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**NEUTRON - CHARGED PARTICLE REACTIONS:
THE ANALYSIS OF THIN LITHIUM AND BORON FILMS**

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INTRODUCTION

The most familiar application of a nuclear technique for chemical analysis is that of neutron activation, which depends upon an n, γ or n -charged particle reaction to produce a radionuclide whose activity can be conveniently measured. Thus, certain a priori limitations are imposed; e.g., the product radionuclide must have (1) a convenient half-life, preferably a few minutes to several hours, (2) characteristic radiations which can be resolved from other activities produced, and (3) high yield relative to other activities.

Rather than measure a fraction of the radioactivity produced by nuclear reactions, one can detect the reactions as

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they occur.^{1,2} The products of such prompt phenomena include capture gamma rays, product neutrons, and emergent charged particles such as fission fragments, alpha particles, and protons. These reactions may be induced by neutrons, charged particles, and in some cases gamma rays.

The prompt phenomena resulting from neutron irradiation offer several advantages: e.g., (1) the analysis does not depend upon the half-life of the product nuclide, (2) the sensitivity is increased by as much as several orders of magnitude, (3) there is greater selectivity due to the highly characteristic nature of prompt phenomena, and (4) the method of analysis may be absolute rather than empirical. Measurement of prompt phenomena depends only upon the characteristics of the reaction and is independent of the properties of the product nuclide.

The fundamental equation for activation analysis is

$$A = d/s = N \sigma \phi (1 - e^{-\lambda t}) \quad (1)$$

and A represents a fraction of the total radionuclide produced during irradiation. The comparable equation for a prompt phenomenon is

$$R = N \sigma \phi t \quad (2)$$

where R is the total number of reactions which have occurred. In

Equation (1) the quantity $(1 - e^{-\lambda t})$ has a maximum value of unity and most frequently is significantly less than unity. When $t \gg \lambda$,

$$A = N \sigma \phi. \quad (3)$$

Consequently, analysis by measurement of a prompt phenomenon is more sensitive than activation analysis by a factor of t .

The increased selectivity results from several factors: there is often a dependence upon a critical threshold energy; there are, in general, fewer competing reactions of the same type; and those reactions which involve an emergent charged particle permit energy and intensity measurements by pulse-height spectrometry with resolution of the order of 1%.

A knowledge of the quantities σ and ϕ and the ability to measure R and t permits an absolute determination of N . The reactions

and

offer a very favorable test of this technique because of the high cross sections. The present study is an investigation of the non-destructive analysis of thin Li^6F and $\text{B}^{10}_2\text{O}_3$ films in the range 30

to 1000 Å.

EXPERIMENTAL

Thin films of Li^6F and $\text{B}^{10}_2\text{O}_3$ were bombarded with a beam of neutrons, and the product charged particles were detected with a silicon solid state device. The pulse-height distribution was recorded with a multichannel analyzer.

The thin films were prepared by vacuum deposition onto a Mylar substrate. Varying thicknesses were obtained by varying the distances from the filament. The films were mounted by sandwiching between 0.05 cm thick Al plates 4 cm x 4 cm provided with a 3.2 cm diameter hole in the center.

Neutrons of selected energies were obtained at the Brookhaven National Laboratory Graphite Research Reactor with a spectrometer equipped with a monochromating crystal. Energy selection was accomplished by reflections from a given plane at various angles (Figure 1). Fluxes were determined by foil activation for each of two different neutron spectrometers at the Brookhaven Graphite Research Reactor. For the initial experiments, the samples and

detector were mounted in an Al chamber 3" in diameter and 12" long provided with a Be window on the front face for entrance of neutrons, and having an 8"-long collimator of high density masonite inserted between window and sample. The window and collimator define a 1"-diameter neutron beam which impinges on the sample inserted at an angle of 45° to the beam. A solid state detector was mounted in the chamber perpendicular to the plane of the sample with a sample-detector distance of ≈ 3 cm. Since the counting efficiency of this chamber proved too low, a second chamber was constructed with a larger diameter detector and a source-detector distance of ≈ 0.5 cm (Figures 2, 3, and 4).

