



Contamination, Transportation or Transmutation in LENR Material Analyses

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1. Raison d'être
2. Material Assay Techniques
 1. Non-nuclear
 2. Nuclear
3. Two Electrolytic Experiments
 - University of Rochester: Palladium Deuteride
 - Naval Research Laboratory: Thorium & Uranium Deuteride
4. Contamination, Transportation or Transmutation?
5. Conclusions



The hallmark of Low Energy Nuclear Reactions is excess power. LENR excess power is often correlated with helium production or leaves tritium “ash”.

Other nuclear reaction products may be detectable during or after device operation including evidence of transmutation from capture, activation, spallation or fission.

The difficulty is in knowing the background when the experiment began and as it ran to correctly identify any new reaction products. These products are necessary to elucidate the nuclear exit channels.

This talk concentrates on post-run material assay techniques and their use in two independent electrolysis experiments.

Non-Nuclear Material Analyses

- *XPS (X-ray Photoelectron Spectroscopy)
 - Sensitive to surface only (10 nm), measure binding, oxides, 100 ppm sensitivity, high vacuum
- *SEM/EDX: Scanning Electron Microscopy/Energy Dispersive X-ray Analysis
 - Elemental B to U, surface or near surface, parts/thousand sensitivity, elemental x-ray interference
- *ICP-OES: Inductively Coupled Plasma-Optical Emission Spectroscopy (or AEC: Atomic Emission Spectroscopy)
 - Elemental Li to U, < ppb, some elemental interferences, sample prep reduces sensitivity
 - Hydride interference
- ICP-MS: Inductively Coupled Plasma-Mass Spectroscopy
 - Isotopic Li to U, < ppb, “bulk” sample prep reduces sensitivity, carrier gas and charge interference:
 - Isobaric interference: ^{64}Ni and ^{64}Zn , ^{40}Ar (feed gas) and ^{40}Ca , et al.
 - ^{67}Zn and $^{134}\text{Ba}^{++}$
 - Hydride and Argand interferences
 - Some (Finnegan) favor higher A isotopes for a given Z
- TOF-SIMS: Time of Flight-Secondary Ion Mass Spectroscopy
 - Isotopic H to U high sensitivity, approximately 10 ppb, surface, qualitative, small sample area, high vacuum
 - Hydride interference

Nuclear Material Analyses

- **Liquid Scintillator, alpha-beta Spectroscopy**
 - **Low specificity, high sensitivity, detects tritium in aqueous solution, range from a few keV to 2 MeV**
 - **Low spectral resolution**
- **X-rays, and γ : HPGe, LaBr₃, NaI, GM Tube (β):**
 - **High specificity, HPGe has high spectral resolution, *background interference***
- **NAA: Neutron Activation Analysis:**
 - ***isotopic, high sensitivity, quantitative < ppm, matrix interferences, not all isotopes, may become radioactive***
- **Solid State Nuclear Track Detectors (CR-39)**
 - **Integrating detector, works with low flux**
 - **Detect charged particles > 100 keV, nearly 100% efficiency**
 - **$10^{-3} - 10^{-4}$ neutron detection efficiency**
 - **Works best in vacuum, used in aqueous electrolytes, < 50 °C**



Experiment 1

Deuterated Pd using D₂O and Li₂SO₄

- Tanaka Precious Metals Pd plate¹
- 2.54 cm x 2.54 cm x 1 mm, split into 1 cm x 2.54 cm x 1 mm
- Cell run for a couple weeks at the University of Rochester, Department of Chemical Engineering
- Reaction gas collected in Summa canisters and analyzed with ambiguous results.
- SEM/EDX analyses conducted at NASA GRC

