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Lead isotope fingerprinting of nanoscale mineral particles via single particle inductively coupled plasma mass spectrometry

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The determination of Pb isotopic ratios in solid samples is of broad interest across geoscience. Measurements are performed *in situ* on individual grains (e.g. LA-ICP-MS), or bulk samples are digested and the Pb is measured with mass spectrometry (TIMS, MC-ICP-MS). While bulk measurements are useful for providing the average isotopic composition of a sample, individual particle types cannot be distinguished. Moreover, if multiple sources of Pb are present in a single sample, end-members are typically required for quantitative source apportionment. Finally, for many samples (e.g. soils, atmospheric particles), Pb is enriched in the fine-grained fraction. Therefore, a method to determine Pb isotope ratios in individual small particles is needed. Single particle ICP-MS is a powerful tool for analysis of nano and micro particles, with the development of spICP-time-of-flight-MS (spICP-TOF-MS) enabling precise multi-nuclide and multi-isotopic characterization in individual particles. Recently, spICP-MS measurements have also been performed on a multi-collector ICP-MS (spMC-ICP-MS) to improve precision in isotopic ratios. Thus far, studies of isotopic signatures in individual particles have been limited to synthetic particles to either confirm natural abundance ratios or measure enriched particles.

In this study we measured Pb isotope ratios in individual particles generated from mineral, rock, and ore samples using spICP-TOF-MS and spMC-ICP-MS. To accurately measure isotope ratios on the ICP-TOF-MS, we corrected the signal ratios for instrumental mass bias using a thallium double spike. After mass bias correction, we determined that unique isotope ratios could be measured in nanoscale galena particles of different origins, with large enough differences to overcome analytical uncertainty from the ICP-TOF-MS (^{206}Pb/ ^{207}Pb ranging from 0.89 to 1.30). The average ratios measured in the particles closely agreed with digested samples, and there was close agreement between the ICP-TOF-MS and MC-ICP-MS. Measuring the digested samples in single particle mode revealed the presence of undigested Pb particles in the rock powder sample, possibly indicating trapped sulfide grains in the silicate matrix, which could have implications for sample preparation for conventional Pb isotopic analysis. This study represents the first step in Pb isotope fingerprinting in individual particles, which would have broad applications across geoscience and environmental science.