Organometallics 1990, 9, 275-277

ligand.[10] The ratios achieved with use of diphosphine ligand 1 were 57:43, 83:17, and 88:12 and with the myrtenol-derived diphosphine 2 were 64:36, 64:36, and 88:12 for the complexes with allyl, 1,3-diphenylallyl, and cyclohexenyl, respectively.[14] In both cases the stereoselectivity for the formation of the cyclohexenyl complexes is better than that for the diphenylallyl compounds. This would be expected since the staggered form of the diphenylallyl complex has the allyl substituents gauche to both phenyl groups on each phosphine, whereas the staggered cyclohexenyl complex has its substituents gauche to only one phenyl group, resulting in a smaller energy difference in the two diphenylallyl isomers.

Having established the ability to coordinate at least one class of C_s-symmetrical π-allyls selectively to C_s-symmetric (diphosphine)palladium complexes containing diphenylphosphines, we are now pursuing the preparation of related diphosphines in which the two phosphines are electronically different, which we anticipate will function as efficient asymmetric ligands.

Acknowledgment. We thank the Graduate Research School of Boston University for financial support of this work.

Supplementary Material Available: A table listing ^{1}H, ^{13}C, and ^{31}P NMR data for the diastereomeric mixtures of the nine (η^3-allyl)(diphosphine)palladium complexes (2 pages). Ordering information is given on any current masthead page.

---

[14] The ratios of all compounds were determined by examination of the crude ^{1}H NMR spectra. Characterization was done on purified materials. The ratios reflect kinetically formed products (reaction time 30 min). Both diastereomers of 3-Pd$^+$(η^3-1,3-diphenylallyl) were isolated by crystallization, and both isomers were observed to equilibrate slowly in solution, reaching the same 55:45 ratio in 48 h, where the major isomer corresponds to the original minor isomer.

$(t\text{-Bu})_2$GaAs$(t\text{-Bu})_2$: A Volatile Monomeric Arsinogallane

Kelvin T. Higa*

Chemistry Division, Research Department
Naval Weapons Center, China Lake, California 93555

Clifford George

Laboratory for the Structure of Matter
Naval Research Laboratory, Washington, D.C. 20375

Received July 31, 1989

Summary: The 1:1 reaction of t-Bu$_2$GaCl and t-Bu$_2$AsLi in benzene affords the volatile monomeric compound (t-Bu)$_2$GaAs(t-Bu)$_2$ as a yellow solid. The single-crystal X-ray structure, ^{1}H and ^{13}C NMR, IR, and mass spectroscopy, elemental analysis, and isopiestic molecular weight determination are reported.

Although arsinogallanes have been known for over 25 years,[1] monomeric arsinogallanes are rare. Arsinogallanes are usually found as dimers, trimers, or adducts due to the proclivity of Ga(III) toward tetracoordination.[2-5] Monomeric arsinogallanes are expected to exhibit a pπ–pπ interaction between the unoccupied p orbital on gallium and the filled p orbital on arsenic. Multiple bonding between main-group elements[6-8] has been well documented, but not for third-row or higher group III–V compounds. Attempts to prepare monomeric arsinogallanes have focused on the use of the bulky substituents. The first monomeric arsinogallane, [(mesityl)$_2$As]$_3$Ga, was reported by Wells et al.[9] in 1986, and subsequently, the *tert*-butyl derivative was reported by Cowley[10] and co-workers. Only recently, Theopold[11] and co-workers reported the first mono(arsino)gallane monomer, (C$_5$Me$_5$)$_2$GaAs(SiMe$_3$)$_2$, and its conversion to amorphous GaAs powder via reaction with alcohol.

