stirred for approximately 1 h (at −78 °C), the mixture was orange. At this time the selected ligand was added (2.0 mmol) and the resulting mixture was allowed to warm slowly to room temperature with stirring. The mixture was filtered, and the resulting filtrate was concentrated in vacuo to leave the crude product complex as a burgundy solid ($PPh_3$ and dppe adducts) or a yellow oil (tmeda adduct). These were washed with several portions of dry benzene to remove any excess ligand present and dried under vacuum (40–50% yield). Attempts to obtain analytically pure samples of the $PPh_3$ and tmeda adducts by recrystallization or chromatography (decomposition on alumina, silica gel, Florisil) were unsuccessful. Spectral properties of these products follow.

**OMo(mnt)($PPh_3$)$_2$·THF (1a)**: IR (KBr) 2200 ($\nu$(CN)), 960 ($\nu$(MoO)) cm$^{-1}$; UV/vis (THF) 500, 400, 320, 250 nm; $^1$H NMR (acetone-$d_6$) $\delta$ 7.0–8.0 (m, $C_6H_5$), 3.6 (t, $OCH_2$), 1.8 (m, $CH_2CH_2$).

**OMo(mnt)(dppe)·THF (1b)**: IR (KBr) 2200 ($\nu$(CN)), 970 ($\nu$(MoO)) cm$^{-1}$; UV/vis (THF) 530, 450, 400, 315, 250 nm,; $^1$H NMR (acetone-$d_6$) $\delta$ 7.2–8.0 (m, $C_6H_5$), 3.6 (t, $OCH_2$), 3.4 (t, $PCH_2$), 3.05 (m, $PCH_2'$), 1.8 (m, $CH_2CH_2$).

**OMo(mnt)(tmeda) (1c)**: IR (KBr) 2200 ($\nu$(CN)), 925 ($\nu$(MoO)) cm$^{-1}$; UV/vis (THF) 425, 360, 280, 250 nm; $^1$H NMR (acetone-$d_6$) $\delta$ 3.1 (s, 4 H, $NCH_2$), 2.6 (s, 6 H, $NCH_3$); $^{13}$C NMR (acetone-$d_6$) $\delta$ 53.9, 43.9.

**Crystal Structure of OMo(mnt)(dppe)·$(CH_3)_2CO$ (2b).** Deep red crystals of **2b** were obtained when **1b** was dissolved in acetone and let stand overnight under $N_2$. The crystal selected was mounted on a glass fiber, and the data were collected on an Enraf-Nonius CAD-4 diffractometer by the methods standard in this laboratory.[16] The data were corrected for Lorentz and polarization effects; no absorption correction was applied since it was judged to be negligible. The structure was solved by the heavy-atom method and refined by least squares (SHELX-76)[17] minimizing $\sum w(|F_o| - |F_c|)^2$. Positional and anisotropic thermal parameters were refined for all the non-hydrogen atoms in two blocks. Only the methyl hydrogen atoms were refined isotropically, while all the other hydrogen atoms were included in the idealized positions (C–H = 0.95 Å).

The atomic scattering factors were taken from ref 18. Data pertaining to data collection and refinement are summarized in Table I. Table II lists the atomic coordinates, and selected bond lengths and angles are given in Table III.

**Supplementary Material Available:** Listings of hydrogen atom coordinates, anisotropic thermal parameters, and supplementary bond lengths and angles (6 pages); a table of calculated and observed structure factors (20 pages). Ordering information is given on any current masthead page.

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# Ditellurene, Selenatellurene, and Thiatellurene Complexes. The Structure of Pt(1,2-$Te_2C_6H_4$)($PPh_3$)$_2$

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Metal complexes formally derived from alkenedichalcogenides or 1,2-benzenedichalcogenides have been heavily studied for more than 20 years.[1] Best known of these ligand types are the sulfur derivatives (dithiolenes) although recent work has focused on the selenium analogues (diselenenes).[2] Interest in this class of chelating ligands continues to grow, particularly with regard to their applications in materials science.[3,4] Large and highly polarizable main-group centers in alkenedichalcogenide complexes should enhance the intermolecular electronic coupling leading to the formation of conduction bands. It is the last property in particular which points to the desirability of complexes of 1,2-alkeneditellurolate (1,2-alkeneditelluride, ditellurene) ligands.

