

### **TEST REPORT FOR:**

SECHAN ELECTRONICS 525 Furnace Hills Pike Lititz, PA 17543

Attn: Jeff Filer

DATE IN:

November 1, 2013

**P/O #:** 

74080

# **QUOTE IDENTIFICATION:**

SechanElectronics, Inc131030

### SUBMISSION IDENTIFICATION:

One (1) partially assembled printed circuit assembly (PCA) sample, identified as "PN8423627001", was submitted for a Black Pad/Contamination Analysis.

We offer our results and observations.

**APPROVED:** 

Keith M. Sellers Managing Scientist





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### **RESULTS SUMMARY**

Visual Examination of the submitted sample found a "residue/discoloration" present at multiple surface mount (SMT) pad locations. Additionally, two (2) plated through-hole (PTH) locations were identified by Sechan Electronics as locations of interest. Based on this information, it was determined that the PTH locations would be identified via Microsection Analysis (including Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDS)), while the SMT pads would be identified via SEM/EDS as well as by Fourier Transform Infrared Spectroscopy (FTIR).

Plating Thickness analysis performed via X-ray Fluorescence (XRF) found the immersion gold and nickel plating layers to be within the "required" ranges as given by IPC-4552.

FTIR analysis performed on the observed "residue/discoloration" revealed only "noise", suggesting that little or no organic material was present. Moreover, the anomaly was not able to be observed within the FTIR microscope. Several scans taken at the approximate location of the "residue/discoloration" all revealed only "noise".

Microsection Analysis on the PTH locations of interest did not reveal any anomalies. Both PTH locations appeared to be fully wetted, with no evidence of dewetting/nonwetting.

"Aerial" SEM/EDS analysis performed on the SMT pads found the pads to be "typical" in appearance for an ENIG-plated sample. Again, the "residue/discoloration" observed during the Visual Examination was not able to be observed via SEM. EDS scans taken were dominated by the presence of gold (Au) and nickel (Ni), with lesser amounts of phosphorus (P) and copper (Cu) also detected. These results are as expected for an ENIG-plated board.

Finally, cross-sectional analysis performed on the PTH locations of interest found the phosphorus-content values for the nickel plating layer to be within the desired 6-11 weight percent range at all locations.

Ultimately, there is no evidence of Black Pad syndrome present. The only anomaly observed was the apparent "residue/discoloration" observed during the Visual Examination, but this anomaly was not able to be observed either via FTIR or via SEM.





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# VISUAL EXAMINATION

### **METHOD:**

Each sample was visually examined using various light sources and magnifications ranging from 10X to 100X.

### **RESULTS:**

Visual Examination of the submitted sample found multiple surface mount (SMT) pad locations which appeared to contain a residue or simple discoloration. This characteristic was seen in multiple locations across the surface of the sample.

Additionally, two (2) plated through-hole (PTH) locations had been identified by Sechan Electronics as specific locations of interest.

Based on the Visual Examination, it was determined that contamination analysis via FTIR and SEM/EDS would be performed on the "residue/discoloration", and that cross-sectional analysis would be performed at the locations identified by Sechan Electronics.

Photographs #1 through #4, below, display overviews of the submitted PCA sample, as well as close-ups of the characteristics described above. Photograph #3 also identified the PTH locations of interest as "Hole 1" and "Hole 2", as identified by Trace Laboratories.





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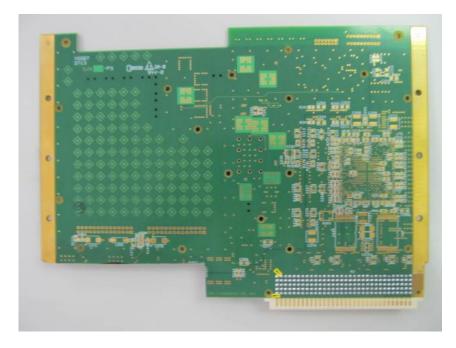
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Photograph #1: Overview of Submitted PCA Sample



