

INTERNATIONAL STANDARD

Semiconductor devices –
Part 5-13: Optoelectronic devices – Hydrogen sulphide corrosion test for
LED packages

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INTERNATIONAL STANDARD

**Semiconductor devices –
Part 5-13: Optoelectronic devices – Hydrogen sulphide corrosion test for
LED packages**

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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SEMICONDUCTOR DEVICES –

Part 5-13: Optoelectronic devices –
Hydrogen sulphide corrosion test for LED packages

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The text of this International Standard is based on the following documents:

| | |
|--------------|------------------|
| FDIS | Report on voting |
| 47E/746/FDIS | 47E/751/RVD |

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 International Standard 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.

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- reconfirmed,
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- amended.

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INTRODUCTION

This part of IEC 60747 provides the accelerated test method to assess effects of the tarnishing of silver and silver alloys used for LED packages due to hydrogen sulphide, because sulphide gas (H_2S) tarnishes silver used in LED packages and causes lumen degradation.

There are some existing environmental stress test standards, but they intend to test contacts and connections, not LED lumen degradation. IEC 60068-2-43 provides useful information to assess effects to the contact resistance for contacts and connections due to corrosion of silver and silver alloy. Because the criterion performance in IEC 60068-2-43 is contact resistance, it is not applicable to LED packages to determine effects to the luminous/radiant flux maintenance.

For LEDs, light output should be measured, but there is no such provision in existing standards. Therefore, this document has been drawn up.

This document provides the accelerated test method with mixture gas (H_2S & NO_2) test which has the following merits:

- the test method in this document can reproduce the real failure mode;
- the test method in this document works to reproduce the in-situ linear kinetics;
- the test method in this document can reduce the testing duration.

In all tests, the major criterion of performance will be the change in the luminous/radiant flux and/or electric characteristics (e.g. forward voltage and forward current) caused by sulphide corrosion.

This test may not be suitable as a general corrosion test, i.e. it may not predict the behaviour of flux and/or electric characteristics and connections in industrial atmospheres.

This document also contains an informative Annex A that gives information to predict luminous/radiant flux degradation due to the silver and silver alloy tarnishing in particular conditions from test results.

SEMICONDUCTOR DEVICES –

Part 5-13: Optoelectronic devices – Hydrogen sulphide corrosion test for LED packages

1 Scope

This part of IEC 60747 provides the accelerated test method to assess effects of the tarnishing of silver and silver alloys used for LED packages due to hydrogen sulphide. Particularly, this test method is intended to give information on silver and silver alloy tarnishing effects to the luminous/radiant flux maintenance of LED packages. Additionally, this test method can give information on electric performances of LED packages due to corrosion of silver and silver alloys.

The object of this test is to determine the influence of atmospheres containing hydrogen sulphide on parts of LED packages made of:

- silver or silver alloy;
- silver or silver alloy protected with another layer;
- other metals covered with silver or silver alloy.

Testing other degradations that are susceptible to affect luminous/radiant flux maintenance and/or electric performance (e.g. degradation of copper or silicone parts) is not the object of this test.

This document is applicable to LED packages for lighting applications only if referenced by an IEC SC 34A document.

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 60068-1, *Environmental testing – Part 1: General and guidance*

IEC 60068-2-60:2015, *Environmental testing – Part 2-60: Tests – Test Ke: Flowing mixed gas corrosion test*

IEC 60747-5-6, *Semiconductor devices – Part 5-6: Optoelectronic devices – Light emitting diodes*

CIE 127, *Measurement of LEDs*

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 luminous flux

 Φ_v

quantity derived from radiant flux Φ_e by evaluating the radiation according to its action upon the CIE standard photometric observer

[SOURCE: IEC 60050-845:2020, 845-21-039, modified – The explanation for photopic vision has been removed as well as the notes.]

3.2 radiant flux

 Φ_e

power emitted, transmitted or received in the form of radiation

[SOURCE: IEC 60050-845:2020, 845-21-038, modified – The definition has been reviewed and the notes have been removed.]

