Table 2. *Main interatomic distances* (Å), *bond angles* (°) *and hydrogen-bond scheme* (Å,°) *in* $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$

$P(1)O_4$ tetrahedron

P(1)	O(*E*11)	O(*E*12)	O(*L*12)	O(*L*21)
O(*E*11)	1·490 (2)	2·575 (3)	2·555 (3)	2.472 (3)
O(*E*12)	120·2 (1)	1·480 (2)	2·482 (3)	2·549 (3)
O(*L*12)	110·4 (1)	106·4 (1)	1·619 (2)	2·515 (3)
O(*L*21)	105·3 (1)	110·7 (1)	102·4 (1)	1·617 (2)

$P(2)O_4$ tetrahedron

P(2)	O(*E*21)	O(*E*22)	O(*L*21)	O(*L*12)
O(*E*21)	1·492 (2)	2·574 (3)	2·550 (2)	2·474 (3)
O(*E*22)	119·5 (1)	1·488 (2)	2·480 (3)	2·552 (3)
O(*L*21)	110·9 (1)	106·6 (1)	1·603 (2)	2·515 (3)
O(*L*12)	105·4 (1)	110·5 (1)	102·7 (1)	1·617 (2)

2 × P(1)—P(2)	2·9503 (9)	P(1)—P(2)—P(1)	88·66 (3)
2 × P(2)—P(1)	2·9489 (9)	P(2)—P(1)—P(2)	88·50 (3)

P(1)—O(*L*12)—P(2)	131·4 (1)
P(2)—O(*L*21)—P(1)	132·8 (1)

$Na(1)O_6$ polyhedron		$Na(2)O_6$ polyhedron	
2 × Na(1)—O(*E*12)	2·327 (2)	2 × Na(2)—O(*E*12)	2·326 (2)
2 × Na(1)—O(*E*21)	2·589 (2)	2 × Na(2)—O(*E*21)	2·506 (2)
2 × Na(1)—O(*W*)	2·432 (3)	2 × Na(2)—O(*W*)	2·694 (3)

NH_3—$(CH_2)_2$—NH_3 group

N—C	1·484 (4)	C—C	1·513 (6)	N—C—C	110·7 (3)

Hydrogen bonds

	H—H		N—O	N—H···O
	O—H	H···O	O—O	O—H···O
N—H(1N)···O(*R*21)	0·92 (5)	1·94 (4)	2·860 (4)	178 (4)
N—H(2N)···O(*E*11)	0·81 (5)	2·02 (5)	2·281 (3)	169 (4)
N—H(3N)···O(*E*22)	0·85 (4)	1·98 (4)	2·797 (3)	159 (3)
O(*W*)—H(1*w*)···O(*E*11)	0·70 (5)	2·25 (5)	2·902 (4)	167 (5)
O(*W*)—H(2*w*)···O(*E*22)	0·93 (5)	1·91 (5)	2·818 (4)	164 (5)

P_4O_{12} groups are located around the twofold axes. They twist around the 2_1 axes, situated at $x = \frac{1}{2}$; $z = \frac{1}{4},\frac{3}{4}$, to form a channel with section (3 × 7 Å) perpendicular to the *c* axis.

The two crystallographically independent sodium atoms Na(1) and Na(2) both have sixfold coordination, formed by two water molecules and four external oxygen atoms belonging to four ring anions $P_4O_{12}^{4-}$. The $Na(1)O_6$ and $Na(2)O_6$ distorted octahedra link together through the O(*E*12)—O(*E*21)—O(*W*) faces to form an infinite one-dimensional chain parallel to the *c* axis, and are located at the centre of the channel (Fig. 2).

The organic group, present as the dication $(CH_2)_2(NH_3)_2^{2+}$, has twofold symmetry. The groups alternate with P_4O_{12} rings and NaO_6 polyhedra to build, by hydrogen bonds, a three-dimensional network. It should be noticed that only the external oxygen atoms of the ring anions and sodium polyhedra are involved in hydrogen bonds; the oxygen atoms O(*E*11) and O(*E*22) taking part in this scheme act twice as acceptors. The potassium salt is isotypic with the following non-refined unit-cell dimensions: *a* = 7·92, *b* = 14·89, *c* = 13·11 Å, *β* = 92·03°.

