भारतीय मानक

Indian Standard

IS 16792 (Part 1/Sec 6) : 2023
IEC 62788-1-6 : 2017+AMD 1 : 2020

फोटोवोल्टिक मॉड्यूल में प्रयुक्त सामग्री के लिए मापन प्रक्रिया

भाग 1 एन्कैप्स्युलेंटस

अनुभाग 6 एथिलीन-विनाइल एसीटेट में इलाज की डिग्री निर्धारित करने के लिए परीक्षण विधियाँ

(पहला पुनरीक्षण)

Measurement Procedures for Materials Used in Photovoltaic Modules

Part 1 Encapsulants

Section 6 Test Methods for Determining the Degree of Cure in Ethylene-Vinyl Acetate

(First Revision)

ICS 27.160

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

May 2023 **Price Group 11**

Solar Photovoltaic Energy Systems Sectional Committee, ETD 28

NATIONAL FOREWORD

This Indian Standard (First Revision) which is identical with IEC 62788-1-6:2017 + Amd 1 : 2020 'Measurement procedures for materials used in photovoltaic modules – Part 1-6: Encapsulants – Test methods for determining the degree of cure in ethylene-vinyl acetate' issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Solar Photovoltaic Energy Systems Sectional Committee and approval of the Electrotechnical Division Council.

This standard was first published in 2019. This first revision has been undertaken to take into consideration the developments that have taken place subsequently and also to align with the latest version of IEC 62788-1-6:2017 + Amd 1 : 2020 .

The text of IEC Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain terminologies and conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.
b) Comma (,) has been used as a decimal marker, while in Indian Standards the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 6427 : 2013 Plastics – determination of matter extractable by organic solvents (conventional methods)	IS 13360 (Part 11/Sec 7) : 2018 Plastics — Methods of Testing: Part 11 Special properties, Section 7 Setermination of matter extractable by organic solvents (Conventional method) (*first revision*)	Identical
ISO 14577-1 Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method	IS 17144 (Part 1) : 2019 Metallic materials — Instrumented indentation test for hardness and materials parameters: Part 1 Test method	Identical
ISO/IEC 17025 : 2005 General requirements for the competence of testing and calibration laboratories	IS/ISO/IEC 17025 : 2017 General requirements for the competence of testing and calibration laboratories (*second revision*)	Identical
IEC 61215-1 Terrestrial photovoltaic (PV) modules — Design qualification and type approval — Part 1: Test requirements	IS 14286 (Part 1) : 2019 Terrestrial photovoltaic (PV) modules — Design qualification and type approval: Part 1 Test requirements (*second revision*)	Identical

The technical committee has reviewed the provisions of the following international standards referred in this adopted standard and decided that they are acceptable for use in conjunction with this standard:

(*Continued on third cover*)

CONTENTS

Indian Standard

MEASUREMENT PROCEDURES FOR MATERIALS USED IN PHOTOVOLTAIC MODULES

PART 1 ENCAPSULANTS

Section 6 Test Methods for Determining the Degree of Cure in Ethylene-Vinyl Acetate

(First Revision)

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 6721-1, *Plastics – Determination of dynamic mechanical properties – Part 1: General principles*

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*

ISO 14577-1, *Metallic materials – Instrumented indentation test for hardness and materials parameters – Part 1: Test method*

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

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.

3.6
degree of cure

G_i

<indentation method> parameter that correlates with the extent of cross-linking within the EVA using the indentation method

Note 1 to entry: Unit: dimensionless.

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 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 $\text{min}^{-1}$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.

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(s) for the same fabrication lay-up configuration (backsheet/EVA combination). Changes in the encapsulant that affect the curing process, including but not limited to a change of the material supplier, would require validating the correlation between $G_{\%}$ and the degree of cure. For example, if the percentage vinyl acetate content in the EVA resin changes, a new correlation between $G_{\%}$ and the degree of cure (from a secondary method) should be obtained because the percentage vinyl acetate content is known to significantly affect the viscoelastic-dependent cure characteristics of the encapsulant.

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.

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 $°C \cdot min^{-1}$;

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

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

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 $°C \cdot min^{-1}$, 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.

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):

$$G_e = \frac{h_u - h_t}{h_u} 100 \tag{1}$$

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, $J \cdot kg^{-1}$; and h_t, the measured specific enthalpy of EVA of the test specimen, $J \cdot kg^{-1}$.

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 %.

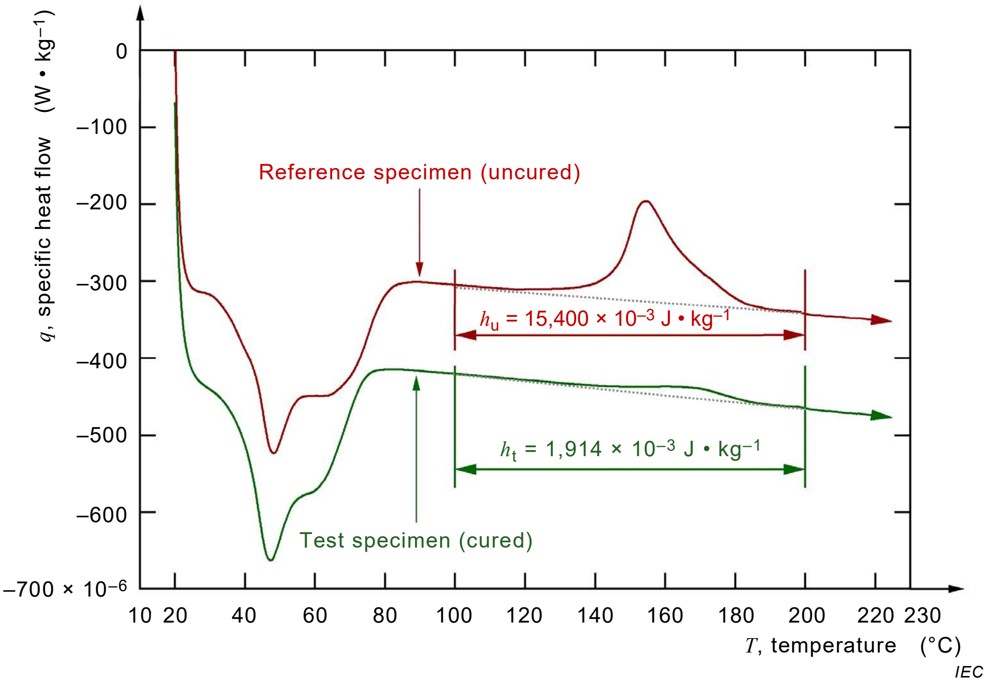


Figure 1 – Example result for the DSC residual enthalpy method

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):

$$G_a = \left(\frac{G_c + G_o + G_{SF}}{3} \right) \tag{2}$$

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

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

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

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.

