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Soft soldering fluxes — Test methods —

Part 15:
Copper corrosion test

Flux de brasage tendre — Méthodes d'essai —

Partie 15: Essai de corrosion du cuivre

Reference number
ISO 9455-15:1996(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 9455-15 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee 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 (excluding fluoride) 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: Surface insulation resistance comb test and electrochemical migration test of flux residues*

Annex A of this part of ISO 9455 is for information only.

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Soft soldering fluxes — Test methods —

Part 15:
Copper corrosion test

1 Scope

This part of ISO 9455 specifies a qualitative method for determination of the corrosive properties of flux residues on a copper substrate, when subjected to controlled environmental conditions. The test is applicable to type 1 fluxes, 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 1634-1:1987, *Wrought copper and copper alloy plate, sheet and strip — Part 1: Technical conditions of delivery for plate, sheet and strip for general purposes.*

ISO 9453:1990, *Soft solder alloys — Chemical compositions and forms.*

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 9455-2:1993, *Soft soldering fluxes — Test methods — Part 2: Determination of non-volatile matter, ebulliometric method.*

IEC 68-2-3:1969, *Environmental testing — Part 2: Tests — Test Ca: Damp heat, steady state.*

3 Principle

A pellet of solder is melted in contact with the flux to be tested on a test piece of copper sheet. The test piece is then exposed to a controlled temperature/humidity environment and the resulting corrosion of the copper, if any, is assessed using a low-power microscope.

4 Reagents and materials

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

4.1 Ammonium peroxodisulfate solution, prepared as follows.

Dissolve 250 g of ammonium peroxodisulfate [$(NH_4)_2S_2O_8$] in water and add cautiously 5 ml of sulfuric acid (density 1,84 g/ml). Mix, cool, dilute to 1 litre and mix. This solution is to be freshly prepared before use.

4.2 Sulfuric acid, 5 % (V/V) solution.

Add cautiously, with stirring, 50 ml of sulfuric acid (density 1,84 g/ml) to 400 ml of water and mix. Cool, dilute to 1 litre and mix well.

4.3 Degreasing solvent, such as acetone or petroleum ether.

4.4 0,5 mm thick copper sheet, complying with ISO 1634-1, grade Cu—ETP, condition HA.

4.5 Solder wire or pellets, complying with ISO 9453, grade S-Sn63Pb37.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Solder bath, either circular with diameter not less than 120 mm, or rectangular with dimensions not less than 100 mm × 75 mm, containing tin-lead solder having a liquidus less than 200 °C. The depth of the solder in the bath shall be not less than 40 mm. The bath shall be capable of being maintained at a temperature of (233 ± 5) °C.

5.2 Humidity chamber, conforming to the temperature and humidity requirements of IEC 68-2-3, test Ca.

5.3 Cupping device (such as an Erichsen cupping machine).

The device shall be fitted with a 27 mm diameter die and a 20 mm diameter steel ball (see figure 1).

5.4 Drying oven (air circulating), suitable for use at (60 ± 2) °C.

5.5 Low-power stereomicroscope, capable of × 20 magnification, equipped with quartz-halogen illumination.

5.6 Tongs, or other suitable mechanical device, to lift the test piece from the surface of the molten solder bath.

6 Preparation of tests pieces

From a sheet of copper 0,5 mm thick (4.4), cut square test pieces each 50 mm × 50 mm.

Clamp each of the test pieces, in turn, centrally onto the 27 mm diameter die of the cupping device (5.3). Using the 20 mm diameter steel ball, make a 3 mm deep depression in the centre of each test piece by forcing the ball into the die (see figure 1). One corner of the test piece may be bent up to facilitate handling with the tongs (5.6).

Immediately before use, pretreat the test pieces in accordance with the following sequence of operations a) to h), ensuring that clean tongs (5.6) are used for handling.

a) Degrease the test pieces with a suitable neutral organic solvent (4.3).

b) Immerse them in sulfuric acid solution (4.2) at (65 ± 5) °C for 1 min to remove the tarnish film.

c) Immerse them in the ammonium peroxodisulfate solution (4.1) at 20 °C to 25 °C for 1 min to etch the surface uniformly.

d) Wash them under running tap water for a maximum of 5 s.

e) Immerse them in sulfuric acid solution (4.2) at a temperature not greater than 25 °C for 1 min.

f) Wash them under running water for about 5 s and rinse in distilled or deionized water. Immerse immediately in the degreasing solvent (4.3).

g) Allow the test pieces to dry in clean air.

h) Use the test pieces immediately or after a maximum storage period of up to 60 min in a closed container.

