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New electron-deficient chiral phosphines: (4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl] substituted amines

Denés Konya, Christian Philouze, Yves Gimbert* and Andrew E. Greene

LEDSS, UMR CNRS 5616, Université Joseph Fourier, BP 53, 38041 Grenoble CEDEX 9, France
Correspondence e-mail: yves.gimbert@ujf-grenoble.fr

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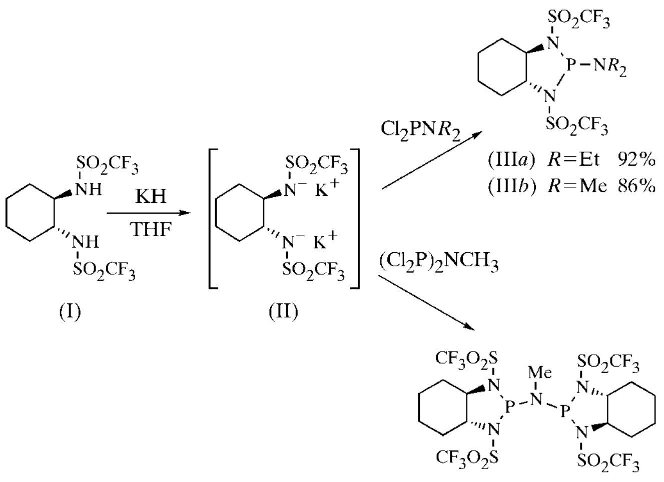
Three chiral electron-deficient phosphine ligands, [(4*R*,-15*R*)-,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl]diethylamine, $C_{12}H_{20}F_6N_3O_4PS_2$, (III*a*), [(4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl]dimethylamine, $C_{10}H_{16}F_6N_3O_4PS_2$, (III*b*), and bis[(4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl]methylamine, (IV), as the chloroform solvate, $C_{17}H_{23}F_{12}N_5O_8P_2S_4\cdot0.98CHCl_3$, have been prepared from (1*R*,2*R*)-*N*,*N'*-bis(trifluoromethanesulfonyl)-1,2-cyclohexanediamine and diethyl phosphoramidous dichloride, dimethyl phosphoramidous dichloride or methyl imidodiphosphorus tetrachloride. The π-acceptor abilities of these new types of ligands have been evaluated by X-ray determination of the P—N bond lengths; it has been found that the most promising ligand is the bis(phosphine) (IV).

Comment

Phosphorus(III) ligands of the type PR_3, where *R* is, for example, an alkyl, alkoxy or aryl group, are ubiquitous in homogeneous catalysis (Collman *et al.*, 1987; Levason, 1990; McCauliffe, 1987; Pignolet, 1983). While certain *R* groups can impart significant π-acceptor character to these ligands, the number of known ligands that approach the π-acceptor ability of CO is rather small (Brookhart *et al.*, 1992; Moloy & Petersen, 1995; Phillips *et al.*, 1988; Schnabel & Roddick, 1993). This may change rapidly, however, because recent publications (Gimbert *et al.*, 1999; Jolly *et al.*, 1990; Kundig *et al.*, 1994; van Leeuwen & Roobeck, 1983, 1981) have shown that certain transition-metal-catalyzed transformations are considerably more successful when less electron-rich phosphorus ligands are used. In contrast with the large number of non-electron-deficient phosphines, phosphinites (Jacobsen *et al.*, 1999), phosphites (Hilgraf & Pfaltz, 1999), diazaphospholes (Brunel *et al.*, 1998) and, to a lesser degree, phosphoramidites (Feringa, 2000) that have frequently been employed as ligands in asymmetric catalysis, electron-deficient mono- and bidentate tris(amino)phosphine derivatives have rarely been used (see, however, Hersh *et al.*, 1996; Denmark *et al.*, 1994) in asymmetric procedures.



(IV) 82%

The trifluoromethanesulfonylamino group is strongly electron withdrawing and should, therefore, substantially affect phosphorus σ-donation and π-acidity. With this in mind, two new types of chiral phosphorus ligands that could be expected to approach the π-acceptor ability of CO have been prepared from (1*R*,2*R*)-*N*,*N'*-bis(trifluoromethanesulfonyl)-1,2-cyclohexanediamine, (I) (Denmark *et al.*, 1998; Takahashi *et al.*, 1992), namely [(4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)-perhydro-1,3,2-benzodiazaphosphol-2-yl)diethylamine, (III*a*), and [(4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl)dimethylamine, (III*b*), and bis[(4*R*,5*R*)-1,3-bis(trifluoromethanesulfonyl)perhydro-1,3,2-benzodiazaphosphol-2-yl]methylamine, (IV).

