Supporting Information

Selective Synthesis of Allylated Oxime Ethers and Nitrones Based on Palladium-Catalyzed Allylic Substitution of Oximes

Hideto Miyabe, Kazumasa Yoshida, Valluru Krishna Reddy, Akira Matsumura and Yoshiji Takemoto*

Graduate School of Pharmaceutical Sciences, Kyoto University, Yoshida, Sakyo-ku, Kyoto 606-8501, Japan.

FAX: +81-75-753-4569; E-mail: takemoto@pharm.kyoto-u.ac.jp

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1. Experimental procedure

General. Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded at 500 MHz, and at 125 MHz, respectively. IR spectra were recorded using FTIR apparatus. Mass spectra were obtained by EI, CI or FAB methods. Preparative TLC separations were carried out on precoated silica gel plates (E. Merck $60F_{254}$). *E*-Oximes were employed, after the separation of *E*- and *Z*-isomers prepared from corresponding aldehydes and $HONH_2 \cdot HCl$. Regarding the configurations of nitrones, *Z*-nitrone (major isomer) and *E*-nitrone (minor isomer) were obtained in all cases. These *E*- and *Z*-isomers were easily separated by preparative TLC.

General procedure for palladium (0)-catalyzed reaction of 1A with carbonate 2a. A mixture of oxime **1A** (60.6 mg, 0.50 mmol), allylic carbonate **2a** (144 mg, 0.75 mmol), and $Pd(PPh_3)_4$ (23.1 mg, 0.020 mmol) in the solvent (2.5 mL) shown in Table 1 was stirred under argon atmosphere at 20 °C. After the reaction was completed, the reaction mixture was concentrated at reduced pressure. The ratio of products and combined yield were determined after rough purification by column chromatography (hexame:AcOEt=1:1). Purification of the residue by preparative TLC (hexane:AcOEt=25:1, 2-fold development) afforded the products **3Aa-5Aa**.

General procedure for palladium (0)-catalyzed reaction of 1A-G with acetates in the presence of Et_2Zn. To a solution of oxime **1A-G** (0.50 mmol) in THF (1.0 mL) was added Et_2Zn (1.0 M in hexane, 0.50 mL, 0.50 mmol) under argon atmosphere at 20 °C. After being stirred at the same temperature for 10 min, a solution of allylic acetate **6a-i** (0.75 mmol) and $Pd(PPh_3)_4$ (46.2 mg, 0.040 mmol) in THF (1.0 mL) was added to the reaction

mixture at 20 °C. After the reaction was completed, the reaction mixture was diluted with saturated aqueous potassium sodium (+)-tartrate and then extracted with AcOEt. The organic phase was dried over $MgSO_4$ and concentrated at reduced pressure. Purification of the residue by preparative TLC (hexane:AcOEt=25:1, 2-fold development) afforded the product **3Aa-Ga**.

General procedure for palladium (0)-catalyzed reaction of 1A-G with acetates in the presence of K_2CO_3. A mixture of oxime **1A-G** (0.50 mmol), allylic acetate **6a-f** (0.75 mmol), $Pd(PPh_3)_4$ (34.7 mg, 0.030 mmol) and K_2CO_3 (69 mg, 0.50 mmol) in CH_2Cl_2 (1.0 mL) was stirred under argon atmosphere at 20 °C. After the reaction was completed, the reaction mixture was diluted with water and then extracted with AcOEt. The organic phase was dried over $MgSO_4$ and concentrated at reduced pressure. Purification of the residue by preparative TLC (hexane:AcOEt=25:1, 2-fold development) afforded the product **3Aa-Ga**.

General procedure for palladium (II)-catalyzed reaction of 1A and 1E with acetates. A mixture of oxime **1A** or **1E** (0.50 mmol), allylic acetate **6a-j** or **7** (0.75 mmol), $Pd(cod)Cl_2$ (14.3 mg, 0.050 mmol) was stirred under argon atmosphere at 90 °C. After the reaction was completed, purification of the reaction mixture by preparative TLC (hexane:AcOEt=5:1, 2-fold development) afforded the product **4Aa-Ga**.

General procedure for palladium (II)-catalyzed reaction of 1A with 7 using ligand 9. A mixture of oxime **1A** (33.5 mg, 0.277 mmol), allylic acetate **7** (105 mg, 0.415 mmol), ligand **9** (11.0 mg, 0.0332 mmol), $Pd(cod)Cl_2$ (7.9 mg, 0.0277 mmol) was stirred under argon atmosphere at 30 °C. After the reaction was stirred for 24 h, purification of the reaction mixture by preparative TLC (hexane:AcOEt=5:1, 2-fold development) afforded the product **8** (59.1 mg, 68% yield). Enantioselectivity of nitrone **8** was determined by HPLC analysis using Chiralcel AD-H (hexane/2-propanol=80/20, 0.5 ml/min, 254 nm, t_r = 57.1 and 63.2 min.

2. Characterization data of all obtained compounds

(*E*)-*O*-(3-Phenylprop-2-enyl)benzaldehyde oxime (3Aa).[1)] A colorless oil. IR ($CHCl_3$) 2926, 1494, 1448 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.13 (1H, s), 7.63-7.48 (2H, m), 7.46-7.16 (8H, m), 6.67 (1H, d, *J*=15.9 Hz), 6.42 (1H, dt, *J*=15.9, 6.2 Hz), 4.83 (2H, d, *J*=6.2 Hz). ^{13}C NMR ($CDCl_3$) δ 148.9, 136.6, 133.5, 132.2, 129.8, 128.7, 128.5, 127.8, 127.1, 126.6, 125.1, 74.9. MS (EI^+) m/z: 237 (M^+, 2), 117 (100). HRMS calcd for $C_{16}H_{15}NO$: 237.1154, Found: 237.1155.

(*E*)-*O*-[3-(4-Trifluoromethylphenyl)prop-2-enyl]benzaldehyde oxime (3Ab). A colorless crystal. mp 55-58 °C (hexane). IR ($CHCl_3$) 2926, 1492, 1447, 1415 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.15 (1H, s), 7.65-7.55 (4H, m), 7.49 (2H, d, *J*=7.9 Hz), 7.40-7.36 (3H, m), 6.71 (1H, d, *J*=16.2 Hz), 6.50 (1H, dt, *J*=16.2, 6.1 Hz), 4.86 (2H, d, *J*=6.1 Hz). ^{13}C NMR ($CDCl_3$) δ 149.2, 140.2, 132.1, 131.6, 130.0, 129.5 (q, *J*=32 Hz), 128.7, 128.1, 127.1, 126.7, 125.5 (q,

J=4.1 Hz), 124.2 (q, *J*=272 Hz), 74.4. MS (EI^+) m/z: 305 (M^+, 2), 185 (100). HRMS calcd for $C_{17}H_{14}F_3NO$: 305.1027, Found: 305.1025. Anal. Calcd for $C_{17}H_{14}F_3NO$: C, 66.88; H, 4.62; N, 4.59; F, 18.67. Found: C, 66.65; H, 4.73; N, 4.44; F, 18.96.

(*E*)-*O*-[3-(4-Methoxyphenyl)prop-2-enyl]benzaldehyde oxime (3Ac). A colorless oil. IR ($CHCl_3$) 2935, 1512, 1464, 1445 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.14 (1H, s), 7.63-7.57 (2H, m), 7.40-7.28 (5H, m), 6.84 (2H, d, *J*=8.6 Hz), 6.63 (1H, d, *J*=15.9 Hz), 6.29 (1H, dt, *J*=15.9, 6.5 Hz), 4.81 (2H, d, *J* = 6.5 Hz), 3.78 (3H, s). ^{13}C NMR ($CDCl_3$) δ 159.4, 148.8, 133.3, 132.3, 129.8, 129.4, 128.7, 127.8, 127.1, 122.7, 113.9, 75.1, 55.2. MS (EI^+) m/z: 267 (M^+, 6), 147 (100). HRMS calcd for $C_{17}H_{17}NO_2$: 267.1259, Found: 267.1253.

(*E*)-*O*-[3-(1-Naphtyl)prop-2-enyl]benzaldehyde oxime (3Ad). A colorless oil. IR ($CHCl_3$) 2925, 1507, 1493, 1446 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.17 (1H, s), 8.12 (1H, d, *J*=7.9 Hz), 7.82 (1H, d, *J*=6.4 Hz), 7.75 (1H, d, *J*=8.2 Hz), 7.68-7.55 (3H, m), 7.51-7.30 (7H, m), 6.44 (1H, dt, *J*=15.8, 6.1 Hz), 4.94 (2H, d, *J*=6.1 Hz), 3.78 (3H, s). ^{13}C NMR ($CDCl_3$) δ 149.0, 134.5, 133.6, 132.3, 131.2, 130.6, 129.9, 128.7, 128.5, 128.4, 128.1, 127.1, 126.1, 125.8, 125.6, 124.1, 123.8, 75.0. MS (EI^+) m/z: 287 (M^+, 10), 167 (100). HRMS calcd for $C_{20}H_{17}NO$: 287.1310, Found: 287.1311.

(*E*)-*O*-[3-(2-Naphtyl)prop-2-enyl]benzaldehyde oxime (3Ae). A colorless crystal. mp 70-73 °C (hexane). IR ($CHCl_3$) 2927, 1506, 1446 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.16 (1H, s), 7.83-7.75 (4H, m), 7.66-7.58 (3H, m), 7.49-7.34 (5H, m), 6.85 (1H, d, *J*=15.9 Hz), 6.56 (1H, dt, *J*=15.9, 6.1 Hz), 4.90 (2H, d, *J*=6.1 Hz). ^{13}C NMR ($CDCl_3$) δ 149.0, 134.1, 133.6, 133.1, 132.2, 129.9, 128.7 (2C), 128.2, 128.0, 127.7, 127.1, 126.7, 126.3, 126.0, 125.5, 123.6, 75.0. MS (EI^+) m/z: 287 (M^+, 6), 167 (100). HRMS calcd for $C_{20}H_{17}NO$: 287.1310, Found: 287.1312. Anal. Calcd for $C_{20}H_{17}NO$: C, 83.59; H, 5.96; N, 4.87. Found: C, 83.72; H, 5.97; N, 4.83.

(*E*)-*O*-[3-(4-Fluorophenyl)prop-2-enyl]benzaldehyde oxime (3Af). A colorless crystal. mp 54-57 °C (hexane). IR ($CHCl_3$) 2926, 1509, 1447, 1414 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.14 (1H, s), 7.63-7.57 (2H, m), 7.42-7.34 (5H, m), 7.05-6.97 (2H, m), 6.65 (1H, d, *J*=15.8 Hz), 6.34 (1H, dt, *J*=15.8, 6.1 Hz), 4.82 (2H, d, *J*=6.1 Hz). ^{13}C NMR ($CDCl_3$) δ 162.5 (d, *J*=247 Hz), 149.0, 132.8, 132.3, 132.2, 129.8, 128.7, 128.1 (d, *J*=8.3 Hz), 127.1, 124.9, 115.4 (d, *J*=21.7 Hz), 74.7. MS (EI^+) m/z: 255 (M^+, 1), 135 (100). HRMS calcd for $C_{16}H_{14}FNO$: 255.1059, Found: 255.1062. Anal. Calcd for $C_{16}H_{14}FNO$: C, 75.28; H, 5.53; N, 5.49; F, 7.44. Found: C, 75.33; H, 5.72; N, 5.45; F, 7.46.

(*E*)-*O*-[3-(3-Chlorophenyl)prop-2-enyl]benzaldehyde oxime (3Ag). A colorless oil. IR ($CHCl_3$) 2925, 1477, 1447, 1425 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.13 (1H, s), 7.63-7.56 (2H, m), 7.43-7.33 (4H, m), 7.28-7.17 (3H, m), 6.59 (1H, d, *J*=15.9 Hz), 6.42 (1H, dt, *J*=15.9, 5.5 Hz), 4.82 (2H, d, *J*=5.5 Hz). ^{13}C NMR ($CDCl_3$) δ 149.1, 138.6, 134.5, 132.2, 131.8, 129.9, 129.8, 128.7, 127.7, 127.1, 126.9, 126.5, 124.8, 74.5. MS (EI^+) m/z: 271 (M^+, 3), 151 (100). HRMS calcd for $C_{16}H_{14}ClNO$: 271.0764, Found: 271.0764.

(*E*)-*O*-[3-(4-Chlorophenyl)prop-2-enyl]benzaldehyde oxime (3Ah). A colorless crystal. mp 68-71 ^{o}C (hexane). IR ($CHCl_3$) 2926, 1491, 1448, 1405 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.14 (1H, s), 7.23-7.57 (2H, m), 7.40-7.27 (7H, m), 6.64 (1H, d, *J*=15.9 Hz), 6.40 (1H, dt, *J*=15.9, 6.2 Hz), 4.83 (2H, d, *J*=6.2 Hz). ^{13}C NMR ($CDCl_3$) δ 149.1, 135.1, 133.4, 132.2, 132.1, 129.9, 128.7 (2C), 127.8, 127.1, 125.9, 74.6. MS (EI^{+}) m/z: 271 (M^{+}, 2), 151 (100). HRMS calcd for $C_{16}H_{14}ClNO$: 271.0764, Found: 271.0769. Anal. Calcd for $C_{16}H_{14}ClNO$: C, 70.72; H, 5.19; N, 5.15; Cl, 13.05. Found: C, 70.78; H, 5.32; N, 5.18; Cl, 13.25.

(*E*)-*O*-[3-(4-Methylphenyl)prop-2-enyl]benzaldehyde oxime (3Ai). A colorless crystal. mp 70-72 ^{o}C (hexane). IR ($CHCl_3$) 2925, 1512, 1493, 1448, 1414 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.14 (1H, s), 7.63-7.56 (2H, m), 7.40-7.34 (3H, m), 7.31 (2H, d, *J*=7.9 Hz), 7.12 (2H, d, *J*=7.9 Hz), 6.65 (1H, d, *J*=15.9 Hz), 6.38 (1H, dt, *J*=15.9, 6.4 Hz), 4.83 (2H, d, *J*=6.4 Hz), 2.34 (3H, s). ^{13}C NMR ($CDCl_3$) δ 148.9, 137.7, 133.9, 133.5, 132.3, 129.8, 129.3, 128.7, 127.1, 126.5, 124.0, 75.0, 21.1. MS (EI^{+}) m/z: 251 (M^{+}, 1), 131 (100). HRMS calcd for $C_{17}H_{17}NO$: 251.1310, Found: 251.1314. Anal. Calcd for $C_{17}H_{17}NO$: C, 81.24; H, 6.82; N, 5.57. Found: C, 81.54; H, 6.92; N, 5.53.

(*E*)-*O*-(3-Phenylprop-2-enyl)-4-trifluoromethylbenzaldehyde oxime (3Ba). A colorless crystal. mp 90-93 ^{o}C (hexane). IR ($CHCl_3$) 2928, 1494, 1450, 1413 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.15 (1H, s), 7.71 (2H, d, *J*=8.1 Hz), 7.60 (2H, d, *J*=8.1 Hz), 7.42 (2H, d, *J*=7.6 Hz), 7.36-7.23 (3H, m), 6.70 (1H, d, *J*=15.9 Hz), 6.42 (1H, dt, *J*=15.9, 6.4 Hz), 4.86 (2H, d, *J*=6.4 Hz). ^{13}C NMR ($CDCl_3$) δ 147.4, 136.5, 135.7, 133.8, 131.4 (*J*=32.1 Hz), 128.6, 128.0, 127.2, 126.6, 125.6, 124.8, 124.0 (*J*=272 Hz), 75.3. MS (EI^{+}) m/z: 305 (M^{+}, 1), 117 (100). HRMS calcd for $C_{17}H_{14}F_3NO$: 305.1027, Found: 305.1023. Anal. Calcd for $C_{17}H_{14}F_3NO$: C, 66.88; H, 4.62; N, 4.59; F, 18.67. Found: C, 67.16; H, 4.81; N, 4.59; F, 18.67.

(*E*)-*O*-(3-Phenylprop-2-enyl)-4-methoxybenzaldehyde oxime (3Ca). A colorless crystal. mp 72-75 ^{o}C (hexane). IR ($CHCl_3$) 2935, 1513, 1463, 1419 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.09 (1H, s), 7.53 (2H, d, *J*=8.6 Hz), 7.40 (2H, d, *J*=7.5 Hz), 7.31 (2H, d, *J*=7.5 Hz), 7.23 (1H, m), 6.88 (2H, d, *J*=8.6 Hz), 6.69 (1H, d, *J*=15.9 Hz), 6.43 (1H, dt, *J*=15.9, 6.2 Hz), 4.81 (2H, d, *J*=6.2 Hz), 3.82 (3H, s). ^{13}C NMR ($CDCl_3$) δ 161.0, 148.6, 136.7, 133.3, 128.5 (2C), 127.8, 126.6, 125.3, 124.9, 114.1, 74.7, 55.2. MS (EI^{+}) m/z: 267 (M^{+}, 10), 117 (100). HRMS calcd for $C_{17}H_{17}NO_2$: 267.1259, Found: 267.1260. Anal. Calcd for $C_{17}H_{17}NO_2$: C, 76.38; H, 6.41; N, 5.24. Found: C, 76.52; H, 6.39; N, 5.22.

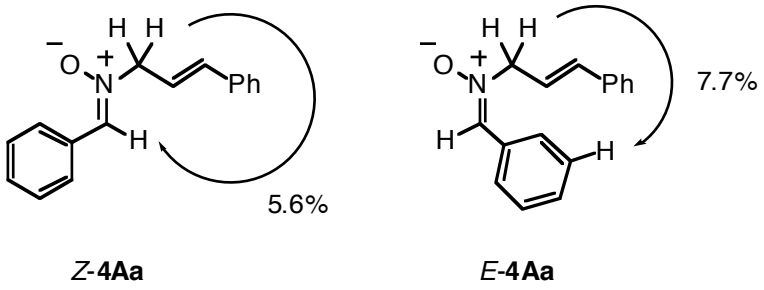
(*E*)-*O*-(3-Phenylprop-2-enyl)-2-pyridinecarboxaldehyde oxime (3Da). A colorless oil. IR ($CHCl_3$) 2928, 1494, 1470, 1436 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.61 (1H, d, *J*=4.0 Hz), 8.23 (1H, s), 7.80 (1H, d, *J*=7.9 Hz), 7.67 (1H, m), 7.41 (2H, d, *J*=7.6 Hz), 7.32 (2H, d, *J*=7.6 Hz), 7.27-7.20 (2H, m), 6.69 (1H, d, *J*=15.9 Hz), 6.42 (1H, dt, *J*=15.9, 6.4 Hz), 4.89 (2H, d, *J*=6.4 Hz). ^{13}C NMR ($CDCl_3$) δ 151.7, 149.7, 149.6, 136.5, 136.4, 133.7, 128.6, 127.9, 126.6, 124.8, 124.0, 121.1, 75.4. MS (EI^{+}) m/z: 238 (M^{+}, 2), 117 (100). HRMS calcd for $C_{15}H_{14}N_2O$: 238.1106, Found: 238.1114.

***O*-(3-Phenylprop-2-enyl)benzophenone oxime (3Ea).**[2] A colorless crystal. mp 66-68 °C (hexane). IR ($CHCl_3$) 2924, 1494, 1444 cm^{-1}. ^1H NMR ($CDCl_3$) δ 7.55-7.20 (15H, m), 6.61 (1H, d, *J*=16.1 Hz), 6.41 (1H, dt, *J*=16.1, 6.1 Hz), 4.86 (2H, d, *J*=6.1 Hz). ^{13}C NMR ($CDCl_3$) δ 157.0, 136.8, 136.6, 133.4, 132.9, 129.4, 129.3, 128.9, 128.6, 128.2, 128.1, 128.0, 127.7, 126.6, 125.8, 75.2. MS (EI^+) m/z: 313 (M^+, 4), 117 (100). HRMS calcd for $C_{22}H_{19}NO$: 313.1467, Found: 313.1469. Anal. Calcd for $C_{22}H_{19}NO$: C, 84.31; H, 6.11; N, 4.47. Found: C, 84.44; H, 6.16; N, 4.43.

***O*-(3-Phenylprop-2-enyl)cyclohexanone oxime (3Fa).**[3] A colorless oil. IR ($CHCl_3$) 2938, 1495, 1449 cm^{-1}. ^1H NMR ($CDCl_3$) δ 7.39 (2H, d, *J*=7.6 Hz), 7.30 (2H, d, *J*=7.6 Hz), 7.22 (1H, br t, *J*=7.6 Hz), 6.62 (1H, d, *J*=15.9 Hz), 6.37 (1H, dt, *J*=15.9, 6.0 Hz), 4.68 (2H, d, *J*=6.0 Hz), 2.50 (2H, br t, *J*=6.1 Hz), 2.21 (2H, br t, *J*=6.1 Hz), 1.73-1.52 (6H, m). ^{13}C NMR ($CDCl_3$) δ 160.5, 136.9, 132.5, 128.5, 127.6, 126.5, 125.9, 73.8, 32.1, 26.9, 25.7, 25.6, 25.3. MS (EI^+) m/z: 229 (M^+, 2), 117 (100). HRMS calcd for $C_{15}H_{19}NO$: 229.1467, Found: 229.1467.

Methyl (*E*)-2-[(3-Phenylprop-2-enyloxy)imino]ethanate (3Ga). A colorless oil. IR ($CHCl_3$) 2954, 1727, 1494, 1442 cm^{-1}. ^1H NMR ($CDCl_3$) δ 7.52 (1H, s), 7.40 (2H, d, *J*=7.3 Hz), 7.32 (2H, t, *J*=7.3 Hz), 7.26 (1H, br t, *J*=7.3 Hz), 6.67 (1H, d, *J*=15.9 Hz), 6.36 (1H, dt, *J*=15.9, 6.6 Hz), 4.91 (2H, d, *J*=6.6 Hz), 3.86 (3H, s). ^{13}C NMR ($CDCl_3$) δ 162.4, 140.8, 136.2, 134.7, 128.6, 128.1, 126.7, 123.4, 76.6, 52.4. MS (EI^+) m/z: 219 (M^+, 6), 117 (100). HRMS calcd for $C_{12}H_{13}NO_3$: 219.0895, Found: 219.0897.

Benzylidene(3-phenylprop-2-enyl)amine *N*-oxide (4Aa). Minor isomer (*E*-isomer): A colorless solid. IR ($CHCl_3$) 2993, 1587, 1493, 1451 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.18-8.12 (2H, m), 7.39-7.19 (9H, m), 6.80 (1H, d, *J*=11.6 Hz), 6.04 (1H, dt, *J*=11.6, 7.0 Hz), 4.75 (2H, d, *J*=7.0 Hz). ^{13}C NMR ($CDCl_3$) δ 135.7, 134.9, 134.1, 130.5, 130.4, 128.7, 128.6, 128.5, 127.9, 123.4, 65.0. One peak of ^{13}C NMR was missing due to overlap. MS (EI^+) m/z: 237 (M^+, 5), 117 (100). HRMS calcd for $C_{16}H_{15}NO$: 237.1154, Found: 237.1159. Major isomer (*Z*-isomer): A colorless solid. IR ($CHCl_3$) 2989, 1585, 1494, 1450 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.20-8.14 (2H, m), 7.38-7.16 (9H, m), 6.67 (1H, d, *J*=15.9 Hz), 6.45 (1H, dt, *J*=15.9, 6.1 Hz), 4.62 (2H, d, *J*=6.1 Hz). ^{13}C NMR ($CDCl_3$) δ 136.6, 135.7, 134.0, 130.5, 130.4, 128.7, 128.6, 128.5 (2C), 126.8, 121.0, 69.4. MS (EI^+) m/z: 237 (M^+, 8), 117 (100). HRMS calcd for $C_{16}H_{15}NO$: 237.1154, Found: 237.1164.



Z-**4Aa** *E*-**4Aa**

FIGURE 1. The nOe studies of *Z*- and *E*-nitrones 4Aa

Benzylidene[3-(4-trifluoromethylphenyl)prop-2-enyl]amine *N*-oxide (4Ab). Minor isomer: A colorless solid. IR ($CHCl_3$) 1582, 1452 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.65 (2H, d, *J*=8.2 Hz), 7.47 (2H, d, *J*=8.2 Hz), 7.43 (3H, m), 7.38 (1H, s), 6.89 (1H, d, *J*=11.3 Hz), 6.21 (1H, dt, *J*=11.3, 7.0 Hz), 4.79 (2H, d, *J*=7.0 Hz). ^{13}C NMR ($CDCl_3$) δ 139.2, 134.4, 133.6, 130.7, 130.3, 129.9 (q, *J*=33.1 Hz), 129.0, 128.6 (2C), 125.6 (q, *J*=3.1 Hz), 125.5, 124.0 (q, *J*=272 Hz), 64.7. MS (FAB^+) m/z: 306 ($M+H^+$, 51), 185 (100). HRMS calcd for $C_{17}H_{15}NOF_3$ ($M+H^+$): 306.1105, Found: 306.1101. Major isomer: A colorless solid. IR ($CHCl_3$) 1585, 1452 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.24 (2H, m), 7.59 (2H, d, *J*=8.2 Hz), 7.53 (2H, d, *J*=8.2 Hz), 7.49-7.41 (4H, m), 6.80 (1H, d, *J*=15.9 Hz), 6.65 (1H, dt, *J*=15.9, 5.8 Hz), 4.74 (2H, d, *J*=5.8 Hz). ^{13}C NMR ($CDCl_3$) δ 139.2, 134.7, 134.3, 130.6, 130.3, 130.1 (q, *J*=32.1 Hz), 128.6, 128.5, 126.9, 125.6 (q, *J*=3.1 Hz), 124.1 (q, *J*=272 Hz), 124.0, 69.1. MS (FAB^+) m/z: 306 ($M+H^+$, 55), 185 (100). HRMS calcd for $C_{17}H_{15}NOF_3$ ($M+H^+$): 306.1105, Found: 306.1107.

Benzylidene[3-(1-naphtyl)prop-2-enyl]amine *N*-oxide (4Ad). Minor isomer: A colorless solid. IR ($CHCl_3$) 1588, 1452 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.17 (2H, m), 7.96 (1H, m), 7.89 (1H, m), 7.85 (1H, d, *J*=8.2 Hz), 7.56-7.38 (7H, m), 7.34 (1H, d, *J*=11.3 Hz), 7.22 (1H, s), 6.43 (1H, dt, *J*=11.3, 7.0 Hz), 4.66 (2H, d, *J*=7.0 Hz). ^{13}C NMR ($CDCl_3$) δ 134.2, 133.6, 133.3, 132.8, 131.6, 130.5, 130.4, 128.6 (2C), 128.5 (2C), 126.5, 126.4, 126.2, 125.6, 125.4, 124.7, 65.2. MS (FAB^+) m/z: 288 ($M+H^+$, 25), 167 (100). HRMS calcd for $C_{20}H_{18}NO$ ($M+H^+$): 288.1388, Found: 188.1393. Major isomer: A colorless solid. IR ($CHCl_3$) 1588, 1451 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.26 (2H, m), 8.11 (1H, d, *J*=8.2 Hz), 7.85 (1H, d, *J*=8.2 Hz), 7.81 (1H, d, *J*=8.2 Hz), 7.66 (1H, d, *J*=7.3 Hz), 7.56-7.39 (8H, m), 6.56 (1H, dt, *J*=15.6, 6.7 Hz), 4.82 (2H, d, *J*=6.7 Hz). ^{13}C NMR ($CDCl_3$) δ 134.2, 133.9, 133.6, 133.3, 131.0, 130.5, 130.4, 128.8, 128.7, 128.6, 128.5, 126.4, 125.9, 125.6, 124.4, 124.1, 123.5, 69.5. MS (FAB^+) m/z: 288 ($M+H^+$, 4.1), 167 (100). HRMS calcd for $C_{20}H_{18}NO$ ($M+H^+$): 288.1388, Found: 188.1382.

Benzylidene[3-(4-chlorophenyl)prop-2-enyl]amine *N*-oxide (4Ah). Minor isomer: A colorless solid. IR ($CHCl_3$) 1589, 1490, 1452 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.46-7.24 (8H, m), 6.81 (1H, d, *J*=11.6 Hz), 6.12 (1H, dt, *J*=11.6, 7.0 Hz), 4.78 (2H, d, *J*=7.0 Hz). ^{13}C NMR ($CDCl_3$) δ 134.3, 134.1, 133.8, 130.6, 130.4, 130.0, 128.8, 128.6 (2C),

124.1, 64.8. One peak of ^{13}C NMR was missing due to overlap. MS (FAB^+) m/z: 272 ($M+H^+$, 55), 151 (100). HRMS calcd for $C_{16}H_{15}NOCl$ ($M+H^+$): 272.0842, Found: 272.0841. Major isomer: A colorless solid. IR ($CHCl_3$) 1590. 1492, 1452 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.47-7.25 (8H, m), 6.70 (1H, d, *J*=15.9 Hz), 6.50 (1H, dt, *J*=15.9, 7.0 Hz), 4.68 (2H, d, *J*=7.0 Hz). ^{13}C NMR ($CDCl_3$) δ 135.1, 134.2, 134.1, 130.5, 130.3, 128.8, 128.6, 128.5, 128.0, 121.8, 69.2. One peak of ^{13}C NMR was missing due to overlap. MS (FAB^+) m/z: 272 ($M+H^+$, 29), 151 (100). HRMS calcd for $C_{16}H_{15}NOCl$ ($M+H^+$): 272.0842, Found: 272.0851.

Benzylidene[3-(4-methylphenyl)prop-2-enyl]amine *N*-oxide (4Ai). Minor isomer: A colorless solid. IR ($CHCl_3$) 1596, 1450 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.48-7.10 (8H, m), 6.83 (1H, d, *J*=11.6 Hz), 6.06 (1H, dt, *J*=11.6, 7.0 Hz), 4.82 (2H, d, *J*=7.0 Hz), 2.37 (3H, s). ^{13}C NMR ($CDCl_3$) δ 137.8, 134.8, 134.0, 132.8, 131.7, 130.5, 129.3, 128.6, 128.5, 122.6, 65.1, 21.2. One peak of ^{13}C NMR was missing due to overlap. MS (FAB^+) m/z: 252 ($M+H^+$, 11), 131 (100). HRMS calcd for $C_{17}H_{18}NO$ ($M+H^+$): 252.1389, Found: 252.1393. Major isomer: A colorless solid. IR ($CHCl_3$) 1586, 1513, 1451 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.45 (1H, s), 7.41 (3H, m), 7.33 (2H, d, *J*=7.9 Hz), 7.15 (2H, d, *J*=7.9 Hz), 6.73 (1H, d, *J*=15.9 Hz), 6.46 (1H, dt, *J*=15.9, 7.0 Hz), 4.69 (2H, d, *J*=7.0 Hz), 2.34 (3H, s). ^{13}C NMR ($CDCl_3$) δ 138.5, 136.7, 133.9, 133.0, 130.5 (2C), 129.4, 128.6, 128.5, 126.7, 119.8, 69.5, 21.2. MS (FAB^+) m/z: 252 ($M+H^+$, 9), 131 (100). HRMS calcd for $C_{17}H_{18}NO$ ($M+H^+$): 252.1389, Found: 252.1386.

