

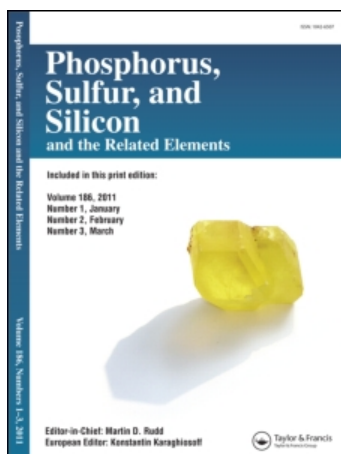
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Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

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To cite this Article Karaghiosoff, Konstantin and Schmidpeter, Alfred(1988) 'THE CHEMICAL SHIFT OF TWO-COORDINATE PHOSPHORUS, II^{1,2} HETEROCYCLES', Phosphorus, Sulfur, and Silicon and the Related Elements, 36: 3, 217 – 259

To link to this Article: DOI: 10.1080/03086648808079020

URL: <http://dx.doi.org/10.1080/03086648808079020>

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THE CHEMICAL SHIFT OF TWO-COORDINATE PHOSPHORUS, II^{1,2}

HETEROCYCLES

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(Received October 9, 1987)

A compilation of the chemical shifts of two-coordinate phosphorus in fully unsaturated cyclic compounds is presented. (Acyclic as well as partly unsaturated cyclic compounds are covered in Part I). Data are arranged according to the ring size and to the neighbours to the phosphorus in the ring. $\delta^{31}\text{P}$ tends to lower field in the series C, N, P for the neighbouring ring members; the whole range extends roughly from $\delta^{31}\text{P} = 470$ to 0 with most of the shifts between 300 and 50. Parallels to $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ in heterocycles with CH and N, respectively, in place of P are shown.

Compounds of two-coordinate phosphorus(III) derive their stability from either one of two effects: Bulky substituents can prevent the formation of di- or oligomers with phosphorus of co-ordination number three. Or two-coordination can be made the intrinsically more stable alternative by including the phosphorus into a delocalized π -system.³ The acyclic and not fully unsaturated cyclic compounds covered in Part I of this review belong to the one or other group. This second part covers two-coordinate phosphorus heterocycles of maximal unsaturation; they belong to the second group alone. As a consequence their ^{31}P chemical shift range (extending essentially from $\delta^{31}\text{P} = 470$ to 0) is considerably smaller than the range in Part I (extending essentially from $\delta^{31}\text{P} = 600$ to -300).¹ In the majority of the compounds of this part the two-coordinate phosphorus participates in a cyclic $(4n + 2)\pi$ -system. In some cases the cyclic delocalization may be debated. Some borderline cases are included here although they are already contained in Part I.

Some ^{31}P chemical shifts of two-coordinate phosphorus in both acyclic and cyclic compounds have been collected in recently published books.^{4,5}

ORGANIZATION OF THE $\delta^{31}\text{P}$ COMPILATION

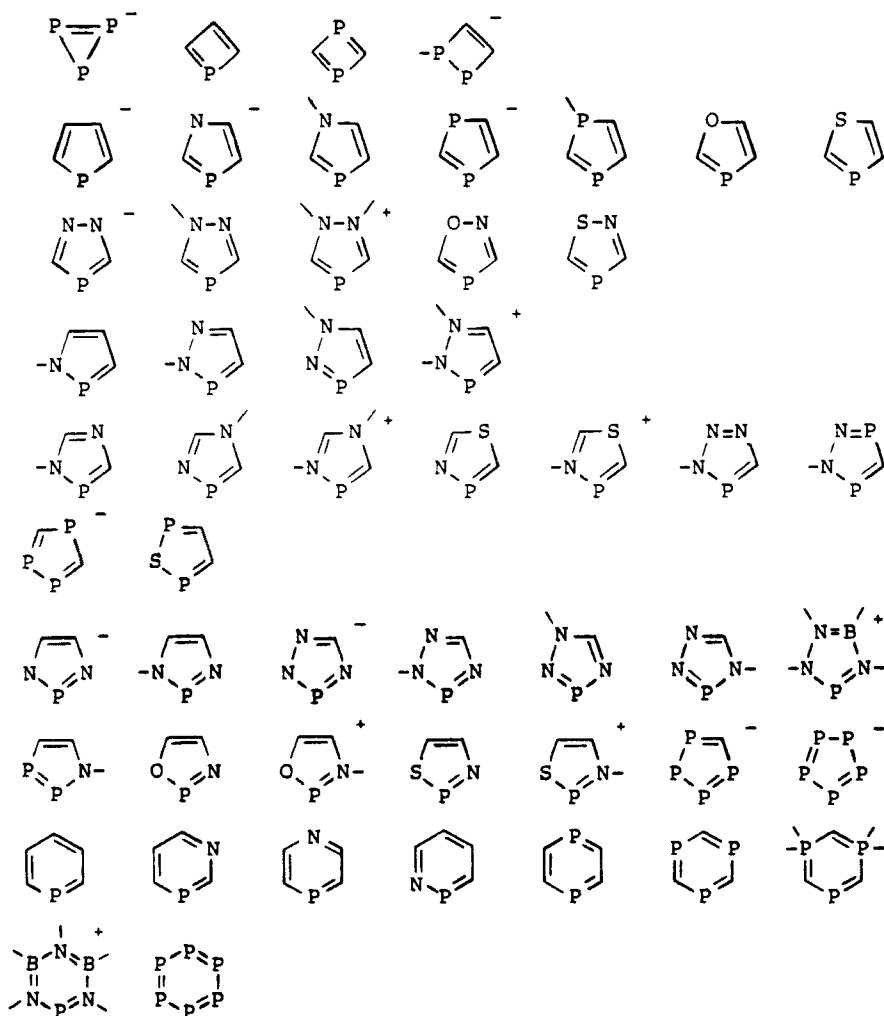
The compilation (Table I) is meant to include all pertinent data reported up to summer 1987. The compounds are arranged according

- (1) to the ring size,
- (2) to the nature of the ring members adjacent to the phosphorus,
- (3) to the remaining ring members,
- (4) to the number of the substituents at the ring,
- (5) to the nature of these substituents.

For (2) and (3) the ring members are ordered according to the PSE: C, N, P, O, S. Two different locations may result for a ring containing two non-equivalent two-coordinate phosphorus members; in such a case the ring is found at the second location. The same principle of order as before applies to the α -atom of the substituents: H, C, Si, Ge, Sn, N, P, As, Sb, O, S, F, Cl, Br, I.

Condensed systems are treated together with the parent phosphorus heterocycle. σ - and π -complexes are also included (in contrast to Part I even if the phosphorus two-coordination is not preserved in the complex formation).

The scheme below shows the known delocalized monocyclic systems of two-coordinate phosphorus ordered as in Table I according to points (1) to (3). It starts with three- and four-membered rings which may be regarded as 4π -systems (triphosphirene, phosphete and 1,3-diphosphete) and which are known only as η^3 - and η^4 -complexes, respectively. Almost all the five- and six-membered heterocycles can be considered as 6π -systems.⁶ Two eight-membered rings already contained in Part I are not repeated here.



