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Analytical method for the determination of dissolved iron isotopes in polar snow and ice using small sample volumes

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Dissolved iron (dFe) is a limiting micronutrient for phytoplankton growth in the ocean, influencing oceanic CO_2 uptake and regulating global climate. To distinguish the relative importance of dFe fluxes from different sources to the ocean, stable Fe isotope analysis has emerged as a distinctive tool to constrain the dFe sources. Polar snow and ice are unique paleoclimate archives, providing records of past atmospheric composition. Despite their potential, determining dFe isotopic composition (δ^{56}Fe relative to the IRMM-524B standard as follows: $([^{56}Fe/^{54}Fe]_{sample}/[^{56}Fe/^{54}Fe]_{IRMM524B} - 1) \times 1000$) in snow and ice remains challenging because of extremely low dFe concentrations down to the picogram per gram level (1 pg = 10^{-12} g) and the small sample sizes available for analysis. Here, we present an ultraclean analytical protocol for determining dFe isotopes in snow and ice using less than 100 mL of sample. Since contamination is a critical factor affecting the reliable measurement of Fe isotopes, the entire cleaning and analytical procedures were conducted under ultraclean conditions (Class 10). The total procedural blanks were observed to be 357 ± 92 pg, which is equivalent to approximately 20% of the average dFe concentration in recent Greenland snow. This indicates that our ultraclean procedures attain high quality contamination control. The internal precision (2σ) of dFe isotope ratio measurement by multiple collector-ICP-MS was estimated to be 0.019–0.022‰ for 5 ng of IRMM-524B. To validate the effectiveness of our protocol, two ice core sections from the Vostok Antarctic deep ice core were selected from depths of 2984 and 3014 m, corresponding to 289 kyr and 297 kyr ago, respectively, during the penultimate glacial period. These sections show high concentrations of total dFe (51 ng and 11 ng, respectively) and the δ^{56}dFe values of 0.158 ± 0.16‰ and 0.243 ± 0.05‰. Crustal dust, the dominant source of atmospheric Fe deposition during cold glacial periods, has an average δ^{56}Fe value of 0.09 ± 0.10‰. Therefore, the dFe isotopic compositions measured in the Vostok samples are in general agreement with typical crustal dust composition, confirming the cleanness and effectiveness of the overall analytical process.