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The Crystal and Molecular Structure of Chloropyridine-*N,N'*-tetramethylene-bis(salicylaldiminato)rhodium(III)

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The structure of the title compound has been determined by single-crystal X-ray analysis and refined by least-squares methods to an R of 0·060 with 3207 counter intensities. The crystals are monoclinic, space group $P2_1/c$, with $Z=4$ and $a=8·893$, $b=11·519$, $c=22·087$ Å, $\beta=104·17°$. The molecule has the pyridine and Cl ligands 91° apart with the Schiff-base twisted into a *cis-β*-configuration.

Introduction

While the *cis-β* configuration of a tetradentate Schiff-base ligand (Fig. 1) has been crystallographically demonstrated in several compounds where this arrangement is forced by a bidentate ligand occupying the other two sites in the coordination octahedron of a metal (Calligaris, Nardin & Randaccio, 1970; Bailey, Higson & McKenzie, 1972; Calligaris, Manzini, Nardin & Randaccio, 1972), there has been no previous report of an X-ray structure for a compound with a tetradentate Schiff-base and two monodentate ligands in which a *cis-β* configuration is adopted. Such a structure was proposed (van den Bergen, Cozens & Murray, 1970) for Me₂SnSalen [Salen²⁻ = N,N'-ethylenebis-(salicylaldiminato)dianion] on the basis of the ¹H n.m.r. spectrum, but an X-ray determination (Calligaris, Nardin & Randaccio, 1972a) showed the molecule to have the methyl groups *trans* with an essentially planar ligand. A more recent ¹H n.m.r. and infrared spectroscopic study has suggested that Me₂SnSalen exists in two forms both in solution and in the solid state, one having the methyl groups *trans* and the other having them *cis* (Kawakami, Miya-Uchi & Tanaka, 1974), although this awaits confirmation.

The recently reported Rh complex RhSalbnPyCl [Salbn²⁻ = N,N'-tetramethylenebis(salicylaldiminato) dianion] (Rogers & West, 1974a) was, on the basis of its ¹H n.m.r. spectrum, believed to have the Schiff-base

ligand twisted into a *cis-β* configuration with the pyridine and Cl also *cis* to one another (Rogers & West, 1974b). We report the structure of this compound which confirms the *cis-β* arrangement of the tetradentate ligand, and is the first X-ray determination to show such a configuration in the absence of a bidentate ligand.

Experimental

Crystals, grown by slow evaporation of a pyridine solution, were provided by Mr C. A. Rogers of this department. The orange-red compound crystallizes as elongated monoclinic prisms.

Crystal data

$C_{23}H_{23}N_3O_2ClRh$, $M=511·8$, monoclinic, $a=8·893$ (4), $b=11·519$ (6), $c=22·087$ (11) Å, $\beta=104·17°$, $U=2192$ Å³, $D_m=1·55$ (1) g cm⁻³ (by flotation in a mixture of carbon tetrachloride and hexane), $Z=4$, $D_c=1·55$ g cm⁻³. $F(000)=1040$, $\mu=9·01$ cm⁻¹ for Mo $K\alpha$ radiation ($\lambda=0·7107$ Å). Systematic absences indicate space group $P2_1/c$ (No. 14). The cell parameters were obtained with a Philips PW 1100 computer-controlled diffractometer by least-squares refinement of the reciprocal spacings for the $h00$, $0k0$, $00l$, $h0h$, $hh0$ and $0kk$ zones.

Intensity measurements

Intensities were collected on the diffractometer from a crystal $0·30 \times 0·25 \times 0·15$ mm with graphite-monochromated Mo $K\alpha$ radiation. A unique data set was collected out to 2θ (Mo $K\alpha$)=56° by the ω-scan

technique with a symmetric scan range of $\pm(0.5+0.1 \tan \theta)°$ in ω from the calculated scattering angle and a scan rate of $0.05°$ s^{-1}; no reflexion was of sufficient intensity to require the insertion of an attenuation filter.

The intensities of 4550 independent reflexions were measured of which 3207 obeyed the condition $F_o^2 > 3\sigma(F_o^2)$ and were used in subsequent calculations. Three standard reflexions measured at hourly intervals showed less than 2% decrease in intensity over the period of the data collection and no correction was made for decomposition.

