P-P Bond Activation of P_4 Tetrahedron by Group 13 Carbenoid and its Bis Molybdenum Pentacarbonyl Adduct

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Supporting Information

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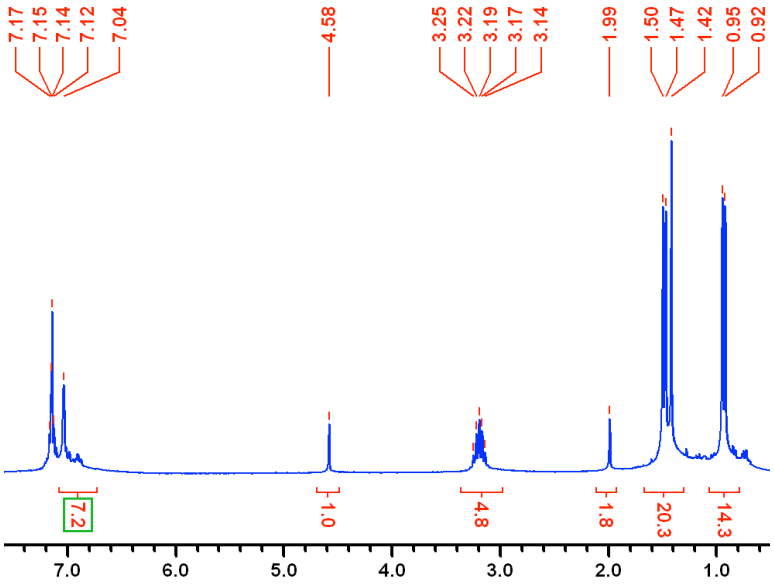

Figure S1. ^{1}H NMR spectrum of **1** in C_6D_6 at RT.

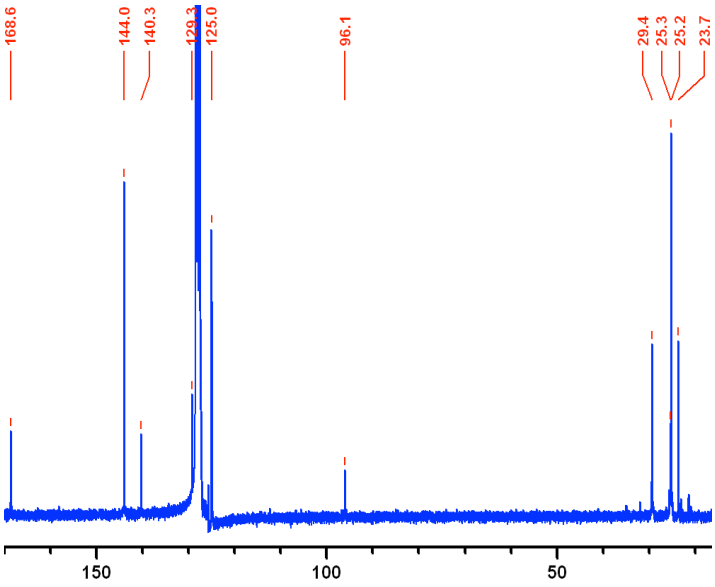

Figure S2. ^{13}C NMR spectrum of **1** in C_6D_6 at RT.

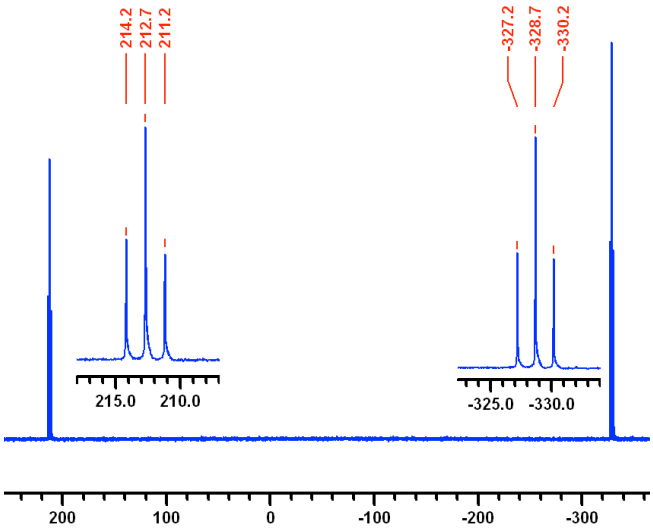

Figure S3. ^{31}P NMR spectrum of **1** in C_6D_6 at RT.