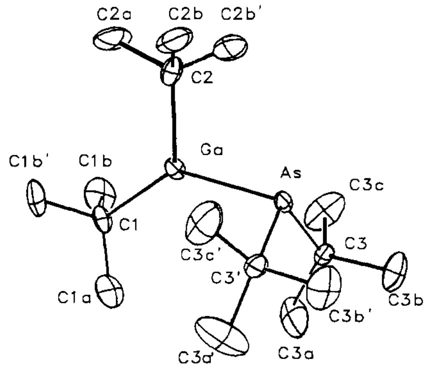


Figure 1. Structure of (t-Bu)$_2$GaAs(t-Bu)$_2$ (1). Hydrogen atoms are omitted for clarity.

Table I. Selected Bond Lengths (Å) and Bond Angles (deg) for (t-Bu)$_2$GaAs(t-Bu)$_2$

Ga–As	2.466 (3)	C(2)–C(2b)	1.510 (16)
Ga–C(1)	2.016 (13)	C(2)–C(2b′)	1.510 (16)
Ga–C(2)	1.961 (17)	C(3)–C(3a)	1.490 (18)
As–C(3)	1.982 (10)	C(3)–C(3b)	1.504 (14)
As–C(3′)	1.982 (10)	C(3)–C(3c)	1.505 (17)
C(1)–C(1a)	1.483 (24)	C(3′)–C(3a′)	1.490 (18)
C(1)–C(1b)	1.536 (14)	C(3′)–C(3b′)	1.504 (14)
C(1)–C(1b′)	1.536 (14)	C(3′)–C(3c′)	1.505 (17)
C(2)–C(2a)	1.541 (22)		
As–Ga–C(1)	132.7 (4)	Ga–C(1)–C(1b)	106.6 (8)
As–Ga–C(2)	106.8 (4)	Ga–C(2)–C(2a)	118.3 (11)
C(1)–Ga–C(2)	120.5 (6)	Ga–C(2)–C(2b)	108.9 (8)
Ga–As–C(3)	103.4 (3)	As–C(3)–C(3a)	110.2 (6)
Ga–As–C(3′)	103.4 (3)	As–C(3)–C(3b)	110.0 (7)
C(3)–As–C(3′)	110.2 (6)	As–C(3)–C(3c)	107.3 (7)
Ga–C(1)–C(1a)	116.3 (9)		

Here we report the synthesis and crystal structure of (t-Bu)$_2$GaAs(t-Bu)$_2$ (1), the first volatile monomeric mono(arsino)gallane, and its conversion to polycrystalline GaAs by pyrolysis at 400–450 °C. Benzene (20 mL) was added to t-Bu$_2$GaCl[12] (0.66 g, 3.0 mmol) and t-Bu$_2$AsLi[13]

---

[1] Coates, G. E.; Graham, J. *J. Chem. Soc.* **1963**, 233.

[2] Beachley, O. T.; Coates, G. E. *J. Chem. Soc.* **1965**, 3241.

[3] Wells, R. L.; Purdy, A. P.; McPhail, A. T.; Pitt, C. G. *J. Organomet. Chem.* **1986**, *308*, 281.

[4] Purdy, A. P.; Wells, R. L.; McPhail, A. T.; Pitt, C. G. *Organometallics* **1987**, *6*, 2099.

[5] Wells, R. L.; Holley, W. K.; Shafieezad, S.; McPhail, A. T.; Pitt, C. G. Fifth International Symposium on Inorganic Ring Systems, Amherst, MA, 1988.

[6] Bartlett, R. A.; Chen, H.; Rasika Dias, H. V.; Olmstead, M. M.; Power, P. P. *J. Am. Chem. Soc.* **1988**, *110*, 446.

[7] Dräger, M.; Escudie, J.; Couret, C.; Ranaivonjatovo, H.; Satge, J. *Organometallics* **1988**, *7*, 1010.

[8] Jutzi, P.; Meyer, U. *J. Organomet. Chem.* **1987**, *326*, C6.

[9] Pitt, C. G.; Higa, K. T.; McPhail, A. T.; Wells, R. L. *Inorg. Chem.* **1986**, *25*, 2483.

