Phosphorus–Tellurium Compounds. Part 3.[1] Cyclopentadienyliron Tellurophosphorane Complexes: Tellurium-bridged Iron Phosphine Compounds?

Norbert Kuhn * **and Hans Schumann**
Fachbereich 6 (Chemie) der Universität (GH) Duisburg, Lotharstrasse 1, D-4100 Duisburg 1, Germany

Cyclopentadienyliron tellurophosphorane cations $[Fe(C_5H_5)(CO)_2(TePR_3)]^+$ (**5**; R = Me, Pr^i, Bu^t, NMe_2, or morpholino) have been prepared by reaction of $[Fe(C_5H_5)(CO)_2(thf)]^+$ (thf = C_4H_8O) with $TePR_3$. The bonding properties of the Fe–Te–P fragment are discussed using n.m.r. data which indicate π bonding between the iron and tellurium atoms.

The tellurium-atom-bridged di- and tri-nuclear transition metal complexes $[\{Mn(C_5Me_5)(CO)_2\}_2Te]$ (**1**) and $[\{Mn(C_5H_5)(CO)_2\}_3Te]$ (**2**) have been reported recently.[2–4] The ability of tellurium atoms to connect phosphines as a transition state $Te(PR_3)_2$ (**3**) in a fluxional redox process is also well established[5] while the existence of stable compounds of this type[6] needs further proof.

Recently, we have described the first tellurophosphorane complexes $[M(CO)_5(TePR_3)]$(R = Bu^t; M = Cr, Mo, or W)[7] in which structural and spectroscopic data indicate the tellurophosphorane to be a strong donor ligand. A detailed investigation on the cations $[Fe(C_5H_5)(CO)_2(XPR_3)]^+$ (X = O, S, or Se) has been reported previously.[8] The present investigation of cyclopentadienyliron tellurophosphorane cations should give some insight into the properties of both the metal–tellurium and tellurium–phosphorus bonds.

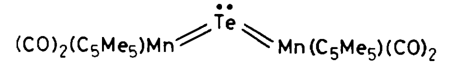
Results and Discussion

As well as with other phosphine chalcogenides[8] the co-ordination of tellurophosphoranes is achieved without problems starting from the cation $[Fe(C_5H_5)(CO)_2(thf)]^+$ (thf = tetrahydrofuran)[9] and the dark brown complexes $[Fe(C_5H_5)(CO)_2(TePR_3)]^+$ (**5**) are obtained in almost quantitative yields (see Scheme). The compounds (**5**) are fairly air-stable in the solid state. Solutions of (**5a**)—(**5d**) slowly eliminate elemental tellurium and the formation of the corresponding phosphine complexes $[Fe(C_5H_5)(CO)_2(PR_3)]^+$ can be observed. The stability depends on the nature of the substituent R according to the series R = $Pr^i \gg Bu^t > NMe_2 >$ Me.

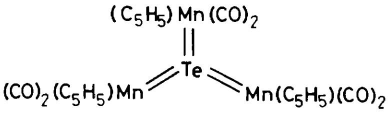
Additional decomposition of (**5c**), forming inorganic iron phosphine complexes occurs on exposure of the solution to light. In contrast to this route involving phosphorus–tellurium bond fission the morpholino-substituted complex (**5e**), which is the least stable one of this series, decomposes to give $[Fe(C_5H_5)(CO)_3]^+$ and $TeP(mor)_3$ (**4e**) (mor = morpholino), the phosphorus–tellurium bond remaining intact here.

We have not been able to prepare cations of the type $[Fe(C_5H_5)(CO)(TePR_3)_2]^+$, either by heating or by irradiation of the cations (**5**) with additional amounts of tellurophosphoranes (**4**). In both cases extensive decomposition occurs. On the other hand, $[Fe(C_5H_5)(CO)(TeMe_2)_2]^+$ has been found to be stable.[10] Reaction of (**5**) with iodide gives the dicarbonyl complex $[Fe(C_5H_5)(CO)_2I]$. Possibly the tellurium–iron bond is destabilized by co-ordination of a second ligand with high electron density at the donor atom as a consequence of repulsion between the ligand co-ordinating atoms.

