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Measurement procedures for materials used in photovoltaic modules – Part 1-6: Encapsulants – Test methods for determining the degree of cure in Ethylene-Vinyl Acetate

Procédures de mesure des matériaux utilisés dans les modules photovoltaïques – Partie 1-6: Encapsulants – Méthodes d'essai pour déterminer le degré de durcissement dans l'éthylène-acétate de vinyle

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MEASUREMENT PROCEDURES FOR MATERIALS USED IN PHOTOVOLTAIC MODULES –

Part 1-6: Encapsulants – Test methods for determining the degree of cure in Ethylene-Vinyl Acetate

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

FDIS	Report on voting
82/1197/FDIS	82/1231/RVD

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

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

A list of all parts in the IEC 62788 series, published under the general title *Measurement procedures for materials used in photovoltaic modules*, can be found on the IEC website.

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MEASUREMENT PROCEDURES FOR MATERIALS USED IN PHOTOVOLTAIC MODULES –

Part 1-6: Encapsulants – Test methods for determining the degree of cure in Ethylene-Vinyl Acetate

1 Scope

This part of IEC 62788 defines the terminology, test equipment, test environment, specimen preparation, test procedures, and test report for measuring the degree of cure of Ethylene-Vinyl Acetate (EVA) encapsulation sheet used in photovoltaic (PV) modules. The differential scanning calorimetry (both residual enthalpy and melt/freeze protocols) and gel content methods are included herein. This procedure can be used by material- or module-manufacturers to verify that the cross-linking additive is present and is active. The procedure can also be used to verify the module manufacturing (lamination) process for the purposes of quality- and process-control. The procedure can also be used to assess the uniformity of the EVA formulation within a roll as well as to compare variation of the EVA formulation from roll to roll. This procedure can be applied to uncured or recently cured EVA sheet as well as uncured or recently cured EVA from PV modules.

This test procedure can also be applied to cross-linking ethylenic co-polymers other than EVA. The temperatures identified for the calorimetry measurements in this procedure have been optimized for EVA. Therefore, if the test procedure is applied to other encapsulation materials, the range of the test temperatures can have to be adjusted based on the active temperature of the curing agent and/or the melt/freeze temperature of the base material.

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.

IEC 61215-1, *Terrestrial photovoltaic (PV) modules – Design qualification and type approval – Part 1: Test requirements*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

ISO 291:2008, *Plastics – Standard atmospheres for conditioning and testing*

ISO 6427:2013, *Plastics – Determination of matter extractable by organic solvents (conventional methods)*

ISO 11357-1:2009, *Plastics – Differential scanning calorimetry (DSC) – Part 1: General principles*

ISO 10147:2011, *Pipes and fittings made of crosslinked polyethylene (PE-X) – Estimation of the degree of cross-linking by determination of the gel content*

ASTM D2765-11, *Standard test methods for determination of gel content and swell ratio of crosslinked ethylene plastics*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC TS 61836 and the following 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

NOTE Calculations related to these definitions are given in 5.5.

3.1
degree of cure
G
unitless parameter that characterizes the extent of cross-linking within EVA

Note 1 to entry: Unlike the cross-link density, which is a physical quantity defined by the theory of rubber elasticity, the degree of cure in a polymer may be assessed by any experimental method that distinguishes partially cured specimens with respect to one another or with respect to a reference material. The degree of cure can be correlated to the gel content, which is the mass percentage of insoluble material (assumed to be cross-linked) within the specimen.

3.2
heat flow
Q
thermal flux across a specified area in the direction of a temperature gradient, W

Note 1 to entry: The specific heat flow, q, is defined as the thermal flux per unit mass of the specimen, W g^{-1}.

3.3
differential scanning calorimetry
DSC
thermoanalytical technique described in ISO 11357-1, in which the difference in the amount of heat flow required to change the temperature of a material specimen and a reference is measured as a function of temperature or time

Note 1 to entry: Both the specimen and reference are maintained at nearly the same temperature during DSC characterization. DSC may be applied to quantify the amount of heat generated or absorbed during the processing (curing) of EVA. The effects of cross-linking, which occur from changes in the molecular structure of the EVA, may also be examined using DSC at the phase transitions (glass transition, melting point, and crystallization temperature). The determination of the phase transition temperatures is described in ISO 11357-2 and ISO 11357-3.

