

# TECHNICAL REPORT



**Printed board assemblies –  
Part 9: Electrochemical reliability and ionic contamination on printed circuit  
board assemblies for use in automotive applications – Best practices**

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## INTERNATIONAL ELECTROTECHNICAL COMMISSION

## PRINTED BOARD ASSEMBLIES –

**Part 9: Electrochemical reliability and ionic contamination on  
printed circuit board assemblies for use in automotive applications –  
Best practices**

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Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at [www.iec.ch/members\\_experts/refdocs](http://www.iec.ch/members_experts/refdocs). The main document types developed by IEC are described in greater detail at [www.iec.ch/publications](http://www.iec.ch/publications).

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## INTRODUCTION

The document applies to electronic and electromechanical automotive circuit board assemblies. It describes current best practices for dealing with electrochemical reactions like migration or corrosion and ionic contamination on the surface of a printed circuit board as one failure mode under humidity load.

This document is an informative document which serves to illustrate the technically feasible options and provide a basis for customer and supplier agreements. It is not intended to be regarded as a specification or standard.

Related standards are gathered in the Bibliography.

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## PRINTED BOARD ASSEMBLIES –

### Part 9: Electrochemical reliability and ionic contamination on printed circuit board assemblies for use in automotive applications – Best practices

#### 1 Scope

This part of IEC 61191, which is a Technical Report, applies to electronic and electromechanical automotive circuit board assemblies and describes current best practices for dealing with electrochemical reactions like migration or corrosion and ionic contamination on the surface of a circuit board as one failure mode under humidity load. This document deals with the evaluation of materials and manufacturing processes for the manufacturing of electronic assemblies with focus on their reliability under humidity loads. The electrical operation of a device in a humid environment can trigger electrochemical reactions that can lead to short circuits and malfunctions on the assembly. In this context, a large number of terms and methods are mentioned, such as CAF (conductive anodic filament), anodic migration phenomena, dendrite growth, cathodic migration, ROSE (resistivity of solvent extract), ionic contamination, SIR (surface insulation resistance), impedance spectroscopy, etc., which are used and interpreted differently. The aim of the document is to achieve a uniform use of language and to list the possibilities and limitations of common measurement methods. The focus of the document is on the error pattern of electrochemical migration on the surface of assemblies with cathodic formation of dendrites.

Evaluation of different test methods of control units under high humidity load are not part of this document.

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60194-1, *Printed boards design, manufacture and assembly – Vocabulary – Part 1: Common usage in printed board and electronic assembly technologies*

IEC 60194-2, *Printed boards design, manufacture and assembly – Vocabulary – Part 2: Common usage in electronic technologies as well as printed board and electronic assembly technologies*

#### 3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in IEC 60194-1, IEC 60194-2 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

### 3.1 Terms and definitions related to management

#### 3.1.1

##### **design authority**

individual, organization, company, contractually designated authority, or agency responsible for the design of electrical/electronic hardware, having the authority to define variations or restrictions to the requirements of applicable standards, i.e., the originator/custodian of the applicable design standard and the approved or controlled documentation

#### 3.1.2

##### **manufacturer**

individual, organization, or company responsible for the assembly process and verification operations

#### 3.1.3

##### **production part approval process**

###### PPAP

procedure in accordance to IATF 16949 [1] to regulate the sample submission process within the supply chain, primarily used for the series release of new parts

Note 1 to entry: The main objective of the procedure is the regulated start-up assurance with regard to quality and quantity of mass production.

#### 3.1.4

##### **user**

individual, organization, company, or agency responsible for the procurement of electrical/electronic hardware and having the authority to define any variation or restrictions to requirements

EXAMPLE Originator/custodian of the contract detailing the requirements.

### 3.2 Technical terms and definitions

#### 3.2.1

##### **conductive anodic filament**

###### CAF

migration which occurs along the monofilament of reinforcing material such as glass cloth in an inner layer part of a printed wiring board

#### 3.2.2

##### **no-clean**

produced with a no-clean solder material and optimized process parameters throughout the entire process chain (e.g. design, printing, soldering), for which flux residues are usually not critical and removal of these residues is not necessary

Note 1 to entry: There could be additional requirements of customers.

#### 3.2.3

##### **resistivity of solvent extract**

###### ROSE

analytical method to determine the integral contamination load on a CB or CBA causing electrical conductivity

#### 3.2.4

##### **surface insulation resistance**

###### SIR

electrical resistance of an insulating material between a pair of contacts, conductors or grounding devices in various combinations, which is determined under specified environmental and electrical conditions

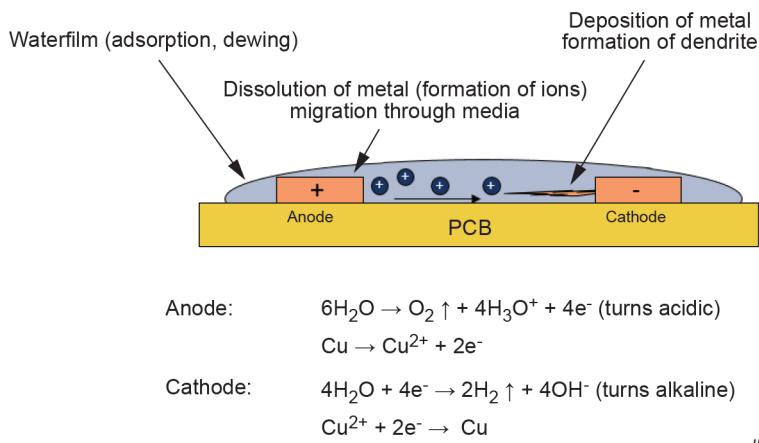
### 3.3 Abbreviated terms

AIT	assembly and interconnect technology
AMP	anodic migration phenomena
CB	circuit board
CBA	circuit board assembly
DI-water	deionized water
ECM	electrochemical migration
ECU	electronic control unit (CBA with housing)
IC	ion chromatography
ICont	ionic contamination
iSn	immersion tin
OSP	organic surface protection
PB	printed circuit board (bare board as delivered by PB manufacturer)
	Note 1 to entry: This abbreviated term is not preferred.
PBA	printed circuit board assembly (unit without housing)
	Note 1 to entry: This abbreviated term is not preferred.
PCB	printed circuit board (bare board as delivered by CB manufacturer)
	Note 1 to entry: This abbreviated term is not preferred.
PCBA	printed circuit board assembly (populated CB without housing)
	Note 1 to entry: This abbreviated term is not preferred.
SMD	surface mounted devices
SMT	surface mounting technology
THR	through hole reflow
THT	through hole technology
WOA	weak organic acid

## 4 Failure mode electrochemical migration

### 4.1 Background of electrochemical migration

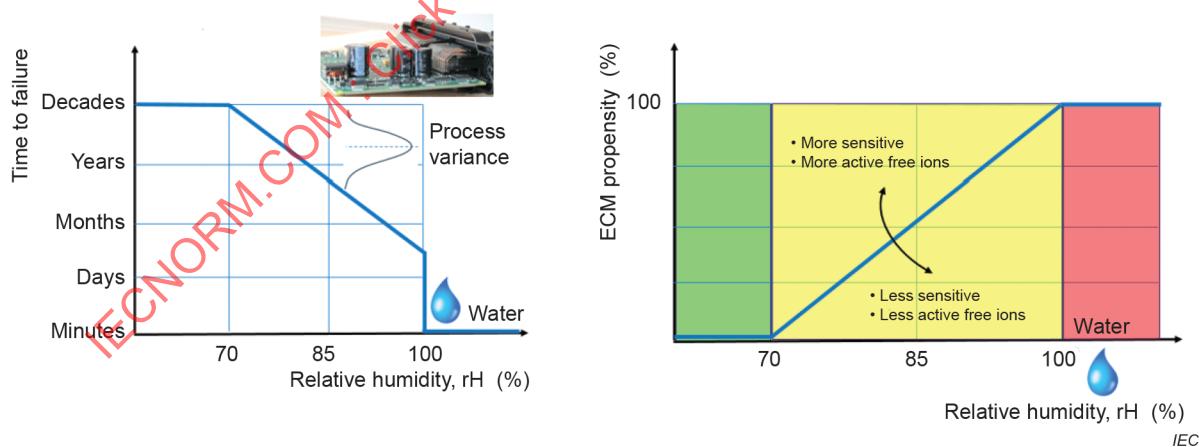
The electrochemical migration on electronic assemblies is understood as the migration of metallic ions such as Ag, Cu, Sn, Ni in a water film on the assembly. The ions are released at the anode (the positive pole of the assembly, e.g. terminal 30), migrate by a diffusion controlled mechanism in the water film to the cathode (the negative pole, e.g. ground, GND) and are deposited there again by reduction with dendrite formation (electrocrystallisation). The dendrite can then grow back towards the anode and create an electrical short circuit. This process can only take place if there is a closed water film between the anode and cathode and if there is a corresponding potential difference. The electrolysis of water always occurs as a reaction, so that a local change in the pH value take place. This can trigger further reactions (e.g. hydrolysis of materials or the formation of poorly soluble metal salts). The processes are shown in Figure 1.



**Figure 1 – Principal reaction mechanism of ECM**

The process of electrochemical migration itself is fast. Dendrite growth occurs within seconds to a few minutes if a sufficiently thick ( $> 50 \mu\text{m}$ ) water film (e.g. droplet formation by direct condensation) is formed on the assembly. Thus, electrochemical migration is not an ageing effect of materials, but is triggered by the event of direct condensation. Classical lifetime laws cannot therefore be applied. With very thin closed water films ( $< 70 \%$  rH with few molecular layers,  $< 50 \text{ nm}$  thickness), this process is significantly slowed down for molecular-structural reasons.

Therefore, ECM is only found if a sufficient amount of water is present locally at a design element having a potential difference ( $> 1,5 \text{ V}$ ). It is also visualized that small deviations in material or processing properties can drastically change the criticality of the system concerning ECM. The principal dependencies were presented on [2]<sup>1</sup> (Figure 2). It illustrates very well the problem that a failure prognosis based on widely used Peck or Lawson models regarding ECM is not possible due to uncertainty of surface conditions and the fact that dewing events do not follow an ageing law.

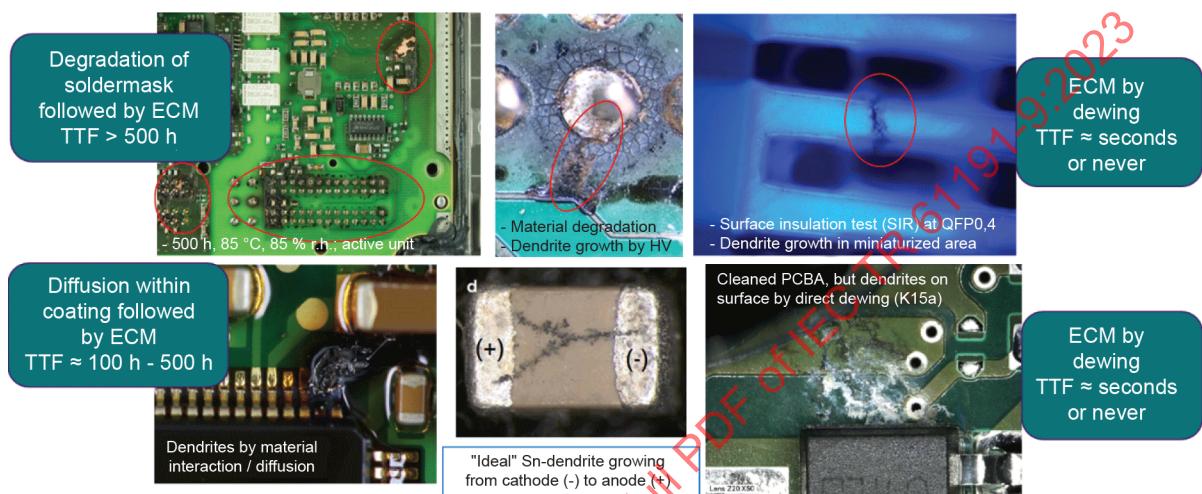


See [2].

**Figure 2 – Uncertainty in local conditions determines ECM failures**

<sup>1</sup> Numbers in square brackets refer to the Bibliography.

The ECM error pattern can also occur with a time delay due to decomposition reactions or diffusion processes. For example, ECM defects below a solder resist or within a conformal coating only occur after sufficient saturation of the materials with water but are significantly delayed by the slow diffusion processes within a polymer. Slow material decompositions (e.g. hydrolysis of polymers by the local change of the pH-value) with an accompanying change of their function (e.g. loss of the insulating effect of a solder resist) also belong to this group. In these cases, ECM will only occur after extended periods of exposure to moisture (weeks to months in high humidity tests). Those findings are illustrated in Figure 3. For degradation or diffusion processes as rate determining step complex, models for failure prognosis could be derived in contrast to the condensation case. However, those models are merely system specific and cannot be transferred to different constructions and materials.



See [3]

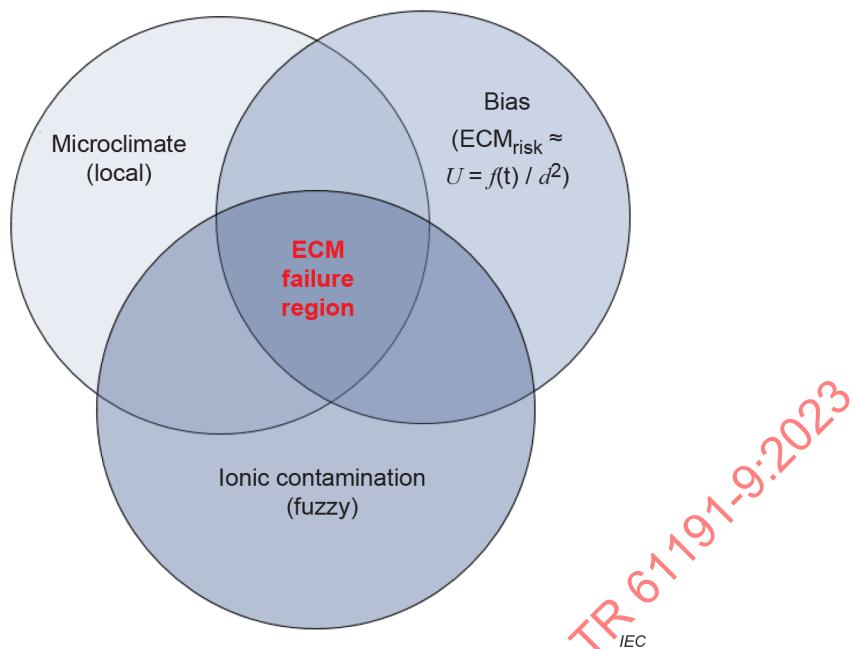
NOTE Same failure mode but different reasons

**Figure 3 – Occurrence of ECM failures during humidity tests**

In all tests and evaluations of assemblies operated in humid environment, it needs to be minded that humidity can only be accelerated to a limited extent. An acceleration factor cannot be derived in most cases. It needs always to be noted that the failure mechanism is not changed by any test condition or acceleration approaches in a way that will not appear in the intended end-use environment of the product. IEC 60068-3-4:2001 [4] already clearly emphasizes this special feature for electrochemical failure mechanisms where humidity is necessary.

#### 4.2 Complexity of electrochemical migration

The error pattern of electrochemical migration on assemblies is complex. Its occurrence depends on various factors, which are shown in the VENN diagram (Figure 4). The attempt to summarize the complex relationships in a single, easily accessible measured quantity, such as only the reduction of ionic contamination, was therefore incorrect and is a frequent cause of misinterpretations.



**Figure 4 – VENN diagram showing the factors influencing ECM**

In order to understand the error pattern of electrochemical migration in its totality, the 3 following main influencing factors need to be analyzed and understood in more detail.

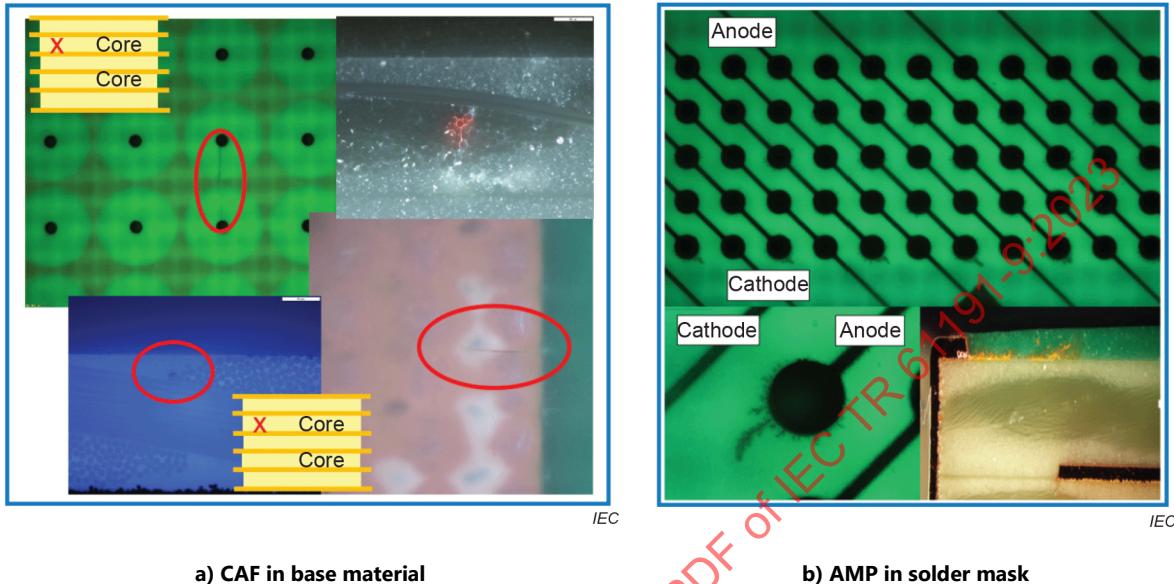
- Bias translated by voltage  $U$  and operation time  $t$  when  $U$  is applied as well as distances  $d$  between metals with different potential, where the following relationship applies to the risk of electrochemical migration  $P_{ECM} \approx U \times f(t) / d^2$ .
- Microclimate translated by local humidity (stress at design element) considering retarded buildup of water path on circuit-board assemblies in a housing, heating-up and dry-out effects, thermal mass of systems.
- Ionic contamination translated by materials properties considering processing, hygroscopic behaviour, degradation of materials, chemical interactions.

For the complex system circuit-board assembly with a multitude of materials and interactions, the measurement of the SIR value in the context of material characterizations as well as active humidity testing of ECUs currently represents the most reliable method for evaluating the electrochemical reliability of an assembly. The measurement of ionic contamination alone does not provide any information about the reliability of an assembly in the intended end-user environment. In 2017, IPC took this into account and eliminated a historical definition with a limit of  $1,56 \mu\text{g}/\text{cm}^2$  NaCl equivalent for cleaned assemblies in IPC-J-STD-001 [5] starting with Amendment 1 to Rev G [6]. A more detailed background is given by the whitepaper IPC-WP-019 [7].

#### 4.3 Conductive anodic filament (CAF) and anodic migration phenomena (AMP)

CAF and AMP belong to degradation mechanisms in CBs that are also triggered by electrochemical processes. In both cases, dendrite-like structures occur, but unlike classical dendrite growth, they propagate from the anode toward the cathode and consist of semiconducting salts. In the CAF failure case, the electrochemical degradation mechanism occurs preferentially along the glass fibre in the epoxy glass fibre composite of a printed circuit board.

In the AMP failure case, the electrochemical degradation mechanism occurs preferentially in HV application in the bulk phase of polymers such as the solder mask. In both cases, these are slow processes with material degradation that can be captured by classical lifetime model approaches. Typical occurrence is shown in Figure 5. The mechanisms, analytical methods, or derivation of lifetime laws for CAF and AMP are not discussed in detail in this document. Details are given in [8] and [9].

