Preparation and Properties of *cis*-$[W(N_2)_2(Ph_2PCH_2PPh_2)_2]$ and *trans*-$[M(N_2)_2(Ph_2PCH{=}CHPPh_2)_2]$ (M = Mo or W), and the Crystal Structure of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$†

Hengameh Dadkhah, Jonathan R. Dilworth, Kevin Fairman, Chi Tat Kan, and Raymond L. Richards *
A.F.R.C. Unit of Nitrogen Fixation and School of Chemistry and Molecular Sciences, University of Sussex, Brighton BN1 9RQ
David L. Hughes
Molecular Structures Department, Rothamsted Experimental Station, Harpenden, Herts AL5 2JQ

The complexes *cis*-$[W(N_2)_2(Ph_2PCH_2PPh_2)_2]$ (**A**) and *trans*-$[W(N_2)_2(Ph_2PCH{=}CHPPh_2)_2]$ (**B**) have been prepared by reduction of WCl_6 or $[WCl_4(Ph_2PCH{=}CHPPh_2)]$ with Mg in tetrahydrofuran (thf) under N_2 in the presence of the appropriate phosphine. Dinitrogen is displaced from (**A**) by CO to give *cis*-$[W(CO)_2(Ph_2PCH_2PPh_2)_2]$. Compound (**B**) reacts with I_2, $FeCl_3$, and $CuCl_2$ to give $[W(N_2)_2(Ph_2PCH{=}CHPPh_2)_2]Y$ (Y = I_3, $FeCl_4$, or $CuCl_2$) and with HCl to give the cation $[WCl(NNH_2)(Ph_2PCH{=}CHPPh_2)_2]^+$. Both complexes (**A**) and (**B**) undergo reversible one-electron oxidation in thf. The complex *trans*-$[Mo(N_2)_2(Ph_2PCH{=}CHPPh_2)_2]$ gives *trans*-$[Mo(N_2)(RCN)(Ph_2PCH{=}CHPPh_2)_2]$ (R = Me or Et) on treatment with RCN. *X*-Ray analysis of the related complex, *cis*-$[W(N_2)_2(PMe_2Ph)_4]$, shows a well defined octahedral structure in which the dinitrogen ligands have different environments and the W–N distances are 1.983(3) and 2.015(3) Å.

The properties of complex compounds of phosphine ligands often vary considerably depending upon the nature of the ligands. Thus increasing bulk of the phosphine can lead to unusual co-ordination numbers and stereochemistry or a small bite angle of a chelate may lead to the formation of binuclear species.[1,2] We and others have extensively investigated dinitrogen complexes of molybdenum and tungsten generally carrying the phosphine co-ligands PMe_2Ph and $Ph_2PCH_2CH_2PPh_2$ (dppe).[3] Herein we describe the chemistry of analogous dinitrogen complexes derived from the ligands $Ph_2PCH_2PPh_2$ (dppm) and $Ph_2PCH{=}CHPPh_2$ (dppen) which we used to see whether the smaller bite angle of dppm or unsaturation of dppen caused any change of their chemistry relative to that previously observed with other co-ligands. We also describe the crystal structure of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$, the first reported determination for a mononuclear dinitrogen complex of tungsten.

Results and Discussion

Treatment of WCl_6 with Mg in tetrahydrofuran (thf) under N_2 with dppm gives orange *cis*-$[W(N_2)_2(dppm)_2]$ (**A**) in moderate yield (15—25%). The yield depends critically on the reaction conditions, poor yields resulting if the molar ratio of dppm to WCl_6 is greater than 2:1 or the reaction time exceeds 3 h. The complex *trans*-$[W(N_2)_2(dppen)_2]$ (**B**) was obtained in 30% yield by magnesium reduction of $[WCl_4(dppen)]$ in thf in the presence of dppen under dinitrogen at *ca.* 60 °C. A lower yield results if the reaction is carried out at lower temperature because the necessarily long reaction time (*ca.* 10 h) allows competing decomposition reactions to occur.

