

RAPID FUSION METHOD FOR PU, NP, AM AND CM IN 10 G SOIL SAMPLES

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Large soil aliquots are often needed to enable lower detection limits to be met for the assay of plutonium, neptunium, americium and curium in environmental soil samples. This laboratory has previously reported rapid fusion methods for 1-2 g soil samples, as well as 5 g soil samples after acid digestion with HF to lower Si levels. The scientific literature typically shows lower tracer yields (50-70%) for the analysis of soil samples. Tracer yields are often even lower when larger sample aliquots are analyzed. Testing in this laboratory has shown that large amounts of residual soil after acid digestion with HF tend to stubbornly retain plutonium and other actinides, despite successive treatments with nitric acid /boric acid. This may be one cause of lower tracer yields in some existing methods in the literature that utilize acid digestion with HF where a soil residue remains.

Rugged soil dissolution methods are essential. the recent failure by ~80% of participating labs in MAPEP Session 30 to digest insoluble uranium in the MAPEP soil samples, even using acid digestion even with HF, points to the need for robust sample digestion.

A new rapid fusion method has been developed by the Savannah River National Laboratory (SRNL) to prepare 10 g soil samples in just hours. The soil aliquot is heated for 1-2 hours in a 250 ml zirconium crucible to destroy organics, then fused with sodium hydroxide. This enables complete dissolution of the sample. No pre-treatment with HF to remove Si prior to fusion is required. Sample preconcentration techniques utilized with large seawater samples containing large amounts of Ca and Mg salts were applied. A new stacked TEVA Resin + DGA Resin column method was employed to separate the Pu, Np, Am and Cm isotopes in the 10g soil samples. This approach has a sample preparation time of <8 hours.

Sodium hydroxide fusion offers advantages over other fusion options, and in combination with iron/titanium hydroxide and lanthanum fluoride pre-concentration steps presents a rapid effective approach to soil analysis, even with large soil samples. Test results with MAPEP soil containing refractory Pu, as well as Am, Cm and Np will be presented.