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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.

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 reference

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.

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].

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[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).

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 methylene-ether 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 % (m/m) of organic solvents.

3.5 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* 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 semi-crystalline 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 resins — 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.*

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2) 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	60	4.3.1.3
Density or relative density, determination of, hydrometer method	3675	4.3.1.4
Density of paints and varnishes, determination of	2811	4.3.1.4
Differential scanning calorimetry, determination of heats and temperatures of reaction	11409	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

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

Sieves, test sieves, nominal sizes of openings	565	4.3.1.2
Viscosity of liquid resins and emulsions or dispersions, Brookfield Test method	2555	4.3.1.5
Viscosity using a falling-ball viscometer	12058 (both parts)	4.3.1.5
Viscosity using a rotary viscosimeter working at defined speed	3219	4.3.1.5
Water determination, Karl Fischer method	760	4.3.2.8
Water miscibility of phenolic resins, determination of	8989	4.3.1.8

4.3 Description of test methods

4.3.1 Physical properties

4.3.1.1 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 test sample is heated in a capillary tube at a controlled rate and monitored visually for change in shape.

Note for 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 main body of the resin becomes totally fluid or (as opposed to the melting point of partially crystalline substances) the temperature at which the sintered 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 influence on the melting range. Since the melting range is to be determined on products as received, it is not permitted to dry the resin 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 test sample:

Use a test sample of resin in powder form.

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

4.3.1.2 Sieve analysis using an air-jet sieve apparatus

The test shall be performed in accordance with ISO 4610.

Principle of ISO 4610:

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 used (see ISO 565), the underpressure and the duration of sieving.

4.3.1.3 Determination of apparent density

The test shall be performed in accordance with ISO 60.

Principle of ISO 60:

A special funnel is placed 20 mm to 30 mm above a 100 ml measuring cylinder. The lower orifice of the funnel is closed with a finger and the material poured into the funnel. The finger is removed and the material flows into the cylinder. When the cylinder is full, a straight edge is drawn across the top of the cylinder to remove excess material. The contents of the cylinder are then weighed.

Note for phenolic powder resins:

A change due to compaction can occur with finely milled or powdered phenolic resins as a result of storage and transportation. Screening through a vibratory sieve with a mesh aperture of 1 mm will eliminate the effect of this compaction on the test result. However, there may still be variations of up to 5 % in the measured values as a result of the previous history of the sample.

4.3.1.4 Determination of density

The test shall be performed in accordance with ISO 2811-1 (pyknometer method) or ISO 3675 (hydrometer method).

Unless otherwise specified, the determination shall be carried out at 23 °C. The results are expressed in grams per cubic centimetre to three places of decimals.

The pyknometer method may be used for all liquid resins. The hydrometer method is suitable for resins with a viscosity < 1 Pa·s.

4.3.1.5 Determination of viscosity

Three procedures are proposed for the determination of viscosity:

- a reference method: determination using a viscometer with a defined speed gradient, for example a plate/cone viscometer (ISO 3219);
- two verification methods: determination with a rotary viscometer (ISO 2555) and determination with a Hoeppler falling-ball viscometer [ISO 12058 (both parts)].

NOTE Since, in a number of cases, liquid phenolic resins or phenolic-resin solutions are non-Newtonian fluids, the measured values may be dependent on the test method. When carrying out comparative tests, it is therefore necessary to ensure that the same method is always used.

4.3.1.6 Determination of pH

The test shall be performed in accordance with ISO 8975.

Principle of ISO 8975:

The potential difference between a glass electrode and a reference electrode immersed in a solution is a function of the pH of the solution. The pH is determined by measuring the potential difference with a potentiometric device.

NOTE The pH determined with a glass electrode is, in principle, a measure of the hydrogen-ion activity, but not of the hydrogen-ion concentration. Since, in dilute aqueous solutions, the activity coefficient is close to unity, the difference between concentration and activity is small. In a medium containing little water, such as liquid phenolic resins and phenolic-resin solutions, the differences can, however, be considerable. In the case of alkali-containing phenolic resins, there is no precise relationship between the pH and the stoichiometric alkalinity.

