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Plastics — Epoxy resins — Determination of degree of crosslinking of crosslinked epoxy resins by Fourier Transform Infrared (FTIR) Spectroscopy

Plastiques — Résines époxy — Détermination du degré de réticulation des résines époxy réticulées par spectroscopie infrarouge à transformée de Fourier (FTIR)

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
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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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

Introduction

It is possible to determine the degree of crosslinking of a crosslinked epoxy resin by observing changes in its mechanical, electrical or thermal properties.

However, such approaches are inadequate in cases where the test sample is to be examined under various crosslinking conditions. This document provides a method whereby the degree of crosslinking is determined without the need for complicated procedures for preparing, conditioning or configuring the test sample. The degree of crosslinking is determined by measuring the disappearance of the epoxy group during the resin crosslinking, using Fourier Transform Infrared spectroscopy.

The advantages of this method are that sample preparation is simple, measurements can be made with very small amounts of resin and it is possible to measure the reaction rate of the epoxy group for an epoxy compound containing another active functional group. For these reasons, this document is useful for investigations and for establishing conditions for crosslinking reactions. It can also be used for production and quality control.

Since epoxy resin systems are highly diverse, the applicability of this document to each resin system is intended to be established prior determination. A technique to test the applicability to any epoxy resin system is included in this document.

Plastics — Epoxy resins — Determination of degree of crosslinking of crosslinked epoxy resins by Fourier Transform Infrared (FTIR) Spectroscopy

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 and to determine the applicability of any other restrictions.

1 Scope

This document specifies a method for determining the degree of crosslinking of crosslinked epoxy resins by the disappearance of the epoxy group during epoxy resin crosslinking measured by Fourier Transform Infrared (FTIR) (with a transmittance mode) spectroscopy.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
degree of crosslinking
value which indicates the degree of crosslinking of an epoxy resin system

Note 1 to entry: See Formula (4) in Clause 8.

Note 2 to entry: It is expressed as a percentage.

3.2
height of absorption peak
greatest distance in the ordinate direction between the virtual baseline and the FTIR absorption curve during a peak

Note 1 to entry: The virtual baseline is drawn simply by connecting the peak onset and peak end by a straight line.

Note 2 to entry: When the separation of the specified peak and other peaks is incomplete, the virtual baseline can be drawn by connecting a peak onset and a peak end which are selected optionally in serial peaks including the specified peak by a straight line.

3.3
peak height ratio
value calculated by dividing the peak height of the epoxy group absorption (wave number 900~915 cm^{-1}) by the peak height of the internal standard group absorption which is not changed by a crosslinking reaction

Note 1 to entry: The methylene group absorption band (near 2 930 cm^{-1}) is recommended for use as the internal standard group absorption.

4 Principle

The degree of crosslinking of the epoxy resin is determined by the peak height ratio of the crosslinked state and the uncrosslinked state of the epoxy resin as measured through FTIR (with a transmittance mode).

Firstly, the height of the epoxy group absorption peak (Pa1) and the height of the internal standard group absorption peak (Pa2) are measured by FTIR using a sample taken from the epoxy resin in an uncrosslinked state (see Figure 1).

The peak height ratio of the uncrosslinked state (X) is obtained using Formula (1):

$$X=Pa1/Pa2 \quad (1)$$

Secondly, the height of the epoxy group absorption peak (Pb1) and the height of the internal standard group absorption peak (Pb2) are measured by FTIR using a sample taken from the epoxy compound in a crosslinked state (see Figure 2).

The peak height ratio of the crosslinked state (Y) is obtained using Formula (2):

$$Y=Pb1/Pb2 \quad (2)$$

The degree of crosslinking is determined by Formula (4) in Clause 8.

