

INTERNATIONAL STANDARD



**Resin based reactive compounds used for electrical insulation –
Part 2: Methods of test**

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**Resin based reactive compounds used for electrical insulation –
Part 2: Methods of test**

INTERNATIONAL
ELECTROTECHNICAL
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CONTENTS

FOREWORD.....	5
INTRODUCTION.....	2
1 Scope.....	8
2 Normative references	8
3 Terms and definitions	11
4 General notes on methods of test	11
4.1 Preparation and conditioning.....	11
4.2 Sequence of tests	12
4.3 Test report	12
5 Methods of test for reactive compounds and their components	12
5.1 Flash point.....	12
5.2 Density	12
5.3 Viscosity	12
5.4 Viscosity after storing at elevated temperature.....	12
5.5 Content of volatile organic components.....	13
5.6 Isothermal increase of viscosity (processing time)	13
5.7 Shelf life	13
5.8 Colour.....	14
5.9 Softening temperature	14
5.10 Ash content.....	14
5.11 Filler content.....	14
5.12 Chlorine content.....	14
5.12.1 Total chlorine content of unsaturated polyesters and epoxide resins.....	14
5.12.2 Inorganic chlorine content of epoxide resins and glycidyl esters	14
5.12.3 Easily saponifiable chlorine content of epoxide resins and related materials	14
5.13 Tendency of crystallisation.....	14
5.14 Epoxide equivalent of epoxide resins	14
5.15 Content of isocyanate	14
5.16 Water content (Karl Fischer method).....	15
5.17 Hydroxyl value	15
5.17.1 Polyester resins.....	15
5.17.2 Resins other than polyester	15
5.18 Acid value of polyester resins	15
5.19 Amount of double bonds of unsaturated polyester and acrylate resins	15
5.20 Acid and acid-anhydride content of acid-anhydride hardeners.....	15
5.21 Amine value	15
5.22 Pot life	15
5.22.1 General	15
5.22.2 Resinous compounds for cable accessories.....	15
5.23 Gel time	16
5.23.1 Unsaturated polyester based compounds	16
5.23.2 Phenolic resin based compounds.....	16
5.23.3 Other compounds	16
5.24 Exothermic temperature rise	16
5.24.1 Unsaturated polyester based compounds	16
5.24.2 Resinous compounds for cable accessories.....	16

~~5.24.3~~	~~Other compounds~~	~~17~~
5.25	Total volume shrinkage of epoxide and unsaturated polyester based compounds	17
5.26	Curing in presence of water	17
5.26.1	General	17
5.26.2	Apparatus and materials	17
5.26.3	Pouring device	18
5.26.4	Procedure	18
5.26.5	Test report	18
5.27	Determination of the degree of curing	19
5.28	Curing in thick layer and emissions during curing	19
5.28.1	General	20
5.28.2	Equipment	20
5.28.3	Test specimen	20
5.28.4	Procedure	20
6	Methods of test for cured reactive compounds	21
6.1	General	22
6.2	Test specimens	22
6.2.1	General	22
6.2.2	Preparation of the reactive compound	22
6.2.3	Preparation of test specimens	22
6.2.4	Type and number of test specimens	22
6.3	Density	23
6.4	Mechanical properties	23
6.4.1	Tensile properties	23
6.4.2	Compressive properties	23
6.4.3	Flexural properties	23
6.4.4	Impact strength	23
6.4.5	Hardness	24
6.5	Thermal properties	24
6.5.1	Bond strength at elevated temperature	24
6.5.2	Linear thermal expansion	24
6.5.3	Thermal conductivity	24
6.5.4	Glass transition	24
6.5.5	Flammability	24
6.5.6	Thermal shock	25
6.5.7	Dry heat resistance of resins for cable accessories – Method of test	25
6.5.8	Wet heat resistance of resins for cable accessories	26
6.5.9	Loss of mass	28
6.5.10	Temperature index	29
6.6	Chemical properties	29
6.6.1	Water absorption	29
6.6.2	Effect of liquid chemicals	29
6.6.3	Resistance to mould growth	30
6.6.4	Water vapour permeability	30
6.7	Electrical properties	30
6.7.1	Effect of water immersion on volume resistivity	30
6.7.2	Dielectric dissipation factor ($\tan \delta$) and relative permittivity	30
6.7.3	Break down voltage and electric strength	31

6.7.4	Proof tracking index (PTI)	33
6.7.5	Electrolytic corrosion	33
Annex A (informative)	Health and safety	38
Bibliography		39

~~Figure 1 – Test apparatus for curing in presence of water test~~

~~Figure 2 – Test set up for volume resistivity~~

Figure 1 – Examination grid

19

Figure 2 – Position of examination grid on the specimen

19

Figure 3 – Example of electrode arrangement for flexible cured compound

36

Figure 4 – Example of electrode arrangement for rigid cured compound

37

Table 1 – Condition of the top side

20

Table 2 – Condition of the bottom side

21

Table 3 – Condition of the interior

21

Table 4 – Voids

21

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FOR ELECTRICAL INSULATION –****Part 2: Methods of test****FOREWORD**

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This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC 60455-2:2015. A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text.

IEC 60455-2 has been prepared by IEC technical committee 15: Solid electrical insulating materials. It is an International Standard.

This fourth edition cancels and replaces the third edition published in 2015. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) Introduction of test methods related to IEC 60455-3-8;
- b) Additional and updated test methods for resins.

The text of this International Standard is based on the following documents:

Draft	Report on voting
15/1006/FDIS	15/1015/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in the IEC 60455 series, published under the general title *Resin based reactive compounds used for electrical insulation*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

This part of IEC 60455 is one of a series which deals with solvent-free resin based reactive compounds and their components used for electrical insulation.

The series consists of three parts:

- Part 1: Definitions and general requirements;
- Part 2: Methods of test;
- Part 3: Specifications for individual materials.

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RESIN BASED REACTIVE COMPOUNDS USED FOR ELECTRICAL INSULATION –

Part 2: Methods of test

1 Scope

This part of IEC 60455 specifies methods of test to be used for testing resin based reactive compounds, their components and cured compounds used for electrical insulation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050 (all parts), *International Electrotechnical Vocabulary* (available at <http://www.electropedia.org>)

IEC 60068-2-10:2005, *Environmental testing – Part 2-10: Tests – Test J and guidance: Mould growth*

~~IEC 60093:1980, Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials~~

IEC 60112:20032020, *Method for the determination of the proof and the comparative tracking indices of solid insulating materials*

IEC 60216 (all parts), *Electrical insulating materials – Thermal endurance properties*

IEC 60243-1:19982013, *Electrical strength of insulating materials – Test methods – Part 1: Tests at power frequencies*

~~IEC 60250:1969, Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths~~

IEC 60296:20122020, *Fluids for electrotechnical applications – ~~Unused~~ Mineral insulating oils for ~~transformers and switchgear~~ electrical equipment*

IEC 60426:2007, *Electrical insulating materials – Determination of electrolytic corrosion caused by insulating materials – Test methods*

IEC 60455-1:1998, *Resin based reactive compounds used for electrical insulation – Part 1: Definitions and general requirements*

IEC 60455-3 (all parts), *Resin based reactive compounds used for electrical insulation – Part 3: Specifications for individual materials*

IEC 60455-3-8:20132021, *Resin based reactive compounds used for electrical insulation – Part 3-8: Specifications for individual materials – Resins for cable accessories*

IEC 60695-11-10: ~~1999~~2013, *Fire hazard testing – Part 11-10: Test flames – 50 W horizontal and vertical flame test methods*

IEC 60814:1997, *Insulating liquids – Oil-impregnated paper and pressboard – Determination of water by automatic coulometric Karl Fischer titration*

IEC 61033:1991, *Test methods for the determination of bond strength of impregnating agents to an enamelled wire substrate*

IEC 61099:2010, *Insulating liquids – Specifications for unused synthetic organic esters for electrical purposes*

ISO 37:2011, *Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties*

ISO 62:2008, *Plastics – Determination of water absorption*

ISO 75 (all parts), *Plastics and ebonite – Determination of temperature of deflection under load*

ISO 175:2010, *Plastics – Methods of test for the determination of the effects of immersion in liquid chemicals, ~~including water~~*

ISO 178:2010, *Plastics – Determination of flexural properties*

ISO 179-1:2010, *Plastics – Determination of Charpy impact properties – Part 1: Non-instrumented impact test*

ISO 179-2:1997, *Plastics – Determination of Charpy impact properties – Part 2: Instrumented impact test*

ISO 291, *Plastics – Standard atmospheres for conditioning and testing*

ISO 306: ~~2004~~2013, *Plastics – Thermoplastic materials – Determination of Vicat softening temperature (VST)*

ISO 527 (all parts), *Plastics – Determination of tensile properties*

ISO 584:1982, *Plastics – Unsaturated polyester resins – Determination of reactivity at 80 degrees C (conventional method)*

ISO 604:2002, *Plastics – Determination of compressive properties*

ISO 868:2003, *Plastics and ebonite – Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1183-1: ~~2012~~2019, *Plastics – Methods for determining the density of non-cellular plastics – Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1513:2010, *Paints and varnishes – Examination and preparation of test samples ~~for testing~~*

ISO 1523:2002, ~~Paints, varnishes, petroleum and related products~~ *Determination of flash point – Closed cup equilibrium method*

ISO 1675:1985, *Plastics – Liquid resins – Determination of density by the pycnometer method*

ISO 2039-1:1993, *Plastics – Determination of hardness – Part 1: Ball indentation method*

ISO 2114:19962000, *Plastics — ~~Unsaturated~~ (polyester resins) and paints and varnishes (binders) – Determination of partial acid value and total acid value*

ISO 2431:1993, *Paints and varnishes – Determination of flow time by use of flow cups*

ISO 2535:1997, *Plastics – Unsaturated polyester resins – Measurement of gel time at 25 degrees C*

ISO 2554:1997, *Plastics – Unsaturated polyester resins – Determination of hydroxyl value*

ISO 2555:1989, *Plastics – Resins in the liquid state or as emulsions or dispersions – Determination of apparent viscosity ~~by the Brookfield test~~ using a single cylinder type rotational viscometer method*

ISO 2592:1973, *Petroleum products – Determination of flash and fire points – Cleveland open cup method*

ISO 3001:1997, *Plastics — ~~Epoxide~~ Epoxy compounds – Determination of ~~epoxide~~ epoxy equivalent*

ISO 3219:1993, *Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 3451-1:1997, *Plastics – Determination of ash – Part 1: General methods*

ISO 3521:1997, *Plastics – Unsaturated polyester and epoxy resins – Determination of overall volume shrinkage*

ISO 3679:1983, *Paints, varnishes, petroleum and related products – Determination of flashpoint – Rapid equilibrium method*

ISO 4573:1978, *Plastics – Epoxide resins and glycidyl esters – Determination of inorganic chlorine*

ISO 4583:1998, *Plastics – Epoxide resins and related materials – Determination of easily saponifiable chlorine*

ISO 4615:1979, *Plastics – Unsaturated polyesters and epoxide resins – Determination of total chlorine content*

ISO 4625:1980, *Binders for paints and varnishes – Determination of softening point – Ring-and-ball method*

ISO 4895, *Plastics – Liquid epoxy resins – Determination of tendency to crystallize*

ISO 7056, *Plastics laboratory ware – Beakers*

ISO 9396:1997, *Plastics – Phenolic resins – Determination of the gel time ~~at a given temperature~~ of resols under specific conditions using automatic apparatus*

ISO 11357-2:1999, *Plastics – Differential scanning calorimetry (DSC) – Part 2: Determination of glass transition temperature and step height*

ISO 11359-2:1999, *Plastics – Thermomechanical analysis (TMA) – Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

ISO 11359-3:2002, *Plastics – Thermomechanical analysis (TMA) – Part 3: Determination of penetration temperature*

ISO 14896:2009, *Plastics – Polyurethane raw materials – Determination of isocyanate content*

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes – Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60455-1 and IEC 60050 and the following shall apply.

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

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

3.1

volume resistance

part of the insulation resistance which is due to conduction through the volume and excluding surface current

3.2

volume resistivity

volume resistance reduced to a cubical unit volume

3.3

dielectric dissipation factor

$\tan \delta$

numerical value of the ratio of the imaginary to the real part of the complex permittivity

3.4

relative permittivity

ϵ_r

ratio of the absolute permittivity to the electric constant

Note 1 to entry: In practical engineering, it is usual to employ the term 'permittivity' when referring to relative permittivity.

4 General notes on methods of test

4.1 Preparation and conditioning

Unless otherwise specified in the relevant specification standard or in the method of test, all tests shall be carried out at atmospheric conditions in a temperature range of between 21 °C and 29 °C and a relative humidity range of between 45 % and 70 %. Before measurements are made, the sample or test specimen shall be pre-conditioned under these atmospheric conditions for a time sufficient to allow the sample or the test specimen to reach stability. For taking samples in liquid or paste form, ISO 15528 shall be applied. For preparation of such samples for testing, ISO 1513 shall be applied.

NOTE For definitions of terms for standard atmospheres, see ISO 558. The test atmosphere as specified above does not comply with any of the two standard atmospheres as specified in ISO 291 but covers both ranges inclusive of their tolerances

Normally, all requirements for a method of test are given in the description, and diagrams are intended only to illustrate one possible arrangement for conducting the test. In case of inconsistencies between this document and the specification sheets of the IEC 60455-3 series, the latter shall prevail. When another standard is invoked for a test method, reference to that standard shall be included in the report.

4.2 Sequence of tests

To avoid unnecessary efforts, tests shall be carried out on the samples in the following sequence:

- 1) tests on individual components prior to mixing;
- 2) tests on reactive compound just after mixing (ready to use);
- 3) tests on cured compound;
- 4) tests on cured compound after pre treatment (thermal, humidity, water etc.).

If the sample under test fails a test the following tests may become obsolete.

4.3 Test report

If not otherwise specified, the test report shall include the following data:

- 1) resin designation and identification;
- 2) lot number or other identification;
- 3) confirmation of marking and labelling according to the material safety data sheet (MSDS);
- 4) test results;
- 5) major test parameters, including conditioning and calibration, if any;
- 6) processing conditions used to reactive compound;
- 7) copy of the technical data sheet (TDS) and (MSDS).

5 Methods of test for reactive compounds and their components

5.1 Flash point

For flash point temperatures of 79 °C and above, the method given in ISO 2592 shall be used. For flash point temperatures below 79 °C, the method given in ISO 1523 shall be used with any of the closed-cup apparatus as described in Annex A of ISO 1523:2002. ISO 1523 shall be read in conjunction with ISO 3679. Two measurements shall be made on two separate samples, and the two results of the flash point shall be reported along with reference to the standards applied.

5.2 Density

The method given in ISO 1675 shall be used. Two measurements shall be made, and the two results of the density shall be reported.

5.3 Viscosity

The viscosity shall be determined with a suitable device at $(23 \pm 0,5)$ °C if not otherwise specified. If a rotating type of device is used, it shall be in accordance with ISO 2555 (Brookfield type) or with ISO 3219 (a type working at a defined shear rate). If an efflux type of equipment is used, the method of test and the flow cup shall be in accordance with ISO 2431. Two measurements shall be made, and the two results of the viscosity shall be reported, along with reference to the standards applied.

5.4 Viscosity after storing at elevated temperature

This method is not applicable to one-component systems or components containing hardener.

If not otherwise specified, a sample of sufficient amount is stored for $(20 \pm 0,5)$ h at a temperature of (100 ± 3) °C in a sealed container. After cooling down to room temperature the viscosity is measured according to 5.3. The increase of viscosity is calculated using the following equation:

$$\text{Increase of viscosity in \%} = (\eta_2 - \eta_1) \times 100 / \eta_1$$

where

η_1 dynamic viscosity before storing

η_2 dynamic viscosity after storing

5.5 Content of volatile organic components

This method is not applicable to one-component systems or components containing hardener. If not otherwise specified, the test shall be carried out in the following way:

The mass of an empty weighing bottle (about 80 mm × 30 mm) is taken to 0,001 g (m_1). A mass of 0,4 g to 0,5 g resin (m_2) is weighed to 0,001 g into the weighing bottle (well closed during weighing). Some drops of toluene are added to dilute the resin. The liquid is spread on the floor of the weighing bottle with a slight twist.

The open weighing bottle is placed into an oven with forced air circulation for at least 2 h at (110 ± 2) °C. After cooling down to room temperature in a desiccator the weighing bottle is weighed again to 0,001 g (m_3).

~~$$\text{Volatile organic components} = 100 \times (m_2 - (m_3 - m_1)) / m_2$$~~

$$\text{Volatile organic components} = 100 \times (m_2 - (m_3 - m_1)) / m_2$$

where:

m_1 mass of an empty weighing bottle (g)

m_2 mass of resin before heating (g)

m_3 mass of an empty weighing bottle with resin after heating and cooling (g)

5.6 Isothermal increase of viscosity (processing time)

This method is designed for PUR and EP resins. For UP resins gel time shall be used. If not otherwise specified, the test shall be carried out in the following way:

All components and equipment shall be at room temperature. The components of the resin are mixed according to the manufacturer's instructions. The mixing procedure shall not take more than 3 min. The time measurement starts after adding and mixing of the last component. After 10 min the first viscosity measurement is taken as the initial value. The measurement is repeated until the specified maximum viscosity is reached. The time between the initial value and the maximum value is reported as processing time.

5.7 Shelf life

The shelf life shall be determined by measurement of the change in a specified characteristic property after a certain storage time and temperature. Experience has shown that viscosity according to 5.3 and gel time according to 5.23 are appropriate characteristics. To assess shelf life, viscosity and/or gel time shall be determined according to 5.3 and/or 5.23 respectively, at a temperature and with an end-point as agreed upon between supplier and purchaser. Two measurements shall be made on both fresh material and on material stored for a time and at a temperature as agreed between supplier and purchaser. The two results of shelf life shall be reported, along with reference to the standards applied. The results shall contain the viscosity

and/or the gel time before and after storing, the storing time and temperature and the test temperature.

5.8 Colour

The method given in ISO 6271 shall be used. Two measurements shall be made, and the two results of colour shall be reported along with reference to the standard applied.

5.9 Softening temperature

The method given in ISO 306 or ISO 4625 shall be used. Two measurements shall be made, and the two results of softening temperature shall be reported along with reference to the standard applied.

5.10 Ash content

The method given in ISO 3451-1, method A, shall be used. Two measurements shall be made, and the two results of the ash content shall be reported.

5.11 Filler content

To be agreed between supplier and purchaser.

5.12 Chlorine content

5.12.1 Total chlorine content of unsaturated polyesters and epoxide resins

The method given in ISO 4615 shall be used. Two measurements shall be made, and the two results of the total chlorine content shall be reported.

5.12.2 Inorganic chlorine content of epoxide resins and glycidyl esters

The method given in ISO 4573 shall be used. Two measurements shall be made, and the two results of the inorganic chlorine content shall be reported.

5.12.3 Easily saponifiable chlorine content of epoxide resins and related materials

The method given in ISO 4583 shall be used. Two measurements shall be made, and the two results of the saponifiable chlorine content shall be reported.

5.13 Tendency of crystallisation

This method is applicable to epoxy resins only.

The method given in ISO 4895 shall be used. Two measurements shall be made, and the two results shall be reported.

5.14 Epoxide equivalent of epoxide resins

The method given in ISO 3001 shall be used. Two measurements shall be made, and the two results of the epoxide equivalent shall be reported.

5.15 Content of isocyanate

This method is applicable to polyurethane hardeners only.

The method given in ISO 14896 shall be used. Two measurements shall be made, and the two results shall be reported.

5.16 Water content (Karl Fischer method)

The method given in IEC 60814 shall be used. Two measurements shall be made, and the two results of the water content shall be reported.

5.17 Hydroxyl value

5.17.1 Polyester resins

The method given in ISO 2554 shall be used. Two measurements shall be made, and the two results of the hydroxyl value shall be reported.

5.17.2 Resins other than polyester

To be agreed between supplier and purchaser.

5.18 Acid value of polyester resins

The method given in ISO 2114 shall be used. Two measurements shall be made, and the two results of the acid value shall be reported.

5.19 Amount of double bonds of unsaturated polyester and acrylate resins

To be agreed between supplier and purchaser.

5.20 Acid and acid-anhydride content of acid-anhydride hardeners

The method given in ISO 2114 shall be used for acid content. The method given in ISO 7327 shall be used for anhydride content. Two measurements shall be made, and the two results of the acid value and/or anhydride content shall be reported.

