



IPC-TM-650 TEST METHODS MANUAL

Number 2.3.27.1	
Subject Rosin Flux Residue Analysis—HPLC Method	
Date 1/95	Revision
Originating Task Group New Methods Task Group (5-32f)	

1.0 Scope

1.1 This High Performance Liquid Chromatography (HPLC) procedure outlines the analysis of rosin flux residues remaining on a printed wiring board (PWB) after defluxing. This test can be used for the evaluation of processes used to clean rosin based soldering fluxes.

Applicable Documents

IPC-TP-383 "Organic Surface Contamination - Its Identification Characterization, Record Effects on Surface Insulation Resistance and Conformal Coating Adhesion."

1PC-TR-580 "Cleaning and Cleanliness Test Program Phase 1 Results."

3.0 Test Specimens

3.1 Printed wiring board (PWB) for extraction

4.0 Apparatus and Materials

4.1 HPLC systems with UV detection

4.2 Waters C18 Novapak column, or equivalent

4.3 Suitable extraction vessel, KAPAK® bag, or equivalent, to extract PWB

4.4 Volumetric Flasks

4.5 Acetonitrile, HPLC grade

4.6 Deionized water, HPLC grade

4.7 Rosin Standards: abietic acid, dehydroabietic acid, neoabietic acid (Helix BioTech, 604-270-7468, Aldrich Chemical, Alltech Associates)

4.8 2-Propanol (IPA), HPLC grade

4.9 Sodium phosphate monobasic, NaH₂PO₄oH₂O

4.10 Hot water bath, 80° ± 5°C

5.0 Procedure

5.1 Extraction

5.1.1 Record area of PWB. General rule on surface area is (length x width x 2)+10% for a populated PWB.

5.1.2 Place processed PWB in extraction bag, or equivalent

5.1.3 Prepare 75/25 (by volume), IPA/H₂O solutions for the extraction.

5.1.4 Add 75-200 mls of IPA/H₂O solution to extraction bag, enough to cover PWB.

5.1.5 Heat seal bag and place in water bath at 80° ± 5°C for 1 hour (cut vent hole in bag).

5.1.6 Dilute (with IPA/H₂O solution) or concentrate extract to get approximately 100 ml of extract per 35 sq inch of PWB area.

5.1.7 Extract unprocessed PWB blank, in same manner as sample.

5.2 Standard and sample analysis

5.2.1 Set HPLC instrument conditions as follows:

Wavelength 220 & 240 nanometers
 (The two wavelengths are needed to get optimum response from all constituents. See attached chromatograms.)
 Column temp 60°C
 Mobile phase Acetonitrile/water 60/40
 25 millimolar Na₂PO₄H₂O
 Flow rate 2 milliliters/minute
 Sample size 10 microliters
 (Instrument conditions may be changed to optimize separation)

5.2.2 Prepare standards of known concentration

5.2.3 Establish retention times and areas of rosin standards

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5.2.4 Prepare calibration curves for each of the identifiable peaks in the extract chromatogram.

5.2.5 Run extracts obtained in 5.1

5.3 Calculation of residue concentration

5.3.1 Concentration of material in solution

$$(\text{milligrams/liter}) = (A \times B \times C) / (D \times E)$$

A = Area of material peak

B = Concentration of standard (milligrams/liter)

C = Injection volume of standard (microliters)

D = Area of standard peak

E = Injection volume of sample (microliters)

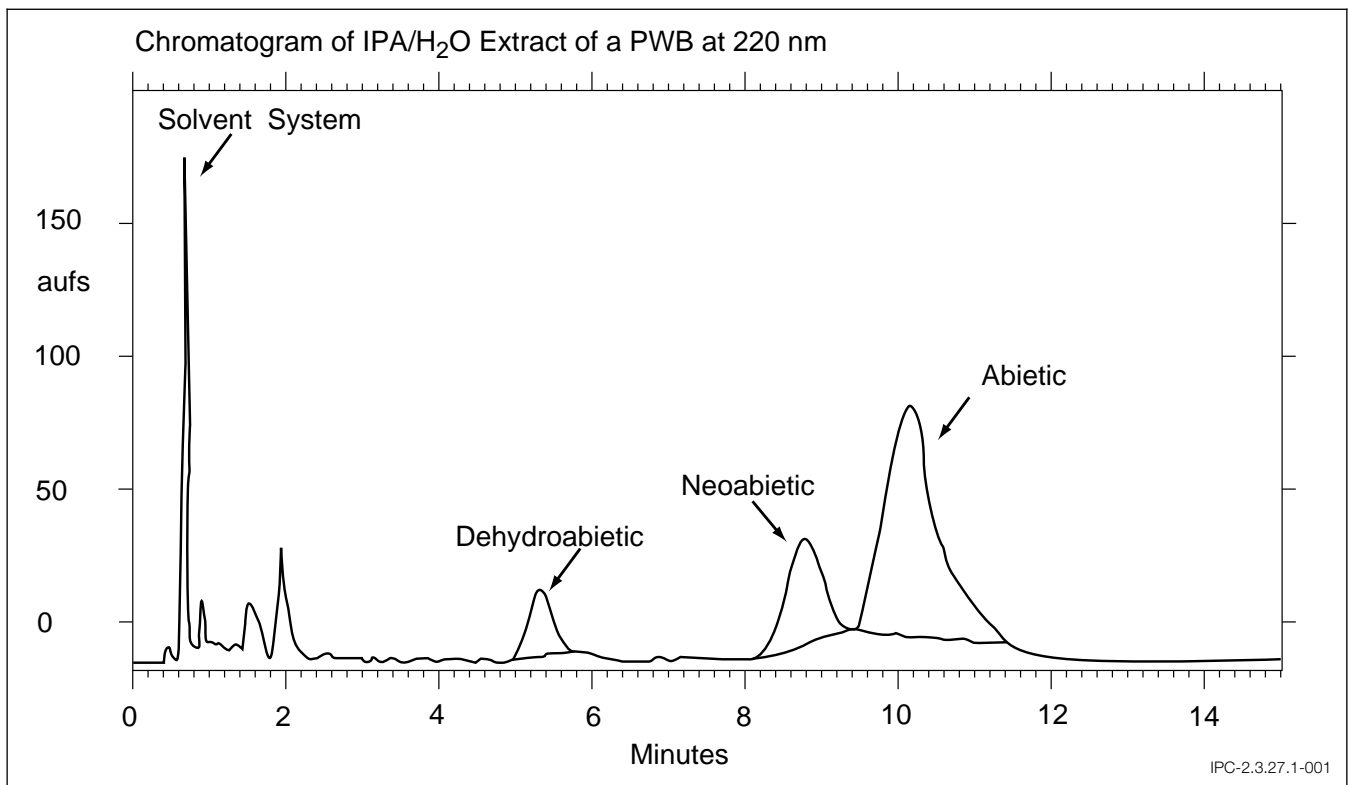
Residual material {(micrograms/inch squared)(ug/in²)} =

