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The Use of Radioisotopes to Determine the Chemistry of Solder Flux

Abstract: When rosin flux activated with amine halides is used in soldering printed circuit boards, metal halides are formed; these halides cannot be removed with organic solvents and thus may cause corrosion. The use of alkanol amines as solder fluxes has been investigated. After soldering, the flux can be removed by rinsing with water. Throughout this investigation radioisotopes were used to identify the compounds studied.

Introduction

The purpose of flux[1] in soldering is to remove oxide surface films on metal and molten solder and to prevent reoxidation of the surfaces during soldering. The flux commonly used for dip soldering printed circuit panels is a solution of rosin in an alcohol to which is added an "activator." The commercially available solutions differ mainly in the type of activator used. (The activated fluxes used in this study contained hydrochloric acid salts of aliphatic amines.) After soldering, the boards are immersed in a solvent to remove the flux. Nevertheless, certain electrical failures, especially occurring at high humidity, and the formation of corrosion products have been attributed to the use of activated flux in the soldering process.

An investigation was made using commercial flux with radioactive amine salt added, the radioactivity being located in the halide part of the molecule in one series of tests, and in the amine part of the molecule in another. It was found that during soldering, metal halides ($PbCl_2$, $SnCl_2$, $CuCl_2$), formed by chemical reaction between the amine salt and solder, cannot be removed with the type of commercial solvent generally used. Depending on experimental conditions, 30 to 50 percent of the activator can react in this way.

Because $PbCl_2$ is slightly soluble in dilute NH_4OH solutions, circuit panels can be effectively cleaned using such a solution; they remove about 90 percent of the metal halides. However, the cause of the trouble remains —the use of amine salts. Metal oxides can be dissolved in a variety of acids, but this will always produce metal salts which are difficult to remove quantitatively and thus eventually will cause corrosion. In this respect, amine-hydrochlorides hardly differ from free hydrochloric acid.

Rather, the removal of oxide films must be accomplished by predominantly non-ionic reactions, leaving non-ionic reaction products. Such residues remaining after cleaning will be far less corrosive than metal salts. Investigation revealed that a mixture of triethanolamine (90 percent) and monoethanolamine (10 percent) satisfies these requirements rather well. The mixture can be applied with a spray gun to the panels to be soldered. It solders just as well as commercial activated flux, but it has the advantage that excess flux can be removed by rinsing with water.

Variations of this formulation are possible, e.g., a small amount of ethanolamine in glycerine will solder nearly as well; for some applications the monoethanolamine can be omitted; triethanolamine can be mixed with polyethylene glycols to form a flux, either solid or liquid at room temperature depending on the molecular weight of the polyglycol. Without doubt other compounds may be found which can be used as a water-soluble flux.

When radioactive triethanolamine was used, it was found that the amount left on the panels after rinsing was of the order of a few milligrams, depending mainly on the length of time the panels were rinsed.

A close correlation was found between the insulation resistance of rinsed test panels measured at high relative humidity and at the amount of flux left on the panels. Thus the insulation resistance of test panels may be used to estimate the degree of cleanness obtained in production.

The average insulation resistance of test panels soldered with triethanolamine as flux and cleaned by immersing the panels for about two minutes in running water is the same as the insulation resistance of the original panels at the same level of humidity. On the other hand, panels soldered with activated rosin flux and cleaned with organic solvents using ultrasonic agitation, as is customary, show insulation resistance at least ten times smaller.

In the sections that follow, experiments with labeled commercial flux are described which lead to the search for a better agent. The properties of a good flux are also listed. Next, experiments with either monoethanolamine or *triethanolamine*-labeled flux are described, followed by a short discussion of the insulation resistance of test panels.

All data are calculated from observed count rates of incorporated radioactive tracers. Although the counting techniques involved are mentioned, their discussion is beyond the scope of this paper.

Evaluation of activated rosin flux

◆ *Activated rosin flux labeled with ethanolamine hydrobromide*—Br-82

For reasons of economy, hydrobromides rather than hydrochlorides were used.[2] The two types of amine salts are sufficiently alike in their chemical behavior to permit this substitution. The short half life of Br-82, nearly 36 hours, is no serious disadvantage if the experiments are carefully planned.

Isotope Br-82 is a beta-gamma emitter. Samples were assayed by gamma counting, thus avoiding complications caused by self-absorption when beta counting is used.

The isotope is available from the Oak Ridge National Laboratory in the form of a KBr-82 solution. The solution is acidified with the calculated amount of H_2SO_4 solution and frozen. H_2O and HBr are sublimed in vacuum into a flask containing free amine solution cooled to liquid N_2 temperature.[3] The recipient is heated slowly to room temperature allowing the acid to react with the free amine. The solution is then frozen again and H_2O and excess amine are sublimed into a second recipient. The amine salt remains and is dissolved by rinsing the flask with 5 to 10 ml of commercial flux solution.

