have equivalent space groups as a subgroup have recently been defined (Billiet, 1973). As with $P6_3/m$, it is necessary that a single same-class relationship exist between a space group and its equivalent subgroup.

Because of their unusual structural features, $La_7(OH)_{18}I_3$-type materials might be useful as host lattices for studies of crystal-field effects on the behavior of lanthanides and other doped ions. The wide range of halide accommodation results in a correspondingly wide range of distortion at certain cationic sites. Although the miscibility levels of the isostructural hydroxide chlorides, hydroxide bromides, and hydroxide iodides have not been investigated, they are expected to be substantial and ranges of continuous structural distortion might be easily achieved.

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Dichlorodioxobis(triphenylphosphine oxide)tungsten(VI), $WO_2Cl_2[OP(C_6H_5)_3]_2$

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$C_{36}H_{30}O_4P_2Cl_2W$, M_r = 843·1; monoclinic, $P2_1/c$, a = 18·89 (1), b = 9·99 (1), c = 19·23 Å, β = 112·20 (5)°; Z = 4, D_c = 1·666, D_m = 1·670 g cm^{-3}; U = 3359·9 Å^3; μ(Mo $K\alpha$) = 35·5 cm^{-1}. The molecule adopts a distorted octahedral coordination around the W atom, the two triphenylphosphine ligands, as well as the two double-bonded O atoms, being oriented *cis* to each other. The final R for 3007 observed reflexions was 0·046.

Introduction

Colourless single crystals of the title compound were prepared as described by Brisdon (1967), and recrystallized from nitromethane. Intensity data were collected on a crystal which was ground and solvent-etched to spherical shape with dimensions sufficiently small (radius 0·072 ± 0·07 mm) to render absorption errors negligible. Accurate cell dimensions were obtained by least-squares refinement from angular data on 25 high-order reflexions measured on a Philips PW 1100

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automated four-circle diffractometer. The intensities of 4411 unique reflexions in the θ range 3–22° were measured with Mo $K\alpha$ radiation, by use of the ω–2θ scan technique (scan width 1·0° θ, scan speed 0·04° θ s^{-1}), and background counts for half the scan time were made on each side of the intensity peak. The number of systematically absent reflexions was 291, a further 1113 with $|F_o| < 3{\cdot}00\sigma(F_o)$ were classified as unobserved; the remaining 3007 intensities were used in the solution and refinement of the structure. Although the compound appears to be quite stable towards the atmosphere, the intensities of the three monitor reflexions, which were measured periodically, decreased during data collection by about 13%, indicating some irradiative decomposition.

After application of Lorentz and polarization corrections, the structure was solved by conventional heavy-atom techniques with three-dimensional Patterson and difference electron density syntheses; several H atom peaks were recognizable in the final synthesis. Three models with increasing freedom of refinement were used: (i) with all atoms thermally isotropic and phenyl C atoms constrained to refine as rigid groups with idealized benzene geometry (C–C = 1·395 Å); (ii) all atoms isotropic, of which three sets of parameters (the non-carbon atoms and two groups of unconstrained C atoms) were grouped into blocked matrices (see below); (iii) all atoms anisotropic, of which four parameter sets were blocked similarly (the non-carbon atoms and three groups of C atoms); phenyl H atoms with common isotropic temperature factor were added artificially, each H riding on its parent C atom, and contributing only to the structure factors. Because of limitations in computer memory, no more than 112 parameters could be refined simultaneously by the full-matrix procedure so that for refinement of models (ii) and (iii) a blocked-matrix technique had to be used. The parameter blocks were constituted so as to ensure least-squares interaction between any two parameters at least once during a series of refinement cycles.