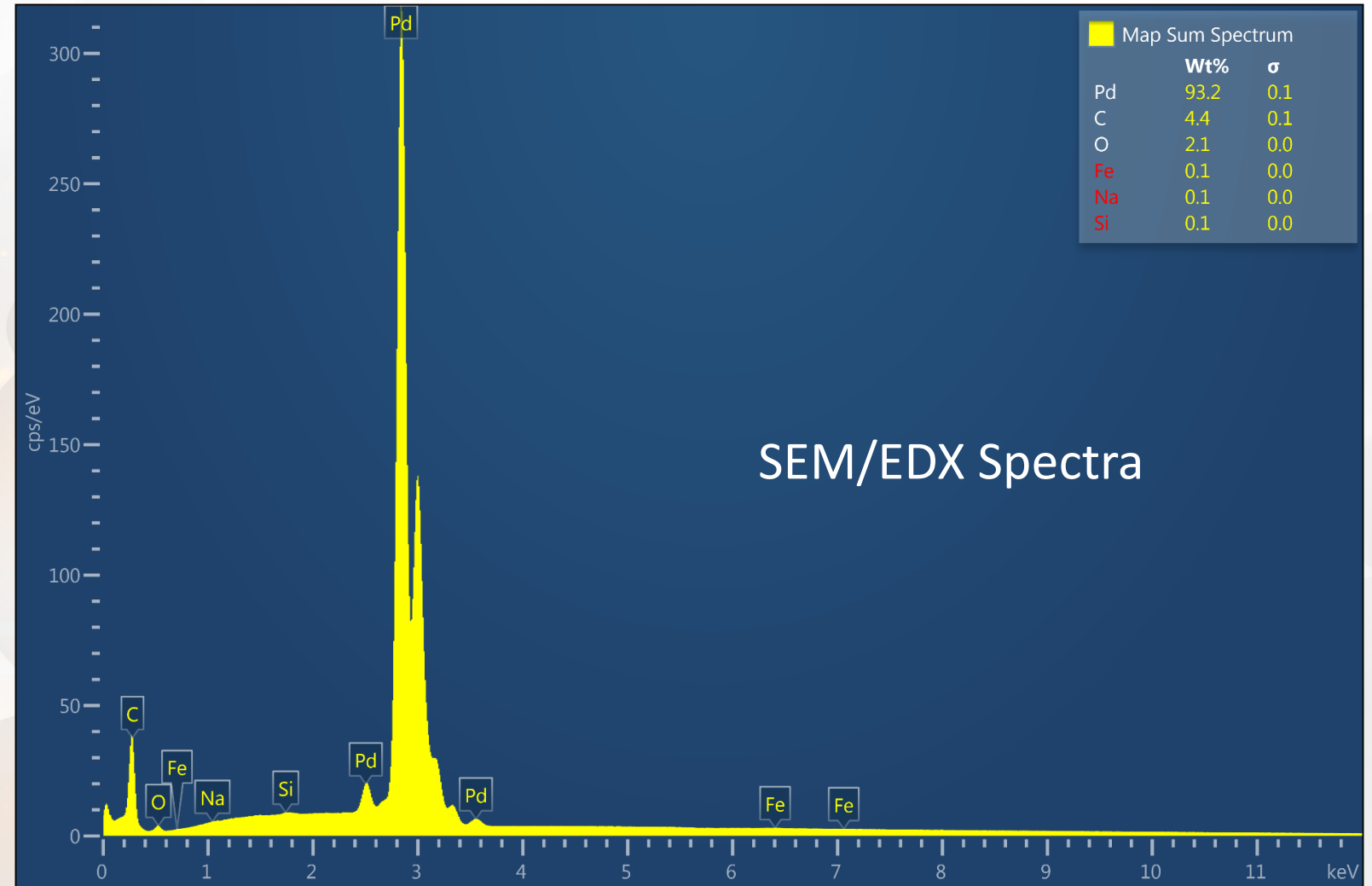
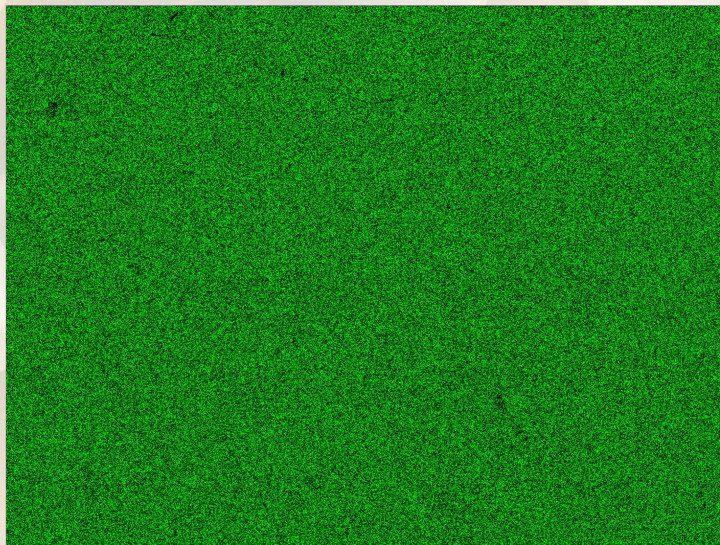
¹. Dr. Gene Mallove provided two Tanaka Precious Metals Pd plates.