Carbon-13 n.m.r. and i.r. data of the $[Fe(C_5H_5)(CO)_2(TePR_3)]^+$ cations (Table 1) indicate the tellurophosphoranes to be stronger donor ligands than the corresponding thio- and seleno-phosphoranes.[8] This may be a consequence of the more 'ylidic' nature of the phosphorus–chalcogen bond in the tellurium compounds. In fact, for thiophosphoranes as well as for their co-ordination compounds, a phosphorus–sulphur double bond has been proposed[11,12] while the



(**1**)



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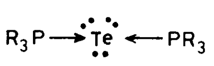

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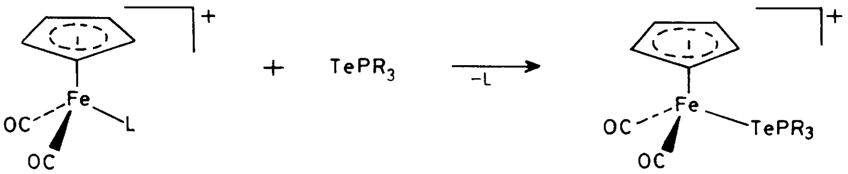
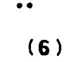


(**4**) (**5**)

R = Me(**a**) R = Pr^i(**b**) R = Bu^t (**c**)
R = NMe_2 (**d**) R = mor (**e**)



(**6**)

Scheme. L = thf

Table 1. Infrared,[a] ^1H n.m.r.,[b] and ^{13}C n.m.r.[c] data for $[Fe(C_5H_5)(CO)_2(TePR_3)]BF_4$ (**5**)

Compound	R	I.r. $\nu(CO)/cm^{-1}$	^1H N.m.r. δ/p.p.m.	^{13}C N.m.r. δ/p.p.m.
(**5a**)	Me	1 988, 2 037	2.20 [d, Me, *J*(PH) 14.4], 5.53 (s, C_5H_5)	16.73 [d, Me, *J*(PC) 39.8], 86.81 (s, C_5H_5), 214.22 [d, CO, *J*(PCO) 5.6]
(**5b**)	Pr^i	1 985, 2 030	1.37 [dd, Me, *J*(HH) 7, *J*(PH) 17], 2.53 [m, CH, *J*(HH) 7, *J*(PH) 9.9], 5.52 (s, C_5H_5)	18.71 [d, Me, *J*(PC) 2.9], 25.92 [d, CH, *J*(PC) 26.3], 87.30 (s, C_5H_5), 214.44 [d, CO, *J*(PCO) 3.1]
(**5c**)	Bu^t	1 994, 2 038	1.72 [d, Me, *J*(PH) 15.3], 5.68 (s, C_5H_5)	31.43 (s, Me), 37.86 [d, CMe_3, *J*(PC) 29], 87.72 (s, C_5H_5), 212.85 [d, CO, *J*(PCO) 2]
(**5d**)	NMe_2	1 983, 2 028	2.71 [d, Me, *J*(PH) 11], 5.46 (s, C_5H_5)	37.37 [d, Me, *J*(PC) 3.5], 86.95 (s, C_5H_5), 214.64 [d, CO, *J*(PCO) 8.4]
(**5e**)	mor	1 980, 2 025	3.17 (m, br, NCH_2), 3.72 (m, br, OCH_2), 5.26 (s, C_5H_5)	47.73 (s, NCH_2), 66.49 (s, OCH_2), 86.28 (s, C_5H_5), 213.13 (s, CO)

[a] Recorded on a Perkin-Elmer 397 instrument in CH_2Cl_2. [b] Recorded on Bruker WP 80 (80 MHz) in $[^2H_6]$acetone, (**5a**)—(**5d**), or Bruker WH 270 instruments (270 MHz) in CD_2Cl_2, (**5e**); *J* values in Hz. [c] Recorded on Varian XL-100 FT (25.5 MHz) in $[^2H_6]$acetone at 200 K, (**5a**)—(**5d**) (proton decoupled), or Bruker AM 400 instruments (100.6 MHz) in CD_2Cl_2 at 300 K, (**5e**); *J* values in Hz.