A program specifically written for the PW 1100 diffractometer (Hornstra & Stubbe, 1972) was used to process the intensities. The standard deviations for the background-corrected intensities were calculated from

$$\sigma(I) = [CT + (t_c/t_b)^2(B_1 + B_2) + (pI)^2]^{1/2}$$

where CT is the total integrated peak count obtained in scan time t_c, B_1 and B_2 are background counts each obtained in time $\frac{1}{2}t_b$, and

$$I = CT - (t_c/t_b)(B_1 + B_2).$$

The factor p was taken as 0.04 to allow for other error sources. The data were corrected for Lorentz and polarization effects, but not for extinction or absorption.

Structure solution and refinement

The structure was solved by the heavy-atom method with scattering factors for Rh^0, Cl^0, O^0, N^0, C^0 and H^0 tabulated by Cromer & Waber (1965). The Patterson map and difference syntheses were calculated with *MONFR* (White, 1965). The function $\sum w(|F_o| - |F_c|)^2$ was minimized with *MONLS*, a modification of the full-matrix program of Busing, Martin & Levy (1962), and the block-diagonal program *MONDLS*, adapted from the '*SF*' series of Shiono (1968). The program *PLUTO* (Motherwell, 1974) was used for drawing the diagrams. Calculations were performed on the Monash University CDC 3200 and B 6700 computers, and the Cambridge University IBM 370/165 computer.

The Patterson synthesis indicated that the Rh atoms were in the $y=0$ and $y=\frac{1}{2}$ planes, and a structure-factor calculation based on the Rh position with the 1892 reflexions with $F_o^2 > 10\sigma(F_o^2)$ gave $R_1 = 0.469$ and $R_2 = 0.539$ where $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$ and $R_2 = (\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2)^{1/2}$.
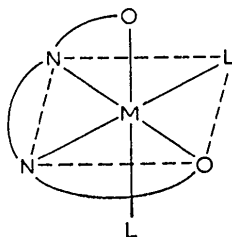

Fig. 1. The *cis-β* configuration for a coordinated O_2N_2 tetradentate Schiff-base ligand.

A difference synthesis calculated from these structure factors displayed mirror symmetry about the $y=0$ plane due to the heavy atom. Chemically reasonable atoms in this difference map were located with the aid of a model made assuming the *cis-β* arrangement suggested by the ^1H n.m.r. spectrum, and several further difference syntheses made it possible to locate all the non-hydrogen atoms.

Full-matrix least-squares refinement with isotropic temperature factors for all atoms, varying positional and thermal parameters separately, and then block-diagonal refinement with anisotropic temperature factors for Rh, led to convergence with $R_1 = 0.070$, $R_2 = 0.084$.

The positional parameters were used to calculate idealized H positions (C–H = 1.0 Å) and a difference

Table 1. *Final positional and thermal parameters of non-hydrogen atoms*

Numbers in parentheses here and in succeeding tables are estimated standard deviations in the least significant digits. The B values are the parameters in the isotropic temperature-factor expression $\exp[-B \sin^2 \theta/\lambda^2]$. The atomic scattering factors for Rh and Cl are expressed as $f = f_0 \exp[-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{12}a^*b^*hk + 2U_{13}a^*c^*hl + 2U_{23}b^*c^*kl]$.

(*a*) Isotropic temperature factors (fractional coordinates $\times 10^4$ and $B \times 10$)

	x	y	z	B (Å²)
O(1)	−1339 (12)	−1544 (8)	1251 (5)	33 (2)
O(2)	−2463 (11)	520 (8)	672 (4)	29 (2)
N(1)	784 (13)	98 (10)	896 (5)	28 (2)
N(2)	−353 (15)	1845 (10)	1642 (6)	32 (2)
N(3)	−2395 (14)	130 (10)	1956 (5)	32 (2)
C(1)	1257 (18)	−842 (13)	700 (7)	31 (3)
C(2)	802 (18)	−2017 (13)	801 (7)	30 (3)
C(3)	−484 (17)	−2295 (12)	1056 (6)	26 (3)
C(4)	−877 (20)	−3488 (14)	1088 (7)	37 (3)
C(5)	−57 (22)	−4334 (16)	855 (8)	45 (4)
C(6)	1161 (22)	−4061 (16)	597 (8)	47 (4)
C(7)	1589 (20)	−2919 (14)	570 (7)	38 (3)
C(8)	1438 (19)	1195 (14)	707 (7)	35 (3)
C(9)	2716 (22)	1707 (15)	1244 (8)	46 (4)
C(10)	2229 (24)	2621 (16)	1651 (9)	50 (4)
C(11)	1144 (21)	2270 (14)	2052 (8)	39 (3)
C(12)	−1371 (19)	2643 (13)	1432 (7)	32 (3)
C(13)	−2829 (18)	2493 (12)	967 (7)	30 (3)
C(14)	−3760 (21)	3519 (15)	839 (8)	43 (4)
C(15)	−5083 (23)	3528 (17)	349 (9)	51 (4)
C(16)	−5484 (22)	2544 (16)	−17 (8)	46 (4)
C(17)	−4593 (18)	1538 (14)	101 (7)	35 (3)
C(18)	−3241 (17)	1500 (12)	606 (6)	26 (3)
C(19)	−3688 (19)	−527 (14)	1753 (7)	37 (3)
C(20)	−4845 (23)	−553 (16)	2083 (8)	48 (4)
C(21)	−4677 (25)	97 (16)	2616 (9)	54 (4)
C(22)	−3357 (24)	776 (18)	2818 (9)	55 (4)
C(23)	−2216 (21)	772 (15)	2488 (8)	43 (4)

