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Plastics — Phenolic resins — Determination of pH

Plastiques — Résines phénoliques — Détermination du pH

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Foreword

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Annex A of this International Standard is for information only.

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Plastics — Phenolic resins — Determination of pH

1 Scope

This International Standard specifies a method for the determination of the pH of liquid phenolic resins at the manufacturing stage or on the market.

NOTE 1 A variant of this method, applicable to solid phenolic resins, is described in annex A. With liquid resins having a viscosity greater than 2 Pa·s, it may be necessary to use this variant, which calls for a magnetic stirrer.

2 Principle

The potential difference between a glass electrode and a reference electrode immersed in the same solution is used to determine the pH.

3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Standard solutions, with acid pH ($\approx$ 4), neutral pH ($\approx$ 7) or alkaline pH ($\approx$ 9), or **buffer solutions**.

The following standard or buffer solutions are specified:

3.1 Potassium hydrogen phthalate, standard solution, $c(KCOOC_6H_4COOH) = 0{,}05$ mol/l, pH = 4,00 at 23 °C.

Dissolve 10,21 g of potassium hydrogen phthalate, previously dried by heating at 100 °C to 130 °C for 2 h, in water and make up to 1 000 ml.

This solution shall be stored in a glass vessel and kept away from any traces of acid or base; it may be kept longer by adding a crystal of thymol. Slight cloudiness in the solution indicates microbial contamination. The thymol is no longer effective and such a solution shall be discarded.

3.2 Hydrogen phosphate buffer solution, containing approximately 0,025 mol of potassium dihydrogen phosphate (KH_2PO_4) per litre and approximately 0,025 mol of sodium hydrogen phosphate (Na_2HPO_4) per litre, pH = 6,88 at 23 °C.

Dissolve 3,39 g of potassium dihydrogen phosphate (KH_2PO_4) and 3,53 g of sodium hydrogen phosphate (Na_2HPO_4), previously dried by heating at 110 °C to 130 °C for 2 h, in water and make up to 1 000 ml.

This solution may be kept longer by adding a crystal of thymol.

3.3 Sodium tetraborate (Borax), standard solution, $c(Na_2B_4O_7.10H_2O) = 0{,}01$ mol/l, pH = 9,22 at 23 °C.

Dissolve 3,80 g of sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$) in water and dilute to 1 000 ml.

4 Apparatus

4.1 pH-meter, accurate to 0,1 pH-units.

4.2 Electrodes

4.2.1 Measuring electrode: glass electrode.

4.2.2 Reference electrode: calomel electrode.

4.2.3 Maintenance of the electrodes

For all types of electrode, follow the manufacturer's instructions specifying the maintenance procedures necessary to keep the electrodes in the correct condition.

In particular, clean the glass electrode at regular intervals as directed by the manufacturer.

NOTE 2 When not in use over a long period, glass electrodes and certain calomel electrodes may be stored in the dry condition if permitted by the manufacturer.

For reconditioning, soak the glass electrode in acidified distilled water (pH 4 to 5) for several hours and the calomel electrode in saturated potassium chloride solution.

Before each determination, wash the electrodes with water and leave them to soak in water for at least 2 min before making any measurements.

5 Procedure

5.1 Test temperature

Carry out the test at 23 °C ± 0,5 °C.

5.2 Calibration of the pH-meter/electrode unit

Carry out the calibration in accordance with the manufacturer's instructions.

For the calibration, use two of the standard or buffer solutions (clause 3) which bracket the assumed value to be measured.

5.2.1 Adjustment of the apparatus

Rinse the electrodes with water and lightly wipe the electrode tips (with filter paper, for example) to remove any excess water.

Rinse the electrodes with one of the standard or buffer solutions by pouring the liquid down the outside of the stem.

Pour an adequate volume of the same standard or buffer solution into the clean, dry measuring vessel and immerse the electrodes in the solution.

Set the pH-meter reading to the pH-value of the standard or buffer solution, taking into account its temperature.

Remove the electrodes and discard the standard or buffer solution contained in the measuring vessel.

5.2.2 Verification of the response accuracy

Rinse the electrodes with water and then with the second standard or buffer solution, as described in 5.2.1.

Wash the measuring vessel with water (or use another clean, dry vessel), rinse it with the second standard or buffer solution, pour an adequate volume of the solution into the vessel and immerse the electrodes in the solution.

Record the pH-meter reading without changing the setting of the apparatus and, in particular, without touching the temperature correction or calibration/buffer control.

