

TECHNICAL PAPER ≡

IPC Cleaning and Cleanliness Test Program Phase 3 Water Soluble Fluxes Part 1

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IPC Cleaning and Cleanliness Test Program Phase 3 Water Soluble Fluxes Part 1: B-24 Interactions of Water Soluble Fluxes with Printed Wiring Board Substrates/Metallizations September 30, 1992

A joint program sponsored by the EPA/DOD/IPC/Industry Ad Hoc Solvents Working Group. This evaluation was performed at the Naval Air Warfare Center, Aircraft Division, Indianapolis, Indiana.

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IPC Cleaning and Cleanliness Test Program

Phase 3 Water Soluble Fluxes Part 1

1 Overview

Chlorofluorocarbon (CFC) compounds have been identified as agents contributing to the reduction of stratospheric ozone. It is incumbent upon the electronics industry, as a primary user of CFC solvents, to find alternative processes and materials which eliminate the need for CFCs. One such alternative is the substitution of water-soluble fluxes and aqueous cleaning for rosin fluxes and CFC cleaning.

The tests and results presented in this paper represent a portion of the third phase of a test program being coordinated by the Institute for Interconnecting and Packaging Electronic Circuits (IPC) and the ad hoc Solvents Working Group, a committee of Department of Defense representatives, military defense contractors, commercial manufacturers, material suppliers, equipment manufacturers and representatives of the Environmental Protection Agency (EPA). The goal of the overall program, divided into three phases, is to determine alternatives to chlorofluorocarbon (CFC) cleaning of printed wiring assemblies. Phase 1 of the program established a set of benchmark values for the cleaning efficiency of nitromethane-stabilized CFC-113/methanol azeotrope in printed wiring assembly applications which use rosin-based solder fluxes. Phase 2, still in progress, tests alternative cleaning materials and processes for removing rosin fluxes and compares their performance to the Phase 1 benchmark values. Phase 3 examines soldering materials and cleaning processes which eliminate the need for CFC cleaning. The Phase 3 Program, chaired by Dr. Laura Turbini of the Georgia Institute of Technology, has identified three areas of research: water soluble fluxes, controlled atmosphere soldering, and low-solids/no-clean fluxes.

The first Phase 3 test, an evaluation of water soluble flux (WSF) technology, was performed at the Naval Air Warfare Center, Aircraft Division, Indianapolis (formerly the Naval Avionics Center) in 1991. This study was divided into two parts. Part 1 examined the interaction between water soluble fluxes and the substrate/metallization of the test assembly. The IPC-B-24 standard test assembly was used for this test. Part 2, not covered in this paper, repeated the rosin-based benchmark experiment from Phase 1 of the test program, for a direct comparison between rosin fluxes and water soluble fluxes. Reference (1), Appendix G, gives the details of the test plan.

2 Equipment

A listing of the materials and equipment, and associated machine parameters, used at NAWC-Indianapolis is listed in Tables 1 through 9.

2.1 Wave Solder Equipment and Parameters

TABLE 1

Model	Electrovert 2000S
Conveyor Speed	3.0 Feet/Min
Air Knife Pressure	8 si
PWB Temperature	200°F
Wave Fluxer Setting	48
Thermal Factor	1.0
Pro Relay Setting	160
Upper Preheat	350°F
Omega Wave	Off
Preheat 1	6
Preheat 2	6
Board Spacing	6 inches
Thermal Profiling	ECD Super-MOLE
Orientation	Fingers leading with titanium solder boots covering old fingers.
Delay Time	15 ± 5 minutes from exit of soldering to start of cleaning.

2.2 Aqueous Cleaning Equipment and Parameters

TABLE 2

Model	Electrovert 2000C See Figure 2
Media	Deionized Water Only
Water Purity	1 Megohm-cm (System setting)
Conveyor Speed	3.5 Feet Min
Water Flow Rate	4 Gallons Min
First and Second Stage Washes	Temperature = 150°F Top Pressure = 40 psi Bottom Pressure = 24 si
Third Stage Rinse	Temperature = 150°F Top Pressure = 33 psi Bottom Pressure = 21 psi
Final Stage Rinse	Temperature = 150°F Top Pressure = 22 psi Bottom Pressure = 12 psi
Air Knife Temperature	450°F

2.3 Solder Paste Application Equipment and Parameters

TABLE 3

Model	Forslund 1224 Screen Stencil Printer
Squeegee Hardness	75 Shore A
Print Mode	Single non-flood
Target Paste Weight	0.53 ± 0.03 grams
Stencil Thickness	0.008 inches (8 mils)

2.4 Infrared Reflow Equipment and Parameters

TABLE 4

Model	Electrovert Infraflow 5000 Turb Air (on loan from Electrovert)
Panel and Edge Heater Temperatures	Zone 1: 115°C Zone 2: 125°C Zone 3: 190°C Zone 4: 175°C Zone 5: 260°C Rear and Front Edge: 155°C
Conveyor Setting	30 (equals 45 cm/sec)
Board Spacing	12 inches between boards
Cooling Time	15 minutes after exit from reflow
Thermal Profile	ECD Super-MOLE

2.5 Ionic Cleanliness Test Equipment and Parameters

TABLE 5

Ionic Cleanliness Tester	Omegameter 600SMD
Calibration	Electrical and Chemical Daily
Solution Volume	4000 ml
Solution Temperature	41 - 43°C
Test Time	15 Minutes
PWB Board Area	35 square inches
Pass/Fail Limit	None

2.6 Ion Chromatography Equipment and Parameters

TABLE 6

Equipment	Dionex 4000i System, AS4A Anion Columns AMMS Chemical Suppression Unit, Conductivity Detector
Eluent	0.0018N Sodium Bicarbonate
Temperature	25°C
Chemical Suppression	0.005N sulfuric acid
Injection Loop	100 microliters
Flow Rate	2 ml/minute

2.7 High Pressure Liquid Chromatography (HPLC) Equipment and Parameters

TABLE 7

Equipment	Waters HPLC System, Hewlett Packard Photodiode Array
Columns	Brownlee Labs Polpore H
Wavelength	210 nm
Bandwidth	30 nm
Reference Wavelength	350 nm
Column Temperature	35°C
Mobile Phase	0.01 N sulfuric acid
Flow Rate	0.4 ml/minute
Sample Size	10 microliters

2.8 Surface Insulation Resistance (SIR) Equipment and Parameters

TABLE 8

Equipment	Despatch 16512 microprocessor controlled humidity chamber, NAWC custom fixtures, shielded PTFE ribbon cables
Data Acquisition	Keithley 706 Scanner with 7158 Cards Keithley 617 Electrometer Keithley 230 Voltage Source Custom software in QuickBasic
Power Supply	NJE Corp model 125-75
Instrument Control	IBM XT with Iomega GPIB Controller Card
Test Length	28 days
Ambient Conditions	25°C 50% RH
Elevated Conditions	85°C 85% RH
Ramp Method	IPC Phase 1 profile 30 minute simultaneous ramp (condensation possible)
Bias Voltage	- 50 Volts DC (applied prior to ramp-up)
Measurement Voltage	+ 100 Volts DC
Measurements Taken At	0, 24, 96, 168 hours, 14, 21, 28, 29 days
Visual Examination	7-10x with backlighting (40W source)

2.9 Other Equipment and Materials

As with all IPC test programs, vendor support was critical. A list of donated materials and services is listed as Table 9.