Two types of amine salts were prepared:

Dimethylamine-hydrobromide Br-82 $(CH_3)_2$ NH HBr^{82}

Monoethanolamine-hydrobromide Br-82

$HOCH_2CH_2NH_2 \cdot HBr^{82}$

These were to be used with two commercial fluxes containing the corresponding hydrochlorides as activator. As it turned out, the two fluxes were very similar. For brevity, the data for the ethanolamine-HCl flux will be used throughout this discussion.

Flux was applied to printed circuit panels with a long-handled brush. The coatings are quite reproducible. The amounts applied are of the same order of magnitude as those in production. The circuit panels were ½ by 1-inch sections of production panels containing copper conductors and two ½-watt resistors. These small circuit panels fit into the well of the gamma spectrometer crystal, used to assay all samples. Both epoxy-glass and epoxy-paper laminates were used. The fluxed boards were soldered by dipping the side to be soldered in molten solder for about seven seconds, while holding the sample with long tweezers. To avoid contamination, fresh solder was used for each sample. Conventional nickel laboratory crucible covers, each holding a few ml of molten solder, were used as "solder pots."

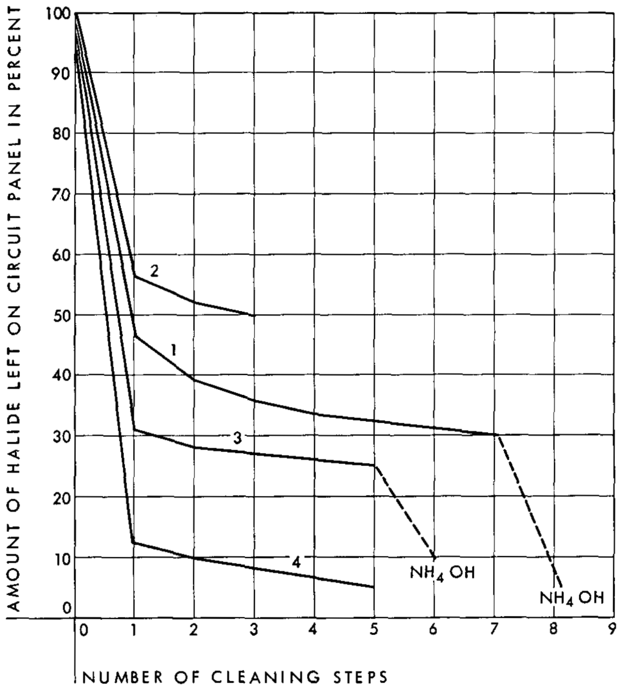
After soldering, the circuit panels were cleaned in about 10 ml of solvent in a test tube in an Ultra Sonorator for two minutes. Then the panel was removed, rinsed with a ml of solvent, and circuit panel and solvents were assayed in the gamma spectrometer. The observed count rate is proportional to the amount of Br-82 in the sample. Neglecting the differences between Br and Cl, the count rates can be expressed as mg halogen in the sample.

Originally all this halogen was in the form of amine-hydrochloride, but it cannot be assumed that the halogen remains in this form. The radioactivity recorded is associated with the halogen and is independent of chemical reactions which have taken place.

The amount of halogen on the circuit boards after successive cleaning steps is plotted in Fig. 1. The

Figure 1 **Cleaning experiments using commercial flux with ethanolamine hydrobromide (Br-82) tracer.**

The curves resulted using (1) Isopropanol followed with 3N ammonia; (2) 1,1,1, Trichloroethane, no agitation; (3) 1,1,1, Trichloroethane with ultrasonic agitation followed by 3N ammonia without agitation; (4) 3N ammonia, no agitation.



amount is expressed as a fraction of the original amount applied to the board. To compare different solvents, this initial amount, corresponding to a layer of flux approximately 0.002 inch thick, was kept constant.

Isopropanol dissolves both rosin and activator. After an initial decrease to about 40 or 50 percent, no substantial amounts are removed by successive cleaning steps (see curve 1, Fig. 1).

Curves 2 and 3 in Fig. 1 refer to experiments with 1,1,1 trichloroethane which is a solvent for rosin but not for amine salts. Curve 2 refers to experiments in which the ultrasonic generator was purposely detuned, so that there is virtually no agitation; Curve 3 refers to maximum ultrasonic agitation. Apparently ultrasonic agitation removes small salt crystals from the surface and suspends them in the solution.

Curve 4 shows the cleaning effect of a 3N NH_4OH solution. Rosin dissolves slowly in alkaline solutions forming a collodial solution. Amine salts, of course, are very soluble in water. Ultrasonic agitation has no effect in this case.

