

KEYNOTE PRESENTATION 2

David S. Sill

Senior Technical Manager for Chemistry at the Department of Energy's Radiological and Environmental Sciences Laboratory

Biography

David Sill is currently the Senior Technical Manager for Chemistry at the Department of Energy's Radiological and Environmental Sciences Laboratory where he has direct technical oversight of the Mixed Analyte Performance Evaluation Program and DOE Laboratory Accreditation Program for Radiobioassay. He has over 30 years of experience as an analytical radiochemist.

Mr. Sill started his storied analytical career in 1983 working as a bench chemist for the contractor at the INL. During the next 11 years, he had the opportunity of a lifetime; learning from and working with the "Jedi Master" of radiochemistry, Claude W. Sill. This awe inspiring experience shaped and molded Mr. Sill's scientific thought process; an experience he will never forget and for which he is eternally grateful.

Mr. Sill's professional interests and experience are in the areas of dissolution of difficult to dissolve sample matrices containing siliceous and other refractory material; and development of analytical procedures for the determination of the actinides in a variety of different sample matrices.

He has been instrumental in developing proficiency testing samples specifically designed for MAPEP, DOELAP and specialized proficiency testing programs over the last twenty years that challenge analytical laboratories with real world matrices and analytes of interest to DOE. In the last five years he has lead his RESL chemistry staff in the development of new Certified Reference Materials containing homogeneously distributed radionuclides, including the actinides, that are directly traceable to NIST which are not currently available to the analytical community.

He has directly worked with the National Institute of Standards & Technology to develop a one of a kind Radiological Traceability Program unique to RESL and NIST. Through this Radiological Traceability Program RESL is directly traceable to NIST through proven demonstration of production of proficiency testing materials and analyses of NIST prepared proficiency testing materials. Through his leadership RESL has also developed a proven Quality System through the NIST/RESL Radiological Traceability and RESL's accreditation to the International Standards Organization 17025 for Chemical Measurements, 17043 as a Proficiency Testing Provider and the latest G34 as a Certified Reference Material Provider.

Mr. Sill's professional career has been dedicated to improving the state of the analytical art with respect to radiochemical measurements and developing Reference Materials that test the actual problems encountered in real world samples.

He has presented numerous talks and published papers in peer reviewed scientific journals on the subject of analytical radiochemistry and quality control.

Contrary to popular belief he is an all-around nice guy, loves molten salt fusions and despises leaching of samples with nitric acid.

Contact information:

Email: sillds@id.doe.gov

THE IMPORTANCE OF TOTAL SAMPLE DISSOLUTION AND GUIDELINES FOR THE PREPARATION AND USE OF STANDARDS TO EVALUATE PERFORMANCE OF RADIOCHEMICAL ANALYSES

David S. Sill

Extensive experimental evidence indicates that variable and incomplete sample dissolution is the major cause of the inaccurate results being routinely reported by radioanalytical laboratories. This conclusion is easily demonstrated by analysis of the residues remaining after the sample has been "dissolved" by an inadequate dissolution technique such as leaching with nitric and hydrochloric acids. The residue often contains a significant fraction of the radionuclide being determined.

Complete dissolution of the sample is undoubtedly one of the most important aspects of any chemical procedure. For a desired chemical reaction to take place the element of interest must be in a specific oxidation state; otherwise the intended reaction will not occur. If an analytical procedure is expected to produce an accurate result, the sample and the analyte of interest must be dissolved completely, and isotopic exchange of the analyte with tracer must be guaranteed. These elementary concepts should be well known and should be the foundation from which every analytical procedure is developed. Unfortunately, the initial dissolution of the sample is usually neglected; especially when dealing with compounds that are difficult to dissolve such as quadrivalent oxides, siliceous materials, and samples like feces that contain high concentrations of calcium and phosphate. The omission of the appropriate type of front-end dissolution chemistry, by itself, will lead to the failure of even the finest chemical procedure. Isotopic exchange of the tracer with the analyte of interest is usually assumed rather than guaranteed. Even when dissolution techniques properly address the matrix and analyte of interest, the rigor necessary to control the oxidation state and prevent hydrolysis are too often overlooked. The importance of the appropriate type of front end dissolution technique and the conditions needed to avoid hydrolysis of large ter- and quadrivalent elements cannot be over emphasized.

It is frequently assumed that because an isotopic tracer was used in the analysis that exchange with the element being determined will be immediate and complete, and the accuracy of the analytical results is assured. Nothing could be further from the truth, isotopic exchange between an acid-soluble tracer and the analyte of interest does not occur with insoluble or refractory compounds. If complete sample dissolution is not achieved, use of isotopic tracers will not result in accurate results because the tracer recovery will apply only to the portion of the sample that was dissolved.