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- 2-Phenyl-1,3-disilapropane (1): GC/MS (m/e (relative intensity)): 152 (40) (M⁺), 121 (53), 120 (100), 119 (38), 105 (33), 93 (19), 91 (15), 53 (12).
Benzylsilane: GC/MS (m/e (relative intensity)): 122 (30) (M⁺), 121 (45), 119 (10), 105 (100), 93 (50), 91 (20), 53 (10).
1-Phenyl-3,5-disilapentane (2): GC/MS (m/e (relative intensity)): 180 (20) (M⁺), 163 (1), 152 (19), 149 (31), 135 (41), 119 (2), 102 (100), 91 (45), 77 (20), 74 (80), 71 (20), 53 (81), 51 (10).
1-Phenyl-4-silyl-3,5-disilapentane (3): GC/MS (m/e (relative intensity)): 210 (20) (M⁺), 179 (35), 154 (10), 147 (12), 130 (80), 105 (100), 91 (50), 77 (25), 73 (60), 71 (50), 65 (20), 51 (12).
Phenethylsilane: GC/MS (m/e (relative intensity)): 136 (62) (M⁺), 133 (18), 119 (1), 108 (80), 105 (75), 91 (100), 77 (30), 65 (20), 58 (21), 51 (20).
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Synthesis and Characterization of Dicationic Diammine Complex of Palladium(II) Chelating with DPPE

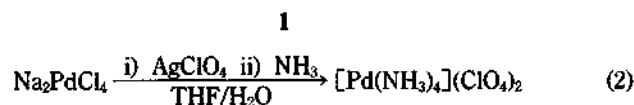
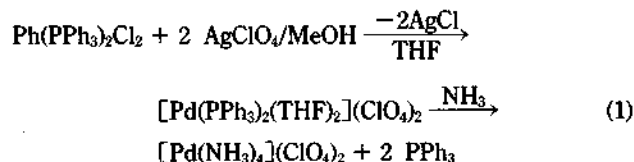
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Although ammine complexes of transition metals have been known since the early history in the classical coordination chemistry, such complexes containing tertiary phosphines or organic moieties are relatively rare.¹ Recently there has been an increasing interest in the amido complexes of transition metals because of their potential applications for catalysis.² One class in this category is such complexes of late transition metals having unsubstituted amide ligand NH₂.³ Ammine complexes having tertiary phosphines are useful synthetic precursors for unsubstituted amido complexes in which the coordinated tertiary phosphines can stabilize electronically and sterically with a variety of substituents.⁴ Ammonia is a very weak acid (*pK_a*=33) and the N-H bond dissociation energy in ammonia is very high (107 kcal/mol).⁵ Ammonia upon coordination to cationic metal center, however, becomes considerably acidic to be deprotonated by appropriate base.

Reported here is the synthesis and characterization of a novel dicationic palladium(II) diammine complex containing 1,2-bis(diphenylphosphino)ethane as a supporting ligand. Our initial attempt to synthesize dicationic diammine complex having monodentate bis-triphenylphosphine has been proven not successful. When gaseous ammonia was added to a THF solution of [Pd(PPh₃)₂(THF)](ClO₄)₂ prepared *in situ* by the reaction of PdCl₂(PPh₃)₂ and 2 equivalents of AgClO₄, dicationic tetraammine complex [Pd(NH₃)₄](ClO₄)₂ (1) was obtained (eq. 1). The formation of 1 can be explained by a sequence of reactions involving the initial formation of [Pd(PPh₃)₂(NH₃)₂](ClO₄)₂, and then further substitution of coordinated triphenylphosphines with excess ammonia. We can not observe the intermediate complex [Pd(PPh₃)₂(NH₃)₂](ClO₄)₂. The formulation of 1 has been confirmed by its independent synthesis from the reaction of aqueous THF solution of [PdCl₄]²⁻, 4 equivalents of AgClO₄ and ammonia (eq. 2).⁶



Targeting diammine complex of bis-phosphine palladium (II) has been successfully prepared by employing 1,2-bis(diphenylphosphino)ethane as chelating ligand in the displace-

7. Spectroscopic data for 2: IR (KBr pellet) $\nu(\text{NH})=3180$, 3250 , 3330 cm^{-1} (w, br), $\nu(\text{SO})=1260$, 1160 cm^{-1} (vs, br); ^1H NMR (CDCl_3) δ 3.02 br (6H, NH_3), δ 2.76m (4H, CH_2), δ 7.6-7.8m (20H, phenyl); $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3) δ 64.83 s. Anal. Calcd for $\text{C}_{30}\text{H}_{38}\text{F}_6\text{N}_2\text{O}_{6.5}\text{PdS}_2$: C, 41.22; H, 4.04; N 3.20; S, 7.34. Found: C, 40.93; H, 4.21; N, 3.27; S, 7.37.
8. The ^1H NMR resonance for the coordinated NH_3 in the platinum complex $[\text{Pt}(\text{NH}_3)\text{Me}(\text{DPPE})]^+$ was observed at δ 3.46, see ref 3 (f).
9. For the complex $[\text{PdL}_3(\text{NH}_3)]^+$ ($\text{L}_3=2,6\text{-bis}(\text{dicyclohexylphosphinomethyl})\text{phenyl}$), the coordinated ammonia slowly undergoes substitution reaction with water; unpublished result in this laboratory.
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