References

Bdiri, M. & Jouini, A. (1989). *C.R. Acad. Sci.* **308**, 1353–1358.

Enraf–Nonius (1980). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Jouini, A. & Dabbabi, M. (1985). *C. R. Acad. Sci.* **301**, 1347–1350.

Jouini, A., Dabbabi, M. & Durif, A. (1985). *J. Solid State Chem.* **60**, 6–12.

Jouini, A. & Durif, A. (1983). *C. R. Acad. Sci.* **297**, 573–575.

Jouini, A., Soua, M. & Dabbabi, M. (1986). *J. Solid State Chem.* **69**, 135–144.

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Structure of [*o*-Bis(diisopropylphosphino)benzene]tetracarbonyltungsten(0)

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Abstract. $[W(CO)_4(C_{18}H_{32}P_2)]$, $M_r = 606{\cdot}30$, monoclinic, $P2_1/c$, $a = 17{\cdot}599\,(4)$, $b = 14{\cdot}672\,(2)$, $c = 19{\cdot}844\,(3)$ Å, $\beta = 102{\cdot}25\,(2)°$, $V = 4996\,(2)$ Å^3, $Z = 8$, $D_m = 1{\cdot}60\,(2)$, $D_x = 1{\cdot}61$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 4{\cdot}87$ mm^{-1}, $F(000) = 1152$, $T = 300$ K, $R = 0{\cdot}034$ for 6268 observed reflections. The structure of the title compound shows that the two phosphorus donors coordinate to the metal centre with *cis* geometry. The average W—P distance is 2·510 (2) Å. All the methyl groups point toward the $W(CO)_4$ moiety in order to minimize steric interaction.

Introduction. Unlike the bidentate behavior of bis(diphenylphosphino)ethane (dppe), the *o*-phenylenebisphosphines have the interesting feature of

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adopting a coplanar arrangement of two phosphorus sites and their attached aromatic ring. Part of our research interest in the coordination behavior of phosphines led us to study the metal complexes containing *o*-phenylenebisphosphine moieties. In this work, we have determined the structure of the title complex.

Experimental. The title complex was obtained from the reaction of $W(CO)_6$ and the corresponding ligand in refluxing toluene. Recrystallization from dichloromethane and methanol gave the title compound as a clear, colorless crystalline solid: IR (CH_2Cl_2) 2007, 1999, 1900, 1877 cm^{-1}; ^{31}P NMR 70·5 p.p.m.; elemental analysis, calculated for $C_{22}H_{32}O_4P_2W$: C 43·58, H 5·32; found: C 43·60, H 5·49%. Density measured by flotation.

Crystal 0·3 × 0·5 × 0·5 mm. CAD-4 diffractometer. Unit cell: 25 reflections, 2θ range 18·40 to 24·40°. $2\theta_{max} = 50°$. Ranges of h, k, l: 0 to 20, 0 to 17, −23 to 23, respectively. Three standard reflections every hour: variation < 2%. 8782 unique reflections, 6268 observed with $I > 2{\cdot}5\sigma(I)$. $R = 0{\cdot}034$, $S = 2{\cdot}382$. Weighting scheme from counting statistics. Structure solved by heavy-atom method. Two independent molecules were found and there was no symmetry relation between them. H atoms found in difference Fourier map after isotropic refinement and then refined. $\sum w(F_o - F_c)^2$ minimized. $(\Delta/\sigma)_{max} = 0{\cdot}026$. Peaks in final map ±1·01 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing program: NRCC *SDP/VAX* package (Gabe & Lee, 1981).

