ASSOCIATION CONNECTING
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Number	
2.3.35.2	
Subject	
Fluoride Concentration, Fluxes-Quantitative	
Date	Revision
06/04	**A**
Originating Task Group	
Flux Specifications Task Group (5-24a)	

IPC-TM-650
TEST METHODS MANUAL

1 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 nonvolatile portion of the flux. Ion specific electrodes are used for the determination. This method uses Orion equipment and solutions.

2 Applicable Documents

IPC-TM-650 Test Methods Manual

2.3.34 Solids Content, Flux

IPC-J-STD-004 Requirements for Soldering Fluxes

3 Test Specimen

3.1 A minimum of 50 ml of first article or production specimen of liquid flux or prepared flux solution.

3.2 For solid or paste flux, 50 ml of the diluted material.

3.3 For solder paste, preform, and cored wire, 50 ml of the extracted flux.

3.4 The solids content of the sample must be known or determined by IPC-TM-650, Test Method 2.3.34.

Note: Recommended procedures for dilution of solid or paste flux, and extraction of solder paste, cored wire, or preforms, can be found in J-STD-004.

4 Apparatus and Reagents

4.1 Apparatus

4.1.1 Autocalibrating ion analyzer.

4.1.2 Fluoride specific electrode.

4.1.3 Double junction reference electrode.

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 pipettes.

4.1.10 Analytical balance capable of 0.001 g.

4.2 Reagents

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

4.2.2 Sodium fluoride, reagent grade.

4.2.3 Chloroform, reagent grade.

4.2.4 Total ionic strength adjustment buffer.

4.2.5 Whatman #41 ashless filter paper, or equivalent.

5 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 a 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 five minutes.

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

5.2 Sample Preparation - Nonwater 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 a 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 five minutes.

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

5.3 Standard Solutions

5.3.1 10 ppm fluoride (0.001%) – This standard can be purchased ready-to-use. Alternately, a solution can be prepared. Weigh 0.0221 gram reagent grade NaF (dried to 140 °C [284 °F]). Add 500 ml DI water. Dilute to one liter in a volumetric flask with total ionic strength adjustment buffer (TISAB).

5.3.2 1.0 ppm fluoride (0.0001%) – This standard can be purchased ready-to-use. Alternately, a solution can be prepared. Weigh 0.0023 gram reagent grade NaF (dried to 140 °C [284 °F]). Add 500 ml DI water. Dilute to one liter in a volumetric flask with TISAB.

5.3.2.1 Another method is to dilute 100 ml of 10 ppm fluoride standard with 450 ml DI water and dilute to one liter in a volumetric flask with TISAB.

5.3.3 0.1 ppm fluoride (0.00001%) – This standard is prepared by diluting 100 ml of 1.0 ppm fluoride standard with 450 ml DI water and diluting to one liter in a volumetric flask with TISAB.

5.3.3.1 Another method is to dilute 10 ml of 10 ppm fluoride standard with 495 ml DI water and dilute to one liter in a volumetric flask with TISAB.

5.4 Double Reference Electrode Filling Solutions

Inner chamber filling solution saturated with AgCl.

Outer chamber filling solution filled with 10% KNO_3.

5.5 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.

5.5.1 Prepare two standards that differ in concentration by a factor of 10. The standards should bracket the expected sample concentration.

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

5.5.3 While stirring with a magnetic stirrer, immerse electrodes in the more dilute standard solution.

5.5.4 Remove and rinse electrodes with DI water. Immerse electrodes in the more concentrated standard solution.

5.5.5 To the sample prepared per instructions for water soluble flux, add 25 ml TISAB. While stirring, immerse electrodes. Record indicated value.

5.6 Evaluation The fluoride value is typically displayed in parts per million (ppm) fluoride. To obtain fluoride as weight percentage of the nonvolatile 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 (nonvolatile chemicals) in the flux sample

6 Notes

6.1 Standardization and Limits Restandardization should be done every few hours or as recommended by the equipment manufacturer to compensate for electrode drift.

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

6.1.2 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.

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

6.1.4 If glassware is used during testing, it is recommended that it should be cleaned thoroughly prior to use to eliminate the possibility of interfering contaminants.

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