

Mixed Analyte Performance Evaluation Program - MAPEP

The Performance Testing (PT) material used in the MAPEP is designed to test the accuracy of analytical procedures on a variety of samples being submitted for analysis to the United States Department of Energy (DOE). To address specific analytical issues routinely encountered on actual samples, MAPEP varies the analytes, matrices, concentrations, solubility, chemical and radiological interferences and other parameters that affect the accuracy of the results.

Insoluble forms of plutonium, uranium, and other analytes are often found in analytical samples of interest to DOE. It is well known that these compounds are not soluble and do not dissolve even with extended treatment with concentrated HNO_3 and HCl acids. Even when HF is added to the HNO_3/HCl mixture, the dissolution is slow; prolonged aggressive treatment is needed to achieve complete dissolution of the sample.

The uranium present in a naturally occurring sample of soil is disproportionately distributed between two distinct fractions with different solubilities. One fraction contains uranium that is fairly soluble in mixtures of concentrated HNO_3 and HCl acid. The other fraction contains uranium that is fundamentally insoluble in these acids and will not dissolve without additional rigorous and prolonged treatment with HF . "Rigorous and prolonged" is a highly subjective term because the concentration of the insoluble form is unknown and there is no definitive way to determine when the dissolution is complete. However, the use of molten salt fusion techniques ensures rapid and complete dissolution of the entire sample.

Soils used in previous MAPEP distributions contained a relatively low concentration of the more acid insoluble fraction containing the uranium. The total amount of this insoluble form was not large enough to show the inaccuracy of the measurement when an inadequate dissolution technique such as acid leaching without HF was employed.

The soil used for MAPEP Series 30 contained a much higher concentration of this naturally occurring and more insoluble form of uranium. The total amount of this insoluble fraction was high enough to contribute significantly to and demonstrate the inaccuracy of the analytical measurement when an inadequate dissolution technique such as acid leaching without HF is employed.

The laboratories using chemical procedures capable of dissolving the entire soil sample reported accurate results; while the laboratories using dissolution techniques that were unable to dissolve the insoluble form of the uranium reported results that were approximately 60% low.