The Crystal and Molecular Structure of Iododicarbonylbis[bis(dimethylphosphino)-ethane]tungsten(II) Iodide

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Crystals of the title compound are monoclinic, space group $P2_1/m$ with $a = 13.853$ (8), $b = 10.752$ (11) $c = 16.584$ (11) Å, $\beta = 90.75$ (8)°, $Z = 4$. 2438 independent reflexions measured on a diffractometer have been refined to R 0.080. There are two independent cations in the asymmetric unit, each with crystallographically imposed C_s symmetry, which have slightly different geometries although in both the metal atom environment is best described as capped trigonal prismatic with the I atom in the capping position, four P atoms in the capped quadrilateral face and two carbonyls in the remaining edge. The geometry of one cation is considerably distorted from C_{2v} symmetry. All facets of the distortion can be related to the asymmetric puckered conformation of the five-membered chelate ring. The other cation has ideal C_{2v} geometry with the chelate rings in the envelope conformation, but such an unusual conformation for saturated rings is unlikely and it may be that the cation is disordered, but this could not be confirmed.

Introduction

The geometry of the capped trigonal prism (Fig. 1) is well established by X-ray structure determinations of such molecules as $[Mo(CNR)_7]^{2+}$ (Lewis & Lippard, 1975) and $[Mo(CNR)_6I]^+$ (Lewis & Lippard, 1972) with R = *tert*-butyl; and $|W(CO)_4(diars)I|^+$ (Drew & Wilkins, 1974) and $[Mo(CO)_2(diars)_2Cl]^+$ (Drew & Wilkins, 1973), diars = *o*-phenylenebis(dimethylarsine). Drew & Wilkins (1973, 1974) have shown that the diars ligand is particularly well suited to occupy the *b* edge of the capped quadrilateral face of a capped trigonal prism with the five-membered ring in the envelope conformation, the angle between MAs$_2$ and the As$_2$C$_6$ planes being *ca* 20°. This conformation minimizes repulsions between the methyl groups and atoms in the coordination sphere and incidentally maintains the C_{2v} geometry of the cations.

We were therefore interested to see what effect the replacement of the diars ligand by dmpe [bis(dimethylphosphino)ethane] would have on the geometry of the capped trigonal prism particularly as the envelope conformation is not likely to be

observed for such a saturated ligand. Therefore we report here the crystal and molecular structure of $[W(CO)_2(dmpe)_2I]^+ I^-$.

Experimental

The crystals were prepared according to the method of Connor, McEwan & Rix (1974).

Crystal data

$C_{14}H_{32}O_2P_4I_2W$, monoclinic, space group $P2_1/m$, $M_r = 793.9$, $a = 13.853$ (8), $b = 10.752$ (11), $c = 16.584$ (11) Å, $\beta = 90.75$ (8)°, $Z = 4$, $U = 2472.8$ Å^3, $D_m = 2.11$ (2), $D_c = 2.13$ g cm^{-3}, λ (Mo $K\alpha$) = 0.7107 Å, $\mu = 75.7$ cm^{-1}, $F(000) = 1488$. The space group $P2_1/m$ was indicated by the systematic absence 0k0, $k = 2n + 1$ and confirmed by the successful structure determination. The analogous Mo compound is isomorphous.

A crystal of approximate size $0.8 \times 0.15 \times 0.50$ mm was mounted with **a*** perpendicular to the instrument axis of a General Electric XRD-5 diffractometer which was used to measure diffraction intensities and cell dimensions, the latter *via* least-squares refinement of the 2θ values of high-angle reflexions. It was equipped with a manual goniostat, scintillation counter and pulse-height discriminator. Zr-filtered Mo X-radiation was used with a 4° take-off angle and a counting time of 10 s. Individual backgrounds were taken for those reflexions seriously affected by the streaking of other orders. For other reflexions, backgrounds were taken from plots as a function of 2θ. The
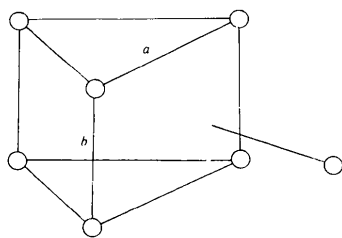

Fig. 1. The capped trigonal prism.