5.21 Amine value

The method given in ISO 9702 shall be used if not otherwise agreed between supplier and purchaser.

5.22 Pot life

5.22.1 General

The time which a particular method requires to achieve a viscosity of 50 Pas is measured.

The viscosity can be determined in any viscosimeter.

5.22.2 Resinous compounds for cable accessories

5.22.2.1 Apparatus and materials

Equipment to be used:

- beaker in accordance with ISO 7056, polyethylene, polypropylene or glass with dimensions: 70 mm to 100 mm diameter and 70 mm to 130 mm high;
- environmental chamber;
- time measuring device;
- viscosimeter.

5.22.2.2 Preparation of the sample

The beaker shall be marked to a volume of (300 ± 25) ml.

The beaker and a standard manufacturer pack shall be stored in an environmental chamber for 24 h at the temperature specified with a tolerance of $\begin{smallmatrix} 0 \\ -2 \end{smallmatrix}$ K.

5.22.2.3 Procedure

The components shall be removed from the environmental chamber and start mixing immediately according to the supplier's instructions. The time starts at the commencement of mixing the components.

Remove the beaker from the environmental chamber, and pour the mixed compound into the beaker up to the mark previously made.

5.22.2.4 Test report

The test report shall include the following information:

- the time is determined from the start of mixing until a viscosity of 50 Pas is reached. The pot life shall be rounded to the nearest of a second;
- the viscosimeter, test parameters and system used (e.g. spindle size, rotation speed etc.).

The viscosimeter with the system used shall be calibrated at 50 Pas.

5.23 Gel time

5.23.1 Unsaturated polyester based compounds

Gel time is the period of time after which the reactive compound reaches the gel state. The method given in ISO 2535 shall be used at a test temperature as agreed upon between supplier and purchaser. Two measurements shall be made, and the two results of the gel time shall be reported along with the test temperature.

5.23.2 Phenolic resin based compounds

The method given in ISO 9396 shall be used. Two measurements shall be made, and the two results of the gel time shall be reported.

5.23.3 Other compounds

If applicable, the methods given in ISO 2535 or ~~ISO 9696~~ ISO 9396, or ISO 8987 shall be used.

5.24 Exothermic temperature rise

5.24.1 Unsaturated polyester based compounds

The method given in ISO 584 shall be used. Two measurements shall be made, and the two results of the exothermic temperature rise shall be reported.

5.24.2 Resinous compounds for cable accessories

5.24.2.1 Apparatus and materials

Equipment to be used:

- beaker in accordance with ISO 7056, polyethylene, polypropylene or glass with dimensions: 70 mm to 100 mm diameter and 70 mm to 130 mm high;
- thermocouple made with twisted wires;
- temperature recorder;
- time measurement device.

5.24.2.2 Preparation of the sample: Test at 23 °C

The beaker shall be marked to a volume of (300 ± 25) ml. The beaker and a sufficient amount of resinous compound components (prepare 400 ml to 700 ml of the compound) shall be stored for 24 h at (23 ± 2) °C.

The components shall be mixed according to manufacturer's instructions. The time starts at the commencement of mixing the components.

The mixed components shall be poured into the beaker up to the mark previously made. The thermocouple shall be inserted vertically in the centre of the mixture to a depth of (25 ± 5) mm under the compound surface and held in position by a suitable device (e.g. ring-stand).

5.24.2.3 Procedure

The temperature increase shall be checked, and the maximum temperature rise and the time to peak recorded. The test is finished after the highest temperature has been reached and the temperature starts to drop significantly.

NOTE For ease of testing, the temperature can be recorded as a curve.

5.24.2.4 Test report

The maximum temperature reached (rounded to the nearest °C) and the time to peak (rounded to the nearest min) shall be reported.

~~5.24.3 Other compounds~~

~~Method of test required, but not available.~~

5.25 Total volume shrinkage of epoxide and unsaturated polyester based compounds

The method given in ISO 3521 shall be used. Two measurements shall be made, and the two results of the total volume shrinkage shall be reported. The report shall contain the test temperature, the density of the compound at test temperature, and the density of the specimen made of the cured compound.

5.26 Curing in presence of water

5.26.1 General

This test applies only to ~~polyurethane resinous~~ compound for cable accessories. This test is to measure ~~the gas evolution and~~ the physical structure change when the polyurethane resinous compound cures in presence of water. Voids created by generation of gas in the resin may lead to electrical break down.

The density of the cured resin mentioned in the technical data sheet shall be $> 1,05 \text{ g/cm}^3$.

5.26.2 Apparatus and materials

Equipment to be used:

- 250 ml transparent/translucent PE squirt bottle ~~with screw-on squirt cap~~;
- funnel that meets the requirements of 5.26.3;
- ~~tubing, inner diameter corresponding to squirt cap tube outer diameter;~~
- ~~beaker or bucket;~~
- ~~100 ml graduated cylinder.~~

NOTE Take care that the equipment is clean (no algae).

5.26.3 Pouring device

Use a funnel with a capability to fill the bottle within 30 s to 60 s at room temperature.

5.26.4 Procedure

~~The test apparatus shall be assembled as shown in Figure 1. The graduated cylinder shall be completely filled with water from the bucket. The squirt bottle shall then be screwed off from the squirt cap, be~~ marked with 2 marks at 100 ml and 200 ml respectively and filled up to the 100 ml mark with de-mineralized water of $(23 \pm 2) ^\circ\text{C}$.

The ~~bucket~~, squirt bottle and resinous compound left in its original packaging shall be allowed to reach room temperature. The compound package shall be opened, the stopwatch started, and the components mixed according to manufacturer's instructions. The mixed compound is poured into the closed funnel. The funnel shall be emptied immediately into the squirt bottle until the water level reaches the 200 ml mark. Care shall be taken to pour in the centre of the bottle and avoid touching of the inner bottle wall.

~~Within the next 30 s, screw the screw cap onto the squirt bottle until the connection is gastight, taking care not to deform the flexible tubing or squirt bottle. During the connection and subsequent curing the flexible tubing and squirt bottle shall not be deformed; if the tubing and/or the bottle become deformed, restart the test.~~ The compound shall be allowed to cure for the pot life specified by the manufacturer. At least one hour after the pot life has elapsed, ~~the volume of gas fill in the graduated cylinder shall be read.~~ The water shall be poured out of the squirt bottle. After hardening of the resin at room temperature for 24 h, the specimen shall be vertical cut through the centre of the bottle and clean the cut surface with compressed air. Examine the physical structure ~~examined~~ for the presence of blisters or cracks in the compound using the examination grid.

Examination grid

Evaluate concentration and dimension of bubbles created by using an examination grid. The grid measures 45×24 mm (width $\times$ height) and is made up of 15 cells of dimensions 9×8 mm, as shown in Figure 1.

5.26.5 Test report

The following parameters shall be measured and reported:

• ~~gas volume;~~

- physical structure/presence of blisters or cracks or bubbles and/or inclusions;
- picture of the cut surface of the compound with scale.

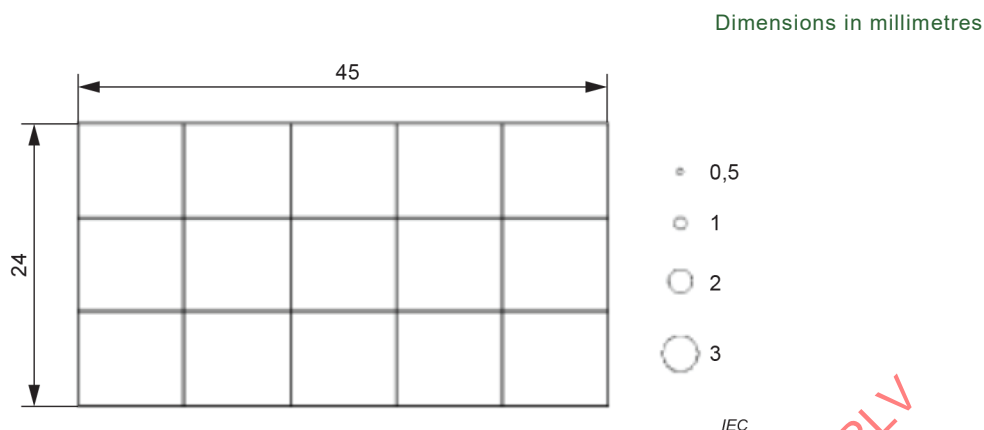


Figure 1 – Examination grid

Position the grid centrally on the cut surface. To pass the examination no bubbles > 3 mm are allowed and at least 5 cells must be free of bubbles. Count only bubbles with diameter ≥ 1 mm.

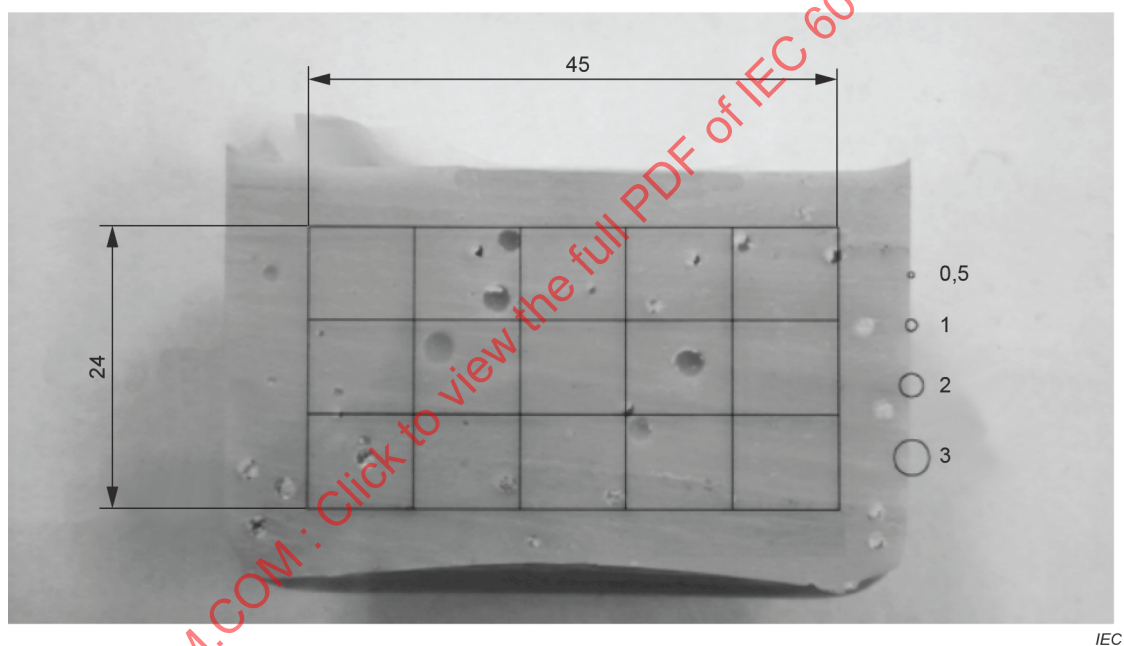


Figure 2 – Position of examination grid on the specimen

Figure 2 shows the position of the examination grid on the specimen.

5.27 Determination of the degree of curing

The reaction enthalpie of the uncured resin is determined by using DSC. An amount of 10 mg has been found adequate.

If not otherwise specified, about 10 g of resin is cured under conditions (temperature and time) as agreed between supplier and purchaser. The residual enthalpie is determined using a sample of about 10 mg.

The degree of curing is calculated as follows:

$$\text{Degree of curing in \%} = 100 - H_c \times 100 / H_u$$

where

H_c = enthalpie of cured resin

H_u = enthalpie of uncured resin

$$Doc = 100 - H_c \times 100 / H_u$$

where

Doc degree of curing (%)

H_c enthalpie of cured resin (J/g)

H_u enthalpie of uncured resin (J/g)

5.28 Curing in thick layer and emissions during curing

5.28.1 General

This test is designed for the examination of cured impregnating materials. Curing in thick layer is expressed by the condition after curing of the top side, bottom side and interior of the specimen. The determination of emissions during the curing process is also part of this method.

5.28.2 Equipment

The following equipment shall be used:

- flat and smooth square pieces of aluminium foil, 0,1 mm to 0,15 mm in thickness and of (95 ± 1) mm side length;
- a former made of metal or any other suitable solid material, (25 ± 1) mm in thickness and (45 ± 1) mm square;
- an oven with forced air circulation and with a minimum rate of 8 fresh air changes per hour. The oven shall be of the type and design used for drying and/or curing specimens;
- a balance with an accuracy of 0,01 g.

5.28.3 Test specimen

A piece of aluminium foil shall be cleaned by adequate means and shall be folded around the former to produce a square mould of about 45 mm side length. The square mould shall be dried at (110 ± 5) °C for (10 ± 1) min, cooled down and stored in a desiccator.

The mass of the square mould shall be weighed to 0,01 g (m_1). A resin sample with a mass between 9,90 g and of 10,10 g shall be weighed to 0,01 g into the mould (m_2).

The specimen shall be cured at a temperature and for a time as agreed between supplier and purchaser. The specimen shall be cooled down to room temperature in a desiccator and the mass shall be taken to 0,01 g (m_3). Subsequently the aluminium foil is removed.

5.28.4 Procedure

5.28.4.1 Description of the specimen

~~The specimen shall be assessed by description of the conditions of its top side, bottom side and interior, expressed according to statements for visual appearance and for tackiness by the symbols given in the following Tables 1, 2 and 3:~~

Table 1, Table 2, Table 3 and Table 4 give descriptions of the specimen.

Table 1 – Condition of the top side

Condition	Symbol
Smooth	S1
Wrinkled	S2

Table 2 – Condition of the bottom side

Condition	Symbol
Non-tacky	U1
Tacky	U2

Table 3 – Condition of the interior

Condition	Symbol
Rigid	I1.X
Horn-like machinable	I2.X
Leather-like	I3.X
Rubber-like	I4.X
Gel-like	I5.X
Liquid	I6.X

Table 4 – Voids

The specimen contains	X
No voids	1
Not more than five voids	2
More than five voids	3

For the condition of the interior a statement shall be added whether the interior is uniform or not uniform.

NOTE It can be necessary to cut the specimen, to bend the specimen with the fingers or to use a knife for describing the mechanical properties.

5.28.4.2 Emissions

The emissions shall be calculated by the following equation:

$$E = 100 - ((m_2 - (m_3 - m_1)) \times 100 / m_2)$$

where

m_1 = mass of the empty square mould

m_2 = mass of the uncured resin (without mass of the square mould)

m_3 = mass of the square mould with cured resin

$$E = 100 - ((m2 - (m3 - m1)) \times 100 / m2)$$

where:

E emissions

m1 mass of the empty square mould

m2 mass of the uncured resin (without mass of the square mould)

m3 mass of the square mould with cured resin

6 Methods of test for cured reactive compounds

6.1 General

The cured compound is self-supporting and thus allows the preparation of rigid and flexible test specimens.

6.2 Test specimens

6.2.1 General

When the term "test specimen" is used it means solid parts of the cured material in a shape as required for the method of test concerned. In 6.2.2, 6.2.3 and 6.2.4 such "test specimens" are referred to as "specimens".

6.2.2 Preparation of the reactive compound

The reactive compound shall be a homogeneous mixture of such portions of the components as specified by the supplier. Also drying, de-aerating, heating, and other measures to treat the components and the compound shall comply with the instructions given by the supplier. When compounds contain fillers, the possibility of settlement needs to be taken into account.

6.2.3 Preparation of test specimens

Specimens shall be prepared under conditions as specified in the particular method of test in the relevant specification sheet of IEC 60455-3, or as agreed upon between supplier and purchaser. This includes the casting process with respect to temperature and vacuum, the curing conditions with respect to temperature, and time or temperature-time programme, the removal from the mould and annealing and cooling. Reactive compounds, which according to the instructions given by the supplier, cure at ambient temperature, generally reach the final state at ambient temperature only after days or weeks. To achieve a defined degree of cure in such conditions, the compounds shall be cured for 24 h at ambient temperature and immediately thereafter for 24 h at 80 °C, or as agreed between supplier and purchaser. Specimens shall be cast in the proper shape according to the dimensions given in the method of test or shall be prepared from cast pieces. They shall be free of voids, bubbles, nicks, and scratches. During machining, excessive heating of the machined surfaces shall be avoided by cooling, for instance with water.

NOTE Removal of the cured compound from the mould is facilitated by the use of release agents and moulds made of chromium-plated or other adequate material.

6.2.4 Type and number of test specimens

The type and number of specimens required for a particular method of test are specified in the method of test, in the relevant specification sheet of IEC 60455-3 or shall be agreed upon between supplier and purchaser.

6.3 Density

A method given in ISO 1183-1 shall be used. Two measurements shall be made. The kind of preparation and the dimensions of the specimen, the method used, and the two results of the density shall be reported.

6.4 Mechanical properties

6.4.1 Tensile properties

6.4.1.1 Rigid material

The method given in ISO 527 shall be used with a speed of testing to break the specimen within (60 ± 15) s. The type of specimen shall be selected from ISO 527. Five specimens shall be tested. The kind of preparation, the dimensions and the type of the specimen, the speed of testing, and the five results of the tensile properties shall be reported. As far as applicable, the report shall contain tensile stress at yield, at maximum load and at break, percentage elongation at yield and at break, and modulus of elasticity.

6.4.1.2 Flexible material

The method given in ISO 37 shall be used for a dumb-bell specimen. Five specimens shall be tested. The kind of preparation and the type of dumb-bell specimen, and the five results of the tensile properties shall be reported. The report shall contain tensile strength, percentage elongation at break, and modulus of elasticity.

6.4.2 Compressive properties

The method given in ISO 604 shall be used. Five specimens shall be tested. The kind of preparation and the dimensions of the specimen, the rate of deformation, and the five results of the compressive properties shall be reported. As far as applicable, the report shall contain compressive strength at maximum load, compressive yield stress, and percentage compressive strain at rupture.

6.4.3 Flexural properties

The method given in ISO 178 shall be used with a relative rate of movement of the loading nose and supports, so that the specimen ruptures or reaches the maximum load within (60 ± 15) s. Five specimens shall be tested. The kind of preparation and the dimensions of the specimen, the relative rate of movement, and the five results of the flexural properties shall be reported. As far as applicable, the report shall contain the flexural stress at rupture or at maximum load, the corresponding deflection, and the modulus of elasticity.

6.4.4 Impact strength

6.4.4.1 Unnotched specimens

The method given in ISO 179-1 or ISO 179-2 shall be used. Ten specimens shall be tested. The kind of preparation, the dimensions, and the type of specimen, as well as the 10 results of the impact strength shall be reported.

6.4.4.2 Notched specimens

The method given in ISO 179-1 or ISO 179-2 shall be used with a notched specimen. Ten specimens shall be tested. The kind of preparation, the dimensions and type of specimen as well as the 10 results of the impact strength shall be reported.

6.4.5 Hardness

6.4.5.1 Rigid material

The method given in ISO 2039-1 (ball indentation method), or the method given in ISO 868 (Shore D hardness) shall be used. Five measurements shall be made on one or more specimens. The kind of preparation and the dimensions of the specimen, the test load applied, and the five results of the hardness shall be reported.

6.4.5.2 Flexible material

The method given in ISO 868 (preferably Shore A hardness) shall be used. Five measurements shall be made on one or more specimens. The kind of preparation, and the dimensions of the specimen, the type of durometer (A or D) used, and the five results of the indentation hardness shall be reported.

6.5 Thermal properties

6.5.1 Bond strength at elevated temperature

The twisted coil test, method A, or the helical coil test, method B, given in IEC 61033, shall be used. The test temperature shall be in accordance with the relevant specification sheet of IEC 60455-3, or shall be agreed upon between supplier and purchaser. Five specimens shall be tested. The method, the types of enamelled winding wire used as substrate, and the five results shall be reported.

6.5.2 Linear thermal expansion

The method given in ISO 11359-2 shall be used. The kind of preparation, the dimensions of the specimen and the result shall be reported.

6.5.3 Thermal conductivity

To be agreed between supplier and purchaser.

6.5.4 Glass transition

6.5.4.1 Glass transition temperature

One of the methods given in ISO 11357-2, ISO 11359-2 or ISO 11359-3 shall be used. Two measurements shall be made. The kind of preparation and, if applicable, the dimensions of the specimen, the method used (A: DSC or DTA, B1: TMA, expansion mode, or B2: TMA, penetration mode), and the two results of the glass transition temperature shall be reported.