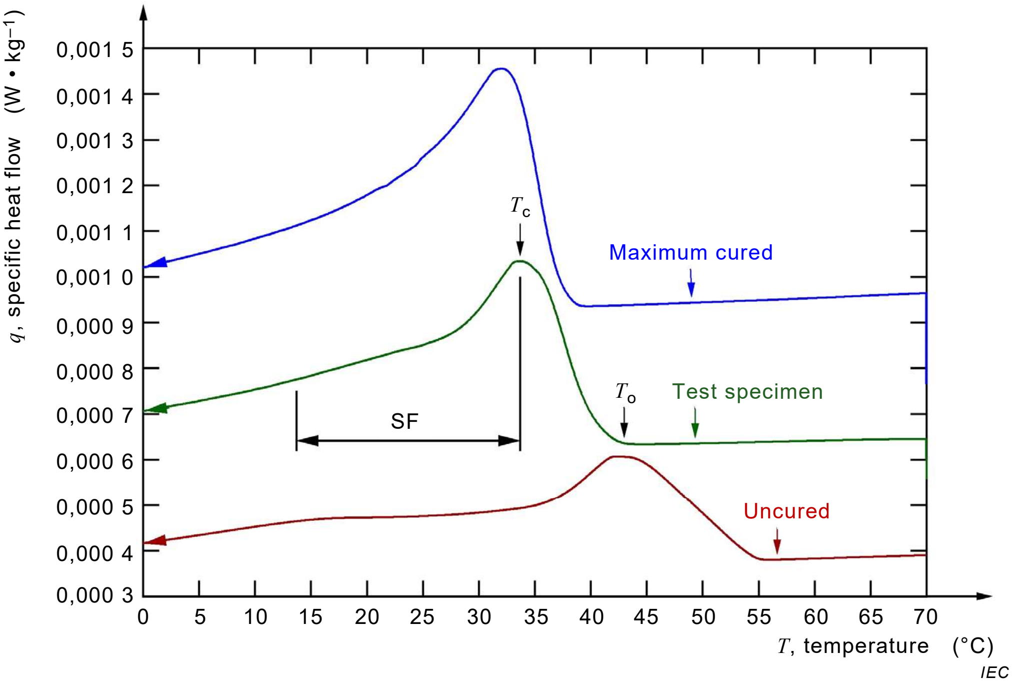


Figure 2 – Location of temperatures and temperature ranges used in the melt/freeze DSC method

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.

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

MEASUREMENTS	SPECIMEN	T_c °C	T_o °C	SF %
	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.

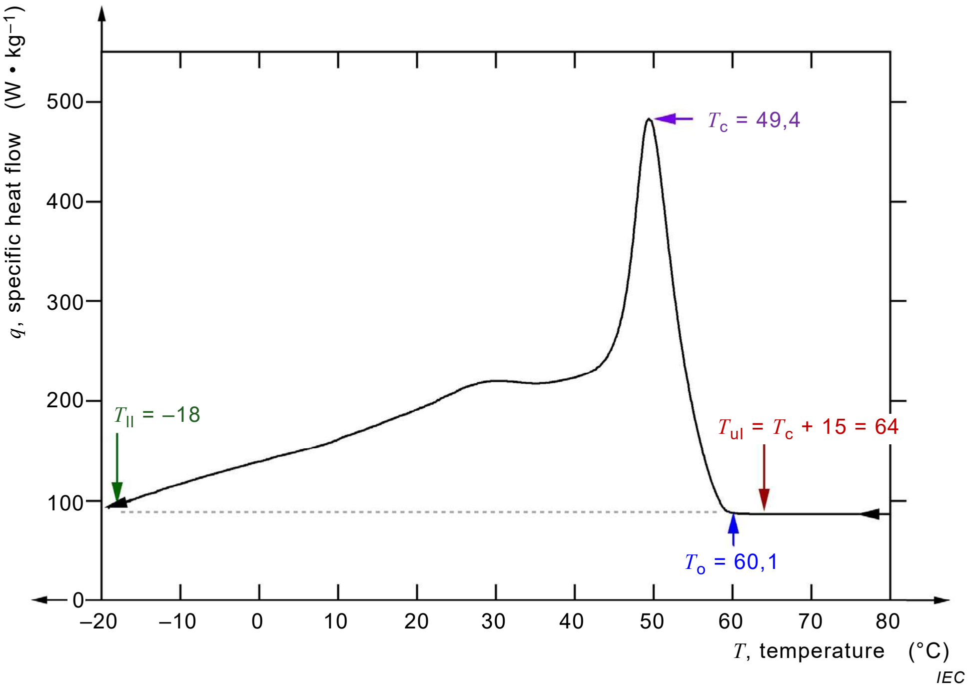


Figure 3 – Example of the temperature bounds applied for an automated software integration algorithm

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.

