

cis positions. Similar coordination geometries have been noted for the derivatives [MnCl(CO)₃(As₂C₇H₁₈)] (Bear & Trotter, 1973) [MnBr(CO)₃{P(OCH₃)₂-C₆H₅}₂] (Kruger, Heckrodt, Reimann & Singleton, 1975), [MnBr(CO)₃{(C₆H₅)₂PC(S)N(C₆H₅)H}] (Just, Klein, Kopf, Steinhäuser & Kramolowsky, 1982) and [MnBr(CO)₃{S₂CC(CH₃)₂P(C₆H₅)₃}] (Winter, Merkel & Kunze, 1983). Further, the Re^I compounds [Re(PO₂F₂)(CO)₃(bpy)] and [ReF(CO)₃(C₆H₁₆N₂)] (Horn & Snow, 1980, 1984) also adopt similar coordination geometries.

The Australian Research Grants Scheme is thanked for support.

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Acta Cryst. (1987). **C43**, 794–797

Structures of Three Glycine-Bridged Polymeric Complexes: [Mn(glycine)(H₂O)₂Cl₂], [Co(glycine)(H₂O)₂Cl₂] and [Co(glycine)(H₂O)₄](NO₃)₂

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(Received 11 November 1986; accepted 11 December 1986)

Abstract. (1): Diaquadichloro(glycine)manganese(II), [MnCl₂(C₂H₅NO₂)(H₂O)₂], *M_r* = 236.94, monoclinic, *P*2₁/*c*, *a* = 8.3975 (4), *b* = 5.6068 (2), *c* = 16.7901 (9) Å, β = 90.175 (4)°, *V* = 790.53 Å³, *Z* = 4, *D_x* = 1.991 Mg m⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 2.25 mm⁻¹, *F*(000) = 476, *T* = 298 K, *R* = 0.017 for 1338 unique reflections with *F* > 4σ(*F*). (2): Diaquadichloro(glycine)cobalt(II), [CoCl₂(C₂H₅NO₂)(H₂O)₂], *M_r* = 240.94, monoclinic, *P*2₁/*n*, *a* = 6.3746 (3), *b* = 15.8638 (8), *c* = 7.7460 (5) Å, β = 97.640 (4)°, *V* = 776.36 Å³, *Z* = 4, *D_x* = 2.061 Mg m⁻³, μ = 2.86 mm⁻¹, *F*(000) = 484, *T* = 298 K, *R* = 0.015 for 1313 unique reflections with *F* > 4σ(*F*). (3): Tetraqua(glycine)cobalt(II) nitrate, [Co(C₂H₅NO₂)(H₂O)₄](NO₃)₂, *M_r* = 330.07, monoclinic, *P*2₁/*n*, *a* = 9.2429 (5), *b* = 6.7203 (3), *c* = 19.0408 (10) Å, β = 96.508 (6)°, *V* = 1175.1 Å³, *Z* = 4, *D_x* = 1.865 Mg m⁻³, μ = 1.52 mm⁻¹, *F*(000) = 676, *T* = 298 K, *R* = 0.026 for 1868 unique reflections with *F* > 4σ(*F*). In all three complexes, coordination of the metal ions is distorted octahedral, with bonds to chloride, water and glycine ligands; nitrate is not coordinated to Co, the octahedral coordination being completed in this case by extra water ligands. In (1), the

chloride ligands are mutually *cis*, the H₂O mutually *trans*; in (2), both chloride and H₂O are mutually *cis*. In all three structures, *cis* positions are occupied by glycine oxygen atoms. The glycine is in its zwitterionic

Table 1. Atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10⁴) for (1)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Mn	7889 (1)	-519 (1)	3347 (1)	187 (1)*
Cl(1)	5411 (1)	1915 (1)	3793 (1)	250 (1)*
Cl(2)	7818 (1)	-2876 (1)	4593 (1)	285 (1)*
O(1)	7568 (1)	1014 (2)	2152 (1)	251 (3)*
O(2)	9889 (1)	2898 (2)	2045 (1)	243 (3)*
C(1)	8556 (2)	2252 (2)	1780 (1)	185 (4)*
C(2)	8125 (2)	3012 (3)	943 (1)	290 (5)*
H(21)	8873 (27)	2387 (36)	602 (13)	513 (62)
H(22)	8134 (28)	4723 (37)	915 (13)	507 (59)
O(3)	9353 (1)	2564 (2)	3679 (1)	283 (3)*
H(31)	9040 (35)	3745 (54)	3951 (16)	708 (76)
H(32)	9715 (24)	3016 (38)	3264 (13)	407 (61)
O(4)	6482 (2)	-3379 (2)	2826 (1)	415 (4)*
H(41)	6238 (29)	-4557 (42)	3050 (14)	513 (67)
H(42)	6029 (27)	-3292 (43)	2467 (14)	477 (69)
N(1)	6525 (2)	2167 (3)	708 (1)	266 (4)*
H(11)	5655 (40)	3071 (61)	978 (20)	1089 (103)
H(12)	6368 (41)	847 (59)	798 (19)	916 (106)
H(13)	6358 (35)	2210 (51)	168 (19)	918 (92)