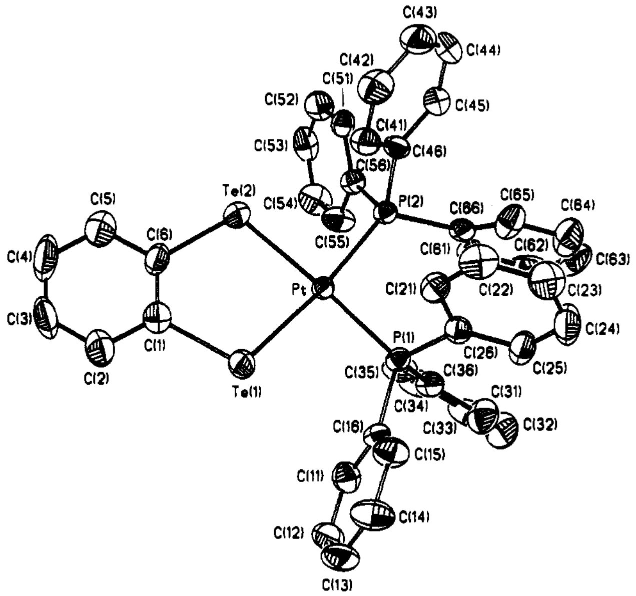
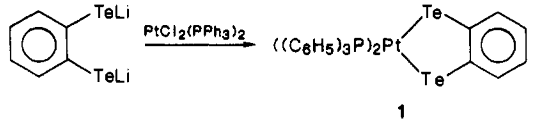


Figure 1. ORTEP plot of Pt($Te_2C_6H_4$)($PPh_3$)$_2$ (**1**).

Research on tetrathia- and tetraselenafulvalenes[5] has evolved in parallel with work on metal dithiolenes and diselenenes. Recent work has resulted in the syntheses of the first examples of tetratellurafulvalenes.[6,7] This paper describes the extension of these synthetic advances leading to the preparation of transition-metal ditellurene complexes.

## Results and Discussion

Solutions of dilithium 1,2-benzeneditelluride, prepared in two steps from the phenylenemercury hexamer[8] according to Cowan,[7] were found to react at room temperature with *cis*-$PtCl_2(PPh_3)_2$ to afford dark red-orange solutions from which orange air-stable crystals of Pt($Te_2C_6H_4$)($PPh_3$)$_2$ (**1**) could be isolated. Compound **1** was characterized by mass spectrometry as well as $^1$H and $^{31}$P



(1)

**1**

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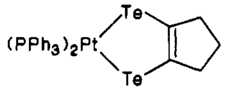
**Figure 2.** 200-MHz $^1$H NMR spectrum of Pt(STeC$_6$H$_4$)(PPh$_3$)$_2$. The intense signal at ~7.3 ppm is due to CHCl$_3$.

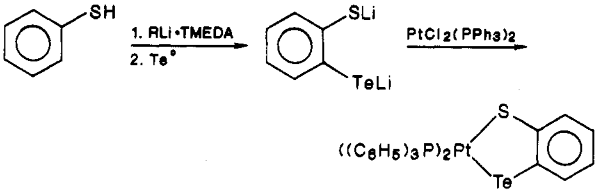
NMR spectroscopy. The $^1$H NMR spectrum of **1** shows well-defined multiplet on the high-field side of the complex phenyl manifold. Decoupling experiments indicate that this multiplet is half of an AA'BB' spin system that we attribute to the ortho protons on the Te$_2$C$_6$H$_4$ group. In a similar manner we found that solutions of 1,2-C$_5$H$_6$(TeLi)$_2$[6] when treated with *cis*-PtCl$_2$(PPh$_3$)$_2$ gave Pt(1,2-Te$_2$C$_5$H$_6$)(PPh$_3$)$_2$. In terms of its physical properties, this new compound resembles **1**.