The *cis* structure of (A) follows from its two N_2-stretching i.r. bands (Table 1) and its ^{31}P n.m.r. spectrum. The latter consists of two doublets centred at −153.18 and −161.51 p.p.m. [thf solvent, $P(OMe)_3$ reference]. Singlet N_2-stretching and ^{31}P resonance absorptions would be expected for a *trans* structure, as are observed for (**B**). The ^{31}P spectrum of complex (A) might be expected to be more complicated, *i.e.* a doublet of triplets, since the system is formally of A_2X_2 type rather than the AB type observed. This can be explained if the observed P–P coupling is $|J_{PP}|_{obs.} = |J_{PP}{}^{b} \pm J_{PP}{}^{m}|$ where $J_{PP}{}^{b}$ = coupling through the ligand backbone and $J_{PP}{}^{m}$ = coupling through the metal.[4] If $J_{PP}{}^{b}$ and $J_{PP}{}^{m}$ are equal and opposite, coupling between the phosphorus atoms on the same ligand can effectively cancel and the system then reduces to the AB type, the observed coupling being between the non-equivalent phosphorus atoms from each ligand across the metal. The factors controlling the *cis*/*trans* geometry of the dinitrogen complexes are subtle, since the analogues of (A) with $Ph_2PCH_2CH_2PPh_2$, $Ph_2PCH{=}CHPPh_2$, and $PMePh_2$ are *trans*, whereas (A) and the complex with PMe_2Ph are *cis* (see below).[3,5]

Both complexes (A) and (**B**) undergo reversible one-electron oxidations in thf containing 0.2 mol dm^{-3} $[NBu_4{}^n][BF_4]$. The $E_{\frac{1}{2}}{}^{ox}$ of (A) [−0.15 V relative to the standard calomel electrode (s.c.e.)] is identical to that of its dppe analogue,[6] but that of (**B**) (0.00 V) is high relative to both. Presumably the unsaturated backbone of dppen lowers its electron-donor capacity relative to dppm and dppe so that the tungsten atom of (**B**) is somewhat less electron rich than those of its analogues. This does not, however, appear to lead to any marked loss of reactivity (see below).

As might be expected on the basis of the electrochemical data, chemical oxidation of (**B**) (I_2, $FeCl_3$, or $CuCl_2$) leads to the isolation of the stable cation $[W(N_2)_2(dppen)_2]^+$ as its $I_3{}^-$, $FeCl_4{}^-$, or $CuCl_2{}^-$ salts (Table 1). Similar oxidations of (**A**), however, lead to the loss of dinitrogen and the formation of uncharacterised products, although clearly the cation $[W(N_2)_2(dppm)_2]^+$ has a finite lifetime under electrochemical conditions.

A stable hydrazide complex $[WCl(NNH_2)(dppen)_2][BPh_4]$

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† *cis*-Tetrakis(dimethylphenylphosphine)bis(dinitrogen)tungsten.

Supplementary data available (No. SUP 56236, 6 pp.): thermal parameters, H-atom co-ordinates, complete bond lengths and angles. See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1985, Issue 1, pp. xvii—xix. Structure factors are available from the editorial office.

Non-S.I. units employed: mmHg ≈ 133 Pa, B.M. ≈ 9.27 × 10^{-24} A m^2.

