Electronic Supplementary Information

Stereoselective synthesis of 1,6-diazecanes by tandem aza-Prins type dimerization and cyclization process

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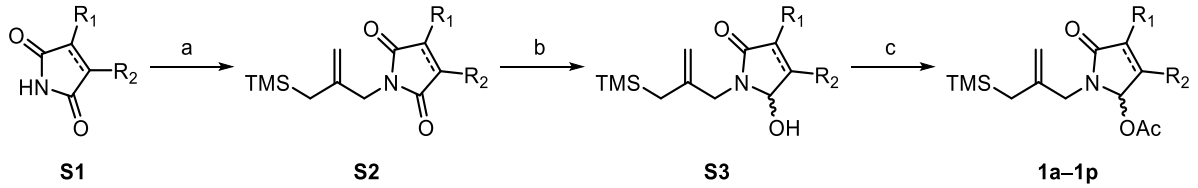
1. Experimental Procedures

1) General Information

All reactions were conducted using oven-dried glassware under an atmosphere of argon (Ar_2). Commercially available reagents were purchased from Merck, TCI, Thermo Fisher Scientific, Combi-blocks and used without further purification. Reactions were followed by thin layer chromatography (TLC) analysis using Merck pre-coated silica gel 60 F_{254} plates. Visualization on TLC was achieved by the use of UV light and treatment with acidic anisaldehyde or ceric ammonium molydate, $KMnO_4$ stain followed by heating. Flash column chromatography was carried out using silica gel (60, 0.040-0.060mm). The ^1H NMR , ^{13}C NMR, ^{19}F NMR spectra were measured with Brucker Avance III HD (400 MHz) and Agilent Technologies DD2 (600 MHz). The ^1H NMR chemical shifts are expressed in parts per million (δ) downfield to $CDCl_3$ resonance (δ = 7.26), CD_3OD resonance (δ = 3.31), $(CD_3)_2CO$ resonance (δ = 2.05). The ^{13}C NMR chemical shifts are expressed in parts per million (δ) relative to the central $CDCl_3$ resonance (δ = 77.16), CD_3OD resonance (δ = 49.00), $(CD_3)_2CO$ resonance (δ = 29.84 or 206.26). The ^{19}F NMR chemical shifts are expressed in parts per million (δ). Coupling constans in ^1H NMR and ^{19}F NMR are in Hz. The following abbreviations were used to designate multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublet, m = multiplet, brs = broad singlet. High resolution mass spectra (HRMS) were obtained by using Bruker Compact System (ESI) or Jeol JMS-700 high resolution mass spectrometer (EI) from the Korea Basic Science Institute (Daegu).

2) General Procedure for the Preparation of Substrate **1a**–**1p**



Note 1 : **S1** substrates were used commercially avilable reagent or prepared from corresponding cyclic anhydride or maleimide according to the reported prodceudres.[1]

a. DIAD (1.5 equiv) was added dropwise over 5 min to a stirred solution of PPh_3 (1.5 equiv) in THF (0.10 M) at 0 °C. After 20 min, the 2-((trimethylsilyl)methyl)prop-2-en-1-ol[2] (1.0 equiv) was added at 0 ℃. The mixture was stirred for 5 min and added the substrate **S1** (1 equiv) at 0 °C. The resulting mixture was allowed to warm to room temperature and sitrred for overnight. The THF solvent was removed under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to obtain product **S2** in 50–85% yields.

Note 2 : **S2** substrate of **1p** was prepared according to the reported procedure.[3]

b. $NaBH_4$ (1.5 equiv) was portionwisely added to a stirred solution of **S2** (1 equiv) in CH_3OH (0.10 M) at 0 ℃. The reaction mixture was stirred for 1 h at 0 ℃. The CH_3OH solvent was removed under reduced pressure. The reaction mixture was diluted with EtOAc and quenched with water. The aqueous layer was extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was used without further purification.

Note 3 : **S3** substrates of **1h**, **1i**, **1l** and **1p** were prepared according to the reported procedure.[4]

c. Acetic anhydride (1.2 equiv) was added to a solution of **S3** (1 equiv) in CH_2Cl_2 (0.10 M) at room temperature. Then, TEA (1.2 equiv) was added, followed by DMAP (0.1 equiv). The reaction mixture was stirred for 1 h at room temperature. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with CH_2Cl_2. The aqueous layer was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to obtain **1a**–**1p** in 64–96% yields.

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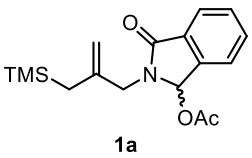
1. (a) G. B. Gill, G. D. James, K. V. Oates and G. Pattenden, *J. Chem. Soc. Perkin Trans 1*, 1993, 2567–2579; (b) K. I. Booker-Milburn, J. R. Baker and I. Bruce, *Org. Lett*., 2004, **6**, 1481–1484.

2. B. M. Trost, D. M. T. Chan and T. N. Nanninga, *Org. Synth.* 1984, **62**, 58.

3. M. A. Walker, *J. Org. Chem.*, 1995, **60**, 5352–5355.

4. N. Mase, T. Nishi, M. Hiyoshi, K. Ichihara, J. Bessho, H. Yoda and K. Takabe, *J. Chem. Soc. Perkin Trans 1*, 2002, 707–709.

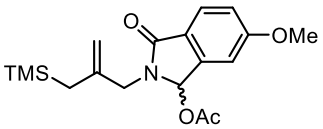
3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1a



1a

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.84 (dt, *J* = 5.9, 1.6 Hz, 1H), 7.58–7.53 (m, 3H), 6.97 (s, 1H), 4.69 (d, *J* = 3.2 Hz, 2H), 4.30 (d, *J* = 15.7 Hz, 1H), 3.75 (d, *J* = 15.7 Hz, 1H), 2.12 (s, 3H), 1.54 (d, *J* = 13.6 Hz, 1H), 1.46 (d, *J* = 13.6 Hz, 1H), 0.08 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.1, 167.9, 142.1, 141.3, 132.6, 132.0, 130.4, 124.2, 123.9, 109.7, 81.3, 46.7, 24.1, 21.1, -1.3; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{17}H_{23}NNaO_3Si$ 340.1339, Found 340.1342.

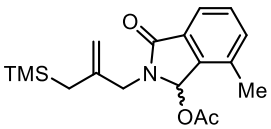
6-methoxy-3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1b



1b

colorless oil; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.2 Hz, 1H), 7.03 (d, *J* = 8.5Hz, 2H), 6.90 (s, 1H), 4.67 (d, *J* = 7.0 Hz, 2H), 4.26 (d, *J* = 15.8 Hz, 1H), 3.86 (s, 3H), 3.71 (d, *J* = 15.8 Hz, 1H), 2.12 (s, 3H), 1.52 (d, *J* = 13.6 Hz, 1H), 1.44 (d, *J* = 13.6 Hz, 1H), 0.06 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.1, 167.8, 163.6, 143.5, 142.2, 125.2, 124.2, 116.6, 109.5, 109.2, 81.1, 55.9, 46.7, 24.0, 21.1, -1.4; **HRMS** (ESI) (m/z): $[M+H]^+$ calcd for $C_{18}H_{26}NO_4Si$ 348.1631, Found 348.1638.

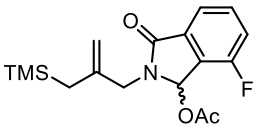
7-methyl-3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1c



1c

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.17 (s, 1H), 4.68 (d, *J* = 7.9, 2H), 4.15 (d, *J* = 15.9 Hz, 1H), 3.84 (d, *J* = 15.9 Hz, 1H), 2.30 (s, 3H), 2.11 (s, 3H), 1.54 (d, *J* = 13.7 Hz, 1H), 1.48 (d, *J* = 13.7 Hz, 1H), 0.08 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.6, 168.3, 142.3, 139.0, 134.0, 132.1, 130.5, 121.5, 109.4, 80.0, 47.2, 24.1, 20.8, 17.5, -1.3; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{25}NO_3Si$ 331.1604, Found 331.1606

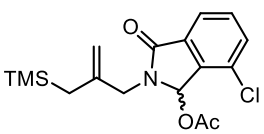
7-fluoro-3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1d



1d

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 7.3 Hz, 1H), 7.57–7.51 (m, 1H), 7.26–7.21 (m, 2H), 4.68 (s, 2H), 4.17 (d, *J* = 15.8 Hz, 1H), 3.82 (d, *J* = 15.9 Hz, 1H), 2.11 (s, 3H), 1.53 (d, *J* = 13.9 Hz, 1H), 1.46 (d, *J* = 13.8 Hz, 1H), 0.07 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.0, 166.8 (d, $J_{C\text{-}F}$ = 2.5 Hz), 157.6 (d, $J_{C\text{-}F}$ = 255.1 Hz), 141.9, 134.8 (d, $J_{C\text{-}F}$ = 3.3 Hz), 132.8 (d, $J_{C\text{-}F}$ = 6.6 Hz), 126.8 (d, $J_{C\text{-}F}$ = 16.3 Hz), 119.9, 119.8 (d, $J_{C\text{-}F}$ = 23.2 Hz), 109.6, 78.0, 47.2, 24.1, 20.8, -1.4; **^{19}F NMR** (375 MHz, $CDCl_3$): δ -119.34 (dd, $J_{F\text{-}H}$ = 8.8, $J_{F\text{-}H}$ = 4.5 Hz); **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{17}H_{22}FNNaO_3Si$ 358.1245, Found 358.1245.

7-chloro-3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1e

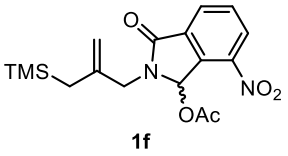


1e

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 6.6, 1.8 Hz, 1H), 7.55–7.49 (m, 2H), 7.21 (s, 1H), 4.71–4.68 (m, 2H), 4.15 (d, *J* = 15.9 Hz, 1H), 3.84 (d, *J* = 15.9 Hz, 1H), 2.13 (s, 3H), 1.53 (d, *J* = 13.6 Hz, 1H), 1.48 (d, *J* = 13.7 Hz, 1H), 0.08 (s, 9H); **^{13}C NMR** (100 MHz,

$CDCl_3$): δ 170.2, 166.9, 141.9, 138.3, 134.3, 133.0, 132.0, 130.0, 122.4, 109.6, 79.1, 47.3, 24.1, 20.7, -1.3; **HRMS** (ESI) (m/z): $[M]^+$ calcd for $C_{17}H_{22}ClNO_3Si$ 351.1057, Found 351.1056.

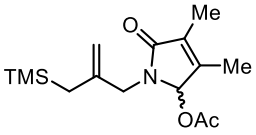
7-nitro-3-oxo-2-(2-((trimethylsilyl)methyl)allyl)isoindolin-1-yl acetate, 1f



1f

yellow solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.40 (dd, *J* = 8.2, 1.0 Hz, 1H), 8.17 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.80 (t, *J* = 7.8 Hz, 1H), 7.61 (s, 1H), 4.70 (dd, *J* = 5.7, 1.2 Hz, 2H), 4.17 (d, *J* = 16.0 Hz, 1H), 3.92 (d, *J* = 16.1 Hz, 1H), 1.52 (s, 2H), 0.09 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.9, 165.4, 143.6, 141.7, 135.9, 135.3, 132.1, 129.7, 127.8, 109.2, 79.8, 47.6, 24.3, 20.5, -1.3; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{17}H_{22}N_2O_5Si$ 362.1298, Found 362.1301.

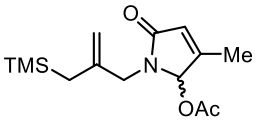
3,4-dimethyl-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1g



1g

coloreless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.40 (s, 1H), 4.61 (dd, *J* = 8.6, 1.4 Hz, 2H), 3.96 (d, *J* = 15.9 Hz, 1H), 3.70 (d, *J* = 15.9 Hz, 1H), 2.10 (s, 3H), 1.87 (s, 3H), 1.84 (s, 3H), 1.49(d, *J* = 13.6 Hz, 1H), 1.42 (d, *J* = 13.6 Hz, 1H), 0.05 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.6, 170.8, 145.4, 142.8, 131.1, 108.9, 83.2, 47.1, 24.0, 21.0, 11.5, 8.7, -1.3; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{15}H_{25}NNaO_3Si$ 318.1496, Found 318.1495.

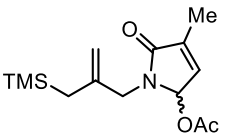
3-methyl-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1h



1h

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.45 (s, 1H), 5.90 (d, *J* = 1.7 Hz, 1H), 4.60 (dd, *J* = 9.4, 1.3 Hz, 2H), 3.94 (d, *J* = 15.9 Hz, 1H), 3.65 (d, *J* = 15.9 Hz, 1H), 2.10 (s, 3H), 1.95 (d, *J* = 1.7 Hz, 3H), 1.47 (d, *J* = 13.7, 1H), 1.40 (d, *J* = 13.7, 1H), 0.03 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.5 (2C), 154.7, 142.6, 124.3, 109.0, 83.7, 46.8, 24.0, 20.9, 13.8, -1.4; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{14}H_{23}NNaO_3Si$ 304.1339, Found 304.1339.

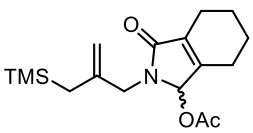
4-methyl-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1i



1i

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.60 (t, *J* = 1.8 Hz, 1H), 6.36 (t, *J* = 1.6 Hz, 1H), 4.62 (dd, *J* = 18.7, 1.4 Hz, 2H), 4.07 (d, *J* = 16.0 Hz, 1H), 3.63 (d, *J* = 16.0 Hz, 1H), 2.08 (s, 3H), 1.94 (t, *J* = 1.5 Hz, 3H), 1.49 (d, *J* = 12.7 Hz, 1H), 1.42 (d, *J* = 12.7 Hz, 1H), 0.06 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.8, 170.6, 142.4, 138.4, 135.0, 109.1, 81.7, 46.8, 24.1, 21.0, 11.1, -1.4; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{14}H_{23}NNaO_3Si$ 304.1339, Found 304.1339.

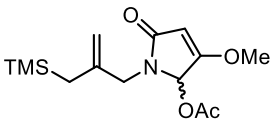
3-oxo-2-(2-((trimethylsilyl)methyl)allyl)-2,3,4,5,6,7-hexahydro-1H-isoindol-1-yl acetate, 1j



1j

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.41 (s, 1H), 4.61 (dd, *J* = 4.8,1.6 Hz, 2H), 3.97 (d, *J* = 16.0 Hz, 1H), 3.67 (d, *J* = 16.0 Hz, 1H), 2.26–2.20 (m, 2H), 2.19–2.14 (m, 2H), 2.09 (s, 3H), 1.77–1.66 (m, 4H), 1.49 (d, *J* = 13.6 Hz, 1H), 1.41 (d, *J* = 13.3 Hz, 1H), 0.04 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.9, 170.7, 149.9, 142.9, 134.5, 108.9, 82.6, 46.8, 24.0, 22.6, 22.0, 21.7, 20.9, 20.2, -1.4; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{17}H_{27}NNaO_3Si$ 344.1652, Found 344.1655.

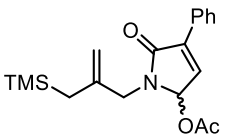
3-methoxy-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1k



1k

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.48 (s, 1H), 5.10 (s, 1H), 4.63 (dt, *J* = 2.8, 1.3 Hz, 2H), 3.95 (d, *J* = 16.0 Hz, 1H), 3.82 (s, 3H), 3.63 (d, *J* = 16.0 Hz, 1H), 1.49 (d, *J* = 13.7 Hz, 1H), 1.42 (d, *J* = 13.7 Hz, 1H), 0.04 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.4, 170.6, 170.3, 142.7, 109.1, 94.6, 79.6, 58.7, 46.4, 24.0, 20.9, -1.3; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{14}H_{23}NNaO_4Si$ 320.1288, Found 320.1289.

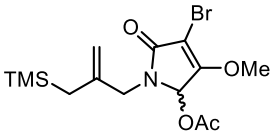
5-oxo-4-phenyl-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1l



1l

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 6.9 Hz, 2H), 7.43 – 7.31 (m, 3H), 7.08 (s, 1H), 6.49 (s, 1H), 4.66 (d, *J* = 3.7 Hz, 2H), 4.17 (d, *J* = 15.8 Hz, 1H), 3.71 (d, *J* = 15.9 Hz, 1H), 2.10 (s, 3H), 1.52 (d, *J* = 13.6 Hz, 1H), 1.45 (d, *J* = 13.6 Hz, 1H), 0.07 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.5, 168.9, 142.1, 138.8, 133.9, 130.4, 129.5, 128.6, 127.5, 109.2, 81.0, 46.7, 24.0, 20.9, -1.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{19}H_{25}NO_3Si$ 343.1604, Found 343.1606.

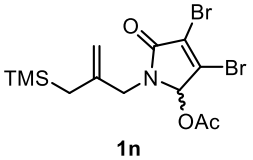
4-bromo-3-methoxy-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1m



1m

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): 6.47 (s, 1H), 4.63 (s, 2H), 4.20 (s, 3H), 3.94 (d, *J* = 15.8 Hz, 1H), 3.69 (d, *J* = 15.8 Hz, 1H), 2.10 (s, 3H), 1.48 (d, *J* = 13.7 Hz, 1H), 1.40 (d, *J* = 13.4 Hz, 1H), 0.04 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.0, 166.7, 163.7, 142.2, 109.7, 87.1, 79.4, 59.5, 47.6, 23.9, 20.8, -1.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{14}H_{22}BrNO_4Si$ 375.0501, Found 375.0502.

3,4-dibromo-5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1n

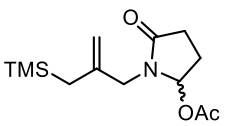


1n

pale yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.60 (s, 1H), 4.66 (d, *J* = 8.4 Hz, 2H), 4.01 (d, *J* = 15.7 Hz, 1H), 3.77 (d, *J* = 15.7 Hz, 1H), 2.14 (s, 3H), 1.48 (d, *J* = 13.6 Hz, 1H), 1.41 (d, *J* = 13.6 Hz, 1H), 0.05 (s, 9H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 169.7, 163.8, 141.7, 135.2, 123.7, 110.2, 82.3, 48.3, 23.9, 20.7, -1.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{13}H_{19}Br_2NO_3Si$ 422.9501, Found 422.9502.

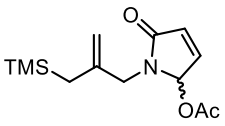
5-oxo-1-(2-((trimethylsilyl)methyl)allyl)pyrrolidin-2-yl acetate, 1o



1o

coloreless oli; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.18 (dd *J* = 6.0 Hz, 1H), 4.66 (dd, *J* = 11.1, 1.2 Hz, 2H), 4.10 (d, *J* = 15.6 Hz, 1H), 3.42 (d, *J* = 15.6 Hz, 1H), 2.66–2.55 (m, 1H), 2.46–2.30 (m, 2H), 2.09–2.04 (s, 4H), 1.49 (d, *J* = 13.6 Hz, 1H), 1.40 (d, *J* = 13.6 Hz, 1H), 0.05 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 175.7, 170.6, 141.4, 109.4, 84.6, 46.9, 28.4, 26.2, 24.1, 21.1, -1.4; **HRMS** (ESI) (m/z): $[M+Na–OAc]^+$ calcd for $C_{11}H_{21}NNaO_2Si$ 250.1234, Found 250.1226.

5-oxo-1-(2-((trimethylsilyl)methyl)allyl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 1p

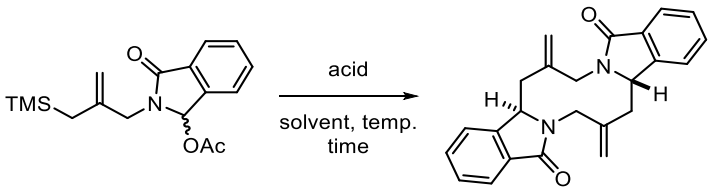


1p

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.98 (d, *J* = 6.0 Hz, 1H), 6.48 (s, 1H), 6.27 (d, *J* = 5.9 Hz, 1H), 4.65 (s, 1H), 4.60 (s, 1H), 4.08 (d, *J* = 16.0 Hz, 1H), 3.62 (d, *J* = 15.9 Hz, 1H), 2.09 (s, 3H), 1.49 (d, *J* = 13.6 Hz, 1H), 1.41 (d, *J* = 13.7 Hz, 1H), 0.05 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 170.4, 169.9, 142.4, 142.2, 129.9, 109.2, 82.9, 46.4, 24.0, 20.9, -1.4; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{13}H_{21}NNaO_3Si$ 290.1183, Found 290.1185.

3) Experiment Procedure for the Optimization Study

Substrate **1a** (1 equiv, 0.60 mmol) was dissolved in indicated solvent (0.10 M, 6.0 mL) under argon atmosphere. The resulting mixture was cooled to indicated temperature. After 10 min, acid was added dropwise to the reaction mixture. The resulting mixture was stirred and allowed to warm to indicated temperature slowly over time. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with $CHCl_3$. The aqueous layer was extracted with $CHCl_3$. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to give product.

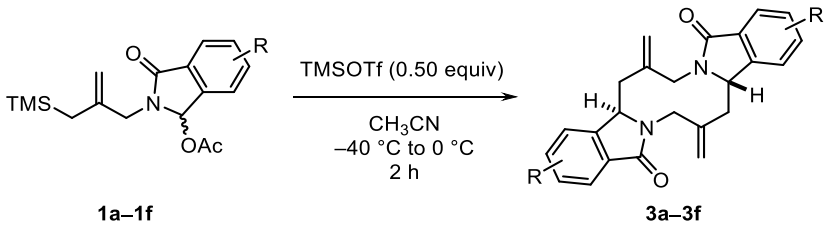


1a **3a**

entry	solvent	acid (equiv)	temp. (°C)	time (h)	yield (%)
1	CH_2Cl_2	TfOH (1.1)	–78 to rt	6	ND
2	CH_2Cl	TfOH (0.50)	–78 to rt	6	ND
3	CH_2Cl_2	$Bi(OTf)_3$ (1.1)	–78 to rt	6	ND
4	CH_2Cl_2	$TiCl_4$ (1.1)	–78 to rt	6	ND
5	CH_2Cl_2	TMSOTf (1.1)	–78 to rt	18	42
6	CH_2Cl_2	TMSOTf (1.1)	–78 to rt	6	67
7	CH_2Cl_2	TMSOTf (2.0)	–78 to rt	18	71
8	CH_2Cl_2	TMSOTf (2.0)	–40 to rt	18	70
9	Et_2O	TMSOTf (1.1)	–78 to rt	6	47
10	Et_2O	TMSOTf (2.0)	–78 to rt	6	54
11	THF	TMSOTf (1.1)	–78 to rt	6	-[a]
12	CH_3CN	TMSOTf (1.1)	–40 to rt	6	78
13	CH_3CN	TMSOTf (1.1)	–40 to rt	18	72
14	CH_3CN (0.5M)	TMSOTf (1.1)	–40 to rt	6	64
15	CH_3CN	TMSOTf (2.0)	–40 to rt	6	70
16	CH_3CN	TMSOTf (1.1)	–40 to 0	2	81
17	CH_3CN	TMSOTf (1.1)	0	2	71
18	CH_3CN	TMSOTf (0.50)	–40 to 0	2	92
19	CH_3CN	TMSOTf (0.20)	–40 to 0	2	83
20	CH_3CN	TfOH (0.50)	–40 to 0	2	81
21	CH_3CN	$Bi(OTf)_3$ (0.50)	–40 to 0	2	42
22	CH_3CN	$TiCl_4$ (0.50)	–40 to 0	2	ND

[a] The reaction mixture bacame viscous; ND = Not Detected.

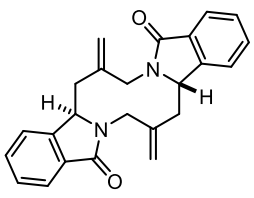
4) General Procedure for the Synthesis of 1,6-Diazecanes **3a**–**3f**



1a–1f **3a–3f**

Substrate **1a**–**1f** (1 equiv) was dissolved in CH_3CN (0.10 M) under argon atmosphere. The resulting mixture was cooled to –40 °C. After 10 min, TMSOTf (0.50 equiv) was added dropwise to the reaction mixture. The resulting mixture was stirred and allowed to warm to 0 °C slowly over 2 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with $CHCl_3$. The aqueous layer was extracted with $CHCl_3$. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to give product **3a**–**3f**.

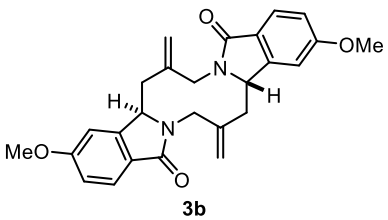
(9aR,18aS)-8,17-dimethylene-7,8,9,9a,16,17,18,18a-octahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3a



3a

Yield: 92%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.89–7.82 (m, 2H), 7.62–7.55 (m, 2H), 7.49 (t, *J* = 7.4, 7.3 Hz, 4H), 4.92 (s, 2H), 4.86 (dd, *J* = 7.5, 4.0 Hz, 2H), 4.68 (s, 2H), 4.47 (d, *J* = 15.5 Hz, 2H), 4.15 (d, *J* = 15.4 Hz, 2H), 3.13 (dd, *J* = 15.2, 5.2 Hz, 2H), 2.63 (dd, *J* = 15.2, 7.5 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.7, 145.6, 140.4, 131.9 (131.94, 131.93, 2C), 128.5, 124.0, 122.4, 116.6, 59.2, 47.4, 37.3; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{24}H_{22}N_2NaO_2$ 393.1574, Found 393.1577.

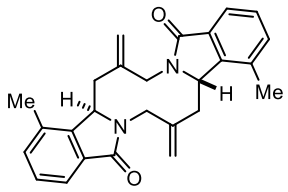
(9aR,18aS)-2,11-dimethoxy-8,17-dimethylene-7,8,9,9a,16,17,18,18a-octahydro-5H,1 4H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3b



3b

Yield: 84%; white solid; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.4 Hz, 2H), 6.99 (dd, *J* = 8.4, 2.2 Hz, 2H), 6.94 (d, *J* = 2.2 Hz, 2H), 4.92 (s, 2H), 4.78 (dd, *J* = 7.3, 4.1 Hz, 2H), 4.67 (s, 2H), 4.44 (d, *J* = 15.6 Hz, 2H), 4.10 (d, *J* = 15.7 Hz, 2H), 3.89 (s, 6H), 3.09 (dd, *J* = 15.3, 4.0 Hz, 2H), 2.61 (dd, *J* = 15.3, 7.3 Hz, 2H) **^{13}C NMR** (150 MHz, $CDCl_3$): δ 169.7, 163.2, 148.0, 140.6, 125.3, 124.6, 116.4, 115.1, 107.2, 59.1, 55.9, 47.6, 37.2; **HRMS** (ESI) (m/z): $[M+H]^+$ calcd for $C_{26}H_{27}N_2O_4$ 431.1971, Found 431.1979.

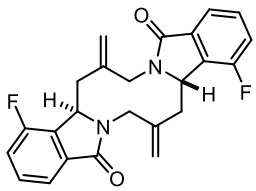
(9aR,18aS)-1,10-dimethyl-8,17-dimethylene-7,8,9,9a,16,17,18,18a-octahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3c



3c

Yield: 79%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.67 (d, *J* = 7.0 Hz, 2H), 7.40–7.31 (m, 4H), 4.92 (s, 2H), 4.82 (dd, *J* = 6.1, 4.1 Hz, 2H), 4.51 (d, *J* = 15.2 Hz, 2H), 4.43 (s, 2H), 4.05 (d, *J* = 15.3 Hz, 2H), 3.16 (dd, *J* = 15.3, 4.0 Hz, 2H), 2.77 (dd, *J* = 15.4, 6.0 Hz, 2H), 2.50 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.9, 143.9, 140.5, 133.7, 132.6, 132.3, 128.6, 121.5, 116.9, 60.2, 48.9, 34.5, 18.6; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{26}H_{26}N_2O_2$ 398.1994, Found 398.1998.

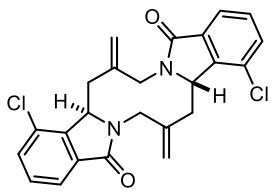
(9aR,18aS)-1,10-difluoro-8,17-dimethylene-7,8,9,9a,16,17,18,18a-octahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3d



3d

Yield: 77%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 7.5 Hz, 2H), 7.51–7.44 (m, 2H), 7.23 (t, *J* = 8.3 Hz, 2H), 4.96 (s, 2H), 4.91 (dd, *J* = 6.0, 4.0 Hz, 2H), 4.65 (s, 2H), 4.40 (d, *J* = 15.5 Hz, 2H), 3.92 (d, *J* = 15.4 Hz, 2H), 3.18 (dd, *J* = 15.3, 4.1 Hz, 2H), 2.85 (dd, *J* = 15.5, 6.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.9 (d, $J_{C\text{-}F}$ = 2.2 Hz), 157.7 (d, $J_{C\text{-}F}$ = 250.8 Hz), 140.0, 135.1 (d, $J_{C\text{-}F}$ = 4.3 Hz), 131.2 (d, $J_{C\text{-}F}$ = 16.2 Hz), 130.8 (d, $J_{C\text{-}F}$ = 6.7 Hz), 119.9 (d, $J_{C\text{-}F}$ = 3.7 Hz), 118.8 (d, $J_{C\text{-}F}$ = 20.1 Hz), 116.5, 58.6, 48.6, 33.7; **^{19}F NMR** (375 MHz, $CDCl_3$): δ -119.40 (dd, $J_{F\text{-}H}$ = 9.2, $J_{F\text{-}H}$ = 4.5 Hz); **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{24}H_{20}F_2N_2NaO_2$ 429. 1385, Found 429.1385.

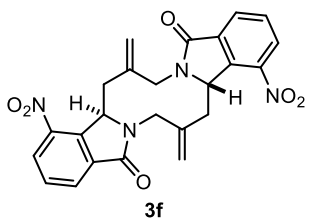
(9aR,18aS)-1,10-dichloro-8,17-dimethylene-7,8,9,9a,16,17,18,18a-octahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3e



3e

Yield: 79%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.5 Hz, 2H), 7.53 (d, *J* = 7.9 Hz, 2H), 7.44 (t, *J* = 7.7 Hz, 2H), 4.95 (s, 2H), 4.83 (t, *J* = 4.8 Hz, 2H), 4.53 (s, 2H), 4.44 (d, *J* = 15.2 Hz, 2H), 3.87 (d, *J* = 15.4 Hz, 2H), 3.22 (dd, *J* = 15.5, 4.2 Hz, 2H), 3.11 (dd, *J* = 15.6, 5.3 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 168.6, 142.4, 139.9, 132.6, 130.2, 129.3, 122.5, 116.5, 60.7, 49.3, 31.8; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{24}H_{20}Cl_2N_2O_2$ 438.0902, Found 438.0905.

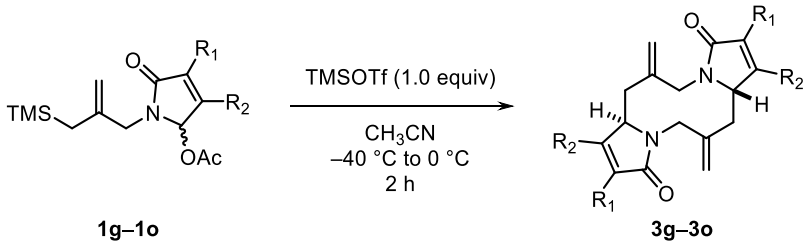
(9aR,18aS)-8,17-dimethylene-1,10-dinitro-7,8,9,9a,16,17,18,18a-octahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3f



3f

Yield: 38%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.39 (dd, *J* = 8.2, 1.0 Hz, 2H), 8.16 (dd, *J* = 7.6, 1.1 Hz, 2H), 7.71 (t, *J* = 7.8 Hz, 2H), 5.58 (dd, *J* = 9.8, 2.0 Hz, 2H), 5.47 (s, 2H), 5.31 (s, 2H), 4.66 (d, *J* = 16.2 Hz, 2H), 4.21 (d, *J* = 16.3 Hz, 2H), 3.07 (d, *J* = 15.3 Hz, 2H), 2.06 (dd, *J* = 15.2, 9.8 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.6, 143.8, 141.9, 140.7, 135.2, 130.3, 130.1, 127.7, 117.0, 62.6, 47.9, 37.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{24}H_{20}N_4O_6$ 460.1383, Found 460.1380.

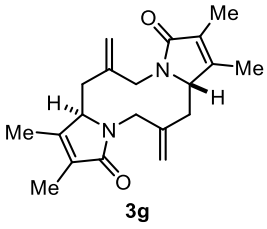
5) General Procedure for the Synthesis of 1,6-Diazecanes **3g**–**3o**



1g–1o **3g–3o**

Substrate **1g**–**1o** (1 equiv) was dissolved in CH_3CN (0.10 M) under argon atmosphere. The resulting mixture was cooled to –40 °C. After 10 min, TMSOTf (1.0 equiv) was added dropwise to the reaction mixture. The resulting mixture was stirred and allowed to warm to 0 °C slowly over 2 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with $CHCl_3$. The aqueous layer was extracted with $CHCl_3$. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to give product **3g**–**3o**.

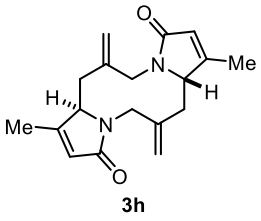
(7aR,14aS)-1,2,8,9-tetramethyl-6,13-dimethylene-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3g



3g

Yield: 73%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.95 (s, 2H), 4.83 (s, 2H), 4.22 (d, *J* = 15.2 Hz, 2H), 3.98 (s, 2H), 3.79 (d, *J* = 15.3 Hz, 2H), 2.81 (dd, *J* = 15.4, 4.5 Hz, 2H), 2.35 (dd, *J* = 15.3, 6.0 Hz, 2H), 1.94 (s, 6H), 1.79 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.0, 150.4, 141.2, 129.1, 115.4, 63.3, 48.3, 33.2, 12.4, 8.8; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{20}H_{26}N_2NaO_2$ 349.1887, Found 349.1888.

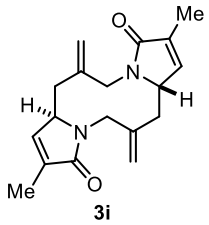
(7aR,14aS)-1,8-dimethyl-6,13-dimethylene-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3h



3h

Yield: 75%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.88 (s, 2H), 5.01 (s, 2H), 4.93 (s, 2H), 4.22 (d, *J* = 15.4 Hz, 2H), 4.12 (t, *J* = 5.1 Hz, 2H), 3.84 (d, *J* = 15.4 Hz, 2H), 2.83 (dd, *J* = 15.3, 4.2 Hz, 2H), 2.39 (dd, *J* = 15.2, 6.2 Hz, 2H), 2.05 (d, *J* = 1.5 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.1, 159.7, 140.8, 123.3, 116.0, 64.6, 48.0, 33.6, 14.8; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{18}H_{22}N_2NaO_2$ 321.1574, Found 321.1574.

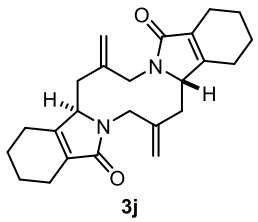
(7aR,14aS)-2,9-dimethyl-6,13-dimethylene-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3i



3i

Yield: 76%; white solid; **^{1}H NMR** (400 MHz, CD_3OD): δ 5.78 (s, 2H), 4.93 (d, *J* = 6.5 Hz, 4H), 4.27 (t, *J* = 4.8 Hz, 2H), 4.12 (d, *J* = 15.7 Hz, 2H), 3.86 (d, *J* = 15.5 Hz, 2H), 2.79 (dd, *J* = 15.4, 4.2 Hz, 2H), 2.36 (dd, *J* = 15.2, 6.6 Hz, 2H), 2.02 (d, *J* = 1.6 Hz, 6H); **^{13}C NMR** (100 MHz, CD_3OD): δ 175.3, 163.6, 142.2, 122.9, 116.6, 66.1, 48.6, 34.6, 14.6; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{18}H_{22}N_2NaO_2$ 321.1574, Found 321.1574.

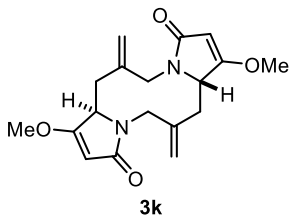
(9aR,18aS)-8,17-dimethylene-1,2,3,4,7,8,9,9a,10,11,12,13,16,17,18,18a-hexadecahydro-5H,14H-[1,6]diazecino[2,1-a:7,6-a']diisoindole-5,14-dione, 3j



3j

Yield: 72%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.96 (s, 2H), 4.86 (s, 2H), 4.21 (d, *J* = 15.3 Hz, 2H), 4.05 (s, 2H), 3.84 (d, *J* = 15.4 Hz, 2H), 2.79 (dd, *J* = 15.2, 4.5 Hz, 2H), 2.38–2.15 (m, 10H), 1.79–1.59 (m, 8H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.3, 154.5, 141.3, 131.9, 115.2, 62.3, 47.8, 33.7, 23.5, 22.3, 22.0, 20.4; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{24}H_{30}N_2NaO_2$ 401.2205, Found 401.2202.

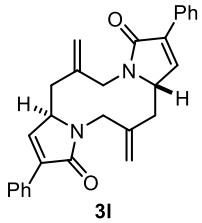
(7aR,14aS)-1,8-dimethoxy-6,13-dimethylene-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3k



3k

Yield: 85%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.05 (s, 4H), 4.82 (s, 2H), 4.20 (d, *J* = 15.2 Hz, 2H), 4.06 (t, *J* = 4.6 Hz, 2H), 3.78 (s, 6H), 3.61 (d, *J* = 15.2 Hz, 2H), 2.77 (dd, *J* = 14.9, 4.6 Hz, 2H), 2.54 (dd, *J* = 14.9, 4.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 176.0, 173.8, 140.9, 115.5, 94.7, 61.7, 58.1, 49.0, 31.8; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{22}N_2O_4$ 330.1580, Found 330.1583.

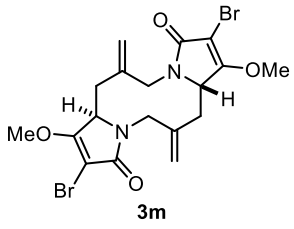
(7aR,14aS)-6,13-dimethylene-2,9-diphenyl-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3l



3l

Yield: 59%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 6.8 Hz, 4H), 7.42–7.34 (m, 6H), 7.19 (d, *J* = 1.9 Hz, 2H), 5.07 (d, *J* = 23.4 Hz, 4H), 4.41–4.37 (m, 2H), 4.34 (d, *J* = 15.2 Hz, 2H), 3.89 (d, *J* = 15.5 Hz, 2H), 2.94 (dd, *J* = 15.0, 4.3 Hz, 2H), 2.55 (dd, *J* = 15.0, 6.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.9, 141.9, 140.8, 136.1, 131.6, 128.8, 128.6, 127.2, 116.3, 60.2, 48.5, 34.5; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{28}H_{26}N_2O_2$ 422.1994, Found 422.1993.

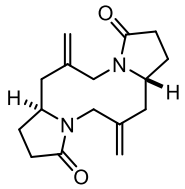
(7aR,14aS)-2,9-dibromo-1,8-dimethoxy-6,13-dimethylene-5,6,7,7a,12,13,14,14a-octahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diazecine-3,10-dione, 3m



3m

Yield: 55%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 5.13 (s, 2H), 4.88 (s, 2H), 4.28–4.23 (m, 8H), 4.00 (t, *J* = 4.3 Hz, 2H), 3.57 (d, *J* = 15.0 Hz, 2H), 2.74 (dd, *J* = 15.1, 4.6 Hz, 2H), 2.58 (dd, *J* = 15.0, 4.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 169.7, 168.1, 140.0, 116.4, 85.5, 62.8, 59.4, 50.5, 31.2; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{20}Br_2N_2O_4$ 485.9790, Found 485.9793.

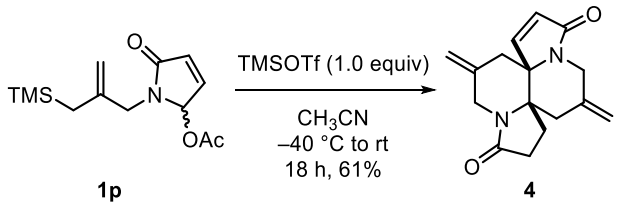
(7aR,14aS)-6,13-dimethylenedodecahydro-3H,10H-dipyrrolo[1,2-a:1',2'-f][1,6]diaze cine-3,10-dione, 3o



3o

Yield: 22%; white solid; **^{1}H NMR** (400 MHz, CD_3OD): δ 5.27 (s, 2H), 4.97 (s, 2H), 4.19–4.11 (m, 2H), 4.10–3.98 (m, 4H), 2.54 (d, *J* = 15.2 Hz, 2H), 2.49–2.42 (m, 4H), 2.35–2.20 (m, 4H), 1.72–1.65 (m, 2H); **^{13}C NMR** (100 MHz, CD_3OD): δ 178.7, 143.3, 115.2, 59.3, 46.8, 42.7, 31.2, 26.9; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{16}H_{22}N_2NaO_2$ 297.1574, Found 297.1574.

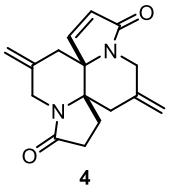
6) Procedure for the Synthesis of **4**



1p **4**

Substrate **1p** (56.4 mg, 0.211 mmol, 1 equiv) was dissolved in CH_3CN (2.10 mL) under argon atmosphere at room temperature. The resulting mixture was cooled to –40 °C. After 10 min, TMSOTf (38.0 μL, 0.211 mmol, 1.00 equiv) was added dropwise to the reaction mixture. The resulting mixture was allowed to warm to room temperature slowly over 5 h and additionally stirred for 12 h at room temperature. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution (3 mL) and diluted with CH_2Cl_2 (3 mL). The aqueous layer was extracted with CH_2Cl_2 (3 × 3 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by column chromatography on silica gel (CH_3OH : EtOAc = 1 : 5) to obtain product **4** (17.3 mg, 60.7%).

