

Table 4. *Hydrogen bonds*

O—H...O	O...O	H...O	∠O—H—O
O(5)—H(5)...O(2)	2.897 (2) Å	2.10 (3) Å	168°
O(5)—H(6)...O(1)	2.951 (2)	2.19 (3)	178

Table 5. *Deviations from best planes (Å)*

P1		P2		P3	
C(1)	0.008	C(1)	0.000	C(6)	0.003
C(2)	-0.010	C(7)	-0.001	C(8)	-0.011
C(3)	-0.001	O(1)	0.001	O(3)	0.004
C(4)	0.010	O(2)	0.001	O(4)	0.004
C(5)	-0.012				
C(6)	0.003	Angles between planes (°)			
C(7)	-0.108*	P1—P2	78		
C(8)	0.049*	P1—P3	34		
H(1)	-0.103*	P2—P3	67		
H(2)	-0.013*				
H(3)	0.037*				
H(4)	-0.024*				

* Atoms not included in the calculation of the plane.

The structure has two hydrogen bonds (Table 4). Both involve the carboxyl group with the more normal O—C—O angle of 125.9°. They can be classified as rather weak.

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Polymeric 1,10-Phenanthrolinebis(phosphinato)manganese(II)

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Abstract. $C_{12}H_{12}MnN_2O_4P_2$, $P2_1/c$, $a = 10.791$ (9), $b = 11.173$ (8), $c = 13.15$ (2) Å, $\beta = 118.65$ (3)°, $Z = 4$, $D_x = 1.74$ g cm⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 98.4$ cm⁻¹. Each six-coordinate Mn atom is bonded to a phenanthroline ligand and is also linked through $H_2PO_2^-$ groups to four other Mn atoms so that a puckered net is obtained parallel to the bc plane.

Introduction. The title compound was obtained by the method of Sala-Pala, Kergoat & Everchais (1972) as yellow laths elongated along c . Equi-inclination Weissenberg photographs of the levels 0–7 kl and $h0$ –7 l were recorded from two fragments (each 0.1 × 0.3 × 0.3 mm), and were scanned by the SRC Microdensitometer Service, Daresbury. The structure was solved by the heavy-atom method after the data had been corrected for absorption. Twelve of the strongest peaks in a difference synthesis, weighted to emphasize low-angle reflections, at R 0.069 were clearly assign-

This system is described by GS as a bifurcated hydrogen-bond system where the same H atom of water is involved in both bonds. Our results indicate, however, a participation of both water H atoms in the two bonds. The maximum deviation of the atoms from the plane of the phenyl ring is 0.01 Å. Both carboxyl groups are planar (Table 5); their planes make angles of 78 and 34° with the phenyl ring and the angle between them is 67°.

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able to the H atoms; they occurred at distances of 1.38 to 1.55 Å from P and 0.94 to 1.17 Å from C. In the last cycles, the H atoms were included at calculated positions (d_{P-H} 1.40, d_{C-H} 1.05 Å, U_H 0.05 Å²). Full-matrix least-squares refinement with anisotropic thermal parameters for all non-hydrogen atoms converged at R 0.063, R_w 0.065 [weights given by $w = 1/(1 + 0.0116F^2)$]; 190 parameters; 1657 unique reflections above background]. The final atomic coordinates are given in Table 1, and distances and angles in Table 2. Fig. 1 illustrates the numbering scheme. G. M. Sheldrick's *SHELX* 76 system was used in all calculations.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Lending Library Division as Supplementary Publication No. SUP 33856 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. In the 2,2'-bipyridyl analogue (Weakley, 1978) the Mn atoms are linked by double Mn—O—P—O—Mn bridges in a chain structure. The substitution of the bulkier and more rigid ligand 1,10-phenanthroline for 2,2'-bipyridyl is accompanied by a

change in both space group and topology. The Mn atoms retain distorted octahedral coordination but are now each linked by single Mn—O—P—O—Mn bridges (Fig. 2) to the four nearest Mn atoms related to them by the screw and glide operations so that a layer network, puckered about the *bc* plane, is formed. Bond lengths in the MnN₂O₄ group (Table 2) are similar to those in Mn(bpy)(H₂PO₂)₂ (Weakley, 1978) and Mn(phen)(acac)₂ (Stephens, 1977). The dimensions of the H₂PO₂⁻ groups are normal (Williams, 1966; Matsuzaki & Iitaka, 1969; Galigné & Dumas, 1973). The phenanthroline ligand shows no unusual features, the variation in the C—N and C—C lengths (Table 2) following the pattern observed in other phenanthroline complexes (Scaringe, Singh, Eckberg, Hatfield & Hodgson, 1975; Lim, Sinn & Martin, 1976; Stephens, 1977). The maximum deviation of an atom from the phenanthroline mean plane is 0.047 Å [C(3)]. This plane is, however, tilted 7.2° from the plane of the Mn—N bonds, apparently to minimize contacts between phenanthroline molecules in adjacent layers