4 Test apparatus

4.1 General

The test apparatus consists of a climatic system, test enclosure, gas delivery system and means for measuring gas concentration, detailed in IEC 60068-2-60:2015, Annex B.

Details of design and construction are optional but shall be such that the conditions specified for the method are fulfilled throughout the working volume and shall comply with the following requirements:

- water droplets or aerosols shall not be injected into the test enclosure;
- air and water used shall be sufficiently clean in order not to affect performance of the test;
- the test atmosphere shall flow through the enclosure in such a manner as to ensure uniform test conditions within the working volume;
- the sampling point for gas analyses shall be in the working volume of the test enclosure;
- the exhaust gases shall be treated in accordance with the relevant regulatory stipulations;
- the wet bulb pod shall be placed in the test chamber in such a manner not to exceed 0,1 % of the cross-section of the test chamber.

4.2 Test jig

If jigs are used to set specimens under test, the jigs shall be made of corrosion-free materials (e.g. UPVC tube, PTFE, glass, etc.).

The jigs shall also allow air to pass through easily enough so that the wind speed in the test enclosure is not influenced significantly.

4.3 Test setup

If multiple specimens are tested, each specimen shall be set in the same posture. See Figure 1 as an example.

The distance between specimens should be 10 mm or more.

Corrosive materials or objects including corrosive materials (e.g. silver) other than the test specimens shall not be set in the test enclosure.

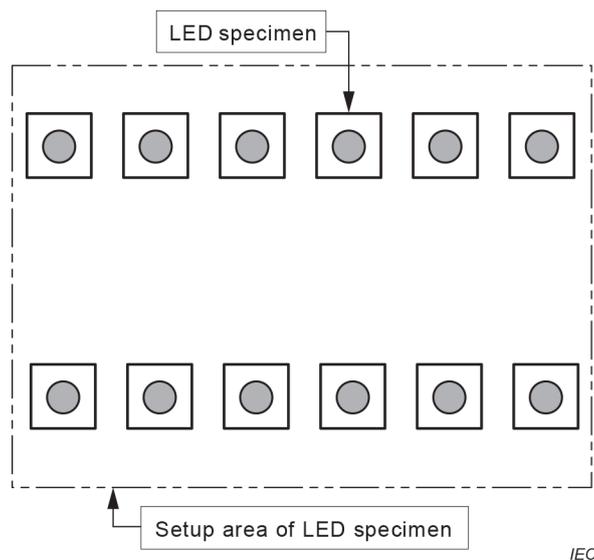


Figure 1 – Example of setup

5 Test atmosphere

The composition of the atmosphere within the test chamber shall satisfy the following conditions:

- hydrogen sulphide: 2×10^{-6} vol/vol;
- nitrogen dioxide: 4×10^{-6} vol/vol;
- temperature: $40 \text{ °C} \pm 2 \text{ °C}$;
- relative humidity: $75 \% \pm 5 \%$
- rate of ventilations: 3 cycles/hour to 10 cycles/hour

The test atmosphere may be obtained by mixing hydrogen sulphide and nitrogen dioxide (from any convenient source) with air and water vapour in a way to ensure a homogeneous mixture. (More than one stage may be necessary to obtain a homogeneous distribution of the small quantity of hydrogen sulphide.)

6 Preconditioning

6.1 General

The specimens under test shall not be cleaned in any way unless required by the manufacturer's instructions, the relevant detail specification, or agreed upon between the interested parties.

6.2 Hygroscopic treatment

Before the test, test specimens shall absorb moisture under the condition below.

- Temperature: $85 \text{ °C} \pm 2 \text{ °C}$
- Relative humidity: $85 \% \pm 5 \%$
- Duration: more than 24 h and more than a period for the test specimens to reach a moisture saturation condition.

The specimens shall be set in the test closure and the test shall be started within one hour after the hygroscopic treatment.

The period for the test specimens to reach a moisture saturation condition may be determined by a test in advance or technical documents.

Initial measurement is done before this treatment.

There is the concern of the different results of sulphide corrosion test caused by the difference amounts of moisture absorption in LEDs in case that LEDs are stored in different conditions (see Figure 2 as an example).