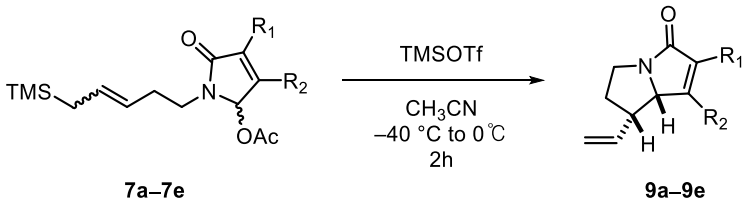
(3aS,10aR)-5,12-dimethylene-2,3,5,6,12,13-hexahydro-1H,4H,8H,11H-dipyrrolo[2,1-e:2',1'-j][1,5]naphthyridine-1,8-dione, 4



4

Yield: 61%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.02 (d, *J* = 6.0 Hz, 1H), 6.27 (d, *J* = 6.0 Hz, 1H), 5.11 (d, *J* = 10.8 Hz, 2H), 4.92 (d, *J* = 3.4 Hz, 2H), 4.70–4.61 (m, 2H), 3.49 (dd, *J* = 14.7, 6.5 Hz, 2H), 3.24–3.16 (m, 2H), 2.44–2.33 (m, 1H), 2.22–2.13 (m, 1H), 2.07 (dd, *J* = 13.7, 1.8 Hz, 2H), 1.61–1.54 (m, 1H), 1.47–1.39 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.2, 168.1, 148.2, 137.2, 137.1, 129.0, 114.7 (114.73, 114.71, 2C), 68.0, 63.5, 42.6, 41.8, 39.6, 36.9, 29.5, 24.6; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{16}H_{18}N_2NaO_2$ 293.1261, Found 293.1263.

7) General Procedure for the Synthesis of **9a**–**9e**



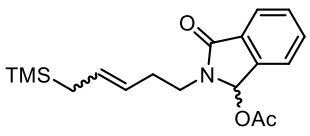
7a–7e **9a–9e**

Note 1 : Substrates **7a**–**7e** were synthesized from 5-(trimethylsilyl)pent-3-en-1-ol[5] according to the general procedure for the preparation of substrate **1a**–**1p**. ^{1}H and ^{13}C NMR spectral data of substrates **7a**–**7e** for the major isomer were reported.

Substrate **7a**–**7e** (1 equiv) was dissolved in CH_3CN (0.10 M) under argon atmosphere. The resulting mixture was cooled to –40 ℃. After 10 min, TMSOTf (0.50 or 1.0 equiv, **Note 2**) was added dropwise to the reaction mixture. The resulting mixture was stirred and allowed to warm to 0 ℃ slowly over 2 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with CH_2Cl_2. The aqueous layer was extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel to give product **9a**–**9e**.

Note 2 : 0.50 equiv of TMSOTf was used for **7a**–**7c** and 1.0 equiv of TMSOTF was used for **7d** and **7e**.

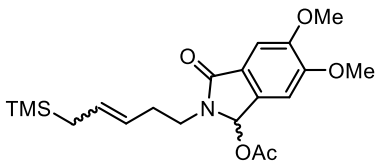
3-oxo-2-(5-(trimethylsilyl)pent-3-en-1-yl)isoindolin-1-yl acetate, 7a



7a

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.84–7.76 (m, 1H), 7.53 (d, *J* = 1.8 Hz, 3H), 7.02 (d, *J* = 7.9 Hz, 1H), 5.56–5.41 (m, 1H), 5.31–5.16 (m, 1H), 3.96–3.69 (m, 1H), 3.34–3.15 (m, 1H), 2.60–2.24 (m, 2H), 2.16 (s, 3H), 1.38 (d, *J* = 7.9 Hz, 2H), -0.11 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.3, 167.9, 142.7, 141.1, 132.3, 130.3, 129.7, 124.5, 123.9, 123.7, 81.3, 40.3, 31.8, 22.9, 21.2, -2.0; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{25}NO_3Si$ 331.1604, Found 331.1607.

5,6-dimethoxy-3-oxo-2-(5-(trimethylsilyl)pent-3-en-1-yl)isoindolin-1-yl acetate, 7b



7b

white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 (s, 1H), 7.00 (s, 1H), 6.89 (s, 1H), 5.54–5.42 (m, 1H), 5.29–5.16 (m, 1H), 3.93 (s, 6H), 3.84–3.72 (m, 1H), 3.26–3.14 (m, 1H), 2.47–2.22 (m, 2H), 2.17 (s, 3H), 1.38 (d, *J* = 8.4 Hz, 2H), -0.10 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.5, 168.1, 152.9, 151.2, 134.5, 129.5, 124.8, 124.5, 106.3, 105.4, 81.4, 56.5, 56.4, 40.4, 31.9, 22.9, 21.3, -2.0; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{20}H_{29}NO_5Si$ 391.1815, Found 391.1813.

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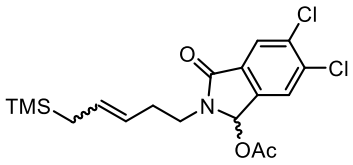
5. M. Tredwell, J. A. R. Luft, M. Schuler, K. Tenza, K. N. Houk and V. Gouverneur, *Angew. Chem., Int. Ed.*, 2008, **47**, 357–360

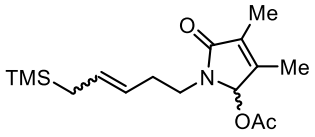
5,6-dichloro-3-oxo-2-(5-(trimethylsilyl)pent-3-en-1-yl)isoindolin-1-yl acetate, 7c



7c

white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.65 (s, 1H), 6.92 (s, 1H), 5.55–5.41 (m, 1H), 5.27–5.13 (m, 1H), 3.79 (dq, *J* = 14.2, 7.0 Hz, 1H), 3.28–3.15 (m, 1H), 2.41–2.24 (m, 2H), 2.17 (s, 3H), 1.37 (d, *J* = 7.9 Hz, 2H), -0.05 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.0, 165.7, 140.2, 136.8, 135.3, 132.1, 130.0, 126.4, 125.5, 124.1, 80.5, 40.6, 31.7, 22.9, 21.1, -2.0; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{23}Cl_2NO_3Si$ 399.0824, Found 399.0827.

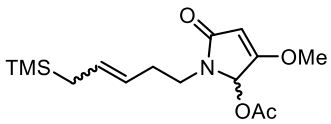
3,4-dimethyl-5-oxo-1-(5-(trimethylsilyl)pent-3-en-1-yl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 7d



7d

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.40 (s, 1H), 5.51–5.38 (m, 1H), 5.24–5.11 (m, 1H), 3.64–3.51 (m, 1H), 3.11–2.99 (m, 1H), 2.34–2.16 (m, 2H), 2.15 (s, 3H), 1.83 (s, 3H), 1.79 (s, 3H), 1.38 (d, *J* = 8.0 Hz, 2H), -0.06 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.5, 171.1, 144.8, 131.4, 129.3, 124.8, 82.8, 40.3, 32.0, 22.9, 21.1, -11.4, -8.6, -1.9; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{16}H_{27}NO_3Si$ 309.1760, Found 309.1758.

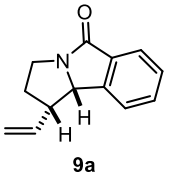
3-methoxy-5-oxo-1-(5-(trimethylsilyl)pent-3-en-1-yl)-2,5-dihydro-1H-pyrrol-2-yl acetate, 7e



7e

colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.46 (s, 1H), 5.50–5.38 (m, 1H), 5.21–5.09 (m, 1H), 5.03 (s, 1H), 3.77 (s, 3H), 3.60–3.48 (m, 1H), 3.06–2.92 (m, 1H), 2.27–2.14 (m, 2H), 2.13 (s, 3H), 1.37 (d, *J* = 8.2 Hz, 2H), -0.07 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.0, 170.6, 170.5, 129.3, 124.6, 94.8, 79.4, 58.6, 39.7, 31.9, 22.9, 21.0, -1.9; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{15}H_{25}NO_4Si$ 311.1553, Found 311.1554.

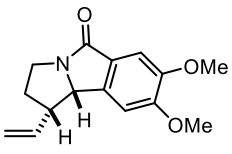
(1S,9bS)-1-vinyl-1,2,3,9b-tetrahydro-5H-pyrrolo[2,1-a]isoindol-5-one, 9a



9a

Yield: 77%; colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.5 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.36 (d, *J* = 7.5 Hz, 1H), 5.11–4.95 (m, 2H), 4.88 (d, *J* = 6.1 Hz, 1H), 4.81 (d, *J* = 12.0 Hz, 1H), 3.84–3.73 (m, 1H), 3.45 (t, *J* = 10.5 Hz, 1H), 3.12 (q, *J* = 6.4 Hz, 1H), 2.62–2.50 (m, 1H), 2.36–2.26 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.4, 143.9, 135.0, 134.8, 131.6, 128.4, 123.9, 123.5, 116.7, 67.7, 42.9, 40.8, 34.7; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{13}H_{13}NO$ 199.0997, Found 199.0994.

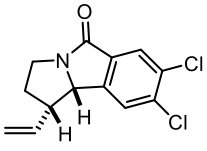
(1S,9bS)-7,8-dimethoxy-1-vinyl-1,2,3,9b-tetrahydro-5H-pyrrolo[2,1-a]isoindol-5-one, 9b



9b

Yield: 84%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (s, 1H), 6.80 (s, 1H), 5.09–4.94 (m, 2H), 4.85–4.76 (m, 2H), 3.91 (d, *J* = 5.0 Hz, 6H), 3.77–3.69 (m, 1H), 3.43–3.35 (m, 1H), 3.09–3.02 (m, 1H), 2.57–2.46 (m, 1H), 2.30–2.23 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.2, 152.8, 149.9, 137.7, 135.0, 127.1, 116.5, 105.6, 105.5, 67.5, 56.3(56.34, 56.25, 2C) 42.9, 40.1, 34.9; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{15}H_{17}NO_3$ 259.1208, Found 259.1211.

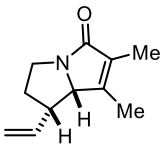
(1S,9bS)-7,8-dichloro-1-vinyl-1,2,3,9b-tetrahydro-5H-pyrrolo[2,1-a]isoindol-5-one, 9c



9c

Yield: 83%; white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.46 (s, 1H), 5.05–5.01 (m, 2H), 4.90–4.86 (m, 1H), 4.83 (d, *J* = 6.0 Hz, 1H), 3.82–3.73 (m, 1H), 3.47–3.40 (m, 1H), 3.14–3.08 (m, 1H), 2.60–2.50 (m, 1H), 2.34–2.27 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.0, 143.0, 136.2, 134.9, 134.1, 133.3, 125.8, 125.7, 117.6, 67.1, 42.9, 41.0, 34.6; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{13}H_{11}Cl_2NO$ 267.0218, Found 267.0218.

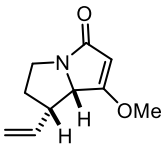
(7S,7aS)-1,2-dimethyl-7-vinyl-5,6,7,7a-tetrahydro-3H-pyrrolizin-3-one, 9d



9d

Yield: 77%; colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.30–5.17 (m, 1H), 5.04 (dd, *J* = 17.1, 1.8 Hz, 1H), 4.93 (dd, *J* = 10.1, 1.7 Hz, 1H), 4.14 (d, *J* = 5.9 Hz, 1H), 3.56–3.47 (m, 1H), 3.27 (ddd, *J* = 11.1, 9.1, 2.1 Hz, 1H), 2.88–2.80 (m, 1H), 2.49–2.37 (m, 1H), 2.18–2.10 (m, 1H), 1.84 (s, 3H), 1.73 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 176.8, 150.0, 134.6, 131.1, 116.1, 71.2, 42.5, 40.8, 35.3, 12.9, 8.7; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{11}H_{15}NO$ 177.1154, Found 177.1152.

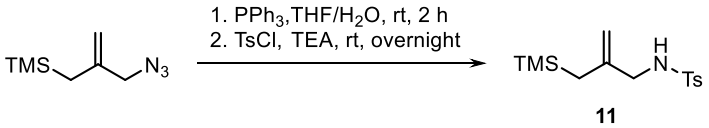
(7S,7aS)-1-methoxy-7-vinyl-5,6,7,7a-tetrahydro-3H-pyrrolizin-3-one, 9



9e

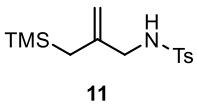
Yield: 86%; colorless oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.49–5.37 (m, 1H), 5.08 (d, *J* = 17.1 Hz, 1H), 5.00 (d, *J* = 10.3 Hz, 1H), 4.95 (s, 1H), 4.27 (d, *J* = 6.4 Hz, 1H), 3.73 (s, 3H), 3.62–3.53 (m, 1H), 3.20–3.14 (m, 1H), 2.90 (q, *J* = 7.4 Hz, 1H), 2.38–2.27 (m, 1H), 2.17–2.09 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 177.1, 176.1, 134.2, 116.7, 96.3, 68.1, 58.4, 42.2, 41.4, 34.3; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{10}H_{13}NO_2$ 179.0946, Found 179.0948.

8) Procedure for the Reaction of **11**

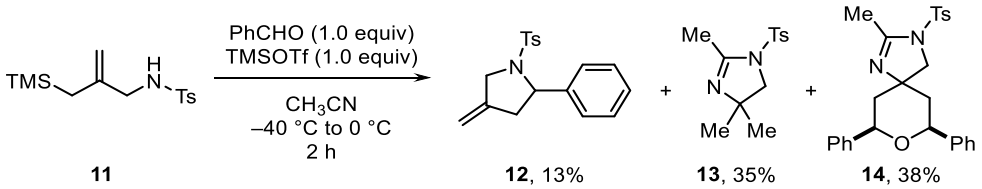


PPh_3 (600 mg, 2.29 mmol, 1.10 equiv) was added to a stirred solution of (2-(azidomethyl)allyl) trimethylsilane[6] (352 mg, 2.08 mmol, 1 equiv) in THF/H_2O (6 mL/2.4 mL) at room temperature. The reaction mixture was sitrred for 20 h at room temperature. After 20 h, TsCl (476 mg, 2.50 mmol, 1.20 equiv) was added, followed by TEA (0.348 mL, 2.50 mmol, 1.20 equiv). The resulting mixture was stirred for overnight at room temperature. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution (8 mL) and diluted with EtOAc (8 mL). The aqueous layer was extracted with EtOAc (3 × 8 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel (EtOAc : Hexane = 1 : 6) to obtain product **11** (467 mg, 76.5%).

4-methyl-N-(2-((trimethylsilyl)methyl)allyl)benzenesulfonamide, 11



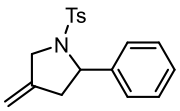
white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 4.76 (s, 1H), 4.61 (s, 1H), 4.49 (t, *J* = 6.3 Hz, 1H), 3.41 (d, *J* = 6.4 Hz, 2H), 2.42 (s, 3H), 1.46 (s, 2H), -0.04 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.6, 142.5, 137.1, 129.8, 127.3, 109.8, 49.5, 24.2, 21.7, -1.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{14}H_{23}NO_2SSi$ 297.1219, Found 297.1217.



Substrate **11** (160 mg, 0.538 mmol, 1 equiv) was dissolved in CH_3CN (5.40 mL) under argon atmosphere. The resulting mixture was cooled to –40 °C. After 10 min, PhCHO (55.0 µL, 0.538 mmol, 1.00 equiv) was added the reaction mixture, followed by TMSOTf (97.0 µL**,** 0.538 mmol**,** 1.00 equiv) dropwisely. The resulting mixture was stirred and allowed to warm to 0 °C slowly over 2 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and diluted with CH_2Cl_2. The aqueous layer was extracted with CH_2Cl_2. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel (EtOAc : Hexane = 5 : 5) to obtain the products **12** (21.4 mg, 12.7%), **13** (50.7 mg, 35.4%) and **14** (93.7 mg, 37.8%).

6. M. Taddei, S. Ferrini and E. Cini, *Synlett*, 2013, **24**, 491–495.

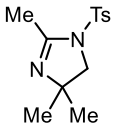
4-methylene-2-phenyl-1-tosylpyrrolidine, 12



12

white solid; **^{1}H NMR** (400 MHz, $(CD_3)_2CO$) δ 7.71 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.35–7.20 (m, 5H), 5.05–4.96 (m, 2H), 4.90 (s, 1H), 4.10 (s, 2H), 2.72–2.63 (m, 1H), 2.48–2.41 (m, 4H); **^{13}C NMR** (100 MHz, $(CD_3)_2CO$) δ 144.9, 144.4, 143.8, 136.3, 130.6, 129.1, 128.4, 127.9, 127.2, 108.3, 63.9, 53.3, 41.9, 21.4; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{18}H_{19}NO_2S$ 313.1136, Found 313.1138.

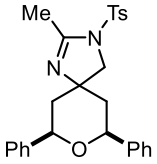
2,4,4-trimethyl-1-tosyl-4,5-dihydro-1H-imidazole, 13



13

white solid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 3.46 (s, 2H), 2.44 (s, 3H), 2.25 (s, 3H), 1.13 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 153.4, 144.7, 135.6, 130.2, 127.3, 64.5, 60.1, 28.6, 21.7, 17.0; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{13}H_{18}N_2O_2S$ 266.1089, Found 266.1091.

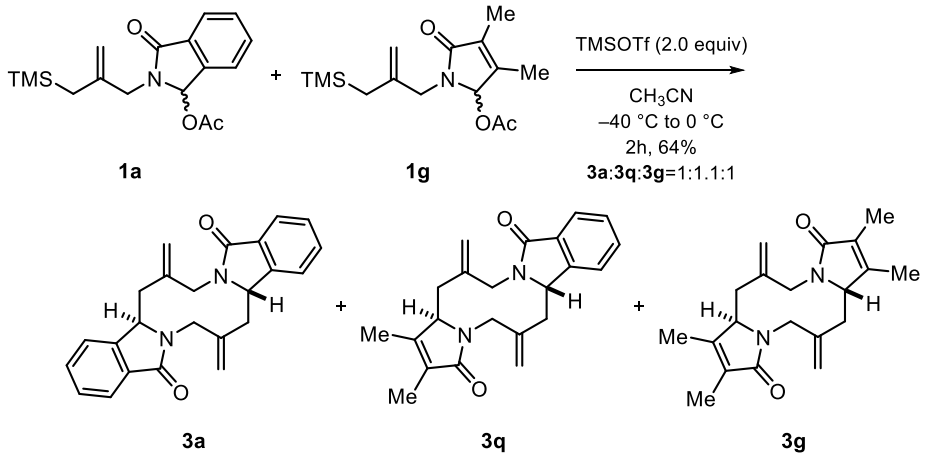
(7R,9S)-2-methyl-7,9-diphenyl-3-tosyl-8-oxa-1,3-diazaspiro[4.5]dec-1-ene, 14



14

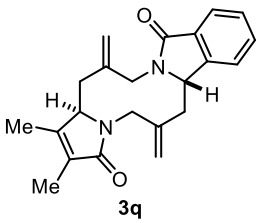
white solid; **^{1}H NMR** (400 MHz, $(CD_3)_2CO$) δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 6.8 Hz, 4H), 7.34 (t, *J* = 7.5 Hz, 4H), 7.26 (t, *J* = 7.2 Hz, 2H), 4.59 (d, *J* = 12.1 Hz, 2H), 3.99 (s, 2H), 2.40 (s, 3H), 2.25 (s, 3H), 1.73 (t, *J* = 12.7 Hz, 2H), 1.38 (d, *J* = 13.1 Hz, 2H); **^{13}C NMR** (100 MHz, Acetone) δ 154.5, 145.7, 143.4, 136.2, 131.1, 129.0, 128.1 (128.14, 128.07, 2C), 126.6, 76.6, 68.5, 57.5, 45.5, 21.5, 17.1; **HRMS** (EI) (m/z): $[M]^+$ calcd for $C_{27}H_{28}N_2O_3S$ 460.1821, Found 460.1819.

9) Cross-over Experiment



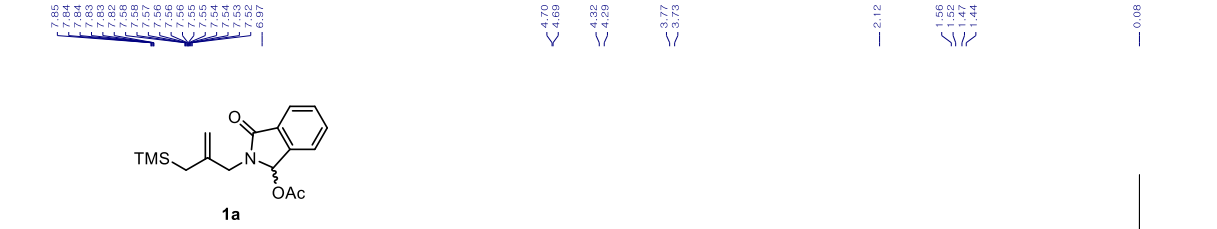
Substrate **1a** (118 mg, 0.372 mmol, 1 equiv) and **1g** (110 mg, 0.372 mmol, 1 equiv) were dissolved in CH_3CN (7.40 mL) under argon atmosphere at room temperature. The resulting mixture was cooled to –40 ℃. After 10 min, TMSOTf (0.135 mL, 0.744 mmol, 2.00 equiv) was added dropwise to the reaction mixture. The resulting mixture was stirred and allowed to warm to 0 ℃ slowly over 2 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution (10 mL) and diluted with $CHCl_3$ (10 mL). The aqueous layer was extracted with $CHCl_3$ (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The resulting residue was purified by flash column chromatography on silica gel ($CHCl_3$: EtOAc = 8 : 2) to obtain the corresponding 1,6-diazecans **3a** (28.1 mg, 20.4%), **3q** (29.3 mg, 22.6%,) and **3g** (25.0 mg, 20.6%,) respectively.

(7aS,16aR)-1,2-dimethyl-6,15-dimethylene-5,6,7,7a,14,15,16,16a-octahydro-3H,12H-pyrrolo[1',2':6,7][1,6]diazecino[2,1-a]isoindole-3,12-dione, 3q

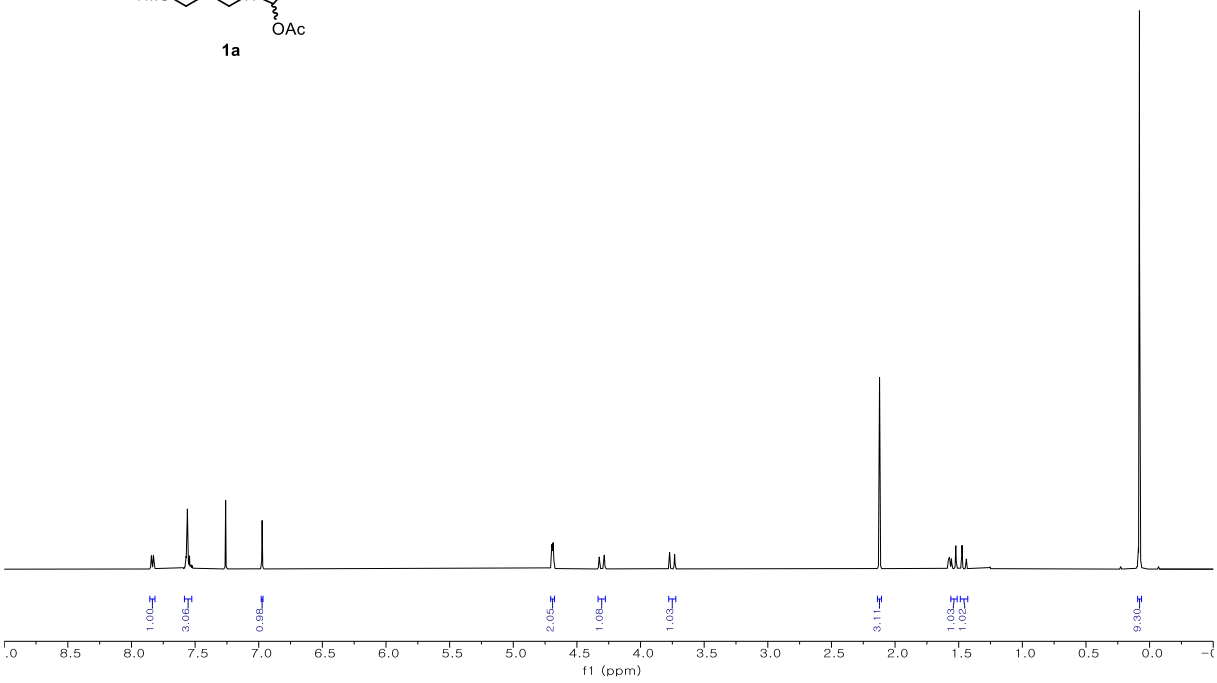


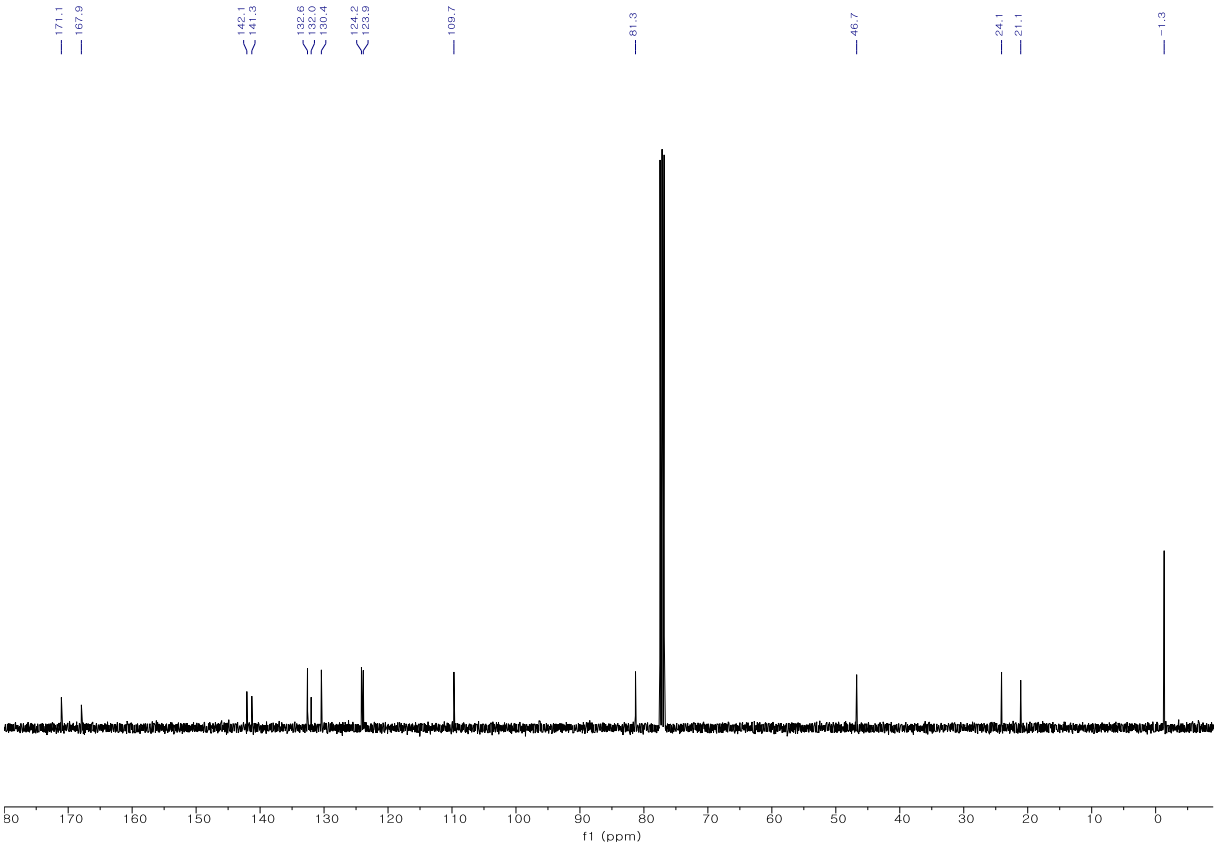
white solid; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.85–7.81 (m, 1H), 7.57 (td, *J* = 7.4, 1.2 Hz, 1H), 7.49–7.43 (m, 2H), 5.03 (s, 1H), 4.93 (s, 1H), 4.83 (s, 1H), 4.80 (dd, *J* = 6.9, 4.2 Hz, 1H), 4.52 (s, 1H), 4.41 (d, *J* = 15.4 Hz, 1H), 4.27 (d, *J* = 15.5 Hz, 1H), 4.12–4.02 (m, 2H), 3.86 (d, *J* = 15.5 Hz, 1H), 3.08 (dd, *J* = 15.2, 4.1 Hz, 1H), 2.86 (dd, *J* = 14.6, 4.3 Hz, 1H), 2.60 (dd, *J* = 15.3, 6.8 Hz, 1H), 2.39 (dd, *J* = 15.2, 6.7 Hz, 1H), 1.97 (s, 3H), 1.81 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.8, 169.9, 150.3, 145.7, 140.9, 140.7, 132.0, 131.9, 129.1, 128.4, 123.9, 122.5, 116.1, 115.9, 62.9, 59.5, 47.8, , 47.7, 36.4, 34.2, 12.5, 8.9; **HRMS** (ESI) (m/z): $[M+Na]^+$ calcd for $C_{22}H_{24}N_2NaO_2$ 371.1730, Found 371.1731.

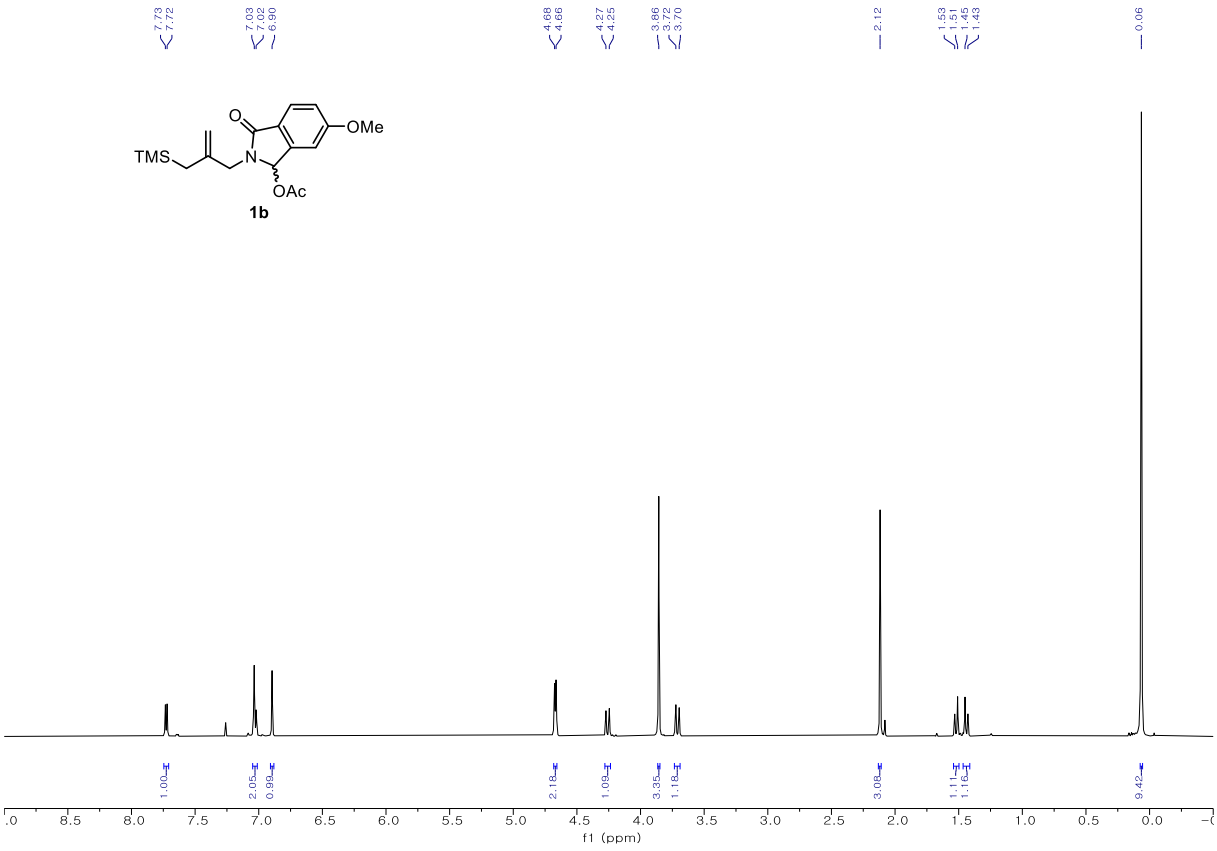
2. ^{1}H, ^{13}C and ^{19}F NMR Spectra of Compounds



1a



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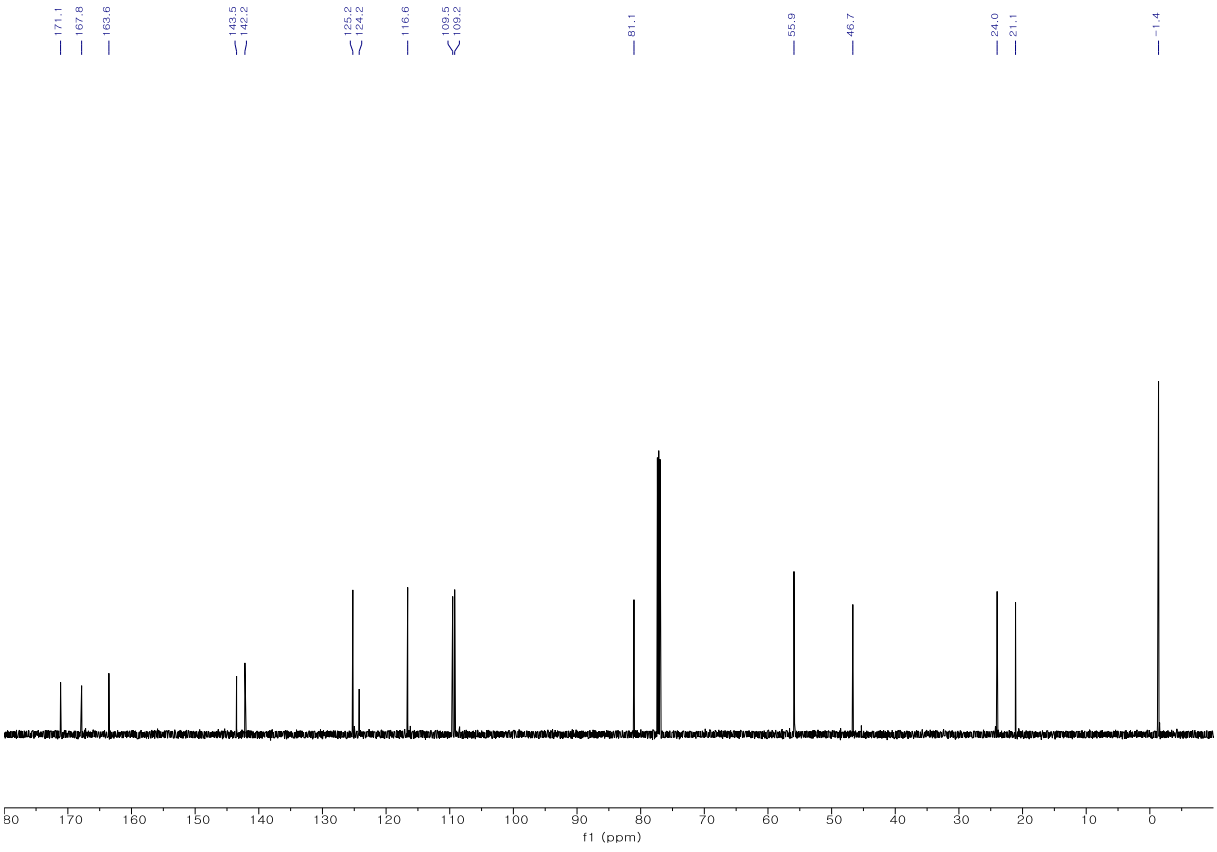
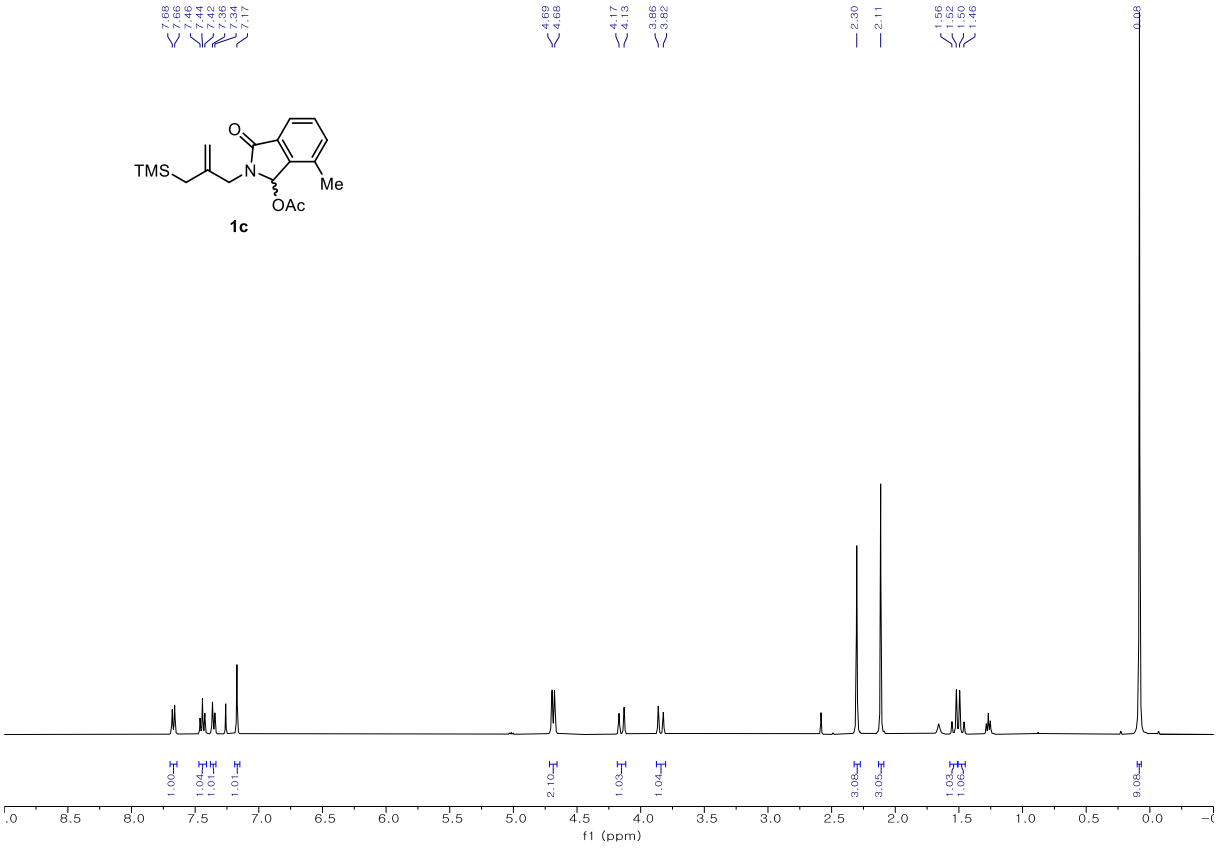
7.72
7.03
7.02
6.90
4.68
4.66
4.27
4.25
3.86
3.72
3.70
2.12
1.53
1.51
1.45
1.43
0.06
O
OMe
TMS
N
OAc
1b
1.00
2.05
0.99
2.18
1.09
3.35
1.18
3.08
1.11
1.16
9.42
f1 (ppm)


171.1
167.8
163.6
143.5
142.2
125.2
124.2
116.6
109.5
109.2
81.1
55.9
46.7
24.0
21.1
1.4
f1 (ppm)


