Electronic Supplementary Information

$NbCl_3$-catalyzed [2+2+2] intermolecular cycloaddition of alkynes and alkenes to 1,3-cyclohexadiene derivatives

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Experimental

Genaral Remarks and Meterials

All reagents were purchased from commercial suppliers and used without further purification. ^{1}H (270 or 400 MHz) and ^{13}C NMR (67.5 or 100 MHz) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard. A GC analysis was performed with a flame ionization detector using a 0.22 mm × 25 m capillary column (BP-5). Mass spectra were determined at an ionizing voltage of 70 eV. The yields of products were estimated from the peak areas based on the internal standard technique using GLC. ^{1}H NMR, ^{13}C NMR, HMQC, HMBC and NOESY spectra were measured with a JEOL EX-270, a JEOL AL-400, or a JEOL ECA-400 spectrometers. GC analysis was carried out using a Shimadzu GC-2010 equipped with a flame ionization detector using a 0.22 mm × 25 m capillary column (BP-5). Mass spectra (EI, GCMS) were measured with an Agilent 5973 Network Mass Selective Detector. High resolution mass spectra were measured on a Micromass GCT instrument.

Compounds **3ad**,[1] **3bd**,[2] **4ad**,[3] **4bd**,[4] **5a**,[5] **5b**[6], **5c**[7] and **5d**[6] are known compounds and reported previously.

3aa: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.87 (t, ^3J(H,H)= 7 Hz, 3H), 1.05 (s, 9 H), 1.09 (s, 9H), 1.12 - 1.36 (m, 4H), 1.49 - 1.59 (m, 2H), 2.04 - 2.12 (m, 2H), 2.31 (d, ^3J(H,H)= 15 Hz, 1H) and 5.69 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.2 (CH_3), 22.8 (CH_2), 27.8 (CH_2), 28.2 (CH_2), 28.5 (3C, CH_3), 29.6 (3C, CH_3), 29.8 (CH_2), 33.2 (CH), 35.0 (C), 35.6 (C), 115.4 (CH), 115.5 (CH), 143.6 (C) and 150.3 (C); ν_{max}/cm^{-1} 2965, 2870, 1654, 1465, 1368, 1264 and 835; *m/z* (EI) 248.2504 (M^+. $C_{18}H_{32}$ requires 248.2504), 175 (7%), 119 (6), 91 (6), 57 (100).

3ab: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.79 (t, ^3J(H,H)= 7 Hz, 3H), 0.98 (s, 9 H), 1.02 (s, 9H), 1.06 - 1.28 (m, 8H), 1.42 - 1.51 (m, 2H), 1.96 - 2.04 (m, 2H), 2.25 (d, ^3J(H,H)= 15 Hz, 1H) and 5.62 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.1 (CH_3), 22.7 (CH_2), 27.6 (CH_2), 28.2 (CH_2), 28.3 (CH_2), 28.5 (3C, CH_3), 29.5 (CH_2), 29.6 (3C, CH_3), 32.0 (CH), 33.3 (CH), 35.0 (C), 35.6 (C), 115.5 (CH), 115.6 (CH), 143.6 (C) and 150.2 (C); ν_{max}/cm^{-1} 2966, 2867, 1644, 1464, 1360, 1266 and 835; *m/z* (EI) 276.2820 (M^+. $C_{20}H_{36}$ requires 276.2817), 175 (5%), 119 (3), 91 (3), 57 (100).

3ac: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.80 (t, ^3J(H,H)= 7 Hz, 3H), 0.98 (s, 9H), 1.02 (s, 9H), 1.11 - 1.26 1.42 - 1.51 (m, 14H), 1.96 - 2.04 (m, 2H), 2.25 (d, ^3J(H,H)= 15 Hz, 1H) and 5.62 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.1 (CH_3), 22.7 (CH_2), 27.6 (CH_2), 28.2 (CH_2), 28.3 (CH_2), 28.5 (3C, CH_3), 29.4 (CH_2), 29.6 (3C, CH_3), 29.7 (CH_2), 29.8 (CH_2), 32.0 (CH_2), 33.3 (CH), 35.0 (C), 35.6 (C), 115.5 (CH), 115.6 (CH), 143.5 (C) and 150.2 (C); ν_{max}/cm^{-1} 2925, 2855, 1464, 1364, 1261 and 803; *m/z* (EI) 304.3122 (M^+. $C_{22}H_{40}$ requires 304.3130), 175 (8%), 119 (4), 91 (3), 57 (100).

3ad: δ_H (270 MHz; $CDCl_3$, Me_4Si) 1.00 (s, 18 H), 2.04 (s, 4H) and 5.65 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 24.6 (2C, CH_2), 28.7 (6C, CH_3), 35.2 (2C, C), 115.6 (2C, CH), 146.2 (2C, C).

3ae: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.72 (t, ^3J(H,H)= 7 Hz, 3H), 0.85 (s, 3H), 0.86 (s, 3 H), 0.99 (s, 9H), 1.04 (s, 9H), 1.11 – 1.24 (m, 4H), 1.69 (dd, ^3J(H,H)= 15, 10 Hz, 1H), 2.04 – 2.10 (m, 1H), 2.30 – 2.37 (m, 1H) and 5.64 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 8.4 (CH_3), 26.9 (CH_3), 27.1 (CH_3), 28.8 (3C, CH_3), 28.9 (CH), 30.0 (3C, CH_3), 31.3 (CH_2), 34.0 (C), 35.1 (C), 35.8 (C), 36.9 (CH_2), 38.0 (CH_2), 116.2 (CH), 116.3 (CH), 143.9 (C) and 150.5 (C); ν_{max}/cm^{-1} 2967, 2870, 1740, 1464, 1364, 1254 and 834; *m/z* (EI) 276.2814 (M^+. $C_{20}H_{36}$ requires 276.2817), 175 (3%), 119 (3), 91 (3), 57 (100).

3af: δ_H (270 MHz; $CDCl_3$, Me_4Si) 1.02 (s, 9H), 1.20 (s, 9H), 1.99 – 2.17 (m, 2H), 2.59 – 2.67 (m, 3H), 5.77 – 5.85 (m, 2H) and 7.15 – 7.30 (m, Ph); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 28.0 (CH_2), 28.9 (3C, CH_3), 29.8 (3C, CH_3), 34.66 (CH_2), 34.68 (CH), 35.2 (C), 35.9 (C), 115.9 (CH), 116.5 (CH), 125.8 128.2 129.0 141.2 (6C, Ph), 143.6 (C) and 148.4 (C); ν_{max}/cm^{-1} 2963, 2866, 1453, 1359, 1269, 837, 734 and 696; *m/z* (EI) 282.2341 (M^+. $C_{21}H_{30}$ requires 282.2348), 190 (14%), 175 (12), 91 (23), 57 (100).

3ag: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.66 (s, 9H), 0.92 (s, 9H), 2.22 (dd, ^3J(H,H)= 16, 2 Hz, 1H), 2.49 (ddd, ^3J(H,H)= 16, 8, 3 Hz, 1H), 3.48 (dd, ^3J(H,H)= 8, 2 Hz, 1H), 5.68 (dd, ^3J(H,H)= 6, 3 Hz, 1H), 6.03 (d, ^3J(H,H)= 6 Hz, 1H) and 7.00 – 7.14 (m, Ph); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 27.8 (3C, CH_3), 29.7 (3C, CH_3), 34.6 (C), 34.8 (CH_2), 35.7 (C), 39.3 (CH), 116.0 (CH), 118.7 (CH), 125.7 127.6 128.0 142.8 (6C, Ph), 142.5 (C) and 146.6 (C); ν_{max}/cm^{-1} 3058, 2965, 2905, 1463, 1361, 1258, 840, 760 and 700; *m/z* (EI) 268.2196 (M^+. $C_{20}H_{28}$ requires 268.2191), 211 (18%), 155 (20), 91 (18), 57 (100).

3ah: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.67 (s, 9H), 0.92 (s, 9H), 2.18 (s, 3H), 2.24 (d, ^3J(H,H)= 6 Hz, 1H), 2.47 (ddd, ^3J(H,H)= 16, 8, 3 Hz, 1H), 3.44 (d, ^3J(H,H)= 7 Hz, 1H), 5.67 (dd, ^3J(H,H)= 6, 3 Hz, 1H), 6.01 (d, ^3J(H,H)= 6 Hz, 1H) and 6.88 - 7.55 (m, Ph); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 21.0 (CH_3), 27.8 (3C, CH_3), 29.7 (3C, CH_3), 34.6 (C), 34.8 (CH_2), 35.7 (C), 38.8 (CH), 115.9 (CH), 118.5 (CH), 127.8 128.3 134.9 139.4 (6C, Ph), 142.8 (C) and 146.7 (C); ν_{max}/cm^{-1} 2962, 2903, 1509, 1462, 1361, 1259 and 812; *m/z* (EI) 282.2349 (M^+. $C_{21}H_{30}$ requires 282.2348), 225 (24%), 169 (30), 105 (23), 57 (100).

3ba: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.74 (t, ^3J(H,H)= 7 Hz, 3H), 1.14 - 1.49 (m, 6H), 2.77 - 2.85 (m, 3H), 6.41 (s, 2H) and 7.15 - 7.46 (m, 2Ph); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.0 (CH_3), 22.8 (CH_2), 29.5 (CH_2), 30.1 (CH_2), 30.2 (CH_2), 35.0 (CH), 120.5 (CH), 121.0 (CH), 125.1 125.5 127.0 127.04 128.45 128.48 (10C, 2Ph), 134.3 (2C, C), 139.9 (C) and 141.2 (C); ν_{max}/cm^{-1} 3031, 2954, 2925, 1495, 1446, 758 and 688; *m/z* (EI) 288.1876 (M^+. $C_{21}H_{30}$ requires 288.1878), 231 (100%), 216 (31), 152 (11), 96 (20).

3bd: δ_H (270 MHz; $CDCl_3$, Me_4Si) 2.72 (s, 4H), 6.46 (s, 2H) and 7.18 - 7.45 (m, 2Ph); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 26.1 (2C, CH_2), 121.6 (2C, CH), 124.9 127.1 128.4 135.9 (12C, 2Ph) and 140.7 (2C, C).

3ca: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.81 (t, ^3J(H,H)= 7 Hz, 3H), 0.93 - 1.48 1.64 - 1.89 (m, 28H), 2.00 - 2.15 (m, 1H), 2.34 - 2.40 (m, 1H), 2.66 - 2.68 (m, 1H) and 5.46 - 5.49 (m, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.1 (CH_3), 22.9 (CH_2), 26.3 (CH_2), 26.39 (CH_2), 26.44 (CH_2), 26.5 (CH_2), 26.7 (CH_2), 26.8 (CH_2), 27.0 (CH_2), 27.2 (CH_2), 27.3 (CH_2), 29.1 (CH_2), 29.6 (CH_2), 30.1 (CH_2), 31.4 (CH_2), 31.7 (CH_2), 34.5 (CH_2), 34.6 (CH_2), 34.68 (CH_2), 34.71 (CH_2), 34.74 (CH_2), 35.8 (CH), 39.0 (CH), 39.3 (CH), 43.3 (CH), 44.3 (CH), 44.8 (CH), 45.1 (CH), 115.7 (CH), 116.5 (CH), 122.9 (CH), 123.8

(CH), 124.4 (CH), 125.6 (CH), 140.1 (C), 142.0 (C), 145.0 (C) and 147.7 (C); ν_{max}/cm^{-1} 2909, 2849, 1600, 1448, 1348, 1261, 997, 908, 855, 820 and 734; *m/z* (EI) 300.2828 (M^+. $C_{22}H_{36}$ requires 300.2817), 161 (25%), 91 (10), 83 (100), 55 (31).

3da: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.81 (t, ^3J(H,H)= 7 Hz, 9H), 1.05 - 1.56 (m, 22H), 1.84 - 2.00 (m, 5H), 2.14 - 2.20 (dd, ^3J(H,H)= 17, 8 Hz, 1H), 2.45 - 2.51 (m, 1H) and 5.48 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.1 (CH_3), 22.6 (CH_2), 22.7 (CH_2), 22.9 (CH_2), 27.7 (CH_2), 28.6 (CH_2), 28.9 (CH_2), 29.07 (CH_2), 29.14 (CH_2), 29.2 (CH_2), 29.43 (CH_2), 29.46 (CH_2), 29.52 (CH_2), 29.7 (CH_2), 31.4 (CH_2), 31.6 (CH_2), 31.77 (CH_2), 31.84 (CH_2), 32.4 (CH_2), 32.6 (CH_2), 32.8 (CH_2), 35.2 (CH_2), 35.6 (CH_2), 36.0 (CH_2), 36.5 (CH), 37.3 (CH_2), 118.0 (CH), 118.4 (CH), 125.7 (CH), 125.8 (CH), 128.9 (CH), 129.2 (CH), 130.4 (CH), 132.4 (CH), 135.3 (C), 137.7 (C), 140.1 (C), 140.3 (C), 142.1 (C) and 142.7 (C); ν_{max}/cm^{-1} 2927, 2856, 1716, 1598, 1466, 1378 and 722; *m/z* (EI) 304.3137 (M^+. $C_{22}H_{40}$ requires 304.3130), 247 (36%), 85 (83), 57 (31), 43 (100).

3dd: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.81 (t, ^3J(H,H)= 7 Hz, 6H), 1.20 - 1.36 (m, 16H), 1.97 (t, ^3J(H,H)= 8 Hz, 4H), 2.01 (s, 4H) and 5.52 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.1 (2C, CH_3), 22.7 (2C, CH_2), 27.3 (2C, CH_2), 27.7 (2C, CH_2), 29.1 (2C, CH_2), 31.8 (2C, CH_2), 37.1 (2C, CH_2), 118.7 (2C, CH) and 137.2 (2C, C); ν_{max}/cm^{-1} 2958, 2855, 1599, 1466, 1378 and 724; *m/z* (EI) 248.2509 (M^+. $C_{18}H_{32}$ requires 248.2504), 177 (61%), 93 (100), 79 (32), 43 (36).

4aa: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.78 - 0.85 (m, 3H), 0.97 (s, 9H), 1.00 (s, 9H), 1.19 - 1.46 (m, 6H), 1.67 - 1.76 (m, 1H), 1.97 - 2.09 (m, 2H), 5.24 - 5.25 (m, 1H) and 5.60 - 5.73 (m, 1H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 14.2 (CH_3), 23.0 (CH_2), 28.7 (3C, CH_3), 29.2 (3C, CH_3), 29.4 (CH_2), 29.8 (CH_2), 34.0 (CH_3), 34.1 (CH_2), 34.6 (CH), 35.5 (C), 115.4 (CH), 119.8 (CH), 144.2 (C) and 147.1 (C); *m/z* (EI) 248.2507 (M^+. $C_{18}H_{32}$ requires 248.2504), 175 (3%), 119 (2), 91 (3), 57 (100).

4ab: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.76 – 0.87 (m, 3H), 0.99 (s, 9H), 1.00 (s, 9H), 1.08 – 1.51 (m, 10H), 1.59 – 1.75 (m, 1H), 1.96 – 2.08 (m, 2H), 5.22 – 5.32 (m, 1H) and 5.71 – 5.82 (m, 1H); *m/z* (EI) 276.2819 (M^+. $C_{20}H_{36}$ requires 276.2817), 175 (2%), 119 (2), 91 (2), 57 (100).

4ac: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.76 – 0.85 (m, 3H), 0.99 (s, 9H), 1.00 (s, 9H), 1.06 – 1.26 1.42 – 1.51 (m, 14H), 1.61 – 1.75 (m, 1H), 1.96 – 2.11 (m, 2H), 5.19 – 5.32 (m, 1H) and 5.60 – 5.73 (m, 1H); *m/z* (EI) 304.3137 (M^+. $C_{22}H_{40}$ requires 304.3130), 175 (2%), 119 (2), 91 (2), 57 (100).

4ae: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.76 – 0.79 (m, 3H), 0.93 (s, 3H), 0.96 (s, 3H), 0.97 (s, 9H), 1.00 (s, 9H), 1.34 – 1.48 (m, 4H), 1.52 – 1.58 (m, 1H), 2.04 – 2.15 (m, 2H) and 5.26-5.74 (m, 2H); *m/z* (EI) 276.2820 (M^+. $C_{20}H_{36}$ requires 276.2817), 175 (4%), 119 (2), 91 (3), 57 (100).

4af: δ_H (270 MHz; $CDCl_3$, Me_4Si) 1.04 (s, 9H), 1.06 (s, 9H), 1.86 – 1.93 (m, 2H), 2.53 – 2.54 (m, 3H), 5.09 – 5.28 (m, 2H) and 7.04 – 7.30 (m, 5H); *m/z* (EI) 282.2354 (M^+. $C_{21}H_{30}$ requires 282.2348), 190 (9%), 175 (2), 91 (17), 57 (100).

4ag: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.94 (s, 9H), 1.02 (s, 9H), 2.05 – 2.13 (m, 1H), 2.28 – 2.34 (m, 1H), 3.35 – 3.42 (m, 1H), 5.39 – 5.40 (m, 1H), 5.85 (s, 1H) and 7.00 – 7.31 (m, 5H); *m/z* (EI) 268.2201 (M^+. $C_{20}H_{28}$ requires 268.2191), 211 (12%), 155 (11), 91 (10), 57 (100).

4ah: δ_H (270 MHz; $CDCl_3$, Me_4Si) 0.95 (s, 9H), 1.02 (s, 9H), 2.03 – 2.31 (m, 5H), 3.32 – 3.39 (m, 1H), 5.36 – 5.38 (m, 1H), 5.83 (s, 1H) and 6.88 – 7.55 (m, 4H); *m/z* (EI) 282.2353 (M^+. $C_{21}H_{30}$ requires 282.2348), 225 (12%), 169 (18), 105 (12), 57 (100).

7: δ_H (270 MHz; $CDCl_3$, Me_4Si) 1.08 (s, 18 H), 1.35 – 1.39 1.52 – 1.57 1.69 – 1.72 (m, 6H), 2.36 – 2.37 (m, 2H), 2.59 (d, ^3J(H,H)= 0.7 Hz, 2H) and 5.57 (s, 2H); δ_C (67.5 MHz; $CDCl_3$, Me_4Si) 30.4 (2C, CH_2), 31.2 (6C, CH_3), 33.6 (CH_2), 35.1 (2C, C), 45.0 (2C, CH), 47.3 (2C, CH), 117.6 (2C, CH) and 143.3 (2C, C); ν_{max}/cm^{-1} 2956, 2907, 2868, 1466, 1360, 1244 and 843; *m/z* (EI) 258.2354 (M^+. $C_{19}H_{30}$ requires 258.2348), 243 (17%), 187 (15), 175 (100), 57 (65).