[10] Arif, A. M.; Benac, B. L.; Cowley, A. H.; Geerts, R.; Jones, R. A.; Kidd, K. B.; Power, J. M.; Schwab, S. T. *J. Chem. Soc., Chem. Commun.* **1986**, 1543.

[11] Byrne, E. K.; Parkanyi, L.; Theopold, K. H. *Science* **1988**, *241*, 334.

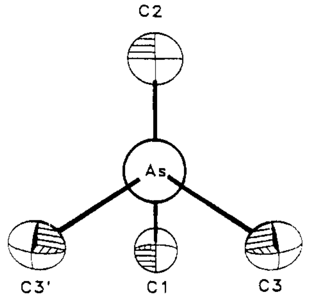
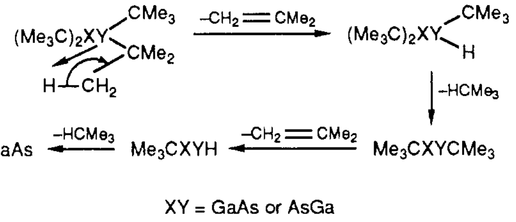


Figure 2. Newman projection down the As–Ga bond.

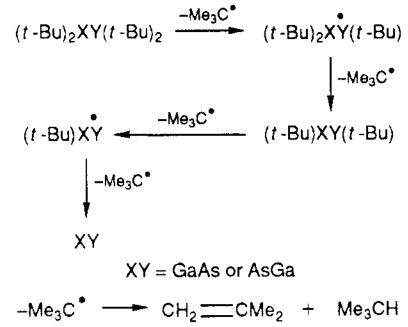
(0.60 g, 3.1 mmol) at room temperature. After it was stirred for 2.5 days under Ar, the yellow-orange solution was filtered with use of a fine frit and the solvent removed under vacuum to give crude $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$ (1.1 g, 2.9 mmol, 97% yield) as a yellow-orange oil. The product, which began to sublime at 18 °C at 10^{-3} Torr, was purified by sublimation at 65 °C at 10^{-3} Torr. Recrystallization in pentane at −78 °C gave yellow crystals[14] suitable for X-ray analysis; mp 41–44 °C.

The degree of association of 1 in the gas phase was investigated by mass spectroscopy and in solution by molecular weight determinations. The chemical ionization mass spectrum[15] contains major peaks (m/e) due to the monomeric molecular ion (372, 374), parent + 1 (373, 375), and the expected fragmentation byproducts. However, higher mass peaks in very low intensity were observed that could be due to either the dimer or, more likely, fragment recombination. Two isopiestic molecular weight determinations in pentane gave molecular weights of 419 and 395, which are consistent with a monomeric structure (molecular weight of 373) in solution.

Scheme I



Scheme II



The structure of 1, determined by single-crystal X-ray analysis,[16] is illustrated in Figure 1, with selected bond distances and angles listed in Table I. Compound 1 is monomeric, with the gallium atom in a trigonal-planar configuration while the three-coordinate arsenic atom is pyramidal. The Ga–As single-bond distance of 2.466 (3) Å is consistent with the sum (2.46 Å) of the covalent radii of Ga and As and is comparable to those found in other three-coordinate arsinogallanes, 2.433 (1) Å in $(C_5Me_5)_2GaAs(SiMe_3)_2$[11] and 2.470 (1)–2.508 (1) Å in $(Mes_2As)_3Ga$.[9] In contrast, the dimeric structures of the type $R_2GaAsR'_2$ exhibit longer Ga–As bond distances of 2.524, 2.550, and 2.558 Å in $[Ph_2GaAs(CH_2SiMe_3)_2]_2$,[2] $[Me_2GaAs(t\text{-Bu})_2]_2$,[10] and $[n\text{-Bu}_2GaAs(t\text{-Bu})_2]_2$,[10] respectively. The dimers $\{BrGa[As(CH_2SiMe_3)_2]_2\}_2$[17] and $\{Ga[As(CH_2SiMe_3)_2]_3\}_2$[18] also exhibit longer endocyclic Ga–As bond distances of 2.521 (1) and 2.540 (1)–2.581 (1) Å, while the exocyclic distances are 2.437 (1) and 2.470 (1)–2.478 (2) Å, respectively. The longer endocyclic Ga–As distances are consistent with those of the other dimers, and the shorter exocyclic distances are consistent with the distances in the monomeric compounds. The longer Ga–As distances cannot be attributed to tetracoordination or ring formation since the trimer $[Br_2GaAs(CH_2SiMe_3)_2]_3$[19] contains short

