Reactions of Octacarbonyldicobalt with some Ditertiary Phosphines and Arsines

By **D. J. Thornhill** and **A. R. Manning,*** Department of Chemistry, University College, Dublin 4, Ireland

Octacarbonyldicobalt reacts with ditertiary phosphines and arsines (L–L) to give $[(L{-}L)Co(CO)_2]_2$ or $[(L{-}L)Co_2(CO)_6]$ complexes *via* the ionic intermediates $[(L{-}L)_3Co_2(CO)_4][Co(CO)_4]$ and $[(L{-}L)Co(CO)_3][Co(CO)_4]$ respectively. The route adopted depends on (L–L). The i.r. spectra of all compounds between 1700 and 2100 cm^{-1} are reported, and structures proposed which, for the covalent complexes, are based on the two tautomers of $Co_2(CO)_8$. The isomer obtained with the carbonyl-bridged $[(Me_2As[CH_2]_nAsMe_2)Co_2(CO)_6]$ derivatives depends on *n*.

THE initial products of the reactions of monotertiary phosphines, arsines, and stibines, L, with octacarbonyldicobalt are the ionic complexes, $[trans\text{-}L_2Co(CO)_3][Co(CO)_4]$. They are readily converted to covalent $[LCo(CO)_3]_2$ derivatives.[1-3] When the work described here was started, the related reactions of polytertiary ligands had received little attention, although 1,2-bis(diphenylphosphino)ethane was reported to give $[(Ph_2PCH_2CH_2PPh_2)_3Co_2(CO)_4][Co(CO)_4]_2$.[4]

It seemed probable that if $Co_2(CO)_8$ were reacted with a series of $R_2M(CH_2)_nMR_2$ ligands ($R =$ alkyl or aryl; $M = P$ or As; and $n > 1$), the products would vary with R, M, or *n*. Of particular interest to us was the preparation of CO-bridged complexes of the general type $[\{R_2M(CH_2)_nMR_2\}Co_2(CO)_6]$ in which the particular isomer obtained would depend on *n* (see ref. 5).

Whilst this work was in progress, it was reported that some fluorocarbon-bridged diarsines and diphosphines gave two types of carbonyl-bridged $[(L{-}L)Co_2(CO)_6]$ complexes with $Co_2(CO)_8$.[6,7] The structure of one of these has been confirmed by an *X*-ray diffraction study.[6,8]

EXPERIMENTAL

Chemicals were purchased from the usual commercial sources. All reactions were carried out under an atmosphere of nitrogen in purified solvents at room temperature unless it is stated otherwise.

The bidentate ligands were prepared using previously published methods or modifications thereof.[9-11] The melt-

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ing or boiling points of new ligands are: $Me_2As[CH_2]_nAsMe_2$ b.p. 103—104 °C, 15 mmHg when $n = 3$, 108—110 °C, 9 mmHg when $n = 4$, *ca.* 98 °C, 2·5 mmHg when $n = 5$, 108—112 °C, 2·5 mmHg when $n = 6$, and 121 °C, 1·0 mmHg when $n = 8$; $Ar_2PCH_2CH_2PAr_2$ melt at 93—94 °C when Ar = *o*-tol, and at 135—136 °C when Ar = *p*-tol.

The reactions of equimolar amounts of the ligands, (L–L), and octacarbonyldicobalt (*ca.* 1 g) in acetone (*ca.* 25 ml) at 0 °C gave the ionic complexes $[(L\text{–}L)_3Co_2(CO)_4][Co(CO)_4]_2$ or $[(L\text{–}L)Co(CO)_3][Co(CO)_4]$. The products were purified by recrystallization from methanol–acetone.

Gentle warming of tetrahydrofuran solutions of the $[(L\text{–}L)Co(CO)_3][Co(CO)_4]$ derivatives gave the covalent $[(L\text{–}L)Co_2(CO)_6]$ complexes. The $[(L\text{–}L)Co(CO)_2]_2$ compounds were obtained by refluxing equimolar amounts of $[(L\text{–}L)_3Co_2(CO)_4][Co(CO)_4]_2$ and free (L–L) in benzene for *ca.* 1·5 h. When the reactions were complete, the solvents were removed at reduced pressures. The products were recrystallized from benzene–heptane or toluene–pentane mixtures.

