ASSOCIATION CONNECTING
ELECTRONICS INDUSTRIES®

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Number	
2.3.35	
Subject	
Halide Content, Quantitative (Chloride & Bromide)	
Date	Revision
06/04	**C**
Originating Task Group	
Flux Specifications Task Group (5-24a)	

IPC-TM-650
TEST METHODS MANUAL

1 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 (nonvolatile) portion of the flux or as milliequivalent per gram of flux solids. A sample of flux or flux extract is titrated to an end-point and the percentage chloride or meq/g of halides is calculated.

2 Applicable Documents

IPC-TM-650 Test Methods Manual

2.3.34 Solids Content, Flux

3 Test Specimen A minimum of 50 ml of liquid flux, diluted paste flux, or diluted solid flux, or 10-50 ml of flux extract from solder paste, solder preforms or flux-cored wire. The solids content of the sample must be known or determined by IPC-TM-650, Test Method 2.3.34.

4 Apparatus and Reagents

4.1 Apparatus

4.1.1 Magnetic stirrer

4.1.2 Analytical balance capable of reading to 0.001 g

4.1.3 Pipettes

4.1.4 Burettes

4.1.5 100 ml beakers, Pyrex

4.1.6 125 ml separatory funnel

4.1.7 125 ml Erlenmeyer flasks

4.1.8 1000 ml volumetric flasks

4.1.9 Water bath

4.2 Reagents

4.2.1 0.1N silver nitrate, standardized: dissolve 17.000 g silver nitrate in deionized water and dilute to 1000 ml in a volumetric flask.

4.2.2 1M sodium hydroxide: 40.0 g of sodium hydroxide diluted to 1000 ml with deionized water in a volumetric flask.

4.2.3 0.2M nitric acid: add 12.6 ml concentrated (16M) nitric acid to deionized water and dilute to 1000 ml in a volumetric flask.

4.2.4 1M Potassium chromate: 194 g diluted to 1000 ml using deionized water in a volumetric flask.

4.2.5 0.03M phenolphthalein solution. (Reagent Grade).

4.2.6 Chloroform (Reagent Grade).

4.2.7 18 megohm or better Deionized water.

5 Procedures

5.1 Sample Preparation

5.1.1 For Rosin/Resin Fluxes

5.1.1.1 Use an analytical balance to accurately weigh about 3-5 g of flux sample in a tared 100 ml beaker.

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 Using a water bath, heat the water extract in the Erlenmeyer flask to expel any chloroform which may be present.

5.1.1.9 Do not heat above 80 °C [176 °F]. Allow solution to cool to room temperature.

5.1.2 For Organic and Inorganic (Water Soluble) Fluxes

5.1.2.1 Use an analytical balance to accurately weigh about 3-5 g of flux sample in a tared 125 ml Erlenmeyer flask.

5.1.2.2 Add 50 ml deionized water.

5.2 Test

5.2.1 Add two drops of 0.03 M phenolphthalein solution to the Erlenmeyer flask (from step 5.1.1.9 or 5.1.2.2).

5.2.2 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.

5.2.3 Dilute to about 60 ml with deionized water.

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

5.3 Calculations

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

$$\text{Halides, as \% chlorides} = \frac{3.55VNx100}{mS}$$

5.3.2 Calculate halides milliequivalent per gram of flux solids (nonvolatiles) using the following formula:

$$\text{Halides, meq/g solids} = \frac{VxMx100}{mS}$$

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 grams.
S is the percentage of solids (nonvolatiles) of the flux.

6 Notes

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