The detector used with the first chamber was a 1-cm diameter diffuse-junction silicon diode manufactured by RCA Canada, Ltd. The second chamber incorporated a 2-cm-diameter guard-ring surface-barrier detector prepared by G. L. Miller of the Instrumentation Division of Brookhaven National Laboratory. The signals from the detector were passed through a charge-sensitive preamplifier and a linear amplifier to a multichannel pulse-height analyzer.

The chamber was evacuated and maintained at a pressure of < 1 mm Hg during use. The counting efficiency was determined with Am^{241} standard prepared with a geometry identical to that of the

thin films by evaporation from solution onto insulin-coated Mylar film. An efficiency of 0.1% was determined for the first chamber and 8.5% for the second chamber.

RESULTS

The pulse-height distribution of the product charged particles was recorded for each sample. The spectra from the Li^6 and B^{10} samples are shown in Figures 5 and 6, respectively. In Figure 5 the two peaks arise from the 2.1-MeV alpha particle and the 2.7-MeV triton. The spectrum in Figure 6 also contains two peaks, due to the 1.8-MeV alpha particle and a 1.0-MeV Li^7 . The area under each peak in the spectrum, when corrected for detector efficiency, gives the number of reactions which occurred.

The original experimental arrangement with both low efficiency and low flux permitted the determination only of relative amounts of Li present in a series of Li^6F films. The data are compared with subsequent flame photometric analyses in Table I.

Table I

Sample No.	Relative Amount Li^6	
	By n irradiation	By flame analysis
1	0.28	0.36
2*	1.0	1.0
3	1.1	1.1
4	2.2	2.1
5	3.6	3.2
6	3.7	3.8
7	7.7	8.3

*The results of the relative analyses by both methods are normalized to sample No. 2.

A second series of Li^6F films was analyzed under more favorable conditions; the efficiency for detection was 8.5%, and the flux corresponded to 1.17×10^5 neutrons/cm²-sec at an energy of 0.0745 eV. The cross section for Li^6 at this energy has a value of 502 b, calculated from the reported cross section³ for natural lithium corrected for isotopic abundance. The weight of Li was calculated from Equation (2), and the results are compared

with subsequent flame analyses in Table II.

Table II		
Weight of Li^6 (ug)		
Sample No.	By n irradiation	By flame analysis
5	7.0	6.8
6	15	14
7	14	13
8	30	29
9	30	28
10	76	75

A series of $\text{B}^{10}_2\text{O}_3$ films were analyzed under the same conditions as the second set of Li^6F films. A value of 2245 b was calculated for the cross section from the reported value³ for natural B corrected for isotopic abundance. The weight of B was calculated from Equation (2), and the results are listed in Table III.

Table III

Sample No.	Weight of B (mg)
11	0.76
12	3.5
13	3.2
14	7.1
15	7.6
16	22

DISCUSSION

The results obtained indicate the feasibility of the neutron - charged particle reaction as an analytical tool. The agreement with flame analyses is within the precision of this method (5 to 10%). The errors associated with detection of prompt phenomena are due primarily to uncertainty in the incident flux ($\pm 5\%$) which arise from inaccuracies in the cross sections of the monitor reactions. Other errors arise from uncertainties in the cross sections of the reactions investigated and from counting statistics.

The latter were $< 1\%$, and the errors of the cross section values selected for Li^6 and B^{10} are estimated as $< 2\%$. Since the major errors introduced are constant, this technique can be the basis for an extremely precise empirical analysis. As an absolute method, it has an accuracy equal or superior to that of the more conventional methods for this concentration range. The major advantages of the method are (1) that it is nondestructive and (2) that it is considerably more sensitive than other methods.

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FIGURE 1

12

FIGURE 2

W

FIGURE 3

14

FIGURE 4

FIGURE 5

FIGURE 6

FIGURE 7