7.68
7.66
7.46
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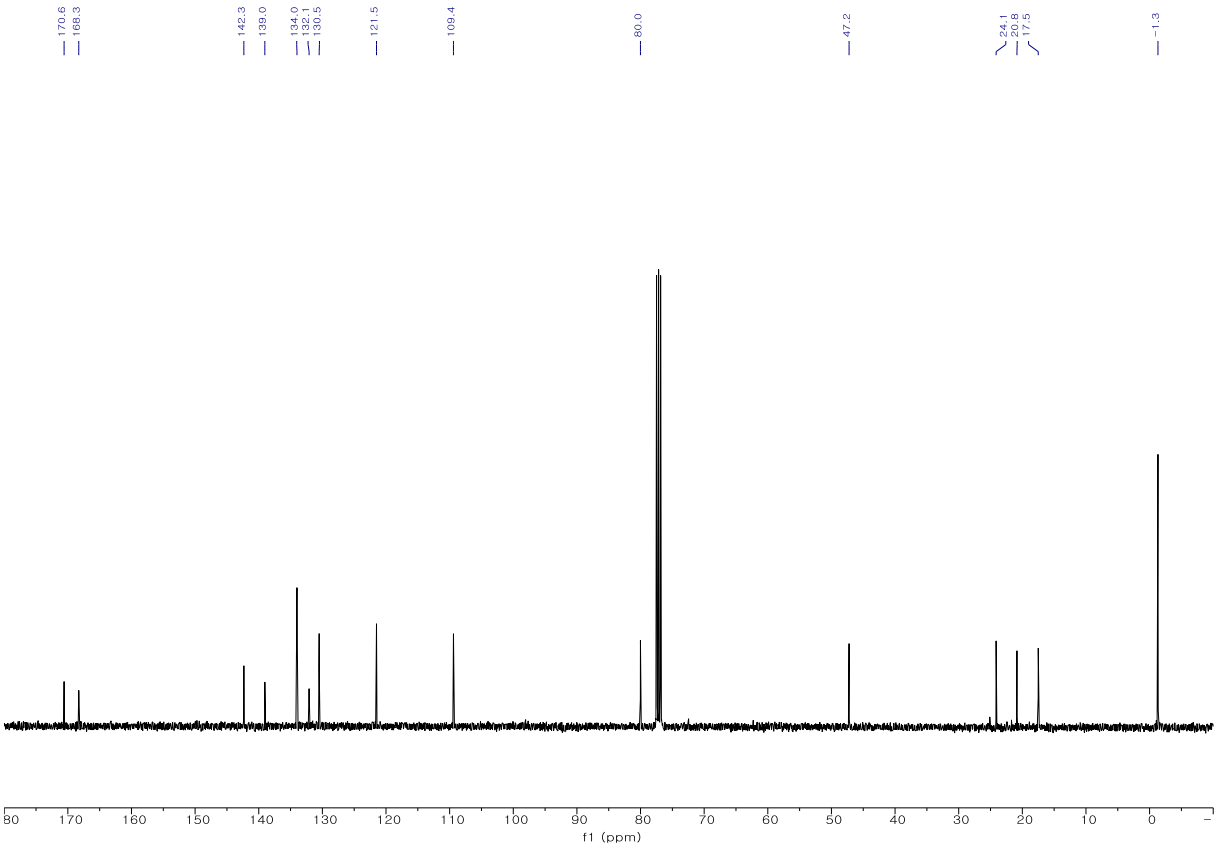
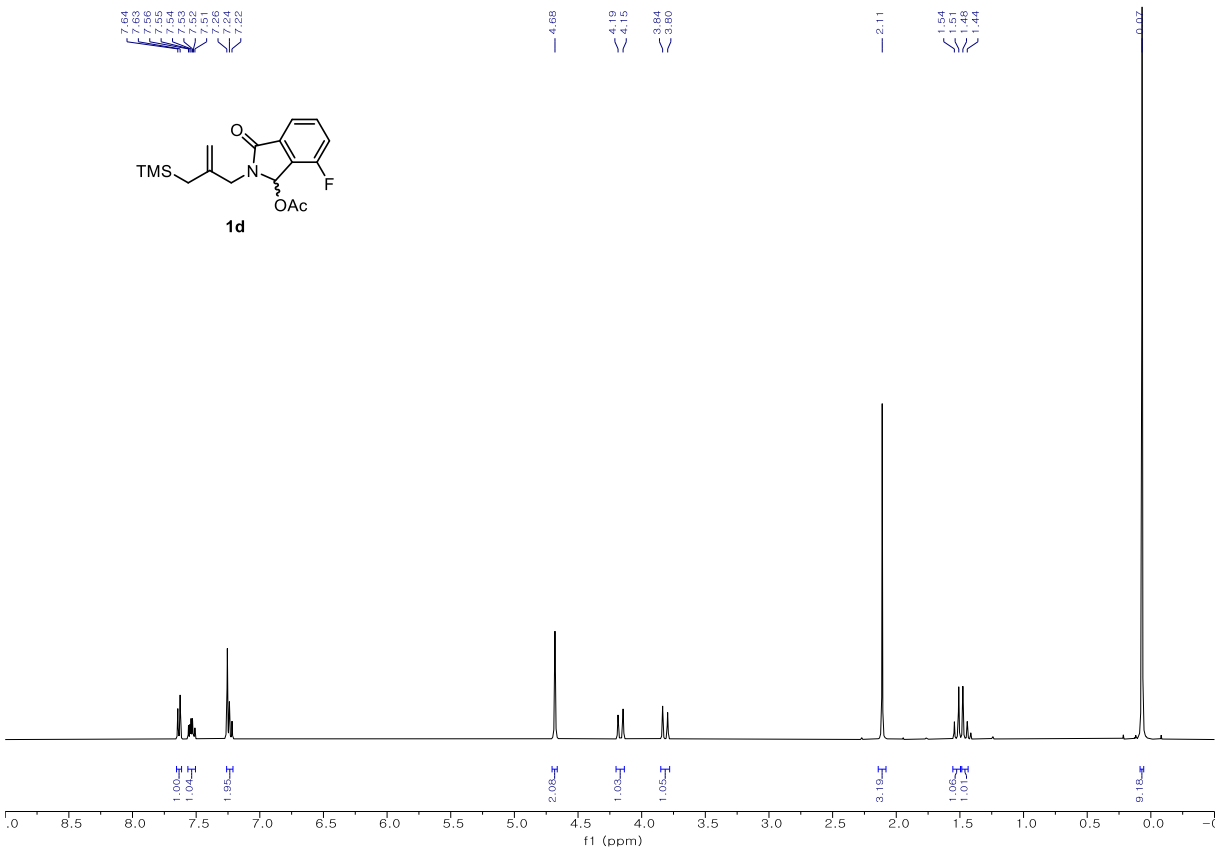
7.42
7.36
7.34
7.17
4.69
4.68
4.17
4.13
3.86
3.82
2.30
2.11
1.56
1.52
1.50
1.46
0.08
O
N
TMS
Me
OAc
1c
1.00
1.04
1.01
1.01
2.10
1.03
1.04
3.08
3.05
1.03
1.06
9.08
f1 (ppm)


170.6
168.3
142.3
139.0
134.0
132.1
130.5
121.5
109.4
80.0
47.2
24.1
20.8
17.5
-1.3
f1 (ppm)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7.63
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7.55
7.54
7.53
7.52
7.51
7.26
7.24
7.22
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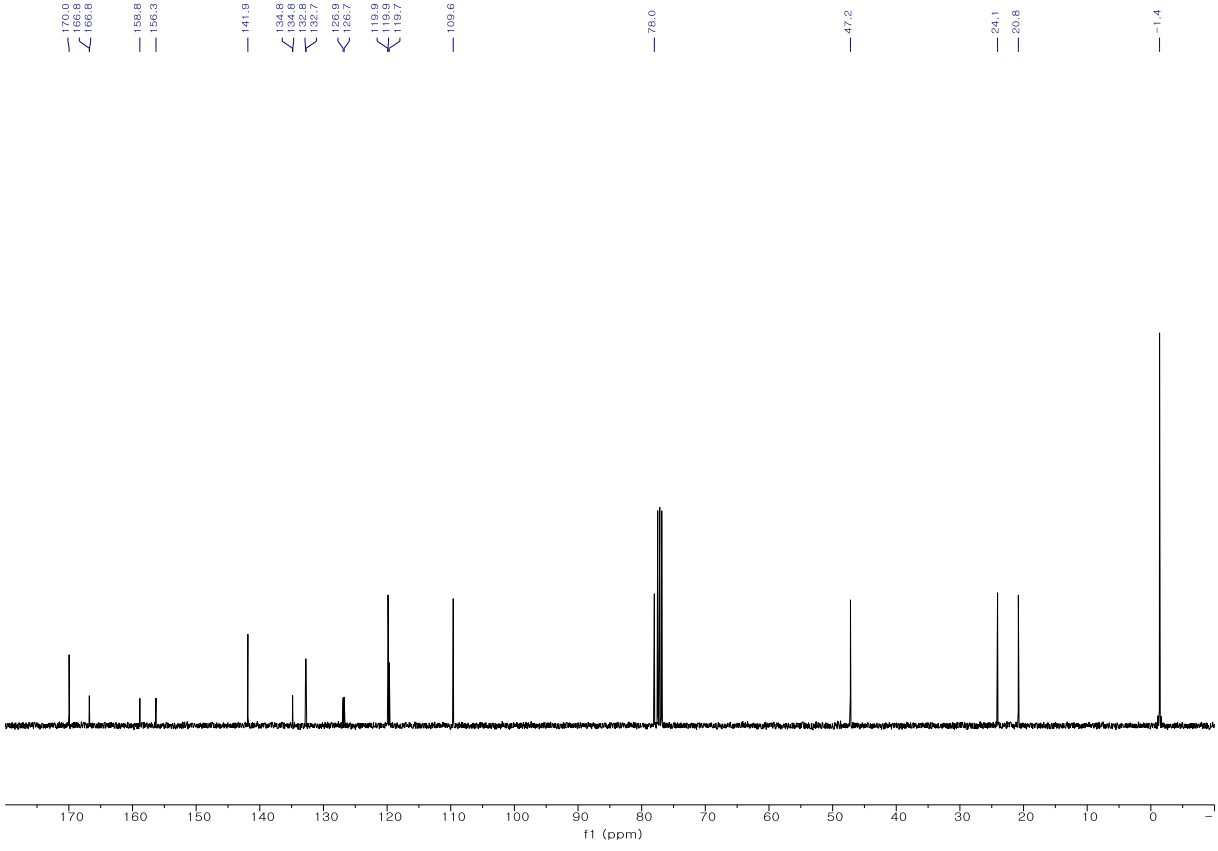
4.19
4.15
3.84
3.80
2.11
1.54
1.51
1.48
1.44
0.07
O
N
TMS
F
OAc
1d
1.00
1.04
1.95
2.06
1.03
1.05
3.19
1.06
1.01
9.18
f1 (ppm)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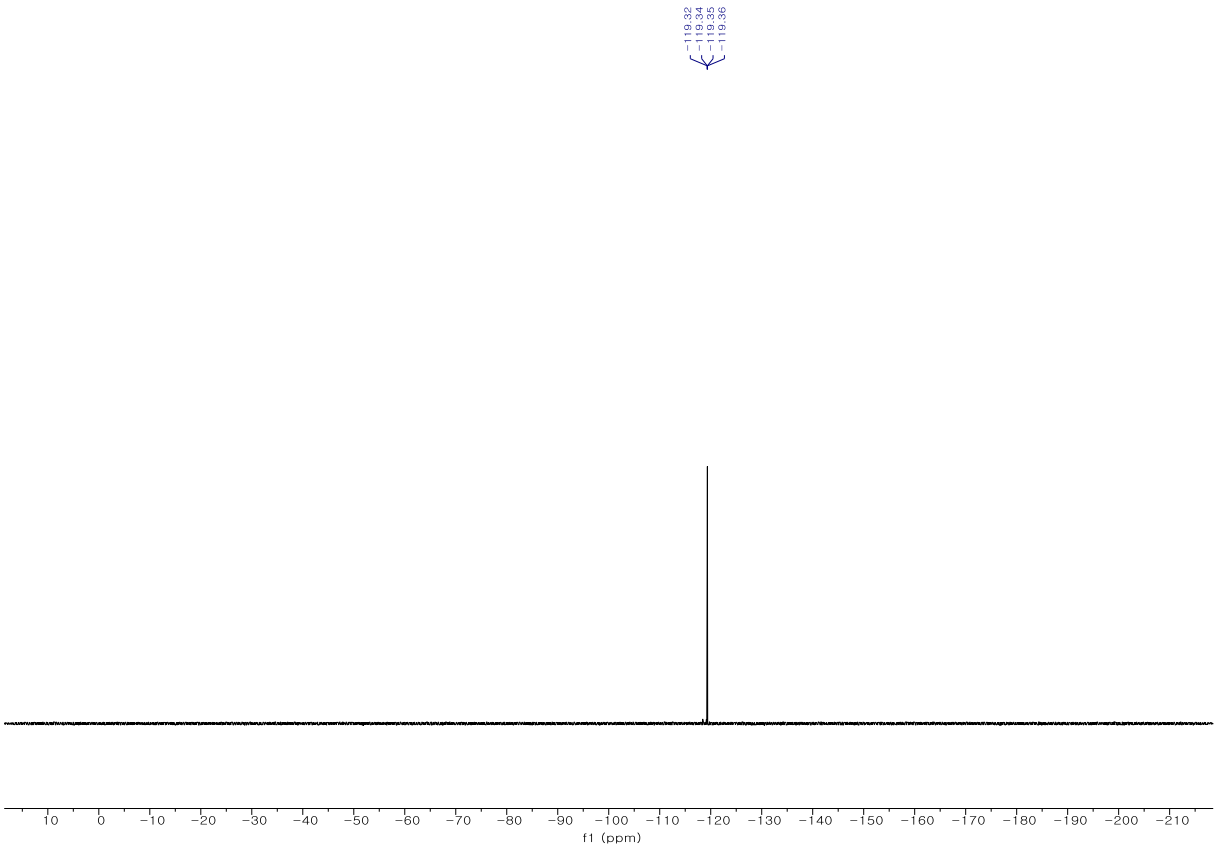
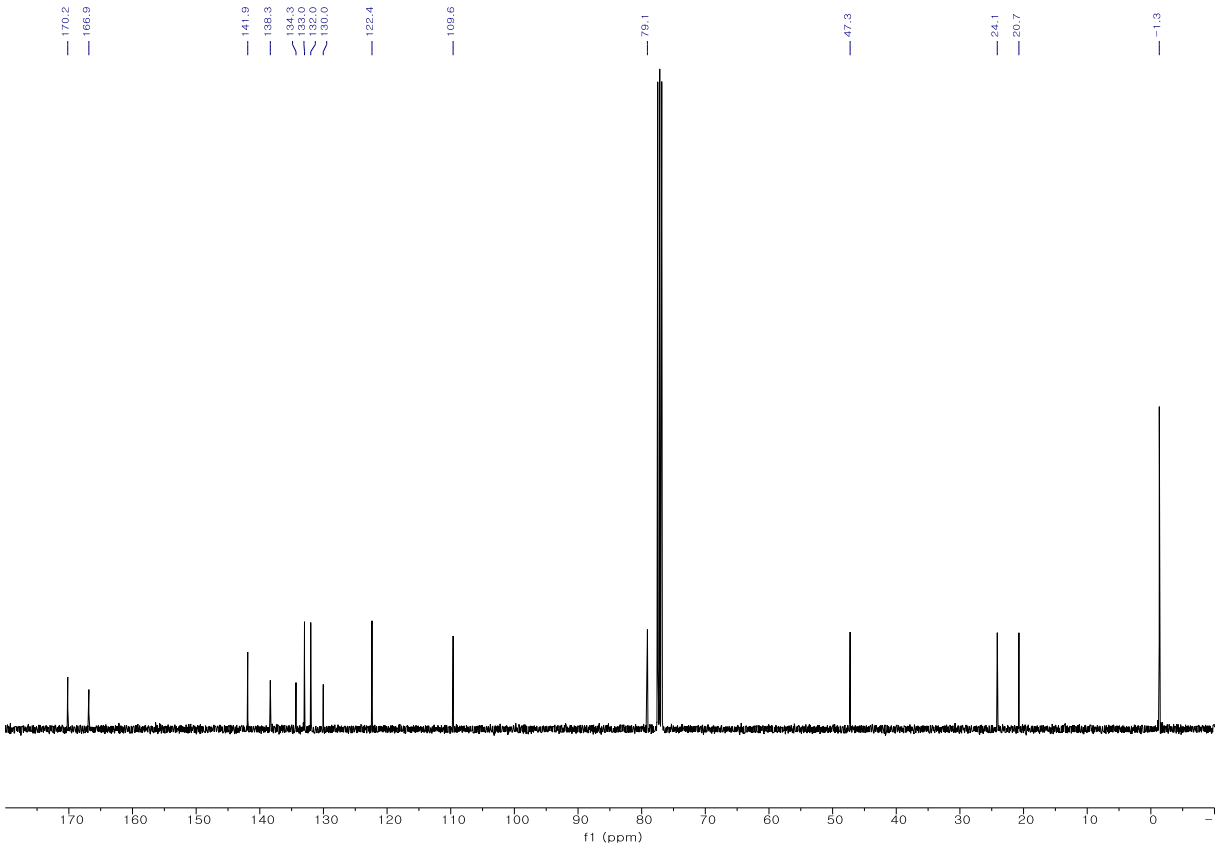
166.8
158.8
156.3
141.9
134.8
134.8
132.8
132.7
126.9
126.7
119.9
119.9
119.7
109.6
78.0
47.2
24.1
20.8
-1.4
f1 (ppm)


−119.32
−119.34
−119.35
−119.36
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)


7.76
7.75
7.74
7.74
7.55
7.54
7.53
7.52
7.51
7.50
7.49
7.21
4.70
4.70
4.70
4.69
4.69
4.17
4.13
3.86
3.82
2.13
1.55
1.52
1.50
1.46
0.08
TMS
O
N
Cl
OAc
1e
1.00
2.05
0.99
2.11
1.05
1.04
3.16
1.05
1.04
9.44
f1 (ppm)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134.3
133.0
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130.0
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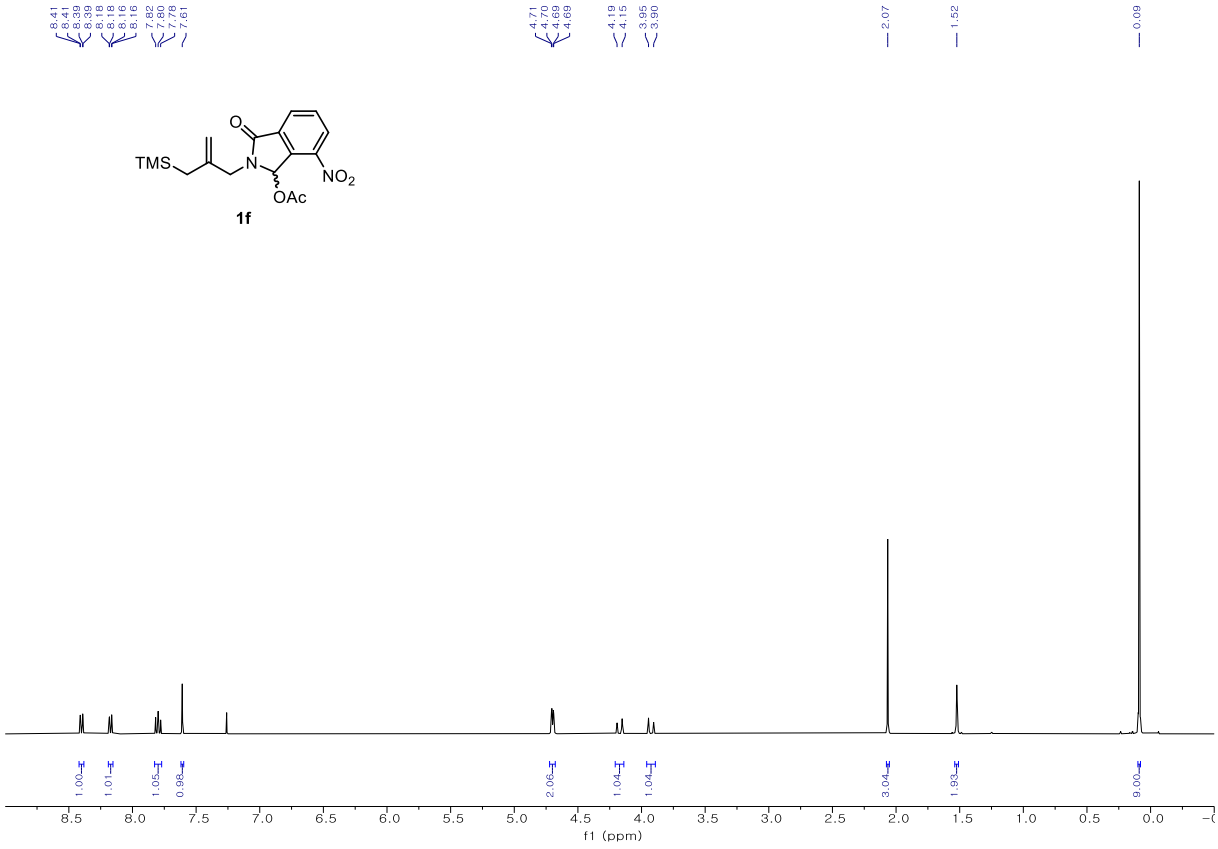
109.6
79.1
47.3
24.1
20.7
-1.3
f1 (ppm)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8.41
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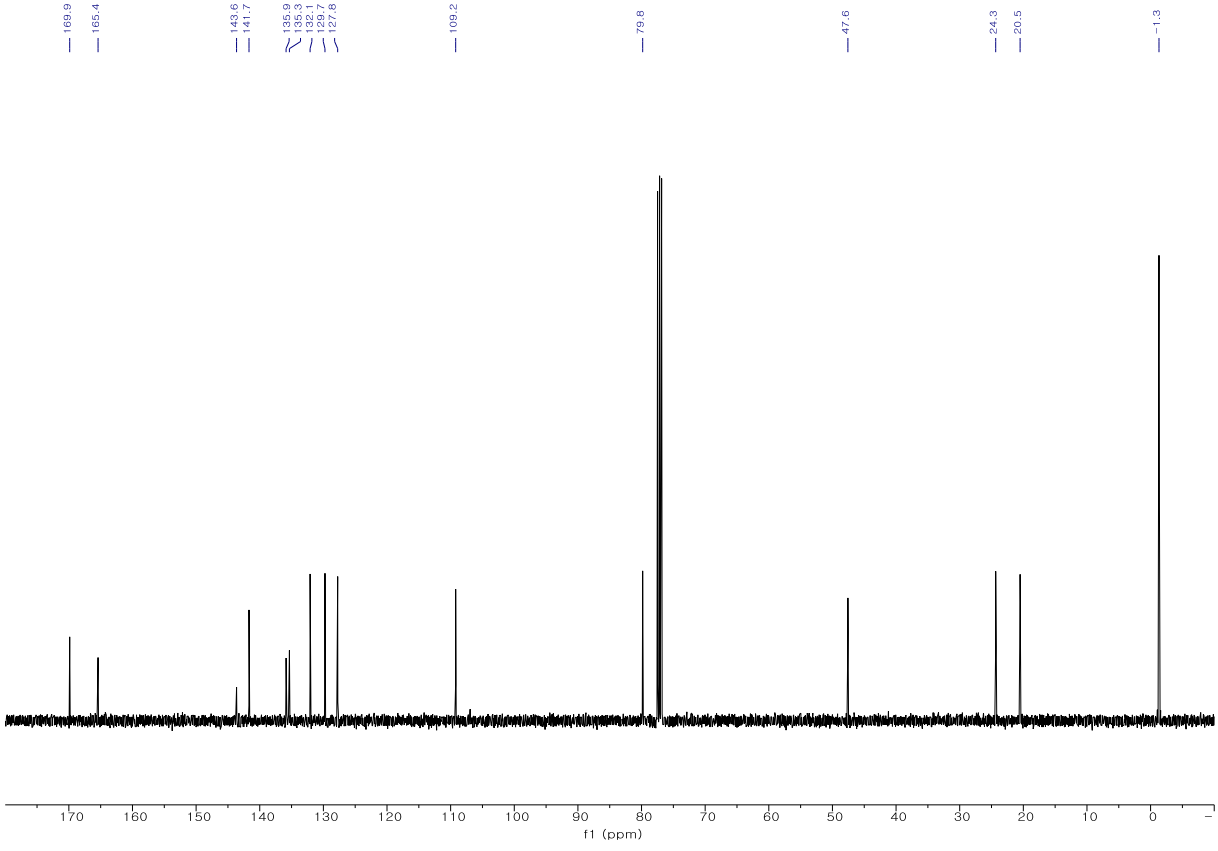
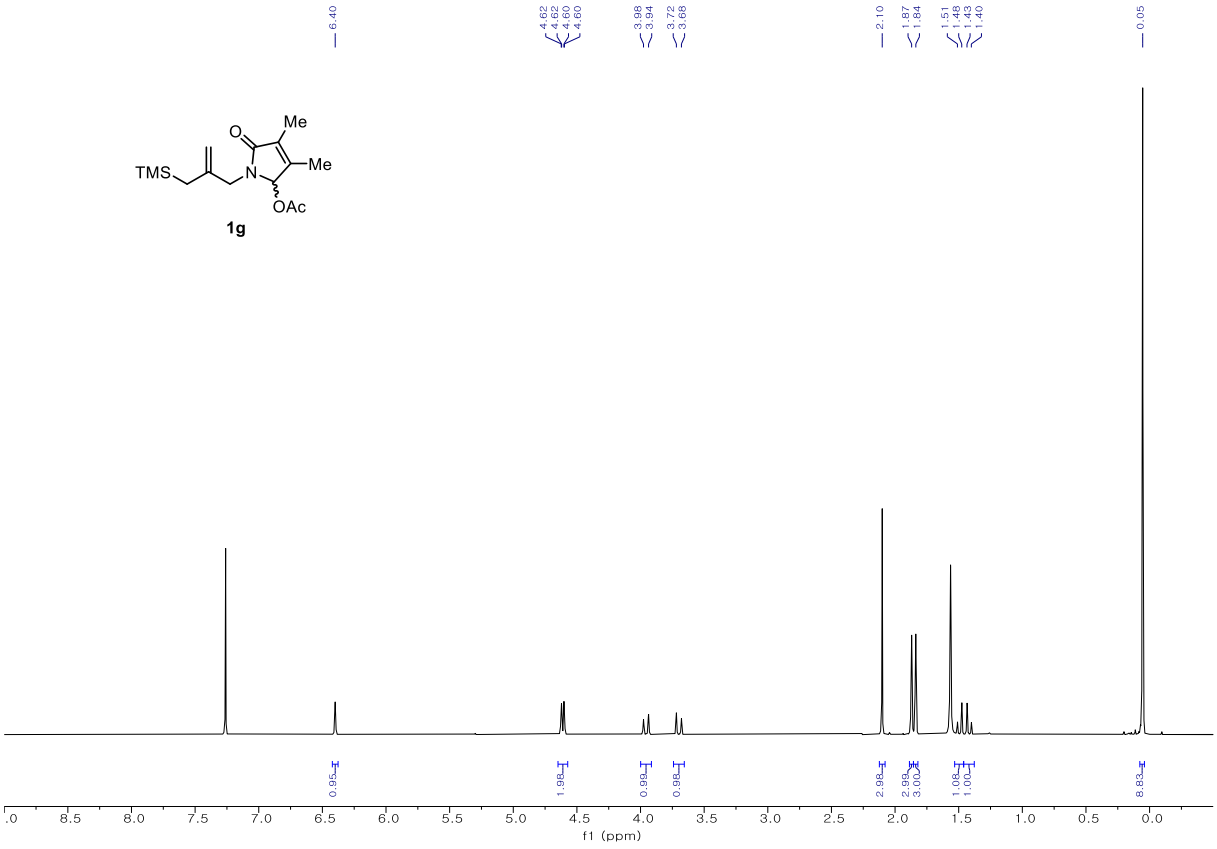
8.39
8.18
8.18
8.16
8.16
7.82
7.80
7.78
7.61
4.71
4.70
4.69
4.69
4.19
4.15
3.95
3.90
2.07
1.52
0.09
O
N
TMS
NO2
OAc
1f
1.00
1.01
1.05
0.98
2.06
1.04
1.04
3.04
1.93
9.00
f1 (ppm)


169.9
165.4
143.6
141.7
135.9
135.3
132.1
129.7
127.8
109.2
79.8
47.6
24.3
20.5
-1.3
f1 (ppm)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4.62
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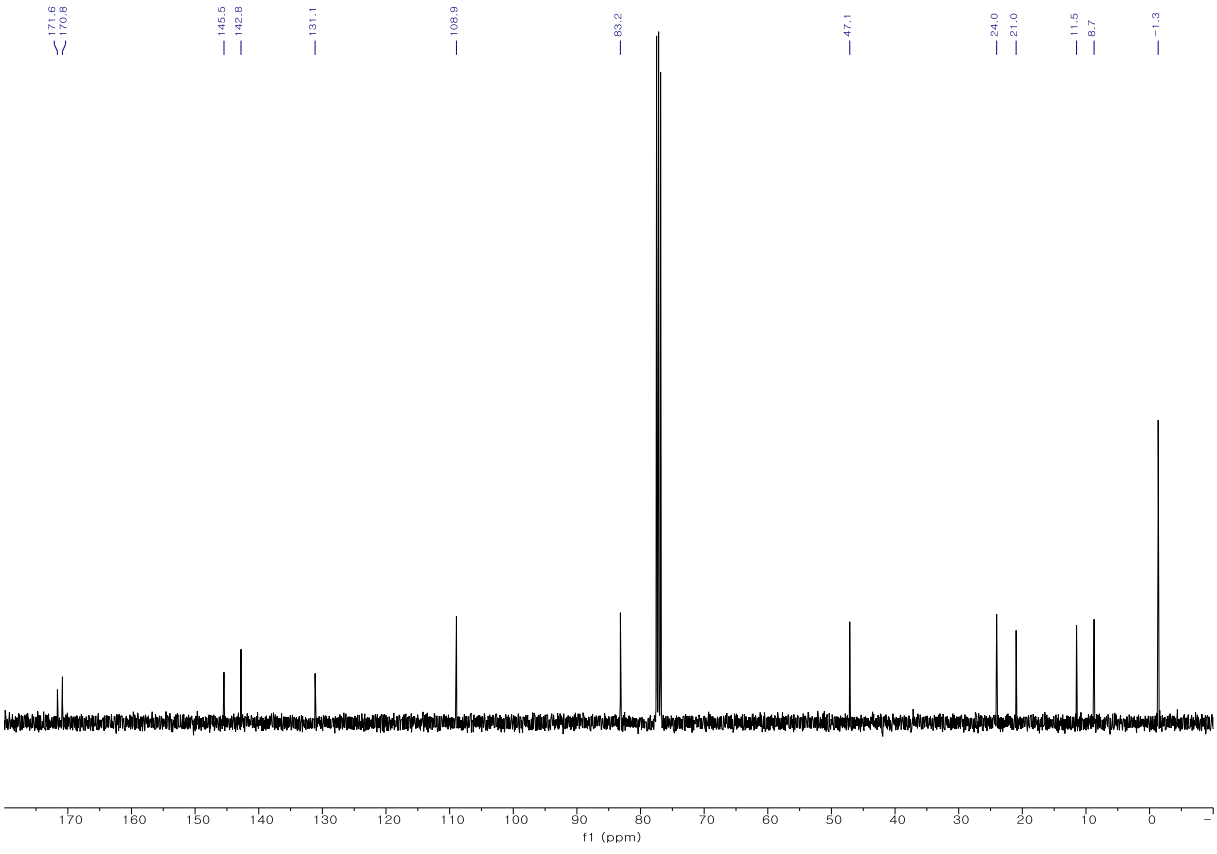
4.60
3.98
3.94
3.72
3.68
2.10
1.87
1.84
1.51
1.48
1.43
1.40
0.05
Me
O
Me
TMS
N
OAc
1g
0.95
1.98
0.99
0.98
2.98
2.99
3.00
1.08
1.00
8.83
f1 (ppm)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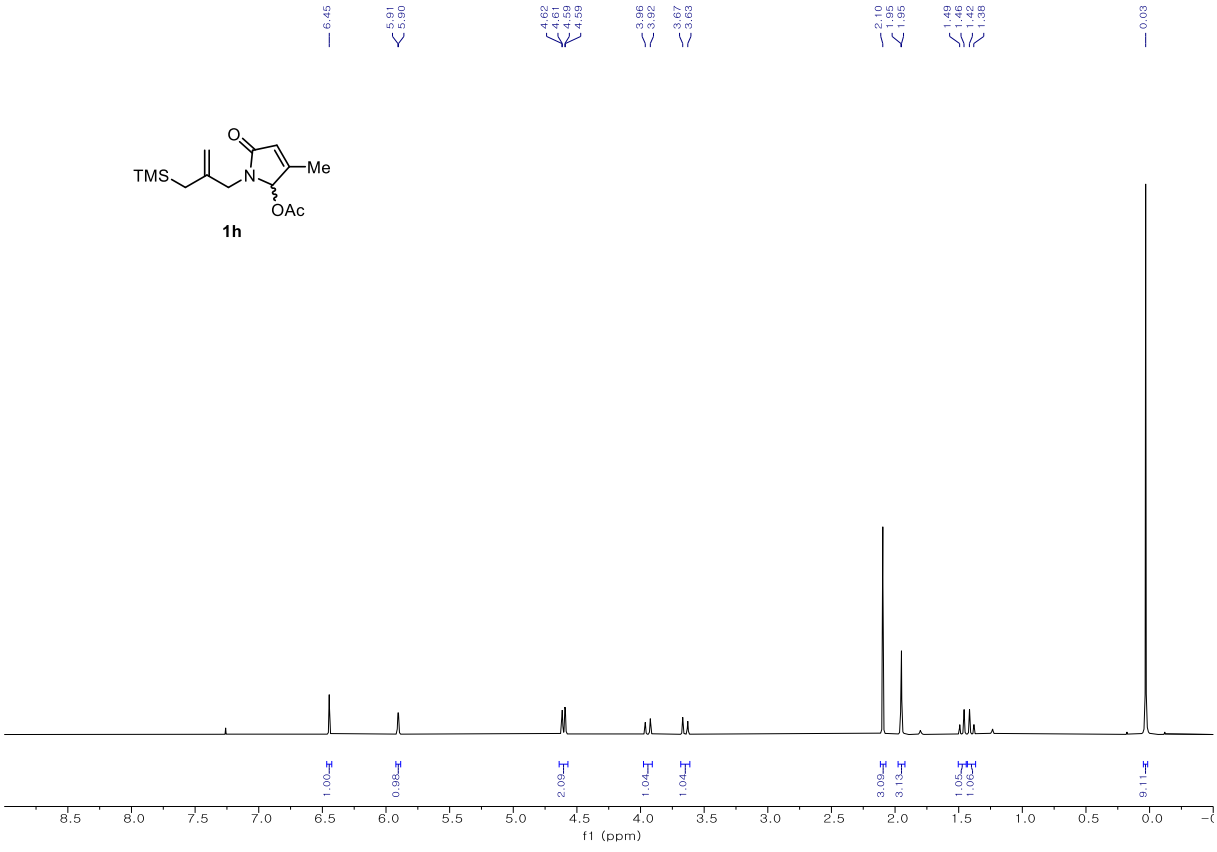
170.8
145.5
142.8
131.1
108.9
83.2
47.1
24.0
21.0
11.5
8.7
-1.3
f1 (ppm)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5.91
5.90
4.62
4.61
4.59
4.59
3.96
3.92
3.67
3.63
2.10
1.95
1.95
1.49
1.46
1.42
1.38
0.03
O
TMS
N
Me
OAc
1h
1.00
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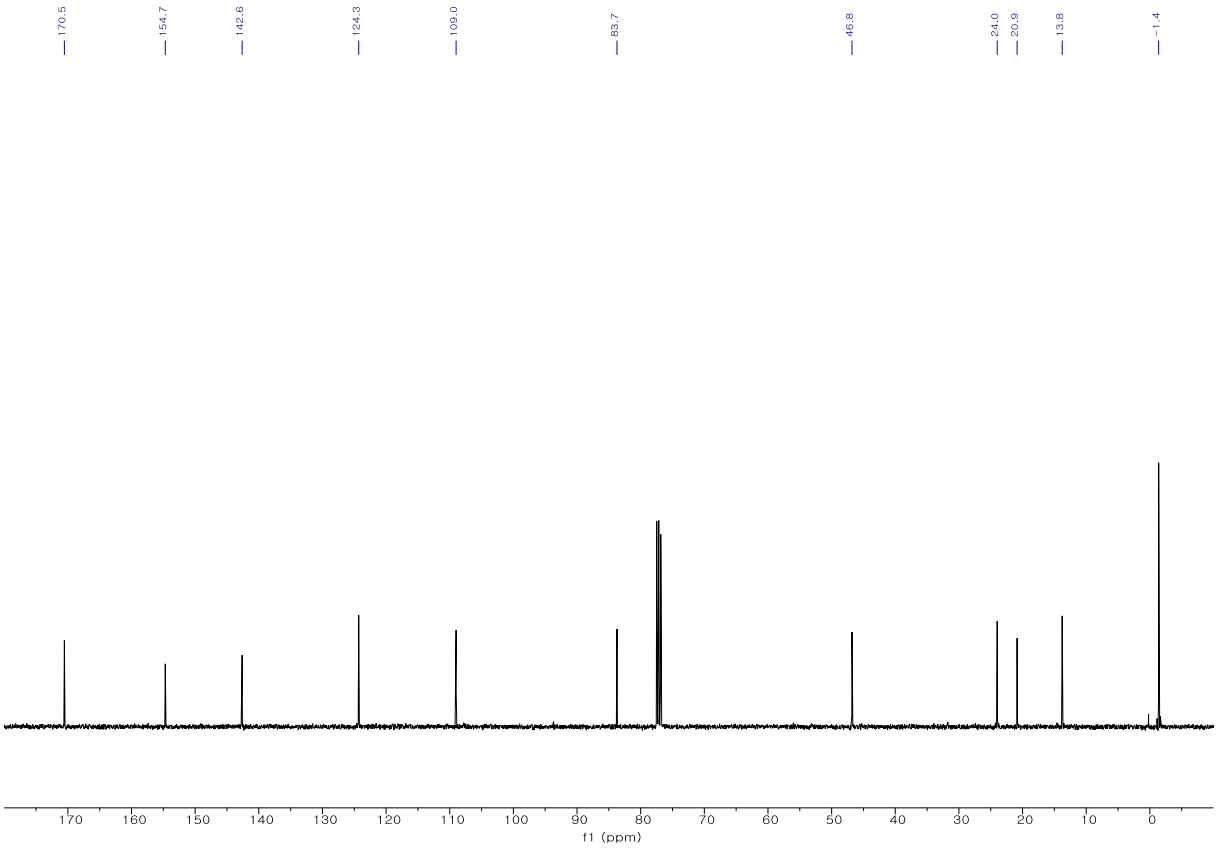
2.09
1.04
1.04
3.09
3.13
1.05
1.06
9.11
f1 (ppm)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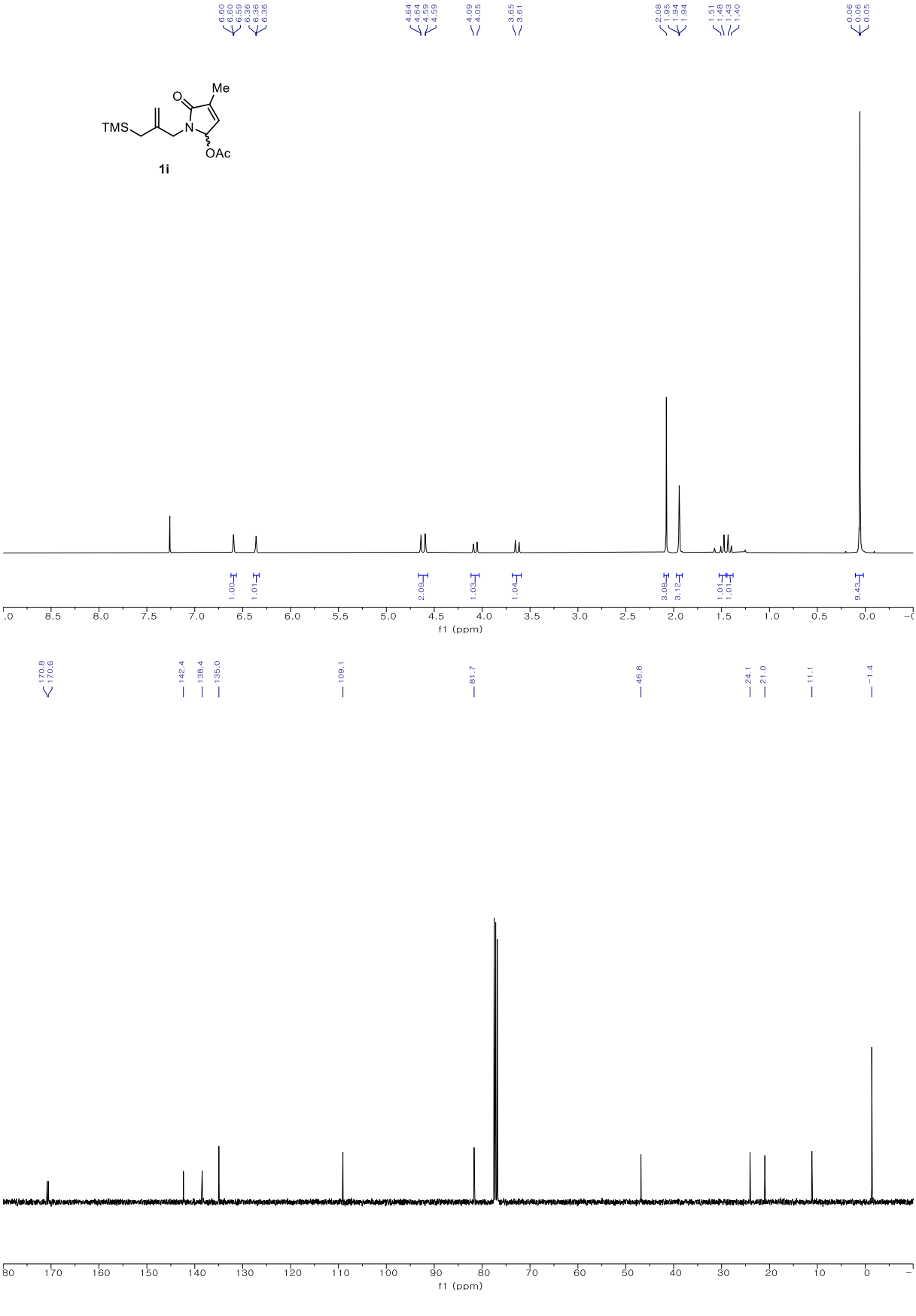
83.7
46.8
24.0
20.9
13.8
1.4
f1 (ppm)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6.36
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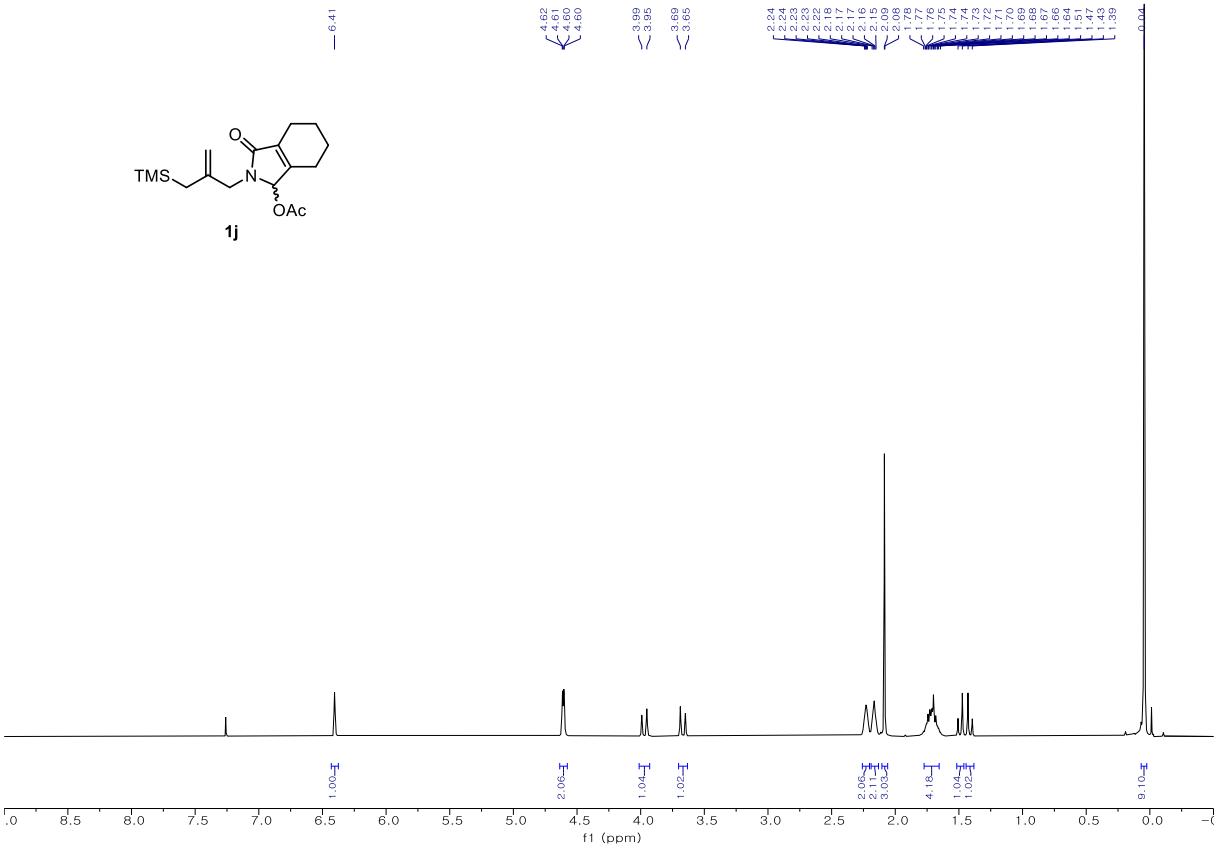
4.64
4.59
4.59
4.09
4.05
3.65
3.61
2.08
1.95
1.94
1.94
1.51
1.48
1.43
1.40
0.06
0.06
0.05
Me
O
N
TMS
OAc
1i
1.00
1.01
2.09
1.03
1.04
3.08
3.12
1.01
1.01
9.43
f1 (ppm)
170.8
170.6
142.4
138.4
135.0
109.1
81.7
46.8
24.1
21.0
11.1
-1.4
f1 (ppm)