6.5.4.2 Temperature of deflection under load

Method A or method B given in ISO 75 shall be used. Two specimens shall be tested. The kind of preparation and the dimensions of the specimen, the method used, and the two results of the temperature of deflection under load shall be reported.

NOTE The temperature of deflection under load is of the same nature as the glass transition temperature, but the methods given in ISO 75 do not allow temperatures below 40 °C to be determined. The methods according to 6.5.4.1 can preferably be applied.

6.5.5 Flammability

The methods A and B given in IEC 60695-11-10 shall be used. For each method five specimens shall be tested. Method ~~B~~ A shall be applied only in the case where the result according to method ~~A~~ B is worse than category V-2. The kind of preparation and the dimensions of the test specimen, as well as the result of flammability obtained with method B and, if applicable, with method A, shall be reported.

6.5.6 Thermal shock

To be agreed between supplier and purchaser.

6.5.7 Dry heat resistance of resins for cable accessories – Method of test

6.5.7.1 General

This test is used to determine the effect of extended exposure to dry heat on loss of mass and if required impact strength.

6.5.7.2 Specimen

Prepare the specimen from the same batch of resinous compound. The resinous compound shall be prepared and cured in accordance with the manufacturer's instructions.

- Loss of mass: a set of 3 test specimens shall be prepared with the dimensions of 80 mm × 40 mm × 5 mm and individually identified.
- Impact strength: a set of 5 test specimens shall be prepared in accordance with ISO 179.

6.5.7.3 Apparatus and materials

Equipment to be used:

- analytic balance with accuracy of ± 1 mg;
- forced air oven with temperature control;
- impact tester;
- desiccators.

6.5.7.4 Procedure

The test specimens, prepared in accordance with 6.5.7.2, shall be used as follows:

- The loss of mass specimen shall be marked individually. Take the mass by weighing it individually to the nearest of 1 mg (= mass m_1).
- All test specimens shall be placed in a forced air oven for 28 days at (120 ± 2) °C or temperature stated in IEC 60455-3-8. Any mechanical loading of the specimen or any direct contact between them shall be avoided. Only one type of resinous compound shall be tested at one time. At the end of the 28 days, the set of test specimen shall be removed from the forced air oven.
- The set of test specimen shall then be placed for 8 h in a desiccator and cooled to (23 ± 2) °C.
- The loss of mass specimens shall be weighed individually to the nearest 1 mg (= mass m_2) immediately after removal from the desiccator.
- The impact strength shall be measured in accordance with ISO 179.

6.5.7.5 Assessment

The percent loss of mass shall be calculated for each individual sample, in accordance with the following equation:

~~$$M = ((m_1 - m_2) / m_1) \times 100$$~~

$$M = ((m_1 - m_2) / m_1) \times 100$$

Where:

M loss of mass (%)

*m*1 mass of weighing of test specimen individually identified before heating (g)

*m*2 mass of weighing of test specimen individually identified after heating and cooling (g)

6.5.7.6 Test report

The test report shall include the following information:

- the average of percentage loss of mass;
- the average of the impact strength if required.

6.5.8 Wet heat resistance of resins for cable accessories

6.5.8.1 General

This test shall be carried out to the following requirements:

- a) the samples shall be prepared in accordance with the methods given in the relevant sheet of IEC 60455-3, to carry out the following tests: hardness, tensile and elongation, electric strength;
- b) final measurement of reference samples and treated samples shall be done the same day successively.

6.5.8.2 Test sequence

The test sequence shall be as follows:

- 1) Preparation of the test specimen from the same lot of resinous compound.
- 2) Immersion for 28 days at $(70 \pm 2) ^\circ\text{C}$ in water.
- 3) Drying for 48 h at $(60 \pm 2) ^\circ\text{C}$ under vacuum.
- 4) Drying 24 h at $80 ^\circ\text{C}$ with open ventilation slot.
- 5) Cooling in desiccator to $(23 \pm 2) ^\circ\text{C}$ (8 h).
- 6) Test of specimens.

6.5.8.3 General

This test is used to determine the resistance to hydrolysis of resinous compounds exposed to the simultaneous influence of water and temperature. It determines any irreversible change in selected mechanical properties by means of a tensile test and of a hardness test, and electrical properties by means of dielectric strength.

6.5.8.4 Specimen

The resinous compound shall be prepared and cured in accordance with the manufacturer's instructions.

Mechanical test:

A set of 10 test specimens no. 1B shall be prepared in accordance with 6.4.1.2 and ISO 527-1, ISO 527-2 and ISO 527-3 from blister free plates of resinous compound of thickness of $(4 \pm 0,2)$ mm.

NOTE 1 For highly filled compound, specimens can be cast in a mould of adequate dimension for the test.

Electrical test:

A set of 5 test specimens shall be prepared in accordance with ~~IEC 60093~~ IEC 60243-1 for the electric strength tester.

NOTE 2—IEC 60093 will be replaced by IEC 62631 in the future. Users are encouraged to investigate the status of standardisation of IEC 62631 and the possibility of applying the standard. Members of ISO and IEC maintain registers of currently valid International Standards.

6.5.8.5 Apparatus and materials

Equipment to be used:

- hydrolysis vessel with a minimum capacity of 1 l for 5 test specimens each;
- forced air oven with temperature control;
- heating /vacuum cabinet;
- Shore hardness tester;
- tensile tester;
- ~~volume resistivity tester;~~
- dielectric strength tester;
- desiccator.

6.5.8.6 Procedure

The test specimens, shall be used as follows:

- A set of 5 mechanical test specimens shall be kept as reference samples. They shall be stored in the test room at standard atmosphere 23/50 in accordance with ISO 291.
- A set of 5 each of the mechanical test and of the electrical test specimens shall be immersed in water in the hydrolysis vessel for 28 days at 70 °C. The test specimens shall be placed in the hydrolysis vessel in such a manner that they are surrounded by water and immersed to a minimum water head of 10 mm. Any mechanical loading of the specimens or any direct contact between them shall be avoided. Only one type of resinous compound shall be tested at one time. The water conductivity shall be less than 500 µS/m and its pH value shall be 7,0 ± 0,5. The hydrolysis vessel shall then be closed with a suitable cover and shall be placed in a ~~heating cabinet~~ forced air oven at (70 ± 2) °C and kept at that temperature during the 28 days. At the end of the 28 days, the set of test specimens shall be removed from the ~~heating cabinet~~ forced air oven and from the vessel.

The set of test specimens shall then be placed respectively in a separate heating/vacuum cabinet and dried for 48 h at (60 ± 2) °C under a (1,5 ± 0,2) kPa vacuum, ~~in storage~~ then store for 24 h at (80 ± 2) °C ~~with an open ventilation slot~~ in a forced air oven, and then shall be cooled down to (23 ± 2) °C for 8 h in desiccators.

6.5.8.7 Conditioning and assessment

The test specimens shall be conditioned before the hardness, the tensile/elongation and the electric strength tests for 24 h in accordance with ISO 291 using an atmosphere 23/50. The mechanical test specimens (reference and wet aged) shall be subjected to hardness measurement, followed by tensile/elongation measurement.

The hardness test shall be carried out in accordance with ISO 868 on ends of the mechanical test specimens outside their clamping length and in the centre of their surfaces.

The tensile and elongation test shall be carried out in accordance with ISO 527, with a jaw speed of 50 mm/min up to Shore D hardness 70.

A jaw speed of 5 mm/min shall be applied if the Shore hardness is ≥ 70.

The electrical test specimens shall be subjected to electric strength measurement in accordance with ~~IEC 60093~~ IEC 60243-1.

For mechanical tests the following arithmetic mean for each property shall be calculated:

e_1 value on the reference test specimens;

e_2 value on the aged test specimens.

The retained percentage of the values for these properties after the temperature/water exposure shall be calculated using the following equation:

$$E = (E_2 / E_1) \times 100$$

$$E = (e_2 / e_1) \times 100$$

where:

E values for these properties after the temperature/water exposure (%)

e_1 value on the reference test specimens

e_2 value on the aged test specimens

For the electric strength test, the arithmetic mean of the test specimens shall be calculated and compared to the values in IEC 60455-3-8:2013, Table 2.

6.5.8.8 Test report

The test report shall include the following information:

- retained percent of Shore hardness;
- retained percent of tensile strength;
- retained percent of elongation at break;
- value of dielectric strength.

6.5.9 Loss of mass

6.5.9.1 General

This method applies to resinous compounds for cable accessories.

6.5.9.2 Apparatus and materials

Equipment to be used:

- analytic balance with accuracy ± 1 mg;
- constant temperature oven with ventilation;
- desiccator with calcium dichloride as a drying agent.

6.5.9.3 Specimen

Three specimens with the dimension of 80 mm $\times$ 40 mm $\times$ 5 mm shall be conditioned for 24 h at standard atmosphere 23/50 in accordance with ISO 291 and individually identified.

6.5.9.4 Procedure

The three conditioned test specimens shall be weighed individually to the nearest 1 mg (= mass m_1) and stored for one week at $(120 \pm 2)^\circ\text{C}$ in the vented oven. They shall then be cooled in a desiccator. Immediately after removal from the desiccator, the test specimens shall be weighed individually to the nearest 1 mg (= mass m_2).

6.5.9.5 Results

The percent loss of mass, M , shall be calculated for each individual sample, in accordance with the following equation:

$$M = \frac{m_1 - m_2}{m_1} \times 100$$

$$M = ((m1 - m2) / m1) \times 100$$

where

M Loss of mass (%)

$m1$ mass of test specimens individually before heating (mg)

$m2$ mass of test specimens individually after cooling (mg)

The average value of the three measurements shall be reported.

6.5.10 Temperature index

NOTE The temperature index depends on the choice of test criterion and of the end-point criterion. Therefore, for one and the same material, results for the temperature index can vary by 80 K or more.

6.5.10.1 Procedure

The method given in IEC 60216 shall be used. The test and end-point criteria shall be in accordance with the relevant specification sheet of IEC 60455-3, or shall be agreed upon between supplier and purchaser. Two test criteria shall be used. For each test criterion at least three exposure temperatures shall be applied. The difference between two subsequent exposure temperatures shall not exceed 20 K. If the correlation coefficient is less than 0,95, one more set of specimens shall be tested at an exposure temperature different from the temperature originally chosen.

NOTE ISO 2578 is based on the principles laid down in IEC 60216. By deleting all information that is not required for planning and running a temperature index experiment and for calculation of results, ISO 2578 has become a practical short version as required for use in a laboratory.

6.5.10.2 Result

For each test criterion, the kind of preparation and the type and dimensions of the specimen, the number of specimens for each test, the exposure temperatures, and the results shall be reported along with reference to the standards applied. The results for each test set shall contain the specimen end-point times, the time to end-point for each exposure temperature, a graph showing the property values as a function of the logarithm of the times to end-point, the thermal endurance graph (first order regression line) on thermal endurance graph paper, the temperature index, and the correlation coefficient.

6.6 Chemical properties

6.6.1 Water absorption

Method 1 (water at 23 °C) and method 3 (boiling water) given in ISO 62 shall be used. For each method three specimens shall be tested. The kind of preparation and the dimensions of the specimen, and the three results of water absorption obtained with each of method 1 and method 3 shall be reported. One untreated specimen shall be kept for reference.

6.6.2 Effect of liquid chemicals

The method given in ISO 175 shall be used. Unless otherwise specified, the temperature of the test liquid shall be (23 ± 2) °C and the immersion time shall be (168 ± 1) h (seven days). For each test liquid three specimens shall be tested. The kind of preparation and the dimensions of

the specimen, the type of test liquid and the three results for each of the test liquids shall be reported. For each test liquid the result shall contain the change of appearance, dimensions and mass for each of the three specimens. One untreated specimen shall be kept for reference.

6.6.3 Resistance to mould growth

The method given in IEC 60068-2-10 shall be used. Three specimens according to 6.7.3.3 shall be tested, and the three results of resistance to mould growth shall be reported. One untreated specimen shall be kept for reference.

6.6.4 Water vapour permeability

To be agreed between supplier and purchaser.

6.7 Electrical properties

6.7.1 Effect of water immersion on volume resistivity

~~6.7.1.1 General~~

~~The method given in IEC 60093 shall be used. If IEC 60093 is not applicable for the material under test, then the following method may be used.~~

~~NOTE—IEC 60093 will be replaced by IEC 62631 in the future. Users are encouraged to investigate the status of standardisation of IEC 62631 and the possibility of applying the standard. Members of ISO and IEC maintain registers of currently valid International Standards.~~

~~6.7.1.2 Equipment~~

~~The following equipment shall be used:~~

- ~~• any commercially available tera-ohmmeter with an accuracy of $\pm 10\%$;~~
- ~~• a metal cylinder to be used as voltage electrode (top electrode) of at least 60 mm diameter having a mass to provide a pressure on the specimen of about 0,015 MPa;~~
- ~~• two conducting rubber disks having the same diameter as the top electrode and of 3 mm to 5 mm thickness with a maximum resistance of 1 000 Ω , and with a Shore A hardness of 65 to 85;~~
- ~~• a metal cylinder having the same diameter as the top electrode and of about 70 mm height;~~
- ~~• (a bottom electrode).~~

~~6.7.1.3 Test specimen~~

~~The specimen shall be in the form of a disk or a square with a diameter or an edge length exceeding the diameter of the top electrode by at least 10 mm. The thickness shall not exceed 3 mm and the flat surfaces shall be in parallel. Three specimens shall be prepared.~~

~~NOTE—The specimen can be cast between plates, with a wound piece of enamelled round winding wire used as a spacer.~~

~~6.7.1.4 Procedure~~

~~The test set-up shall consist of the specimen placed between the two metal cylinders with the rubber disks as intervening layers. For an example of the complete test arrangement, see Figure 1. The d.c. test voltage shall be adjusted to provide an electrical field strength of not more than 1 000 V/mm. The specimen shall be tested before and after immersion in demineralized water. Unless otherwise specified, the temperature of the water shall be $(23 \pm 2)^\circ\text{C}$ and the time of immersion shall be (168 ± 1) h (seven days). After immersion in water the test set-up shall be made immediately after removing the specimen from the water and blotting it between filter papers to remove excessive water. The resistance measurement shall be taken (15 ± 1) min after the test set-up is made. The reading shall be taken (60 ± 5) s after electrification. In the~~

~~case where, for example, the diameter of the top electrode is 60 mm, resistivity shall be calculated as:~~

$$\rho = (2,83 - R)/d$$

~~where~~

~~ρ is the resistivity (Ωm);~~

~~d is the specimen thickness (mm);~~

~~R is the measured resistance (Ω).~~

~~For a different diameter D of the top electrode, replace the factor 2,83 by:~~

~~$2,83 D^2 / 3\,600$ with D in millimetres.~~

6.7.1.5 Result

~~Three specimens shall be tested and the kind of preparation, the diameter of the electrodes, the dimensions of the test specimen, the test voltage used, and the three results before and after immersion in water shall be reported, along with reference to the standard applied. The results shall contain volume resistance and volume resistivity.~~

A method given in the IEC 62631-3 series shall be used. Three specimens shall be tested.

6.7.2 Dielectric dissipation factor ($\tan \delta$) and relative permittivity (ϵ_r)

6.7.2.1 General

~~The method given in IEC 60250 shall be used. If IEC 60250 is not applicable for the material under test, then the following method may be used.~~

~~NOTE IEC 60250 will be replaced by IEC 62631 in the future. Users are encouraged to investigate the status of standardisation of IEC 62631 and the possibility of applying the standard. Members of ISO and IEC maintain registers of currently valid International Standards.~~

6.7.2.2 Equipment

~~Any commercially available and adequate impedance meter may be used, indicating the dielectric dissipation factor ($\tan \delta$) and the relative permittivity (ϵ_r).~~

6.7.2.3 Test specimen

~~A test specimen in accordance with 6.7.1.3 shall be used.~~

6.7.2.4 Procedure

~~The top electrode shall have a diameter of at least 40 mm and may or may not be surrounded by a shield electrode. The bottom electrode shall have a diameter exceeding the diameter of the top electrode by at least 20 mm and shall be applied concentrically to the upper electrode. The electrodes shall be provided by brushing a conductive dispersion such as graphite or silver, or by applying a metal foil of a thickness of not more than 0,005 mm, made to adhere with a drop of oil, or by any other equally suitable procedure. Unless otherwise specified, the test shall be carried out at $(23 \pm 2)^\circ\text{C}$ with a sinusoidal test voltage at a frequency of 1 kHz. The connections to the specimen shall be in accordance with the instruction manual of the testing device.~~

6.7.2.5 Result

~~Two specimens shall be tested. The kind of preparation and the dimensions of the test specimen, the test temperature, the electrodes employed, the test voltage and frequency used,~~

~~and the two results shall be reported along with reference to the standard applied. The report shall contain the dielectric dissipation factor and the relative permittivity.~~

The method given in IEC 62631-2-1 or IEC 62631-2-2 shall be used. Two specimens shall be tested.

6.7.3 Break down voltage and electric strength

6.7.3.1 General

Breakdown voltage shall be measured by using IEC 60243-1. If IEC 60243-1 is not applicable to the material under test, Clauses 4 and 6 may be amended as specified in 6.7.3.2.

6.7.3.2 Electrodes

The electrode arrangement shall be the ball-to-plate type. The high-voltage electrode shall consist of a polished steel ball with a radius of $(3 \pm 0,0005)$ mm for rigid material, and $(10 \pm 0,0005)$ mm for flexible material. Polished steel balls with a surface roughness of less than 0,001 mm as used in ball bearings (class III) are easily available and have been found adequate for the purpose. The earth electrode shall be a plate with a diameter of (75 ± 1) mm and with rounded edge of a radius of $(3 \pm 0,1)$ mm. For the complete test arrangement for flexible material, see Figure 2. In the case of rigid material, the upper electrode and the specimen shall be as shown in Figure 3.

NOTE 1 The ball-to-plate electrode arrangement gives, compared to a plate-to-plate set, a slightly increased field strength depending on the radius of the ball electrode and the thickness of the specimen.

EXAMPLE For a radius of 10 mm and a thickness of 0,1 mm, the increase in field strength compared to that of plate-to-plate arrangement is about 10 %.

NOTE 2 If a round cylindrical glass container of sufficient size is used to accommodate the test set-up and the fluid with the earth electrode at the bottom of it, such a container makes it possible to observe visually the process when the voltage is applied. It also permits the earth connection and the fluid supply through the bottom electrode, with a fluid overflow at the top of the container, see Figure 2 Figure 4. If an elevated test temperature is required, such an arrangement allows the fluid to be used for heating purposes.

6.7.3.3 Test specimen

6.7.3.3.1 General

The thickness of that part of the specimen which is subject to breakdown shall not exceed 1 mm. The thickness of any two of the specimens of one set shall not vary by more than 10 %.

NOTE It is generally found that the electric strength for cured reactive compounds with a glass transition temperature above 80 °C is from 50 kV/mm to 100 kV/mm and can be even more, for instance, for hot curing cycloaliphatic epoxide-based compounds. Consequently, testing specimens of a thickness of significantly more than 1 mm and with an electrode arrangement of, for instance, 25/75 mm as specified in IEC 60243-1, can require voltage levels above 200 kV. This can lead to conditions where flashover or partial flashover with subsequent breakdown outside the electrode area cannot be avoided.

6.7.3.3.2 Rigid material

The specimen shall form a cylindrical rod of cast compound with a diameter of about 30 mm and with a length, in millimetres, twice the assumed numerical value of breakdown voltage in kilovolts. This rod shall contain a central lead wire with a steel ball attached to it at one end and apart from the other end completely embedded in the casting compound.

After the mould is removed, that end of the specimen which is close to the ball electrode shall be ground to the specified thickness, then polished and coated with a conductive layer, for instance a dispersion of graphite or silver, which serves as the earth electrode. During grinding the thickness shall be controlled by means of a permeameter type device calibrated in thickness. For an example of the specimen set-up, see Figure 4. This set-up can be accommodated in a glass container as in Figure 2 Figure 4.

NOTE For casting, a glass tube can serve as a mould with the lead wire and the ball electrode properly centred by adequate means. A piece of welding wire of, for instance, 3 mm diameter can serve as a lead wire, with one end soldered to the ball electrode.