After a number of cleaning steps in isopropanol or trichloroethane, if the sample is immersed in dilute NH_4OH solution, a substantial amount of activity is removed (as illustrated by the dashed sections of curves 1 and 3).

Other solvents which were tried:

1. Trichloroethylene, similar to 1,1,1 trichloroethane. Initial cleaning leaves on between 40 and 50 percent; subsequent cleaning removes only small amounts. After five cleaning steps the remaining activity is still 35 to 40 percent. If now the sample is immersed in 3N NH_4OH only 10 percent remains.

2. 3N KOH Solutions. This solvent is about as effective as 3N NH_4OH solution, but there is some damaging effect to the circuit panel. Dilute Na_2CO_3 solutions do not clean as well as 3N NH_4OH. The detergent solutions tried were ineffective.

3. Dimethylformamide. This solvent removes the activity even faster than NH_4OH but it dissolves the epoxy resin in the circuit board.

The optimum NH_4OH concentration is 3N to 5N; more dilute solutions do not remove the activity as well and more concentrated solutions have adverse effects on circuit panels or electrical components. Adding detergents to 3 to 5N NH_4OH solutions does not increase the removal but tends to leave a detergent film on the panel. At high levels of humidity this would decrease the resistance between adjacent conductors on the panel.

At elevated temperatures (from room temperature to about 50°C), the removal process accelerates slightly but the total amount removed is about the same.

Figure 1 shows a significant difference between alcohol and ammoniacal solutions. The small quantities of amine salts on the panels should be readily soluble in both alcohol and aqueous solutions and thus alcohol should clean just as well as NH_4OH solutions. Notice furthermore that 3N NH_4OH does not remove all the activity.

It was found that NH_4OH solutions can remove flux from the surface of epoxy paper laminates nearly quantitatively. If a 10 lambda drop (1 lambda is 0.001 ml) of flux solution is put on a circuit panel and dried under an infared lamp, the spot can be removed with a cotton swab dipped in 3N NH_4OH solution leaving on less than 3 percent in one cleaning and less than 0.5 percent if the board is subsequently rinsed with some NH_4OH.

The failure of alcohol to remove the radioactivity from the soldered boards could be caused by strong absorption of flux on the board material in alcoholic solutions. To check this point, the absorption of flux on circuit board material was determined in the usual way.

Solutions of labeled flux were prepared ranging in concentration from 0.01λ flux per ml solvent to 2λ flux per ml solvent. In one series alcohol was used as solvent, in another series 3N NH_4OH was the solvent. To 5 ml of each solution was added 0.1 g of powdered circuit board material and the mixture was shaken until equilibrium was established. After centrifuging, a sample of the supernatant liquid was assayed. From initial and final concentrations the amount of flux absorbed on the circuit board can be calculated. Absorption curves are obtained by plotting the amount of flux absorbed on 1 g powdered circuit board material versus the equilibrium concentration.

It was found that there is no absorption from ammoniacal solution and weak absorption from alcoholic solution. However, this absorption from alcoholic solution is far too small to account for the observed activity in curve 1, Fig. 1.

Thus it is unlikely that ammoniacal solutions or isopropanol leave any flux on the boards. The remaining activity consistently found on soldered boards must refer to something different from the original amine salt.

It was found that the radioactivity remaining after cleaning with 3N NH_4OH solutions can be removed by dissolving the solder in 8N HNO_3 followed by a rinse with water. Only 3 percent of the activity of the cleaned samples remains. Thus the major part of the radioactivity remaining after cleaning is indeed located at the soldered junctions rather than at the surface of the board. It is unlikely that this activity refers to flux occluded in the solder. If the soldered spots are remelted—which should bring any flux to the surface—and the boards are immersed again in 3N NH_4OH, about 90 percent of the activity still remains.

Thus there is evidence to indicate that the remaining activity is the product of a reaction involving the hydrochloric-acid part of the amine salt and solder, mainly $SnCl_2$ and $PbCl_2$ and possibly $CuCl_2$. These salts are insoluble in alcohol but soluble in dilute NH_4OH solution. Remelting of the soldered spots would not bring a major portion of these salts to the surface.

Summarizing these results, the following picture of the solder process emerges. During soldering, part of the activator[4] in the flux reacts with the solder and the metals on the circuit board. After soldering the excess flux, chemically unchanged, can easily be removed, even with solvents in which the rosin but not the activator is soluble, provided there is sufficient agitation to remove the salt crystals mechanically. The metal chlorides formed are not soluble in chlorinated hydrocarbons or alcohols and cannot be removed in this way. These salts may well cause corrosion over periods of time. Three to 5N NH_4OH solutions will remove both the excess flux and much of the metal salts but a remainder of 5 to 10 percent persists probably because it is not accessible to the NH_4OH solution. Corrosion will be reduced but not eliminated.