Photograph #2: Overview of Submitted PCA Sample



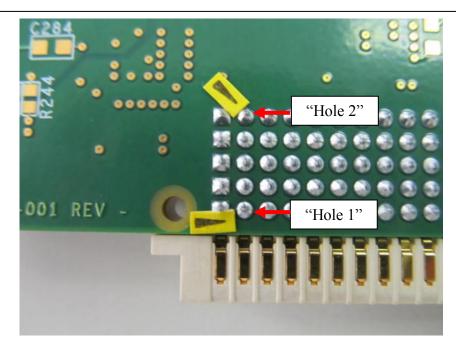


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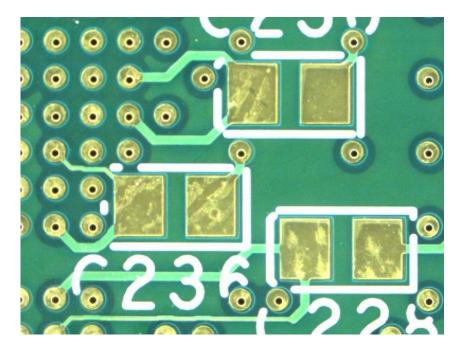


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Photograph #3: Close-up of PTH Locations of Interest as Identified by Sechan Electronics



Photograph #4: Representative Close-up of "Residue/Discoloration" of SMT Pads









# X-RAY FLUORESCENCE (XRF)

### **METHOD:**

The sample was positioned on the measuring stage of the x-ray equipment. The instrumentation was then activated causing a beam of primary x-rays to strike the sample. This caused the elements present to emit characteristic fluorescent x-ray lines that can be identified and quantified. The fluorescent x-rays were detected by a proportional counter and converted to a plating thickness measurement.

XRF analysis was performed at ten (10) random locations across the surface of the sample.

### **RESULTS:**

The results of the XRF analysis are found in Table 1, below.

From IPC-4552 – Specification for Electroless Nickel / Immersion Gold (ENIG) Plating for Printed Circuit Boards, the immersion gold thickness should be a minimum of  $1.97\mu$ in (0.05 µm) and the electroless nickel thickness should be 118.1 to 236.2 µin (3 to 6 µm).

Each location analyzed was found to meet these requirements.





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Location	Plating Thickness		
	Au	Ni	
R69	4.91	155.9	
C76	3.97	171.6	
R8	3.71	166.4	
R28	4.50	148.2	
C2	3.92	169.1	
C276	3.95	149.4	
R239	4.67	140.3	
C278	4.09	140.1	
L201	4.16	155.4	
R237	3.89	149.9	
Average	4.18	154.6	

### Table 1: Results of XRF Analysis (microinches)









# FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

### **SPECIMEN PREPARATION:**

A representative surface mount (SMT) pad which appeared to display the "residue/discoloration" observed during the Visual Examination was analyzed in-situ via micro-FTIR. An IR-reflective brass plate was used as a background.

# **METHOD:**

A Fourier Transform Infrared Spectrometer was used to collect and process infrared wavelength absorbance / transmission spectra of the specimens. These spectra are created when a molecule converts infrared radiation into molecular vibrations. There are two types of molecular vibrations, stretching and bending. These vibrational movements create bands in a spectrum that occur at specific wavelengths (cm<sup>-1</sup>). Each wavelength is dependent on a number of things. Most importantly of which are the mass of the atoms present, the force constants of the bonds present, and the geometry of the molecule present.

Infrared spectra can help in identifying the chemical composition and / or bonding present in an unknown molecule. The greatest use of infrared spectroscopy is that the tool provides structural information about the presence of certain functional groups that are present in the sample.

The spectrometer radiates a broad band of infrared light through the specimen. Depending on the bonding present, the light will be absorbed, transmitted, or reflected at various wavelengths. From the spectrum produced, information about the bonding present is obtained from the location of group frequency peaks. Also, most spectra contain additional "fingerprint" peaks that are unique to a particular molecular structure.