* *U_{eq}* = $\frac{1}{3}$ (trace of the orthogonalized *U_{ij}* matrix).

form, and the carboxylate oxygen atoms bond to different metal atoms, thus forming a glycine bridge. In this way, polymeric chains are formed: these chains are helical in (1) and (3), but essentially planar zigzag in (2). The polymer chains [and nitrate anions in (3)] are held together by extensive hydrogen bonding.

Experimental. Compounds prepared by reaction of the corresponding hydrated metal(II) salt (chloride or nitrate) with glycine (1:2 molar ratio) in ethanol-water, the pH being adjusted to 2 by addition of dilute HCl or HNO₃ ($T = 333$ K for 2 h). Crystals obtained by slow evaporation of reaction solutions.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for (2)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Co	4113 (1)	1703 (1)	3163 (1)	168 (1)*
Cl(1)	7271 (1)	1362 (1)	1941 (1)	273 (1)*
Cl(2)	2795 (1)	220 (1)	3113 (1)	243 (1)*
O(1)	5177 (2)	2987 (1)	3322 (1)	213 (3)*
O(2)	7392 (2)	2971 (1)	5803 (1)	221 (3)*
C(1)	6608 (3)	3300 (1)	4372 (2)	170 (4)*
C(2)	7568 (3)	4130 (1)	3913 (2)	260 (5)*
H(21)	8979 (37)	4056 (13)	4005 (28)	405 (58)
H(22)	7230 (31)	4554 (12)	4648 (25)	287 (48)
O(3)	5382 (2)	1521 (1)	5754 (1)	256 (3)*
H(31)	6191 (39)	1939 (15)	6007 (29)	530 (64)
H(32)	5911 (34)	1103 (13)	5990 (28)	440 (57)
O(4)	1384 (2)	2037 (1)	4300 (2)	287 (4)*
H(41)	1354 (49)	2414 (20)	4890 (39)	745 (96)
H(42)	331 (42)	1926 (15)	3797 (32)	436 (71)
N(1)	6820 (3)	4385 (1)	2103 (2)	236 (4)*
H(11)	5459 (42)	4548 (15)	1943 (29)	504 (67)
H(12)	7549 (42)	4835 (16)	1861 (30)	601 (75)
H(13)	6861 (36)	3943 (15)	1332 (30)	479 (62)

* $U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

Table 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^4$) for (3)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Co	4156 (1)	2415 (1)	1969 (1)	271 (1)*
O(1)	3816 (1)	2786 (2)	3029 (1)	319 (4)*
O(2)	1844 (1)	4690 (2)	2929 (1)	386 (4)*
C(1)	2792 (2)	3645 (2)	3277 (1)	271 (5)*
C(2)	2676 (2)	3389 (3)	4059 (1)	323 (6)*
H(21)	1846 (27)	2756 (29)	4110 (13)	434 (62)
H(22)	2686 (22)	4556 (33)	4275 (10)	484 (57)
N(1)	3861 (2)	2110 (3)	4395 (1)	361 (5)*
H(11)	4674 (31)	2021 (39)	4087 (16)	728 (80)
H(12)	4124 (33)	2546 (32)	4816 (16)	736 (88)
H(13)	3491 (31)	1042 (42)	4416 (14)	700 (87)
O(3)	2101 (1)	3629 (2)	1565 (1)	336 (4)*
H(31)	1402 (33)	2836 (38)	1426 (15)	616 (80)
H(32)	1775 (24)	4094 (35)	1888 (12)	626 (68)
O(4)	4630 (2)	2006 (3)	946 (1)	404 (5)*
H(41)	4209 (31)	2490 (31)	683 (15)	400 (80)
H(42)	5516 (34)	1903 (41)	855 (16)	727 (85)
O(5)	4935 (2)	5327 (2)	1965 (1)	503 (5)*
H(51)	5197 (30)	5911 (40)	2311 (14)	643 (81)
H(52)	5340 (31)	5648 (44)	1655 (15)	743 (88)
O(6)	6130 (1)	1184 (3)	2365 (1)	568 (6)*
H(61)	6389 (28)	1168 (39)	2773 (15)	623 (80)
H(62)	6919 (34)	1338 (44)	2179 (16)	811 (92)
N(2)	8647 (2)	1535 (3)	1087 (1)	373 (5)*
O(21)	8656 (2)	1681 (3)	1733 (1)	603 (6)*
O(22)	9833 (1)	1517 (3)	824 (1)	480 (5)*
O(23)	7507 (1)	1447 (3)	692 (1)	612 (6)*
N(3)	7685 (2)	1495 (2)	4356 (1)	368 (5)*
O(31)	7358 (2)	1543 (3)	4974 (1)	559 (6)*
O(32)	8957 (2)	1686 (3)	4243 (1)	652 (6)*
O(33)	6695 (2)	1279 (3)	3868 (1)	736 (7)*