**2**

The structure of **1** was established crystallographically. It consists of a distorted square-planar PtTe$_2$P$_2$ core (Figure 1). The environment about the platinum center resembles that found in (Ph$_3$P)$_2$PtSe$_2$Fe$_2$(CO)$_6$.[9] The PtTe$_2$P$_2$C$_6$ core is essentially planar: The maximum deviation of the atoms Pt, Te(1), Te(2), and C(1)–C(6) from planarity is 0.03 Å. The Pt–Te distances appear normal although few other complexes containing Te–Pt bonds have been characterized crystallographically.[10] In [Pt(*o*-PhTeC$_6$H$_4$PPh$_2$)$_2$]$^{2+}$, the Pt–Te distance is 2.575 Å.[11]

In an attempt to extend the range of tellurene ligands, we developed a synthetic approach to the related S–Te chelating ligands. PhSLi is known to undergo ortho metalation with butyllithium.[12] The resulting 1,2-C$_6$H$_4$SLi$_2$ is a very promising ligand precursor and is already known to be reactive toward a range of simple electrophiles.[12] We found that hexane slurries of 1,2-C$_6$H$_4$SLi$_2$ take up elemental tellurium to give an orange suspension that when treated with PtCl$_2$(PPh$_3$)$_2$ gave yellow crystals of Pt(STeC$_6$H$_4$)(PPh$_3$)$_2$ in 20% overall yield (eq 2). Microanalysis and mass spectrometry confirm the stoichiometry while the $^1$H (Figure 2) and $^{31}$P NMR results support the unsymmetrical cis structure. Addition of *cis*-PtCl$_2$(PPh$_3$)$_2$ to a solution of 1,2-C$_6$H$_4$SeTeLi$_2$, prepared in situ from Ph$_2$Se$_2$, BuLi (2 equiv), and Te, gave a mixture of products, the major component of which is Pt(SeTeC$_6$H$_4$)(PPh$_3$)$_2$. This orange crystalline complex was characterized by $^{31}$P NMR spectroscopy and fast atom bombardment mass spectrometry.



(2)

Metal complexes of each member of the 1,2-C$_6$H$_4$E$_2$ series (E = S, Se, or Te[13]) are now known.

## Experimental Section

Purification procedures and spectroscopic methods have been described previously.[14] Reagents were obtained commercially and were used without purification.

**Pt(Te$_2$C$_6$H$_4$)(PPh$_3$)$_2$.** A slurry of 0.414 g (1.5 mmol) of (HgC$_6$H$_4$)$_6$[10] and 0.080 g of 30% (w/w) Li in mineral oil in 20 mL of ether was stirred at 25 °C for 3 days, giving a red solution and gray solids. The solution was filtered into a slurry of 0.384 (3.0 mmol) of Te powder in 40 mL of THF at −20 °C. The mixture was stirred at 25 °C for 2 days, giving a red-orange slurry, to which was added a slurry of 1.2 g (1.5 mmol) of *cis*-PtCl$_2$(PPh$_3$)$_2$ in 40 mL of THF. The red-orange slurry was stirred overnight (14 h), and 2 g of silica gel was added. Volatiles were removed in vacuo, and the orange solid was chromatographed on a 4 × 2 cm column of silica gel. The fast-moving orange band was collected, diluted with hexanes, and concentrated to crystallization. Filtration and recrystallization from CH$_2$Cl$_2$/hexanes gave 0.325 g (0.31 mmol, 21%) of orange-brown crystals. Anal. C, H, N, P, Pt, Te. Fast atom bombardment mass spectrum (ion, % relative intensity): *m/z* 1052 (M$^+$, 40), 790 (M$^+$ − PPh$_3$, 22), 718 (M$^+$ − Te$_2$C$_6$H$_4$, 60). $^{31}$P{$^1$H} NMR (positive shifts are downfield of 85% H$_3$PO$_4$): 17.6 ppm ($J$(Pt,P) = 2990 Hz). The analogous C$_6$H$_4$S$_2$ complex was prepared in 94% yield from 1,2-C$_6$H$_4$(SH)$_2$, Et$_3$N, and *cis*-PtCl$_2$(PPh$_3$)$_2$. This yellow compound exhibits NMR spectroscopic characteristics similar to those for the C$_6$H$_4$Te$_2$ complex.