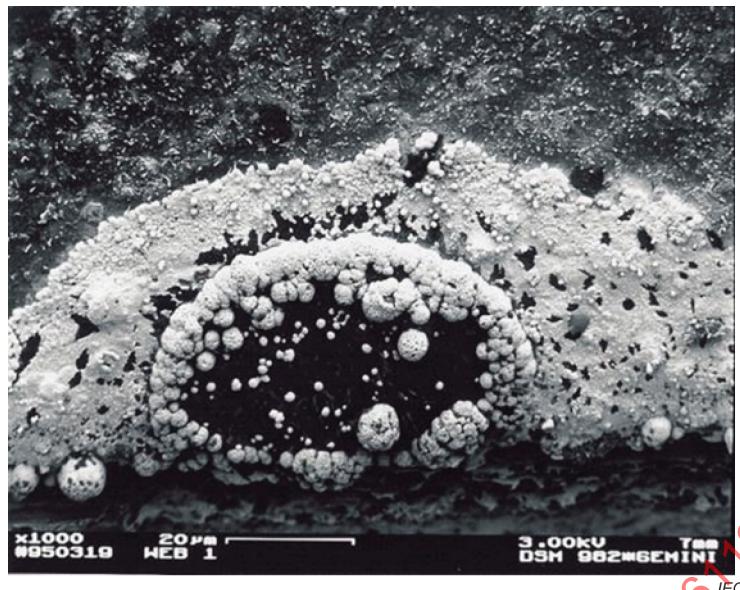


See [8]

**Figure 5 – Occurrence of CAF and AMP**

#### 4.4 Creep corrosion

In addition to classical electrochemical migration with dendrite growth, another contamination-related failure mechanism is creep corrosion (Figure 6). Creep corrosion primarily requires susceptible metallization or final finishes. Ag is the most susceptible, followed by Cu and Ni. A moisture film, well below the dewing point, is enough to trigger the slow corrosion mechanism. A critical humidity is already reached at 60 % to 70 % rH. In addition, corrosive gases such as nitrogen oxides ( $\text{NO}_x$ ),  $\text{H}_2\text{S}$  and  $\text{CO}_2$  tend to dissolve in the moisture film and promote water adsorption by lowering the necessary so-called Gibbs energy. Contamination by atmospheric pollutants such as  $\text{SO}_x$  or  $\text{NO}_x$  as well as sulfur-containing substances from flux residues, especially from natural doping of rosin, can cause creep corrosion. In very rare cases, halide contamination from the manufacturing process of epoxy materials for CB fabrication also causes creep corrosion of the base copper. Creep corrosion is a slow degradation mechanism that can be captured by metal corrosion models. The mechanisms, analytical methods, or derivation of lifetime laws for creep corrosion are not discussed in detail in this document. Details are given in [10] [11] [12].



**Figure 6 – Creep corrosion caused by corrosive gases**

## 5 Electrochemical migration and relevance of ionic contamination

### 5.1 General aspects

The presence of free ions and also critical ions such as halides or WOA on an assembly can increase the risk of electrochemical migration. On the other hand, the use of substances and materials with technical quality and thus with certain ionic loads is typical for the industry, which does not lead to ECM failures per se. The ionic contamination and its measurement method need therefore to be considered in a differentiated way.

### 5.2 Background of ionic contamination measurement

In order to understand the importance of ionic contamination measurement and their limitations, it is important to know their historical origin. This is described in detail in IPC-5703:2013 [13], Chapter 7, and IPC-TP-1113 [14]. The methodology and conclusions date back to a time when soldering was still done with rosin and ammonium chloride or ammonium bromide. Such assemblies had to be washed to ensure that these highly corrosive substances were safely removed. To monitor the cleaning results, these assemblies were rinsed again with an isopropanol-water mixture after the washing process and the conductivity of this washing solution was determined. In this context, the method ROSE (resistivity of solvent extract) was developed. The conductivity of the solution is converted into a NaCl-equivalent: to this end, the (hypothetical) amount of NaCl on the surface of the assembly, which, upon dissolving, would result in the measured resistivity, is calculated. The measurement result, as expressed by the NaCl equivalent does not indicate that NaCl is actually measured/present on the surface of the assembly. The measured value rather represents the conductivity resulting from the multitude of all ions that can be washed off an assembly by the selected solvent mixture. The correlation to the electrochemical reliability of the assembly can be established by providing objective evidence that a given assembly with a certain amount of soluble ionic contaminants on the surface and with the given layout can be operated safely in a given end-use environment.

The ROSE method was then used as a standard method to check the cleaning process and measurement results were tracked in control charts. Since this is a process control, the absolute measurement values have limited significance and the ROSE method itself was never specified exactly. Different solvent mixtures and analyzers are in use and this does not cause any issues as long as the measuring conditions for a given product remain unchanged. The absolute measurement ROSE values are not important as long as selected measurement conditions are applied unchanged and only changes in the process are observed. Due to a series of

unfortunate circumstances, however, a requirement of  $1,56 \mu\text{g}/\text{cm}^2$  NaCl equivalent after a cleaning process was included in military (e.g. MIL-STD-2000A [15]) and commercial (e.g. IPC-J-STD-001 [5]) standards. However, this value was never correlated with the reliability of assemblies in different end-user environment. In addition, more analysers came on the market over time, local measurement methods for ionic contamination were developed and the method of ion chromatography became more widely used, which led to repeated and controversial discussions about absolute values of the different measurement methods. The IPC reacted to this in 2017 and clarified that a single measurement value such as ROSE  $< 1,56 \mu\text{g}/\text{cm}^2$  is obsolete and does not represent general product reliability (see [7]).

The processes for measuring ionic contamination by ROSE and ion chromatography are shown schematically in Figure 7. The effects of the different extraction methods are highlighted. By chance, ion chromatography has established itself as a worldwide, largely uniform procedure with so-called "bag extraction" at  $80^\circ\text{C}$ , for 1 h and a solvent mixture of 75 Vol % 2-propanol/25 Vol % DI-water. For measured values based on ion chromatography, a certain comparability of numerical values is therefore possible. But also for a discussion about absolute values based on ion chromatography, the correlation to reliability tests and field experiences is necessary.

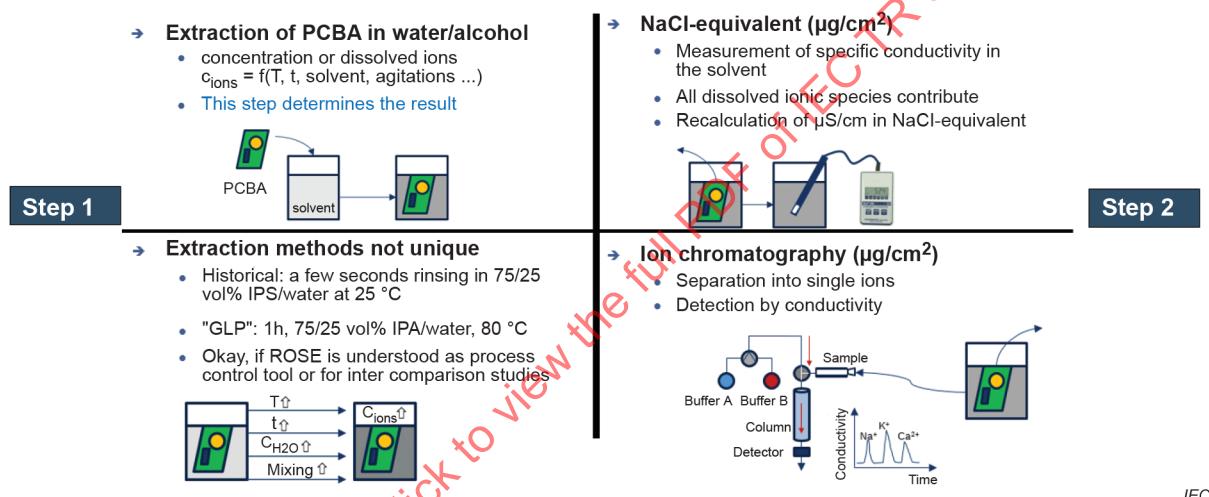


Figure 7 – Ionic contamination measurement

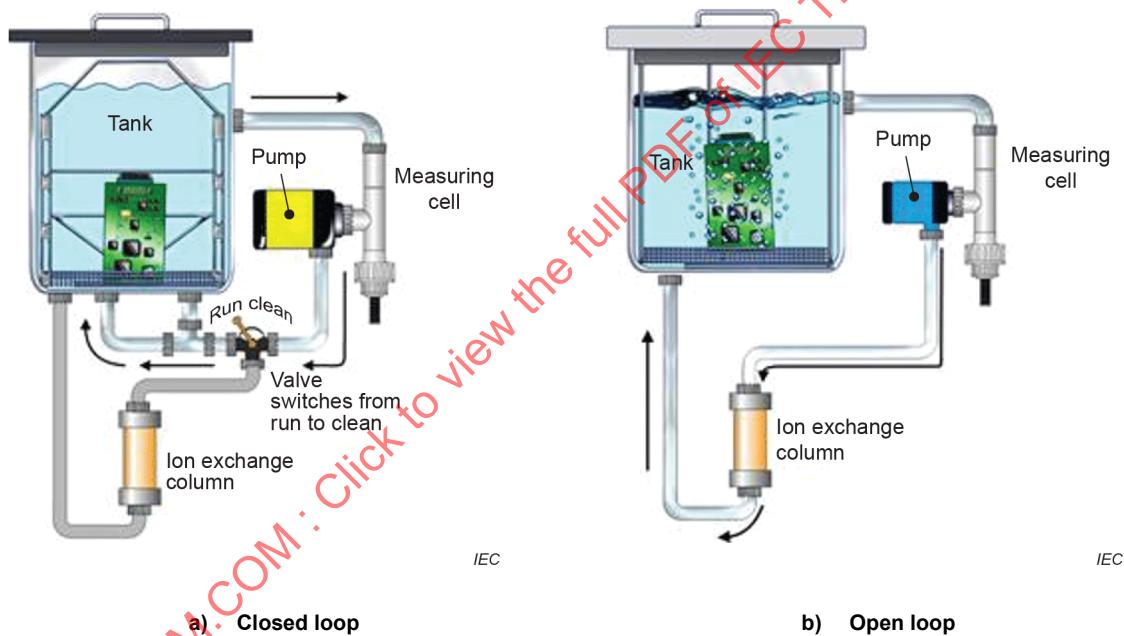
The measuring methods for ionic contamination measurement are described in the following documents.

- IPC-TM-650, method 2.3.25D [17], describes the ROSE measurement for CBs and CBAs; the analysis requires the use of automatic ROSE testers. Solvent mixtures based on 75 Vol % 2-propanol/25 Vol % DI-water or 50 Vol % 2-propanol/50 Vol % DI-water are allowed.
- IPC-TM-650, method 2.3.25.1, [18] describes the ROSE measurement only for CBs; the analysis can be done by bag-extraction and conductivity measurement, but also by automatic ROSE testers. Only the solvent mixture of 75 Vol % 2-propanol/25 Vol % DI-water is allowed.
- IPC-TM-650, method 2.3.26A [19] and method 2.3.26.1A [20], originally described the static and dynamic ROSE measurement methods respectively. These measurement methods will be no longer be referenced, as their contents have now been incorporated into IPC-TM 650, 2.3.25D [17].
- IPC-TM-650, method 2.3.28B, [21] describes the measurement by ion chromatography and lists 21 species to be measured. Preferably, the bag-extraction is carried out with 75 Vol %

2-propanol/25 Vol % DI-water at 80 °C and 1 h extraction time. Deviations from these conditions are allowed, but only if agreed between user and manufacturer.

- IPC-TM-650, method 2.3.28.2, [22] describes the measurement by ion chromatography and lists 17 species to be measured. The bag-extraction is fixed at 80 °C for 1 h, but only allows the use of the solvent mixture 10 Vol % 2-propanol/90 Vol % DI-water. This measuring method will no longer be referenced, the contents of which have now been included in IPC-TM-650, 2.3.28B [21].

In order to overcome the historic misunderstandings about the cleanliness of assemblies and the associated term ROSE, the IEC produced IEC 61189-5-504:2020 [23], in which the term ROSE was replaced by PICT. PICT stands for "process ionic contamination testing" to make the original purpose of the ROSE measurement method clearer. IEC 61189-5-504:2020 [23] also explains two basic design options of ROSE testers. For this purpose, the terms "open loop system" were introduced, which are called "dynamic method" in IPC documents, and "closed loop system", which are called "static method" in IPC documents. These two fundamentally different construction principles already have an influence on the absolute measured value as NaCl equivalent. Figure 8 illustrates the two principal operation modes of ROSE testers. More details on the operation modes and measurement system capability are provided in IEC 61189-5-504:2020, 6.6 and Clause 7 [23].



NOTE Operation mode influences the measured value of NaCl equivalent (see [23]).

**Figure 8 – Principal operation mode (fluid flow) of ROSE**

Readings of NaCl equivalent derived by other methods or with different testers, available on the market, cannot be compared. Comparison is not possible as the way of extraction is different in all testers (solvent, agitation, time, heating). All attempts to do correlation studies or attempts by using correction factors between different ROSE testers failed (IPC-TR-583:1995 [24]).

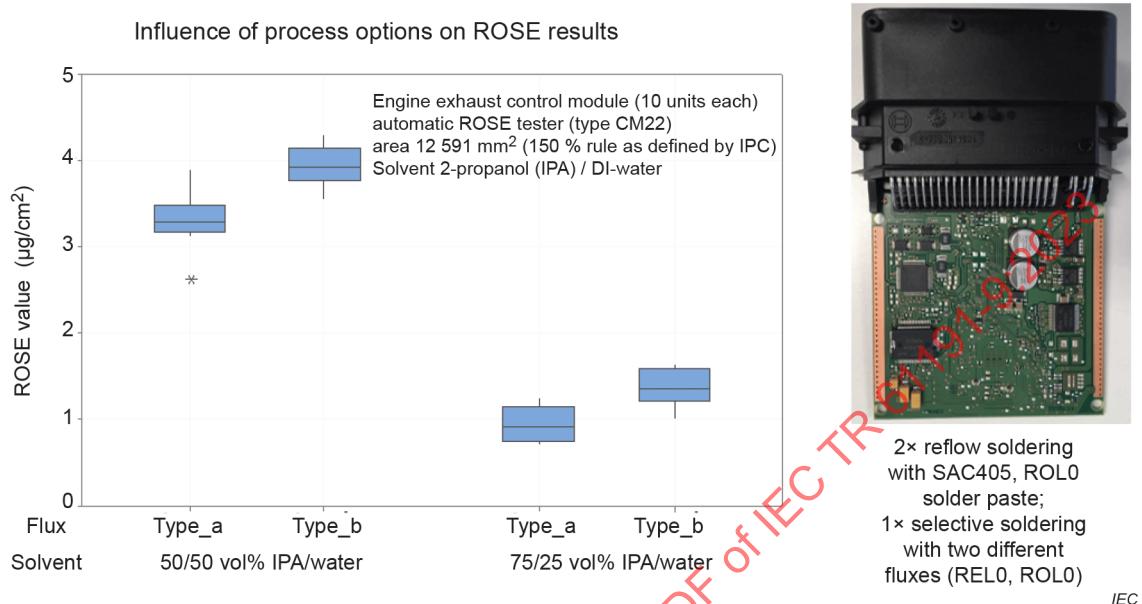
### 5.3 Restrictions and limitations of ionic contamination measurement for no-clean assemblies

#### 5.3.1 Factors determining the result

As described in 5.2, the selection of the extraction and measurement methods based on automatic ROSE testers, bag extraction with dip-probe method or local extractions significantly affect the measurement results for ionic contamination.

### 5.3.2 Influence by solvent on measurement of no-clean assemblies

The effect of different solvent mixtures on the measured ROSE values of no-clean assemblies is shown in Figure 9 and regarding bromide, as an example of an element from ion chromatography, is shown in Figure 10.



NOTE The measurement alone does not predict reliability for no-clean assemblies (see [16]).

**Figure 9 – Effect of solvent composition on the obtained ROSE results**



NOTE The measurement alone does not predict reliability (see [16]).

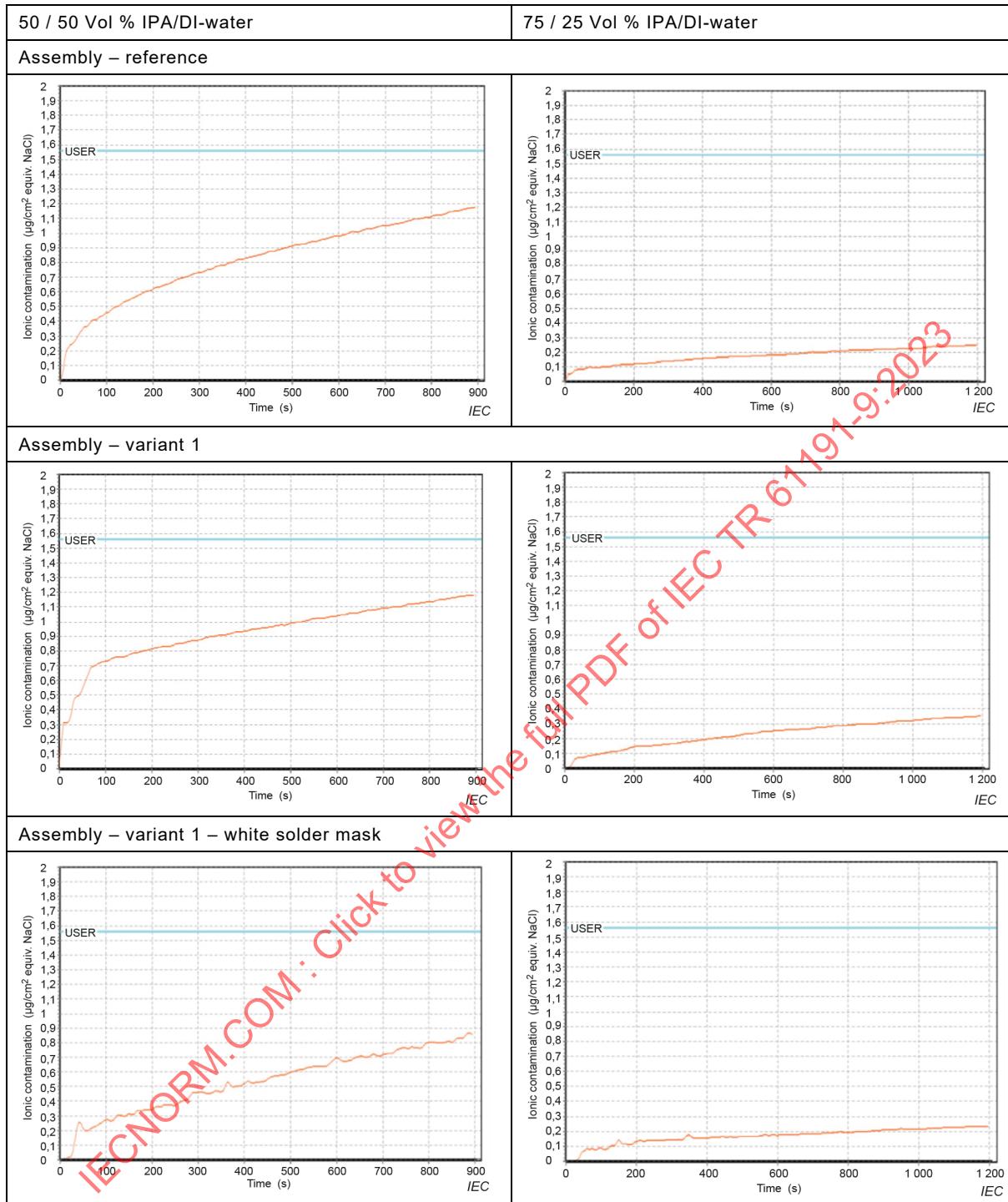
**Figure 10 – Effect of solvent composition on the obtained ion chromatography result**

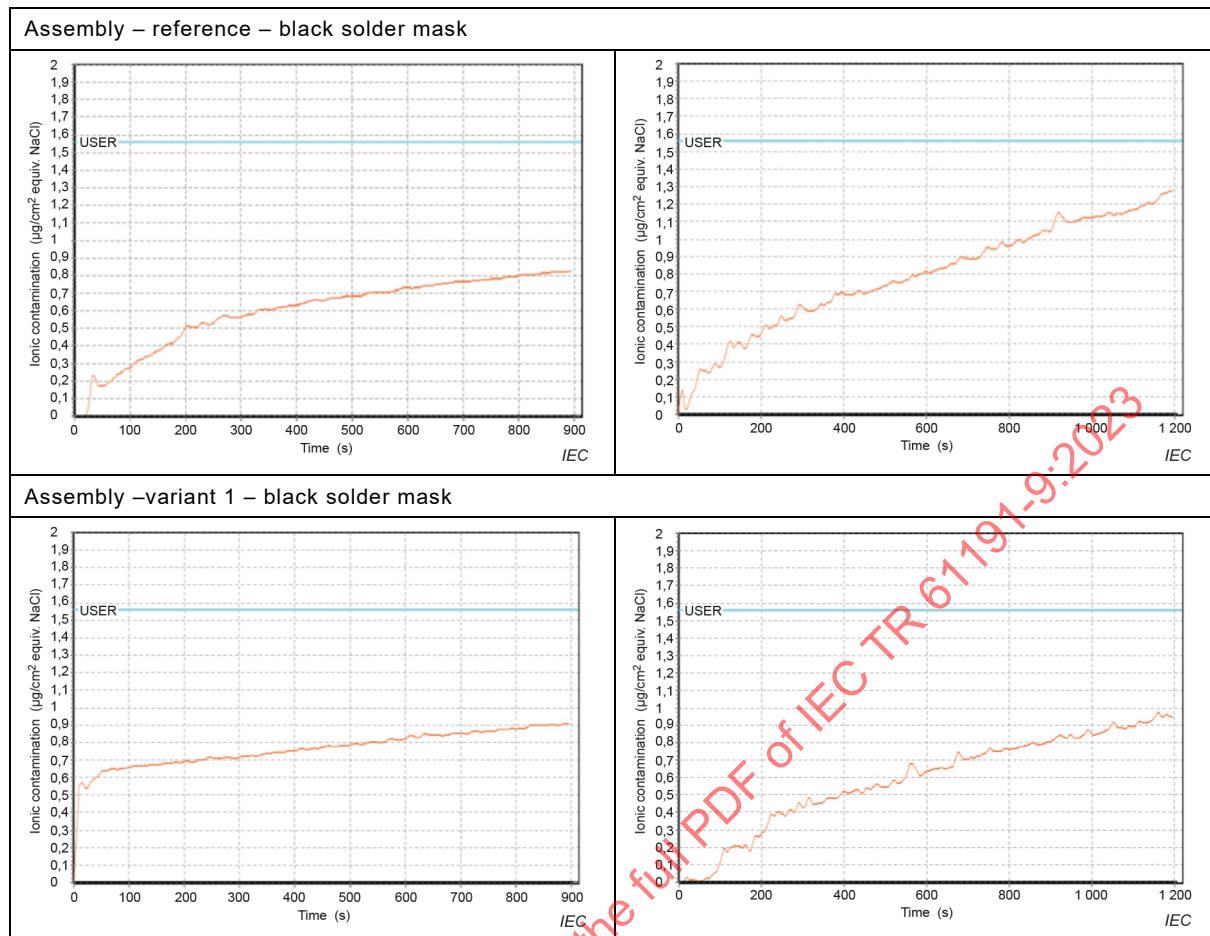
It can be seen that the absolute values are significantly different for given assemblies from a production batch if variations in the measurement methods (here: solvent composition) are considered. Therefore, the absolute value alone cannot give an indication of the reliability of the assembly in the end-user environment. The significant differences here are only due to the measurement method, the solvent mixture selected and its destructive effect on materials (e.g. resin matrix). The link to a moisture robustness of the product is missing and needs therefore to be provided separately for an evaluation of the product reliability.

