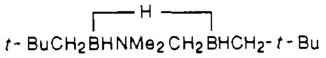
doublet centered at 1.68 ppm and a 1.93 ppm complex multiplet coalescing to the doublet of doublets signal at 1.84/1.88 ppm. The resonances are inherently broadened by the boron quadrupole and spin splittings. Other small, broad multiplets in the region near TMS are presumedly associated with the methylene protons of the neopentyls. Two resonances at −0.06 and −0.72 ppm are present in the trans isomer, and three multiplets, one looking like AB splitting, are present in the cis isomer at −75 °C: 0.3 (0.40, 0.36, 0.28, 0.24 ppm AB pattern), −0.06, and −0.16 ppm, coalescing to a very broad resonance at 0.14 ppm at room temperature. Taken together, these observations suggest an AB methylene pattern for the axial neopentyl methylene of the cis isomer and a less defined multiplet for the equatorial methylene.

The neopentyl methyl resonance shift is slightly temperature dependent, even in the nonexchanging trans isomer, moving about 0.03 ppm upfield on cooling.

The stereochemistry of the isomers allows a better understanding of the high resolution mass spectral data of the separated isomers (Table I). Significantly, the 100% peaks are different, being the 211 peak for the cis isomer and the 70 peak for the trans isomer. The one neopentyl group on the cis isomer that is axial (at the time of fragmentation) would have strong contact with axial groups and should be more easily lost than the equatorial groups on the trans isomer, whence the high 211 (parent less neopentyl) of the cis isomer. A stable structure, probably cyclic, may be inferred for the 223/224 fragment because of its significant intensity. One, with hydrogen bridging over the gap from the lost CH_2NMe_2 moiety, satisfies the valencies believably:



$t\text{-}BuCH_2BHNMe_2CH_2BHCH_2\text{-}t\text{-}Bu$

Conclusion

Characterization of two diastereomers of the dineopentyl-substituted BCN-sequenced heterocycle and study of the temperature dependent proton resonance spectrum of the cis isomer establish firmly the dynamic nature of the ring system, a feature that previously was a matter of conjecture. Additionally, the substituted ring is found to be robust toward atmospheric conditions and toward ring opening at room temperature. The cis isomer rearranges to the thermodynamically favored trans isomer, but only after prolonged heating at 160–170 °C. Possibilities are therefore promising that other 2,5-disubstituted diastereomers may be made and separated, and these possibilities would allow investigation of whether substitution reactions at boron proceed with or without retention. Finally, a synthesis of 2,5-dialkyl-substituted six-membered BCN-sequenced heterocycles is uncovered.

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Registry No. **1**-*cis*, 114492-12-5; **1**-*trans*, 114530-91-5; $H_2BCH_2N(Me)_2CH_2$, 71371-27-2.

Additions and Corrections

1986, Volume 25

Ahmad Moini, Roberta Peascoe, Philip R. Rudolf, and Abraham Clearfield*: Hydrothermal Synthesis of Copper Molybdates.

Page 3783. In Table II the *y* positional parameter for Mo2 should read 0.70979 (6) instead of 0.79079 (6).—Abraham Clearfield