tisite magnetization transfer data and EXSY data, respectively. Dr. B. E. Mann is also thanked for a preprint of ref 29 prior to publication.

Supplementary Material Available: Tables of equilibrium constants for cis-trans isomerization and optimized parameters from magnetization transfer experiments using DANTE inversion and plot of ln K versus $1/T$ for cis-trans isomerization (Figure S1), a ^{13}C EXSY spectrum of 1 in toluene-d_8 at 224 K (Figure S2), plots of the experimental and optimized magnetization versus time curves (Figure S3), a plot of ln (k/T) versus $1/T$ for the rate constants k_{12} (Figure S4), and experimental and simulated ^{13}C NMR spectra of 1 in the carbonyl region (Figure S5) (9 pages). Ordering information is given on any current masthead page.

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Synthesis and Reactivity of Cyclic Phosphetanes. Oligomerization, Quaternization, and Complexation with Platinum(II)

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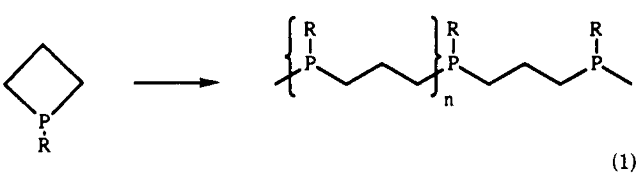
Summary: The new cyclic phosphetane *cis*- and *trans*-3-*tert*-butyl-1-phenylphosphetane (1) was prepared by a heteroatom metallacycle transfer reaction from 3-*tert*-butyltitanacyclobutane and dichlorophenylphosphine. Phosphetane 1 is stable to polymerization in the presence of electrophilic initiators in solution but forms low oligomers when the neat liquid is heated (>250 °C for 120 h). Phosphetane 1 was employed to form the phosphetane complex *cis*-dichlorobis(3-*tert*-butyl-1-phenylphosphetane)platinum(II), which was characterized by X-ray diffraction.

Polynuclear transition-metal complexes bridged by bis(diarylphosphino)- and bis(dialkylphosphino)methanes often possess novel chemical and photochemical properties.[1-4] The corresponding bis(diarylphosphino)propanes may also serve as bridging ligands to two metal centers but are sufficiently flexible to serve in some cases as chelating ligands to one metal center.[5] In the course of our recent studies of di- and trinuclear diphosphine-bridged transition-metal complexes,[1-4] we sought a general method of preparing polyphosphine ligands which were capable of supporting linear metal clusters of higher nuclearity. Polymers functionalized with phosphines are also of significant interest as catalyst supports[6-9] and in the stabilization of metal/polymer interfaces.[10] One approach to the synthesis of poly(phosphinopropanes) is the ring-opening polymerization of cyclic phosphetanes (eq 1).



(1)

Previous reports by Cremer et al. have noted the possibility of phosphetane polymerization. Upon sitting, an evacuated tube containing neat 2,2,3-trimethyl-1-phenylphosphetane was observed to become very viscous and apparently had polymerized or decomposed.[11] In addition, concentration of a highly strained tetracyclic phosphine was observed to result in polymerization.[12]

Here we describe the synthesis of the new phosphetanes *cis*- and *trans*-3-*tert*-butyl-1-phenylphosphetane (1), their reactivity toward polymerization, and their use as ligands in the preparation of the complex *cis*-dichlorobis(3-*tert*-butyl-1-phenylphosphetane)platinum(II).

Results and Discussion

Phosphetane 1 was prepared by the recently reported heteroatom metallacycle transfer reaction from 3-*tert*-butyltitanacyclobutane and dichlorophenylphosphine (eq 2).[13] Essentially equal quantities of the cis (δ −38.3 ppm) and trans (δ −6.8 ppm) isomers are evident by $^{31}P\{^1H\}$ NMR spectroscopy. Earlier $^{31}P\{^1H\}$ studies established that *cis*-phosphetanes generally exhibit chemical shifts which occur 20–30 ppm upfield of the trans isomers.[14] Similarly, the corresponding ^1H NMR data for *cis*- and *trans*-1 are

