Gunshot Residue Analysis and Other Applications of ICP-MS

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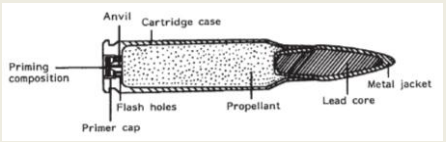
Overview

- Gunshot Residue Analysis (GRA) - overview
- GSR using ICPMS
- ICP-MS instrument operation and calibration
- ICP-MS analysis of archaeological bone
- Comments on operation of ICPMS facility
- Other applications of ICP-MS at ITSligo

Gunshot Residue Analysis



- Evidence of proximity to discharge of weapon
- Matching and comparing weapons or bullets.

Alternative techniques for Gunshot Residue Analysis

- Presumptive Colour Tests
- Scanning Electron Microscopy with Energy-Dispersive X-Ray Fluorescence (SEM-EDX)
- Flame Atomic Absorbance Spectroscopy (FAAS)
- Graphite Furnace Atomic Absorbance Spectroscopy (GFAAS)
- Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES)

Gunshot Residue Analysis by ICPMS

- Quantitative multi-element analysis
- Relatively simple sample preparation
- Relatively interference free for most relevant elements
- Low detection limits
- Possible isotopic ratios

Forensic Investigation and Analysis student at shooting range near Riverstown, Co Sligo

Gunshot residue sampling:The GSR sampling kit

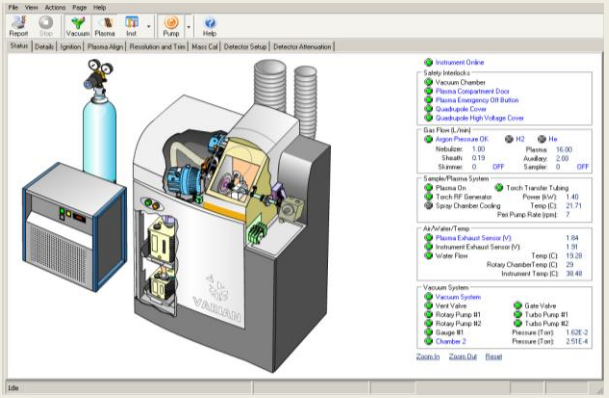
- Tape lifts from fingers, hands and face .
- Comb to remove residue from hair
- Cotton buds to remove residue from between fingers
- Evidence bags and chain of evidence

Gunshot residue sampling plan and sample preparation for ICPMS

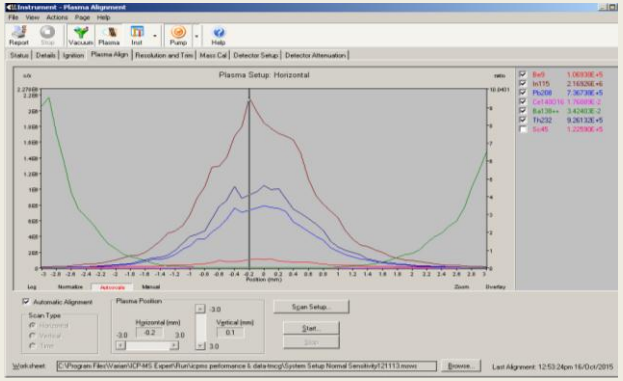
- Control sample collected at shooting range prior to using weapons
- Random samples taken from population for purpose Bayesian statistical analysis
- Dissolution and digestion in 10% ultra pure nitric acid
- Working sample and standards prepared in 2% nitric acid
- Multi-element standards prepared from single element standards certified for ICPMS analysis
- Internal standard added to all samples, standards and blanks

ICP-MS Laboratory

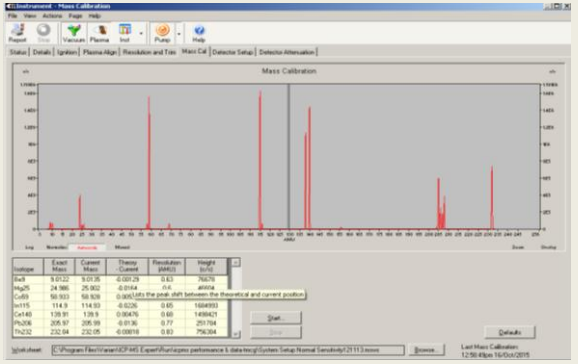
ICP-MS Operating conditions



ICPMS signal optimisation

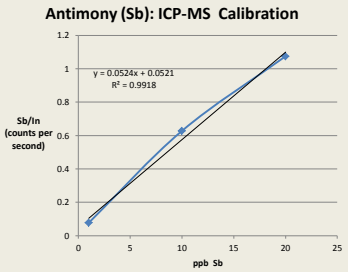


Mass calibration and resolution



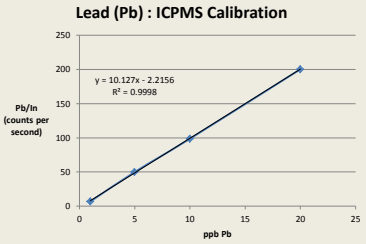
Gunshot residue – Antimony (Sb) (student ICP-MS results)