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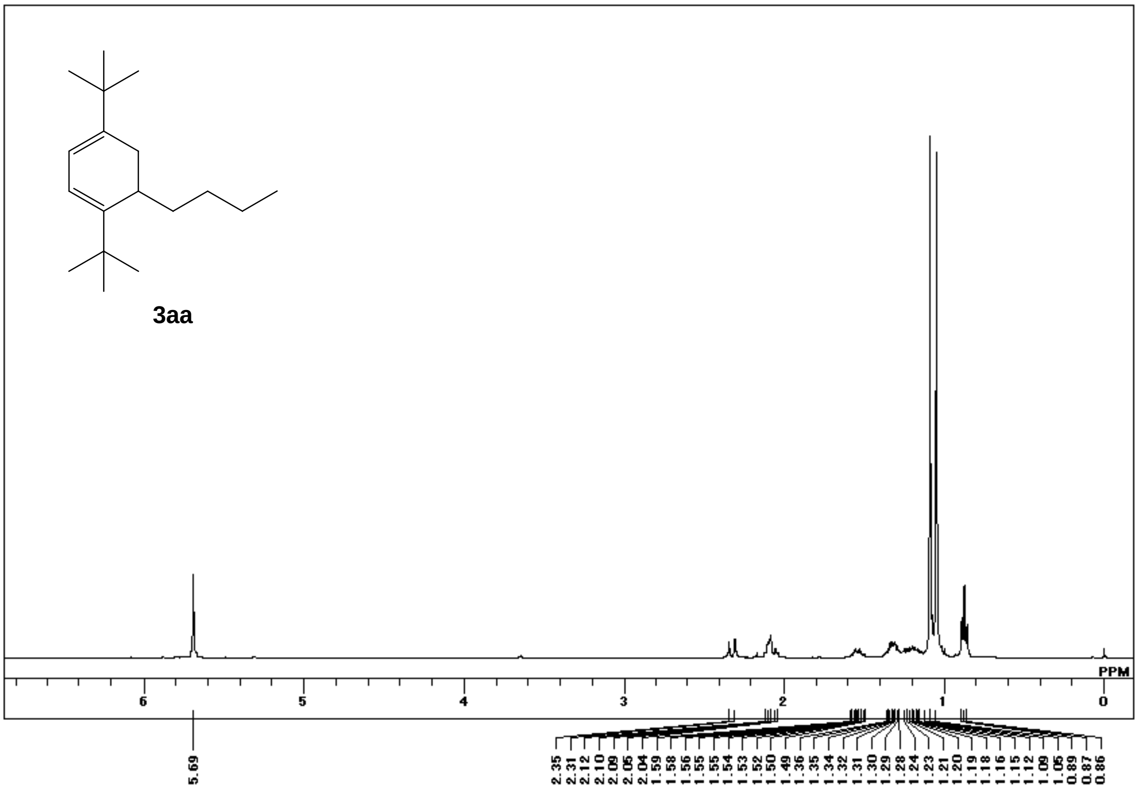
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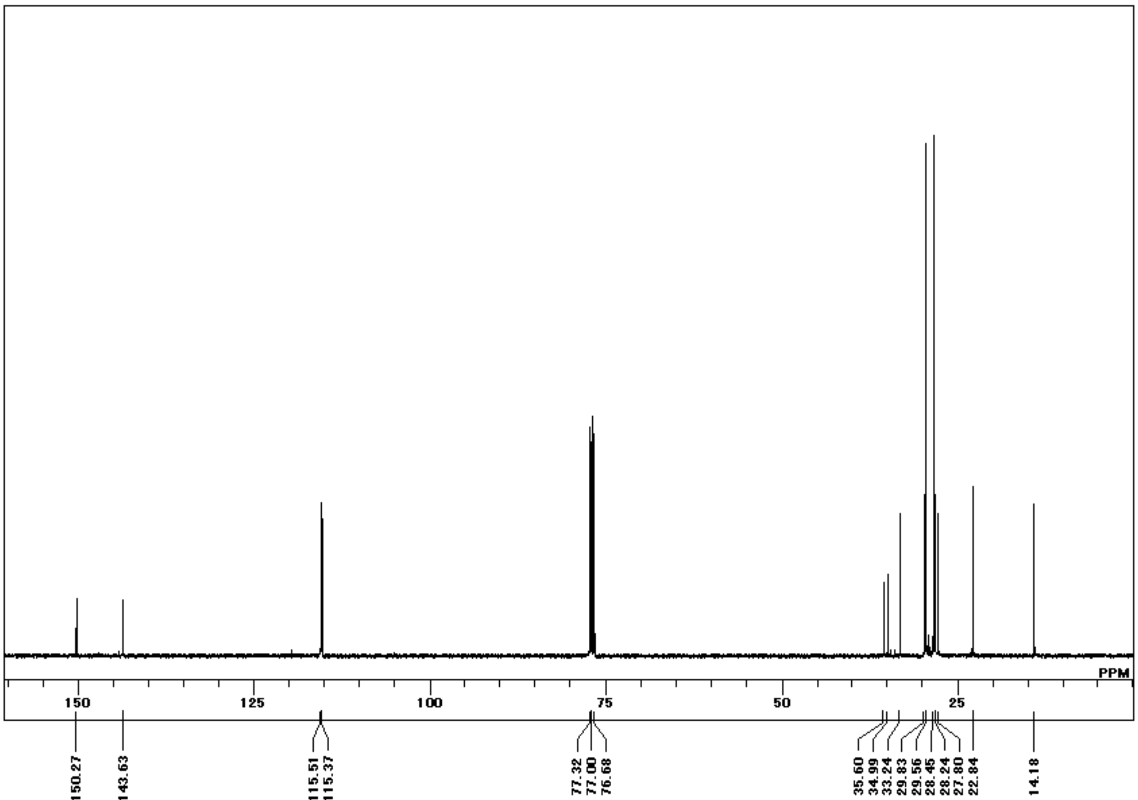
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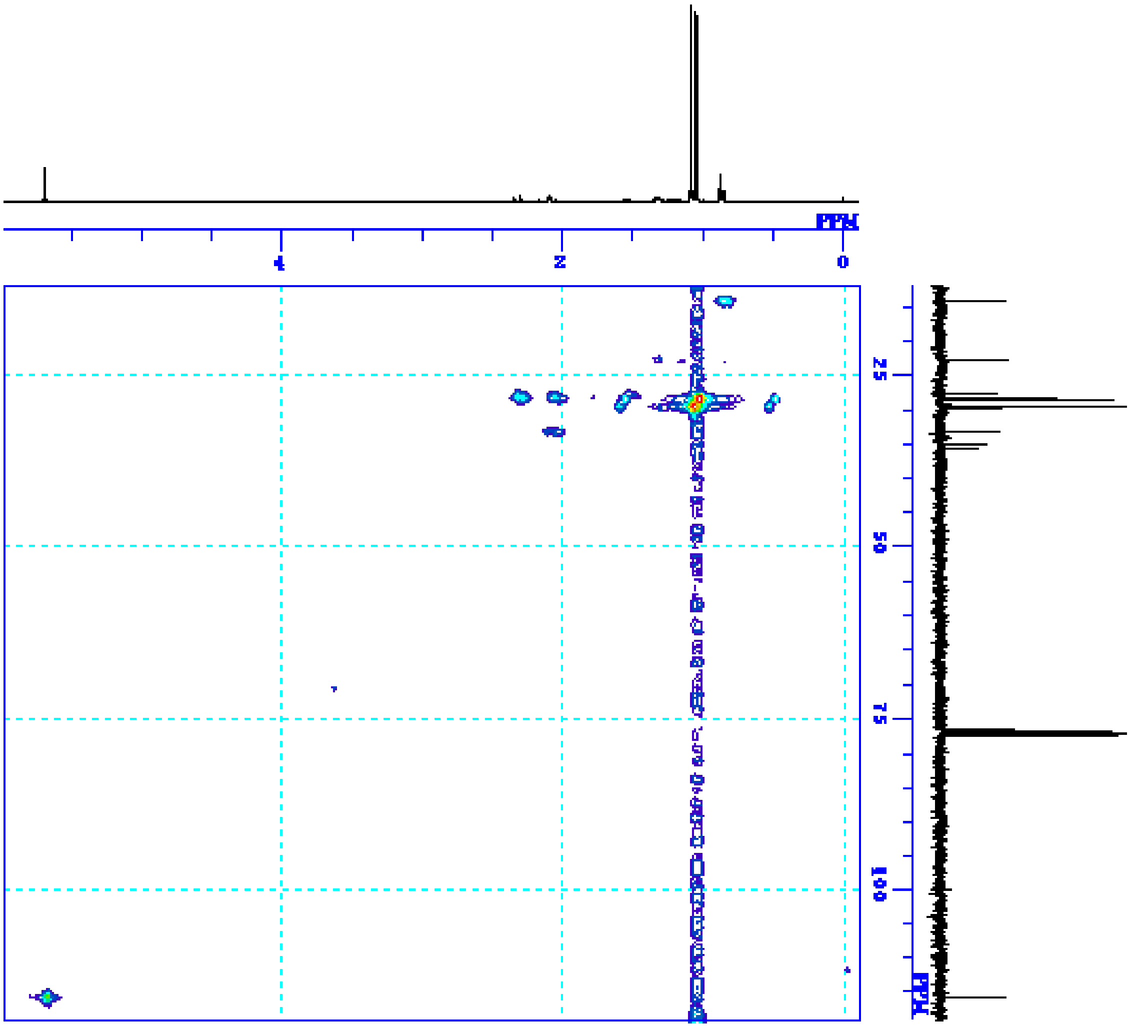

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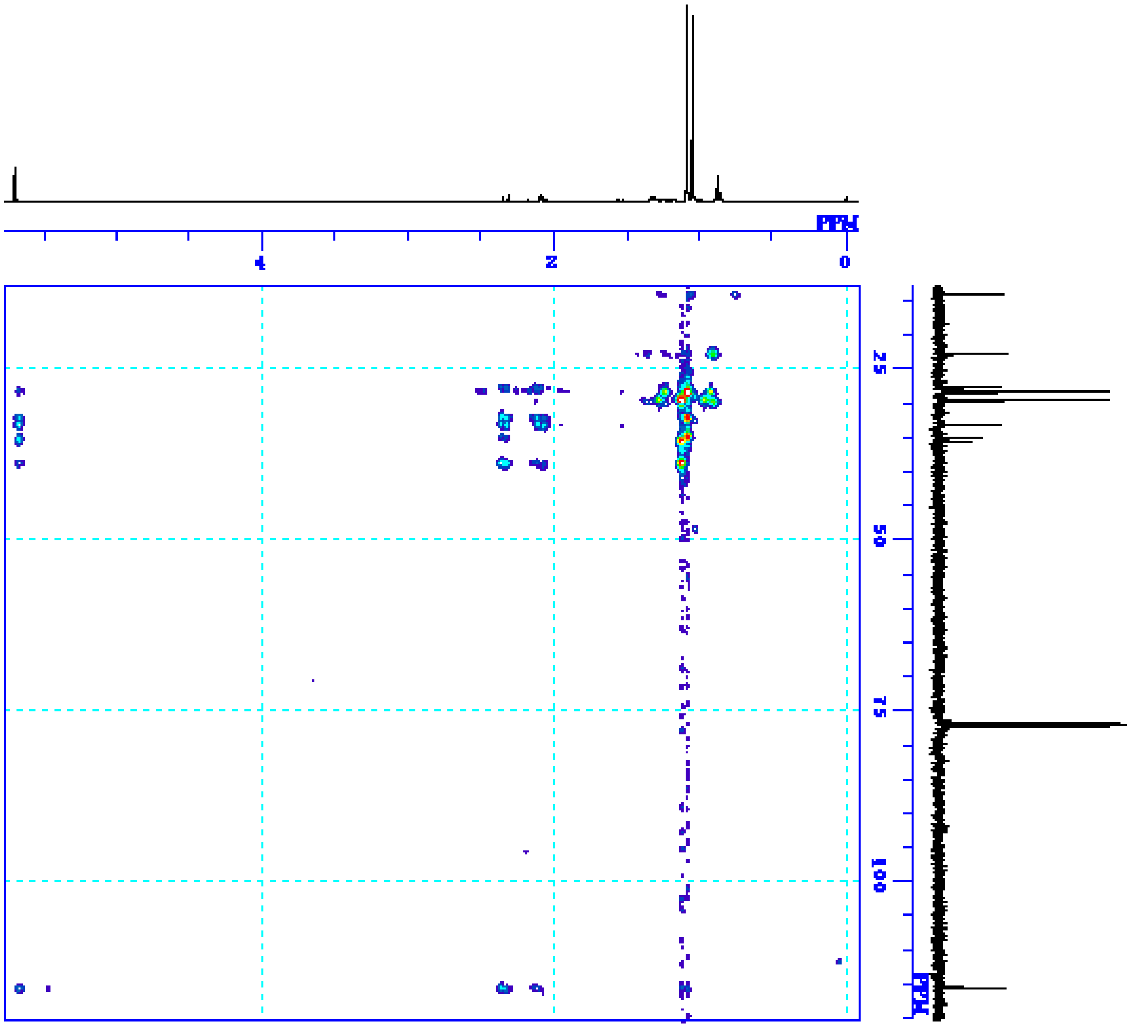

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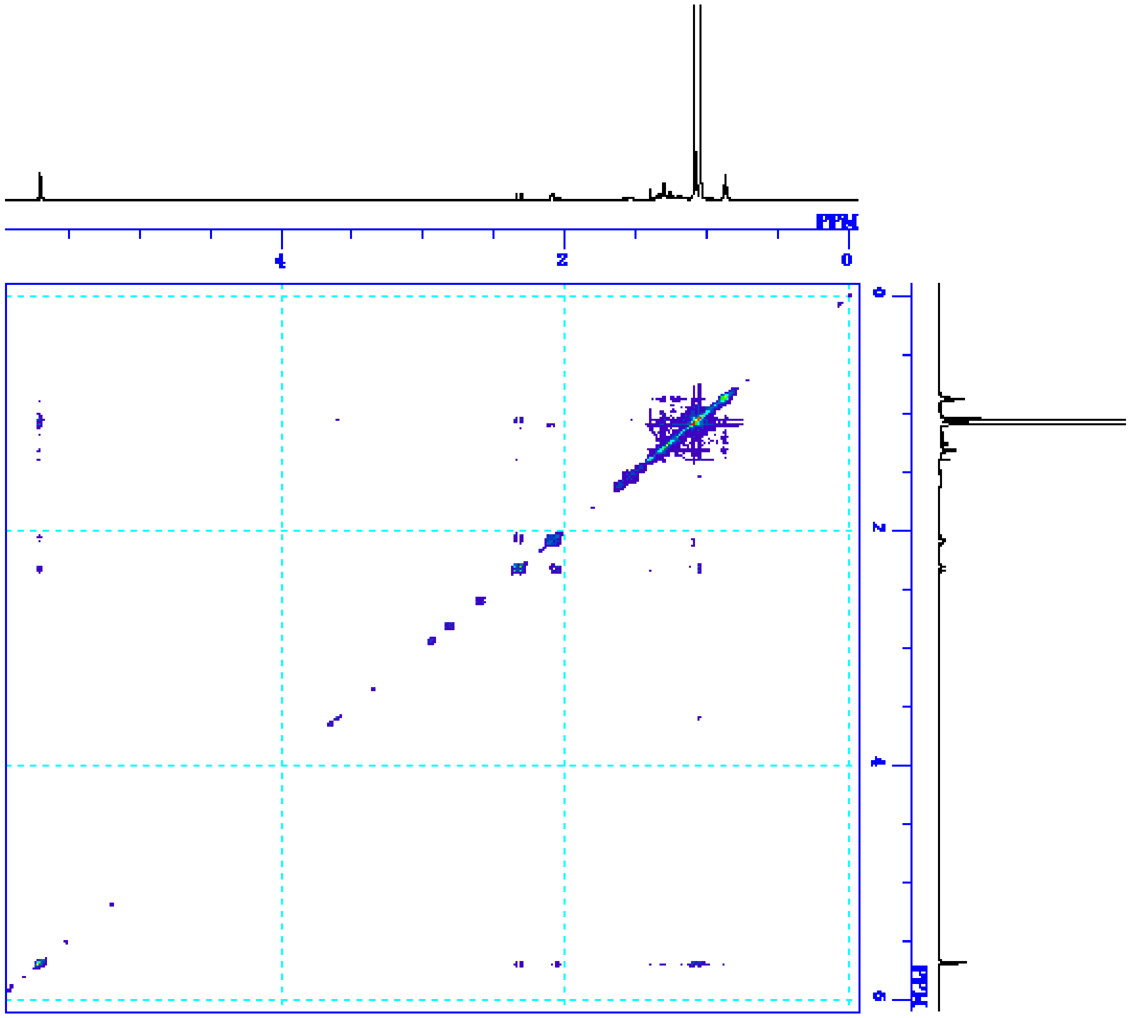

