Microwave-assisted decomposition of solid matrices, and analysis of radiostrontium and plutonium.

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Soil, fish, vegetation, crops, and glass fiber filter were decomposed using a close-vessel microwave oven system. One-gram samples were treated with mineral acids, used singly, or combined. The acid strength, volume, pressure, and decomposition time were varied to determine optimum conditions.

Fish, vegetation, and crops were dry-ashed at 650°C for 24 or 48 hours. Soil and glass fiber filters were dry-ashed at 550°C, for 16 hours.

Silica containing samples were decomposed in a two step process. Soil samples were treated with concentrated hydrofluoric acid. Remaining solids were dissolved with a boric acid/mineral acid mixture. Glass fiber filters were decomposed with a dilute hydrofluoric acid solution. Remaining solids were dissolved with dilute nitric acid. Hydrofluoric acid was more effective when used alone than when combined with hydrochloric and/or nitric acid.

Fish and crop samples were decomposed in a single step using a dilute nitric acid solution.

Residues were analyzed by X-ray spectrometry to guide sample treatment.

Strontium carrier and plutonium tracer were used to study the behavior of these nuclides during the digestion step, and to determine the chemical yield of the final separation. The yield was determined gravimetrically and by alpha spectrometry.

Under the most favorable conditions, greater than 99.5% of the sample matrix was dissolved with less than 40cc solution. This was achieved within one hour for the single step, and two hours for the two step process. Carrier and tracer yields of 70% or better were obtained in these cases. The final digestate, a dilute mineral acid solution, is amenable to chemical purification.

Further study is in progress to determine the feasibility of dissolving larger sample sizes. Preliminary results suggest that there is no simple linear relationship between sample size and reagent volume.