---

[12] t-Bu_2GaCl was prepared from a neat mixture containing a 2:1 molar ratio of t-Bu_3Ga and $GaCl_3$ heated to 120 °C for 2 h in a closed, evacuated flask. The product was purified by sublimation at 95 °C at 10^{-3} Torr. t-Bu_3Ga was prepared from a 3:1 reaction of t-BuLi and $GaCl_3$. t-BuLi (1.7 M in pentane) was slowly added to a 0.5 M solution of $GaCl_3$ in benzene at 0 °C. After 0.5 h, the reaction mixture was heated to 65 °C for 15 h, cooled to room temperature, and filtered and the solvent removed via distillation under Ar. The product was purified by distillation at 69–73 °C at 4 Torr. ^1H NMR (C_6D_6): t-Bu_3Ga δ 1.16 (s); t-Bu_2GaCl, δ 1.24 (s). ^{13}C NMR (C_6D_6): t-Bu_3Ga δ 31.3 m (C), 30.9 (quartet of septets, $^1J_{CH}$ = 123.8 Hz, $^3J_{CH}$ = 6.3 Hz, CH_3).

[13] t-Bu_2AsLi was prepared from the 1:1 reaction of t-Bu_2AsH and n-BuLi in hexane. The reaction mixture was heated to 55 °C for 1 h and the yellow product filtered, washed with hexane, and then dried under vacuum. t-Bu_2AsCl was prepared by the addition of 2 equiv of t-BuLi (1.7 M in pentane) to a 0.5 M solution of $AsCl_3$ in pentane at −78 °C. The reaction mixture was stirred for 4 h at room temperature and filtered and the pentane removed via distillation under Ar. $LiAlH_4$ was added to t-Bu_2AsCl (1 M in diethyl ether) in a 1:1 molar ratio at −78 °C. The reaction mixture was warmed and stirred at room temperature for 4 h, the solvent removed, and t-Bu_2AsH purified by distillation at 56 °C at 25 Torr. ^1H NMR of t-Bu_2AsH: δ 3.1 (s, 1 H), 1.25 (s, 18 H). ^{13}C NMR of t-Bu_2AsH: δ 32.34 (CH_3) 32.06 (C).

