



IPC-TM-650 TEST METHODS MANUAL

1.0 Scope To quantify the residual rosin left from solder paste and/or wave soldering flux after the reflow and cleaning process.

2.0 Applicable Documents

IPC-TR-580 Cleaning and Cleanliness Test Program Phase 1 Results

3.0 Test Specimen A printed circuit board assembly which has been cleaned after soldering.

4.0 Apparatus

- a) Ultraviolet spectrophotometer
- b) Balance capable of measuring 0.0001 grams
- c) Balance capable of measuring 500 grams
- d) 100 ml volumetric flasks (5)
- e) 250 ml volumetric flask
- f) 1000 ml volumetric flask
- g) Squeeze bottle
- h) Soxhlet Extraction Tube
- i) 500 ml round bottomed flask
- j) Friedrich condenser
- k) Heating Mantle capable of heating a 500 ml flask
- l) 250 ml Erlenmeyer flask

Materials:

- a) TEST SOLUTION—HPLC TEST SOLUTION with 1.0% v/v phosphoric acid and 0.1% v/v water added.
- b) Solder paste to be used on assembly line
- c) GLAD-LOCK recloseable freezer bags (7 inch x 8 inch x 2.7 mil)

5.0 Procedure

5.1 Preparing the Standards First, analyze the components of the solder paste. To do this assemble a Soxhlet extraction apparatus. Set the round bottomed flask in the heating mantle and plug the mantle into a power regulator so that the temperature can be controlled. DO NOT TURN THE POWER ON AT THIS TIME. Hook the inlet of the condenser to a water supply, and the outlet to a drain.

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Once the extraction unit is assembled, weigh a cotton cellulose thimble on an analytical balance to the fourth decimal place. Record the weight.

Remove the top of the solder paste jar and use a spatula to stir the solder paste. Scoop about 25 grams of fresh solder paste and put it into the thimble. Immediately weigh the thimble and paste together to the fourth decimal place. Record that weight.

Carefully remove the round bottomed flask and the Soxhlet extraction tube from the condenser. Place the thimble containing the paste into the Soxhlet extraction tube and set it aside. Pour about 300 ml of TEST SOLUTION into the round bottomed flask. This amount is not critical. Reassemble the Soxhlet extraction tube and the round bottomed flask back together with the condenser and mantle.

Turn on the water supply to a slow but steady flow. Turn on the power to the mantle and gradually heat the TEST SOLUTION to a boil. As the solvent boils, vapors will condense in the coil and start dripping into the cellulose thimble containing the paste. As the condensate fills the Soxhlet tube, it will eventually fill to a point that a siphon will drain the condensate back down into the round bottom flask. Continued filling and draining of the Soxhlet tube will wash away the flux residues and leave only the solder in the thimble.

Allow the condensate to flush for two hours. At the end of the two hours, disconnect the soxhlet and round bottom flask from the condenser. If TEST SOLUTION is not covering the paste already, add some so that the paste is covered by at least two inches. Using a long object such as a spatula, stir the solder paste to release any remaining trapped flux. Reassemble the tube and flask back together and let the solvent boil for another two hours.

After four hours of boiling, disconnect the Soxhlet and round bottomed flask from the condenser and turn off the water. Pick the thimble up and let the TEST SOLUTION drain back into the Soxhlet tube. Set the thimble on a paper towel to air dry for about one hour then place it in an oven at 150°F for two hours. Remove the thimble from the oven and let it cool to room temperature. Weigh the thimble and dry solder together to four decimal places and record the weight.

Pour the TEST SOLUTION that is in the Soxhlet tube into the round bottomed flask with the rest of the solvent. Place the

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round bottomed flask in the mantle and bring the solvent back to a boil. Let the TEST SOLUTION evaporate until the volume is around 50 to 100 ml then remove it from the mantle. Weigh a 250 ml erlenmeyer flask to at least 2 decimal places and record the weight. Pour the solvent from the round bottomed flask two times to make sure all of the residue is in the erlenmeyer.

Set the erlenmeyer on a hot plate and evaporate the solvent dry. The flask will probably not go completely dry, but it will reduce to a thick, viscous looking solvent. Remove the flask from the heat and let it cool. When the flask is at room temperature, weigh the flask and residue together and record the weight.

Repeat this testing at least four times to get an average of each weight.

****CAUTION: SOLDER IS A HAZARDOUS MATERIAL. DISPOSE OF SOLDER AND SOLDER PASTE PROPERLY.**

5.1.1 Standards Calculations:

(Thimble + wet paste wt.) – thimble wt. = wet paste wt.

