#### BS EN 16245-2:2013



## **BSI Standards Publication**

# Fibre-reinforced plastic composites — Declaration of raw material characteristics

Part 2: Specific requirements for resin, curing systems, additives and modifiers



BS EN 16245-2:2013 BRITISH STANDARD

#### **National foreword**

This British Standard is the UK implementation of EN 16245-2:2013.

The UK national committee are of the opinion that in all parts of EN 16245 the use of the terms "guaranteed minimum value" and "guaranteed maximum value" is not appropriate within a voluntary based standard. Furthermore, the absolute "minimum value" and "maximum value" cannot be determined practicably, so that "statistical minimum value" (defined in EN 16245-1) and "statistical maximum value" (calculated in a similar manner using maximal values) are preferred.

The UK participation in its preparation was entrusted to Technical Committee PRI/42, Fibre reinforced thermosetting plastics and prepregs.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Compliance with a British Standard cannot confer immunity from legal obligations.

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### EUROPEAN STANDARD

#### EN 16245-2

# NORME EUROPÉENNE

**EUROPÄISCHE NORM** 

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#### **English Version**

# Fibre-reinforced plastic composites - Declaration of raw material characteristics - Part 2: Specific requirements for resin, curing systems, additives and modifiers

Composites plastiques renforcés de fibres - Déclaration des caractéristiques des matières premières - Partie 2: Exigences particulières pour les résines, les systèmes de polymérisation, les additifs et les modificateurs

Faserverstärkte Verbundwerkstoffe - Angabe von Werkstoffeigenschaften - Teil 2: Spezifische Anforderungen an Harz, Aushärtungssysteme, Zusatzstoffe und Modifizierer

This European Standard was approved by CEN on 21 March 2013.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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#### **Foreword**

This document (EN 16245-2:2013) has been prepared by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by NBN.

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 November 2013, and conflicting national standards shall be withdrawn at the latest by November 2013.

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.

EN 16245 consists of the following parts, under the general title *Fibre-reinforced plastic composites* — *Declaration of raw material characteristics*:

- Part 1: General requirements
- Part 2: Specific requirements for resin, curing systems, additives and modifiers (the present document)
- Part 3: Specific requirements for fibre
- Part 4: Specific requirements for fabrics
- Part 5: Specific requirements for core materials

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

#### 1 Scope

This European Standard specifies the minimum information to be declared for resins, curing systems, additives and modifiers to be used for the manufacturing of composites products.

These specific declaration requirements are in addition to the general requirements given in EN 16245-1.

This document includes requirements for the Certificate of Analysis (CoA). The purpose of the CoA is to verify that material properties and quality conforms to the declared values.

#### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 16245-1:2013, Fibre-reinforced plastic composites — *Declaration of raw material characteristics* — *Part 1:* General requirements

EN 59, Glass reinforced plastics — Measurement of hardness by means of a Barcol impressor

EN ISO 62, Plastics — Determination of water absorption (ISO 62)

EN ISO 75-2:2013, Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite (ISO/FDIS 75-2:2012)

EN ISO 178:2010, Plastics — Determination of flexural properties (ISO 178:2010)

EN ISO 291, Plastics — Standard atmospheres for conditioning and testing (ISO 291)

EN ISO 527 (all parts), Plastics — Determination of tensile properties (ISO 527)

EN ISO 584, Plastics — Unsaturated polyester resins — Determination of reactivity at 80 °C (conventional method) (ISO 584)

EN ISO 787-5, General methods of test for pigments and extenders — Part 5: Determination of oil absorption value (ISO 787-5)

EN ISO 1183 (all parts), Plastics — Methods for determining the density of non-cellular plastics (ISO 1183)

EN ISO 2114, Plastics (polyester resins) and paints and varnishes (binders) — Determination of partial acid value and total acid value (ISO 2114)

EN ISO 2555, Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method (ISO 2555)