TABLE 9

Phototooling and Stencils	Magnavox
Components (LCCCs)	Intel
Pastes	Alpha Metals, Kester Solder, Multicore
Fluxes	Alpha Metals, Kester Solder, London Chemical (Lonco)
Glass Boards	Texas Instruments
Thermal Profiling System	ECD Super-MOLE
Edge Card Connectors	AMP
Titanium Solder Boots	Electrovert
Levchecks	Hexacon
IPC-B-24 Test Boards	Sandia National Laboratories, Georgia Institute of Technology, Hughes Aircraft
HCFC Cleaning Solvent	Allied Signal
Copper Mirror Coupons	Georgia Institute of Technology
Vinyl Gloves	Delco Electronics
Ion Chromatography Analyses	Delco Electronics
HPLC Analyses and Surface Analytical Work	Alliant Techsystems, NAWC, Indpls
Additional SIR Testing	Hughes Aircraft - El Segundo AT&T - Oklahoma City

3 Developmental Work

3.1 Test Plan Development

The test plan for the water soluble flux evaluation had taken form over the course of several IPC semi-annual meetings. In July, 1990, a group of people from the Ad Hoc Solvents Working Group met at Georgia Tech to finalize the details of the test plan. Most of the specifics were decided, but some items were equipment specific and were left for the test site to determine during process development work. Figure 1 shows the final process flow diagram for the flux-metal-substrate evaluation (Part 1).

The thermal profiles used in the test plan were agreed to by all of the vendors participating in the study. Single profiles were used, rather than profiles for each paste or flux, in order to limit variability between materials. Appendix B shows the thermal profiles used in this evaluation.

3.2 Sample Sizes

Table 10 shows the number of test boards processed for each cleanliness characterization test. A single set of boards served for both the ion chromatography and HPLC tests.

TABLE 10 Sample Sizes

Material	600SMD	IC HPLC	SIR	Photo	Extra	Total
Control	3	5	3	1	2	14
Flux #1	3	5	3	1	2	14
Flux #2	3	5	3	1	2	14
Flux #3	3	5	3	1	2	14
Paste #1	3	5	3	1	2	14
Paste #2	3	5	3	1	2	14
Paste #3	3	5	3	1	2	14

3.3 Test Vehicle Development

Previous phases of the test program used the IPC-B-36 standard test assembly. One criticism of the B-36 was that flux may not directly contact many of the test patterns and therefore, the true measure of the flux interaction with the test vehicle was not made. Therefore, the test plan committee developed a new test vehicle, the IPC-B-24. This was a single sided test board with four comb patterns having nominal 0.4 mm (0.016") lines and 0.5 mm (0.020") spaces. This configuration was selected to minimize cost and maximize the amount of data for each test coupon. These comb patterns had a nominal square count of 1000 squares. Actual square count varied due to variations in line spacing. Table 11 gives a comparison of the B-24 and B-36 test assemblies. The 0.4 mm lines and 0.5 mm spacings were chosen to minimize bridging during wave soldering.

TABLE 11 Comparison of B-24 and B-36 Test Vehicles

Parameter	IPC-B-24 (Part 1)	IPC-B-36 (Part 2)
Dimensions	4.0" x 4.375"	4.0" x 4.375"
Copper Circuit	Single Sided	Double Sided
Laminate Material	0.062" FR-4 Epoxy Glass	0.062" FR-4 Epoxy Glass
Test Patterns	4 Comb Patterns, 1000 squares each	10 patterns, 200-3000 squares
Line Widths	0.4 mm 16 mil	0.15 mm (6 mil)
Line Spacings	0.5 mm 20 mil	0.15 mm (6 mil)
Components	None	Two 68 I/O LCCCs
Solder Applied	Directly to patterns	To non-pattern side with exception of the mounting pads

3.4 Test-Site Process Development

Process development work was done at the Naval Avionics Center (now the Naval Air Warfare Center, Aircraft Division, Indianapolis) during the fall of 1990. Process development covered such elements as:

- obtain the donated materials and equipment from the vendors
- develop very detailed instruction sets for the operators
- fully characterize the materials
- schedule the testing with the manufacturing department
- train operators
- fabricate/obtain the necessary B-24 assemblies
- inspect the assemblies
- prepare the assemblies for testing
- arrange for Test Monitoring and Validation Team (TMVT) visitation
- coordinate the efforts of several test services
- calibrate equipment
- determine the optimum settings for each piece of equipment
- arrange photographic work and video recording
- fully document every facet of the test

3.5 Materials Characterization

Each of the three fluxes and three pastes was subjected to characterization tests per IPC-SF-818 and IPC-SP-819, respectively. The results of these tests are listed in Tables 12 and 13.

TABLE 12 Liquid Fluxes

Material	Percent Solids	Specific Gravity	Water Extract Resistivity	Chloride % Extract/Direct	Lot Number	Thinner Lot Number
Alpha 857	17.90	0.866	7,105 ohm-cm	7.38%/7.88%	0090135A	0080092A Alpha 425
Kester 2331-ZX	31.70	0.900	6,065 ohm-cm	6.84%/6.80%	57986	Unknown Kester 301
Lonco 3355-HB	27.45	0.966	3,082 ohm-cm	7.06%/7.05%	901544	901895 3355 Additive

TABLE 13 Solder Pastes

Material	%Tin %Lead	%Antimon	%Metals	Powder Type	Particle Shape	Ave. Diameter
Alpha WS601	63.0/36.8	0.19	90.59	3	Spherical	43 microns
Kester R-577T	62.9/37.0	0.01	90.00	3	Spherical	41 microns
Multicore PHX21A88	63.2/36.5	0.27	87.07	3	Spherical	48 microns

Material	Lot Number	Viscosity	Slump	Fineness of Pitch	Deposit Thickness
Alpha WS601	10104902	340,000 cps	None	Bridging	10 mil
Kester R-577T	DS	Not Available	None	Bridging	10 mil
Multicore PHX21A88	011528	480,000 cps	None	Bridging	7 mil

3.6 Process Documentation

It was a requirement of the test program that every process be fully documented. This included development of written process instruction sets for every operation. Copies of all process instruction sets are included as Appendix A. In order to document the conditions that each test board was subjected to, a process traveler sheet accompanied every test board. This traveler was used to record process times, pastes weights, etc., along with any anomalies or variations from the process instructions. Samples of the process traveler sheets are included as Appendix C. Extensive photography and videotape documentation was also arranged.

