ASSOCIATION CONNECTING
ELECTRONICS INDUSTRIES®

2215 Sanders Road
Northbrook, IL 60062-6135

Number	
2.3.28.1	
Subject	
Halide Content of Soldering Fluxes and Pastes	
Date	Revision
06/04	
Originating Task Group	
Flux Specifications Task Group (5-24a)	

IPC-TM-650
TEST METHODS MANUAL

1 Scope This test procedure is designed to measure the level of chloride, bromide, and fluoride present in a soldering flux or paste by Ion Chromatography.

2 Applicable Documents

IPC J-STD-004 Requirements for Soldering Fluxes

IPC-TM-650 Test Methods Manual

2.3.34 Solids Content, Flux

3 Test Specimens

3.1 Liquid flux, solder paste, paste flux, extracted solder preform flux, or cored wire. The reflow/extraction process should be carried out in accordance with J-STD-004 for the solder preforms.

4 Apparatus and Material

4.1 Ion Chromatograph capable of 50 ppb or better detection. The equipment and chemistry should be set up and standardized per the manufacturer's instructions.

4.2 Hot Water Bath capable of maintaining 80 ± 5 °C [176 ± 9 °F].

4.3 Clean, heat sealable bags, i.e., KAPAK® 500 series or equivalent, with less than 250 ppb extractable contaminants. (Specify cleanliness level or manufacturers' part number.)

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

4.5 Deionized water with a resistivity of at least 18.0 megohm centimeter.

4.6 HPLC or ASC grade chemicals for eluent and regenerant preparation.

4.7 NIST traceable standards for chloride, bromide and fluoride.

4.8 2-Propanol (IPA), Electronic grade or better.

4.9 Solder Pot.

4.10 Analytical Balance capable of measuring 0.001g.

4.11 50 mm [1.969 in] mandrel.

4.12 Clean wire cutters.

4.13 Heat sealer.

4.14 Ion-free containers.

4.15 Ultrasonic bath or magnetic stirrer and stir bar.

4.16 Ion-free syringe filters.

4.17 Test Tube.

5 Procedures

5.1 Liquid Flux / Extracted Flux

5.1.1 Prepare a solution of 10% by volume isopropyl alcohol and 90% by volume deionized water and dilute the liquid flux sample to 200X.

5.1.2 Filter samples through ion-free syringe filters to remove particulate prior to analysis.

5.2 Solder Paste/Paste Flux

5.2.1 Weigh to the nearest 0.001 g., approximately one (1) gram of solder paste and place it into a tared 165 mm [6.496 in] by 200 mm [7.874 in] Kapak® bag containing 50 ml of a solution of 10% by volume isopropyl alcohol and 90% by volume deionized water.

5.2.2 Prepare a blank by adding 50 ml of the extract solution to an empty bag.

5.2.3 Label and heat seal the bags and place them into a hot water bath maintained at 80 °C [176 °F].

5.2.4 After 16 hours ± one hour, remove the bags from the water bath and shake vigorously for 10 seconds to mix the contents.

5.2.5 Allow the solutions to cool for 30 minutes minimum before transferring the extract solutions to ion-free plastic containers.

5.2.6 Filter samples through ion-free syringe filters to remove particulate prior to analysis.

5.3 Flux-Cored Solder Wire

5.3.1 Weigh approximately fifty (50) grams of the wire sample.

5.3.2 Wrap the specimen around a 50 mm [1.969 in] diameter mandrel (*Note:* Touch the cut ends of the sample with a soldering iron to seal the sample while cleaning). Place the resultant sample coil into a beaker of boiling deionized water.

5.3.3 After approximately five (5) minutes, remove the coil from the boiling water, rinse with isopropyl alcohol, and allow to air dry.

5.3.4 After drying, cut the sample into approximately one (1) centimeter [0.394 in] length pieces and place inside of a test tube.

5.3.5 Place the test tube, with diced sample, in a solder bath. Thirty (30) seconds after the sample is completely reflowed, remove the test tube and then allow the solder to solidify.

5.3.6 Upon solidification of the solder and before solidification of the flux, pour approximately 0.2 grams of the flux into a test tube of known weight.

5.3.7 Weigh the second test tube containing the flux and subtract off the test tube weight to determine the weight of the flux sample.

5.3.8 Add a known volume of isopropyl alcohol to the sample test tube and place the test tube into a sonicator or on a magnetic stirrer with a stir bar in the test tube for approximately thirty (30) minutes.

5.3.9 Once the flux sample is dissolved, dilute the sample solution such that the overall concentration of isopropyl alcohol is approximately 10%.

5.4 Test Method

5.4.1 Extract solutions shall be analyzed using an Ion Chromatograph with a three to five level calibration.

5.4.2 Dilute extract solutions further with a 10% by volume isopropyl alcohol solution, if necessary, to lower ion concentration levels within the calibration ranges of the chromatograph.

5.5 Calculations

5.5.1 Liquid Flux

5.5.1.1 Determine the weight of each halide ion in the liquid flux:

Weight of halide ion (g) in liquid flux =

[ppm from IC (µg/mL)] x
[Dilution factor, if necessary] x
[Volume of dilution solution (ml)] x 10^{-6}

Note: ''ppm from IC'' value is actually specimen value minus blank value.

5.5.1.2 Determine the weight of flux solids in the liquid flux:

Weight of flux solids (g) in liquid flux =

[Volume of liquid flux sample (ml)] x
[Density of liquid flux sample (g/ml)] x
[% Solids in liquid flux / 100]

5.5.1.3 Calculate percent of each halide ion in the nonvolatile solid portion of the liquid flux:

Percent of halide ion in solid portion of liquid flux =

$$\frac{\text{[Weight of halide ion (g) in liquid flux]} \times 100}{\text{[Weight of flux solids (g) in liquid flux]}}$$

5.5.2 Solder Paste

5.5.2.1 Determine the weight of each halide ion in the solder paste flux:

Weight of halide ion (g) in solder paste flux =

[ppm from IC (µg/mL)] x
[Dilution factor, if necessary] x
[Volume of extract solution (mL)] x 10^{-6}

5.5.2.2 Determine the weight of flux solids in the solder paste flux:

Weight of flux solids (g) in solder paste flux =

[Weight of solder paste sample (g)] x
[% Flux in solder paste / 100] x
[% Solids in paste flux / 100]

5.5.2.3 Calculate the percentage of each halide ion in the nonvolatile solid portion of the liquid flux:

Percent of halide ion in solid portion of solder paste flux =

$$\frac{\text{[Weight of halide ion (g) in solder paste]} \times 100}{\text{[Weight of flux solids (g) in solder paste]}}$$

5.5.3 Flux-Cored Solder Wire

5.5.3.1 Determine the weight of each halide ion in the solder wire flux:

Weight of halide ion (g) in solder wire flux =

[ppm from IC (µg/mL)] x
[Dilution factor, if necessary] x
[Volume of dilution solution (ml)] x 10^{-6}

5.5.3.2 Determine the weight of flux solids in the solder wire flux:

Weight of flux solids (g) in solder wire flux =

[Weight of flux sample (g)] x [% Solids in flux / 100]

5.5.3.3 Calculate percent of each halide ion in the nonvolatile solid portion of the solder wire flux:

Percent of halide ion in solid portion of solder wire flux =

$$\frac{\text{[Weight of halide ion (g) in flux]} \times 100}{\text{[Weight of flux solids (g) in flux]}}$$

6 Notes

6.1 When establishing an ion chromatography method, the containers utilized for extraction and sample processing should be evaluated to confirm that error is not introduced by the chosen ''ion-free'' containers.

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