Therefore, LED test specimens shall be saturated with moisture absorption before hydrogen sulphide corrosion test to reduce the difference of test results caused by the difference amounts of moisture absorption in LEDs.

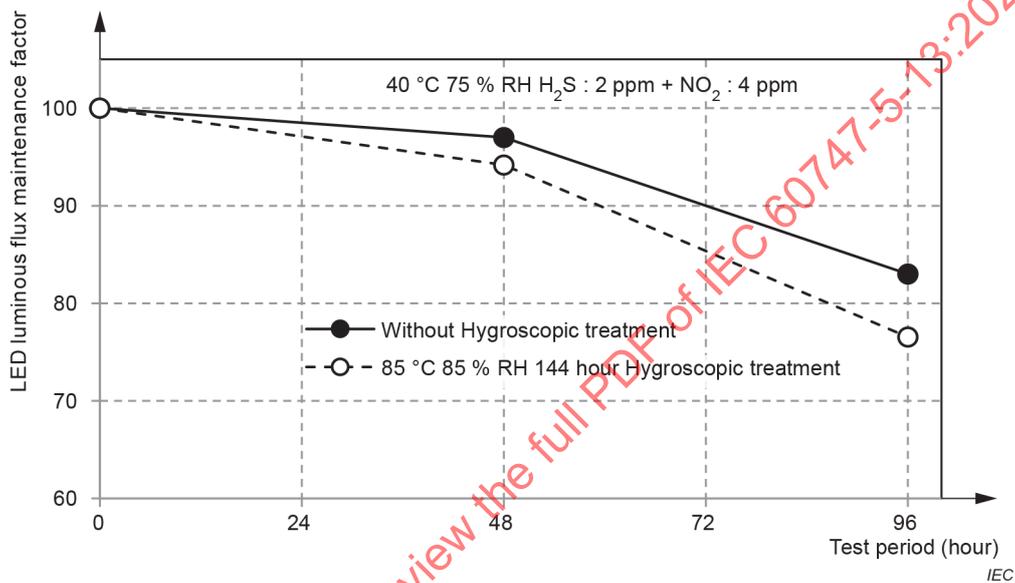


Figure 2 – Example of LED luminous flux maintenance factor before & after hygroscopic treatment

7 Method

7.1 Initial measurements

The relevant specification may require that, before and after test, a luminous/radiant flux measurement be carried out in combination with an electric measurement.

Unless otherwise specified in the relevant specification, the luminous/radiant flux measurement and the electric measurements shall be conducted in accordance with IEC 60747-5-6 or CIE 127.

7.2 Procedure

Prior to the commencement of the test, it shall be established by suitable measurements that stable conditions for the concentration of hydrogen sulphide and nitrogen dioxide, temperature, and relative humidity have been achieved. Periodic checks for temperature and relative humidity shall be made during the test to ensure that these conditions are maintained.

Care shall be taken that the specimens are placed in a way so that they do not come in contact with each other, that they do not cover or shield each other from the test atmosphere, and that they do not shield air flow in the test chamber significantly.

For the total volume and the total surface area of the test specimens, the provisions in 7.2 of IEC 60068-2-60:2015 shall be applied.

Adequate precautions shall be taken to ensure that the luminous/radiant flux and electric performances are not disturbed during the exposure period.

In the exposure period, the number of specimens in the test chamber should not be changed.

The specimens shall be exposed without any electrical load or as required by the detail specification.

The specimens shall be continuously exposed to the test atmosphere for a duration as required by the detail specification and the following steps:

- The gas concentrations set up of test atmosphere is recommended in Annex D;
- Set the test specimens in the working volume of the test chamber, after the specified temperature is stabilized, start the flow of humid air, allow to stabilize and adjust temperature and humidity not to accumulate the condensation on the inner wall of the test chamber and the surface of the test specimen;
- Start the flow of the gases into the humid air stream and allow to be stabilized. Exposure period shall be counted from the time the gas is started to flow;
- Measure and adjust, if necessary, temperature, humidity and gas concentrations. During these adjustments, any overshooting of gas concentration shall be avoided. Maximum allowed duration of this period of stabilization and adjustments, to prescribed values, is 24 h;
- During the course of testing, temperature, humidity and gas concentrations shall be kept within the prescribed limits;
- At the end of the test period, remove the specimens after the humidity in the chamber is decreased around room temperature and RH of 75 % to avoid condensation.

