*Inorg. Chem.* **1993**, *32*, 5394–5395

# Synthesis and Pyrolysis Studies of $[(t\text{-Bu})_2GaPR_2]_x$

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*Received December 17, 1992*

## Introduction

Although the search for an infrared window material with high durability and multispectral capability has been in progress for over three decades, results have not been satisfactory.[1] Zinc sulfide and zinc selenide are currently being used as compromise materials, especially for large-size hardware, aboard high-speed aircraft or infrared-guided missiles. Major disadvantages of zinc sulfide are its low durability and weak resistance to rain impact.[2]

Spherical, monodispersed, submicron zinc sulfide powders were recently prepared by homogeneous precipitation.[3] In order to improve the mechanical properties of zinc sulfide as an infrared window material, it was decided to impregnate ZnS powder with gallium phosphide. GaP was chosen because the GaP lattice matches that of ZnS, which may prevent microcrack formation in the densified ceramic object and it has a higher resistance to rain.[4] To impregnate ZnS, it would seem desirable to have the precursor in either a liquid or a solution form and to generate a precursor that contains Ga and P in a 1:1 ratio. Here, we report the preparation and characterization of $[(t\text{-Bu})_2GaP(i\text{-Pr})_2]_2$ (**1**) and $(t\text{-Bu})_2GaP(t\text{-Bu})_2$ (**2**). While this work was in progress, these compounds were described by Higa et al. in the abstracts of a recent meeting.[5] We also describe a mechanism for the pyrolysis of **1** to GaP which contrasts that reported earlier for the pyrolysis of the analogue $(t\text{-Bu})_2GaAs(t\text{-Bu})_2$.[6]

## Experimental Section

**General Procedures.** All reactions were carried out under an atmosphere of prepurified argon by using standard inert-atmosphere and Schlenk techniques.[7] Toluene, benzene, $Et_2O$, and *n*-pentane were distilled from Na/benzophenone under $N_2$. $^1H$, $^{13}C$, and $^{31}P$ spectra were recorded on a Nicolet NT-300 300-MHz, a Varian VXR-300 300-MHz, or a Bruker WM-200 200-MHz instrument. FTIR spectra were recorded on an IBM-ZR 98 spectrometer as KBr pellets or Nujol mulls. Mass spectra were obtained on a Finnigan 4000 instrument or a Kratos MS-50 spectrometer. $GaCl_3$, $ClP(i\text{-Pr})_2$, $LiAlH_4$, *n*-BuLi, and *t*-BuLi were purchased from Aldrich Chemical Co. $LiP(i\text{-Pr})_2$,[8,9] $LiP(t\text{-Bu})_2$,[8a,9] $(t\text{-Bu})_3Ga$,[10] and $ClGa(t\text{-Bu})_2$[11] were prepared by literature methods. Elemental analyses and molecular weight determinations were performed by Galbraith Laboratories, Knoxville, TN.

$(i\text{-Pr})_2PH$.[8] This compound was prepared by a literature method[8b] except that the reaction was refluxed for 4 h instead of 2 h and at the end of the reaction excess $LiAlH_4$ was carefully destroyed by methanol instead of a concentrated aqueous solution of $NH_4Cl$. As a a result, the yield improved from 42.7%[8b] to 65%. Bp: 115–118 °C (lit. 118.0–118.5,[8b] 119° [8a]). $^1H$ NMR ($CDCl_3$): $\delta$ 0.99 (dd, 12H, $CH_3$, $^3J_{CH} = 7.2$ Hz, $^3J_{PH} = 7.8$ Hz), 1.85 (dsept, 2H, CH). $^{31}P$ NMR (toluene-$d_8$): $\delta$ −25.36 (lit.[8c] $\delta$ −26.4). MS, *m/e* (relative intensity): 118 ($M^+$, 18), 103 (3, $M^+ - Me$), 76 (43, $M^+ - CMe_2$), 75 (7, $M^+ - i\text{-Pr}$), 43 (100, *i*-Pr). The MS closely matched that in the literature.[8b] IR (KBr), $cm^{-1}$: 2957 (s), 2865 (s), 2721 (w), 2267 (s) (P–H), 1463 (s), 1383 (m), 1261 (s), 1223 (m), 1150 (m), 1057 (m), 957 (m), 882 (m), 786.2 (m), 692 (m), 642 (m).

