

**Project Title: Radiochemical Analysis by High Sensitivity Dual-Optic Micro X-ray Fluorescence**

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**Research Objective**

A novel dual-optic micro X-ray fluorescence instrument will be developed to do radiochemical analysis of high-level radioactive wastes at DOE sites such as Savannah River Site and Hanford. This concept incorporates new X-ray optical elements such as monolithic polycapillaries and double bent crystals, which focus X-rays. The polycapillary optic can be used to focus X-rays emitted by the X-ray tube thereby increasing the X-ray flux on the sample over 1000 times. Polycapillaries will also be used to collect the X-rays from the excitation site and screen the radiation background from the radioactive species in the specimen. This dual-optic approach significantly reduces the background and increases the analyte signal thereby increasing the sensitivity of the analysis. A doubly bent crystal used as the focusing optic produces focused monochromatic X-ray excitation, which eliminates the bremsstrahlung background from the X-ray source. The coupling of the doubly bent crystal for monochromatic excitation with a polycapillary for signal collection can effectively eliminate the noise background and radiation background from the specimen. The integration of these X-ray optics increases the signal-to-noise and thereby increases the sensitivity of the analysis for low-level analytes. This work will address a key need for radiochemical analysis of high-level waste using a non-destructive, multi-element, and rapid method in a radiation environment. There is significant potential that this instrumentation could be capable of on-line analysis for process waste stream characterization at DOE sites.

**Research Progress and Implications**

This report summarizes work after 9 months of a 3-year project as of May 2002. The project is 3 months ahead of the original milestone timetable. The X-ray optics were designed and constructed and performance tested. The associated instrumentation was purchased and a breadboard instrument constructed of the components. This breadboard system was tested with a model analyte system utilizing 1 M sodium chloride as a test matrix for the anticipated sodium hydroxide matrix from the sites. In addition, an iron 55 source was also used to determine the radiation background reduction with the dual capillary optic design. This breadboard system was designed with a 100-mm focal distance for both the excitation and detection optics. This was primarily chosen to allow for additional shielding for high radiation field use. The testing on the model system showed that this focal distance compromised the sensitivity of the X-ray detection system and did not screen the radiation from the iron 55 source as effectively as with a short focal distance optic on the detection side. The model system consisted of a 100 ppm solution of lead, platinum, palladium, copper, strontium and molybdenum (as a substitute for technetium). All the target elements were detected at 100 ppm with a 1200-sec acquisition time except for palladium due to the inability to effectively excite this element with the polycapillary optic. The results clearly show that we do not need to experiment with the filters. The air filter is sufficient to remove the lower energy X-rays that could swamp the detector from the high concentration sodium and chlorine of the matrix. This was not evident and simplifies the instrument experimentation. Some doubly curved crystal (DCC) measurements were made on the same analytes. The results showed improved S/N over the capillary excitation, however the larger excitation spot size requires a comparable collection optic to maximize the dual optic approach. The results indicate that we need to determine in conjunction with Savannah River and Hanford contacts whether they need higher sensitivity. This will determine if we need to redesign the optics for the prototype for year 2 of our research effort. In addition, the palladium issue needs to be addressed as well. Additional feedback is necessary to determine if we have to incorporate a helium purge to acquire the L line of palladium. Overall the results are encouraging and we will endeavor to iterate with the site contacts to insure that the instrumentation will address their HLW measurement needs.

### **Planned Activities**

06/01/02-08/01/02 – iterate with site representatives to determine best compromise of sensitivity and elemental range for desired HLW process lines.

07/01/02 – 09/30/02 – redesign optics for optimized sensitivity and working distance; model expected radiation field and needed shielding for instrumentation in anticipation for design work in FY'03/

07/01/02 – 11/01/02 – test Mylar resistance to NaOH and its lifetime for future model testing

09/01/02 – initiate design of prototype system and potential shielding

### **Information Access**

One Page Adobe Acrobat File attached.

### **OPTIONAL ADDITIONAL INFORMATION**

### **OPTIONAL PROPRIETARY INFORMATION**