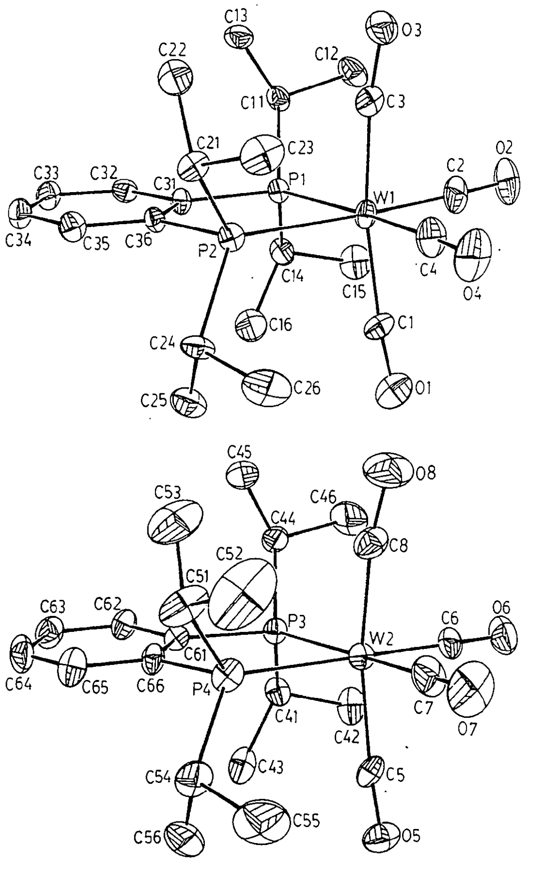

Fig. 1. *ORTEP* plots of the two independent molecules of [bis(diisopropylphosphino)benzene]tetracarbonyltungsten(0).

Table 1. *Atomic coordinates and isotropic temperature factors*

B_{iso} is the mean of the principal axes of the thermal ellipsoid.

	x	y	z	B_{iso}(Å^2)
W1	0·63388 (2)	0·17749 (2)	0·32468 (2)	3·28 (2)
P1	0·6356 (1)	0·2453 (1)	0·44113 (9)	2·9 (1)
P2	0·6011 (1)	0·3399 (1)	0·29082 (9)	3·2 (1)
C1	0·7466 (4)	0·1906 (5)	0·3329 (3)	4·4 (4)
O1	0·8139 (4)	0·1914 (4)	0·3359 (3)	7·8 (4)
C2	0·6573 (5)	0·0536 (5)	0·3605 (4)	5·3 (5)
O2	0·6712 (4)	−0·0199 (3)	0·3814 (3)	7·8 (4)
C3	0·5240 (5)	0·1428 (5)	0·3101 (3)	4·0 (4)
O3	0·4605 (3)	0·1127 (3)	0·3012 (3)	5·9 (3)
C4	0·6287 (5)	0·1381 (5)	0·2276 (4)	5·8 (5)
O4	0·6292 (4)	0·1154 (4)	0·1724 (3)	8·5 (4)
C11	0·5632 (5)	0·2037 (4)	0·4904 (3)	3·8 (4)
C12	0·5621 (5)	0·1016 (5)	0·4968 (4)	6·1 (5)
C13	0·4822 (5)	0·2387 (5)	0·4605 (4)	5·1 (5)
C14	0·7275 (5)	0·2440 (5)	0·5085 (4)	4·4 (4)
C15	0·7649 (5)	0·1493 (5)	0·5186 (4)	6·6 (5)
C16	0·7845 (5)	0·3112 (6)	0·4951 (4)	6·8 (5)
C21	0·5064 (5)	0·3662 (4)	0·2328 (3)	3·8 (4)
C22	0·4392 (5)	0·3564 (5)	0·2699 (4)	5·8 (5)
C23	0·4903 (5)	0·3057 (6)	0·1687 (4)	6·6 (6)
C24	0·6702 (4)	0·4084 (5)	0·2514 (3)	4·1 (4)
C25	0·7435 (6)	0·4319 (6)	0·3034 (4)	6·9 (6)
C26	0·6911 (6)	0·3590 (6)	0·1896 (4)	7·2 (6)
C31	0·6133 (4)	0·3655 (4)	0·4333 (3)	2·8 (4)
C32	0·6088 (4)	0·4204 (4)	0·4906 (3)	3·4 (4)
C33	0·5890 (5)	0·5097 (4)	0·4844 (3)	4·1 (4)
C34	0·5733 (5)	0·5505 (4)	0·4213 (4)	4·4 (5)
C35	0·5774 (5)	0·5006 (4)	0·3627 (3)	4·1 (4)
C36	0·5965 (4)	0·4079 (4)	0·3689 (3)	2·9 (3)
W2	0·19296 (2)	0·21655 (2)	0·33932 (2)	3·55 (2)
P3	0·1366 (1)	0·2403 (1)	0·44342 (9)	3·28 (9)
P4	0·0902 (2)	0·3319 (2)	0·2936 (1)	4·5 (1)
C5	0·2750 (4)	0·3117 (5)	0·3651 (4)	4·5 (4)
O5	0·3268 (3)	0·3621 (4)	0·3795 (3)	6·7 (3)
C6	0·2711 (5)	0·1284 (5)	0·3836 (4)	5·4 (5)
O6	0·3179 (4)	0·0751 (4)	0·4109 (3)	8·2 (4)
C7	0·2286 (6)	0·2027 (6)	0·2506 (4)	7·2 (6)
O7	0·2499 (4)	0·1937 (5)	0·2006 (3)	10·3 (5)
C8	0·1282 (5)	0·1088 (5)	0·3108 (4)	6·0 (5)
O8	0·0963 (4)	0·0388 (4)	0·2941 (3)	11·0 (5)
C41	0·1991 (4)	0·2902 (5)	0·5240 (3)	4·3 (4)
C42	0·2757 (5)	0·2409 (6)	0·5444 (4)	6·7 (5)
C43	0·2115 (5)	0·3920 (5)	0·5158 (4)	5·9 (5)
C44	0·0886 (5)	0·1426 (5)	0·4774 (4)	4·9 (4)
C45	0·0113 (6)	0·1172 (6)	0·4299 (5)	8·0 (6)
C46	0·1426 (6)	0·0606 (5)	0·4930 (5)	8·4 (7)
C51	0·0075 (6)	0·3043 (6)	0·2218 (4)	7·3 (6)
C52	0·0321 (8)	0·265 (1)	0·1619 (5)	17·0 (1)
C53	−0·0453 (6)	0·2394 (7)	0·2413 (5)	11·0 (8)
C54	0·1182 (5)	0·4445 (6)	0·2645 (4)	6·6 (5)
C55	0·1723 (8)	0·4371 (8)	0·2133 (6)	12·9 (9)
C56	0·1579 (7)	0·5039 (5)	0·3244 (6)	10·2 (8)
C61	0·0567 (4)	0·3226 (4)	0·4256 (3)	3·2 (4)
C62	0·0146 (5)	0·3461 (5)	0·4744 (3)	4·5 (4)
C63	−0·0420 (5)	0·4092 (5)	0·4630 (4)	5·4 (5)
C64	−0·0603 (5)	0·4511 (6)	0·3997 (4)	6·7 (5)
C65	−0·0217 (5)	0·4294 (5)	0·3496 (4)	5·3 (5)
C66	0·0373 (4)	0·3637 (5)	0·3613 (3)	3·8 (4)