* $U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

(1) [(2); (3) in square brackets where different]: crystal size $0.31 \times 0.38 \times 0.15$ [$0.33 \times 0.35 \times 0.38$; $0.27 \times 0.35 \times 0.54$] mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections with $20 < 2\theta < 25^\circ$, intensity measurements in ω/θ scan mode, scan width = 1.02 [1.19 ; 1.19] $^\circ$ + α -doublet separation, scan time = 14 – 56 s, $2\theta_{max} = 50^\circ$, h 0→10, k -6→6, l -20→20 [h -8→2, k 0→19, l -9→9; h 0→10, k -7→1, l -22→22], no significant variation for three standard reflections, semi-empirical absorption correction, transmission 0.374 – 0.499 [0.254 – 0.290 ; 0.411 – 0.470]. 2824 [1863; 2364] reflections measured, 1376 [1367; 2052] unique, $R_{int} = 0.018$ [0.015; 0.018], 1338 [1313; 1868] with

Table 4. Coordination geometry (\AA and $^\circ$)

Compound (1)			
Mn-Cl(1)	2.601 (1)	Mn-Cl(2)	2.475 (1)
Mn-O(1)	2.199 (1)	Mn-O(3)	2.192 (1)
Mn-O(4)	2.174 (2)	Mn-O(2 ^h)	2.170 (1)
Cl(1)-Mn-Cl(2)	90.9 (1)	Cl(1)-Mn-O(1)	87.8 (1)
Cl(2)-Mn-O(1)	167.6 (1)	Cl(1)-Mn-O(3)	87.8 (1)
Cl(2)-Mn-O(3)	102.8 (1)	O(1)-Mn-O(3)	89.5 (1)
Cl(1)-Mn-O(4)	93.9 (1)	Cl(2)-Mn-O(4)	86.1 (1)
O(1)-Mn-O(4)	81.7 (1)	O(3)-Mn-O(4)	171.0 (1)
Cl(1)-Mn-O(2 ^h)	172.5 (1)	Cl(2)-Mn-O(2 ^h)	93.5 (1)
O(1)-Mn-O(2 ^h)	89.2 (1)	O(3)-Mn-O(2 ^h)	85.3 (1)
O(4)-Mn-O(2 ^h)	92.5 (1)		
Compound (2)			
Co-Cl(1)	2.398 (1)	Co-Cl(2)	2.496 (1)
Co-O(1)	2.146 (1)	Co-O(3)	2.082 (1)
Co-O(4)	2.119 (2)	Co-O(2 ^h)	2.069 (1)
Cl(1)-Co-Cl(2)	94.6 (1)	Cl(1)-Co-O(1)	87.8 (1)
Cl(2)-Co-O(1)	177.2 (1)	Cl(1)-Co-O(3)	96.4 (1)
Cl(2)-Co-O(3)	88.4 (1)	O(1)-Co-O(3)	89.8 (1)
Cl(1)-Co-O(4)	178.0 (1)	Cl(2)-Co-O(4)	87.1 (1)
O(1)-Co-O(4)	90.5 (1)	O(3)-Co-O(4)	82.5 (1)
Cl(1)-Co-O(2 ^h)	94.7 (1)	Cl(2)-Co-O(2 ^h)	94.8 (1)
O(1)-Co-O(2 ^h)	86.5 (1)	O(3)-Co-O(2 ^h)	168.2 (1)
O(4)-Co-O(2 ^h)	86.3 (1)		
Compound (3)			
Co-O(1)	2.092 (1)	Co-O(3)	2.129 (1)
Co-O(4)	2.062 (2)	Co-O(5)	2.086 (2)
Co-O(6)	2.067 (2)	Co-O(2 ^h)	2.070 (1)
O(1)-Co-O(3)	94.4 (1)	O(1)-Co-O(4)	176.3 (1)
O(3)-Co-O(4)	89.2 (1)	O(1)-Co-O(5)	88.9 (1)
O(3)-Co-O(5)	86.2 (1)	O(4)-Co-O(5)	90.7 (1)
O(1)-Co-O(6)	85.3 (1)	O(3)-Co-O(6)	178.9 (1)
O(4)-Co-O(6)	91.1 (1)	O(5)-Co-O(6)	94.9 (1)
O(1)-Co-O(2 ^h)	84.1 (1)	O(3)-Co-O(2 ^h)	89.0 (1)
O(4)-Co-O(2 ^h)	96.6 (1)	O(5)-Co-O(2 ^h)	171.2 (1)
O(6)-Co-O(2 ^h)	89.8 (1)		

