GEREMIA, S. (1988). Thesis, Univ. of Trieste, Italy.
HALPERN, J. (1985). *Science*, **227**, 869–875.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor: Kluwer Academic Publishers, Dordrecht.)
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
MARZILLI, L. G., BAYO, F., SUMMERS, M. F., THOMAS, L. B., ZANGRANDO, E., BRESCIANI PAHOR, N., MARI, M. & RANDACCIO, L. (1987). *J. Am. Chem. Soc.* **109**, 6045–6052.
PARKER, W. O., ZANGRANDO, E., BRESCIANI PAHOR, N., MARZILLI, P. A., RANDACCIO, L. & MARZILLI, L. G. (1988). *Inorg. Chem.* **27**, 2170–2180.
PATTABHI, V., NETHAJI, M., GABE, E. J., LEE, F. L. & LE PAGE, Y. (1984). *Acta Cryst.* C**40**, 1155–1156.
UKIDA, A., SASADA, Y. & OHASHI, Y. (1988). *Acta Cryst.* B**44**, 249–254.
ZANGRANDO, E., BRESCIANI PAHOR, N., RANDACCIO, L., CHARLAND, J. P. & MARZILLI, L. G. (1986). *Organometallics*, **5**, 1938–1944.

Acta Cryst. (1989). C**45**, 566–568

Structure of [*N*,*N*-Bis(carboxymethyl)glycinato](1,3-propanediamine)cobalt(III) Monohydrate

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Abstract. $[Co(C_6H_6NO_6)(C_3H_{10}N_2)].H_2O$, $M_r = 338.90$, orthorhombic, $Pna2_1$, $a = 10.457$ (2), $b = 17.141$ (4), $c = 7.233$ (2) Å, $V = 1296.39$ Å^3, $Z = 4$, $D_x = 1.737$, D_m(by flotation) = 1.730 g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 12.83$ cm^{-1}, $F(000) = 692$, $T = 295$ K, $R = 0.0254$ for 1156 observed reflections. The two N atoms of the 1,3-propanediamine (pd) ligand, the N atom and three carboxyl O atoms of the *N*,*N*-bis(carboxymethyl)glycinato (nta) ligand give distorted octahedral coordination around Co. The Co—O(6) bond distance is shorter than the other axial Co—O(4) bond distance. The amine groups and the water molecule are involved in hydrogen bonding.

Introduction. In continuation of research on metal complexes involving tripod ligands (Swaminathan, Sinha, Chatterjee, Patel & Padmanabhan, 1988; Swaminathan, Sinha, Chatterjee, Yadava & Padmanabhan, 1989), the structural investigation of the title compound was undertaken to establish the details of coordination.

Experimental. The complex was synthesized by a similar method to that reported in the literature (Chatterjee, Singh, Phulambrikar & Das, 1988):

$$CoCO_3 + H_3nta + pd \xrightarrow{333\ K} [Co(pd)(nta)].H_2O.$$

Recrystallized from hot water, washed with water and ethanol and finally air-dried. Intensity data were first collected on an indigenously fabricated four-circle diffractometer at BARC, Bombay. As the number of reflections with $|F_o| > 5\sigma(|F_o|)$ was inadequate to locate the H atoms, data were again collected (crystal dimensions $0.35 \times 0.13 \times 0.15$ mm) on an Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scan. Lattice parameters obtained by photographs were refined by least-squares analysis using 22 reflections, $7 < \theta < 11°$. Intensities of two standard reflections recorded for every 1800 s showed no significant changes. Range $2 < \theta < 25°$ (resolution $d = 0.84$ Å, $h_{max} = 12$, $k_{max} = 20$, $l_{max} = 8$). 1362 unique reflections were collected of which 1164 reflections had $|F_o| > 3\sigma(|F_o|)$. Lp corrections, no absorption correction. Structure was solved by Patterson method, *SHELXS*86 (Sheldrick,

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Table 1. *Positional parameters* ($\times 10^4$, *for* Co $\times 10^5$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10^3$) *for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{\rm eq} = \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Co	18435 (5)	2514 (3)	0	20 (<1)
N(1)	2934 (3)	1176 (2)	262 (7)	21 (3)
N(2)	599 (3)	−586 (2)	−118 (8)	26 (3)
N(3)	3152 (3)	−476 (2)	−837 (6)	27 (3)
O(1)	−99 (4)	2131 (2)	1106 (8)	65 (5)
O(2)	462 (3)	895 (2)	808 (5)	31 (3)
O(3)	4104 (3)	152 (2)	−5739 (5)	42 (3)
O(4)	2361 (3)	45 (2)	2539 (5)	27 (3)
O(5)	2247 (3)	1435 (2)	−4512 (5)	40 (4)
O(6)	1566 (3)	570 (2)	−2420 (5)	26 (3)
O*W*	2509 (4)	−1203 (2)	−4524 (6)	51 (4)
C(1)	899 (4)	−1341 (3)	769 (8)	33 (4)
C(2)	2060 (4)	−1716 (2)	−38 (13)	41 (4)
C(3)	3273 (4)	−1245 (2)	100 (12)	38 (4)
C(4)	2109 (4)	1829 (2)	967 (7)	29 (4)
C(5)	702 (4)	1625 (3)	932 (8)	35 (5)
C(6)	3947 (4)	979 (3)	1623 (7)	30 (4)
C(7)	3452 (4)	363 (3)	2931 (7)	27 (4)
C(8)	3385 (5)	1342 (3)	−1639 (7)	31 (5)
C(9)	2326 (4)	1117 (3)	−3000 (7)	29 (4)

