The metal framework is surrounded by a crowded and compact ligand sphere of 19 carbonyl groups. A minimization of their intra- and intermolecular repulsive forces in the packing to the present equilibrium C···O, C···C and O···O contact lengths is a suitable explanation of the differences obtained in the values of analogous Fe—Fe—Fe and Fe—Ge—Fe bond angles and of metal–metal bond lengths. The mean values of such bond lengths along the edges of the polyhedron are Fe—Fe = 2·719 (4) and Ge—Fe = 2·379 (4) Å. By comparison with the average Fe—Fe covalent single-bond length of 2·673 (7) Å – resulting from the two unbridged Fe—Fe bonds in $Fe_3(\mu\text{-}CO)_2(CO)_{10}$ (Wei & Dahl, 1969) – and the average Ge—Fe single-bond distance of 2·412 (1) Å in $\{(\mu_4\text{-}Ge)[\eta^5\text{-}C_5H_4CH_3\text{-}Mn(CO)_2][Fe(CO)_4]_3\}$ (Forster, Mackay & Nicholson, 1985), it can be assumed that the corresponding bond lengths of the title compound are also single bonds. Finally, the same bond type is realized in the remaining Ge—Re bond [ave. 2·542 (3) Å] because of the univalent chemical property of the related $Re(CO)_5$ group.

Intermolecular distances do not indicate any interactions exceeding van der Waals forces.

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Structure of a Cyclic Hydroxo-Bridged Pt^{II} Trimer with Platinum–Silver Bonds

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Abstract. *cyclo*-Tri-μ-hydroxo-(trinitroargentio)tris-[(ethylenediamine)platinum(II)] nitrate, $[AgPt_3(C_2H_8N_2)_3(OH)_3(NO_3)_3]NO_3$, $M_r = 1172·48$, monoclinic, Pc, $a = 8·283$ (4), $b = 8·319$ (5), $c = 17·507$ (9) Å, $\beta = 96·37$ (4)°, $V = 1199$ (1) Å^3, $Z = 2$, $D_x = 3·247$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 18·79$ mm^{-1}, $F(000) = 1068$, $T = 295$ K, $R = 0·046$ for 2094 unique observed reflections. The structure consists of a cyclic hydroxo-bridged trimer $[Pt(en)OH]_3^{3+}$ with an Ag atom located at equal distances from the three Pt atoms. The Pt_3O_3 ring assumes a chair conformation with approximate C_{3v} symmetry. The three Pt atoms possess square-planar coordination with minor distortion towards the square-pyramidal arrangement caused by the presence of the Ag atom. The Pt—Ag distances [2·838 (2), 2·890 (2) and 2·893 (2) Å] suggest the presence of metal–metal bonds. The coordination around the Ag atom defines a distorted trigonal antiprism including three nitrate O atoms with Ag—O distances = 2·47 (2), 2·60 (2) and 2·62 (2) Å. The amine and hydroxo ligands are hydrogen bonded to the nitrate groups.

Introduction. Neutral *cis*-dichloro Pt^{II} amine complexes are effective antitumor agents. The neutrality of the drug is believed to be important for its passage through the cell membranes. Within the cells where the chloride concentration is much lower than in the blood plasma, the platinum(II) compound is believed to hydrolyze. Hydrolysis of cisplatin, *cis*-$[Pt(NH_3)_2Cl_2]$, has been studied by several authors. The presence of several hydrated species, which are believed to play an important role in the antitumor activity of the drug, has been reported. At physiological pH, the hydrolyzed products isolated so far, for which X-ray structural data are available, were shown to contain oligomeric species. The crystal structures of hydroxo-bridged dimers (Faggiani, Lippert, Lock & Rosenberg, 1977*a*; Stanko, Hollis, Schreifels & Hoeschele, 1977; Lippert, Lock, Rosenberg & Zvagulis, 1978) and trimers (Faggiani, Lippert, Lock & Rosenberg, 1977*b*, 1978) have been reported. These oligomers are toxic (Rosenberg, 1978) and might be partly responsible for the toxicity of *cis*-$[Pt(NH_3)_2Cl_2]$.