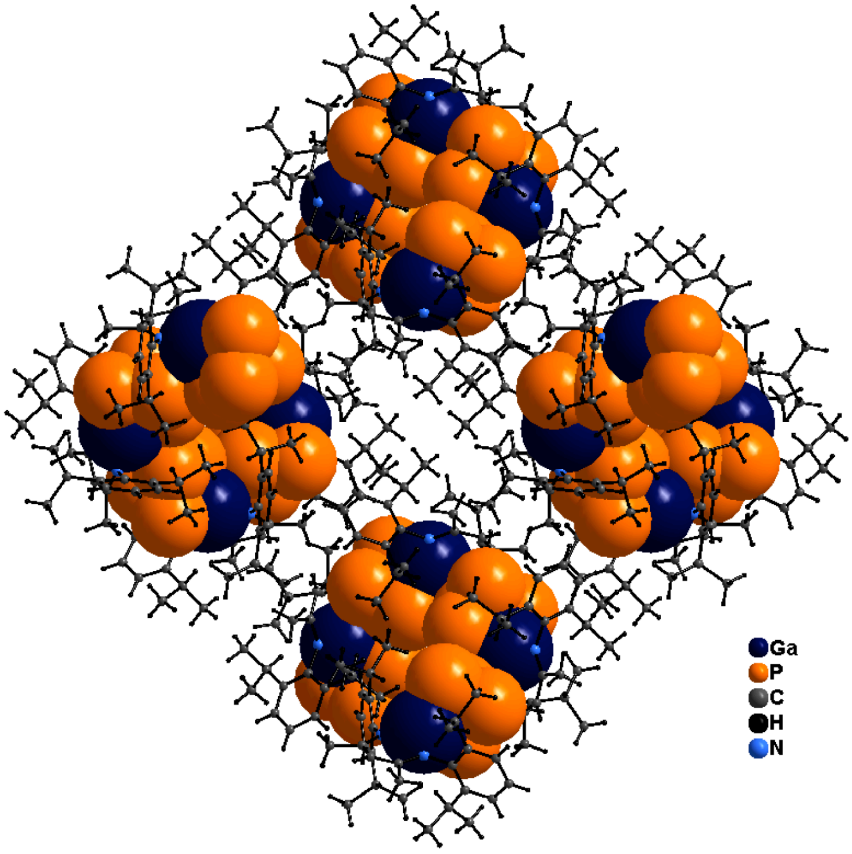

Figure S4. Molecular packing of **1** (view along X-axis).

