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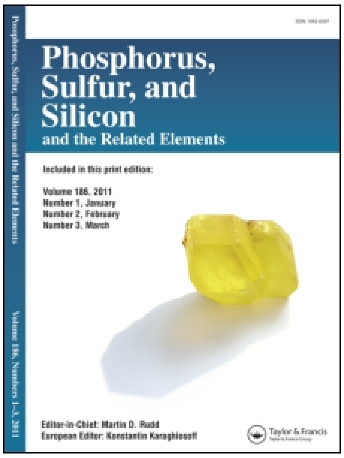

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NOVEL ANIONIC CHALCOGENO LIGANDS. TELLUROPHOSPHINITES R2PTe- AND CHALCOGENOTELLUROPHOSPHINATES $R_2P(Ch)Te$-[Ch = O,S,Se,Te]

Benno Bildstein[a]; Fritz Sladky[a]
[a] Institut für Anorgankche und Analytische Chemie der Universität Innsbruck, Innsbruck, Austria

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NOVEL ANIONIC CHALCOGENO LIGANDS. TELLUROPHOSPHINITES R_2PTe^- AND CHALCOGENOTELLUROPHOSPHINATES $R_2P(Ch)Te^-$ [Ch = O,S,Se,Te]

BENNO BILDSTEIN and FRITZ SLADKY†
Institut für Anorganische und Analytische Chemie der Universität Innsbruck, Innrain 52a, A-6020 Innsbruck, Austria

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Elemental tellurium reacts with diorganylphosphides R_2P^- to give tellurophosphinites R_2PTe^- and with chalcogenophosphinites R_2PCh^- to give chalcogenotellurophosphinates $R_2P(Ch)Te^-$ [$R = C_6H_{11}$, C_6H_5; Ch = O,S,Se,Te], respectively. Borane- or chromium-pentacarbonyl complexed phosphides also react with elemental tellurium to the corresponding tellurophosphinito complexes $R_2P(BH_3)Te^-$ and $R_2P[Cr(CO)_5]Te^-$. The new ligands are characterized by multinuclear NMR spectroscopy. The analogous reaction of elemental tellurium with organylphosphides RP^{2-} ($R = C_6H_5$) does not lead to tellurophosphonites $RPTe_2^{2-}$ or tellurophosphonates $RPTe_3^{2-}$, instead, tellurium couples the phosphide oxidatively to cyclic tetraphosphine.

Key words: Tellurophosphinites; ditellurophosphinates; selenotellurophosphinates; thiotellurophosphinates; tellurophosphinates.

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INTRODUCTION

The chemical and coordinating properties of anionic ligands with phosphorus and chalcogen donor atoms are well developed. Chalcogenophosphito[1,2] $(RO)_2PCh^-$, chalcogenophosphinito[1,2,3] R_2PCh^-, and dichalcogenophosphinato[1,4] $R_2PCh_2^-$ complexes where R = alkyl or aryl and Ch = O,S,Se are known. Surprisingly, the analogous ligands or complexes with tellurium as donor atom are virtually unknown. Potassium diethyltellurophosphinite, reported first by Foss[5] in 1950, was used by Clive and Menchen[6] in 1980 as deoxygenating reagent for epoxides, and no chalcogenotellurophosphinato compounds are yet known. In this paper we report the synthesis and properties of tellurophosphinites R_2PTe^- and chalcogenotellurophosphinates $R_2P(Ch)Te^-$. The coordination chemistry of these anions is under investigation. The first tellurophosphinito complex, $[L]Rh[TeP(C_6H_{11})_2]$[7] ($L = bupp_2ph_4$), was reported only very recently.

In an extension of this work we also attempted to synthesize tellurophosphonites $RPTe_2^{2-}$ and tellurophosphonates $RPTe_3^{2-}$.

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† Author to whom all correspondence should be addressed.

RESULTS AND DISCUSSION

Synthesis

Diorganyl-tellurophosphinites R_2PTe^- (R = phenyl, cyclohexyl) and diorganylchalcogenotellurophosphinates R_2PChTe^- (R = phenyl, cyclohexyl; Ch = oxygen, sulfur, selenium, tellurium). Diorganylphosphides, prepared by metallation of phosphines with butyl lithium in tetrahydrofuran (THF), react with one equivalent of elemental tellurium to tellurophosphinites **1a**, **1b** (Scheme 1). Addition of one further equivalent of tellurium oxidizes **1a**, **1b** to the ditellurophosphinates **5a**, **5b**. Seleno-, thio-, (oxo)phosphinites **2a–4b**, prepared from the corresponding phosphinous acids by lithium–hydrogen exchange with butyl lithium, yield with tellurium "mixed" chalcogenotellurophosphinates **6a–8b**. Phosphinous acids $R_2P(Ch)H$ are prepared by oxidation of phosphines with elemental selenium or sulfur and, in the case of diorganylphosphinoxides, by hydrolysis of chlorophosphines with oxygen-free water. In contrast, elemental tellurium does not oxidize diorganylphosphines.