Chelates in which the ammine ligands have been replaced by 1,2-cyclohexanediamine (dach) seem superior to NH_3 complexes as antitumor drugs especially because of their reduced toxicity. Oligomers of dach are much less toxic than those of NH_3 (Gill & Rosenberg, 1982). The crystal structure of a hydroxo-

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bridged trimer $[Pt(trans\text{-dach})OH]_3^{3+}$ has been reported (Macquet, Cros & Beauchamp, 1985).

Compounds of ethylenediamine (en) have also shown antitumor properties and they are less toxic than *cis*-$[Pt(NH_3)_2Cl_2]$ (Cleare, 1977). These compounds have been less studied than cisplatin. Lim & Martin (1976) have shown that $[Pt(en)(H_2O)(OH)]^+$ which is formed at neutral pH slowly produces the Pt^{II} dihydroxo-bridged dimer $[Pt(en)OH]_2^{2+}$. Broomhead, Fairlie & Whitehouse (1980) studied the immunosuppressive, nephrotoxic and gastrointestinal effects of the hydroxo-bridged dimer in rats. The dimer was synthesized from the following reactions:

$$[Pt(en)X_2] + 2AgNO_3 \xrightarrow{H_2O} [Pt(en)(H_2O)_2](NO_3)_2 + 2AgX\downarrow$$

$$\downarrow \text{NaOH} \mid \text{pH}\sim 6\cdot 5$$

$$[Pt(en)(OH)]_2(NO_3)_2$$

We have recently attempted to prepare the hydroxo-bridged dinuclear complex by this method in order to determine its crystalline structure. The first crystal which was analyzed by X-ray diffraction was found to be a hydroxo-bridged cyclic tetramer (Rochon, Morneau & Melanson, 1987).

We have now determined the crystal structure of a second hydrolyzed product, which was found to contain an Ag atom. This compound is a hydroxo-bridged trimer which can be formulated as $[\{Pt(en)(\mu\text{-}OH)\}_3Ag(NO_3)_3]NO_3$. This is the first example of a Pt^{II} hydroxo-bridged cyclic oligomer with an Ag atom.

Experimental. The compound was synthesized from the aqueous reaction of $[Pt(en)I_2]$ with $AgNO_3$ (1:2 ratio) in the dark. The AgI precipitate was filtered out and the filtrate was adjusted to a pH of 5·8 with NaOH. The next day the precipitate was filtered and recrystallized in water.

Yellow platelet, dimensions (mm): 0·104 [(100)–($\bar{1}00$)] × 0·230 [(010)–($0\bar{1}0$)] × 0·058 [(001)–($00\bar{1}$)] mounted roughly along the *b* axis; Syntex $P\bar{1}$ diffractometer: graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from refined angles of 15 centered reflections; 2761 independent reflections measured up to $2\theta < 55°$ by θ–2θ-scan technique; range of *hkl*: $h = 0\rightarrow 10$, $k = 0\rightarrow 10$, $l = -22\rightarrow 22$; standard reflections $40\bar{2}$, $10\bar{6}$ and 133, variations < 2·3%; reflections with $I_{net} < 2\cdot 5\sigma(I)$ unobserved, $\sigma(I)$ calculated as in Melanson, Hubert & Rochon (1975); absorption correction based on equations of crystal faces, transmission factors from 0·159 to 0·331; data corrected for Lorentz and polarization effects; 2094 unique observed reflections; atomic scattering factors of Cromer & Waber (1965) for Pt, Ag, O, N, C and of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion terms of Pt and Cl from Cromer (1965); isotropic secondary-extinction corrections from Coppens & Hamilton (1970).

The coordinates of the three Pt atoms were determined by direct methods on a Nicolet *SHELXTL* system; other non-H atoms located by structure-factor and Fourier-map calculations; refinement by block-diagonal least-squares calculations; H atoms fixed at calculated positions (C—H = 0·95 and N—H = 0·85 Å) with isotropic $B = 6\cdot 0$ Å^2; individual weights $w = 1/\sigma^2(F)$; ratio of max. least-squares shift to e.s.d. in final refinement cycle (on F) < 0·61 [for C(4)]; $\Delta\rho_{max} = 0\cdot 5$ e Å^{-3} (close to Pt) in final Fourier synthesis; goodness-of-fit 1·290; 307 refined parameters; $R = 0\cdot 046$ and $wR = 0\cdot 039$; calculations on a Cyber 830 with programs of Melanson, Hubert & Rochon (1975).*

Discussion. The refined atomic parameters are listed in Table 1. A labeled diagram of the molecule is shown in Fig. 1. The structure consists of a cyclic hydroxo-bridged trimer $[Pt(en)(\mu\text{-}OH)]_3^{3+}$ with an Ag atom located at equal distances from the three Pt atoms.