TMS
O
N
OAc
1j
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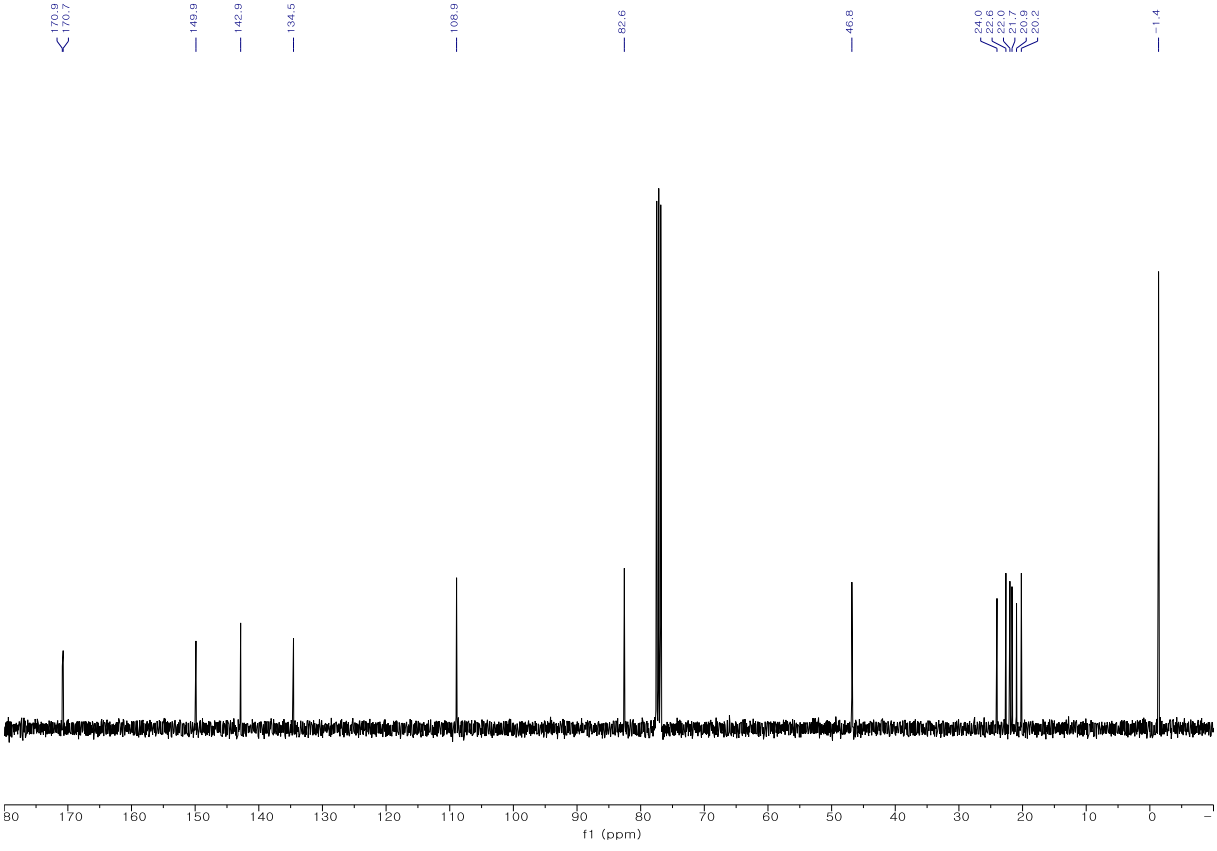
4.61
4.60
3.99
3.95
3.69
3.65
2.24
2.23
2.22
2.18
2.17
2.16
2.15
2.09
2.08
1.78
1.77
1.76
1.75
1.74
1.73
1.72
1.71
1.70
1.69
1.68
1.67
1.66
1.64
1.51
1.47
1.43
1.39
0.04
1.00
2.06
1.04
1.02
2.06
2.11
3.03
4.16
1.04
1.02
9.10
f1 (ppm)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170.7
149.9
142.9
134.5
108.9
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46.8
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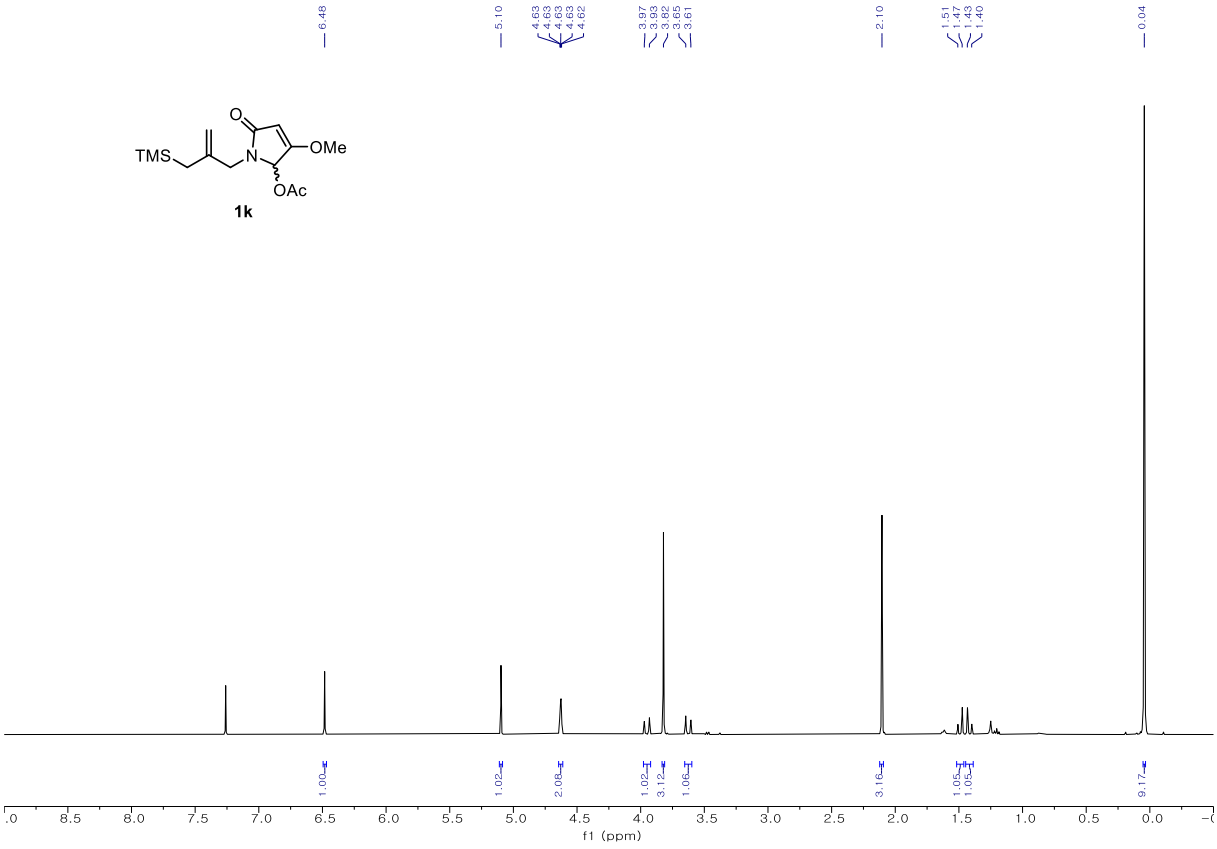
22.6
22.0
21.7
20.9
20.2
1.4
f1 (ppm)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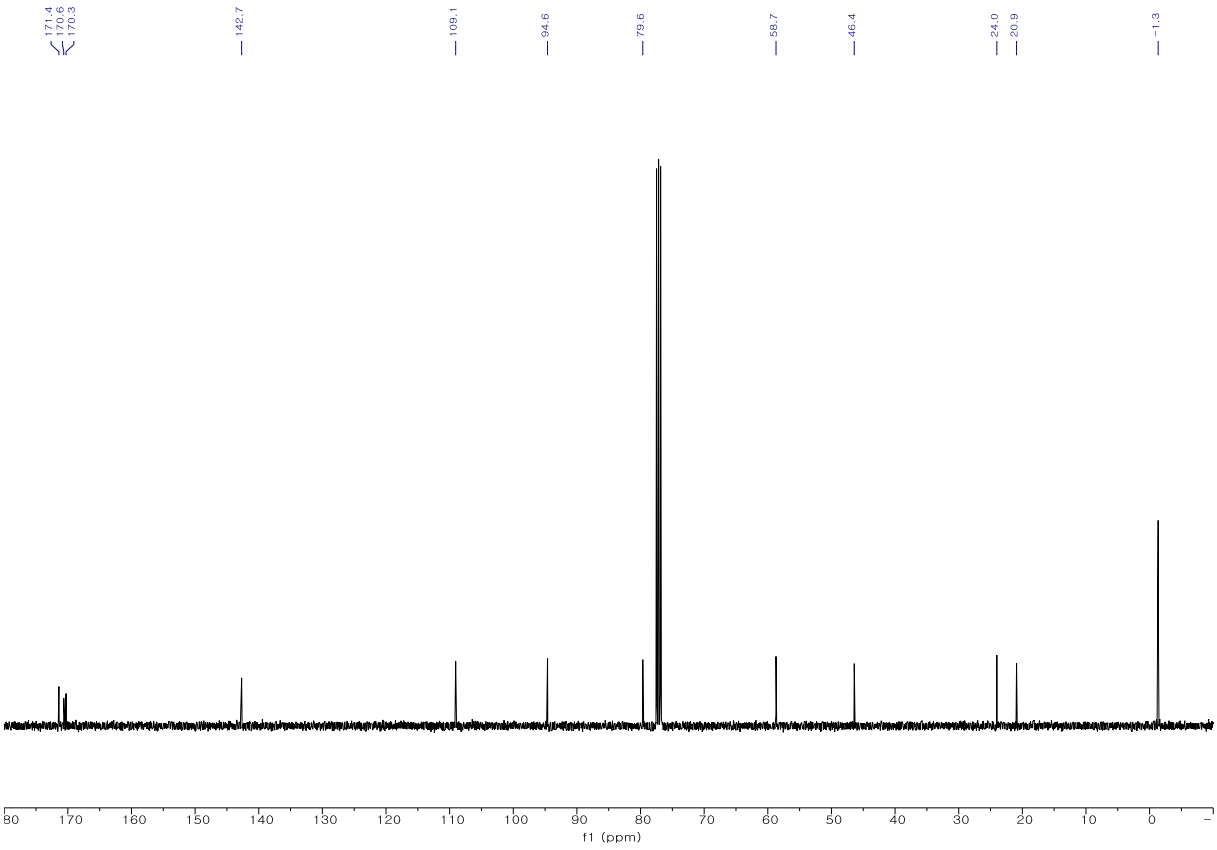
5.10
4.63
4.63
4.63
4.62
3.97
3.93
3.82
3.65
3.61
2.10
1.51
1.47
1.43
1.40
0.04
TMS
O
N
OMe
OAc
1k
1.00
1.02
2.08
1.02
3.12
1.06
3.16
1.05
1.05
9.17
f1 (ppm)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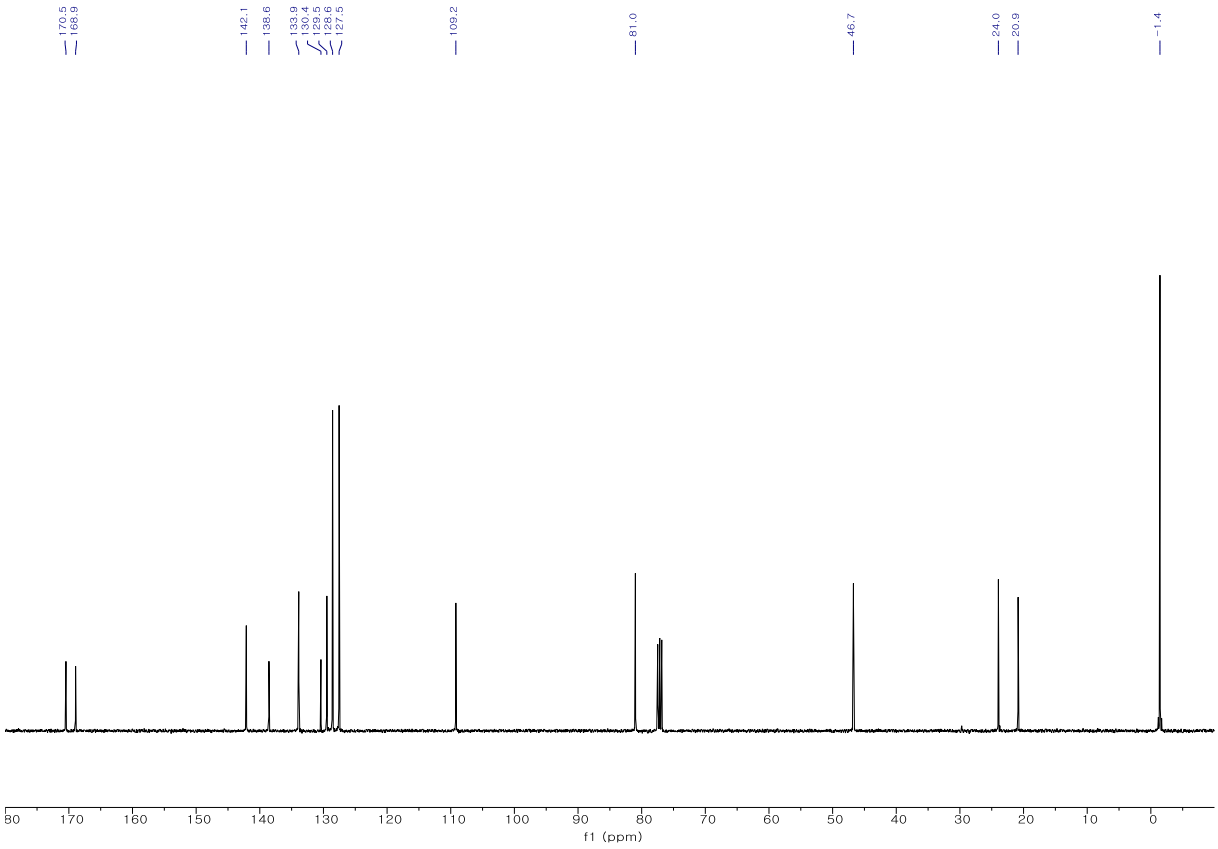
170.6
170.3
142.7
109.1
94.6
79.6
58.7
46.4
24.0
20.9
-1.3
f1 (ppm)


7.89
7.88
7.41
7.40
7.39
7.38
7.37
7.36
7.35
7.35
7.08
6.49
4.67
4.66
4.19
4.15
3.73
3.69
2.10
1.54
1.50
1.47
1.44
0.07
Ph
O
N
TMS
OAc
1l
2.00
3.16
0.95
0.96
1.97
1.02
1.00
2.86
1.15
0.99
8.94
f1 (ppm)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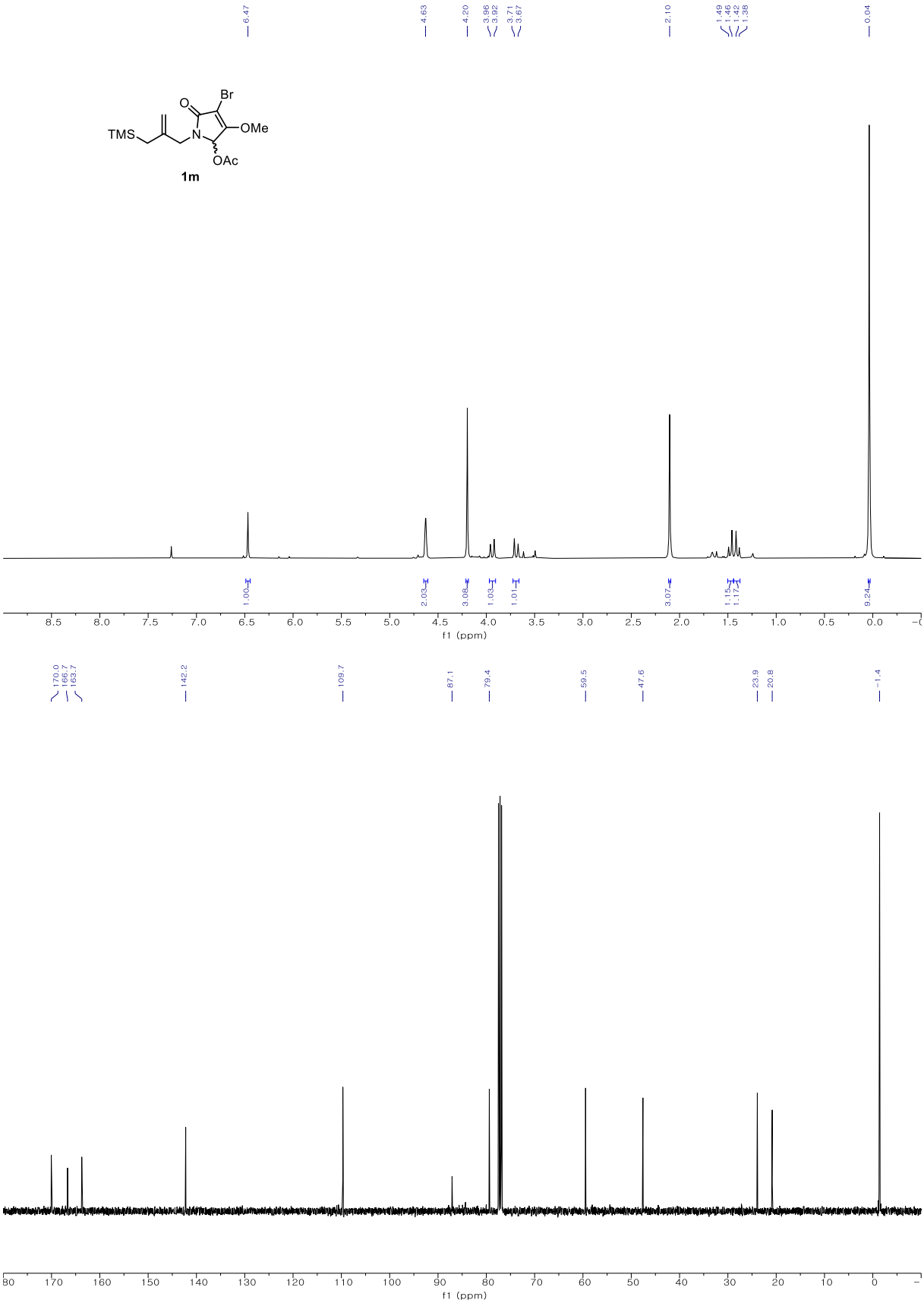
168.9
142.1
138.6
133.9
130.4
129.5
128.6
127.5
109.2
81.0
46.7
24.0
20.9
-1.4
f1 (ppm)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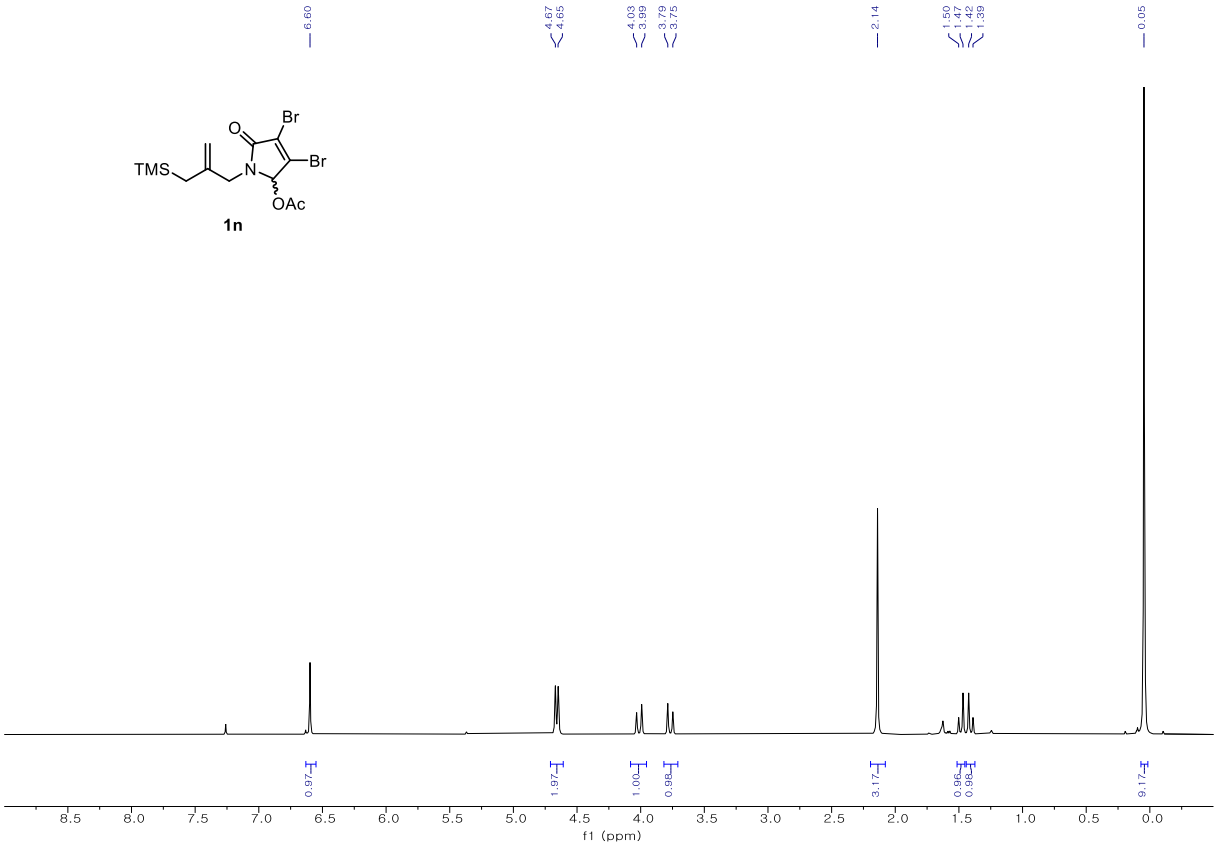
4.20
3.96
3.92
3.71
3.67
2.10
1.49
1.46
1.42
1.38
0.04
Br
O
TMS
N
OMe
OAc
1m
1.00
2.03
3.08
1.03
1.01
3.07
1.15
1.17
9.24
f1 (ppm)
170.0
166.7
163.7
142.2
109.7
87.1
79.4
59.5
47.6
23.9
20.8
1.4
f1 (ppm)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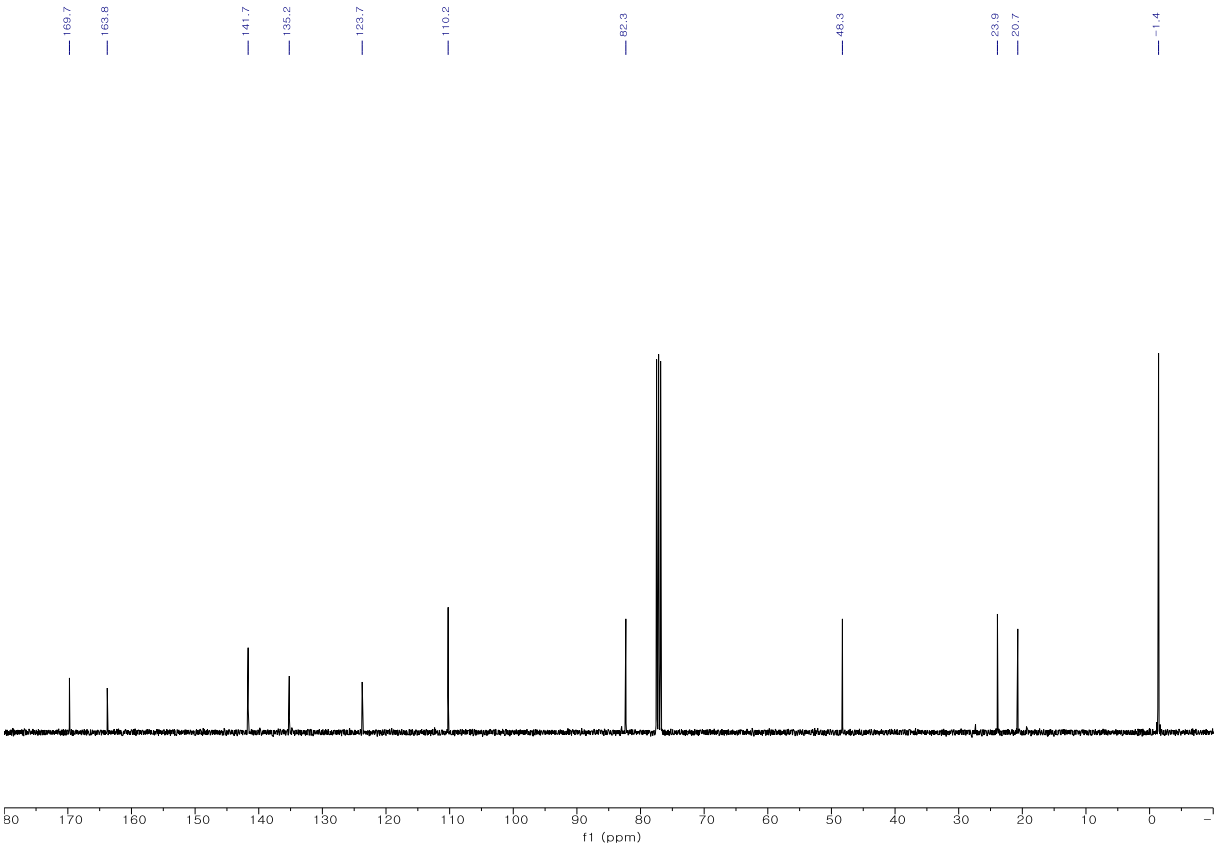
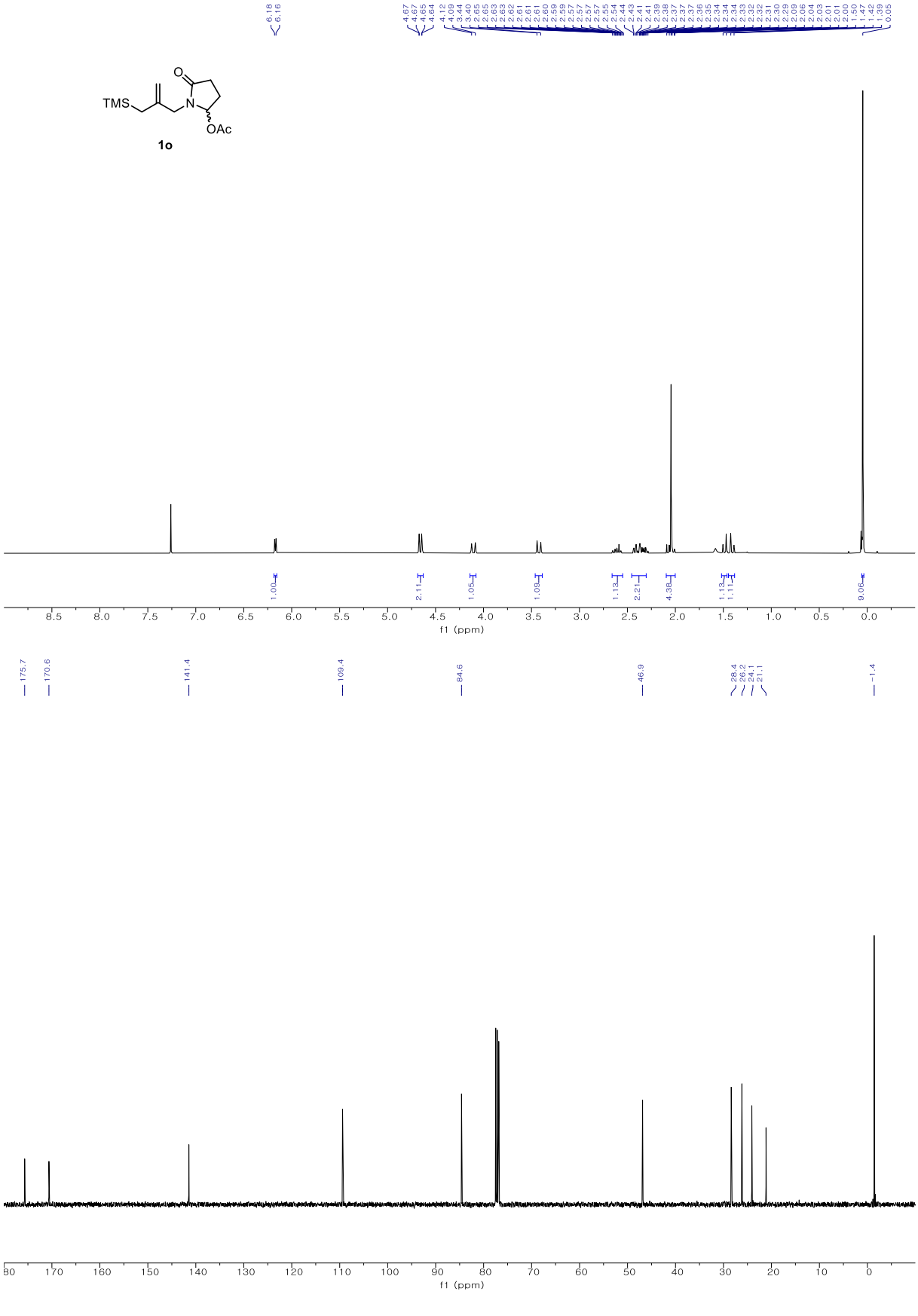
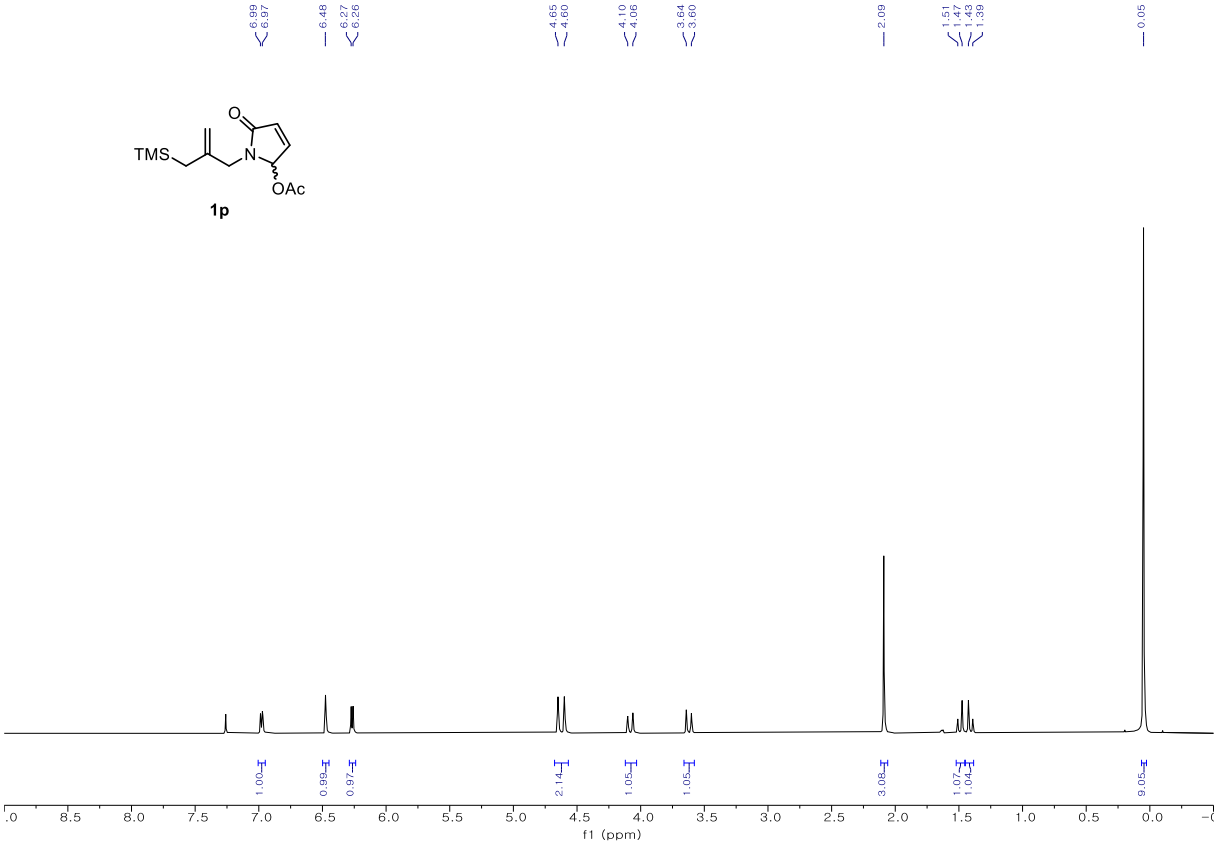
4.67
4.65
4.03
3.99
3.79
3.75
2.14
1.50
1.47
1.42
1.39
0.05
Br
O
TMS
N
Br
OAc
1n
0.97
1.97
1.00
0.98
3.17
0.96
0.98
9.17
f1 (ppm)


169.7
163.8
141.7
135.2
123.7
110.2
82.3
48.3
23.9
20.7
1.4
f1 (ppm)