7.3 Final measurements

The specimens shall be removed from the chamber and stored under standard recovery conditions specified in IEC 60068-1 for not less than 1 h before luminous/radiant flux and electric measurements are made. The measurements should be done in 2 h after the removal from the chamber if the measurements cannot be done within the specified time, they shall be performed within up to 24 hours after the removal from the chamber. In that case, the specimens shall be kept under standard recovery condition specified in IEC 60068-1, and such situation shall be mentioned in the test report with the period of storage.

Adequate precautions shall be taken to ensure that the luminous/radiant flux and electric performances are not disturbed. The method used for measuring the luminous/radiant flux and the electric performances should be the same as used for the initial measurement.

Treatment of the specimens before final measurements and the details of measurements shall be made as required by the relevant specification.

Visual inspection of the specimens may be required by the detail specification.

8 Details to be specified

The relevant specification shall specify the following details:

- a) measurements and checks to be made prior to the test;
- b) electrical loading or operational conditions of the specimens, if applicable;
- c) duration of the test;
- d) measurements, correction, checks and visual inspection to be made at the end of the test.

NOTE Annex A provides information to predict the luminous flux degradation in particular indoor environments from the results of this hydrogen sulphide corrosion test.

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Annex A (informative)

Information to predict luminous/radiant flux degradation in particular conditions from the test results

A.1 Correspondence relation between hydrogen sulphide corrosion test and indoor corrosivity categories

The result of hydrogen sulphide corrosion test for 96 h corresponds to the luminous flux degradation in the indoor environment of corrosivity category IC 3 in ISO 11844-1:2006¹ for approximately ten years usage as shown in Table A.1.

Therefore, it is possible to predict the luminous flux degradation in particular indoor environments from the results of this hydrogen sulphide corrosion test.

**Table A.1 – Description of typical environments related to the estimation
of indoor corrosivity categories in ISO 11844-1:2006, Table D.3²**

| Corrosivity category (IC) | Corrosivity | Typical environments |
|---------------------------|------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| IC 1 | very low indoor | <p><u>Heated spaces</u> with controlled stable relative humidity (< 40 %) without risk of condensation, low levels of pollutants, no specific pollutants, e.g. computer rooms, museums with controlled environment</p> <p><u>Unheated spaces</u> with dehumidification, low levels of indoor pollution, no specific pollutants e.g. military stores for equipment</p> |
| IC 2 | low indoor | <p><u>Heated spaces</u> with low relative humidity (< 50 %) with certain fluctuation of relative humidity without risk of condensation, low levels of pollution, without specific pollutants e.g. museums, control rooms</p> <p><u>Unheated spaces</u> with only temperature and humidity changes, with no risk of condensation, low levels of pollution without specific pollutants, e.g. storage rooms with low frequency of temperature changes</p> |
| IC 3 | medium indoor | <p><u>Heated spaces</u> with risk of fluctuation of temperature and humidity, medium levels of pollution, certain risks for specific pollutants, e.g. switchboards in the power industry</p> <p><u>Unheated spaces</u> with elevated relative humidity (> 50 % – 70 %) with periodic fluctuation of relative humidity, without risk of condensation, elevated levels of pollution, low risk of specific pollutants, e.g. churches in non-polluted areas, outdoor telecommunication boxes in rural areas</p> |
| IC 4 | high indoor | <p><u>Heated spaces</u> with fluctuation of humidity and temperature, elevated levels of pollution including specific pollutants, e.g. electrical service rooms in industrial plants</p> <p><u>Unheated spaces</u> with high relative humidity (> 70 %) with some risk of condensation, medium levels of pollution, possible effect of specific pollutants, e.g. churches in polluted areas, outdoor boxes for telecommunication in polluted areas</p> |
| IC 5 | very high indoor | <p><u>Heated spaces</u> with limited influence of relative humidity, higher levels of pollution including specific pollutants like H₂S, e.g. electrical service rooms, cross-connection rooms in industries without efficient pollution control</p> <p><u>Unheated spaces</u> with high relative humidity and risk for condensation, medium and higher levels of pollution, e.g. storage rooms in basements in polluted areas</p> |

NOTE The general characterization of indoor atmospheres with respect to corrosion of metals is summarized in ISO 11844-1:2006, Annex C.