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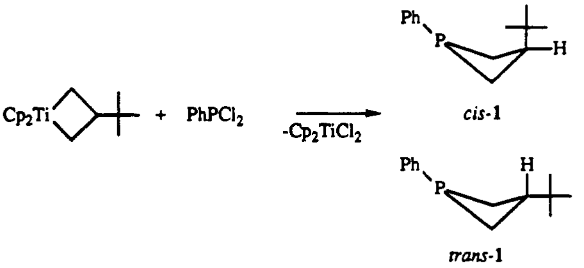
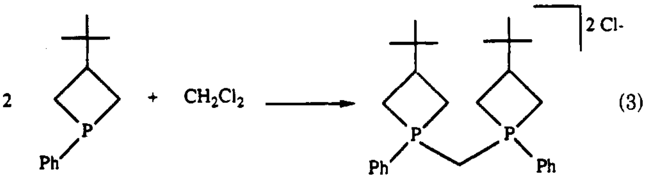


(2)

in qualitative agreement with data reported for known phosphetanes. In particular, the lone methine protons of the cis isomers are generally observed 0.4–0.7 ppm upfield of those for the trans isomer.[14] In the case of 1, the multiplet at 2.81–2.96 ppm is assigned to the trans isomer and that at 2.41–2.60 ppm is assigned to the cis isomer. A 200-MHz NOESY correlation experiment permitted the further assignment of the *tert*-butyl proton signals of the two isomers but did not resolve the individual phenyl or methylene signals.

The phosphetane ring of 1 is remarkably robust. For example, azetidines, the corresponding four-membered nitrogen heterocyclic compounds, are readily polymerized by electrophilic initiation.[15,16] However, treatment of 1 with 5% ethyl trifluoromethanesulfonate in $CDCl_3$ (11 h, 100 °C) or 10% triphenylcarbenium hexafluorophosphate in $CDCl_3$ (608 h, 85–105 °C) in sealed-tube reactions results in initial quaternization, but not in polymerization. Heating 1 in a sealed tube at 70 °C in methylene chloride with or without initiators also does not lead to polymerization but instead to the methylenebis(phosphonium)salt 2, derived from methylene chloride solvent (eq 3).



(3)

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Ring opening of 1 was achieved by heating the neat liquid phosphetane in a vacuum-sealed thick-walled glass tube for 5 days at 250 °C. Triphenylcarbenium hexafluorophosphate and methyl iodide initiators do not improve the rate of ring opening. The viscous orange product was characterized by $^{31}P\{^1H\}$ NMR spectroscopy and mass spectrometry. The $^{31}P\{^1H\}$ spectra of the product show a broad signal centered at −27 ppm along with less intense signals at −7.9 and −14.7 ppm. The $^{31}P\{^1H\}$ signal at −27 ppm compares well with the only other reported data for a poly(phenylphosphino)propane. Saegusa et al. prepared poly(phenylphosphino)propane in two steps by initial cationic ring-opening polymerization of 2-phenyl-1,2-oxaphospholane followed by reduction of the oxygenated material. The $^{31}P\{^1H\}$ spectrum of the material with an average molecular weight of 4100 (n = 27) is reported[17,18] to consist of one signal at −26.8 ppm. In separate studies, we have prepared poly(phenylphosphino)propylenes by condensation polymerization of dilithiated phosphines with 1,3-dichloropropane and by Michael addition of phenylphosphine to bis(allyl)phenylphosphine.[19] In all cases, $^{31}P\{^1H\}$ NMR data for samples are characterized by one signal at approximately −27 ppm. Mass spectral analysis (CI; 70 eV; T(source) = 250 °C; T(probe) = 240 °C) of the product obtained by ring-opening 1 revealed only low oligomers with the most intense parent ions observed at m/z = 413 (n = 2), 619 (n = 3). The stability of phosphetane 1 with respect to polymerization compared to that for azetidines is likely due to reduced ring strain resulting from longer P–C (~1.8 Å) compared to N–C (~1.4 Å) bonds. The presence of *tert*-butyl groups at the carbon atom β to phosphorus is also likely to contribute to a steric barrier to polymerization due to gauche interactions.

