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Test methods for soldering fluxes

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry, through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Welding Engineering Society (JWES) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS Z 3197:1999** is replaced with this Standard.

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Attention is drawn to the possibility that some parts of this Standard may conflict with patent rights, applications for a patent after opening to the public or utility model rights. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying any of such patent rights, applications for a patent after opening to the public or utility model rights.

Test methods for soldering fluxes

Introduction

This Japanese Industrial Standard has been prepared based on the first editions of **ISO 9454-1** published in 1990, **ISO 9455-1** published in 1990, **ISO 9455-3** published in 1992, **ISO 9455-5** published in 1992, **ISO 9455-6** published in 1995, **ISO 9455-10** published in 1998, **ISO 9455-13** published in 1996, **ISO 9455-14** published in 1991, **ISO 9455-15** published in 1996, **ISO 9455-16** published in 1998, **ISO 9455-17** published in 2002, **ISO 12224-2** published in 1997, **IEC 61189-5** published in 2006, **IEC 61189-6** published in 2006 and **IEC 61190-1-1** published in 2002, but with some modifications of the technical contents in the said International Standards, which specify fluxes intended for all soldering purposes, whereas this **JIS** specifies fluxes intended for soldering for connection of wiring and parts in electric and electronic apparatuses and communication devices.

The portions with continuous sidelines or dotted underlines are the matters in which the contents of the corresponding International Standards have been modified. A list of modifications with the explanations is given in Annex JA.

1 Scope

This Standard specifies the test methods for soldering fluxes (hereafter referred to as “fluxes”) mainly intended for connection of wiring and parts in electric and electronic apparatuses and communication devices.

NOTE : The International Standards corresponding to this Standard and the symbol of degree of correspondence are as follows.

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 9455-3:1992 *Soft soldering fluxes—Test methods—Part 3: Determination of acid value, potentiometric and visual titration methods*

ISO 9455-5:1992 *Soft soldering fluxes—Test methods—Part 5: Copper mirror test*

ISO 9455-6:1995 *Soft soldering fluxes—Test methods—Part 6: Determination and detection of halide (excluding fluoride) content*

ISO 9455-10:1998 *Soft soldering fluxes—Test methods—Part 10: Flux efficacy tests, solder spread method*

ISO 9455-13:1996 *Soft soldering fluxes—Test methods—Part 13: Determination of flux spattering*

ISO 9455-14:1991 *Soft soldering fluxes—Test methods—Part 14: Assessment of tackiness of flux residues*

ISO 9455-15:1996 *Soft soldering fluxes—Test methods—Part 15: Copper corrosion test*

ISO 9455-16:1998 *Soft soldering fluxes—Test methods—Part 16: Flux efficacy tests, wetting balance method*

ISO 9455-17:2002 *Soft soldering fluxes—Test methods—Part 17: Surface insulation resistance comb test and electrochemical migration test of flux residues*

ISO 12224-2:1997 *Flux cored solder wire—Specification and test methods—Part 2: Determination of flux content*

IEC 61189-5:2006 *Test methods for electrical materials, interconnection structures and assemblies—Part 5: Test methods for printed board assemblies*

IEC 61189-6:2006 *Test methods for electrical materials, interconnection structures and assemblies—Part 6: Test methods for materials used in manufacturing electronic assemblies*

IEC 61190-1-1:2002 *Attachment materials for electronic assembly—Part 1-1: Requirements for soldering fluxes for high-quality interconnections in electronics assembly* (Overall evaluation: MOD)

The symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

WARNING Persons carrying out tests based on this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS B 7502 *Micrometer callipers*

JIS B 7525 *Density hydrometers*

JIS C 6480 *General rules of copper-clad laminates for printed wiring boards*

JIS H 3100 *Copper and copper alloy sheets, plates and strips*

JIS H 3260 *Copper and copper alloy wires*

JIS K 2265-1 *Determination of flash point—Part 1: Tag closed cup method*

JIS K 2265-2 *Determination of flash point—Part 2: Rapid equilibrium closed cup method*

JIS K 2265-3 *Determination of flash point—Part 3: Pensky-Martens closed cup method*

JIS K 2265-4 *Determination of flash points—Part 4: Cleveland open cup method*

JIS K 3351 *Glycerines for industrial use*

JIS K 5600-2-1 *Testing methods for paints—Part 2: Characteristics and stability of paints—Section 1: Colour number (Gardner method)*

