

# Evaluation of Cleanliness Test Methods for Spacecraft PCB Assemblies

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## Abstract

Ionic contamination on printed-circuit-board assemblies may cause current leakage and short-circuits. The present cleanliness requirement in ECSS-Q-70-08, "The manual soldering of high-reliability electrical connections", is that the ionic contamination shall be less than 1.56  $\mu\text{g}/\text{cm}^2$  NaCl equivalents. The relevance of the method used for measurement of the ionic contamination level, resistivity of solvent extract, has been questioned. Alternative methods are ion chromatography and measurement of surface insulation resistance, but these methods also have their drawbacks. These methods are first described and their advantages and drawbacks are discussed. This is followed by an experimental evaluation of the three methods. This was done by soldering test vehicles at four manufacturers of space electronics using their ordinary processes for soldering and cleaning printed board assemblies.

The experimental evaluation showed that the ionic contamination added by the four assemblers was very small and well below the acceptance criterion in ECSS-Q-70-80. Ion-chromatography analysis showed that most of the ionic contamination on the cleaned assembled boards originated from the hot-oil fusing of the printed circuit boards. Also, the surface insulation resistance was higher on the assembled boards compared to the bare printed circuit boards. Since strongly activated fluxes are normally used when printed circuit boards are hot-oil fused, it is essential that they are thoroughly cleaned in order to achieve low contamination levels on the final printed-board assemblies.

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## 1 Introduction

Contamination on PCB assemblies may cause current leakage and, in worst case, short circuits. Ionic compounds from flux residues are the most common contaminants causing current leakage. Therefore, ESA standards like ECSS-Q-70-08 [1] require monitoring of the cleanliness of PCB assemblies after soldering. Two basic methods are referred to in ECSS-Q-70-08: resistivity of solvent extract test and sodium chloride (NaCl) equivalent ionic contamination test. When the resistivity of solvent extract test is used, a mixture of 75% by volume isopropanol (IPA) and 25% by volume deionised water is poured in a fine stream onto both sides of the PCB assembly, with 1.55 ml solution for each cm<sup>2</sup> of assembly area. The starting resistivity of the test solution shall be greater than 6 Mohm-cm and the pass requirement is that the end value shall be greater than 2 Mohm-cm. The sodium-chloride equivalent ionic contamination test is also based on the measurement of change in resistivity of solvent extract, but in this case an instrument is used for performing the extraction. Furthermore, by calibrating the instrument using solutions with known concentrations of sodium chloride (NaCl), the change in resistivity can be transformed to the equivalent amount of NaCl that would give the same change in resistivity. Thereby, the result can be calculated as  $\mu\text{g}/\text{cm}^2$  NaCl equivalents. There are several automatic instruments available on the market that can be used for this type of test. However, the relevance of these test methods has been questioned for several reasons.

The first part of this report consists of a description of the effect of various contaminants on the current leakage, the historical background to the test methods referred to in ECSS-Q-70-08, the shortcomings of these methods, and a summary of alternative test methods. The second part consists of an experimental comparison of various test methods used for cleanliness assessment. The intention is to assess the adequateness of ESA's present cleanliness requirements.

## 2 Effect of Various Contaminants on Current Leakage

Current leakage due to contamination may cause unacceptably low insulation resistance between conductors or solder joints. It may also cause electrochemical migration, resulting in the formation of dendrites that cause short-circuits.

### 2.1 Effect of Contaminants on Insulation Resistance

Between two conductors of different potential separated by a dielectric material, a current leakage will occur. If the conductors are embedded in the dielectric material, the current leakage will depend on the resistivity of the bulk material. Besides the properties of the dielectric material, the resistivity will also be affected by the amount of absorbed humidity and various types of contaminants.

When conductors are located on the surface of a dielectric material, the current leakage is determined by the integrated effect of both surface and bulk resistivity. Since measurement of the resistance between two conductors on the surface of a dielectric material will inevitably include both bulk and surface resistance, the measured resistance is usually denoted as the Surface Insulation Resistance (SIR) [2, 3]. A special case occurs when the conductors are located at the interface between two materials, for example a laminate and a conformal coating. A large fraction of the current leakage then can take place at the interface.

Dielectric materials that have been properly qualified for production of PCB assemblies have high surface and bulk resistivity. Low surface and/or bulk resistivity is almost always caused by either improper processing (not fully cured) or by contaminants being present. Normally, the decrease in resistivity is the result of ionic conduction caused by non-reacted constituents or by ionic contaminants. Since migration of ions is involved in

the current leakage, the presence of water is normally mandatory for ions to take part in the process. Without water, ions will not be formed. Therefore, ionic contamination has a negligible effect on the resistivity in dry conditions [3].

In humid conditions, the bulk resistivity is normally much higher than the surface resistivity [4]. The reason for this behaviour is that a film of adsorbed water is formed on the surface. On  $\alpha$ -alumina substrates, roughly one monolayer of water is adsorbed at 35% RH, and more than 5 layers of water molecules at relative humidities higher than 70% [5]. Since the surface of the alumina substrate is hydrophobic, thicker water films can be expected to be adsorbed on hydrophilic organic substrates, but it is difficult to determine the thickness of adsorbed water films on organic substrates such as the epoxy- and polyimide-glass laminates selected for space use [6]. These substrates are somewhat different from the alumina ones as these organic substrates will absorb water.

Due to the intrinsic ionisation of water into hydrogen and hydroxide ions, an adsorbed water film has some conductive properties. This may be further enhanced by adsorption of some gases from the atmosphere that form ionic compounds with water, such as carbon dioxide. Nevertheless, the impact of a pure water film on the surface resistivity does not normally pose a problem. It is the function of the water film as a medium for ionisation of ionic compounds that renders adsorbed water films hazardous.

The conductivity of an adsorbed water film is much less than for bulk water [5]. A water film five layers of molecules thick has a conductivity approximately two orders of magnitude lower than that of bulk water, and a water film of 20 layers has a conductivity that is still one order of magnitude lower than that of bulk water. The reduced conductivity is due to a strong interaction between the adsorbed water molecules and the substrates, which affects the properties of the water film. Probably, it is also affected by a non-uniform distribution of the water on the surface. Therefore, at a relative humidity below a certain value, ionic conduction will be insignificant unless hygroscopic contaminants are present. A hygroscopic compound absorbs water above a certain critical relative humidity and thereby increases the thickness of the adsorbed water film. In Table 1, the critical relative humidity is given for a number of inorganic compounds containing halides.

Compound	Temperature (°C)	Relative humidity (%)
LiCl.H <sub>2</sub> O	20	15
KF	100	22.9
NaBr	100	22.9
CaCl <sub>2</sub> .6H <sub>2</sub> O	24.5	31
CaCl <sub>2</sub> .6H <sub>2</sub> O	5	39.8
KBr	100	69.2
NaCl	20	75
KCl	80	78.9
KBr	20	84
KCl	0	88.6
NaF	100	96.6

Table 1. Critical relative humidities for a number of inorganic compounds [7]

As can be seen in Table 1, the critical relative humidity varies considerably for the presented compounds. Furthermore, the data for calcium chloride, potassium bromide, and potassium chloride show that the critical relative humidity is temperature-dependent, the compounds become more hygroscopic at higher temperatures.

Anderson et al. [8] have shown that the resistivity of a surface contaminated with a hygroscopic compound decreases dramatically when the relative humidity is increased to values above the critical relative humidity for the hygroscopic compound. The resistivity is then rather little affected by a further increase in relative humidity. For an assembled PCB, the surface would be contaminated with a mixture of contaminants from various process steps having varying critical relative humidities. Therefore, the surface resistivity for an ordinary production assembly can be expected to decrease more or less gradually with increasing relative humidity.

Not only ionic compounds can have hygroscopic properties. Polyglycols and many other types of organic non-ionic surfactants common in some types of fluxes and fusing oils are very hygroscopic even at relative humidities down to 0% [9]. When present alone, they contribute to a decrease in the surface resistivity mainly by increasing the thickness of the adsorbed water film. Since they do not contribute to ionic conduction, the decrease in surface resistivity is rather small at low concentrations. What makes them hazardous is the fact that they promote a dissolving medium for ionic contaminants with low hygroscopicity that would otherwise be rather harmless except at very high relative humidities. Thereby, strong synergistic effects may be observed when hygroscopic non-ionic compounds are mixed with ionic compounds of low hygroscopicity. For example, as shown in Table 2, a mixture of polyethylene glycol and adipic acid has the same impact on SIR as sodium chloride [10]. The figures in Table 2 also show that ionic compounds have a negligible impact on SIR at relative humidities below the critical relative humidities for the compounds.

Substance	Added amount ( $\mu\text{g}/\text{cm}^2$ )	Surface conductivity ( $\text{ohm}^{-1}$ )	Critical RH* (%)
NaCl	2.00	$1.3 \times 10^{-8}$	76
NaF	1.44	$4.0 \times 10^{-11}$	97
NaBr	3.52	$3.8 \times 10^{-11}$	84
KCl	2.55	$3.1 \times 10^{-11}$	84
MgCl <sub>2</sub>	1.63	$2.9 \times 10^{-8}$	44
CaCl <sub>2</sub>	1.90	$9.5 \times 10^{-8}$	29
Adipic acid	5.00	$2.7 \times 10^{-11}$	99.6
PEG 400**	13.70	$3.2 \times 10^{-10}$	0
Adipic acid + PEG 400	5.00 + 13.70	$1.4 \times 10^{-8}$	
Reference	0.00	$3.5 \times 10^{-11}$	

\* Calculated values

\*\* PEG 400 = polyethylene glycol with a molecular weight of 400.

Fortunately, not all contaminants are hazardous - some are even beneficial. Rosin, a common base in many fluxes, is hydrophobic, i.e. water repellent [9]. Therefore, rosin residues decrease the amount of water adsorbed on the surface and thereby improve the surface resistivity. They may also encapsulate ionic contamination and thereby immobilise ions. In addition, water that condenses on a hydrophobic surface tends to form isolated droplets, as water does on a greasy surface. Thus, even if condensation occurs, rosin residues improve the situation. It should be noted that pure rosin residues might be acceptable in this instance for manned spacecraft, but for space units that operate under vacuum all flux residues are to be avoided as they are known to have a high outgassing rate and can cause adjacent units to be contaminated (e.g. optical systems, commutators, etc.).

