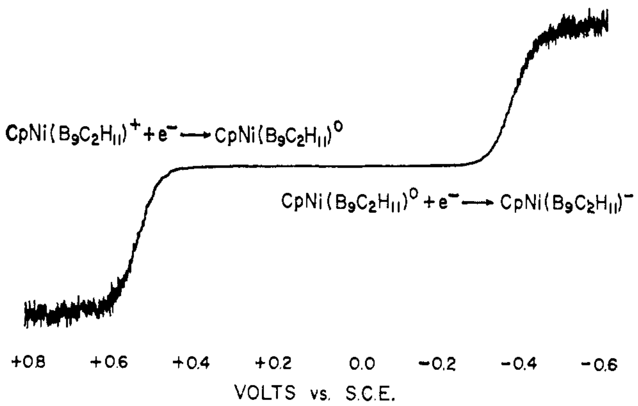

Figure 2. Rotating platinum polarogram of $(\pi\text{-}C_5H_5)Ni[\pi\text{-}(3)\text{-}1,2\text{-}B_9C_2H_{11}]$ run in acetonitrile solution at room temperature.

$C_5H_5NiB_9C_2H_{11}$: C, 32.82; H, 6.29; B, 37.98; Ni, 22.91. Found: C, 31.18; H, 6.19; B, 36.73; Ni, 21.44. High-resolution mass spectroscopy verified the formulation: Calcd for $^{60}Ni^{11}B_9{}^{12}C_7{}^1H_{16}$, 259.144; found, 259.142. The mixed ligand compound is electrochemically similar to the two analogous nickel systems discussed above; cyclic voltammetry gave two reversible redox couples (Table I). The polarogram (rotating platinum electrode) of **1** at room temperature in acetonitrile is shown in Figure 2. Here again the presence of a formal d^6 Ni(IV) species, which must be the cation $(\pi\text{-}C_5H_5)Ni[\pi\text{-}(3)\text{-}1,2\text{-}B_9C_2H_{11}]^+$, is demonstrated. The high reduction potential of the Ni(IV) complex, $E_{1/2} = +0.55$ V, indicates that the species is strongly oxidizing, and attempts to isolate it, chemically and electrochemically, have as yet been unsuccessful. The cation decomposed slowly in acetonitrile at room temperature, though much more slowly than its less stable bis(cyclopentadienyl)nickel(IV) analog.

Although several cationic boron hydride derivatives containing one boron atom[7] have been synthesized, with few exceptions, all the higher boron hydrides, polyhedral boranes, carboranes, etc., have been exclusively anionic or electrically neutral species.[8] However, electrochemistry has shown here that the driving force of a metal to attain its inert gas configuration apparently overcomes this charge precedence established by the electron-deficient boron hydrides.

The lowering of the Ni(IV) reduction potentials with successive dicarbollide ion substitution for cyclopentadienide ion is seen in Table I. This effect appears to be due to the higher formal negative charge of the dicarbollide ligand (−2, compared to −1 for cyclopentadienide), resulting in a less highly charged, hence more stable, formal Ni(IV) complex. A similar correlation regarding the lower +2 oxidation state cannot be made; the presence of one dicarbollide ligand makes the Ni(II) species obtained from **1** very strongly reducing compared to nickelocene, while the presence of two dicarbollide ion ligands does not appreciably alter the potential of this oxidation state.

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[8] For two exceptions, see W. R. Hertler, W. K. Knoth, and E. L. Muetterties, *Inorg. Chem.*, **4**, 288 (1965), regarding the protonated amino complex, $[(CH_3)_2SB_{10}H_7NH_3 \cdot N_2]^+$, and E. L. Muetterties and W. H. Knoth, "Polyhedral Boranes," Marcel Dekker, Inc., New York, N. Y., 1968, p 110, concerning the $B_{10}H_7[S(CH_3)_2]_3{}^+$ cation.

(9) U. S. Public Health Service Predoctoral Fellow.

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Determination of Specific Surface Areas of Powders by Means of Dyes

Sir:

Van den Hul and Lyklema report[1] measurements of specific surface of samples of silver iodide by several methods. The values from dye adsorption differ considerably from those obtained by nitrogen adsorption. They used two cationic dyes: methylene blue (MB) and diethyl-2,2′-cyanine (DEC). They assumed flatwise adsorption of both dyes and a projected molecular area of 108 Å^2 for MB.

Investigations here[2] have shown that the best results with adsorption of cationic dyes are given if (a) the molecular area is that of the smallest enclosing rectangle rather than the actual projected area, and (b) account is taken of the fact that the dyes are adsorbed as ionic micelles from water. Thus for MB, 120 Å^2 is the best molecular area, and since this dye is adsorbed as a dimer, the specific surface thus calculated must be divided by two.

If the results given by Van den Hul and Lyklema are recalculated in this way, they are found to be in much better agreement with their nitrogen results, as shown in Table I. The lower results quoted by Van den Hul and Lyklema for DEC, compared with nitrogen data, are readily accounted for by the fact that they used ethanolic solution. This solvent would disaggregate the dye and interfere with its adsorption.

Table I

Sample	Specific surface, m^2/g N_2	Methylene blue Quoted[1]	Recalcd
A1	0.45	0.87	0.48
A2	0.38	0.61	0.34
B2	0.52	0.98	0.54
B3	0.30	0.65	0.36
C1	0.97	2.18	1.21
C2	1.99	2.14	1.19

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(1) H. J. Van den Hul and J. Lyklema, *J. Am. Chem. Soc.*, **90**, 3010 (1968).

(2) C. H. Giles, I. A. Easton, R. B. McKay, C. C. Patel, N. B. Shah, and D. Smith, *Trans. Faraday Soc.*, **62**, 1963 (1966).

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