back donation from the W atom. The comparative shortness of the W—As(2) bond compared to the W—As(1) bond can be explained by consideration of their respective *trans* ligands. The Br(1) atom *trans* to the As(2) ligand is not as strong a $d\pi$ acceptor as is the P atom which is *trans* to As(1), which results in a greater back donation to As(2) and a shortening of the W—As(2) bond length.

The room temperature ^{1}H NMR spectrum in the arsenic methyl region is not consistent with the general geometry shown in Fig. 1. The solution ^{1}H NMR (90 MHz) data shows four well resolved peaks at 190 (1) K, which indicates that the structure is static, consistent with the solid state results. However, at 219 (1) K the four peaks coalesce into one peak. The single resonance line becomes very sharp at 298 K indicative of a fast-exchange limiting spectrum. In the related complex $(L–L)W(CO)_2I_2P$-$(OCH_3)_3$ (Mihichuk, Giesinger, Robertson & Barton, 1987), four peaks were observed at 216 K, which coalesce into two peaks at 221 K and then to one at 239 K. The reason that the title compound remains fluxional at comparatively lower temperature is not clear, but presumably involves the relative radius of the halogen atoms.

References

BEAUCHAMP, A. L., BELANGER-GARIEPY, F. & ARALI, S. (1985). *Inorg. Chem.* **24**, 1860–1863.

COTTON, F. A., FALVELLO, L. R. & MEADOWS, J. H. (1985). *Inorg. Chem.* **24**, 514–517.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

CROW, J. P., CULLEN, W. R., HERRING, F. G., SAMS, J. R. & TAPPING, R. L. (1971). *Inorg. Chem.* **10**, 1616–1623.

DREW, M. G. B. & WILKINS, J. D. (1973). *J. Chem. Soc. Dalton Trans.* pp. 2664–2669.

HOFFMANN, R., BEIER, E. L., MUETTERTIES, E. L. & ROSSI, A. R. (1977). *Inorg. Chem.* **16**, 511–522.

MERCER, A. & TROTTER, J. (1974). *Can. J. Chem.* **52**, 3331–3336.

MIHICHUK, L. M., GIESINGER, C. L., ROBERTSON, B. E. & BARTON, R. J. (1987). *Can. J. Chem.* **65**, 2634–2638.

MIHICHUK, L. M., PIZZEY, M., ROBERTSON, B. E. & BARTON, R. J. (1986). *Can. J. Chem.* **64**, 991–995.

MUETTERTIES, E. L. & GUGGENBERGER, L. J. (1974). *J. Am. Chem. Soc.* **96**, 1748–1756.

PORAI-KOSHITS, M. A. & ASLANOV, L. A. (1972). *J. Struct. Chem.* **13**, 244–253.

STEWART, J. M. & HALL, S. R. (1986). *XTAL85*. Tech. Rep. TR-1675. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

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Structures of [cis-1,2-Bis(diphenylphosphino)ethylene]tetracarbonylmolybdenum(0) (1) and Bis[cis-1,2-bis(diphenylphosphino)ethylene]dicarbonylmolybdenum(0) (2)

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Abstract. (1) $C_{30}H_{22}MoP_2O_4$, $M_r = 604.4$, orthorhombic, *Pbcn*, $a = 15.884$ (5), $b = 16.719$ (3), $c = 20.807$ (4) Å, $V = 5526$ (2) Å^3, $Z = 8$, $D_m = 1.5$ (1), $D_x = 1.45$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7093 Å, $\mu = 0.30$ mm^{-1}, $F(000) = 1224$, $T = 298$ K, final $R = 0.032$ for 2708 observed reflections. (2) $C_{54}H_{44}$-MoP_4O_2, $M_r = 944.8$, monoclinic, *C2/c*, $a = 21.644$ (4), $b = 11.361$ (2), $c = 18.708$ (5) Å, $\beta = 99.02$ (2)°, $V = 4544$ (2) Å^3, $Z = 4$, $D_m = 1.4$ (1), $D_x = 1.38$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7093 Å, $\mu = 0.46$ mm^{-1}, $F(000) = 1944$, $T = 298$ K, final $R = 0.033$ for 3040 observed reflections. The two Mo—P lengths for compound (1) are almost the same, but the two Mo—P lengths in the same chelate ring for compound (2) are significantly different. The two

mutually *cis* CO groups in compound (1) have larger Mo—C distances than those in compound (2) owing to the presence of only two stronger π-acceptor CO ligands in the latter compound. The bite angle, $\angle$P—Mo—P, of compound (1) is slightly smaller than that of compound (2).

