References

Begley, M. J., Sowerby, D. B. & Haiduc, I. (1980). *J. Chem. Soc. Chem. Commun.* pp. 64–65.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Gottardi, G. (1961). *Z. Kristallogr.* **115**, 451–459.

Hall, M. & Sowerby, D. B. (1980). *J. Chem. Soc. Dalton Trans.* pp. 1292–1296.

International Tables for X-ray Crystallography (1974). Vol. IV. pp. 77–151. Birmingham: Kynoch Press.

Kavounis, C. A., Kokkou, S. C., Rentzeperis, P. J. & Karagiannidis, P. (1980). *Acta Cryst.* B**36**, 2954–2958.

Kokkou, S. C., Fortier, S., Rentzeperis, P. J. & Karagiannidis, P. (1983). *Acta Cryst.* C**39**, 178–180.

Lipka, A. (1980). *Z. Anor. Allg. Chem.* **466**, 195–202.

Main, P. (1970). Private communication. Univ. of York, England.

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1984). C**40**, 946–949

The Low-Temperature X-ray Diffraction Study of Bis[diisopropyl(phenyl)phosphine]-hexahydridoosmium, [Os($C_{12}H_{19}P$)$_2$$H_6$]

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Abstract. $M_r = 584{\cdot}8$, monoclinic, $P2_1/n$, $a = 17{\cdot}196\,(5)$, $b = 13{\cdot}799\,(5)$, $c = 10{\cdot}969\,(4)$ Å, $\beta = 98{\cdot}31\,(3)°$, $U = 2575\,(1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}48$ g cm^{-3} [$U = 2632\,(1)$ Å^3, 292 K], $D_x = 1{\cdot}49$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 50{\cdot}9$ cm^{-1}, $F(000) = 1176$, 200 K, $R(R_w) = 0{\cdot}034$ (0·032) for 5696 reflections. The Os—P bond lengths are 2·340 (1) and 2·347 (1) Å and the P—Os—P bond angle is 156·2 (1)°. Crystallographic evidence was obtained for only three of the six hydride ligands. The positions of the five located atoms bound to osmium are consistent with a triangular dodecahedral coordination geometry. The structure is compared with those of other eight-coordinate polyhydride complexes.

Introduction. Polyhydridophosphine complexes of the elements towards the centre of the second and third transition series have recently been the subject of renewed interest in terms of their synthesis (Chaudret, Commenges & Poilblanc, 1982; Crabtree & Hlatky, 1982; Green, Huffman & Caulton, 1983), structure (Howard, Mead & Spencer, 1983; Chaudret, Devillers & Poilblanc, 1983; Gregson, Howard, Nicholls, Spencer & Turner, 1980) and because they have been shown to be active in the cleavage of carbon–hydrogen bonds (Baudry, Ephritikhine & Felkin, 1980, 1982).

Douglas & Shaw (1970) reported the first osmium hexahydride, [$OsH_6(PMe_2Ph)_2$], as a pale yellow oil. Recent work on the polyhydrides of tungsten and rhenium (Howard, Gregson, Spencer & Turner, 1981; Gregson, Howard, Nicholls, Spencer & Turner, 1980) suggested that the ligand diisopropyl(phenyl)phosphine afforded polyhydride complexes of improved stability and crystallinity. This proved to be the case with osmium also and [$OsH_6(PPhPr^i_2)_2$] was synthesized in good yield (Connelly, Howard, Spencer & Woodley, 1984) as colourless crystals. Previous structural work on eight-coordinate polyhydrides has revealed the prevalence of the triangular dodecahedral coordination geometry. However, the structural preferences of $ML_6L'_2$ are not well established (Kepert, 1982). For this reason, [$OsH_6(PPhPr^i_2)_2$] represents an interesting extension to the series of polyhydride complexes we have previously characterized by X-ray and neutron diffraction. It is also a key starting material in our investigation of osmium hydride chemistry and we therefore undertook the low-temperature X-ray structure determination.

