
**Plastics — Unsaturated-polyester resins —
Determination of reactivity at 130 °C**

*Plastiques — Résines de polyesters non saturés — Détermination de la
réactivité à 130 °C*

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ISO 14848:1998

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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.

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 14848 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

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Introduction

The processing of unsaturated-polyester resins, as well as formulated products such as bulk moulding compound (BMC) and sheet moulding compound (SMC), depends on their reactivity, i.e. on their ability to polymerize under the action of a catalyst and under the effect of heat.

The reactivity of such resins may be determined by different methods, such as this International Standard which makes use of the variation in temperature as a function of time.

The use of the standardized conditions given in this International Standard make it possible to carry out comparisons between similar types of unsaturated-polyester resin.

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Plastics — Unsaturated-polyester resins — Determination of reactivity at 130 °C

1 Scope

This International Standard specifies a method of measuring

– the time required for the temperature of a solution of unsaturated-polyester resin and a catalyst, heated in a thermostatically controlled bath at 130 °C, to rise

- from 80 °C to 140 °C,
- from 80 °C to the maximum temperature reached;
- the maximum temperature reached;
- the speed of the reaction, determined by drawing a tangent at the point of inflection of the temperature/time curve.

It applies in particular to unsaturated-polyester resins processed at a temperature higher than 100 °C.

NOTE – The results are closely linked to the conditions. The aim of this International Standard is to define them as exactly as possible, particularly the following:

- a) The quantity and form of the resin sample being examined (test tube diameter, height of resin in the tube).
- b) The rate at which the sample is heated in the bath. For example, the use of a liquid having a relatively low heat capacity (ethylene glycol, silicone oil) leads to slower heating.
- c) The type of thermometer used. Thermometers having a response slower than that of a thermocouple, and resistance probes having too large a heat-sensitive element, are unsuitable.
- d) The position of the heat-sensitive element in the resin, both heightwise and relative to the axis of the test tube containing the resin.
- e) The nature of the catalyst.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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 15038:—¹⁾, *Plastics — Organic perester crosslinking agents for unsaturated-polyester thermosetting materials — Determination of active-oxygen content.*

EN 60584-2:1993, *Thermocouples — Part 2: Tolerances.*

3 Principle

Unsaturated-polyester resins are crosslinked by heating with a perester catalyst.

A resin/catalyst mixture (in the ratio 100 g to 1 g) is placed in a test tube kept in a thermostatically controlled bath at 130 °C and

- the rise in temperature recorded as a function of time for the entire duration of the reaction;
- the tangent drawn at the point of greatest slope of the recorded curve, thus giving an indication of the speed of the reaction.

4 Reagents

4.1 Catalyst: *tert*-butyl perbenzoate, having a purity of over 96 % (i.e. an active-oxygen content greater than 7,9 % determined in accordance with ISO 15038).

NOTE – It is advisable not to store *tert*-butyl perbenzoate (m.pt. 8 °C) at a temperature

- lower than 10 °C (to avoid crystallization);

- higher than 30 °C (to avoid deterioration in the quality of the product).

5 Apparatus

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5.1 Thermostatically controlled bath, with a capacity between 4 litres and 5 litres, containing silicone oil having a viscosity between 100 mPa·s and 500 mPa·s at 23 °C determined in accordance with ISO 3219, maintained at a temperature of 130 °C ± 1 °C and equipped with a thermostat, a circulation pump and a stirrer located at a distance of 10 cm from the test tube (5.2). The stirring action shall be sufficient to maintain a uniform temperature in the bath.

5.2 Borosilicate-glass test tube, having an internal diameter of 18 mm, a length of at least 180 mm and a wall thickness of 1,2 mm ± 0,2 mm, for use with the centering device (5.5).

5.3 Type J iron-constantan thermocouple, with a 1,5 mm wide protective sheath, 0,25 mm ± 0,01 mm thick wires and a welded junction of maximum diameter 0,54 mm (reaction time 28/100 s), inserted in a 1,6 mm diameter metal guide fitted with a stop 17 cm from the end, and connected to a temperature recorder equipped with an automatic cold-source compensator (see figure 1).

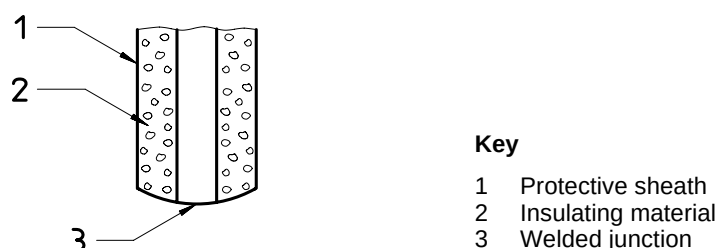


Figure 1 — Thermocouple

1) To be published.

5.4 Chart recorder, allowing the temperature to be read off the trace to within 1 °C and having a chart speed of 60 mm/min, or a **computer-based data-acquisition system**.

