Acta Cryst. (1974). B**30**, 300

The Crystal Structure of Dioxobis-(1,3-diphenylpropanedionato)molybdenum(VI), $(C_{15}H_{11}O_2)_2MoO_2$

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(*Received* 24 *September* 1973; *accepted* 26 *September* 1973)

The crystal structure of dioxobis-(1,3-diphenylpropanedionato)molybdenum(VI) has been determined from three-dimensional X-ray film data. The crystals are monoclinic, space group $P2_1/c$, with $a=9{\cdot}37$, $b=13{\cdot}52$, $c=20{\cdot}43$ Å, $\beta=94{\cdot}5°$, $Z=4$. Full-matrix least-squares refinement resulted in $R=0{\cdot}073$. The octahedrally coordinated molybdenum atom has two oxo oxygen atoms in the *cis* configuration with Mo–O distances of 1·695 and 1·697 Å. The Mo–O distances in the chelate rings are 2·176 and 2·157 Å for the oxygen atoms *trans* to the oxo oxygen atoms, and 2·006 and 1·979 Å for the other two oxygen atoms.

Introduction

The crystal structures of many metal complexes with β-diketones have been solved, and the stereochemistry of the chelate rings has been reviewed and discussed (Lingafelter & Braun, 1966; Cotton & Eiss, 1968). The influence of the ligand geometry on the coordination polyhedron of the metal atom cannot be predicted in general. Several years ago, when the present crystal structure analysis was undertaken, the configuration of the oxo oxygen atoms in the dioxomolybdenum(VI) complexes (molybdenyl complexes) was unknown. In the meantime other investigators (Atovmyan & Sokolova, 1971) found that in all these complexes the oxo oxygen atoms are *cis* to each other. The only exception is the octahedral dioxotetracyanomolybdate(VI) ion in $NaK_3[MoO_2(CN)_4].6H_2O$ with a *trans* configuration (Larson & Moore, 1966). The *cis* configuration was also found quite recently in the structure of dioxobis(acetylacetonato)molybdenum(VI) (Kamenar & Penavić, 1973).

Experimental

Dioxobis(1,3-diphenylpropanedionato)molybdenum(VI), ('molybdenylbisdibenzoylmethanate'), $(C_{15}H_{11}O_2)_2MoO_2$, first obtained by Morgan & Castell (1928), was prepared by the method described by Korpar (1961). The crystals are monoclinic with:

$$a=9{\cdot}37\,(2),\ b=13{\cdot}52\,(3),\ c=20{\cdot}43\,(5)\ \text{Å},\ \beta=94{\cdot}5\,(2)°,\ V=2539{\cdot}16\ \text{Å}^3,\ D_m=1{\cdot}48\ \text{g cm}^{-3},\ D_c=1{\cdot}51\ \text{g cm}^{-3},\ Z=4,\ \mu=48{\cdot}69\ \text{cm}^{-1}\ (\text{Cu }K\alpha\text{ radiation},\ \lambda=1{\cdot}5418\ \text{Å}).$$

The space group $P2_1/c$ was determined unambiguously from systematic absences. The density was determined pycnometrically at 25°C with decalin as the liquid.

The isomorphism between dioxobis-(1,3-diphenylpropanedionato)molybdenum(VI) and -tungsten(VI) was established.*

Dioxobis-(1,3-diphenylpropanedionato)tungsten(VI) crystals, $(C_{15}H_{11}O_2)_2WO_2$, have:

$$a=9{\cdot}33\,(1),\ b=13{\cdot}50\,(2),\ c=20{\cdot}41\,(5)\ \text{Å},\ \beta=94{\cdot}8\,(3)°.$$

Equi-inclination Weissenberg photographs were taken with nickel-filtered Cu $K\alpha$-radiation and the multiple-film technique. From 4418 independent reflexions recorded, 2561 had observable intensities which were measured with a microdensitometer. Two cylindrical crystals were used: with the first (0·0168 cm in diameter) six layers about [100] and with the second (0·0452 cm in diameter) seven layers about [010] were recorded. The intensities were corrected for absorption (Bond, 1967). The usual Lorentz and polarization corrections were applied.