Table 2. ^{31}P N.m.r. data (δ/p.p.m.) for $[Fe(C_5H_5)(CO)_2(TePR_3)]BF_4$, $TePR_3$, and R_3PTeMe^+

R	$[M(TePR_3)]BF_4$ [a]	$\Delta\delta(^{31}P)$ [b]	$TePR_3$ [c]	R_3PTeMe^+ [d]
Me	−56.0 (1 295)	9.3	−65.3 (br)[e]	89.8
Pr^i	29.2 (1 399)	−10.0	39.2 (1 728)	82.9
Bu^t	53.8 (br)	−20.6	74.4(1 600)[f]	97.8
NMe_2	23.8 (1 877)	−34.5	58.3 (2 112)	28.7
mor	57.7 (1 821)	−0.5	58.2 (2 096)	20.5

[a] M = $Fe(C_5H_5)(CO)_2$; recorded on a Bruker WP 80 instrument (32 MHz) in $[^2H_6]$acetone; *J*(PTe)/Hz in parentheses. [b] Co-ordination shift: δ(complex) − δ(unco-ordinated ligand). [c] Recorded on Bruker WP 80/Bruker WP 80 SY WG instruments (32 MHz) in toluene at 245 K; *J*(PTe)/Hz in parentheses. [d] Recorded on a Bruker WP 80 instrument (32 MHz) in $[^2H_3]$nitromethane at 300 K; see ref. 1. [e] Due to rapid tellurium exchange between phosphine and tellurophosphorane no *J*(PTe) is observed even at 200 K (W. W. du Mont, personal communication). [f] From W. W. du Mont and H. J. Kroth, *Z. Naturforsch., Teil B*, 1981, **36**, 332.

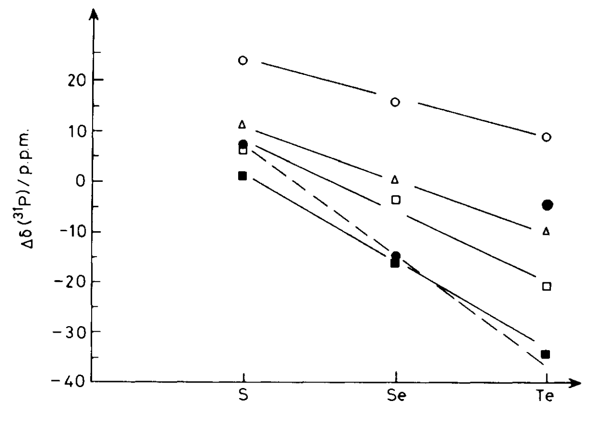


Figure. Co-ordination shifts of the phosphane chalcogenides XPR_3 in the cations $[Fe(C_5H_5)(CO)_2(XPR_3)]^+$ (X = S, Se, or Te); Δδ values from ref. 8 (X = S or Se) and Table 2 (X = Te). R = Me(○), Pr^i (△), Bu^t (□), NMe_2 (■), or mor (●). The values plotted on the abscissa are normalized to give a straight line for R = Pr^i

phosphorus–tellurium bond in compounds of the type $TePR_3$ seems to have a bond order near 1.5,[13] which is decreased markedly on co-ordination to give a phosphorus–tellurium single bond.[7] On the other hand, the increase of electron density in going from sulphur donor ligands to their tellurium analogues is also observed for cations of the type $[Fe(C_5H_5)(CO)_2(XR_2)]^+$ (X = S, Se, or Te),[10] and is possibly a consequence of the nature of the chalcogen donor atom, as discussed elsewhere.[14]