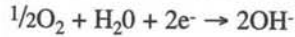
Table 2. Surface conductivity for copper comb patterns on FR-4 substrate contaminated with various compounds [10]

## 2.2 Effect of Contaminants on Formation of Dendrites

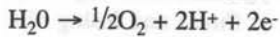
The current leakage due to ionic conduction must be accompanied by a reduction process at the cathode and an oxidation process at the anode. The reduction process at the cathode may be:



or



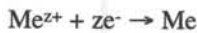
and the anode reaction:



The cathodic reaction gives rise to an increase in the pH at the cathode, whereas the anodic reaction brings about a decrease in the pH at the anode. The anode reaction may also involve oxidation of metals forming the anode. All common metals used in conductors can be oxidised, including gold, platinum, and palladium, although these three can only be oxidised in the presence of contaminants forming strong complexes with the metals, for example chloride, bromide, and iodide [11], the first two of which are common contaminants on assemblies. The oxidation of metals at the anode can be written:



Under favourable (or rather unfavourable) conditions, the dissolved metal ions can migrate to the cathode and there be reduced back to metal:



These are the same reactions that occur during electroplating. However, in contrast to electroplating, the metal in this case is not plated as a metal film on the cathode. Instead, it is plated in tree-like structures called dendrites (Figure 1). The failure mechanism is usually referred to as **electrochemical migration**. With time, the dendrites may completely bridge the space between the conductors, causing short-circuits. Since the dendrites are very thin, short-circuits usually burn off part of or the whole dendrite. Thus, failures caused by the formation of dendrites are normally intermittent in nature, and it may be very difficult to track the reason for such failures, especially if the dendrites have been burned off or if they are formed beneath components where it is difficult to see them.

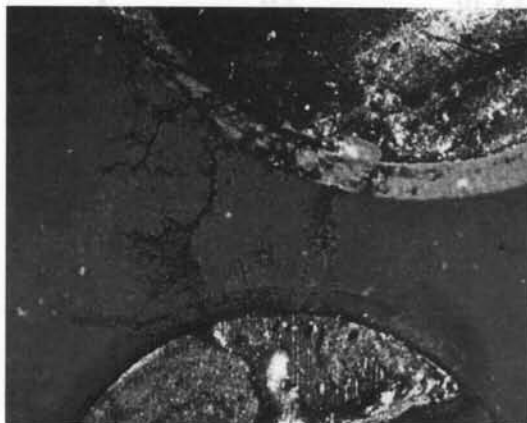


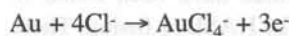
Figure 1. Dendrites formed on an assembly exposed to condensation. The dendrite to the right has caused a short circuit and is partly blown away

For dendrites to form, the metal ions formed at the anode must be able to migrate all the way to the cathode without being precipitated as insoluble compounds. As already indicated, acidic conditions can be expected to prevail at the anode and alkaline at the cathode. However, the actual pH of the surface will also depend on the contaminants present on the surface. Some common contaminants are alkaline, for example sodium and potassium carbonate from alkaline solder-mask developers or residues from alkaline



cleaning solutions. Examples of acidic residues are weak organic acids, such as adipic acid, forming a part of many no-clean fluxes. Rather few, if any, of the metal ions normally found in dendrites are soluble across the whole pH-range that may prevail between the conductors, especially at neutral and high pH. For example, copper dendrites require a pH of less than 5 in order to be able to form [12]. Silver(I) ions are among those metal ions that are soluble at rather high pH [13], which is probably one of the reasons why silver has been found to be the metal that forms dendrites most easily. When tin is oxidised, tin(II) ions are first formed which may be further oxidised to tin(IV). Tin(II) and especially tin(IV) require very low pH if they are not to be precipitated as hydroxides in water solutions. Lead(II), which is formed when lead is oxidised, is less soluble than silver(I), but more soluble than tin(II) and tin(IV). Thus, lead is more prone to form dendrites than tin. For an assembly exposed to field conditions, exposure to high humidity levels will usually be followed by drying-up periods. Soluble metal salts may then be converted into insoluble compounds.

As already mentioned, gold can only be oxidised in the presence of certain anions, notably chloride, bromide and iodide. These are anions that form very strong soluble complexes with gold ions. In the presence of chloride ions, the anode reaction can be written:



The strong complexes formed with chloride also make gold ions soluble in solutions with rather high pH, thus facilitating the migration of gold ions towards the cathode and thereby the formation of gold dendrites. Chloride and bromide ions also form strong complexes with cations of silver, copper, tin and lead. In fact, that is the reason why they are added to fluxes. Their strong tendency to form complexes with the metals commonly used as surface finishes facilitates the breaking of the oxides on these surfaces. Consequently, many of the substances added to fluxes in order to facilitate soldering can be expected to form strong complexes with metals common in the surface finishes of printed boards, although not necessarily as strong as those formed with chloride and bromide. The presence of complex-forming compounds is highly favourable for the formation of dendrites and is probably a prerequisite for dendrites to form in most cases. An interesting fact is that most complexes formed with chloride and bromide have a negative charge. For example, the complexes formed between silver and chloride,  $\text{AgCl}$ ,  $\text{AgCl}_2^-$ ,  $\text{AgCl}_3^{2-}$  and  $\text{AgCl}_4^{3-}$ , are either neutral or negatively charged. The concentration of each species depends on the concentration of chloride ions in relation to silver ions. The larger the surplus of chloride ions, the higher is the concentration of the species with silver ions surrounded with three and four chloride ligands. Thus, in the presence of chloride and bromide ions, migration of silver ions from the anode to the cathode will not be driven by the potential difference since it will work in the opposite direction. Migration would be driven by difference in concentration. Under field conditions, most of the migration may take place during turn-off periods.

A special case of migration can occur under certain conditions along glass-fibre/epoxy interfaces within board laminates, when a glass fibre bundle is in contact with biased surfaces of different potential [14, 15]. The connection can be between two conductors, between a conductor and a plated through-hole, or between two plated through-holes. The migration results in the formation of Conductive Anodic Filaments (CAFs), which may cause short-circuits. The first stage in the process is believed to be degradation of the glass/epoxy interface, followed by oxidation of the copper anode and migration of copper ions along the glass-fibre bundles. The filaments formed have a high content of copper and either fluoride or bromide [16, 17]. The most likely source for the halides are fluxes used when fusing or hot-air solder-levelling printed boards.

### 2.3 Contamination from Manufacturing of Assemblies

The production of PCBs includes many wet chemical treatments which all may leave residues. Examples of chemical processes are desmearing of drilled holes, etching

processes, plating of copper and surface finishes, and cleaning processes. It is important that thorough cleaning is performed so that as much as possible of the residues is removed.

The most contaminating process used for PCB fabrication is application of solder, whether it is applied using Hot-Air Solder Levelling (HASL) or hot-oil fusing. This process involves fluxing of the boards followed by heating to 240-260°C during solder application or fusing, i.e. at temperatures far above the glass transition temperature ( $T_g$ ) for FR-4 laminates and around the  $T_g$  for polyimide laminates [18]. The polymer resin in the laminate becomes soft at temperatures around and above  $T_g$  and flux ingredients are absorbed into epoxy resins through this treatment [19, 20] and probably also into polyimide resins. Epoxy and polyimide laminates may absorb flux ingredients to some extent even at temperatures as low as 125°C [21].

One group of ingredients that may be absorbed is polyglycols. It has been known for a long time that polyglycol materials are absorbed into the epoxy resin of FR-4 boards and that it is very difficult to remove these residues by cleaning [22, 23]. Inorganic ionic compounds may also be absorbed. High concentrations of chloride and bromide have been found in FR-4 laminates and solder masks [20, 24]. It is not possible to remove these residues completely even if very efficient cleaning processes are used. As mentioned earlier, large concentrations of chloride and bromide have been found in CAFs. The halides are believed to originate from fluxes used for HASL or fusing. There are indications that halides may diffuse through several layers in multilayer boards [17]. Some polyglycols have also been found to enhance CAF formation [25].

Fluxes used for soldering of assemblies will also leave residues on the surface, and some of the flux ingredients may be absorbed into the resin.

### 3 Historical Background of Ionic Contamination Measurements

Electrochemical migration was recognised as a failure mechanism in the sixties, and ionic residues from fluxes were identified to be the main cause. Consequently, work was done to develop a test method for analysing the amount of ionic contamination on the assemblies. In 1972, Hobson and DeNoon presented a method based on extracting ionic contamination by a mixture of water and isopropanol, usually 75% isopropanol by volume [26]. The resistivity of the solution was measured before and after the extraction using a conductivity bridge. The change in resistivity indicated the amount of ionic substances extracted. Originally, the assembly was flushed manually with a pre-determined quantity of the solvent, namely 10 millilitres per square inch (1.55 ml/cm<sup>2</sup>). Later, automatic test instruments were developed. This test was incorporated into the military standard MIL-P-28809 (replaced in 1989 by MIL-STD-2000 [27]) with an acceptance requirement that the resistivity of the solution after extraction should not be less than 2 Mohm-cm when the starting solution had a resistivity greater than 6 Mohm-cm. By comparing the resistivity of test solutions with the resistivity of sodium-chloride solutions of known concentrations, the registered values could be converted to  $\mu\text{g}/\text{cm}^2$  NaCl equivalents. Thereby, the acceptance criterion was transformed to read that the ionic contamination should be a maximum 1.56  $\mu\text{g}/\text{cm}^2$  NaCl equivalents (10  $\mu\text{g}/\text{in}^2$ ). The acceptance criterion was originally more or less arbitrarily chosen [28], but in 1978 a group of companies met and it was concluded that the acceptance criterion had indeed been used successfully for over 5 years in producing high-reliability electronic gear [29]. Thus, it was afterwards verified that the acceptance criterion was severe enough for the products of that time. This test method is often referred to as the Resistivity of Solvent Extract (ROSE) test or Solvent Extract Conductivity (SEC) test. It can be noted that the instrumental test method is also referred to as a ROSE or SEC test.

Since then, this test method has been adopted by most military and non-military standards having cleanliness requirements, usually with the same acceptance

requirement. It has been the main test used for ascertaining adequate cleanliness of printed boards and printed-board assemblies. At the time when the test was developed, through-hole mount technology was used exclusively. Military standards, for which the test method was developed, allowed only mildly activated rosin-based fluxes to be used, and it was required that the assemblies be cleaned after soldering using CFC-based cleaning solvents. Furthermore, the assemblies had to be conformally coated after cleaning. Since then, the materials and processes used for manufacturing assemblies have changed considerably.

As already mentioned, the test was originally developed for wave-soldered through-hole mounted assemblies, i.e. the flux residues were fairly evenly distributed over the board area. When using surface-mount technology, and especially reflow-soldering of surface-mount components, flux residues are more unevenly distributed. At locations beneath components that are difficult to clean, the concentration of flux residues may be considerably higher than on surrounding areas. Since ROSE testing gives the result as the mean value for the whole board area, it may be well within the required level despite high local concentrations. There was a note in MIL-STD-2000 (the standard was cancelled in 1995) pointing out this fact but, for some reason, this note has not been included in any other standard that has adopted the test method.

