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ISO 9455-10

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Soft soldering fluxes — Test methods —

Part 10:
Flux efficacy tests, solder spread method

Flux de brasage tendre — Méthodes d'essai —

Partie 10: Essais d'efficacité du flux, méthode d'étalement

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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 9455-10 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 and detection 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*

— *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*

Annexes A to C of this part of ISO 9455 are for information only.

Soft soldering fluxes — Test methods —

Part 10:
Flux efficacy tests, solder spread method

1 Scope

This part of ISO 9455 specifies a method for the determination of the efficacy of a soft soldering flux. The method is known as the solder spread method and is applicable to all flux classes defined in ISO 9454-1.

NOTES

1 This part of ISO 9455 is only applicable for liquid fluxes > 10 % (*m*/*m*).

2 An alternative method for the determination of the flux efficacy, applicable to liquid fluxes only, known as the wetting balance method, is specified in ISO 9455-16.

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 edition of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 197-1:1983, *Copper and copper alloys — Terms and definitions — Part 1: Materials*.

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 3611:1978, *Micrometer callipers for external measurement*.

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*.

3 Principle

A specially prepared brass test sheet is treated with a known quantity of the flux under test and a standard quantity of specified solder alloy. Upon heating, the solder alloy melts and spreads across the surface of the brass test sheet, the extent of spread being a measure of the flux efficacy. The area covered by the solder is determined by means of a planimeter, or other suitable technique.

This test method is applicable to all fluxes. If required, the efficacy of the flux sample under test can be compared with that of a standard flux (see annex A).

NOTE — It is possible to use copper as the test surface. However, it is preferable to use brass as the test surface in order to distinguish between various fluxes because it provides a better surface for differentiation than copper would.

4 Reagents

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

4.1 Acid preparation solution, prepared in a fume cupboard by cautiously mixing the following ingredients in the order given, cooling as necessary.

— 140 ml water;

— 225 ml nitric acid ($\rho = 1{,}42$ g/ml);

— 600 ml sulphuric acid ($\rho = 1{,}84$ g/ml);

— 5 g sodium chloride;

— 18 ml copper solution (4.2).

Thoroughly mix the solution.

This preparation solution shall be freshly prepared every day.

WARNING: The mixture is extremely corrosive and produces hazardous fumes.

4.2 Copper solution, prepared by dissolving 10 g pure copper (analytical grade) turnings in 100 ml of 50 % (*V*/*V*) nitric acid solution ($\rho = 1{,}42$ g/ml).

4.3 Industrial methylated spirits.

4.4 Acetone.

5 Apparatus

5.1 Solder bath, containing not less than 4 kg of molten tin-lead solder, having a liquidus of less than 200 °C. The liquid solder in the bath shall be at least 25 mm in depth, with a surface area easily capable of accommodating the test specimen.

The bath shall be capable of being maintained at a temperature of 300 °C ± 5 °C.

5.2 Tongs, or other suitable mechanical device, for lowering the brass test plate (6.1), in a horizontal plane, onto the surface of the liquid solder in the bath (5.1) and raising it again, also in a horizontal plane.

5.3 Planimeter, suitable for measuring surface areas of the order of 100 mm^2.

5.4 Microsyringe, or micropipette, capable of delivering 25 µl (i.e. 0,025 ml).

5.5 Plastic tweezers or tongs, for use in the cleaning procedure for test plates.

5.6 Micrometer, as specified in ISO 3611.

5.7 Filter paper, for use in cleaning procedure for test plates.

6 Test specimens

6.1 Brass test plates

Each plate shall be of 40 mm × 40 mm. Ten (10) plates cut from brass sheet, 0,5 mm thick, complying with ISO 1634-1 alloy CuZn37 or CuZn40, condition HA, for each flux being tested.

NOTES

1 Annex B gives the chemical composition of these two brasses.

2 Copper test plates, each 40 mm × 40 mm. Ten (10) plates cut from phosphorus deoxidized sheet, 0,5 mm thick, complying with ISO 197-1, for each flux being tested, may also be used.

3 One corner of the test plate may be bent, to facilitate handling with the tongs (5.2).

6.2 Solder sample

Clean a length of S-Sn60 Pb40 solder wire, with a diameter of 1 mm ± 0,05 mm, weighing between 0,49 g and 0,50 g and complying with ISO 9453 with a filter paper soaked in industrial methylated spirits (4.3) or acetone (4.4). Wind the length of wire into a tight, flat spiral for use in the test. After cleaning, handle the solder wire only with clean cotton gloves.

7 Procedure

7.1 Preparation of test plates

Immediately before use pretreat the ten test pieces in the following manner; handle with clean tongs or with a suitable mechanical device (5.2).

Degrease the test plates (6.1) thoroughly in acetone (4.4) and allow to dry in clean air.

