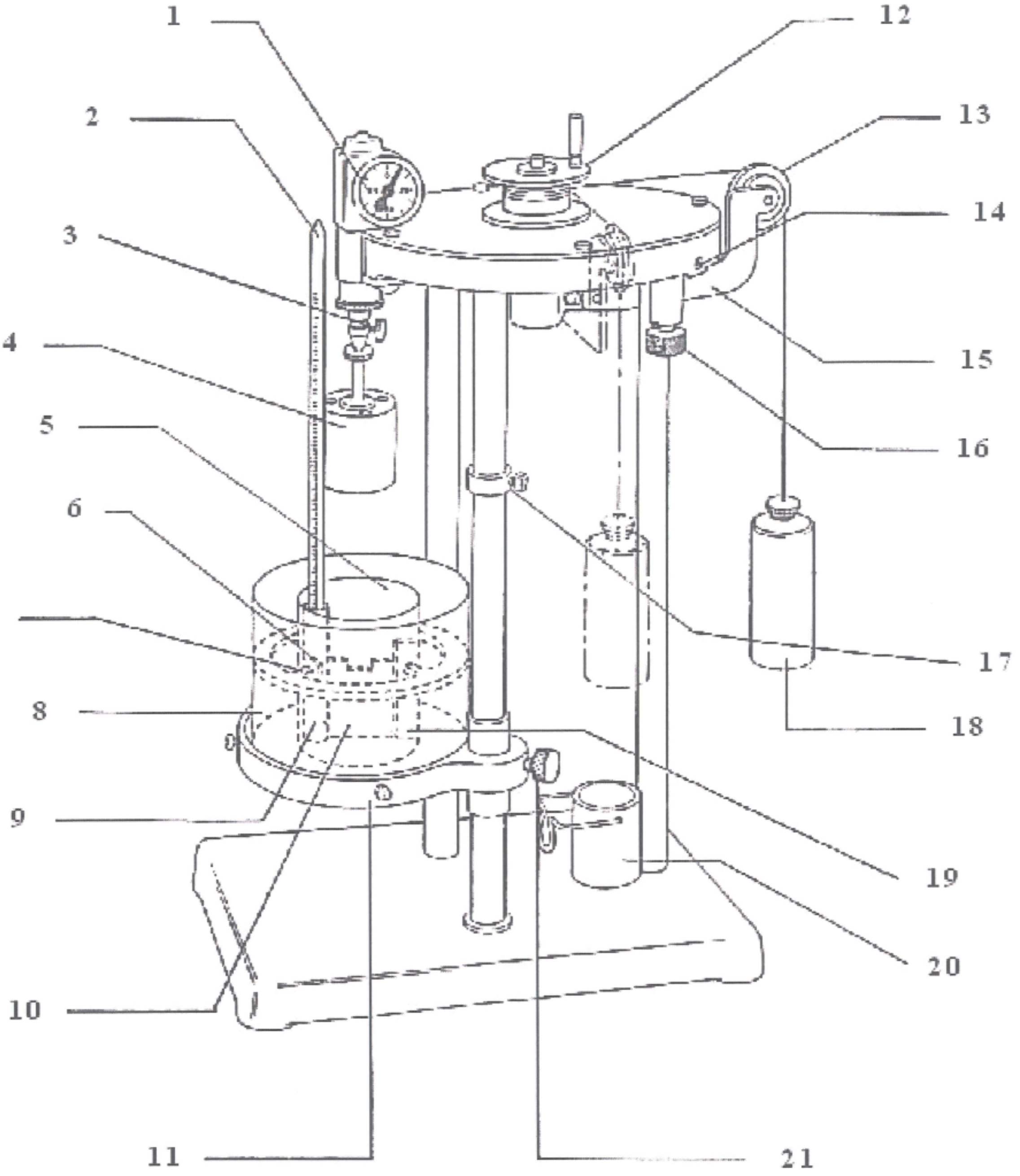
NOTE In apparatus with stroboscopic timer select the weights necessary to produce the 200 rpm pattern where the lines appear to be stationary.

G.4 Expression of results

The consistency of the sample can be expressed in the following ways:

G.4.1 Report the consistency as the weight in grams necessary to produce 100 in 30 s.

G.4.2 Krebs units, plotting in the following table the weight in grams applied and the time in seconds.