**$ClP(t\text{-Bu})_2$.**[12] $ClP(t\text{-Bu})_2$ was previously prepared by the reaction of $PCl_3$ with *t*-BuMgCl in 50–60% yield,[12a–c] and by the reaction of tetra-*tert*-butyldiphosphine with chlorobiphenyl although no yield was reported.[12d] The procedure used in this work led to a considerably increased yield. This compound was prepared by slow addition of 20 mL (34 mmol) of *t*-BuLi (1.7 M in pentane) to 17.0 mmol (2.33 g, 1.50 mL) of $PCl_3$ dissolved in 25 mL of dried, degassed pentane at −78 °C. The reaction was allowed to reach room temperature and then was stirred for an additional 4 h. Pentane was removed by distillation at ambient pressure and the product was purified by distillation under 0.3-Torr vacuum at 47–50 °C and obtained in 90% yield. $^1H$ NMR ($CDCl_3$): $\delta$ 1.16 (d, 36H, $^3J_{PH} = 12.3$ Hz). $^1H$ NMR ($C_6D_6$): $\delta$ 1.14 (d, $^3J_{PH} = 12.3$ Hz). $^{31}P$ NMR ($CDCl_3$): $\delta$ 147.3. The $^1H$ and $^{31}P$ NMR spectra favorably correspond to those given in the literature.[12b,12d] MS, *m/e* (relative intensity): 180 ($M^+$, 5), 57 (*t*-Bu, 100). IR (KBr) $cm^{-1}$: 2950 (s), 2862 (s), 2362 (vw), 2307 (m), 2274 (vw), 1469 (s), 1387 (m), 1260 (m), 1174 (m), 1097 (w), 1014 (m), 934 (w), 803 (s), 667 (w).

**$HP(t\text{-Bu})_2$.**[13] This compound was prepared analogously to $HP(i\text{-Pr})_2$, and in contrast to the literature report,[13a] saturated $NH_4Cl$ solution was not used. This led to an increase of the literature yield (60%[12a]) to 75%. $^1H$ NMR ($C_6D_6$): $\delta$ 1.16 (d, 18H, $^3J_{PH} = 11.4$ Hz), 3.16 (d, 2H, $^1J_{PH} = 198.9$ Hz). $^{13}C$ NMR ($C_6D_6$): $\delta$ 31.8 (d, $CH_3$, $^2J_{PC} = 13.4$ Hz). $^{31}P$ NMR ($C_6D_6$): $\delta$ 20.26. The $^1H$, $^{13}C$, and $^{31}P$ NMR data favorably correspond to the literature reports.[13b,c] MS, *m/e* (relative intensity): 146 ($M^+$, 9), 131 ($M^+ - Me$, 1), 57 (*t*-Bu, 100). The mass spectral data were in agreement with those in the literature.[9b] IR (KBr), $cm^{-1}$: 2950 (s), 2861 (s), 2710 (vw), 2274 (s), 1731 (w), 1470 (s), 1387 (w), 1245 (w), 1192 (m), 1122 (w), 1028 (m), 815 (m), 760 (w).

