*Refinement*

Refinement on $F$
$R = 0.038$
$wR = 0.044$
2434 reflections
117 parameters
$w = 1/[\sigma^2(F) + 0.003352F^2]$
$(\Delta/\sigma)_{\max} = 0.032$
$\Delta\rho_{\max} = 1.4$ e Å$^{-3}$
$\Delta\rho_{\min} = -1.3$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| Ni | 1/2 | 1/2 | 1/2 | 0.0175 (1) |
| Cl | 0.86552 (6) | 0.39203 (7) | 0.75640 (6) | 0.0323 (1) |
| O1 | 0.5051 (2) | 0.4076 (2) | 0.6713 (2) | 0.0254 (3) |
| O2 | 0.7674 (2) | 0.6204 (2) | 0.4991 (2) | 0.0290 (3) |
| O3 | 0.2030 (3) | 1.1479 (2) | 0.9817 (2) | 0.0446 (5) |
| N | 0.4173 (2) | 0.7825 (2) | 0.7075 (2) | 0.0205 (3) |
| C1 | 0.4154 (3) | 0.7593 (3) | 0.8547 (2) | 0.0263 (4) |
| C2 | 0.3784 (2) | 0.5427 (3) | 0.8249 (2) | 0.0269 (4) |
| C3 | 0.5690 (3) | 0.9187 (2) | 0.6746 (2) | 0.0286 (4) |
| C4 | 0.7649 (3) | 0.8256 (3) | 0.6212 (2) | 0.0334 (5) |
| C5 | 0.2262 (2) | 0.8510 (3) | 0.7236 (2) | 0.0269 (4) |
| C6 | 0.1811 (3) | 1.0763 (3) | 0.8222 (3) | 0.0344 (5) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Ni—O1 | 2.068 (1) | O1—C2 | 1.438 (2) |
| Ni—O2 | 2.067 (1) | O2—C4 | 1.433 (2) |
| Ni—N | 2.113 (1) | O3—C6 | 1.424 (3) |
| C1—N | 1.496 (2) | C1—C2 | 1.513 (2) |
| C3—N | 1.482 (2) | C3—C4 | 1.513 (3) |
| C5—N | 1.495 (2) | C5—C6 | 1.518 (2) |
| N—Ni—O1 | 83.64 (6) | C1—N—Ni | 107.6 (1) |
| N—Ni—O2 | 81.69 (6) | C3—N—Ni | 103.7 (1) |
| O1—Ni—O2 | 91.31 (6) | C5—N—Ni | 110.9 (1) |
| C2—O1—Ni | 106.8 (1) | C1—N—C5 | 111.2 (1) |
| C4—O2—Ni | 113.9 (1) | C3—N—C5 | 111.5 (1) |

Table 3. *Hydrogen-bonding geometry* (Å, °)

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—HO1$\cdots$Cl | 0.72 (4) | 2.34 (4) | 3.039 (2) | 164 (4) |
| O2—HO2$\cdots$Cl$^{\rm i}$ | 0.81 (4) | 2.23 (4) | 3.023 (2) | 167 (4) |
| O3—HO3$\cdots$Cl$^{\rm ii}$ | 0.71 (4) | 2.48 (4) | 3.166 (2) | 161 (4) |

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $1 - x, 2 - y, 2 - z$.

Data collection used $2\theta$ scan rates of 5.33 ($I_p < 150$) to 58.6° min$^{-1}$ ($I_p > 2500$), where $I_p$ is the prescan intensity. Scan widths were 2.4 to 2.8° ($2\theta$). Refinement was by full-matrix least squares. All non-H atoms were refined anisotropically. The initial positions of hydroxyl H atoms were determined by rigid-body refinement of O-centred 'methyl' groups, each with two dummy H atoms having zero site-occupancy factors, and then refined in the usual manner but with a common group $U_{\rm iso}$. Methylene H atoms were placed in calculated positions (C—H 0.95 Å) and refined riding on the C atom to which they were attached with a second common group $U_{\rm iso}$. All computations were performed on the SUN SPARCserver (UNIX operating system) of the Computing Centre of the University of Aberdeen.

Data reduction: *RDNIC* (Howie, 1980). Structure solution: *SHELXS*86 (Sheldrick, 1990). Structure refinement: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLOTAID* (Cradwick, 1980).

