ORGANIC LETTERS

Toward Interlocked Molecules Beyond Catenanes and Rotaxanes

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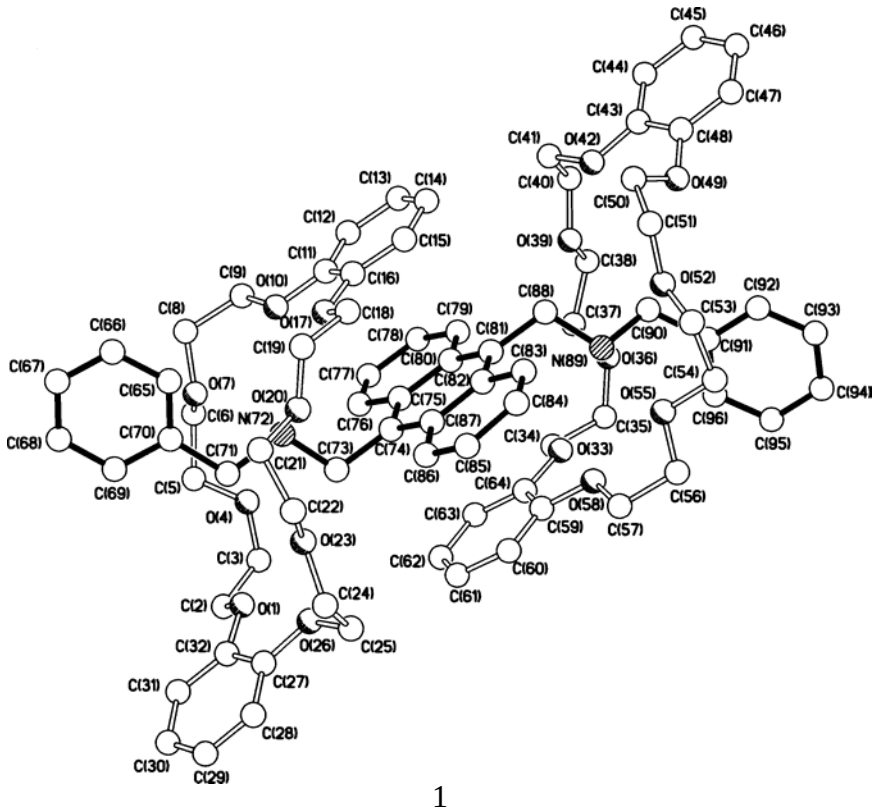
SUPPORTING INFORMATION

(2 PAGES)

EXPERIMENTAL PROCEDURES FOR **1**-H_2· $2PF_6$

CRYSTAL DATA FOR [**DB24C8**· **1**-H_2][$2PF_6$] CAN BE FOUND IN THE ACCOMPANYING FILE CALLED "FS9911.CIF"



Experimental Section

General

All reagents and solvents were used as received unless otherwise stated. Reactions were carried out under an atmosphere of anhydrous argon. Reactions were monitored by TLC on silica plates (Merck, 0.25 mm) and visualized with UV light (254 nm). Melting points given are uncorrected. NMR Spectra were recorded on either a Bruker ARX 400 MHz or ARX 500 spectrometer. Chemical shifts reported are referenced to the residual solvent peak.

Synthesis

1-$H_2 \cdot 2PF_6$:

A solution of 9,10-bis(aminomethyl)anthracene (1.77 g, 0.008 mol) and benzaldehyde (1.59 g, 0.015 mol) was heated under reflux for 20 h in PhMe (500 mL) using a Dean-Stark apparatus. The solvent was evaporated *in vacuo* and the residue (3.04 g, 0.007 mol) dissolved in distilled THF (50 mL) and MeOH (100 mL). $NaBH_4$ (2.78 g, 0.074 mol) was added portionwise to the stirring solution. The reaction mixture was quenched with ice-water and the aqueous layer extracted with CH_2Cl_2. The solvent was evaporated to give a yellow powder. The powder was purified by passing through a short pad of SiO_2 (EtOAc/C_6H_{14} 3:1, 1% Et_3N). The resulting yellow solid was recrystallized (EtOAc/C_6H_{14}) to yield compound **1** (1.04 g, 34%). Protonation of **1** followed by counterion exchange by standard procedures, yielded **1**–$H_2 \cdot 2PF_6$ (1.37 g, 84%); mp > 226°C (dec); ^{1}H NMR (400 MHz, CD_3CN): δ = 4.53 (s, 4H), 5.29 (s, 4H), 7.55 (m, 6H), 7.72 (m, 4H), 7.73 (m, 4H), 8.32 (m, 4H); ^{13}C NMR (100 MHz, CD_3CN): δ = 42.9, 52.3, 124.2, 124.9, 127.7, 129.4, 129.7, 130.2, 130.4, 130.6; $C_{30}H_{30}N_2P_2F_{12}$ (708.5): calcd C 50.86, H 4.27, N 3.95; found C 51.02, H 4.22, N 3.76; FABMS: *m/z* (%): 563.3 (100) $[M - PF_6]^+$, 417.2 (81) $[M - 2PF_6]^+$.