3.7 Test Board Inspection

The B-24 boards were purchased from Magnavox with funds provided by Sandia National Laboratories, and received in November and December, 1990. The boards were manufactured from a single lot of tetra-functional, 0.062" FR-4, epoxy glass laminate, 0.5 oz copper, with no preservative finish on the copper circuitry. Each test board was visually inspected for defects. A representative measurement of the line width and line spacing of the comb patterns was taken for each test board. The line width and spacing were assumed to be constant for each board. Each board was tested electrically for the presence of dead shorts or low resistance paths. Two representative boards were subjected to full MIL-P-55110D, Group A, testing. Samples from these boards were also subjected to thermal analysis. The results are shown in Tables 14A and 14B. Boards which failed the electrical test, or contained imperfections (scratches), were marked as practice boards. During the inspection process, the boards were handled by the edges only, and with gloved hands. All surfaces which came into contact with the test boards were thoroughly cleaned with isopropyl alcohol.

TABLE 14A MIL-P-55110 Inspection Results

NAWC Lab Number	Z901750
Inspector	Pe PI mate
Inspection Date	12/17/90
Copper Thickness	0.87 mils (0.00087")
Nickel Strike Thickness	0.14 mils (0.00014")
Gold Thickness	0.05 mils (0.00005")

TABLE 14B Results of Thermal Analysis

Operator	Gerald Rhoades
Thermal Analysis	Differential Scanning Calorimet DSC
Sample Size	21.8 mg
Percent Cure	98.95 %
Midpoint Tg	142°C

3.8 Test Board Preparation

All test boards were prepared for testing using the following procedures.

3.8.1 Oven Bake

All boards were baked in a forced air oven at 150°C for one hour. This bake step was done to fully polymerize the epoxy matrix of the boards and to liberate as much free Bromide from the boards as possible.

3.8.2 Microetch Process

The oven bake procedure caused a heavy layer of oxide to develop on the copper circuitry of the test boards. A microetch procedure was needed to remove the oxide layer. Boards were processed in accordance with Table 15.

TABLE 15 Microetch Procedure

Step	Solution	Temperature	Dwell Time
1	Enplate PC-499*	160°F	5 Minutes
2	Tap Water Spray Rinse	Room Temperature	30 Seconds
3	1st Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
4	2nd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
5	3rd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
6	Deionized Water Overflow Rinse	Room Temperature	1 Minute
7	Enplate AD-485	Room Temperature	15 Seconds
8	Tap Water Spray Rinse	Room Temperature	30 Seconds
9	1st Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
10	2nd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
11	3rd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
12	Deionized Water Overflow Rinse	Room Temperature	1 Minute
13	10% v/v Sulfuric Acid	Room Temperature	2 Minutes
14	Tap Water Spray Rinse	Room Temperature	30 Seconds
15	1st Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
16	2nd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
17	3rd Stage Tap Water Cascade Rinse	Room Temperature	30 Seconds
18	Deionized Water Overflow Rinse	Room Temperature	1 Minute

* Enplate PC-499 (an alkaline cleaner) and Enplate AD-485 (a mild copper microetch) were prepared according to the manufacturer's (i.e., Enthone's) directions. The deionized water used for rinsing was a minimum one megohm-cm resistivity.

3.8.3 IPA/Water Clean

Immediately following the microetch procedure, all boards were subjected to cleaning in an Omegameter 600SMD until the ionic levels had not changed for five minutes. This differs from the test plan method of cleaning to lower than two micrograms per square inch. Boards were cleaned in batches of six boards. Boards were separated to allow full solution access. Solution resistivity values were not recorded during this operation. This step was included to ensure that all boards were free of ionic residues prior to any test processing. All boards were cleaned until no further ionic residues could be removed.

3.8.4 Board Drying

After removal from the Omegameter, the boards were placed in clean aluminum racks and batch-dried in a nitrogen autoclave at room temperature. After nitrogen-drying, the boards were heat sealed in Kapak® bags (6.5" x 8") until ready for testing.

3.9 Formulations Tested

This test program was intended to evaluate water soluble flux technology, rather than any individual flux or paste formulation. Three vendors' products were chosen for the solder pastes and three vendors' formulations for the liquid fluxes. The chosen vendors were long-standing active members of the Ad Hoc Solvents Working Group and had contributed to the development of the test plan.

3.10 Other Developmental Work

In October, 1990, the Materials Laboratory's four-stage IR reflow unit was moved to another facility. Electrovert donated a five-stage Turb-Air 500C IR convection reflow oven for this test.

During the process development phase, the thermal profiles of all machines were determined. A single IR profile to reflow all three pastes was identified. Similarly, a single thermal profile was used for preheat and reflow in the wave solder process.

An evaluation of the aqueous cleaner parameters was done using a procedure developed by Hollis, and modified by Texas Instruments (Reference 2). Using a glass board and this method, the set of parameters for the aqueous cleaner was optimized for the removal of water soluble fluxes using DI water. Water quality tests were performed to gauge the water entering the aqueous cleaner. Prior to this test, NAWC used city tap water in combination with detergents for removal of rosin-based flux. For the Phase 3 tests, four in-line industrial water deionizing columns were installed. The water purity at the exit of the columns was set at a minimum of one megohm-cm. The aqueous cleaner was de-calcified prior to the test and the cleaner was completely flushed between cleaning cycles for each of the fluxes and pastes. The aqueous cleaner used only deionized water. No wetting agents or detergents were added.

Samples of the fluxes and pastes were forwarded to Terry Munson, Delco, and Heather Getty, Alliant Techsystems, for development of Ion Chromatography (IC) and High Pressure Liquid Chromatography (HPLC) analytical procedures. Appendices D and E contain the test procedures used for these residue characterization tests.

Process development also included development of the detailed instruction sets and forms for documenting the test processes.

3.11 Dry Run

On January 11, 1991, NAWC personnel performed a dry run of the experiment to verify the timing of all processes and to iron out any problems in the operation of any equipment. The "Paste No-Clean" and "Flux No-Clean" processes (See Figure 1) were evaluated during the dry run. The No-Clean processes were included to determine the worst case values for each paste and flux. Terry Munson, a member of the TMVT, was present for the dry run experiments. The ionic cleanliness tests and SIR tests were performed on the uncleaned test boards. HPLC testing was not performed.

Difficulties were experienced when trying to measure the total ionic contamination levels on the wave fluxed (Flux No-Clean) test boards. Ionic levels exceeded the maximum levels which the Omegameter 600SMD could record (>100 ug/in²). The results of the ionic contamination tests on the uncleaned boards are given in Table 18 (see section 5.1).

A set of the uncleaned test boards were tested for Surface Insulation Resistance (SIR) for seven days (85°C/85% RH). The initial measurements, taken at ambient conditions, indicated that all patterns registered as electrical shorts. The NAWC SIR data acquisition system made use of current limiting resistors (10^5 ohms); therefore, a short would have a value of 10^5 ohms. These low values of resistance were not unexpected considering the highly ionic nature of the uncleaned flux residues. SIR measurements were taken at elevated conditions (85°C/85% RH) after seven days. The electrical shorts remained.

After removal from the humidity chamber, the boards were visually examined. All boards showed extensive signs of electrochemical corrosion.

After the conclusion of the dry run, all process instructions were reviewed and corrected as needed. The process instructions of Appendix A reflect those changes made after the dry run.