Sufficient evidence to substantiate this hypothesis was obtained from experiments using activator labeled in the amine part, to be discussed next. At the beginning of the program experiments were planned using labeled rosin, however the development of the new flux made these experiments unnecessary.

Corrosion caused by salts such as $SnCl_2$ even in minute quantities, can be quite severe over a period of time. Moisture and oxygen in the air will convert the halide into the dioxide liberating hydrochloric acid which in turn can react with solder or oxide surface film. The chain reaction would stop only after all the HCl had escaped.

The mechanism may be expressed as follows:

$$SnCl_2+H_2O \rightarrow Sn(OH)Cl+HCl$$

$$Sn(OH)Cl+H_2O+\tfrac{1}{2}O_2 \rightarrow SnO(OH)_2+HCl$$

$$SnO(OH)_2 \rightarrow SnO_2+H_2O$$

$$SnO+2HCl \rightarrow SnCl_2+H_2O$$

$$PbO+2HCl \rightarrow PbCl_2+H_2O$$

In addition, the electrochemical potential set up between Cu and Pb or Sn, with electrolytes like $SnCl_2$ or $PbCl_2$ present, would result in the oxidation of solder.

- *Activated rosin flux labeled with ethanolamine*-C-14 *hydrochloride*

Ethanolamine-C-14 hydrocloride is commercially available and flux can be labeled by dissolving a few mg of the radioactive salt in 5 ml of flux. Since C-14 is a weak beta-emitter, samples were assayed in a liquid scintillation spectrometer.[5] The scintillation solutions were prepared by dissolving diphenyloxazole (3 g/l) and bis-(phenyloxazoyl)-benzene (.1 g/l) in freshly distilled toluene. It is possible to assay the amount of flux on circuit panels immersed in this solution. However, the panel interferes with the energy transfer mechanism and with the propagation of the emitted light, reducing the counting efficiency. It is necessary to determine this counting efficiency before count rates can be expressed as mg of flux.

It was observed that part of the radioactive amine on the panel dissolves in the counting liquid. Therefore panels should not be used for further cleaning experiments after they are assayed.

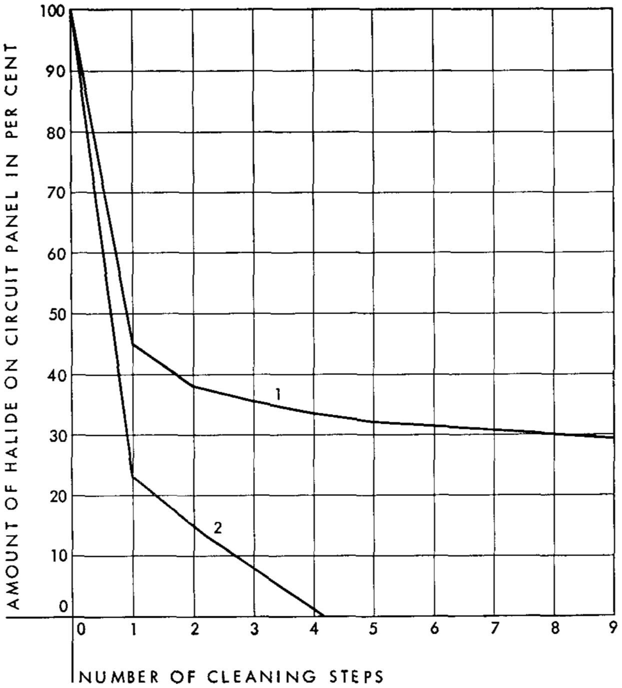


Figure 2 **Cleaning experiment using commercial activated flux with (1) Br-82 labeled activator, and (2) C-14 labeled activator.**

To avoid these complications, the flux removal process was studied by assaying the cleaning solutions rather than the panels.

After soldering, each panel was immersed in 10 ml of solvent (isopropanol) and agitated ultrasonically for two minutes in the same manner as previously described. The panel was then removed and rinsed with a ml of solvent. The solution was assayed by introducing an aliquot into the liquid scintillation solution and counting. The cleaning was repeated a number of times using fresh solvent each time and assaying each solution. Finally as a check the panel was assayed. After determining the counting efficiency the amount of flux removed in each step was calculated from the observed count rates.

Cleaning experiments with Br-82 labeled flux are compared in Fig. 2 with identical experiments using C-14 labeled flux. The solvent used in both test series was isopropanol. The original amount of flux on the panels is about the same. Data are the averages of three experiments.

It is evident that the amine part of the activator can readily be removed, leaving a residue of 1 or 2 percent; but the halide part is not removed even after 10 cleaning cycles. This must mean that the Br-82 activity remaining after the first few cleaning cycles does not refer to the original activator but to reaction products of the amine salt and metal on the board as discussed above.