Dimensions in millimetres

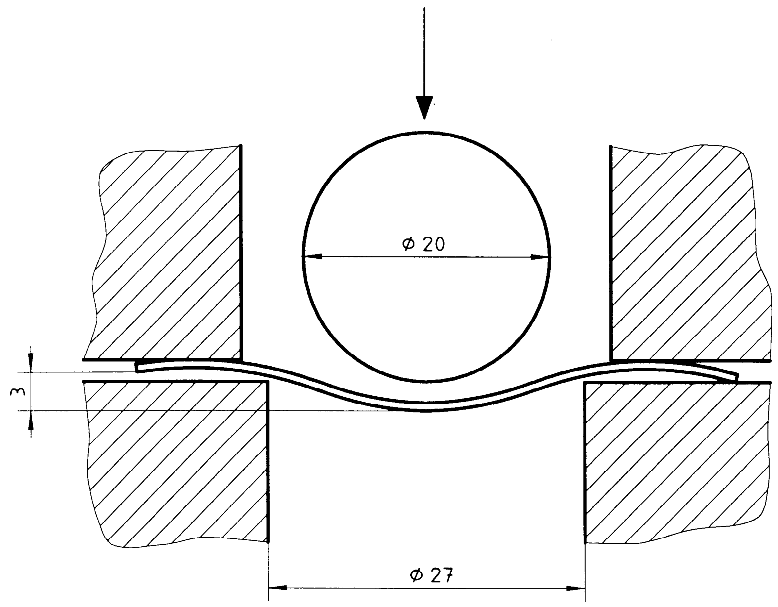

Figure 1 — Dimensions of penetrator, die and blank-holder

7 Procedure

7.1 General

Select three cleaned copper test pieces (clause 6), one of which will serve as the blank. Follow the procedures in 7.2 to 7.6 on the three test pieces, omitting the procedures in 7.4 in the case of the blank.

7.2 Fluxing the test pieces

7.2.1 For solid, paste and liquid flux samples

Weigh (1,00 ± 0,05) g of the solder wire or pellets (4.5), previously degreased with a neutral organic solvent (4.3), and transfer to the centre of the depression in one of the test pieces (7.1).

NOTE 1 This may conveniently be done, if wire is used, by forming the wire into a small flat coil.

Repeat for the other two test pieces (7.1).

According to the form of the flux under test, continue with the preparation of the test pieces by following the procedure given in either a) or b) as follows.

a) If the flux under test is in solid or paste form, weigh between 0,035 g and 0,040 g of the solid or paste flux and add this to the solder in the depression of the test piece. Repeat for the other two test pieces.

b) If the flux under test is in liquid form, first determine its non-volatile matter content by the use of the method described in ISO 9455-1 or ISO 9455-2, then add the appropriate volume of the liquid flux, to contain between 0,035 g and 0,040 g of non-volatile matter, to the solder in the depression of the test piece. If the non-volatile matter content of the liquid flux is so low that the volume required would more than fill the depression, then use sufficient flux to just fill the depression. Repeat for the other two test pieces.

Evaporate the solvent at (60 ± 2) °C for 10 min in the drying oven (5.4).

7.2.2 For flux-cored solder samples

If the flux is present in the form of flux-cored solder, degrease the surface of a suitable length of the cored solder sample, using a cloth dampened with the solvent (4.3). Weigh (1,00 ± 0,05) g of the degreased sample, form it into a small flat coil and place it in the centre of the depression in one of the test pieces (7.1). Repeat for the other two test pieces.

7.2.3 For solder paste samples

If the flux is present in the form of solder paste, weigh (0,50 ± 0,05) g of the solder paste sample into the centre of the depression in one of the test pieces (7.1). Repeat for the other two test pieces.

7.3 Heating the test pieces

Using the tongs (5.6) or other suitable means, carefully lower the fluxed test piece from (7.2) onto the surface of the molten solder, maintained at (233 ± 5) °C in the solder bath (5.1).

Allow the test piece to remain in contact until the solder melts and leave the test piece in this position for a further 5 s.

Maintaining the test piece in a horizontal position, remove it carefully from the heating bath and allow it to cool for 30 min.

Repeat for the other two test pieces.