Experimental. [$OsH_6(PPhPr^i_2)_2$] prepared as described by Connelly, Howard, Spencer & Woodley (1984), crystals suitable for X-ray diffraction grown by slow cooling of a saturated solution in petroleum ether (b.p. 313–333 K), D_m by flotation in aqueous thallous formate. A room-temperature single-crystal X-ray investigation preceded the low-temperature study, but only the results of the latter are presented here. No phase changes were observed between 293 and 200 K, the temperature at which the diffracted intensities were recorded using a Nicolet *P*3*m* four-circle automated diffractometer. Measurement parameters are given in Table 1. Structure solved by Patterson and Fourier

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techniques using unique data (Table 1) corrected for the effect of X-ray absorption; all non-hydrogen atoms given anisotropic thermal parameters, aromatic and aliphatic hydrogen atoms refined satisfactorily with isotropic thermal parameters. 3 of the highest peaks in the later difference density syntheses were at distances (*ca* 1·7 Å) from the osmium atom consistent with the presence of hydride ligands, and gave sensible interbond angles P—Os—H and H—Os—H. Simultaneous refinement of positional and thermal parameters for these atoms was not stable although independent refinement gave plausible molecular parameters; all attempts to rationalize the remaining peaks failed, as did attempts to locate the other hydride ligands by the use of low-angle ($0 < \sin\theta < 0{\cdot}23$) difference density maps.*

Table 1. *Additional crystal and experimental data for* $[OsH_6(PPhPr^i_2)_2]$

Independent data	6169
Data used in final refinement $[I > \sigma(I)]$	5696
$R(R'_w)$	0·034 (0·032)
Function minimized	$\sum w(F_o - \lvert F_c \rvert)^2$
Weighting scheme $w = [\sigma^2(F) + g(F^2)]^{-1}$: g	0·00021
Scan type	ω:2θ
Scan range min., max. 2θ(°)	2·9, 60
Refinement type	Block-cascade least squares
Program system	*SHELXTL* (Sheldrick, 1978)
Scattering factors and corrections for anomalous dispersion	*International Tables for X-ray Crystallography* (1974)
Crystal shape, size (mm)	Irregular prism, 0·12 × 0·15 × 0·28
Absorption correction: empirical (μR)	0·6
Range of *hkl*	0,0,$\overline{16}$–22,19,15
Standard reflections	222, $\bar{2}\bar{4}0$
Frequency of standards	50
Intensity variation of standards (%)	1·4
$\Delta\rho$ final difference Fourier (e Å^{-3})	±1
Δ/σ	0·6
Reflections used for lattice-parameter determination (15)	083, 22$\bar{4}$, 253, $\overline{12}$,$\bar{1}$,3, $\bar{8}\bar{4}$2, 27$\bar{2}$, $\overline{10}$,5,1, 824, 45$\bar{3}$, 61$\bar{5}$, 613, 6$\bar{5}$1, $\bar{8}\bar{3}$1, 483, $\overline{10}$,$\bar{1}$,3

Table 2. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2 ×10^3)

	x	*y*	*z*	*U*
Os	2509 (1)	305 (1)	207 (1)	27 (1)*
P(1)	2996 (1)	1001 (1)	−1475 (1)	23 (1)*
P(2)	2311 (1)	−929 (1)	1603 (1)	23 (1)*
C(11)	3530 (3)	140 (3)	−2376 (4)	35 (1)*
C(12)	3711 (4)	529 (4)	−3607 (5)	53 (2)*
C(13)	4275 (3)	−238 (4)	−1612 (5)	43 (2)*
C(21)	2237 (3)	1532 (3)	−2656 (4)	32 (1)*
C(22)	1646 (3)	770 (4)	−3206 (5)	47 (2)*
C(23)	1817 (3)	2380 (4)	−2138 (5)	42 (2)*
C(31)	3710 (2)	1994 (3)	−1150 (4)	27 (1)*
C(32)	3816 (3)	2700 (3)	−2022 (4)	37 (1)*
C(33)	4343 (3)	3451 (3)	−1730 (5)	43 (2)*
C(34)	4776 (3)	3510 (3)	−572 (5)	46 (2)*
C(35)	4688 (3)	2807 (4)	286 (5)	41 (2)*
C(36)	4170 (3)	2048 (3)	−1 (4)	33 (1)*
C(41)	2178 (3)	−482 (3)	3167 (4)	35 (1)*
C(42)	2304 (3)	−1250 (4)	4184 (4)	47 (2)*
C(43)	1375 (4)	−18 (4)	3141 (5)	55 (2)*
C(51)	3127 (3)	−1808 (3)	1931 (4)	34 (1)*
C(52)	3892 (3)	−1322 (4)	2480 (5)	51 (2)*
C(53)	3232 (3)	−2385 (4)	768 (5)	47 (2)*
C(61)	1444 (2)	−1711 (3)	1208 (4)	27 (1)*
C(62)	1405 (3)	−2651 (3)	1675 (4)	35 (1)*
C(63)	731 (3)	−3203 (3)	1404 (5)	43 (2)*
C(64)	72 (3)	−2842 (4)	677 (5)	50 (2)*
C(65)	103 (3)	−1913 (4)	219 (5)	55 (2)*
C(66)	780 (3)	−1352 (4)	464 (5)	43 (2)*
H(1)	2355	1515	410	50†
H(2)	2117	−633	−834	50
H(3)	3433	722	890	50

