Supporting Information

for

Synthesis of 5-(2-methoxy-1-naphthyl)- and 5-[2-(methoxymethyl)-1-naphthyl]-11*H*-benzo[*b*]fluorene as 2,2'-disubstituted 1,1'-binaphthyls via benzannulated enyne–allenes

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Dedicated to Professor Anthony Winston on the occasion of his 85th birthday.

Experimental procedures, spectroscopic data, and ^{1}H and/or ^{13}C NMR spectra of **5a**,**b**, **6a**,**b**, **8a**,**b**, **9a**,**b**, **13a**,**b**, **17**, **18b**, **19a**–**c**, **20a**–**c**, **21**–**24**, and the (1*S*)-camphanates of **24**.

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Trimethyl[[2-[(2-methoxyphenyl)ethynyl]phenyl]ethynyl]silane (5a). To a mixture of 0.318 g of 1-iodo-2-[2-(trimethylsilyl)ethynyl]benzene (1.059 mmol), $Pd(PPh_3)_2Cl_2$ (0.027 g, 0.039 mmol), and copper(I) iodide (0.008 g, 0.04 mmol) in 15 mL of triethylamine, was added via cannula a solution of 0.103 g of **4a** (0.780 mmol) in 5 mL of triethylamine. After stirring at 70 °C for 5 h, 20 mL of a saturated ammonium chloride solution and 20 mL of diethyl ether were added. The organic layer was separated and the aqueous layer back extracted with diethyl ether. The combined organic layers were washed successively with brine and water, dried over sodium sulfate, and concentrated. Purification of the residue by flash column chromatography (silica gel/30% methylene chloride in hexanes) afforded 0.206 g of **5a** (0.677 mmol, 87%) as a colorless liquid: ^{1}H ($CDCl_3$, 270 MHz) δ 7.59−7.47 (3 H, m), 7.35−7.20 (3 H, m), 6.94 (1 H, t, J = 7.7 Hz), 6.91 (1 H, d, J = 7.7 Hz), 3.91 (3 H, s), 0.26 (9 H, s) ; ^{13}C ($CDCl_3$, 67.9 MHz) δ 159.9, 133.9, 132.2, 131.9, 129.9, 128.1, 127.6, 126.4, 125.4, 120.4, 112.5, 110.7, 103.6, 98.4, 92.0, 89.9, 55.8, −0.1.

Benzannulated Enediyne 6a. To 0.201 g (0.660 mmol) of **5a** in 10 mL of diethyl ether were added 4 mL of a 10% sodium hydroxide solution and 10 mL of methanol. After stirring at room temperature for 30 min, 20 mL of water and 20 mL of diethyl ether were added. The organic layer was separated and the aqueous layer back extracted with diethyl ether. The combined organic layers were washed successively with brine and water, dried over sodium sulfate, and concentrated. Purification of the residue by flash column chromatography (silica gel/30% methylene chloride in hexanes) afforded 0.137 g of **6a** (0.591 mmol, 90%) as a yellow liquid: ^{1}H ($CDCl_3$, 270 MHz) δ 7.60−7.51 (3 H, m), 7.36−7.23 (3 H, m), 6.95 (1 H, t, J = 7.6 Hz), 6.90 (1 H, d, J = 8.2 Hz), 3.91 (3 H, s), 3.37

(1 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 160.0, 133.8, 132.5, 131.8, 130.0, 128.4, 127.7, 126.6, 124.4, 120.4, 112.3, 110.7, 91.8, 90.0, 82.3, 81.0, 55.8.

α-[[2-[(2-Methoxyphenyl)ethynyl]phenyl]ethynyl]-α-(1,1-dimethylethyl)benzenemethanol (8a). To 0.065 g (0.28 mmol) of **6a** in 5 mL of anhydrous diethyl ether under a nitrogen atmosphere at 0 °C, was added 0.18 mL of a 1.6 M solution of *n*-butyllithium (0.28 mmol) in hexanes. After stirring for 30 min, a solution of 0.064 g of **7** (0.39 mmol) in 10 mL of THF was introduced via cannula, and the reaction mixture allowed to warm to room temperature. After an additional 3 h, 15 mL of water was introduced and the reaction mixture extracted with diethyl ether. The combined organic extracts were washed successively with brine and water, dried over sodium sulfate, and concentrated. The residue was purified by flash column chromatography (silica gel/15% diethyl ether in hexanes) to provide 0.109 g (0.276 mmol, 99%) of **8a** as a light yellow liquid: ^{1}H ($CDCl_3$, 270 MHz) δ 7.78−7.71 (2 H, m), 7.61−7.57 (1 H, m), 7.53−7.49 (1 H, m), 7.41 (1 H, dd, J = 7.9, 1.7 Hz), 7.35−7.20 (6 H, m), 6.94−6.87 (2 H, m), 3.84 (3 H, s), 2.67 (1 H, s), 1.07 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 159.9, 142.1, 133.7, 132.2, 131.9, 129.9, 127.9, 127.8, 127.2, 126.9, 126.1, 125.1, 120.5, 112.4, 110.9, 96.2, 92.1, 89.5, 84.6, 79.4, 55.9, 39.7, 25.5.

Benzannulated Enediyne 9a. To a mixture of **8a** (0.109 g, 0.276 mmol) and triethylsilane (0.048 g, 0.41 mmol) in 10 mL of methylene chloride, was added trifluoroacetic acid (0.126 g, 1.11 mmol). After stirring at room temperature for 1 h, sodium carbonate (0.11 g, 4.6 mmol) was added, followed by 10 mL of water and 10 mL of diethyl ether. The organic layer was separated, dried over sodium sulfate and concentrated. Purification of the residue by flash column chromatography (silica gel/25%

methylene chloride in hexanes) provided 0.097 g (0.256 mmol, 93%) of **9a** as a light yellow liquid: ^{1}H ($CDCl_3$, 270 MHz) δ 7.61−7.55 (1 H, m), 7.48−7.16 (10 H, m), 6.92−6.85 (2 H, m), 3.87 (3 H, s), 3.69 (1 H, s), 1.04 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 160.0, 139.2, 133.7, 132.2, 132.0, 129.8, 129.7, 127.7, 127.5, 127.2, 126.5, 126.3, 125.9, 120.3, 112.5, 110.5, 95.5, 92.4, 89.1, 82.6, 55.7, 50.6, 35.5, 27.7.

5-(2-Methoxyphenyl)-10-(1,1-dimethylethyl)-11*H*-benzo[*b*]fluorene (13a) To 0.097 g of **9a** (0.256 mmol) in 10 mL of anhydrous toluene under a nitrogen atmosphere, was added 0.33 mL of a 1.0 M solution of potassium *t*-butoxide (0.33 mmol) in 2-methyl-2-propanol. The reaction mixture was then heated under reflux for 5 h. After the reaction mixture was allowed to cool to room temperature, 10 mL of water and 20 mL of methylene chloride were introduced. The organic layer was separated, dried over sodium sulfate and concentrated. The residue was purified by flash column chromatography (silica gel/20 % methylene chloride in hexanes) to provide 0.073 g of **13a** (0.192 mmol, 75%) as a light yellow liquid and 0.002g of **14a** (0.005 mmol, 2%). **13a**: IR 1330, 811, 739 cm^{-1}; ^{1}H (C_6D_6, 600 MHz) δ 8.62 (1 H, d, J = 9.0 Hz), 7.89 (1 H, d, J = 8.4 Hz), 7.35−7.27 (4 H, m), 7.22 (1 H, t, J = 7.2 Hz), 7.11 (1 H, t, J = 7.2 Hz), 7.04−7.02 (2 H, m), 6.86 (1 H, d, J = 7.8 Hz), 6.78 (1 H, d, J = 8.4 Hz), 4.21 (1 H, d, J = 21.0 Hz), 4.13 (1 H, d, J = 21.0 Hz), 3.06 (3 H, s), 1.71 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 157.7, 144.2, 140.7, 140.5, 138.4, 137.7, 134.4, 131.8, 131.5, 129.6, 129.3, 128.3, 128.0, 127.2, 126.7, 126.3, 124.0, 123.8, 123.2, 123.1, 121.5, 111.6, 55.8, 40.2, 38.8, 34.4; MS *m/z* 378 (M^+), 363, 349, 321; HRMS calcd for $C_{28}H_{26}O$ 378.1984, found 378.1971. The ^{1}H NMR signals attributable to **14a** (ca. 2%) were observed at δ ($CDCl_3$) 6.25 (1 H, s), 3.86 (3 H, s), and 1.23 (9 H, s).

Trimethyl[[2-[[2-(methoxymethyl)phenyl]ethynyl]phenyl]ethynyl]silane (5b). The same procedure as described for **5a** was repeated except that 0.445 g of 1-iodo-2-[2-(trimethylsilyl)ethynyl]benzene (1.482 mmol), $Pd(PPh_3)_2Cl_2$ (0.040 g, 0.056 mmol), and copper(I) iodide (0.015 g, 0.080 mmol) in 10 mL of triethylamine were treated with a solution of 0.167 g of **4b** (1.140 mmol) in 5 mL of triethylamine to afford 0.287 g of **5b** (0.900 mmol, 79%) as a colorless liquid: ^{1}H ($CDCl_3$, 600 MHz) δ 7.31 (1 H, d, J = 7.2 Hz), 7.28−7.23 (2 H, m), 7.11 (1 H, t, J = 7.2 Hz), 7.06−7.00 (4 H, m), 4.52 (2 H, s), 3.22 (3 H, s), 0.01 (9 H, s); ^{13}C ($CDCl_3$, 150 MHz) δ 140.0, 132.6, 132.3, 131.9, 128.7, 128.2, 127.9, 127.4, 127.2, 125.9, 125.4, 121.8, 103.6, 98.6, 92.5, 90.9, 72.6, 58.5, −0.04.

Benzannulated Enediyne 6b. The same procedure as described for **6a** was repeated except that a solution of 0.255 g (0.800 mmol) **5b** in 15 mL of diethyl ether was treated with 6 mL of a 10% sodium hydroxide solution and 15 mL of methanol to afford 0.193 g of **6b** (0.785 mmol, 98%) as a yellow liquid: ^{1}H ($CDCl_3$, 600 MHz) δ 7.56 (1 H, dd, J = 7.8, 1.2 Hz), 7.55−7.53 (2 H, m), 7.50 (1 H, d, J = 8.4 Hz), 7.36 (1 H, td, J = 7.7, 1.2 Hz), 7.33 (1 H, td, J = 7.8, 1.2 Hz), 7.30−7.26 (2 H, m), 4.80 (2 H, s), 3.47 (3 H, s), 3.37 (1 H, s); ^{13}C ($CDCl_3$, 150 MHz) δ 140.3, 132.7, 132.2, 131.9, 128.8, 128.6, 128.0, 127.21, 127.18, 126.3, 124.3, 121.5, 92.2, 91.0, 82.5, 81.1, 72.6, 58.5.

α-[[2-[[2-(Methoxymethyl)phenyl]ethynyl]phenyl]ethynyl]-α-(1,1-dimethylethyl)benzenemethanol (8b). The same procedure as described for **8a** was repeated except that 0.165 g (1.015 mmol) of **7** was treated with the lithium acetylide derived from 0.178 g (0.725 mmol) of **6b** and 0.45 mL of a 1.6 M solution of *n*-butyllithium (0.72 mmol) in hexanes to afford 0.284 g (0.696 mmol, 96%) of **8b** as a light yellow liquid: IR 3410, 2961, 1490, 758 cm^{-1}; ^{1}H ($CDCl_3$, 270 MHz) δ 7.74−7.69 (2 H,

m), 7.57−7.53 (2 H, m), 7.46−7.21 (7 H, m), 4.67 (1 H, d, *J* =12.6 Hz), 4.40 (1 H, d, *J* =12.4 Hz), 3.31 (3 H, s), 1.68 (1 H, s, broad), 1.07 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 142.4, 139.4, 132.4, 132.2, 128.5, 128.2, 128.07, 128.02, 127.8, 127.5, 127.2, 127.0, 125.6, 125.3, 122.3, 96.8, 92.6, 90.8, 84.4, 79.3, 72.5, 57.7, 39.7, 25.6.

Benzannulated Enediyne 9b. The same procedure as described for **9a** was repeated except that 0.163 g of **8b** (0.400 mmol) and 0.070 g of triethylsilane (0.601 mmol) was treated with 0.182 g of trifluoroacetic acid (1.602 mmol) to afford 0.141 g (0.360 mmol, 90%) of **9b** as a light yellow liquid: ^{1}H ($CDCl_3$, 600 MHz) δ 7.54−7.52 (1 H, m), 7.50−7.48 (1 H, m), 7.47 (1 H, d, *J* = 7.2 Hz), 7.41−7.38 (3 H, m), 7.34 (1 H, t, *J* =7.8 Hz), 7.29−7.27 (2 H, m), 7.21 (1 H, t, *J* =7.8 Hz), 7.19−7.17 (3 H, m), 4.70 (1 H, d, *J* =12.6 Hz), 4.63 (1 H, d, *J* =13.2 Hz), 3.69 (1 H, s), 3.41 (3 H, s), 1.03 (9 H, s); ^{13}C ($CDCl_3$, 150 MHz) δ 140.2, 139.1, 132.3, 132.11, 132.05, 129.7, 128.5, 128.0, 127.6, 127.4, 127.2, 127.1, 126.7, 126.3, 125.5, 121.7, 95.7, 93.0, 90.3, 82.5, 72.5, 58.5, 50.6, 35.5, 27.7.

5-[2-(Methoxymethyl)phenyl]-10-(1,1-dimethylethyl)-11*H*-benzo[*b*]fluorene (13b). The same procedure as described for **13a** was repeated except that 0.119 g of **9b** (0.303 mmol) was treated with 0.39 mL of a 1.0 M solution of potassium *t*-butoxide (0.39 mmol) in 2-methyl-2-propanol to afford 0.075 g of a mixture of **13b** and **14b** in a 5:1 ratio as a light yellow liquid. The mixture was then dissolved in 8 mL of THF and treated with 1 mL of a 1.0 M BH_3·THF solution under a nitrogen atmosphere. After stirring at room temperature for 5 h, 10 mL of water and 10 mL of diethyl ether were added. The organic layer was separated and the aqueous layer back extracted with diethyl ether. The combined organic layers were washed successively with brine and water, dried over

sodium sulfate, and concentrated. Purification of the residue by flash column chromatography (silica gel/30% methylene chloride in hexanes) afforded 0.057 g of **13b** (0.145 mmol, 48%) as a colorless liquid: IR 2923, 1088, 764, 732 cm^{-1}; ^{1}H ($CDCl_3$, 600 MHz) δ 8.62 (1 H, d, J = 9.6 Hz), 7.77 (1 H, d, J = 7.8 Hz), 7.60 (1 H, t, J = 7.8 Hz), 7.49−7.41 (4 H, m), 7.29 (1 H, t, J = 7.2 Hz), 7.22 (1 H, d, J = 7.8 Hz), 7.19 (1 H, t, J = 7.2 Hz), 6.96 (1 H, t, J = 7.8 Hz), 6.22 (1 H, d, J = 7.8 Hz), 4.522 (1 H, d, J = 21 Hz), 4.504 (1 H, d, J = 21 Hz), 4.07 (1 H, d, J = 13.8 Hz), 4.04 (1 H, d, J = 13.8 Hz), 3.10 (3 H, s), 1.93 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 144.1, 141.2, 140.1, 138.3, 137.9, 137.5, 137.4, 134.1, 131.4, 130.7, 130.3, 128.2, 128.0, 127.0, 126.5, 124.3, 123.9, 123.4, 123.1, 71.7, 58.3, 40.2, 38.9, 34.4; MS *m/z* 392 (M^+), 377, 343; HRMS calcd for $C_{29}H_{28}O$ 392.2140, found 392.2135. The ^{1}H NMR signals attributable to **14b** (ca. 10%) before treatment with BH_3·THF were observed at δ ($CDCl_3$) 6.36 (1 H, s), 4.31 (1 H, d, J = 13.2 Hz), 3.83 (1 H, d, J = 13.2 Hz), 2.9 (3 H, s), and 1.21 (9 H, s).

Benzannulated Enediyne 19a. To a mixture of 0.193 g of **17** (0.516 mmol), $Pd(PPh_3)_2Cl_2$ (0.018 g, 0.026 mmol), and copper(I) iodide (0.010 g, 0.053 mmol) in 10 mL of triethylamine, was added via cannula a solution of 0.102 g of **18a** (0.670 mmol) in 3 mL of triethylamine. After stirring at 60 °C for 12 h, 15 mL of a saturated ammonium chloride solution and 15 mL of diethyl ether were added. The organic layer was separated and the aqueous layer back extracted with diethyl ether. The combined organic layers were washed successively with brine and water, dried over sodium sulfate, and concentrated. Purification of the residue by flash column chromatography (silica gel/5% methylene chloride in hexanes) afforded 0.189 g of **19a** (0.475 mmol, 92%) as a light yellow liquid: IR 2227, 1481, 758 cm^{-1}; ^{1}H ($CDCl_3$, 270 MHz) δ 8.53−8.46 (1 H, m),

7.90−7.82 (2 H, m), 7.68−7.62 (2 H, m), 7.56−7.48 (3 H, m), 7.44−7.38 (3 H, m), 7.34−7.30 (2 H, m), 7.16−7.12 (3 H, m) 3.71 (1 H, s), 1.01 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 138.9, 133.2, 133.1, 132.3, 132.1, 130.4, 129.7, 128.7, 128.1, 128.0, 127.5, 127.4, 126.7, 126.6, 126.4, 126.3, 125.6, 125.2, 120.9, 95.8, 93.4, 90.9, 82.6, 50.6, 35.5, 27.7.

10-(1,1-Dimethylethyl)-5-(1-naphthyl)-11*H*-benzo[*b*]fluorene (20a). To 0.189 g of **19a** (0.475 mmol) in 10 mL of anhydrous toluene under a nitrogen atmosphere, was added 0.50 mL of a 1.0 M solution of potassium *t*-butoxide (0.50 mmol) in 2-methyl-2-propanol. The reaction mixture was then heated under reflux for 6 h. After the reaction mixture was allowed to cool to room temperature, 10 mL of water and 40 mL of methylene chloride were introduced, the organic layer separated, dried over sodium sulfate, and concentrated. The residue was purified by flash column chromatography (silica gel/5 % methylene chloride in hexanes) to provide 0.131 g of **20a** (0.328 mmol, 69%) as a bright yellow liquid: IR 1191, 1015 cm^{-1}; ^{1}H (C_6D_6, 600 MHz) δ 8.68 (1 H, d, J = 9.0 Hz), 7.84 (1 H, d, J =7.8 Hz), 7.76 (1 H, d, J = 8.4 Hz), 7.60 (1 H, dd, J = 8.4, 1.2 Hz), 7.54 (1 H, d, J = 8.4 Hz), 7.45 (1 H, dd, J = 6.6, 1.2 Hz), 7.41 (1 H, dd, J = 8.1, 6.9 Hz), 7.27 (1 H, ddd, J = 9.0, 6.6, 1.2 Hz), 7.23 (1 H, d, J = 7.2 Hz), 7.19 (1 H, ddd, J = 8.4, 6.6, 1.2 Hz), 7.01 (1 H, ddd, J = 8.4, 6.6, 1.2 Hz), 6.96 (1 H, td, J = 7.2, 1.2 Hz), 6.90 (1 H, ddd, J = 8.4, 6.6, 1.2 Hz), 6.65 (1 H, t, J = 7.2 Hz), 6.32 (1 H, d, J = 7.8 Hz), 4.27 (1 H, d, J = 21 Hz), 4.25 (1 H, d, J = 21 Hz), 1.81 (9 H, s); ^{1}H ($CDCl_3$, 270 MHz) δ 8.66 (1 H, d, J = 8.7 Hz), 8.07 (1 H, d, J = 8.4 Hz), 8.00 (1 H, d, J = 8.4 Hz), 7.68 (1 H, t, J = 7.7 Hz), 7.52−7.15 (10 H, m), 7.11 (1 H, td, J = 7.4, 1.0 Hz), 6.75 (1 H, t, J = 7.4 Hz), 5.89 (1 H, d, J = 8.2 Hz), 4.56 (2 H, s), 1.96 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 144.2, 141.2, 139.9, 139.1, 137.6, 137.4, 134.9, 133.8, 132.8, 131.4, 130.8, 128.16, 128.03,

127.97, 127.88, 127.7, 126.7, 126.3, 126.2, 124.2, 123.8, 123.5, 123.4, 40.2, 38.9, 34.4; MS *m/z* 398 (M^+), 383, 341; HRMS calcd for $C_{31}H_{26}$ 398.2035, found 398.2028.

1-Ethynyl-2-methoxynaphthalene (18b). To a solution of 1.05 g (5.64 mmol) of 2-methoxy-1-naphthaldehyde and 2.80 g of potassium carbonate in 20 mL of anhydrous methanol, was added 1.37 g (7.15 mmol) of dimethyl (1-diazo-2-oxopropyl)phosphonate, and the reaction mixture was stirred at room temperature for 6 days. The reaction mixture was then diluted with diethyl ether, washed with a 5% aqueous sodium bicarbonate solution, dried over sodium sulfate and concentrated. Purification of the residue by flash column chromatography (silica gel/25% methylene chloride in hexanes) afforded 0.545 g of **18b** (3.00 mmol, 53%) as a white solid: IR 3260, 1274, 1083, 809 cm^{-1}; ^1H ($CDCl_3$, 270 MHz) δ 8.27 (1 H, d, J = 8.4 Hz), 7.86 (1 H, d, J = 8.9 Hz), 7.79 (1 H, d, J = 8.2 Hz), 7.56 (1 H, ddd, J = 8.2, 6.9, 1.2 Hz), 7.39 (1 H, ddd, J = 8.2, 6.9, 1.2 Hz), 7.27 (1 H, d, J = 9.2 Hz), 4.05 (3 H, s), 3.76 (1 H, s); ^{13}C ($CDCl_3$, 150 MHz) δ 159.8, 134.8, 130.7, 128.4, 128.1, 127.6, 125.1, 124.3, 112.5, 105.0, 86.5, 78.2, 56.6; HRMS calcd for $C_{13}H_{11}O$ (MH^+) 183.0810, found 183.0804.

Benzannulated Enediyne 21. The same procedure as described for **5a** was repeated except that 0.200 g of **17** (0.535 mmol), $Pd(PPh_3)_2Cl_2$ (0.016 g, 0.023 mmol), and copper(I) iodide (0.015 g, 0.080 mmol) in 10 mL of triethylamine was treated with a solution of 0.263 g of (trimethylsilyl)acetylene (2.675 mmol) in 5 mL of triethylamine to afford 0.140 g of **21** (0.407 mmol, 76%) as a colorless liquid: ^1H ($CDCl_3$, 270 MHz) δ 7.50−7.39 (4 H, m), 7.35−7.20 (5 H, m), 3.69 (1 H, s), 1.09 (9 H, s), 0.24 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 139.1, 132.8, 132.2, 129.8, 128.0, 127.6, 127.2, 126.6, 126.4, 125.3, 104.0, 98.0, 95.5, 82.3, 50.6, 35.5, 27.9, −0.1.

Benzannulated Enediyne 22. The same procedure as described for **6a** was repeated except that a solution of 0.140 g (0.407 mmol) of **21** in 20 mL of diethyl ether was treated with 8 mL of a 10% sodium hydroxide solution and 20 mL of methanol to afford 0.110 g of **22** (0.403 mmol, 99%) as a light yellow liquid: ^{1}H ($CDCl_3$, 270 MHz) δ 7.55−7.44 (4 H, m), 7.38−7.22 (5 H, m), 3.72 (1 H, s), 3.31 (1 H, s), 1.11 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 139.1, 132.5, 132.0, 129.8, 128.4, 127.5, 127.3, 127.1, 126.7, 124.4, 95.7, 82.8, 82.0, 80.7, 50.5, 35.5, 27.8.