VTY and Hİ wish to thank The Scientific and Technical Research Council of Turkey (TUBITAK) for the financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Trichloro[tris(2-pyridyl)methanol]-rhodium(III)–Methanol (1/1)

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(*Received* 27 *January* 1995; *accepted* 28 *February* 1995)

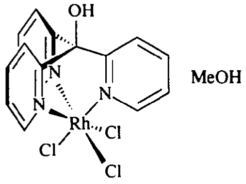
## Abstract

An X-ray diffraction study of [Rh{$(C_5H_4N)_3COH$}$Cl_3$].-$CH_3OH$ showed that the tris(2-pyridyl)methanol ligand coordinates to the Rh atom in a tridentate fashion, with the N atoms positioned at three corners of an octahedron

in a facial (*fac*) configuration. The remaining octahedral sites are occupied by the three $Cl^-$ ions. The coordination polyhedron is slightly distorted. The analysis of the structure revealed the presence of solvent molecules in the crystal. The methanol 'guest' is hydrogen bonded by the alcoholic hydroxy group of the organic ligand. Inspection of the intermolecular bonds showed relatively short distances between the methanol O atom and two of the $Cl^-$ ions. These O—H···Cl contacts may possibly be considered as attractive electrostatic interactions and are certainly important in the crystal packing.

## Comment

In recent years, ligands with $C_2$ symmetry have been employed extensively in catalytic asymmetric synthesis (Whitesell, 1989). While such ligands show beneficial properties in reactions where the selectivity is determined by an intermediate with square-planar geometry, $C_3$-symmetrical ligands should be preferred in cases where the selectivity-determining step involves an octahedral complex. In connection with studies of catalytic reactions involving chiral tripodal $C_3$-symmetric ligands (Adolfsson, Wärnmark & Moberg, 1992), it was of interest to study whether the threefold rotational axis in tris(2-pyridyl)methanol (tpmo) is maintained upon coordination to $Rh^{III}$. Thus, the complex [$Rh\{(C_5H_4N)_3COH\}Cl_3$] was prepared (Adolfsson & Moberg, 1995) and studied by X-ray diffraction. Here we report the results of the crystal structure analysis of the title compound, (I).



(I)

The coordination geometry around the $Rh^{III}$ ion may be described as a slightly distorted octahedral. The ligand acts as a tridentate donor, and the three coordinating N atoms are positioned in a facial (*fac*) configuration, while the $Cl^-$ ions occupy the remaining three octahedral positions (Fig. 1). A comparison of the Rh—Cl and Rh—N distances indicates that the donor atoms of each type have almost equivalent chemical surroundings (Table 2). The mean Rh—Cl and Rh—N distances in the present complex [2.355 (6) and 2.037 (7) Å, respectively, with the root mean square deviations given in parentheses] are comparable with those observed in related complexes of $Rh^{III}$ (Acharya, Tavale & Guru Row, 1984; Muir, Ma & Muir, 1989; Nishiyama *et al.*, 1989; Nishiyama, Kondo, Nakamura & Itoh, 1991; Sakaba, Kabuto, Horino & Arai, 1990; Lahuerta, Latorre, Martínez-Máñez, García-Granda & Gómez-Beltrán, 1991; Ma, Li, Muir & Muir, 1993). In the title complex, any two N atoms form a basal plane with two of the $Cl^-$ ions. The one formed by N1 and N1′ and the Cl1 and Cl3 ions, for example, is planar to within 0.095 (3) Å, and the $Rh^{III}$ ion is displaced by only −0.018 (1) Å from the plane, towards the Cl2 ion.

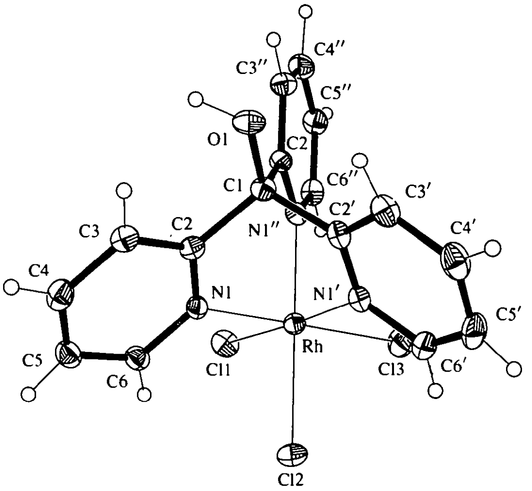


Fig. 1. Perspective molecular view of the title compound displaying displacement ellipsoids at the 50% probability level.