Melting points and analyses for all products are summarized in Table 1. Reaction yields ranged between 36 and 78% for the ionic compounds, and between 1 and 50% for the covalent complexes.

TABLE 1

Melting points and analyses for the ionic compounds of type (I), $[(L\text{–}L)_3Co_2(CO)_4][Co(CO)_4]_2$, and (II), $[(L\text{–}L)Co(CO)_3][Co(CO)_4]$, and the covalent derivatives of type (III), $[(L\text{–}L)Co(CO)_2]_2$, and (IV), $[(L\text{–}L)Co_2(CO)_6]$

Ligand (L–L)	Compound type	M.p. (°C) [a]	Found C	Found H	Found Co	Required C	Required H	Required Co
$Ph_2PCH_2CH_2PPh_2$	(I)		60·7	4·0	13·8	61·1	4·1	13·4
$(p\text{-tol})_2PCH_2CH_2P(p\text{-tol})_2$	(I)		57·4	4·7		63·3	4·0	
$(p\text{-tol})_2PCH_2CH_2P(p\text{-tol})_2$	(I) [b]		62·8	5·1		63·9	5·4	
$Ph_2AsCH_2CH_2AsPh_2$	(II)		49·2	3·7	14·7	49·5	3·0	14·7
$Me_2As[CH]_3AsMe_2$	(II)		28·9	3·0	20·4	29·6	3·2	20·8
$Me_2As[CH_2]_4AsMe_2$	(II)		29·8	3·7	20·9	31·0	3·5	20·4
$Me_2As[CH_2]_5AsMe_2$	(II)		31·6	3·7	20·1	32·2	3·7	19·9
$Me_2As[CH_2]_6AsMe_2$	(II)		33·1	4·0	19·9	33·6	4·0	19·4
$Ph_2PCH_2CH_2PPh_2$	(III)	dec. 194	65·9	4·7	11·8	65·5	4·7	11·5
$(p\text{-tol})_2PCH_2CH_2P(p\text{-tol})_2$	(III)	125—127	66·8	5·4		67·5	5·6	
$Ph_2AsCH_2CH_2AsPh_2$	(IV)	dec. 119	49·8	3·5	15·5	49·7	3·1	15·3
$Me_2As[CH_2]_3AsMe_2$	(IV)	106—108	28·4	3·4	22·0	29·0	3·3	21·9
$Me_2As[CH_2]_4AsMe_2$	(IV)	133—135	30·5	3·7	21·8	30·4	3·6	21·4
$Me_2As[CH_2]_6AsMe_2$	(IV)	dec. 115	33·5	4·3	20·3	33·1	4·1	20·4
$Me_2As[CH_2]_8AsMe_2$	(IV)	Oil	35·0	4·5		35·5	4·6	
$Ph_2P[CH_2]_4PPh_2$	(IV) [c]	196—198	57·1	4·5	16·0	57·4	3·9	16·6
$Ph_2P[CH_2]_4PPh_2$	(IV) [d]		55·6	3·9	17·4	57·4	3·9	16·6
$o\text{-}(Me_2As)_2C_6H_4$	(IV)	dec. 140	33·8	3·1	20·4	33·5	2·8	20·6

[a] Determined in sealed tubes. The ionic compounds of types (I) and (II) decomposed without melting over wide, and irreproducible, temperature ranges. dec. = decomposed without melting. [b] The formula of this compound $[(L\text{–}L)_3Co_2(CO)_4][BF_4]_2$. [c] Soluble product (see text). [d] Insoluble product (see text).

I.r. spectra were obtained as described elsewhere.[5] The frequencies and relative intensities of absorption bands which lie between 1700 and 2100 cm^{-1} are given in Tables 2—4. Some spectra are illustrated in Figure 1.

