A new procedure for high precision measurement of selenium isotopes using hydride generation MC-ICP-MS with double spike

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Selenium isotopes have been used to trace pollution sources and reconstruct paleo-redox conditions. However, high-precision determination of Se isotopes is still challenged by difficulties in chemical purification for various geological and environmental samples.

Here, utilizing a novel silicon (Si) based thiol resin (Si-$(CH_2)_3SH$), we developed a new procedure for high precision measurement of Se isotopes by hydride generation (HG)-MC-ICP-MS using a ^{74}Se-^{77}Se double spike (DS) technique. First, Se in sample solution mixed with DS was completely converted into Se (IV) in 5mol/L HCl. Then, the solution was adjusted to 6mol/L HCl. Most of iron was removed from sample through an Ag1-X8 anion resin column procedure. After Fe removal, the collected elute was diluted to 1 mol/L HCl and immediately loaded onto a Bio-Rad column filled with 0.25g Si-thiol resin. Matrix elements including Ge were eluted by 10mol/L HCl and H_2O. 1-1.5mL concentrated HNO_3 was used to extract Se from thiol resin. Samples were evaporated to incipient dryness and then dissolved in 0.1mL 0.1mol/L HNO_3. Se was again oxidized by adding 0.1mL 0.1mol/L $K_2S_2O_8$ and 0.8mL H_2O. The oxidized solution was loaded onto Ag1-X8 resin again to remove As using 0.03 mol/L HNO_3. Se was eluted by 0.6mol/L HNO_3 and evaporated to incipient dryness. Finally, followed by the sixth step in Zhu et al [1], Se isotopes were measured on a Nu Plasma II HG-MC-ICP-MS. Our new procedure makes Se purification simpler and better than traditional TCF procedure. Using our new method, we determined a series of reference materials (relative to NIST SRM3149): house standard MH495 $\delta^{82/76}Se$ = -3.41±0.1‰ (2SD, n=4), SGR-1b (USGS) $\delta^{82/76}Se$ = -0.20±0.1‰ (2SD, n=4) and 2711a (NIST) $\delta^{82/76}Se$ = -0.48±0.14‰ (2SD, n=5), which are in excellent agreement with the values reported previously.

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[1] Zhu et al. (2008) *Chinese J Anal Chem* 36, 1385-1390.