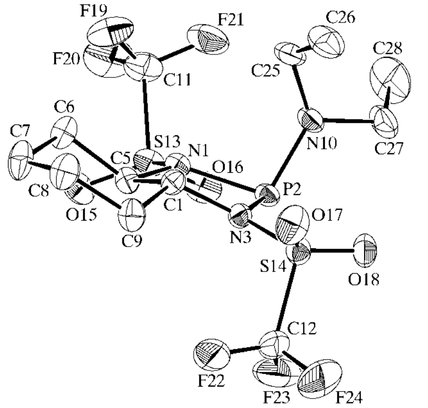


Figure 1
A view of the molecule of compound (III*a*) with displacement ellipsoids shown at the 25% probability level. H atoms have been omitted for clarity.

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X-ray diffraction analyses of these new compounds (Figs. 1, 2 and 3) indicate some interesting differences. In particular, in the potential monodentates [(III*a*) and (III*b*)], the trifluoromethyl groups are *trans* and perpendicular to the mean plane defined by the five-membered ring, whereas in the potential bidentate (IV), the trifluoromethyl groups are *cis*. On considering the distances of the S atoms from the least-squares weighted plane through each ring (Table 4), it should be noted that pairs having the same sign are on the same side with respect to the plane. The first set of torsion angles in Table 4 indicates the orientation of the trifluoromethyl groups with respect to the P—N$_{amine}$ bond (not the plane through the ring). Finally, the relative orientations of the trifluoromethyl groups are revealed by the non-bonded torsion angles in Table 4.

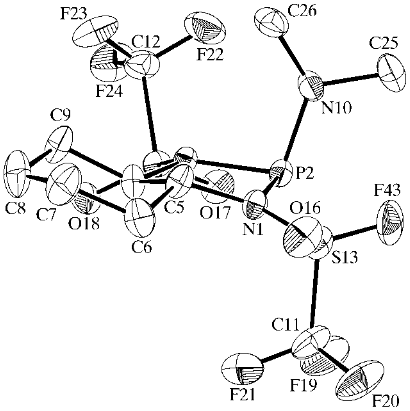

Figure 2
A view of the molecule of compound (III*b*) with displacement ellipsoids shown at the 25% probability level. H atoms have been omitted for clarity.

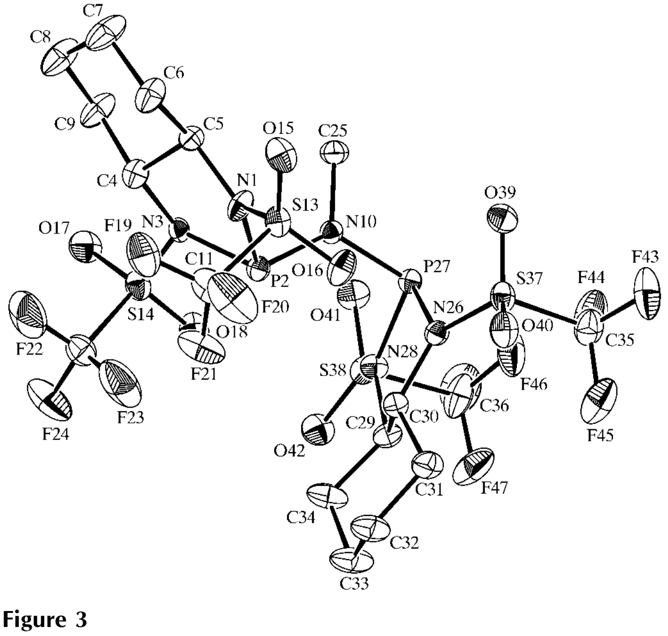

Figure 3
A view of the molecule of compound (IV) with displacement ellipsoids shown at the 25% probability level. H atoms and the disordered solvent molecule have been omitted for clarity.

Despite the strong *sp*^{2} character of the N atoms of the five-membered rings, the bonds about these atoms are not strictly coplanar, as indicated by the distances of the N atoms from the planes defined by the atoms surrounding them (Table 4).