Immerse each test plate separately for approximately 15 s, in the acid preparation solution (4.1) using the plastic tweezers or tongs (5.5). Hold the solution at a temperature between 15 °C and 25 °C.

Wash the test plates in distilled or deionized water for a maximum of 5 s.

Repeat the acid preparation operation, not more than three times, until a uniform matt lustre on the surface of the test plates has been attained.

After the final rinsing in deionized water, rinse the test plates in methylated spirits (4.3) and dry them with filter paper (5.7).

NOTE — This preparation method is applicable to both brass and copper test plates.

7.2 Test method

Immediately after the cleaning operation described in 7.1, take one of the test plates and apply the flux sample under test to the central region of the plate, in accordance with a) or b) as follows:

a) For liquid flux samples: measure 0,025 ml ± 5 % of the sample by means of the microsyringe or micropipette (5.4);

b) For paste flux samples: weigh 0,025 g ± 5 % of the sample.

Place a solder sample (6.2) in the middle of the test plate. Lower the test plate, with its plane horizontal, on to the surface of the solder in the solder bath (5.1) which is maintained at 300 °C ± 5 °C.

After 5 s, remove the test plate from the solder bath, keeping it in a horizontal plane until it has cooled to room temperature.

Remove the flux residue with a suitable solvent.

Determine the area, in mm^2, covered by the melted solder alloy by use of the planimeter (5.3), or measure the height of the solder by use of a micrometer (5.6) and calculate the ratio of spread given in % by the formula:

$$\text{Ratio of spread } (\%) = \frac{D - H}{D} \times 100$$

where

H is the height of spread solder, in millimetres;

D is the diameter, in millimetres, when the solder used is considered as a sphere, $D = 1{,}24 \times V^{1/3}$;

V is the volume, in millilitres, of the solder used.

7.3 Replicate tests

Using the remaining nine test plates, re-prepare as described in 7.1, repeat the test method described in 7.2, calculating either the area or the ratio of spread as in the original sample.

8 Expression of results

The efficacy of the flux sample under test is expressed as the arithmetic mean and the standard deviation of the ten areas or ratios of spread, in mm^2, measured in the tests carried out as described in clause 7.

The efficacy of the flux sample under test may also be expressed in comparison with results obtained by using the test described in clause 7 on a standard flux, prepared as described in annex A.

9 Precision

Interlaboratory tests were carried out on two colophony fluxes without addition of halide and with 0,6 % added halide.

Nine laboratories took part in the tests with the results given in tables 1 to 3.

Table 1 — Precision for halide- and non-halide-type fluxes

Parameter		Flux type 1.1.2 0,6 % halide	Flux type 1.1.3 non halide
Arithmetic mean in mm^2	m	283,1	155,7
Standard deviations			
— within laboratory	s_w [1)]	34,4	14,3
— between laboratories	s_b [1)]	62,8	20,4
Repeatability	r [1)]	96,4	40,0
Reproducibility	R [1)]	175,7	57,2
1) The evaluation is based on single values.			

Table 2 — Precision for ratio of spread values on brass plates

Parameter		Flux type				
		1.1.	1.1.3	1.2.2	2.2.2	3.2.1
Arithmetic mean in mm^2	m	106,95	122,68	155,07	192,95	215,57
Standard deviations						
— within laboratory	s_w	7,44	8,33	11,70	10,77	10,36
— between laboratories	s_b	18,42	8,42	12,71	14,41	14,19
Repeatability	r	20,84	23,23	32,77	30,16	29,02
Reproducibility	R	51,58	23,57	35,60	40,36	39,72

Table 3 — Precision for area of spread and ratio of spread on copper plates

Parameter		Flux type									
		1.1.1		1.1.3		1.2.2		2.2.2		3.2.1	
		Area of spread	Ratio of spread	Area of spread	Ratio of spread	Area of spread	Ratio of spread	Area of spread	Ratio of spread	Area of spread	Ratio of spread
Arithmetic mean	m	42,17	55,12	146,02	85,90	124,17	80,40	214,23	89,81	224,21	90,41
Standard deviations											
— within laboratory	s_w	5,45	4,10	4,22	0,45	6,36	1,39	18,12	1,88	11,22	0,45
— between laboratories	s_b	7,25	5,34	4,59	0,53	23,89	4,37	25,31	2,33	21,10	0,58
Repeatability	r	15,25	11,48	11,80	1,26	17,82	3,89	50,74	5,26	31,41	1,27
Reproducibility	R	20,31	14,96	12,86	1,49	66,89	12,23	70,87	6,52	59,08	1,63

10 Test report

The test report shall include the following information:

a) identification of the test sample;

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

c) the result obtained (i.e. area or ratio of spread 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.