3.12 NAWC Personnel

A test program of this magnitude could not have been done without the active participation of many dedicated personnel at NAWC, Indianapolis. Many people contributed their days off and countless hours of careful and detailed work to ensure the success of this test program. The following people are gratefully acknowledged for their many contributions to the program.

Douglas Pauls and Mike Hook (co-managers), Robin Sellers, Tom Tierney, Dusty Herrings, Barbara Malburg, Linda Redenbarger, Teresa Birdsong, Jack Williams, Jim Cole, Earl Johnson, Ed Arvin, Gary Smith, Ed Ostack, Frank Renfro, Dave Loby, Melissa Pirtle, David Pond, Barbara Belt, Sharon Wagner, Gary Moore, Gerald Rhoades, Linda Lubber, John O'Connor, Peggy Plymate, Malcom Griner, Tim Bowren, Tim Berg, Steve Weddle, Laura Stocker, Joe Kurek, Jim Hovelsrud, Gordon Morris, and Mike Bratcher.

Thanks are also due to the management at NAWC AD Indianapolis for their support of the Phase 3 test program.

4 Test Procedure

4.1 Test Monitoring and Validation Team (TMVT)

A TMVT was established from the Ad Hoc Solvents Working Group and assembled at NAWC on February 1, 1991. Following an initial briefing, members divided into teams to observe and document the process, and to note any anomalies or deviations which occurred. A listing of TMVT members and their affiliations is shown in Table 17.

TABLE 17 TMVT Members

Name	Affiliation	Name	Affiliation
Dr. Laura Turbini	Georgia Tech	Mr. Robert Burress	Texas Instruments
Mr. David Bergman	IPC	Mr. Tom Carroll	Hughes Aircraft
Mr. Mike Oborn	Sandia National Labs	Ms. Quynhgio Le	Boeing
Mr. Carl Buchanan	Army	Mr. Terry Munson ¹	Delco
Mr. Hyun Soo Park	NASA	Ms. Heather Getty	Alliant Techsystems
Mr. Steve Sauer ³	Navy	Mr. Michael Brownfield	Magnavox
Mr. Jim Moffitt ³	Navy	Mr. Les Hymes ²	G.E. Medical
Mr. Bill Vuono	Navy (EMPF)	Mr. Bill Down	Electrovert
Mr. David Ross	Air Force	Mr. Al Schneider	Alpha Metals
		Mr. David Dodgen	Interflux USA
		Mr. Raoul Smoczinski	London Chemical

1 - Mr. Munson now with CSL Labs, Kokomo, IN.

2 - Mr. Hymes now with Plexus, Inc., Neenah, WI.

3 - Mr. Sauer and Mr. Moffitt observed the testing but were not formal TMVT members.

4.2 Board Handling

All processing and handling of boards was done with gloved hands and scrupulously cleaned instruments. Contact with the board surface was minimized wherever possible. The gloves had been previously shown to contribute no ionic contamination by testing a glove in the Omegameter 600SMD. All surfaces which could contact the test boards were cleaned with isopropyl alcohol (IPA) and lint-free paper towels. All racks were either wiped down with IPA or degreased with a hydro-generated chlorofluorocarbon (HCFC) between process sequences. These measures were taken to minimize any outside sources of contamination in the test.

4.3 Wave Solder Operations

All soldering and cleaning operations were thermal profiled prior to testing. Five thermocouples were attached to a B-24 test board using a thermally conductive epoxy adhesive. One thermocouple was placed in the center of each comb pattern with an additional thermocouple placed in the center of the test board. The thermocouples were then attached to an ECD SuperMOLE thermal profiling device. The SuperMOLE was protected during the thermal operations by an insulating jacket and by a rubber enclosure for the aqueous cleaning operations.

The Flux No-Clean processes (see Figure 1) had been evaluated during the dry run of the experiment. The Flux Clean processes, which were used to evaluate the three liquid water soluble fluxes, were performed on February 1, 1991. The thermal profile for the wave solder is shown as Appendix B, Profile 1. Prior to testing, the wave fluxer assembly and reservoir were cleaned thoroughly with isopropyl alcohol. The isopropyl alcohol cleaning procedure was repeated each time a new water soluble flux was introduced.

Process Instruction 1, Appendix A, was followed for all wave solder operations. Several practice boards were run across the wave fluxer, removed from the machine prior to preheat, and weighed to give a representative weight of flux applied to each board. Differences were noted in average applied flux weights due to differences in the specific gravity and solids content of the fluxes.

The B-24 test boards were wave soldered with the comb side down (contacting the flux and solder), and the contact fingers leading. Clean titanium solder boots were used to cover the gold contact fingers of the test boards. After the boards exited the wave solder, the solder boots were removed and the boards were placed in clean, horizontal aluminum racks to cool for 15 minutes. The time from soldering to cleaning was held constant at 15 ± 5 minutes. The wave solder unit and aqueous cleaner were located in the same area, so board transport from the solder station to the aqueous cleaning station was not required.

4.4 Aqueous Cleaning

Process Instruction 2, Appendix A, was followed for all aqueous cleaning operations. All aqueous cleaning was accomplished using only deionized water. No saponifiers or surfactants were used in the aqueous cleaner. The thermal profile for the aqueous cleaner is shown as Appendix B, Profile 2. After the 15 minute post-solder delay was completed, the boards were placed one at a time onto the aqueous cleaner conveyor. A diagram of the aqueous cleaner is shown as Figure 2. The final stage of the aqueous cleaner was a heated air knife to dry the assemblies.

Water samples were taken from the aqueous cleaner before and after the running of each flux material and tested for Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), and general water quality. These tests were performed to estimate potential environmental hazards.

After exiting the aqueous cleaner, the boards were placed into clean aluminum racks and transported to the board sort operation.

4.5 Transport

Process Instruction 3, Appendix A, was followed for all board transporting. Transporting of test boards between processing stations was necessary due to equipment locations. The clean aluminum racks were covered with degreased aluminum foil and placed into an insulated plastic cooler. The cooler top was put in place and heat sealed. The coolers were then carried to the necessary processing stations. This arrangement was done to ensure that no contaminants contacted the test boards during transport, and that the boards would see no thermal variations due to outside weather conditions.

4.6 Board Sorting

Process Instruction 4, Appendix A, was followed for board sort operations. Table 10 shows the sample size for each residue characterization test. Test boards were removed from the aluminum racks and electrically tested using a digital multi-meter. If a test board had a short, it was unacceptable for SIR testing, and was slated for one of the other tests. Electrical checking was terminated after three "good" assemblies for SIR testing had been found. If three non-shortened test boards could not be found, the boards with the least amount of bridging were re-worked using a dry solder iron and dry solder braid to remove any existing bridges. The bridge removal technique was needed for only three of the wave soldered boards. Two boards had one bridge each, and the remaining board required the removal of two bridges.

Boards slated for Omegameter 600SMD testing were returned to the aluminum racks, recovered with foil, placed into the insulated cooler, and transported to the Omegameter test station.