Figure 11 provides another example showing the influence of the solvent mixture on the measurement result of the ionic contamination. Therefore, there are always discussions about which of the commonly used solvent mixtures, such as 75 vol % 2-propanol/25 vol % DI-water or 50 vol % 2-propanol/50 vol % DI-water, is the "better" one and provides more reliable measured values. However, both solvent mixtures are formally permissible for the automatic ROSE testers. It has not yet been possible to find a binding and uniform extraction method in expert groups for ROSE measurements. Depending on the respective assembly or even its variations, however, very different measurement results can be generated.

The measurement curves listed in Figure 11 which were obtained from an automatic ROSE tester from CBAs following the no-clean approach, clearly illustrate the facts described. Comparative measurements in both solutions were carried out on CBAs with different, slightly modified assembly variants and three different solder mask coatings (with different colours). The assembly contained a usual mixture of different electronic components, for example several LEDs, an SMT connector and about 30 chip components. It is clearly visible that there is a significant dependence on the solvent mixture ratio, but also on assembly variations and different colours of the solder resist, both in the curve progression and in the maximum value of the ionic contamination.

Therefore, it is very important to basically perform all investigations (qualification, validation, series analysis) with a constant solvent ratio in the ROSE test in order to obtain comparable measurement results. A comparison of absolute ROSE values from different ROSE testers and thus also extraction conditions, is not possible.





**Figure 11 – Comparison of ROSE values with different solvent mixtures and material variations of the CBA**

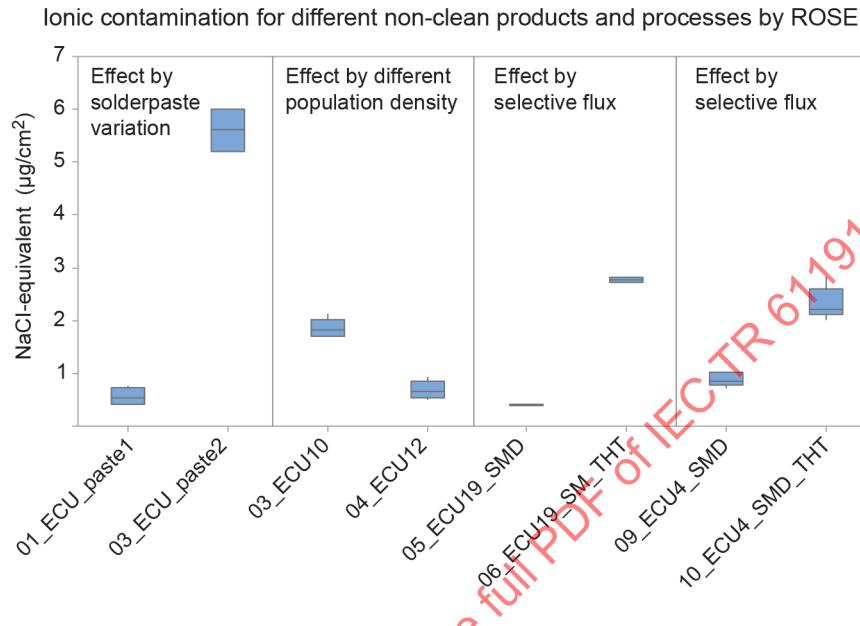
### 5.3.3 Influence of extraction time on measurement of no-clean assemblies

Figure 11 and Figure 7 reveal that the extraction time in a ROSE analyzer can have an influence on the amount of dissolved ionic constituents and thus on the measured value. For example, a lack of temperature compensation or the absorption of  $\text{CO}_2$  from the air in the organic solvent in a ROSE tester can lead to a continuous increase in electrical conductivity [25]. In addition, the use of an organic solvent results in continuous leaching of ionic constituents from the assembly, which would not occur in this manner under an aqueous moisture load on the product (see Figure 13 and Figure 14).

Even though IPC-TM-650, 2.3.25D, [17] requires stabilization of the measurement curve, this has been formulated with the background of process control after intensive cleaning of the assembly. This cannot be achieved for the transfer of the method to no-clean CBAs in most cases. However, since the greatest change in the measurement of electrical conductivity in the ROSE measurement occurs at the beginning and critical, free ionic residues are detected within a few minutes, the ROSE measurement is nevertheless suitable for characterizing ionic species on assemblies. For this purpose, according to the specifications of the instrument manufacturer, the measurement is stopped after a fixed time and the measured value then obtained is documented. Typically, a measurement time of 15 min is adopted for no-clean assemblies. Prolonged exposure time needs to be prevented due to secondary effects. In the context of validation or continuous process control, this fixed measurement time in a ROSE tester needs to be kept constant and documented in order to obtain comparable measurement results.

### 5.3.4 Influence by assembly and interconnect technology on measurement of no-clean assemblies

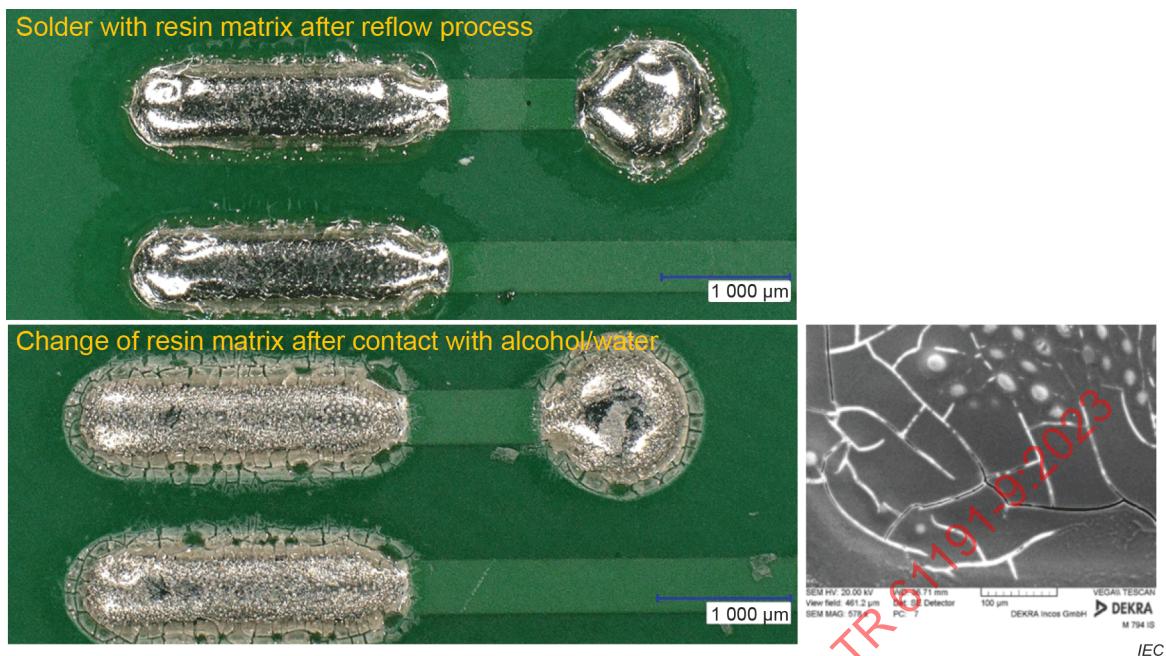
The absolute values are also depending on the assembly and interconnect technology used. Typical values of ROSE measurements are shown in Figure 12. Variations are seen depending on the materials and processes used as well as on the population density of the product. All shown product examples passed active humidity tests and are working properly in the intended end-use environment.



NOTE All products are working properly in the intended end-use environment. Even with ROL0 or REL0 materials, ionic contaminations occur (see [3]).

**Figure 12 – Variation in ROSE values depending on technology used**

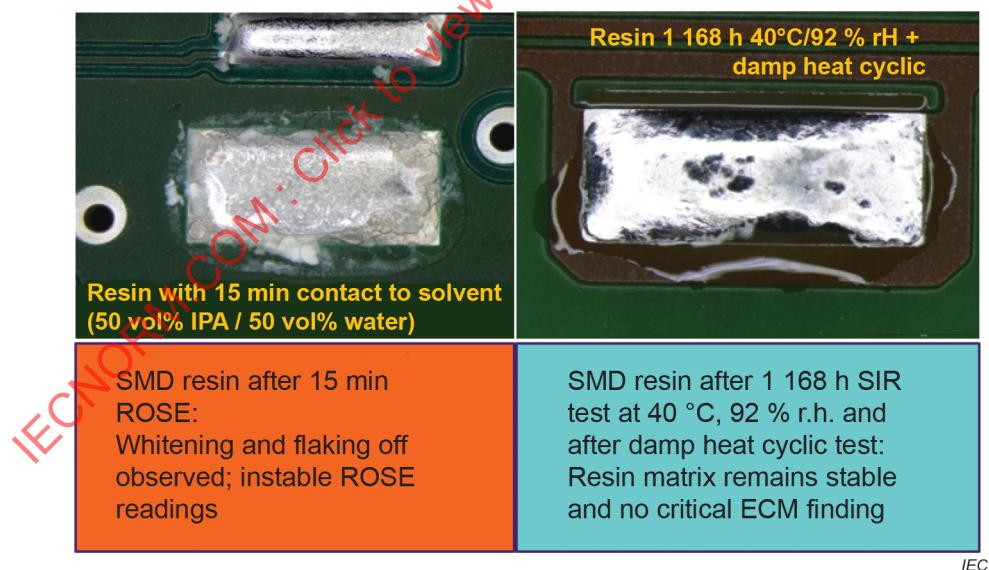
In Figure 9 to Figure 12, scattering in ROSE data and ion chromatography are seen. This scattering is normal for such kind of chemical analysis with many manual handling steps. In addition, the solvent typically based on 2-Propanol/water is destructive for a no-clean assembly. The materials on CBAs that are explicitly designed for use in humid environment and by which reactive constituents (e.g. tin-ions, residues from weak organic acids) are immobilized become soluble in alcohol. And this take place in a random way, so that even for identical parts a strong scattering in repetitions can be measured. Figure 13 shows the destructive effect of the 2-Propanol/water mixture on the resin matrix of modern no-clean SMT solder pastes. The resin matrix turns white as micropores are formed due to the action of the solvent. This does not take place in high humid conditions as shown from long-term SIR-tests.



NOTE The destructive action determines the result of the ionic contamination measurement. The REM image clearly shows the crack formation of the resin after contact with 2-Propanol/DI-water.

**Figure 13 – Destructive action of solvent on resin matrix**

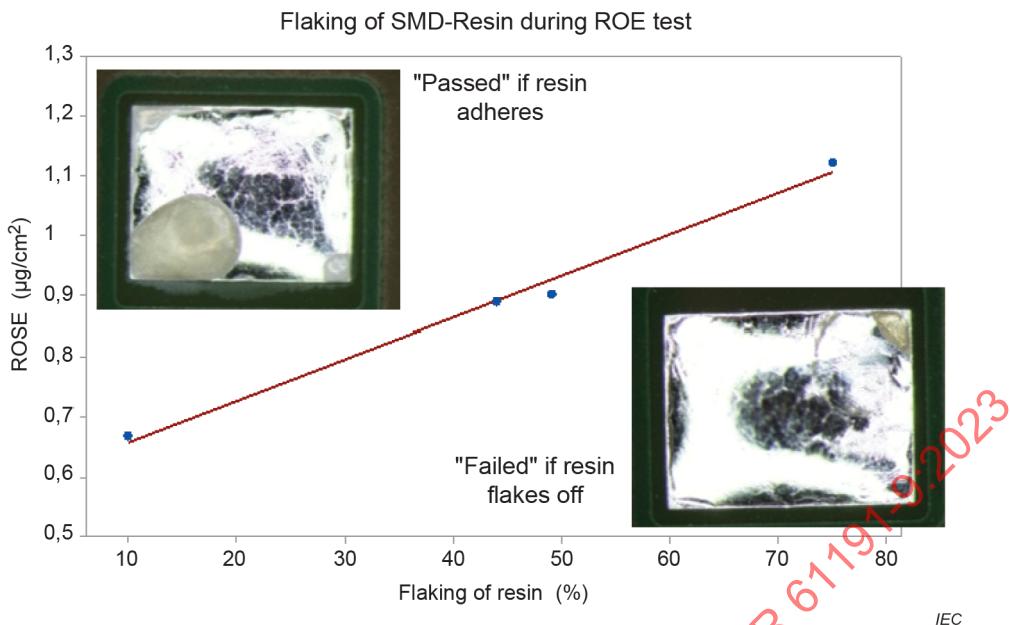
This embrittlement and crack formation of the resin matrix does not take place in humid test conditions as shown from long-term SIR-tests (Figure 14). The contact with alcohol does not simulate the behaviour of the resin in a humid end-use environment.



NOTE The resin matrix remains stable under moisture load (see [3]).

**Figure 14 – Comparison of the resin change**

The destructive action of the resin residues on the CBA take place in a random way. ROSE values are high if flaking or chipping is seen after the test, but ROSE values are lower if the resin adheres at the solder joint. This correlation is shown in Figure 15.



NOTE The degree of chipping determines the result in ionic contamination (see [16]).

**Figure 15 – Destructive action of solvent on resin matrix and chipping effect**

To summarize, absolute values of ionic contamination derived from any extraction method depend on the extraction conditions, the materials and processes used as well as the analyser types. The measurement of ionic contamination alone does not predict reliability. Objective evidence of the robust operation of the assembly in the end-use environment is mandatory. IPC uses the following description for this context [5]: "The use of extraction testing, i.e., ROSE, IC, etc., with no supporting objective evidence shall not [N1D2D3] be used to qualify a manufacturing process (see IPC-WP-019 [7]). Supporting objective evidence shall [N1D2D3] be test data and/or other documentation demonstrating that the performance of the actual hardware is not adversely affected under conditions anticipated in the service environment".

NOTE "[N1D2D3]" means that no requirement has been established for Class 1, Defect Classes 2 and 3.

### 5.3.5 Ion chromatography of no-clean assemblies CBA

The way of extraction method also affects the results obtained by ion chromatography. However, in ion chromatography, a worldwide uniform practice has been established in which an extraction for 1 h at 80 °C in 75 Vol % 2-Propanol/25 Vol % DI-water is performed. Measured values obtained in this way, thus, allow a certain degree of comparability, although even in ion chromatography the measured values themselves do not initially allow an adequate statement to be made about reliability. The same factors of influence that are shown for ROSE, like materials used, the way of processing or population density, will also influence the absolute values obtained. In ion chromatography, too, objective evidence needs to be provided to show that products with given set of ionic residues can be operated without electrochemical failures in the end-use environment.

The advantage of ion chromatography is that the ions found can be used to assign their origin in many cases (Table 1). Furthermore, ion chromatography is a good tool for failure analysis. Significant deviations in the ionic composition measured from a product can be found by ion chromatography if compared with the ionic load (fingerprint) typical for the respective product. This can be an indication of a material or process deviation.

Table 2 shows an example of a product-specific fingerprint of an assembly from a no-clean production process shown in Figure 16.

The values in Table 2 have been normalized to avoid generalization of absolute values to all product types, in this case on Bromide level.

The origin of all ions can be explained, and they do not deviate significantly from similar product classes from the manufacturer. For this product, objective evidence has been provided by passing validation tests in humid climate and experience during mass production. The manufacturing state is documented by this unique fingerprint within its process related data scattering.

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Table 1 – List of ions based on IPC-TM650, 2.3.28 [21]

Ions	Source <sup>a</sup>	Rating	Recommendation for trouble shooting	Recommendation for fingerprint
Chloride	Handling, rinsing, cleaning flux, contamination	Typically only found after wrong handling or rinsing (tap water); critical if significantly present	CB	
Sulfate <sup>b</sup>	Etching processes, iSn electrolyte, flame retardant	Typically relatively low values due to solvent	CB	
Bromide	TBBA, SMT solder paste, flux	Flame-retardant from high Tg-base materials is nowadays not significantly found; Br is released from SMT resin by the solvent and not critical for ROL0 or REL0 materials; critical if from cleaning flux	CB, CBA	CB, CBA
Nitrite	Not present, infrequent flux	Typically not found	CBA	
Nitrate	Process chemicals, cleaning agents, infrequent flux	Typically not found	CBA	x
Phosphate	iSn-Antitarnish, cleaning agent; ENiG, ENiPG	Indication of lifetime problems in NiAu process	CB	
Fluoride	PTFE or similar, flux	Typically not found; only from PTFE-based substrates	CBA	
Acetate	Solder mask	Can point on insufficiently cured solder mask	CB	CB
Formiate	Solder mask	See Acetate	CB	
Methan-sulfonate (MSA) <sup>b</sup>	iSn-electrolyte	MSA based iSn; relatively high values are found	CB	CB
Citrate	iSn-electrolyte	iSn electrolyte; infrequent found		
Succinate	Solder mask, flux		CB, CBA	
Malate	Flux		CBA	
Glutarate <sup>d</sup>	Flux		CBA	
Adipate	Flux			CBA
Phthalate	Packing material	Typically not found		
Sodium	Etching process, handling	Typically found in low concentration	CB	
Ammonia	Cleaning agents; Amine-based chemicals from the solder mask; antitarnish, flux	Relatively high values can be found	CBA	CBA
Potassium <sup>c</sup>	Etching process; Interference with Amine-based components; flame retardant	K itself on low level; but separation from Ammonia and Amines quite often difficult so that apparent high K values are reported	CB	
Magnesium	Water source, solder mask filler	Low values if DI-water is used	CB	If CBA is cleaned
Calcium	Water source, solder mask	Low values if DI-water is used	CB	If CBA is cleaned
Lithium		Typically not found		

CB (state of delivery from supplier); CBA (assembly after all soldering processes without housing)

<sup>a</sup> Origin of ions is indicated based on modern high Tg base materials, solder masks and final finishes used in the automotive industry.<sup>b</sup> Depending on electrolyte and process (high variance).<sup>c</sup> Depending on solder mask, final finish and IC-analyzers; in most cases not K-ions.<sup>d</sup> IPC-TM650, 2.3.28, mentioned Glutamate, which is probably a mistake.

