AD-A057 364 NAVAL AVIONICS FACILITY INDIANAPOLIS IND F/G 9/5

PRINTED-WIRING ASSEMBLIES; DETECTION OF IONIC CONTAMINANTS ON.(U)

JUL 78 W T HOBSON

UNCLASSIFIED NAFI-MRR-3-72 NL

| OF |

AD A057364

END

DATE FILMED

9-78

DDC

DEPARTMENT OF THE NAVY
NAVAL AVIONICS FACILITY
INDIANAPOLIS, INDIANA 46218

IN REPLY REFER TO:
033.2:WTH:mjr
10380/1
25 May 1972
(A) Revised 9 June 1972
(B) Revised 30 Apr 1973
(C) Revised 21 Nov 1973
(D) Revised 19 Dec 1974
(E) Revised 24 Jul 1978

(9) MATERIALS RESEARCH REPORT NO. 3-72

SUBJECT: (6) Printed-wiring assemblies; detection of ionic contaminants on.

PERFORMED BY: R. J. DeNoon [redacted]
W. T. Hobson [redacted]

PREPARED BY: (10) W. T. Hobson 033.2

STATISTICAL ANALYSIS BY: W. D. Watkins, 033.3

REFERENCE: (a) Log Book 033-241, p. 79

I. Scope. A test for determining the ionic contaminants on printed-wiring assemblies was proposed, defined, and found feasible. The basis of the test is the change in electrical resistivity of the solvent with which the chemist washed the printed-wiring assembly. The solvent was 75% by volume, ACS Reagant Grade isopropyl alcohol. The resistivity is lowered when contaminants, such as flux residues, are dissolved. Establishing acceptance criteria for clean printed-wiring assemblies was beyond the scope of this investigation.

II. Introduction. The need for a test to determine the amount of ionizable residue on a printed-wiring assembly is recognized. The existence of a simple dependable accepted test was unknown to us. An ideal test method should:

1. Detect both water-soluble ionic residues and ionic materials within resinous residues.

2. Be simple and accurate.

3. Be performed with readily-available laboratory chemicals and equipment. This rationale was the basis of the study, reference (a).

III. Procedure. A test procedure was developed. "(Measured at 25°C)" (D)

A. Selection of solvent. The first task was the selection of a suitable solvent mixture. It was reasoned that isoproply alcohol would dissolve resinous materials and that water would dissolve ionic materials. Three different alcohol-water mixtures were evaluated. The results can be seen in Table I.

Table I.

Resistivity and Solvency of Isopropl Alcohol-water Mixtures for Rosin Flux

	ACS Reagent grade isopropyl alcohol make-up volume	620,000 ohm-cm. Distilled water make-up volume	Resistivity of mixture ohm-cm	Solvency of mixture for rosin flux
(a)	25 ml	75 ml	960,000	Inadequate
(b)	50 ml	50 ml	1,900,000	Adequate
(c)	75 ml	25 ml	8,600,000	Superlative

B. Other test procedures. The 25% isopropyl alcohol mixture (a) was eliminated because of its poor solvency for rosin flux. The water-extract resistivity test was not used because of the inadequate solvency of water for organic residues. The well known azeotrope of isopropyl alcohol, defined as 87.8% by volume isopropyl alcohol, (on page D-14 in the Chemical Rubber Company's "Handbook of Chemistry and Physics", 45th Ed.,) was considered. However, experiments were not conducted with the azeotrope because the resistivity of the 75% isopropyl alcohol and 25% distilled water mixture (c) proved to be convenient, and the azeotrope may have marginal solvency for ionic residues.

C. For both the 50% and 75%, by volume, isopropyl alcohol mixtures, it was found that the sensitivity of the electrical resistivity test was excellent. This fact was established by preliminary experiments, in which it was easy to detect known amounts of sodium chloride which had been deliberately added to isopropyl alcohol solutions of known resistivity. The test was sufficiently sensitive to detect extremely small quantities of sodium chloride. In other preliminary experiments, the electrical resistivity of known amounts of flux solids was monitored for both the 50% and 75% isopropyl alcohol mixtures, (b) and (c). The degree of response was greater using 75% alcohol. Therefore, the 75%, by volume, isopropyl alcohol mixture was used to develop and optimize the ionic-contaminants test.