5.5 Centering device, with a diameter of $(18_{-0,04}^0)$ mm, made of PTFE for instance, for centering the thermocouple in the middle of the test tube (see figure 2).

Dimensions in millimetres

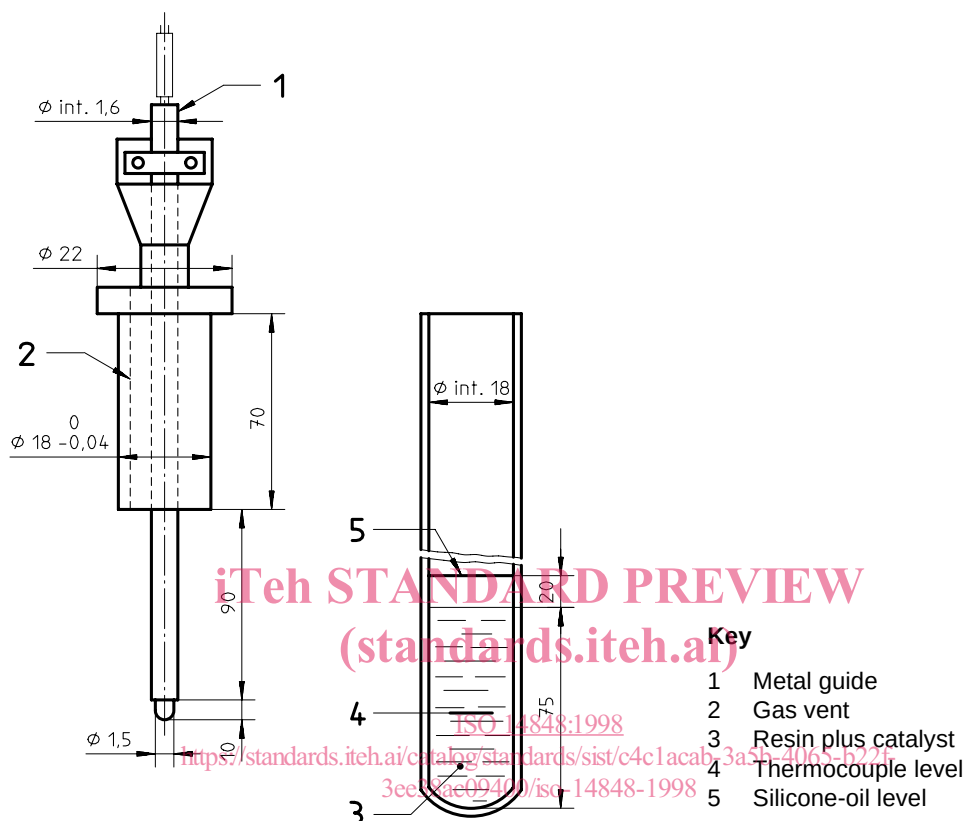


Figure 2 — Example of centering device

5.6 Balance, accurate to 0,01 g.

5.7 Stopwatch.

5.8 Beaker, capacity 150 ml.

5.9 Watchglass.

5.10 Glass rod.

6 Procedure

6.1 Weigh the glass beaker (5.8) and then weigh into it 100 g $\pm$ 0,1 g of resin at a temperature of 23 °C $\pm$ 2 °C.

6.2 Add a quantity of catalyst (4.1) such that the ratio of catalyst to resin is 1:100 by mass. Stir with the glass rod (5.10) to homogenize.

6.3 Cover the beaker with a watchglass (5.9) and allow to stand for 45 min $\pm$ 5 min.

Fill the test tube (5.2) to a height of 7,5 cm $\pm$ 0,2 cm.

Fit the centering device (5.5) in place and insert the thermocouple (5.3).

Immerse the test tube in the bath (5.1), maintained at 130 °C, ensuring that the surface of the resin is 2 cm below the level of the silicone oil in the bath.

To ensure the reproducibility of the determination, the centering device shall be checked every six months to confirm that its dimensions are still within the specified tolerances. If they are not, the device shall be replaced.

6.4 Record the temperature as a function of time throughout the duration of the reaction (see figure 3).

6.5 Remove the thermocouple-guide/thermocouple assembly immediately after the determination, when the recorder has stopped (see figure 3), to avoid resin adhering to the thermocouple.

