

Australian/New Zealand Standard®

**Plastic roof and wall cladding materials —
Methods of test****Method 8: Determination of residual
styrene**

[ISO title: Reinforced plastic based on unsaturated polyester resins —
Determination of residual styrene monomer content]

PREFACE

This Standard was prepared by the Joint Standards Australia/ Standards New Zealand Committee PL/22 on Plastic Building Sheets. It is identical with and has been reproduced from ISO 4901—1985, *Reinforced plastics based on unsaturated polyester resins — Determination of residual styrene monomer content*.

Statements expressed in mandatory terms in notes to clauses are deemed to be requirements of this Standard.

As this Standard is reproduced from an international Standard, the following applies:

- (a) Its number does not appear on each page of text and its identity is shown only on the cover and title page.
- (b) In the source text, 'this International Standard' should read 'this Joint Australian/New Zealand Standard'.
- (c) A full point substitutes for a comma when referring to a decimal marker.

There are no Australian, New Zealand or Joint Standards that correspond to the following referenced International Standards:

ISO	
1172	Textile glass reinforced plastics — Determination of loss on ignition
2561	Plastics — Determination of residual styrene monomer in polystyrene by gas chromatography

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METHOD

Reinforced plastics based on unsaturated polyester resins — Determination of residual styrene monomer content

1 Scope and field of application

This International Standard specifies a method of determining the residual styrene monomer content in reinforced plastics based on unsaturated polyester resins in the polymerized state (hereinafter called *UP resins in the polymerized state*), by gas chromatography. The residual styrene monomer content is an important criterion in evaluating the degree of cure of UP resins in the polymerized state. This method is not applicable to UP resins of high chemical resistance.

NOTE — If other volatile aromatic hydrocarbon monomers are present in significant quantities, see ISO 2561 for the procedure to be followed in their determination.

This International Standard also specifies a method of determining residual styrene monomer by the Wijs method for laboratories in which gas chromatography is not available.

2 References

ISO 1172, *Textile glass reinforced plastics — Determination of loss on ignition*.

ISO 2561, *Plastics — Determination of residual styrene monomer in polystyrene by gas chromatography*.

3 Determination by gas chromatography

3.1 Principle

Extraction of the styrene from the UP resin in the polymerized state using dichloromethane, followed by determination by gas chromatography.

3.2 Reagents

During the analysis, use only reagents of recognized analytical grade.

3.2.1 Dichloromethane.

3.2.2 Methanol.

3.2.3 *n*-Butylbenzene.

3.2.4 Styrene, freshly distilled and stored at 0 °C until used.

When mixed with an equal volume of methanol, the styrene shall give a clear solution.

3.2.5 Polyethylene glycol, relative molecular mass 15 000 to 20 000. (Carbowax, 15 to 20 M, has been found satisfactory.)

3.2.6 Diatomaceous earth, particle size 210 to 250 µm. (Celite, acid-washed grade, has been found satisfactory.)

3.2.7 Helium, hydrogen, and **air**, as carrier and fuel gases for gas chromatography.

For use with the flame ionization detector, nitrogen may be applied as the carrier gas instead of helium.

NOTE — Changes in carrier gas are permissible only if the replacement gas has been demonstrated to give the same results.

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 Cutting device, consisting of a water-cooled diamond blade for cutting the UP resin in the polymerized state to strips of width 1 to 2 mm.

3.3.2 Gas chromatograph, with injection port for liquid samples, flame ionization detector, and recorder.

Some other type of ionization detector may be used and, in exceptional cases, a thermal conductivity detector. The latter, however, has lower sensitivity.

3.3.3 Microsyringe, of capacity 1 to 50 µl.

3.3.4 Analytical balance, accurate to 1 mg.

3.4 Preparation of sample

3.4.1 Cutting

Polymerized UP resin pieces of any shape that will permit the production of strips of width 1 to 2 mm may be used. Cut the polymerized pieces into strips of width

1 to 2 mm. Dry the strips and break into pieces of length about 10 mm. During cutting and drying, avoid any overheating which could modify the styrene monomer content.