Benzylidene(3-cyclohexylprop-2-enyl)amine *N*-oxide (4Aj). Minor isomer: A colorless solid. IR ($CHCl_3$) 1596, 1450 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.47-7.39 (3H, m), 5.75-7.60 (2H, m), 4.61 (2H, d, *J*=6.7 Hz), 2.35 (1H, m), 1.82-1.45 (5H, m), 1.37-1.18 (5H, m). MS (FAB^+) m/z: 244 ($M+H^+$, 67), 81 (100). HRMS calcd for $C_{16}H_{22}NO$ ($M+H^+$): 244.1701, Found: 244.1700. Major isomer: A colorless solid. IR ($CHCl_3$) 2853, 1582, 1450 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.23 (2H, m), 7.45-7.35 (4H, m), 5.84 (1H, dd, *J*=15.6, 6.4 Hz), 5.76 (1H, dt, *J*=15.6, 6.7 Hz), 4.48 (2H, d, *J*=6.7 Hz), 2.05 (1H, m), 1.80-1.62 (5H, m), 1.35-1.05 (5H, m). ^{13}C NMR ($CDCl_3$) δ 144.8, 133.5, 130.5, 130.3, 128.6, 128.5, 119.6, 69.5, 40.4, 32.4, 26.0, 25.8. MS (FAB^+) m/z: 244 ($M+H^+$, 100). HRMS calcd for $C_{16}H_{22}NO$ ($M+H^+$): 244.1701, Found: 244.1710.

Diphenylmethylidene(3-phenylprop-2-enyl)amine *N*-oxide (4Ea). A colorless solid. IR ($CHCl_3$) 1655, 1438 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.03 (2H, m), 7.53-7.47 (3H, m), 7.39-7.24 (10H, m), 6.52 (1H, dt, *J*=15.6, 6.4 Hz), 6.40 (1H, d, *J*=15.6 Hz), 4.59 (1H, d, *J*=6.4 Hz). ^{13}C NMR ($CDCl_3$) δ 136.1, 135.1, 133.6, 130.3, 130.1, 130.0, 129.8, 129.7, 129.1, 128.6, 128.3, 128.2, 127.9, 126.7, 122.0, 66.1. MS (EI^+) m/z: 313 (M^+, 1.9), 117 (100). HRMS calcd for $C_{22}H_{19}NO$ (M^+): 313.1467, Found: 313.1476.

(*E*)-*O*-(1-Phenylprop-2-enyl)benzaldehyde oxime (5Aa). A colorless oil. IR ($CHCl_3$) 2908, 1494, 1450, 1414 cm^{-1}. ^1H NMR ($CDCl_3$) δ 8.18 (1H, s), 7.60-7.50 (2H, m), 7.45-7.23 (8H, m), 6.16 (1H, ddd, *J*=17.1, 10.5, 6.3 Hz), 5.71 (1H, d, *J*=6.3 Hz), 5.34 (1H, d,

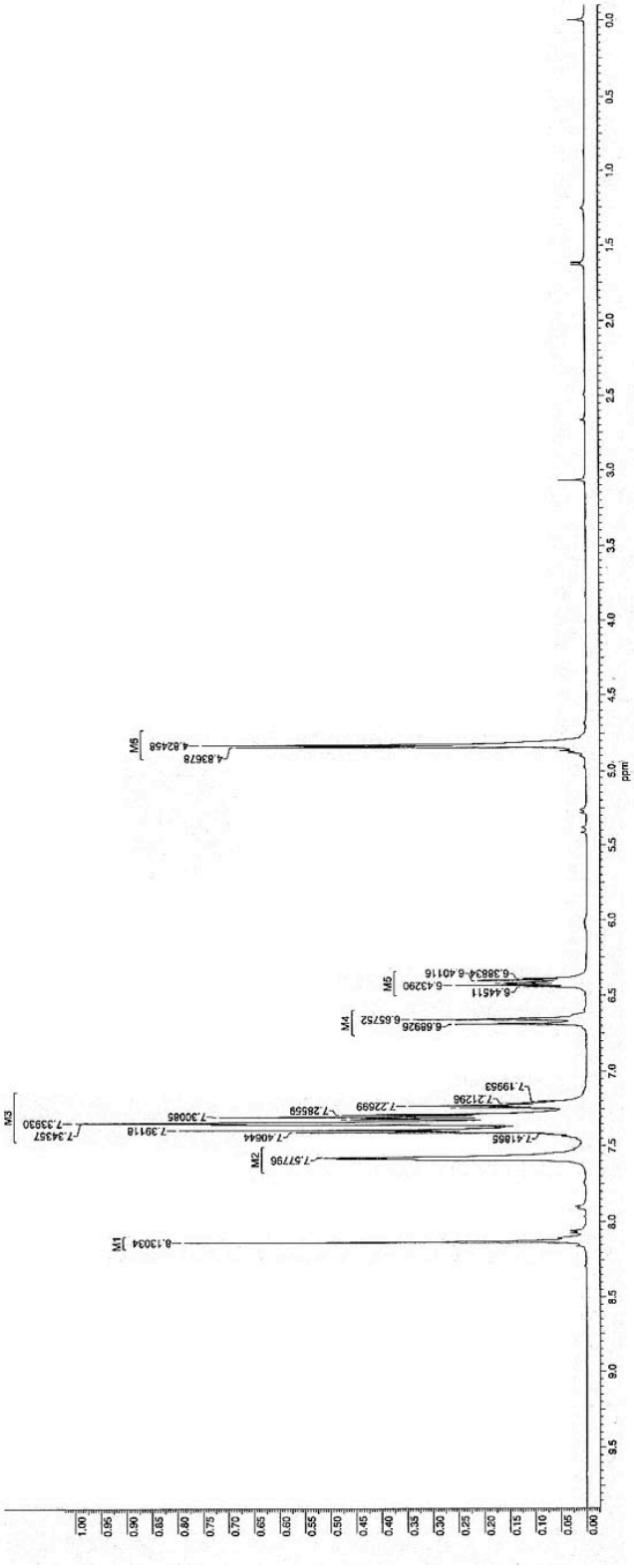
J=17.1 Hz), 5.29 (1H, d, *J*=10.5 Hz). ^{13}C NMR ($CDCl_3$) δ 149.2, 140.1, 137.6, 132.3, 129.8, 128.6, 128.4, 127.9, 127.4, 127.1, 117.3. MS (EI^+) m/z: 237 (M^+, 0.2), 117 (100). HRMS calcd for $C_{16}H_{15}NO$: 237.1154, Found: 237.1156.

Benzylidene(1,3-diphenylprop-2-enyl)amine *N*-oxide (8). A colorless solid. IR ($CHCl_3$) 1581, 1494, 1451 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 8.25 (2H, m), 7.60-7.25 (14H, m), 6.86 (1H, dd, *J*=16.2, 7.9 Hz), 6.69 (1H, d, *J*=16.2 Hz), 5.75 (1H, d, *J*=7.9 Hz). ^{13}C NMR ($CDCl_3$) δ 137.2, 135.9, 135.2, 133.5, 130.5, 130.4, 128.9, 128.8, 128.7, 128.5 (2C), 127.8, 126.9, 125.2, 82.0. One peak of ^{13}C NMR was missing due to overlap. MS (CI^+) m/z: 314 ($M+H^+$, 0.6), 193 (100). HRMS calcd for $C_{22}H_{20}NO$ ($M+H^+$): 314.1545, Found: 314.1543.

References

1) (a) Davies, S. G.; Jones, S.; Sanz, M. A.; Teixeira, F. C.; Fox, J. F. *Chem. Commun.* **1998**, 2235. (b) Tiecco, M.; Testaferri, L.; Tingoli, M.; Bagnoli, L. *Chem. Commun.* **1995**, 235.
2) (a) Tiecco, M.; Testaferri, L.; Marini, F.; Sternativo, S.; Santi, C.; Bagnoli, L.; Temperini, A. *Tetrahedron: Asymmetry.* **2001**, *12*, 3059. (b) Tiecco, M.; Testaferri, L.; Tingoli, M.; Bagnoli, L. *Chem. Commun.* **1995**, 235.
3) (a) Koyama, J.; Ogura, T.; Tagahara, K.; Miyashita, M.; Irie, H. *Chem. Pharm. Bull.* **1993**, *41*, 1297. (b) Koyama, J.; Ogura, T.; Tagahara, K.; Irie, H. *Chemistry Express* **1991**, *6*, 197.