U of R: Unused Tanaka Precious Metals Pd

Electron Image 1



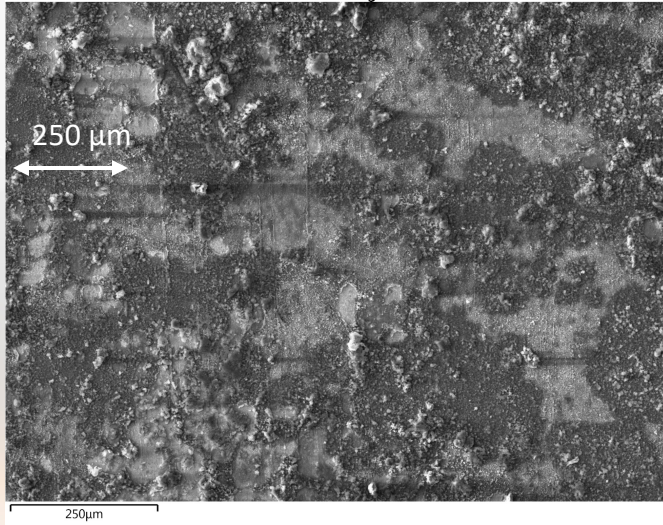
Pd L series



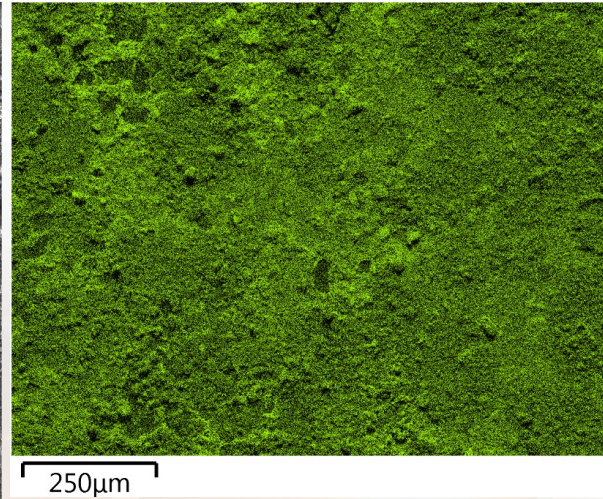
Lines from polishing indicate a source of initial contamination!