The Pt_3O_3 ring assumes a chair conformation with approximate C_{3v} symmetry (Fig. 1). The torsion angles in the Pt_3O_3 ring are −79·1 (8), 76·7 (8), −80·3 (8), 85·2 (8), −83·1 (8) and 79·6 (8)°. Similar conformations were observed for the SO_4^{2-} trimers $[Pt(dach)(\mu\text{-}OH)]_3^{3+}$ and $[Pt(NH_3)_2(\mu\text{-}OH)]_3^{3+}$ (Faggiani, Lippert, Lock & Rosenberg, 1978; Macquet *et al.*, 1985).

The three Pt atoms possess the normal square-planar coordination with minor distortion towards the square-pyramidal arrangement. Each Pt atom is slightly out of the plane defined by its four donor atoms, by 0·056 and 0·094 Å towards the Ag atom. The deviations from the best planes have been deposited. The angles between the square planes and the three-Pt-atom plane are 36·3° for Pt(1), 144·1° for Pt(2) and 38·5° for Pt(3) while the interplane angles are 121·0° for the Pt(1) plane–Pt(2) plane, 64·8° for the Pt(1) plane–Pt(3) plane and 115·9° for the Pt(2) plane–Pt(3) plane. The angles around each Pt atom are close to the expected 90 and 180° (Table 2) but there are some deviations owing to a slight strain caused by the bidentate ligands. The chelate angles N—Pt—N are smaller [82·9 (9), 84·8 (7) and 86·4 (8)°] as observed in other Pt–ethylenediamine complexes (Rochon, Morneau & Melanson, 1987; Faggiani, Lippert & Lock, 1980; Bau, Gellert, Lehovec & Louie, 1977) and in the chelate trimer $[Pt(dach)(\mu\text{-}OH)]_3^{3+}$ (Macquet *et al.*, 1985). The O—Pt—O angles are also smaller than expected [83·8 (6),

* Lists of anisotropic thermal factors, fixed coordinates of the H atoms, bond distances and angles in the nitrate ions, weighted least-squares planes, torsion angles, distances and angles involving possible hydrogen bonds and observed and calculated structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44550 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

85·3 (6) and 86·9 (6)°] as observed in $[Pt(dach)(\mu\text{-}OH)]_3^{3+}$ (Macquet *et al.*, 1985) whereas these angles were normal in the SO_4^{2-} and NO_3^- trimers $[Pt(NH_3)_2(\mu\text{-}OH)]_3^{3+}$ (Faggiani, Lippert, Lock & Rosenberg, 1977*b*, 1978). The *cis* angles O—Pt—N are larger than expected [93·6 (8) to 98·5 (7)°] while the *trans* angles are smaller than 180° [173·8 (7) to 177·2 (7)°] because of the square-pyramidal distortion. These departures from the square-planar coordination are mainly caused by the presence of the Ag atom located on top of the three Pt atoms. The Pt—O—Pt angles [111·8 (7) to 115·5 (7)°] are close to the tetrahedral value.