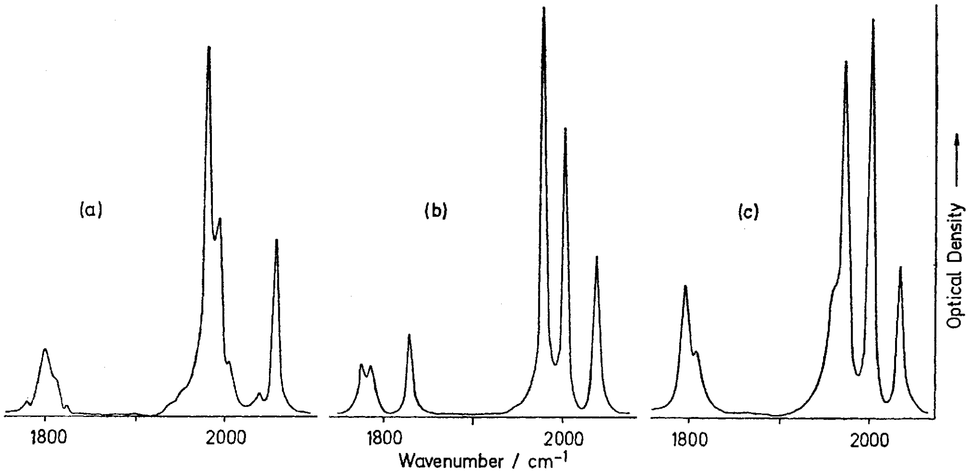

FIGURE 1 The i.r. spectra of hexane solutions of (a), $[(Me_2As[CH_2]_3AsMe_2)Co_2(CO)_6]$, a 1,1′-isomer; (b), $[(Me_2As[CH_2]_4AsMe_2)Co_2(CO)_6]$, a 2,2′-isomer; and (c), $[(Me_2As[CH_2]_8AsMe_2)Co_2(CO)_6]$, a 2,3′-isomer between, 1700 and 2100 cm^{-1} at 25 °C

RESULTS AND DISCUSSION

Salts are the initial products of the reactions of octacarbonyldicobalt with ditertiary phosphines and arsines at 0 °C in polar and non-polar solvents {*cf.* Ph_3P and $Co_2(CO)_8$ [1]}. They are of type (I), $[(L\text{–}L)_3Co_2(CO)_4][Co(CO)_4]_2$, when (L–L) = (a) $Ph_2PCH_2CH_2PPh_2$ and

(b) $(p\text{-tol})_2PCH_2CH_2P(\text{tol-}p)_2$ (tol = MeC_6H_4), and of type (II), $[(L\text{–}L)Co(CO)_3][Co(CO)_4]$, when (L–L) = $Ph_2AsCH_2AsPh_2$, (b) $Me_2As(CH_2)_nAsMe_2$ (n = 3—6 and 8), (c) $o\text{-}(Me_2As)_2C_6H_4$, and (d) $Ph_2P(CH_2)_4PPh_2$. Neither (IIb, n = 8) nor (IId) could be purified, but the cation from the former was isolated as $[(L\text{–}L)_3Co_2(CO)_4][BF_4]_2$. Complex (IIc) could not be isolated. Both $Ph(C_6H_{11})PCH_2CH_2P(C_6H_{11})Ph$ and $(o\text{-tol})_2PCH_2CH_2P(\text{tol-}o)_2$ gave unstable salts containing the $[Co(CO)_4]^-$ anion.

The ionic complexes are yellow to brown solids which decompose in the solid state. Even if (IIb) are stored at −25°, the odours of the free α,ω-bis(dimethylarsino)alkanes may always be detected.

The i.r. spectra of the salts (Table 2) show absorption bands which may be attributed to ν(CO) vibrations of the $[Co(CO)_4]^-$ anion (*cf*. ref. 12) and the cations. The numbers and relative intensities of those due to the cations are consistent with trigonal bipyramidal co-ordination about the cobalt atoms. In (I) the CO ligands occupy two equatorial co-ordination positions on each of the two metal atoms which are bridged by one or three (L–L) ligands. In (IIb) and (IId) the axial co-ordination positions are occupied by chelating or bridging bidentate ligands,* whilst in (IIa) $Ph_2AsCH_2CH_2AsPh_2$ appears to chelate through an axial and an equatorial co-ordination site.

