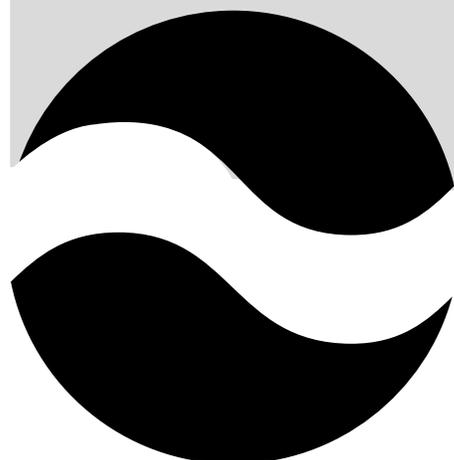


AMERICAN NATIONAL  
STANDARD

ANSI/J-STD-004  
JANUARY 1995

# ***JOINT INDUSTRY STANDARD***

Requirements  
for  
Soldering  
Fluxes



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**JOINT  
INDUSTRY  
STANDARD**

**J-STD-004**

# **Requirements for Soldering Fluxes**

**A joint standard developed by the Flux Specifications Task  
Group of the Institute for Interconnecting and Packaging  
Electronic Circuits**

**Users of this standard are encouraged to participate in the  
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## Acknowledgment

Members of the Flux Specifications Task Group have worked to develop this document. We would like to thank them for their dedication to this effort.

Any Standard involving a complex technology draws material from a vast number of sources. While the principle members of the Flux Specifications Task Group are shown

below, it is not possible to include all of those who assisted in the evolution of this Standard. To each of them, the members of the EIA and IPC extend their gratitude.

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# Requirements for Soldering Fluxes

## 1.0 SCOPE AND DESIGNATION

**1.1 Scope** This standard prescribes general requirements for the classification and testing of soldering fluxes for high quality interconnections. This standard is a flux characterization, quality control, and procurement document for solder flux and flux containing material.

**1.1.1 Purpose** This standard defines the classification of soldering materials through specifications of test methods and inspection criteria. These materials include: liquid flux, paste flux, solder-paste flux, solder-preform flux, and flux cored solder. It is not the intent of this standard to exclude any acceptable flux or soldering aid material; however, these materials must produce the desired electrical and metallurgical interconnection.

The requirements for soldering fluxes are defined in general terms for standardized classification. In practice, where more stringent requirements are necessary or other manufacturing processes are used, these shall be defined as additional requirements by the user.

**1.2 Designation** For ordering purposes and designation by other specifications, the following flux identification system shall be used. (See Table 1.)

**Table 1 Flux Identification, Materials of Composition, Activity Levels<sup>1</sup>**

Flux Materials of Composition <sup>2</sup>	Flux Activity Levels (% Halide)/Flux Type <sup>3</sup>		Flux Designator
Rosin (RO)	Low (0%)	L0	ROL0
	Low (<0.5%)	L1	ROL1
	Moderate (0%)	M0	ROM0
	Moderate (0.5–2.0%)	M1	ROM1
	High (0%)	H0	ROH0
	High (>2.0%)	H1	ROH1
Resin (RE)	Low (0%)	L0	REL0
	Low (<0.5%)	L1	REL1
	Moderate (0%)	M0	REM0
	Moderate (0.5–2.0%)	M1	REM1
	High (0%)	H0	REH0
	High (>2.0%)	H1	REH1
Organic (OR)	Low (0%)	L0	ORL0
	Low (<0.5%)	L1	ORL1
	Moderate (0%)	M0	ORM0
	Moderate (0.5–2.0%)	M1	ORM1
	High (0%)	H0	ORH0
	High (>2.0%)	H1	ORH1
Inorganic (IN)	Low (0%)	L0	INL0
	Low (<0.5%)	L1	INL1
	Moderate (0%)	M0	INM0
	Moderate (0.5–2.0%)	M1	INM1
	High (0%)	H0	INH0
	High (>2.0%)	H1	INH1

<sup>1</sup> Fluxes of Type A–Y are available in S (Solid), P (Paste/Cream) or L (Liquid) forms

<sup>2</sup> See paragraphs 6.2 and 6.3 for comparisons of RO, RE, OR and IN composition classes and L, M and H activity levels with the traditional classes such as R, RMA, RA, water soluble and low solids “no-clean.”

<sup>3</sup> The 0 and 1 indicate absence and presence of halides, respectively. See paragraph 3.2.3 for an explanation of L, M and H nomenclature.

## 2.0 APPLICABLE DOCUMENTS

The following documents of the issue currently in effect, form a part of this specification to the extent specified herein. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

### 2.1 Joint Standards<sup>1</sup>

**J-STD-003** Solderability Tests for Printed Boards

### 2.2 Government<sup>2</sup>

#### 2.2.1 Military<sup>2</sup>

**MIL-STD-45662** Calibration Systems Requirements

### 2.3 International Standards Organization<sup>3</sup>

**ISO 9000** Quality Management and Quality Assurance Standards Guidelines Selection and Use

**ISO 9001** Quality Systems—Model for Quality Assurance in Design/Development, Production, Installation and Servicing.

**ISO 9002** Quality Systems—Model for Quality Assurance in Production and Installation

### 2.4 The Institute for Interconnecting and Packaging Electronic Circuits (IPC)<sup>1</sup>

**IPC-T-50** Terms and Definitions for Interconnecting and Packaging Electronic Circuits

**IPC-TM-650** Test Methods Manual

**2.3.13** Determination of Acid Value of Liquid Solder Flux—Potentiometric and Visual Titration Methods

**2.3.28** Ionic Analysis of Circuit Boards—Chromatography Method.

**2.3.32** Flux Induced Corrosion (Copper Mirror Method)

**2.3.33** Presence of Halides in Flux, Silver Chromate Method

**2.3.34** Solids Content, Flux

**2.3.35** Halide Content, Quantitative (Chloride and Bromide)

**2.3.35.1** Fluorides by Spot Test, Fluxes—Qualitative

**2.3.35.2** Fluoride Concentration, Fluxes—Quantitative

**2.4.14.2** Liquid Flux Activity, Wetting Balance Method

**2.4.34.4** Paste Flux Viscosity - T-Bar Spindle Method

**2.4.46** Spread Test, Liquid or Extracted Solder Flux, Solder Paste and Extracted Cored Wires or Preforms

**2.6.1** Fungus Resistance Printed Wiring Materials

**2.6.3.3** Surface Insulation Resistance (SIR), Fluxes

**2.6.15** Corrosion, Flux

### 2.5 American Society for Testing and Materials (ASTM)<sup>4</sup>

**ASTM D-465** Acid Number of Rosin, Test Methods For

**ASTM D-1298** Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

## 3.0 REQUIREMENTS

**3.1 General Requirements** Except when otherwise specified on the design or assembly drawings or instructions by the user, the soldering fluxes covered by this document shall conform with the following paragraphs.

**3.1.1 Conflict** In the event of conflict between the requirements of this specification and other requirements of the applicable acquisition documents, the precedence in which documents shall govern, in descending order, is as follows:

1. The applicable acquisition document
2. The applicable specification sheet/drawing
3. This specification
4. Applicable documents (see paragraph 2.0)

**3.1.2 Terms and Definitions** Definitions applicable to this specification shall conform to IPC-T-50, and the following:

**3.1.2.1 Form** The flux is additionally classified according to the form that it takes—liquid (L), solid (S), or paste (P).

**3.1.2.2 Inorganic Flux** A solution of inorganic acids and/or salts.

1. Application for copies should be addressed to the IPC, 2215 Sanders Road, Northbrook, IL 60062-6135

2. Publications are available from Standardization Documents Order Dept., Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

3. Publications are available from the International Standards Organization, 1 Rue de Varembe, Case Postale 56, CH-1211 Geneve 20 Switzerland

4. American Society for Testing Materials, 1916 Race Street, Philadelphia, PA 19103-1187

**Table 2 Test Requirements for Flux Activity Classification**

Flux Type <sup>1</sup>	Copper Mirror <sup>2,3</sup>	Qualitative Halide		Quantitative Halide	Corrosion Test <sup>2,6</sup>	Conditions for Passing 100 Megohm SIR Requirements <sup>8,10</sup>
		Silver Chromate (Cl, Br) <sup>2,4</sup>	Spot Test (F) <sup>2</sup>	(Cl, Br, F) <sup>5</sup>		
L0	No evidence of mirror breakthrough	Pass <sup>7</sup>	Pass <sup>7</sup>	0.0%	No evidence of corrosion	Both cleaned and uncleaned <sup>9,10</sup>
L1		Pass <sup>7</sup>	Pass <sup>7</sup>	<0.5%		
M0	Breakthrough in less than 50% of test area	Pass <sup>7</sup>	Pass <sup>7</sup>	0.0%	Minor corrosion acceptable	Cleaned <sup>8</sup> or uncleaned <sup>9,10</sup>
M1		Fail <sup>11</sup>	Fail <sup>11</sup>	0.5 to 2.0%		
H0	Breakthrough in more than 50% of test area	Pass <sup>7</sup>	Pass <sup>7</sup>	0.0%	Major corrosion acceptable	Cleaned
H1		Fail <sup>11</sup>	Fail <sup>11</sup>	>2.0%		

- 0 and 1 in Flux Type column indicate absence and presence of halide, respectively.
- Qualitative
- Refer to Figure 1.
- If the total absence of covalently bonded halogens is required by the user the Beilstein test (*Practical Organic Chemistry*, Mann and Saunders; *A New Dictionary of Chemistry*, Stephen Miall, Editor, Longmans Green & Co., 1940, p. 68) should be performed.
- Quantitative
- A quantitative test for corrosion is currently under development.
- False failure could result from non-halide constituents (see 3.2.4.2).
- If the M0 or M1 flux passes SIR when cleaned, but fails when not cleaned, this flux shall always be cleaned.
- Fluxes which are not meant to be removed require testing only in the uncleaned state.
- If an assembly using no-clean flux is to be cleaned prior to conformal coating, then the user should verify the SIR values after cleaning.
- Depending on the type of halide, one or both tests will be failed.

**3.1.2.3 Organic Flux** Primarily composed of organic materials other than rosin or resin.

**3.1.2.4 Resin Flux** Primarily composed of natural resins other than rosin types, and/or synthetic resins.

**3.1.2.5 Rosin Flux** Primarily composed of natural resin, extracted from the oleoresin of pine trees and refined. Consists of one or more of the following types of rosin: gum rosin, wood rosin, tall oil rosin, modified or natural rosin. The rosins used must have a minimum acid value of 130, as determined per ASTM D-465.

## 3.2 Flux Classification and Testing

**3.2.1 Standard Classification for Products** Fluxes used in the process of soldering shall be classified according to the corrosive or conductive properties of the flux or flux residue (Table 1).

**3.2.2 Composition** Soldering fluxes shall also be classified according to the general chemical composition of the non-volatile portion. Based on a minimum 51% composition of the non-volatile portion, the flux shall be classified as either rosin, resin, organic or inorganic (Table 1).

**3.2.3 Activity** The soldering fluxes of Table 1 shall be further classified by test requirements relating to the activity of the flux and its residue. Soldering fluxes shall be characterized according to one of the following three types:

L = Low or no flux/flux residue activity

M = Moderate flux/flux residue activity

H = High flux/flux residue activity

These classes shall be further characterized using 0 or 1 to indicate absence or presence of halide in the flux. Both the L, M, H and 0, 1 classifications shall be determined per the test methods in Table 2.

It is the responsibility of the material suppliers to characterize their fluxes in accordance with the classification requirements in this standard.

**3.2.3.1 Flux Activity Classification** In order to be classified as a specific type, a flux must meet all the characterization requirements as shown in Table 2.

Flux used for the soldering of electronic assemblies shall be further assessed by the impact of flux residue on the assembly's performance. Conductive properties of the flux residue shall meet the SIR requirements contained in Table 2.

(It is conceivable that certain corrosive fluxes could meet one or more tests for the L-type flux. However, failure to meet all test requirements will cause the given flux to be classified as either type M or H.)

### 3.2.4 Flux Type Classification (Table 2)

**3.2.4.1 Copper Mirror Test** The corrosive properties of flux shall be determined in accordance with IPC-TM-650, Test Method 2.3.32.

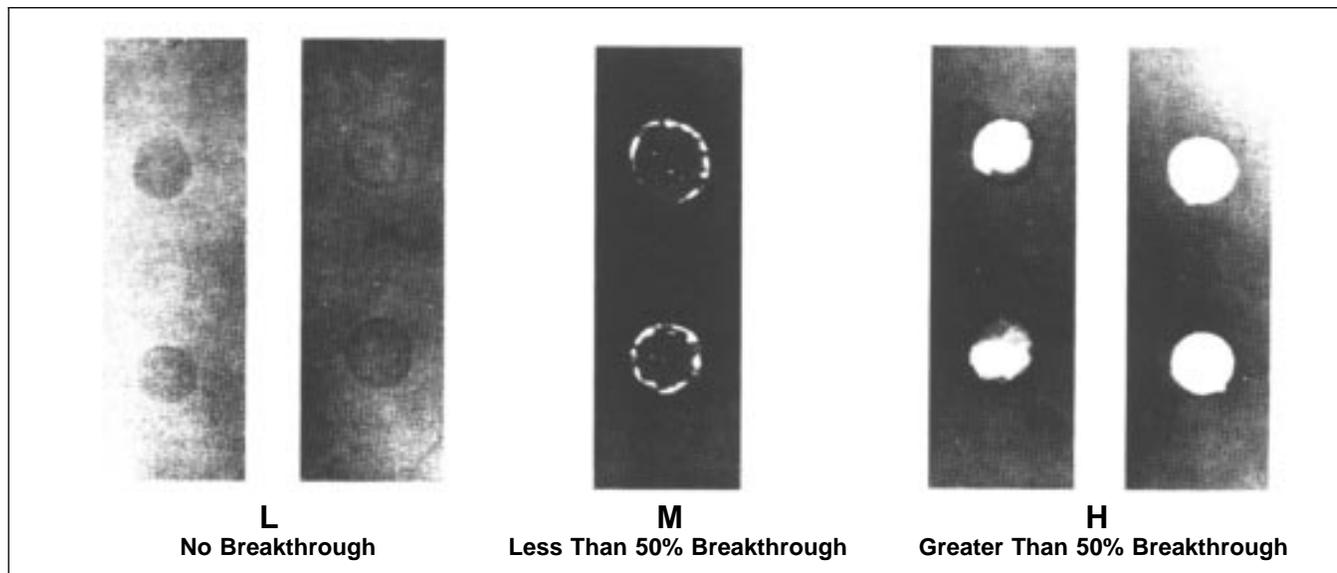


Figure 1 Flux type classification by copper mirror test

**3.2.4.2 Qualitative Halide Tests** The following tests are used to determine the presence of halides, specifically, chlorides (Cl), bromides (Br), and fluorides (F), in liquid fluxes or extracted flux solutions.

**3.2.4.2.1 Chlorides and Bromides by Silver Chromate Method** The presence of chlorides and bromides shall be determined in accordance with IPC-TM-650, Test Method 2.3.33.

**3.2.4.2.2 Fluorides By Spot Test** The presence of fluorides shall be determined in accordance with IPC-TM-650, Test Method 2.3.35.1.

**3.2.4.3 Quantitative Halide Content Tests** If the flux fails the test in 3.2.4.2.1 and/or 3.2.4.2.2, the following tests shall be used to determine the concentration of chlorides (Cl), bromides (Br), and fluorides (F), in liquid fluxes or extracted flux solutions. The halide content is reported as the equivalent weight percentage of chloride to the solid (nonvolatile) portion of the flux. The solids content is determined in accordance with 3.2.4.3.3. The total halide content of the flux is obtained by adding together the halide contents from sections 3.2.4.3.1 (Cl and Br) and 3.2.4.3.2 (F). Ion chromatography is also an acceptable test method for quantitative halide content.

**3.2.4.3.1 Chloride and Bromide Concentrations** The combined concentration of chlorides and bromides shall be determined in accordance with IPC-TM-650, Test Method 2.3.35 or Test Method 2.3.28.

**3.2.4.3.2 Fluoride Concentration** For fluxes giving a positive result when tested per 3.2.4.2.2, the concentration of fluorides shall be determined using IPC-TM-650, Test Method 2.3.35.2 or Test Method 2.3.28.

**3.2.4.3.3 Flux Solids (Nonvolatile) Determination** The determination of the amount of residual solids content of liquid fluxes shall be made in accordance with IPC-TM-650, Test Method 2.3.34 or as agreed between the manufacturer and the user. For fluxes with solids contents less than 10%, the solids content shall not differ by more than 10% of the manufacturer's nominal value. For all other fluxes, flux solids content shall not differ by more than 5% of the manufacturer's nominal value.

**3.2.4.4 Corrosion Test** The corrosive properties of flux residue shall be determined in accordance with IPC-TM-650, Test Method 2.6.15.