(*b*) Anisotropic temperature factors (fractional coordinates $\times 10^5$ and $U_{ij} \times 10^4$)

	x	y	z
Rh	−7561 (13)	1373 (9)	14368 (5)
Cl	11546 (45)	−5435 (39)	22937 (18)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh	288 (5)	396 (5)	281 (5)	6 (6)	47 (4)	31 (5)
Cl	310 (19)	692 (27)	369 (18)	32 (19)	23 (17)	187 (19)

synthesis at this stage confirmed these positions as well as indicating substantial anisotropy in the thermal motion of the Cl atom. The calculated H positions were included in subsequent structure-factor calculations (with isotropic temperature factors one unit greater than the attached C) and further refinement, with anisotropic temperature factors for Rh and Cl, led to convergence with $R_1 = 0.060$, $R_2 = 0.074$. Table 1 shows the final positional and thermal parameters for the non-hydrogen atoms with standard deviations derived from the inverse least-squares matrix, and Table 2 gives the calculated H atom positions.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31120 (20 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Idealized hydrogen atom positions*

Numbering corresponds to the attached carbon atoms
(fractional coordinates $\times 10^4$).

	x	y	z
H(1)	2049	−755	450
H(4)	−1741	−3721	1279
H(5)	−356	−5166	873
H(6)	1730	−4687	430
H(7)	2473	−2709	382
H(12)	−1136	3438	1605
H(14)	−3462	4232	1099
H(15)	−5752	4235	262
H(16)	−6423	2562	−376
H(17)	−4912	838	−167
H(19)	−3815	−996	1361
H(20)	−5787	−1067	1933
H(21)	−5500	93	2851
H(22)	−3227	1253	3201
H(23)	−1258	1240	2642
H(8)	1898	1032	344
H(8′)	590	1782	580
H(9)	3178	1046	1522
H(9′)	3522	2058	1056
H(10)	3199	2929	1935
H(10′)	1705	3254	1366
H(11)	1635	1636	2347
H(11′)	944	2955	2301

A final difference synthesis showed no features greater than 1·8 e Å^{-3}.

Discussion of the structure

The asymmetric unit consists of one molecule, and a stereoscopic view of this, giving the labelling scheme, is shown in Fig. 2. H atoms have been omitted for clarity. Bond lengths and angles with their standard deviations are given in Table 3. The coordination about Rh is approximately octahedral, with angles between neighbouring vertices of the octahedron varying from
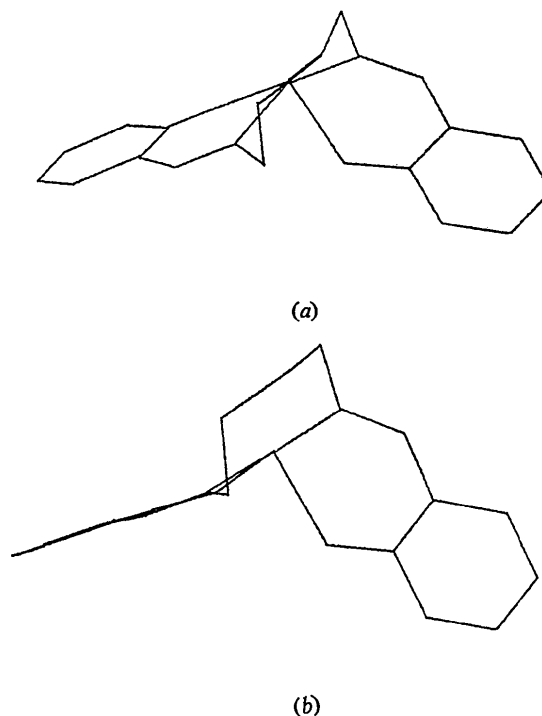

