

Fig. 1. Structure of the anion, with 50% probability ellipsoids and the labelling of the independent atoms.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent thermal parameters ($\text{\AA}^2 \times 10^4$)

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

	x	y	z	U_{eq}
Mo	5575 (1)	7500	5933 (1)	546 (2)
O	4778 (3)	7500	6715 (5)	837 (20)
N(1)	6658 (3)	7500	4892 (5)	588 (20)
C(1)	7203 (3)	7500	4355 (6)	452 (20)
S(1)	8002 (1)	7500	3607 (2)	680 (7)
N(2)	5234 (3)	7500	3843 (6)	640 (21)
C(2)	5154 (4)	7500	2614 (9)	583 (25)
S(2)	5050 (1)	7500	898 (2)	862 (8)
N(3)	6194 (3)	7500	7771 (6)	781 (24)
C(3)	6681 (4)	7500	8539 (7)	644 (25)
S(3)	7345 (1)	7500	9629 (2)	892 (9)
N(4)	5701 (2)	6082 (2)	5890 (4)	656 (15)
C(4)	5919 (2)	5328 (3)	5968 (5)	528 (15)
S(4)	6204 (1)	4282 (1)	6089 (1)	851 (6)
N(5)	6405 (2)	4865 (2)	1479 (3)	526 (12)
C(51)	6628 (3)	5690 (3)	2323 (5)	965 (25)
C(52)	6446 (4)	5054 (4)	-62 (5)	1182 (31)
C(53)	5656 (3)	4653 (5)	1875 (8)	1516 (40)
C(54)	6845 (4)	4064 (4)	1884 (7)	1642 (43)

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fac-(2,2'-Bipyridyl)tricarbonylchloromanganese(I)

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Abstract. $[\text{MnCl}(\text{CO})_3(\text{C}_{10}\text{H}_8\text{N}_2)]$, $M_r = 330.6$, triclinic, $P\bar{1}$, $a = 11.040$ (1), $b = 11.491$ (1), $c = 10.9649$ (7) \AA , $\alpha = 94.03$ (1), $\beta = 102.45$ (1), $\gamma = 84.80$ (1) $^\circ$, $V = 1351$ (2) \AA^3 , $Z = 4$, $D_x = 1.627$ Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.7107$ \AA , $\mu = 1.01$ mm^{-1} , $F(000) = 664$, $T = 295$ (2) K, $R = 0.037$ for 3245

0108-2701/87/040792-03\$01.50

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Mo—O	1.646 (5)	Mo—N(1)	2.228 (5)
Mo—N(2)	2.050 (6)	Mo—N(3)	2.063 (6)
Mo—N(4)	2.053 (4)	N(1)—C(1)	1.127 (8)
C(1)—S(1)	1.636 (6)	N(2)—C(2)	1.157 (10)
C(2)—S(2)	1.612 (8)	N(3)—C(3)	1.151 (9)
C(3)—S(3)	1.596 (7)	N(4)—C(4)	1.158 (6)
C(4)—S(4)	1.599 (5)	N(5)—C(51)	1.482 (6)
N(5)—C(52)	1.465 (6)	N(5)—C(53)	1.465 (7)
N(5)—C(54)	1.461 (8)		
O—Mo—N(1)	179.5 (2)	O—Mo—N(2)	98.4 (2)
N(1)—Mo—N(2)	82.1 (2)	O—Mo—N(3)	97.4 (2)
N(1)—Mo—N(3)	82.1 (2)	N(2)—Mo—N(3)	164.2 (2)
O—Mo—N(4)	96.4 (1)	N(1)—Mo—N(4)	83.6 (1)
N(2)—Mo—N(4)	91.0 (1)	N(3)—Mo—N(4)	87.3 (1)
N(4)—Mo—N(4')	166.7 (2)	Mo—N(1)—C(1)	179.4 (5)
N(1)—C(1)—S(1)	178.8 (5)	Mo—N(2)—C(2)	169.4 (6)
N(2)—C(2)—S(2)	179.5 (6)	Mo—N(3)—C(3)	162.3 (6)
N(3)—C(3)—S(3)	178.9 (7)	Mo—N(4)—C(4)	165.4 (4)
N(4)—C(4)—S(4)	178.9 (4)	C(51)—N(5)—C(52)	111.0 (4)
C(51)—N(5)—C(53)	107.2 (4)	C(52)—N(5)—C(53)	109.6 (4)
C(51)—N(5)—C(54)	109.8 (4)	C(52)—N(5)—C(54)	111.7 (4)
C(53)—N(5)—C(54)	107.3 (5)		

N(4') is related to N(4) by the mirror plane (symmetry operator $x, \frac{1}{2}-y, z$).