The discussion of bonding properties in relation to heteronuclear n.m.r. shifts is difficult because of the uncertain influence of the paramagnetic shielding term.[15,16] Nevertheless, some trends can be seen comparing the present complexes with structurally similar compounds. Examination of the ^{31}P n.m.r. co-ordination shift (Δδ) of the ligand XPR_3 in the cations $[Fe(C_5H_5)(CO)_2(XPR_3)]^+$ (X = S, Se, or Te) gives an unexpected result (Table 2, Figure): if upfield co-ordination shifts are assigned to be positive, the Δδ values give regular sequences of increasing shielding for the tellurium ligands for R = Me, Pr^i, Bu^t, and NMe_2 while thiophosphoranes are deshielded on co-ordination. The change of sign of Δδ may be interpreted as a superposition of at least two effects of which the deshielding one predominates for the thiophosphoranes, while for the co-ordination of tellurophosphoranes an upfield shift effect gives the major contribution. Thiophosphoranes are suggested to be co-ordinated on metals *via* a σ-donor bond only,[12] and the downfield shift observed here is paralleled by the co-ordination of methylenephosphoranes.[17] In both cases an electron-withdrawing effect may influence the *s* character of the P–X (X = S, Se, Te, CH_2, *etc.*) bond without change of the bond order.

Structural data, however, indicate π bonding from the tellurium atom to the metallic centre in addition to the σ-donor bond in the complexes $[M(CO)_5(TePR_3)]$ (R = Bu^t; M = Cr, Mo, or W)[7] corresponding to the resonance hybrid $M^{2-}{=}Te^+{-}P^+R_3$ which has been confirmed by the preliminary results of Hückel molecular orbital calculations.[18] The electrostatic repulsion between the phosphorus and the tellurium atom here causes a weakening of this bond; π bonding by use of the occupied metal *d* orbitals and the empty σ* P–Te orbital also weakens the phosphorus–tellurium bond. If we suggest that weakening of the phosphorus–tellurium bond increases the *s* character of the orbital used by the phosphorus atom we have an explanation for the ^{31}P n.m.r. upfield co-ordination shift of tellurophosphoranes (**4b**)—(**4d**) in contrast to the behaviour of thiophosphoranes, no metal–sulphur π bonding being present here on co-ordination.

The $XPMe_3$ co-ordination shifts are shown in the Figure to form a series similar to those of the Pr^i, Bu^t, and NMe_2 analogues, though deshielding occurs for all $XPMe_3$ ligands. We suppose this result to be influenced strongly by the small size of the methyl group which favours the *s* character of the phosphorus–chalcogen bond in the XPR_3 species. This effect causes the phosphorus–tellurium bond in (**4a**) to be weaker compared with those of (**4b**)—(**4e**) and additional weakening on co-ordination possibly does not play an important role here.

On the other hand, Me_3PTeMe^+ shows a ^{31}P n.m.r. shift of 89.8 p.p.m., typical for this type of compound, and apparently pure σ-donor co-ordination of (**4a**) causes the phosphorus–tellurium bond to be a 'normal phosphonium *sp*^{3} hybrid' one with respect to the other R_3PTeMe^+ cations (R = Pr^i or Bu^t). It could be argued the change of sign in Δδ values going from SPR_3 to $TePR_3$ may be a consequence of chalcogen electronegativities. However, tellurophosphonium cations R_3PTeMe^+,[1] which may be considered as co-ordination compounds of tellurophosphoranes and methyl cations, show shift values downfield with respect to the corresponding tellurophosphoranes (**4a**)—(**4c**)* (see Table 2), no π bonding between tellurium and the co-ordination centre being possible here and this confirms the importance of metal–tellurium π bonding for the upfield co-ordination shift of tellurophosphoranes in transition metal complexes.

