

**NPL REPORT
DEPC MPR 032**

**The Role of Permeability and
Ion Transport in Conformal
Coating Protection**

A MENSAH and C HUNT

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National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

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authorised by Director, Engineering and Process Control Division

The Role of Permeability and Ion Transport in Conformal Coating Protection

A Mensah and C Hunt
Engineering and Process Control Division

ABSTRACT

The level of protection offered by a range of conformal coatings on electronic assemblies has been evaluated. The role of permeability and ion transport is the primary interest. Testing was carried out on 6 coatings of the main generic types currently used by industry either conformally coated onto FR-4 laminate boards or, as free films. The methods utilised were surface insulation resistance (SIR), sequential electrochemical reduction analysis (SERA), PermeGear diffusion cells and gas chromatography mass spectrometry (GC-MS). Conformally coated boards were used for the SIR and SERA measurements while free films of the selected coatings were used for the diffusion and GC-MS measurements.

Each method revealed aspects of the level of protection offered by the coatings as well as the extent to which the coatings are permeable to contaminants in high environmental stress regimes. The coatings acted as an effective barrier to NaCl penetration but were permeable to dibasic acids found in electronic fluxes. The importance of board cleanliness is also highlighted by the results obtained from these investigations.

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1 INTRODUCTION

Increasingly, more safety critical systems are being controlled by electronics. Furthermore, these systems are being mounted close to the functional part. This inevitably requires electronics to operate in high environmental stress regimes. To try and achieve reliable electronics in such situations, protective coatings can be applied to electronic circuits and are commonly known as conformal coatings. Conformal coatings are applied to printed circuit boards in thin layers by dipping or spraying, and may cover all or part of the electronic assembly. They are able to achieve a high degree of protection even in environments that would normally be considered fatal for electronic equipment¹. However, there is very little information currently available to engineers to accurately predict the performance over the lifetime of the product. It is not currently known what levels of protection are offered to specific contaminants, and what transport rates and degradation will be observed. For example it has now been shown that they let moisture permeate^{2, 3}, contradicting the previously accepted understanding.

This report aims to identify the potential protection mechanisms in conformally coated electronic assemblies and to assess the level of protection offered by a range of conformal coating materials. The major criteria for success are not just based on the generic chemical base of the coating, as there are good and bad in all categories¹.

The conformal coatings and contaminants investigated in this project were selected following industrial consultation. The six selected coatings consisted of two water-based acrylics, a solvent-based acrylic, a silicone, an epoxy, and a polyurethane/alkyd. The boards were coated by the coating suppliers in accordance with their recommended standard procedures.

The rate of degradation or permeability of these coatings was investigated using surface insulation resistance (SIR), sequential electrochemical reduction analysis (SERA), PermeGear diffusion cells and gas chromatography mass spectrometry. Salt, in the form of sodium chloride, wave soldering flux and solder paste were the contaminants used in these investigations.

2 EXPERIMENTAL

2.1 BOARD

The test coupons were 106.5 x 102.6 mm boards manufactured from epoxy FR-4 laminate. The design of the test pattern (NPL TB67) is shown in Figure 1. The test pattern comprises of four combs of dimensions 25 mm x 25 mm with minimum track and gap 152 μm and 172 μm respectively. The boards, which were 1.6 mm thick, had an electroless NiAu finish.

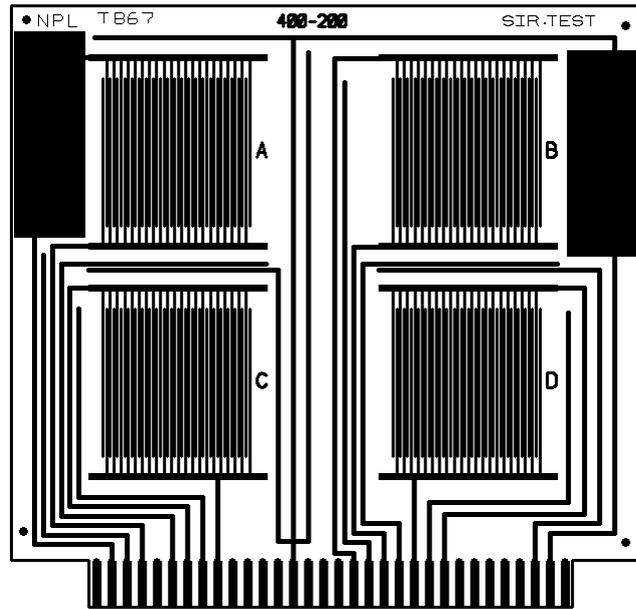


Figure 1. Layout of test board

2.2 METHODS

2.2.1 Surface Insulation Resistance (SIR)

The Surface Insulation Resistance (SIR) technique has been widely used to assess the effect of contaminants on the reliability of assemblies. It is a measurement of the resistance across two interdigitated combs while subjecting the test vehicle to a hot/humid environment⁴.

A Concoat AutoSIR (Mk II) that has a current sensitivity of 1×10^{-11} A was used to monitor the SIR values on 16 channels at 10-minute intervals during the test period of 72 hours. A +50 V DC biased voltage was continuously applied across the comb patterns during the test period and a $10^6 \Omega$ limiting resistor was included in each measurement channel.

The edge connector was masked on both sides during coating of the boards to ensure that the SIR measurements were a true reflection of the resistance of the test pattern and not contaminated by the edge connector. The measured SIR values discussed later represent the average value for the four combs contained on each board.

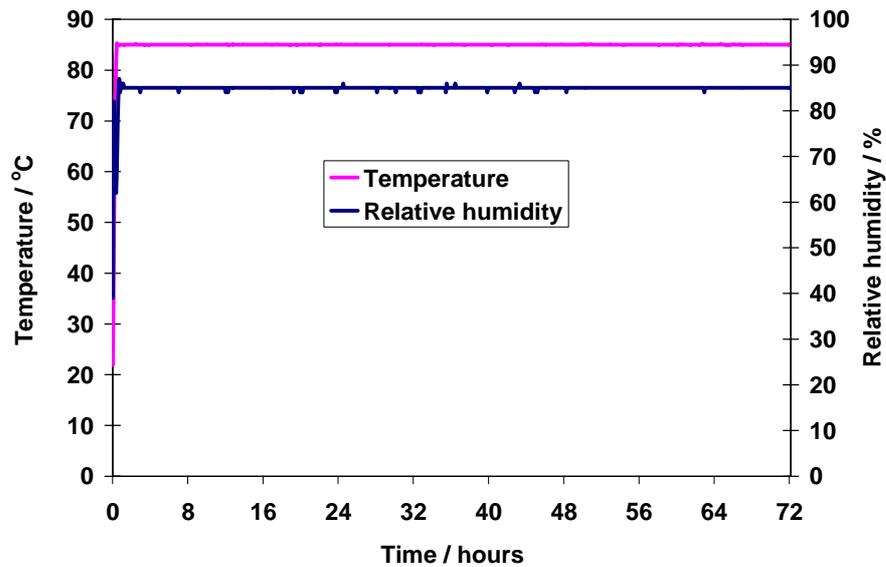


Figure 2. Typical temperature / humidity profile during SIR testing at 85 °C and 85% RH

Exposure tests were carried out with the some of the boards contaminated prior to coating and also some after coating. The boards were contaminated with 50 μ L of NaCl solution of varying concentration per each comb pattern and left to air dry for up to an hour before the boards were sent off for coating.

The temperature and humidity conditions within the environmental chamber (Sanyo Format 550 Programme⁵) were monitored by wet and dry platinum resistance thermometers and a typical measured profile for humidity and temperature during a test is shown in Figure 2. The SIR environmental conditions were initially selected to match the maximum temperature of the diffusion cell experiments (40 °C). However, the resistance values tended to be very high and preliminary measurements at 40 °C and 90% RH, when conformally coated boards where contaminated after coating, showed very little effect of the contaminants on the SIR values. Increasing the test conditions to 85°C and 85% RH showed a reduced resistance (Figure 3) and greater differentiation of the effect of contaminants. These test parameters are also much closer to most in-service conditions, and were the test conditions adopted.

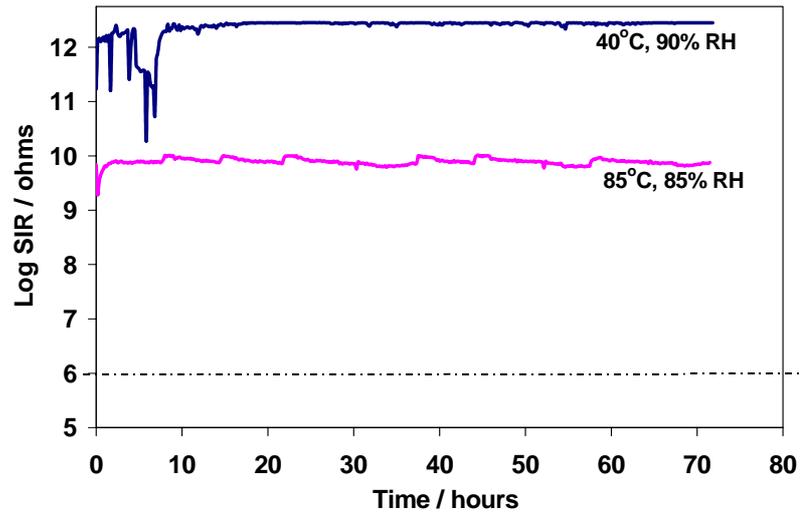


Figure 3. SIR plot showing the effect of environmental conditions on the resistance of a conformally coated board

2.2.2 Sequential Electrochemical Reduction Analysis (SERA)

SERA is an electrochemical technique used to determine a variety of key coating parameters. It involves isolating a small well-defined area with a gasket, which is then filled with an electrolyte. A controlled current is then applied between the tested surface and an auxiliary electrode to reduce or oxidise the surface species⁶. The electrode potential of the tested surface is measured as a function of time yielding a series of plateaus corresponding to the sequential reduction or oxidation of surface compounds⁷. The associated potentials identify the types of species present, and the decay characteristics with time at each level give a measure of the amount present. If the total amount is required the current should be allowed to fall to zero. All SERA measurements were performed using the SurfaceScan QC-100™ shown in Figure 4.

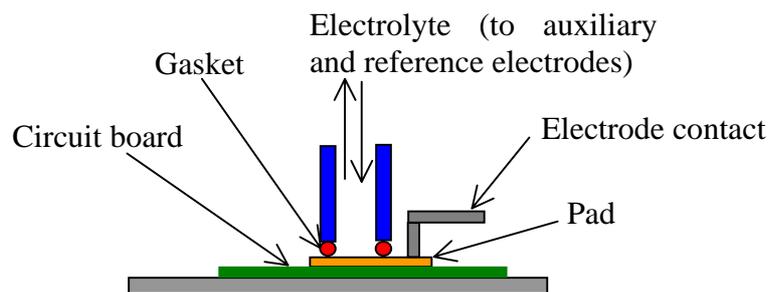


Figure 4. Diagram of SurfaceScan QC-100™

Prior to SERA testing, the coatings were stripped off the boards after 72 hours exposure at 85°C and 85% RH with isopropyl alcohol (IPA), acetone or dichloromethane. A solution made up of 10g/l potassium thiocyanate and 2.5g/l potassium hydroxide was used for the analysis. This electrolyte was deaerated prior to the tests by purging it with ultra high purity nitrogen for ten minutes, sufficient for the very small volumes.

The reduction potentials were measured versus a Ag/AgCl reference electrode using a pad area of 0.02cm^2 and a $30\ \mu\text{Acm}^{-2}$ applied current at room temperature.

2.2.3 Diffusion cell

To measure the diffusivity of contaminants, such as NaCl, through a coating, PermeGear horizontal diffusion cells (Figure 5) were used with 15 mm orifice diameters and the test membrane (coating) fixed between. When an excess amount of the diffusant of interest is added to one compartment the time evolution of its concentration in the other compartment reflects the diffusion of species through the membrane/coating. In this study, diffusion of chloride ions and of flux was evaluated in separate tests. In both cases, $5\ \text{mol dm}^{-3}$ NaNO₃ was used as a supporting electrolyte.

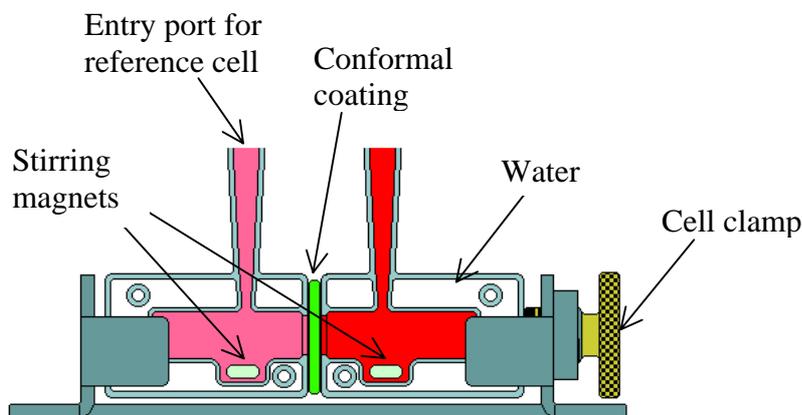


Figure 5. PermeGear Horizontal Cell

All of the glassware was cleaned with acetone and DI water until a conductivity reading of less than 4 mS was obtained from the washings. This indicates that there were no ions present that would interfere with the experiment. Free films of the selected coatings were cast for the diffusion cell experiments to determine both chloride ion and flux migration.