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observed reflections. The distorted octahedral Mn environment is comprised of three CO ligands, defining one octahedral face, two N atoms and the Cl atom. The greatest distortion from the ideal geometry arises as a result of the restricted bite distance of the chelate; N(1)—Mn—N(2) 78.5 (1), 78.8 (1) $^\circ$. There are no

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significant differences in chemically equivalent parameters describing the two molecules constituting the asymmetric unit.

Experimental. $[\text{MnCl}(\text{CO})_3(\text{bpy})]$ prepared according to the literature procedure (Abel & Wilkinson, 1959). Orange crystal $0.08 \times 0.23 \times 0.50$ mm grown from

CH_2Cl_2 . Enraf-Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; $\omega:2\theta$ -scan technique. Cell parameters from least-squares procedure (De Boer & Duisenberg, 1984) on 25 reflections ($13 \leq \theta \leq 15^\circ$). Analytical absorption correction: max./min. transmission factors 0.9209, 0.7776 (Sheldrick, 1976). Total of 5663 reflections ($1 \leq \theta \leq 25^\circ$) measured in the range $-13 \leq h \leq 13$, $-13 \leq k \leq 13$, $-13 \leq l \leq 1$. No significant variation in the intensities of three standards (372, 536, 625) monitored every 3600 s. 4765 unique reflections ($R_{\text{int}} = 0.021$), 3245 satisfied $I \geq 2.5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement of 361 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms located from difference map. At convergence $R = 0.037$, $wR = 0.046$, $w = 0.212/[\sigma^2(F) + 0.0082F^2]$, $S = 0.29$, $(\Delta/\sigma)_{\text{max}} \leq 0.001$, $\Delta\rho_{\text{max}} = 0.36$, $\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{Å}^{-3}$; no extinction correction. Scattering factors for H, C, Cl, N and O given in *SHELX76* (Sheldrick, 1976) and those for neutral Mn corrected for f' and f'' (Hamilton & Ibers, 1974). University of Adelaide's VAX11/780 computer system. Atomic parameters are given in Table 1, bond distances and angles in Table 2,* and the numbering scheme is shown in Fig. 1.

Table 1. Fractional atomic coordinates and B_{eq} (Å^2) values

$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Mn(a)	0.34334 (5)	0.49607 (5)	0.22305 (5)	3.09
Cl(a)	0.1573 (1)	0.3941 (1)	0.1715 (1)	3.82
C(1a)	0.3994 (4)	0.4159 (4)	0.0953 (4)	4.14
O(1a)	0.4380 (4)	0.3670 (3)	0.0161 (3)	6.31
C(2a)	0.4132 (4)	0.3791 (3)	0.3240 (4)	3.92
O(2a)	0.4580 (3)	0.3075 (3)	0.3894 (3)	5.74
C(3a)	0.4848 (4)	0.5687 (4)	0.2631 (4)	4.20
O(3a)	0.5771 (3)	0.6099 (4)	0.2893 (4)	6.69
N(1a)	0.2472 (3)	0.6303 (3)	0.1182 (3)	3.18
N(2a)	0.2666 (3)	0.5954 (3)	0.3533 (3)	3.21
C(4a)	0.2412 (4)	0.6421 (4)	-0.0039 (4)	4.27
C(5a)	0.1702 (5)	0.7311 (4)	-0.0694 (4)	4.87
C(6a)	0.1056 (5)	0.8137 (4)	-0.0064 (5)	5.20
C(7a)	0.1125 (4)	0.8051 (4)	0.1195 (5)	4.65
C(8a)	0.1851 (3)	0.7117 (3)	0.1795 (4)	3.32
C(9a)	0.1980 (3)	0.6941 (3)	0.3136 (3)	3.31
C(10a)	0.1439 (4)	0.7710 (3)	0.3939 (4)	4.27
C(11a)	0.1627 (5)	0.7468 (4)	0.5187 (4)	4.88
C(12a)	0.2326 (5)	0.6453 (4)	0.5589 (4)	4.90
C(13a)	0.2815 (4)	0.5718 (4)	0.4752 (4)	4.19
Mn(b)	0.29848 (5)	0.6407 (5)	-0.21373 (5)	3.18
Cl(b)	0.0970 (1)	-0.0119 (1)	-0.2721 (1)	3.82
C(1b)	0.3682 (5)	-0.0831 (4)	-0.1994 (4)	4.79
O(1b)	0.4122 (4)	-0.1770 (3)	-0.1881 (4)	7.49
C(2b)	0.2898 (5)	0.0663 (4)	-0.0514 (4)	4.87
O(2b)	0.2853 (5)	0.0678 (4)	0.0509 (3)	8.58
C(3b)	0.4495 (4)	0.1223 (4)	-0.1734 (4)	4.01
O(3b)	0.5464 (3)	0.1557 (3)	-0.1473 (4)	5.83
N(1b)	0.2903 (3)	0.0742 (3)	-0.4014 (3)	4.41
N(2b)	0.2059 (3)	0.2254 (3)	0.2457 (3)	3.24
C(4b)	0.3368 (5)	-0.0078 (5)	-0.4740 (5)	6.50
C(5b)	0.3186 (7)	0.0037 (7)	-0.6048 (6)	8.83
C(6b)	0.2513 (8)	0.1036 (9)	-0.6545 (6)	9.75
C(7b)	0.2037 (6)	0.1868 (6)	-0.5801 (5)	7.35
C(8b)	0.2260 (4)	0.1717 (5)	-0.4531 (4)	4.81
C(9b)	0.1817 (4)	0.2569 (4)	-0.3644 (4)	3.95
C(10b)	0.1157 (5)	0.3686 (5)	-0.3954 (5)	5.57
C(11b)	0.0770 (5)	0.4388 (4)	-0.3032 (6)	5.71
C(12b)	0.1008 (4)	0.4041 (4)	-0.1855 (5)	5.07
C(13b)	0.1673 (4)	0.2985 (3)	-0.1589 (4)	3.96