All molecules have a unique spectrum in IR. The "fingerprint" region, below  $\sim 1500 \text{ cm}^{-1}$ , is typically used to identify the molecule or molecules present. This region is different when comparing two molecules even with identical group frequency peaks. The group frequency peaks are typically strong and convey the presence of numerous organic functional groups. These include, but are not limited to, alcohols (-O-H), amines (-N-H), carbonyls (-C=O), etc.

The spectrometer has the ability to analyze samples in two distinctly different ways. The methods are referred to as bench analysis and micro-FTIR analysis.

Bench analysis consists of forming the specimen to be analyzed into a thin film between two zinc selenide (ZnSe) windows. The infrared beam is then passed through the windows and a spectrum is recorded. This method is typically used on homogenous liquid samples.









Micro-FTIR analysis consists of using a microscope attachment on the spectrometer to analyze areas as small as 25 microns in diameter. This technique is particularly useful in analyzing small areas of surface contamination (given the proper sample geometry) or microscopic materials. Specimens can be analyzed in reflective or transmission mode with this method. In reflective mode, the infrared beam is passed through the sample and then reflected off of a reflective IR plate and passed back through the sample where it is detected and analyzed. In transmission mode, the infrared beam is passed through the sample where it is detected and analyzed. Both modes allow for in situ analysis, limited by the specimen's geometry. In addition, this type of analysis is typically performed on specimen residues or when a precise location analysis is required.

A specimen's spectrum can then be compared with spectra of standard materials from an IR reference library or compared with customer-supplied references. Upon analysis of some specimens, a spectrum is obtained that is nothing more than an erratic signal with no distinguishable features. A spectrum of this type is referred to as a noise spectrum. Noise is created in a specimen's spectrum when the absorbance by the specimen of a particular frequency is extremely low. With extremely low absorbance values, any minor increase or decrease in the absorbance of a frequency can create what appears to be a peak. Primary absorbance peaks lower than approximately 0.4 are generally considered indistinguishable in comparison to any noise present. Additionally, low absorbance values of primary absorbance peaks cause comparisons and identifications to be extremely difficult.

The photograph below shows the FTIR unit.







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### **RESULTS:**

The "residue/discoloration" was not able to be observed in the FTIR microscope. With that, multiple scans were taken at the approximate location of the "residue/discoloration" as seen visually.

The spectra obtained were found to consist basically of "noise" with no apparent organic material detected. Characteristic carbon-hydrogen bonding peaks were not detected and neither were any other distinguishable characteristic peak structures.

This result suggests that no organic residue is present on the pads of the sample.

A representative spectrum obtained can be found attached at the end of the report.





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### **MICROSECTION ANALYSIS**

### **METHOD:**

The area of the sample containing the plated through-hole (PTH) areas of interest was removed from the whole and was mounted in an encapsulating mold, which had been treated with a release agent, such that the sample could be viewed in cross section. (This release agent helps to remove the cured microsection from the mold).

A two-part liquid-to-liquid polyester base resin compound was then carefully mixed and poured over the prepared sample. The compound was cured at room temperature for approximately twenty-four hours. After curing, the microsection was removed and the following procedure was utilized to prepare and evaluate the sample:

- a. The microsection was ground on 180 and 600 grits to the intended plane of evaluation. Running water was used during the grinding procedure, to prevent overheating of the sample.
- b. A 6-micron diamond slurry was used to ensure the removal of the scratches created from the grinding process.
- c. A final polish using a 0.6-micron alumina was used to rid the sample of all fine line scratches.

### **RESULTS:**

No anomalies were observed as a result of the microsection analysis. Each PTH location analyzed appeared to have fully wetted.

At the conclusion of this analysis, the locations were analyzed via SEM/EDS as described in the section below.

Photographs #5 through #8, below, display cross-sectional overview and close-up images of each PTH location analyzed.