After testing, the cured compound is removed at the point of breakdown to allow measurement of the space between the polished surface and the ball electrode. The space shall be measured by means of a micrometre probe and reported as thickness.

6.7.3.3.3 Flexible material

A specimen in accordance with 6.7.3.3 shall be used.

6.7.3.4 Procedure

The rate of increase of the voltage shall be not more than 500 V/s. Unless otherwise specified, the test temperature shall be $(23 \pm 2) ^\circ\text{C}$. The test shall be carried out with the specimen and the electrodes in a dielectric fluid, which is circulated and maintained at the specified test temperature. Unless otherwise specified, unused mineral insulating oil according to IEC 60296, or unused synthetic organic ester according to IEC 61099, shall be employed.

6.7.3.5 Result

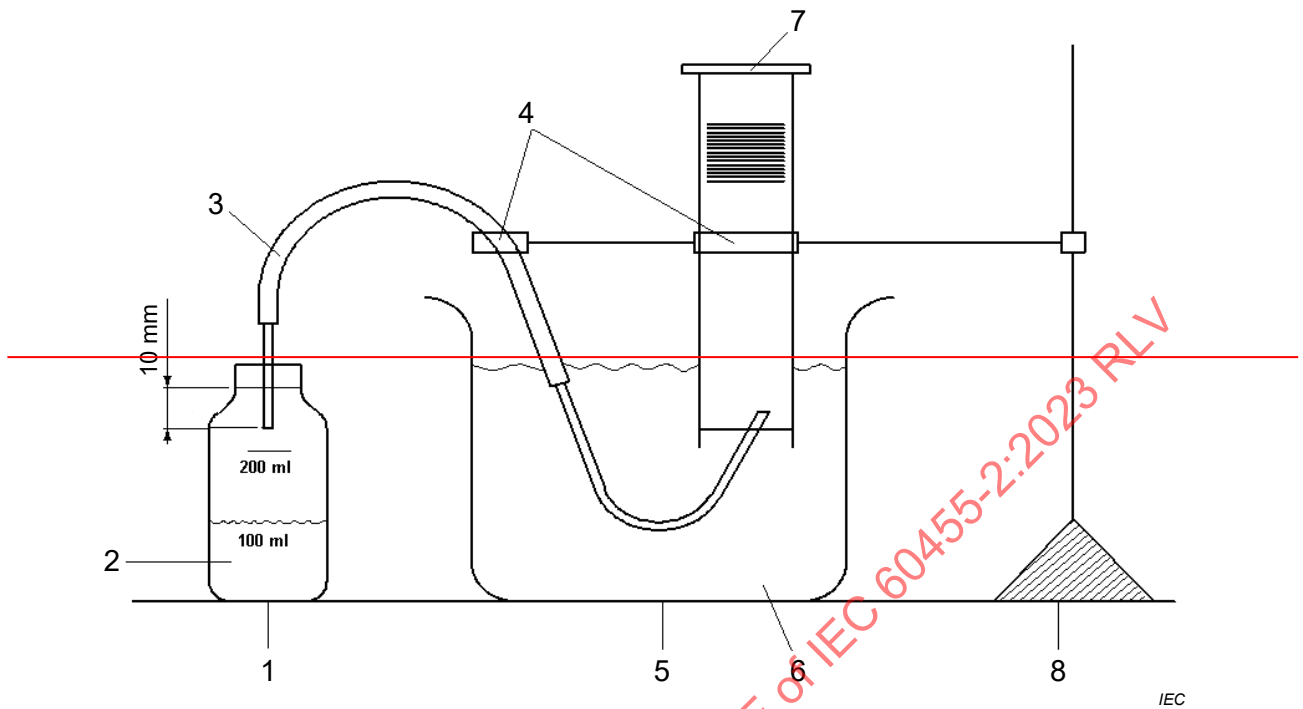
Five specimens shall be tested. The type and method of preparation of the specimen and its dimensions, the test temperature, the radius of the ball electrode, the type of dielectric fluid used, and the five results shall be reported, along with reference to the standards applied. The results shall contain the thickness of the specimen at the point of breakdown, the breakdown voltage and the electric strength.

6.7.4 Proof tracking index (PTI)

The method given in Clause 10 of IEC 60112:2003 shall be used. Three specimens shall be tested with a proof voltage as specified in the relevant specification sheet, or as agreed upon between supplier and purchaser. The kind of preparation and the dimensions of the specimen, as well as the three results of PTI, shall be reported. The results shall contain the proof voltage applied and the number of drops obtained.

6.7.5 Electrolytic corrosion

The visual method given in IEC 60426 shall be used. Three specimens shall be tested. The three results of electrolytic corrosion shall be reported.



- | | |
|------------------------|-----------------------|
| 1. spray bottle | 5. water basin |
| 2. demineralized water | 6. tap water |
| 3. flexible tube | 7. measuring cylinder |
| 4. fixture | 8. stand |

Figure 1 — Test apparatus for curing in presence of water test

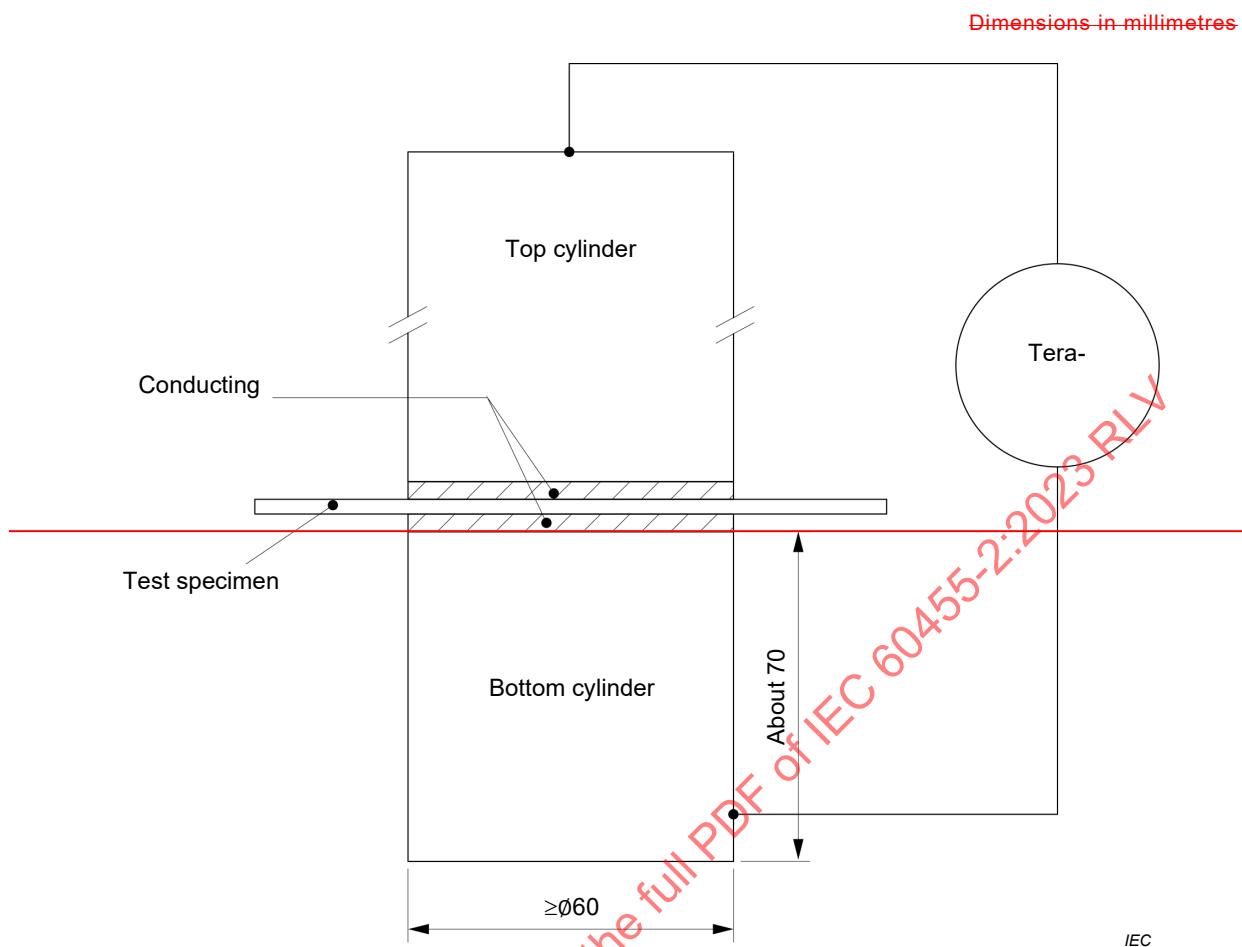


Figure 2 – Test set-up for volume resistivity

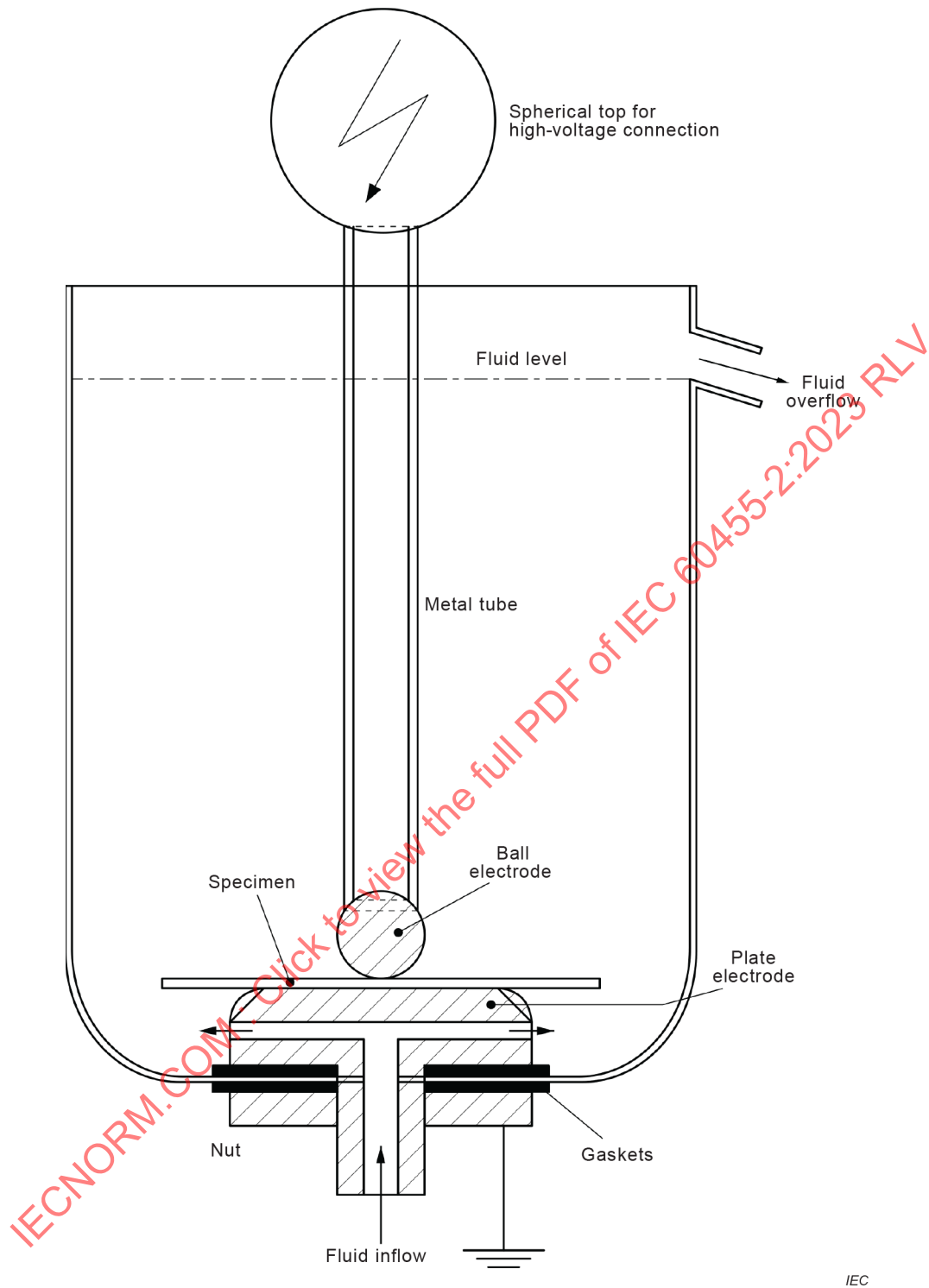


Figure 3 – Example of electrode arrangement for flexible cured compound

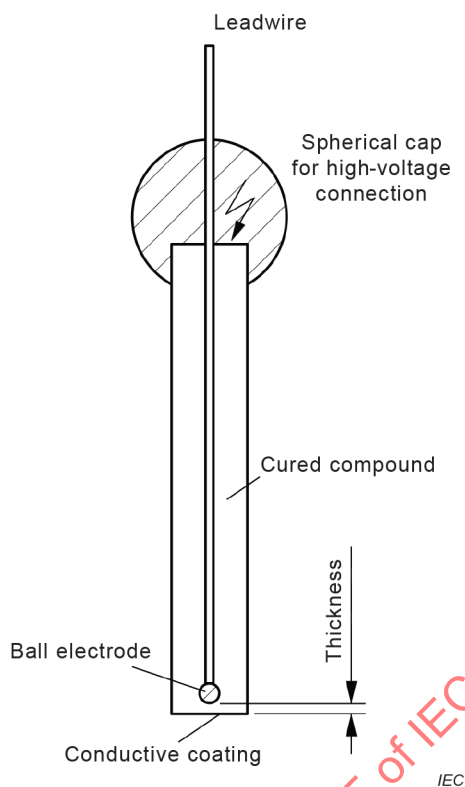


Figure 4 – Example of electrode arrangement for rigid cured compound

Annex A (informative)

Health and safety

The manufacturer shall make available on request the MSDS for each component, in order to enable the purchaser to transport, use and dispose of the components and their packaging in a safe manner, in relevant languages. Further relevant information concerning health and safety linked to installation conditions shall be made available on request.

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Bibliography

- [1] ISO 558:1980, *Conditioning and testing – Standard atmospheres – Definitions*
 - [2] ISO 2578:1993, *Plastics – Determination of time-temperature limits after prolonged exposure to heat*
-

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INTERNATIONAL STANDARD

NORME INTERNATIONALE

**Resin based reactive compounds used for electrical insulation –
Part 2: Methods of test**

**Composés réactifs à base de résines utilisés comme isolants électriques –
Partie 2: Méthodes d'essai**

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CONTENTS

FOREWORD.....	5
INTRODUCTION.....	7
1 Scope.....	8
2 Normative references	8
3 Terms and definitions	11
4 General notes on methods of test	11
4.1 Preparation and conditioning.....	11
4.2 Sequence of tests	12
4.3 Test report	12
5 Methods of test for reactive compounds and their components	12
5.1 Flash point.....	12
5.2 Density	12
5.3 Viscosity	12
5.4 Viscosity after storing at elevated temperature.....	12
5.5 Content of volatile organic components.....	13
5.6 Isothermal increase of viscosity (processing time)	13
5.7 Shelf life	13
5.8 Colour.....	13
5.9 Softening temperature	14
5.10 Ash content.....	14
5.11 Filler content.....	14
5.12 Chlorine content.....	14
5.12.1 Total chlorine content of unsaturated polyesters and epoxide resins.....	14
5.12.2 Inorganic chlorine content of epoxide resins and glycidyl esters	14
5.12.3 Easily saponifiable chlorine content of epoxide resins and related materials	14
5.13 Tendency of crystallisation.....	14
5.14 Epoxide equivalent of epoxide resins	14
5.15 Content of isocyanate	14
5.16 Water content (Karl Fischer method).....	14
5.17 Hydroxyl value	15
5.17.1 Polyester resins.....	15
5.17.2 Resins other than polyester	15
5.18 Acid value of polyester resins	15
5.19 Amount of double bonds of unsaturated polyester and acrylate resins	15
5.20 Acid and acid-anhydride content of acid-anhydride hardeners.....	15
5.21 Amine value	15
5.22 Pot life	15
5.22.1 General	15
5.22.2 Resinous compounds for cable accessories.....	15
5.23 Gel time	16
5.23.1 Unsaturated polyester based compounds	16
5.23.2 Phenolic resin based compounds.....	16
5.23.3 Other compounds	16
5.24 Exothermic temperature rise	16
5.24.1 Unsaturated polyester based compounds	16
5.24.2 Resinous compounds for cable accessories.....	16

5.25	Total volume shrinkage of epoxide and unsaturated polyester based compounds	17
5.26	Curing in presence of water	17
5.26.1	General	17
5.26.2	Apparatus and materials	17
5.26.3	Pouring device	17
5.26.4	Procedure	17
5.26.5	Test report	18
5.27	Determination of the degree of curing	19
5.28	Curing in thick layer and emissions during curing	19
5.28.1	General	19
5.28.2	Equipment	20
5.28.3	Test specimen	20
5.28.4	Procedure	20
6	Methods of test for cured reactive compounds	21
6.1	General	21
6.2	Test specimens	21
6.2.1	General	21
6.2.2	Preparation of the reactive compound	21
6.2.3	Preparation of test specimens	22
6.2.4	Type and number of test specimens	22
6.3	Density	22
6.4	Mechanical properties	22
6.4.1	Tensile properties	22
6.4.2	Compressive properties	22
6.4.3	Flexural properties	23
6.4.4	Impact strength	23
6.4.5	Hardness	23
6.5	Thermal properties	23
6.5.1	Bond strength at elevated temperature	23
6.5.2	Linear thermal expansion	23
6.5.3	Thermal conductivity	23
6.5.4	Glass transition	24
6.5.5	Flammability	24
6.5.6	Thermal shock	24
6.5.7	Dry heat resistance of resins for cable accessories – Method of test	24
6.5.8	Wet heat resistance of resins for cable accessories	25
6.5.9	Loss of mass	27
6.5.10	Temperature index	28
6.6	Chemical properties	29
6.6.1	Water absorption	29
6.6.2	Effect of liquid chemicals	29
6.6.3	Resistance to mould growth	29
6.6.4	Water vapour permeability	29
6.7	Electrical properties	29
6.7.1	Effect of water immersion on volume resistivity	29
6.7.2	Dielectric dissipation factor ($\tan \delta$) and relative permittivity	29
6.7.3	Break down voltage and electric strength	29
6.7.4	Proof tracking index (PTI)	31

6.7.5 Electrolytic corrosion	31
Annex A (informative) Health and safety	34
Bibliography	35
Figure 1 – Examination grid	18
Figure 2 – Position of examination grid on the specimen.....	19
Figure 3 – Example of electrode arrangement for flexible cured compound	32
Figure 4 – Example of electrode arrangement for rigid cured compound	33
Table 1 – Condition of the top side.....	20
Table 2 – Condition of the bottom side.....	20
Table 3 – Condition of the interior	20
Table 4 – Voids.....	21

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**RESIN BASED REACTIVE COMPOUNDS USED
FOR ELECTRICAL INSULATION –****Part 2: Methods of test****FOREWORD**

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IEC 60455-2 has been prepared by IEC technical committee 15: Solid electrical insulating materials. It is an International Standard.

This fourth edition cancels and replaces the third edition published in 2015. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) Introduction of test methods related to IEC 60455-3-8;
- b) Additional and updated test methods for resins.

The text of this International Standard is based on the following documents:

Draft	Report on voting
15/1006/FDIS	15/1015/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

A list of all parts in the IEC 60455 series, published under the general title *Resin based reactive compounds used for electrical insulation*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

This part of IEC 60455 is one of a series which deals with solvent-free resin based reactive compounds and their components used for electrical insulation.

The series consists of three parts:

- Part 1: Definitions and general requirements;
- Part 2: Methods of test;
- Part 3: Specifications for individual materials.