Table 2. *Important bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

N(1)—Co	1·962 (3)	C(5)—O(1)	1·213 (6)
N(2)—Co	1·940 (3)	C(5)—O(2)	1·279 (5)
N(3)—Co	1·948 (4)	C(7)—O(3)	1·233 (6)
O(2)—Co	1·910 (3)	C(7)—O(4)	1·295 (5)
O(4)—Co	1·947 (3)	C(9)—O(5)	1·225 (6)
O(6)—Co	1·857 (3)	C(9)—O(6)	1·299 (5)
C(4)—N(1)	1·503 (6)	C(2)—C(1)	1·493 (7)
C(6)—N(1)	1·485 (6)	C(3)—C(2)	1·507 (6)
C(8)—N(1)	1·481 (7)	C(5)—C(4)	1·512 (7)
C(1)—N(2)	1·477 (6)	C(7)—C(6)	1·509 (7)
C(3)—N(3)	1·487 (7)	C(9)—C(8)	1·531 (7)

N(3)—Co—N(2)	89·0 (1)	C(7)—O(4)—Co	112·0 (3)
O(2)—Co—N(1)	86·8 (1)	C(9)—O(6)—Co	114·9 (3)
O(4)—Co—N(1)	83·9 (2)	C(2)—C(1)—N(2)	112·3 (4)
O(6)—Co—N(1)	86·8 (2)	C(3)—C(2)—C(1)	115·3 (4)
O(6)—Co—O(4)	170·5 (1)	C(2)—C(3)—N(3)	111·9 (4)
C(4)—N(1)—Co	107·5 (2)	C(5)—C(4)—N(1)	112·3 (3)
C(6)—N(1)—Co	107·1 (3)	C(4)—C(5)—O(2)	114·8 (4)
C(8)—N(1)—Co	104·5 (3)	C(7)—C(6)—N(1)	109·3 (4)
C(1)—N(2)—Co	119·1 (3)	C(6)—C(7)—O(4)	117·3 (4)
C(3)—N(3)—Co	119·1 (3)	C(9)—C(8)—N(1)	108·6 (4)
C(5)—O(2)—Co	115·9 (3)	C(8)—C(9)—O(6)	114·6 (4)

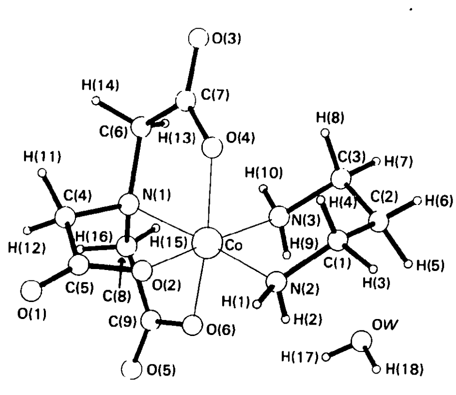

Fig. 1. General view of the molecule.

Table 3. *Hydrogen-bond distances with e.s.d.'s* ≃ 0·008 Å

$X\cdots$O		Acceptor code
N(2)···O(6)	2·989	$-x, -y, \frac{1}{2}+z$
N(3)···O(3)	2·924	$-x+1, -y, \frac{1}{2}+z$
O*W*···O(3)	2·991	x, y, z
O*W*···O(4)	3·019	x, y, z
O*W*···O(1)	3·015	$-x, -y, \frac{1}{2}+z$

1986). H atoms were located from difference Fourier maps. Structure refinement by *SHELX76* (Sheldrick, 1976), using least squares based on F values with anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. In the final calculation reflections with $|F_o| < 5\sigma(|F_o|)$ and those for which $F_o < F_c$ with $(F_o - F_c)/\sigma(|F_o|) > 4{\cdot}0$ were omitted. Number of parameters refined: 252. $(\Delta/\sigma)_{\max} = 0{\cdot}364$ and $\Delta\rho$ in the final difference Fourier map −0·09 to 0·17 e Å^{-3}. For 1156 reflections $R = 0{\cdot}0254$, unit weights. CYBER-180 computer was used for all the calculations. Atomic scattering factors those of *SHELX76*, for Co atom from *International Tables for X-ray Crystallography* (1974).