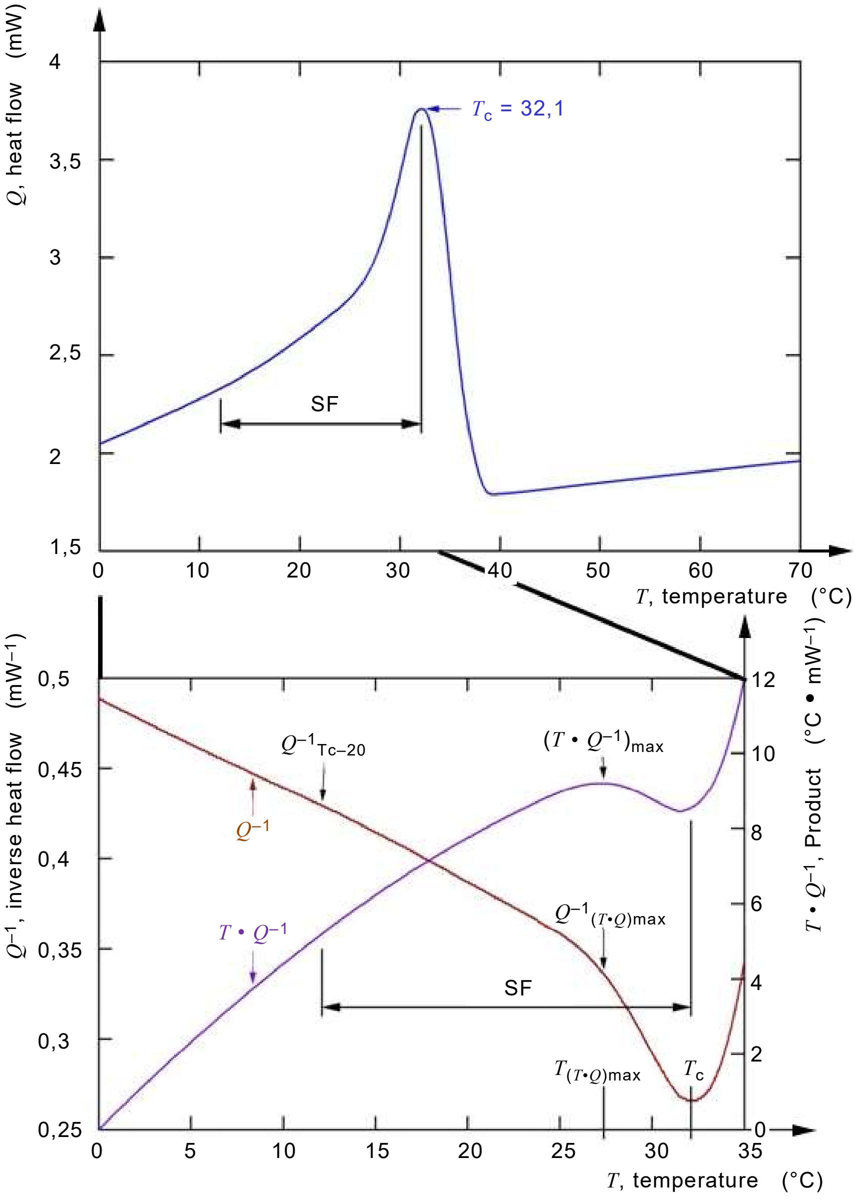
5.5.2.2.4 Identify the coordinates, [T, $T \cdot Q^{-1}$]max, for which the product reaches a maximum.

5.5.2.2.5 Calculate the shape factor from Formula (6):

$$SF = \frac{\left[T \cdot Q^{-1}\right]_{\text{max}}}{T_{\text{c}} \cdot \left[Q^{-1}\right]_{Tc-20}} 100 \tag{6}$$

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}, $[T \cdot Q^{-1}]_{\text{max}}$, is labelled in the bottom of the figure, along with: the temperature at $[T \cdot Q^{-1}]_{\text{max}}$, $T_{[\text{T}\cdot\text{Q}]\text{max}}$; the inverse heat flow at $T_{[\text{T}\cdot\text{Q}]\text{max}}$, $Q^{-1}_{[\text{T}\cdot\text{Q}]\text{max}}$; 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 $T_{[\text{T}\cdot\text{Q}]\text{max}}$ = 27,4 °C, $Q^{-1}_{[\text{T}\cdot\text{Q}]\text{max}}$ = 0,336 mW^{-1}, T_{c} = 32,1 °C, and $Q^{-1}_{\text{Tc} - 20}$ = 0,429 mW^{-1}.



IEC

Figure 4 – Representation of the measurement profile for an EVA test specimen

5.6 Uncertainty of measurements for the secondary method

When multiple specimens are examined from the same encapsulation sample or module fabrication batch, the uncertainty of measurements for DSC should be reported for a 95 % confidence interval.

6 The primary method

6.1 Principle for the primary method

The results from the DSC residual enthalpy and melt/freeze methods may vary according to the formulation (additives present), molecular weight (M_w) or vinyl acetate content (VaC) of the EVA. The DSC residual enthalpy method could also readily vary with the type and concentration of peroxide originally present in the EVA. A more absolute method of assessing the degree of cure is desired, because the DSC methods provide "secondary" results, i.e., the results depend on the make of EVA examined. The method of "gel content analysis" also characterizes the degree of cure, providing results that may be interpreted more universally. To clarify the gel content method does not measure the degree of cure directly, but is used to infer the degree of cure based on the solubility of the material that is not cross linked. The gel content test may be applied to any EVA specimen, regardless of its thermal history (including EVA from fielded modules), to quantify its degree of cure. Some of the limitations for the primary method for the gel content test, described in Annex A, are addressed by the standardized test procedure for the primary method.

6.2 Instrument and equipment for the primary method

6.2.1 Electronic balance

An electronic balance shall be used with a measurement accuracy ≤ 1 mg.

6.2.2 Soxhlet extractor

A Soxhlet or similar (*e.g.*, Kumagawa) extractor [1] shall be used for the gel content measurements. To prevent the risk of explosion of a closed system, the condenser for the extractor may use a pinhole or pressure relief device. The distillation path of the extractor (containing the thimble) shall be cleaned prior to use to improve the accuracy of the measurement, i.e., to remove EVA condensed from a prior test or to prevent contamination with other chemicals. It is recommended to use PTFE sleeves for all ground joints in the apparatus, for example to avoid the inadvertent dissolution of joint grease.

NOTE The use of a closed loop extractor system and thimble improves the accuracy of measurement over a flask/reflux condenser as in ISO 10147 and ASTM D2765 or sealed bottle as is sometimes used in the industry. The use of a Soxhlet extractor is identified here to provide the greatest accuracy, when the gel content test is used to calibrate the DSC methods. The procedure here does not prohibit the use of other apparatuses or established methods, such as a pressure relieved jar, if the gel content test is used for other purposes, for example, supplemental inspection of encapsulation, for process control or inspection of incoming goods in mass production.

6.2.3 Thimble

A standard single thickness thimble with a wall thickness of approximately 1 mm (10,0 μm nominal particle retention) shall be used to contain the specimen within the distillation path. An alpha cellulose cotton linter or glass fibre thimble may be used. For cellulose thimbles, the manufacturer’s recommended operating temperature range should be consistent with the Soxhlet extraction process (140 °C for xylenes). The specimen may also be placed inside a stainless steel screen bag or pouch that is placed within the thimble.

6.2.4 Heating apparatus

A hot plate, heating mantle, or similar apparatus shall be used to control the temperature of the still pot. If a hot plate is used, the still pot may be situated within a heated container containing a liquid, such as silicone or mineral oil. Electronic stirring capability in addition to the heating apparatus may be used to maintain a uniform temperature within the still pot of the extractor and prevent it from boiling over. Alternatively, boiling chips may be added to the still pot to prevent boiling over.

For safety, it is preferred to monitor and regulate the temperature of the heating apparatus as close to the still pot as is practical. For example, if a heated container is used, the temperature should be examined within the heated liquid and not at the hot plate.

At the end of the experiment, the same hot plate may be used to dry the specimen. Alternatively, a vented or vacuum oven may be used to dry the specimen. The accuracy of temperature measurement for the inside of an oven shall be at least ±1 °C.

---

[1] This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of these products.