TMS
O
N
OAc
1o
f1 (ppm)
f1 (ppm)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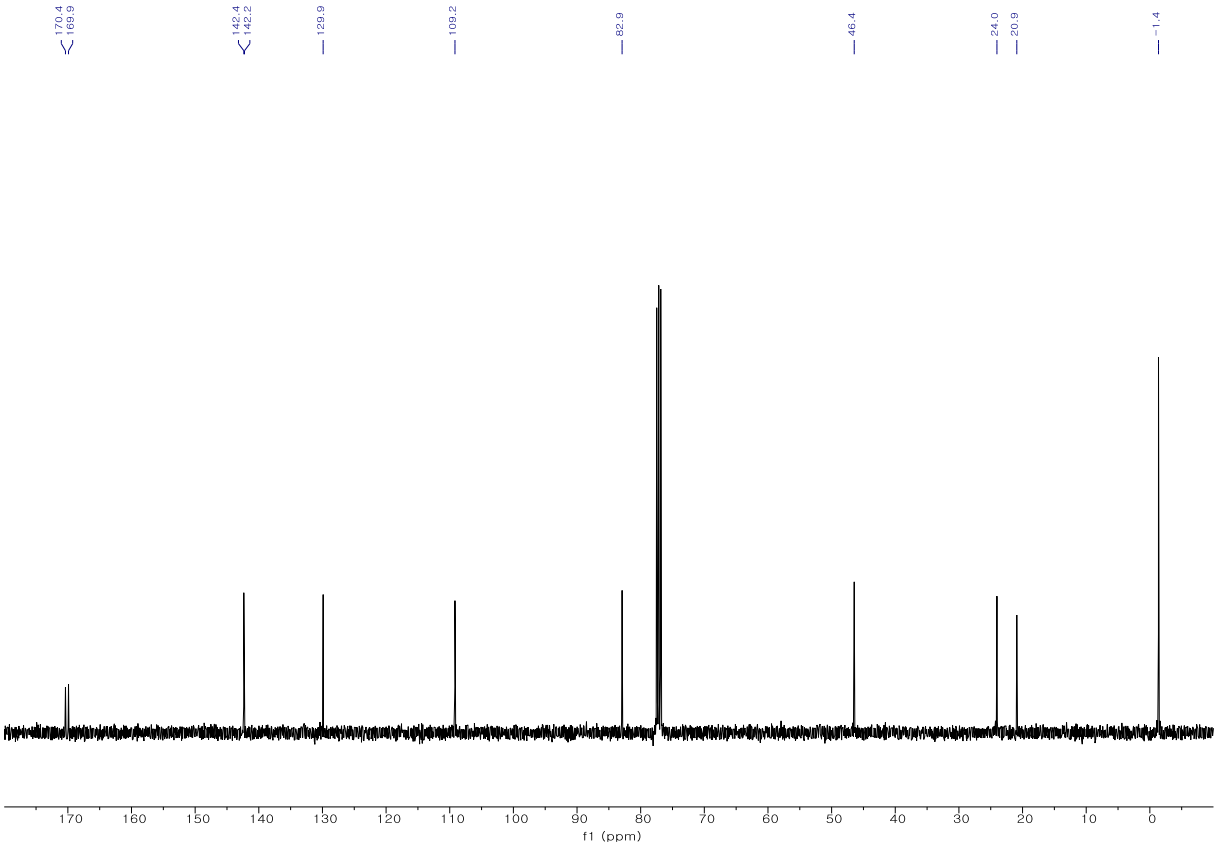
6.97
6.48
6.27
6.26
4.65
4.60
4.10
4.06
3.64
3.60
2.09
1.51
1.47
1.43
1.39
0.05
O
N
TMS
OAc
1p
1.00
0.99
0.97
2.14
1.05
1.05
3.08
1.07
1.04
9.05
f1 (ppm)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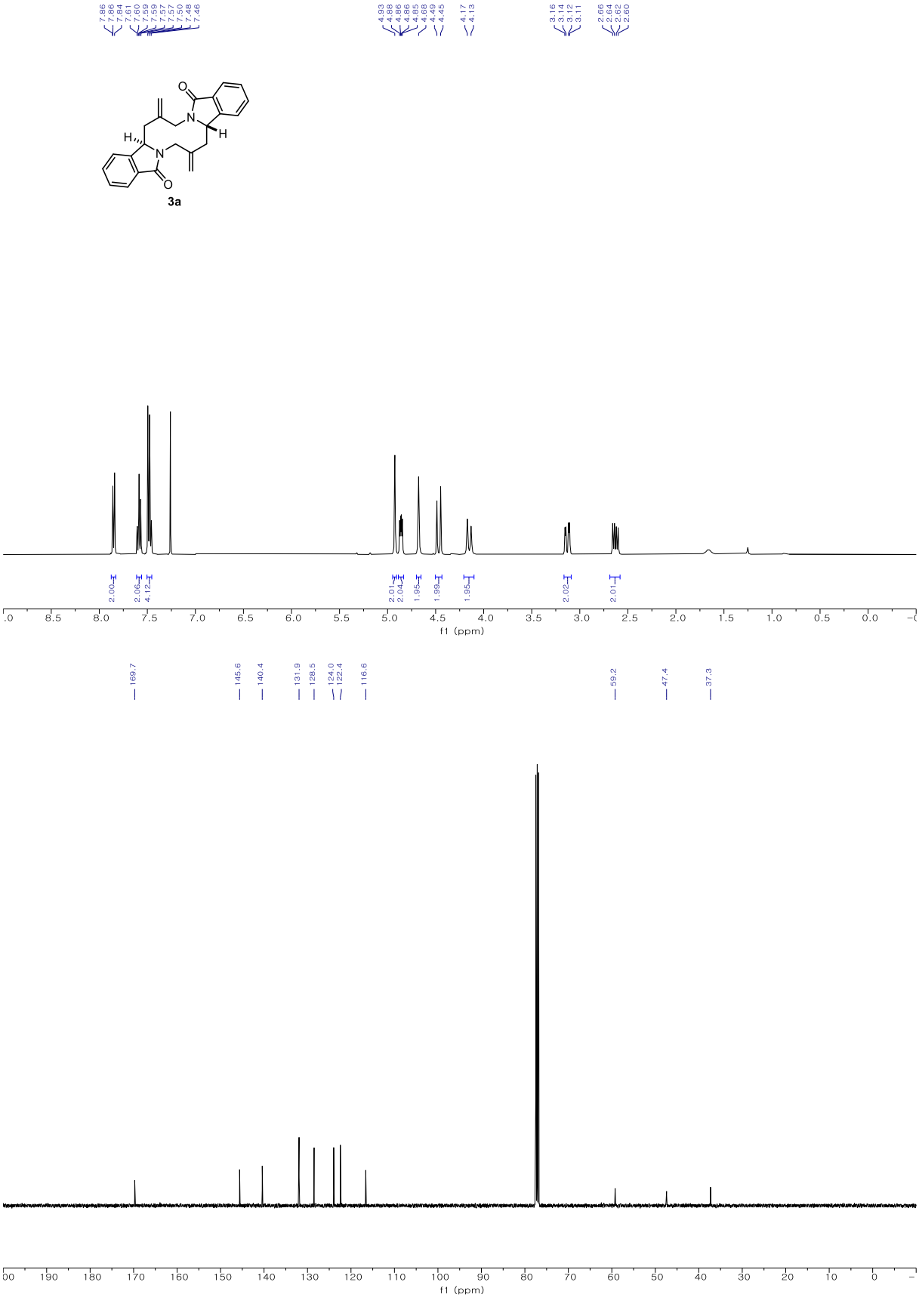
169.9
142.4
142.2
129.9
109.2
82.9
46.4
24.0
20.9
1.4
f1 (ppm)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7.59
7.59
7.57
7.57
7.50
7.48
7.46
4.93
4.88
4.86
4.86
4.85
4.68
4.49
4.45
4.17
4.13
3.16
3.14
3.12
3.11
2.66
2.64
2.62
2.60
O
N
H
H
N
O
3a
2.00
2.06
4.12
2.01
2.04
1.95
1.99
1.95
2.02
2.01
f1 (ppm)
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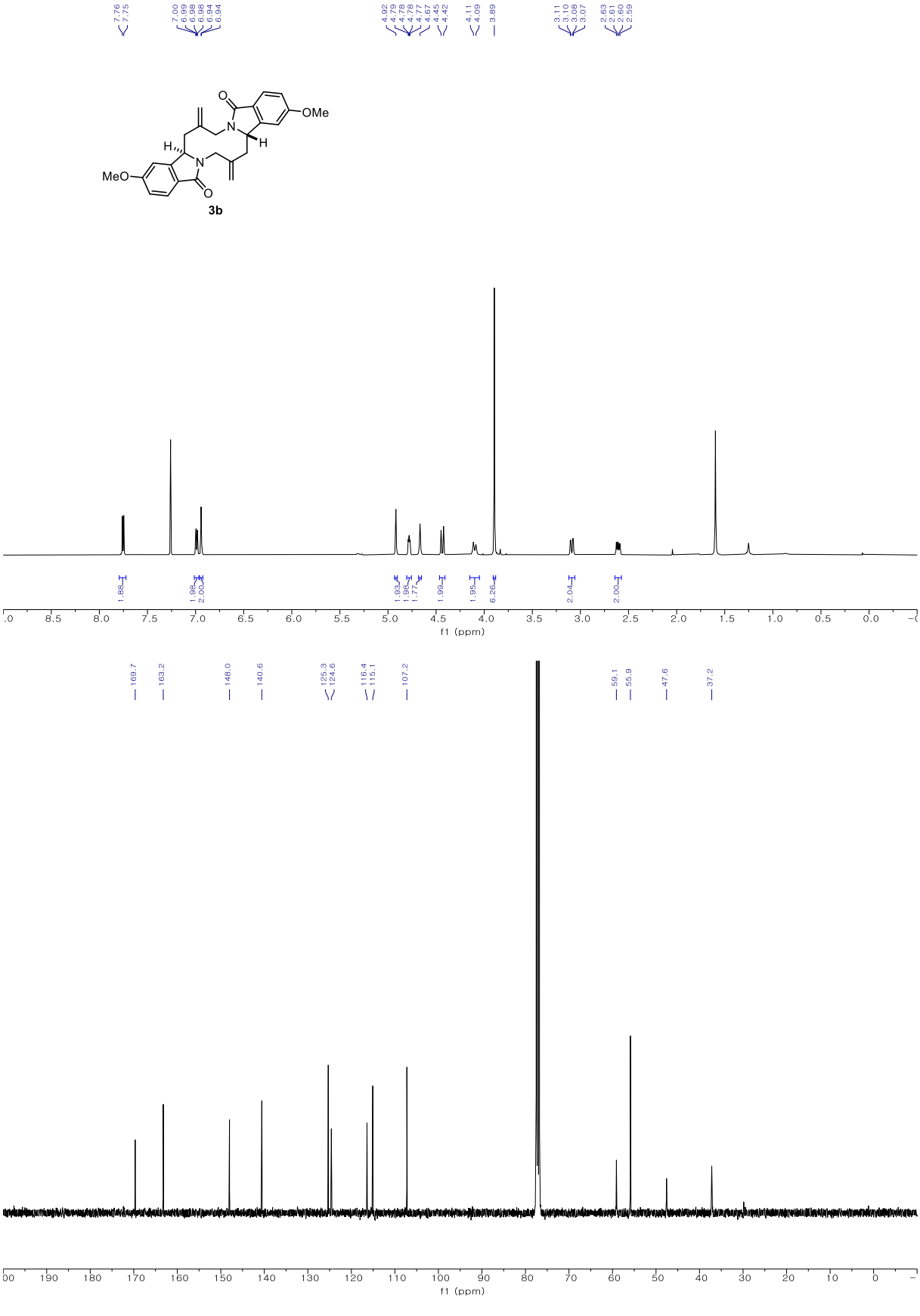
145.6
140.4
131.9
128.5
124.0
122.4
116.6
59.2
47.4
37.3
f1 (ppm)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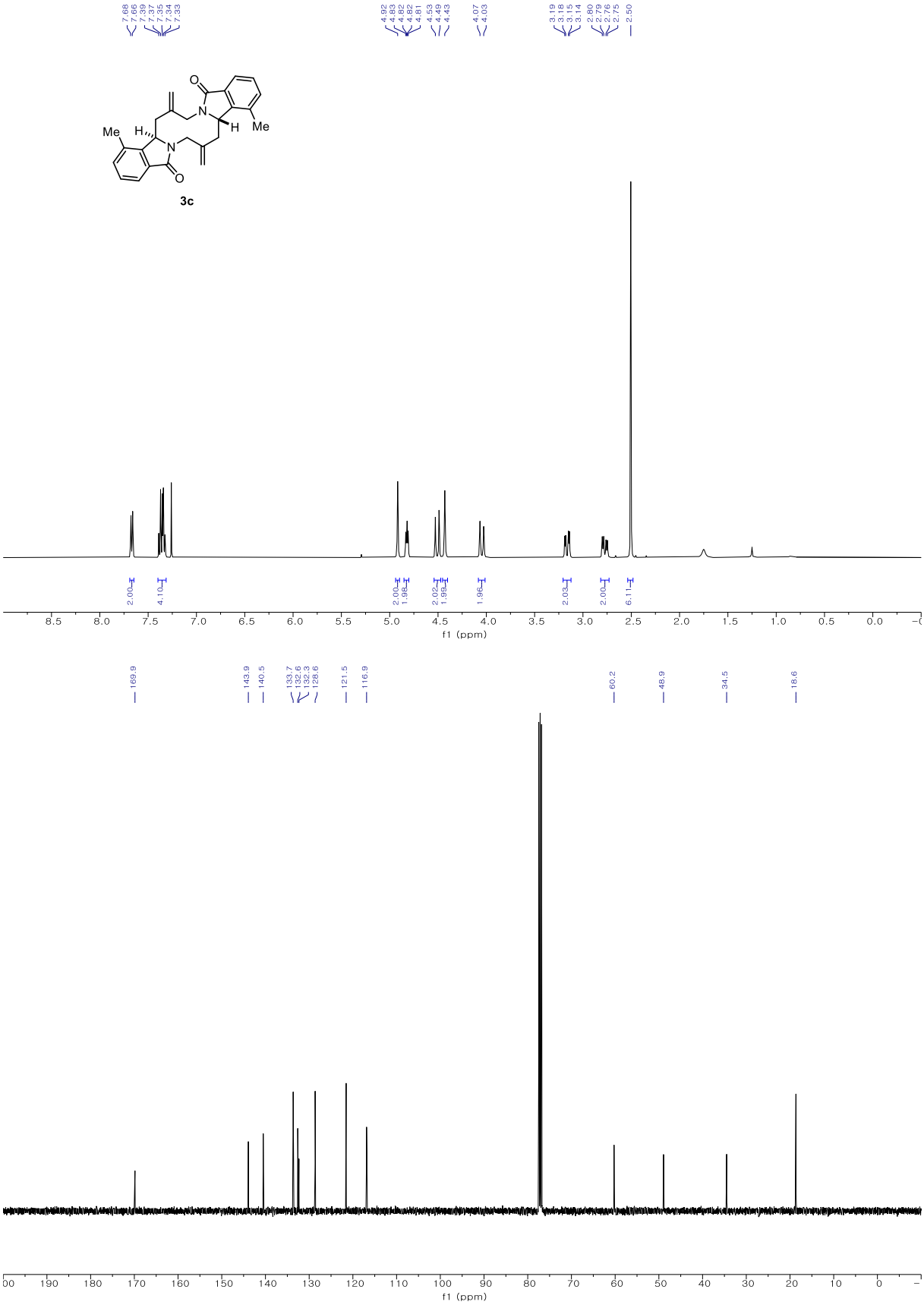
6.98
6.94
6.94
4.92
4.79
4.78
4.78
4.67
4.45
4.42
4.11
4.09
3.89
3.11
3.10
3.08
3.07
2.63
2.61
2.60
2.59
OMe
MeO
H
H
N
N
O
O
3b
1.88
1.98
2.00
1.93
1.98
1.77
1.99
1.95
6.26
2.04
2.00
f1 (ppm)
169.7
163.2
148.0
140.6
125.3
124.6
116.4
115.1
107.2
59.1
55.9
47.6
37.2
f1 (ppm)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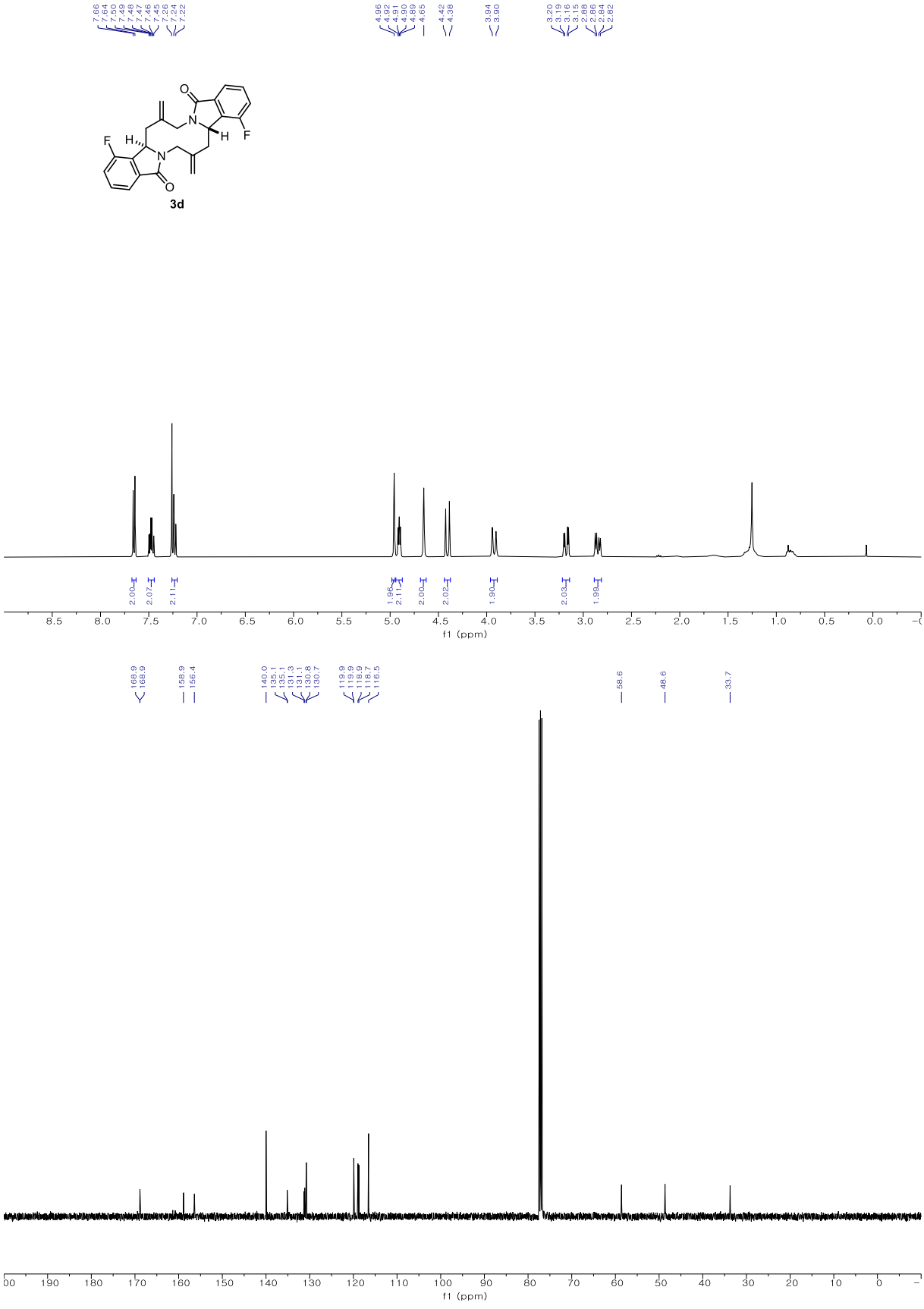
7.34
7.33
4.92
4.83
4.82
4.82
4.81
4.53
4.49
4.43
4.07
4.03
3.19
3.18
3.15
3.14
2.80
2.79
2.76
2.75
2.50
O
N
H
Me
Me
H
N
O
2.00
4.10
2.00
1.98
2.02
1.98
1.96
2.03
2.00
6.11
f1 (ppm)
169.9
143.9
140.5
133.7
132.6
132.3
128.6
121.5
116.9
60.2
48.9
34.5
18.6
f1 (ppm)

3c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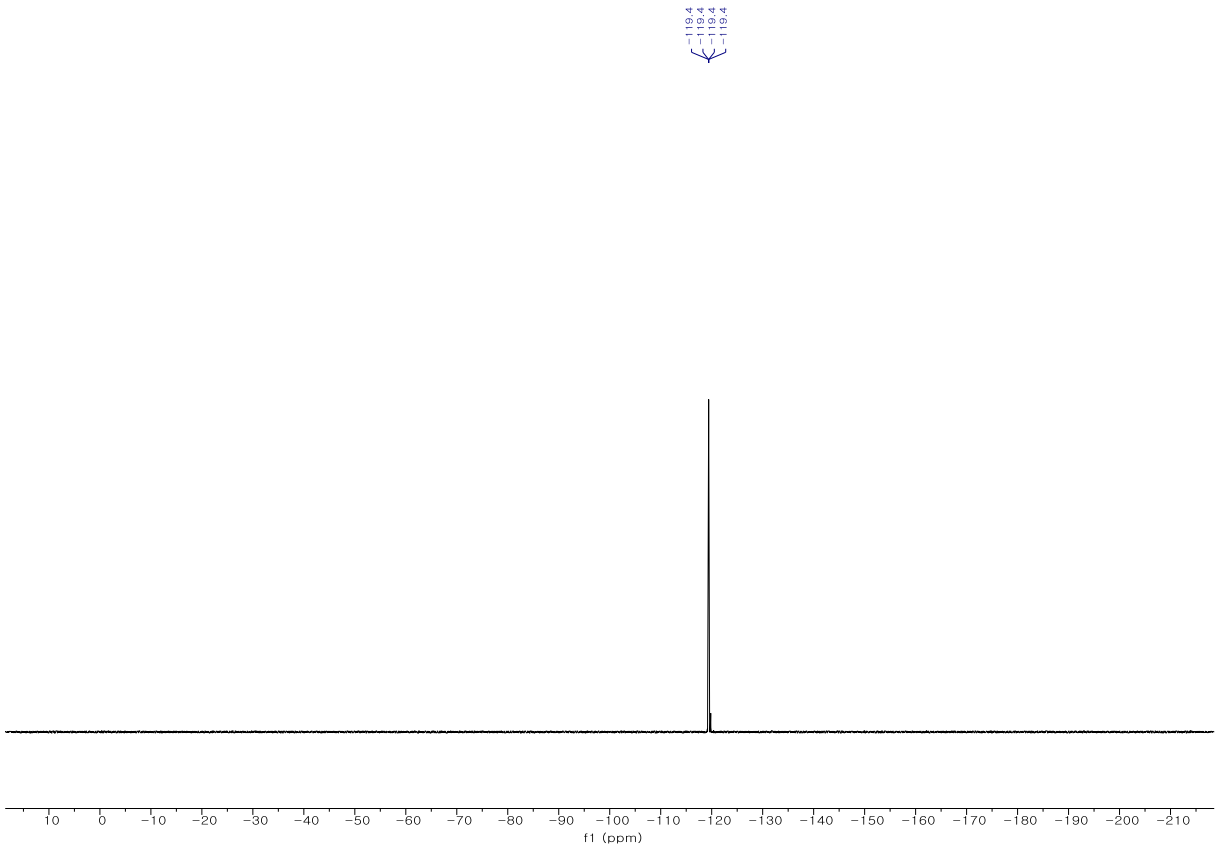
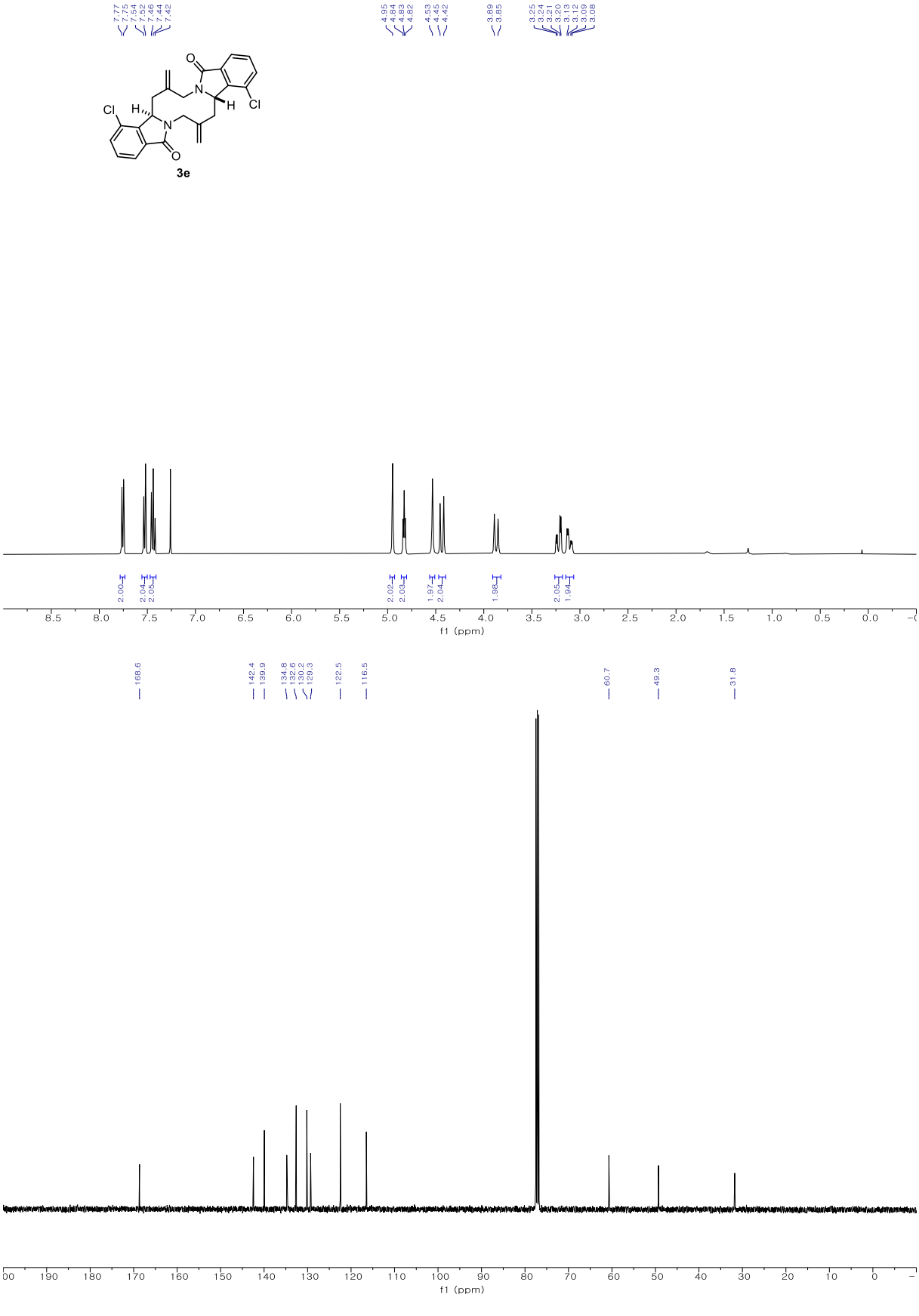
7.64
7.50
7.49
7.48
7.47
7.46
7.45
7.26
7.24
7.22
4.96
4.92
4.91
4.90
4.89
4.65
4.42
4.38
3.94
3.90
3.20
3.19
3.16
3.15
2.88
2.86
2.84
2.82
O
N
H
F
H
F
N
O
3d
2.00
2.07
2.11
1.96
2.11
2.00
2.02
1.90
2.03
1.99
f1 (ppm)
168.9
168.9
158.9
156.4
140.0
135.1
135.1
131.3
131.1
130.8
130.7
119.9
119.9
118.9
118.7
116.5
58.6
48.6
33.7
f1 (ppm)


-119.4
-119.4
-119.4
-119.4
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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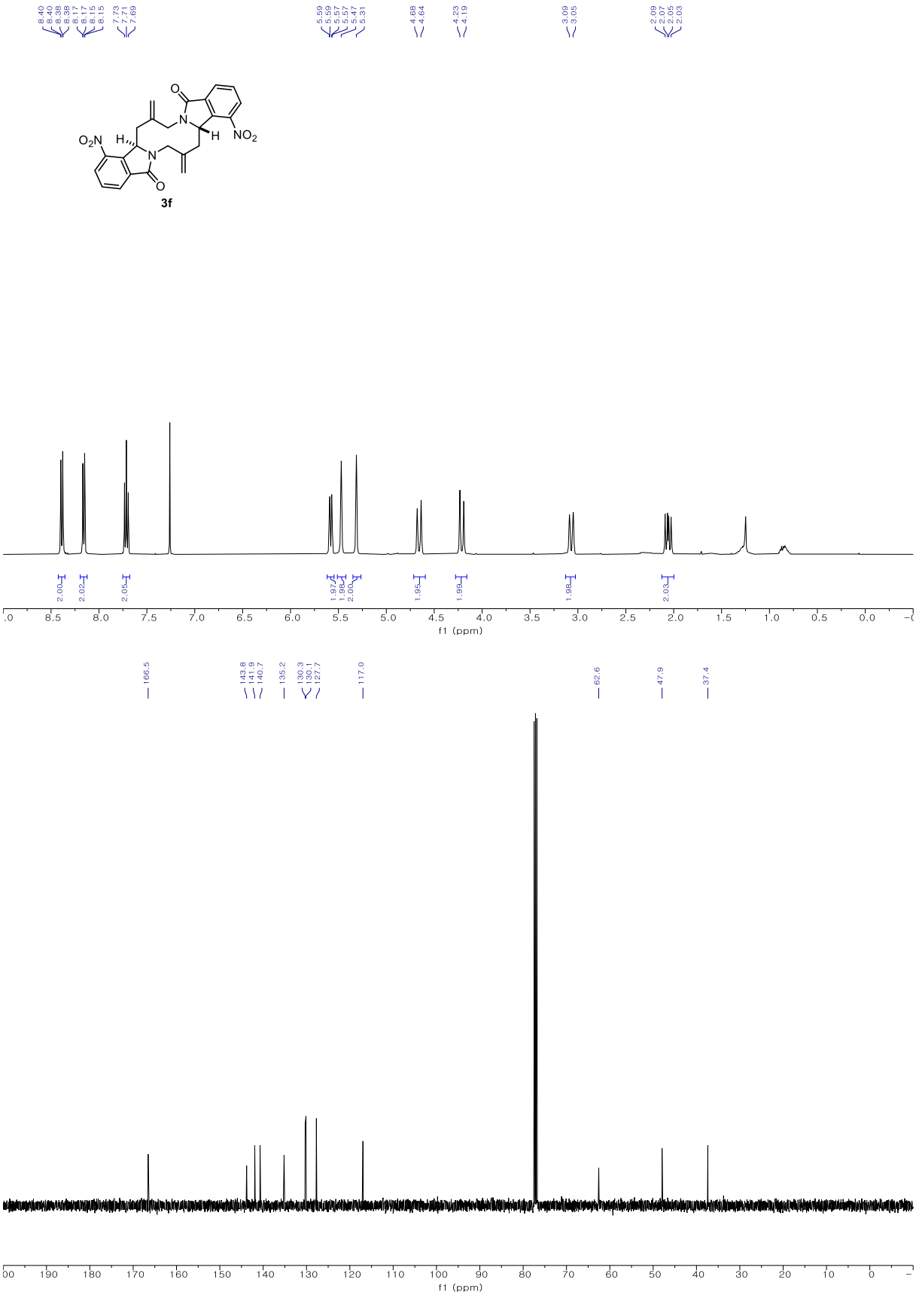
7.46
7.44
7.42
4.95
4.84
4.83
4.82
4.53
4.45
4.42
3.89
3.85
3.25
3.24
3.21
3.20
3.13
3.12
3.09
3.08
Cl
H
N
O
O
N
H
Cl
3e
2.00
2.04
2.05
2.02
2.03
1.97
2.04
1.98
2.05
1.94
f1 (ppm)
168.6
142.4
139.9
134.8
132.6
132.2
129.3
122.5
116.5
60.7
49.3
31.8
f1 (ppm)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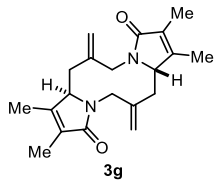
8.40
8.38
8.38
8.17
8.17
8.15
8.15
7.73
7.71
7.69
5.59
5.59
5.57
5.57
5.47
5.31
4.68
4.64
4.23
4.19
3.09
3.05
2.09
2.07
2.05
2.03
O
N
H
H
NO2
O2N
N
O
O
3f
2.00
2.02
2.05
1.97
1.96
2.00
1.95
1.99
1.98
2.03
f1 (ppm)
166.5
143.8
141.9
140.7
135.2
130.3
130.1
127.7
117.0
62.6
47.9
37.4
f1 (ppm)


O
Me
N
Me
H
H
Me
N
Me
O

3g

4.95
4.83
4.23
4.20
3.98
3.81
3.77
2.84
2.83
2.80
2.79
2.38
2.36
2.34
2.32
1.94
1.79
2.00
1.98
1.97
2.01
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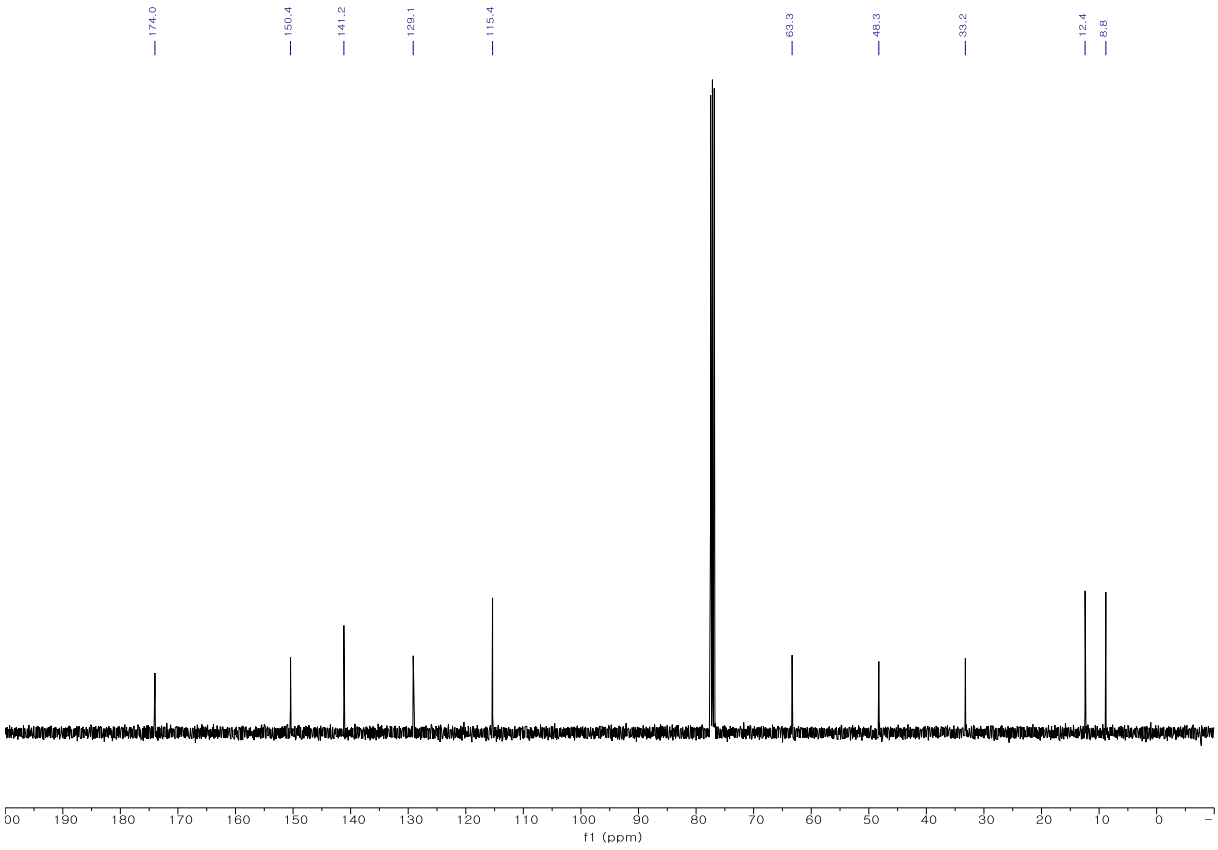
1.97
1.96
5.93
6.01
f1 (ppm)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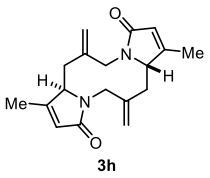
150.4
141.2
129.1
115.4
63.3
48.3
33.2
12.4
8.8
f1 (ppm)


O
N
Me
H
H
Me
N
O

3h

5.88
5.01
4.93
4.24
4.20
4.13
4.12
4.11
3.86
3.83
2.86
2.85
2.82
2.81
2.41
2.40
2.38
2.36
2.06
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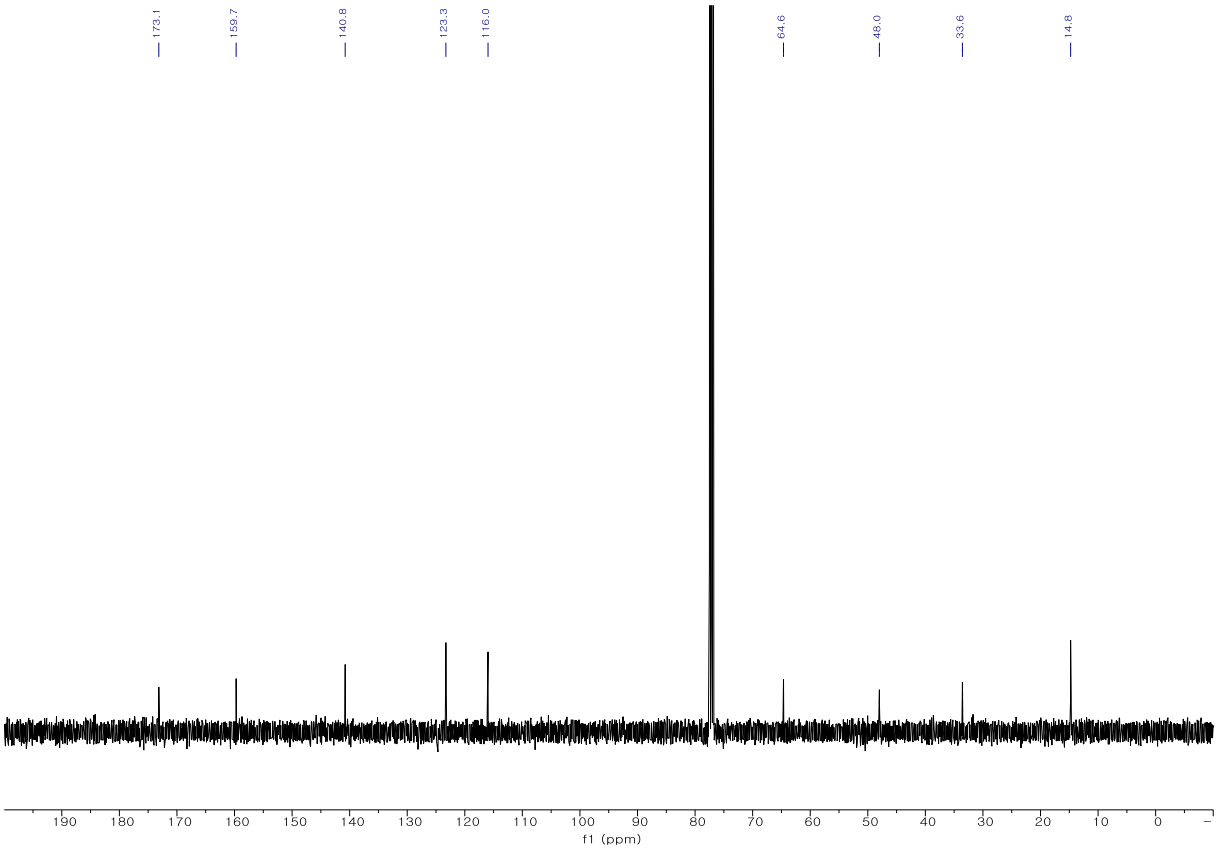
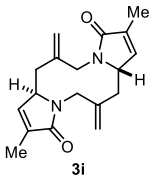
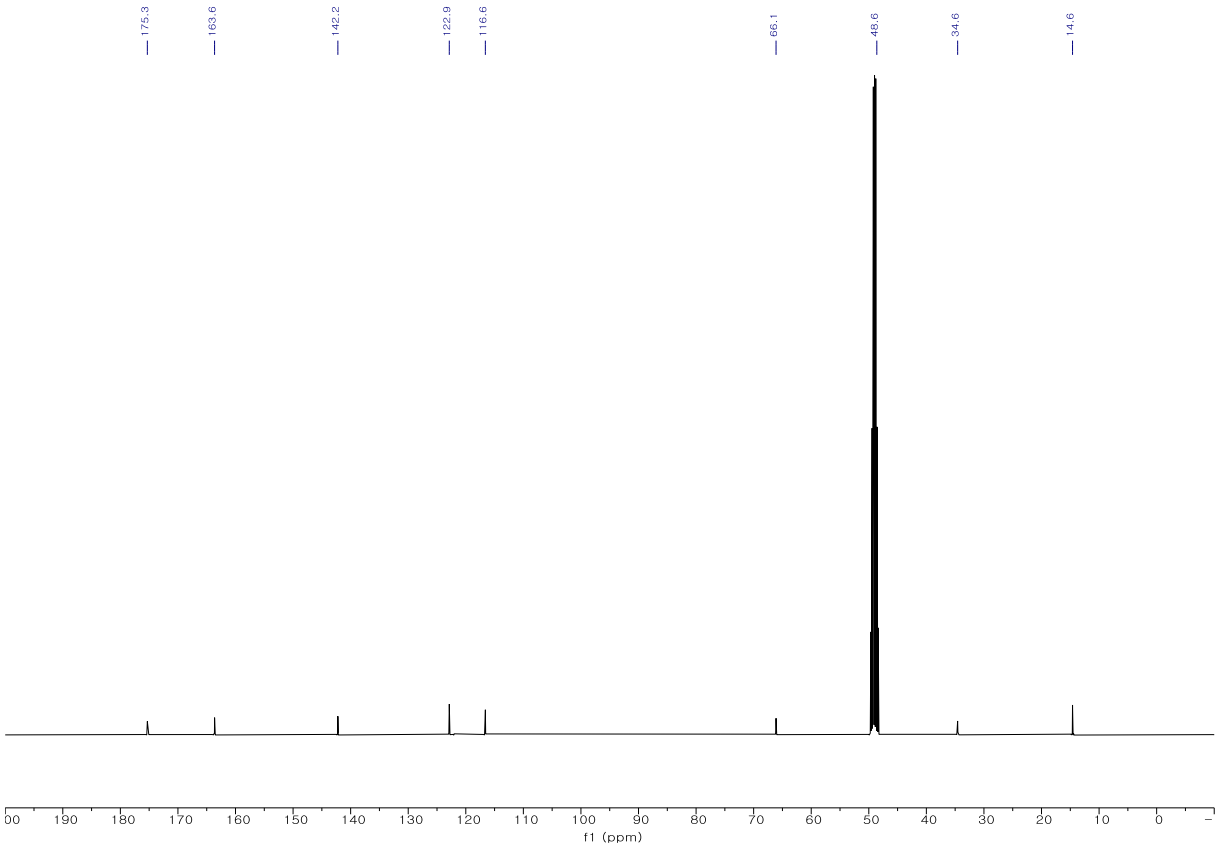
1.84
1.94
2.05
6.01
f1 (ppm)

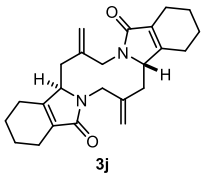

173.1
159.7
140.8
123.3
116.0
64.6
48.0
33.6
14.8
f1 (ppm)


O
Me
N
H
H
N
Me
O

3i

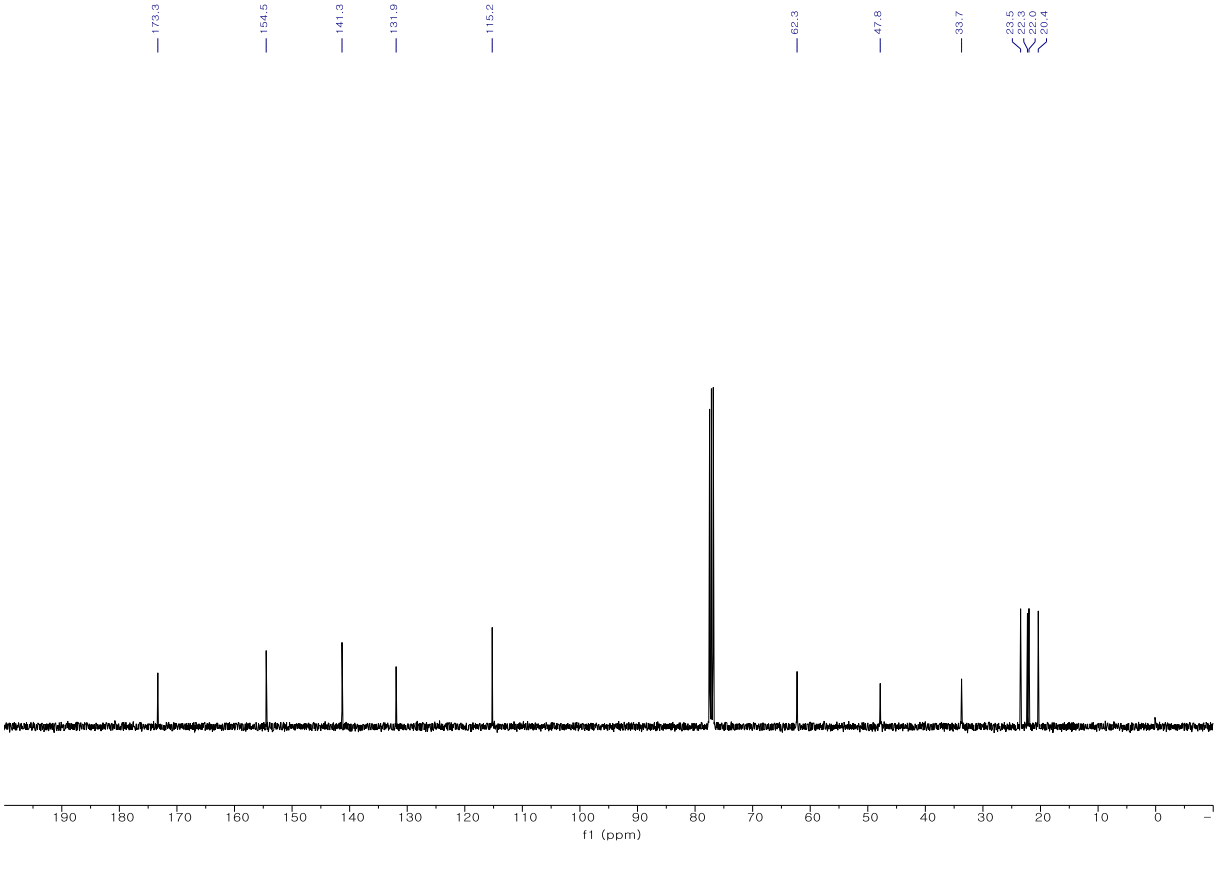
5.78
4.94
4.92
4.29
4.27
4.26
4.14
4.10
3.88
3.84
2.82
2.81
2.78
2.77
2.38
2.37
2.35
2.33
2.02
2.02
2.00
3.95
2.01
1.99
1.94
1.99
2.01
6.03
f1 (ppm)