**3.2.4.5 Surface Insulation Resistance (SIR) Test** The surface insulation requirements for fluxes shall be determined in accordance with IPC-TM-650, Test Method 2.6.3.3.

**3.2.4.5.1 Reporting Flux Surface Insulation Resistance Values** When specifying the SIR test results, the supplier shall clearly indicate the type of cleaning procedure used prior to SIR testing (See Appendix A - Qualification Test Report). SIR values shall be measured at 24, 96, and 168 hours, while the specimen is still under elevated temperature and humidity. Specimens shall comply with the requirements specified in Table 2 (100 megohms) measured at 96 and 168 hours.

**3.2.4.6 Fungus Test** When specified in the procurement document fungus resistance shall be evaluated in accordance with IPC-TM-650, Test Method 2.6.1.

**3.2.5 Qualification** For qualification testing the flux vendor shall perform the testing in accordance with tests listed in Table 4 and complete a qualification test report (See Appendix A).

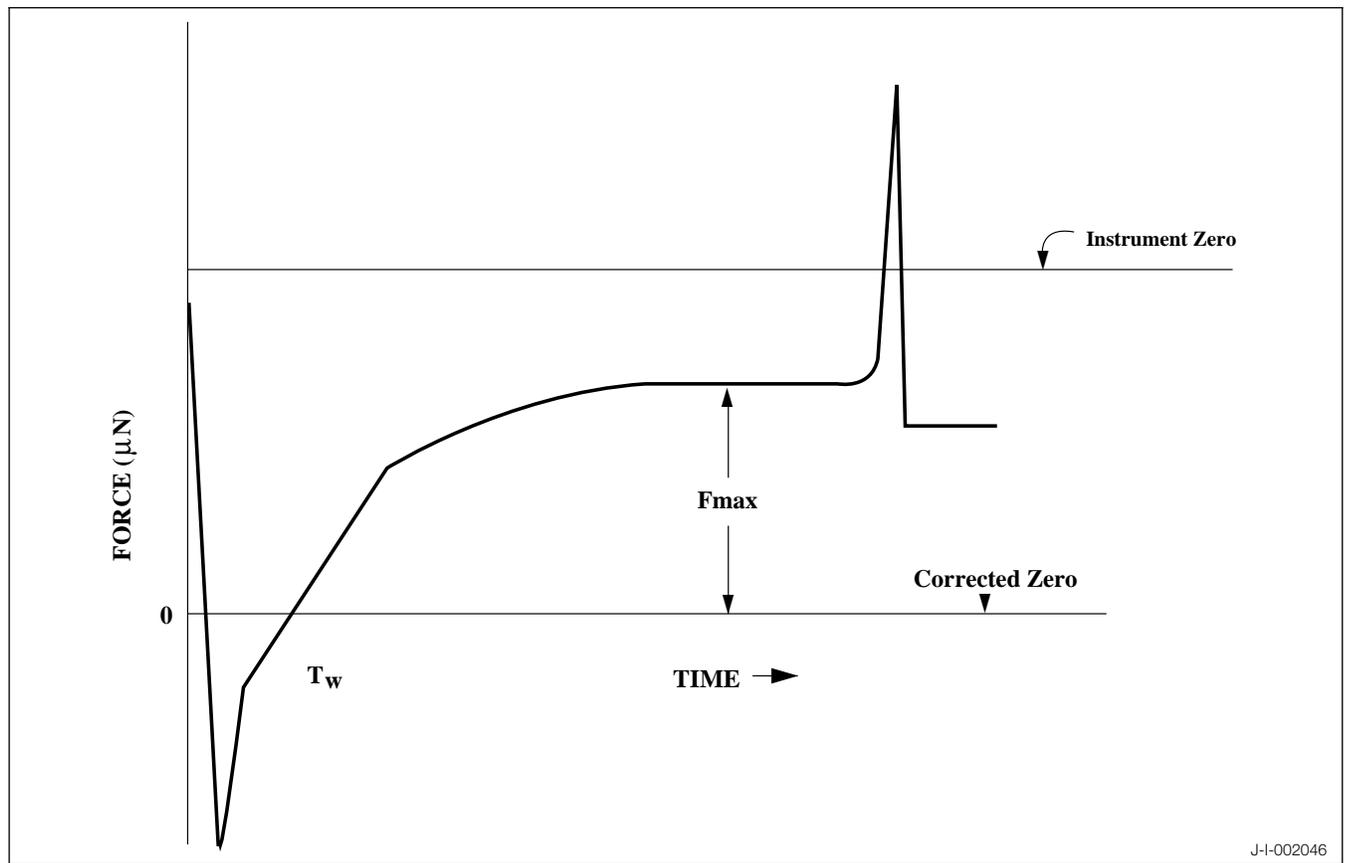


Figure 2 Wetting balance curve

**3.2.6 Quality Conformance** The following tests shall be performed to evaluate product consistency per the flux manufacturers technical data sheets. (See 4.6)

**3.2.6.1 Acid Value Determination** The acid value of liquid flux shall be evaluated in accordance with IPC-TM-650, Test Method 2.3.13.

**3.2.6.2 Flux Specific Gravity Determination** For fluxes with solids contents of 10% or less flux density shall be supplied within 0.003 g/ml from the nominal value at specified temperature. For fluxes with solids higher than 10%, flux density shall be supplied within 0.005 g/ml from the nominal value at specified temperature. Density (specific gravity) determination shall be performed using the hydrometer method in accordance with ASTM D-1298 (static method).

**3.2.6.3 Determination of Viscosity of Paste or Solder Paste Flux** The viscosity of the paste flux shall be evaluated in accordance with IPC-TM-650, Test Method 2.4.34.4.

**3.2.6.4 Visual** The flux material shall be examined for clarity and the absence of precipitation.

**3.2.7 Performance** When specified, the following tests shall be used to determine the flux's performance.

If the stated shelf life on a product is expired, but the product meets the performance test, it may be used.

**3.2.7.1 Wetting Balance Test (Optional)** When specified, the wetting balance shall be evaluated in accordance with IPC-TM-650, Test Method 2.4.14.2. Using the coupon as specified, the acceptance to satisfactory solderability shall be defined as those fluxes which exhibit the following:

1. A wetting time ( $T_w$ ) for the wetting curve to cross the corrected zero axis after the start of the test in less than 2.0 seconds. (See Figure 2)
2. A maximum wetting force,  $F_{max}$ , taken after correction for buoyancy (see J-STD-003) greater than the minimum acceptance force of 150 microneutons/mm.
3. If the "dip and look" requirement is specified, the sample from the above procedure can be used, and a minimum of 95% of the surface area immersed shall exhibit good wetting.

**3.2.7.2 Spread Test—Liquid Flux (Optional)** The solder spread may be determined by means of a flux wetting/spreading test (static method) per IPC-TM-650, Test Method 2.4.46. (Table 3 is intended as an aid in defining areas in  $mm^2$ .)

**Table 3 Typical Spread Areas Defined in mm<sup>2</sup>**

Diameter in mm	Area in mm <sup>2</sup> Circles
10.00	78.54
10.70	90.00*
11.28	100.00**

\* Minimum for L1

\*\* Minimum for M1

**3.2.8 Labeling** The manufacturer shall label each container of solder flux with the following:

1. The manufacturer's name and address.
2. The number of this standard and the soldering product part number (see paragraph 6.6).
3. The manufacturer's designation of the flux .
4. The net mass of flux.
5. The batch number.
6. The date of manufacture.
7. Shelf life.
8. Any health, safety and environmental markings applicable in the country of use.

#### 4.0 QUALITY ASSURANCE PROVISIONS

**4.1 Responsibility for Inspection** The flux manufacturer is responsible for the performance of all inspection specified herein except the performance inspections which are the responsibility of the user. The flux manufacturer may use its own or any other facilities suitable for the performance of the inspections specified herein, unless disapproved by the user. The user reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to ensure that supplies and services conform to prescribed requirements.

**4.1.1 Responsibility for Compliance** Materials covered by this specification shall meet all requirements of Section 3. The inspection(s) excluding the performance inspections defined in this specification shall become a part of the contractor's overall inspection system or quality program. The vendor has responsibility of ensuring that all products or supplies submitted to the user for acceptance comply with all requirements of the purchase order contract.

**4.1.1.1 Quality Assurance Program** When required by the user, a quality assurance program for material furnished under this specification shall be established and maintained in accordance with ISO 9002, or as otherwise agreed on between user and manufacturer, and shall be monitored by the qualifying activity.

**4.1.2 Test Equipment and Inspection Facilities** Test/measuring equipment and inspection facilities, of sufficient accuracy, quality, and quantity to permit performance of

the required inspection(s), shall be established and maintained or designated by the supplier. Establishment and maintenance of a calibration system to control the accuracy of the measuring and test equipment shall be in accordance with MIL-STD-45662.

**4.1.3 Inspection Conditions** Unless otherwise specified herein, all inspections shall be performed in accordance with the test conditions specified in Section 3.

**4.2 Classification of Inspections** The inspections specified herein are classified as follows:

1. Materials Inspection (4.3)
2. Qualification Inspection (4.4)
3. Performance Inspection (4.5)
4. Quality Conformance (4.6)

**4.3 Materials Inspection** Materials inspection shall consist of certification supported by verifying data that the materials used in compounding the flux, are in accordance with the applicable referenced specifications or requirements prior to such compounding. The verifying data and certification applicable to a qualification test sample shall be made a part of the qualification test report.

**4.4 Qualification Inspection** Qualification inspection shall be performed at a laboratory acceptable to the user on samples produced with equipment and procedures normally used in production.

**4.4.1 Sample Size** Sample sizes shall be appropriate to the solder form being inspected and the inspection being performed.

**4.4.2 Inspection Routine** The sample shall be subjected to the inspections specified in Table 4.

**4.5 Performance Inspection** Performance Inspections are non-mandatory inspections and are for the benefit of the user.

**4.6 Quality Conformance** The material manufacturer shall perform those inspections necessary to insure that the process is in control and to insure that the product is within specification limit.

**4.6.1 Sampling Plan** Statistical sampling and inspection shall be in accordance with an approved quality program. (See 4.1.1.1).

**4.6.2 Rejected Lots** If an inspection lot is rejected, the supplier may rework it to correct the defects, or screen out the defective units and resubmit for reinspection. Resubmitted lots shall be inspected using tightened inspection.

**Table 4 Solder Flux Test Method Classification**

Test Method		Qualification	Quality Conformance Inspection Certificate of Compliance	Performance
<b>Name (Reference Paragraph)</b>	<b>IPC-TM-650</b>			
<i>Materials</i>				
Copper Mirror (3.2.4.1)	2.3.32	X		
<i>Halides, Qualitative (3.2.4.2)</i>				
Silver Chromate (3.2.4.2.1)	2.3.33	X		
Fluoride Spot (3.2.4.2.2)	2.3.35.1	X		
<i>Halides Quantitative (3.2.4.3)</i>				
Chloride, Bromide (3.2.4.3.1)	2.3.35 or 2.3.28	X		
Fluoride (3.2.4.3.2)	2.3.35.2 or 2.3.28	X		
Nonvolatile Determination (3.2.4.3.3)	2.3.34	X		
Corrosion (3.2.4.4)	2.6.15	X		
SIR (3.2.4.5)	2.6.3.3	X		
Acid Value Determination (3.2.6.1)	2.3.13		X	
Spread Test, Liquid Flux (3.2.7.2)	2.4.46			(O)
Wetting Balance (3.2.7.1)	2.4.14.2			(O)
Flux Specific Gravity Determination (3.2.6.2)			X	
Viscosity—Paste Flux (3.2.6.3)	2.4.34.4		X	
Visual (3.2.6.4)			X	
Fungus (3.2.4.6)	2.6.1	(O)		

(O) – Optional

**Table 5 Flux Form for Test**

Flux Material	Copper Mirror	Halides	SIR	Corrosion
Liquid flux as received	X <sup>1</sup>	X	X	X
Paste flux as received	X	X	X	X
Solder paste as received	X <sup>2</sup>	X	X	X
Melted & Extracted Solder Paste	X <sup>2</sup>			
Preforms as received				X
Cored-wire as received				X
Extracted preforms <sup>3</sup>	X	X	X	
Extracted cored wire <sup>3</sup>	X	X	X	

<sup>1</sup> A flux containing >50% water may be oven dried at 80°C and reconstituted in an alcohol solvent for use in this test.

<sup>2</sup> See 4.7.5 and 4.7.5.1

<sup>3</sup> Extracted then concentrated/diluted

Such lots shall be separate from new lots, and shall be clearly identified as reinspected lots.

#### 4.7 Preparation of Fluxes for Testing

**4.7.1 Flux Form for Test** The form of the flux to be used for each classification test is shown in Table 5.

**4.7.2 Liquid Fluxes** Testing for flux and flux residue activity is done on liquid fluxes and other (liquid) fluxing materials, such as flux thinners, infrared reflow fluids, soldering (intermix) oils, preparation fluid (from inert atmosphere soldering), etc., in the “as supplied” form.

**4.7.3 Solid Fluxes** Testing for flux activity will be done on material as a 35% weight percent solution in 2-propanol, reagent grade (or other solvent recommended by the manufacturer).

**4.7.4 Paste Flux** Testing for flux activity will be done on material as a 35% weight percent solution in 2-propanol, reagent grade (or other solvent recommended by the manufacturer).

**4.7.5 Solder Paste** Solder paste shall be tested as received. If the solder paste, as received, does not pass the copper mirror test as an L-type flux, the reflowed solder paste flux (see 4.7.5.1) may be used for the test. If this passes in the L category, the solder paste flux shall be listed as an L for this test.

**4.7.5.1 Procedure for Preparing Reflowed Solder-Paste Flux** To assess the corrosivity of reflowed solder-paste flux, the following sample preparation procedure shall be used. Two containers are required. Container No. 1 may be a test tube or small beaker. Preferably container No. 2 should be a low-profile container, such as a Petri dish or watch glass, to enhance heat transfer. Label Container No. 1 and record its weight. Place 10 grams of solder paste (A) in Container No. 2 and hold in a solder bath maintained at 215°C for 20 seconds beyond the time required for the pool of liquid solder alloy to form. While the solder is molten,

carefully pour the liquified hot solder-paste flux into Container No. 1 and allow to cool. Weigh Container No. 1 to determine the weight of reflowed solder-paste flux. Add an excess amount of 2-propanol solvent or other more suitable solvent such that after evaporation a 25% by weight solution of reflowed solder paste flux will result. Stir or agitate the mixture as required. If a flocculent precipitate results, this precipitate should be allowed to settle and the supernatant liquid used for subsequent tests. Store the solution in a closed container.

**4.7.6 Other Materials** Flux-cored solder wires and flux-cored solder preforms shall be extracted for testing, as specified in paragraph 4.7.6.1 and 4.7.6.2. Testing for flux extract activity shall be performed on extracts dissolved in a suitable solvent. Fluxes intended to be cleaned shall be tested as 10% solid solutions, while those no-clean flux extracts shall be tested as a 5% solid solution.

**4.7.6.1 Flux Extraction Procedure for Flux-Cored Solder Wire** Cut a length of wire to correspond to approximately 150 grams. Seal the ends with a soldering iron. Wipe the surface clean with a cloth moistened with 2-propanol. Wind the wire around a 50 mm mandrel. Place the coil into a beaker filled with deionized water to cover the coil and boil for 5–6 minutes. Decant, rinse the coil with 2-propanol and dry. Cut the wire into approximately 3 mm lengths with a sharp blade. Discard the sealed ends of the wire. Two containers are required. Container No. 1 may be a test tube or small beaker. Preferably container No. 2 should be a low-profile container, such as a Petri dish or watch glass, to enhance heat transfer. Label Container No. 1 and record its weight. Place the accumulated 3 mm lengths in Container No. 2 and hold in a solder bath maintained at 215°C for 20 seconds beyond the time required for the pool of liquid solder alloy to form. While the solder is molten, carefully pour the liquified hot cored-solder flux into Container No. 1 and allow to cool. Weigh Container No. 1 to determine the weight of the reflowed cored-solder flux. Add an excess amount of 2-propanol or other suitable

solvent such that after evaporation a 10% by weight solution of reflowed cored- solder flux will result. For low solids, no clean fluxes a 5% solution shall be used. Stir or agitate the mixture as required. If a flocculent precipitate results, this precipitate should be allowed to settle and the supernatant liquid used for subsequent tests. Store the solution in a closed container.