[14] $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$ decomposes to an uncharacterized white solid over a period of several hours in the air. The ^1H NMR spectrum consists of two equal-intensity singlets at δ 1.40 and 1.26 ppm due to $(t\text{-Bu})_2As$ and $(t\text{-Bu})_2Ga$ fragments, respectively. The chemical shift for the $(t\text{-Bu})_2As$ fragment is identical with those found for $[R_2GaAs(t\text{-Bu})_2]_2$ (R = Me, n-Bu), and the shift for the $(t\text{-Bu})_2Ga$ fragment is similar to the shift of δ 1.24 ppm found for $(t\text{-Bu})_2GaCl$. The ^{13}C NMR spectrum contains peaks at δ 35.14 (quartet of septets, $^1J_{CH}$ = 125.1 Hz, $^3J_{CH}$ = 4.9 Hz), 33.86 m, 33.50 m, 30.20 (quartet of septets, $^1J_{CH}$ = 124.1 Hz, $^3J_{CH}$ = 5.8 Hz). The multiplets at 35.14 and 33.86 ppm have been assigned to the methyl and quaternary carbons, respectively, of the $(t\text{-Bu})_2As$ fragment. The multiplets at 33.50 and 30.20 ppm have been assigned as the quaternary and methyl carbons, respectively, of the $(t\text{-Bu})_2Ga$ fragment. These assignments are consistent with those of $(t\text{-Bu})_2AsH$ (32.34 ppm, methyl; 32.06 ppm, quaternary) and $(t\text{-Bu})_3Ga$[12] (31.3 ppm, quaternary; 30.9 ppm, quartet of septets, $^1J_{CH}$ = 123.8 Hz, $^3J_{CH}$ = 6.3 Hz). The NMR data agree with the formulation $[(t\text{-Bu})_2GaAs(t\text{-Bu})_2]_n$, where n = 1–3, but the degree of association cannot be determined by NMR spectroscopy. IR for $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$ (Nujol mull): 2990 w, 2960 m, 2950 m, 2930 m, 2910 m, 2860 m, 2850 s, 2830 vs, 2765 w, 2710 w, 1466 m, 1384 w, 1361 m, 1155 m, 1012 w, 939 w, 802 m, 527 w. Anal. Calcd for $C_{16}H_{36}AsGa$: C, 51.51; H, 9.73; As, 20.08; Ga, 18.68. Found: C, 48.86; H, 9.36; As, 20.23; Ga, 18.36.

[15] Methane chemical ionization mass spectral data for $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$ in the range of m/z 100–900 (relative amplitudes in parentheses): found, 183 (22.2, $[t\text{-Bu}_2Ga]$), 315 (100.0), 317 (64.8, [molecular ion − t-Bu]), 372 (98.1), 374 (72.2, [molecular ion]), 373 (75.9), 375 (40.7, [molecular ion + 1]), 441 (20.4), 443 (29.6, [molecular ion + Ga]), 555 (20.4), 557 (25.9, [molecular ion + t-Bu_2Ga]), 687 (33.3), 689 (38.9, [(2 × molecular ion − t-Bu]).

[16] $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$ crystallizes in the orthorhombic space group $Pnma$ with a = 9.481 (7) Å, b = 13.064 (11) Å, c = 16.301 (14) Å, V = 2019 (3) Å^3, and Z = 4. X-ray diffraction data were collected at −40 °C on a Nicolet $R3_{m/v}$ diffractometer using graphite-monochromated Mo Kα radiation. A total of 1398 independent reflections were collected with use of the θ–2θ scanning technique between 3.0 and 45.0° in 2θ. A total of 1074 reflections with $F > 3\sigma(F)$ were used in the full-matrix least-squares refinement. The structure was solved by using direct methods with SHELXTL PLUS. The final values of the agreement indices were R = 0.066 and R_w = 0.060 with 106 variables.

[17] Purdy, A. P.; Wells, R. L.; McPhail, A. T.; Pitt, C. G. *Organometallics* **1987**, *6*, 2099.

[18] Wells, R. L.; Purdy, A. P.; Higa, K. T.; McPhail, A. T.; Pitt, C. G. *Gov. Rep. Announce. Index (U.S.)* **1987**.

[19] Wells, R. L.; Holley, W. K.; Shafieezad, S.; McPhail, A. T.; Pitt, C. G. *Phosphorus Sulfur* **1989**, *41*, 15.

Ga-As bond lengths of 2.432 (2)-2.464 (1) Å. The long Ga-As bond lengths in the dimers are primarily the result of ring strain due to endocyclic bond angles of 83-96° and steric repulsion. In $[Br_2GaAs(CH_2SiMe_3)_2]_3$, the Ga-As bond length is short since the ring strain is minimal, with endocyclic bond angles ranging from 103 to 121°. The Ga-As bond length is also reduced by electronic effects due to the electronegative bromide on the gallium. Electronic effects leading to bond shortening can be seen in the Ga-As and Ga-Sb bond lengths in $[Br_2GaAs(CH_2SiMe_3)_2]_3$ and $[Cl_2GaSb(t\text{-}Bu)_2]_3$,[20] which are consistent with the sum of their covalent radii. In contrast, the In-As bond length in $[Me_2InAsMe_2]_3$[20] of 2.669 (3)-2.679 (2) Å is slightly longer than the sum of their covalent radii (2.64 Å). A more dramatic shortening of the Ga-As bond is seen in $[(THF)Br_2Ga]_3As$,[21] which has the shortest known Ga-As distance of 2.404 (4) Å.