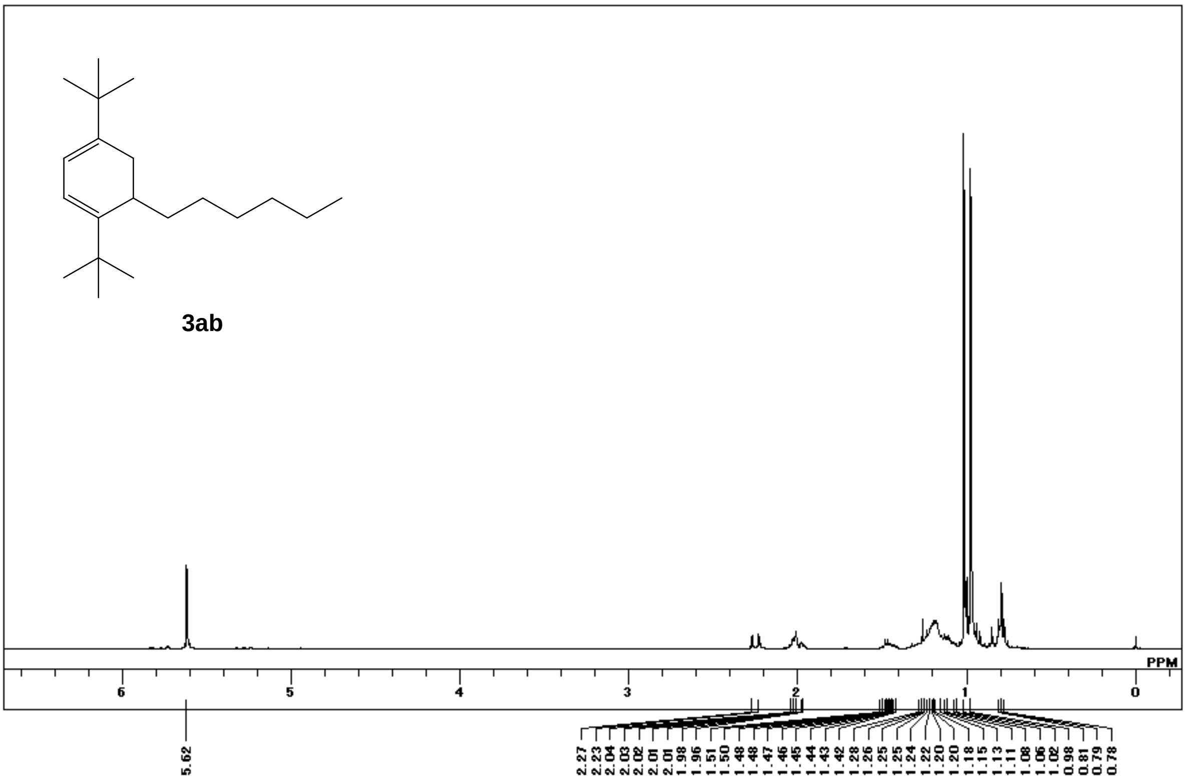
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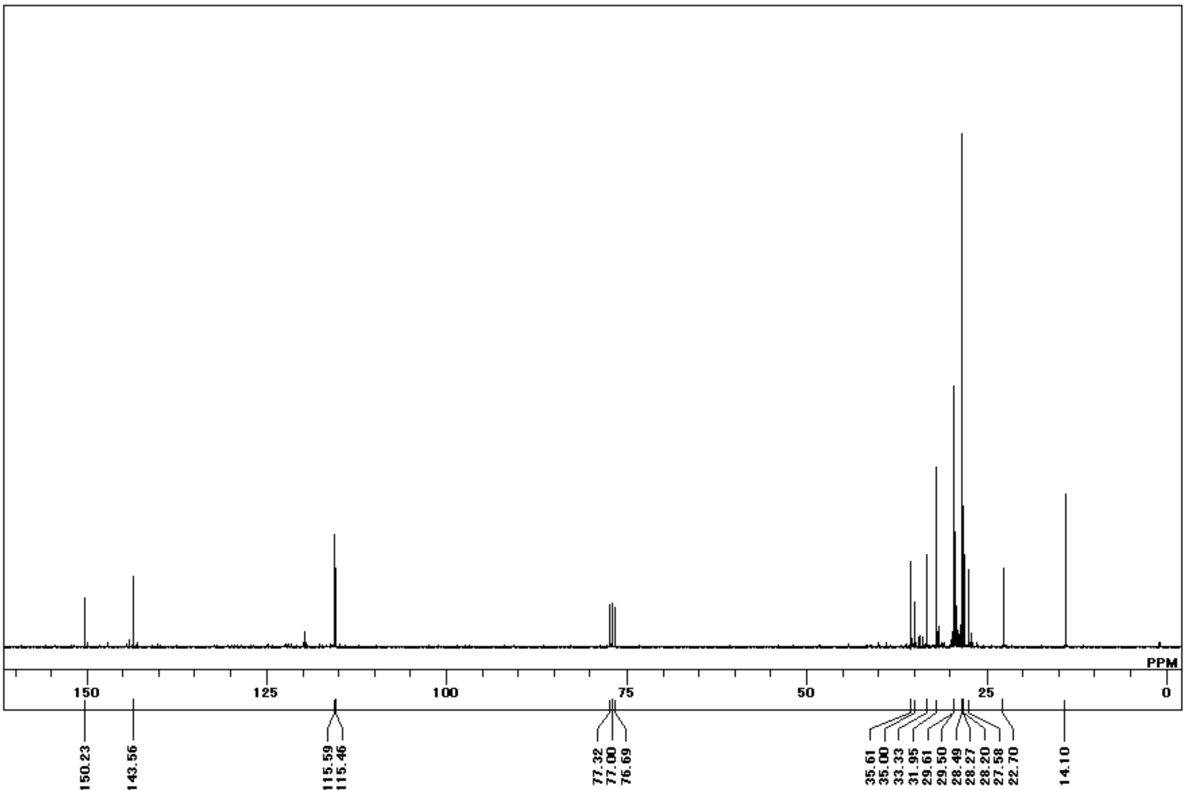
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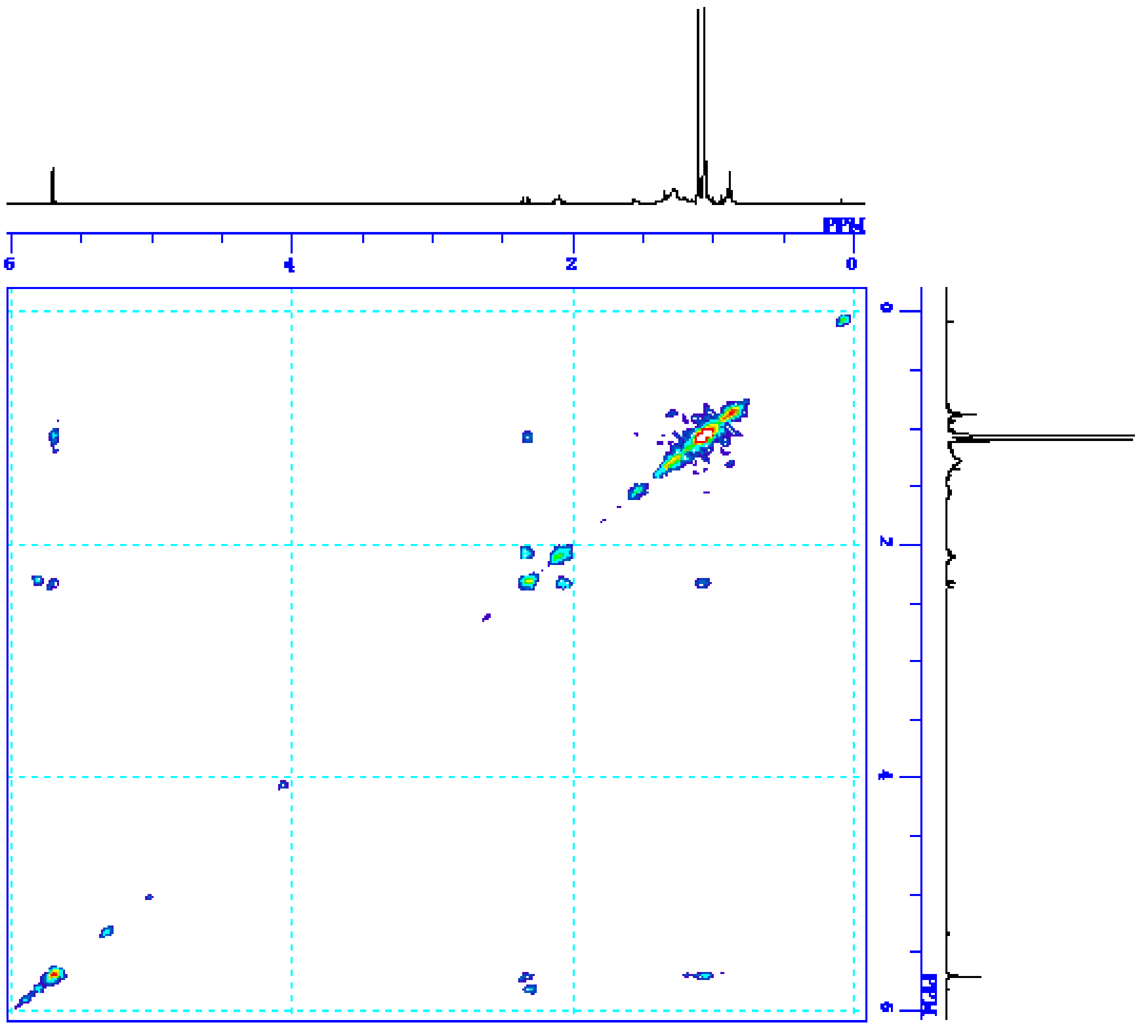
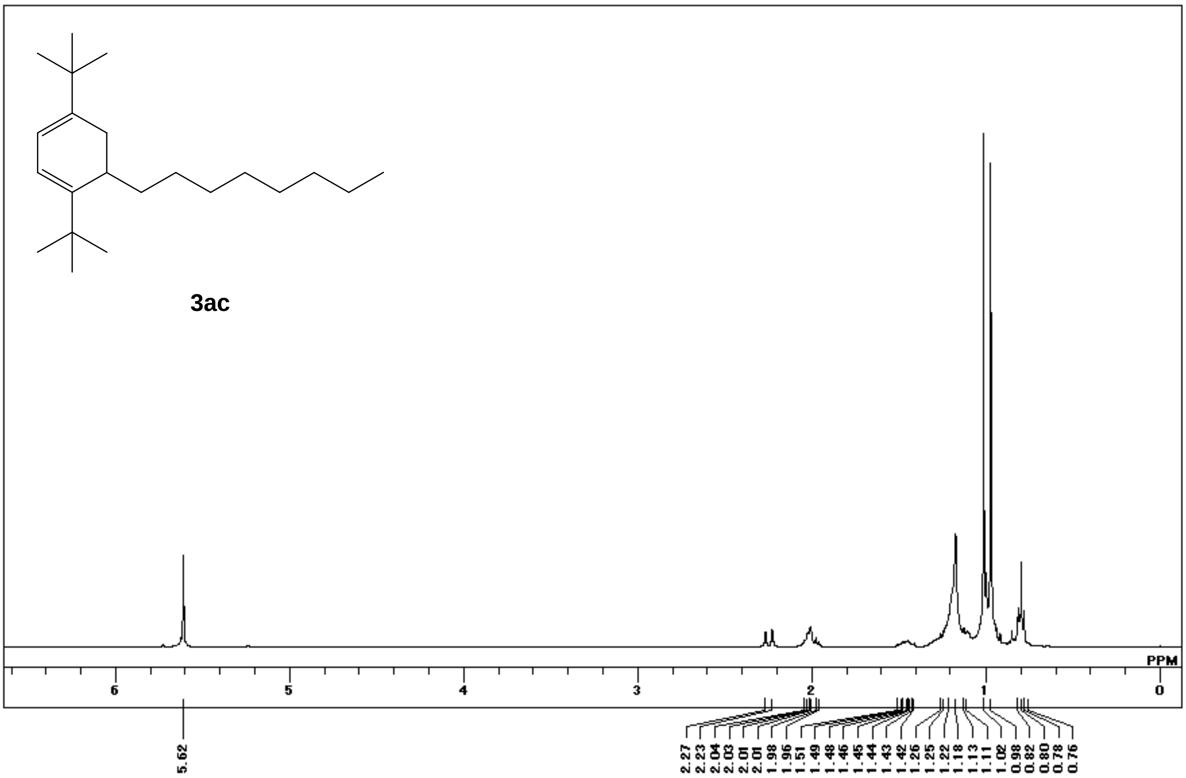
NOESY

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1.46
1.45
1.44
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1.26
1.25
1.22
1.18
1.13
1.11
1.02
0.98
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0.80
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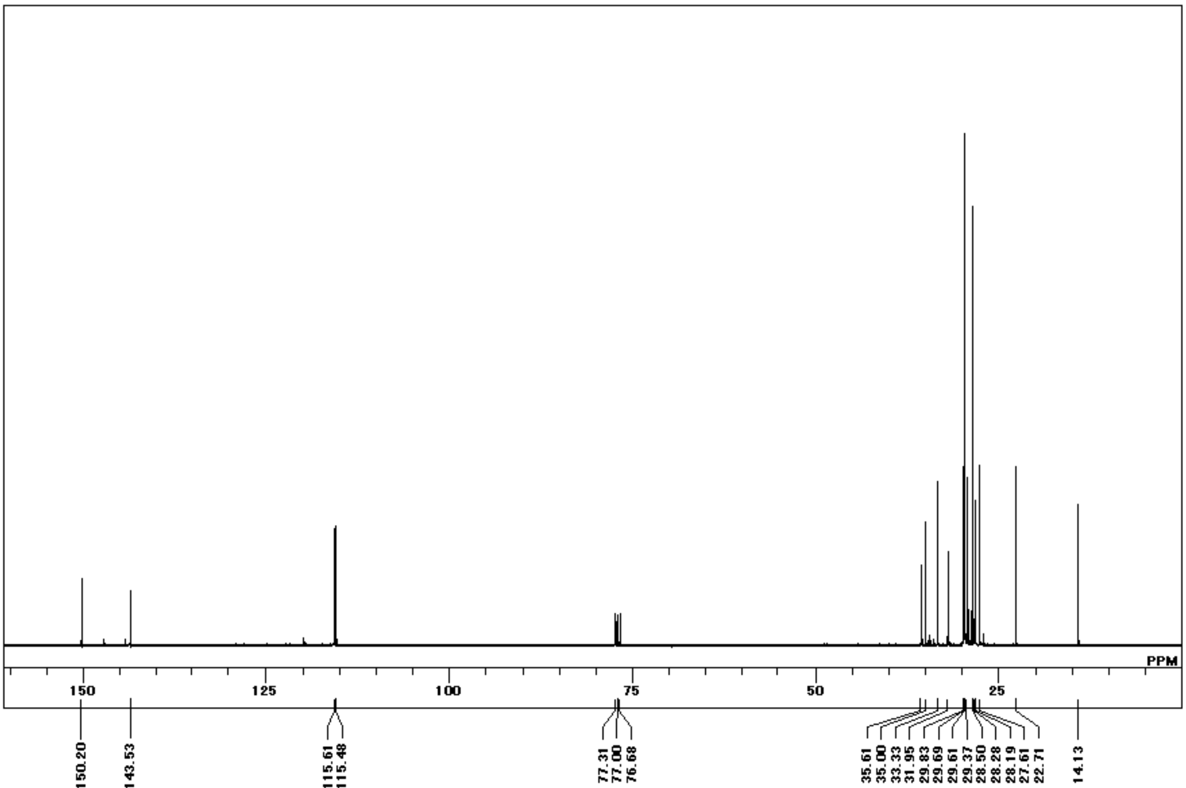
0.76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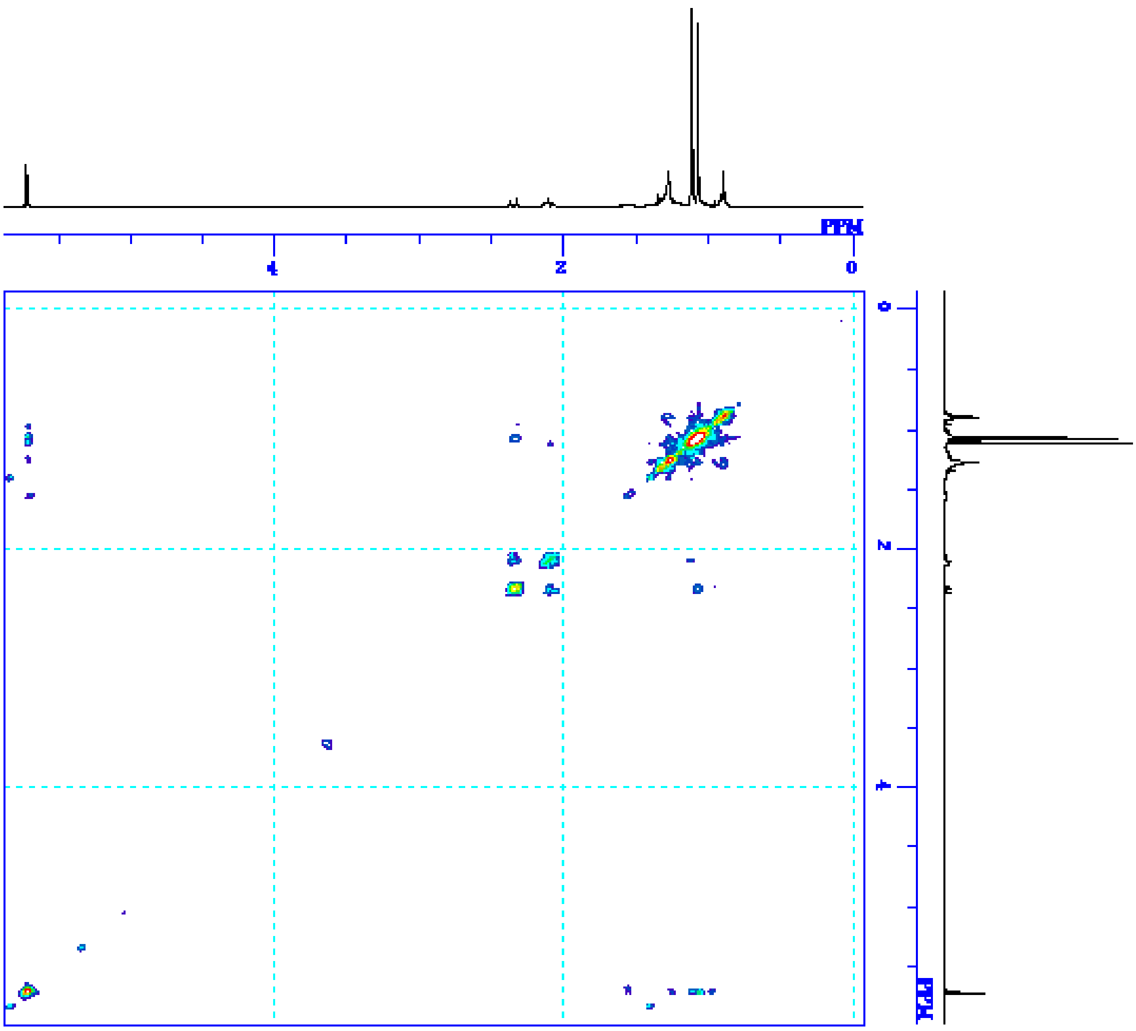