Due to temperature limitations on the effective use of the combined ion selective electrode used for taking measurements in the diffusion cell experiments, all measurements were carried out at $40^\circ\text{C} \pm 1$, with the temperature controlled by water recirculation.

Casting films

Preparation of the films to act as the membrane in the diffusion cells was undertaken in the following way.

The PTFE and polythene sheets were taped onto the back of a metal panel using double sided tape. The polymer sheets were then cleaned by wiping with acetone and water several times before finishing with an acetone wipe and then allowed to dry in ambient conditions before casting films. Films were prepared using various thickness of tape, to give the desired dry film thickness, and the coatings were drawn down using a doctor blade, two coats were applied. Samples of the coatings were prepared according to

individual suppliers instructions, where possible, to give a dry film thickness of ~50 μm . After 24 hours, the films were removed from the backing PTFE or polythene sheets and conditioned for 1 week at 23°C and 50% RH.

Test procedure for chloride ion diffusion

The sodium chloride and sodium nitrate were dried in an airflow oven at 110°C for at least 2 hours to ensure that any water was removed and then cooled in a desiccator for 1 hour. Fresh DI water was used for all solutions. A stock solution of each of the salts was made of 1L of NaCl (0.42 mol dm^{-3}) and 250 ml of NaNO₃ (5 mol dm^{-3}). Then using the exact values for the molarity, the amounts of NaCl solution needed to make up solutions of 10, 50, 100, 250, 500, 750, 1000 and 5000 ppm of Cl⁻ ion were calculated. Once all solutions had been made they were left overnight in a constant temperature room (CTR) at ~25°C. Ion-selective electrodes were used to monitor the chloride level. The electrode was connected to the meter and also left in the CTR. The water bath, pump, cells and stirrers were set up and the bath filled with deionised water. The bath was set to a temperature of 43°C, which maintained a temperature of 40.3°C inside the cells when they were filled with water.

Each electrode was calibrated at 25°C and 43°C using a selection of the Cl⁻ ion solutions and applying the Nernst equation, a calibration graph for the electrodes expected response at 40°C was derived. It is important to note that the electrode needs to be calibrated just prior to use. This should be done, at the very minimum, with two concentrations of chloride spanning the test levels of interest.

A portion of the free film was then clamped between the two halves of the diffusion cell and the remainder was used for film thickness measurement using a Minitest 4100. Three drops of NaNO₃ (5 mol dm^{-3}) solution were added to either side of the cell and on the right hand side the magnetic stirrer was added. Two solutions of different chloride ion concentrations were added, 100 ppm Cl⁻ ion in the right compartment and 15000 ppm in the left compartment. This was done simultaneously to avoid any undue strain on the thin film. Both sides were then stoppered; the PTFE stoppers for the cells were wrapped in ParaFilm to enable a good airtight seal. When the cell was secure, it was placed on the stirrer so that the magnetic stirrer was not touching the film. Once the apparatus was set up the electrode was connected and a measurement taken. Cl⁻ ion concentration measurements were taken at regular intervals over 400 hours in the right hand side of the cell that contained an initial solution of 100 ppm Cl⁻.

Hydrogen Ion Diffusion

The glassware and apparatus was prepared as above with the addition of neoprene rubber gaskets to hold the free films in place between the two halves of the cell. The temperature of the cells during the experiment was 40°C and the average difference in the cell temperature during operation was 0.6°C.

A solution of water-based flux B (Table 1) was added to the left hand cell while deionised water was added to the right hand side. To both solutions, five drops of NaNO₃ (5 mol dm^{-3}) solution were added and to the right hand side, the magnetic stirrer

was added. pH readings of both halves of the cell were taken at regular intervals over a 50-hour period.

2.2.4 Gas Chromatography Mass Spectrometry (GC-MS)

Samples of the individual acids used in fluxes were dissolved in water, and then 1- 6 μL injected into the GC (Hewlett Packard 5890 with a Hewlett Packard MSD 5970 as detector) and the chromatograms recorded. The injected volume was varied depending on the peak strength. A sample of flux was then injected and the peaks of the resulting chromatogram recorded. A sample was then taken from the initially uncontaminated side of one of the diffusion cell experiments after 50 hours and was also injected and the resulting peaks identified. Between each sample, a blank injection containing deionised water was performed to ensure minimal chance of crossover between samples. It must be noted that there was a minor air leak apparent in the MSD and this does affect the baseline in some runs and the ions at elapsed times of 32 and 28 (O_2 and N_2 respectively) have been removed from the representation of the chromatograms.

Mass spectra of the peaks identified in the chromatogram of the initially uncontaminated side of the cell were recorded and compared with the library records of the individual acids present in the flux.

3 RESULTS

3.1 SIR RESULTS WITH NaCl CONTAMINATION

The effect of NaCl contamination on boards prior to coating can be seen in Figure 6 for the solvent based acrylic coating, where SIR values of boards that have not been conformally coated are compared to boards contaminated either before or after conformal coating application. From this figure, it is evident that boards that were contaminated after conformal coating have higher log SIR values than boards that are either contaminated before coating or not coated at all. A similar behaviour to addition of contamination was seen with the other coatings.

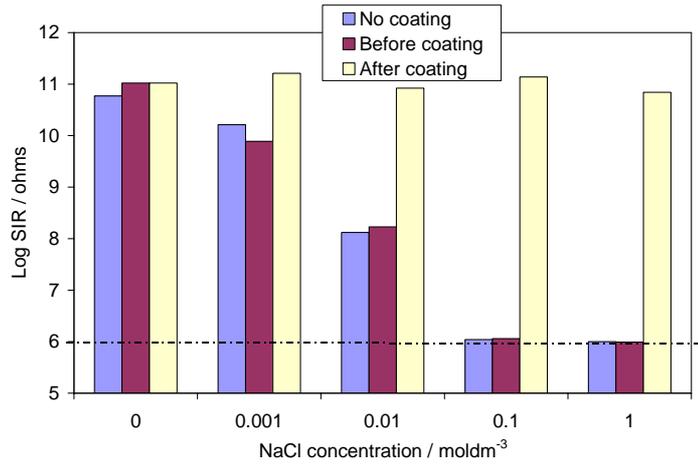


Figure 6. Final SIR values of boards with NaCl contamination applied before or after acrylic coating

Figures 7 to 12 show SIR measurements taken at 85°C and 85% RH for the various coatings when the boards are contaminated with NaCl after coating. A bar chart, showing the summary of the results can be seen in Figure 13. It is evident from these figures that the various conformal coatings provide varying degrees of protection for the boards. It should however be noted that with the exception of the epoxy coating and the water-based acrylic (2) coating, the coatings provided similar levels of protection for the different NaCl concentrations. Also, a low log SIR value does not necessarily mean the coating was permeable to the NaCl contamination, as each coating will have a different level of moisture permeability, which will affect its SIR results under the set environmental conditions.

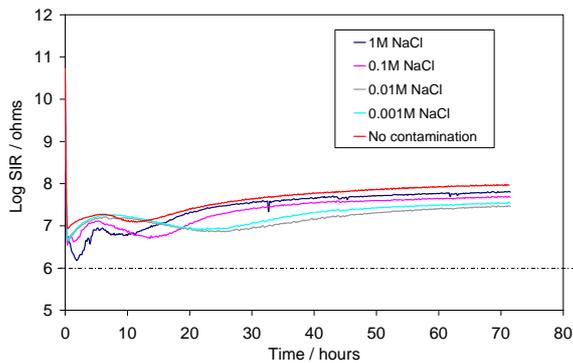


Figure 7. SIR plot of NaCl contamination on water-based acrylic (1) coated boards

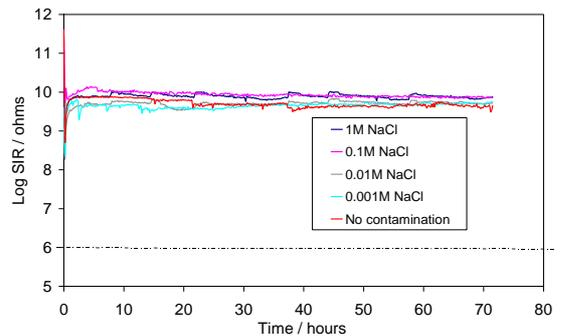


Figure 8. SIR plot of NaCl contamination on solvent-based acrylic coated boards

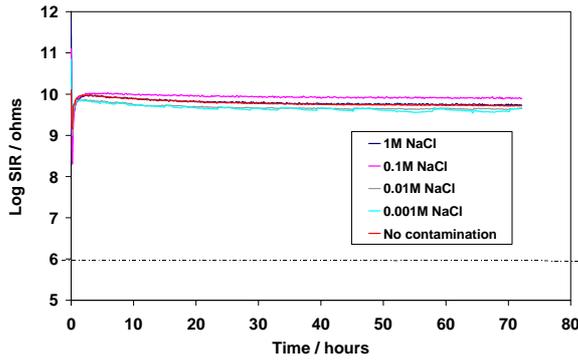


Figure 9. SIR plot of NaCl contamination on silicone coated boards

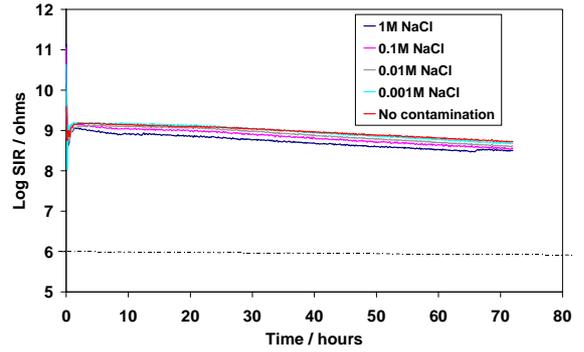


Figure 10. SIR plot of NaCl contamination on polyurethane / alkyd coated boards

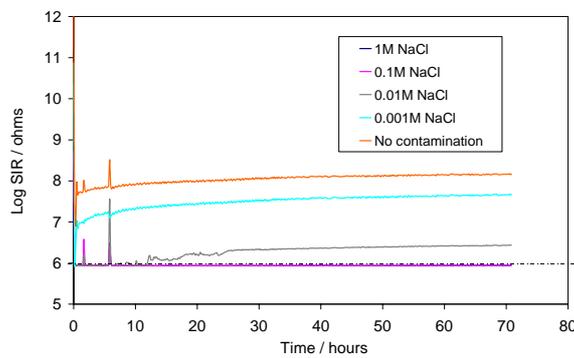


Figure 11. SIR plot of NaCl contamination on epoxy coated boards

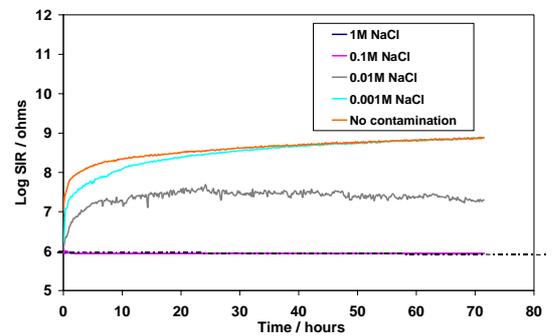


Figure 12. SIR plot of NaCl contamination on water-based acrylic (2) coated boards

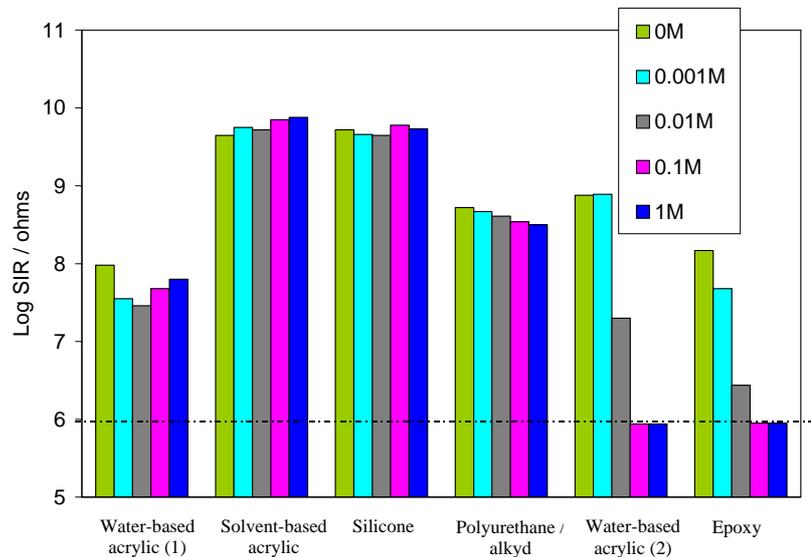


Figure 13. Final SIR values of conformally coated boards contaminated with NaCl after coating

3.2 SIR RESULTS WITH FLUX CONTAMINATION

Flux contamination of the boards after conformal coating application showed a more significant decrease in SIR compared to the results from NaCl contamination. Figures 14 to 20 show the SIR results from contaminating the boards with flux.