Key

1	revolution counter
2	thermometer
3	knurled set screw
4	cylindrical rotor
5	test cup
6	stop plate
7	projecting arm
8	water or oil bath
9	thermometer holder
10	central baffle
11	movable platform
12	winding drum
13	pulley
14	brake assembly set screw
15	pulley bracket
16	brake control knob
17	stop
18	driving weight (weight box)
19	side vane
20	weight box holder
21	platform set screw

Figure G.1 – General purpose outfit, with component parts identified

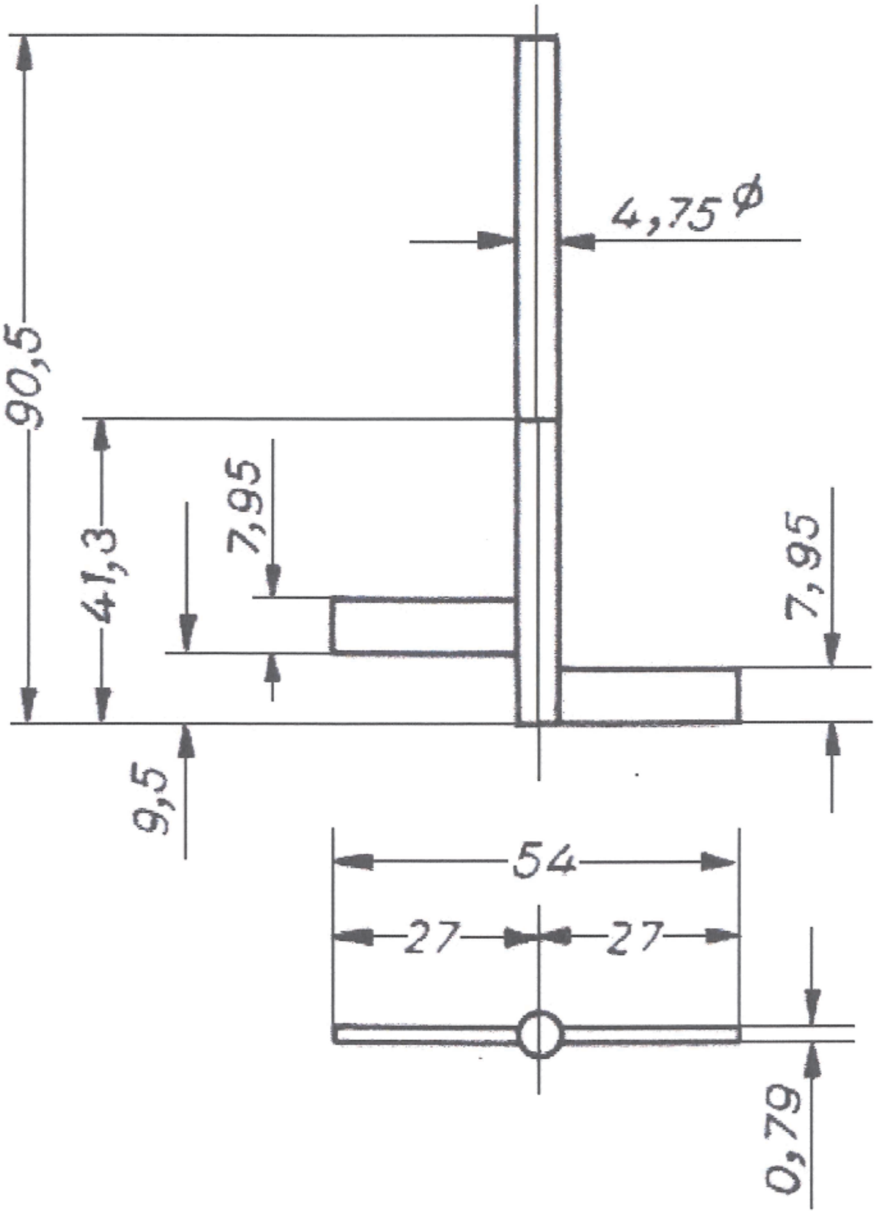


Figure G.2 – Paddle-type rotor in mm

Table G.1 - Units Krebs against the weight in grams applied and the time in seconds obtained

Weight (g) Time (s)	75	100	125	150	175	200	225	250	275	300	325	350	375	400	425	450	475	500
24	42	52	--	65	--	75	--	83	--	90	--	95	--	99	--	103	--	108
25	45	54	--	66	--	76	--	84	--	90	--	95	--	100	--	104	--	109
26	47	56	--	68	--	78	--	85	--	91	--	96	--	101	--	105	--	110
27	49	57	63	69	74	79	83	86	89	92	95	97	100	102	104	106	109	111
28	51	59	65	70	75	80	84	87	90	93	96	98	100	102	105	107	110	112
29	53	60	66	71	76	81	85	88	91	94	97	99	101	103	105	107	110	112
30	54	61	67	72	77	82	86	89	92	95	98	100	102	104	106	108	110	112
31	55	62	68	73	78	82	86	90	93	95	98	100	102	104	106	108	111	113
32	56	63	69	74	79	83	87	90	93	96	99	101	103	105	107	109	111	113
33	57	64	70	75	80	84	88	91	94	96	99	101	103	105	107	109	112	114
34	58	64	--	75	--	84	--	91	--	97	--	102	--	106	--	110	--	114
35	59	65	--	76	--	85	--	92	--	98	--	102	--	106	--	110	--	114
36	60	66	--	76	--	85	--	92	--	98	--	103	--	107	--	111	--	115
37	61	67	--	77	--	86	--	93	--	99	--	103	--	107	--	111	--	115
38	62	68	--	78	--	87	--	93	--	99	--	104	--	108	--	112	--	116
39	62	68	--	78	--	88	--	94	--	100	--	104	--	108	--	112	--	116
40	63	69	--	79	--	88	--	94	--	100	--	104	--	108	--	112	--	116

