IEC TS 62876-3-1

Edition 1.0 2022-02

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Reliability assessment –
Part 3-1: Graphene-based material – Stability: Temperature and humidity test

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CONTENTS

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NANOMANUFACTURING – RELIABILITY ASSESSMENT –

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FOREWORD

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IEC TS 62876-3-1 has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

The text of this Technical Specification is based on the following documents:

Draft	Report on voting
113/592/DTS	113/616/RVDTS

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

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

A list of all parts in the IEC TS 62876 series, published under the general title *Nanomanufacturing – Reliability assessment*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

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- withdrawn,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

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INTRODUCTION

Graphene, a single-layer of carbon atoms arranged in a honeycomb lattice, has a high potential for future nanotechnology applications due to the excellent conductivity, transparency and flexibility of the material. Many research organizations and industrial companies are developing fabrication technologies for graphene films on substrates, as dry powders or graphene in liquid dispersions for a wide variety of applications. Therefore, the need for a system of standardized tests for the key control characteristics to benchmark graphene material in its different physical and chemical modifications remains critical. The absence of such a system slows down the development process and hinders companies to be commercially successful. To improve this situation, IEC TC 113 is developing a comprehensive system of material specifications (IEC 62565-3-X series) which list application relevant key control characteristics (KCCs) and the related measurement standards (IEC 62607-6-X series) for graphene materials in general. That includes graphene, few-layer graphene and multilayer graphene, graphene oxide and other graphene-related materials.

In addition to the KCCs to qualify graphene material after fabrication, it is also important to obtain information on its long-term stability. This document defines the conditions for a set of stress tests to qualify graphene material for an acceptable level of reliability and durability in its performance category and operating service environment.

These tests are performed using samples with graphene material layers on the same substrate as is used in the final product. If the graphene material layer in the final product is embedded between other materials, the test samples are prepared in the same way. The basic idea is to prepare test samples which are representative for the application, so that the results of the reliability screening allow the prediction of the reliability of the layer in the final product. The results of these tests will help to establish acceptance criteria for the graphene material including its manufacturing process. This document does not address the quantitative reliability measures and life predictions of the product, which would involve further testing based on the knowledge of failure mechanisms.

The objectives of this document are to:

– specify the requirements for a general reliability stress screening (RSS) standard for nano-enabled electrotechnical products using graphene and other graphene-based materials;
– give direction to the supplier and to the end user on the production and purchase of nano-enabled electrotechnical products to meet and verify reliability qualification standards for certain specified service environments;
– provide a list of reliability qualification stress tests and conditions;
– establish guidance for selection of appropriate measurements and pass/fail criteria.
– give relevant references; and
– establish the minimum reporting requirements.

This document is meant to be a general document that can be applied to all modifications of graphene materials. Even if this document focuses on graphene materials, an analogue systematics might be useable for other 2D materials. Nevertheless, as it is expected that failure mechanisms are material dependent, a direct comparison of time-to-failure values is not recommended.

As such, the results of this reliability screening test give guidance to optimize graphene material fabrication processes and the use of graphene materials in a product design.

In some cases, it may be required that prior to the tests the environmental chamber be evacuated to vacuum so the intrinsic KCCs are measured (without influence from other environmental contaminants).

It is the intent of this document to be compatible with and work in conjunction with the performance standards defined in the IEC 62607 series.

**NANOMANUFACTURING –
RELIABILITY ASSESSMENT –**

**Part 3-1: Graphene-based material –
Stability: Temperature and humidity test**

1 Scope

This part of IEC TS 62876 establishes a standardized method to determine the

- stability

of films of graphene-based material by a

- temperature and humidity test.

It establishes a general methodology for reliability stress screening (RSS) to qualify the use of graphene-based material in its subsequent product value stage. The intention is to prepare test samples undergoing the same or similar failure mechanisms as the graphene-based material in the final product.

- Selected stress factors in this test to drive the failure mechanisms are low temperature, high temperature, change of temperature and damp heat with respect to test severity, test sequence, sample quantities and acceptance criteria.
- The RSS test procedure defined here supports reliability assessment in the design phase of a product by providing guidelines for selecting appropriate measurement methods and how to define specific pass/fail criteria.
- The described methodology does not replace traditional reliability tests on the final product level and will not provide full reliability data which allow the estimation of product lifetimes. If the samples pass the test, the design is called qualified in accordance with IEC 62876-3-1.