U of R: Deuterated Pd using D₂O and Li₂SO₄

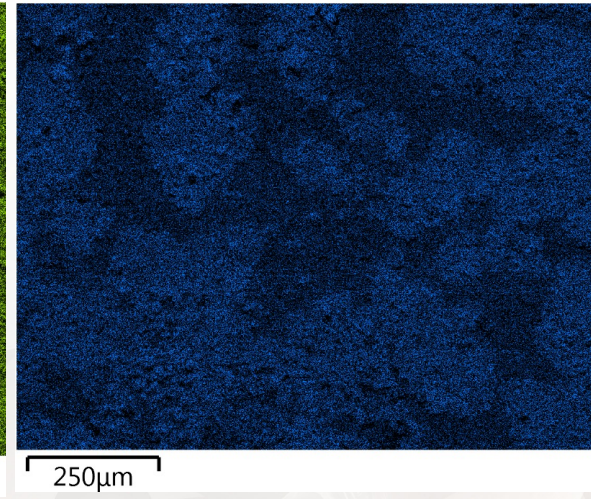
Electron Image 2



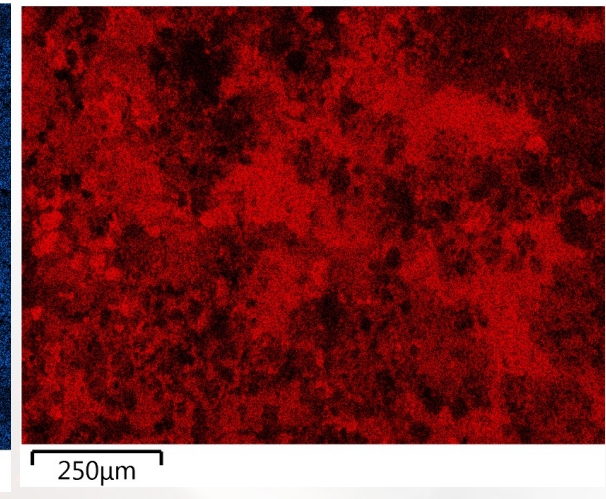
O K series



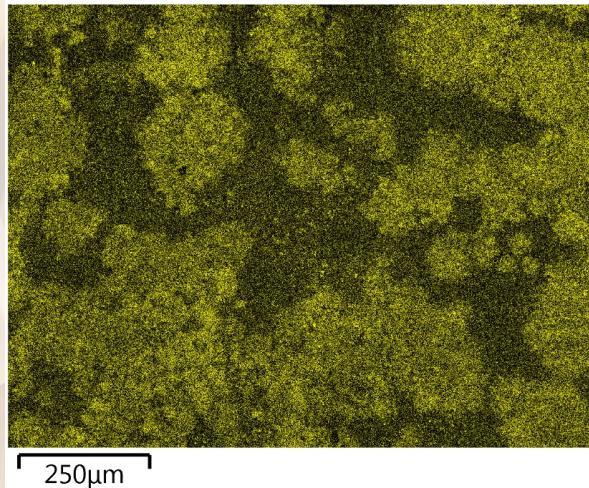
C K series



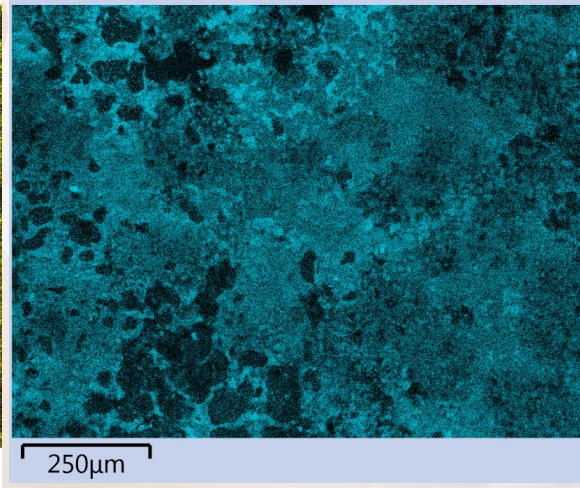
Sn L series



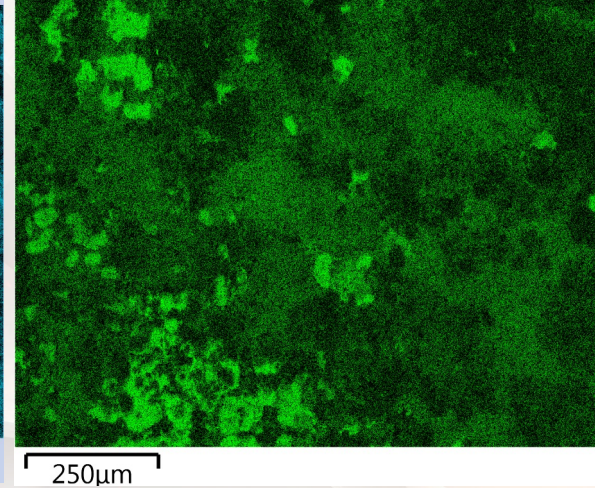
Pb M series



Si K series



Pd L series