$\delta^{31}\text{P}$ RANGES

In Figure 1 the phosphorus chemical shifts of Table I are classified according to the immediate surrounding of phosphorus and to the ring size. The other ring members and the substituents prove to be of similar influence however; thus only broad and overlapping ranges result. Four of them are enough populated to allow some statements nevertheless: Phosphorus is less shielded in six-membered rings than in five-membered rings, as shown for the CPC case. A nitrogen instead of carbon as neighbour also causes a phosphorus shift to lower field, as shown by the five-membered rings with CPC, CPN, and NPN fragments; a much more pronounced downfield shift is caused by phosphorus neighbours.

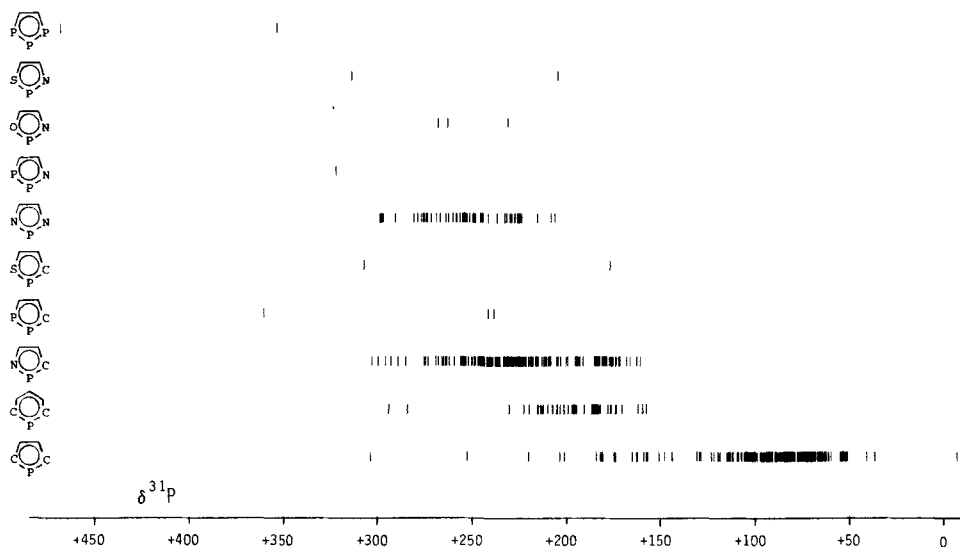
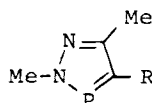


FIGURE 1 The chemical shifts $\delta^{31}\text{P}$ from Table I arranged according to the ring members adjacent to phosphorus and to the ring size.

The effect of an α -substituent on $\delta^{31}\text{P}$ is similar to that in the acyclic case¹; the 2,5-dimethyl-1,2,3-diazaphospholes provide a representative example for it:⁷



R	$\delta^{31}\text{P}$	R	$\delta^{31}\text{P}$	R	$\delta^{31}\text{P}$	R	$\delta^{31}\text{P}$
PSF ₂	275	POP _h ₂	252	P(CN) ₂	257	C(CF ₃) ₂ OH	237
PSFCl	269	PO(OMe) ₂	247	PCl ₂	249	SPh	236
PSCl ₂	251			PBr ₂	249	H	229
PS(OMe) ₂	254			PPhCl	243	Me	226
PSBr ₂	249			PPh ₂	241	Br	217
PSClMe	249			PMeCl	240	Cl	212
PSClPh	248			P(OMe) ₂	240	NHPh	172

$\delta^{13}\text{C}/^{31}\text{P}$ AND $\delta^{15}\text{N}/^{31}\text{P}$ CORRELATIONS

The properties of the phosphorus heterocycles discussed here suggest bonding situation comparable to that of the parent compounds in which a CH unit occupies the site of phosphorus. On this assumption the effects of the molecular environment on the chemical shift of this carbon and on $\delta^{31}\text{P}$ in the corresponding phospho derivative should parallel each other. For 37 pairs of compounds identical except for the CH/P replacement $\delta^{13}\text{C}$ and $\delta^{31}\text{P}$ data were found available.⁸ They are plotted in Figure 2 and clearly demonstrate the expected parallel between the carbon and phosphorus chemical shift. $\delta^{31}\text{P}$ responds, however, much more sensitive (almost five times as strongly) to the effects than $\delta^{13}\text{C}$. The shown $\delta^{13}\text{C}/^{31}\text{P}$ correlation is closely related to the one found for methylene and phosphinidene compounds in Part I.¹

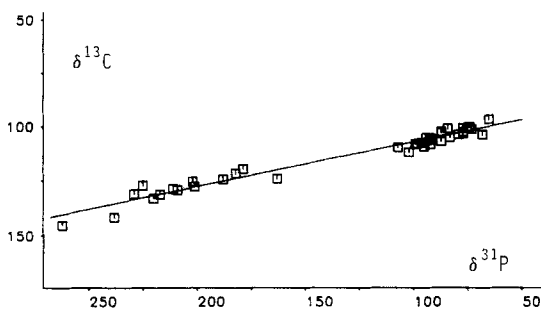
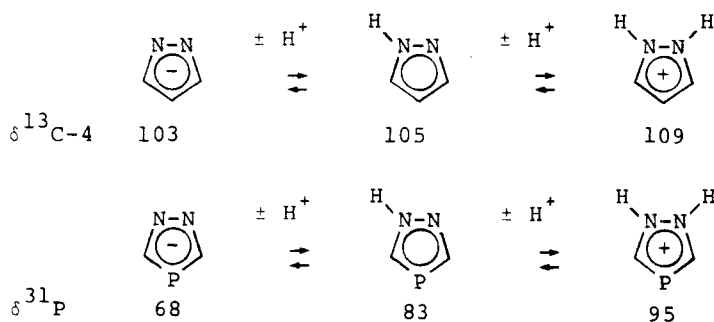


FIGURE 2 Correlation of $\delta^{31}\text{P}$ in two-coordinate phosphorus heterocycles (from Table I) with $\delta^{13}\text{C}$ of the CH unit replacing the phosphorus in otherwise identical heterocycles.⁸ A linear dependency is found (correlation coefficient 0.980): $\delta^{13}\text{C} = 0.205\delta^{31}\text{P} - 86.5$.

A specific example for the parallel response of $\delta^{13}\text{C}$ and $\delta^{31}\text{P}$ is provided by the two-step protonation of the pyrazole and of the 1,2,4-diazaphosphole anion. The ^{13}C -4 as well as the ^{31}P resonance shifts to lower field in both steps. Here again the effect on the former is much smaller than on the latter:



The comparison between nitrogen and phosphorus chemical shifts in corresponding aza- and phospho-heterocycles is somewhat hampered by the lack of $\delta^{15}\text{N}$ data. The pairs available⁹ are plotted in Figure 3.

