
**Thermosetting resin and UV curable
resin — Determination of shrinkage
by continuous measurement method**

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The use of resin first requires curing it under specific conditions that vary depending on the product specification. During this curing process, chemical reactions occur and volatiles evaporate, and so the resin shrinks. This can cause defects, strength reduction, and the deformation of the finished parts or products, especially in high precision required applications.

The conventional method which measures the shrinkage of resin based on specific gravity requires long measurement time and an amount of resin about a few cubic centimetres. This sample size is larger than what is actually used in many applications such as the epoxy encapsulation compounds for integrated circuits, resin coating or adhesive for electronic devices. In order to improve the quality control and further promote the technical advancement of high precision production, a convenient and high accuracy method for determining the shrinkage of resin is essential.

A totally new measurement method has been developed to meet this demand, allowing to measure curing shrinkage continuously with just a trace amount of resin. Moreover, since measurements are taken continuously, the curing behaviour of resin such as thermal expansion and thermal contraction are also observed. This measurement method is described in this document.

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Thermosetting resin and UV curable resin — Determination of shrinkage by continuous measurement method

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies the continuous measurement method of shrinkage for thermosetting resin and/or UV curable resin.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

UV curable resin

resin which is cured by receiving energy from UV rays

3.2

thermosetting resin

resin which is cured by receiving energy from heat

3.3

curing condition

UV irradiation and/or heating condition for curing resin

3.4

curing shrinkage

ratio of the change in resin volume due to curing process to the resin volume before curing

Note 1 to entry: Percentage of shrinkage due to curing of resin.

4 Principle

Cure the resin inside a sample container and continuously measure the changes of sample thickness. Since the horizontal cross-sectional area of resin sample remains constant due to the sidewalls of the sample container, the changes in sample volume are proportional to the changes in the sample

thickness. Therefore, shrinkage of resin is calculated from the changes in sample thickness, as shown in [Formula \(1\)](#).

$$A = (T_1 - T_2) / T_1 \times 100 \tag{1}$$

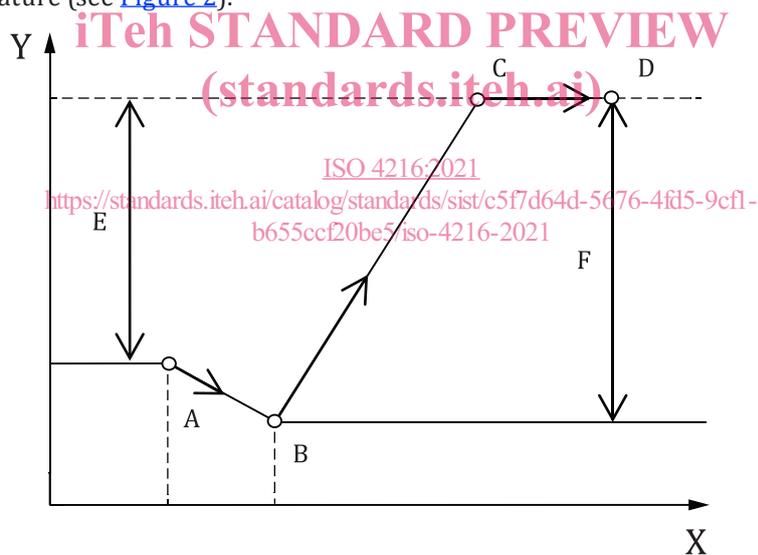
Where

- A is the shrinkage, in %;
- T_1 is the initial sample thickness;
- T_2 is the sample thickness at an arbitrary time.

In addition to the determination of the curing shrinkage, this continuous measurement technique also allows to see the volumetric changing behaviour of a resin at the time it is being irradiated by UV, heated, or cooled during the curing process.

NOTE 1 Typically, upon irradiation by UV rays, UV curable resins expand immediately then contract as curing proceeds (see [Figure 1](#)). However, some types of UV curable resin exhibit fast reactions wherein shrinkage starts immediately right after irradiation (see [Figure B.1](#)).

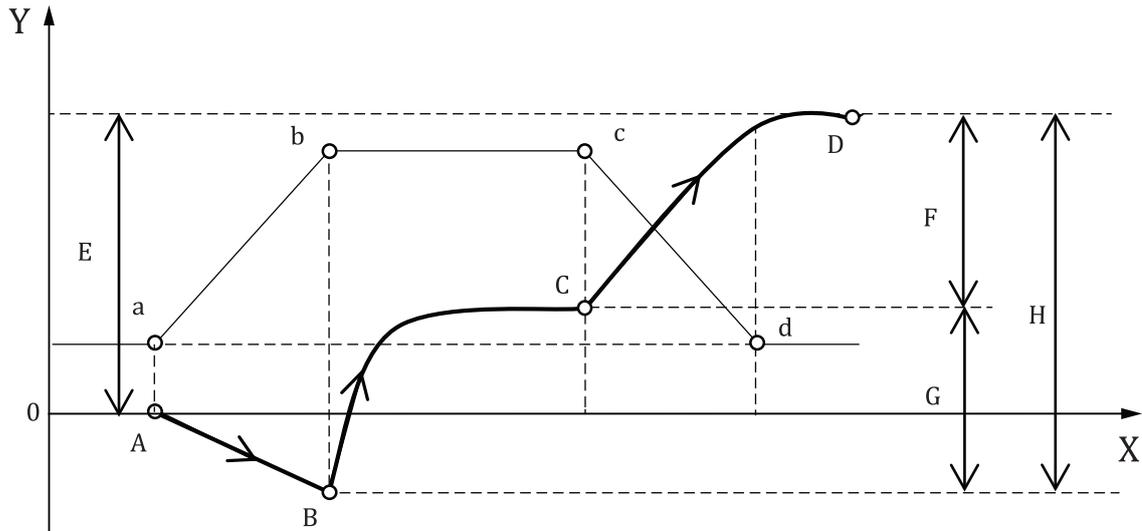
NOTE 2 When a thermosetting resin undergoes heating, thermal expansion occurs due to the increase in the resin temperature. This expansion continues as the temperature rises. When the curing temperature is reached, the curing begins and the resin starts to contract. This shrinkage continues until the resin is fully cured and returned to room temperature (see [Figure 2](#)).