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

† Parameters constrained.

Discussion. Atomic coordinates are given in Table 2; bond lengths and interbond angles in Table 3. Fig. 1 shows the $Os(PPhPr^i_2)_2$ fragment with the atomic numbering scheme and Fig. 2 depicts the OsH_3P_2 skeleton of the molecule. This X-ray investigation establishes the geometry of the OsP_2 fragment and gives probable atomic positions for three of the hydride ligands. However, the other three hydride ligands were not located and a detailed discussion of the coordination geometry is therefore precluded. The previous structural studies on eight-coordinate polyhydride complexes are summarized in Table 4. Of the two most plausible structures, the square antiprism (D_{4d}) and the triangular dodecahedron (D_{2d}) (Kepert, 1982), the latter appears to be favoured in the MH_4L_4 case because it offers two types of coordination site, designated *A* and *B* by Hoard & Silverton (1963). For the MH_4L_4 compounds the *A* sites, with four close neighbours in the coordination sphere, are occupied by the hydride ligands whereas the phosphine ligands adopt the *B* sites which have five neighbouring ligands. The *B* sites are less constricted than the *A* sites (Kepert, 1982; Hoard & Silverton, 1963), and this presumably explains the preference of phosphorus for the former. Table 4 shows the significant variations in the P—*M*—P angle which have been observed. As has been pointed out by Lobkovskii, Borisov, Makhaev & Semenenko (1980), there is an inverse correlation between the larger (*transoid*) P—*M*—P angle and the cone angle of the phosphine (Tolman, 1977). In other words, as the size of the phosphine ligand increases, the arrangement of phosphorus atoms about the metal more closely approaches the tetrahedral limit. The ReH_5L_3 structures are also described in terms of distorted dodecahedra, there being a large variation in the *transoid* P—Re—P angle on changing the phosphine ligand from PPh_3 to PMe_2Ph, although again this may be rationalized in terms of the phosphine cone angle. On the basis of this study the geometry of $[OsH_6(PPhPr^i_2)_2]$ also appears to be derived from the dodecahedron with the phosphine ligands occupying two opposed *B* sites. As there are no close non-bonded contacts between the phosphine ligands the geometry is

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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 39208 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and angles* (°)

Os–P(1)	2·340 (1)	Os–P(2)	2·347 (1)
Os–H(1)	1·711	Os–H(2)	1·792
Os–H(3)	1·753	P(1)–C(11)	1·869 (5)
P(1)–C(21)	1·852 (4)	P(1)–C(31)	1·840 (4)
P(2)–C(41)	1·868 (5)	P(2)–C(51)	1·850 (4)
P(2)–C(61)	1·840 (4)	C(11)–C(12)	1·527 (8)
C(11)–C(13)	1·518 (7)	C(21)–C(22)	1·524 (6)
C(21)–C(23)	1·527 (7)	C(31)–C(32)	1·395 (6)
C(31)–C(36)	1·389 (6)	C(32)–C(33)	1·384 (6)
C(33)–C(34)	1·378 (7)	C(34)–C(35)	1·374 (7)
C(35)–C(36)	1·383 (6)	C(41)–C(42)	1·531 (6)
C(41)–C(43)	1·519 (8)	C(51)–C(52)	1·522 (7)
C(51)–C(53)	1·537 (7)	C(61)–C(62)	1·400 (6)
C(61)–C(66)	1·395 (6)	C(62)–C(63)	1·383 (7)
C(63)–C(64)	1·382 (7)	C(64)–C(65)	1·380 (8)
C(65)–C(66)	1·391 (7)		