The ligand molecule possesses a near threefold axis of symmetry ($C_{3v}$). Each pyridine ring lies roughly in one of the vertical mirror planes (Fig. 1). The ring *C* [N1″–C6″] forms, for example, dihedral angles of 52.1 (1) and 61.7 (1)° with the pyridine rings *A* (N1–C6) and *B* (N1′–C6′), respectively. A comparison with the free ligand (tpmo) (Keene, Snow & Tiekink, 1988*a*), tpmoH.ClO$_4$ (Boggess, 1992) and related structures such as tris(2-pyridyl)methane (tpm) (Keene & Tiekink, 1990) and tris(2-pyridyl)phosphine (tpp) (Keene, Snow & Tiekink, 1988*b*) is given in Table 5.

In the free tpmo ligand, two of the N atoms point in the opposite direction to the alcohol function while the N atom in the third pyridine ring lies on the same side of the molecule as the COH group, probably due to a weak intramolecular hydrogen bond between the N atom and the alcoholic OH group (Keene *et al.*, 1988*a*). The pyridine rings in the free ligand are almost perpendicular to one another and are forced to adjust upon complexation to the $Rh^{III}$ ion (Table 5). The tpmo ligand coordinates readily to several transition metal ions, either through the three pyridine N atoms or through two N and one O atom. X-ray diffraction studies of complexes with tridentate N coordination (Table 5) have been reported by Canty, Chaichit, Gatehouse & George (1981) and by Szalda & Keene (1986), with methylmercury(II) and cobalt(III), respectively, as cations. Ruthenium(II) complexes with a mixed mode of coordination, through two N and one O ligand atoms, have also been reported (Keene, Szalda & Wilson, 1987; Moritz, Diamantis, Keene, Snow & Tiekink, 1988). The related ligand tpm ($py_3CH$) has been observed to act as a bidentate ligand for dimethylgold(III) (Canty, Minchin, Healy & White, 1982) and palladium(II) cations (Canty, Minchin, Skelton & White, 1992).

Table 2 lists selected torsion angles within the trispyridylmethanol unit. The C—O bond is almost coplanar with the pyridine rings. The mean deviations of the O—C—C—C and O—C—C—N torsion angles from 180° are 3 (2) and 2 (2)°, respectively. Upon coordination, three six-membered chelate rings, each with an approximate boat conformation, are formed. The ring puckering parameters calculated according to Cremer & Pople (1975) for the chelate rings are reported in Table 2.

The X-ray investigation revealed that in the crystallization process of the rhodium complex, one methanol molecule per complex was retained in the crystal structure. The methanol O1*M* atom was found to be hydrogen bonded by the alcoholic OH group of the organic ligand (Table 3). Further inspection of the intermolecular bonds showed relatively short distances between the methanol O atom and two of the $Cl^-$ ions. As seen in Fig. 2, H1O*M* points towards the $Cl^-$ ions of the neighbouring complex. The distances between O1*M* and the $Cl^-$ ions are actually longer than the sum of the van der Waals radii (3.2 Å; Pauling, 1960). However, because electrostatic interactions fall off much more slowly with distance than van der Waals interactions (Desiraju, 1991), the O1*M*—H1O*M*···Cl contacts (Table 3) may possibly be considered as attractive electrostatic interactions. These long-range attractive forces are certainly important in the crystal packing. Accordingly, besides the ordinary van der Waals forces, the crystal structure seems to be stabilized by electrostatic interactions (Table 4).

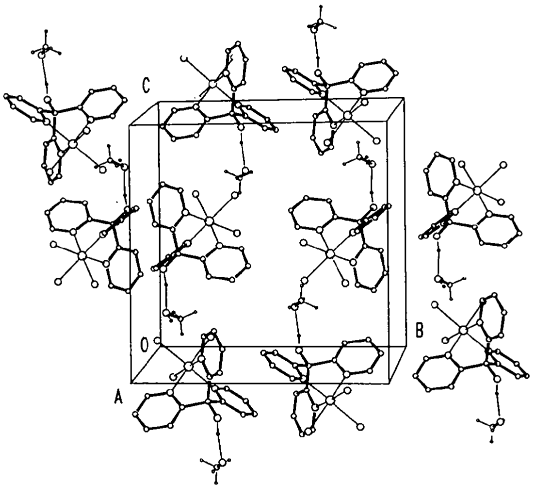


Fig. 2. Illustration of the crystal packing of the title compound (*PLUTO*; Motherwell & Clegg, 1978).

