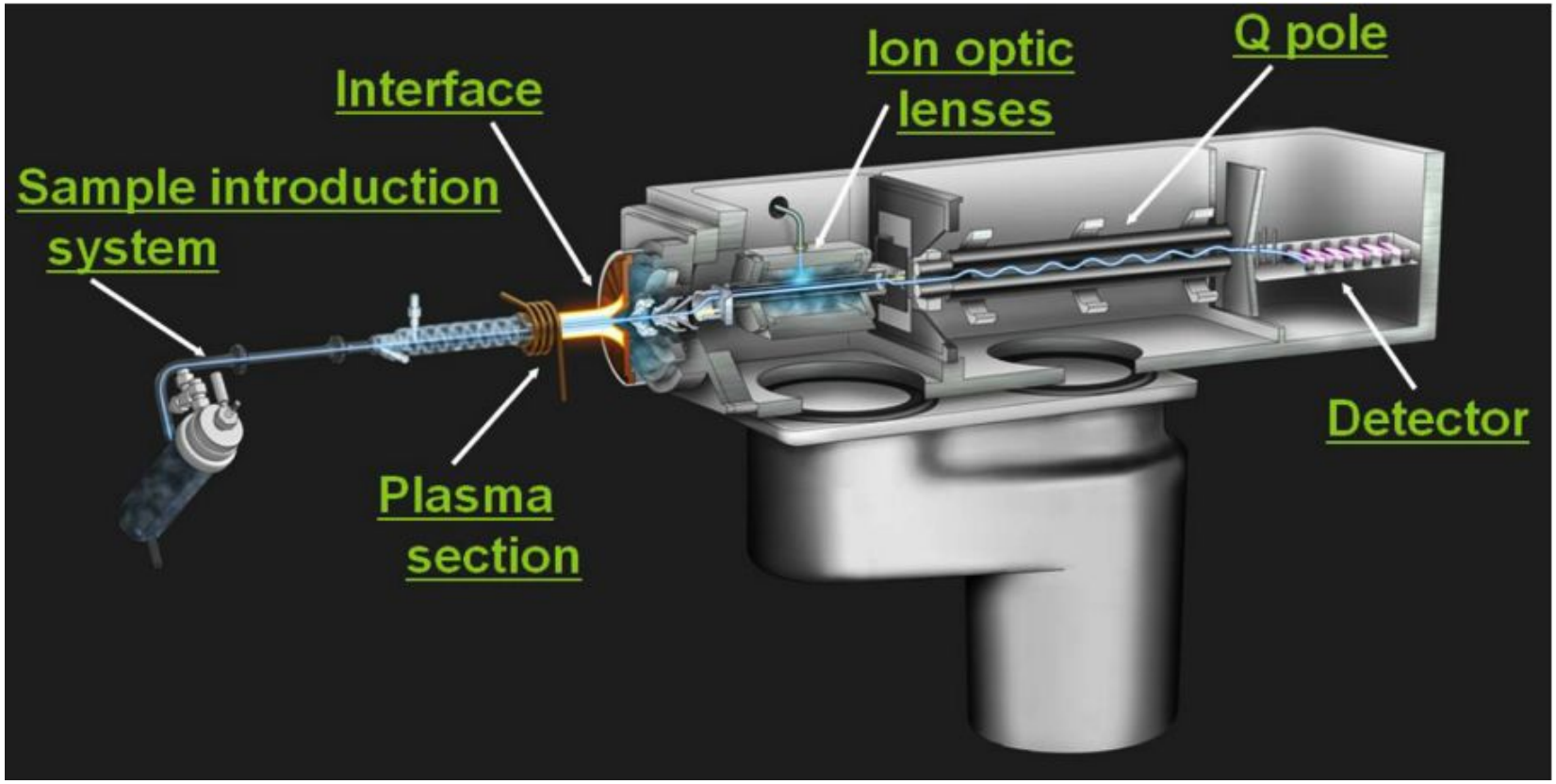
A brief intro to ICP-MS

UCL

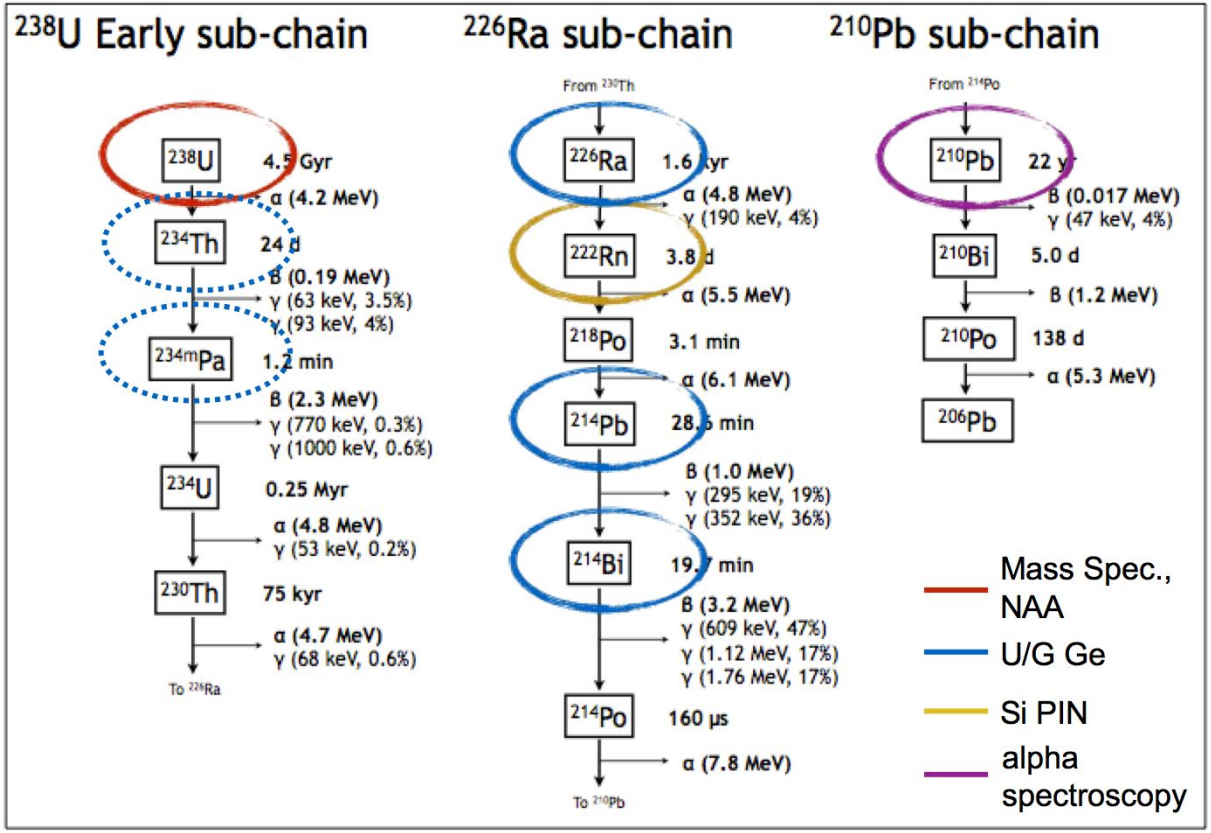
ICP-MS = Inductively Coupled Plasma Mass Spectrometry



Can analyse almost all stable elements on earth (Li - U): ionises elements, selects them based on ion mass to charge ratio, and counts. Fast analysis at ppm - ppt in seconds.

