International Standard

ISO 9455-18

First edition
2024-08

Soft soldering fluxes — Test methods —

Part 18: Cleanliness of soldered printed circuit assemblies before and/or after cleaning

Reference number
ISO 9455-18:2024(en)

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Published in Switzerland

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Foreword

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This document was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 12, *Soldering materials*.

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. Official interpretations of ISO/TC 44 documents, where they exist, are available from this page: https://committee.iso.org/sites/tc44/home/interpretation.html.

Soft soldering fluxes — Test methods —

Part 18: Cleanliness of soldered printed circuit assemblies before and/or after cleaning

1 Scope

This document specifies test methods for the cleanliness of soldered printed circuit assemblies before and/or after soldering and cleaning. The test is applicable to all fluxes as defined in ISO 9454-1.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
flux residue
flux components remaining around the soldering area after reflow

3.2
white residue
flux components remaining around the soldering area after cleaning

4 Principle

Test sample before and/or after cleaning the soldered printed circuit assemblies is prepared without contamination. Then, the test sample is tested by the flow chart given in Figure 1. The test methods are detailed in Table 1.

The first step is to check for the presence or absence of flux residue on the PCB by microscope and/or SEM.

Next step is identification and/or reliability test of the flux residue. Identification of flux residue is carried out by SEM/EDX and/or FT-IR. Reliability tests are carried out by ionic contamination tester (Ref. ROSE test) and/or SIR and/or dielectric property test. This second step tests are optional, where users can select the test methods to meet user's requirements. An example of test methods and test results of cleanliness of the soldered printed circuit boards before and/or after cleaning is provided in Annex A. An example for a FT-IR analysis is shown in Annex B.

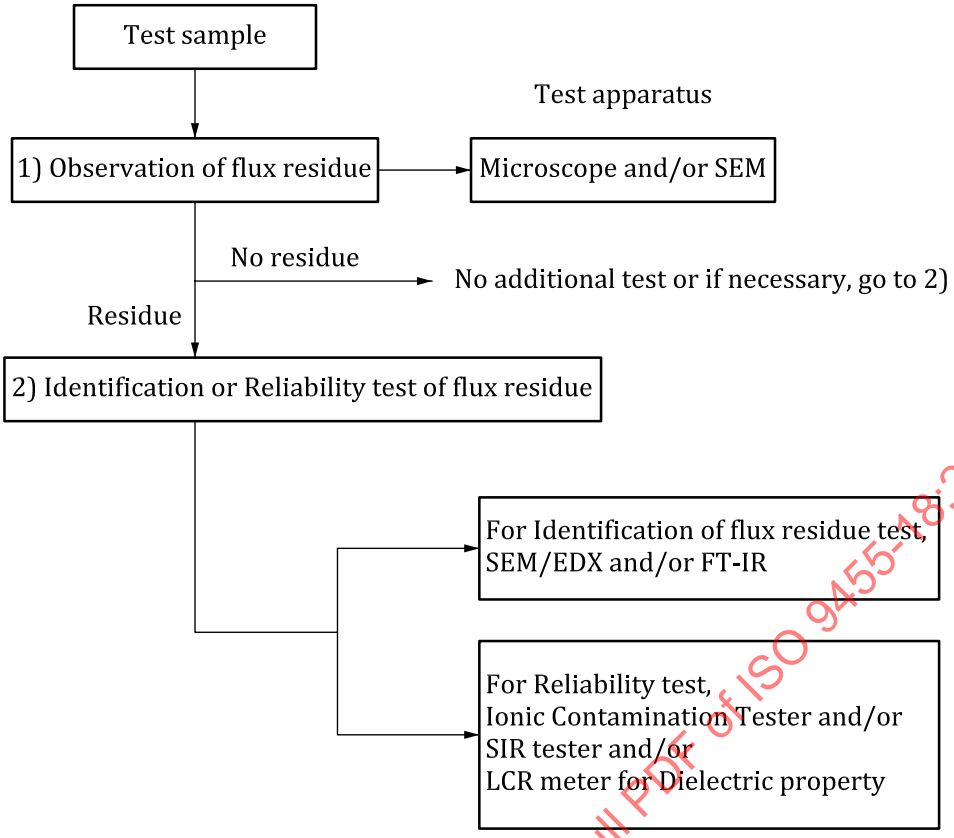


Figure 1 — Flowchart of test methods of cleanliness of the soldered printed circuit assemblies before and/or after cleaning