When suspensions of (I) in benzene are heated with more of the diphosphine, $[(L\text{–}L)Co(CO)_2]_2$ derivatives (III) are obtained as rather unstable brown solids, [L–L = (a) $Ph_2PCH_2CH_2PPh_2$, and (b) $(p\text{-tol})_2PCH_2CH_2P(\text{tol-}p)_3$]. The i.r. spectra between 1700 and 2100 cm^{-1} are consistent with structures based on the carbonyl-bridged isomer of $Co_2(CO)_8$ with a non-planar $Co(\mu\text{-}CO)_2Co$ bridging system,[14] and chelating bidentate ligands. Similar complexes have been prepared previously by an indirect route.[15]

When $[L_2Co(CO)_3][Co(CO)_4]$ complexes of monodentate phosphines, arsines, and stibines, L, are heated, $[LCo(CO)_3]_2$ derivatives are obtained.[1] Similar treatment converts the salts (II) to the covalent $[(L\text{–}L)Co_2(CO)_6]$ complexes (IV) {L–L = (a) $Ph_2AsCH_2CH_2AsPh_2$, (b) $Me_2As(CH_2)_nAsMe_2$ (n = 3—5 and 8), (c) $o\text{-}(Me_2As)_2C_6H_4$ (*cf*. refs. 6 and 7), and (d) $Ph_2P[CH_2]_4PPh_2$}. With the exception of (IVb, n = 8) which is a liquid, all are unstable red-brown solids. (IVb, n = 5) was too unstable to isolate.

Two isomeric forms of (IVd) were obtained. The spectra of both (Table 3) indicate a $Co_2(CO)_6$ moiety with D_{3d} local symmetry {*cf*. $[Ph_3PCo(CO)_3]_2$ [13]}. Thus it is probable that both products are best formulated as $[\text{–}CH_2CH_2(Ph)_2PCo(CO)_3\cdot Co(CO)_3P(Ph)_2CH_2CH_2\text{–}]_n$ with n = *ca*. 1—3 for the soluble isomer and >*ca*. 4 for the insoluble one.

The remaining $\{(L\text{–}L)Co_2(CO)_6\}$ complexes are of ditertiary arsines. Unlike the corresponding derivatives of monodentate arsines,[5] they exist as CO-bridged isomers only. It is probable that all are monomeric and have structures based on the bridged form of $Co_2(CO)_8$ [14] with (L–L) occupying one terminal co-ordination position on each cobalt atom, *cf*. $[\{\overline{CF_2(Me_2As)C{=}C(AsMe_2)CF_2}\}Co_2(CO)_6]$.[6,8] † Four isomers are possible (Figure 2).

The six complexes may be divided into three groups on the basis of their i.r. spectra in the regions 1750—1850 and 1900—2100 cm^{-1} where the C–O stretching vibrations of bridging and terminal carbonyl groups give rise to their respective absorption bands. An example of each type is illustrated in Figure 1.

It is probable that 1,1′-isomers are the principal species present in solutions of (IVa), (IVb, n = 3), and (IVc). This is consistent with the small frequency separation of the absorption bands at *ca*. 1800 and 1810 cm^{-1} due to their ν(COμ) vibrations (see ref. 5). At the same time the patterns of the absorption bands due to their terminal ν(CO) modes are similar to those observed for, *e.g.* $\{(\mu\text{-}MeS)Fe(CO)_2PPh_3\}_2$ [16] or $\{(\mu\text{-}PhC{=}CH)Co_2(CO)_4[P(OMt)Ph_2]_2\}$ [17] (MtO^- = mentholate anion) where phosphine ligands occupy 1 and 1′ co-ordination positions of $Co_2(CO)_8$-type molecules. A similar structure has been suggested for $\{Me_2AsC(CF_3){=}C(CF_3)AsMe_2\}Co_2(CO)_6$.[6,7]

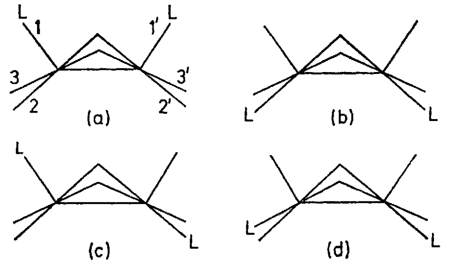

FIGURE 2 The four possible isomers of $[L_2Co_2(CO)_6]$ (a), 1,1′-; (b) 2,2′-; (c) 1,2′-; and (d) 2,3′-. The numbering scheme for the terminal co-ordination positions of a bridged $Co_2(CO)_8$-type molecule is given on (a). Co atoms and CO ligands have been omitted for clarity

The i.r. spectrum of (IVb, n = 4) in the terminal ν(CO) region is similar to that of the known 2,2′-$[\{\overline{CF_2(Me_2As)C{=}C(AsMe_2)CF_2}\}Co_2(CO)_6]$ [6-8] (Figure 1), and is virtually independent of solvent and temperature. Unexpectedly, there are three absorption bands due to its ν(COμ) vibrations which are affected by these variables in a manner which is consistent with the presence of two species in equilibrium (Table 4). We suggest that they differ only in the conformations adopted by the tetra-

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* The spectra should be compared with that of $[trans\text{-}(Ph_3P)_2Co(CO)_3]^+$.[13]

† Species containing a chelating L–L ligand may be discounted; see ref. 5.

