RADIATIVE-NEUTRON-CAPTURE REACTIONS IN ORTHOPHOSPHATES: THE CATION EFFECT

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INTRODUCTION

In continuing the general study of the radiative-neutron-capture effect in solid orthophosphates, a series of experiments was performed on the mono- and di-alkali metal orthophosphates and tri-lithium orthophosphate. The object of these experiments was to collect data on the several different oxidation states that are formed and to note trends and possible correlation with a physical property of the metal. The overall objective is a consistent theory and mechanism that will explain all radiative-neutron-capture reactions. In the experiments all salts were similarly treated, i. e., they were irradiated at the same facility, handled and treated in identical fashion. Separations were performed on a strong-base anion-exchanger using a chloride gradient.

EXPERIMENTAL

Syntheses:

Salts of the alkali metal orthophosphates, not commercially available, were prepared by neutralization of orthophosphoric acid with the appropriate base. A pH meter was used to indicate the equivalence point followed by evaporation of the solvent, filtration, and finally drying of the salt over P_4O_{10} .

Anion-Exchange Chromatography:

Gradient-elution anion-exchange chromatography was used in a manner as previously described (1). Identification of activities was based on the retention

(1) Anselmo, V. C., A. E. C. Report COO 1618-1.

volume of the pure compounds. The fraction as ^{32}P in each peak was found by cutting the peak from the recorder tracing and finding the weight relative to the weight of all the peaks.

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Neutron Irradiations:

Neutron irradiations were performed at the OWR reactor at Los Alamos Scientific Laboratory for 10 to 15 minutes at 2.5×10^{12} n cm² sec⁻¹. Immediately following the irradiation the samples were quenched in liquid nitrogen and stored in this manner until subject to isothermal annealing. The samples were annealed at $120 \pm 3^\circ$ C for predetermined times and then dissolved in the aqueous eluant and separated on the ion-exchange column. Samples, weighing about 0.1 g, were wrapped in aluminum foil for irradiation.

RESULTS AND DISCUSSION

The results are shown in Table I for the entire series of experiments. The percentages reported here are the "plateau values", i. e., the percentage reached by the activity after annealing for at least 3 hours at $120 \pm 3^\circ$ C. The annealing curves go from an initially high or low value to the plateau, or there is no change on annealing. Several conclusions may be drawn regarding trends in percentage of a particular oxidation state for the different salts. For example, for the mono-alkali metal orthophosphates, there is a reduction as hypophosphite and orthophosphite, with an increase in the fraction as orthophosphate, as the atomic weight of the cation increases. And also there is a slight increase in all the +5 states. This category includes not only orthophosphate, but also pyrophosphate and the activity that is eluted with 6M HCl that for the purposes of this report may be considered to be a +5 oxidation state. The hypophosphate percentage is irregular but appears to go to zero.

With respect to the di-alkali metal orthophosphates, the results are quite different. First of all, there appears to be no dependence on the cation. The fraction as each individual species is fairly constant, at least there is not the change shown for the mono-alkali metal salt. Also, there is a tremendous increase in the fraction as orthophosphite; it is much larger than in the case of the mono-alkali metal orthophosphate. In addition, the orthophosphate fraction is consistently less, but the summation as pyrophosphate and condensed phosphate is about the same in both the mono and di-alkali metal salt. The only tri-alkali

metal salt that was irradiated was tri-lithium orthophosphate, and its results are included in Table I for comparison purposes.

In conclusion, the results of these experiments indicate trends in the percents of the different oxidation states of phosphorus in alkali metal orthophosphates that are irradiated with thermal neutrons. Further analysis into the implications of these trends is continuing at the present time.

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TABLE I

PERCENT ^{32}P FOUND IN ALKALI-METAL ORTHOPHOSPHATES THAT WERE IRRADIATED WITH THERMAL NEUTRONS, ISOTHERMALLY ANNEALED AT $120 \pm 30^\circ \text{C}$ FOR AT LEAST THREE HOURS, AND DISSOLVED IN AQUEOUS SOLVENT

Salt	P ¹	P ³	P ⁴ -P ⁴	Summation of Cols 2, 3, and 4	P ⁵	P ⁵ -O-P ⁵	P _x	Summation of Cols 6, 7, and 8
LiH ₂ PO ₄	14	2	1	17	40	18	26	84
NaH ₂ PO ₄	13	7	0	20	58	0	22	80
KH ₂ PO ₄	20	1	0	21	69	0	11	80
RbH ₂ PO ₄	7	~4	3	~14	~72	19	7	~98
CsH ₂ PO ₄	<1	0	0	<1	77	0	22	99
Na ₂ HPO ₄	5	29	(b)	34	46	20		66
K ₂ HPO ₄	3	23	(b)	26	53	21		74
Rb ₂ HPO ₄	4	23	2	29	42	12	17	71
Li ₃ PO ₄	10	6	4	20	44	12	24	80

(a) Using the Blaser notation, where P¹ is hypophosphite, etc.

(b) Not actually separated but included with P⁵-O-P⁵ and P_x peak.

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