(Thimble + dry solder wt.) – thimble wt. = dry solder wt.

(Flask + residue wt.) – flask wt. = residue wt.

$$\frac{100}{\text{wet paste wt.}} \times \text{dry solder wt.} = \% \text{ metal}$$

$$\frac{100}{\text{wet paste wt.}} \times \text{residue wt.} = \% \text{ residue}$$

$$\frac{\text{all \% metals}}{\text{number of times run}} \times \text{average \% residue}$$

$$\frac{\text{all \% residues}}{\text{number of times run}} \times \text{average \% residue}$$

***Note:** All % metals run and the amount listed in technical data sheets should be within 2% of each other. All % rosins run and the amount listed in technical data sheets should be within 1% of each other.

5.2 Standards: Making the Stock Solution Once the solder paste components have been verified, standards can be made.

Weigh a cotton cellulose thimble to the fourth decimal place and record the weight. Add about 25 grams of fresh solder paste to the thimble and weigh it immediately. Record the weight.

Put the thimble into the Soxhlet tube and assemble the extraction unit as described earlier. Turn on the water to the condenser and the power to the mantle. Gradually bring the solvent to a boil.

As done previously, let the paste flux for two hours then stir it with a spatula. Flux for another two hours. When flushing is complete, lift the thimble up and let the TEST SOLUTION drip back into the Soxhlet tube. Set the thimble on a paper towel and let it air dry for one hour. Place the thimble in an oven at 150°F for two hours. Remove the thimble from the oven and let it cool to room temperature. Weigh the thimble and paste together to the fourth decimal place and record the weight.

Pour the solvent remaining in the Soxhelt tube into the round bottom flask and let it boil down to about 100 ml. Remove the flask from the heat. Weigh a 500 ml volumetric flask to the second decimal place and record the weight. Pour the TEST SOLUTION from the round bottomed flask into the volumetric. Rinse the round bottomed flask with TEST SOLUTION at least twice to be sure all of the residue is in the volumetric. Rinse the inside of the volumetric down and continue to add TEST SOLUTION until the flask is about half full. Weigh the flask and the solvent together to the second decimal place and record the weight. Cap the flask and mix thoroughly.

5.2.1 Calculations

(thimble + paste wt.) – thimble wt. = paste wt.

Example: 32.8293 – 4.2686 = 28.5607

(thimble + dry solder wt.) – thimble wt. = metal wt.

Example: 29.6060 – 4.2686 = 25.3374

*this weight should be within 1% of your average metal wt.

paste wt. – metal wt. = flux wt.

Example: 28.5607 – 25.3374 = 3.2233

(volumetric flask + solvent wt.) – volumetric wt. = solvent wt.

Example: 175.22 = 199.96

flux wt. x average % residue = wt of residue in flask

Example: 3.2233 X 0.55 = 1.77

$$\frac{\text{residue wt.}}{\text{solvent wt.}} = \% \text{ residue in stock solution}$$

Example:

$$\frac{1.77}{199.96} = 0.00885 \text{ grams or } 0.885\%$$

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5.3 Making the Standards from the Stock Solution

First, calculate the amount of stock solution you will need for each standard. Standards that will be used are 0.002%, 0.004%, 0.006%, 0.008% and 0.010%.

5.3.1 Calculations

To make 50 grams of 0.002% standard:

$$\frac{0.002}{\% \text{ residue in volumetric}} \times 50 = \text{wt. of stock solution to be used for standard} *$$

***Note:** If the amount of stock solution to be taken from the 500 ml volumetric is less than one gram, dilute the concentration of the solvent. This will decrease your margin of error.

Example:

$$\frac{0.002\%}{0.885\%} \times 50 = 0.1129 \text{ grams}$$

So:

Dilute 10 grams of 0.885% = 190 grams TEST SOLUTION

$$\frac{10 \times 0.885\%}{200} = 0.04425\%$$

Now:

$$\frac{0.002\%}{0.04425\%} \times 50 = 2.26 \text{ grams}$$

Calculate the rest of the standard using the same calculation, substituting the standard number for 0.002.

5.4 Making the Standards

Get five, 100 ml volumetric flasks and label them:

- 1) 0.002% residue
- 2) 0.004% residue
- 3) 0.006% residue
- 4) 0.008% residue
- 5) 0.010% residue

Weigh or zero the first volumetric labeled 0.002%. Add the amount of stock solution to the flask (*Example:* 2.26). Add TEST SOLUTION until the weight reaches 50 grams not including the weight of the flask. Cap the flask immediately and mix thoroughly.