Introduction. The crystal structures of some bidentate phosphine derivatives of molybdenum(0) carbonyl, [bis(diphenylphosphino)methane]tetracarbonyl-molybdenum(0), $Mo(CO)_4$(dppm) (Cheung, Lai & Mok, 1971), [1,2-bis(diphenylphosphino)ethane]-tetracarbonylmolybdenum(0), $Mo(CO)_4$(dppe) (Bernal, Reisner, Dobson & Dobson, 1986), bis[1,3-bis-(diphenylphosphino)propane]dicarbonyl-molybdenum(0), $Mo(CO)_2$(dppp)$_2$ (Chow, Wang, Sheu & Peng, 1986), [1,3-bis(diphenylphosphino)pro-

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pane]tetracarbonylmolybdenum(0), Mo(CO)$_4$(dppp), and[1,4-bis(diphenylphosphino)butane]tetracarbonyl-molybdenum(0), Mo(CO)$_4$(dppb) (Ueng & Hwang, 1991), have been studied to elucidate the detailed three-dimensional architecture of the molecule, to study the chelate effect for the bidentate phosphine ligand, and to investigate the ring effect by tuning the number of the chelate ring. In order to make a comparison with other bidentate phosphine derivatives, especially with Mo(CO)$_4$(dppe), and as part of a series of studies on bidentate phosphine derivatives of molybdenum carbonyl, the two cis-1,2-bis(diphenylphosphino)ethylene derivatives, Mo(CO)$_4$(cis-vpp) and Mo(CO)$_2$(cis-vpp)$_2$, were synthesized and the crystal structures determined.

Experimental. (1) Compound was synthesized from Mo(CO)$_6$ and cis-vpp in n-decane (King & Eggers, 1968; Grim, Briggs, Barth, Tolman & Jesson, 1974). Crystal was prepared from CH$_2$Cl$_2$/n-hexane by diffusion method. Crystal 0·17 × 0·38 × 0·50 mm. CAD-4 diffractometer. Intensities measured by θ–2θ technique. Unit cell: 24 reflections, 2θ range 19·48–22·84°. D_m by flotation (CH$_2$Cl$_2$/CH$_3$Cl). $2\theta_{max}$ = 49·8°. Ranges of h, k, l: 0 to 18, 0 to 19, 0 to 24, respectively. Three standard reflections monitored every 2 h: variation on $I < 2\%$, 4877 unique reflections, R_{int} = 0·010, 2708 observed with $I \geq 2\cdot5\sigma(I)$, the quantity minimized $w(KF_o - F_c)^2$, $R(F)$ = 0·032, wR = 0·025, S = 1·40. Weighting scheme from counting statistics. Structure solved by heavy-atom method. H atoms of phenyl groups calculated after isotropic refinement, the others found in difference Fourier map. $(\Delta/\sigma)_{max}$ = 0·076. Peaks in final ΔF map 0·360 to −0·250 e Å^{-3}. Secondary-extinction coefficient 0·33 (2) (length in μm). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computing programs: *NRCC SDP VAX* package (Gabe & Lee, 1981), *ORTEP*II (Johnson, 1976) and *Structure Determination Package* Enraf–Nonius (1979).