## Experimental

The title compound was prepared as described by Adolfsson & Moberg (1995) and crystallized from methanol.

*Crystal data*

$[RhCl_3(C_{16}H_{13}N_3O)].CH_3OH$
$M_r = 504.605$
Monoclinic
$P2_1/n$
$a = 8.3218 (5)$ Å
$b = 15.1916 (12)$ Å
$c = 14.9796 (9)$ Å
$\beta = 102.43 (1)°$
$V = 1849.39 (23)$ Å$^3$
$Z = 4$
$D_x = 1.8123 (2)$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 34 reflections
$\theta = 10.5–17.2°$
$\mu = 1.249$ mm$^{-1}$
$T = 153 (2)$ K
Plate
0.22 × 0.18 × 0.10 mm
Yellow

*Data collection*

Siemens Stoe AED-2 diffractometer
$\omega$–$2\theta$ scans
Absorption correction: numerical by integration from crystal shape (Norrestam, 1989)
$T_{min} = 0.797$, $T_{max} = 0.847$
8814 measured reflections
8105 independent reflections

3514 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.0367$
$\theta_{max} = 34.995°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 24$
$l = -24 \rightarrow 24$
5 standard reflections
frequency: 60 min
intensity decay: 2%

*Refinement*

Refinement on $F$
$R = 0.0301$
$wR = 0.0334$
3514 reflections
254 parameters
H atoms refined using a riding model
$w = 1/[\sigma^2(F) + 0.0002F^2]$

$(\Delta/\sigma)_{max} = 0.016$
$\Delta\rho_{max} = 0.240$ e Å$^{-3}$
$\Delta\rho_{min} = -0.104$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Rh | 0.06971 (3) | 0.23270 (2) | 0.07570 (2) | 0.0149 (1) |
| Cl1 | −0.1562 (1) | 0.1473 (1) | 0.0018 (1) | 0.0237 (3) |
| Cl2 | −0.0866 (1) | 0.3043 (1) | 0.1684 (1) | 0.0235 (3) |
| Cl3 | 0.1483 (1) | 0.1232 (1) | 0.1872 (1) | 0.0277 (3) |
| N1 | 0.0029 (4) | 0.3287 (2) | −0.0192 (2) | 0.0163 (8) |
| C2 | 0.1201 (4) | 0.3706 (2) | −0.0523 (2) | 0.0158 (9) |
| C3 | 0.0846 (4) | 0.4458 (2) | −0.1063 (2) | 0.0196 (10) |
| C4 | −0.0789 (5) | 0.4749 (2) | −0.1294 (2) | 0.0237 (11) |
| C5 | −0.2001 (5) | 0.4291 (2) | −0.0984 (2) | 0.0218 (10) |
| C6 | −0.1555 (4) | 0.3570 (2) | −0.0426 (2) | 0.0192 (10) |
| C1 | 0.2976 (4) | 0.3335 (2) | −0.0250 (2) | 0.0161 (9) |
| O1 | 0.4036 (3) | 0.3802 (2) | −0.0696 (2) | 0.0209 (8) |
| C2″ | 0.3048 (4) | 0.2337 (2) | −0.0448 (2) | 0.0169 (8) |
| C3″ | 0.4098 (4) | 0.2009 (2) | −0.0982 (2) | 0.0191 (10) |
| C4″ | 0.4181 (5) | 0.1094 (2) | −0.1105 (2) | 0.0239 (11) |
| C5″ | 0.3226 (5) | 0.0548 (2) | −0.0692 (2) | 0.0230 (11) |
| C6″ | 0.2202 (4) | 0.0907 (2) | −0.0165 (2) | 0.0202 (10) |
| N1″ | 0.2130 (3) | 0.1788 (2) | −0.0043 (2) | 0.0170 (8) |
| N1′ | 0.2697 (4) | 0.3073 (2) | 0.1335 (2) | 0.0181 (8) |
| C2′ | 0.3587 (4) | 0.3477 (2) | 0.0790 (2) | 0.0174 (9) |