D. Precision of data. At various known concentrations of flux solids, in 100 ml of 75%, by volume, isopropyl alcohol, the resistivity of the mixture was measured. This was done with four commercially available fluxes. This data on resistivity of flux mixtures was curve fitted using the least squares method. The curve chosen was a "right hyperbola". The general formula is: $(Y + H)(X + J) = K$. In this instance "Y" is the measured resistivity in ohm-centimetres and "X" is the weight of flux solids in grams per 100 ml of 75% isopropyl alcohol. The curves and the data points are shown in Graph I. The curves don't appear as hyperbolas because they are plotted on log-log paper. This was done for ease in displaying the data. The tabular data, from reference (a) may be plotted linearly if the reader so desires.

Note: Graph I appears on following page.

E. Trial procedure. Encouraged by the precision of the measured data, the following trial test procedure was developed:

1. Position a 115-mm diameter Pyrex funnel over a 180-ml electrolytic beaker. Suspend, by a wire hook, the printed-wiring assembly within the funnel. From a wash bottle, direct a fine stream of wash solution upon both sides of the assembly until 100-ml of the wash solution is collected in the beaker. It is imperative that the initial washings be included in the 100-ml volume to be measured for resistivity. Initial resistivity of the 75% by volume ACS grade isopropyl alcohol - 25% by volume distilled water wash solution must be equal to or greater than 6×10^6 ohm-cm.

2. Measure the resistivity of collected washings with a conductivity bridge or another instrument of equivalent range and accuracy. (A)

F. Proof of test procedure:

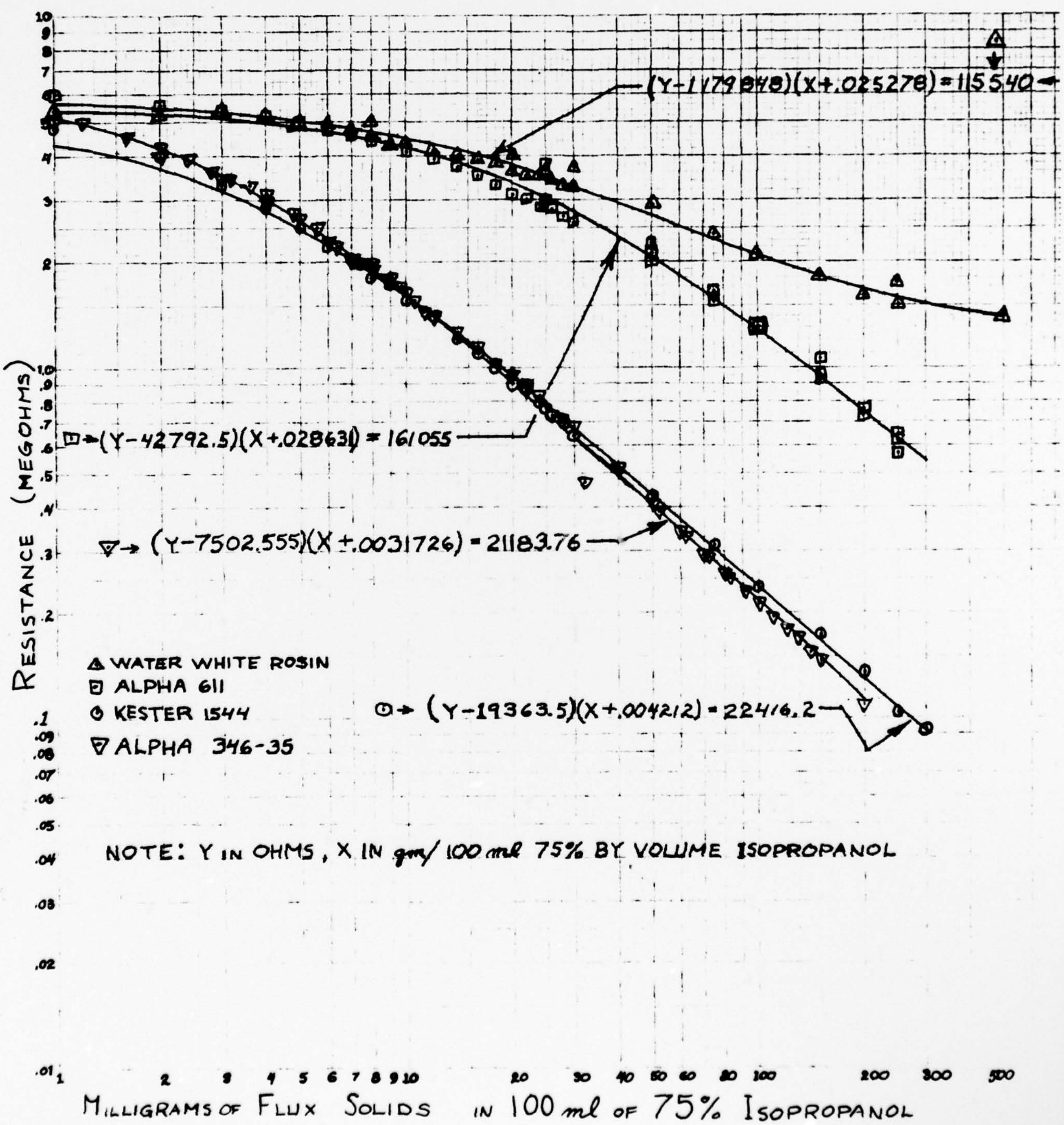
1. Jumper wires were installed and clinched in 17 AV2231 test-specimen printed-wiring boards. See Figure 1.