JIS K 5902 *Colophonium*

JIS K 8034 *Acetone (Reagent)*

JIS K 8057 *Alizarin red S (Reagent)*

JIS K 8101 *Ethanol (99.5) (Reagent)*

JIS K 8150 *Sodium chloride (Reagent)*

JIS K 8180 *Hydrochloric acid (Reagent)*

JIS K 8252 *Ammonium peroxodisulfate (Reagent)*

JIS K 8312 *Potassium chromate (Reagent)*

JIS K 8322 *Chloroform (Reagent)*

JIS K 8541 *Nitric acid (Reagent)*

JIS K 8550 *Silver nitrate (Reagent)*

JIS K 8723 *Nitrobenzene (Reagent)*

JIS K 8839 *2-Propanol (Reagent)*

JIS K 8891 *Methanol (Reagent)*

JIS K 8951 *Sulfuric acid (Reagent)*

JIS K 8982 *Ammonium iron (III) sulfate 12-water (Reagent)*

JIS K 9000 *Ammonium thiocyanate (Reagent)*

JIS P 3801 *Filter paper (for chemical analysis)*

JIS R 3503 *Glass apparatus for chemical analysis*

JIS R 3505 *Volumetric glassware*

JIS R 3703 *Slide glasses for microscope*

JIS R 6252 *Abrasive papers*

JIS Z 3001 (standard series) *Welding and applied processes—Vocabulary*

JIS Z 3282 *Soft solders—Chemical compositions and forms*

JIS Z 3283 *Resin flux cored solders*

JIS Z 3284 *Solder paste*

JIS Z 8401 *Guide to the rounding of numbers*

JIS Z 8703 *Standard atmospheric conditions for testing*

JIS Z 8803 *Methods for viscosity measurement of liquid*

3 Terms and definitions

For the purposes of this Standard, the terms and definitions given in **JIS Z 3001** standard series and the following apply.

3.1 resin type flux

flux of which the base compound is resin such as rosin (colophony)

NOTE : Rosin includes modified rosin. Organic solvent is used as the solvent.

3.2 organic type flux

flux of which the base compound is organic acid and organic activator

NOTE : Water-based or organic solvents are used as the solvent.

3.3 inorganic type flux

flux of which the base compound is inorganic acids, inorganic salts and inorganic alkalis

NOTE : As a solvent, water-soluble materials such as glycerine, polyethylene glycol and polyoxyethylene glycol, or non-water-soluble materials such as wax and Vaseline are used. Fluxes of this type have strong active power by the base compound, but their residues have strong corrosiveness.

3.4 halide content

total content of chlorine (Cl), bromine (Br), and iodine (I) contained in flux converted to chlorine (Cl) content

NOTE : This is contained in activator which is mixed in or added to the flux by a chemical procedure for improving the solderability.

3.5 purified water

distilled water and ion exchanged water with specific resistivity (electric resistivity) of 5 000 Ωm or over at 20 °C

3.6 solder paste

paste flux mixed with solder powder in such a way that solder powder is dispersed, to which components are added for preventing separation of flux and solder powder, and for providing proper viscosity, thixotropy and printability

4 Types, components and forms of fluxes

The types, components and forms of fluxes specified in this Standard are shown in table 1.

Table 1 Types, components and forms of fluxes

Type	Components			Form
	Basis component	Activation	Fluoride	
1 Resin	1 Rosin ^{a)}	1 No activator added	F (contained)	A Liquid
	2 Synthetic resin ^{b)}	2 Halide activated ^{c)}	N (not contained)	B Solid
2 Organic	1 Water-soluble	3 Non-halide activated		C Paste
	2 Non-water-soluble			
3 Inorganic	1 Inorganic salts	1 With ammonium chloride (ammonium halide)		
		2 Without ammonium halide		
	2 Inorganic acids	1 Phosphoric acid		
		2 Other acids		
	3 Inorganic alkalis	1 Amines and/or ammonium		
Notes ^{a)} Modified rosin is included.				
^{b)} Resins such as organic acid ester of pentaerythritol, phenol or cresol derivative, styrene-maleic acid resin and acrylic resin. Many of them are used for flux with trace amount of residue.				
^{c)} Other activators may be present.				

5 Classification of test methods and types of test methods

Test methods are classified by the type, purpose of test, sampling method according to the form of flux as shown in table 2.

Table 2 Classification, types, purposes of test methods and sampling methods

Test				Suitable sampling method and applicable tests for respective forms of flux						
				Resin flux cored solder		Liquid flux	Solid flux		Solder paste	
				Product itself 7.4.2 a)	2-Propanol solution of 25 % mass fraction 7.4.2 b)	Product itself 7.4.4	Product itself 7.4.5 a)	2-Propanol solution of 25 % mass fraction 7.4.5 b)	Product itself 7.4.3 a)	Paste flux 7.4.3 b)
Classification of test method	Type of test method		Purpose of test	Test subclause						
1 Tests of contents	1 Test of resistivity (electric resistivity) of aqueous solution		In order to examine the quantity of ionic substances in flux that affect the corrosion characteristics, the resistivity of extracted solution is measured.	8.1.1	—	○	○	—	○	—
	2 Test of flux content		The flux content that affects the solderability of the resin flux cored solder and the solder paste is measured.	8.1.2	○	—	—	—	○	—
	3 Test of solid content		In order to examine solderability of liquid flux and the amount of flux residues, the solid content is measured.	8.1.3	—	—	○	—	—	—
	4 Activator evaluation test	1 Organic	1 Acid value (resin and organic)	8.1.4.1.1	○	—	○	○	—	○
			2 Acid value (water-soluble)	8.1.4.1.2	○	—	○	○	—	○