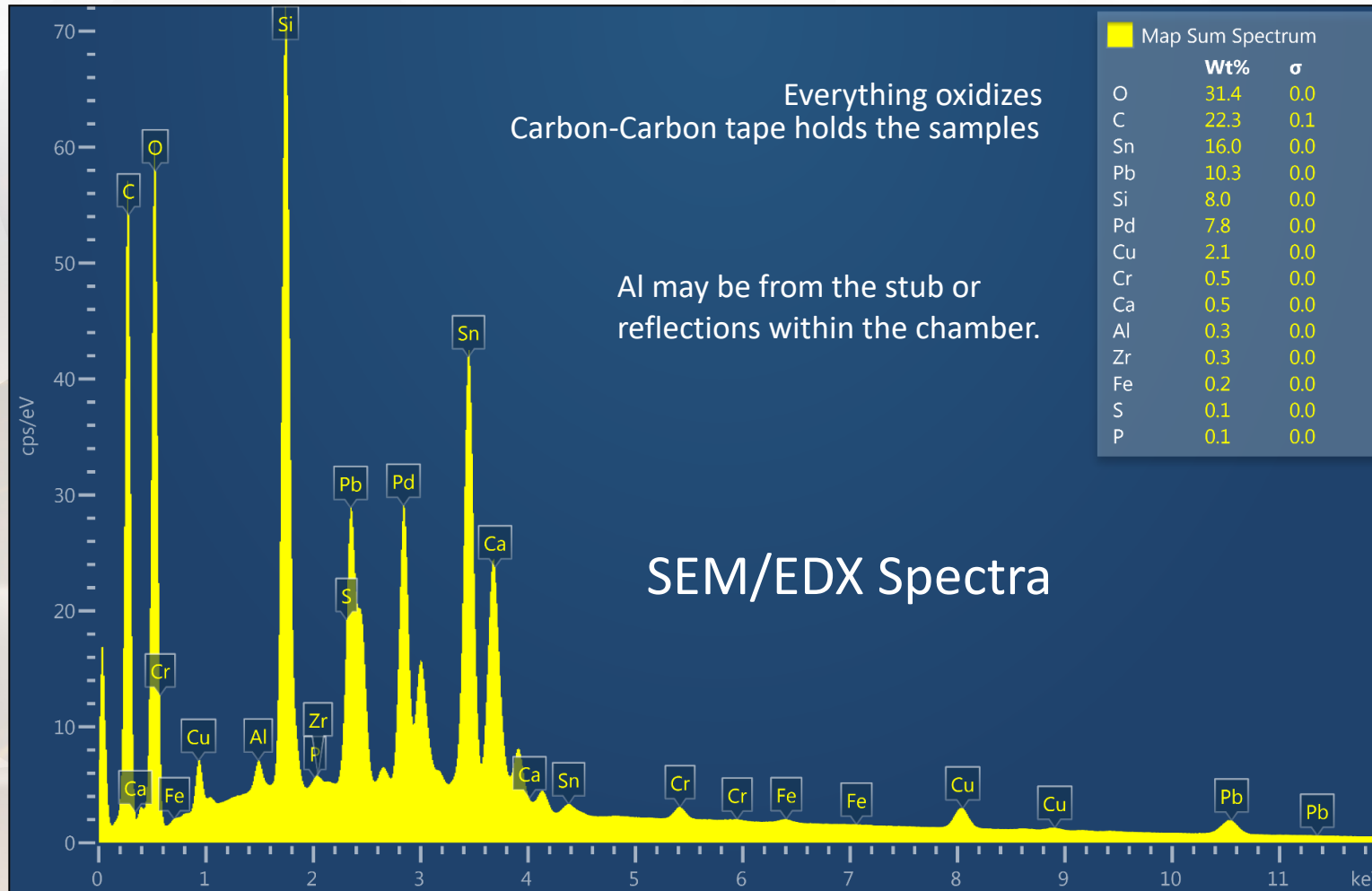


Element	% Before	% After
O	2.1	31.1
C	4.4	22.3
Sn	NA	16.0
Pb	NA	10.3
Si	0.1	8.0
Pd	93.2	7.8

Used Pd

U of R: Deuterated Pd using D₂O and Li₂SO₄

Electrolyzed Tanaka Pd plate in Li₂SO₄



Comparison before and after

Element	% Before	% After
O	2.1	31.1
C	4.4	22.3
Sn	NA	16.0
Pb	NA	10.3
Si	0.1	8.0
Pd	93.2	7.8

