Supplementary Information

Structure of 3-(Diethylborylethynyl)pyridine: Nonplanarly Cyclic Trimer

Shigeharu Wakabayashi,*[a] Mitsumi Kuse,[a] Aimi Kida,[a] Seiji Komeda,[b]

Kazuyuki Tatsumi,[c] and Yoshikazu Sugihara[d]

[a]*Department of Clinical Nutrition, Faculty of Health Science, Suzuka University of Medical Science, Suzuka, Mie 510-0293, Japan*

[b]*Department of Pharmaceutical Sciences, Faculty of Pharmaceutical Sciences, Suzuka University of Medical Science, Suzuka, Mie 513-8670, Japan*

[c]*Department of Chemistry, Graduate School of Science and Research Center for Materials Science, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8602, Japan*

[d]*Department of Chemistry, Faculty of Science, Yamaguchi University, Yamaguchi 753-8512, Japan*

s-waka@suzuka-u.ac.jp

Contents

General methods. Melting points are uncorrected. All ^{1}H and ^{13}C NMR spectra in $CDCl_3$ were recorded at 400 and 100 MHz, respectively, on a Varian 400-MR instrument. The chemical shifts are described as values in ppm relative to a $Si(CH_3)_4$ standard for ^{1}H NMR. ^{11}B NMR spectra in $CDCl_3$ were recorded at 192 MHz using BF_3-etherate as internal standard on Jeol JNM-A600. Infrared spectra were recorded with a Shimadzu IRAffinity-1 Fourier transform infrared spectrophotometer. EI mass spectra were recorded at a voltage of 20 eV on a Jeol JMS T100GCV mass spectrometer. ESIMS spectra were recorded in a mixed solution of CH_3CN and THF on an PerkinElmer Sciex API 300 triple quadrupole LC/MS/MS mass spectrometer. UV/Vis spectra were recorded on a Hitachi U-3000 spectrometer. Vapor pressure osmometry was performed using Knauer A0280 and K-7000 instruments. Analytical thin-layer chromatography was conducted on a plate coated with silicagel plate (0.25 mm thickness), and visualization was effected with short wavelength UV light (254 nm). Most reactions were carried out in septum-capped, oven-dried, and argon-purged flasks. All reagents were used directly as obtained commercially unless otherwise noted.

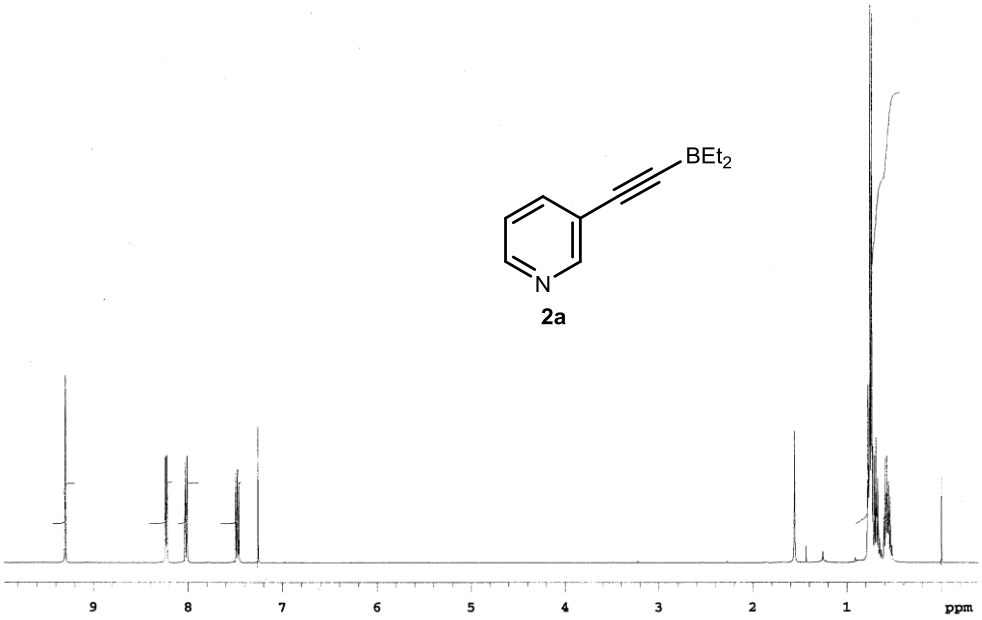

Figure S1. ^{1}H NMR spectrum of **2a** (400 MHz, $CDCl_3$)

