

1,3,2-Diazaalumina-[3]ferrocenophanes with Alkyn-1-yl Substituents at Aluminum

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The 1,3,2-diazaalumina-[3]ferrocenophane-ethyl(dimethyl)amine adduct **2**, containing an Al–H function, reacts with terminal alkynes $R-C\equiv C-H$ [$R = nBu$ (**a**), tBu (**b**), Ph (**c**), $SiMe_3$ (**d**)] by elimination of H_2 to the amine adducts **4a–d** containing an Al–C \equiv C–R function. Addition of pyridine leads to the corresponding pyridine adducts **5a–d**, of which the molecular structure of **5d** could be determined by single crystal X-ray diffraction. The formation of **4** is accompanied by side reactions such as trimerization of the alkynes to the 1,3,5-trisubstituted benzene derivatives **6a, c**, and some polymerization of the alkynes. The solution-state structures of **4** and **5** were confirmed by multinuclear magnetic resonance spectroscopy (1H , ^{13}C , ^{27}Al , ^{29}Si NMR). Structural features and molecular dynamics were investigated by appropriate $^1H/^1H$ NOE and magnetization transfer experiments, and particular attention was paid to the correct assignment of $^{13}C(Al-C\equiv C-R)$ NMR signals.

Key words: Aluminum, Ferrocenophane, Alkynes, NMR, X-Ray