It is no longer permitted to use CFCs for cleaning electronics. This has led to the development of new types of fluxes many of which are rosin-free, for example water-soluble fluxes and fluxes based on weak organic acids. Furthermore, a large number of various types of cleaning solvents are used, including pure water and water with added saponifier. It was expressed in a note in MIL-STD-2000 that "The cleanliness test methods designed for rosin-based fluxes may not be adequate for cleanliness testing when non-rosin fluxes are used". Again, no other standard has adopted this reservation, nor has anyone shown that the test method is adequate when non-rosin fluxes are used. The cleaning methods used for extracting contaminants in automatic test instruments are usually much less efficient than the processes used for cleaning assemblies after soldering. The cleaning efficiency of a commercial test instrument may be less than 1% when evaluating the cleanliness of hot-air solder-levelled FR-4 boards [20]. Although the cleaning efficiency may be improved by using an instrument with better agitation and a heating system, even those systems may have cleaning efficiencies as low as 10% [28]. The most likely explanation for the low cleaning efficiencies is that the absorption of the flux ingredients into the laminate during the soldering process when the boards are fused or hot-air solder-levelled makes it very difficult to extract them. Thus, very efficient cleaning processes and extensive cleaning times are necessary if most of the ionic contamination is to be removed from the boards, both during cleaning after soldering and during cleanliness measurements. This conclusion is valid both for bare boards and assembled boards.

When automatic test instruments for cleanliness measurements were first developed, it was soon discovered that the various instruments gave quite different results. This is not surprising, bearing in mind the poor cleaning efficiency. Increasing the amount of detected ions from 5% to 10%, which is still an exceptionally poor cleaning efficiency, results in a 100% increase in detected ions. In 1978, a report was presented comparing the cleaning efficiency of various test instruments [29]. This evaluation of the cleaning efficiency was performed using the following procedure. Assembled through-hole mounted boards were produced and these were then extensively cleaned in order to achieve very clean assemblies. The assembled boards were then dipped into an RA flux, after which they were withdrawn at a specified rate in order to achieve a reproducible level of contamination. The assemblies were allowed to drain for five minutes at ambient conditions and were then dried at 107°C for 30 minutes. These assemblies were used for testing the cleaning efficiencies of various test instruments. The type of board laminate used was not specified, but most probably it was FR-4. That is, the fluxed test boards had never been exposed to temperatures above the  $T_g$  for the board laminate after the fluxes were applied, and very little of the flux residues could be expected to have been absorbed into the laminate. Furthermore, because of the low drying temperature, the rosin residues

should still be easy to dissolve in isopropyl alcohol compared to after a soldering process. Thus, the ionic residues ought to have been rather easy to extract. Nevertheless, large variations for the evaluated instruments were found, with cleaning efficiencies 1.39 to 3.25 times better than the manual method.

To solve the dilemma with instruments giving different results, an 'equivalence factor' was calculated for each instrument tested. The acceptance criterion was then multiplied by the equivalence factor for the instrument used for a cleanliness test. This was an interim solution that became permanent. New models and new instruments have been developed since then, but only for one of these has an 'equivalence factor' been determined [30]. This led to a situation in which you could choose between using the manual method or an old test instrument with poor cleaning efficiency and adjusting the acceptance requirement using the equivalence factor, or use more modern equipment with much better cleaning efficiency for which no equivalence factor has been determined. Passing the test was more a question of which test instrument was used, rather than the cleanliness of the board.

Even under ideal conditions, large differences in the amounts of contamination detected are observed for various instruments [31]. Test instruments use one of two alternative methods for measuring the amount of extracted ionic contamination, the static and the dynamic method. If the dynamic method is used, the solution is passed through a conductivity cell, which measures the conductivity continuously. These conductivity values are integrated over the time of the extraction. The mixture is then pumped through a resins deionisation column before being recirculated back to the test tank. When the static method is used, the solution is not regenerated during the measurement and the amount of ionic contamination is determined from the end resistivity of the extract. For equipment utilising the static method, weak organic acids will only be partly ionised. Therefore, the higher the concentration, the lower the fraction of the weak acid that will be ionised, causing the registered value to be dependent on residue quantity, flux composition, and volume of solvent to board area ratio. In all instruments, the solvent will absorb heat due to pumps moving the solvent and friction in the plumbing. A temperature increase of 5°C is not uncommon. This will have an impact on the measured resistivity. Other parameters that affect the results are alcohol concentration, stand-off of components, 'deadband', and absorption of carbon dioxide. 'Deadband' means an inability to register some contamination, if the solvent is regenerated to a resistivity above the system's probe capability.

Another drawback with the ROSE test method is that it is not possible to distinguish between different types of ions. The contamination may mainly consist of halides or no halides at all. Thus, it is hard to draw any conclusions regarding the origin or hazard of the detected contamination.

Since the test method was originally developed for military equipment to be used in harsh environments, there was no reason to have different acceptance requirements for various applications. Today, the same acceptance criterion that is used for products that will be used under harsh outdoor conditions and with a very long lifetime requirement is also used for products that will be used under dry indoor conditions and with a short lifetime requirement. Obviously, this will lead to some over-specified PBAs, whereas others are under-specified, i.e. some are unnecessarily expensive to produce, whereas others will not meet reliability requirements [32].

Although these objections are severe enough to question the use of ROSE testing, the main drawback is that it is not a true reliability test, in that it does not accelerate a failure mechanism. If the test results are to be of any value, there must be a known relationship between what is analysed, i.e. the total amount of ionic contamination, and the reliability of the product. No such relationship has ever been shown for any type of application, not even when the test method was developed. However, as concluded in 1978, the ROSE test using 1.56  $\mu\text{g}/\text{cm}^2$  NaCl equivalents as the acceptance criterion of ionic contaminants had been used successfully for over 5 years in producing high-reliability

gear [29]. Thus, actual field service confirmed that the cleanliness requirement was severe enough to give reliable products with the materials and manufacturing methods used at that time, i.e. assemblies with through-hole mounted components soldered using rosin-based fluxes, cleaned using CFCs, and conformally coated.

Even if the extraction efficiency is improved and test methods are employed that can analyse the presence and concentration of specific ions, for example ion chromatography, these are still not accelerated reliability tests. The problem remains that there must be a known relationship between what is analysed and the reliability of the product. Since synergistic effects may occur (see Table 2) it would, in practice, be impossible to establish such relationships. However, these types of test methods can be useful for tracking the origin of contaminants. They can also be used to check that the cleanliness does not change with time in a running process, which would indicate that the manufacturing process is not under control.

## 4 Alternative Test Methods

### 4.1 Ion Chromatography

An alternative method for measuring cleanliness is ion chromatography. It is also based on solvent extraction, but the cleaning efficiency can be improved by using ultrasonic agitation (may not be acceptable for some assemblies). The IPC has standardised a method for ion chromatography, but no acceptance criteria are specified [33].

The largest advantage with this method is that it identifies the types and amounts of anions on the assemblies. However, this method also has the drawbacks that the measured value is an average for the whole board area, and there is no clear correlation between measured value and reliability.

### 4.2 Surface Insulation Resistance

Adequate evaluation of the impact of contamination on reliability must be based on acceleration of the crucial failure mechanisms that are caused by various contaminants. In the case of current leakage, these failure mechanisms are deterioration of surface insulation resistance and electrochemical migration. Surface insulation resistance testing has long been used to evaluate the impact of contaminants on current leakage and dendrite formation, but even using this test method it may be difficult to interpret the results. Misconceptions and poor understanding of the objectives of SIR testing are common [32]. The difficulties with using SIR testing as a tool for reliability evaluations of PBAs are caused by several factors, the most important of which are:

- Lack of definitions of expressions used or inconsistent use of defined expressions.
- Insufficient knowledge of the failure mechanism.
- Poor understanding of what one is trying to achieve with SIR testing.
- Difficulties in testing functional PBAs. Specially designed test patterns are required for adequate testing.

One major reason for the confusion that exists about SIR testing is that surface insulation resistance is a material property, whereas the purpose of the majority of SIR test methods is to accelerate electrochemical migration, which is only indirectly related to SIR [34]. SIR is defined in IPC-9201 [35] as “...a property of the material and electrode system. It represents the electrical resistance between two electrical conductors separated by some dielectric material(s). This property is loosely based on the concept of sheet resistance, but also contains elements of bulk conductivity, leakage through electrolytic contaminants, multiple dielectric and metallisation materials and air”.

It is far more complicated to measure SIR than it at first appears. SIR is determined using Ohm's law  $R=U/I$ . In humid conditions, most of the current on contaminated boards is generated by the migration of ions. This will cause a depletion of ions in the area between

the biased surfaces, which will result in a decrease in the current, i.e. an increase in SIR. Thus, the actual measuring of SIR will affect the measured value. As shown by Chan [36], the increase in SIR on a contaminated board is fast at the beginning and then gets slower and slower. When a board was contaminated with  $10 \mu\text{g}/\text{in}^2$  ( $1.56 \mu\text{g}/\text{cm}^2$ ) of table salt, the initial SIR was  $5.3 \times 10^6$  ohms, compared to about  $1 \times 10^{10}$  ohms for a clean board. When a bias of 100 V DC was then applied to the contaminated board, the SIR increased to  $3.7 \times 10^7$  ohms after 60 seconds,  $1 \times 10^9$  ohms after a few hours, and  $8 \times 10^9$  ohms after 100 hours (see Figure 1). A clean test board showed almost no drift in SIR values. Thus, the SIR on the contaminated board approached the values of the clean board after 100 hours of testing.

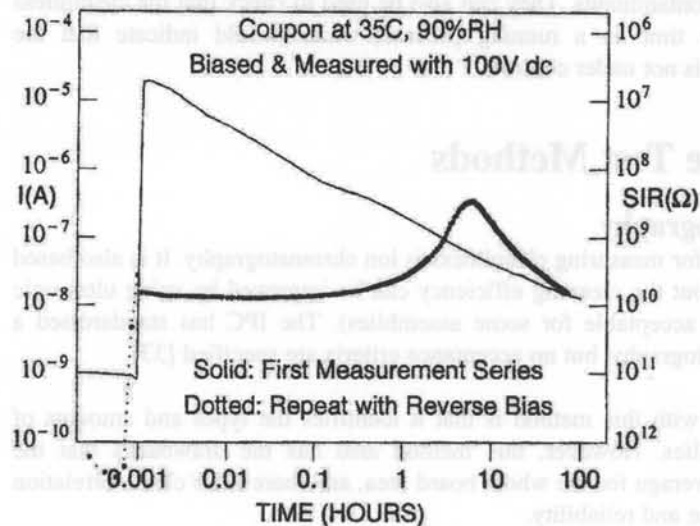


Figure 2. Current and SIR for a coupon contaminated with  $10 \mu\text{g}/\text{in}^2$  of table salt (from Ref. 36, © 1996 IEEE)

Obviously, in order to get reproducible data one, must define a stabilisation (electrification) time after the voltage has been applied and before a measurement is taken. Many standards prescribe a stabilisation time of 60 seconds, but that may be a little too short since the SIR may still drift rather fast after one minute [36, 37]. On the other hand, waiting too long will cause too large a drift in SIR. Furthermore, a measurement of SIR will be affected by previously performed measurements. This influence will be larger, the longer the stabilisation time is. Another implication of the ionic contribution to current leakage is that the measured SIR will depend on the applied voltage, as will probably the optimal stabilisation time also.