Table 1 — Test methods of cleanliness of the soldered printed circuit assemblies

Step	Test item	Test apparatus	Details
1	Observation of flux residue	Microscope	Observation of flux residue
		SEM	Usually more detectable than Microscope Qualitative test of the flux residue determining organic or inorganic residue
2	Identification of flux residue	SEM/EDX	Determination of residual element, for example, C, O, Cl, Br, Sn, Ag · · · and mapping of each element
		FT-IR	Functional group, for example, -COOH, -NH, et al.
	Reliability test Ionic residue	Ionic contamination tester	Electrical conductivity of the extract by mixture of IPA and DI water
	Reliability test SIR	SIR tester	Surface insulation resistance test of flux residue
	Reliability test Dielectric property	LCR meter	Dielectric property of flux residue

5 Reagents and cleaning solvent

In the test, use only reagents of recognized analytical grade or higher.

Cleaning solvent as recommended by manufacturer or supplier.

NOTE Cleaning solvent to be used can vary with the flux composition.

6 Apparatus

6.1 Microscope, capable of magnification x20 to x100.

6.2 SEM-EDX, capable of magnification x20 to x1 000 and detecting organic and inorganic elements.

6.3 FT-IR, capable of detecting functional group like -COOH, -NH.

6.4 Ionic contamination tester, capable of detecting ionic contamination as µg-NaCl calculated from conductivity of the extract solution.

6.5 Temperature/humidity oven, controlled temperature and humidity chamber.

7 Procedure

7.1 Preparation of the test sample

Test sample should be prepared without contamination. When test boards are cleaned, cleaning solvent and cleaning condition should be agreed between manufacturer and supplier. If necessary, test sample should be pre-treated to meet the instruction manual of the test apparatus.

7.2 Test method

7.2.1 SIR

Testing procedure is performed with reference to standards such as, ISO 9455-17, IEC 61189-5-501.

7.2.2 Ionic contamination test

Testing procedure is performed with reference to IEC 61189-5-504.

8 Assessment and expression of results

The cleanliness level is tested and decided by each test method. Step 1 tests in Table 1 are to check if there is residue or no residue on the test board. Step 2 tests give more detailed information of flux residue such as identification of the flux residue like elements or function groups, and/or electrical reliability like SIR or ionic contamination or dielectric property of the flux residue.

9 Test report

The test report shall include the following information:

a) the identification of the test sample;

b) the test method used;

c) the results obtained;

d) any usual features noted during the determination;

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

f) details of the cleaning solution and cleaning condition, equipment and parameters used in the preparation of the test sample.

Annex A
(informative)

Example of test methods and test results of cleanliness of the soldered printed circuit boards before and/or after cleaning

A.1 Test condition

A.1.1 Preparation

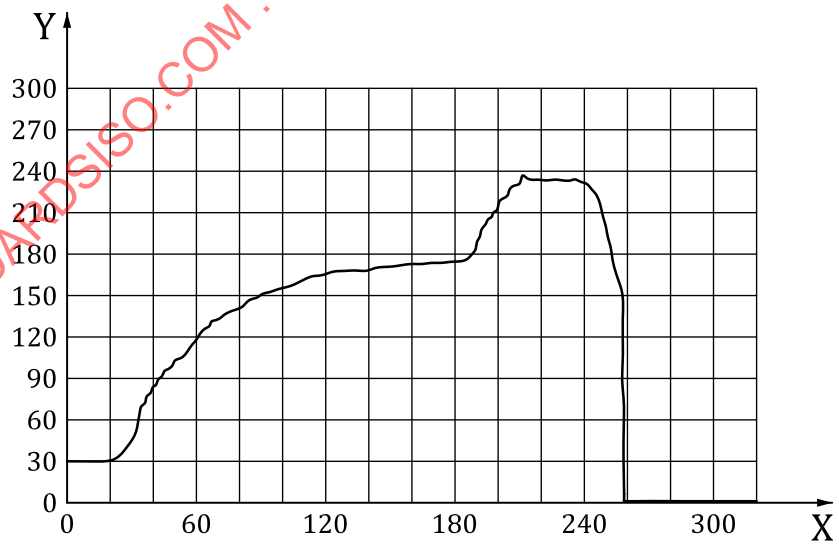
Soldering paste: Mixture of Sn-Ag3-Cu0.5 type 3 powder and controlled flux defined in IEC 60068-2-83. (See Table A.1.)

Printing the soldering paste on the comb pattern test board (land and space 0,318 mm each) and reflow (see Figure A.1 Reflow profile: 150 °C to 180 °C for 98,2 sec and above 221 °C for 48,2 sec).