¹ A new edition of ISO 11844-1 was published in 2020.

² Reproduced (from ISO 11844-1:2006), with the permission of ISO.

A.2 Correspondence relation between the result of this corrosion test and the corrosion in the field environment (Case example)

Hydrogen sulphide corrosion test for 96 h gets silver test pieces mass increase by about 4 500 mg/m² (see the test results by test apparatus in Figure A.1). Method of confirmation of the mass increase of silver test pieces is detailed in Annex B.

This mass increase corresponds to the ten times the upper limit of mass increase to silver test pieces in the indoor environment of corrosivity category IC 3 in ISO 11844-1:2006 for approximately one year's usage (see Table A.1 and Table A.2).

The result of hydrogen sulphide corrosion test for 96 h corresponds to the corrosion in the indoor environment of corrosivity category IC 3 in ISO 11844-1:2006 for approximately ten years' usage, because the mass increase to silver test pieces in the field environment linearly changes.

Table A.2 – The upper limit of mass increase of silver test pieces in the indoor environment of corrosivity category in ISO 11844-1:2006 for approximately ten years' usage

| Class | The upper limit of mass increase of silver test pieces mg/m ² for approximately 1 year | The upper limit of mass increase of silver test pieces mg/m ² for approximately 10 years |
|-------|------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|
| IC 1 | 25 | 250 |
| IC 2 | 100 | 1 000 |
| IC 3 | 450 | 4 500 |
| IC 4 | 1 000 | 10 000 |
| IC 6 | 2 500 | 25 000 |

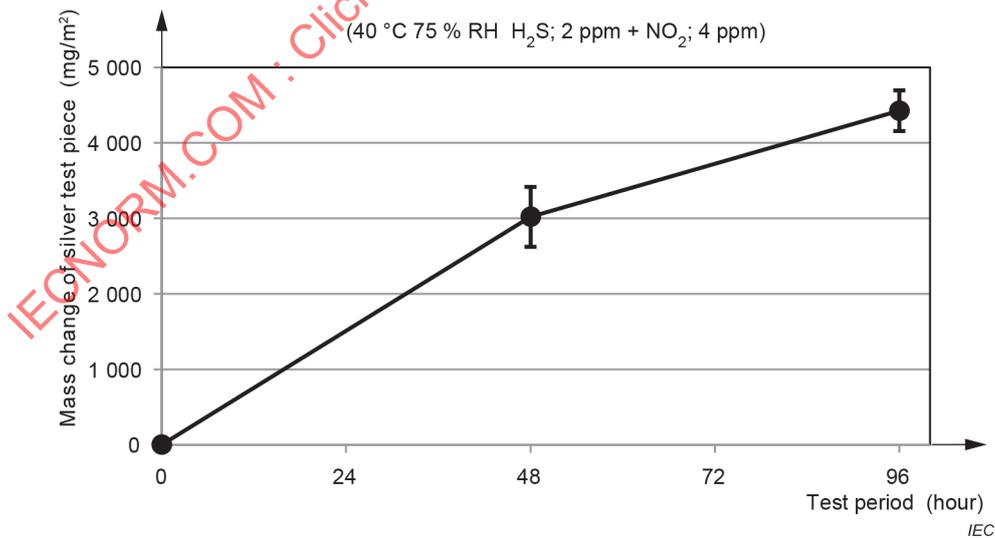


Figure A.1 – Mass increase example of silver test piece hydrogen sulphide corrosion test

Annex B (informative)

Method for determining the mass increase of silver test pieces

B.1 Purpose

The purpose here is to explain how to determine the increase amount of silver test pieces.

The specification of the silver test pieces is described in Annex C.

