

COMMUNICATIONS TO THE EDITOR

THE TETRAVALENT AND PENTAVALENT STATES OF PROTACTINIUM

Sir:

It was presumed for some time that protactinium exists in the V state though it had been suggested that the III and IV states might be found.¹ Recently several workers have reported its existence in the IV state.^{2,3,4} The analytical data and other evidence for the existence of these states are not unequivocal.

We have recently prepared on the microgram scale two anhydrous compounds of protactinium (IV) which demonstrate the stability of tetravalent protactinium and, in addition, one compound which is definitely in the V state.

The dioxide, PaO₂, a black solid, was prepared during an attempt to reduce the white volatile chloride (PaCl₅?) with zinc at 600°. Evidently enough water was present to hydrolyze the product to the oxide. The tetrachloride, PaCl₄, a yellow-green solid, volatile *in vacuo* at 400°, was prepared by action of hydrogen on the "PaCl₅" at 800°.

The formulas of both compounds were established by analysis of the X-ray diffraction patterns. The tetrachloride has the tetragonal UCl₄-type structure.⁵ The unit cell dimensions for the set of isostructural compounds are

	<i>a</i> ₁ , Å.	<i>a</i> ₃ , Å.
ThCl ₄	8.490 ± 0.001	7.483 ± 0.001
PaCl ₄	8.377 ± .004	7.482 ± .004
UCl ₄	8.303 ± .001	7.483 ± .001
NpCl ₄	8.27 ± .01	7.48 ± .01

The dioxide has the fluorite structure and is thus isostructural with the dioxides of the other 5f-elements from thorium to americium. The unit cell constant is *a* = 5.05 ± 0.001 Å.

The cubic form of Pa₂O₅, with a unit cell constant of 5.416 ± 0.001 Å., was obtained as a white solid by heating the dioxide to 1100° in oxygen. The oxide obtained by heating the hydrated oxide precipitated from solution is also cubic with a unit cell constant of 5.46 ± 0.01 Å. Its approximate composition is PaO_{2.25} and *not* Pa₂O₅ as has been reported.¹ Other intermediate compositions were obtained by heating PaO_{2.25} at elevated temperatures in pure oxygen or hydrogen. The cubic oxide phase extends over the entire range from PaO₂ to Pa₂O₅, the unit cell constant decreasing with increasing oxygen content.

(1) A. von Grosse, *THIS JOURNAL*, **52**, 1744 (1930); *Proc. Roy. Soc. (London)*, **A150**, 369 (1935); *J. Russ. Phys.-Chem. Soc.*, **60**, 844 (1928).

(2) W. H. Zachariasen, *Acta Cryst.*, **2**, 388 (1949).

(3) G. Bouissieres and M. Haissinsky, *J. Chem. Soc.*, Supplementary Issue No. 2, S-256 (1949).

(4) R. Elson, ANL-4252, p. 9, Oct.-Dec. 1948; paper presented before Detroit Meeting of Am. Chem. Soc., April, 1950.

(5) R. C. L. Mooncy, *Acta Cryst.*, **2**, 189 (1949).

A second form of Pa₂O₅, orthorhombic and isostructural with U₂O₅, Ta₂O₅ and Nb₂O₅, was obtained as a white solid in the course of an attempt to prepare a fluoride by action of bromine pentafluoride on PaO_{2.25} at 400°. This oxide represented material volatilized out of the original sample, suggesting the existence of a volatile fluoride or oxyfluoride.

Attempts to prepare the tetravalent bromide and iodide of protactinium by methods analogous to those used for the preparations of the corresponding compounds of the other heavy elements have yielded compounds whose X-ray diffraction patterns have not yet been interpreted.

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SYNTHESIS OF PHENYLALANINE AND TYROSINE IN YEAST

Sir:

Little evidence exists to indicate the biological origin of the benzenoid amino acids phenylalanine and tyrosine. In the present experiments yeast (*S. cerevisiae*) was grown in a medium containing glucose and small amounts of acetate. The labeled substrate was 1-C¹³, 2-C¹⁴ acetate in one experiment and 1-C¹⁴ glucose¹ in another. The hydrolysates from the yeast proteins were separated on columns of Dowex 50.² It will be seen (Table I) that in a medium containing glucose

TABLE I

ISOTOPE CONCENTRATIONS IN YEAST CONSTITUENTS
Yeast grown in medium containing 0.555 mole *d*-glucose, 0.054 mole of acetic acid, 100 mg. *meso*-inositol.

	Labeled substrate		1-C ¹⁴ glucose 800 c.p.m. C ¹⁴ c.p.m. C ¹⁴
	C ¹⁴ H ₃ C ¹³ OOH c.p.m. C ¹⁴	at % excess C ¹³	
Acetate added	23,000	10.4	0
Acetate recovered	1,300	0.70	800
Protein	2,000	0.91	690
Glutamic a.	3,700	1.83	...
Aspartic a.	730	0.25	...
Tyrosine	20	0.03	530
Phenylalanine	20	0.02	540
Leucine	3,300	1.27	...
Lysine	6,200	2.97	...

^a All samples counted after conversion to BaCO₃ and correction to infinite thickness.

(1) D. E. Koshland and F. H. Westheimer, *THIS JOURNAL*, **72**, 3383 (1950).

(2) W. H. Stein and S. Moore, *Cold Spring Harbor Symp. Quant. Biol.*, **XIV**, 179 (1950).