The function minimized during refinement was $\Sigma\, w\Delta^2$ [$\Delta = |F_o - kF_c|$, and $w \propto 1/(gF_o^2 + \sigma_{F_o}^2)$]. After three cycles of refinement with model (i), the value of the conventional R decreased to 0·098. A further three cycles with model (ii) (each parameter refined once), and eight cycles with model (iii) (each parameter refined twice), resulted in a final R value of 0·046.[*] $U_{\rm iso}$ for the H atoms was 0·077 Å². For the final refined value of the weighting parameter $g = 5{\cdot}6 \times 10^{-4}$, R_w $(= \Sigma\, w|\Delta|/\Sigma\, |F_o|)$ was 0·042. One-way analyses of $\langle w\Delta^2\rangle$, sampled over the range of values of $\sin\theta/\lambda$ and $[F_o/F_o(\max)]^{1/2}$, revealed no trends due to residual systematic error. Although R had not converged completely (the penultimate value was 0·048), further refinement was not considered to be worth the effort in view of the overall precision in positional parameters (~0·02 Å) attained for the lighter atoms; this was considered to be satisfactory, even if allowance is made for the fact that the standard deviations computed for blocked-matrix refinements are somewhat underestimated (Cruickshank, 1965). In addition, the distri-

Table 1. *Fractional coordinates* (×10⁴) *of non-hydrogen atoms*

	x	y	z
W	2585 (0·3)	2668 (0·4)	5732 (0·2)
Cl(1)	1228 (2)	2447 (3)	5270 (2)
Cl(2)	3909 (2)	2216 (4)	6131 (2)
P(1)	2215 (1)	326 (3)	4202 (1)
P(2)	2803 (2)	−368 (3)	6836 (2)
O(1)	2597 (4)	3824 (7)	5083 (4)
O(2)	2635 (6)	3647 (9)	6474 (5)
O(3)	2489 (4)	1070 (7)	4933 (4)
O(4)	2591 (4)	884 (7)	6372 (4)
C(11)	3004 (6)	−33 (9)	3937 (6)
C(12)	3679 (6)	−506 (11)	4474 (7)
C(13)	4292 (6)	−820 (12)	4286 (7)
C(14)	4261 (7)	−621 (11)	3558 (8)
C(15)	3596 (6)	−167 (11)	3006 (7)
C(16)	2971 (6)	144 (10)	3196 (6)
C(21)	1527 (5)	1228 (10)	3458 (5)
C(22)	1609 (6)	2608 (11)	3389 (5)
C(23)	1113 (7)	3302 (11)	2798 (6)
C(24)	538 (6)	2655 (12)	2248 (6)
C(25)	408 (7)	1287 (12)	2304 (7)
C(26)	916 (8)	600 (11)	2899 (6)
C(31)	1804 (6)	−1270 (9)	4284 (6)
C(32)	2026 (6)	−2437 (10)	4029 (6)
C(33)	1705 (8)	−3615 (12)	4145 (8)
C(34)	1207 (8)	−3688 (12)	4492 (7)
C(35)	964 (7)	−2495 (13)	4716 (6)
C(36)	1273 (6)	−1294 (11)	4620 (6)
C(41)	1945 (5)	−1274 (10)	6699 (5)
C(42)	1929 (5)	−2652 (12)	6817 (6)
C(43)	1245 (8)	−3297 (14)	6689 (7)
C(44)	596 (8)	−2605 (17)	6452 (7)
C(45)	573 (7)	−1258 (15)	6330 (7)
C(46)	1262 (6)	−577 (13)	6436 (6)
C(51)	3432 (6)	−1492 (9)	6629 (5)
C(52)	3142 (6)	−2463 (11)	6066 (5)
C(53)	3629 (7)	−3361 (11)	5933 (6)
C(54)	4400 (7)	−3275 (12)	6322 (7)
C(55)	4702 (6)	−2309 (13)	6857 (7)
C(56)	4216 (6)	−1397 (12)	7013 (6)
C(61)	3247 (6)	40 (10)	7822 (5)
C(62)	3490 (7)	−957 (11)	8346 (6)
C(63)	3821 (7)	−619 (12)	9097 (7)
C(64)	3924 (6)	661 (12)	9327 (6)
C(65)	3702 (6)	1631 (11)	8810 (6)
C(66)	3354 (6)	1372 (10)	8046 (6)

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[*] Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32987 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

bution of shift/e.s.d. magnitudes for the 405 non-hydrogen positional and thermal parameters was acceptable. The cumulative percentages for intervals in the range of these magnitudes were, finally:

0——0·2——0·4——0·6——1·0——2·5
47·2 68·1 80·7 94·1 99·8.