6.2.5 Handling apparatus

Tweezers, tongs, or similar devices shall be used to handle the prepared specimens or thimble. Metallic tweezers with no polymeric coating shall be used to prevent spurious contamination of the extractor.

6.2.6 Solvent

Reagent grade xylene (mixed, CAS 1330-20-7, containing the meta-, ortho- and para-isomers) shall be used as the solvent for the gel content test. The solvent shall be replaced prior to each characterization to ensure accurate results.

An antioxidant shall be added (2 % wt. of the EVA) to the thimble to prevent further cross-linking of the specimen and improve the longevity of the solvent. Antioxidants used in EVA solvent extraction include: 2,6 di-tert-butyl-4-methylphenol (butylated hydroxytoluene, "BHT", CAS 128-37-0); pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate) (e.g., Irganox ®1010, CAS 6683-19-8); octadecyl-3-(3,5-di-tert.butyl-4-hydroxyphenyl)-propionate (e.g., Irganox ®1076, CAS 2082-79-3); 2,2-methylene-bis(4-methyl-6-tert-butylphenol), CAS 119-47-1; or 3-(3,5-di-tert-butyl-4-hydroxyphenyl), CAS 20170-32-5.

Solvents other than xylenes, such as toluene or tetrahydrofuran (THF), are sometimes used in the literature to study the degree of cure of EVA. When used with a Soxhlet extractor, these solvents would inherently have a different operating temperature (boiling point) and may require different extraction times. When comparing data between institutions, a correlation is required to interpret the results relative to the primary method when solvents (or procedures) other than that described in Clause 6 (using xylenes) are used.

6.3 Specimen preparation for the primary method

6.3.1 Sampling and storage

6.3.1.1 To reduce the variability associated with different formulations of EVA, test specimens for the primary should come from the same manufacturer and fabrication batch of EVA examined using the secondary method.

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

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

6.3.1.4 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 the results of the primary method do not change with storage time.

6.3.1.5 A minimum of 2 samples shall be used from a roll of EVA to compare with the secondary method.

6.3.2 Preparation procedures

6.3.2.1 The thimble should be baked at (110 ± 2,5) °C for 2 h to remove moisture, and then cooled to ambient temperature in a dessicator to prevent moisture absorption.

6.3.2.2 Record the mass of the empty thimble, m_t.

6.3.2.3 Prepare EVA specimens of the nominal size (1 ± 0,5) g. If possible, specimens shall be obtained from a single sheet of material. The accuracy of the measurement of the recorded specimen mass, m_i, should be within 1 %. The specimen may be cut to smaller pieces to assist in fitting in the thimble or preventing it from swelling and escaping the thimble during extraction.

NOTE One gram of 0,45 mm thick EVA sheet will be approximately 4,7 cm × 4,7 cm in size.

6.4 Test requirements for the primary method – Environment requirements

As in ISO 291, the recommended laboratory environment is: (25 ± 2) °C and the relative humidity of (50 ± 5) %.

6.5 Test procedure for the primary method

6.5.1 The gel content test shall be carried out as follows.

6.5.2 Use a handling apparatus to place the specimen in the thimble, and then place the thimble within the distillation path of the extractor.

6.5.3 Fill the still pot of the extractor with at least 4 to 5 times the nominal volume of the extractor chamber of xylene and assemble it to the extractor. The volume of solvent in the still pot shall run between 1/3 and 2/3 the nominal volume of the still pot during the test.

6.5.4 The combination of the still pot size and heating power shall be chosen such that the extraction is performed for ≥ 15 cycles per hour.

6.5.5 Maintain the still pot at the test temperature for at least 8 h. For specimens with expected gel content below 70 %, the minimum test time of 12 hours is recommended.

6.5.6 Remove the thimble and specimen from the extractor and place them in a secondary container such as a funnel or beaker.

6.5.7 Place the secondary container in/on a heating apparatus, maintained at (115 ± 2,5) °C.

6.5.8 Dry the secondary container (with the specimen and thimble) to constant weight at the drying temperature for at least 4 h.

6.5.9 Remove the thimble and specimen from the secondary container, allow them to cool to room temperature, and record their combined final mass. The thimble containing the specimen should be cooled in a desiccator to prevent moisture absorption.

It is recommended to verify that the results do not change with time, i.e. that the extraction in 6.5.5 and the drying performed in 6.5.8 is complete.

6.6 Calculation and expression of the results for the primary method

The gel content shall be calculated using Formula (7):

$$G_{\%} = \frac{m_{\mathrm{f}} - m_{\mathrm{t}}}{m_{\mathrm{i}}} 100 \qquad (7)$$

In the formula, $G_{\%}$ represents the cross-linked gel content, %; m_f, the final combined mass of the thimble and encapsulation specimen after drying, g; m_t, the initial mass of the thimble before extraction, g; and m_i, the initial mass of the encapsulation specimen before extraction.

7 Test report

A report of the tests including at least the following information shall be prepared. The report should contain the detail specification for the specimens.

a) a title;

b) name and address of the test laboratory and location where the tests were carried out;

c) unique identification of the certification or report and of each page;

d) name and address of client, where appropriate;

e) description and identification of the item tested, including the specimen size;

f) characterization and condition of the test item;

g) date of receipt of test item and date(s) of test, where appropriate;

h) identification of test method used and test instrument and other equipment used, including the laminator and the temperature, pressure, and time settings used, when applicable. In the case of the indentation secondary method (per correlation or usual use of method), the test temperature, tip material, tip geometry, and tip size, maximum indentation load, maximum indentation depth, and frequency of modulation (if applicable) shall also be reported;

i) reference to sampling procedure, where relevant, including the number of tests per specimen;

j) any deviations from, additions to, or exclusions from, the test method and any other information relevant to a specific test, such as environmental conditions, gas content and flow rate to the oven;

k) measurements (associated and their uncertainty), examinations and derived results supported by tables, graphs, sketches and photographs as appropriate including degree of cure, specimen mass, measured enthalpy, graphs of the enthalpy/temperature data, graphs of the crystallization peaks, and gel content;

l) a statement of the estimated uncertainty of the test results (where relevant);

m) a signature and title, or equivalent identification of the person(s) accepting responsibility for the content of the certificate or report, and the date of issue;

n) where relevant, a statement to the effect that the results relate only to the items tested;

o) a statement that the certificate or report should not be reproduced except in full, without the written approval of the laboratory.

8 Indentation secondary method

8.1 General

An alternative secondary method, using indentation to characterize the degree of cure of EVA, has emerged from the PV industry. The method is presently being used by module manufacturers and has been demonstrated in in-line application. A general description of the principle, related equipment, and use of the method is given herein.