An example of a table is given in Table 1.

Table A.1 — Test methods of cleanliness of the soldered printed circuit assemblies

Component	Composition	Content (wt%)
Rosin	Polymerized rosin	30,0
	Modified rosin	30,0
Solvent	Diethylene glycol Monobutyl ether	34,7
Activator	1.3-Diphenyl guanidine Hydrobromide	0,8
Organic acid	Adipic acid	0,5
Thixotropic agent	Hydrogenated castor oil	4,0



Key

X (sec)

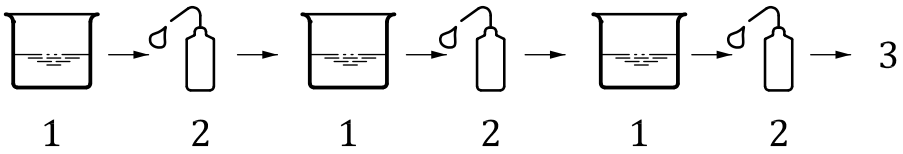
Y (°C)

Figure A.1 — Reflow profile

A.1.2 Cleaning

Cleaning the test board by IPA after reflow. (See Figure A.2.)

IPA was selected as a cleaning solution to make it an insufficient cleaning example.



Key

1 beaker IPA1.5L (40 °C), stirring 620 rpm for 1 min
2 rinse by IPA 100 mL
3 hot air blow drying (80 °C/1 min)

Figure A.2 — Cleaning the test board by IPA after reflow process

A.2 Test results

A.2.1 Microscope

If the cleaning is insufficient, a white residue can be found around the soldering. (See Figure A.3.)

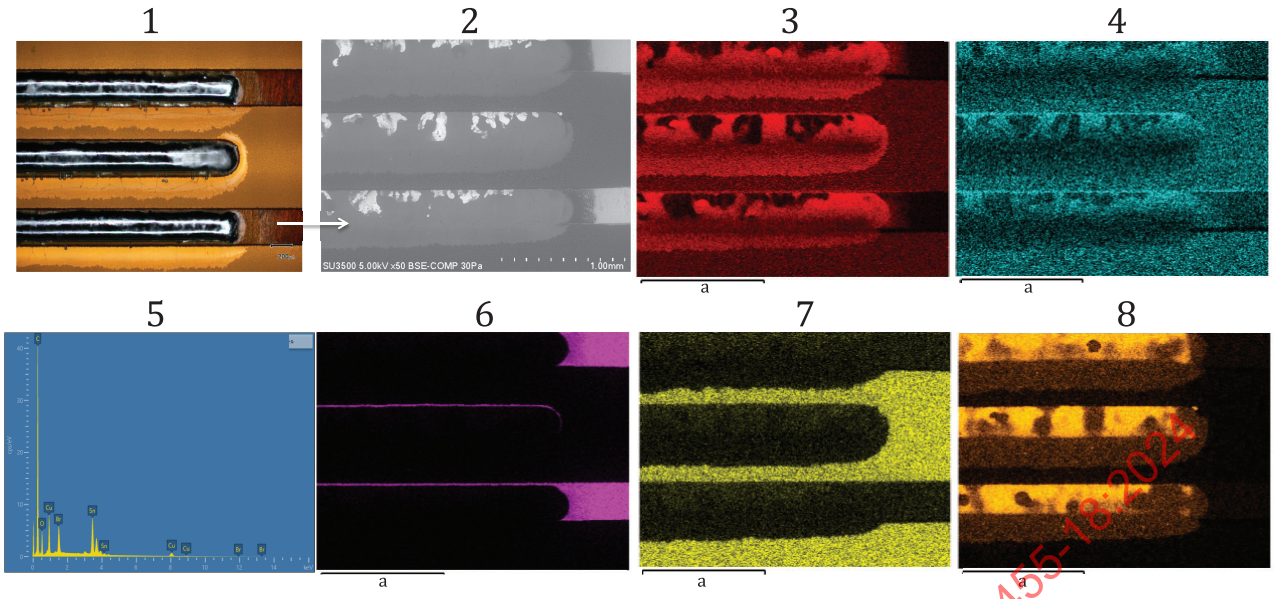
a) Before cleaning **b) Insufficient cleaning**

Figure A.3 — Microscope observation

A.2.2 SEM-EDX

Observation and measurement conditions should be set with optimal values for the target sample and EDX resolution.

If cleaning is insufficient, the value of carbon and oxygen tends to be higher. (See Figure A.4 and Table A.2.)