Key

- X time (s)
- Y volumetric shrinkage (%)
- A irradiation start point = curing start point
- B shrinkage start point
- C curing finish point
- D shrinkage finish point
- E curing shrinkage
- F maximum shrinkage

Figure 1 — Curing behaviour of UV curable resin

**Key**

X	time (min)	a, d	room temperature
Y	volumetric shrinkage (%)	b, c	curing temperature
	temperature(°C)		
A	start heating point		
B	start curing point		
C	curing finish point		
D	shrinkage finish point		
E	curing shrinkage		
F	shrinkage by cooling		
G	shrinkage by reaction		
H	maximum shrinkage		

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Figure 2 — Curing behaviour of thermosetting resin

5 Test methods and test conditions

5.1 Test methods

The test methods are classified according to the curing conditions applied to the resin. There are three different types of curing condition: UV curing, thermal curing, and a combination of UV and thermal curing.

5.2 Test conditions

Conduct measurements in the standard laboratory atmosphere of $23\text{ °C} \pm 2\text{ °C}$ ($73,4\text{ °F} \pm 3,6\text{ °F}$) and $50\% \pm 5\%$ relative humidity, unless otherwise specified in the experiment conditions.

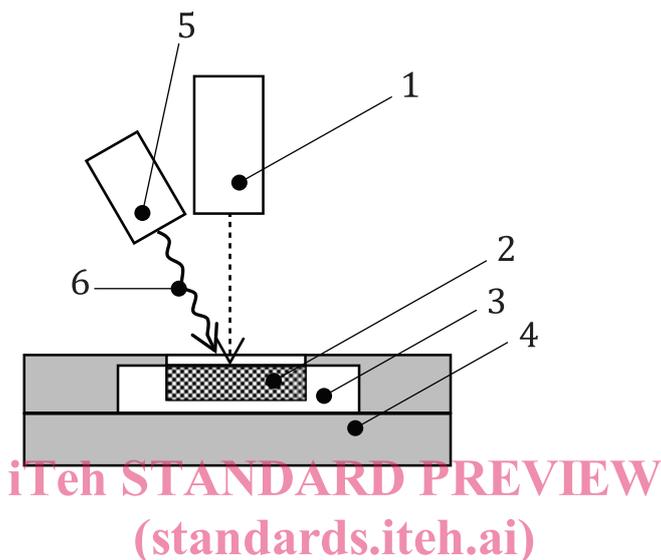
6 Number of measurements

Three or more samples shall be tested for each curing condition.

7 Apparatus

7.1 Apparatus configuration

An apparatus for measuring the curing shrinkage of resin by this method is a system that consists of different units. Primarily, it includes a sample container, displacement gauge, UV irradiation device, heating/cooling device, etc. [Figure 3](#) shows an example of an apparatus configuration compatible with a UV curable resin, and [Figure 4](#) shows an example of an apparatus configuration corresponding to a thermosetting resin.

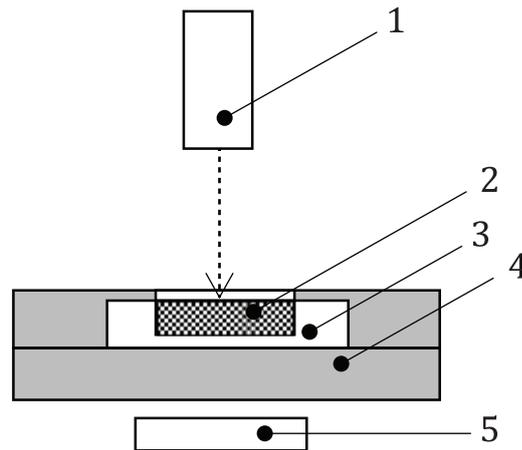


Key

- 1 displacement gauge
- 2 sample
- 3 sample container
- 4 measuring stage
- 5 UV irradiation device
- 6 UV beams

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Figure 3 — Example of an apparatus applied for UV curable resin

**Key**

- 1 displacement gauge
- 2 sample
- 3 sample container
- 4 measuring stage
- 5 heating/cooling device

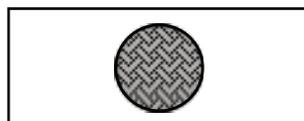
Figure 4 — Example of an apparatus applied for thermosetting resin

7.2 Sample container (standards.iteh.ai)

A sample container of accurately known volume and dimensions shall be used. To ensure an accurate measurement, container material should be used which does not cause either the detachment of resin from the bottom after curing or absorb the sample via the wall surfaces. Moreover, in order to calculate easily the container capacity/volume, the sample container should have a smooth bottom surface and cylindrical shape.



a) Sectional view



b) Top view

Figure 5 — Sample container

(Typical dimensions)

Depth: 0,5 mm to 3 mm

Inner diameter: 10 mm to 15 mm