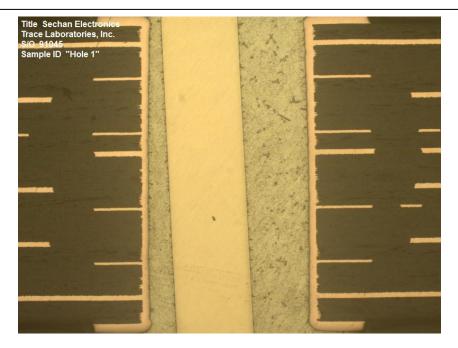
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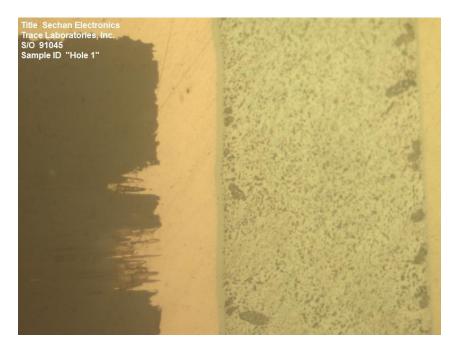
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Photograph #5: Microsection Overview of PTH Location "Hole 1"



Photograph #6: Microsection Close-up of PTH Location "Hole 1"

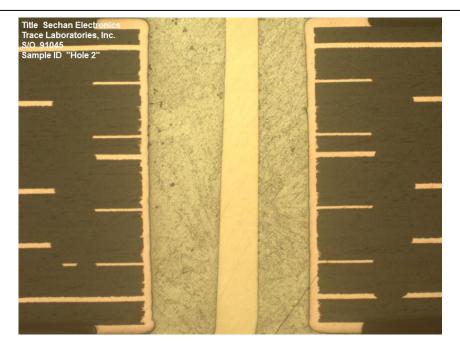








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Photograph #7: Microsection Overview of PTH Location "Hole 2"



Photograph #8: Microsection Close-up of PTH Location "Hole 2"









# SCANNING ELECTRON MICROSCOPY / ENERGY DISPERSIVE X-RAY SPECTROSCOPY (SEM/EDS)

# **SPECIMEN PREPARATION:**

Initially, an "aerial" analysis was performed by removing a representative portion of the sample containing the "residue/discoloration" observed during the Visual Examination. The sample was mounted directly to an SEM stage using electrically conductive tape, which helps to ground the object and aids in stabilizing the SEM image.

Additionally, the encapsulated sample, prepared as described in the Microsection Analysis portion above, was similarly mounted.

# **METHOD:**

SEM allows an area to be examined at extremely high magnifications. SEM produces images of high resolution and detailed depth of field. Surface structures, anomalies, and contamination can be detected and isolated for further analysis.

The specimen is located within a vacuum chamber at the bottom of the SEM column. An electron source is located at the top of the column. The source produces electrons that pass through the column and are incident upon the specimen. The electron beam is directed and focused by magnets and lens as it approaches the sample. The beam swings across the sample causing some of the electrons to be reflected by the specimen and some to be absorbed. Specialized detectors receive these electrons and process the signal. The three different detectors used are referred to as: secondary electron, backscatter, and x-ray.

The secondary electron detector converts the reflected electrons into a signal that can be displayed on a monitor or captured digitally. Photographs are black and white because the electrons being detected are beyond the light spectrum. This detector is primarily used to observe the structure of a specimen.

The backscatter detector reads the pattern in which the electrons are being reflected and displays and image for observation and or photography. Denser areas in the specimen will absorb more electrons than a less dense area. Thus, for example, carbon (C) will appear as a much darker area than lead (Pb). The x-ray detector (EDS) is used to qualitatively and possibly quantitatively determine the elemental composition of any particles or suspect areas, which were observed using the secondary and backscatter detectors. As the electron beam strikes the specimen, the electrons within the atoms of the specimen are elevated to an excited state. As the electrons return to their ground state, a characteristic x-ray is emitted. The EDS detector observes these x-rays as they are emitted from the specimen. EDS can detect elements





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from carbon (C) to uranium (U) and in quantities as low as 1.0%. The area of analysis for a specimen can be adjusted based on the magnification at which the specimen is being observed.

