

Accurate and Precise determination of Pb isotope ratio by single collector QQQ-ICP-MS: Application to environmental samples

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We have developed a new method for accurate and precise determination of Pb isotope ratios ($^{208}\text{Pb}/^{206}\text{Pb}$ and $^{207}\text{Pb}/^{206}\text{Pb}$) in environmental samples utilizing a single step column purification and subsequent determination by single collector quadrupole plasma mass spectrometry (SC-QQQ-ICP-MS; AgilentTM 8900). Our improved column method is characterized by low blanks (0.4 ± 0.2 pg; $n=9$), high yield ($93.4\pm 1.2\%$; 2σ , $n=9$) and isotopic fractionation free Pb elution utilizing small volume ($600\mu\text{L}$) of 6 mol L^{-1} HCl. This method is optimized for complex matrices such as seawater, dust, and sediment samples. We utilized 0.23 ng of Pb per analysis resulting in a total Pb consumption of 0.46 ng when analyzed in duplicate. The key advantages of our Pb isotope determination method are sub pico-gram levels of procedural blanks, rapidity of sample processing and analysis time (172s), low mass requirement (0.23ng per analysis), and a relatively high tolerance for potential matrix mismatch.

Quantitative separation of Pb from matrix elements was done by a single-step anion exchange chromatographic method utilizing teflon micro columns ($\sim 250\mu\text{l}$ wet resin volume), Biorad AG-1X8 (chloride form, 200–400 mesh) anion exchange resin. The average Pb isotopic composition of pure NIST 981 ($^{208}\text{Pb}/^{206}\text{Pb}=2.1681\pm 0.0034$, $^{207}\text{Pb}/^{206}\text{Pb}=0.9146\pm 0.0014$, 2σ , $n=40$) determined over 40 analytical sessions ($n=294$) is identical to certified values ($^{208}\text{Pb}/^{206}\text{Pb}=2.1681\pm 0.0008$, $^{207}\text{Pb}/^{206}\text{Pb}=0.9144\pm 0.0005$). Additionally, column processed NIST 981 loaded in pure form ($^{208}\text{Pb}/^{206}\text{Pb}=2.1678\pm 0.0018$, $^{207}\text{Pb}/^{206}\text{Pb}=0.9144\pm 0.0007$, $n=3$) and doped in seawater matrix (Pb:Na= $1:10^6$ ng/ng) ($^{208}\text{Pb}/^{206}\text{Pb}=2.1695 \pm 0.0058$, $^{207}\text{Pb}/^{206}\text{Pb}=0.9136 \pm 0.0011$, $n=19$) are analytically indistinguishable from certified values. We report an external reproducibility of 0.3% RSD for $^{208}\text{Pb}/^{206}\text{Pb}$ and 0.5% RSD for $^{207}\text{Pb}/^{206}\text{Pb}$, determined through repeat analysis ($n=21$) of multiple aliquots of ab-initio processed and column eluted NIST SRM 8704 buffalo river sediment. Comparison of Pb isotope ratios ($^{208}\text{Pb}/^{206}\text{Pb}$ and $^{207}\text{Pb}/^{206}\text{Pb}$) of column processed natural samples (soil, dust, and plant tissues) determined by our SC-ICP-MS method and established MC-ICP-MS method are statistically indistinguishable ($\Delta^{208}\text{Pb}/^{206}\text{Pb}=0.004$, $\Delta^{207}\text{Pb}/^{206}\text{Pb}=0.001$). To summarize, we have established a low blank, high precision and accurate method for rapid analysis of Pb isotope ratios