**Pt(Te$_2$C$_5$H$_6$)(PPh$_3$)$_2$.** At −78 °C, 0.55 mL of 1.8 M *t*-BuLi was added to 0.063 mL (0.5 mmol) of 1,2-dibromocyclopentene. After 5 min 0.064 g (0.5 mmol) of Te was added to the pale yellow solution. The murky grey slurry was allowed to slowly warm, and when the slurry cleared, the mixture was recooled to −78 °C. The reaction with *t*-BuLi/Te was repeated. Then 0.3909 g (0.5 mmol) of PtCl$_2$(PPh$_3$)$_2$ was added and the slurry allowed to warm to room temperature. The red-orange solution was worked up as described above and washed with ether until the washings came through clear. Recrystallization from CH$_2$Cl$_2$/hexane afforded 0.026 g (0.025, 5%) of orange solid. Anal. C, H, Pt, Te. FABMS: *m/z* 1041 (M$^+$, 50), 780 (M$^+$ − PPh$_3$, 35), 718 (M$^+$ − Te$_2$C$_5$H$_6$, 25). $^{31}$P{$^1$H} NMR: 14.8 ppm ($J$(Pt,P) = 2860 Hz).

**Pt(STeC$_6$H$_4$)(PPh$_3$)$_2$.** At 0 °C, 0.105 mL (1 mmol) of benzenethiol was added to a solution consisting of 3.3 mL of 1.4 M *n*-BuLi and 0.34 mL of tetramethylethylenediamine in 10 mL of hexane. After 1 h, the solution was allowed to warm to room temperature. After 12 h, 0.128 g (1 mmol) of Te was added to the cream-colored slurry. When the Te had completely dissolved (ca. 5 h), a solution of 0.79 g (1 mmol) of PtCl$_2$(PPh$_3$)$_2$ in 50 mL of THF was added. Workup was conducted as above to give 0.192 g (0.2 mmol, 20%) of yellow-brown crystals. Anal. C, H, P, S. FABMS: *m/z* 957 (M$^+$, 42), 880 (M$^+$ − C$_6$H$_4$, 8), 847 (M$^+$ − SC$_6$H$_4$, 10), 718 (M$^+$ − STeC$_6$H$_4$, 22), 695 (M$^+$ − PPh$_3$, 43). $^{31}$P{$^1$H} NMR: 20.49 (J(Pt,P) = 2857 Hz, J(P,P) = 27 Hz), 15.71 ppm ($J$(Pt,P) = 2968 Hz).

**Pt(SeTeC$_6$H$_4$)(PPh$_3$)$_2$.** At 0 °C, 0.312 g (1 mmol) of Ph$_2$Se$_2$ was added to a solution of 1.4 mL of 1.6 M *n*-BuLi and 0.400 mL of tetramethylenediamine in 20 mL of hexanes. After 1 h the solution was allowed to warm to room temperature to afford a pale orange slurry. After 24 h, 0.128 g (1 mmol) of Te was added. Within 5 h all of the Te had dissolved, and 0.68 g (0.86 mmol) of PtCl$_2$(PPh$_3$)$_2$ in 20 mL of THF was added. Workup was conducted as above to give 0.115 g (0.11, 14%) of yellow-brown microcrystals. $^{31}$P{$^1$H} NMR: 19.35 ($J$(Pt,P) = 2881 Hz, $J$(P,P) = 26 Hz), 15.35 ppm ($J$(Pt,P) = 3024 Hz, $J$(P,P) = 26 Hz). FABMS: *m/z* 1003 (M$^+$, 100), 730 (M$^+$ − PPh$_3$, 35) 718 (M$^+$ − SeTeC$_6$H$_4$, 25).

**X-ray Crystallography.** A greenish yellow crystal (0.15 × 0.24 × 0.30 mm) of Pt(Te$_2$C$_6$H$_4$)(PPh$_3$)$_2$ was grown by diffusion of hexanes into a

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**Table I.** Atomic Coordinates ($\times 10^4$) and Isotropic Thermal Parameters ($Å^2 \times 10^3$) for **1**[a]

