INTERNATIONAL STANDARD

ISO 9455-5

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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

Reference number
ISO 9455-5:1992(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9455-5 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Sub-Committee SC 12, *Soldering and brazing materials*.

ISO 9455 consists of the following parts, under the general title *Soft soldering fluxes — Test methods*:

— *Part 1: Determination of non-volatile matter, gravimetric method*

— *Part 2: Determination of non-volatile matter, ebulliometric method*

— *Part 3: Determination of acid value, potentiometric and visual titration methods*

— *Part 5: Copper mirror test*

— *Part 6: Determination of halide content*

— *Part 8: Determination of zinc content*

— *Part 9: Determination of ammonia content*

— *Part 10: Flux efficacy tests, solder spread method*

— *Part 11: Solubility of flux residues*

— *Part 12: Steel tube corrosion test*

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— *Part 13: Determination of flux spattering*

— *Part 14: Assessment of tackiness of flux residues*

— *Part 15: Copper corrosion test*

— *Part 16: Flux efficacy tests, wetting balance method*

— *Part 17: Determination of surface insulation resistance of flux residues (Comb test)*

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Soft soldering fluxes — Test methods —

Part 5:
Copper mirror test

1 Scope

This part of ISO 9455 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 standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9455. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9455 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9454-1:1990, *Soft soldering fluxes — Classification and requirements — Part 1: Classification, labelling and packaging*.

ISO 9455-1:1990, *Soft soldering fluxes — Test methods — Part 1: Determination of non-volatile matter, gravimetric method*.

ISO 9455-2:—[1], *Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method*.

3 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 1 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.

4 Reagents

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

4.1 Acetone.

4.2 Propan-2-ol.

4.3 Degreasing agent, such as a permitted chlorinated hydrocarbon, or an aqueous, non-ionic detergent.

4.4 Rosin reference solution, 25 % (*m*/*m*), prepared by dissolving 25 g of W-W grade colophony in 75 g of propan-2-ol (4.2).

4.5 Ethylenediaminetetraacetic acid (EDTA), 5 % (*m*/*m*) solution.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

1) To be published.

5.1 Temperature/humidity oven, capable of maintaining a temperature of 25 °C ± 2 °C and a relative humidity of (50 ± 5) %.

5.2 Copper mirrors.

Thoroughly clean a number of glass test plates, approximately 25 mm × 50 mm in size, degrease them using the degreasing agent (4.3) and dry them. Deposit copper, by vacuum deposition, onto one surface of the dry test plates to a thickness of 26 nm ± 5 nm, which corresponds to a mass deposition of about $2{,}3 \times 10^{-5}$ g copper per square centimetre of glass. 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[2] and may be used instead.

Store the copper mirrors under dry nitrogen until use.

5.3 Soxhlet extraction apparatus.

6 Procedure

6.1 Preparation of the flux test solution

6.1.1 Liquid flux samples

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

6.1.2 Solid flux samples

6.1.2.1 Prepare by dissolution with propan-2-ol (4.2) (but see 6.1.2.2), a flux test solution containing 25 % by mass of the solid flux sample.

6.1.2.2 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 8, item f)].

6.1.3 Flux-cored solder

6.1.3.1 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 (4.1). 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 (4.1) and allow to dry.

Whilst protecting the solder surface from contamination, cut the sample into short lengths (maximum 10 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 (5.3) and extract the flux with propan-2-ol (4.2) (but see 6.1.2.2) until the return condensate is clear.

6.1.3.2 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 (4.2) (but see 6.1.2.2) to 25 % by mass to produce the flux test solution.

6.2 Preparation of copper mirrors for test

Select two copper mirrors (5.2) free from visible defects. Immediately before carrying out the test in 6.3, immerse the copper mirrors in the EDTA solution (4.5) for not more than 5 s, to remove any copper oxide. Rinse immediately in running water, then in acetone (4.1) and dry using warm air.

6.3 Determination

Place the two freshly cleaned copper mirrors (6.2) onto a clean horizontal surface, mirror side up. Place one drop (maximum 0,05 ml) of the flux test solution (6.1) on one of the mirrors and, at a distance of approximately 35 mm, one drop (maximum 0,05 ml) of the rosin reference solution (4.4). 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 (5.1) 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 (4.2), or using the solvent used in 6.1. Dry the mirrors using a stream of warm air.

Examine the copper mirrors against a white background.

NOTE 2 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.

2) Copper mirror test plates suitable for this test method may be obtained from Evaporated Metal Films Corporation, Ithaca, New York, USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named.

7 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 (4.4) has failed the test, then repeat the determination using freshly prepared copper mirrors (6.2).

8 Test report

The test report shall include the following information:

a) the identification of the test sample;

b) the test method used (i.e. reference to this part of ISO 9455);

c) the results obtained;

d) any unusual features noted during the determination;

e) details of any operation not included in this part of ISO 9455, or regarded as optional;

f) details of the solvent used in the preparation of the flux test solution (6.1) if not propan-2-ol.

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