(2) Compound was synthesized from one mole of Mo(CO)$_6$ and two moles of cis-vpp in n-decane (King, Houk & Kapoor, 1969). Crystal was prepared from CH$_2$Cl$_2$/n-hexane with diffusion method. Crystal 0·10 × 0·25 × 0·65 mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range 20·96 to 26·12°. D_m by flotation (CH$_2$Cl$_2$/CH$_3$Cl). $2\theta_{max}$ = 49·8°. Ranges of h, k, l: −25 to 25, 0 to 13, 0 to 22, respectively. Three standard reflections monitored every 2 h: variation on $I < 4\%$, 3988 unique reflections, R_{int} = 0·015, 3040 observed with $I \geq 2\sigma(I)$, $R(F)$ = 0·033, wR = 0·027, S = 1·95. Weighting scheme from counting statistics. Structure solved by heavy-atom method. $(\Delta/\sigma)_{max}$ = 0·001. Peaks in final ΔF map 0·470 to −0·570 e Å^{-3}. Secondary-extinction coefficient 1·0 (1) (length in μm). Other details as for (1).

Discussion. Atomic positional parameters and equivalent isotropic temperature factors are listed in Table 1.* Selected bond lengths and bond angles are shown in Table 2. The molecular structures are shown in Fig. 1.

The molecules consist of a central Mo atom bonded to carbonyl groups and a bidentate phosphine ligand having two P atoms as donors. The coordination around the metal is a distorted octa-

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* Lists of structure factors, anisotropic thermal parameters, complete bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53583 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
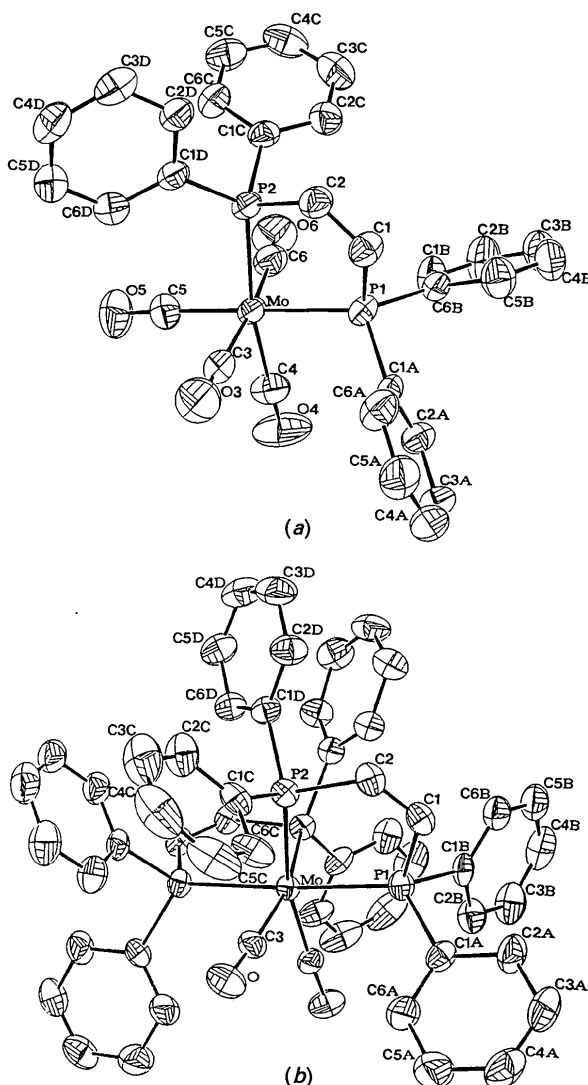

(a)

(b)

Fig. 1. Molecular structures of (a) [cis-1,2-bis(diphenylphosphino)-ethylene]tetracarbonylmolybdenum(0) (1) and (b) bis[cis-1,2-bis(diphenylphosphino)ethylene]dicarbonylmolybdenum(0) (2).

Table 1. *Atomic positional parameters and equivalent isotropic temperature factors of* (1) *and* (2)