O, C, Sn, Pb, Si, Pd, Cu, Cr, Ca, Al, Zr, Fe, S, P

Another Site, #2

Electron Image 3

“hill” causes shadow

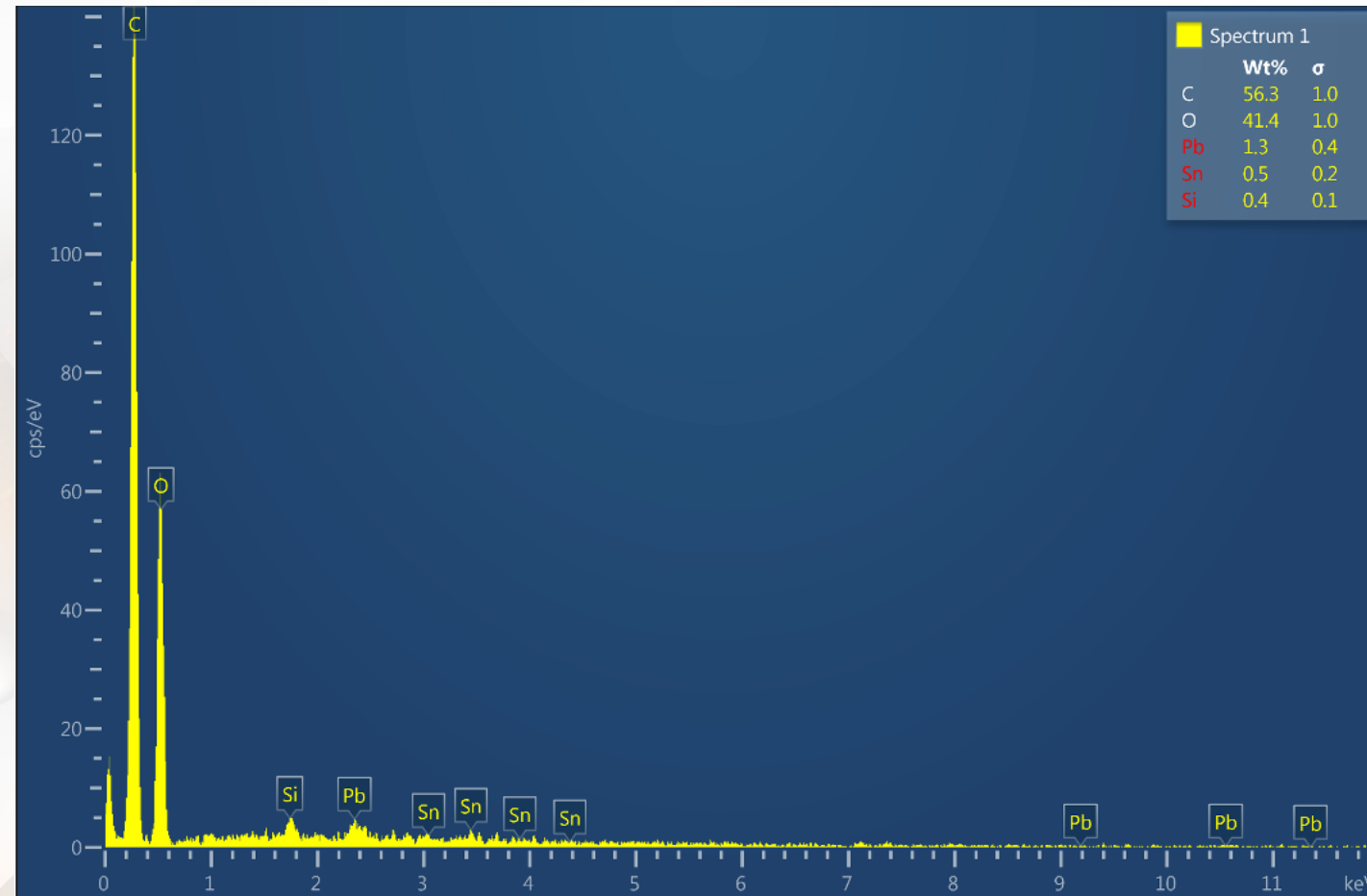
Spectrum 1

250µm

Si K series

x-ray detector shadowed

250µm



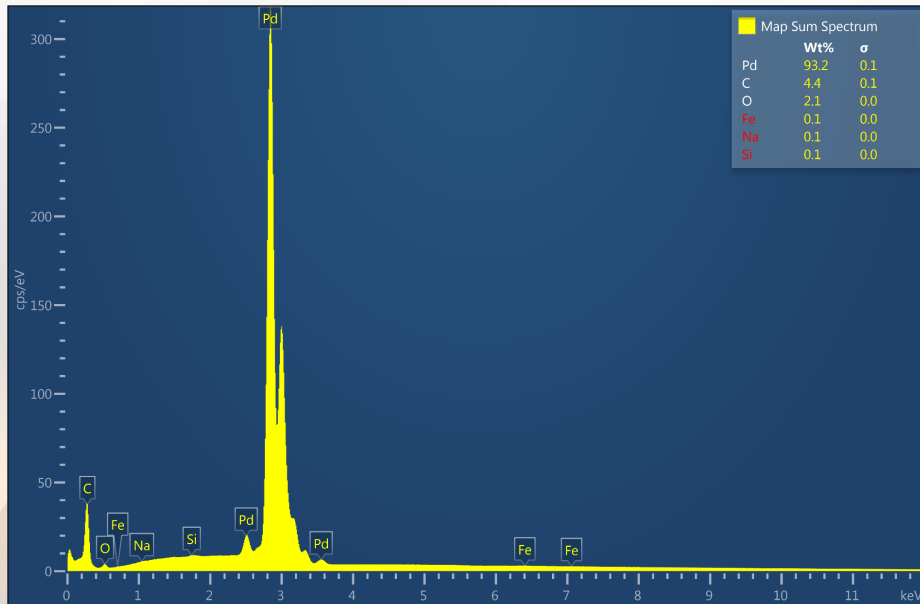
C 56.3%
O 41.4%
Pb 1.3%
Sn .5%
Si .4%

Just an inclusion on the surface

Where you look makes a difference!

Contamination, Transportation or Transmutation?

