

Table 2. *Bond lengths and angles*

	Old	New		Old	New
N(1)-C(2)	1.497 Å	1.491 Å	C(7)-O(8)	1.262 Å	1.260 Å
C(2)-C(3)	1.528	1.519	C(7)-O(9)	1.239	1.232
C(3)-O(4)	1.249	1.249	N(1)···O(8)	2.712	2.747
C(3)-N(5)	1.328	1.319	N(1)···O(9)	2.729	2.728
N(5)-C(6)	1.462	1.451	N(1)···O(8)	2.782	2.790
C(6)-C(7)	1.516	1.514	N(5)···O(4)	2.966	2.963
Standard deviation ± 0.007 Å					
N(1)-C(2)-C(3)	110.3°	109.5°	N(5)-C(6)-C(7)	112.7°	112.4°
C(2)-C(3)-O(4)	120.1	120.4	C(6)-C(7)-O(8)	117.6	117.9
C(2)-C(3)-N(5)	116.8	116.3	C(6)-C(7)-O(9)	115.6	115.5
O(4)-C(3)-N(5)	123.1	123.3	O(8)-C(7)-C(9)	126.7	126.5
C(3)-N(5)-C(6)	121.6	121.2			
Standard deviation ± 0.4 °					

earlier measurements agree with the new values within their own much larger standard deviations. The differences between the hydrogen and deuterium compounds, although perhaps significant, are now quite small and the earlier remarks (Biswas, Hughes, Sharma & Wilson, 1968 p.50) regarding these discrepancies should be largely disregarded.

Because the chief error is in the length of *a* and because the molecular plane is nearly parallel to (100), the errors have only a small effect on the intramolecular bond lengths and angles. The maximum error is 0.011 Å and the r.m.s. error is ± 0.007 Å. Table 2 shows the new values of the bond lengths. The maximum error in an H-bond length is 0.035 Å, for the N(1)···O(8) bond, which is nearly parallel to *a*. Other H-bond lengths (Table 2) and van der Waals contacts have in general much smaller errors, the r.m.s. value being well under 0.01 Å. With the exception of the comments on the perdeutero compound the other arguments given in

the original paper still stand. In particular, the old estimates of standard deviations apply to the newly calculated bond lengths and angles given in Table 2.

I am indebted to Dr Freeman for prepublication use of his lattice constants and for the newly calculated bond lengths, which are based on our parameters and the new lattice constants.

References

- BISWAS, A. B., HUGHES, E. W., SHARMA, B. D. & WILSON, J. N. (1968). *Acta Cryst.* **B24**, 40.
 FREEMAN, H. C., PAUL, G. L. & SABINE, T. M. (1967). Private communication.
 HUGHES, E. W. & MOORE, W. J. (1949). *J. Amer. Chem. Soc.* **71**, 2618.

Acta Cryst. (1968). **B24**, 1129

Crystal data for $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{AsO}$ and $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{AsO}]_2$. BY GIULIANO BANDOLI, RODOLFO GRAZIANI and BRUNO ZARLI, *Centro Chimica delle Radiazioni e dei Radioelementi and Centro Strutturistica Chimica C.N.R. Padova, via Loredan 4, Italy.*

(Received 26 March 1968)

Crystal data are reported for $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{AsO}$ and $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{AsO}]_2$, which are crystallographically isomorphous with their respective triphenylphosphine analogues.

Four uranyl acetate complexes with triphenylphosphine and triphenylarsine oxides were recently prepared (Volponi, Zarli & Panattoni, 1967). They are $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{PO}]_2$ and $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{PO}$, for which the crystal data were obtained from precession and Weissenberg photographs and have been reported elsewhere (Graziani, Zarli & Bandoli, 1967), and $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{AsO}]_2$ and $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{AsO}$.

From oscillation and precession photographs taken with Cu $K\alpha$ radiation, $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{AsO}]_2$ was shown to be isomorphous with $[\text{UO}_2(\text{OAc})_2 \cdot \text{Ph}_3\text{PO}]_2$, the structure of

which has been successfully determined (Panattoni, Bandoli, Graziani & Croatto, 1968). The presence of dimers was recognized for both compounds by molecular weight measurements.