EN ISO 2592, Determination of flash and fire points — Cleveland open cup method (ISO 2592)

EN ISO 2719, Determination of flash point — Pensky-Martens closed cup method (ISO 2719)

EN ISO 2811 (all parts), Paints and varnishes — Determination of density (ISO 2811)

EN ISO 2884 (all parts), Paints and varnishes — Determination of viscosity using rotary viscometers (ISO 2884)

EN ISO 3251, Paints, varnishes and plastics — Determination of non-volatile-matter content (ISO 3251)

EN ISO 3521, Plastics — Unsaturated polyester and epoxy resins — Determination of overall volume shrinkage (ISO 3521)

EN ISO 3838, Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838)

EN ISO 4629, Binders for paints and varnishes — Determination of hydroxyl value — Titrimetric method (ISO 4629)

EN ISO 4630 (all parts), Clear liquids — Estimation of colour by the Gardner colour scale (ISO 4630)

EN ISO 15512, Plastics — Determination of water content (ISO 15512)

ISO 760, Determination of water — Karl Fischer method (General method)

ISO 3105, Glass capillary kinematic viscometers — Specifications and operating instructions

ISO 5661, Petroleum Products — Hydrocarbon liquids — Determination of refractive index

ISO 14848, Plastics — Unsaturated-polyester resins — Determination of reactivity at 130 degrees C

ASTM D1135-86, Standard Test Methods for Chemical Analysis of Blue Pigments

ASTM D2196-10, Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer

ASTM D3278-96, Standard Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus

ASTM D4835-08, Standard Specification for Propylene Glycol Monomethyl Ether Acetate

ASTM D6420-99, Standard Test Method for Determination of Gaseous Organic Compounds by Direct Interface Gas Chromatography-Mass Spectrometry

ASTM E298-08, Standard Test Methods for Assay of Organic Peroxides

ASTM E1473-09, Standard Test Methods for Chemical Analysis of Nickel, Cobalt, and High-Temperature Alloys

UOP523, Glycol and Carom Solvent Distribution in Mixtures by GC

DIN 16945:1989, Testing of resins, hardeners and accelerators, and catalyzed resins

#### EN 16245-2:2013 (E)

#### 3 Terms and definitions

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

#### 3.1

#### accelerator

promoter

substance used in small proportion to increase the reaction rate of a chemical system (reactants, plus other additives)

[SOURCE: EN ISO 472:2013, 2.1]

#### 3.2

#### additive

modifier

substance added to a resin to improve or modify one or more properties (i.e. UV stability, surface tensions, air release etc.)

#### 3.3

#### **Barcol hardness**

hardness value obtained by measuring the resistance to penetration of a sharp, spring-loaded steel point with an instrument called the Barcol Impressor

Note 1 to entry: The value can be used as an indicative measure of the degree of cure of a thermosetting resin.

#### 3.4

#### cure

process of chemically converting an unsaturated polymeric composition into a more stable three-dimensional network by cross-linking

#### 3.5

#### curing system

initiator, promoter, accelerator and inhibitor, all related to the chemical reaction of the resin

#### 3.6

#### flash point

lowest temperature at which a combustible liquid will give off a flammable vapour that will burn momentarily by ignition near the surface of the liquid or within the vessel used

#### 3.7

#### gel time

interval of time, in connection with the use of synthetic thermosetting resins, extending from the introduction of an initiator and a catalyst into a liquid resin system until the interval of gel formation/solidification

#### 3.8

#### inhibitor

substance used in small proportion to suppress a chemical reaction

[SOURCE: EN ISO 472:2013, 2.488]

#### 3.9

#### initiator

substance, used in small proportion, that starts a chemical reaction, for example, by providing free radicals

[SOURCE: EN ISO 472:2013, 2.490]

#### 3.10

#### monomer

relatively simple substance, usually containing carbon and of low molecular mass, which can react to form a polymer by combination with itself or with other similar molecules or substances

Note 1 to entry: To be distinguished from the term reactive monomer, which within the context of unsaturated polyester/vinyl ester is defined as a substance with unsaturated groups which can undergo cross-linking reactions with the unsaturated polymers through the curing reaction.