2. These boards, with installed jumper wires, were cleaned per MEPI 210.1.1 (see Appendix).

3. A 5% solids, by weight, solution of Alpha 346-35 flux was prepared. One half millilitre of this solution was applied to each specimen, on the side to be soldered. In other words, 25 mg flux solids was applied to each specimen. Afterwards the specimens were allowed to air dry.

4. Each board was float soldered for 15 sec. at 500 ± 10°F and allowed to cool.

GRAPH I


(Y-1179848)(X+.025278)=115540
□→(Y-42792.5)(X+.028631) = 161055
▽→ (Y-7502.555)(X+.0031726) = 2118.3.76
⊙→ (Y-19363.5)(X+.004212) = 22416.2
△ WATER WHITE ROSIN
□ ALPHA 611
⊙ KESTER 1544
▽ ALPHA 346-35
NOTE: Y IN OHMS, X IN gm/100 ml 75% BY VOLUME ISOPROPANOL
RESISTANCE (MEGOHMS)
MILLIGRAMS OF FLUX SOLIDS IN 100 ml OF 75% ISOPROPANOL

MRR 3-72
033.2:WTH:mjr
10380/1
25 May 1972

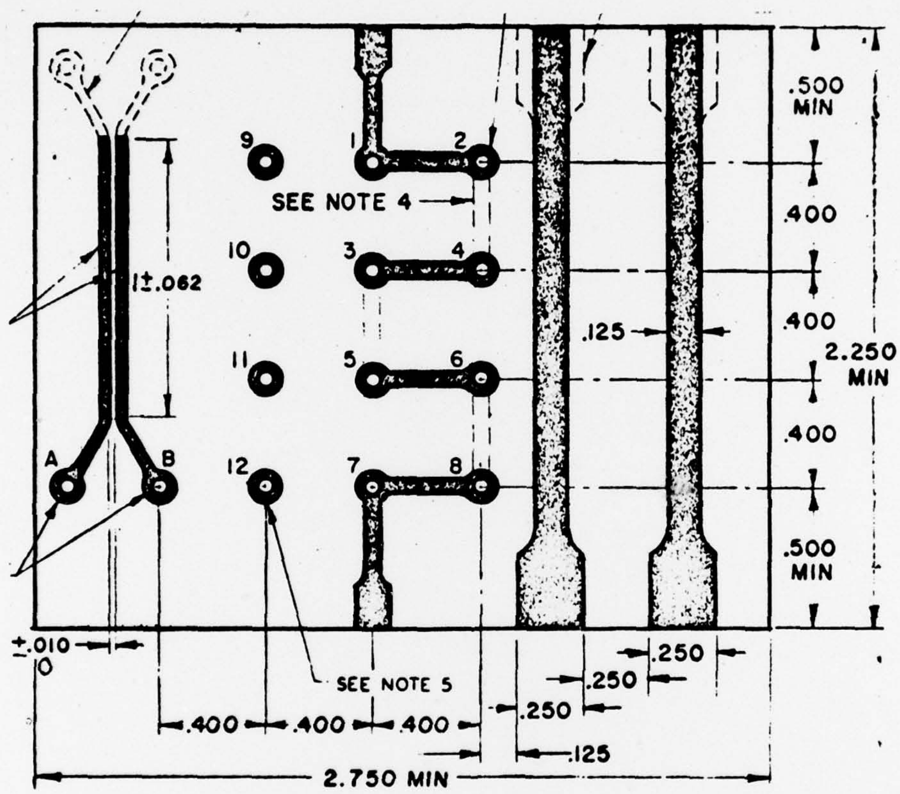


Figure 1.

5. Cleaning methods:

a. Cleaning. Method 1. Four boards were cleaned as follows:

(1) Soaked 1 minute in a 75% by volume mineral spirits and 25%, by volume, isopropyl alcohol mixture, (62A4B1) at room temperature, followed by an in-tank soft-bristle brushing.

(2) Step 1 was repeated in a second tank containing clean solvent (62A4B1) and using a clean brush.

(3) A wash bottle was used to rinse the board with ACS reagent grade isopropyl alcohol.

(4) Blown dry with air.

b. Cleaning. Method 2. Five boards were cleaned as follows:

(1) Cleaned per Method 1.

(2) Cleaned 2 minutes in a mild alkaline cleaner (Amchem Ridoline® 53, 4 oz. / gal., 150 ± 5°F).

(3) Rinsed 30 sec. in each of 4 cascading tap-water rinse tanks.

(4) Rinsed 30 sec. in deionized water from a weak anion base 2 column deionizer.

(5) Blown dry with air.

c. Cleaning. Method 3. Four boards were cleaned as follows:

(1) Cleaned per Method 2.

(2) Rinsed 30 sec. in $> 4 \times 10^6$ ohm-cm. water.

(3) Blown dry with air.