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 60068-2-1, *Environmental testing – Part 2-1: Tests – Test A: Cold*

IEC 60068-2-2, *Environmental testing – Part 2-2: Tests – Test B: Dry heat*

IEC 60068-2-14, *Environmental testing – Part 2-14: Tests – Test N: Change of temperature*

IEC 60068-2-78, *Environmental testing – Part 2-78: Tests – Test Cab: Damp heat, steady state*

3 Terms and definitions

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

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

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

3.1 General terms

3.1.1
key control characteristic
KCC
key performance indicator
material property or intermediate product characteristic which can affect safety or compliance with regulations, fit, function, performance, quality, reliability or subsequent processing of the final product

Note 1 to entry: The measurement of a key control characteristic is described in a standardized measurement procedure with known accuracy and precision.

Note 2 to entry: It is possible to define more than one measurement method for a key control characteristic if the correlation of the results is well-defined and known.

3.1.2
graphene
graphene layer
single-layer graphene
monolayer graphene
single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.

Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single-layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layer graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.1]

3.1.3
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate and functionalized variations thereof as well as graphene oxide and reduced graphene oxide

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.2 Terms regarding reliability

3.2.1
end of life
life cycle stage of a product starting when it is removed from its intended use stage

Note 1 to entry: In the context of IEC TS 62876-3-1, "end of life" is reached when a failure is observed.

3.2.2
failure
loss of ability to perform as required

Note 1 to entry: A failure of an item is an event that results in a *fault* (IEV 192-04-01) of that item.

Note 2 to entry: Qualifiers, such as catastrophic, critical, major, minor, marginal and insignificant, can be used to categorize failures according to the severity of consequences, the choice and definitions of severity criteria depending upon the field of application.

Note 3 to entry: Qualifiers, such as misuse, mishandling and weakness, can be used to categorize failures according to the cause of failure.

[SOURCE: IEC 60050-192:2015, 192-03-01]

3.2.3
failure criterion
pre-defined condition for acceptance as conclusive evidence of failure

Note 1 to entry: In the context of IEC TS 62876-3-1, the failure criterion is a pre-defined change of a key control characteristic (KCC) which is tested during reliability stress screening.

Note 2 to entry: If more than one KCC is observed during the test, end of life is reached if any of the KCCs reaches the failure criterion.

[SOURCE: IEC 60050-192:2015, 192-03-03, modified – The term KCC has been integrated into Note 1 to entry in the explanation of the failure criterion in this specific case. Note 2 to entry has been added.]

3.2.4
lot tolerance percent defective
LTPD
percent defective units that the sampling plan will reject 90 % of the time

Note 1 to entry: A single sampling plan for attributes consists of a sample of size n and an acceptance number c. The procedure operates as follows: select n items at random from the lot. If the number of defective items in the sample set is less than c, the lot is accepted. Otherwise, the lot is rejected.

3.2.5
mean time to failure
MTTF
expectation of the time to failure under stress

Note 1 to entry: In the case of non-repairable items with an exponential distribution of operating times to failure (i.e. a constant failure rate), the MTTF is numerically equal to the reciprocal of the failure rate. This is also true for repairable items if after restoration they can be considered to be "as-good-as-new".

Note 2 to entry: Operating time is the time accumulated from the first use, or from restoration, until failure.

Note 3 to entry: For the estimation of the MTTF from test results, the MTTF is calculated as the arithmetic mean value of the measured times to failure. If not all samples have failed, the behaviour of the remaining units might be extrapolated to the time when the sample under test falls below the failure criterion.

[SOURCE: IEC 60050-192:2015, 192-05-11, modified – In the definition, "under stress" has been added after the word "failure" and Note 3 to entry has been added.]

3.2.6
reliability
ability to perform as required, without failure, for a given time interval, under given conditions

Note 1 to entry: The time interval duration may be expressed in units appropriate to the item concerned, e.g. calendar time, operating cycles, distance run, etc., and the units should always be clearly stated.

Note 2 to entry: Given conditions include aspects that affect reliability, such as: mode of operation, stress levels, environmental conditions, and maintenance.

[SOURCE: IEC 60050-192:2015, 192-01-24, modified – Note 3 to entry has been omitted.]

3.2.7
reliability stress screening
RSS
process for detecting flaws by applying environmental and/or operational stresses to precipitate them as detectable failures.

Note 1 to entry: In the context of IEC TS 62876-3-1, the flaws are related to changes in the key control characteristics of the graphene layers for a given application.

[SOURCE: IEC 60050-192:2015, 192-09-19]

3.2.8
sample under test
SUT
manufactured product or clearly defined part of a product undergoing testing

3.2.9
time to failure
TTF
time accumulated from the first application of stress until failure

Note 1 to entry: The functionality of SUT is tested before and after the application of stress.

Note 2 to entry: SUT need not be functionally operating under stress test. Nevertheless, it is mentioned in the test report if the SUT was operating or not.

[SOURCE: IEC 60050-192:2015, 192-05-01, modified – In the term and definition, "operating time" has been replaced by "time". In the definition, "accumulated from the first use, or from restoration," has been replaced by "accumulated from the first application of stress" to render the definition more appropriate for the scope. The original Note 1 to entry has been replaced and Note 2 to entry has been added to take into account the specific application and scope.]

4 General

4.1 Sample under test

The sample under test (SUT) shall be clearly defined. In the content of this document it is always a substrate covered with a layer of a graphene material. Typical examples for substrates are the following:

– silica on silicon (SiO_2 on Si) – typical values of the thickness of the SiO_2 layer are 90 nm and 300 nm;
– SiC (0001);
– copper (Cu);
– polyethylene terephthalate (PET);
– quartz.

The graphene material layer may consist of, for example,

a) graphene,
b) few-layer graphene, or
c) graphene oxide,

which might be

1) grown on the substrate,
2) transferred from another substrate to the substrate, or
3) printed on the substrate as an ink.

The SUTs shall be selected randomly from a larger population of samples such that the SUTs are representative for the product.

Although it is anticipated that a homogeneous set of the SUTs be used for all tests in a reliability stress screening programme, it is recognized that this is not always possible or practical. When this is the case, the specific description of the samples used in each test shall be stated. Justification for the comparability of the samples shall be documented.

In addition, the level of manufacturing maturity of each SUT type shall be defined; for example, the product is in production with all documentation or the product is in development and was made with significant engineering support.

4.2 Sample description

The SUT shall be described in a way that all information regarding the preparation of the sample is disclosed, especially those preparations which reduce the severity of the test conditions with respect to the expected failure mechanisms. Cover layers or housings that are applied only for the test and are not part of the product design are not allowed as they distort the test results.

The thickness of the substrate, any kind of buffer layers, cover layers and residuals remaining from the manufacturing method of the layer can have a strong influence on the results of the reliability stress screening. Therefore, this information shall be provided in the test report, for example as a drawing showing the substrate layer sequence with dimensions and manufacturing method.

5 Reliability stress screening

5.1 General

The design of the tests in this document is based on assumptions on the failure mechanisms which are driven by the stress test level (severity). In order to qualify and certify the reliability of graphene layers on a substrate it is necessary to identify potential failure mechanisms and thus to underpin/confirm those assumptions using failure mode and effect analysis (FMEA). This can be done as a minimum by an analysis of failed samples during the development of the final product design. It can and should also be done proactively, based on general principles of physics and chemistry, established knowledge about material failure mechanisms for the material or device type under test, and/or experience with similar materials or devices. The test report shall contain the justification that the failure mechanisms and related failure modes are understood and relevant for the normal use of the final product.

5.2 End of life criteria

End of life criteria for the SUTs shall be defined based on changes of key control characteristics. The key control characteristics selected depend on the application. The sample reaches its end of life if any of the selected key control characteristics leaves its allowed range defined by its failure criterion.

EXAMPLE In case where the electrical resistivity is of relevance for the application, the KCC sheet resistance is assumed to be a mandatory key control characteristic. If the sheet resistance is not used, the reasons for that are given.