Table 2 (continued)

Test						Suitable sampling method and applicable tests for respective forms of flux						
						Resin flux cored solder		Liquid flux	Solid flux		Solder paste	
						Product itself 7.4.2 a)	2-Propanol solution of 25 % mass fraction 7.4.2 b)	Product itself 7.4.4	Product itself 7.4.5 a)	2-Propanol solution of 25 % mass fraction 7.4.5 b)	Product itself 7.4.3 a)	Paste flux 7.4.3 b)
Classifica- tion of test method	Type of test method			Purpose of test	Test subclause							
1 Tests of contents (concluded)	4 Activator evalua- tion test	2 Halide	1 Potentiometric titration method	The halide content in the flux that affects solderability and corrosion characteristics is examined.	8.1.4.2.1	○	—	○	○	—	—	○
			2 Volhard method (total halide in water- soluble flux)		8.1.4.2.2	○	—	○	○	—	—	○
			3 Silver chromate paper test (quality of halide)	The presence of halide in the flux that affects solderability and corrosion characteristics is examined.	8.1.4.2.3	—	○	○	—	○	—	○
			4 Colour reaction method (fluoride content test)	The presence of fluoride in the flux that affects solderability and corrosion characteristics is examined.	8.1.4.2.4	—	○	○	—	○	—	○
2 Physical character- istics tests	1 Viscosity test			The viscosity that affects quality and workability of liquid flux is measured.	8.2.1	—	—	○	—	—	—	—
	2 Specific gravity test			The specific gravity that affects quality and workability of liquid flux is measured.	8.2.2	—	—	○	—	—	—	—
	3 Colour number test			The colour tone of flux that affects quality and finishing of liquid flux is evaluated in comparison with the colour standard.	8.2.3	—	—	○	—	—	—	—
	4 Flash point test			In order to examine the flam- mability of liquid flux, the flash point is measured.	8.2.4	—	—	○	—	—	—	—

Table 2 (continued)

Test				Suitable sampling method and applicable tests for respective forms of flux						
				Resin flux cored solder		Liquid flux	Solid flux		Solder paste	
				Product itself 7.4.2 a)	2-Propanol solution of 25 % mass fraction 7.4.2 b)	Product itself 7.4.4	Product itself 7.4.5 a)	2-Propanol solution of 25 % mass fraction 7.4.5 b)	Product itself 7.4.3 a)	Paste flux 7.4.3 b)
3 Soldering behaviour tests	1 Flux effect	1 Solder spreading method	In order to examine solderability of liquid flux, solid flux, resin flux cored solder and solder paste, the effect of flux is evaluated by the solder spreading characteristics.	8.3.1.1	○	—	○	—	○	○
		2 Wetting balance method	In order to examine solderability of liquid flux, solid flux, resin flux cored solder and solder paste, the wetting characteristics are evaluated by wetting balance.	8.3.1.2	—	○	○	—	○	○
	2 Flux spitting test		The flux spatter of resin flux cored solder at the time of soldering that affects the finishing quality after soldering is examined.	8.3.2	○	—	—	—	—	—
	3 Foaming test		The foaming characteristics that affect workability of liquid flux is examined.	8.3.3	—	—	○	—	—	—

Table 2 (continued)

Test				Suitable sampling method and applicable tests for respective forms of flux						
				Resin flux cored solder		Liquid flux	Solid flux		Solder paste	
				Product itself 7.4.2 a)	2-Propanol solution of 25 % mass fraction 7.4.2 b)	Product itself 7.4.4	Product itself 7.4.5 a)	2-Propanol solution of 25 % mass fraction 7.4.5 b)	Product itself 7.4.3 a)	Paste flux 7.4.3 b)
4 Corrosion tests	1 Copper plate corrosion test	In order to examine the corrosion characteristics of flux residue after soldering, it is evaluated by discolouration of flux residue on a copper plate or presence of corrosion product.	8.4.1	○	—	○	○	—	○	○
	2 Copper mirror corrosion test	In order to examine the corrosion characteristics of flux, the degree of corrosion of copper on the copper mirror is compared with the standard rosin solution (WW rosin) for evaluation.	8.4.2	—	○	○	—	○	—	○
5 Tests of characteristics after soldering	1 Dryness test	The dryness of flux residue that affects the finishing quality after soldering is examined.	8.5.1	○	—	○	○	—	○	—
	2 Ionic residue test	In order to examine the reliability of printed circuit board after soldering, the quantity of ionic residue in flux residue is measured, and the obtained quantity is displayed after conversion to sodium chloride.	8.5.2	—	○	○	—	○	○	—
	3 Insulation resistance test	In order to examine the reliability of flux, the insulation resistance of flux is measured under high temperature and high humidity by using comb-patterned printed circuit boards.	8.5.3	—	○	○	—	○	○	○