d. Cleaning. Method 4. Four boards were cleaned as follows:

(1) Soaked 1 minute in a mixture of 75%, by volume, mineral spirits and 25% isopropyl alcohol (62A4B1), at room

temperature followed by an in-tank soft-bristle brushing.

(2) A wash bottle was used to rinse the board with ACS reagent grade isopropyl alcohol.

(3) Blown dry with air.

(4) Cleaned 2 minutes in Shipley's NeutraClean® 68, 33% by vol., 120 ± 5°F.

(5) Rinsed 15 sec. in each of 4 cascading tap-water rinse tanks.

(6) Rinsed 15 sec. in deionized water from a weak anion base 2 column deionizer.

(7) Blown dry with air.

6. The results of the Proof-of-Test Procedure are given in Table 2. Proposed Test Procedure Evaluation.

Table 2 on following page.

TABLE 2.

PROPOSED TEST PROCEDURE EVALUATION

Cleaning Method	Individual Board Washings Resistivity ohm-cm	Statistical Analysis of Each Cleaning Method
1	1,950,000 2,600,000 2,500,000 2,200,000	N = 4, $\bar{X}$ = 2,312,500 ohm-cm σ = 255,868.6 ohm-cm $\bar{X} + 3\sigma$ = 3,080,106 ohm-cm $\bar{X} - 3\sigma$ = 1,544,894 ohm-cm t test method 1 vs 2 $\overline{\Delta X}$ = -1,757,500 ohm-cm t = -7.307 considered very significant
2	4,500,000 4,350,000 3,700,000 3,600,000 4,200,000	N = 5, $\bar{X}$ = 4,070,000 ohm-cm σ = 357,211 ohm-cm $\bar{X} + 3\sigma$ = 5,141,634 ohm-cm $\bar{X} - 3\sigma$ = 2,998,366 ohm-cm Predicts 42% < 4,000,000 ohm-cm Predicts none < 2,000,000 ohm-cm t test method 2 vs 3 $\overline{\Delta X}$ = -680,000 ohm-cm t = -2.089 10% > P > 5% considered significant
3	3,900,000 5,200,000 5,000,000 4,900,000	N = 4, $\bar{X}$ = 4,750,000 ohm-cm σ = 502,494 ohm-cm $\bar{X} + 3\sigma$ = 6,257,481 ohm-cm $\bar{X} - 3\sigma$ = 3,242,519 ohm-cm Predicts 6.68% < 4,000,000 ohm-cm Predicts none < 2,000,000 ohm-cm
4	1,900,000 1,650,000 1,250,000 2,000,000	N = 4, $\bar{X}$ = 1,700,000 ohm-cm σ = 289,395 ohm-cm $\bar{X} + 3\sigma$ = 2,568,188 ohm-cm $\bar{X} - 3\sigma$ = 831,812 ohm-cm