Discussion. The *ORTEP* (Johnson, 1965) plot of the title complex is shown in Fig. 1. Atomic coordinates and isotropic temperature factors are given in Table

1.* Selected bond distances and angles are listed in Table 2. As shown in the *ORTEP* plot, the two P atoms coordinate to the metal center with an average W—P distance of 2·510 (2) Å, which is similar to those reported in $[(OC)_4WPPh_2CH(CH_2CH_2PPh_2)CH_2PPh_2]$ (1), 2·523 (2) and 2·522 (2) Å (Churchill, Rheingold & Keiter, 1981), and $[(CO)_4W\{Ph_2PNHN{=}C(Ph)CH_2PPh_2\}]$ (2), 2·531 (2) and 2·497 (1) Å (Al-Jibori, Hall, Hutton & Shaw, 1984). The P—W—P bond angle, 80·06 (6)°, is smaller than those in (1) [87·58 (6)°] and (2) [87·8 (1)°]. This observation is apparently due to the coplanar, rigid ligand, which forces the decrease in the P—W—P bond angle. The four atoms P1, C31, C36 and P2 are coplanar; distances from the least-squares plane [P1, C31, C36, P2] are P1 −0·001 (3), C31 0·015 (9), C36 −0·014 (9), and P2 0·001 (3) Å. As expected, the W atom is located in the same plane; its deviation from the plane is 0·018 (7) Å.