#### 3.11

#### resin

<thermosetting materials> the uncured liquid material that is used as matrices to bind together the reinforcement materials in a fibre-reinforced composite product

#### 3.12

#### stvrene

colourless liquid produced from the catalytic dehydrogenation of ethylbenzene which is easily polymerized by exposure to light, heat or a peroxide initiator

Note 1 to entry: The main reactive monomer used in unsaturated polyester/vinyl ester materials.

#### 3.13

#### unsaturated polyester

thermosetting resins of polyester molecules, dissolved in a reactive monomer (e.g. styrene) capable of copolymerisation with the polyester molecules

Note 1 to entry: The polyester molecules are condensation products of polyols (e.g. ethylene glycol, dipropylene glycol, glycerol) and saturated/unsaturated difunctional carboxylic acids/anhydrides (e.g. terephthalic acid, phthalic anhydride, fumaric acid, maleic anhydride) and/or cyclic unsaturated hydrocarbons (e.g. dicyclopentadiene).

Note 2 to entry: At least one of the acids/anhydrids is unsaturated (usually maleic anhydride/fumaric acid is used).

#### 3.14

#### vinyl ester resins

thermosetting resins of vinyl ester molecules, dissolved in a reactive monomer (e.g. styrene) capable of copolymerisation with the vinyl ester molecules

Note 1 to entry: The vinyl ester is a product of the esterification reaction of any epoxy resin (e.g. Novolac, Bisphenol A epoxy) with an unsaturated monocarboxylic acid (e.g. methacrylic acid).

#### 3.15

#### viscosity

measure of the resistance to flow due to internal friction when one layer of fluid is caused to move in relationship to another layer

Note 1 to entry: In the context of thermosetting resins for the FRP (Fibre-Reinforced Plastic) industry, viscosity is one of the important parameters that express the ability of a resin to flow in reinforcements and release entrapped air often formed during the application a production process.

#### 3.16

#### water absorption

moisture absorption

amount of water absorbed by a material under specified test conditions

Note 1 to entry: The conditions may be immersion in water or exposure to a humid atmosphere, in the latter case the process is also referred to as water vapour absorption.

[SOURCE: EN ISO 472:2013, 2.1246]

#### 4 Content of a declaration

A declaration for the resin, curing system, additive and modifier shall consist of information according to EN 16245-1:2013, Clause 5 and Clause 5 of this standard.

#### 5 Specific declaration requirements

#### 5.1 General

The specific requirements for resins, curing systems, additives and modifiers are given below.

All declaration requirements, i.e. the general information according to EN 16245-1 and the specific declaration requirements according to this part (i.e. EN 16245-2), and application dependant requirements as agreed between manufacturer/supplier and customer, shall be declared by the supplier as information to the customer. The following also apply:

- if the property given has reference to a test method standard or test method, this test method standard or test method shall be used;
- the values given shall be in accordance with the test method standard given;
- if the test environment is not clearly stated in the specific test method standard, the standard atmosphere conditioning and testing shall be carried out in accordance with EN ISO 291;
- the manufacturer shall be responsible for the performance and results of all tests required for the declaration;
- the declaration is for the delivered material and not for its constituents.

#### 5.2 Declaration for resin

#### 5.2.1 Declaration for unsaturated polyester and vinyl ester resin

#### 5.2.1.1 Properties of liquid resin as delivered to customer

The specific declaration requirements for liquid resin are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 1 and Table 2.

The following specific declaration requirements a) to f) apply for all liquid resins independent of application:

- a) identification (name/number/code used by the manufacturer for identification purposes);
- b) density [kg/m<sup>3</sup>];
- c) viscosity [mPa·s] (cP):
  - 1) The supplier will define and declare which standard is to be used.
- d) acid number [mg KOH/g];
- e) non-volatile content [wt.%];

NOTE Defines the amount of solid content from which styrene content in the material can be calculated.

f) flash point [°C].