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RESIN BASED REACTIVE COMPOUNDS USED FOR ELECTRICAL INSULATION –

Part 2: Methods of test

1 Scope

This part of IEC 60455 specifies methods of test to be used for testing resin based reactive compounds, their components and cured compounds used for electrical insulation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050 (all parts), *International Electrotechnical Vocabulary* (available at <http://www.electropedia.org>)

IEC 60068-2-10:2005, *Environmental testing – Part 2-10: Tests – Test J and guidance: Mould growth*

IEC 60112:2020, *Method for the determination of the proof and the comparative tracking indices of solid insulating materials*

IEC 60216 (all parts), *Electrical insulating materials – Thermal endurance properties*

IEC 60243-1:2013, *Electric strength of insulating materials – Test methods – Part 1: Tests at power frequencies*

IEC 60296:2020, *Fluids for electrotechnical applications – Mineral insulating oils for electrical equipment*

IEC 60426:2007, *Electrical insulating materials – Determination of electrolytic corrosion caused by insulating materials – Test methods*

IEC 60455-1:1998, *Resin based reactive compounds used for electrical insulation – Part 1: Definitions and general requirements*

IEC 60455-3 (all parts), *Resin based reactive compounds used for electrical insulation – Part 3: Specifications for individual materials*

IEC 60455-3-8:2021, *Resin based reactive compounds used for electrical insulation – Part 3-8: Specifications for individual materials – Resins for cable accessories*

IEC 60695-11-10:2013, *Fire hazard testing – Part 11-10: Test flames – 50 W horizontal and vertical flame test methods*

IEC 60814:1997, *Insulating liquids – Oil-impregnated paper and pressboard – Determination of water by automatic coulometric Karl Fischer titration*

IEC 61033:1991, *Test methods for the determination of bond strength of impregnating agents to an enamelled wire substrate*

IEC 61099:2010, *Insulating liquids – Specifications for unused synthetic organic esters for electrical purposes*

ISO 37:2011, *Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties*

ISO 62:2008, *Plastics – Determination of water absorption*

ISO 75 (all parts), *Plastics and ebonite – Determination of temperature of deflection under load*

ISO 175:2010, *Plastics – Methods of test for the determination of the effects of immersion in liquid chemicals*

ISO 178:2010, *Plastics – Determination of flexural properties*

ISO 179-1:2010, *Plastics – Determination of Charpy impact properties – Part 1: Non-instrumented impact test*

ISO 179-2:1997, *Plastics – Determination of Charpy impact properties – Part 2: Instrumented impact test*

ISO 291, *Plastics – Standard atmospheres for conditioning and testing*

ISO 306:2013, *Plastics – Thermoplastic materials – Determination of Vicat softening temperature (VST)*

ISO 527 (all parts), *Plastics – Determination of tensile properties*

ISO 584:1982, *Plastics – Unsaturated polyester resins – Determination of reactivity at 80 degrees C (conventional method)*

ISO 604:2002, *Plastics – Determination of compressive properties*

ISO 868:2003, *Plastics and ebonite – Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1183-1:2019, *Plastics – Methods for determining the density of non-cellular plastics – Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1513:2010, *Paints and varnishes – Examination and preparation of test samples*

ISO 1523:2002, *Determination of flash point – Closed cup equilibrium method*

ISO 1675:1985, *Plastics – Liquid resins – Determination of density by the pycnometer method*

ISO 2039-1:1993, *Plastics – Determination of hardness – Part 1: Ball indentation method*

ISO 2114:2000, *Plastics (polyester resins) and paints and varnishes (binders) – Determination of partial acid value and total acid value*

ISO 2431:1993, *Paints and varnishes – Determination of flow time by use of flow cups*

ISO 2535:1997, *Plastics – Unsaturated polyester resins – Measurement of gel time at 25 degrees C*

ISO 2554:1997, *Plastics – Unsaturated polyester resins – Determination of hydroxyl value*

ISO 2555:1989, *Plastics – Resins in the liquid state or as emulsions or dispersions – Determination of apparent viscosity using a single cylinder type rotational viscometer method*

ISO 2592:1973, *Petroleum products – Determination of flash and fire points – Cleveland open cup method*

ISO 3001:1997, *Plastics – Epoxy compounds – Determination of epoxy equivalent*

ISO 3219:1993, *Plastics – Polymers/resins in the liquid state or as emulsions or dispersions – Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 3451-1:1997, *Plastics – Determination of ash – Part 1: General methods*

ISO 3521:1997, *Plastics – Unsaturated polyester and epoxy resins – Determination of overall volume shrinkage*

ISO 3679:1983, *Paints, varnishes, petroleum and related products – Determination of flashpoint – Rapid equilibrium method*

ISO 4573:1978, *Plastics – Epoxide resins and glycidyl esters – Determination of inorganic chlorine*

ISO 4583:1998, *Plastics – Epoxide resins and related materials – Determination of easily saponifiable chlorine*

ISO 4615:1979, *Plastics – Unsaturated polyesters and epoxide resins – Determination of total chlorine content*

ISO 4625:1980, *Binders for paints and varnishes – Determination of softening point – Ring-and-ball method*

ISO 4895, *Plastics – Liquid epoxy resins – Determination of tendency to crystallize*

ISO 7056, *Plastics laboratory ware – Beakers*

ISO 9396:1997, *Plastics – Phenolic resins – Determination of the gel time of resols under specific conditions using automatic apparatus*

ISO 11357-2:1999, *Plastics – Differential scanning calorimetry (DSC) – Part 2: Determination of glass transition temperature and step height*

ISO 11359-2:1999, *Plastics – Thermomechanical analysis (TMA) – Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

ISO 11359-3:2002, *Plastics – Thermomechanical analysis (TMA) – Part 3: Determination of penetration temperature*

ISO 14896:2009, *Plastics – Polyurethane raw materials – Determination of isocyanate content*

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes – Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60455-1 and IEC 60050 and the following shall apply.

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

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

3.1

volume resistance

part of the insulation resistance which is due to conduction through the volume and excluding surface current

3.2

volume resistivity

volume resistance reduced to a cubical unit volume

3.3

dielectric dissipation factor

$\tan \delta$

numerical value of the ratio of the imaginary to the real part of the complex permittivity

3.4

relative permittivity

ϵ_r

ratio of the absolute permittivity to the electric constant

Note 1 to entry: In practical engineering, it is usual to employ the term 'permittivity' when referring to relative permittivity

4 General notes on methods of test

4.1 Preparation and conditioning

Unless otherwise specified in the relevant specification standard or in the method of test, all tests shall be carried out at atmospheric conditions in a temperature range of between 21 °C and 29 °C and a relative humidity range of between 45 % and 70 %. Before measurements are made, the sample or test specimen shall be pre-conditioned under these atmospheric conditions for a time sufficient to allow the sample or the test specimen to reach stability. For taking samples in liquid or paste form, ISO 15528 shall be applied. For preparation of such samples for testing, ISO 1513 shall be applied.

NOTE For definitions of terms for standard atmospheres, see ISO 558. The test atmosphere as specified above does not comply with any of the two standard atmospheres as specified in ISO 291 but covers both ranges inclusive of their tolerances

Normally, all requirements for a method of test are given in the description, and diagrams are intended only to illustrate one possible arrangement for conducting the test. In case of inconsistencies between this document and the specification sheets of the IEC 60455-3 series, the latter shall prevail. When another standard is invoked for a test method, reference to that standard shall be included in the report.

4.2 Sequence of tests

To avoid unnecessary efforts, tests shall be carried out on the samples in the following sequence:

- 1) tests on individual components prior to mixing;
- 2) tests on reactive compound just after mixing (ready to use);
- 3) tests on cured compound;
- 4) tests on cured compound after pre treatment (thermal, humidity, water etc.).

If the sample under test fails a test the following tests may become obsolete.

4.3 Test report

If not otherwise specified, the test report shall include the following data:

- 1) resin designation and identification;
- 2) lot number or other identification;
- 3) confirmation of marking and labelling according to the material safety data sheet (MSDS);
- 4) test results;
- 5) major test parameters, including conditioning and calibration, if any;
- 6) processing conditions used to reactive compound;
- 7) copy of the technical data sheet (TDS) and (MSDS).

5 Methods of test for reactive compounds and their components

5.1 Flash point

For flash point temperatures of 79 °C and above, the method given in ISO 2592 shall be used. For flash point temperatures below 79 °C, the method given in ISO 1523 shall be used with any of the closed-cup apparatus as described in Annex A of ISO 1523:2002. ISO 1523 shall be read in conjunction with ISO 3679. Two measurements shall be made on two separate samples, and the two results of the flash point shall be reported along with reference to the standards applied.

5.2 Density

The method given in ISO 1675 shall be used. Two measurements shall be made, and the two results of the density shall be reported.

5.3 Viscosity

The viscosity shall be determined with a suitable device at $(23 \pm 0,5)$ °C if not otherwise specified. If a rotating type of device is used, it shall be in accordance with ISO 2555 (Brookfield type) or with ISO 3219 (a type working at a defined shear rate). If an efflux type of equipment is used, the method of test and the flow cup shall be in accordance with ISO 2431. Two measurements shall be made, and the two results of the viscosity shall be reported, along with reference to the standards applied.

5.4 Viscosity after storing at elevated temperature

This method is not applicable to one-component systems or components containing hardener.

If not otherwise specified, a sample of sufficient amount is stored for $(20 \pm 0,5)$ h at a temperature of (100 ± 3) °C in a sealed container. After cooling down to room temperature the viscosity is measured according to 5.3. The increase of viscosity is calculated using the following equation:

Increase of viscosity in % = $(\eta_2 - \eta_1) \times 100 / \eta_1$

where

η_1 dynamic viscosity before storing

η_2 dynamic viscosity after storing

5.5 Content of volatile organic components

This method is not applicable to one-component systems or components containing hardener. If not otherwise specified, the test shall be carried out in the following way:

The mass of an empty weighing bottle (about 80 mm × 30 mm) is taken to 0,001 g (m_1). A mass of 0,4 g to 0,5 g resin (m_2) is weighed to 0,001 g into the weighing bottle (well closed during weighing). Some drops of toluene are added to dilute the resin. The liquid is spread on the floor of the weighing bottle with a slight twist.

The open weighing bottle is placed into an oven with forced air circulation for at least 2 h at $(110 \pm 2) ^\circ\text{C}$. After cooling down to room temperature in a desiccator the weighing bottle is weighed again to 0,001 g (m_3).

Volatile organic components = $100 \times (m_2 - (m_3 - m_1)) / m_2$

where:

m_1 mass of an empty weighing bottle (g)

m_2 mass of resin before heating (g)

m_3 mass of an empty weighing bottle with resin after heating and cooling (g)

5.6 Isothermal increase of viscosity (processing time)

This method is designed for PUR and EP resins. For UP resins gel time shall be used. If not otherwise specified, the test shall be carried out in the following way:

All components and equipment shall be at room temperature. The components of the resin are mixed according to the manufacturer's instructions. The mixing procedure shall not take more than 3 min. The time measurement starts after adding and mixing of the last component. After 10 min the first viscosity measurement is taken as the initial value. The measurement is repeated until the specified maximum viscosity is reached. The time between the initial value and the maximum value is reported as processing time.

5.7 Shelf life

The shelf life shall be determined by measurement of the change in a specified characteristic property after a certain storage time and temperature. Experience has shown that viscosity according to 5.3 and gel time according to 5.23 are appropriate characteristics. To assess shelf life, viscosity and/or gel time shall be determined according to 5.3 and/or 5.23 respectively, at a temperature and with an end-point as agreed upon between supplier and purchaser. Two measurements shall be made on both fresh material and on material stored for a time and at a temperature as agreed between supplier and purchaser. The two results of shelf life shall be reported, along with reference to the standards applied. The results shall contain the viscosity and/or the gel time before and after storing, the storing time and temperature and the test temperature.

5.8 Colour

The method given in ISO 6271 shall be used. Two measurements shall be made, and the two results of colour shall be reported along with reference to the standard applied.

5.9 Softening temperature

The method given in ISO 306 or ISO 4625 shall be used. Two measurements shall be made, and the two results of softening temperature shall be reported along with reference to the standard applied.

5.10 Ash content

The method given in ISO 3451-1, method A, shall be used. Two measurements shall be made, and the two results of the ash content shall be reported.

5.11 Filler content

To be agreed between supplier and purchaser.

5.12 Chlorine content

5.12.1 Total chlorine content of unsaturated polyesters and epoxide resins

The method given in ISO 4615 shall be used. Two measurements shall be made, and the two results of the total chlorine content shall be reported.

5.12.2 Inorganic chlorine content of epoxide resins and glycidyl esters

The method given in ISO 4573 shall be used. Two measurements shall be made, and the two results of the inorganic chlorine content shall be reported.

5.12.3 Easily saponifiable chlorine content of epoxide resins and related materials

The method given in ISO 4583 shall be used. Two measurements shall be made, and the two results of the saponifiable chlorine content shall be reported.

5.13 Tendency of crystallisation

This method is applicable to epoxy resins only.

The method given in ISO 4895 shall be used. Two measurements shall be made, and the two results shall be reported.

5.14 Epoxide equivalent of epoxide resins

The method given in ISO 3001 shall be used. Two measurements shall be made, and the two results of the epoxide equivalent shall be reported.

5.15 Content of isocyanate

This method is applicable to polyurethane hardeners only.

The method given in ISO 14896 shall be used. Two measurements shall be made, and the two results shall be reported.

5.16 Water content (Karl Fischer method)

The method given in IEC 60814 shall be used. Two measurements shall be made, and the two results of the water content shall be reported.

5.17 Hydroxyl value

5.17.1 Polyester resins

The method given in ISO 2554 shall be used. Two measurements shall be made, and the two results of the hydroxyl value shall be reported.

5.17.2 Resins other than polyester

To be agreed between supplier and purchaser.

5.18 Acid value of polyester resins

The method given in ISO 2114 shall be used. Two measurements shall be made, and the two results of the acid value shall be reported.

5.19 Amount of double bonds of unsaturated polyester and acrylate resins

To be agreed between supplier and purchaser.

5.20 Acid and acid-anhydride content of acid-anhydride hardeners

The method given in ISO 2114 shall be used for acid content. The method given in ISO 7327 shall be used for anhydride content. Two measurements shall be made, and the two results of the acid value and/or anhydride content shall be reported.

5.21 Amine value

The method given in ISO 9702 shall be used if not otherwise agreed between supplier and purchaser.

5.22 Pot life

5.22.1 General

The time which a particular method requires to achieve a viscosity of 50 Pas is measured.

The viscosity can be determined in any viscosimeter.

5.22.2 Resinous compounds for cable accessories

5.22.2.1 Apparatus and materials

Equipment to be used:

- beaker in accordance with ISO 7056, polyethylene, polypropylene or glass with dimensions: 70 mm to 100 mm diameter and 70 mm to 130 mm high;
- environmental chamber;
- time measuring device;
- viscosimeter.

5.22.2.2 Preparation of the sample

The beaker shall be marked to a volume of (300 ± 25) ml.

The beaker and a standard manufacturer pack shall be stored in an environmental chamber for 24 h at the temperature specified with a tolerance of ± 2 K.

5.22.2.3 Procedure

The components shall be removed from the environmental chamber and start mixing immediately according to the supplier's instructions. The time starts at the commencement of mixing the components.

Remove the beaker from the environmental chamber, and pour the mixed compound into the beaker up to the mark previously made.

5.22.2.4 Test report

The test report shall include the following information:

- the time is determined from the start of mixing until a viscosity of 50 Pas is reached. The pot life shall be rounded to the nearest of a second;
- the viscosimeter, test parameters and system used (e.g. spindle size, rotation speed etc.).

The viscosimeter with the system used shall be calibrated at 50 Pas.

5.23 Gel time

5.23.1 Unsaturated polyester based compounds

Gel time is the period of time after which the reactive compound reaches the gel state. The method given in ISO 2535 shall be used at a test temperature as agreed upon between supplier and purchaser. Two measurements shall be made, and the two results of the gel time shall be reported along with the test temperature.

5.23.2 Phenolic resin based compounds

The method given in ISO 9396 shall be used. Two measurements shall be made, and the two results of the gel time shall be reported.

5.23.3 Other compounds

If applicable, the methods given in ISO 2535 or ISO 9396, or ISO 8987 shall be used.

5.24 Exothermic temperature rise

5.24.1 Unsaturated polyester based compounds

The method given in ISO 584 shall be used. Two measurements shall be made, and the two results of the exothermic temperature rise shall be reported.

5.24.2 Resinous compounds for cable accessories

5.24.2.1 Apparatus and materials

Equipment to be used:

- beaker in accordance with ISO 7056, polyethylene, polypropylene or glass with dimensions: 70 mm to 100 mm diameter and 70 mm to 130 mm high;
- thermocouple made with twisted wires;
- temperature recorder;
- time measurement device.

5.24.2.2 Preparation of the sample: Test at 23 °C

The beaker shall be marked to a volume of (300 ± 25) ml. The beaker and a sufficient amount of resinous compound components (prepare 400 ml to 700 ml of the compound) shall be stored for 24 h at (23 ± 2) °C.

The components shall be mixed according to manufacturer's instructions. The time starts at the commencement of mixing the components.

The mixed components shall be poured into the beaker up to the mark previously made. The thermocouple shall be inserted vertically in the centre of the mixture to a depth of (25 ± 5) mm under the compound surface and held in position by a suitable device (e.g. ring-stand).

5.24.2.3 Procedure

The temperature increase shall be checked, and the maximum temperature rise and the time to peak recorded. The test is finished after the highest temperature has been reached and the temperature starts to drop significantly.

NOTE For ease of testing, the temperature can be recorded as a curve.

5.24.2.4 Test report

The maximum temperature reached (rounded to the nearest °C) and the time to peak (rounded to the nearest min) shall be reported.

5.25 Total volume shrinkage of epoxide and unsaturated polyester based compounds

The method given in ISO 3521 shall be used. Two measurements shall be made, and the two results of the total volume shrinkage shall be reported. The report shall contain the test temperature, the density of the compound at test temperature, and the density of the specimen made of the cured compound.

5.26 Curing in presence of water

5.26.1 General

This test applies only to compound for cable accessories. This test is to measure the physical structure change when the polyurethane resinous compound cures in presence of water. Voids created by generation of gas in the resin may lead to electrical break down.

The density of the cured resin mentioned in the technical data sheet shall be $> 1,05 \text{ g/cm}^3$.

5.26.2 Apparatus and materials

Equipment to be used:

- 250 ml transparent/translucent PE squirt bottle;
- funnel that meets the requirements of 5.26.3;

NOTE Take care that the equipment is clean (no algae).

5.26.3 Pouring device

Use a funnel with a capability to fill the bottle within 30 s to 60 s at room temperature.

5.26.4 Procedure

The squirt bottle shall be marked with 2 marks at 100 ml and 200 ml respectively and filled up to the 100 ml mark with de-mineralized water of (23 ± 2) °C.

The squirt bottle and resinous compound left in its original packaging shall be allowed to reach room temperature. The compound package shall be opened, the stopwatch started, and the components mixed according to manufacturer's instructions. The mixed compound is poured into the closed funnel. The funnel shall be emptied immediately into the squirt bottle until the water level reaches the 200 ml mark. Care shall be taken to pour in the centre of the bottle and avoid touching of the inner bottle wall.

The compound shall be allowed to cure for the pot life specified by the manufacturer. At least one hour after the pot life has elapsed. The water shall be poured out of the squirt bottle. After hardening of the resin at room temperature for 24 h, the specimen shall be vertical cut through the centre of the bottle and clean the cut surface with compressed air. Examine the physical structure for the presence of blisters or cracks in the compound using the examination grid.

Examination grid

Evaluate concentration and dimension of bubbles created by using an examination grid. The grid measures 45 × 24 mm (width × height) and is made up of 15 cells of dimensions 9 × 8 mm, as shown in Figure 1.

5.26.5 Test report

The following parameters shall be measured and reported:

- physical structure/presence of blisters or cracks or bubbles and/or inclusions;
- picture of the cut surface of the compound with scale.