Discussion. Positional parameters and equivalent isotropic temperature factors for non-H atoms are given in Table 1 and some important bond lengths and bond angles in Table 2.* The *PLUTO* diagram (Motherwell & Clegg, 1978) of the molecule is given in Fig. 1. The Co atom is six-coordinated with a distorted octahedral geometry. The equatorial plane consists of two N atoms of the pd ligand, the N atom and one O atom of one of the glycinato arms of the nta ligand while the axial positions are occupied by O atoms of the other two glycinato arms. The maximum deviation from the equatorial plane is 0·0093 (8) Å. The pd ring is found to have the chair conformation. The angle between the equatorial plane and that of atoms N(2), C(1), C(3), N(3) is 41·7 (4)°. The Co—N(2) and Co—N(3) bond lengths are nearly equal [1·940 (3), 1·948 (4) Å] but less than Co—N(1) [1·962 (3) Å]. This is explained by the depletion of electron density on N(1) as it is bonded to electron-withdrawing glycinato groups making the Co—N(1) bond weaker. The N—C distances of the nta ligand are nearly equal. It has previously been observed (Weakliem & Hoard, 1959; Okazaki, Tomioka & Yoneda, 1983) that the *G* ring (the glycinato ring coplanar with the equatorial plane) is more strained than the *R* rings (outer glycinato rings). In contrast, considering the deviations of the constituent atoms of these rings as a measure of ring strain, the strain can be

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and further hydrogen-bond details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51547 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

represented to follow the order $G < R_2 < R_1$, where R_1 and R_2 are the glycinato rings coordinated to Co through O(4) and O(6) respectively. This strain makes the Co—O(4) bond length [1·947 (3) Å] longer than Co—O(6) [1·857 (3) Å]. The possible hydrogen bonds are given in Table 3.

References

CHATTERJEE, C., SINGH, R. S., PHULAMBRIKAR, A. & DAS, S. (1988). *J. Chem. Soc. Dalton Trans.* pp. 159–162.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

OKAZAKI, H., TOMIOKA, K. & YONEDA, H. (1983). *Inorg. Chim. Acta*, **74**, 169–178.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS86. Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

SWAMINATHAN, K., SINHA, U. C., CHATTERJEE, C., PATEL, R. P. & PADMANABHAN, V. M. (1988). *Acta Cryst.* C**44**, 447–449.

SWAMINATHAN, K., SINHA, U. C., CHATTERJEE, C., YADAVA, V. S. & PADMANABHAN, V. M. (1989). *Acta Cryst.* C**45**, 21–23.

WEAKLIEM, H. A. & HOARD, J. L. (1959). *J. Am. Chem. Soc.* **81**, 549–555.

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Structure of Triphenyltin(IV) 2-Thiophenecarboxylate

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Abstract. [(C_6H_5)$_3$SnOC(O)C_4H_3S], $M_r = 477\cdot15$, monoclinic, $P2_1/n$, $a = 14\cdot237$ (3), $b = 11\cdot785$ (2), $c = 13\cdot448$ (2) Å, $\beta = 116\cdot65$ (1)°, $V = 2017$ (1) Å^3, $Z = 4$, $D_x = 1\cdot571$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 13\cdot8$ cm^{-1}, $F(000) = 952$, $T = 295$ (1) K, $R = 0\cdot041$ for 2617 [$I > 3\sigma(I)$] reflections. The Sn is four-coordinate and the tetrahedral geometry is distorted owing to an intramolecular tin–carbonyl oxygen contact of 2·768 (4) Å.

Introduction. Triorganotin(IV) alkanoates are generally polymeric because of carboxylate bridging which results in the *trans*-C_3SnO_2 trigonal bipyramidal geometry at tin (Davies & Smith, 1982), especially in the absence of other donor atoms in the carboxylate unit (Ng, Chen Wei & Kumar Das, 1988). The triphenyltin arylcarboxylates are a fairly consistent series of four-coordinate tin structures (Harrison, Lambert, King & Majee, 1983; Holmes, Day, Chandrasekhar, Vollano & Holmes, 1986*a*; Swisher, Vollano, Chandrasekhar, Day & Holmes, 1984; Vollano, Day, Rau, Chandrasekhar & Holmes, 1984) whose distortion from idealized geometry arises from the short intramolecular acyl oxygen–tin (2·463–2·861 Å) distances which, although less than the sum of the van der Waals radii (3·70 Å) of tin and oxygen, are nevertheless longer than the covalent Sn—O bonds (2·048–2·115 Å). On the basis of infrared and tin-119*m* Mössbauer data, the heterocyclic analogues, triphenyltin 2-thiophene- and 2-furan- (Sandhu & Verma, 1986) carboxylates are reported to be four-coordinate; the report prompted the present crystal study of triphenyltin 2-thiophenecarboxylate.

Experimental. Crystals of triphenyltin 2-thiophenecarboxylate were grown from an alcoholic solution of triphenyltin hydroxide and 2-thiophenecarboxylic acid. A crystal measuring 0·32 × 0·15 × 0·16 mm was mounted on an Enraf–Nonius CAD-4 diffractometer for the X-ray data collection. 25 reflections (15° < 2θ < 18°) were used to determine the cell constants. Lorentz–polarization, linear decay based on repeatedly measuring three independent reflections (from 0·987 to 1·000 on I), reflection averaging (agreement on I 1·8%) and empirical absorption (from 7·21% to 0·08% on I) corrections were applied. The maximum 2θ was 50·0° with the *hkl* ranges being h −16 to 15, k 0 to 13 and l 0 to 15. Of the 3887 total reflections, 3721 were unique

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