Table 1. Compounds of molybdenum and tungsten

Compound	Colour	Analysis (%)[a] C	H	N	I.r.[b] (cm^{-1})	N.m.r.[c]	Other data
(**A**) *cis*-$[W(N_2)_2(dppm)_2]$	Orange	60.2 (60.1)	4.8 (4.2)	5.5 (4.5)	1 935s (N_2) 2 000s (N_2)	δ_H 4.5 (CH_2), 6.7—7.75 (Ph) δ_P −153.18(dd), −161.51(dd); J_{PP} 6 Hz	
(**B**) *trans*-$[W(N_2)_2(dppen)_2]$	Orange	60.5 (60.6)	4.5 (4.1)	5.4 (5.4)	1 965s (N_2) 2 022w (N_2)	δ_H 6.7—7.5 (Ph) δ_P −80.19	*M* 1 005 (1 036)[d]
trans-$[W(N_2)_2(dppen)_2][I_3]$	Red	44.2 (44.1)	2.8 (3.4)	4.0 (4.0)	2 020s (N_2)		Λ[d] 53.45 S cm^2 mol^{-1} μ 1.91 B.M.
trans-$[W(N_2)_2(dppen)_2][FeCl_4]$	Red	50.3 (50.6)	3.8 (3.9)	4.3 (4.5)	2 020s (N_2)		Λ[d] 60.15 S cm^2 mol^{-1}
trans-$[W(N_2)_2(dppen)_2][CuCl_2]$	Red	53.5 (53.3)	4.3 (4.1)	4.4 (4.8)	2 020s (N_2)		Λ[d] 57.85 S cm^2 mol^{-1}
$[WCl(NNH_2)(dppen)_2][BPh_4]$	Yellow	65.0 (64.0)	5.0 (4.6)	1.7 (1.9)	3 250 (NH) 3 440 (NH)	δ_H 6.7—7.5 (Ph)	Λ[d] 52.65 S cm^2 mol^{-1}
trans-$[Mo(N_2)(MeCN)(dppen)_2]$	Red-brown	66.8 (67.6)	5.5 (5.2)	4.8 (4.4)	1 942s (N_2) 2 220m (N_2)	*e*	
trans-$[Mo(N_2)(EtCN)(dppen)_2]$	Red-brown	67.0 (67.2)	6.3 (5.9)	4.3 (4.0)	1 945s (N_2) 2 202m (NC)	δ_H 6.8—7.5 (Ph), 1.0—1.5 (t, CH_3CH_2CN), 2.1—2.3 (q, CH_2CN)	*M* 839 (1 020)[d,e]

[a] Calculated values in parentheses. [b] Nujol mulls, s = strong, m = medium, and w = weak. [c] In $C^2H_2Cl_2$ solvent, δ_H relative to $SiMe_3$, δ_P relative to $P(OMe)_3$. dd = Doublet of doublets, t = triplet, and q = quartet. [d] In *ca.* 10^{-3} mol dm^{-3} $C_2H_4Cl_2$ solution. [e] Decomposition in solution.

was also isolated after treatment of (**B**) with anhydrous HCl [reaction (1)]. It resembles its analogues with dppe[7] (Table 1).

$$trans\text{-}[W(N_2)_2(dppen)_2] \xrightarrow[\text{toluene}]{\text{HCl (excess)}} [WCl(NNH_2)(dppen)_2]Cl \xrightarrow[\text{MeOH}]{\text{Na[BPh}_4]} [WCl(NNH_2)(dppen)_2][BPh_4] \quad (1)$$

Dinitrogen is displaced from (**A**) by CO in thf to give the known complex *cis*-$[W(CO)_2(dppm)_2]$.[8]

Although *trans*-$[Mo(N_2)_2(dppen)_2]$ (**C**) has been known for some time,[9] there has been little investigation of its chemical properties, mainly because its solubility is surprisingly low compared to that of (**B**), which may indicate some structure difference which is not apparent from the usual spectroscopic properties. So far, unfortunately, it has not proved possible to obtain suitable crystals for *X*-ray analysis. The poor solubility has restricted our investigations, but treatment of (**C**) with refluxing RCN (R = Me or Et) leads to the formation of $[Mo(N_2)(NCR)(dppen)_2]$ (Table 1) whose analogues with dppe as ligand are well known.[10] Thus one would conclude that the chemical properties of (**C**) are dominated by its solubility rather than any noticeable electronic difference from its analogues.

We also include in this paper the crystal structure of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$ because of its relevance to the structure of *cis*-$[W(N_2)_2(dppm)_2]$. Crystal structures of tungsten dinitrogen complexes are few, the only other so far published being that of $[\{W(N_2)_2(PEt_2Ph)_3\}_2(\mu\text{-}N_2)]$ which could not be accurately refined.[11] We have, however, also obtained the structures[12] of *trans*-$[W(N_2)_2(PEt_2Ph)_4]$ and $[W(\eta^6\text{-}C_6H_5PPr^n{}_2)(N_2)(PPr^n{}_2Ph)_2]$, which will be reported in detail elsewhere.

Crystal Structure of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$.—The results of the *X*-ray analysis show a well defined structure in which the discrete molecules of the complex pack tightly together and allow precise measurement of molecular dimensions. The slight distortions from a perfect octahedral co-ordination of the two *cis* dinitrogen and four phosphine ligands (Figure) result from steric effects; there are many interligand and intermolecular contacts which are rather shorter than the sums of van der Waals radii.

Table 2. Principal molecular dimensions [bond lengths (Å) and angles (°)] about the W atom and in the dinitrogen ligands of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$. Estimated standard deviations (e.s.d.s) are in parentheses; those for P–W–P angles are less than 0.05°

W–P(1)	2.480(1)	P(1)–W–P(3)	166.3
W–P(2)	2.481(1)	Other P–W–P	91.2—99.3
W–P(3)	2.444(1)	P(2)–W–N(5)	173.5(1)
W–P(4)	2.473(1)	P(4)–W–N(6)	176.9(1)
W–N(5)	1.983(3)	Other P–W–N	83.3—89.8
W–N(6)	2.015(3)	N(5)–W–N(6)	88.9(1)
N(5)–N(51)	1.125(4)	W–N(5)–N(51)	177.8(3)
N(6)–N(61)	1.120(4)	W–N(6)–N(61)	178.0(3)

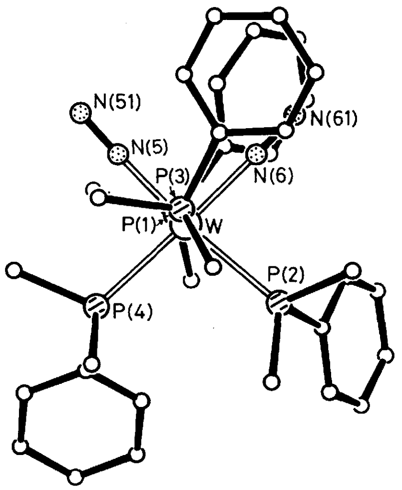

Figure. A molecule of *cis*-$[W(N_2)_2(PMe_2Ph)_4]$ projected on to the plane of W,N(5),N(6). In the phosphine ligand of P(*n*), the C atoms of the phenyl ring are labelled C(*n*1)—C(*n*6) and the methyl C atoms are C(*n*7) and C(*n*8)

The W–P bond distances and those in the linear W–N–N systems all lie in the normal ranges and are listed in Table 2, but there are significant differences in the supposedly equivalent dimensions, and these too appear to arise from steric interactions. The phosphine ligands pack tightly together, *e.g.* (*i*) the plane of P(1),C(17),C(18) is nearly parallel to that of P(4),C(41)–C(46),C(48), and the distance C(18) ··· C(42) is 3.315(5) Å, (*ii*) the plane of P(1),C(11)–C(16),C(18) is approximately parallel to that of P(2),C(21)–C(26). The contacts within the W(phosphine)$_4$ group are not symmetrical and result in different nitrogen ··· phosphine interactions. The Figure shows P(1), almost hidden behind W and P(3), and P(3) distorted much more towards N(5) than N(6). The phenyl groups in these two ligands make close contact with N(6) and N(61), and the W–N(6) bond is thus extended rather more than W–N(5).

The ^{15}N n.m.r. spectrum of *cis*-[W(N$_2$)$_2$(PMe$_2$Ph)$_4$] showed a significant deshielding of the terminal (N$_\beta$) nitrogen atom relative to those of analogous compounds of *trans* geometry.[13] Thus one might expect to see some interaction of the *cis*-N$_2$ ligands in *cis*-[W(N$_2$)$_2$(PMe$_2$Ph)$_4$]. Such an interaction is not observed in the solid state, however, and possibly the ^{15}N n.m.r. technique is a more sensitive indicator of such interactions in solution than is the solid-state structural method. The asymmetric N$_2$ environment observed in the solid is not detected in the ^{15}N spectrum, which shows no line broadening, and is probably absent in solution.

Conclusions

The chelating phosphine ligands dppm and dppen affect the geometry and solubility of their complexes of molybdenum and tungsten with dinitrogen. However, electronic differences between these complexes and their analogues with dppe appear to be too minor to lead to pronounced differences in patterns of reactivity.

Experimental

The manipulation of air-sensitive materials, preparation of phosphine ligands and *cis*-[W(N$_2$)$_2$(PMe$_2$Ph)$_4$], purification of solvents, and electrochemistry followed standard techniques.[5,6] Spectra were determined with Unicam SP200 and JEOL FX90Q instruments and magnetic moments with a Faraday balance.

cis-*Bis*[*bis*(*diphenylphosphino*)*methane*]*bis*(*dinitrogen*)*tungsten*(0), *cis*-[W(N$_2$)$_2$(dppm)$_2$] (**A**).—Magnesium turnings (1 g) and WCl$_6$ (1 g, 2.5 mmol) were added to a solution of dppm (1 g, 2.4 mmol) in thf (100 cm^3). The resulting solution was stirred under N$_2$, when the colour changed from brown through greenish yellow to orange. After about 1 h the solution was filtered, concentrated under reduced pressure to about 20 cm^3, and MeOH (50 cm^3) added to give a yellow-brown solid which gave orange *crystals* (0.6 g, 25%) from thf–Et$_2$O.

trans-*Bis*[1,2-*bis*(*diphenylphosphino*)*ethene*]*bis*(*dinitrogen*)*tungsten*(0), *trans*-[W(N$_2$)(dppen)$_2$] (**B**).—Magnesium (1 g) and dppen (0.5 g, 1.2 mmol) were added to a suspension of [WCl$_4$(dppen)] (0.9 g, 1.2 mmol) in thf (100 cm^3). The mixture was stirred under N$_2$ at 60 °C for 3 h when the colour changed from greenish yellow to orange. The solution was then filtered, concentrated under reduced pressure to about 20 cm^3, and MeOH (50 cm^3) added to give a yellow-orange solid which gave orange *crystals* (0.4 g, 30%) from thf–Et$_2$O.

trans-*Bis*[1,2-*bis*(*diphenylphosphino*)*ethene*]*bis*(*dinitrogen*)*tungsten*(I) *Tri-iodide*, *trans*-[W(N$_2$)$_2$(dppen)$_2$][I$_3$].—An excess of I$_2$ (1 g) was added to a suspension of complex (**B**) (0.5 g) in MeOH (20 cm^3). The reaction mixture was stirred under N$_2$ for 3 h to produce a red *solid* which was filtered off, washed with thf, MeOH, and Et$_2$O, and dried (10^{-3} mmHg) (0.55 g, 80%). Similarly prepared were the FeCl$_4{}^-$ salt (from FeCl$_3$, 85%) and the CuCl$_2{}^-$ salt (from CuCl$_2$, 85%).

Bis[1,2-*bis*(*diphenylphosphino*)*ethene*]*chloro*[*hydrazido*-(2−)]*tungsten*(IV) *Tetraphenylborate*, [WCl(NNH$_2$)(dppen)$_2$][BPh$_4$].—(*a*) Hydrogen chloride gas was passed through a solution of complex (**B**) (0.3 g) in toluene (20 cm^3) for 1 min, then the reaction mixture was stirred for 1 h under N$_2$ to give a yellow solid which was filtered off and dissolved in MeOH (20 cm^3). The mixture was stirred under N$_2$ until complete dissolution (1 h). Sodium tetraphenylborate was then added to precipitate a yellow *solid* which was filtered off and recrystallised from CH$_2$Cl$_2$–MeOH (0.3 g, 80%).

(*b*) Trimethylsilyl chloride (0.2 g) was added to a suspension of complex (**B**) (0.3 g) in MeOH (20 cm^3) and the mixture stirred under N$_2$ until complete dissolution (1 h). Sodium tetraphenylborate was then added to precipitate a yellow *solid* which was filtered off and recrystallised from CH$_2$Cl$_2$–MeOH (0.3 g, 80%).

Bis[1,2-*bis*(*diphenylphosphino*)*ethene*]*dinitrogen*(*propionitrile*)*molybdenum*(0)–*Tetrahydrofuran* (1/1), [Mo(N$_2$)(EtCN)(dppen)$_2$]·thf.—Complex (**C**) (0.5 g) was heated under reflux in EtCN (60 cm^3) for 2 h to give a red solution which was quickly cooled to 20 °C, giving a red solid which was filtered off and recrystallised from hot thf containing a drop of EtCN as red *crystals* (0.4 g, 80%). An analogous procedure was used to prepare the MeCN analogue as red *crystals* (80% yield).

Reactions of cis-[W(N$_2$)$_2$(dppm)$_2$].—(*a*) *With* CO. Carbon monoxide was passed into a solution of complex (**A**) (1.05 g) in thf (30 cm^3) for 24 h under tungsten light irradiation[5,6] to give a pale yellow solution. Concentration of the solution under reduced pressure to one third volume followed by addition of Et$_2$O (50 cm^3) gave yellow *crystals* (65%) of *cis*-[W(CO)$_2$(dppm)$_2$], identified by its spectroscopic properties[8] (Found: C, 61.5; H, 4.2. C$_{52}$H$_{44}$O$_2$P$_4$W requires C, 61.8; H, 4.4%).

(*b*) *With* H$_2$. Treatment of complex (**A**) (0.75 g) in thf (70 cm^3) with H$_2$ for 12 h under tungsten light irradiation led to a colour change from orange to pale yellow. Removal of solvent to about one third volume under reduced pressure followed by addition of Et$_2$O (60 cm^3) gave an impure yellow solid which exhibited no N$_2$ peak, nor hydride i.r. or n.m.r. absorptions (Found: C, 59.2; H, 4.4%).

X-*Ray Crystallographic Study of* cis-[W(N$_2$)$_2$(PMe$_2$Ph)$_4$].—Crystals of the complex from thf–MeOH solution are bright orange, and have a modified octahedral shape. Some have dimensions greater than 1 mm, but the specimen, mounted on a glass-fibre for study, was 0.15 × 0.25 × 0.35 mm. After photographic examination, accurate cell parameters and diffraction intensities were measured on an Enraf–Nonius CAD4 diffractometer with monochromated Mo-K_α radiation.

Crystal data. C$_{32}$H$_{44}$N$_4$P$_4$W, $M = 792.5$, monoclinic, space group $P2_1/n$ (equivalent to no. 14), $a = 18.653(2)$, $b = 13.995(2)$, $c = 13.322(3)$ Å, $\beta = 96.30(1)°$, $U = 3\,456.8(9)$ Å^3, $Z = 4$, $D_c = 1.523$ g cm^{-3}, $F(000) = 1\,592$, μ(Mo-K_α) = 36.2 cm^{-1}, λ(Mo-$K_{\bar{\alpha}}$) = 0.710 69 Å.