G_i is obtained using an indentation instrument, where the result may follow from the analysis of the applied load, penetration depth, use of a modulated applied load (such as harmonic stiffness, storage modulus, loss modulus, or tan[δ]), and/or the specimen viscoelastic response (including the relaxation or recovery response). G_i may be obtained from a more complicated dimensionless fit, including a fit applied to the specimen's viscoelastic response (e.g., using a Maxwell model) or a combination of characteristics monitored during indentation. G_i may be obtained from a dimensionless fit of the response of the specimen (for example

$$G_i = \frac{C_\mathrm{t} - C_\mathrm{n}}{C_\mathrm{m} - C_\mathrm{n}}$$

where

C is the characteristic of interest,

C_t is the test specimen;

C_m is the reference specimen with the greatest thermal history (“maximum correlated”, e.g., most-cross-linked); and

C_n is the reference specimen with minimal or no thermal history ("minimum correlated", i.e., not-laminated or not-cured).

NOTE A suitable apparatus is supplied by LayTec AG[2] in Germany. For this presently available commercial equipment, G_i can range from 0,3 to 2,5..

The terminology and definitions related to dynamic indentation, including harmonic stiffness, storage modulus, loss modulus may be found in ISO 14577-1. The terminology and definitions related to tan[δ] may be found in ISO 6721-1.

8.2 Principle

The indentation method probes the mechanical response (viscoelastic characteristics) of cured EVA layers, which change due to the cross-linking of the EVA during the curing process. In the method, a probe tip (connected to a force transducer) is pressed against the flexible back-sheet affecting the EVA. The corresponding reaction force acting on the tip during indentation and subsequent relaxation is recorded by the force transducer and analyzed, giving a figure of merit (G_i) describing the viscoelastic properties of the material. The principle of the indentation method is described in the related references in the bibliography.

A correlation between the degree of cure and the gel content can be established by a series of measurements on samples, each with a different degree of cure. Indentation can thus be used as a secondary method to quantify the degree of cure. To enable more widespread comparison, the same set of sample materials shall be characterized using the gel content method (Clause 6 of IEC 62788-1-6) after indentation to establish a correlation between gel content and the degree of cure. The indentation method is valid for the specific combination of EVA and back-sheet examined.

This test procedure may also be applied to cross-linking ethylenic co-polymers other than EVA. The maximum tip displacement, maximum applied load, and temperatures identified for the indentation measurements in this procedure have been optimized for EVA. For other materials, the optimum temperature depends on the stack of materials subject to indentation, including the encapsulant and backsheet.

The method and instrumentation is designed for non-destructive examination of PV modules with a flexible back-sheet for the purpose of manufacturing process control. The method may not be applied to modules with a glass/glass laminated construction. The indentation method may be used for quality control in production lines. The variation of the method (one standard deviation) is typically ≤ ±3 % (see Kunath et al.).

8.3 Instrument and equipment for the indentation method

Equivalent instruments that contain the following components and can be shown to provide repeatability and reproducibility of ≤ ±5 % for two standard deviations for correlation of G_i as defined in Section 3.6 may be used.

The instrument consists of:

- a rigid probe;
- a force transducer connected to the probe, measuring the reaction force acting on the probe during indentation;
- a temperature regulation system, to maintain the test specimen (module) at a designated temperature;
- a mechanically rigid linear stage that may be used to move the probe and force transducer for site-specific indentation.

---

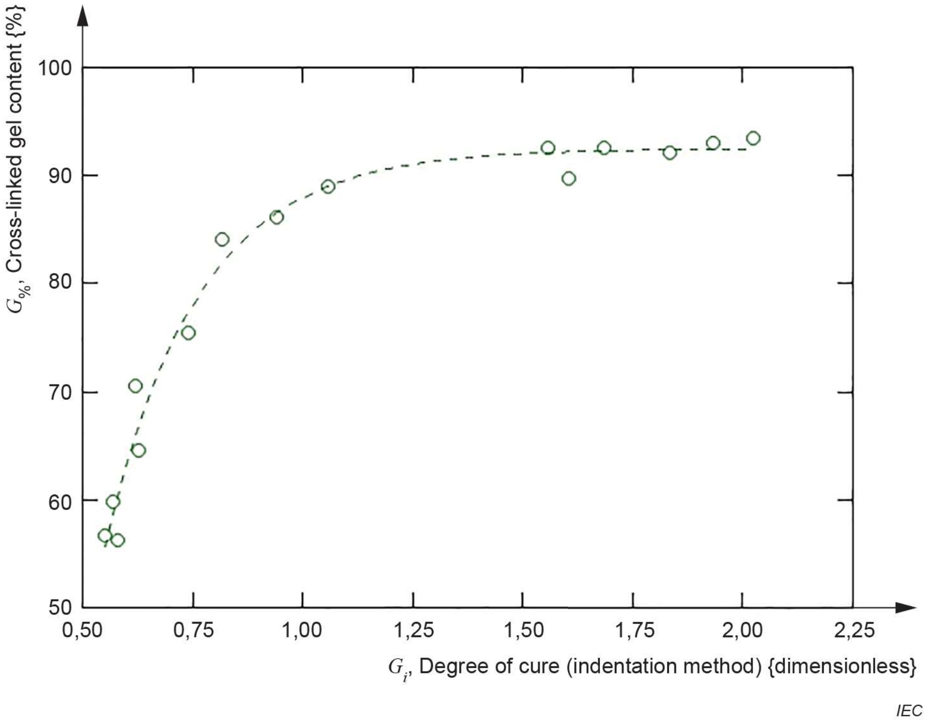
[2] This information is given for the convenience of users of this International Standard and does not constitute an endorsement by IEC of the product named.

8.4 Instrument calibration

The tip, force transducer, and temperature regulation system shall be verified and calibrated regularly according to the specifications of the equipment vendor.

8.5 Correlation of the degree of cure

For EVA, to establish a correlation between the figure of merit (G_i) given by the instrument and the gel content, a set of specimens with different cross-linked gel content ($G_{\%}$) including the range of the manufacturer's specification limits (e.g., between 70 % and 93 %) shall be used. Specimens for correlation, usually prepared by varying the lamination time or temperature, shall first be examined using the indentation tester. The correlation specimens shall be subsequently analysed using the gel content method (Clause 6 of IEC 62788-1-6) so that an empirical correlation (best fit or series of best fits through the range of correlation) between $G_{\%}$ and G_i is established. Figure 5 (similar to Lux et al.) shows an example of a correlation for an EVA material. The figure shows an example of correlation data for a single material rather than the application of indentation to multiple module specimens, therefore no error bars are given. Because of the nonlinear nature of the curing process, the results of the secondary methods (including indentation) should not be extrapolated beyond the range of $G_{\%}$ and G_i of the correlation specimens.



