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CRYSTAL AND MOLECULAR STRUCTURE OF THE CLATHRATE COMPOUND (ALLYLIDENETRIPHENYLPHOSPHORANE)-MOLYBDENUM TETRACARBONYL BENZENE

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SUMMARY

The crystal and molecular structures of the clathrate compound (allylidenetriphenylphosphorane)molybdenum tetracarbonyl benzene, have been determined from an X-ray diffraction study, performed with a Picker Facs-1, computer controlled, diffractometer. The phosphorus ylide is linked to the metal through a π-allylic system involving molybdenum and the three carbon atoms of the starting allylidene group. The coordination at the molybdenum atom corresponds to a distorted octahedron. The coordination at the phosphorus atom substantially corresponds to those found in phosphorus ylides. The crystals of $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ have $P2_1/c$ symmetry with four molecules in a unit cell defined by the parameters $a=15.049$ (7) Å, $b=10.798$ (5) Å, $c=18.47$ (1) Å, $\beta=111°$ (8′). The structure has been refined by several least-squares cycles. The final value of $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$ is 0.068 for 3058 independent observed reflections.

INTRODUCTION

Allylidenetriphenylphosphorane reacts with $M(CO)_6$ ($M = Mo$, W) in boiling tetrahydrofuran, to give $M(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]$[1]. The objective of this paper is the definition of the X-ray crystal structure of $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ (I).

EXPERIMENTAL

The crystals of (I) were firmly mounted in Lindemann glass capillary tubes filled with dry nitrogen and flame sealed. The crystals were first examined with a Weissenberg camera (Ni filtered Cu-K_α radiation). The reciprocal lattice symmetry and the absence of ($0k0$) reflections with k odd and of ($h0l$) reflections with l odd indicated that the crystals of (I) are monoclinic and that the space group is $P2_1/c$ (C_{2h}^5).

A crystal, essentially a parallelepiped, having dimensions approximately 0.30 × 0.20 × 0.60 mm, was mounted on a Picker FACS 1, four-circle, computer-controlled diffractometer, equipped with a scintillation counter and a pulse-height analyser. Zirconium-filtered molybdenum radiation was employed in all the measurements.

TABLE 1

FRACTIONAL COORDINATES OF THE $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ INDEPENDENT UNIT.

The e.s.d., in parenthesis, in this and in the following tables, occurs in the last significant digit.