**Table 2 – Fingerprint after ion chromatography of no-clean assembly shown in Figure 16**

Analysis (bag extraction) 1h, 80 °C, 75 Vol % 2-Propanol		SMT only	SMT+THT (incl. connecto r)	Comment (LDL: lowest detection limit)
	Surface / cm <sup>2</sup>	218	434	Factor 2 can be seen in result
Anions	Fluoride	< LDL	< LDL	Not present
	Chloride	3,6 %	1,2 %	At LDL
	Bromide	100,0 %	47,6 %	Released from SMT resin (activator)
	Iodide	< LDL	392,3 %	Released from connector (thermostabilizer)
	Nitrite	< LDL	< LDL	Not present
	Nitrate	< LDL	< LDL	Not present
	Phosphate	< LDL	< LDL	Not present
	Sulfate	< LDL	< LDL	Not present
	MSA	< LDL	< LDL	Not present
WOAs	Acetate	20,2 %	8,3 %	Released from solder mask
	Adipate	10,7 %	84,5 %	Selective flux (WOA)
	Citrate	< LDL	< LDL	Not present
	Formiate	20,2 %	7,1 %	Released from solder mask
	Malate/ Succinate	28,6 %	13,1 %	Released from SMT resin (WOA)
	Oxalate	< LDL	< LDL	Not present
	Phthalate	< LDL	< LDL	Not present
	Propionate	< LDL	< LDL	Not present
Cations	Ammonium	4,8 %	6,0 %	At LDL
	Calcium	8,3 %	13,1 %	Connector (Ca-Stearate as form release agent)
	Lithium	< LDL	< LDL	Not present
	Potassium	< LDL	117,9 %	Released from connector (thermostabilizer)
	Magnesium	< LDL	< LDL	Not present
	Sodium	6,0 %	6,0 %	At LDL
<b>ROSE as different method (normalized NaCl equivalent)</b>				
ROSE (bag extraction)	75/25 solvent	100 %	166 %	Increase due to THT flux, connector
ROSE (analyzer)	50/50 solvent		247 %	Higher than ROSE-bag due to other solvent
Typical ions according to IPC-TM650,665 2.3.28 [21] are listed. Data are only valid product specifically and are normalized on Bromide level.				



**Figure 16 – Assembly manufactured with 2x SMT and 1x THT process for the connector**

#### **5.4 Restrictions and limitations of ionic contamination measurement for cleaned products**

As explained in 5.3, the measurement result of the ionic contamination of no-clean assembly is clearly dependent on the measurement method. If, on the other hand, easily soluble residues are found on the surface after washing, rinsing, or cleaning processes of printed circuit boards or assemblies, different ROSE testers and also different extraction methods can give comparable results. For these cases, the specification of a target value can also be useful in order to control a cleaning result. In the case of no-clean technologies, in which process residues are deliberately left behind but immobilized, significantly different absolute values of ionic contamination needs always to be expected due to the measurement method itself.

##### **5.4.1 Ionic contamination of unpopulated CBs (bare board, state of delivery)**

The manufacture of CBs involves a sequence of chemical process steps. To ensure that residues from these processes (e.g. etching, solder mask application, curing, final finishing) are removed from the CB as far as possible, intensive rinsing processes needs to be carried out. Intensive rinsing and cleaning is mandatory during CB manufacturing. For this reason, it is now useful to set target values for the cleaning performance in order to prevent undesirable residues from chemical processes. Printed circuit board suppliers for the automotive industry are asked to follow the instructions that are given in IPC-5703 [13] for set-up of rinsing and cleaning equipment.

The measurement of ionic contamination is part of the CB supplier's process control as described in the IPC-6012EA [26]. As part of the regular process monitoring, the control is done at the following 3 process gates on a daily basis:

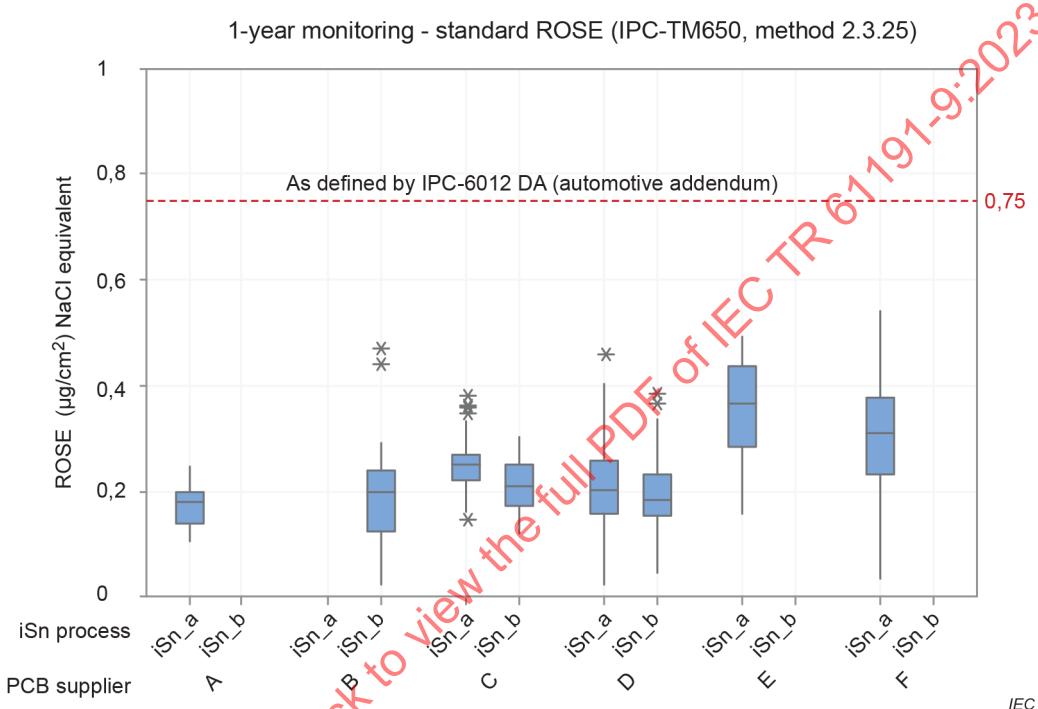
- before lamination process;
- before application of the solder mask;
- after final finishing.

The limit value of ionic contamination by ROSE according to IPC-TM650, method 2.3.25D, [17] is  $0,75\mu\text{g}/\text{cm}^2$  NaCl equivalent [26].

The limit value of  $0,75\mu\text{g}/\text{cm}^2$  NaCl-equivalent after the final finish applies to surfaces with immersion tin (iSn), immersion silver (iAg), organic surface protection (OSP) and the nickel-gold or nickel-palladium-gold variants. As defects of ROSE testers during continuous measurement of OSP-CBs were reported, an SPC based on CB samples before OSP application in combination with continuous monitoring of the electrical conductivity of the final rinsing water in the OSP-line is an acceptable option.

In the case of leadfree hot air solder levelling (Lf-HASL) as final finish process, flux residues will cause quite often higher ROSE values than  $0,75 \mu\text{g}/\text{cm}^2$  NaCl-equivalent. These flux residues from Lf-HASL processes are rated as critical for no-clean assemblies. There is no general limit for this case. A ROSE value in the range between  $0,75 \mu\text{g}/\text{cm}^2$  to  $1,00 \mu\text{g}/\text{cm}^2$  NaCl-equivalent is set for this case. A limit can be derived product specifically and needs to be agreed between the design authority and the manufacturer.

The measured values are recorded in a statistical control chart with a  $C_{pk} \geq 1,33$ . Figure 17 shows an example of process monitoring from various CB suppliers using the immersion tin (iSn) process. Robust processes are achieved as they are well below the limit value of  $0,75 \mu\text{g}/\text{cm}^2$  NaCl-equivalent.



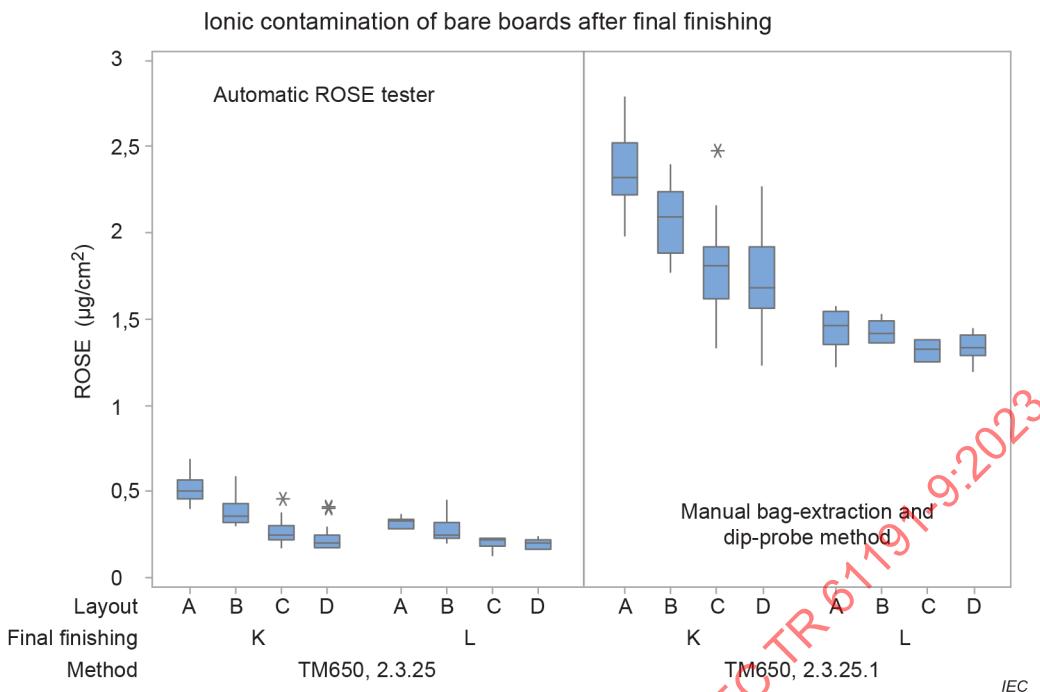
Robust processes were achieved.

**Figure 17 – Comparison of SPC-charts from 1-year monitoring of different CB suppliers and two different iSn final finish processes**

The measurement of ionic contamination is more comprehensive as part of the production part approval process (PPAP). As part of the PPAP, subsequent measurements on unpopulated CBs (state of delivery from CB manufacturer) are carried out and documented:

- surface insulation resistance (SIR), see also Clause 6;
- resistivity of solvent extract (ROSE and/or modified ROSE);
- ion chromatography as option.

Beside the standard ROSE test by automatic analyzers (IPC-TM650, method 2.3.25D [17]), a modified ROSE can be applied for the PPAP, if agreed between design authority and manufacturer. This method is based on IPC-TM650, method 2.3.25D.1 [18]. The extraction is carried out by bag extraction with 1 h at 80 °C in a mixture of 75 Vol % 2-Propanol and 25 Vol % deionized water. The measurement of the resistivity is subsequently done by dip probe method or by pouring the extract into a calibrated automatic ROSE tester. As the extraction method is drastically intensified, entrapped or absorbed process chemicals and insufficiently cured solder mask can more easily be detected. The situation is shown in Figure 18. An aligned target value as part of a process optimization in CB manufacturing is required between design authority and manufacturer if use of modified ROSE (IPC-TM650, 2.3.25.1 [18]) is specified.



Standard ROSE IPC-TM650, 2.3.25D, [17] versus modified ROSE IPC-TM650, 2.3.25.1 [18]. Modified ROSE as tool for process optimization in CB manufacturing

**Figure 18 – Differences in ROSE values for unpopulated CBs depending on the extraction method**

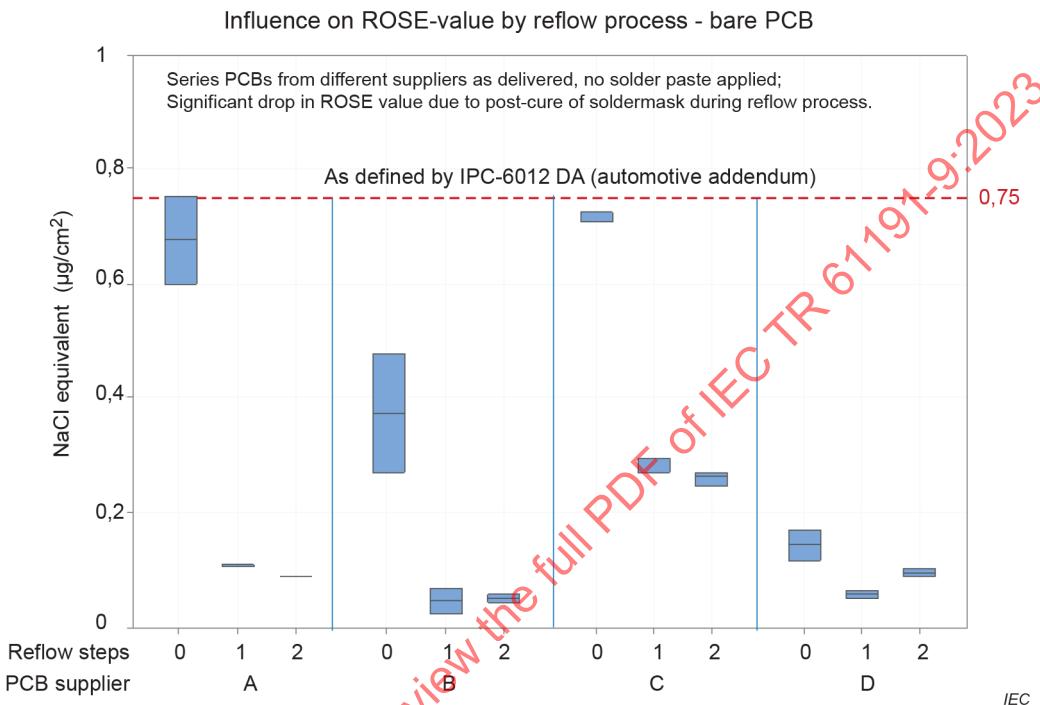
Ion chromatography needs only be carried out for the PPAP and only if agreed between design authority and manufacturer. Ion chromatography is carried out according to IPC-TM650, method 2.3.28, [21] with an extraction time of 1 h at 80 °C in a mixture of 75 Vol % 2-Propanol and 25 Vol % deionized water. Limits are determined product-specifically between design authority and manufacturer. The current contents of IPC-5704:2009 [27] will no longer be valid for CBs for the automotive industry. For modern high Tg base materials and thoroughly conducted rinsing processes of CBs, the found ions Cl, Br, Na, K are much lower than the limits defined by IPC-5704:2009 [27].

Table 3 shows an example of typical fingerprints for bare CBs (after final rinsing, state of delivery, high Tg base material) used in the automotive industry. The values in Table 3 have been normalized to avoid generalization of absolute values to all product types, in this case on Acetate level. In the case of failure analysis and if significant deviations from the reference are found, for example obtained during the PPAP, a root cause analysis needs to be performed.

**Table 3 – Fingerprint after ion chromatography of bare CBs (state of delivery)**

Analysis (bag extraction) 1h, 80 °C, 75Vol % 2-Propanol		iSn final finish	OSP final finish	Comment (LDL: lowest detection limit)
Anions	Fluoride	< LDL	< LDL	Not present, if not PTFE similar base material
	Chloride	6,3 %	6,3 %	At LDL
	Bromide	6,3 %	6,3 %	At LDL
	Iodide	< LDL	< LDL	< LDL (but traces from OSP possible)
	Nitrite	< LDL	< LDL	Not present
	Nitrate	6,3 %	6,3 %	At LDL
	Phosphate	25,0 %	12,5 %	Cleaning, antitarnish, flame retardant
	Sulfate	37,5 %	12,5 %	Etching processes, sulfuric acid based plating
WOAs	MSA	187,5 %	18,8 %	MSA based plating
	Actetate	100,0 %	50,0 %	Released from solder mask; OSP process
	Adipate	< LDL	< LDL	Not present
	Citrate	37,5 %	25,0 %	From plating process
	Formiate	18,8 %	50,0 %	Released from solder mask; OSP process
	Malate/ Succinate	< LDL	< LDL	Not present
	Oxalate	< LDL	< LDL	Not present
	Phthalate	< LDL	< LDL	Not present
Cations	Propionate	< LDL	< LDL	Not present
	Ammonium	25,0 %	12,5 %	Antitarnish, from solder mask
	Calcium	6,3 %	18,8 %	Water source
	Lithium	< LDL	< LDL	Not present
	Potassium	37,5 %	12,5 %	Interference with Amine
	Magnesium	6,3 %	6,3 %	At LDL (water source)
ROSE as different method (normalized NaCl equivalent)				
ROSE (Analyzer)	75/25 solvent	100,0 %	100,0 %	Standard ROSE (IPC-TM650, 2.3.25D [17])
ROSE (bag extraction)	75/25 solvent	325,0 %	175,0 %	Modified ROSE (IPC-TM650, 2.3.25.1 [18])
Bare CBs manufactured with final rinsing processes as state of the art. Typical ions according to IPC-TM650, 2.3.28, [21] are listed. Data are only valid product specifically (layout, solder mask process, plating process) and are normalized on Acetate level.				

It is known that the remaining contamination level of bare CBs after state-of-the-art rinsing is mainly caused by photo-initiators from the solder mask in combination with the final finish process and that this level is additionally reduced by the first reflow step. Already a single reflow soldering step without solder paste reduces this contamination load by evaporation and degradation so that those residues will not remain on the assembly (Figure 19). Also this effect needs to be considered for evaluation of CBs and CBAs based on ion chromatography data. The change in contamination load by processes or adding new materials like a SMT solder paste to the products complicates the evaluation of measurement data and also makes failure analysis more difficult.



See [3].

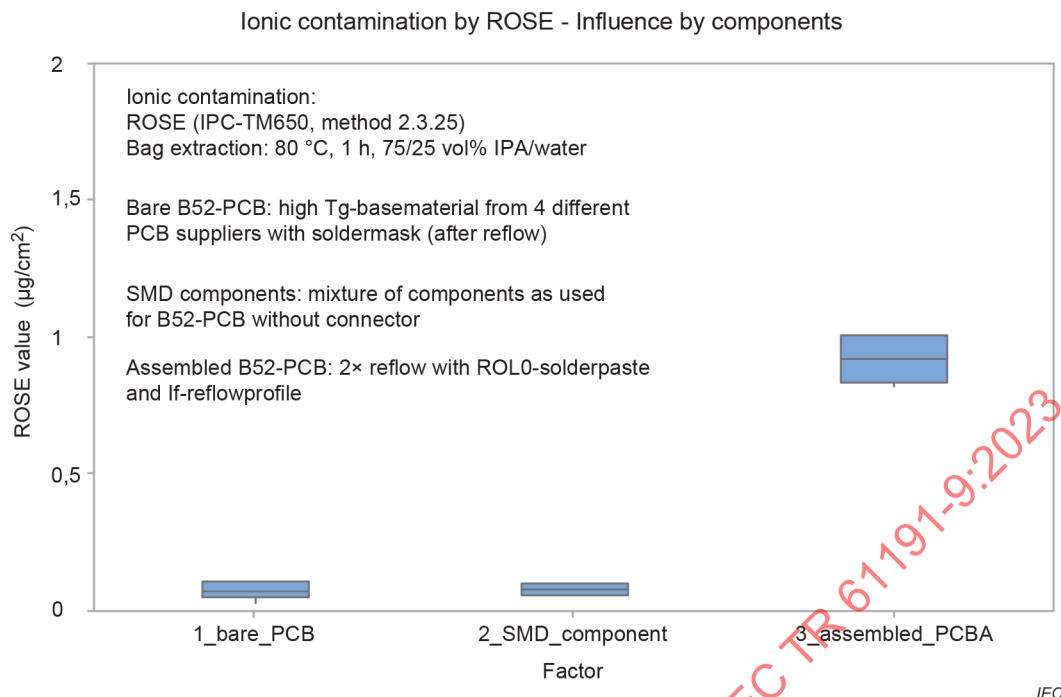
**Figure 19 – Reduction of ionic contamination on bare CBs (state of delivery from CB supplier) by leadfree reflow step without solder paste or components**

It is also known that the layout of the CB has a significant influence on the ionic contamination level. Extended undercut of the solder mask on base material or Copper or realization of solder mask defined pads with formation of crevices can lead to entrapment of process chemicals which are difficult to rinse. Countermeasures are defined in an early design phase. If process technology in CB manufacturing is used to achieve media-resistant adhesion of the solder mask to a pad, solder mask defined pads provide a robust solution without bleeding effects of process chemicals.

#### 5.4.2 Ionic contamination of electronic and electromechanic components

Components in mold housings (e.g. QFP, QFN, BGA, DPAK, SOIC etc.) are not a common source of ionic contamination. In rare cases of process failures during component manufacturing, residues from the tin-electroplating of the leadframe can be found. Residues from sulfate or methansulfonate are typical for such cases as well as calcium or magnesium ions from insufficient cleaning processes.

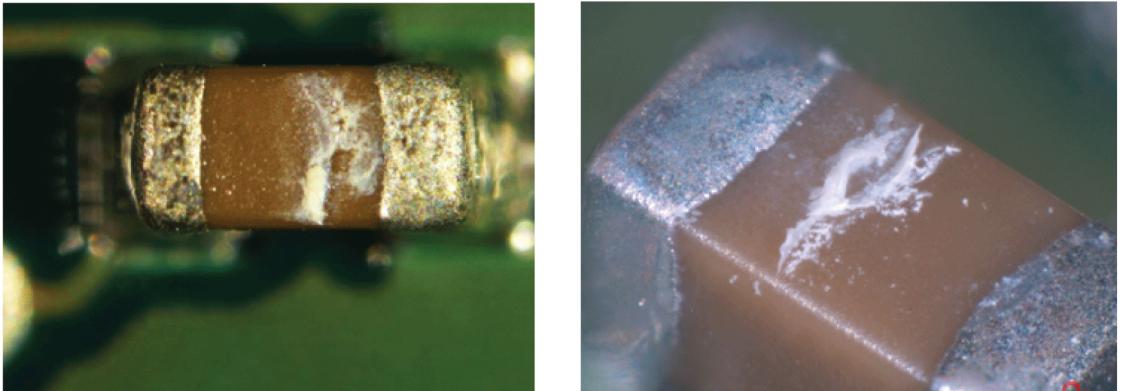
Figure 20 shows the factor of influence for the assembly of B52-CBs [28]. Main source of ionic contamination is the soldering process. The random sample of components for B52 assembly does not show a significant increase in ionic load.