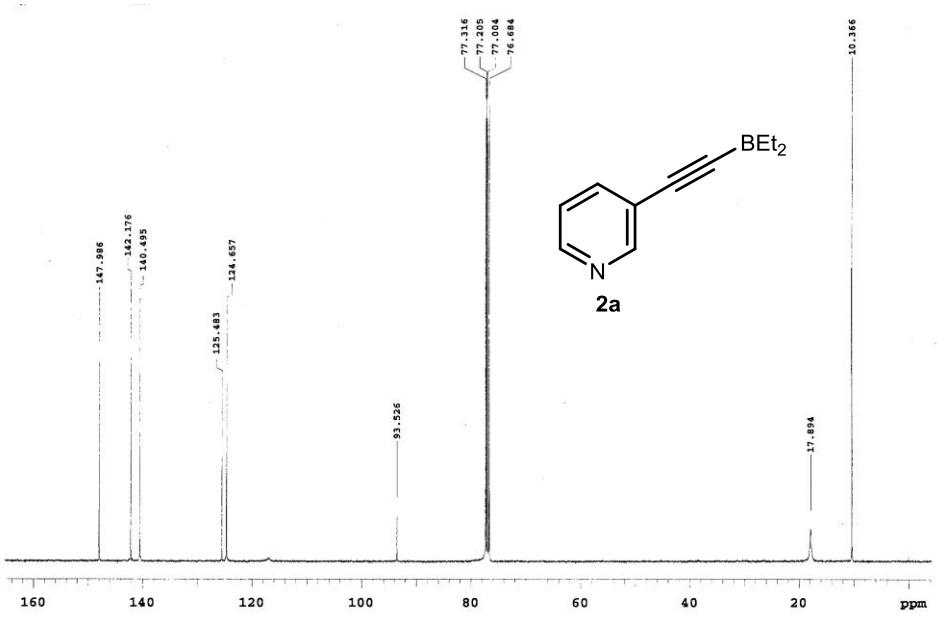

Figure S2. ^{13}C NMR spectrum of **2a** (100 MHz, $CDCl_3$)

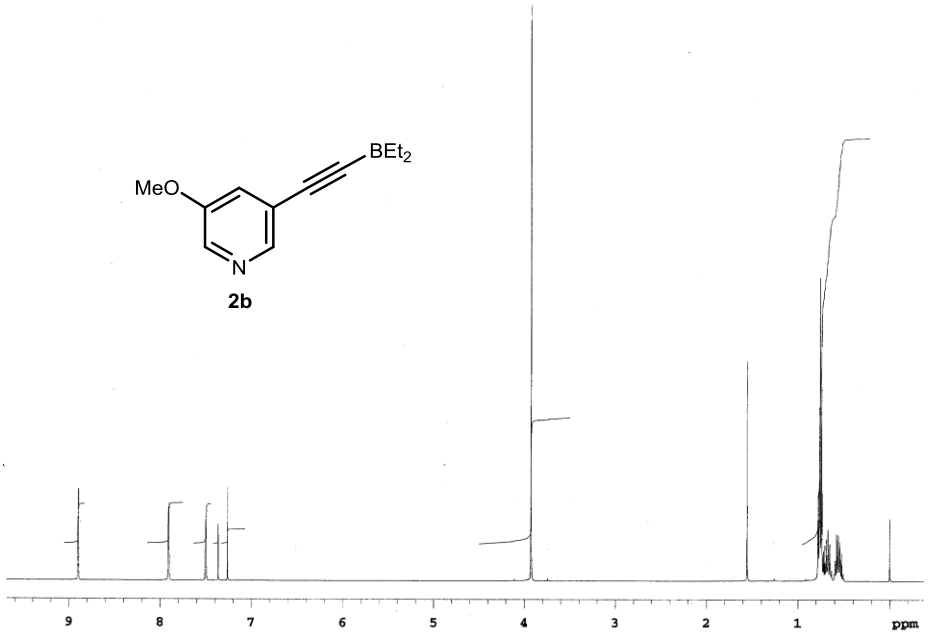

Figure S3. ^{1}H NMR spectrum of **2b** (400 MHz, $CDCl_3$)