ICP-MS for low-BG experiments

- Measure U-238 and Th-232 → early chain info
- ~10 ppt sensitivity achievable
- High-throughput: sample digestion + analysis 1-2 days
- Complementary to γ-screening → build full picture of background model
- Risk mitigation for fast accept/reject of materials



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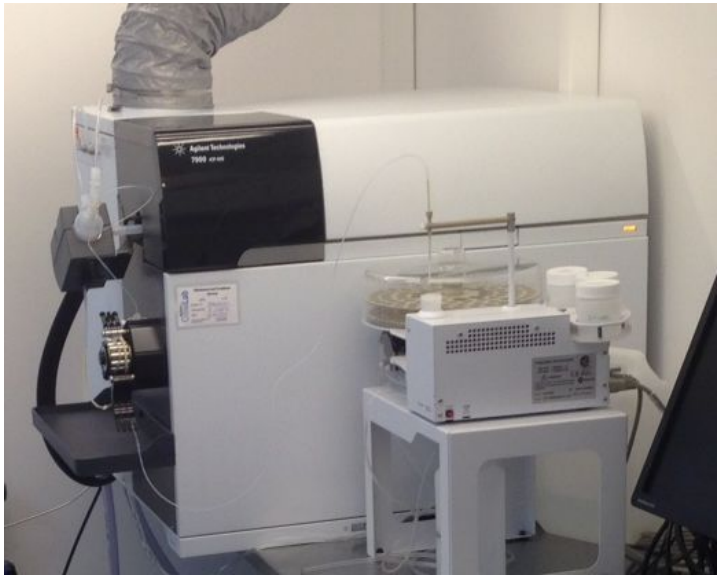
ICP-MS facility at UCL

- Agilent 7900 ICP-MS + HF capability
- ISO Class 6 (1000) cleanroom (ISO class 5 (100) laminar flow unit
- Microwave digestion: use ultra-clean TFM vessels
- Ashing oven
- Ability to purify our own acid with sub-boiling point distillation system