$$B_{iso} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{iso}(\text{Å}^2)$
Compound (1)				
Mo	0·79383 (3)	0·16410 (2)	0·81827 (2)	2·94 (2)
P1	0·73588 (8)	0·05905 (7)	0·74498 (6)	3·16 (6)
P2	0·72721 (8)	0·07057 (7)	0·89655 (5)	3·15 (6)
C1	0·6593 (3)	−0·0002 (3)	0·7894 (2)	3·7 (2)
C2	0·6576 (3)	0·0034 (3)	0·8534 (2)	3·7 (2)
C3	0·6832 (3)	0·2236 (3)	0·8051 (2)	3·8 (3)
C4	0·8481 (4)	0·2231 (3)	0·7460 (3)	4·8 (3)
C5	0·8282 (3)	0·2495 (3)	0·8784 (2)	4·1 (3)
C6	0·9076 (3)	0·1163 (3)	0·8387 (2)	4·3 (3)
C1a	0·6763 (3)	0·0965 (3)	0·6764 (2)	3·3 (2)
C2a	0·7186 (3)	0·1152 (3)	0·6205 (2)	3·9 (2)
C3a	0·6756 (4)	0·1467 (3)	0·5685 (2)	5·0 (3)
C4a	0·5906 (4)	0·1590 (4)	0·5715 (3)	5·7 (3)
C5a	0·5488 (4)	0·1412 (4)	0·6263 (3)	5·5 (3)
C6a	0·5899 (3)	0·1101 (3)	0·6789 (2)	4·6 (3)
C1b	0·7978 (3)	−0·0209 (3)	0·7079 (2)	3·1 (2)
C2b	0·7592 (3)	−0·0833 (3)	0·6754 (2)	4·6 (3)
C3b	0·8069 (4)	−0·1436 (3)	0·6485 (3)	5·4 (3)
C4b	0·8926 (4)	−0·1417 (3)	0·6531 (3)	5·5 (3)
C5b	0·9304 (3)	−0·0808 (4)	0·6838 (3)	6·1 (3)
C6b	0·8844 (3)	−0·0199 (3)	0·7111 (2)	4·6 (3)
C1c	0·8003 (3)	0·0004 (3)	0·9350 (2)	3·1 (2)
C2c	0·8296 (3)	−0·0660 (3)	0·9031 (2)	4·1 (3)
C3c	0·8910 (4)	−0·1135 (3)	0·9302 (3)	5·1 (3)
C4c	0·9245 (3)	−0·0929 (3)	0·9888 (3)	5·3 (3)
C5c	0·8959 (4)	−0·0274 (4)	1·0211 (3)	4·9 (3)
C6c	0·8342 (3)	0·0191 (3)	0·9946 (2)	4·0 (3)
C1d	0·6624 (3)	0·1014 (3)	0·9651 (2)	3·1 (2)
C2d	0·6187 (3)	0·0453 (3)	1·0006 (2)	3·7 (2)
C3d	0·5734 (3)	0·0686 (3)	1·0539 (2)	4·4 (3)
C4d	0·5706 (3)	0·1474 (4)	1·0718 (2)	4·5 (3)
C5d	0·6139 (4)	0·2036 (3)	1·0375 (2)	4·8 (3)
C6d	0·6593 (3)	0·1806 (3)	0·9836 (2)	4·0 (3)
O3	0·6229 (2)	0·2588 (2)	0·7993 (2)	6·5 (2)
O4	0·8811 (3)	0·2547 (3)	0·7047 (2)	8·5 (3)
O5	0·8492 (3)	0·3007 (2)	0·9118 (2)	6·4 (2)
O6	0·9729 (2)	0·0959 (3)	0·8537 (2)	6·9 (2)
Compound (2)				
Mo	0	0·35050 (4)	0	1·96 (2)
P1	−0·08509 (4)	0·3562 (1)	0·14803 (5)	2·48 (4)
P2	0·04399 (4)	0·4965 (1)	0·16925 (5)	2·43 (4)
C1	−0·0673 (2)	0·4576 (3)	0·0785 (2)	2·9 (2)
C2	−0·0141 (1)	0·5158 (3)	0·0881 (2)	2·8 (2)
C3	0·0491 (1)	0·2295 (3)	0·2109 (2)	2·6 (2)
C1a	−0·1072 (2)	0·2264 (3)	0·0905 (2)	2·8 (2)
C2a	−0·1559 (2)	0·2335 (4)	0·0324 (2)	3·9 (2)
C3a	−0·1693 (2)	0·1384 (4)	−0·0138 (2)	4·5 (2)
C4a	−0·1355 (2)	0·0364 (4)	−0·0026 (2)	4·8 (2)
C5a	−0·0882 (2)	0·0273 (4)	0·0551 (2)	5·2 (2)
C6a	−0·0746 (2)	0·1227 (4)	0·1013 (2)	4·1 (2)
C1b	−0·1627 (1)	0·4076 (4)	0·1624 (2)	2·8 (2)
C2b	−0·1978 (2)	0·3351 (4)	0·2016 (2)	3·7 (2)
C3b	−0·2543 (2)	0·3745 (5)	0·2177 (2)	4·7 (3)
C4b	−0·2761 (2)	0·4846 (5)	0·1961 (2)	4·9 (2)
C5b	−0·2421 (2)	0·5567 (4)	0·1576 (2)	4·3 (2)
C6b	−0·1863 (2)	0·5162 (4)	0·1401 (2)	3·5 (2)
C1c	0·1113 (2)	0·4546 (4)	0·1247 (2)	3·0 (2)
C2c	0·1669 (2)	0·5183 (4)	0·1351 (2)	4·2 (2)
C3c	0·2165 (2)	0·4840 (5)	0·1008 (2)	5·8 (3)
C4c	0·2102 (2)	0·3877 (5)	0·0565 (3)	6·1 (3)
C5c	0·1561 (2)	0·3230 (4)	0·0458 (2)	5·3 (3)
C6c	0·1065 (2)	0·3558 (4)	0·0802 (2)	4·0 (2)
C1d	0·0623 (1)	0·6498 (3)	0·1929 (2)	2·7 (2)
C2d	0·0617 (2)	0·7412 (4)	0·1436 (2)	3·7 (2)
C3d	0·0707 (2)	0·8560 (4)	0·1665 (2)	4·6 (2)
C4d	0·0816 (2)	0·8819 (4)	0·2394 (3)	4·6 (2)
C5d	0·0849 (2)	0·7926 (4)	0·2889 (2)	4·0 (2)
C6d	0·0747 (2)	0·6780 (3)	0·2660 (2)	3·1 (2)
O	0·0808 (1)	0·1560 (2)	0·1919 (1)	4·2 (1)