P(1)–Os–P(2)	156·2 (1)	P(1)–Os–H(1)	77·4
P(2)–Os–H(1)	125·7	P(1)–Os–H(2)	86·5
P(2)–Os–H(2)	79·4	H(1)–Os–H(2)	137·4
P(1)–Os–H(3)	78·3	P(2)–Os–H(3)	99·7
H(1)–Os–H(3)	76·8	H(2)–Os–H(3)	138·1
Os–P(1)–C(11)	114·5 (2)	Os–P(1)–C(21)	114·7 (1)
C(11)–P(1)–C(21)	103·4 (2)	Os–P(1)–C(31)	117·3 (1)
C(11)–P(1)–C(31)	102·1 (2)	C(21)–P(1)–C(31)	102·9 (2)
Os–P(2)–C(41)	114·1 (1)	Os–P(2)–C(51)	115·1 (1)
C(41)–P(2)–C(51)	103·2 (2)	Os–P(2)–C(61)	117·4 (1)
C(41)–P(2)–C(61)	102·0 (2)	C(51)–P(2)–C(61)	103·1 (2)
P(1)–C(11)–C(12)	115·0 (4)	P(1)–C(11)–C(13)	111·5 (3)
C(12)–C(11)–C(13)	110·2 (5)	P(1)–C(21)–C(22)	111·5 (3)
P(1)–C(21)–C(23)	111·5 (3)	C(22)–C(21)–C(23)	110·6 (4)
P(1)–C(31)–C(32)	122·5 (3)	P(1)–C(31)–C(36)	119·4 (3)
C(32)–C(31)–C(36)	118·1 (4)	C(31)–C(32)–C(33)	120·7 (4)
C(32)–C(33)–C(34)	120·4 (5)	C(33)–C(34)–C(35)	119·4 (4)
C(34)–C(35)–C(36)	120·7 (4)	C(31)–C(36)–C(35)	120·7 (4)
P(2)–C(41)–C(42)	114·6 (3)	P(2)–C(41)–C(43)	110·7 (3)
C(42)–C(41)–C(43)	109·8 (4)	P(2)–C(51)–C(52)	112·1 (3)
P(2)–C(51)–C(53)	110·8 (3)	C(52)–C(51)–C(53)	111·0 (4)
P(2)–C(61)–C(62)	122·5 (3)	P(2)–C(61)–C(66)	119·8 (3)
C(62)–C(61)–C(66)	117·7 (4)	C(61)–C(62)–C(63)	121·0 (4)
C(62)–C(63)–C(64)	121·1 (4)	C(64)–C(65)–C(66)	121·4 (5)
C(63)–C(64)–C(65)	118·3 (5)	C(61)–C(66)–C(65)	120·5 (4)

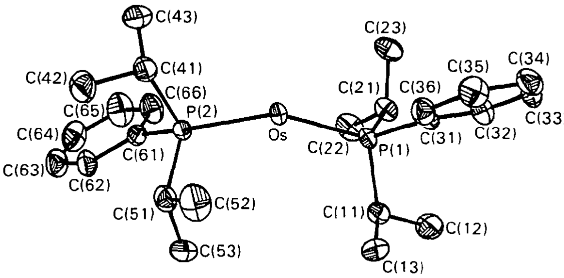

Fig. 1. Molecular configuration showing atomic numbering scheme. Hydride ligands and hydrogen atoms omitted.

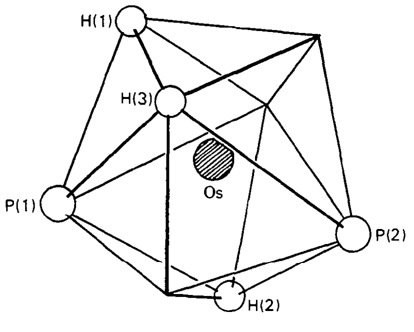

Fig. 2. Schematic representation of core atoms as vertices of idealized triangular dodecahedron. Those atoms occupying 'vacant' vertices were not located by the X-ray experiment.