4.3.1.7 Determination of electrical conductivity

The test shall be performed in accordance with ISO 9944.

Principle of ISO 9944:

An acetone/water mixture is added to a solution of the resin. After settling, the conductivity of the suspension above the resin is measured.

4.3.1.8 Determination of miscibility with water

The test shall be performed in accordance with ISO 8989.

Principle of ISO 8989:

The percentage by mass of water needed to obtain turbidity in the liquid phenolic resin is determined. The determination is performed at a temperature of 23 °C ± 0,1 °C, water being added to the resin until persistent turbidity remains after a minimum of 30 s agitation.

4.3.2 Chemical properties

4.3.2.1 Determination of flow distance on a heated glass plate

The test shall be performed in accordance with ISO 8619.

Principle of ISO 8619:

Tablets are first prepared under defined conditions and placed on a heated glass plate. The plate is inserted in a naturally ventilated oven which is kept at a constant temperature of 125 °C ± 1 °C. The plate is kept in the oven for 3 min in the horizontal position and 20 min in a tilted position. The flow distance is then measured.

4.3.2.2 Hardening characteristics of phenolic resins

The methods described give the reaction time required by a phenolic resin at a particular temperature under the specified test conditions to be transformed to the B-stage (see 3.5).

4.3.2.2.1 Determination of reactivity on a B-transformation test plate

The test shall be performed in accordance with ISO 8987.

Principle of ISO 8987:

The B-transformation time of a phenolic resin is determined on a hot plate at a specified temperature and under specified conditions. Two types of plate are described:

- a plate with depressions in the form of segments of spheres (method A);
- a flat plate without depressions (method B).

4.3.2.2.2 Determination of gel time under specific conditions by automatic apparatus

The test shall be performed in accordance with ISO 9396.

Principle of ISO 9396:

When phenolic resins polymerize, the resin passes through a critical point at which viscosity increases abruptly. An automatic apparatus is used to measure the time taken to reach this point.

The resin and a mixer rod are placed in a test tube. The rod is driven to execute a vertical reciprocating motion. The gel point is reached when an abrupt increase in viscosity causes the rod immersed in the resinous mass to lift the test tube with it, thus stopping a stopwatch and the apparatus.

4.3.2.2.3 Determination of heats and temperatures of reaction by differential scanning calometry

The test shall be performed in accordance with ISO 11409.

Principle of ISO 11409:

The heat flux (power) supplied to a test sample in a pressuretight steel container is measured as a function of time or temperature. The heating rate is preferably 5 °C ± 1 °C per minute. The difference is measured between the heat flux supplied to a sample holder containing the test sample and the heat flux supplied to a reference holder.

4.3.2.3 Acid reactivity of phenolic resols

The test shall be performed in accordance with ISO 9771.

Principle of ISO 9771:

A phenolic resol is mixed with an acid hardener and allowed to harden. The highest temperature reached by the test sample and the time taken to reach this temperature are recorded.

4.3.2.4 Determination of non-volatile matter

The test shall be performed in accordance with ISO 8618.

Principle of ISO 8618:

A test portion of resin is weighed into a disposable dish. The non-volatile matter is the percentage residue remaining when the volatile components are evaporated under specified conditions of temperature and time. The proposed temperature is 135 °C ± 1 °C. Special procedures for testing at 150 °C are described in the annex.

4.3.2.5 Gas-chromatographic determination of residual phenol content

The test shall be performed in accordance with ISO 8974.

Principle of ISO 8974:

A test portion of resin is dissolved in a suitable solvent, preferably acetone, and the phenol content is determined by gas chromatography, using *m*-cresol as the internal standard.