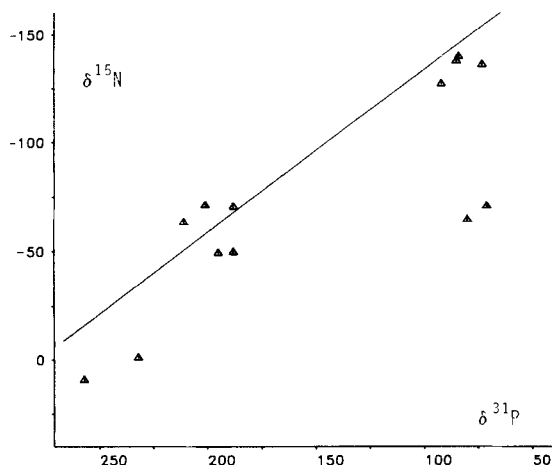


FIGURE 3 Correlation of $\delta^{31}\text{P}$ in two-coordinate phosphorus heterocycles (from Table I) with $\delta^{15}\text{N}$ of the specific nitrogen replacing the phosphorus in otherwise identical heterocycles.⁹ The straight line is taken from a broader $\delta^{15}\text{N}/^{31}\text{P}$ correlation.¹⁰ Its slope is much steeper than that of the $\delta^{13}\text{C}/^{31}\text{P}$ correlation: $\delta^{15}\text{N} = 0.75\delta^{31}\text{P} - 209$.

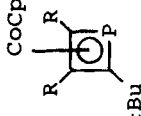
ACKNOWLEDGMENTS

We thank all colleagues who made unpublished data available to us. Thanks are also due to the Fonds der Chemischen Industrie for its support.

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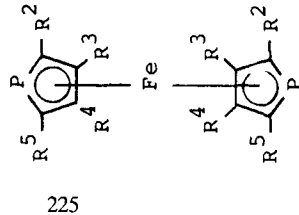
TABLE I
Chemical shifts $\delta^{31}\text{P}$ of dicoordinate phosphorus heterocycles

	$\delta^{31}\text{P}_A$	$\delta^{31}\text{P}_{B,C}$	J_{PP}	Lit.
ML_n 	$\text{ML}_n = \text{Cr}(\text{C}_3\text{Me}_3)(\text{CO})_2$ $\text{MoCp}(\text{CO})_2$ $\text{Mo}(\text{C}_3\text{Me}_3)(\text{CO})_2$ $\text{WCp}(\text{CO})_2$ $\text{Co}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Rh}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Ir}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$	-270 -352 -337 -396 -276 -261 -313		135 133 134 162 149 149 149
ML_n^+ 	$\text{ML}_n = \text{Ni}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Pd}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Pt}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$	+16 +5 -19	14 11 9	149 149 149
ML_n^+ 	$\text{ML}_n = \text{Co}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Co}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$	+33 +33	12 12	150 150
ML_n^+ 	$\text{ML}_n = \text{Pd}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{CMe}$ $\text{Pd}(\text{Ph}_2\text{P}_8\text{CH}_2)_3\text{N}$	-13 -5	20 32	163 163
CoCp 	$\text{R} = \text{SiMe}_3$	+3		157

	$ML_n = Fe(C_6H_5Me)$ $CoCp$	+21 +38 +39 -32 +86 +27 +49 +39 +32	+135	161 26 54 26 54 54 54
	$P_B-CoCp(CH_2=CH_2)$ complex $P,P'-[CoCp(CH_2=CH_2)]_2$ complex $Co(C_5Me_5)$ $RhCp$ $Rh(C_5Me_5)$ $Ir(C_5Me_5)$	-53	-18	34
		+168		89
		+178		89
		+43		78
		+72 +77		6 99

TABLE I
(contd.)

						$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_{\text{B,C}}$	J_{PP}	Lit.		
	M^+	$\text{R}^2 = \text{H}$	$\text{R}^3 = \text{H}$	$\text{R}^4 = \text{H}$	$\text{R}^5 = \text{H}$	$\text{M} = \text{Li}$					
		Ph	H	H	Ph	Li	+77			33	
		H	Me	Me	H	Li	+79			33	
			$P\text{-Cr(CO)}_5$ complex				+59			33	
			$P\text{-W(CO)}_5$ complex				-30			58	
			$P\text{-[W(CO)}_5\text{]}_2$ complex				-55			59, 148	
			$\text{Mn}_3(\text{CO})_{11}$ complex				-100			58, 59	
			$\text{Mn}_3(\text{CO})_{12}^{2+}$ complex				+74			101	
			$\text{Co}_4(\text{CO})_{10}^{2+}$ 2:1-complex				+42			101	
			H	Me	Me	H	Na	+56			57
			H	Me	Me	H	$(\text{Ph}_3\text{P})_2\text{N}$	+57			105
			H	Ph	Ph	H	Li	+58			60
			Ph	Me	Me	H		+54	+21		112
				$P\text{-Mo}_2(\text{CO})_8^{2+}$ 2:1-complex				+207			106
				$P\text{-W}_2(\text{CO})_8^{2+}$ 2:1-complex				+208			106
				$P\text{-Fe}_2(\text{CO})_6^{2+}$ 2:1-complex				+161			106
		Ph	Ph	Ph	Ph	Li					
		Ph	Ph	Ph	Ph	Li	+90			57	
		Ph	Ph	Ph	Ph	Li	+103			32	
							+98			33	
						K	+99			33	
		$\text{R}^2 = \text{H}$	$\text{R}^3 = \text{H}$	$\text{R}^4 = \text{H}$	$\text{R}^5 = \text{H}$	$\text{ML}_n = \text{Mn(CO)}_3$	-26			101	
		Ph	H	H	H	FeCp	-70			106	
		H	Me	H	H	Mn(CO)_3	-29			101	
		H	Me	H	H	$\text{Fe(C}_5\text{Me}_5\text{)}$	-58			132	
		Ph	H	H	Ph	Mn(CO)_3	-30			27	
		H	Me	Me	H	$\text{W(CO)}_3\text{I}$	-32			59	
			$P\text{-W(CO)}_5$ complex				-14			59	
		H	Me	Me	H	Mn(CO)_3	-47			101	
			$P\text{-Fe(CO)}_4$ complex				+52			27	
		H	Me	Me	H	FeCp	-83			102	
			$P\text{-BF}_3$ complex				+23			122	
	$P\text{-Fe(CO)}_4$ complex				+26			102			

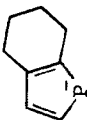
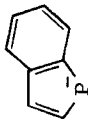
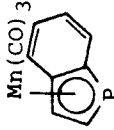
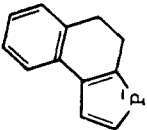
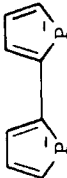