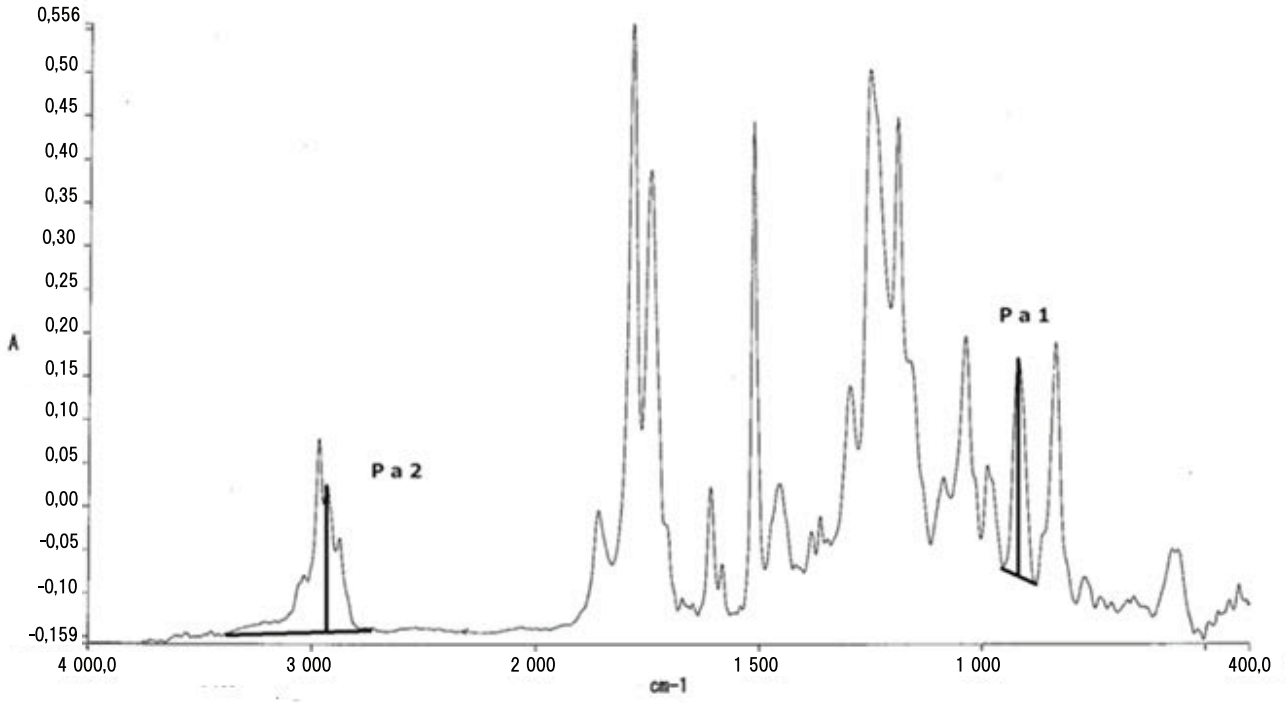

Figure 1 — Spectrum of uncrosslinked epoxy resins

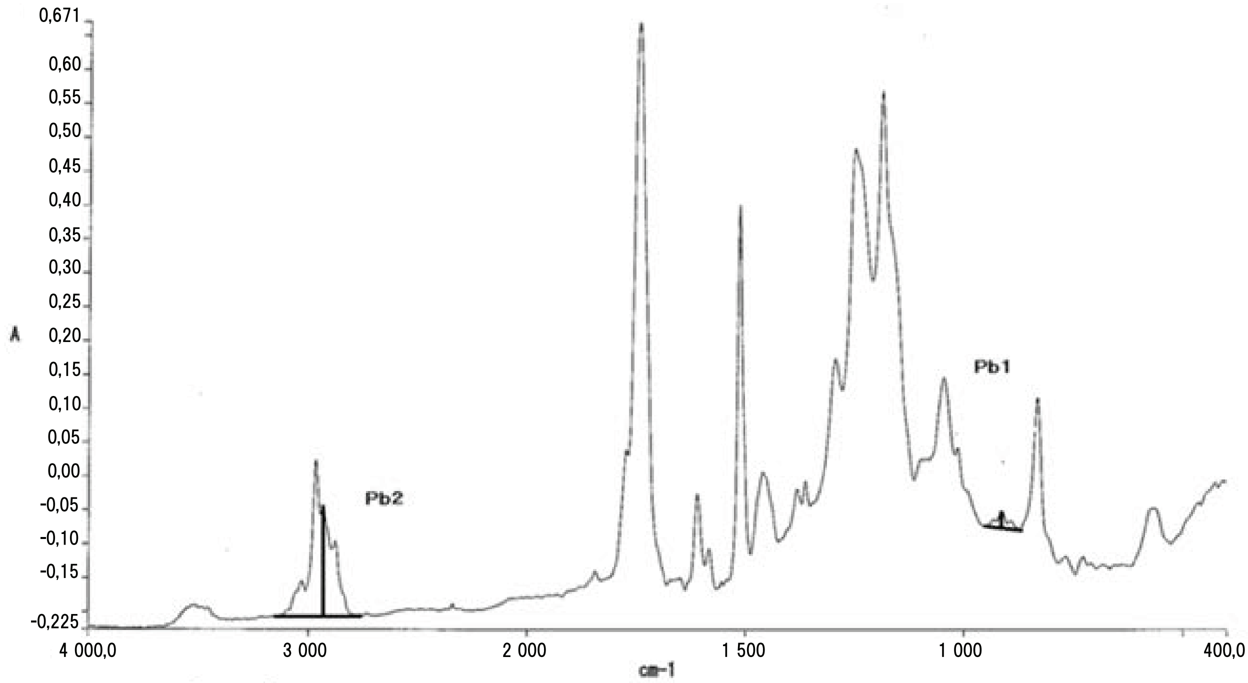

Figure 2 — Spectrum of crosslinked epoxy resins

5 Materials

5.1 Epoxy resin, as specified in the test resin system formulation.

5.2 Hardener, as specified in the test resin system formulation.

5.3 Catalyst, as specified in the test resin system formulation.

6 Apparatus

6.1 FTIR Spectrophotometer, having the following characteristics:

a) an operating range of the wave number from 4 000 cm^{-1} to 650 cm^{-1};

b) the capable cumulative number shall be more than 16.

6.2 Micropipette, with capacity 1 µl to 10 µl.

6.3 Silicon wafer, having the following characteristics:

a) single-side polished monocrystalline silicon wafer;

b) thickness of 625 µm ± 15 µm;

c) total thickness variation (TTV)of 10 µm;

d) total dimensions of the wafer (150 mm; 6 inch wafer).

6.4 Analytical balance, accurate to 0,1 mg.