| | *x* | *y* | *z* | $U^a$ |
|---|---|---|---|---|
| Pt | 1732 (1) | 1122 (1) | 2032 (1) | 32 (1) |
| Te(1) | 459 (1) | 1270 (1) | 3152 (1) | 52 (1) |
| Te(2) | 302 (1) | 1705 (1) | 742 (1) | 56 (1) |
| P(1) | 2915 (2) | 561 (1) | 3191 (2) | 35 (1) |
| P(2) | 2799 (2) | 1133 (1) | 955 (2) | 35 (1) |
| C(1) | −893 (8) | 1761 (5) | 2341 (7) | 53 (4) |
| C(2) | −1768 (10) | 1908 (7) | 2741 (9) | 80 (6) |
| C(3) | −2627 (10) | 2230 (7) | 2223 (9) | 84 (6) |
| C(4) | −2668 (11) | 2446 (8) | 1326 (12) | 102 (7) |
| C(5) | −1835 (10) | 2304 (6) | 910 (10) | 79 (6) |
| C(6) | −926 (8) | 1950 (5) | 1433 (8) | 56 (4) |
| C(11) | 1788 (5) | −16 (3) | 4408 (4) | 54 (4) |
| C(12) | 1492 | −98 | 5257 | 64 (5) |
| C(13) | 1969 | 290 | 6026 | 78 (6) |
| C(14) | 2743 | 761 | 5946 | 77 (6) |
| C(15) | 3039 | 843 | 5098 | 57 (4) |
| C(16) | 2561 | 455 | 4329 | 38 (3) |
| C(21) | 4084 (4) | 1699 (3) | 3438 (5) | 47 (4) |
| C(22) | 4987 | 2101 | 3729 | 64 (5) |
| C(23) | 5989 | 1817 | 4104 | 70 (5) |
| C(24) | 6087 | 1129 | 4187 | 75 (5) |
| C(25) | 5184 | 727 | 3895 | 66 (5) |
| C(26) | 4183 | 1012 | 3521 | 43 (4) |
| C(31) | 3921 (5) | −708 (4) | 3370 (5) | 72 (5) |
| C(32) | 4015 | −1360 | 3084 | 86 (7) |
| C(33) | 3279 | −1610 | 2308 | 89 (7) |
| C(34) | 2450 | −1209 | 1818 | 81 (6) |
| C(35) | 2356 | −557 | 2104 | 60 (5) |
| C(36) | 3091 | −306 | 2880 | 44 (4) |
| C(41) | 3124 (5) | 2496 (3) | 1295 (4) | 46 (4) |
| C(42) | 3591 | 3109 | 1196 | 65 (5) |
| C(43) | 4312 | 3170 | 619 | 71 (5) |
| C(44) | 4567 | 2617 | 142 | 64 (5) |
| C(45) | 4100 | 2004 | 241 | 52 (4) |
| C(46) | 3379 | 1944 | 818 | 40 (3) |
| C(51) | 1947 (5) | 1334 (3) | −988 (4) | 43 (3) |
| C(52) | 1305 | 1160 | −1858 | 62 (5) |
| C(53) | 714 | 573 | −1959 | 66 (5) |
| C(54) | 766 | 158 | −1190 | 72 (5) |
| C(55) | 1408 | 332 | −320 | 61 (4) |
| C(56) | 1999 | 920 | −220 | 40 (3) |
| C(61) | 3908 (6) | −58 (3) | 832 (5) | 63 (5) |
| C(62) | 4812 | −467 | 1002 | 91 (7) |
| C(63) | 5807 | −221 | 1477 | 93 (7) |
| C(64) | 5900 | 463 | 1783 | 88 (6) |
| C(65) | 4996 | 846 | 1614 | 64 (5) |
| C(66) | 4000 | 599 | 1138 | 46 (4) |