An example of the empirical correlation (dashed line for a first order exponential fit) is shown for the correlation data (circles) for a representative combination of encapsulant and backsheet.

Figure 5 – Example of the correlation applied between G_i (indentation) and $G_{\%}$ (gel content)

8.6 Specimen preparation for the indentation method

The indentation method does not require sample specific preparation. Test specimens may consist of full-size modules or mini-modules. Specimens shall be placed in the test device with the backsheet-side facing the probe tip. Measurements shall only be conducted at locations within the specimen where just a cell is present (i.e., not between cells or at a location where the interconnect ribbon is present) and at least 10 mm away from the edge of a cell. The measurement locations should be clean and without defects or damage in the specimen materials.

If measurements are performed for the purpose of correlation between $G_{\%}$ and G_i, subsequent preparation requirements for the gel content method may apply.

8.7 Test procedure for the indentation method

A module specimen is placed under the indentation test probe, with both the specimen and probe being held rigidly in place. The specimen is heated to a temperature above the melt transition temperature of the encapsulant (for example, within the range of 85 °C and 95 °C for EVA) and maintained constant at the designated temperature ±1 °C for all measurements. For example, heating can be achieved from below with a heater, such as a halogen light source that irradiates the front (glass side) of the PV module. When the designated test temperature is achieved and stabilized, the probe is pressed into the backsheet/encapsulant stack and the measurement is performed. For contemporary backsheet materials laminated on EVA encapsulant, the stabilization time of at least 45 s is recommended; however, shorter stabilization times may be used if validated for indentation. For contemporary backsheet materials laminated on EVA encapsulant, the maximum indentation depth may be in the order of 120 µm and the applied load may be in the order of 15 N. The optimum measurement temperature range for crosslinking encapsulant materials other than EVA has not yet been established. If the test procedure is applied to other encapsulation materials, the range of the test temperatures may have to be adjusted.

The measurement for the indentation method gives the figure of merit, G_i, characterizing the degree of cure based on the viscoelastic response of the specimen.

Annex A
(informative)

Limitations of the primary and secondary measurement methods

A variety of methods may be used to characterize the degree of cure of EVA, including: dynamic mechanical analysis (DMA); rheometric cure metering, gel content; swell ratio; differential scanning calorimetry (DSC, both the residual enthalpy and melt/freeze methods); indentation; vibrometry; scanning acoustic microscopy; chemical analysis (including Fourier transform infrared spectroscopy and Raman spectroscopy); mechanical elongation; and spectrophotometry, for example, haze measurement. While DMA (which among its different possible applications may refer to a parallel plate or cone/plate rheology measurement, performed in the melt state) most directly verifies the desired mechanical characteristics for EVA, the method is cumbersome. Specifically, DMA requires a relatively large volume of test material, requires careful sample preparation, is time consuming, and makes use of specialized equipment. DMA results, however, directly correlate with the molecular structure of EVA, including the cross-link density and the mean molecular weight between crosslinks within the polymer network. Rheometric metering, which may be performed using a rotary instrument, also directly examines the mechanical properties. Rheometric metering is subject to many of the same limitations as DMA, including large sample size. The other methods have limitations and requirements, as described.

Gel content is foremost a time consuming process, typically requiring 8 h to 24 h. Factors that may limit the accuracy of the test, include the multiple mass measurements (each with an associated uncertainty), and possible cross-contamination (samples are transferred in weighing/dissolving containers between ovens to perform the processes of filtering, drying, and weighing). The method furthermore makes use of heated, toxic chemicals, a potential safety hazard. Gel content specifically examines the solubility of EVA, which is presumed to be negligible for the cross-linked portion of the EVA. A hazard of the gel content test is that EVA specimens cured below a minimally cured threshold may entirely dissolve through the thimble or filter. To explain, the terminal CH_3 groups participating in cross-linking are on the order of 1 % of the available CH_3 groups, even in "maximum cured" EVA. The range of measurement from 60 % (where the thimble may trap the gel effectively) to 90 % (above which only the dissolving of additives may be detected) has been suggested for the gel content method. Separately, inadvertent curing of the specimen during the test would compromise the results. This possibility is minimized in a Soxhlet extractor, because the solvent temperature will be decreased below its boiling point upon condensation; furthermore an antioxidant is added to the thimble to specifically mitigate chemical reactions, such as curing. In addition to cross-linking, solubility may be influenced by other parameters (such as crystallinity). The gel content test does not provide information regarding the cross-linked structure, for example, the average number of bonds between molecules. If correctly performed, however, gel content should correlate well with the results of rheological tests.

While the DSC methods may both be applied over a greater range of degree of cure than the gel content method, it is suggested that quantitive correlation to the gel content method be limited to the range of 60 % to 90 % gel content, based on the effectiveness of the gel content method. One practical limitation of the DSC residual enthalpy method is that it cannot be correlated to a gel content measurement until a sufficient curing (threshold) has been performed. Another limitation of DSC residual enthalpy method is that its correlation (calibration) may be unique to each formulation of EVA examined. In particularly, the results would be expected to readily vary with the type and concentration of peroxide originally present in the EVA. The DSC residual enthalpy method examines the heat of reaction for the peroxide-enabled curing of the EVA. The measurement is therefore indirect (it does not query the molecular structure) and subject to aging, for example, evaporation of the peroxide during storage. Furthermore, EVA formulations often contain additional peroxide that does not contribute to cross-linking of the EVA, but would be characterized by DSC.

One limitation of the DSC melt/freeze method is that it is subject to the characteristics of the base EVA resin. For example, variation in the molecular weight ($\mathrm{M_w}$) or vinyl acetate content (VAc) could affect the results of the DSC melt/freeze method. The results of the DSC melt/freeze method shall therefore be recalibrated using a primary method if base resin is changed, for example, from 33 % to 28 % VAc. For example, the degree of cure associated with the concavity will readily become reduced with VAc. The degree of cure associated with the crystallization or onset of crystallization may vary in an even more complex manner with VAc. The DSC melt/freeze method, however, provides three different estimates for the degree of cure; it is therefore recommended to apply all three estimates to determine the degree of cure from their average. The DSC melt/freeze method is a more direct measurement than the DSC residual enthalpy method, because the phase transition varies with the molecular structure of the EVA. The DSC melt/freeze method would incorporate the effects of degradation of the EVA (as opposed to degradation of the peroxide, which is examined using the DSC residual enthalpy method).