In (III*a*), (III*b*) and (IV), the internal N—P—N angle is approximately 86°. The N—P bond distances for all three compounds are compared in Table 4, and it can be seen that significant differences are found between the distances involving N$_{ring}$ and those involving N$_{amine}$. The averages for P—N$_{ring}$ are 1.784 (5) Å for (III*a*) and (III*b*), and 1.755 (3) Å for (IV), while the averages for P—N$_{amine}$ are 1.639 (3) Å for (III*a*) and (III*b*), and 1.690 (9) Å for (IV). [For a discussion of the nature of the P—N bond, see Kremer *et al.* (1996).] The observed difference in the P—N$_{amine}$ distances suggest that there is a reduced electron density at P in the bis(phosphine), and thus greater π-accepting ability. This apparent electronic difference may contribute to its ability to give rise to metal complexes. For example, (IV), but not (III*a*) or (III*b*), has been shown to react with the hexacarbonylphenylacetylene dicobalt complex to produce, by thermal substitution of two additional carbonyl groups, the bis(phosphine) dicobalt tetracarbonylphenylacetylene complex (D. Konya, unpublished results).

Experimental

The bis(amide) (I) was prepared according to the literature method of Denmark *et al.* (1994) from commercially available (1*R*,2*R*)-1,2-cyclohexanediamine and triflic anhydride. In the presence of KH in tetrahydrofuran (THF), the bis(amide) yields the dipotassium salt, (II), which, on treatment with diethyl phosphoramidous dichloride (Whitaker *et al.*, 1995), provides phosphine (III*a*) (92% yield; m.p. 336–338 K). Phosphine (III*b*) was obtained by an analogous procedure (86% yield; m.p. 339–341 K). Bis(phosphine) ligand (IV) was prepared from (II) by reaction with methyl imidodiphosphorus tetrachloride (King & Gimeno, 1978) (82% yield; m.p. 473–475 K). Phosphines (III*a*), (III*b*) and (IV) are stable to hydrolysis and oxidation and can be stored for several months at room temperature without decomposition.

Spectroscopic data for (III*a*): $[\alpha]_D^{25} = -35$ (*c* 0.8, THF); ^{1}H NMR ($CDCl_3$, 200 MHz, δ, p.p.m.): 1.15 (*t*, $J = 7.2$ Hz, 6H), 1.20–1.65 (*m*, 4H), 1.80–1.95 (*m*, 2H), 2.55–2.70 (*m*, 2H), 2.90–3.40 (*m*, 5H), 3.59 (*td*, $J = 3.1$ and 10.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 50 MHz, δ, p.p.m.): 13.8, 13.9, 23.9, 24.4, 29.6, 30.6, 41.4, 41.7, 65.7 (*d*, $J = 5.5$ Hz), 67.3 (*d*, $J = 5.5$ Hz); ^{19}F NMR ($CDCl_3$, 188.3 MHz, δ, p.p.m.): −76.1 (*br s*, 3F), −77.6 (*d*, $J = 11.4$ Hz, 3F); ^{31}P NMR ($CDCl_3$, 81 MHz, δ, p.p.m.): 99.3 (*s* large, 2P); IR (cm^{-1}): 1458, 1448, 1392, 1219, 1196; MS (CI) *m*/*z*: 480 [100%, $(M+H)^+$], 407 (3.7%), 396 (10.8%), 346 (2.5%). Analysis calculated for $C_{12}H_{20}F_6N_3O_4PS_2$: C 30.06, N 8,77, H 4.20%; found: C 29.87, N 9.01, H 3.92%.

Spectroscopic data for (III*b*): $[\alpha]_D^{25} = -42$ (*c* 0.9, THF); ^{1}H NMR ($CDCl_3$, 200 MHz, δ, p.p.m.): 1.15–1.65 (*m*, 4H), 1.75–1.95 (*m*, 2H), 2.50–2.70 (*m*, 2H), 2.73 (*s*, 3H), 2.78 (*s*, 3H), 3.57 (*td*, $J = 3.5$ and 10.6 Hz, 1H), 3.66 (*td*, $J = 3.0$ and 10.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 50 MHz, δ, p.p.m.): 23.4, 23.9, 29.3, 29.9, 37.4, 37.9, 65.4 (*d*, $J = 4.5$ Hz), 66.9 (*d*, $J = 4.5$ Hz); ^{19}F NMR ($CDCl_3$, 188.3 MHz, δ, p.p.m.): −76.5

(d, J = 11.5 Hz, 3F), −77.60 (d, J = 9.8 Hz, 3F); ^{31}P NMR ($CDCl_3$, 81 MHz, δ, p.p.m.): 102.90 (s, 2P); IR (film, ν, cm^{-1}): 3293, 2952, 1462, 1451, 1392; MS (Cl) m/z: 452 [100%, $(M+H)^+$], 318 (6.9%). Analysis calculated for $C_{10}H_{16}F_6N_3O_4PS_2$: C 26.61, N 9.31, H 3.57%; found: C 26.42, N 9.47, H 3.84%.

