PKS
267
5/8/67

MASTER

IN-1081
April 1967

**A RAPID SOURCE PREPARATION TECHNIQUE
FOR HIGH-RESOLUTION ALPHA PARTICLE SPECTROSCOPY**

R. A. Deal and R. N. Chanda

IDAHO NUCLEAR CORPORATION
NATIONAL REACTOR TESTING STATION
IDAHO FALLS, IDAHO

U. S. ATOMIC ENERGY COMMISSION

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

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.

DISCLAIMER

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

Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

GESTI PRICES

RC. \$3.00 : NTV. 65

IN-1081
Issued: April 1967
Chemistry
TID-4500

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

A RAPID SOURCE PREPARATION TECHNIQUE FOR HIGH-RESOLUTION ALPHA PARTICLE SPECTROSCOPY

by

R. A. Deal

R. N. Chanda

IDAHO NUCLEAR CORPORATION

A JOINTLY OWNED SUBSIDIARY OF
AEROJET ALLIED
GENERAL CHEMICAL
CORPORATION CORPORATION

U. S. Atomic Energy Commission Research and Development Report
Issued Under Contract AT(10-1)-1230
Idaho Operations Office

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ABSTRACT

A rapid method has been developed for the preparation of sources for high-resolution alpha particle spectroscopy. The method involves the extraction of a nuclide into an organic phase. An aliquot of the organic is then transferred to a counting plate, evaporated to dryness and finally ignited leaving a mass-free plate. Sources prepared in this manner are of a quality equal to other methods currently in use.

CONTENTS

I. INTRODUCTION	1
II. EXPERIMENTAL	2
III. RESULTS	3
IV. REFERENCES	8

FIGURES

1. Alpha particle spectrum of Pu-240 from source prepared by TTA evaporation on a platinum plate. Energies are given in MeV with relative intensities in parentheses	4
2. Alpha particle spectrum of Cm-242 from source prepared by vacuum sublimation on a platinum plate. Energies are given in MeV with relative intensities in parentheses	5
3. Alpha particle spectrum of U-234 from source prepared by electro-deposition on a platinum plate. Energies are given in MeV with relative intensities in parentheses	6
4. Alpha particle spectrum of Ra-224 from source prepared by TTA evaporation on an aluminum plate. Energies are given in MeV with relative intensities in parentheses	7

A RAPID SOURCE PREPARATION TECHNIQUE
FOR HIGH-RESOLUTION ALPHA PARTICLE SPECTROSCOPY
(R. A. Deal and R. N. Chanda)

I. INTRODUCTION

High-resolution alpha particle pulse-height analysis with surface barrier detectors requires, in addition to stable electronic and detection systems, thin uniform sources that do not appreciably degrade the energy of the alpha particle leaving the source. For resolutions in the range of 25 to 100 keV FWHM, crudely prepared sources such as those obtained by evaporation from aqueous solution are often quite adequate, but when higher resolution is desired, some care must be taken in the preparation of the sources. In our work we routinely obtain α -resolutions of < 14 keV, and, thus, special care must be taken in the preparation of sources.

There are several methods for preparing α -sources currently in use. Among these are vacuum sublimation from a hot filament^[1,2], electro-spraying^[3,4], and electrodeposition^[5,6] [a]. All of these techniques, with the exception of electrodeposition, require quite elaborate apparatus. Another disadvantage with all three methods is the time involved in preparing the source and cleaning the equipment to prevent cross-contamination of samples. In addition, vacuum sublimation requires additional time to pump down the system. Certainly the simplest approach is direct evaporation of a solution onto a counting plate. The rapid method we describe below is the evaporation of a volatile organic solution on a metal disc. Sources prepared in this manner are of a quality equal to those prepared by the techniques referred to above.

[a] For a more complete review of sample preparation techniques, see: W. C. Parker and H. Slatis, "Sample and Window Techniques", in α -, β - and γ -Ray Spectroscopy, Kai Siegbahn (ed.), Vol. I, p 379-407 (1965), and Proceedings of the Seminar on the Preparation and Standardization of Isotopic Targets and Foils, M. L. Smith (ed.), AERE-R-5097 (1965).

II. EXPERIMENTAL

The procedure that was developed for the preparation of sources for semiconductor detector α -spectroscopy involves extracting the nuclide into an organic phase. An aliquot of the organic is then transferred to a counting plate, evaporated to dryness, and finally ignited leaving a mass free plate. TTA (thenoyltrifluoroacetone) was chosen since it is a very selective extraction reagent for all of the elements above lead ($Z > 82$) with the obvious exceptions of radon, francium, and astatine. (A complete summary of TTA extraction coefficients for the heavy elements is given by A. M. Poskanzer and B. M. Foreman, Jr. [7].)

The nuclide is first purified from daughters or contaminants and extracted into a 0.25 M TTA-benzene solution. The TTA-nuclide organic extract is then transferred to a plate by means of a micropipette or spitzer and evaporated to dryness under an infrared heat lamp. The organic extract can be contained in a small area on the plate by maintaining the edges at a higher temperature than the center [8], but we found that a slight depression in the center of the plate is sufficient to confine the material to a small area during evaporation. The organic extract is evaporated in 10 to 25 γ portions. After evaporation, the plate is flamed slowly over a burner until no organic deposits remain.

Any backing with a highly polished surface that does not melt or oxidize upon flaming will yield good plates. We have found 1-inch-diameter by 0.002-inch polished platinum discs to be satisfactory since scratch-free depressions are easily made using a fire-polished glass rod. In addition, because of the high melting point of platinum and its resistance to oxidation, no special care need be exercised during the flaming.

