



IPC-TM-650 TEST METHODS MANUAL

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

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

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

2 Applicable Documents

ISO 9455 Soft Soldering Fluxes, Test Methods

IPC-TM-650 Test Methods Manual

2.3.34 Solids Content, Flux

IPC J-STD-004 Requirements for Soldering Fluxes

3 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 Apparatus and Reagents

4.1 General

4.1.1 Use only reagents of recognized analytical quality and only deionized water.

4.1.2 Ordinary laboratory apparatus.

4.2 For Potentiometric Titration Method (Method A)

4.2.1 Tetrabutyl ammonium hydroxide, 0.1 M (0.1 mole/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 a 0.1 M tetrabutyl ammonium hydroxide solution by diluting a 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.

Number 2.3.13	
Subject Determination of Acid Value of Liquid Solder Flux - Potentiometric and Visual Titration Methods	
Date 06/04	Revision A
Originating Task Group Flux Specifications Task Group (5-24a)	

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 phenolphthalein as an indicator.

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

4.2.6 Phenolphthalein Indicator.

4.2.7 Millivoltmeter or pH meter.

4.2.8 Glass electrode.

4.2.9 Saturated calomel, or silver chloride/silver, electrode.

4.2.10 Magnetic or mechanical stirrer with variable speed drive.

4.2.11 Burette capable of delivering aliquots of 0.1 ml to 1.0 ml.

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 neutralized anhydrous ethanol (4.3.1) and neutralized toluene (4.3.2).

4.3.4 2-Propanol. Neutralized with potassium hydroxide, 0.1 M in alcohol (4.3.5), to a faint pink color using phenolphthalein as an indicator.

IPC-TM-650		
Number 2.3.13	Subject Determination of Acid Value of Liquid Solder Flux - Potentiometric and Visual Titration Methods	Date 06/04
Revision A		

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 approximate 0.1 M 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 Procedures

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 the sample is not completely soluble in any of these solvents, select the one in which the sample appears to be the most soluble. If the sample is equally soluble in all four solvents, then use neutralized 2-propanol (4.2.2).

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 to 5 g of the liquid flux sample, taking steps to prevent loss of volatile matter during the weighing. The larger size (≈ 5 g) sample is required for very low solids fluxes. Transfer the weighed sample to a 250 ml low form beaker.

5.1.2.2 Dilute the sample to 100 ml with 2-propanol (4.2.2), 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 0.1 M tetrabutyl ammonium hydroxide solution (4.2.1), adding 1.0 ml portions and recording the pH or mV meter reading 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 reagents only, 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 the sample is not completely soluble in any of these solvents, select the one in which the sample appears to be the most soluble. If the sample is equally soluble in all four solvents, then use neutralized ethanol (4.3.1) 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 flux sample to correspond to approximately 1 g of nonvolatile 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 three drops of phenolphthalein indicator solution (4.3.6) and titrate with the 0.1 M potassium hydroxide solution (4.3.5) until a faint pink color persists throughout the titrated solution for 15 seconds.

5.2.2.4 Carry out a blank determination, using reagents only, for comparison purposes.

5.3 Calculation of Results The acid value is expressed in milligrams of potassium hydroxide per gram of nonvolatile 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 nonvolatile matter) is given by:

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

IPC-TM-650		
Number 2.3.13	Subject Determination of Acid Value of Liquid Solder Flux - Potentiometric and Visual Titration Methods	Date 06/04
Revision A		

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 nonvolatile matter, expressed as a fraction, determined as described in IPC-TM-650, Test Method 2.3.34.

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 Notes

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