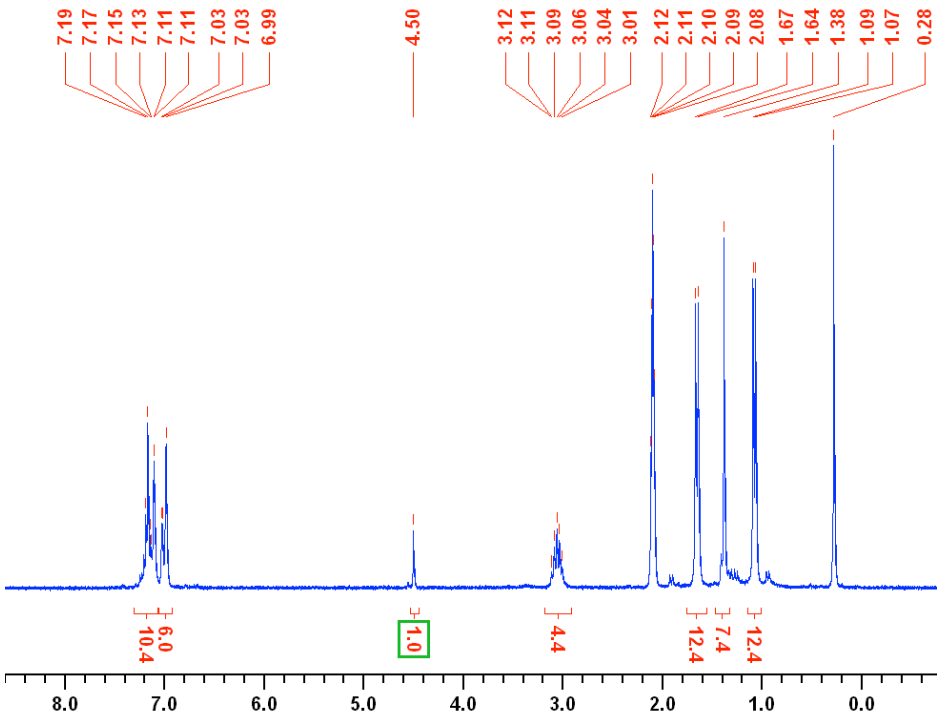

Figure S5. ^{1}H NMR spectrum of **2** in toluene-d_8 at RT.

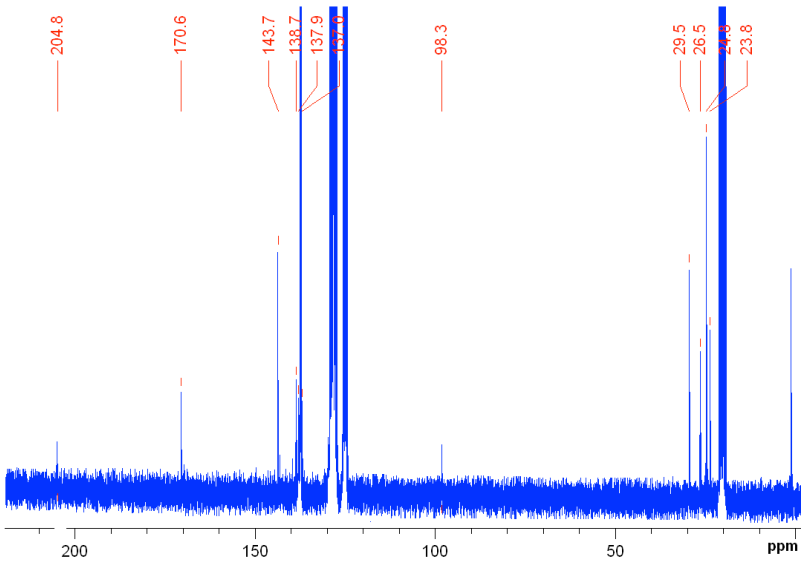

Figure S6. ^{13}C NMR spectrum of **2** in toluene-d_8 at RT. Although two carbonyl signals are expected for molecule **2,** the additional signal was not observed. The similar observation also reported in the literature.[1] Attempt to increase the sample concentration was not successful due to the poor solubility of **2** in toluene-d_8 at RT.