The only limitations as to the quantity of organic reagent that can be evaporated while still obtaining "good plates" are the purity of the TTA-benzene solution and the total mass of the nuclide extracted. In some instances, where a direct separation could not be obtained with TTA, other reagents such as EHPPA (2-ethyl-hexyl phosphoric acid) or Aliquat 336 [9] (tricaprylyl-monomethyl ammonium chloride) were first employed with the nuclide finally being extracted into 0.25 M TTA-C₆H₆ for the source preparation [10]. A partial separation could also be made on an ion-exchange column if so desired.

III. RESULTS

All sources were analyzed using ORTEC^[11] silicon surface barrier detectors (25 mm² active area) cooled to 0°C. We designed a counting chamber^[12] which permitted the detector to remain at constant vacuum and bias while changing samples. Signal amplification and analysis was provided by a solid-state system consisting of an FET preamplifier^[13], main amplifier and biased amplifier system (Ortec 220 or Tennelec TC 200-250), and a 1024-channel Nuclear Data Analyzer.

Sources prepared by the TTA evaporation method (Figure 1) have been analyzed and compared with sources prepared by vacuum sublimation (Figure 2) and electrodeposition (Figure 3). Within the capabilities of our detection and electronic systems, we have observed little or no difference in resolution^{[14][a]} between sources prepared by our technique and the other two source preparation techniques.

It is not imperative that platinum be used for the counting plate; in fact, a low Z backing material aids in reducing back scattering of alpha particles. Stainless steel backings have been used, with results comparable to platinum. It is, however, difficult to make a depression on a stainless steel plate without the use of a die. If the highest resolution is not required, sources prepared on 0.005-inch-thick aluminum (Figure 4) give a resolution of 15 keV which is quite adequate for most applications. In addition, low Z backing materials, ie, aluminum, are advantageous in certain types of experiments such as α - γ coincidence studies where critical absorption of gamma rays may be important.

The procedure described above has been used in the preparation of alpha sources of ≈ 4 mm diameter on 1-inch-diameter metal discs. Starting with the nuclide in a TTA-nuclide solution, a source can be prepared for counting in less than 5 minutes. While we have concerned ourselves exclusively with the actinide elements and the preparation of alpha sources, the TTA evaporation method should also find application in the preparation of electron sources, especially of samples of rare earth nuclides.

[a] Alpha partical energies and relative intensities are from Reference 14.

FIG. 1 ALPHA PARTICLE SPECTRUM OF Pu^{240} FROM SOURCE PREPARED BY TTA EVAPORATION ON A PLATINUM PLATE. ENERGIES ARE GIVEN IN MEV WITH RELATIVE INTENSITIES IN PARENTHESES.

FIG. 2 ALPHA PARTICLE SPECTRUM OF CM-242 FROM SOURCE PREPARED BY VACUUM SUB-LIMATION ON A PLATINUM PLATE, ENERGIES ARE GIVEN IN MEV WITH RELATIVE INTENSITIES IN PARENTHESES.

FIG. 3 ALPHA PARTICLE SPECTRUM OF U-234 FROM SOURCE PREPARED BY ELECTRO-DEPOSITION ON A PLATINUM PLATE, ENERGIES ARE GIVEN IN MEV WITH RELATIVE INTENSITIES IN PARENTHESES.

FIG. 4 ALPHA PARTICLE SPECTRUM OF RA-224 FROM SOURCE PREPARED BY TTA EVAPORATION ON AN ALUMINUM PLATE, ENERGIES ARE GIVEN IN MEV WITH RELATIVE INTENSITIES IN PARENTHESES.

IV. REFERENCES

1. N. Jackson, "Vacuum Sublimation Apparatus for Preparation of Thin Sources of Active Materials", J. Sci. Instrum., 37 (May 1960) pp 169-171.
2. J. Milsted, The Preparation of Thin Alpha Sources by Vacuum Sublimation, AERE-C/R-1379 (February 1954).
3. D. J. Carswell and J. Milsted, "A New Method for the Preparation of Thin Films of Radioactive Material", J. Nucl. Energy, 4 (January 1957) pp 51-54.
4. P. S. Robinson, "The Production of Radioactive Sources by the Electro-spraying Method", Nucl. Instrum. Methods, 40 (February 1966) pp 136-140.
5. H. J. S. Sand, Electrochemistry and Electrochemical Analysis, 1, Blackie and Son Ltd., London (1946).
6. W. Parker, M. De Croes, and K. Sevier, Jr., "Some Methods in the Preparation of Radioactive Materials for Use in Beta-Spectroscopy", Nucl. Instrum. Methods, 7 (April 1960) pp 22-36.
7. A. M. Poskanzer and B. M. Foreman, Jr., "A Summary of TTA Extraction Coefficients", J. Inorg. and Nuclear Chem., 16 (February 1961) pp 323-336.
8. Ibid, p 334.
9. Trademark of General Mills, Kankakee, Illinois.
10. R. N. Chanda and R. A. Deal, "Alpha Spectrum of Ac-227", Nuclear Technology Branches Quarterly Report, July 1 - September 30, 1965, IDO-17147 (April 1966) pp 19-20.
11. Oak Ridge Technical Enterprises Corporation, Oak Ridge, Tennessee, USA.
12. R. N. Chanda and R. A. Deal, Semiconductor Counting System for High Resolution Alpha Particle Spectroscopy, IN-1082 (1967).
13. K. F. Smith and J. E. Cline, "A Low-Noise Charge Sensitive Preamplifier for Semi-Conductor Detectors Using Paralleled Field-Effect-Transistors", IEEE, Trans. Nucl. Sci., NS-13, No. 3 (June 1966) pp 468-476.
14. E. K. Hyde, I. Perlman, and G. T. Seaborg, The Nuclear Properties of the Heavy Elements, Vol. II, Prentice-Hall, Inc. (1964).