H	Me	Me	H	Fe(1,2-Me ₂ -4-PhC ₃ H ₃)	-75	41
H	Me	Me	H	Fe(C ₅ Me ₅)	-69	132
H	Me	Me	H	Fe(CO) ₃	+200	
<i>P</i> -Fe ₂ (CO) ₆ 2:1-complex						
Et	Me	Me	H	Mn(CO) ₃	-45	57
CH ₂ OH	Me	Me	H	FeCp	-80	27
CH(OH)Me	Me	Me	H	Mn(CO) ₃	-7	75
CHO	Me	Me	H	FeCp	-62	27
COMe	Me	Me	H	Mn(CO) ₃	-16	75
COCH ₂ Bu	Me	Me	H	Mn(CO) ₃	-21	101
CO ₂ Pr	Me	Me	H	Mn(CO) ₃	-20	27
COCH=CHMe	Me	Me	H	Mn(CO) ₃	-21	27
COCH=CHPh	Me	Me	H	Mn(CO) ₃	-21	27
COPh	Me	Me	H	Mn(CO) ₃	-22	101
CO(4-FC ₆ H ₄)	Me	Me	H	Mn(CO) ₃	-26	27
Ph	Me	Me	H	FeCp	-72	100
Ph	Ph	Ph	Ph	Fe(C ₅ Me ₅)	-50	132
R ² = H	R ³ = H	R ⁴ = H	R ⁵ = H		-58 (CDCl ₃)	121
					-59	75
Me	H	H	H		-123 (CF ₃ SO ₃ H)	121
					-54 (CDCl ₃)	121
					-55 (CDCl ₃)	121
					-116 (CF ₃ SO ₃ H)	121
					-119 (CF ₃ SO ₃ H)	121
AgOSO ₂ CF ₃ complex						
					-145	122
					-149	122
					-58 (CDCl ₃)	121
					-59 (CDCl ₃)	121
					-128 (CF ₃ SO ₃ H)	121
					-129 (CF ₃ SO ₃ H)	121
					-72 (CF ₃ CO ₂ H)	121
AgOSO ₂ CF ₃ complex						
					-139	122
					-155	122

TABLE I
(contd.)

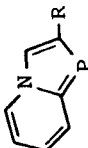
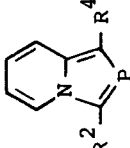
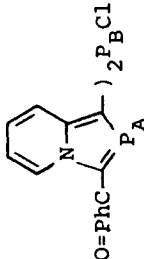
			δ^3P_A	$\delta^3P_{B,C}$	J_{PP}	Lit.
Ph	H	H	-61 (CDCl ₃)			121
			-64			75
		AlCl ₃ complex	-137 (CF ₃ SO ₃ H)			121
		CuOSO ₂ CF ₃ complex	-137			122
		AgOCOCF ₃ complex	-109			122
		AgOSO ₂ CF ₃ complex	-133			122
		Me	-146			122
H	Me	H	-72 (CDCl ₃)			42
			-146 (CF ₃ SO ₃ H)			121
		<i>P</i> _B -BF ₃ complex	-63	+31		122
		<i>P</i> _B -Cr(CO) ₅ complex	-68	+12	5	42
		<i>P</i> ₁ <i>P</i> ₁ -[Cr(CO) ₅] ₂ complex	+14			42
		<i>P</i> _B -Mo(CO) ₅ complex	-67	-23	5	42
		<i>P</i> ₁ <i>P</i> ₁ -[Mo(CO) ₅] ₂ complex	-22			42
		<i>P</i> _B -W(CO) ₅ complex	-67	-48	5	42
		<i>P</i> ₁ <i>P</i> ₁ -[W(CO) ₅] ₂ complex	-46			42
		<i>P</i> _A -Cr(CO) ₅ - <i>P</i> _B -Mo(CO) ₅ complex	+14	-22	5	42
		<i>P</i> _A -Cr(CO) ₅ - <i>P</i> _B -W(CO) ₅ complex	+14	-47	5	42
		<i>P</i> _A -Mo(CO) ₅ - <i>P</i> _B -W(CO) ₅ complex	-21	-47	5	42
		<i>P</i> _B -Mn ₂ (CO) ₁₀ complex	-67	+22		42
		<i>P</i> ₁ <i>P</i> ₁ -[Mn ₂ (CO) ₁₀] ₂ complex	+29			42
		<i>P</i> _B -Fe(CO) ₄ complex	-64	+32		74
		<i>P</i> ₁ <i>P</i> ₁ -[Fe(CO) ₄] ₂ complex	+35			74
		<i>P</i> _B -RuCl ₃ 4:1-complex	-73	+12	0	42
		<i>P</i> ₁ <i>P</i> ₁ -[Ru ₂ (P ^{Ph} ₃) ₂ Cl ₄] 2:1-complex	+18	+56	42	42
		CuOSO ₂ CF ₃ complex	-154			122
		CuI complex	-144			122
		AgOSO ₂ CF ₃ complex	-141 (MeCN)			122
			-161 (DMF)			122
COMe	Me	H	-52			75
			-49			75

TABLE I
(*contd.*)

	$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.
 K^+	+73			116
 Li^+	+40 -36			112 112
 $\text{P-Mn}_2(\text{CO})_8\text{Br}^+$ complex	-55			112
 K^+	+82			116
 $(\text{Li}^+)_2$	+57			107

	-60	104
	-41	104
	+78	52
	+98 +114 +92 +86 +96 +122 +97 +102 +99 +94 +94 +92 +96 +97	52 52 52 52 52 52 91 117 91 1 91 91 91 91 129
	+98 +114 +92 +86 +96 +122 +97 +102 +99 +94 +94 +92 +96 +97	52 52 52 52 52 52 91 117 91 1 91 91 91 91 129
	+98 +114 +92 +86 +96 +122 +97 +102 +99 +94 +94 +92 +96 +97	52 52 52 52 52 52 91 117 91 1 91 91 91 91 129
	+98 +114 +92 +86 +96 +122 +97 +102 +99 +94 +94 +92 +96 +97	52 52 52 52 52 52 91 117 91 1 91 91 91 91 129

TABLE I
(*contd.*)

			$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.	
	Me	Ph	Ph	Ph	+102	91	
	Me	Ph	Ph	Ph	+63	95	
	Me	Ph	4-MeOC ₆ H ₄	Ph	+62	95	
	Me	Ph	Ph	Ph	+73	95	
	Me	Ph	Ph	Ph	+72	95	
	Me	Ph	Ph	Ph	+72	95	
	Me	Ph	4-MeOC ₆ H ₄	Ph	+71	95	
	Me	4-MeOC ₆ H ₄	Ph	Ph	+101	91	
	Ph	Ph	Ph	Ph	+105	91	
	Ph	4-MeOC ₆ H ₄	Ph	Ph	+102	91	
230	Ph	4-ClC ₆ H ₄	Ph	Ph	+106	91	
	R = Me						
	<i>t</i> Bu						
	Ph				+76	94	
					+70	94	
					+69	94	
	R ² = C(=O)Ph	R ⁴ = H				20	
	C(=O)Ph	Me				20	
	C(=O)Ph	Ph				20	
	C(=O)Ph	P _B Cl ₂				20	
	CO ₂ Et	Me		+156	175	20	
	CO ₂ Et	Ph				20	
	CN	Me				20	
	CN	Ph			+161	20	
			+204	+66	48	20	

	R ¹ = H Me P ₈ Cl ₂ H H H H	R ² = H H H Me <i>t</i> Bu Ph NMe ₂ Li Me Ph CO ₂ H CO ₂ SiMe ₃ SiMe ₃ SMe Ph Ph	+77 +73 +73 +70 +66 +72 +77 -5 +109 +69 +76 +127 +126 +121 +109 +73 +74	+160	62 49 62 62 128 62 62 49 49 49 49 50 62 62
	P-Cr(CO) ₃ complex				128
	R ¹ = Me SiMe ₃ SiMe ₃	R ² = NMe ₂ NPhSiMe ₃ OSiMe ₃	+104 +178 +178	+2 -7 -7	147 63 63
	R ¹ = Me Et <i>t</i> Bu Ph 4-MeOC ₆ H ₄ 4-ClC ₆ H ₄ <i>t</i> Bu	R ⁵ = H H H H H H Me	+85 +81 +76 +86 +78 +88 +77		51 51 51 51 51 51 51