The flux formulations, typical of wave soldering fluxes, are shown in Table 1. Water-based flux A and B were used as contaminants in order to get some insight into the effect of the weak organic acids, present in fluxes, on conformal coatings.

Table 1. Flux formulations

Flux	IPA	DI water	Adipic	Succinic	Glutaric	Rosin	Surfactant	Co-solvent
Solvent-based	93.2	-	1.7	1.7	1.7	1.7	-	-
Water-based	-	90.4	1.7	1.7	1.7	-	0.1	5.0
Water-based A	-	90.5	1.7	1.7	1.7	-	-	5.0
Water-based B	-	95.5	1.7	1.7	1.7	-	-	-

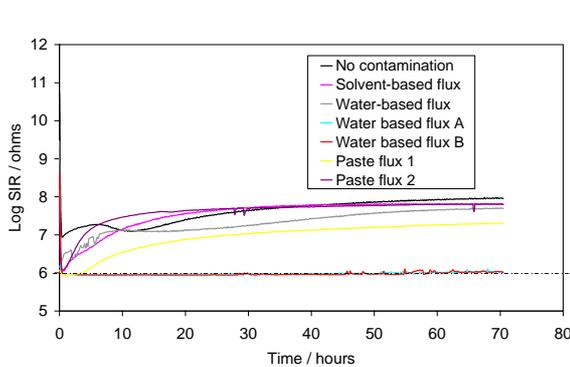


Figure 14. SIR plot of flux contamination on water-based acrylic (1) coated boards

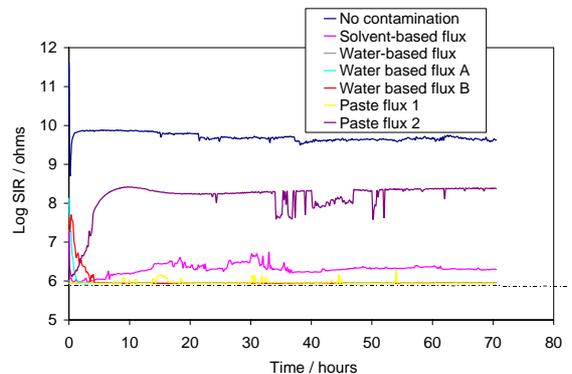


Figure 15. SIR plot of flux contamination on solvent-based acrylic coated boards

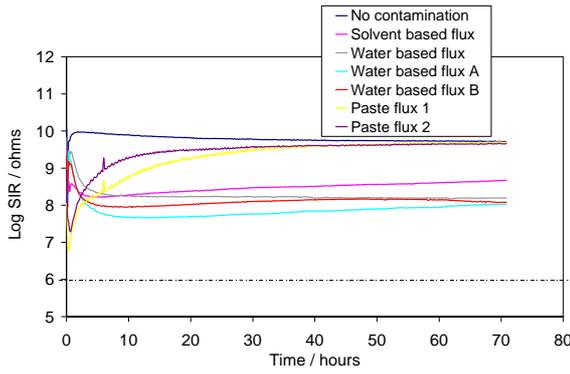


Figure 16. SIR plot of flux contamination on silicone coated boards

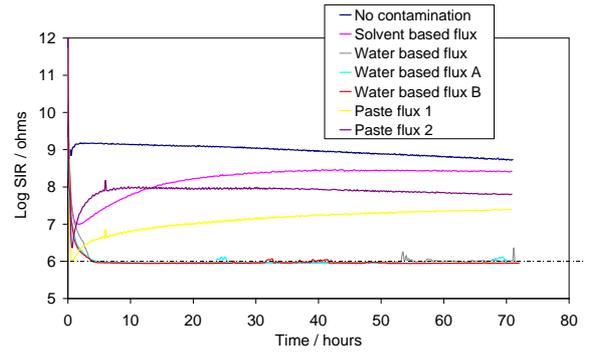


Figure 17. SIR plot of flux contamination on polyurethane / alkyd coated boards

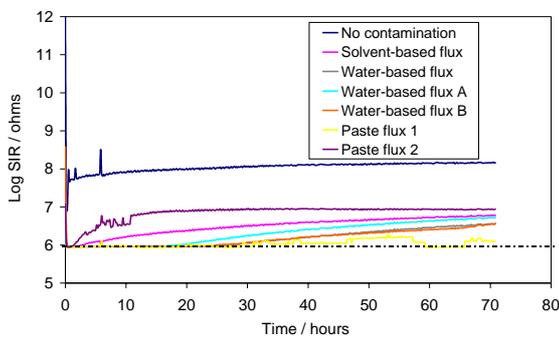


Figure 18. SIR plot of flux contamination on epoxy coated boards

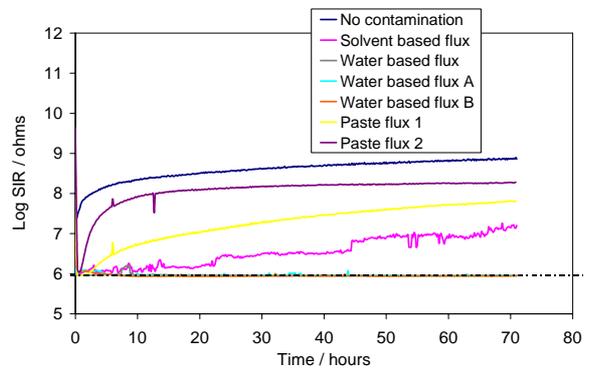


Figure 19. SIR plot of flux contamination on water-based acrylic 2 coated boards

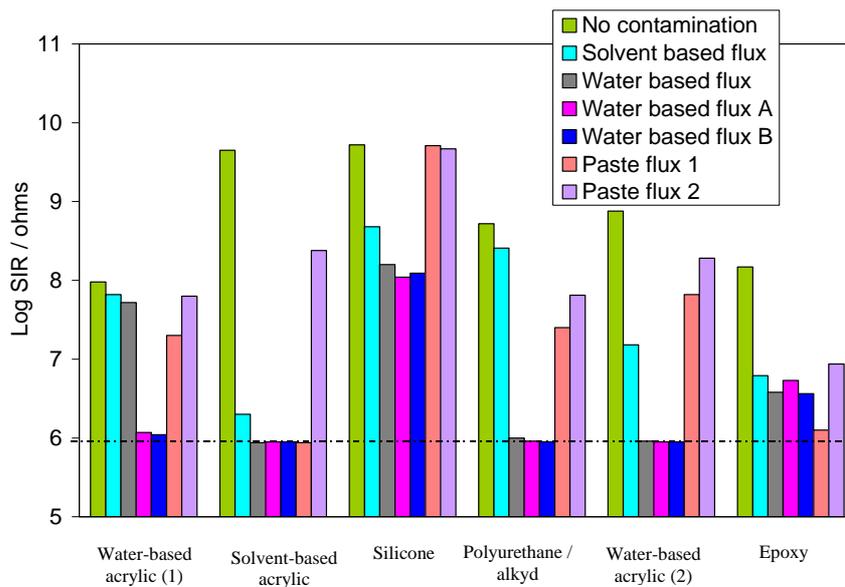


Figure 20. Final SIR values of conformally coated boards contaminated with flux after coating

3.3 SERA RESULTS WITH NaCl CONTAMINATION

A typical SERA curve as shown in Figure 21 illustrates the principle, showing plateaus associated with different reduction potentials for a copper-based system⁶. For each of the SERA curves shown in Figures 21 to 27, the final potential corresponds to the hydrogen evolution potential.

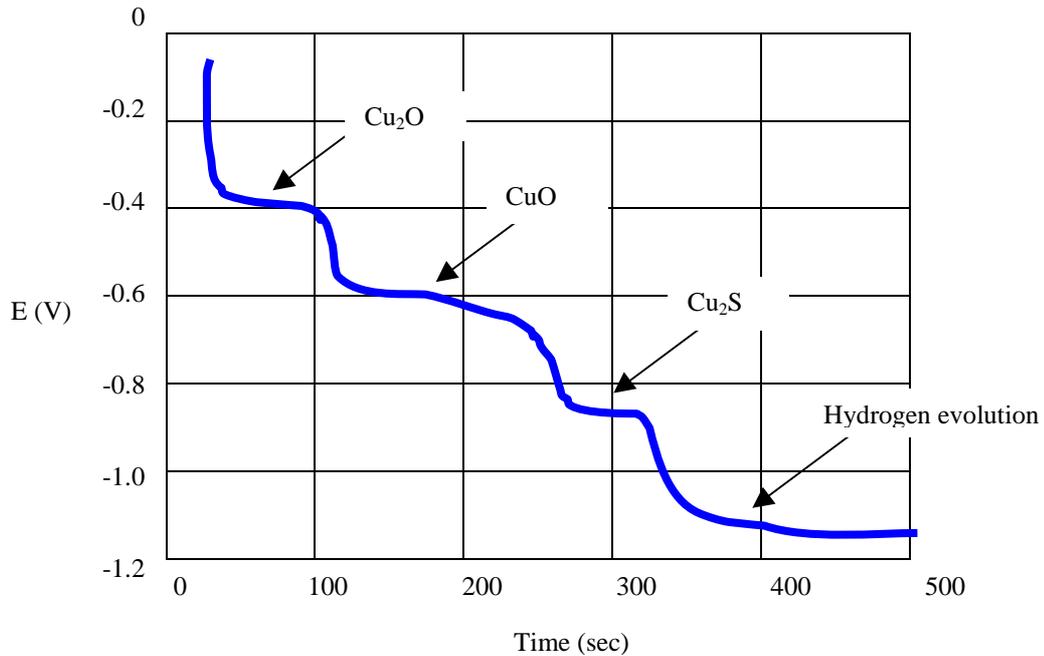


Figure 21. A copper surface shown with different oxidation states of copper, and copper sulphide

At ambient conditions, gold does not form any oxides when in contact with moisture and air; thus, upon application of the reduction current, gold does not undergo electrochemical reduction⁸, the potential attaining a value associated with reduction of water. Thus, for the boards that are sufficiently protected from contamination by conformal coatings the potential transient should be similar to that for gold. This is indeed observed for boards that were contaminated by NaCl after conformal coating application as can be seen in Figures 22 – 27. This means the coatings were sufficiently robust against the NaCl contamination.

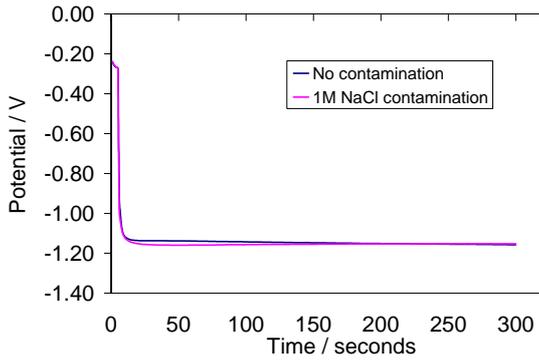


Figure 22. SERA curve of NaCl contamination on water-based acrylic (1) coated boards

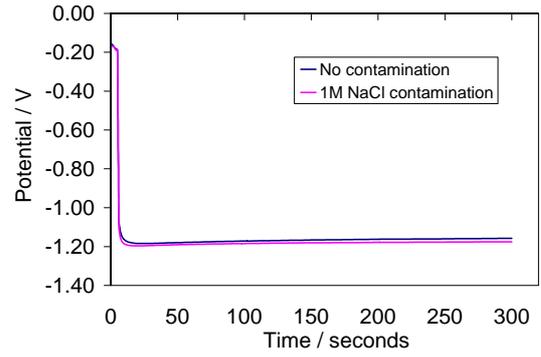


Figure 23. SERA curve of NaCl contamination on solvent-based acrylic coated boards

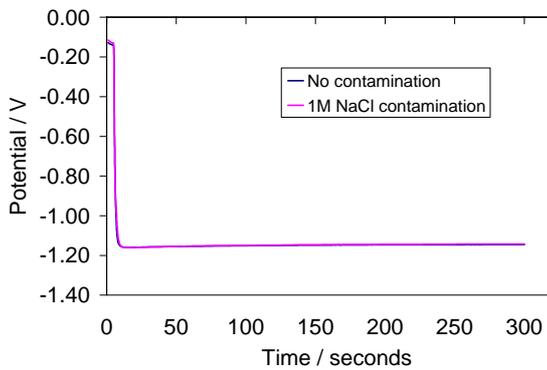


Figure 24. SERA curve of NaCl contamination on silicone coated boards

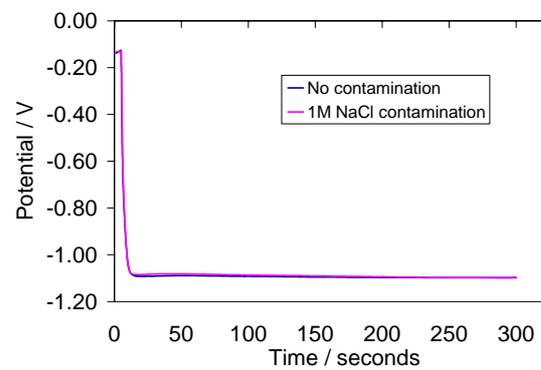


Figure 25. SERA curve of NaCl contamination on polyurethane / alkyd coated boards

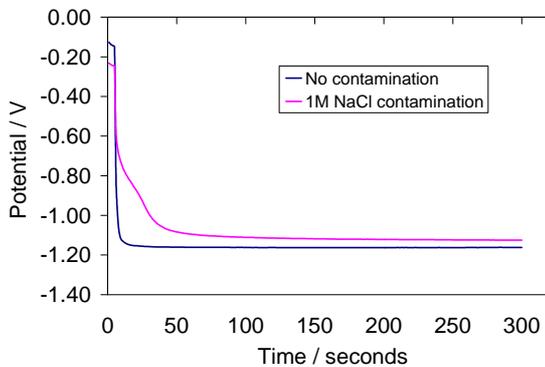


Figure 26. SERA curve of NaCl contamination on epoxy coated boards

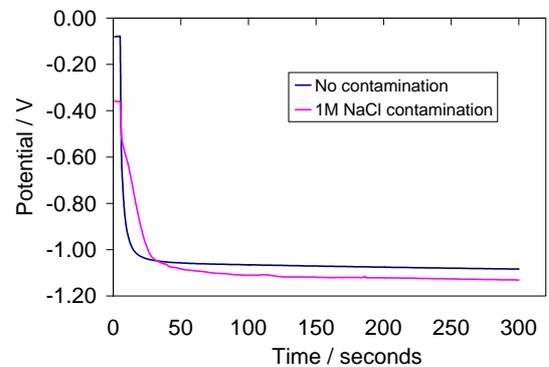


Figure 27. SERA curve of NaCl contamination on water-based acrylic (2) coated boards

3.4 SERA RESULTS WITH FLUX CONTAMINATION

A possible failure that might occur in electroless nickel/immersion gold finish boards is due to the oxidation of the nickel through the pores in the gold coating⁹. The presence of reducible species was evident on the boards where the conformal coatings were permeable to the applied contamination, the potential attaining values significantly more noble than that for gold.