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

**Table II.** Selected Bond Distances (Å) and Angles (deg) for **1**

| | | | |
|---|---|---|---|
| Distances | | | |
| Pt–Te(1) | 2.586 (1) | Pt–Te(2) | 2.592 (1) |
| Pt–P(1) | 2.298 (2) | Pt–P(2) | 2.317 (3) |
| Te(1)–C(1) | 2.106 (10) | Te(2)–C(6) | 2.117 (12) |
| C(1)–C(2) | 1.411 (18) | C(1)–C(6) | 1.379 (16) |
| C(2)–C(3) | 1.350 (17) | C(3)–C(4) | 1.377 (23) |
| C(4)–C(5) | 1.375 (22) | C(5)–C(6) | 1.427 (16) |
| Angles | | | |
| Te(1)–Pt–Te(2) | 88.4 (1) | P(1)–Pt–P(2) | 97 (1) |
| Te(1)–Pt–P(1) | 89.3 (1) | Pe(2)–Pt–P(2) | 85.6 (1) |
| Te(1)–Pt–P(2) | 172.2 (1) | Te(2)–Pt–P(1) | 176.4 (1) |
| Te(1)–C(1)–C(2) | 119.2 (8) | Te(1)–C(1)–C(6) | 121.1 (8) |
| Te(2)–C(6)–C(5) | 117.4 (9) | Te(2)–C(6)–C(1) | 122.6 (7) |
| C(1)–C(6)–C(5) | 119.9 (11) | C(2)–C(1)–C(6) | 119.7 (10) |
| C(4)–C(5)–C(6) | 118.8 (13) | C(1)–C(2)–C(3) | 119.0 (13) |
| C(3)–C(4)–C(5) | 119.9 (13) | C(2)–C(3)–C(4) | 122.6 (14) |
| Dihedral Angles | | | |
| C(1)–C(6)/Te(1), C(1), C(6), Te(2) | | | 2.9 (3) |
| Te(1), C(1), C(6), Te(2)/Te(1), Pt, Te(2) | | | 1.3 (2) |
| Te(1), C(1), C(6), Te(2)/P(1), P(2), Pt, Te(1), Te(2) | | | 1.3 (2) |

$CH_2Cl_2$ solution of **1**. The crystal belonged to the monoclinic space group $P2_1/n$ (295 K): $a = 12.782$ (2) Å, $b = 20.174$ (3) Å, $c = 14.704$ (3) Å, $\beta = 103.36$ (1)°, $V = 3688.9$ (10) Å$^3$, $Z = 4$, $D$(calcd) = 1.892 g cm$^{-3}$, $\mu$(Mo K$\alpha$) = 57.2 cm$^{-1}$. By the use of a Nicolet R3 diffractometer with a graphite monochromator and $\omega$ scans ($4° \leq 2\theta \leq 48°$), 6274 reflections were collected. A total of 5732 data were unique ($R_{int} = 1.12\%$), and 4241 data with $F_o \geq 5\sigma(F_o)$ were considered observed. An empirical absorption correction ($T_{max} = 0.237$, $T_{min} = 0.163$) and a 4% linear decay correction were applied. The structure was solved by direct methods (Pt and two Te atoms) and refined by subsequent difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically while the phosphine phenyl rings were constrained as rigid bodies and hydrogen atoms were constrained as idealized, updated isotropic contributions. $R_F = 0.0397$, $R_{wF} = 0.0424$, GOF = 1.008. In the final refinement cycle the maximum shift/$\sigma$ was 0.018 and the maximum nonassigned electron density in the difference map was 0.64 e/Å$^3$ (0.84 Å from Pt). The data/parameter ratio was 12.0. The atomic coordinates and isotropic thermal parameters are collected in Table I. Selected bond angles and bond distances are in Table II.

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**Registry No.** 1, 107494-87-1; 2, 107494-88-2; $Pt(STeC_6H_4)(PPh_3)_2$, 107494-89-3; $Pt(SeTeC_6H_4)(PPh_3)_2$, 107494-90-6; $(HgC_6H_6)_6$, 256-24-6; Li, 7439-93-2; Te, 13494-80-9; *cis*-$PtCl_2(PPh_3)_2$, 15604-36-1; $Pt(S_2C_6H_4)(PPh_3)_2$, 107494-91-7; *t*-BuLi, 594-19-4; *n*-BuLi, 109-72-8; $Ph_2Se_2$, 1666-13-3; 1,2-dibromocyclopentene, 75415-78-0; benzenethiol, 108-98-5.

**Supplementary Material Available:** A table of anisotropic thermal parameters (1 page); a table of structure factors (25 pages). Ordering information is given on any current masthead page.

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# A New Synthetic Route to Cyclic Polyarsines

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Cyclic polyarsines have been of continued interest[1-7] owing to the extreme adaptability of arsenic as a bridging element in metal clusters and their unique coordinating modes in transition-metal complexes. A variety of reactions have been reported for the synthesis of polyarsines.[8-13] Typically, low yields of the cyclic polyarsines are obtained. Furthermore, the products are often contaminated with difficult to remove byproducts. In this paper, we report a new and convenient synthetic pathway to high yields of pure cyclic polyarsines.

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