Table 4. *Summary of mean* P–*M*–P *angles* (°) *for eight-coordinate polyhydride complexes*

	P–*M*–P angles	Reference
$[MoH_4(PMePh_2)_4]$	95·8, 142·8	(*a*)
$[WH_4(PEtPh_2)_4]$	96·6, 140·3	(*b*)
$[WH_4\{P(OPr^i)_3\}_4]$	93·7, 150·6	(*c*)
$[CrH_4(dmpe)_2]$*	85·0, 158·5	(*d*)
$[ReH_5(PPh_3)_3]$	107·1, 134·5	(*e*)
$[ReH_5(PMe_2Ph)_3]$	100·9, 149·5	(*f*)
$[OsH_6(PPhPr^i_2)_2]$	156·2	(*g*)

References: (*a*) Meakin, Guggenberger, Peet, Muetterties & Jesson (1973); (*b*) Lobkovskii, Makhaev, Borisov & Semenenko (1979); (*c*) Lobkovskii, Borisov, Makhaev & Semenenko (1980); (*d*) Girolami, Salt, Wilkinson, Thornton-Pett & Hursthouse (1983); (*e*) Ginsberg, Abrahams & Jamieson (1973); (*f*) Teller, Carroll & Bau (1983); (*g*) this work.

* dmpe = 1,2-bis(dimethylphosphino)ethane.

determined by the steric requirements of the atoms immediately attached to the osmium and this is reflected in the large P–Os–P angle, which is a common feature of those structures in which phosphine packing is of lesser importance (Table 4). The dodecahedral geometry is frequently described in terms of two orthogonal intersecting trapezia, each formed by two *A* and two *B* type sites. In the present structure P(1), P(2) and H(1) presumably form part of one such trapezoid. As expected, the osmium atom lies very close (displacement 0·09 Å) to the plane of these atoms. The other trapezoid includes H(2) and H(3), and the plane defined by these two atoms and the osmium makes an angle of 78·5° with the previous plane. This 11·5° deviation from 90° may reflect the poorly defined hydride positions.

Other features of the structure of $[OsH_6(PPhPr^i_2)_2]$ are in accord with expectations. Vectors joining the centroids *X*(1) and *X*(2) of the triangles C(11)C(21)C(31) and C(41)C(51)C(61) respectively to the appropriate phosphorus atoms form angles *X*(1)–P(1)–Os 178·0° and *X*(2)–P(2)–Os 177·6°, indicating that the phosphine ligands point directly at the metal atom. The Os–P separations of 2·340 (1) and 2·347 (1) Å are comparable with previously recorded values which lie in the range 2·31–2·56 Å, the shorter of these distances deriving from the neutron diffraction study of $[OsH_4(PMe_2Ph)_3]$ (Bau, Carroll, Hart, Teller & Koetzle, 1978) and the longer from the X-ray diffraction study of $[OsHBr(CO)(PPh_3)_3]$ (Orioli & Vaska, 1962). Variations in the P–C distances in the phosphine ligands reflect the smaller covalent radius of sp^2 C as compared to sp^3 C, and the angles Os–P–C > C–P–C as observed previously (Howard, Mead & Spencer, 1983).

Note added in proof (*February* 1984). In collaboration with Dr D. Gregson (ILL, Grenoble) we have found that very preliminary neutron diffraction data confirm the three given hydride positions and support the geometrical assignments.

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References

Bau, R., Carroll, W. E., Hart, D. W., Teller, R. G. & Koetzle, T. F. (1978). *Adv. Chem. Ser.* **167**, 73–92.

Baudry, D., Ephritikhine, M. & Felkin, H. (1980). *J. Chem. Soc. Chem. Commun.* pp. 1243–1244.

Baudry, D., Ephritikhine, M. & Felkin, H. (1982). *J. Organomet. Chem.* **224**, 363–376.

Chaudret, B., Commenges, G. & Poilblanc, R. (1982). *J. Chem. Soc. Chem. Commun.* pp. 1388–1390.

Chaudret, B., Devillers, J. & Poilblanc, R. (1983). *J. Chem. Soc. Chem. Commun.* pp. 641–643.

Connelly, N. G., Howard, J. A. K., Spencer, J. L. & Woodley, P. K. (1984). *J. Chem. Soc. Dalton Trans.* In the press.

Crabtree, R. H. & Hlatky, G. G. (1982). *J. Organomet. Chem.* **238**, C21–C23.

Douglas, P. G. & Shaw, B. L. (1970). *J. Chem. Soc.* (*A*), pp. 334–338.

Ginsberg, A. P., Abrahams, S. C. & Jamieson, P. B. (1973). *J. Am. Chem. Soc.* **95**, 4751–4752.