**4.7.6.2 Flux Extraction Procedure for Flux-Coated and Flux-Cored Preforms** Two containers are required. Container No. 1 may be a test tube or small beaker. Preferably container No. 2 should be a low-profile container, such as a Petri dish or watch glass, to enhance heat transfer. Label Container No. 1 and record its weight. Place 10 grams of solder preforms in Container No. 2 and hold in a solder bath maintained at 215°C for 20 seconds beyond the time required for the pool of liquid solder alloy to form. While the solder is molten, carefully pour the liquified hot solder-preform flux into Container No. 1 and allow to cool. Weigh Container No. 1 to determine the weight of reflowed solder-preform flux. Add an excess amount of 2-propanol or other suitable solvent such that after evaporation a 10% by weight solution of reflowed solder preform flux will result. For low solids, no clean fluxes a 5% solution shall be used. Stir or agitate the mixture as required. If a flocculent precipitate results, this precipitate should be allowed to settle and the supernatant liquid used for subsequent tests. Store the solution in a closed container.

If the user requires an assessment of the as-received solid or paste flux activity, paste or solid fluxes identical to those in the solder containing materials shall be obtained from a supplier and applied to the test vehicles as 10 weight percent solutions in 2-propanol or other suitable solvent. If the flux component is a low-solids flux, a 5 weight percent solution in 2-propanol shall be used.

## 5.0 PREPARATION FOR DELIVERY

**5.1 Preservation-packing and Packaging** Unless otherwise specified, the preservation-packing, packaging, and marking of soldering products shall be equivalent to or better than the supplier's standard commercial practice.

## 6.0 NOTES

**6.1 Intended Use** This document is intended to be applicable to all types of flux as used for soldering in general and to soldering in electronics particularly. The fluxes involved relate to all aspects of application, such as for wave soldering, PWB fabrication, lead tinning, and reflow, and include those in solder pastes, flux-cored wire, and flux-coated preforms. Soldering fluxes covered by this standard are intended for use in various consumer, industrial and commercial electronics soldering applications of industry and, when adopted by government, in applications on that government's electronic hardware.

**6.2 Flux Composition** Low solids "no-clean" fluxes may have rosin compositions (symbol RO), resin compositions (symbol RE), or they may be substantially free of rosins and resins, in which case they are classified as organic type (symbol OR). They typically have type L or M activity levels. Water soluble fluxes for electronic soldering applications and synthetic activated fluxes generally have organic compositions (symbol OR). They typically have type H activity levels.

**6.3 Flux Activity** In order to illustrate an approximate, though not necessarily always accurate, analogy between L, M, and H type fluxes with the traditional classes of rosin-based fluxes (R, RMA, RA, and RSA), as well as other fluxes such as water soluble or synthetic activated fluxes, the following guideline is offered:

- L0 Type Fluxes – All R,  
Some RMA  
Some Low Solids "No-Clean"
- L1 Type Fluxes – Most RMA,  
Some RA
- M0 Type Fluxes – Some RA,  
Some Low Solids "No-Clean"
- M1 Type Fluxes – Most RA, Some RSA
- H0 Type Fluxes – Some Water Soluble
- H1 Type Fluxes – Some RSA,  
Most Water Soluble and Synthetic  
Activated

**6.4 Flux and Cleaning Relationship** The type of flux used impacts cleaning requirements of the assembly, so any changes in flux type, or cleanliness requirements may require changes in cleaning method and test methods.

**6.5 Ordering Data** Purchasers should select the preferred options permitted herein and include the following information in procurement documents:

- a. Title, number, revision, and date of this specification
- b. Part number of product
- c. Flux type or type symbol (see Table 1)
- d. Flux form
- e. Viscosity
- f. Viscosity tolerance, if different
- g. Slump criteria, if different
- h. Tack criteria
- i. Detailed requirements for nonstandard solder flux and/or solder flux characteristics, when specified

**6.6 Formic Acid** Formic acid is not considered a flux for the purposes of this document.

**Appendix A  
Qualification Test Report**

<b>I.D. Number:</b>				
<b>Flux Classification:</b>			<b>Date of Manufacture:</b>	
<b>Manufacturer's Identification:</b>			<b>Original Use by Date:</b>	
<b>Manufacturer's Batch Number:</b>			<b>Requalified Use by Date:</b>	
<b>Date Original Qualification Tests Completed:</b>			<b>Original:</b>	
<b>Date Requalification Tests Completed:</b>				
<b>Tested By:</b>		<b>Pass: _</b>	<b>Tested By:</b>	
			<b>Pass: _</b>	
<b>Witnessed By:</b>		<b>Fail: _</b>	<b>Witnessed By:</b>	
			<b>Fail: _</b>	
<b>Certification Test</b>	<b>Requirement Paragraph</b>	<b>IPC-TM-650 Method</b>	<b>Test Requirement</b>	<b>Result Pass/Fail/NA</b>
Copper Mirror	3.2.4.1	2.3.32		
<i>Halides Qualitative</i>	3.2.4.2			
Silver Chromate	3.2.4.2.1	2.3.33		
Fluoride Spot	3.2.4.2.2	2.3.35.1		
<i>Halides Quantitative</i>	3.2.4.3			
Chloride, Bromide	3.2.4.3.1	2.3.35, 2.3.28		
Fluoride	3.2.4.3.2	2.3.35.2		
Nonvolatile Determination	3.2.4.3.3	2.3.34		
Total as Chloride	3.2.4.3.1			
Corrosion	3.2.4.4	2.6.15		
SIR	3.2.4.5	2.6.3.3	Cleaned Not Cleaned	
Fungus (optional)	3.2.4.6	2.6.1		
<i>Cleaning Procedure for Flux Characterization</i>				
Cleaning Material _____				
Cleaning Equipment _____				
Cleaning Process Parameters _____				
_____				
_____				



# IPC-TM-650 TEST METHODS MANUAL

**1.0 Scope** This test method specifies two methods for the determination of the acid value of a flux of types L, M, or H.

Method A is a potentiometric titration method and is to be considered as the reference method.

Method B is an alternative, visual end-point, titration method.

## 2.0 Applicable Documents

**ISO 9455** Soft Soldering Fluxes, Test Methods

**IPC-TM-650 - Test Methods Manual**

**2.3.34** Solids Content, Flux

**3.0 Test Specimen** A minimum of 2.0 grams of liquid flux, 10 grams of solder paste, 150 grams of cored wire or 10 grams of solder preforms.

## 4.0 Apparatus and Reagents

### 4.1 General

1. Use only reagents of recognized analytical quality and only distilled or deionized water.
2. Ordinary laboratory apparatus.

**4.2** For Potentiometric Titration Method (Method A).

**4.2.1** Tetrabutyl ammonium hydroxide. 0.1 M (0.1 mol/l). Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with 2-propanol (4.2.2). Alternatively, prepare an 0.1 mol/l tetrabutyl ammonium hydroxide solution by diluting commercial concentrated solution with 2-propanol and standardize this solution against an accurately weighed amount of benzoic acid (about 0.5 g) dissolved in dimethylformamide, previously neutralized to thymol blue.

**4.2.2** 2-Propanol. Neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink color using phenolphthalein as an indicator.

**4.2.3** Ethanol. Anhydrous, neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink color using phenolphthalein as an indicator.

**4.2.4** Toluene. Neutralized with tetrabutyl ammonium hydroxide solution (4.2.1) to a faint pink color using phenol-

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Subject <b>Determination of Acid Value of Liquid Solder Flux—Potentiometric and Visual Titration Methods</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

phthalein as an indicator.

**4.2.5** Ethanol/toluene mixture. Mix equal volumes of the anhydrous ethanol (4.2.3) and toluene (4.2.4).

**4.2.6** Millivoltmeter or pH meter.

**4.2.7** Glass electrode.

**4.2.8** Saturated calomel, or silver chloride/silver, electrode.

**4.2.9** Magnetic or mechanical stirrer with variable speed drive.

**4.3** For titration with visual End-Point (Method B).

**4.3.1** Ethanol, anhydrous. Neutralized with potassium hydroxide, 0.1 M in alcohol (4.3.5), to a faint pink color using phenolphthalein as an indicator.

**4.3.2** Toluene. Neutralized with potassium hydroxide, 0.1 M in alcohol (4.3.5), to a faint pink color using phenolphthalein as an indicator.

**4.3.3** Ethanol/toluene mixture. Mix equal volumes of the anhydrous ethanol (4.3.1) and toluene 4.3.2).

**4.3.4** 2-Propanol. Neutralized with potassium hydroxide, 0.1 M in alcohol (4.3.1), to a faint pink color using phenolphthalein as an indicator.

**4.3.5** Potassium hydroxide solution. 0.1 M in alcohol. Use a commercially available standard solution or one prepared from a commercially available concentrated standard solution by dilution with ethanol (4.3.1). Alternatively, prepare an 0.1 Mol/l potassium hydroxide solution by dissolving 3 +/-0.1 g potassium hydroxide (KOH) in 500 ml of ethanol (4.3.1), and standardize this solution against an accurately weighed amount of benzoic acid (about 0.5 g) dissolved in ethanol (4.3.1).

**4.3.6** Phenolphthalein indicator solution. Add 1g of phenolphthalein to approximately 50 ml methanol and mix. When dissolved, dilute to 100 ml with methanol and mix.

## 5.0 Procedures

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## 5.1 Potentiometric Titration (Method A)

**5.1.1** By preliminary experiments, determine whether the sample is soluble in 2-propanol, anhydrous ethanol, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents then use 2-propanol.

**5.1.2** Carry out the following procedure, in triplicate, on the flux sample.

**5.1.2.1** Weigh, to the nearest 0.001 g, 2.0 to 5.0 g of the liquid flux sample taking steps to prevent loss of volatile matter during the weighing. The larger size sample is required for very low solids fluxes. Transfer the weighed sample to a 250 ml low form beaker.

**5.1.2.2** Dilute sample to 100 ml with 2-propanol, or the selected solvent (4.2.3 to 4.2.5), according to the solubility characteristics of the flux. Cover with a watch glass and dissolve the flux by gentle agitation.

**5.1.2.3** Place the beaker on the stand of the titration assembly with the electrodes, stirrer and burette in position. Adjust the speed of the stirrer to give vigorous stirring without splashing. Titrate with the tetrabutyl ammonium hydroxide solution (4.2.1), adding 1 ml portions and recording the pH, or mV meter readings after each addition. As the end point is approached, reduce the additions of titrant to 0.1 ml and continue titrating past the end point.

**5.1.2.4** Plot the pH, or potential values against the volume of titrant added to obtain the titration curve. The point of inflection of the curve corresponds to the end-point of the titration.

*Note:* The point of inflection of the curve may conveniently be determined by using the derivative curve.

**5.1.2.5** Carry out a blank determination, using all reagents, for comparison purposes.

## 5.2 Visual Titration (Method B)

**5.2.1** By preliminary experiments, determine whether the sample is soluble in 2-propanol anhydrous ethanol, toluene or the ethanol/toluene mixture. If it is not completely soluble in any of these solvents, select the one in which it appears to be the most soluble. If it is equally soluble in all four solvents then use ethanol as the selected solvent.

**5.2.2** Carry out the following procedure, in triplicate, on the flux sample.

**5.2.2.1** Weigh, to the nearest 0.001 g sufficient of the flux sample to correspond to approximately 1 g of non-volatile matter (see IPC-TM-650, test method 2.3.34), taking steps in the case of liquid flux samples to prevent loss of volatile matter during the weighing.

**5.2.2.2** Transfer the weighed sample to a suitable flask or beaker and add 100 ml of the selected solvent. Stir until the sample has dissolved as completely as possible. Do not heat.

**5.2.2.3** Add 3 drops of phenolphthalein indicator (4.3.6) and titrate with the potassium hydroxide (4.3.5) until a faint pink color persists throughout the titrated solution for 15 seconds.

**5.2.2.4** Carry out blank determination, using all reagents, for comparison purposes.

**5.3 Calculation of Results** The acid value is expressed in milligrams of potassium hydroxide per gram of non-volatile matter, regardless of the alkali used to perform the titration.

**5.3.1** The acid value (expressed in milligrams of potassium hydroxide per gram of non-volatile matter) is given by:

$$\frac{56.11 \text{ VM}}{\text{mS}}$$

Where:

V is the volume, in ml, of alkali used (tetrabutyl ammonium hydroxide for method A, potassium hydroxide for method B).

M is the molarity of the alkali used.

m is the mass, in grams of the sample taken.

S is the percentage non-volatile matter determined as described in IPC-TM-650, test method 2.3.34.

The acid value (expressed in milligrams of potassium hydroxide per gram of flux) is given by:

$$\frac{56.11 \text{ VM}}{\text{m}}$$

**5.3.2** The acid value of the flux under test is calculated as the mean of the results obtained on each of the three test samples.

## 6.0 Notes

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**6.1 Safety** Operator should be trained and familiar with the hazards inherent to the chemicals being used and analyzed. Proper personal safety equipment, such as safety glasses, gloves and splash apron, and adequate ventilation should be used.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.28</b>	
Subject <b>Ionic Analysis of Circuit Boards Ion Chromatography Method</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>New Methods Task Group (5-32f)</b>	

## 1.0 Scope

**1.0** This test procedure is designed to measure the level of anionic contaminants on the surface of circuit boards by ion chromatography.

## 2.0 Applicable Documents

**IPC-TP-1043** "Cleaning and Cleanliness Test Program, Phase III, Water Soluble Fluxes, Part 1: B-Z4, Interactions of Water Soluble Fluxes with Metal/Substrates. October, 1992."

**IPC-TP-1044** "Cleaning and Cleanliness Test Program, Phase III, Water Soluble Fluxes, Part 2: B-36, Comparison to Phase 1 Rosin Benchmark", September 1992.

## 3.0 Test Specimens

**3.1** Printed Wiring Board (PWB) for extraction

## 4.0 Apparatus and Material

**4.1** Dionex 4000i (Ion Chromatograph) or equivalent. The system consists of a gradient pump and an anion column (AS4A-SC or equivalent), and a conductivity detector. A system which is operating properly should be capable of 50 ppb or better. The equipment and chemistry should be set up and standardized per manufacturers' instructions.

**4.2** Hot Water Bath capable of holding 80°C ±5°C.

**4.3** Use a clean heat sealable bag, ie. KAPAK® 500 series or equivalent, with less than 250 ppb extractable contaminants. (Specify cleanliness level or manufacturers' part number.)

**4.4** Cleanroom vinyl gloves. (<3ppm of Cl)

**4.5** Hi-purity deionized water 18.3 meg-ohm grade and chloride levels of less than 50 ppb.

**4.6** Hi-purity chemicals for eluent and regenerant preparation.

**4.7** NIST traceable standards

**4.8** 2-Propanol (IPA), Electronic grade

## 5.0 Procedure

## 5.1 Extraction

**5.1.1** Record area of PWB. General rule on surface area is (length x width x 2) + 10% for a populated PWB.

**5.1.2** Use clean gloves when handling the samples to be tested, and then place each sample in the extraction bag.

**5.1.3** Prepare 75/25 IPA/H<sub>2</sub>O solutions for the extraction.

**5.1.4** Add 100-250 mls of the extraction solution to the extraction bag (enough to cover the PWB).

**5.1.5** Heat seal the extraction bag and place in the 80°C water bath for one hour (cut a vent hole in the bag).

**5.1.6** Measure solution volume after extraction.

**5.1.7** Prepare unprocessed PWB as control.

## 5.2 Standard and Sample Analysis.

**5.2.1** Inject solution into Ion Chromatograph (IC) and calculate against known standards.

**5.2.2** Values from the IC are in the ppb in solution range.

**5.2.3** Standards should be used per mfg. instruction. (Chloride levels of 100 ppb are recommended).

**5.2.4** A calculation to take into account for surface area and evaporation must be done so as to compare all different sizes of circuit boards.

$$\text{ug/cm}^2 = \frac{(\text{ppb value from IC}/1000) \times (\text{final volume}/\text{original volume})}{\text{Surface Area (cm}^2\text{)}}$$



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.32</b>	
Subject <b>Flux Induced Corrosion (Copper Mirror Method)</b>	
Date <b>1/95</b>	Revision <b>C</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to determine the removal effect the flux has (if any) on the bright copper mirror film which has been vacuum deposited on clear glass.

## 2.0 Applicable Documents

**ASTM E104** Maintaining Constant Relative Humidity by means of Aqueous Solutions

**LLL-R-626** Rosin, Gum, Rosin Wood and Rosin Tall Oil

**3.0 Test Specimen** A minimum of 100 ml of liquid flux, a representative container of solder paste, reflowed solder-paste flux, extracted solder preform flux or extracted flux-cored wire.

## 4.0 Apparatus and Reagents

**4.1** 0.5L of control standard rosin flux, class A, type II, grade WW, of LLL-R-626.

**4.2** 0.5L of reagent grade (99% pure) 2-propanol.

**4.3** A vacuum deposition system or the means to procure glass test panels having a copper mirror coating as described in paragraph 5 below.