3. Copies of ^{1}H and ^{13}C NMR spectra of all obtained compounds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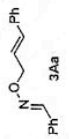
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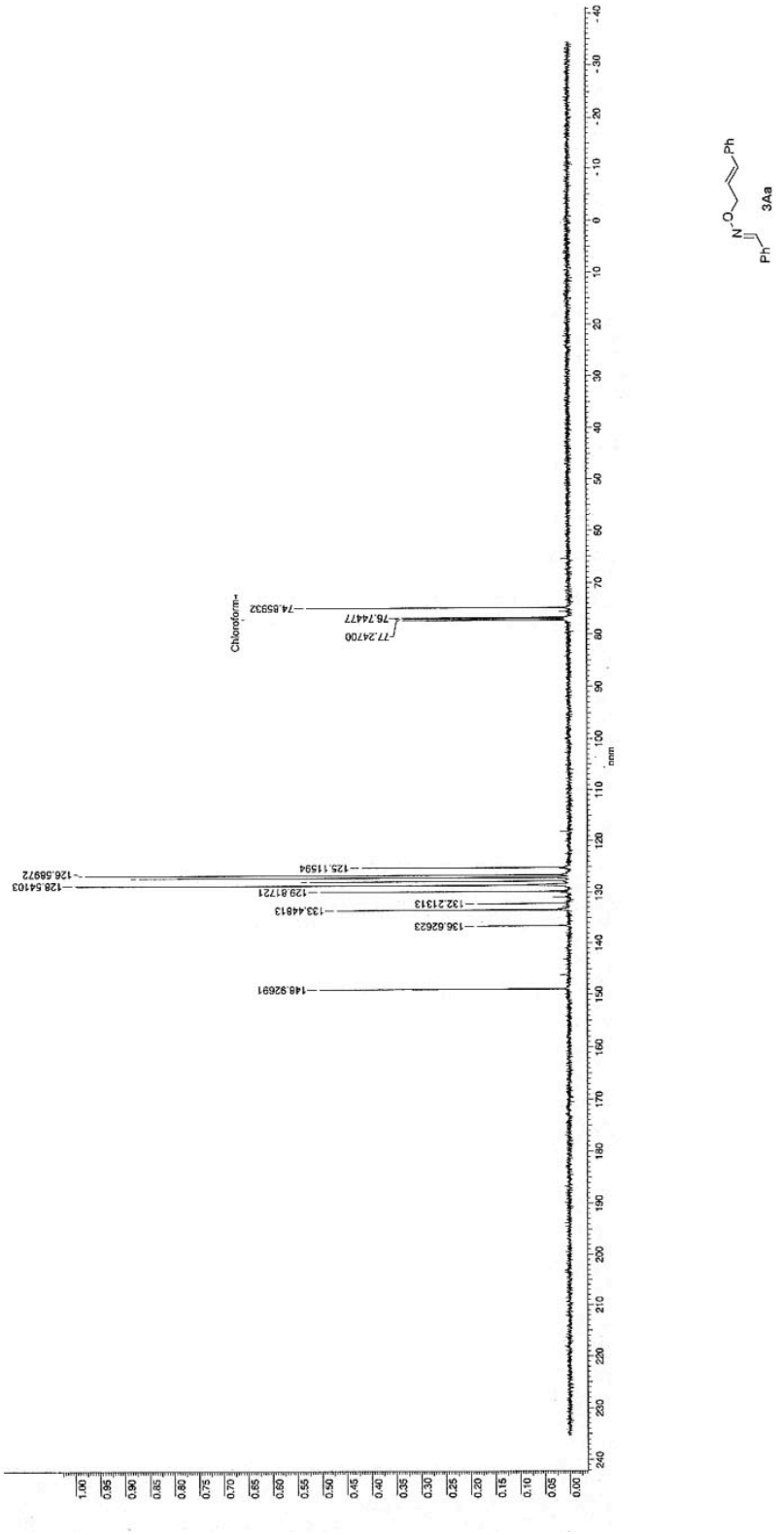
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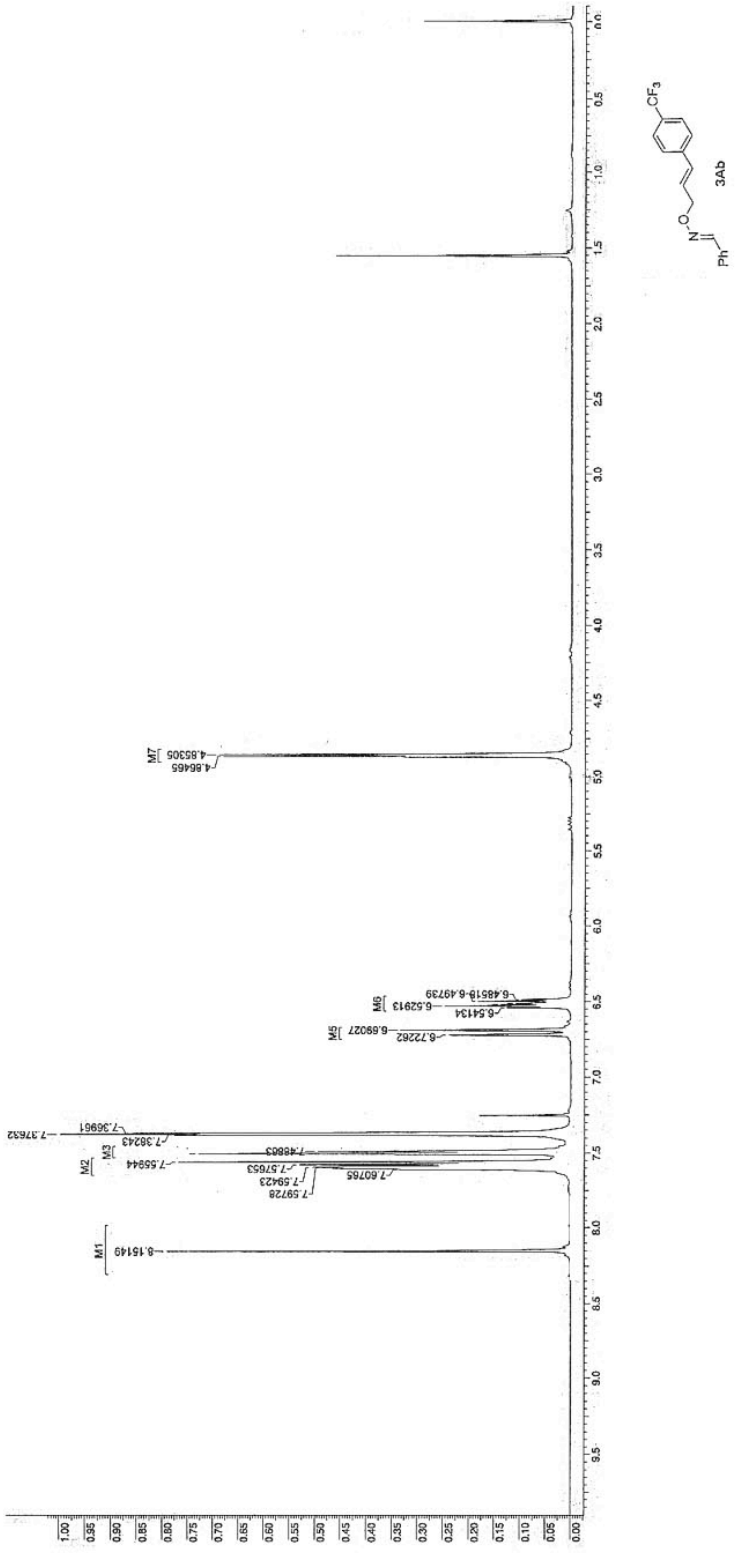
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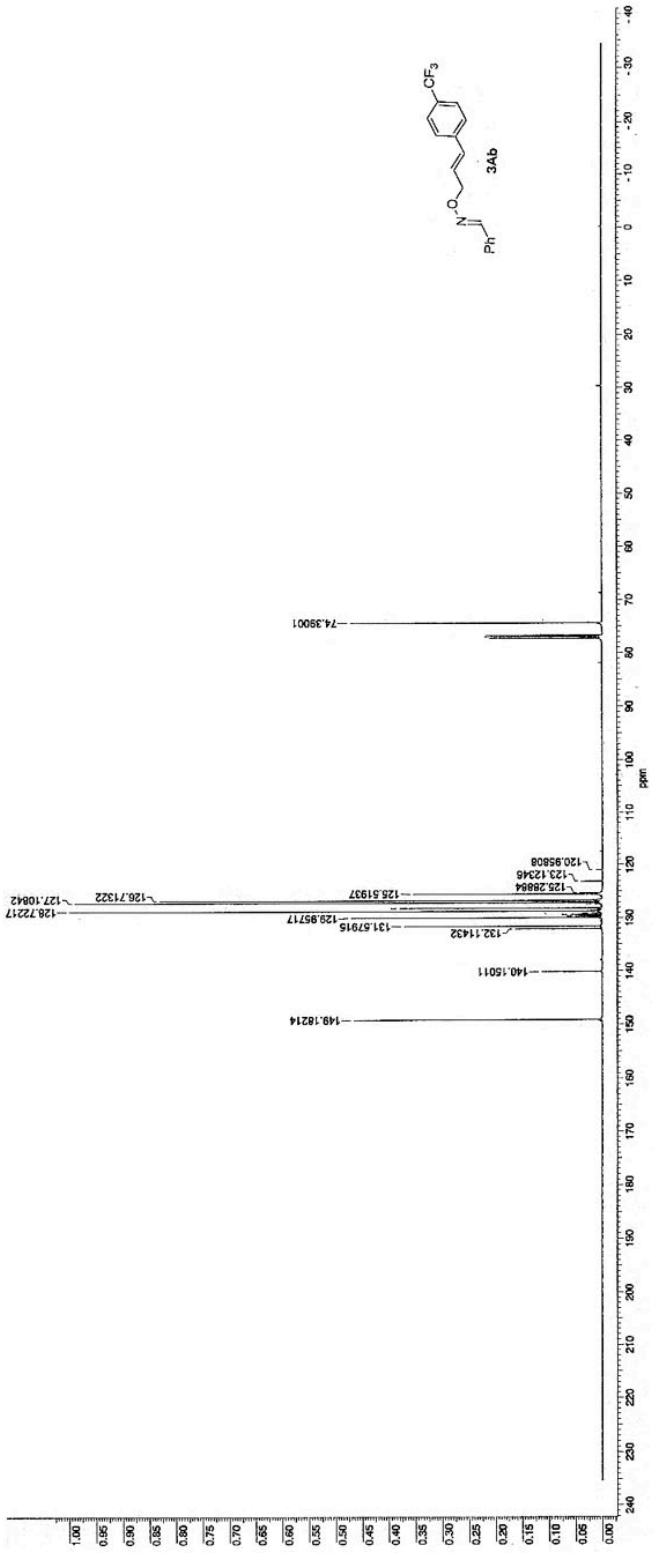
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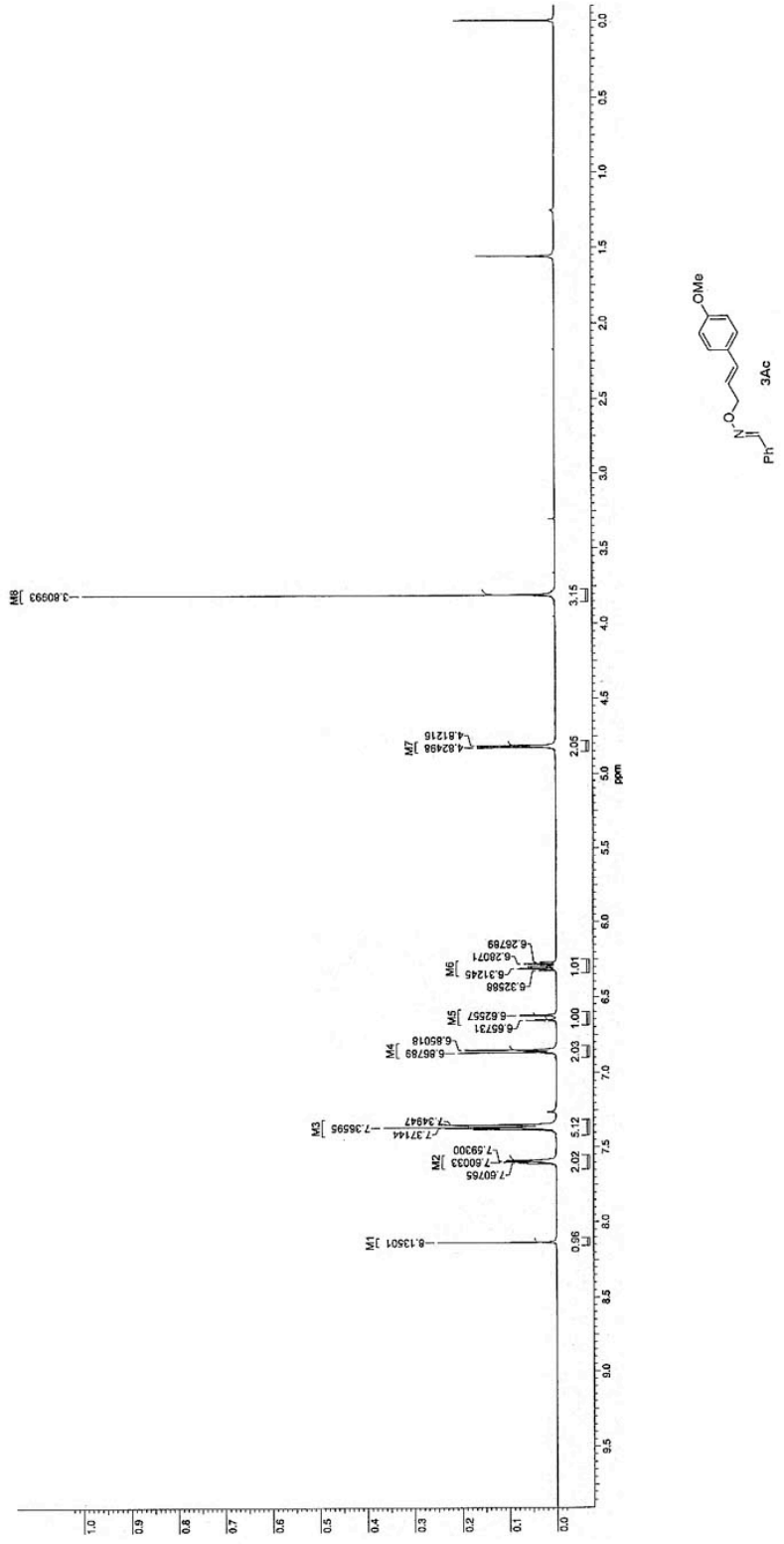
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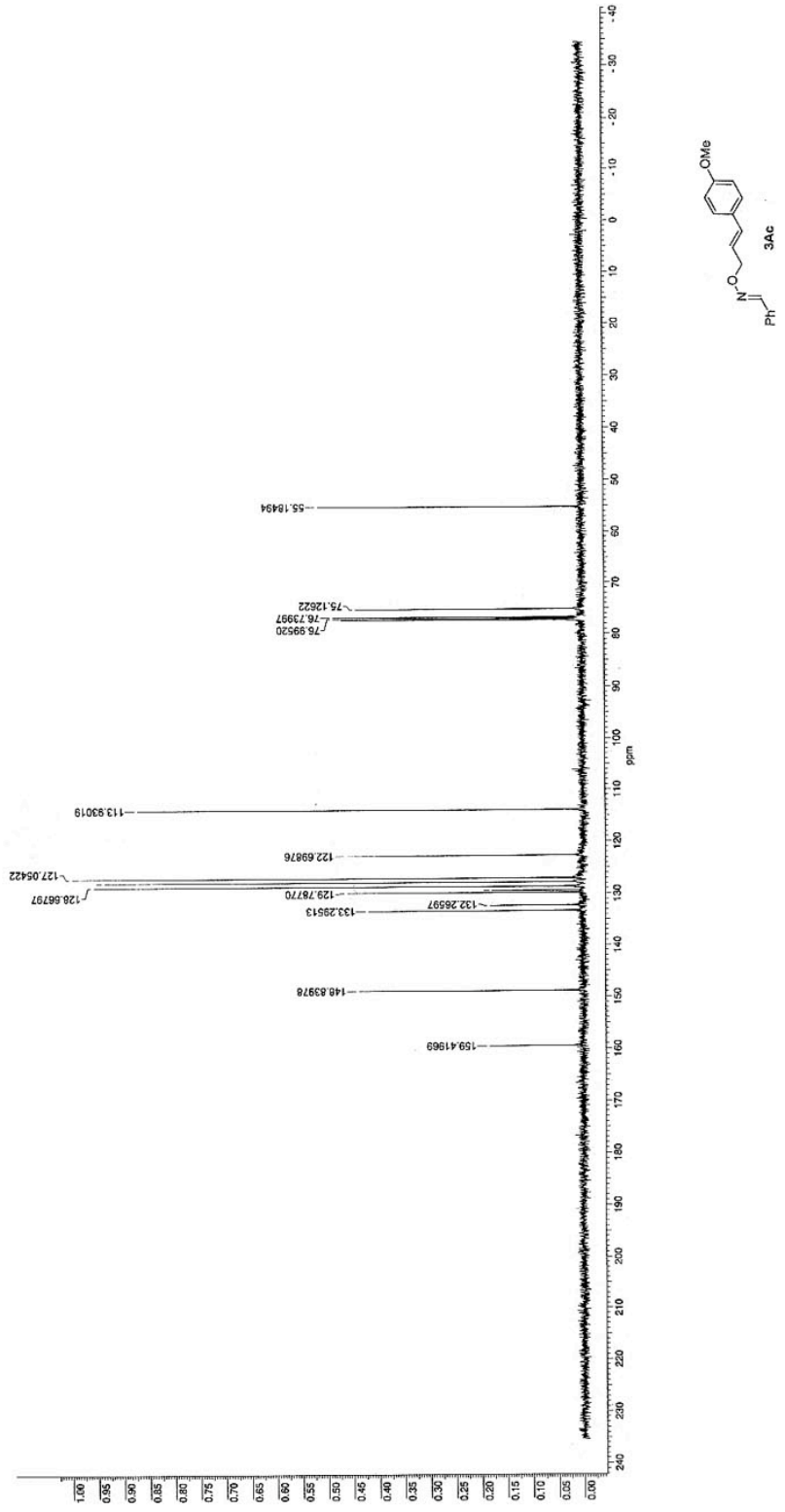
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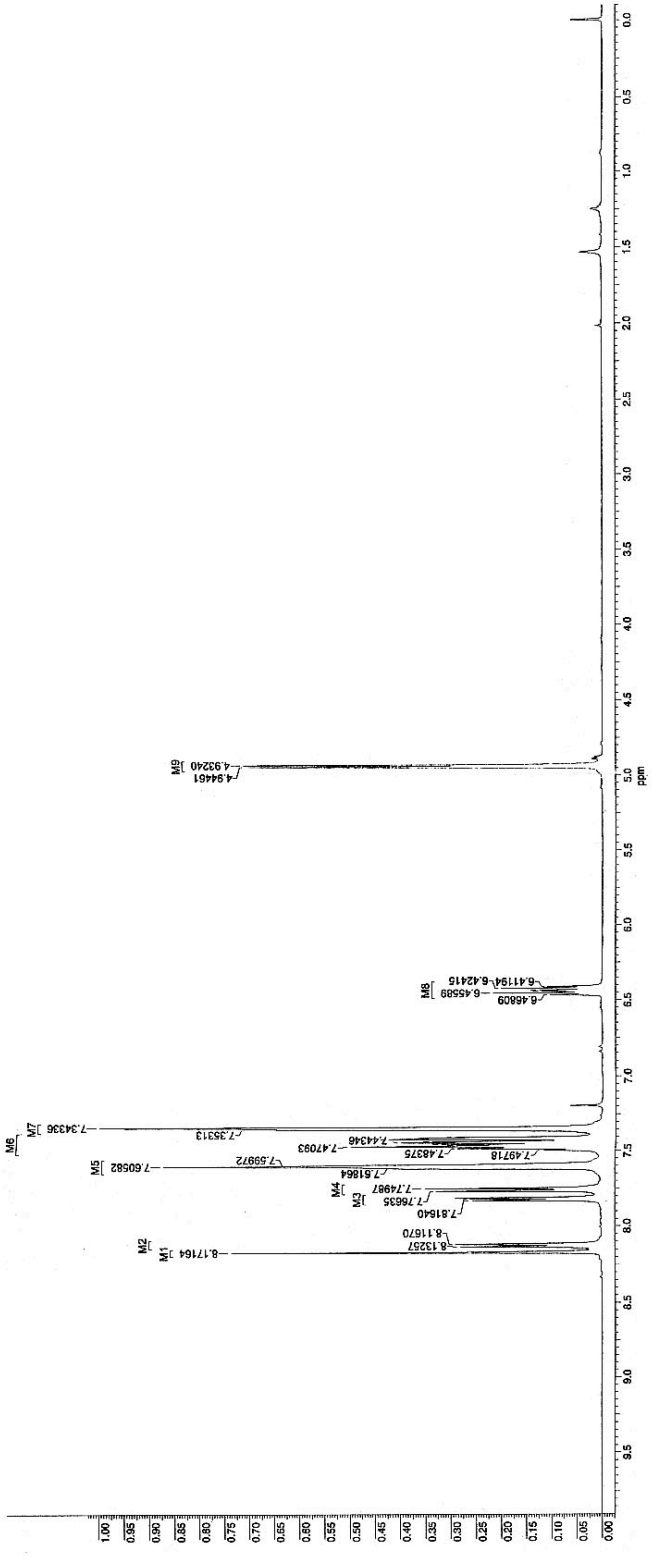
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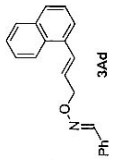
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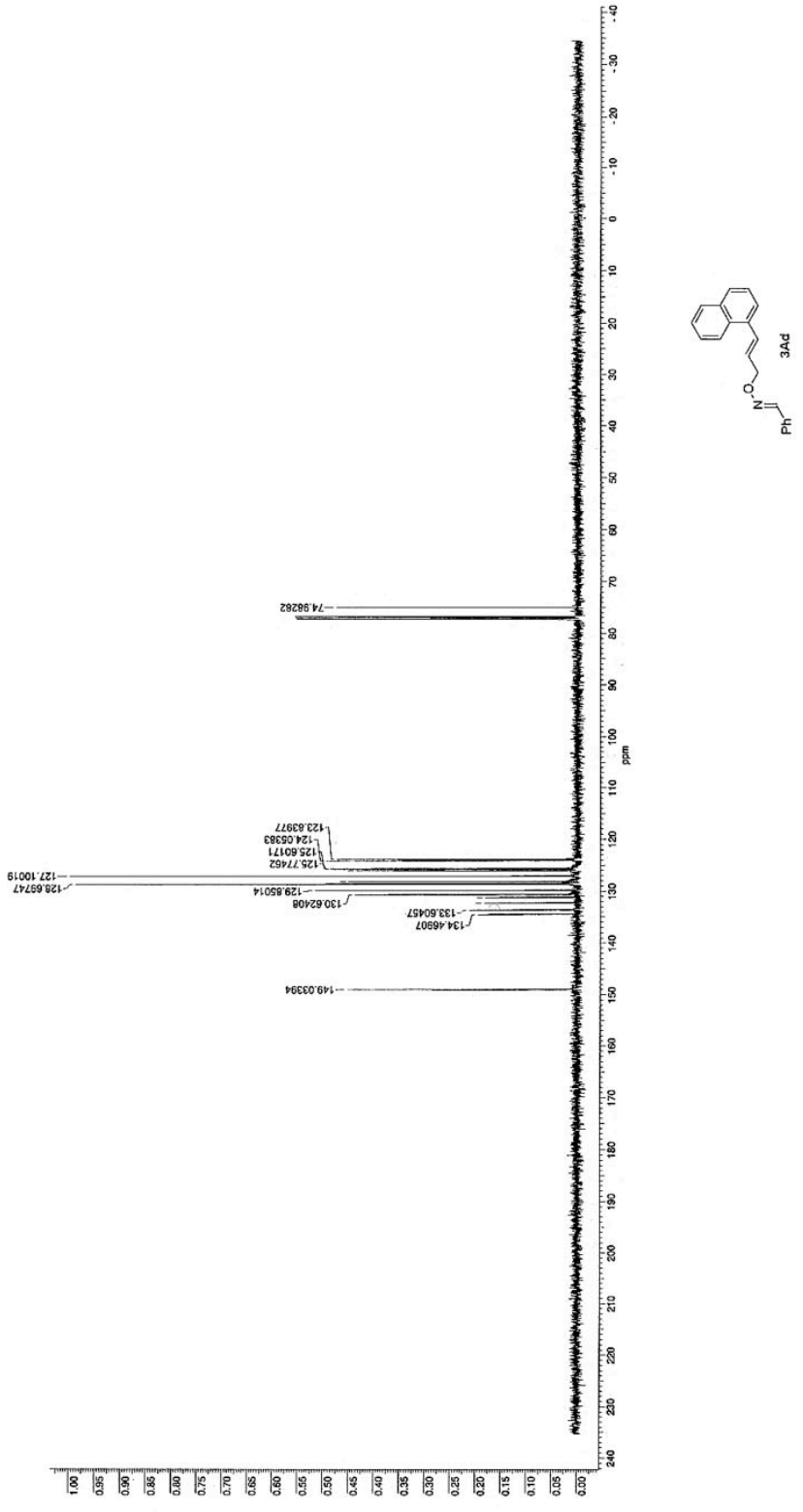
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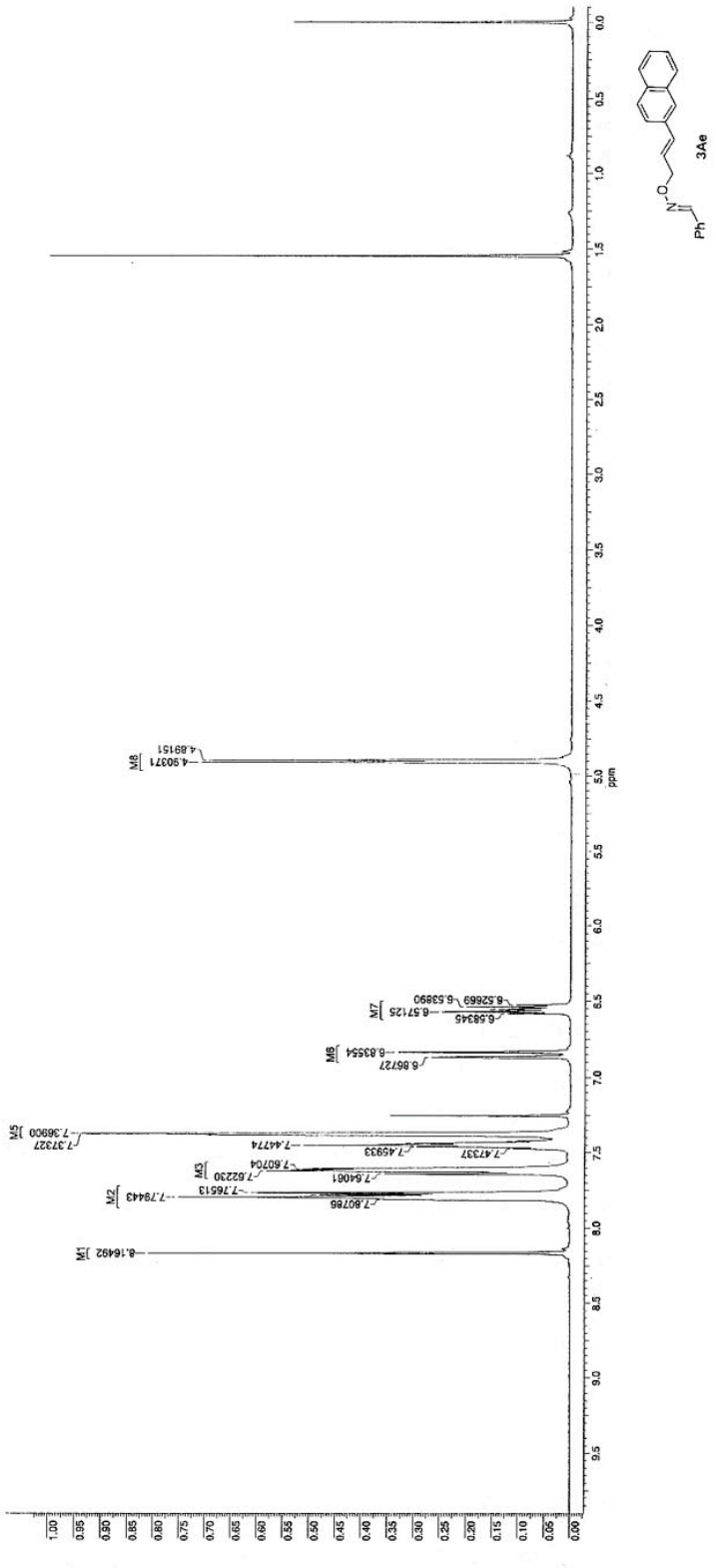
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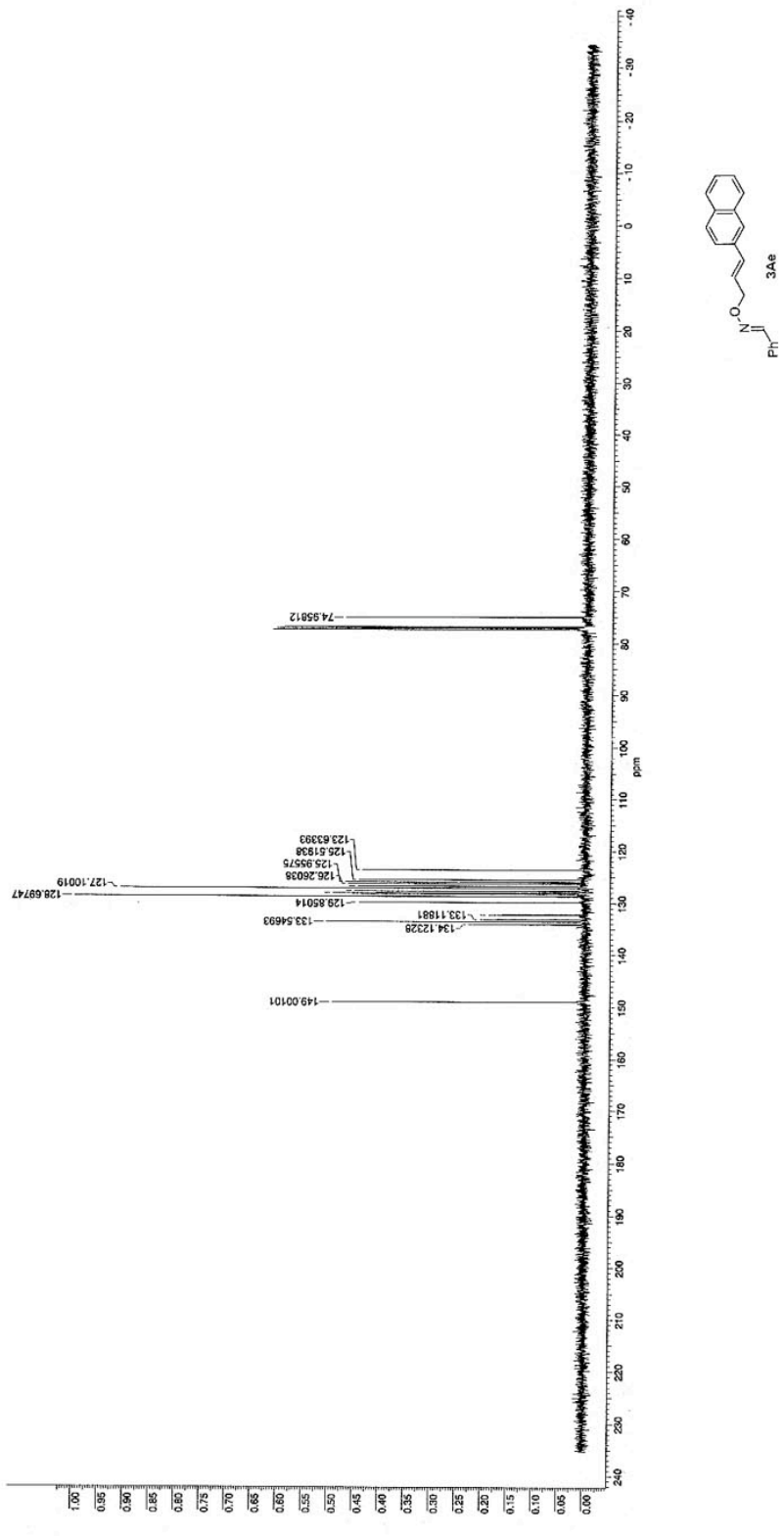
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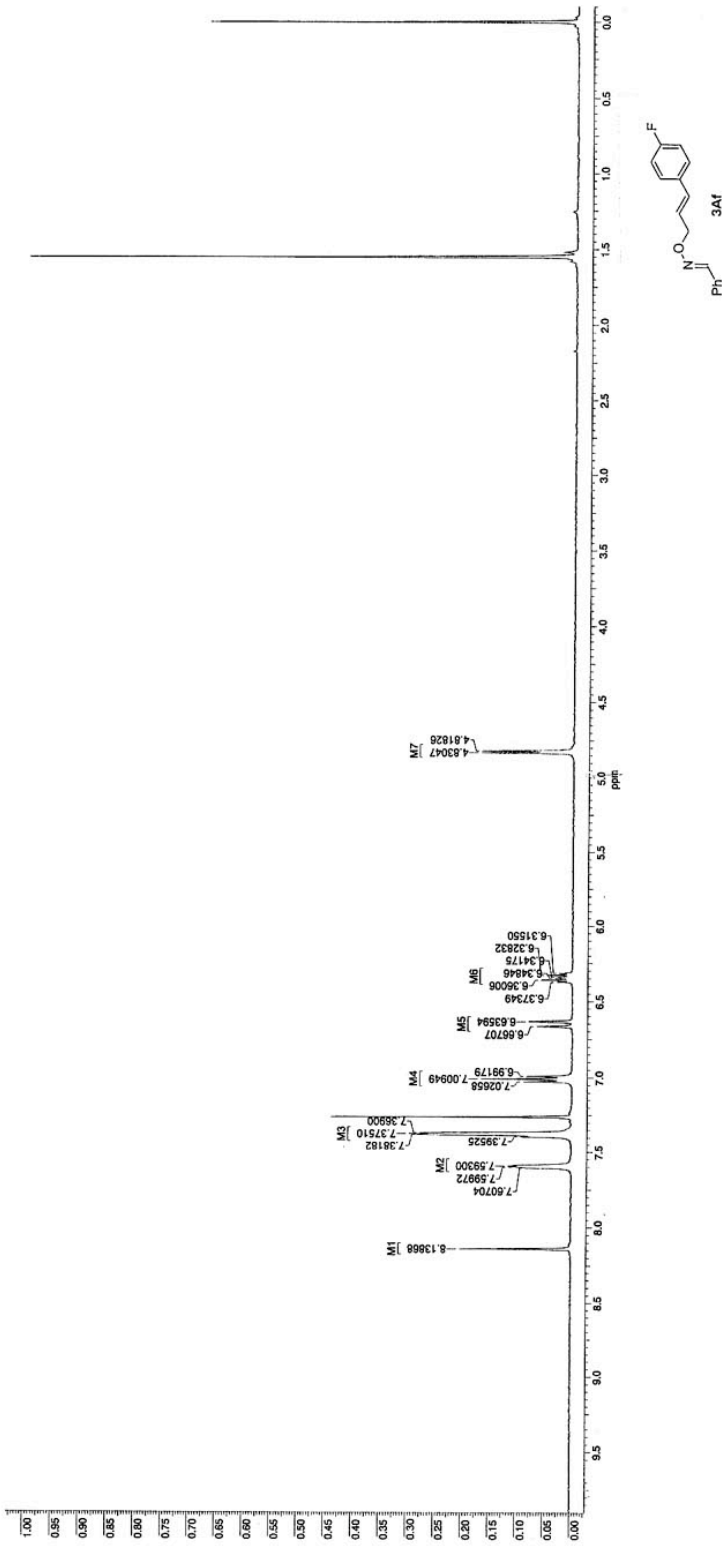
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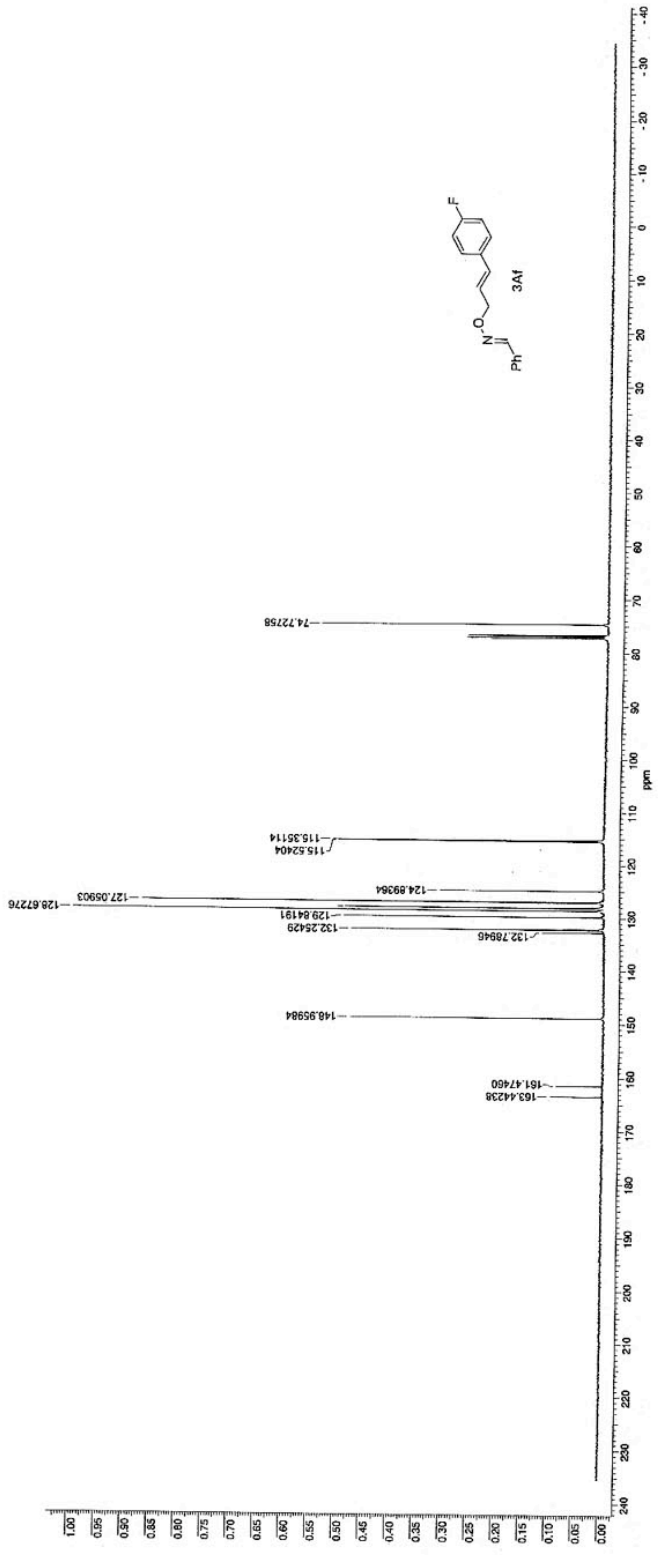
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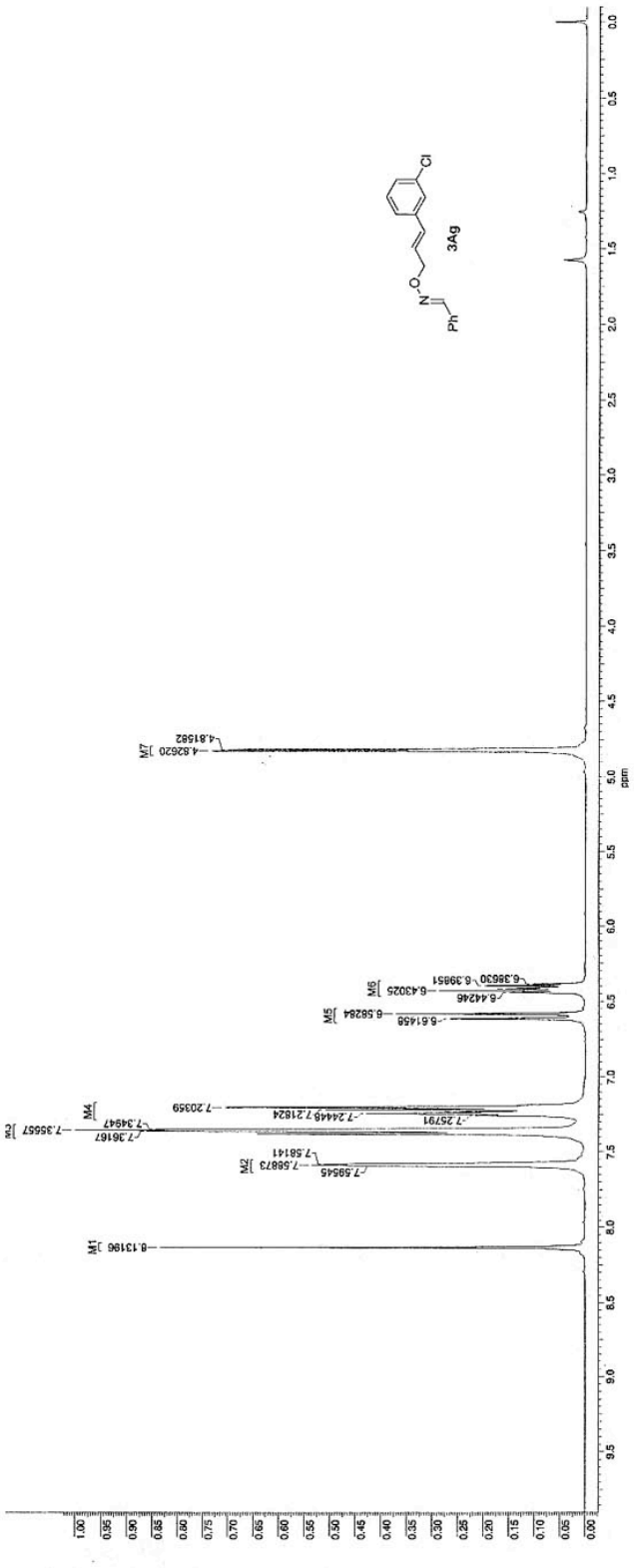
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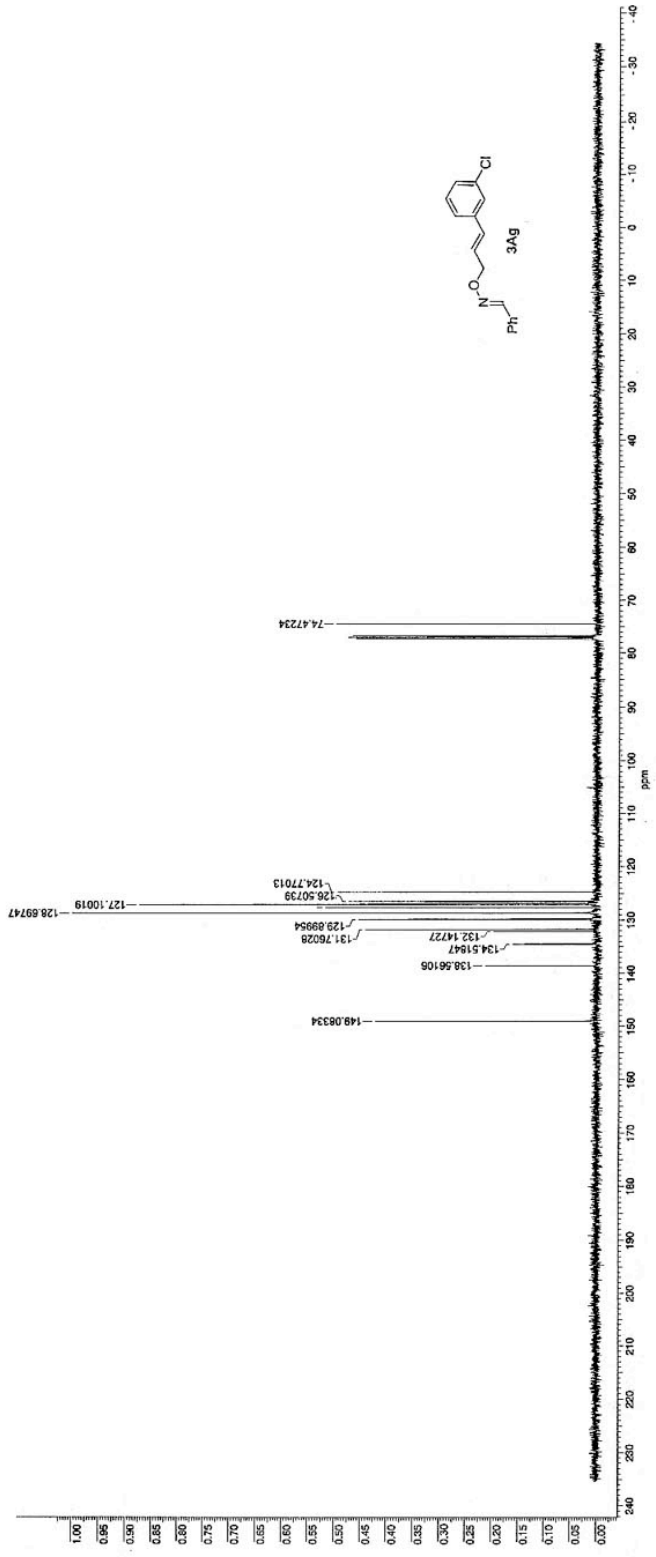
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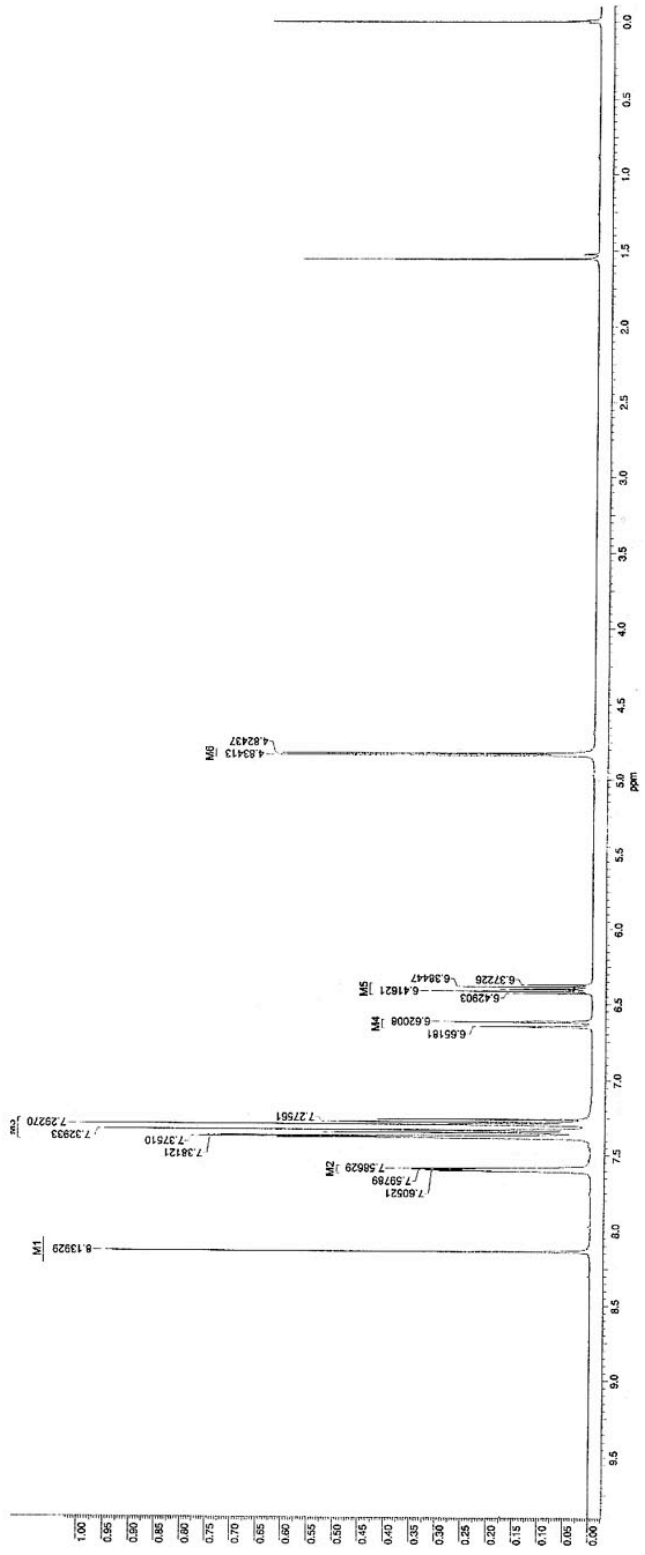
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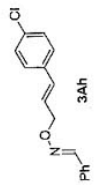
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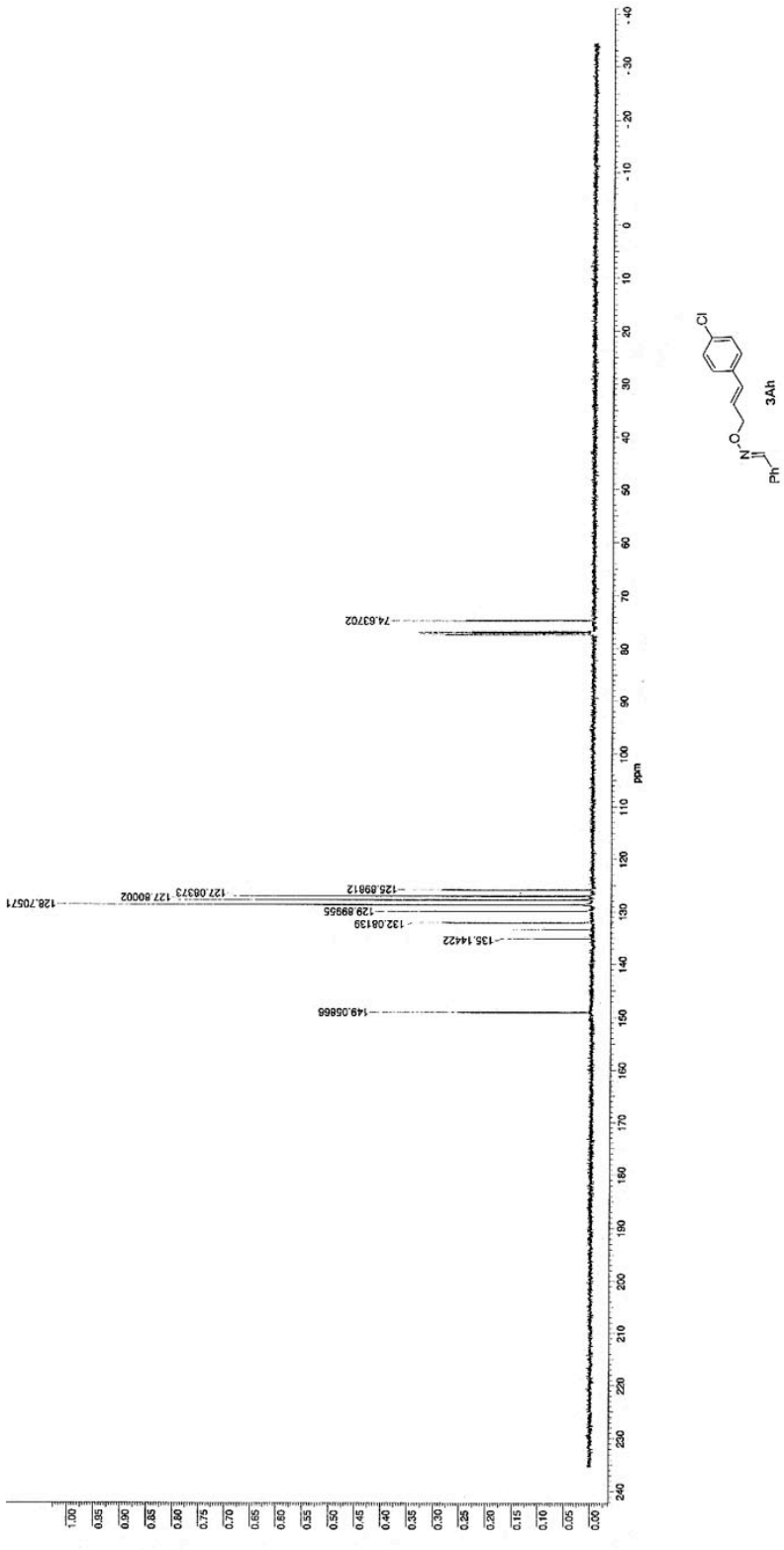
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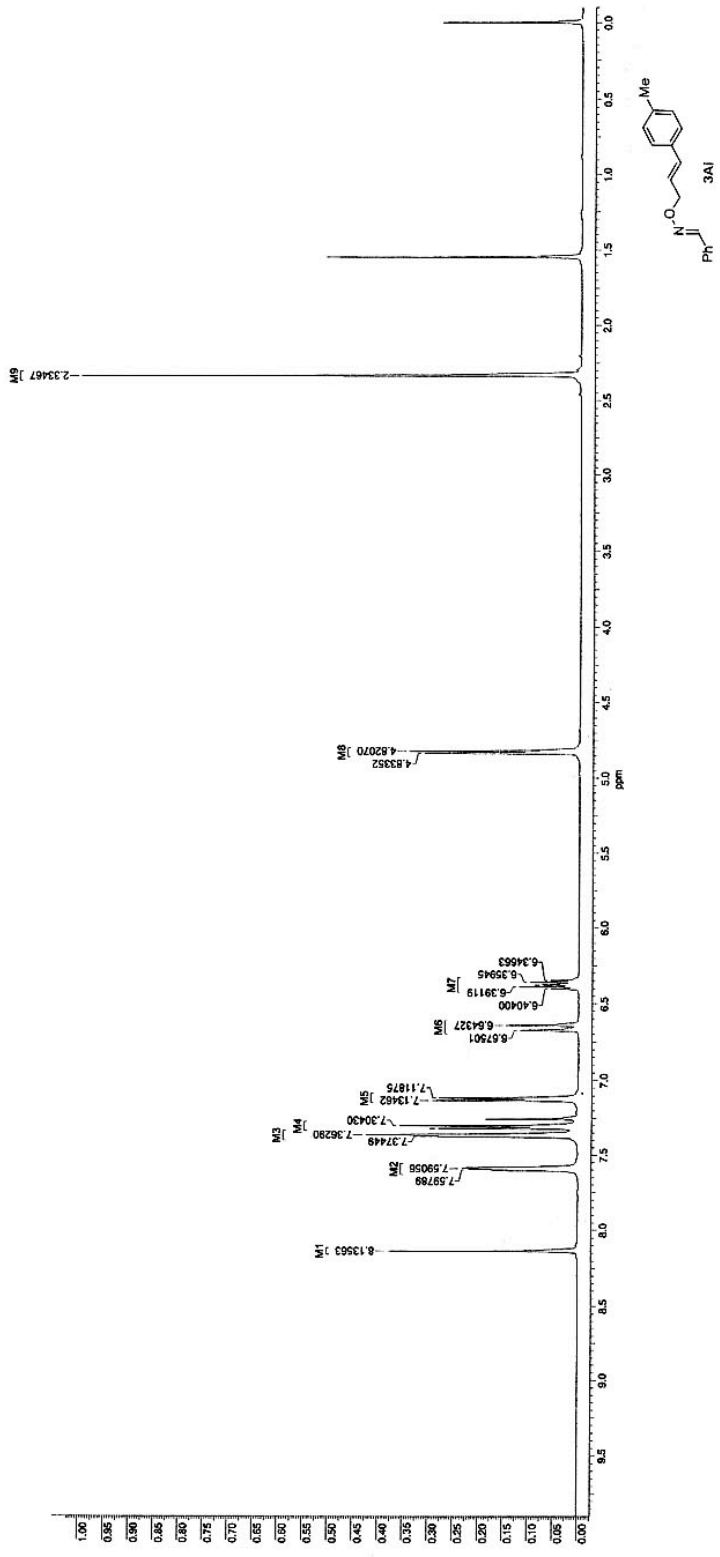
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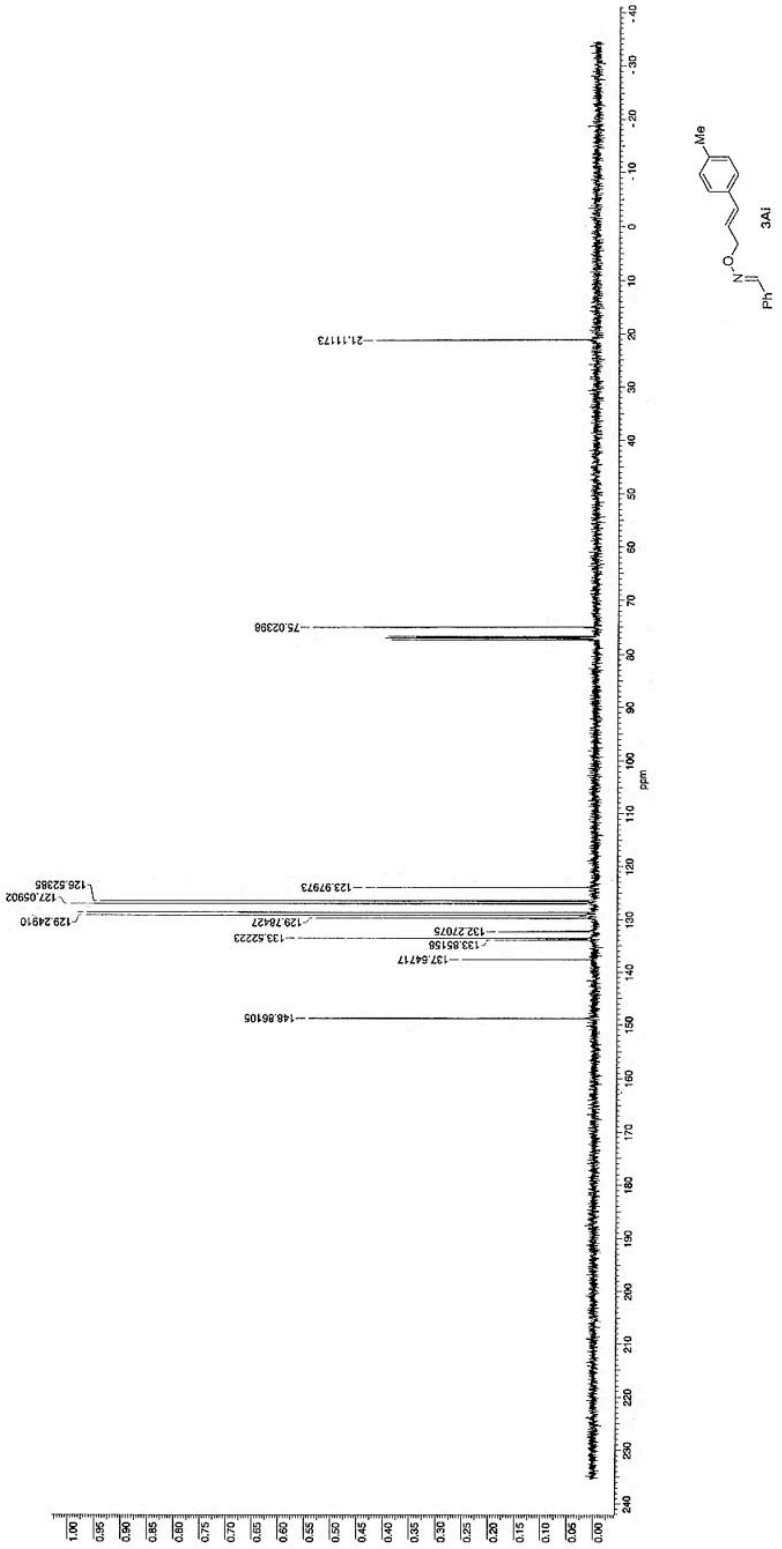
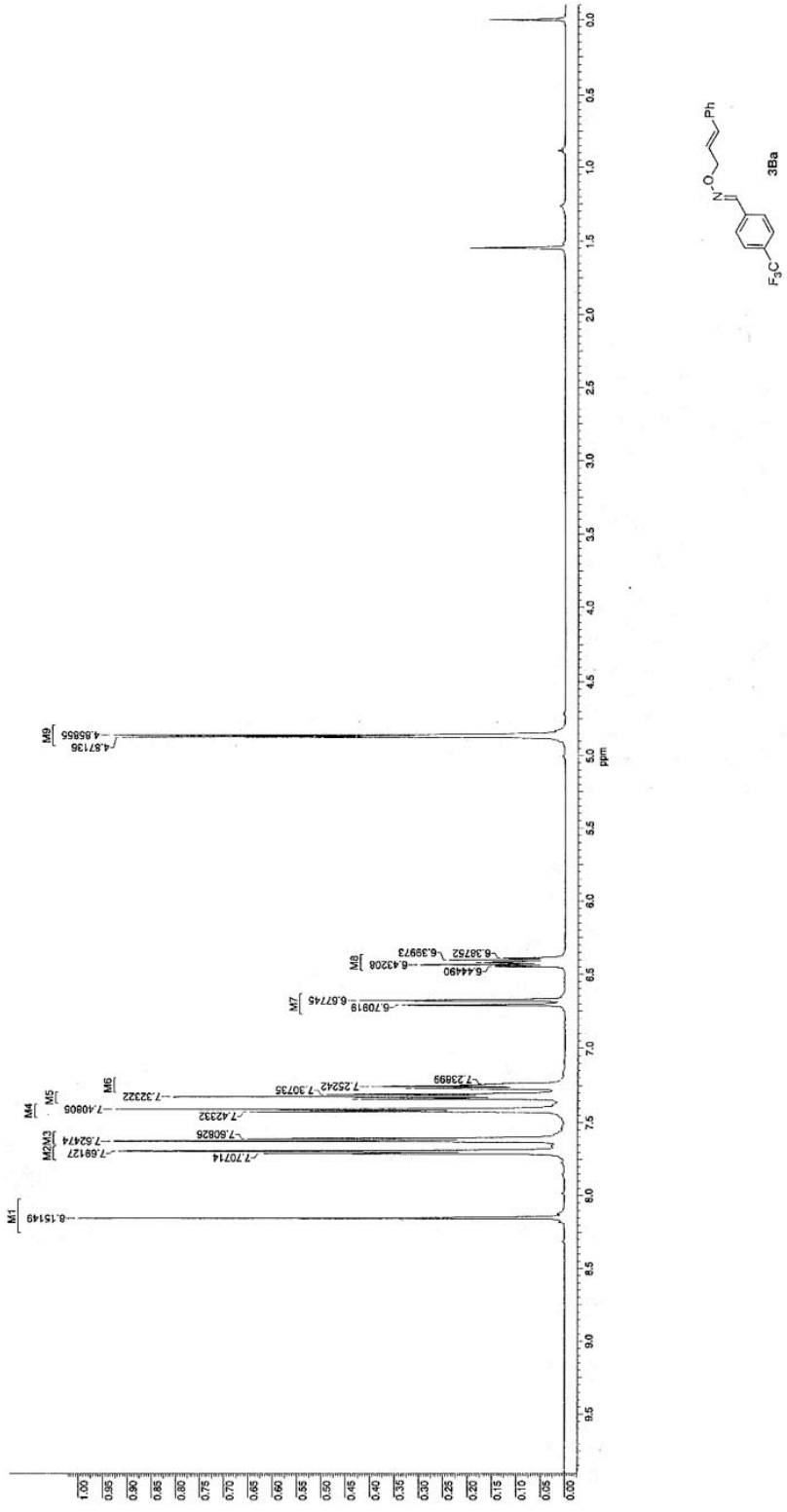
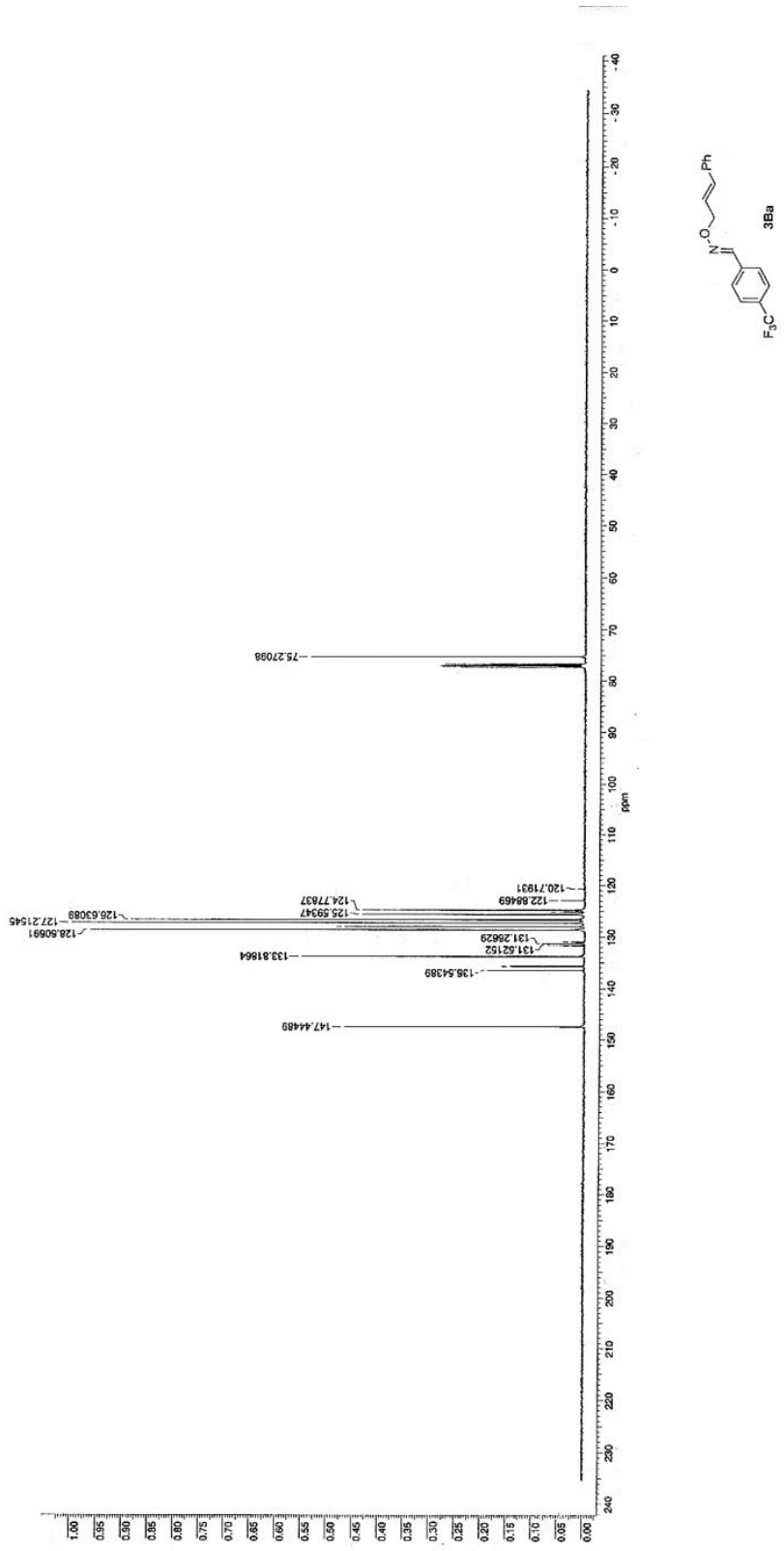
M4 7.30430
M3 7.37449 7.36280
M2 7.59789 7.59056
M1 8.13563
ppm


