INTERNATIONAL STANDARD

ISO 9455-10

Second edition
2012-09-15

Soft soldering fluxes — Test methods —

Part 10:
Flux efficacy test, solder spread method

Flux de brasage tendre — Méthodes d'essai —

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

Reference number
ISO 9455-10:2012(E)

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Foreword

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9455-10 was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering 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 test, solder spread method*

— *Part 11: Solubility of flux residues*

— *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*

Requests for official interpretations of any aspect of this part of ISO 9455 should be directed to the Secretariat of ISO/TC 44/SC 12 via your national standards body. A complete listing of these bodies can be found at www.iso.org.

Soft soldering fluxes — Test methods —

Part 10:
Flux efficacy test, 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 of filler metal and is applicable to all flux classes defined in ISO 9454-1.

NOTE 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.[3]

2 Normative references

The following referenced documents are indispensable for the application 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 9453, *Soft solder alloys — Chemical compositions and forms*

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

3 Symbols

$\bar{A}$ arithmetic mean spread area, in square millimetres

H height of spread solder, in millimetres

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

R reproducibility

r repeatability

s_w standard deviation within laboratory

s_b standard deviation between laboratories

ρ density, in grams per millilitre, of solder under test at the test temperature

V volume, in millilitres, of the solder used

4 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 does.

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 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 (ρ = 1,42 g/ml);

— 600 ml sulfuric acid (ρ = 1,84 g/ml);

— 5 g sodium chloride;

— 18 ml copper solution (5.2).

Thoroughly mix the solution.

This preparation solution shall be freshly prepared every day.

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

5.2 Copper solution, prepared by dissolving 10 g pure copper turnings in 100 ml of 50 % by volume nitric acid solution (ρ = 1,42 g/ml).

5.3 Industrial methylated spirits.

5.4 Acetone.

6 Apparatus and materials

6.1 Solder bath, containing not less than 4 kg of molten solder. 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. As a minimum, the bath temperature shall be capable of being maintained at a set temperature of the liquidus temperature of the alloy in the test plus 35 °C. The test temperature shall be documented.

6.2 Tongs, or other suitable mechanical device, for lowering the brass test plate (7.1), in a horizontal plane, onto the surface of the liquid solder in the bath (6.1) and raising it again, also in a horizontal plane.

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

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

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

6.6 Micrometer, as specified in ISO 3611.[2]

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

7 Test specimens

7.1 Brass test plates

Each plate shall be of dimensions 40 mm × 40 mm. Prepare 10 plates, of alloy CuZn37 or CuZn40, cut from brass sheet 0,5 mm thick, for each flux being tested.

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

NOTE 2 An alternative is 10 oxidized copper test plates, each of dimensions 40 mm × 40 mm, cut from phosphorus deoxidized sheet, 0,5 mm thick, complying with ISO 197-1,[1] for each flux being tested.

NOTE 3 One corner of the test plate can be bent, to facilitate handling with the tongs (6.2).

7.2 Solder sample

Clean a length of solid solder wire as agreed between the contracting parties, with a diameter of 1 mm ± 0,05 mm, weighing between 0,49 g and 0,50 g with a filter paper soaked in industrial methylated spirits (5.3) or acetone (5.4). 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.

The solder used for the test and the test temperature shall be selected from one of the following:

— S-Sn60Pb40 (in accordance with ISO 9453) at 235 °C ± 3 °C;

— S-Sn96Ag3Cu1 (in accordance with ISO 9453) at 255 °C ± 3 °C;

— any other solder or temperature combinations as agreed between the customer and the flux supplier — for test temperatures, see 6.1.

8 Procedure

8.1 Preparation of test plates

Immediately before use, pretreat the 10 test pieces in the following manner; handle with clean tongs or with a suitable mechanical device (6.2).

Degrease the test plates (7.1) thoroughly in acetone (5.4) and allow to dry in clean air.

Immerse each test plate separately for approximately 15 s in the acid preparation solution (5.1) using the plastic tweezers or tongs (6.5). Hold the solution at a temperature between 15 °C and 25 °C.

Wash the test plates in 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 water, rinse the test plates in methylated spirits (5.3) and dry them with filter paper (6.7).

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

8.2 Test method

Immediately after the cleaning operation specified in 8.1, 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):

a) for liquid flux samples: measure 0,025 ml ± 0,001 25 ml of the flux sample by means of the microsyringe or micropipette (6.4);

b) for paste and solid flux samples: weigh 0,025 g ± 0,001 25 g of the flux sample.

Place a solder sample (7.2) 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 of filler metal (6.1).

After 5 s of the start of spreading, 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 square millimretres, covered by the melted solder alloy by use of the planimeter (6.3), or measure the height of the solder by use of a micrometer (6.6) and calculate the ratio of spread, expressed as a percentage, by the formula:

$$\frac{D-H}{D}\times 100$$

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.