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The following additional declaration requirements g) to I) apply for liquid resin for specific application:

- g) gel time [min] [s]: defines the gel time of the resin at a given temperature and for a given curing system. To be given as the arithmetic mean value of at least two tests that are both within 10 % of their mean value, including tolerances;
- h) reactivity [min or s] and [°C];
- i) maximum recommended laminating thickness [mm]:
  - 1) Amount and type of initiator shall be given in connection to recommended laminate thickness applied wet in wet. Wet in wet is defined by impregnation of all layers through the thickness prior to curing.
- j) colour:
  - 1) The supplier shall decide and declare the procedure to be used. Accepted standards are: ISO 2211 and EN ISO 4630. Acceptable standards for spectrophotometric methods are: ASTM D156-12, ASTM D263-05, ASTM D1045-08, ASTM D1209-05, ASTM D1500-12, EN 1557 and DIN 6162.
- k) water content [wt.%]:
  - 1) The supplier shall decide and declare the procedure to be used together with Karl Fischer Titration.
- I) chemical thickening (i.e. valid for Sheet Moulding Compound resin (SMC resin)).

The Certificate of Analysis (CoA) verifies that the properties selected for the CoA for the delivered liquid resin material comply with the declared values, in accordance with the methods specified in Table 1 and Table 2. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 1 — Material declaration of liquid resin properties relevant for CoA (independent of application)

Ref. no 5.2.1.1	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
b)	Density	Mean value	Minimum and maximum value	kg/m <sup>3</sup>	EN ISO 2811 (all parts)
c)	Viscosity, high shear Viscosity, low shear	Mean value	Minimum and maximum value	mPa·s cP	EN ISO 2884 (all parts) EN ISO 2555 ASTM D2196-10
d)	Acid number	Mean value	Maximum value	mg KOH/g	EN ISO 2114
e)	Non-volatile content	Mean value	Minimum and maximum value	%	EN ISO 3251
f)	Flash point	Mean value	Minimum and maximum value	°C	ASTM D3278-96 EN ISO 2592

Table 2 — Material declaration of liquid resin properties relevant for CoA (application specific)

Ref. no 5.2.1.1	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
g)	Gel time	Mean value	Minimum and maximum value	min or s	To be specified by the supplier
h)	Reactivity ambient temp.  Reactivity elevated temp.  Sheet Moulding Compound resin (SMC resin)	Mean value	Minimum and maximum value	min or s and °C	DIN 16945:1989, 6.2.2.2 EN ISO 584 ISO 14848
j)	Colour	Mean value	Maximum value		
k)	Water content	Mean value	Maximum value	wt.%	

#### 5.2.1.2 Properties of cured non-reinforced resin (clear cast)

Clear cast test sample shall be produced according to Annex A.

The specific declaration requirements for cured non-reinforced resin are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 3.

The following specific declaration requirements a) to m) apply for all cured non-reinforced resins independent of application:

a) heat distortion temperature (HDT) [°C];

NOTE Defines at which temperature the cured resin deflects a specific amount with a specified load applied.

- b) tensile strength [MPa];
- c) tensile modulus [MPa] or [GPa];
- d) tensile strain at break [%];
- e) flexural strength [MPa];
- f) flexural modulus [MPa] or [GPa];
- g) Barcol hardness;
- h) water absorption [wt.%] or [mg/test specimen];
- i) density [kg/m<sup>3</sup>];
- j) volumetric shrinkage [%];

- k) curing system name, number and code (i.e. the exact curing system is to be given);
- mixing ratio [wt.% and/or vol.%];
- m) post cure schedule temperature as function of time [°C and h].

