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METHOD AND APPARATUS FOR
QUANTITATIVE NEUTRON ACTIVATION
ANALYSIS OF LARGE SAMPLES

Livermore, California

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ABSTRACT

A method and apparatus have been devised to irradiate multiple samples of large physical size simultaneously in a nonuniform neutron flux. A capsule containing the samples and flux monitors is rotated about an axis at constant speed with samples fixed in a symmetrical geometry so that each position receives the same integrated neutron flux.

INTRODUCTION

There are several methods for accomplishing a quantitative neutron activation analysis with samples that the analyst generally handles. If the sample is physically small, he will perhaps have a similar known sample, called a flux monitor, placed in close proximity to the unknown sample so that the samples are subjected to the same neutron flux environment. If the sample is large, he may choose to add a suitable flux monitor internally mixed with the unknown sample but of different elemental character. Ideally, it is preferable to monitor the flux using the same isotope as that which one would produce upon exposure of the unknown sample to the neutron flux. This necessitates external monitoring.

In the case where the sample is large, external monitoring becomes difficult to accomplish without serious errors due to the nonuniformity of the flux over the large dimensions involved. This report describes a method and apparatus for overcoming the difficulty.

EXPERIMENTAL

The neutron source used in this work was provided by the C-curve irradiation ports or S-tubes of the Livermore Pool Type Reactor (LPTR).¹ These 4-in.-i.d. tubes are water-filled and curved on a constant 10-foot radius for shielding purposes since they terminate near the core. The length of the S-tubes is approximately 11 feet.

The apparatus provides for placing the samples and flux monitors about an axis and rotating them at a constant rate about this axis. The arrangement of all samples is completely symmetrical, so that they all experience an equal flux exposure for an equal length of time.

The apparatus is constructed entirely from polyethylene, except for a rubber O-ring seal in the capsule (Fig. 1). Since the S-tubes (Fig. 2) are filled with water, the addition of a lead donut was required to overcome the buoyancy of the capsule. The speed of rotation was fixed at 2 rpm. This speed is adequate when the isotope half-life is 1 hour or more. Irradiations were usually of 1 to 2 hours duration. For this length of irradiation and a thermal neutron flux of 5×10^{12} n/cm²-sec, the polyethylene had a useful life of at least 40 hours. The O-rings were discarded after each use.

All samples irradiated in this work were liquid samples. They were heat-sealed in polypropylene tubes whose outside dimensions were 115 mm by 15 mm with a capacity of 13 ml. These were chosen over the readily available polyethylene conical tubes since their consistency in dimension produced equal solution height for all tubes when resting on the base of the clamping device (see Fig. 1). After irradiation the polypropylene tubes were centrifuged and cut open with a single-edge razor blade heated to redness. The solution was then transferred quantitatively to centrifuge tubes containing suitable carriers, and radiochemical separations were performed.

RESULTS

Three duplicate samples were tied together in a cluster so that they were as close together as was physically possible. These samples were packaged in the tubes described above. They were then irradiated in the S-tube without rotation. The results disagreed by as much as 25% (Table I).

¹ John B. Radcliffe, Jr., "The Livermore Pool Type Reactor (LPTR)," UCRL-4919.

Fig. 1. From left to right: capsule for enclosing samples, with lead donut weight at top; clamping device, which fits inside capsule, shown loaded with six samples in polypropylene tubes with tops heat-sealed and trimmed for ease of fit and alignment; empty clamping device.

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Fig. 2. View showing S-tube entrance of LPTR, motor used for rotation, and 1/2-inch polyethylene rod used to connect capsule and motor over 12-foot distance.

When the irradiation was repeated using the device to rotate the cluster of tubes, the error was reduced to $\pm 1.3\%$. When six duplicate samples were irradiated using the apparatus shown in Fig. 1, the results agreed to $\pm 1.3\%$.

Table I. Comparison of uniformity of activation of three identical samples in a cluster, with and without rotation during irradiation.

Sample No.	Activation, in 10^4 cpm of Eu^{152} at zero time	
	Without rotation	Rotated 2 rpm
1	8.28	2.99
2	10.01	2.93
3	8.04	2.96

A word of caution might be appropriate here. The environment of the flux monitor should resemble the environment of the unknown. For example, a high Cl^- concentration represents a large neutron sink; consequently both standard and unknown should have approximately the same chloride concentration. An attempt should be made to avoid or keep at a minimum substances which will depress the effective flux and also to match standard and unknown. A 6 M Cl^- concentration was found to depress the effective flux by 9.7%.

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