21.11173
75.02398
123.97973
126.52385
127.05902
129.24810
129.78427
132.27075
133.85223
133.85156
137.64717
148.86105
ppm
Me
O
N
Ph
3Ai


3Ba
Ph
O
N
F3C
M1
8.15149
M2M3
7.89127
7.82474
7.70714
7.60826
M4
7.40806
M5
7.42332
M6
7.32322
7.25242
7.30735
7.23899
M7
6.67745
6.70919
M8
6.44490
6.43208
6.38752
6.39973
M9
4.87135
4.85855
ppm


3Ba
Ph
O
N
F3C
ppm
75.27038
120.71831
122.88469
124.77837
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126.63089
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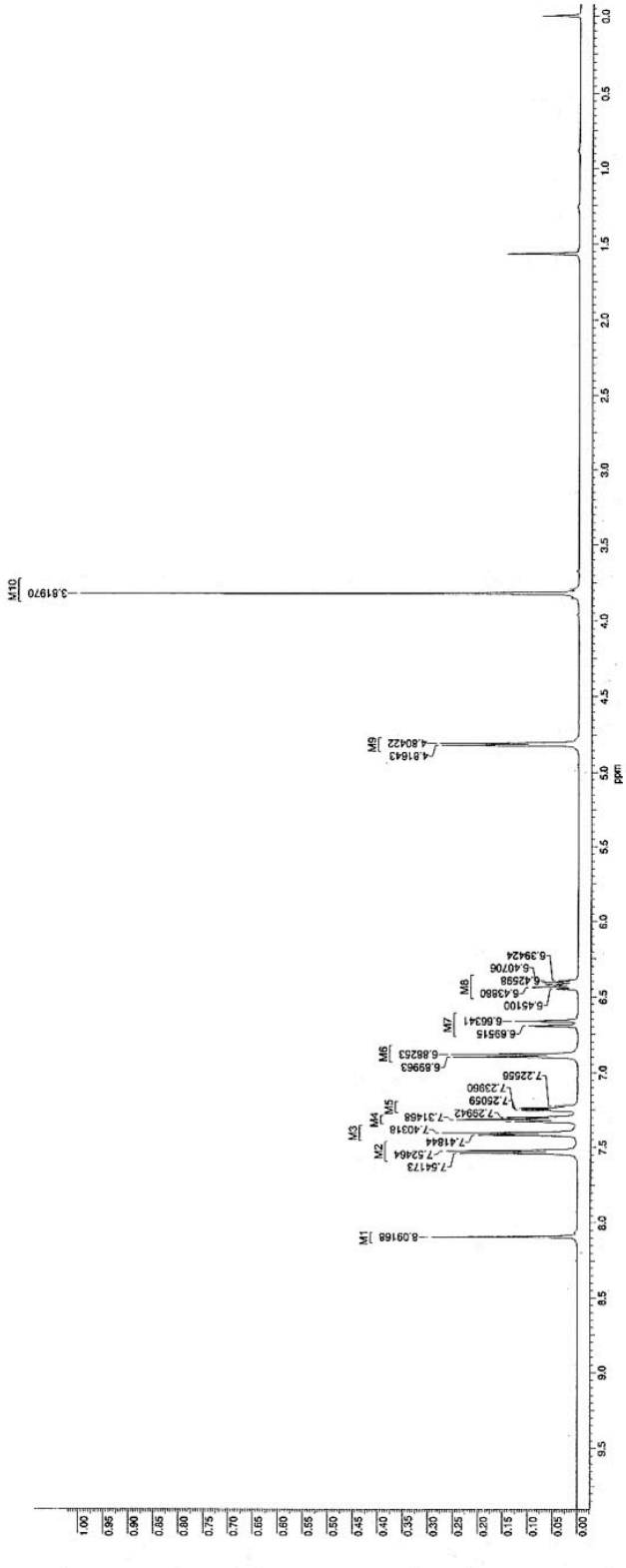
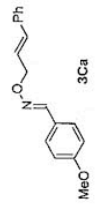
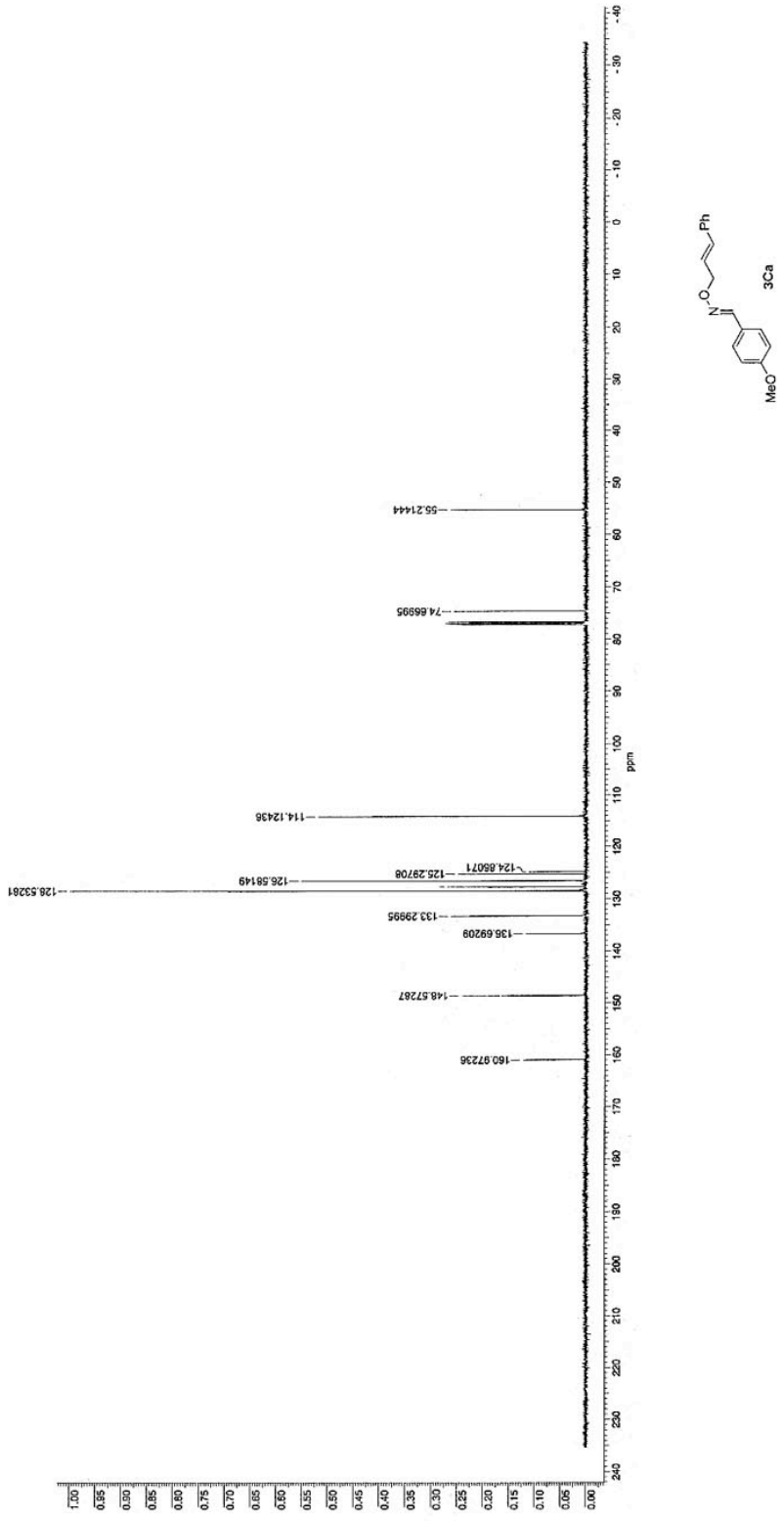
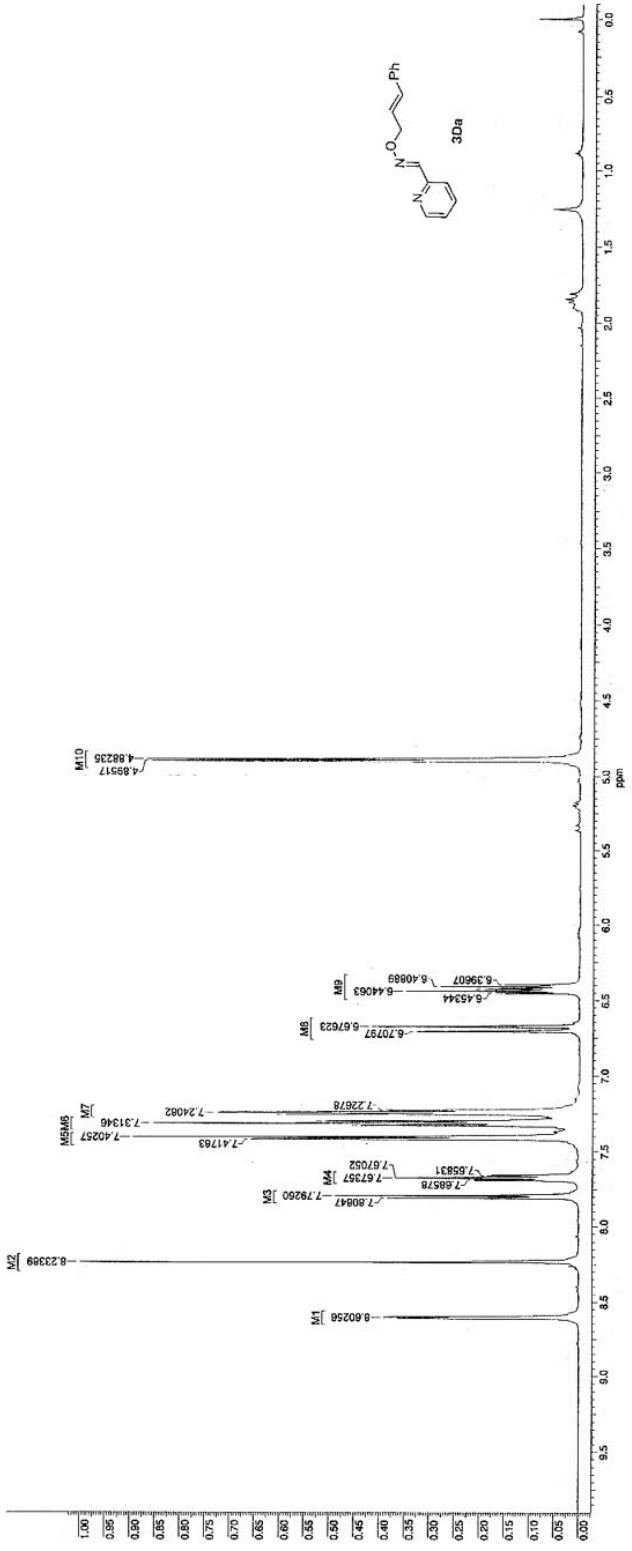
131.26629
131.52152
133.81864
135.54389
147.44489

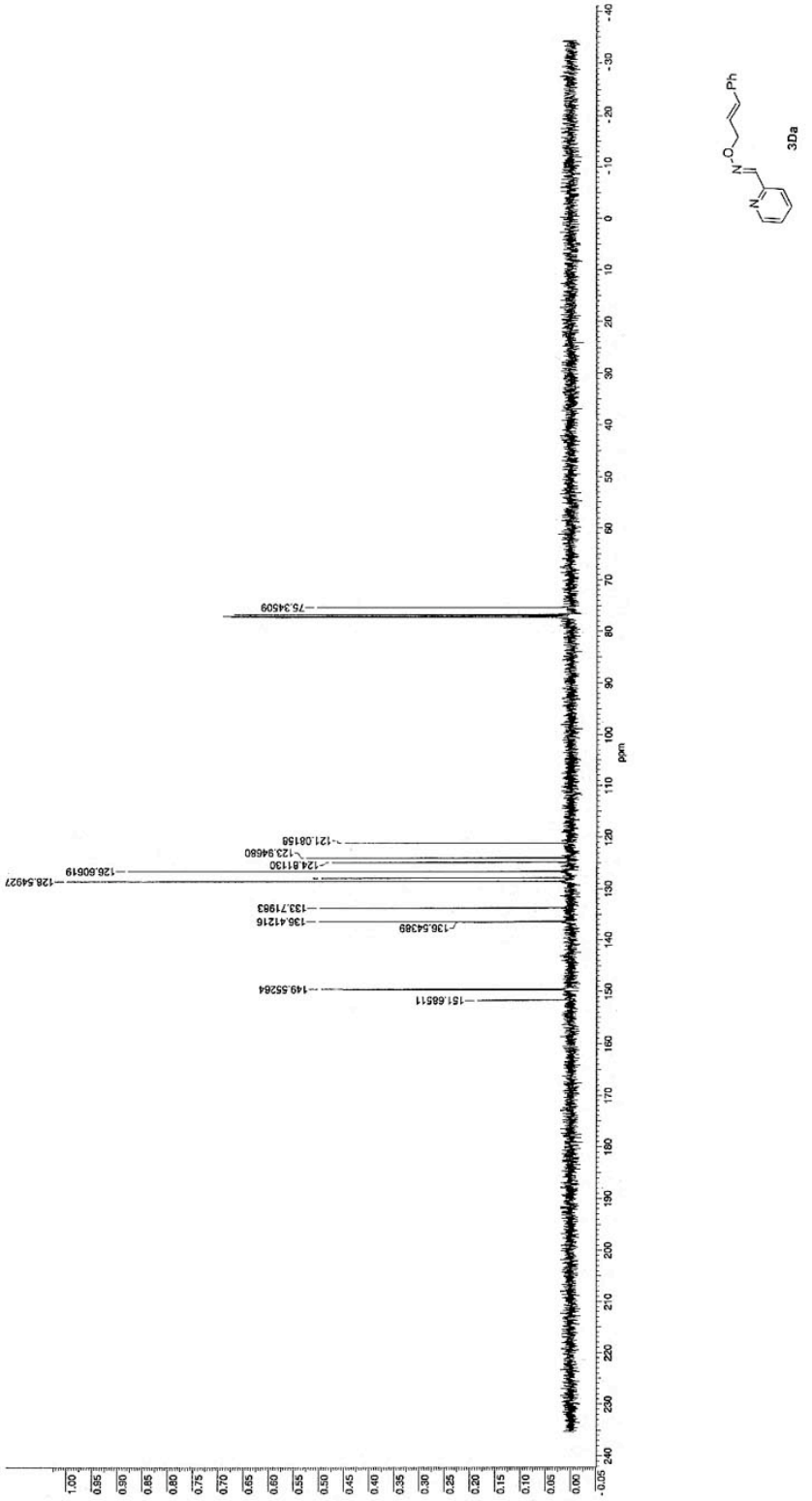

M10
3.81970
M9
4.81643
4.80422
M8
6.45100
6.43880
6.42588
6.40706
6.39424
M7
6.66341
6.65515
M6
6.88263
6.87963
M5
7.25642
7.25059
7.23960
7.22556
M4
7.31458
M3
7.40318
7.41844
M2
7.53464
7.54173
M1
8.09168
ppm


Ph
O
N
MeO

3Ca


55.21444
74.66995
114.12436
124.85071
125.28708
126.58149
128.53281
133.29995
136.69209
148.57287
160.97236
ppm
Ph
O
N
MeO
3Ca


3Da
Ph
N
O
N
M1 8.60256
M2 8.23389
M3 7.80847 7.79260
M4 7.68578 7.67357 7.67052 7.65831
M5M6 7.41763 7.40257
M7 7.31346 7.24082 7.22678
M8 6.70797 6.67623
M9 6.45344 6.44063 6.40689 6.39607
M10 4.89517 4.88236
ppm