Spectroscopic data for (IV): $[\alpha]_D^{25}$ = −34 (c 1.0, THF); ^{1}H NMR ($CDCl_3$, 200 MHz, δ, p.p.m.): 1.22 (t, J = 10.4 Hz, 4H), 1.4–1.7 (m, 4H), 1.84 (br s, 4H), 2.5–2.7 (m, 4H), 3.01 (t, J = 9.1 Hz, 3H), 3.34 (t, J = 10.4 Hz, 2H), 3.98 (s, 2H); ^{13}C NMR ($CDCl_3$, 50 MHz, δ, p.p.m.): 23.8, 24.4, 28.9, 30.0, 34.3, 67.5, 68.8; ^{19}F NMR ($CDCl_3$, 188.3 MHz, δ, p.p.m.): −73.58 (br s, 6F), −76.82 (s, 6F); ^{31}P NMR ($CDCl_3$, 81 MHz, δ, p.p.m.): 103.5 (s, 2P); IR (cm^{-1}): 3054, 1265; MS (Cl) m/z: 861 [100%, $(M+NH_4^+)$], 844 (24.4%), 774 (9.3%), 436 (14.2%), 397 (93.6%). Analysis calculated for $C_{17}H_{23}F_{12}N_5O_8P_2S_4$: C 24.35, N 8.10, H 2.46%; found: C 24.19, N 8.30, H 2.72%.

Table 1
Selected geometric parameters (Å, °) for (IIIa).

P2—N1	1.783 (2)	P2—N10	1.637 (2)
P2—N3	1.792 (2)		
N1—P2—N3	85.47 (7)	N3—P2—N10	107.71 (8)
N1—P2—N10	101.42 (8)		

Table 2
Selected geometric parameters (Å, °) for (IIIb).

P2—N1	1.791 (2)	P2—N10	1.642 (2)
P2—N3	1.771 (2)		
N1—P2—N3	86.05 (8)	N3—P2—N10	100.80 (8)
N1—P2—N10	107.3 (1)		

Compound (III*a*)

Crystal data

$C_{12}H_{20}F_6N_3O_4PS_2$
M_r = 479.39
Triclinic, $P\overline{1}$
a = 8.657 (2) Å
b = 10.352 (2) Å
c = 12.859 (3) Å
α = 104.81 (1)°
β = 93.08 (2)°
γ = 111.21 (2)°
V = 1024.9 (5) Å^3
Z = 2
D_x = 1.553 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.0–11.1°
μ = 0.41 mm^{-1}
T = 293 K
Prism, colourless
0.20 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
6193 measured reflections
5947 independent reflections
3678 reflections with $I > 3\sigma(I)$
$R_{\rm int}$ = 0.025
$\theta_{\rm max}$ = 30°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = 0 \rightarrow 18$
2 standard reflections every 120 reflections intensity decay: 2.0%

Refinement

Refinement on F
R = 0.044
wR = 0.063
S = 1.95
3678 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00063|F_o|^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.005
$\Delta\rho_{\rm max}$ = 0.43 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.34 e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (IV).

P2—N1	1.749 (4)	P27—N10	1.699 (2)
P2—N3	1.758 (3)	P27—N26	1.749 (3)
P2—N10	1.681 (2)	P27—N28	1.763 (3)
N1—P2—N3	87.3 (1)	N10—P27—N28	103.6 (2)
N1—P2—N10	105.9 (2)	N26—P27—N28	86.4 (1)
N3—P2—N10	103.3 (2)	P2—N10—P27	124.0 (1)
N10—P27—N26	106.9 (2)		

Compound (III*b*)