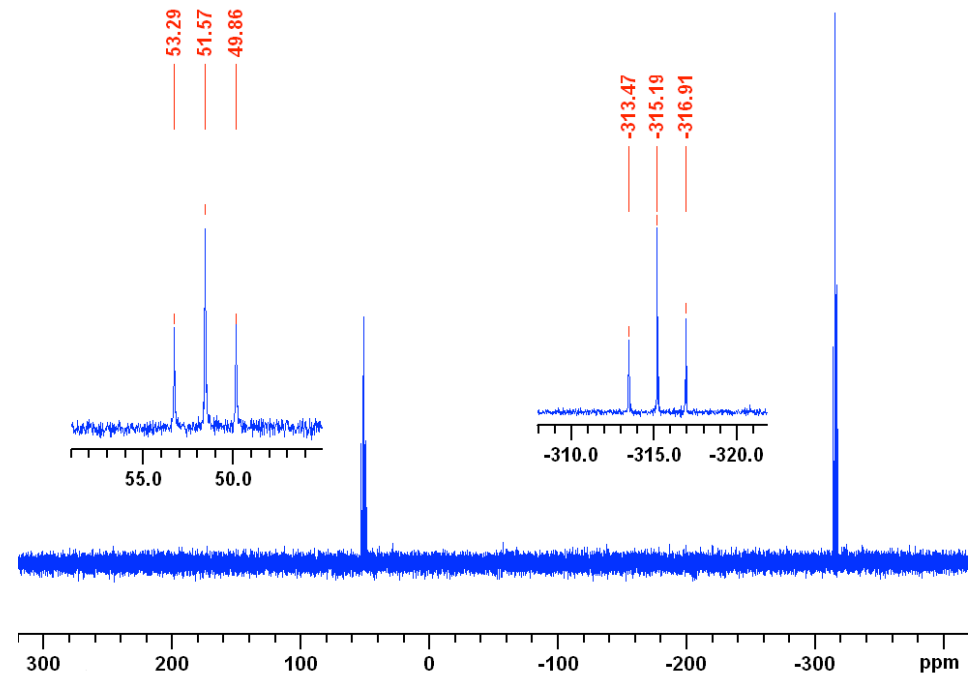

Figure S7. ^{31}P NMR spectrum of **2** in C_6D_6 at RT.

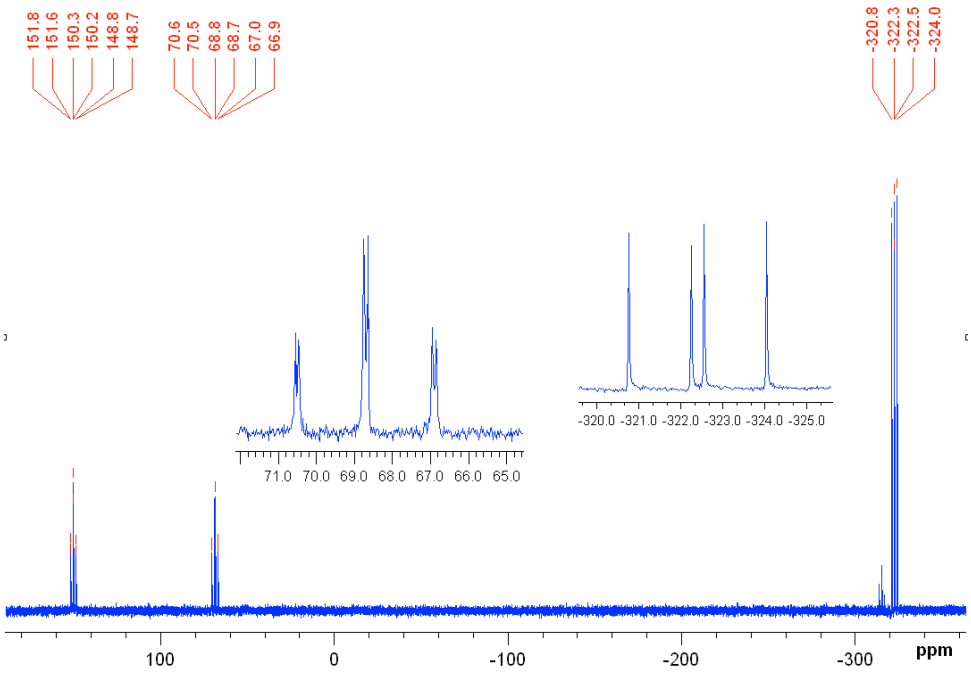

Figure S8: ^{31}P NMR spectrum of mother liquor of **2** in C_6D_6 at RT.