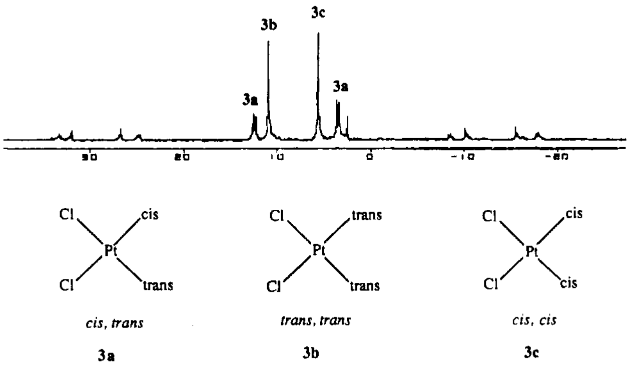
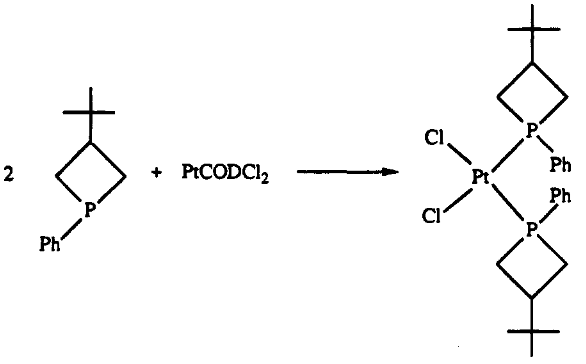


Figure 1. $^{31}P\{^1H\}$ NMR spectrum of *cis*-dichlorobis(3-*tert*-butyl-1-phenylphosphetane)platinum(II) (**3**). Three distinct platinum complexes are observed, which differ in the stereochemistry of the phosphetane rings: cis,trans (**3a**), trans,trans (**3b**), and cis,cis (**3c**).

Phosphetane 1 was also characterized by its reaction with $Pt(COD)Cl_2$ (COD = 1,5-cyclootadiene). The reaction of 2 equiv of *cis*- and *trans*-1 with $Pt(COD)Cl_2$ affords *cis*-dichlorobis(3-*tert*-butyl-1-phenylphosphetane)platinum(II) (3; eq 4). $^{31}P\{^1H\}$ NMR spectroscopy indicates three isomers of complex 3 exist (Figure 1). These can be distinguished by the configuration of the phosphetane ligands attached to platinum. The $^{31}P\{^1H\}$ NMR spectrum was assigned by assuming that the chemical shift of the coordinated *cis*-phosphetane isomer remains upfield from that of the trans isomer. Thus, the isomer of 3 which contains one *cis*- and one *trans*-phosphetane ring (**3a**) exhibits an AB multiplet centered between the signals of the trans,trans isomer (**3b**) at 10.88 ppm and the cis,cis isomer (**3c**) at 5.56 ppm.



(4)

3

Complex 3 was also characterized by a single-crystal diffraction study. The crystallographic study revealed two independent molecules per unit cell in the single crystal that was analyzed. One molecule corresponds to the *cis,trans*-phosphetane ring isomer **3a** (Figure 2), while the second independent molecule is the trans,trans isomer **3b** (Figure 3). Phosphetane complexation generally occurs with retention of configuration;[20] thus, the cis,cis isomer

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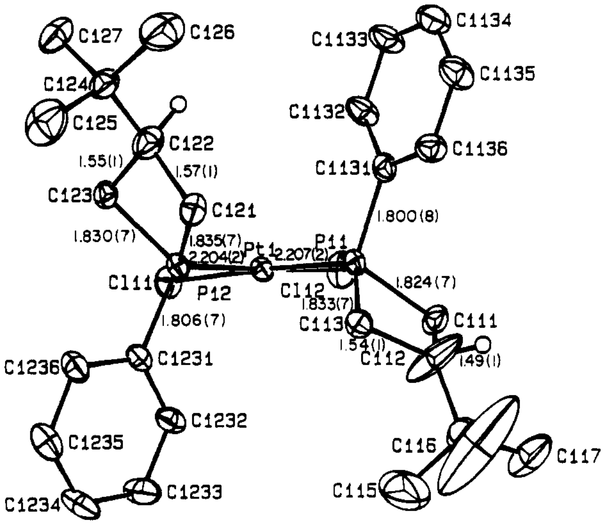

Figure 2. ORTEP drawing of the *cis,trans*-phosphetane ring stereoisomer **3a**.