3.4
differential scanning calorimeter
instrument used to measure the heat flow difference between the test crucible (containing the specimen) and reference (typically empty) crucible

3.5
gel content
percentage of mass content of polymer insoluble in a specified solvent after extraction according to the specified test conditions

Note 1 to entry: The gel is typically composed of insoluble cross-linked material.

4 Principle

The degree of cure of EVA may be quickly inferred using a "secondary method", such as differential scanning calorimetry (DSC, ISO 11357-1), described in Clause 5. Established alternative secondary methods, as identified in Annex A, or specialized equipment may also be used directly for the purpose of manufacturing and quality control. When the results of the

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secondary method are to be compared between different institutions, they shall be calibrated using a slower, more universal "primary" method, the gel content test as described in Clause 6, similar to the procedures described in ISO 6427, ISO 10147 and ASTM D2765. The primary method may also be applied for research and development when the results of the secondary method are to be compared between formulations of EVA. A test procedure for the primary method is described in Clause 6. The results of the primary or secondary methods may be correlated to the module qualification tests (IEC 61215 series) or additional field durability data to identify the minimum degree of cure necessary. Examples correlating between the secondary and primary methods may be found in the bibliography of Annex A.

The DSC measurements for EVA may be interpreted based on the enthalpy of the cross-linking reaction or the characteristics of the melt/freeze transition as described in 5.5. Because the melt/freeze transition does not depend on the concentration of residual peroxide, the DSC melt/freeze method may be applied to specimens obtained from fielded modules. Limitations of the primary and secondary methods are discussed in Annex A.

5 DSC secondary method

5.1 Instrument and equipment for the secondary method

5.1.1 General

References for the application of the DSC method are provided in Annex A.

5.1.2 Electronic balance

The micro balance should have a measurement resolution of at least 0,01 mg, and a maximum range of at least 20 mg.

5.1.3 Differential scanning calorimeter

5.1.3.1 The calorimeter should have a temperature accuracy of at least ±0,1 °C, temperature precision of at least 0,01 °C, calorimetric accuracy of at least ±0,5 % (or 0,2 mW), calorimetric sensitivity of at least 2 μW, and calorimetric repeatability of at least ±0,5 %.

5.1.3.2 The oven heating/cooling rate should be adjustable between 5 °C $min^{-1}$and 30 °C min^{-1} measured with a thermometric accuracy of at least ±0,1 °C min^{-1}.

5.1.3.3 The baseline drift (absolute value of signal change between the two integration limit, for an empty cell) should be less than 50 μW, for the temperature range from –50 °C to 250 °C.

5.1.3.4 The baseline curvature (the biggest deviation from the integration baseline) should be less than 50 μW, for the temperature range from –50 °C to 250 °C.

5.1.4 Instrument calibration

The instrument should be calibrated routinely according to the instrument manufacturer's specification, using the instrument supplier's recommended calibration methods. Accuracy calibration should be performed using standard substances, for example, indium or tin, as the temperature and heat-flow verification material. Sapphire may be used to quantify the baseline (curvature) of the instrument drift. The instrument should specifically be recalibrated if the test rate, type of pan, or test atmosphere has been changed before DSC measurements.

NOTE The importance, performance, and considerations related to DSC instrument calibration are described further in D. Chen, A. Green, D. Dollimore, "DSC: the Importance of Baseline Calibration", Thermochimica Acta, 284 (2), 1996, 429–433.

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5.2 Specimen preparation for the secondary method

5.2.1 Sampling and storage

5.2.1.1 Because the results for the secondary method may depend on the make of EVA, test results may only be directly compared for the same formulation of EVA. Therefore, test specimens should come from the same manufacturer and fabrication batch.

5.2.1.2 Additional experimentation shall be performed using uncured EVA to establish a baseline for the uncured state (for both the DSC residual enthalpy and melt/freeze methods) and a previously cured ("maximum cured") EVA used to establish a baseline for the final cured state (for the melt/freeze method).

