Acta Cryst. (1988). C**44**, 2199–2201

Structure of Tris(glycinato)cobalt(III) Dihydrate

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(*Received* 16 *May* 1988; *accepted* 19 *July* 1988)

Abstract. $[Co(C_2H_4NO_2)_3].2H_2O$, $M_r = 317{\cdot}14$, monoclinic, $P2_1/n$, $a = 13{\cdot}860$ (3), $b = 13{\cdot}153$ (3), $c = 13{\cdot}996$ (3) Å, $\beta = 112{\cdot}88$ (1)°, $V = 2351$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}792$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 15{\cdot}61$ cm^{-1}, $F(000) = 1312$, $T = 295$ K, final $R = 0{\cdot}044$ for 2691 unique observed reflections with $I_o > 2\sigma(I_o)$. The structure determination was undertaken to characterize the compound and to establish its atom connectivity and stereochemistry. There are two complex molecules, which are geometric isomers, and four water molecules of crystallization per asymmetric unit. The Co–O distances average 1·894 Å and range from 1·876 (3) to 1·911 (4) Å. For Co–N these values are 1·931, 1·926 (4) and 1·936 (4) Å. Possible hydrogen-bonding interactions (<3·7 Å) listing minimum, maximum and average distances are as follows: O(water)···O(water) 2·903 (6); O(ligand)···O(water) 2·719 (6), 3·626 (6), 3·198; N(ligand)···O(water) 2·943 (6), 3·645 (6), 3·148; N(ligand)···O(ligand) 2·922 (6), 3·560 (5), 3·110 Å.

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Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = \frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}]$.

	x	*y*	*z*	B_{eq}
Co(1)	0·18691 (5)	0·74428 (6)	0·70770 (5)	1·42 (3)
N(11)	0·0432 (3)	0·7211 (3)	0·6877 (3)	1·8 (2)
C(11)	0·0203 (5)	0·6117 (5)	0·6762 (5)	3·5 (3)
C(12)	0·1111 (4)	0·5516 (4)	0·6718 (4)	1·8 (2)
O(11)	0·1942 (3)	0·6021 (3)	0·6878 (3)	2·0 (2)
O(12)	0·1017 (3)	0·4596 (3)	0·6549 (3)	2·7 (2)
N(21)	0·1745 (3)	0·8899 (3)	0·7189 (3)	1·8 (2)
C(21)	0·1689 (5)	0·9383 (4)	0·6208 (4)	2·6 (2)
C(22)	0·1464 (4)	0·8599 (4)	0·5361 (4)	2·0 (2)
O(21)	0·1484 (3)	0·7667 (3)	0·5637 (2)	2·0 (1)
O(22)	0·1299 (3)	0·8866 (3)	0·4469 (3)	3·0 (2)
N(31)	0·3344 (3)	0·7538 (3)	0·7339 (3)	1·8 (2)
C(31)	0·3950 (4)	0·7181 (4)	0·8409 (4)	2·1 (2)
C(32)	0·3270 (4)	0·7182 (4)	0·9033 (4)	1·9 (2)
O(31)	0·2279 (3)	0·7264 (3)	0·8511 (2)	2·0 (1)
O(32)	0·3661 (3)	0·7089 (3)	0·9979 (3)	3·5 (2)
Co(2)	0·80601 (5)	0·25236 (6)	0·81088 (4)	1·52 (3)
N(41)	0·7783 (3)	0·2500 (4)	0·6647 (3)	2·2 (2)
C(41)	0·6693 (4)	0·2843 (5)	0·6062 (4)	2·4 (2)
C(42)	0·6078 (4)	0·2792 (4)	0·6736 (4)	2·1 (2)
O(41)	0·6601 (3)	0·2638 (3)	0·7715 (2)	2·1 (1)
O(42)	0·5125 (3)	0·2916 (3)	0·6364 (3)	3·1 (2)
N(51)	0·7976 (4)	0·1066 (4)	0·8212 (4)	2·2 (2)
C(51)	0·9025 (5)	0·0611 (5)	0·8451 (5)	3·2 (3)
C(52)	0·9829 (4)	0·1415 (4)	0·8547 (4)	2·1 (2)
O(51)	0·9514 (3)	0·2337 (3)	0·8453 (3)	2·1 (1)
O(52)	1·0736 (3)	0·1156 (3)	0·8709 (3)	2·9 (2)
N(61)	0·8277 (3)	0·2730 (3)	0·9545 (3)	1·8 (2)
C(61)	0·8603 (5)	0·3779 (4)	0·9835 (4)	2·7 (2)
C(62)	0·8382 (4)	0·4446 (4)	0·8895 (4)	1·9 (2)
O(61)	0·8175 (3)	0·3967 (3)	0·8042 (3)	2·2 (2)
O(62)	0·8434 (3)	0·5377 (3)	0·8967 (3)	3·1 (2)
O(71)	1·2184 (4)	−0·0399 (3)	0·9846 (3)	4·4 (2)
O(72)	0·1103 (3)	0·6029 (3)	0·4247 (3)	3·2 (2)
O(73)	0·0478 (3)	1·0589 (4)	0·3385 (4)	4·5 (2)
O(74)	0·9055 (4)	0·5950 (3)	1·1102 (4)	4·2 (2)