Table 2 (concluded)

Test				Suitable sampling method and applicable tests for respective forms of flux						
				Resin flux cored solder		Liquid flux	Solid flux		Solder paste	
				Product itself 7.4.2 a)	2-Propanol solution of 25 % mass fraction 7.4.2 b)	Product itself 7.4.4	Product itself 7.4.5 a)	2-Propanol solution of 25 % mass fraction 7.4.5 b)	Product itself 7.4.3 a)	Paste flux 7.4.3 b)
5 Tests of characteristics after soldering (concluded)	4 Voltage-applied moisture resistance test—Migration test	In order to examine the occurrence of electrochemical migration that affects the reliability of flux, flux applied on comb-patterned printed circuit boards is tested by leaving the circuit boards in a humidity chamber under high temperature and high humidity conditions for a specified time with bias voltage applied, and removing from the humidity chamber to check the presence of dendrites.	8.5.4	—	○	○	—	○	○	○
NOTE: ○ indicates that the test is applicable.										

6 General conditions of tests

6.1 Standard conditions of tests

The test shall be, unless otherwise specified, conducted in the standard atmospheric condition of temperature 20 °C Class 15 (± 15 °C) and relative humidity 65 % Class 20 (± 20 %) as specified in **JIS Z 8703**. In cases of any doubt arising as to judgement of measured values obtained under the said condition, the test shall be performed at the atmospheric condition of temperature 20 °C Class 2 (± 2 °C) and relative humidity of 65 % Class 5 (± 5 %) specified in **JIS Z 8703**.

6.2 Test pieces

Tests that require test pieces shall be conducted by preparing three test pieces each, unless otherwise specified.

6.3 Rounding of numbers

Unless otherwise specified, rounding of numbers shall be in accordance with Rule A given in **JIS Z 8401**.

7 Sample

7.1 Test materials

The test materials shall be resin flux cored solder specified in **JIS Z 3283**, solder paste specified in **JIS Z 3284**, liquid flux and solid flux.

7.2 Reagents

The reagents to be used shall be as follows.

- a) **Acetone**, specified in **JIS K 8034**.
- b) **2-Propanol**, specified in **JIS K 8839** (isopropyl alcohol).

7.3 Apparatus and instruments

The apparatus and instruments to be used shall be as follows.

- a) **Soxhlet extractor**
- b) **Hot plate**, adjustable to temperature $150\text{ °C} \pm 5\text{ °C}$.
- c) **Constant temperature bath**, adjustable to temperature $100\text{ °C} \pm 5\text{ °C}$.

7.4 Sampling

7.4.1 General

For sampling from test materials, apply the method for the relevant form of flux as given in table 2. The procedures for respective sampling methods are detailed in the following.

7.4.2 Resin flux cored solder

Sampling in the case of resin flux cored solder shall be as follows.

- a) **Product itself** Use the product itself as a sample, after washing its surface with acetone, rinsing with purified water and then with 2-propanol, and drying.
- b) **2-Propanol solution of 25 % mass fraction** Cut off about 150 g of resin flux cored solder and crush both ends and crimp them. Clean the surface with a cloth dampened with acetone. Place this in a beaker, pour sufficient purified water, cover the sample and boil for about 5 min to 6 min. Remove the sample from the beaker, rinse with acetone and dry. Cut the sample piece into 2 mm to 3 mm lengths using a razor or scissors with care not to contaminate the surface of soldering and crush the cut ends. Dispose the crimped ends. Put the pieces into the clean extraction pipe of Soxhlet extractor and extract with 2-propanol or other appropriate solvent until the recovered condensate becomes transparent. Pour the flux extract into a beaker and evaporate and dry in the constant temperature bath at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. Dilute this with the solvent used for extraction until the solid content (25 ± 0.5 mass fraction) % is obtained, to prepare the 2-propanol solution of 25 % mass fraction.

NOTE : In extraction of flux or preparation of 2-propanol solution of 25 % mass fraction, if the flux cannot be dissolved in 2-propanol, other appropriate solvent miscible with purified water shall be used.

7.4.3 Solder paste

Sampling in the case of solder paste shall be as follows.