IV. Discussion.

A. The test results indicate that if the boards contain very little flux residue, the first 100 ml of washings collected will have a resistivity greater than 2×10^6 ohm-cm. Hence the test procedure does have practical feasibility.

B. Also, the Students "t" test was used to determine if the resistivity values obtained on solutions from boards cleaned by different processes would be significantly different. The "t" test indicates that Methods 2 and 3 are significantly different.

C. The statistics of Methods 2 and 3 are of interest. Method 2 indicates that 42% of boards cleaned would have resistivities less than 4×10^6 ohm-cm, with no boards below 2×10^6 ohm-cm. Method 3 indicates that only 6.7% of boards cleaned would have resistivities less that 4×10^6 ohm-cm, with no boards below 2×10^6 ohm-cm. The confidence level of the statistical analysis, while defensible, would have been higher had the sample size been larger.

D. For consistent results it is mandatory that ACS reagent grade isopropyl alcohol and distilled water with a minimum resistivity of 4×10^6 ohm-cm be used to mix the wash solution. It is also very important that scrupulously clean laboratory ware be used to store and handle the wash solution. Any time that the wash solution does not have a resistivity equal to or greater than 6×10^6 ohm-cm, it must be discarded and a new solution prepared. The laboratory ware must be clean.

V. Conclusion. A simple test, based upon the ability of ionic contaminants to lower the resistivity of a liquid, has been developed. The test procedure is simple, straightforward, and appears to be a useful method for detecting ionic contaminants on printed-wiring assemblies.

B. C. Vaughn

B. C. VAUGHN, Chief, Metallurgical Materials Branch

Norton Gerber

NORTON GERBER, Chief, Physico Chemical Materials Branch

APPROVED BY:

Carl Ferguson

CARL FERGUSON
Manager, Materials Laboratory and Consultants Division

MRR No. 3-72
033.2:WTH:mjr
10380/1
25 May 1972

APPENDIX A.

List of apparatus used.

1. Printed-wiring specimen AV-2231 (Illustrated in MIL-P-55110A)

2. Conductivity meter, Model RC 1632, Industrial Instruments, Inc.

3. Wash bottle, polyethylene, squeeze-type, Nalgene® 78-0518-7B

4. Electrolytic beaker 180 ml. Corning® 422410

5. Brush, natural bristle, 7510-550-8446

6. Funnel, ribbed, Mooney Airvent® 115 mm top dia., Fisher Scientific 10-381B

List of chemicals used.

1. Rosin, water-white, LLL-R-626, Class A, Type 1, Grade WW

2. Flux, liquid, Kester® 1544 (Control No. C-7641)

3. Flux, liquid, Alpha® 611, MIL-F-14256, Type A (Lot No. 8012436)

4. Flux, liquid, Alpha® 346-35

5. Distilled water, minimum resistivity shall be 4×10^5 ohm-centimetres

6. Isopropyl alcohol, ACS reagent grade (See reference (a) for analysis)

7. 75% isopropyl alcohol - 25% distilled water. See Table 1. Minimum resistivity shall be 6×10^6 ohm-centimeters

8. Mineral spirits - isopropyl alcohol mixture (62A4B1)

   a. Thinner, paint (mineral spirits) TT-T-291, Gr. 1 75% by volume

   b. Isopropyl alcohol (99%) TT-T-735, Gr. A 25% by volume

9. Sodium chloride ACS reagent grade
10. Solder 60:40
11. Cleaner, Amchem Ridoline® 53
12. Cleaner, Shipley's Neutra Clean® 68
13. Cleaner, Kelite® No. 351
14. Cleaner, Oakite® 90
15. Hydrochloric acid, ACS reagent grade

TITLE: CLEANING PRINTED WIRING BOARDS FOR SCREENING

1. General: This process is used to prepare the surface of printed wiring boards or blanks to receive a screened resist for etching, plating or solder dipping.

