Low Background Studies of Pb – Free Solder Using ICP-MS

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Introduction

Inductively Coupled Plasma Mass Spectrometry (ICP-MS), was used to look for radioactive isotopes Uranium-238 and Thorium-232 in two lead-free Solder pastes used for electronics built for ultra-low background in rare-event searches.

Uranium and Thorium were examined for their decay-chain that causes a bulk of γ-ray, alpha-particle, and neutron-induced backgrounds.

The ICP-MS, a sensitive multielement detection instrument, was used to determine these trace elements within the Solder.

ICP-MS

ICP-MS:

- a sensitive multielement detection instrument.
- useful in determining ultratrace amounts of specific elements in the sample.

- works by up-taking a sample into a spray chamber via a nebulizer
- This allows small droplets to pass into the plasma while large droplets flow into the waste.

Nebulizer creates an aerosol spray

Spray Chamber removes larger aerosol droplets

- Once in the plasma the droplets are vaporized and turned into atoms and then dry ions.
- Ions then enter the quadrupole mass spectrometer.

ICP-MS Data

Results for the digestion of solder pastes

	Conc. (ppm)			
Solder ID		Pb	Th	U
NC-SMQ80	Avg	200	-0.00031	0.00027
Indium5.7LT	Avg	26.7	0.0073	0.0011

Unique Sample Preparation

Low background analysis includes several unique samples that do not have well established digestion procedures.

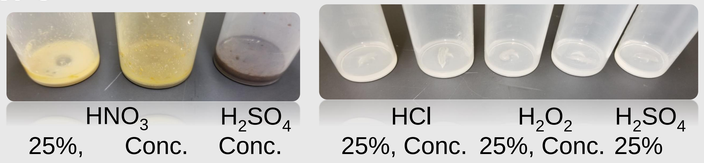
Before analysis the sample must be tested to determine a suitable digestion protocol. A variety of digestions were analyzed using different temperatures, acid concentrations, and acid combinations.

Trial 1

Trial 1: Vary:

Temp ^oC	Digestant	Concentration of Digestant
30	HNO_3	Concentrated
50	HCl	25%
100	H_2O_2	
120	H_2SO_4	

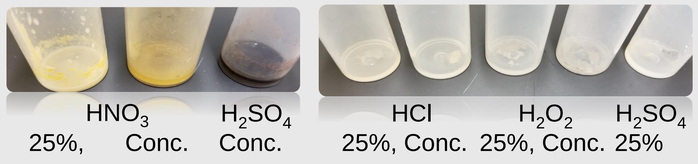
30^{o}C



HNO_3 25%, Conc. | H_2SO_4 Conc. | HCl 25%, Conc. | H_2O_2 25%, Conc. | H_2SO_4 25%

-Nitric turns white, conc. Sulfuric is too strong

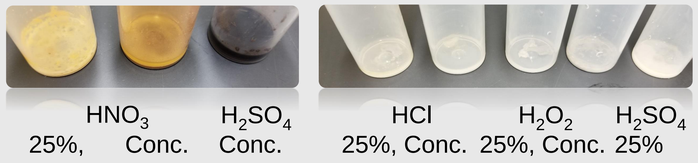
100^{o}C



HNO_3 25%, Conc. | H_2SO_4 Conc. | HCl 25%, Conc. | H_2O_2 25%, Conc. | H_2SO_4 25%

-Same as 30, but the others are starting to digest.

120^{o}C



HNO_3 25%, Conc. | H_2SO_4 Conc. | HCl 25%, Conc. | H_2O_2 25%, Conc. | H_2SO_4 25%

-Temp is a little high, dried out the HNO_3 samples.

Trial 2

Trial 2: Vary: Digestant Mixtures

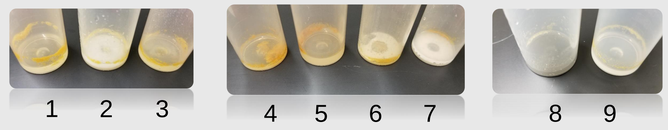
	Digestant 1	Digestant 2
1	50% HNO_3	50% HCl
2	50% HNO_3	Conc. H_2O_2
3	50% HNO_3	Conc. H_2SO_4
4	1 mL of 1:2 HNO_3:HCl mixture	
5	2 mL of 1:2 HNO_3:HCl mixture	
6	1 mL of 1:2 HNO_3:H_2O_2 mixture	
7	2 mL of 1:2 HNO_3:H_2O_2 mixture	
8	Conc. H_2O_2	Conc. H_2SO_4
9	Conc. H_2O_2	Conc. HCl

In each run:

- 1 mL of the first digestant was added.
- the solution is heated at 100^{o}C for 5 minutes.
- 1 mL of the second digestant was added.

Except where noted otherwise ▪

all Digestions occurred at 100^{o}C



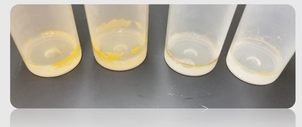
1 2 3 | 4 5 6 7 | 8 9

-mixed digestants do not work well for this.
- 1, 8 , and 9 are clear and mostly digested.

Trial 3

Trial 3: 3 digestants

Digestant 1	Digestant 2	Digestant 3
50% HNO_3	50% HCl	Conc. H_2O_2
50% HNO_3	50% HCl	50% H_2SO_4
Conc. H_2O_2	50% HCl	50% H_2SO_4
Conc. H_2O_2	50% HCl	50% HNO_3



-all samples digested, but solution 3 didn't have undissolved solids.

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