The Newman projection (Figure 2) of 1 down the Ga-As bond clearly shows that the lone pair on arsenic must be orthogonal to the unoccupied p orbital on gallium, which precludes the possibility of Ga-As double-bond character. The stabilization of the monomeric unit is due to the fact that the Ga and As atoms are effectively shielded from intermolecular association by the bulky *tert*-butyl substituents on both gallium and arsenic. Therefore, the yellow to yellow-orange color observed in these monomeric arsinogallanes must originate from charge-transfer processes. In contrast, associated compounds in which charge transfer cannot occur, containing tetracoordinated gallium and arsenic, are colorless solids.

$(t\text{-}Bu)_2GaAs(t\text{-}Bu)_2$ was heated to 150 °C for 10 min without decomposition but decomposed to red oligomers and/or polymers at 188-190 °C. Pyrolysis of 1 under a He atmosphere with a cool yellow flame (~400 °C) resulted in crystalline GaAs and an approximately 1:1 mole ratio of 2-methylpropane and 2-methylpropene.[22] The decomposition can be rationalized by a β-elimination followed by alkane elimination (Scheme I), a free-radical mechanism (Scheme II), or both.

β-Elimination in group III-V chemistry is not new. The thermal decomposition[23] of Et_3Ga occurs by this route to give Et_2GaH and ethylene, and $t\text{-}BuPH_2$ decomposes to give PH_3, 2-methylpropene, and 2-methylpropane.[24] In a recent pyrolysis study of diorganotellurium compounds, it was demonstrated that $(t\text{-}Bu)_2Te$ decomposes by both β-elimination and free-radical mechanisms to give 2-methylpropene and 2-methylpropane.[25] Both mechanisms may also be involved in the pyrolytic decomposition of 1.

Single-source organometallic precursors have been utilized to prepare epitaxial films of GaP,[26] GaAs,[27] and InP.[27,28] Volatile monomeric mono(arsino)gallanes, such as $(t\text{-}Bu)_2GaAs(t\text{-}Bu)_2$, may be better source compounds for the metal-organic chemical vapor deposition of GaAs. The expected advantages of $(t\text{-}Bu)_2GaAs(t\text{-}Bu)_2$ are as follows: (1) the lower vapor pressure and reduced air sensitivity[14] reduces the toxicity (AsH_3) and safety hazards (flammability of Me_3Ga) in GaAs film deposition; (2) the incorporation of *tert*-butyl groups, which can undergo β-elimination reactions, could result in lower carbon incorporation into the GaAs film; (3) highly crystalline GaAs films can be prepared at less than 550 °C; (4) high-purity source compounds can be achieved through multiple sublimations; (5) the correct stoichiometry of Ga to As is always maintained.

Acknowledgment. The financial support of the Office of Naval Research is gratefully acknowledged. We thank Drs. R. A. Nissan, M. P. Nadler, and T. A. Vanderah for their assistance in obtaining ^{13}C NMR and infrared spectra and Gandolfi data. We also thank R. Woolever for SEM X-ray analysis.

Supplementary Material Available: Atomic coordinates and isothermal displacement coefficients (Table S-I), bond lengths (Table S-II), bond angles (Table S-III), anisotropic displacement coefficients (Table S-IV), hydrogen atom coordinates and isotropic displacement coefficients (Table S-V), structure determination summary (Table S-VI), data collection summary (Table S-VII), and solution refinement summary (Table S-VIII) (6 pages); observed and calculated structure factors (Table S-IX) (5 pages). Ordering information is given on any masthead page.