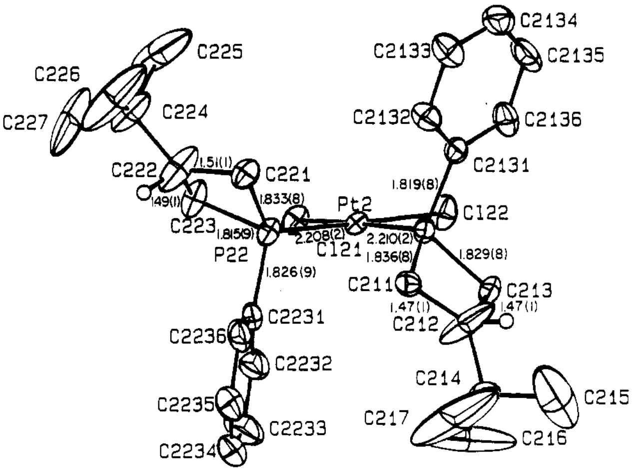

Figure 3. ORTEP drawing of the *trans,trans*-phosphetane ring stereoisomer **3b**.

Table I. Selected Bond Distances (Å) and Angles (deg) for 3a and 3b

	Bond Distances for **3a**		
Pt1–P11	2.207 (2)	C111–C112	1.49 (1)
Pt1–P12	2.204 (2)	C112–C113	1.54 (1)
Pt1–Cl11	2.363 (2)	C113–P11	1.833 (7)
Pt1–Cl12	2.342 (2)	P12–C121	1.835 (7)
P11–C1131	1.800 (8)	C112–C123	1.55 (1)
P11–C111	1.824 (7)	C123–P12	1.830 (7)
	Bond Angles for **3a**		
Cl11–Pt1–Cl12	91.94 (7)	C112–C113–P11	89.4 (5)
Cl11–Pt1–P12	86.48 (7)	C113–P11–C111	78.7 (3)
Cl12–Pt1–P11	88.83 (7)	P12–C121–C122	87.8 (4)
P11–Pt1–P12	92.84 (7)	C121–C122–C123	96.4 (6)
P11–C111–C112	91.5 (5)	C122–C123–P12	88.7 (5)
C111–C112–C113	99.8 (7)	C123–P12–C121	78.8 (3)
	Bond Distances for **3b**		
Pt2–P21	2.210 (2)	C211–C212	1.47 (1)
Pt2–P22	2.208 (2)	C212–C213	1.47 (1)
Pt2–Cl21	2.342 (2)	C213–P21	1.829 (8)
Pt2–Cl22	2.355 (2)	P22–C221	1.833 (8)
P21–C2131	1.819 (8)	C221–C222	1.51 (1)
P22–C2231	1.826 (9)	C222–C223	1.49 (1)
P21–C211	1.836 (8)	C223–P22	1.815 (9)
	Bond Angles for **3b**		
Cl21–Pt2–Cl22	90.13 (8)	C212–C213–P21	88.2 (6)
Cl21–Pt2–P22	89.39 (8)	C213–P21–C211	79.0 (4)
Cl22–Pt2–P21	86.51 (8)	P22–C221–C222	88.5 (6)
P21–Pt2–P22	94.00 (8)	C221–C222–C223	100.1 (8)
P21–C211–C212	87.7 (6)	C222–C223–P22	89.7 (6)
C211–C212–C213	105.1 (8)	C223–P22–C221	78.2 (4)

Table II. Phosphetane Dihedral Angles (deg) for 3a and 3b

plane 1	plane 2	angle
	Compound **3a**	
C121–C122–C123	C121–P12–C123	30.67 ± 0.68
C111–C112–C113	C111–P11–C113	7.98 ± 2.07
	Compound **3b**	
C221–C222–C223	C221–P22–C223	20.01 ± 1.52
C211–C212–C213	C211–P21–C213	0.77 ± >10

presumably may be present in a separate crystal. A summary of selected bond distances and angles for **3a** and **3b** are presented in Table I. The coordinated phosphetane rings are staggered above and below the approximately square coordination plane about platinum. The dihedral angles of the coordinated phosphetanes (Table II) were found to vary from ring to ring, with one phosphetane ring having a significantly larger dihedral angle than the second phosphetane ring contained within the molecule. This was also observed in the crystal structure study of *trans*-tricarbonylbis(*trans*-2,2,3,4,4-pentamethyl-1-phenylphosphetane)iron, where the phosphetane rings had dihedral angles of 20.3 and 6.1°.[20]

It is noteworthy that the *cis*-bis(phosphetane)platinum complex is formed exclusively. The relatively unusual characteristics of phosphetane **1** as a phosphine donor ligand are reflected in ^1J(Pt–P) values for all three isomers of **3** which lie below the typical range of 3500–3700 Hz for *cis*-$PtCl_2(PR_3)_2$ complexes.[21] This suggests that the phosphetane ligand exerts a very weak σ-trans effect and has little π-acceptor character. The $^{31}P\{^1H\}$ NMR data for the phosphetane ligands in **3**, the structure of phosphetane **1**, and the relative lack of reactivity of **1** all tend to suggest that the donor ability of **1** as a ligand is restricted more by steric considerations than by basicity of the phosphorus atom.