intensities of several standard reflexions monitored throughout data collection showed no significant changes. Of 3394 independent reflexions with $2\theta < 45°$ measured by the stationary-crystal stationary-counter method, 2438 with $I > 2\sigma(I)$ were used in subsequent

Table 1. *Positional parameters* ($\times 10^4$) *and isotropic thermal parameters* ($\times 10^3$) *with estimated standard deviations in parentheses*

	x	y	z	U
W(A)	2705 (1)	2500*	3956 (1)	†
I(A)	4645 (2)	2500*	4566 (2)	†
P(1A)	3333 (5)	608 (7)	3226 (4)	†
P(2A)	2476 (5)	680 (7)	4928 (4)	†
C(1A)	1270 (26)	2500*	4131 (20)	42 (8)
C(2A)	2146 (24)	2500*	2924 (20)	41 (8)
O(1A)	478 (18)	2500*	4206 (14)	52 (6)
O(2A)	1733 (21)	2500*	2274 (17)	66 (8)
C(3A)	2978 (27)	−863 (43)	4541 (22)	95 (11)
C(4A)	3829 (23)	−579 (38)	3890 (19)	79 (9)
C(5A)	2530 (21)	−245 (34)	2568 (16)	64 (8)
C(6A)	4375 (24)	902 (41)	2528 (20)	85 (10)
C(7A)	1317 (24)	109 (40)	5122 (20)	83 (10)
C(8A)	2910 (29)	814 (46)	6032 (24)	105 (13)
W(B)	−1767 (1)	2500*	1025 (1)	†
I(B)	−2887 (2)	2500*	2496 (1)	†
P(1B)	−2875 (5)	4346 (7)	697 (4)	†
P(2B)	−968 (5)	4352 (6)	1706 (4)	†
C(1B)	−496 (27)	2500*	594 (22)	51 (10)
C(2B)	−1837 (22)	2500*	−139 (18)	32 (7)
O(1B)	301 (24)	2500*	325 (19)	82 (9)
O(2B)	−1816 (19)	2500*	−820 (15)	56 (7)
C(3B)	−1880 (29)	5668 (45)	1849 (24)	102 (12)
C(4B)	−2678 (35)	5694 (57)	1354 (29)	133 (17)
C(5B)	−2786 (28)	5120 (45)	−312 (22)	98 (12)
C(6B)	−4181 (16)	4149 (29)	760 (13)	42 (6)
C(7B)	87 (22)	5053 (37)	1229 (18)	72 (8)
C(8B)	−449 (27)	4072 (35)	2762 (18)	69 (8)
I(1)	−257 (2)	2500*	6637 (2)	†
I(2)	5207 (2)	2500*	8461 (2)	†

* Parameter fixed.
† Anisotropic thermal parameters in Table 2.

Table 3. *Molecular dimensions: distances* (Å) *and angles* (°)