Examine the specimens using the microscope (5.5) at × 20 magnification and record their surface appearance for subsequent comparison after conditioning (see 7.4 and 7.6.2).

NOTE 2 It is recommended that a camera be used to obtain a photographic record to facilitate this comparison.

7.4 Conditioning the test pieces

Retaining one of the specimens from 7.3 as a blank, preheat the other two to 40 °C (see note 3) and then place them in a vertical position in the humidity chamber (5.2) operating at a temperature of (40 ± 2) °C and at a relative humidity of 90 % to 95 %.

NOTE 3 Preheating the test pieces avoids condensation onto the test pieces when they are placed in the chamber, which, if it occurred, would invalidate the test.

Allow the two specimens to remain in the chamber, under these conditions of temperature and humidity, for 3 days.

7.5 Examination of the test pieces

Remove the test pieces from the humidity chamber. Examine for evidence of corrosion, using the microscope (5.5) at × 20 magnification and compare with the surface appearance of the pieces before conditioning (see 7.3) and with the blank, assessing the corrosion in accordance with 7.6.

7.6 Assessment of corrosion

7.6.1 General

In this test, corrosion results from a progressive chemical reaction between the copper, the solder and the constituents in the flux residues. For a corrosive flux, the corrosion occurs after soldering and during exposure to the test conditions given in 7.4.

When corrosion occurs, growths of solid corrosion product resulting from the chemical reaction become visible

1) at the boundaries of the flux residues on the copper, and/or

2) through discontinuities or cracks in the residues, and/or

3) as spots under the residues.

In all these cases, the corrosion products may be green, blue-green or white in colour. Examples illustrating corrosion of these types are given in annex A.

Each of the figures shown in annex A illustrates a typical used test piece after conditioning, showing the silver/blue solder in the centre of the depression in the test piece and surrounded by the flux residue. Each of these is accompanied by a magnified view of a specific area of the test piece (two magnified views in the case of figure A.1 e)].

NOTE 4 The figures shown in annex A are provided for guidance only.

Figures A.1 a) and A.1 b) illustrate test pieces corresponding to typical results obtained from fluxes which PASS the test. Figures A.1 c) and A.1 e) show increasing degrees of corrosion, and all correspond to fluxes which FAIL the test. The principal features of these figures are as follows.

- Figure A.1 a): The flux residue is of a brown to brownish-green translucent appearance. The residue is cracked, but there is no evidence at all of any growth of corrosion product. This flux PASSES the test.

- Figure A.1 b): The flux residue is translucent, and in the magnified view olive-green areas are evident in the flux residue due to dissolved copper salts. There is no evidence at all of any growth of corrosion product. This flux PASSES the test.

- Figure A.1 c): In the magnified area the small blue spots at the flux/copper boundary are clear evidence of the growth of corrosion product. This flux FAILS the test.

- Figure A.1 d): A considerable amount of blue corrosion growth in the darker areas of the residue, in both views illustrated, is evidence of severe corrosion. This flux FAILS the test.

— Figure A.1 e): The magnified views reveal extensive blue spots of corrosion product growth under the flux residue and in cracks in the residue. This flux FAILS the test

7.6.2 Examination after fluxing and melting

When the test pieces are examined after melting the solder, but before conditioning in the humidity chamber (see 7.3), and show signs of blue spots as in figure A.1 c) or A.1 d), the flux shall be deemed to have failed the test, and further testing is unnecessary. However, any change in the colour of the residue which may be observed at this stage of the test procedure, usually to a green hue, without the growth of solid corrosion products, shall be ignored.

7.6.3 Examination after conditioning

When the test pieces are examined after conditioning in the humidity chamber (see 7.4 and 7.5), and appear comparable with the typical examples given in figures A.1 a) and A.1 b), the flux shall be deemed to have passed the test. However, if at this stage the test pieces reveal the evidence of corrosion comparable with the typical examples given in figures A.1 c), A.1 d) or A.1 e), the flux shall be deemed to have failed the test.

NOTE 5 During conditioning in the humidity chamber, the flux residue and/or the copper test pieces may change in appearance without exhibiting corrosion. For example, the copper may become tarnished or the flux residue may become opaque. These effects, if they occur, should be ignored.

8 Expression of results

The results shall be assessed in accordance with the criteria given in 7.5 and 7.6 and recorded as PASS or FAIL.

9 Test report

The test report shall include the following information:

a) identification of the test sample;

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

c) results obtained;

d) any unusual features noted during the test;

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