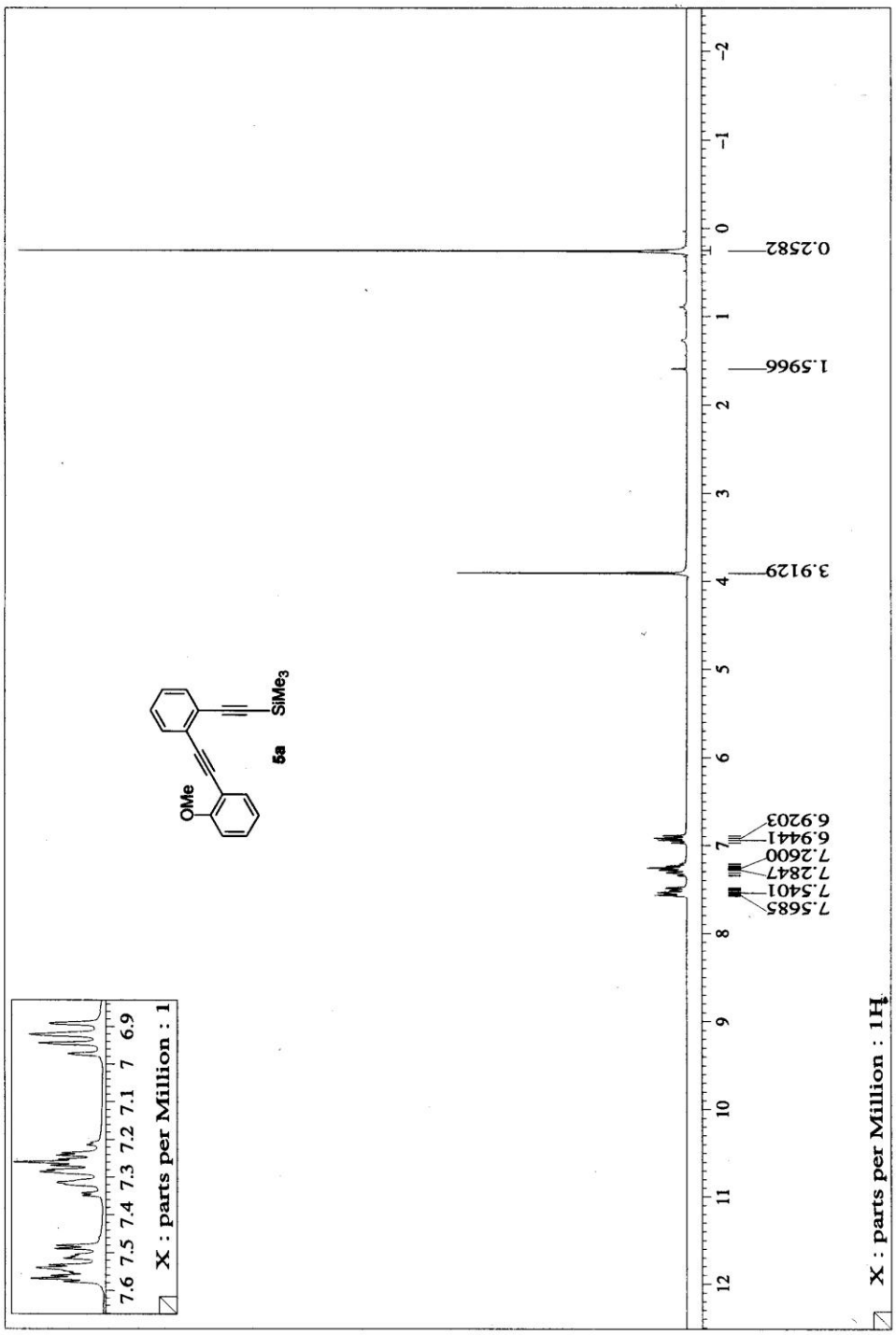
1-Iodo-2-(methoxymethyl)naphthalene (23). A solution of *n*-butyllithium in hexanes (1.6 M, 1.8 mL, 3.06 mmol) was added dropwise to a solution of 0.500 g of 1-bromo-2-(methoxymethyl)naphthalene (1.99 mmol) in 10 mL of THF at −78 °C. After stirring at −78 °C for 1 h, a solution of 1.02 g of iodine (4.02 mmol) in 10 mL of THF was added dropwise via cannula. The reaction mixture then was allowed to warm to room temperature before the addition of 20 mL of a 5% sodium thiosulfate solution. The organic layer was separated and the aqueous layer back extracted with diethyl ether. The combined organic layers were washed successively with brine and water, dried over sodium sulfate, and concentrated. Purification of the residue by flash column chromatography (silica gel/30% methylene chloride in hexanes) afforded 0.516 g of **23** (1.73 mmol, 87%) as a pale yellow liquid: IR 2922, 1500, 1113, 814 cm^{-1}; ^{1}H ($CDCl_3$, 270 MHz) δ 8.26 (1 H, d, J = 7.7 Hz), 7.83 (1 H, d, J = 8.4 Hz), 7.78 (1 H, dd, J = 7.9, 1.5 Hz), 7.61−7.48 (3 H, m), 4.74 (2 H, s), 3.53 (3 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 139.8, 134.6, 133.6, 132.2, 128.7, 128.3, 127.7, 126.4, 125.8, 103.1, 79.8, 58.6.

1,1′-Binaphthyl 24. Boron tribromide (0.5 mL) was added dropwise to a solution of **20b** (0.234 g, 0.546 mmol) in 10 mL of methylene chloride of at 0 °C. The reaction mixture

was stirred at 0 °C for 2 h before 10 mL of water and 20 mL of methylene chloride were introduced. The organic layer was separated, dried over sodium sulfate and concentrated. The residue was purified by flash column chromatography (silica gel/50% methylene chloride in hexanes) to provide 0.172 g (0.42 mmol, 76%) of **24** as a pale yellow solid: ^{1}H ($CDCl_3$, 270 MHz) δ 8.69 (1 H, d, J = 8.9 Hz), 8.03 (1 H, d, J = 8.9 Hz), 7.92 (1 H, d, J = 7.9 Hz), 7.47 (1 H, d, J = 7.4 Hz), 7.48−7.12 (8 H, m), 7.05 (1 H, d, J = 8.4 Hz), 6.83 (1 H, t, J = 7.4 Hz), 6.21 (1 H, d, J = 7.9 Hz), 4.89 (1 H, br s), 4.59 (1 H, d, J = 21 Hz), 4.54 (1 H, d, J = 21 Hz), 1.97 (9 H, s); ^{13}C ($CDCl_3$, 67.9 MHz) δ 151.1, 144.2, 142.8, 141.1, 139.3, 138.2, 134.6, 133.5, 132.0, 130.1, 129.3, 128.3, 128.1, 127.5, 126.8, 126.6, 125.1, 124.8, 124.1, 123.9, 123.6, 123.0, 117.6, 117.5, 40.3, 39.1, 34.4; HRMS calcd for $C_{31}H_{26}ONa$ (MNa^+) 437.1881, found 437.1877.

(1*S*)-Camphanates of 24. To a solution of 0.039 g of **24** (0.095 mmol) and 0.2 mL of triethylamine (0.4 mmol) in 3 mL of anhydrous methylene chloride at room temperature under a nitrogen atmosphere, was added 0.062 g of (1*S*)-(−)-camphanoyl chloride (0.29 mmol). The mixture was stirred for 12 h and then quenched with a saturated sodium bicarbonate solution. After 5 mL of methylene chloride was added, the organic layer was separated and the aqueous layer back extracted with methylene chloride. The combined organic extracts were washed successively with brine and water, dried over sodium sulfate, and concentrated. The residue was purified by flash column chromatography (silica gel/20% ethyl acetate in hexanes). A small fraction of the eluent contained partially separated (1*S*)-camphanates in a 5:1 ratio. The solvent of this fraction was evaporated to afford a white solid: ^{1}H ($CDCl_3$, 600 MHz) δ 8.54 (1 H, d, J = 9.0 Hz), 8.08 (1 H, d, J = 9.0 Hz), 7.96 (1 H, d, J = 8.4 Hz), 7.48 (1 H, dd, J = 9.0, 1.8 Hz), 7.43 (1 H,

t, J = 7.2 Hz), 7.39−7.32 (2 H, m), 7.29−7.16 (4 H, m), 7.13 (1 H, t, J = 7.2 Hz), 7.05 (1 H, t, J = 7.8 Hz), 5.96 (1 H, d, J = 8.4 Hz), 4.47 (1 H, d, J = 21.6 Hz), 4.41 (1 H, d, J = 21.0 Hz), 1.87 (9 H, s), 1.63−1.57 (1 H, m), 1.50−1.45 (2 H, m), 1.38−1.32 (1 H, m), 0.80 (3 H, s), 0.07 (3 H, s), 0.00 (3 H, s). A minor set of the ^{1}H NMR signals at δ 5.93 (1 H, d, J = 8.4 Hz), 4.48 (1 H, d, J = 21.6 Hz), 4.43 (1 H, d, J = 21.0 Hz), 0.02 (3 H, s), and 0.01 (3 H, s) attributable to the other camphanate diastereomer were also observed.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X : parts per Million : 1
7.6 7.5 7.4 7.3 7.2 7.1 7 6.9