	Cation A	Cation B
W−I	2·859 (3)	2·907 (3)
W−P(1)	2·527 (7)	2·563 (7)
W−P(2)	2·559 (7)	2·537 (6)
W−C(1)	2·01 (3)	1·91 (4)
W−C(2)	1·87 (3)	1·93 (3)
I−W−P(1)	80·9 (2)	81·6 (2)
I−W−P(2)	84·3 (2)	82·0 (2)
I−W−C(1)	151·0 (9)	145·0 (9)
I−W−C(2)	134·4 (10)	144·9 (10)
P(1)−W−P(2)	74·5 (2)	75·1 (2)
P(1)−W−C(1)	114·6 (5)	118·2 (5)
P(1)−W−C(2)	72·7 (6)	76·4 (6)
P(1)−W−P(1^v)	107·3 (2)	101·5 (2)
P(1)−W−P(2^v)	164·6 (2)	163·6 (2)
P(2)−W−C(1)	77·2 (6)	76·5 (7)
P(2)−W−C(2)	121·6 (5)	117·4 (4)
P(2)−W−P(2^v)	99·8 (2)	103·4 (2)
C(1)−W−C(2)	74·6 (14)	70·1 (14)
P(1)−C(4)	1·81 (4)	1·83 (6)
P(1)−C(5)	1·80 (3)	1·87 (4)
P(1)−C(6)	1·89 (3)	1·82 (2)
W−P(1)−C(4)	114 (1)	114 (2)
W−P(1)−C(5)	119 (1)	119 (1)
W−P(1)−C(6)	115 (1)	119 (1)
C(4)−P(1)−C(5)	104 (2)	100 (2)
C(4)−P(1)−C(6)	102 (2)	101 (2)
C(5)−P(1)−C(6)	101 (1)	101 (2)
P(2)−C(3)	1·91 (4)	1·91 (4)
P(2)−C(7)	1·75 (4)	1·83 (3)
P(2)−C(8)	1·92 (4)	1·91 (3)
W−P(2)−C(3)	114 (1)	111 (1)
W−P(2)−C(7)	120 (1)	118 (1)
W−P(2)−C(8)	120 (1)	116 (1)
C(3)−P(2)−C(7)	96 (2)	106 (2)
C(3)−P(2)−C(8)	106 (2)	104 (2)
C(7)−P(2)−C(8)	97 (2)	100 (1)
C(3)−C(4)	1·64 (5)	1·37 (6)
P(2)−C(3)−C(4)	109 (3)	118 (4)
P(1)−C(4)−C(3)	105 (2)	117 (4)
C(1)−O(1)	1·11 (4)	1·20 (5)
C(2)−O(2)	1·21 (4)	1·13 (4)
W−C(1)−O(1)	178 (3)	180 (3)
W−C(2)−O(2)	176 (3)	176 (3)

Table 2. *Anisotropic thermal parameters* ($\times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(A)	35·4 (7)	23·6 (8)	29·9 (7)	0·0*	−4·6 (6)	0·0*
I(A)	52·7 (16)	56·3 (18)	90·3 (19)	0·0*	−21·7 (15)	0·0*
P(1A)	54·3 (39)	32·4 (38)	53·5 (37)	−1·1 (34)	7·6 (31)	−9·7 (33)
P(2A)	50·0 (37)	38·3 (40)	50·5 (35)	−1·0 (34)	2·2 (30)	14·9 (34)
W(B)	37·8 (7)	21·3 (7)	26·5 (6)	0·0*	4·1 (5)	0·0*
I(B)	66·6 (16)	47·2 (15)	45·7 (12)	0·0*	13·1 (11)	0·0*
P(1B)	58·0 (41)	36·3 (39)	55·0 (36)	17·1 (36)	−1·5 (31)	7·4 (35)
P(2B)	50·8 (35)	27·4 (33)	41·7 (31)	−1·5 (32)	−4·7 (27)	0·6 (30)
I(1)	91·8 (21)	36·1 (15)	72·9 (17)	0·0*	3·5 (15)	0·0*
I(2)	80·2 (20)	36·9 (15)	96·3 (21)	0·0*	14·7 (16)	0·0*

* Parameter fixed.

calculations. An absorption correction was applied with the X-RAY system (Stewart, Kundell & Baldwin, 1970).

Structure determination

The structure was solved from a Patterson function and successive Fourier maps. W, I and P atoms were refined anisotropically and C and O atoms isotropically by full-matrix least squares to R 0·080. The weighting scheme, chosen to give average values of $w\Delta^2$ for groups of reflexions independent of the value of F_o and $\sin \theta/\lambda$, was $\sqrt{w} = 1$ for $F_o \leq 80$ and $\sqrt{w} = 80/F_o$ for $F_o > 80$. Calculations were made on a CDC-7600 computer at the University of London Computer Centre with the X-RAY system (Stewart *et al.*, 1970). Atomic scattering factors for W, I, P, C and O were taken from *International Tables for X-ray Crystallography* (1974) as were the corrections for the real and imaginary parts of the anomalous dispersion for W, P and I. The 956 unobserved reflexions showed no serious discrepancies. In the final cycle of the refinement, no shift exceeded 0·10σ. Final positional coordinates and thermal parameters are given in Tables 1 and 2. Bond lengths and angles in the cations are given in Table 3.*

Discussion

There are two independent $[W(CO)_2(dmpe)_2I]^+$ cations in the asymmetric unit, each with crystallographically imposed C_s symmetry. The W and I atoms and carbonyl groups in both cations lie on the mirror planes as indeed do the two I$^-$ anions. The geometries of both independent cations are based on the capped trigonal prism but are not identical. The two cations are called

