Solvent Extract Matrix Selection and its Potential Affects on Cleanliness Test Results

By Keith M. Sellers
Managing Scientist
NTS Baltimore

Contents

Abstract

This presentation covers an internal study focused on determining the affects, if any, of the solvent extract matrix selection on the cleanliness results of a given sample.

If differences are present…

- Do these differences show any trends between the different matrices that may help in explaining why one matrix should be used instead of another?
- Should the products' end use have any determining factor in which matrix is selected?

Background

Ion Chromatography (IC) is the predominant instrument used in the determination of overall cleanliness of printed circuit boards (PCB) and assemblies (PCA) in the medical device industry.

Cleanliness testing should be one of many steps in your quality assurance process and its importance is critical as a first step in predicting overall product reliability.

Background

In the past, cleanliness testing was performed through bulk conductivity readings of a “solvent extract”.

- IPC-TM-650, method 2.3.25 – “ROSE” Testing

Currently, the cleanliness test methods called out in most industry specifications use IC…

- IPC-TM-650, method 2.3.28A
- Delphi Delco Automotive Systems
  - methods Q-1000-119 and Q-1000-127
- MIL-STD-883, method 5011.4

Background

The various cleanliness methodologies are designed to cover all aspects of the “industry”.

- PCB’s, PCA’s, components, etc.
- Housing materials, adhesives, underfills, etc.

Within the various IC-based cleanliness test methods, the one striking variation in each is the solvent extract matrix selected for use…

- Deionized (DI) water
- Isopropyl alcohol (IPA) / DI water solutions

Background

From these choices…

- Do the various extract matrices yield different results?
- Do the differences show any trends between the various extract matrices?
  - A specific ion, overall ionic conductivity, etc.

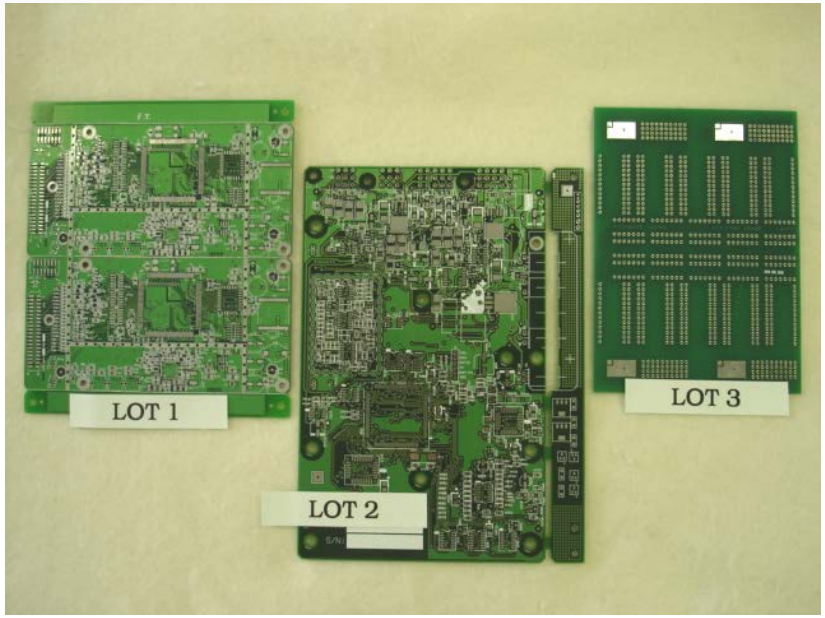
Why is a solution of 75% IPA / 25% DI the “Standard” extraction matrix?

- It was chosen by the US military back in the 1960’s
- Later adopted by IPC for use in its methodologies
- Was able to “dissolve” flux residues at the time

Methodology

For the study, three (3) lots of PCB's from three (3) different PCB manufacturers were used.