Table 1. *Positional parameters* ($\times 10^3$; Pt, Ag $\times 10^4$), *with their e.s.d.'s and temperature factors*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å$^2\times 10^4$)
Pt(1)	8730	2720 (1)	940	260
Pt(2)	10159 (1)	4597 (1)	2670 (1)	256
Pt(3)	6744 (1)	5964 (1)	1572 (1)	261
Ag	7194 (2)	2893 (2)	2341 (1)	321
O(1)	1064 (2)	380 (2)	159 (1)	259
O(2)	904 (2)	655 (2)	212 (1)	293
O(3)	798 (2)	504 (2)	72 (1)	324
N(1)	966 (3)	41 (3)	111 (1)	531
N(2)	704 (2)	159 (2)	23 (1)	384
N(3)	1136 (2)	278 (3)	324 (1)	448
N(4)	996 (3)	548 (3)	372 (1)	515
N(5)	558 (3)	696 (3)	238 (1)	363
N(6)	458 (2)	555 (3)	102 (1)	410
C(1)	880 (3)	−63 (4)	51 (2)	567
C(2)	706 (3)	−6 (4)	37 (1)	443
C(3)	1118 (4)	282 (5)	408 (2)	873
C(4)	1087 (5)	447 (5)	434 (2)	894
C(5)	371 (3)	664 (3)	216 (1)	358
C(6)	340 (3)	664 (3)	135 (2)	434
N(7)	404 (2)	115 (3)	201 (1)	418
N(8)	824 (2)	−30 (3)	331 (1)	393
N(9)	562 (2)	395 (3)	398 (1)	419
N(10)	266 (2)	147 (3)	988 (2)	607
O(4)	542 (2)	52 (2)	207 (1)	495
O(5)	285 (2)	27 (2)	213 (1)	570
O(6)	378 (2)	249 (3)	181 (1)	628
O(7)	788 (2)	−157 (3)	359 (1)	545
O(8)	771 (3)	103 (3)	355 (1)	665
O(9)	900 (3)	−31 (3)	273 (1)	686
O(10)	659 (3)	501 (3)	417 (1)	858
O(11)	516 (2)	366 (2)	332 (1)	548
O(12)	499 (2)	323 (3)	447 (1)	700
O(13)	300 (3)	48 (3)	1037 (1)	726
O(14)	143 (2)	145 (3)	938 (2)	895
O(15)	359 (3)	266 (4)	984 (2)	1211

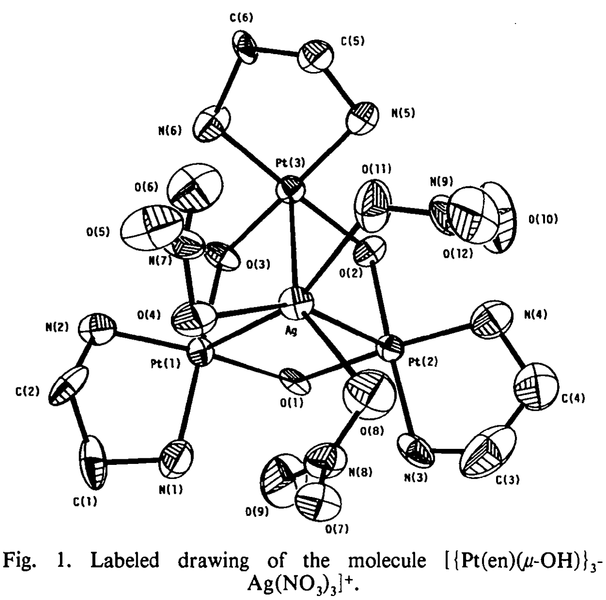

Fig. 1. Labeled drawing of the molecule $[\{Pt(en)(\mu\text{-}OH)\}_3Ag(NO_3)_3]^+$.

Table 2. *Bond distances* (Å) *and angles* (°)

