The Crystal and Molecular Structure of 1,2-Bis-(diphenylphosphino)ethane-bis(trimethylgallium)

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The ligand 1,2-bis(diphenylphosphino)ethane (diphos) forms weak complexes with several group III metal alkyls; the complexes readily undergo thermal dissociation and since the ligand has low volatility at the adduct's dissociation temperature, the dissociative process effects a means of purification of the Lewis acids. The volatile metal aklys $(CH_3)_3M$ (M = Ga, In) which are used in synthesizing III–V compounds may be purified by this method [1]. We here report the crystal structure of the gallium compound.

Experimental

The adduct was prepared by mixing benzene solutions of trimethylgallium and diphos *in vacuo*. Solvent was then removed to concentrate the solution and colourless crystals, suitable for X-ray analysis, were deposited. The structure of $(C_6H_5)_2PCH_2CH_2P(C_6H_5)_2\{Ga(CH_3)_3\}_2$ was solved by a single crystal X-ray study.

Crystal Data (20 °C)

Triclinic, space group $P\bar{1}$, $a = 8.970(5)$, $b = 11.94(1)$, $c = 9.935(5)$ Å, $\alpha = 118.3(1)$, $\beta = 65.8(1)$, $\gamma = 103.2(1)°$, $\mu = 1.62$ mm^{-1}. Intensity measurements were made on a Stoe STADI-2 diffractometer using Mo Kα radiation. 3299 reflections were measured, and after elimination of those for which $I < 3\sigma(I)$ there remained 2220 unique reflections which were used in the final refinement. The structure was solved using the SHELX suite of programs [2]. Ga atom positions, obtained from Patterson maps, were used to phase $(F_o - F_c)$ maps to give the positions of the remaining atoms (except hydrogen). Full matrix least-squares refinement of all atomic positions, anisotropic U_{ij} for Ga and P, and isotropic U values for C were carried out until convergence was reached at $R = 0.078$. Fractional atomic coordinates are given in Table I. See also 'Supplementary Material'.

Results and Discussion

The X-ray crystallographic study revealed the presence of discrete molecules in the solid state (Fig. 1).

TABLE I. Fractional Atomic Coordinates (×10^4)

Atom	*x*	*y*	*z*
C(1)	5011(13)	2041(10)	4104(13)
C(2)	3902(16)	1459(12)	5139(16)
C(3)	4186(18)	1558(14)	6542(18)
C(4)	5582(18)	2246(14)	6870(18)
C(5)	6662(18)	2860(14)	5878(18)
C(6)	6375(15)	2743(12)	4473(16)
C(7)	5511(15)	3992(12)	1691(15)
C(8)	6004(12)	2807(9)	1336(12)
C(9)	7562(14)	2399(11)	233(15)
C(10)	8638(16)	3163(13)	−586(17)
C(11)	8131(16)	4338(12)	−249(16)
C(12)	6596(17)	4744(13)	888(17)
C(13)	5108(13)	191(10)	838(13)
C(14)	1537(21)	2593(17)	1195(24)
C(15)	289(21)	705(16)	3405(21)
C(16)	1391(19)	3913(15)	5318(19)
Ga	1545(2)	2321(1)	3161(2)
P	4522(3)	1834(3)	2306(3)

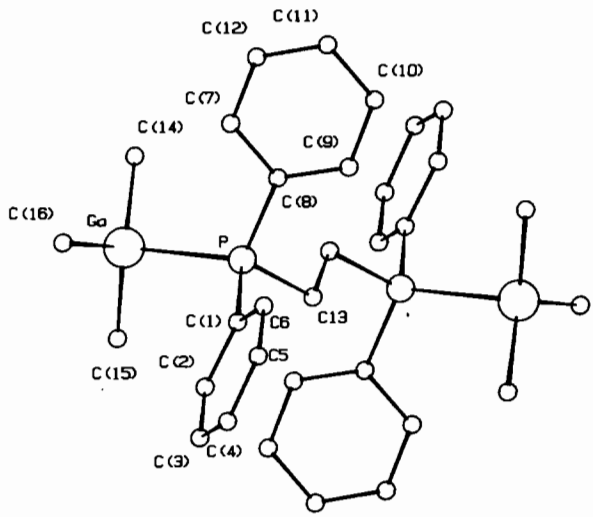

Fig. 1. The molecular structure of 1,2-bis(diphenylphosphino)ethane-bis(trimethylgallium).

The complex has a centre of symmetry imposed crystallographically and there are no short intermolecular contacts. Selected bond distances and angles are given in Table II. The weak interaction between $Ga(CH_3)_3$ and the Lewis base is reflected in the long Ga–P bond and the large C–Ga–C bond angles (average 116.3°). The Ga–P distance (2.563(3) Å) greatly exceeds that observed in $(CH_3)_3P$–$GaCl_3$ (2.353(2) Å) [3] which is close to the sum of the single-bond covalent radii for Ga and P (2.30 Å). The Ga–P bond is slightly longer than observed in the analogous bis(diphenylphosphino)methane–chlorodimethylgallium (2.535(2) Å) [4].

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TABLE II. Selected Bond Distances (Å) and Angles (°) in Diphos·2Ga(CH_3)$_3$

Distances			
Ga–P	2.563(3)	Ga–C(14)	2.13(2)
Ga–C(15)	2.07(2)	Ga–C(16)	2.06(2)
P–C(1)	1.90(1)	P–C(8)	1.83(1)
P–C(13)	1.84(1)	C(13)–C(13′)	1.58(2)
Angles			
P–Ga–C(16)	97.9(4)	C(14)–Ga–C(16)	117.4(7)
P–Ga–C(15)	102.2(5)	C(14)–Ga–C(15)	116.1(7)
P–Ga–C(14)	103.7(5)	C(15)–Ga–C(16)	115.3(7)
C(8)–P–C(1)	108.6(5)	C(13)–P–C(1)	102.1(5)
C(13)–P–C(8)	104.0(5)	Ga–P–C(1)	111.8(3)
Ga–P–C(8)	113.2(3)	Ga–P–C(13)	116.2(4)

Supplementary Material

Lists of structure factors and thermal parameters are available on request from the authors.

References

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