In addition to the decomposition of peroxide with time (which affects the results of the DSC residual enthalpy method), and UV facilitated cross-linking with age (which affects the results of the DSC melt/freeze method), the DSC characterization of EVA obtained fielded modules may be affected by other factors, including the other components within the PV module. Contamination or interaction with components within a module, such as cell coatings, ribbon and string surfaces, flux residues, back sheet primers, and adhesive layers, should be considered when EVA obtained from field-deployed modules is examined.

Inline measurement capability has been recently demonstrated for indentation, which may find more widespread use as an alternate secondary method. Advantages of the method include the non-destructive nature of the test, ability to use the method on product modules (rather than test coupons or scrap modules), ease of automation, and the speed of measurement (on the order of seconds). The method may also be used as a research tool to map the distribution of the degree of cure across the module.

One limitation of the indentation method is that the results are unique to the combination of encapsulant and backsheet used in the module, and therefore must be correlated against another method (such as gel content) to be interpreted. Conversely, the method cannot be used for glass/glass modules as indentation on a rigid glass outer surface would mask the deformation of and interior-located encapsulation layer. Because the mechanical response for EVA is temperature sensitive, the indentation method does require temperature control for the specimen – for example, whether the modules are measured at the same interval after lamination in the manufacturing line, after controlled temperature modulation downstream on the manufacturing line, or after reheating to a designated temperature. Like the DSC methods, the indentation method would be affected by field aging, where UV or temperature mediated crosslinking could alter the results to be different from the recently laminated condition.

Additional details and limitations of the secondary measurement techniques, when applied to the evaluation of the degree of cure of EVA, may be found in the bibliography.

Bibliography

IEC 61215 (all parts), *Terrestrial photovoltaic (PV) modules – Design qualification and type approval*

ISO 11357-2, *Plastics – Differential scanning calorimetry (DSC) – Part 2: Determination of glass transition temperature and glass transition step height*

ISO 11357-3, *Plastics – Differential scanning calorimetry (DSC) – Part 3: Determination of temperature and enthalpy of melting and crystallization*

J.A. Reyes-Labarta, M.M. Olaya, A. Marcilla, *"DSC and TGA study of the transitions involved in the thermal treatment of binary mixtures of PE and EVA copolymer with a crosslinking agent", Polym., 47, 2006, 8194-8202*

Z. Xia, D.W. Cunningham, J.H. Wohlgemuth*, "A Non-solvent Extraction Method for Measuring Gel Content of Ethylene Vinyl Acetate", Proc. IEEE PVSC 2009, 124-126*

Heng-Yu Li, Laure-Emmanuelle Perret-Aebi, Ricardo Théron, Christophe Ballif, Yun Luo, Ronald F. M. Lange, "Towards in-line Determination of EVA Gel Content During PV Modules Lamination Processes", Proc. Euro PVSEC 2010, 4044-4046

P. Ye, A. Salomon, K. Menard, Kevin*, "EVA curing study by DSC for solar application", Proc. 68th Annual Technical Conference – ANTEC, 1, 2010, 608-610*

K. Agroui, G. Collins, J. Farenc*, "Measurement of Glass Transition Temperature of Crosslinked EVA Encapsulant by Thermal Analysis for Photovoltaic Application", Renew Energy, 2011*

M. Hidalgo, F. Medlege, M. Vite, C. Corfias-Zuccalli, P. Voarino, J. Gonzalez-Leon, *"New DSC Method for the Quality Control of PV Modules: Simple and Quick Determination of the Cross-Linking Degree of EVA Encapsulants, and Other Properties", PV Intl. J., 14, 2011, 130-140*

H.Y. Li, L.-E. Perret-Aebi, R. Théron, C. Ballif, Y. Luo, R.F.M. Lange, *"Optical Transmission as a Fast and Non-Destructive Tool for Determination of EVA Curing State in PV modules", PIP, 2011. DOI: 10.1002/pip.1175*

R. Mickiewicz, *"Non-Destructive Measurement of the Degree of Cross-Linking of EVA Solar Module Encapsulation", Proc. ATLAS/NIST Work. PV Mats. Durability, 2011*

J. Lloyd, T. Christian, D. Doble, R.A. Mickiewicz, *"Non-Destructive Measurement of the Degree of Cross-linking of EVA in Solar Module Encapsulation", Proc. IEEE PVSC, 2011*

W.Mühleisen, M. Biebl-Rydlo, M. Spielberger, "Determining the Degree of EVA Cross-Linking in Assembled PV Modules Acoustically and In-Situ", Proc. Euro PVSEC, 2011, 3480-3483

S.-H. Schulze, M. Pander, S. Dietrich, M. Ebert, *"Encapsulation Polymers – a Key Issue in Module Reliability", PV Intl, 2011, 118-126*

W. Stark, M. Jaunich, *"Investigation of Ethylene/Vinyl Acetate Copolymer (EVA) by Thermal Analysis DSC and DMA", Polym Test., 30, 2011, 236-242*

W. Stark, M. Jaunich, W. Bohmeyer, K. Lange, *"Investigation of the Crosslinking Behaviour of Ethylene Vinyl Acetate (EVA) for Solar Cell Encapsulation by Rheology and Ultrasound", Polym Test., 31, 2012, 904-908*

M. Winkler, R. Baeuerlein, B. Goetz, F. Sandau, *Proc. Euro. PVSEC, 2012, 3482-3485*

K. Agroui, G. Collins*, "Determination of Thermal Properties of Crosslinked EVA Encapsulant Material in Outdoor Exposure by TSC and DSC Methods", Renewable Energy, 2013*

B.S. Chernev, Ch. Hirschl, G.C. Eder, "Non-Destructive Determination of Ethylene Vinyl Acetate CrossLinking in Photovoltaic (PV) Modules by Raman Spectroscopy", Appl. Spectroscopy, 67 (11), 2013, 1296-1301

Ch. Hirschl, M. Biebl-Rydlo, M. DeBiasio, W. Mühleisen, L. Neumaier, W. Scherf, G. Oreski, G. Eder, B. Chernev, W. Schwab, M. Kraft, *"Determining the Degree of Crosslinking of Ethylene Vinyl Acetate Photovoltaic Module Encapsulants — A comparative Study", Solmat, 116, 2013, 203-218*

Y.S.H. Johnson, Y.-L.L. Lawrence, D. Israel, M. Baiardo, Y.-H. Chou, G.-Z. Fan, S.-Y. Huang, *"Simple Modeling for Quick Determination of the Gel Content of EVA Encapsulants in PV Module Fabrication", Proc. Euro. PVSEC, 2013*