Pt(1)–Ag	2·890 (2)	Pt(3)–N(6)	1·97 (2)
Pt(2)–Ag	2·838 (2)	N(1)–C(1)	1·48 (4)
Pt(3)–Ag	2·893 (2)	N(2)–C(2)	1·39 (4)
Pt(1)–O(1)	2·050 (15)	N(3)–C(3)	1·50 (4)
Pt(1)–O(3)	2·053 (15)	N(4)–C(4)	1·50 (4)
Pt(2)–O(1)	2·083 (16)	N(5)–C(5)	1·58 (3)
Pt(2)–O(2)	2·057 (16)	N(6)–C(6)	1·49 (3)
Pt(3)–O(2)	2·090 (15)	C(1)–C(2)	1·51 (4)
Pt(3)–O(2)	2·057 (16)	C(3)–C(4)	1·48 (6)
Pt(1)–N(1)	2·08 (2)	C(5)–C(6)	1·41 (4)
Pt(1)–N(2)	2·00 (2)	Ag–O(4)	2·47 (2)
Pt(2)–N(3)	2·01 (2)	Ag–O(11)	2·60 (2)
Pt(2)–N(4)	2·01 (2)	Ag–O(8)	2·62 (2)
Pt(3)–N(5)	1·98 (2)		
Pt(1)–O(1)–Pt(2)	115·5 (7)	Pt(1)–Ag–Pt(2)	75·2 (1)
Pt(1)–O(3)–Pt(3)	112·0 (7)	Pt(1)–Ag–Pt(3)	72·2 (1)
Pt(2)–O(2)–Pt(3)	111·8 (7)	Pt(2)–Ag–Pt(3)	73·6 (1)
O(1)–Pt(1)–O(3)	83·8 (6)	Ag–Pt(1)–O(1)	83·8 (4)
O(2)–Pt(3)–O(3)	85·3 (6)	Ag–Pt(1)–O(3)	87·9 (4)
O(1)–Pt(2)–O(2)	86·9 (6)	Ag–Pt(3)–O(3)	87·8 (4)
O(1)–Pt(1)–N(1)	94·3 (8)	Ag–Pt(3)–O(2)	85·9 (4)
O(1)–Pt(1)–N(2)	173·8 (7)	Ag–Pt(2)–O(1)	84·5 (4)
O(3)–Pt(1)–N(1)	174·8 (8)	Ag–Pt(2)–O(2)	88·0 (4)
O(3)–Pt(1)–N(2)	98·5 (7)	N(1)–C(1)–C(2)	108 (2)
O(3)–Pt(3)–N(5)	177·2 (7)	N(2)–C(2)–C(1)	109 (2)
O(3)–Pt(3)–N(6)	94·4 (7)	N(3)–C(3)–C(4)	111 (3)
O(2)–Pt(3)–N(5)	93·7 (7)	N(4)–C(4)–C(3)	113 (3)
O(2)–Pt(3)–N(6)	176·3 (7)	N(5)–C(5)–C(6)	108 (2)
O(1)–Pt(2)–N(3)	94·4 (7)	N(6)–C(6)–C(5)	110 (2)
O(1)–Pt(2)–N(4)	173·2 (8)	Ag–O(4)–N(7)	102 (1)
O(2)–Pt(2)–N(3)	176·5 (7)	Ag–O(11)–N(9)	122 (2)
O(2)–Pt(2)–N(4)	93·6 (8)	Ag–O(8)–N(8)	106 (2)
N(1)–Pt(1)–N(2)	82·9 (9)	Pt(1)–Ag–O(4)	95·9 (4)
N(3)–Pt(2)–N(4)	84·8 (9)	Pt(3)–Ag–O(4)	124·8 (4)
N(5)–Pt(3)–N(6)	86·4 (8)	Pt(2)–Ag–O(4)	156·8 (4)
Pt(1)–N(1)–C(1)	107 (2)	Pt(1)–Ag–O(8)	127·5 (5)
Pt(1)–N(2)–C(2)	111 (2)	Pt(3)–Ag–O(8)	154·1 (5)
Pt(3)–N(5)–C(5)	107 (1)	Pt(2)–Ag–O(8)	94·0 (5)
Pt(3)–N(6)–C(6)	108 (2)	Pt(1)–Ag–O(11)	161·0 (4)
Pt(2)–N(3)–C(3)	112 (2)	Pt(3)–Ag–O(11)	91·7 (4)
Pt(2)–N(4)–C(4)	112 (2)	Pt(2)–Ag–O(11)	110·6 (4)
		O(4)–Ag–O(8)	74·1 (6)
		O(4)–Ag–O(11)	84·8 (6)
		O(8)–Ag–O(11)	71·0 (6)