The presence of reducible species in the presence of flux contamination is evident in Figures 28 to 33, which show the results from contaminating conformally coated boards with flux. This implies that the coatings were permeable to the fluxes. Table 2 gives the reduction potentials of some reducible species for copper though no data were available directly for nickel.

Table 2 Reduction potentials of surface species

Reducible compound	Reduction Potentials vs. Ag/AgCl electrode, V
Cu ₂ O	-0.45 to -0.60
CuO	-0.60 to -0.70
Cu ₂ S	-0.85 to -0.90
Hydrogen on copper	-1.05 to -1.15

* Measured at 30 μAcm⁻²

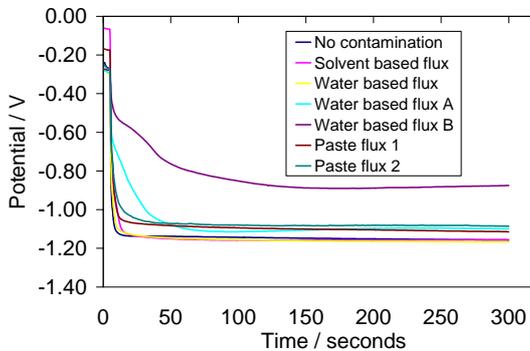


Figure 28. SERA curve of flux contamination on water-based acrylic (1) coated boards

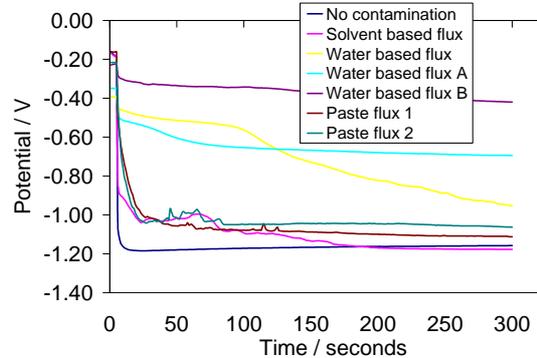


Figure 29. SERA curve of flux contamination on solvent-based acrylic coated boards

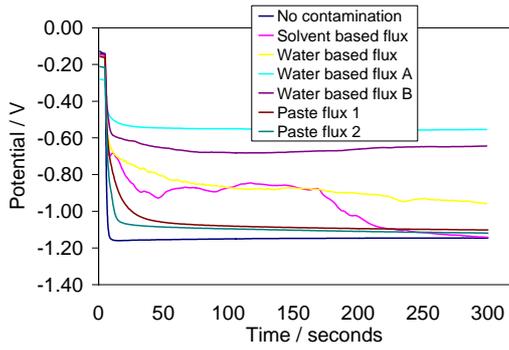


Figure 30. SERA curve of flux contamination on silicone coated boards

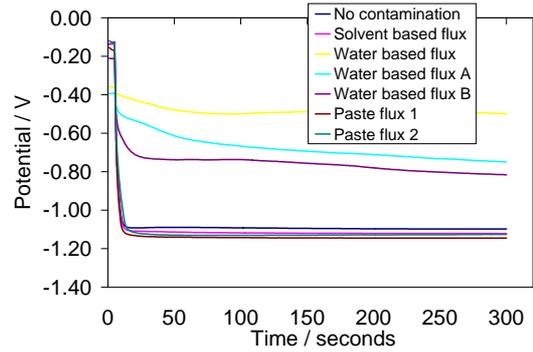


Figure 31. SERA curve of flux contamination on polyurethane / alkyd coated boards

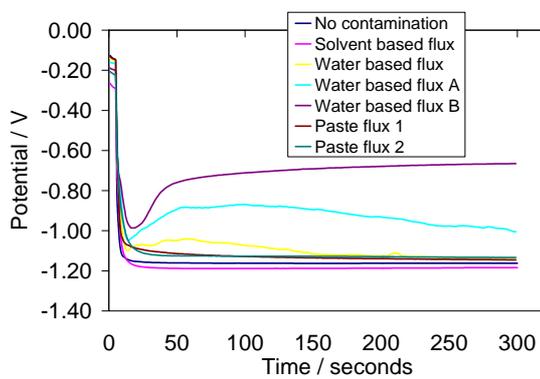


Figure 32. SERA curve of flux contamination on epoxy coated boards

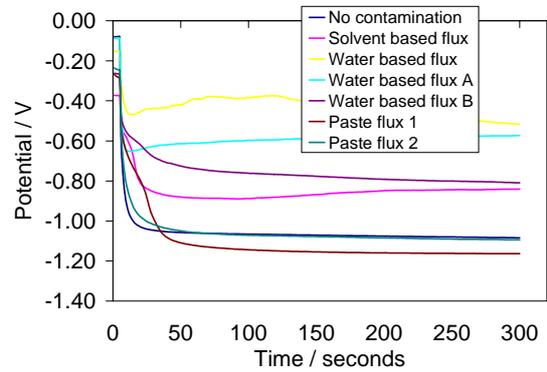


Figure 33. SERA curve of flux contamination on water-based acrylic (2) coated boards

3.5 RESULTS FROM CHLORIDE ION MIGRATION MEASUREMENTS

The results from measuring Cl^- ion migration across conformal coatings are shown in Figures 34 and 35 where the change in Cl^- ion concentration with time is monitored. These measurements were made at 40°C and confirm the results obtained from the SIR measurements at the same temperature, where very little effect of Cl^- ions on the coatings' performance was observed.

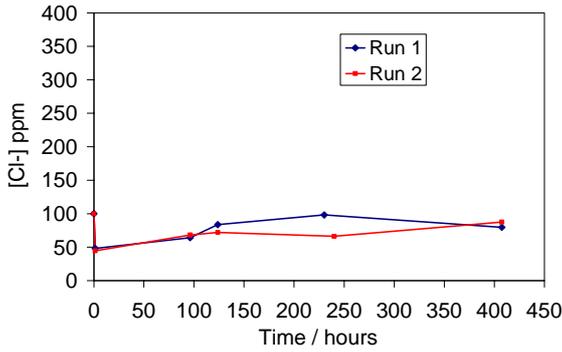


Figure 34. Chloride diffusion trends for water-based acrylic (1) coated boards

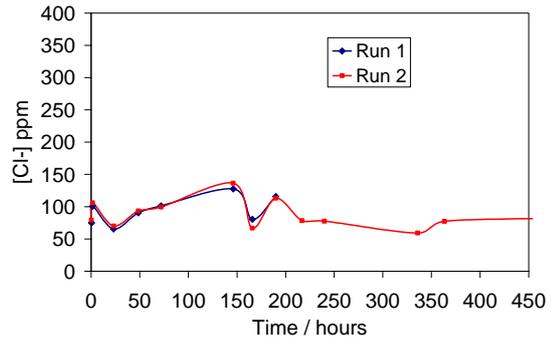


Figure 35. Chloride diffusion trends for solvent-based acrylic coated boards

3.6 RESULTS FROM FLUX MIGRATION MEASUREMENTS

Figures 36 to 41 show the pH measurements of both sides of the diffusion cell over the 50-hour test period. It should be noted that the left-hand side of the diffusion cell contained a sample of water-based flux B at about pH 2 while the right-hand side of the cell contained deionised water. The graphs below show the change in pH with time in both compartments.

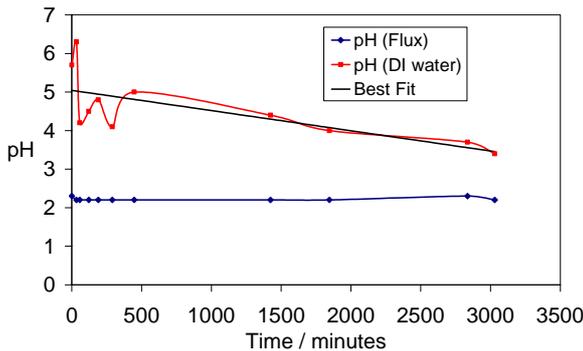


Figure 36. Variation in pH reflecting hydrogen ion diffusion trends for water-based acrylic (1) coated boards

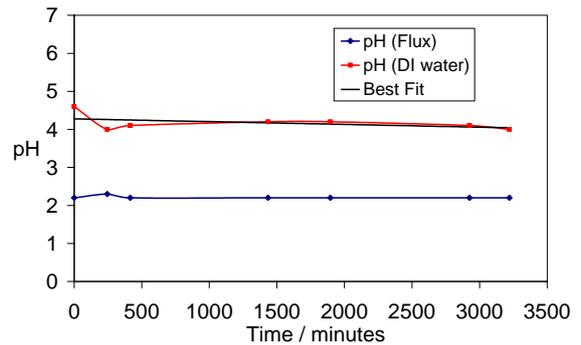


Figure 37. Variation in pH reflecting hydrogen ion diffusion trends for solvent-based acrylic coated boards

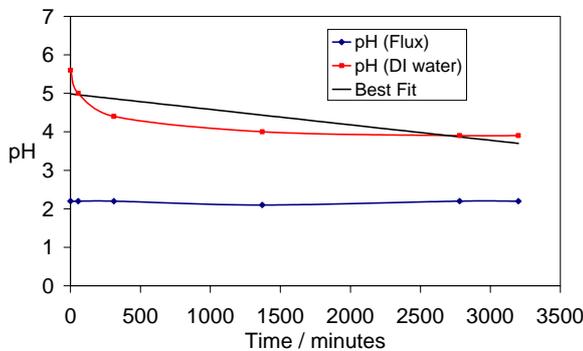


Figure 38. Variation in pH reflecting hydrogen ion diffusion trends for silicone coated boards

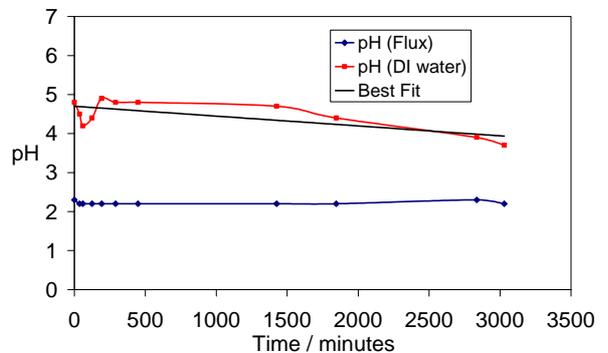


Figure 39. Variation in pH reflecting hydrogen ion diffusion trends for polyurethane / alkyd coated boards

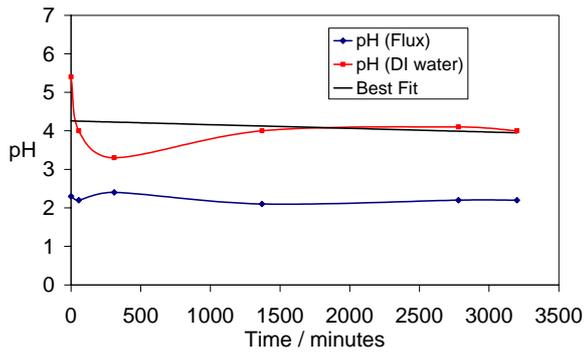


Figure 40. Variation in pH reflecting hydrogen ion diffusion trends for epoxy coated boards

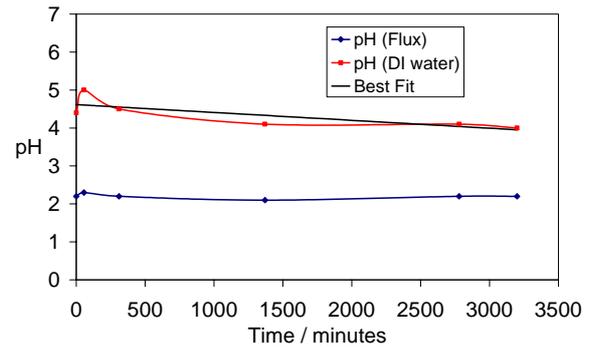


Figure 41. Variation in pH reflecting hydrogen ion diffusion trends for water-based acrylic (2) coated boards

The chromatograms of the acids present in the fluxes, used in these investigations, are shown in Figures A1 to A3. To identify whether these acids were migrating through the coatings, a chromatogram of water-based flux B (Figure A4) is compared to the chromatogram of the solution from the initially uncontaminated side of the diffusion cell at the end of the test period (Figure A5).