Boards slated for Ion Chromatography or HPLC analysis were individually placed into Kapak® bags and heat sealed. Previous testing by Terry Munson had shown that these bags do not contribute ionic or organic contaminants. A single set of boards were extracted to remove residues for IC and HPLC analysis. Boards slated for SIR testing were heat sealed in Kapak® bags until the SIR test was begun on 4 February 1991.

4.7 Solder Paste Printing

Process Instruction 5, Appendix A, was followed for all solder paste application operations. The Paste No-Clean processes (see Figure 1) had been evaluated during the dry run of the experiment. The Paste Clean processes, which we used to evaluate the three water soluble flux solder pastes, were performed on February 2, 1991. Solder paste was applied to the fingers of each comb pattern using an eight-mil (0.008") stainless steel stencil and a semi-automatic stencil print operation. The solder pastes had been taken from refrigerated storage the night before testing and were at room temperature at the time of printing. Each jar of paste was thoroughly mixed with a spatula prior to any printing operation.

Paste was printed onto practice boards until the applied paste weight was consistent within the 0.53 ± 0.03 gram target range, and the registration and print quality were acceptable. Test boards were weighed prior to and after printing to determine the applied paste weight. The weight of paste applied was recorded for each test board. After printing, the boards were placed horizontally into clean aluminum racks. After all test boards for a particular paste had been printed, the boards went to the infrared reflow station. A baking step, normally needed for rosin solder pastes, was not used at the advice of the solder paste vendors.

Every surface at the solder print station was thoroughly cleaned with IPA between printings with different solder pastes, to eliminate cross-contamination between solder pastes. The stencil and squeegees were also thoroughly cleaned with IPA.

4.8 Infrared (IR) Reflow

Process Instruction 6, Appendix A, was followed for all IR reflow operations. Thermal profiles were taken periodically to ensure consistency. The thermal profile for the IR oven is shown as Appendix B, Profile 3.

After the solder print operations had been completed for each paste, the test boards were removed from the aluminum racks and placed on the IR oven conveyor belt, spaced 12 inches apart. When the boards exited the oven, they were placed into clean aluminum racks. The full racks were then covered with degreased aluminum foil, placed into insulated coolers, and transported to the aqueous cleaning station. The fifteen minute delay between reflow and cleaning was strictly adhered to for all test boards.

4.9 Ionic Cleanliness Tests

Process Instruction 7, Appendix A, was followed for all Omegameter 600SMD testing. Total ionic contamination testing was performed in the same manner as in the Phase 1 Benchmark test.

An Omegameter 600SMD was used to evaluate ionic residues remaining on the test assemblies after processing. The Omegameter system is an automated system capable of meeting the cleanliness test requirements of MIL-C28809, MIL-STD-2000, and MIL-STD-2000A. The 600SMD used a heated isopropanol/water (75%/25%) solution and a pressure spray system to remove ionic contaminants. This removal method is more effective than some of the unheated, static systems which could also have been used.

Each of the test assemblies was tested individually. The Omegameter was programmed with the parameters listed in Table 5. The Omegameter was calibrated daily with hydrometer and thermometer. HPLC-grade isopropanol was added to the solution when necessary to bring the solution to a 75%, by volume, isopropanol concentration. The remaining 25% was distilled water.

The Omegameter heater had been turned on the night before the testing. Three days prior to testing, the Omegameter was electrically calibrated. At the start of each day of testing, the Omegameter was chemically calibrated using the manufacturer's recommended calibration procedure of placing five milliliters of standard NaCl solution (750 ppm NaCl in water) in 5000 milliliters of test solution. At all periods when ionic testing was not being done, the Omegameter was kept in the CLEAN/FILL cycle to keep the solution and internal plumbing of the Omegameter at thermal equilibrium.

Recent experimentation with the Omegameter has shed new light on the operating characteristics of the machine. As the IPA/water solution is continuously cycled through the deionizing columns, the resistivity of the solution continually increases as the solution becomes cleaner. The electronics of the machine has a maximum resistivity limit of 60.35 megohm-cm and equates a resistivity of 44.5 megohm-cm to 0.0 micrograms/in². If the Omegameter had been cycling the fluid for several minutes (or hours), the true resistivity could have been much higher, requiring a small amount of ionic contamination to bring the machine back to the zero point. Therefore, the testing procedure of Phases I and II was modified to begin each test cycle from a standard starting point. To accomplish this, if the Omegameter had been running on the CLEAN/FILL cycle for three minutes or longer, one milliliter of standard NaCl solution was pipetted into the test solution and a single CLEAN/FILL cycle was initiated.

Each test assembly was carefully placed in the Omegameter test cell using clean tongs, and the test initiated. The tongs did not contact the test solution. The Omegameter continuously monitored the solution's resistivity and charted the total amount of ionic contamination. At the end of the 15 minute test, the Omegameter reported the level of ionic contamination in total micrograms of equivalent sodium chloride per square inch.

4.10 Ion Chromatography (IC) and HPLC Tests

The boards slated for IC and High Pressure Liquid Chromatography (HPLC) testing were taken by Terry Munson and extracted on the day following the testing. The extraction procedure (Appendix D) used for the IC and HPLC tests was developed by Terry Munson. Funding for Mr. Munson's work was provided by Delco Electronics. The extract solution was divided into two aliquots. The first aliquot was used for IC analysis. The second aliquot was shipped to Mr. Tom Barrett, Alliant Techsystems, Hopkins, MN, for HPLC analysis.

Ion Chromatography was used to separate, identify, and quantify ions in solution. The combination of this technology with an effective extraction procedure provided the analytical means to better understand flux residues left on printed circuit boards after IR reflow, wave soldering, and cleaning processes. The Delco DIONEX 4000i system was sensitive in the low ppb and ppm ranges for most anions.

The ion chromatography analyses of the WSF extracts quantified contaminants in micrograms per square inch of: (1) chlorides, (2) bromides, and (3) weak organic acids (WOAs).

The HPLC analytical work was done by Heather Getty and Tom Barrett of Alliant Techsystems, Hopkins, Minnesota. The testing was jointly funded by Alliant Techsystems and Honeywell. The following description of the HPLC analytical technique was furnished by Ms. Getty.

High pressure liquid chromatography (HPLC) is an analytical technique which is used to separate chemicals dissolved in a solvent. The separation is performed by passing the solvent through a column. Each chemical in the solvent will be slowed down by the column dependent on its properties. A solvent containing a mixture of chemical A, B and C will be separated by the column into discrete groups of A, B and C. A detector identifies the time each material emerges from the column, and based on peak area, the quantity present. Standard chemicals A, B and C are necessary to determine the time it takes a chemical to pass through the column and to create a calibration curve which relates the peak area to a given quantity of material.

For the Phase 3 Water Soluble Flux Evaluation, HPLC was used to identify the amount of flux or paste residue remaining on a circuit board after cleaning. The circuit board extract, furnished by Mr. Munson, was analyzed by HPLC. The HPLC analytical procedure is shown in Appendix E. Since only the fluxes and pastes were available, rather than each individual constituent, the standards were the fluxes and pastes. The result is that the material detected can only be quantified as "X" of Flux 1; rather than X% of Chemical A, Y% of Chemical B and Z% of Chemical C. The latter format is preferred, but not possible. HPLC was performed on 35 samples (5 control and 5 each flux 1,2, and 3 and paste 1, 2, and 3).