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32150 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.
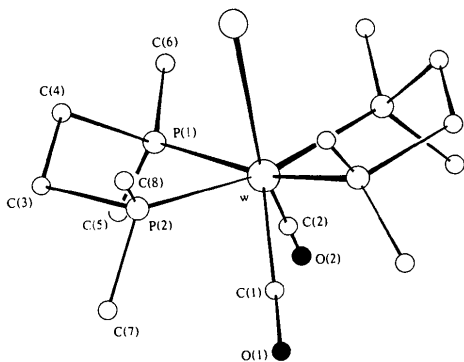

Fig. 2. Cation *A*.

A and B and are numbered in an identical fashion. The numbering scheme for the atoms is illustrated in Fig. 2 which shows cation A.

In the capped trigonal prism, I occupies the capping position, four P atoms the capped quadrilateral face and two carbonyl groups the remaining edge. The geometry is thus equivalent to that found for $[Mo(CO)_2(diars)_2Cl]^+$ (Drew & Wilkins, 1973) in most particulars, with the dmpe ligands occupying the same b edges as diars. The geometry of $[Mo(CO)_2(diars)_2Cl]^+$ exhibited C_{2v} (*mm*) symmetry but this was only possible because the five-membered ring had the envelope conformation. However, one would not expect such a conformation for the saturated rings in dmpe.

Indeed in cation A the ring has an asymmetric puckered conformation with C(1) and C(2) on the same side of the Mo, P(1), P(2) plane at distances of 0·12 and 0·83 Å. Such a geometry has been shown to be one of the most stable for a five-membered ring (Gollogly & Hawkins, 1971). This conformation much affects the geometry of the coordination sphere. The two torsion angles C(1)—W—P(2)—C(7) and C(2)—W—P(1)—C(5) are not identical as they would be in a C_{2v} cation for example {25·0, −27·5° in $[Mo(diars)_2(CO)_2Cl]^+$} but are 12·0 and −38·1° respectively (Table 4). Thus, because of the ring conformations, C(1) is potentially in a more crowded position than C(2). To compensate, the C(1)—W—P(2) angle becomes significantly greater than the C(2)—W—P(1) angle: 77·2 (6), 72·7 (6)°. Also the I—W—P(1)—C(4) torsion angle at 56·5° is larger than

Table 4. *Torsion angles* (°)

(*a*) Of the form L—W—P—C where the L—W—P angle is <95°

	Cation A	Cation B
I—W—P(1)—C(4)	56·5	66·4
I—W—P(1)—C(5)	179·5	−176·5
I—W—P(1)—C(6)	−60·6	−53·2
P(2)—W—P(1)—C(4)	−30·0	−17·5
P(2)—W—P(1)—C(5)	92·9	99·6
P(2)—W—P(1)—C(6)	−147·2	−137·1
C(2)—W—P(1)—C(4)	−161·0	−141·2
C(2)—W—P(1)—C(5)	−38·1	−24·1
C(2)—W—P(1)—C(6)	81·8	99·2
I—W—P(2)—C(3)	−78·2	−63·4
I—W—P(2)—C(7)	169·5	173·7
I—W—P(2)—C(8)	48·7	55·0
P(1)—W—P(2)—C(3)	3·9	20·0
P(1)—W—P(2)—C(7)	−108·4	−103·0
P(1)—W—P(2)—C(8)	130·8	138·4
C(1)—W—P(2)—C(3)	124·3	144·5
C(1)—W—P(2)—C(7)	12·0	21·5
C(1)—W—P(2)—C(8)	−108·8	−97·1

(*b*) Others in the five-membered ring

W—P(1)—C(4)—C(3)	53·4	10·4
P(1)—C(4)—C(3)—P(2)	−46·5	8·2
C(4)—C(3)—P(2)—W	24·5	−23·1

the I—W—P(2)—C(8) angle of 48·7° and this may be why the I—W—P(1) angle is 80·9 (2)° compared to I—W—P(2) at 84·3 (2)°, and the W—P(1) bond is shorter [2·527 (7) Å] than the W—P(2) bond [2·559 (7) Å]. A consequence of all these changes is that the I—W—C(1) and I—W—C(2) angles are very different at 151·0 (9), 134·4 (10)°. The W—I bond length [2·859 (3) Å] is equivalent to that found in [W(CO)$_4$(diars)I]$^+$ [2·842 (3) Å]. The geometry of the coordination sphere of cation A is thus severely distorted from the capped trigonal prism and becomes intermediate between this ideal and the capped octahedron (see below).