Comparing the mass spectra of the identified peaks in Figure A5 with the library records of the individual acids (Figures A6 – A13) showed evidence of permeation of at least two of the acids present in the sample taken from the initially uncontaminated side of the diffusion cell (succinic acid and either adipic and/or glutaric acids).

4 DISCUSSION

These experiments have characterised, with a range of techniques, the protection capabilities of conformal coatings when exposed to water salt and flux components. The techniques have been used in a complementary manner and have led to a better understanding of coating capabilities.

SIR generally declines with increasing temperature. At constant temperature there was no effect on SIR after exposing the coated board to sodium chloride (Figure 3). This suggests that the conformal coatings are permeable to moisture even though they remained very much less permeable to NaCl. Tautscher¹¹ and Waryold¹⁰ have observed that conformal coatings act as semi-permeable membranes allowing some moisture penetration to occur on exposure of boards to a damp atmosphere or immersed in water that reduces the SIR. Despite the decline in SIR, the coating will stop the circuit from water bridging following adventitious splashing or short term condensation and hence prevent signal cross talk or short circuits².

The preliminary SIR measurements carried out on boards that were contaminated before coating showed lower SIR as compared to boards that were contaminated after conformal coating application (Figure 6). This indicates the importance of board cleanliness in maintaining the performance of circuitry. Also, if Cl⁻ ions were to penetrate the coating, then a notable effect on the SIR would be observed. This is an

important point as Cl⁻ ions do have a strong effect on SIR but the conformal coating acts as an effective barrier.

The efficiency of different coatings in preserving high values of SIR during exposure to increasingly harsh environments depends both on the generic type of coating and the specific chemistry of the formulation. In general, the water-based acrylic coatings recorded lower SIR values than the solvent-based acrylic, silicone and polyurethane.

The SIR measurements were used to evaluate the application of flux residues and significant effects were observed, as shown in Figures 14 – 20. These fluxes were generic formulations from commonly used dibasic acids used in industry. Only the silicone coating appeared resistant to these acids, and in general the water-based variants of the fluxes were more aggressive than the solvent based version. The SIR results were very important, showing that these coatings can be relatively susceptible to these acids and directing further work to monitor closely these effects.

The surface analysis of the boards by SERA provided information about the presence of reducible species on the surface. Since gold is a precious metal and it is not oxidising, the surface analysis will show a SERA curve that drops rapidly to the water reduction potential. This makes it easier for SERA to detect the presence of reducible species on the board surface, which will be recorded as a series of plateaus corresponding to the sequential reduction of surface compounds. The SERA curves of the boards that were sufficiently protected from the contaminants by conformal coating show reduction potentials that rapidly dropped to the value corresponding to the hydrogen evolution reaction. The boards that recorded very low SIR values, 10⁶ ohms, show the presence of reducible compounds on the surface during SERA. These reducible compounds are the products of nickel and oxygen. There is also the possibility of some oxides of copper present where the nickel has been 'removed' by the flux to expose the underlying copper to oxidation. The SERA results are in agreement with, and complementary to, the SIR results as it shows the conformal coatings are more robust against the ionic contaminant than they are against flux.

A contaminated surface can cause a conformal coating to blister, as a result of underlying track corrosion, leading to loss of adhesion. This was evident in a number of the conformally coated boards that were contaminated with flux. The coatings appear to perform better against the solvent-based flux than they did against the water-based flux. Water-based flux B, which is made up of the weak organic acids (adipic, succinic and glutaric) show the lowest SIR values. This emphasizes the great influence these acids have on conformal coatings performance and is of primary concern as these organic acids are common in fluxes. Re-work is an issue of particular concern since it is quite possible for flux residues to be left following this operation. The permeability of the coatings to the fluxes has also raised questions about their permeability to other fluids that have similar organic compounds as the main constituents.

The hydrogen ion diffusion cell experiments were carried out to establish whether the conformal coatings are actually permeable to these fluxes as suggested by the SIR and SERA measurements. The results show that shortly after the test set-up, there is a drop in pH on the uncontaminated side of the cell. This is due to H₃O⁺ ions quickly migrating through the coating until equilibrium is reached. There is then further change in pH with time, which could be due to either the diffusion of the organic acid

molecules (and then dissociation) or the anions of the dissociated acids across the coating membrane. The GC-MS proved very sensitive to minute amounts of contaminants and the sample from the uncontaminated side of the cell was indeed shown to have small amounts of succinic acid and either adipic and/or glutaric acids present. A more comprehensive study would be required if quantitative analysis of migrating species is to be determined.

New work will be performed at NPL to evaluate the permeability of conformal coatings to organic compounds. This is of primary concern because a large number of fluids, such as hydraulic fluids, de-icing fluids and surfactants, come into contact with conformally coated boards in-service, as indicated in a survey of conformal coatings users. It will therefore be useful to find out whether the coatings are as permeable to these organic compounds.

5 CONCLUSIONS

The suite of techniques used in these investigations, SIR, SERA, diffusion cell and GC-MS, have proved complementary and have been successful in characterising the protective properties of conformal coatings. In summary, these studies have shown that while the coatings are robust against corrosion induced by exposure to NaCl when this is external to the coating, exposure to dibasic weak acids can cause corrosion of the underlying metallisation. Diffusion experiments, with free films as permeable membranes, followed by GC-MS were used to indicate that these acids do penetrate the film.

The SIR measurements provide an indication of the permeability of the coatings to moisture and contaminants. It also gives the relative performance of different coatings with similar or dissimilar chemistry. The measurements indicate that the coatings provided protection from NaCl contamination but not from the flux contamination. That the SIR results showed that the dibasic acids in the flux proved so aggressive was a surprise and further investigations were undertaken with the other techniques. Also, the permeability of the coatings to water does not appear to affect their ability to protect circuitry from corrosion as none of the conformally coated, but uncontaminated, boards showed any signs of corrosion.

The SERA technique has proved to be very useful for the evaluation of the level of corrosion attack under the conformal coatings. SIR runs out of discrimination when the results hit the limit at $10^6 \Omega$; here the complementary aspect of the SERA technique becomes evident. For SIR results of $10^6 \Omega$ the SERA results reveal associated levels of corrosion attack in terms of the amount of nickel and copper oxides. The correlation of SERA results and the SIR results makes the SERA technique a promising tool for evaluating the permeability of conformal coatings.

The diffusion cell experiments used to evaluate chloride ion migration confirmed the results obtained from the SIR measurements at the same temperature; that is, the coatings are reasonably robust against NaCl penetration. The flux migration evaluations, under same conditions, however showed that the coatings are permeable to the weak organic acids present in the fluxes. This result was confirmed by the GC-MS measurements of extract solutions, which showed the presence of small amounts of at least two of the acids in the uncontaminated side of the diffusion cell at the end of the

50-hour test period. This is an important result, confirming the diffusion of these acids through conformal coatings.

Permeability of the conformal coatings to the organic acids is an interesting development and future work at NPL will explore a wider family of similar organic compounds from a range of industrial applications.

6 ACKNOWLEDGEMENTS

The authors would like to thank Concoat, Henkel, Dow Corning, Beck Insulation and HK Wentworth for conformally coating boards for testing and also providing us with samples of their conformal coatings. The authors would also like to gratefully acknowledge Anthony Buxton and Richard Holman of the Paints Research Association for their consultation and experimental work on the diffusion cell and GC-MS.

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- [5] Sanyo Gallenkamp Plc, Leicester, UK.
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- [10] J. Waryold, J. Lawrence and E. Mines, 'A conformal coatings selection guide', SMT February 1998, p84.
- [11] C.J. Tautscher, 'Conformal coatings – selection criteria', SMT, July 1999, p64.