**4.4** 0.5L of reagent grade 0.5% solution of ethylene diamine tetra acetic acid (EDTA).

**4.5** 0.5L of reagent grade ethanol or methanol.

**4.6** 100 ml medicine bottle with dropper.

**4.7** Test cabinet capable of achieving  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity.

## 4.8 Glass slides

**4.9** A relative humidity gauge having a  $\pm 2\%$  accuracy, or better, shall be used to continuously monitor the test environment. The gauge should be calibrated periodically.

## 5.0 Procedures

### 5.1 Preparation

**5.1.1 Preparation of Control Standard Flux** Dissolve 35

g of Federal Specification LLL-R-626 rosin into 100 ml of reagent grade 99% 2-propanol and stir thoroughly.

### 5.1.2 Preparation of Temperature/Humidity Chamber

When acid or salt solutions, such as reported in ASTM E104, are used the environment shall be monitored for a minimum of 48 hours prior to exposing the copper mirror samples, to assure compliance with the  $50\% \pm 5\%$  relative humidity requirement.

### 5.1.3 Preparation of Copper Mirror Test Panels.

**5.1.3.1** Apply by vacuum deposition, a film of copper metal on one surface of a flat sheet or clear, polished glass.

**5.1.3.2** Apply a uniform thickness of approximately 50 nm and assure that the finished mirror permits  $10 \pm 5\%$  transmission of normal incident light of nominal wave length of 500 nm. This may be determined using a suitable photoelectric spectrophotometer. Commercially available copper mirrors meeting the above specifications are acceptable. (See 6.2.)

**5.1.3.3** Prevent oxidation of the copper mirror by storing in a closed container which has been flushed with nitrogen.

**5.1.3.4** Immediately before testing, immerse the copper mirror in a 5 g/l solution of EDTA for copper oxide removal. Mirrors stored in a non-oxidizing environment, do not require cleaning with the EDTA solution prior to testing. The cleaning step must be used if test results are in dispute.

**5.1.3.5** Rinse thoroughly in running water, immerse in clean ethanol or methanol and dry with clean, oil free air.

**5.1.3.6** Carefully examine the mirror before testing. There must be no oxide.

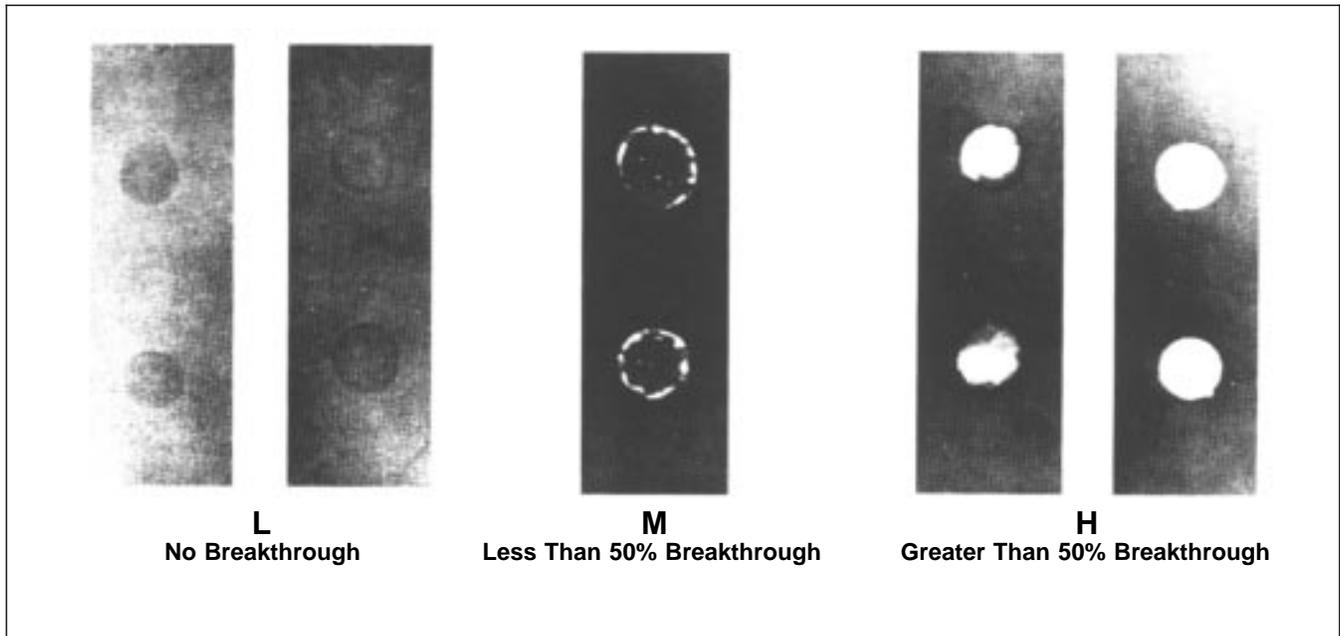
## 5.2 Test

**5.2.1** Place the copper mirror test panel on a flat surface, mirror side up, and protect from dust and dirt at all times.

**5.2.2** Place one drop of test flux or extract to be tested (approximately 0.05 ml) on each copper mirror test panel. Do not allow the dropper to touch the test panel.

**5.2.3** Solder-paste shall be applied directly to the mirror

Number <b>2.3.32</b>	Subject <b>Flux Induced Corrosion (Copper Mirror Method)</b>	Date <b>1/95</b>
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**Figure 1 Flux type classification by copper mirror test**

without scratching the copper mirror, with a volume approximating a 0.5 mm thickness and 8 mm diameter. (It has been determined that significant variations from this quantity have little effect for most materials.)

**5.2.4** Immediately also place one drop of the control standard flux adjacent to the test flux. Do not allow drops to touch.

**5.2.5** Place test panels in a horizontal position in the dust free cabinet at  $23 \pm 2^\circ\text{C}$  and  $50 \pm 5\%$  relative humidity for  $24 \pm 1/2$  hours.

**5.2.6** At the end of the 24 hour period, remove the test panels and remove the test flux and control standard fluxes by immersion in clean 2-propanol.

### 5.3 Evaluation

**5.3.1** Carefully examine each test panel for possible copper removal or discoloration.

**5.3.2** If there is any complete removal of the copper film as evidenced by the background showing through the glass, the test flux has failed the L category. Complete removal of the copper only around the perimeter of the drop defines the flux as M. Complete removal of the copper places the flux in the H category. (See Figure 1).

**5.3.3** If the control flux fails, repeat the entire test, using new copper mirror test panels.

**5.3.4** Discoloration of the copper film due to a superficial reaction or only a partial reduction of the copper film thickness is not considered a failure.

**5.3.5** A number of chemicals can cause failure of copper mirror: free halides, stronger organic and inorganic acids and free amines.

### 6.0 Notes

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.

### 6.2 Sources for prepared copper mirrors

**6.2.1** Evaporated Metal Films, Inc., Ithaca, NY 14850.

**6.2.2** Clausing PA Co., 8038 Monticello Ave., Skokie, IL, 708/267-3399



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.33</b>	
Subject <b>Presence of Halides in Flux, Silver Chromate Method</b>	
Date <b>1/95</b>	Revision <b>C</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to determine the presence (if any) of chlorides and bromides in soldering flux by visual examination after placement on test paper.

**2.0 Applicable Documents** None

**3.0 Test Specimen** A minimum of 100 ml of liquid flux, a representative container of solder paste, reflowed solder paste flux, extracted solder preform flux or extracted flux-cored wire.

#### 4.0 Apparatus and Reagents

**4.1** Six pieces of silver chromate test paper 51 mm x 51 mm

**4.2** 0.25L of reagent grade 2-propanol.

#### 5.0 Procedures

##### 5.1 Preparation

a. The silver chromate paper is extremely light sensitive and must be stored in a closed container away from light until used for testing.

b. To avoid contamination, the paper must be handled with forceps and must never be touched with bare hands.

##### 5.2 Test for Liquid Flux or Flux Extract Solution

**5.2.1** Place one drop of test flux or flux extract (approximately 0.05 ml) on each piece of silver chromate test paper. Allow the droplet to remain on each test paper for a minimum of 15 seconds.

**5.2.2** After the 15 seconds, immediately immerse each test paper in clean 2-propanol to remove the residual organic materials.

**5.2.3** Allow each test paper to dry for 10 minutes, then examine for color change.

##### 5.3 Test for Paste Flux or Solder Paste Flux as Obtained from the Supplier

**5.3.1** Clean a glass microscope slide with 2-propanol and air dry.

**5.3.2** Moisten a piece of silver chromate reagent paper of suitable size with deionized water.

**5.3.3** Apply the wet paper to the glass slide and remove the excess water with blotting paper.

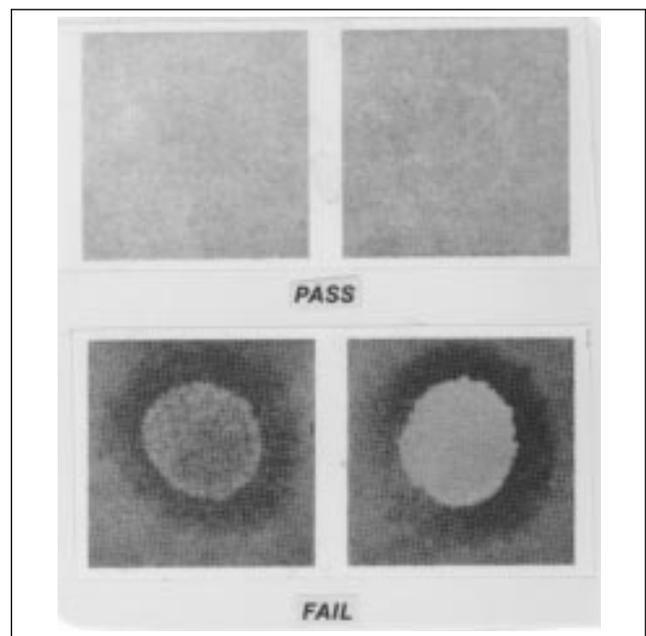
**5.3.4** Using a spatula, apply a thin coating of the paste flux or solder paste flux directly to the moist reagent paper.

**5.3.5** Allow the paste flux or solder paste flux to remain in contact with the paper for 1 minute, then remove the flux with 2-propanol without disturbing the paper.

**5.4 Evaluation** Carefully examine each test sheet for possible color change. A change to off-white or yellow-white indicates the presence of chlorides or bromides. See Figure 1.

**5.4.1 Interferences** A number of chemicals besides free halides may cause test failures. (Representative examples are, but are not limited to, amines, cyanides, isocyanates, etc.)

**5.4.2** Certain acidic solutions may react with the reagent paper to provide a color change similar to that obtained with chlorides and bromides. When a color change is observed, it



**Figure 1 Chlorides and/or bromides test results**

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Number <b>2.3.33</b>	Subject <b>Presence of Halides in Flux, Silver Chromate Method</b>	Date <b>1/95</b>
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is advisable to check the acidity of the affected area by means of a pH indicating paper. If pH values of less than 3 are obtained, the presence of chlorides and bromides should be verified by other analytical means.

## **6.0 Notes**

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.

## **6.2 Source For Silver Chromate Test Paper**

**6.2.1** Quantek, PO Box 136, Lyndhurst, NJ 07071, (201) 935-4103



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.34</b>	
Subject <b>Solids Content, Flux</b>	
Date <b>1/95</b>	Revision <b>B</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to determine the residual solids content of the liquid flux after evaporation of the volatile chemicals from within the liquid flux; typically 15% by weight minimum.

**2.0 Applicable Documents** None

**3.0 Test Specimen** A minimum of 6 grams by weight per test of liquid flux or flux extracted from solder paste, solder preforms or flux-cored wire.

## 4.0 Apparatus and Reagents

1. A circulating air drying oven capable of maintaining  $85 \pm 5^\circ\text{C}$ .
2. An analytical balance capable of weighing to the 4th decimal of a gram.
3. Glass pipettes
4. Glass petri dish, 30 ml capacity
5. Silica gel desiccant, or equivalent, in a glass desiccator
6. Analytical balance capable of 0.001 g. Carry out the following procedures in triplicate.

## 5.0 Procedures

### 5.1 Preparation

**5.1.1** Dry 3 empty glass petri dishes in the drying oven, then cool in the desiccator to room temperature.

**5.1.2** Weigh each dish to the nearest 0.001 gram.

### 5.2 Test

**5.2.1** Pipette approximately 6 grams (see 6.1) by weight of test flux specimen into each specimen dish and weigh to the nearest 0.001 gram.

**5.2.2** Heat in the drying oven for one hour, then reweigh after allowing the sample to come to room temperature.

**5.2.3** Repeat heating and drying procedure until the weight is constant to within 0.005 gram.

**5.3 Evaluation** Calculate the residual solids as follows:

$$\frac{100 \times m_2}{m_1}$$

where:

$m_2$  = the mass of residual after drying, in grams

$m_1$  = the mass of original test flux specimen, in grams

## 6.0 Notes

**6.1 Sample Size** Larger sample sizes may be required to obtain accurate data on low solids (<15%) fluxes.

**6.2 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.35</b>	
Subject <b>Halide Content, Quantitative (Chloride &amp; Bromide)</b>	
Date <b>1/95</b>	Revision <b>B</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to determine the halide content of fluxes attributable to chlorides and bromides. The halide content is reported as the weight percentage of chloride to the solid (non-volatile) portion of the flux or as milliequivalent per gram of flux solids. A sample of flux or flux extract is tritrated to an end-point and the percentage chloride or meq/g of halides is calculated.

**2.0 Applicable Documents:** None

**3.0 Test Specimen:** A minimum of 100 ml of liquid flux, 10-50 ml flux extract of known solids content from solder paste, solder preforms or flux-cored wire.

#### 4.0 Apparatus and Reagents:

- (1) Hot plate with magnetic stirrer.
- (2) Analytical balance capable of reading to 0.001 g.
- (3) Pipettes
- (4) Burettes
- (5) 100 ml beakers, Pyrex
- (6) 125 ml separatory funnel
- (7) 125 ml Erlenmeyer flasks
- (8) 1000 ml volumetric flasks
- (9) Reagents
  - a. 0.1N silver nitrate, standardized: dissolve 17.000 g silver nitrate in deionized water and dilute to 1000 ml in a volumetric flask.
  - b. 1M sodium hydroxide: 40.0 g of sodium hydroxide diluted to 1000 ml with deionized water in a volumetric flask.
  - c. 0.2M nitric acid: add 12.6 ml concentrated (16M) nitric acid to deionized water and dilute to 1000 ml in a volumetric flask.
  - d. 1M Potassium chromate: 194 g diluted to 1000 ml using deionized water in a volumetric flask.
  - e. 0.03M phenolphthalein solution. (Reagent Grade)
  - f. Chloroform (Reagent Grade)
  - g. Deionized water

## 5.0 Procedures

### 5.1 Sample Preparation

#### 5.1.1 For Rosin/Resin Fluxes

**5.1.1.1** In a tared 100 ml beaker, accurately weigh about 3-5 g of flux sample on an analytical balance.

**5.1.1.2** Quantitatively transfer the flux sample to a 125 ml separatory funnel using three 10 ml aliquots of chloroform.

**5.1.1.3** Add 15 ml of deionized water to the funnel and shake the funnel for 10 seconds.

**5.1.1.4** Allow the funnel to stand until the layers completely separate.

**5.1.1.5** Draw off the bottom (chloroform) layer into a beaker and save for the next extraction.

**5.1.1.6** Transfer the top (water) layer to a 125 ml Erlenmeyer flask.

**5.1.1.7** Transfer the chloroform layer from the beaker to the funnel and repeat the extraction with 15 ml of water two more times, each time adding the water extract portion to the flask.

**5.1.1.8** Heat the water extract in the Erlenmeyer flask using a steam bath to expel any chloroform which may be present.

**5.1.1.9** Do not heat above 80°C. Allow for solution to cool to room temperature.

#### 5.1.2 For Organic and Inorganic Fluxes

**5.1.2.1** In a tared 125 ml Erlenmeyer flask, accurately weigh about 3-5 g of flux sample on an analytical balance.

**5.1.2.2** Add 50 ml deionized water.

**5.1.2.3** Add 2 drops of 0.03 M phenolphthalein solution to the Erlenmeyer flask (from step 5.1.1.9 or 5.1.2.2).

**5.1.2.4** Add 1 M sodium hydroxide until the solution turns red. Add 0.2 M nitric acid dropwise until the red color is just completely discharged.

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**5.1.2.5** Dilute to about 60 ml with deionized water.

**5.1.2.6** Add 6 drops of 1 M potassium chromate and titrate with standardized 0.1N silver nitrate to the red-brown end point.

**5.1.3** For extracted preforms and extracted cored-wire follow the procedure in paragraph 5.1.1 if the extract contains a rosin or resin; follow the procedure in paragraph 5.1.2 if the extract is water soluble.