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methylene chain. In one 'conformer' of the 2,2′-isomer, the chain is close to the bridging CO ligand, in the other it is further away.* Although these differences would be expected to have little or no effect on the terminal ν(CO) modes, both forms will give rise to two ν(COμ) vibrations. Those of higher frequencies are coincident. This type of isomerism would not be possible if the L–L ligand were rigid, *e.g.* $CF_2(Me_2As)C{=}C(AsMe_2)CF_2$.

TABLE 2

The i.r. spectra of the ionic complexes of type (I), $[(L\text{–}L)_3Co_2(CO)_4][Co(CO)_4]_2$, and type (II), $[(L\text{–}L)Co(CO)_3][Co(CO)_4]$, between 1800 and 2100 cm^{-1} (tetrahydrofuran solutions)

Ligand (L–L)	Compound type	Absorption bands [a]			
$Ph_2PCH_2CH_2PPh_2$	(I)	1885(10) [b]	1955(3·8)		2007(1·7)
$(p\text{-tol})_2PCH_2CH_2P(p\text{-tol})_2$	(I)	1884(10)	1950(3·0)		2003(1·4)
$Ph_2AsCH_2CH_2AsPh_2$	(II)	1885(10)	1951(2·8)	1990(5·8)	2006(sh)
$Me_2As[CH_2]_3AsMe_2$	(II)	1884(10)		1994(4·6)	
$Me_2As[CH_2]_4AsMe_2$	(II)	1884(10)		1998(5·0)	
$Me_2As[CH_2]_5AsMe_2$	(II)	1885(10)		1994(5·0)	
$Me_2As[CH_2]_6AsMe_2$	(II)	1885(10)		1997(4·1)	
$Me_2As[CH_2]_8AsMe_2$	(II)	1884(10)		1993(4·0)	
$Ph_2P[CH_2]_4PPh_2$	(II)	1886(10)		2001(3·6)	

[a] Peak positions (cm^{-1}) with relative peak heights in parentheses. [b] The absorption bands at 1884—1886 cm^{-1} are due to the $[Co(CO)_4]^-$ anion; the remainder are due to the various cations.

TABLE 3

I.r. spectra [a] of the covalent compounds of type (III), $[(L\text{–}L)Co(CO)_2]_2$ and (IV), $[(L\text{–}L)Co_2(CO)_6]$

Ligand	Compound type	Absorption bands										
$Ph_2PCH_2CH_2PPh_2$ [b]	(III)		1728 (14·2)	1752 (4·6)				1922 (10)	1957(8·7)			
$(p\text{-tol})_2PCH_2CH_2P(p\text{-tol})_2$ [b]	(III)		1727 (11·2)	1750 (3·6)				1918 (10)	1954(7·7)			
$Ph_2AsCH_2CH_2AsPh_2$	(IV)			1798 (4·0) [c]	1812 (sh) [c]	1832 (1·0) [d]		1990 (8·9) [c]	2000 (10) [c]	2012 (2·4) [d]	2046 (2·0) [d]	2062 (5·4) [c]
$Me_2As[CH_2]_3AsMe$	(IV)		1781 (0·4) [d]	1800 (1·5) [c]	1812 (sh) [c]	1822 (0·3) [d]		1983 (10) [c]	1995 (5·4) [c]	2006 (1·4) [d]	2041 (0·6) [d]	2058 (5·0) [c]
$Me_2As[CH_2]_4AsMe_2$	(IV)	1777 (1·3) [d]	1788 (1·2) [d]			1829 (2·0) [d]	1980 (10) [d]			2002 (7·0) [d]	2038 (4·0) [d]	
$Me_2As[CH_2]_6AsMe_2$	(IV)		1796 (3·2) [e]	1808 (1·6) [e]			1964 (sh) [e]	1972 (8·8) [e]		2003 (10) [e]	2036 (4·0) [e]	
$Me_2As[CH_2]_8AsMe_2$	(IV)		1795 (3·2) [e]	1806 (sh) [e]			1957 (sh) [e]	1969 (8·1) [e]		2002 (10) [e]	2034 (3·0) [e]	
$o\text{-}(Me_2As)_2C_6H_4$	(IV)		1791 (sh) [d]	1799 (2·1) [c]	1811 (sh) [c]	1821 (sh) [d]		1989 (10) [c]	1994 (6·7) [c]	2012 (1·2) [d]	2047 (sh) [d]	2057 (5·1) [c]
$Ph_2P[CH_2]_4PPh$ [b,f]	(IV)							1953(10)		1973(sh)		
$Ph_2P[CH_2]_4PPh_2$ [g]	(IV)							1946 (s)	1973(sh)		2026(vw)	

