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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Test methods for electrical materials, printed boards and other interconnection structures and assemblies –
Part 11: Measurement of melting temperature or melting temperature ranges of solder alloys

Méthodes d'essai pour les matériaux électriques, les cartes imprimées et autres structures d'interconnexion et ensembles –
Partie 11: Mesure de la température de fusion ou des plages de températures de fusion des alliages à braser

IEC 61189-11:2013

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FOREWORD

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International Standard IEC 61189-11 has been prepared by IEC technical committee 91: Electronics assembly technology.

The text of this standard is based on the following documents:

FDIS	Report on voting
91/1086/FDIS	91/1097/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of IEC 61189 under the general title *Test methods for electrical materials, printed boards and other interconnection structures and assemblies* can be found in the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

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- withdrawn,
- replaced by a revised edition, or
- amended.

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TEST METHODS FOR ELECTRICAL MATERIALS, PRINTED BOARDS AND OTHER INTERCONNECTION STRUCTURES AND ASSEMBLIES –

Part 11: Measurement of melting temperature or melting temperature ranges of solder alloys

1 Scope

This part of IEC 61189 describes the measurement method of melting ranges of solder alloys that are mainly used for wiring of electrical equipment, for electrical and communication equipment, and for other apparatus, as well as for connecting components.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60194,— *Printed board design, manufacture and assembly – Terms and definitions*[1]

IEC 61189-3, *Test methods for electrical materials, printed boards and other interconnection structures and assemblies – Part 3: Test methods for interconnection structures (printed boards)*

IEC 61190-1-3, *Attachment materials for electronic assembly – Part 1-3: Requirements for electronic grade solder alloys and fluxed and non-fluxed solid solders for electronic soldering applications*

ISO 9453, *Soft solder alloys – Chemical compositions and forms*

ISO 11357-1, *Plastics – Differential scanning calorimetry (DSC) – Part 1: General principles*

3 Terms and definitions

For the purposes of this document the terms and definitions of IEC 60194, IEC 61189-3, IEC 61190-1-3, ISO 9453 and ISO 11357-1, as well as the following apply.

3.1
melting temperature ranges
total range of solidus and liquidus temperature of solder alloys

3.2
solidus temperature
temperature when solder alloys start to melt measured by DSC (method A)

1 Sixth edition to be published.

3.3
solidus temperature
temperature when solidification of solder alloys ends measured by the cooling curve of molten solder (method B)

3.4
liquidus temperature
temperature when melting ends measured for various heating temperature levels by DSC (method A)

3.5
liquidus temperature
solidification temperature measured by the cooling curve of molten solder (method B)

3.6
DSC curve
curve measured by differential scanning calorimetry (DSC)

4 Summary of measuring methods

The melting temperature range of solder alloys is measured by using the following methods.

Method A: Differential scanning calorimetry (DSC).

Method B: Cooling curve of molten solder.

Test report shall be made according to Annex A.

5 Test equipment

5.1 Method A: DSC

5.1.1 DSC

See ISO 11357-1.

5.1.2 Balance

The balance shall have a resolution of 0,1 mg or better.

5.1.3 Pans

Pans shall be constructed of a material with a high heat transfer rate and which is not corroded by the samples. Usually, aluminium is used.

5.1.4 Inert gas

Inert gas (example N2 or Ar: of a purity higher than 99,9 %) should be used to avoid the sample oxidation.

5.1.5 Alumina powder

Alumina powder should be used as a reference material. It is stable for the temperature range of the measurement. See ISO 11357-1.

5.2 Method B: Cooling curve of molten solder

5.2.1 Electric furnace

It shall be capable of heating its content to a temperature of 400 °C or higher and provide good heat insulation.

5.2.2 Thermocouple

A thermocouple that is suitable for the temperature being used, shall be selected. The compensating lead used shall be suitable for the thermocouple being used.

5.2.3 Measuring instrument

The heat flow measuring instrument shall be capable of measurements of one second intervals or less.