**Figure 20 – Influence of components on the ionic contamination based on B52-standard**

However, due to the package form and increasing use of "leadless components" (bottom terminated components), mounting on the CB can create a risk with regard to ECM. Components such as QFNs, QFPs with ePad, BGAs and SMD electrolytic capacitors can hinder the deactivation of flux residues or the outgassing of flux components due to their "lid effect". A low stand-off of such components can hinder the efficiency of cleaning processes, so that reactive flux residues (WOA) increasingly remain as residue. The special feature of this package shape with lid effect needs to be evaluated in the context of electrochemical reliability investigations, for example in SIR tests (Clause 7).

MLCCs are also subject of cleaning processes and are not a significant source of ionic contamination. However, in active tests in humid environment, quite often white residues are formed on the ceramic body. This is due to the hydrophilic nature of the ceramic and the ease of formation of a closed water film on the surface. By ECM tin ions are released from the termination but they precipitate as inert  $\text{Sn}(\text{O})\text{OH}$  on the surface. After drying, the precipitate remains as non-conductive tin-oxide on the surface. This tin-oxide shown in Figure 21 is not bridging the non-common potentials and is not a metallic dendrite, and this state is acceptable after humidity tests, if leakage currents were not negatively affecting the operation of the product.



The residue is no-conductive tin-oxide.

**Figure 21 – Formation of a white veil or residue on MLCCs during active humidity test**

Coils can be a source of ionic contamination as these consist of different materials and also soldering steps were part of the manufacturing process. Their influence on ionic contamination and electrochemical reliability of the system need to be individually assessed.

Connectors can be a source of ionic contamination. Form release agents like Ca-stearate can remain on their surface. Red phosphor as flame retardants is critical. The use of Potassiumiodide (KI) as thermostabilizer needs to be reduced, because of their influence on ionic contamination and electrochemical reliability of the system.

#### 5.4.3 Ionic contamination of cleaned CBAs

Cleaning of CBAs after soldering is sometimes an additional step in achieving required reliability of the final assembly. Not only it is able to enhance surface conditions prior to subsequent processes like conformal coating, but also can significantly reduce the amount of ions present on a CBA to increase SIR and minimize the risk of ECM. For this reason, cleaning of assemblies can also be useful, even if no-clean solder pastes and fluxes are used. What sounds contradictory in first place might still be necessary if reliability in environmental testing cannot be achieved otherwise. A wet chemical cleaning process is always mandatory after soldering with corrosive, water-soluble fluxes.

In this context, ion chromatography is well suitable to be used as a tool for process validation beside SIR testing. This analysis method is not only able to visualize the amount of critical ions remaining on the substrate after cleaning, but can also be used as a tool to verify the capability of the process steps cleaning and rinsing with DI-water.

Table 4 shows an example of typical fingerprints of a substrate from its state of bare CB to assembled CBA as well as after cleaning. The values in Table 4 have been normalized to avoid generalization of absolute values to all product types, in this case on Adipate level.

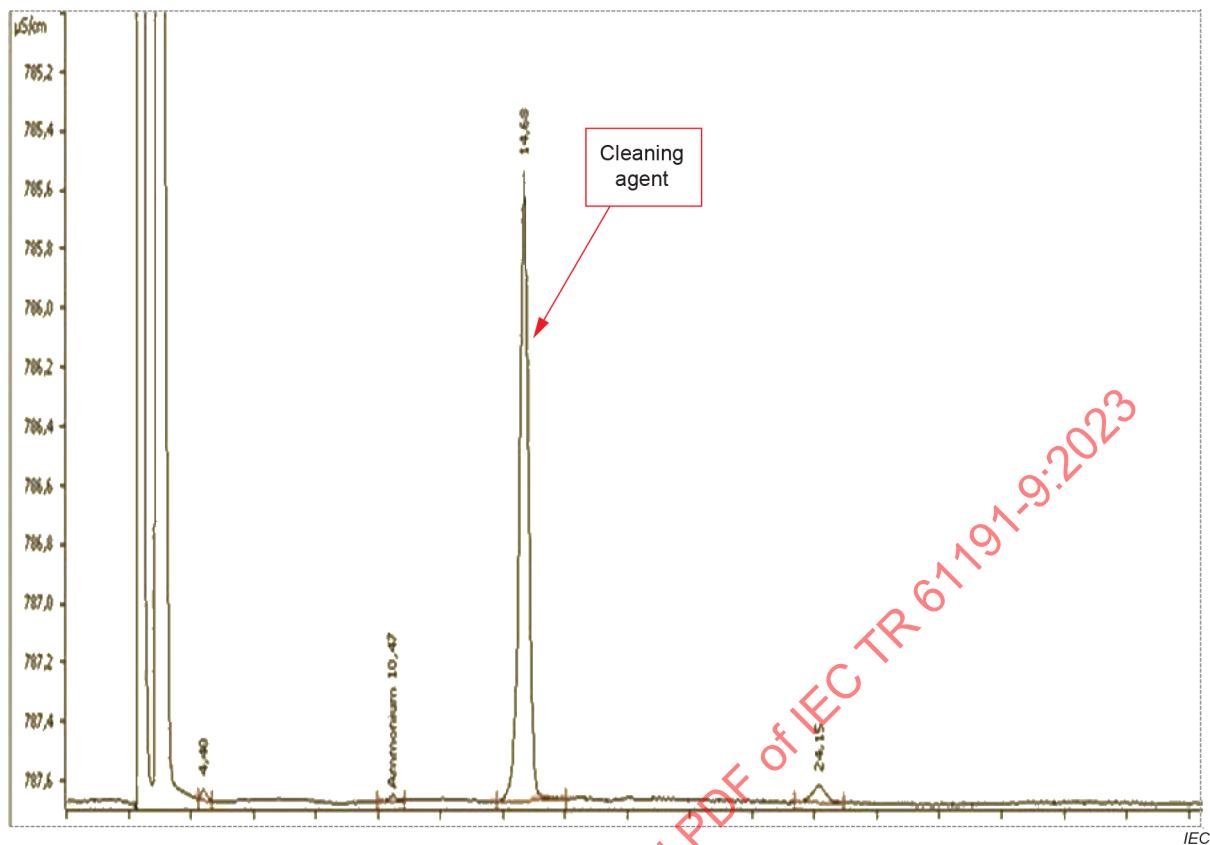
**Table 4 – Fingerprint after ion chromatography of a bare CB and the respective PBA in uncleaned and cleaned condition**

Ion chromatography method (normalized values)				
Analysis (bag extraction) 1 h, 80 °C, 75 Vol % 2-Propanol		Bare CB	Assembled, uncleaned	Assembled, cleaned
Anions	Bromide	1,0 %	1,0 %	< LDL
	Chloride	38,0 %	24,0 %	16,0 %
	Fluoride	< LDL	< LDL	< LDL
	Nitrate	< LDL	< LDL	< LDL
	Nitrite	< LDL	< LDL	< LDL
	Phosphate	< LDL	< LDL	< LDL
	Sulfate	< LDL	< LDL	< LDL
WOAs	Acetate	1,0 %	1,0 %	1,0 %
	Adipate	< LDL	100,0 %	22,0 %
	Formiate	6,0 %	11,0 %	11,0 %
	Glutarate	< LDL	< LDL	< LDL
	Malate	< LDL	< LDL	< LDL
	Methansulfonate	4,0 %	2,0 %	1,0 %
	Succinate	< LDL	< LDL	< LDL
Cations	Ammonium	3,0 %	2,0 %	3,0 %
	Calcium	13,0 %	5,0 %	5,0 %
	Lithium	< LDL	< LDL	< LDL
	Potassium	2,0 %	1,0 %	1,0 %
	Magnesium	4,0 %	9,0 %	3,0 %
	Sodium	3,0 %	3,0 %	2,0 %

Cleaned with wet chemical cleaning processes as state of the art. Data are only valid product specifically (layout, solder paste, cleaning process) and are normalized on adipate level.

As stated in 5.3, soldering processes are adding a significant number of ions to the overall contamination of the CBA. In this case, adipate, a weak organic acid often used for the activation of no-clean solder pastes, is the main contributor. The wet cleaning process after assembly is reducing the overall contamination level considerably. Besides WOAs, activators based on halides are targeted by the cleaning and reduced significantly. In addition, even residues from bare board manufacturing are reduced.

Since it is an additional production step, cleaning is often considered critical. A common concern is that flux residues are only replaced by cleaning chemicals and remain on the CB, posing also a high risk to product reliability. In this case again, the ion chromatography can be used as a control tool. As seen in Figure 22, the chromatogram can deliver valuable information in this context. Besides the known species like ammonium, a foreign peak was detected which later could be led back to traces of the used cleaning agent with the help of a reference sample. This information can then be used for adjusting process parameters like rinsing times accordingly.



With information about the used cleaning chemistry and its fingerprint, unidentified peaks can be traced back to insufficient rinsing.

**Figure 22 – Chromatogram derived from ion chromatography measurement of a cleaned CBA**

Another concern is that cleaning might be insufficient in narrow gaps and under low stand-off components like QFNs. This can also be evaluated by ion chromatography. In the example shown in Table 5, an uncleared CBA was compared with its cleaned version and finally after the components have been removed mechanically after cleaning.

Like in Table 5, the level of adipate is decreased significantly after cleaning. But even after the removal of components, the weak organic acids stay at a comparable level. This proves that the cleaning removed critical ions from underneath the components as well.

Overall, this means that the cleaning process, if well tuned to the product, can contribute to the overall reliability of the product.

Proof of performance and effectiveness of cleaning can be delivered by ion chromatography.

**Table 5 – Fingerprint after ion chromatography of an uncleaned CBA compared to the cleaned CBA and after removing the components**

Ion chromatography method (normalized values)				
Analysis (bag extraction) 1 h, 80 °C, 75 Vol % 2-Propanol		Assembled, uncleaned	Assembled, cleaned	Cleaned, components removed
Anions	Bromide	< LDL	< LDL	< LDL
	Chloride	< LDL	< LDL	< LDL
	Fluoride	< LDL	< LDL	< LDL
	Nitrate	< LDL	< LDL	< LDL
	Nitrite	< LDL	< LDL	< LDL
	Phosphate	< LDL	< LDL	< LDL
	Sulfate	< LDL	< LDL	< LDL
WOAs	Actetate	17,0 %	17,0 %	17,0 %
	Adipate	100,0 %	33,0 %	33,0 %
	Formiate	50,0 %	67,0 %	50,0 %
	Malate	< LDL	< LDL	< LDL
	Methansulfonate	133,0 %	83,0 %	83,0 %
	Succinate	< LDL	< LDL	< LDL
Cations	Ammonium	17,0 %	17,0 %	17,0 %
	Calcium	17,0 %	< LDL	< LDL
	Lithium	< LDL	< LDL	< LDL
	Potassium	< LDL	< LDL	< LDL
	Magnesium	< LDL	< LDL	< LDL
	Sodium	< LDL	< LDL	17,0 %

Cleaned with wet chemical cleaning processes as state of the art. Data are only valid product specifically (layout, solder paste, cleaning process) and are normalized on adipate level.

## 5.5 How to do – Guidance to use cases

### 5.5.1 When is the use of ROSE measurements reasonable?

#### 5.5.1.1 Summary

Despite the many limitations in the measurement of ionic contamination mentioned above, the ROSE tool can be usefully implemented

- for process optimization in manufacturing of CBs (5.5.1.2),
- for process optimization during assembly of CBs (CBA manufacturing) (5.5.1.2),
- to document the production quality as a "fingerprint" parallel to the product validation (5.5.1.3), and
- for process control based on the results of product validation (5.5.1.4).

#### 5.5.1.2 For the definition of production process windows (optimization of processes)

A ROSE measurement can be used as a method to optimize manufacturing processes during the production of the CB or during the assembly (CBA manufacturing).

CB manufacturing: Many chemical treatment steps are carried out during the manufacturing of CBs so that repeated intensive rinsing of CBs is required. In addition, the solder mask

application (type, processing, curing state) is a source of ionic contamination which can interact negatively with the assembly in active humidity testing.

Especially the modified ROSE test (IPC-TM650, 2.3.25.1 [18]) can be used in this context to optimize production processes. The aim of the CB manufacturer needs to be to minimize the load of ionic contamination on/inside the CB (state of delivery) on an acceptable level and in alignment with the design authority (see also 5.4.1). Sampling is carried out directly after the process that is to be optimized.

CBA manufacturing, no-clean: With the no-clean manufacturing approach, substances remain on the assembly that are detected as ionic contamination. The main causes are soldering processes (in particular the application of flux before selective soldering), for example if an excess of flux is applied, or if insufficiently deactivated flux residues remain on the assembly (e.g. under components or by spraying into the SMT area). Improper handling of assemblies, for example without gloves, the use of brushing processes, rework with reactive fluxes and local cleaning, are further strong sources of ionic contamination. Such uncontrolled contamination can reduce the electrochemical reliability of the product under certain circumstances.

Process steps that have an influence on the electrochemical reliability of an ECU needs to be evaluated, excluded or optimized by experts during the product development procedure. Such manufacturing processes include steps with flux application (SMT, THR, THT, hand and robot soldering) as well as the application of polymer materials (dispensing of TIM, gluing, underfilling, protective coating, potting, etc.) including interactions with flux residues and any handling. ROSE measurements can help to define optimal process windows if they are not yet or not sufficiently described in process instructions. Sampling is done directly after the process to be optimized. The optimization goal is to achieve ROSE values as low as possible without negatively affecting other quality criteria at the ECU or the production flow (e.g. minimum ROSE values so that soldering results on the product are still sufficiently good according to IPC-A-610 [29]).

#### **5.5.1.3 For documentation of the production quality as fingerprint parallel to product validation**

It is advisable to determine ROSE values for products as a so-called "fingerprint based on ROSE". This is done at the time when products are manufactured under conditions of mass production also for product validation. The measured values obtained are documented together with the results of the humidity tests. This procedure now creates a comparison between an ionic contamination load on the CBA resulting from the no-clean production and a passed active humidity test. At this moment, objective evidence according to IPC-J-STD001 [5] is achieved. This procedure thus represents the relationship between humidity robust products and a given ionic contamination load from the no-clean production including its process-related fluctuations, depending on the measuring method used and the materials and processes. A fingerprint characteristic for the product with proof of its humidity robustness was thus determined. This fingerprint can now be used, for example, to randomly verify the consistency of production processes. This fingerprint can also be used as a basis for approval, for example, if production lines are duplicated or a production site is relocated.

The procedure can be transferred to as "continuous process control" or "ramp-up assurance".

#### **5.5.1.4 For process control based on results during product validation or ramp-up assurance**

Even though robust processes and manufacturing consistency are already required by IAF-T 16949 [1] in the automotive industry, ROSE can be introduced as a measure of a continuous improvement process in plants to track compliance of manufacturing processes within the permitted process windows and product specific requirements. The use of ROSE as process control tool as well as frequency need to be product specifically agreed between design authority and user. If ROSE is used as process control tool, the measurement is performed with products from mass production or a technically representative assembly that can be minor populated on an identical manufacturing line on a regular basis. The comparison is made with

the measured values that were determined and fixed during product validation. The ROSE measurement is typically considered to be a destructive measurement for a CBA, so that the PBA need to be scrapped after the measurement.

Since, in addition, the ROSE measurement cannot be implemented at the same time as the production flow in modern electronics manufacturing, it is only an indication method. As a good basis, if regular process control based on ROSE is required, the measurement of two CBAs from mass production per month take place within a limited period of 6 months as part of a ramp-up assurance (see 5.6).

### **5.5.2 When is the use of ion chromatography reasonable?**

#### **5.5.2.1 Summary**

In contrast to ROSE measurements, tests using ion chromatography require trained chemical-technical personnel as well as the infrastructure of a comprehensive chemical laboratory. The use of ion chromatography is therefore limited to a few use cases:

- to document the production quality as a fingerprint parallel to product validation (5.5.2.2);
- as a tool for root cause identification in failure analysis (5.5.2.3).

#### **5.5.2.2 A fingerprint parallel to product validation**

The determination of measured values via ion chromatography for new product groups and new materials as an unique fingerprint is currently rising. Available data from IC is also a product-specific fingerprint with higher significance than the ROSE measurement. This applies to both CBs and CBAs. The limitations listed above due to effort and the capability of external analytical laboratories to use IC measuring equipment needs to be taken into account. The determination of IC data needs to be product specifically agreed between design authority and manufacturer and can only be defined for specific cases. The best time for determination is again parallel to the production of CBs and CBAs for product validation. The measured values obtained need to be documented together with the results of the environmental test or as part of the PPAP, so that a comparison with the results from humidity tests is possible.

#### **5.5.2.3 A tool for failure analysis**

Ion chromatography is an excellent tool for failure analysis. If significant amounts of specific ions are detected from the CB or CBA, which are significantly above typical values, this is an indicator of issues with materials, processes or handling. The detected element can give an indication of the origin of this deviation. Within the framework of failure analysis, other methods of extraction (e.g. local extraction, wiping techniques, etc.) and measurement of ionic contamination can also be used than those described in IPC-TM650, 2.3.28, [21] with bag extraction. The different extraction methods lead to differences in absolute values, so that a reference point is always required. Therefore, historical data needs to be already available, which were determined with the same methods as used for failure analysis, or analyses of "good parts" carried out in parallel are used to provide a point of reference, so that an evaluation of measured contamination levels is possible.

### **5.5.3 At what point in the manufacturing sequence ionic contamination measurements are carried out, if a fingerprint or the basis for process control is to be established?**

For CBs, see 5.4.1.

For CBA, no-clean: Ionic contamination measurements as a fingerprint for product validation or for process control only make sense if production is carried out under conditions of mass production. Materials and optimal process windows are already defined for the product.

Samples are typically taken directly after the last soldering process directly from the production line, for example

- after 2 x reflow soldering but before the press-fit technology,
- after 2 x reflow soldering, THR- or THT-soldering but before mounting the housing, or
- after all soldering processes, but before encapsulation (conformal coating, potting, etc.).

Further handling steps that can lead to contaminations (e.g. handling in milling processes) need also to be considered.

In special cases, instead of products from mass production for measuring ionic contamination levels, technically representative assemblies with reduced components can be used. Other material, processing and handling need to be identical to mass production.

#### 5.5.4 How is the sampling for ROSE and IC done?

Samples (CBs or CBAs) can only be measured once. The measurements are destructive for CBAs from a no-clean production so that these parts need to be scrapped after the extraction step. Sampling needs to be carried out in such a way that no contamination occurs during sampling, packaging, transport or by handling in the analysis laboratory. This can be achieved by taking samples directly from the production line without placing them on carriers, using powder-free disposable gloves (nitrile, low chloride) when handling the samples and immediately wrapping them in new aluminium foil. Only then the samples are packed in ESD-bags. Labelling the CB or CBA surface with foil pens needs to be avoided as well.

#### 5.5.5 How is a product-specific process control limit based on ROSE determined?

Materials, processes and also the measuring method for ROSE needs to be defined.

The calculation is carried out with the usual methods of quality assurance and creation of a statistical process control chart. The mean value and the standard deviation  $\sigma$  needs to be determined for this purpose. The upper process control limit (UCL) is then calculated according to:

$$UCL = \text{mean value} + 3 \times \sigma$$

The calculation,  $+ 3 \times \sigma$ , takes into account the expected distribution of 99,7 % of all parts from this statistical population. A statistically relevant sample size from different production lots is a basis for the calculation.

The specification of generally applicable limits without objective evidence can no longer be accepted due to the explanations given here and the change in IPC-J-STD-001 [5] made in this regard.

The product-specific process control limit valid for the CBA needs to be defined in a test document that becomes part of the product specification.