B.2 Method

The mass increase of silver test pieces d_m should be obtained by the hydrogen sulphide corrosion test with only silver test pieces done by the same test apparatus, test jig, test atmosphere and test method described in this document.

Mass increase of silver test piece d_m (mg/m²) is calculated by the following formula:

$$d_m = (ms1 - ms2) / sa$$

where

$ms1$ (mg) is the mass of silver test piece after the test;

$ms2$ (mg) is the mass of silver test piece before the test;

sa (m²) is the surface area of silver test piece.

No material to be eroded or something to contain it shall be allowed in test chamber.

In particular, this test inside the same chamber for hydrogen sulphide corrosion test for LED should be avoided not to have the influence on the result of hydrogen sulphide corrosion test for LED.

B.3 Silver test pieces

Five silver test pieces prepared based on Annex C should be exposed.

The increase amount measured by appropriate ability after the test shall be used to monitor the test repeatability and repetitiveness, one of criteria for corrosivity.

Apply 7.2 of IEC 60068-2-60:2015 to total cubic volume and surface area of test pieces.

B.4 How to place silver test pieces

One silver test piece should be placed at the centre of the area to place test pieces and other 4 pieces should be placed at the locations to surround the area.

They should be hanged, holding one end of longer direction on test pieces.

It is advisable that the distance between test pieces is longer than 10 mm not to disturb homogeneous air flow.

Example of layout of silver test pieces is shown in Figure B.1.

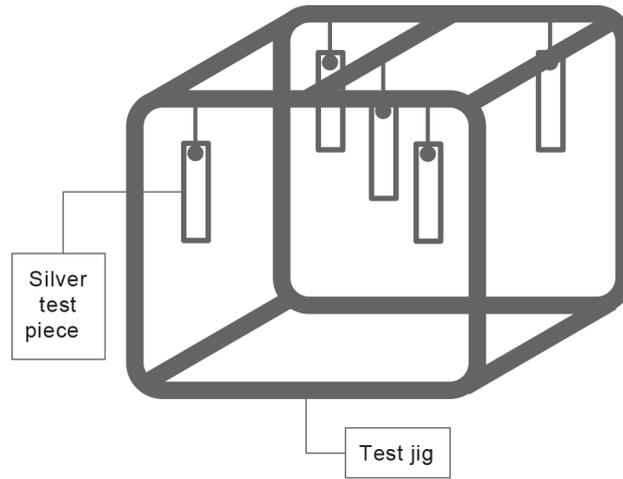


Figure B.1 – Example of layout of silver test pieces

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Annex C (informative)

Silver test piece for corrosion monitoring

C.1 Specimens

Silver specimens should be pure pieces from silver purity not lower than 99,98 %.

It is preferable to use rectangular specimens in the form of flat sheets, as they can be readily weighed by the analytical balance (readability down to 0,1 mg). A convenient specimen size is 10 mm × 50 mm.

Specimens may be larger provided that they can be accurately weighed. The specimen thickness may preferably be 0,5 mm.

C.2 Preparation

Silver specimens should, before weighing, be prepared as follows:

- a) A hole with diameter not greater than 4 mm is cut at the upper side of the specimen;
Abrading³ with deionised water and silicon carbide paper to P 1 000 specified in ISO 6344-3.
- b) Cleaning in deionised water in an ultrasonic bath for 3 min to 5 min;
- c) Degreasing in ethanol in an ultrasonic bath for 3 min to 5 min;
- d) Drying 60 °C for 5 min;
- e) Store in small plastic bags with open top. The small plastic bags are placed in a desiccator or sealed plastic bag with desiccant that have no corrosiveness before and after the weighing and the exposure.

After final surface cleaning before exposure, it is important that limited handling occurs. The specimens are only handled with a clean pair of tweezers. After the preparation, the specimens are allowed to be kept for maximum of 120 h in the desiccator or sealed plastic bag with desiccants that have no corrosiveness.

To avoid marking on the specimens, the identity of the specimens may preferably be marked on the small plastic bags.

³ To avoid the risk of contamination, the abrading papers for polishing specimens of different materials are not recommended for this use.