Scale factors were determined for the reflexions of each layer line separately and further improved in the course of the refinement.

Determination and refinement of the structure

The coordinates of the molybdenum atom were determined from a three-dimensional Patterson synthesis.

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* Crystals of the molybdenum compound were kindly supplied by Dr B. Korpar-Čolig, Faculty of Science, the University of Zagreb and of the tungsten compound by A. Nikolovski, Faculty of Science, the University of Skopje.

Table 1. *Final coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^2$)

The thermal parameters refer to the expression:

$$\exp\{-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*)\}.$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo	508 (0)	1181 (0)	1657 (0)	5·21 (4)	4·59 (5)	4·20 (4)	−0·10 (5)	1·80 (3)	−0·27 (4)
O(1)	47 (8)	−339 (6)	1361 (4)	6·79 (49)	5·62 (50)	7·10 (50)	−1·38 (45)	3·97 (41)	−1·76 (41)
O(2)	1945 (8)	1039 (6)	1003 (3)	6·35 (43)	5·58 (51)	5·99 (43)	−0·83 (44)	2·43 (35)	−0·64 (38)
O(3)	−729 (7)	828 (6)	2381 (3)	5·79 (42)	5·77 (48)	4·58 (38)	1·15 (43)	1·49 (32)	−0·01 (34)
O(4)	1984 (7)	288 (6)	2267 (4)	4·09 (39)	5·53 (48)	7·05 (50)	−1·12 (41)	0·65 (34)	0·33 (40)
O(5)	−894 (8)	1558 (7)	1144 (3)	6·52 (46)	7·65 (59)	5·19 (41)	1·72 (49)	0·84 (35)	0·40 (40)
O(6)	1255 (9)	2241 (6)	1970 (3)	8·42 (55)	5·20 (49)	6·18 (47)	−0·99 (48)	3·37 (42)	−1·06 (39)
C(1)	498 (9)	−857 (8)	903 (4)	2·43 (40)	5·13 (64)	4·63 (52)	−0·06 (53)	−1·30 (37)	0·47 (47)
C(2)	1453 (12)	−496 (8)	464 (5)	5·48 (66)	4·97 (72)	5·31 (61)	−0·16 (64)	2·21 (52)	−0·12 (52)
C(3)	2112 (9)	409 (8)	529 (4)	3·30 (49)	5·17 (65)	2·97 (42)	0·87 (54)	0·74 (37)	−0·24 (42)
C(4)	−696 (11)	158 (8)	2828 (4)	3·99 (56)	4·39 (61)	4·27 (53)	−0·37 (60)	0·13 (42)	0·09 (46)
C(5)	401 (11)	−499 (9)	2941 (5)	3·80 (54)	5·71 (78)	6·56 (66)	−1·25 (64)	2·78 (49)	0·05 (58)
C(6)	1723 (11)	−429 (8)	2664 (5)	4·72 (56)	4·93 (64)	4·60 (54)	−1·07 (59)	0·03 (44)	−1·00 (49)
C(7)	3070 (10)	783 (8)	49 (4)	3·24 (48)	5·09 (61)	4·23 (50)	0·46 (57)	−0·15 (39)	0·96 (46)
C(8)	3770 (11)	1678 (9)	162 (4)	4·81 (60)	6·87 (81)	4·03 (53)	−0·66 (68)	0·87 (44)	0·12 (52)
C(9)	4743 (15)	2019 (10)	−265 (6)	7·81 (85)	6·55 (84)	7·56 (82)	−1·15 (78)	3·44 (69)	0·52 (68)
C(10)	4980 (13)	1469 (12)	−818 (6)	5·97 (69)	9·90 (1·13)	5·72 (67)	−0·17 (88)	2·83 (56)	1·78 (70)