Atom	*x/a*	*y/b*	*z/c*	*B* (Å^2)
Mo	0.19422 (6)	−0.01231 (7)	0.19281 (4)	
P	0.2340 (2)	0.2236 (2)	0.3431 (1)	
O_1	0.0452 (5)	−0.1289 (6)	0.2563 (4)	
O_2	0.1204 (7)	−0.2256 (8)	0.0754 (5)	
O_3	0.3408 (6)	0.0238 (9)	0.1085 (5)	
O_4	0.3362 (7)	−0.1818 (8)	0.3155 (5)	
C_1	0.0974 (7)	−0.0782 (7)	0.2352 (5)	
C_2	0.1494 (7)	−0.1476 (9)	0.1200 (5)	
C_3	0.2898 (7)	0.0183 (10)	0.1402 (5)	
C_4	0.2820 (8)	−0.1205 (9)	0.2684 (6)	
C_{23}	0.2177 (6)	0.1909 (8)	0.2464 (5)	
C_{24}	0.1246 (7)	0.1789 (8)	0.1920 (5)	
C_{25}	0.1076 (8)	0.1647 (9)	0.1122 (5)	
C_5	0.3588 (7)	0.2223 (9)	0.3982 (5)	
C_6	0.4183 (9)	0.1411 (11)	0.3804 (7)	
C_7	0.5167 (10)	0.1386 (15)	0.4234 (9)	
C_8	0.5513 (9)	0.2199 (15)	0.4843 (8)	
C_9	0.4939 (10)	0.2984 (15)	0.5045 (8)	
C_{10}	0.3959 (9)	0.3031 (13)	0.4597 (7)	
C_{11}	0.1733 (6)	0.1154 (8)	0.3840 (5)	
C_{12}	0.2219 (7)	0.0126 (9)	0.4232 (5)	
C_{13}	0.1722 (8)	−0.0721 (8)	0.4516 (6)	
C_{14}	0.0781 (9)	−0.0575 (10)	0.4397 (6)	
C_{15}	0.0311 (9)	0.0459 (11)	0.4013 (6)	
C_{16}	0.0784 (8)	0.1333 (10)	0.3723 (6)	
C_{17}	0.1914 (6)	0.3746 (7)	0.3552 (5)	
C_{18}	0.1694 (8)	0.4058 (10)	0.4190 (6)	
C_{19}	0.1409 (9)	0.5267 (11)	0.4266 (7)	
C_{20}	0.1341 (8)	0.6122 (10)	0.3712 (7)	
C_{21}	0.1545 (9)	0.5831 (10)	0.3062 (8)	
C_{22}	0.1830 (7)	0.4612 (8)	0.2983 (6)	
C_{26}	0.2972 (12)	0.4822 (17)	0.1455 (10)	
C_{27}	0.3485 (14)	0.5429 (17)	0.2165 (12)	
C_{28}	0.4283 (14)	0.4908 (21)	0.2670 (11)	
C_{29}	0.4619 (12)	0.3832 (21)	0.2451 (11)	
C_{30}	0.4158 (14)	0.3288 (15)	0.1750 (11)	
C_{31}	0.3345 (12)	0.3791 (17)	0.1262 (10)	
H_{23}	0.267	0.236	0.234	5.0
H_{24}	0.075	0.175	0.208	5.0
H_{25}	0.167	0.200	0.100	5.0
H_{251}	0.042	0.150	0.067	5.0
H_6	0.388	0.079	0.332	5.0
H_7	0.563	0.075	0.408	5.0
H_8	0.626	0.223	0.516	5.0
H_9	0.525	0.355	0.557	5.0
H_{10}	0.351	0.370	0.474	5.0
H_{12}	0.296	−0.002	0.432	5.0

(*continued*)

TABLE 1 (contd.)

Atom	x/a	y/b	z/c	B (Å^2)
H_{13}	0.207	−0.152	0.483	5.0
H_{14}	0.041	−0.126	0.461	5.0
H_{15}	−0.042	0.057	0.393	5.0
H_{16}	0.041	0.213	0.341	5.0
H_{18}	0.177	0.337	0.464	5.0
H_{19}	0.119	0.550	0.475	5.0
H_{20}	0.117	0.707	0.380	5.0
H_{21}	0.146	0.651	0.262	5.0
H_{22}	0.199	0.437	0.248	5.0
H_{26}	0.230	0.518	0.107	5.0
H_{27}	0.324	0.630	0.232	5.0
H_{28}	0.467	0.536	0.321	5.0
H_{29}	0.526	0.340	0.285	5.0
H_{30}	0.444	0.244	0.157	5.0
H_{31}	0.298	0.334	0.071	5.0

The orientation matrix and the unit cell dimensions, with their e.s.d., were obtained from a least squares fit of χ, ϕ, ω and 2θ values from 12 independent reflections. The room temperature unit cell dimensions are: $a = 15.049$ (7) Å, $b = 10.798$ (5) Å, $c = 18.47$ (1) Å, $\beta = 111°$ (8′), $V = 2802$ Å^3, $Z = 4$, $d_{\text{calcd.}} = 1.39$ g/cm^3, $d_{\text{obs.}} = 1.40$ g/cm^3.

The intensity data were collected using zirconium filtered Mo-K_α radiation in the range $3° \leqslant 2\theta \leqslant 50°$. The moving-crystal moving-counter technique was used, with a 2θ scan rate of 1° min^{-1} and a scan range of $(2.00° + \Delta)$, where Δ is introduced to take into account the 2θ separation of the K_{α_1} K_{α_2} peaks at increasing values of 2θ. Background counts of 10 sec each, were measured at the extremes of each 2θ scan. Three monitoring, symmetry-equivalent reflections, have been measured after every 50 reflections. The intensities of these monitoring reflections decreased slightly with time (about 20% by the end of the collection), thus indicating a progressive alteration of the crystal. The decomposition of the crystal has been accounted for on the basis of the monitoring reflections considering the decomposition to be isotropic. A total of 4784 independent reflections were measured, 3058 of which were accepted as significant under the criterion $I \geqslant 3\sigma$. An arbitrary intensity equal to 0.5 of the observable limit, was assigned to the 1726 non-significant reflections. All the intensities were corrected for Lorentz and polarization effects. No corrections have been introduced for absorption ($\mu = 4.8$ cm^{-1}).