## 5.2 Calculations

**5.2.1** Calculate the percentage of halides as chloride based on flux solids content, using the following formula:

$$\text{Halides, as \% chlorides} = \frac{3.55 \text{ VN} \times 100}{\text{mS}}$$

**5.2.2** Calculate halides milliequivalent per gram of flux solids (non volatiles) using the following formula:

$$\text{Halides, meq/g solids} = \frac{\text{V} \times \text{N} \times 100}{\text{mS}}$$

Where:

V is the volume of 0.1N silver nitrate in ml.

N is the normality of silver nitrate solution.

m is the mass (weight) of flux sample in gram.

S is the percentage of solids (non-volatiles) of the flux.

## 6.0 Notes

**6.1 Safety:** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.35.1</b>	
Subject <b>Fluorides By Spot Test, Fluxes—Qualitative</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to determine the presence (if any) of fluoride(s) in soldering flux by visual examination after placement of a drop of liquid test flux in a zirconium - alizarin purple lake.

**2.0 Applicable Documents** None

### 3.0 Test Specimen

**3.1** A minimum of 10 ml of first article or production specimen of liquid flux, solderpaste flux, paste flux, or extracted flux from preform or wire furnished in a new clean glass container.

**3.2** For paste flux or solder paste flux dilute the sample in 2-propanol or another suitable solvent.

Note: The solvent used in dilution/extraction must be water miscible.

### 4.0 Apparatus and Reagents

#### 4.1 Apparatus

**4.1.1** White spot plate

**4.1.** Glass droppers

**4.1.** Glass rods

#### 4.2 Reagents:

**4.2.1** Zirconium nitrate solution: 0.05g dissolved in 50 ml of deionized water.

**4.2.2** Sodium alizarin sulfate solution: 0.05g dissolved in 50 ml of deionized water.

### 5.0 Procedure

#### 5.1 Preparation

**5.1.1** Prepare a fresh zirconium - alizarin purple lake in three spots of the white spot plate by adding 1 drop each of zirconium nitrate and sodium alizarin sulfate solutions.

#### 5.2 Test

**5.2.1** Add one drop of the test flux to each of the spots.

**5.2.2** Mix each spot with a clean glass rod.

**5.2.3** Examine for any color change.

**5.3 Evaluation** A change in color of the lake from purple to yellow is an indication of the presence of fluoride(s).

### 6.0 Notes

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.3.35.2</b>	
Subject <b>Fluoride Concentration, Fluxes—Quantitative</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is used to determine the concentration of fluorides in soldering flux. The fluoride content is reported as the weight percentage of fluoride to the non-volatile portion of the flux. Ion specific electrodes are used for the determination. This method uses Orion equipment and solutions.

**2.0 Applicable Documents** None

### 3.0 Test Specimen

**3.1** A minimum of 100 ml of first article or production specimen of liquid rosin flux or prepared flux solution furnished in a new clean glass container.

**3.2** For paste flux and solder paste flux 100 ml of the diluted material.

**3.3** For preform and cored wire 100 ml of the extracted material.

### 4.0 Apparatus and Reagents

#### 4.1 Apparatus

**4.1.1** Autocalibrating ion analyzer (Orion EA 920 or equivalent)

**4.1.2** Fluoride specific electrode (Orion 94-09-00 or equivalent)

**4.1.3** Double junction reference electrode (Orion 90-02 or equivalent)

**4.1.4** Magnetic stirrer and stir bars

**4.1.5** 250 ml and 500 ml beakers, glass or polypropylene

**4.1.6** Funnel stand and funnel

**4.1.7** 50 ml graduated cylinders

**4.1.8** 100 ml and 1000 ml volumetric flasks

**4.1.9** 10 ml and 100 ml pipets

**4.1.10** Analytical balance capable of 0.001 g

### 4.2 Reagents

**4.2.1** Deionized water

**4.2.2** Sodium fluoride, reagent grade

**4.2.3** Chloroform, reagent grade

**4.2.4** Orion TISAB II #940909 or equivalent

**4.2.5** Whatman #41 ashless filter paper

### 5.0 Procedure

#### 5.1 Sample Preparation—Water Soluble Flux

Depending on the expected concentration of fluoride, use the following amount of sample and deionized water.

ppm Fluoride	Sample ml	DI Water ml
0.1–1.0	25	50
1.0–10.0	5	50

**5.1.1** Tare the beaker and add sample, record the weight of the flux to 0.001 gram

**5.1.2** Add DI water and mix with stirrer for 10 minutes. Stop mixing and allow to stand for 5 minutes

**5.1.3** Pour solution through filter paper in funnel into a clean beaker. Allow sample to drain completely

#### 5.2 Sample Preparation—Non-water Soluble Flux

**5.2.1** Depending on the expected concentration of fluoride, use the following amount of sample and chloroform.

ppm Fluoride	Sample ml	Chloroform ml
0.1–1.0	25	25
1.0–10.0	5	5

**5.2.2** Tare the beaker and add sample, record the weight of the flux to 0.001 gram

**5.2.3** Add chloroform and 50 ml DI water and mix with stirrer for 10 minutes. Stop mixing and allow to stand for 5 minutes.

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**5.2.4** Carefully decant the water solution through filter paper in funnel into a clean beaker. Allow sample to drain completely.

### 5.2.5 Standard Solutions

**5.2.5.1** 10 ppm fluoride (0.001%)—This standard can be purchased ready-to-use from Orion, catalog #040908. Alternatively, a solution can be prepared. Weigh 0.0221 gram (dried to 140°C) reagent grade NaF. Add 500 ml DI water. Dilute to one liter in a volumetric flask with Orion catalog #940909 TISAB (total ionic strength adjustment buffer).

**5.2.5.2** 1.0 ppm fluoride (0.0001%)—This standard can be purchased ready-to-use from Orion, catalog #040906. Alternatively, a solution can be prepared. Weigh 0.0023 gram (dried to 140°C) reagent grade NaF. Add 500 ml DI water. Dilute to one liter in a volumetric flask with Orion catalog #940909 TISAB.

**5.2.5.2.1** Another method is to dilute 100 ml 10 ppm fluoride standard with 450 ml DI water and dilute to one liter in a volumetric flask with Orion #940909 TISAB. 0.1 ppm fluoride (0.00001%)—This standard is prepared by diluting 100 ml, 1.0 ppm fluoride standard with 450 ml DI water and dilute to one liter in a volumetric flask with Orion catalog #940909 TISAB.

**5.2.5.2.2** Another method is to dilute 10 ml, 10 ppm fluoride standard with 495 ml DI water and dilute to one liter in a volumetric flask with Orion catalog #940909 TISAB.

### 5.2.6 Double Reference Electrode Filling Solutions

Inner chamber filling solution (green). Solution saturated with AgCl, Orion catalog #900002.

Outer chamber filling solution. 10% KNO<sub>3</sub> - Orion catalog #900003.

**5.3 Test** For accurate results, allow all samples and standards to reach the same temperature before attempting a measurement. Stir the samples and standards while a measurement is being made.

Prepare two standards that differ in concentration by a factor of 10. The standards should bracket the expected sample concentration. Select concentration mode and STD 1 function. Significant digit selection is accomplished by pressing the X10 key the required number of times.

Dilute 50 ml of the two standard solutions, each with 50 ml of DI water.

While stirring with a magnetic stirrer, immerse electrodes in the more dilute standard solution. Adjust the display using arrows or X10 keys to the correct value. When “Ready” illuminates, press “Enter.” The display automatically proceeds to “STD 2.”

Remove and rinse electrodes with DI water. Immerse electrodes in the more concentrated standard solution. Adjust the display to read the correct value for STD 2. When “Ready” illuminates, press “Enter.” The display automatically proceeds to “Sample” function.

To the sample prepared per instructions for water soluble flux, add 25 ml TISAB. While stirring, immerse electrodes. When display indicates “Ready,” record indicated value.

**5.4 Evaluation** The indicated value is equal to the parts per million (ppm) fluoride. To obtain weight % of fluoride as percentage of the non-volatile content, use the following formula:

$$\text{Fluoride \% of Solids} = \frac{I}{(MS)100}$$

Where:

I is indicated value in ppm

M is mass of the flux sample in grams

S is the percentage of solids (non-volatile chemicals) of the flux

### 6.0 Notes

**6.1 Standardization and Limits** Restandardization should be done every few hours to compensate for electrode drift.

Reproducibility is typically ±2% obtainable with hourly recalibration. Temperature fluctuation, drift and noise will limit reproducibility.

Detection limits are 0.02 ppm minimum and no known maximum. A list of possible interfering ions has not yet been determined for this electrode method.

**6.2 Safety** Operator should be trained and familiar with the hazards inherent to the chemicals being used and analyzed. Proper personal safety equipment, such as safety glasses, gloves and splash apron, and adequate ventilation should be used.

Using the same batch of DI water for dilutions will eliminate the need to run blanks.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.4.14.2</b>	
Subject <b>Liquid Flux Activity, Wetting Balance Method</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test prescribes the recommended test method for assessing the activity of liquid fluxes using a wetting balance.

## 2.0 Applicable Documents

ISO 1634

## 3.0 Test Specimen

**3.1** The test specimen shall be a copper coupon complying with ISO 1634-CU-ETP Condition HA. The width shall be 6.0 ±0.25 mm wide; the length should be 25.0 ± 1 mm long or as appropriate to the test equipment. The thickness shall be 0.5 ±0.05 mm.

## 4.0 Apparatus and Reagents

### 4.1 Apparatus

**4.1** A meniscus force measuring device (wetting balance) which includes a temperature-controlled solder pot containing solder maintained at 245 ±3°C. *Note:* Reaction rate is very

sensitive at this temperature.

Solder composition shall be Sn60/Pb40 or Sn63/Pb37.

**4.2** A chart recorder, data logger, or computer capable of recording force as a function of time with a minimum recorder speed of 10 mm/s.

**4.3** A mechanical dipping device as shown in Figure 1 shall be used. This device shall be present to produce an immersion and emersion rate of 20–25 mm per second to a depth of 6.0 ±0.1 mm and a dwell time of 5.0 ±0.5 seconds.

## 5.0 Procedure

### 5.1 Preparation

**5.1.1** The test coupon should be cleaned (degreased) by immersion in a suitable solvent, then cleaned using a 10±1% fluoroboric acid dip.

**5.1.2** The coupon shall then be washed with water and dried.

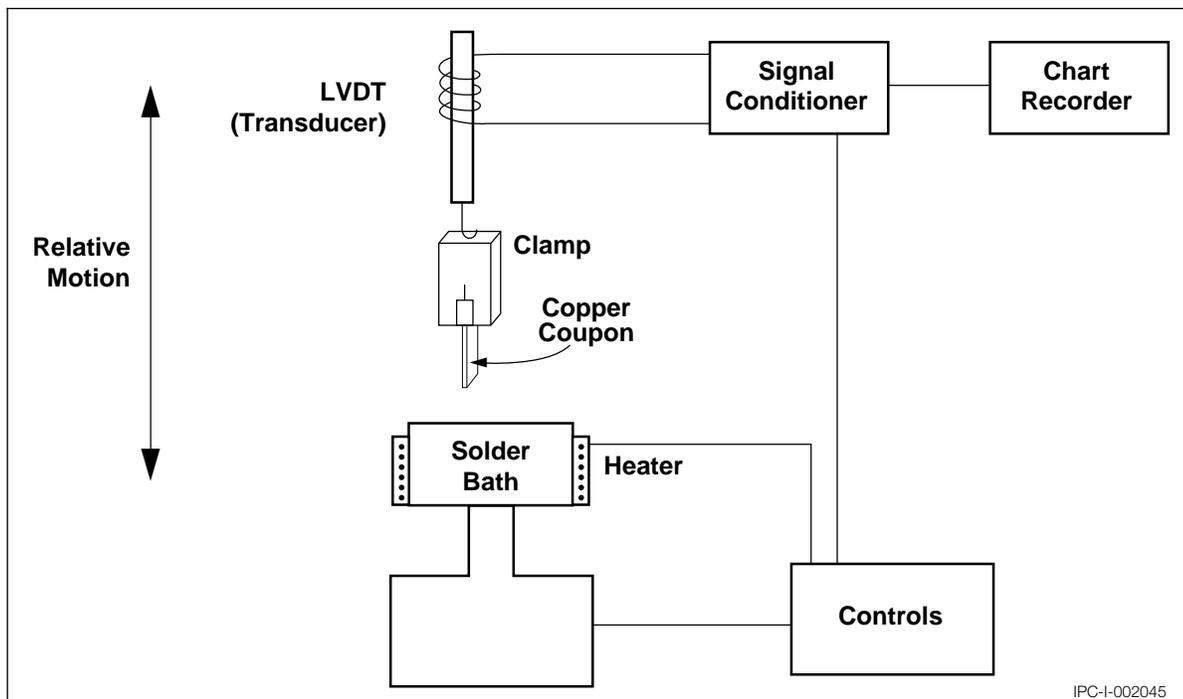


Figure 1 Wetting Balance Apparatus

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## 5.2 Test

**5.2.1** After mounting the specimen in a suitable holder, the coupon should be immersed in the liquid flux at room temperature to a minimum depth of 10 mm.

**5.2.2** Excess flux is to be immediately drained off by standing the specimen vertically on a clean filter paper for 1–5 seconds.

**5.2.3** After partial drying, it should be mounted in the test equipment.

**5.2.4** The surface of the molten solder shall be skimmed just prior to immersing the specimen in the solder.

**5.2.5** The specimen in its holder shall be held for approximately 10±1 second, 3 mm above the solder pot. The test shall be started and the specimen immersed only once using an immersion and emersion rate of 20–25 mm per second to a depth of 5.0 ±0.1 mm and a dwell time of 5.0 ±0.5 seconds.

**5.2.6** During the test, the wetting curve shall be recorded on a suitable device for use in the evaluation.

**5.3 Evaluation** Using the coupon as specified, fluxes which shall be evaluated for the following:

1. A wetting time ( $T_w$ ) for the wetting curve to cross the corrected zero axis after the start of the test. (See Figure 2)
2. A maximum wetting force,  $F_{max}$ , taken after correction for buoyancy (See 6.2).

**6.0 Note** This test method can be useful in requalifying materials that have exceeded the recommended shelf life. In addition, the method can help evaluate fluxing power prior to manufacturing operations on critical applications.

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.

**6.2 Correction for Buoyancy** For the wetting balance to obtain wetting force values that are relatable to one another, it is necessary to correct for the variability in specimen sizes, in particular width and thickness. This is done by correcting for the volume of the sample immersed in the solder. The following formula may be used to calculate the buoyant force correction:

$$P_b = \rho gV$$

$\rho$  = Density of solder @ 245°C (8.15g/cm<sup>3</sup>)\*

$g$  = Acceleration of gravity (9810 mm/s<sup>2</sup>)

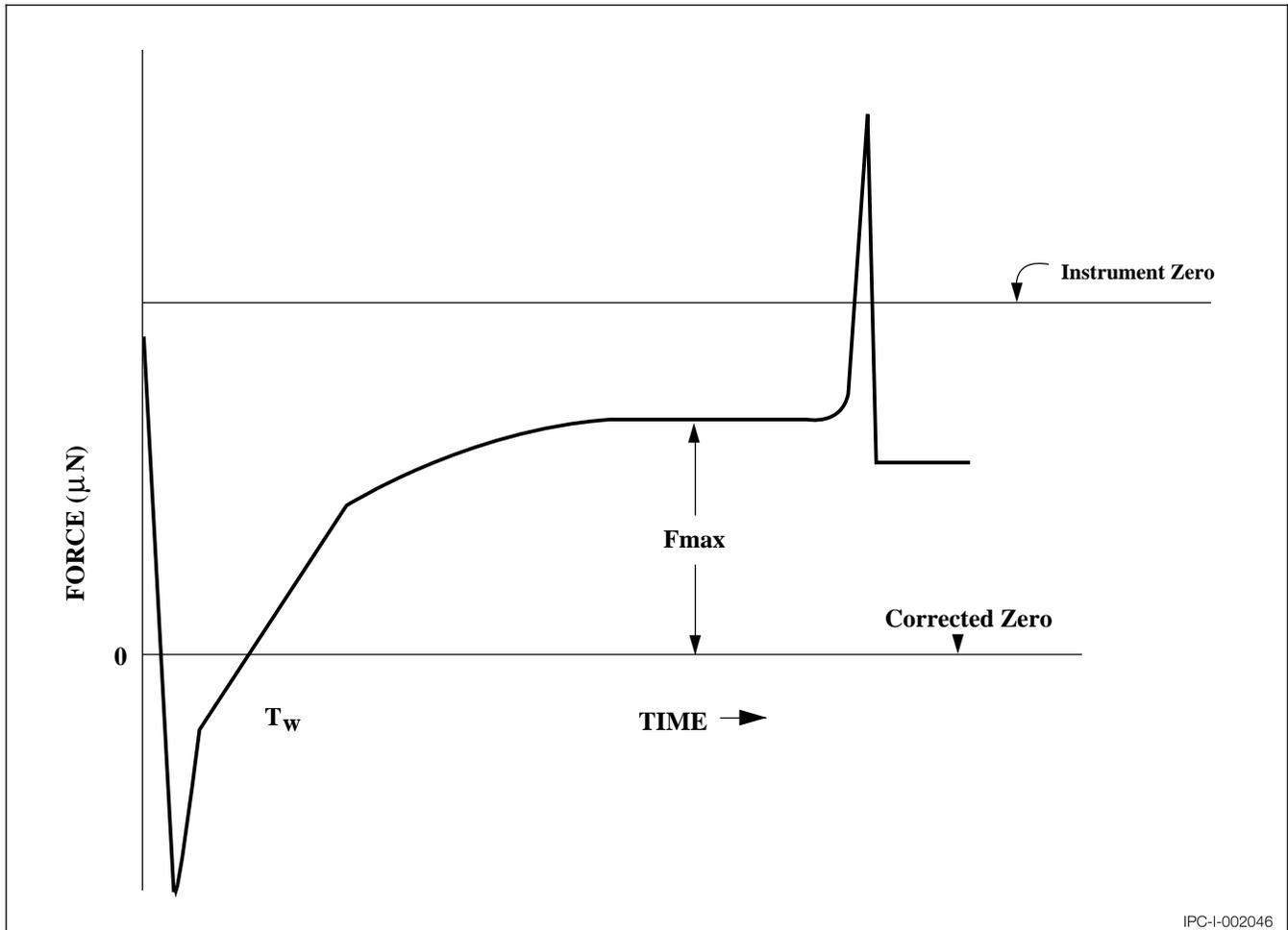
$V$  = Immersed volume of coupon (cm<sup>3</sup>)

= width x thickness x immersion depth

When the buoyancy force is calculated it should be used to correct the zero axis. This correction is required to obtain both the proper measurement of wetting times as well as wetting forces. All measurements of wetting times and wetting forces must be made from the corrected zero axis. In the case of an upright curve, the new corrected zero axis will be below the instrument zero.