Ph
O
N
N
3Da
75.34509
121.08158
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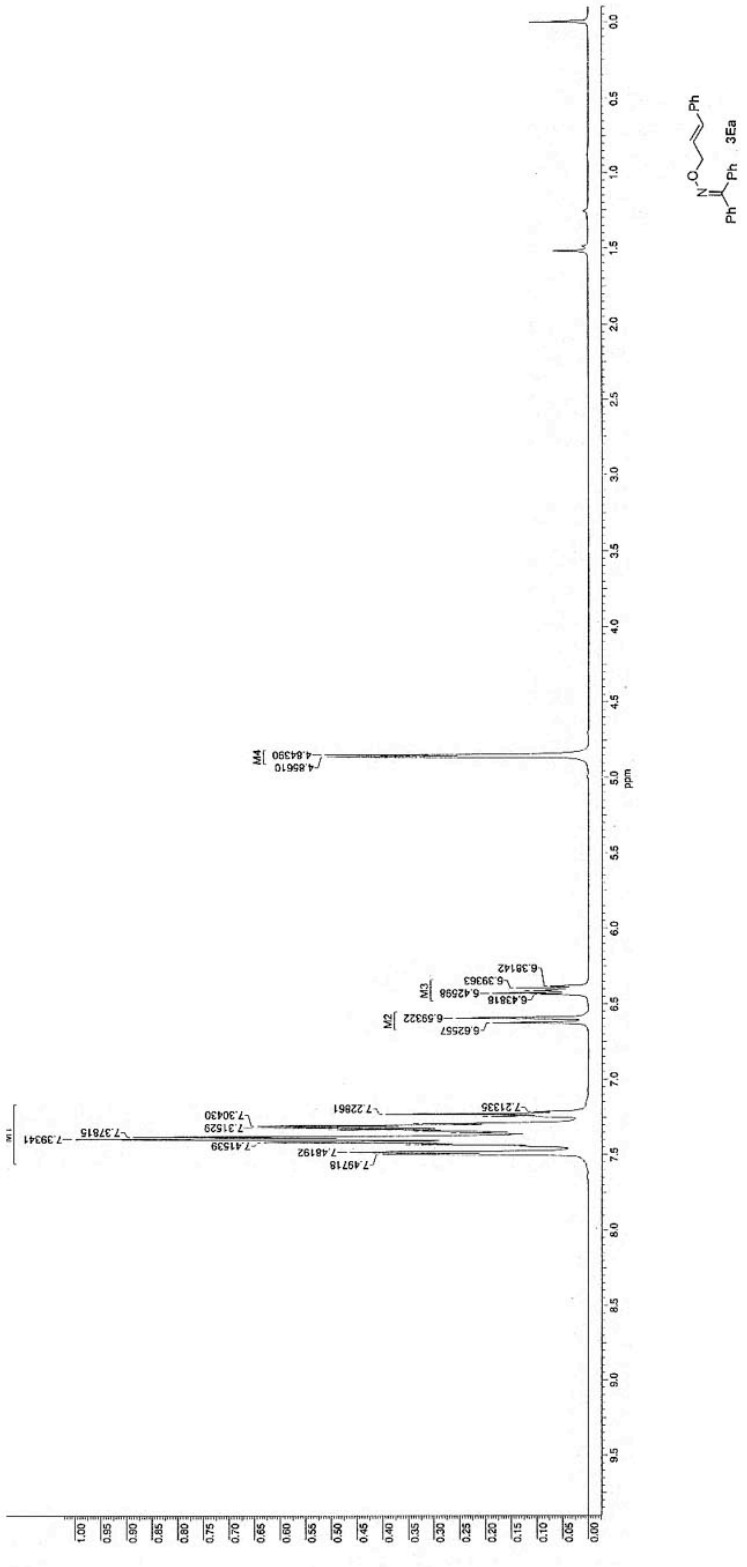
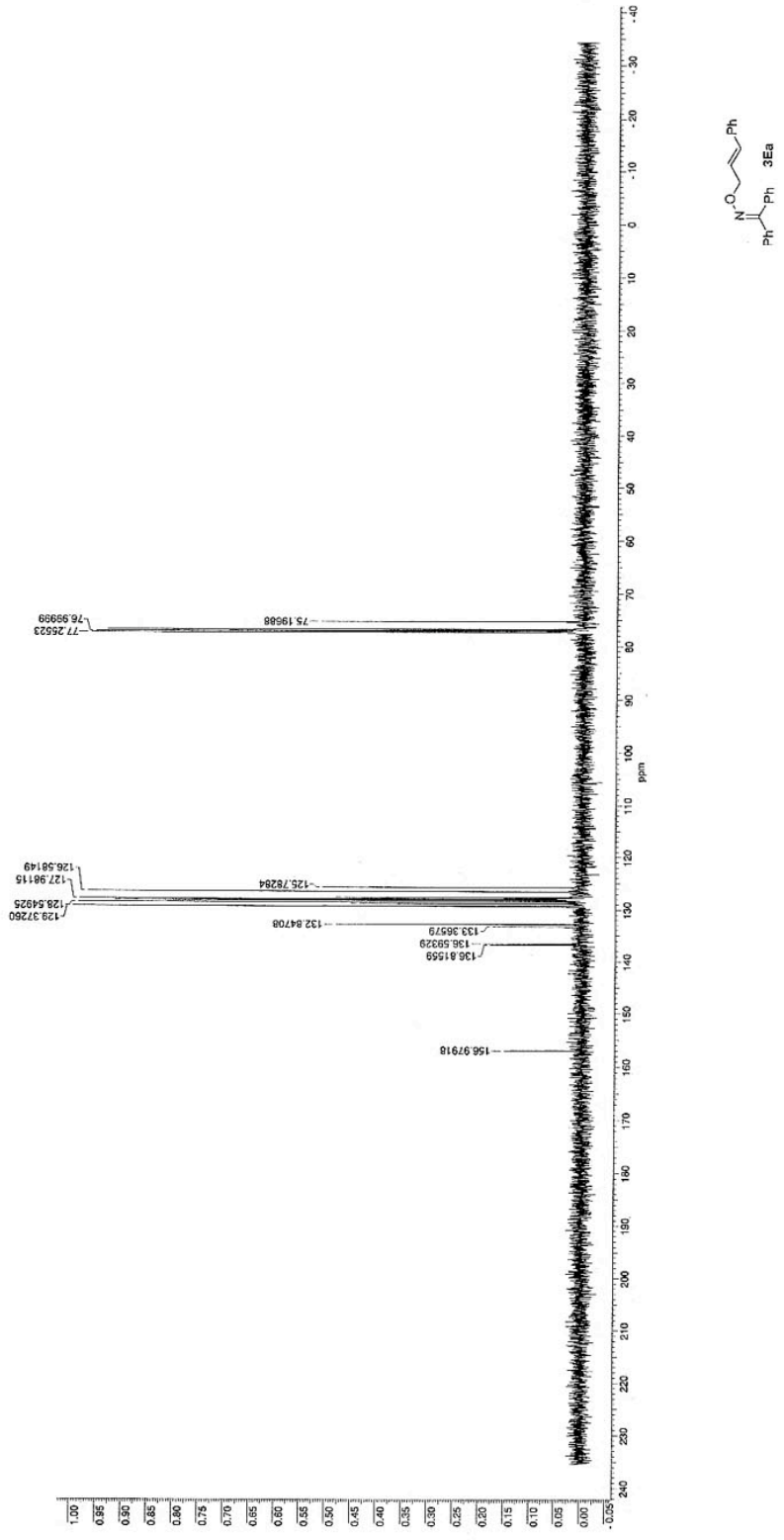
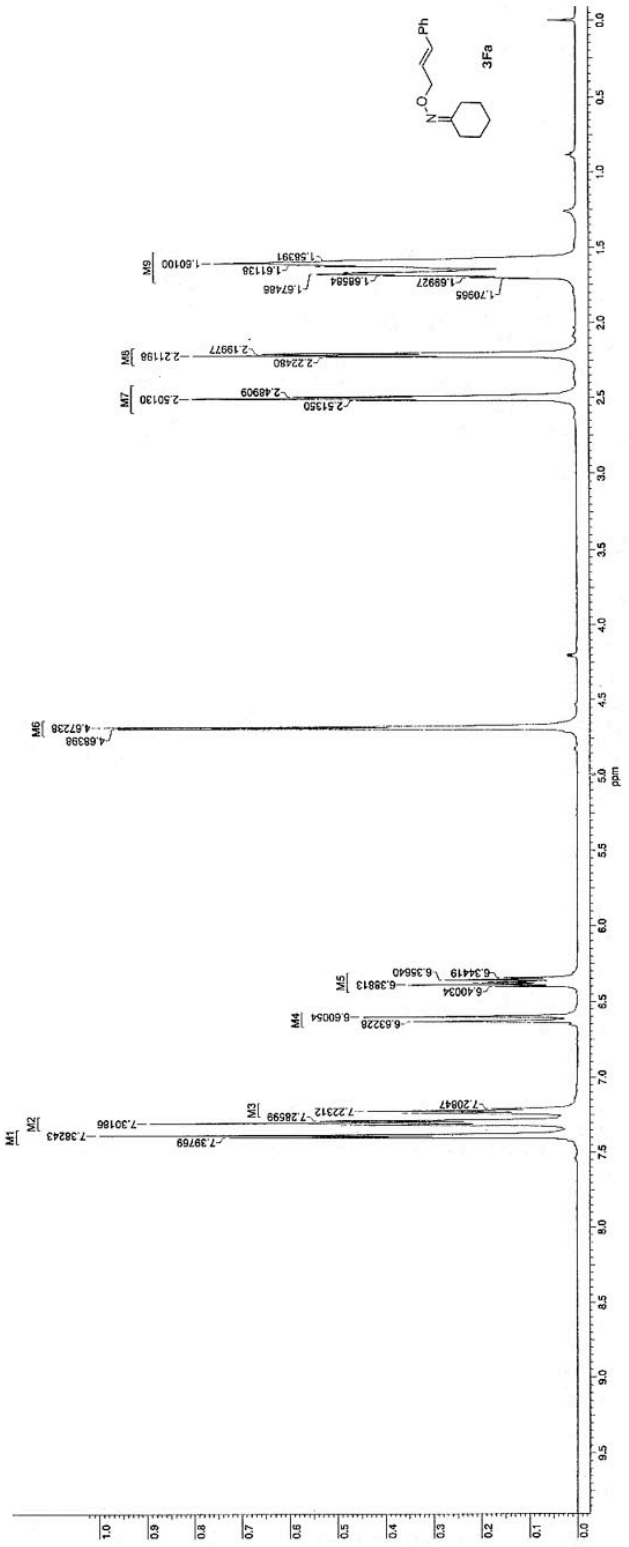
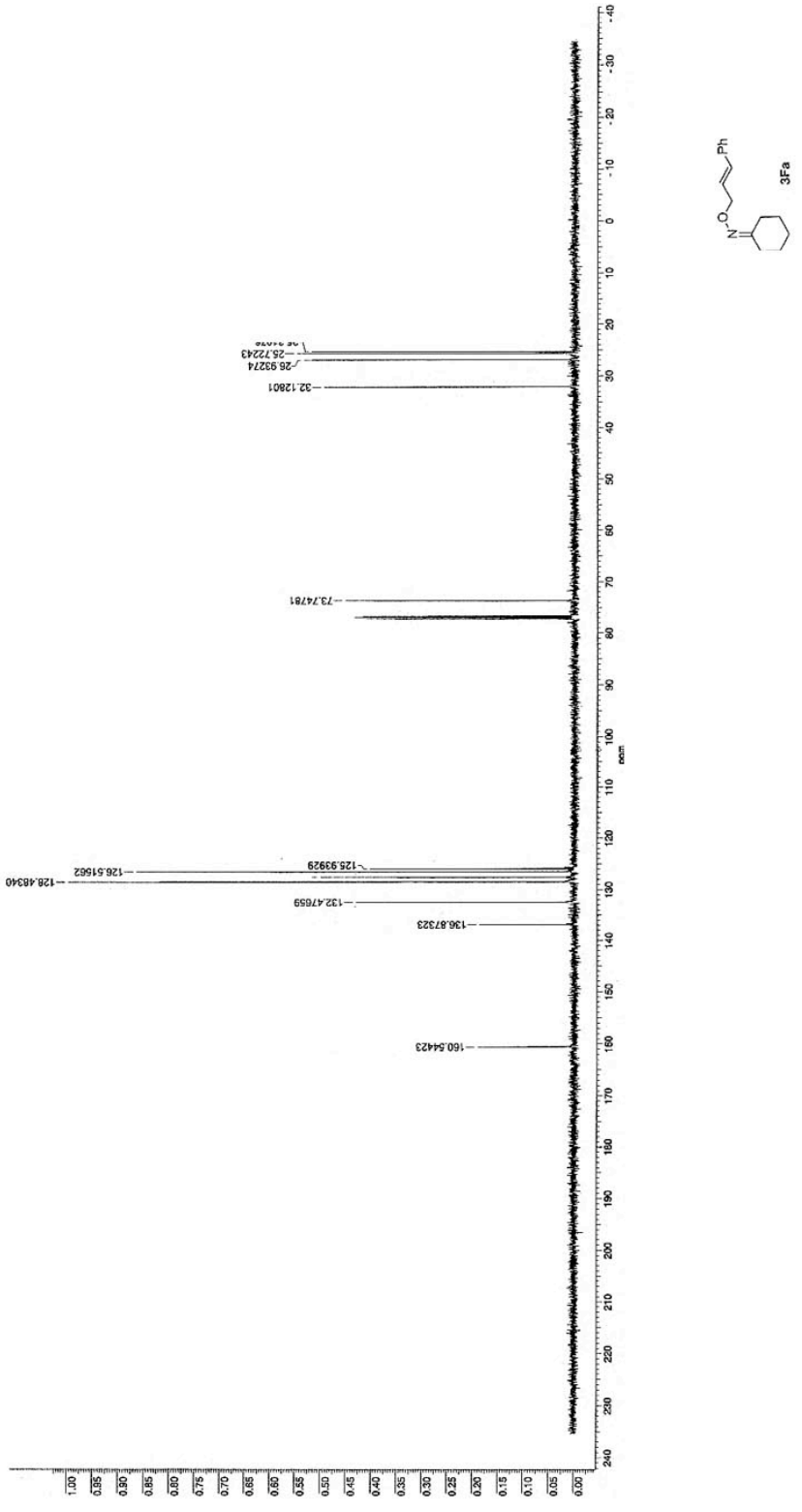
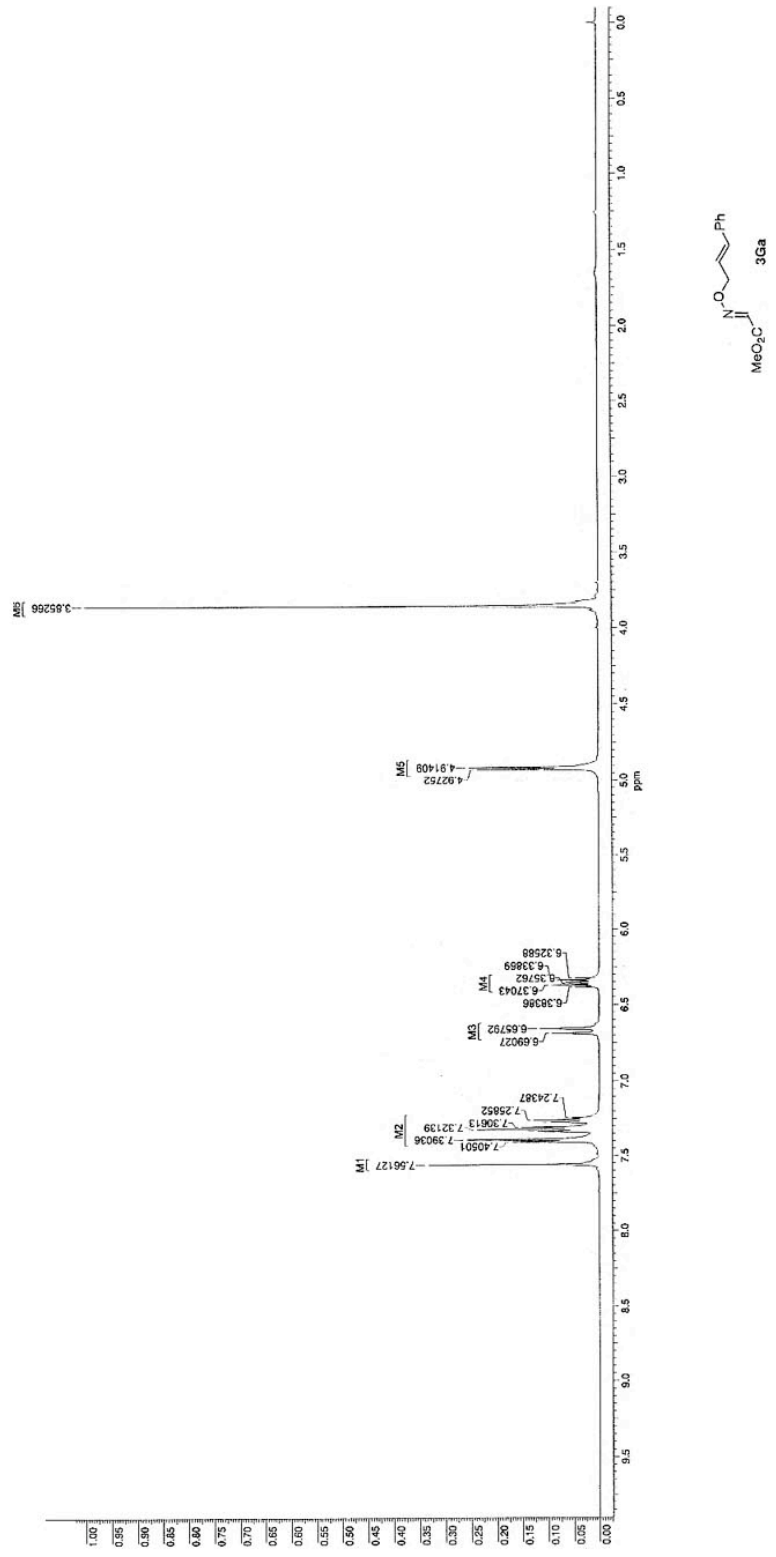
124.81130
126.60619
128.54927
133.71983
136.41216
136.54389
149.55264
151.68511
ppm

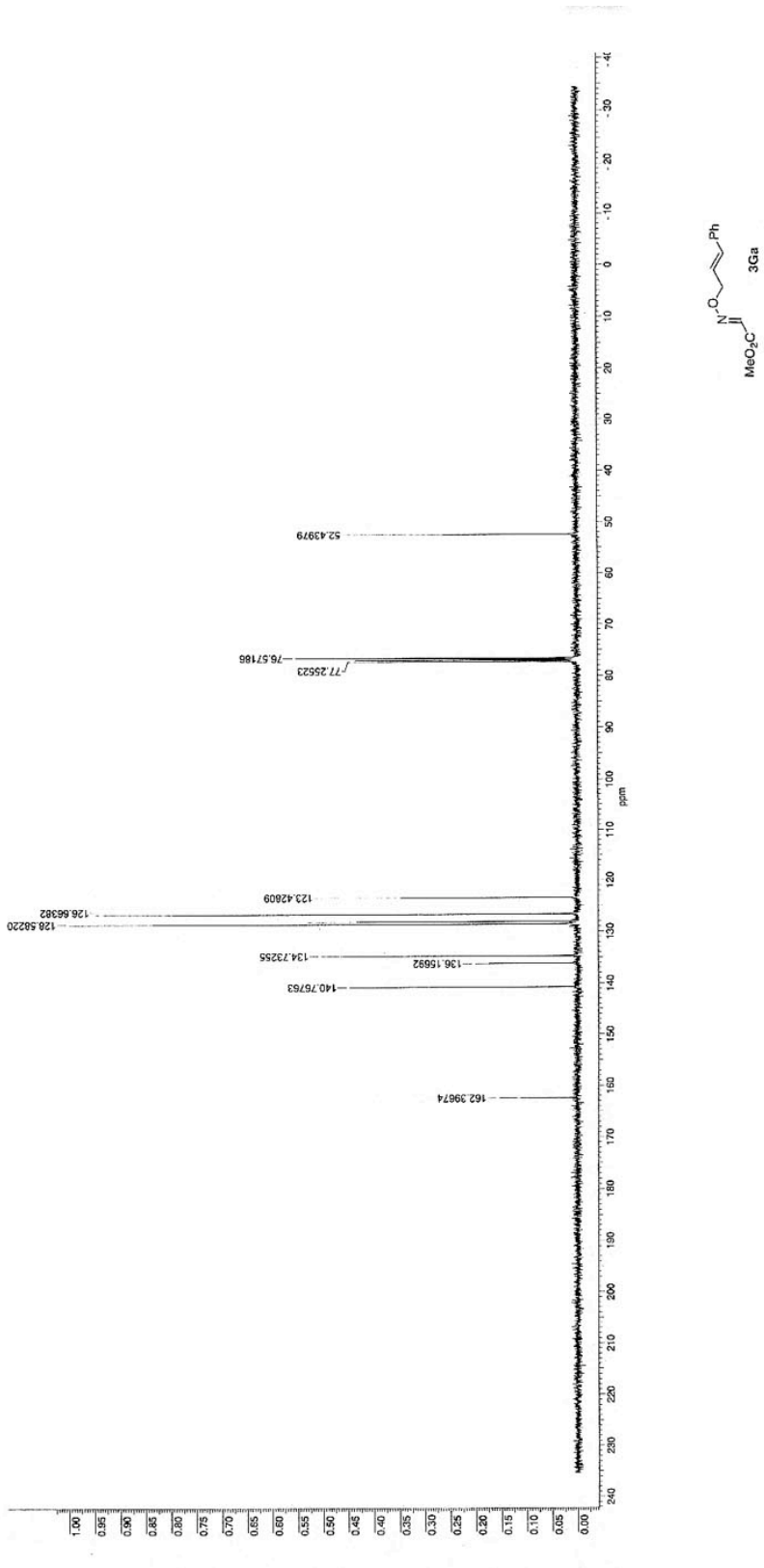

Ph
Ph
Ph
3Ea
4.85610
4.84390
M4
6.38142
6.39363
6.42598
6.43818
M3
6.62557
6.63322
M2
7.21335
7.22861
7.30430
7.31529
7.37615
7.39341
7.41539
7.48192
7.49718
1.00
ppm

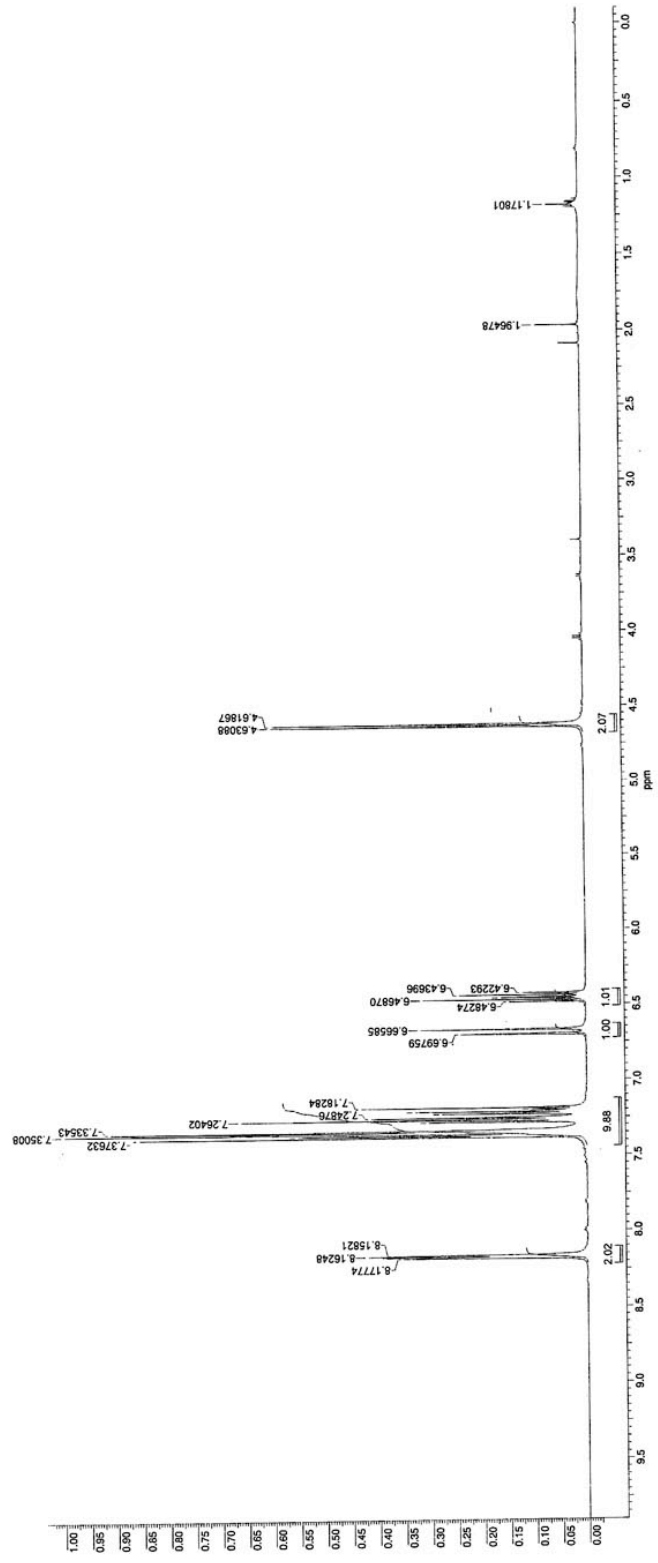

156.9791
136.8155
136.5932
133.3657
132.8470
129.3726
128.5492
127.9811
126.5814
125.7828
77.2562
76.9899
75.1958
ppm
Ph
N
O
Ph
Ph
3Ea

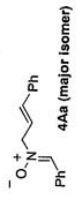

3Fa
Ph
O
N
M1
M2
M3
M4
M5
M6
M7
M8
M9
7.39769
7.38243
7.30186
7.28699
7.22312
7.20847
6.63228
6.60054
6.40034
6.38813
6.35640
6.34419
4.85398
4.67238
2.51350
2.48909
2.50130
2.24480
2.19977
2.21198
1.70965
1.69927
1.68584
1.67486
1.61136
1.58391
1.60100
ppm


160.54423
136.87323
132.47659
128.48340
126.51562
125.99929
73.74781
32.12801
25.72243
26.93224
3Fa
Ph
O
N
ppm

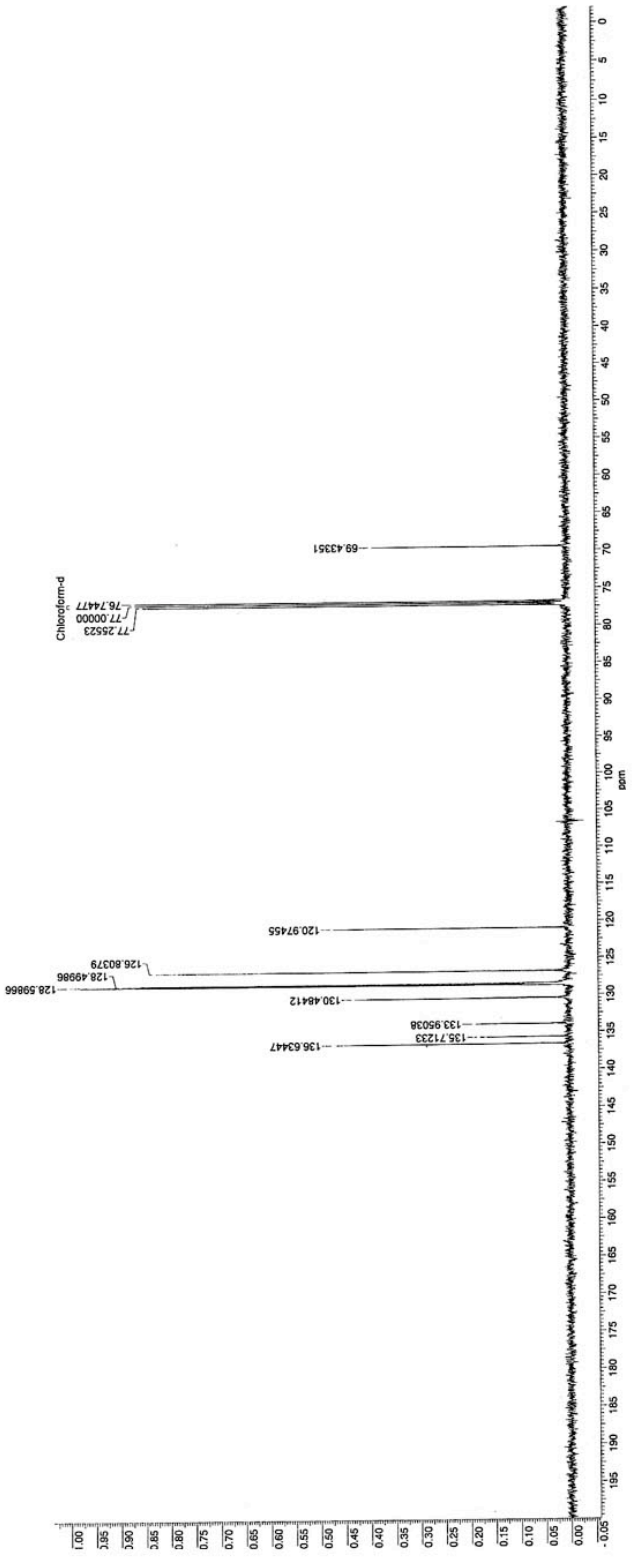

3Ga
MeO2C
Ph
M5 3.85266
M5 4.91409
4.92752
M3 6.65792
6.69027
M4 6.35762
6.37043
6.38386
6.33869
6.32588
M2 7.30613
7.32139
7.39036
7.40501
M1 7.56127
7.24387
7.25852
ppm

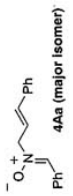

52.43979
76.57186
77.25523
123.42809
126.66362
128.58220
134.73255
136.15692
140.75763
162.39874
ppm
Ph
O
N
MeO2C
3Ga

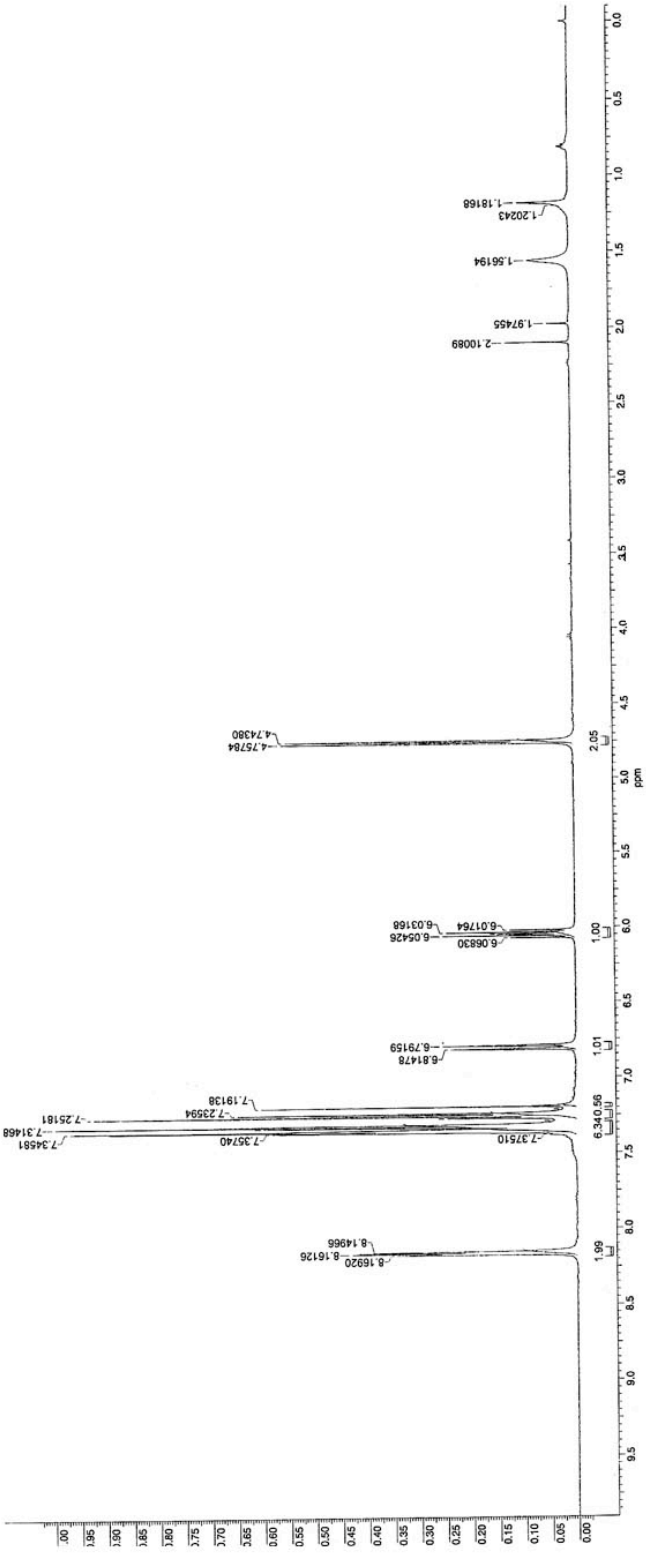


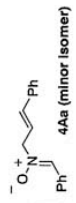


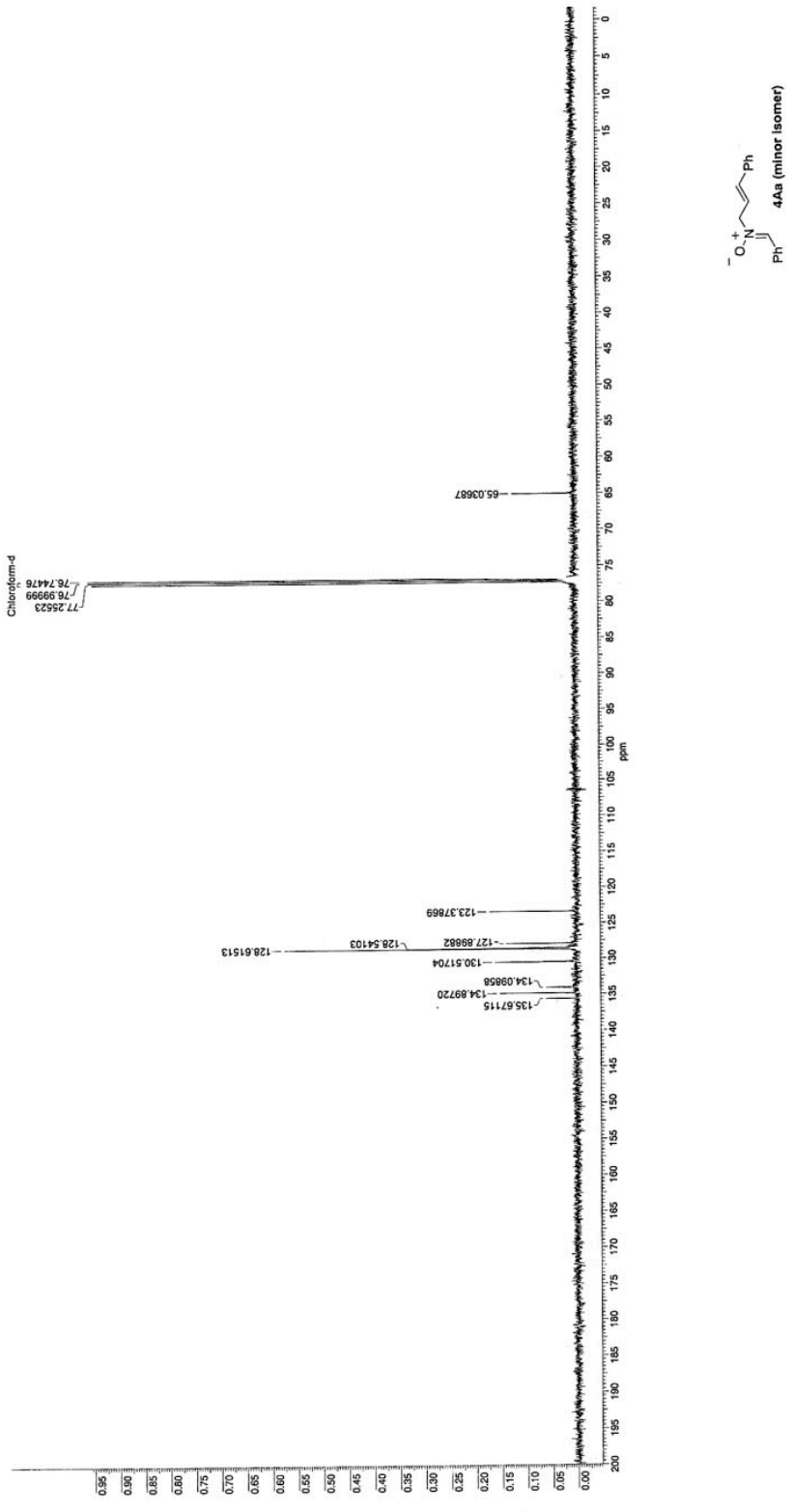
4Aa (major isomer)