DETERMINATION AND REFINEMENT OF THE STRUCTURE

The structure was solved by the heavy atom method. The coordinates of the Mo and P atoms were derived from a three-dimensional Patterson map. The y coordinates of the molybdenum and phosphorus atoms (≈ 0 and ≈ 0.250 respectively) are such as to introduce a false higher symmetry in the Fourier map phased on these atoms only. The Fourier map clarification was achieved by a stepwise introduction of the carbon and oxygen atoms into the structure factors calculations, their location being derived both on Fourier and on stereochemical grounds. The final Fourier map

TABLE 2

ANISOTROPIC THERMAL PARAMETERS (Å^2) FOR THE NON-HYDROGEN ATOMS OF THE $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ INDEPENDENT UNIT

The anisotropic temperature factor is in the form: $T_j = \exp[-\frac{1}{4}[B_{11}\cdot a^{*2}\cdot h^2 + B_{22}\cdot b^{*2}\cdot k^2 + B_{33}\cdot c^{*2}\cdot l^2 + 2\,B_{12}\cdot a^*\cdot b^*\cdot h\cdot k + 2\,B_{13}\cdot a^*\cdot c^*\cdot h\cdot l + 2\,B_{23}\cdot b^*\cdot c^*\cdot k\cdot l)]$.

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo	4.29 (4)	2.90 (3)	3.04 (3)	−0.04 (3)	1.50 (3)	−0.45 (3)
P	3.57 (11)	2.23 (8)	2.26 (8)	0.17 (7)	1.06 (8)	−0.14 (7)
O_1	5.46 (38)	3.67 (31)	4.85 (35)	−0.62 (28)	2.14 (31)	0.06 (27)
O_2	10.50 (65)	5.61 (44)	5.22 (42)	−2.05 (43)	3.75 (44)	−1.84 (35)
O_3	5.70 (44)	8.28 (55)	6.23 (45)	0.50 (40)	3.45 (38)	0.95 (41)
O_4	6.64 (47)	5.49 (43)	6.22 (46)	1.19 (37)	1.46 (38)	0.97 (36)
C_1	3.98 (43)	2.19 (32)	2.87 (36)	−0.62 (30)	1.04 (32)	−0.62 (28)
C_2	5.30 (52)	3.85 (42)	2.04 (33)	−0.78 (37)	1.51 (34)	−1.05 (31)
C_3	3.73 (43)	4.61 (47)	3.44 (40)	−0.27 (37)	1.43 (34)	0.05 (36)
C_4	4.56 (51)	3.91 (44)	4.19 (46)	0.15 (38)	1.93 (40)	0.10 (37)
C_{23}	3.70 (42)	2.99 (36)	2.73 (36)	0.68 (31)	1.34 (32)	−0.49 (28)
C_{24}	4.94 (49)	2.12 (33)	3.44 (40)	0.70 (32)	1.60 (37)	0.10 (29)
C_{25}	4.79 (53)	3.98 (44)	3.11 (41)	0.81 (38)	0.71 (38)	0.26 (34)
C_5	3.96 (44)	3.48 (39)	2.66 (36)	0.07 (33)	1.39 (33)	−0.06 (30)
C_6	4.74 (56)	5.01 (55)	5.71 (60)	0.68 (45)	1.24 (47)	−0.57 (46)
C_7	4.88 (67)	8.48 (90)	7.99 (84)	1.26 (62)	1.33 (61)	−0.95 (73)
C_8	4.93 (65)	8.24 (85)	6.37 (72)	0.23 (59)	0.79 (56)	−0.49 (64)
C_9	5.36 (63)	7.96 (84)	6.48 (69)	−0.35 (59)	1.36 (53)	−2.54 (63)
C_{10}	4.93 (60)	7.15 (71)	4.54 (54)	0.09 (51)	1.10 (47)	−1.42 (50)
C_{11}	3.54 (41)	2.85 (34)	2.77 (35)	−0.22 (30)	1.48 (32)	−0.31 (28)
C_{12}	4.60 (48)	3.28 (40)	3.51 (40)	0.73 (36)	1.36 (36)	−0.12 (33)
C_{13}	5.89 (57)	2.63 (37)	3.79 (43)	−0.62 (37)	1.95 (41)	0.10 (33)
C_{14}	6.79 (62)	4.28 (47)	3.00 (40)	−0.32 (44)	2.56 (42)	0.38 (35)
C_{15}	5.50 (59)	5.38 (55)	4.39 (51)	−0.11 (46)	2.17 (45)	0.08 (43)
C_{16}	5.67 (57)	4.30 (47)	4.07 (46)	−0.04 (41)	2.73 (43)	0.70 (38)
C_{17}	3.46 (42)	2.17 (33)	3.67 (40)	0.21 (29)	1.16 (34)	−0.61 (29)
C_{18}	5.19 (54)	4.63 (49)	3.45 (43)	1.19 (42)	1.63 (39)	−0.57 (37)
C_{19}	6.40 (65)	4.84 (54)	4.72 (53)	1.61 (48)	1.24 (47)	−1.92 (44)
C_{20}	5.24 (59)	3.13 (43)	6.51 (63)	0.57 (40)	1.11 (49)	−1.21 (43)
C_{21}	5.23 (59)	3.81 (49)	7.32 (70)	−0.10 (44)	2.27 (53)	0.27 (48)
C_{22}	4.77 (51)	2.13 (34)	4.99 (49)	0.00 (32)	1.78 (41)	0.39 (32)
C_{26}	7.55 (86)	10.81 (116)	9.27 (101)	−0.92 (79)	4.66 (80)	3.74 (88)
C_{27}	11.81 (127)	8.43 (98)	13.33 (141)	−1.54 (93)	9.58 (121)	−1.27 (98)
C_{28}	9.73 (117)	13.48 (157)	7.86 (98)	−4.87 (108)	2.62 (88)	−2.08 (99)
C_{29}	7.02 (92)	12.90 (145)	9.65 (110)	−0.39 (94)	2.67 (85)	2.39 (107)
C_{30}	12.11 (126)	6.65 (82)	10.56 (114)	0.02 (84)	6.91 (103)	0.07 (80)
C_{31}	9.24 (103)	9.67 (108)	7.94 (91)	−2.07 (85)	4.56 (82)	0.23 (82)

phased on all the non-hydrogen atoms revealed no unusual features. The positional and the isotropic thermal parameters of all the non-hydrogen atoms were refined by use of some cycles of the block-diagonal least-squares, by minimizing the quantity $[\Sigma\, w(|F_o|-|F_c|)^2]$. A general computation program prepared by Immirzi[2] was employed. The atomic scattering factors were calculated by use of the expression suggested by Vand, Eiland and Pepinski[3], using for the parameters the values suggested by Moore[4]. The weighting scheme suggested by Cruikshank, Pilling, Bujosa, Lovel and Truter[5] was adopted. The conventional $R=\Sigma|F_o|-|F_c||/\Sigma|F_o|$ was at this point 0.12 for the 3058 non-zero reflections.

Four block-diagonal cycles of refinement were run by assuming anisotropic thermal parameters for all the non-hydrogen atoms of the independent unit. The hydrogen atoms of benzene and of the C_6H_5 groups have been introduced (not refined) into the structures factors calculations, their location being derived on stereochemical grounds. The location of the hydrogen atoms of the allylidene group H_{23}, H_{24}, H_{25} and H_{251} has been derived by a difference map. The refinement gave convergence to a final discrepancy factor $R=0.068$ for the 3058 significant reflections. The final shifts of the atomic parameters were negligibly small, all of them being well below the corresponding e.s.d.

The final fractional coordinates and the corresponding e.s.d. for the atoms of the independent unit are reported in Table 1. In Table 2 are reported the anisotropic thermal parameters of all the non-hydrogen atoms. The list of the calculated and observed structure amplitudes can be obtained by application to the authors.

