

ICP-MS Project Report

Project Request Details

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Project Title: Elemental composition of monk relic

Sample Type: Monk relic

Requested Elements: Total Quant

PerkinElmer NexION 350D ICP Mass Spectrometer Settings

Ar Gas flow (L min ⁻¹)	1		
Ar Auxiliary gas flow (L min ⁻¹)	1.2		
Ar Plasma flow (L min ⁻¹)	18		
Cell A gas flow (NH ₃ , mL m ⁻¹)	0		
Cell B gas flow (He, mL m ⁻¹)	4.5		
Nebulizer gas flow (L min ⁻¹)	0.94		
Mode of operation	KED		
RF power (W)	1600		

ICP-MS – Method parameters

Elements mass	Total Quant		
Dwell time (ms)	100-500		
RPa	0		
RPq	0.25		
Calibration (µg/L)	50		
Replicates per sample	3		
LOD (µg/L)			
LOQ (µg/L)			
Internal standard (µg/L)			

Sample preparation

The monk relic sample was transferred to a 7 mL PTFE microwave digester vessel and 2.5 mL of Optima grade concentrated HNO₃ (67–69% w/w; Fisher Scientific) was added to the vessel. The vessel was then placed in a Milestone UltraWAVE microwave digestion system, which was ramped up to a temperature of 220 °C over the course of a 15-minute period and maintained at that temperature for a further 10 minutes to complete the digestion process. Sample aliquots of 250 µL were transferred to acid cleaned trace metal grade HDPE centrifuge tube (VWR) and diluted to 5 mL (factor of 20x dilution) using purified water with a resistivity ≥18.2 MΩ cm from a Milli Q system (Millipore, Merck).

A calibration standard was made volumetrically using a 100 mg/L Sigma Aldrich TraceCert multi-element standard and ~0.7 M HNO₃ stock solution made from Optima grade concentrated HNO₃ and purified water to ensure the calibrant and sample were matrix matched.

Measurements

Total quantification of the sample (elemental scan) was conducted using a Perkin Elmer NexION 350D Inductively Coupled Plasma Quadrupole Mass Spectrometer (ICP-QMS) under Kinetic Energy Discriminator (KED) mode at the London Metallomics Facility, King's College London. The introduction system to the instrument was a Cetac ASX-100 autosampler coupled to a SeaSpray glass nebulizer fitted to a quartz cyclonic spray chamber. Argon plasma flow and nebulizer gas flow rates were 18 L/min and 0.93 L/min, respectively.

Results

The absolute and proportional elemental composition of the monk relic is shown in the table and figure below. The monk relic contains 10 major elements that make up 99% of the total elemental composition while the remaining 73 elements represent ~1% of the monk relic (figure). The major elements include biologically important elements such as calcium, potassium and iron. Surprisingly, the monk relic contains a large proportion of silver (5%), silicon (5%) and gold (Au; 0.96 µg/L).

Element	Concentration (µg/L)
H	
He	
Li	0.107
Be	0.000
B	1.212
C	68.164
N	0.000
O	
F	
Ne	
Na	1068.132
Mg	330.405
Al	229.150
Si	394.505
P	173.391
S	
Cl	
Ar	
K	1570.135
Ca	2418.878
Sc	0.041
Ti	14.068
V	0.316
Cr	3.358
Mn	18.436
Fe	1302.260
Co	0.064
Ni	1.458
Cu	5.815

Zn	8.805
Ga	0.046
Ge	0.015
As	0.035
Se	0.019
Br	0.000
Kr	
Rb	0.417
Sr	0.931
Y	0.025
Zr	0.076
Nb	0.021
Mo	0.264
Ru	0.000
Rh	0.000
Pd	0.003
Ag	363.647
Cd	0.000
In	0.000
Sn	0.153
Sb	0.211
Te	0.000
I	0.067
Xe	
Cs	0.012
Ba	7.853
La	0.239
Ce	0.601
Pr	0.056
Nd	0.230
Sm	0.044
Eu	0.010
Gd	0.076
Tb	0.010
Dy	0.049
Ho	0.012
Er	0.027
Tm	0.004
Yb	0.021
Lu	0.002
Hf	0.005
Ta	0.001
W	0.092
Re	0.001
Os	0.000
Ir	0.000
Pt	0.000
Au	0.959
Hg	0.000
Tl	0.000
Pb	0.633
Bi	0.001
Th	0.019
U	0.016

