

Extraction and quantitative analysis of iodine in solid matrices

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^{129}I is gaining interest as a contaminant of concern at numerous federal and privately owned facilities. Several techniques have been identified to extract iodine from solid matrices; however, all of them rely on two fundamental approaches: liquid extraction or chemical/heat facilitated volatilization. While these methods are typically chosen for their ease of implementation, they lack the ability to result in total sample dissolution. Small partition coefficients have been measured for iodine on soil; therefore, extraction methods that do not result in total sample dissolution could underestimate the total iodine content of samples.

Approach

We conducted laboratory tests to define an extraction method contingent upon complete sample dissolution. Testing consisted of potassium nitrate/potassium hydroxide fusion of the sample, followed by sample dissolution in a mixture of sulfuric acid and sodium bisulfite. Direct analysis of the dissolved sample was performed via inductively coupled plasma mass spectrometry (Perkin Elmer Elan DRC II) using a tertiary amine (Spectrasol CFA-C) carrier solution.

Discussion of results

Use of the fusion extraction method resulted in complete sample dissolution of all solid matrices tested: sediment, glass samples containing low-levels of iodine, as well as tank waste material collected from the Hanford Site. Quantitative analysis of iodine (^{127}I and ^{129}I) was better than $\pm 10\%$ of certified reference standards, with the linear operating range extending more than three orders of magnitude (0.01 to 25 $\mu\text{g/L}$). Extraction and analysis of four replicates of standard reference material (San Joaquin Soil) from the National Institute of Standards and Technology, Gaithersburg, MD, resulted in an average recovery of 98% with a relative percent deviation of 6%.

Conclusions

These data highlight the success of the extraction and analytical techniques to quantitate total iodine (^{127}I and ^{129}I) in solid samples. Furthermore, these simple and cost-effective techniques can be applied to samples from multiple disciplines with little to no adaptation.

Depth profile of iodine and bromine in pore waters collected from the Nankai Trough

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Pore waters associated with methane have been found to show considerable enrichment of iodine (e.g. Fehn et al., 2003). In order to understand the detailed vertical distribution of iodine and bromine in marine sediments associated with methane hydrates, we have carried out ICP-MS analyses for a set of pore water samples collected from a borehole in the Nankai Trough area. Iodine concentrations in pore waters increased strongly with depth in the first 100 mbsf. The maximum iodine concentration of 60 ppm, corresponding to enrichment of 1000 times compared to seawater, was observed in layers below 100 mbsf, closely associated with the occurrence of methane hydrate. Concentrations of bromine also followed the iodine pattern with depth, but the highest enrichment compared to seawater (80 ppm) was only a factor of about 2. The iodine concentrations in the depth of 100 - 200 mbsf varied considerably. A similar depth profile was also observed for bromine and chlorine, indicating the presence of methane hydrate in this horizon. Iodine concentrations in solid phase of the sediment, after squeezing of pore fluid, were considerably lower than those in pore waters. The results indicate that most of iodine associated with gas hydrates resides in the pore waters under reducing conditions.

Reference

Fehn U. et al., (2003) *Geology*, 31, 521-524