The metal—C bond distances of carbonyls *trans* to phosphorus sites are in the range 1·953 (8)–1·999 (8) Å, whereas the other two metal bonds (*cis* to phosphorus) are 1·956 (8)–1·993 (8) Å, and they are all normal bond lengths. Generally, those metal–C bonds *trans* to phosphorus sites in metal carbonyl systems have shorter distances; however, in the present species there is no significant difference. All W—C—O bond angles range from 172·9 (8)–179·7 (6)°, deviating slightly from linearity.

The diastereotropic property of methyl groups on the isopropyl unit can be observed by ^1H NMR spectroscopy, which shows absorptions at 1·34 (*dd*, $J = 6{\cdot}8$ Hz, $J_{P—H} = 16{\cdot}6$ Hz, 12 H) and 0·68 p.p.m. (*dd*, $J = 7{\cdot}0$ Hz, $J_{P—H} = 15$ Hz, 12 H). In this crystalline structure, all the methyl groups point toward the $W(CO)_4$ moiety, as shown in Fig 1, in order to minimize steric interaction.

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

W1—P1	2·510 (2)	W2—P3	2·497 (2)
W1—P2	2·509 (2)	W2—P4	2·499 (2)
W1—C1	1·960 (8)	W2—C5	1·993 (8)
W1—C2	1·963 (7)	W2—C6	1·953 (8)
W1—C3	1·956 (8)	W2—C7	1·999 (8)
W1—C4	1·994 (7)	W2—C8	1·960 (8)
P1—C11	1·864 (7)	P3—C41	1·885 (7)
P1—C14	1·865 (8)	P3—C44	1·861 (7)
P1—C31	1·806 (6)	P3—C61	1·828 (7)
P2—C21	1·852 (7)	P4—C51	1·851 (9)
P2—C24	1·871 (7)	P4—C54	1·851 (8)
P2—C36	1·859 (6)	P4—C66	1·847 (7)
C1—O1	1·17 (1)	C5—O5	1·160 (9)
C2—O2	1·164 (9)	C6—O6	1·18 (1)
C3—O3	1·178 (9)	C7—O7	1·140 (9)
C4—O4	1·147 (9)	C8—O8	1·18 (1)
P1—W1—P2	80·06 (6)	P3—W2—P4	79·82 (7)
P1—W1—C4	173·2 (2)	P3—W2—C7	174·5 (3)
P2—W1—C2	174·1 (2)	P4—W2—C6	174·6 (2)
C1—W1—C3	169·8 (3)	C5—W2—C8	169·3 (3)
C11—P1—C14	101·8 (3)	C41—P3—C44	102·6 (3)
C21—P2—C24	101·8 (3)	C51—P4—C54	99·8 (4)
W1—C1—O1	174·7 (6)	W2—C5—O5	174·9 (6)
W1—C2—O2	179·7 (6)	W2—C6—O6	179·3 (7)
W1—C3—O3	173·1)6)	W2—C7—O7	178·8 (8)
W1—C4—O4	177·0 (8)	W2—C8—O8	172·9 (8)

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References

Al-Jibori, S., Hall, M., Hutton, A. T. & Shaw, B. L. (1984). *J. Chem. Soc. Dalton Trans.* pp. 863–867.

Churchill, M. R., Rheingold, A. L. & Keiter, R. L. (1981). *Inorg. Chem.* **20**, 2730–2724.

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

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52117 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetrakis(1-ethoxy-4,4,4-trifluoro-1,3-butanedionato)uranium(IV)

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Abstract. $[U(C_6H_6F_3O_3)_4]$, $M_r = 970{\cdot}46$, monoclinic, $P2_1$, $a = 10{\cdot}034\ (1)$, $b = 15{\cdot}181\ (2)$, $c = 11{\cdot}842\ (1)$ Å, $\beta = 111{\cdot}25\ (2)°$, $V = 1681{\cdot}2\ (7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}917$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 4{\cdot}694$ mm^{-1}, $F(000) = 928$, $T = 295$ K, $R = 0{\cdot}027$ for 2371 unique reflections with $I > 3\sigma(I)$. The U atom is eight coordinated by the O atoms of four independent β-diketone ligands, forming a dodecahedral

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