Table 1. Atomic coordinates ($\times 10^4$)

	<i>x</i>	<i>y</i>	<i>z</i>
Mn	1737 (1)	2365 (1)	2821 (1)
P(1)	760 (2)	-630 (2)	2240 (2)
P(2)	1185 (2)	2035 (2)	5085 (1)
O(1)	1040 (6)	567 (5)	2798 (5)
O(2)	72 (6)	-1537 (5)	2628 (5)
O(3)	2158 (6)	2357 (5)	4625 (4)
O(4)	1208 (6)	2804 (5)	6023 (5)
N(1)	4074 (6)	1825 (5)	3498 (5)
N(2)	3086 (6)	4096 (5)	3069 (5)
C(1)	4589 (8)	704 (7)	3722 (7)
C(2)	6026 (8)	443 (7)	4296 (7)
C(3)	7001 (7)	1345 (7)	4617 (6)
C(4)	6519 (7)	2542 (7)	4375 (6)
C(5)	5038 (7)	2732 (6)	3814 (6)
C(6)	7463 (9)	3535 (9)	4669 (7)
C(7)	6952 (8)	4678 (8)	4405 (7)
C(8)	4490 (7)	3934 (6)	3553 (6)
C(9)	5474 (8)	4877 (7)	3869 (6)
C(10)	4893 (8)	6080 (7)	3638 (7)
C(11)	3469 (9)	6248 (7)	3127 (7)
C(12)	2597 (8)	5227 (7)	2859 (7)

Table 2. Interatomic distances (Å) and angles (°)

Mn—O(1)	2.144 (5)	N(2)—C(12)	1.330 (8)
Mn—O(2)	2.134 (5)	C(1)—C(2)	1.388 (10)
Mn—O(3)	2.191 (5)	C(2)—C(3)	1.365 (10)
Mn—O(4 ⁱⁱ)	2.158 (5)	C(3)—C(4)	1.413 (10)
Mn—N(1)	2.315 (5)	C(4)—C(5)	1.418 (9)
Mn—N(2)	2.351 (5)	C(4)—C(6)	1.434 (10)
P(1)—O(1)	1.481 (5)	C(5)—C(8)	1.447 (8)
P(1)—O(2)	1.491 (5)	C(6)—C(7)	1.344 (11)
P(2)—O(3)	1.491 (5)	C(7)—C(9)	1.419 (10)
P(2)—O(4)	1.492 (5)	C(8)—C(9)	1.403 (9)
N(1)—C(1)	1.337 (8)	C(9)—C(10)	1.451 (10)
N(1)—C(5)	1.366 (8)	C(10)—C(11)	1.348 (10)
N(2)—C(8)	1.350 (8)	C(11)—C(12)	1.415 (10)
N(1)—Mn—N(2)	71.0 (2)	C(4)—C(5)—N(1)	123.6 (6)
O(1)—Mn—O(3)	84.4 (2)	C(4)—C(5)—C(8)	119.3 (6)
N(1)—Mn—O(1)	94.2 (2)	C(6)—C(4)—C(5)	119.7 (6)
N(2)—Mn—O(3)	93.3 (2)	C(4)—C(6)—C(7)	121.1 (6)
N(2)—Mn—O(1)	165.1 (2)	C(6)—C(7)—C(9)	120.0 (6)
N(1)—Mn—O(2 ⁱ)	160.2 (3)	C(7)—C(9)—C(8)	122.1 (6)
O(3)—Mn—O(4 ⁱⁱ)	173.9 (3)	C(9)—C(8)—C(5)	117.7 (5)
O(1)—P(1)—O(2)	117.7 (3)	N(2)—C(8)—C(9)	123.8 (6)
O(3)—P(2)—O(4)	117.7 (3)	C(8)—C(9)—C(10)	116.0 (6)
C(1)—N(1)—C(5)	116.3 (6)	C(9)—C(10)—C(11)	120.4 (6)
C(2)—C(1)—N(1)	124.0 (6)	C(10)—C(11)—C(12)	118.0 (6)
C(3)—C(2)—C(1)	120.2 (6)	C(11)—C(12)—N(2)	124.1 (6)
C(4)—C(3)—C(2)	118.8 (6)	C(8)—N(2)—C(12)	117.7 (5)
C(5)—C(4)—C(3)	117.2 (6)		