5.3 Acceptance criteria

The number of failures in a test shall be equal to or less than the number allowed by the agreed LTPD value according to Table B.2. For an initial product qualification as described in this document, an LTPD value of 20 % is sufficient. This translates to zero failed samples out of 11 tested. If this criterion is fulfilled, the specific combination of graphene and substrate can be called qualified according to this document.

NOTE Table B.2 gives an overview of the numbers of allowed failures via the number of samples tested depending on the chosen LTPD value.

5.4 Environmental stress tests

The minimum set of tests is defined in Annex A. Other tests may be added depending on the application. For each test there are two values given for the severity of the test.

– Required: These conditions are relevant for the pass/fail decision in the test.
– Optional, for information only: It is suggested to extend the test duration if the samples do not fail during the required part of the test in terms of testing time and temperature cycles to gather additional information. Failed samples provide a lot of important information regarding failure modes and the related failure mechanisms. The failures shall be analysed to improve the product. These results are not part of the test report.

5.5 Sequence

The tests listed in Annex A shall be run in parallel because the stress applied during the test can induce unnoticed damage on the sample even if the sample does not fail. This damage can lead to early failures in the subsequent test. Therefore, sequential tests are more difficult to interpret, and results may not be comparable. Nevertheless, if for any reason only a sequential test procedure is practical, it should be noted in the test report that the tests were sequential and that an unrepresentative fail due to the sequential test could have been the result. A good reason for sequential testing is the cost of testing. The cost of testing rises fast if the tests with many samples are done in parallel. For preliminary results, the uncertainty resulting from sequential testing can therefore be acceptable.

5.6 Severity

The severity and duration of the variables in the tests shall be clearly defined. For the purpose of this document, the values in Table A.1 shall apply. Annex B provides additional information on test conditions regarding the selection of temperature and relative humidity (Clause B.1) and sample numbers (Clause B.2) to achieve the targeted LTPD.

5.7 Measurement methods

The measurement method to evaluate changes of the key control characteristics shall be clearly defined for each reliability stress screening. Most of the measurements are defined in the IEC 62607 series. Where this is not possible other test methods may be defined. If a previously undefined test method is used, the test method and details used shall be technically justified and included in the RSS.

EXAMPLE If sheet conductance or sheet resistance is assumed to be a mandatory key control characteristic, one of the following methods may be used:

– IEC TS 62607-6-4, *Nanomanufacturing – Key control characteristics – Part 6-4: Graphene – Surface conductance measurement using resonant cavity*
– IEC TS 62607-6-9, *Nanomanufacturing – Key control characteristics – Part 6-9: Graphene-based material – Sheet resistance: Eddy current method*
– IEC TS 62607-6-10, *Nanomanufacturing – Key control characteristics – Part 6-10: Graphene-based material – Sheet resistance: Terahertz time-domain spectroscopy*

Measurement of sheet resistance by the four-point probe method will form the subject of a future Part 6-8.

The decision on which method is the best is made based on an analysis of the failure mechanisms including the results from the FMEA.

6 Measurements

6.1 General

Measurements of the key control characteristics shall be performed according to the procedures described in the related measurement standard.

6.2 Measurements during the test

The key control characteristics selected as pass/fail indicators shall be monitored during the test. Mandatory is a measurement before and after the test but continuous monitoring during the test is recommended, even for one or a small number of key control characteristics. If the samples need to be taken out of the climatic chamber from time to time for intermediate measurements, it shall be justified that this does not influence the test results. The test results shall be plotted versus testing time to establish the time to failure for each sample.

6.3 Identification of permanent changes

It is well known that key control characteristics can change temporarily during the test. The change of sheet resistance under humidity is such an example. Therefore, it is recommended to define a conditioning process which needs to be performed before each measurement. Clearly this requires the SUTs to be removed from the climatic chamber for each measurement. This procedure shall be reported in the test results.

7 Data analysis / Interpretation of results

7.1 Presentation of the results

The results of the measurement of the selected key control characteristics shall be plotted versus test duration for each of the SUTs. These plots are part of the test report. If the failure criterion is reached during the reliability stress screening, this point in time is called the TTF (time to failure). If the SUT remains intact, the measured KCC values may be extrapolated to estimate the future TTF. Figure 1 shows examples for both cases: TTF1 can directly be taken from the measurements because the SUT fails during the test. In case of TTF2, this is not possible as the SUT still functions after the test has been finished. Therefore, the measurement curve shall be extrapolated. The method of extrapolation shall be explained in the test report.

