An improved low-blank method for the preconcentration of Pb isotopes from seawater

EDWARD ALLEN BOYLE

Massachusetts Institute of Technology
Presenting Author: eaboyle@mit.edu

In order to measure high-precision Pb isotope ratios on seawater samples, ultra-low Pb in seawater (0.5-100 pmol/kg) has to be concentrated and separated from other seawater constituents. In the past, we have used $Mg(OH)_2$ coprecipitation to concentrate Pb and separate it from most sea salts followed by anion exchange purification. But this method has a problem with ocean waters that have high silicate concentrations because the Si is also concentrated, and when the $Mg(OH)_2$ precipitate is dissolved in a small volume for anion exchange purification, silica gel precipitates and prevents passage through the anion exchange column. To overcome this problem, we had switched to a batch Nobias PA1 chelating exchange resin method similar to that used for Fe, Zn, and Cd isotope measurements (Conway et al., 2013). But we found that when sufficient PA1 is used to get near-quantitative recovery from a two-step batch process, the PA1 blank is a significant factor in calculating the results. So more recently, we have switched to a combined $Mg(OH)_2$-PA1 method that reduces the PA1 blank by a factor of ~25. We start with an $Mg(OH)_2$ coprecipitation using high-purity NH_3 addition followed by gravitational separation of the precipitate from a 1 liter separatory funnel. This step is nearly quantitative and has an extremely low blank. The precipitate is then dissolved in 40 mL of dilute HCl and then buffered to pH 5-6. This volume is sufficient to avoid silica gel nucleation. We then add a small amount of PA1 in two batches with agitation for one day, separate the resin from the solution, and then extract the Pb with 1.2 mL of 0.1M HNO_3 for one day. This solution is then dried down, and then 400 μL 1.1M HBr is added for anion exchange purification. We will illustrate this method with data from the US GEOTRACES Pacific Meridional Transect (PMT) GP-15 between Alaska and Tahiti, comparing PA1 and $Mg(OH)_2$-PA1 stations and with new data from the northern portion of the section.

Reference: Conway, T.M, A.D. Rosenberg, J.F. Adkins, and S.G. John (2013) Analytica Chimica Acta 73:44-52.