- Lot 1 = 76.6 in^2
  - solder mask coated
- Lot 2 = 94.0 in^2
  - solder mask coated
- Lot 3 = 52.2 in^2
  - non-solder mask coated



Methodology

Five (5) PCB specimens were tested from each lot via IC after sample extraction using:

- DI water
- 10% isopropyl alcohol in DI water (10% IPA)
- 75% isopropyl alcohol in DI water (75% IPA)

Each specimen was placed into an individual extraction bag along with an appropriate amount of extract solution.

- Approximately 1.0 – 1.3 mL solution per square inch of sample area (mL/in^2)

Methodology

All bags were heat sealed and placed into a water bath at 80°C for 1 hour.

- Per IPC-TM-650, method 2.3.28A

A fourth set of samples, a set of “rinse” solutions, were prepared using the 75% IPA matrix in a different preparation technique.

- The “ROSE” test – IPC-TM-650, method 2.3.25

All “extract” and “rinse” solutions were analyzed via IC and traditional bulk conductivity methods.

Methodology

For the IC analysis…

- Dionex DX-500 Ion Chromatograph
  - AS11 column for anion analysis
    - NaOH eluents
  - CS12A column for cation analysis
    - MSA eluents
  - A five-level calibration for both anions and cations
  - Self-regenerating suppression

Methodology

From the IC analysis, ionic data was obtained for the following:

- Seven (7) anions
  - Fluoride (F), chloride (Cl), nitrite (NO_2), bromide (Br), nitrate(NO_3), sulfate (SO_4), and phosphate (PO_4)
- Five (5) weak organic acid (WOA) anions
  - Acetate, formate, MSA, adipate, and succinate
- Six (6) cations
  - Lithium (Li), sodium (Na), ammonium (NH_4), magnesium (Mg), and calcium (Ca)

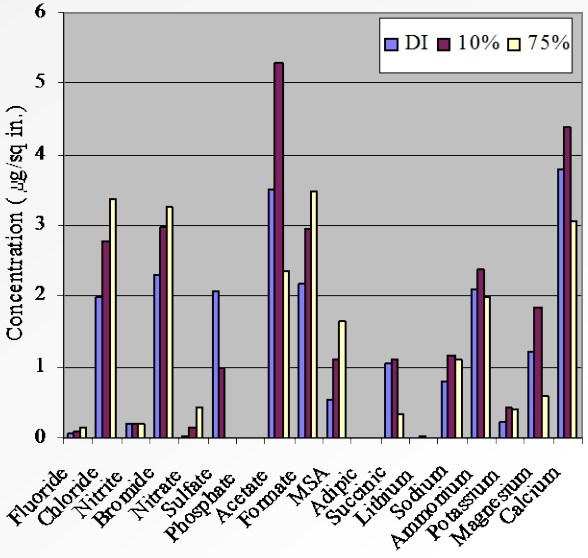
Methodology

For the Bulk Conductivity analysis…

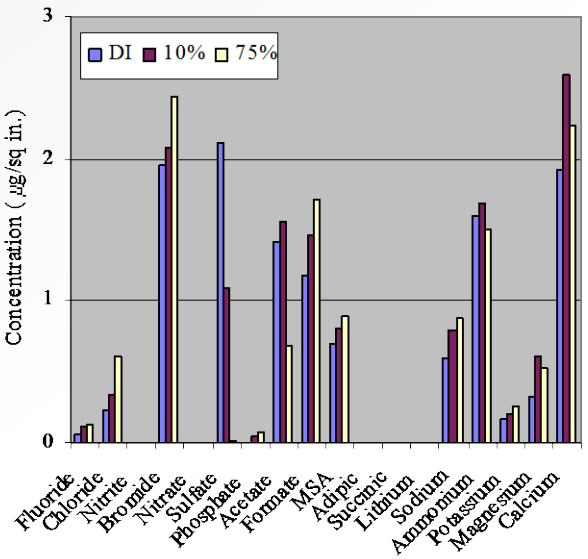
- Omega CDB-410 Conductivity Meter
  - Conductivity cell with cell constant of 0.98 cm^{-1}