Microwave digestion and ashing closed, clean systems for sample prep

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From the outside

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Inside cleanroom

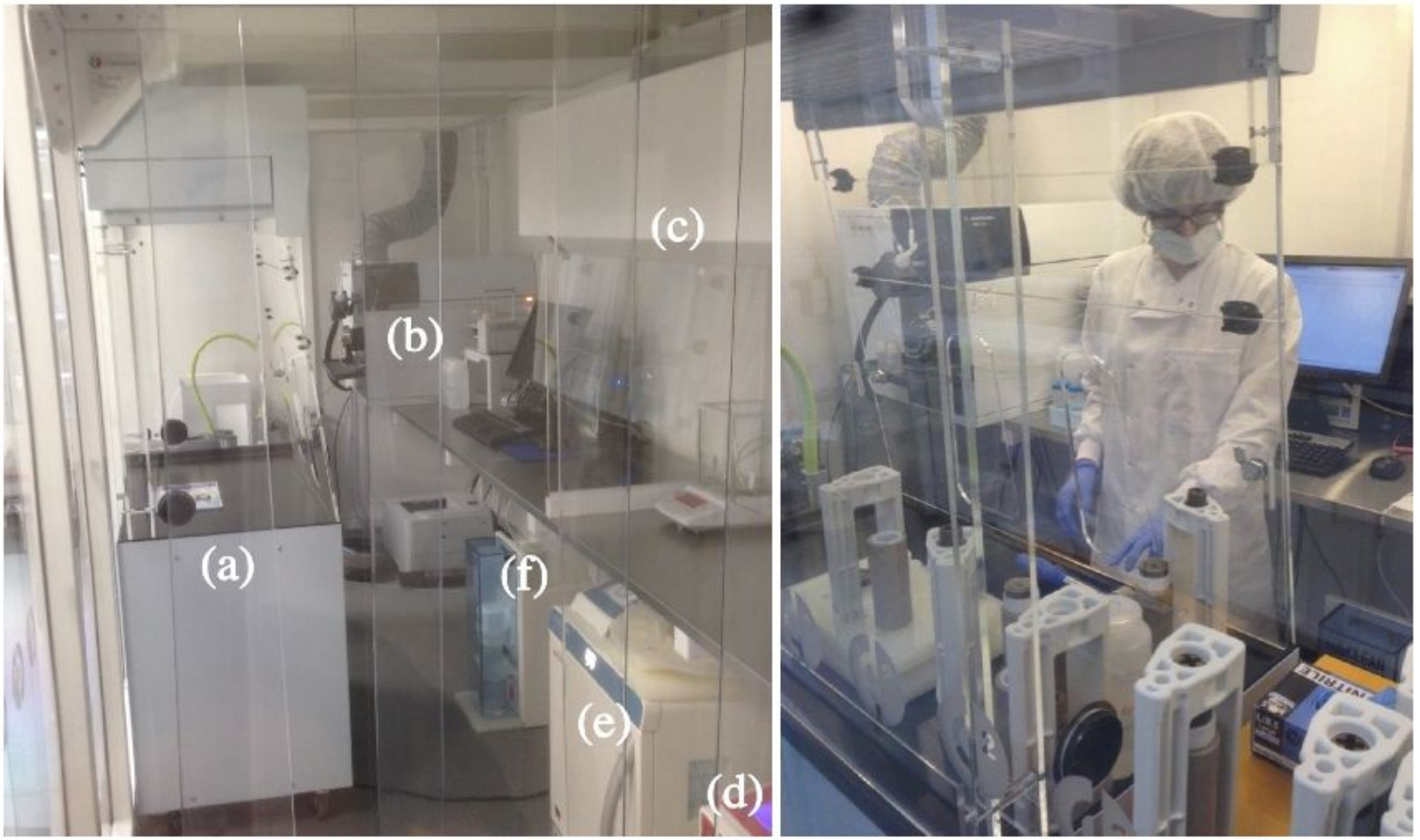

FIG. 1: *Left:* view of the ISO Class 6 cleanroom through changing room curtain showing the fume cupboard (a), Agilent 7900 ICP-MS (including integrated auto-sampler and control PC) (b), the class 5 laminar flow hood (c), the Milestone Pyro 260 microwave ashing (d) and ETHOS UP microwave digestion (e) systems, and the SubCLEAN subboiling distillation unit (f). *Right:* sample preparation in the fume cupboard, an unassembled TFM^{TM}PTFE sample preparation vessel can be seen in the workstation jig.