[a] Spectra between 1700 and 2100 cm^{-1} in hexane solution unless stated otherwise, peak positions (cm^{-1}) with relative peak heights in parentheses. Those lying below 1850 cm^{-1} are due to the ν(CO) vibrations of bridging ligands, the remainder are due to terminal CO groups. [b] CS_2 Solution. [c] Due to 1,1′-isomer. [d] Due to 2,2′-isomer. [e] Due to 2,3′-isomer. [f] Soluble product. [g] Insoluble product. Nujol mull.

TABLE 4

The effects of solvent and temperature on the i.r. spectrum of $[(Me_2As[CH_2]_4AsMe_2)Co_2(CO)_6]$ between 1700 and 2100 cm^{-1}

Solvent [a]	Absorption bands [b]					
Hexane	1777(1·3)	1788(1·2)	1829(2·0)	1980(10)	2002(7·0)	2038(4·0)
CS_2	1776(2·0)	1783(2·0)	1816(2·0)	1971(10)	1995(8·5)	2032(6·2)
CS_2(−80 °C)	1775(3·7)	1784(sh)	1813(2·0)	1969(10)	1993(8·8)	2031(6·5)
Xylene	1779(2·0)	1785(2·0)	1816(1·7)	1973(10)	1997(6·7)	2034(5·0)
THF	1775(sh)	1783(2·5)	1810(1·7)	1969(10)	1992(7·0)	2030(5·2)

[a] THF = tetrahydrofuran. [b] Peak position (cm^{-1}) with relative peak heights in parentheses. Absorption band with frequencies less than 1850 cm^{-1} are due to the ν(CO) vibrations of bridging carbonyl groups, whilst the remainder are due to terminal CO ligands.

A third isomeric form is adopted by (IVb, $n = 6$) and (IVb, $n = 8$) (Figure 1 and Table 3). The small frequency separation of the absorption bands due to their ν(COμ) modes suggests that the two bridging ligands are in similar environments. Thus these complexes must be 2,3′-isomers (Figure 2).

Despite the inherent instability of (IVa), (IVb, $n = 3$), and (IVc), there are a number of weak, real peaks in

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* That this is possible may be confirmed by the use of models.

their i.r. spectra which may be attributed to small amounts of the 2,2′-isomers. These appear to be in equilibrium with the more important 1,1′-forms, and decline in importance as the solvent polarity increases. Similar behaviour has been reported for [$(Me_2AsCHFCF_2$-$AsMe_2)Co_2(CO)_6$].[6,7]

The configuration adopted by (IVa), (IVb), and (IVc) appears to be that which minimises the steric interactions between arsenic atoms. If it is assumed that in these compounds the As atoms have replaced the CO ligands of $Co_2(CO)_8$ [*cf.* $Co_2(CO)_8$ [14] with [$CF_2(Me_2As)C{=}C(AsMe_2)CF_2$]$Co_2(CO)_6$,[6,8]] then the 2,2′-species would be expected for the L–L having the shortest polymethylene chain, *e.g.* for (IVb, $n = 3$) rather than (IVb, $n = 4$). However, the effective span of a ligand may be limited by its rigidity, *e.g.* *o*-$(Me_2As)C_6H_4$, or by internal non-bonded interactions, *e.g.* within the polymethylene chain of $R_2As(CH_2)_nAsR_2$.

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