8 APPENDIX 1

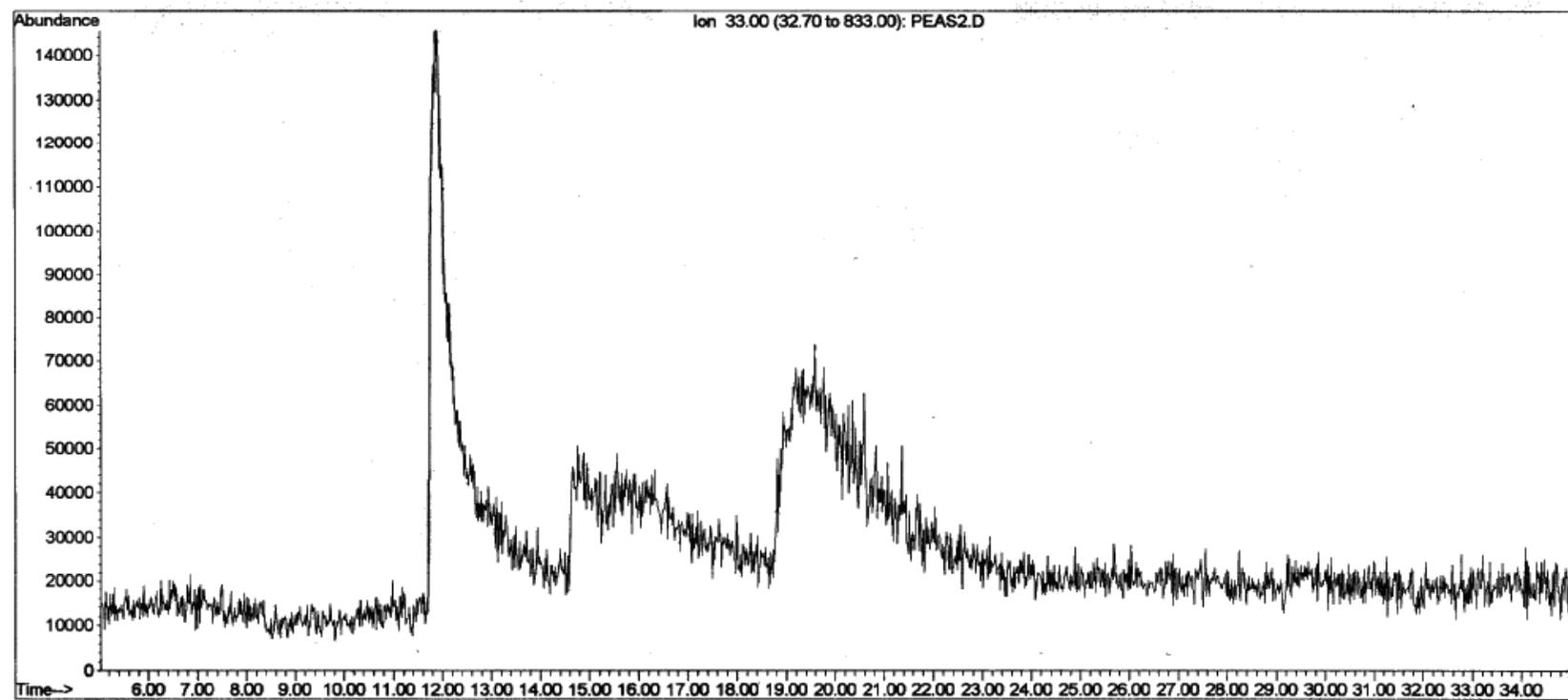
GC-MS Flux Analysis

Figure A1. Chromatogram of succinic acid

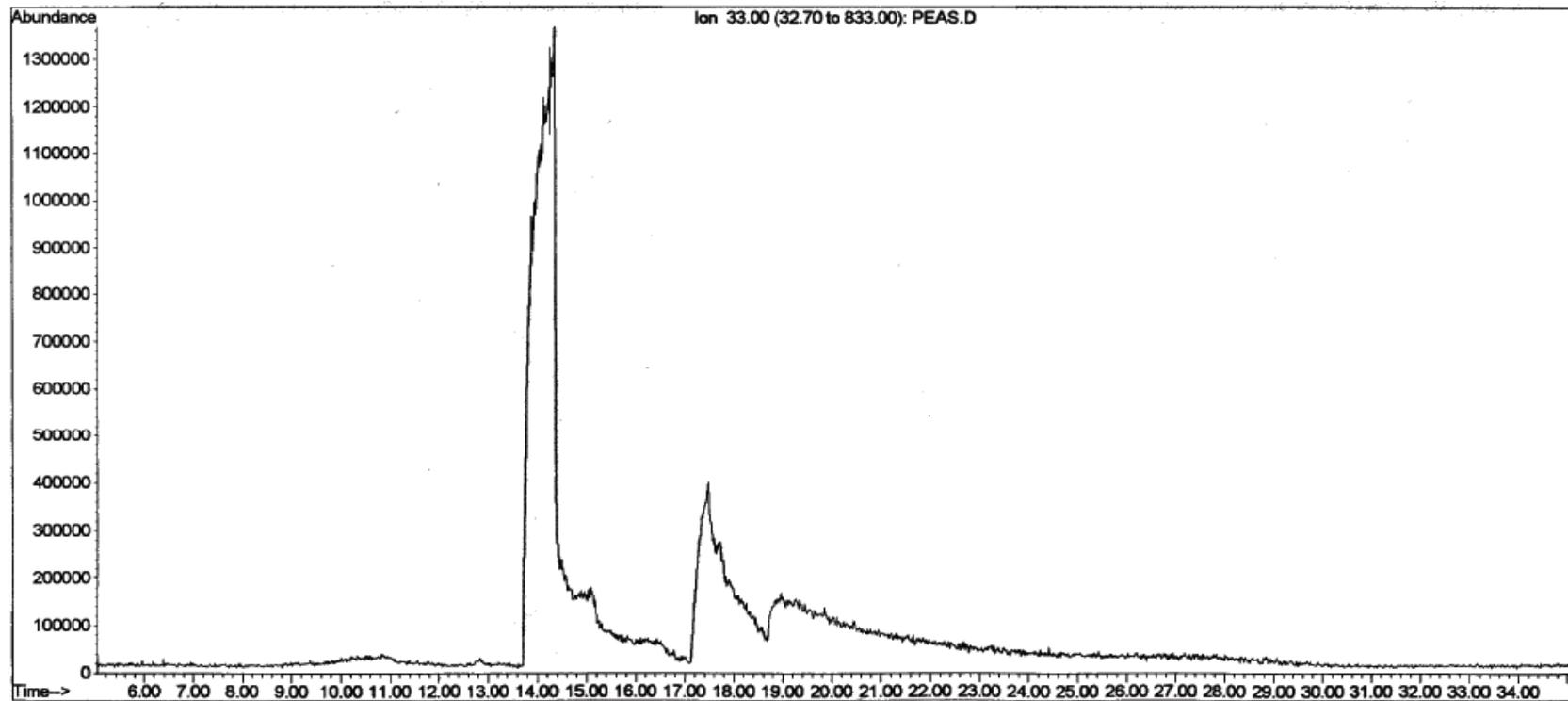


Figure A2. Chromatogram of glutaric acid

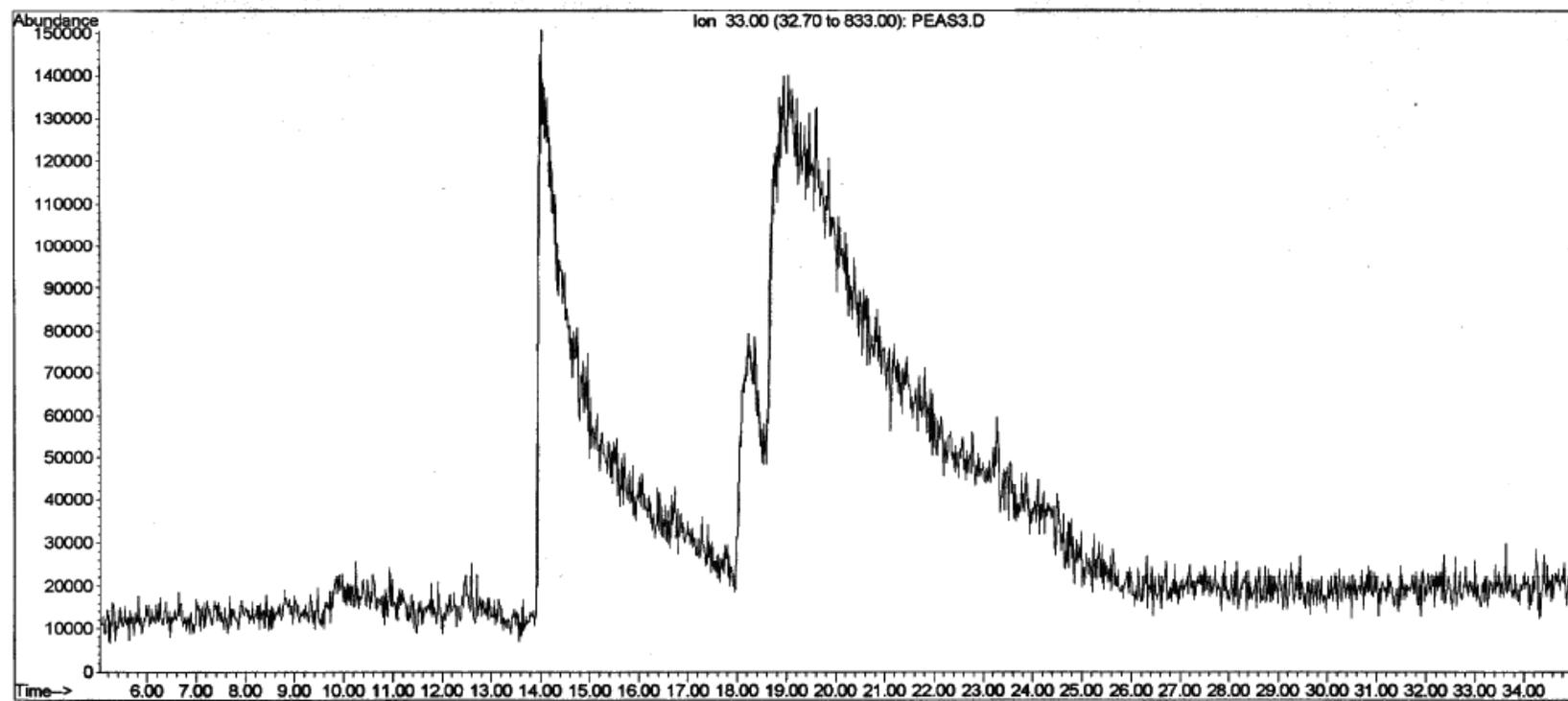


Figure A3. Chromatogram of adipic acid

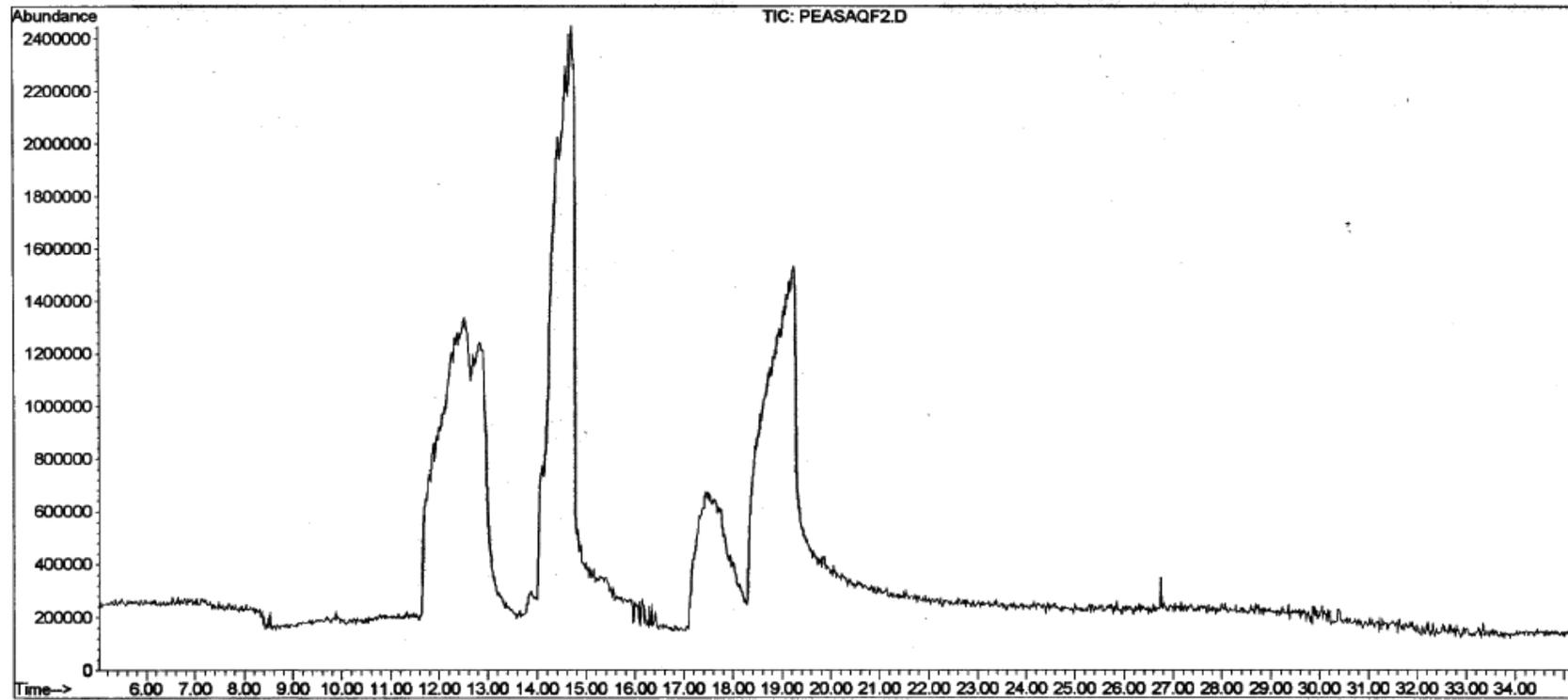


Figure A4. Chromatogram of water-based flux B

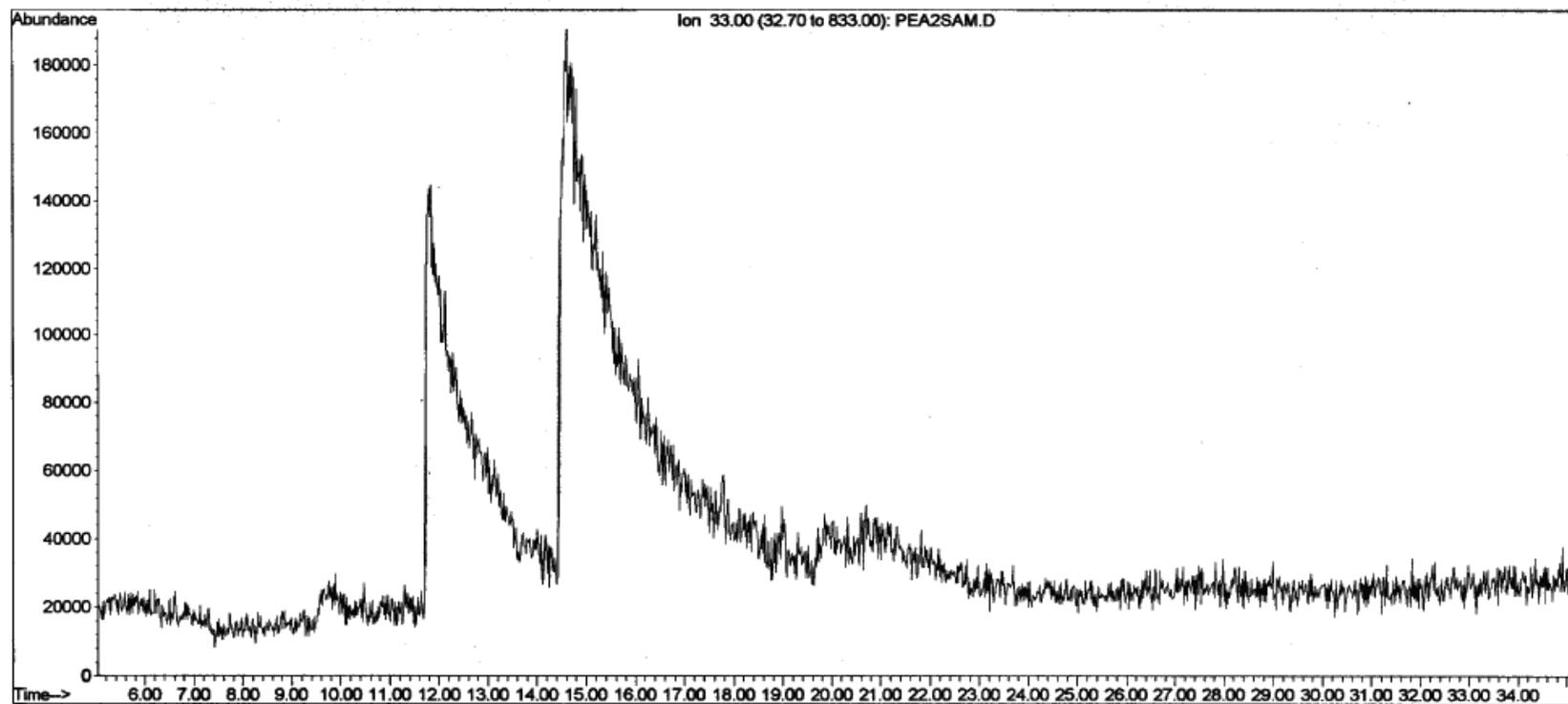


Figure A5. Chromatogram of initially uncontaminated solution from the diffusion cell experiment