OMe
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5a
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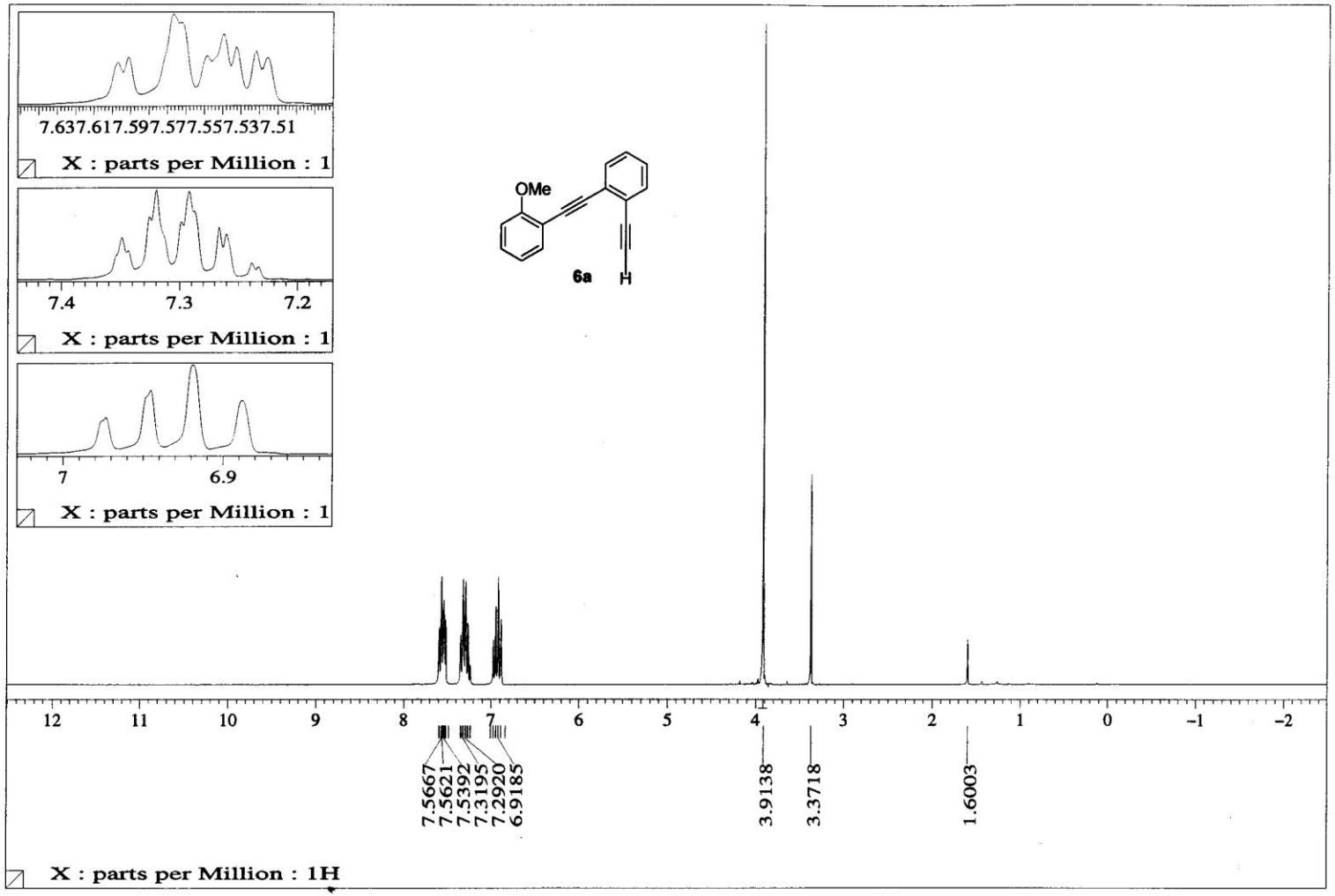
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H
12
11
10
9
8
7
6
5
4
3
2
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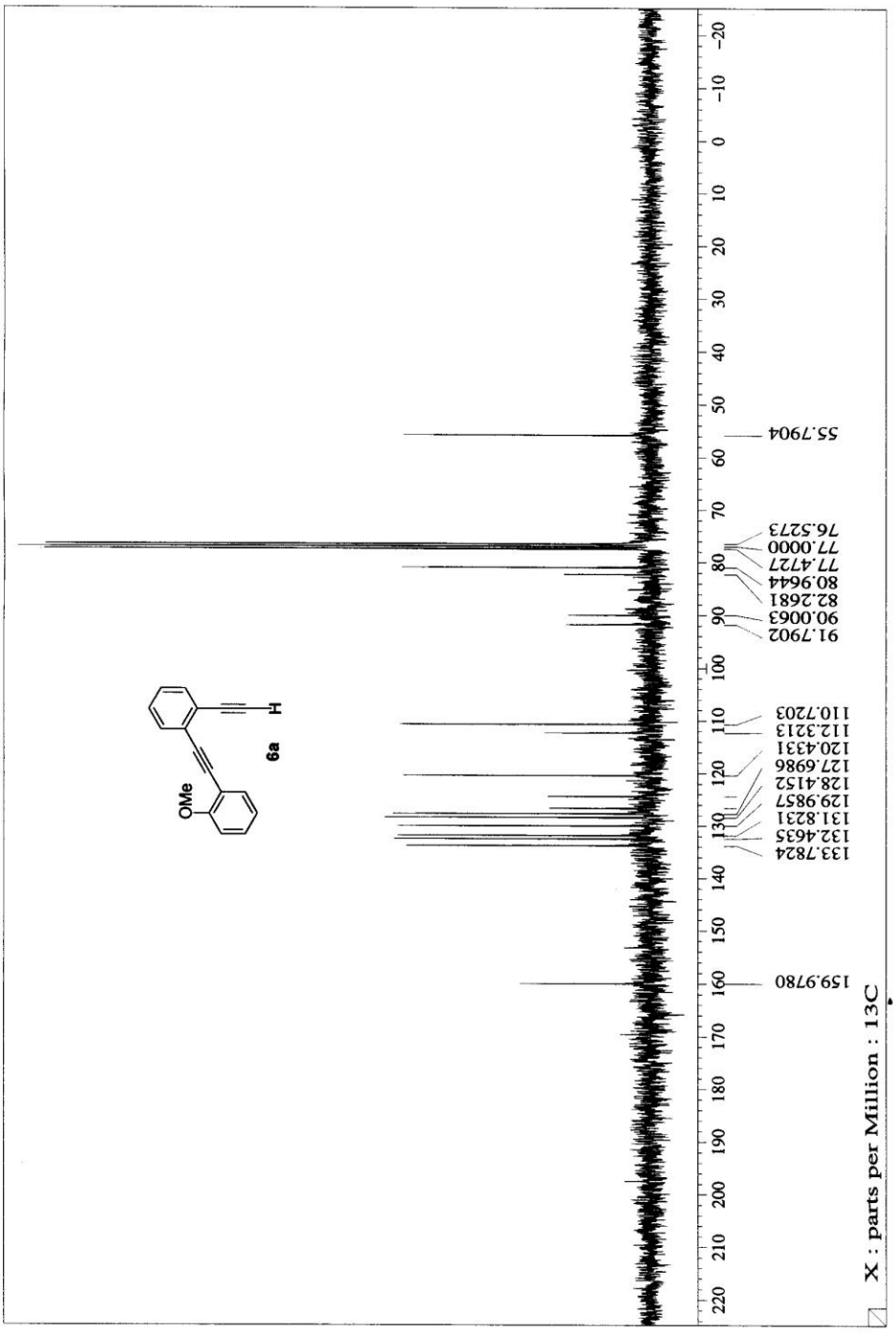
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H
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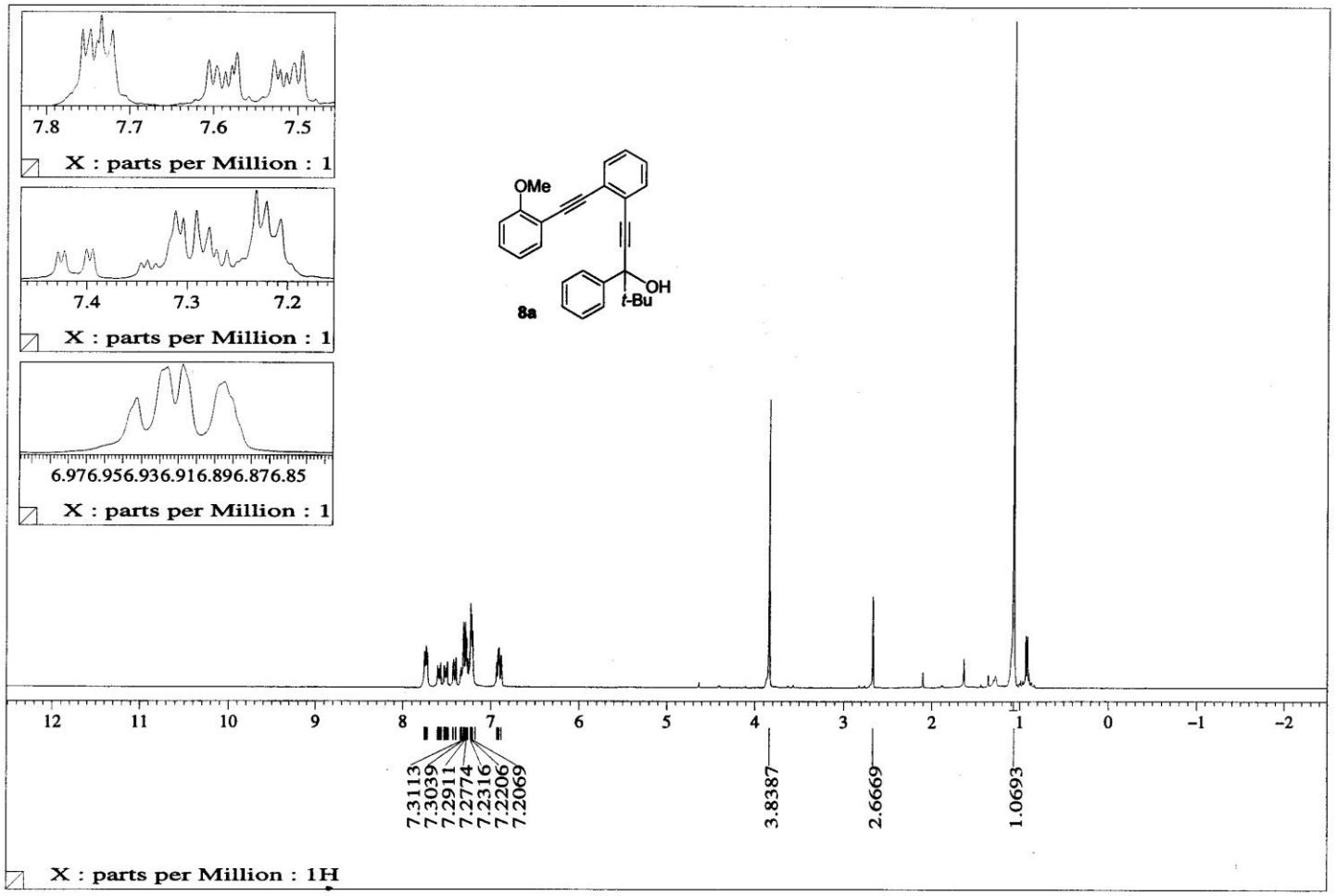
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t-Bu
8a
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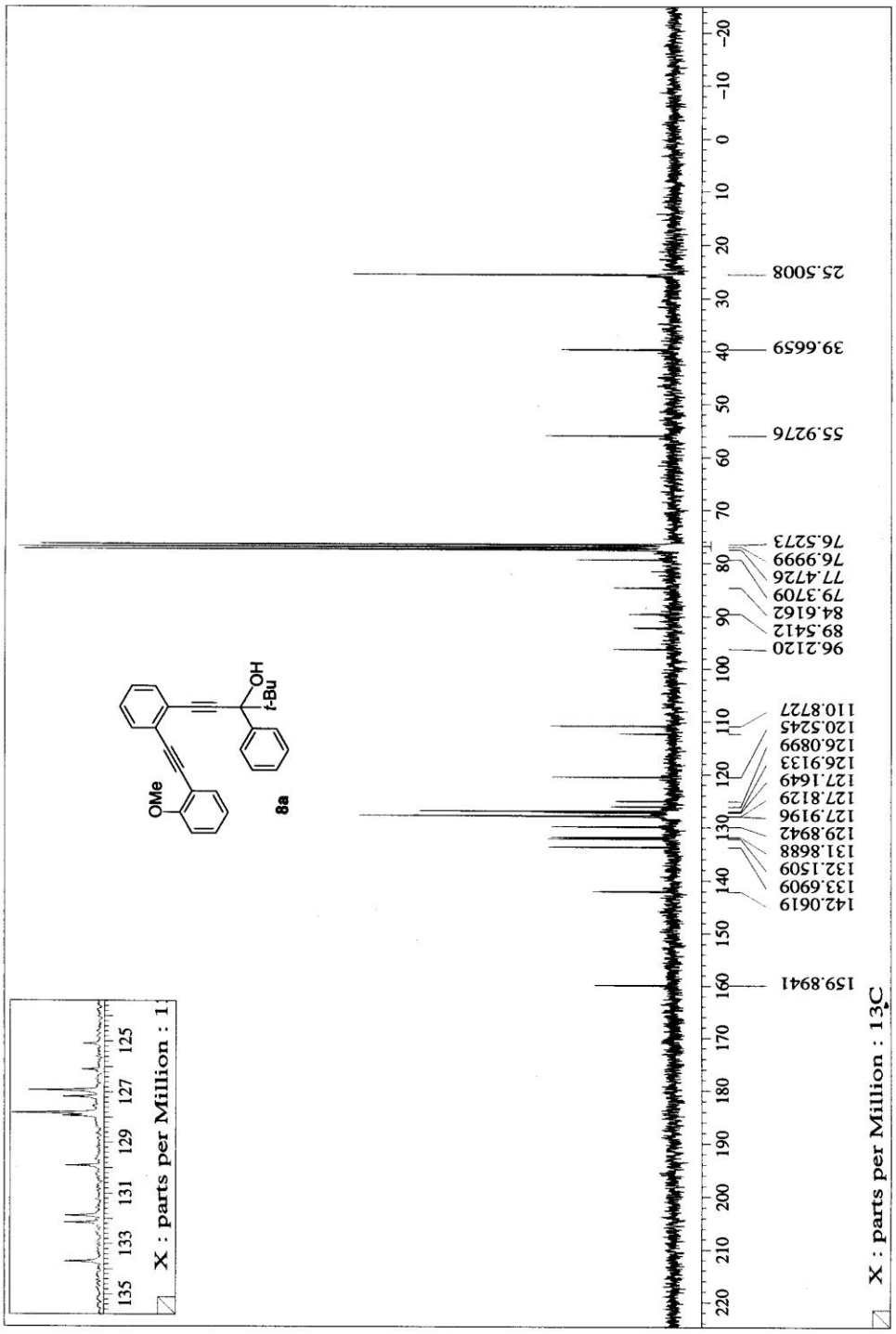
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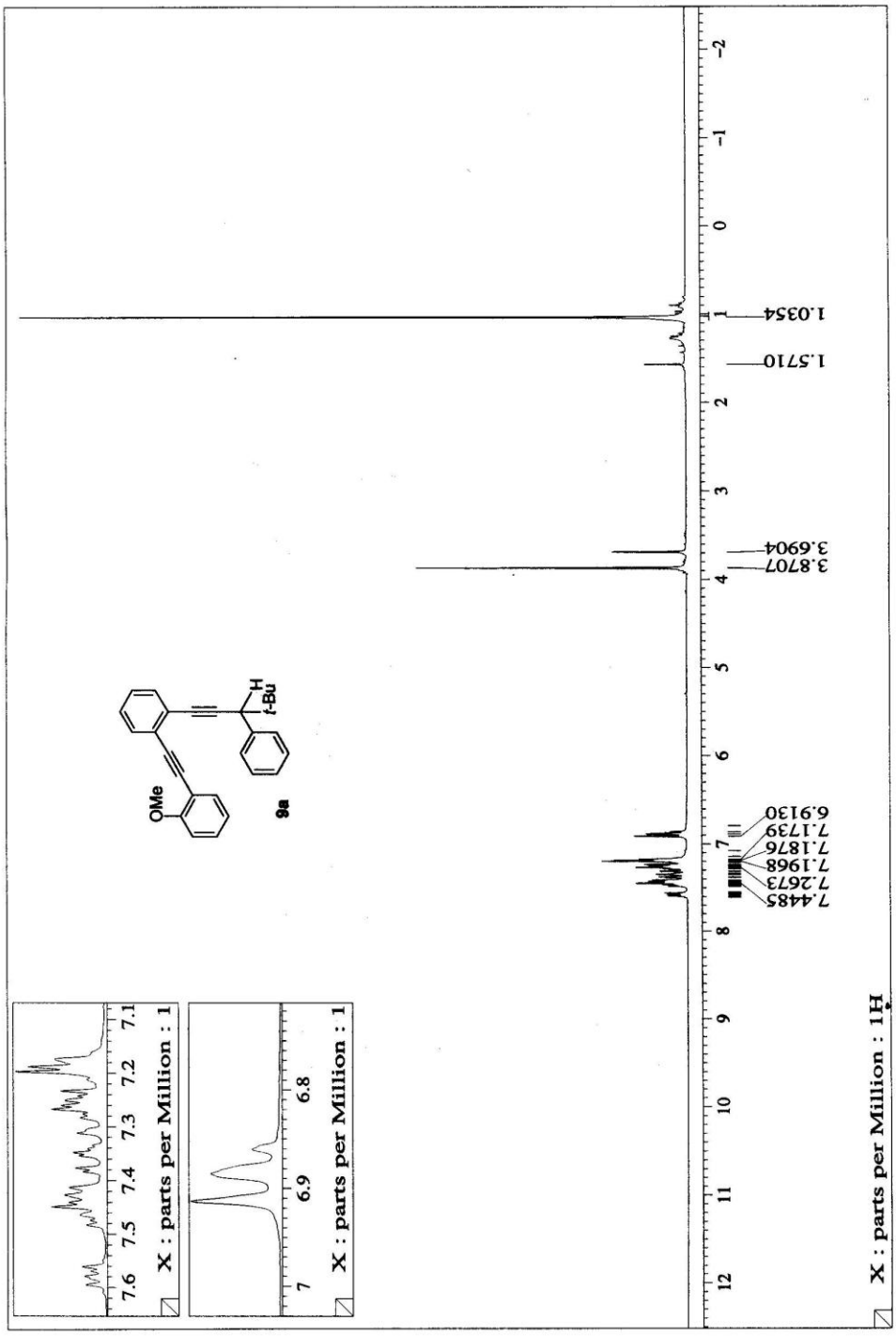
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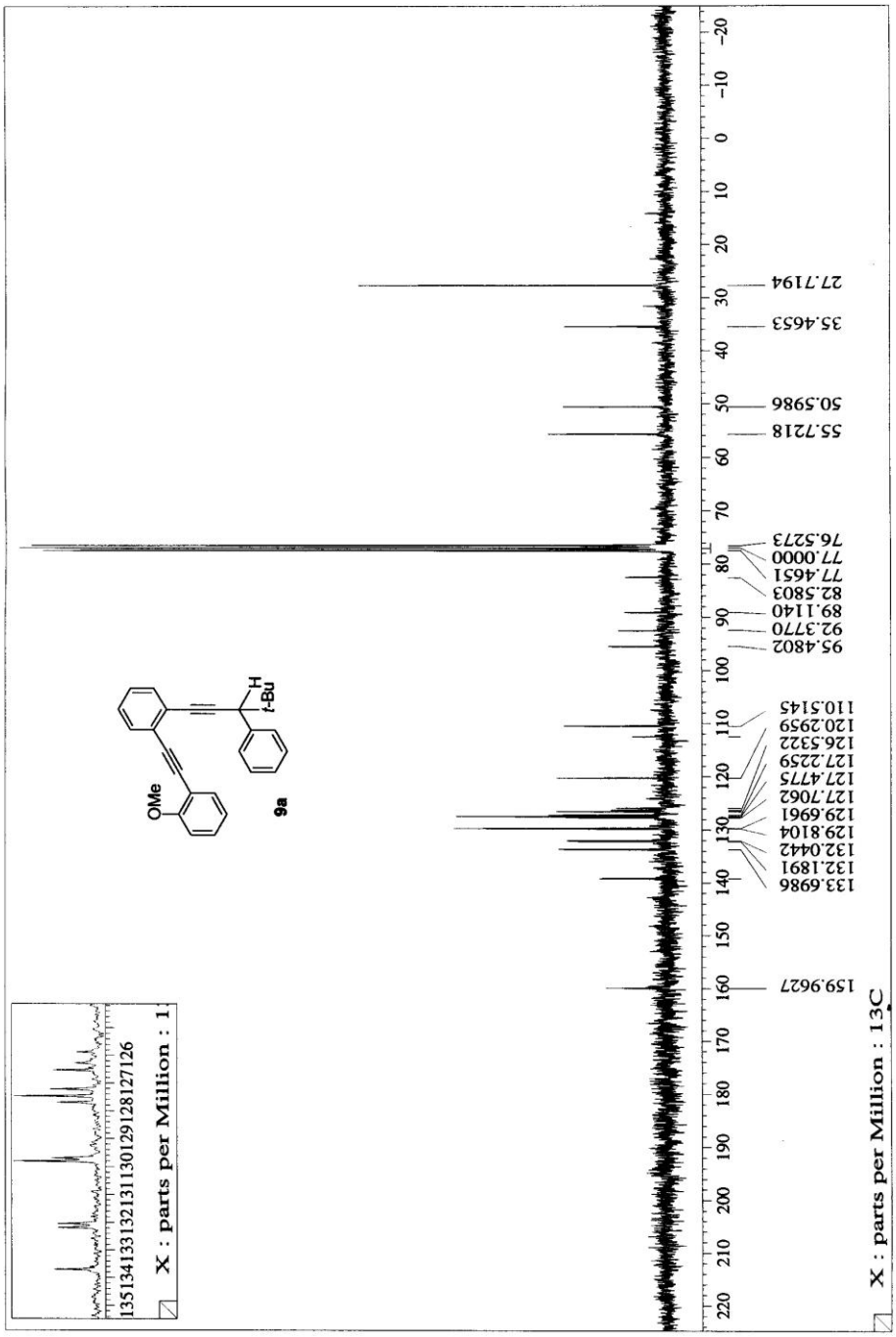
X : parts per Million : 1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H
9a
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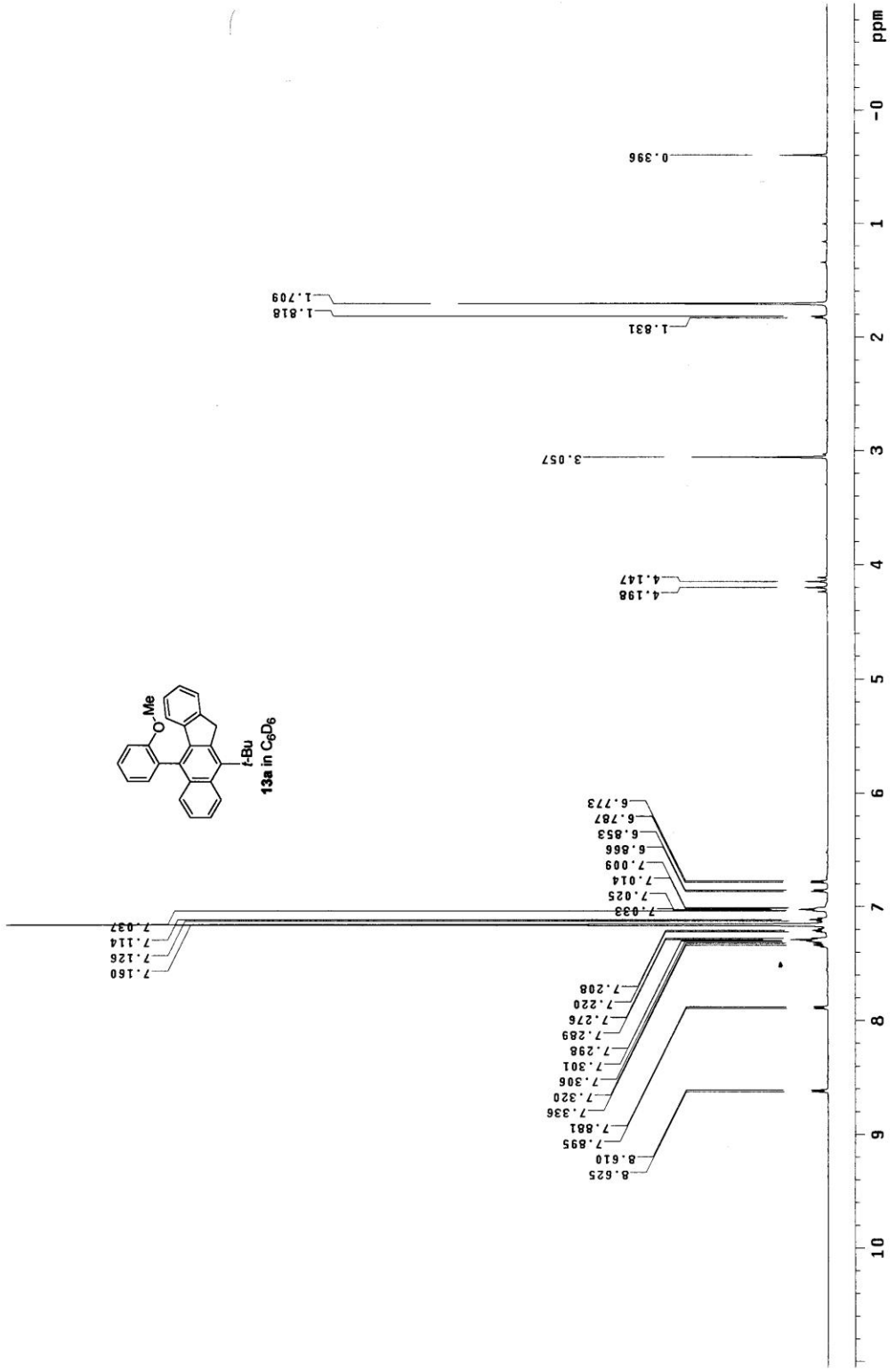
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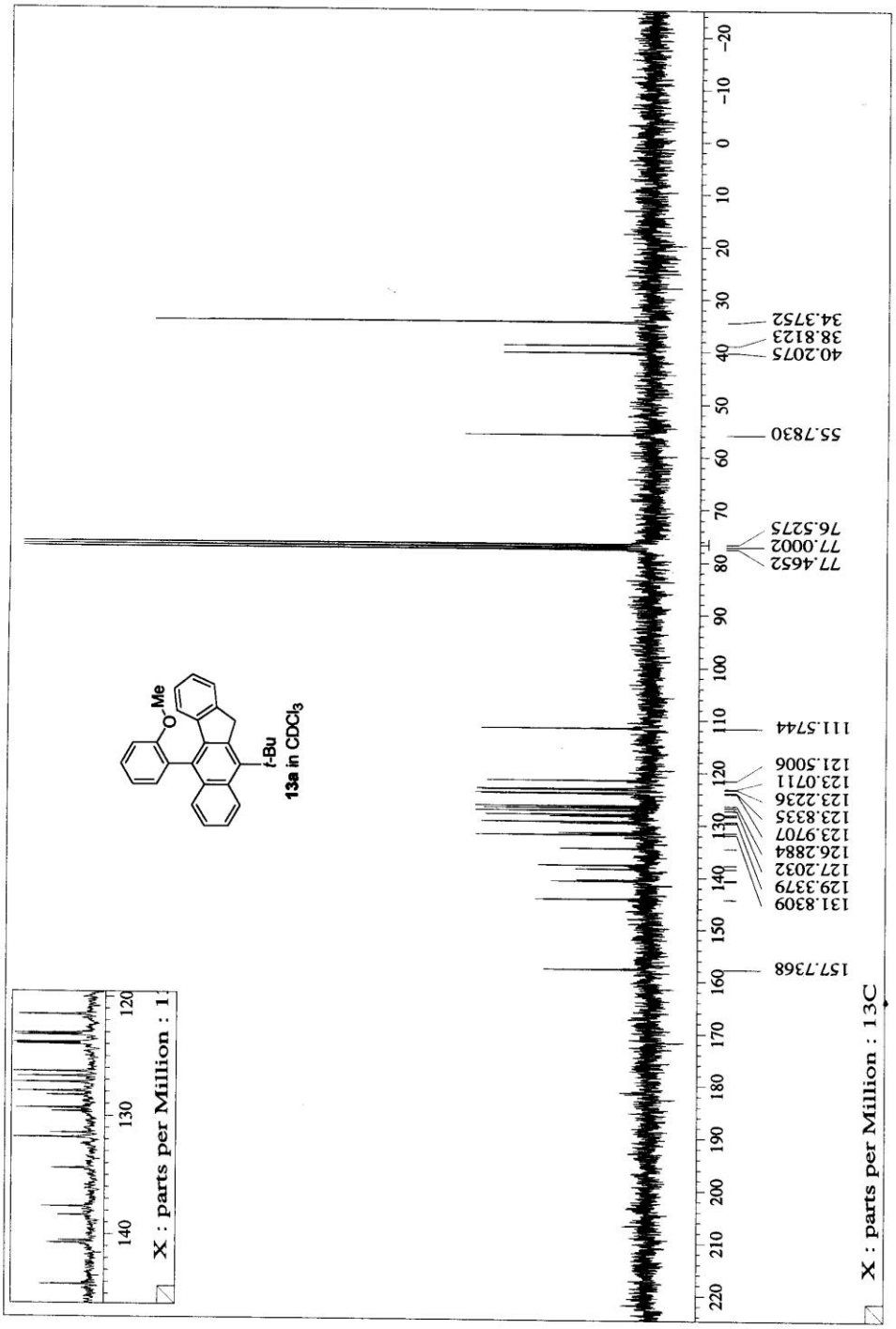
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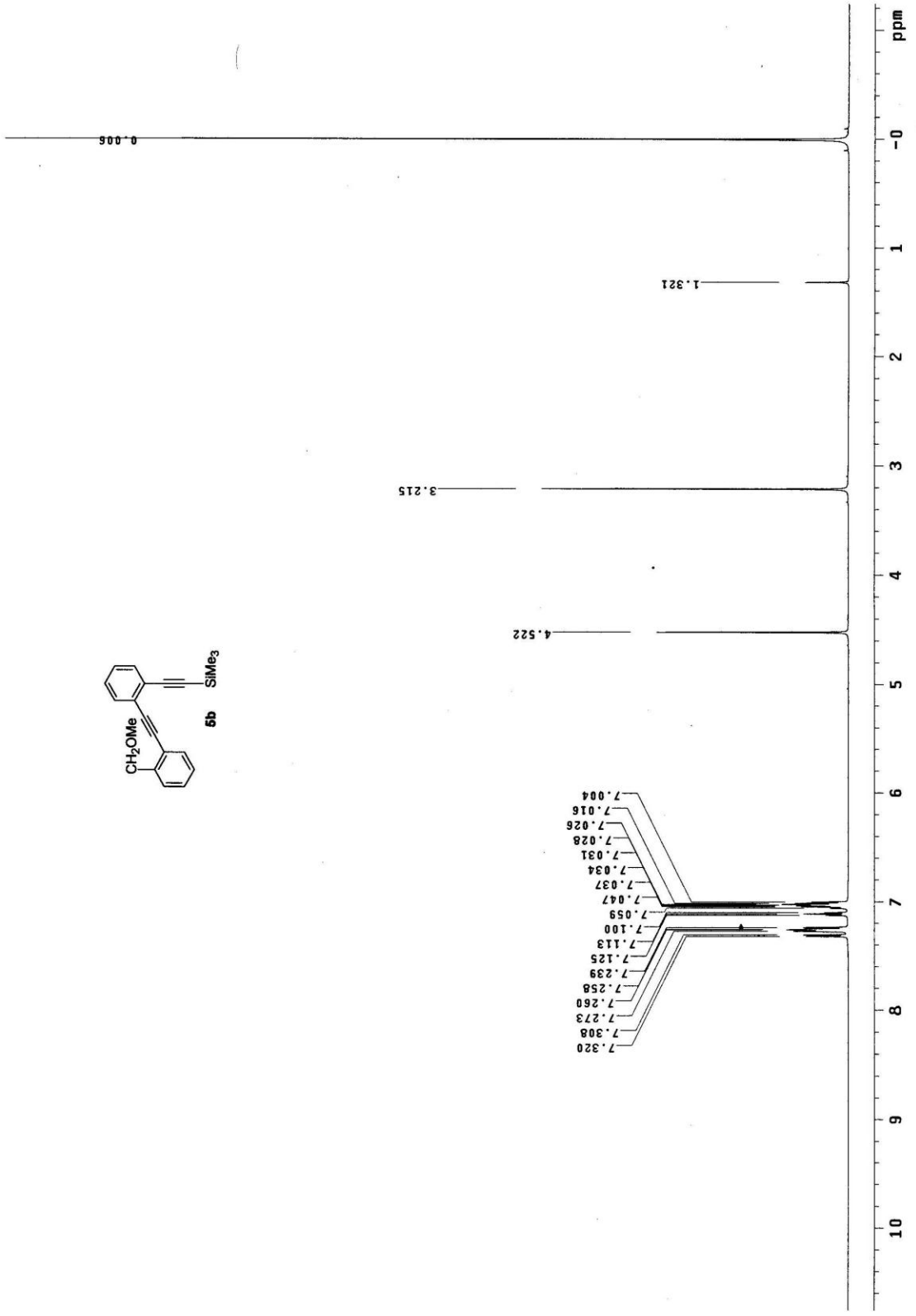
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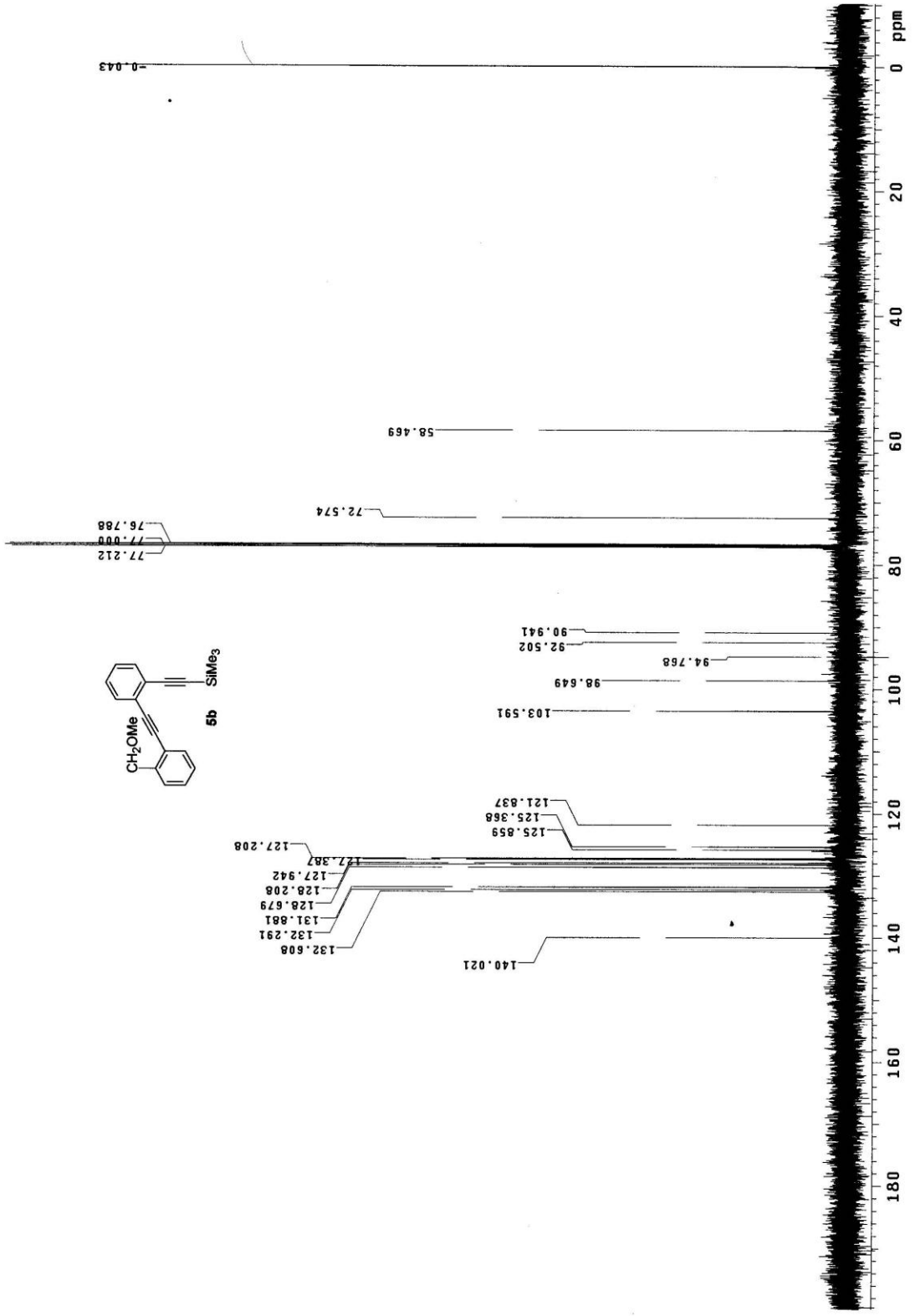
5b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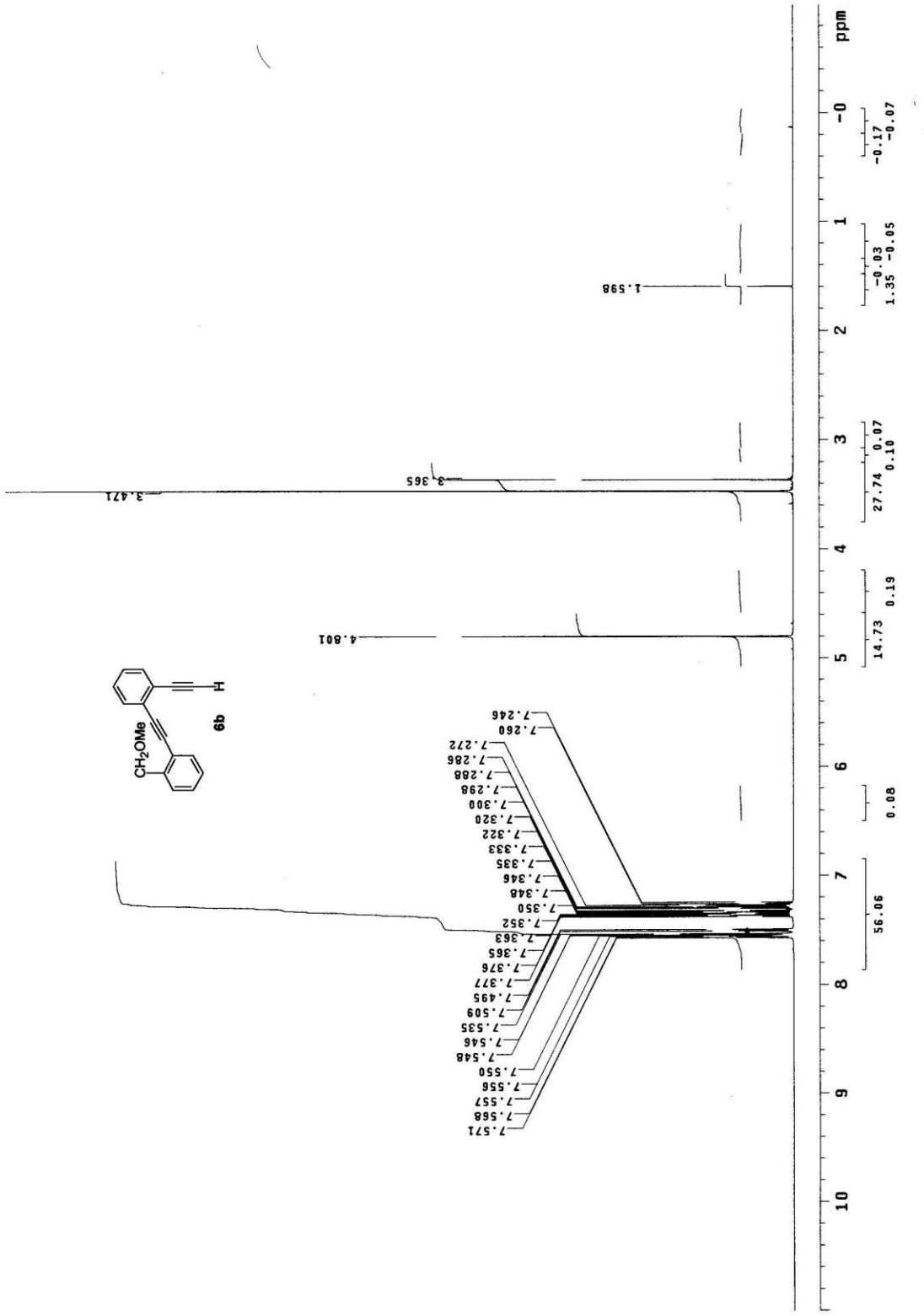
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6b
CH2OMe
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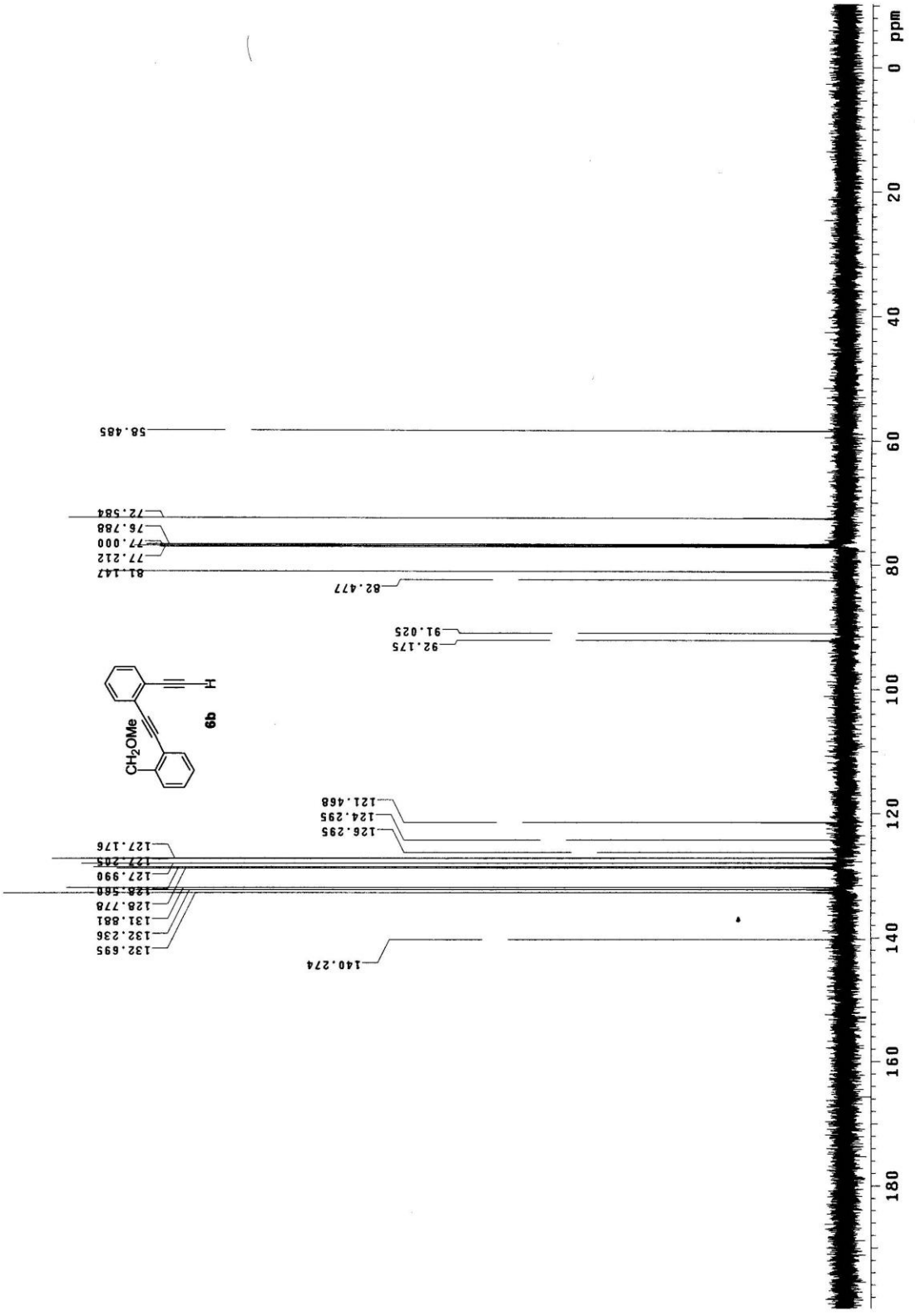
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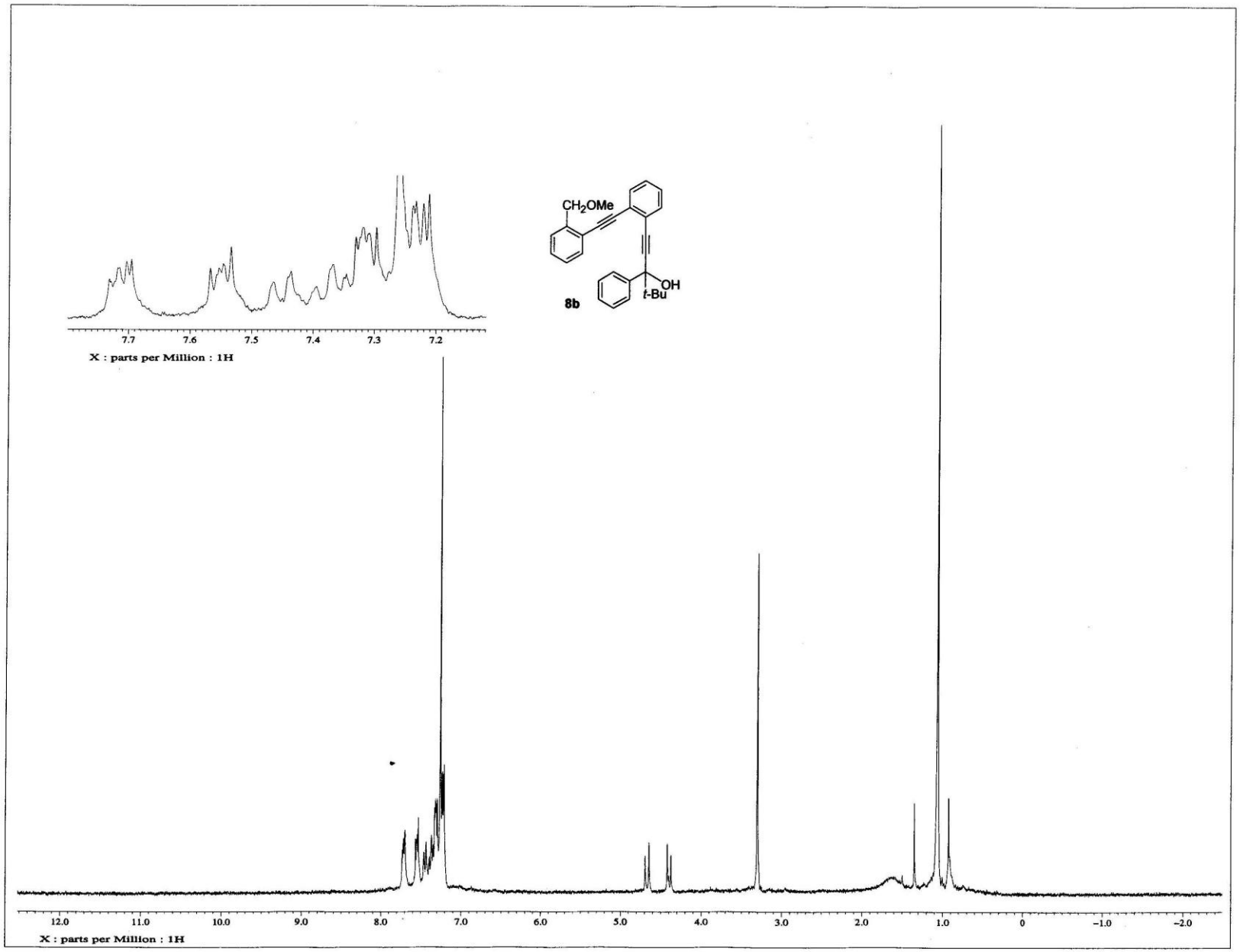
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t-Bu
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7.3
7.2
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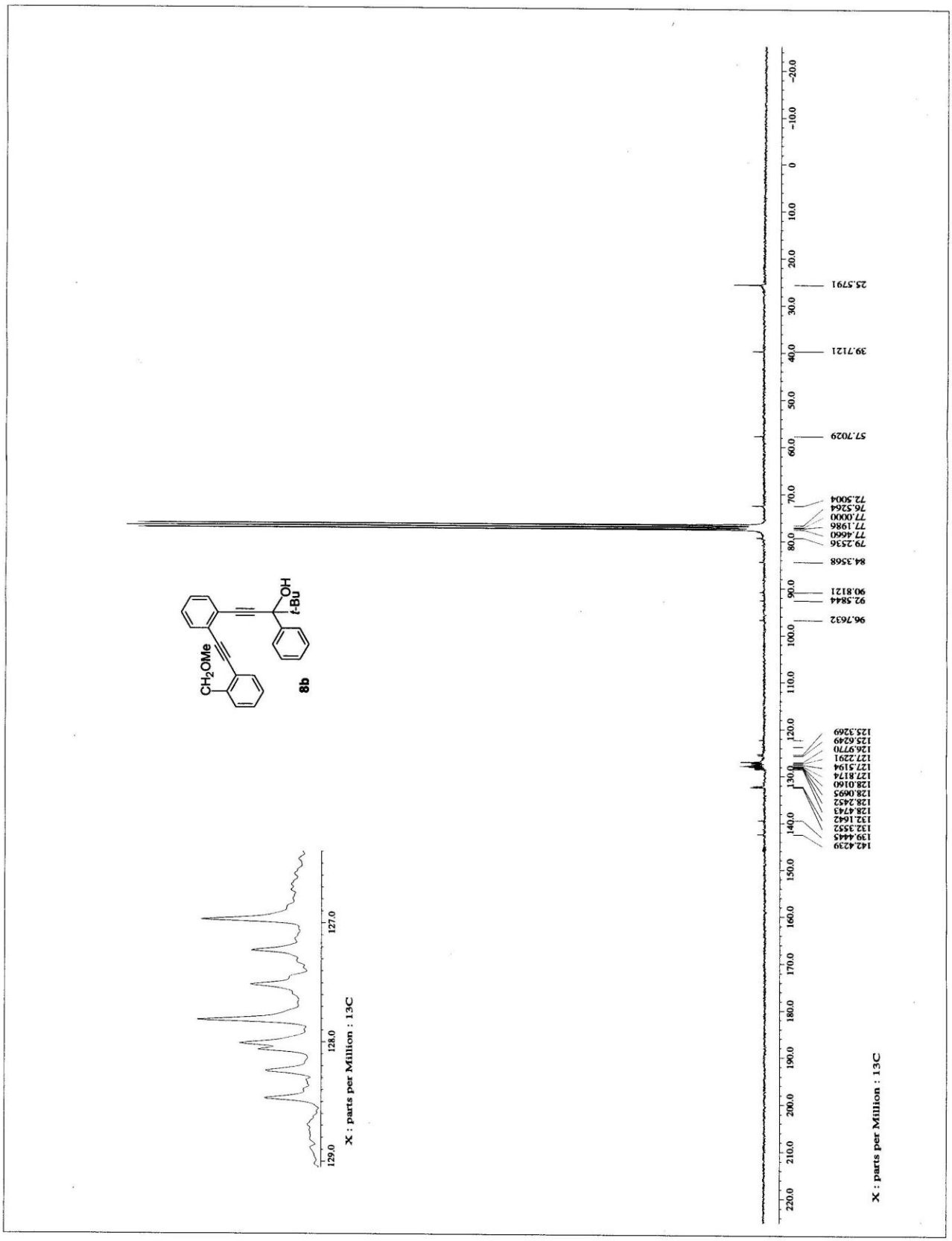
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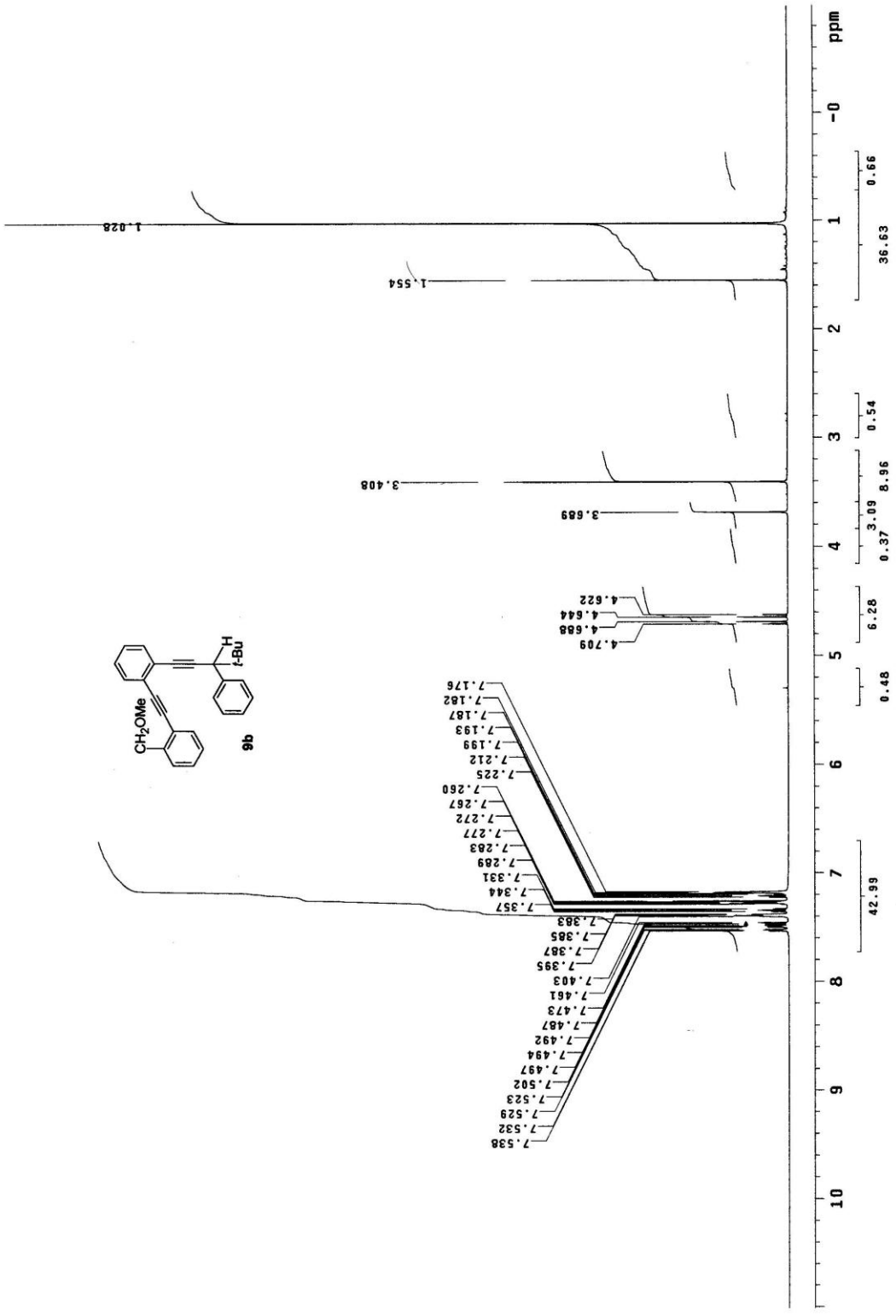
X : parts per Million : 13C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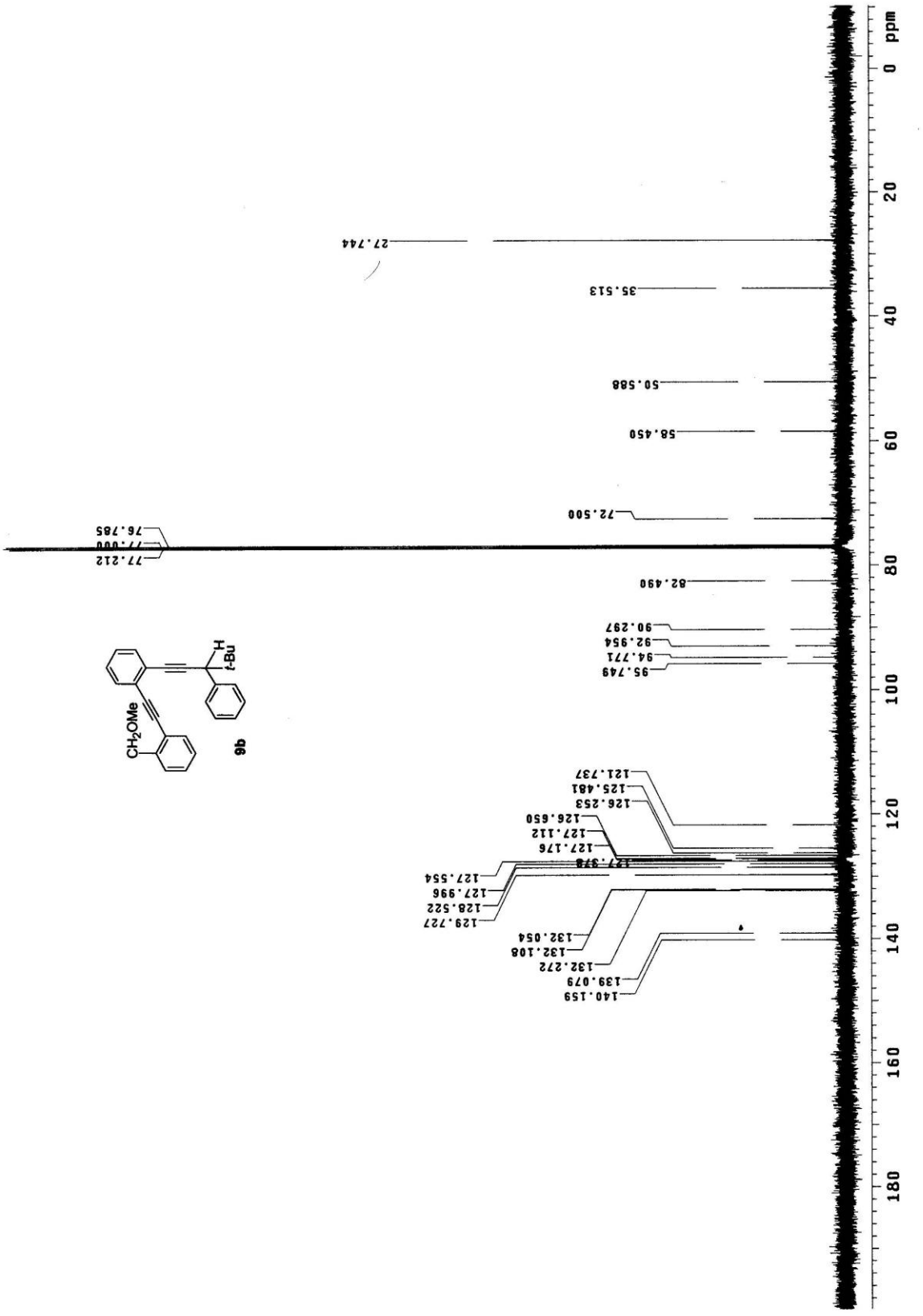
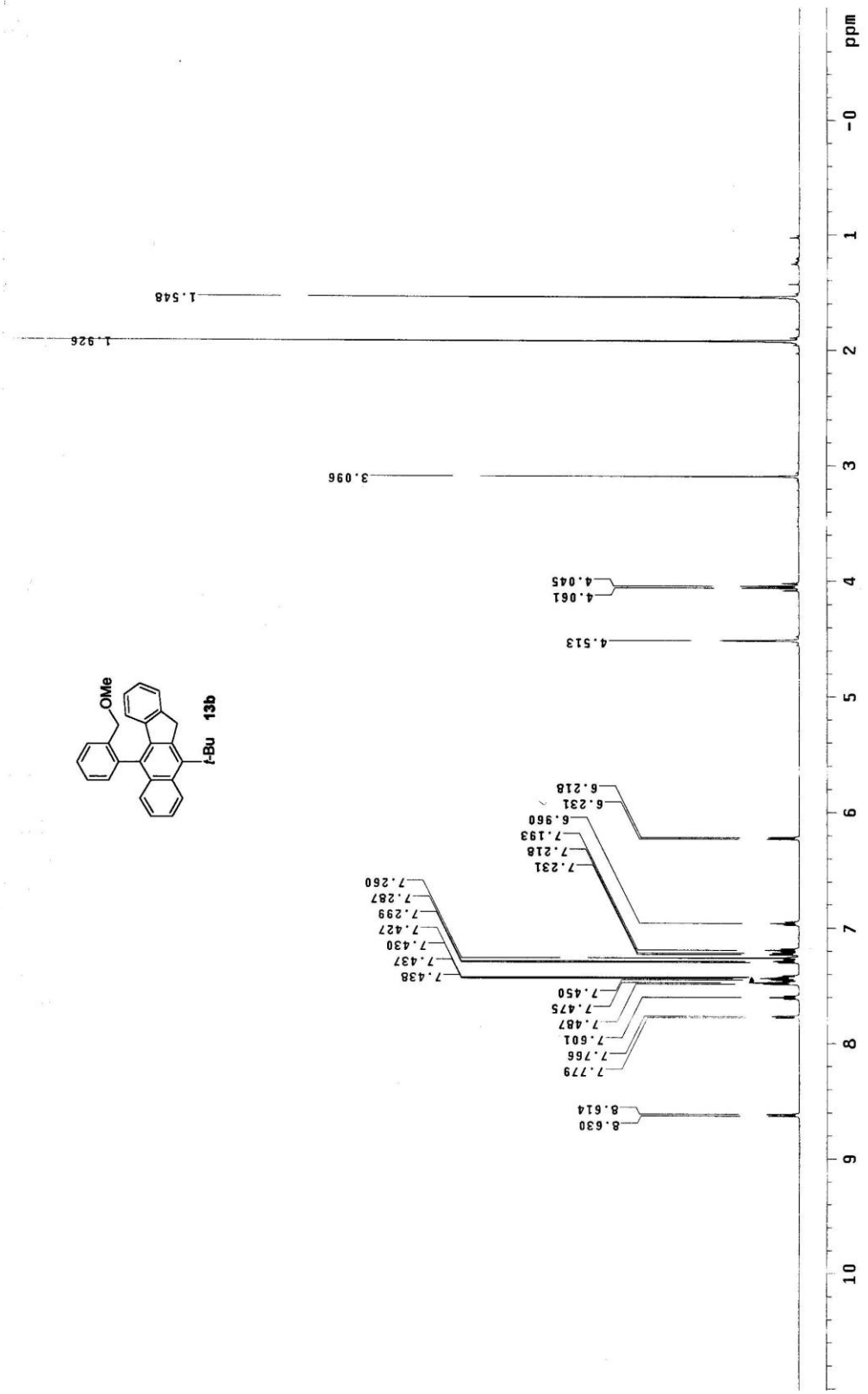
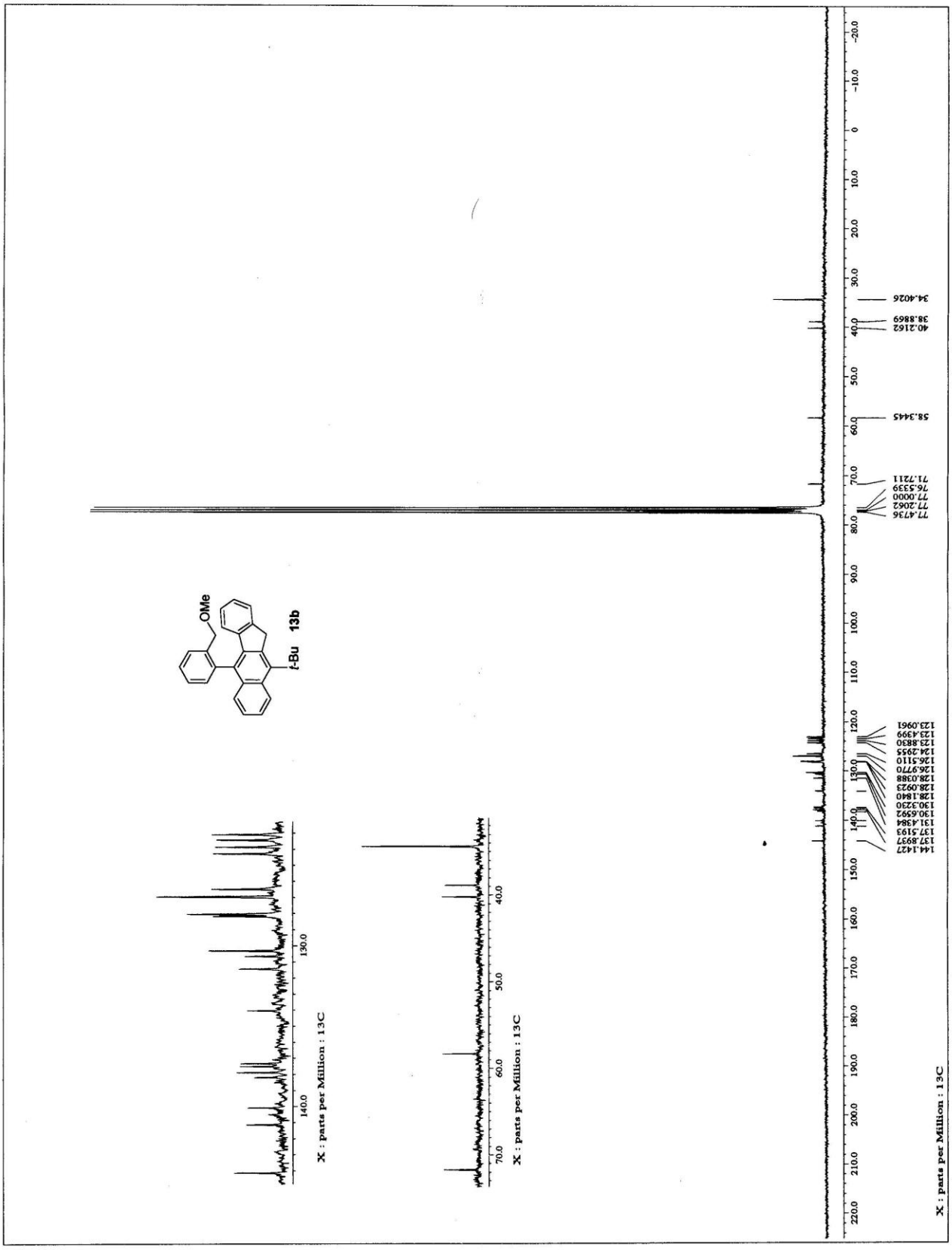
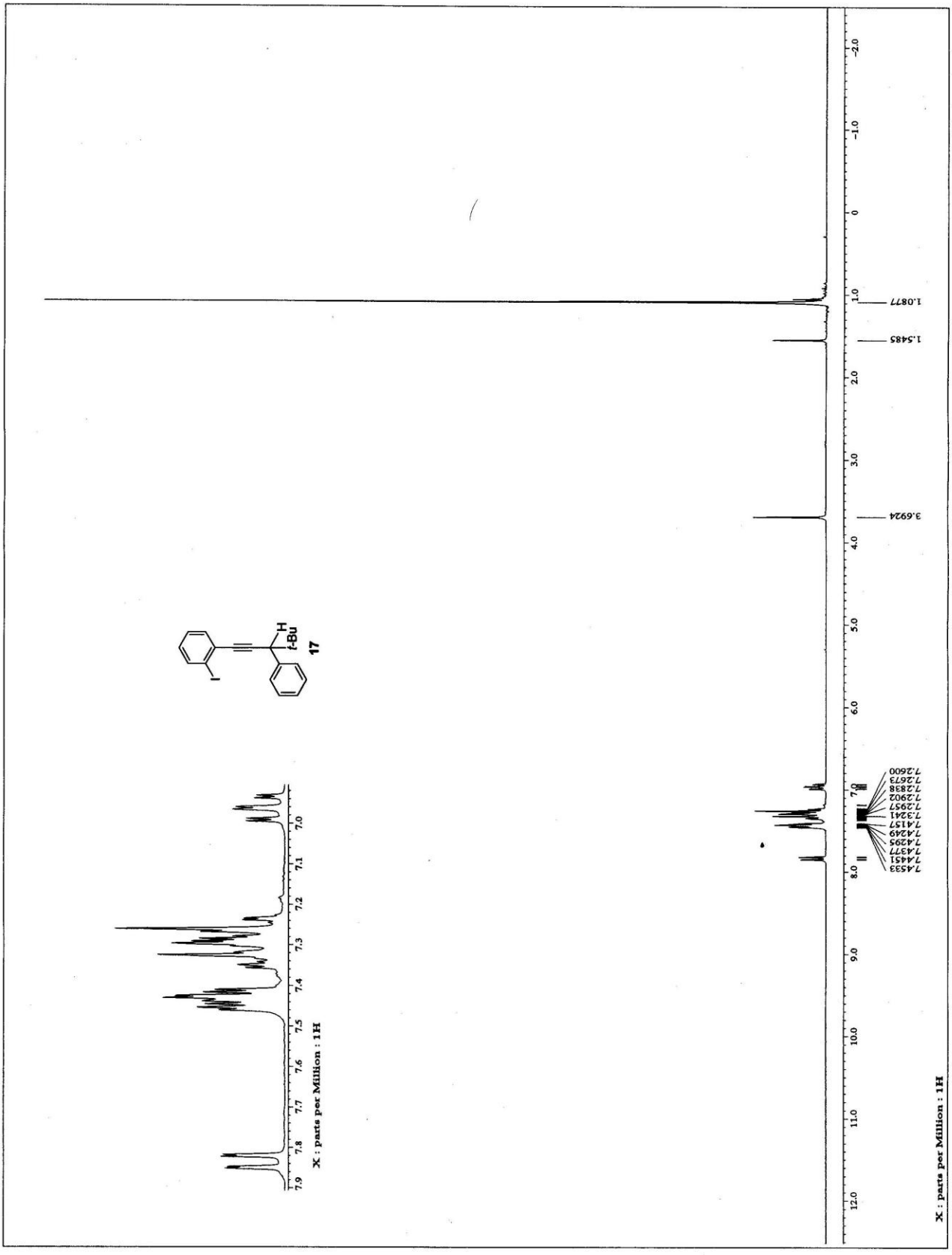
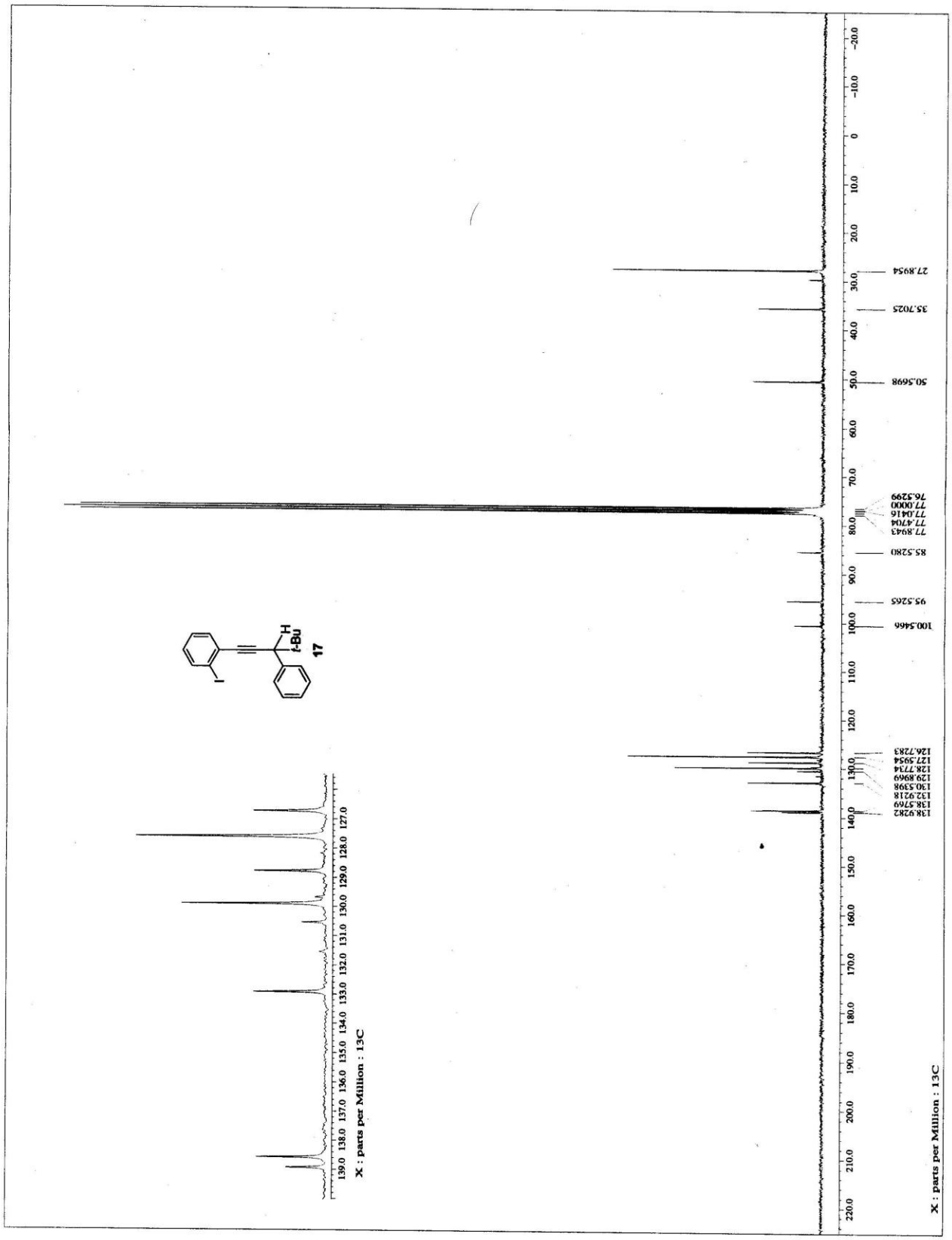
7.494
7.497
7.502
7.523
7.529
7.532
7.538
t-Bu
CH2OMe
9b
ppm
0.66
36.63
0.54
8.96
3.09
0.37
6.28
0.48
42.99

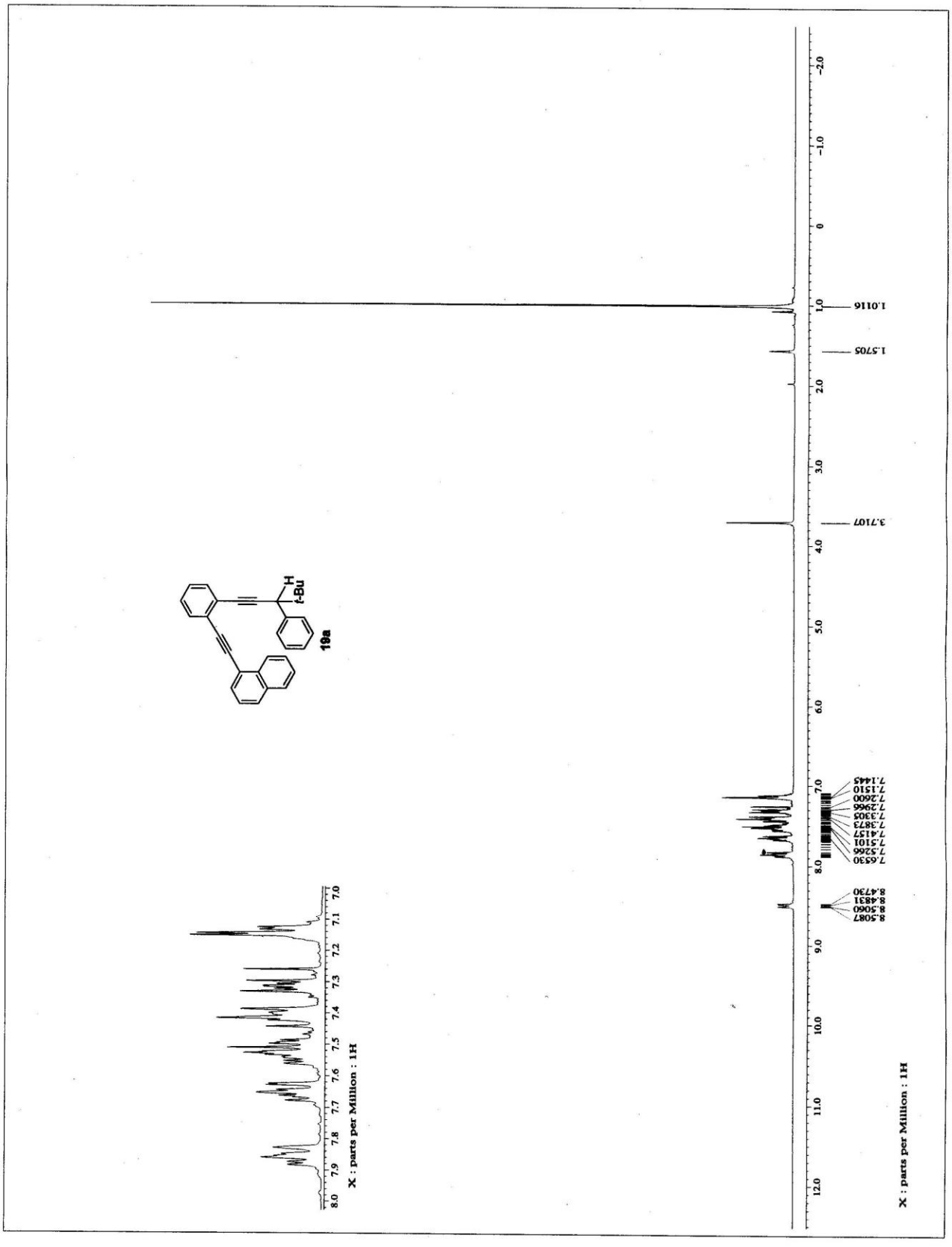

27.744
35.513
50.588
58.450
72.500
76.785
77.000
77.212
82.490
90.297
92.954
94.771
95.749
121.737
125.481
126.253
126.650
127.112
127.176
127.554
127.996
128.522
129.727
132.054
132.108
132.272
139.079
140.159
CH2OMe
t-Bu
9b
0
20
40
60
80
100
120
140
160
180
ppm


1.548
1.926
3.096
4.045
4.061
4.513
6.218
6.231
6.960
7.193
7.218
7.231
7.260
7.287
7.299
7.427
7.430
7.437
7.438
7.450
7.475
7.487
7.601
7.766
7.779
8.614
8.630
ppm
OMe
t-Bu
13b

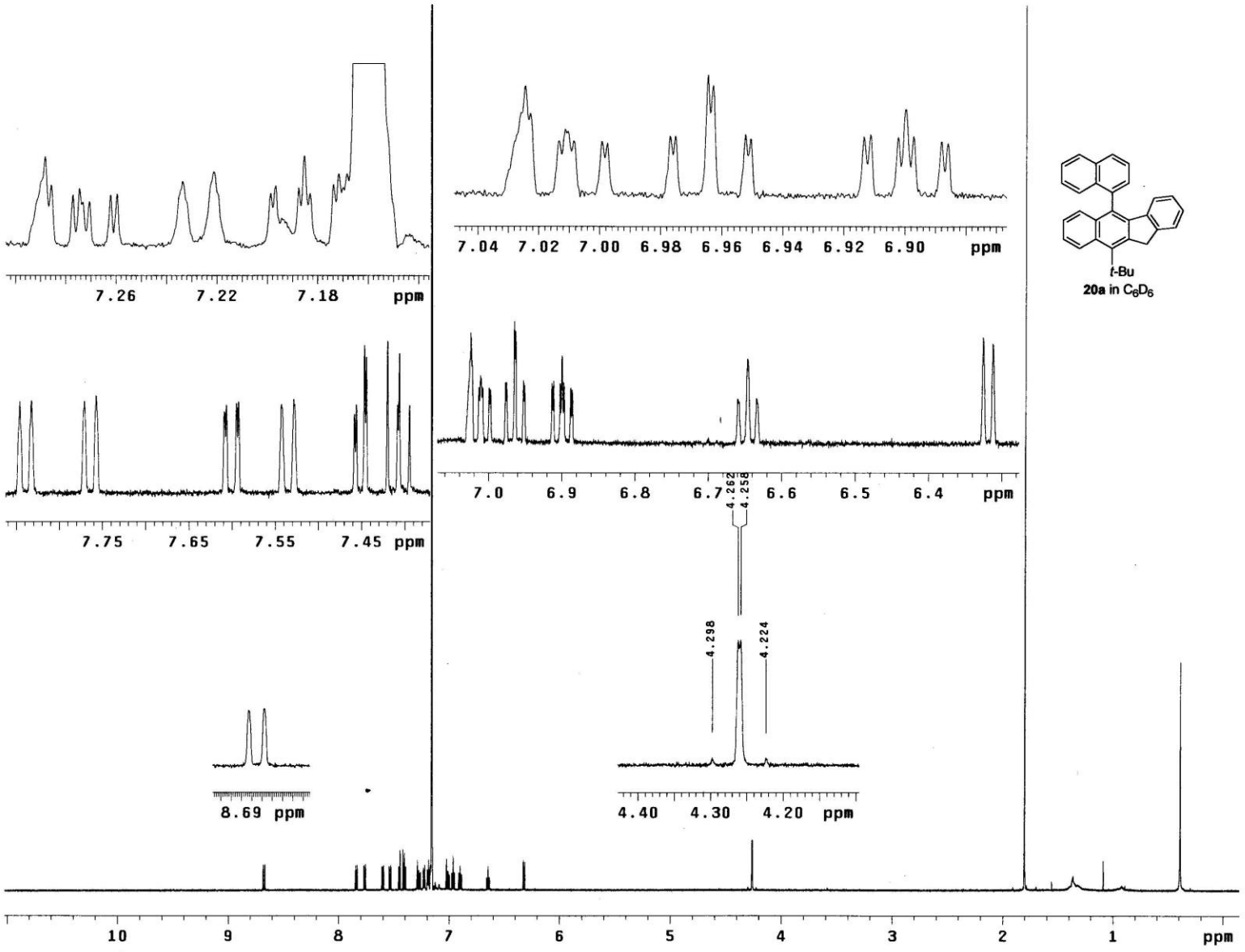

OMe
t-Bu
13b
144.1427
137.8937
137.5193
131.4384
130.3230
130.0992
128.1840
128.0923
128.0388
126.9770
126.5110
124.2955
123.8830
123.4399
123.0961
77.4736
77.2062
77.0000
76.5359
71.7211
58.3445
40.2162
38.8869
34.4026
X : parts per Million : 13C
X : parts per Million : 13C
X : parts per Million : 13C

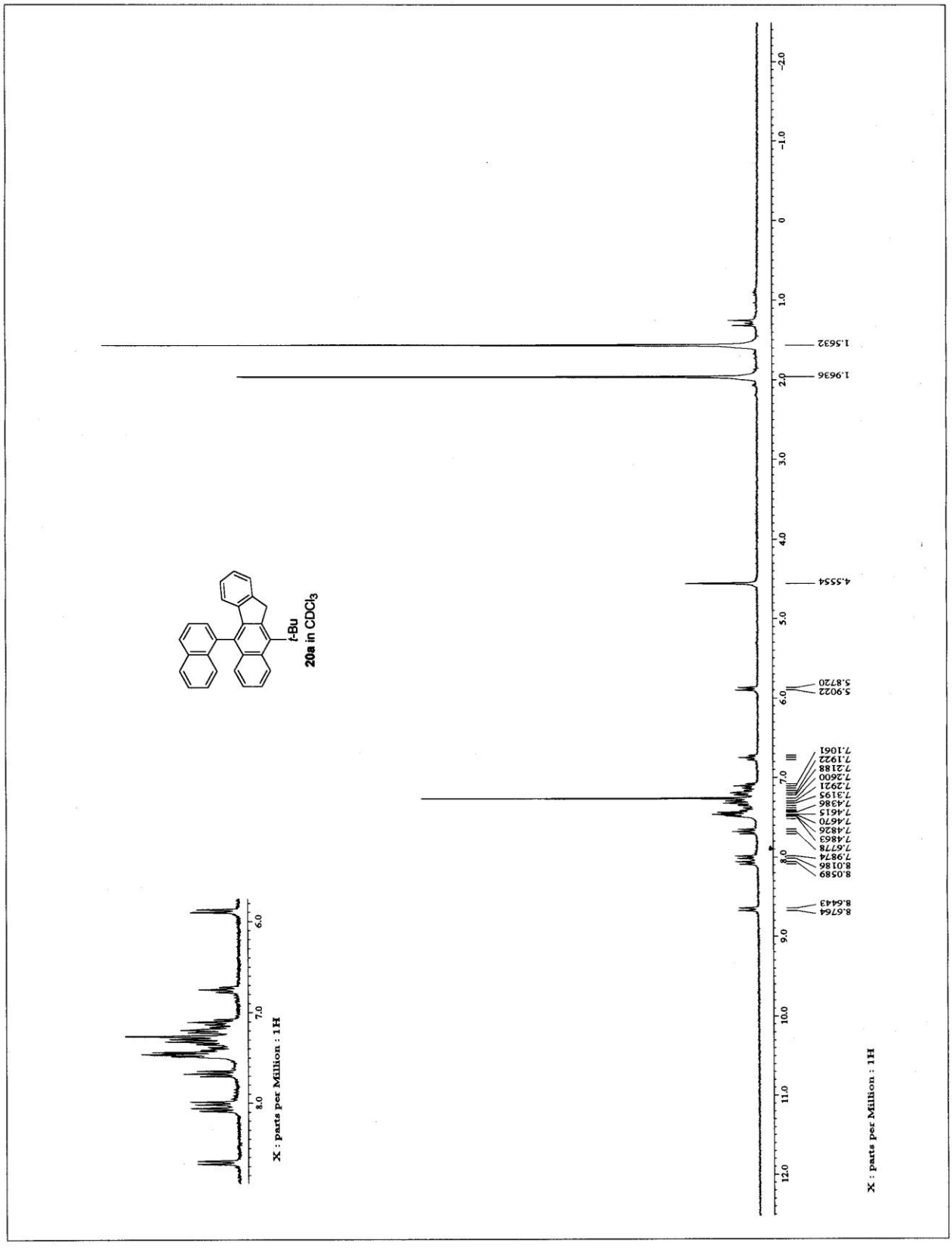

1.0877
1.5485
3.6924
7.4533
7.4451
7.4377
7.4295
7.4249
7.4157
7.3241
7.2957
7.2902
7.2838
7.2673
7.2600
X : parts per Million : 1H
i-Bu
17
X : parts per Million : 1H

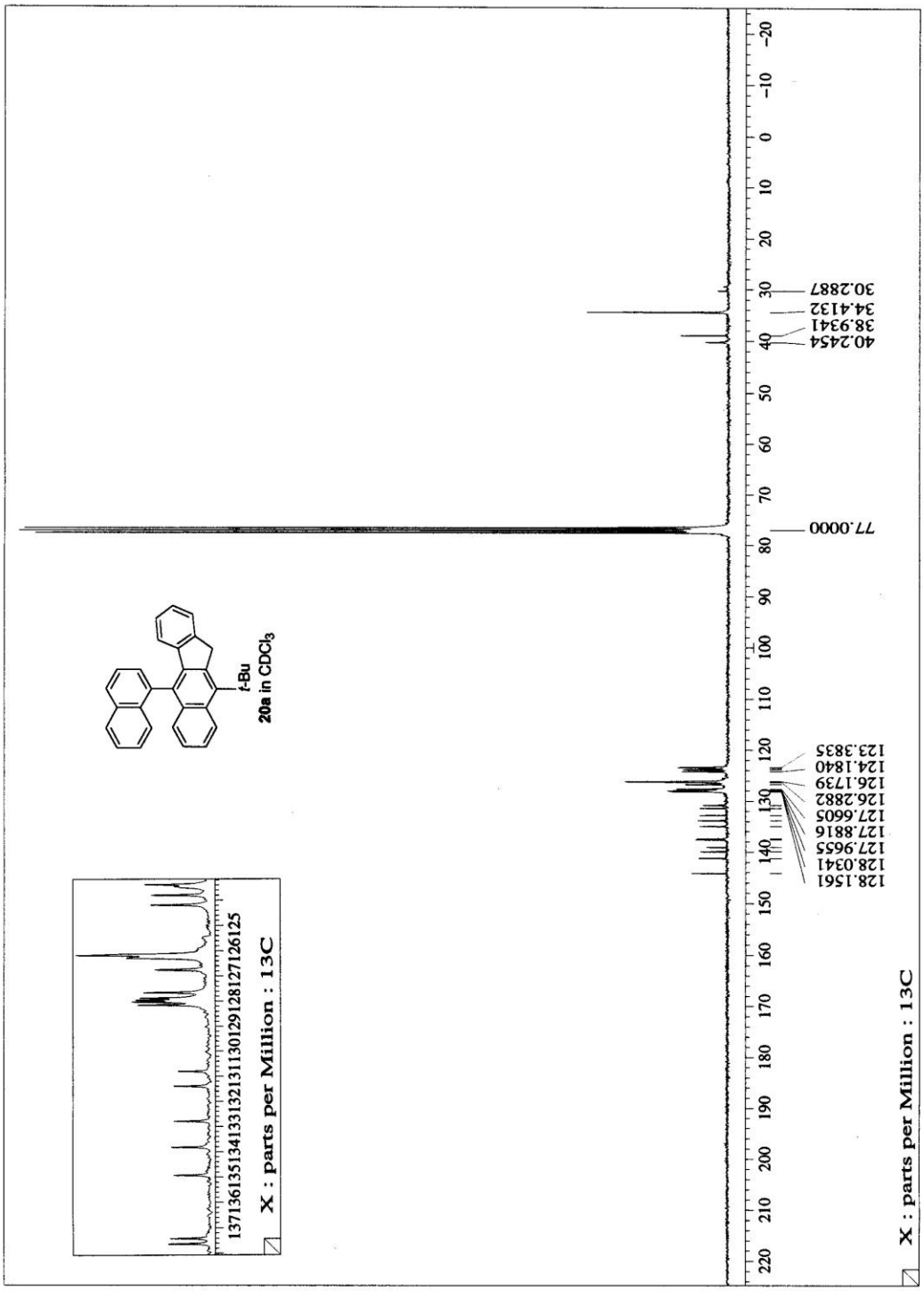

27.8954
35.7025
50.5698
76.3299
77.0000
77.0416
77.4704
77.8943
85.5280
95.5265
100.5466
126.7283
127.3954
128.7734
129.8969
130.5398
132.9218
138.5769
138.9282
X : parts per Million : 13C
t-Bu
17
X : parts per Million : 13C

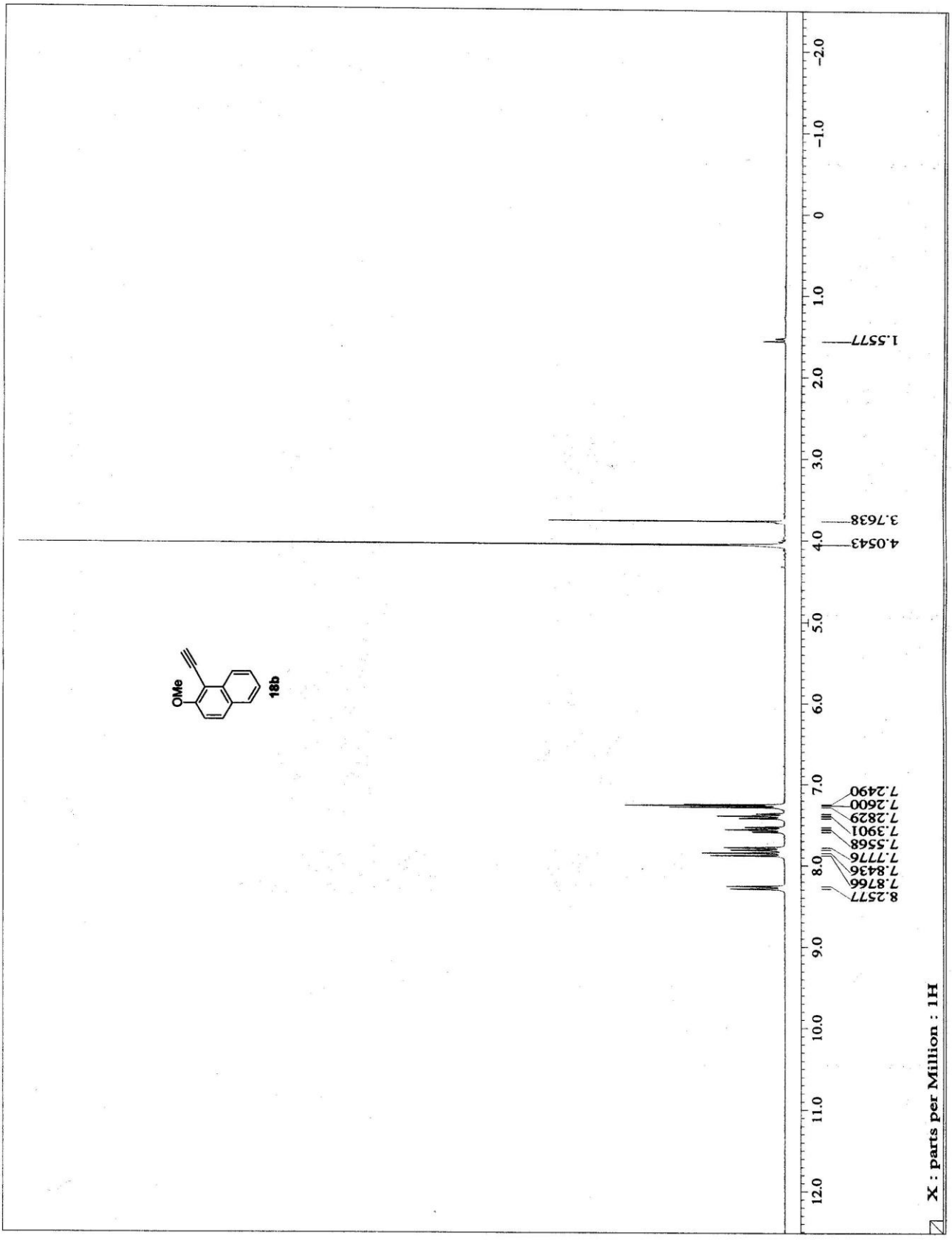

1.0116
1.5705
3.7107
7.6530
7.5266
7.5101
7.4157
7.3873
7.3305
7.2966
7.2600
7.1510
7.1445
8.5087
8.5060
8.4831
8.4730
X : parts per Million : 1H
t-Bu
H
19a
X : parts per Million : 1H

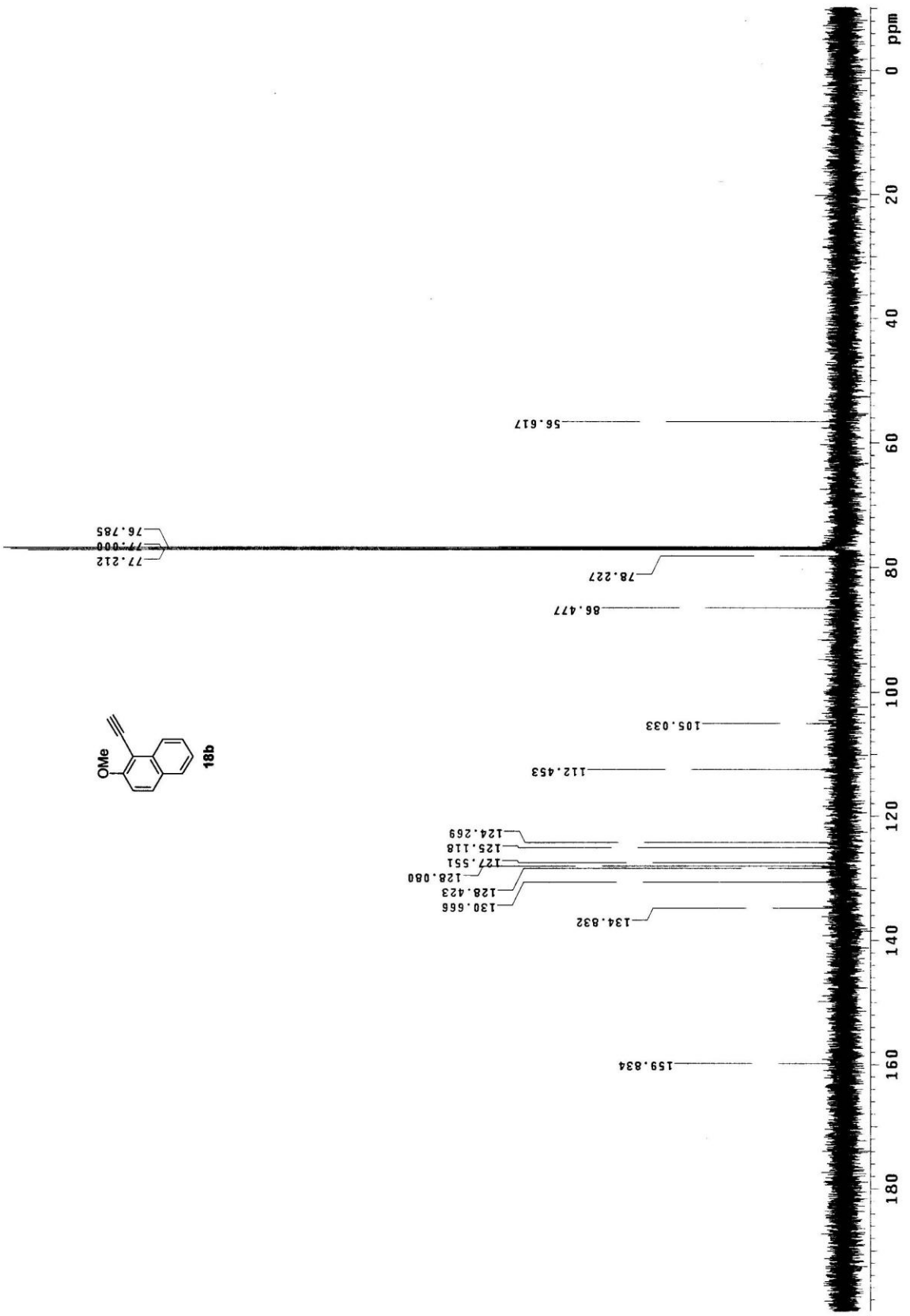
19a
X : parts per Million : 13C
27.7347
35.4958
50.5834
50.5300
76.5274
77.0000
77.4651
82.5502
90.8754
93.3608
95.8309
126.3187
126.4407
126.5551
126.6770
127.4089
127.4852
128.1408
129.7037
X : parts per Million : 13C

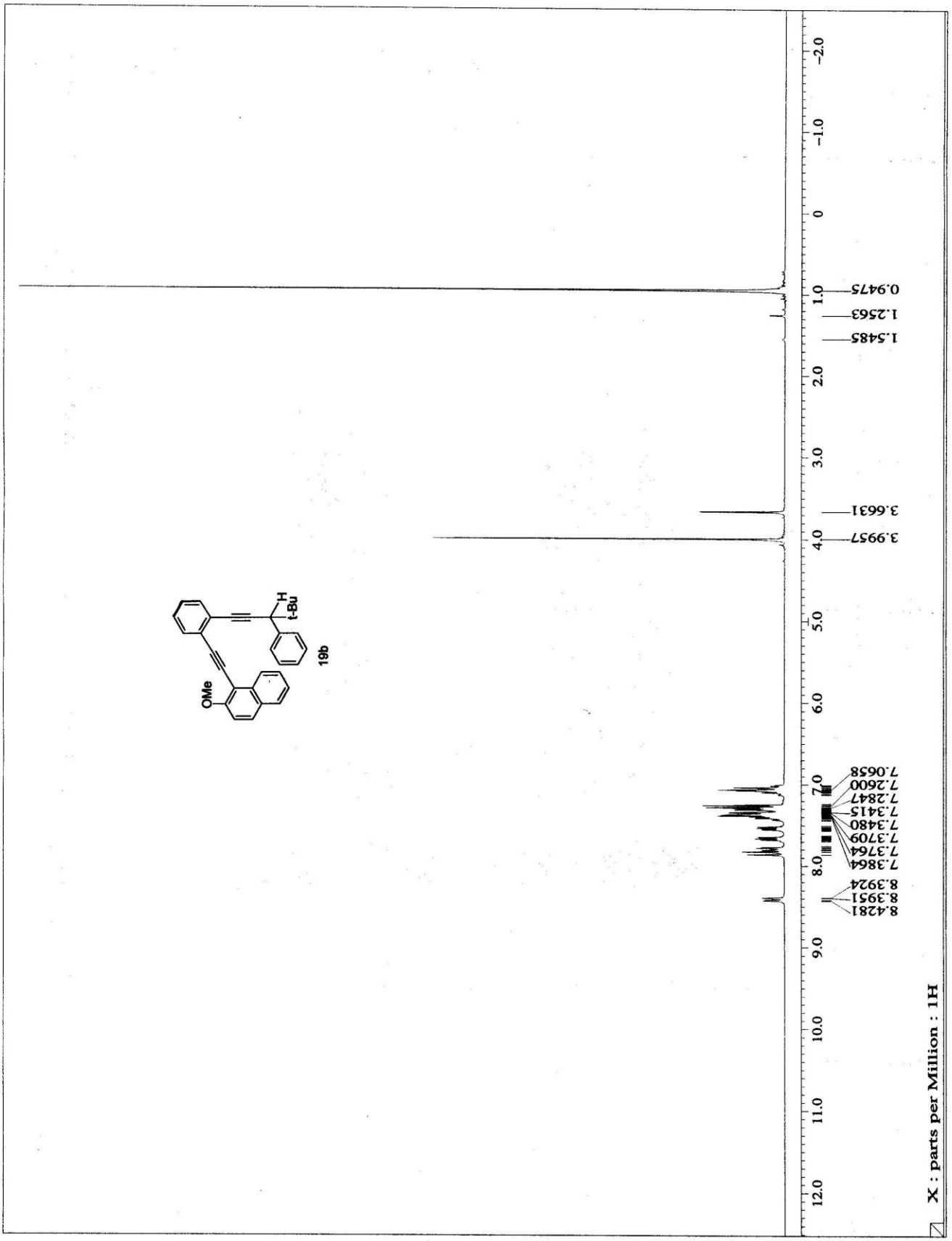

7.26 7.22 7.18 ppm
7.04 7.02 7.00 6.98 6.96 6.94 6.92 6.90 ppm
7.75 7.65 7.55 7.45 ppm
7.0 6.9 6.8 6.7 6.6 6.5 6.4 ppm
4.262
4.258
4.298
4.224
8.69 ppm
4.40 4.30 4.20 ppm
10 9 8 7 6 5 4 3 2 1 ppm
t-Bu
20a in C6D6

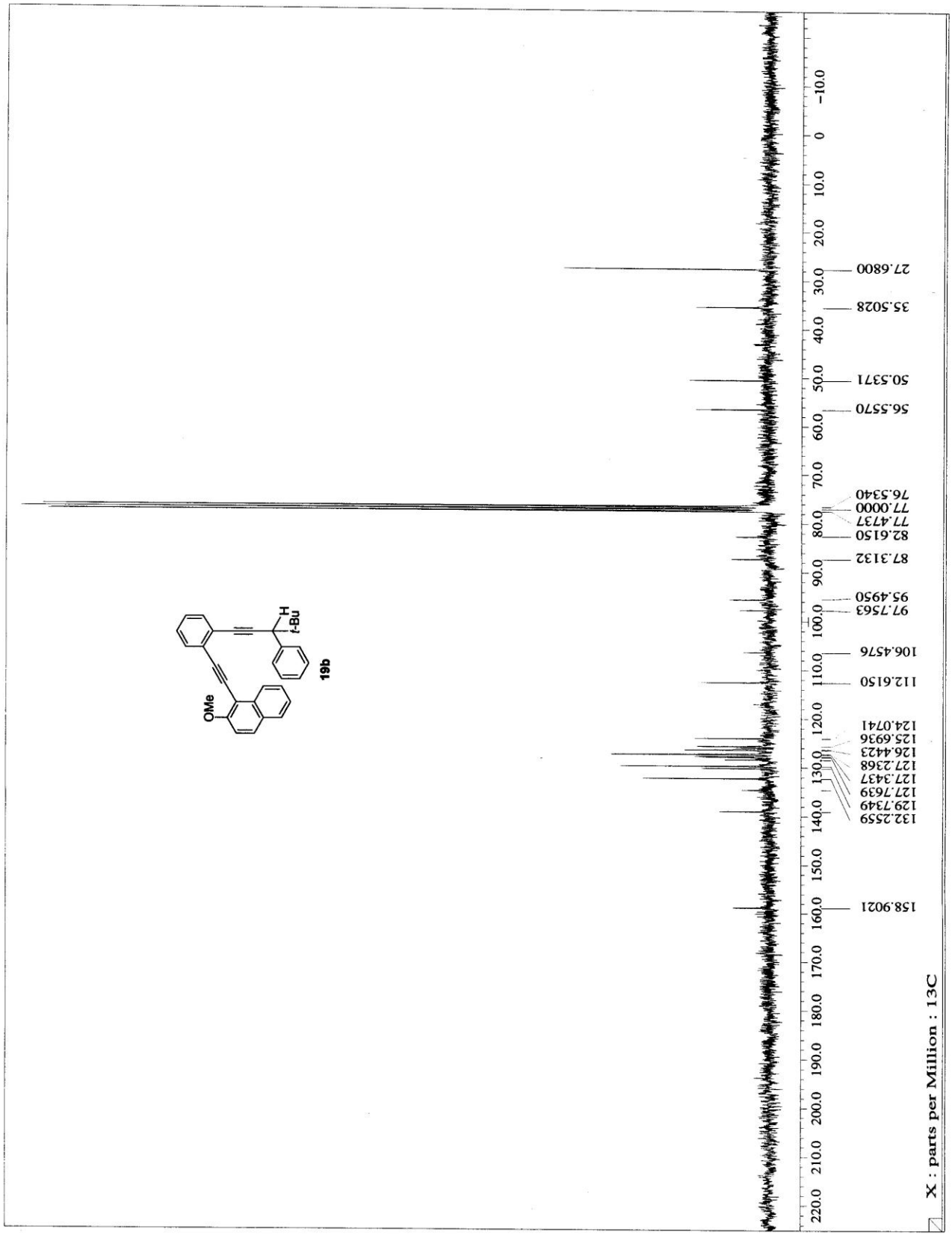

t-Bu
20a in CDCl3
1.5632
1.9636
4.5554
5.9022
5.8720
8.0589
8.0186
7.9874
7.6778
7.4863
7.4826
7.4670
7.4615
7.4386
7.3195
7.2921
7.2600
7.2188
7.1922
7.1061
8.6764
8.6443
X : parts per Million : 1H
X : parts per Million : 1H

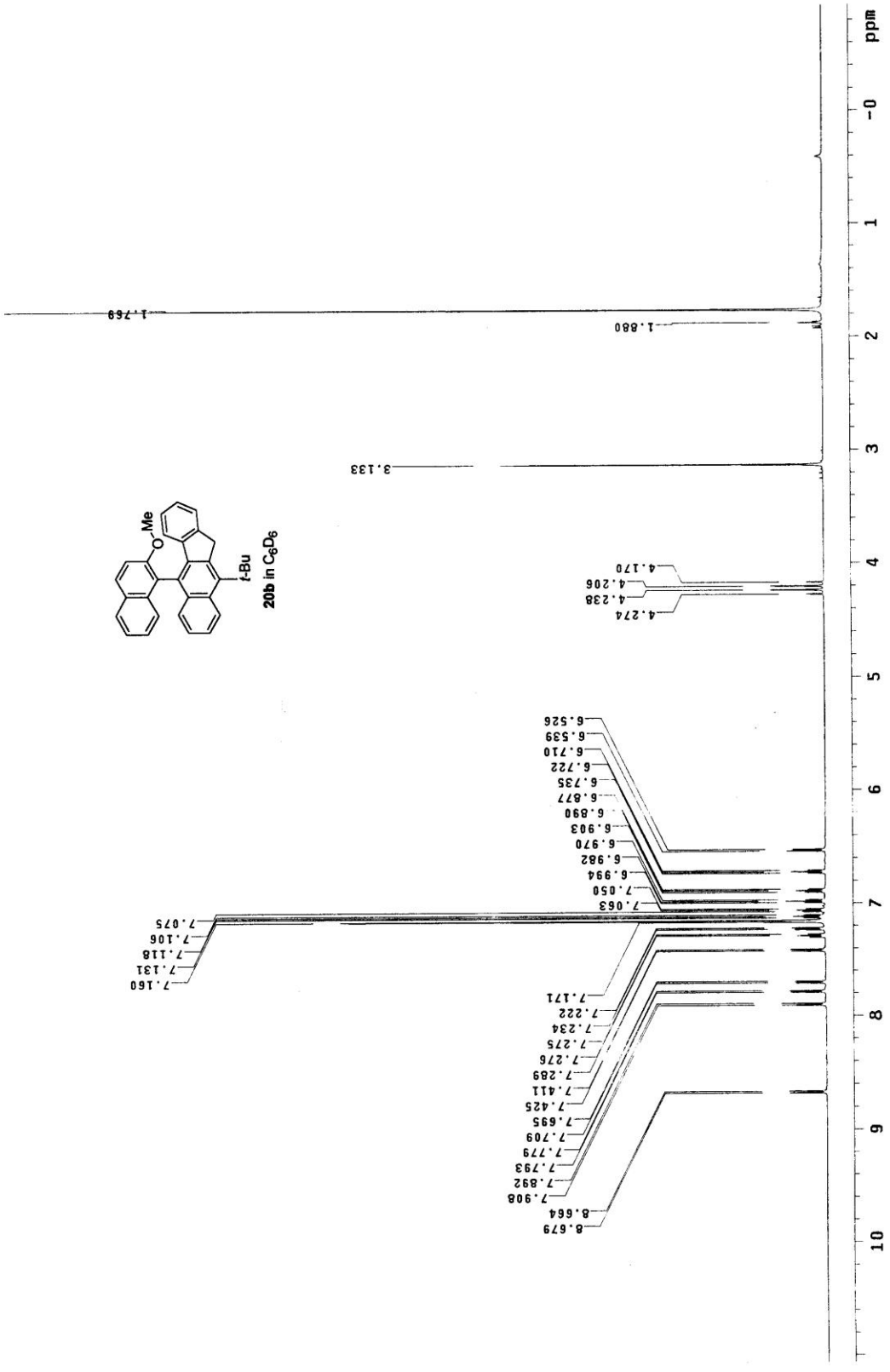

t-Bu
20a in CDCl3
77.0000
40.2454
38.9341
34.4132
30.2887
128.1561
128.0341
127.9655
127.8816
127.6605
126.2882
126.1739
124.1840
123.3835
X : parts per Million : 13C
X : parts per Million : 13C

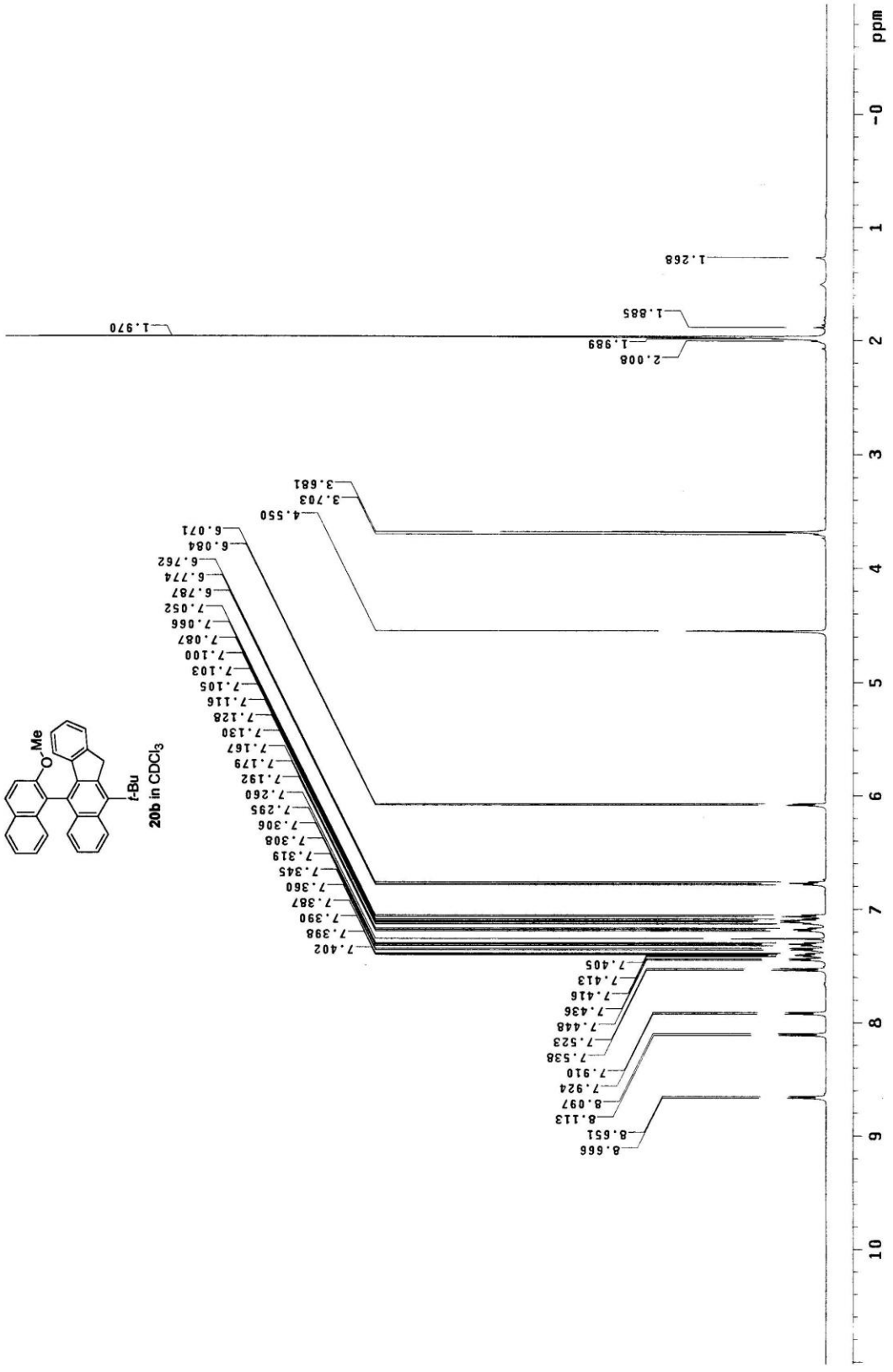

OMe
18b
1.5577
3.7638
4.0543
7.2490
7.2600
7.2829
7.3901
7.5568
7.7776
7.8436
7.8766
8.2577
X : parts per Million : 1H

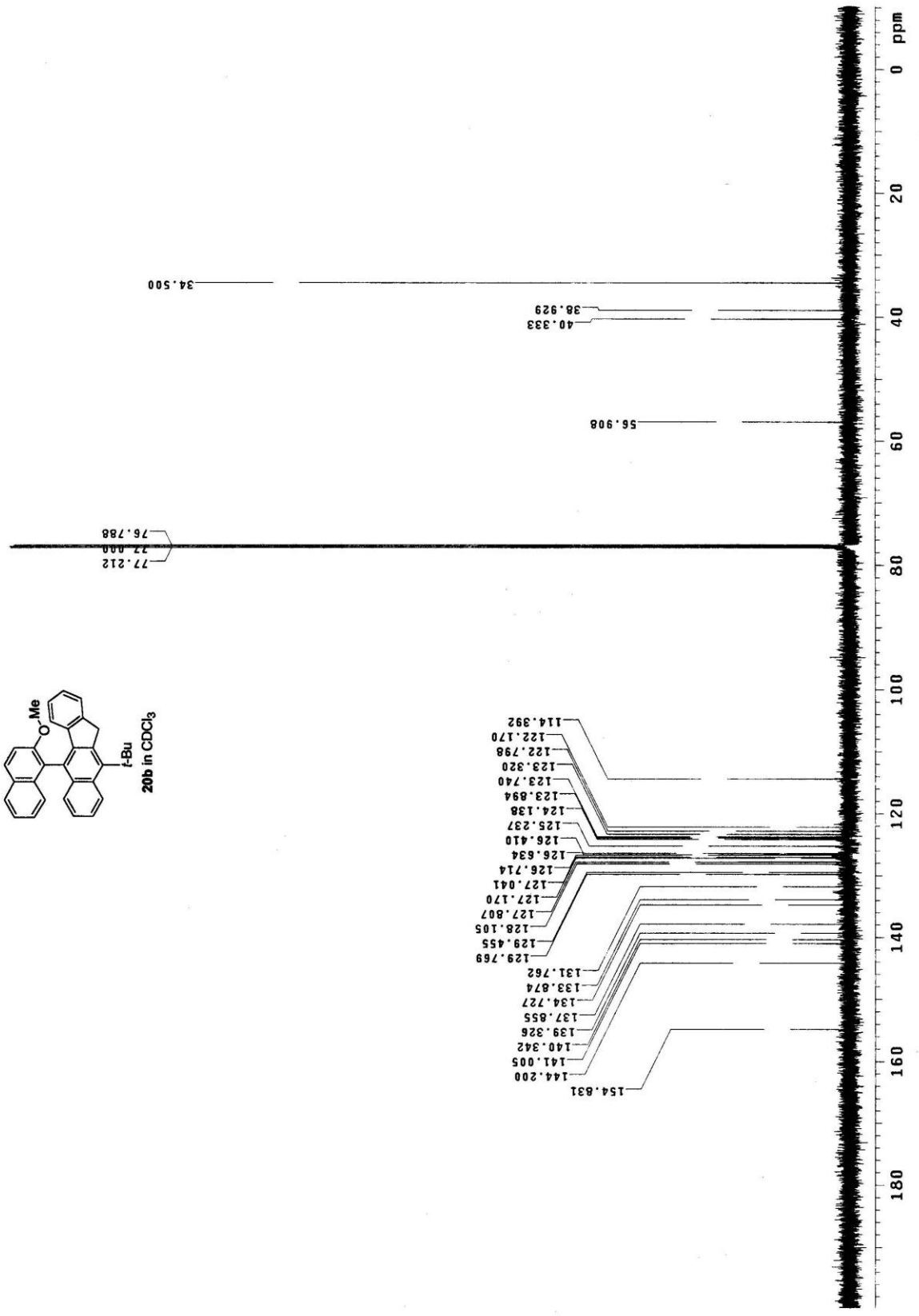

56.617
76.785
77.000
77.212
78.227
86.477
105.033
112.453
124.269
125.118
127.551
128.080
128.423
130.666
134.832
159.834
OMe
18b
0
20
40
60
80
100
120
140
160
180
ppm

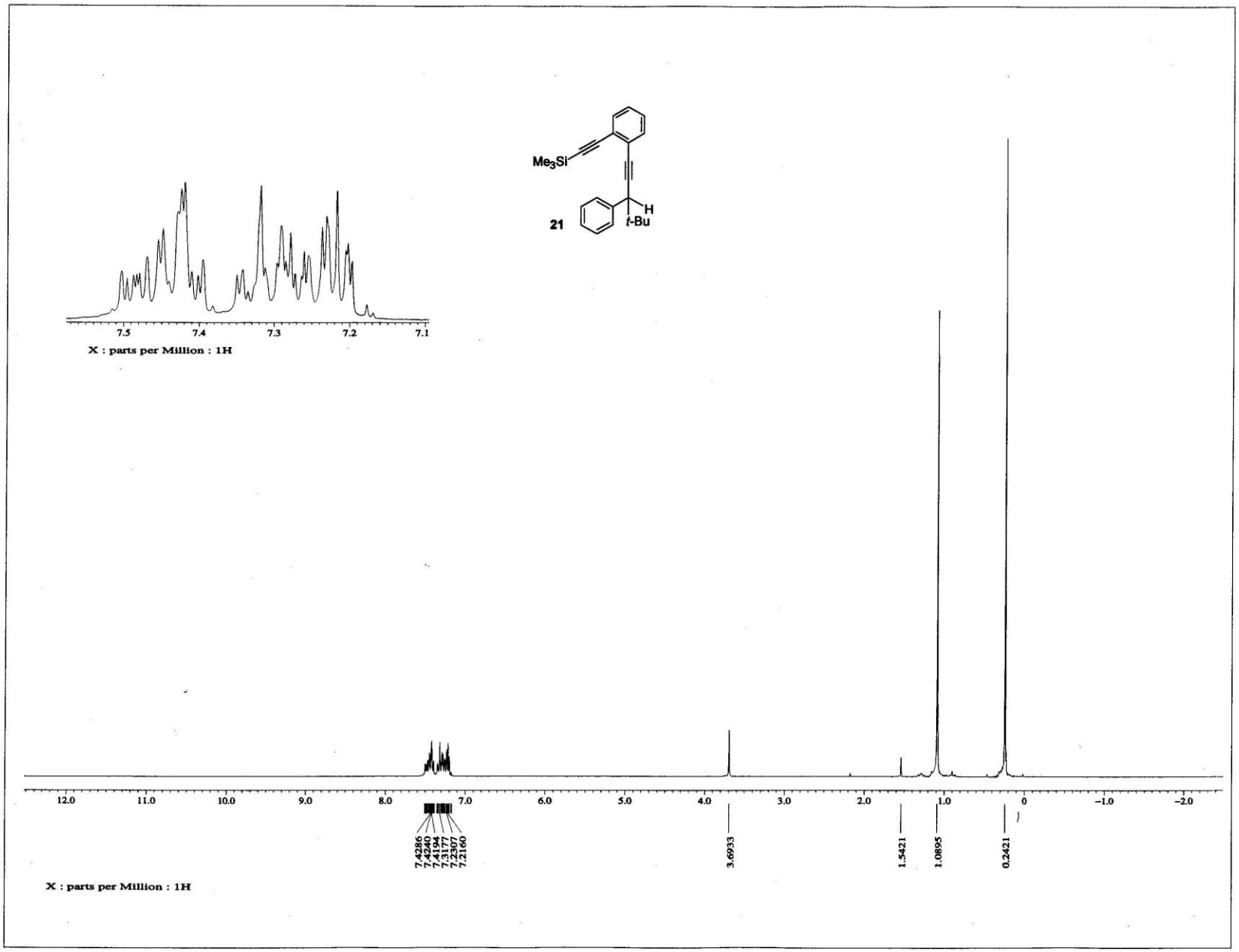

0.9475
1.2563
1.5485
3.6631
3.9957
7.0658
7.2600
7.2847
7.3415
7.3480
7.3709
7.3764
7.3864
8.3924
8.3951
8.4281
X : parts per Million : 1H
t-Bu
OMe
19b

