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Resin based reactive compounds used for electrical insulation - Part 2: Methods of test (IEC 60455-2:1998)

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

Reaktionsharzmassen für die Elektroisolierung -- Teil 2: Prüfverfahren

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

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

(IEC 60455-2:1998)

Composés réactifs à base de résines
utilisés comme isolants électriques
Partie 2: Méthodes d'essai
(CEI 60455-2:1998)

Reaktionsharzmassen für
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CENELEC

European Committee for Electrotechnical Standardization
Comité Européen de Normalisation Electrotechnique
Europäisches Komitee für Elektrotechnische Normung

Central Secretariat: rue de Stassart 35, B-1050 Brussels

Foreword

The text of document 15C/893 + 1000/FDIS, future edition 2 of IEC 60455-2, prepared by SC 15C, Specifications, of IEC TC 15, Insulating materials, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 60455-2 on 1999-04-01.

This European Standard supersedes HD 307.2 S1:1986.

The following dates were fixed:

- latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement (dop) 2000-01-01
- latest date by which the national standards conflicting with the EN have to be withdrawn (dow) 2002-01-01

Annexes designated “normative” are part of the body of the standard.

Annexes designated “informative” are given for information only.

In this standard, Annex ZA is normative and Annex A is informative.

Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60455-2:1998 was approved by CENELEC as a European Standard without any modification.

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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* (IEC 60455-1)¹⁾;
- *Part 2: Methods of test* (IEC 60455-2);
- *Part 3: Specifications for individual materials* (IEC 60455-3).

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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of IEC 60455. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of IEC 60455 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

- IEC 60050(212):1990, *International Electrotechnical Vocabulary (IEV) — Chapter 212: Insulating solids, liquids and gases*.
- IEC 60068-2-10:1988, *Environmental testing — Part 2: 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 60112:1979, *Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions*.
- IEC 60216-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 1: General guidelines for ageing procedures and evaluation of test results*.
- IEC 60216-2:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 2: Choice of test criteria*.

IEC 60216-3-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 3: Instructions for calculating thermal endurance characteristics — Section 1: Calculations using mean values of normally distributed complete data*.

IEC 60216-3-2:1993, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 3: Instructions for calculating thermal endurance characteristics — Section 2: Calculations for incomplete data: proof test results up to and including the median time to end-point (equal test groups)*.

IEC/TR 60216-4-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 4: Ageing ovens — Section 1: Single-chamber ovens*.

IEC 60216-5:1998, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 5: Guidelines for application of thermal endurance characteristics*.

IEC 60243-1:1998, *Electric 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 60296:1982, *Specification for unused mineral insulating oils for transformers and switchgear*.

IEC 60426:1973, *Test methods for determining electrolytic corrosion with insulating materials*.

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

IEC 60455-3 (all parts), *Specification for solventless polymerisable resinous compounds used for electrical insulation — Part 3: Specifications for individual materials*.

IEC 60707:1981, *Methods of test for the determination of the flammability of solid electrical insulating materials when exposed to an igniting source*.

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

IEC 61006:1991, *Methods of test for the determination of the glass transition temperature of electrical insulating materials*.

¹⁾ To be published.

- IEC 61033:1991, *Test methods for the determination of bond strength of impregnating agents to an enamelled wire substrate.*
- IEC 61099:1992, *Specifications for unused synthetic organic esters for electrical purposes.*
- ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*
- ISO 62:1980, *Plastics — Determination of water absorption²⁾.*
- ISO 75 (all parts), *Plastics and ebonite — Determination of temperature of deflection under load.*
- ISO 175:1981, *Plastics — Determination of the effects of liquid chemicals, including water.*
- ISO 178:1993, *Plastics — Determination of flexural properties.*
- ISO 179:1993, *Plastics — Determination of Charpy impact strength²⁾.*
- ISO 306:1994, *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 604:1993, *Plastics — Determination of compressive properties.*
- ISO 688:1985, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness).*
- ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics.*
- ISO 1512:1991, *Paints and varnishes — Sampling of products in liquid or paste form.*
- ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing.*
- ISO 1523:1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — 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 2114:1996, *Plastics — Unsaturated polyester resins — 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 method.*
- ISO 2592:1973, *Petroleum products — Determination of flash and fire points — Cleveland open cup method²⁾.*
- ISO 3001:1997, *Plastics — Epoxide compounds — Determination of epoxide equivalent²⁾.*
- 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 7327:1994, *Plastics — Hardeners and accelerators for epoxide resins — Determination of free acid in acid anhydride.*
- ISO 9396:1997, *Plastics — Phenolic resins — Determination of the gel time at a given temperature using automatic apparatus.*
- ISO 9702:1998, *Plastics — Amine epoxide hardeners — Determination of primary, secondary and tertiary amine group nitrogen content.*

²⁾ Revision to be published.

