ANALYSIS OF U AND PU RESIN BEAD SAMPLES WITH A SINGLE STAGE MASS SPECTROMETER*

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Restrictions in sample size caused by recently imposed limits on the amounts of fissionable isotopes that can be shipped has created a keen interest in minimizing the amount of sample required for analysis for Safeguards purposes. Use of the resin bead sampling technique[1] allows shipment of nanogram quantities of U and Pu for mass spectrometric analysis for isotopic composition and, through the use of isotope dilution, quantity.

Application of this technique to Safeguards has been investigated with a single-stage mass spectrometer.[2] The instrument has a 90-degree sector magnet with a 30 cm central radius of curvature. The instrument was designed and built at ORNL, but does not differ in any significant way from several widely used commercially available mass spectrometers. The small samples require a pulse-counting detection system to supply the necessary sensitivity.

To evaluate the single stage-resin bead combination, several samples were analyzed. Standards (Table 1) gave results in good agreement with NBS certified values. Other samples, previously run on multi-stage instruments, were analyzed and gave good agreement with the earlier results (Table 2).

Precision of our results is not as good as that obtainable under the optimum conditions of very large samples and a Faraday cup collector; however, our results are comparable in quality to those obtainable from current integrating detectors. External precisions of ±0.5% are obtained on isotopic ratios of about 0.01; precisions on quantitative measurements are ±1.0%.

Table 1. Results from Standards

	NBS-010		NBS-500	
	234/235	235/238	234/235	235/238
NBS	0.00538	0.01014	0.01043	0.9998
ORNL	0.00549 ±0.00032	0.01013 ±0.00004	0.01041 ±0.00010	1.0006 ±0.0036
	8 analyses		13 analyses	

	NBS-947			
	238/239	240/239	241/239	242/239
NBS*	0.00370	0.24147	0.04309	0.01559
ORNL	0.00377 ±0.00013	0.24119 ±0.00069	0.04327 ±0.00031	0.01557 ±0.00021
	22 analyses			

*Corrected to August 13, 1978.

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Table 2. Results from Comparison Samples

SAU-14

U	No. of Analyses	234/235	235/238		
2-stage		0.01030	0.01216		
1-stage	4	0.01015 ±0.00025	0.01220 ±0.00008		
Pu		**238/239**	**240/239**	**241/239**	**242/239**
2-stage		0.0093	0.3116	0.1020	0.03149
1-stage	4	0.0097 ±0.0006	0.3121 ±0.0008	0.0992 ±0.0008	0.03153 ±0.00011

K-1

U	No. of Analyses	234/235	235/238	238/233	
2-stage (unspiked)	2		0.00198 ±0.00001		
1-stage (unspiked)	4		0.00202 ±0.00003		
2-stage (spiked)	2	2.42 ±0.03	0.00236 ±0.00004	2.113 ±0.010	
1-stage (spiked)	4	2.34 ±0.04	0.00244 ±0.00004	2.096 ±0.013	
Pu		**238/239**	**240/239**	**241/239**	**242/239**
2-stage (unspiked)	2	0.00057 ±0.00028	0.05858 ±0.00022	0.00219 ±0.00001	0.00019 ±0.00001
1-stage (unspiked)	4	0.00022 ±0.00006	0.05862 ±0.00014	0.00224 ±0.00001	0.00037 ±0.00001
2-stage (spiked)	2	0.00033 ±0.00007	0.05863 ±0.00006	0.00223 ±0.00001	0.3415 ±0.0002
1-stage (spiked)	4	0.00023 ±0.00003	0.05864 ±0.00024	0.00231 ±0.00001	0.3397 ±0.0017

References

1. R. L. Walker, R. E. Eby, C. A. Pritchard, and J. A. Carter, Anal. Lett., 7, 563 (1974).

2. D. H. Smith, ed., USDOE Report ORNL/TM-6485, Oak Ridge, TN, November, 1978.