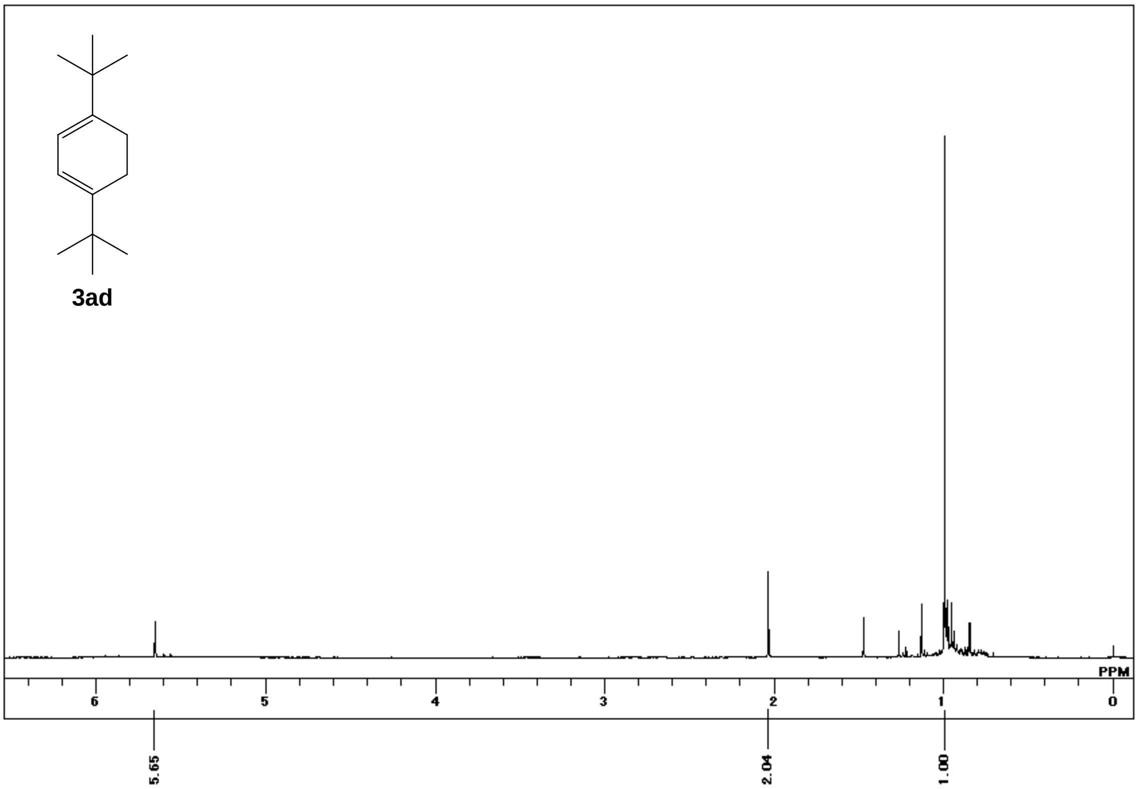
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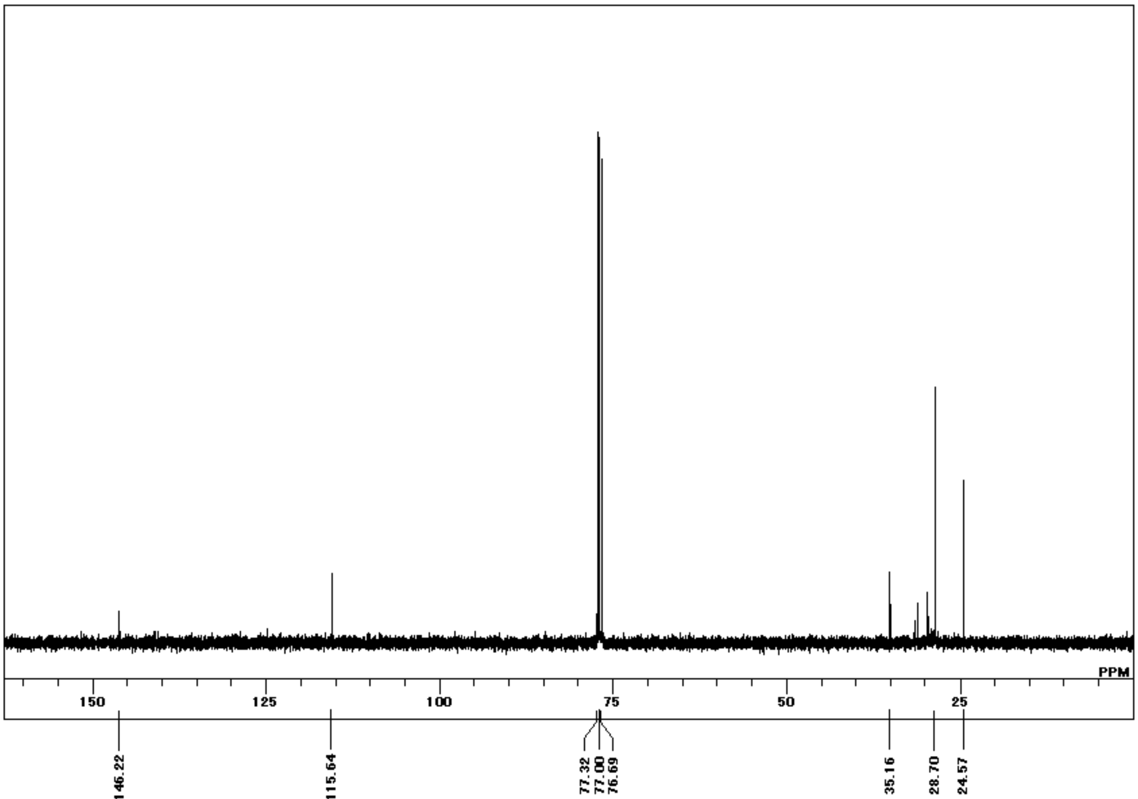
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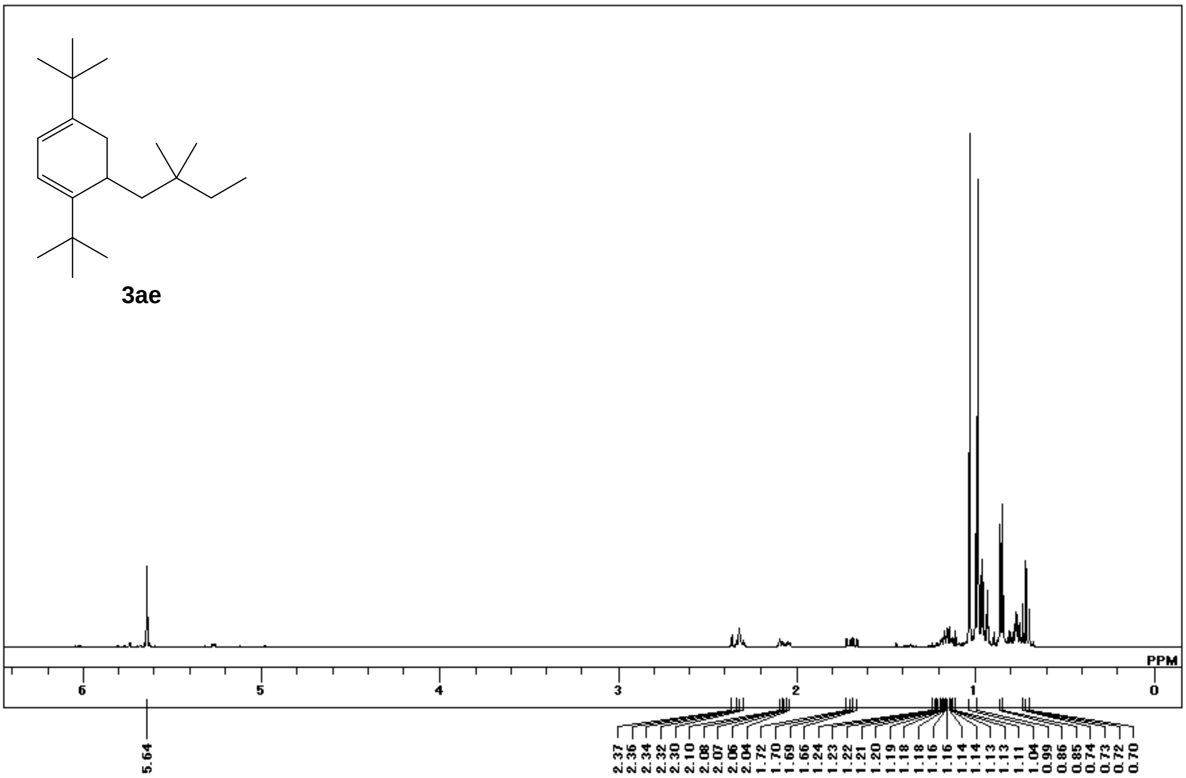
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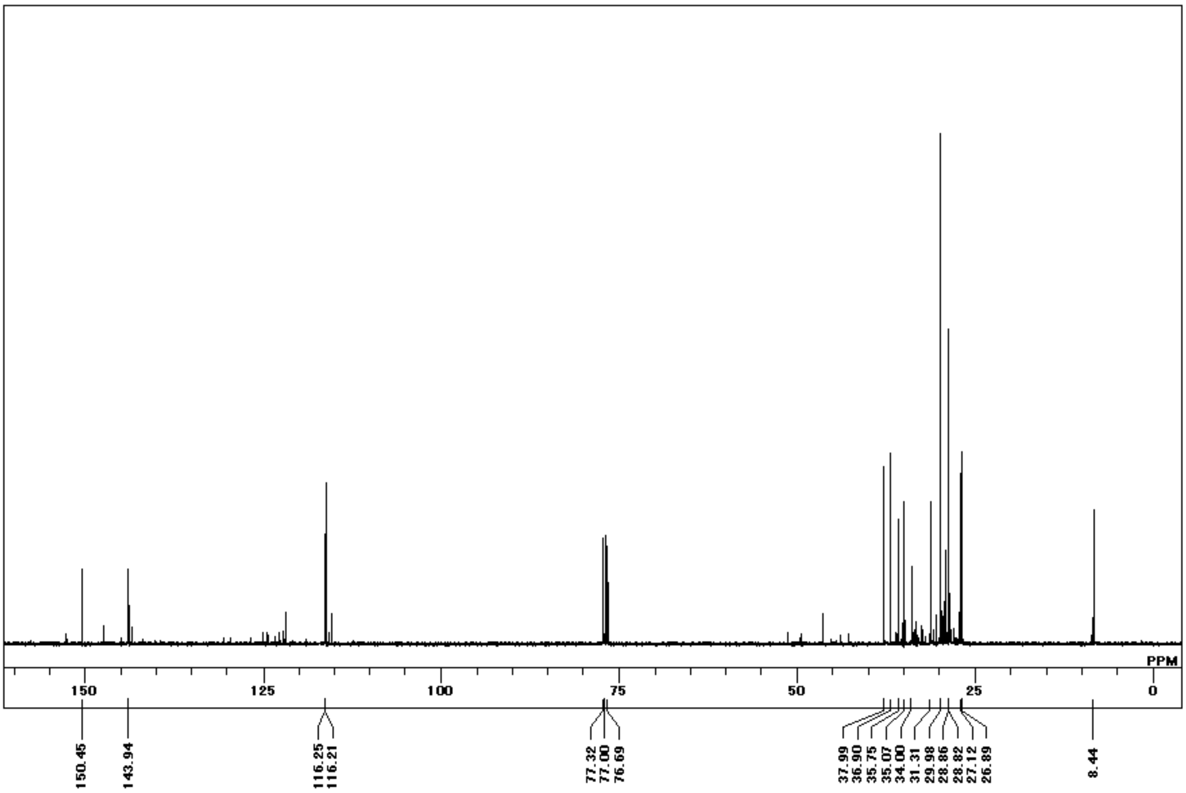
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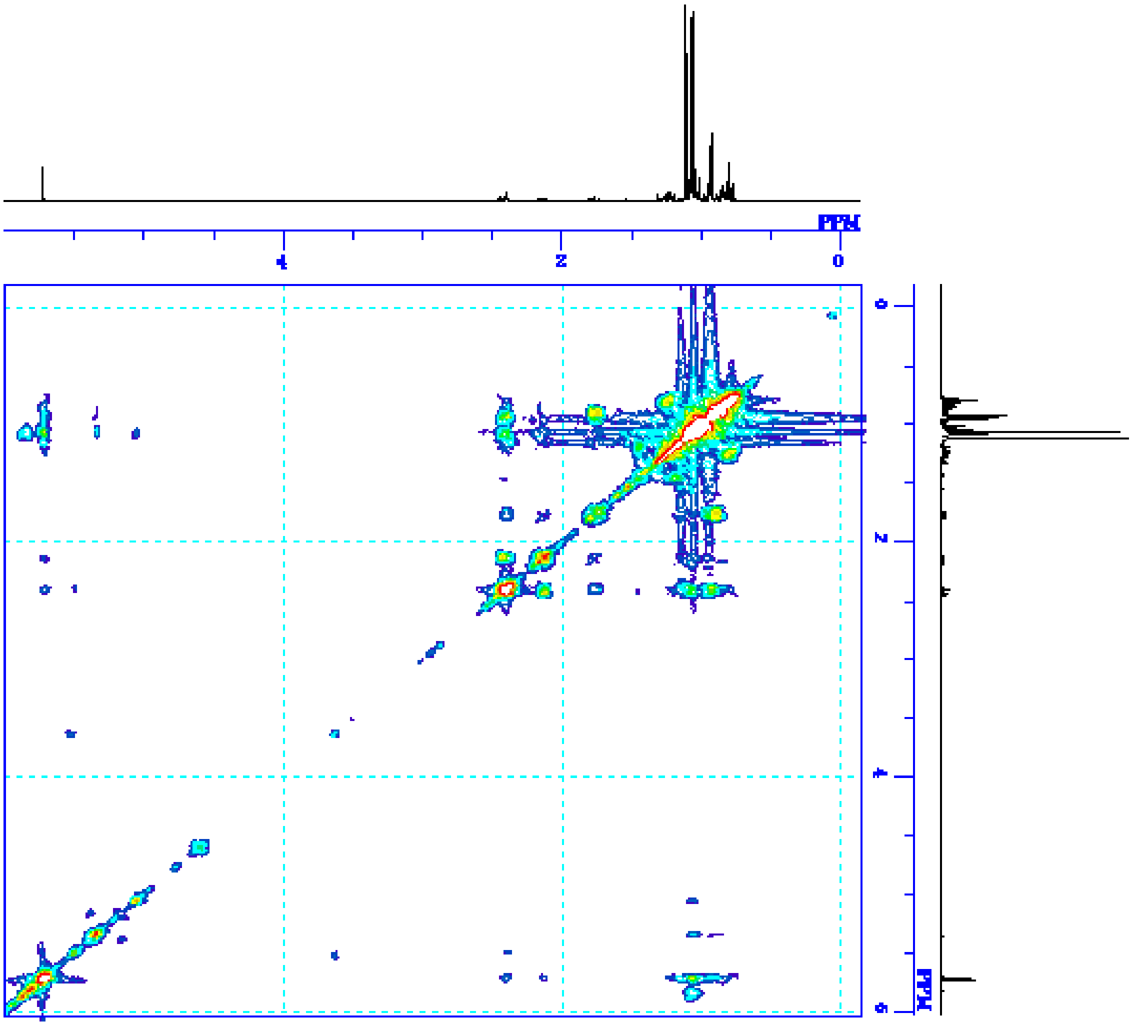

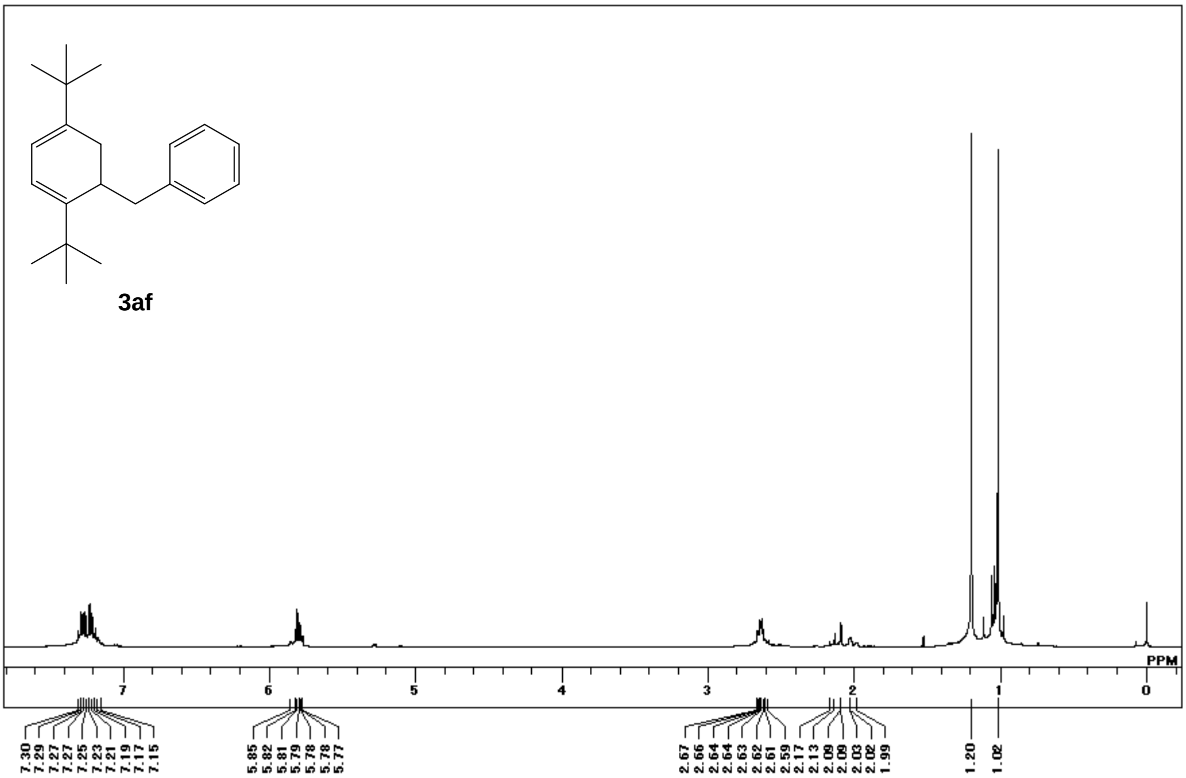
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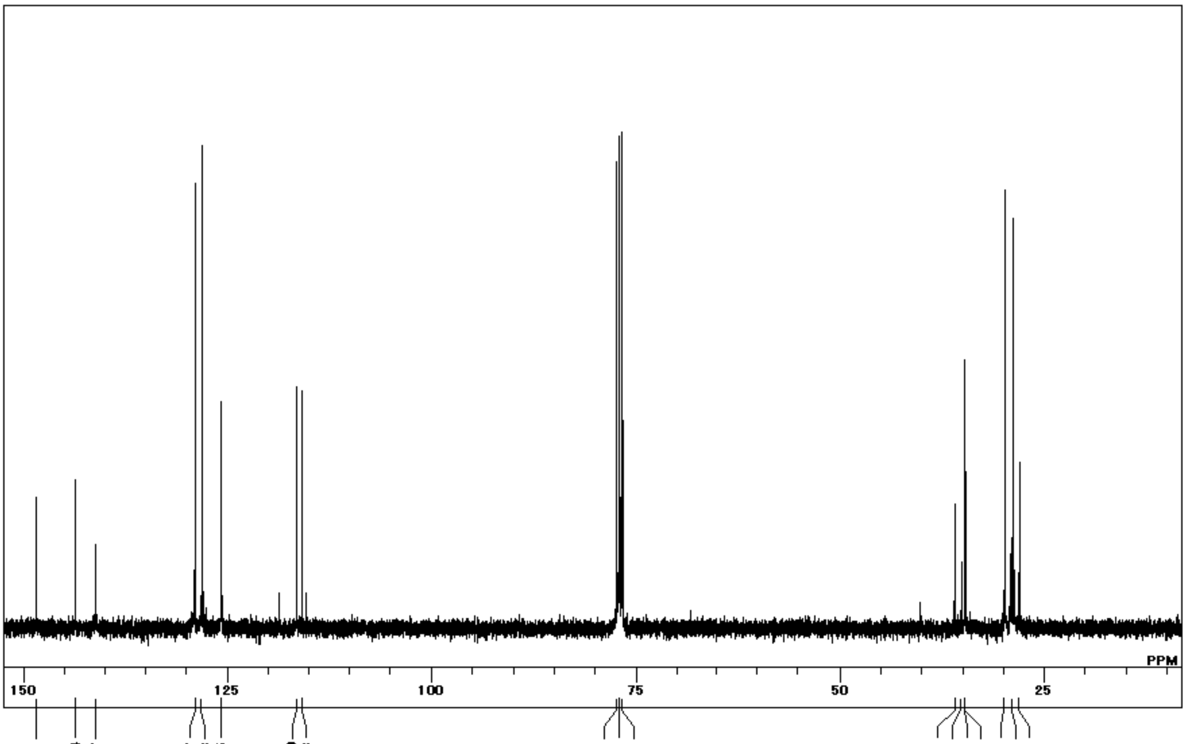
1.20 1.02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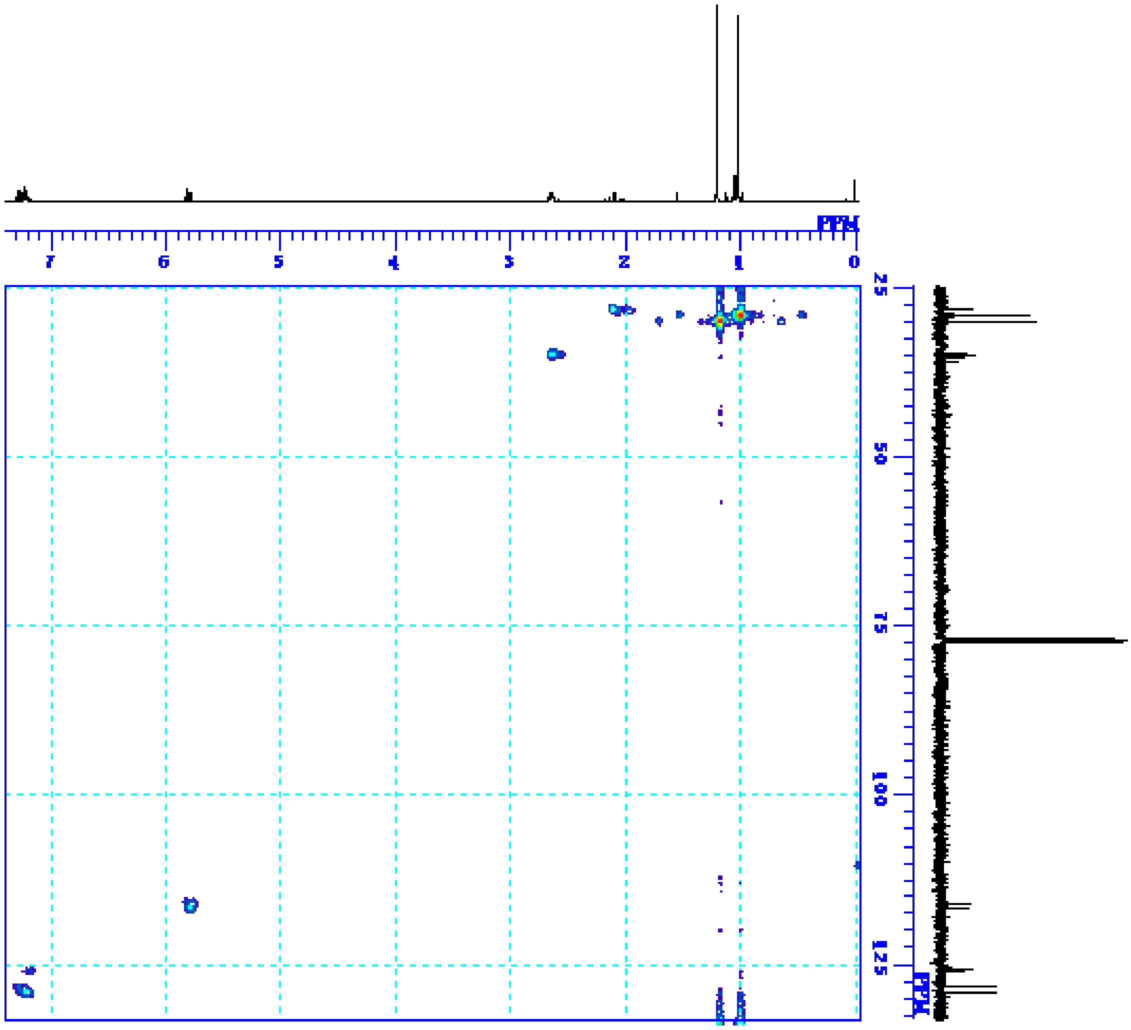
HMQC



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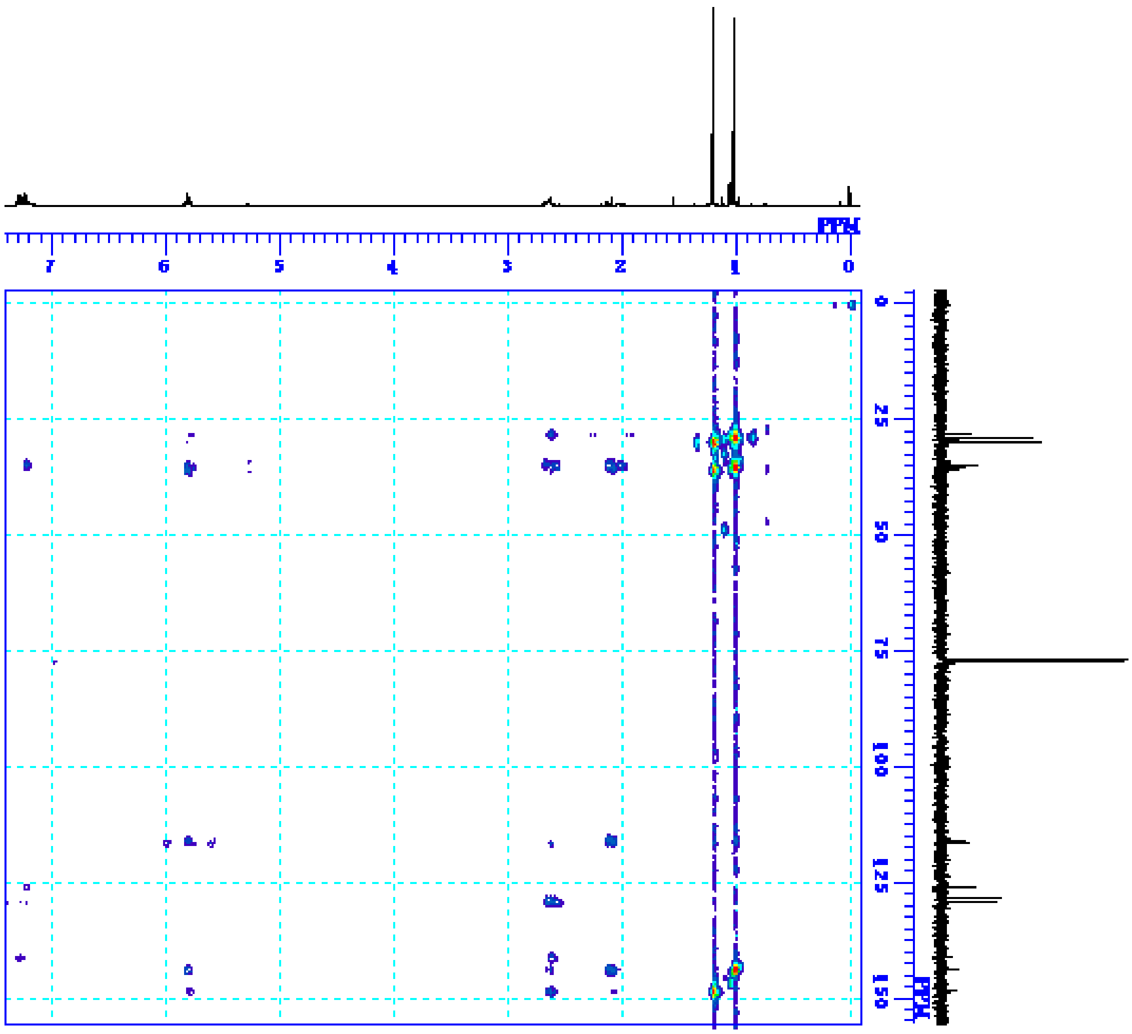