Library Searched : C:\DATABASE\sollib.1
Quality : 83
ID : Succinic Acid

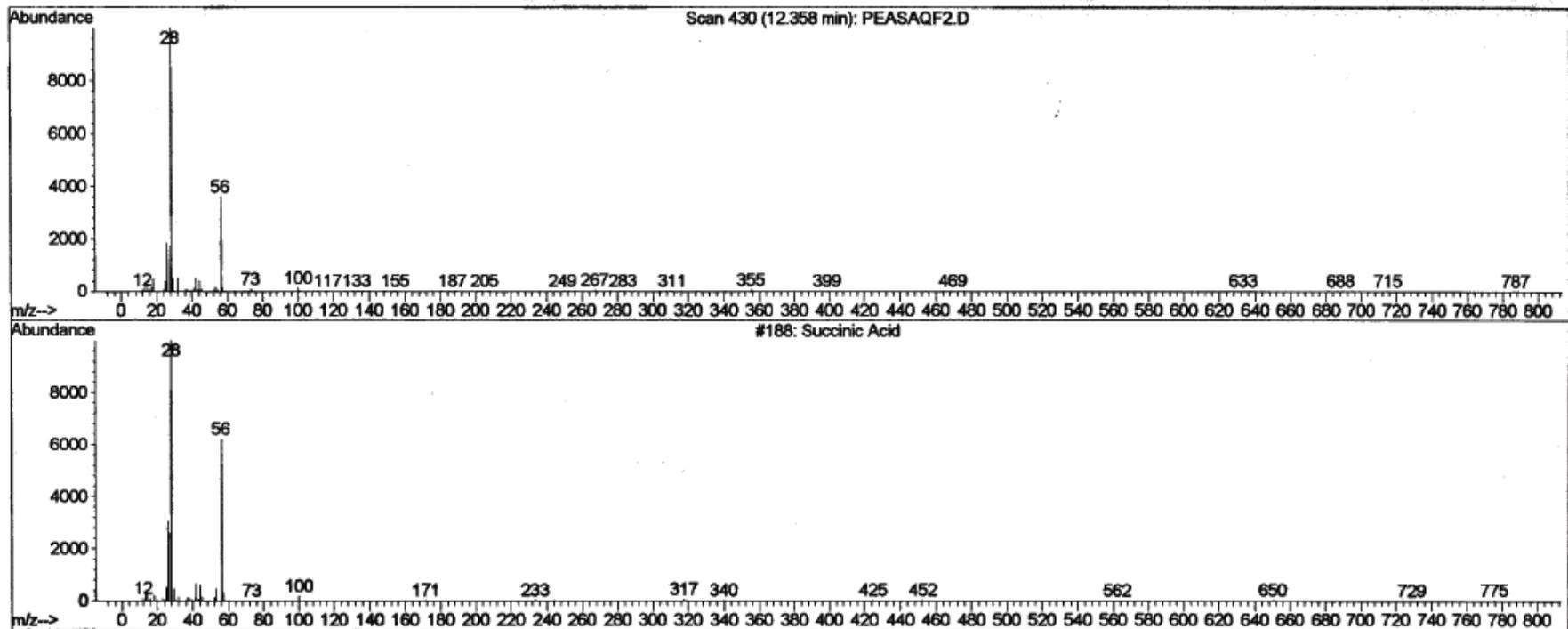


Figure A6. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 12.358 minutes (top) and library match succinic acid (bottom)

Library Searched : C:\DATABASE\sollib.1
Quality : 74
ID : Adipic Acid

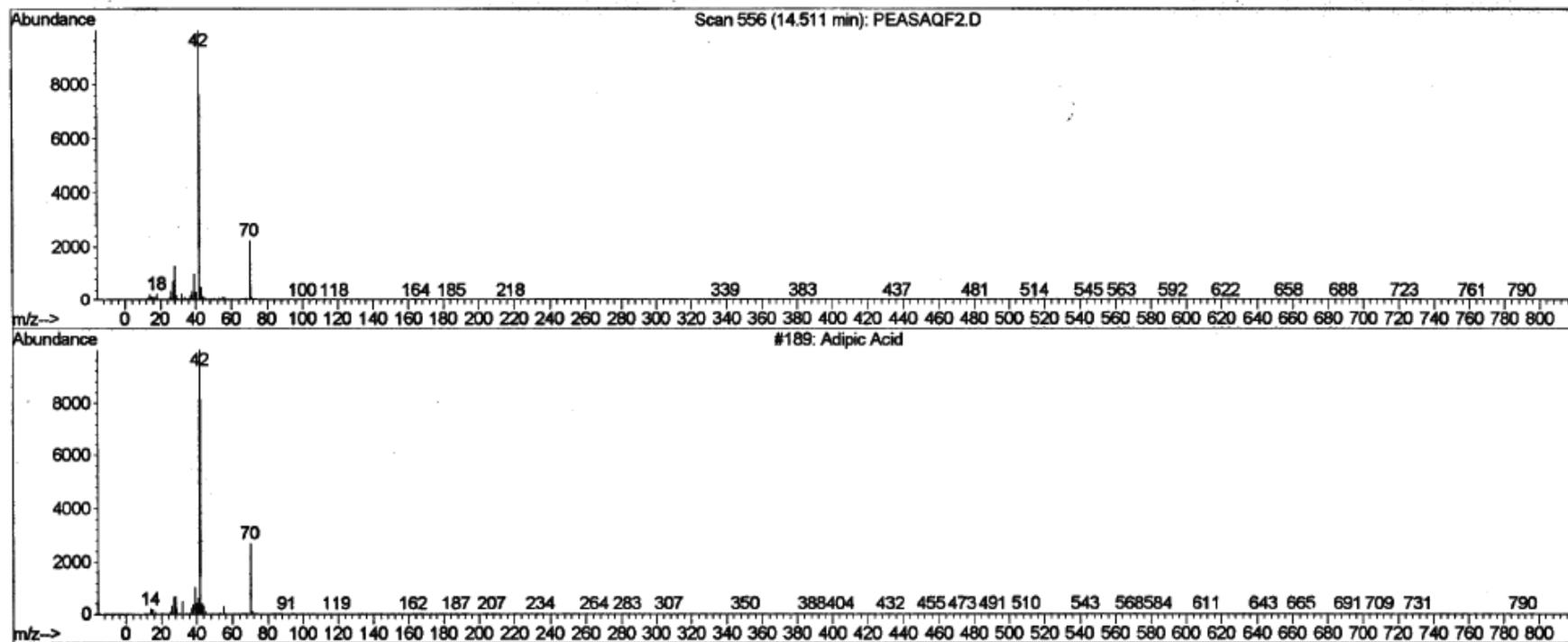


Figure A7. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 14.511 minutes (top) and library match adipic acid (bottom)

Library Searched : C:\DATABASE\sollib.1
Quality : 83
ID : Glutaric Acid

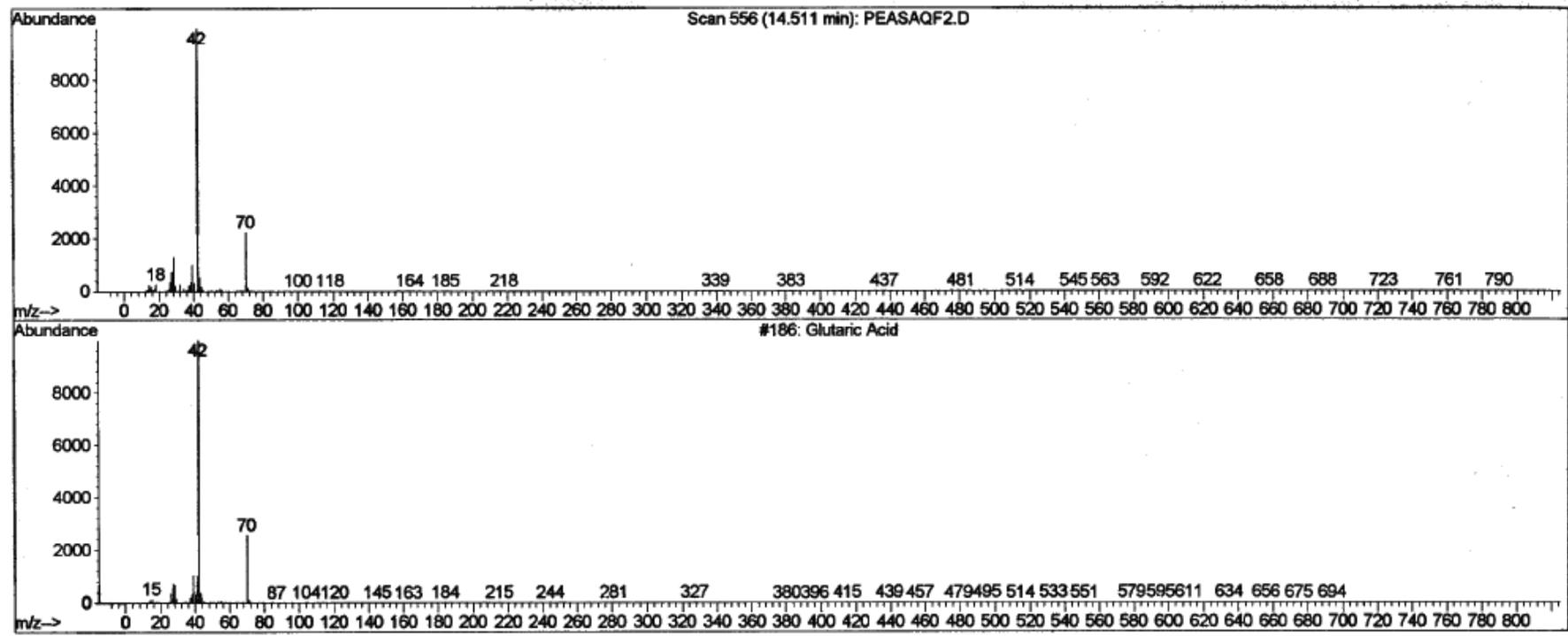


Figure A8. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 14.511 minutes (top) and library match glutaric acid (bottom)

Library Searched : C:\DATABASE\sollib.l
Quality : 58
ID : Minor Component of Glutaric Acid

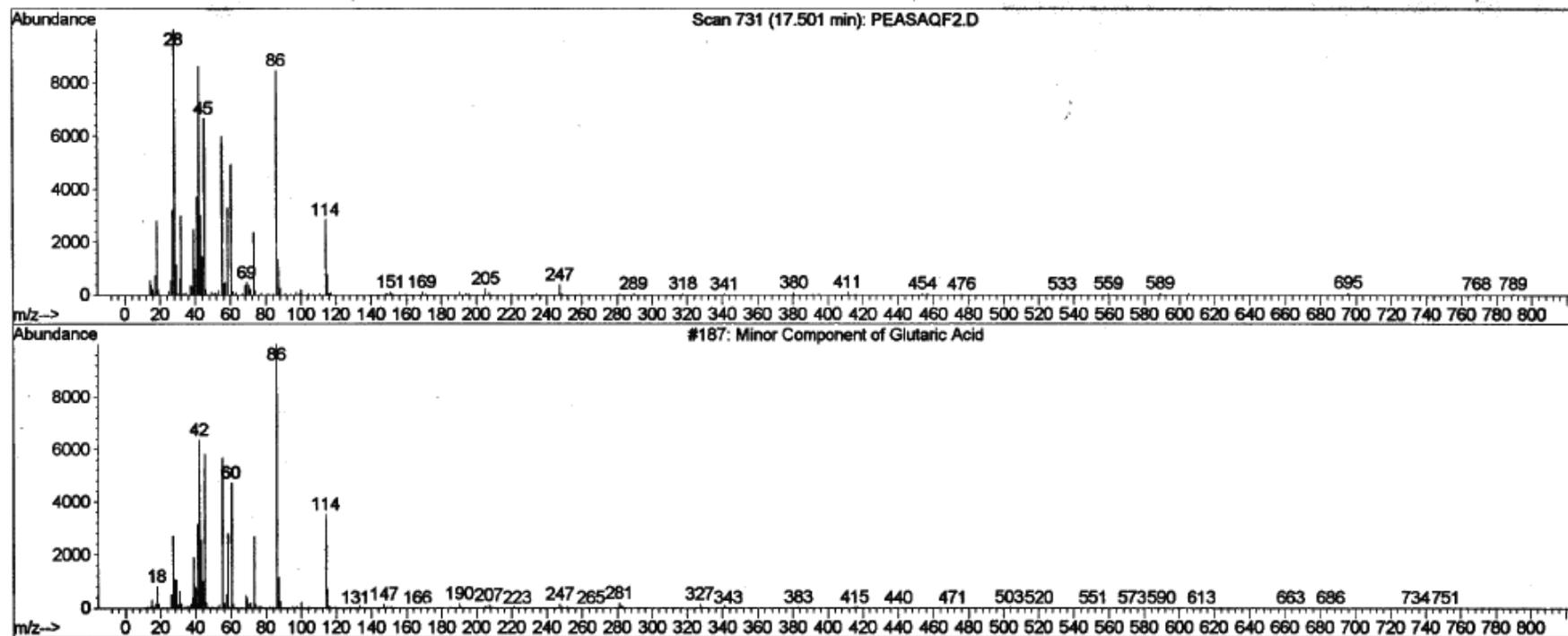


Figure A9. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 17.501 minutes (top) and library match the minor component of glutaric acid (bottom)

Library Searched : C:\DATABASE\sollib.l
Quality : 50
ID : Minor Component of Adipic Acid