C(11)	4249 (13)	596 (12)	−946 (5)	5·58 (70)	9·38 (1·09)	4·82 (61)	0·48 (82)	2·04 (53)	1·03 (65)
C(12)	3307 (10)	235 (9)	−516 (5)	4·48 (56)	6·63 (77)	4·34 (52)	0·21 (63)	1·15 (43)	−0·39 (51)
C(13)	−1968 (11)	167 (8)	3216 (4)	4·55 (57)	5·31 (68)	4·22 (51)	−0·97 (62)	1·00 (42)	−0·82 (48)
C(14)	−2991 (13)	915 (11)	3103 (7)	6·41 (71)	7·00 (95)	8·87 (90)	1·52 (80)	4·16 (66)	1·20 (72)
C(15)	−4154 (16)	953 (12)	3493 (8)	8·67 (94)	8·45 (1·16)	12·09 (1·25)	2·04 (95)	6·71 (94)	1·76 (96)
C(16)	−4279 (16)	286 (13)	3979 (7)	8·32 (96)	8·70 (1·07)	7·65 (89)	−0·12 (1·00)	4·00 (76)	−0·43 (81)
C(17)	−3296 (13)	−449 (12)	4092 (5)	5·87 (73)	9·25 (1·05)	5·18 (65)	−0·70 (85)	0·93 (54)	1·04 (67)
C(18)	−2122 (13)	−515 (11)	3717 (5)	6·06 (67)	8·33 (98)	5·27 (64)	0·75 (75)	1·48 (53)	−0·09 (64)
C(19)	2916 (11)	−1107 (10)	2824 (5)	4·76 (53)	6·93 (78)	5·25 (57)	−0·78 (70)	−0·38 (44)	−0·62 (61)
C(20)	4159 (11)	−1043 (11)	2505 (5)	5·44 (58)	8·65 (96)	5·02 (57)	−0·00 (76)	1·22 (46)	1·05 (64)
C(21)	5246 (12)	−1698 (13)	2624 (6)	4·96 (69)	10·72 (1·14)	5·88 (70)	1·40 (80)	0·80 (54)	0·60 (74)
C(22)	5179 (14)	−2463 (13)	3078 (7)	6·55 (85)	8·89 (1·09)	8·79 (99)	1·26 (93)	0·64 (72)	1·02 (86)
C(23)	3943 (15)	−2516 (14)	3419 (8)	5·98 (78)	9·58 (1·17)	10·44 (1·13)	0·91 (99)	−0·35 (74)	2·59 (99)
C(24)	2820 (12)	−1851 (10)	3302 (6)	4·80 (63)	6·79 (86)	7·54 (78)	−0·32 (71)	0·69 (55)	2·08 (67)
C(25)	−0070 (10)	−1914 (9)	841 (5)	3·27 (48)	5·89 (72)	5·05 (56)	0·98 (58)	−0·79 (42)	0·36 (52)
C(26)	289 (15)	−2541 (10)	349 (6)	8·61 (91)	5·95 (83)	6·27 (75)	0·40 (80)	0·63 (65)	−0·92 (63)
C(27)	−297 (20)	−3509 (10)	321 (7)	13·52 (1·36)	3·81 (71)	7·90 (94)	−1·37 (84)	−0·55 (92)	−1·14 (64)
C(28)	−1205 (20)	−3815 (11)	765 (7)	13·13 (1·33)	5·47 (80)	7·47 (87)	−1·53 (1·01)	−2·24 (88)	1·36 (83)
C(29)	−1551 (15)	−3188 (10)	1259 (7)	7·56 (89)	5·36 (80)	8·17 (89)	−1·02 (76)	0·03 (70)	0·43 (69)
C(30)	−957 (12)	−2224 (10)	1303 (5)	5·61 (65)	6·96 (85)	4·38 (56)	−1·51 (70)	−0·85 (48)	1·10 (55)

A Fourier synthesis based on the molybdenum coordinates revealed the positions of the two oxo oxygen atoms, and four oxygen and six carbon atoms belonging to the chelate rings. The partially known structure was refined on the basis of 39 positional parameters, 13 isotropic thermal parameters and 13 scale factors to $R=0{\cdot}26$. A difference synthesis ($F_o-F_{c\,\mathrm{Mo,O}}$) was used to locate the carbon atoms of the phenyl rings.