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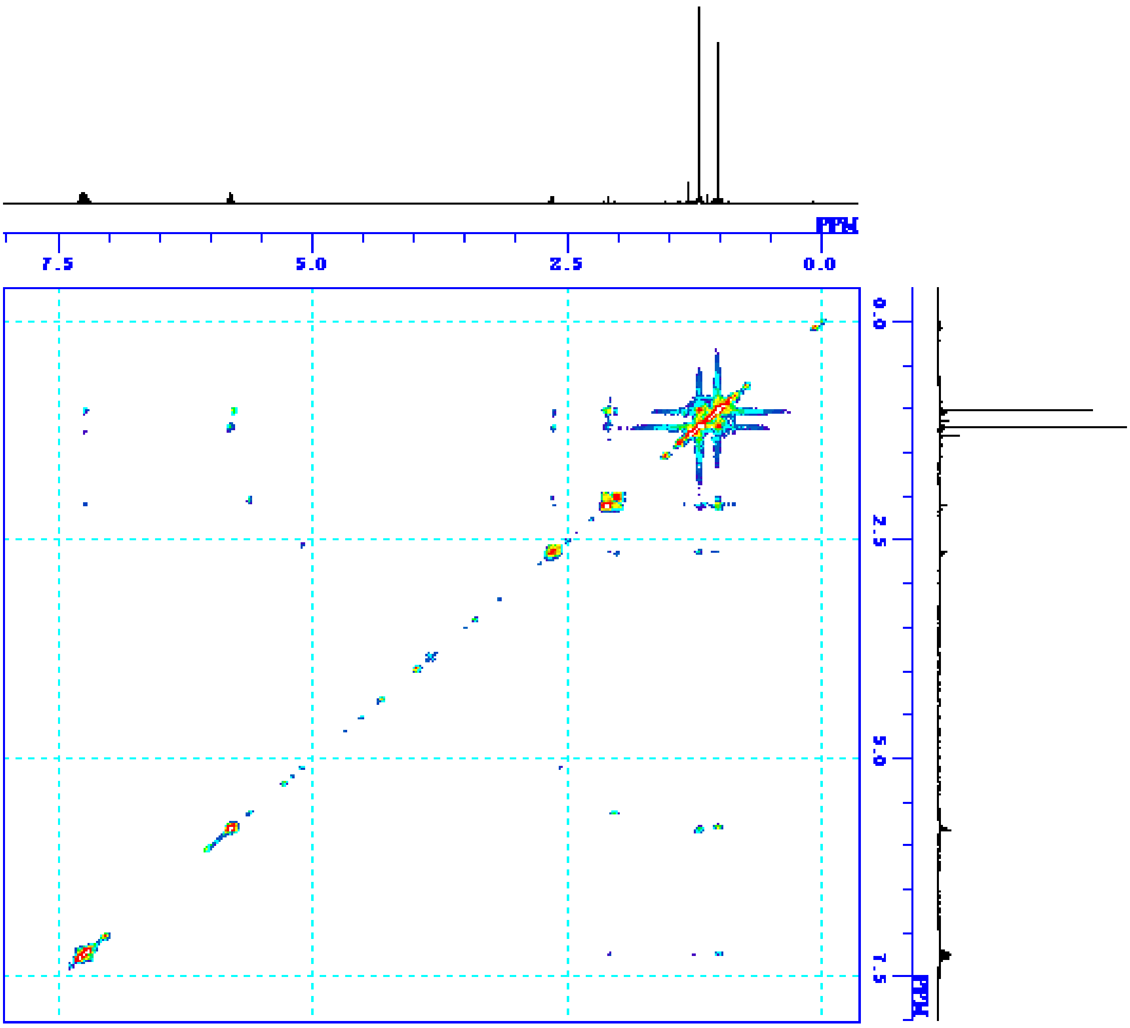

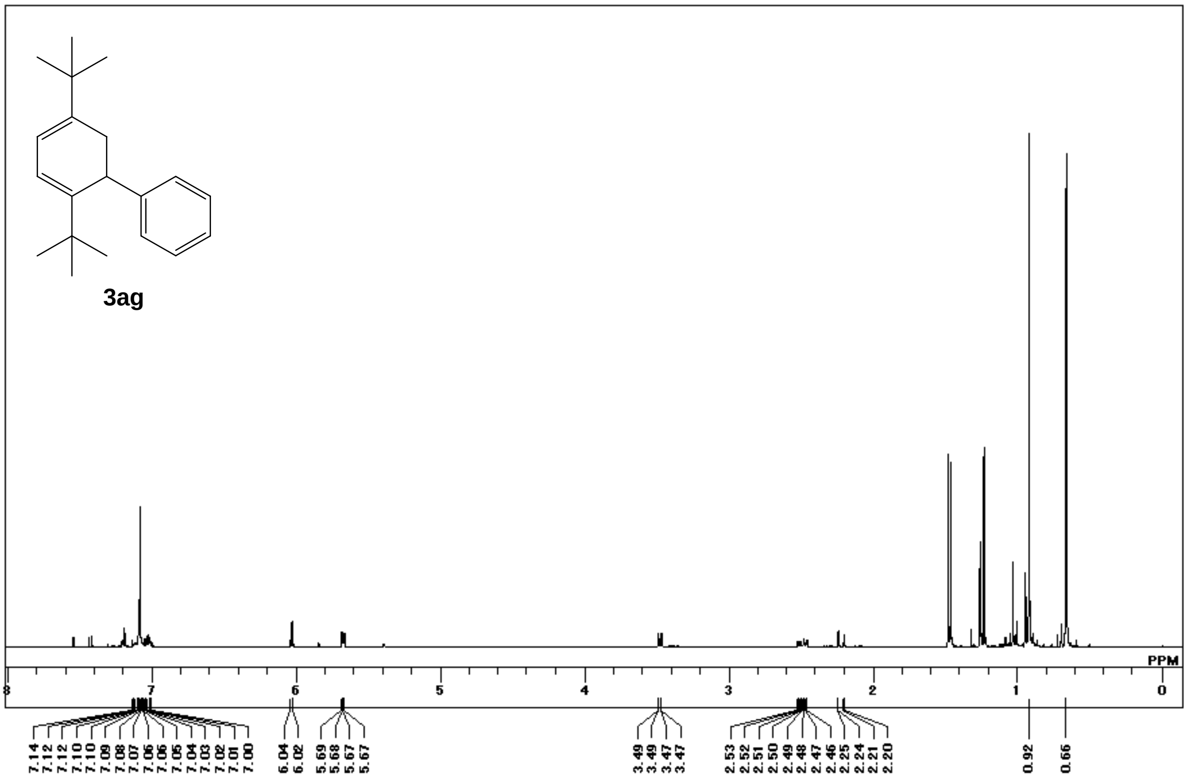
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6.04 6.02
5.69 5.68 5.67 5.67
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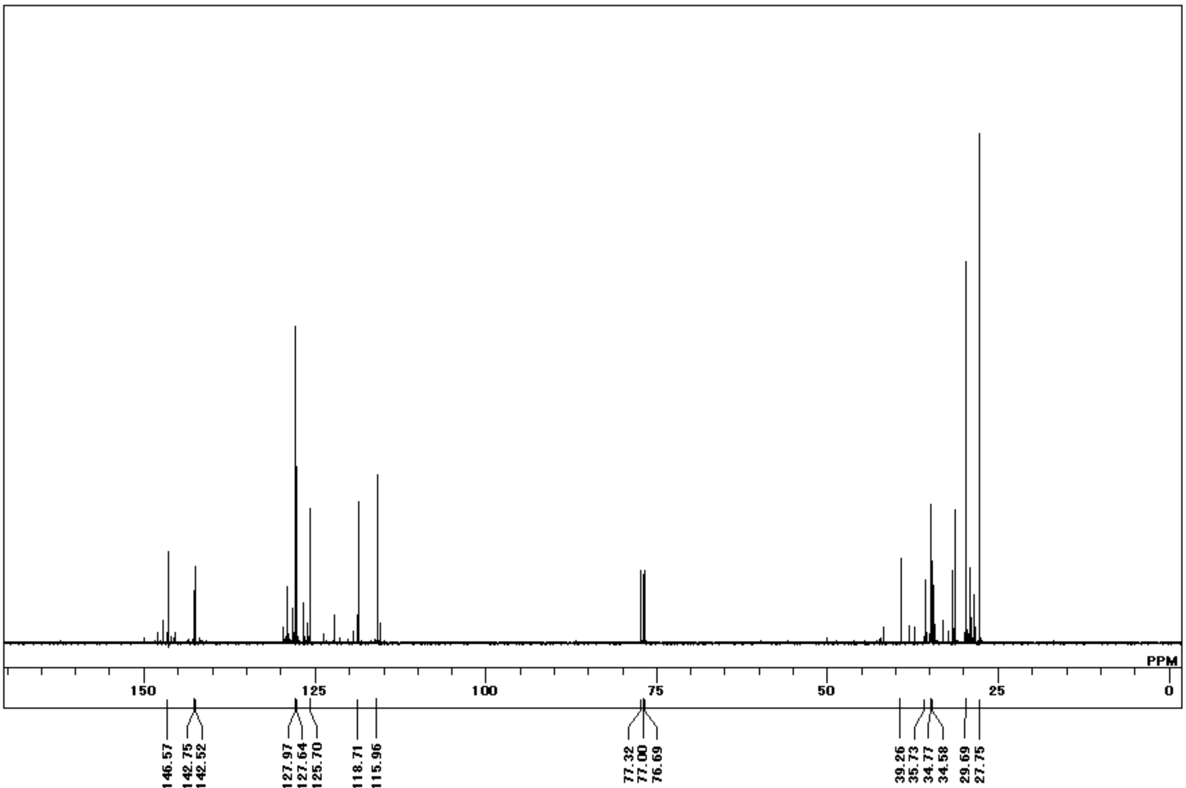
0.66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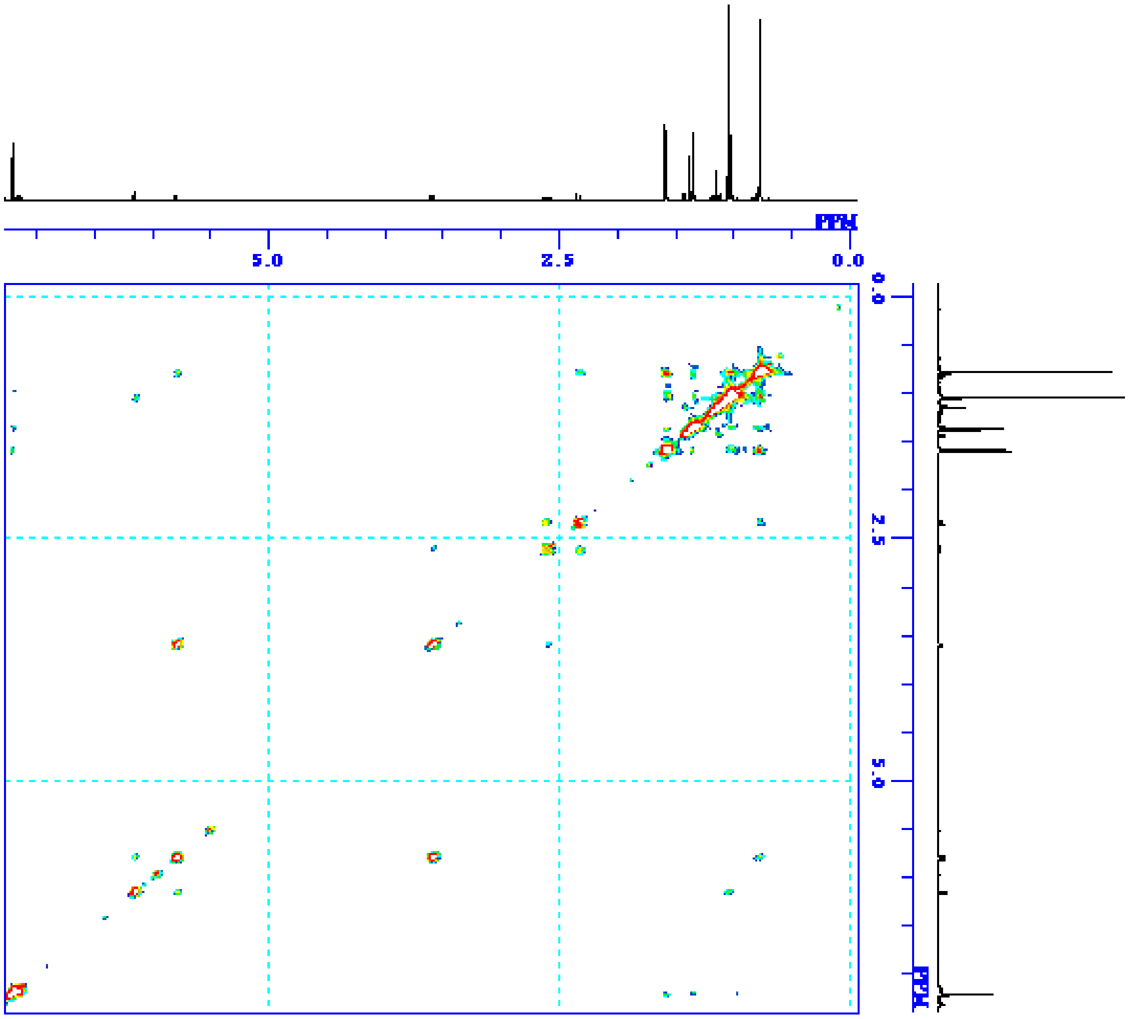
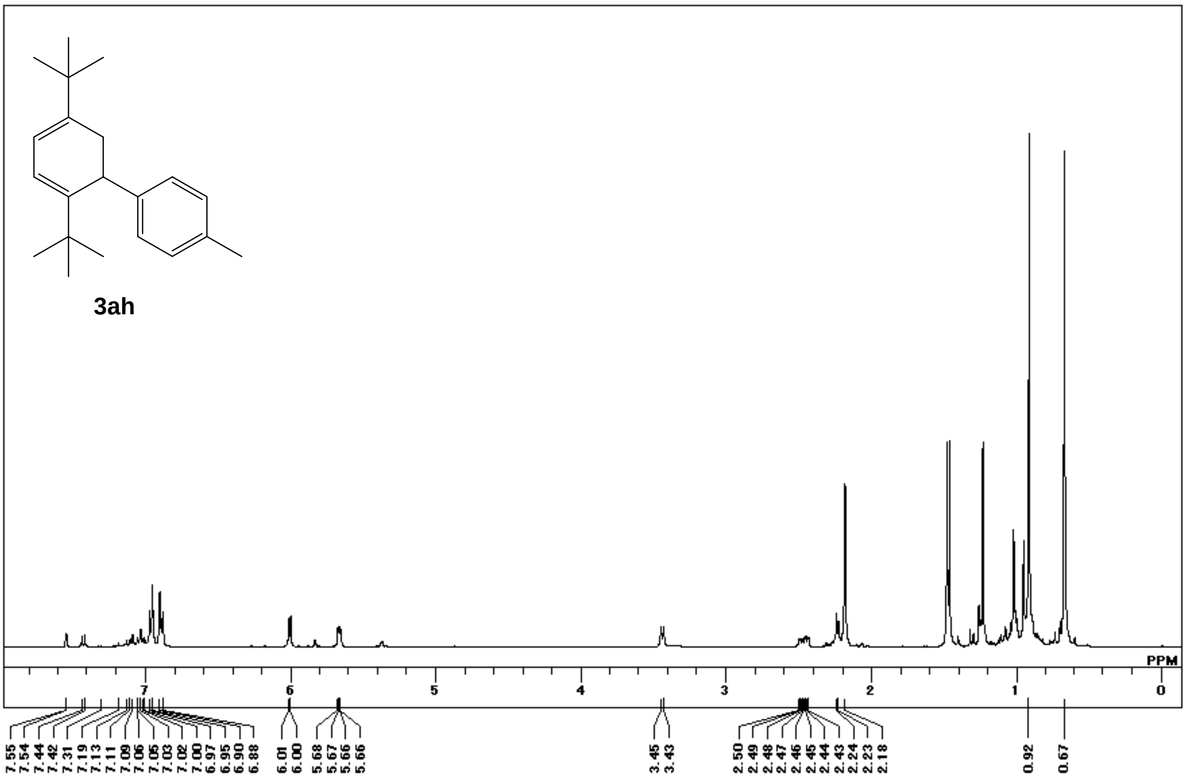
NOESY

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6.01 6.00
5.68 5.67 5.66 5.66
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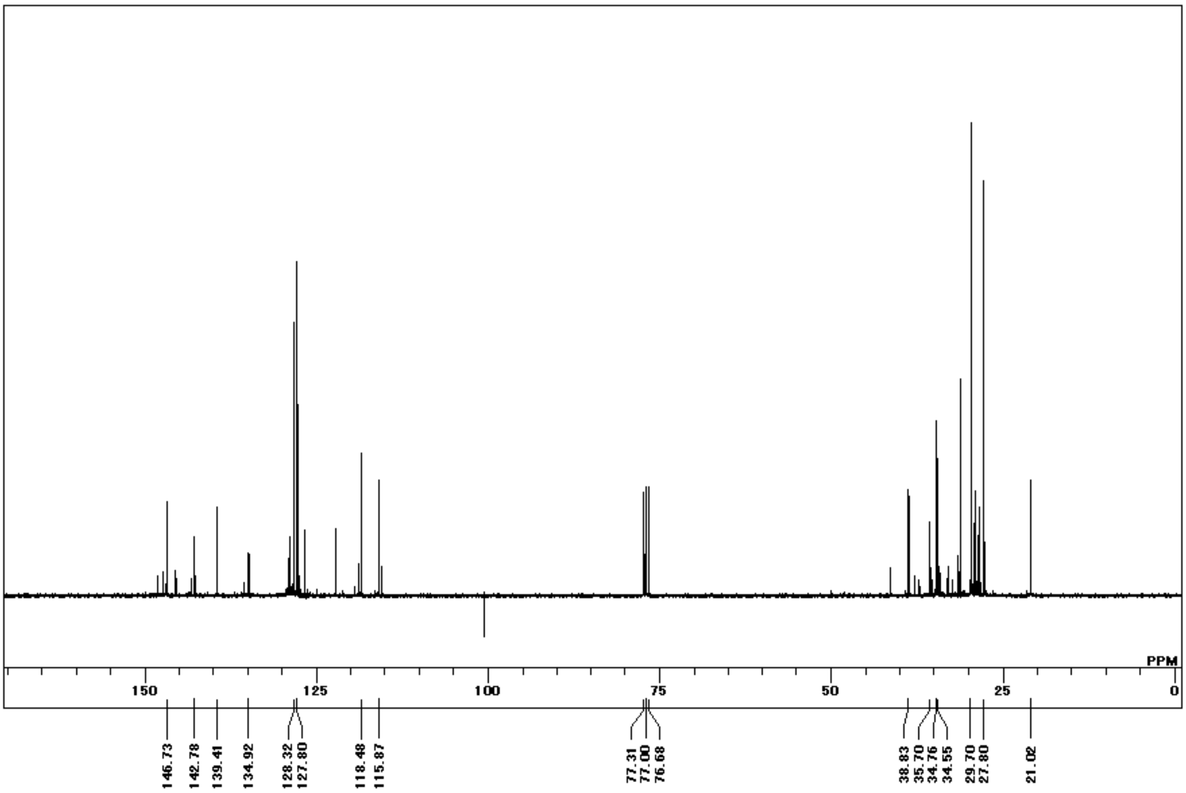
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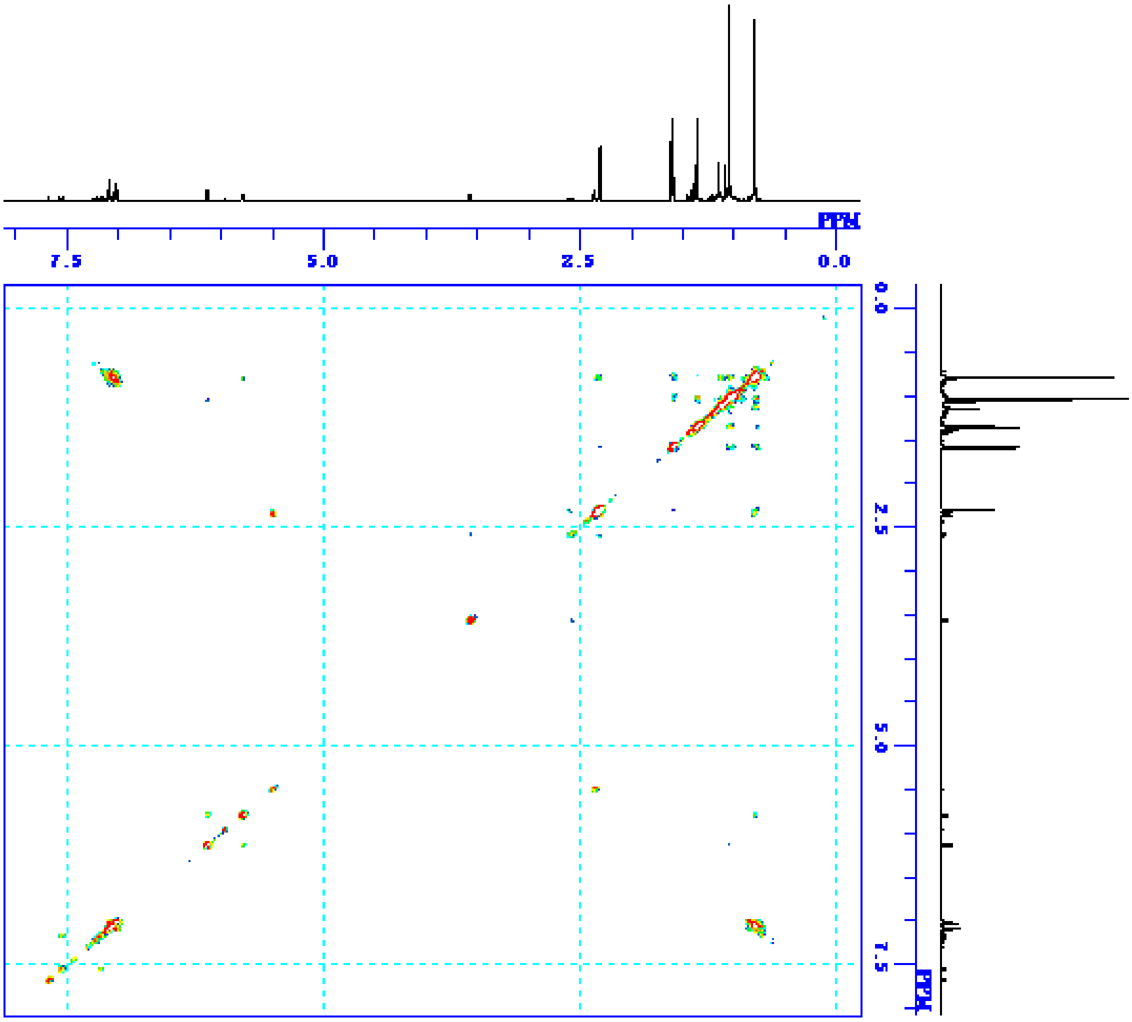