4.11 SIR Tests

SIR testing was used as an indicator of the electrical characteristics of a substrate after it had been exposed to various manufacturing processes. There are many forms of SIR testing commonly used in the electronics industry. They all consist of exposure to an environment that is hotter and more humid than ambient conditions. In selecting the temperature-humidity profile for the test, the committee wanted to maintain the same test levels (85°C/85% RH) used in Phases I and II of the test program, and to adhere to the Class 3 test specifications of IPC-SF-818. The SIR test run in Phase 1 was a seven day (168 hour) test. The SIR test called out in IPC-SF-818 is a 28 day test. The 28 day test was chosen to extend the evaluation period for the WSF/substrate interactions.

A diagram of the Phase 1 SIR temperature-humidity profile is shown as Figure 3. As in Phases 1 and 2, the profile called for temperature and humidity to be simultaneously ramped from ambient conditions (25°C/50% RH) up to elevated conditions (85°C/85% RH) over a 30 minute period.

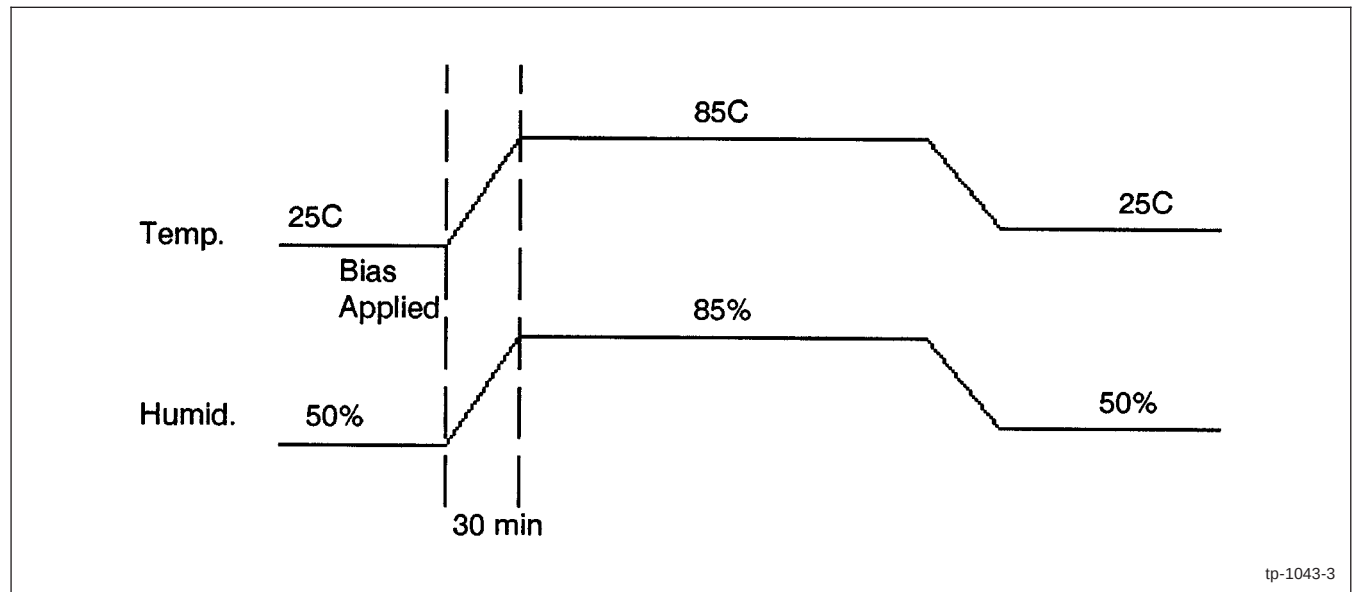


Figure 3 Phase 1 Temperature-Humidity Profile

An electrical bias of -50 volts DC was applied to the test patterns prior to the start of the temperature-humidity ramp up. This bias remained on all test patterns throughout the test, except when resistance measurements were made. The elevated conditions were maintained for 28 days. After 28 days, the chamber was ramped down to ambient conditions over a 30 minute period, using a simultaneous ramping of temperature and humidity. The -50VDC bias voltage was applied during the ramp-down period.

Resistance measurements were made using + 100 volts DC electrification voltage applied to each test pattern for 60 seconds. Electrical resistance measurements were taken at 0, 24, 96, and 168 hours, and then weekly thereafter until the 28th day. The readings at 0 hours and the final two readings (day 29) were at ambient conditions. All other readings were at elevated temperatures. One hour after the chamber had stabilized at ambient conditions after the ramp-down, a measurement set was taken. A final set of readings was taken 24 hours later.

The IPC-B-24 test board has four individual, identical comb patterns. Each comb has a square count of approximately 1000 squares. Actual square count is dependent on the line spacings of each comb pattern, which varied slightly from the nominal 0.4 mm. Three test boards (a total of 12 comb patterns) from each flux/paste process were SIR tested. A total of 21 boards were tested.

Reference 3 is a NAWC AD Indianapolis report which gives greater detail on the SIR test set-up equipment, and measurement procedures.

5 Test Results

This test program was intended to characterize water soluble flux technology, rather than any specific formulations. Therefore, all test data shown in this report is given in a coded format. The codes for the fluxes and pastes were randomly assigned.

5.1 Results from Dry Run (Uncleaned Boards)

TABLE 18 Uncleaned Boards

Material	Weight Applied	Ionic Levels	Ambient SIR Levels	Elevated SIR Levels
Controls	0.00 grams	0.76 $\mu\text{g}/\text{in}^2$	10^{13} ohms	10^9 ohms
Flux #1	0.080 grams	$>100 \mu\text{g}/\text{in}^2$	$<10^5$ ohms	<10 ohms
Flux #2	0.203 grams	$>100 \mu\text{g}/\text{in}^2$	$<10^5$ ohms	<10 ohms
Flux #3	0.200 grams	$>100 \mu\text{g}/\text{in}^2$	$<10^5$ ohms	<10 ohms
Paste #1	0.570 grams	34.9 $\mu\text{g}/\text{in}^2$	$<10^5$ ohms	<10 ohms
Paste #2	0.519 grams	18.1 $\mu\text{g}/\text{in}^2$	$<10^5$ ohms	<10 ohms
Paste #3	0.530 grams	46.7 $\mu\text{g}/\text{in}^2$	<10 ohms	<10 ohms

Values of 10^5 ohms are system minimums and should be viewed as a “dead short.” Some of the test boards did attain slightly higher values (10^7 - 10^8 ohms) at the completion of the test; however, all boards exhibited heavy corrosion.

5.2 Ionic Cleanliness by Omegameter 600SMD (Cleaned)

Table 19 gives the results of the total ionic contamination test. All values are expressed in micrograms per square inch of sodium chloride equivalent. Figure 4 illustrates the results graphically.

