



September 14, 2012

Joe Cerniglia
The International Group for Historic Aircraft Recovery (TIGHAR)

Subject: ICP-MS Report
Job Number: S0CGF653

Dear Joe:

Please find enclosed the procedure report for the analysis of your samples.

Thank you for using the analytical services of the Evans Analytical Group - NY. We appreciate your business and welcome any suggestions you may have for improving the quality and efficiency of our service. Please do not hesitate to call us if you have any questions regarding this report.

Sincerely,

A handwritten signature in black ink, appearing to read "Paul K. Anseele".

Manager ICP, IGA and TG Services
(Tel. 315-431-9900; risensee@eaglabs.com)

Enclosures:



**ICP-MS REPORT
JOB NUMBER: S0CGF653**

for

Joe Cerniglia
The International Group for Historic Aircraft Recovery (TIGHAR)

Analyzed by:

A handwritten signature in black ink, appearing to read "G. Infantino", written over a horizontal line.

Gabriel Infantino
Lead Analyst
(Tel. 315-431-9900; ginfantino@eaglabs.com)

Reviewed by:

A handwritten signature in black ink, appearing to read "Robert K. Isensee", written over a horizontal line.

Robert K Isensee
Manager ICP, IGA and TG Services
(Tel. 315-431-9900; risensee@eaglabs.com)

Evans Analytical Group
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Syracuse, NY 13211, USA

Purpose:

To determine compositional and leachable elements present in one glass sample.

ICP-MS Method:

Aqueous samples are aspirated and converted to an aerosol through a nebulizer and directed into an argon based plasma. Here the sample is dried, vaporized, atomized and ionized in the argon plasma. The resulting ions are then directed into a quadrupole mass analyzer where they are then separated and measured according to their mass to charge ratios. The constituents of an unknown sample can then be identified and quantified. ICP-MS offers extremely high sensitivity to a wide range of elements.

Analytical Set-up: ICPMS

Instrument: Perkin Elmer Elan DRC II equipped with a Cetac ASX-520 auto sampler.

ICP-MS conditions

Instrument	ELAN DRC II
Nebulizer	Quartz Meinhardt
Spray Chamber	Cyclonic
RF power	1400 W
Ar Flow	15.0 L/min
Auxillary Ar Flow	1.2 L/min
Nebulizer Gas Flow	0.88 L/min
Integration time	80 S
Scanning mode	Peak hopping
Replicates	1
RPq for Cerium as CeO (m/z 156)	< 2 %

Procedure:

For this material two different tests were performed. The first test was an acid leach comparison of the the exterior and interior cavity of the glass jar. The goal was to determine if any elemental residue was left behind from the jars contents. The second test involved total digestion of the glass to determine the elements present in the bulk material. Both tests involved the use trace metals grade acids and high purity water. Reagent information, manufacturer and lot numbers are listed below:

Nitric Acid - ARISTAR PLUS - trace metal grade - Lot number 1111010
Hydrochloric Acid - ARISTAR PLUS - trace metal grade - Lot number 4111040
Hydrofluoric Acid - Fisher - trace metal grade - Lot number 5111093
Aqua Regia - mix of hydrochloric and nitric trace metal acids in a 3:1 ratio
High Purity Water - 18.2MΩ*cm, deionized water

In the first test an acid leach was performed separately on the exterior and the interior of the glass jar. In both cases the leaching involved 1ml of 1:1 aqua regia being allowed to sit on the desired surface for 4 hours in a trace metals hood. Upon completion of the leach time the acid was rinsed into an acid cleaned 50ml polypropylene sample tube using 18.2M Ω *cm deionized water and brought to a final volume of 50ml. These samples were then analyzed for a full sweep of 69 elements using ICPMS. An aqua regia acid blank was also run to correct for background and possible interferences.

For the second test a total digestion of the bulk glass was done to determine compositional and trace elemental components. For this digestion an Anton Paar multiwave microwave digestion unit was used. This allows for digestion in a closed environment under high temperature and pressure with minimum loss of volatile elements. To perform the digestion two separate pieces were chipped off from the outer side of the sample. The pieces were chipped off using a file covered in acid cleaned parafilm. The sample itself was also covered in acid cleaned parafilm except for the areas to be chipped from. A small amount of force was used against the file to chip the pieces away. Once free the pieces were rinsed in high purity water, dried and weighed out for microwave digestion. This digestion involved a 1:1 combination of nitric and hydrochloric acids along with 0.5ml of hydrofluoric acid. An acid blank was also taken through the digestion process to correct for background and possible interferences. The sample was digested from two locations to ensure any elemental hit was part of the bulk and not some surface artifact. Each sample digestion used approximately 100mg of sample.