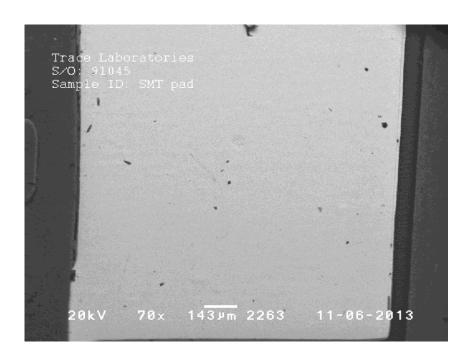
### **RESULTS:**

### "Aerial" Analysis:

The "residue/discoloration" observed during the Visual Examination was not able to be observed visually via SEM, just as it was not able to be observed visually via FTIR. With that, the pad had a "typical" appearance of an ENIG-plated surface.

From an elemental standpoint, the sample was dominated by the presence of gold (Au) and nickel (Ni), with small amounts of phosphorus (P) and copper (Cu) also being detected. The elements detected would be those expected for an ENIG-plated board pad.

Photographs #9 and #10, below, display the SEM images obtained. The EDS can obtained can be found attached at the end of the report.



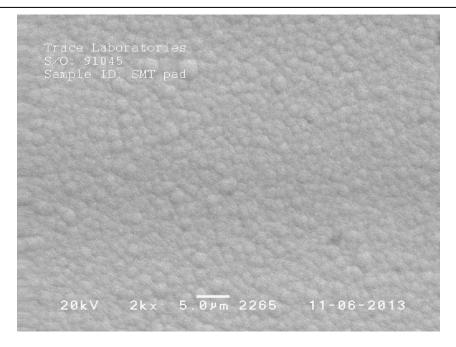
Photograph #9: SEM Overview Image of Representative SMT Pad











Photograph #10: SEM Close-up Image of Representative SMT Pad

# Cross-sectional Analysis:

EDS analysis was also performed on the microsection samples prepared from the locations of interest by focusing solely on the nickel layer at each selected location to determine the presence of phosphorous in different regions of the layer. The analysis was completed by obtaining an EDS spectrum at a location in the center of the nickel layer, identified as "bulk", and obtaining an EDS spectrum at a location near the top of the layer, identified as "surface". Comparison of the phosphorous levels at these locations can help in determining whether or not Black Pad Syndrome is present.

Table 2, below, summarizes the results obtained for each location; quantification was completed based only on nickel and phosphorous. The results found in the table are expressed in weight percent (wt%) and are representative of the pads found at each location. (Note: The values reported in the table below have been "corrected" based on the analysis of ENIG "standards" with known levels of phosphorous, as determined by ICP-OES.)





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Location ID	"Bulk" Layer		"Surface" Layer	
	Р	Ni	Р	Ni
<b>"Hole 1"</b>	6.03	93.97	6.80	93.20
<b>"Hole 2"</b>	6.12	93.88	6.12	93.88

# Table 2: Interfacial EDS Results, in wt%

\* TR signifies "Trace amount" ( < 1 wt. %)

Confirmation of the presence of Black Pad Syndrome is typically characterized by evaluating the phosphorous concentrations at the "surface" layer and the "bulk" layer. In this case, Black Pad Syndrome does not appear to be present at either of the locations analyzed. Specifically, high phosphorous levels in relation to the nickel (> 11 wt%) and/or an increasing phosphorous concentration gradient are the key elemental characteristics present within a Black Pad "infected" board. Neither of these characteristics was observed for the submitted sample.

On the other hand, a lack of phosphorous content in the Ni-layer (< 6 wt%) can lead to a nickel corrosion issue, which could inhibit proper solderability. Phosphorous is present in the electroless nickel-plating as a "corrosion-inhibitor". If the P-level is not near the "desired" range (approximately 6-11 wt%), the electroless nickel layer will be more susceptible to corrosion thus leading to solderability and proper joint formation issues. Again, this characteristic does not appear to be present at the locations analyzed, though most measurements taken were near the "lower end" of the desired range.