4.3.2.6 Free-formaldehyde content of condensation resins

Table 1 — Selection of procedure

Procedure	Suitable for testing of
Hydroxylammonium chloride procedure	Phenolic resins, furan resins (unmodified with urea or melamine resin)
Sulfite procedure	Urea resins, melamine resins, furan resins, urea-melamine resins, furan-urea resins
KCN procedure	Melamine-phenolic resins, urea-phenolic resins, urea-melamine-phenolic resins

4.3.2.6.1 Free formaldehyde by hydroxylamine hydrochloride method

The test shall be performed in accordance with ISO 9397.

Principle of ISO 9397:

The free formaldehyde present in a test portion is converted to the oxime by the action of hydroxylamine hydrochloride. The hydrochloric acid formed during the reaction is back-titrated by potentiometric titration using a solution of sodium hydroxide.

4.3.2.6.2 Free formaldehyde by sodium sulfite titrimetric method

The test shall be performed in accordance with ISO 9020.

Priniciple of ISO 9020:

Free formaldehyde and alcohol-formaldehyde semiacetals in a test portion are reacted with excess sodium sulfite solution at a temperature of 0 °C to form hydroxymethane sulfonate. The excess sodium sulfite is titrated with iodine solution. The hydroxymethane sulfonate is decomposed with sodium carbonate solution and the sodium sulfite liberated is titrated with iodine solution.

4.3.2.6.3 Free formaldehyde by KCN method

The test shall be performed in accordance with ISO 11402.

Principle of ISO 11402:

Free formaldehyde in a test portion is converted to cyanohydrin with excess potassium cyanide. The excess potassium cyanide is subsequently back-titrated with mercury(II) nitrate solution using diphenylcarbazone as an indicator.

4.3.2.7 Hexamethylenetetramine (HMTA) content

The test shall be performed in accordance with ISO 8988.

Principle of ISO 8988:

Two methods are described:

- **Kjeldahl method**

  The HMTA is converted to ammonium sulfate by decomposing it by heating in the presence of concentrated sulfuric acid and a catalytic mixture. The ammonium sulfate is subsequently converted to sodium sulfate and ammonia by adding sodium hydroxide. The ammonia is driven off by steam distillation and dissolved in a solution of hydrochloric acid. The excess hydrochloric acid is titrated with sodium hydroxide in the presence of an indicator.

— **Perchloric acid method**

The tertiary-amine group in HMTA is titrated with perchloric acid.

The two methods are equivalent. The Kjeldahl method is not applicable if there are other components containing nitrogen in the phenolic resin. The perchloric acid method is only applicable of there are no other basic or acidic additives in the resin.

4.3.2.8 Measurement of water content by the Karl Fischer method

The test shall be performed in accordance with ISO 760.

For the determination of water in phenolic resins, modified Karl Fischer reagents may be used. For example, reagents free from pyridine and reagents suitable for working in the presence of aldehydes and ketones may be used.

4.3.2.9 Determination of ash

The test shall be performed in accordance with ISO 3451-1:1997, method A.

Principle of ISO 3451-1:1997, method A:

The ash is determined by direct calcination, i.e. by burning off the organic matter and heating the residue at high temperature until constant mass is reached.

4.3.2.10 Separation by liquid chromatography

The test shall be performed in accordance with ISO 11401.

Principle of ISO 11401:

Three methods are described:

Method A: Gel-permeation chromatography

A test sample of the phenolic resin is dissolved in tetrahydrofuran and the molecular-mass distribution is determined by separation on columns with polymer gels having different pore diameters.

Method B: High-performance liquid chromatography on polar columns

A test sample of the phenolic resin is dissolved in a suitable solvent and separated on the polar column. The eluent is a solvent blend run with a concentration gradient. Novolaks and tetrahydrofuran-soluble resols may be analysed.

Proposed solvent blend: tetrahydrofuran-heptane.

Method C: High-performance liquid chromatography on a non-polar (reversed-phase) column

A test sample of the phenolic resin is dissolved in a suitable solvent and separated on the non-polar column. The eluent is a solvent blend run with a concentration gradient.

Proposed solvent blend: water-methanol.

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