
International Standard**584**

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Unsaturated polyester resins — Determination of reactivity at 80 °C (conventional method)*Plastiques — Résines de polyesters non saturés — Détermination conventionnelle de la réactivité à 80 °C***First edition — 1982-12-01**

UDC 678.674 : 543.878**Ref. No. ISO 584-1982 (E)****Descriptors :** plastics, polyester resins, chemical analysis, determination, chemical reactivity.

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 584 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in July 1980.

It has been approved by the member bodies of the following countries :

Australia	Hungary	New Zealand
Austria	India	Poland
Canada	Iran	Romania
China	Ireland	South Africa, Rep. of
Czechoslovakia	Israel	Spain
Egypt, Arab Rep. of	Italy	Sweden
Finland	Japan	USA
France	Korea, Rep. of	USSR
Germany, F. R.	Netherlands	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Belgium
United Kingdom

This International Standard cancels and replaces ISO Recommendation R 584-1967 of which it constitutes a technical revision.

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0 Introduction

The processing of unsaturated polyester resins depends on their reactivity, i.e., their rate of polymerization in the presence of a catalyst. The evaluation of their reactivity can be carried out by various methods. This International Standard is limited to measuring certain characteristics relating to the change in temperature as a function of time of a resin-catalyst mix. Using arbitrarily standardized test conditions, these characteristics allow comparison of similar types of unsaturated polyester resins.

The results obtained in this test are dependent upon the following test conditions :

- a) quantity and spatial parameters of the test portion examined (test-tube diameter, height of resin in the tube);
- b) speed of heating the test portion placed in a temperature controlled bath. For example, the replacement of water in the bath by another fluid with a lower heat capacity (glycol, oil, etc.) leads to slower heating;
- c) type of temperature measuring element placed in the test portion and its ability to respond to rapid changes in temperature. Thermometers with a slow response and thermocouples with large junctions between the resistance leads are unsatisfactory;
- d) accurate centring of the thermocouple in the test portion, both in height and in line with the axis.

These conditions which are mandatory are specified in the text of this International Standard.

1 Scope and field of application

This International Standard specifies a method for the determination of the reactivity of unsaturated polyester resins as characterized by the maximum temperature reached by a solution of unsaturated polyester resin and catalyst when heated in a water-bath at 80 °C, by the time taken for the temperature of

the mixture to rise from 65 °C to this maximum temperature, and also, if the maximum temperature exceeds 90 °C, by the time taken for the temperature to rise from 65 to 90 °C.

These characteristics give an indication of the processing behaviour of unsaturated polyester resins and can be used to compare resins of similar type. As the test results depend very much on the conditions under which the test is carried out, these conditions are described in detail.

NOTES

- 1 If the method is used to test commercial benzoyl peroxide catalyst, the results should be compared with those obtained for the same resin using pure benzoyl peroxide.
- 2 By agreement between the interested parties, certain test conditions can be modified : such as the test temperature (temperature of the thermostatically controlled bath) and the quantity and type of catalyst system (mixtures of catalysts, combinations of catalysts and accelerators).

2 Principle

Placing of a mixture of 100 parts of resin with 1 part of catalyst in a test-tube of defined size. Heating of the test-tube and its contents in a water-bath at 80 °C for the duration of the reaction, and noting (or recording) the rise in temperature with time.

3 Reagent

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

Benzoyl peroxide, catalyst.

WARNING — Attention is drawn to the dangers involved in handling pure benzoyl peroxide.

A mixture of 50 % (*m/m*) of benzoyl peroxide catalyst in dibutyl phthalate having an active oxygen content between 3,25 and 3,33 % (*m/m*), as measured by known analytical methods.

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4 Apparatus

4.1 Water-bath,¹⁾ maintained at $80 \pm 0,5$ °C and provided with a thermostat and a circulation pump or stirrer.

4.2 Test-tube, of borosilicate glass or any other glass giving the same results, of internal diameter 18 ± 1 mm, length 210 ± 5 mm and wall thickness $1 \pm 0,2$ mm.

4.3 Iron-constantan thermocouple, consisting of wires of $1 \pm 0,05$ mm thickness, with a welded junction of 2 mm diameter maximum and a meter for measuring temperatures up to 250 °C, graduated in divisions of 2 °C.

NOTE — It is desirable to use screened thermocouples that can be positioned centrally.

4.4 If available a **recorder**, permitting the readings of temperature on the graph paper to the nearest 1 °C and the time to the nearest 15 s.

4.5 Device, for locating the thermocouple centrally in the test-tube (see the figure).

4.6 Balance, accurate to 0,01 g.

4.7 Stop watch.

5 Procedure

5.1 Weigh $2 \pm 0,01$ g of the benzoyl peroxide-dibutyl phthalate mixture (clause 3) into a glass beaker of capacity 250 ml and add 100 ± 1 g of resin at a temperature between 15 and 30 °C. Continue stirring for 2 to 3 min.

5.2 Then immediately pour part of the mixture into the test-tube (4.2) to a height of 7 to 8 cm. Introduce the thermocouple into the mixture in a position on the axis of the tube and equidistant from the bottom and the surface of the mixture (see the figure). Then place the tube in the water-bath at $80 \pm 0,5$ °C, so that the surface of the resin is well beneath the level of the surface of the water.