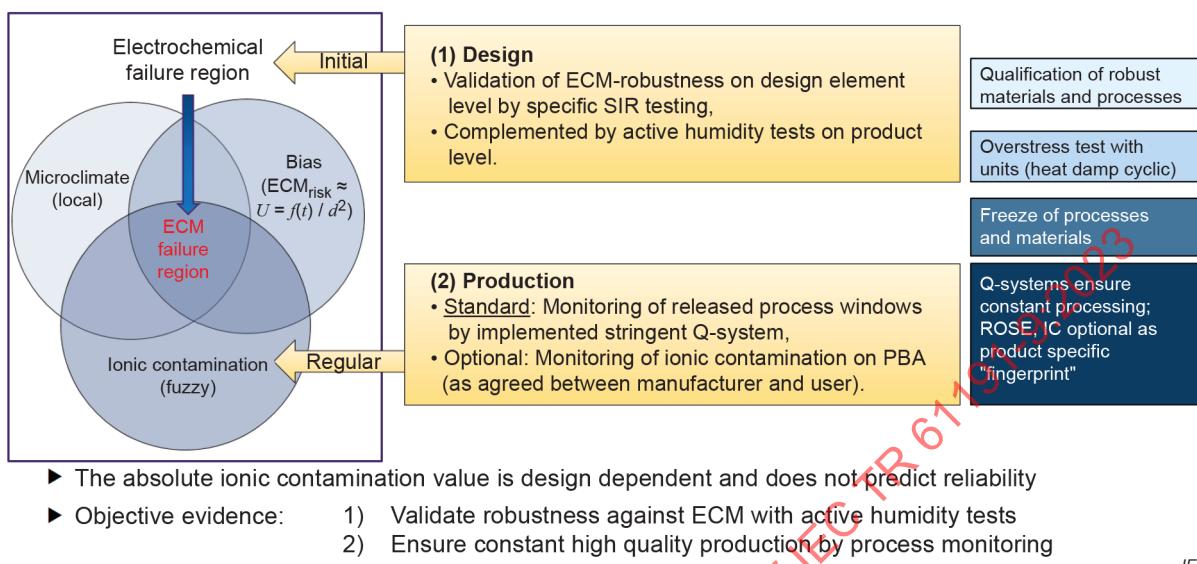
Measured values or control charts for ionic contamination need to be available on request of the user. The measured values or control charts need to show that agreed process control values are adhered to, thus ensuring controlled manufacturing processes.

### 5.6 Examples for good practice

#### 5.6.1 Ways to achieve objective evidence

One way towards objective evidence for a qualified manufacturing process is shown in Figure 23. In a first step, the materials and processes of electronic production are subjected to extensive qualification tests. This involves advanced SIR tests that are based on single material characterization (e.g. IPC-TM650, 2.6.3.3 [30]) and go to interactions tests as described by IPC-9202 [28] during the design phase. The procedure is described in Clause 6 and Clause 7. It is followed by overstress tests on product level by active constant climate and cyclic climate

tests. If these tests are passed successfully, objective evidence is achieved. Even if the constancy of materials and processes is ensured by quality management systems, an optional measurement of the ionic contamination for mass production can be agreed between design authority and user.



IEC

See [16]

**Figure 23 – Approach for achieving objective evidence for a qualified manufacturing process in the automotive industry**

### 5.6.2 Introduction of a new product family with new materials

A supplier to the automotive industry (high volume electronic manufacturing) has designed a new airbag control unit. In addition to layout changes compared with earlier versions and the implementation of new functions, a new CB base material and a new SMT solder paste will be used. The CB will be sourced from two different suppliers in Asia. The assembly and interconnect technology is a no-clean production involving both surface-mount and through-hole soldering technology typical for the automotive industry.

In a first step, the base material was selected and investigated regarding its CAF performance according to IPC-TM650, 2.6.25D [31]. First samples of CAF (customized test board) and SIR test coupons (B52 test board) including solder mask application and final finish were then ordered for production from the two CB suppliers. Through automotive typical long-term tests at 85 °C, 85 % rH for 1 000 h, the capability of all CB materials and the CB manufacturer as well as their interactions within the PPAP was demonstrated with regard to CAF and SIR performance of unpopulated CBs.

In parallel, the new solder paste was released based on positive results of an SIR test according to IPC-TM650, 2.6.3.3B, [30] and interaction tests according to IPC-9202 [28].

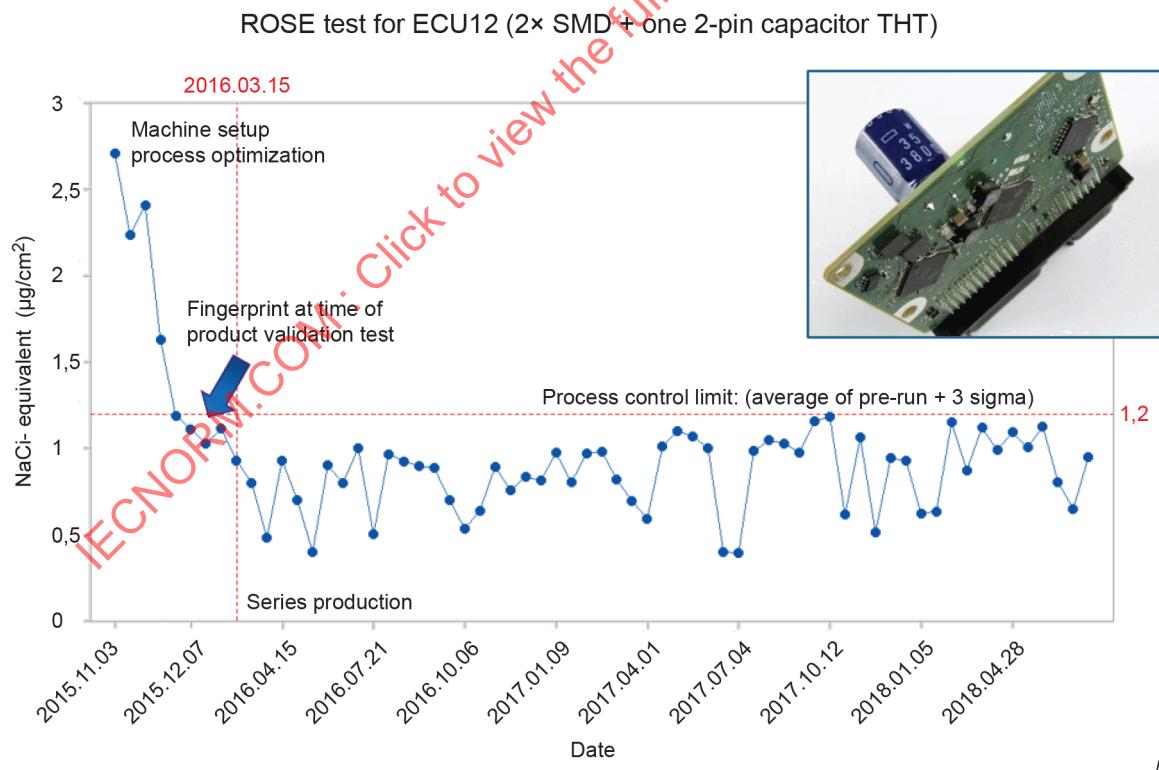
During these material release tests, an investigation of the nature of ionic contamination on the test coupons is also carried out by ion chromatography as a material characteristic fingerprint.

With the materials now defined, the ECU was built under mass production conditions. However, the initial settings on the selective soldering system led to high ionic contamination of 2,5 µg/cm<sup>2</sup> NaCl equivalent (based on the fixed measurement conditions of the ROSE tester used) due to a flux excess in selective soldering. The ROSE method was therefore used to optimize the process. The aim was to reduce the ionic contamination load on the no-clean assembly without negatively affecting the solder joint quality and also to take into account given fluctuations in high volume production. The determination of an optimal process window for

fluxing prior to the selective soldering process was then made in coordination between the manufacturing plant, development and product managers and was  $0,8 \mu\text{g}/\text{cm}^2$  to  $1,0 \mu\text{g}/\text{cm}^2$  NaCl equivalent. The process parameters for manufacturing the new airbag control unit were fixed. At this time, the determination of the ionic contamination as a characteristic fingerprint was also carried out.

A product-specific process control value with respect to ROSE was derived and documented from the mean value and three times the standard deviation, which amounted to  $1,2 \mu\text{g}/\text{cm}^2$  NaCl equivalent (based on the fixed measurement conditions of the ROSE tester used).

Within the scope of product validation, a further testing step involved the manufacturing of a total of 12 complete control units from the 2 CB suppliers under mass production conditions. The devices including housing were again tested in humidity tests. These are active tests without self-heating (battery on, switch between idle and wake-up mode) for 1 000 h at  $85^\circ\text{C}$ , 85 % rh and for 10 days in a cyclic climate test according to IEC 60068-2-38 [32] (considerably overstressing real field loads). Leakage currents  $< 200 \mu\text{A}$  and no indication of electrochemical migration on the CBA in the optical analysis are typical pass/fail criteria. The tests were successfully passed. For this CBA and the fixed materials and process conditions, objective evidence was now achieved. The manufacturing quality for this product at the time of the passed environmental tests was documented by an average and the corresponding standard deviation of the ROSE value. A random measurement of the ionic contamination by ROSE on the no-clean assembly was agreed between user and supplier. This has been carried out for more than 4 years on two assemblies per month and per production plant. The results proved that the product-specific ionic contamination was consistently below  $1,2 \mu\text{g}/\text{cm}^2$ , thus ensuring constant production processes and the continued delivery of humidity robust products. The realization of this procedure is visualized in Figure 24.



The process is kept constant during mass production. Known data scattering for no-clean assemblies (see [16]).

**Figure 24 – ROSE as process control tool**

### 5.6.3 Adaptation of an ECU for a new vehicle type

In another example from the automotive industry (high volume electronic manufacturing), an engine control unit, which is already in mass production with  $> 1$  million ECUs delivered per

year, is to be adapted for a new vehicle type. This can be realized by using available assembly options in the SMT process. Only the component spectrum and the layout change slightly; however, minimum electrical clearance as well as case sizes of components including pitch are not affected.

All CB materials, already qualified CB suppliers and soldering processes remain unchanged. After the assembly of test samples on mass production lines, the ionic contamination is measured by means of ROSE on some CBAs from the no-clean production after selective soldering. This results in measured values between  $2 \mu\text{g}/\text{cm}^2$  to  $3 \mu\text{g}/\text{cm}^2$  NaCl equivalent (based on the fixed measurement conditions of the ROSE tester used). These are typical measured values for this type of devices, which result from the no-clean production and the large selectively soldered through-hole connector. The ROSE fingerprint of this modified engine control unit is therefore not significantly different from those for which extensive humidity tests and comprehensive field data are already available. This means that objective evidence has now been achieved: proof of the humidity robustness of the product was provided by measurements of ionic contamination and comparison of test data and field data of devices with comparable assembly and interconnect technology and unchanged housing concept. Thus, a reference release was agreed upon. Testing of the modified ECUs in long-term humidity tests typical for automotive applications was not necessary.

## 6 Surface insulation resistance (SIR)

### 6.1 SIR – An early stage method to identify critical material combinations and faulty processing

As shown in Figure 4, the electrochemical reliability of an assembly is determined by the interaction of microclimate, layout with potential differences and material properties. To characterize the electrochemical reliability of these systems on a first level, measurement of the surface insulation resistance (SIR) is currently the best method. The SIR measurement is a level of abstraction, with approximation of simple material combinations (e.g. SMT flux on a B24 or B53 test board without final finish and without solder mask) towards increasingly complex systems (e.g. by B52-CBA with applied coating). The aim is to use SIR tests to exclude materials and material combinations and faulty processing at an early stage, which could lead to humidity induced faults on a product. The second level would be active humidity tests of the product in its entire complexity and in a climate simulating a critical environment for the product as shown in Figure 23.

### 6.2 Fundamental parameters of influence on SIR

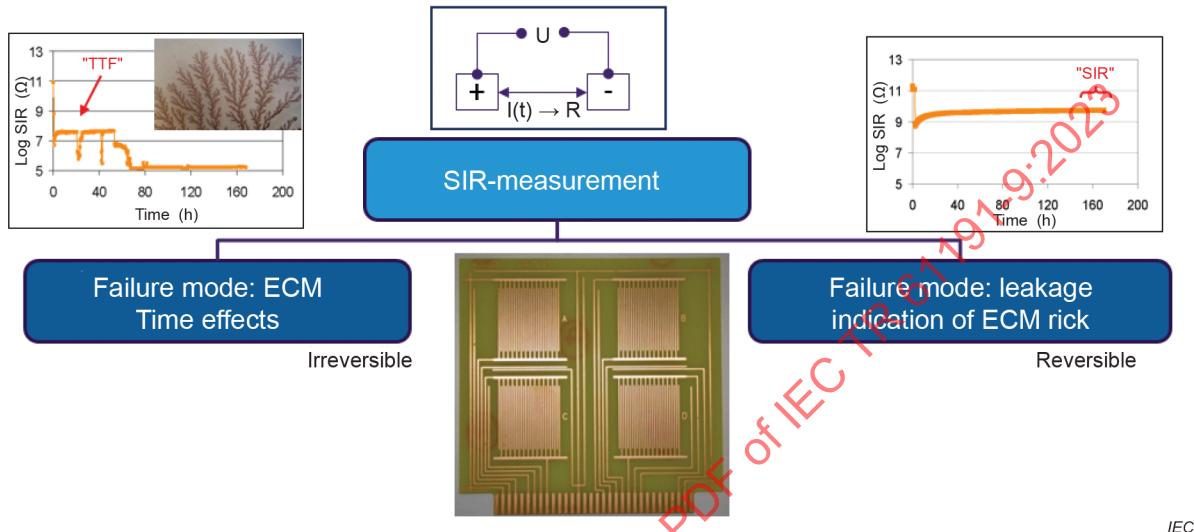
#### 6.2.1 General aspects

In the SIR measurement, a leakage current is measured across the surface of a simplified printed circuit board or CBA without housing in a humid environment and converted into the surface resistance by means of the applied DC voltage (IPC-9201 [33]). There are now two possibilities for characterizing the system, which are illustrated in Figure 25. Using standardized test coupons (e.g. B24, B52, B53), after a short measurement period, during which the SIR measurement value has stabilized, its absolute value is taken and transferred to databases for this specific test condition and material combination. A higher SIR reading for a material system is often considered more robust than a lower reading for the same test condition. The alternative view is to use the method as an event tracker around short circuits, which are represented as spikes in the SIR measurement curves. In this case, the evaluation is often done as a pure pass/fail criterion.

The first method focuses more on the electrochemical events directly caused by the applied materials and can easily be seen within 168 h test duration. People who follow this approach therefore insist on proper adherence to test conditions, mandatory use of a standardized test layout, and round robin comparisons (IEC 61189-5-506 [34]). For this reason, a fixed measurement period of 168 h is also mentioned in many standards for the SIR test. This view is consistent with the original setting of the SIR test.

The second approach is based on material degradation and diffusion processes, i.e. more on long-term effects, with the consequential error of an electrical short circuit. In later case, a test duration of 1 000 h is quite common. As test conditions by this approach deviates significantly in most cases from SIR-standards, this approach is rather be called a temperature-humidity-bias (THB) test based on SIR test equipment.

A combination of both methods with identification of the test conditions and display of the SIR measurement curves is recommended nowadays.



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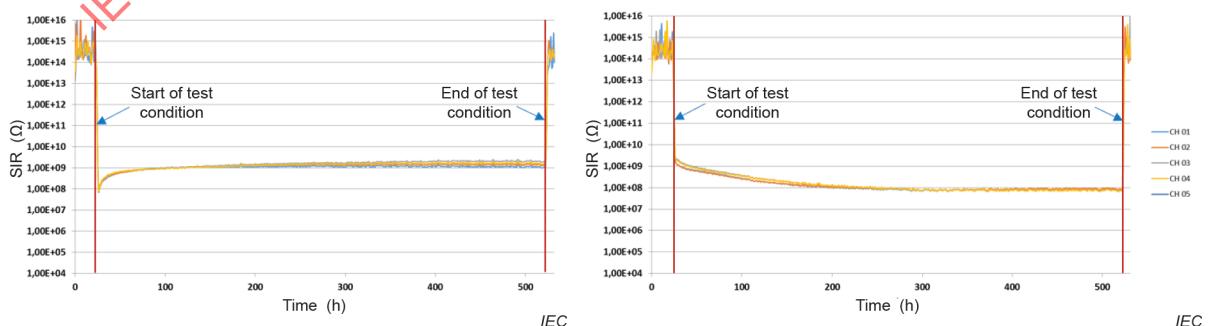
Detection of shorts over time due to material degradation or diffusion process (left) and evaluation of SIR-level after fix time due to electrochemical material characteristics (right) (see [35]).

**Figure 25 – View on SIR measurement**

Figure 26 illustrates in addition a common behaviour of SIR curves which often falls into two broad categories:

- 1) an initial drop in insulation resistance followed by a steadily improving SIR level with time – Figure 26 a);
- 2) a lesser drop in insulation resistance, followed by a shallow declining trend with time – Figure 26 b).

Although exceptions are known, experts rate the first case as an indicator of robust long-term stability of the material, while in the second case failures are more likely to occur after a longer test period. This could be due to contamination in many cases.



**a) Example SIR test of soldermask with B53 coupon  
Soldermask A**

**b) Example SIR test of soldermask with B53 coupon  
Soldermask B**

Left with significant drop at the beginning of measurement and steadily improving SIR value; right with a less drop at the beginning of measurement and slightly ongoing decrease.

### Figure 26 – Principal course of SIR curves

For simple, clean SIR test boards with bare epoxy and Cu-foil based comb structures (like a B24 or B53 coupon), important factors of influence on the SIR-value are merely the climate and the test voltage. Figure 27 shows this effect on the leakage current, respectively the stabilized SIR value after 168 h measurement. The surface resistance remains nearly unchanged for the Cu-Cu distance in the stabilized condition. Also the application of a final finish with correctly applied rinsing of the test coupon does not change the stabilized SIR value for this design.

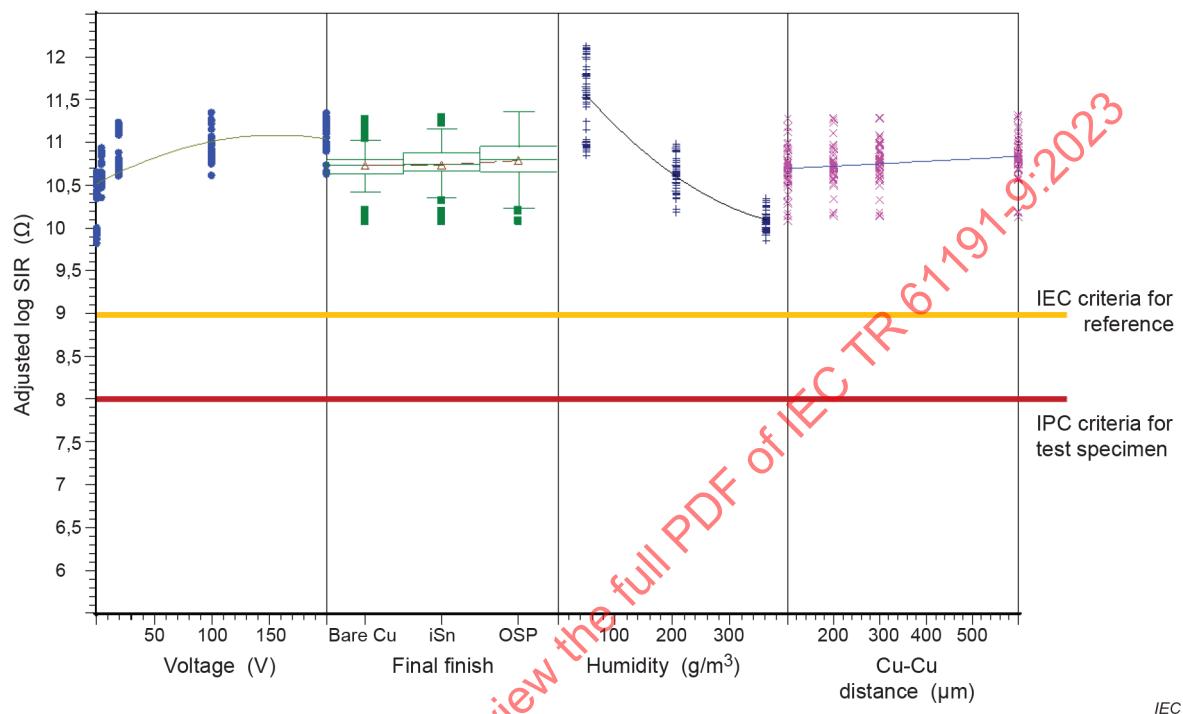


Figure 27 – Response graph concerning stabilized SIR-value after 168 h from a DoE with B53-similar test coupons (bare CB)

The consequences from this observation are explained subsequently.

### 6.2.2 Influence of climate

The formation of a water film on the surface of a SIR test coupon, but also the absorption of water in the bulk phase of material (e.g. the solder mask), determines foremost the SIR value. Transport of charge carriers by diffusion and migration in the thicker water film is facilitated. A gradual approach to the dew point leads to a decrease in the SIR. This process is now massively disturbed by materials that accelerate charge transport or the formation of the water film. The SIR level drops faster or is already at a low level from the beginning of the measurement. Therefore, the climate needs to be perfectly monitored and controlled during the SIR measurement in a test chamber. A comparison of SIR measurements is only possible under identical climatic conditions. In Figure 28, the trend depending on the climate is shown for a simple comb structure covered with SMT flux after reflow. Up to two decades difference in SIR between the widely used conditions at 40 °C, 90 % rH and 85 °C, 85 % rH are seen.