(a)

(b)

Fig. 3. Projections of the coordinated Schiff-base ligand, looking towards the Rh atom (a) from the mid-point of C(9)–C(10) and (b) along the bisector of N(1)–Rh–N(2).
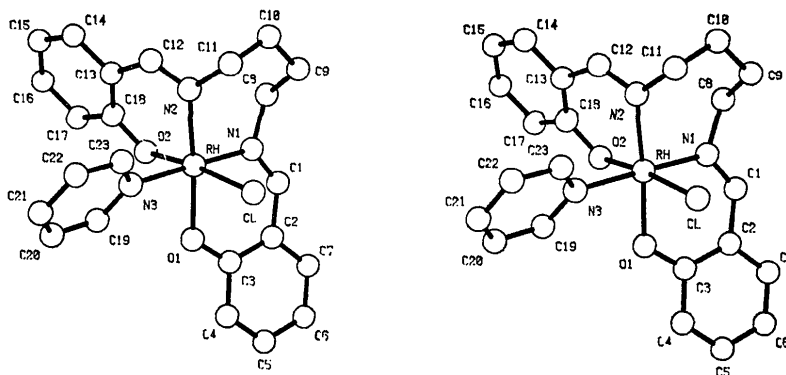

Fig. 2. A stereoscopic view of the RhSalbnPyCl molecule showing the labelling scheme.
(Hydrogen atoms have been omitted for clarity.)

Table 3. *Bond lengths and angles*

(a) Bond lengths (Å)

Rh—Cl	2·347 (4)	C(10)–C(11)	1·52 (3)
Rh—O(1)	2·021 (10)	N(2)—C(11)	1·50 (2)
Rh—O(2)	2·024 (9)	N(2)—C(12)	1·29 (2)
Rh—N(1)	2·026 (13)	C(12)–C(13)	1·45 (2)
Rh—N(2)	2·030 (12)	C(13)–C(14)	1·43 (2)
Rh—N(3)	2·064 (14)	C(14)–C(15)	1·39 (2)
N(1)–C(1)	1·28 (2)	C(15)–C(16)	1·39 (3)
C(1)–C(2)	1·45 (2)	C(16)–C(17)	1·39 (3)
C(2)–C(3)	1·43 (2)	C(17)–C(18)	1·43 (2)
C(3)–C(4)	1·42 (2)	C(13)–C(18)	1·39 (2)
C(4)–C(5)	1·39 (3)	O(2)—C(18)	1·31 (2)
C(5)–C(6)	1·38 (3)	N(3)–C(19)	1·36 (2)
C(6)–C(7)	1·38 (3)	C(19)–C(20)	1·40 (3)
C(2)–C(7)	1·42 (2)	C(20)–C(21)	1·37 (3)
O(1)–C(3)	1·29 (2)	C(21)–C(22)	1·39 (3)
N(1)–C(8)	1·49 (2)	C(22)–C(23)	1·39 (3)
C(8)–C(9)	1·55 (2)	N(3)—C(23)	1·37 (2)
C(9)–C(10)	1·52 (3)		

(b) Angles (°)

