
**Plastics — Liquid epoxy resins —
Determination of tendency to crystallize**

*Plastiques — Résines époxydes liquides — Détermination de la tendance
à la cristallisation*



Foreword

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International Standard ISO 4895 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This second edition cancels and replaces the first edition (ISO 4895:1987), which has been technically revised.

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Printed in Switzerland

Introduction

The tendency of liquid epoxy resins to crystallize varies depending on factors such as basic composition, purity, additives, homogeneity and water content, in addition to external factors such as storage history and ambient temperature.

As it is rather difficult to indicate the tendency quantitatively by observation and comparison, the tendency is therefore to express the results by observing, at intervals, the changes in fluidity and appearance of samples.

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Plastics — Liquid epoxy resins — Determination of tendency to crystallize

1 Scope

This International Standard specifies a method for determining the tendency of liquid epoxy resins to crystallize. The tendency to crystallize is determined by observing, at specified time intervals, changes in fluidity and the onset of crystallization.

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 3001:1997, *Plastics — Epoxy compounds — Determination of epoxy equivalent*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*.

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

3 Principle

Calcium carbonate powder is mixed with the liquid epoxy resin dissolved in ethanol. The mixture is kept at a specified low temperature and observed at specified time intervals to compare the changes in fluidity and crystallization.

4 Reagents

4.1 Calcium carbonate, as specified in ISO 6353-3:1987, R 53.

4.2 Ethanol, as specified in ISO 6353-2:1983, R 11.

5 Apparatus

5.1 Refrigerator or cold enclosure, maintained at $10\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

5.2 Glass test tube, of capacity approximately 100 ml, 40 mm in diameter and 80 mm in height, with a polyethylene-covered stopper.

5.3 Glass rod, of hard glass, approximately 10 mm in diameter.

5.4 Oven.

5.5 Analytical balance.

6 Procedure

6.1 Weigh 20 g of liquid epoxy resin into the test tube (5.2).

6.2 Stopper the test tube and place it in the oven (5.4) at $60\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 16 h.

6.3 Cool the test tube to room temperature ($23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$), add 20 g of calcium carbonate (4.1) and 2 g of ethanol (4.2), and mix the sample thoroughly with the glass rod for 2 min.

6.4 Stopper the test tube again and place it in the vertical position in the refrigerator or cold enclosure (5.1) at $10\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

6.5 Observe the sample twice a day at a specified time (for example at an interval of 8 h during the day and 16 h during the night):

- | | |
|----------------------|------------------------|
| — start | 9 a.m. |
| — first observation | 5 p.m. |
| — second observation | 9 a.m. (next day) |
| — third observation | 5 p.m. (next day) |
| — fourth observation | 9 a.m. (the day after) |

Observe the sample in the following way:

Allow the test tube to warm to room temperature ($23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$), then place it in the horizontal position and leave it for 1 min.

- If the sample is still in the liquid state, record “a”.
- If the sample does not flow during the 1 min, record “b”.
- If the sample has completely solidified due to crystallization, record “c”.

After each observation, if the sample has not yet crystallized, mix thoroughly with the glass rod for 2 min.

Record the number of days which elapse to each stage of crystallization (see figure 1).

7 Expression of results

The tendency of a liquid epoxy resin to crystallize is expressed as the number of days which elapse until each stage specified in 6.5, "a", "b" and "c", is reached.

8 Precision

Precision in the generally accepted sense of the term for test methods cannot be determined here since it is a tendency which is being assessed.