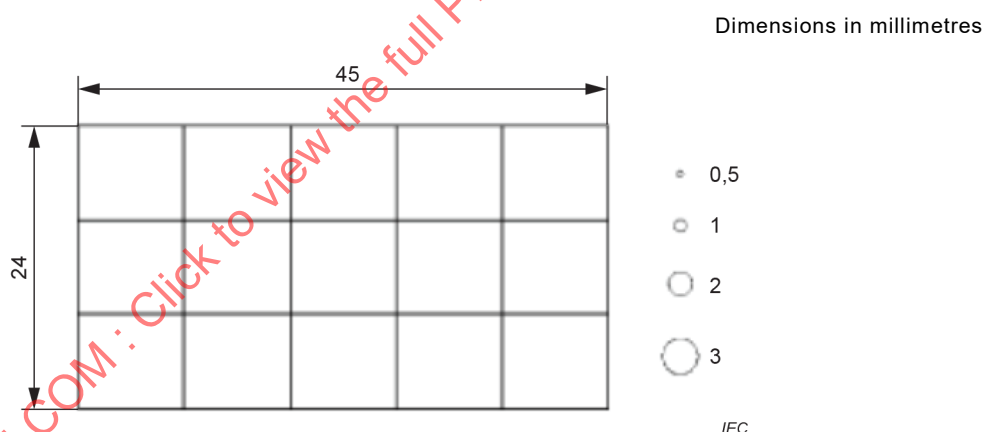
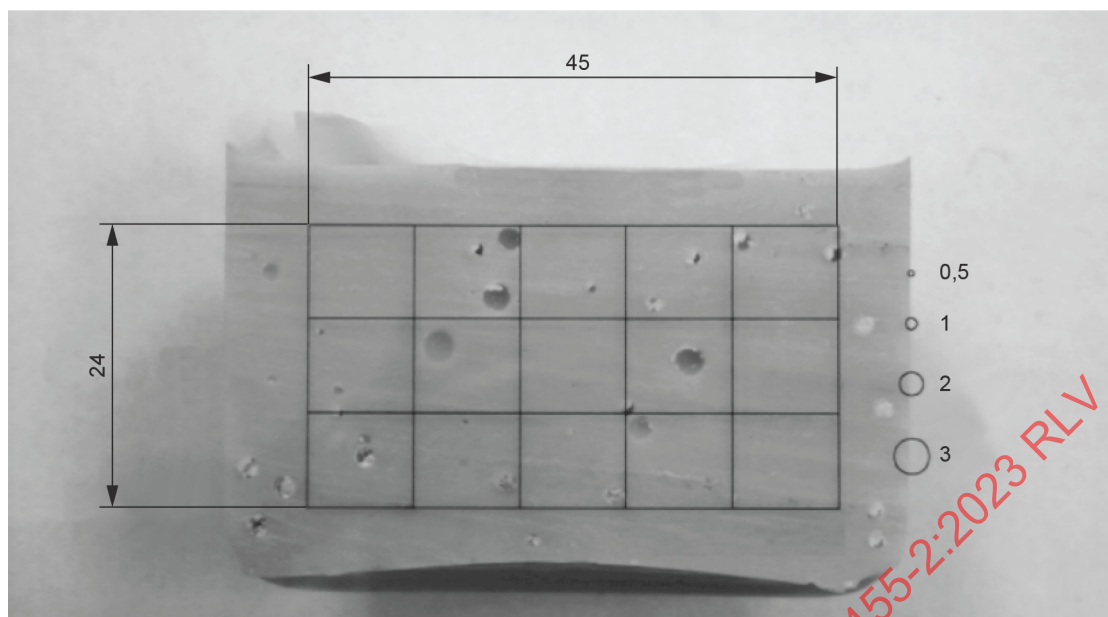


Figure 1 – Examination grid

Position the grid centrally on the cut surface. To pass the examination no bubbles > 3 mm are allowed and at least 5 cells must be free of bubbles. Count only bubbles with diameter ≥ 1 mm.



IEC

Figure 2 – Position of examination grid on the specimen

Figure 2 shows the position of the examination grid on the specimen.

5.27 Determination of the degree of curing

The reaction enthalpie of the uncured resin is determined by using DSC. An amount of 10 mg has been found adequate.

If not otherwise specified, about 10 g of resin is cured under conditions (temperature and time) as agreed between supplier and purchaser. The residual enthalpie is determined using a sample of about 10 mg.

The degree of curing is calculated as follows:

$$Doc = 100 - H_c \times 100 / H_u$$

where

Doc degree of curing (%)

H_c enthalpie of cured resin (J/g)

H_u enthalpie of uncured resin (J/g)

5.28 Curing in thick layer and emissions during curing

5.28.1 General

This test is designed for the examination of cured impregnating materials. Curing in thick layer is expressed by the condition after curing of the top side, bottom side and interior of the specimen. The determination of emissions during the curing process is also part of this method.

5.28.2 Equipment

The following equipment shall be used:

- flat and smooth square pieces of aluminium foil, 0,1 mm to 0,15 mm in thickness and of (95 ± 1) mm side length;
- a former made of metal or any other suitable solid material, (25 ± 1) mm in thickness and (45 ± 1) mm square;
- an oven with forced air circulation and with a minimum rate of 8 fresh air changes per hour. The oven shall be of the type and design used for drying and/or curing specimens;
- a balance with an accuracy of 0,01 g.

5.28.3 Test specimen

A piece of aluminium foil shall be cleaned by adequate means and shall be folded around the former to produce a square mould of about 45 mm side length. The square mould shall be dried at (110 ± 5) °C for (10 ± 1) min, cooled down and stored in a desiccator.

The mass of the square mould shall be weighed to 0,01 g (m_1). A resin sample with a mass between 9,90 g and of 10,10 g shall be weighed to 0,01 g into the mould (m_2).

The specimen shall be cured at a temperature and for a time as agreed between supplier and purchaser. The specimen shall be cooled down to room temperature in a desiccator and the mass shall be taken to 0,01 g (m_3). Subsequently the aluminium foil is removed.

5.28.4 Procedure

5.28.4.1 Description of the specimen

Table 1, Table 2, Table 3 and Table 4 give descriptions of the specimen.

Table 1 – Condition of the top side

Condition	Symbol
Smooth	S1
Wrinkled	S2

Table 2 – Condition of the bottom side

Condition	Symbol
Non-tacky	U1
Tacky	U2

Table 3 – Condition of the interior

Condition	Symbol
Rigid	I1.X
Horn-like machinable	I2.X
Leather-like	I3.X
Rubber-like	I4.X
Gel-like	I5.X
Liquid	I6.X

Table 4 – Voids

The specimen contains	X
No voids	1
Not more than five voids	2
More than five voids	3

For the condition of the interior a statement shall be added whether the interior is uniform or not uniform.

NOTE It can be necessary to cut the specimen, to bend the specimen with the fingers or to use a knife for describing the mechanical properties.

5.28.4.2 Emissions

The emissions shall be calculated by the following equation:

$$E = 100 - ((m_2 - (m_3 - m_1)) \times 100 / m_2)$$

where:

E emissions

m_1 mass of the empty square mould

m_2 mass of the uncured resin (without mass of the square mould)

m_3 mass of the square mould with cured resin

6 Methods of test for cured reactive compounds

6.1 General

The cured compound is self-supporting and thus allows the preparation of rigid and flexible test specimens.

6.2 Test specimens

6.2.1 General

When the term "test specimen" is used it means solid parts of the cured material in a shape as required for the method of test concerned. In 6.2.2, 6.2.3 and 6.2.4 such "test specimens" are referred to as "specimens".

6.2.2 Preparation of the reactive compound

The reactive compound shall be a homogeneous mixture of such portions of the components as specified by the supplier. Also drying, de-aerating, heating, and other measures to treat the components and the compound shall comply with the instructions given by the supplier. When compounds contain fillers, the possibility of settlement needs to be taken into account.

6.2.3 Preparation of test specimens

Specimens shall be prepared under conditions as specified in the particular method of test in the relevant specification sheet of IEC 60455-3, or as agreed upon between supplier and purchaser. This includes the casting process with respect to temperature and vacuum, the curing conditions with respect to temperature, and time or temperature-time programme, the removal from the mould and annealing and cooling. Reactive compounds, which according to the instructions given by the supplier, cure at ambient temperature, generally reach the final state at ambient temperature only after days or weeks. To achieve a defined degree of cure in such conditions, the compounds shall be cured for 24 h at ambient temperature and immediately thereafter for 24 h at 80 °C, or as agreed between supplier and purchaser. Specimens shall be cast in the proper shape according to the dimensions given in the method of test or shall be prepared from cast pieces. They shall be free of voids, bubbles, nicks, and scratches. During machining, excessive heating of the machined surfaces shall be avoided by cooling, for instance with water.

NOTE Removal of the cured compound from the mould is facilitated by the use of release agents and moulds made of chromium-plated or other adequate material.

6.2.4 Type and number of test specimens

The type and number of specimens required for a particular method of test are specified in the method of test, in the relevant specification sheet of IEC 60455-3 or shall be agreed upon between supplier and purchaser.

6.3 Density

A method given in ISO 1183-1 shall be used. Two measurements shall be made. The kind of preparation and the dimensions of the specimen, the method used, and the two results of the density shall be reported.

6.4 Mechanical properties

6.4.1 Tensile properties

6.4.1.1 Rigid material

The method given in ISO 527 shall be used with a speed of testing to break the specimen within (60 ± 15) s. The type of specimen shall be selected from ISO 527. Five specimens shall be tested. The kind of preparation, the dimensions and the type of the specimen, the speed of testing, and the five results of the tensile properties shall be reported. As far as applicable, the report shall contain tensile stress at yield, at maximum load and at break, percentage elongation at yield and at break, and modulus of elasticity.

6.4.1.2 Flexible material

The method given in ISO 37 shall be used for a dumb-bell specimen. Five specimens shall be tested. The kind of preparation and the type of dumb-bell specimen, and the five results of the tensile properties shall be reported. The report shall contain tensile strength, percentage elongation at break, and modulus of elasticity.

6.4.2 Compressive properties

The method given in ISO 604 shall be used. Five specimens shall be tested. The kind of preparation and the dimensions of the specimen, the rate of deformation, and the five results of the compressive properties shall be reported. As far as applicable, the report shall contain compressive strength at maximum load, compressive yield stress, and percentage compressive strain at rupture.

6.4.3 Flexural properties

The method given in ISO 178 shall be used with a relative rate of movement of the loading nose and supports, so that the specimen ruptures or reaches the maximum load within (60 ± 15) s. Five specimens shall be tested. The kind of preparation and the dimensions of the specimen, the relative rate of movement, and the five results of the flexural properties shall be reported. As far as applicable, the report shall contain the flexural stress at rupture or at maximum load, the corresponding deflection, and the modulus of elasticity.

6.4.4 Impact strength

6.4.4.1 Unnotched specimens

The method given in ISO 179-1 or ISO 179-2 shall be used. Ten specimens shall be tested. The kind of preparation, the dimensions, and the type of specimen, as well as the 10 results of the impact strength shall be reported.

6.4.4.2 Notched specimens

The method given in ISO 179-1 or ISO 179-2 shall be used with a notched specimen. Ten specimens shall be tested. The kind of preparation, the dimensions and type of specimen as well as the 10 results of the impact strength shall be reported.

6.4.5 Hardness

6.4.5.1 Rigid material

The method given in ISO 2039-1 (ball indentation method), or the method given in ISO 868 (Shore D hardness) shall be used. Five measurements shall be made on one or more specimens. The kind of preparation and the dimensions of the specimen, the test load applied, and the five results of the hardness shall be reported.

6.4.5.2 Flexible material

The method given in ISO 868 (preferably Shore A hardness) shall be used. Five measurements shall be made on one or more specimens. The kind of preparation, and the dimensions of the specimen, the type of durometer (A or D) used, and the five results of the indentation hardness shall be reported.

6.5 Thermal properties

6.5.1 Bond strength at elevated temperature

The twisted coil test, method A, or the helical coil test, method B, given in IEC 61033, shall be used. The test temperature shall be in accordance with the relevant specification sheet of IEC 60455-3, or shall be agreed upon between supplier and purchaser. Five specimens shall be tested. The method, the types of enamelled winding wire used as substrate, and the five results shall be reported.

6.5.2 Linear thermal expansion

The method given in ISO 11359-2 shall be used. The kind of preparation, the dimensions of the specimen and the result shall be reported.

6.5.3 Thermal conductivity

To be agreed between supplier and purchaser.

6.5.4 Glass transition

6.5.4.1 Glass transition temperature

One of the methods given in ISO 11357-2, ISO 11359-2 or ISO 11359-3 shall be used. Two measurements shall be made. The kind of preparation and, if applicable, the dimensions of the specimen, the method used (A: DSC or DTA, B1: TMA, expansion mode, or B2: TMA, penetration mode), and the two results of the glass transition temperature shall be reported.

6.5.4.2 Temperature of deflection under load

Method A or method B given in ISO 75 shall be used. Two specimens shall be tested. The kind of preparation and the dimensions of the specimen, the method used, and the two results of the temperature of deflection under load shall be reported.

NOTE The temperature of deflection under load is of the same nature as the glass transition temperature, but the methods given in ISO 75 do not allow temperatures below 40 °C to be determined. The methods according to 6.5.4.1 can preferably be applied.

6.5.5 Flammability

The methods A and B given in IEC 60695-11-10 shall be used. For each method five specimens shall be tested. Method A shall be applied only in the case where the result according to method B is worse than category V-2. The kind of preparation and the dimensions of the test specimen, as well as the result of flammability obtained with method B and, if applicable, with method A, shall be reported.

6.5.6 Thermal shock

To be agreed between supplier and purchaser.

6.5.7 Dry heat resistance of resins for cable accessories – Method of test

6.5.7.1 General

This test is used to determine the effect of extended exposure to dry heat on loss of mass and if required impact strength.

6.5.7.2 Specimen

Prepare the specimen from the same batch of resinous compound. The resinous compound shall be prepared and cured in accordance with the manufacturer's instructions.

- a) Loss of mass: a set of 3 test specimens shall be prepared with the dimensions of 80 mm × 40 mm × 5 mm and individually identified.
- b) Impact strength: a set of 5 test specimens shall be prepared in accordance with ISO 179.

6.5.7.3 Apparatus and materials

Equipment to be used:

- analytic balance with accuracy of ± 1 mg;
- forced air oven with temperature control;
- impact tester;
- desiccators.

6.5.7.4 Procedure

The test specimens, prepared in accordance with 6.5.7.2, shall be used as follows:

- The loss of mass specimen shall be marked individually. Take the mass by weighing it individually to the nearest of 1 mg (= mass m_1).
- All test specimens shall be placed in a forced air oven for 28 days at $(120 \pm 2)^\circ\text{C}$ or temperature stated in IEC 60455-3-8. Any mechanical loading of the specimen or any direct contact between them shall be avoided. Only one type of resinous compound shall be tested at one time. At the end of the 28 days, the set of test specimen shall be removed from the forced air oven.
- The set of test specimen shall then be placed for 8 h in a desiccator and cooled to $(23 \pm 2)^\circ\text{C}$.
- The loss of mass specimens shall be weighed individually to the nearest 1 mg (= mass m_2) immediately after removal from the desiccator.
- The impact strength shall be measured in accordance with ISO 179.

6.5.7.5 Assessment

The percent loss of mass shall be calculated for each individual sample, in accordance with the following equation:

$$M = ((m_1 - m_2) / m_1) \times 100$$

Where:

M loss of mass (%)

m_1 mass of weighing of test specimen individually identified before heating (g)

m_2 mass of weighing of test specimen individually identified after heating and cooling (g)

6.5.7.6 Test report

The test report shall include the following information:

- the average of percentage loss of mass;
- the average of the impact strength if required.

6.5.8 Wet heat resistance of resins for cable accessories

6.5.8.1 General

This test shall be carried out to the following requirements:

- a) the samples shall be prepared in accordance with the methods given in the relevant sheet of IEC 60455-3, to carry out the following tests: hardness, tensile and elongation, electric strength;
- b) final measurement of reference samples and treated samples shall be done the same day successively.

6.5.8.2 Test sequence

The test sequence shall be as follows:

- 1) Preparation of the test specimen from the same lot of resinous compound.
- 2) Immersion for 28 days at $(70 \pm 2)^\circ\text{C}$ in water.
- 3) Drying for 48 h at $(60 \pm 2)^\circ\text{C}$ under vacuum.
- 4) Drying 24 h at 80°C with open ventilation slot.
- 5) Cooling in desiccator to $(23 \pm 2)^\circ\text{C}$ (8 h).

6) Test of specimens.

6.5.8.3 General

This test is used to determine the resistance to hydrolysis of resinous compounds exposed to the simultaneous influence of water and temperature. It determines any irreversible change in selected mechanical properties by means of a tensile test and of a hardness test, and electrical properties by means of dielectric strength.

6.5.8.4 Specimen

The resinous compound shall be prepared and cured in accordance with the manufacturer's instructions.

Mechanical test:

A set of 10 test specimens no. 1B shall be prepared in accordance with 6.4.1.2 and ISO 527-1, ISO 527-2 and ISO 527-3 from blister free plates of resinous compound of thickness of $(4 \pm 0,2)$ mm.

NOTE 1 For highly filled compound, specimens can be cast in a mould of adequate dimension for the test.

Electrical test:

A set of 5 test specimens shall be prepared in accordance with IEC 60243-1 for the electric strength tester.

6.5.8.5 Apparatus and materials

Equipment to be used:

- hydrolysis vessel with a minimum capacity of 1 l for 5 test specimens each;
- forced air oven with temperature control;
- heating /vacuum cabinet;
- Shore hardness tester;
- tensile tester;
- dielectric strength tester;
- desiccator.

6.5.8.6 Procedure

The test specimens, shall be used as follows:

- A set of 5 mechanical test specimens shall be kept as reference samples. They shall be stored in the test room at standard atmosphere 23/50 in accordance with ISO 291.
- A set of 5 each of the mechanical test and of the electrical test specimens shall be immersed in water in the hydrolysis vessel for 28 days at 70 °C. The test specimens shall be placed in the hydrolysis vessel in such a manner that they are surrounded by water and immersed to a minimum water head of 10 mm. Any mechanical loading of the specimens or any direct contact between them shall be avoided. Only one type of resinous compound shall be tested at one time. The water conductivity shall be less than 500 $\mu\text{S/m}$ and its pH value shall be $7,0 \pm 0,5$. The hydrolysis vessel shall then be closed with a suitable cover and shall be placed in a forced air oven at (70 ± 2) °C and kept at that temperature during the 28 days. At the end of the 28 days, the set of test specimens shall be removed from the forced air oven and from the vessel.

The set of test specimens shall then be placed respectively in a separate heating/vacuum cabinet and dried for 48 h at $(60 \pm 2) ^\circ\text{C}$ under a $(1,5 \pm 0,2)$ kPa vacuum, then store for 24 h at $(80 \pm 2) ^\circ\text{C}$ in a forced air oven, and then shall be cooled down to $(23 \pm 2) ^\circ\text{C}$ for 8 h in desiccators.

6.5.8.7 Conditioning and assessment

The test specimens shall be conditioned before the hardness, the tensile/elongation and the electric strength tests for 24 h in accordance with ISO 291 using an atmosphere 23/50. The mechanical test specimens (reference and wet aged) shall be subjected to hardness measurement, followed by tensile/elongation measurement.

The hardness test shall be carried out in accordance with ISO 868 on ends of the mechanical test specimens outside their clamping length and in the centre of their surfaces.

The tensile and elongation test shall be carried out in accordance with ISO 527, with a jaw speed of 50 mm/min up to Shore D hardness 70.

A jaw speed of 5 mm/min shall be applied if the Shore hardness is ≥ 70 .

The electrical test specimens shall be subjected to electric strength measurement in accordance with IEC 60243-1.

For mechanical tests the following arithmetic mean for each property shall be calculated:

e_1 value on the reference test specimens;

e_2 value on the aged test specimens.

The retained percentage of the values for these properties after the temperature/water exposure shall be calculated using the following equation:

$$E = (e_2 / e_1) \times 100$$

where:

E values for these properties after the temperature/water exposure (%)

e_1 value on the reference test specimens

e_2 value on the aged test specimens

For the electric strength test, the arithmetic mean of the test specimens shall be calculated and compared to the values in IEC 60455-3-8:2013, Table 2.

6.5.8.8 Test report

The test report shall include the following information:

- retained percent of Shore hardness;
- retained percent of tensile strength;
- retained percent of elongation at break;
- value of dielectric strength.

6.5.9 Loss of mass

6.5.9.1 General

This method applies to resinous compounds for cable accessories.

6.5.9.2 Apparatus and materials

Equipment to be used:

- analytic balance with accuracy ± 1 mg;
- constant temperature oven with ventilation;
- desiccator with calcium dichloride as a drying agent.

6.5.9.3 Specimen

Three specimens with the dimension of 80 mm × 40 mm × 5 mm shall be conditioned for 24 h at standard atmosphere 23/50 in accordance with ISO 291 and individually identified.