O(1)–Rh—N(2)	175·0 (5)	C(9)—C(10)–C(11)	118 (2)
N(1)–Rh—N(3)	177·3 (5)	N(2)—C(11)–C(10)	105 (1)
O(2)–Rh—Cl	173·1 (3)	O(2)—C(18)–C(13)	126 (1)
O(1)–Rh—N(1)	92·0 (4)	C(12)–C(13)–C(18)	125 (1)
N(1)–Rh—O(2)	89·4 (4)	N(2)—C(12)–C(13)	126 (1)
O(2)–Rh—N(2)	91·7 (5)	C(11)–N(2)—C(12)	115 (1)
N(2)–Rh—N(3)	89·4 (5)	O(1)—C(3)—C(4)	117 (1)
N(3)–Rh—Cl	91·2 (4)	C(1)—C(2)—C(7)	117 (1)
Cl—Rh—O(1)	86·7 (3)	C(12)–C(13)–C(14)	115 (1)
O(1)–Rh—O(2)	86·4 (4)	O(2)—C(18)–C(17)	116 (1)
O(1)–Rh—N(3)	85·9 (5)	C(2)—C(7)—C(6)	121 (2)
N(2)–Rh—N(1)	92·6 (5)	C(5)—C(6)—C(7)	119 (2)
N(1)–Rh—Cl	90·4 (3)	C(4)—C(5)—C(6)	122 (2)
O(2)–Rh—N(3)	88·7 (4)	C(3)—C(4)—C(5)	120 (2)
Cl—Rh—N(2)	95·2 (4)	C(2)—C(3)—C(4)	118 (1)
Rh—O(1)–C(3)	125 (1)	C(3)—C(2)—C(7)	119 (1)
Rh—N(1)–C(1)	123 (1)	C(13)–C(14)–C(15)	120 (2)
Rh—O(2)–C(18)	123 (1)	C(14)–C(15)–C(16)	120 (2)
Rh—N(2)–C(11)	122 (1)	C(15)–C(16)–C(17)	121 (2)
Rh—N(2)–C(12)	123 (1)	C(16)–C(17)–C(18)	120 (2)
Rh—N(3)–C(19)	118 (1)	C(13)–C(18)–C(17)	118 (1)
Rh—N(3)–C(23)	122 (1)	C(14)–C(13)–C(18)	121 (1)
Rh—N(1)–C(8)	121 (1)	N(3)—C(19)–C(20)	121 (2)
O(1)–C(3)–C(2)	125 (1)	C(19)–C(20)–C(21)	120 (2)
C(1)–C(2)–C(3)	124 (1)	C(20)–C(21)–C(22)	119 (2)
N(1)–C(1)–C(2)	128 (1)	C(21)–C(22)–C(23)	120 (2)
C(1)–N(1)–C(8)	116 (1)	N(3)—C(23)–C(22)	121 (2)
N(1)–C(8)–C(9)	112 (1)	C(19)–N(3)—C(23)	120 (1)
C(8)–C(9)–C(10)	117 (2)		

85·9 to 95·2°. The tetradentate Schiff-base ligand adopts a *cis-β* configuration and the pyridine and Cl ligands are therefore *cis*, with an angle between them of 91·2°. The distances from Rh of the O and N donors are all very similar, although that of the pyridine N is marginally longer, perhaps indicating weaker coordination. The bond lengths within the Schiff-base are all similar to those found in the many complexes of the related ligand Salen (Calligaris, Nardin & Randaccio, 1972*b*). Furthermore the bond lengths in the two salicylaldimine groups show no significant differences.

Several least-squares planes were calculated (Blow, 1960). Equations of these, with distances of relevant atoms from them, are shown in Table 4. As expected, the pyridine molecule and benzene rings are planar. However the chelate rings of the salicylaldimine groups

and Rh form poor planes, being noticeably puckered. The 'coordination planes' defined by Cl, N(1) O(2), N(3) and by Cl, O(1), O(2), N(2) are only moderate, but that defined by N(1), N(2), N(3), O(1) is very good. The displacement of the Rh atom from these planes, while significant, is quite small.

Table 4. *Equations of least-squares planes and distances of individual atoms (Å) from the planes*

X, Y, Z are coordinates in Å referred to the set of Cartesian axes a, b, c^*. These are related to fractional coordinates x, y, z in the crystal system by the matrix equation

$$\begin{pmatrix} X \\ Y \\ Z \end{pmatrix} = \begin{pmatrix} 8\cdot893 & 0 & -5\cdot404 \\ 0 & 11\cdot519 & 0 \\ 0 & 0 & 21\cdot413 \end{pmatrix} \begin{pmatrix} x \\ y \\ z \end{pmatrix}$$

(1) Plane through N(3), C(19), C(20), C(21), C(22), C(23)
$-0\cdot3332X + 0\cdot7691Y - 0\cdot5455Z + 1\cdot1098 = 0$

N(3)	0·002 (11)	C(21)	−0·003 (19)
C(19)	0·003 (16)	C(22)	0·008 (20)
C(20)	−0·003 (19)	C(23)	−0·008 (17)

(2) Plane through C(2), C(3), C(4), C(5), C(6), C(7)
$-0\cdot4282X + 0\cdot0892Y - 0\cdot8993Z + 1\cdot8598 = 0$

C(2)	−0·010 (15)	C(5)	−0·012 (18)
C(3)	0·020 (14)	C(6)	0·022 (18)
C(4)	−0·008 (16)	C(7)	−0·011 (17)

(3) Plane through C(13), C(14), C(15), C(16), C(17), C(18)
$0\cdot7210X + 0\cdot3177Y - 0\cdot6158Z + 2\cdot5583 = 0$