| | | | | |
|---|---|---|---|---|
| C3′ | 0.4951 (4) | 0.4002 (2) | 0.1149 (2) | 0.0224 (11) |
| C4′ | 0.5363 (5) | 0.4148 (3) | 0.2087 (3) | 0.0275 (12) |
| C5′ | 0.4435 (5) | 0.3742 (2) | 0.2645 (2) | 0.0262 (11) |
| C6′ | 0.3124 (4) | 0.3211 (2) | 0.2258 (2) | 0.0224 (10) |
| C1*M* | 0.1248 (6) | 0.3465 (3) | −0.3006 (3) | 0.0352 (14) |
| O1*M* | 0.2812 (4) | 0.3847 (2) | −0.2558 (2) | 0.0278 (9) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Rh—Cl1 | 2.3557 (9) | Cl2—N1 | 3.081 (3) |
| Rh—Cl2 | 2.3625 (10) | Cl2—N1′ | 3.119 (3) |
| Rh—Cl3 | 2.3482 (9) | Cl3—N1″ | 3.145 (3) |
| Rh—N1 | 2.030 (3) | N1—N1″ | 2.851 (4) |
| Rh—N1″ | 2.035 (3) | N1″—N1′ | 2.807 (4) |
| Rh—N1′ | 2.046 (3) | Cl1—Cl2 | 3.411 (1) |
| Cl1—Cl3 | 3.353 (1) | C2—C1 | 1.551 (5) |
| Cl1—N1 | 3.103 (3) | C1—O1 | 1.407 (4) |
| Cl3—N1′ | 3.137 (3) | C1—C2″ | 1.549 (5) |
| N1—N1′ | 2.844 (4) | C1—C2′ | 1.546 (4) |
| Cl1—N1″ | 3.130 (3) | C1*M*—O1*M* | 1.451 (5) |
| Cl2—Cl3 | 3.352 (1) | | |
| N1″—Rh—N1′ | 86.9 (1) | Cl1—Rh—N1 | 89.8 (1) |
| N1—Rh—N1′ | 88.5 (1) | Cl1—Rh—Cl3 | 90.9 (1) |
| N1—Rh—N1″ | 89.1 (1) | Cl1—Rh—Cl2 | 92.6 (1) |
| Cl3—Rh—N1′ | 90.8 (1) | N1—C2—C1 | 117.1 (3) |
| Cl3—Rh—N1″ | 91.4 (1) | C2—C1—C2′ | 107.6 (3) |
| Cl3—Rh—N1 | 179.2 (1) | C2—C1—C2″ | 112.3 (3) |
| Cl2—Rh—N1′ | 89.8 (1) | C2—C1—O1 | 110.5 (3) |
| Cl2—Rh—N1″ | 176.1 (1) | C2″—C1—C2′ | 108.1 (3) |
| Cl2—Rh—N1 | 88.7 (1) | O1—C1—C2′ | 107.7 (3) |
| Cl2—Rh—Cl3 | 90.7 (1) | O1—C1—C2″ | 110.4 (3) |
| Cl1—Rh—N1′ | 177.0 (1) | C1—C2″—N1″ | 117.8 (3) |
| Cl1—Rh—N1″ | 90.6 (1) | C1—C2′—N1′ | 116.0 (3) |
| N1—C2—C1—O1 | 175.9 (3) | C2—C1—C2″—N1″ | −58.2 (4) |
| N1—C2—C1—C2″ | 52.2 (4) | C2″—C1—C2′—C3′ | 120.3 (4) |
| N1—C2—C1—C2′ | −66.7 (4) | O1—C1—C2′—C3′ | 1.0 (4) |
| C3—C2—C1—O1 | −6.0 (4) | C2″—C1—C2′—N1′ | −61.0 (4) |
| C3—C2—C1—C2″ | −129.8 (3) | O1—C1—C2′—N1′ | 179.7 (3) |
| C3—C2—C1—C2′ | 111.4 (4) | O1—C1—C2″—C3″ | 1.7 (4) |
| C2—C1—C2′—C3′ | −118.2 (4) | O1—C1—C2″—N1″ | 178.0 (3) |
| C2—C1—C2′—N1′ | 60.5 (4) | C2′—C1—C2″—N1″ | 60.4 (4) |
| C2—C1—C2″—C3″ | 125.5 (4) | C2′—C1—C2″—C3″ | −115.9 (4) |