Related literature. The coordination geometry found for $[\text{MnCl}(\text{CO})_3(\text{bpy})]$ closely resembles that reported for the related compound $[\text{MnBr}(\text{CO})_3(\text{NCCCH}_3)_2]$ (Sarapu & Fenske, 1972) in which the N donor atoms occupy

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bipyridyl geometries have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43544 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Interatomic distances (Å) and angles ($^\circ$)

	Molecule a		Molecule b		
	a	b	a	b	
Mn-Cl	2.399 (1)	2.404 (1)	Mn-N(2)	2.044 (3)	2.049 (3)
Mn-C(1)	1.812 (5)	1.796 (4)	C(1)-O(1)	1.140 (5)	1.146 (5)
Mn-C(2)	1.812 (4)	1.801 (5)	C(2)-O(2)	1.136 (5)	1.133 (5)
Mn-C(3)	1.794 (5)	1.807 (5)	C(3)-O(3)	1.135 (5)	1.140 (6)
Mn-N(1)	2.062 (3)	2.051 (3)			
Cl-Mn-C(1)	89.9 (1)	89.2 (2)	C(3)-Mn-N(1)	93.6 (2)	92.0 (2)
Cl-Mn-C(2)	87.9 (1)	89.9 (2)	C(3)-Mn-N(2)	92.6 (2)	93.4 (2)
Cl-Mn-C(3)	178.4 (1)	178.7 (1)	N(1)-Mn-N(2)	78.5 (1)	78.8 (1)
Cl-Mn-N(1)	88.0 (1)	86.7 (1)	Mn-C(1)-O(1)	177.9 (4)	178.8 (5)
Cl-Mn-N(2)	88.1 (1)	86.0 (1)	Mn-C(2)-O(2)	178.5 (4)	179.5 (5)
C(1)-Mn-C(2)	89.1 (2)	88.2 (2)	Mn-C(3)-O(3)	177.0 (4)	177.9 (4)
C(1)-Mn-C(3)	89.6 (2)	91.3 (2)	Mn-N(1)-C(4)	126.1 (3)	125.3 (4)
C(1)-Mn-N(1)	95.9 (2)	97.3 (2)	Mn-N(1)-C(8)	115.8 (2)	115.1 (3)
C(1)-Mn-N(2)	174.0 (2)	174.1 (2)	C(4)-N(1)-C(8)	118.2 (3)	119.5 (4)
C(2)-Mn-C(3)	90.6 (2)	91.3 (2)	Mn-N(2)-C(13)	125.6 (3)	125.3 (3)
C(2)-Mn-N(1)	173.5 (2)	173.5 (2)	Mn-N(2)-C(9)	116.3 (2)	115.7 (3)
C(2)-Mn-N(2)	96.4 (2)	95.3 (2)	C(9)-N(2)-C(13)	118.1 (3)	119.0 (3)

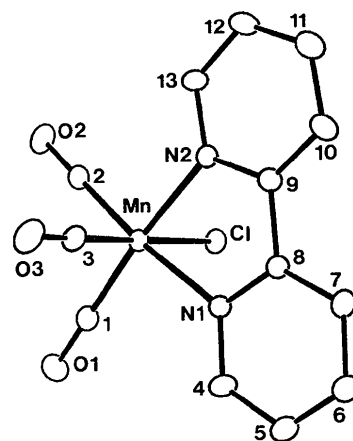


Fig. 1. The numbering scheme employed for $[\text{MnCl}(\text{CO})_3(\text{bpy})]$ (Johnson, 1971).

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.

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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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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).