- a) **Product itself** Use the product itself as a sample, after stirring several times to make it homogenous.
- b) **Paste fluxes** If the user of this Standard requires assessing only the flux in the solder paste, the same flux as that in the solder paste shall be obtained from the solder paste manufacturer. If this is impossible, the following procedures shall be performed to extract the flux from the solder paste. Weigh out $100\text{ g} \pm 2\text{ g}$ of solder paste to the nearest 0.1 g in a 300-ml beaker 1 of which the tare mass has been weighed. Add 100 ml of 2-propanol or other suitable solvent to this, and boil on a hot plate at $150\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for about 3 min while stirring with a glass rod so as not to bump. After leaving to stand for about 1 min, filter the warm supernatant solution through Grade 1 filter paper specified in **JIS P 3801**, while taking care that soldering powder is not mixed in, into a 300-ml beaker 2 of which the tare mass has been weighed. At this time, it is not necessary to filter the whole solution, and the filtered solution need not be completely transparent. Repeat this extraction procedure several times using 30 ml of 2-propanol or other appropriate solvent until colourless extracted solution is obtained. Mix the obtained extracted solution with the solution in beaker 2. Heat in the constant temperature bath maintained at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 20 min or longer until the residue in beaker 1 is completely dry, and evaporate the remaining solvent in a beaker containing solder powder. Weigh beaker 1, and repeat evaporation of solvent until the mass is constant (0.1 g or less). Calculate the mass of extracted flux to one decimal place by the following formula (1):

$$F = W_1 - (W_3 - W_2) \dots\dots\dots (1)$$

where, F : mass of extracted flux (g)

W_1 : sampling mass of solder paste (g)

W_2 : mass of beaker 1 (g)

W_3 : mass of beaker 1 containing solder powder (g)

Then, heat beaker 2 containing the extract on a hot plate to evaporate the solvent until the flux mass (F) in formula (1) is obtained, and take the remaining non-volatile residue as paste flux in solder paste.

NOTE : In extraction of flux or preparation of 2-propanol solution of 25 % mass fraction, if the flux cannot be dissolved in 2-propanol, other appropriate solvent miscible with purified water shall be used.

7.4.4 Liquid flux

In the case of liquid flux, use the product itself as a sample.

7.4.5 Solid flux

Sampling in the case of solid flux shall be as follows.

- a) **Product itself** Use the product itself as a sample.
- b) **2-Propanol solution of 25 % mass fraction** Using 2-propanol or other appropriate solvent, prepare a 2-propanol solution containing 25 % of solid part.

NOTE : In extraction of flux or preparation of 2-propanol solution, if the flux cannot be dissolved in 2-propanol, other appropriate solvent miscible with purified water shall be used.

8 Test methods

8.1 Tests of contents

8.1.1 Test of resistivity (electric resistivity) of aqueous solution

This test is to measure the resistivity of extracted water from a solution of a certain density in flux in an appropriate solvent to determine the mass of water-soluble conductive materials in resin or organic flux (see the "Type" column in table 1) and as follows.

- a) **Outline of test** The 2-propanol solution of 25 % mass fraction is added to purified water, and boiled. The resistivity of this solution is measured at 20 °C.
- b) **Reagent** Purified water shall be used as reagent.
- c) **Apparatus and instruments** The apparatus and instruments to be used shall be as follows.
 - 1) **Hot plate**
 - 2) **Water bath**
 - 3) **Microsyringe**
 - 4) **Electric conductivity meter**, with conductive cell of about 0.1 cell constant.
 - 5) **Ordinary laboratory apparatus** All glass instruments to be used in this test shall be those specified in **JIS R 3503** or equivalent instruments. They shall be exclusively used for this test and cleaned before storage.

d) **Test procedure** The test procedure shall be as follows.

1) **General** Wash all glass instruments thoroughly with cleaning solution prior to use. Rinse several times with running water, and then at least five times with purified water. Pour 50 ml of a solution in a 50-ml tall beaker (hereafter referred to as “tall beaker”) and check that, when a conductive cell is immersed, its electrode is sufficiently dipped in the solution.

2) **Measurement** Wash thoroughly five watch glasses and five tall beakers.

Fill each of the five tall beakers with purified water up to the 50-ml mark, and immerse them in a water tank maintained at $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Upon reaching thermal equilibrium, measure the resistivity of purified water in each of the tall beakers at this temperature using the electric conductivity meter. All the measured values of resistivity at this time shall exceed $5\,000\text{ }\Omega\text{m}$. If any of the measurements is below this value, repeat the above procedure. In three beakers out of the five, setting aside the other two, add $0.100\text{ ml} \pm 0.005\text{ ml}$ of 2-propanol solution of 25 % mass fraction by means of microsyringe or the like.

For sampling paste flux from solder paste, after checking the cleanliness of the tall beakers as described above, discard the purified water in the beakers, weigh out $0.100\text{ g} \pm 0.005\text{ g}$ of paste flux and add purified water.

Cover all of the five tall beakers with watch glasses and heat on hot plate and boil for 60 s to obtain extracted water. Then, after cooling with running water, place in a water bath maintained at $20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. Upon reaching thermal equilibrium, measure the resistivity of purified water in five beakers and that of the flux sample solution.

NOTE : After washing, all the tall beakers should be covered with watch glasses in order to prevent contamination until the time of use.

