Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1367.
Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.
Keene, F. R., Snow, M. R. & Tiekink, E. R. T. (1988*a*). *Acta Cryst.* C**44**, 937–938.
Keene, F. R., Snow, M. R. & Tiekink, E. R. T. (1988*b*). *Acta Cryst.* C**44**, 757–758.
Keene, F. R., Szalda, D. J. & Wilson, T. A. (1987). *Inorg. Chem.* **26**, 2211–2216.
Keene, F. R. & Tiekink, E. R. T. (1990). *Acta Cryst.* C**46**, 1562–1563.
Lahuerta, P., Latorre, J., Martínez-Máñez, R., García-Granda, S. & Gómez-Beltrán, F. (1991). *Acta Cryst.* C**47**, 519–522.
Ma, R., Li, Y.-J, Muir, J. A. & Muir, M. M. (1993). *Acta Cryst.* C**49**, 89–91.
Moritz, P. S., Diamantis, A. A., Keene, F. R., Snow, M. R. & Tiekink, E. R. T. (1988). *Aust. J. Chem.* **41**, 1353–1362.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.
Muir, M. M., Ma, R. & Muir, J. A. (1989). *Acta Cryst.* C**45**, 1634–1635.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Nishiyama, H., Kondo, M., Nakamura, T. & Itoh, K. (1991). *Organometallics*, **10**, 500–508.
Nishiyama, H., Sakaguchi, H., Nakamura, T., Horihata, M., Kondo, M. & Itoh, K. (1989). *Organometallics*, **8**, 846–848.
Norrestam, R. (1989). *STOEABS. Program for Absorption Correction of STOE Data*. Stockholm Univ., Sweden.
Pauling, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.
Sakaba, H., Kabuto, C., Horino, H. & Arai, M. (1990). *Bull. Chem. Soc. Jpn*, **63**, 1822–1824.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Stoe & Cie (1988*a*). *DIF4. Diffractometer Control Program*. Version 7. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*b*). *REDU4. Data Reduction Program*. Version 7. Stoe & Cie, Darmstadt, Germany.
Szalda, D. J. & Keene, F. R. (1986). *Inorg. Chem.* **25**, 2795–2799.
Whitesell, J. K. (1989). *Chem. Rev.* **89**, 1581–1590.

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Silver(I) *N*-Phthaloylglycinate

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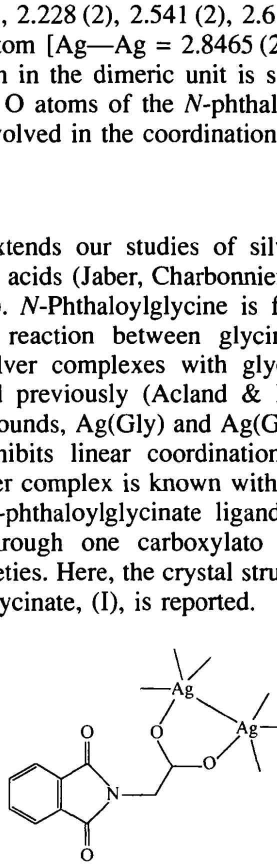
Abstract

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$_2$O, 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

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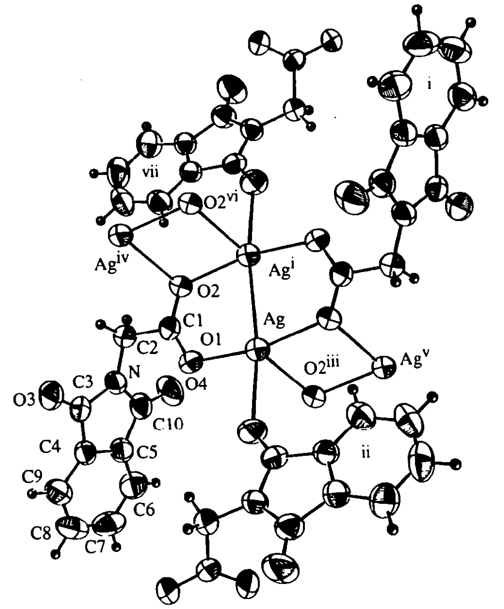

Fig. 1. Silver coordination around the eight-membered ring [Ag-carboxylato-*O*,*O*′]$_2$ with 74% probability displacement ellipsoids. The H atoms are scaled arbitrarily. Symmetry codes: (i) $-x, -y, 1-z$; (ii) $-x, y-1, \frac{1}{2}-z$; (iii) $x, y-1, z$; (iv) $x, 1+y, z$; (v) $-x, -1-y, 1-z$; (vi) $-x, 1-y, 1-z$; (vii) $x, 1-y, \frac{1}{2}+z$.

