

mol. wt., 897.8. Found: B, 3.51; C, 77.04; Cl, 0.00; N, 4.85; Si, 9.19; mol. wt., 881.2. Calcd. for  $B_3[Si(C_6H_5)_3]_3N_3(C_6H_5)_3$ : B, 2.99; C, 79.76; Cl, 0.00; N, 3.88; Si, 7.77; mol. wt., 1084. Found: B, 3.02; C, 79.70; Cl, 0.00; N, 3.98; Si, 7.50; mol. wt., 1052.

In addition to the method of synthesis and analysis, the assignment of a substituted borazine structure to these compounds is supported by the molecular weights, determined cryoscopically in benzene, and by certain features of their infrared spectra in carbon tetrachloride. The salient features are: bands in the  $7\mu$  region characteristic of the borazine ring<sup>3</sup>

N-Trimethyl compound: 6.94 (s) 7.02 (ms) 7.27 (s)  
N-Triphenyl compound: 6.68 (ms) 7.00 (s) 7.35 (vs)

Both compounds have absorption at  $14.4\mu$  which is due to a monosubstituted phenyl group.<sup>4</sup> Also, the C-H band at about  $3.5\mu$  is diffuse and seems to be characteristic of the triphenylsilyl group.<sup>5</sup>

Both compounds react with moist air, but do not take up dry oxygen at room temperature or at  $160^\circ$ . Reaction with bromine proceeds readily in carbon tetrachloride and at room temperature, producing triphenylbromosilane as the only soluble product (identified by infrared spectrum and bromine analysis) and a precipitate which does not exhibit the borazine ring frequency in a KBr pellet infrared spectrum. This suggests that ring cleavage had occurred.

(3) W. C. Price, R. P. B. Fraser, T. S. Robinson and H. C. Longuet-Higgins, *Disc. Faraday Soc.*, **9**, 131 (1950).

(4) H. M. Randall, "Infrared Determination of Organic Structures," D. Van Nostrand Co., New York, N. Y., 1949.

(5) The authors found that the infrared spectrum of hexaphenyldisilane had this same flattening effect.

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#### THE SYNTHESIS AND STRUCTURE OF BIS-(CYCLOPENTADIENYLNICKEL)-ACETYLENE

Sir:

The first synthesis of an acetylene complex of nickel is herein reported, bis-(cyclopentadienyl-nickel)-acetylene being a product of the reaction between nickelocene and acetylene. Alkyne metal complexes in which the alkyne as such is bonded via its  $\pi$  electron system have been reported with the cobalt<sup>1</sup> and iron carbonyls<sup>2</sup> and various derivatives of platinum.<sup>3,4</sup> Recently certain alkyne nickel complexes have been reported as the products of reaction between alkynes and dicyclopentadienylnickel dicarbonyl.<sup>5</sup> The isolation of bis-(cyclopentadienylnickel)-acetylene through a dif-

(1) H. W. Sternberg, H. Greenfield, R. A. Friedel, J. Wotiz, H. R. Markby and I. Wender, *THIS JOURNAL*, **76**, 1457 (1954); **78**, 120 (1956).

(2) W. Hubel and E. H. Braye, *J. Inorg. Nucl. Chem.*, **10**, 250 (1959).

(3) S. V. Bukhovets and K. A. Molodava, *Zhur. Neorg. Khim.*, **2**, 776 (1957); S. V. Bukhovets and N. K. Pukhova, *ibid.*, **3**, 1714 (1958).

(4) J. Chatt, C. A. Rowe and A. A. Williams, *Proc. Chem. Soc.*, 208 (1957); J. Chatt, L. A. Duncanson and R. G. Guy, *Chem. and Ind.*, 430 (1959).

(5) J. F. Tilney-Bassett and O. S. Mills, *THIS JOURNAL*, **81**, 4757 (1959).

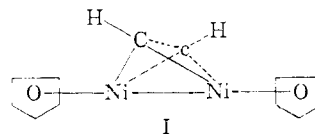
ferent route represents the first member of this novel series of nickel alkyne complexes.

Bis-(cyclopentadienylnickel)-acetylene was prepared by contacting 18 g. of nickelocene dissolved in tetrahydrofuran with acetylene at 180 p.s.i. and  $80^\circ$  for 15 hours. The reaction mixture was filtered and the solvent was removed at reduced pressure. Fractional vacuum sublimation of the dark green residue resulted in a recovery of 5.5 g. (30%) of unreacted nickelocene and the isolation of a less volatile dark green crystalline solid which crystallized from petroleum ether at  $-60^\circ$  as light green lustrous plates m.p.  $143-144^\circ$  (dec.).

*Anal.* Calcd. for  $(C_5H_5Ni)_2$ : C, 52.7; H, 4.39; Ni, 42.9. Found: C, 52.3; H, 4.49; Ni, 42.6.

A Signer molecular weight determination of 290 supported the above written empirical formula. The total yield of product was 6.3 g. (48%).

The product exhibits moderate solubility in saturated hydrocarbons but is very soluble in all other common organic solvents. Oxidatively, it appears to be more stable than nickelocene and it can be handled readily in air for short periods of time. The infrared spectrum of the material contains bands characteristic of a  $\pi$  bonded cyclopentadienyl metal grouping. Magnetic susceptibility measurements<sup>6</sup> indicate that the product is diamagnetic which suggests that nickel has attained rare gas electronic structure. On the basis of the above data, Structure I corresponding to bis-(cyclopentadienylnickel)-acetylene is proposed in which acetylene is bonded via four  $\pi$  electrons to a binuclear cyclopentadienyl nickel system. The



exact orientation of the acetylene carbons relative to the binuclear nickel bonding is unknown. However, in view of the recent X-ray studies by Sly<sup>7</sup> with the binuclear cobalt carbonyl complex of diphenylacetylene, the bonds probably are at right angles. The proposed structure is in agreement with the structures of the alkyne nickel complexes as outlined by Tilney-Bassett and Mills.

The extension of the above reaction to substituted alkynes will be discussed in forthcoming publications.

(6) We are indebted to Dr. Stanley Kirschner of Wayne State University for carrying out the magnetic measurements.

(7) W. G. Sly, *THIS JOURNAL*, **81**, 18 (1959).

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#### SYNTHESIS OF THE LUPININE SYSTEM PATTERNED AFTER THE BIOGENETIC SCHEME OF SCHÖPF AND ROBINSON

Sir:

Consideration of structural relationships leads to the inference that the simple lupin alkaloids (III) arise in nature by Mannich cyclization of the amine dialdehyde I, formed in the plant by