- 2.1) Wash thoroughly the conductive cell in purified water, and immerse one sample in the flux sample solution and read the resistivity.
- 2.2) Wash thoroughly the conductive cell in purified water, immerse in purified water and read the resistivity.
- 2.3) Wash thoroughly the conductive cell in purified water, and immerse in the next flux sample solution and read the resistivity.
- 2.4) Wash thoroughly the conductive cell in purified water, and continue measurement of the resistivity of the remaining purified water and flux sample solution.

3) **Requirements** The resistivity of each purified water shall exceed $5\,000\text{ }\Omega\text{m}$. If this value is below $5\,000\text{ }\Omega\text{m}$, it indicates that the purified water is contaminated with water-soluble ionized substance, in which case, the entire test shall be repeated. The resistivity of the flux sample solution shall be expressed with the average of three results obtained with three test samples.

8.1.2 Test of flux content

The flux content test is to measure the flux content in resin flux cored solder and solder paste (see the “Form” column in table 1) and as follows.

- a) **Outline of test** The soft solder of resin flux cored solder or solder paste, whose masses have been measured, is fused. The flux portion is separated and its mass is measured. The flux content is calculated and indicated by mass fraction with respect to the resin flux cored solder or solder paste and expressed in %.
- b) **Reagents** The reagents to be used shall be as follows.
- 1) **Industrial glycerin**, specified in **JIS K 3351**.
 - 2) **2-Propanol**, specified in **JIS K 8839**.
- c) **Implements** The following implements shall be used.
- 1) **Hot plate**, capable of use at a temperature 50 °C higher than the liquidus-line temperature of the solder used for the test.
 - 2) **Analytical balance**, with reciprocal sensitivity of 0.000 1 g.
- d) **Test procedure** The test procedure shall be as follows.

- 1) **Resin flux cored solder** For resin flux cored solder, after cleaning the surface of flux with a cloth dampened with 2-propanol, weigh out about 30 g ± 2 g of the sample to the nearest 0.001 g by means of a chemical balance into a 100-ml beaker. Add about 20 ml of glycerin and fuse the solder by heating with a hot plate set at a temperature 50 °C above the liquidus-line temperature of the solder. When solder and flux are separated completely, leave the beaker to cool until the solder has solidified.

Then, take out the solidified solder from the beaker and wash it with water, and after immersing it in 2-propanol for about 5 min, wash and dry it at room temperature. Weigh the mass of it to the nearest 0.001 g by means of a chemical balance and calculate the flux content according to formula (2) and round the result to one decimal place.

$$F = \frac{W_1 - W_2}{W_1} \times 100 \dots\dots\dots (2)$$

where, F : the flux content (%)
 W_1 : the mass of sample taken (g)
 W_2 : the mass of solidified solder (g)

- 2) **Solder paste** For solder paste, weigh out 30 g ± 1 g of sample to the nearest 0.001 g, from evenly mixed solder paste, into a 100-ml beaker. Then add about 20 ml of glycerin and fuse the solder by heating on a hot plate set at a temperature 50 °C above the liquidus-line temperature of the solder. When solder and flux are separated completely, leave the beaker to cool until the solder has solidified.

Then, take out the solidified solder from the beaker and wash it with water, and after immersing it in 2-propanol for about 5 min, wash and dry it at room temperature. Weigh its mass to the nearest 0.001 g by means of an analytical balance, and calculate the flux content according to formula (3) and round the result to one decimal place.

$$F = \frac{W_1 - W_2}{W_1} \times 100 \dots\dots\dots (3)$$

where, F : the flux content (%)
 W_1 : the mass of sample taken (g)
 W_2 : the mass of solidified solder (g)

8.1.3 Tests of solid content

This test is to measure the solid content of liquid flux (see the “Form” column in table 1) and as follows.

- a) **Outline of test** Flux whose mass has been weighed is heated in a drying oven. After the flux is allowed to cool, its mass is re-weighed to determine the solid content.
- b) **Apparatus and instruments** The apparatus and instruments to be used shall be as follows.

- 1) **Flat weighing bottle with ground-in stopper**, having an inner diameter of 40 mm and depth of 40 mm.
- 2) **Analytical balance**, with reciprocal sensitivity of 0.000 1 g.
- 3) **Drying oven**, capable of maintaining at a temperature of $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
- 4) **Desiccator**

- c) **Test procedure** The test procedure shall be as follows.

- 1) **For organic solvent flux (boiling point below $100\text{ }^{\circ}\text{C}$)** Dry a flat weighing bottle with ground-in stopper for 1 h in a drying oven maintained at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, remove it from the drying oven, and allow it to cool down to room temperature in a desiccator. After cooling, weigh the mass of the bottle to the nearest 0.001 g by means of the analytical balance (W). Weigh $1.0\text{ g} \pm 0.1\text{ g}$ of flux to the nearest 0.001 g by means of the analytical balance (W_1). At this time, take measures to prevent loss of volatile matter and dry for 1 h in the drying oven maintained at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. Take out from the drying oven, and allow to cool to room temperature in the desiccator. After cooling, weigh the mass to the nearest 0.001 g by means of the analytical balance (W_2).