The geometry of cation B is, however, significantly different from that of cation A as it has C_{2v} symmetry, within experimental error, with the five-membered ring in the envelope conformation and with the two C atoms 0·61 and 0·50 Å from the W, P(1), P(2) plane. The angle between the W, P, P plane and the P, C, C, P plane in the ring is 19·8 compared with 21·2° in [Mo(CO)$_2$(diars)$_2$Cl]$^+$ and 15·6° in [W(CO)$_4$(diars)I]$^+$. This conformation is so surprising that we considered whether the dmpe ligand was disordered in cation B. Other workers have shown that this ligand is prone to disorder in metal complexes, one example being TaH(CO)$_2$(dmpe)$_2$ (Meakin, Guggenberger, Tebbe & Jesson, 1974). A possible type of disorder would be that in which cation B is made up of two superimposed mirror-image cations each with the geometry of cation A. The consequence of such disorder would be that in B all atomic positions bar W and I would be the mean of two positions. These two positions would be very close together except for C(3), C(4) where differences of ca 0·5 Å would occur. After considerable investigation including refinement in $P2_1$, much study of difference Fourier maps, use of anisotropic thermal parameters, refinement of half-atoms in space groups $P2_1$ and $P2_1/m$ we are unable to state whether disorder occurs. This failure contrasts with the successful location of disorder in other structures and we conclude that cation B might well have an ordered C_{2v} geometry. The bond lengths are equivalent to those found in cation A with the exception of W—I which is a good 0·05 Å longer. We can see no reason for this but comment that the Mo—Cl bond length in [Mo(CO)$_2$(diars)$_2$Cl]$^+$ is also longer than expected and both may be due to halogen—methyl contacts. These will, of course, be less in cation A and in the [W(diars)(CO)$_4$I]$^+$ structure.

We have discussed elsewhere (Drew, 1977) that the differences in geometry between the capped trigonal prism (CTP) and the capped octahedron (CO) are very small and that chelate rings can often distort one geometry towards the other. This is particularly true for a large number of molecules with stoichiometry ML$_5$(L—L), L being a monodentate and L—L a bidentate ligand, whose geometries are intermediate between the CTP and the CO.

Table 5. *Intermolecular distances* < 3·75 Å *not involving hydrogen*

C(8B)···C(1A)	3·68	C(8B)···O(2A)	3·56
C(8B)···O(1A)	3·19	C(7A)···C(7A^{ii})	3·67
O(1B)···C(7B^i)	3·72	C(6A)···C(6B^{iii})	3·57
O(2B)···C(7B^i)	3·63	C(3A)···C(3A^v)	3·53

Roman numeral superscripts refer to the following equivalent positions relative to the set at x,y,z (Table 1):

(i) $-x, 1-y, -z$ (ii) $-x, -y, 1-z$ (iii) $1+x, \frac{1}{2}-y, z$
(iv) $x, -\frac{1}{2}-y, z$ (v) $x, \frac{1}{2}-y, z$

Cation A is an example of a molecule of type ML$_3$(L—L)$_2$ with this intermediate geometry which is brought about by the asymmetric puckered conformations of the bidentate dmpe ligands. As a capped octahedron, cation A has C(2) in the capping position, C(1), P(1), P(1^v) in the capped face and I, P(2), P(2^v) in the uncapped face. As an indication of the closeness to the CO we note that the angles subtended at the metal by C(2) and atoms in the capped face are 74·6, 72·7, 72·7° and by C(2) and atoms in the uncapped face 134·4, 121·6, 121·6°. The other L—M—L angles in cation A also indicate this intermediate geometry.