	Average Concentration (blank subtracted) (µg/L)	Standard Deviation (µg/L)
Random Individuals (n=10)	0.02	0.046
GSR samples (n=4)	24	59
Control	0.3	



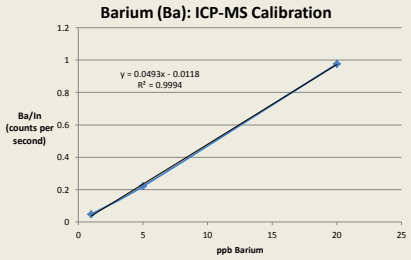
Gunshot residue – Lead (Pb) (student ICP-MS results)

	Average Concentration (blank subtracted) (µg/L)	Standard Deviation (µg/L)
Random Individuals (n=10)	1.0	0.3
GSR samples (n=4)	3.9	2.6
Control	2.8	



Gunshot residue – Barium(Ba) (student ICP-MS results)

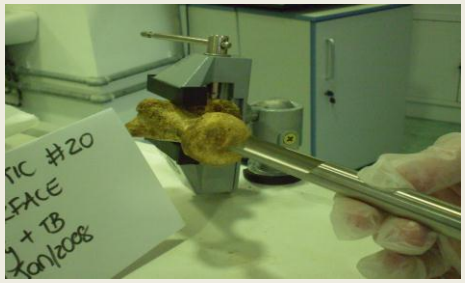
	Average Concentration (blank subtracted) (µg/L)	Standard Deviation (µg/L)
Random Individuals (n=10)	141	35
GSR samples (n=4)	2.3	1.1
Control	1.1	



What archaeological research questions can multi-element analysis results address ?

- **Diagenesis**
- **Biogenic indicators**
- **Paleodietary Reconstruction**
- **Health status information**

Bone sampling for ICPMS: Core sampling of cortical bone from the femur head using a manual coring drill

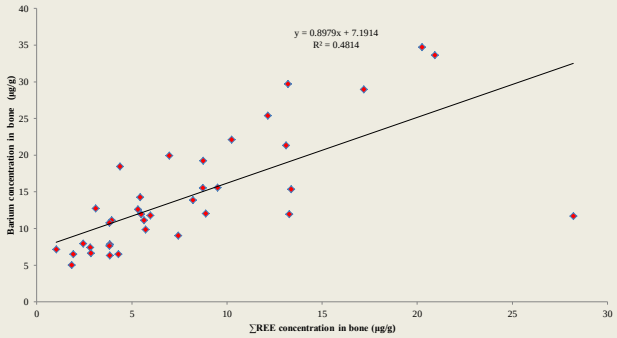


0.5 g bone sample microwave digested with ultrapure nitric acid

ICPMS % Recoveries for individual rare earth elements in a bone matrix (SRM 1486 bone meal)

	Spike (ppb)	% Recovery		Spike (ppb)	% Recovery
Ce	1	76.58	Pr	1	98.54
Dy	1	100.11	Sm	1	100.36
Er	1	99.58	Tb	1	72.22
Eu	1	98.36	Th	1	100.16
Gd	1	98.81	Tl	1	99.19
Ho	1	99.46	Tm	1	99.38
La	1	78.32	U	1	97.88
Lu	1	104.22	Yb	1	99.4
Nd	1	98.32			

Total rare earth element (∑REE) concentration versus barium concentration in the bones of 38 articulate skeletons



Comments on practical use of ICPMS

- Good in house technical support
- Skilled operator
- Use calibration standards specified for multi-element (ICPMS & ICPAES)
- Must use internal standardisation as the norm
- Plan your analysis well to avoid waste of argon and other resource
- Keep spares (e.g. nebulisers, actuators, vacuum and flow sensors)

Other ICP-MS projects at IT Sligo

- Analysis of fish otoliths and tracking fish migration
- Determination of gold and silver nano-particles
- Determination of trace elements in battery acid
- Environmental analysis e.g. bio-remediation of toxic effluents with algae
- Trace elements in seaweed extracts