Weight (g) Time (s)	525	550	575	600	625	650	675	700	725	750	775	800	825	850	875	900	950	1000
24	--	111	--	115	--	118	--	122	--	125	--	128	--	130	--	132	--	136
25	--	112	--	116	--	119	--	122	--	125	--	129	--	131	--	133	--	137
26	--	113	--	117	--	120	--	123	--	126	--	130	--	132	--	134	--	138
27	113	114	116	118	120	121	123	124	126	127	129	130	131	132	133	134	136	138
28	114	115	117	118	120	121	123	124	126	127	129	130	131	132	133	134	137	139
29	114	115	117	119	121	122	124	125	127	128	130	131	132	133	134	135	137	139
30	114	116	118	120	121	122	124	125	127	128	130	131	133	134	135	136	138	140
31	115	116	118	120	122	123	125	126	128	129	131	132	133	134	135	136	138	140
32	115	116	118	120	122	123	125	126	128	129	131	132	133	134	135	136	138	140
33	116	117	119	121	122	123	125	126	129	129	131	132	134	135	136	137	139	141
34	--	118	--	122	--	124	--	127	--	130	--	132	--	135	--	137	--	141
35	--	118	--	122	--	124	--	127	--	130	--	133	--	135	--	137	--	142
36	--	118	--	122	--	125	--	128	--	130	--	133	--	135	--	137	--	142
37	--	119	--	123	--	125	--	128	--	131	--	133	--	136	--	138	--	142
38	--	119	--	123	--	126	--	129	--	131	--	134	--	136	--	138	--	142
39	--	120	--	124	--	126	--	129	--	131	--	134	--	136	--	138	--	143
40	--	120	--	124	--	127	--	130	--	132	--	134	--	136	--	138	--	143

Annex H
(normative)

Paint, thermoplastics and cold plastics – Test method for the determination of the ash content

H.1 Scope and field application

This document specifies a method for determining the residue obtained by calcinating materials under fully specified conditions after evaporation of the volatile matter.

The determination of the ash content constitutes a standard and comparative means for estimating the content of minerals in the materials.

H.2 References

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling (ISO 15528:2000)*

H.3 Sampling

Take a sample of the product to be tested in accordance with EN ISO 15528.

H.4 Apparatus

a) flat bottom evaporating basin, of capacity approximately 25 ml;

b) flat bottom evaporating basin, of capacity approximately 100 ml;

c) laboratory scales, accurate to 0,1 mg;

d) oven with natural ventilation;

e) sand bath;

f) electric muffle furnace, capable of being raised to a temperature of 900 °C ± 20 °C;

g) desicator.

H.5 Procedure

Carry out all the weighings to within 0,1 mg.

Carry out the operation on three separate test portions of approximately 2 g.

NOTE 1 The test portion can be increased to 5 g or 10 g in the case of paints with an ash content of less than 1 %. In this case, use a 100 ml evaporating basin.

Calcinate the empty evaporating basin in the electric muffle furnace for half an hour at 900 °C ± 20 °C; remove it and place it in the desiccator to cool.

Weigh the evaporating basin, let M_o be its mass.

Place the test portion in the evaporating basin and weigh; let M_1 be the mass.

Distribute the test portion as uniformly as possible in the evaporating basin and evaporate the volatile products (solvents) by placing the evaporating basin in a sand bath at 40 °C, beneath a ventilated hood, for 24 h.

NOTE 2 For varnishes with an ash content of less than 1 %, use a 100 ml evaporating basin.

Then place the evaporating basin in the oven and dry the test portion at 105 °C ± 2 °C for 3 h in order to eliminate the solvent

Then calcinate in the electric muffle furnace for 2 h at 900 °C ± 20 °C.

Remove the evaporating basin and allow it to cool in the desiccator and weigh it.

Let M_2 be the mass of the evaporating basin and the calcinated product.

NOTE 3 This calcination will be achieved gradually, for example, the evaporating basin can be heated prior to its insertion in the furnace with a laboratory burner that is removed immediately after igniting the product so as to burn most of the binder without splashing.

H.6 Calculation and expression of results

Express the result by the arithmetic mean of three measurements, calculated as a percentage of the initial mass $M_1 - M_0$ of the test portion, using the expression:

$$\frac{M_2 - M_0}{M_1 - M_0} \times 100 \qquad \text{(H.1)}$$

Each of the individual measurements shall not be less than 2 % of the mean value, except in the case of varnishes with an ash content less than 1 %, for which a difference of 5 % can be permitted.

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

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