The amount of flux on the panels after soldering (but before cleaning) was determined in each experiment. For C-14 labeled flux about 70 percent of the original activity was found on the panels immediately after soldering, while for Br-82 labeled flux about 90 percent was left. The amine salt is not volatile at soldering temperature, but the free amine will evaporate during soldering. Since free amine is formed by the reaction, the difference in activity probably corresponds to the amount of metal halides formed.

Development of a new flux

It is not likely that flux can be removed quantitatively on a production scale. Therefore, the use of acids or salts as a flux should be avoided since the residue after cleaning will be corrosive. Other reactions which would remove oxide films are (1) reduction by organic compounds at elevated temperature, and (2) complex formation.

The conductors on printed circuit panels are copper. The leads of components usually are "tinned" copper. Copper oxide can be reduced by a variety of compounds. The element is also known for the ease with which it forms complexes, often at much lower temperatures. It should, then, be relatively easy to find chemicals which can be used as flux.

Cu-Ammin coordination compounds are formed with NH_3, primary amines, α-aminoacids and some heterocyclic compounds, but not with tertiary amines.

Cu-Oxygen complexes are formed with α-oxy acids, α-dihydroxy compounds, with enols like β-diketons or β-ketoesters, salicylaldehyde and similar compounds.

Many of these coordination compounds are water soluble, mostly under dissociation, with Cu either in the cation (e.g., $Cu(Amm)_n(OH)_2$) or in the anion (e.g., Cu-tartrato complex), but a few are not ionized (glycol or glycerol-chelates). This last group should receive special attention.

Further considerations limit this long list of possible chemicals to relatively few:

1. Regardless of the type of reaction, the boiling point of the compound should be higher than the melting point of the solder used, otherwise the flux evaporates leaving the surface without protection against reoxidation.

2. Since water is the cheapest cleaning agent, flux components should preferably be water soluble. Also the reaction products formed during soldering should dissolve in water. Since carbonaceous residues will not dissolve, the flux must be stable at temperatures up to 200°C.

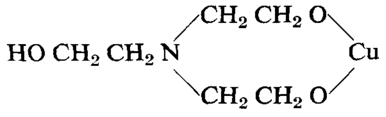
3. For industrial use, the compounds must be inexpensive, readily available and non-toxic.

Eutectic PbSn melts at 183°C (62 percent Sn). This eliminates nearly all water soluble primary amines since their boiling points are too low. It leaves polyalcohols, aminoalcohols and aminoacids. Salicylaldehyde (boiling point 196°) and ethylacetoacetate (boiling point 180°) are not reactive enough, are not water soluble and the ester is much too expensive.

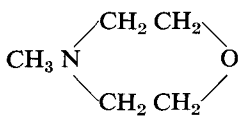
Glycerol (boiling point 290°) can be used as a flux for very clean surfaces. Triethanolamine (boiling point >300°) will solder badly oxidized Cu surfaces. The boiling point of monoethanolamine (170°) is too low for normal solder but it can be used with low melting solder (e.g., indium solder). However, if some monoethanolamine is mixed with glycerine the mixture will solder nearly as well as triethanolamine. Glycine (boiling point 290°) will not remove copper oxide.

These solder experiments are, of course, straightforward. All that is needed is solder, the compound to be tested and copper strip or printed circuit panels. It is more illustrative to proceed in a different way. A strip of copper is oxidized with a Bunsen burner; solid solder is placed on it and the assembly is heated in the liquid to be tested. As the temperature rises the black CuO should slowly change into bright Cu; at about 180 to 185° the solder should flow easily over the surface. Triethanolamine is about the best in this respect of all compounds which were tested.

The chemical reactions involved have not been studied quantitatively. The color of triethanolamine becomes slightly green indicating that Cu complexes are formed. If this is so, the complex must be a Cu-O complex as indicated below:



No color change takes place with N-methyl-morpholine, which can no longer form complex Cu compounds.



N-methyl-morpholine

There is undoubtedly some oxidation of triethanolamine as well. If badly oxidized Cu strips are used, finely divided Cu collects at the bottom of the beaker after some time.

Monoethanolamine is more reactive as evidenced by the observation that, even at room temperature and normal humidity, the amine will react with Cu surfaces forming a deep blue Amino-complex, easily soluble in water. If the amine is rinsed off with water, a bright copper surface remains. Triethanolamine reacts very slowly at room temperature. Thus monoethanolamine is a corrosive agent while triethanolamine is far less corrosive.

Triethanolamine was evaluated as a solder flux for printed circuit boards on a production scale. The liquid was applied with a spray gun to printed circuit panels and soldered on a wave solder machine. It was found that the amine will spread over the panels better if about 10 percent monoethanolamine is added. The spreading can be improved further if about 1 percent polyoxyethylene-sorbitan monooleate in a small amount of water is added.

