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Soft soldering fluxes — Test methods —

Part 5:
Copper mirror test

Flux de brasage tendre — Méthodes d'essai —

Partie 5: Essai au miroir de cuivre

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering materials*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 121, *Welding*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 9455-5:2014), of which it constitutes a minor revision.

The main changes compared to the previous edition are as follows:

— Footnote 1 in old 5.2 (now 6.2) has been deleted;

— Clause 3, Terms and definitions, has been added;

— subsequent numbering and cross-references have been updated.

A list of all parts in the ISO 9455 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Soft soldering fluxes — Test methods —

Part 5:
Copper mirror test

1 Scope

This document specifies a qualitative method for assessing the aggressiveness of a flux towards copper.

The test is applicable to all fluxes of type 1 as defined in ISO 9454-1.

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.

ISO 9455-1, *Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method*

ISO 9455-2, *Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

For flux samples in the form of a solid or paste, and for flux-cored solder, a flux test solution containing 25 % (*m*/*m*) of solids is prepared. For liquid flux samples, the liquid is used full strength as the flux test solution. The flux test solution is then evaluated in terms of its attack on a copper film previously vacuum deposited onto a glass plate (copper mirror). A rosin reference solution, which should not cause removal of the copper film, is used as a control. The object of the test is to determine the flux reactivity due to the presence of free halide activators.

NOTE The presence of amines in the flux can cause misleading results in that the flux appears to pass the test, when in fact it has a highly reactive composition.

5 Reagents

Use only reagents of recognized analytical grade and only distilled, or deionized, water.

5.1 Acetone.

5.2 Propan-2-ol.

5.3 Degreasing agent, such as a suitable neutral organic solvent such as acetone or petroleum ether.

5.4 Rosin reference solution, 25 % (*m*/*m*), prepared by dissolving 25 g of W-W grade colophony in 75 g of propan-2-ol (see 5.2).

5.5 Ethylenediaminetetraacetic acid (EDTA), 0,1 % (*m*/*m*) solution.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Temperature/humidity oven, capable of maintaining a temperature of 25 °C and a relative humidity of (50 ± 5) %.

6.2 Copper mirrors. Thoroughly clean a number of glass test plates, approximately 25 mm × 50 mm or bigger (e.g. 50 mm × 75 mm) in size, degrease them, if necessary, using the degreasing agent (see 5.3) and dry them.

Deposit copper, by vacuum deposition, onto one surface of the dry test plates to a thickness of approximately 50 nm. The transmittance of the plate to normal incident monochromatic light at a wavelength of 500 nm shall be between 5 % and 15 %.

Copper mirror test plates complying with these requirements are available commercially and can be used instead.

Store the copper mirrors under dry nitrogen until use.

6.3 Soxhlet extraction apparatus.

7 Procedure

7.1 Preparation of the flux test solution

7.1.1 Liquid flux samples

Use liquid flux samples at full strength as the flux test solution.

7.1.2 Solid flux samples

7.1.2.1 Prepare by dissolution with propan-2-ol (see 5.2, but see also 7.1.2.2), a flux test solution containing 25 % by mass of the solid flux sample.

7.1.2.2 If it is found that the flux is not soluble in propan-2-ol, then use another suitable water miscible solvent and give details of this solvent in the test report [see Clause 9, item f)].

7.1.3 Flux-cored solder

7.1.3.1 Cut a length of the flux-cored solder with a mass of approximately 150 g and seal the ends by crimping. Wipe the surface clean with a cloth moistened with acetone (see 5.1). Place the sample in a beaker, add sufficient water to cover the sample, and boil for 5 min to 6 min. Remove the sample, rinse it with acetone (see 5.1), and allow to dry.

While protecting the solder surface from contamination, cut the sample into short lengths (maximum 19 mm) using a razor blade so as not to crimp the cut ends. Place the cut segments into the extraction

tube of a clean Soxhlet extraction apparatus (see 6.3) and extract the flux with propan-2-ol (see 5.2, but see also 7.1.2.2) until the return condensate is clear.

7.1.3.2 Determine the non-volatile matter content of the extract using the method in ISO 9455-1 or ISO 9455-2, and adjust the content by evaporation or by dilution with propan-2-ol (see 5.2, but see also 7.1.2.2) to 25 % by mass to produce the flux test solution.

7.2 Preparation of copper mirrors for test

Select two copper mirrors (see 6.2) free from visible defects. Immediately before carrying out the test in 7.3, immerse the copper mirrors in the EDTA (see 5.5) for not more than 5 s, to remove any copper oxide. Rinse immediately in running water, then in acetone (see 5.1) and dry using warm air.

7.3 Determination

Place the two freshly cleaned copper mirrors (see 7.2) onto a clean horizontal surface, mirror side up. Place one drop (maximum 0,05 ml) of the flux test solution (see 7.1) on one of the mirrors and, at a distance of approximately 35 mm, one drop (maximum 0,05 ml) of the rosin reference solution (see 5.4). During this operation, do not allow the dropper to touch the copper mirror.

Repeat the procedure with the second copper mirror.

Place the two mirrors in a horizontal position in the temperature/humidity oven (see 6.1) and condition the mirrors at 25 °C ± 2 °C and (50 ± 5) % relative humidity for 24 h. Remove the mirrors from the oven and wash off the flux residues using propan-2-ol (see 5.2), or using the solvent used in 7.1. Dry the mirrors using a stream of warm air.

Examine the copper mirrors against a white background.

NOTE The presence of free halide activators in the flux test solution results in partial or complete removal of the copper film at the location of the drop, the copper mirror becoming progressively more transparent as the flux reactivity increases. The presence of amines in the flux can cause misleading results.

8 Assessment and expression of results

The flux shall have passed the test if there is no removal of the film on either copper mirror by the flux test solution. Removal is defined as the complete penetration of the copper film over the whole or part of the area, which causes the white background to be visible.

However, if the rosin reference solution (see 5.4) has failed the test, then repeat the determination using freshly prepared copper mirrors (see 7.2).

9 Test report

The test report shall include the following information:

a) the identification of the test sample;

b) a reference to this document (i.e. ISO 9455-5);

c) the results obtained;

d) any unusual features noted during the determination;

e) details of any operation not included in document, or regarded as optional;

f) details of the solvent used in the preparation of the flux test solution (see 7.1) if not propan-2-ol.

Bibliography

[1] ISO 9454-1, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging*