Conjugate Addition of Acetylides to Complexes of Type $[M(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$ (M = Cr, Mo, or W): Crystal Structure of $[M(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$† formed by the Base-catalysed Isomerization of $[M(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CPh\}]$

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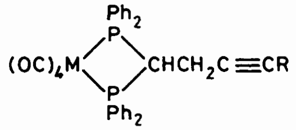
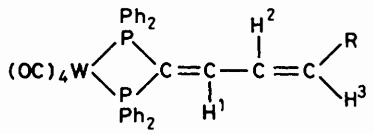
Although treatment of $[W(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$ with $Li(C{\equiv}CPh)$ resulted in no conjugate addition, in the presence of $Me_2NCH_2CH_2NMe_2$ smooth addition occurred to give $[W(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CPh\}]$ (**1c**). Similar additions were effected with the corresponding complexes of Cr and Mo, and other acetylides, $Li(C{\equiv}CR)$ where R = C_6H_4Me-*p*, Me, Pr^n, or Bu^n, underwent addition. Treatment of $[W(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CPh\}]$ with $KOBu^t$ in Pr^iOH gave the dienephosphine complex $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$ (**2a**); the *p*-tolylactylide complex $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHC_6H_4Me\text{-}p\}]$ was also prepared. Crystals of compound (**2a**) are monoclinic, space group $P2_1/c$ with a = 988.4(3), b = 1 069.7(2), c = 3 233.7(4) pm, β = 94.73(2)°, and Z = 2; final R = 0.0334 and R' = 0.0316 for 3 929 observed reflections. Hydrogen-1 and $^{31}P\text{-}\{^1H\}$ n.m.r. and i.r. data are given.

We have described the synthesis of $[M(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$ either by treating the carbanions $[M(CO)_4\{(Ph_2P)_2CH\}]^-$ with CH_3OCH_2Cl (for M = Cr or W)[1,2] or, more conveniently, by treating $[M(CO)_6]$ with $(Ph_2P)_2C{=}CH_2$ [vinylidenebis(diphenylphosphine), vdpp].[2] The synthesis of $[M(CO)_4(\text{vdpp-}PP')]$ (M = Mo or W) has also been described by Colquhoun and McFarlane.[3] We have also shown that, although the free diphosphine vdpp is hardly activated towards Michael additions across the $CH_2{=}C$ bond, when complexed, even to a relatively poorly activating group such as $M(CO)_4$, then activation towards Michael addition is greatly increased, even an amine such as pyrrolidine being added on.[1,2] Carbanions, *e.g.* the lithium reagents LiMe or $LiBu^n$ or $NaCH(COOEt)_2$ readily add to $[M(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$.[1,2] In this paper we report the addition of lithium acetylides to $[M(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$ and the subsequent prototropic (base-catalysed) isomerizations of some of them.



	M	R
(**1a**)	Cr	Ph
(**1b**)	Mo	Ph
(**1c**)	W	Ph
(**1d**)	Mo	C_6H_4Me-p
(**1e**)	Mo	Bu^n
(**1f**)	Mo	Me
(**1g**)	W	Pr^n
(**1h**)	W	Bu^n
(**1i**)	Mo	Pr^n
(**1j**)	Cr	Me
(**1k**)	W	C_6H_4Me-p



	R
(**2a**)	Ph
(**2b**)	C_6H_4Me-p

Results and Discussion

Treatment of $[M(CO)_4(\text{vdpp-}PP')]$ with $Li(C{\equiv}CPh)$ in tetrahydrofuran resulted in no conjugate addition and recovery of the vdpp starting complex, even after prolonged reflux. We therefore increased the carbanion character of the acetylide by adding $Me_2NCH_2CH_2NMe_2$ (tmen) and found that in its presence $Li(C{\equiv}CPh)$ added smoothly to $[M(CO)_4(\text{vdpp-}PP')]$ over an 18-h period at 20 °C. After this period the reaction solution was extremely dark but addition of methanol then gave $[M(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CPh\}]$ (M = Cr, Mo, or W) as yellow or orange crystals in excellent yields. The complexes were characterized by elemental analyses, i.r. spectroscopy (Table 1), and particularly by $^{31}P\text{-}\{^1H\}$, $^1H\text{-}\{^{31}P\}$, and ^1H n.m.r. spectroscopy (Table 2). Other complexes of type $[M(CO)_4\{(Ph_2P)CHCH_2C{\equiv}CR\}]$ were formed similarly, *viz.* with M = Mo or W, R = *p*-tolyl, Pr^n, or Bu^n; M = Cr or Mo, R = Me. Details of the preparation are in the Experimental section and characterizing analytical and spectroscopic data in Tables 1 and 2. All the complexes showed far-i.r. absorption in the carbonyl region (Table 2), characteristic of a *cis*-$M(CO)_4$ moiety. The $^{31}P\text{-}\{H\}$ n.m.r. spectra consisted of a single line (complexes of Cr or Mo) or a single line with satellites (W) due to ^{183}W (14.3% abundance, $I = \frac{1}{2}$). The $^1H\text{-}\{^{31}P\}$ n.m.r. spectrum of $[Mo(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CMe\}]$ showed a triplet, a

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† [1,1-Bis(diphenylphosphino)-4-phenylbuta-1,3-diene-*PP'*]tetracarbonyltungsten.

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1988, Issue 1, pp. xvii—xx.

Table 1. Analytical and i.r. (cm^{-1}) data

Complex	Analysis (%)[a]		Carbonyl ν(C≡O)[b]
	C	H	
(1a)	68.65 (68.9)	4.2 (4.25)	2 000, 1 920, 1 900, 1 875
(1b)	64.95 (64.6)	3.85 (4.00)	2 020, 1 920, 1 910, 1 870
(1c)	57.15 (57.4)	3.45 (3.55)	2 018, 1 920, 1 895, 1 855
(1d)	61.6 (61.5)	4.0 (4.05)	2 020, 1 935, 1 920, 1 870
(1e)	63.25 (63.0)	4.65 (4.7)	2 020, 1 935, 1 920, 1 870
(1f)	65.15 (65.0)	4.2 (4.25)	2 020, 1 920, 1 910, 1 885
(1g)	54.15 (58.8)	3.65 (3.85)	
(1h)	55.75 (55.8)	4.05 (4.15)	
(1i)	62.05 (62.5)	4.55 (4.5)	2 020, 1 930, 1 875, 1 840
(1j)	66.05 (66.0)	4.10 (4.15)	2 005, 1 920, 1 910, 1 840
(1k)	57.75 (57.8)	3.85 (3.9)	2 005, 1 930, 1 910, 1 875
(2a)	57.15 (57.4)	3.6 (3.55)	2 010, 1 910, 1 880, 1 855
(2b)·0.33CH_2Cl_2	56.15 (56.25)	3.6 (3.65)	2 005, 1 915, 1 860, 1 825

[a] Calculated values in parentheses. [b] As Nujol mulls (solid).

Table 2. $^{31}P-\{^1H\}$ and ^1H n.m.r. data for complexes of type (**1**)[a]

Complex	^{31}P[b]		^1H[c]					
	δ(P)	^1J(W–P)	δ(CH)	^2J(PCH)	δ(CH_2)	^3J(PCH_2)	^3J(CHCH_2)	δ(CH_3)
(1a)	49.2		4.96	11	2.51	10	7.3	—
(1b)	25.4		4.93	11	2.45	11	7.3	—
(1c)	0.8	205	5.11	11	2.42	11	7.3	—
(1d)	25.3		5.10	11	2.60	10	7.3	2.51
(1e)	24.3		4.78	10	2.16	11	7.1	0.85
(1f)[d]	25.6		4.77	10	2.17	11	7.3	1.61
(1g)	1.2	205	4.52	8	1.95	12	8.1	0.71
(1h)	2.3	207	4.96	n.r.	2.16	11	7.3	0.85
(1i)	25.7		4.79	n.r.	2.21	10	7.1	0.88
(1j)	49.1		4.57	n.r.	1.93	9	7.6	1.24
(1k)	0.8	205	5.09	11	2.36	11	7.3	2.33

[a] Recorded in $CDCl_3$ solution at 21 °C. [b] Positive shifts, δ values ± 0.01 p.p.m., to high frequency of external 85% H_3PO_4. Coupling constants *J* ± 3 Hz. [c] Positive shifts, δ values ± 0.01 p.p.m., to high frequency of $SiMe_4$. Coupling constants *J* ± 0.3 Hz. [d] $^5J(CH_2CH_3)$ 2.5 Hz.

Table 3. $^{31}P-\{^1H\}$ and ^1H n.m.r. data for complexes of type (**2**)

Complex	^{31}P[a]		^1H[b]						
	δ(P)	^1J(W–P)	δ(CH^1)	δ(CH^2)	δ(CH^3)	$^3J(PH^1)$	$^3J(H^1H^2)$	$^4J(H^1H^3)$	$^3J(H^2H^3)$
(2a)	3.8	210	6.46	6.32	6.62	23	11.4	*ca.* 1	15.3
(2b)	3.4[c]	210	6.46	6.34	6.61	23	11.4	<1	15

[a] In $CDCl_3$, measured at 40.25 MHz; chemical shifts, δ, to high frequency of 85% H_3PO_4, *J* values ± 2 Hz. [b] In $CDCl_3$ at 360 MHz; chemical shifts, δ, to high frequency of $SiMe_4$, *J* values ± 0.1 Hz. [c] δ(CH_3) 2.28 p.p.m.

doublet of quartets, and a triplet, of relative intensities corresponding to 1, 2, and 3 H, respectively. The ^1H n.m.r. spectrum showed coupling to phosphorus by both the CH and the CH_2 protons but not the CH_3 protons. The mass spectra of compounds (**1e**), (**1h**), and (**1k**) were recorded and showed the expected intensity patterns for their molecular ions with the most intense peaks at *m*/*e* values of 681, 714, and 809, respectively.

It is known that the hydrogen on the carbon atom α to the P nuclei in Group 6 metal carbonyl complexes of type $[M(CO)_4\{(Ph_2P)_2CHR\}]$ (R = alkyl or H) is sufficiently acidic to be removed by a strong base.[4–6] We therefore hoped that we could effect a base-catalysed prototropic rearrangement of an acetylene to a conjugated diene system, *viz.* $[M(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CR\}] \longrightarrow [M(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHR\}]$. When a solution of $[W(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CPh\}]$ with KOBu^t in Pr^{i}OH (*i.e.* effectively KOPr^i) was heated for 1.5 h an intensely green solution formed which, on acidification, gave a deep red crystalline solid in excellent (>90%) yield. This was established to be the hoped for di-enediphosphine complex $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$, on the basis of elemental analysis (see Experimental section); the mass spectrum, which showed the most intense peak of the parent molecular ion (M^+) to be at *m*/*e* = 794, was as expected. The i.r. spectrum, as a Nujol mull, showed ν(C≡O) bands at 2 010, 1 910, 1 880, and 1 855 cm^{-1}, all very strong. The ^1H and $^{31}P-\{^1H\}$ n.m.r. data are shown in Table 3. We similarly made and characterized the *p*-tolyl analogue (**2b**); preparative details and some characterizing data are in the Experimental section and n.m.r. data in Table 3.

X-*Ray Crystal Structure of* $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$ (**2a**).—We considered this compound to be of sufficient interest to determine its crystal structure by *X*-ray diffraction.

The molecular structure is shown in the Figure and selected bond lengths and angles are given in Table 4. The carbon–carbon bond lengths C(131)–C(13), C(131)–C(132), and C(132)–C(133) of 133.9(9), 143.5(9), and 131.6(9) pm and the associated bond angles confirm the presence of the *trans*-C=CHCH=CHPh grouping. The bond angle P(2)–C(13)–P(1) of 100.7(4)° is small for sp^2 carbon but is presumably forced to adopt this small value by the requirements of the four-membered chelate ring.

Although the complexes $[W(CO)_4\{(Ph_2P)_2CHCH_2C{\equiv}CR\}]$ with R = Ph or *p*-tolyl rearranged to the dienediphosphine complexes $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHR\}]$ in very high yield, similar treatments of the alkylacetylide complexes, R = Pr^n or Bu^n, gave intensely coloured solutions but these on acidification gave complex mixtures which we could not separate. Treatment of the Cr- or Mo-containing adducts with $KOPr^i$ caused extensive changes (^{31}P-$\{^1H\}$ n.m.r. evidence) and we were unable to isolate any product complexes from the reaction solutions.

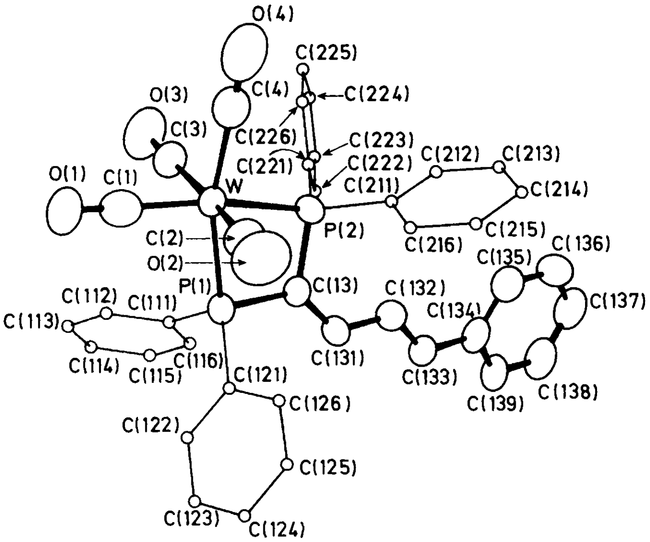

Figure. Molecular structure of $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$ (**2a**) showing the principal atom numbering

Experimental

The general procedures and apparatus used were the same as in other recent publications from this laboratory.[7]

Preparations.—$[Mo(CO)_4\{(Ph_2P)_2CHCH{\equiv}CPh\}]$ (**1b**). A solution of lithium phenylacetylide was made by the addition of butyl-lithium (1.55 mol dm^{-3} in hexane, 0.21 cm^3, 0.33 mmol) to a cooled (0 °C) solution of phenylacetylene (0.035 g, 0.35 mmol) in dry tetrahydrofuran (10 cm^3). *NNN'N'*-Tetramethylethylenediamine (tmen) (0.038 g, 0.33 mmol) was then added under dinitrogen and the mixture was stirred for 1 h at 0 °C. Solid $[Mo(CO)_4\{(Ph_2P)_2C{=}CH_2\}]$ (0.20 g, 0.33 mmol) was then added to the warm (21 °C) stirred mixture; the yellow solution changed immediately through orange to brown in 1 h, and black after 18 h. The solvent was evaporated under reduced pressure

Table 4. Selected bond lengths (pm) and angles (°) for $[W(CO)_4\{(Ph_2P)_2C{=}CHCH{=}CHPh\}]$ (**2a**)

P(1)–W	251.3(4)	C(131)–C(132)	143.5(9)
P(2)–W	250.9(4)	C(133)–C(134)	144.8(8)
C(1)–W	198.0(9)	H(131)–C(131)	96.8(52)
C(2)–W	203.6(9)	H(132)–C(132)	90.7(62)
C(3)–W	202.1(9)	H(133)–C(133)	105.5(70)
C(4)–W	202.1(10)	O(1)–C(1)	116.2(8)
C(13)–P(1)	182.2(7)	O(2)–C(2)	114.0(8)
C(13)–P(2)	183.0(7)	O(3)–C(3)	113.8(8)
C(131)–C(13)	133.9(9)	O(4)–C(4)	115.2(9)
C(132)–C(133)	131.6(9)		
P(2)–W–P(1)	68.1	O(2)–C(2)–W	175.9(6)
P(2)–C(13)–P(1)	100.7(4)	O(4)–C(4)–W	177.6(7)
C(132)–C(131)–C(13)	126.6(7)	H(131)–C(131)–C(13)	117.2(30)
C(134)–C(133)–C(132)	127.1(7)	C(132)–C(131)–H(131)	116.1(31)
O(1)–C(1)–W	175.9(6)	H(133)–C(133)–C(132)	120.0(34)
O(3)–C(3)–W	173.4(6)	C(134)–C(133)–H(133)	112.7(34)

Table 5. Atom co-ordinates (× 10^4)

Atom	*x*	*y*	*z*
W	2 181.0(2)	1 614.4(2)	1 197.0(1)
P(1)	3 347(2)	2 724(1)	641(1)
P(2)	3 312(2)	3 592(1)	1 460.9(4)
C(111)	2 312(4)	3 471(3)	218(1)
C(112)	1 282(4)	2 755(3)	15(1)
C(113)	500(4)	3 248(3)	−325(1)
C(114)	749(4)	4 457(3)	−462(1)
C(115)	1 779(4)	5 172(3)	−259(1)
C(116)	2 561(4)	4 679(3)	82(1)
C(121)	4 775(4)	2 103(4)	383(1)
C(122)	4 744(4)	2 037(4)	−48(1)
C(123)	5 853(4)	1 552(4)	−236(1)
C(124)	6 992(4)	1 133(4)	8(1)
C(125)	7 023(4)	1 199(4)	440(1)
C(126)	5 915(4)	1 684(4)	627(1)
C(13)	4 086(6)	3 949(5)	980(2)
C(211)	4 634(4)	3 656(4)	1 890(1)
C(212)	4 724(4)	4 627(4)	2 179(1)
C(213)	5 762(4)	4 630(4)	2 499(1)
C(214)	6 712(4)	3 663(4)	2 529(1)
C(215)	6 623(4)	2 692(4)	2 240(1)
C(216)	5 584(4)	2 688(4)	1 921(1)
C(221)	2 200(4)	4 883(3)	1 571(1)
C(222)	2 284(4)	6 053(3)	1 384(1)
C(223)	1 429(4)	7 019(3)	1 490(1)
C(224)	490(4)	6 816(3)	1 782(1)
C(225)	406(4)	5 646(3)	1 969(1)
C(226)	1 261(4)	4 680(3)	1 863(1)
C(131)	5 095(7)	4 740(6)	913(2)
C(132)	5 657(7)	5 683(6)	1 193(2)
C(133)	6 550(7)	6 543(6)	1 113(2)
C(134)	7 080(4)	7 528(3)	1 388(1)
C(135)	6 889(4)	7 512(3)	1 810(1)
C(136)	7 387(4)	8 488(3)	2 065(1)
C(137)	8 075(4)	9 481(3)	1 898(1)
C(138)	8 266(4)	9 498(3)	1 475(1)
C(139)	7 769(4)	8 521(3)	1 220(1)
C(1)	1 213(7)	295(6)	861(2)
O(1)	577(6)	−431(5)	660(2)
C(2)	3 712(7)	355(7)	1 308(2)
O(2)	4 511(6)	−406(5)	1 368(2)
C(3)	428(8)	2 578(6)	1 091(2)
O(3)	−620(6)	3 023(5)	1 046(2)
C(4)	1 499(7)	1 144(6)	1 748(3)
O(4)	1 121(6)	918(6)	2 067(2)

and the residue was triturated with methanol (3 cm^3) to give the required product as yellow microcrystals. Yield 0.23 g (97%). The product could be recrystallized from dichloromethane–ethanol as yellow needles.

The corresponding tungsten, (**1c**), and chromium, (**1a**), complexes were made similarly (98 and 91% yields respectively).

$[Mo(CO)_4\{(Ph_2P)_2CHCH_2C\equiv CC_6H_4Me\text{-}p\}]$ (**1d**). This complex was made in a similar fashion to the above complexes using lithium *p*-tolylacetylide in tetrahydrofuran. Yield 81%.

$[Mo(CO)_4\{(Ph_2P)_2CHCH_2C\equiv CBu^n\}]$ (**1e**). A solution of lithium n-butylacetylide was made by the addition of butyllithium (1.7 mol dm^{-3} in hexane, 0.19 cm^{-3}, 0.33 mmol) to a cooled (0 °C) solution of n-butylacetylene (0.028 g, 40 μl, 0.35 mmol) in dry tetrahydrofuran (10 cm^3). *NNN′N′*-Tetramethylethylenediamine (0.038 g, 0.33 mmol) was added and the mixture was stirred for 1 h at (0 °C) under nitrogen. The solid $[Mo(CO)_4\{(Ph_2P)_2C=CH_2\}]$ (0.20 g, 0.33 mmol) was then added to the warm mixture (21 °C), which changed through orange-red to deep red and brown-black in 1 h. The solvent was evaporated under reduced pressure and the residue was triturated with methanol (3 cm^3) to give the required product as yellow microcrystals. Yield 0.21 g (93%). The product could be recrystallized from dichloromethane as yellow needles.

The corresponding tungsten complex, (**1h**), was made similarly. Yield 40%.

$[Mo(CO)_4\{(Ph_2P)_2CHCH_2C\equiv CMe\}]$ (**1f**). A solution of lithium methylacetylide was made by the addition of butyllithium (1.7 mol dm^{-3} in hexane, 0.194 cm^3, 0.33 mmol) to a cooled solution (0 °C) of methylacetylene (0.033 g, 20 cm^3, 0.832 mmol) in dry tetrahydrofuran (5 cm^3). *NNN′N′*-Tetramethylethylenediamine (0.038 g, 0.33 mmol) was added and the mixture was stirred for 30 min (0 °C) under dinitrogen. The solid $[Mo(CO)_4\{(Ph_2P)_2C=CH_2\}]$ (0.20 g, 0.33 mmol) was added to the solution (21 °C), which changed colour to brown in 30 min. The solvent was evaporated under reduced pressure and the residue was triturated with methanol (3 cm^3) to give the yellow solid product. Yield 0.133 g (62%). The product could be recrystallized from dichloromethane–ethanol as yellow needles.

The corresponding chromium complex, (**1j**), was similarly prepared in 37% yield.

$[W(CO)_4\{(Ph_2P)_2CHCH_2C\equiv CPr^n\}]\cdot 0.33CH_2Cl_2$ (**1g**). This compound was made in a similar fashion to the above complex using lithium n-propylacetylide in tetrahydrofuran. Yield 46%. The corresponding molybdenum complex, (**1i**), was made similarly, in 47% yield.

$[W(CO)_4\{(Ph_2P)_2C=CHCH=CPh\}]$ (**2a**). A suspension of $[W(CO)_4\{(Ph_2P)_2CHCH_2C\equiv CPh\}]$ (0.10 g, 0.13 mmol) in propan-2-ol (3 cm^3) and $KOBu^t$ (0.050 g, 0.45 mmol) was heated under reflux for 1.5 h under nitrogen. The resultant deep green solution and orange solid were cooled to room temperature and dilute hydrochloric acid (*ca.* 1 cm^3) was added to neutralize the excess of base. The deep red product formed was filtered off, washed with diethyl ether (2 cm^3), and dried *in vacuo*. Yield 0.092 g (91%). The product could be crystallized from dichloromethane–ethanol as deep red prisms. The corresponding *p*-tolyl complex, (**2b**), was made and isolated similarly.

X-Ray Crystallography.—Crystals suitable for single-crystal diffraction analysis were obtained by diffusion of ethanol into a CH_2Cl_2 solution of complex (**2a**).

Crystal data. $C_{38}H_{28}O_4P_2W$, $M = 794.435$, monoclinic, $a = 988.4(3)$, $b = 1\,069.7(2)$, $c = 3\,233.7(4)$ pm, $\beta = 94.73(2)°$, $U = 3.407$ nm^3, space group $P2_1/c$, $Z = 2$, $D_c = 1.55$ g cm^{-3}, $\mu = 33.52$ cm^{-1}, $F(000) = 1\,608$.

Data collection. Scans running from 0.9° below $K_{\alpha 1}$ to 0.9° above $K_{\alpha 2}$, scan speeds 2.0—29.3° min^{-1}, $4.0 < 2\theta < 45.0°$. 4 245 Unique data collected, 3 929 observed $[I > 2.0\sigma(I)]$, $T = 290$ K.

Structure refinement. Weighting factor $g = 0.000\,05$, number of parameters = 363, $R = 0.0334$, $R' = 0.0316$.

All crystallographic measurements were made on a Nicolet P3/F diffractometer operating in the ω–2θ scan mode using graphite-monochromatized Mo-K_α radiation ($\lambda = 71.069$ pm) following a standard procedure described in detail elsewhere.[8] The data set was corrected for absorption empirically once the structure had been solved.[9] The compound was solved *via* standard heavy-atom methods and refined by full-matrix least squares using the SHELX program system.[10] All non-hydrogen atoms were assigned anisotropic thermal parameters with phenyl rings included in the refinement as rigid bodies with idealized hexagonal symmetry (C–C 139.5 pm). Phenyl hydrogen atoms were included in calculated positions (C–H 108 pm) and were refined with an overall isotropic thermal parameter. All other hydrogen atoms were located in difference syntheses and freely refined with individual isotropic thermal parameters. The weighting scheme $w = [\sigma^2(F_o) + g(F_o)]^{-1}$ was used at the end of refinement in order to obtain acceptable agreement analyses. Final non-hydrogen atomic co-ordinates are given in Table 5.

Acknowledgements

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