**Synthesis of $[(t\text{-Bu})_2GaP(i\text{-Pr})_2]_2$ (1).** To a mixture of 0.56 g (4.5 mmol) of LiP $(i\text{-Pr})_2$ and 1.00 g (4.50 mmol) of $ClGa(t\text{-Bu})_2$ was added about 25 mL of dry, degassed benzene. The mixture was stirred overnight at room temperature and then filtered. Benzene was removed under vacuum to give 1.1 g of white product in 80% yield, which was purified by sublimation at 90 °C and $5 \times 10^{-3}$ Torr. Mp: 151–153 °C (decomposing to yellow monomer and melt). $^1H$ NMR ($C_6D_6$): $\delta$ 1.41 (s, 18H, $(CH_3)_3C$), 1.47 (q, $(CH_3)_2C$, $^3J_{HH} = 6.6$ Hz), 2.67 (dsept, CH, $^2J_{PH} = 0.6$ Hz, $^3J_{HH} = 6.6$ Hz). $^{13}C$ NMR ($C_6D_6$): $\delta$ 26.64 (t, $CH_3)_2\mathit{C}$, $^2J_{PC} = 5.3$ Hz), 27.31 (t, $(\mathit{C}H_3)_2C$, $^3J_{PC} = 2.2$ Hz), 31.99 (s, $(CH_3)_3\mathit{C}$), 33.87 (br s, $(\mathit{CH}_3)_3C$). $^{31}P$ NMR ($C_6D_6$): $\delta$ 16.65. MS, *m/e* for the $^{69}Ga$ and $^{71}Ga$ isotopes (relative intensity): 547, 545, 543 (5, 14, 11, $M^+ - t\text{-Bu}$), 489, 487, 485 (1, 6, 13, $M^+ - 1 - 2t\text{-Bu}$), 374, 372 ($M^+ - 3t\text{-Bu}$, 2, 9), 373, 371, 369 (21, 67, 49), 303 (14), 302 (20), 301, 300 (20, 28, $^1/_2M^+$), 245, 243 (53, 73, $^1/_2M^+ - t\text{-Bu}$), 188, 186 (2, 9, $^1/_2M^+ - 2t\text{-Bu}$), 71, 69 (66, 100, Ga), 57 (67, *t*-Bu). CIMS ($NH_3$), *m/e* for the $^{69}Ga$ and $^{71}Ga$ isotopes (relative intensity): 604, 602, 600 (1, 1, 1, $M^+$), 547, 545, 543 (1, 1, 1, $M^+ - t\text{-Bu}$), 502, 500 498 (3, 3, 2, $M^+ - t\text{-Bu} - i\text{-Pr}$), 402, 400, 398 (6, 13, 10, $M^+ - 2t\text{-Bu} - 2i\text{-Pr}$), 303 (5), 302 (3),

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301, 300 (8, 2, $^1/_2$M), 117 (100, P(*i*-Pr)$_2$). Anal. Calcd for $\mathrm{C_{28}H_{64}Ga_2P_2}$: C, 55.85; H, 10.71. Found: C, 55.28; H, 10.15. The molecular weight of 637 was measured in benzene at a concentration of 0.054 M.

**Synthesis of (*t*-Bu)$_2$GaP(*t*-Bu)$_2$ (2).** The synthesis of this compound was analogous to that of **1** except that 1.1 g (5.0 mmol) of ClGa(*t*-Bu)$_2$ and 0.76 g (5.0 mmol) of LiP(*t*-Bu)$_2$ were used. A yellow oil was obtained that begins to sublime at room temperature at $10 \times 10^{-3}$ Torr. Sublimation was carried out at 60 °C and $10^{-2}$ Torr, however, and a yellow crystalline product of **2** was obtained in 80% yield. Mp: 41–43 °C. $^1$H NMR ($\mathrm{C_6D_6}$): $\delta$ 1.30 (d, 18H, $^3J_{\mathrm{PH}} = 11.5$ Hz, $\mathrm{PC(CH_3)_3}$), 1.28 (s, 18H, $\mathrm{GaC(CH_3)_3}$). $^1$H NMR ($\mathrm{C_7D_8}$): $\delta$ 1.23 (d, 18H, $^3J_{\mathrm{PH}} = 11.5$ Hz, $\mathrm{PC(CH_3)_3}$), 1.20 (s, 18H, $\mathrm{GaC(CH_3)_3}$). $^{13}$C NMR ($\mathrm{C_6D_6}$): $\delta$ 34.66 ($\mathrm{PC(\mathit{C}H_3)_3}$, $^2J_{\mathrm{PC}} = 13.4$ Hz), 32.25 ($\mathrm{P\mathit{C}(CH_3)_3}$, $^1J_{\mathrm{PC}} = 30.5$ Hz), 31.96 ($\mathrm{Ga\mathit{C}(CH_3)_3}$, 30.37 ($\mathrm{PGaC(\mathit{C}H_3)_3}$, $^4J_{\mathrm{PC}} = 2$ Hz). $^{13}$C NMR ($\mathrm{C_7D_8}$): $\delta$ 34.19 ($\mathrm{PC(\mathit{C}H_3)_3}$, $^2J_{\mathrm{PC}} = 13.4$ Hz), 31.68 ($\mathrm{P\mathit{C}(CH_3)_3}$, $^1J_{\mathrm{PC}} = 30.5$ Hz), 31.43 ($\mathrm{Ga\mathit{C}(CH_3)_3}$), 29.96 ($\mathrm{PGaC(\mathit{C}H_3)_3}$, $^4J_{\mathrm{PC}} = 2$ Hz). $^{31}$P NMR ($\mathrm{C_6D_6}$): $\delta$ 23.8. MS, *m/e* for the $^{69}$Ga and $^{71}$Ga isotopes (relative intensity): 330, 328 (M$^+$, 1, 1), 273, 271 (5, 8, M$^+$ – *t*-Bu), 216, 214 (3, 4, M$^+$ – 2*t*-Bu), 57 (100, *t*-Bu). Anal. Calcd for $\mathrm{C_{16}H_{36}GaP}$: C, 58.38; H, 11.02. Found: C, 56.29; H, 10.96. The molecular weight of 381 was measured in benzene at a concentration of 0.059 M.