It is worth mentioning that the resistance of graphene films such as CVD grown and on SiC(0001) can many times be restored following vacuum annealing.

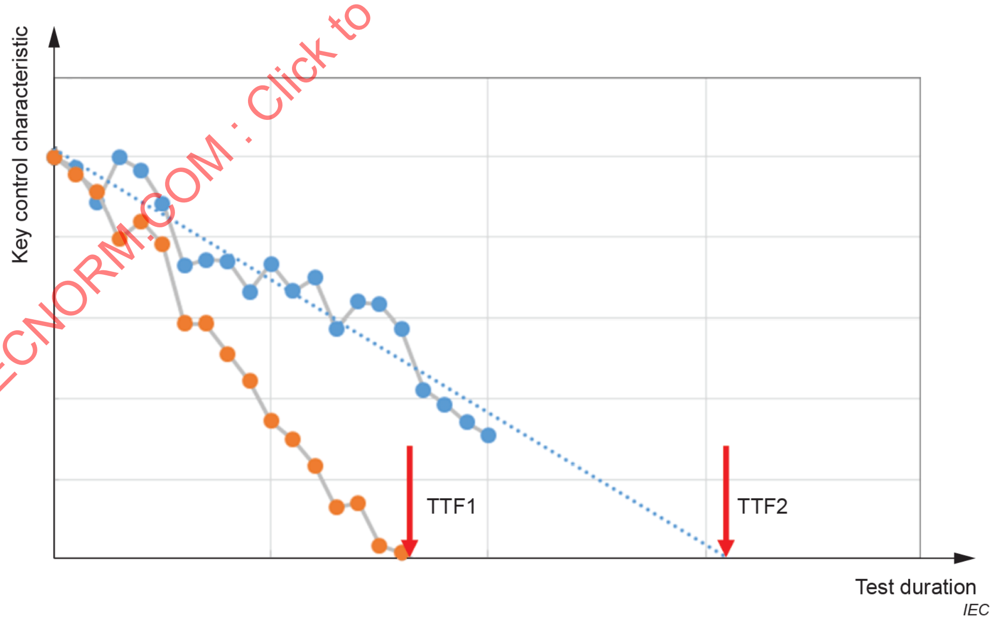

Figure 1 – Determination of the TTF by direct reading from the experimental data or extrapolation (arbitrary example)

The test duration is the actual time for which the SUT has undergone the reliability test screening.

After ordering the N screened SUTs according to their TTFs, the results of all test conditions shall be presented in a single table, see Table 1 for example, and for further evaluation. This table is part of the test report.

Table 1 – Times to failure (TTF) for devices in two life test conditions

Failure number	**Condition 45 °C / 85 % RH**	**Condition 85 °C / 85 % RH**
	TTF [h]	TTF [h]
1	1 700	850
2	1 900	1 100
3	Not yet apparent	1 300
…		
N − 1	Not yet apparent	1 950
N	Not yet apparent	Not yet apparent

7.2 Evaluation of pass/fail criteria

The product, i.e. the combination of substrate and graphene layer, has passed the test if the number of failed SUTs during the required testing time (Table A.1) is less than the number of LTPD listed in Table B.2 (e.g. LTPD 20 %: zero failures out of 11 SUTs or one failure out of 18 SUTs). Failures which occur after the standard RSS in the extended optional screening time will not be taken into account.

7.3 Calculation of the MTTF (mean time to failure)

For each test condition, the mean time to failure shall be calculated as the simple arithmetic mean value of the observed TTFs. Even if it is recommended to test until all SUTs fail, this might not be practical if the TTFs are very long. In those cases, a best guess extrapolation of the SUT behaviour to failure might be an adequate way to estimate the TTF for the surviving SUTs.

NOTE Extrapolation to the TTF might be difficult if the exact failure mechanism and failure mode are not known. In most cases a linear extrapolation is a good strategy. Strictly speaking the best way is to test until all SUTs have failed. Nevertheless there is always a trade-off between cost of testing and quality of the results. It is practical experience that a test duration which leads to 80 % of failed SUTs is sufficient.