If the experiment is intended to monitor a production process, the "maximum cured" samples should be taken from a laminated module or test sample subjected to the thermal history used in lamination. If the experiment is intended to monitor the complete consumption of peroxide (which often does not occur during the lamination of a PV module), additional processing (time or temperature) may be required.

5.2.1.3 Operators should wear clean gloves when preparing and handling samples.

5.2.1.4 When storage is required, the EVA should be packaged in a marked, sealed bag for later use.

5.2.1.5 Specimens should be kept dry (stored at below 50 % relative humidity), maintained at ambient temperature, and not exposed to light.

It is recommended to verify that the results of the secondary method do not change with storage time.

5.2.2 Preparation procedures

5.2.2.1 Weigh the empty specimen crucible and empty reference crucibles.

The use of aluminium crucibles is recommended for use with EVA.

5.2.2.2 Prepare EVA specimens 5 mg to 9 mg in size (or of a size recommended by the DSC instrument manufacturer), obtained from a single sheet of material. The accuracy of the measurement of the specimen mass should be at least 1 %.

5.2.2.3 A minimum of 2 samples shall be used from each roll of EVA for process control.

5.2.2.4 Place each specimen in a separate crucible, and seal the crucible with a lid.

A non-hermetic aluminium crucible meets the requirement for this test.

The final geometry, specifically the flatness of bottom surface of the crucible, can affect its thermal contact to the instrument, which is critical to the measurement. If the crucible geometry is compromised during preparation, the specimen should be discarded.

If the specimen or lid is not well seated relative to the crucible, it will affect the measurement, and the specimen should be discarded.

5.2.2.5 Record the measured mass of each specimen and its crucible.

5.3 Test requirements for the secondary method

5.3.1 Environment requirements

The recommended laboratory environment of (25 ± 2) °C and the relative humidity of (50 ± 5) % shall be used, as in ISO 291.

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DSC tests should be performed using a dry inert carrier gas, such as nitrogen, in the DSC instrument. The gas flow rate shall be specified by the user, for example, (50 ± 5) ml·min^{-1}. The purity of gas should be at least 99,99 %.

5.3.2 Parameter settings (residual enthalpy method)

The following test parameters are recommended for use during the DSC residual enthalpy method:

Data acquisition rate: 5 Hz (0,2 s·point^{-1});

Initial temperature: 25 °C;

End temperature: 225 °C;

Heating rate: 10 °C·min^{-1}.

The completion of the peroxide reaction may be verified using a second thermal cycle (cool to 25 °C and reheat to 225 °C). The residual enthalpy for maximum cured EVA should be $< 0{,}1$ J·g^{-1} during this second thermal cycle.

5.3.3 Parameter settings (melt/freeze method)

The following test procedure is recommended for use during the DSC melt/freeze method:

Data acquisition rate: 5 Hz (0,2 s·point^{-1});

Initial temperature: 25 °C;

Heat to 100 °C at the rate of 10 °C·min^{-1};

Cool to –20 °C at the rate of 10 °C·min^{-1}.

Care should be taken to ensure that the heating used to melt the specimen and erase structure-related effects is limited to temperatures less than that capable of activating the peroxide. If a reaction is evident in the data profile for heating, a temperature less than 100 °C should be used.

5.3.4 Parameter settings (combined enthalpy and melt/freeze method)

The following test procedure (with no dwell time occurring between the separate steps) is recommended for performing the DSC enthalpy and melt/freeze characterization on the same specimen, in a single test:

Data acquisition rate: 5 Hz (0,2 s·point^{-1});

Initial temperature: 25 °C;

Heat to 100 °C at the rate of 10 °C·min^{-1};

Cool to –20 °C at the rate of 10 °C·min^{-1};

Heat to 225 °C at the rate of 10 °C·min^{-1}.