Since the commercial grade of triethanolamine contains about 10 percent of the mono- and diethanolamine, the 90 percent triethanolamine, 10 percent monoethanolamine mixture was adopted for further experiments.

Triethanolamine C-14 labeled flux

Two stock solutions were prepared, each containing 10 percent monoethanolamine[6] and 90 percent triethanolamine,[6] labeled respectively with monoethanolamine C-14 and triethanolamine C-14. The labeled free amines were prepared from the corresponding HCl salts by vacuum distillation from alkaline solutions.

For the initial experiments the small circuit boards previously described were used. The radioactive flux was painted on. The boards were dip-soldered and cleaned in water. The amount of flux originally applied, the amount left on after soldering, and the amount left on after cleaning was determined by extracting the panels with methanol followed by assay of an aliquot of the extract. The activity of the panels remaining after extraction was determined by direct assay of the panel.[7]

For the purpose of this discussion it will be sufficient to summarize the results of these initial experiments very briefly.

Nearly all of the monoethanolamine in the flux evaporates during the soldering operation. Samples soldered using monoethanolamine labeled flux retain only 4 percent of the original activity after soldering, and before cleaning. Triethanolamine does not evaporate to any extent. After soldering about 90 percent of the original amount is still left on the panel. Because monoethanolamine left in contact with copper will cause corrosion, the evaporation of the monoamine is fortuitous.

The bulk of the flux on the panels after soldering can be removed in a matter of seconds. Panels, soldered with triethanolamine-labeled flux and cleaned in a water spray for 10 seconds, retain about 10 percent of the original activity. It takes progressively longer to remove more of the flux. For example, if it is desired to remove more than 99 percent of the original activity, panels must be immersed in running water for about half an hour.

Since long cleaning times are impractical for commercial application, an attempt was made to determine how much flux can be left on the board, and the time necessary to achieve this desired degree of cleanliness. The insulation resistance of cleaned panels was chosen as a criterion.

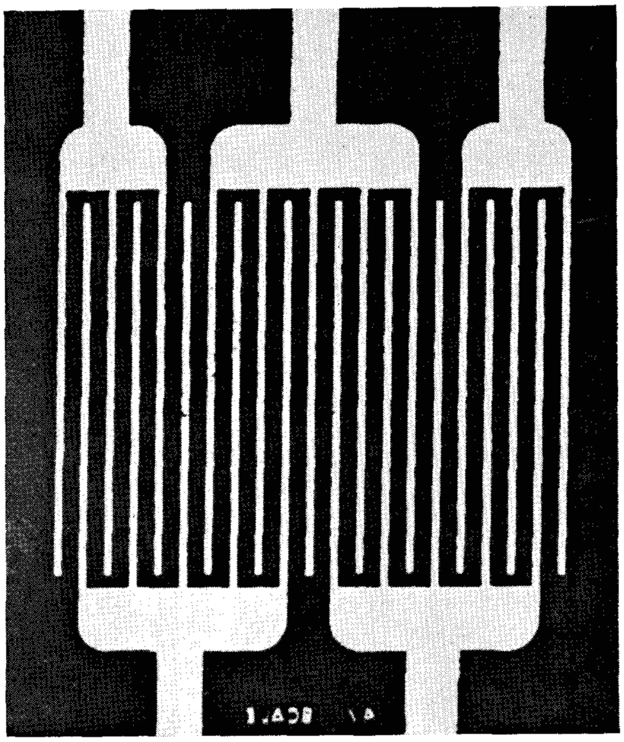
Insulation resistance and amount of flux on cleaned panels

At room temperature triethanolamine is a viscous hygroscopic liquid with a specific resistance larger than the specific resistance of distilled water. Thus pure, dry triethanolamine is not ionized. However, the flux residue left on soldered panels after cleaning in water is an ethanolammonium solution, either in the hydroxide or salt form, sufficiently ionized to show appreciable conductivity. As the amount of flux on the panels decreases, the insulation resistance should increase.

Insulation resistance measurements should be made at high humidity levels because the board material must contain enough water to form the ethanolammonium solution. The resistance of the board material itself decreases sharply as the amount of water absorbed by it increases. It was found that the amount of flux left on panels after cleaning in water for two or five minutes is so small that the insulation resistance of these panels is the same as the insulation resistance of the board material measured at the same level of humidity, regardless of the level of humidity.

The panels adopted for insulation resistance measurements have 21 parallel equidistant conductors. The odd- and even-numbered conductors are connected. Ignoring end effects, the patterns can be regarded as 20 parallel resistances, each resistance formed by an area approximately 1-1/4″ long and 1/32″ wide. Fig. 3 is a photograph of a test panel.