5.2.4 Recorder

The recorder shall be capable of recording a cooling curve and reading in 0,1 °C units.

5.2.5 Container

The graphite or ceramic crucible shall be used.

6 Calibration of the temperature

Temperature calibration shall be conducted using the pure materials listed in Table 1, whose purity shall be 99,99 % or higher. The melting points of two or more pure materials that are close to the temperature to be measured, shall be measured under the same conditions as those applied to the sample, and a compensation formula with a linear function shall be determined for the temperature correction from the obtained measurements and the melting temperatures given in Table 1.

Table 1 – Metal list for calibration

Metal	Melting temperature °C
In (indium)	156,6
Sn (tin)	231,9
Pb (lead)	327,4

7 Procedure for the measuring method

7.1 Method A: DSC

7.1.1 Test condition

7.1.1.1 Sample mass

The sample mass shall be from 5 mg to 50 mg.

7.1.1.2 Inert gas flow

Inert gas shall be used. Gas flow rate shall be from 10 ml/min to 50 ml/min.

7.1.1.3 Heating rate

Heating rate shall be from 0,5 °C/min to 10 °C/min. Recommended heating rate are 0,5, 1, 2, 5 and 10 °C/min.

7.1.2 Procedure for measuring the DSC curve

7.1.2.1 Instructions

Carry out the measuring DSC curve as follows.

a) The sample is placed in the centre part of pans, and the cap of the pans is put and clamped.

b) The pan containing the sample is placed on to the pan holder, and the pan with alumina powder is placed on the other pan holder.

c) Flow the inert gas (example N2 or Ar) until the measurement ends.

d) Carry out the measuring DSC curve with a heating rate of 0,5 °C/min up to a temperature about 30 °C higher than the heat flow peak.

Repeat procedures a) through d) using a new sample, except heating rate of 1, 2, 5 and 10 °C/min.

7.1.2.2 Solidus temperature

The data of a heating rate of 2 °C/min are used. A typical DSC curve is shown in Figure 1. T1 or T2 represent the solidus temperature.

a) If melting occurs abruptly, then the temperature when melting starts shall be the temperature T1 at the intersection of the extrapolation of the low-temperature side baseline towards the high-temperature side and the tangent drawn from the low-temperature side endothermic peak at the point with the steepest slope, as shown in Figure 1a. In this case, correct the temperature, using T1 of the pure materials.

b) If melting occurs gradually, then determine temperature T2 at the point at which the curve starts to leave the baseline, as shown in Figure 1b. In this case, correct the temperature, using T2 of the pure materials. Repeat the measurement several times and then determine the average.