Significant influence by climate on the absolute SIR-value.

**Figure 28 – SIR measurement with B24-CB, no-clean SMT solder paste**

IEC 60068-3-4:2001 [4] requires that the test climate used for material characterization by SIR tests does not change the mechanism of electrochemical reactions and is appropriate to the end-use environment of the CB or CBA as tested without housing.

The content of [4] is important. This document describes and justifies in detail the use of various climatic conditions that affect the electrical properties of circuits. In order to cover adsorption, absorption and diffusion of moisture in materials, a constant climate test needs to be carried out in a way that the electrochemical mechanism is not changed. The constant climate test IEC 60068-2-78 [36] with  $(40 \pm 2)^\circ\text{C}$ ,  $(93 \pm 3)\%$  r.h. is recommended for 1 000 h for CBs and CB as with open structures in this case. This condition is reflecting a real field load. In case of polymer coatings on the CB (e.g. solder mask or encapsulations), the test climate could be enhanced so that the diffusion process is accelerated. For this condition, the climate condition with  $(65 \pm 2)^\circ\text{C}$ ,  $(93 \pm 3)\%$  r.h., 1 000 h has proven to be more advantageous than using the 85 °C, 85 % r.h. condition. The test condition with 85 °C, 85 % r.h. (IEC 60068-2-67 [37]) was defined to trigger special degradation mechanism. Under these severe conditions, other failure mechanisms may occur than under normal operating conditions. Therefore, this test condition can only be used in justified cases (e.g. solder paste qualification).

In case of short-term dewing on the surface of CBs and CBAs without housing that will change the electrical characteristic, IEC 60068-3-4:2001 [4] recommends the damp heat cyclic test in accordance to IEC 60068-2-30 (6 cycles from 25 °C to 55 °C at 96 % r.h., 6 days [38]). This test reveals real weaknesses well without significantly overstressing the CB or CBA and reflects experience from field performance of electrical products [16], [39].

The basic prerequisite for reliable SIR tests is the use of measurement equipment that is capable in accordance to IEC 60068-3-6 [40] and IEC TR 61189-5-506 [34]. See also IPC-TM-650, method 2.6.3.7, for equipment requirements [41]. Awareness for maintenance of the climatic chambers before each series of measurements is also prerequisite for reliable SIR tests. The complete cleaning of the inner chamber walls, for example with hot water steam and wipe with lint- and salt-free cleaning tissues with DI-water, and the replacement of the reservoir of DI water are good practice. The use of DI water with a residual conductivity of  $< 20 \mu\text{S}/\text{cm}$  is common practice. The humidity inside the climatic chamber needs to be realized aerosol-free.

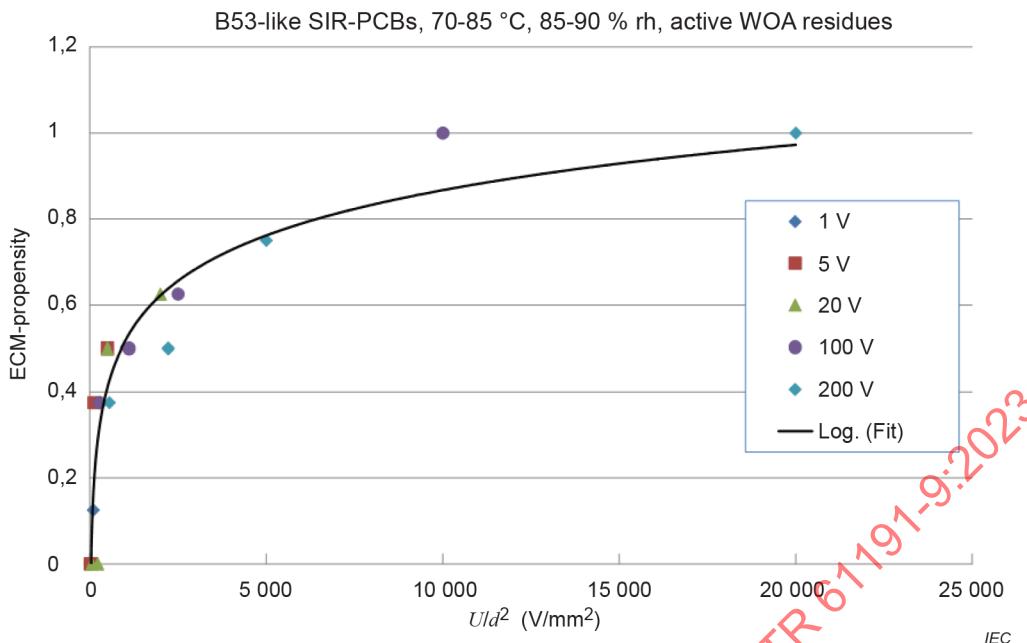
The minimum requirements for the climatic chamber are specified in IEC 60068-2-78:2012 [36], IEC 60068-2-30:2005 [38], or IEC 60068-2-38 [32]. To meet these requirements, the following points are essential.

- The air flow in the chamber is not hindered by the installation of the test specimens.
- CBs and CBAs are installed vertically to the air flow (along with the air flow) with a mounting distance of typically  $\geq 20 \text{ mm}$ .
- Cabinets are not be loaded above 15 % of the chamber volume.
- Distance of the test specimens to the wall is at least 10 % of the chamber width or height (see also calibration test [42]).

### 6.2.3 Influence of voltage

Some publications state that 5 V is the most critical situation for SIR testing. Thus, the trend in SIR with increasing voltage, as shown in Figure 27, seems surprising at first. But, in accordance to Ohm's law and a low and nearly constant leakage current in a stabilized state, the measured surface resistance must increase with intensification of the voltage. The effect shown in Figure 27 is merely a pseudo increase in robustness. This voltage of 5 V is beyond the decomposition potential of water, leads to lowest SIR values and is above the operation mode for many logic circuits. With this view, it is already critical. In addition, it is reported that, at 5 V voltage, a dendrite is less likely to burn off in the SIR test and thus more likely to be found as a drop in the measurement curves or by optical inspection than at higher voltages. However, an increasing voltage leads to formation of more crystal sites in electrochemical migration (= more chances to form a bridging dendrite), can trigger other humidity induced failures like treeing effect, arcing, or partial discharge so that the propensity of a humidity induced failure will increase with the voltage (Figure 29). For comprehensive SIR investigations on standard test coupons like B52 or B53, a voltage of 50 V is considered as a good practice for automotive applications. A 48 V board-supply is covered as well as the higher propensity for ECM failures but the overstress situation with 100 V is avoided.

For tests of ECU-BGAs or BGA-footprints on B52-like test CBs, the test voltage is set at 5 V as usual for the operation of logic circuits.



1 = 100 % of structures in the test failed with signs of ECM (see [35]).

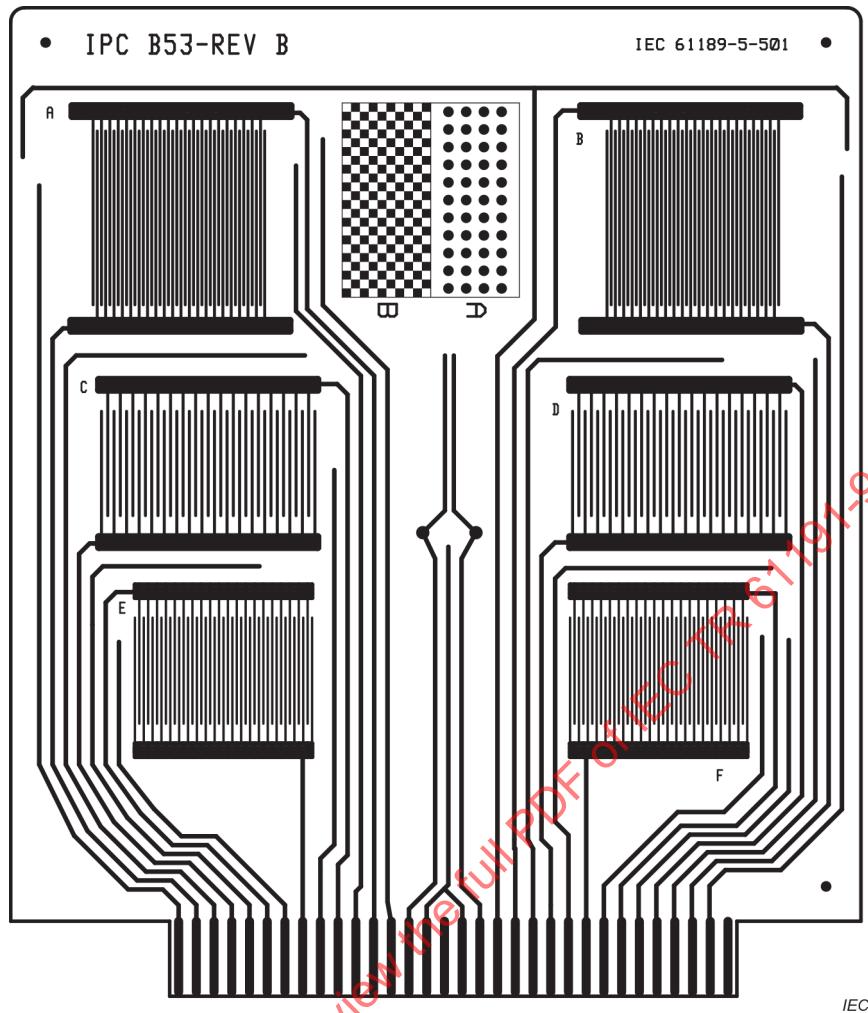
**Figure 29 – Increase in ECM propensity depending on voltage applied ( $U$ ) and Cu-Cu distances ( $d$ ) of comb structures**

High voltage SIR testing with  $> 200\text{V}$  for materials that are used, for example, for e-mobility is currently not covered by international standards. New failure modes like anodic migration phenomena, treeing effects, or partial discharge need to be considered [8], [34]. A simple field strength approach done with low voltage  $< 100\text{V}$  is insufficient for this case.

#### 6.2.4 Influence of distance

The influence of the distance between two open conductors with different potential on the surface insulation resistance is small in the case of equilibrium in absence of ECM promotors (Figure 27). The charge transport through a very thin water film is mainly diffusion controlled and is not significantly accelerated by the electric field strength.

The trend towards miniaturization has replaced the B24 test coupon. For this purpose, the B53 test coupon (listed as TB144 at IEC), as shown in Figure 30, was designed by experts (IEC TR 61189-5-506 [34]). It was done in a way that, on the one hand, different Cu-Cu distances down to 200  $\mu\text{m}$  were realized and, on the other hand, measured SIR values from earlier investigations in databases are still valid (based on 500  $\mu\text{m}$  and 318  $\mu\text{m}$  Cu-Cu distance). The B53 CB also offers the opportunity to assess the influence of spacing on creepage current behaviour during the early stages of material investigations. For this reason, the B53 test coupon is used for first material characterizations.



A/B with 400/200 $\mu\text{m}$  line/space, C/D with 400/500 line/space, E/F with 318/318 $\mu\text{m}$  line/space.

**Figure 30 – Layout of B53 test coupon**

#### 6.2.5 The limit 100 M $\Omega$ and optical inspection

There is no law of nature behind the frequently used 100 M $\Omega$  limit in the evaluation of SIR results. Rather, it is an empirical value of technical experts according to which the risk of failures due to electrochemical migration increases significantly if the surface insulation resistance is close to or below 100 M $\Omega$ . With increasing surface insulation resistance, the robustness of the system in the SIR test against moisture-induced faults rises since fewer charge carriers are also transported.

Nevertheless, even in high-impedance systems which are significantly higher than 100 M $\Omega$  in the SIR measurement, errors due to ECMs can occur (e.g. due to degradation). But even systems in which the leakage current flows through the bulk phase (e.g. solder resists, coatings) and thus have relatively low resistance values (so called volume resistance) close to 100 M $\Omega$  can be humidity robust over their service life without ECM failures.

The distance of the SIR measurement curve from the empirical value of 100 M $\Omega$  and the general trend of the curve (clearly declining over the test duration, strong scattering of the measurement curve with significant downward peaks) are rather risk indicators which are taken into account in the evaluation for material selection. The presentation of the SIR measurement curves is therefore general good practice. The simple classification into a pass/fail criterion is no longer appropriate.

When carrying out SIR measurements, it has proven positive to integrate a reference (e.g. only the CB without SMT solder paste) in parallel in the test. In the case of ECM errors, this allows a decision to be made as to whether the material to be tested is causing the error or whether the test CB has already brought a weakness with it. In the best case, a good reference in the SIR measurement value is one decade higher than the test object.

After each SIR test, an optical inspection of the SIR test structures under the microscope is necessary. Since SIR curves can display anomalies such as unstable course or drops, the optical inspection needs to clarify the physical causes for this. The procedure is described in IPC-9202 [28]. IPC-9203 [43] provides additional guidance.

- a) All specimens are examined under a 10x to 30x microscope using preferred backlighting or UV light. For SIR test patterns that are covered by components, the components need to be carefully removed to examine the covered SIR patterns.
- b) Any pattern that exhibits dendrites that extend more than 20 % of the conductor spacing are considered as fail. In case that dendrites are present, representative documentation of worst case dendrites, typical dendrites, and weakest dendrites by images is required.
- c) Fibres between conductors need to be documented but can be excluded from the rating.
- d) Discoloration between conductors needs to be documented and criticality be rated.
- e) Water spots on test patterns are evidenced by faint staining of the conductors. If a dendrite occurs in a water spot, that data point can be excluded. If more than two points are excluded, the test needs to be repeated with new samples.
- f) Subsurface metal migration needs to be documented and criticality be rated.
- g) Corrosion needs to be documented. Staining can be accepted. Significant corrosion is rated as fail.
- h) Blistering of solder mask needs to be documented and criticality be rated.
- i) Delamination of solder mask or base material needs to be documented and criticality be rated.

In case dendrites and corrosion defects were found during optical inspection, a systematic root cause analysis needs to be performed so that the occurrence of the defect can be explained via a "5x why approach". Typical steps include the use of initially non-destructive analyses for material detection such as SEM-EDS, XPS, TOF-SIMS to clarify the elemental composition. FTIR microscopy can be used to additionally search for local organic impurities. Finally, as a destructive investigation, the use of ion chromatography is also advantageous here, preferably after local extraction in the damage area, to search for unusual ions and deviations from the material fingerprint. Based on all this material information, a logical defect sequence based on physical-chemical fundamentals needs to be established.

### 6.2.6 Influence of materials

The final finish used on B53-like SIR-CBs with standard rinsing processes does not show a significant influence on the SIR value for the reference samples as shown in Figure 27. There are therefore no residues on the printed circuit board which lead to increased creepage currents. The ionic contamination caused by the manufacturing process is low, as high SIR values are achieved. In addition, there are no residues which are for example hygroscopic and would therefore influence the formation of the water film on the surface and, thus, the SIR value.

The understanding of these relationships, as shown in Figure 27, provides the basis for further SIR measurements to characterize materials used for assembly and interconnect technology. By applying additional materials to an SIR-CB, such as solder paste or solder mask, their influence on the SIR value can be evaluated. Thus, within the scope of material characterization, it is now examined whether free ions are present through the additional material and its processing, whether free ions are released, and whether diffusion or degradation processes take place under voltage and moisture load. Clause 7 then looks at the interactions of the different materials when they are combined on a SIR-CBA (B52 approach as described by IPC-9202 [28]). In later case housing effects from bottom terminated components are also considered.

### 6.3 Harmonization of SIR test conditions for characterization of materials for automotive applications

The performance of SIR tests is described very differently in the literature and in standards (e.g. IPC-9201 [33]). The aim of this document for the automotive industry is therefore to provide a common procedure, taking into account current trends and the state of knowledge. Based on the contents of 6.2, the following conditions are recommended.

The conditions in Table 6 can be applied to all different steps of SIR testing for material- and process qualification. Deviations will be explained on a case-by-case basis.

In material- and process characterization investigations, the duration of the constant climate phase can be reduced down to 504 h or 168 h so that extended test times are avoided, and the benefit of internal comparison studies of absolute SIR data can be used.

**Table 6 – Common test conditions for basic material evaluation**

Factor	Parameter	Comment
CB test coupon	B53	See 6.2.4
Voltage	50V	Bias and test voltage with same polarity, see 6.2.3
Polarity	Cathode	In case of covered Cu-lines on the B53-CB (e.g. for solder mask testing), the polarity of the covered line is the cathode (ground, GND). As hydroxyl ions (alkaline pH-value) are formed at this polarity, degradation mechanisms by hydrolysis will be initiated.
SIR interval	≤ 20 min	IPC-TM650, 2.6.3.7 [41]
Evaluation I	> 100 MΩ	See 6.2.5; curves need to be provided so that trend and scattering can be seen.
Evaluation II	Optical inspection	See 6.2.5; optical inspection with documentation of dendrites, corrosion, blistering, delamination, chipping, significant discoloration, water spots etc. is required.
Climate	<b>A constant climate depending on the material to be qualified; cyclic test preferred with B52 CBAs</b>	
Climate const. I	(40 ± 2) °C, (93 ± 3) % rh, 1 000 h (IEC 60068-2-78 [36])	Realistic field load for open CBs, CBAs
Climate const. II	(65 ± 2) °C, (93 ± 3) % rh, 1 000 h	For permanent polymers, coatings, encapsulation (CBs, CBAs)
Climate const. III	(85 ± 2) °C, (85 ± 5) % rh, 1 000 h (IEC 60068-2-67 [37])	See 6.2.2; only special cases as material decomposition can occur.  In the first instance, this test was applied to accelerate the corrosion of aluminum metallization in both integrated circuits and other semiconductor devices in plastic encapsulation. However, when considering the application of the test to other products, it is important that the failure mode or modes are determined, and the appropriate degradation process and test severity selected in response to each failure mode.
Climate cyclic	25°C to 55 °C, 96 % rh, 6 cycles (IEC 60068-2-30 [38])	See 6.2.2; preferred used with B52-CBA subsequently after constant climate tests.  Short term dewing simulation on surfaces.

### 6.4 Different steps of SIR testing

#### 6.4.1 General procedure

The conditions as defined by Table 6 need to be applied on all different steps of SIR testing. Deviations will be explained on a case-by-case basis.

The implementation of SIR tests for material qualification according to the current state of the art is described in detail using the example of the examination of SMT solder pastes (Clause A.1).

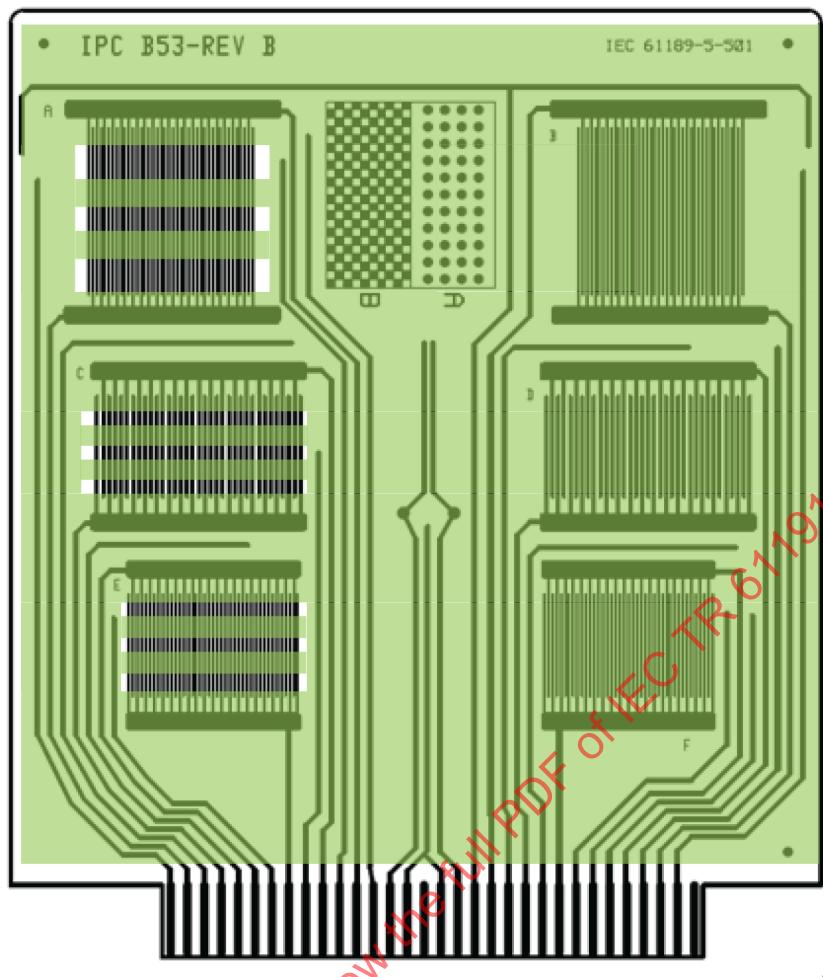
The measurement conditions and procedures as described in Clause A.1 can also be transferred to material qualification of, for example, solder resists or coatings, if the B53 test coupon and material application are adapted to the material to be qualified. Necessary adjustments to the test conditions and the B53 test coupon and its preparation are therefore listed below for the various steps of basic material characterization by SIR.