The morpholino-substituted complex (**5e**), whose chemical behaviour differs from that of its analogues (see above), shows a significant difference in the ^{31}P n.m.r., also (Figure). While the shifts of $P(mor)_3$, $TeP(mor)_3$, and $(mor)_3PTeMe^+$ resemble closely those of the corresponding dimethylamino compounds, the shift for (**5e**) is *ca.* 40 p.p.m. downfield from the expected value, and no significant co-ordination shift for $TeP(mor)_3$ is observed here. The lack of iron–tellurium π bonding would explain the route of decomposition which indicates a relatively weak iron–tellurium bond with a strong phosphorus–tellurium bond in (**5e**), but the reason for this differing behaviour is unknown, as yet.

Increasing attention has been focussed on ^{125}Te n.m.r. in recent years.[15] Though the interpretation of shifts needs further work, deshielding seems to occur on increasing co-ordination number of the tellurium atom. Thus, the tellurium atom in tellurophosphoranes is deshielded on co-ordination.[7] On the other hand, an upfield shift is observed on changing the alkyl groups in TeR_2 for acceptor groups (Y) like SnR_3,[20] indicating an additional shielding by Te–Y π bonding. Looking at the series M–$TePR_3$ (R = Bu^t) we see an increase of shielding in the sequence M = Me^+ (752)[21] > $Fe(C_5H_5)(CO)_2{}^+$ (−532) > $W(CO)_5$ (−770 p.p.m.)[7] which may also be a consequence of increasing M–Te π bonding. Interestingly, no co-ordination shift could be detected for $TeP(NMe_2)_3$ (**4d**); it seems that the downfield shift caused by the increase of the co-ordination number at the tellurium atom is compensated by the π-bonding effect here. Unfortunately, we have not been able to obtain ^{125}Te n.m.r. data for (**4e**) and (**5e**) and therefore we cannot say whether the different behaviour of the complexes (**5d**) and (**5e**) influences the ^{125}Te n.m.r. shift.

The coupling constants $^1J(^{125}Te-^{31}P)$ are reported[15] to have positive sign and we therefore suppose the values reported in Table 3 to be positive. Methylenephosphoranes are considered to be of ylidic structure $H_2C^-{-}P^+R_3$ using a carbon *sp*^{2} orbital for the phosphorus–carbon bond. By analogy, in tellurophosphoranes $:\ddot{T}e^-{-}P^+R_3$ the tellurium atom may be attached at the phosphorus *via* an *sp* orbital, and co-ordination should decrease the *s* character of the phosphorus–tellurium bond as well as the positive value of the coupling constant.[15] If, on the other hand, π bonding between the metal and the tellurium atom increases the *s* character of the phosphorus orbital used for the phosphorus–tellurium bond, the coupling constant should be increased by this effect. Looking at the series M–$TePR_3$ (R = Bu^t) we see the $^{125}Te-^{31}P$ coupling constant being increased with increasing π-donor ability of M: M = Me^+ (<50)[21] < $Fe(C_5H_5)(CO)_2{}^+$ (1 480) < $W(CO)_5$ (1 600 Hz).[7] This also may demonstrate the existence of π bonding between tellurium and the iron co-ordination centre in the complexes (**5**).

Table 3. ^{125}Te N.m.r. data for $[Fe(C_5H_5)(CO)_2(TePR_3)]BF_4$ and $TePR_3$

R	$[Fe(C_5H_5)(CO)_2(TePR_3)]BF_4$[a]	Δδ[b]	$TePR_3$[c]
Me	−462.6 (1 333)	50.8	−513.4[d]
Pr^i	−781.8 (1 410)	218.5	−1 000.3 (1 735)
Bu^t	−531.9 (1 480)	307.8	−839.7 (1 600)
NMe_2	−825.8 (2 086)	0.6	−826.4 (2 105)
mor	*e*		*e*

[a] Recorded on a Bruker WM 300 instrument (94.7 MHz) in [$^2H_6$]-acetone; values (δ/p.p.m.) relative to $TeMe_2$, *J*(PTe)/Hz in parentheses. [b] Co-ordination shift: δ(complex) − δ(unco-ordinated ligand). [c] Recorded on a Bruker WM 300 instrument (94.7 MHz) in [$^2H_6$]benzene; values (δ/p.p.m.) relative to $TeMe_2$, *J*(PTe)/Hz in parentheses. [d] *J*(PTe) not resolved due to rapid tellurium exchange between phosphine and tellurophosphorane (W. W. du Mont, personal communication). [e] Not observed.