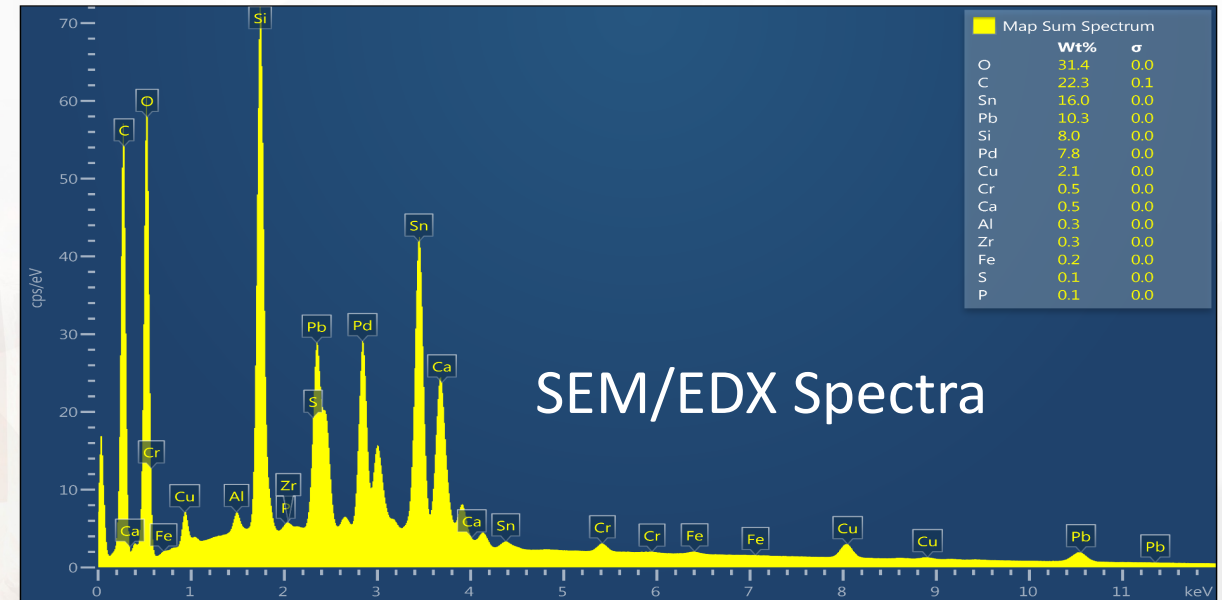
Virgin Tanaka Precious Metals Pd Plate



Predominately Pd with trace Si, Fe, Na

Pd-Plates_LCF-041-Pd-NL-NL-20220323-041_2022-03-23_11-49-35

Electrolyzed Tanaka Pd plate in Li_2SO_4



SEM/EDX Spectra

O, C, Sn, Pb, Si, Pd, Cu, Cr, Ca, Al, Zr, Fe, S, P

Pd-Plates_LCF-042-Pd-WC-D-20220323-042-Site1_2022-03-23_11-47-56

The experiment had:

Borosilicate glass Pyrex[®] (4.0% B, 54.0% O, 2.8% Na, 1.1% Al, 37.7% Si, and 0.3% K).

Cu feed wires that were soldered with Sn/Pb solder. (ratio close to 60:40 ratio)

Li_2SO_4 Electrolyte

Maintained in an Al calorimeter

Pd plate background includes trace Fe, Na, Si

Li and B have low Z
Hard to see with EDX

*What about:
Ca, Cr, Zr?*

The SEM/EDX had an Al stub and a Carbon-Carbon tape holder (though neither should have been visible to the beam).

Electrolytic Experiment Two

Deuterated U and Th using D₂O and Li₂SO₄

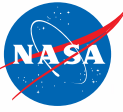


- U and Th loaded proton-conducting ceramic
- Fabricated by J. Patterson, CETI, Inc. and L. Forsley, JWK¹, under CETI License
- Cell ran for 4 hours, < 0.2 amps in a CETI RIFEX™ Kit at NRL
- HPGe γ analyses conducted at NRL
- NAA analyses at University of Illinois

¹ Under purchased CETI Research License

Electrolytic Experiment Two¹

Results



ICP/MS

> Ba(+104x), Ni(+18.5x), Pb (+15x), Zn(+10x), Ca, Mg, Al, trace amounts of Cr, Fe, Ni and Pt

< U and Th.

no isotope shifts from natural abundance within experimental error for Mg, Ni, Cu, Zn, Ba, or U.

