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; Agilent™ 8900). Our improved column method is characterized by low blanks (0.4±0.2 pg; n=9), high yield (93.4±1.2%; 2σ, n=9) and isotopic fractionation free Pb elution utilizing small volume (600µL) 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 (~250µl wet resin volume), Biorad AG-1X8 (chloride form, 200–400 mesh) anion exchange resin. The average Pb isotopic composition of pure NIST 981 ($^{208}\text{Pb}/^{206}\text{Pb}=2.1681±0.0034,$ $^{207}\text{Pb}/^{206}\text{Pb}=0.9146±0.0014,$ 2σ, n=40) determined over 40 analytical sessions (n=294) is identical to certified values ($^{208}\text{Pb}/^{206}\text{Pb}=2.1681±0.0008,$ $^{207}\text{Pb}/^{206}\text{Pb}=0.9144±0.0005$). Additionally, column processed NIST 981 loaded in pure form ($^{208}\text{Pb}/^{206}\text{Pb}=2.1678±0.0018,$ $^{207}\text{Pb}/^{206}\text{Pb}=0.9144±0.0007,$ n=3) and dopped in seawater matrix (Pb:Na=1:10$^6$ ng/ng) ($^{208}\text{Pb}/^{206}\text{Pb}=2.1695 ± 0.0058,$ $^{207}\text{Pb}/^{206}\text{Pb}=0.9136 ± 0.0011,$ 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 ($^{208}\text{Pb}/^{206}\text{Pb}$ and $^{207}\text{Pb}/^{206}\text{Pb}$) from mass-limited samples. This method is applicable to diverse environmental samples and utilizes readily available instrumentation.