Table 2. *Bond lengths* (Å) *and angles* (°) *involving the non-*H *atoms*

Co(1)–O(31)	1·876 (3)	Co(2)–O(41)	1·885 (3)
Co(1)–O(21)	1·896 (3)	Co(2)–O(51)	1·897 (4)
Co(1)–O(11)	1·899 (4)	Co(2)–O(61)	1·911 (4)
Co(1)–N(11)	1·926 (4)	Co(2)–N(51)	1·929 (5)
Co(1)–N(21)	1·935 (5)	Co(2)–N(41)	1·929 (4)
Co(1)–N(31)	1·936 (4)	Co(2)–N(61)	1·932 (4)
N(11)–C(11)	1·469 (7)	N(41)–C(41)	1·482 (7)
C(11)–C(12)	1·509 (8)	C(41)–C(42)	1·499 (7)
C(12)–O(12)	1·230 (6)	C(42)–O(42)	1·228 (6)
C(12)–O(11)	1·271 (6)	C(42)–O(41)	1·294 (6)
N(21)–C(21)	1·488 (6)	N(51)–C(51)	1·485 (7)
C(21)–C(22)	1·509 (8)	C(51)–C(52)	1·504 (8)
C(22)–O(22)	1·231 (6)	C(52)–O(52)	1·236 (6)
C(22)–O(21)	1·283 (6)	C(52)–O(51)	1·278 (6)
N(31)–C(31)	1·480 (6)	N(61)–C(61)	1·459 (7)
C(31)–C(32)	1·513 (7)	C(61)–C(62)	1·511 (8)
C(32)–O(32)	1·227 (6)	C(62)–O(62)	1·228 (6)
C(32)–O(31)	1·286 (6)	C(62)–O(61)	1·279 (6)

O(31)–Co(1)–O(21)	177·9 (1)	O(41)–Co(2)–O(51)	176·5 (2)
O(31)–Co(1)–O(11)	91·3 (2)	O(41)–Co(2)–O(61)	90·4 (2)
O(31)–Co(1)–N(11)	90·1 (2)	O(41)–Co(2)–N(51)	90·7 (2)
O(31)–Co(1)–N(21)	92·1 (2)	O(41)–Co(2)–N(41)	86·7 (2)
O(31)–Co(1)–N(31)	87·0 (2)	O(41)–Co(2)–N(61)	90·4 (2)
O(21)–Co(1)–O(11)	90·4 (2)	O(51)–Co(2)–O(61)	92·3 (2)
O(21)–Co(1)–N(11)	91·3 (2)	O(51)–Co(2)–N(51)	86·6 (2)
O(21)–Co(1)–N(21)	86·3 (2)	O(51)–Co(2)–N(41)	91·2 (2)
O(21)–Co(1)–N(31)	91·8 (2)	O(51)–Co(2)–N(61)	91·9 (2)
O(11)–Co(1)–N(11)	86·2 (2)	O(61)–Co(2)–N(51)	178·4 (2)
O(11)–Co(1)–N(21)	176·5 (2)	O(61)–Co(2)–N(41)	87·1 (2)
O(11)–Co(1)–N(31)	88·8 (2)	O(61)–Co(2)–N(61)	85·9 (2)
N(11)–Co(1)–N(21)	92·8 (2)	N(51)–Co(2)–N(41)	94·1 (2)
N(11)–Co(1)–N(31)	174·1 (2)	N(51)–Co(2)–N(61)	92·9 (2)
N(21)–Co(1)–N(31)	92·4 (2)	N(41)–Co(2)–N(61)	172·5 (2)
C(11)–N(11)–Co(1)	109·8 (3)	C(41)–N(41)–Co(2)	108·7 (3)
N(11)–C(11)–C(12)	111·7 (5)	N(41)–C(41)–C(42)	110·2 (4)
O(12)–C(12)–O(11)	124·7 (5)	O(42)–C(42)–O(41)	122·5 (5)
O(12)–C(12)–C(11)	119·6 (5)	O(42)–C(42)–C(41)	120·6 (5)
O(11)–C(12)–C(11)	115·6 (5)	O(41)–C(42)–C(41)	116·9 (5)
C(12)–O(11)–Co(1)	116·4 (3)	C(42)–O(41)–Co(2)	114·7 (3)
C(21)–N(21)–Co(1)	108·8 (3)	C(51)–N(51)–Co(2)	109·6 (3)
N(21)–C(21)–C(22)	110·7 (5)	N(51)–C(51)–C(52)	111·3 (5)
O(22)–C(22)–O(21)	123·5 (5)	O(52)–C(52)–O(51)	124·3 (5)
O(22)–C(22)–C(21)	120·2 (5)	O(52)–C(52)–C(51)	119·2 (5)
O(21)–C(22)–C(21)	116·3 (5)	O(51)–C(52)–C(51)	116·5 (5)
C(22)–O(21)–Co(1)	115·7 (3)	C(52)–O(51)–Co(2)	115·8 (3)
C(31)–N(31)–Co(1)	108·3 (3)	C(61)–N(61)–Co(2)	109·2 (3)
N(31)–C(31)–C(32)	110·4 (4)	N(61)–C(61)–C(62)	111·7 (4)
O(32)–C(32)–O(31)	123·2 (5)	O(62)–C(62)–O(61)	123·5 (5)
O(32)–C(32)–C(31)	120·7 (5)	O(62)–C(62)–C(61)	121·4 (5)
O(31)–C(32)–C(31)	116·1 (4)	O(61)–C(62)–C(61)	115·0 (5)
C(32)–O(31)–Co(1)	115·5 (3)	C(62)–O(61)–Co(2)	115·9 (3)