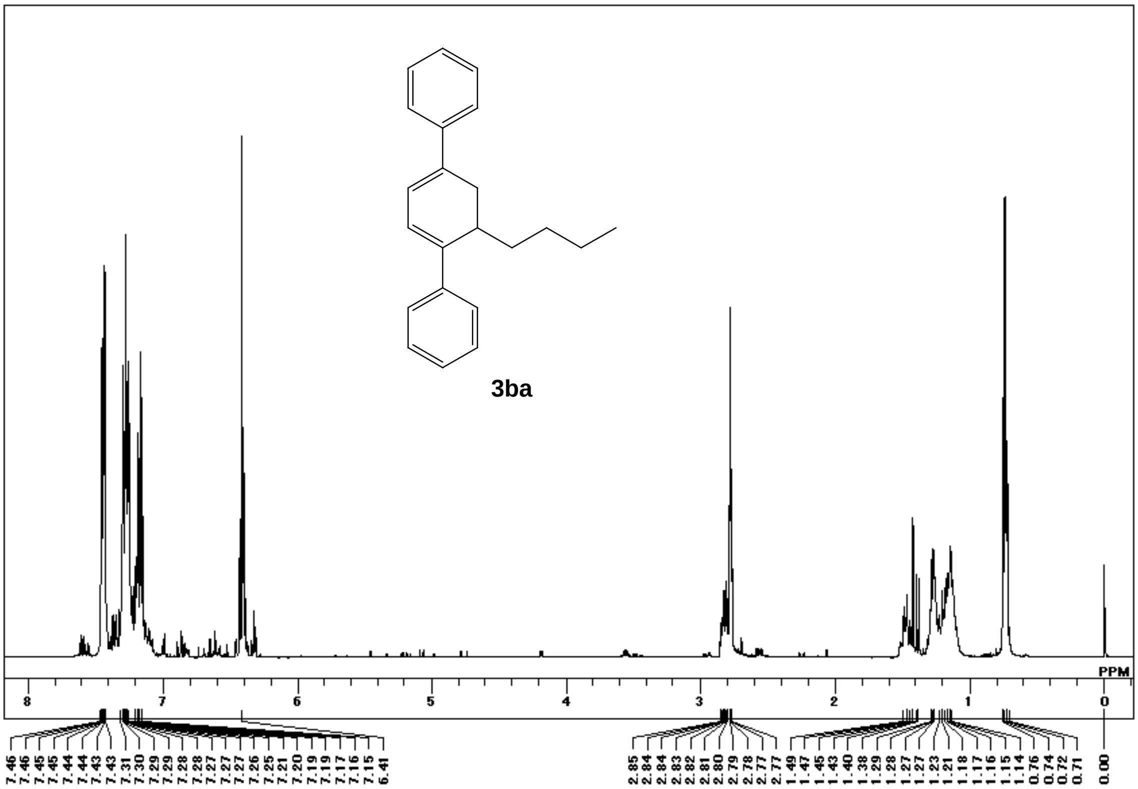
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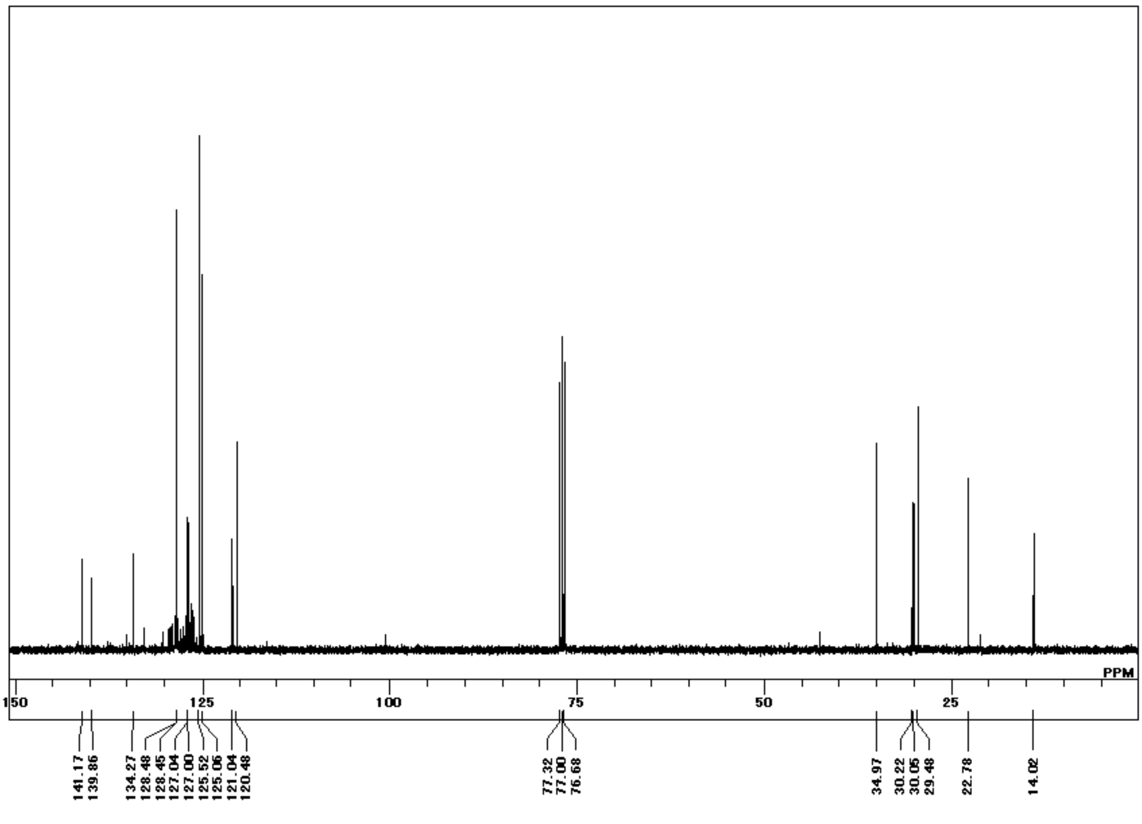
2.85 2.84 2.84 2.83 2.82 2.81 2.80 2.79 2.78 2.77 2.77 1.49 1.47 1.45 1.43 1.40 1.38 1.29 1.28 1.27 1.27 1.23 1.21 1.18 1.17 1.16 1.15 1.14 0.76 0.74 0.72 0.71 0.00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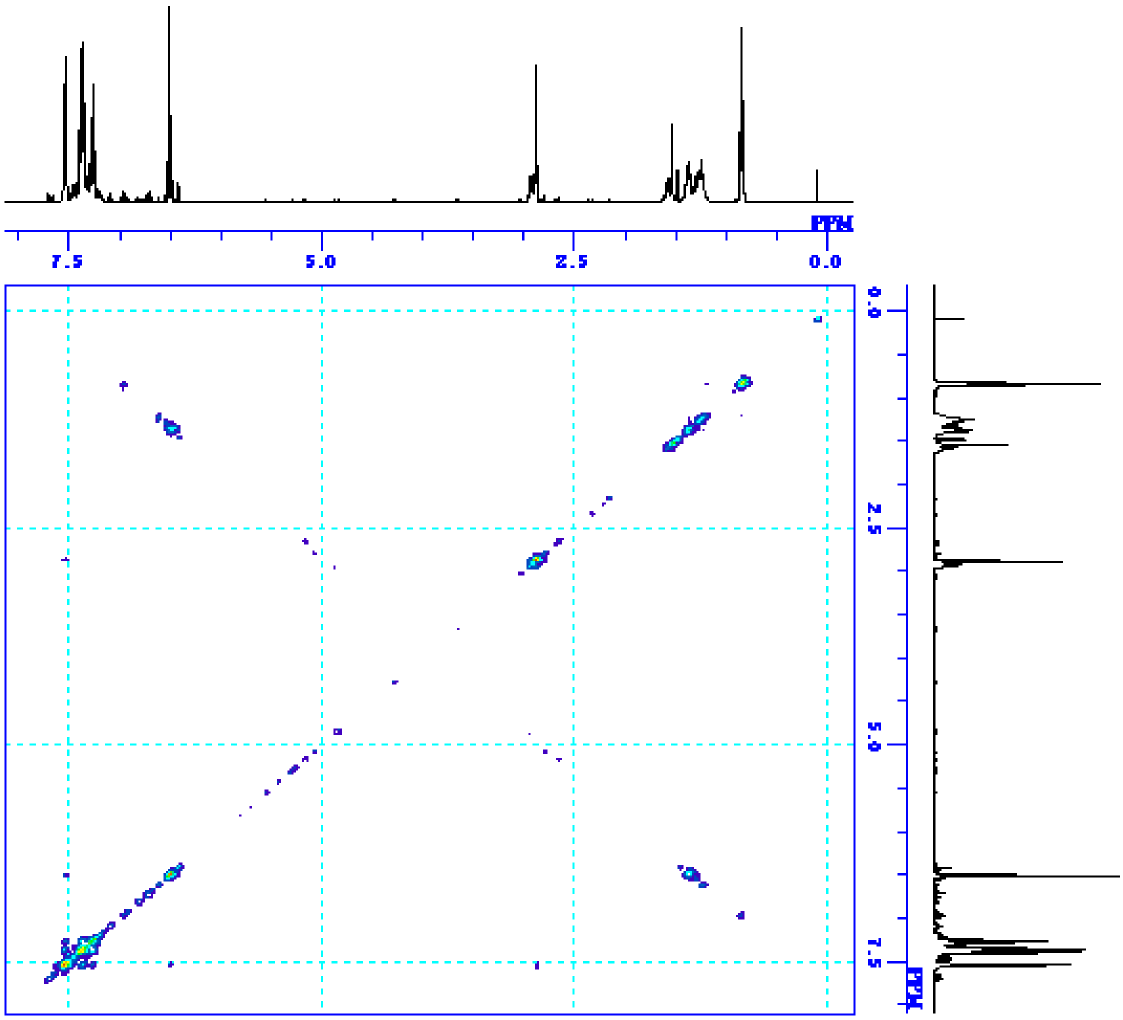
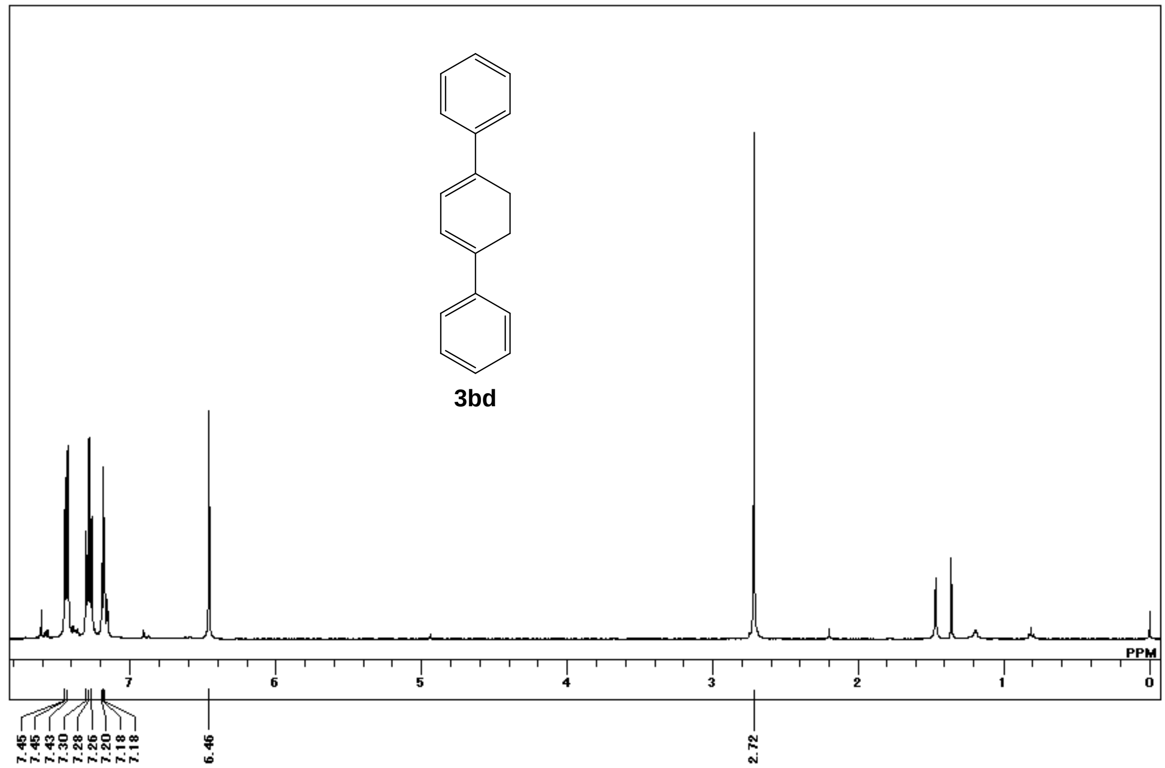
NOESY