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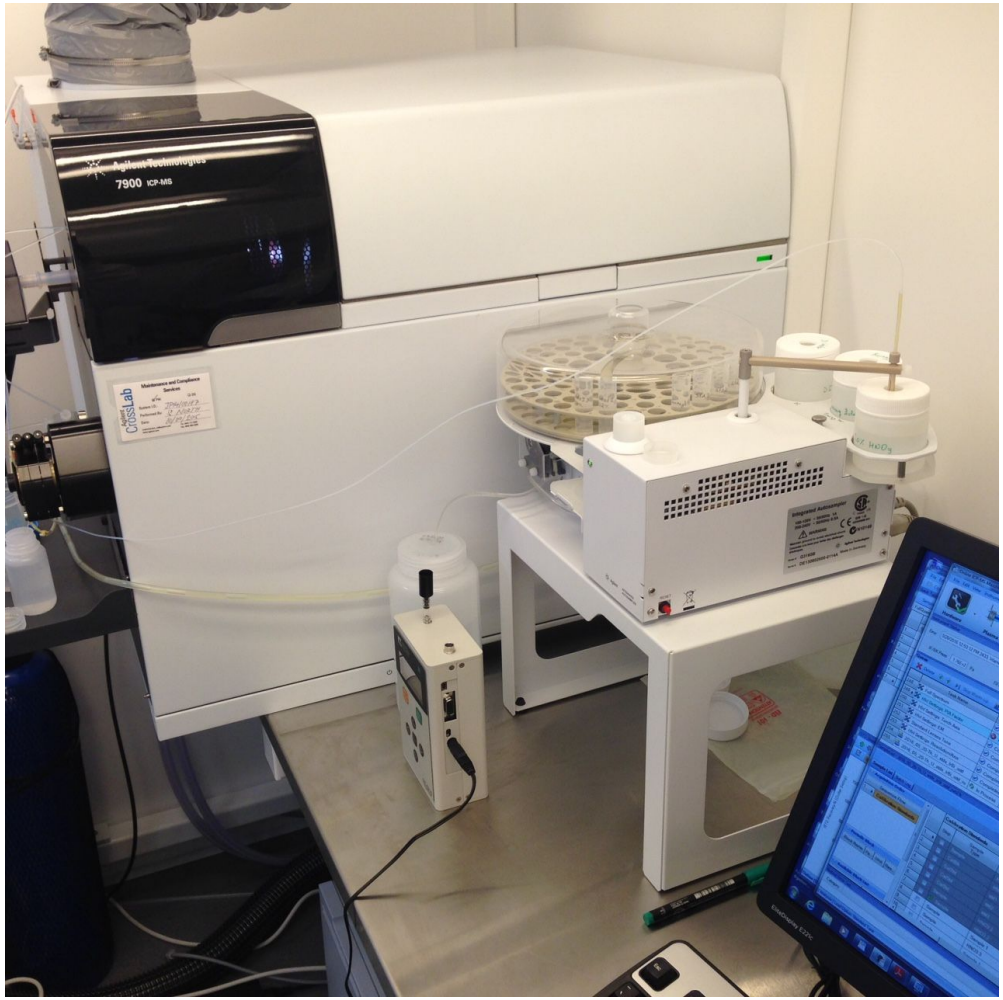
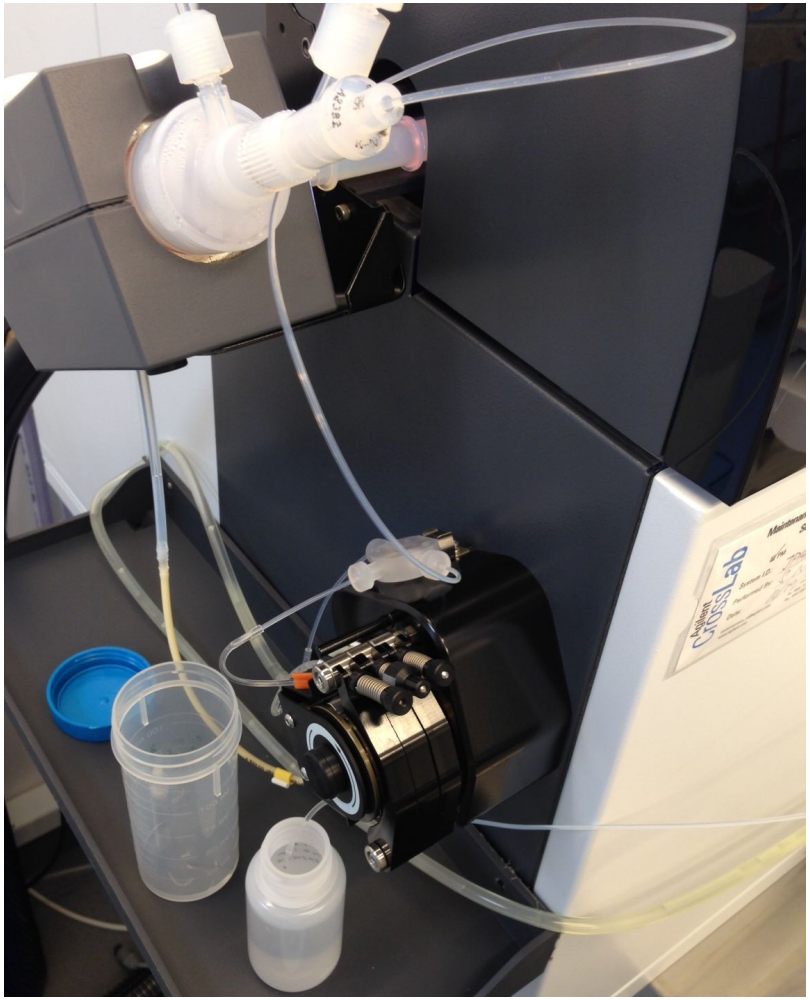
ICP-MS + auto-sampler