Table 2. *Selected bond lengths* (Å) *and angles* (°) *of* (1) *and* (2)

Compound (1)

Mo—P1	2·501 (1)	P2—C2	1·814 (5)
Mo—P2	2·494 (1)	P2—C1c	1·833 (5)
Mo—C3	2·038 (5)	P2—C1d	1·833 (5)
Mo—C4	1·993 (5)	C1—C2	1·332 (7)
Mo—C5	1·976 (5)	C3—O3	1·129 (6)
Mo—C6	2·022 (5)	C4—O4	1·138 (7)
P1—C1	1·821 (5)	C5—O5	1·152 (6)
P1—C1a	1·823 (5)	C6—O6	1·135 (7)
P1—C1b	1·830 (5)		
P1—Mo—P2	78·56 (4)	Mo—P1—C1b	124·9 (2)
P1—Mo—C3	86·8 (1)	C1—P1—C1a	103·8 (2)
P1—Mo—C4	92·7 (2)	C1—P1—C1b	100·2 (2)
P1—Mo—C5	174·4 (2)	C1a—P1—C1b	101·5 (2)
P1—Mo—C6	100·4 (2)	Mo—P2—C2	108·9 (2)
P2—Mo—C3	91·6 (1)	Mo—P2—C1c	114·7 (2)
P2—Mo—C4	170·5 (2)	Mo—P2—C1d	124·8 (2)
P2—Mo—C5	99·0 (1)	C2—P2—C1c	101·9 (2)
P2—Mo—C6	89·7 (2)	C2—P2—C1d	102·6 (2)
C3—Mo—C4	91·7 (2)	C1c—P2—C1d	101·3 (2)
C3—Mo—C5	88·3 (2)	P2—C2—C1	120·6 (4)
C3—Mo—C6	172·9 (2)	P1—C1—C2	119·8 (4)
C4—Mo—C5	90·0 (2)	Mo—C3—O3	177·4 (4)
C4—Mo—C6	88·2 (2)	Mo—C4—O4	177·6 (5)
C5—Mo—C6	84·6 (2)	Mo—C5—O5	177·8 (4)
Mo—P1—C1	108·5 (2)	Mo—C6—O6	173·4 (5)
Mo—P1—C1a	115·3 (2)		