The structure (excluding the hydrogen atoms) was refined by minimizing $\sum W(|F_o|-|F_c|)^2$ by full-matrix least-squares calculations based on 2561 observable reflexions. The weights W were assigned as: $W=W_1 . W_2$, where $W_1=1$ for $|F_o|\le 34$ and $W_1=34/F_o$ for $|F_o|>34$; $W_2=1$ for $\sin\theta\ge 0{\cdot}4$ and $W_2=(\sin\theta)/0{\cdot}4$ for $\sin\theta<0{\cdot}4$. Isotropic full-matrix refinement led to $R=0{\cdot}124$. After three cycles of anisotropic refinement of all non-hydrogen atoms and hydrogen atoms at calculated positions after each cycle R was 0·073 and the weighted R for 2511 reflexions was 0·092. Some reflexions with very large F_c and small $\sin\theta$ values were omitted, as they were affected by extinction.

Table 2. *Hydrogen positions* ($\times 10^4$)

An isotropic temperature factor $U=4{\cdot}432$ Å^2 was used.

	x	y	z
H(2)	1777	−960	57
H(5)	160	−1115	3257
H(8)	3569	2129	604
H(9)	5302	2748	−154
H(10)	5731	1775	−1160
H(11)	4441	168	−1384
H(12)	2739	−467	−617
H(14)	−2861	1467	2721
H(15)	−4986	1549	3391
H(16)	−5252	321	4265
H(17)	−3481	−1001	4474
H(18)	−1320	−1104	3810
H(20)	4241	−449	2135
H(21)	6178	−1633	2352
H(22)	6026	−3020	3170
H(23)	3853	−3108	3788
H(24)	1858	−1919	3561
H(26)	1031	−2302	−11
H(27)	17	−4009	−84
H(28)	−1617	−4565	731
H(29)	−2298	−3447	1614
H(30)	−1294	−1722	1683

Table 3. *Observed and calculated structure factors*

[illegible]