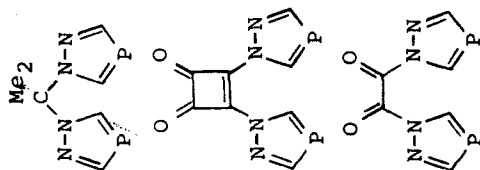
	$\delta^{31}\text{P}_A$	$\delta^{31}\text{P}_{B,C}$	J_{PP}	Lit.
	$R^2 = \text{Ph}$	$R^4 = i\text{Bu}$	$R^5 = \text{Ph}$	
	Ph	Ph	Me	129
	Ph	Ph	Ph	159
	Ph	Ph	$\text{N}(\text{CH}_2)_5$	159
	Ph	Ph	$\text{Ni}[(\text{CH}_2)_2]_2\text{O}$	159
	4-MeC ₆ H ₄	Ph	Ph	159
	4-MeOC ₆ H ₄	Ph	Ph	159
	4-BrC ₆ H ₄	Ph	Ph	159
	4-BrC ₆ H ₄	OSiMe ₃	Ph	159
				95
	$R^2 = \text{H}$			61
	Me			61
	Ph			61
	$R = \text{H}$			146
	<i>i</i> Bu			146
	Ph			146
	$R^1 = \text{H}$	$R^3 = \text{H}$	$R^3 = \text{H}$	177, 141, 146
				146
	Me	$N(2)\text{-BF}_3$ complex		146
	$\text{CH}(\text{OMe})(\text{NMe}_2)$			146
	$\text{C}_6\text{O}_2\text{Cl}$			146
	Ph			146
		$N(2)\text{-BF}_3$ complex		146
		$N(2)\text{-AlCl}_3$ complex		92, 141
	4-NO ₂ C ₆ H ₄			146
				146
	$\text{C}(\text{Me})=\text{NH}$			146
	2-C ₆ H ₄ N			146
	COMe			146

COPh	H	H	+102 (toluene)	146
C(O)COCl	H	H	+104 (Me ₂ CO)	146
CO(4-MeOC ₆ H ₄)	H	H	+119	146
CO(4-NO ₂ C ₆ H ₄)	H	H	+101	146
CONHMe	H	H	+108	146
	H	H	+104 (CH ₂ Cl ₂)	146
			+104 (CHCl ₃)	146
SiMe ₃	H	H	+92	146
P _B (S)Ph ₂	H	H	+98	146
SO ₄ (4-MeC ₆ H ₄)	H	H	+104	146
H	Me	H	+84	120
H	<i>i</i> Pr	H	+77	126
H	<i>t</i> Bu	H	+77	124, 127
H	CH ₂ <i>t</i> Bu	H	+87	126
H	CM ₂ Et	H	+81	118
H	1-MeC ₅ H ₈	H	+78	126
H	CM ₂ <i>t</i> Bu	H	+88	118
H	1-MeC ₆ H ₁₀	H	+80	126
H	Ph	H	+81	90
H	CO ₂ <i>t</i> Bu	H	+99	119
Me	<i>t</i> Bu	H	+83	129
Me	Ph	H	+87	92
Et	Ph	H	+85	92
Ph	<i>t</i> Bu	H	+91	129
Ph	Ph	H	+93	92
Me	H	Me	+94	69
Me	H	BMe ₂	+104	146
Me	H	SnMe ₃	+131	69
Me	H	SnMe ₂ (CMe=CEt)BEt ₂	+132	69
Ph	H	D	+96	69
SiMe ₃	H	<i>t</i> Bu	+86	127
H	Me	Me	+79	146
H	Me	<i>t</i> Bu	+75	127
H	<i>t</i> Bu	<i>t</i> Bu	+65	146
H	<i>t</i> Bu	1-MeC ₅ H ₈	+69	127
H	<i>t</i> Bu	1-Ad	+67	118
H	<i>t</i> Bu	(CH ₂) ₃ CO ₂ Me	+74	4
H	<i>t</i> Bu	Ph	+74	127
H	<i>t</i> Bu	COPh	+106	127
H	<i>t</i> Bu	CONHPh	+87	127
H	<i>t</i> Bu	CO ₂ Me	+96	156
			+98	127

+7

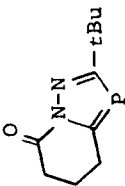
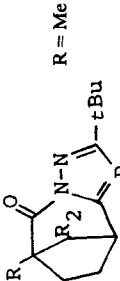
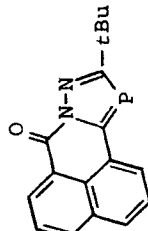
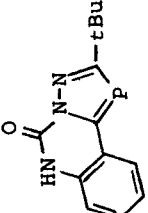
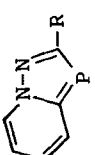
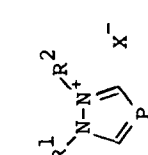
TABLE I
(*contd.*)

		$\delta^3\text{P}_A$	$\delta^3\text{P}_{B,C}$	J_{PP}	Lit.
H	<i>i</i> Bu	+97			127
H	<i>i</i> Bu	+111			127
H	<i>i</i> Bu	+90	+23	54	127
H	C_6H_{11}	+66			146
H	1-Ad	+104			4
H	1-Ad	+95			4
H	Ph	+74			141
		+76			90
H	Ph	+105			90
H	Ph	+99			90
H	Ph	+113	+23	54	90
H	Ph	+111	+27	56	90
H	Ph	+111	+27	65	90
H	COPh	+122			115
H	CONMe ₂	+113			115
H	CO ₂ Et	+118			115
H	CO ₂ Me	+134			90, 155
H	SiMe ₃	+151			25
H	SiMe ₃	+147			25
H	SiMe ₃	+84	+56	61	127
Me	<i>i</i> Bu	+83			141
Me	<i>i</i> Bu	+88			127
Me	<i>i</i> Bu	+76			146
Me	C_6H_{11}	+87			92
Me	Ph	+87			130
Me	Ph	+89			90, 141
Me	4-MeOC ₆ H ₄	+85			146
Et	Ph	+88			92
Ph	D	+96			69
Ph	Me	+96			92
Ph	Me	+95			92
		+92			130
Ph	<i>i</i> Bu	+88			129
Ph	<i>i</i> Bu	+83			141
Ph	<i>i</i> Bu	+97			124



Substrate	Temperature (°C)	Yield (%)	Product	Temperature (°C)	Yield (%)
Ph	+78	146	C ₆ H ₁₁		
Ph	+90	92	Me		
Ph	+80	92	<i>i</i> Pr		
Ph	+87	92, 130	<i>t</i> Bu		
Ph	+95	92, 141	Ph		
Ph	+90	146	4-MeOC ₆ H ₄		
COMe	+117	120	<i>t</i> Bu		
COMe	+109	127	Me		
COMe	+117	127	Ph		
COMe	+110	127	CONHPh		
COMe	+149	127	SO ₂ Ph		
COMe	+110	120	<i>t</i> Bu		
COMe	+110	146	Ph		
COPh	+106	127	Ph		
COPh	+95	127	<i>t</i> Bu		
SiMe ₃	+178	25	SiMe ₃		
SiMe ₃	+187	83	P _B (S)(NiPr ₂) ₂		
P _B Cl ₂	+100	146	<i>t</i> Bu		
P _B (O)Ph ₂	+107	127	Ph		
P _B (O)(OMe) ₂	+99	127	Me		
P _B (O)(OMe) ₂	+103	127	Ph		
	+97	146			
	+114	146			
	+117 (CHCl ₃)	146			
	+115 (C ₆ H ₆)	146			