In the case of organic flux of which the boiling point is $100\text{ }^{\circ}\text{C}$ or higher, the test shall be carried out as agreed upon between the purchaser and the supplier.

- 2) **For water-soluble flux** Perform the test procedure in 1) using the same instruments as in 1), except that flux shall be dried at $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 3 h.

- d) **Calculation of solid content** Express the solid content in mass fraction (%), and calculate from formula (4). Round the result to one decimal place.

$$S = \frac{W_2 - W}{W_1} \times 100 \dots\dots\dots (4)$$

where, S : solid content (%)

W : mass of flat weighing bottle with ground-in stopper (g)

W_1 : mass of sample taken (g)

W_2 : mass of flat weighing bottle with ground-in stopper and residue after drying (g)

Express the solid content with the average of measurements of three samples.

8.1.4 Activator evaluation test

8.1.4.1 Organic activator content test

8.1.4.1.1 Acid value test (resin and organic)

This test is to evaluate the acid content of resin flux and organic flux (see the "Type" column in table 1) by acid value and as follows.

- a) **Outline of test** The flux component of the test material is dissolved in ethanol. This solution is titrated with N/10 potassium hydroxide (ethanol solution) to obtain the acid value.
- b) **Reagents** The reagents to be used shall be as follows.

- 1) **General** Use only purified water and reagents of recognized analytical quality of Grade 1 or guaranteed reagents.
- 2) **N/10 potassium hydroxide (ethanol solution)** Commercially available N/10 potassium hydroxide, or commercially available potassium hydroxide concentrated solution diluted with ethanol specified in **JIS K 8101**. The factor is calculated as follows.

Titrate 25 ml of N/10 potassium hydroxide (ethanol solution) with N/10 hydrochloric acid standard solution with known factor, using phenolphthalein as an indicator, and calculate the factor by formula (5). Round the result to four decimal places.

$$f_k = \frac{x - f_H}{25} \dots\dots\dots (5)$$

where, f_k : factor of N/10 potassium hydroxide

x : titre of N/10 hydrochloric acid standard solution (ml)

f_H : factor of N/10 hydrochloric acid standard solution

- 3) **Phenolphthalein indicator** Dissolve 1 g of phenolphthalein in 50 ml of methanol specified in **JIS K 8891** and dilute to 100 ml with methanol.
- 4) **Ethanol** Neutralize ethanol specified in **JIS K 8101** with N/10 potassium hydroxide (ethanol solution) using phenolphthalein as an indicator.
- c) **Apparatus and instruments** The apparatus and instruments to be used shall be as follows.
 - 1) **General analysis instruments** The glass instruments used for this test shall be those specified in **JIS R 3503** or equivalent instruments.

- 2) **Drying oven**, capable of setting and maintaining the temperature at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
- 3) **Magnetic stirrer**, with variable speed drive.
- 4) **Analytical balance**, with reciprocal sensitivity of 0.000 g .
- 5) **Volumetric pipette**, specified in **JIS R 3505**.

d) **Test procedure** The test procedure shall be as follows.

- 1) **Resin flux cored solder** The test procedure for resin flux cored solder is as follows.

- 1.1) Wash the surface of resin flux cored solder in accordance with **7.4.2 a)**, and from this solder, weigh out the amount which will produce about 1 g of flux, and cut into chips of 2 mm to 3 mm lengths. Measure the total mass of these solder chips to the nearest 0.001 g by means of an analytical balance (W_1).
- 1.2) Put all of these solder chips in a suitable flask or beaker and add 100 ml of ethanol. Covering it with a watch glass, heat it gently to extract flux. After extraction, pour the supernatant liquid into another beaker to take it as the extracted solution. Wash the solder chips two or three times with 25 ml of ethanol and add the washings to the extracted solution to take it as the test solution.
- 1.3) Dry the solder chips after extraction of flux for 1 h in a drying oven maintained at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. After cooling, weigh the total mass of the solder chips to the nearest 0.001 g by means of an analytical balance (W_2). Calculate the mass of flux according to formula (6), and round the result to one decimal place.

$$B = W_1 - W_2 \dots\dots\dots (6)$$

where, B : mass of flux (g)

W_1 : mass of resin flux cored solder (g)

W_2 : total mass of solder chips after extraction of flux (g)

- 1.4) Add three drops of phenolphthalein as an indicator to the test solution, and put the beaker on a magnetic stirrer. Stir it vigorously while taking care not to allow spattering. Titrate it with $N/10$ potassium hydroxide (ethanol solution). The end point is reached when the light red (pink) colour first persists in the solution for 30 s . The volume of $N/10$ potassium hydroxide (ethanol solution) added up to the end point is taken as V_k .

- 2) **Solid flux** The test procedure for solid flux is as follows.