Only 1 -2 percent isotopic changes between the used and unused samples, *within error bars*.

Ti was notably absent, despite the Ti electrodes presenting the largest surface area for contact. This may be indicative of an oxide forming on the Ti, thereby sealing it from contact with the electrolyte, as well as reducing its electrical conductivity.

HPGe

Real-time during operation showed no change in ²³⁵U, or ²³⁸U or ²³²Th decay chains

Prolonged not operating, closed, showed expected growth of ²¹²Pb and ²⁰⁸Tl from decay chains

NAA

Results ambiguous due to fission of U and Th.

¹ L. Forsley, et al., "Analyzing Nuclear Ash From the Electrocatalytic Reduction of Radioactivity in Uranium and Thorium", ICCF-7, Eneco, Inc., Vancouver, Canada (1997) 128-132.

² Under purchased CETI Research License

Electrolytic Experiment Two¹

Diagnostic Issues



ICP/MS Instrumentation Issues

Inability to distinguish between ^{64}Ni and ^{64}Zn : was ^{64}Ni enhanced and ^{64}Zn diminished over natural abundance?

^{67}Zn overlaps with $^{134}\text{Ba}^{++}$ and can not be reported.

Contamination and Transportation

O-rings (Viton) are a possible source of barium.

Cr, Fe, Ni, and Pt present in electrical feed wires and Ti electrodes.

Al and Si were present in the alumina pump head used prior to the adoption of a contact-less peristaltic pump.

¹ L. Forsley, et al., "Analyzing Nuclear Ash From the Electrocatalytic Reduction of Radioactivity in Uranium and Thorium", ICCF-7, Eneco, Inc., Vancouver, Canada (1997) 128-132.

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Conclusion



- Know your experiment
- Know your diagnostics and instrumentation
- Have statistically significant sized samples
- Be aware of:
 - Confounding interferences and backgrounds
 - Loss of sensitivity through sample preparation and dilution
 - Unrealized investigational bias
- *Be suspicious!*

Be wary of the tale of looking for your keys around the lamppost because the light is better there!

Acknowledgements



“Back to the Future”

Experiments conducted in the 1990s to the last analyses in 2022!

Pd/D Electrolysis: [University of Rochester \(UofR\) 1990-1993](#)

L. Forsley and J. Jorne, Department of Chemical Engineering, UofR

Tanaka Precious Metals Pd provided by E. Mallove

Accelerator Mass Spectroscopy Pd Isotope characterization offered by Nuclear Structures Research Laboratory (NSRL), UofR, *but denied by J. Huizenga¹*, Department of Chemistry, UofR

Conducted under a grant from ENECO, Inc. courtesy of F. Jaeger

SEM/EDAX conducted at NASA GRC under the NASA Lattice Confinement Fusion Project

Proton-Conducting Ceramic Encapsulated Th/U RIFEX™ Electrolysis²: [Naval Research Laboratory \(NRL\) 1996](#)

L. Forsley, R. August and G. Phillips, supported by D. Nagel, at NRL

Ceramic structure fabricated by J. Patterson, CETI, Inc. and L. Forsley, JWK, under CETI License

Neutron Activation Analysis (NAA) conducted by S. Landsburger, University of Illinois

SEM/EDAX and ICP/MS at NRL

Funded by JWK International Corporation

¹ John Huizenga told Harry Gove, NSRL Director, that the proposed analyses were, *“Inappropriate science.”*

² L. Forsley, *et al.*, “Analyzing Nuclear Ash From the Electrocatalytic Reduction of Radioactivity in Uranium and Thorium”, *ICCF-7*, Eneco, Inc., Vancouver, Canada (1997) 128-132.



Backup

Real-time Techniques



1. Calorimetry
 - **Various means:** *Room variations, accurately measuring input power, stable calibration,*
2. Photons
 - **RF:** various spectrum analyzers and software-radio: *Electromagnetic Interference*
 - **Infrared:** FLIR camera, un-backed Si (NIR): *Background interference*
 - **Visible:** digital cameras and spectrometers: *Background interference*
 - **Polarization:** presence of magnetic fields, Stokes parameters: *Interference with probe source*
 - **Spectroscopy:** black body vs non-black body radiation, e.g. down shifted bremsstrahlung
 - **X-rays and γ :** HPGe, LaBr₃, NaI, GM Tube: *Background interference*
3. Neutrons
 - **Counting, spectroscopy:** *low count rate background interference*
4. Charged Particles
 - **Counting, spectroscopy:** *Easiest in vacuum, difficult in aqueous electrolyte, EMI and light interference*