69.43351
Chloroform-d
77.25523
77.00000
76.74477
120.97455
126.80379
128.49986
128.59866
130.48412
133.95038
135.71233
136.63447
ppm

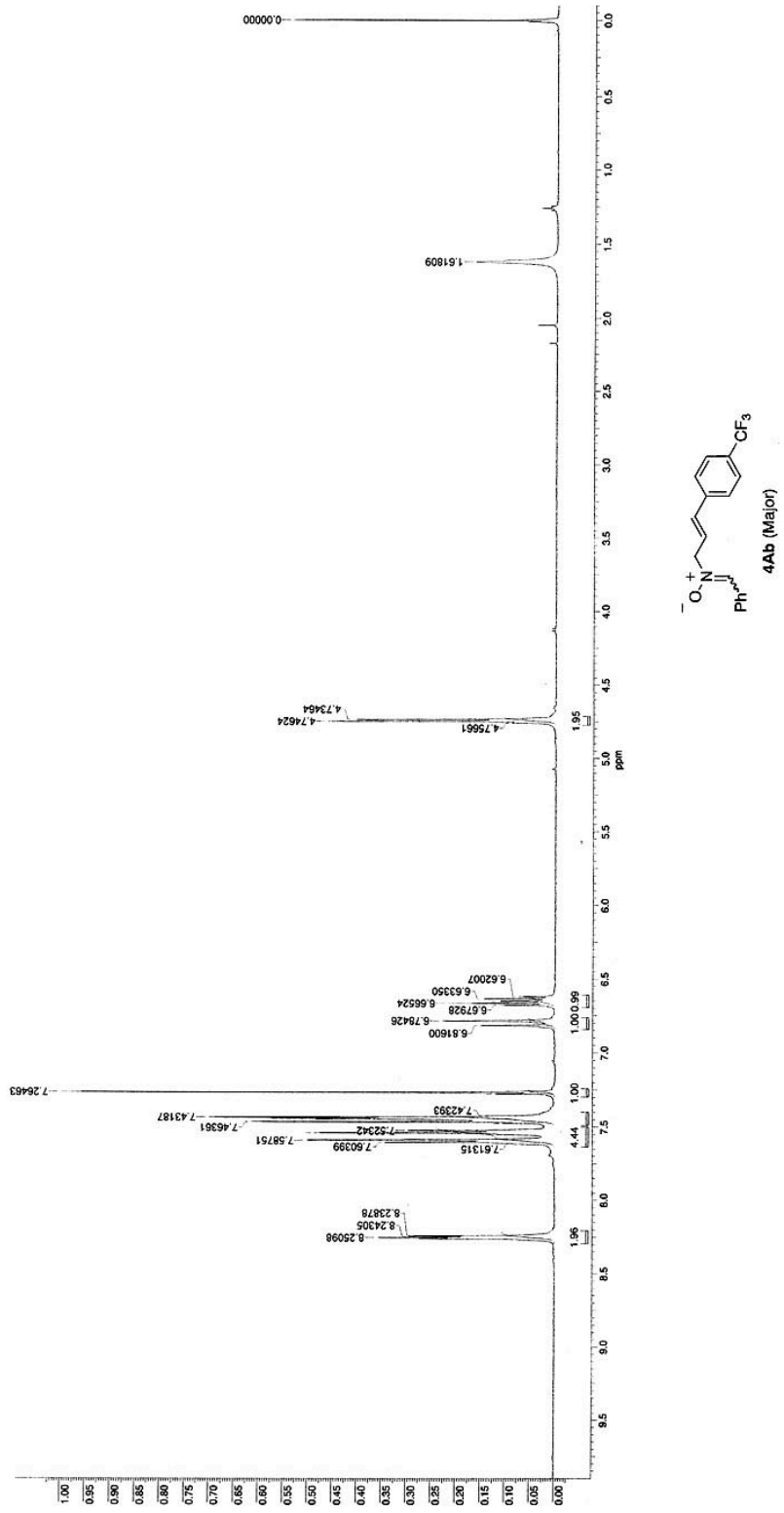

Ph
Ph
4Aa (major isomer)

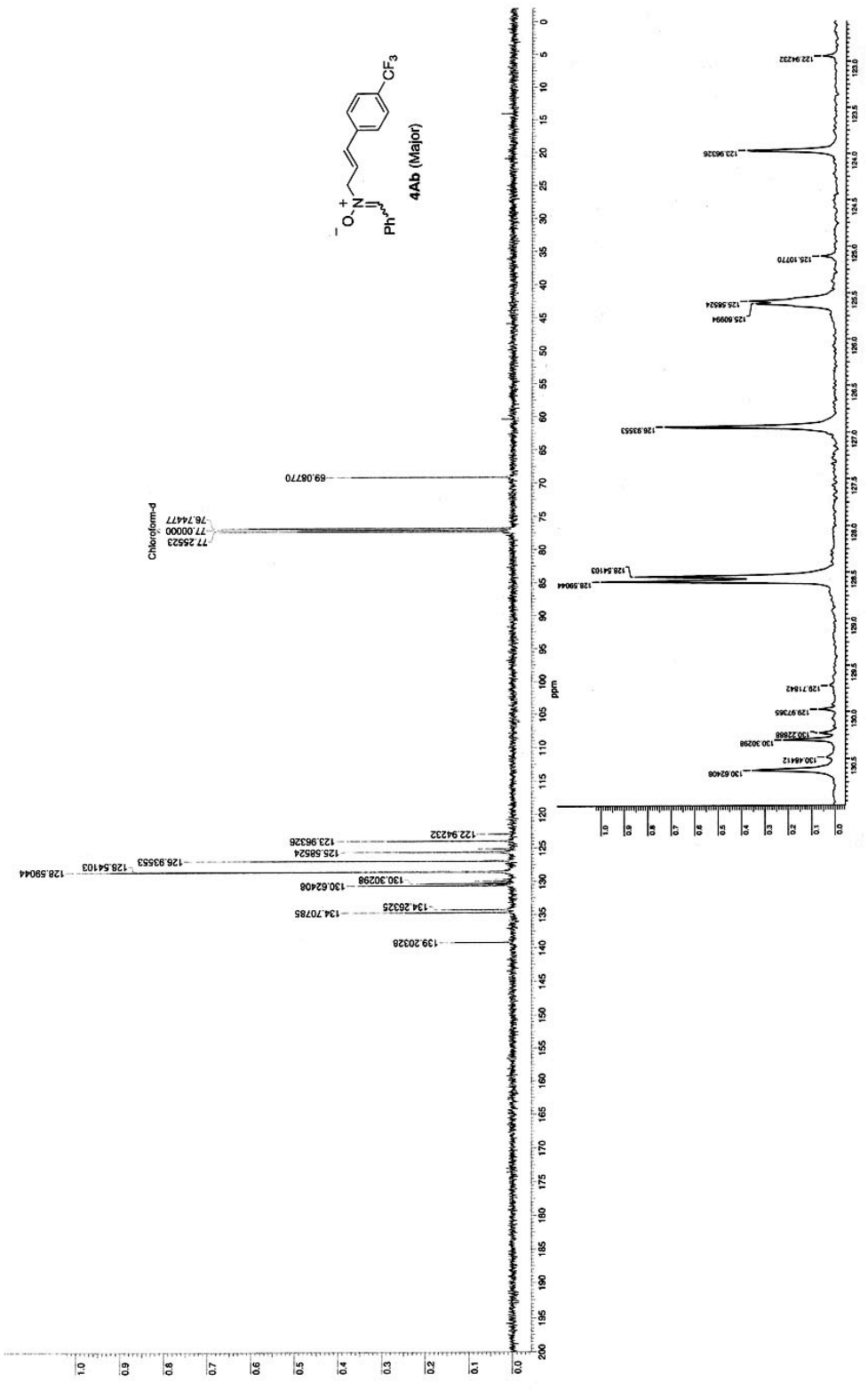

8.16920
8.16126
8.14966
7.37510
7.35740
7.34581
7.31468
7.25181
7.23594
7.19138
6.81478
6.79159
6.06830
6.05426
6.03168
6.01764
4.75784
4.74380
2.10089
1.97455
1.56194
1.20243
1.18168
6.34.56
2.05
1.00
1.01
1.99
ppm

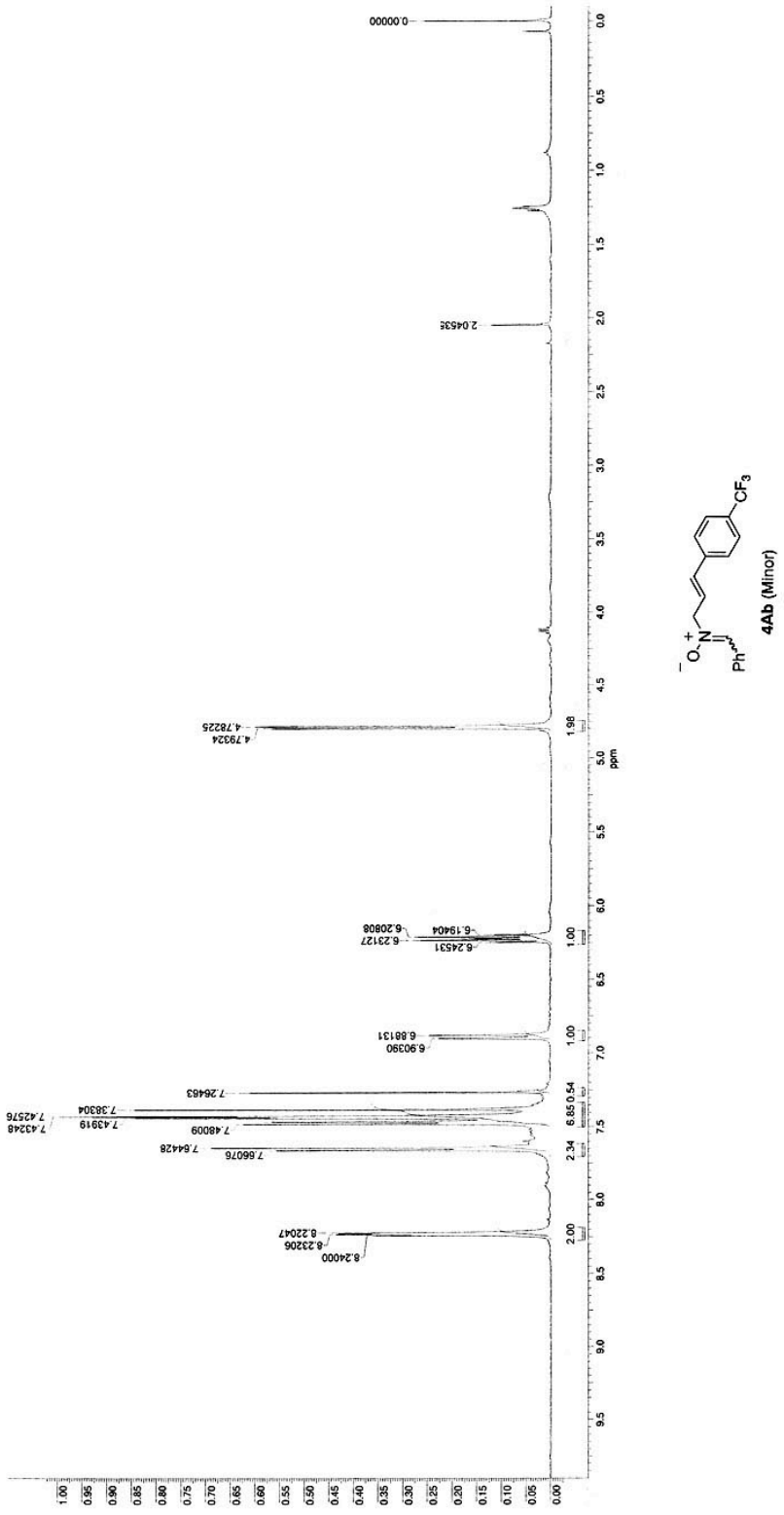

Ph
Ph
4Aa (minor isomer)

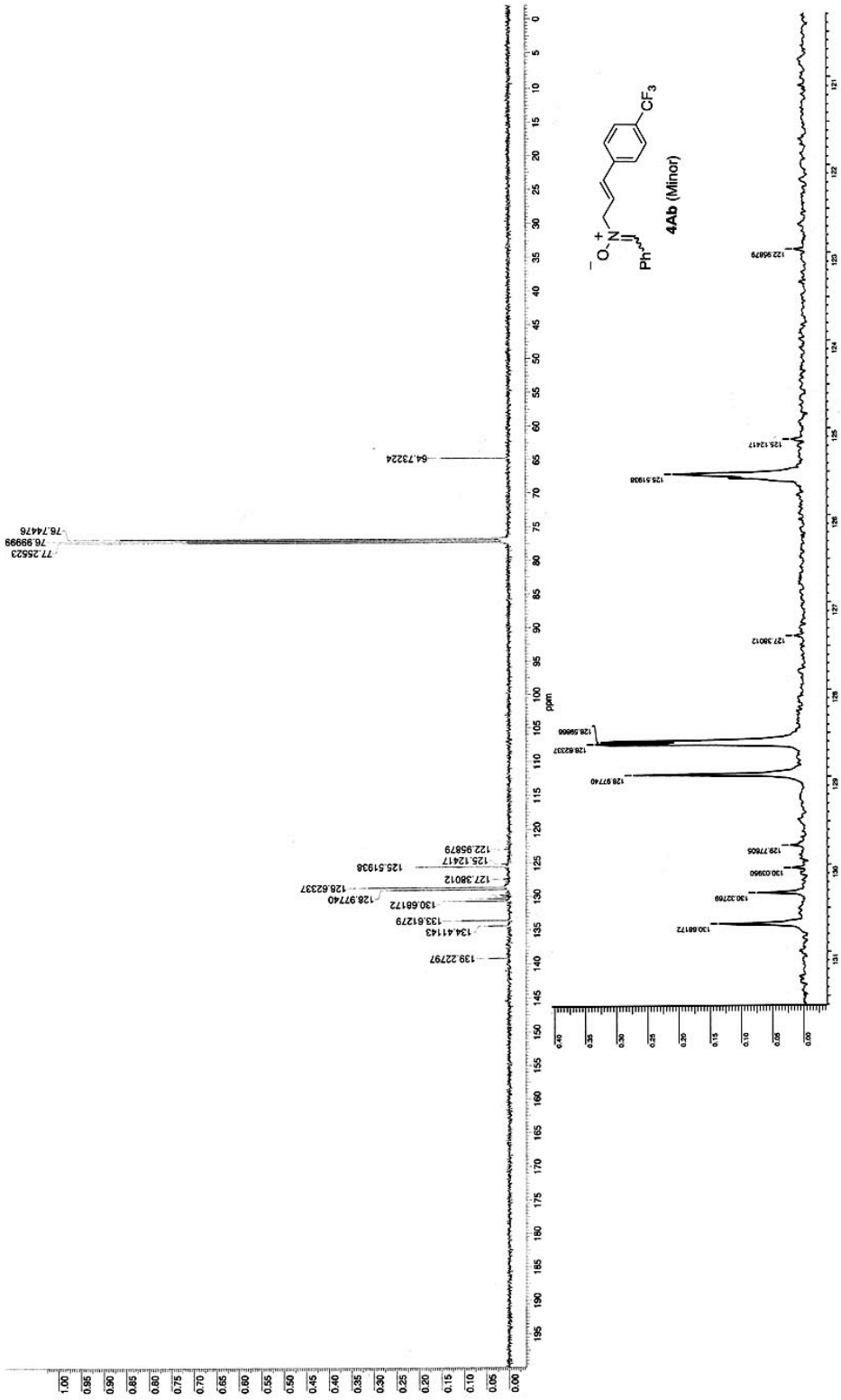


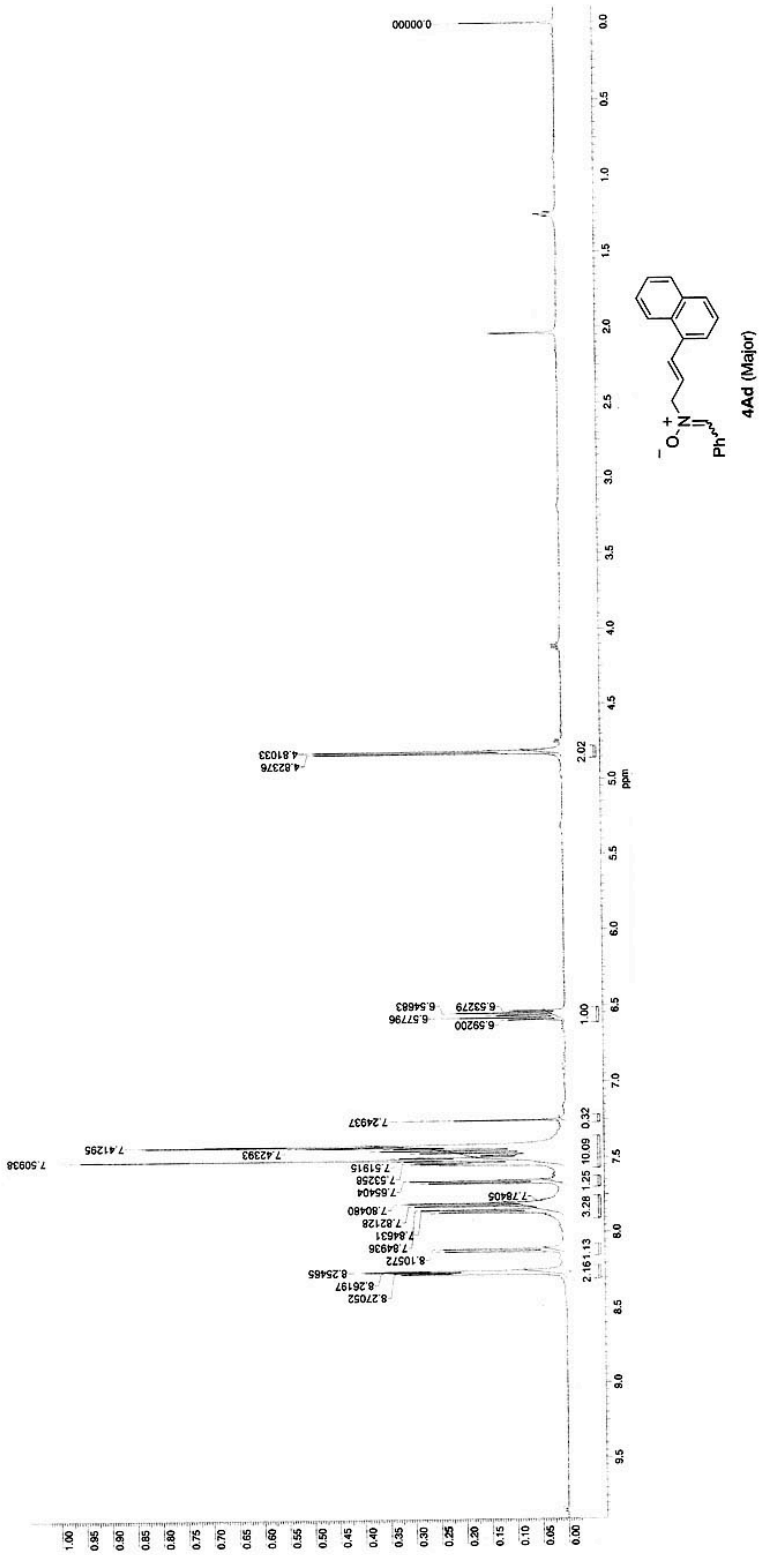
4Aa (minor isomer)

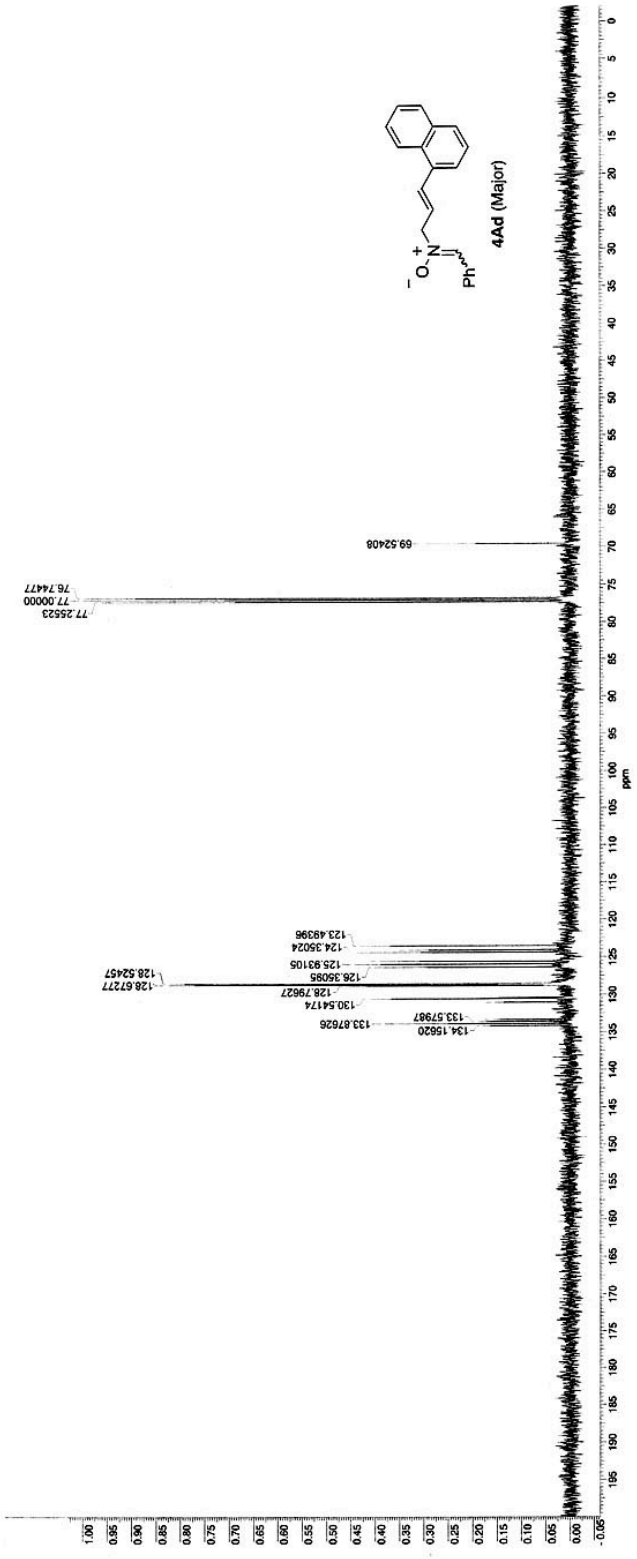

0.00000
1.61809
4.73464
4.74624
4.75661
6.62007
6.63350
6.66524
6.67928
6.79426
6.81600
7.26463
7.42393
7.43187
7.46361
7.52342
7.58751
7.60399
7.61315
8.23878
8.24305
8.25098
ppm
4Ab (Major)

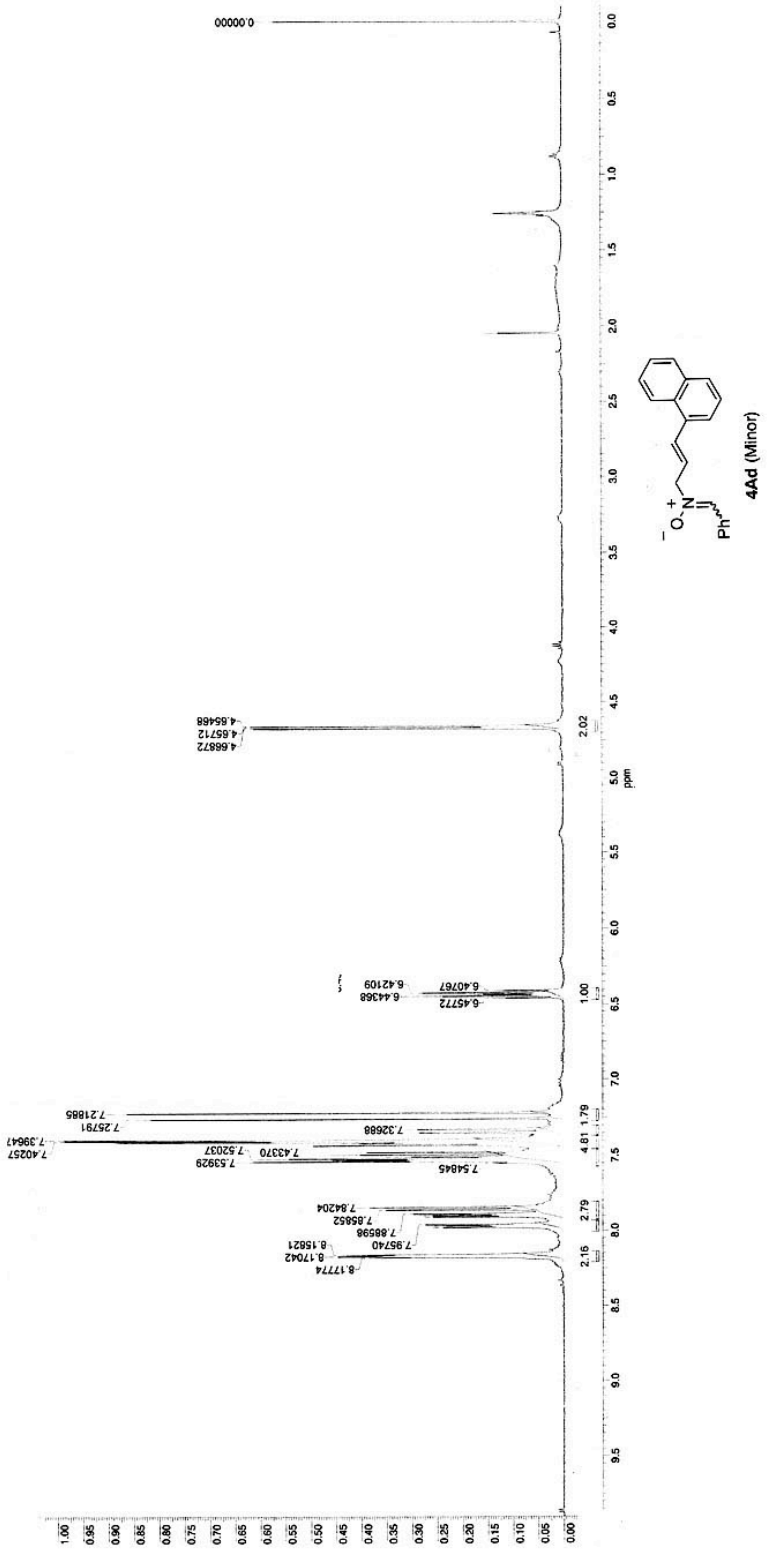

4Ab (Major)
Chloroform-d
77.25523
77.00000
76.74477
69.08770
139.20328
134.26325
134.70785
130.30298
130.62408
128.59044
128.54103
126.93553
125.58524
123.96326
122.94232
ppm

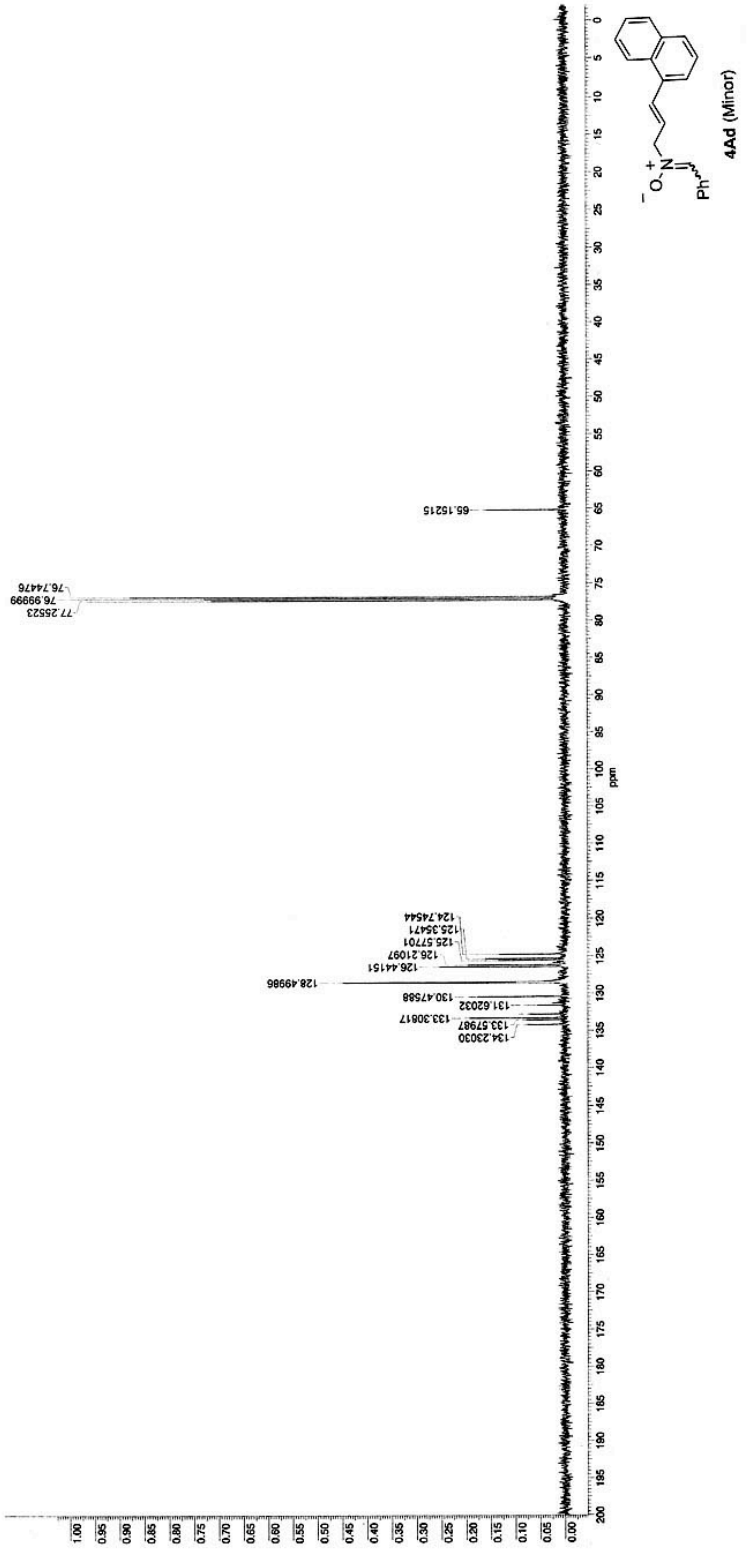

0.00000
2.04535
4.79324
4.78225
6.19404
6.20808
6.23127
6.24531
6.88131
6.90390
7.26463
7.38304
7.42576
7.43248
7.43919
7.48009
7.64428
7.66076
8.22047
8.22306
8.24000
ppm
4Ab (Minor)