0108-2701/88/122199-03$03.00

Experimental. Purple crystal, dimensions 0·25 × 0·25 × 0·07 mm, space group $P2_1/n$ [non-standard setting of $P2_1/c$ (No. 14)], Enraf–Nonius CAD-4F-11 κ-geometry diffractometer, monochromated Mo $K\alpha$ radiation, $\omega/2\theta$-scan technique, variable scan width where $\Delta\omega = (0{\cdot}8 + 0{\cdot}35\tan\theta)°$, scan rate 4° min^{-1} in ω, scan extended 25% on either side for background measurement, $3 < 2\theta < 50°$, lattice parameters from 25 reflections with $2\theta > 30°$, empirical absorption correction with *DIFABS* (Walker & Stuart, 1983), transmission factors range from 0·89 to 1·29, three intensity standards showed no decay. Solved by direct methods using *MITHRIL* (Gilmore, 1984) followed by difference-Fourier syntheses. 4338 unique reflections measured (h 0→16, k 0→15, l −16→14), 2691 with $I_o > 2\sigma(I_o)$ used in structure refinement, full-matrix least squares on F (325 variables) using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985). All non-H atoms refined anisotropically, H atoms placed in calculated positions (C—H = 0·95 Å) and assigned a temperature factor of 1·2 times B_{eq} of the atom to which they are attached. Refined occupancies of water molecules converged to unity and were fixed there in final cycles. Neutral-atom scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}044$, $wR = 0{\cdot}045$, GOF = 1·48. Weights given by $w = 4F_o^2/\sigma^2(F_o^2)$. In final cycle maximum LS shift/e.s.d. 0·001. Final difference-Fourier map maximum and minimum peaks 0·45 and −0·39 e Å^{-3}, respectively. Further details of data-collection procedures given by Silverman, Dewan, Giandomenico & Lippard (1980). Table 1* gives atomic positional parameters, Table 2 bond lengths and angles, and Table 3 intermolecular contacts. Fig. 1 shows the molecular geometry and atom-labeling scheme.

