A simplified form of the relationship discussed in the previous paragraph is that the ratio of D values for the triplet and the corresponding quintets should be approximately constant for a series of *meta* quintets. We find $D_{\mathrm{Va}}/D_{\mathrm{IIIa}} = 5.8$, $D_{\mathrm{Vb}}/D_{\mathrm{IIIb}} = 6.1$, and $D_{\mathrm{VI}}/D_{\mathrm{IV}} = 6.4$.

The theoretical value of the D/E ratio should be ~ 5 for IV or for IIIa,b if the bonds to the divalent carbon are colinear.[9,10] With IIIb, the one case in which E was determined directly, the ratio is 3.6. A possible explanation for this lower value lies in the potentiality for isomers in the methylenes as these are known to be bent about the divalent carbon atom.[4,13] Simple theoretical arguments[9] indicate that $D/E < 5$, ~ 5, >5 for IIIb, VII, and VIII, respectively. Thus the absorptions used for the assignment of parameters may arise from the particular isomer IIIb.

The possibility exists that some of the epr lines are due to the quartet states such as IX which could be formally produced from IIIb. Arguments analogous to those used above to relate the parameters of the quintet to those of the corresponding triplet may be used to relate the quartet to those of a triplet and doublet.[9,10] The predicted values for the quartet state should not allow lines at as high or low magnetic fields as we observe experimentally. While quartet states may be present in the observed systems, the assignments we are making are most probably based on quintet resonances.

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Electrochemical Oxidation of $B_{12}H_{12}^{2-}$ [1]

Sir:

Recent work[2] has shown that the electrochemical oxidation of the $B_{10}H_{10}^{2-}$ ion in acetonitrile parallels the aqueous chemical oxidation,[3-5] and that controlled-potential electrolysis is a useful method of carrying out the oxidative coupling of $B_{10}H_{10}^{2-}$ and its derivatives.[6] Previous attempts to oxidize $B_{12}H_{12}^{2-}$ in aqueous solution have been unsuccessful or have yielded only borates as products.[7,8] We now report the controlled-potential oxidation of $B_{12}H_{12}^{2-}$ in acetonitrile.

Voltammetry of $[(C_2H_5)_4N]_2B_{12}H_{12}$ at a rotating platinum electrode in acetonitrile (0.1 M tetraethylammonium perchlorate as supporting electrolyte) shows an anodic wave with $E_{1/2} = +1.5$ v (*vs.* sce). At a stationary platinum electrode, an anodic peak is observed at +1.5 v, with a shoulder at +1.85 v on a large anodic wave. In a typical run, 30 mmoles of $Na_2B_{12}H_{12}$ in 150 ml of acetonitrile was exhaustively electrolyzed under nitrogen at +1.45 v, using a graphite cloth anode, with no supporting electrolyte. The current was monitored using a strip chart recorder; the value $n = 0.91$ equiv/mole of $Na_2B_{12}H_{12}$ was found. The yellow solution was evaporated to dryness at room temperature, and the residue was dissolved in H_2O. Addition of CsF to the aqueous solution gave white crystals of $Cs_3B_{24}H_{23} \cdot 3H_2O$ (I) which were recrystallized from an acetonitrile–water mixture.

Anal. Calcd for $Cs_3B_{24}H_{23} \cdot 3H_2O$: B, 35.29. Found: B, 35.10. The compound does not melt below 300°.

In a similar experiment, addition of $(C_2H_5)_4NCl$ to the aqueous solution gave the corresponding tetraethylammonium salt of $B_{24}H_{23}^{3-}$.

Anal. Calcd for $[(C_2H_5)_4N]_3B_{24}H_{23}$: C, 42.82; H, 12.42; N, 6.24; B, 38.52. Found: C, 42.93; H, 12.72; N, 6.18; B, 38.66.

Stationary-electrode voltammetry of I in acetonitrile showed only the +1.85-v shoulder found in the voltammetry of $B_{12}H_{12}^{2-}$. The conductivity of I in H_2O gave $\Lambda_m = 462\ \mathrm{ohm}^{-1}\ \mathrm{cm}^{-1}$, typical of a 3:1 electrolyte.

A solution of compound I was passed through a strong acid ion-exchange column, and the liberated acid was neutralized with sodium hydroxide (equivalent weight: calcd, 245.1; found, 246 ± 1). The infrared spectrum of I in KBr showed bands at 2500, 1050, and 940 and multiple bands from 750 to 710 cm^{-1}.

The ^{11}B nmr spectrum of aqueous I at 32.0 Mc consisted of an unsymmetrical doublet at +15.4 ppm relative to external $BF_3 \cdot O(C_2H_5)_2$, with $J = 130$ cps. The 100-Mc ^1H spectrum irradiated at 32.0 Mc consisted of two peaks of approximately equal intensities at +3.02 and +3.21 ppm from H_2O. The optimum decoupling frequencies for the two peaks differed by about 30 cps, which implies that the ^{11}B unsymmetrical doublet includes two doublets separated by less than 1 ppm. The low-field ^1H peak was considerably broader under optimum decoupling conditions than was the high-field peak.

The controlled-potential electrolysis of $B_{12}H_{12}^{2-}$ apparently parallels that of $B_{10}H_{10}^{2-}$, a one-electron oxidation followed by a dimerization reaction with the B_{12} cage left intact.

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Steroid Conjugates. III.[1] The Synthesis of a Sulfoglucuronide Derivative of Estriol

Sir:

We wish to describe the first chemical synthesis of estriol 3-sulfate 16-glucuronide (3-sulfooxy-17β-hydroxyestra-1,3,5(10)-trien-16α-yl-β-D-glucopyranosidur-

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