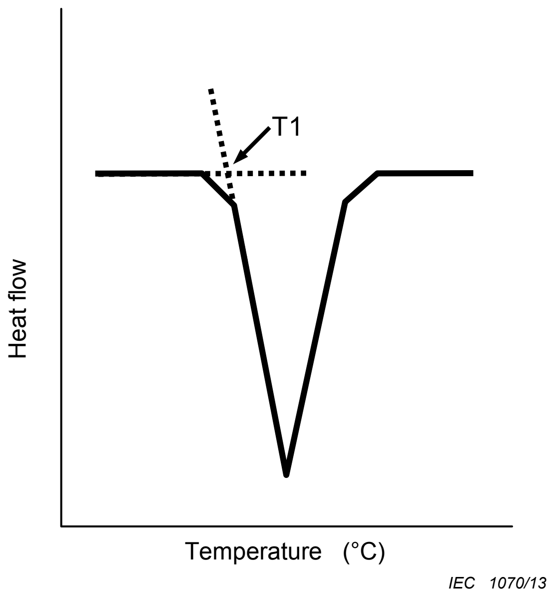

Figure 1a – Abruptly melting alloy

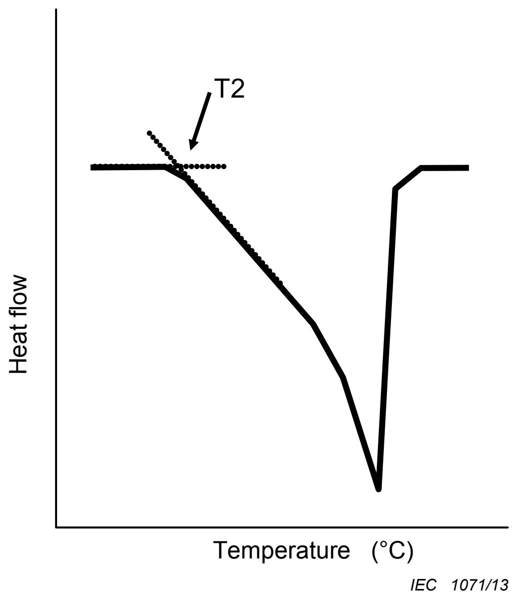

Figure 1b – Gradually melting alloy

Figure 1 – Determination of solidus temperature

7.1.2.3 Liquidus temperature

The data of a heating rate of 0,2, 0,5, 1, 2, 5 and 10 °C/min are used. A typical DSC curve is shown in Figure 2. T3 or T4 indicate the temperature where melting ends.

a) If melting occurs with a single peak, the temperature of melting ends shall be the temperature T3 at the intersection of the extrapolation of the high-temperature side baseline towards the low-temperature side and the tangent drawn from the high-temperature side endothermic peak at the point with the steepest slope, as shown in Figure 2a.

b) If melting occurs with double or more peaks, the temperature where the melting ends shall be the temperature T4 at the intersection of the extrapolation of the high-temperature side baseline towards the low-temperature side and the tangent drawn from the most highest-temperature side endothermic peak at the point with the steepest slope, as shown in Figure 2b.

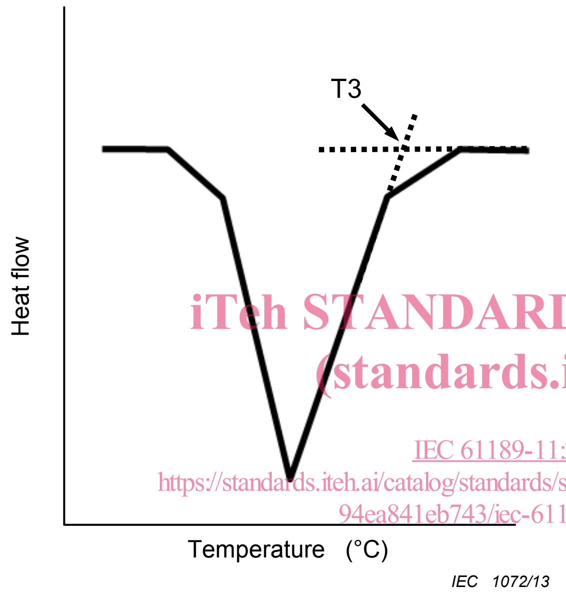

Figure 2a – Single melting peak

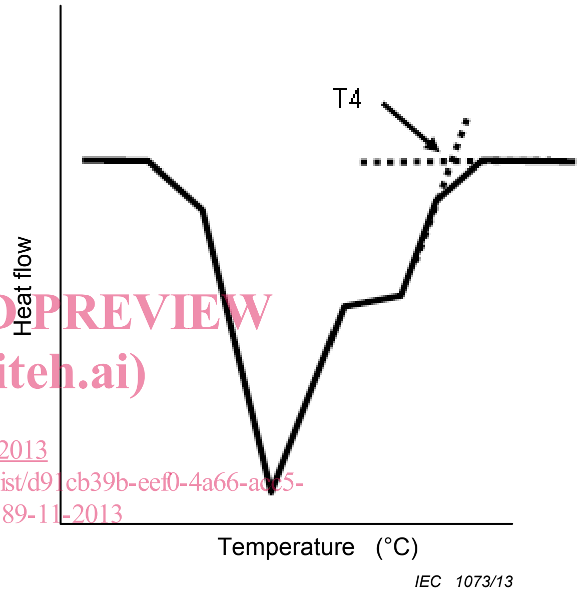

Figure 2b – Double or more melting peaks

Figure 2 – Determination of temperature of melting ends

c) The extrapolated end temperature of endothermic peak is a linear function of the square root of the heating rate. Therefore, the point of interception on a temperature axis of the linear function is assumed to be the liquidus temperature, as shown in Figure 3.