All elements were calibrated using NIST traceable multi-element standards keeping a minimum calibration coefficient criteria of 0.999. Quality control standards were run at the start, finish and periodically throughout the runs with a minimum acceptance criteria of $\pm 10\%$.

Results:

Table 1 shows a comparison of the leach performed on the inner and outer surfaces. Table 2 shows the compositional and trace components found in digestion of the glass bulk.

Table 1. Results of leach from inner and outer glass surfaces.

Element	Inner Surface Mass Fraction (ug/L)	Outer Surface Mass Fraction (ug/L)	Element	Inner Surface Mass Fraction (ug/L)	Outer Surface Mass Fraction (ug/L)
Li	1	< 1	In	< 1	< 1
Be	< 1	< 1	Sn	7	3
B	27	2	Sb	< 1	< 1
Na	6100	3900	Te	< 1	< 1
Mg	10 mg/L	2700	Cs	< 1	< 1
Al	4300	960	Ba	26	6
Si	2300	36	La	< 1	10
P	160	10	Ce	< 1	< 1
K	6800	110	Pr	< 1	< 1
Ca	10 mg/L	120	Nd	< 1	2
Sc	< 1	< 1	Sm	< 1	< 1
Ti	7	3	Eu	< 1	< 1
V	26	14	Gd	< 1	< 1
Cr	7	4	Tb	< 1	< 1
Mn	2	< 1	Dy	< 1	< 1
Fe	83	73	Ho	< 1	< 1
Co	< 1	4	Er	< 1	< 1
Ni	12	15	Tm	< 1	< 1
Cu	18	24	Yb	< 1	< 1
Zn	88	28	Lu	< 1	< 1
Ga	< 1	2	Hf	< 1	< 1
Ge	< 1	< 1	Ta	< 1	< 1
As	12	6	W	2	5
Se	< 1	< 1	Re	< 1	< 1
Rb	2	< 1	Os	< 1	< 1
Sr	19	1.5	Ir	< 1	< 1
Y	1	< 1	Pt	< 1	< 1
Zr	< 1	3	Au	< 1	< 1
Nb	< 1	< 1	Hg	4	< 1
Mo	< 1	13	Tl	< 1	< 1
Ru	< 1	< 1	Pb	5	< 1
Rh	< 1	< 1	Bi	1	< 1
Pd	< 1	< 1	Th	< 1	< 1
Ag	< 1	< 1	U	< 1	< 1
Cd	< 1	< 1			

Table 2. Results of digestion of glass bulk.

Element	Mass Fraction (ppm wt)	Element	Mass Fraction (ppm wt)
Li	3	In	< 1
Be	< 1	Sn	7
B	< 1	Sb	< 1
Na ₂ O	13.1 wt%	Te	< 1
MgO	4.3 wt%	Cs	< 1
Al ₂ O ₃	0.74 wt%	BaO	0.74 wt%
SiO ₂	Matrix	La	< 1
P	35	Ce	3
K ₂ O	0.24 wt%	Pr	< 1
CaO	8.5 wt%	Nd	< 1
Sc	< 1	Sm	< 1
Ti	100	Eu	< 1
V	6	Gd	< 1
Cr	< 1	Tb	< 1
Mn	11	Dy	< 1
Fe	340	Ho	< 1
Co	< 1	Er	< 1
Ni	< 1	Tm	< 1
Cu	< 1	Yb	< 1
Zn	16	Lu	< 1
Ga	< 1	Hf	< 1
Ge	< 1	Ta	39
As	< 1	W	38
Se	< 1	Re	< 1
Rb	13	Os	< 1
Sr	94	Ir	< 1
Y	< 1	Pt	< 1
Zr	89	Au	< 1
Nb	< 1	Hg	89
Mo	< 1	Tl	< 1
Ru	< 1	Pb	7
Rh	< 1	Bi	< 1
Pd	< 1	Th	< 1
Ag	< 1	U	< 1
Cd	< 1		