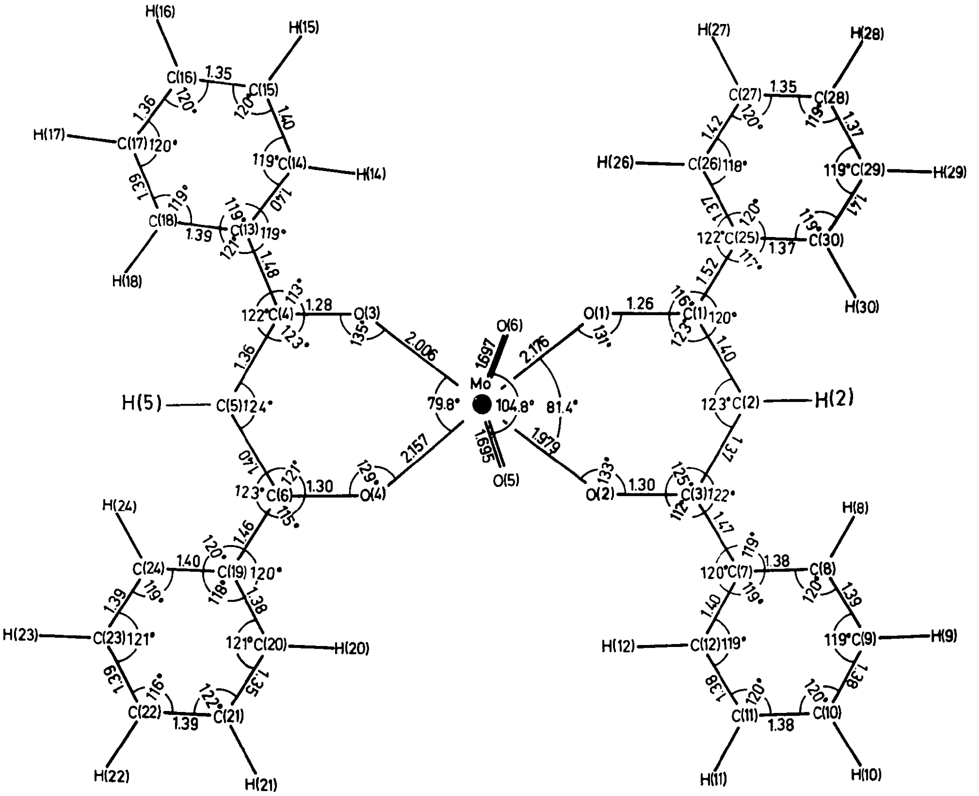

Fig. 1. One molecular unit of $(C_{15}H_{11}O_2)_2MoO_2$ including numbering of atoms, bond lengths and angles.

Atomic scattering factors given by Cromer & Waber (1965) and the anomalous dispersion correction for molybdenum (Cromer, 1965) were used.

Atomic positional and thermal parameters for the non-hydrogen atoms are listed in Table 1. The positions of the hydrogen atoms, calculated on stereochemical grounds, are given in Table 2. Observed and calculated structure factors are listed in Table 3.

Calculations were carried out on the CAE-90-40 computer (Zagreb) and the CDC 6600 computer (Ljubljana) with the X-RAY-70 system.

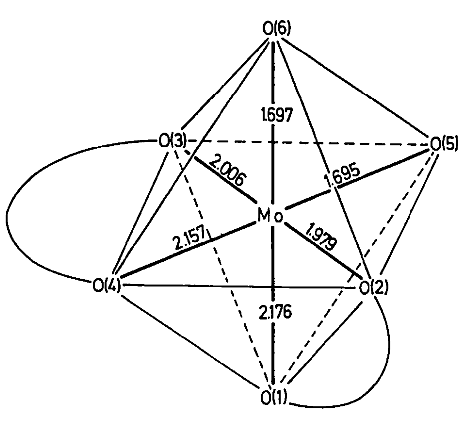

Fig. 2. The coordination around the molybdenum atom in $(C_{15}H_{11}O_2)_2MoO_2$.

Description and discussion of the structure

The molybdenum atom is coordinated by two oxo oxygen atoms O(5), O(6) and four oxygen atoms O(1),

Table 4. *Interatomic distances and angles*

(*a*) Distances

Within the Mo octahedron

Mo–O(1)	2·176 (9) Å	Mo–O(4)	2·157 (8) Å
Mo–O(2)	1·979 (8)	Mo–O(5)	1·695 (8)
Mo–O(3)	2·006 (7)	Mo–O(6)	1·697 (8)

Within the organic part of the molecule

O(1)—C(1)	1·26 (1)	C(15)–C(16)	1·35 (2)
O(2)—C(3)	1·30 (1)	C(16)–C(17)	1·36 (2)
O(3)—C(4)	1·28 (1)	C(17)–C(18)	1·39 (1)
O(4)—C(6)	1·30 (1)	C(18)–C(13)	1·39 (1)
C(1)—C(2)	1·40 (1)		
C(2)—C(3)	1·37 (1)	C(6)—C(19)	1·46 (1)
C(4)—C(5)	1·36 (1)	C(19)–C(20)	1·38 (1)
C(5)—C(6)	1·40 (1)	C(20)–C(21)	1·35 (1)
		C(21)–C(22)	1·39 (2)
C(3)—C(7)	1·47 (1)	C(22)–C(23)	1·39 (2)
C(7)—C(8)	1·38 (1)	C(23)–C(24)	1·39 (2)
C(8)—C(9)	1·39 (1)	C(24)–C(19)	1·40 (1)
C(9)—C(10)	1·38 (2)		
C(10)–C(11)	1·38 (2)	C(1)—C(25)	1·52 (1)
C(11)–C(12)	1·38 (1)	C(25)–C(26)	1·37 (1)
C(12)–C(7)	1·40 (1)	C(26)–C(27)	1·42 (2)
		C(27)–C(28)	1·35 (2)
C(4)—C(13)	1·48 (1)	C(28)–C(29)	1·37 (2)
C(13)–C(14)	1·40 (1)	C(29)–C(30)	1·41 (2)
C(14)–C(15)	1·40 (2)	C(30)–C(25)	1·37 (1)