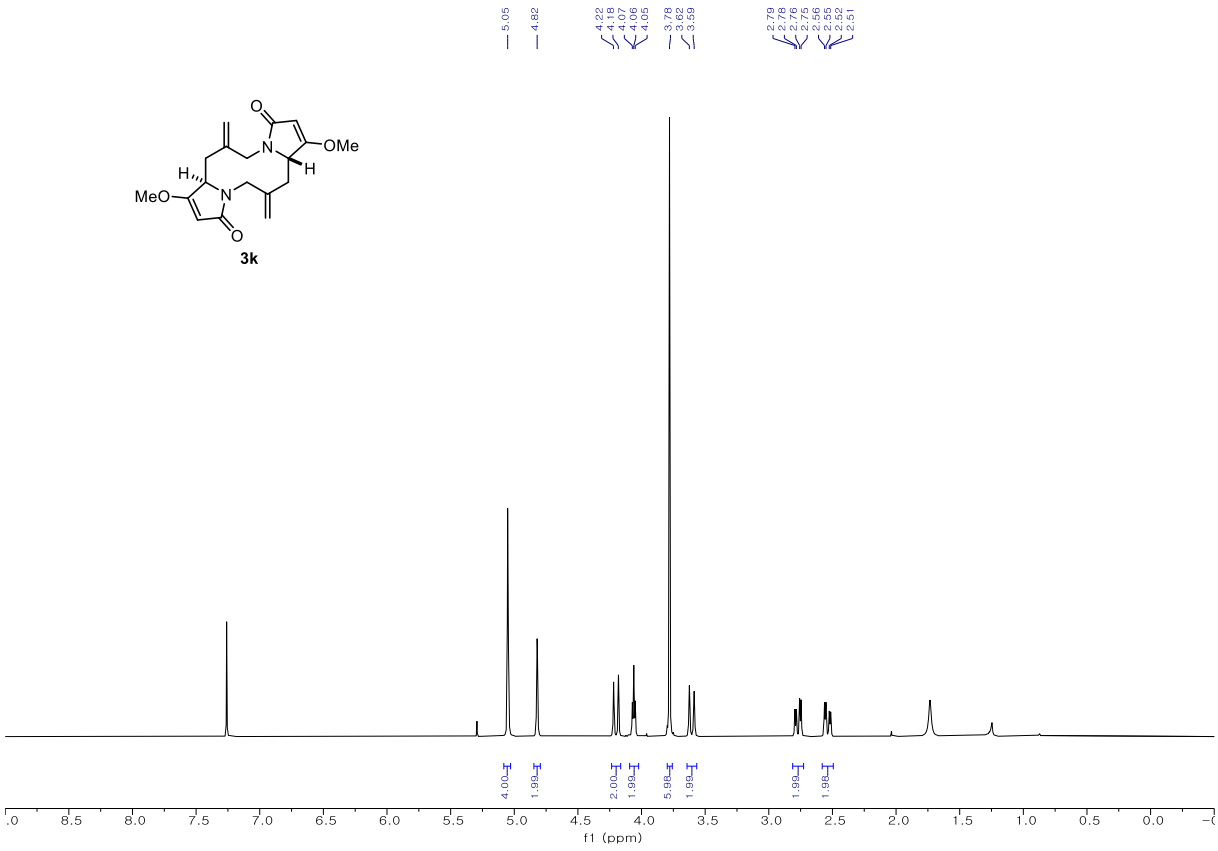

175.3
163.6
142.2
122.9
116.6
66.1
48.6
34.6
14.6
f1 (ppm)

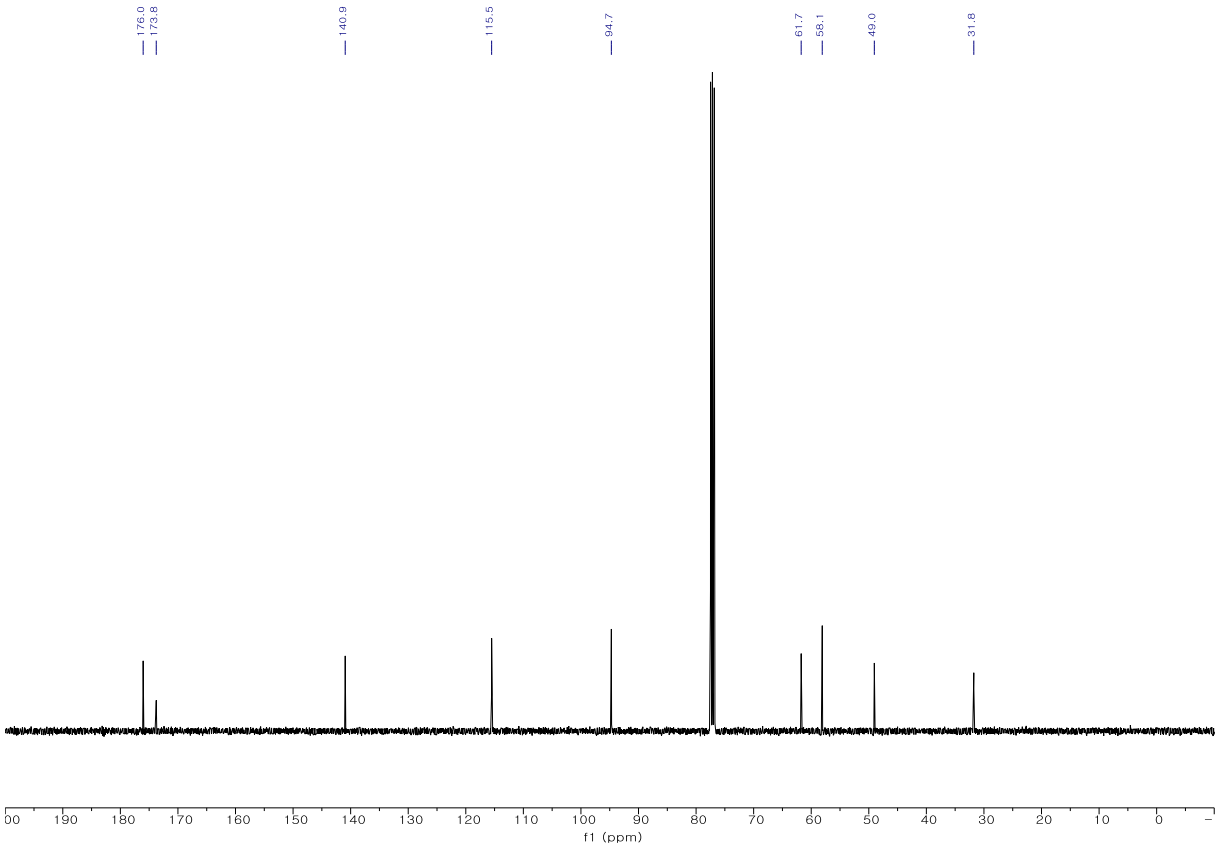

O
N
H
H
N
O

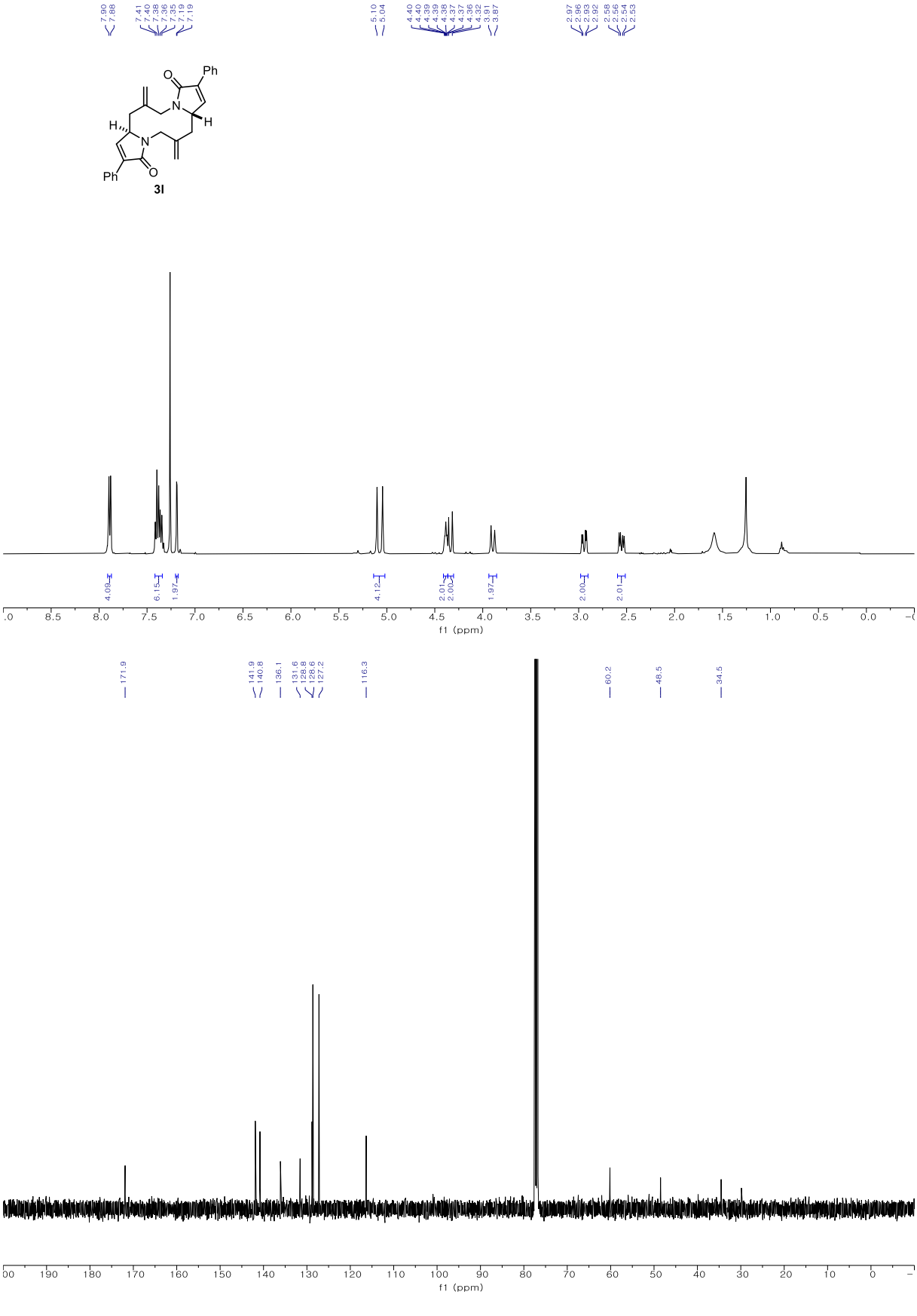
3j

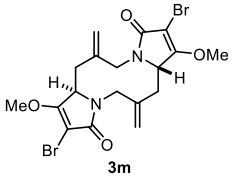
4.96
4.86
4.23
4.20
4.05
3.86
3.82
2.81
2.80
2.77
2.76
2.35
2.33
2.31
2.29
2.28
2.27
2.23
2.22
2.21
2.20
2.19
1.75
1.74
1.73
1.72
1.71
1.69
1.68
1.67
1.98
2.00
2.01
2.03
1.95
1.98
10.11
8.10
f1 (ppm)


173.3
154.5
141.3
131.9
115.2
62.3
47.8
33.7
23.5
22.3
22.0
20.4
f1 (ppm)


5.05
4.82
4.22
4.18
4.07
4.06
4.05
3.78
3.62
3.59
2.79
2.78
2.76
2.75
2.56
2.55
2.52
2.51
OMe
MeO
H
H
N
N
O
O
3k
4.00
1.99
2.00
1.99
5.98
1.99
1.99
1.98
f1 (ppm)

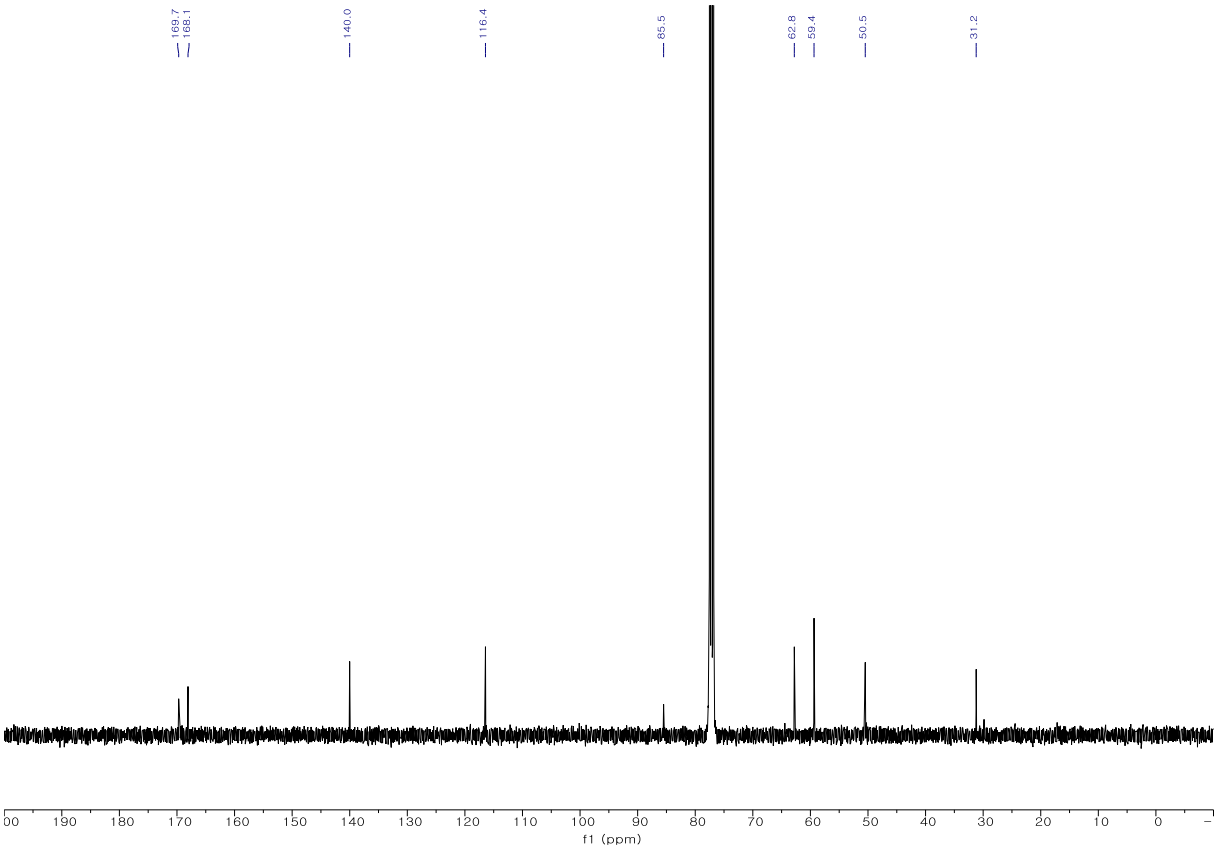

176.0
173.8
140.9
115.5
94.7
61.7
58.1
49.0
31.8
f1 (ppm)

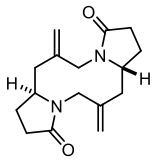

7.90
7.88
7.41
7.40
7.38
7.36
7.35
7.19
7.19
5.10
5.04
4.40
4.40
4.39
4.39
4.38
4.37
4.37
4.36
4.32
3.91
3.87
2.97
2.96
2.93
2.93
2.58
2.56
2.54
2.53
Ph
O
N
H
H
N
O
Ph
3l
4.09
6.15
1.97
4.12
2.01
2.00
1.97
2.00
2.01
f1 (ppm)
171.9
141.9
140.8
136.1
131.6
128.8
128.6
127.2
116.3
60.2
48.5
34.5
f1 (ppm)


O
Br
OMe
H
H
MeO
N
N
O
Br

3m

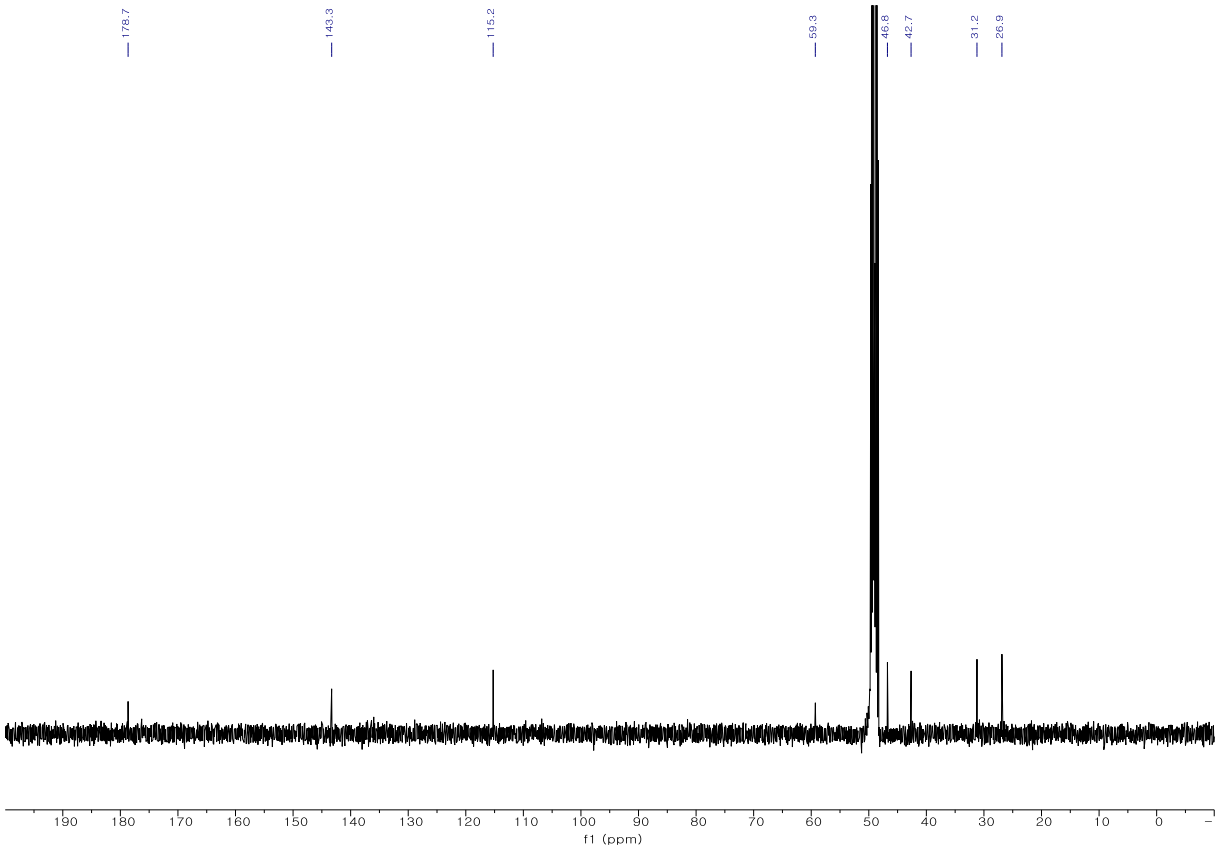
5.13
4.88
4.26
4.23
4.01
4.00
3.99
3.59
3.55
2.77
2.75
2.73
2.72
2.61
2.60
2.57
2.56
1.00
1.01
4.14
1.00
0.97
1.03
1.01
f1 (ppm)

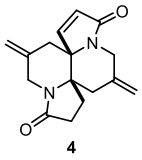

169.7
168.1
140.0
116.4
85.5
62.8
59.4
50.5
31.2
f1 (ppm)



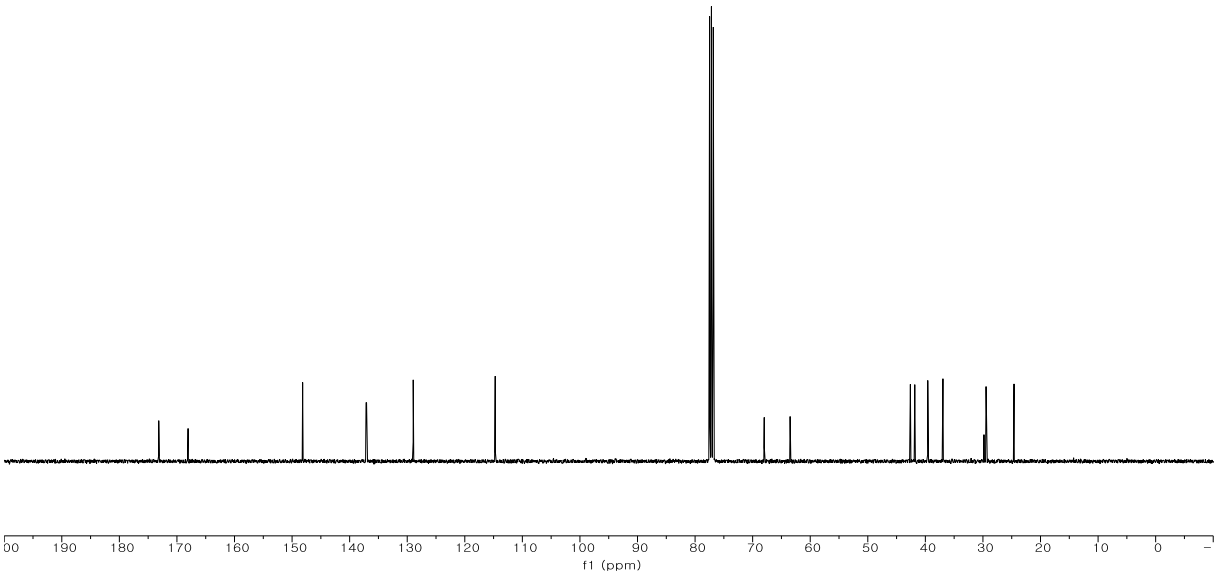
3o

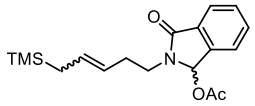
5.27
4.97
4.19
4.17
4.14
4.12
4.11
4.06
4.04
4.00
2.56
2.52
2.48
2.48
2.47
2.45
2.45
2.43
2.43
2.35
2.33
2.32
2.31
2.30
2.28
2.27
2.24
2.23
2.20
1.71
1.69
1.68
1.66
2.04
2.06
2.21
4.09
2.35
4.03
4.22
2.28
f1 (ppm)


178.7
143.3
115.2
59.3
46.8
42.7
31.2
26.9
f1 (ppm)



4

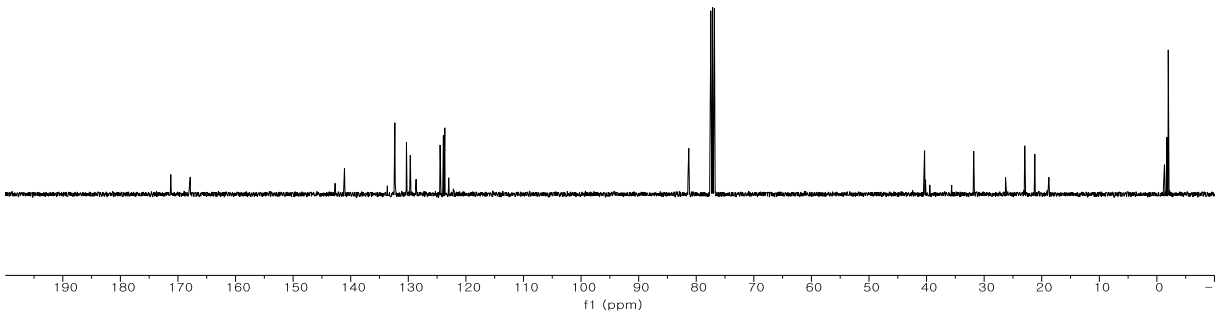


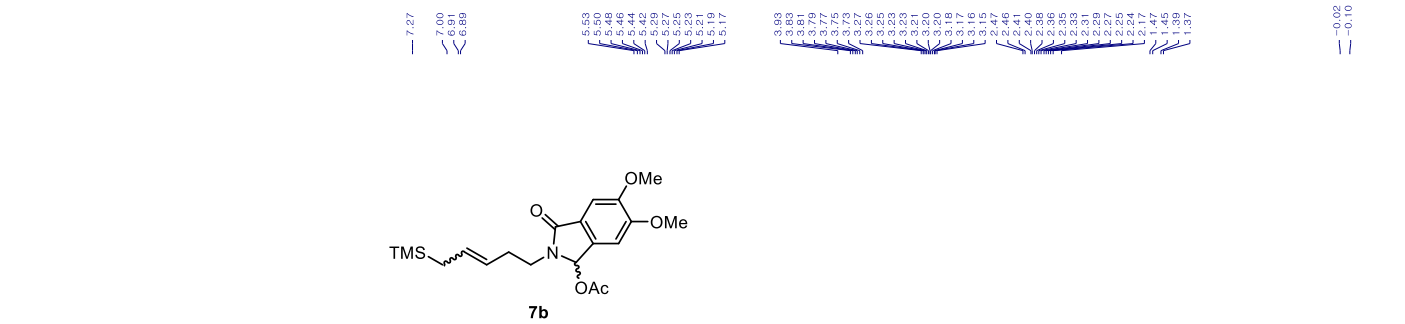

TMS
O
N
OAc

7a

1.00
3.09
1.01
0.78
0.83
1.02
0.94
1.90
3.09
1.82
9.15
f1 (ppm)

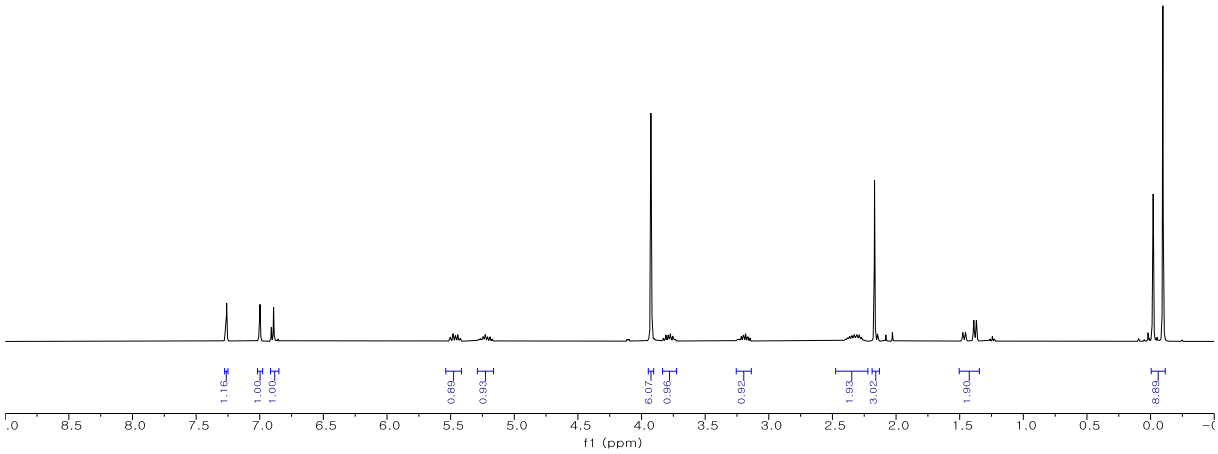

171.3
167.9
142.7
141.1
132.3
130.3
129.7
124.5
123.9
123.7
81.3
40.3
31.8
22.9
21.2
-2.0

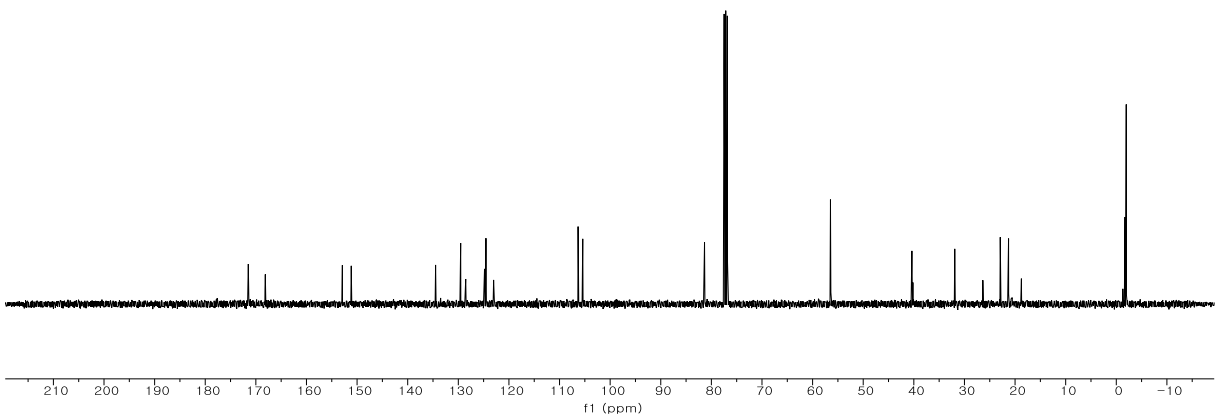

f1 (ppm)

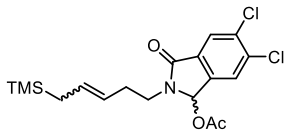


OMe
OMe
O
N
TMS
OAc

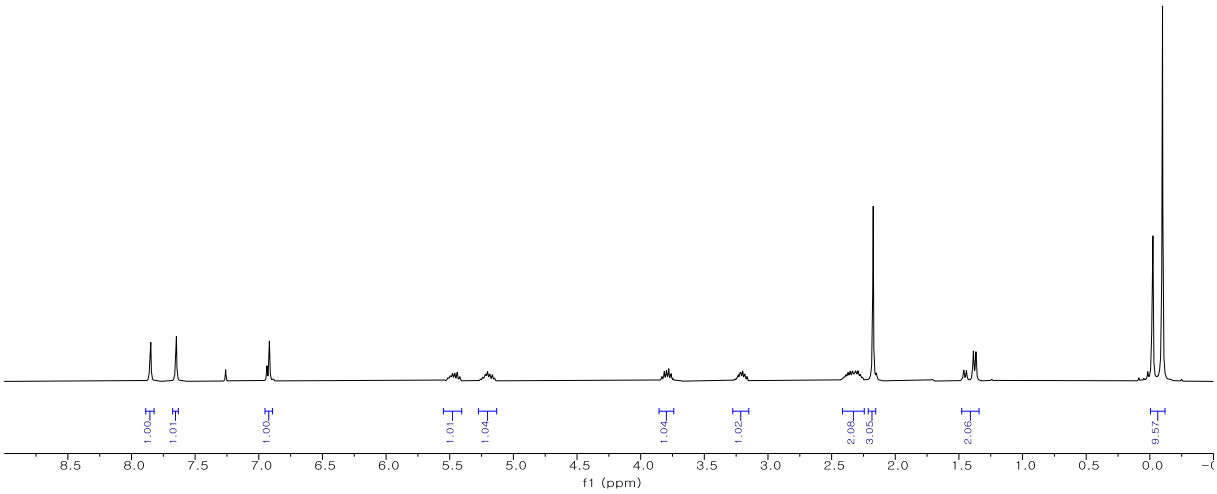
7b

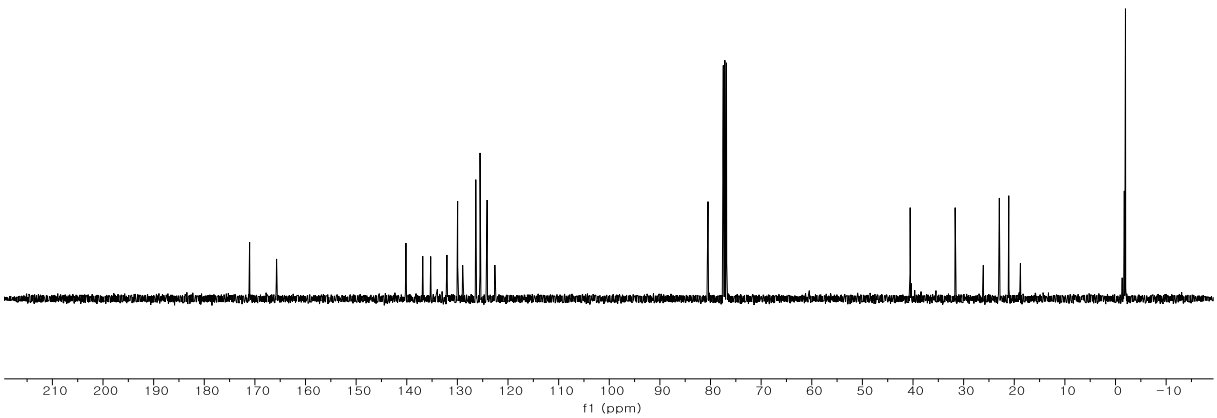

1.16
1.00
1.00
0.89
0.93
6.07
0.96
0.92
1.93
3.02
1.90
8.89
f1 (ppm)

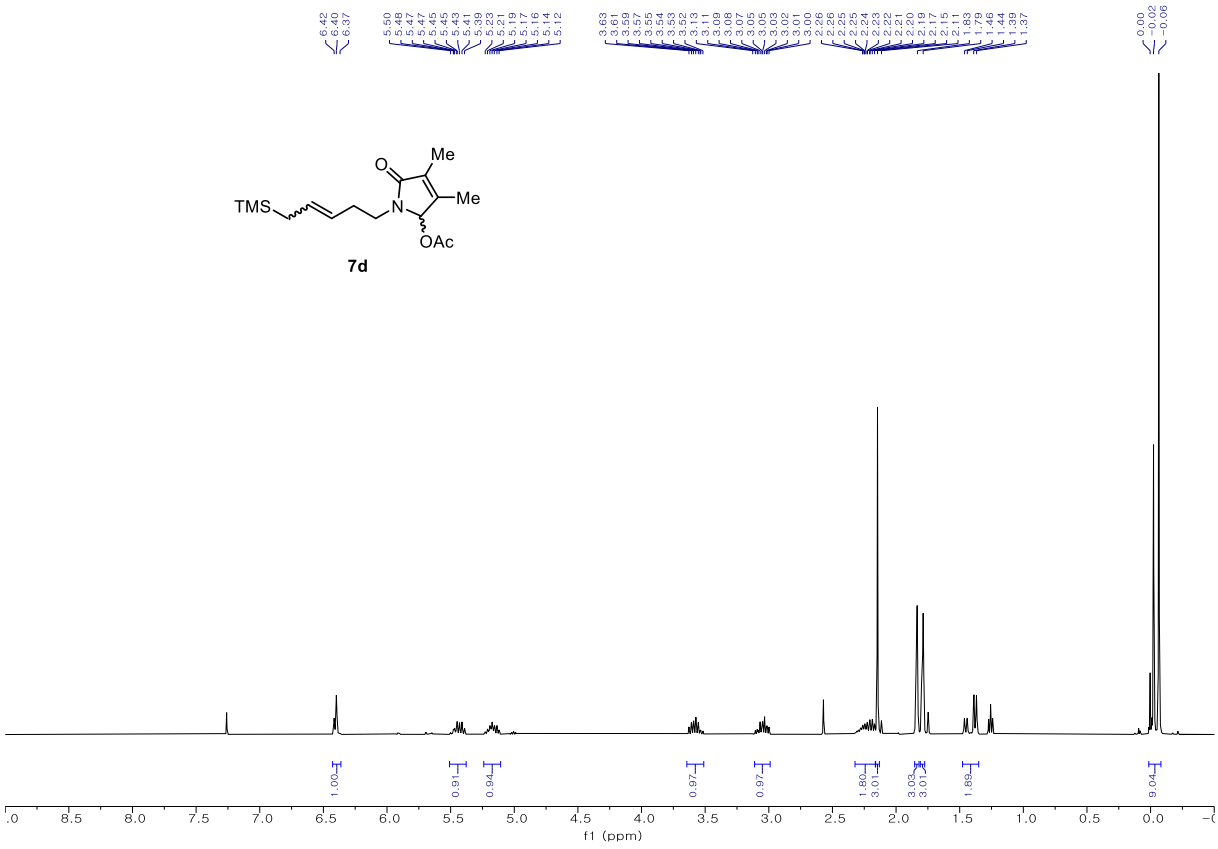

f1 (ppm)

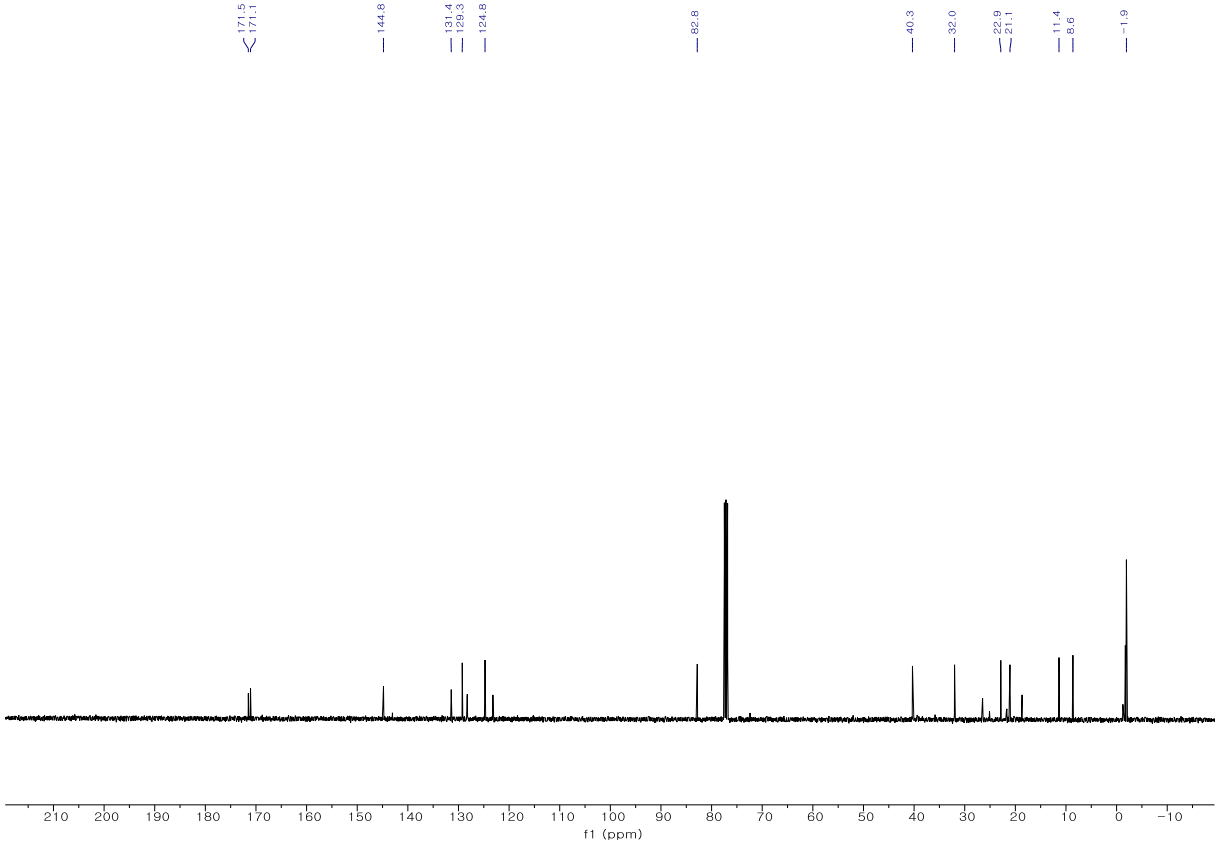


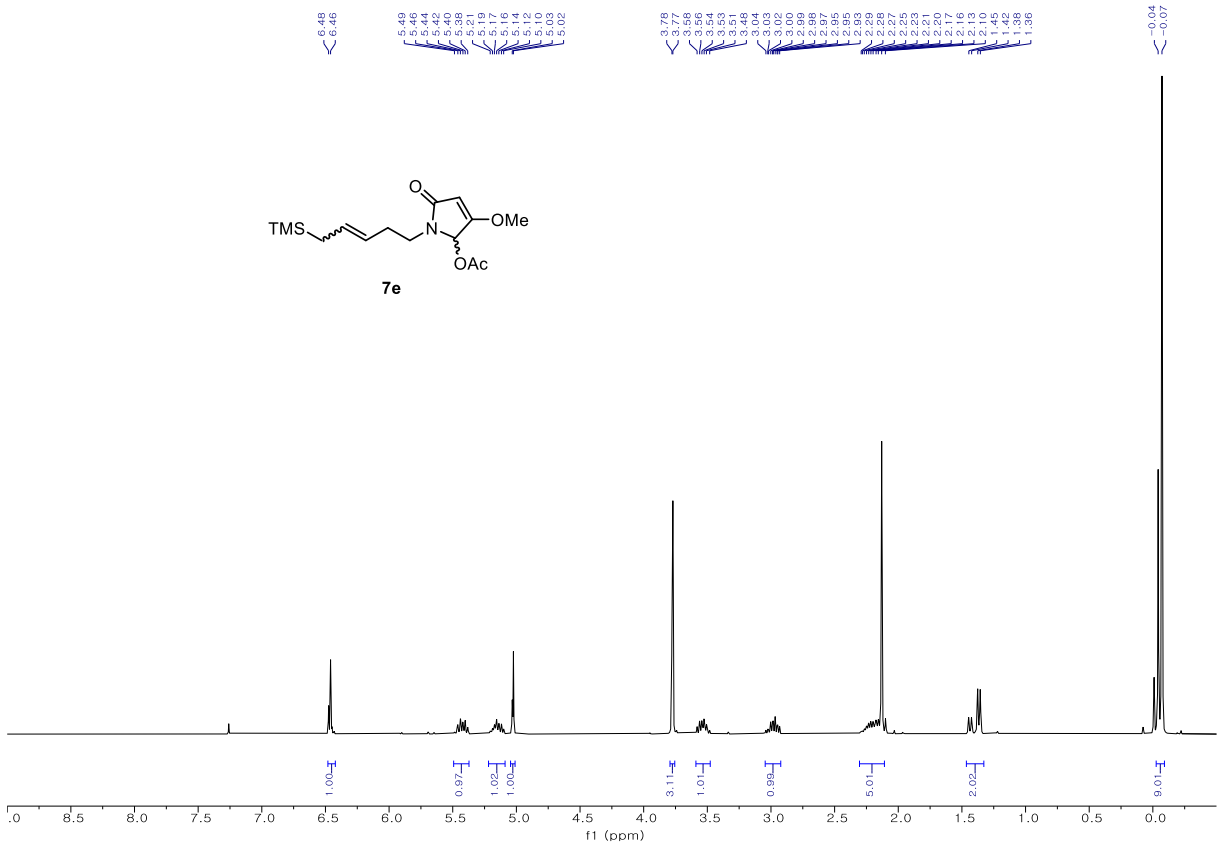
7c

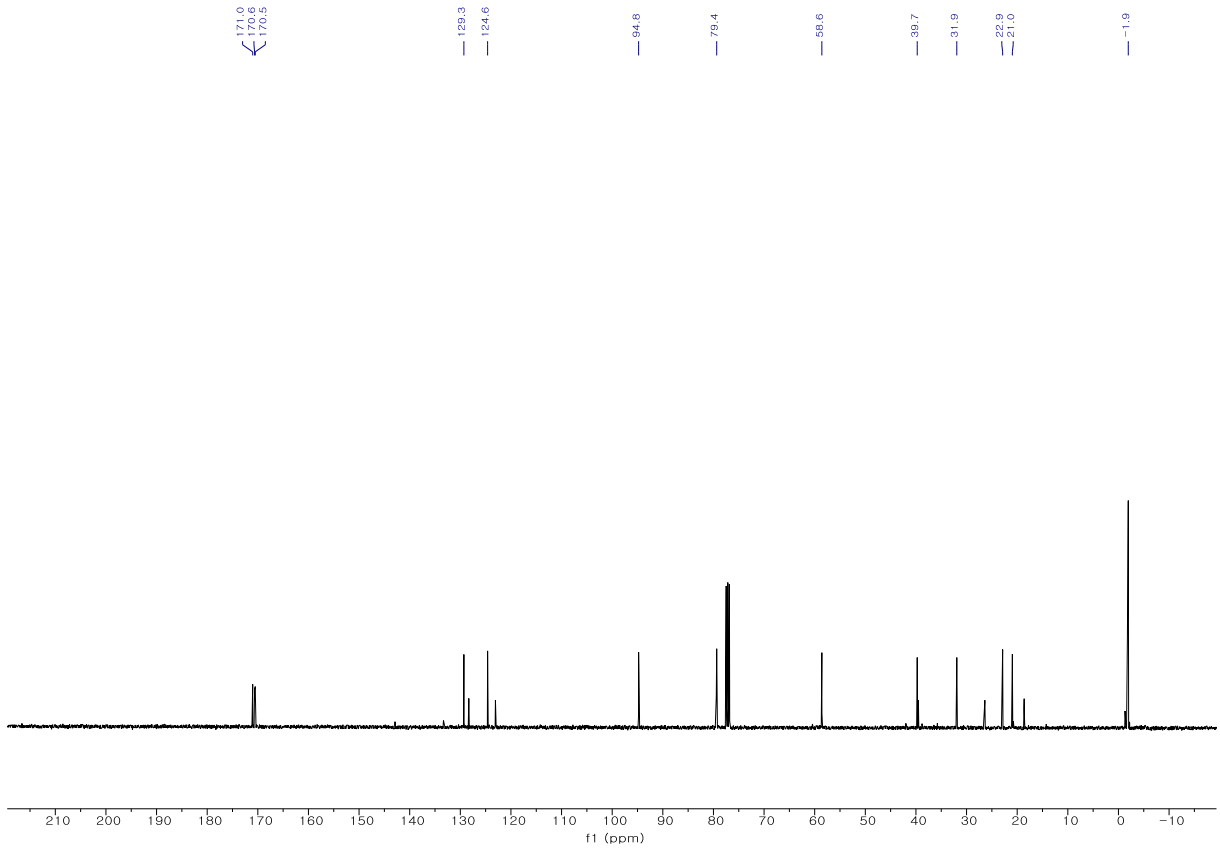


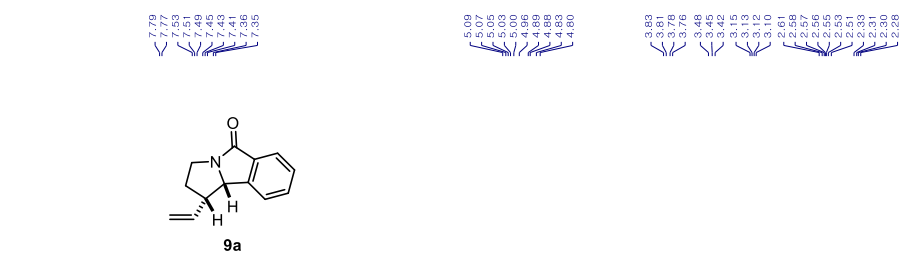



6.42
6.40
6.37
5.50
5.49
5.48
5.47
5.47
5.45
5.45
5.43
5.41
5.39
5.23
5.21
5.19
5.17
5.16
5.14
5.12
3.63
3.61
3.59
3.57
3.55
3.54
3.53
3.52
3.33
3.11
3.09
3.08
3.07
3.05
3.05
3.03
3.02
3.01
3.00
2.99
2.26
2.25
2.25
2.24
2.23
2.22
2.21
2.20
2.19
2.17
2.15
2.11
1.83
1.79
1.46
1.44
1.39
1.37
0.00
-0.02
-0.06
Me
O
Me
TMS
N
OAc
7d
1.00
0.91
0.94
0.97
0.97
1.80
3.01
3.03
3.01
1.88
9.04
f1 (ppm)