8 Test report

The test report shall contain the following information:

– full description of the SUTs;
– sketch showing the structure of the sample, including the substrate, sequence of layers on top the substrate and their thickness;
– description of sample preparation;
– the sequence of tests for every group of SUTs;
– a table with headings for the test name, test duration and severity of the test, number of devices in the test, number of devices that failed during the test results;
– key control characteristics and test method to verify the pass/fail criteria;
– description of and justification for all measurements reported, including the plots of the KCC measurement results during the test;
– description of and justification for all passes/fails reported including the TTF table.

Annex A
(normative)

Environmental tests

Table A.1 – Tests and their severities

Test name	Requirements	LTPD [%]	Quantity	# fail	Reference
Cold – low temperature storage	Required: Temperature: −40 °C, uncontrolled humidity Duration: 500 h	20	11	0	IEC 60068-2-1
	Optional, for information only: Extension of the duration up to 2 000 h or to failure				
Dry heat – high temperature storage	Required: Temperature 85 °C at < 40 %RH Duration: 500 h	20	11	0	IEC 60068-2-14
	Optional, for information only: Extension of the duration up to 2 000 h or to failure				
Change of temperature	Required: Temperature cycle −40 °C to +85 °C, dwell time at extremes 1 h, ramp 1 °C/min, uncontrolled humidity Number of cycles: 500	20	11	0	IEC 60068-2-2
	Optional, for information only: Extension up to 2 000 cycles or to failure				
Damp heat, steady state	Required: Temperature/humidity 85 °C/85 % RH Duration: 500 h	20	11	0	IEC 60068-2-78
	Optional, for information only: Extension to 2 000 h or to failure				

Annex B
(normative)

Additional information regarding test conditions and procedures

B.1 Test matrix for demonstrating acceleration factors

It is widely assumed that nanomaterials and nano-subassemblies can have degradation mechanisms driven by temperature and humidity. Even if the details are not clear at this point in time, it seems to be practical to make simple assumptions like Arrhenius type acceleration factors or other models. It is not the intention of this document to give guidelines regarding degradation mechanisms, but it seems appropriate to suggest recommended values for temperature and humidity to reduce the manifold possible selections. This will help to compare results taken in different research laboratories and the comparison of material modifications as well as the understanding of failure mechanisms.

Table B.1 – Test matrix for temperature and humidity relation

Absolute humidity [g/m^3]	**Relative humidity [%]**									
	Temperature [°C]									
	25	**40**	**45**	**55**	**65**	**75**	**85**	**95**	**110**	**125**
19,5	85	38	30	19	12	8	6	4	2	2
43,5	sat.	85	67	42	27	18	12	9	5	3
55,5	-	sat.	85*	53	34	23	16*	11	7	4
88,0	-	-	sat.	85	55	37	25	18	11	7
136,0	-	-	-	sat.	85	57	39	27	17	11
203,0	-	-	-	-	sat.	85	58	41	25	16
296,0	-	-	-	-	-	sat.	85*	60	37	23
421,0	-	-	-	-	-	-	sat.	85	52	33
585,0	-	-	-	-	-	-	-	sat.	85	46
1 070,0	-	-	-	-	-	-	-	-	sat.	85
NOTE sat. means saturated.										

In order to define priorities regarding test conditions, the following test conditions should be used to validate alternative acceleration models (identified by an asterisk (*) in Table B.1):

– damp heat, steady state: 85 °C/85 % RH

– damp heat, steady state: 45 °C/85 % RH

– dry heat: 85 °C (which is basically identical to damp heat, steady state): 85 °C/16 % RH

The minimum sample size shall be determined with respect to the LTPD = 20 % sampling plan.

B.2 Procedure to select test samples

Test samples shall be selected from fabrication statistically independently. Table B.2 provides the number of test samples required to achieve the required LTPD value. For the purpose of this document, an LTPD value of 20 % seems to be sufficient. Nevertheless the LTPD value shall be specified in the test report together with the number of tested and failed samples.

NOTE An LTPD value of 20 % allows zero failures if 11 samples are tested or one failure if 18 samples are tested and so on.