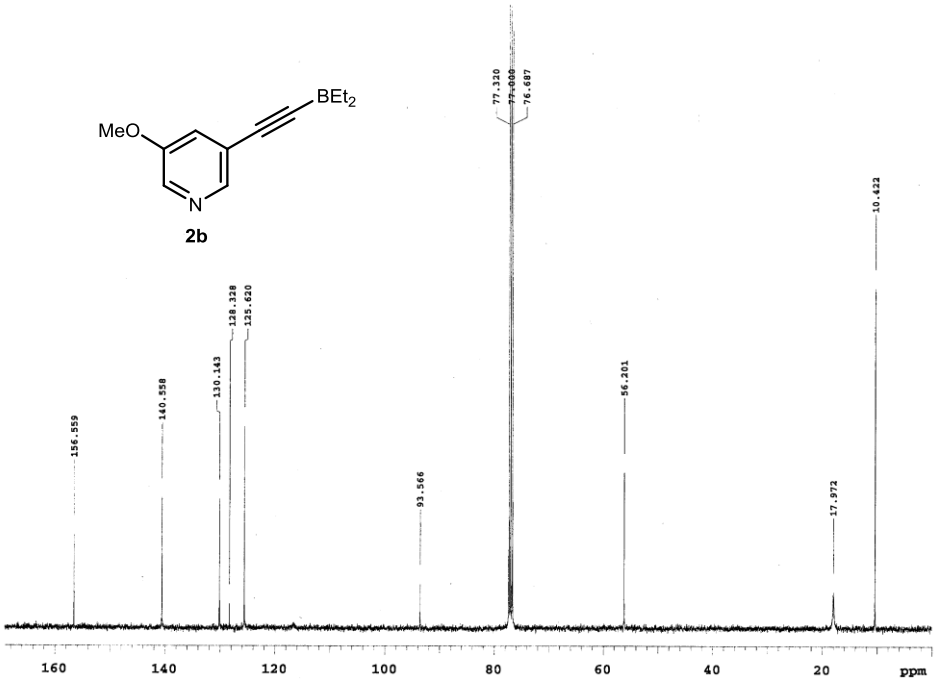

Figure S4. ^{13}C NMR spectrum of **2b** (100 MHz, $CDCl_3$)

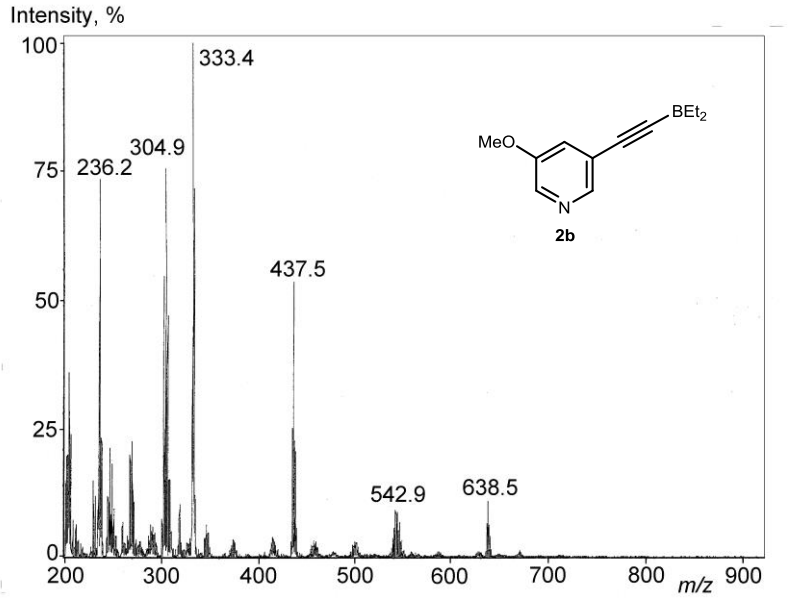

Figure S5. ESI-MS spectrum of **2b** in THF–CH_3CN in the presence of LiCl.