RESULTS AND DISCUSSION

The molecules of (I) are represented in Figs. 1 and 2 as obtained from the coordinates reported in Table 1. The geometrical parameters of the molecule with their e.s.d. are reported in Table 3. The coordination at the molybdenum atom corresponds to a distorted octahedron. The C_2, C_4, C_{23}, C_{25} and Mo atoms are nearly coplanar (r.m.s. distance 0.06 Å) and the C_{24} atom is out of this plane (0.31 Å). The three carbon atoms of the starting allylidene group are π-coordinated to the molybdenum atom.

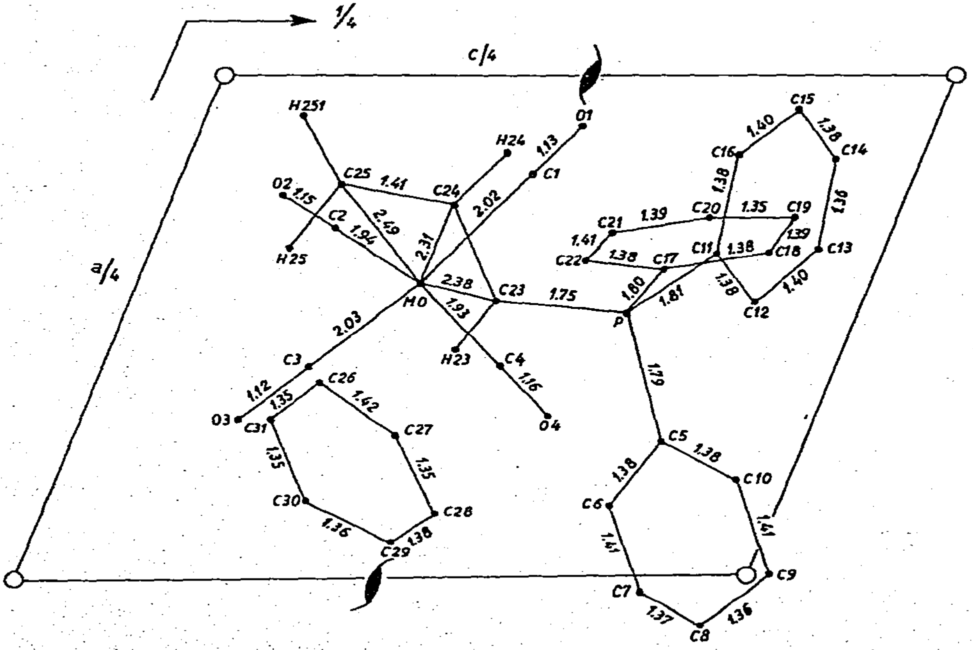


Fig. 1. Projection along the *b* axis unit cell of the $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ molecule, indicating the atom labelling scheme (interatomic distances in Å).