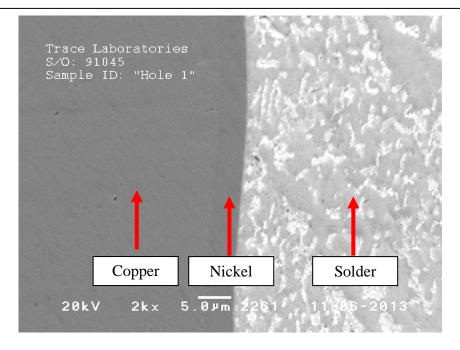
Photograph #11, below, displays a representative SEM image which identifies the plating layers of interest. All of the EDS scans obtained can be found at the end of the report.











Photograph #11: Representative SEM Image Displaying Plating Layers of Interest





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Trace Laboratories, Inc. certifies that the test equipment used complies with the calibration test purposes of ISO 10012-1, ANSI/NCSL Z540-1-1994, and MIL-STD-45662A and that the data contained in this report is accurate within the tolerance limitation of this equipment.

All test procedures detailed within this report are complete. The results in this report relate only to those items tested. If any additional information or clarification of this report is required, please contact us. This test report shall not be reproduced except in full, without the written approval of Trace Laboratories, Inc.

Thank you for selecting Trace Laboratories, Inc. for your testing purposes.

### **PERFORMED BY:**

Daniel D. Phillips Engineer

ATTACHMENTS:	one (1) FTIR scan five (5) EDS scans one (1) Spectroscopy Log sheet
SAMPLE DISPOSITION:	samples returned to Sechan Electronics
DATE COMPLETED:	November 6, 2013