3 Definitions

For the purpose of this part of IEC 60455, the following definitions, as well as the definitions of IEC 60455-1 and IEC 60050(212), apply:

3.1

volume resistance

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

3.2

volume resistivity

the 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 In practical engineering, it is usual to employ the term "permittivity" when referring to relative permittivity.

4 General notes on methods of test

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 1512 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 standard and the specification sheets of IEC 60455-3 of this series, the latter shall prevail.

When another standard is invoked for a test method, reference to that standard shall be included in the report.

5 Methods of test for reactive compounds and their components

The materials before curing are resins (1), other reactive or non-reactive components (2) (for instance hardener, accelerator, stabilizer, filler), and the ready-to-use reactive compound (3). The numbers 1, 2 and/or 3 indicate the possible application of the methods of test described below.

5.1 Flash point (1, 2 and 3)

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 that standard. ISO 1523 shall be read in conjunction with ISO 3679.

Two measurements shall be made on two separate samples, and the two results of flash point shall be reported along with reference to the standards applied.

5.2 Density (1, 2 and 3)

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

5.3 Viscosity (1, 2 and 3)

The viscosity shall be determined with a suitable device at $(23 \pm 0,5)$ °C. 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 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 viscosity shall be reported, along with reference to the standards applied.

5.4 Shelf life (1, 2 and 3)

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 5.17 are appropriate characteristics. To assess shelf life, viscosity and/or gel time shall be determined according to 5.3 and/or 5.17 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.

5.5 Softening temperature (1 and 2)

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.

5.6 Ash content (1 and 2)

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

5.7 Filler content (1 and 2)

Method of test required, but not available.

5.8 Chlorine content**5.8.1 Total chlorine content of unsaturated polyesters and epoxide resins (1 and 2)**

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

5.8.2 Inorganic chlorine content of epoxide resins and glycidyl esters (1)

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

5.8.3 Easily saponifiable chlorine content of epoxide resins and related materials (1)

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

5.9 Epoxide equivalent of epoxide resins (1)

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

5.10 Water content (Karl Fischer method) (1 and 2)

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

5.11 Hydroxyl value**5.11.1 Polyester resins (1)**

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

5.11.2 Resins other than polyester (1)

Method of test required, but not available.

5.12 Acid value of polyester resins (1)

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

5.13 Amount of double bonds of unsaturated polyester and acrylate resins (1)

Method of test required, but not available.

5.14 Acid and acid-anhydride content of acid-anhydride hardeners (2)

Method of test required, but not available.

5.15 Amine value (2)

Method of test required, but not available.

5.16 Pot life (3)

Pot life shall be determined by measurement of the change in a specified characteristic property after mixing. To assess pot life, viscosity and/or gel time shall be determined according to 5.3 and/or 5.17 respectively, at a temperature and with an end-point as agreed upon between supplier and purchaser. Two measurements shall be made, both on freshly prepared material and on material stored after preparation for a time and at a temperature as agreed upon between supplier and purchaser. The two results of pot life shall be reported along with reference to the standards applied. The report shall contain the viscosity and/or the gel time before and after storing, the storing time and temperature and the test temperature.

5.17 Gel time**5.17.1 Unsaturated polyester based compounds (3)**

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 gel time shall be reported along with the test temperature.

5.17.2 Phenolic resin based compounds (3)

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

5.17.3 Other compounds (3)

Method of test required, but not available.

5.18 Exothermic temperature rise**5.18.1 Unsaturated polyester based compounds (3)**

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

5.18.2 Compounds other than unsaturated polyester based (3)

Method of test required, but not available.

5.19 Total volume shrinkage of epoxide and unsaturated polyester based compounds (3)

The method given in ISO 3521 shall be used. Two measurements shall be made, and the two results of 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.

6 Methods of test for cured reactive compounds

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

6.1 Test specimens

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

6.1.1 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.

6.1.2 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.

6.1.3 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.

6.2 Density

Method A or method B given in ISO 1183 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 density shall be reported.

6.3 Mechanical properties

6.3.1 Tensile properties

6.3.1.1 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 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.

6.3.1.2 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 tensile properties shall be reported. The report shall contain tensile strength, percentage elongation at break, and modulus of elasticity.

6.3.2 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 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.