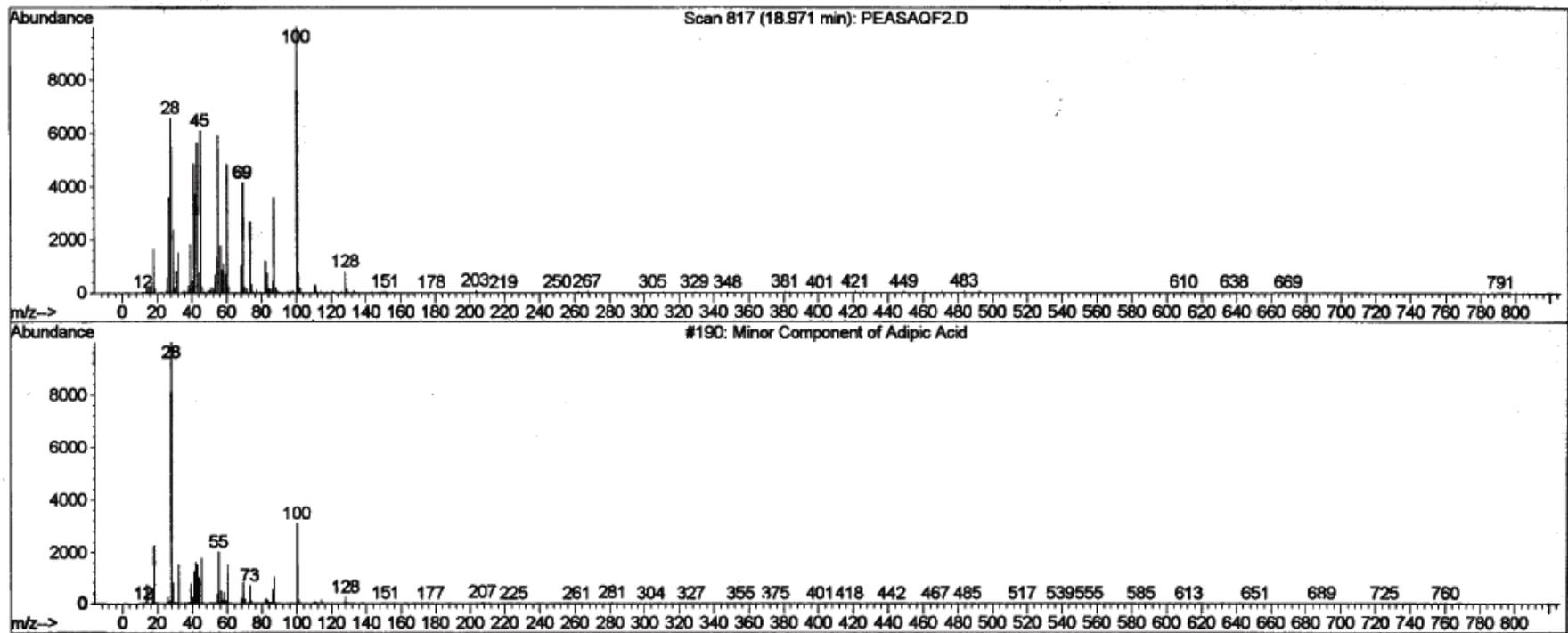


Figure A10. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 18.971 minutes (top) and library match the minor component of adipic acid (bottom)

Library Searched : C:\DATABASE\sollib.1
Quality : 53
ID : Succinic Acid

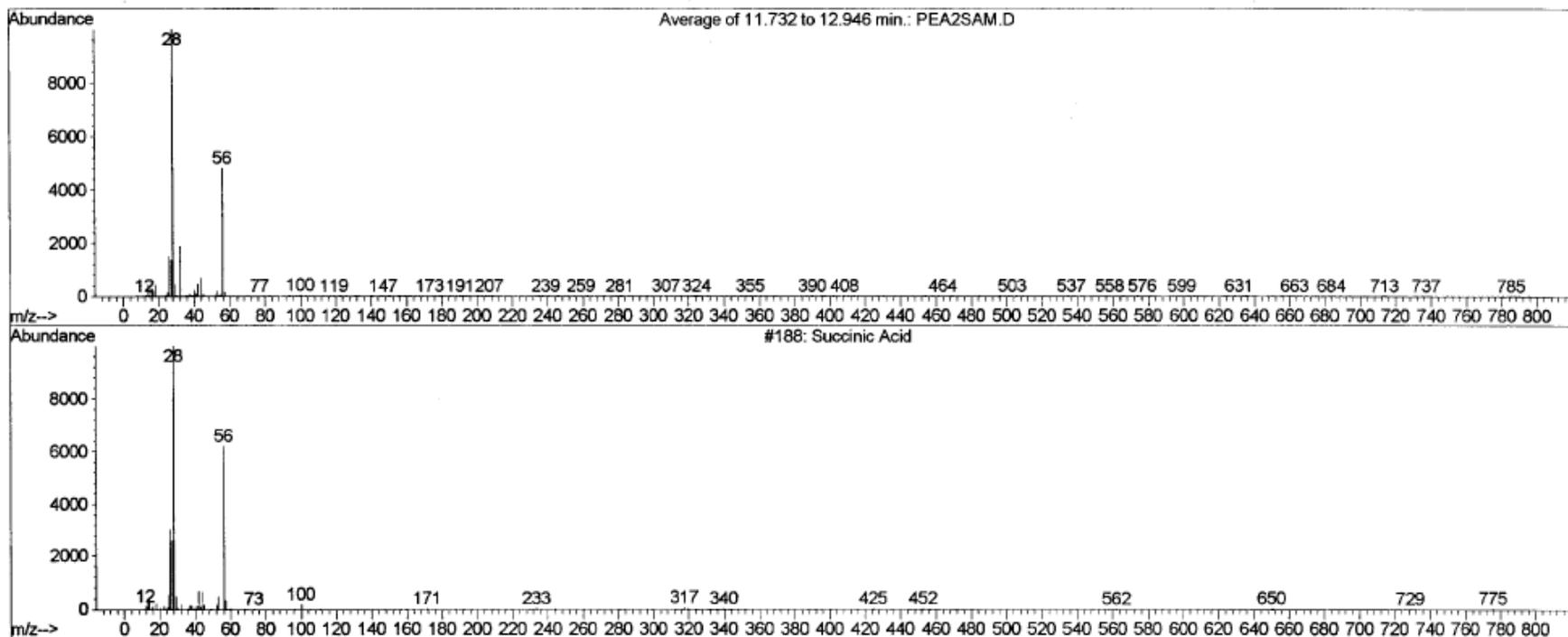


Figure A11. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 11.7 -to- 12.9 minutes (top) and library match succinic acid (bottom)

Library Searched : C:\DATABASE\sollib.
Quality : 72
ID : Glutaric Acid

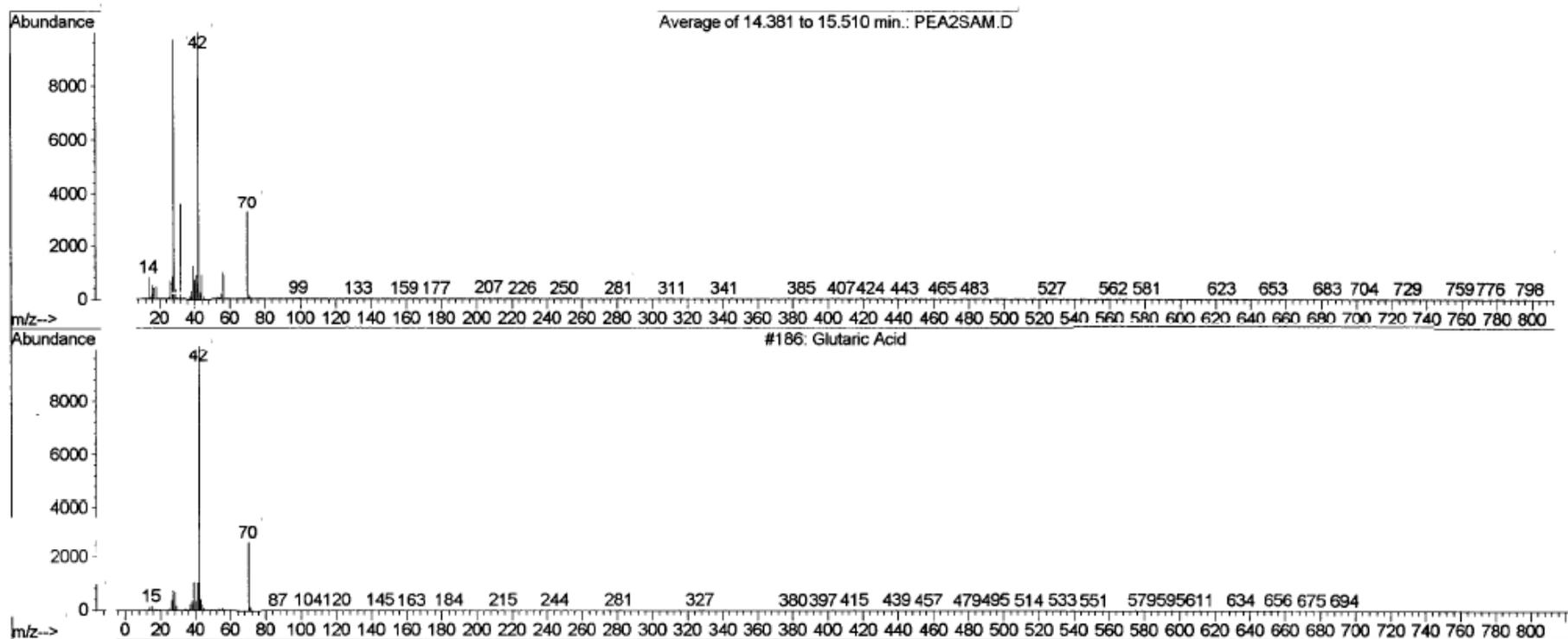


Figure A12. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 14.3 -to- 15.5 minutes (top) and library match glutaric acid (bottom)

Library Searched C:\DATABASE\sollib.l
Quality 64
ID Adipic Acid

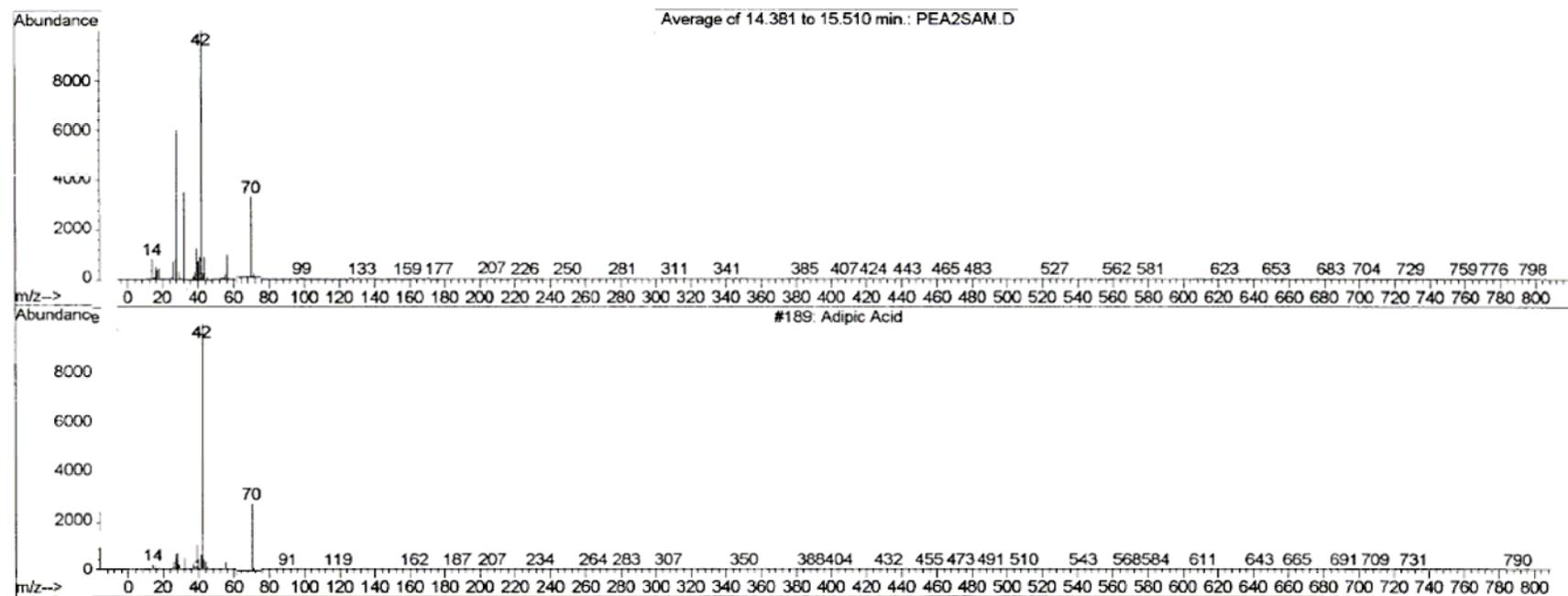


Figure A13. Mass spectrum of water-based flux B looking at the peak from Figure A5 at 14.3 -to- 15.5 minutes (top) and library match adipic acid (bottom)