TABLE I
(*contd.*)

	$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.
	+96			127
	+91			127
	+98			127
	+84			127
	+63 +57 +55 +58			94 94 94 94
	+95 +87 +86 +102 +92 +92 +79 +93	$\text{R}^2 = \text{H}$ Me Me H H Me Ph H	$\text{X} = \text{Cl}$ Cl BF ₄ BF ₄ HSO ₄ MeSO ₄ Cl Cl	146 146 146 146 146 146 146 146

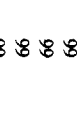
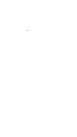
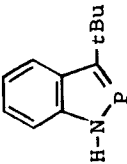
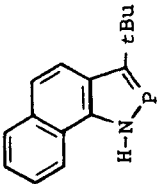
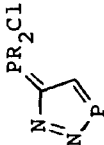
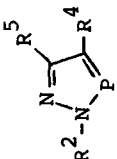
	R ⁵ = H <i>t</i> Bu	+84 +69 (CHCl ₃) +67 (<i>n</i> BuOH) +69 (Toluol) +68 (C ₆ D ₆) +67 +77 +72 +69 +79 +71 +70 (CH ₂ Cl ₂) +72 (CHCl ₃) +83 +87 +66 +127 +77 +74 +82	146 146 146 146 130 126 130 126 130 126 146 146 118 118 156 155 124, 130 4 130
	Mes 2,4,6- <i>i</i> Pr ₃ C ₆ H ₂ <i>t</i> Bu SiMe ₃ <i>t</i> Bu 1-Ad <i>t</i> Bu	+78	146
	R ⁵ = Ph <i>t</i> Bu	+178 +183	129 129
	R ⁵ = Pr Bu CH ₂ NMe ₂ Ph CO ₂ Et Me H H H	+219 +218 +220 +218 +221 +227 +227 +226 +217	66 66 66 66 66 66 66 66 66

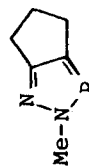
TABLE I
(*contd.*)

		$\delta^3\text{P}_A$	$\delta^3\text{P}_{B,C}$	J_{PP}	Lit.
	Me	CO ₂ Et	+226		66
	Me	OCOPh	+205		66
	Me	H	+230		66
	Me	CO ₂ Me	+230		66
	Me	Ph	+231		66
	Me	CO ₂ Et	+225		66
	Me	CO ₂ Me	+227		66
	Me	OCOPh	+227		66
	SiMe ₃	CO ₂ Me	+238		66
			+210		128
			+180		128
	R = <i>i</i> Pr				25
	R ² = H	R ⁴ = H	R ⁵ = Me		108
	H	H	CO ₂ <i>i</i> Bu		119
	Bu	Et	H		44
	SiMe ₃	CO ₂ Et	H		140
	P _B Ph ₂	CO ₂ Et	H		140
	Me	H	Me	+64	152, 154
				72	

[illegible]

TABLE I
(*contd.*)

		$\delta^{31}\text{P}_A$	$\delta^{31}\text{P}_{B,C}$	J_{PP}	Lit.
Me	$\text{P}_B(\text{S})(\text{OMe})_2$	+254	+82	87	154
Me	$\text{P}_A\text{-Cr}(\text{CO})_5$ complex	+266	+80	81	154
Me	$\text{P}_B(\text{S})\text{F}_2$	+275	+93	112	154
Me	$\text{P}_B(\text{S})\text{FCl}$	+269	+90	105	154
Me	$\text{P}_B(\text{S})\text{Cl}_2$	+251	+56	95	154
Me	$\text{P}_B(\text{S})\text{Br}_2$	+245	-11	85	154
Me	$\text{P}_B(\text{OMe})_2\text{O}_2\text{C}_2\text{Me}_2$	+252	-36	75	154
Me	$\text{P}_B(\text{OMe})_2\text{O}_2\text{C}_3\text{Ph}_2$	+255	-36	72	154
Me	SMe	+237			123
		+222			56
Me	SPh	+236			56
Me	Cl	+212			123
Me	Br	+217			56, 123
Me	Me	+212			53
Me	CH_2iPr	+229			11
<i>t</i> Bu	<i>t</i> Bu	+219			130
Ph	CH_2iBu	+220			11
Ph	$1\text{-C}_3\text{H}_9$	+228			10
Ph	$1\text{-C}_6\text{H}_{11}$	+231			10
Ph	$1\text{-(2-HO)C}_8\text{H}_{10}$	+223			10
Ph	$\text{P}_B(\text{O})\text{Ph}_2$	+246	+21	74	18
Ph	Cl	+206			123
Ph	Br	+211			123
		+210			56
COMe	CONHCCl_3	+249			9
COMe	CONHPh	+246			12
COMe	$\text{CONH(2-C}_{10}\text{H}_7)$	+242			12
SiMe_3	Ph	+247			145
SiMe_3	CO_2Me	+262			140
P_BPh_2	CO_2Me	+267	+66	68	140
		+204			152



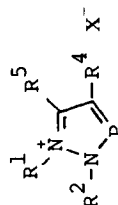
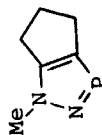
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TABLE I
(*contd.*)

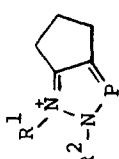
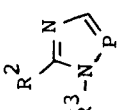
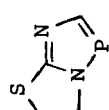
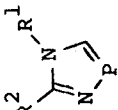
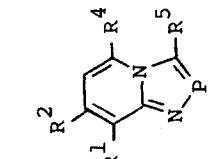
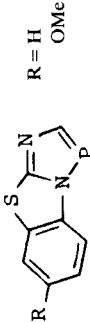
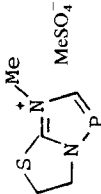
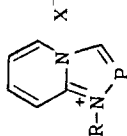
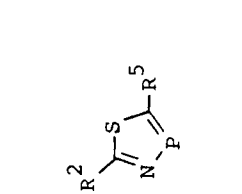
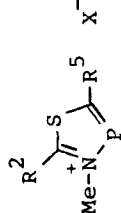
	$\delta^{31}\text{P}_A$	$\delta^{31}\text{P}_{B,C}$	J_{PP}	Lit.					
	H Me Me	Ph Me Me	$\text{P}_B(\text{O})\text{Ph}_2$ Me Me	Me NEt_2 NEt_2	Cl MeBCl_3 MeBBF_3	+228 +231 +232	+33	45	18 114 114
	$\text{R}^1 = \text{Me}$ H	$\text{R}^2 = \text{H}$ Me				+195 +209			152 47
	$\text{R}^2 = \text{Me}$ Ph Ph SMe	$\text{R}^3 = \text{H}$ H Ph Me				+209 +185 +212 +213			68 68 144 68
						+194			68
	$\text{R}^1 = \text{H}$ Me Ph	$\text{R}^2 = \text{Me}$ SMe Ph				+199 +183 +182			68 68 144
	$\text{R}^1 = \text{H}$ Me H H H H H H H Me H H H H Me H	$\text{R}^2 = \text{H}$ H Me H H H H H H Me H H H H Me H	$\text{R}^4 = \text{H}$ H H Me H H H H H H H H H H H H	$\text{R}^5 = \text{H}$ H H H P_BPh_2 P_BCl_2 P_BPh_2 CO_2Et CO_2Et CO_2Et CO_2Et		+195 +195 +194 +195 +227 +244 +228 +233 +233 +230 +234	-38 +123 -38	2 166 5	68 144 144 144 144 68 144 144 144 144 144