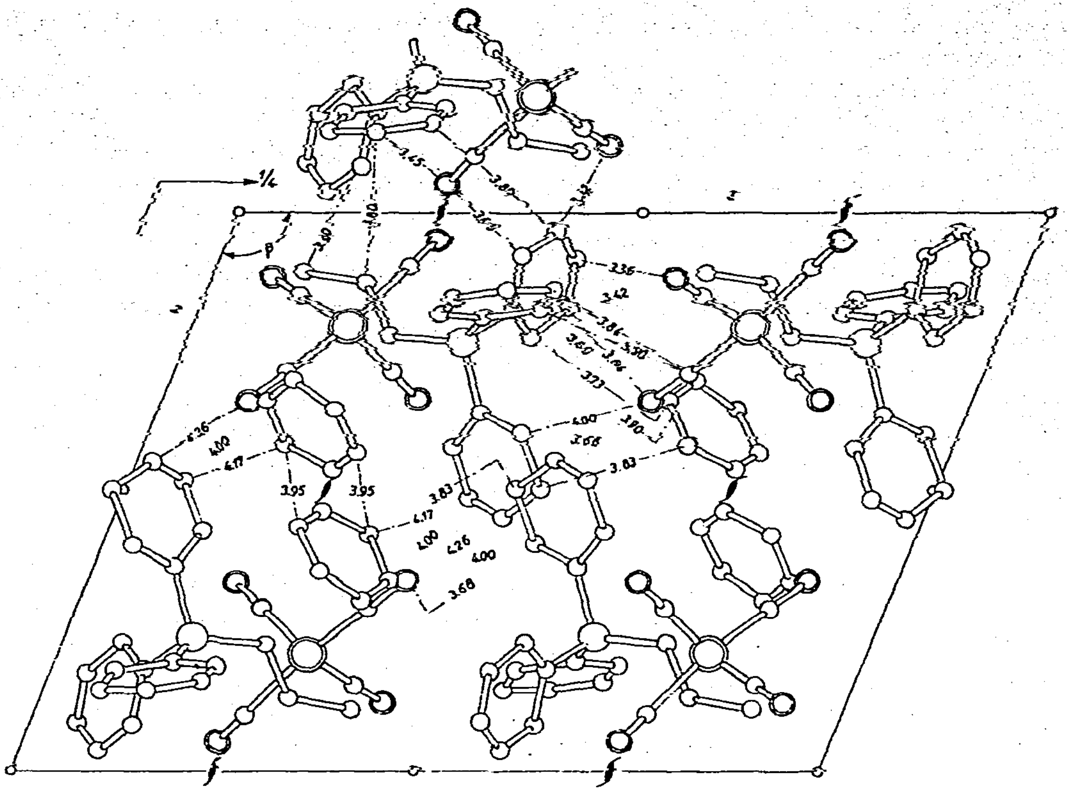
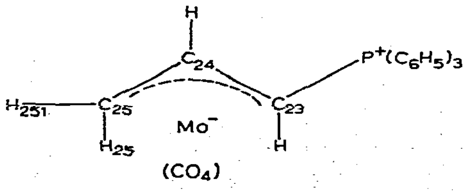


Fig. 2. The packing arrangement of the $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ molecules as viewed down the *b* axis. Some of the most significant intermolecular contact distances are indicated (interatomic distances in Å). Doubly ringed atoms: large circles Mo, small circles O; singly ringed atoms: large circles P, small circles C.

The $Mo{-}C_{23}$, $Mo{-}C_{24}$ and $Mo{-}C_{25}$ bond lengths are 2.38 (1) Å, 2.31 (1) Å and 2.49 (1) Å respectively (average 2.39 (1) Å).

Compound (I) is diamagnetic. The bonding situation can be described as follows: one of the two electrons of the carbon–phosphorus double bond of the starting ylide is allowed to take part in the formation of the π allylic system while the second is digested by the electronic shell of the metal, leaving a formal positive charge on the phosphorus atom:



This π-allylic formulation is in agreement with the coplanarity of the C_{25}, C_{24}, C_{23} and P atoms (r.m.s. distance 0.07 Å) and with the bond angles $C_{25}{-}C_{24}{-}C_{23}$, $C_{24}{-}C_{23}{-}$

TABLE 3

GEOMETRICAL PARAMETERS OF THE $Mo(CO)_4[(C_6H_5)_3P{=}CH{-}CH{=}CH_2]\cdot C_6H_6$ MOLECULE IN THE CRYSTALS

Bond lengths (Å)	
$Mo{-}C_1$	2.02 (1)
$Mo{-}C_2$	1.94 (1)
$Mo{-}C_3$	2.03 (1)
$Mo{-}C_4$	1.93 (2)
$Mo{-}C_{23}$	2.38 (1)
$Mo{-}C_{24}$	2.31 (1)
$Mo{-}C_{25}$	2.49 (1)
$P{-}C_{23}$	1.75 (1)
$P{-}C_5$	1.79 (1)
$P{-}C_{11}$	1.81 (1)
$P{-}C_{17}$	1.80 (1)
$C_1{-}O_1$	1.13 (1)
$C_2{-}O_2$	1.15 (1)
$C_3{-}O_3$	1.12 (1)
$C_4{-}O_4$	1.16 (2)
$C_{23}{-}C_{24}$	1.41 (2)
$C_{24}{-}C_{25}$	1.41 (2)
$C_{23}{-}H_{23}$	0.98
$C_{24}{-}H_{24}$	0.90
$C_{25}{-}H_{25}$	1.06
$C_{25}{-}H_{251}$	1.06

