Chemical purification and isotopic analysis of Nickel by MC-ICPMS

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The use of the shortlived ^{60}Fe-^{60}Ni decay system ($T_{1/2}$ = 1.49 Myr) in cosmochemistry requires the ability to determine Ni isotopic ratios to high precision. Towards this end, two chemical procedures were established for the separation of Ni from metal and silicate matrices. Metal samples were digested in 6 M HCl and Ni was isolated using TEVA spec resin and high molarity HCl. Silicate samples were digested by standard HF-HNO_3 techniques, and Ni was purified in a three column procedure as follows: 1) separation of Ni from matrix elements in 1 M HNO_3 on cation exchange resin, 2) removal of Ca on TODGA resin in 1 M HNO_3, and 3) final purification of Ni on anion exchange resin in mixed HCl-$H_2C_2O_4$-H_2O_2 media. Both techniques facilitate Ni yields in excess of 99% and total procedural blanks less than 1 ng, which are insignificant compared to the amount of Ni analyzed (10-15 µg).

Ni isotopic compositions were determined by MC-ICPMS using the sample-standard bracketing technique. Samples were analysed in 5% HNO_3 and introduced into the plasma source using an Aridus desolvating nebuliser with Ar and N sweepgases. Ni isotopic compositions were determined in two separate analytical runs, covering the mass ranges from ^{57}Fe to ^{62}Ni and ^{61}Ni to ^{66}Zn, respectively. Samples and standards were analysed with signal intensities of 8 volts on ^{58}Ni in the low-mass routine and 3 volts on ^{62}Ni in the high-mass routine. Typical instrumental backgrounds after washouts were less than 0.001 V on ^{58}Ni and below detection limits on ^{62}Ni in the low- and high-mass routines, respectively. Each sample was analysed 8-12 times. Ni isotopic ratios determined in the low-mass routine were internally normalized to ^{61}Ni/^{58}Ni = 0.0167442 using the exponential law. Ni isotopic ratios in the high-mass routine were internally normalized to the ^{61}Ni/^{62}Ni obtained in the low-mass routine, also using the exponential law.

Ni isotopic compositions are reported in delta units as the per mil deviation of internally normalized isotope ratios in samples relative to standards. We achieve a long-term (6 months) external reproducibility for δ^{60}Ni, δ^{62}Ni, and δ^{64}Ni of 0.010‰, 0.015‰ and 0.040‰ in both synthetic standards and rock matrices.