Compound (2)

Mo—P1	2·436 (1)	P2—C2	1·827 (3)
Mo—P2	2·528 (1)	P2—C1c	1·851 (3)
Mo—C3	1·948 (4)	P2—C1d	1·826 (4)
P1—C1	1·822 (4)	C1—C2	1·316 (5)
P1—C1a	1·845 (4)	C3—O	1·171 (4)
P1—C1b	1·838 (3)		
P1—Mo—P1	176·94 (4)	Mo—P1—C1	111·0 (1)
P1—Mo—P2	79·85 (3)	Mo—P1—C1a	122·1 (1)
P1—Mo—P2	98·11 (3)	Mo—P1—C1b	119·4 (1)
P1—Mo—C3	96·6 (1)	C1—P1—C1a	98·9 (2)
P1—Mo—C3	85·6 (1)	C1—P1—C1b	101·8 (2)
P1—Mo—P2	98·11 (3)	C1a—P1—C1b	100·3 (2)
P1—Mo—P2	79·85 (3)	Mo—P2—C2	107·5 (1)
P1—Mo—C3	85·6 (1)	Mo—P2—C1c	119·8 (1)
P1—Mo—C3	96·6 (1)	Mo—P2—C1d	124·7 (1)
P2—Mo—P2	98·00 (4)	C2—P2—C1c	98·3 (2)
P2—Mo—C3	87·7 (1)	C2—P2—C1d	100·5 (2)
P2—Mo—C3	164·9 (1)	C1c—P2—C1d	101·4 (2)
P2—Mo—C3	164·9 (1)	P1—C1—C2	119·9 (2)
P2—Mo—C3	87·7 (1)	P2—C2—C1	121·7 (3)
C3—Mo—C3	90·3 (1)	Mo—C3—O	175·7 (3)

hedron in (1) and (2), the equatorial plane being formed by two mutual *cis*-CO and the two P atoms for compound (1) and by the two P atoms of one of the bidentate ligands and two atoms (P and C) *trans* to it for compound (2). The deviations of the Mo atom from this plane are 0·020 (2) and 0·019 (6) Å. The dihedral angles between the three mutually perpendicular planes of the octahedron are 92·5 (1), 96·0 (1) and 80·5 (1)° for (1) and 105·9 (1), 85·1 (1) and 89·6 (2)° for (2). Distortions from the theoretical octahedron geometry are largely due to the formation of the five-membered chelate ring and also to the differences between the Mo—P [2·436 (1)–2·528 (1) Å] and Mo—CO bond lengths [1·948 (4)–2·038 (5) Å].

The two Mo—P lengths for (1) are almost the same [2·501 (1) and 2·494 (1) Å], which is expected since the environments about the two P atoms are similar. But the two Mo—P lengths in the same bidentate chelate ligand for (2) are significantly different [2·528 (1) and 2·436 (1) Å], which is also expected since the *trans* position for the longer one is the carbonyl group which is a stronger π-acceptor group than the phosphine group which is *trans* for