**Pyrolysis Studies.** The thermolysis of compounds **1** and **2** was studied by three different methods. A sample was heated to 600 °C in a TGA apparatus (TG/DTA 300; Seiko Instrument Inc.) at a ramping rate of 10 °C/min in a flowing argon atmosphere using ultra high purity grade argon flowing at a rate of 55 mL/min through an $\mathrm{O_2}$ trap (Oxy-Trap, Alltech Associates Inc., Deerfield, IL). Compounds **1** and **2** were also pyrolyzed in a flash pyrolysis apparatus at a ramping rate of 300 °C/min. The material remaining after pyrolysis was characterized by X-ray powder diffraction using a Scintag XDS 2000 powder X-ray diffractometer. The powder was identified as GaP.[14] A convergent beam electron diffraction (CBED) pattern for the pyrolysis residue of **1** was observed at 300 kV on a Phillips CM30 S/TEM instrument. A pyrolysis tube was also interfaced with a VG SX300 quadrupole mass spectrometer using a 70-eV electron impact filament. The sample of **1** (250 mg) was heated over a period of 45 min, from 200 to 500 °C while mass spectral data were collected. Volatiles from the pyrolysis were also collected in a liquid-$\mathrm{N_2}$ cold trap for subsequent injection into a GC/IR/MS apparatus (HP-MS-5970-IRD-5965A).

## Discussion

The preparation of $\mathrm{(Me_2GaPR_2)_\mathit{x}}$ (R = Me, Et, Ph) via alkane elimination from the reaction of trimethylgallium with the corresponding $\mathrm{HPR_2}$ was reported in the 1960s.[15,16] A salt elimination method to prepare a number of GaP precursors from a mixture of lithium dialkylphosphide, $\mathrm{GaCl_3}$, and alkyllithium was subsequently described.[9a,17] In this study we prepared the gallium phosphide precursors **1** and **2**[5] by reacting $\mathrm{LiPR_2}$ (R = *i*-Pr, *t*-Bu) with ClGa(*t*-Bu)$_2$. During the preparation of ClGa(*t*-Bu)$_2$ from Ga(*t*-Bu)$_3$ and $\mathrm{GaCl_3}$, it is important to utilize a ratio of 2:1. Excess $\mathrm{GaCl_3}$ produces the byproduct $\mathrm{Cl_2Ga}$(*t*-Bu) (a chloride-bridged dimer) which is a solid at room temperature and a liquid at ~80 °C.[18] Its presence inhibited the sublimation of ClGa(*t*-Bu)$_2$.