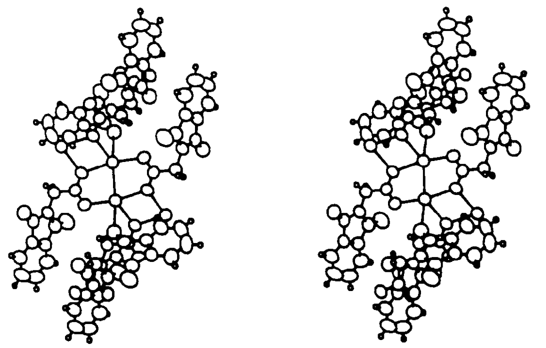

Fig. 2. *ORTEP* (Johnson, 1965) stereoscopic view of the title compound.

bonds are part of the dimeric unit [Ag-carboxylato-*O*,*O*′]$_2$, as well as forming the two adjacent four-membered $(AgO)_2$ rings, 'fused' on both sides of the eight-membered ring (see Fig. 1). This structure type, possessing alternating eight- and four-membered rings, is known for other silver(I) carboxylates (Jaber, Charbonnier, Faure & Gebicki, 1994; Mak *et al.*, 1988; Chen & Mak, 1991*a*,*b*; Huang *et al.*, 1991; Smith *et al.*, 1990; Charbonnier *et al.*, 1984; Hedrich & Hartl, 1983). Both the eight- and four-membered rings are centrosymmetric, as found in some related crystals (Chen & Mak, 1991*a*,*b*; Huang *et al.*, 1991; Smith *et al.*, 1990).

The *N*-phthaloylglycinate ligand presents two donor-type functional groups: the carboxylic and carbonyl groups. In the complex, the two O atoms of the carboxylato group act in different ways: O1 forms only one coordination bond, whereas O2 forms two. In the same way, only one of the two carbonyl O atoms plays a role in the coordination. This can be explained by the steric hindrance caused by the large volume occupied by the ligand. The short Ag—Ag separation is comparable with those found in other dimeric silver(I) carboxylates: 2.892 (1) (Jaber, Charbonnier, Faure & Gebicki, 1994), 2.809 (1) (Mak *et al.*, 1988), 2.898 (1) and 2.814 (2) (Chen & Mak, 1991*a*), 2.881 (2) (Charbonnier *et al.*, 1984), 2.866 (2) (Smith *et al.*, 1990), 2.855 (1) (Movsumov, Antsyshkina, Ostrikova, Karaevea & Porai-Koshits, 1990), 2.800 (2), 2.854 (1) and 2.901 (2) (Chen & Mak, 1991*b*), 2.861 (1) (Smith, Kennard & Mak, 1988), 2.856 (2) and 2.928 (1) (Huang *et al.*, 1991), 2.866 (2) and 2.836 (2) Å (Smith *et al.*, 1990).

The crystal is structure is polymeric; a three-dimensional network is formed by the four- and eight-membered centrosymmetric rings. All the four-membered rings are parallel, as are all the eight-membered rings. The dihedral angle between an eight-membered ring and a four-membered ring is 59.0 (1)°.

Experimental

A $0.05\ \text{mol l}^{-1}$ aqueous solution of *N*-phthaloylglycine (200 ml) was adjusted to pH 3. On addition of 0.01 mol of solid silver nitrate, a white precipitate was obtained; this was isolated by filtration and carefully washed. This precipitate was redissolved in hot water and colourless transparent crystals were formed by slow evaporation in the dark.

Crystal data

$Ag^+.C_{10}H_6NO_4^-$
$M_r = 312.0$
Monoclinic
$C2/c$
$a = 27.571\ (4)$ Å
$b = 5.062\ (1)$ Å
$c = 17.284\ (3)$ Å
$\beta = 127.63\ (1)°$
$V = 1910\ (1)$ Å^3
$Z = 8$
$D_x = 2.17$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 11.6$–$23.1°$
$\mu = 2.08$ mm^{-1}
$T = 291$ K
Needle
0.5 × 0.1 × 0.05 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–$4/3\theta$ scans
Absorption correction:
ψ scan
$T_{\min} = 0.852$, $T_{\max} = 1.000$
4721 measured reflections
2401 independent reflections

1839 observed reflections [$I > 3\sigma(I)$]
$R_{\text{int}} = 0.028$
$\theta_{\max} = 30°$
$h = -38 \rightarrow 38$
$k = -7 \rightarrow 7$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.017$
$wR = 0.019$
$S = 1.48$
1839 reflections
164 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}
Extinction correction: none
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)

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ag	−0.01553 (1)	−0.18509 (4)	0.42605 (1)	2.821 (4)
O1	0.04167 (6)	0.0631 (3)	0.4039 (1)	2.95 (4)
O2	0.04948 (6)	0.3831 (3)	0.50013 (9)	2.75 (4)
O3	0.05855 (6)	0.5620 (4)	0.2307 (1)	3.04 (4)
O4	0.18642 (7)	0.0546 (4)	0.5006 (1)	4.00 (5)
N	0.11646 (7)	0.3336 (4)	0.3759 (1)	2.37 (4)
C1	0.05892 (8)	0.2835 (4)	0.4437 (1)	2.18 (5)
C2	0.09540 (9)	0.4623 (5)	0.4251 (1)	2.60 (5)
C3	0.09530 (8)	0.3911 (4)	0.2811 (1)	2.20 (5)
C4	0.12825 (8)	0.2072 (5)	0.2609 (1)	2.23 (4)
C5	0.16705 (8)	0.0525 (5)	0.3428 (1)	2.40 (5)
C6	0.20392 (9)	−0.1377 (6)	0.3457 (2)	3.26 (6)
C7	0.20019 (9)	−0.1682 (6)	0.2623 (2)	3.64 (6)
C8	0.16195 (9)	−0.0122 (6)	0.1807 (2)	3.43 (6)
C9	0.12515 (9)	0.1808 (6)	0.1781 (1)	2.86 (5)
C10	0.16010 (9)	0.1338 (5)	0.4179 (1)	2.61 (5)