| Planarity of pyridine rings<br>Ring | planar to within (Å) | Dihedral angles (°)<br>between pyridine rings | |
|---|---|---|---|
| *A* (unprimed) | 0.032 (3) | *A*—*B* | 113.3 (1) |
| *B* (primed) | 0.026 (4) | *A*—*C* | 52.1 (1) |
| *C* (double-primed) | 0.008 (3) | *B*—*C* | 61.7 (1) |

Puckering parameters (Å, °) for rings (1) N1—C2—C1—C2″—N1″—Rh, (2) N1′—C2′—C1—C2″—N1″—Rh and (3) N1—C2—C1—C2′—N1′—Rh

| | (1) | (2) | (3) |
|---|---|---|---|
| $q_2$ | 0.869 (3) | 0.932 (3) | 0.905 (3) |
| $q_3$ | −0.103 (3) | 0.084 (3) | 0.040 (3) |
| $\varphi_2$ | 123.2 (2) | −60.9 (2) | −62.9 (2) |
| Q | 0.875 (3) | 0.936 (3) | 0.906 (3) |
| $\theta_2$ | 96.8 (2) | 84.9 (2) | 87.5 (2) |

Table 3. *Hydrogen-bonding geometry* (Å, °)

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1O···O1*M* | 1.004 | 1.76 | 2.757 (4) | 170 |
| O1*M*—H1O*M*···Cl2$^{i}$ | 0.863 | 2.66 | 3.359 (3) | 140 |
| O1*M*—H1O*M*···Cl3$^{i}$ | 0.863 | 2.63 | 3.346 (3) | 142 |

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$.

Table 4. *Contact distances* (Å)

| | | | |
|---|---|---|---|
| Cl2···C1*M*$^{i}$ | 3.423 (5) | O1···C5$^{iii}$ | 3.497 (5) |
| Cl3···C4$^{ii}$ | 3.499 (4) | O1*M*···C3 | 3.183 (5) |
| Cl3···C5$^{ii}$ | 3.285 (4) | O1*M*···C4′$^{iv}$ | 3.410 (5) |

Symmetry codes: (i) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1+x, y, z$; (iv) $1-x, 1-y, -z$.

Table 5. *Comparison of selected conformational features of related tripodal ligand structures*

Dihedral angles (°) formed by the pyridine ring planes

| | | | | |
|---|---|---|---|---|
| Compound (1) | 113.3 (1) | 52.1 (1) | 61.7 (1) | Present paper |
| Tpmo free | 89.7 | 100.8 | 81.5 | Keene *et al.* (1988*a*) |
| TpmoH.ClO$_4$ | 103.4 | 74.6 | 29.9 | Boggess (1992) |
| Tpm | 103.6 | 106.8 | 88.4 | Keene & Tiekink (1990) |
| Tpp | 87.8 | 97.0 | 97.5 | Keene *et al.* (1988*b*) |

Mean deviations from 0 and 180° for selected torsion angles (°) and average metal–N(py) distances (Å)

| Compound | Coordination mode | O—C—C—C | O—C—C—N | Me—N |
|---|---|---|---|---|
| (*a*) | irregular, tetrahedral | 6 (4) | 5 (4) | 2.42 (10) |
| (*b*) | octahedral | 2 (2) | 1 (1) | 2.042 (6) |
| (*c*) | octahedral | 2 (2) | 3 (1) | 2.063 (4) |
| (*d*) | octahedral | 2 (1) | 3 (1) | 1.938 (6) |

Compounds: (*a*) [MeHg{(py)$_3$COH}]NO$_3$ (Canty *et al.*, 1981); (*b*) [Ru{(py)$_3$COH}{(py)$_3$CO}]Br.2C$_2$H$_5$OH (Keene *et al.*, 1987); (*c*) [Ru{(py)$_3$COH}$_2$](CH$_3$SO$_4$)$_2$ (Keene *et al.*, 1987); (*d*) Li[Co{(py)$_3$COH}$_2$](S$_2$O$_6$)$_2$.10H$_2$O (Szalda & Keene, 1986). [Source: Cambridge Structural Database (1994).]