The Certificate of Analysis (CoA) verifies that the clear cast properties selected for the CoA for the delivered resin comply with the declared values, in accordance with the methods specified in Table 3. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 3 — Material declaration of cured non-reinforced resin (clear cast) properties and conditions relevant for CoA (independent of application)

Ref. no 5.2.1.2	Property/condition	Nominal value	Minimum and/or maximum value	Unit	Test method
a)	Heat distortion temp. (HDT)	Mean value	Minimum and maximum value	°C	ISO 75-2:2013 Method A
b)	Tensile strength	Mean value	Minimum and maximum value	MPa	EN ISO 527 (all parts)
c)	Tensile modulus	Mean value	Minimum and maximum value	MPa GPa	EN ISO 527 (all parts)
d)	Tensile strain at break	Mean value	Minimum and maximum value	%	EN ISO 527 (all parts)
e)	Flexural strength	Mean value	Minimum and maximum value	MPa	ISO 178:2010 Method A
f)	Flexural modulus	Mean value	Minimum and maximum value	MPa GPa	ISO 178:2010 Method A
g)	Barcol hardness	Mean value	Minimum and maximum value	Units	EN 59
h)	Water absorption	Mean value	Minimum and maximum value	wt.% or mg/test specimen	EN ISO 62
i)	Density	Mean value	Minimum and maximum value	kg/m <sup>3</sup>	EN ISO 1183 (all parts)
j)	Volumetric shrinkage	Mean value	Minimum and maximum value	%	EN ISO 3521
l)	Mixing ratio	Mean value	Minimum and maximum value	wt.% and/or vol.%	
m)	Post cure schedule	Mean value	Minimum and maximum value	°C and h	

#### 5.3 Declaration for curing systems

#### 5.3.1 Properties of resin initiator

The specific declaration requirements for resin initiators are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 4 and Table 5.

The following specific declaration requirements a) to g) apply for all resin initiators independent of application:

a) identification (name/number/code used by the manufacturer for identification purposes);

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- b) type of initiator;
- c) total active oxygen content [wt.% and/or vol.%];
- d) flash point [°C];
- e) viscosity (nominal) [mPa·s] [cP];
- f) density [kg/m<sup>3</sup>];
- g) recommended mixing ratio [wt.% and/or vol.%].

The following additional declaration requirements h) to l) apply for resin initiator for specific application:

- h) hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) content [wt.% and/or vol.%];
- i) water content [wt.%];
- j) salt content [ppm];
- k) glycol content [wt.%];
- I) methyl ethyl ketone (MEK) content [wt.%].

The Certificate of Analysis (CoA) verifies that the properties selected for the CoA for the delivered initiator material comply with the declared values, in accordance with the methods specified in Table 4 and Table 5. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 4 — Material declaration of resin initiator properties relevant for CoA (independent of application)

Ref. no 5.3.1	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
c)	Total active oxygen content	Mean value	Minimum and maximum value	wt.% vol.%	ASTM E298-08
d)	Flash point	Mean value	Minimum and maximum value	°C	ASTM D3278-96
e)	Viscosity	Mean value	Minimum and maximum value	mPa·s cP	ISO 3105
f)	Density	Mean value	Minimum and maximums value	kg/m <sup>3</sup> g/cm <sup>3</sup>	EN ISO 3838

Table 5 — Material declaration of resin initiator properties relevant for CoA (application specific)

Ref. no 5.3.1	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
h)	Hydrogen peroxide (H <sub>2</sub> O <sub>2</sub> ) content	Mean value	Minimum and maximum value	wt.% vol.%	Modified ASTM E298-08
i)	Water content	Mean value	Minimum and maximum value	wt.%	EN ISO 15512 by Karl Fischer Titration
j)	Salt content	Mean value	Minimum and maximum value	ppm	ASTM D1135 (conductivity method)
k)	Glycol content	Mean value	Minimum and maximum value	wt.%	ASTM D4835-08 or UOP523
l)	Methyl ethyl ketone (MEK) content	Mean value	Minimum and maximum value	wt.%	ASTM D6420-99

#### 5.3.2 Properties of resin accelerator/promoter

The specific declaration requirements for resin accelerators/promoters are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 6.