2. Procedure:

   a. Rack boards on a stainless steel fixture.

   b. Vapor degrease (vapors only).

   c. Hot water rinse - 200°F. - 5 to 10 seconds.

   d. Acid clean (Solution 1) - 4 minutes.

   e. Spray rinse - 10 to 15 seconds.

   f. Alkaline rinse (Solution 2) - 10 to 15 seconds.

   g. Hot water rinse - 200°F. - 10 to 15 seconds.

   h. Cold water rinse - 10 to 15 seconds.

   i. Acid rinse (Solution 3) - 5 to 10 seconds.

   j. Cold water rinse - 10 to 15 seconds.

   k. Hot water rinse - 200°F. - 5 to 10 seconds.

   l. Blow dry with clean air.

   m. Unrack boards and place on clean paper covered tray.

SOLUTION 1

Kelite #351 --------------------- 10% (by volume)

160 to 180°F.

SOLUTION 2

Oakite 90 ---------------------- 4 oz./gal.

160 to 170°F.

SOLUTION 3

HCL ---------------------------- 50% (by volume)

Room temperature

3. SAFETY:

> Avoid breathing degreaser vapors. The aqueous acid and alkaline solutions used are poisonous and are skin irritants. In case these solutions contact the skin or clothing, flush well with water.

Prepared by J. L. Stockdale
J. L. STOCKDALE, MFE-31

J. L. Bryant
J. L. BRYANT
Mgr., Mfg. Engr. Div.

John E. Van Osdol
J. E. VAN OSDOL
Chief, Process Engr. Br.

2

IMPORTANT!

It is imperative that all apparatus used in this procedure be scrupulously clean! Glassware was washed with MICRO laboratory cleaning solution (2 oz/gal). It was rinsed with tap and distilled water and further cleaned with the traditional chromic-sulfuric cleaning solution until there were no water-breaks observed with distilled water rinses. Beakers and funnels were allowed to air dry inverted on a polyethylene-coated wire mesh rack. If glassware stood overnight, it was rinsed with distilled water. If there was a water-break, the items were recleaned.

PROCEDURE

1. Support a convenient sized funnel over an electrolytic beaker. (In our investigations a Mooney Airvent® funnel, 115-mm top diameter (Fisher Scientific 10-381B) and 180-ml electrolytic beakers (Corning® 422410) were used.)

2. Suspend the specimen over the funnel.

(C) 3. Wash both sides of the specimen using 100-ml of a 75% ACS 2-Propanol (Fisher CERTIFIED A-416) - 25% distilled water mixture dispensed from a polyethylene wash bottle, squeeze type (Nalgene® 2401-0250). NOTE: The ratio of wash solution to surface area shall be maintained at 10 ml/sq. in. of board area. Otherwise, the results will be erroneous.

4. Collect the washings in a 180-ml electrolytic beaker. (For convenience, the beakers were pre-marked at 100-ml capacity with a Vibroengraver (FSN 5130-596-8404).

5. Thoroughly rinse funnel with 50-ml of the alcohol-water wash solution between specimens.

6. Measure resistivity of the 100-ml washings with a Type RC-16B2 Conductivity Bridge (Beckman Instruments). Between measurements, flush the probe with a stream from a wash bottle containing the alcohol-water mixture. The probe should be conditioned before the first measurement by immersing in 100-ml of the alcohol-water mixture. "(Measure at 25°C)" (D)

Distilled water is prepared by passing tap water through a Pressure Bantam Demineralizer (Model BD-2, Barnstead Still and Sterilizer Co.) using a Standard (Barnstead 0802) demineralizer cartridge. This demineralized water is fed to a Barnstead ELH-3 Water Still. Non-demineralized water is used to cool the condensor. Distilled water is collected in a 13-gallon polyethylene carboy (FSN 8125-993-8040) equipped with a Type "T" Spigot (14114, U. S. Plastic Corp., Lima, Ohio).

