

## The Mixed Analyte Performance Evaluation Program Sample Dissolution

The Performance Testing (PT) material used in the MAPEP is specifically 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<sub>3</sub> and HCl acids. Even when HF is added to the HNO<sub>3</sub>/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<sub>3</sub> 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. Laboratories should use chemical procedures capable of dissolving the analytes of interest in the complex matrices encountered in real world samples.

MAPEP's philosophy regarding sample dissolution is:

- Results should accurately reflect what is in the sample.
- Chemical dissolution techniques should address the common problems encountered in real world samples.
- Results obtained by acid leaching of the sample will not be accurate if insoluble compounds are present.
- There is no way to determine if insoluble compounds are present without analysis.
- Leaching techniques should only be used when proven to be effective and demonstrated to produce accurate results equal to total dissolution methods.
- MAPEP does not mandate the use of specific analytical techniques; MAPEP evaluates analytical results compared to NIST-traceable reference values.

There are many dissolution methods in the scientific literature that may be appropriate for real-world samples found in the DOE complex. MARLAP devotes an entire chapter to sample dissolution. <http://www.epa.gov/radiation/docs/marlap/402-b-04-001b-13-final.pdf>. This chapter not only discusses the vital importance of sample dissolution, but also provides an overview of many different sample dissolution techniques. Other publications are found on the MAPEP web site <http://www.id.energy.gov/resl/mapep/mapep.html>.