Mix the rest of the standards using the different amounts of stock solutions calculated.

5.5 Procedure: Measuring the Standards

- 1) Turn on the spectrophotometer and let the system warm up.
- 2) Fill the sample cuvette (fill both cuvettes if your system is a double beam) with clean TEST SOLUTION. Place the cuvette(s) into the holder(s).
- (3) Go to 241 nm wavelength and zero or record the absorbance reading as the 0.00% standard. This is also known as the "Y ntercept."
- (4) Remove the sample cuvette and dump the TEST SOLUTION. Fill the cuvette with the 0.002% standard and insert it back into the spectrophotometer. Record the absorbance reading.
- (5) Repeat step four until all of the standards have been measured. Rinse the cuvette at least two times with TEST SOLUTION between each standard. Be sure that the outside of the cuvette is clean and dry before each measurement.
- (6) When all of the standards have been measured, graph the absorbance readings versus the percent concentration. Draw a line that best fits the points plotted. Ideally, the line should run through all of the points plotted (see Figure 1). If it does not, a problem has occurred in either the standards or a data entry. Check the printout to see that all data was entered properly. Look at the graph to see if one of the data points is located off line with the others as in Figure 2. If all of the points seem to be scattered, there is probably something wrong with the stock solution and the whole process will need to be redone.

- (7) Calculate the slope of the line using the following calculation:

$$\text{slope} = \frac{\text{change in concentration}}{\text{change in absorbance}}$$

5.6 Procedure: Sample Analysis

- 1) Take a GLAD-LOCK bag and add exactly 100 ml of TEST SOLUTION. To reduce the chance of leaking, keep the sealing part of the bag as dry as possible when adding the solvent.
- 2) Handle the board being tested with gloved hands and insert it into the bag along with the TEST SOLUTION.
- 3) Remove as much of the air from the bag as possible then seal the top of the bag closed. Fold the top of the bag over twice to reduce the size of the bag and to add to the seal.

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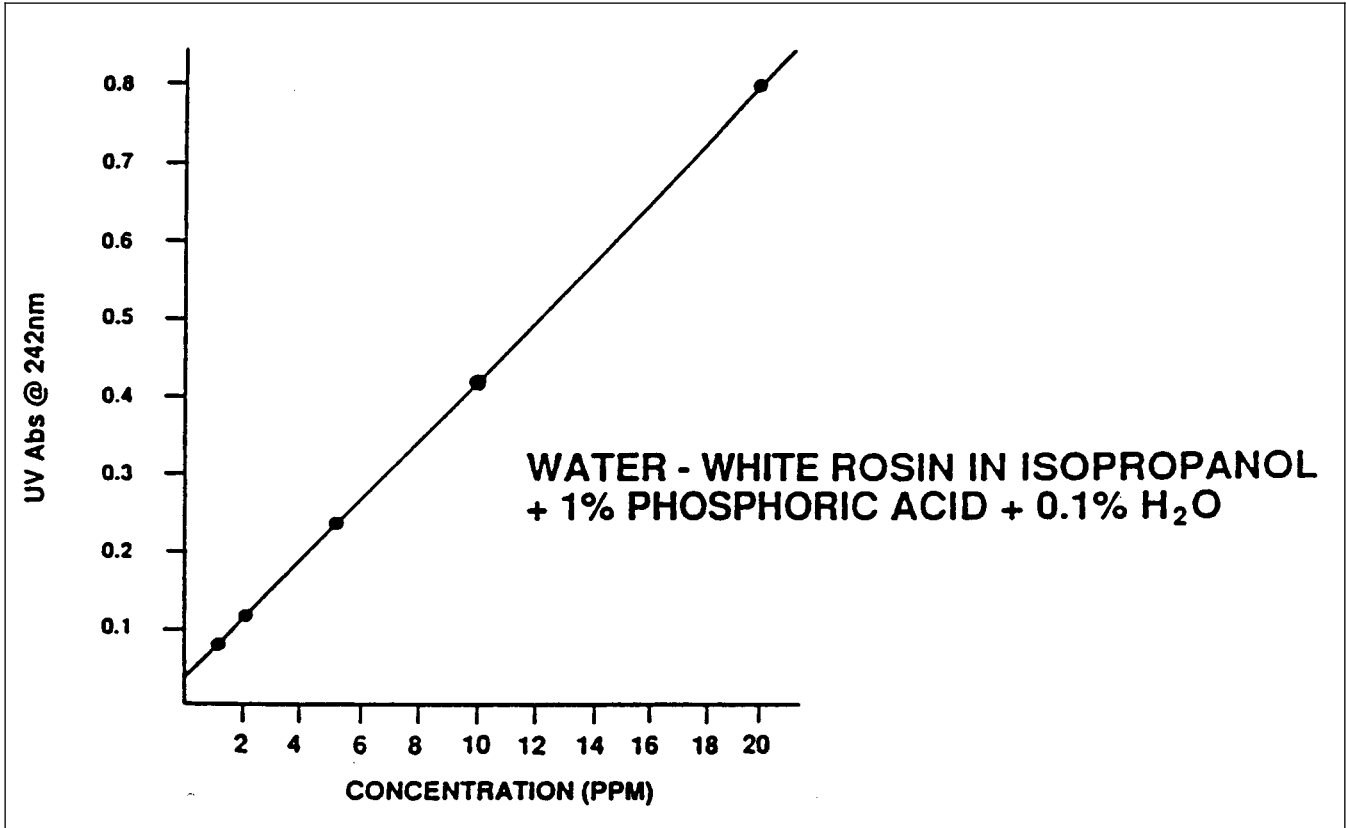