In view of the stability of phosphetane **1** toward ring-opening polymerization and its reactivity with solvents such as CH_2Cl_2, we are continuing synthetic studies of polymeric phosphines along other lines.

Experimental Section

General Data. All manipulations were done under an oxygen-free atmosphere using Schlenk techniques or an inert-atmosphere box. Anhydrous pentane was purchased from Aldrich and used as is. Other organic solvents were dried and degassed using standard procedures. ^{31}P and ^1H NMR spectra were recorded on Varian XL-200 spectrometers. The ^{31}P chemical shifts are reported relative to external 85% H_3PO_4.

Procedure for *cis*- and *trans*-1. A slurry of 3-*tert*-butyltitanacyclobutane (6.15 g, 22.2 mmol) in 450 mL of anhydrous pentane was cooled to −41 °C (CH_3CN/N_2). $PhPCl_2$ (3.03 mL, 22.3 mmol) was slowly added to the stirred solution by syringe. The reddish solution gradually turned orange, and an orange precipitate (Cp_2TiCl_2) was produced. The mixture was stirred at −41 °C for 1.5 h followed by another 1 h of stirring at room temperature. A coarse frit was used to rapidly filter the orange precipitate. Solvent was removed from the slightly orange filtrate. Kugelrohr distillation (78 °C, 0.05 Torr) afforded a clear liquid: yield 34%; ρ = 0.96 g/mL. $^{31}P\{^1H\}$ NMR (200 MHz, $CDCl_3$): δ −6.8 (s, *trans*-1), −38.3 (s, *cis*-1). ^1H NMR (200 MHz, CD_2Cl_2): δ 0.79 (s, 9 H, $C(CH_3)_3$ of *cis*-1), 0.85 (s, 9 H, $C(CH_3)_3$ of *trans*-1), 1.71–2.22 (m, 8 H, CH_aH_b of *cis*-1 and *trans*-1), 2.41–2.60 (m, 1 H, $CHC(CH_3)_3$ of *cis*-1), 2.81–2.96 (m, 1 H, $CHC(CH_3)_3$ of *trans*-1), 7.29–7.60 (m, 10 H, C_6H_5 of *cis*-1 and *trans*-1).

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Procedure for 2. Phosphetane 1 (0.096 g, 0.465 mmol) and methylene chloride (1.5 mL, 13.3 mmol) were syringed into a Teflon-screw-top pressure NMR tube (Wilmad). The tube was heated in an oil bath at 90 °C for 68 h. The reaction is quantitative by ^{31}P NMR spectroscopy. X-ray-quality crystals were obtained by reacting 0.960 g (4.65 mmol) of the phosphetane with 0.5 mL (4.44 mmol) of CH_2Cl_2. Crystals formed upon heating at 90 °C overnight. A partial X-ray structure was determined for 2. However, due to the presence of isomers of 1, there was disorder in the structural determination. $^{31}P\{^1H\}$ NMR (200 MHz, CD_2Cl_2): δ 22.3 (s, *trans,trans*-**2**), 19.8 (AB quartet, *cis,trans*-**2**), 17.3 (s, *cis,cis*-**2**). The singlet peak assignments were made by assuming that the cis isomer remains upfield of the trans isomer.

Procedure for 3. To a solution of Pt(1,5-COD)Cl_2 (0.16 g, 0.45 mmol) in 10 mL of CH_2Cl_2 was added the phosphetane 1 (0.17 mL, 0.86 mmol). The solution was stirred overnight, followed by removal of solvent under vacuum. The product was recrystallized by slow diffusion of ether into a CH_2Cl_2 solution of 3. $^{31}P\{^1H\}$ NMR (200 MHz, CD_2Cl_2): isomer **3a**, δ 3.44 (d, ^1J(Pt–P) = 3453 Hz, ^2J(P–P) = 20.4 Hz), 12.36 (d, ^1J(Pt–P) = 3378 Hz, ^2J(P–P) = 20.0 Hz); isomer **3b**, δ 10.88 (s, ^1J(Pt–P) = 3415 Hz); isomer **3c**, δ 5.56 (s, ^1J(Pt–P) = 3418 Hz).