However, the distinction between one geometry or another can best be checked *via* δ' angles (Muetterties & Guggenberger, 1974) or by r.m.s. deviations (Drew & Rix, 1975). For both these calculations bond lengths have been normalized to 1·0 Å. There are three unique δ' angles required to evaluate a molecule as a CO or a CTP. In A these come to 24·8, 6·9, 6·9° while in B they are 38·2, 0·8, 0·8°. (There are no ideal angles to compare these with but in an ideal CO the three angles are equivalent and in an ideal CTP two angles are 0°.) R.m.s. deviations from C_{3v} and C_{2v} symmetry are, for A, 0·077, 0·063 Å and, for B 0·121, 0·022 Å respectively. This intermediate geometry of cation A may be indicative of a reaction path CTP $\rightleftharpoons$ CO in which C_s symmetry is maintained.

There are no intermolecular contacts significantly less than the sum of van der Waals radii. Those distances less than 3·75 Å are given in Table 5.

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Structure Cristalline d'un Oxychlorure de Manganèse, Mn₈O₁₀Cl₃

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The crystal structure of a new compound, $Mn_8O_{10}Cl_3$, has been determined from three-dimensional intensity data. Single-crystals were 'twinned out' by cooling from 400 °C under axial pressure. Independent data were collected on a Hilger & Watts automatic diffractometer. The structure was solved by Patterson and Fourier syntheses, and was then refined by least-squares calculations. The final discrepancy index for 461 X-ray independent reflexions was 10·5%. Mn has two valences. One of the eight Mn of the formula is Mn^{2+}, probably located on two sites, one on a Cl octahedron, the other one on a cube of eight O atoms. Mn^{3+} is located on three sites; two are octahedral with four O and two Cl, the third has an O octahedron coordination. Distances $Mn^{3+}-Cl^-$ are very long (2·84 Å) and it is the first time that data are published on the occurrence of these two ions in one compound. [Space group $I4/mmm$, $a = b = 9·2898$ (6), $c = 13·0247$ (9) Å, $Z = 4$.]

Trois oxychlorures de manganèse sont actuellement connus, $MnOCl_3$, MnO_2Cl_2, MnO_3Cl (Briggs, 1968), dans lesquels l'ion manganèse prend respectivement les valences 5+, 6+ et 7+. La structure cristalline de ces composés n'est pas connue. Bien que nous connaissions FeOCl (Lind, 1970), CrOCl (Christensen, Johansson & Quezel, 1975) dont les propriétés physiques font l'objet de nombreuses études, nous n'avons pas connaissance d'un oxychlorure de manganèse où Mn a une valence identique.

Cette publication est relative à la détermination de la structure cristalline d'un nouvel oxychlorure de manganèse, dans lequel Mn a les valences 3+ et 2+, et de formule $Mn^{2+}Mn_7^{3+}O_{10}Cl_3$.

Préparation des cristaux

Dans un four tubulaire chauffé à une température comprise entre 650 et 680°C, on introduit un creuset de platine rempli de $MnCl_2$ anhydre ou hydraté, sur lequel on fait passer un mélange gazeux, comprenant 80% d'azote et 20% d'oxygène. Au début de la réaction, le débit gazeux doit être suffisamment important pour entraîner les vapeurs de chlore qui se dégagent. A cette température, $MnCl_2$ qui est fondu, sert de solvant, et de petits cristaux de $Mn_8O_{10}Cl_3$ croissent à la surface du bain. On extrait ces cristaux par simple lavage à l'eau; ils se présentent sous forme de pyramide à base carrée; les plus gros mesurent 0,1 mm de côté.

Données cristallographiques

A la température ordinaire, la maille est quadratique. Le Tableau 1 résume les différentes données cristallographiques. L'absence systématique des raies hkl avec $h + k + l \neq 2n$, $hk0$ avec $h + k \neq 2n$ et $00l$ où $l \neq 2n$, sélectionne six groupes spatiaux possibles: deux

Tableau 1. *Données cristallographiques*

$a = b = 9,28984$ Å	$\sigma = 0,00059$ Å
$c = 13,02474$	$\sigma = 0,00094$
$Z = 4$	$D_c = 4,18$ g cm^{-3}

Groupes spatiaux possible: $I4/mmm$
ou $I4$, $I4/m$, $I422$, $I4mm$, $I\bar{4}m2$