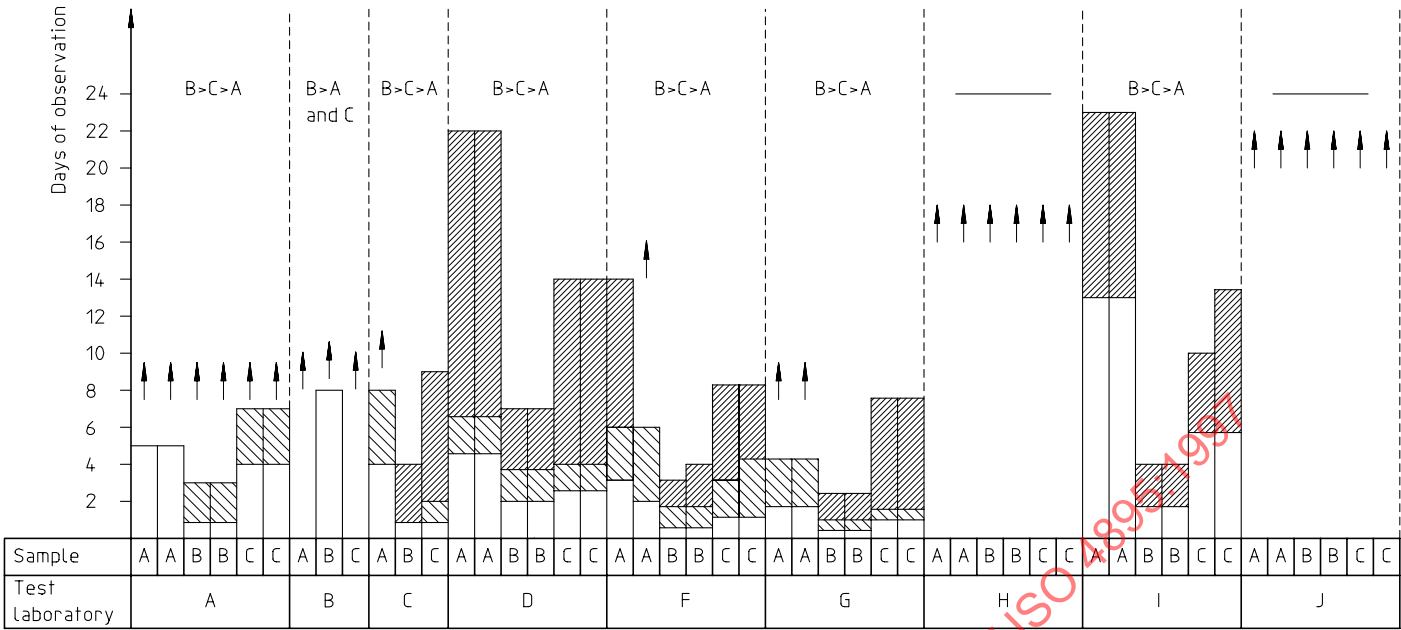
Inter-laboratory tests conducted in Japan have shown, however, that this method is currently the state of the art: it is more reliable and environmentally more acceptable than ISO 4895:1987.

9 Test report

The test report shall include the following particulars:

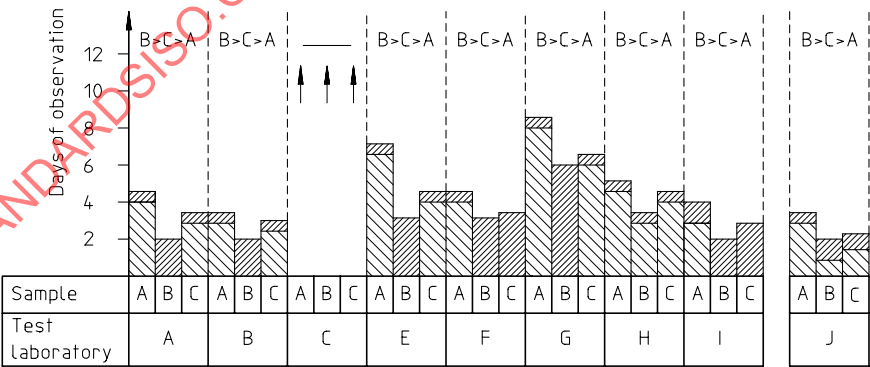
- a) a reference to this International Standard;
- b) all details necessary for identification of the sample tested;
- c) the results of the test;
- d) the date of the test;
- e) any deviation, by agreement or otherwise, from the specified procedure.

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An arrow ↑ indicates the time when the test was discontinued because of no further change.

Figure 1 — NBGE method of ISO 4895:1987



An arrow ↑ indicates the time when the test was discontinued because of no further change.

Owing to the presence of the CaCO_3 , it is not possible to judge when crystallization actually starts and hence not possible to differentiate between "contains no crystals" and "contains some crystals but flows" as in figure 1.

Figure 2 — CaCO_3 method (this edition of ISO 4895)