TABLE 19 Ionics Values for Cleaned Boards

	Controls	Flux #1 (F1)	Flux #2 (F2)	Flux #3 (F3)	Paste #1 (P1)	Paste #2 (P2)	Paste #3 (P3)
Sample 1	1.6	16.0	5.0	8.3	0.8	1.60	2.00
Sample 2	0.7	15.5	4.10	8.0	1.0	2.4	1.4
Sample 3	0.5	13.6	5.2	11.4	2.1	2.1	1.4
Mean	0.93	15.10	4.83	9.23	1.30	2.03	1.60
Std. Dev.	0.59	1.35	0.64	1.88	0.70	0.40	0.35

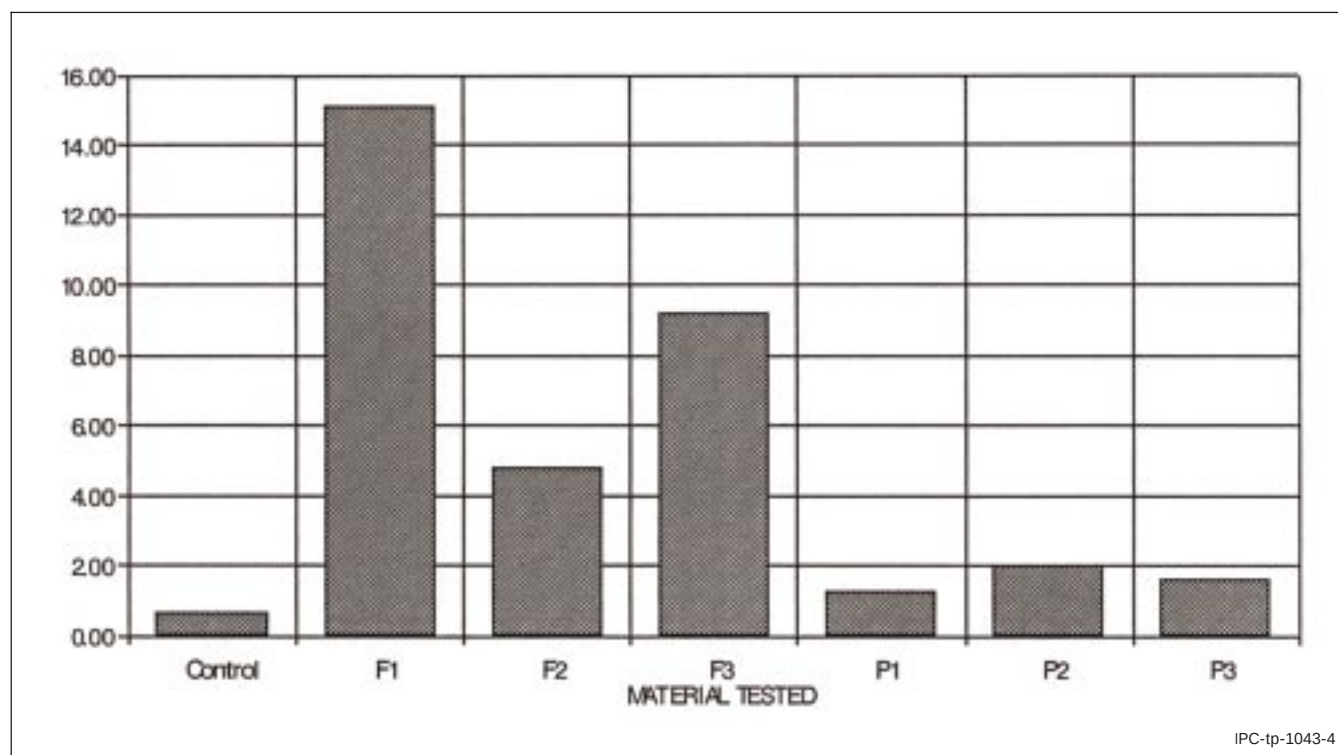


Figure 4 Ionic Contamination Levels by Omegameter 600SMD

The control boards were used as blanks to baseline the results. The values for the WSF pastes were generally lower due to lower levels of flux contained in the paste. Flux was applied only where solder paste was stenciled; directly on the copper fingers of the test pattern. The boards processed with the liquid WSFs were wave fluxed, and therefore the entire board surface was flooded with flux.

The values for Flux #1 are above the military limit of 14.0 micrograms per square inch. It should be noted however, that this limit was derived from a test with room temperature static solutions of IPA and DI water. The Omegameter 600SMD used a heated, agitated solution of IPA and DI water, and is therefore a more effective extractor of ionic material. No equivalency factor has been determined between the unheated, unagitated solution, and the heated agitated solution. It is unclear whether the boards processed with Flux #1 would have failed in the non-heated solution.

It should also be noted that these test boards contained no components and therefore presented no cleaning challenges. The high level of ionic contaminants illustrates how ionically active and aggressive WSFs can be.

5.3 Ion Chromatography Results

The values for the Ion Chromatography (IC) are shown in Table 20 and graphically in Figure 5. All values listed are in micrograms of contaminant species per square inch.

Table 20 Ion Chromatography Results

	Chloride	Bromide	Weak Organic Acids	Total
Control	1.6	0.0	0.0	1.6
Flux #1	5.7	12.8	1.8	20.3
Flux #2	4.2	0.5	0.0	4.7
Flux #3	5.3	0.8	0.0	6.1
Paste #1	1.1	0.4	0.0	1.5
Paste #2	1.4	0.9	0.0	2.3
Paste #3	1.6	1.2	0.0	2.9