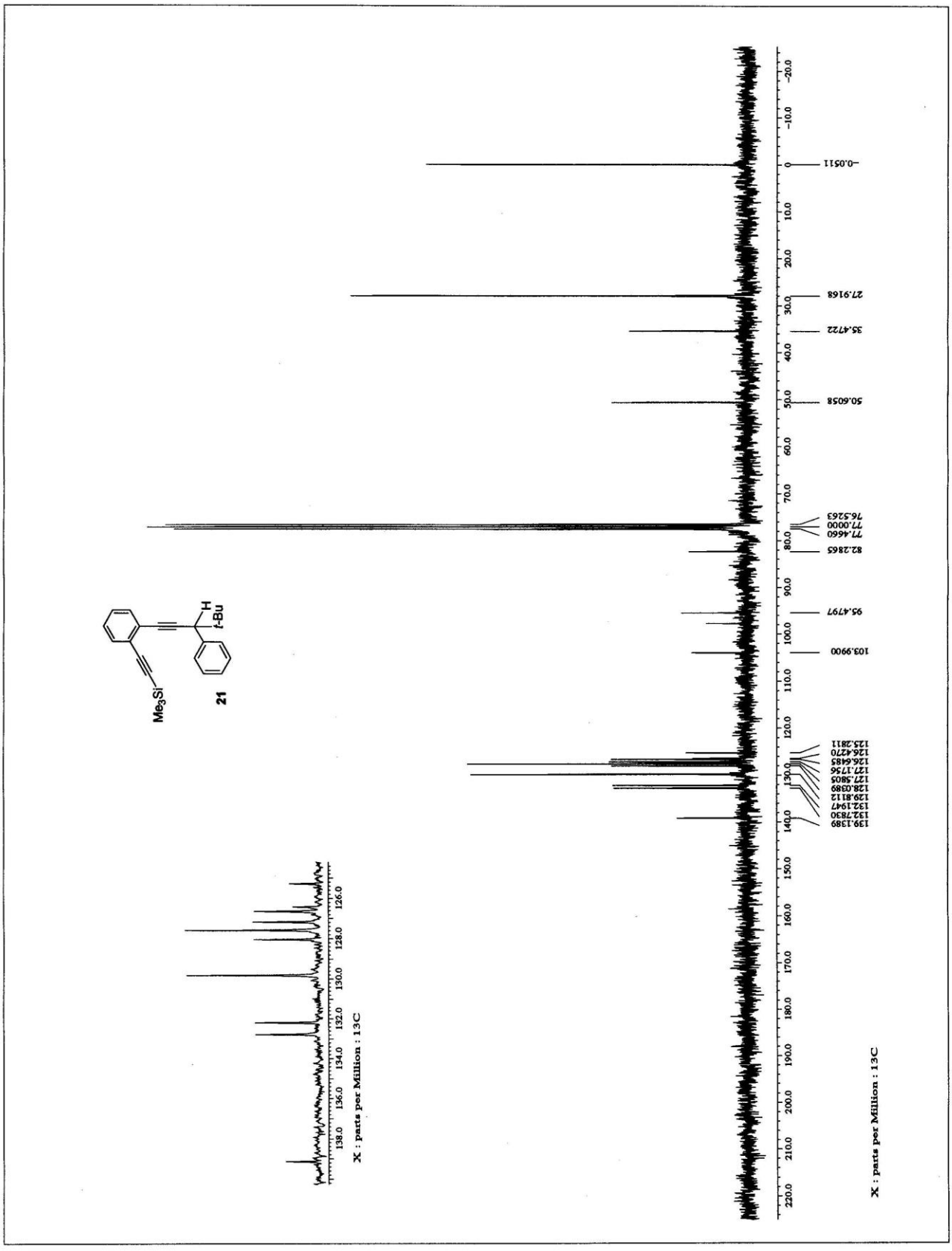

27.6800
35.5028
50.5371
56.5570
76.5340
77.0000
77.4737
82.6150
87.3132
95.4950
97.7563
106.4576
112.6150
124.0741
125.6936
126.4423
127.2368
127.3437
127.7639
129.7349
132.2559
158.9021
X : parts per Million : 13C
OMe
t-Bu
H
19b

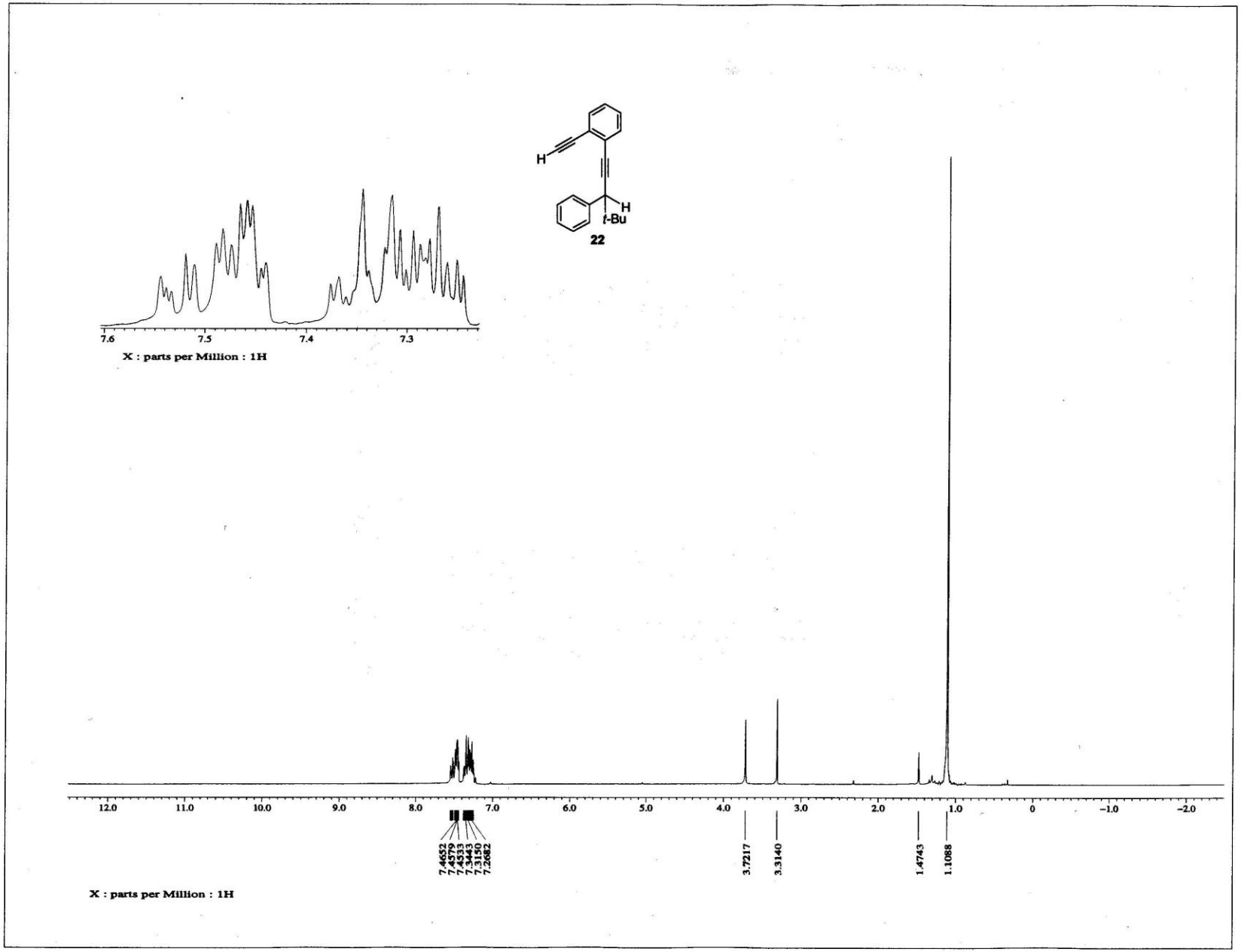

Me
O
t-Bu
20b in C6D6
1.758
1.880
3.133
4.170
4.206
4.238
4.274
6.526
6.539
6.710
6.722
6.735
6.877
6.890
6.903
6.970
6.982
6.994
7.050
7.063
7.075
7.106
7.118
7.131
7.160
7.171
7.222
7.234
7.275
7.276
7.289
7.411
7.425
7.695
7.709
7.779
7.793
7.892
7.908
8.664
8.679
ppm

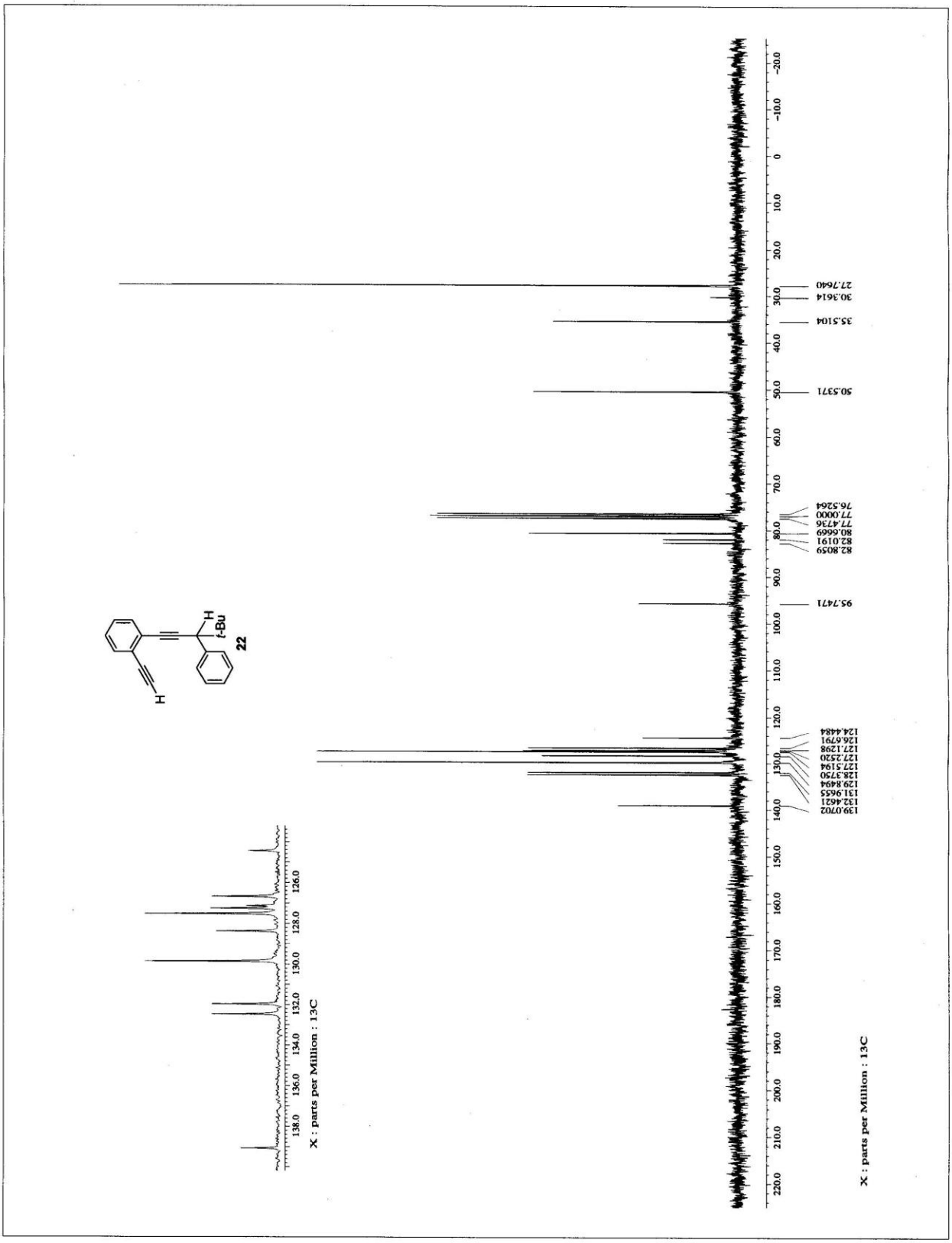

1.268
1.885
1.970
1.989
2.008
3.681
3.703
4.550
6.071
6.084
6.762
6.774
6.787
7.052
7.066
7.087
7.100
7.103
7.105
7.116
7.128
7.130
7.167
7.179
7.192
7.260
7.295
7.306
7.308
7.319
7.345
7.360
7.387
7.390
7.398
7.402
7.405
7.413
7.416
7.436
7.448
7.523
7.538
7.910
7.924
8.097
8.113
8.651
8.666
Me
O
t-Bu
20b in CDCl3
ppm

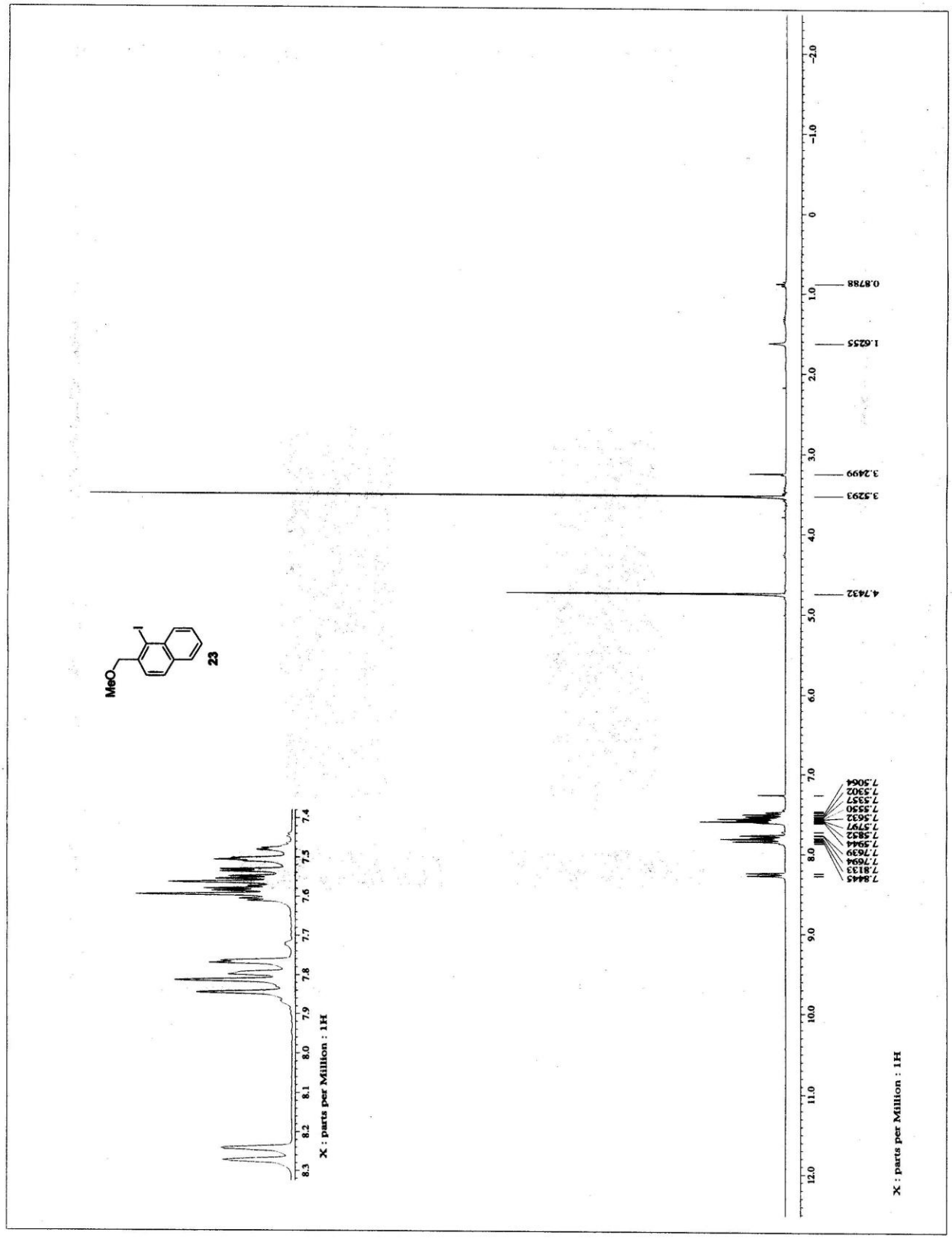

34.500
38.929
40.333
56.908
76.788
77.000
77.212
114.392
122.170
122.798
123.320
123.740
123.894
124.138
125.237
126.410
126.634
126.714
127.041
127.170
127.807
128.105
129.455
129.769
131.762
133.874
134.727
137.855
139.326
140.342
141.005
144.200
154.831
Me
O
t-Bu
20b in CDCl3
0
20
40
60
80
100
120
140
160
180
ppm

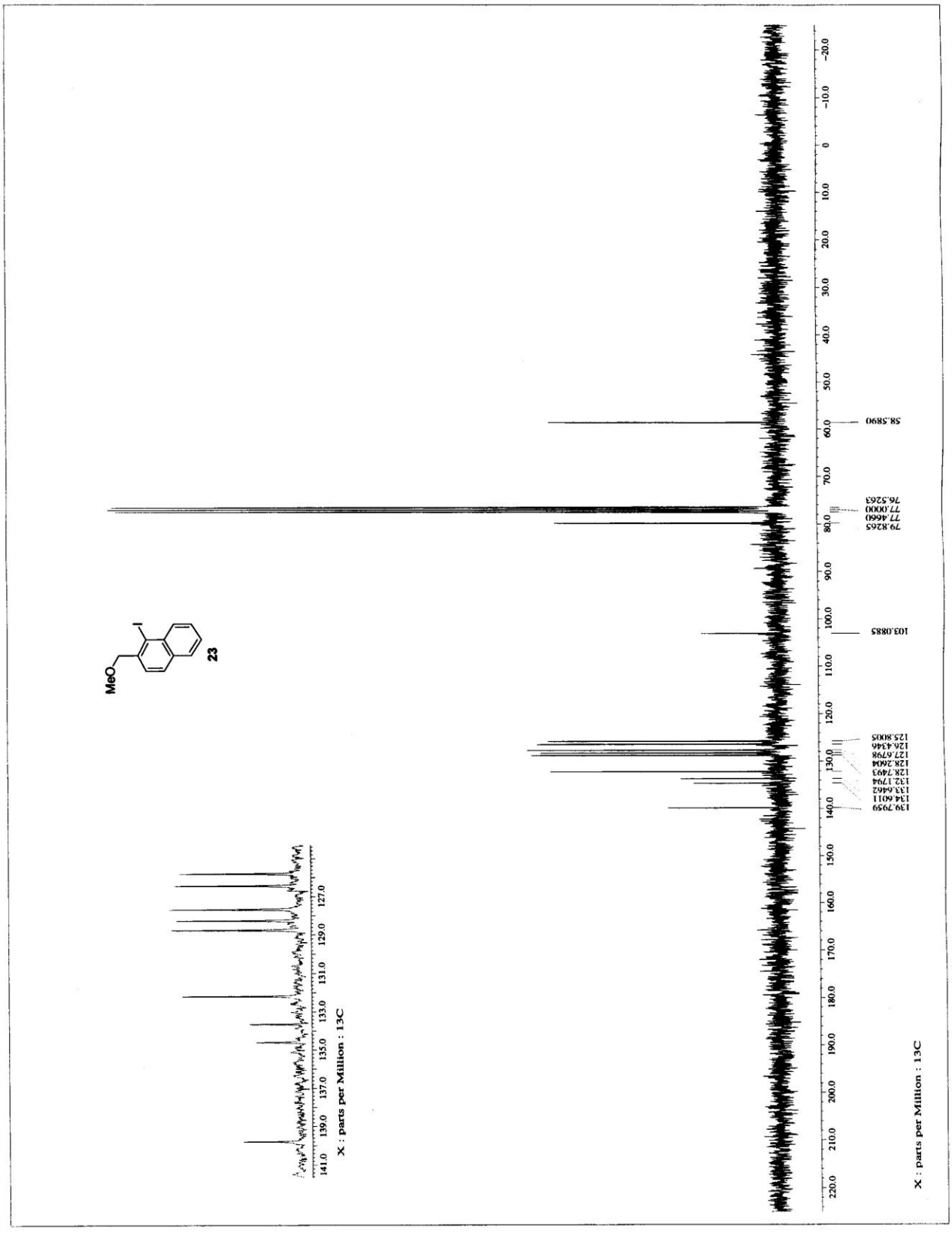

Me3Si
H
t-Bu
21
7.5
7.4
7.3
7.2
7.1
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
−2.0
7.4286
7.4240
7.4194
7.3177
7.2107
7.2160
3.6933
1.5421
1.0895
0.2421
X : parts per Million : 1H

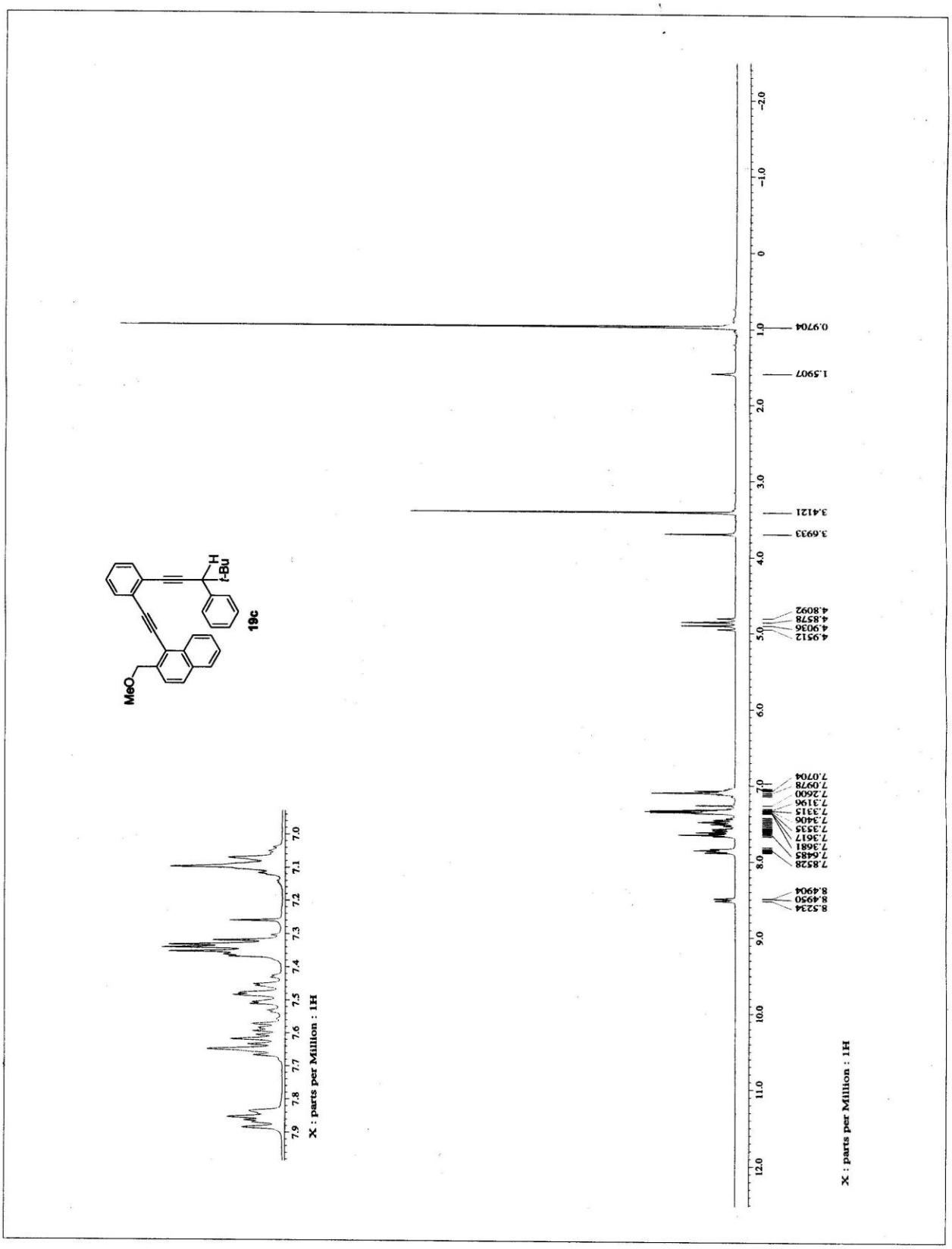

Me3Si
t-Bu
H
21
-0.0511
27.9168
35.4722
50.6058
76.5263
77.0000
77.4660
82.2865
95.4797
103.9900
125.2811
126.4270
126.6485
127.1756
127.3805
128.0589
129.8112
132.1947
132.7830
139.1389
X : parts per Million : 13C
X : parts per Million : 13C