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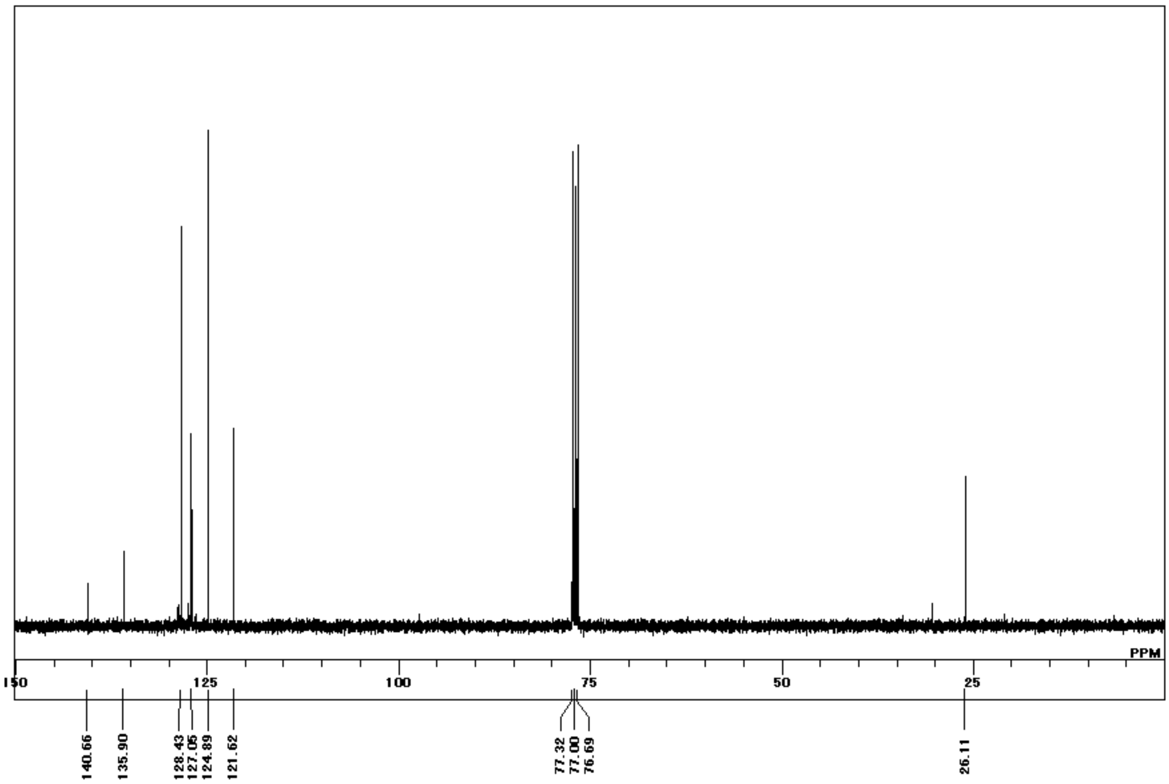
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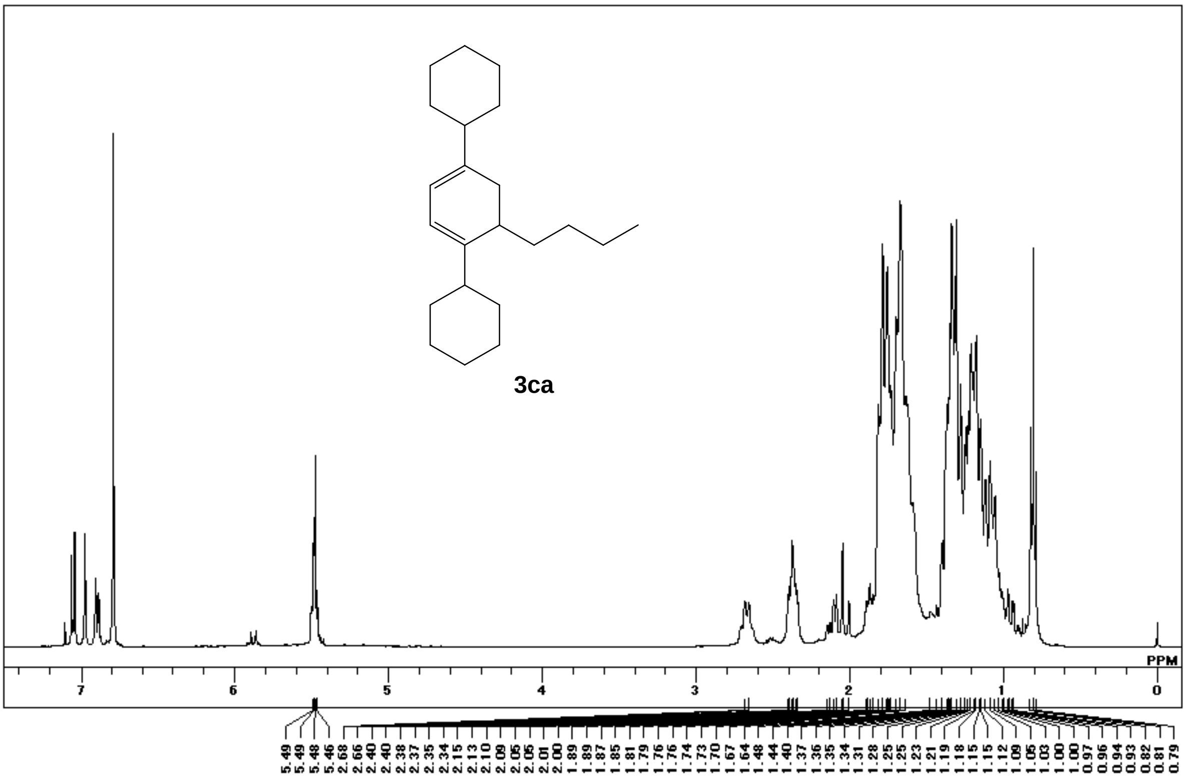
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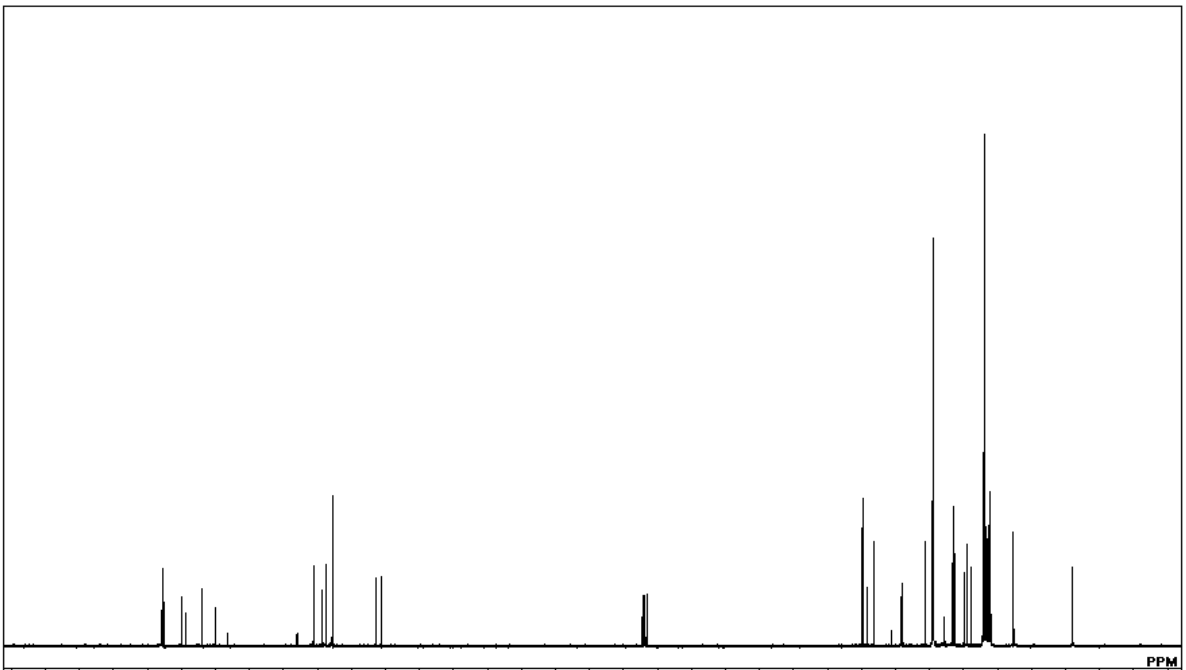
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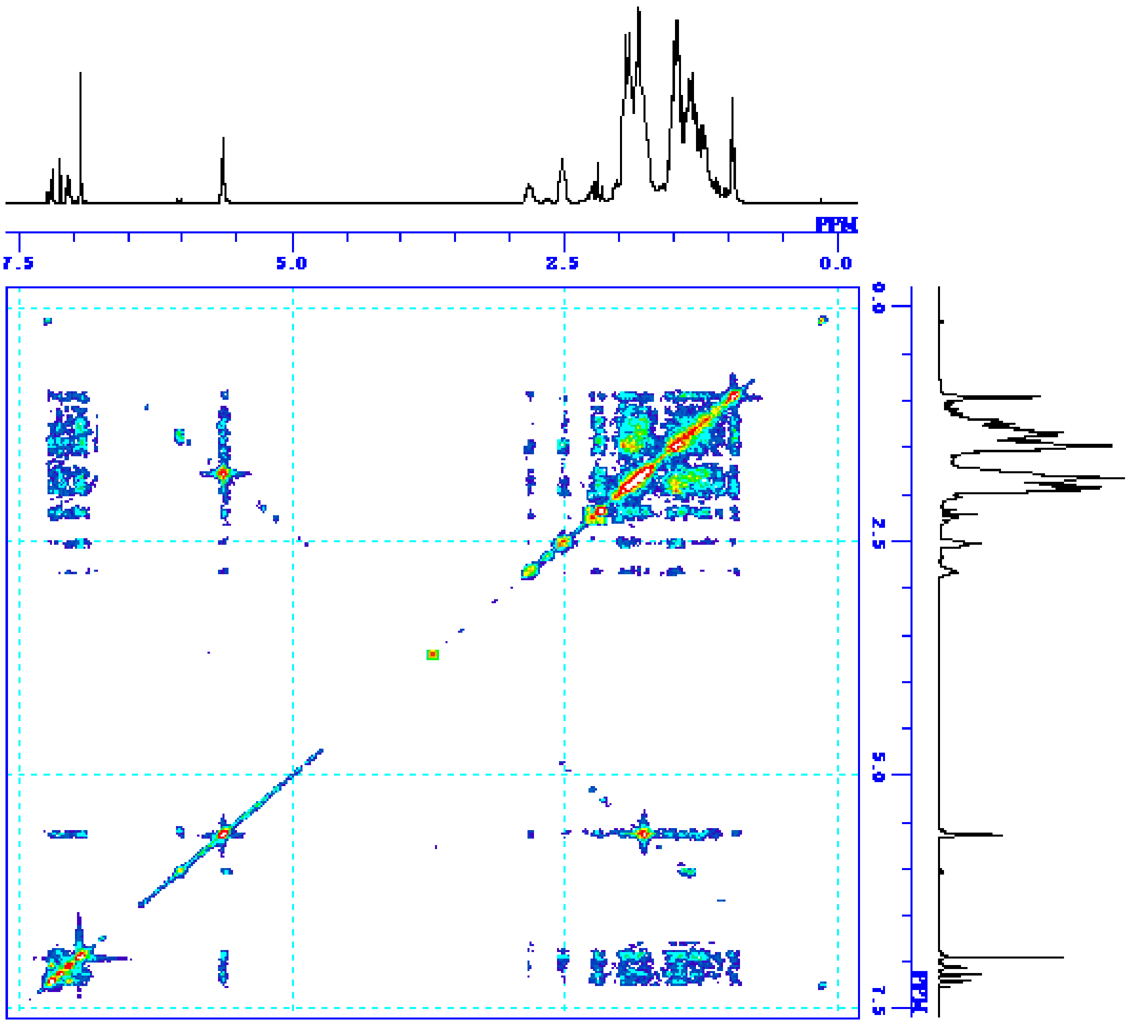
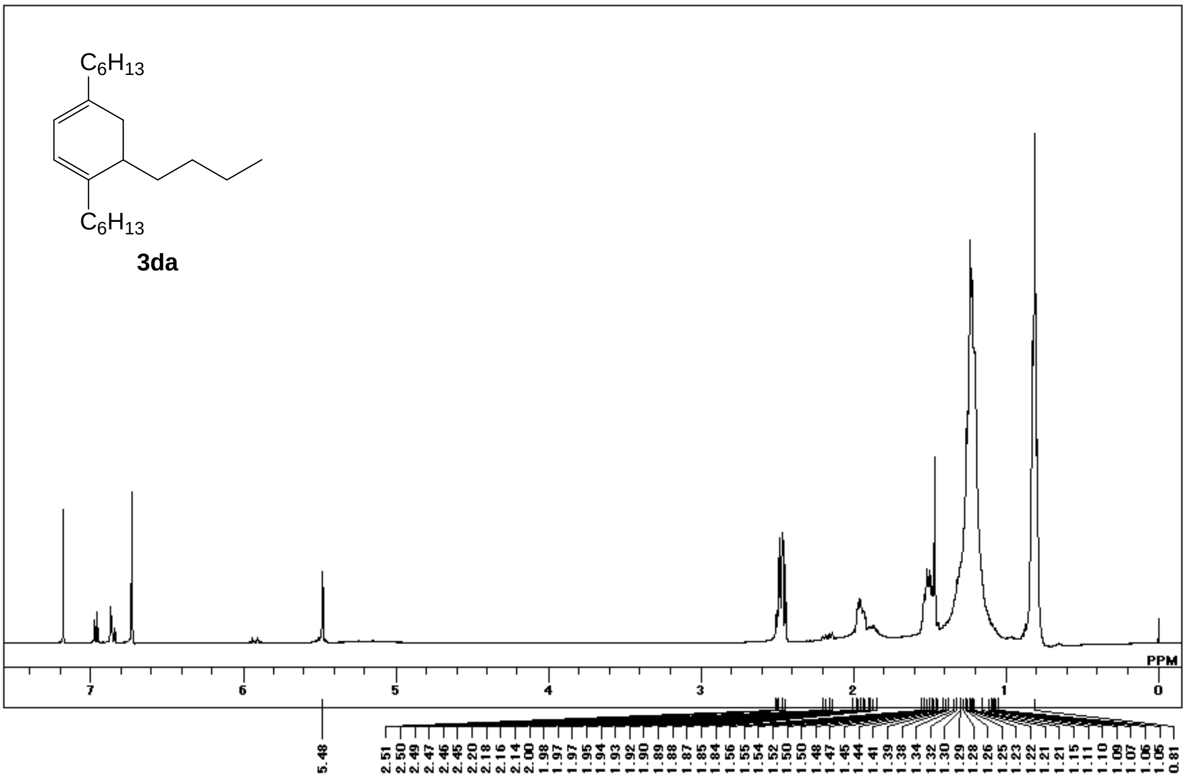
NOESY





C_6H_{13}
C_6H_{13}
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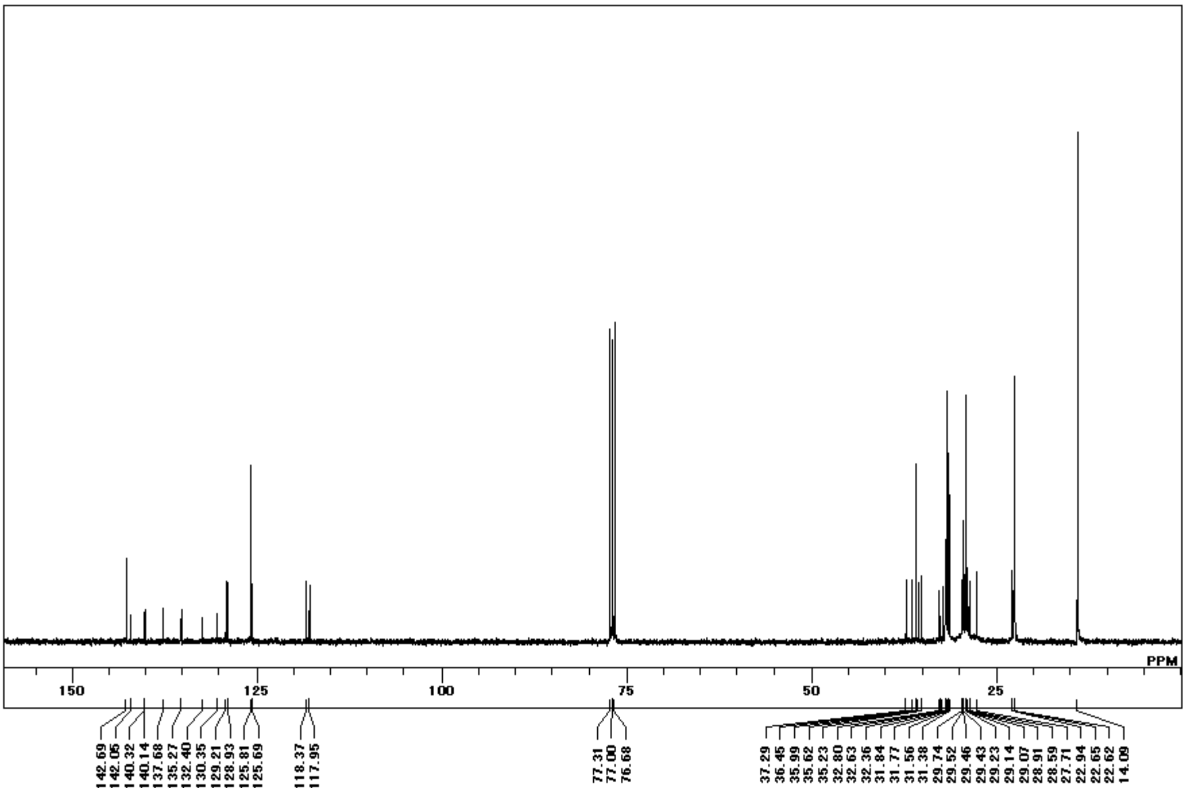

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77.31 77.00 76.68
37.29 36.45 35.99 35.62 35.23 32.80 32.63 32.36 31.84 31.77 31.56 31.38 29.74 29.52 29.46 29.43 29.23 29.14 29.07 28.91 28.59 27.71 22.94 22.65 22.62 14.09

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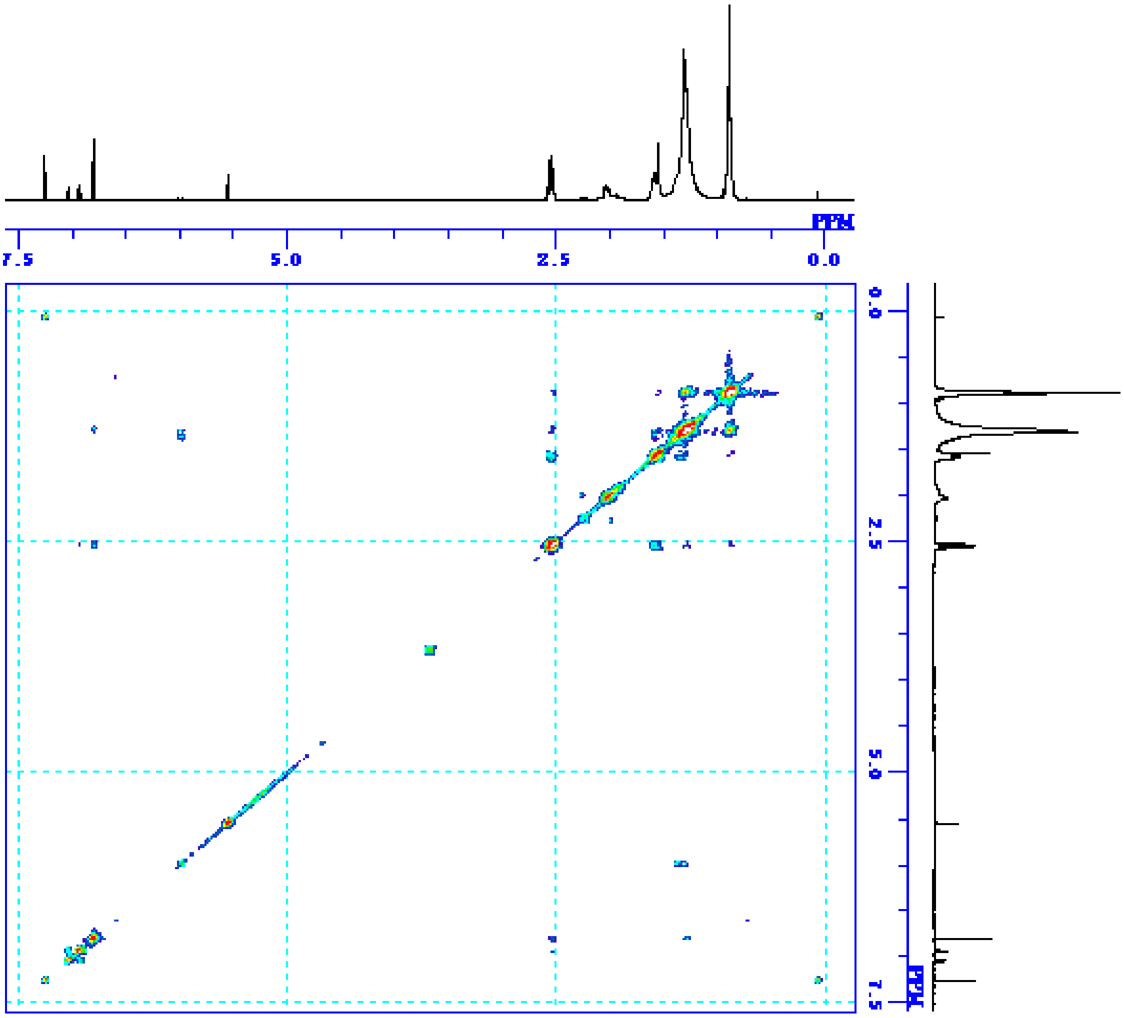

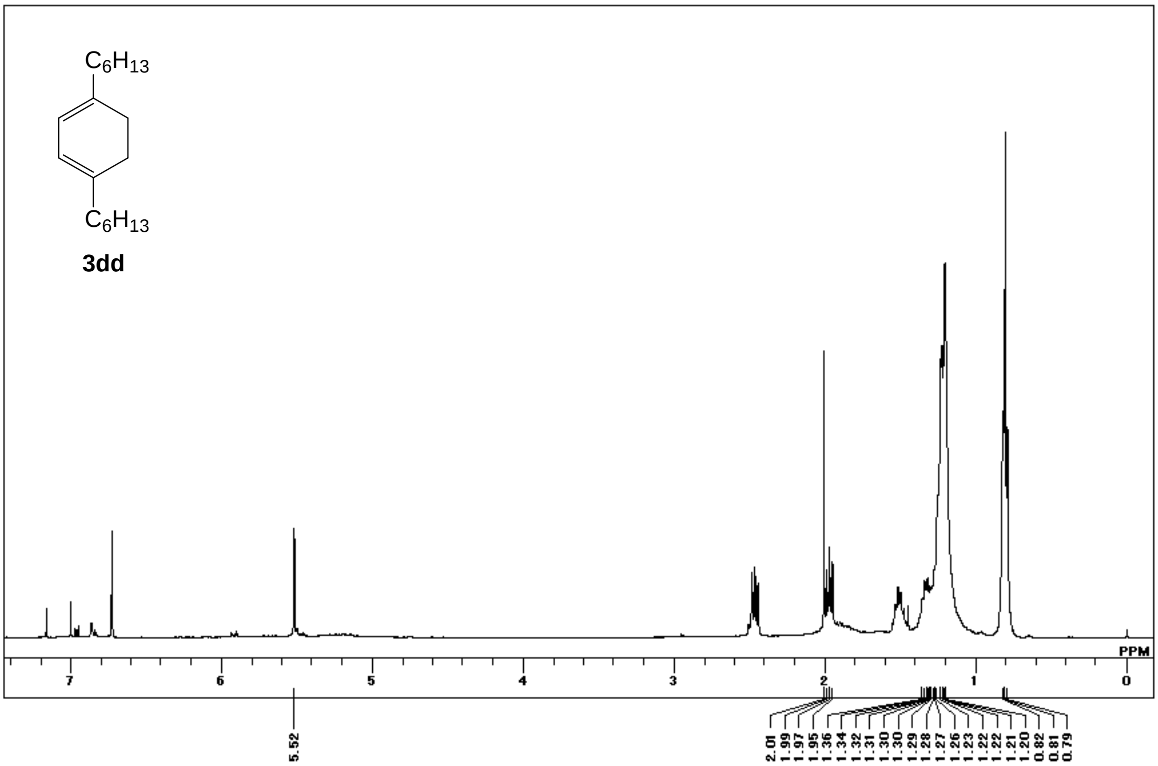

C6H13
C6H13
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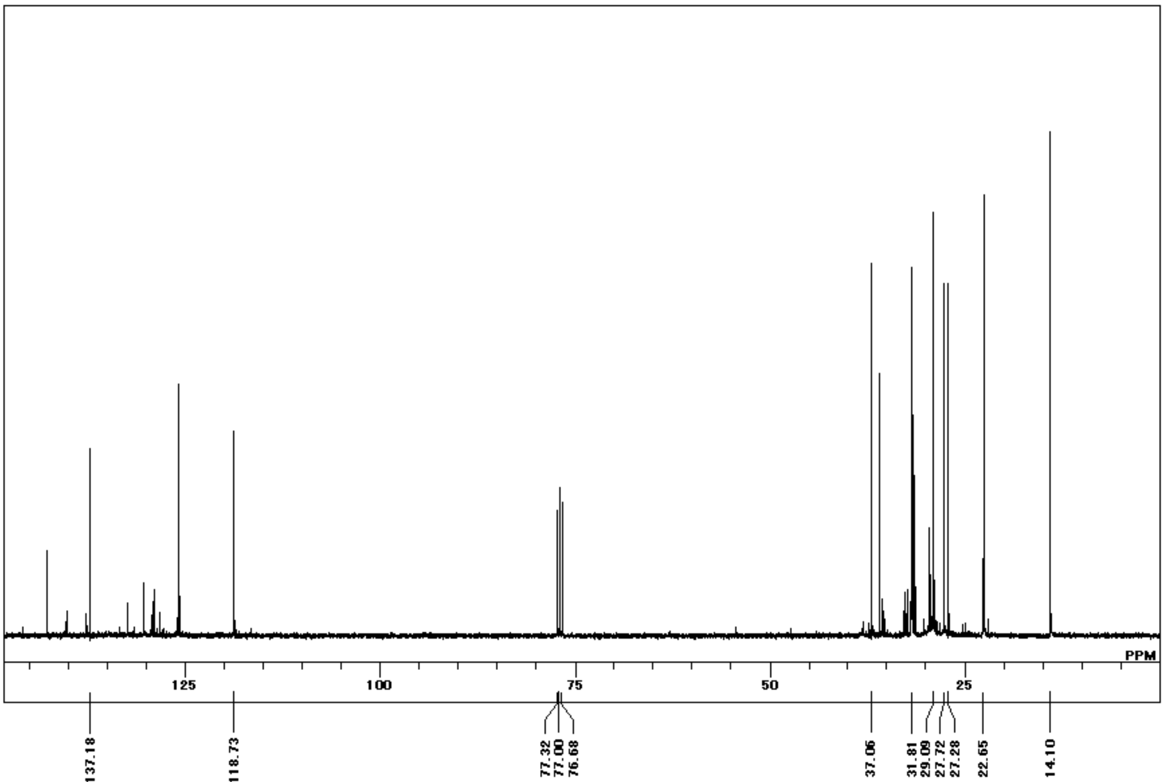

