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Boron-11 Decoupling Studies on the Proton Nmr of $C_2B_4H_8$

by

Thomas Onak

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Department of Chemistry
California State College at Los Angeles
Los Angeles, California

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Thomas Onak

Department of Chemistry, California State College at Los Angeles

The proton nmr of $C_2B_4H_8$ has been recorded and consists of a single peak $\tau = 3.68$, a 1:1:1:1 quartet $\tau = 6.56$, another 1:1:1:1 quartet $\tau = 10.13$, and a broad peak $\tau = 12.6$ which have been assigned to the H-C, $H_{terminal}$-$B_{4,5,6}$, $H_{terminal}$-B_1 and H_{bridge} hydrogen nuclei, respectively.[1] The assignment of the high field quartet to H_t-B_1, however, was based more on analogy[2] than on direct evidence. On this assumption it was necessary, from area considerations, to assign the low field quartet to two overlapping resonances, H_t-$B_{4,6}$ and the chemically unique H_t-B_5. Without direct evidence available there still remained the ambiguity of the H_t-B_1 and H_t-B_5 assignments. With this in mind the present study was undertaken.

The structure of $C_2B_4H_8$ has been verified by X-ray studies[3] and it is clear that each of the basal boron atoms (4,6 and 5) of the pentagonal pyrimidal framework has, in addition to a terminal hydrogen, an attached bridge hydrogen(s) whereas the apex boron has an attached terminal hydrogen only. With boron-11 decoupling experiments carried out at 19.2 Mc it is possible to selectively irradiate the high- and low-field proton nmr quartets. When the high field quartet is collapsed the rest of the spectrum remains unchanged. In striking contrast, collapse of the low field quartet is accompanied by a markedly sharpened bridge

hydrogen region. Such observations are consistent only with the original H_t-B_1 (no bridge hydrogens) assignment to the high field quartet.

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References

1. T. Onak, R. P. Drake and G. B. Dunks, Inorg. Chem., 3, 1686 (1964).

2. B. Figgis and R. L. Williams, Spectrochim. Acta, 331 (1959); see also references mentioned on pgs. 312 and 313, T. Onak, Advan. Organomet. Chem., 3, 263 (1966).

3. W. E. Streib, F. P. Boer and W. N. Lipscomb, J. Am. Chem. Soc., 85, 2331 (1963); Inorg. Chem., 3, 1666 (1964).