Results

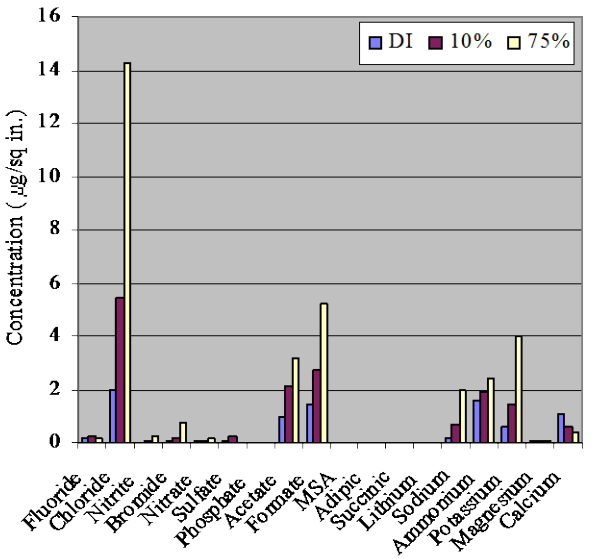
Graphical representation of IC results…



Lot1



Lot 2

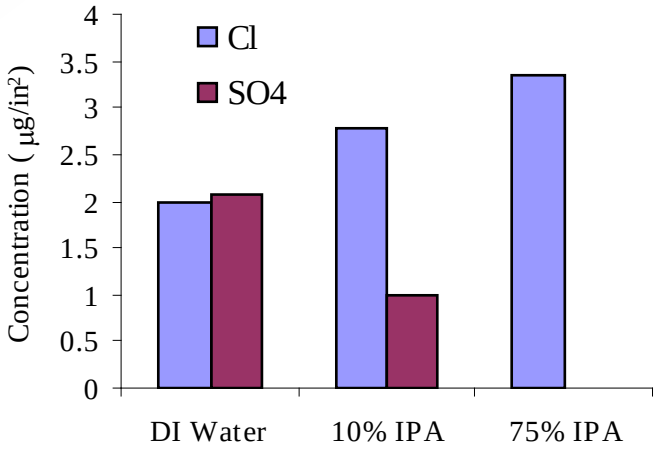


Lot 3

Interpretation

Sample type and processing history may affect the results, however a few trends are still evident and are pertinent.

- The concentrations for chloride, bromide, nitrate, formate, and MSA all increase with increasing IPA concentration in the matrix
- The concentration of sulfate decreases to zero with an increasing concentration of IPA



Interpretation

More trends…

- “Fluctuation” within the data is always present
- For most of the cation species, the ionic concentrations typically increase from the DI water matrix to the 10% IPA matrix and then either hold steady for the 75% IPA matrix or decrease back down towards the DI water matrix value
- The IC results obtained from the “rinse” solutions, in comparison to the “extract” solution results, suggests that the ROSE sample preparation method fails to extract similar ionic levels

Discussion

Ionic cleanliness level requirements vary depending on the industry in which the products are used.

The ultimate reliability desired for a given product is the critical issue.

The following tables summarize some “known” IC test requirements.

- These requirements are not industry-wide standards, but levels established by individual corporations

Discussion

Various IC analysis requirements…

Ions	Bare PCB	Components, Bare PCB, PCA
Fluoride	Each < 0.52	---
Chloride		< 3.0
Nitrite		---
Bromide		< 6.0
Nitrate		< 4.0
Sulfate		< 4.0
Phosphate		< 4.0
Acetate	None specified	< 4.0
Formate		< 4.0
MSA		< 2.0
Adipate		< 30.0
Succinate		
Lithium	Each < 1.00	---
Sodium		< 4.0
Ammonium		< 4.0
Potassium		< 4.0
Magnesium		---
Calcium		---
Total Anions	< 1.61	---
Total WOA	< 5.00	---
Total Cations	< 3.20	---