8.3 Replicate tests

Using the remaining nine test plates, re-prepare as specified in 8.1, repeat the test method specified in 8.2, calculating either the area or the ratio of spread as in the original sample.

9 Expression of results

The efficacy of the flux sample under test is expressed as the arithmetic mean and the standard deviation of the 10 areas of spread, in square millimetres or ratios of spread, expressed as a percentage, measured in the tests carried out as described in Clause 8.

The efficacy of the flux sample under test may also be expressed in comparison with results obtained by using the test described in Clause 8 on a standard flux, prepared as described in Annex A.

10 Precision

Interlaboratory tests have been carried out on two colophony fluxes without addition of halide and with 0,6 % by mass 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 % by mass halide	Flux type 1.1.3 halide-free
Arithmetic mean, mm²	$\bar{A}$	283,1	155,7
Standard deviations			
— within laboratory	s_w [a]	34,4	14,3
— between laboratories	s_b [a]	62,8	20,4
Repeatability	r [a]	96,4	40,0
Reproducibility	R [a]	175,7	57,2

[a] The evaluation is based on single values.

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

Parameter		Flux type				
		1.1.1	1.1.3	1.2.2	2.2.2	3.2.1
Arithmetic mean, mm^2	$\bar{A}$	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	$\bar{A}$	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

11 Test report

The test report shall include at least the following information:

a) identification of the test sample;

b) the test method used, i.e. reference to this part of ISO 9455 (ISO 9455-10:2012);

c) identification of test plate;

d) grade of solder tested;

e) temperature of test;

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

g) any unusual features noted during the determination;

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

Annex A
(informative)

Method for the preparation of standard reference rosin (colophony) based liquid fluxes, having 25 % by mass non-volatile content

A.1 General

This annex gives a method for the preparation of two standard rosin (colophony) based liquid fluxes having 25 % by mass non-volatile content, one non-activated and the other halogen activated (i.e being class 1.1.1.A and class 1.1.2.A, respectively, as defined in ISO 9454-1. The specifications for the flux constituents are based on IEC 60068-2-20:2008,[4] Annex B.

The standard flux may be used as a reference against which the efficacy of the flux under test may be compared (see Clauses 4 and 9).

A.2 Principle

The non-activated flux is prepared by dissolving a special grade of rosin (colophony) in propan-2-ol. The halogen-activated flux is prepared in a similar way, with the addition of diethylamine hydrochloride.

A.3 Apparatus

Usual laboratory apparatus, including an oven capable of being maintained at 110 °C ± 2 °C, is required.

A.4 Reagents

Use only reagents of recognized analytical grade.

A.4.1 Rosin (colophony). Water white grade gum rosin, or equivalent, which shall comply with the following requirements:

— acid value (as KOH): 155 mg/g to 180 mg/g

— softening point: 70 °C minimum

— flow point: 76 °C minimum

— ash: 0,05 % by mass maximum

— solubility: to give a clear 1 + 1 solution in propan-2-ol

A.4.2 Diethylamine hydrochloride. Dried for 2 h at 110 °C ± 2 °C.

A.4.3 Propan-2-ol (isopropanol), which shall comply with the following requirements:

— propan-2-ol: 99,5 % by mass minimum

— acid content: 0,002 % by mass maximum

— non-volatile content: 0,2 % by mass maximum

A.5 Procedure

A.5.1 Non-activated rosin (colophony)

Weigh 25 g ± 0,1 g rosin (A.4.1) and dissolve it with gentle mixing in 75 g ± 0,1 g propan-2-ol (A.4.3).

A.5.2 Halogen-activated rosin (colophony)

Weigh 0,39 g ± 0,01 g diethylamine hydrochloride (A.4.2) and dissolve it in 75 g ± 0,1 g propan-2-ol (A.4.3). Then add 25 g ± 0,1 g rosin (A.4.1) and dissolve it with gentle mixing. This flux solution contains a nominal 0,5 % by mass active chloride.

A.6 Storage

Store the standard flux solutions, prepared as described in A.5, in a container, properly closed at all times, away from heat or extreme cold.

Annex B
(informative)

Chemical composition of brass test plates

Table B.1 — Chemical composition of brass test plates

Values as percentages by mass

Alloy	Cu	Zn	Fe	Pb
CuZn37	62,0 min. 64,0 max.	balance	— 0,1 max.	— 0,1 max.
CuZn40	59,5 min. 61,5 max.	balance	— 0,2 max.	— 0,3 max.

Bibliography

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

[2] ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

[3] ISO 9455-16, *Soft soldering fluxes — Test methods — Part 16: Flux efficacy test, wetting balance method*

[4] IEC 60068-2-20:2008, *Environmental testing — Part 2-20: Tests — Test T: Test methods for solderability and resistance to soldering heat of devices with leads*

[5] IEC 60068-2-54, *Environmental testing — Part 2-54: Tests — Test Ta: Solderability testing of electronic components by the wetting balance method*

ICS 25.160.50

Price based on 9 pages