The drift of SIR during measuring and the influence of previous measurements on later measurements may be avoided by using an AC voltage at a low frequency of about 1 Hz [36]. Very little has been done in this field, but it may be the most relevant method for SIR measurement and efforts ought to be made to investigate this alternative more thoroughly.

A low SIR may alone be the cause of failures. The SIR level at which failures will occur depends on the impedance requirements of the circuitry. Thus, SIR measurements are useful for verification of the performance of a PBA, but SIR should then be measured directly with only a short stabilisation period or using AC voltages. As already discussed, SIR is affected to a very large extent by temperature and humidity. An increase in temperature by 20-30°C or an increase in the relative humidity by about 20-30% have been found to decrease the SIR by about one decade on boards without solder mask [3, 37, 38, 39]. However, the presence of a hygroscopic contaminant can cause a sudden large drop in SIR, when the relative humidity surpasses the critical humidity at which the contaminant becomes hygroscopic. Hence, an informative test would be to screen the relationship between SIR and the relative humidity in order to detect the presence of hygroscopic contaminants and determine critical relative humidities. Since the critical relative humidity decreases for many substances with increasing temperature (see Table 1) this screening should preferably be performed at both a low and a high temperature.

Some residues may be decomposed or evaporated at high temperatures, for example adipic acid and polyethylene glycol. For that reason, an extended test performed at 85°C may be more benign than a test performed at 65°C [40]. Furthermore, in many field environments, corrosive gas pollution and deposition of particles containing ionic material may have a large impact on the amount of ionic material on the surface and SIR. This must be considered when performing a SIR test and specifying acceptance criteria. In many SIR test methods, a bias is applied to the test patterns during only part of, or for the whole, test period. These tests may last up to 56 days. The purpose of such extended SIR tests is not to determine the SIR itself, but instead to assess the risk of electrochemical migration. Thus, a more appropriate name for such tests is 'electrochemical migration testing'.

According to the IPC, electrochemical migration is defined as [41] "*..the growth of conductive metal filaments on a printed board under the influence of a DC voltage bias. This may occur at an external surface, an internal interface, or through the bulk material of a composite. Growth is by electrodeposition from a solution containing metal ions, which are dissolved from the anode, transported by the electric field and redeposited at the cathode. We are thus excluding phenomena such as field-induced metal transport in semiconductors and diffusion of the products arising from metallic corrosion*".

The fact that SIR is measured during an electrochemical migration test may give the impression that the risk of electrochemical migration can be measured and given a numerical value. That is not the case. Although electrochemical migration causes migration of metal ions, there is no way to separate electric current due to migrating metal ions from current due to other migrating ions. In fact, the bias will usually in time cause an increase in SIR due to the ion-sweeping effect, indicating an improvement of the reliability. The formation of a dendrite normally does not have any impact on SIR, except for a few seconds when a short-circuit is formed and the dendrite is burned off [41]. Since SIR in most tests is measured once a day, or even with larger time intervals, and each measurement takes less than a second, it is very unlikely that a drop in SIR would be detected. Some modern instruments are designed to automatically measure SIR at a high frequency [42]. Although the chance of detecting a drop in SIR increases, there is still a large risk of missing most of the SIR drops that will occur. This could be mastered by using an event detector similar to the type used for detecting intermittent open circuits in solder joints, but in this case for detecting intermittent short-circuits. The time elapsing before a short-circuit occurs would be a good measure of the propensity for dendrite formation.

As long as event detectors are not used to detect short-circuits, dendrites have to be detected using some other method. Today, this is done by visual examination of the test boards using an optical microscope after the test has been run. If test patterns are located beneath components, it may be necessary to remove the components before the inspection. Using visual inspection, only the number, size, and location of dendrites can be evaluated. It is not possible to say when they were formed during the test. In order to prevent that dendrites are burned off when a short-circuit is formed, current-limiting resistors should be connected in series with each test pattern.

If electrochemical migration cannot be detected by SIR measurements, why measure SIR at all during an electrochemical migration test? Although there is no clear correlation between SIR and the tendency for dendrite formation, a high SIR indicates that electrochemical migration is unlikely and a low SIR indicates an increased risk for dendrite formation. However, it is not possible to exclude that dendrites will form if the SIR is above a certain value, nor will dendrite formation be inevitable if the SIR is below a certain value. A large number of parameters not directly correlated with SIR have a large impact on the tendency for dendrite formation, for example, as previously discussed, the pH of the surface and the presence of substances forming strong complexes with metal ions.

There are also other reasons for questioning much of the practice used today for SIR measuring during electrochemical migration tests. Originally, SIR measurements were

made with an instrument using 500 V DC as the measurement potential [34]. Several standards still prescribe that 500 V DC should be applied. Today, with much shorter insulation distances and lower voltages, this is a far too high a potential for many applications. Even if the applied polarisation bias is generally lower, polarisation biases of 100 V DC are not uncommon. They may induce failure mechanisms that would not occur under field conditions. Not only will the increased electrical field cause an increased ion migration rate, it will also increase the rate of the oxidation and reduction reactions at the anode and cathode, respectively. This will cause concentrations of ionic species that would not be found on a true product. Furthermore, the pH gradient would be larger than normal, i.e. the pH would be lower at the anode and higher at the cathode. The surroundings for migrating metal ions may be quite different from those in the field. Therefore, it is advisable not to use polarisation and measurement voltages that differ too much from the voltage to be used for the final product. To be on the safe side, polarisation and measurement voltages no higher than twice the service voltage gradient have been recommended [34].

As shown by Chan [36], SIR increases continuously when a bias is applied to a board contaminated with table salt, and approaches the value of a clean board after a few days (Figure 2). Thus, it seems odd to disregard initial measurements and have requirements on SIR only after some days of measuring, as several standards do. The impact of low initial SIR values needs to be explored more deeply.

The continuous increase in SIR due to depletion of ions in the area between the biased conductors during an electrochemical migration test is probably the reason why some standards prescribe that SIR measurements should be made using a voltage with reversed polarity after a stabilisation time of 60 seconds. As expected, by reversing the polarity, the SIR will first drop as ions start to migrate in the opposite direction, and then increase again after some time when the area between the conductors once more becomes depleted of ions. The drop in SIR may begin immediately but, as shown in Figure 2, it may also take more than an hour before starting and ten hours to reach a minimum [36]. Therefore, using reverse bias for SIR measurements only makes it even more difficult to interpret the results of electrochemical migration tests. Since a change of voltage with retained polarity will also cause an unpredictable drift in SIR, the best results are probably obtained if measurements are taken using the same voltage and polarity as the polarisation bias and without any interruption of the bias.

An unbiased period before measurement could be an alternative for another reason, but then it should be of some length. According to the IPC's definition of electrochemical migration, metal ions should be transported from the anode by the electric field and redeposited at the cathode, while diffusion of ions is excluded. There is a reason for questioning this definition. As in the case of electrochemical migration of gold, metal ions may be, and probably usually are, present as negatively charged complexes. Thus, the transportation of these to the cathode must be by diffusion against the electrical field. A test consisting of periods with applied bias followed by periods without any bias to allow for 'free' diffusion would perhaps be a more adequate test producing dendrites in a shorter time.

For products that will be used in such a way that PBAs will be contaminated during service, the impact of these contaminants on electrochemical migration must be considered. Corrosive gases and deposited particles containing ions may have a large impact not only on SIR, but also on dendrite formation. This must be considered when assessing the risk of dendrite formation. A test method for contamination with ammonium sulphate particles has been developed by AT&T Bell Laboratories [43].

### Test Vehicles

Special test patterns are necessary for SIR and electrochemical migration testing. The test patterns most frequently used are interdigitated comb patterns of various designs, some of which are shown in Figure 3. This type of test pattern can be useful for preliminary



screening of materials and processes used for production of PBAs. They are not suitable for evaluation of the impact of contaminants on the reliability of PBAs, for reasons that will be discussed later.

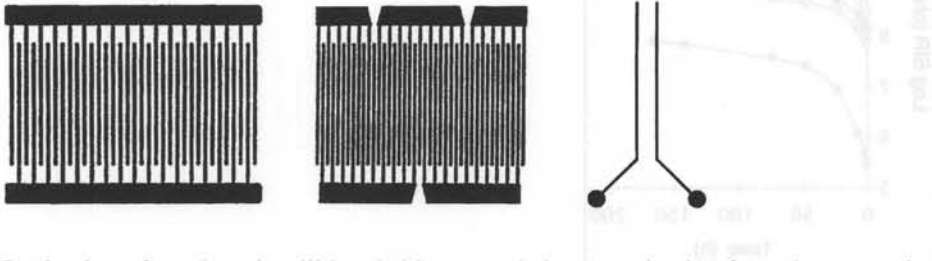


Figure 3. Examples of patterns used for SIR and electrochemical migration testing, showing comb patterns from Test Boards IPC-B-24 and IPC-B-25, and a military Y-pattern

Production of test boards will inevitably cause their contamination from the processing solutions. To minimise this contamination, test boards with bare copper patterns, without any solder mask applied and extensively cleaned, are often used. The effect of a single material, chemical, or process can thereby be evaluated. Passing such a test is mandatory but not sufficient for approval. When various materials and processes are combined, synergistic effects may occur. For example, as shown by Adams et al. [10], polyethylene glycol, which is a common ingredient in fluxes used for HASL, gives a strong synergistic effect when combined with adipic acid, a common ingredient in many no-clean fluxes used for soldering of PBAs (see Table 2).