X-ray Structure Determination for 3. X-ray-quality crystals of 3 were obtained by slow diffusion of ether into a CH_2Cl_2 solution. The complex crystallized as $3\cdot0.5Et_2O$ in the triclinic space group $P\bar{1}$, with a = 14.140 (3) Å, b = 15.784 (5) Å, c = 16.047 (3) Å, α = 68.18 (3)°, β = 83.47 (2)°, γ = 69.94 (2)°, V = 3122 (1) Å^3, Z = 4, and d_{calcd} = 1.522 g/cm^3 for the formula $C_{28}H_{43}O_{0.5}P_2Cl_2Pt$. Crystal dimensions were 0.63 × 0.19 × 0.10 mm. A total of 8116 unique data were collected over the *hkl* limits −14 to 15, −15 to 16, 0–17. The structure was solved by MULTAN–least-squares–Fourier methods and was refined to R and R_w values of 0.033 and 0.040 for 289 variables and 6121 unique data with $I > 3.0\sigma(I)$. All programs were from the Enraf-Nonius SDP package. All calculations were performed on a micro-VAX computer.

Acknowledgment is made to NASA (Grant No. NAG-3-977) and the NSF (Grant No. CHE-9016513) for support of this work. We are also grateful to Drs. Perry Pellechia and Dean Carlson for Assistance with the NOESY experiment and to Professor Sheldon E. Cremer of Marquette University for helpful comments on this work. J.C.H. gratefully acknowledges Upjohn and U.S. Department of Education National Needs Graduate Fellowships.

Supplementary Material Available: Tables of crystal data and data collection parameters, positional parameters, general temperature factor expressions, and bond distances and angles for 3 (22 pages). Ordering information is given on any current masthead page.

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Synthesis and Characterization of Volatile Trifluoromethyl Alkyl Tellurides

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Summary: Ligand exchange reactions between bis(trifluoromethyl)tellurium and di-*tert*-butyl or dibenzyl telluride were used to prepare the odorless, unsymmetrical trifluoromethyl alkyl tellurides $CF_3TeC(CH_3)_3$ and $CF_3TeCH_2C_6H_5$, respectively. These new compounds, potentially useful as source reagents for chemical vapor deposition of tellurium alloys, are considerably more volatile than the corresponding MeTeR (R = ^{t}Bu, Bz) derivatives and decompose at comparable temperatures.

Volatile organotellurium compounds are commonly used as source reagents for metal organic chemical vapor deposition (MOCVD) of mercury cadmium telluride (MCT). Fabrication of devices based on MCT requires the ability to grow abrupt junctions over large areas at low temperatures (<300 °C). High growth temperatures coupled with low growth rates result in diffusion of mercury across the heterojunction, thus destroying its integrity and usefulness.[1] In this note we report the synthesis of two novel highly volatile trifluoromethyl alkyl tellurides potentially suitable as tellurium source reagents for growth of mercury cadmium telluride.

MOCVD of mercury cadmium telluride is presently limited by the availability of appropriate tellurium source reagents. Volatility of the tellurium source reagent and efficient, low-temperature decomposition of the source reagent to elemental tellurium, without incorporation of impurities into the product film, are among the most important criteria for effective tellurium source reagents. Competing trends in volatility and decomposition temperature currently require a compromise between growth rate and temperature (Table I).[2]

Table I. Trends in Volatility and Decomposition Temperatures of Organotellurium Compounds

compd	vapor pressure, mmHg (temp, °C)	growth temp,[a] °C
dimethyl telluride	760 (82)	>450
diisopropyl telluride	5.6 (30)	390
di-*tert*-butyl telluride	4 (40)	320
diallyl telluride	3.5 (45)	280
methyl allyl telluride	8 (30)	290

[a] Growth temperatures are from literature data and are the temperatures at which the growth rate becomes temperature-independent.[4,17]

Table II. Boiling Points of Selected Organometallic Compounds at 1 atm

compd	bp, °C	perfluoro compd	bp, °C
$(CH_3)_2S$	37.3	$(CF_3)_2S$	−22
$(CH_3)_2Se$	54.5	$(CF_3)_2Se$	
$(CH_3)_2Te$	82	$(CF_3)_2Te$	23

The absence of intermolecular forces in perfluoroalkyl compounds often leads to increased volatility as compared to the hydrocarbon analogues (Table II).[3] We sought to

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