NOTE Examples of the test results are shown in Annex B.

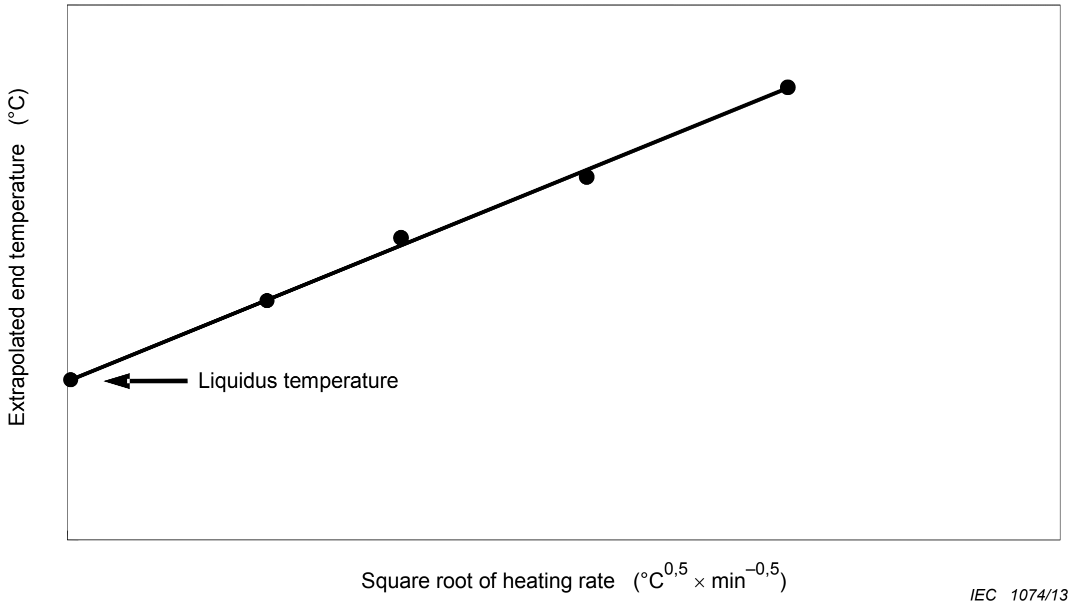

Figure 3 – Determination of liquidus temperature

7.2 Method B: Cooling curve of molten solder

7.2.1 Test condition

7.2.1.1 Sample mass

The sample mass shall be 500 g or more.

7.2.1.2 Sample melting

Place the sample in the container and then heat it in the electric furnace until it melts.

7.2.1.3 Thermocouple installation

Position the temperature-measuring junction of the thermocouple in the centre of the molten solder.

7.2.1.4 Reference junction

The reference junction shall be of the cryoscopic, thermoelectric cooling, or compensating type.

7.2.2 Procedure for measuring the cooling curve of molten solder

7.2.2.1 Instructions

Melt the entire sample in the crucible, then turn off the power to the electric furnace and measure the temperature as the sample cools down.

Typical cooling curves of molten solder are shown in Figure 4.

7.2.2.2 Solidus temperature

The solidus temperature shall be determined from the parallel portion, (T6, as shown in Figure 4b). If undercooling occurs, as shown in Figure 4c, then the temperature T7 at the intersection of the extrapolation of the parallel portion toward the short-time side and the cooling curve shall be assumed to be the solidus temperature.