Figure 3 **Typical panel prepared for insulation resistance measurements.**



The flux used for these experiments was the 90 percent triethanolamine, 10 percent monoethanolamine mixture containing radioactive triethanolamine as described earlier. Panels were dip-soldered and cleaned in running water for two to five minutes. After superficial drying the panels were conditioned at about 90 percent relative humidity for approximately 100 hours. Then the resistance of each panel was measured using a General Radio Megohmmeter. Next the amount of flux on each panel was determined using the extraction technique previously described.

Included in this test series was a group of panels which were not fluxed or soldered, to determine the resistance of the board material, and another group of panels soldered with commercial activated flux and cleaned using the customary procedure. All panels were conditioned together, all resistances were measured in the same way.

The results are collected in Table 1. The table lists: (1) the insulation resistance of control panels; (2) the insulation resistance of panels soldered with triethanolamine flux and subsequently rinsed in running water for 2 to 5 minutes. (Average amount applied to panels is 80 mg.) (3) the corresponding amounts of flux, determined—as before—by extracting the panel with methanol in a Soxhlet apparatus and assaying an aliquot of

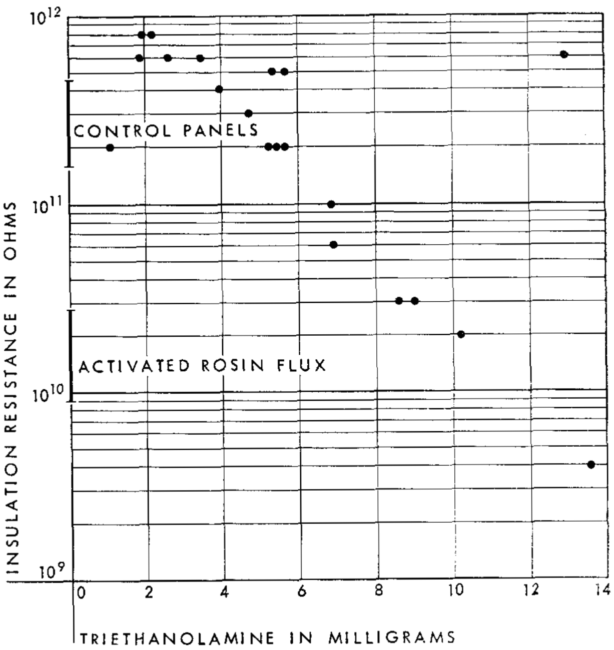


Figure 4 **Resistance in ohms against amounts of triethanolamine remaining after cleaning.**

Table 1 **Resistances in ohms of test panels conditioned at 90 percent relative humidity.**

Control Panels	*Soldered with Triethanolamine*			*Soldered with Activated Rosin Flux*
Resistance	*Resistance*	*mg Flux*	*Correction*	*Resistance*
1×10^{11}	3×10^{11}	4.7	0.05	1×10^{10}
1×10^{11}	2×10^{11}	5.6	0.08	1×10^{10}
3×10^{11}	4×10^{11}	3.9	0.11	1×10^{9}
4×10^{11}	8×10^{11}	1.9	0.03	1×10^{9}
4×10^{11}	6×10^{11}	3.4	0.04	4×10^{10}
5×10^{11}	6×10^{11}	2.5	0.02	1×10^{10}
1×10^{11}	1×10^{11}	6.8	0.05	1×10^{10}
3×10^{11}	5×10^{11}	5.6	0.27	7×10^{9}
4×10^{11}	5×10^{11}	5.3	0.17	4×10^{9}
4×10^{11}	3×10^{10}	9.0	0.10	2×10^{10}
4×10^{11}	6×10^{11}	12.9	0.11	3×10^{9}
5×10^{11}	3×10^{10}	8.6	0.04	3×10^{10}
5×10^{11}	6×10^{10}	6.9	0.13	4×10^{9}
4×10^{11}	2×10^{11}	5.4	0.06	1×10^{10}
4×10^{11}	8×10^{11}	2.1	0.01	5×10^{10}
2×10^{11}	2×10^{11}	5.4	0.11	1×10^{10}
2×10^{11}	2×10^{11}	1.0	0.05	4×10^{10}
3×10^{10}	2×10^{10}	10.2	0.27	1×10^{10}
	6×10^{11}	1.8	0.02	5×10^{9}
	4×10^{9}	13.6	0.53	1×10^{9}
Av 3.1×10^{11}*	Av 3.4×10^{11}*			Av 1.4×10^{10}
s. d 1.5×10^{11}	s. d 2.7×10^{11}			s. d 1.4×10^{10}

*A t-test indicated no systematic difference between the two averages.

the extract. As a check on the extraction, a part of the panel was counted. The amounts found on the extracted panel are listed under Correction; (4) the insulation resistance of panels soldered with activated rosin flux and cleaned in chloroethane under ultrasonic agitation.

