

# INTERNATIONAL STANDARD

**ISO**  
**10082**

First edition  
1991-05-15

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## Plastics — Phenolic resins — Definitions and test methods

**iTeh STANDARD PREVIEW**  
*Plastiques — Résines phénoliques — Définitions et méthodes d'essai*  
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ISO 10082:1991

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Reference number  
ISO 10082:1991(E)

## 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.

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.

International Standard ISO 10082 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It includes the contents of Technical Report ISO/TR 8244:1988.

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International Organization for Standardization  
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

## Introduction

This International Standard gives an overview of the different types of phenolic resin used in the manufacture of thermosetting plastics (section 2) and a summary of available test methods (section 3).

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# Plastics — Phenolic resins — Definitions and test methods

## Section 1: General

### 1.1 Scope

This International Standard defines, in section 2<sup>1)</sup>, terms relating to the chemical structure of phenolic resins, their physical state and their degree of condensation and polycondensation.

In section 3, a summary of test methods is given. These methods apply to the determination of properties which are technical significant for the production, processing and application of phenolic resins as described in section 2. 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.

### 1.2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary*.

1) Previously published as ISO/TR 8244:1988, *Plastics — Phenolic resins — Basis for classification*.

## Section 2: Definitions

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

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

### 2.1 General

**2.1.1** 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)<sup>2)</sup>.

NOTE 1 Resins in which the original properties are so changed by the modification (for example by rosin) that they resemble more closely those of the modifying medium are not regarded as phenolic resins.

**2.1.2** Phenolic resins may be classified in a number of ways, such as by

- a) the type of raw material (see clause 2.2);
- b) the type of resin produced (see clause 2.3);
- c) the type of modifying agent (see 2.3.2);
- d) the physical form of the commercial product (see clause 2.4);
- e) the degree of condensation (see clause 2.5);
- f) the type of catalyst (see 2.6.1);
- g) the type of hardener (see 2.6.2).

### 2.2 Raw materials

#### 2.2.1 Phenols

Besides unsubstituted phenol, derivatives of phenol used for the manufacture of phenolic resins 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).

#### 2.2.2 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.

### 2.3 Types of phenolic resin

#### 2.3.1 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.

##### 2.3.1.1 Novolaks (two-step)

Novolaks are non-self-curing, soluble, fusible phenolic resins that remain stable when stored, the phenol nuclei of which are linked primarily by methylene bridges. Novolaks can be made to react further and crosslink by the addition of hardeners (see 2.6.2); heating is also usually necessary.

See also *novolak* in ISO 472.

##### 2.3.1.2 Resols (one-step)

Resols are soluble, fusible phenolic resins which, in contrast to novolaks, contain methylol groups and methylene-ether and sometimes also methylene-amine bridges. Resols are self-curing; they crosslink into 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.

#### 2.3.2 Modified phenolic resins

Phenolic resins can be modified by chemical reaction of the methylol or the phenolic hydroxyl groups and/or by physical dispersion of the modify-

2) 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.

ing agent. Such modified phenolic resins can be either self-curing or non-self-curing, depending on their structure.

### 2.3.3 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.

## 2.4 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 % (*m/m*) of organic solvents.

## 2.5 Degree of condensation

Phenolic resins can be crosslinked by the use of heat and/or hardeners and/or catalysts. They pass through the following transitional stages of condensation:

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*; *resitol* in ISO 472.

## 2.6 Catalysts and hardeners

### 2.6.1 Catalysts

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

### 2.6.2 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).

## Section 3: Test methods

### 3.1 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 2811:1974, *Paints and varnishes — Determination of density.*

ISO 3146:1985, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers.*

ISO 3219:1977, *Plastics — Polymers in the liquid emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate.*

ISO 3451-1:1981, *Plastics — Determination of ash — Part 1: General methods.*

ISO 3675:1976, *Crude petroleum and liquid petroleum products — Laboratory determination of density or relative density — Hydrometer method.*

ISO 8618:1987, *Plastics — Liquid phenolic resins — Conventional determination of non-volatile matter.*

ISO 8619:1988, *Plastics — Phenolic resin powder — Determination of flow distance on a glass plate.*

ISO 8620:1986, *Plastics — Phenolic resin powder — Sieve analysis using air-jet sieve apparatus.*

ISO 8974:1988, *Plastics — Phenolic resins — Determination of residual phenol content by gas chromatography.*

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

ISO 8987:1988, *Plastics — Phenolic resins — Determination of reactivity on a B-transformation test plate.*

ISO 8988:1989, *Plastics — Phenolic resins — Determination of hexamethylenetetramine content.*

ISO 8989:1988, *Plastics — Liquid phenolic resins — Determination of water miscibility.*

ISO 9371:1990, *Plastics — Phenolic resins in the liquid state or in solution — Determination of viscosity.*

ISO 9396:1989, *Plastics — Phenolic resins — Determination of the gel time at a given temperature using automatic apparatus.*

ISO 9397:1989, *Plastics — Phenolic resins — Determination of free formaldehyde content.*

ISO 9771:1989, *Plastics — Phenolic resins — Determination of reactivity of resols under acid conditions.*