In summary, n.m.r. data for the cations (**5**) compared with those of thio- and seleno-phosphoranes may be discussed in terms of π bonding between tellurium and the co-ordination centre which does not occur in the case of thiophosphorane co-ordination while selenophosphoranes seem to resemble more closely their sulphur analogues. On the other hand, the phosphorus–chalcogen bond seems to be more weakened on co-ordination of tellurophosphoranes than has been observed for thio- and seleno-phosphoranes. Therefore it seems useful, in our opinion, to consider the cations (**5**) as dinuclear iron–tellurium complex fragments stabilized by a phosphine ligand co-ordinated at the tellurium atom, (**6**). This type of bonding parallels the tellurium-atom-bridged structures (**1**)—(**3**), *i.e.* tellurophosphorane complexes may be considered as tellurium-atom-bridged metal phosphine compounds.

Experimental

The complex $[Fe(C_5H_5)(CO)_2(thf)]BF_4$[9,22] and the ligands (**4**)[23] were prepared according to published procedures. Reactions were routinely carried out under argon in purified solvents through which argon had been bubbled.

Preparation of $[Fe(C_5H_5)(CO)_2(TePR_3)]BF_4$ [R = Me (**5a**), Pr^i (**5b**), Bu^t (**5c**), NMe_2 (**5d**), or mor (**5e**)].—The appropriate tellurophosphorane (**4**) (4 mmol) was added to a stirred solution of $[Fe(C_5H_5)(CO)_2(thf)]BF_4$ (*ca.* 1 g, 3 mmol) in CH_2Cl_2 (40 cm^3) at room temperature. After 2 h the colour had changed from red to brown and the solution was filtered through alumina (5 cm) to remove tellurium. The filtrate was concentrated *in vacuo* and ether (80 cm^3) was added to precipitate the products. For purification all compounds were recrystallized from dichloromethane–diethyl ether at −78 °C. $[Fe(C_5H_5)(CO)_2(TePMe_3)]BF_4$ (**5a**) (1.1 g, 78%), brown powder (Found: C, 25.75; H, 3.05. $C_{10}H_{14}BF_4FeO_2PTe$ requires C, 25.65; H, 3.00%). $[Fe(C_5H_5)(CO)_2(TePPr^i{}_3)]BF_4$ (**5b**) (1.4 g, 86%), dark brown crystals (Found: C, 34.9; H, 4.70. $C_{16}H_{26}BF_4FeO_2PTe$ requires C, 34.8; H, 4.70%). $[Fe(C_5H_5)(CO)_2(TePBu^t{}_3)]BF_4$ (**5c**) (1.5 g, 83%), dark brown powder (Found: C, 38.5; H, 5.55. $C_{19}H_{32}BF_4FeO_2PTe$ requires C, 38.4; H, 5.40%). $[Fe(C_5H_5)(CO)_2\{TeP(NMe_2)_3\}]BF_4$ (**5d**) (1.5 g, 88%), dark brown crystals (Found: C, 28.25; H, 4.20. $C_{13}H_{23}BF_4FeN_3O_2PTe$ requires C, 28.1; H, 4.15%). $[Fe(C_5H_5)(CO)_2\{TeP(mor)_3\}]BF_4$ (**5e**) (1.3 g, 65%), brown solid (Found: C, 33.7; H, 4.25. $C_{19}H_{29}BF_4FeN_3O_5PTe$ requires C, 33.5; H, 4.25%).

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* The upfield shift observed on methylation of (**4d**) and (**4e**) parallels the results obtained for $XP(NR_2)_3$ (X = O, S, or Se) and is suggested to be a consequence of changed P–N π bonding.[1,19]

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