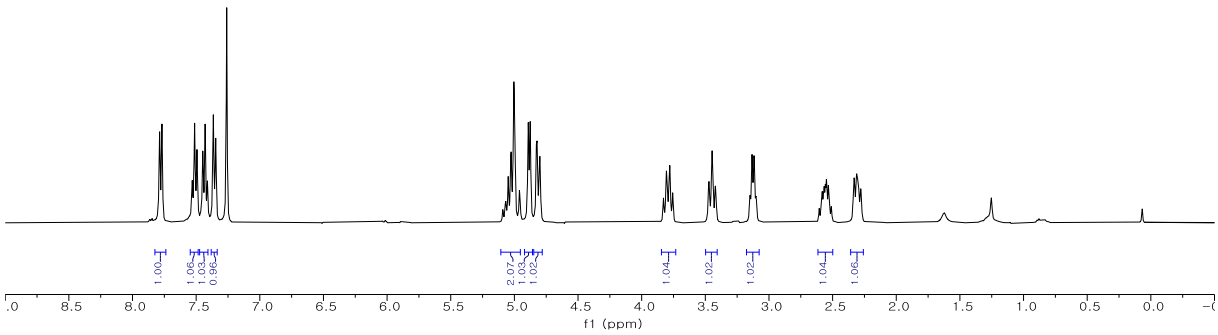

171.5
171.1
144.8
131.4
129.3
124.8
82.8
40.3
32.0
22.9
21.1
11.4
8.6
-1.9
f1 (ppm)

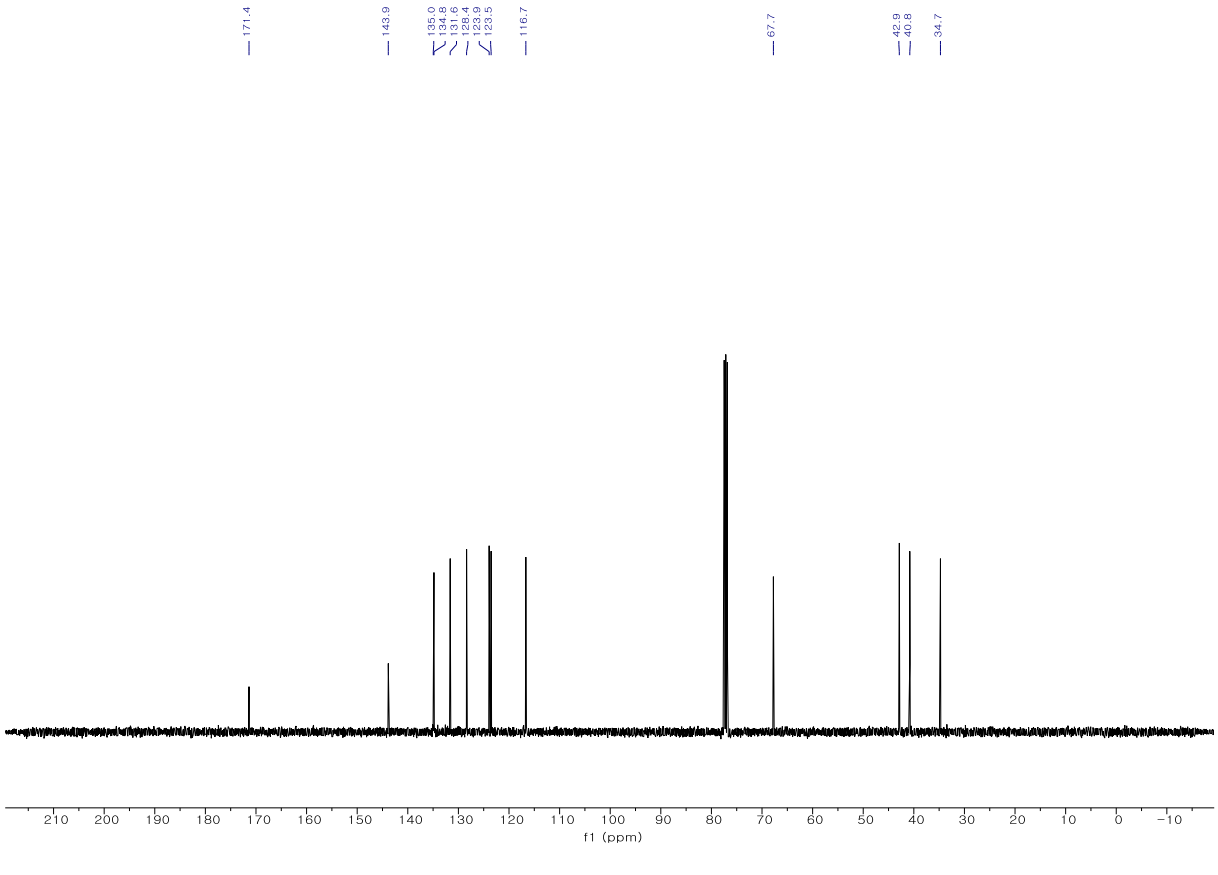

6.48
6.46
5.49
5.46
5.44
5.42
5.40
5.38
5.21
5.19
5.17
5.16
5.14
5.12
5.10
5.03
5.02
3.78
3.77
3.58
3.56
3.54
3.53
3.51
3.48
3.04
3.03
3.02
3.00
2.99
2.98
2.97
2.95
2.93
2.29
2.28
2.27
2.25
2.23
2.21
2.20
2.17
2.16
2.13
2.10
1.45
1.42
1.38
1.36
-0.04
-0.07
O
N
TMS
OMe
OAc
7e
1.00
0.97
1.02
1.00
3.11
1.01
0.99
5.01
2.02
9.01
f1 (ppm)

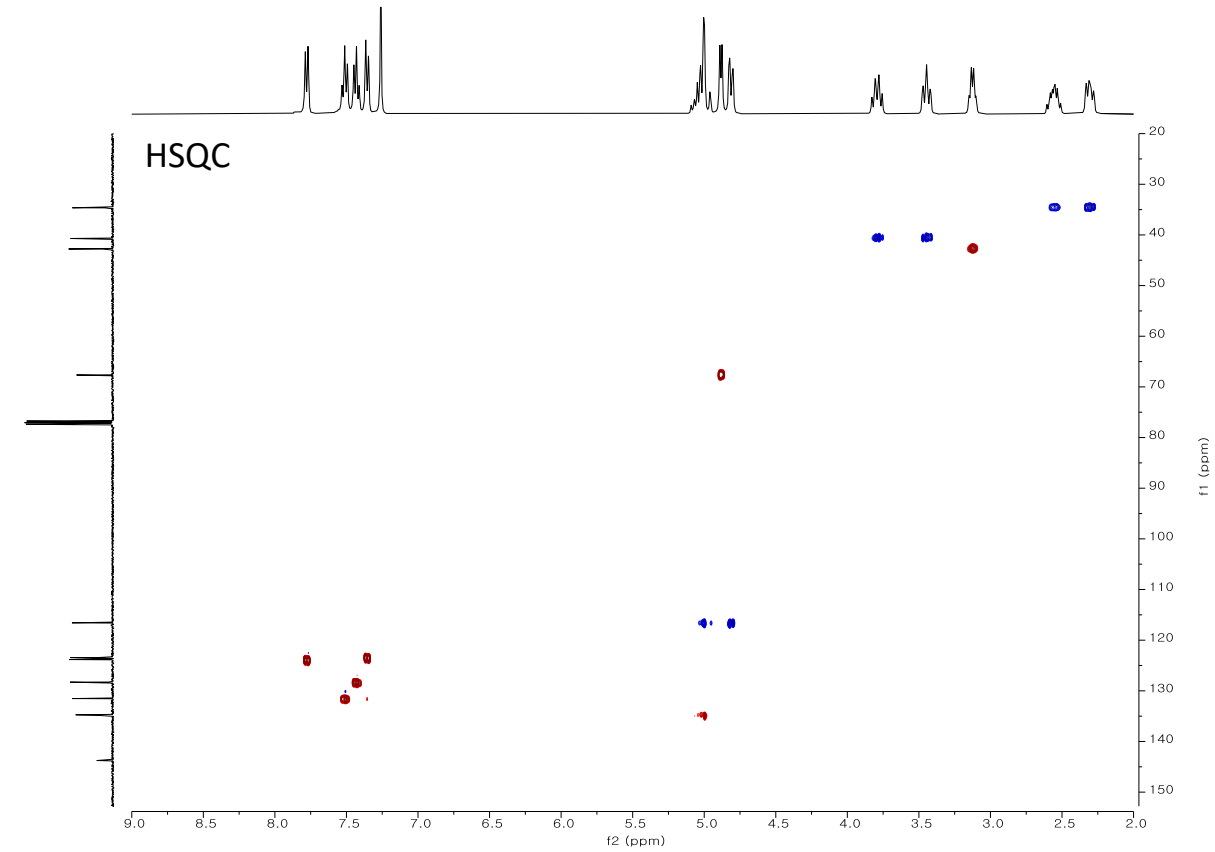

171.0
170.6
170.5
129.3
124.6
94.8
79.4
58.6
39.7
31.9
22.9
21.0
-1.9
f1 (ppm)

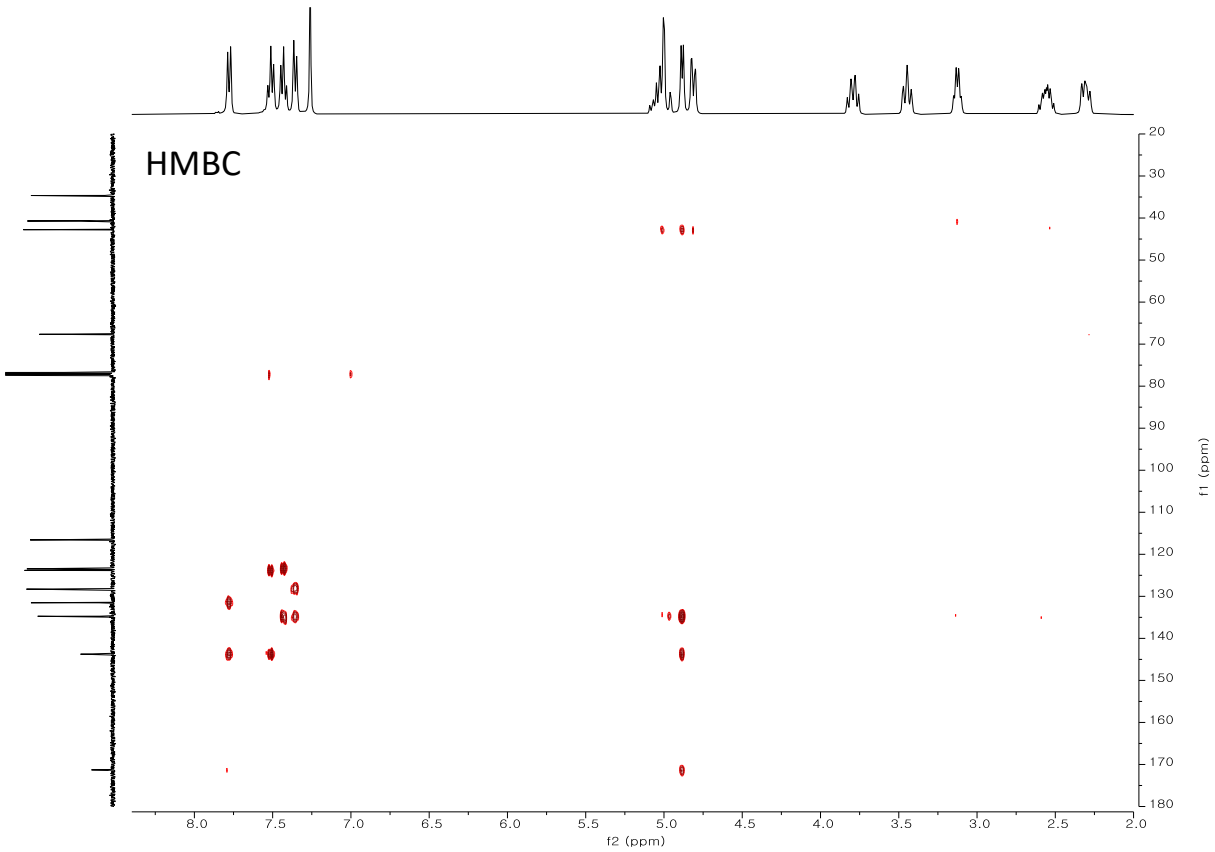

7.79
7.77
7.53
7.51
7.49
7.45
7.43
7.41
7.36
7.35
5.09
5.07
5.05
5.03
5.00
4.96
4.89
4.88
4.83
4.80
3.83
3.81
3.78
3.76
3.48
3.45
3.42
3.15
3.13
3.12
3.10
2.61
2.58
2.57
2.56
2.55
2.53
2.51
2.33
2.31
2.30
2.28
O
N
H
H

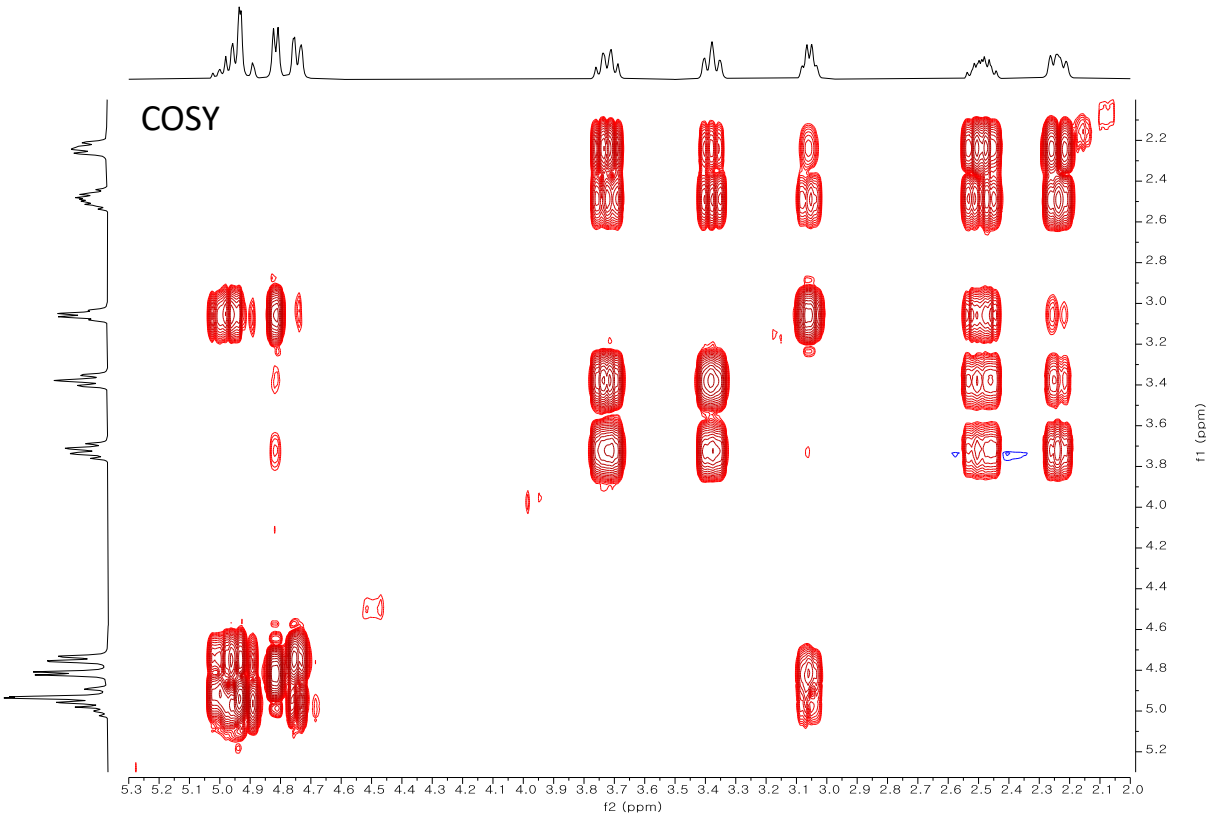
9a

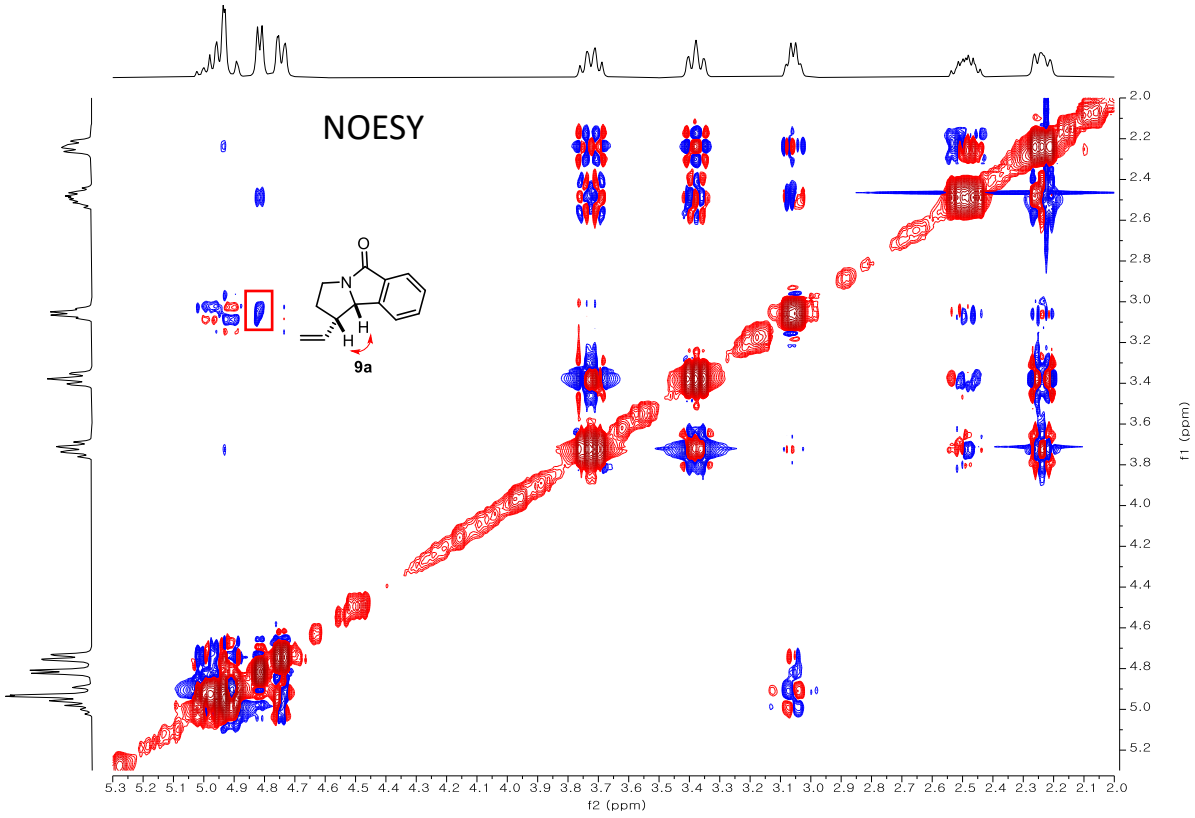

1.00
1.06
1.03
0.96
2.07
1.03
1.02
1.04
1.02
1.02
1.04
1.06
f1 (ppm)

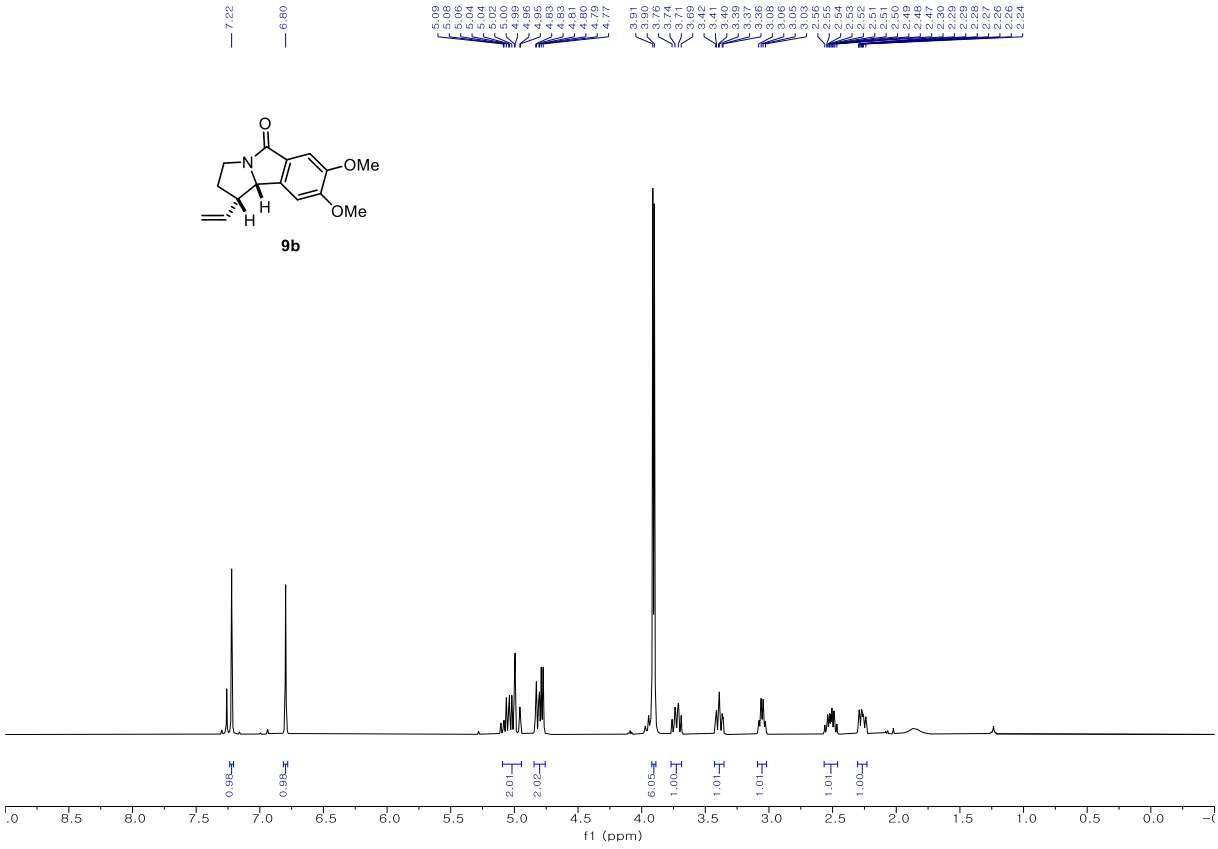

171.4
143.9
135.0
134.8
131.6
128.4
123.9
123.5
116.7
67.7
42.9
40.8
34.7
f1 (ppm)

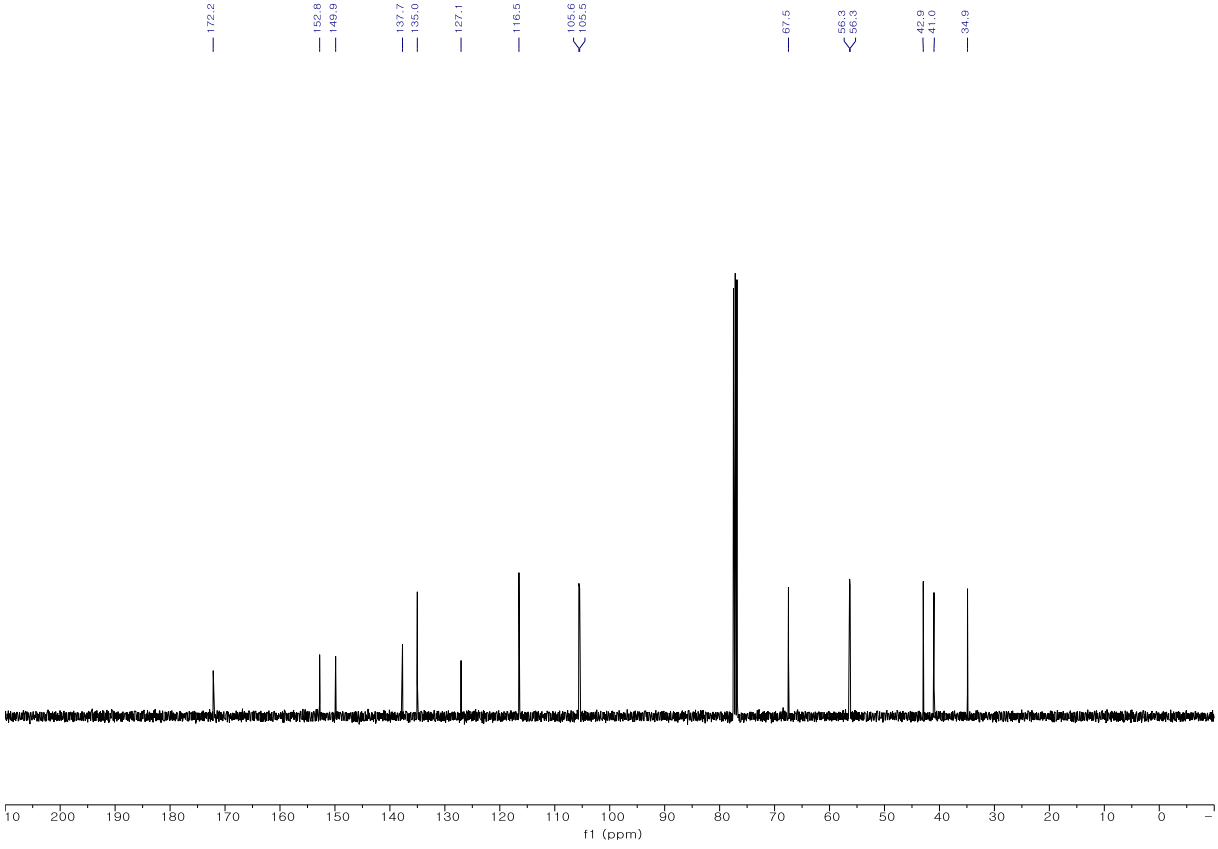

HSQC
f2 (ppm)
f1 (ppm)

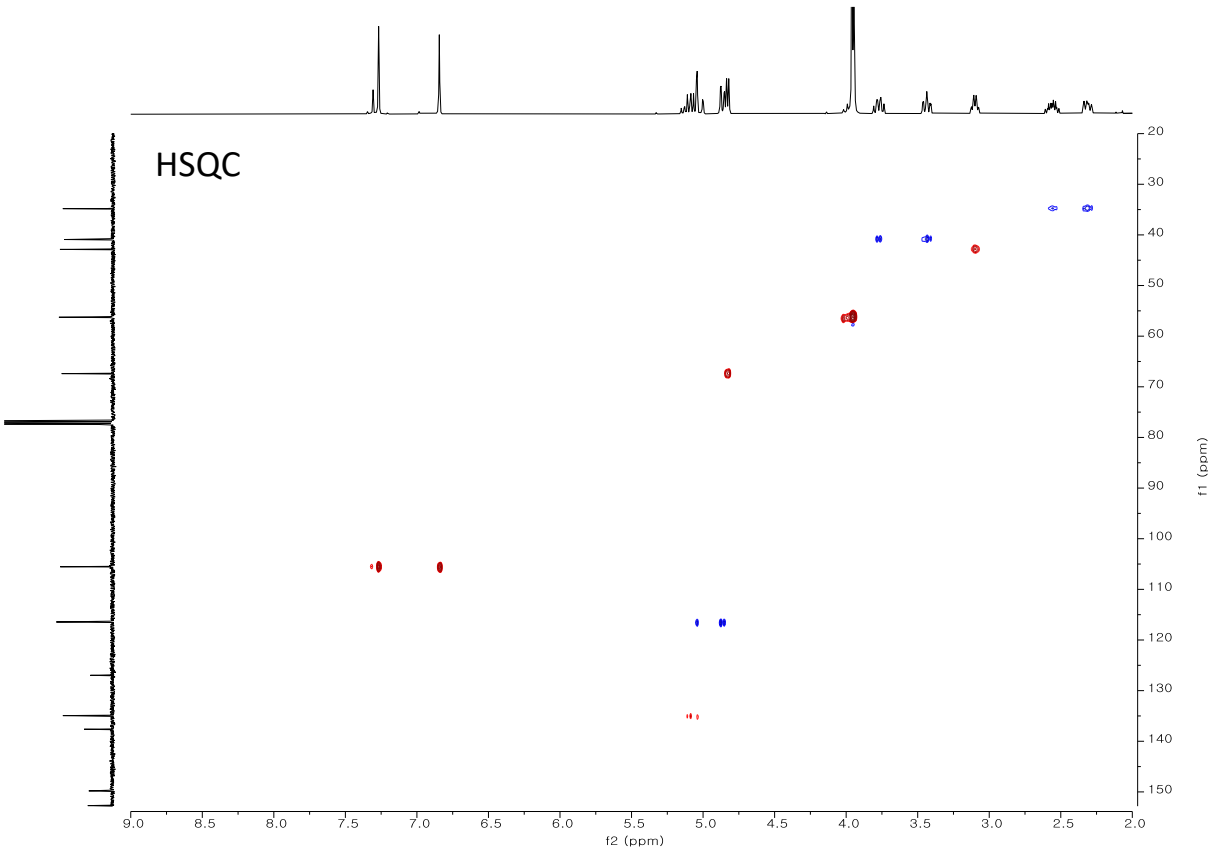

HMBC
f2 (ppm)
f1 (ppm)

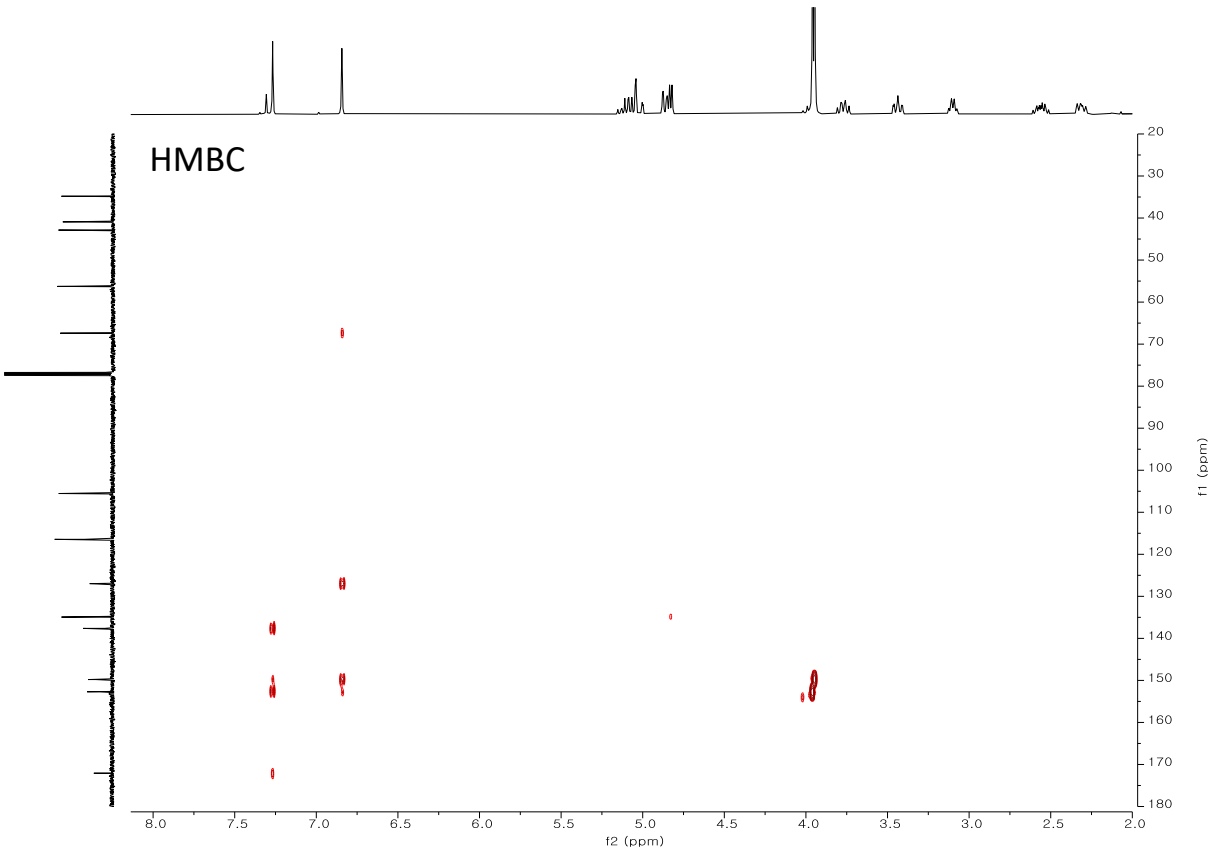

COSY
f2 (ppm)
f1 (ppm)


NOESY
O
N
H
H
9a
f2 (ppm)
f1 (ppm)


7.22
6.80
5.09
5.08
5.06
5.04
5.04
5.02
5.00
4.99
4.96
4.95
4.83
4.83
4.81
4.80
4.79
4.77
3.91
3.90
3.76
3.74
3.71
3.69
3.42
3.41
3.40
3.39
3.37
3.36
3.08
3.06
3.05
3.03
2.56
2.55
2.54
2.53
2.52
2.51
2.51
2.50
2.49
2.48
2.47
2.30
2.29
2.29
2.28
2.27
2.26
2.26
2.24
O
N
H
H
OMe
OMe
9b
0.98
0.98
2.01
2.02
6.05
1.00
1.01
1.01
1.01
1.00
f1 (ppm)


172.2
152.8
149.9
137.7
135.0
127.1
116.5
105.6
105.5
67.5
56.3
56.3
42.9
41.0
34.9
f1 (ppm)


HSQC
f2 (ppm)
f1 (ppm)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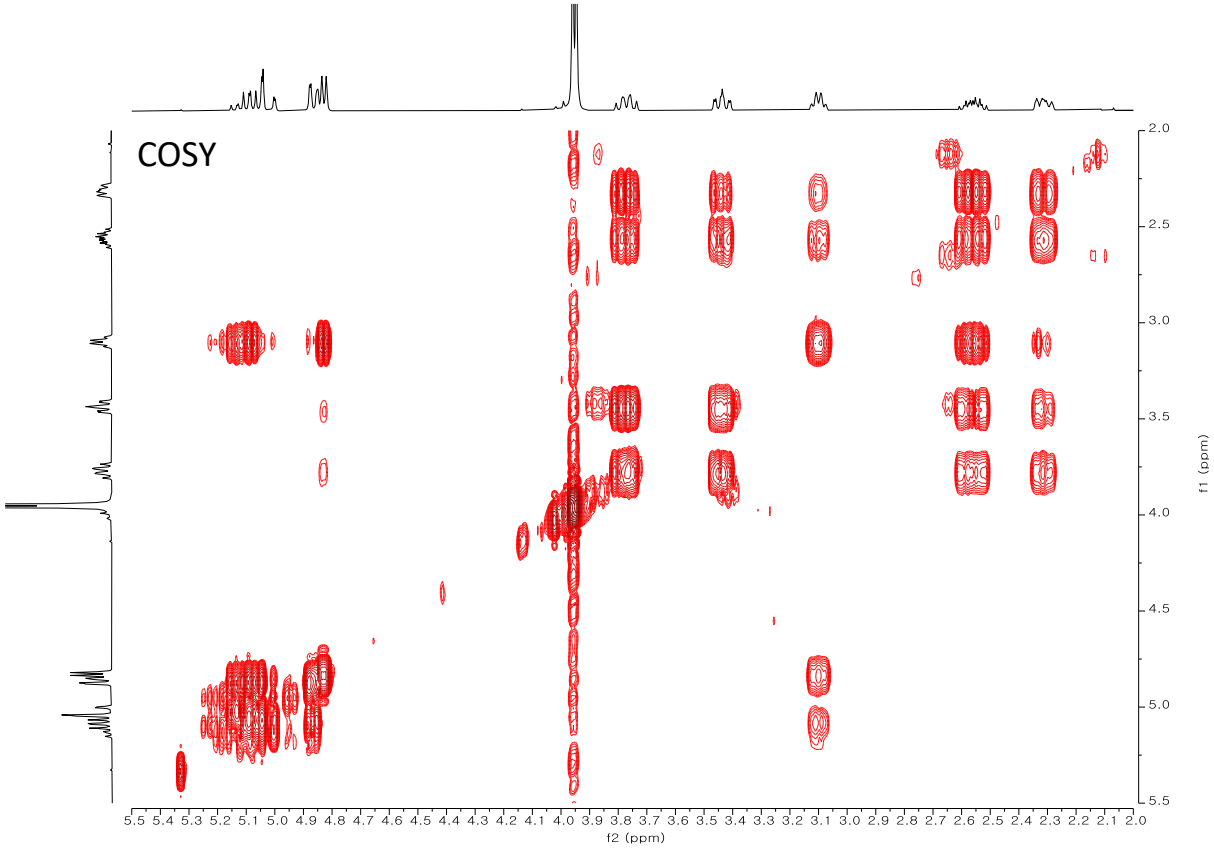
f2 (ppm)
f1 (ppm)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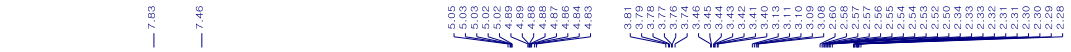
f1 (ppm)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4.86
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4.83
3.81
3.79
3.78
3.77
3.76
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3.46
3.45
3.44
3.43
3.42
3.41
3.40
3.13
3.11
3.10
3.09
3.08
3.00
2.58
2.57
2.57
2.56
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2.54
2.54
2.53
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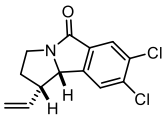
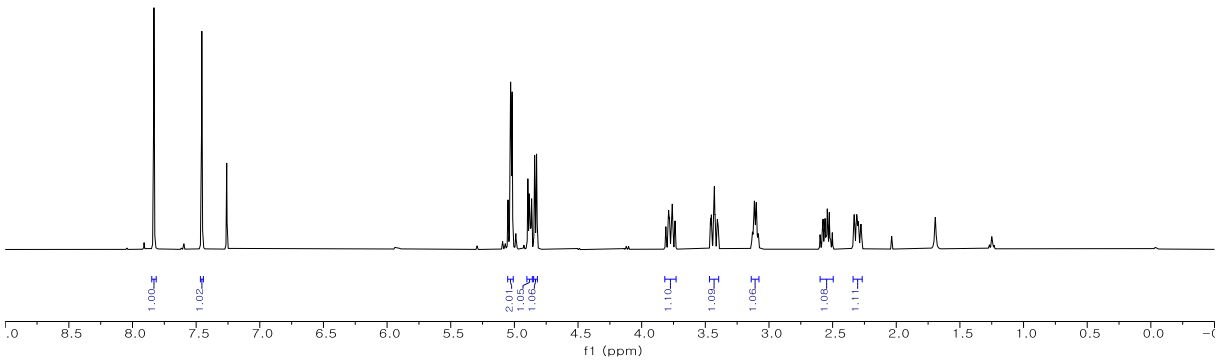
2.30
2.29
2.28