Figure 4 shows the SIR for printed boards with different surface finishes produced by one printed-board manufacturer using ordinary production processes, including cleaning [44]. Initially, the boards with HASL finish have an about five decades lower SIR than the boards with OSP (organic solderability preservative) or ENIG (electroless nickel/immersion gold). This clearly demonstrates the detrimental effect of flux residues from hot-air solder levelling on SIR. Due to the impossibility of cleaning away all residues [20, 24], HASL boards will always have rather low SIR. However, the impact of flux residues from different printed-board manufacturers may vary greatly. Figure 4 shows the results from SIR measurements on HASL printed boards from four different manufacturers [20]. Initially, the SIR values differ by three decades between the best and the worst boards. With two hours of extensive cleaning using ultrasonic agitation of boards from Manufacturer A, the SIR was improved by about two decades, i.e. to about the same value as OSP and ENIG boards if the SIR is adjusted for the difference in test temperature. However, when the board went through a soldering process without any added flux, the SIR decreased almost back to the original value, indicating that absorbed flux residues diffused up to the surface during the heat treatment [20].

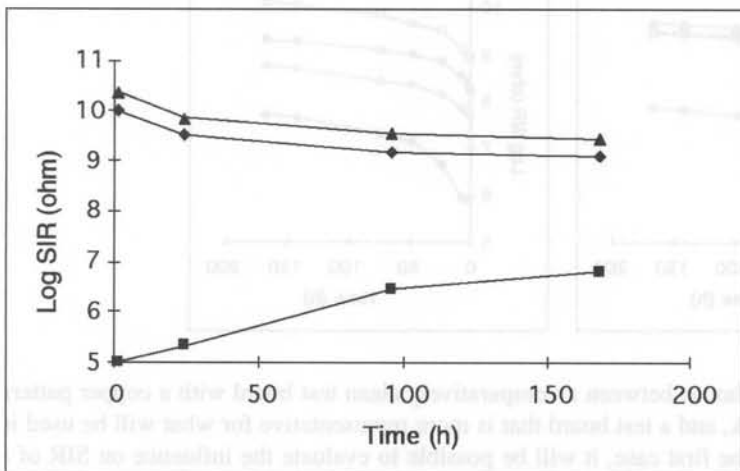
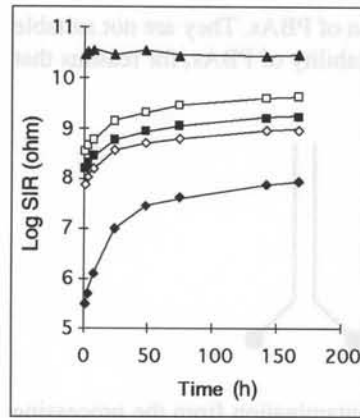


Figure 4. Surface insulation resistance measured during an electrochemical migration test performed at 85°C, 85% RH and 5 V DC polarisation and measurement voltage for comb patterns on printed boards with the following surface finishes: HASL (■), ENIG (▲) and OSP (◆)

Figure 5. Surface insulation resistance measured during an electrochemical migration test performed at 65°C, 85% RH and 5 V DC polarisation and measurement voltage for comb patterns on HASL printed boards from four printed-board manufacturers: A (■), B (□), C (◆), and D (◇). For reference, results are given for a board from Manufacturer A cleaned for 2 h before the test (▲)



The cleanliness of a printed board will affect the SIR of a PBA whether or not cleaning of the PBA is performed. Figure 6 shows the results from measuring SIR on PBAs soldered at some assemblers having different types of soldering processes using printed boards from the four manufacturers presented in Figure 5 [20]. The variation in SIR of the printed boards was reflected in the SIR of the assemblies independent of the type of flux and cleaning process used, although the absolute measured SIR values varied considerably for the various assemblies. As expected, rosin residues were found to have a very benign influence on SIR even if cleaning was performed.

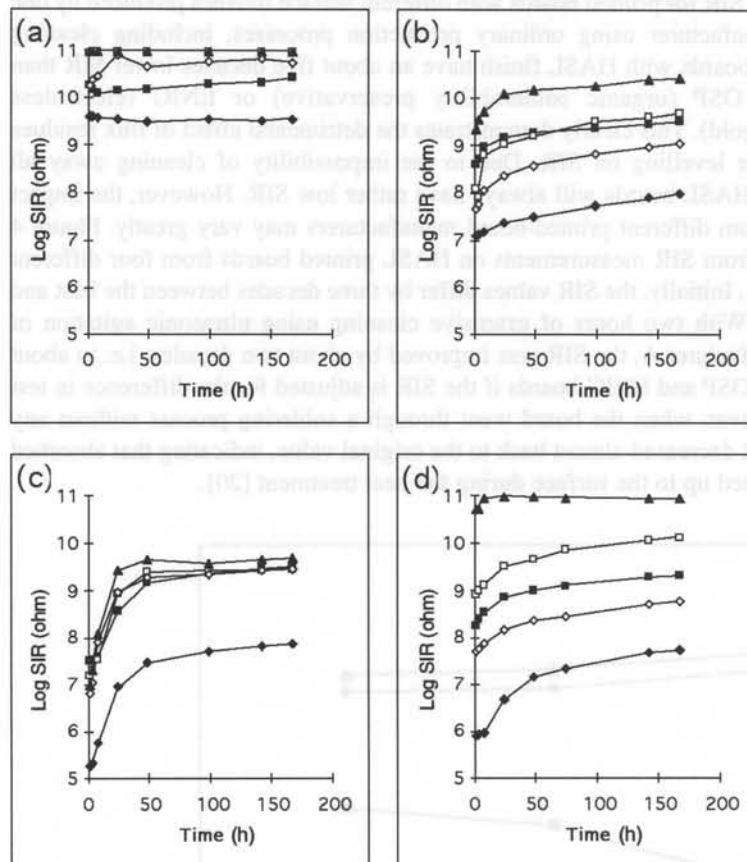


Figure 6. Surface insulation resistance measured between solder joints to wave-soldered SO28 components on assemblies manufactured by three different companies using their ordinary processes: RA flux and solvent cleaning (a), low-solid, rosin-based flux and no cleaning (b), water-soluble flux and cleaning using pure water (c), and non-soldered reference boards (d). The printed boards used were produced by Manufacturers A (■), B (□), C (◆), D (◇) and, as reference, boards from Manufacturer A cleaned for 2 hours before soldering (▲)

Thus, one has to choose between a comparatively clean test board with a copper pattern and no solder mask, and a test board that is more representative for what will be used in true products. In the first case, it will be possible to evaluate the influence on SIR of a single material, chemical, or process and to compare with data from other investigations, but it will not be possible to draw reliable conclusions regarding the SIR of the final product. In the second case, the results will show the SIR properties of the final product, but they will only be representative for the combination of materials, chemicals, and

processes used for producing the test boards. Hence, the primary field of application for 'clean' test boards is the screening of materials and processes, although it is not desirable that material qualification be limited to that. This may lead to approval of many materials that later will be found to cause unreliable products. Test boards used for final verification need to be representative of the materials and processes used for production of the final product. Consequently, final verification can only be done for specific applications. This is reflected in the IPC's standard for PBAs: J-STD-001D, Appendix C, Material and Process Compatibility Testing, which specifies that the test vehicle should represent the substrate materials, assembly materials, and fabrication processes used in production [45].

However, comb patterns are not appropriate for such evaluations. The locations most contaminated and difficult to clean on a PBA are areas between solder joints and beneath components. Hence, to truly represent manufacturing processes, it is necessary to mount components on the test boards. By mounting dummy components without any electrical connections inside (open circuit) on test patterns of the type shown in Figure 7, SIR can be measured between solder joints to soldered components. In fact, a test board can be designed and produced exactly in the same way as production PBAs. By placing comb patterns under components, SIR can also be checked at such locations, although it must be pointed out that the comb pattern can have some influence on the efficiency of a cleaning process for components with small stand-offs. Ceramic chip capacitors and resistors usually have a very small stand-off and it is difficult to clean beneath them, especially if they are glued to the boards using glue dots at the sides of the components. The best way to measure SIR beneath this type of component is to use a test pattern as shown in Figure 7, but then only chip capacitors can be used. After the voltage has been applied and the capacitors have been charged, the SIR can be measured.

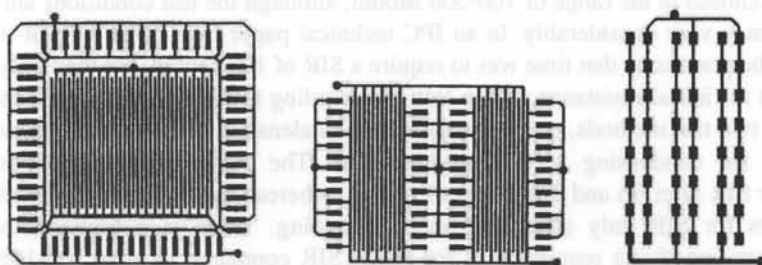


Figure 7. Test patterns for measuring SIR between solder joints to a PLCC68 component, two SO28 components, and 0805 chip capacitors. Comb patterns are located beneath the PLCC and SO components

### Acceptance Criteria

Even if the knowledge of how to adequately test SIR and electrochemical migration is far from satisfactory, much has been written about the subject compared to what has been written about acceptance criteria. Most standards have numerical requirements, which usually differ between the various standards. Even if the numerical requirement is the same, the test conditions normally vary which, in practice, means that the requirements differ. Usually, there is a requirement that SIR shall not be less than a certain value. Some electrochemical migration tests also require that the SIR not decrease more than one decade during the test. Due to the experience that dendrites normally form without any impact on SIR, some later standards also have visual requirements regarding the presence of corrosion, dendritic formation, and mealing of conformal coating.

As already discussed, there are two aspects on SIR requirements. Firstly, a certain level of SIR is required for the functioning of a PBA. Secondly, a certain level is believed to be needed in order to avoid electrochemical migration, although this is not well verified. There is no standard that makes any distinction between these two aspects, and thus it is not clear to which aspect the requirements refer. Obviously, in the first case, the required SIR may differ considerably between different products even if they will be used in the same environment and, therefore, acceptance criteria have to be application-specific. The resistance required to avoid dendrite formation will probably be less dependent on the function, i.e. in the second case the required SIR will be the same for products that will

be used in similar environments. However, since humidity and contamination levels in the field environment will have a very large impact on both SIR and electrochemical migration, it will be necessary to have different acceptance criteria for various types of applications also when considering the risk for electrochemical migration.

