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Plastics — Phenolic resins — Classification and test methods

Plastiques — Résines phénoliques — Classification et méthodes d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10082 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 10082:1991) and ISO/TR 8244:1988, which have been technically revised.

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Plastics — Phenolic resins — Classification and test methods

1 Scope

This International Standard gives an overview of the different types of phenolic resin used in the manufacture of thermosetting plastics and a summary of available test methods.

Clause 3 defines terms relating to the chemical structure of phenolic resins, their physical state and their degree of condensation or polycondensation.

Clause 4 gives a summary of test methods. These methods apply to the determination of properties which are relevant to the production, processing and use of phenolic resins as described in clause 3. The user should select from the tests those that are appropriate for a particular application. For a given method, only the standard(s) listed may be used.

2 Normative referencereh STANDARD PREVIEW

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document 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. 1316101502cb/iso-10082-1999

ISO 472:1999, Plastics - Vocabulary.

3 Basis of classification of phenolic resins

This clause defines terms relating to the chemical structure of phenolic resins, their physical state and their degree of condensation or polycondensation for classification purposes in the manufacturing and processing of the resins.

The definitions apply to all fields of application of phenolic resins in plastics (see also related definitions in ISO 472).

3.1 General

- 3.1.1 The term "phenolic resins" as used in this International Standard refers to
- a) synthetic resins or modified products obtained by condensation of phenol with aldehydes, particularly formaldehyde;
- b) products of the addition of phenols to unsaturated compounds (for example acetylene, terpenes and natural resins)¹).

¹⁾ Strictly speaking, these resins are not what are normally regarded as resins for the production of plastics. They are used particularly in certain surface coatings.

NOTE Resins in which the original properties are so changed by the modification that they resemble more closely those of the modifying medium are not regarded as phenolic resins.

- 3.1.2 Phenolic resins may be classified in a number of ways, such as by
- a) the type of raw material (see 3.2);
- b) the type of resin produced (see 3.3);
- c) the type of modification (see 3.3.2);
- d) the physical form of the commercial product (see 3.4);
- e) the degree of condensation (see 3.5);
- f) the type of catalyst used (see 3.6.1);
- g) the type of hardener used (see 3.6.2).

3.2 Raw materials

3.2.1 Phenols

Besides unsubstituted phenol, derivatives of phenol are used for the manufacture of phenolic resins. These include cresols, xylenols and other alkylphenols (for example *p-tert*-butylphenol, *p-tert*-octylphenol and *p-tert*-nonylphenol), arylphenols (for example phenylphenol and naphthols) and divalent phenols (such as resorcinol and bisphenol A).

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3.2.2 Aldehydes

The most important aldehyde component is formaldehyde, which is used in various forms, including aqueous solution and solid paraformaldehyde, and also as compounds which give rise to formaldehyde. Other aldehydes (for example acetaldehyde, acrolein, benzaldehyde and furfural) are employed to a more limited extent, as also are ketones.

3.3 Types of phenolic resin

3.3.1 Unmodified phenolic resins

Unmodified phenolic resins are produced by condensation reactions of phenols with an aldehyde. Depending on factors such as the volume and molar ratio of the raw materials, the reaction conditions and the catalysts employed, two different classes of product are obtained: novolaks and resols.

3.3.1.1 Novolaks (two-step)

Novolaks are non-self-curing, soluble, fusible phenolic resins that remain stable when stored. The phenol nuclei are linked primarily by methylene bridges. Novolaks can be made to react further and crosslink by the addition of hardeners (see 3.6.2); heating is also usually necessary.

See also *novolak* in ISO 472.

3.3.1.2 Resols (one-step)

Resols are soluble, fusible phenolic resins which, in contrast to novolaks, contain methylol groups and methyleneether and sometimes also methylene-amine bridges. Resols are self-curing: they crosslink to give insoluble products when heated and/or mixed with catalysts, without addition of further reaction components. Resols are perishable and can be stored for a limited time only.

See also *resol* in ISO 472.

3.3.2 Modified phenolic resins

Phenolic resins can be modified by chemical reaction of the methylol or the phenolic hydroxyl groups and/or by physical dispersion in the modifying agent. Such modified phenolic resins can be either self-curing or non-self-curing, depending on their structure.

3.3.3 Phenolic addition resins

Phenolic resins can also be produced without condensation with formaldehyde by reaction of phenol with unsaturated hydrocarbons. Examples of these are phenol/acetylene and phenol/terpene resins, which are not always self-curing.

3.4 Forms of commercial product

Phenolic resins are manufactured and used in various forms, such as

- a) liquids;
- b) solids, as pelletized, flake, granular or finely powdered products;
- c) solutions or dispersions in water and/or organic solvents.

A so-called "aqueous solution" (it is not a real aqueous solution) is one that has a water content of more than 5 % (m/m) and an organic-solvent content of less than 5 % (m/m) (free phenol is not counted as organic solvent).

A solvent-containing phenolic resin solution is one that has more than 5 % (*mlm*) of organic solvents.

3.5 Degree of condensation

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Phenolic resins can be crosslinked by the use of heat and/or hardeners and/or satalysts. They pass through the following transitional stages of condensation: 16101502cb/iso-10082-1999

- A-stage: starting state (resol or novolak); liquid or fusible, and soluble in alcohol and acetone;
- B-stage: intermediate state (resitol); infusible, but still mouldable with heat and capable of swelling in alcohol or acetone;
- C-stage; final state (resite); infusible, and insoluble in alcohol or acetone.