X-ray powder diffraction photographs were taken for $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{AsO}$ and $\text{UO}_2(\text{OAc})_2 \cdot 2\text{Ph}_3\text{PO}$ with Cu $K\alpha$ radiation on a 114.6 mm camera using sodium fluoride as internal standard, and by comparison the compounds were found to be isomorphous. Densities were measured by the flotation method with the use of mixtures of *sym*-tetrabromoethane and *sym*-dichloroethane. The cell di-

Table 1. Agreement of calculated and observed interplanar spacings

UO ₂ (OAc) ₂ ·2Ph ₃ PO				UO ₂ (OAc) ₂ ·2Ph ₃ AsO	
<i>d</i> _{calc}	<i>d</i> _{obs}	<i>I</i> _{rel}	<i>hkl</i>	<i>d</i> _{obs}	<i>I</i> _{rel}
9.568	9.571	6	020	9.561	5
8.676	8.674	9	01 $\bar{1}$	8.672	8
7.980	7.971	10	11 $\bar{1}$	7.971	10
6.469	6.464	9	12 $\bar{1}$	6.464	8
			111	5.246	3
4.704	4.710	5	12 $\bar{2}$	4.709	4
4.390	3.387	5	{ 20 $\bar{2}$ 22 $\bar{1}$ }	4.387	5
4.390					
4.142	4.144	8	131		
4.014	4.004	3	220		
3.869	3.873	2	03 $\bar{2}$	3.873	2
3.570	3.562	7	112	3.566	6
3.412	3.409	6	04 $\bar{2}$	3.414	6
3.234	3.227	4	24 $\bar{2}$	3.236	2
2.892	2.890	7	{ 03 $\bar{3}$ 14 $\bar{2}$ 31 $\bar{3}$ 33 $\bar{2}$ 10 $\bar{4}$ }	2.898	5
2.894					
2.893					
2.891					
2.671					
2.674	2.675	3	{ 21 $\bar{4}$ 330 42 $\bar{4}$ }	2.683	2
2.676					
2.139					
2.138	2.140	4	{ 44 $\bar{3}$ 124 }	2.143	3
2.067					

Table 2. Crystal data

Compound	[UO ₂ (OAc) ₂ ·Ph ₃ AsO] ₂	UO ₂ (OAc) ₂ ·2Ph ₃ AsO
M.W.	1420.8	1032.6
M.P.(°C)	245	260
System	Triclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$ *	<i>P</i> 2 ₁ / <i>c</i>
<i>a</i> (Å)	8.31 ± 0.02	9.868 ± 0.013
<i>b</i> (Å)	11.05 ± 0.03	19.166 ± 0.016
<i>c</i> (Å)	13.66 ± 0.04	10.880 ± 0.011
α	101°50' ± 12'	
β	91°10' ± 10'	116°32' ± 5'
γ	109°12' ± 15'	
<i>V</i> (Å ³)	1150.5	1841.1
<i>D</i> _m (g.cm ⁻³)	2.03	1.85
<i>D</i> _x (g.cm ⁻³)	2.05	1.86
<i>Z</i>	1	2
<i>F</i> (000)	668	996

* Space group *P* $\bar{1}$ was chosen since the structure of the phosphine analogue was shown to be centrosymmetric.

mensions for UO₂(OAc)₂·2Ph₃AsO were calculated by the least-squares method.

The main reflexions in the powder diagram are listed in Table 1. Table 2 shows the crystal data. Since UO₂(OAc)₂·2Ph₃AsO crystallizes in *P*2₁/*c* with only two molecules per cell, the uranium atoms must be at centres of symmetry. The most probable resulting structure is consistent with eight-coordination to the heavy atom, realized by two chelate acetate groups and two ligand oxygen atoms in hexagonal transplanar arrangement normal to the linear uranyl group.

No further X-ray work on these compounds is contemplated at present.

References

- GRAZIANI, R., ZARLI, B. & BANDOLI, G. (1967). *Ric. Sci.* **37**, 984.
 PANATTONI, C., BANDOLI, G., GRAZIANI, R. & CROATTO, U. (1968). *Chem. Comm.* p.278.
 VOLPONI, L., ZARLI, B. & PANATTONI, C. (1967). Annual Meet. 163. Ist. Chim. Gen. Univ. Padova.