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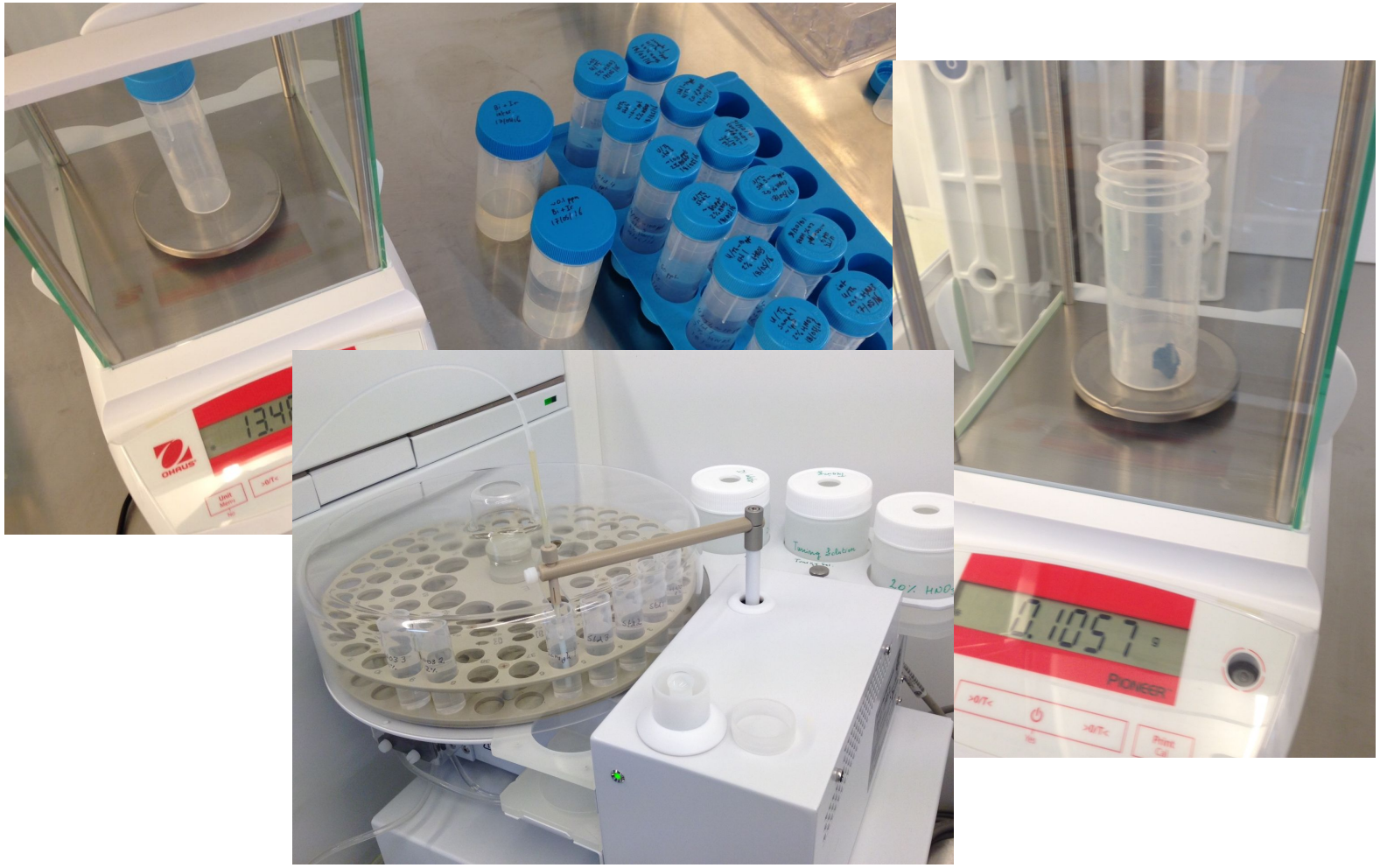
Cleaning protocols & sample prep. key



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Role of ICP-MS for Pb

(i) to measure isotopic composition of lead in water at UCL IC-PMS facility to correlate the Pb-210 to the total lead content in water, and quantify the typical variance across water samples (lead academic: Ghag);