Table 4 (*cont.*)

(*b*) Angles

O(1)—Mo—O(2)	81·4 (3)°
O(1)—Mo—O(3)	82·3 (3)
O(1)—Mo—O(4)	74·7 (3)
O(1)—Mo—O(5)	89·1 (3)
O(1)—Mo—O(6)	165·6 (3)
O(2)—Mo—O(3)	159·6 (3)
O(2)—Mo—O(4)	84·0 (3)
O(2)—Mo—O(5)	98·4 (3)
O(2)—Mo—O(6)	93·0 (3)
O(3)—Mo—O(4)	79·8 (3)
O(3)—Mo—O(5)	93·5 (3)
O(3)—Mo—O(6)	99·7 (3)
O(4)—Mo—O(5)	163·1 (3)
O(4)—Mo—O(6)	91·6 (3)
O(5)—Mo—O(6)	104·8 (4)
Mo—O(1)—C(1)	131 (1)
Mo—O(2)—C(3)	133 (1)
O(1)—C(1)—C(2)	123 (1)
O(1)—C(1)—C(25)	116 (1)
C(2)—C(1)—C(25)	120 (1)
C(1)—C(2)—C(3)	123 (1)
O(2)—C(3)—C(2)	125 (1)
O(2)—C(3)—C(7)	112 (1)
C(2)—C(3)—C(7)	122 (1)
Mo—O(3)—C(4)	135 (1)
Mo—O(4)—C(6)	129 (1)
O(3)—C(4)—C(5)	123 (1)
O(3)—C(4)—C(13)	113 (1)
C(5)—C(4)—C(13)	122 (1)
C(4)—C(5)—C(6)	124 (1)
C(5)—C(6)—C(19)	123 (1)
O(4)—C(6)—C(5)	121 (1)
O(4)—C(6)—C(19)	115 (1)
C(3)—C(7)—C(8)	119 (1)
C(3)—C(7)—C(12)	120 (1)
C(8)—C(7)—C(12)	119 (1)
C(7)—C(8)—C(9)	120 (1)
C(8)—C(9)—C(10)	119 (1)
C(9)—C(10)—C(11)	120 (1)
C(10)–C(11)–C(12)	120 (1)
C(11)–C(12)–C(7)	119 (1)
C(4)—C(13)–C(14)	119 (1)
C(4)—C(13)–C(18)	121 (1)
C(14)–C(13)–C(18)	119 (1)
C(13)–C(14)–C(15)	119 (1)
C(14)–C(15)–C(16)	120 (1)
C(15)–C(16)–C(17)	120 (1)
C(16)–C(17)–C(18)	120 (1)
C(17)–C(18)–C(13)	119 (1)
C(6)—C(19)–C(20)	120 (1)
C(6)—C(19)–C(24)	120 (1)
C(20)–C(19)–C(24)	118 (1)
C(19)–C(20)–C(21)	121 (1)
C(20)–C(21)–C(22)	122 (1)
C(21)–C(22)–C(23)	116 (1)
C(22)–C(23)–C(24)	121 (1)
C(23)–C(24)–C(19)	119 (1)
C(1)—C(25)–C(26)	122 (1)
C(1)—C(25)–C(30)	117 (1)
C(26)–C(25)–C(30)	120 (1)
C(25)–C(26)–C(27)	118 (1)
C(26)–C(27)–C(28)	120 (1)
C(27)–C(28)–C(29)	119 (1)
C(28)–C(29)–C(30)	119 (1)
C(29)–C(30)–C(25)	119 (1)

O(2), O(3), O(4) from the ligands, in a distorted octahedron (Figs. 1 and 2) with the oxo oxygen atoms in the *cis* configuration.