Girolami, G. S., Salt, J. E., Wilkinson, G., Thornton-Pett, M. & Hursthouse, M. B. (1983). *J. Am. Chem. Soc.* **105**, 5954–5956.

Green, M. A., Huffman, J. C. & Caulton, K. E. (1983). *J. Organomet. Chem.* **243**, C78–C82.

Gregson, D., Howard, J. A. K., Nicholls, J. N., Spencer, J. L. & Turner, D. G. (1980). *J. Chem. Soc. Chem. Commun.* pp. 572–573.

Hoard, J. L. & Silverton, J. V. (1963). *Inorg. Chem.* **2**, 235–243.

Howard, J. A. K., Gregson, D., Spencer, J. L. & Turner, D. G. (1981). *Acta Cryst.* A**37**, C240.

Howard, J. A. K., Mead, K. A. & Spencer, J. L. (1983). *Acta Cryst.* C**39**, 555–559.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Kepert, D. L. (1982). *Inorganic Stereochemistry.* Berlin: Springer-Verlag.

Lobkovskii, E. B., Borisov, A. P., Makhaev, V. D. & Semenenko, K. N. (1980). *Zh. Strukt. Khim.* **21**, 126–130; Engl. Transl: *J. Struct. Chem.* **21**, 100–103.

Lobkovskii, E. B., Makhaev, V. D., Borisov, A. P. & Semenenko, K. N. (1979). *Zh. Strukt. Khim.* **20**, 944–946; Engl. Transl: *J. Struct. Chem.* **20**, 812–813.

Meakin, P., Guggenberger, L. J., Peet, W. G., Muetterties, E. L. & Jesson, J. P. (1973). *J. Am. Chem. Soc.* **95**, 1467–1474.

Orioli, P. L. & Vaska, L. (1962). *Proc. Chem. Soc.* p. 333.

Sheldrick, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

Teller, R. G., Carroll, W. E. & Bau, R. (1983). Private communication.

Tolman, C. A. (1977). *Chem. Rev.* **77**, 313–348.

Acta Cryst. (1984). C**40**, 949–951

Structure of Bis(2-methyl-1,2-propanediamine-*N*,*N'*)bis(trichloroacetato-*O*)nickel(II), $[Ni(C_2Cl_3O_2)_2(C_4H_{12}N_2)_2]$

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Abstract. $M_r = 559{\cdot}8$, monoclinic, $P2_1/n$, $a = 7{\cdot}3163\,(8)$, $b = 16{\cdot}198\,(1)$, $c = 9{\cdot}943\,(4)$ Å, $\beta = 95{\cdot}44\,(1)°$, $V = 1173\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}58$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}54$ mm^{-1}, $F(000) = 572$, $T = 298$ K, final $R = 0{\cdot}060$ for 1481 reflexions. The central Ni atom is octahedrally coordinated by four N atoms in a plane and two trichloroacetate ions in *trans* positions. The bond distances and angles of the ligands are as expected. The large anisotropy in the Cl-atom thermal parameters indicates some disorder in the $-CCl_3$ group.

Introduction. As a continuation of our investigations of the structure of the Ni and Co transition-metal complexes of ethylenediamines and C-substituted ethylenediamines (Alcalá Aranda, Fernández, Gómez Beltrán & Larena, 1977; Gómez Beltrán, Valero Capilla & Alcalá Aranda, 1978, 1979; Valero Capilla, Alcalá Aranda & Gómez Beltrán, 1980), the crystal structure of another compound of this series is communicated herein.

Experimental. Blue crystal $0{\cdot}1 \times 0{\cdot}17 \times 0{\cdot}35$ mm, Nonius CAD-4-F automatic diffractometer, cell dimensions by least-squares fitting of the θ values of 25 reflexions, intensities collected by $\omega/2\theta$ scans, no appreciable drop in intensity of 3 standard reflexions checked every hour; 3451 reflexions within $0 < \theta < 29°$ and *hkl* range from $-10,0,0$ to $10,22,13$; 1481 with $I > 3\sigma(I)$ used in subsequent calculations; intensities corrected for Lorentz and polarization effects, not for absorption ($\mu R = 0{\cdot}54$); scattering factors for neutral atoms and anomalous-dispersion corrections for Ni and Cl from *International Tables for X-ray Crystallography* (1974); statistical test showed a centric intensity distribution; structure solved using

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