After correction for Lorentz-polarisation effects, slight deterioration (overall 3.8%) (the processing program CAD4[14]), and absorption (EMPABS[15]), and application of Bayesian methods to ensure no negative net intensities (using BAYES[16]), 6 066 independent reflections, with $\theta_{max.} = 25°$, were entered into the SHELX system.[17] The structure was solved by the heavy-atom method, and refinement by large-block-matrix least-squares procedures converged rapidly, giving $R = 0.027$ and $R' = 0.024$,[17] for 6 053 reflections (omitting only the lowest

Table 3. Final co-ordinates (fractional × 10^4) of the non-hydrogen atoms in *cis*-$[W(N_2)_2(PMe_2Ph)_4]$ with e.s.d.s in parentheses

Atom	*x*	*y*	*z*	Atom	*x*	*y*	*z*
W	3 092.6(1)	2 752.1(1)	5 451.6(1)	C(32)	4 941(2)	1 445(3)	7 227(3)
P(1)	1 954.1(4)	2 284.5(6)	4 431.8(6)	C(33)	5 337(2)	609(3)	7 374(3)
C(11)	1 801(2)	985(3)	4 346(2)	C(34)	5 681(2)	246(3)	6 604(4)
C(12)	1 173(2)	541(3)	4 587(3)	C(35)	5 625(2)	700(3)	5 699(4)
C(13)	1 115(3)	−454(4)	4 539(4)	C(36)	5 229(2)	1 547(3)	5 550(3)
C(14)	1 660(3)	−1 022(3)	4 246(4)	C(37)	4 647(2)	3 673(3)	7 310(3)
C(15)	2 278(3)	−578(3)	4 010(3)	C(38)	4 903(2)	3 669(3)	5 295(4)
C(16)	2 347(2)	410(3)	4 055(3)	P(4)	2 956.7(4)	4 437.1(6)	4 905.3(6)
C(17)	1 943(2)	2 556(3)	3 077(3)	C(41)	2 073(2)	5 044(2)	4 728(2)
C(18)	1 048(2)	2 732(3)	4 609(3)	C(42)	1 590(2)	4 878(2)	5 427(3)
P(2)	2 632.9(4)	2 957.5(6)	7 116.7(6)	C(43)	954(2)	5 390(3)	5 408(3)
C(21)	1 689(2)	2 637(2)	7 237(2)	C(44)	778(2)	6 068(3)	4 666(3)
C(22)	1 187(2)	3 256(3)	7 587(2)	C(45)	1 242(2)	6 232(3)	3 959(3)
C(23)	447(2)	2 957(3)	7 636(3)	C(46)	1 888(2)	5 731(2)	3 976(3)
C(24)	267(2)	2 050(3)	7 353(3)	C(47)	3 411(2)	5 393(2)	5 689(3)
C(25)	762(2)	1 430(3)	7 010(3)	C(48)	3 319(2)	4 686(3)	3 705(3)
C(26)	1 462(2)	1 724(2)	6 947(2)	N(5)	3 526(1)	2 467(2)	4 191(2)
C(27)	3 055(2)	2 154(3)	8 104(3)	N(51)	3 785(2)	2 282(2)	3 491(2)
C(28)	2 732(2)	4 090(2)	7 805(3)	N(6)	3 257(1)	1 379(2)	5 873(2)
P(3)	4 346.3(4)	3 029.0(6)	6 131.3(7)	N(61)	3 369(2)	622(2)	6 110(2)
C(31)	4 884(2)	1 933(2)	6 322(2)				

θ-angle reflections, affected by extinction) weighted according to $w = \sigma_F^{-2}$.

All non-hydrogen atoms were refined with anisotropic thermal parameters, and hydrogen atoms (in idealised positions on the phenyl rings, or in rigid methyl groups) were given independent isotropic thermal parameters which refined satisfactorily. Scattering factors were from ref. 18. The highest peaks in a final difference map (maximum 0.55 e $Å^{-3}$) were close to the tungsten atom.

Refined atomic co-ordinates are in Table 3. Geometrical calculations were made, and some tables prepared, using the program GEOM.[19] The figure was drawn by ORTEP.[20]

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Received 1*st November* 1984; *Paper* 4/1867