The arrangement of the molecules in the crystal projected on the (010) plane is shown in Fig. 3. The bond distances and angles are listed in Table 4.

The values of 1·695 and 1·697 Å for Mo–O(5) and Mo–O(6), respectively, indicate a double bond between the molybdenum atom and these oxygen atoms as expected for oxo oxygen atoms (Atovmyan & Sokolova, 1971). The O(5)–Mo–O(6) bond angle of 104·8° also agrees with the average value found in oxo-molybdenum complexes. The Mo–O distances in the chelate rings are different. Mo–O(1) and Mo–O(4) *trans* to the oxo oxygen atoms are longer (2·176 and 2·157 Å); Mo–O(2) and Mo–O(3) are shorter (2·006 and 1·979 Å). The bond angles (Table 4) also show that the coordination about molybdenum is far from regular octahedral.

The bond distances and angles within the chelate rings are in a good agreement with values found in analogous β-diketone complexes (Lingafelter & Braun, 1966; Cotton & Eiss, 1968). Both rings are planar with a similar departure of the atoms from the best plane. All four phenyl rings are planar within the limits of error (Table 5), while the C–C bond length of 1·38 (2) Å is in good agreement with the value of 1·395 (3) Å in aromatic compounds (*International Tables for X-ray Crystallography*, 1968).

References

Atovmyan, L. O. & Sokolova, J. A. (1971). *Zh. Strukt. Khim.* **12**, 851–858.

Bond, W. L. (1967). *International Tables for X-ray Crystallography*, Vol. II, pp. 295–298. Birmingham: Kynoch Press.

Cotton, F. A. & Eiss, R. (1968). *J. Amer. Chem. Soc.* **90**, 38–46.

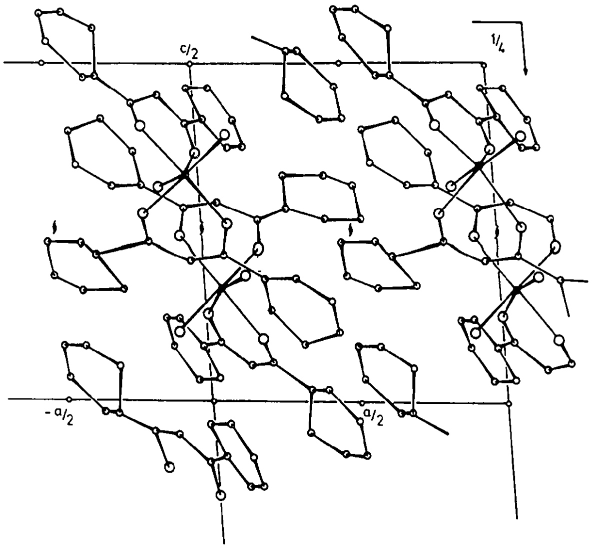

Fig. 3. Molecular packing projected on the *ac* plane.

Table 5. *Least-squares planes* (*expressed as* $Px+Qy+Rz=S$ *in direct space*)

Plane determined by

	Phenyl ring 1	Phenyl ring 2	Phenyl ring 3	Phenyl ring 4	Chelate ring 1	Chelate ring 2
P	6·606	4·475	3·356	6·918	6·489	2·582
Q	−6·622	8·040	8·171	−4·583	−5·621	8·209
R	9·313	12·403	13·919	10·690	10·894	14·735
S	1·544	3·244	4·021	1·738	1·742	4·051

Distances of the atoms from least-squares planes in Å and the corresponding standard deviation