See also A-stage, B-stage, C-stage, resite and resitol in ISO 472.

3.6 Catalysts and hardeners

3.6.1 Catalysts

Catalysts accelerate the crosslinking reaction. Both acid and basic compounds are suitable for this purpose.

3.6.2 Hardeners (crosslinking agents)

Formaldehyde, or compounds that release formaldehyde, for example hexamethylenetetramine (hexamine), can be used as hardeners. Resols and isocyanates can also be used as hardeners. Hardeners are generally added only to non-self-curing phenolic resins and cure by chemical reaction (three-dimensional crosslinking).

4 Test methods

4.1 Numerical list

ISO 60:1977, Plastics — Determination of apparent density of material that can be poured from a specified funnel.

ISO 565:1990, Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.

ISO 760:1978, Determination of water — Karl Fischer method (General method).

ISO 2555:1989, Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method.

ISO 2811-1:1997, Paints and varnishes — Determination of density — Part 1: Pyknometer method.

ISO 3146:—²⁾, Plastics — Determination of melting behaviour (melting temperature or melting range) of semicrystalline polymers by capillary tube and polarizing-microscope methods.

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 3675:1998, Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method.

ISO 4610:1977, Plastics — Vinyl chloride homopolymer and copolymer resins — Sieve analysis using air-jet sieve apparatus.

ISO 8618:1995, Plastics — Liquid phenolic resing loc Conventional determination of non-volatile matter.

ISO 8619:1995, Plastics — Phenolic resin powder — Determination of flow distance on a heated glass plate.

ISO 8974: —³⁾, Plastics — Phenolic resins — Determination of residual phenol content by gas chromatography.

ISO 8975:1989, Plastics — Phenolic resins — Determination of pH.

ISO 8987:1995, Plastics — Phenolic resins — Determination of reactivity on a B-transformation test plate.

ISO 8988:1995, Plastics — Phenolic resins — Determination of hexamethylenetetramine content — Kjeldahl method and perchloric acid method.

ISO 8989:1995, Plastics — Liquid phenolic resins — Determination of water miscibility.

ISO 9020:1994, Binders for paints and varnishes — Determination of free-formaldehyde content of amino resins — Sodium sulfite titrimetric method.

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

ISO 9397:1995, *Plastics* — *Phenolic resins* — *Determination of free-formaldehyde content* — *Hydroxylamine hydrochloride method.*

To be published. (Revision of ISO 3146:1985)

³⁾ To be published. (Revision of ISO 8974:1997)

ISO 9771:1995, Plastics — Phenolic resins — Determination of the pseudo-adiabatic temperature rise of liquid resols when cured under acid conditions.

ISO 9944:1990, Plastics — Phenolic resins — Determination of electrical conductivity of resin extracts.

ISO 11401:1993, Plastics — Phenolic resins — Separation by liquid chromatography.

ISO 11402:—⁴), Phenolic, amino and condensation resins — Determination of free-formaldehyde content.

ISO 11409:1993, Plastics — Phenolic resins — Determination of heats and temperatures of reaction by differential scanning calorimetry.

ISO 12058 (both parts), Plastics — Determination of viscosity using a falling-ball viscometer.

4.2 Alphabetical list

	ISO standard	Subclause
Acid reactivity of phenolic resols	9771	4.3.2.3
Ash, determination of	3451-1	4.3.2.9
Conductivity, electrical, of phenolic resin extracts, measurement of	9944	4.3.1.7
Density, apparent, determination of		4.3.1.3
Density or relative density, determination of, hydrometer method REVIEW	3675	4.3.1.4
Density of paints and varnishes, determination of dards.iteh.ai)	2811	4.3.1.4
Differential scanning calorimetry, determination of heats and temperatures of reaction https://standards.iteh.ai/catalog/standards/sist/a5c582ab-7c0d-4a10-9'	11409 7e2-	4.3.2.2.3
Flow distance, on a heated glass plate, of phenolic resins, determination of	8619	4.3.2.1
Formaldehyde, free, content in amino resins, sodium sulfite titrimetric method	9020	4.3.2.6.2
Formaldehyde, free, content in condensation resins	11402	4.3.2.6.3
Formaldehyde, free, content in phenolic resins, hydroxylamine hydrochloride method	9397	4.3.2.6.1
Gel time under specific conditions by automatic apparatus, determination of	9396	4.3.2.2.2
Hexamethylenetetramine content of phenolic resins, Kjeldahl method and perchloric acid method	8988	4.3.2.7
Liquid chromatography of phenolic resins, separation of	11401	4.3.2.10
Melting behaviour of semi-crystalline polymers	3146	4.3.1.1
Non-volatile matter in liquid phenolic resins, determination of	8618	4.3.2.4
pH of phenolic resins, determination of	8975	4.3.1.6
Phenol, residual content in phenolic resins, gas chromatographic determination	8974	4.3.2.5
Reactivity on a B-transformation test plate	8987	4.3.2.2.1
Sieve analysis, air-jet apparatus method	4610	4.3.1.2

⁴⁾ To be published. (Revision of ISO 11402:1993)