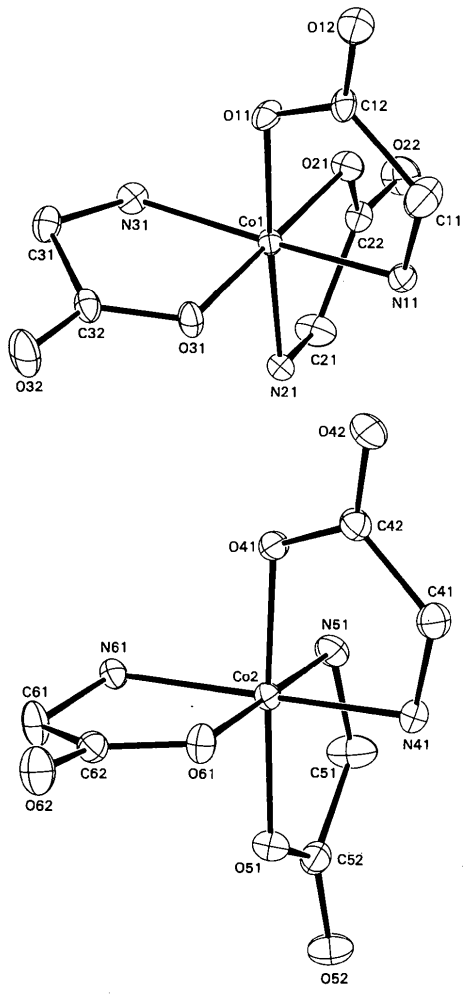

Fig. 1. *ORTEP*II diagrams (Johnson, 1976) showing the two crystallographically independent geometric isomers of the molecule with the atom-labeling scheme and 30% probability thermal ellipsoids. H atoms omitted for clarity.

Table 3. *Possible hydrogen-bonding interactions* (<3·7 Å)

Water molecules of crystallization are labeled O(71), O(72), O(73) and O(74).

N(11)···O(32^{ii})	2·976 (6)	O(31)···N(61^{viii})	3·103 (5)
N(11)···O(42^{iii})	2·998 (6)	O(31)···O(71^{ix})	3·626 (6)
N(11)···O(73^{iv})	3·120 (6)	O(32)···O(73^{v})	2·904 (6)
N(11)···O(41^{iii})	3·134 (5)	O(32)···N(61^{viii})	3·018 (6)
O(11)···N(21^{v})	3·310 (6)	N(41)···O(72^{x})	3·032 (6)
O(11)···O(72^{i})	3·403 (5)	N(41)···O(62^{xi})	3·203 (6)
O(11)···O(71^{vi})	3·612 (5)	O(41)···O(73^{xii})	3·142 (6)
O(12)···O(72^{vii})	2·829 (6)	O(42)···N(61^{xiii})	2·949 (6)
O(12)···N(21^{v})	3·054 (6)	O(42)···O(73^{xii})	3·320 (6)
O(12)···N(31^{v})	3·076 (6)	N(51)···O(71^{xiv})	2·946 (6)
O(12)···O(74^{viii})	3·404 (6)	N(51)···O(62^{xi})	3·051 (6)
N(21)···O(73^{iv})	2·943 (6)	N(51)···O(61^{xi})	3·327 (6)
N(21)···O(71^{ix})	3·645 (6)	O(51)···O(74^{xv})	2·905 (6)
O(21)···O(72^{i})	2·813 (5)	O(52)···O(71^{i})	2·873 (6)
O(22)···O(73^{i})	2·719 (6)	O(52)···O(73^{x})	3·585 (6)
O(22)···N(41^{x})	2·972 (6)	N(61)···O(71^{xiv})	3·311 (6)
O(22)···O(74^{ii})	3·617 (7)	O(61)···O(74^{xv})	3·551 (6)
N(31)···O(52^{vi})	2·922 (6)	O(62)···O(74^{i})	2·870 (6)
N(31)···O(74^{ii})	3·042 (6)	O(71)···O(72^{xi})	2·903 (6)
N(31)···O(51^{vi})	3·560 (5)		

Symmetry code: (i) $+x, +y, +z$; (ii) $-\frac{1}{2}+x, \frac{3}{2}-y, -\frac{1}{2}+z$; (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $-x, 2-y, 1-z$; (v) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (vii) $-x, 1-y, 1-z$; (viii) $1-x, 1-y, 2-z$; (ix) $-1+x, 1+y, +z$; (x) $1-x, 1-y, 1-z$; (xi) $\frac{3}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z$; (xii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (xiii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (xiv) $2-x, -y, 2-z$; (xv) $2-x, 1-y, 2-z$.

Related literature. The structure determination has established the atom connectivity and stereochemistry of the compound to be that displayed in Fig. 1. The structure of one of the geometric isomers reported here, namely that represented by Co(2), has been published previously (Miyanaga, Sakaguchi, Morimoto, Kushi & Yoneda, 1982).