Additional data obtained after cooling from 225 °C, can be used for the "maximum cured" reference specimen required for the DSC melt/freeze method. To make use of the combined DSC method to also obtain data for the maximum cured reference specimen, cooling should be carried out to –20 °C at the rate of 10 °C·min^{-1}, so that the freeze transition is accurately characterized after thoroughly curing the test specimen in the calorimeter. If the maximum cured reference specimen is measured after the combined DSC characterization of a set of EVA specimens, the specimens with the greatest previous thermal history (temperature and time) should be used, to ensure that the EVA is thoroughly cured.

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5.4 Test procedure for the secondary method

5.4.1 The DSC tests shall be carried out as follows.

5.4.2 The test parameters in 5.3.2 shall be used for the DSC residual enthalpy method; the test parameters in 5.3.3 shall be used for the DSC melt/freeze method; or the test parameters in 5.3.4 shall be used for the combined DSC residual enthalpy and melt/freeze methods.

5.4.3 Confirm the furnace (flange) temperature for the calorimeter is in a safe temperature range, and open the lid.

5.4.4 Place the specimen crucible and the empty reference crucible in the oven, and close the lid.

5.4.5 Specify the mass of the specimen and reference crucible to the calorimeter and initiate the test. An isothermal hold at the endpoints of the test segments within the method (i.e., initial, hot, or cold temperatures) shall not be used for the residual enthalpy, melt/freeze, or combined methods. In order to obtain the most consistent results, it is recommended to use the same test method (residual enthalpy, melt/freeze, or combined) for the purpose of process control or other sample comparison.

5.4.6 Remove the crucibles from the calorimeter at the end of test.

It is suggested that the specimen(s) be weighed after the test, which may be compared to the initial mass to verify the integrity of the crucible. The final weights of the specimen(s) may also be used to confirm the specimen identity.

5.4.7 Record test data, and calculate the degree of cure according to the method in 5.5.

5.5 Calculation and expression of the results for the secondary method

5.5.1 Enthalpy method

The degree of cure for the DSC residual enthalpy method shall be calculated using Formula (1):

$$G_{\mathrm{e}} = \frac{h_{\mathrm{u}} - h_{\mathrm{t}}}{h_{\mathrm{u}}} 100 \qquad (1)$$

In the formula, G_{e} represents the degree of cure for the enthalpy method, %; h_{u}, the measured specific enthalpy of EVA of an uncured reference specimen, $\mathrm{J \cdot kg^{-1}}$; and h_{t}, the measured specific enthalpy of EVA of the test specimen, $\mathrm{J \cdot kg^{-1}}$.

The specific enthalpy shall be determined using the instrument software from the integral of the measured heat flow, using the limits of integration from 100 °C to 200 °C for the specified heating rate. The specific enthalpy for the test specimen may include multiple peaks within the bounds of integration.

An example result is shown in Figure 1, where an offset has been added to q to distinguish the test and reference data profiles. As in Figure 1, the degree of cure, G_{e}, for the test specimen in the figure is 87,6 %.

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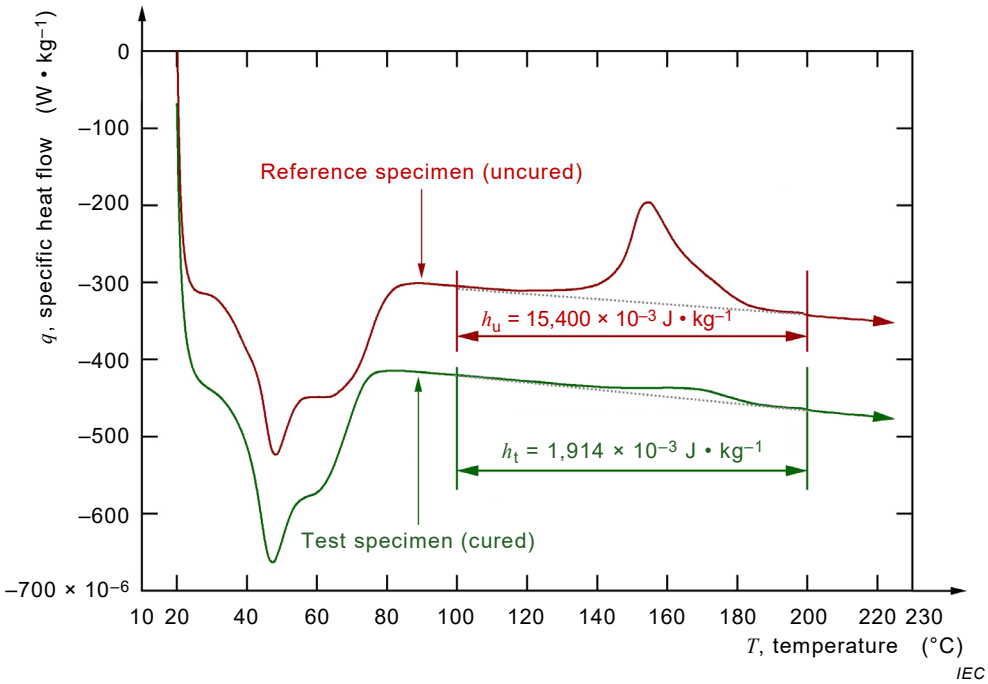