Table 1 – Medical

Ions	Automotive – HASL PCB	Automotive – OSP PCB	Automotive – Ni/Au PCB
Chloride	< 4.84	< 4.84	< 4.84
Bromide	< 6.45	< 6.45	< 6.45
Na + K	< 12.90	< 25.81	< 12.90
Fluoride Chloride Bromide Nitrate Nitrite Sulfate Phosphate Sodium Potassium Lithium Ammonium Magnesium Calcium	Total < 24.52	Total < 38.06	Total < 24.52

Table 2 – Non-Medical

Ions	Automotive – PCB's Any Finish	PCA's
Fluoride	---	< 3.0
Chloride	< 8.0	< 6.0
Nitrite	---	---
Bromide	< 15.0	< 10.0
Nitrate	---	< 7.0
Sulfate	---	< 10.0
Phosphate	---	< 7.0
Acetate	---	< 300.0
Formate	---	
MSA	---	
Adipate	---	
Succinate	---	
Lithium	---	---
Sodium	---	---
Ammonium	---	---
Potassium	---	---
Magnesium	---	---
Calcium	---	---

Table 3 – Non-Medical

Note: Tables 1 & 3 – 75% IPA, Table 2 – 10% IPA

NTS

Discussion

An example of why extract matrix selection is critical…using the "medical" requirements…

- The "< 0.52 μg/in^2" CI requirement
  - Specimens extracted with DI water and 10% IPA for Lot 2 would meet the requirement
  - All others would fail to meet the requirement
- The "< 3.0 μg/in^2" CI requirement
  - Lot 1 – Specimens extracted with DI water and 10% IPA would meet the requirement
  - Lot 2 – All of the specimens would meet the requirement
  - Lot 3 – Only specimens extracted with DI water matrix would meet the requirement

NTS

Discussion

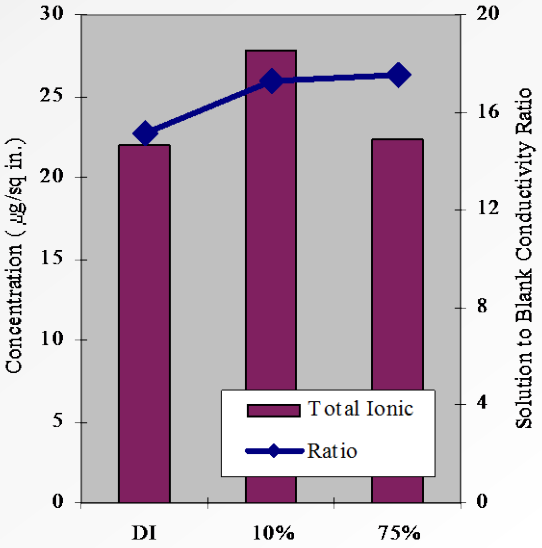
For the IC analysis of the “rinse” solutions…

- None to very little specific ion content was detected
- The specific ion results suggest that the “extraction” procedure is more successful at removing ionic species from the surface of a given specimen than the “rinse” procedure
- From a bulk conductivity standpoint, the “rinse” solutions measured 1,000 to 10,000 times lower in conductivity than the “extract” solutions

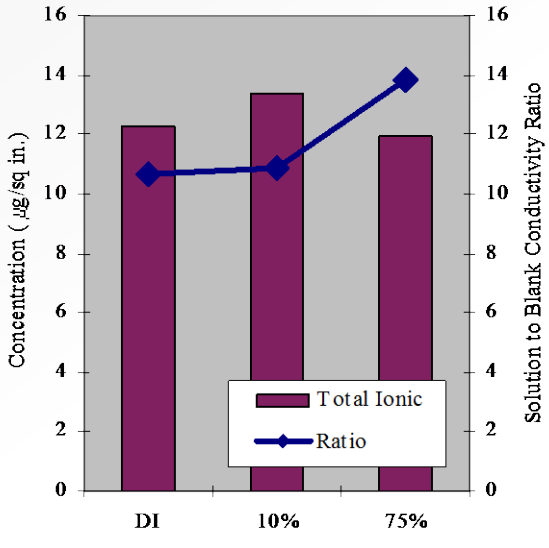
The “ROSE” test does not appear to give an accurate account of the ionic material present.