The IPC standard for soldered PBAs, J-STD-001D [45], is a good example of the many uncertainties that arise when one tries to get a grip on the basis for the acceptance requirement. In Appendix C, Material and Process Compatibility Testing, the acceptance requirement is that the SIR value shall be not less than 100 Mohm. The test pattern must provide for SIR testing similar to that of the IPC-B-36 circuitry, which is a leadless ceramic chip carrier with 68 castellations (open circuit) mounted on a pattern with interdigitated solder lands. Components of the type to be soldered in production representative of the 'hardest-to-clean' configurations shall be included on the PBA. What then does similar circuitry mean? The most likely interpretation is open-circuit components mounted on patterns with interdigitated solder lands. However, any change in the number of connections and/or pitch for a test pattern will, of course, influence the measured SIR and the acceptance criterion ought to be adjusted, but nothing is mentioned in the standard about how this should be dealt with. Ideally, this can be handled using the ohms/square concept [3]. If SIR is assumed to obey Ohm's law, the SIR for a square will be independent of the size of the square. By calculating the total number of squares in the test pattern and then dividing the measured SIR by the total number of squares, the SIR can be expressed in terms of ohms/square. Thus, by presenting the results from a SIR measurement as ohms/square, the SIR will be independent of the test-pattern geometry. In reality, SIR does not obey Ohm's law, so this concept should be used with caution [35,46].

The reason for setting the acceptance criterion to 100 Mohm in J-STD-001D is not given. When looking at previous IPC standards, it can be noted that acceptance criteria for SIR tests are usually chosen in the range of 100-500 Mohm, although the test conditions and the test pattern may vary considerably. In an IPC technical paper from 1985 [48], it is mentioned that the practice at that time was to require a SIR of 100 Mohm, but then only as a requirement for initial resistance. When testing according to J-STD-001D, one can choose between two test methods, one static for a non-condensing service environment and one cyclic for condensing service environments. The static test method has requirements for SIR after 96 and 168 hours of testing, whereas the cyclic test method has requirements for SIR only after 160 hours of testing. There is a tremendous difference between specifying requirements for initial SIR compared to after 100-168 hours of testing. As shown by Chan [36], SIR may increase several decades on a contaminated board during a test period of 100 hours, and approach values of a clean board. Since no information is given on the background to the SIR requirements in J-STD-001D, it is not possible to draw any conclusion about the relevance of the requirements, nor to justify the disregarding of initial SIR values.

A good example of a function-based, application-specific requirement has been described by Mason [39]. The company's most sensitive circuit had a device with 0.5 pA maximum offset current. Two guard tracks are used, each 25 mm long and separated from the guarded track by 1.25 mm. The potential between guarding and guarded tracks was 15 mV. Hence, the minimum required SIR was  $3 \times 10^{10}$  ohms, or  $1.2 \times 10^{12}$  ohms per square.

## 5 Experimental Work

### 5.1 Layout of the Test Vehicle

A specially designed test board was used for the evaluation (see Figure 8). It is a double-sided polyimide board with fused tin-lead. The boards were manufactured at an ESA-approved supplier with qualified materials, processes and quality standards [6]. They were mounted with the following types of components: QFP144, PLCC68, SO28, PLCC20, and ceramic-chip capacitors (0603 and 0805). BGA components were not mounted on the boards, as at this time none of the assemblers soldering the test boards

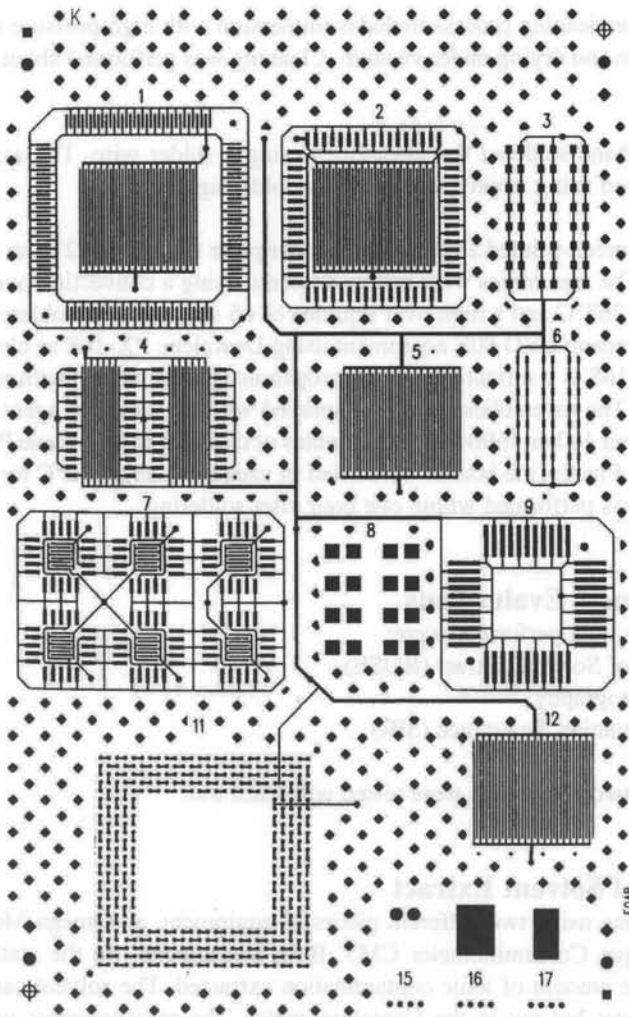


Figure 8. Layout of the test board. It has footprints for mounting of QFP144 (Pos. 1), PLCC68 (Pos. 2), chip capacitors 0805 (Pos. 3), SO28 (Pos. 4), chip capacitors 0603 (Pos. 6), PLCC20 (Pos. 7), surface-mounted connector (Pos. 9), and BGA520 (Pos. 11)

are approved by ESA to perform this process. The solder lands in the footprints are connected to make it possible to measure the SIR between the solder joints under the condition that the QFP, PLCC, and SO components used are dummies with open circuit. The SIR can also be measured for free comb patterns and comb patterns beneath components having leads.

## 5.2 Soldering of Test Vehicles

Test boards and components were sent to four different space-electronics manufacturers for soldering. The test boards were soldered using their ordinary soldering and cleaning processes. Below is a description of the materials and processes used by the four assemblers.

**Assembler A** dispensed a rosin-based solder paste (Microprint P2004 from Warton Metcals) on the PCBs. Soldering was done using a vapour-phase oven with a peak temperature of 230°C and a time over liquidus of 65 seconds. A few dry joints were reworked on each PCB. The assemblies were hand-cleaned during rework and were then immersion-cleaned after rework. In both cases, Librasol 141 ME was used as cleaning agent. Rework and final cleaning were performed within 1 hour and 45 minutes after soldering.

**Assembler B** screen-printed a rosin-based solder paste (Opti-Line 9086 from Alpha Metals) on the PCBs. The assemblies were reflow-soldered using a convection oven with a peak temperature of 215°C and a time over liquidus of 70 seconds. Cleaning was performed in Amsonic EVD 80 equipment using Topklean EL-20D from Avantec as cleaning agent. Topklean EL-20D is an organic solvent with a boiling point of

170-180°C. The cleaning process includes immersion with high-pressure spray, spray in air, vapour rinse and drying under vacuum. Cleaning was performed about 18 hours after soldering.

**Assembler C** hand-soldered the assemblies using a solder wire. The assemblies were manually cleaned using isopropanol just after soldering.

**Assembler D** screen-printed a rosin-based solder paste (NC-SMQ92 from Indium Corp.) on the PCBs. The assemblies were reflow-soldered using a convection oven with a peak temperature of 265°C and a time over liquidus of 65 seconds. The soldered boards were cleaned in a Branson EVD 80E equipment using Dowclene PX-16S as cleaning solvent. Dowclene PX-16S is a mixture of alkoxypropanols (glycol ethers) with a boiling range of 170-193°C. The assemblies were first sprayed with the cleaning agent for 6 minutes at 55°C and about 10 bar, followed by 4 minutes of cleaning in Dowclene PX-16S vapour at about 70°C. Finally, the boards were dried in vacuum at about 70°C for 12.5 minutes. The cleaning was performed within one hour after soldering.

### 5.3 Cleanliness Evaluations

The cleanliness tests performed were:

- Resistivity of Solvent Extract (ROSE).
- Ion chromatography.
- Surface Insulation Resistance (SIR).

In most cases, two test boards were tested with each test.

#### Resistivity of Solvent Extract

Testing was done using two different pieces of equipment, an Omega Meter 600 SMD and a Protonique Contaminometer CM5. Both instruments use the static method for determining the amount of ionic contamination extracted. The solvent can be heated in the Omega Meter but not in the Contaminometer. The measurements made using the Omega Meter were performed at 42°C, whereas those made using the Contaminometer were performed at ambient conditions (about 25°C). Two assemblies from each assembler were analysed using each instrument. In order to evaluate the impact of the solvent temperature, assemblies from one assembler were also analysed at 21°C using the Omega Meter.

#### Ion Chromatography

In order to extract ionic contamination from the boards for the ion-chromatography analysis, these were put into plastic bags and 50 ml of pure water was poured in, after which they were sealed. The water used was cleaned with a Milli-Q unit to a specific resistivity of >18 Mohm cm. The plastic bags were first put in an ultrasonic unit for fifteen minutes at room temperature. Thereafter, small samples of the extract solution were taken directly from the plastic bags using a syringe. The extraction in the ultrasonic equipment was then continued for another 1 hour and 45 minutes, i.e. 2 hours in total. New samples of the extract solution were taken after the 2 hours. The samples taken from the plastic bags were injected into an ion chromatograph (Dionex 300 with an AN-1 colon from MetaChem and anion suppresser from Sequant). Only anions were analysed.

#### Surface Insulation Resistance

SIR was measured between the solder joints to the various components soldered to the test boards, but also for a free comb pattern and the comb patterns beneath the QFP144, PLCC68, SO28 and PLCC20 components.

The SIR measurements were performed under controlled temperature and humidity conditions of 65°C and 85% RH, respectively. When the test was started, the temperature

in the test cabinet was first increased to 65°C without any added humidity. About ten minutes after the temperature had stabilised at 65°C, the humidity was increased to 85% RH. When the humidity had stabilised, a bias voltage of 5 V DC was applied during a test period of seven days. The SIR was measured after 1, 3, 8, 24, 51, 98, and 168 hours without interrupting the applied bias. That is, the test voltage was 5 V DC with the same polarity as the bias voltage. After the test period of 168 hours, the temperature was increased to 85°C in order to evaluate the impact of temperature on the SIR results. The SIR values at 85°C were taken one and a half hours after the temperature had stabilised.

To enable automatic measurements, the test patterns were connected in parallel during the test. Resistors (10 Mohm) were connected in series with each test pattern. This was done in order to limit the current density if short-circuits formed within the test patterns due to electrochemical migration. As for all automatic measurement systems, some current leakage will inevitably occur in switches, which limits the accuracy of the measured values. For this system, it was shown that SIR values of between 1 and 10.000 Mohm could be measured accurately. At 100 kohm and 100 Gohm, the error could be up to 50%.

All test boards were visually inspected after the SIR test using an optical microscope with up to 40 times magnification.