C(7)	0·010	C(13)	−0·001	C(19)	−0·015	C(25)	−0·009	O(1)	−0·037	O(3)	−0·051
C(8)	−0·014	C(14)	0·001	C(20)	0·009	C(26)	0·000	C(1)	0·047	C(4)	0·067
C(9)	0·003	C(15)	−0·004	C(21)	0·004	C(27)	0·007	C(2)	−0·014	C(5)	−0·022
C(10)	0·010	C(16)	0·006	C(22)	−0·011	C(28)	−0·005	C(3)	−0·025	C(6)	−0·032
C(11)	−0·014	C(17)	−0·005	C(23)	0·004	C(29)	−0·004	O(2)	0·028	O(4)	0·039
C(12)	0·003	C(18)	0·002	C(24)	0·008	C(30)	0·011				
e.s.d.	0·011	e.s.d.	0·004	e.s.d.	0·010	e.s.d.	0·008	e.s.d.	0·036	e.s.d.	0·050

CROMER, D. T. (1965). *Acta Cryst.* **18**, 17–23.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

International Tables for X-ray Crystallography (1968). Vol. III 2nd ed., p. 276. Birmingham: Kynoch Press.

KAMENAR, B. & PENAVIĆ, M. (1973). *Cryst. Struct. Commun.* **2**, 41–44.

KORPAR, B. (1961). Ph. D. Thesis, Faculty of Science, Univ. of Zagreb, p. 87.

LARSON, M. L. & MOORE, F. W. (1966). *Inorg. Chem.* **5**, 801–805.

LINGAFELTER, E. C. & BRAUN, R. L. (1966). *J. Amer. Chem. Soc.* **88**, 2951–2956.

MORGAN, G. T. & CASTELL, R. A. S. (1928). *J. Chem. Soc.* 3252–3256.

Acta Cryst. (1974). **B30**, 305

Structure Cristalline de $CdCl_2.H_2O$

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(*Reçu le* 13 *juillet* 1973, *accepté le* 17 *septembre* 1973)

Cadmium chloride monohydrate crystallizes in the orthorhombic system (space group *Pnma*). The unit-cell dimensions are $a=9·25$ (2), $b=3·776$ (8), $c=11·89$ (2) Å; $Z=4$. The structure was refined by anisotropic least-squares calculations to a final *R* of 6·5 %, using 290 independent reflexions registered with a Weissenberg camera. The cadmium atoms are octahedrally coordinated. The crystal structure is described as octahedron chains packed along [010] and linked on their sides by hydrogen bonds. The crystal morphology can be easily explained by periodic bond chain theory.

Nous avons entrepris la détermination de la structure de $CdCl_2.H_2O$ dans le cadre d'une étude des hydrates du chlorure de cadmium (Hering, 1936) à une, deux et demi et quatre molécules d'eau et du bromure de cadmium tétra-hydraté.

Partie expérimentale

Les cristaux de $CdCl_2.H_2O$ s'obtiennent en évaporant à la température de 35 °C une solution saturée de $CdCl_2$ dans l'eau. Ils sont stables à la température ambiante et se présentent toujours sous forme d'aiguilles. Le groupe de Laue indique sans ambiguïté que $CdCl_2.H_2O$ cristallise dans le système orthorhombique. Les paramètres de la maille ont été déterminés à partir de clichés de précession, réalisés avec une chambre étalonnée. La densité a été mesurée au picnomètre en utilisant le benzène. Tous les résultats obtenus figurent dans le Tableau 1.

La direction d'allongement des cristaux est parallèle à l'axe [010]. Ils sont limités le plus fréquemment par les faces du prisme {101} et plus rarement par les faces des deux formes {101} et {001}. En outre, ils présentent les clivages très faciles {101} et {001}. Une très faible contrainte produit une importante déformation de ces cristaux; cette déformation se traduit sur des clichés de Weissenberg *h*0*l* par un allongement très important des taches de diffraction selon la direction [010].

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