In the above analysis, one shift/e.s.d. ratio of 4·26, occurring for U_{11} of atom C(42), was omitted. (It should be noted that these quantities are, of course, not randomly distributed; the mean value of the magnitude of shift/e.s.d. decreases after each blocked cycle since the fraction of parameters in the non-refined set increases after each of these cycles.) Except for the absorption corrections, all the computing was done with the program system *SHELX* (Sheldrick, 1977).

Discussion

IR spectral data on the title compound (Lewis & Whyman, 1965; Brisdon, 1967; Majumdar & Bhattacharyya, 1967) have established the presence of two absorption bands associated with the W=O stretching vibration in the region 900–960 cm^{-1}, these being separated by 45–55 cm^{-1}. Lewis & Whyman (1965)

Table 2. *Fractional atomic coordinates* ($\times 10^3$) *of the hydrogen atoms*

	x	*y*	*z*
H(12)	372	−63	505
H(13)	481	−122	471
H(14)	476	−84	343
H(15)	356	−4	244
H(16)	245	52	277
H(22)	208	313	381
H(23)	117	437	277
H(24)	18	321	176
H(25)	−8	80	190
H(26)	85	−47	293
H(32)	243	−242	375
H(33)	187	−454	395
H(34)	100	−465	460
H(35)	54	−252	496
H(36)	110	−37	481
H(42)	246	−322	702
H(43)	124	−436	678
H(44)	7	−314	634
H(45)	4	−72	616
H(46)	125	48	632
H(52)	254	−251	574
H(53)	340	−414	552
H(54)	478	−398	621
H(55)	531	−225	716
H(56)	445	−63	743
H(62)	343	−199	817
H(63)	400	−141	951
H(64)	418	91	992
H(65)	380	266	899
H(66)	318	217	764

Table 3. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

W–Cl(1)	2·385 (3)	C(26)–C(21)	1·39 (1)
W–Cl(2)	2·366 (3)	C(31)–C(32)	1·39 (2)
W–O(1)	1·706 (8)	C(32)–C(33)	1·38 (2)
W–O(2)	1·702 (9)	C(33)–C(34)	1·35 (3)
W–O(3)	2·175 (8)	C(34)–C(35)	1·40 (2)
W–O(4)	2·163 (7)	C(35)–C(36)	1·38 (2)
O(3)–P(1)	1·499 (8)	C(36)–C(31)	1·38 (2)
O(4)–P(2)	1·500 (7)	C(41)–C(42)	1·40 (2)
P(1)–C(11)	1·78 (1)	C(42)–C(43)	1·38 (2)
P(1)–C(21)	1·77 (1)	C(43)–C(44)	1·33 (2)
P(1)–C(31)	1·81 (1)	C(44)–C(45)	1·36 (2)
P(2)–C(41)	1·79 (1)	C(45)–C(46)	1·41 (2)
P(2)–C(51)	1·79 (1)	C(46)–C(41)	1·38 (2)
P(2)–C(61)	1·81 (1)	C(51)–C(52)	1·40 (1)
C(11)–C(12)	1·39 (1)	C(52)–C(53)	1·38 (2)
C(12)–C(13)	1·37 (2)	C(53)–C(54)	1·37 (2)
C(13)–C(14)	1·39 (2)	C(54)–C(55)	1·37 (2)
C(14)–C(15)	1·38 (2)	C(55)–C(56)	1·41 (2)
C(15)–C(16)	1·40 (2)	C(56)–C(51)	1·39 (1)
C(16)–C(11)	1·42 (2)	C(61)–C(62)	1·37 (2)
C(21)–C(22)	1·40 (1)	C(62)–C(63)	1·38 (2)
C(22)–C(23)	1·36 (1)	C(63)–C(64)	1·34 (2)
C(23)–C(24)	1·36 (1)	C(64)–C(65)	1·34 (2)
C(24)–C(25)	1·40 (2)	C(65)–C(66)	1·39 (2)
C(25)–C(26)	1·37 (1)	C(66)–C(61)	1·39 (1)