#### 6.4.2 Base material

SIR tests with modern high Tg base materials typically show no abnormalities. In addition, interactions between a base material and a solder mask, if correctly applied, are commonly not known. Rather, the CAF performance needs to be given priority when selecting base materials. In this context, the creeping current behaviour over the surface (e.g. between PTH land-eyes) can also be evaluated (CAF tests, IPC-TM650, method 2.6.25D [31]).

#### 6.4.3 Solder mask and final finish

The solder mask, with its ability to absorb process chemicals and to release ingredients like photoinitiators in a humid environment, is an important aspect in SIR investigations. For the characterization of solder masks, SIR test coupons need to be used, which have passed through the basic process steps for the production of a printed circuit board up to the final finish. The application of the immersion tin and/or nickel-gold or nickel-palladium-gold process is recommended as this causes the highest impact on the solder mask. Test coupons need to be tested in the delivery condition of the CB, but also after 3 reflow steps without SMT solder paste to simulate thermal stress with initiation of micro crack formation. It is advisable to modify the B53 test coupon in such a way that completely covered comb structures, but also alternating open and covered comb structures are present so that issues by solder mask interface attack are triggered. This also realizes areas with undercut from which process chemicals can bleed during the SIR test. One possible layout is shown in Figure 31. The humidity load "climate I" or "climate II" is chosen, as humidity saturation is fast for a thin solder mask. "Climate III" is not recommended for tests of solder mask types, as rapid polymer degradation with this overstress load is known. Details on how to perform the test are identical to those described in Annex A.



IEC

**Figure 31 – B53 with solder mask, partially covered and fully covered comb structures**

#### 6.4.4 SMT solder paste

The retention of active flux residues (weak organic acids) or their release from the resin matrix is known to be a major factor in triggering ECM from a no-clean process. Tests of SMT solder pastes regarding SIR performance are therefore of great importance. Several documents also describe SIR tests for SMT solder pastes (IPC-J-STD-004 [44], IEC 61189-5-501 [45] and IPC-TM650, method 2.6.3.7 [41]), some of them with different or even contradicting conditions. The aim therefore is to achieve a standardized procedure for SMT solder paste material releases in the automotive industry. The B53 test coupon is, thus, suitable for comparison of existing data based on B24 test coupons and the trend of smaller distances of Cu-lands is also covered. The B53 test layout was developed in a way that flux residues will reliably bridge the comb pattern with 200 $\mu$ m Cu-Cu distance which is seen as critical condition. For some SMT solder pastes, the condition "Climate I" (more critical if volatile ingredients will remain) and the "Climate III" are best suitable (for use in high temperature applications). Details of the sample preparation and the way of SIR measurement is described in Clause A.1. The content of Clause A.1 is also valid for other material and processes qualification investigations based on SIR test equipment with minor adaptations as described in 6.4.2 to 6.4.7.

#### 6.4.5 THT fluxes

With the application of selective fluxes, the greatest risk is that active weak organic acids remain on the assembly, which can trigger the ECM fault in a humid environment. This risk exists especially for flux residues that are not deactivated or rinsed off by the hot solder wave during selective soldering. The criticality of these conditions can also be investigated with the B53 test coupon and a comparison between different selective fluxes is possible. For this purpose, B53 test coupons with or without final finish are provided with an application-typical amount of

selective flux. This can be done by pipette or by spray fluxer as surface loading. A possible overspray into the SMT area can also be simulated by means of spray fluxing around the comb structures of a B53-CB. Subsequently, the activation/deactivation of the flux residues is only performed by the preheating profile specified for the THT flux, without reaching the temperature peak zone of the soldering process. B53 test coupons prepared in this way are tested for SIR performance according to the contents in Clause A.1. For most flux types, the condition "Climate I" is best suitable (more critical as volatile ingredients will remain).

#### 6.4.6 Encapsulations and adhesives

Unforeseen interactions between flux residues and chemical assembly and interconnect technology (e.g. conformal coating, potting, glues, thermal interface materials) are also a frequent cause of failure with electrochemical migration. Outgassing or degrading ingredients could lower the dewing point, could lead to corrosion, or non-predictable chemical reactions take place between residues from different sources. For this reason, polymer materials applied to SIR test coupons but also in combination with SMT resin residues need to be examined in an early phase with regard to their moisture resistance.

The B53 test board can also be used for the initial characterization and qualification of chemical assembly and interconnection materials. In a first step, a full-surface application (e.g. coating) of the comb structures of the B53 CB based only on epoxy base material and etched foil copper can be carried out with the polymer materials to be tested. With the comb structures fully covered, the SIR measurements are performed according to Clause A.1. Since a fast saturation of the polymer and the interface with moisture needs to be aimed at, "Climate II" is recommended. Polymer-typical degradation phenomena, which are often observed at a temperature of 85 °C, recede into the background with "Climate II" condition.

For the next level of material characterization and qualification, the use of more complex B53 test coupons with final finish and resin residues from the SMT process is recommended. Preferred is the application of SMT solder paste (at least type REL0 or ROL0, better would be the SMT solder paste from mass production) on the B53 comb structures as described in Clause A.1, a reflow step and then the strip-like application of the polymer materials on these comb structures contaminated with flux residues.

B53 test coupons prepared in this way are examined for SIR performance according to the contents in Clause A.1. Use of condition "Climate II" is preferred for this case.

#### 6.4.7 Process qualification at CB manufacturer

The manufacturing process of printed circuit boards comprises a large number of process steps, which in their entirety have an effect on the electrochemical reliability of the printed circuit board and thus also on the product. For example, manufacturer-specific pressing or drilling programs during layer buildup have an influence on CAF performance. Manufacturer-specific adjustments in the application process of the solder mask or also in the scope of the rinsing processes after final finishing lead to supplier-dependent differences, even if identical materials are used. The performance of process audits at the CB manufacturer and the implementation of a CAF and SIR audit protocol for each CB supplier is recommended.

The CAF tests are to be based on the specifications of IPC-TM650, method 2.6.25D, [31] which can be supplemented by customer-specific adaptations and extensions in the scope of testing.

SIR tests need to be performed with test CBs that have gone through all manufacturing processes of a multilayer CB assembly. The use of the simple test coupons B24 or B53 without additional plated through holes and solder resist application is therefore insufficient for the process qualification of the CB manufacturer. A suitable SIR test CB is the IPC-B52 layout or customer-specific modifications with comparable requirements (see 7.2). In addition, the introduction of custom patterns as comb structures, which are fully and/or partially covered with solder resist, is of advantage.

SIR testing is carried out for all material and process variants required for the assembly of outer layers, up to the delivery stage to the customer. Deviating from the specifications in Table 6, the following test conditions are appropriate for the process qualification of a CB manufacturer (Table 7).

**Table 7 – Recommended SIR test conditions for basic material- and process release for the outer layer manufactured by a CB supplier**

Factor	Parameter	Comment
CB test coupon	B52 or similar	Complete process of CB fabrication needs be included
Voltage	50V	Bias and test voltage with same polarity, see 6.2.3
Polarity	Anode	Pin A of B52 layout is joined connection of all patterns which is typically the anode (plus pole).
SIR interval	≤ 20 min	IPC-TM650, 2.6.3.7 [41]
Evaluation I	> 100 MΩ	See 6.2.5; curves need to be provided so that trend and scattering can be seen.
Evaluation II	Optical inspection	See 6.2.5; optical inspection with documentation of dendrites, corrosion, blistering, delamination, chipping, significant discoloration, water spots etc. is required.
Conditions	CBs at stage of delivery and after 1x leadfree reflow (1x reflow is rated as more critical as more electrochemical active residues will remain)	
Sample size	10 test samples in total from at least 3 different production lots (one test sample is a single CB and not the number of comb structures or parallel lines on one CB)	
Climate const. I	(40 ± 2) °C, (93 ± 3) % rh, ≥ 504 h (IEC 60068-2-78 [36])	Realistic field load for open CBs and well established in the meantime. The climate condition 85 °C, 85 % rH is additionally considered in CAF testing.

## 7 Comprehensive SIR testing – B52-approach

### 7.1 General aspects

The manufacturing of a control unit includes many process and handling steps with a combination of many assembly and interconnect materials among each other. Interactions of these material combinations can affect the electrochemical reliability in the end-use environment of the product. For example, chemical ingredients can outgas and be deposited on solder joints and open conductors, ionic residues can remain or be released from the soldering process, non-ionic surface-active substances reach the assembly through the application of polymer materials to the assembly or the direct contact of two AIT substances with each other leads to a chemical reaction in a humid environment.

This complexity of interactions is not perceived by simple SIR tests of the individual materials, as described in Clause 6. This Clause 7 therefore focuses on investigations of the electrochemical reliability of material combinations based on the IPC-B52 and IPC-9202 standards (see also [46]). It serves as a representative demonstrator for assemblies which can be realized with these materials and whose robustness under humid load and with phases of short-term dewing is proven.

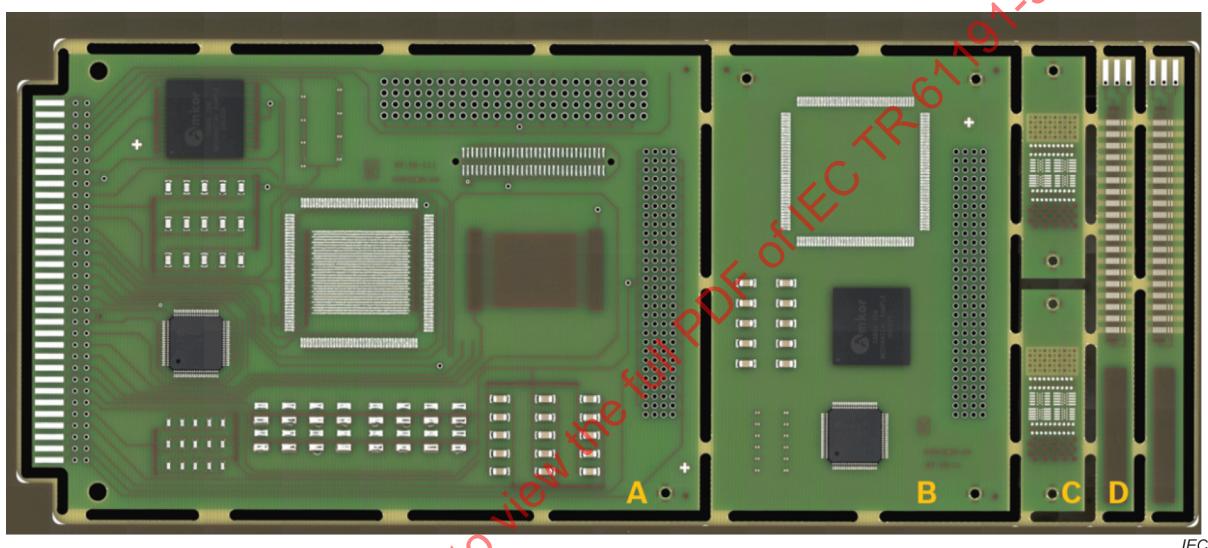
The B52-CBA is a simplification of an assembly whose realization can only be pushed to a certain degree so that the most essential interactions are covered. The totality of all interactions is ultimately verified in active tests of the product under moisture load as part of product validations.

The implementation of IPC-9202 and the IPC-B52 test coupon is rather to be understood as a mindset, according to which materials need to be combined with each other using exactly the processes that will also be used later for mass production. The use of a B52-CBA is therefore an option, since it is standardized, but can be replaced by proprietary release boards if they serve the same purpose. Therefore, also slight adaptation of the B52 test coupon can be used

as well as B52-CBA with minor population, if the deviation can be explained and does not change the overall results of this approach.

## 7.2 The main B52 test board

The history and development of the IPC-B52 standard is documented in IPC-9203 [43], which is the guidance document to IPC-9202 [28]. Even today, the B52 standard still represents a good, typical CBA. A common mix of components and package types is given, with areas for user-specific adjustments. The IPC-B52 test board was originally designed to examine the cleaning performance of the manufacturing process. However, various assembly processes can be used to realize the B52-CBA (e.g. SMT, THT, THR soldering, application of underfill materials, staking adhesives, thermal interface materials, coating). Each of these have the potential for interacting with the materials of construction potentially degrading assembly reliability. Figure 32 gives an overview of a B52-CBA, slightly adapted from standard so that company internal layout rules were realized.



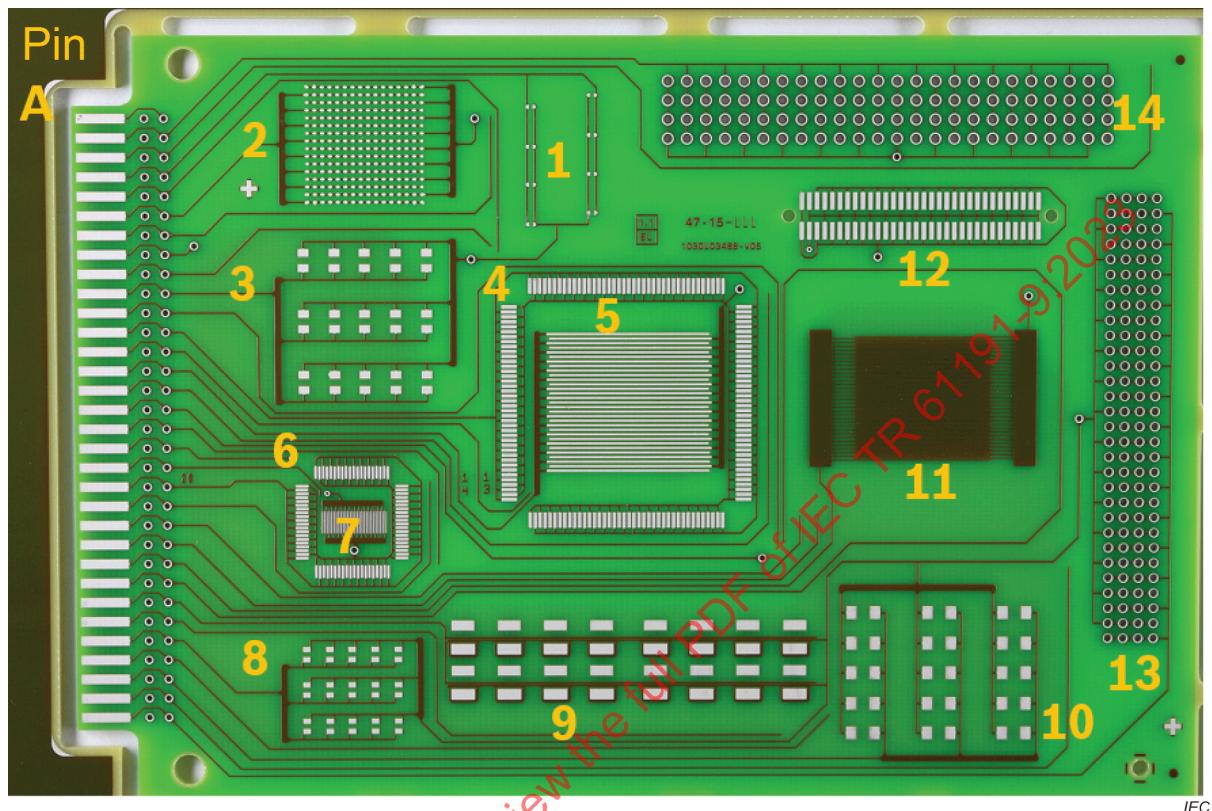
**Figure 32 – B52 CBA after SMT process, layout slightly adapted to fulfil company internal layout rules**

The layout consists of different parts (A: SIR test coupon, B: ion chromatography test coupon, C: solder mask adhesion test coupon, D: mini SIR test coupon). Though originally a part for SIR measurement was designed and a second section for ion chromatography measurement, a different approach has proven advantageous for an automotive typical no-clean approach. For this purpose, it is advisable to build up an excess number of B52-CBAs. In addition to the SIR measurements, an excess number of SIR test coupons is used for ion chromatography. This gives a more typical fingerprint over the placement density and a clearer correlation to the SIR readings than if the measurement is done on the IC coupon. Note that parts that were placed in the climate chamber for SIR measurement are not used for ion chromatography later and vice versa. SIR and IC measurements are done on separate parts that were manufactured at the same time, if used for correlation studies between SIR and IC and for derivation of a fingerprint. In case of failure analysis on tested SIR boards, analysis methods like IC are of course be carried out on the tested specimen (see 6.2.5).

The solder mask adhesion coupon and the SIR mini coupon are also part of the original B52-layout. The use of these parts is optional and not required for SIR investigations as described in this document.

### 7.3 The test patterns

In Figure 33, the different test patterns on the IPC-B52 SIR-part are shown which consists of 14 sections. In Table 8, the different placement positions are explained, and recommendations are given where adaptation possibilities have proven to be advantageous.



**Figure 33 – Pattern of B52 CB, layout slightly adapted to fulfill company internal layout rules**

Pin A is joined connection of all patterns which is typically the anode (plus pole). The other pins are a sequence of alternating shielding lines and the isolated cathode (GND) of each pattern. The contact fingers are based on a 2,54 mm pitch connector. Depending on the SIR test equipment used, rewiring of pin A can be required so that single SIR monitoring is always possible for each pattern.

**Table 8 – List of materials for components with recommendations for minor adaptations**

Figure ID	Component, pattern	Qty	Comment, recommendation
1 <sup>a</sup>	MLCC0402; surface mount ceramic capacitor, 0402 body, $\leq 10 \text{ pF}$ , $\geq 50 \text{ VDC}$ , 5 % tolerance	8	Unchanged; higher capacity of component with $\leq 220 \text{ pF}$ might also work but needs to be verified
2 <sup>b</sup>	BGA, 256 IO, 1,0 mm pitch, isolated, full 16 x 16 array, 17 mm body size	1	Unchanged or change to BGA with 0,8 mm pitch; fully isolated, no internal die or wire; weakness at interposer can lead to pseudo SIR-failures
3 <sup>a</sup>	MLCC0805; surface mount ceramic capacitor, 0805 body, $\leq 10 \text{ pF}$ , $\geq 50 \text{ VDC}$ , 5 % tolerance	15	Unchanged; higher capacity of component with $\leq 220 \text{ pF}$ might also work but needs to be verified
4 <sup>b</sup>	QFP160, 0,65 mm pitch, 28 mm square body, isolated	1	Optional; can be used as lid above the comb structure 5 so that active flux is entrapped under the QFP
5	Comb under QFP160	1	Unchanged if used with QFP165 lid or change to comb structure that can be printed with solder paste without lid. Size could be 318/318 $\mu\text{m}$ line/space as on B53. Application of solder mask dam between single lines recommended
6 <sup>b</sup>	QFP80, 0,5 mm pitch, 12 mm square body, isolated	1	Unchanged, fully isolated, no internal die or wire
7	Comb under QFP80	1	Unchanged, 0,1 8mm space, QFP80 is used as lid above the comb structure so that flux residues can interfere with the comb structure
8 <sup>a</sup>	MLCC0603; surface mount ceramic capacitor, 0603 body, $\leq 10 \text{ pF}$ , $\geq 50 \text{ VDC}$ , 5 % tolerance	15	Unchanged; higher capacity of component with $\leq 220 \text{ pF}$ might also work but needs to be verified
9	SOIC16, 1,27 mm pitch, isolated	4	Changed; size of SOIC is not critical; area could be used for custom patterns
10 <sup>a</sup>	MLCC1206; surface mount ceramic capacitor, 1206 body, $\leq 10 \text{ pF}$ , $\geq 50 \text{ VDC}$ , 5 % tolerance	15	Unchanged; higher capacity of component with $\leq 220 \text{ pF}$ might also work but needs to be verified
11	Area for 2 custom patterns	2	1 or 2 patterns can be integrated in this area like comb structures under solder mask or bottom terminated components (e.g. QFNs)
12	SMT connector, 1,0 mm pitch	1	Optional; can be used unpopulated as comb structure with 0,4 mm space and large flux residue reservoir
13	Plated through holes, 2,0 mm pitch	1	Unpopulated and not soldered used for test of clearance quality. Unpopulated or populated used for test of THT soldering with selective flux contamination. Only required if THT soldering needs to be qualified.
14	Plated through holes, 2,5 mm pitch	1	Unpopulated and not soldered used for test of clearance quality. Unpopulated or populated used for test of THT soldering with selective flux contamination. Only required if THT soldering needs to be qualified.

<sup>a</sup> Within a group of MLCCs, the capacities need to be identical.

<sup>b</sup> It is important that these parts are real dummies without die or internal wiring. Sourcing such components is proving increasingly difficult. Replacing these devices with mechanical dummies based on FR4 CB material can be an option. It needs to be ensured that identical behaviour as from components with mold housing is present.