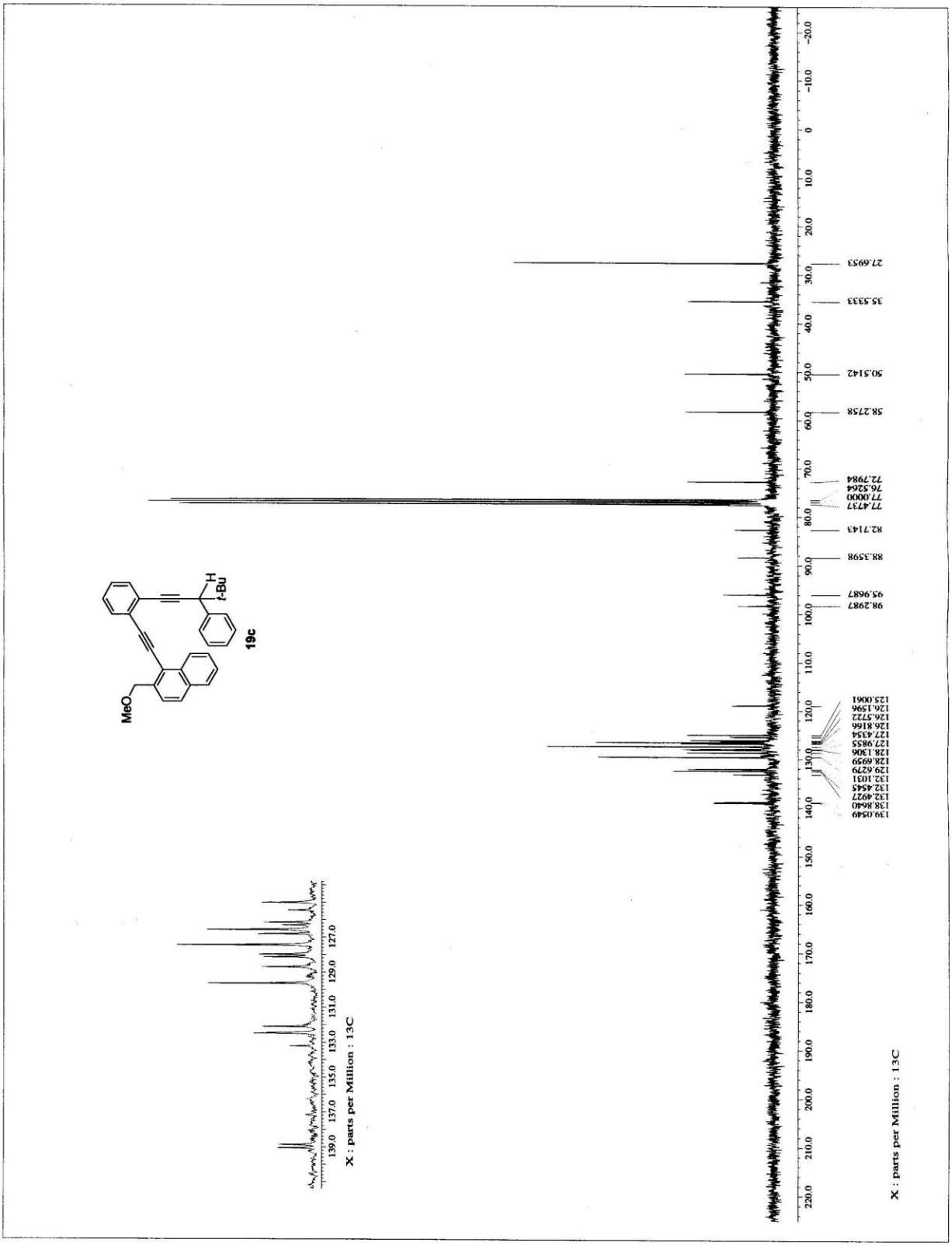

H
H
t-Bu
22
7.6
7.5
7.4
7.3
X : parts per Million : 1H
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
−2.0
7.4652
7.4579
7.4533
7.3443
7.3150
7.2682
3.7217
3.3140
1.4743
1.1088
X : parts per Million : 1H

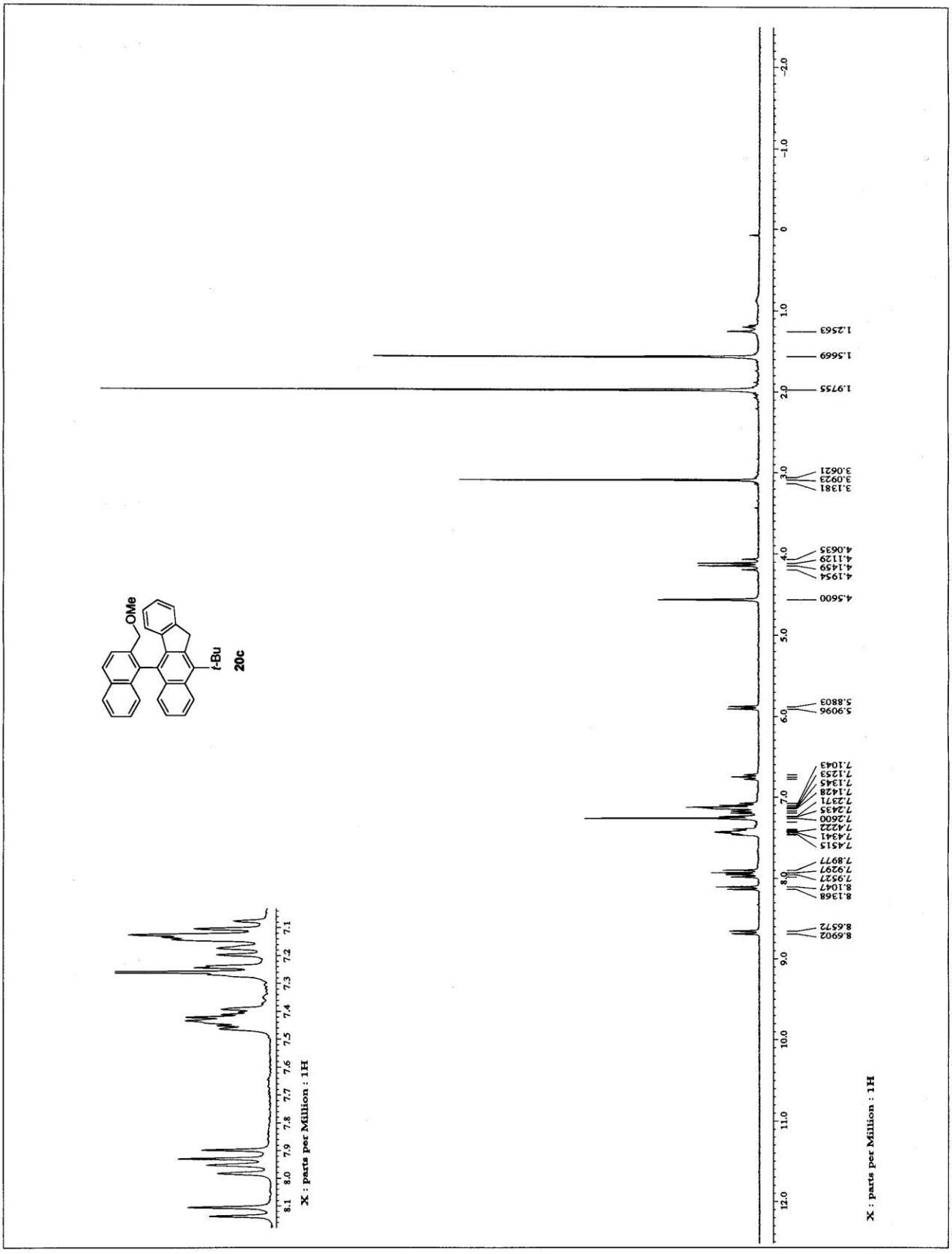

22
t-Bu
H
H
27.7640
30.3614
35.5104
50.5371
76.5264
77.0000
77.4736
80.6669
82.0191
82.8059
95.7471
124.4484
126.6791
127.1298
127.2520
127.5194
128.3750
129.8494
131.9625
132.4621
139.0702
X : parts per Million : 13C
X : parts per Million : 13C

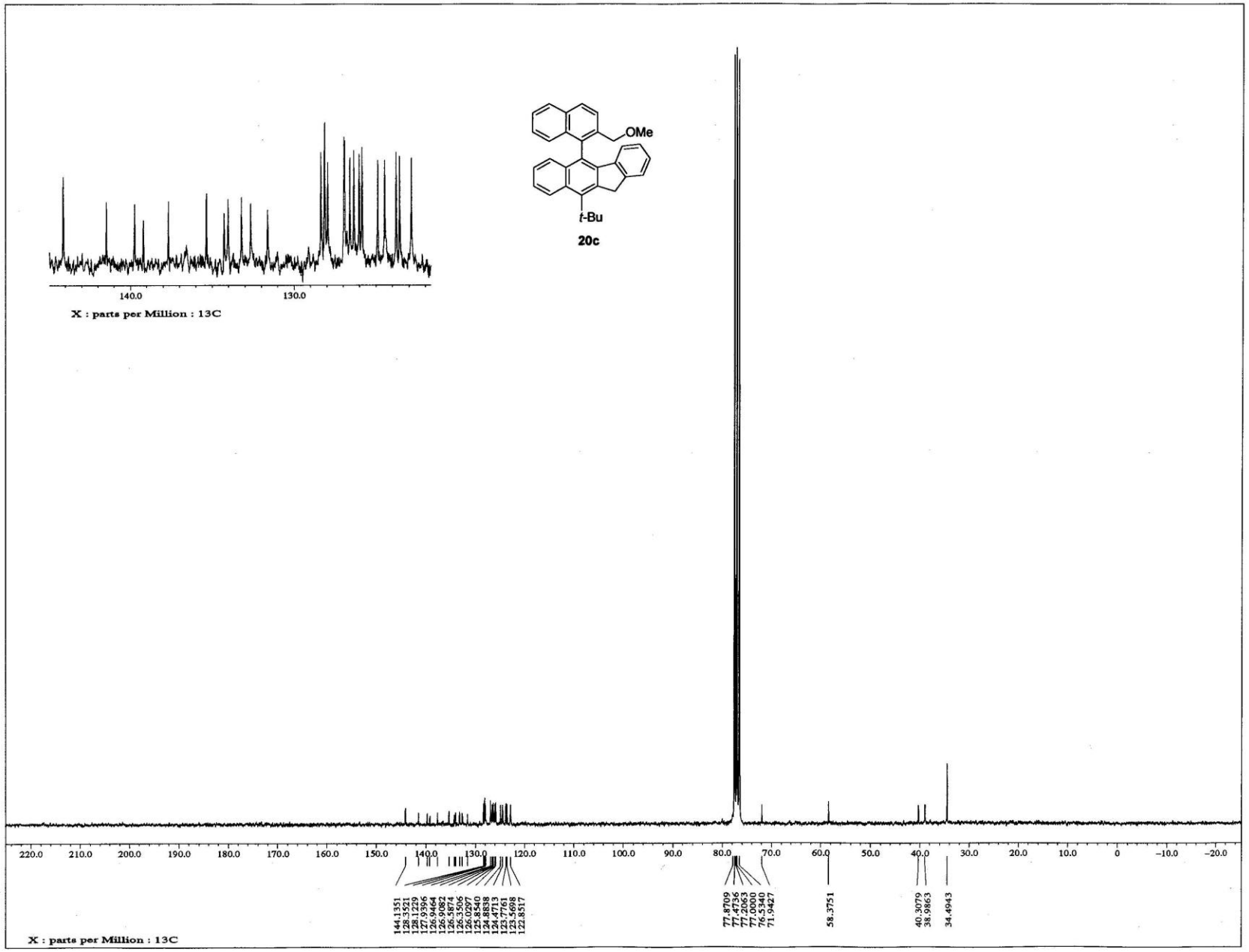

MeO
I
23
X : parts per Million : 1H
0.8788
1.6255
3.2499
3.5293
4.7432
7.8445
7.8133
7.7694
7.7639
7.5944
7.5852
7.5797
7.5632
7.5550
7.5357
7.5302
7.5064
X : parts per Million : 1H

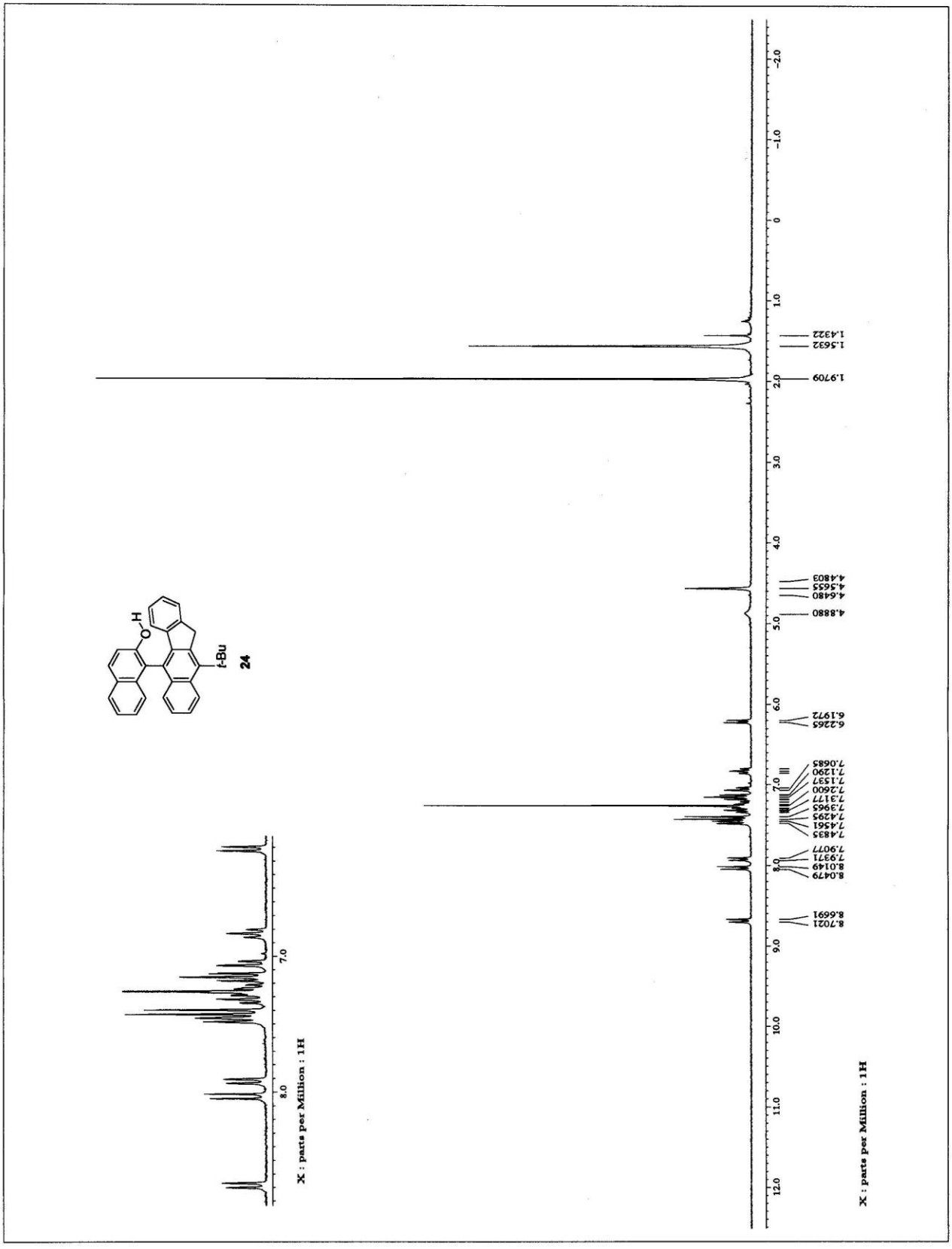

MeO
I
23
58.5890
79.8265
77.4660
77.0000
76.5263
103.0885
139.7959
134.6011
133.6462
132.1794
128.7493
128.2604
127.6798
126.4346
125.8005
X : parts per Million : 13C
X : parts per Million : 13C

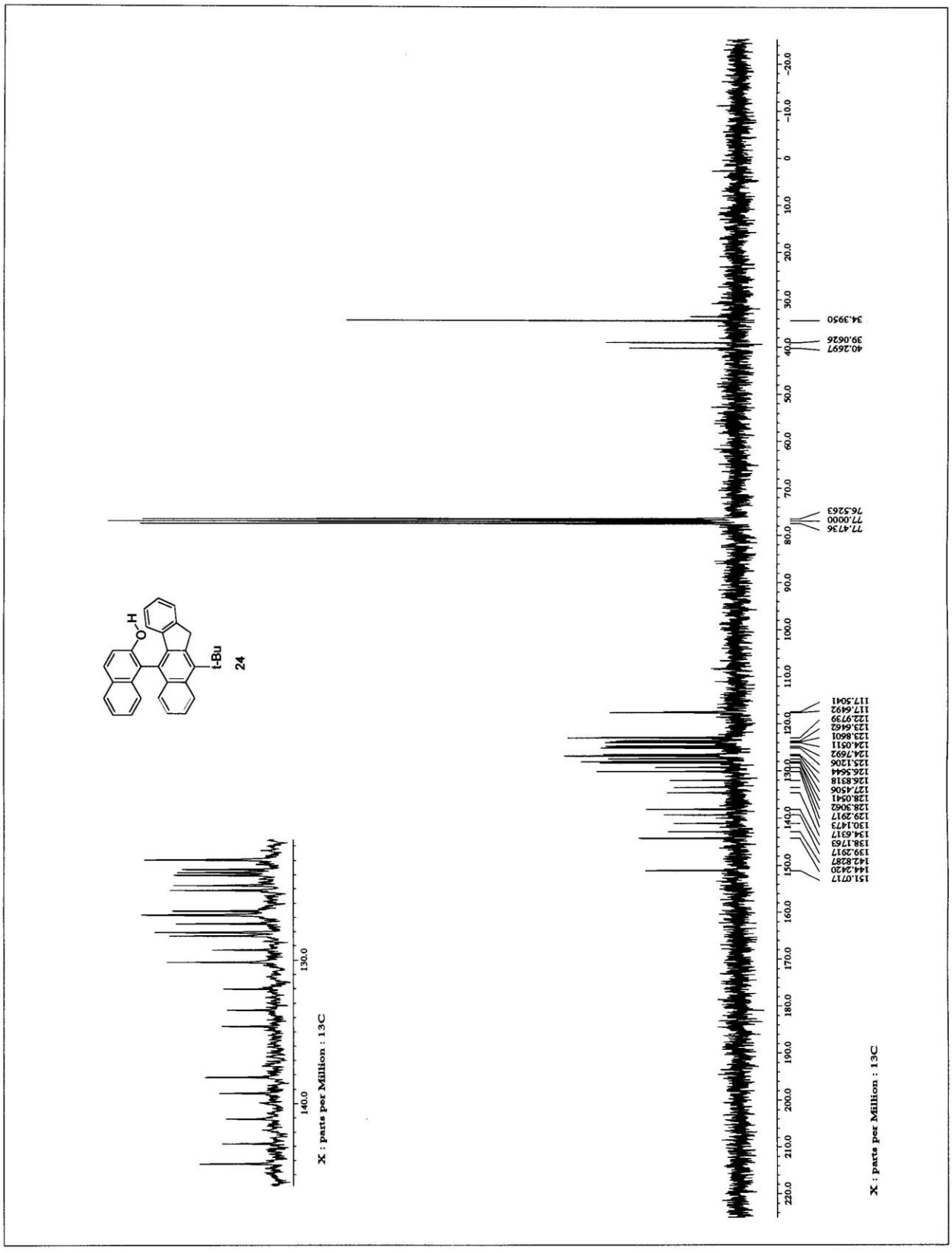

MeO
t-Bu
H
19c
0.9704
1.5907
3.4121
3.6933
4.9512
4.9036
4.8578
4.8092
7.0704
7.0978
7.2600
7.3196
7.3315
7.3406
7.3535
7.3617
7.3681
7.6485
7.8528
8.4904
8.4950
8.5234
X : parts per Million : 1H
X : parts per Million : 1H

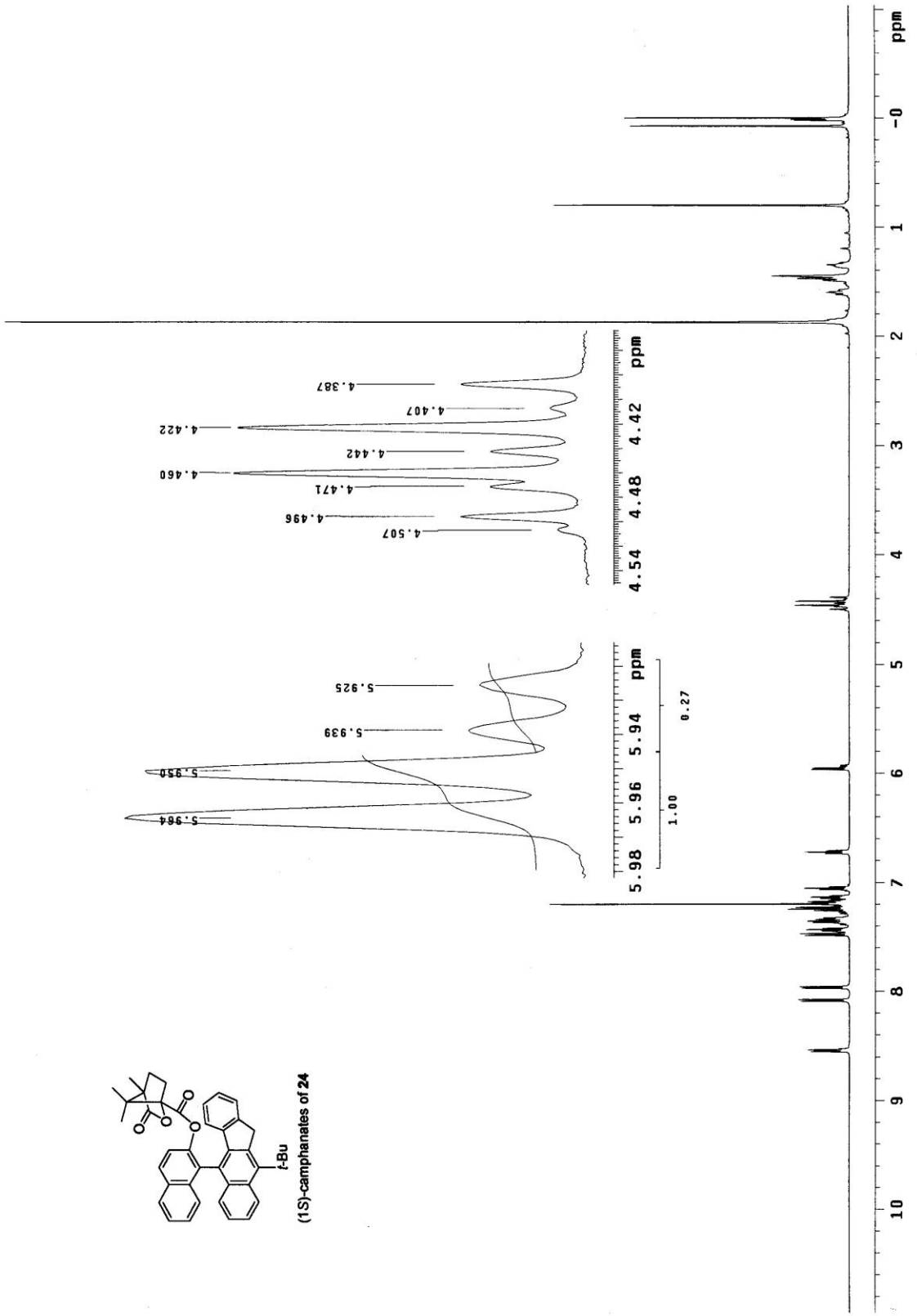

MeO
t-Bu
H
19c
X : parts per Million : 13C
27.6953
35.5333
50.5142
58.2758
72.7984
76.5264
77.0000
77.4737
82.7143
88.3598
95.9687
98.2987
125.0061
126.1596
126.5722
126.8156
127.4554
127.9855
128.1306
128.6959
129.6279
132.1031
132.4545
132.4927
138.8640
139.0549
X : parts per Million : 13C


OMe
t-Bu
20c
X : parts per Million : 1H
8.6902
8.6572
8.1368
8.1047
7.9527
7.9297
7.8977
7.4515
7.4341
7.4222
7.2600
7.2435
7.2271
7.1428
7.1345
7.1253
7.1043
5.9096
5.8803
4.5600
4.1954
4.1459
4.1129
4.0635
3.1381
3.0923
3.0621
1.9755
1.5669
1.2563
X : parts per Million : 1H


OMe
t-Bu
20c
140.0
130.0
X : parts per Million : 13C
144.1351
128.3521
128.1229
127.9396
126.9464
126.9082
126.3574
126.3365
126.0297
125.8540
124.8838
124.4713
123.7761
123.5698
122.8517
77.8709
77.4736
77.2763
77.0000
76.5340
71.9427
58.3751
40.3079
38.9863
34.4943
X : parts per Million : 13C


t-Bu
24
X : parts per Million : 1H
X : parts per Million : 1H


t-Bu
24
X : parts per Million : 13C
34.3950
39.0626
40.2697
77.4736
77.0000
76.5263
151.0717
144.2420
142.8287
139.2917
138.1763
134.6317
130.1473
129.2917
128.3062
128.0541
127.4506
126.8518
126.5644
125.1206
124.7692
124.0511
123.8601
123.6462
122.9739
117.6492
117.5041
X : parts per Million : 13C


(1S)-camphanates of 24
t-Bu
5.964
5.950
5.939
5.925
4.507
4.496
4.471
4.460
4.442
4.422
4.407
4.387
5.98
5.96
5.94
ppm
1.00
0.27
4.54
4.48
4.42
ppm
10
9
8
7
6
5
4
3
2
1
-0
ppm