The following specific declaration requirements a) to i) apply for all resin accelerators/promoters independent of application:

- a) identification (name/number/code used by the manufacturer for identification purposes);
- b) type of accelerator/promoter;
- c) metal content [wt.%] (valid for accelerators only);
- d) active ingredient content [wt.%];
- e) solvent type;
- f) flash point [°C];
- g) viscosity [mPa·s] [cP];
- h) density [kg/m<sup>3</sup>];
- i) recommended mixing ratio [wt.% and/or vol.%].

The Certificate of Analysis (CoA) verifies that the properties selected for the CoA for the delivered resin accelerators/promoters comply with the declared values, in accordance with the methods specified in Table 6. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 6 — Material declaration of resin accelerators/promoters properties relevant for CoA (independent of application)

Ref. no 5.3.2	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
c)	For accelerators:  Metal content (Cobalt, etc.)	Mean value	Minimum and maximum value	wt.%	ASTM E1473-09
d)	For promoters: Active ingredient content/Aniline content	Mean value	Minimum and maximum value	wt.%	ASTM E298-08
f)	Flash point	Mean value	Minimum and maximum value	°C	ASTM D3278-96
g)	Viscosity	Mean value	Minimum and maximum value	mPa·s (cP)	ISO 3105
h)	Density	Mean value	Minimum and maximum value	kg/m <sup>3</sup>	EN ISO 3838

#### 5.3.3 Properties of resin inhibitor

The specific declaration requirements for resin inhibitors are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 7.

The following specific requirements a) to f) apply for all resin inhibitor independent of application:

- a) identification (name/number/code used by the manufacturer for identification purposes);
- b) type of inhibitor;
- c) active ingredient content [wt.%];
- d) solvent type;
- e) flash point [°C];
- f) recommended mixing ration [wt.% and/or vol.%].

The Certificate of Analysis (CoA) verifies that the properties selected for the CoA for the delivered resin inhibitors comply with the declared values, in accordance with the methods specified in Table 7. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 7 — Material declaration of resin inhibitor properties relevant for CoA (independent of application)

	f. no 3.3	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
(	c)	Active ingredient content	Mean value	Minimum and maximum value	wt.%	To be specified by supplier
6	e)	Flash point	Mean value	Minimum and maximum value	°C	ASTM D3278-96

#### 5.4 Declaration for resin additive/modifier

The specific declaration requirements for resin additives/modifiers are listed below. The declaration including tolerances shall be given in accordance with the test method standards stated in Table 8 and Table 9.

The following specific requirements a) to g) apply for all resin additive/modifier independent of application:

a) identification (name/number/code used by the manufacturer for identification purposes);

b)	type of additive/modifier;
c)	total active ingredient content [wt.%];
d)	flash point [°C];
e)	density [kg/m³];
f)	refractive index;
g)	recommended mixing ratio [wt.% and/or vol.%].
	e following additional declaration requirements h) to q) apply for resin additive/modifier for specific olication:
h)	colour;
i)	acid value;
j)	amine value;
k)	OH-number;
l)	non-volatile content [wt.%];
m)	solvent type;
n)	water content [wt.%];
o)	viscosity [mPa·s];
p)	mean particle size, particle size distribution [µm] and particle shape (applicable for particulate fillers only);
q)	particle oil absorption value.

The Certificate of Analysis (CoA) verifies that the properties selected for the CoA for the delivered resin additives and modifiers comply with the declared values, in accordance with the methods specified in Table 8

and Table 9. The CoA shall be given in accordance with EN 16245-1:2013, Clause 6.