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

$F > 4\sigma(F)$. Direct methods [Patterson for (3)], difference syntheses, blocked-cascade least-squares refinement on F , $w^{-1} = \sigma^2(F) + gF^2$, $g = 0$ [0; 0.00025], anisotropic thermal parameters for all non-H atoms, isotropic for H with freely refined coordinates, isotropic extinction correction $F_c^0 = F_c/(1 + xF_c^2/\sin 2\theta)^{1/4}$, $x = 1.14(5) \times 10^{-5}$ [$1.55(5) \times 10^{-5}$; $9.9(7) \times 10^{-6}$]. 128 [128; 216] parameters, $R = 0.017$ [0.015; 0.026],

$wR = 0.021$ [0.021; 0.037], max. $\Delta/\sigma = 0.004$ [0.015; 0.034], mean = 0.001 [0.003; 0.007], max. $\Delta\rho = +0.22$ [+0.26; +0.36] $\text{e}\text{\AA}^{-3}$, min. = -0.22 [-0.18; -0.37] $\text{e}\text{\AA}^{-3}$, slope of normal probability plot = 1.81 [1.85; 1.53]. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* programs (Sheldrick, 1985).

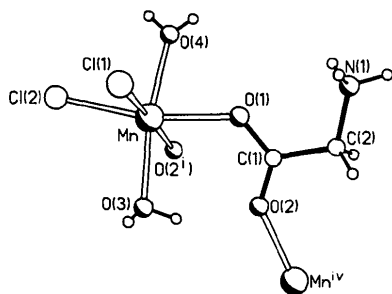


Fig. 1. The asymmetric unit of (1), showing the atom numbering and the connections to the adjoining units. Symmetry code: (i) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iv) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$.

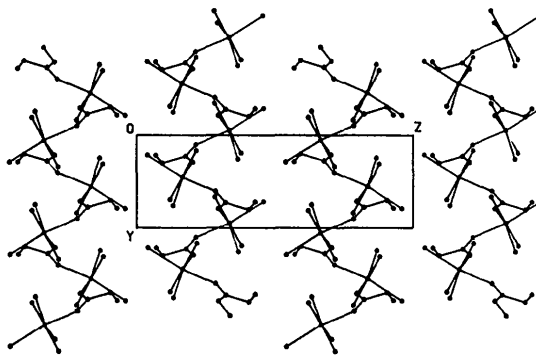


Fig. 4. Projection of (1) down the a axis.

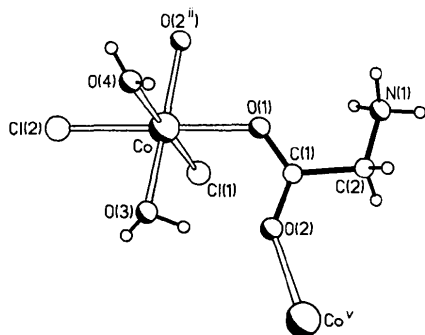


Fig. 2. The asymmetric unit of (2), showing the atom numbering and the connections to the adjoining units. Symmetry code: (ii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (v) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$.

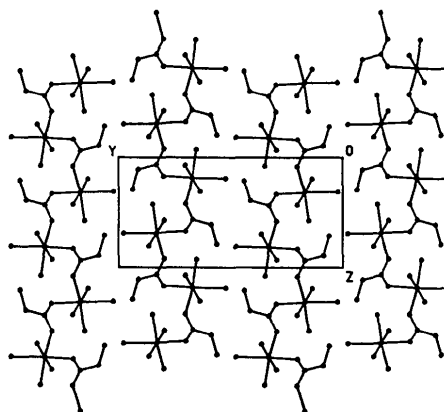


Fig. 5. Projection of (2) down the a axis.