The methyl H atoms were given geometrically assumed positions (C—H = 1.00 Å) which were recalculated after each refinement cycle. The methyl group was treated as rigid with free rotation around the O—C$_{\mathrm{methyl}}$ bond (Sheldrick, 1976).

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

AE is grateful to Docent Ingeborg Csöregh at the Stockholm University for valuable comments on the manuscript. Financial support from the Swedish Natural Science Research Council (NFR) is gratefully acknowledged.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data and torsion angles, have been deposited with the IUCr (Reference: AB1264). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). C**51**, 1765–1767

# Silver(I) *N*-Phthaloylglycinate

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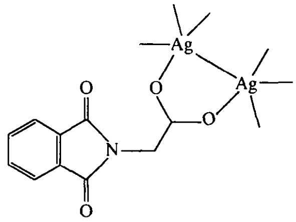
(*Received* 13 *May* 1994; *accepted* 17 *February* 1995)

## Abstract

The title compound, $Ag^+.C_{10}H_6NO_4^-$, was obtained by mixing *N*-phthaloylglycine and silver nitrate in aqueous solution. The structure is a three-dimensional network based on centrosymmetric eight-membered rings of dimeric [Ag-carboxylato-*O*,*O*′]$_2$ units. The Ag polyhedron is a trigonal bipyramid. Each Ag atom is linked to three carboxylic O atoms and one carbonyl O atom belonging to four *N*-phthaloylglycinate ligands [Ag—O = 2.225 (2), 2.228 (2), 2.541 (2), 2.612 (2) Å], and to another Ag atom [Ag—Ag = 2.8465 (2) Å]. This Ag—Ag interaction in the dimeric unit is short. One of the two carbonyl O atoms of the *N*-phthaloylglycinate ligand is not involved in the coordination polyhedron.

## Comment

This work extends our studies of silver coordination by carboxylic acids (Jaber, Charbonnier, Faure & Petit-Ramel, 1994). *N*-Phthaloylglycine is formed from the condensation reaction between glycine and phthalic acid. Two silver complexes with glycine (Gly) have been reported previously (Acland & Freeman, 1971). In both compounds, Ag(Gly) and $Ag(Gly)_2.0.5H_2O$, the Ag atom exhibits linear coordination. On the other hand, no silver complex is known with phthalic acid. *A priori*, the *N*-phthaloylglycinate ligand can potentially coordinate through one carboxylato group and two carbonyl moieties. Here, the crystal structure of silver(I) *N*-phthaloylglycinate, (I), is reported.



(I)

Fig. 1 shows an *ORTEP* (Johnson, 1965) plot of (I) showing the numbering system. The packing is shown as a stereoscopic view in Fig. 2. Each Ag atom has a trigonal bipyramidal environment. The Ag atom is bonded to three carboxylic O atoms belonging to three different ligands [Ag—O1 = 2.225 (2), Ag—O2$^{i}$ = 2.228 (2), Ag—O2$^{vi}$ = 2.612 (2) Å], to one carbonyl O atom belonging to a fourth ligand [Ag—O3$^{vii}$ = 2.541 (2) Å], and to one Ag atom [Ag—Ag$^{i}$ = 2.8465 (2) Å] [symmetry codes: (i) $-x$, $-y$, $1 - z$; (vi) $-x$, $1 - y$, $1 - z$; (vii) $x$, $1 - y$, $\frac{1}{2} + z$]. The O—Ag—O chain is not linear [O1—Ag—O2$^{i}$ = 160.4 (4)°]. The Ag—O bond lengths inside and outside the eight-membered ring and the connected angles are comparable with those found in the same structure types (Jaber, Charbonnier, Faure & Gebicki, 1994; Chen & Mak, 1991*a*,*b*; Huang, Lü, Chen & Mak, 1991; Smith, Sagatys, Campbell, Lynch & Kennard, 1990; Charbonnier, Faure, Petit-Ramel & Loiseleur, 1984; Hedrich & Hartl, 1983; Mak, Yip, Kennard, Smith & O'Reilly, 1988).

The carboxylic function of one *N*-phthaloylglycinate ligand is involved in three coordination bonds. These

*Acta Crystallographica Section C*
ISSN 0108-2701 ©1995