

**Separation and Collection of Iodine, Sulfur, and  
Phosphorous Anion Complexes for Subsequent  
Radiochemical Analysis (U)**

by

A. A. Ekechukwu

Westinghouse Savannah River Company

Savannah River Site

Aiken, South Carolina 29808

R. A. Dewberry

**MASTER**

A document prepared for EMERGING TECHNOLOGIES IN HAZARDOUS WASTE MANAGEMENT VIII at  
Birmingham from 09/09/96 - 09/12/96.

DOE Contract No. DE-AC09-89SR18035

This paper was prepared in connection with work done under the above contract number with the U. S.  
Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S.  
Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper,  
along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

**DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED**

#### **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

### **DISCLAIMER**

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161.

PREPRINT EXTENDED ABSTRACT  
Presented at the I&EC Special Symposium  
American Chemical Society  
Birmingham, AL September 9-12, 1996

SEPARATION AND COLLECTION OF IODINE, SULFUR, AND  
PHOSPHORUS ANION COMPLEXES FOR SUBSEQUENT  
RADIOCHEMICAL ANALYSIS

A.A. Ekechukwu, R.A. Dewberry  
Savannah River Technology Center

SUMMARY

We developed a method to separate anion complexes of radioactive sulfur, iodine, and phosphorus to enable determination by radiochemical techniques. This method involves ion chromatographic separation of the anion complexes from other highly emitting radioactive species such as cesium-137 and strontium-90 which interfere with radiochemical analysis. We essentially use the ion chromatograph as a sample pretreatment method. The samples are injected onto a cation exchange column which allows the anions to pass through while retaining the positively charged species. If more than one negatively charged radioisotope is present, the sample is injected onto an anion exchange column and the individual anions separated from each other as well as from the cations. These anions are collected in the column effluent and measured by nuclear counting methods.

The method was developed to enable measurement of trace radionuclides in radioactive waste and in environmental samples. Trace radionuclides which are present in concentrations of only a few hundred disintegrations per minute per milliliter can be separated and then analyzed using liquid scintillation counting analysis. This paper establishes the separation and collection protocol, collection efficiencies for sulfur, iodine, and phosphorus, and overall efficiencies and detection limits for the separation and subsequent radiochemical analysis.

## INTRODUCTION

Our laboratory provides analytical support for many different types of research programs within SRTC and throughout the Savannah River Site. A wide variety of sample types including ground water, organics, laboratory waste, process control, sludge, soils, and others are received for many different analyses. These samples are both radioactive and non-radioactive and may contain hazardous materials such as RCRA metals, organics, and flammable solvents. Often these analyses involve measurement of trace constituents in the presence of higher concentrations of interfering species. This is especially common when measuring trace radionuclides due to the high concentration of radioactive cesium and strontium isotopes.

We have developed a method for analysis of low levels of radioactive phosphorus, sulfur, and iodine in the waste and environmental samples. This method involves ion chromatographic separation of isotopes from radioactive cesium and strontium isotopes by passage through a cation exchange column. The cesium and strontium (cations) are retained by the column while the iodine, sulfur, and phosphorus, present as anion complexes, pass through unretained. The radionuclides of the separated species are then analyzed using liquid scintillation counting and gamma spectroscopy.

Currently available methods for determination of low-level radionuclides involve chemical separation of each element individually and subsequent radioisotope analysis using nuclear counting techniques. The ion chromatographic method separates all of the elements simultaneously including ones which cannot be determined using existing wet chemical methods. This decreases the length of time required for analysis and the amount of waste generated, as well as limiting personnel radiation and chemical exposure.

## DISCUSSION

### Separation of Interfering Species

Samples which contain many different species present in widely differing concentrations are particularly difficult to analyze. As stated earlier, this chromatographic method was developed to be used in the analysis of low levels of radioactive iodine, phosphorus, and sulfur in the presence of high levels of radioactive cesium and strontium. Cs-137 and Sr-90 activity mask the signal from these

isotopes thus preventing their detection and quantitation. Qualitative identification of the separated isotopes was done by liquid scintillation counting. Standards and spikes samples were run to determine collection efficiency, counting efficiency, and overall recovery of radioisotopes. After the chromatographic separation was complete, the column was flushed with concentrated eluent to remove any remaining species which may have been retained by the column. This wash was counted by LSC and no radionuclides were found.

### **Problems and Interferences**

The major difficulty encountered with this analysis is the nature of the waste samples themselves. High level nuclear waste contains many cationic and anionic species at widely differing concentration levels including chemicals from plant separation processes, laboratory waste, fission products, neutron activation products, uranium, plutonium, and other actinides. Environmental samples often contain high levels of nitrates and chlorides. The high concentrations of individual anions and cations prevent the trace constituents from completely interacting with the column stationary phase and thus inhibit separation into discrete fractions. To counteract this effect, nonradioactive carriers (10 mL of the 1000 ppm standards of stable elemental isotopes per 1 mL of sample) were added to the samples. This enabled the trace constituents to more effectively compete with the column surface and separate effectively.

For radionuclide analysis, ion chromatography was used only to separate the cations into discrete fraction for subsequent analysis by liquid scintillation counting and gamma spectroscopy. The ions were collected in 1 mL fractions. Since only a 50 mL sample volume had been injected onto the column, this represented a 20 fold dilution which prevented detection of most of the trace constituents. To compensate for this, the injection valve was replumbed with a 500 mL injection loop, thus increasing the amount of sample injected onto the column and then recovered by ten-fold. Comparison of chromatographic runs using 1-ppm nonradioactive standard solutions showed no effect in precision or accuracy of the data or the effectiveness of the separation.

## CONCLUSION

We have developed a method which can separate low levels of radioactive anions from high levels of radioactive cations which would prohibit their analysis. The method can be used for both environmental and waste samples which are very difficult to analyze because of the wide variety of species found in them in varying concentrations. As with any chromatographic separation, high concentrations of elements in the sample will saturate the column stationary phase and must be removed from the sample prior to analysis. This method appears applicable to the analysis of trace radionuclides which can be separated and analyzed using liquid scintillation counting and gamma counting.