Table 4. *Bond angles* (°) *with e.s.d.'s in parentheses*

O(1)–W–O(2)	102·2 (4)	C(21)–C(22)–C(23)	121 (1)
O(3)–W–O(4)	77·2 (3)	C(22)–C(23)–C(24)	120 (1)
O(1)–W–O(3)	90·1 (3)	C(23)–C(24)–C(25)	121 (1)
O(2)–W–O(4)	90·6 (4)	C(24)–C(25)–C(26)	118 (1)
O(1)–W–O(4)	167·1 (3)	C(25)–C(26)–C(21)	122 (1)
O(2)–W–O(3)	167·7 (4)	C(26)–C(21)–C(22)	117 (1)
O(1)–W–Cl(1)	95·7 (3)	P(1)–C(31)–C(32)	121 (1)
O(2)–W–Cl(1)	94·4 (4)	P(1)–C(31)–C(36)	118 (1)
O(3)–W–Cl(1)	83·0 (2)	C(31)–C(32)–C(33)	117 (1)
O(4)–W–Cl(1)	84·8 (2)	C(32)–C(33)–C(34)	124 (1)
O(1)–W–Cl(2)	93·6 (3)	C(33)–C(34)–C(35)	119 (1)
O(2)–W–Cl(2)	97·0 (4)	C(34)–C(35)–C(36)	120 (1)
O(3)–W–Cl(2)	83·4 (2)	C(35)–C(36)–C(31)	120 (1)
O(4)–W–Cl(2)	83·2 (2)	C(36)–C(31)–C(32)	121 (1)
Cl(1)–W–Cl(2)	163·5 (1)	P(2)–C(41)–C(42)	123 (1)
W–O(3)–P(1)	159·0 (4)	P(2)–C(41)–C(46)	118 (1)
W–O(4)–P(2)	165·4 (5)	C(41)–C(42)–C(43)	120 (1)
O(3)–P(1)–C(11)	109·8 (5)	C(42)–C(43)–C(44)	120 (1)
O(3)–P(1)–C(21)	113·1 (4)	C(43)–C(44)–C(45)	122 (1)
O(3)–P(1)–C(31)	111·3 (5)	C(44)–C(45)–C(46)	119 (1)
C(11)–P(1)–C(21)	108·0 (5)	C(45)–C(46)–C(41)	120 (1)
C(21)–P(1)–C(31)	108·1 (4)	C(46)–C(41)–C(42)	119 (1)
C(11)–P(1)–C(31)	106·3 (5)	P(2)–C(51)–C(52)	121 (1)
O(4)–P(2)–C(41)	108·1 (4)	P(2)–C(51)–C(56)	120 (1)
O(4)–P(2)–C(51)	116·1 (5)	C(51)–C(52)–C(53)	120 (1)
O(4)–P(2)–C(61)	110·4 (4)	C(52)–C(53)–C(54)	120 (1)
C(41)–P(2)–C(51)	107·0 (5)	C(53)–C(54)–C(55)	121 (1)
C(51)–P(2)–C(61)	107·1 (4)	C(54)–C(55)–C(56)	120 (1)
C(41)–P(2)–C(61)	107·7 (5)	C(55)–C(56)–C(51)	120 (1)
P(1)–C(11)–C(12)	119 (1)	C(56)–C(51)–C(52)	119 (1)
P(1)–C(11)–C(16)	123 (1)	P(2)–C(61)–C(62)	120 (1)
C(11)–C(12)–C(13)	121 (1)	P(2)–C(61)–C(66)	120 (1)
C(12)–C(13)–C(14)	121 (1)	C(61)–C(62)–C(63)	119 (1)
C(13)–C(14)–C(15)	120 (1)	C(62)–C(63)–C(64)	122 (1)
C(14)–C(15)–C(16)	119 (1)	C(63)–C(64)–C(65)	119 (1)
C(15)–C(16)–C(11)	121 (1)	C(64)–C(65)–C(66)	123 (1)
C(16)–C(11)–C(12)	118 (1)	C(65)–C(66)–C(61)	118 (1)
P(1)–C(21)–C(22)	120 (1)	C(66)–C(61)–C(62)	120 (1)
P(1)–C(21)–C(22)	122 (1)		