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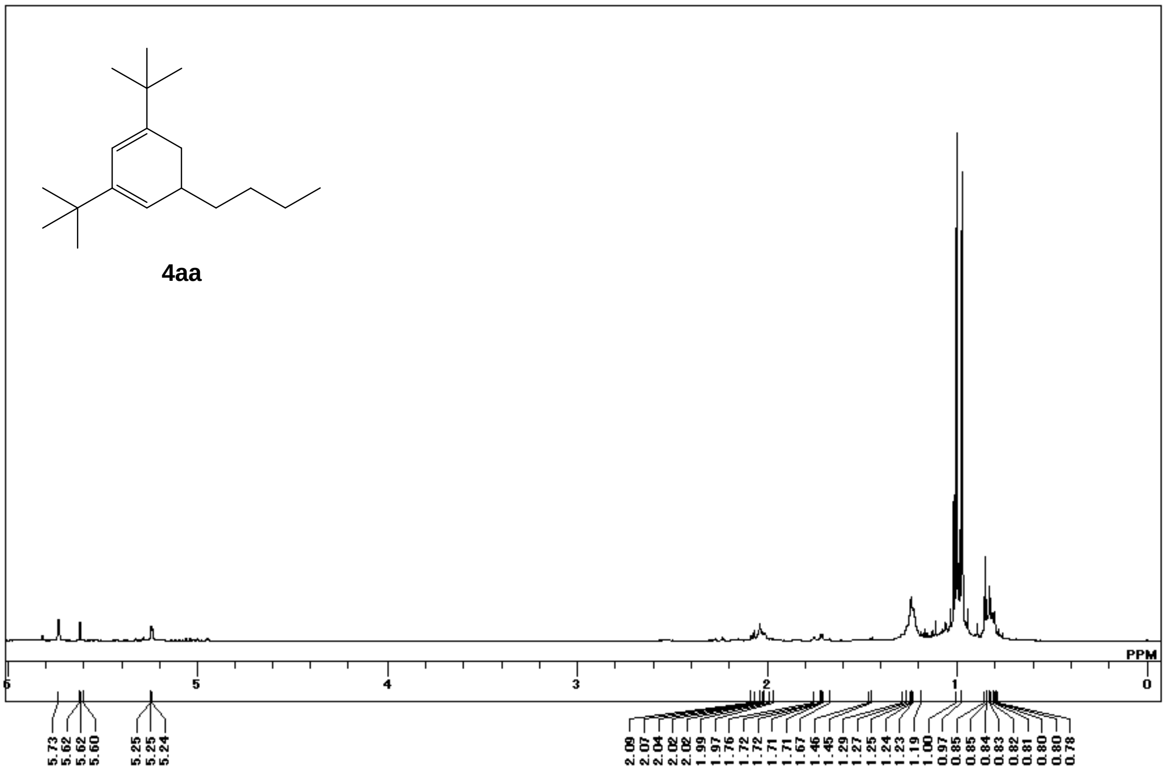
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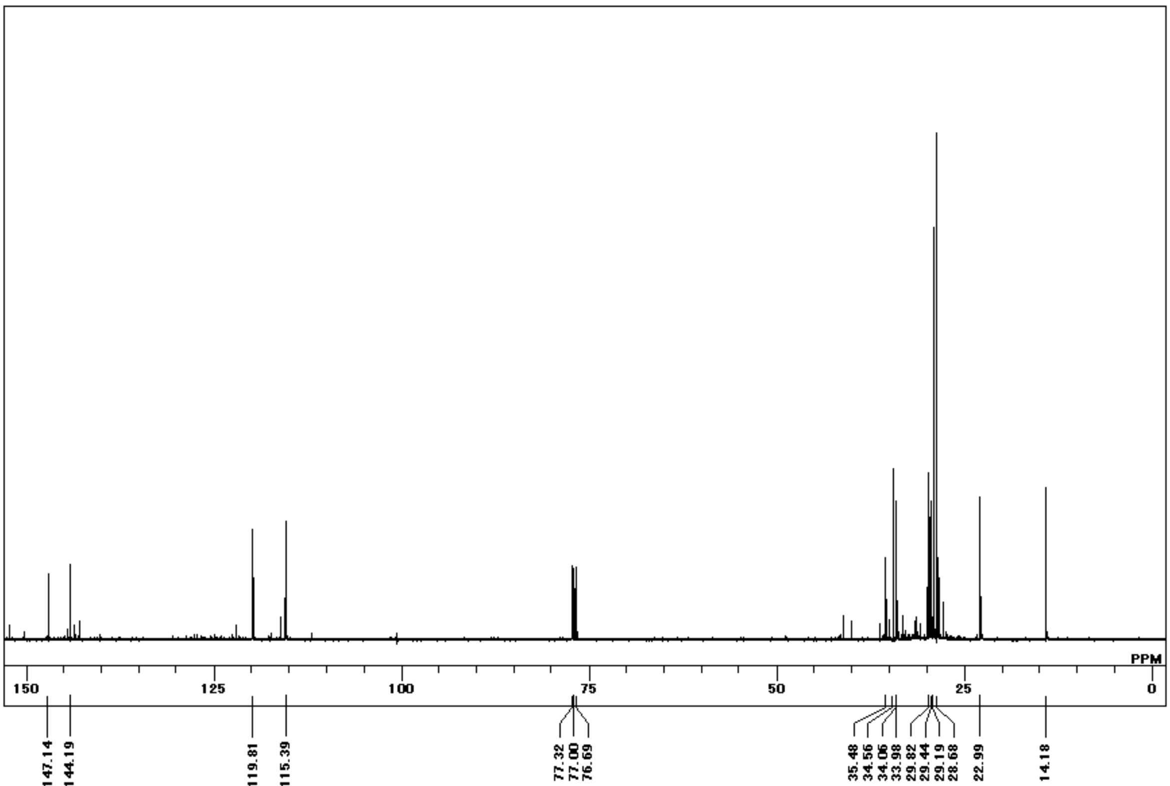
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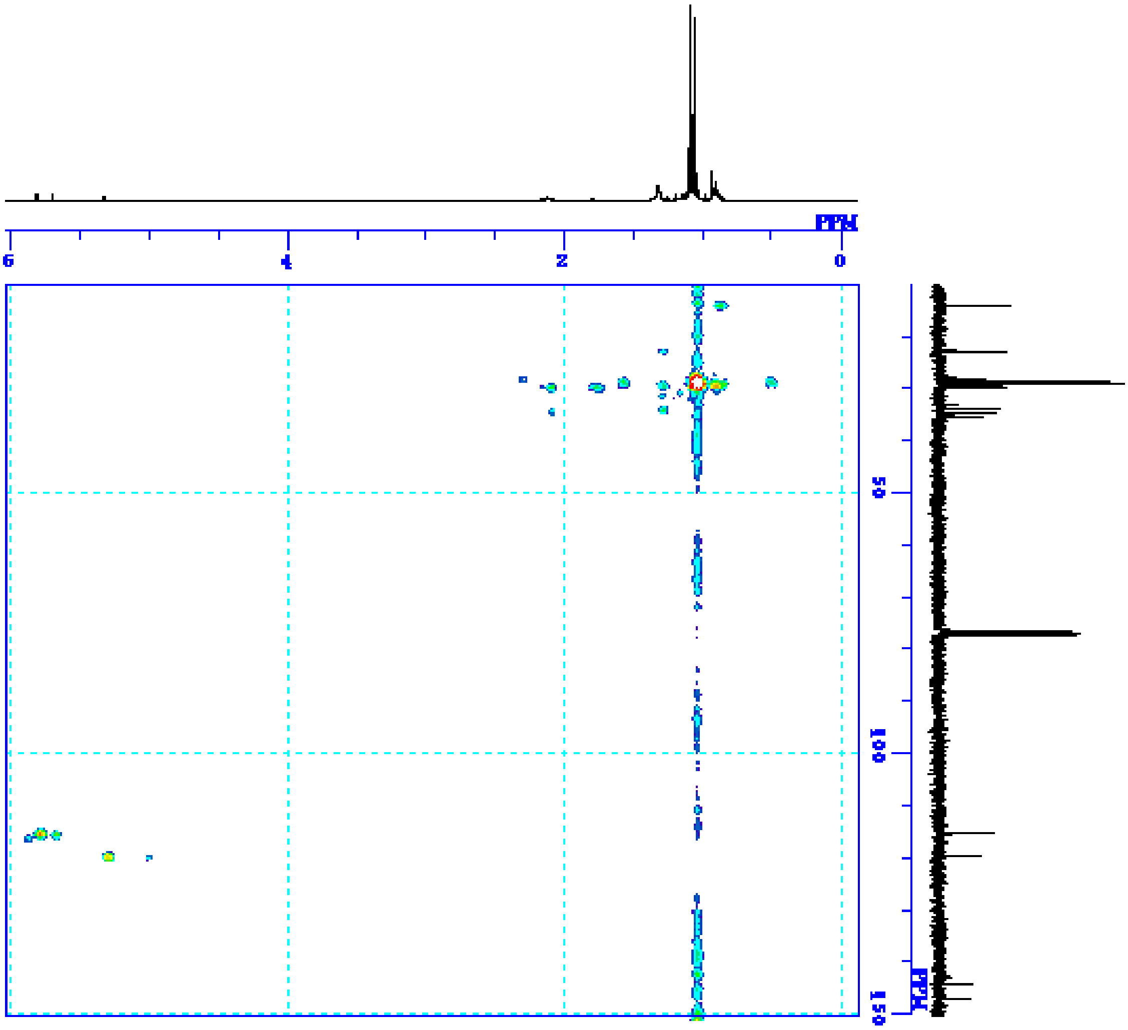
HMQC



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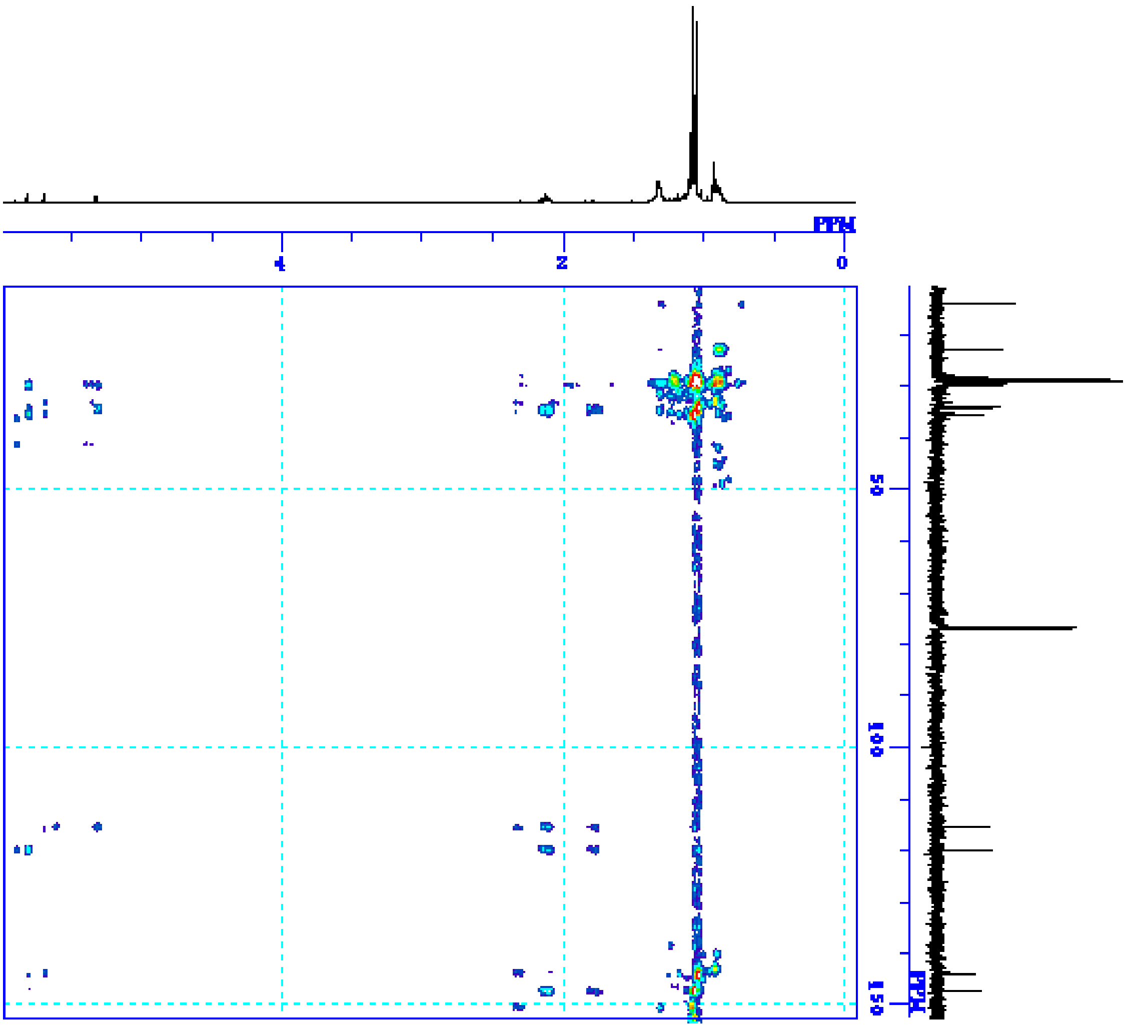

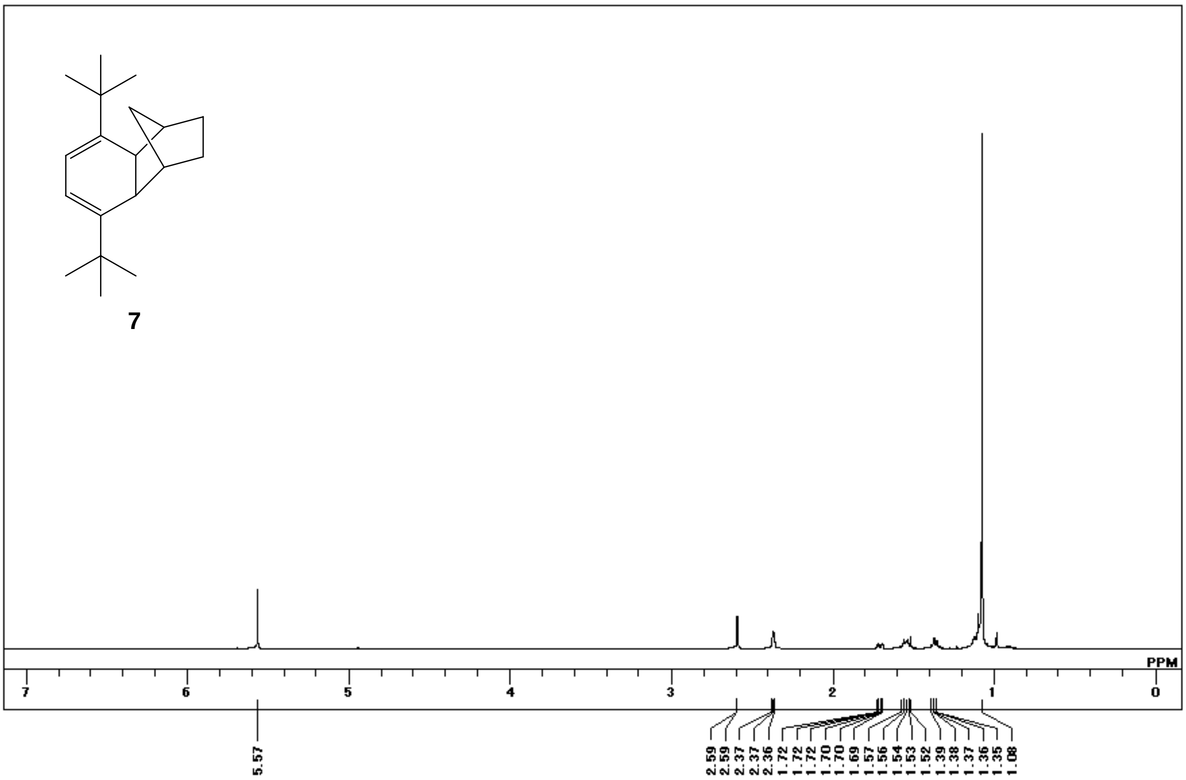
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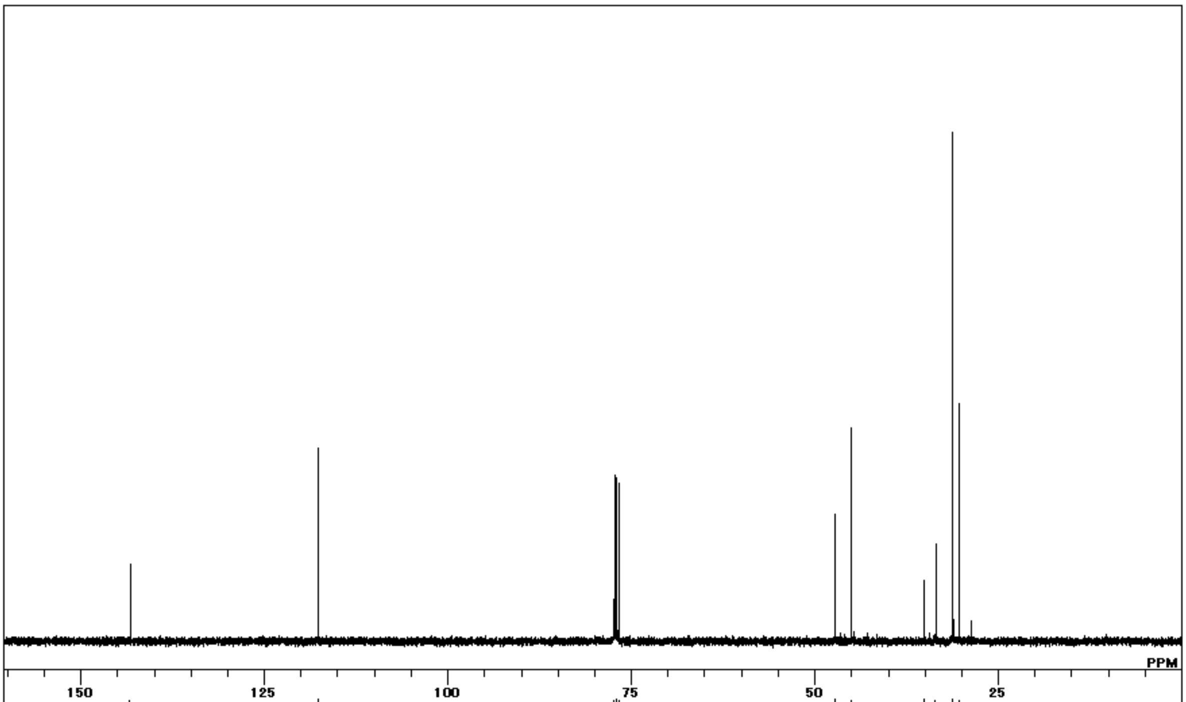
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