C(13)	0·004 (16)	C(16)	0·003 (19)
C(14)	0·002 (18)	C(17)	0·003 (16)
C(15)	−0·005 (20)	C(18)	−0·006 (14)

(4) Plane through Rh, N(1), C(1), C(2), C(3), O(1)
$-0\cdot4734X + 0\cdot1377Y - 0\cdot8700Z + 1\cdot8342 = 0$

Rh	−0·135 (1)	O(1)	0·143 (10)
N(1)	0·080 (11)	C(4)	−0·099 (16)
C(1)	0·046 (15)	C(5)	−0·204 (18)
C(2)	−0·111 (15)	C(6)	−0·227 (18)
C(3)	−0·023 (14)	C(7)	−0·214 (17)

(5) Plane through Rh, N(2), C(12), C(13), C(18), O(2)
$0\cdot7366X + 0\cdot2053Y - 0\cdot6444Z + 2\cdot8441 = 0$

Rh	−0·173 (1)	O(2)	0·158 (10)
N(2)	0·131 (13)	C(14)	−0·278 (18)
C(12)	0·026 (16)	C(15)	−0·271 (20)
C(13)	−0·140 (16)	C(16)	−0·115 (19)
C(18)	−0·001 (14)	C(17)	0·019 (16)

(6) Plane through Cl, N(1), O(2), N(3)
$-0\cdot1643X - 0\cdot9592Y - 0\cdot2302Z + 0\cdot5377 = 0$

Cl	0·043 (5)	N(3)	−0·047 (12)
N(1)	−0·047 (11)	Rh	−0·084 (1)
O(2)	0·051 (10)		

(7) Plane through Cl, O(1), O(2), N(2)
$0\cdot8250X - 0\cdot0181Y - 0\cdot5648Z + 2\cdot9761 = 0$

Cl	0·038 (4)	Rh	0·040 (1)
O(1)	−0·044 (11)	C(12)	−0·455 (16)
O(2)	0·045 (10)	C(13)	−0·753 (16)
N(2)	−0·039 (13)	C(18)	−0·436 (15)

(8) Plane through N(1), N(2), N(3), O(1)
$-0\cdot5339X + 0\cdot2633Y - 0\cdot8035Z + 1\cdot6243 = 0$

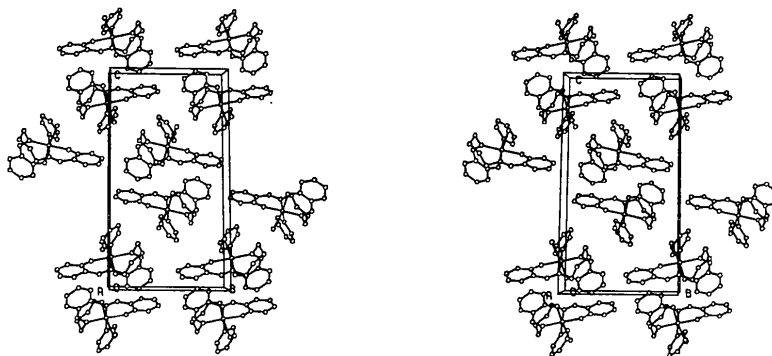|			
N(1)	−0·001 (11)	Rh	−0·033 (1)
N(2)	0·001 (12)	C(1)	−0·231 (15)
N(3)	−0·001 (12)	C(2)	−0·516 (15)
O(1)	0·001 (10)	C(3)	−0·354 (14)

Fig. 4. A stereoscopic view of the packing. The origin of the cell is labelled O and the axes a,b,c are OA,OB,OC respectively. (Hydrogen atoms have been omitted for clarity.)

The conformation of the tetramethylene bridge is of some interest as there are no previous structural determinations for such a unit. While the bond lengths are all normal for C–C single bonds and very similar to that in the ethylene bridge of Salen (Calligaris, Nardin & Randaccio, 1972b), C(8)–C(9)–C(10) and C(9)–C(10)–C(11) of 117 and 118° respectively are much larger than the tetrahedral angle and are closer to the trigonal angle. This suggests considerable strain in the bridge and makes it perhaps surprising that regular octahedral coordination is so well preserved with N(1)–Rh–N(2) = 92·6°. The conformation of the bridge and remainder of the Schiff-base ligand is illustrated by Fig. 3, which gives views from the mid-point of C(9)–C(10) towards Rh (a) and along the bisector of N(1)–Rh–N(2) (b).

Fig. 4 gives a stereoscopic view of the packing of the molecules in the crystal.

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