Figure 1 – Example result for the DSC residual enthalpy method

The data profiles (with an offset added to the specific heat flow to distinguish the data) are shown for a cured test specimen and an uncured reference specimen.

The presence of contamination (from backsheet or other materials) may be verified from the data profile. Contamination will result in unexpected peaks within the data profile, which should be noted in the test report.

5.5.2 Melt/freeze method

5.5.2.1 Determination of the degree of cure

The analysis for the DSC melt/freeze method considers three parameters within the measured data: the maximum of the crystallization (freeze) temperature, the extrapolated onset of the crystallization temperature, and the concavity of the data profile below the crystallization temperature (assessed using a quantitative shape factor). The DSC melt/freeze method may be applied to any EVA specimen, regardless of its thermal history (including EVA from fielded modules), to quantify its degree of cure. The degree of cure for the DSC melt/freeze method shall be calculated from Formulas (2) to (5):

$$G_{\mathrm{a}} = \left(\frac{G_{\mathrm{c}} + G_{\mathrm{o}} + G_{\mathrm{SF}}}{3} \right) \tag{2}$$

$$G_{\mathrm{c}} = \frac{T_{\mathrm{c,u}} - T_{\mathrm{c,t}}}{T_{\mathrm{c,u}} - T_{\mathrm{c,m}}} 100 \tag{3}$$

$$G_{\mathrm{o}} = \frac{T_{\mathrm{o,u}} - T_{\mathrm{o,t}}}{T_{\mathrm{o,u}} - T_{\mathrm{o,m}}} 100 \tag{4}$$

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$$G_{S,F} = \frac{SF_u - SF_t}{SF_u - SF_m} 100 \quad (5)$$

In the formulas, G_a represents the average value for the degree of cure from the DSC melt/freeze method, %; G_c represents the degree of cure determined for the change in the maximum of the crystallization temperature, %; G_o represents the degree of cure determined for the change in the temperature extrapolated at the onset of the crystallization, %; and G_{SF} represents the degree of cure determined for the change in the concavity of the data profile below the crystallization temperature, %. In the formulas, the subscript –a refers to the average (numerical mean); –c, the maximum crystallization temperature; –o, the extrapolated temperature at the onset of the crystallization; –SF, the concavity of the data profile below the crystallization temperature (evaluated using a shape factor – See 5.5.2.2); –t, the cured test specimen; –m, the previously laminated ("maximum cured") reference EVA specimen; and –u, a reference EVA specimen with no prior thermal history ("uncured").

Figure 2 shows an example, where the applicable temperatures (T_c and T_o) and temperature range for the shape factor are identified in the figure for the test specimen. The data is shown for specimens of the same EVA formulation, so that the effects of the curing process are evident. An offset has been added to q to distinguish the test and reference (uncured and maximum cured) data profiles.