- 2.1) Weigh about 1 g of sample flux to the nearest 0.001 g by means of an analytical balance. Put this flux in a suitable flask or a beaker and add 100 ml of ethanol.
- 2.2) Perform titration according to **1.4**).

- 3) **Liquid flux** The test procedure for liquid flux is as follows.

- 3.1) Take a mass of about 15 g of the sample flux in a suitable flask or a beaker, weigh the mass to the nearest 0.01 g by means of an analytical balance (m_1) and add 100 ml of ethanol.

If the specific gravity of the liquid flux is known, the following procedure may be used.

Measure 10 ml to 20 ml of sample flux using a volumetric pipette specified in **JIS R 3505** into a suitable flask or a beaker. Obtain the mass (m_1) as the product of the volume and specific gravity.

3.2) Perform titration according to **1.4**).

4) **Solder paste (when flux is available)** When flux of solder paste is available, the test procedure is as follows.

4.1) Weigh about 3 g of paste flux specified in **7.4.3 b)** to the nearest 0.001 g by means of an analytical balance and put it in a suitable flask or a beaker. Add 100 ml of ethanol, and when paste flux is completely dissolved, take this as the test solution.

4.2) Perform titration according to **1.4**).

5) **Solder paste (when flux is not available)** When flux of solder paste is not available, the test procedure shall be as follows.

5.1) Weigh out solder paste to produce about 3 g of flux and weigh the mass by means of an analytical balance to the nearest 0.001 g.

5.2) Put this in a suitable beaker, add 100 ml of ethanol, stir it at the ordinary temperature and extract flux.

5.3) After extraction of flux, pour the supernatant gently into a beaker through a coarse filter paper while taking care not to let the powder escape. Wash the beaker and the filter paper two or three times with 10 ml of heated ethanol, and put the washings in the beaker for test.

5.4) Dry the solder paste from which flux has been extracted in a drying oven maintained at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 1 h. After cooling, weigh the mass to the nearest 0.001 g by means of the analytical balance.

5.5) The mass of flux shall be the difference between the mass first weighed [see **5.1**)] and the mass after extraction of flux [see **5.4**)].

5.6) Perform titration according to **1.4**).

e) **Calculation of acid value** Calculate the acid value as follows.

Express the acid content in flux by the acid value (amount in mg of potassium hydroxide required to neutralize 1 g of flux) and calculate it from formula (7). Round the result to one decimal place.

$$A_{V1} = \frac{56.11 \times V_K \times M_K \times f_K}{m_1} \dots\dots\dots (7)$$

where, A_{V1} : acid value of flux

V_K : volume of potassium hydroxide (ethanol solution) (ml)

M_K : concentration of potassium hydroxide (ethanol solution) (mol/L)

f_k : factor of potassium hydroxide (ethanol solution)

m_1 : mass of sample flux (g)

Express the acid value with the average of measurements of three samples.

8.1.4.1.2 Acid value test (water-soluble)

This test is to evaluate the content of water-soluble acid in resin flux and organic flux (see the "Type" column in table 1) and as follows.

- a) **Outline of test** Chloroform is added to a test material to dissolve flux, and then water-soluble material is extracted with purified water. This extracted solution is titrated with N/10 potassium hydroxide (ethanol solution) to determine the acid value.
- b) **Reagents** The reagents to be used shall be as follows.

- 1) **General** Use only purified water and reagents of recognized analytical quality of Grade 1 or guaranteed reagents.
- 2) **N/10 potassium hydroxide (ethanol solution)** Commercially available N/10 potassium hydroxide, or its commercially available concentrated solution diluted with ethanol specified in **JIS K 8101**. The factor is calculated as follows.

Titrate 25 ml of N/10 potassium hydroxide (ethanol solution) with N/10 hydrochloric acid standard solution with known factor, using phenolphthalein as an indicator. Calculate the factor by formula (8), and round the result to four decimal places.

$$f_k = \frac{x \times f_H}{25} \dots\dots\dots (8)$$

where, x : titre of N/10 hydrochloric acid standard solution (ml)

f_H : factor of N/10 hydrochloric acid standard solution

f_k : factor of N/10 potassium hydroxide

- 3) **Phenolphthalein indicator** Add 1 g of phenolphthalein to 50 ml of methanol specified in **JIS K 8891** and dilute with methanol to make 100 ml.
 - 4) **Chloroform**, specified in **JIS K 8322**.
- c) **Apparatus and instruments** The apparatus and instruments to be used shall be as follows.
- 1) **General** All the glass instruments used for this test shall be in accordance with **JIS R 3503** or equivalent instruments. The glass instruments shall be exclusively used for this test, and cleaned before storage.
 - 2) **Drying oven**, of air circulation type, capable of setting and maintaining the temperature at $100\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.
 - 3) **Magnetic stirrer**, with variable speed drive.
 - 4) **Analytical balance**, with reciprocal sensitivity of 0.000 1 g.
 - 5) **Volumetric pipette**, specified in **JIS R 3505**.