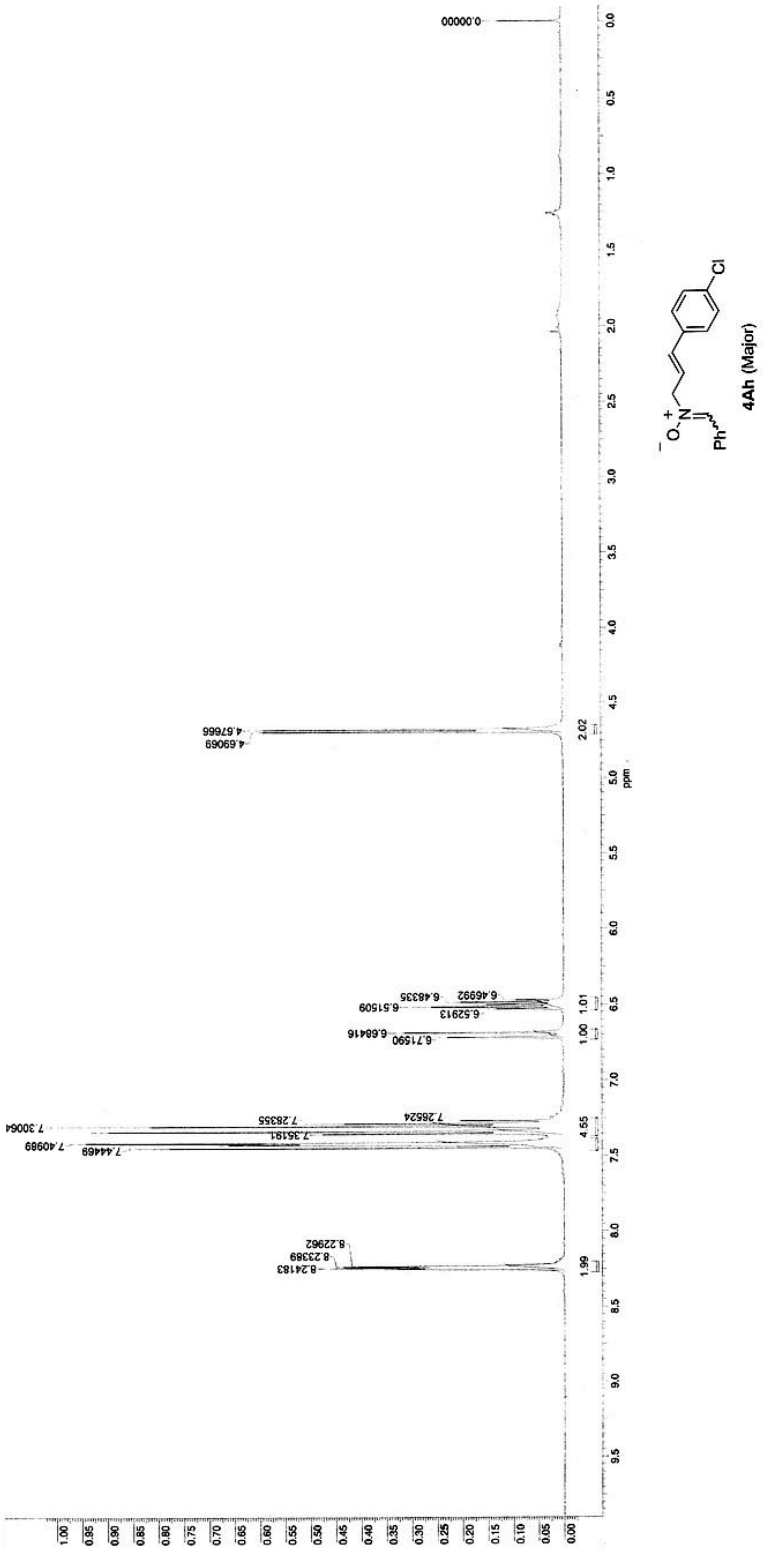

4Ab (Minor)
CF3
Ph
ppm

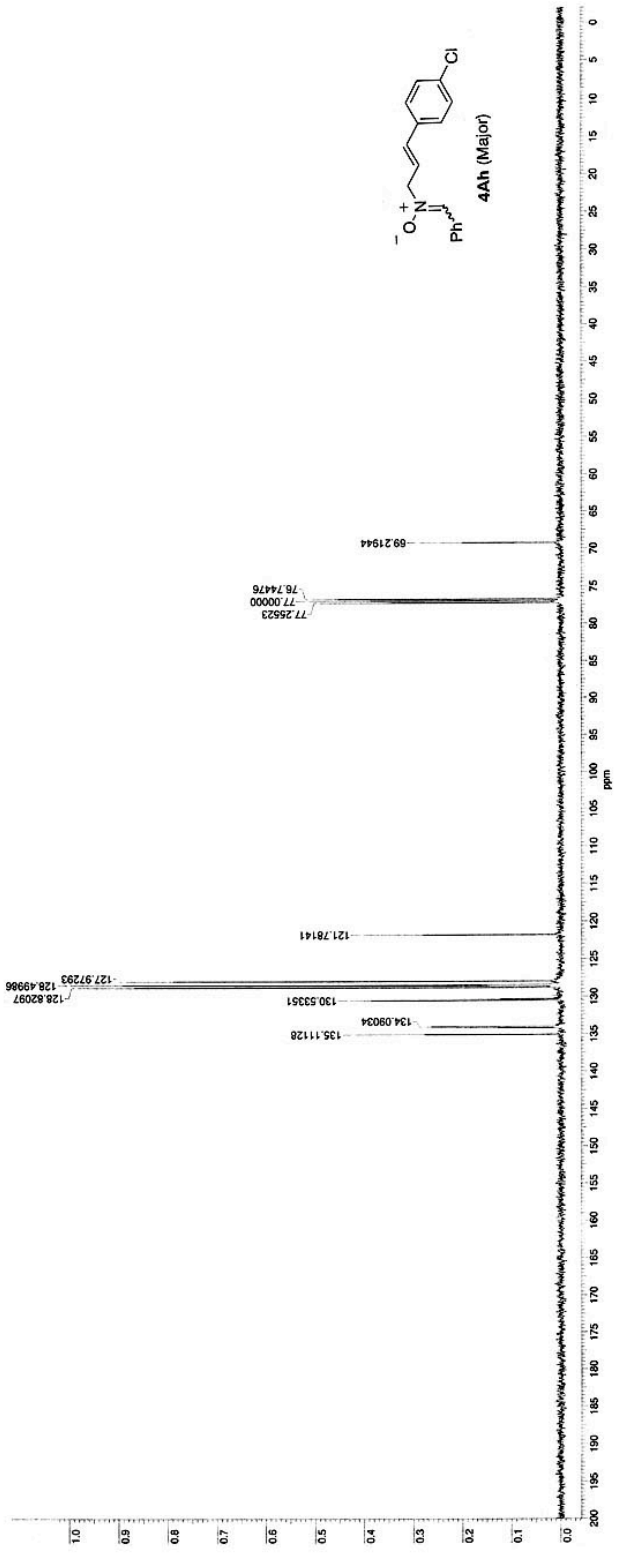

4Ad (Major)
Ph
N
O
0.00000
4.82376
4.81033
6.59200
6.57796
6.54683
6.53279
7.24937
7.42383
7.41295
7.50938
7.51915
7.53258
7.65404
7.78405
7.80480
7.82128
7.84531
7.84936
8.10572
8.25485
8.26197
8.27052
2.02
1.00
3.28
1.25
10.09
0.32
2.16
1.13
ppm

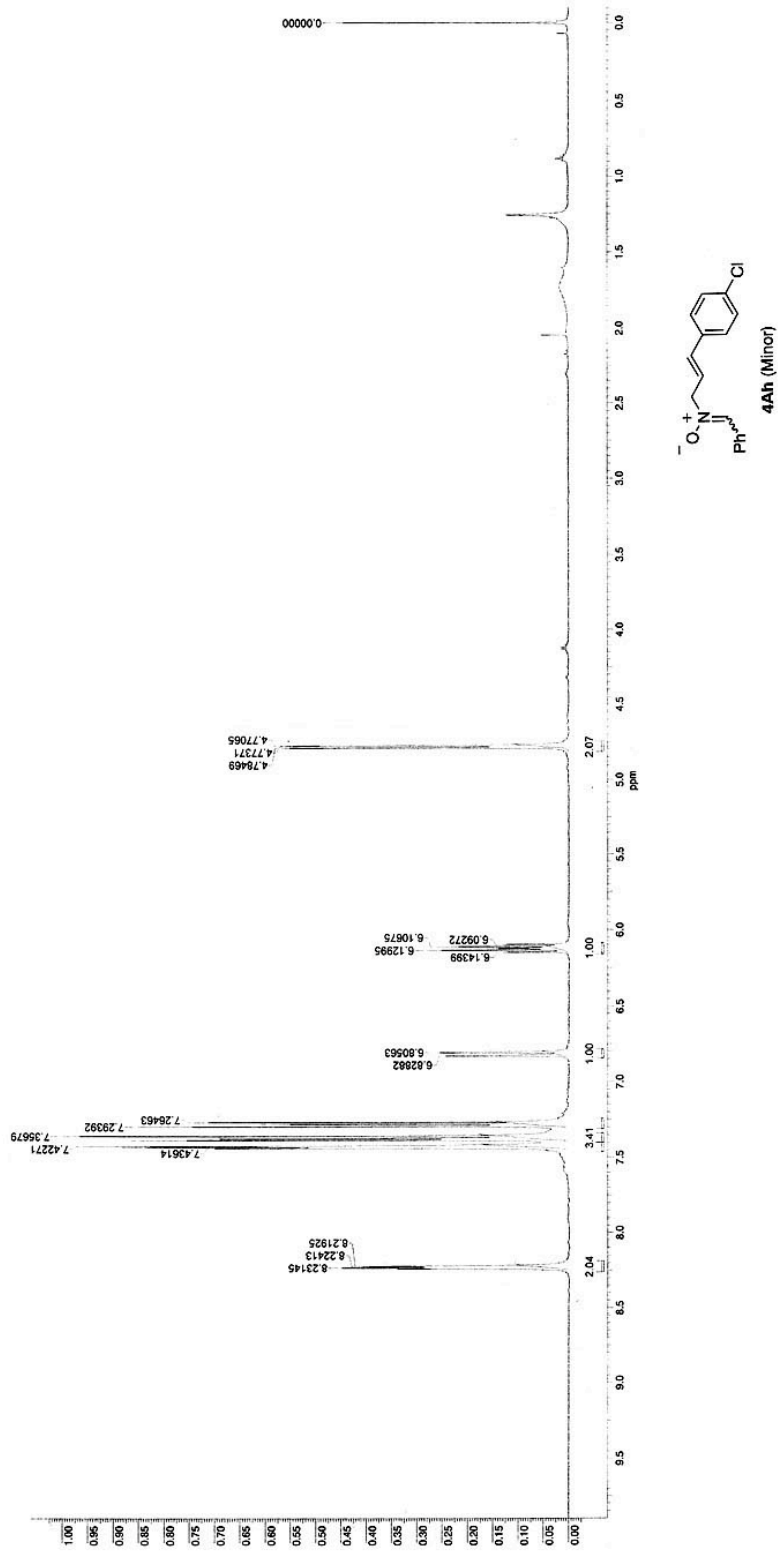

4Ad (Major)
69.52408
77.25523
77.00000
76.74477
128.67277
128.52457
126.36085
125.83105
124.35024
123.48396
133.87626
133.57987
134.15620
130.54174
128.79827
ppm

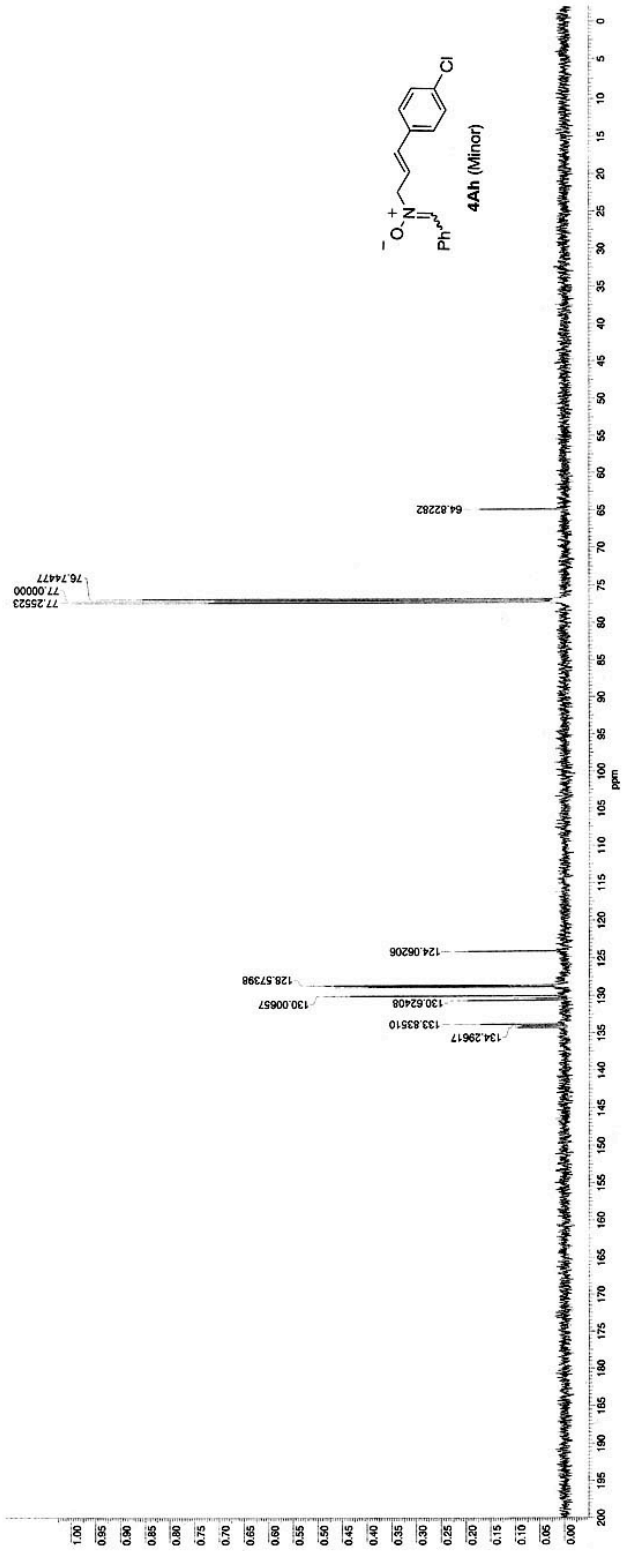

4Ad (Minor)
ppm


4Ad (Minor)
65.15215
77.25523
76.99999
76.74476
128.49986
126.44151
126.21097
125.57701
125.35471
124.74544
134.23030
133.57987
133.30817
131.62032
130.47588
ppm


0.00000
4.69069
4.57666
6.46992
6.46335
6.52913
6.51509
6.71590
6.68416
7.36524
7.28355
7.35181
7.44468
7.40989
7.30064
8.24183
8.23389
8.22962
ppm
4Ah (Major)
Cl
Ph


4Ah (Major)
Cl
Ph
69.21944
77.25623
77.00000
76.74476
121.78141
127.97293
128.49986
128.82097
130.53351
134.09034
135.11128
ppm


0.00000
4.78469
4.77271
4.77065
6.14399
6.12995
6.10675
6.09272
6.82862
6.80563
8.23145
8.22413
8.21925
7.43614
7.42271
7.35679
7.29392
7.26463
ppm
Cl
Ph
4Ah (Minor)


4Ah (Minor)
64.82282
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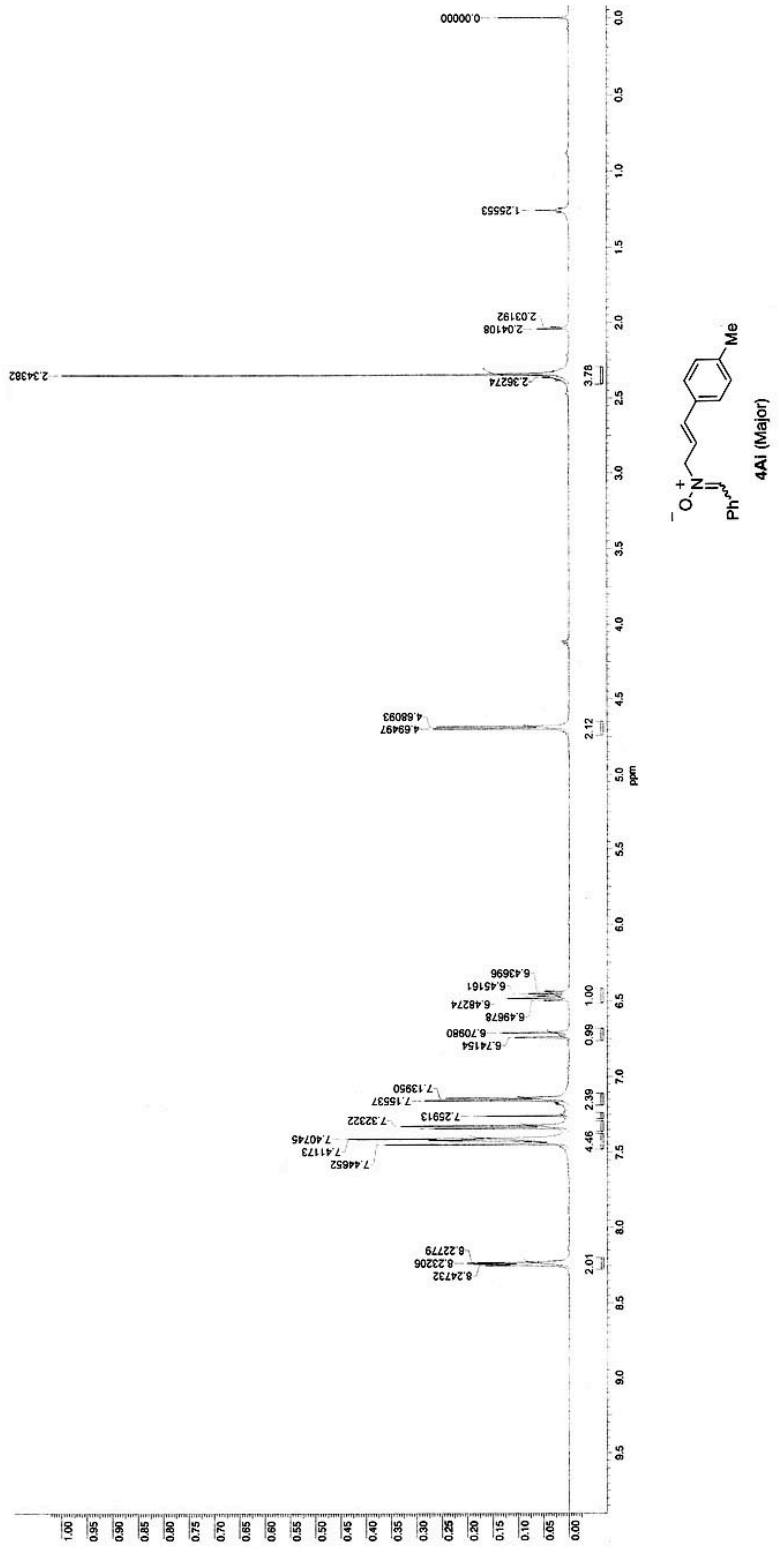
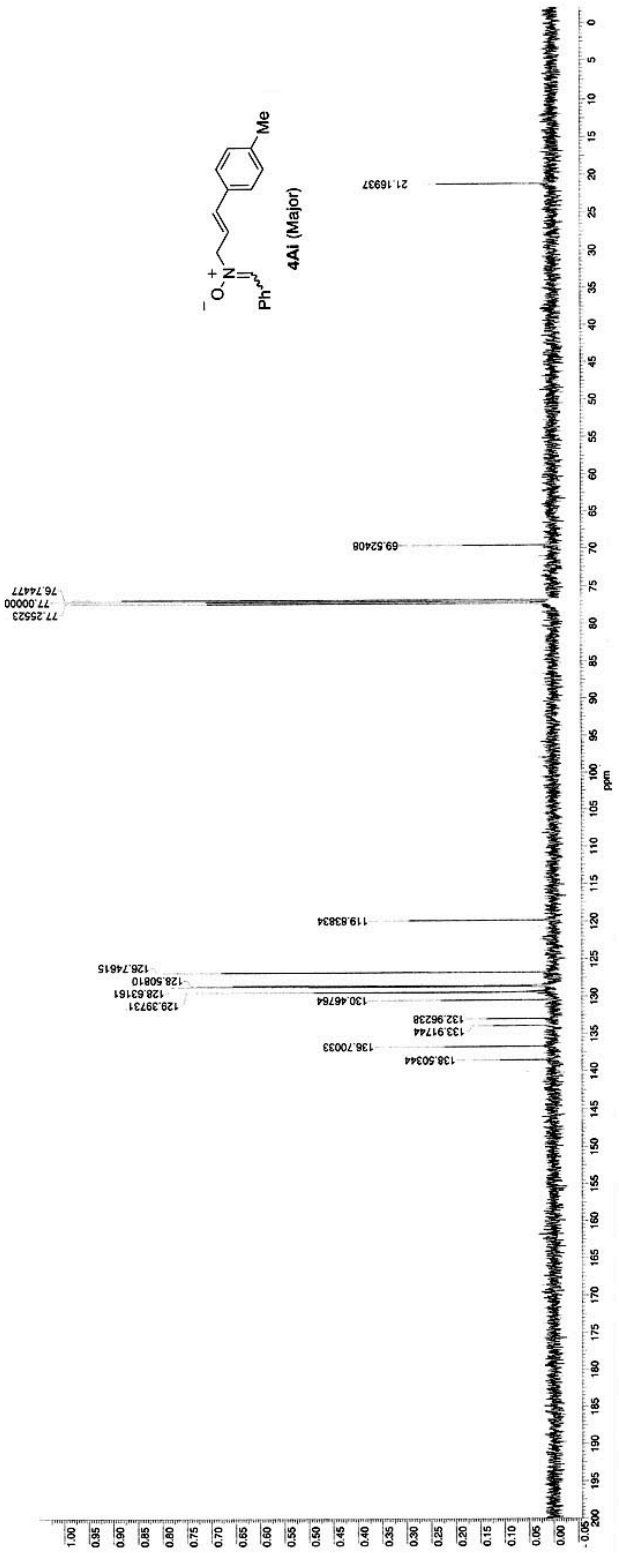
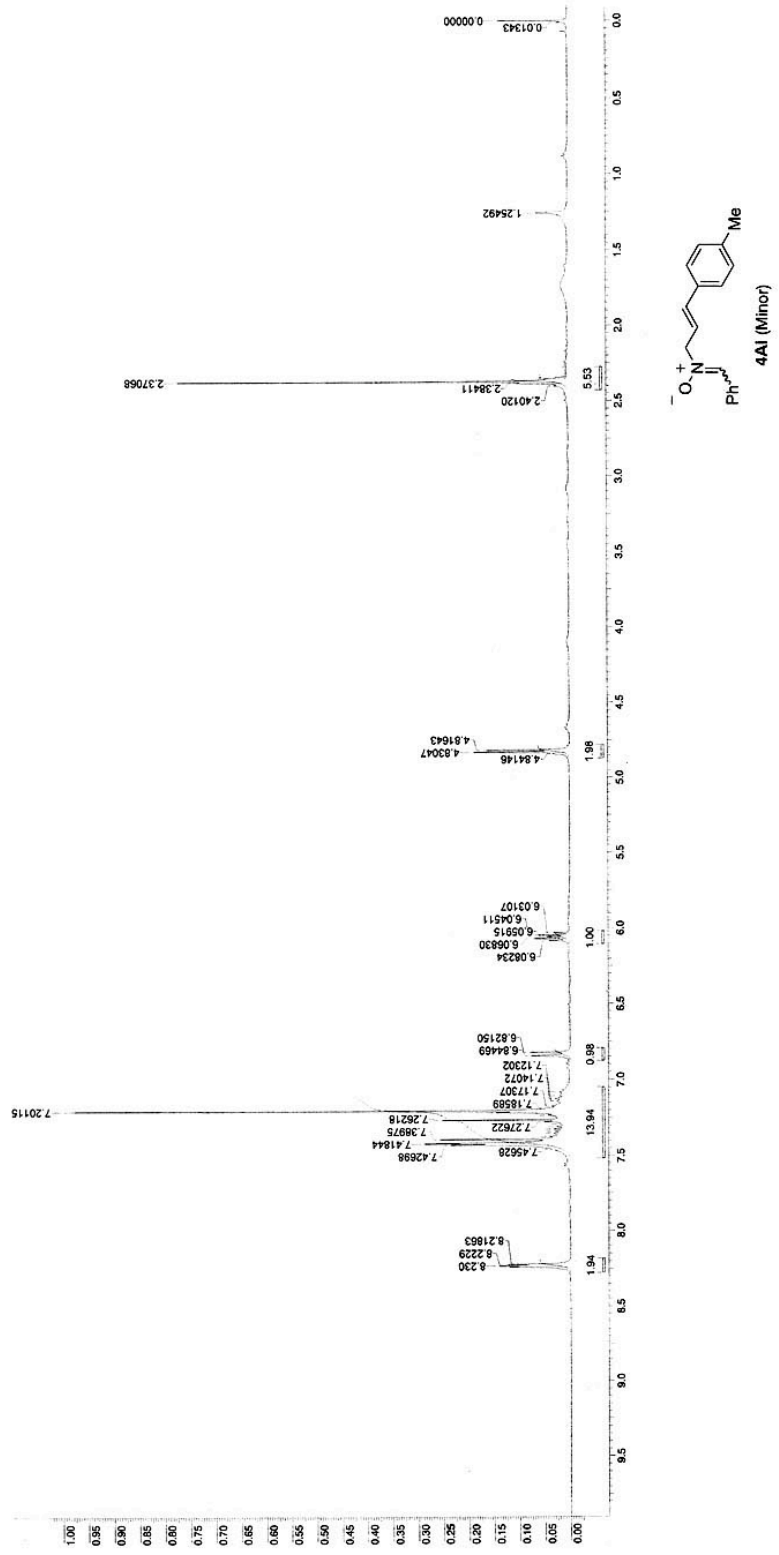
77.00000
76.74477
124.06206
128.57398
130.00657
130.62408
133.83510
134.29617
ppm


0.00000
1.25553
2.04108
2.03192
2.34382
2.36274
4.69497
4.68083
6.43656
6.45161
6.48274
6.49678
6.70980
6.74154
7.13950
7.15537
7.25913
7.32322
7.40745
7.41173
7.44652
8.22779
8.23206
8.24732
3.78
2.12
1.00
0.99
2.39
4.46
2.01
ppm
Me
Ph
4Ai (Major)


Me
4Ai (Major)
Ph
21.16937
69.52408
77.25523
77.00000
76.74477
119.83834
128.74615
128.50810
128.63161
129.39731
130.46764
132.96238
133.91744
138.70033
138.50344
ppm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6.06830
6.08234
6.82150
6.84469
7.12302
7.14072
7.17307
7.18589
7.20115
7.26218
7.27622
7.38975
7.41844
7.42698
7.45628
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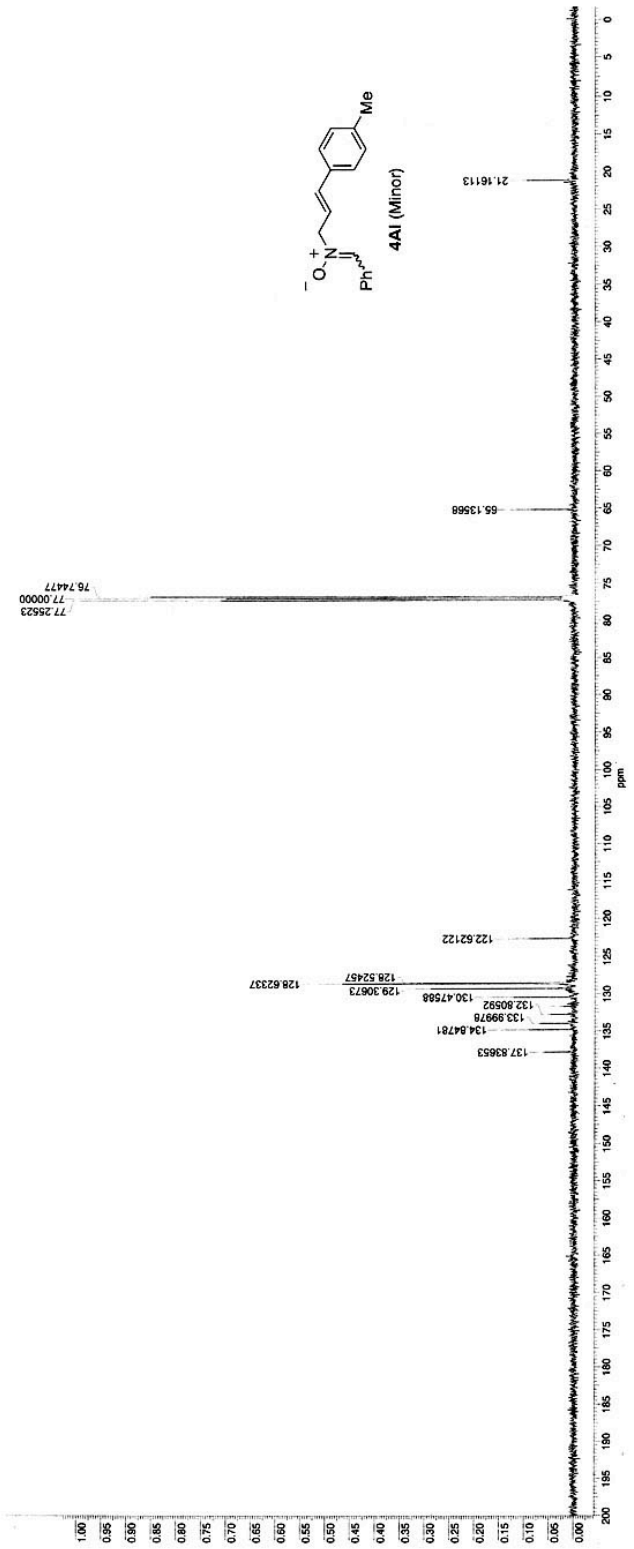
8.230
5.53
1.98
1.00
0.98
13.94
1.94
Me
Ph
4Ai (Minor)