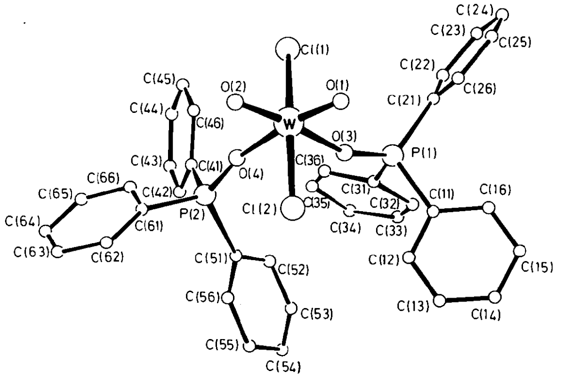

Fig. 1. Atomic numbering scheme and a view of the molecular structure perpendicular to the O(1)–O(2)–Cl(2) plane.

Table 5. *Torsion angles* (°)

O(3)–P(1)–C(11)–C(12)	44·1
O(3)–P(1)–C(21)–C(22)	40·0
O(3)–P(1)–C(31)–C(32)	−130·3
O(4)–P(2)–C(41)–C(42)	156·8
O(4)–P(2)–C(51)–C(52)	−86·9
O(4)–P(2)–C(61)–C(62)	180·0

Table 6. *Intramolecular and intermolecular contacts* (Å)

Symmetry code: no superscript: x,y,z; (i) $1-x, -y, 1-z$; (ii) $x, 1+y, z$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iv) $x, -\frac{1}{2}-y, -\frac{1}{2}+z$.

Cl(1)···H(36)	2·94	Cl(2)···H(14^{i})	2·72
Cl(1)···H(46)	2·81	O(1)···H(33^{ii})	2·66
O(1)···H(22)	2·36	O(1)···H(53^{ii})	2·50
O(2)···H(66)	2·55	O(2)···H(16^{iii})	2·79
O(3)···H(12)	2·81	O(2)···H(15^{iii})	2·44
O(3)···H(22)	2·86		
O(3)···H(36)	2·92		
O(4)···H(46)	2·53	C(26)···C(43^{iv})	3·48
O(4)···H(66)	2·59	C(13)···C(13^{i})	3·45

suggested that these O atoms are *cis* but came to no definite conclusion since, as they pointed out, the mutual coupling between the two bonds which is presumably responsible for the splitting of the IR band has no counterpart in the linear UO_2 group for which only a single absorption band has been observed. In addition, Brisdon refers to the difficulties in making spectral assignments for the heavy-metal d^1 oxo complexes. The present study was therefore undertaken to clear up the uncertainty in the structure of this compound.