- If this reading is within the permissible tolerances (± 0,1 pH-units) at the known pH-value of the standard or buffer solution, taking into account its temperature, the apparatus is in operating condition and is suitably calibrated.
- If this is not the case, determine the cause (such as operating error, defective electrode, incorrect temperature correction) and remedy the defect.

5.3 Determination

5.3.1 Test portion and preparation of the test solution

Allow the temperature of the resin to stabilize at 23 °C ± 0,5 °C .

Prepare a 50 % (*m*/*m*) solution of the resin in water.

If phase separation occurs, wait until a sufficient volume of the aqueous phase for measurement has separated out in a dropping funnel. Transfer the aqueous phase to a graduated flask and carry out the measurement.

5.3.2 Measurement of the pH

Once the apparatus has been calibrated, wash the electrodes (4.2) and the measuring vessel, first with water, then with the test solution, as described in 5.2.1 and 5.2.2. Mix the test solution, pour an adequate volume of it into the measuring vessel (another dry, clean vessel may also be used) and immerse the electrodes in the solution. Check that the pH-meter reading is stable, and, if so, record it.

Repeat these operations with a fresh portion of the test solution.

- If the new reading of the pH-meter is identical to the previous one, or only differs from it by 0,2 pH-units, the test is complete (unless otherwise indicated in special standards).
- If this is not the case, carry out these operations a third time with a fresh portion of the test solution, carrying out all the checks necessary to determine the source of the error. If this third measurement does not give a conclusive result, repeat all the operations, including the calibration.

In order to determine the pH of a solution likely to change with time, particularly when in contact with the carbon dioxide in the air, proceed under the appropriate conditions (purge with a stream of nitrogen, for example).

6 Expression of results

Calculate the mean of the two measurements carried out in accordance with 5.3.2 and round to the nearest 0,1 pH-units.

7 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) a complete identification of the resin tested;

c) the test result, calculated as specified in clause 6, as well as the test temperature if the latter is not 23 °C ± 0,5 °C;

d) the date of the test.

Annex A
(informative)

Plastics — Solid phenolic resins — Determination of pH

A.1 Scope

This annex describes a method for determining the pH of solid phenolic resins at the manufacturing stage or on the market.

A.2 Principle

Preparation of an organo-aqueous solution, using a mixture of solvents previously neutralized to pH 7, and measurement of the pH of this solution with a pH-meter.

A.3 Reagents

The reagents specified in clause 3, and

A.3.1 Hydrochloric acid, solution, $c(\mathrm{HCl}) \approx 0{,}01$ mol/l.

A.3.2 Sodium hydroxide, solution, $c(\mathrm{NaOH}) \approx 0{,}01$ mol/l.

A.3.3 Solvent mixture, with the following composition:

Water:	20 g ± 1 g
Methanol:	40 g ± 1 g
Acetone:	40 g ± 1 g

NOTE 3 If the solid phenolic resin is insoluble in the above solvent mixture, water and toluene or water and xylene mixtures may be used.

A.4 Apparatus

The apparatus specified in clause 4, and

A.4.1 Magnetic mixer, with bar magnet.

A.4.2 Balance, accurate to 1 g.

A.5 Procedure

A.5.1 Calibration of the pH-meter/electrode unit

Calibrate using two standard or buffer solutions (clause 3) with an acid and neutral pH, respectively.

A.5.2 Neutralization of the solvent mixture

Place 80 g ± 1 g, weighed using the balance (A.4.2), of the solvent mixture (A.3.3) in a 250 ml beaker. Mix using the magnetic mixer (A.4.1) and allow the temperature of the mixture to stabilize at 23 °C ± 0,5 °C. Immerse the electrodes of the pH-meter in the beaker and, if necessary, adjust the pH of the mixture to 7,0 ± 0,1 using either the sodium hydroxide solution (A.3.2) or the hydrochloric acid solution (A.3.1).

A.5.3 Determination

A.5.3.1 Test portion and preparation of the test solution

Introduce 20 g ± 1 g, weighed using the balance (A.4.2), of the finely ground solid resin into the 250 ml beaker containing 80 g of the solvent mixture neutralized as described in A.5.2. Mix using the magnetic mixer (A.4.1) until the resin is completely dissolved and allow the temperature of the solution to stabilize at 23 °C ± 0,5 °C .

A.5.3.2 Measurement of the pH

Carry out the measurement as described in 5.3.2 on the solution prepared as specified in A.5.3.1.

A.6 Expression of results

See clause 6.

A.7 Test report

See clause 7.

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