Table 8 — Material declaration of resin additive/modifier properties relevant for CoA (independent of application)

Ref. no 5.4	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
c)	Total active ingredient content	Mean value	Minimum and maximum value	wt.%	
d)	Flash point	Mean value	Minimum and maximum value	°C	EN ISO 2719
e)	Density	Mean value	Minimum and maximum value	kg/m <sup>3</sup>	EN ISO 2811 (all parts)
f)	Refracting index	Mean value	Minimum and maximum value		ISO 5661

Table 9 — Material declaration of resin additive/modifier properties relevant for CoA (application specific)

Ref. no 5.4	Property	Nominal value	Minimum and/or maximum value	Unit	Test method
h)	Colour	Mean value	Minimum and maximum value		EN ISO 4630
i)	Acid value	Mean value	Minimum and maximum value		EN ISO 2114
j)	Amine value	Mean value	Minimum and maximum value		DIN 16945
k)	OH-number	Mean value	Minimum and maximum value		EN ISO 4629
l)	Non-volatile content	Mean value	Minimum and maximum value	wt.%	EN ISO 3251
n)	Water content	Mean value	Minimum and maximum value	wt.%	EN ISO 15512 or Modified Karl Fischer Titration/ ISO 760
0)	Viscosity	Mean value	Minimum and maximum value	mPa·s (cP)	EN ISO 2555
p)	Mean particle size / Particle size distribution	Mean value	Minimum and maximum value	μm	
q)	Particle oil absorption value	Mean value	Minimum and maximum value		EN ISO 787-5

#### Annex A

(normative)

# Non-reinforced/clear cast mouldings of unsaturated polyesters and vinyl ester

#### A.1 Principle

Non-reinforced/clear cast mouldings of unsaturated polyesters and vinyl esters are made to test mechanical and physical properties.

Air voids, curing stresses and other possible defects in the moulding will influence the final results negatively and give results which are not correct. Moulding thixotropic resins and/or Low Styrene Emission (LSE) resins as delivered may also cause reduced moulding quality, as the thixotropic agent and paraffin wax can cause "micro-air bubbles" inclusions which could further influence the tested properties. Therefore, it is always recommended to use only non-thixotropic and non-waxed resins for moulding to test mechanical properties.

The method described is intended to ensure that a non-reinforced moulding of unsaturated polyester resin and/or vinyl ester resin is produced with as few defaults as possible. The method also contains guidance for specimen selection for reporting average values.

#### A.2 Apparatus

The apparatuses used for non-reinforced/clear cast include:

- two clean glass plates, 450 mm x 300 mm (see A.3.2);
- flexible rubber gaskets, E-profile 5 mm thickness x 10 mm width (see A.3.2);
- aluminium spacer sheets with thickness of 4 mm.

#### A.3 Procedure

#### A.3.1 Curing system

Select a cobalt-MEKP (methyl ethyl ketone peroxide) system inhibited with 4-tert-butylcathechol (TBC) to a gel time of 30 min to 50 min at 23 °C. Avoid using curing systems that can give curing stresses, for example too reactive curing systems.

After having carefully stirred in separately the accelerator and the peroxide, let the mix deaerate/degas for approximately 20 min.

#### A.3.2 Mould preparation filling

Thoroughly clean two glass plates with acetone and soap water and clean thoroughly with clean water prior to waxing.

Prepare the glass plates (450 mm x 300 mm) with an appropriate release wax giving good release properties.

Let the waxed glass plates "dry/cure" for a minimum 2 h after polishing.

#### EN 16245-2:2013 (E)

Prepare a gasket, normally a rubber gasket E-profile with thickness 5 mm and width 10 mm. Place the prepared gasket around the edges of one of the prepared glass plates, and put the other glass plate on top. Allow an opening in the gasket at the short edge for filling purposes.

Insert 4 mm aluminium spacers to ensure even and correct thickness.

Tighten the glass mould with an appropriate clamping system to a mould opening of (4 ± 0,1) mm.