Distilled water produced and stored for as long as five days routinely measures 8×10^6 ohm-cm resistivity. (As the carboy empties, the last gallon is thrown away to prevent ionic concentration in the new distilled water placed in the reservoir.)

Originally, distilled water was redistilled but this was found to be unnecessary when freshly distilled water as above was used for the 2-propanol-water wash solution.

Just as important as good water is good 2-propanol. It has been found that ACS 2-propanol must be used to insure attainment of the necessary resistivity of the wash solution. To prevent cross contamination from other sections of the laboratory, a 5-gallon container was reserved to be used for these experiments.

The 2-propanol-distilled water wash solution is routinely retained in the polyethylene wash bottle (Nalgene® 2401-0250) and 1-gallon polyethylene large narrow mouth bottles (Nalgene® 2202-0010). Any time that the wash solution does not have a resistivity equal to or greater than 6×10^6 ohm-cm, it is discarded and a new solution prepared.

APPENDIX C.

IMPORTANT!

It is imperative that all apparatus used in this procedure be scrupulously clean. Plastic ware has been substituted for glass ware. New plastic ware is cleaned with a good detergent, rinsed in distilled/deionized water and air dried. Before testing, the lab ware is rinsed with test solution. During test, a rinse with test solution between specimen testing is adequate to insure against cross-contamination. If this plastic ware is used exclusively for this testing, minimum recleaning is necessary.

PREPARATION OF SOLVENT EXTRACT TEST SOLUTION

Prepare a test solution of 75% by volume ACS reagent grade isopropyl alcohol and 25% by volume distilled/deionized water. Pass this solution through a mixed bed deionizer cartridge (Barnstead D8902, Ultra-Pure, Hose-Nipple Cartridge, or equal). After passage through the cartridge, typical resistivity of the solution will be 25×10^6 ohm-cm (conductivity-0.04 micromho/cm). Replace the deionizer cartridge when the exchanged solution resistivity is less than 6×10^6 ohm-cm (conductivity - greater than 0.166 micromho/cm).

Place test solution in a 500-ml wash bottle (Bel-Art F 11621-0016, or equal). Test solution may also be dispensed in the following manner: Elevate a five-gallon plastic rectangular carboy containing the test solution so that the distance from the top of the contained solution to the top of the work bench is fifty-six inches ± two inches. Pass the solution through Tygon tubing to a mixed bed deionizer cartridge (Barnstead D8902, Ultra-Pure, Hose-Nipple Cartridge, or equal), through an in-line conductivity cell, thence to a stainless steel needle valve, and through additional Tygon tubing terminating in a spray nozzle; (CRES 316) 2.75 inches ± 0.25 inches(70 mm ± 5 mm) long with a nominal inside diameter of 0.0625 inches (1.59 mm).

PROCEDURE

1. Support a convenient sized polyethylene funnel over a suitable polyethylene container. Pre-mark the container for the volume of test solution required for the test.

2. Suspend the specimen within the funnel.

3. Direct the test solution, in a fine stream, onto both sides of the assembly until 10 ml of test solution is collected for each square inch of assembly area. Assembly area includes the area of <u>both</u> sides of the

board <u>plus</u> the area of the components mounted thereon. It is imperative that the initial washings be included in the test sample.

4. Measure and record the resistivity/conductivity of the collected test solution with a conductivity bridge or other instrument of equivalent range and accuracy.

5. Between measurements, rinse the conductivity cell, funnel, and container with fresh test solution. Condition the probe before the first measurement and between tests by immersing it in the fresh test solution.

MRR No. 3-72
033.2:WTH:mjr
10380/1
25 May 1972

DISTRIBUTION:

NAVELEX J. A. Wyatt 05183
NAVAIR F. Schulte 53355
NAVAIR J. J. Gurtowski 52032C
030
033
033.1 J. E. Mitchell
033.2 W. T. Hobson via B. C. Vaughn
033.2 R. J. DeNoon
033.2 F. J. Smith
033.3 W. D. Watkins via W. W. Turner
033.3 D. A. Werner
033.4 N. Gerber
033.4 R. E. Martz