5.3 If a recorder is used, start and allow the recorder to run during the complete reaction. Alternatively, note the maximum temperature reached, as well as the time taken for the temperature of the mixture, determined by the thermocouple, to rise from 65 °C to the maximum temperature, and in the case where this temperature is greater than 90 °C, the time to rise from 65 to 90 °C.

5.4 Repeat the test, beginning at 5.1, until two consecutive determinations do not differ by more than 5 °C in temperature and 10 % in time.

6 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 type of thermocouple used and whether screened or unscreened;
- d) the individual values of the maximum temperature reached in the two tests and the average value;
- e) the individual values of the time taken for the temperature to rise from 65 °C to the maximum temperature in the two tests and the average value;
- f) if the maximum temperature is above 90 °C, the individual values of the time taken for the temperature to rise from 65 to 90 °C in the two tests and the average value;
- g) if a recorder is used, the graphs giving the temperature plotted against time.
- h) any deviations from the standard test conditions specified in this International Standard, for example type and temperature of the heating medium, catalyst system used, thermocouple system used, etc. (see note 2 to clause 1).

1) The water in the bath may be replaced by another fluid agreed upon by the interested parties.

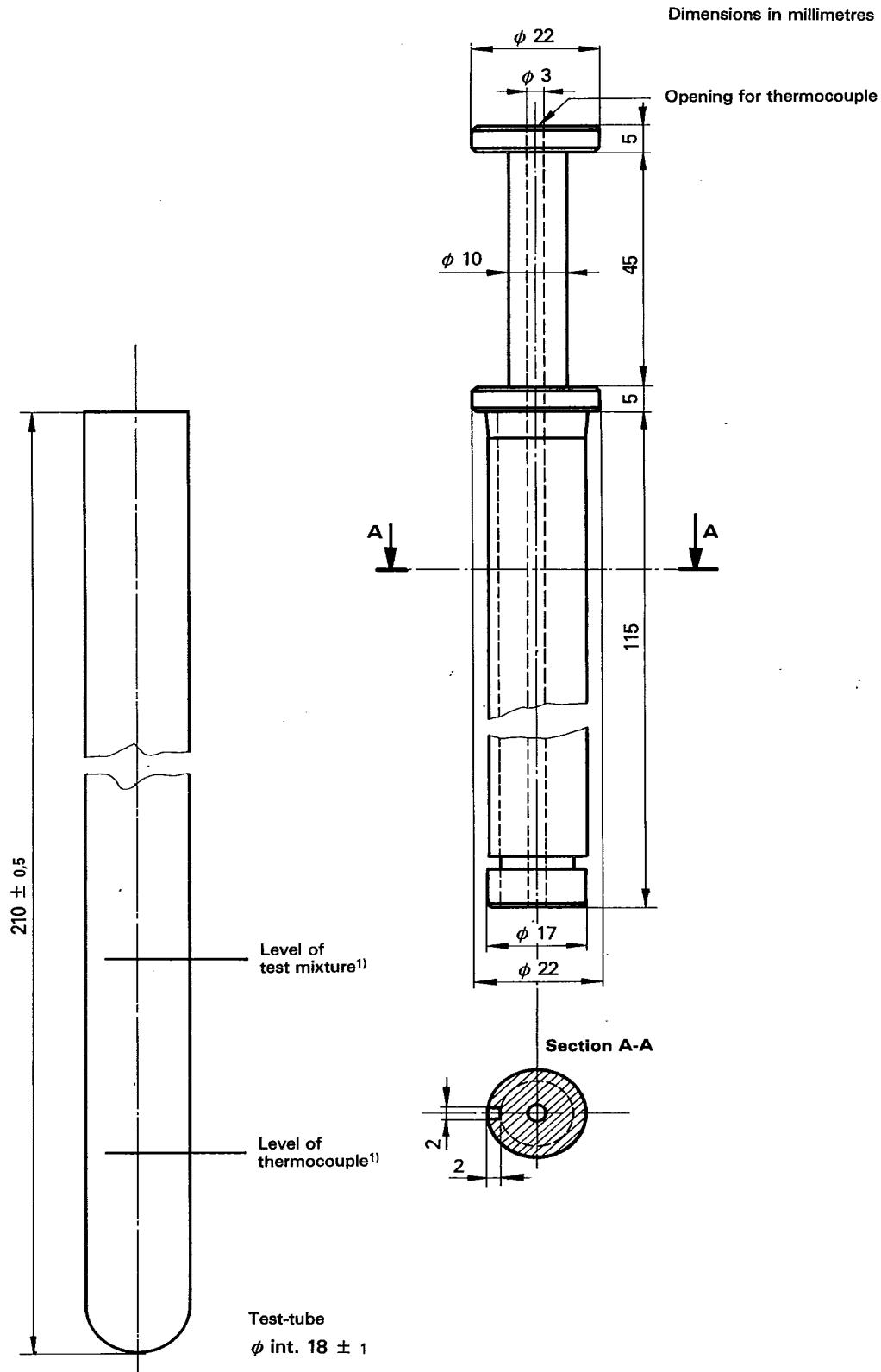


Figure — Example of device for locating the thermocouple centrally in the test-tube

1) The levels for the test mixture and the thermocouple shall be in accordance with the instructions in 5.2.