Crystal data

$C_{10}H_{16}F_6N_3O_4PS_2$
M_r = 451.34
Triclinic, $P\overline{1}$
a = 8.972 (2) Å
b = 9.116 (6) Å
c = 12.990 (7) Å
α = 97.40 (4)°
β = 104.03 (4)°
γ = 60.58 (4)°
V = 897.8 (9) Å^3
Z = 2
D_x = 1.670 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.0–12.3°
μ = 0.47 mm^{-1}
T = 293 K
Prism, colourless
0.25 × 0.18 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
5444 measured reflections
5227 independent reflections
3076 reflections with $I > 3\sigma(I)$
$R_{\rm int}$ = 0.022
$\theta_{\rm max}$ = 30°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 18$
2 standard reflections every 120 reflections intensity decay: 10.8%

Refinement

Refinement on F
R = 0.049
wR = 0.070
S = 1.91
3076 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00090|F_o|^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.008
$\Delta\rho_{\rm max}$ = 0.53 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.39 e Å^{-3}

Compound (IV)

Crystal data

$C_{17}H_{23}F_{12}N_5O_8P_2S_4\cdot0.98CHCl_3$
M_r = 960.65
Monoclinic, $P2_1$
a = 8.812 (4) Å
b = 13.897 (3) Å
c = 14.728 (6) Å
β = 90.21 (3)°
V = 1803 (1) Å^3
Z = 2
D_x = 1.767 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.0–11.6°
μ = 0.68 mm^{-1}
T = 293 K
Monoclinic prism, colourless
0.22 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
5666 measured reflections
5519 independent reflections
4855 reflections with $I > 0$
$R_{\rm int}$ = 0.031
$\theta_{\rm max}$ = 30.9°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 20$
2 standard reflections every 120 reflections intensity decay: 7.0%

Refinement

Refinement on F
R = 0.086
wR = 0.051
S = 1.88
4855 reflections
486 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00013|F_o|^2]$
$(\Delta/\sigma)_{\rm max}$ = 0.007
$\Delta\rho_{\rm max}$ = 0.46 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.61 e Å^{-3}

Table 4
Comparisons of selected parameters between the three title compounds (°, Å).

	(III*a*)	(III*b*)	(IV)
S distances from ring planes			
S13	−0.298 (1)	0.387 (1)	0.667 (1)
S14	−0.321 (1)	0.553 (1)	0.460 (1)
S37			0.774 (1)
S38			0.373 (1)
Bonded torsion angles			
N10—P2—N1—S13	94.9 (1)	−89.8 (1)	−94.6 (2)
N10—P2—N3—S14	−80.5 (1)	99.0 (1)	99.2 (2)
N10—P27—N26—S37			−98.1 (2)
N10—P27—N28—S38			94.9 (2)
Non-bonded torsion angles			
S13—N1···N3—S14	51.5 (5)	30.9(5)	5.4 (9)
S13—N1···N26—S37			−43.5 (2)
S37—N26···N28—S38			−1.9 (9)
S14—N3···N28—S38			43.8 (2)
N distances from surrounding planes			
N1···(P2/C5/S13)	0.260 (1)	0.080 (2)	0.019 (4)
N3···(P2/C4/S14)	0.132 (1)	0.246 (2)	0.258 (4)
N26···(P27/C30/S37)			0.004 (3)
N28···(P27/C29/S38)			0.220 (3)
N—P distances			
P2—N1	1.783 (2)	1.791 (2)	1.749 (4)
P2—N3	1.792 (2)	1.771 (2)	1.758 (3)
P2—N10	1.637 (2)	1.642 (2)	1.681 (2)
P27—N26			1.749 (3)
P27—N28			1.763 (3)
P27—N10			1.699 (2)

Compounds (III*a*) and (III*b*) were analyzed in racemic form and compound (IV) as the *R*,*R*-enantiomer. H atoms were placed in calculated positions and not refined. In compound (IV), the loss of symmetry from $P2_1/m$ to $P2_1$ and the high disorder of the cocrystallized $CHCl_3$ molecule were responsible for the relatively rough *R* factors observed for this structure. The treatment for this disorder was as follows: the occupancy factor of the site of the solvent molecule was first refined and then fixed at 0.983. The three Cl atoms were statistically spread over five different sites. The sum of the five occupancy factors was then set to three times the occupancy factor of the site of the solvent molecule, *i.e.* 3 × 0.983 = 2.949. The loss of symmetry arises from the fact that the pseudo-mirror plane exists neither for the highly disordered solvent molecule nor for the cyclohexyl group, which would become a phenyl group in contradiction with the chemical nature of the compound. Space group $P2_1$ has a twofold screw axis. The heaviest atom can be chosen as an origin, and its coordinate along this twofold screw axis can then be fixed and not refined. Here, the heaviest atom is Cl, but since it is part of a severely disordered solvent molecule, we preferred to choose as origin an S atom on the main molecule, which is far less disordered, and an S atom is not much lighter than a Cl atom. Doing this, we could achieve a better refinement of the molecular structure.