6.6 Carry out at least two determinations.

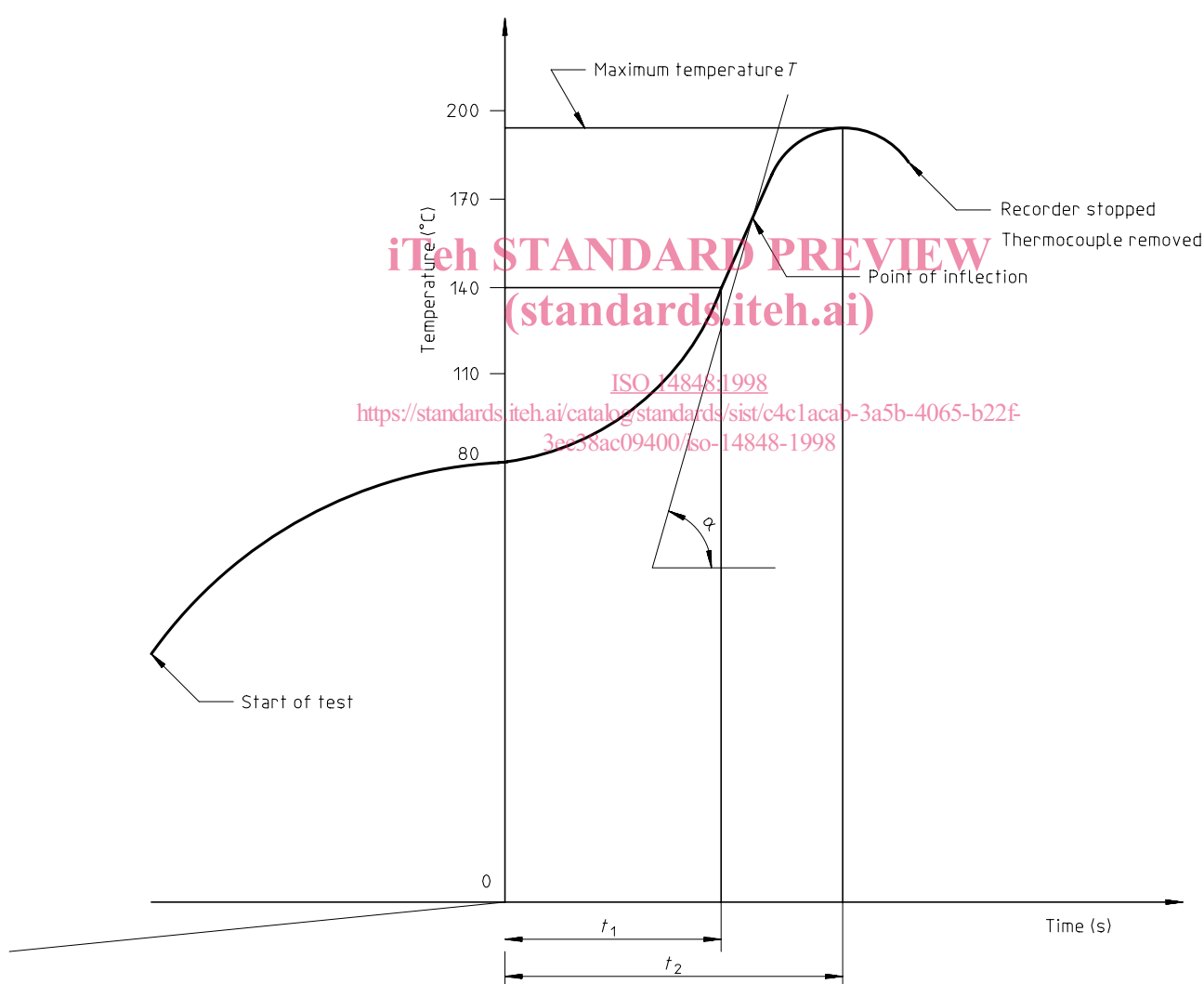


Figure 3 — Typical temperature-time curve

7 Expression of results

The reactivity of the resin is characterized by the following parameters:

- the maximum temperature T , in degrees Celsius, reached during the reaction;
- the time t_1 , in seconds, taken for the temperature to rise from 80 °C to 140 °C;
- the time t_2 , in seconds, taken for the temperature to rise from 80 °C to the maximum temperature.

In addition, the gradient ($\tan \alpha$) of the tangent drawn at the point of inflection of the curve provides an indication of the rate of the reaction, in degrees Celsius per second (see figure 3).

Measure the values of these parameters for each determination and calculate the arithmetic mean in each case.

8 Precision

Following a round robin organized in France in 1996, the precision of this method has been calculated to be as shown in table 1.

Table 1 — Precision data

	$t_1 \leq 60$ s			60 s $< t_1 \leq 120$ s	
	T	t_1	t_2	T	t_2
r	2 %	14 %	12 %	2 %	7,5 %
R	3 %	16 %	19 %	4 %	15 %
r = repeatability R = reproducibility Confidence level 95 %					

9 Test report

The test report shall contain the following information:

- a) a reference to this International Standard;
- b) all details necessary to identify the sample tested;
- c) the temperature at which the determination was carried out, if different from 130 °C;
- d) the type of thermocouple used;
- e) the results, expressed in accordance with clause 7;
- f) details of any operation not specified in this International Standard, and details of any incident which may have affected the results;
- g) the date and location of the test.