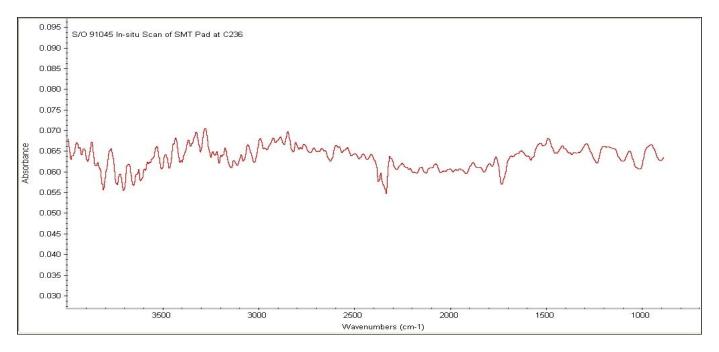






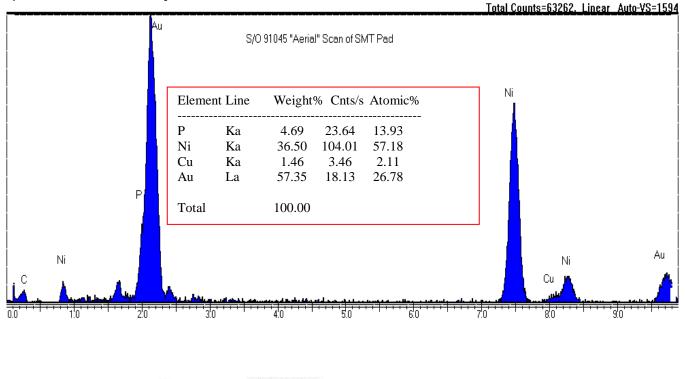






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Range:20 keV

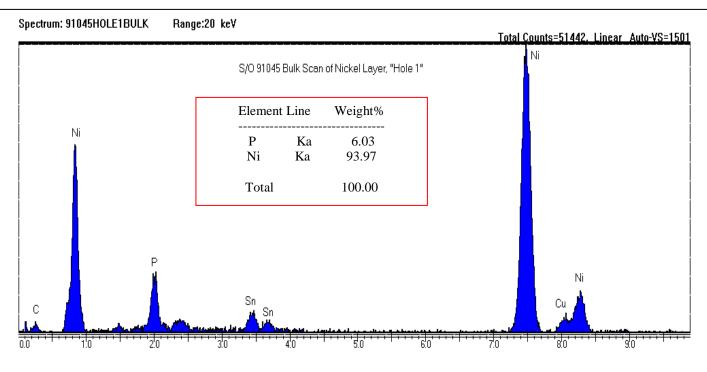


Certificate No. TLE01A

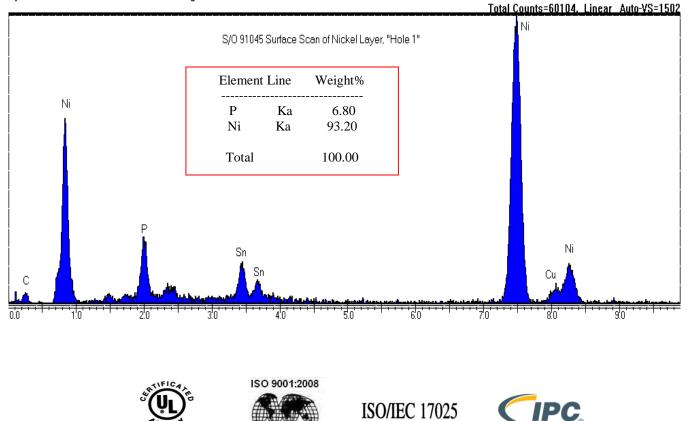








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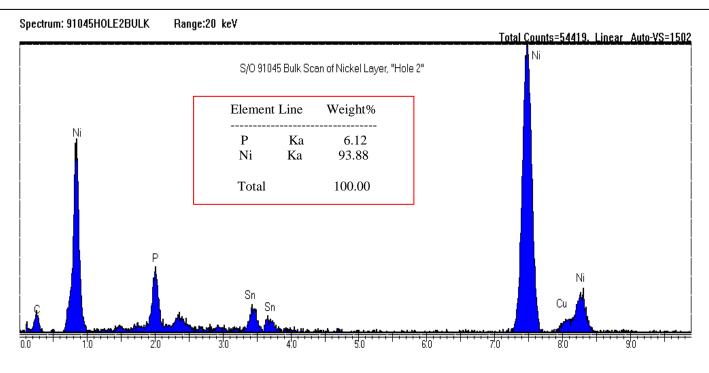
COMPLIANT

TLE01A

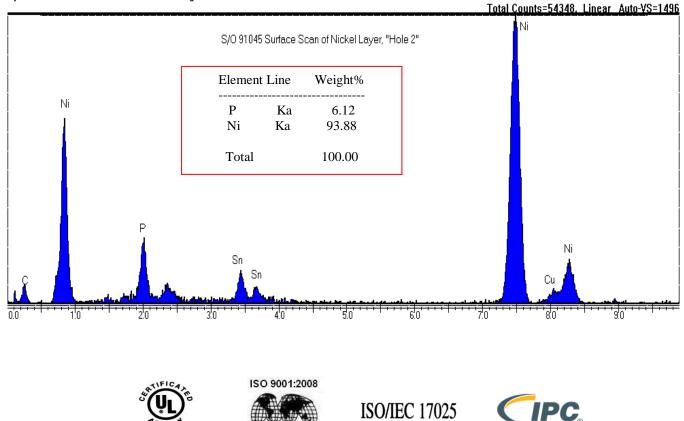
Member

S/O 91045





### Spectrum: 91045HOLE2SURFACE Range:20 keV



COMPLIANT

o. TLE01A

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Member

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Form 313

Revision 130515

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S/O: 91045 EQUIPMENT USED: CA-01, CAL-17

MS-15, MS-16

#### SPECTROSCOPY TEST INFORMATION

#### SAMPLE IDENTIFICATION:

"PN8423627001"

SCAN / SPECTRUM INFORMATION:

Technique	Date	Initials	File Name(s) *
FTIR	11/6/2013	DDP	91045_C236
SEM/EDS	11/6/2013	DDP	91045*.*

\* NOTE: The \*.\* notation will be used when all files starting with the same 5/O number are performed by the same technician.

NOTES:

Form Originated: <u>KMS</u> Date: <u>5/15/13</u> Form Approved: <u>JR</u> Date: <u>5/15/13</u> Uncontrolled Document - See Original in Quality/Office Manager's Office