## 6 Results

### 6.1 Resistivity of Solvent Extract

The results from the ROSE test are presented in Table 1. Due to a hard-disk crash, the assemblies from Assembler C could not be analysed using the Contaminometer. The non-assembled PCBs had a contamination level of less than 0.1  $\mu\text{g}/\text{cm}^2$  NaCl equivalents. The assemblies from Assemblers B and D had a contamination level of about 0.1  $\mu\text{g}/\text{cm}^2$  NaCl equivalents, whereas the assemblies from Assemblers A and C had a contamination level of about 0.3  $\mu\text{g}/\text{cm}^2$  NaCl equivalents. It can be noted that Assembler A had (by mistake) measured the cleanliness before they were tested using the Contaminometer and the Omega Meter, i.e. the cleanliness was measured twice on these assemblies.

Slightly higher values were recorded using the Omega Meter compared with the Contaminometer. Thus the higher solvent temperature used by the Omega Meter seems to increase the amount of ions extracted, but the difference is rather small and the number of assemblies tested is too low to draw a definitive conclusion about the effect of the higher extraction temperature.

Assembler	Contaminometer ( $\mu\text{g}/\text{cm}^2$ )	Omega Meter @ 42°C ( $\mu\text{g}/\text{cm}^2$ )	Omega Meter @ 21°C ( $\mu\text{g}/\text{cm}^2$ )
A*	0.24-0.30	0.15-0.3	
B	About 0.1	About 0.15	
C	-	0.15-0.6	0.3
D	<0.1	About 0.15	
Reference boards	<0.1	<0.1	

Table 3. Results from the resistivity of solvent extract test for the assemblies from the various assemblers

\* Assembler A measured the ionic cleanliness on all assemblies after soldering and cleaning using an Ionograph 500M. The cleanliness varied between 0.00-0.28  $\mu\text{g}/\text{cm}^2$  NaCl equivalents with an average of 0.14  $\mu\text{g}/\text{cm}^2$ .

### 6.2 Ion Chromatography

The results from the ion chromatography measurements are presented in Table 4. The main anions found were formate, chloride, bromide, nitrate and sulphate. The concentrations of these anions were about the same on the assembled boards as on the reference board (printed-circuit boards as received) except for formate, which had almost

twice as high a concentration on the reference boards. Thus, it is likely that most of the contamination found on the assembled boards originates from the manufacturing of the PCBs. Formate, nitrate and sulphate are commonly found in various baths used during the manufacture of PCBs, whereas chloride and bromide are often found in fluxes used for hot-air solder levelling and fusing of PCBs [19, 49]. Rather surprisingly, the amounts of extracted ions were in most cases lower after 2 hours of extraction than after 15 minutes. It was also observed that the extracts become cloudy after 2 hours of extraction time. Possibly, insoluble compounds were formed in the extract during the prolonged extraction time, resulting in lower detected anion concentrations.

Table 4. Results from the ion-chromatography analysis of the assemblies from the various assemblers

Assembler	Formate ( $\mu\text{g}/\text{cm}^2$ )		Chloride ( $\mu\text{g}/\text{cm}^2$ )		Bromide ( $\mu\text{g}/\text{cm}^2$ )		Nitrate ( $\mu\text{g}/\text{cm}^2$ )		Sulphate ( $\mu\text{g}/\text{cm}^2$ )	
	15 min	2 h	15 min	2 h	15 min	2 h	15 min	2 h	15 min	2 h
A	0.35	0.44	0.24	0.24	0.08	0.12	0.10	0.13	0.11	0.09
B	0.44	0.33	0.15	0.13	0.09	0.10	<0.05	<0.05	0.11	0.05
C	0.58	0.31	0.22	0.20	0.08	0.12	0.09	0.09	0.05	0.06
D	0.33	0.18	0.11	0.08	<0.05	0.09	0.09	<0.05	0.12	0.06
Ref.	0.72	0.64	0.18	0.16	<0.05	0.11	<0.05	0.09	0.08	0.06

### 6.3 Surface Insulation Resistance

Figure 9 presents the results from the SIR measurements on the reference boards (printed-circuit boards as received from the PCB manufacturer). The initial SIR varied between 160 Mohm and 1 Gohm and it had increased by about one and a half decades after 168 hours. The drop in SIR at the end of the test was caused by the increase in test temperature from 65°C to 85°C. The temperature increase also caused a decrease in the SIR by about a half to one decade, in accordance with what has previously been reported [3, 37, 38, 39]. The corresponding results for the assemblies from the various manufacturers are presented in Figure 10. Whereas the SIR on the assemblies from Manufacturers B and C is quite similar to that for the reference boards, it is notably higher on the assemblies from Manufacturers A and D, but it also show larger spread in values on these latter assemblies.

In Figures 11 to 13, the results are compared for specific test patterns. In most cases, the registered SIR values are higher on the assembled boards than on the reference boards. For most test patterns, the highest SIR was registered on the assemblies from Manufacturer D, followed by the assemblies from Manufacturer A.

Test Pattern	Reference Board	Manufacturer A	Manufacturer B	Manufacturer C	Manufacturer D
1	>1	>1	>1	>1	>1
2	>1	>1	>1	>1	>1
3	>1	>1	>1	>1	>1



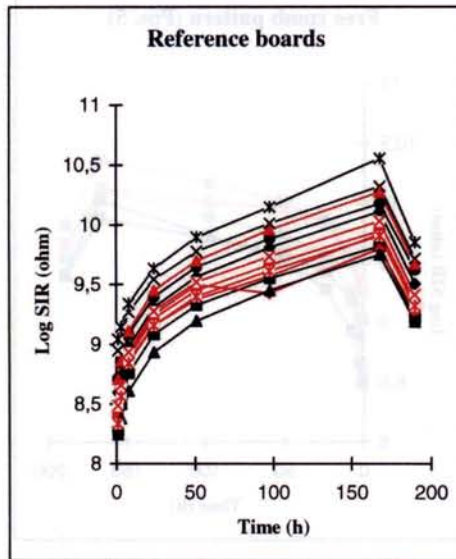


Figure 9. SIR values registered for the various test patterns on the reference boards. Values measured between solder joints are represented with black lines, whereas those measured for comb patterns are represented with red lines

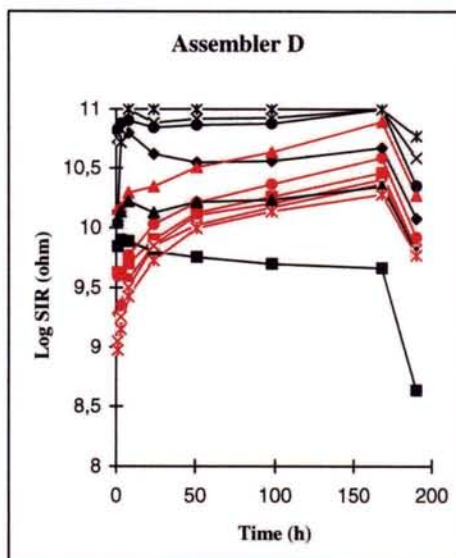
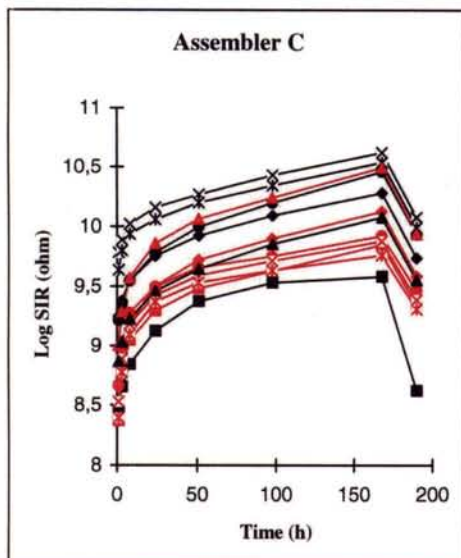
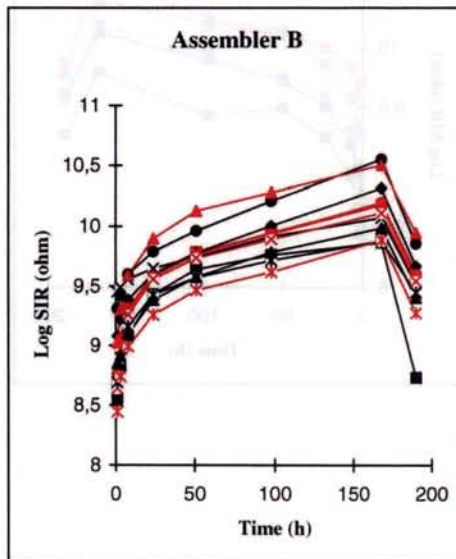
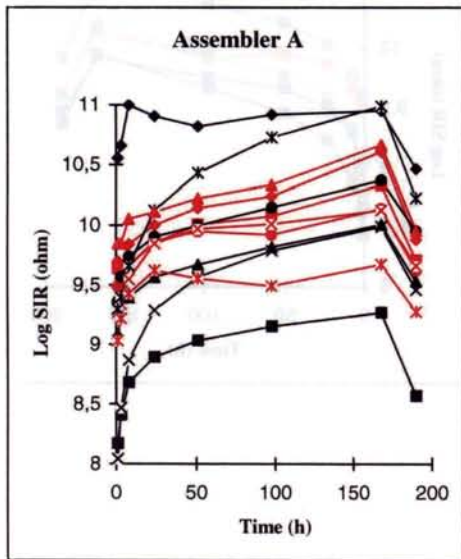


Figure 10. SIR values registered for the various test patterns on the assembled boards. Values measured between solder joints are represented with black lines, whereas those measured for comb patterns are represented with red lines