Me
Ph
4Al (Minor)
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77.25523
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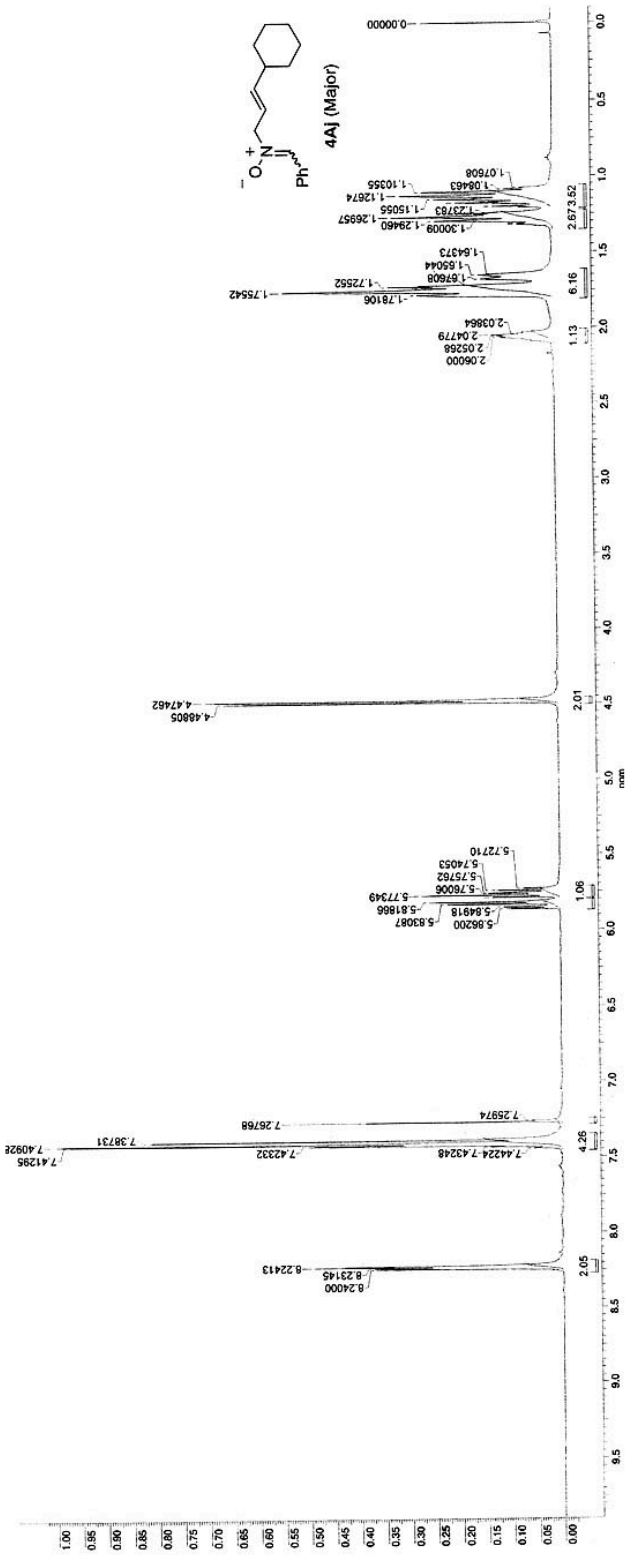
4Aj (Major)
Ph
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8.22413
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7.40928
7.38731
7.28768
7.25974
5.86200
5.84918
5.83087
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5.76006
5.75762
5.74053
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4.48805
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2.06000
2.05268
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1.64373
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1.12674
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1.07606
1.03555
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4.26
1.06
5.00
2.01
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3.62
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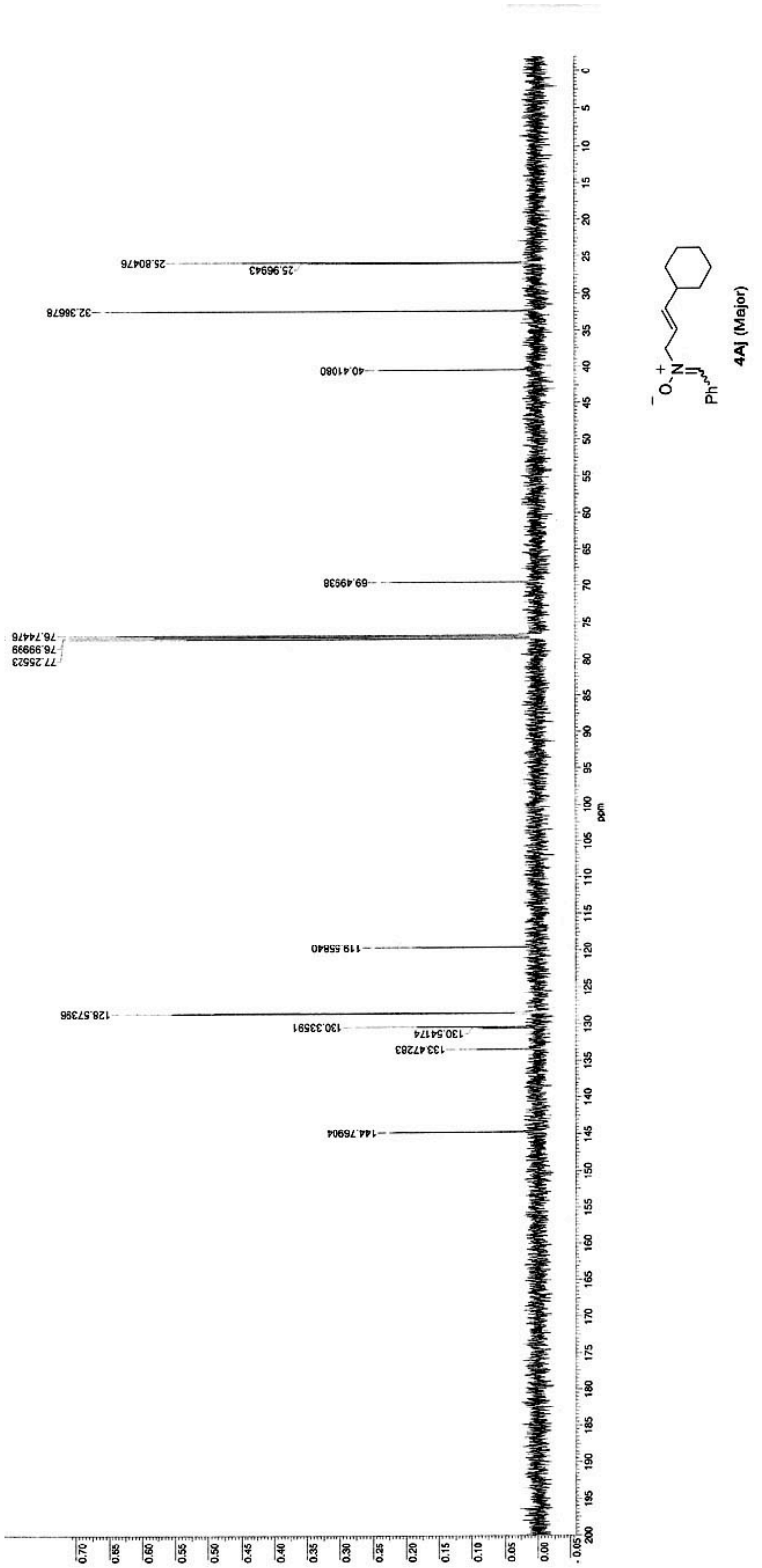
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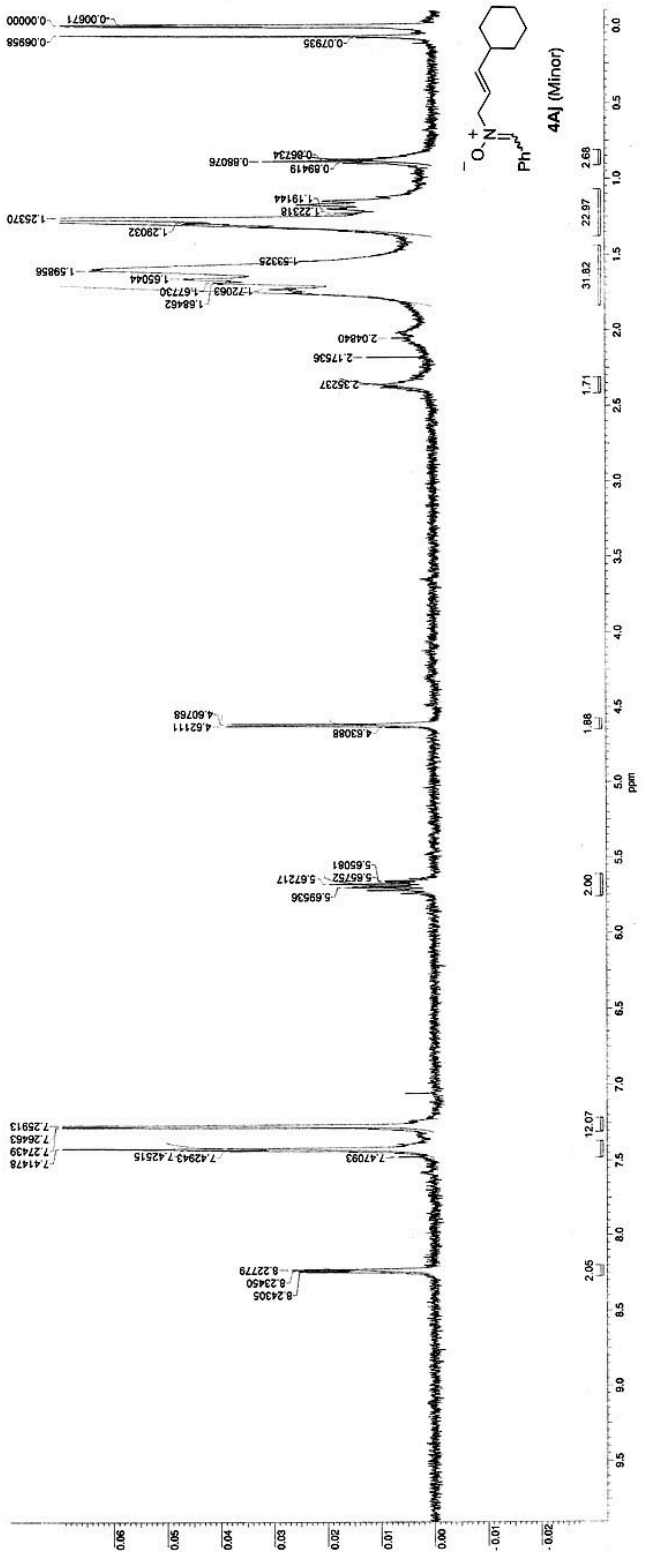
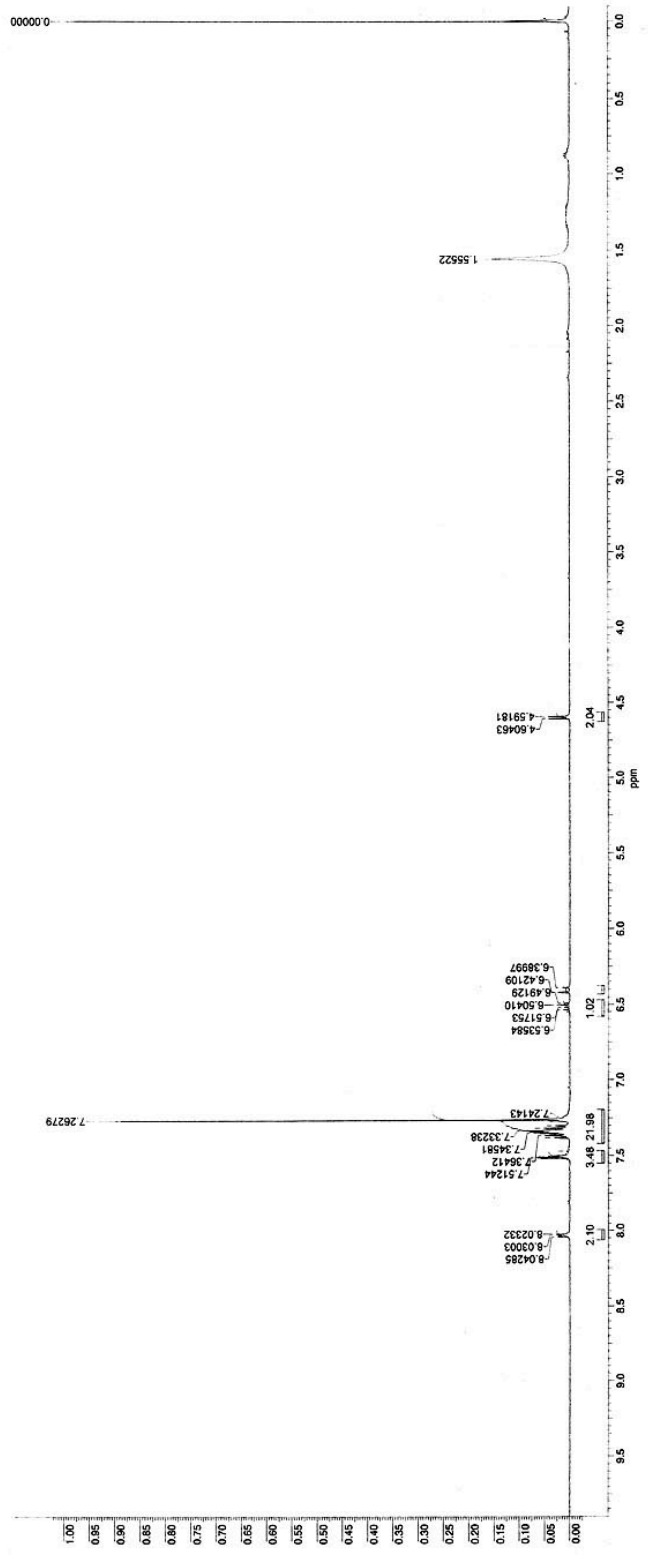
144.75904
ppm
4AJ (Major)
Ph


4Aj (Minor)
Ph
ppm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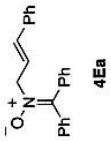
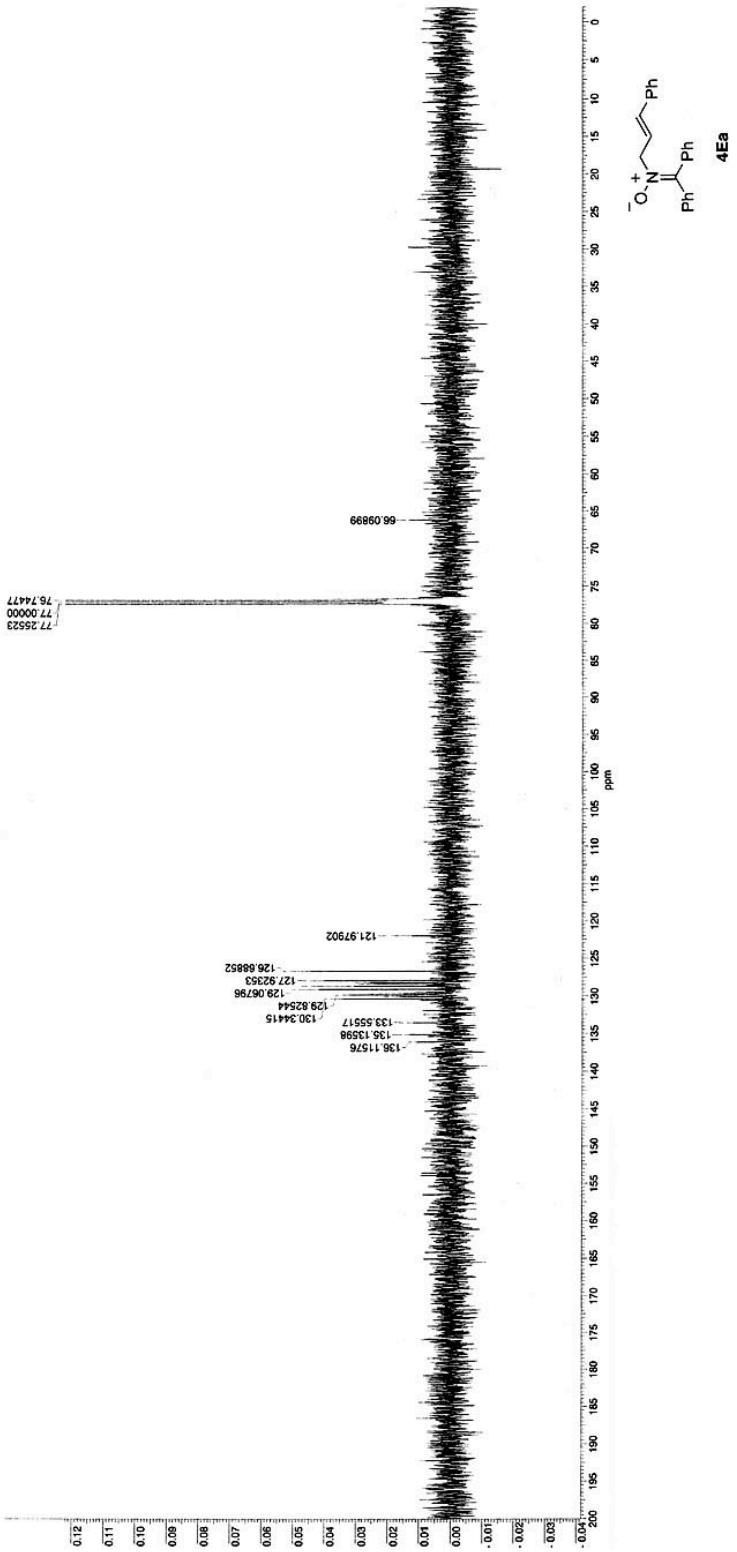
ppm


Ph
Ph
Ph
4Ea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77.00000
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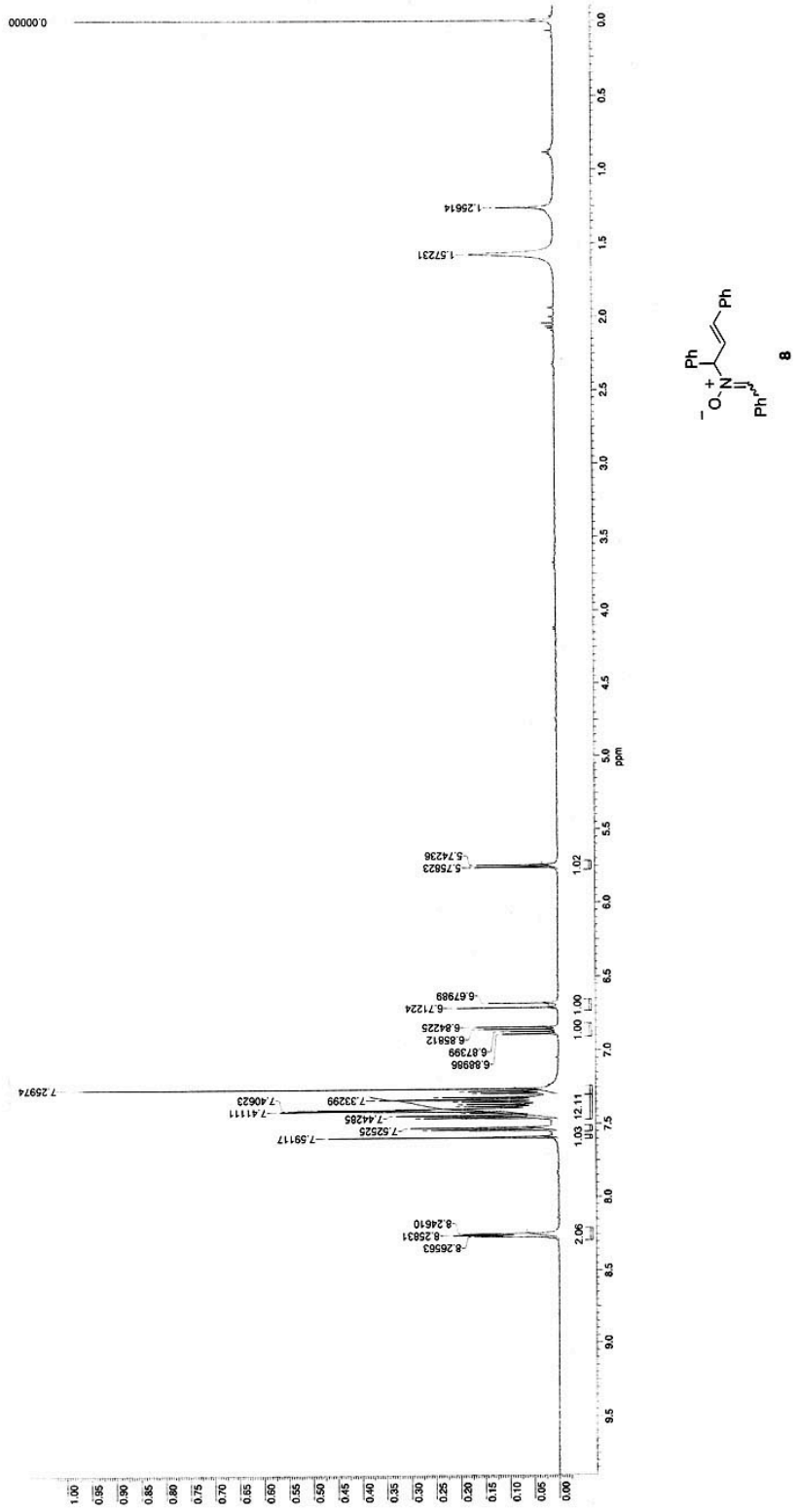
133.55517
135.13598
136.11576
ppm
Ph
Ph
Ph
4Ea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6.85812
6.87399
6.88986
7.25974
7.33299
7.40623
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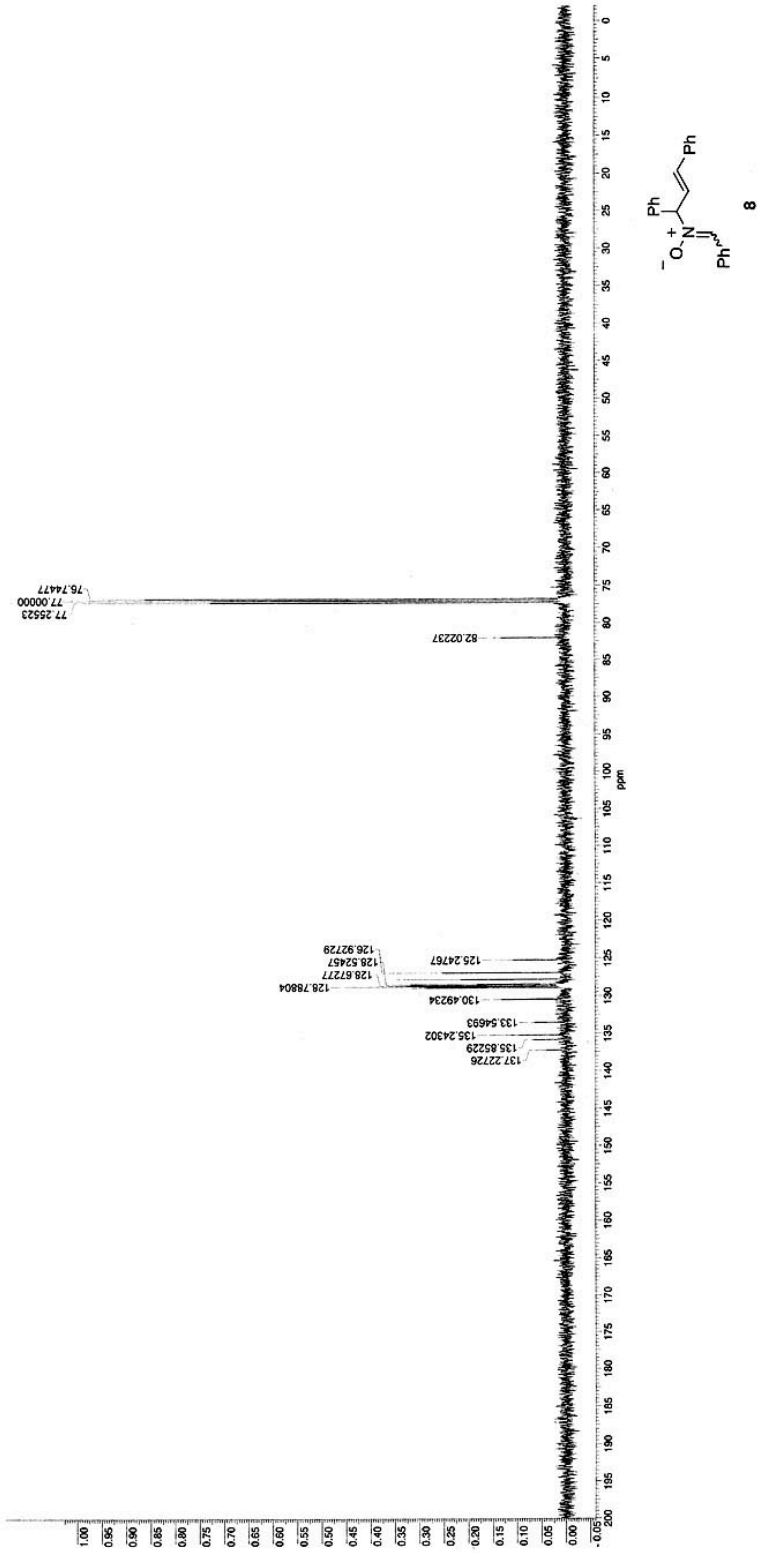
7.44285
7.52525
7.59117
8.24610
8.25331
8.26563
Ph
Ph
Ph
8
ppm


Ph
Ph
Ph
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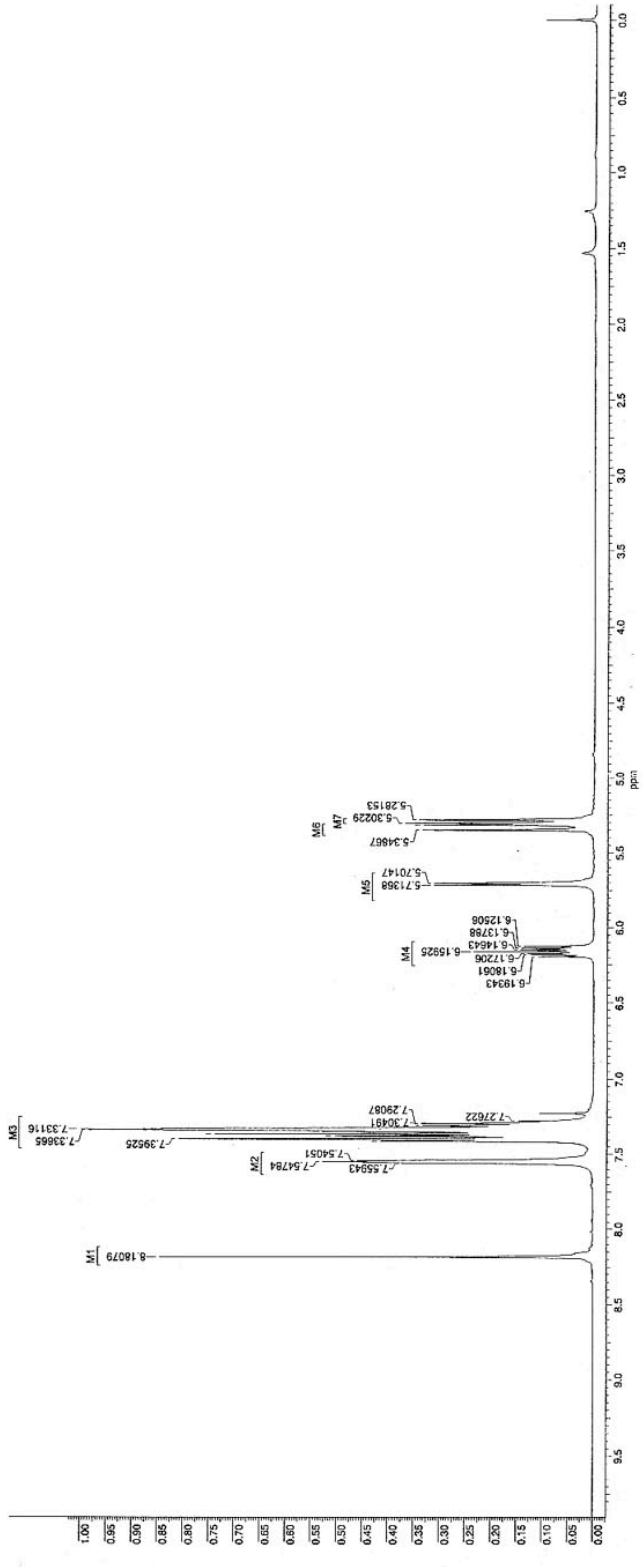
ppm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6.19343
6.18061
6.17206
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6.13788
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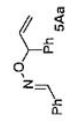
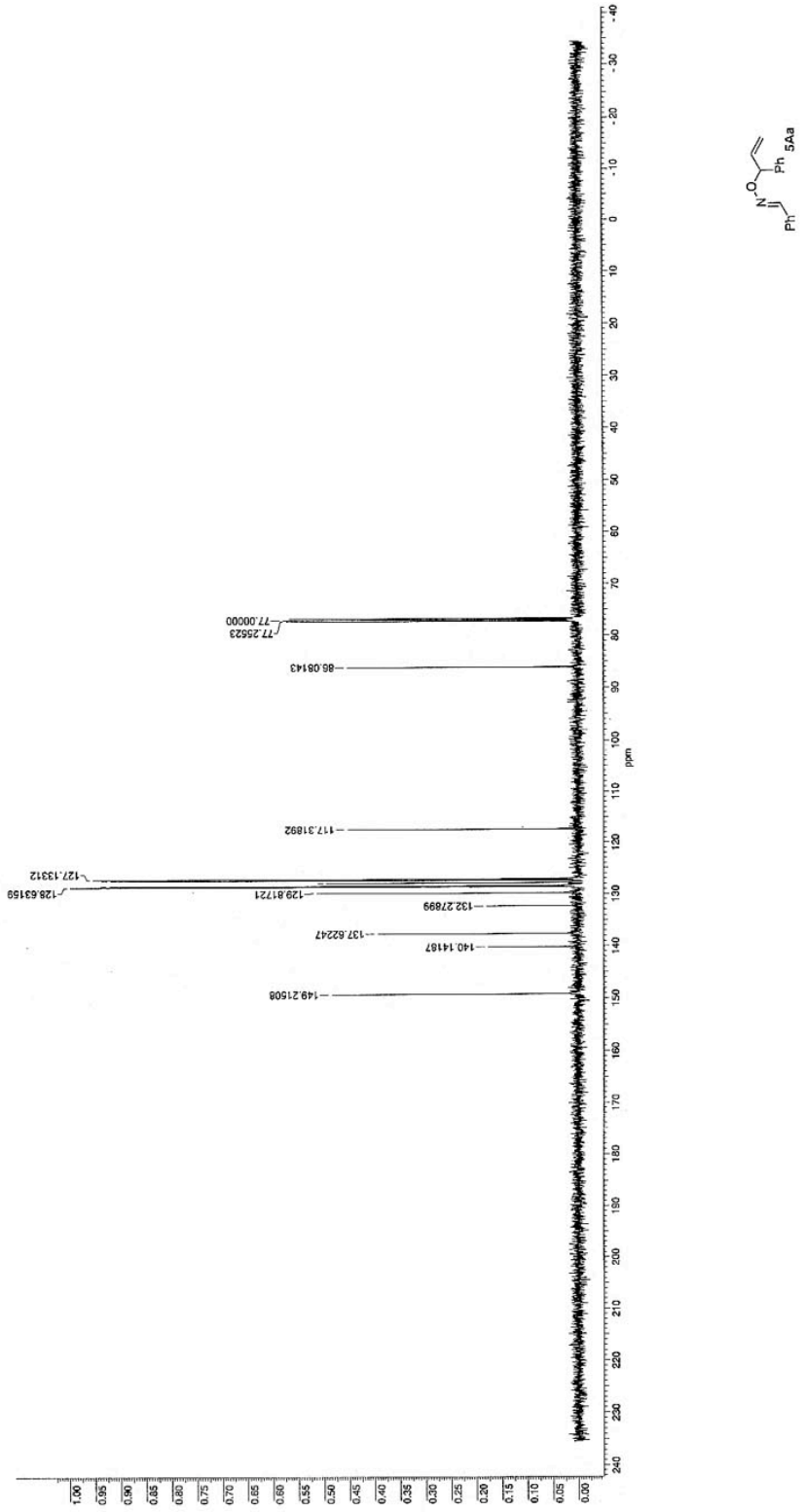
5.28153
M1
M2
M3
M4
M5
M6
M7
ppm


Ph
5Aa
N
O
Ph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77.00000
86.08143
117.31892
127.13312
128.63159
129.81721
132.27899
137.62247
140.14187
149.21508
ppm
5Aa
Ph
Ph