Bond angles, (°)		
$C_1{-}Mo{-}C_2$	83.29	(20)
$C_1{-}Mo{-}C_3$	168.01	(133)
$C_1{-}Mo{-}C_4$	84.72	(24)
$C_1{-}Mo{-}C_{23}$	100.60	(13)
$C_1{-}Mo{-}C_{24}$	85.94	(14)
$C_1{-}Mo{-}C_{25}$	101.96	(19)
$C_2{-}Mo{-}C_3$	85.45	(17)
$C_2{-}Mo{-}C_{23}$	161.24	(49)
$C_2{-}Mo{-}C_{25}$	100.73	(17)
$C_2{-}Mo{-}C_{24}$	128.63	(21)
$C_2{-}Mo{-}C_4$	91.39	(26)
$C_3{-}Mo{-}C_{23}$	91.38	(14)
$C_3{-}Mo{-}C_{24}$	104.38	(17)
$C_3{-}Mo{-}C_{25}$	84.15	(18)
$C_3{-}Mo{-}C_4$	91.47	(27)
$C_4{-}Mo{-}C_{25}$	166.71	(121)
$C_4{-}Mo{-}C_{23}$	107.19	(17)
$C_4{-}Mo{-}C_{24}$	137.30	(30)
$C_{23}{-}Mo{-}C_{25}$	60.52	(23)
$C_{24}{-}Mo{-}C_{25}$	33.93	(94)
$C_{23}{-}Mo{-}C_{24}$	34.84	(69)
$Mo{-}C_{23}{-}C_{24}$	69.90	(42)
$Mo{-}C_{24}{-}C_{25}$	79.79	(53)
$Mo{-}C_{25}{-}C_{24}$	66.28	(69)
$Mo{-}C_{24}{-}C_{23}$	75.26	(39)
$Mo{-}C_{23}{-}P$	123.82	(21)
$C_{23}{-}C_{24}{-}C_{25}$	121.25	(55)
$P{-}C_{23}{-}C_{24}$	119.25	(55)
$C_{23}{-}P{-}C_5$	108.41	(29)
$C_{23}{-}P{-}C_{11}$	112.89	(26)
$C_{23}{-}P{-}C_{17}$	112.64	(22)
$C_5{-}P{-}C_{11}$	109.97	(26)
$C_5{-}P{-}C_{17}$	106.59	(24)
$C_{11}{-}P{-}C_{17}$	106.15	(21)
$P{-}C_{23}{-}H_{23}$	107	
$C_{24}{-}C_{23}{-}H_{23}$	122	
$C_{23}{-}C_{24}{-}H_{24}$	120	
$C_{25}{-}C_{24}{-}H_{24}$	118	
$C_{24}{-}C_{25}{-}H_{25}$	109	
$C_{24}{-}C_{25}{-}H_{251}$	128	
$H_{25}{-}C_{25}{-}H_{251}$	120	

Torsion angles, (°)				
C_{25}	C_{24}	C_{23}	P	173.66
C_{24}	C_{23}	P	C_{17}	−66.32
C_{24}	C_{23}	P	C_{11}	53.89
C_{24}	C_{23}	P	C_5	175.98
C_{23}	P	C_{17}	C_{22}	−22.09
C_{23}	P	C_{17}	C_{18}	159.23
C_{23}	P	C_{11}	C_{16}	−84.02
C_{23}	P	C_{11}	C_{12}	87.28
C_{23}	P	C_5	C_6	−35.04
C_{23}	P	C_5	C_{10}	145.34