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

### 3.2 Alphabetical list

	ISO	Sub-clause
Acid reactivity of phenolic resols	9771	3.3.2.3
Ash, determination of	3451-1	3.3.2.9
Conductivity, electrical, phenolic resins, measurement of	9944	3.3.1.7
Density, apparent, determination of	60	3.3.1.3
Density or relative density, determination of, hydrometer method	3675	3.3.1.4
Density, paints and varnishes, determination of	2811	3.3.1.4
Flow distance on a glass plate, phenolic resins, determination of	8619	3.3.2.1
Formaldehyde, free, content in phenolic resins	9397	3.3.2.6
Gel time at a given temperature by automatic apparatus, determination of	9396	3.3.2.2.2
Hexamethylenetetramine content in phenolic resins	8988	3.3.2.7
Melting behaviour of semi-crystalline polymers	3146	3.3.1.1



Non-volatile matter in liquid phenolic resins, determination of	8618	3.3.2.4	is not limited to one temperature but extends over a range of temperatures.
pH, phenolic resins, determination of	8975	3.3.1.6	The limits of the melting range consist of a "sinter point" (also known as the "stick point") and the "melted stage". The challenge is to establish exact definitions of these "points" for interpretation by various operators.
Phenol, residual, content in phenolic resins, gas-chromatographic determination	8974	3.3.2.5	The repeatability of the method is good, but reproducibility may be poor; hence the necessity for agreement on the definitions of these "points".
Reactivity on a B-transformation test plate	8987	3.3.2.2.1	The melting range is the temperature interval between the "sinter point" and the "melted stage".
Sieve analysis, air-jet apparatus method	8620	3.3.1.2	The sinter point (stick point) is the temperature at which the first physical change is observed in the powdered resin.
Sieves, test sieves, nominal sizes of openings	565	3.3.1.2	The melted stage is the temperature at which the mass of the resin becomes totally fluid or (as opposed to the melting point of partially crystalline substances) the temperature at which the sintered mass of the resin becomes translucent (not transparent) and/or when the resin starts to shrink and separate from the capillary-tube walls.
Viscosity, liquid resins and emulsions or dispersions, Brookfield Test method	2555	3.3.1.5	The moisture content of the resin powder has an effect on the determination of the melting range. Since the melting range is to be determined on products as received, it is not permitted to subject the resin to drying before testing. However, for comparative tests, it may be desirable to take into account the moisture content. In such a case, it is possible to dry the resin powder to constant mass, or at least for 48 h, using diphosphorus pentoxide or a similar dehydrating agent.
Viscosity, phenolic resins, liquid or in solution	9371	3.3.1.5	
Viscosity, using rotary viscosimeter with definite speed gradients	3219	3.3.1.5	
Water determination, Karl Fischer method	760	3.3.2.8	
Water miscibility, phenolic resins, determination of	8989	3.3.1.8	Preparation of the sample:  Use a sample of resin in powder form.  When the resin is in lump or flake form, crush it in a mortar and sift the pulverized resin through a 250 µm mesh sieve. Take the sample from the material that passes through the 250 µm sieve.

### 3.3 Description of test methods

#### 3.3.1 Physical properties

##### 3.3.1.1 Determination of the melting range using the capillary method

The test shall be performed in accordance with ISO 3146, method A.

Principle of ISO 3146, method A:

A specimen is heated in a capillary tube, at a controlled rate, and monitored visually for change in shape.

Note for use with phenolic resins:

Phenolic resins are chemically not single substances, but mixtures or alloys of polymers. This is evident from their melting behaviour — the process

is not limited to one temperature but extends over a range of temperatures.

The limits of the melting range consist of a "sinter point" (also known as the "stick point") and the "melted stage". The challenge is to establish exact definitions of these "points" for interpretation by various operators.

The repeatability of the method is good, but reproducibility may be poor; hence the necessity for agreement on the definitions of these "points".

The melting range is the temperature interval between the "sinter point" and the "melted stage".

The sinter point (stick point) is the temperature at which the first physical change is observed in the powdered resin.

The melted stage is the temperature at which the mass of the resin becomes totally fluid or (as opposed to the melting point of partially crystalline substances) the temperature at which the sintered mass of the resin becomes translucent (not transparent) and/or when the resin starts to shrink and separate from the capillary-tube walls.

The moisture content of the resin powder has an effect on the determination of the melting range. Since the melting range is to be determined on products as received, it is not permitted to subject the resin to drying before testing. However, for comparative tests, it may be desirable to take into account the moisture content. In such a case, it is possible to dry the resin powder to constant mass, or at least for 48 h, using diphosphorus pentoxide or a similar dehydrating agent.

Preparation of the sample:

Use a sample of resin in powder form.

When the resin is in lump or flake form, crush it in a mortar and sift the pulverized resin through a 250 µm mesh sieve. Take the sample from the material that passes through the 250 µm sieve.

##### 3.3.1.2 Sieve analysis using air-jet sieve apparatus

The test shall be performed in accordance with ISO 8620.

Principle of ISO 8620:

A test portion of powdered resin is placed on a sieve in a closed container and subjected to an air stream produced by a rotating jet beneath the sieve and to suction downwards through the sieve. The test results are dependent on the size of the sieve (see ISO 565) used, the negative pressure and the duration of sieving.