For all compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992–1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1537). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Brookhart, M., Chandler, W. A., Pfister, A. C., Santini, C. C. & White, P. S. (1992). *Organometallics*, **11**, 1263–1268.

Brunel, J.-M., DelCampo, B. & Buono, G. (1998). *Tetrahedron Lett.* **39**, 9663–9666.

Collman, J. P., Hegedus, L. S., Norton, J. R. & Finke, R. G. (1987). *Principles and Applications of Organotransition Metal Chemistry*, p. 66. Mill Valley: University Science Books.

Denmark, S. E., Coe, D. M., Pratt, N. E. & Griedel, B. D. (1994). *J. Org. Chem.* **59**, 6161–6163.

Denmark, S. E., O'Connor, S. P. & Wilson, S. R. (1998). *Angew. Chem. Int. Ed.* **37**, 1149–1153.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Feringa, B. L. (2000). *Acc. Chem. Res.* **33**, 346–353.

Gimbert, Y., Robert, F., Durif, A., Averbuch, M.-T., Kann, N. & Greene, A. E. (1999). *J. Org. Chem.* **64**, 3492–3497.

Hersh, W. H., Xu, P., Wang, B., Won Yon, J. & Simpson, C. K. (1996). *Inorg. Chem.* **35**, 5453–5459.

Hilgraf, R. & Pfaltz, A. (1999). *Synlett*, pp. 1814–1816.

Jacobsen, E. N., Pfaltz, A. & Yamamoto, H. (1999). Editors. *Comprehensive Asymmetric Catalysis*, Vols. 1–3. New York: Springer.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Jolly, R. S., Luedtke, G., Sheehan, D. & Livinghouse, T. (1990). *J. Am. Chem. Soc.* **112**, 4965–4966.

King, R. B. & Gimeno, J. (1978). *Inorg. Chem.* **17**, 2390–2395.

Kremer, T., Hampel, F., Knoch, F. A., Bauer, W., Schmidt, A., Gabold, P., Schütz, M., Ellermann, J. & Schleyer, P. v. R. (1996). *Organometallics*, **15**, 4776–4785.

Kundig, E. P., Dupré, C., Bourdin, B., Cunningham, A. Jr & Pons, D. (1994). *Helv. Chim. Acta*, **77**, 421–428.

Leeuwen, P. W. N. M. van & Roobeck, C. F. (1981). *Tetrahedron*, **37**, 1973–1982.

Leeuwen, P. W. N. M. van & Roobeck, C. F. (1983). *J. Organomet. Chem.* **258**, 343–347.

Levason, W. (1990). *The Chemistry of Organophosphorous Compounds*, Vol. 1, edited by F. R. Hartlet, ch. 15. New York: Wiley.

McCauliffe, C. A. (1987). *Comprehensive Coordination Chemistry*, Vol. 2, edited by G. Wilkinson, R. D. Gillard & J. A. McCleverty, p. 989. Oxford: Pergamon.

Molecular Structure Corporation (1992–1997). *TEXSAN*. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Moloy, K. G. & Petersen, J. L. (1995). *J. Am. Chem. Soc.* **117**, 7696–7710.

Phillips, I. G., Ball, R. G. & Cavell, R. G. (1988). *Inorg. Chem.* **27**, 4038–4045.

Pignolet, L. H. (1983). Editor. *Homogeneous Catalysis with Metal Phosphine Complexes*. New York: Plenum.

Schnabel, C. R. & Roddick, D. M. (1993). *Inorg. Chem.* **32**, 1513–1518.

Takahashi, H., Kawakita, T., Ohno, M., Yoshioka, M. & Kobayashi, S. (1992). *Tetrahedron*, **48**, 5691–5699.

Whitaker, C. M., Kott, K. L. & McMahon, R. J. (1995). *J. Org. Chem.* **60**, 3499–3508.