$$((F \times G) / H) \times 1000 \text{ug/mg}$$

F = Concentration of material in solution (milligrams/liter)

G = Volume of extract solvent (liter)

H = PWB surface area (square inch)



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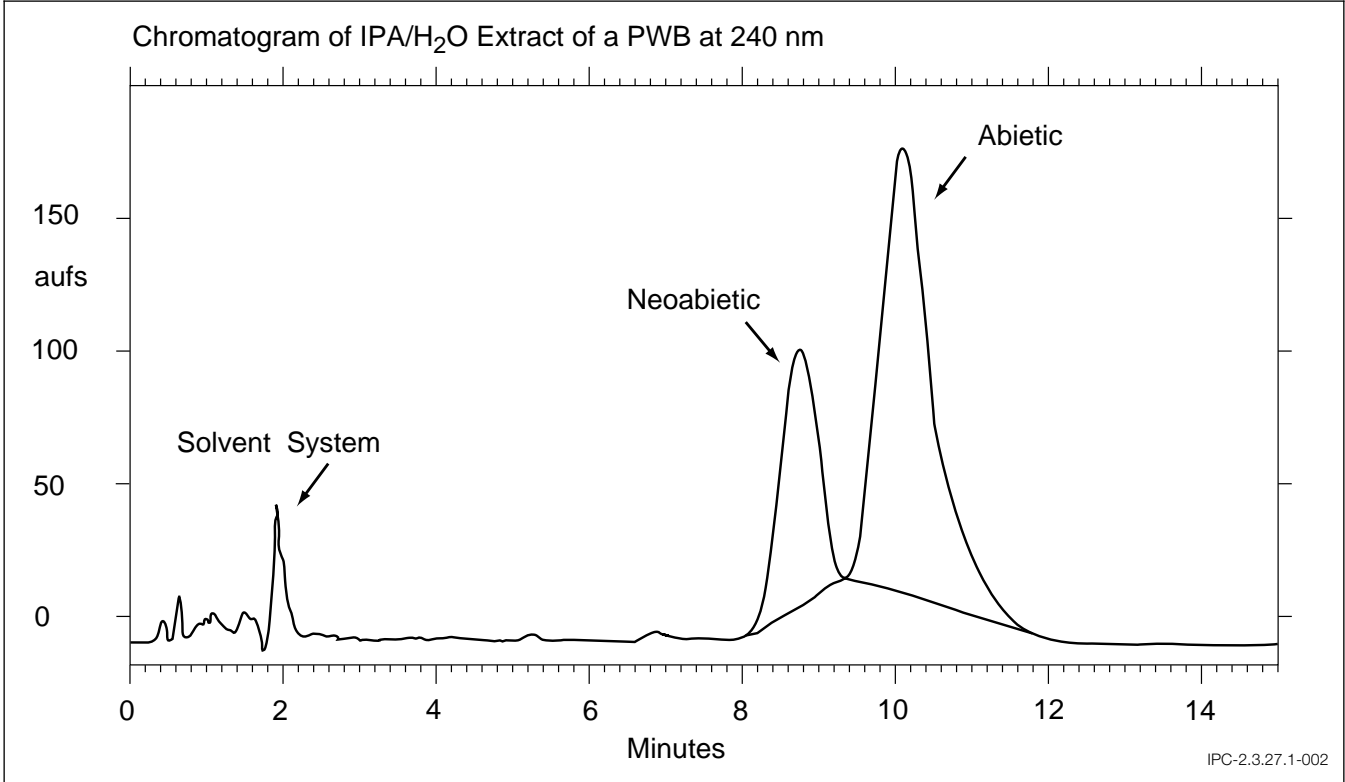


Figure 2