The Pt—Ag distances [2·890 (2), 2·838 (2) and 2·893 (2) Å] suggest the presence of metal–metal bonding. Several heteronuclear complexes containing Pt and Ag atoms have been structurally characterized and most of them showed short Pt—Ag distances. However, the existence of metal–metal bonding is not always certain because the presence of bridging ligands might be responsible for the close approach of the metal atoms (Schöllhorn, Thewalt & Lippert, 1987, and references therein; Usón, Forniés, Tomás, Casas, Cotton & Falvello, 1986, and references therein). A few structures without bridging ligands have been recently published (Usón, Fourniés, Tomás, Casas, Cotton & Falvello, 1985, 1986). Pt—Ag distances of 2·637 (1) to 2·827 Å have been observed. Our values, together with the fact that there are no ligands serving as bridges between the Ag and Pt atoms, strongly suggest metal–metal bonds. The Pt—Ag—Pt angles are 72·2 (1), 73·6 (1) and 75·2 (1)° while the Ag—Pt—O angles vary from 84·5 (4) to 88·0 (4)° (Table 2).

The Pt—N and Pt—O bonds [av. 2·02 (2) and 2·06 (2) Å respectively] agree with the values found in the hydroxo-bridged oligomers mentioned above. The bond lengths and angles within the ethylenediamine ligands are normal but the standard deviations are high owing to the large thermal factors of several of these atoms. The torsion angles have been deposited.

Some of the nitrate ions are disordered as shown by the high thermal factors. Values of U_{eq} up to 0·121 Å^2 have been observed. The N—O bond distances vary from 1·18 (3) to 1·28 (4) Å (mean 1·23 Å) and the O—N—O angles vary from 115 (3) to 126 (3)° (mean 120°).*

The overall coordinating sphere of the Ag atom includes three nitrate O atoms. The closest to the Ag atom is O(4) at 2·47 (2) Å, which is close to the shortest distance of 2·45 Å found in silver nitrate (Gibbons & Trotter, 1971; Lindley & Woodward, 1966). Values between 2·45 and 3·00 Å have been reported for Ag—O bonds (Gibbons & Trotter, 1971; Lindley & Woodward, 1966; Gagnon & Beauchamp, 1977; Gagnon, Hubert, Rivest & Beauchamp, 1977). Two other nitrate O atoms are located at 2·60 (2) and 2·62 (2) Å (Table 2). The angles Ag—O—N are 102 (1), 122 (2) and 106 (2)° while three different values are observed for the angles Pt—Ag—O [av. 93·9 (4), 121·0 (4) and 157·3 (4)°]. The O—Ag—O angles vary from 71·0 (6) to 84·8 (6)°. The coordination around the Ag atom defines a distorted trigonal antiprismatic geometry with three Pt atoms on one side and three O atoms (nitrates) on the other side (Fig. 1).

The packing of the molecules in the unit cell is shown in Fig. 2. It consists of alternate layers of cations and NO_3^- ions parallel to the *bc* plane. The ions are held together by an extensive hydrogen-bonding system involving the hydroxo and amine groups with the nitrate ions and ligands. The distances and angles of atoms possibly involved in hydrogen bonds have been deposited. The three hydroxo ligands form intermolecular hydrogen bonds with the three nitrate groups bonded to Ag. The O···O distances are 2·81 (2) to 2·83 (3) Å and the angles Pt—O···O vary from 104·7 (8) to 125·0 (8)°. All the amine groups except N(3) are also hydrogen bonded to the nitrate O atoms. The N···O distances vary from 2·95 (3) to 3·02 (3) Å with Pt—N···O angles 93 (2) to 139 (2)°.

* See deposition footnote.

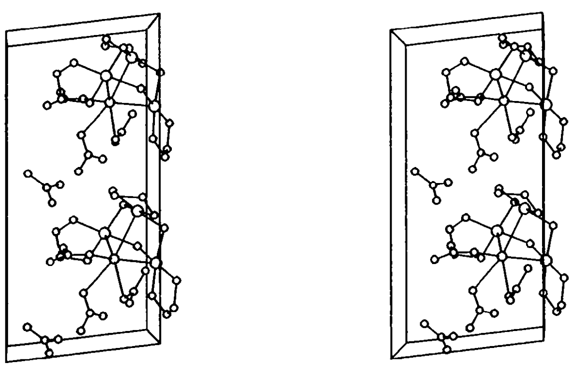

Fig. 2. Packing of the molecules in the unit cell (down *b* axis; *c* axis vertical).

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