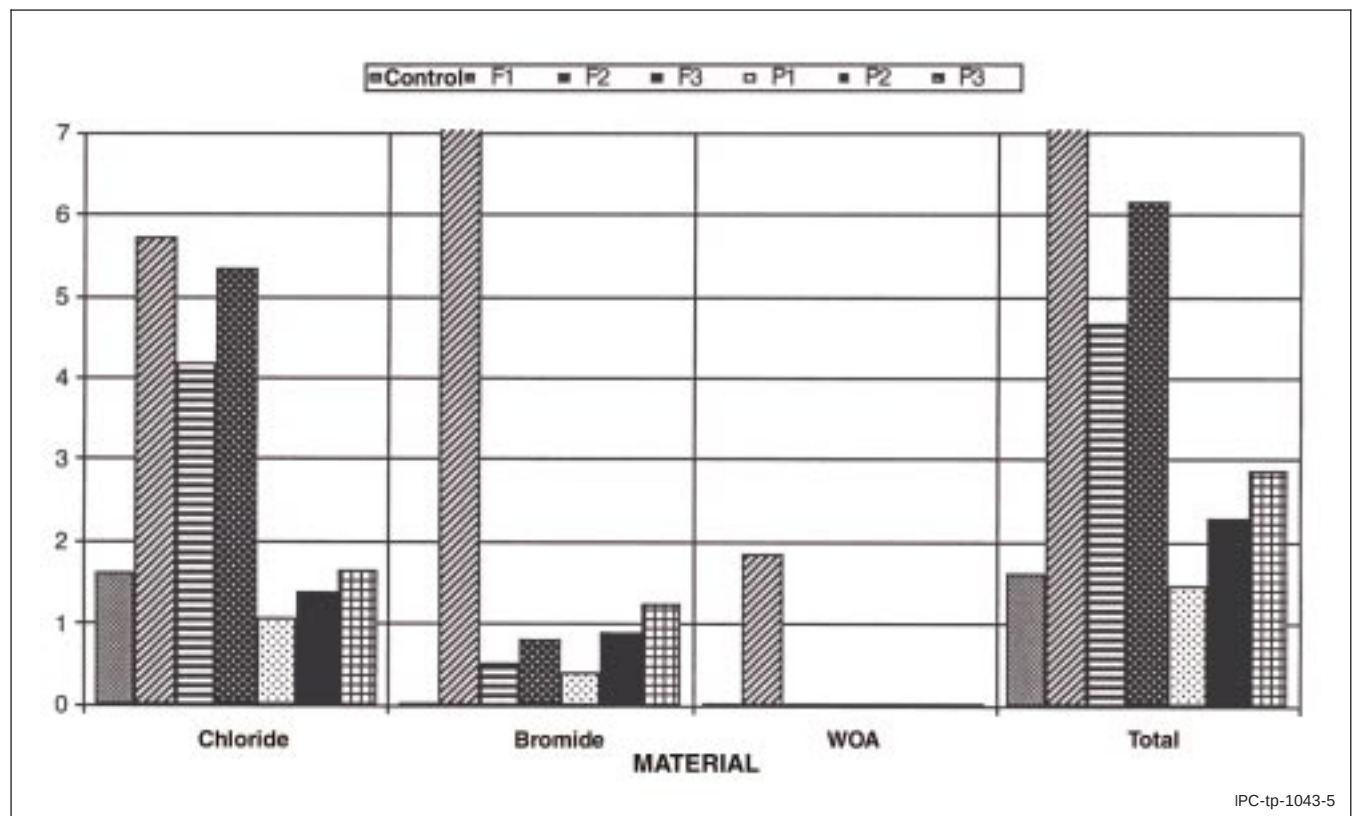


Figure 5 Ion Chromatography Levels

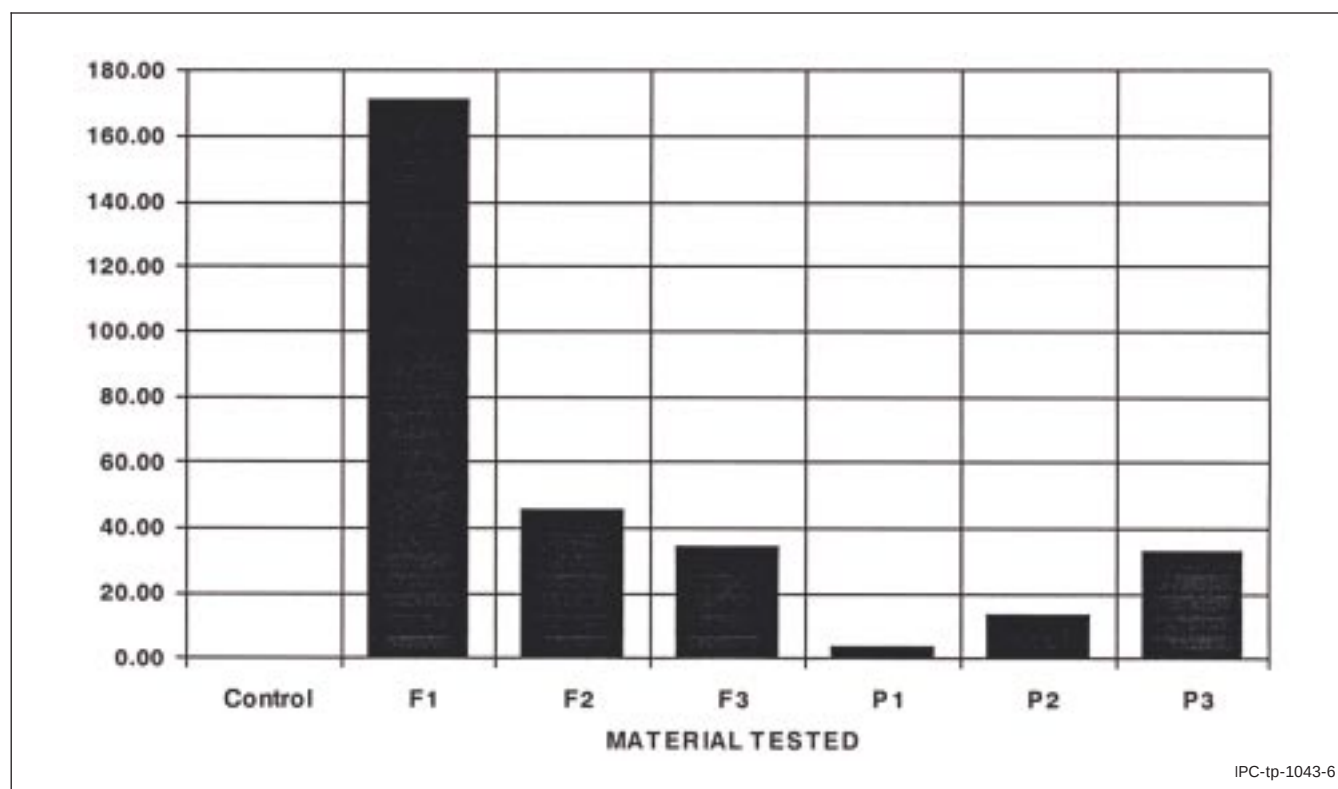
The leftmost bar in each block is “CONTROL.” The next bar to the right is F1, then F2, etc. The “TOTAL” value was determined as the sum of the three constituent elements. Chlorides are usually the greatest contributor to ionic contamination. With the exception of Flux #1, which showed a relatively high bromide level, the bromide content detected may have been contributed by the flame-retardants in the epoxy-glass laminate material and not from the flux. The characterization work indicated that Flux #1 contained bromide in the formulation. The other formulations did not contain bromide. Weak organic acids (WOAs) are found as activators in WSFs. Only Flux #1 exhibited a noticeable WOA level.

5.4 High Pressure Liquid Chromatography Results

The results of the HPLC analyses are shown in Table 21 and illustrated graphically in Figure 6. All values are in micrograms of contaminant per square inch.

TABLE 21 HPLC Results

Material	Residue
Control	0.00
Flux #1	171.00
Flux #2	45.60
Flux #3	34.80
Paste #1	4.00
Paste #2	13.80
Paste #3	33.20



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Figure 6 HPLC Results

HPLC analysis has been used in previous phases of this test program to determine the amount of rosin residues (usually rosin abietates) remaining on a board surface after processing. If the constituent materials of a flux are known, standards for each constituent may be prepared. Residues can then be analyzed and the amount of each constituent may be determined. Due to the proprietary nature of the WSF formulations, the constituent information was not made available; therefore, standards could not be prepared. The data listed above are in micrograms of total organic contaminant per square inch. Comparing this data to the benchmark data (Reference 4), it is apparent that water soluble fluxes have a lower organic residue level than rosin fluxes, which was expected due to the difference in flux chemistries.