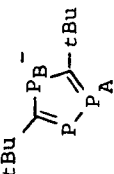
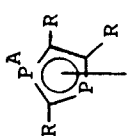
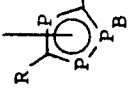
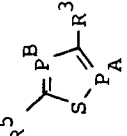
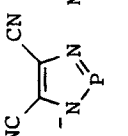
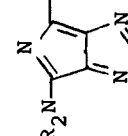


TABLE I
(*contd.*)

	$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.
 R = H OMe	+184 +184			144 144
 Me MeSO ₄ ⁻	+178			68
 R = Me 2-C ₅ H ₄ N	+187 +178	X = MeSO ₄ Cl		68 144
 R ² = Me Ph Ph Ph Ph Ph CuBr complex N(3)-BF ₃ complex Ph 4-CNC ₆ H ₄	+302 +276 +250 +257 +270 +300 +293 +295 +249 +262	R ⁵ = CO ₂ Et H Ph 4-MeC ₆ H ₄ 4-NO ₂ C ₆ H ₄ CO ₂ Et CuBr complex N(3)-BF ₃ complex Ph 4-CNC ₆ H ₄		144 68 143 143 143 143 144 144 143 143
 R ² = Ph NMe ₂ NMe ₂ X ⁻	+290 +239 +240	X = BF ₄ BF ₄ MeSO ₄		144 144 144

[illegible]

TABLE I
(*contd.*)

		$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.
	$\text{Li}(\text{DME})_3^+$	+239	+252	51	24
					
	$\text{R} = t\text{Bu}$	+33	+46		161
	$\text{R}^3 = \text{OSiMe}_3$ SSiMe_3	+180 +310	+160 +304	54 59	8 5
	$\text{M} = \text{H}_2\text{NMe}_2$ H_2NEt_2 K	+262 +262 +264			65 65 145
	$\text{NR}_2 = \text{NMe}_2$ $\text{NMe}(\text{CH}_2\text{Ph})$	+300 +301 +300 +300 +301 +299 +301 +300			65 67 67 67 67 67 67 67

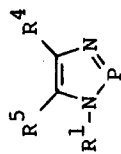
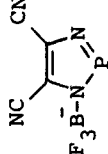
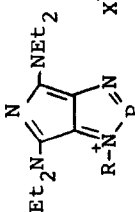
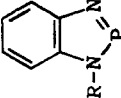
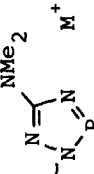
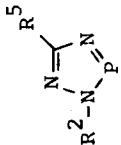
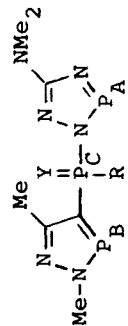
	R ¹ = Me H Me CH ₂ Ph CH ₂ COPh CH ₂ CO ₂ Et SiMe ₃	R ⁴ = NMe ₂ CN CN CN CN CN CN	R ⁵ = H CN CN CN CN CN CN	+210 +255 +234 +230 +219 +234 +250	144 65 139 144 144 144 66
	H ₂ NMe ₂ ⁺			+232	144
	R = Me Me	X = MeSO ₄ I		+278 +280	67 67
	R = H Et Pr <i>i</i> Pr <i>s</i> Bu (CH ₂) ₂ CHMe ₂ CH ₂ Ph Ph	N(3)-BF ₃ complex N(3)-BF ₃ complex N(3)-BF ₃ complex N(3)-BF ₃ complex N(3)-BF ₃ complex N(3)-BF ₃ complex N(3)-BF ₃ complex		+238 +222 +232 +236 +226 +228 +226 +226 +227 +232 +226 +228 +225 +206	98 98 98 98 98 98 98 98 98 98 98 98 98 98
	M = Na K			+291 +263	145 145

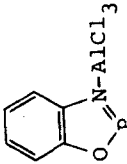
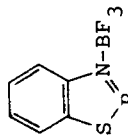
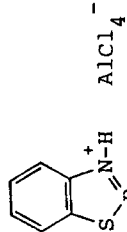
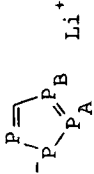
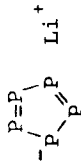
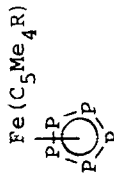
TABLE I
(contd.)

	$\delta^3\text{P}_\text{A}$	$\delta^3\text{P}_\text{B,C}$	J_PP	Lit.
$\text{R}^3 = \text{H}$	$\text{R}^5 = \text{Ph}$			
H	NMe ₂	+252		79
H	SMc	+246		138
Me	Me	+250		145
Me	<i>i</i> Pr	+254		137
Me	Bu	+255		30
Me	CH ₂ Ph	+256		30
Me	Ph	+250		30
Me	BF ₃ complex	+256		96
	<i>P</i> -Cr(CO) ₅ complex	+251		30
	<i>P</i> -Mo(CO) ₅ complex	+281		153
	<i>P</i> -cis-PtBr ₂ P _B Et ₃ complex	+258		153
	<i>N</i> (4)-trans-PdCl ₂ P _B Et ₃ complex	+191	16	73
	<i>N</i> (4)-trans-PtBr ₂ P _B Et ₃ complex	+245	+37	73
	<i>P</i> -AuMe ₂ Cl complex	+242	+1	36
Ph	CH ₂ Ph	+223		30
Ph	Ph	+245		30
SiMe ₃	Ph	+280		79
SiMe ₃	NMe ₂	+275		138
SiMe ₃	SMc	+281		145
SnMe ₃	NMe ₂	+279		138
P _B Me ₂	NMe ₂	+272	89	138
P _B Ph ₂	NMe ₂	+277	69	138
P _B (S)Me ₂	NMe ₂	+270	23	138
P _B (S)Ph ₂	NMe ₂	+275	18	138
R = Me	Y = —	+270	+244(B)	92(AC)
Ph	—	+270	+25(C)	<1(BC)
Me	S	+268	+243(B)	81(AC)
			+30(C)	<1(BC)
			+250(B)	24(AC)
			+51(C)	74(BC)



	$R^1 = H$	$R^5 = Ph$	+254	79
	Me	Me	+257	137
		<i>P</i> -Cr(CO) ₅ complex	+315	153
		<i>P</i> -Mo(CO) ₅ complex	+267	153
		<i>N</i> (4)- <i>trans</i> -PtCl ₂ P ₈ Et ₃ complex	+246	72
		<i>P</i> -cis-PtBr ₂ P ₈ Et ₃ complex	+211	73
		<i>N</i> (4)- <i>trans</i> -PtBr ₂ P ₈ Et ₃ complex	+245	73
		<i>P</i> -Pt(P ₈ Ph ₃) ₃ complex	+234	71
		<i>P</i> -Pt(P ₈ Ph ₃) ₂ 2:1-complex	+231	71
	Me	Bu	+260	30
	Me	CH ₂ Ph	+255	30
	Me	Ph	+261	30
	Ph	CH ₂ Ph	+257	30
			+249	79
			+228	21
		R = Me		
			+323	48
			+244	480
			+270	19
			+234	14