Key

1 microscope
2 BSE
3 C Kα1_2
4 O Kα1
5 EDX SPECTRUM
6 Cu Lα1_2
7 Br Lα1_2
8 Sn Lα1
[a] 1 mm.

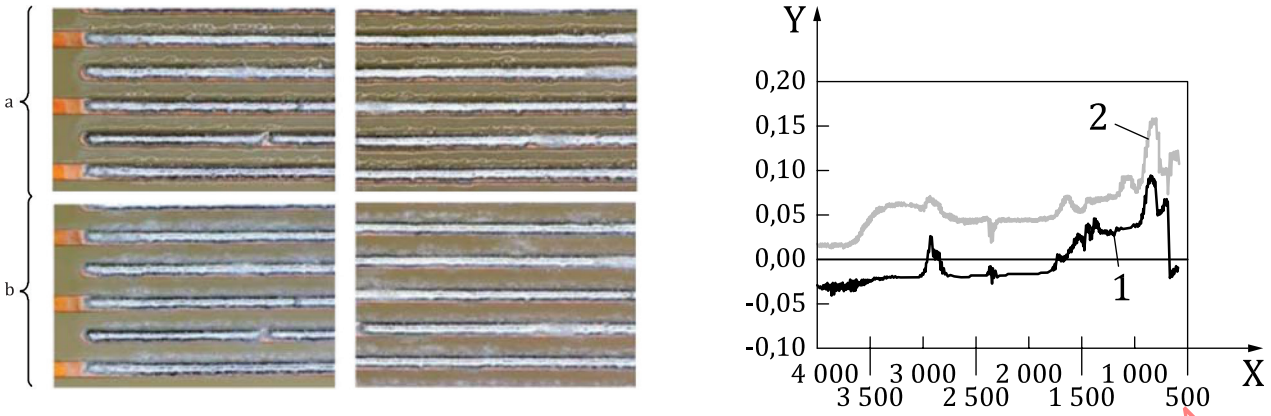
Figure A.4 — SEM observation and EDS analysis with insufficient cleaning

Table A.2 — Element/mass fraction in % of the EDX spectrum in Figure A.4

Element	Mass fraction %
C	53,09
O	8,79
Cu	11,14
Br	5,59
Sn	21,38

A.3 FT-IR

If the cleaning is insufficient, the FT-IR chart is almost the same as before the cleaning. (See Figure A.5.)



a) Microscope **b) FT-IR chart**

Key

X wavenumbers (cm^{-1})
Y absorbance (a.u.)
1 before cleaning
2 insufficient cleaning
a Before cleaning.
b Insufficient cleaning.

Figure A.5 — Microscope observation and FT-IR chart

A.4 Ionic contamination test

The extraction temperature and measurement conditions must be set according to the situation.

If cleaning is insufficient, the value of ionic residue is about the same as before the cleaning. (See Table A.3.)

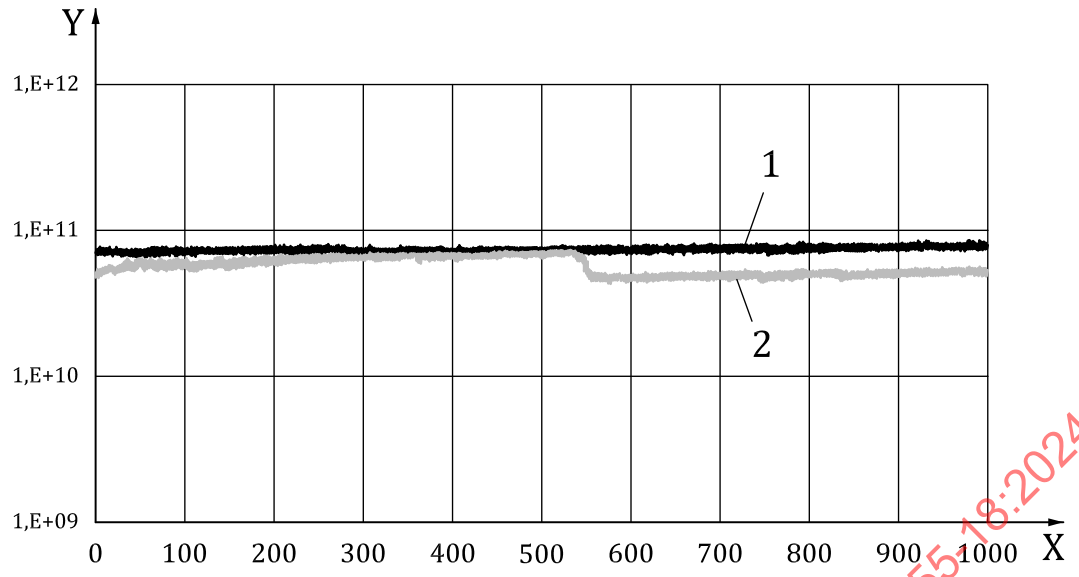
Table A.3 — Measurements of ionic contamination test results in the various conditions