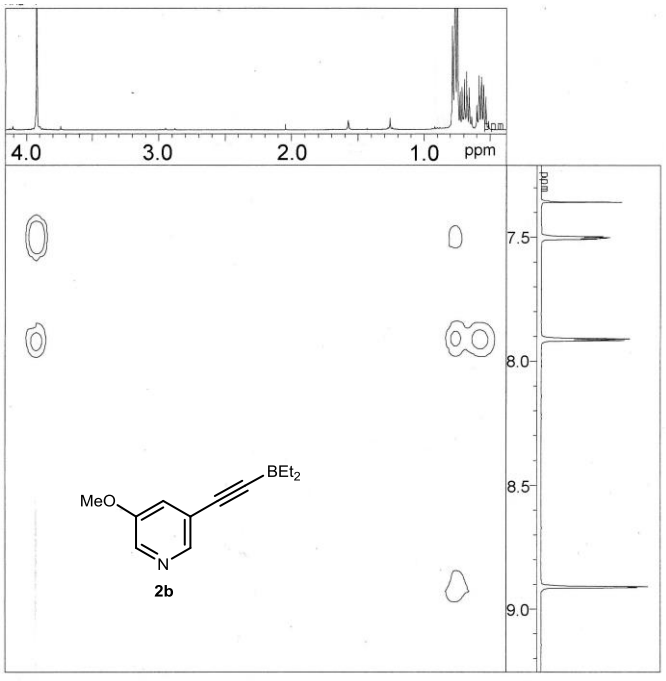

Figure S6. NOESY spectrum of **2b** (400 MHz, $CDCl_3$)

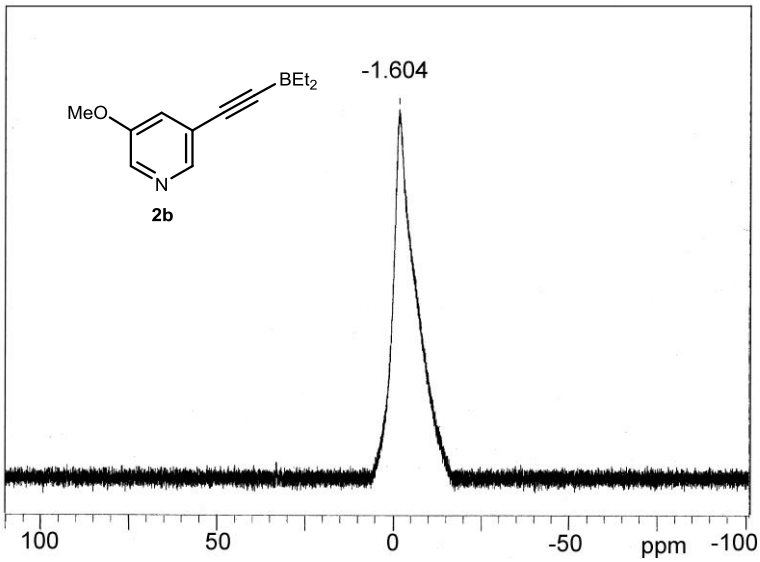

Figure S7. ^{11}B NMR spectrum of **2b** (192 MHz, $CDCl_3$)

Cartesian coordinates of optimized structures of the cyclic trimer of 2b

Method: RB3LYP
Basis set: 6-31G(D)
Number of shells: 276
Number of basis functions: 771
Multiplicity: 1
SCF model:
A restricted hybrid HF-DFT SCF calculation will be
performed using Pulay DIIS + Geometric Direct Minimization
SCF total energy: -1865.1672170 hartrees

Cartesian Coordinates (Angstroms)

Atom		X	Y	Z
1 H	H1	4.0147385	3.9005053	0.0000000
2 C	C1	2.9339724	3.9663493	0.0000000
3 N	N3	0.1521540	4.0672493	0.0000000
4 C	C2	2.1801509	2.7769584	0.0000000
5 C	C6	2.2682778	5.1908306	0.0000000
6 C	C5	0.8702284	5.2019195	0.0000000
7 C	C3	0.7805790	2.8780894	0.0000000
8 H	H5	0.3284929	6.1377411	0.0000000
9 H	H3	0.1442481	2.0039800	0.0000000
10 C	C7	2.8274874	1.5125440	0.0000000
11 C	C8	3.4512059	0.4615117	0.0000000
12 B	B1	4.3988545	-0.8058460	0.0000000
13 N	N1	3.4462642	-2.1653938	0.0000000
14 C	C10	1.9679731	-4.5240692	0.0000000
15 C	C11	4.0698802	-3.3545997	0.0000000
16 C	C12	2.1022091	-2.1150460	0.0000000
17 C	C13	1.3148410	-3.2765453	0.0000000
18 C	C14	3.3612522	-4.5598016	0.0000000
19 H	H8	5.1511933	-3.3533537	0.0000000
20 H	H9	1.6633735	-1.1269125	0.0000000
21 H	H12	1.3705675	-5.4271181	0.0000000

