



January 3, 2013

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

Subject: ICP-MS Report
Job Number: S0CHG693

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. Ansee".

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

Enclosures:



**ICP-MS REPORT
JOB NUMBER: S0CHG693**

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](mailto: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
6707 Brooklawn Parkway
Syracuse, NY 13211, USA

Purpose:

To determine the amount of mercury present in a freckle cream and the amount of mercury leachable from the containing jar.

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	1350 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 and 2
RPq for Cerium as CeO (m/z 156)	< 2 %

Procedure:

For this procedure two different tests were performed to determine the presence of mercury. The first test was an acid leach of the the exterior and interior cavity of a glass jar containing Dr. Berry's Freckle Ointment. The second test involved total digestion of Dr. Berry's Freckle Ointment found inside the jar. 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 1112074
Hydrochloric Acid - ARISTAR PLUS - trace metal grade - Lot number 4112030
Aqua Regia - mix of hydrochloric and nitric trace metal acids in a 3:1 ratio
High Purity Water - 18.2MΩ*cm, deionized water

For the first test an acid leach was preformed separately on the exterior and the interior surfaces of the glass jar. In both cases the leaching involved 1ml of aqua regia being allowed to sit on the desired surface for 4 hours in a trace metals hood at room temperature. Prior to leaching, as much freckle ointment was removed from the jars interior as physically possible without chemical aid. In addition, prior to leaching each surface was rinsed three time with 18.2MΩ*cm deionized water. The exterior leach area was covered with Para film during leaching to prevent evaporation or contamination during the process. During the interior leach the jars opening was covered with Para film also for similar reasons. The jars metal lid was not used to seal the jar during interior leaching to prevent possible acid attack and subsequent contamination of the leach. In both cases upon completion of the leach the acid was rinsed into separate acid cleaned 50ml polypropylene sample tube using 18.2MΩ*cm deionized water and brought to a final volume of 50ml. These samples were then further diluted as necessary and analyzed for mercury 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 Dr Berry's Freckle Ointment using an Anton Paar multiwave microwave digestion unit was preformed. This allows for digestion in a closed environment under high temperature and pressure with minimum loss of volatile elements. To perform the digestion two 100mg samplings of the freckle ointment were weighed out and placed in Teflon digestion vessels. Digestion was accomplished using 5ml of trace metals grade nitric acid and 5ml of 18.2MΩ*cm deionized water. An acid blank was also taken through the digestion process to correct for background and possible interferences. Once complete all digestions, including the blank, were brought to a final volume of 50ml in acid cleaned polypropylene sample tubes using 18.2MΩ*cm deionized water. These samples were then further diluted as necessary and analyzed for mercury using ICPMS.

All elements were calibrated using NIST traceable multielement standards keeping a minimum calibration coefficient criterion of 0.999. Quality control standards were run at the start, finish and after each dilution set with a minimum acceptance criterion of ± 10%.

Results:

Table 1 shows the level of mercury found in the ointment and in the leach of both surfaces.

Table 1. Mercury content found in ointment and jar leaches.

Sample	Total Hg content	% RSD
Ointment	5.4 wt%	6.5
Inside Leach	120 mg/L	NA
Outside Leach	< 0.005 mg/L	NA