Isotope			Decay	
	abun-dance	half-life ($t_{1/2}$)	mode	pro-duct
^{202}Pb	syn	5.25(28)×10^4 y	ε	^{202}Tl
^{204}Pb	1.4%	stable		
^{205}Pb	trace	1.53(7)×10^7 y	ε	^{205}Tl
^{206}Pb	24.1%	stable		
^{207}Pb	22.1%	stable		
^{208}Pb	52.4%	stable		
^{209}Pb	trace	3.253(14) h	β$^-$	^{209}Bi
^{210}Pb	trace	22.3(22) y	β$^-$	^{210}Bi
^{211}Pb	trace	36.1(2) min	β$^-$	^{211}Bi
^{212}Pb	trace	10.64(1) h	β$^-$	^{212}Bi
^{214}Pb	trace	26.8(9) min	β$^-$	^{214}Bi

Isotopic abundances vary greatly by sample

Standard atomic weight 207.2(1)[1]

($A_{r,\ standard}$)

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ICP-MS for stable → should be straightforward to get 10 ppb g/g

γ-screening of same sample to get ^{210}Pb

www.esslab.com/elements/pb.html

analytical periodic table

Lead [Analytical Periodic Table]

82 207.2
1750 1.6
327.6
Pb
[Xe]4f^{14}5d^{10}6s^{2}6p^2
11.4 2,4

Location: Group 14, Period 6
Atomic Weight: 207.2
Coordination Number: 6
Chemical Form in Solution: $Pb(H_2O)_6^{+2}$

Storage & Handling: Keep tightly sealed when not in use. Store and use at 20 ± 4°C. Do not pipet from container. Do not return portions removed for pipetting to container.

Chemical Compatibility: Soluble in HCl, HF, and HNO_3. Avoid H_2SO_4. Stable with most metals and inorganic anions forming insoluble carbonate, borate, sulfate, sulfite, sulfide, phosphate, oxalate, chromate, tannate, iodate, and cyanide in neutral aqueous media.

Stability: 2-100 ppb levels stable for months in 1% HNO_3 / LDPE container. 1-10,000 ppm solutions chemically stable for years in 2-5% HNO_3 / LDPE container.

Pb Containing Samples (Preparation & Solution): Metal (best dissolved in 1:1 H_2O / HNO_3); Oxides (the many different Pb oxides are soluble in HNO_3, with the exception of PbO_2 which is soluble in HCl or HF); Ores and Alloys (best attacked using 1:1 H_2O / HNO_3); Organic Matrices (dry ash and dissolve in dilute HCl. Do not heat when dissolving to avoid precipitation of SiO_2).

Atomic Spectroscopic Information:

Technique / Line	Estimated D.L.*	Order	Type	Interferences
ICP-OES 168.215 nm	0.03/.003 μg/mL	1	ion	Co
ICP-OES 220.353 nm	0.04/.006 μg/mL	1	ion	Bi, Nb
ICP-OES 217.000 nm	0.09/.03 μg/mL	1	atom	W, Ir, Hf, Sb, Th
ICP-MS 208 amu	5 ppt	n/a	M+	^{192}Pt^{16}O, ^{192}Os^{16}O

*ICP-OES D.L.'s are given as radial / axial view

See Inorganic Ventures' Lead (Pb) Standards In Our Store

For discussion: what mechanism would correlate ^{210}Pb to the stables isotopes?

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Using ^{210}Pb as a tracer

https://www.nature.com/articles/srep21707

nature > scientific reports > articles > article

SCIENTIFIC REPORTS

Article | OPEN | Published: 22 February 2016

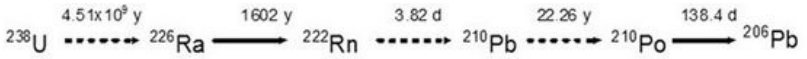
Use of lead-210 as a novel tracer for lead (Pb) sources in plants

Handong Yang & Peter G. Appleby

Scientific Reports **6**, Article number: 21707 (2016) | Download Citation

from Richmond Park, London, the UK. The mean ratio of ^{210}Pb to total Pb in atmospheric depositions collected from a site adjacent to the park during August–October 2012 was 96 Bq mg^{-1}, while the ratio in surface soils from the park was typically an order of magnitude lower. The difference between these values made it possible to trace the source of Pb in the plants. The ^{210}Pb/Pb ratios in plants varied from 0 to 34 Bq mg^{-1} indicating different levels of Pb absorption from the atmosphere. The ratio in mosses had an average value of 22 Bq mg^{-1}. This suggests that only around 20% of the Pb they contain was from direct atmospheric deposition, revealing possible limitations in the use of terrestrial mosses for monitoring atmospheric pollution. As well as

Figure 2: ^{238}U decay series, showing the principal radionuclides concerned with the production of ^{210}Pb and their radioactive half-lives.