The prepared mould is placed in a certain angle (i.e. for example 60 ° to 80 °).

Before the resin is moulded, it might be necessary to dilute with styrene. Generally, a viscosity of approximately 200 mPa s at 23 °C for orthophthalic resins and 300 mPa s at 23 °C for isophthalic and vinyl ester resins are used.

Before filling the mould, the resin shall be filtered, preferably through a 100 microns filter.

The resin is gently poured into mould, so that no air bubbles are created.

#### A.3.3 Curing

After reaching the exothermic peak, the clamps are slightly loosened.

The resin is cured 18 h to 24 h at room temperature (23 °C) in the mould.

#### A.3.4 Demould

The clear casting is placed horizontally between two aluminium plates or glass plates, protecting the casting on both sides with BoPET film. For flexible resin mouldings, a soft paper tissue can be used instead of the BoPET film to reduce possible surface defects. The top plate shall act as a weight.

#### A.3.5 Post curing

Place the demoulded non-reinforced/clear cast, with supporting plates, in a heating oven. It is important that the oven is "cold" when starting the post curing process. The cool down period must be slow and the moulding shall not be removed from the oven at temperatures above 40 °C.

If no other post curing cycle is specified, 24 h at 60 °C plus 3 h at 10 °C above the anticipated heat deflection temperature (HDT) of the resin is used.

If the highest post curing temperature exceeds 90  $^{\circ}$ C to 100  $^{\circ}$ C, one shall post cure step-wise to avoid stresses in the casting. Use intervals of 25  $^{\circ}$ C to 30  $^{\circ}$ C. See the example below:

#### **FXAMPIF**

- 90 °C in 1/2 h;
- increase to 120 °C in 1/2 h;
- increase to 150 °C in 3 h;
- cooling.

#### A.4 Test specimens

The following is prepared to give guidance to the selection of specimens to be used for testing/reporting.

When the moulding is demoulded and post cured as described in A.3.4 and A.3.5 above, the test specimens are cut according to the test method standards to be used.

The cutting edges of the specimens shall be polished.

A visual judgement of the test specimens shall be carried out in order to exclude those that contain airentrapments or voids and those that have been damaged or have edge failures from the cutting or machining of the specimens.

Test specimens that differ considerably from the rest of the specimens concerning width and thickness are excluded.

After testing the specimens, the individual results shall be evaluated. The test specimens that are giving large deviations compared to the others shall be examined. If these test specimens contain air voids or other visual defects, they shall be excluded and shall not be used in the final calculation/report. If other individual specimens show large deviation in results, they shall be evaluated if they shall be part of the final calculation/report.

#### A.5 Quality assurance and control

Typically, six, nine or twelve tensile test specimens and six or nine flexural specimens are made.

If the number of specimens that are excluded according to the above procedure are larger than one-third of the total number of specimens, e.g. more than three specimens of a total selection of nine specimens, a new non-reinforced/clear cast moulding shall be made.

The absolute minimum of repeats to be used for calculation/reporting is five specimens.

Any deviation from the above description/method shall be documented and the equivalence between the deviating method and the described method shall be demonstrated by comparative testing.

#### **Bibliography**

- [1] EN ISO 472:2013, Plastics Vocabulary (ISO 472:2013)
- [2] ISO 2211, Liquid chemical products Measurement of colour in Hazen units (platinum-cobalt scale)
- [3] ISO 13320, Particle size analysis Laser diffraction methods
- [4] ASTM D156-12, Standard Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)
- [5] ASTM D263-05, Standard Specification for Chrome Oxide Green Pigment
- [6] ASTM D1045-08, Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics
- [7] ASTM D1209-05, Standard Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
- [8] ASTM D1500-12, Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
- [9] EN 1557, Surface active agents Colorimetric characterization of optically clear coloured liquids (products) as X, Y, Z tristimulus values in transmission
- [10] DIN 6162, Determination of iodine colour number



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