Darstellung und spektroskopische Charakterisierung von Methylnitro-*closo*-hexaboraten sowie Kristallstrukturen von *cis*-$(Ph_4As)_2[B_6H_4(CH_3)(NO_2)]$, *fac*-$(Ph_4As)_2[B_6H_3(CH_3)(NO_2)_2]$ · CH_3CN und *mer*-$(Ph_4P)_2[B_6H_3(CH_3)(NO_2)^c{}_2]$

Synthesis and Spectroscopic Characterization of Methylnitro-*closo*-hexaborates and Crystal Structures of *cis*-$(Ph_4As)_2[B_6H_4(CH_3)(NO_2)]$, *fac*-$(Ph_4As)_2[B_6H_3(CH_3)(NO_2)_2]$ · CH_3CN and *mer*-$(Ph_4P)_2[B_6H_3(CH_3)(NO_2)^c{}_2]$

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Z. Naturforsch. **54 b,** 349–356 (1999); eingegangen am 11. November 1998

Methylnitrotetrahydro-*closo*-hexaborate(2-), Methyldinitrotrihydro-*closo*-hexaborate(2-), Crystal Structure, ^{11}B NMR Spectra, Vibrational Spectra

By electrochemical oxidation of $(n\text{-}Bu_4N)[B_6H_6(CH_3)]$ in the presence of nitrite ions and of the base DBU in dichloromethane solution *cis*- and *trans*-$[B_6H_4(CH_3)(NO_2)]^{2-}$, *fac*-$[B_6H_3(CH_3)(NO_2)_2]^{2-}$ and *mer*-$[B_6H_3(CH_3)(NO_2)^c{}_2]^{2-}$ are formed. X-ray diffraction analyses have been performed on single crystals of *cis*-$(Ph_4As)_2[B_6H_4(CH_3)(NO_2)]$ (**1**) (monoclinic, space group $P2_1/a$, a = 20.063(2), b = 10.858(1), c = 21.384(2) Å, β = 105.818(9)°, Z = 4), *fac*-$(Ph_4As)_2[B_6H_3(CH_3)(NO_2)_2]\cdot CH_3CN$ (**2**) (triclinic, space group P1, a = 10.333(3), b = 10.695(3), c = 22.833(6) Å, α = 93.91(3), β = 96.79(3), γ = 104.56(2)°, Z = 2), and *mer*-$(Ph_4P)_2[B_6H_3(CH_3)(NO_2)^c{}_2]$ (**3**) (triclinic, space group P1, a = 10.100(1), b = 10.402(3), c = 22.923(3) Å, α = 96.328(18), β = 89.928(12), γ = 107.963(16)°, Z = 2). The ^{11}B NMR spectra and the vibrational spectra of the methylnitro compounds are discussed and compared with those of the monomethyl- and mononitro-*closo*-hexaborates.

* Sonderdruckanforderungen an Prof. Dr. W. Preetz.