The radionuclide ^{210}Pb (half-life 22.26 y) occurs naturally as a member of the ^{238}U decay series (Fig. 2). A fraction of the inert gas ^{222}Rn, a product of ^{226}Ra decay (half-life 1602 y) in soils, escapes to the atmosphere where it decays via a series of short-lived radionuclides to ^{210}Pb. ^{210}Pb atoms in the atmosphere are readily attached to airborne particles which are quickly removed to land surfaces and water bodies by wet and dry deposition. Fallout ^{210}Pb accumulating in soils and sediments is called unsupported ^{210}Pb, to distinguish it from the supported ^{210}Pb that derives from *in situ* decay of the parent radionuclide ^{226}Ra. The unsupported ^{210}Pb will decay to near-zero concentrations over a period of around six ^{210}Pb half-lives (~130 years). Supported ^{210}Pb, which will usually be in radioactive equilibrium with ^{226}Ra, is determined by measuring the ^{226}Ra activity of the sample. Unsupported ^{210}Pb is determined by subtracting the supported activity from the measured total ^{210}Pb activity.

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Calibration standard for total Pb content

Fairly inexpensive:

- Make up test solutions and use to confirm we can recover total Pb content
  - Will give indication of whether we have accounted for interferences and other biases
- Bracket each sample with one of these → correct for instrument drift throughout run

Lead, 1,000 ug/mL, for ICP-MS, 30 mL

List Price: $ 38.00 | In Stock: YES | Quantity: 1 | ADD TO CART

Single-Element Standards for ICP-MS

- Made with acid and ASTM Type I Water
- Inorganic compounds and metals at 99.99% to 99.9999% purity (where commercially available)
- Directly traceable to NIST (where applicable)

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Isotopic reference material

- Certified isotopic abundances → more expensive → can be used to correct for mass bias effects, i.e. a bias in the instrument response to different masses
- Could also use for isotope spike dilution measurement

Details	
Description:	Common Lead Isotopic Standard
Lot:	N/A
Expiration Date:	
Unit Price * :	$470.00
Unit of Issue:	1 g wire
Status:	Now Selling
Certificate Date:	3/25/1991
MSDS Date:	5/7/2014
Technical Contact:	Robert Vocke
Additional Information:	N/A

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 981

Common Lead Isotopic Standard

This Standard Reference Material (SRM) is intended primarily for use as an isotopic standard. SRM 981 consists of 1 gram of a commercially available, high purity lead metal, of 99.9+ percent purity, that was extruded into wire form. The atomic weight of the material is calculated to be 207.215 using the nuclidic masses 203.973044, 205.974468, 206.975903, and 207.976650. The certified isotopic compositions are given below.

Atomic Abundance Ratio, Lead-204/Lead-206 . . . 0.059042 ± 0.000037

Atomic Abundance Ratio, Lead-207/Lead-206 . . . 0.91464 ± 0.00033

Atomic Abundance Ratio, Lead-208/Lead-206 . . . 2.1681 ± 0.0008

Lead-204, atom percent . . . . . 1.4255 ± 0.0012

Lead-206, atom percent . . . . . 24.1442 ± 0.0057

Lead-207, atom percent . . . . . 22.0833 ± 0.0027

Lead-208, atom percent . . . . . 52.3470 ± 0.0086

Overall limits of error are based on 95 percent confidence limits for the mean of the ratio measurements and on allowances for the known sources of possible systematic error.

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EPA method

EPA United States Environmental Protection Agency

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EPA Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma–Mass Spectrometry

This document is included in Selected Analytical Methods for Environmental Remediation and Recovery (SAM).

Citation:

U.S. EPA. 1994. "Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry," Revision 5.4. Cincinnati, OH

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