* Lists of anisotropic thermal parameters, structure factors, H-atom parameters and intermolecular distances, and a complete description of the structure determination have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51258 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1988). C**44**, 2201–2202

Bis(triphenylarsine oxide)hydrogen(I) Tetrachloroaurate(III)

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(*Received* 28 *March* 1988; *accepted* 14 *July* 1988)

Abstract. $C_{36}H_{31}As_2O_2^+.AuCl_4^-$, $M_r = 984{\cdot}3$, monoclinic, $P2_1/n$, $a = 13{\cdot}659\ (5)$, $b = 9{\cdot}955\ (5)$, $c = 14{\cdot}481\ (5)$ Å, $\beta = 110{\cdot}98\ (3)°$, $U = 1838{\cdot}5$ Å^3, $Z = 2$, $D_x = 1{\cdot}78$ Mg m^{-3}, $F(000) = 952$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 6{\cdot}1$ mm^{-1}, $T = 293$ K, final $R = 0{\cdot}072$ for 2095 unique observed reflections. The anions are associated with the centre of symmetry at 0,0·5,0·5, with Au–Cl 2·270, 2·280 (4) Å. The Ph_3AsO moieties of the cation are related across the symmetry centre 0,0,0; the proton that connects these *via* a hydrogen bond of 2·39 (2) Å was not located, but presumably lies on the origin.

Experimental. A yellow prism 0·25 × 0·15 × 0·1 mm was mounted in a glass capillary; 5320 profile-fitted intensities were measured on a Stoe–Siemens four-circle diffractometer using monochromated Mo $K\alpha$ radiation ($2\theta_{max}$ 50°, scan ratio $2\theta/\omega = 1$). Three check reflections showed no significant intensity change. An absorption correction based on ψ scans was performed; transmission factors lay in the range 0·69–0·84. Merging equivalents gave 3218 unique reflections (R_{int} 0·035; index ranges h −15 to 15, k 0 to 11, l 0 to 17), 2095 of which with $F > 4\sigma(F)$ were used for all calculations. Cell constants were refined from 2θ values of 68 reflections in the range 15–25°. The structure was solved by the heavy-atom method and subjected to anisotropic full-matrix least-squares refinement on F. H atoms were included using a riding model with C–H = 0·96 Å, except for the acidic H, which was not located (although it very probably lies on the origin). The final R value was 0·072, with wR 0·052. The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}00015F^2$. 205 parameters; S 1·7; max. Δ/σ 0·013; max. $\Delta\rho$ features ±1·2 e Å^{-3}. Final atomic coordinates are presented in Table 1.† Selected bond lengths and angles are given in the *Abstract* and the caption to Fig. 1.

† Lists of structure factors, anisotropic thermal parameters, further bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51246 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
As	62 (1)	1761 (1)	1276 (1)	50 (1)
Au	0	5000	5000	60 (1)
Cl(1)	1069 (3)	6779 (4)	5636 (3)	84 (2)
Cl(2)	1372 (3)	3797 (4)	4886 (3)	85 (2)
O	−352 (7)	1045 (8)	142 (5)	67 (4)
C(11)	1540 (10)	2070 (12)	1731 (7)	48 (5)
C(12)	2150 (11)	1442 (12)	1283 (9)	59 (6)
C(13)	3218 (13)	1630 (14)	1664 (11)	72 (7)
C(14)	3667 (12)	2424 (14)	2465 (12)	70 (7)
C(15)	3064 (13)	3051 (14)	2894 (10)	69 (7)
C(16)	1997 (11)	2916 (12)	2540 (9)	55 (6)
C(21)	−672 (9)	3407 (12)	1151 (8)	48 (5)
C(22)	−800 (11)	4017 (14)	1947 (10)	68 (7)
C(23)	−1336 (13)	5246 (18)	1818 (13)	94 (9)
C(24)	−1715 (12)	5835 (16)	909 (16)	93 (9)
C(25)	−1587 (12)	5244 (16)	120 (13)	90 (8)
C(26)	−1066 (11)	4013 (13)	225 (10)	70 (7)
C(31)	−224 (10)	646 (12)	2210 (8)	52 (5)
C(32)	490 (10)	506 (13)	3160 (9)	61 (6)
C(33)	257 (13)	−314 (13)	3830 (11)	73 (7)
C(34)	−683 (14)	−987 (15)	3536 (13)	80 (8)
C(35)	−1399 (12)	−881 (15)	2598 (13)	77 (8)
C(36)	−1154 (10)	−40 (15)	1927 (10)	71 (6)

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0108-2701/88/122201-02$03.00