TABLE I
(*contd.*)

	$\delta^{31}\text{P}_A$	$\delta^{31}\text{P}_{B,C}$	J_{PP}	Lit.
	+262			97
	+160 +204			97 144
	+317			144
	+355	+362	-484 -4 -505 -53	(AB) (AB') (AA') (BB')
	+470			23
	+153			136

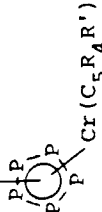
	R = H	R' = H																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																										</
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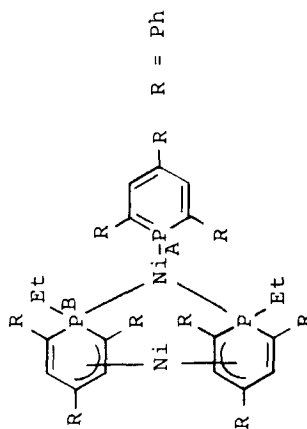
TABLE I
(contd.)

		$\delta^3\text{P}_A$	$\delta^3\text{P}_{B,C}$	J_{PP}	Lit.
	$P\text{-W}(\text{CO})_5$ complex	+161			28
	$P,N\text{-W}(\text{CO})_4$ complex	+205			28
SiMe_3	H	+215			155
<i>t</i> Bu	<i>t</i> Bu	+179			29
<i>t</i> Bu	Ph	+187			77
Ph	H				
	$P\text{-AgOCoCF}_3$ complex	+133			64
Ph	Me				
	H				
	$P\text{-AgOCoCF}_3$ complex	+138			64
Ph	CH_2Ph				
Ph	H	+178			160
Ph	$\text{CH}_2/2$	+191			160
Ph	CH_2CN	+179			77
	H	+176			77
	$P\text{-Ni(COD) 2:1-complex}$	+176			77
	$P\text{-Ni(CDT) complex}$	+178			81
Ph	H	+198			39
	$P\text{-Cr(CO)}_5$ complex	+217			39
	$P\text{-Cr(CO)}_4$ 2:1-complex	+180			39
	$P\text{-Mo(CO)}_5$ complex	+191			39
	$P\text{-Mo(CO)}_4$ 2:1-complex	+156			39
	$P\text{-W(CO)}_5$ complex	+170			39
	$P\text{-W(CO)}_4$ 2:1-complex	+243			110
	$P\text{-Mn(Cp(CO))}_2$ complex	+246			110
	$P\text{-Mn(C}_3\text{H}_4\text{Me)(CO)}_2$ complex	+179			77
	$P\text{-Ni(CDT) complex}$	+205			77
	$P\text{-Ni(CH}_2=\text{CH}_2)_2\text{P}_2(\text{C}_6\text{H}_{11})_3$ complex	+167			77
	$P\text{-Ni(P}_2\text{Ph}_3)_2$ complex	+160			77
	$P\text{-Ni[P}_2\text{(O-2-MeC}_6\text{H}_4)_3]_2$ complex	+179			77
	$P\text{-Ni(COD) 2:1-complex}$	+141			77
	$P\text{-Ni(COT)}_{1/2}$ 2:1-complex	+167			64
	$P\text{-AgOCoCF}_3$ complex	+166			35
	$P\text{-AuCl complex}$	+215			35
	$P\text{-AuI complex}$	+216			160
Ph	H				160
Ph	CO_2Me				93
CO_2Me	Ph				117
<i>t</i> Bu	<i>t</i> Bu				

125
117, 164

Ph +202
Ph +198

Ph
Ph

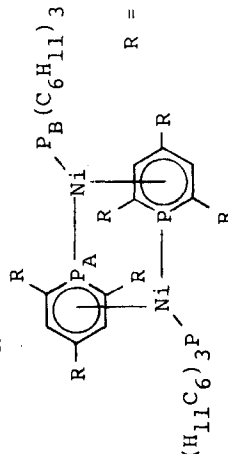


R = Ph

+186

5

76



R = Ph

+108

+29

77

253

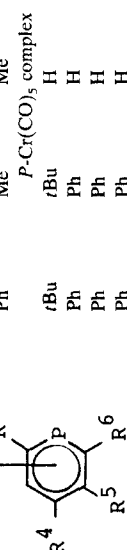
ML_n = Mn(C₅H₄Me)

R² = Ph R⁴ = Me R⁵ = Me R⁶ = H
P-Mn(C₅H₄Me)(CO)₂ complex

Ph Me Me H
P-Cr(CO)₅ complex

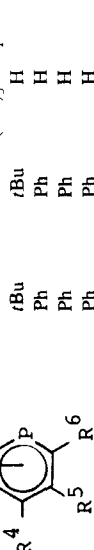
+74

110



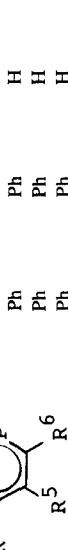
+96

111



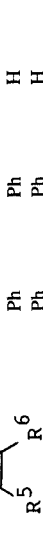
+23

43



+4

37, 43



-3

38



-50

110

FeCp

X = AlCl₄
PF₆⁻

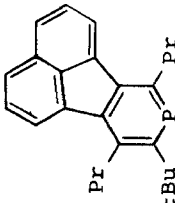
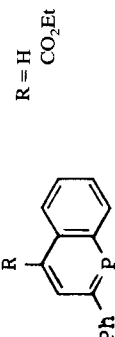
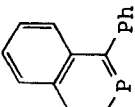
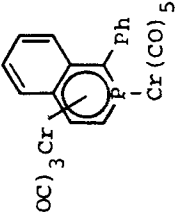
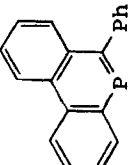
-8

-10

113

113

TABLE I
(*contd.*)

	$\delta^{31}\text{P}_\text{A}$	$\delta^{31}\text{P}_\text{B,C}$	J_PP	Lit.
	+206			117
	+197 +221			82 84
	+188			111
	+103			111
	+186			109

	$R^2 = CF_3$	$R^4 = tBu$	$R \equiv Me$	+163	7
	CF_3	1-Ad	Me	+163	7
	Ph	Ph	Ph	+163	93
	Ph	Ph	4-MeC ₆ H ₄	+161	93
	4-MeC ₆ H ₄	Ph	Ph	+161	93
	4-MeC ₆ H ₄	Ph	4-MeC ₆ H ₄	+160	93
	4-ClC ₆ H ₄	Ph	Ph	+163	93
	4-ClC ₆ H ₄	Ph	4-MeC ₆ H ₄	+161	93
				+245	83
				+287	70
		$R = CF_3$			
				+25	22
				+296	46
			+59 (B)		49 (AB)
			+58 (C)		45 (AC)
					30 (BC)
				+263	158
				+302	21
				-316	134

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