Distances between non-bonded atoms

C(1)—C(2 ⁱⁱⁱ)	3.25 (2)	O(2)—C(12 ^{iv})	3.30 (2)
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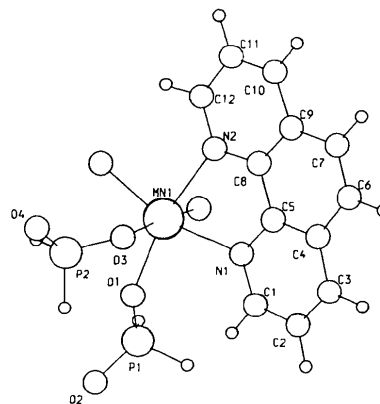
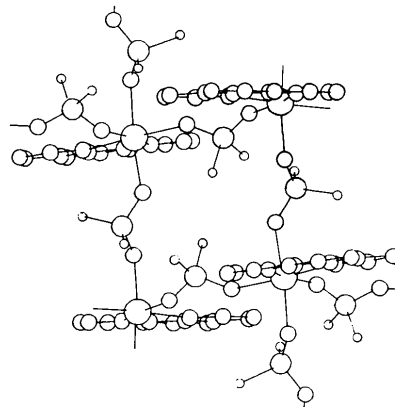
Symmetry code: (i) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$; (iii) $1 - x, -y, 1 - z$; (iv) $-x, -\frac{1}{2} + y, \frac{1}{2} - z$.

Fig. 1. Coordination about one Mn atom.

Fig. 2. Part of one layer (a^* projection).

related by the screw axis at $(\frac{1}{2}, y, \frac{1}{4})$. There are no unusually short distances between non-bonded atoms.

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Structure of Tris(pyridine)mercury(II) Bis(trifluoroacetate)

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Abstract. $\text{Hg}(\text{C}_5\text{H}_5\text{N})_3(\text{CO}_2\text{CF}_3)_2$, $\text{C}_{19}\text{H}_{15}\text{F}_6\text{HgN}_3\text{O}_4$, monoclinic, $P2_1/c$, $a = 13.31$ (1), $b = 20.24$ (2), $c = 8.97$ (1) Å, $\beta = 110.2$ (1)°, $Z = 4$, $D_c = 1.94$ g cm⁻³. The structure has been determined from 1863 diffractometer intensities (Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å) to $R = 0.068$. There is sevenfold coordination around Hg (Hg–N, 2.28, 2.27 and 2.36 Å; Hg–O, 2.66, 2.72, 2.79 and 2.53 Å). The N–Hg–N angles are 161.0, 99.2 and 99.5°. A complex ion, $\text{Hg}(\text{C}_5\text{H}_5\text{N})_3^{2+}$, appears to be formed.

Introduction. Acicular crystals of the title compound occur as the initial product of the reaction between pyridine and mercury(II) trifluoroacetate. Crystals, removed from pyridine and dried, effloresced on exposure to the atmosphere, with a weight loss corresponding to one molecule of pyridine per formula unit. Powder photographs showed that the residue was bis(pyridine)mercury(II) bis(trifluoroacetate). Sealing the crystals in Lindemann-glass tubes prevented efflorescence, but nevertheless decomposition occurred on exposure to X-rays. With Cu $K\alpha$ radiation ($\lambda = 1.542$ Å) the cell dimensions and space group were obtained from Weissenberg photographs. It was not possible to measure the crystal density as it changed rapidly due to decomposition in all suspension liquids. Intensity measurements were made with one crystal of dimensions $0.25 \times 0.25 \times 0.85$ mm using graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). The crystal was set about c on the Stoe STADI 2 automatic two-circle diffractometer. 2386 intensities in the range $2\theta < 40^\circ$ were measured; 1863 for which $I > 3\sigma(I)$ were used for the structure determination.

Absorption corrections ($\mu = 70.2$ cm⁻¹) and data reduction were carried out using the *SHELX* suite of programs (Sheldrick, 1976); the remainder of the calculations were performed with the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Patterson maps were used to obtain the Hg coordinates and a Fourier F_o map phased on Hg revealed the rest of the non-H atoms. In the least-squares refinement, anisotropic U_{ij} for Hg, isotropic U for other non-H atoms and positions of all non-H atoms were refined alternately with scale factors for each layer of the reciprocal lattice measured on the diffractometer. Inclusion of H atoms at positions calculated with C–H = 1.00 Å and $U = 0.05$ Å² was found to improve the agreement; the R value at which convergence was complete was 0.068. Final coordinates and temperature factors are given in Table 1.* Atomic scattering factors for Hg atoms (including anomalous dispersion) were taken from Cromer & Mann (1968); those for all other atoms were from *International Tables for X-ray Crystallography* (1962).

Discussion. One unit of the structure is shown in Fig. 1, the bond distances and angles around Hg are given in Table 2.*

* Lists of structure factors and bond lengths and angles around non-Hg atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33857 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.