H.-Y. Li, L.-E. Perret-Aebi, R. Théron, C. Ballif, Y. Luo, R.F.M. Lange, *"Optical Transmission as a Fast and Non-Destructive Tool for Determination of Ethylene-co-Vinyl Acetate Curing State in Photovoltaic Modules" PIP, 21, 2013, 187-194*

E. Malguth, B. Buhl, T. Schenk, "Fast and Non-Destructive Determination of the EVA Cross-Linking Degree for Inline and Off-Line Application", Proc Euro PVSEC, 2013, 472-475

D.C. Miller, X. Gu, S. Haldeman, M. Hidalgo, E. Malguth, C.G. Reid, T. Shioda, S.-H. Schulze, Z.-Y. Wang, J.H. Wohlgemuth, *"Examination of a Standardized Test for Evaluating the Degree of Cure of EVA Encapsulation", Proc Asian PVSEC, 2013*

A. Morlier, S. Klotz, S. Sczuka, I. Kunze, I. Schaumann, S. Blankemeyer, M. Siegert, T. Döring, T. Alshuth, U. Giese, M. Denz, M. Köntges, *"Influence of the curing state of Ethylene-Vinyl-Acetate on photovoltaic modules aging", Proc. Euro PVSEC, 2013, 2832-2837*

M. Hidalgo, D. Thil, C. Dugué*, "Assessment of the Cross-Linking Degree of EVA PV encapsulants" PV Intl., 2013, 98-103*

C. Lux, U. Blieske, E. Malguth, N. Bogdanski, *"Variations in Cross-link Properties of EVA of Un-aged and Aged PV-modules", Proc. Euro PVSEC, 2014*

G. Oreski, "Advanced Methods for Determining PV Module Process Optimization Potential", PV Intl, 2014, 71-78

G. Oreski, A. Rauschenbach, Ch. Hirschl, L. Neumaier, M. Kraft, G. Pinter, "Challenges in Measuring the Degree of Cross-Linking of Ethylene Vinyl Acetate", Proc. Euro. PVSEC, 2014, 2457-2461

Ch. Hirschl, L. Neumaier, W. Mühleisen, M. DeBiasio, R. Leitner, M. Zauner, G. Oreski, A. Rauschenbach, G. C. Eder, S. Seufzer, Ch. Berge, E. Rüland M. Kraft, "In-Line Quality Control Device for the Degree of Crosslinking of the Embedding Material", Proc. Euro PVSEC, 2015, 5BV4.3

Ch. Hirschl, L. Neumaier, W. Mühleisen, M. DeBiasio, G. Oreski, A. Rauschenbach, "Post-crosslinking in Photovoltaic Modules Under Different Conditions", Proc. Euro PVSEC, 2015

S. Kunath, Marco Vogel, Michael Mette, Enno Malguth, "Evaluation of Gel Conent-Measurement-Method for Mass Production of Glass-Foil Based Crystalline Silicon PV Modules", Proc. Euro. PVSEC, 2015

H.-Y. Li, Y. Luo, C. Ballif, L.-E. Perret-Aebi, "Fast and Nondestructive Detection on the EVA Gel Content in Photovoltaic Modules by Optical Reflection", IEEE J. PV, 2015

S.-H. Schulze, A. Apel, D. Dassler, C. Ehrich, "Cure State Assessment of EVA-Copolymers for PV-Applications Comparing Dynamic-Mechanical, Dielectric and Calorimetric Properties", SOLMAT, 143, 2015, 411–417

Ch. Hirschl, L. Neumaier, W. Mühleisen, M. Zauner, G. Oreski, G.C. Eder, S. Seufzer, Ch. Berge, E. Rüland, M. Kraft, "In-line determination of the degree of crosslinking of ethylene vinyl acetate in PV modules by Raman spectroscopy", SOLMAT, 52, 2016,10–20

D. Chen, A. Green, D. Dollimore, "DSC: the Importance of Baseline Calibration", Thermochimica Acta, 284 (2), 1996, 429–433

Dan Doble, Rafal Mickiewicz, John Lloyd, Marco Jaeger, William F. Hartman, “Methods and Apparatus for Detecting Cross-Linking in a Polymer”, patent application US 20120118071 A1, 2012

B.-M. Chen, C.-Y. Peng, J.-L. Cho, G.A. Porter, "Optimization of Solar Module Encapsulant lamination by Optical Constant Determination of Ethylene-Vinyl Acetate", *Int. J. Photoenergy*, 2015, 1-7

C. Camus, C. Buerhop-Lutz, S. Wrana, J. Adams, T. Pickel, H. Scheuerpflug, C. Zetzmann, E. Malguth, J. Hauch, C.J. Brabec, “Direct Evidence for Hot-Cell-Induced Modifications in PV Module Encapsulants”, *Proc. Euro PVSEC*, 2016, 5BO.10.6

K. Harms, L. Neumaier & C. Hirschl, Determination of Depth-Dependent Variations in the Degree of Crosslinking of EVA due to Changing Lamination Parameters Using Raman Spectroscopy, *Proc. Euro PVSEC Conf.*, 2019, 4AV.1.23

S. Ogier, C. Vidal, D. Chapron, P. Bourson, I. Royaud, M. Ponçot, M. Vite, M. Hidalgo, “A comparative study of calorimetric methods to determine the crosslinking degree of the ethylene-Co-vinyl acetate polymer used as a photovoltaic encapsulant”, *J. Polym. Sci.*, 2017. DOI: 10.1002/polb.24335

G. Oreski, A. Rauschenbach, C. Hirschl, M. Kraft, G.C. Eder, G. Pinter, “Crosslinking and Post-Crosslinking of Ethylene Vinyl Acetate in Photovoltaic Modules”, *J. Appl. Polym. Sci.,* 134 (23), 2017. DOI: 10.1002/app.44912

A.K. Singh, R. Singh, "The Effect of Degree of Gel Content of EVA Encapsulation on the Properties of Encapsulant Itself and Finally on the Reliability of the Solar Module in the Field", *RenewSys White paper*, 1-10, 2017

(*Continued on third cover*)

International Standard	*Title*
ISO 291 : 2008	Plastics — Standard atmospheres for conditioning and testing
ISO 6721-1	Plastics — Determination of dynamic mechanical properties — Part 1: General principles
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

Only the English language text has been retained while adopting it in this Indian Standard, and as such, the page numbers given here are not the same as in the IEC Publication.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

This Indian Standard has been developed from Doc No.: ETD 28 (18117).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor,
DMRC Building, Bhavbhuti Marg, New
Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V,
Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg,
Chandigarh 160019 — 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 — 2254 1442, 2254 1216

Western : Plot No. E-9, Road No.-8, MIDC, Andheri
(East), Mumbai 400093 — 2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

Published by BIS, New Delhi