Discussion

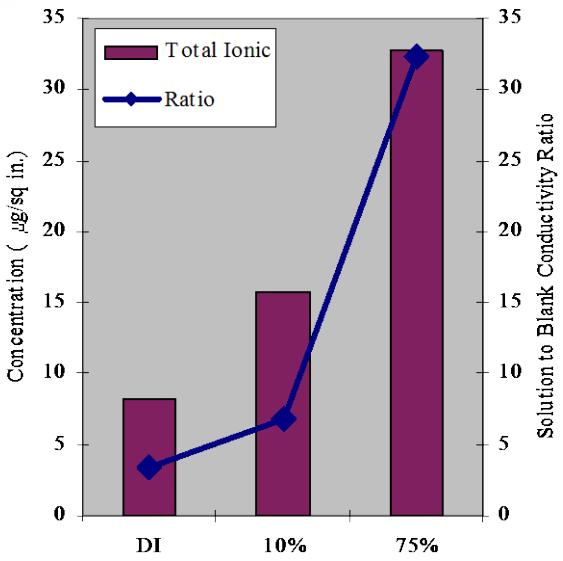
Comparison of IC and Bulk Conductivity results…



Lot 1



Lot 2



Lot 3'

Note: Lots 1 & 2 – Solder Mask Coated, Lot 3 – Non Solder Mask Coated

Discussion

These comparisons reveal a specific trend, not obvious from the IC results alone.

From the results, the addition of IPA to the matrix appears to allow for “extraction” of ions from the material itself and not just “removal” of ions from the material’s surface.

- Lot 3 Results in comparison to Lot 1 & 2 results

The question of each specific ion’s solubility in a given extraction matrix also arises.

Conclusions

The Theory…

- Deionized water removes all soluble ions from the surface of a sample, while the addition of IPA only aids in the extraction of ions from within the sample

With the real-world concern of ionic contamination being its mobility in the presence of moisture, it seems logical that “testing” with DI water is a more realistic choice than testing with an IPA-containing matrix.

Conclusions

Additional testing will hopefully prove this theory correct.

- This detailed experiment will be repeated on materials that are impervious, or relatively impervious, to internal extraction with IPA
  - metal, plastic, and possibly glass

Nonetheless, these findings have suggested that IC testing using differing extraction matrices obtains results which cannot be directly correlated.

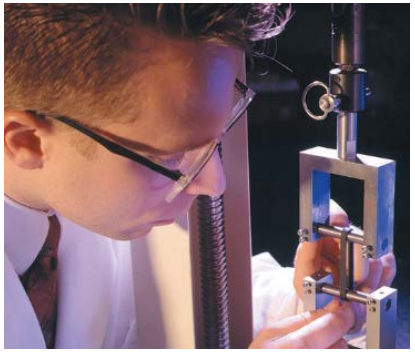
Conclusions

A plausible, and potentially problematic, scenario…

- Samples produce a “passing” ionic cleanliness result based on the extract matrix called out in a particular test method or specification while the same samples would produce a “failing” result using a different extraction matrix – or vice versa

Capabiltiies

- Chemical Analysis
- Electrical/Electronic
- EMC
- Environmental
- Failure/Contamination
- HALT/HASS
- Lead Free/Tin Whisker
- Mechanical
- Printed Circuit Board
- Reliability/Durability
- SLA/Rapid Prototyping
- Thermal Analysis
- Underwriters Laboratories
- Vibration/Shock
- Water Testing
- X-Ray



For more information, please visit www.nts.com

Or contact:

Keith Sellers, Managing Scientist
keith.sellers@nts.com

John Radman, Senior Technical Director
john.radman@nts.com

(410) 584-9099