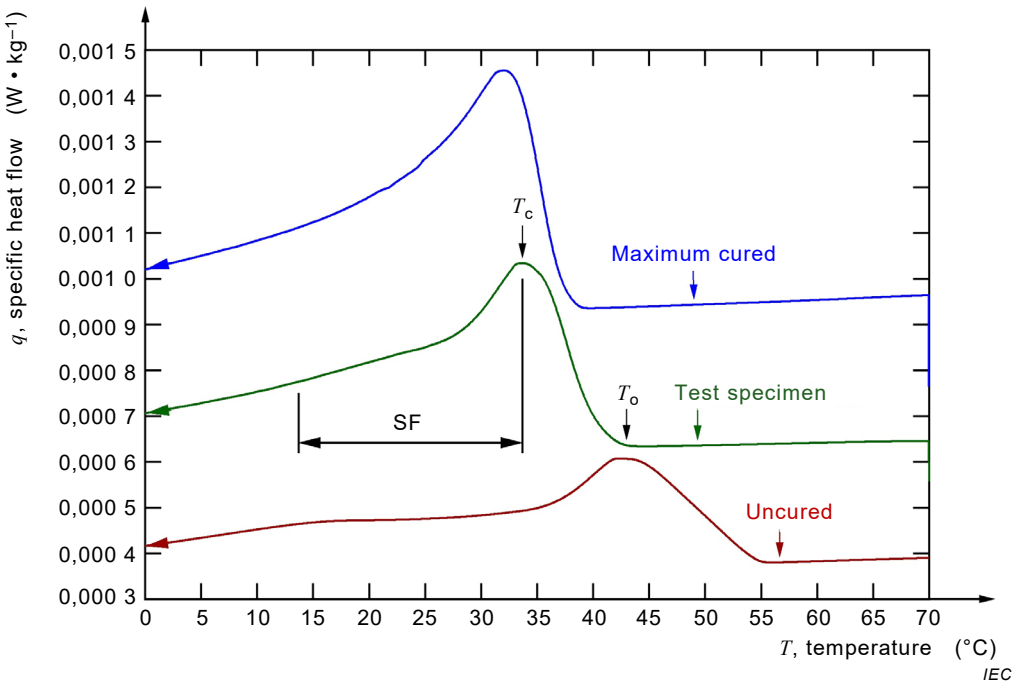

Figure 2 – Location of temperatures and temperature ranges used in the melt/freeze DSC method

The data profiles (with an offset added to the specific heat flow to distinguish the data) are shown for test and reference specimens.

The results shown in Figure 2 are summarized in Table 1. The values, determined from Formulas (2) to (5), are provided as an example. Table 1 formally demonstrates the required data (temperature and shape factor values) and corresponding results for the DSC melt/freeze method.

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Table 1 – Summary of the results for the example measurements shown in Figure 2

	SPECIMEN	T_c °C	T_o °C	SF %
MEASUREMENTS	n, uncured reference	42,3	54,4	79,9
	f, maximum cured reference	32,0	37,2	63,7
	t, test	33,7	40,3	66,4
RESULTS	G_a %	G_c %	G_o %	G_{SF} %
	83,0	83,4	82,4	83,1

For software equipped DSC instruments, an integration algorithm may be used to automatically determine T_c and T_o. The choice of the upper-limit and lower-limit bounds for the integration operation can, however, affect the T_c and T_o values. To achieve repeatable results, it is recommended that the user check for repeatability of the T_c and T_o values given by the software, which can be done by manually varying the limits for the integration. The following steps, which should apply for a broad variety of EVA specimens, are recommended for the automated determination of T_c and T_o.

i) The upper-limit temperature (T_{ul}, °C) shall be fixed by taking the approximate T_c value (based on visual inspection of the measured thermogram), and adding 15 °C, as shown in Figure 3. T_{ul} should fall within the flat region in the thermogram that precedes the crystallization peak during cooling. After the integration analysis, T_{ul}, should be greater than, but located near T_o.

ii) Once T_{ul}, has been assigned, the lower-limit temperature bound (T_{ll}, °C) may be determined by using the software to draw a horizontal line from T_{ul} down to the temperature intersecting the data profile in the thermogram, Figure 3. For EVA, T_{ll} will typically fall in the range from –20 °C to –10 °C.