the shorter (Cotton & Kraihanzel, 1962; Cotton, Darensbourg, Klein & Kolthammer, 1982). There are four carbonyl groups in (1). The Mo—C lengths for the mutually *cis* CO groups (*trans* to the bidentate phosphine ligand) are shorter than that of the mutually *trans* one [1·993 (5), 1·976 (5) vs 2·022 (5), 2·038 (5) Å]. But the two mutually *cis* CO groups in (1) have larger Mo—C distances than in (2) [1·948 (4) Å]. This trend which is also found in $Mo(CO)_4(dppp)$ (Ueng & Hwang, 1991) and $Mo(CO)_2(dppp)_2$ (Chow, Wang, Sheu & Peng, 1986) [1·968 (5) vs 1·913 (8), 1·938 (9) Å] may be attributed to the presence of only two stronger π-acceptor CO ligands in the latter compound instead of four in the former. The bite angle, $\angle$P—Mo—P, of (1) is slightly smaller than that of (2) [78·56 (4) vs 79·85 (3) Å]. This order does not coincide the fact that $\angle$P—Mo—P in $Mo(CO)_4(dppp)$ is larger than that in $Mo(CO)_2(dppe)_2$ [89·74 (4) vs 85·55 (8), 86·76 (8) Å]. The ranges of the P—C bond lengths [1·814 (5) to 1·851 (3) Å] and the C—C bond lengths in the phenyl groups [1·366 (8) to 1·402 (5)] for both title compounds are reasonable. From the C=C bond lengths [1·332 (7); 1·316 (5) Å] and the $\angle$P—C—C bond angles [119·8 (4), 120·6 (4); 119·9 (2), 121·7 (3)°] of the chelate rings, it is obvious that the distortions of the C=C double bonds are negligible when the bidentate ligands are coordinated to the central metal atom.

Neither title compound has intermolecular contacts of structural significance. Compound (1) has pseudo mirror molecular symmetry through Mo, the middle point of C1—C2 and the two *trans* carbonyl groups. Compound (2) has a crystallographic twofold axis that coincides with the bisector of $\angle$C3—Mo—C3 at $[0, y, \frac{1}{4}]$. The dihedral angles between the two phenyl groups at P1 for (1) and (2) are comparable [101·0 (2) vs 95·6 (2)°], and so are those at P2 [76·9 (2) vs 73·2 (2)°].

The fact that compound (1) and $Mo(CO)_4(dppe)$ have similar bite angles [78·56 (4); 80·2 (1)°] and have the same Mo—P bond lengths [2·501 (1), 2·494 (1); 2·500 (2), 2·495 (2) Å] suggests that there may be no significant difference from any major electronic effect between *cis*-vpp and dppe ligands.

References

BERNAL, I., REISNER, G. M., DOBSON, G. R. & DOBSON, C. B. (1986). *Inorg. Chim. Acta*, **121**, 199–206.

CHEUNG, K. K., LAI, T. F. & MOK, K. S. (1971). *J. Chem. Soc. A*, pp. 1644–1647.

CHOW, T. J., WANG, C.-Y., SHEU, S.-C. & PENG, S.-M. (1986). *J. Organomet. Chem.* **311**, 339–347.

COTTON, F. A., DARENSBOURG, D. J., KLEIN, S. & KOLTHAMMER, B. W. S. (1982). *Inorg. Chem.* **21**, 294–299.

COTTON, F. A. & KRAIHANZEL, C. S. (1962). *J. Am. Chem. Soc.* **84**, 4432–4444.

ENRAF-NONIUS (1979). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.

GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* A**37**, S339.

GRIM, S. O., BRIGGS, W. L., BARTH, R. C., TOLMAN, C. A. & JESSON, J. P. (1974). *Inorg. Chem.* **13**, 1095–1100.

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory Tennessee, USA.

KING, R. B. & EGGERS, C. A. (1968). *Inorg. Chim. Acta*, **2**, 33–38.

KING, R. B., HOUK, L. W. & KAPOOR, P. N. (1969). *Inorg. Chem.* **8**, 1792–1794.

UENG, C.-H. & HWANG, G.-Y. (1991). *Acta Cryst.* C**47**, 522–525.

Structure of Sodium Hydrogen Glutarate Dihydrate

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Abstract. $C_5H_7O_4^-.Na^+.2H_2O$, $M_r = 190\cdot1$, monoclinic, $P2_1/c$, $a = 5\cdot371$ (1), $b = 13\cdot367$ (1), $c = 11\cdot587$ (1) Å, $\beta = 93\cdot72$ (1)°, $V = 830\cdot0$ Å^3, $Z = 4$, $D_m = 1\cdot52$ (by flotation in heptane/iodobutane), $D_x = 1\cdot527$ g cm^{-3}, $\lambda(Cu\,K\alpha) = 1\cdot5418$ Å, $\mu = 16\cdot1$ cm^{-1}, $F(000) = 400$, $T = 293$ K, $R = 0\cdot034$ for 1602 unique