Figure 1

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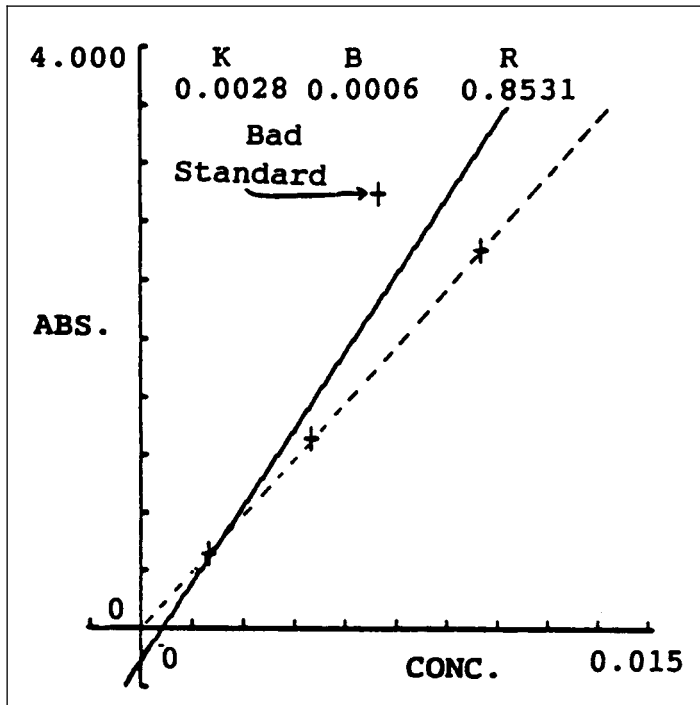


Figure 2

- 4) Hold the folded top of the bag with one hand, and hold the board in place with the other. Damage to the bag is likely to occur if the board is allowed to shift around while shaking. Shake the bag containing the board for 10 minutes.
- 5) At the end of 10 minutes, REMOVE THE BOARD FROM THE BAG.
- 6) Again, rinse the sample cuvette at least twice with clean TEST SOLUTION. Fill the cuvette with a sample of TEST SOLUTION from the bag. Insert the cuvette into the sample chamber of the spectrophotometer and record the absorbance reading.

Note: In the case of the boards that have high levels of contamination, the absorbance reading will be off scale. In that case, add another 100 ml of TEST SOLUTION to the bag and shake it for a couple of minutes. Repeat step six. If it is still off scale, add another 100 ml until the absorbance readings are less than 3.000. Keep track of the amount of TEST SOLUTION that is added.

- 7) Convert the absorbance readings to concentration percent using the following calculation:

$$\text{concentration \%} = \text{slope} \times \text{absorbance} + \text{Y intercept}$$

- 8) Convert the concentration percent to parts per million (ppm) using the following calculation:

$$\frac{\text{\%} \times 1,000,000}{100} = \text{ppm}$$

- 9) To calculate the residual rosin in micrograms per square inch using the following calculation:

$$\frac{\text{residual rosin micrograms/sq. in} = \text{ppm rosin} \times \text{specific gravity of TEST SOL.} \times \text{ml TEST SOL. in bag}}{\text{Surface area of board}}$$