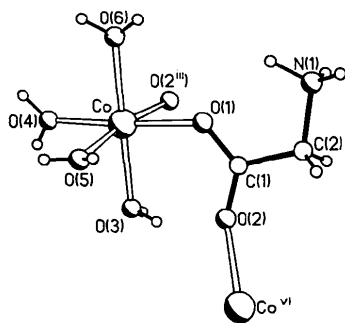


Fig. 3. The asymmetric unit of (3) (without nitrate anions), showing the atom numbering and the connections to the adjoining units. Symmetry code: (iii) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (vi) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

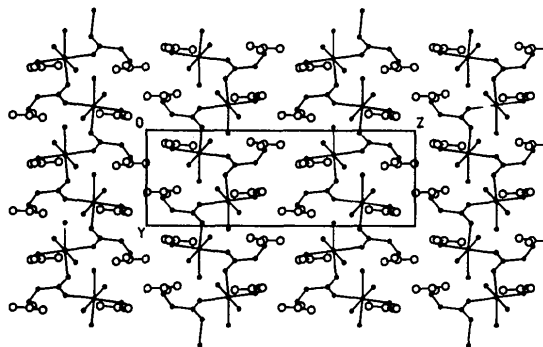


Fig. 6. Projection of (3) down the a axis.

Tables 1 to 3 give the atomic coordinates of compounds (1) to (3) respectively, and Table 4 lists some geometric data.* Views of the compounds are given in Figs. 1 to 3, and views of the projection of each compound along the *a* axis are presented in Figs. 4, 5 and 6.

Related literature. In complexes with metal ions, glycine and other amino acids may coordinate as their anions or as zwitterionic acids, depending on conditions. In the former case the amino group may take direct part in the coordination (Low, Hirshfeld & Richards, 1959; Stosick, 1945; Dejehet & Debuyst, 1978); in the zwitterion form, the protonated amine N atom is prevented from doing so, and coordination is *via* the carboxylate oxygen atoms alone, to give either monodentate or bidentate bonding (Dejehet, Debuyst, Ledieu, Declercq, Germain & Van Meerssche, 1978; Glowiak & Kozlowski, 1980; Ardon, Bino, Cohen & Felthouse, 1984, and references therein). In some cases,

* Lists of structure factors, anisotropic thermal parameters and additional bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43638 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

mixtures of the two types of complex are formed (Herzog & Kalies, 1967).

We thank SERC for a research grant towards crystallographic equipment, and the University of Newcastle upon Tyne for the award of a Ridley Fellowship to OML.

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Acta Cryst. (1987). **C43**, 797–799

Structure of an Antisickling Agent, *N*-Phenylacetyl-L-phenylalanine

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Abstract. C₁₇H₁₇NO₃, *M_r* = 283.3, orthorhombic, *P*2₁2₁2₁, *a* = 8.400 (1), *b* = 5.423 (1), *c* = 32.548 (3) Å, *V* = 1482.7 (1) Å³, *Z* = 4, *D_x* = 1.270 g cm⁻³, λ(CuKα) = 1.5418 Å, μ = 6.25 cm⁻¹, *F*(000) = 600, *T* = 263 K, *R* = 0.048 for 1199 unique observed reflections. The molecule has adopted a compact and amphipathic conformation. Peptide torsion angles: L-Phe: φ = 76.8 (4), ω = 171.5 (4), χ¹ = -61.2 (5), χ^{2,1} = -72.6 (5); phenyl: χ¹ = -72.9 (5), χ^{2,1} = 96.1 (5)°. Intramolecular edge-to-face interaction between phenyl rings: phenyl(L-Phe)···phenyl(phenylacetyl) centroid separation =

5.05 (1) Å and interplanar angle = 70.1 (5)°. Intermolecular hydrogen bond: OH(L-Phe)···O(phenylacetyl') = 2.678 (6) Å. Intermolecular edge-to-face interaction between phenyl rings: phenyl(L-Phe)···phenyl(phenylacetyl') centroid separation = 4.85 (1) Å and interplanar angle = 70.1 (5)°.

Experimental. Rectangular crystal, 0.2 × 0.3 × 0.6 mm, Nicolet P3 diffractometer, Ni-filtered radiation, ω-scan method, (sinθ)/λ < 0.58 Å⁻¹, lattice parameters from the 2θ values of nine reflections with