6.5.9.4 Procedure

The three conditioned test specimens shall be weighed individually to the nearest 1 mg (= mass m_1) and stored for one week at (120 ± 2) °C in the vented oven. They shall then be cooled in a desiccator. Immediately after removal from the desiccator, the test specimens shall be weighed individually to the nearest 1 mg (= mass m_2).

6.5.9.5 Results

The percent loss of mass, M , shall be calculated for each individual sample, in accordance with the following equation:

$$M = ((m_1 - m_2) / m_1) \times 100$$

where

M Loss of mass (%)

m_1 mass of test specimens individually before heating (mg)

m_2 mass of test specimens individually after cooling (mg)

The average value of the three measurements shall be reported.

6.5.10 Temperature index

NOTE The temperature index depends on the choice of test criterion and of the end-point criterion. Therefore, for one and the same material, results for the temperature index can vary by 80 K or more.

6.5.10.1 Procedure

The method given in IEC 60216 shall be used. The test and end-point criteria shall be in accordance with the relevant specification sheet of IEC 60455-3, or shall be agreed upon between supplier and purchaser. Two test criteria shall be used. For each test criterion at least three exposure temperatures shall be applied. The difference between two subsequent exposure temperatures shall not exceed 20 K. If the correlation coefficient is less than 0,95, one more set of specimens shall be tested at an exposure temperature different from the temperature originally chosen.

NOTE ISO 2578 is based on the principles laid down in IEC 60216. By deleting all information that is not required for planning and running a temperature index experiment and for calculation of results, ISO 2578 has become a practical short version as required for use in a laboratory.

6.5.10.2 Result

For each test criterion, the kind of preparation and the type and dimensions of the specimen, the number of specimens for each test, the exposure temperatures, and the results shall be reported along with reference to the standards applied. The results for each test set shall contain the specimen end-point times, the time to end-point for each exposure temperature, a graph showing the property values as a function of the logarithm of the times to end-point, the thermal endurance graph (first order regression line) on thermal endurance graph paper, the temperature index, and the correlation coefficient.

6.6 Chemical properties

6.6.1 Water absorption

Method 1 (water at 23 °C) and method 3 (boiling water) given in ISO 62 shall be used. For each method three specimens shall be tested. The kind of preparation and the dimensions of the specimen, and the three results of water absorption obtained with each of method 1 and method 3 shall be reported. One untreated specimen shall be kept for reference.

6.6.2 Effect of liquid chemicals

The method given in ISO 175 shall be used. Unless otherwise specified, the temperature of the test liquid shall be (23 ± 2) °C and the immersion time shall be (168 ± 1) h (seven days). For each test liquid three specimens shall be tested. The kind of preparation and the dimensions of the specimen, the type of test liquid and the three results for each of the test liquids shall be reported. For each test liquid the result shall contain the change of appearance, dimensions and mass for each of the three specimens. One untreated specimen shall be kept for reference.

6.6.3 Resistance to mould growth

The method given in IEC 60068-2-10 shall be used. Three specimens according to 6.7.3.3 shall be tested, and the three results of resistance to mould growth shall be reported. One untreated specimen shall be kept for reference.

6.6.4 Water vapour permeability

To be agreed between supplier and purchaser.

6.7 Electrical properties

6.7.1 Effect of water immersion on volume resistivity

A method given in the IEC 62631-3 series shall be used. Three specimens shall be tested.

6.7.2 Dielectric dissipation factor ($\tan \delta$) and relative permittivity

The method given in IEC 62631-2-1 or IEC 62631-2-2 shall be used. Two specimens shall be tested.

6.7.3 Break down voltage and electric strength

6.7.3.1 General

Breakdown voltage shall be measured by using IEC 60243-1. If IEC 60243-1 is not applicable to the material under test, Clauses 4 and 6 may be amended as specified in 6.7.3.2.

6.7.3.2 Electrodes

The electrode arrangement shall be the ball-to-plate type. The high-voltage electrode shall consist of a polished steel ball with a radius of $(3 \pm 0,0005)$ mm for rigid material, and $(10 \pm 0,0005)$ mm for flexible material. Polished steel balls with a surface roughness of less than 0,001 mm as used in ball bearings (class III) are easily available and have been found adequate for the purpose. The earth electrode shall be a plate with a diameter of (75 ± 1) mm and with rounded edge of a radius of $(3 \pm 0,1)$ mm. For the complete test arrangement for flexible material, see Figure 2. In the case of rigid material, the upper electrode and the specimen shall be as shown in Figure 3.

NOTE 1 The ball-to-plate electrode arrangement gives, compared to a plate-to-plate set, a slightly increased field strength depending on the radius of the ball electrode and the thickness of the specimen.

EXAMPLE For a radius of 10 mm and a thickness of 0,1 mm, the increase in field strength compared to that of plate-to-plate arrangement is about 10 %.

NOTE 2 If a round cylindrical glass container of sufficient size is used to accommodate the test set-up and the fluid with the earth electrode at the bottom of it, such a container makes it possible to observe visually the process when the voltage is applied. It also permits the earth connection and the fluid supply through the bottom electrode, with a fluid overflow at the top of the container, see Figure 4. If an elevated test temperature is required, such an arrangement allows the fluid to be used for heating purposes.

6.7.3.3 Test specimen

6.7.3.3.1 General

The thickness of that part of the specimen which is subject to breakdown shall not exceed 1 mm. The thickness of any two of the specimens of one set shall not vary by more than 10 %.

NOTE It is generally found that the electric strength for cured reactive compounds with a glass transition temperature above 80 °C is from 50 kV/mm to 100 kV/mm and can be even more, for instance, for hot curing cycloaliphatic epoxide-based compounds. Consequently, testing specimens of a thickness of significantly more than 1 mm and with an electrode arrangement of, for instance, 25/75 mm as specified in IEC 60243-1, can require voltage levels above 200 kV. This can lead to conditions where flashover or partial flashover with subsequent breakdown outside the electrode area cannot be avoided.

6.7.3.3.2 Rigid material

The specimen shall form a cylindrical rod of cast compound with a diameter of about 30 mm and with a length, in millimetres, twice the assumed numerical value of breakdown voltage in kilovolts. This rod shall contain a central lead wire with a steel ball attached to it at one end and apart from the other end completely embedded in the casting compound.

After the mould is removed, that end of the specimen which is close to the ball electrode shall be ground to the specified thickness, then polished and coated with a conductive layer, for instance a dispersion of graphite or silver, which serves as the earth electrode. During grinding the thickness shall be controlled by means of a permeameter type device calibrated in thickness. For an example of the specimen set-up, see Figure 4. This set-up can be accommodated in a glass container as in Figure 4.

NOTE For casting, a glass tube can serve as a mould with the lead wire and the ball electrode properly centred by adequate means. A piece of welding wire of, for instance, 3 mm diameter can serve as a lead wire, with one end soldered to the ball electrode.

After testing, the cured compound is removed at the point of breakdown to allow measurement of the space between the polished surface and the ball electrode. The space shall be measured by means of a micrometre probe and reported as thickness.

6.7.3.3.3 Flexible material

A specimen in accordance with 6.7.3.3 shall be used.

6.7.3.4 Procedure

The rate of increase of the voltage shall be not more than 500 V/s. Unless otherwise specified, the test temperature shall be $(23 \pm 2) ^\circ\text{C}$. The test shall be carried out with the specimen and the electrodes in a dielectric fluid, which is circulated and maintained at the specified test temperature. Unless otherwise specified, unused mineral insulating oil according to IEC 60296, or unused synthetic organic ester according to IEC 61099, shall be employed.

6.7.3.5 Result

Five specimens shall be tested. The type and method of preparation of the specimen and its dimensions, the test temperature, the radius of the ball electrode, the type of dielectric fluid used, and the five results shall be reported, along with reference to the standards applied. The results shall contain the thickness of the specimen at the point of breakdown, the breakdown voltage and the electric strength.

6.7.4 Proof tracking index (PTI)

The method given in Clause 10 of IEC 60112:2003 shall be used. Three specimens shall be tested with a proof voltage as specified in the relevant specification sheet, or as agreed upon between supplier and purchaser. The kind of preparation and the dimensions of the specimen, as well as the three results of PTI, shall be reported. The results shall contain the proof voltage applied and the number of drops obtained.

6.7.5 Electrolytic corrosion

The visual method given in IEC 60426 shall be used. Three specimens shall be tested. The three results of electrolytic corrosion shall be reported.

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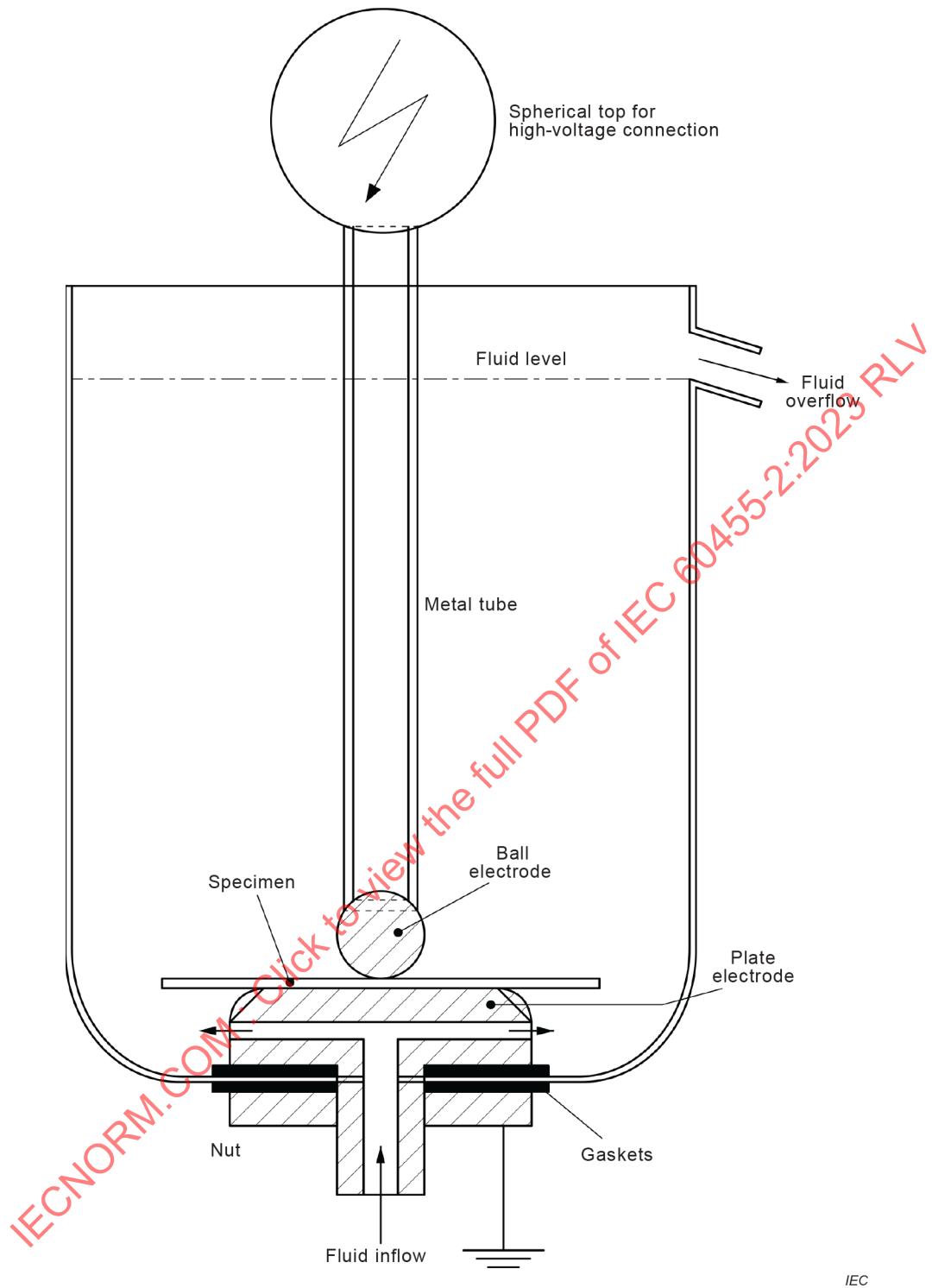


Figure 3 – Example of electrode arrangement for flexible cured compound

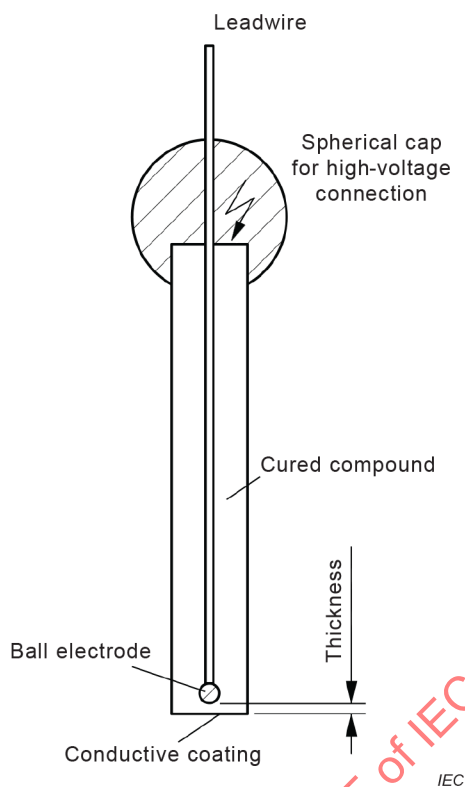


Figure 4 – Example of electrode arrangement for rigid cured compound

Annex A (informative)

Health and safety

The manufacturer shall make available on request the MSDS for each component, in order to enable the purchaser to transport, use and dispose of the components and their packaging in a safe manner, in relevant languages. Further relevant information concerning health and safety linked to installation conditions shall be made available on request.

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Bibliography

- [1] ISO 558:1980, *Conditioning and testing – Standard atmospheres – Definitions*
 - [2] ISO 2578:1993, *Plastics – Determination of time-temperature limits after prolonged exposure to heat*
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SOMMAIRE

AVANT-PROPOS	39
INTRODUCTION.....	41
1 Domaine d'application	42
2 Références normatives	42
3 Termes et définitions	45
4 Remarques générales sur les méthodes d'essai	46
4.1 Préparation et conditionnement.....	46
4.2 Séquence d'essais	46
4.3 Rapport d'essai	46
5 Méthodes d'essai pour les composés réactifs et leurs composants	47
5.1 Point d'éclair	47
5.2 Masse volumique	47
5.3 Viscosité	47
5.4 Viscosité après stockage à température élevée	47
5.5 Teneur des composés organiques volatils	47
5.6 Augmentation isothermique de la viscosité (temps de traitement)	48
5.7 Durée limite de stockage.....	48
5.8 Couleur.....	48
5.9 Température de ramollissement.....	48
5.10 Teneur en cendres	48
5.11 Teneur en charge.....	48
5.12 Teneur en chlore.....	49
5.12.1 Teneur totale en chlore des résines de polyesters non saturés et des résines époxydes.....	49
5.12.2 Teneur en chlore inorganique des résines époxydes et des esters glycidiques	49
5.12.3 Teneur en chlore facilement saponifiable des résines époxydes et des matières apparentées	49
5.13 Tendance à la cristallisation.....	49
5.14 Équivalent époxyde des résines époxydes	49
5.15 Teneur en isocyanate.....	49
5.16 Teneur en eau (méthode de Karl Fischer)	49
5.17 Indice d'hydroxyle	49
5.17.1 Résines de polyesters	49
5.17.2 Résines autres que le polyester.....	49
5.18 Indice d'acide des résines de polyesters	49
5.19 Nombre de doubles liaisons des résines de polyesters non saturés et des résines acryliques	50
5.20 Teneur en acide et en anhydride d'acide des durcisseurs acide-anhydride.....	50
5.21 Teneur en amine.....	50
5.22 Durée de vie en pot	50
5.22.1 Généralités	50
5.22.2 Composés résineux pour accessoires de câbles	50
5.23 Temps de gélification	51
5.23.1 Composés à base de polyesters non saturés	51
5.23.2 Composés à base de résines phénoliques	51
5.23.3 Autres composés	51

5.24	Montée exothermique de température	51
5.24.1	Composés à base de polyesters non saturés	51
5.24.2	Composés résineux pour accessoires de câbles	51
5.25	Retrait total en volume des résines de polyesters non saturés et des résines époxydes	52
5.26	Durcissement en présence d'eau	52
5.26.1	Généralités	52
5.26.2	Appareillage et matériels	52
5.26.3	Dispositif verseur	52
5.26.4	Procédure	52
5.26.5	Rapport d'essai	53
5.27	Détermination du degré de durcissement	54
5.28	Durcissement en couche épaisse et émissions pendant le durcissement	54
5.28.1	Généralités	54
5.28.2	Matériels	55
5.28.3	Éprouvette	55
5.28.4	Procédure	55
6	Méthodes d'essai pour les composés réactifs durcis	56
6.1	Généralités	56
6.2	Éprouvettes	56
6.2.1	Généralités	56
6.2.2	Préparation du composé réactif	57
6.2.3	Préparation des éprouvettes	57
6.2.4	Type et nombre d'éprouvettes	57
6.3	Masse volumique	57
6.4	Propriétés mécaniques	57
6.4.1	Propriétés en traction	57
6.4.2	Propriétés en compression	58
6.4.3	Propriétés en flexion	58
6.4.4	Résistance au choc	58
6.4.5	Dureté	58
6.5	Propriétés thermiques	58
6.5.1	Pouvoir agglomérant à température élevée	58
6.5.2	Dilatation thermique linéaire	59
6.5.3	Conductivité thermique	59
6.5.4	Transition vitreuse	59
6.5.5	Inflammabilité	59
6.5.6	Choc thermique	59
6.5.7	Résistance à la chaleur sèche des résines pour accessoires de câbles – Méthode d'essai	59
6.5.8	Résistance à la chaleur humide des résines pour accessoires de câbles	60
6.5.9	Perte de masse	63
6.5.10	Indice de température	64
6.6	Propriétés chimiques	64
6.6.1	Absorption d'eau	64
6.6.2	Effets des liquides chimiques	65
6.6.3	Résistance aux moisissures	65
6.6.4	Perméabilité à la vapeur d'eau	65
6.7	Propriétés électriques	65

6.7.1	Effet de l'immersion dans l'eau sur la résistivité volumique	65
6.7.2	Facteur de dissipation diélectrique ($\tan \delta$) et permittivité relative	65
6.7.3	Tension de claquage et rigidité diélectrique	65
6.7.4	Indice de tenue au cheminement (ITC)	67
6.7.5	Corrosion électrolytique	67
Annexe A (informative) Santé et sécurité		70
Bibliographie.....		71
Figure 1 – Grille d'examen		53
Figure 2 – Position de la grille d'examen sur l'éprouvette.....		54
Figure 3 – Exemple de montage des électrodes pour les composés durcis souples		68
Figure 4 – Exemple de montage des électrodes pour les composés durcis rigides		69
Tableau 1 – État du côté supérieur		55
Tableau 2 – État du côté inférieur		55
Tableau 3 – État de l'intérieur		56
Tableau 4 – Vides		56

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COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

**COMPOSÉS RÉACTIFS À BASE DE RÉSINES
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Cette quatrième édition annule et remplace la troisième édition parue en 2015. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

- a) introduction des méthodes d'essai de l'IEC 60455-3-8;
- b) ajout et mise à jour des méthodes d'essai pour les résines.

Le texte de cette Norme internationale est issu des documents suivants:

Projet	Rapport de vote
15/1006/FDIS	15/1015/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

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INTRODUCTION

La présente partie de l'IEC 60455 appartient à une série qui traite des composés réactifs à base de résines exemptes de solvant et de leurs composants, utilisés pour l'isolation électrique.

Cette série est composée de trois parties:

- Partie 1: Définitions et prescriptions générales;
- Partie 2: Méthodes d'essai;
- Partie 3: Spécifications pour matériaux particuliers.

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COMPOSÉS RÉACTIFS À BASE DE RÉSINES UTILISÉS COMME ISOLANTS ÉLECTRIQUES –

Partie 2: Méthodes d'essai

1 Domaine d'application

La présente partie de l'IEC 60455 spécifie les méthodes d'essai à utiliser pour vérifier les composés réactifs à base de résines, leurs composants et les composés durcis, utilisés pour l'isolation électrique.

2 Références normatives

Les documents suivants sont cités dans le texte de sorte qu'ils constituent, pour tout ou partie de leur contenu, des exigences du présent document. Pour les références datées, seule l'édition citée s'applique. Pour les références non datées, la dernière édition du document de référence s'applique (y compris les éventuels amendements).