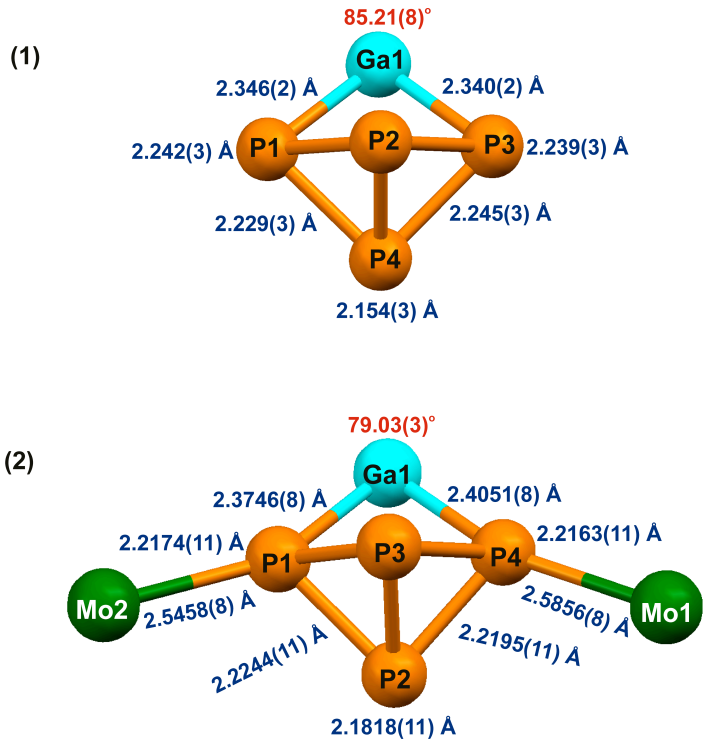

Figure S9. The selected bond lengths and angles comparison of GaP_4 core in **1** and **2**.

Table 1. Crystal data and structure refinement for **1** and **2**.[a]

	1	**2**
formula	$C_{29}H_{41}GaN_2P_4$	$C_{39}H_{41}GaMo_2N_2O_{10}P_4$
mol wt	611.24	1083.22
data / restraints / parameters	6462 / 0 / 325	10628 / 0 / 513
data coll, T, K	113(2)	107(2)
wavelength, Å	0.71073	0.71073
crystal system, space group	tetragonal, $P4_32_12$	Monoclinic, $P2_1/n$
a, Å	19.9661(7)	13.8185(2)
b, Å	19.9661(7)	11.1546(2)
c, Å	18.6059(15)	39.2533(7)
β, deg	-	91.703(2)

V, A^3	7417.2(7)	6047.82(18)
Z, ϱ (Calcd) Mg/m^3	8, 1.095	4, 1.190
μ, mm^{-1}	0.932	0.997
F(000)	2560	2176
crystal size, mm	0.21 x 0.09 x 0.07	0.17 x 0.15 x 0.12
θ range, deg	2.99 to 25.00	2.95 to 25.00
reflections collected/unique	14147/6462	67025 / 10628
	[R(int) = 0.0898]	[R(int) = 0.0292]
observed reflns[I>2sigma(I)]	3574 Fo > 4sig(Fo)	8906 Fo > 4sig(Fo)
completeness to theta = 25.00, %	99.5	99.8
absorption correction	Empirical	Empirical
max. and min. transmission	0.930 and 0.900	0.8897 and 0.8488
goodness-of-fit on F^2	0.873	1.060
final R indices [I>2sigma(I)]	R1 = 0.0570,	R1 = 0.0303,
	wR2 = 0.1330	wR2 = 0.0875
R indices (all data)	R1 = 0.0966,	R1 = 0.0382,
	wR2 = 0.1400	wR2 = 0.0894
largest diff. peak and hole, $e.A^{-3}$	1.275 and -0.421	0.482 and -0.395

a: refinement method - Full-matrix least-squares on F^2;

1. J. Grobe, D. Le Van, *Z. Anorg. Allg. Chem.* **1984,** *518*, 36-54.