LEAD ISOTOPES AND ADDITIONAL ANALYTES IN NIST BONE ASH

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INTRODUCTION

Bones provide a means to evaluate pollution trends and to group specimens according to trace-element patterns. Bones are known [1] to accumulate some toxic elements (such as lead, Pb). There is special interest in Pb because its stable isotopes offer the potential to distinguish the source(s) of exposure. Nutritional elements in bone have been used to assess (retrospectively) the health of animals, while other trace elements in bone may serve to distinguish the regional origins of animals. Bone Ash (SRM 1400) from the National Institute of Standards and Technology (NIST) is a commonly used reference material. To expand the utility of this reference material by characterizing additional analytes, the data reported here were obtained from a multilaboratory effort. Method comparisons among laboratories can serve to verify results and procedures.

EXPERIMENTAL

This multilaboratory effort involved quadrupole and magnetic-sector inductively coupled plasma mass spectrometry (ICPMS), thermal ionization mass spectrometry (TIMS), matrix separation, flow injection, and interference assessments.

RESULTS AND DISCUSSION

Data for each analyte are shown below to the significant figures justified [2] by the measured precision. The data for Cd, Cu, Mn, Pb, Sr, & Zn from three labs range between 91% & 110% of the NIST values, providing confirmation of the dissolution of the ash and the reported digest volumes. Analytes found to be below detection limits (μg/g) in this bone ash include Be (<0.06), Lu (<0.007), Ta (<0.02), and Zr (0.08). The results for Co, Cr, Mo, Ni, and Ti in the bone ash differ more than 2 fold among the laboratories, indicating interferences.

This NIST Bone Ash was issued in December 1992 with a reference value for the total Pb but none for any of the Pb isotopes. Table 1 shows the agreement obtained between ICPMS and TIMS for the stable Pb isotopes in this bone ash. In comparison with the Common Lead Isotopic Standard (SRM 981) from NIST, the Pb in this bone ash is enriched in ^{206}Pb by 3.29% (on a relative basis) and diminished in ^{207}Pb by 3.65% (Figure 1). Table 2 shows the data obtained for some additional analytes (not listed by NIST) in this bone ash.

Table 1. Multilaboratory Data for Pb Isotopes in NIST Bone Ash (SRM 1400)

Isotope	TIMS Data Lab C (n=5)	ICPMS Data (% Abundance) Lab C (n=10)	Lab D (n=10)	Lab E (n=3)	mean	ICPMS % of TIMS
208Pb	52.4287	52.335	52.18	52.412	52.31	99.77
207Pb	21.2767	21.274	21.37	21.275	21.31	100.2
206Pb	24.9375	24.965	25.02	24.945	24.98	100.2
204Pb	1.3571	n/a*	n/a*	1.368	n/a*	100.8

Data are shown to the significant figures justified by the precision,
SRM = Standard Reference Material (NIST trademark),
TIMS = Thermal Ionization Mass Spectrometry,
ICPMS = Inductively Coupled Plasma Mass Spectrometry,
n/a = not available,
*The minor ^{204}Pb value (1.4255%) from the Common Lead Isotopic Standard (SRM 981) was used for Labs C & D because ^{204}Pb was not measured.

Figure 1. Abundance differences for Pb isotopes in Bone Ash vs. Common Lead Isotopic Standard

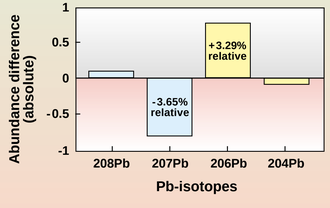


Table 2. Data for Additional Analytes in NIST Bone Ash (SRM 1400) from this Study (μg/g)

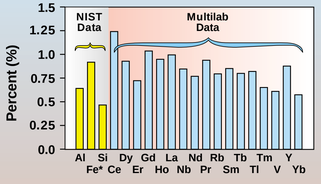
Analyte	Conc (%RSD)	Comment
Ag	0.0987 (8.3)	1 lab, n=9
Ba	240 (4.3)	1 lab, n=10
Bi	0.011 (3.3)	2 labs, n=9,9
Ce	0.821 (12)	2 labs, n=10,10
Dy	0.0479 (5.2)	2 labs, n=10,10
Er	0.0254 (10)	2 labs, n=10,9
Gd	0.064 (16)	2 labs, n=10,10
La	0.386 (21)	2 labs, n=10,10
Li	0.95 (14)	2 labs, n=10,10
Nb	0.170 (22)	1 lab, n=10
Nd	0.316 (5.9)	2 labs, n=10,9
Pr	0.0860 (14)	2 labs, n=10,10
Rb	0.71 (18)	1 lab, n=10
Sb	0.423 (2.9)	2 labs, n=9,10
Sm	0.0595 (7.9)	1 lab, n=10
Sn	0.183 (11)	2 labs, n=10,10
Tb	0.00963 (8.8)	1 lab, n=10
Th	0.123 (0.23)	2 labs, n=10,9
Tl	0.00712 (2.6)	1 lab, n=10
Tm	0.00343 (16)	1 lab, n=10
U	0.066 (4.1)	4 labs, n=10 or 11
V	0.769 (12)	3 labs, n=10 each
Y	0.288 (8.7)	1 lab, n=10
Yb	0.0183 (16)	2 labs, n=10,10

%RSD = Percent Relative Standard Deviation,
n = number of replicate digests used in averages (after Dixon outliers were excluded).

RESULTS AND DISCUSSION *(cont'd)*

It is possible that some analytes in SRM 1400 did not originate (totally or partially) in the bones. The unusually high NIST values for Al (530 μg/g), Fe (660 μg/g), and Si (1300 μg/g) in this reference material, as well as the concentrations for many of the additional analytes found in this multilaboratory study, are consistent with about 1% contamination of this bone ash by material from the Earth's crust [3] (Figure 2).

Figure 2. Percent of Earth's crust that MAY account for some analytes in NIST Bone Ash (SRM 1400)



Crustal data from CRC Handbook, 1995-96
*Fe is 1.17% without subtraction of Bone-Meal Fe
Avg. 20 analytes (%) = 0.82
SD = 0.18
RSD = 22

CONCLUSION

The data provided here will be useful to those interested in measuring these analytes in the presence of a bone-ash matrix, regardless of the origin of these analytes in SRM 1400.

REFERENCES

[1] *Trace Elements in Human and Animal Nutrition*, 3rd ed., E.J. Underwood, Ed., Academic Press, New York, 1971.

[2] *Statistical Manual of the Association of Official Analytical Chemists*, W.J. Youden and E.H. Steiner, AOAC, Arlington, VA, 1975, p. 59.

[3] *CRC Handbook of Chemistry and Physics*, 76th ed., CRC Press, New York, 1995-1996, p. 14-11.

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