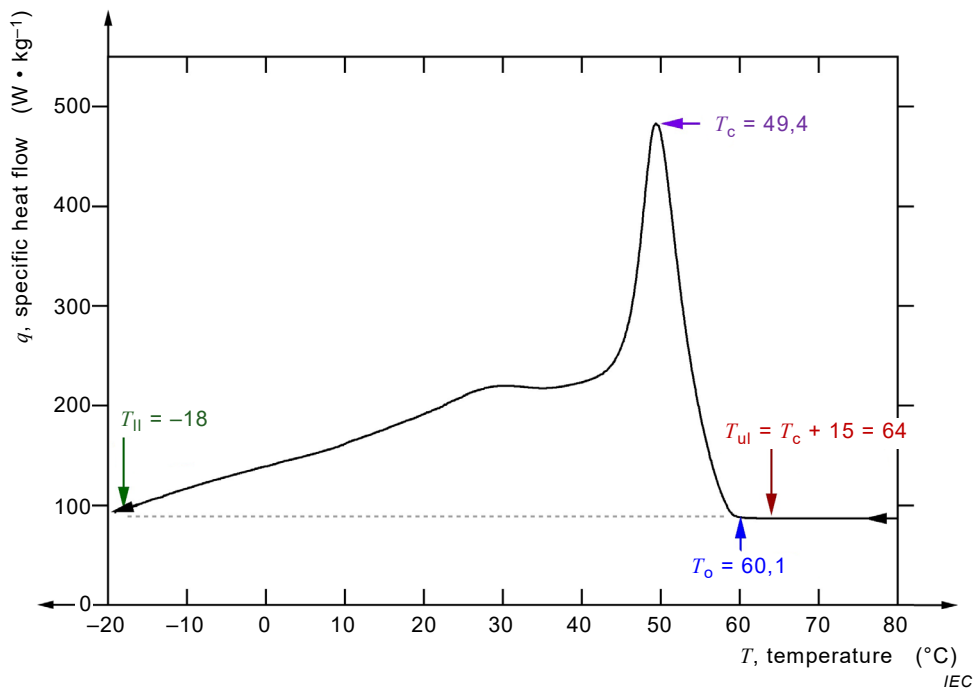

Figure 3 – Example of the temperature bounds applied for an automated software integration algorithm

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5.5.2.2 Determination of the concavity

5.5.2.2.1 The empirical shape factor, characterizing the concavity of the crystallization peak shall be calculated using the following algorithm.

Select all (T, Q) data points between T_c and (T_c – 20 °C).

5.5.2.2.2 Calculate the inverse heat flow, Q^{-1}, for all data points.

5.5.2.2.3 Calculate the product, $T \cdot Q^{-1}$, for all data points.

5.5.2.2.4 Identify the coordinates, [T, $T \cdot Q^{-1}$]max, for which the product reaches a maximum.

5.5.2.2.5 Calculate the shape factor from Formula (6):

$$SF = \frac{\left[T \cdot Q^{-1}\right]_{\max}}{T_c \cdot \left[Q^{-1}\right]_{Tc-20}} 100 \qquad (6)$$

Either the heat flow, Q, or specific heat flow, q, may be used in the concavity analysis.

Figure 4 shows an example of the determination of the shape factor. The measurement profile from the DSC instrument for an EVA test specimen is shown at the top of the figure. The determination of the shape factor is performed from the analysis in 5.5.2.2.1. The region of examination of the shape factor (SF) is identified in both parts of the figure. The maximum of the product of T and Q^{-1}, $[T \cdot Q^{-1}]_{max}$, is labelled in the bottom of the figure, along with: the temperature at $[T \cdot Q^{-1}]_{max}$, $T_{[T \cdot Q]max}$; the inverse heat flow at $T_{[T \cdot Q]max}$, $Q^{-1}_{[T \cdot Q]max}$; the maximum crystallization temperature, T_c; and the inverse heat flow at (T_c – 20 °C), $Q^{-1}_{T_c - 20}$. The shape factor of 66,9 % is determined for $T_{[T \cdot Q]max}$ = 27,4 °C, $Q^{-1}_{[T \cdot Q]max}$ = 0,336 mW^{-1}, T_c = 32,1 °C, and Q^{-1}_{Tc-20} = 0,429 mW^{-1}.

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