Sample	# of test boards	Electric conductivity MΩ·cm	Ionic residue μgNaCl/cm²
Blank	11	20,65	0,36
Before cleaning	11	16,10	0,50
Insufficient cleaning	11	15,86	0,51

A.5 SIR test

SIR test is applied at 50 V bias and 100 V measuring in the same direction and test environment; 85 °C/85 % RH.

If cleaning is insufficient, the resistance value tends to decrease. (See Figure A.6.)



Key

X time (h)
Y resistance value (Ω)
1 blank
2 insufficient cleaning

Figure A.6 — SIR test

Annex B
(informative)

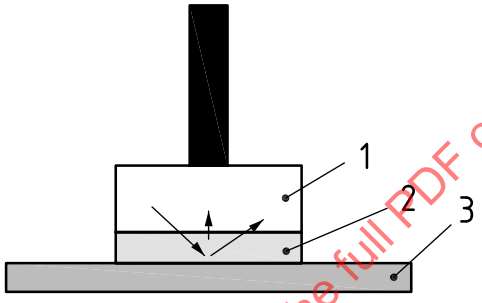
FT-IR analysis

B.1 Measurement methods

B.1.1 ATR (Attenuated Total Reflection)

This is a measurement method that utilizes the light penetration that occurs when infrared light is totally reflected inside the prism. (See Figure B.1.) Highly sensitive measurement is possible.

The size of the prism limits the measurement location.



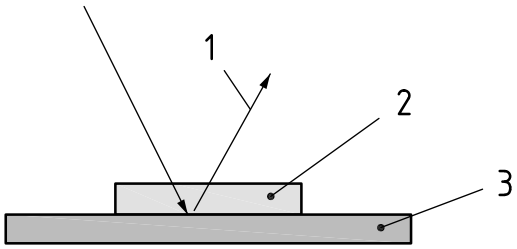
Key

1 prism
2 residue
3 substrate

Figure B.1 — Schematic diagram of ATR

B.1.2 Reflection

Basic measurement method of FT-IR. Although the sensitivity is inferior to that of ATR. It is possible to measure even in a narrow area such as a electrodes or High-density mounted part. (See Figure B.2.)



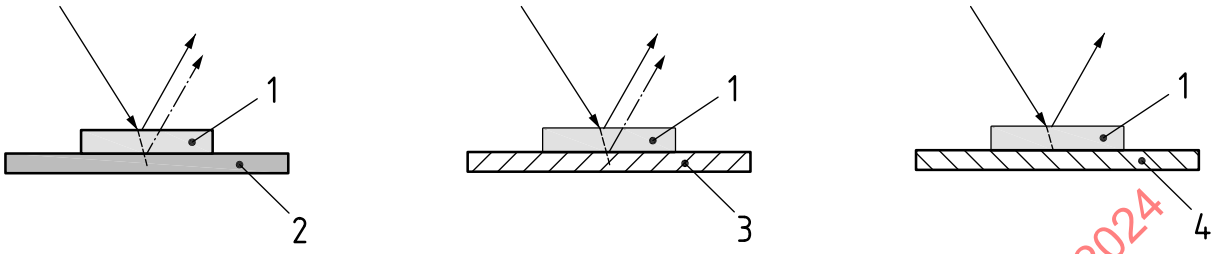
Key

1 IR radiation
2 residue
3 substrate

Figure B.2 — Schematic diagram of FT-TR (reflection)

B.2 Differences depending on the base material

In the case of a substrate such as FR4 or ceramic, the spectrum of substrate material and the spectrum of the residue are mixed. It is necessary to obtain a spectrum of flux residues and make a comparative evaluation. As a general rule, metals do not absorb infrared light, so it is possible to discriminate and measure only the residue. (See Figure B.3.)



a) **b)** **c)**

Key

1 residue
2 substrate (FR4) (substrate spectrum) C-H O-H C=O
3 ceramic (substrate spectrum) Si-O
4 metal (substrate spectrum) not detected, spectrum is residue only

Figure B.3 — Differences depending on the base material in the FT-IR measurement

B.3 Spectrum

Refer to this table for the material frequencies. (See Figure B.4.)