---

(20) Cowley, A. H.; Jones, R. A.; Kidd, K. B.; Nunn, C. M.; Westmoreland, D. L. *J. Organomet. Chem.* **1988**, *341*, C1.

(21) Wells, R. L.; Shafieezad, S.; McPhail, A. T.; Pitt, C. G. *J. Chem. Soc., Chem. Commun.* **1987**, 1823.

(22) A sealed flask equipped with a Teflon valve containing 0.1 g of $(t\text{-}Bu)_2GaAs(t\text{-}Bu)_2$ under He was heated with a cool yellow flame for 10 min. The volatile products and 0.5 mL of deuterated toluene were condensed into an NMR tube at 10^{-4} Torr, and the tube was sealed. The molar ratio of 2-methylpropene to 2-methylpropane was 0.74, but this does not account for the products in the gas phase of the NMR sample. Energy-dispersive X-ray analysis of the GaAs powder showed a 53:47 Ga:As atom ratio. The error associated with SEM-EDX analysis is 5-6%. The sample was confirmed to be highly crystalline GaAs by the X-ray diffraction powder pattern (Gandolfi method).

(23) Yoshida, M.; Watanabe, H.; Uesugi, F. *J. Electrochem. Soc.* **1985**, *132*, 677.

(24) Chen, C. H.; Larsen, C. A.; Stringfellow, G. B.; Brown, D. W.; Robertson, A. J. *J. Cryst. Growth* **1986**, *77*, 11.

(25) Kirss, R.; Brown, D. *Abstracts of Papers*, 198th National Meeting of the American Chemical Society, Miami, FL, Sept 1989; American Chemical Society: Washington, DC, 1989; INOR 285.

(26) Maury, F.; Combes, M.; Constant, G.; Renucci, J. B. *J. Phys., Colloq.* **1982**, C1, 347.

(27) Cowley, A. H.; Benac, B. L.; Ekerdt, J. G.; Jones, R. A.; Kidd, K. B.; Lee, J. Y.; Miller, J. E. *J. Am. Chem. Soc.* **1988**, *110*, 624.

(28) Andrews, D. A.; Davies, G. J.; Bradley, D. C.; Faktor, M. M.; Frigo, D. M.; White, E. A. D. *Semicond. Sci. Technol.* **1988**, *3*, 1053.

Convenient Synthesis of β-Trichlorostannyl Ketones from α,β-Unsaturated Ketones and Their Facile Conversion to β-Trialkylstannyl Ketones

**Hiroyuki Nakahira, Ilhyong Ryu,* Akiya Ogawa,
Nobuaki Kambe, and Noboru Sonoda***

*Department of Applied Chemistry
Faculty of Engineering, Osaka University
Suita, Osaka 565, Japan*

Received August 16, 1989

Summary: Reaction of α,β-unsaturated ketones with tin(II) chloride dihydrate ($SnCl_2 \cdot 2H_2O$) and chlorotrimethylsilane in ether afforded β-trichlorostannyl ketones in good yields. These underwent chemoselective alkylation at Sn to give the corresponding β-trialkylstannyl ketones by the reaction with 3 equiv of Grignard reagents at -78 °C.

In the course of our study on the utilization of β-metallo ketones as synthetic intermediates,[1] we required a variety of β-trichlorostannyl ketones. While the ring-opening reaction of siloxycyclopropanes 1 with tin(IV) chloride ($SnCl_4$), which we have recently reported,[2] gives the β-

---

(1) For recent work, see: (a) Ryu, I.; Suzuki, H.; Ogawa, A.; Kambe, N.; Sonoda, N. *Tetrahedron Lett.* **1988**, *29*, 6137. (b) Ikura, K.; Ryu, I.; Ogawa, A.; Kambe, N.; Sonoda, N. *Ibid.*, in press.

(2) Ryu, I.; Murai, S.; Sonoda, N. *J. Org. Chem.* **1986**, *51*, 2389.

0276-7333/90/2309-0277$02.50/0