3.4.2 Preparation of solvent for extraction

Weigh 600 ± 1 mg of *n*-butylbenzene (3.2.3) into a flask of capacity 25 ml, transfer quantitatively into a one-mark volumetric flask of capacity 1 000 ml, and dilute to the mark with dichloromethane (3.2.1). During the dilution process, maintain the liquid at $20 \pm 0,5$ °C.

3.4.3 Extraction of styrene

Depending on the styrene monomer content expected, weigh, to the nearest 1 mg, 1 to 2 g of the sample (3.4.1) and transfer into a conical flask of capacity 50 ml. Pour $15 \pm 0,1$ ml of the extraction solvent (3.4.2) over the sample and allow the suspension to stand for 15 to 20 h, with occasional shaking, keeping the flask closed. Then filter quickly, using a water-jet pump, and collect the filtrate.

3.4.4 Incineration of filtered residue

If the styrene monomer content is to be calculated on the resin content of UP resins in the polymerized state containing glass fibre or mineral fillers, incinerate the filtered residue (3.4.3) according to ISO 1172 (the final total mass after incineration is designated m_i).

If necessary, another piece of the test sample may be used instead of the filtered residue.

3.5 Procedure

Three test portions of the sample (3.4) shall be analysed.

3.5.1 Preparation of calibration mixtures for gas chromatography

Weigh, to the nearest 1 mg, 600 mg of *n*-butylbenzene with an appropriate amount of styrene and transfer quantitatively into a one-mark volumetric flask of capacity 1 000 ml and dilute to the mark with a mixture of 2 parts by volume dichloromethane (3.2.1) and 1 part by volume methanol (3.2.2). Recommended amounts of styrene are as follows: 100, 200, 500, 800 and 1 000 mg. During the dilution, maintain the liquid at $20 \pm 0,5$ °C.

3.5.2 Gas chromatographic operating conditions

Column: metal or glass tubing of length 4,5 m and inner diameter 4 mm are recommended. The column is packed with a stationary phase of diatomaceous earth (3.2.6) which is coated with 10 % (m/m) polyethylene glycol (3.2.5).

NOTE - Methods of coating and packing are not specified, but may be chosen so as to obtain satisfactory separating efficiency of the column. The column shall be aged for 24 h at 150 °C with gas flow. Changes in column filling materials or column dimensions are permissible only if this has proved to give the same results.

Column temperature: 80 °C isothermal.

Temperature of injection port: 150 °C.

Temperature of detector compartment: 150 °C.

Carrier gas: helium (or nitrogen under the conditions indicated in 3.2.7).

Flow rate of carrier gas: 100 ml/min

Flame ionization detector, in which the hydrogen and air flow rates are adjusted to give

- high sensitivity of response;
- linear response over the range of concentrations being measured;
- only insignificant effect of small changes in flow rates on response or sensitivity.

Recorder speed: 0,5 cm/min is recommended.

3.5.3 Gas chromatogram of test and calibration mixtures

According to the sensitivity of the gas chromatograph used, inject a suitable volume of the test solution (prepared according to 3.4.3) or the calibration mixture (prepared according to 3.5.1). The injected volume is not critical for the calculation of results, but shall be identical for corresponding samples and calibration mixtures. Operate the gas chromatograph until the *n*-butylbenzene is completely eluted.

3.5.4 Evaluation of peaks of gas chromatogram

The retention times of styrene and *n*-butylbenzene shall be known, at least in relation to each other. The exact values vary according to the density of the column, the age of the column, and other gas chromatographic parameters.

NOTE - The approximate retention time of styrene at the conditions specified in 3.5.2 is 16,5 min and of *n*-butylbenzene (standard) is 20,5 min; the approximate retention time of styrene relative to *n*-butylbenzene is 0,80.

The peak areas of styrene and *n*-butylbenzene shall be determined by one of the following methods:

- electronic integration;
- estimation of area, A , on the basis of the equation ¹⁾

$$A = hw_{1/2}$$

1 For terms and definitions see: IUPAC. Preliminary recommendation on nomenclature and presentation of data in gas chromatography. *Pure and Appl. Chem.* **1**, pp. 177-186 (1960).