\*For Sn60/Pb40 Alloy

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IPC-I-002046

**Figure 2 Wetting Balance Curve**

**Note:** The vertical force measured by the wetting balance is made up from two forces—the buoyancy force, and the wetting force caused by the contact angle changing from initial non-wetting to wetting.

The buoyancy force may be considered during the test, and is equal to the weight of the solder displaced, when the specimen is immersed into the solder.

The only changing force is the wetting force, caused by the changing contact angle, as the specimen solders.

The corrected zero (buoyancy) line is the force when the contact angle is 90°, or when the bath surface has returned to horizontal, having been initially depressed by the immersed sample.

The wetting balance curve is centered on the corrected zero (buoyancy) line since the only parameter that changes during the test is the contact angle.  $\theta$ . See equation 1:

$$F = \gamma \rho \cos \theta - g \rho v \quad \text{Equation 1}$$

$$\text{Buoyancy} = g \rho v \quad \text{Equation 2}$$

where

F = The measured force in micronewtons

$\gamma$  = Surface tension of molten solder (400  $\mu\text{N mm}^{-1}$ )

$\rho$  = Specimen perimeter in mm

$\theta$  = Contact angle

g = Gravitational acceleration ( $9.81 \times 10^{-3} \text{ mm—sec}^{-2}$ )

$\rho$  = Solder density (8000  $\mu\text{g mm}^{-3}$ )

v = immersed volume in  $\text{mm}^3$

The corrected zero line (buoyancy) is a fixed reference point from which the force measurements should be taken. This line should also be used as a reference point for any time measurements.

Altering the specimen dimensions changes the immersed volume and hence the buoyancy, and so alters the position of the corrected zero line; but the wetting curve still remains centered on this line. Similarly, any change in immersion depth will also alter the immersed volume, with the same effect on the buoyancy.

Although use of the corrected zero line will cancel small variations in the specimen immersed volume and the immersion depth, large changes will affect the rate of heat transfer into the specimen, which will affect both  $T_w$ , the time to recross the corrected zero (buoyancy) line and the time to reach F max.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.4.34.4</b>	
Subject <b>Paste Flux Viscosity – T-Bar Spindle Method</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is designed to measure the viscosity of paste flux.

**2.0 Applicable Documents** None

**3.0 Test Specimen** Enough paste flux to fill a container with a minimum diameter of 4 cm to a minimum depth of approximately 10 cm.

## 4.0 Apparatus and Reagents

**4.1** Brookfield RVT viscometer or equivalent with helipath stand and a TC spindle.

**4.2** Water bath capable of holding 25 +/-0.5°C.

**4.3** Stopwatch

**4.4** Spatula

## 5.0 Procedure

**5.1** Test

**5.1.1** Place container of paste flux in water bath at 25+/- 0.5°C.

**5.1.2** When medium has attained thermal equilibrium, place container under spindle so that it is at center of surface.

**5.1.3** Start the Brookfield at 5 revolutions per minute and start the Helipath stand on descend.

**5.1.4** Two minutes after the spindle has cut into the top surface of the medium, record the value. Check that spindle is not touching bottom of container.

**5.1.5** Remove spindle from the paste flux. Using spatula, stir the flux vigorously for 15 to 20 seconds and remeasure viscosity.

**5.2 Expression of Results** The viscosities are calculated from the values recorded after 2 minutes of medium penetration. Both stirred and unstirred results should be quoted.

## 6.0 Notes

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

**1.0 Scope** This test method will give an indication of activity of wave solder fluxes, core solder fluxes, and solder paste.

## 2.0 Applicable Documents

**ASTM B-36** Brass Plate, Sheet, Strip, and Rolled Bar

**J-STD-006** Requirements for Solder Wire

## 3.0 Test Specimen

**3.1** A minimum of 10 ml of first article or production specimen of liquid flux furnished in a new clean glass container.

**3.2** For paste flux and solder paste flux 10 ml of the diluted material (35%).

**3.3** For preform and cored wire 10 ml of the extracted material.

## 4.0 Apparatus and Reagents

**4.1** Five (5) replicates of 0.25 mm thick 70/30 brass (per ASTM-B-36 C2600 HO2) approximately 40 x 75 mm.

**4.2** Degreased steel wool #00.

**4.3** Sn60 1.5 mm diameter Type S solid wire solder per J-STD-006.

**4.4** Solder pot containing at least 2 kg of solder and no less than 25 mm in depth.

## 5.0 Test

### 5.1 Specimen Preparation

**5.1.1** Clean five (5) brass coupons with steel wool.

**5.1.2** Using a flat strip of brass, bend the opposite ends parallel to the curve of the metal coil to stiffen and flatten the test coupon.

**5.1.3** Cut a 30 mm length of solid wire solder

**5.1.4** Wrap the cut length of solder around a 3 mm mandrel.

Number <b>2.4.46</b>	
Subject <b>Spread Test, Liquid or Extracted Solder Flux, Solder Paste and Extracted Cored Wires or Preforms</b>	
Date <b>1/95</b>	Revision
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**5.1.5** Cut the coil into individual rings to make a preform of the solder.

## 5.2 Test

**5.2.1** Maintain solder pot at 260 +/-10°C.

**5.2.2** Place the preformed solder in the center of the test coupon.

**5.2.3** Place one drop (0.05ml) of flux in center of preform on the test coupon.

**5.2.4** Carefully place the coupon on the surface of the solder bath for 15 seconds.

**5.2.5** Remove the coupon in a horizontal position and place on a flat surface allowing the adhered solder to solidify undisturbed.

**5.2.6** Remove all flux residue with a suitable solvent.

## 5.3 Evaluation

**5.3.1** Measure the solder spread area by comparing to circles (pre-drawn) with areas similar to those listed in Table 1. The mean of the spread of all five samples tested is to be reported.

Table 1 is intended as an aid in defining areas in mm<sup>2</sup>.

**Table 1 Typical Spread Areas Defined in mm<sup>2</sup>**

Diameter in mm	Area in mm <sup>2</sup>
10.00	78.54
10.70	90.00
11.28	100.00

## 6.0 Notes

**6.1 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.6.1</b>	
Subject <b>Fungus Resistance Printed Wiring Materials</b>	
Date <b>1/95</b>	Revision <b>D</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** The fungus resistance test is used to determine the resistance of materials to fungi and to determine if such material is adversely affected by fungi under conditions favorable for their development, namely high humidity, warm atmosphere, and presence of inorganic salts.

**2.0 Applicable Documents** None

**3.0 Test Specimen** Specimens must be a minimum size of 50 mm x 50 mm with copper foil (if applicable) removed by etching using standard commercial practices.

## 4.0 Apparatus and Reagents

**4.1 Test Chamber** The autoclave shall be capable of maintaining 30°C and 95% relative humidity and an ultra violet (360 nm) source for subsequent decontamination. Provisions shall be made to prevent condensation from dripping on the test item. There shall be free circulation of air around the test item and the contact area of fixtures supporting the test item shall be kept to a minimum.

**4.2** Sterilizer

**4.3** Centrifuge

**4.4** pH Meter

**4.5** Colony Counter

**4.6** Incubator

**4.7** Dishwasher

**4.8** Petri Dishes

**4.9** Filter Paper

**4.10** Media Solutions

**4.11** Microorganism

**4.12** Atomizer, 15,000 ± 3000 spores

## 5.0 Procedures

### 5.1 Preparation of Test Media

**5.1.1 Mineral-Salts Solution** Prepare the solution to contain the following:

Potassium dihydrogen orthophosphate (KH <sub>2</sub> PO <sub>4</sub> ).....	0.7g
Potassium monohydrogen orthophosphate (K <sub>2</sub> HPO <sub>4</sub> ).....	0.7g
Magnesium sulfate heptahydrate (MgSO <sub>4</sub> 7H <sub>2</sub> O.....)	0.7g
Ammonium nitrate (NH <sub>4</sub> NO <sub>3</sub> ).....	1.0g
Sodium chloride (NaCl).....	0.005g
Ferrous sulfate heptahydrate (FeSO <sub>4</sub> 7H <sub>2</sub> O) .....	0.002g
Zinc sulfate heptahydrate (ZnSO <sub>4</sub> 7H <sub>2</sub> O).....	0.002g
Manganous sulfate monohydrate (MnSO <sub>4</sub> H <sub>2</sub> O).....	0.001g
Distilled water .....	1000 ml

Sterilize the mineral salts solution by autoclaving at 121°C for 20 minutes. Adjust the pH of the solution by the addition of 0.01 normal solution of NaOH so that after sterilization the pH is between 6.0 and 6.5. Prepare sufficient salts solution for the required tests.

**5.1.2 Purity of Reagents** Reagent grade chemicals shall be used in all tests. Unless otherwise specified, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

**5.1.3 Purity of Water** Unless otherwise specified, references to water shall be understood to mean distilled water or water of equal purity.

**5.1.4 Preparation of Mixed Spore Suspension** The following test fungi shall be used:

Description	ATCC
Aspergillus niger .....	9642
Chaetomium globosum .....	6205
Gliocladium virans .....	9645
Aureobasidium pullulans.....	9348
Penicillium funiculosum.....	9644

**5.1.5** Maintain cultures of these fungi separately on an appropriate medium such as potato dextrose agar. However, the culture of chaetomium globosum shall be cultured on

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Number <b>2.6.1</b>	Subject <b>Fungus Resistance Printed Wiring Materials</b>	Date <b>1/95</b>
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strips of filter paper on the surface of mineral salts agar. (Mineral salts agar is identical to mineral salts solution, but contains in addition 15.0 g of agar per liter).

**5.1.6** The stock cultures may be kept for not more than 4 months at  $6^{\circ}\pm 4^{\circ}\text{C}$ , at which time subcultures shall be made and new stocks shall be selected from the subcultures.

**5.1.7** If genetic or physiological changes occur, obtain new cultures as specified above. Subcultures used for preparing new stock cultures or the spore suspension shall be incubated at  $30^{\circ}\text{C}$  for 9 to 12 days or longer.

**5.1.8** Prepare a spore suspension of each of the five fungi by pouring into one subculture of each fungus, a 10-ml portion of a sterile solution containing 0.05 g per liter of a non-toxic wetting agent such as sodium dioctyl sulfosuccinate or sodium lauryl sulfate.

**5.1.9** Use a sterile platinum or nichrome inoculating wire to scrape gently the surface growth from the culture of the test organism.

**5.1.10** Pour the spore charge into a sterile 125-ml glass-stoppered Erlenmeyer flask containing 45 ml of sterile water and 50 to 75 solid glass beads, 5 mm in diameter.

**5.1.11** Shake the flask vigorously to liberate the spores from the fruiting bodies and to break the spore clumps.

**5.1.12** Filter the dispersed fungal spore suspension, through a 6 mm layer of glass wool contained in a glass funnel, into a sterile flask.

**5.1.13** This process should remove large mycelial fragments and clumps of agar which could interfere with the spraying process.

**5.1.14** Centrifuge the filtered spore suspension aseptically and discard the supernatant liquid.

**5.1.15** Resuspend the residue in 50 ml of sterile water and centrifuge. Wash the spores obtained from each of the fungi in this manner three times.

**5.1.16** Dilute the final washed residue with sterile mineral-salts solution in such a manner that the resultant spore suspension shall contain  $1,000,000 \pm 200,000$  spores per ml as determined with a counting chamber.

**5.1.17** Repeat this operation for each organism used in the test and blend equal volumes of the resultant spore suspension to obtain the final mixed spore suspension. The spore suspension may be prepared fresh each day or may be held at  $6^{\circ} \pm 4^{\circ}\text{C}$  for not more than 7 days.

**5.2 Viability of Inoculum Control** With each daily group of tests, place each of 3 pieces of sterilized filter paper, 25 mm x 25 mm square, on hardened mineral-salts agar in separate Petri dishes. Inoculate these with the spore suspension by spraying the suspension from a sterilized atomizer until initiation of droplet coalescence. Incubate these at  $30^{\circ}\text{C}$  at a relative humidity not less than 85% and examine them after 7 days of incubation. There shall be copious growth on all three of the filter paper control specimens. Absence of such growth requires repetition of the test.

### 5.3 Control Items

**5.3.1** In addition to the viability of inoculum control, known susceptible substrates shall be inoculated along with the test item to insure that proper conditions are present in the incubation chamber to promote fungus growth.

**5.3.2** The control items shall consist of cotton duck 8.25-ounce strips that are 5 cm, that have been dipped into a solution containing 10% glycerol, 0.1% potassium dihydrogen orthophosphate ( $\text{KH}_2\text{PO}_4$ ), 0.1% ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ), 0.025% magnesium sulfate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ), and 0.05% yeast extract (pH 5.3), and from which the excess liquid has been removed.

**5.3.3** The strips should be hung to air dry before being inoculated and placed into the chamber.

### 5.4 Inoculation of Test and Control Item

**5.4.1** Mount the test and control items on suitable fixtures or suspend from hangers. No cleaning of the test item shall be permitted for 72 hours prior to the beginning of the fungus test.

Equipment handling prior to and during the fungus test shall be accomplished without contamination of the equipment.

**5.4.2** Precondition the chamber and its contents at:  $30^{\circ}\text{C}$  and  $97 \pm 2\%$  relative humidity for at least 4 hours.

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**5.4.3** Inoculate the test and control items with the mixed fungus spore suspension (3.1.2) by spraying it on and into the test and control items (if not hermetically sealed) in the form of a fine mist from a previously sterilized atomizer or nebulizer. In spraying the test and control items, care should be taken to spray all surfaces which are exposed during use or maintenance. If the surfaces are nonwetting, spray until initiation of droplet coalescence. Incubation is to be started immediately following the inoculation.

### 5.5 Test Incubation of Test Items

**5.5.1** Incubate test items under cyclic temperature and humidity conditions to include 20 hours of relative humidity at  $95 \pm 5\%$  at an air temperature of  $30^\circ \pm 1^\circ\text{C}$  followed by 4 hours of 100% relative humidity at  $25^\circ \pm 1^\circ\text{C}$ .

**5.5.2** After 7 days, inspect the growth on the control items to be assured that the environmental conditions are suitable for growth. If inspection reveals that the environmental conditions are unsuitable for growth, the entire test shall be repeated.

**5.5.3** If the control items show satisfactory fungus growth, continue the test for a period of 28 days from the time of inoculation, or as specified.

### 5.6 Evaluation

**5.6.1** Report those specimens which were found to be nutrient to fungus growth.

**5.6.2** Corrosion should be noted separately from the fungus test results.

### 6.0 Notes

### 6.1 Source for Microorganisms

#### 6.1.1

American Type Culture Collection  
12301 Parklawn Drive  
Rockville, MD 20852 USA  
(301) 881-2600 TELEX: 908768 ATCC ROVE

### 6.2 Secondary Sources for Microorganisms

#### 6.2.1

Pioneering Research Division  
U.S. Army Natick Laboratories  
Natick, Massachusetts 01760

#### 6.2.2.

USDA Northern Regional Research Center  
1815 North University St.  
Peoria, IL 61604  
Contact: Dr. Stephen Peterson  
309-685-4011

**6.3** After evaluation, the materials and the test chamber must be decontaminated by exposure on all sides to ultraviolet rays (360 nm) for a minimum of two hours, or sprayed with a solution of 1:750 zephiran chloride solution. (One part zephiran chloride to 750 parts distilled water).