Compound **2**, a yellow highly pyrophoric solid, is a monomer in the gas phase. The electron ionization mass spectrum contains peaks (*m/e*) due to the monomeric molecular ion (330, 328) and its fragmentation products. The CI/MS of the recently reported monomeric arsinogallane (*t*-Bu)$_2$GaAs(*t*-Bu)$_2$ (**3**)[6] shows peaks due to the molecular ion as well as higher mass peaks which were attributed to fragment recombination. For **2**, however, no peaks higher than 330 were observed. An osmometric molecular weight determination in benzene gave a value of 381 for **2** which is 16% higher than expected for a monomeric structure (calcd MW 329) in solution. Variable-temperature $^{13}$C NMR studies of **2** in toluene-$d_8$ revealed the appearance of a new $\mathrm{PC(\mathit{C}H_3)}$ doublet ($^2J_{\mathrm{PC}} = 28.9$ Hz) at 32.18 ppm and a change in the $\mathrm{GaC(\mathit{C}H_3)_3}$ doublet at 29.96 ppm to a broadened singlet. These spectral results are consistent with the presence of approximately 10% of a dimeric species at –70 °C and with about 15% dimer at room temperature, as indicated by the somewhat high molecular weight measurement which is accurate to only ±5%. Because the solid-state structure of (*t*-Bu)$_2$GaAs(*t*-Bu)$_2$ is monomeric,[6] the phosphorus analogue **2** is also expected to be monomeric in the solid state owing to the smaller size of the phosphorus atom compared with arsenic. Compound **1**, on the other hand, is more easily manipulated than **2** and is a dimer in the gas phase. Thus its CI ($\mathrm{NH_3}$) and EI ionization mass spectrum contains peaks ascribable only to a dimer. An osmometric measurement of the MW of **1** in benzene (637) suggests that it is also largely dimeric in solution (calcd MW 602). This dimer is easily purified by vacuum sublimation.

Pyrolysis of **1** or **2** under an argon atmosphere at ~450 °C resulted in the formation of GaP, as indicated by X-ray powder diffraction. This was confirmed in the case of the flash pyrolysis product of **1** by CBED, which revealed the expected cubic lattice and ratios of *d* spacings that matched those of GaP within 0.2% error. Compound **1** was also studied by TGA. Decomposition begins at 150 °C, and at about 400 °C it appears to be complete. A 66.3% overall weight loss was recorded, which corresponds well to the theoretical weight loss of 66.5%. The *tert*-butyl and isopropyl groups in these precursors can undergo $\beta$-elimination reactions,[6] thus resulting in lower carbon contents. The elemental analysis of the products of thermolysis of **1** and **2** reveals the presence of 5 and 2% carbon, respectively. This compares with the 10% carbon present after the solid–state pyrolysis of [*t*-$\mathrm{Bu_2Ga(\mu\text{-}PH_2)]_3}$ under 100-Torr pressure of $\mathrm{N_2}$ in a static system.[19] ZnS impregnation experiments with **1** and **2** are underway.

Mass spectral analysis of the gaseous products of the pyrolysis of **1**[20] revealed the presence of isobutene, isobutane, propene, propane, and hydrogen. From the GC/MS analysis of the pyrolysis condensate, the isobutene:isobutane and propene:propane ratios are each approximately 10:1. This result contrasts that observed for the pyrolysis of (*t*-Bu)$_2$GaAs(*t*-Bu)$_2$, wherein the isobutene:isobutane ratio was observed to be approximately 1:1.[6] One rationale put forth for this ratio was that $\beta$ elimination is followed by alkane elimination.[6] The high alkene to alkane ratio in our case can be ascribed to $\beta$ elimination of isobutene and propene, along with hydrogen, as the dominant pathway. If a free-radical pathway (involving alkyl radical formation followed mainly by hydrogen radical and alkene production) makes a significant contribution in our case, the detection of radical-coupling products such as $\mathrm{Me_3CCMe_3}$, $\mathrm{Me_3CCHMe_2}$, and $\mathrm{Me_2HCCHMe_2}$ might be expected, but no evidence for these products was observed.

**Acknowledgment.** This work was supported in part by the Office of Naval Research (Contract No. N00014-91-J-1974), Iowa State University, and Ames Laboratory (U.S. DOE). We thank Mr. E. Todd Voiles for experimental assistance.

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(20) Compound **2** was too pyrophoric to be successfully introduced into the VG SX300 or the GC/IR/MS.