Tellurophosphinites **1a**, **1b** are also accessible by synproportioning diorganylphosphides with ditellurophosphinates **5a**, **5b** (Equation 1).

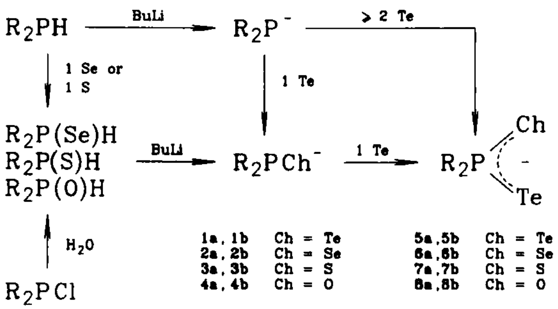
$$R_2P^- + \underset{\textbf{5a, 5b}}{R_2PTe_2^-} \rightarrow \underset{\textbf{1a, 1b}}{2R_2PTe^-} \qquad (1)$$

Selenotellurophosphinates **6a**, **6b** are only obtained as an equilibrium mixture of diselenophosphinates, ditellurophosphinates **5a**, **5b** and selenotellurophosphinates **6a**, **6b** (Equation 2).

$$\underset{\textbf{6a, 6b}}{2R_2PTeSe^-} \rightleftharpoons \underset{\textbf{5a, 5b}}{R_2PTe_2^-} + R_2PSe_2^- \qquad (2)$$

These equilibria are temperature dependent, the asymmetric species being favored at higher temperatures.

The reaction of elemental tellurium with diorganylphosphides works also for



SCHEME 1 Synthesis of tellurophosphinites **1a**, **1b** and chalcogenotellurophosphinates **5a–8b** (Series **a**: R = cyclohexyl; Series **b**: R = phenyl).

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phosphides which engage their free electron pair in complex formation with a metal carbonyl moiety or a Lewis acid (Equations 3,4).

$$\underset{\mathbf{9}}{[(CO)_5Cr]PR_2H} \xrightarrow{BuLi} [(CO)_5Cr]PR_2^- \xrightarrow{Te} \underset{\mathbf{10}}{[(CO)_5Cr]PR_2Te^-} \quad (R = C_6H_5) \tag{3}$$

$$\underset{\mathbf{11}}{(BH_3)PR_2H} \xrightarrow{BuLi} (H_3B)PR_2^- \xrightarrow{Te} \underset{\mathbf{12}}{(H_3B)PR_2Te^-} \quad (R = {}^tBu) \tag{4}$$

Properties

Tellurophosphinites **1a**, **1b**, **10**, **12** and chalcogenotellurophosphinates **5a–8b** are soluble in oxygen-free solvents like THF, Et_2O, CH_3CN, CH_3OH and H_2O. The yellow to red air-sensitive solutions are stable at ambient temperature if kept in the dark under argon. Exposure to light deposits tellurium mirrors on the wall of the Schlenk tubes, whereas on contact with air, the compounds immediately are oxidized and precipitate amorphous tellurium. The crude solid compounds can be isolated as white to yellow air-sensitive powders by removing the solvent *in vacuo*. Redissolution of these powders yields solutions with identical spectral parameters.

Multinuclear NMR analysis (^{31}P, ^{125}Te, ^{77}Se, ^{11}B) is used to characterize the tellurophosphinites **1a**, **1b**, **10**, **12** and chalcogenotellurophosphinates **5a–8b** (Table I). The NMR measurements were performed at low temperatures to avoid

TABLE I

NMR data of tellurophosphinites and chalcogenotellurophosphinates[a]

Compound[b]	$\delta(^{31}P)$	$\delta(^{125}Te)$	$^1J_{Te-P}$	Temp.	Conc.
1a R_2PTe^-	−6.0	−931.6	565	−60	0.40
1b R_2PTe^-	−35.7	−478.2	775	+20	0.28
5a $R_2PTe_2^-$	−70.0	−338.6	1339	−30	0.34
6a R_2PSeTe^-	−4.2	−452.6	1354	−10	[c]
7a R_2PSTe^-	+18.4	−516.0	1417	−30	0.33
8a R_2POTe^-	+52.9	−752.1	1618	−30	0.25
5b $R_2PTe_2^-$	−125.9	+63.3	1442	−20	0.40
6b R_2PSeTe^-	−41.9	−73.4	1511	−60	[d]
7b R_2PSTe^-	−18.1	−141.7	1556	−30	0.14
8b R_2POTe^-	+18.3	−420.4	1690	−40	[e]
10 $R_2P[Cr(CO)_5]Te^-$	−36.0	+15.0	1147	−40	0.16
12 $R_2P[BH_3]Te^-$	+31.2	−407.1	1175	+20	0.90[f]

[a] Chem. shifts rel. $(CH_3)_2Te[^{125}Te]$, $(CH_3)_2Se[^{77}Se]$, 85% $H_3PO_4[^{31}P]$, $BF_3Et_2O[^{11}B]$; coupling constants in Hz, temp. in °C, conc. in mol/l, solvent THF.

[b] **1a**, **5a–8a**: R = cyclohexyl; **1b**, **5b–8b**, **10**: R = phenyl; **12**: R = ^{t}Butyl.

[c] conc. ≤ 0.19 mol/l, mixture of **6a**, **5a**, and $R_2PSe_2^-$; $\delta(^{77}Se)$ not observed (dec.), $^1J(^{77}Se-^{31}P) = 547$ Hz.

[d] conc. ≤ 0.39 mol/l. mixture of **6b**, **5b** and $R_2PSe_2^-$; $\delta(^{77}Se) = -4.0$ ppm, $^1J(^{77}Se-^{31}P) = 641$ Hz.

[e] **8b** is only slightly soluble in THF, solvent CH_3CN.

[f] $\delta(^{11}B) = -34.0$ ppm, $^1J(^{31}P-^{11}B) = 70$ Hz.

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decomposition during accumulation of data and to minimize possible exchange processes (chalcogen transfer) which could lead to line broadening or disappearing of satellite signals.

The highly shielded tellurium of tellurophosphinites **1a**, **1b** reflects the transfer of a substantial amount of negative charge to the tellurium. The magnitude of $^{1}J(^{125}Te–^{31}P)$ of tellurophosphinites **1a**, **1b** is comparable to values obtained for $^{t}Bu_2P–Te–P^{t}Bu_2$ (451 Hz)[8] and $(pCH_3–C_6H_4)_2P–Te–CH(CH_3)_2$ (565 Hz),[9] indicating a formal bond order close to one.

Diorganylphosphinites R_2PCh^- (R = cyclohexyl, phenyl; Ch = O,S,Se,Te) are members of an isoelectronic series whose ^{31}P-NMR shifts correlate linearly with the Pauling electronegativity of the chalcogen bonded to phosphorus (Table II Equations 5,6):

$$\delta(^{31}P)[(C_6H_{11})_2PChLi] = 81.3\,EN - 170.1 \quad (r = 0.993) \tag{5}$$

$$\delta(^{31}P)[(C_6H_5)_2PChLi] = 83.5\,EN - 199.1 \quad (r = 0.981) \tag{6}$$

In comparison to tellurophosphinites **1a**, **1b** the ^{125}Te-NMR resonances of chalcogenotellurophosphinates **5a–8b** are shifted downfield because the negative charge is delocalized.

The ^{31}P and ^{125}Te shifts correlate linearly with the Pauling electronegativity of the second chalcogen O,S,Se,Te, reflecting the increasing ability of withdrawal of charge (Equations 7–10).

$$\delta(^{125}Te)[(C_6H_{11})_2PChTeLi] = -283.5\,EN + 229.4 \quad r = 0.988) \tag{7}$$

$$\delta(^{31}P)[(C_6H_{11})_2PChTeLi] = 73.8\,EN - 194.3 \quad (r = 0.865) \tag{8}$$

$$\delta(^{125}Te)[(C_6H_5)_2PChTeLi] = -331.2\,EN + 726.5 \quad (r = 0.989) \tag{9}$$

$$\delta(^{31}P)[(C_6H_5)_2PChTeLi] = 84.7\,EN - 264.2 \quad (r = 0.840) \tag{10}$$

$$^{1}J(^{125}Te–^{31}P)[(C_6H_{11})_2PChTeLi] = -0.716\,\delta(^{125}Te) + 1063.2 \quad (r = 0.972) \tag{11}$$

$$^{1}J(^{125}Te–^{31}P)[(C_6H_5)_2PChTeLi] = -0.513\,\delta(^{125}Te) + 1476.3 \quad (r = 0.999) \tag{12}$$

The coupling constants $^{1}J(^{125}Te–^{31}P)$ of chalcogenotellurophosphinates **5a–8a**, **5b–8b** also give a linear correlation with the ^{125}Te shifts (Equations 11,12) and are comparable to values obtained for triorganylphosphine tellurides (e.g. $^{t}Bu_3P{=}Te$ $^{1}J(^{125}Te–^{31}P) = 1732$ Hz).[12]

The coordination of the tellurophosphinito ligand to a metal carbonyl moiety (compare **1b** to **10**) shifts the ^{125}Te resonance by 500 ppm downfield whereas the

TABLE II

Correlation of ^{31}P-NMR shifts[a] of chalcogenophosphinites R_2PCh^- with the Pauling electronegativity of Ch

Ch	EN	$\delta(^{31}P)$[**8a–5a**]	$\delta(^{31}P)$[**8b–5b**]
O	3.5	+111.9	+88.9 (ref. 10)
S	2.5	+40.3	+20.7 (ref. 11)
Se	2.4	+26.7	+6.5
Te	2.1	−6.0	−35.7

[a] Chem. shifts rel. 85% H_3PO_4.

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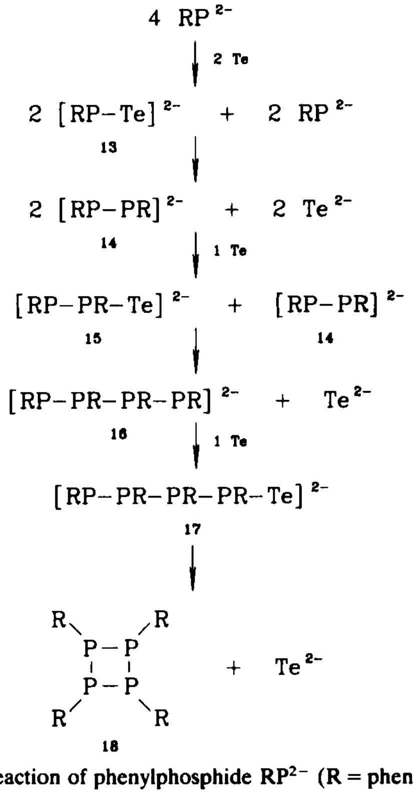
^{31}P resonance remains unaffected, suggesting a terminal coordination mode. The coupling constant of complex **10** $^{1}J(^{125}\text{Te}-^{31}\text{P}) = 1147$ Hz) is similar to the value obtained for the complex $[\text{L}]\text{Rh}[\text{TeP}(\text{C}_6\text{H}_{11})_2]$ ($^{1}J(^{125}\text{Te}-^{31}\text{P} = 1150$ Hz, L = bupp_2ph_4);[7] unfortunately a closer comparison is hampered by the lack of ^{125}Te-NMR data.

Reaction of elemental Tellurium with dipotassium phenylphosphide

The successful synthesis of tellurophosphinites **1a**, **1b** and chalcogenotellurophosphinates **5a–5b** by reaction of elemental tellurium with diorganylphosphides or chalcogenophosphinites prompted us to investigate the analogous reaction of tellurium with organylphosphides RP^{2-} in the hope of getting access to the so far unknown ditellurophosphonites RPTe_2^{2-} or tritellurophosphonates RPTe_3^{2-}. Although tellurium does react with dipotassium phenylphosphide, no tellurophosphonites or tellurophosphonates can be detected by monitoring the reaction mixture with ^{31}P-NMR spectroscopy. Instead, tellurium oxidizes phenylphosphide yielding in a stepwise phosphorus–phosphorus coupling reaction cyclic phenylpolyphosphine **18** ($\delta(^{31}\text{P}) = -7.8$ ppm) and a precipitate of potassium telluride (Scheme 2, Equation (13)).

$$4(\text{C}_6\text{H}_5)\text{PK}_2 + 4\text{Te} \rightarrow \underset{\textbf{18}}{[(\text{C}_6\text{H}_5)\text{P}]_4} + 4\text{K}_2\text{Te} \qquad (13)$$



SCHEME 2 Reaction of phenylphosphide RP^{2-} (R = phenyl) with tellurium.

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The reaction sequence depicted in Scheme 2 is based on the ^{31}P-NMR spectroscopic detection of some of the intermediate coupled products **13–17**. If the reaction is run with a stochiometric ratio RPK_2/Te less than one and if the reaction temperature is kept at low temperatures (−75°C), the intermediate tetraphenyltetraphosphide **16** is identified by its AA′BB′ spin system, the spectral parameters (RP_A–RP_B–$RP_{B'}$–$RP_{A'}$ = δ_A − 74.2 ppm, $\delta_B = -28.2$ ppm, $^1J_{AB} =$ 333.1, $^1J_{BB'} = 266.5$, $^2J_{A'B} = 2.5$, $^3J_{AA'} = 120$ Hz)[13] agree with literature values.[14] The tellurium containing (poly)-phosphides **13**, **15**, **17** give rise to spin systems of higher order with tellurium satellites; due to the transient nature of these species no reproduceable spectra can be obtained.

EXPERIMENTAL

NMR measurements were obtained on a Bruker WP 80 equipped with a variable temperature probe. Chemical shifts refer to external standards (^{31}P: 85% H_3PO_4, ^{125}Te: $(CH_3)_2Te$, ^{77}Se: $(CH_3)_2Se$, ^{11}B: $BF_3.Et_2O$).

Successful results necessitate utilization of conventional organometallic chemical methods (i.e. purified argon atmosphere, Schlenk techniques, purified and deoxygenated solvents). Chemicals were either obtained commercially or prepared according to literature procedures: dicyclohexylphosphine-oxide,[15] -sulfide,[16] diphenylphosphine-oxide,[15] -sulfide,[17] diphenylphosphine chromium pentacarbonyl,[18] dipotassium phenylphosphide tetrahydrofuranate.[19]

R_2PTeLi **1a** *($R = C_6H_{11}$)*, **1b** *($R = C_6H_5$)*. In a representative reaction, 1 ml dicyclohexylphosphine (4.56 mmol) was dissolved in 7 ml THF and cooled to −80°C. After addition of 3.28 ml (4.56 mmol) ^{n}butyl lithium (1.4 molar solution in hexane) the mixture was stirred for 3 hours and allowed to warm to room temperature. 1 ml of this yellow solution was transferred to a NMR tube. A ^{31}P-NMR spectrum showed quantitative conversion to the lithium salt R_2PLi [$\delta(^{31}P) = -19.7$ ppm ($R = C_6H_{11}$); δ (^{31}P) = −23.0 ppm ($R = C_6H_5$)]. To the remaining solution (c = 0.4 mol/l, 10.27 ml) one equivalent tellurium powder (0.524 g, 4.11 mmol) was added and the mixture stirred until all tellurium had dissolved. NMR analysis of this solution showed complete conversion to **1a** (**1b**).

R_2PTe_2Li **5a** *($R = C_6H_{11}$)*, **5b** *($R = C_6H_5$)*. The reaction was run in an analogous manner as described for **1a** except that 2 equivalents or an excess of tellurium were used.

$R_2PSeTeLi$ **6a** *($R = C_6H_{11}$)*, **6b** *($R = C_6H_5$)*. 0.5 ml dicyclohexylphosphine (2.28 mmol) was dissolved in 5 ml THF, 18.1 mg selenium powder (2.3 mmol) were added and this mixture was stirred at room temperature until all selenium had dissolved. 1 ml of this solution was checked by ^{31}P-NMR: $(C_6H_{11})_2P(Se)H$ $\delta(^{31}P) = 33.3$ ppm, $^1J(^{31}P–^1H) = 418$ Hz (d), $^1J(^{77}Se–^{31}P) = 725$ Hz; $(C_6H_5)_2P(Se)H$ $\delta(^{31}P) = 3.6$ ppm, $^1J(^{31}P–^1H) = 470$ Hz (d), $^1J(^{77}Se–^{31}P) = 778$ Hz. The remaining solution was cooled to −80°C, metallated by butyl lithium (1.14 ml, 1.8 mmol, 1.6 molar solution in hexane) to the selenophosphinite **2a** ($\delta(^{31}P) = 26.7$ ppm) respectively **2b** ($\delta(^{31}P) = 6.0$ ppm, $^1J(^{77}Se–^{31}P) = 344$ Hz), and oxidized at room temperature with tellurium (0.250 g, 2 mmol), giving a dark orange solution which was shown by NMR to consist of a mixture of diselenophosphinate, ditellurophosphinates **5a** (resp. **5b**) and selenotellurophosphinates **6a** (resp. **6b**).

$R_2PChTeLi$ **7a** *($R = C_6H_{11}$, $Ch = S$)*, **7b** *($R = C_6H_5$, $Ch = S$)*, **8a** *($R = C_6H_{11}$, $Ch = O$)*, **8b** *($R = C_6H_5$, $Ch = O$)*. The preparations were performed analogously as described for **6a**, **6b**, except that the chalcogenotellurophosphinous acids were isolated. ^{31}P-NMR data of chalcogenophosphinites **3a–4b**: **3a** $\delta(^{31}P) = 40.3$ ppm, **3b** $\delta(^{31}P) = 21.7$ ppm, **4a** $\delta(^{31}P) = 109.7$ ppm, **4b** $\delta(^{31}P) = 87.7$ ppm. After reaction with tellurium the colored THF solutions were analyzed by NMR. **8b** is only slightly soluble in THF; CH_3CN was used as solvent.

$[(CO)_5Cr]P(C_6H_5)_2TeLi$ **10**. 0.270 g (0.7 mmol) $[(CO)_5Cr]P(C_6H_5)_2H$ in 4 ml THF was cooled to −80°C, was lithiated with 0.44 ml BuLi (0.7 mmol) and was stirred for 2 hours at −70°C. ^{31}P-NMR showed complete conversion to $[(CO)_5Cr]P(C_6H_5)_2Li$ $\delta(^{31}P) = -14.0$ ppm). To this solution (3.44 ml, c = 0.158 mol/l, 0.54 mmol) 0.130 g tellurium (1 mmol) was added and the mixture was allowed to warm to room temperature during 2 hours, yielding a red solution of **10** and excess, undissolved tellurium.

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$(H_3B)P^tBu_2H$ **11** *was prepared from ${}^tBuPCl$ and $LiBH_4$ analogously to $(H_3B)P(C_6H_5)_2H$*[20]. Yield: 67%, colorless air sensitive solid, b.p. 113°C/10 torr. ^{1}H-NMR: $\delta = 1.4$ ppm (d), ${}^3J({}^{31}P-{}^1H) = 13$ Hz $(H_3B)P^t\underline{Bu_2}H$; $\delta = 4.08$ ppm (dq), ${}^1J({}^{31}P-{}^1H) = 352$ Hz (d), ${}^3J({}^1H-{}^1H) = 6$ Hz (q) $(H_3B)P^tBu_2\underline{H}$; $\delta = 0.52$ ppm (dq), ${}^3J({}^1H-{}^1H) = 6.0$ Hz (d), ${}^1J({}^{11}B-{}^1H) = 100$ Hz (q) $(\underline{H_3B})P^tBu_2H$; ^{31}P-NMR: $\delta = 45.5$ ppm (dq), ${}^1J({}^{31}P-{}^1H) = 352$ Hz (d), ${}^1J({}^{31}P-{}^{11}B) = 45$ Hz (q); ^{11}B-NMR: $\delta = -42.8$ ppm (dq), ${}^1J({}^{31}P-{}^{11}B) = 45$ Hz (d), ${}^1J({}^{11}B-{}^1H) = 100$ Hz (q).

$(H_3B)P^tBu_2TeLi$ **12**. 0.440 g (2.75 mmol) **11** was lithiated at −30°C in 2 ml THF with 3.44 ml BuLi (2.75 mmol, 0.8 molar solution in pentane), yielding a white precipitate. Solvents were removed *in vacuo*, the lithium salt was redissolved in 3 ml THF [0.9 molar solution; ^{31}P-NMR: $\delta = 7.0$ ppm (q), ${}^1J({}^{31}P-{}^{11}B) = 39$ Hz; ^{11}B-NMR: $\delta = -37.0$ ppm (dq), ${}^1J({}^{31}P-{}^{11}B) = 37$ Hz (d), ${}^1J({}^{11}B-{}^1H) = 83$ Hz (q)], and excess of tellurium powder was added. The mixture was stirred at room temperature for 2 hours yielding a red solution of **12** and excess, undissolved tellurium powder.

Reaction of $(C_6H_5)PK_2$ with Te. 0.210 g (0.86 mmol) $(C_6H_5)PK_2$–(THF) was stirred with 0.110 g (0.86 mmol) tellurium powder at ambient temperature. The reaction started immediately; the mixture turned slightly warm and the color of the solution changed to red; grey potassium telluride precipitated. After 1 hour the phosphide was completely oxidized to cyclic tetraphenyltetraphosphine $(\delta({}^{31}P)) = -7.8$ ppm).

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