In Fig. 4 the observed insulation resistance of panels soldered with triethanolamine and cleaned in water is plotted against the amount of flux on that panel. The correlation is evident. For insufficiently cleaned boards the resistance of the relatively large amount of flux solution is much smaller than the resistance of the panel material. As the amount of flux on the panel decreases, the resistance of the flux solution increases to the point where the resistance of the residue exceeds the resistance of the board material. Panels so cleaned may contain up to about 2 mg of triethanolamine. This amount of flux can be reduced to about 0.1 mg but the insulation resistance will not increase substantially. To substantiate this, 20 panels were soldered and cleaned in water. Ten of these panels were subsequently extracted with methanol. The average resistance of the two groups of 10 panels is the same.

The average resistance of groups of 20 panels is plotted in Fig. 5 as a function of the relative humidity at which the panels were conditioned. Note that the insulation resistance of soldered and cleaned panels is very nearly the same as the resistance of unused panel at the same level of relative humidity.

The resistance of panels after soldering, but before cleaning, is of the order of 10^6 ohms. The resistance of cleaned panels is of the order of 10^{11} ohms. This large range suggests the use of resistance measurements to estimate the "cleanness" of the panels.

Finally, referring again to Table 1 and Fig. 3, triethanolamine may be compared with activated rosin flux. As stated above, the rosin flux soldered samples were cleaned as is customary in production, using organic solvents and ultrasonic agitation. The method is far more cumbersome, necessitating equipment to reclaim the solvent and large ultrasonic transducers. However, the average resistance of panels is still at least ten times lower than the resistance of the board material. As previously stated, metal halides formed during the soldering operation are not removed. The electrolytic conductors of these salts at high levels of humidity result in low insulation resistance.

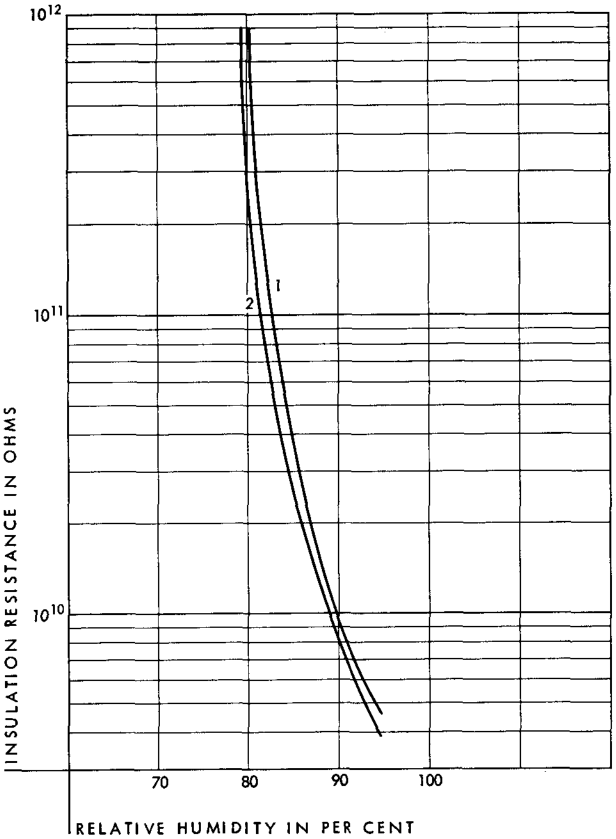


Figure 5 **Average insulation resistance of test panels as a function of relative humidity.** *Curve (1) represents the control panels, curve (2) the soldered and rinsed panels.*

References and footnotes

1. There is little information on solder flux available in the literature. The reader is referred to a *Symposium on Solder* published by the ASTM, 1956.
2. Br-82 costs $0.50 per mC, Cl-36 $500.00 per mC.
3. The technique and the apparatus needed for it are well known; it affords complete control of the invisibly small amounts of material.
4. About one-third or one-half of the amount applied, depending on the amount of flux on the panel, the number of solder points, the duration of soldering, etc.
5. A "liquid scintillator" is a solution which scintillates under nuclear irradiation. While solid scintillators have been known for about half a century, scintillating solutions were discovered only recently. The method, its advantages, limitations, and the instrumentation needed are described in: *Liquid Scintillation Counting,* Carlos J. Bell and F. Newton Hayes, Pergamon Press 1958.
6. U.S.P. grade.
7. If a panel is counted directly, some uncertainty is introduced in the counting efficiency of the liquid scintillator solution. This uncertainty does not exist if the radioactivity is introduced in the form of a methanol solution. It was found that methanol extraction removes 99 percent of the activity on the panel. Thus the uncertain counting efficiency affects only about 1 percent of the activity to be assayed which makes the total effect negligible. Counting the extracted panel forms an additional check on the extraction.

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