Technische Hochschule Lübeck
Department of Applied Sciences
Course of study: Biochemical Engineering (M.Sc.)

# Masterthesis

**Title:**

Development and Validation of an automated analyte-matrix separation for Fe isotope ratio analysis of sediment and carbonate samples.

**Summary:**

In the present work, the purification of Fe from sediment and carbonate samples was investigated and optimized using the low-pressure chromatography system, prepFAST–MC™ (Elemental Scientific, Omaha, Nebraska). A commercially available method originally used on blood samples was adapted and a method development was carried out in order to present a new separation scheme based on DGA resin. The commercially available method (CF-MC FeZnCu-2000) (Elemental Scientific, Omaha, Nebraska) is based on the ion exchange resin AG-MP1 with a column volume of 2 mL. The method was adapted based on the separation of different sediment digestions and the evaluation of the elution behavior of typical elements (Na, Mg, Al, K, Ca, Cr, Fe, Ni, Cu and Zn). The method enables the quantitative separation of matrix elements and subsequent separate elution of Cu, Fe and Zn. Regarding Fe, recoveries of 87% ± 6%, 91% ± 3% and 89% ± 2% could be achieved for different samples in recurring order (four separations per sample). A quantitative reduction of Cr from the Fe fraction was found for all samples. Ni was reduced by 72% ± 35%, 77% ± 19% and 91% ± 12%. The carry-over effect in relation to Fe was < 0.73% ± 0.02%. Furthermore, a new separation scheme for the purification of Fe is presented using a newly developed method based on the ion extraction resin DGA with a column volume of 3 mL. With reference to the same elements, the separation of Fe and Zn from other matrix elements is achieved: recoveries of 91% ± 2%, 97% ± 3% and 96% ± 9% for Fe and 91% ± 2%, 92% ± 2% and 96% ± 7% for Zn could be obtained for different samples in repeating order (five separations per sample). Cr was quantitatively removed in two of the three samples, in the third the removal was 97% ± 5%. Ni was reduced by 95% ± 3%, 93% ± 8% and 99% ± 1%. The carry-over effect in relation to Fe is 1.4% ± 0.1%. Both methods enable fast and cost-effective possibilities for the purification of Fe from sediment and carbonate samples. The newly developed method is also characterized by a more efficient separation of Ni and a low and reproducible carryover effect. The method is to be used for the purification of Fe for the subsequent isotope analysis on sediments. The corresponding sediments have already been digested within the work and elemental composition has been determined.

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