**6.4 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.6.3.3</b>	
Subject <b>Surface Insulation Resistance, Fluxes</b>	
Date <b>1/95</b>	Revision <b>A</b>
Originating Task Group <b>Flux Specifications Task Group (5-24a)</b>	

**1.0 Scope** This test method is to characterize fluxes by determining the degradation of electrical insulation resistance of rigid printed wiring board specimens after exposure to the specified flux. This test is carried out at high humidity and heat conditions.

## 2.0 Applicable Documents

**IPC-B-24** Surface Insulation Resistance Test Board

**IPC-A-600** Acceptability Guidelines

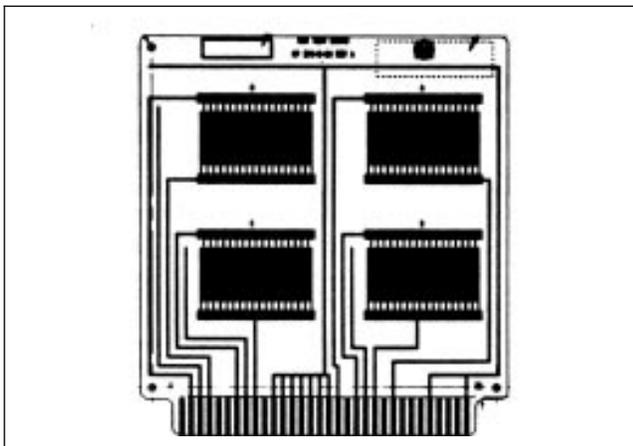
## 3.0 Test Specimens

**3.1 Comb Patterns** Use the IPC-B-24 test pattern which consists of four comb patterns per coupon. The individual comb, pictured in Figure 1, has 0.4 mm lines and 0.5 mm spacing. The test coupon shall be unpreserved bare copper metallization.

**3.2 Laminate** The laminate material for this test shall be FR-4 epoxy-glass.

## 4.0 Apparatus

**4.1** A clean test chamber capable of programming and recording an environment of 25 +10/-2°C to at least 85 ±2°C and 85% ±2% relative humidity. A salt solution and desiccator may be used to maintain humidity if a tight temperature control is maintained on the temperature of the chamber.



**Figure 1** Typical "Comb Pattern" (from IPC-B-24)

**4.2** A power supply capable of producing a standing bias potential of 45–50 volts DC with a tolerance of ±10%.

**4.3** A resistance meter capable of reading high resistance ( $10^{12}$  ohms) with a test voltage of 100 volts or an ammeter capable of reading  $10^{-10}$  amps in combination with 100 volts DC power supply.

**4.4** Three 2000 ml beakers

**4.5** Exhaust ventilation hood.

**4.6** Metal tongs.

**4.7** Soft bristle brush

**4.8** Deionized or distilled water (2 megohm-cm, minimum resistivity recommended).

**4.9** Drying oven capable of maintaining at least 50°C.

## 5.0 Test

**5.1 Test Conditions** All fluxes will be tested at 85 ±2°C, 85±2% relative humidity for 168 hours.

**5.2 Specimen Preparation** There shall be 3 test coupons for each liquid flux to be tested in the cleaned state (see Table 1, A). When testing liquid fluxes which are intended to remain in the uncleaned state, six test coupons are required. Three uncleaned test coupons shall be wave soldered pattern side down (Table 1, B) and three shall be wave soldered pattern side up (Table 1, C).

Solder paste coupons shall be reflowed pattern side up and either cleaned (Table 1, D) or not cleaned (Table 1, E). In addition, there shall be at least 2 unprocessed control coupons for comparison purposes (Table 1, F).

**5.2.1** Positive, permanent and non-contaminating identification of test specimens is of paramount importance. (For example, a vibrating scribe.)

**5.2.2** Visually inspect the test specimens for any obvious defects, as described in IPC-A-600. If there is any doubt about the overall quality of any test specimen, the test specimen should be discarded.

Number <b>2.6.3.3</b>	Subject <b>Surface Insulation Resistance, Fluxes</b>	Date <b>1/95</b>
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**Table 1 Coupons for SIR Testing**

Sample Group	Flux/Solder	Clean	Number of Coupons
A	yes	yes	3
B	yes	no	3
C	yes	no	3
D	yes	yes	3
E	yes	no	3
F	no	no	2

A = Pattern down/clean

B = Pattern down/no clean

C = Pattern up/no clean

D = Solder paste/reflow/clean

E = Solder paste/reflow/no clean

F = Control (precleaned, unprocessed)

**5.2.3** Clean the test coupon with deionized or distilled water and scrub with a soft bristle brush for a minimum of 30 seconds. Spray rinse thoroughly with deionized or distilled water. Rinse cleaned area thoroughly with fresh 2-propanol.

An alternative cleaning method is to place the test coupon in an ionic contamination tester containing 75% 2-propanol, 25% deionized water and process the solution until all ionics have been removed.

During the remainder of the specimen preparation, handle test specimens by the edges only, or use non-contaminating rubber gloves.

**5.2.4** If boards are to be stored before treatment, place the boards in Kapak bags or other contamination-free containers and close bags (do not heat seal). (Kapak bags are available from Fischer, VWR and other distributors).

### 5.3 Procedure

**5.3.1 Sample Preparation** Flux application and soldering.

**5.3.1.1 Liquid Flux or Flux Extract** Coat the comb pattern with a thin coating of the liquid flux or flux extract under test.

**5.3.1.1.1** The test coupons shall be exposed to solder by floating the fluxed comb patterns of the test specimens face down on the solder pot at 245-260°C for 4 ±1 seconds. Wave solder of comb patterns face down at 245-260°C and a conveyor speed with a contact time of 3 ±1 seconds. For fluxes to be tested in the uncleaned state, a second set of comb patterns shall be fluxed and floated pattern up on the solder pot or passed pattern up over the solder wave.

**5.3.1.2 Solder Paste** Stencil print the solder paste on to the comb pattern using a 0.2 mm thick stencil (the IPC-A-24 artwork contains the stencil design).

**5.3.1.2.1** The samples shall be run through a reflow soldering process using the temperature profile recommended by the vendor.

### 5.3.2 Cleaning of Samples

**5.3.2.1** After exposure to flux and solder, samples to be tested *in an uncleaned state* shall be evaluated as in 5.3.3 through 5.4.1.

**5.3.2.2** After exposure to flux and solder, samples to be tested in the cleaned state shall be cleaned using one of the procedures listed below. The cleaning parameters shall be reported in the Qualification Test Report (Appendix A).

**5.3.2.2.1** The samples to be cleaned shall be cleaned with an appropriate environmentally safe solvent or aqueous cleaning medium. The use of a commercial in-line or batch cleaner is preferred. If this is not available, the following laboratory cleaning process shall be followed.

Three samples shall be cleaned (within 30 minutes or less) after soldering. For solvent or aqueous detergent cleaning, three 2000 ml beakers each containing 1000 ml of solvent shall be used such that one beaker serves as the primary cleaning stage and the other two are used for rinsing purposes. Each test coupon shall be agitated in each beaker for 1 minute. In the case of aqueous detergent, one beaker shall contain the cleaning agent and the remaining beakers shall contain deionized water for rinsing purposes. After the cleaning procedure is complete, samples are dried for 2 hours at 50°C. Following cleaning, the specimens shall be tested as outlined in 5.3.3 through 5.4.1.

**5.3.3 Preparation of Samples for Chamber** Visually inspect all combs and discard any combs with bridging of conductors. Use water white rosin to solder teflon-insulated wires to the connection points of the specimens. Do not attempt to remove the flux residues. Connectors may be used in lieu of soldering wires but are not recommended. In the event of a dispute, the samples with soldered wires shall be used as a referee.

**5.3.4** Place the specimens in the environmental chamber in a vertical position such that the air flow is parallel to the direction of the board in the chamber. Set the chamber temperature at 85±2°C and humidity at 20% RH and allow the oven to stabilize at this temperature for 3 hours. Then, slowly ramp the humidity to 85±2% over a minimum 15 minute period.

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Allow the specimens to come to equilibrium for at least 1 hour before applying the bias voltage to begin the test. If a salt solution and dessicator are used for humidity, specimens shall be held for 24 hours before beginning the test.

**5.3.5** Connect the 45–50v DC voltage source to the specimen test points to apply the bias voltage to all specimens.

#### **5.4 Measurements**

**5.4.1** Measurements shall be made with test specimens in the chamber under the test conditions of temperature and humidity at 24, 96 and 168 hours. To take these measurements, the 45 - 50v DC bias voltage source must be removed from the test specimen and a test voltage of -100v DC shall be applied. (Test voltage polarity is opposite the bias polarity.)

#### **5.5 Evaluation**

**5.5.1** Each comb pattern on each test specimen shall be evaluated by the insulation resistance values obtained at 96 and 168 hours. If the control coupon readings are less than 1000 megohms, a new set of test coupons shall be obtained and the entire test repeated. The reading at 24 hours may fall below the required value provided that it recovers by 96 hours. Any reason for deleting values (scratches, condensation, bridged conductors, outlying points, etc., must be noted).

**5.5.2** All specimens shall also be examined under a 10x to 30x microscope using backlighting within 24 hours of completing the testing. If the coupons are to be held longer, they shall be placed in Kapak or other noncontaminating container and stored in a dessicator. All samples must be evaluated within 7 days. If dendritic growth or corrosion is observed, it shall be determined if the dendrite spans 25% or more of the original spacing. This latter condition will constitute a failure. It should be determined whether dendritic growth is due to condensation from the chamber (see paragraph 6.1).

**5.5.3** Rejection of results for more than 2 combs for a given condition shall require the test to be repeated.

#### **6.0 Notes**

**6.1** If condensation occurs on the test specimens in the environmental chamber while the samples are under voltage, dendritic growth will occur. This can be caused by a lack of sufficient control of the humidification of the oven. Water spotting may also be observed in some ovens where the air flow in the chamber is from back to front. In this case, water con-

densation on the cooler oven window can be blown around the oven as microdroplets which deposit on test specimens and cause dendritic growth if the spots bridge the distance between two electrified conductors. Both of these conditions must be eliminated for proper testing.

**6.2** IPC-B-24 test board artwork and electronic data is available from IPC.

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Number <b>2.6.3.3</b>	Subject <b>Surface Insulation Resistance, Fluxes</b>	Date <b>1/95</b>
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**Appendix A  
Qualification Test Report**

<b>I.D. Number:</b>			
<b>Flux Classification:</b>		<b>Date of Manufacture:</b>	
<b>Manufacturer's Identification:</b>		<b>Original Use by Date:</b>	
<b>Manufacturer's Batch Number:</b>		<b>Requalified Use by Date:</b>	
<b>Manufacturer's Original:</b>		<b>Original:</b>	
<b>Date Original Qualification Tests Completed:</b>		<b>Date Requalification Tests Completed:</b>	
<b>Tested By:</b>	<b>Pass: _</b>	<b>Tested By:</b>	<b>Pass: _</b>
<b>Witnessed By:</b>	<b>Fail: _</b>	<b>Witnessed By:</b>	<b>Fail: _</b>
<b>Certification Test</b>	<b>IPC-TM-650 Method</b>	<b>Test Requirement</b>	<b>Result Pass/Fail/NA</b>
Copper Mirror	2.3.32		
<i>Halides Qualitative</i>			
Silver Chromate	2.3.33		
Fluoride Spot	2.3.35.1		
<i>Halides Quantitative</i>			
Chloride, Bromide	2.3.35, 2.3.28		
Fluoride	2.3.35.2		
Nonvolatile Determination	2.3.34		
Total as Chloride			
Corrosion	2.6.15		
SIR	2.6.3.3	Cleaned Not Cleaned	
Fungus (optional)	2.6.1		
<i>Cleaning Procedure for Flux Characterization</i>			
Cleaning Material _____			
Cleaning Equipment _____			
Cleaning Process Parameters _____			
_____			
_____			



# IPC-TM-650 TEST METHODS MANUAL

Number <b>2.6.15</b>	
Subject <b>Corrosion, Flux</b>	
Date <b>1/95</b>	Revision <b>B</b>
Originating Task Group <b>Flux Specifications Task Group (5-22d)</b>	

**1.0 Scope** This test method is designed to determine the corrosive properties of flux residues under extreme environmental conditions. A pellet of solder is melted in contact with the test flux on a sheet metal test piece. The solder is then exposed to prescribed conditions of humidity and the resulting corrosion, if any, is assessed visually.

## 2.0 Applicable Documents

**IPC-TM-650** Test Methods Manual

**2.3.34** Solids Content, Flux

**British Standard Institute BS5625** Specification of Purchasing Requirements and Methods of Test for Fluxes for Soft Soldering

**3.0 Test Specimen** At least 0.035 g of flux solids, 1 g solder paste, 1 g wire, or 1 g preform with an equivalent amount of solids. Flux solids are defined as the residue from the solids content, fluxes test described in IPC-TM-650, Test Method 2.3.34. All solvent must have been evaporated from the specimen in a chemical fume hood.

## 4.0 Apparatus and Reagents

1. Solder pot
2. Humidity chamber capable of achieving  $50 \pm 2^\circ\text{C}$  and  $95 \pm 2\%$  relative humidity.
3. Air circulating drying oven
4. Microscope having 20X minimum
5. Chemicals: All chemicals must be reagent grade and water must be distilled or demineralized:
  - a. Ammonium persulphate
  - b. Sulfuric acid, % v/v
  - c. Degreasing agent, Acetone, Toluene, or Petroleum ether.
6. Analytical balance capable of weighing 0.001 g

## 5.0 Procedures

### 5.1 Preparation

#### 5.1.1 Chemicals

**5.1.1.1** Ammonium persulphate (25% m/v in 0.5% v/v sulfuric acid). Dissolve 250 g of ammonium persulphate in water and add cautiously 5 ml of 5% sulphuric acid (relative density 1.84). Mix, cool, dilute to 1 liter and mix. This solution should be freshly prepared.

**5.1.1.2** Sulfuric acid (5% v/v). To 400 ml of water cautiously add 50 ml of sulfuric acid (relative density 1.84). Mix, cool, dilute to 1 liter and mix.

#### 5.1.2 Test Panel

**5.1.2.1** Cut a  $0.50 \pm 0.05$  mm thick piece of 99% pure copper 51 mm x 51 mm for each test.

**5.1.2.2** Form a circular depression in the center of each test panel 3.2 mm deep by forcing a 19 mm steel ball into a 25.4 mm hole to form a cup.

**5.1.2.3** Bend one corner of each test panel up to facilitate subsequent handling with tongs.

#### 5.1.3 Pre-treating Test Panels

**5.1.3.1** Immediately before performing test, pretreat as follows using clean tongs for handling.

- A. Degrease with a suitable neutral organic solvent such as acetone, toluene, or petroleum ether.
- B. Immerse in 5% sulfuric acid (by volume) at  $65 \pm 5^\circ\text{C}$  for 1 minute to remove the tarnish film.
- C. Immerse in a solution of 25% m/v ammonium persulphate (0.5% v/v sulfuric acid) at  $23 \pm 2^\circ\text{C}$  for 1 minute to etch the surface uniformly.
- D. Wash in running tap water for a maximum of 5 seconds.
- E. Immerse in 5% sulfuric acid (by volume) at  $23 \pm 2^\circ\text{C}$  for 1 minute.
- F. Wash for 5 seconds in running tap water, then rinse thoroughly in demineralized water.
- G. Rinse with acetone.
- H. Allow to dry in clean air.

**5.1.3.2** Use the test piece as soon as possible or store up to 1 hour in a closed container.

IPC-TM-650		
Number <b>2.6.15</b>	Subject <b>Corrosion, Flux</b>	Date <b>1/95</b>
Revision <b>B</b>		

#### 5.1.4 Preparation of Test Solder

**5.1.4.1** Weigh 1.00 gram  $\pm 0.05$  gram specimen of solder for each test and place in center of depression of each test panel.

**5.1.4.2** Degrease solder specimen with a suitable neutral organic solvent such as acetone, toluene, or petroleum ether.

**5.1.4.3** Solder may be in the form of pellets or by forming tight spirals of solder wire.

#### 5.2 Test

**5.2.1** Heat solder pot so that solder bath stabilizes at  $235 \pm 5^\circ\text{C}$ .

**5.2.2 Liquid Flux** Place 0.035 g of flux solids into the depression in the test panel. Add solder sample.

##### 5.2.2.1 Solder Paste, Cored-Wire or Cored-Preform

Place 1 g of solder paste, flux-cored wire or cored-preform into the depression in the test panel.