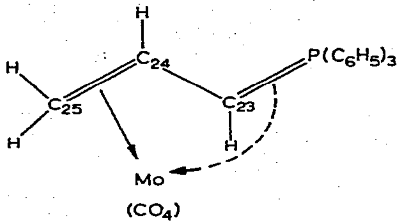
Most relevant intramolecular distances between non-bonded atoms (Å)	
Mo...P	3.66
$P...C_1$	3.99
$O_1...C_{11}$	3.60
$O_1...C_{12}$	3.61
$O_1...C_{13}$	3.48
$O_1...C_{14}$	3.34
$O_1...C_{15}$	3.35
$O_1...C_{16}$	3.48
$O_3...C_{30}$	3.56
$O_3...C_{31}$	3.86
$O_4...C_{12}$	3.71
$O_4...C_6$	3.75
$C_1...C_{11}$	3.31
$C_1...C_{12}$	3.45
$C_1...C_{13}$	3.75
$C_1...C_{14}$	3.90
$C_1...C_{15}$	3.79
$C_1...C_{16}$	3.50
$C_3...C_{30}$	3.79
$C_3...C_{31}$	3.98
$C_4...C_{12}$	3.59
$C_{23}...C_6$	3.19
$C_{23}...C_{12}$	3.77
$C_{23}...C_{16}$	3.70
$C_{23}...C_{22}$	3.17
$C_{23}...C_{30}$	3.96
$C_{23}...C_{31}$	3.87
$C_{24}...C_{11}$	3.42
$C_{24}...C_{16}$	3.67
$C_{24}...C_{17}$	3.52
$C_{24}...C_{22}$	3.56
$C_5...C_{18}$	3.60
$C_5...C_{22}$	3.68
$C_6...C_{12}$	3.60
$C_6...C_{29}$	3.83
$C_{10}...C_{22}$	3.90
$C_{11}...C_{18}$	3.20
$C_{21}...C_{27}$	3.86
$C_{22}...C_{26}$	3.80
$C_{22}...C_{27}$	3.46
$C_{22}...C_{28}$	3.95

The average C–C bond length and C–C–C bond angle for the benzene ring and the phenyl groups are 1.38 Å and 120° respectively.

P that are 121.3(6)° and 119.3(6)° respectively. Furthermore the $C_{25}{-}C_{24}$ and $C_{24}{-}C_{23}$ bonds have the same length [1.41 (2) Å]. The H_{251} and H_{24} hydrogen atoms are slightly out of the plane defined by C_{25}, C_{24}, C_{23} and P while H_{25} and H_{23} are $\approx$0.50 Å out of this plane away from the metal. Very similar arrangements of the hydrogen atoms of diolefin ligands about the coordinating metal have been found in some other complexes[6,7].

The arrangement of the phenyl groups with respect to C_{24} and C_{23} carbon atoms is nearly staggered. The C_{24}–C_{23}–P–C_{17}, C_{24}–C_{23}–P–C_{11} and C_{24}–C_{23}–P–C_5 torsion angles are $-66°$, $54°$ and $176°$ respectively.

The observed P–C_{23} bond length (1.75 (1) Å) is shorter than the P–C(Ph) bond lengths (1.79 (1) Å, 1.81 (1) Å and 1.80 (1) Å) which are in agreement with the quoted P–C single bonds distances[8]. A degree of double bond character in the P–C_{23} bond may arise from a contribution to the bonding system of the structure:



The length of a P=C double bond calculated by the sum of the atomic radii[8], is 1.665 Å. This observation and the coplanarity of C_{25}, C_{24}, C_{23}, and P atoms evidence that, as postulated for the phosphorus ylides[8], a probable electron delocalization takes place also in the molecule of (I).

With regard to the four-fold coordination at the P atom, there is a reduction of the local symmetry from tetrahedral to trigonal, as shown by the smaller values of the C(Ph)–P–C(Ph) bond angles (average 107°) in respect of the C_{23}–P–C(Ph) angles (average 111°). The smallest C_{23}–P–C(Ph)–C(Ph) torsion angles are $-22°$, $-35°$ and $-84°$. Both these observations are in line with the data reported for many phosphorous ylides[8].

The molecular packing of (I) consists of an arrangement of up and down molecules around each inversion centre of the unit cell. No C...C intermolecular distance is shorter than 3.7 Å, while the shortest C...O intermolecular distances are ≈3.4 Å. The carbon atoms of the benzene molecules are at Van der Waals distances from their nearest neighbours.

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