22 C	C15	-0.1038421	-3.2049479	0.0000000
23 C	C16	-1.3259221	-3.2195878	0.0000000
24 B	B2	-2.8973104	-3.4065967	0.0000000
25 N	N2	-3.5984182	-1.9018555	0.0000000
26 C	C18	-4.9019454	0.5577200	0.0000000
27 C	C19	-4.9401086	-1.8473198	0.0000000
28 C	C20	-2.8827881	-0.7630435	0.0000000
29 C	C21	-3.4949920	0.4995869	0.0000000
30 C	C22	-5.6295301	-0.6310291	0.0000000
31 H	H14	-5.4796861	-2.7843874	0.0000000
32 H	H15	-1.8076216	-0.8770674	0.0000000
33 H	H18	-5.3853059	1.5266128	0.0000000
34 C	C23	-2.7236452	1.6924039	0.0000000
35 C	C24	-2.1252837	2.7580762	0.0000000
36 B	B3	-1.5015441	4.2124428	0.0000000
37 C	C4	5.3107353	-0.8401264	-1.3606592
38 H	H4	6.0075408	-1.6936202	-1.3235207
39 H	H7	5.9567919	0.0498188	-1.3325627
40 C	C9	4.5561960	-0.8631767	-2.6984349
41 H	H16	3.9358741	-1.7638447	-2.8015174
42 H	H21	5.2369455	-0.8431859	-3.5595291
43 H	H22	3.8852470	0.0000000	-2.7870929
44 C	C17	5.3107353	-0.8401264	1.3606592
45 H	H2	6.0075408	-1.6936202	1.3235207
46 H	H24	5.9567919	0.0498188	1.3325627
47 C	C25	4.5561960	-0.8631767	2.6984349
48 H	H23	3.9358741	-1.7638447	2.8015174
49 H	H25	3.8852470	0.0000000	2.7870929
50 H	H26	5.2369455	-0.8431859	3.5595291
51 C	C26	-3.3829384	-4.1791685	1.3606592
52 H	H10	-4.4704885	-4.3558728	1.3235207
53 H	H28	-2.9352516	-5.1836425	1.3325627
54 C	C27	-3.0256309	-3.5141931	2.6984349
55 H	H27	-1.9426235	-3.3647226	2.7870929
56 H	H29	-3.3486932	-4.1137349	3.5595291
57 H	H30	-3.4954714	-2.5266446	2.8015174

58 C	C28	-3.3829384	-4.1791685	-1.3606592
59 H	H13	-4.4704885	-4.3558728	-1.3235207
60 H	H31	-2.9352516	-5.1836425	-1.3325627
61 C	C29	-3.0256309	-3.5141931	-2.6984349
62 H	H32	-3.4954714	-2.5266446	-2.8015174
63 H	H33	-3.3486932	-4.1137349	-3.5595291
64 H	H34	-1.9426235	-3.3647226	-2.7870929
65 C	C30	-1.9277969	5.0192948	1.3606592
66 H	H19	-1.5370523	6.0494930	1.3235207
67 H	H36	-3.0215403	5.1338238	1.3325627
68 C	C31	-1.5305651	4.3773698	2.6984349
69 H	H35	-0.4404027	4.2904893	2.8015174
70 H	H37	-1.9426235	3.3647226	2.7870929
71 H	H38	-1.8882524	4.9569208	3.5595291
72 C	C32	-1.9277969	5.0192948	-1.3606592
73 H	H20	-3.0215403	5.1338238	-1.3325627
74 H	H40	-1.5370523	6.0494930	-1.3235207
75 C	C33	-1.5305651	4.3773698	-2.6984349
76 H	H39	-1.9426235	3.3647226	-2.7870929
77 H	H41	-0.4404027	4.2904893	-2.8015174
78 H	H42	-1.8882524	4.9569208	-3.5595291
79 O	O1	4.1236159	-5.6801529	0.0000000
80 C	C34	3.4633594	-6.9420247	0.0000000
81 H	H11	2.8430092	-7.0674905	0.8961157
82 H	H43	4.2575150	-7.6894853	0.0000000
83 H	H44	2.8430092	-7.0674905	-0.8961157
84 O	O2	-6.9809646	-0.7310796	0.0000000
85 C	C35	-7.7436494	0.4716551	0.0000000
86 H	H17	-7.5421309	1.0716271	0.8961157
87 H	H45	-8.7880471	0.1576266	0.0000000
88 H	H46	-7.5421309	1.0716271	-0.8961157
89 O	O3	2.8573488	6.4112326	0.0000000
90 C	C36	4.2802900	6.4703696	0.0000000
91 H	H6	4.6991218	5.9958634	0.8961157
92 H	H47	4.5305321	7.5318588	0.0000000
93 H	H48	4.6991218	5.9958634	-0.8961157