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

Ag—Ag^{i}	2.8465 (2)	N—C10	1.389 (3)
Ag—O1	2.225 (2)	C1—C2	1.525 (4)
Ag—O2^{i}	2.228 (2)	C3—C4	1.484 (4)
Ag—O3^{ii}	2.541 (2)	C4—C5	1.382 (3)
Ag—O2^{iii}	2.612 (2)	C4—C9	1.386 (4)
O1—C1	1.242 (3)	C5—C6	1.378 (4)
O2—C1	1.259 (3)	C5—C10	1.481 (4)
O3—C3	1.207 (2)	C6—C7	1.388 (5)
O4—C10	1.210 (3)	C7—C8	1.381 (3)
N—C2	1.445 (4)	C8—C9	1.389 (4)
N—C3	1.394 (3)		
O1—Ag—Ag^{i}	85.8 (5)	O1—Ag—O2^{i}	160.4 (4)
Ag—O1—C1	118.9 (2)	C3—C4—C9	130.0 (2)
C2—N—C3	124.6 (2)	C5—C4—C9	121.5 (2)
C2—N—C10	123.5 (2)	C4—C5—C6	121.7 (3)
C3—N—C10	111.9 (2)	C4—C5—C10	107.8 (2)
O1—C1—O2	126.7 (3)	C6—C5—C10	130.5 (2)
O1—C1—C2	119.7 (2)	C5—C6—C7	117.0 (2)
O2—C1—C2	113.6 (2)	C6—C7—C8	121.5 (3)
N—C2—C1	114.2 (2)	C7—C8—C9	121.5 (3)
O3—C3—N	124.3 (3)	C4—C9—C8	116.8 (2)
O3—C3—C4	130.2 (2)	O4—C10—N	124.2 (3)
N—C3—C4	105.6 (2)	O4—C10—C5	129.6 (2)
C3—C4—C5	108.5 (2)	N—C10—C5	106.2 (2)

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

Calculations were performed using *SDP* (B. A. Frenz & Associates Inc., 1982) on a DEC MicroVAX 3100-80 computer at the Centre de Diffractométrie Automatique, Université Lyon I.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: PA1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Acland, C. B. & Freeman, H. C. (1971). *Chem. Commun.* pp. 1016–1017.

B. A. Frenz & Associates Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, USA.

Charbonnier, F., Faure, R., Petit-Ramel, M. & Loiseleur, H. (1984). *Rev. Chim. Minér.* **21**, 601–610.

Chen, X. M. & Mak, T. C. W. (1991*a*). *J. Chem. Soc. Dalton Trans.* pp. 1219–1222.

Chen, X. M. & Mak, T. C. W. (1991*b*). *J. Chem. Soc. Dalton Trans.* pp. 3253–3258.

Hedrich, V. M. & Hartl, H. (1983). *Acta Cryst.* **C39**, 533–536.

Huang, W. Y., Lü, L., Chen, X. M. & Mak, T. C. W. (1991). *Polyhedron*, **10**, 2687–2691.

Jaber, F., Charbonnier, F., Faure, R. & Gebicki, K. (1994). *Acta Cryst.* **C50**, 1444–1447.

Jaber, F., Charbonnier, F., Faure, R. & Petit-Ramel, M. (1994). *Z. Kristallogr.* **200**, 536–538.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Mak, T. C. W., Yip, W. H., Kennard, C. H. L., Smith, G. & O'Reilly, E. J. (1988). *J. Chem. Soc. Dalton Trans.* pp. 2353–2356.

Movsumov, E. M., Antsyshkina, A. S., Ostrikova, V. N., Karaeva, K. T. & Porai-Koshits, M. A. (1990). *Sov. J. Coord. Chem.* **16**, 281–284.

Smith, G., Kennard, C. H. L. & Mak, T. C. W. (1988). *Z. Kristallogr.* **184**, 275–280.

Smith, G., Sagatys, D. S., Campbell, C. A., Lynch, D. E. & Kennard, C. H. L. (1990). *Aust. J. Chem.* **43**, 1707–1712.

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Mono(aminocarboxylato)cobalt(III) Complexes Possessing Only Vicinal and Conformational Asymmetry. IV. Silver(I) *mer*-(NO_2),*trans*-(NH_2,NH_3)-[(*S*)-alaninato]-amminetrinitrocobaltate(III) Monohydrate

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Abstract

The structure of $Ag[Co(C_3H_6NO_2)(NO_2)_3(NH_3)].H_2O$ consists of silver cations, complex anions and molecules of water. The five-membered chelate ring adopts a very flat strained λ-envelope conformation with a methyl side

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