**5.2.3** Using tongs, lower each test panel onto the surface of the molten solder.

**5.2.4** Allow the test panel to remain in contact until solder specimen in the depression of the test panel melts. Maintain this position for  $5 \pm 1$  seconds.

**5.2.5** Carefully examine test specimen at 20X magnification for subsequent comparison after humidity exposure. Record observations, especially any discoloration.

**5.2.6** Preheat test panel to  $40 \pm 1^\circ\text{C}$  for  $30 \pm 2$  minutes.

**5.2.7** Preset humidity chamber to  $40 \pm 1^\circ\text{C}$  and  $93 \pm 2\%$  relative humidity.

**5.2.8** Suspend each test specimen vertically (and separately) in the humidity chamber.

**5.2.9** Expose specimens to the above environment for 240 hours (10 days). M and H may be tested in the cleaned, as well as uncleaned, condition.

**5.3 Evaluation** Carefully examine specimens prior to placing them in the environmental chamber. Note any discoloration.

**5.3.1** After the appropriate exposure period, remove test specimens from humidity chamber, examine at 20X magnification and compare with observations noted in paragraph 5.2.5.

**5.3.2** Corrosion is described as follows:

- A. Excrescences at the interfaces of the flux residue and copper boundary, or the residues or discontinuities in the residues.
- B. Discrete white or colored spots in the flux residues.

**5.3.3** An initial change of color which may develop when the test panel is heated during soldering is disregarded, but subsequent development of green-blue discoloration with observation of pitting of the copper panel is regarded as corrosion.

#### 6.0 Notes

**6.1 Definition of Corrosion** For purposes of this test method, the following definition of corrosion shall prevail. "A chemical reaction between the copper, the solder, and the constituents of the flux residues, which occurs after soldering and during exposure to the above environmental conditions."

**6.2** Color photos before and after the test are valuable tools in identifying corrosion. (See 5.2.5.)

**6.3 Safety** Observe all appropriate precautions on MSDS for chemicals involved in this test method.



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**J-STD-004**

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1. I recommend changes to the following:

- Requirement, paragraph number \_\_\_\_\_
- Test Method number \_\_\_\_\_, paragraph number \_\_\_\_\_

The referenced paragraph number has proven to be:

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- Too Rigid
- In Error
- Other \_\_\_\_\_

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2. Recommendations for correction:

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April 28-30, 1998  
Long Beach, California



March 16-18, 1999  
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For more information, contact Kim Behr

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Our facility manufactures and sells to other companies, printed wiring boards or other electronic interconnection products on the merchant market.

WHAT PRODUCTS DO YOU MAKE FOR SALE?

- |   |  |  |
|---|--|--|
| <input type="checkbox"/> One-sided and two-sided rigid printed boards | <input type="checkbox"/> Flexible printed boards | <input type="checkbox"/> Discrete wiring devices |
| <input type="checkbox"/> Multilayer printed boards                    | <input type="checkbox"/> Flat cable              | <input type="checkbox"/> Other interconnections  |
|   | <input type="checkbox"/> Hybrid circuits         |  |

Name of Chief Executive Officer/President \_\_\_\_\_

INDEPENDENT PRINTED BOARD ASSEMBLERS EMSI COMPANIES

Our facility assembles printed wiring boards on a contract basis and/or offers other electronic interconnection products for sale.

- |  |   |                                      |
|--|---|--------------------------------------|
| <input type="checkbox"/> Turnkey               | <input type="checkbox"/> Through-hole     | <input type="checkbox"/> Consignment |
| <input type="checkbox"/> SMT                   | <input type="checkbox"/> Mixed Technology | <input type="checkbox"/> BGA         |
| <input type="checkbox"/> Chip Scale Technology |   |                                      |

Name of Chief Executive Officer/President \_\_\_\_\_

OEM – MANUFACTURERS OF ANY END PRODUCT USING PCB/PCAs OR CAPTIVE MANUFACTURERS OF PCBs/PCAs

Our facility purchases, uses and/or manufactures printed wiring boards or other electronic interconnection products for our own use in a final product. Also known as original equipment manufacturers (OEM).

IS YOUR INTEREST IN:

- purchasing/manufacture of printed circuit boards
- purchasing/manufacturing printed circuit assemblies

What is your company's main product line? \_\_\_\_\_

INDUSTRY SUPPLIERS

Our facility supplies raw materials, machinery, equipment or services used in the manufacture or assembly of electronic interconnection products.

What products do you supply? \_\_\_\_\_

GOVERNMENT AGENCIES/ ACADEMIC TECHNICAL LIAISONS

We are representatives of a government agency, university, college, technical institute who are directly concerned with design, research, and utilization of electronic interconnection devices. (Must be a non-profit or not-for-profit organization.)

Please be sure both sides of this application are correctly completed



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Authorized Signature \_\_\_\_\_

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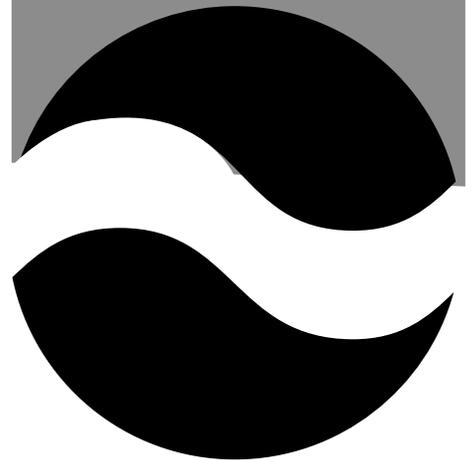
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**J-STD-004**  
JANUARY 1995  
Amendment 1 April 1996

# ***JOINT INDUSTRY STANDARD***

Requirements  
for  
Soldering  
Fluxes

Amendment 1



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The material in this joint standard was developed by the Solder Alloy Task Group (5-24c).

*For Technical Information Contact:*

**Electronic Industries Association**

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2500 Wilson Boulevard  
Arlington, VA 22201  
Phone (703) 907-7500  
Fax (703) 907-7501

**The Institute for Interconnecting  
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Please use the Standard Improvement Form shown at the end of this document.

# Requirements for Soldering Fluxes Amendment 1

**Table 1** Delete and replace with the following:

**Table 1 Flux Identification, Materials of Composition, Activity Levels**

Flux Materials of Composition <sup>2</sup>	Flux Activity Levels (% Halide)/Flux Type <sup>1,3</sup>		Flux Designator
Rosin (RO)	Low (0%)	L0	ROL0
	Low (<0.5%)	L1	ROL1
	Moderate (0%)	M0	ROM0
	Moderate (0.5–2.0%)	M1	ROM1
	High (0%)	H0	ROH0
	High (>2.0%)	H1	ROH1
Resin (RE)	Low (0%)	L0	REL0
	Low (<0.5%)	L1	REL1
	Moderate (0%)	M0	REM0
	Moderate (0.5–2.0%)	M1	REM1
	High (0%)	H0	REH0
	High (>2.0%)	H1	REH1
Organic (OR)	Low (0%)	L0	ORL0
	Low (<0.5%)	L1	ORL1
	Moderate (0%)	M0	ORM0
	Moderate (0.5–2.0%)	M1	ORM1
	High (0%)	H0	ORH0
	High (>2.0%)	H1	ORH1
Inorganic (IN)	Low (0%)	L0	INL0
	Low (<0.5%)	L1	INL1
	Moderate (0%)	M0	INM0
	Moderate (0.5–2.0%)	M1	INM1
	High (0%)	H0	INH0
	High (>2.0%)	H1	INH1

<sup>1</sup> Fluxes are available in S (Solid), P (Paste/Cream) or L (Liquid) forms.

<sup>2</sup> See paragraphs 6.2 and 6.3 for comparisons of RO, RE, OR and IN composition classes and L, M and H activity levels with the traditional classes such as R, RMA, RA, water soluble and low solids “no-clean.”

<sup>3</sup> The 0 and 1 indicate absence and presence of halides, respectively. See paragraph 3.2.3 for an explanation of L, M and H nomenclature.

**Para 6.5** Delete: “c. Flux type or type symbol (see Table 1)”

Replace with: c. Flux designator (see Table 1)

**Appendix A** Delete and replace as follows:

**Appendix A Qualification Test Report**

<b>I.D. Number:</b>				
<b>Flux Classification:</b>			<b>Date of Manufacture:</b>	
<b>Manufacturer's Identification:</b>			<b>Original Use by Date:</b>	
<b>Manufacturer's Batch Number:</b>			<b>Requalified Use by Date:</b>	
<b>Date Original Qualification Tests Completed:</b>			<b>Original:</b>	
<b>Date Requalification Tests Completed:</b>				
<b>Tested By:</b>		<b>Pass: _</b>	<b>Tested By:</b>	<b>Pass: _</b>
<b>Witnessed By:</b>		<b>Fail: _</b>	<b>Witnessed By:</b>	<b>Fail: _</b>
<b>Certification Test</b>	<b>Requirement Paragraph</b>	<b>IPC-TM-650 Method</b>	<b>Test Requirement</b>	<b>Result Pass/Fail/NA</b>
Copper Mirror	3.2.4.1	2.3.32		
<i>Halides Qualitative</i>	3.2.4.2			
Silver Chromate	3.2.4.2.1	2.3.33		
Fluoride Spot	3.2.4.2.2	2.3.35.1		
<i>Halides Quantitative</i>	3.2.4.3			
Chloride, Bromide	3.2.4.3.1	2.3.35, 2.3.28		
Fluoride	3.2.4.3.2	2.3.35.2		
Nonvolatile Determination	3.2.4.3.3	2.3.34		
Total as Chloride	3.2.4.3.1			
Corrosion	3.2.4.4	2.6.15		
SIR	3.2.4.5	2.6.3.3	Cleaned Not Cleaned	
Fungus (optional)	3.2.4.6	2.6.1		
<i>Cleaning Procedure for Flux Characterization</i>				
Cleaning Material_____				
Cleaning Equipment_____				
Cleaning Process Parameters_____				
_____				
_____				



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1. I recommend changes to the following:

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- Unclear
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- Other \_\_\_\_\_

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Message: sign off DesignerCouncil

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Printed Wiring Board Fundamentals	High Speed Design
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Choosing the Right Base Material Laminate	Design for Assembly
Acceptability of Printed Boards	Designers Certification Preparation
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For more information on IPC Video/CD Training, contact Mark Pritchard

tel 505/758-7937 ext. 202 fax 505/758-7938

e-mail: [markp@taos.newmex.com](mailto:markp@taos.newmex.com)

<http://www.ipc.org>

## Training and Certification

### IPC-A-610 Training and Certification Program

"The Acceptability of Electronic Assemblies" (ANSI/IPC-A-610) is the most widely used specification for the PWB assembly industry. An industry consensus Training and Certification program based on the IPC-A-610 is available to your company.

For more information, contact John Riley

tel 847/509-9700 ext. 308 fax 847/509-9798

e-mail: [rilejo@ipc.org](mailto:rilejo@ipc.org) <http://www.ipc.org/html/610.htm>

## IPC Printed Circuits Expo

IPC Printed Circuits Expo is the largest trade exhibition in North America devoted to the PWB industry. Over 90 technical presentations make up this superior technical conference.



April 28-30, 1998  
Long Beach, California



March 16-18, 1999  
Long Beach, California

For more information, contact Kim Behr

tel 847/509-9700 ext. 319 fax 847/509-9798

e-mail: [behrki@ipc.org](mailto:behrki@ipc.org) <http://www.ipc.org>

## How to Get Involved

The first step is to join IPC. An application for membership can be found on page 74.

Once you become a member, the opportunities to enhance your competitiveness are vast. Join a technical committee and

learn from our industry's best while you help develop the standards for our industry. Participate in market research programs which forecast the future of our industry. Participate in Capitol Hill Day and lobby your Congressmen and Senators for better industry support. Pick from a wide variety of educational opportunities: workshops, tutorials, and conferences. More up-to-date details on IPC opportunities can be found on our web page: <http://www.ipc.org>.

For information on how to get involved, contact:

Jeanette Ferdman, Membership Manager

tel 847/509-9700 ext. 309 fax 847/509-9798

e-mail: [JeanetteFerdman@ipc.org](mailto:JeanetteFerdman@ipc.org) <http://www.ipc.org>



# APPLICATION FOR SITE MEMBERSHIP

PLEASE CHECK APPROPRIATE CATEGORY

Thank you for your decision to join IPC members on the "Intelligent Path to Competitiveness"! IPC Membership is **site specific**, which means that IPC member benefits are available to all individuals employed at the site designated on the other side of this application.

To help IPC serve your member site in the most efficient manner possible, please tell us what your facility does by choosing the most appropriate member category.

INDEPENDENT PRINTED BOARD MANUFACTURERS

Our facility manufactures and sells to other companies, printed wiring boards or other electronic interconnection products on the merchant market.

WHAT PRODUCTS DO YOU MAKE FOR SALE?

- One-sided and two-sided rigid printed boards
- Flexible printed boards
- Discrete wiring devices
- Multilayer printed boards
- Flat cable
- Other interconnections
- Hybrid circuits

Name of Chief Executive Officer/President \_\_\_\_\_

INDEPENDENT PRINTED BOARD ASSEMBLERS EMSI COMPANIES

Our facility assembles printed wiring boards on a contract basis and/or offers other electronic interconnection products for sale.

- Turnkey
- Through-hole
- Consignment
- SMT
- Mixed Technology
- BGA
- Chip Scale Technology

Name of Chief Executive Officer/President \_\_\_\_\_

OEM – MANUFACTURERS OF ANY END PRODUCT USING PCB/PCAs OR CAPTIVE MANUFACTURERS OF PCBs/PCAs

Our facility purchases, uses and/or manufactures printed wiring boards or other electronic interconnection products for our own use in a final product. Also known as original equipment manufacturers (OEM).

IS YOUR INTEREST IN:

- purchasing/manufacture of printed circuit boards
- purchasing/manufacturing printed circuit assemblies

What is your company's main product line? \_\_\_\_\_

INDUSTRY SUPPLIERS

Our facility supplies raw materials, machinery, equipment or services used in the manufacture or assembly of electronic interconnection products.

What products do you supply? \_\_\_\_\_

GOVERNMENT AGENCIES/ ACADEMIC TECHNICAL LIAISONS

We are representatives of a government agency, university, college, technical institute who are directly concerned with design, research, and utilization of electronic interconnection devices. (Must be a non-profit or not-for-profit organization.)

Please be sure both sides of this application are correctly completed



# APPLICATION FOR SITE MEMBERSHIP

**Site Information:**

Company Name \_\_\_\_\_

Street Address \_\_\_\_\_

City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_ Country \_\_\_\_\_

Main Phone No. \_\_\_\_\_ Fax \_\_\_\_\_

Primary Contact Name \_\_\_\_\_

Title \_\_\_\_\_ Mail Stop \_\_\_\_\_

Phone \_\_\_\_\_ Fax \_\_\_\_\_ e-mail \_\_\_\_\_

Alternate Contact Name \_\_\_\_\_

Title \_\_\_\_\_ Mail Stop \_\_\_\_\_

Phone \_\_\_\_\_ Fax \_\_\_\_\_ e-mail \_\_\_\_\_

**Please check one:**

- \$1,000.00 Annual dues for Primary Site Membership (Twelve months of IPC membership begins from the time the application and payment are received)
- \$800.00 Annual dues for Additional Facility Membership: Additional membership for a site within an organization where another site is considered to be the primary IPC member.
- \$600.00\*\* Annual dues for an independent PCB/PWA fabricator or independent EMSI provider with annual sales of less than \$1,000,000.00. \*\*Please provide proof of annual sales.
- \$250.00 Annual dues for Government Agency/University/not-for-profit organization

**TMRC Membership**  Please send me information on Membership in the Technology Marketing Research Council (TMRC)

**AMRC Membership**  Please send me information for Membership in the Assembly Marketing Research Council (AMRC)

**Payment Information**

Enclosed is our check for \$ \_\_\_\_\_

Please bill my credit card: (circle one) MC AMEX VISA DINERS

Card No. \_\_\_\_\_ Exp date \_\_\_\_\_

Authorized Signature \_\_\_\_\_

**Mail application with check or money order to:**

IPC  
Dept. 77-3491  
Chicago, IL 60678-3491

**Fax/Mail application with credit card payment to:**

IPC  
2215 Sanders Road  
Northbrook, IL 60062-6135  
Tel: 847 509.9700  
Fax: 847 509.9798

**PLEASE ATTACH BUSINESS CARD OF OFFICIAL REPRESENTATIVE HERE**



*2215 Sanders Road  
Northbrook, Illinois  
60062-6135*

*Tel 847 509.9700  
Fax 847 509.9798  
URL: <http://www.ipc.org>*