IEC 60050 (toutes les parties), *Vocabulaire Électrotechnique International* (disponible à l'adresse <http://www.electropedia.org>)

IEC 60068-2-10:2005, *Essais d'environnement – Partie 2-10: Essais – Essai J et guide: Moisissures*

IEC 60112:2020, *Méthode de détermination des indices de résistance et de tenue au cheminement des matériaux isolants solides*

IEC 60216 (toutes les parties), *Matériaux isolants électriques – Propriétés d'endurance thermiques*

IEC 60243-1:2013, *Rigidité diélectrique des matériaux isolants – Méthodes d'essai – Partie 1: Essais aux fréquences industrielles*

IEC 60296:2020, *Fluides pour applications électrotechniques – Huiles minérales isolantes pour matériel électrique*

IEC 60426:2007, *Matériaux isolants électriques – Détermination de la corrosion électrolytique en présence de matériaux isolants – Méthodes d'essais*

IEC 60455-1:1998, *Composés réactifs à base de résine utilisés comme isolants électriques – Partie 1: Définitions et prescriptions générales*

IEC 60455-3 (toutes les parties), *Composés réactifs à base de résines utilisés comme isolants électriques – Partie 3: Spécifications pour matériaux particuliers*

IEC 60455-3-8:2021, *Composés réactifs à base de résine utilisés comme isolants électriques – Partie 3-8: Spécifications pour matériaux particuliers – Résines pour accessoires de câble*

IEC 60695-11-10:2013, *Essais relatifs aux risques du feu – Partie 11-10: Flammes d'essai – Méthodes d'essai horizontale et verticale à la flamme de 50 W*

IEC 60814:1997, *Isolants liquides – Cartons et papiers imprégnés d'huile – Détermination de la teneur en eau par titrage coulométrique de Karl Fischer automatique*

IEC 61033:1991, *Méthodes d'essai pour la détermination du pouvoir agglomérant des agents d'imprégnation sur fil émaillé*

IEC 61099:2010, *Liquides isolants – Spécifications relatives aux esters organiques de synthèse neufs destinés aux matériels électriques*

ISO 37:2011, *Caoutchouc vulcanisé ou thermoplastique – Détermination des caractéristiques de contrainte-déformation en traction*

ISO 62:2008, *Plastiques – Détermination de l'absorption d'eau*

ISO 75 (toutes les parties), *Plastiques et ébonite – Détermination de la température de fléchissement sous charge*

ISO 175:2010, *Plastiques – Méthodes d'essai pour la détermination des effets de l'immersion dans des produits chimiques liquides*

ISO 178:2010, *Plastiques – Détermination des propriétés en flexion*

ISO 179-1:2010, *Plastiques – Détermination des caractéristiques au choc Charpy – Partie 1: Essai de choc non instrumenté*

ISO 179-2:1997, *Plastiques – Détermination des caractéristiques au choc Charpy – Partie 2: Essai de choc instrumenté*

ISO 291, *Plastiques – Atmosphères normales de conditionnement et d'essai*

ISO 306:2013, *Plastiques – Matériaux thermoplastiques – Détermination de la température de ramollissement Vicat (VST)*

ISO 527 (toutes les parties), *Plastiques – Détermination des propriétés en traction*

ISO 584:1982, *Plastiques – Résines de polyesters non saturés – Détermination conventionnelle de la réactivité à 80 °C*

ISO 604:2002, *Plastiques – Détermination des propriétés en compression*

ISO 868:2003, *Plastiques et ébonite – Détermination de la dureté par pénétration au moyen d'un duromètre (dureté Shore)*

ISO 1183-1:2019, *Plastiques – Méthodes de détermination de la masse volumique des plastiques non alvéolaires – Partie 1: Méthode par immersion, méthode du pycnomètre en milieu liquide et méthode par titrage*

ISO 1513:2010, *Peintures et vernis – Examen et préparation des échantillons pour essai*

ISO 1523:2002, *Détermination du point d'éclair – Méthode à l'équilibre en vase clos*

ISO 1675:1985, *Plastiques – Résines liquides – Détermination de la masse volumique par la méthode du pycnomètre*

ISO 2039-1:1993, *Plastiques – Détermination de la dureté – Partie 1: Méthode de pénétration à la bille*

ISO 2114:2000, *Plastiques (résines de polyesters) et peintures et vernis (liants) – Détermination de l'indice d'acide partiel et de l'indice total*

ISO 2431:1993, *Peintures et vernis – Détermination du temps d'écoulement au moyen de coupes d'écoulement*

ISO 2535:1997, *Plastiques – Résines de polyesters non saturés – Mesurage de la durée de gélification à 25 °C*

ISO 2554:1997, *Plastiques – Résines de polyesters non saturés – Détermination de l'indice d'hydroxyle*

ISO 2555:1989, *Plastiques – Résines à l'état liquide ou en émulsions ou dispersions – Détermination de la viscosité apparente par la méthode du viscosimètre rotatif de type à un cylindre*

ISO 2592:1973, *Produits pétroliers – Détermination des points d'éclair et de feu – Méthode Cleveland en vase ouvert*

ISO 3001:1997, *Plastiques – Compositions époxydiques – Détermination de l'équivalent époxy*

ISO 3219:1993, *Plastiques – Polymères résines à l'état liquide en émulsion ou en dispersion – Détermination de la viscosité au moyen d'un viscosimètre rotatif à gradient de vitesse de cisaillement définie*

ISO 3451-1:1997, *Plastiques – Détermination du taux de cendres – Partie 1: Méthodes générales*

ISO 3521:1997, *Plastiques – Résines d'époxydes et de polyesters non saturés – Détermination du retrait global en volume*

ISO 3679:1983, *Peintures, vernis, produits pétroliers et assimilés – Détermination du point d'éclair – Méthode rapide à l'équilibre*

ISO 4573:1978, *Plastiques – Résines d'époxydes et esters glycidiques – Dosage du chlore inorganique*

ISO 4583:1998, *Plastiques – Résines époxydes et matières apparentées – Dosage du chlore facilement saponifiable*

ISO 4615:1979, *Plastiques – Résines de polyesters non saturés et époxydes – Détermination de la teneur totale en chlore*

ISO 4625:1980, *Liants pour peintures et vernis – Détermination du point de ramollissement – Méthode de l'anneau et de la bille*

ISO 4895, *Plastiques – Résines époxydes liquides – Détermination de la tendance à la cristallisation*

ISO 7056, *Matériel de laboratoire en plastique – Béchers*

ISO 9396:1997, *Plastiques – Résines phénoliques – Détermination du temps de gélification des résols dans des conditions spécifiques sur appareils automatiques*

ISO 11357-2:1999, *Plastiques – Analyse calorimétrique différentielle (DSC) – Partie 2: Détermination de la température de transition vitreuse*

ISO 11359-2:1999, *Plastiques – Analyse thermomécanique (TMA) – Partie 2: Détermination du coefficient de dilatation thermique linéique et de la température de transition vitreuse*

ISO 11359-3:2002, *Plastiques – Analyse thermomécanique (TMA) – Partie 3: Détermination de la température de pénétration*

ISO 14896:2009, *Plastiques – Matières premières des polyuréthanes – Détermination de la teneur en isocyanate*

ISO 15528:2000, *Peintures, vernis et matières premières pour peintures et vernis – Échantillonnage*

3 Termes et définitions

Pour les besoins du présent document, les termes et définitions de l'IEC 60455-1 et de l'IEC 60050 ainsi que les suivants s'appliquent.

L'ISO et l'IEC tiennent à jour des bases de données terminologiques destinées à être utilisées en normalisation, consultables aux adresses suivantes:

- IEC Electropedia: disponible à l'adresse <https://www.electropedia.org/>
- ISO Online browsing platform: disponible à l'adresse <https://www.iso.org/obp>

3.1

résistance volumique

partie de la résistance d'isolement qui est due à la conduction à travers le volume et qui exclut le courant à sa surface

3.2

résistivité volumique

résistance volumique ramenée à l'unité de volume cubique

3.3

facteur de dissipation diélectrique

$\tan \delta$

valeur numérique du rapport de la partie imaginaire sur la partie réelle de la permittivité complexe

3.4

permittivité relative

ϵ_r

rapport de la permittivité absolue sur la constante diélectrique

Note 1 à l'article: En ingénierie, il est courant d'employer le terme "permittivité" pour faire référence à la permittivité relative.

4 Remarques générales sur les méthodes d'essai

4.1 Préparation et conditionnement

Sauf spécification contraire dans la norme de spécifications correspondante ou dans la méthode d'essai, tous les essais doivent être réalisés dans les conditions atmosphériques, avec une température comprise entre 21 °C et 29 °C et une humidité relative comprise entre 45 % et 70 %. Avant d'effectuer les mesurages, l'échantillon ou l'éprouvette doit être préconditionné dans ces conditions atmosphériques pendant une durée suffisante pour permettre à l'échantillon ou à l'éprouvette de se stabiliser. Pour prélever des échantillons au format liquide ou pâteux, l'ISO 15528 doit être appliquée. Pour préparer ces échantillons d'essai, l'ISO 1513 doit être appliquée.

NOTE Pour les définitions des termes relatifs aux atmosphères normales, voir l'ISO 558. L'atmosphère d'essai spécifiée ci-dessus ne satisfait à aucune des deux atmosphères normales spécifiées dans l'ISO 291, mais couvre les deux plages y compris leurs tolérances.

Normalement, toutes les exigences d'une méthode d'essai sont fournies dans la description et les schémas ont uniquement pour objet de montrer les différents montages d'essai possibles. En cas de contradictions entre le présent document et les feuilles de spécifications de la série IEC 60455-3, ces dernières doivent prévaloir. Si une autre norme est citée comme méthode d'essai, la référence à cette norme doit être fournie dans le rapport.

4.2 Séquence d'essais

Pour éviter tout effort inutile, les essais doivent être effectués sur les échantillons selon la séquence suivante:

- 1) essais sur chaque composant avant le mélange;
- 2) essais sur le composé réactif immédiatement après le mélange (prêt à l'emploi);
- 3) essais sur le composé durci;
- 4) essais sur le composé durci après le prétraitement (température, humidité, eau, etc.).

Si l'échantillon en essai échoue à l'essai, les essais suivants peuvent devenir obsolètes.

4.3 Rapport d'essai

Sauf spécification contraire, le rapport d'essai doit inclure les données suivantes:

- 1) désignation et identification de la résine;
- 2) numéro de lot ou autre identification;
- 3) confirmation du marquage et de l'étiquetage selon la fiche de données de sécurité (MSDS, *Material Safety Data Sheet*);
- 4) résultats d'essai;
- 5) principaux paramètres d'essai, notamment le conditionnement et l'étalonnage, s'il y a lieu;
- 6) conditions de traitement utilisées pour le composé réactif;
- 7) copie de la fiche technique (TDS, *Technical Data Sheet*) et de la MSDS.

5 Méthodes d'essai pour les composés réactifs et leurs composants

5.1 Point d'éclair

Pour les températures de point d'éclair supérieures ou égales à 79 °C, la méthode spécifiée dans l'ISO 2592 doit être utilisée. Pour les températures de point d'éclair inférieures à 79 °C, la méthode spécifiée dans l'ISO 1523 doit être utilisée, avec l'un des appareillages en vase clos décrits à l'Annexe A de l'ISO 1523:2002. L'ISO 1523 doit être lue conjointement avec l'ISO 3679. Deux mesurages doivent être effectués sur deux échantillons distincts, et les deux mesures de point d'éclair doivent être consignées avec la référence aux normes utilisées.

5.2 Masse volumique

La méthode spécifiée dans l'ISO 1675 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de masse volumique doivent être consignées.

5.3 Viscosité

Sauf spécification contraire, la viscosité doit être déterminée au moyen d'un dispositif adapté à $(23 \pm 0,5)$ °C. Si un dispositif rotatif est utilisé, celui-ci doit être conforme à l'ISO 2555 (dispositif Brookfield) ou à l'ISO 3219 (dispositif à gradient de vitesse de cisaillement défini). Si un dispositif de type à écoulement est utilisé, la méthode d'essai et la coupe d'écoulement doivent être conformes à l'ISO 2431. Deux mesurages doivent être effectués, et les deux mesures de viscosité doivent être consignées avec la référence aux normes utilisées.

5.4 Viscosité après stockage à température élevée

Cette méthode ne s'applique pas aux systèmes monocomposants ou aux composants qui contiennent des durcisseurs.

Sauf spécification contraire, un échantillon en quantité suffisante est stocké pendant $(20 \pm 0,5)$ h à une température de (100 ± 3) °C dans un récipient fermé. Après refroidissement à température ambiante, la viscosité est mesurée conformément au 5.3. L'augmentation de la viscosité est calculée à l'aide de l'équation suivante:

$$\text{Augmentation de la viscosité en \%} = (\eta_2 - \eta_1) \times 100 / \eta_1$$

où

η_1 viscosité dynamique avant stockage

η_2 viscosité dynamique après stockage

5.5 Teneur des composés organiques volatils

Cette méthode ne s'applique pas aux systèmes monocomposants ou aux composants qui contiennent des durcisseurs. Sauf spécification contraire, l'essai doit être effectué comme suit:

La masse d'un flacon à tare vide (de dimensions 80 mm × 30 mm environ) est fixée à 0,001 g (m_1). Une masse de 0,4 g à 0,5 g de résine (m_2) est pesée à 0,001 g dans le flacon à tare (hermétiquement fermé pendant la pesée). Quelques gouttes de toluène sont ajoutées pour diluer la résine. Le liquide est dispersé au fond du flacon à tare en appliquant une légère torsion.

Le flacon à tare ouvert est placé dans un four à ventilation forcée pendant au moins 2 h à (110 ± 2) °C. Après refroidissement à température ambiante dans un dessiccateur, le poids du flacon à tare est de nouveau pesé à 0,001 g (m_3).

Composés organiques volatils = $100 \times (m_2 - (m_3 - m_1)) / m_2$

où:

m_1 masse d'un flacon à tare vide (g)

m_2 masse de la résine avant chauffage (g)

m_3 masse d'un flacon à tare vide rempli de résine après chauffage et refroidissement (g)

5.6 Augmentation isothermique de la viscosité (temps de traitement)

Cette méthode est utilisée pour les résines polyuréthanes (PUR) et époxydes (EP). Pour les résines UP (polyesters non saturés), le temps de gélification doit être utilisé. Sauf spécification contraire, l'essai doit être effectué comme suit:

Tous les composants et matériels doivent être à température ambiante. Les composants de la résine sont mélangés conformément aux instructions du fabricant. La procédure de mélange ne doit pas durer plus de 3 min. Le chronomètre est démarré après ajout et mélange du dernier composant. Après 10 min, la première mesure de viscosité est prise comme valeur initiale. Le mesurage est répété jusqu'à ce que la viscosité atteigne la valeur maximale spécifiée. L'intervalle de temps entre la valeur initiale et la valeur maximale est consigné comme temps de traitement.

5.7 Durée limite de stockage

La durée limite de stockage doit être déterminée en mesurant les variations d'une propriété caractéristique spécifique après un temps et une température de stockage donnés. L'expérience a montré que la viscosité selon le 5.3 et le temps de gélification selon le 5.23 constituaient des caractéristiques appropriées. Pour déterminer la durée limite de stockage, la viscosité et/ou le temps de gélification doivent être déterminés conformément au 5.3 et/ou au 5.23, respectivement, pour une température et un point limite, fixés par accord entre le fournisseur et l'acheteur. Deux mesurages doivent être effectués sur un matériau fraîchement préparé et sur un matériau stocké pendant la durée et à la température fixées par accord entre le fournisseur et l'acheteur. Les deux mesures de durée limite de stockage doivent être consignées avec la référence aux normes utilisées. Les mesures doivent comprendre la viscosité et/ou le temps de gélification avant et après stockage, le temps et la température de stockage, ainsi que la température d'essai.

5.8 Couleur

La méthode spécifiée dans l'ISO 6271 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de couleur doivent être consignées avec la référence aux normes utilisées.

5.9 Température de ramollissement

La méthode spécifiée dans l'ISO 306 ou l'ISO 4625 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de température de ramollissement doivent être consignées avec la référence aux normes utilisées.

5.10 Teneur en cendres

La méthode spécifiée dans l'ISO 3451-1, méthode A, doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de teneur en cendres doivent être consignées.

5.11 Teneur en charge

À fixer par accord entre le fournisseur et l'acheteur.

5.12 Teneur en chlore

5.12.1 Teneur totale en chlore des résines de polyesters non saturés et des résines époxydes

La méthode spécifiée dans l'ISO 4615 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de teneur totale en chlore doivent être consignées.

5.12.2 Teneur en chlore inorganique des résines époxydes et des esters glycidiques

La méthode spécifiée dans l'ISO 4573 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de teneur en chlore inorganique doivent être consignées.

5.12.3 Teneur en chlore facilement saponifiable des résines époxydes et des matières apparentées

La méthode spécifiée dans l'ISO 4583 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de teneur en chlore saponifiable doivent être consignées.

5.13 Tendance à la cristallisation

Cette méthode s'applique aux résines époxydes uniquement.

La méthode spécifiée dans l'ISO 4895 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures doivent être consignées.

5.14 Équivalent époxyde des résines époxydes

La méthode spécifiée dans l'ISO 3001 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures d'équivalent époxyde doivent être consignées.

5.15 Teneur en isocyanate

Cette méthode s'applique aux durcisseurs en polyuréthane uniquement.

La méthode spécifiée dans l'ISO 14896 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures doivent être consignées.

5.16 Teneur en eau (méthode de Karl Fischer)

La méthode spécifiée dans l'IEC 60814 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures de teneur en eau doivent être consignées.

5.17 Indice d'hydroxyle

5.17.1 Résines de polyesters

La méthode spécifiée dans l'ISO 2554 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures d'indice d'hydroxyle doivent être consignées.

5.17.2 Résines autres que le polyester

À fixer par accord entre le fournisseur et l'acheteur.

5.18 Indice d'acide des résines de polyesters

La méthode spécifiée dans l'ISO 2114 doit être utilisée. Deux mesurages doivent être effectués, et les deux mesures d'indice d'acide doivent être consignées.

5.19 Nombre de doubles liaisons des résines de polyesters non saturés et des résines acryliques

À fixer par accord entre le fournisseur et l'acheteur.

5.20 Teneur en acide et en anhydride d'acide des durcisseurs acide-anhydride

La méthode spécifiée dans l'ISO 2114 doit être utilisée pour la teneur en acide. La méthode spécifiée dans l'ISO 7327 doit être utilisée pour la teneur en anhydride. Deux mesurages doivent être effectués, et les deux mesures de teneur en acide et/ou en anhydride doivent être consignées.

5.21 Teneur en amine

Sauf accord contraire entre le fournisseur et l'acheteur, la méthode spécifiée dans l'ISO 9702 doit être utilisée.

5.22 Durée de vie en pot

5.22.1 Généralités

Le temps exigé par une méthode particulière pour obtenir une viscosité de 50 Pas est mesuré.

La viscosité peut être déterminée au moyen d'un viscosimètre quelconque.

5.22.2 Composés résineux pour accessoires de câbles

5.22.2.1 Appareillage et matériels

Matériels à utiliser:

- b cher conform ment   l'ISO 7056 en poly thyl ne, en polypropyl ne ou en verre, avec les dimensions suivantes: diam tre de 70 mm   100 mm et hauteur de 70 mm   130 mm;
- enceinte climatique;
- chronom tre;
- viscosim tre.

5.22.2.2 Pr paration de l' chantillon

Le b cher doit  tre marqu  pour un volume de (300 ± 25) ml.

Le b cher et un paquet normalis  du fabricant doivent  tre stock s dans une enceinte climatique pendant 24 h   la temp rature sp cifi e avec une tol rance de $\begin{smallmatrix} 0 \\ -2 \end{smallmatrix}$ K.

5.22.2.3 Proc dure

Les composants doivent  tre retir s de l'enceinte climatique et le m lange doit d marrer imm diatement apr s, conform ment aux instructions du fournisseur. L'enregistrement du temps d marre au d but du m lange des composants.

Retirer le b cher de l'enceinte climatique, puis verser le compos  m lang  dans le b cher jusqu'au rep re pr c demment marqu .