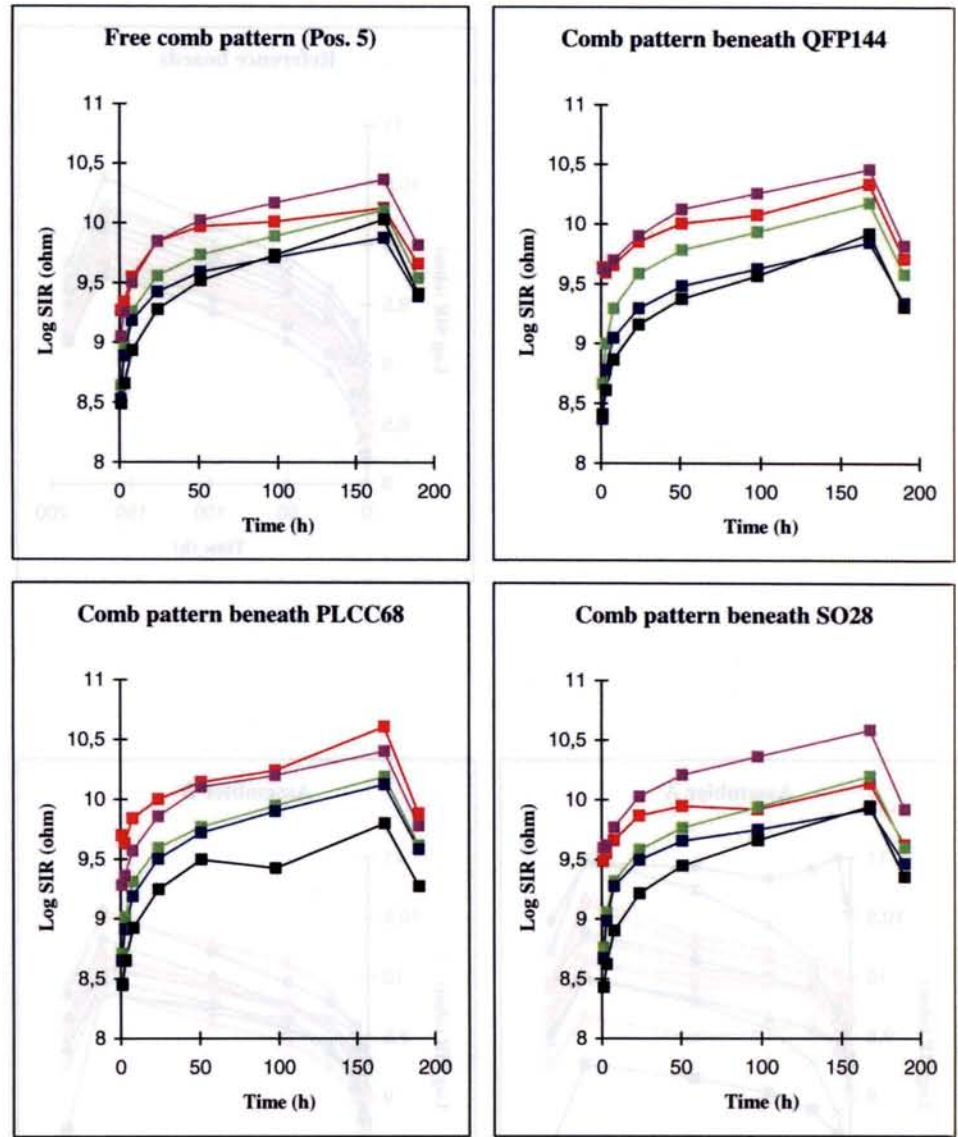


Figure 11. SIR values registered for comb patterns on boards soldered by Assembler A (■), Assembler B (■), Assembler C (■), Assembler D (■), and on reference boards (■)

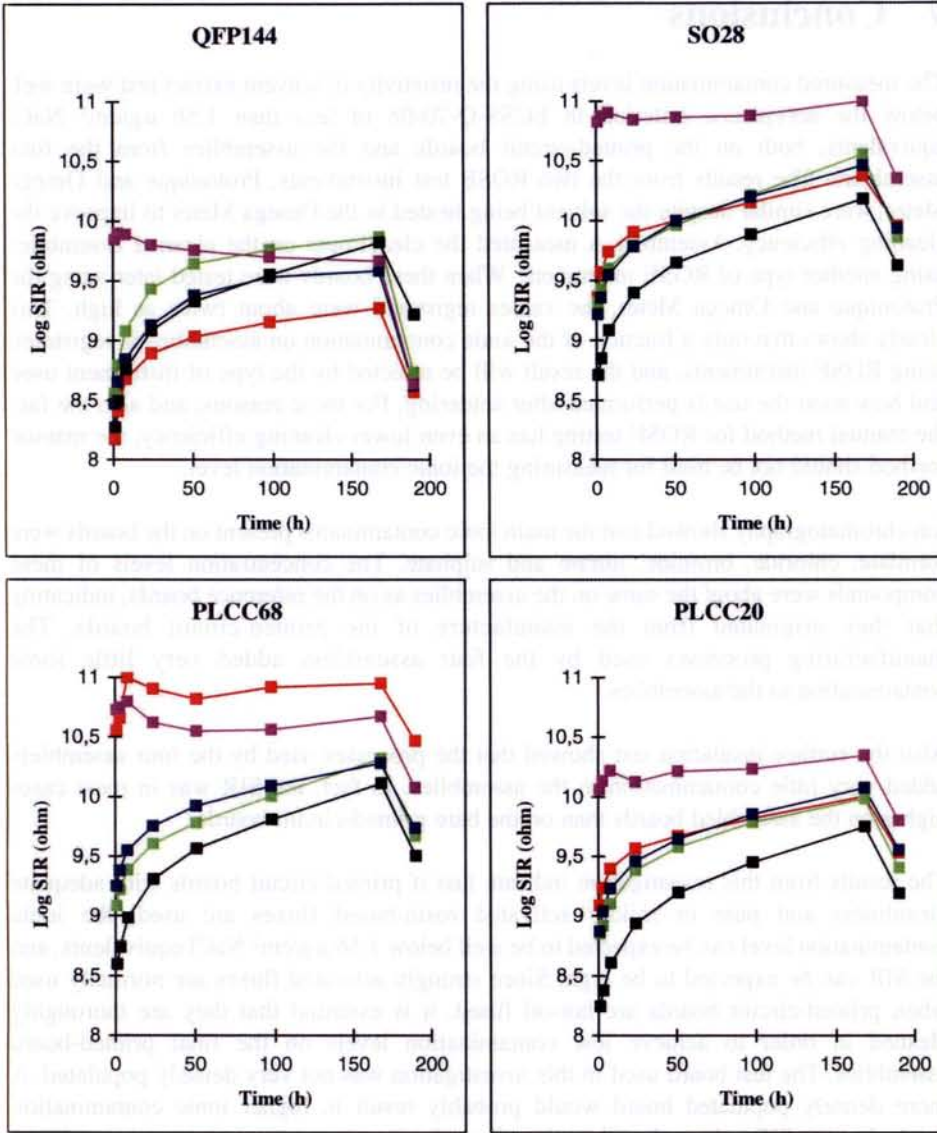


Figure 12. SIR values registered between solder joints to leaded components on boards soldered by Assembler A (■), Assembler B (■), Assembler C (■), Assembler D (■), and on reference boards (■)

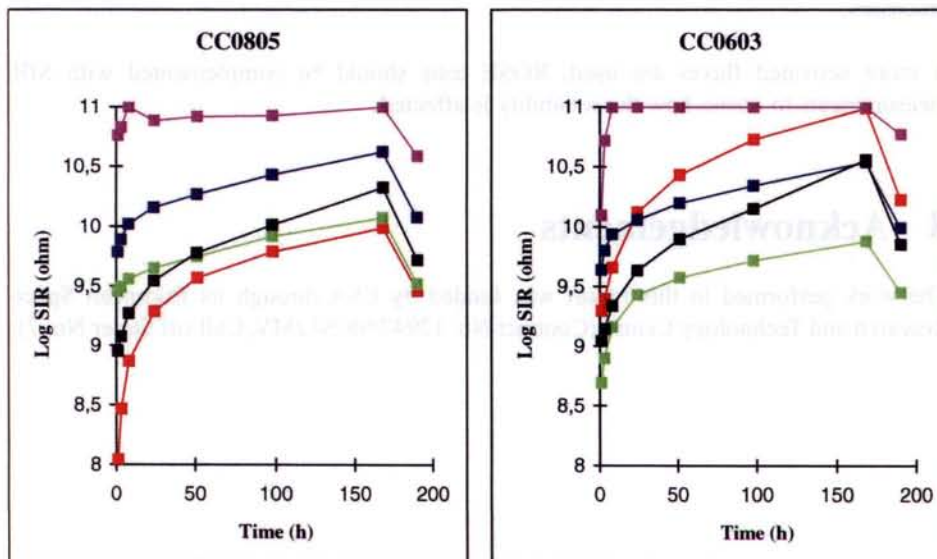


Figure 13. SIR values registered between solder joints to ceramic-chip capacitors on boards soldered by Assembler A (■), Assembler B (■), Assembler C (■), Assembler D (■), and on reference boards (■)

## 7 Conclusions

The measured contamination levels using the resistivity of solvent extract test were well below the acceptance criterion in ECSS-Q-70-08 of less than  $1.56 \mu\text{g}/\text{cm}^2$  NaCl equivalents, both on the printed-circuit boards and the assemblies from the four assemblers. The results from the two ROSE test instruments, Protonique and Omega Meter, were similar despite the solvent being heated in the Omega Meter to improve the cleaning efficiency. Assembler A measured the cleanliness on the cleaned assemblies using another type of ROSE instrument. When these boards were tested later using the Protonique and Omega Meter, the values registered were about twice as high. This clearly shows that only a fraction of the ionic contamination on assemblies is registered using ROSE instruments, and the result will be affected by the type of instrument used and how soon the test is performed after soldering. For these reasons, and also the fact the manual method for ROSE testing has an even lower cleaning efficiency, the manual method should not be used for measuring the ionic contamination level.

Ion chromatography showed that the main ionic contaminants present on the boards were formiate, chloride, bromide, nitrate and sulphate. The concentration levels of these compounds were about the same on the assemblies as on the reference boards, indicating that they originated from the manufacture of the printed-circuit boards. The manufacturing processes used by the four assemblers added very little ionic contamination to the assemblies.

Also the surface-insulation test showed that the processes used by the four assemblers added very little contamination to the assemblies. In fact, the SIR was in most cases higher on the assembled boards than on the bare printed-circuit boards.

The results from this investigation indicate that if printed-circuit boards with adequate cleanliness and pure or mildly activated rosin-based fluxes are used, the ionic contamination level can be expected to be well below  $1.56 \mu\text{g}/\text{cm}^2$  NaCl equivalents, and the SIR can be expected to be high. Since strongly activated fluxes are normally used when printed-circuit boards are hot-oil fused, it is essential that they are thoroughly cleaned in order to achieve low contamination levels on the final printed-board assemblies. The test board used in this investigation was not very densely populated. A more densely populated board would probably result in higher ionic contamination levels, but the SIR values should not be affected.

The results from this investigation do not show how contamination levels around  $1.56 \mu\text{g}/\text{cm}^2$  NaCl equivalents would affect the reliability. However, they would indicate a possible cleanliness problem, since the values registered in this investigation were well below  $1.56 \mu\text{g}/\text{cm}^2$  NaCl equivalents, even when using manual soldering and cleaning processes.

If more activated fluxes are used, ROSE tests should be complemented with SIR measurements to assess how the reliability is affected.

## 8 Acknowledgements

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