Fractional atomic coordinates are given in Tables 1 and 2; bond lengths and angles in Tables 3 and 4, respectively. The molecular structure and atomic numbering scheme are depicted in Fig. 1. The equation of the least-squares plane through the four O atoms, based on a right-handed orthogonal system (in Å) with **Z**∥**c**, **X**∥**a***, is

$$0{\cdot}9978X - 0{\cdot}0450Y - 0{\cdot}0491Z = 3{\cdot}9438.$$

The r.m.s. deviation of these atoms from the plane is 0·028 Å, and the W atom is situated 0·002 Å from it. The coplanarity of the C atoms in each of the six phenyl rings is good, the largest deviation from a least-squares plane (0·026 Å) being of the order of the precision in the positions of these atoms (see above, *Introduction*). The triphenylphosphine (tppo) ligands are oriented *cis* in a configuration which is rather crowded as a result of closure of the angle between the ligand O atoms, O(3)–W–O(4), to 77·2°. In view of the planarity of the WO_4 moiety this is evidently the result of strong repulsions between the double-bonded O atoms, which open out the O(1)–W–O(2) angle to 102·2°, and the maintenance of 90° angles between single- and double-bonded O atoms. The repulsion of the Cl atoms by O(1) and O(2) is also evident in the deviations (+4–7°) of the four O=W–Cl angles from 90°. Similar deviations from octahedral symmetry around W have been observed in the dichlorodioxo(pentane-2,4-dionato)tungstate(VI) anion, $C_5H_7O_2WO_2Cl_2^-$ (Drew, Fowles, Rice & Shanton, 1974), oxotetrachloro(azoxybenzene)tungsten(VI), $(C_6H_5N)_2OWOCl_4$ (Bassi & Scordamaglia, 1975) and $WOCl_4$ (Iijima & Shibata, 1972).

Not only are the tppo ligands asymmetrically attached to the W atom, but considerable crowding of the phenyl substituents is evident from the 6·4° difference in the two P=O–W angles, from the scatter in torsion angles involving these bonds (Table 5), and the large variation (108–116°) in the six O=P–C angles. The constancy of the C–P–C angles shows that the conformational strain is largely accommodated by flexure of the $P(C_6H_5)_3$ moieties around the pivotal P atoms. It is interesting to note that this crowded arrangement is not paralleled in the structure of $UCl_4(tppo)_2$ (Bombieri, Brown & Graziani, 1975), where the two *cis* tppo ligands, although bonded at similar distances from the metal atom, are unstrained and related by twofold symmetry. Evidently the larger angular separation (90°) between the metal–ligand bonds in this structure is sufficient to allow the phenyl groups to be accommodated without strain. The authors ascribe the *cis* conformation in the uranium complex to the more stable packing that is presumably achieved by close, graphite-type contacts that result between phenyl groups of neighbouring molecules. Favourable as such contacts may be in respect of lattice energy, it is apparent from the present structure [in which only two short intermolecular C···C contacts (Table 6) occur] that *cis* tppo substitution is possible not only without graphite-type interactions, but even at the cost of appreciable intramolecular strain. De Wet & du Preez (1978) have furthermore shown that the disposition of ligands in this and other uranium complexes can be accounted for on more fundamental grounds, such as the ionic bonding character of U and ligand–ligand repulsion effects.

The bond lengths observed here between the W atom and its immediate neighbours are comparable with values found for other similar compounds. For example, for the structures quoted above W–Cl is 2·386 (4) Å (mean) in the dionato anion and 2·295 (7) Å in the azoxybenzene complex; W=O is 1·729 (10) (mean) and W–O 2·142 (9) Å in the former. On the other hand, shorter W=O bond lengths, 1·669 (18) for the dionato complex and 1·684 (15) Å in $WOCl_4$ (Iijima & Shibata, 1972), have been observed; the length of this bond is somewhat variable, and is probably sensitive to the nature of the other substituents in the coordination sphere, as is the case for oxomolybdenum complexes (Schröder, 1975).

The structure is stabilized by several short H···Cl and H···O contacts (Table 6), 14 of which surround the terminal O and Cl atoms in each molecule. Although some of the contact distances exceed the sum of the Pauling non-bonded radii, all involve H atoms in *ortho* phenyl positions, and therefore probably influence the particular ring orientations in the crystal. This would be particularly so for the short contacts listed in Table 6 (H···O all <2·6 Å and H···Cl <3 Å).

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