O
N
Cl
H
H
Cl

9c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1.10
1.09
1.06
1.08
1.11
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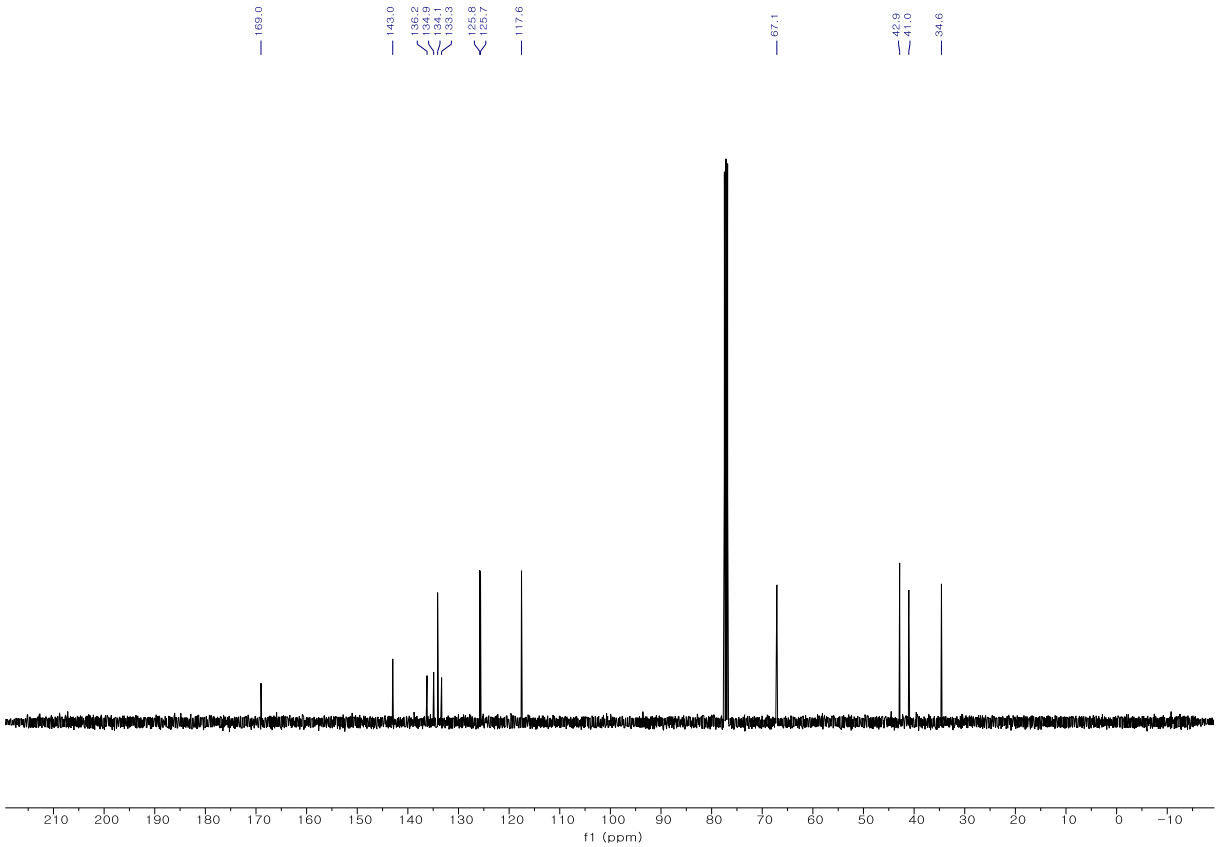
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143.0
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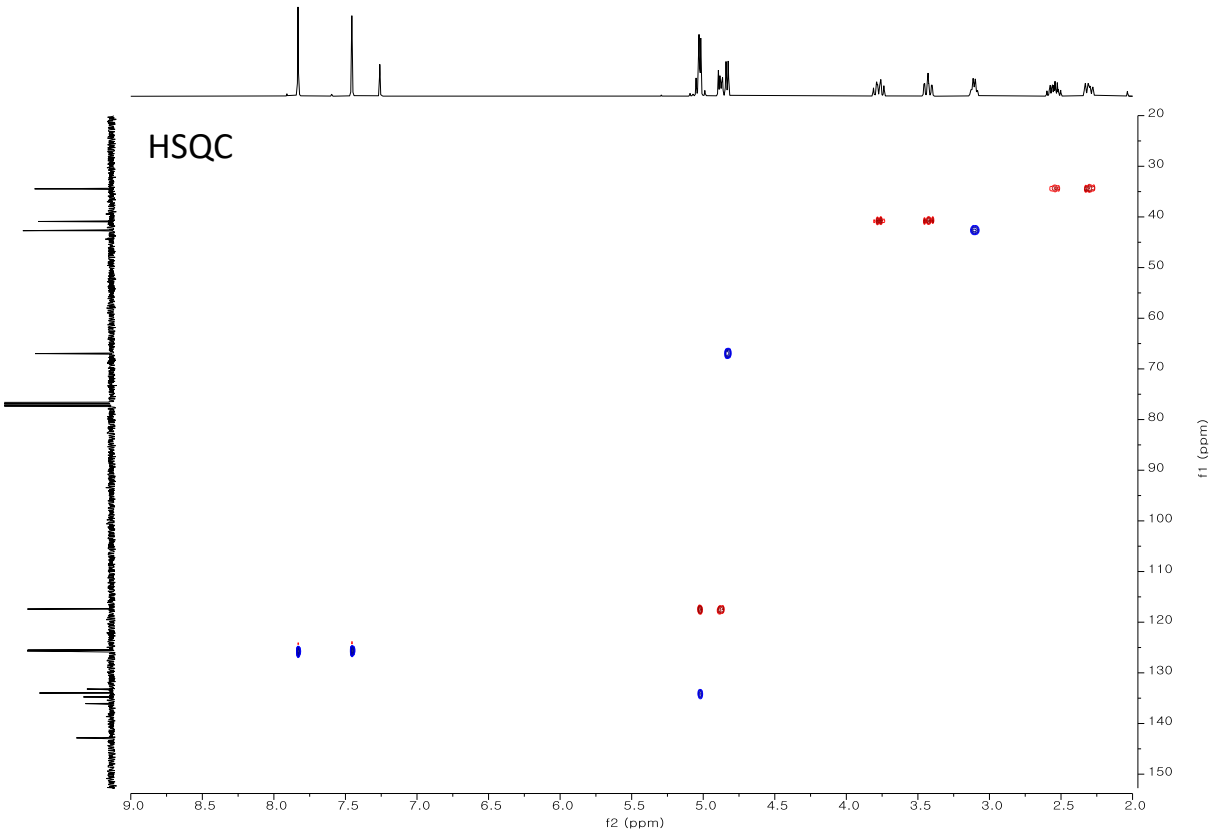
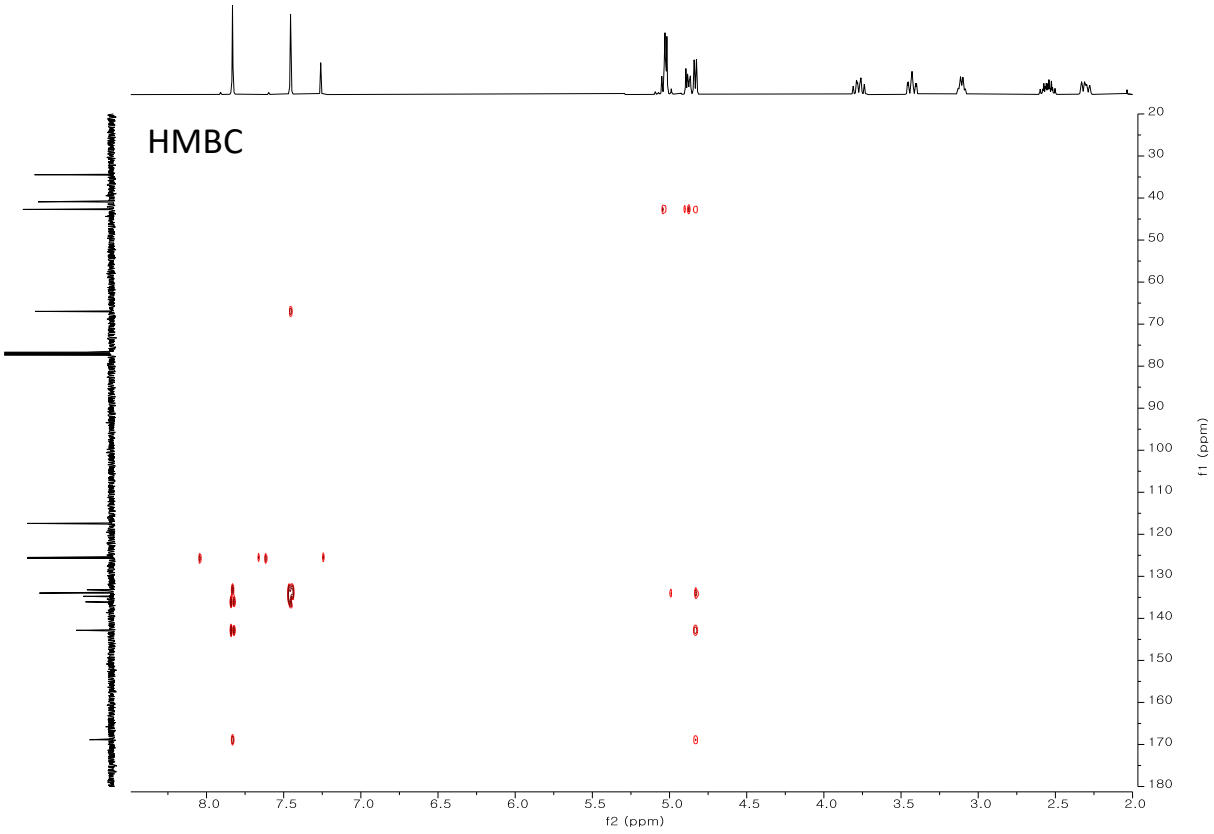
134.9
134.1
133.3
125.8
125.7
117.6
67.1
42.9
41.0
34.6
f1 (ppm)


HSQC
f2 (ppm)
f1 (ppm)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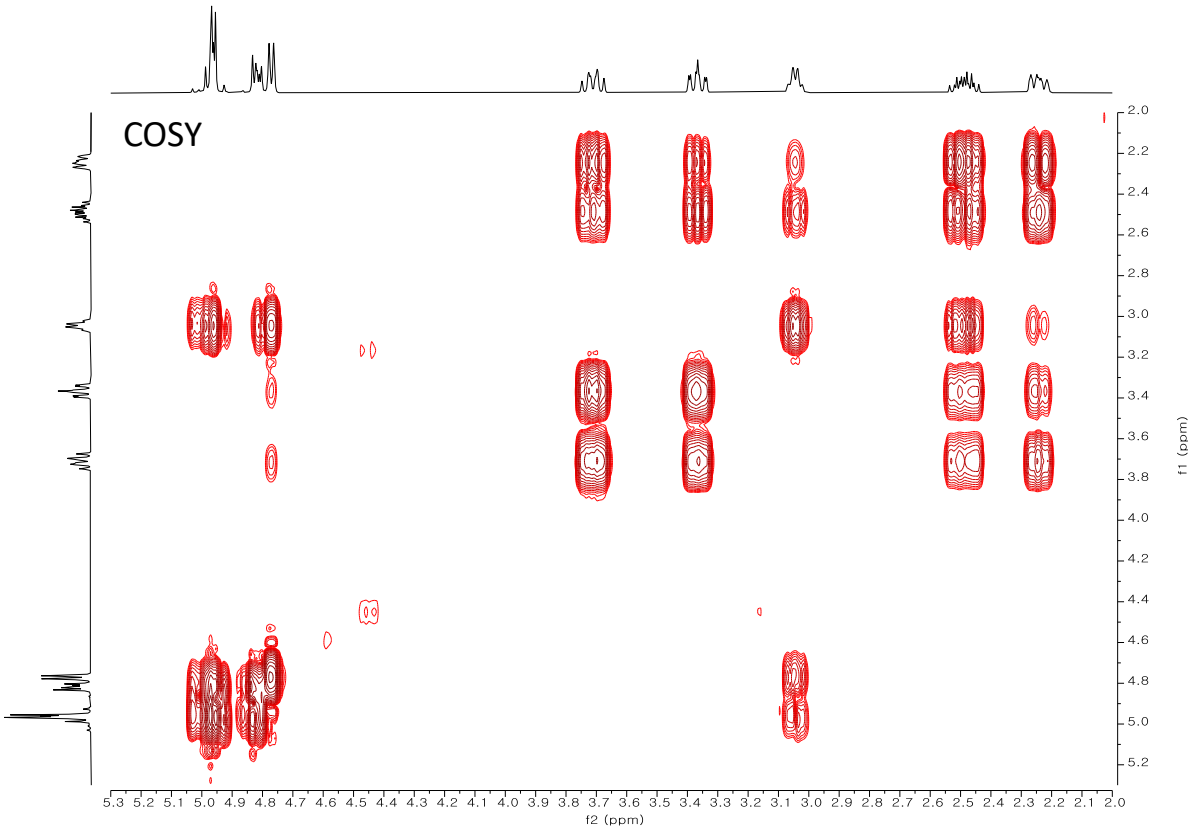
f2 (ppm)
f1 (ppm)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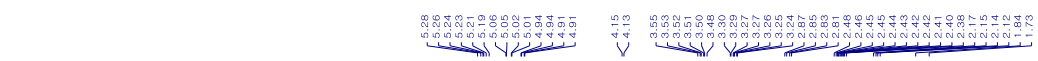
f2 (ppm)
f1 (ppm)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5.01
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4.91
4.15
4.13
3.55
3.53
3.52
3.51
3.50
3.48
3.30
3.29
3.27
3.27
3.26
3.25
3.24
2.87
2.85
2.83
2.81
2.48
2.46
2.45
2.45
2.44
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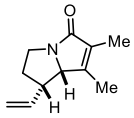
2.17
2.15
2.14
2.12
1.84
1.73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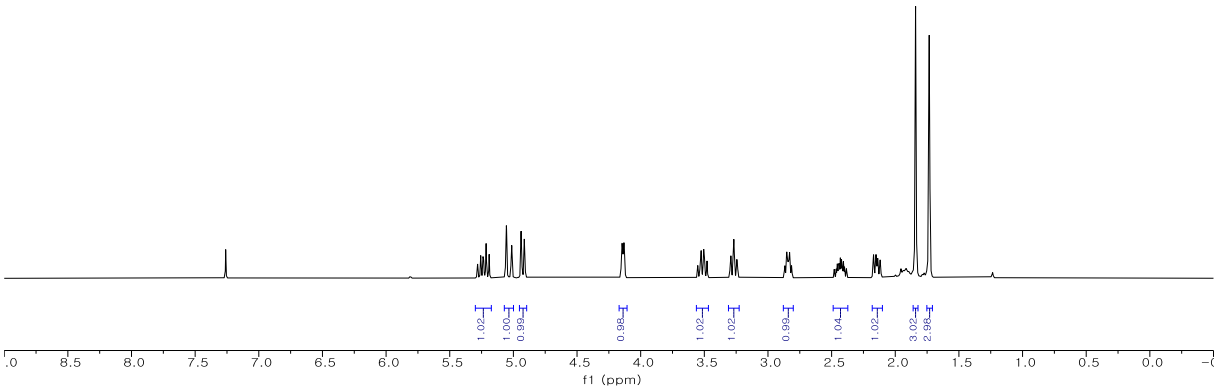
N
Me
Me
H
H

9d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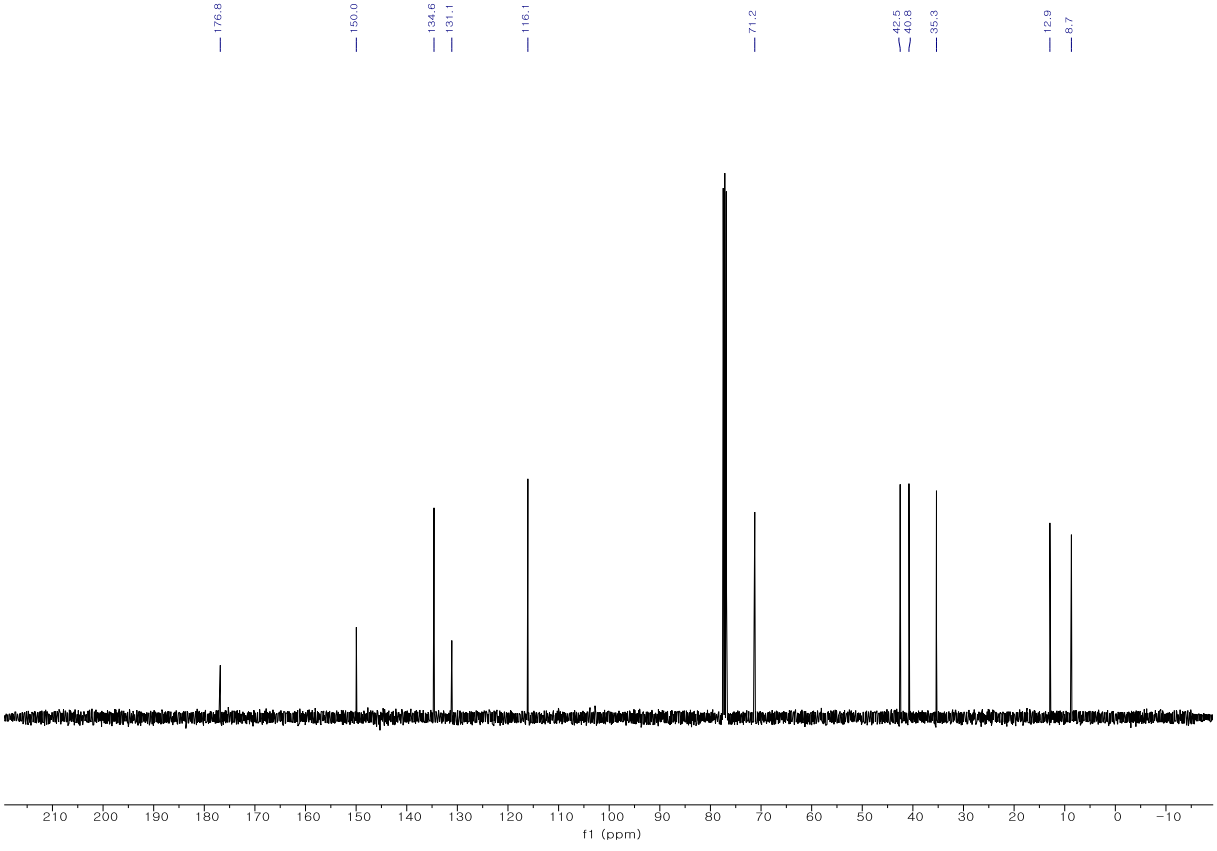
0.99
0.98
1.02
1.02
0.99
1.04
1.02
3.02
2.98
f1 (ppm)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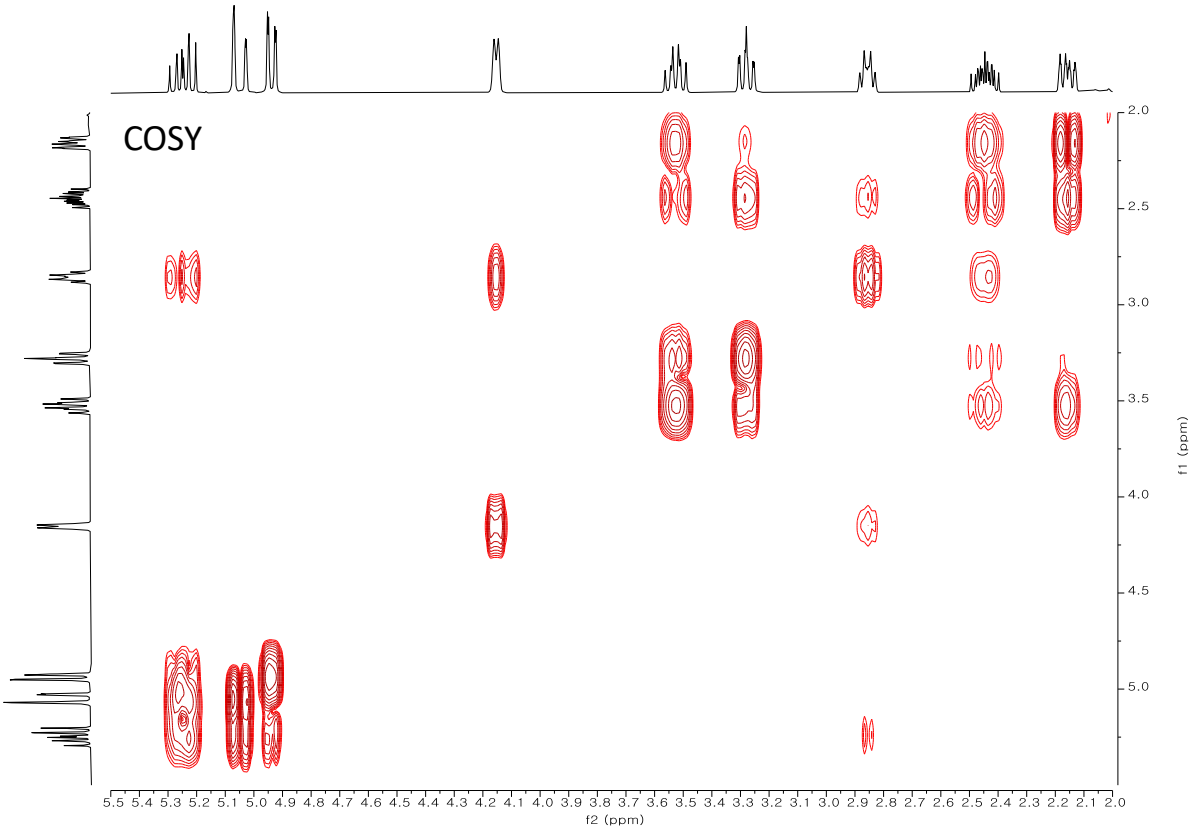
150.0
134.6
131.1
116.1
71.2
42.5
40.8
35.3
12.9
8.7
f1 (ppm)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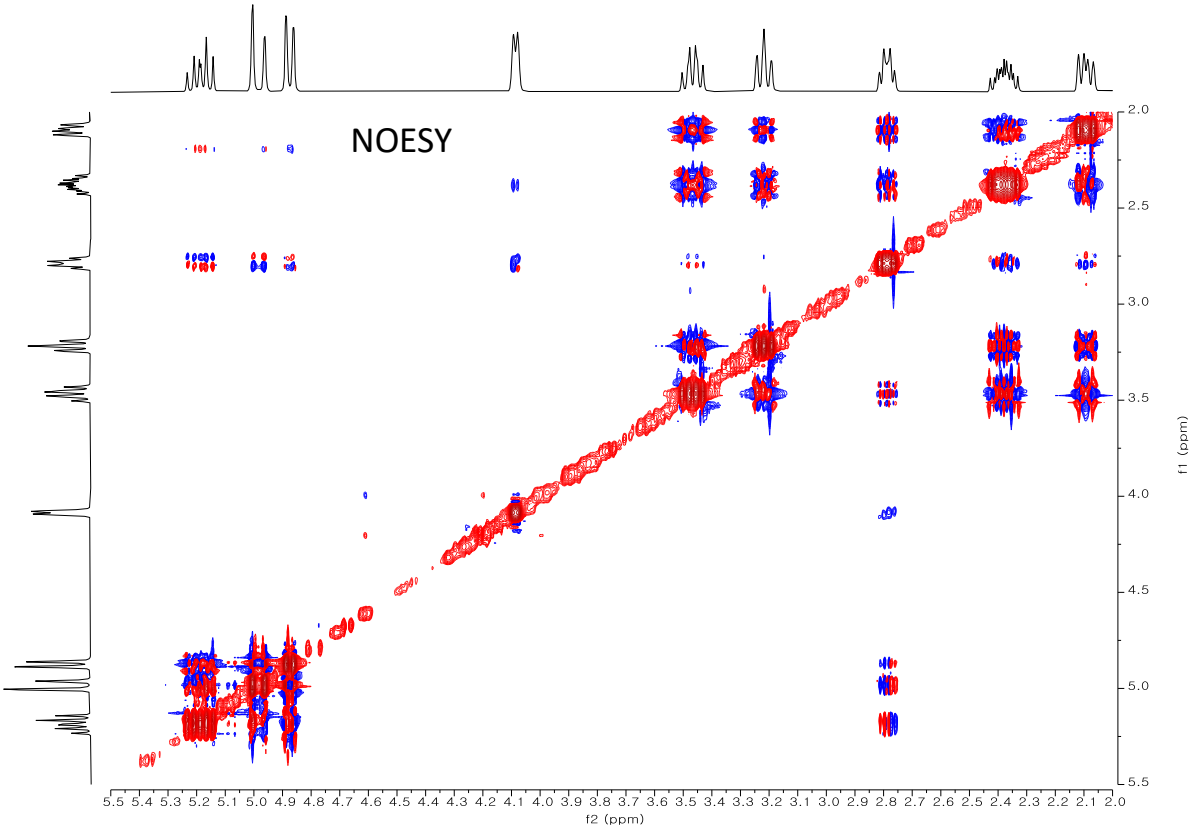
f2 (ppm)
f1 (ppm)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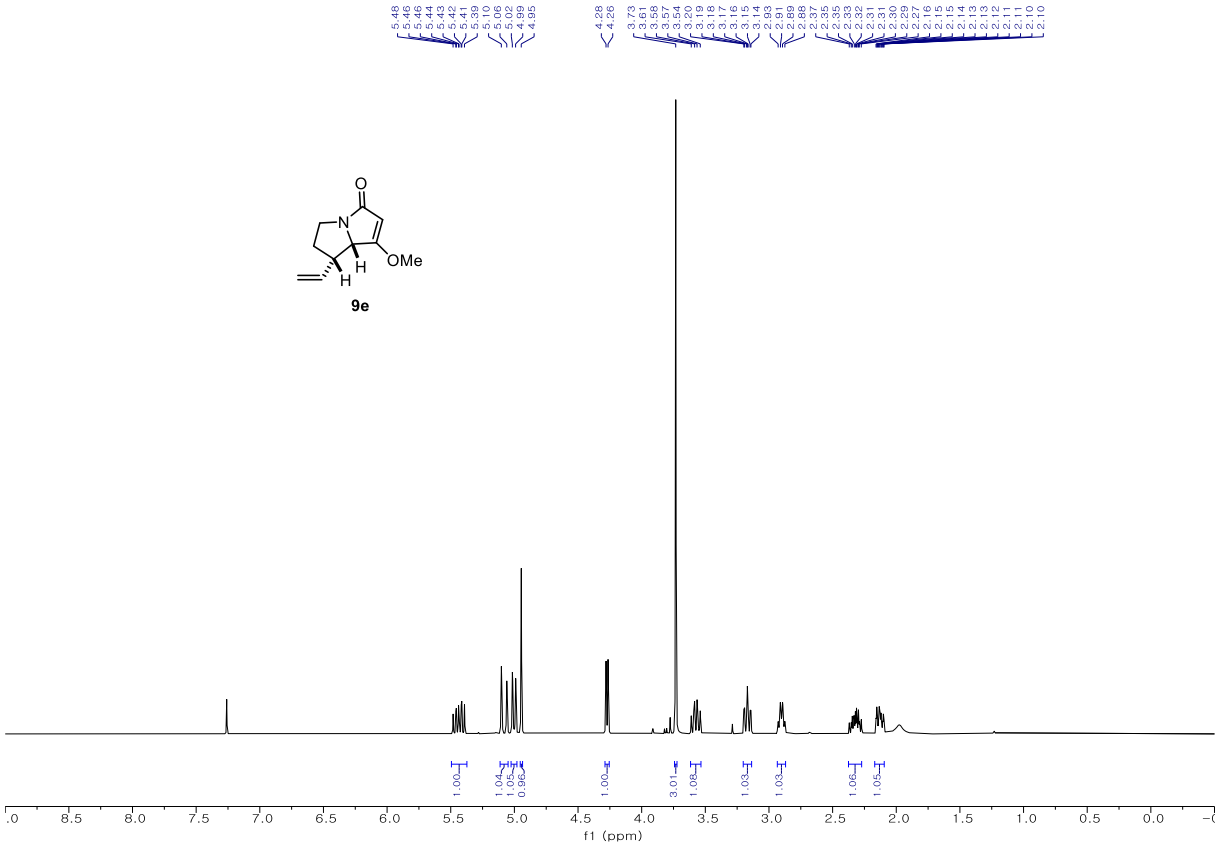
f2 (ppm)
f1 (ppm)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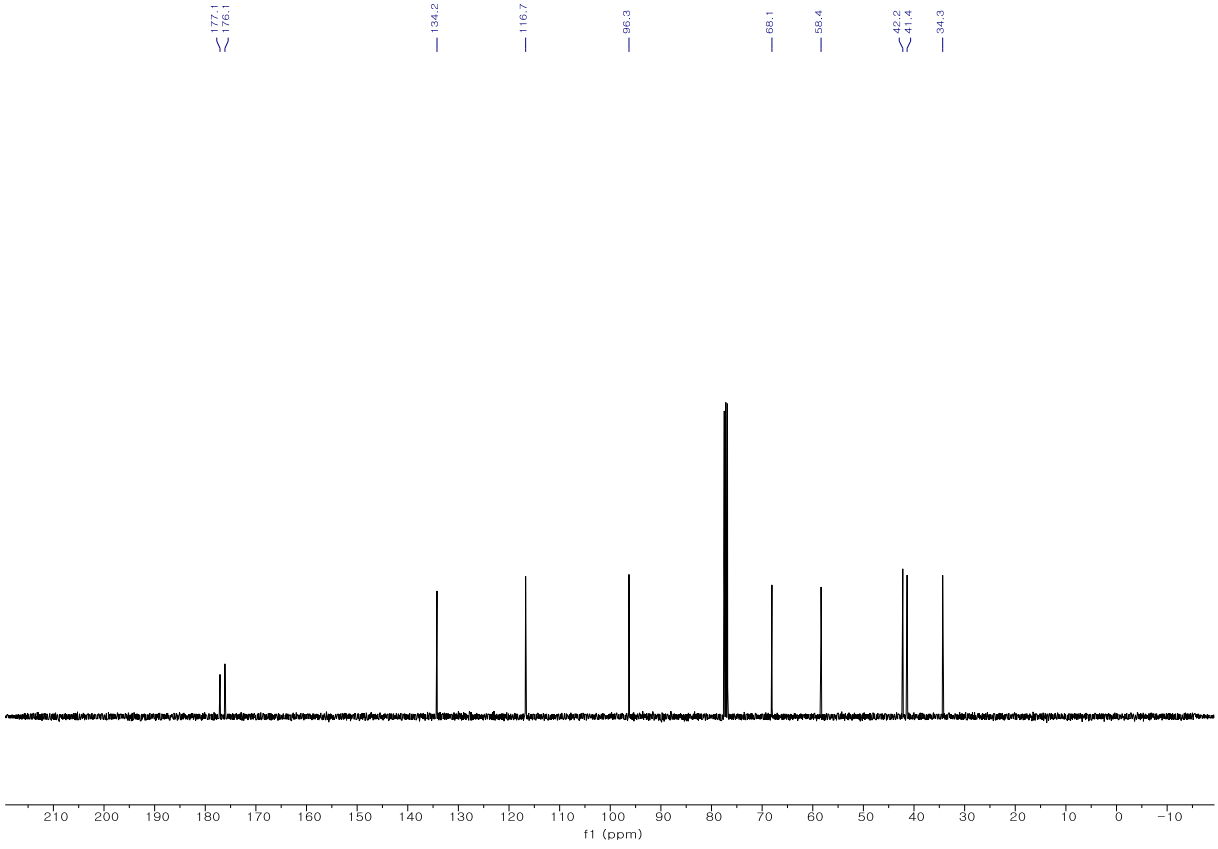
5.43
5.42
5.41
5.39
5.10
5.06
5.02
4.99
4.95
4.28
4.26
3.73
3.61
3.58
3.57
3.54
3.20
3.19
3.18
3.17
3.16
3.15
3.14
2.93
2.91
2.89
2.88
2.37
2.35
2.33
2.32
2.31
2.31
2.30
2.29
2.27
2.16
2.15
2.14
2.13
2.12
2.11
2.11
2.10
2.10
O
N
H
OMe
H
9e
1.00
1.04
1.05
0.96
1.00
3.01
1.08
1.03
1.03
1.06
1.05
f1 (ppm)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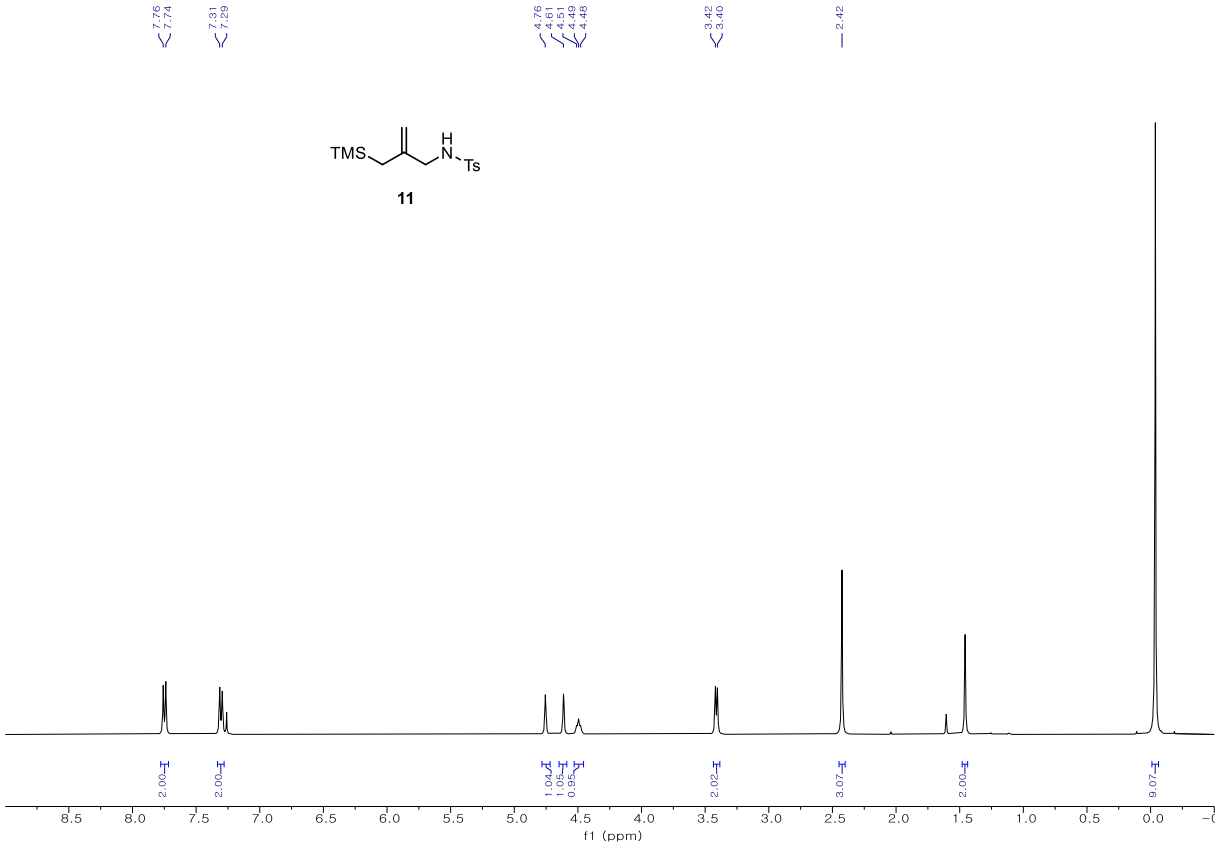
176.1
134.2
116.7
96.3
68.1
58.4
42.2
41.4
34.3
f1 (ppm)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7.74
7.31
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4.76
4.61
4.51
4.49
4.48
3.42
3.40
2.42
TMS
H
N
Ts
11
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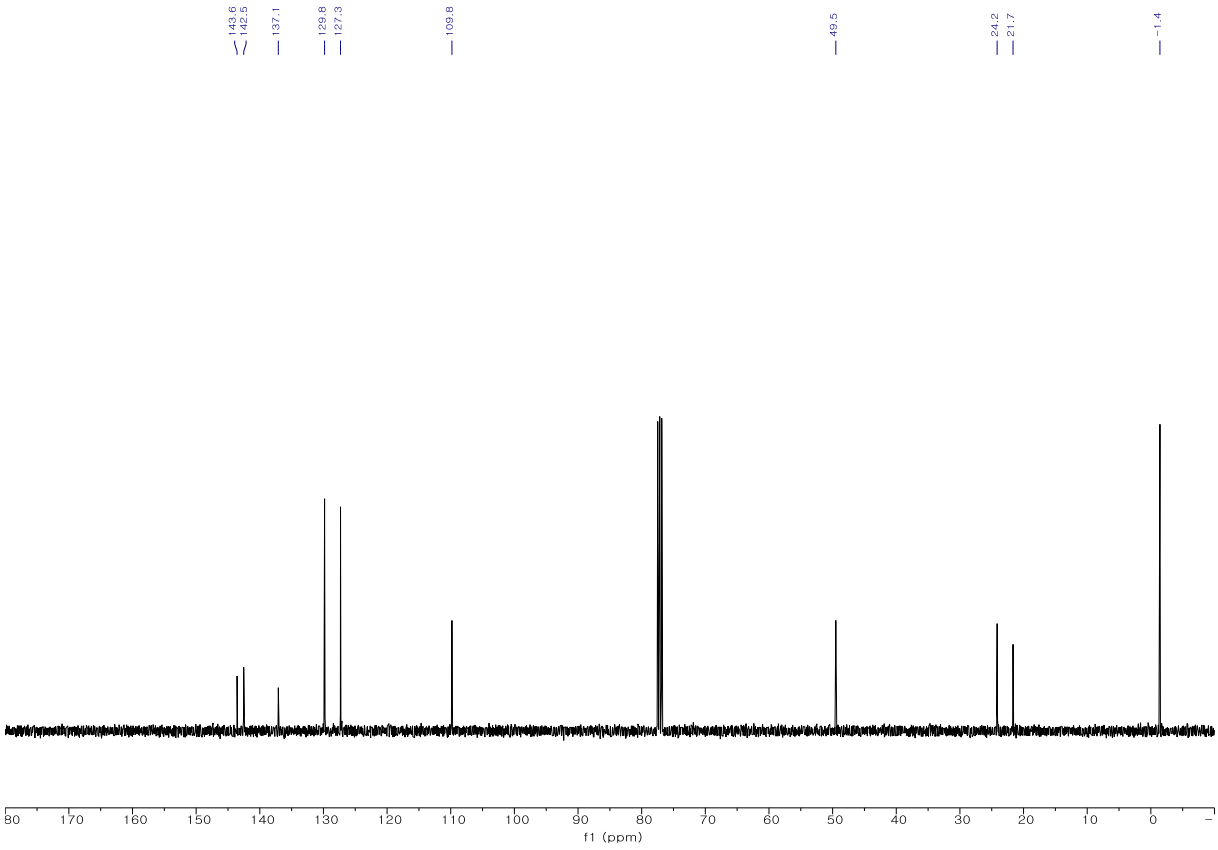
2.00
1.04
1.05
0.95
2.02
3.07
2.00
9.07
f1 (ppm)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142.5
137.1
129.8
127.3
109.8
49.5
24.2
21.7
1.4
f1 (ppm)


7.72
7.70
7.41
7.39
7.34
7.32
7.31
7.29
7.27
7.25
7.23
7.21
5.03
5.02
5.01
4.90
4.10
2.71
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2.68
2.68
2.68
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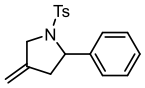
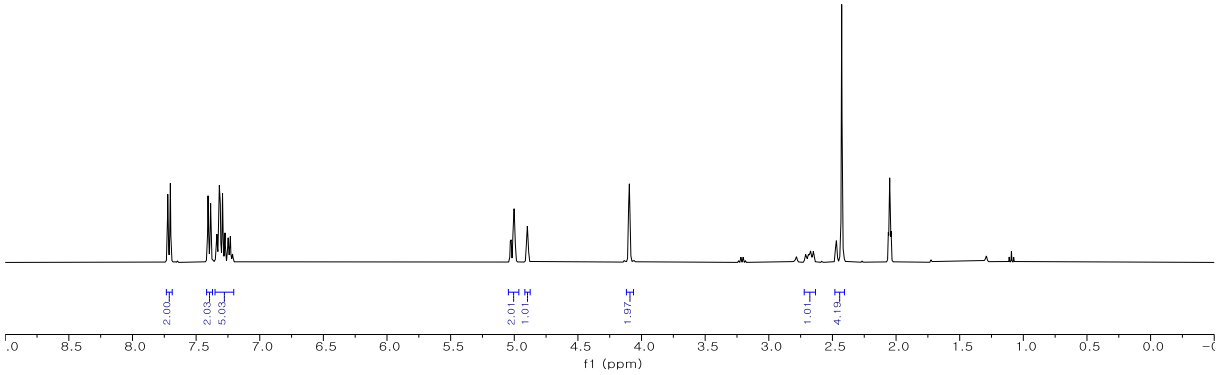
2.67
2.65
2.65
2.64
2.47
2.43


Ts
N

12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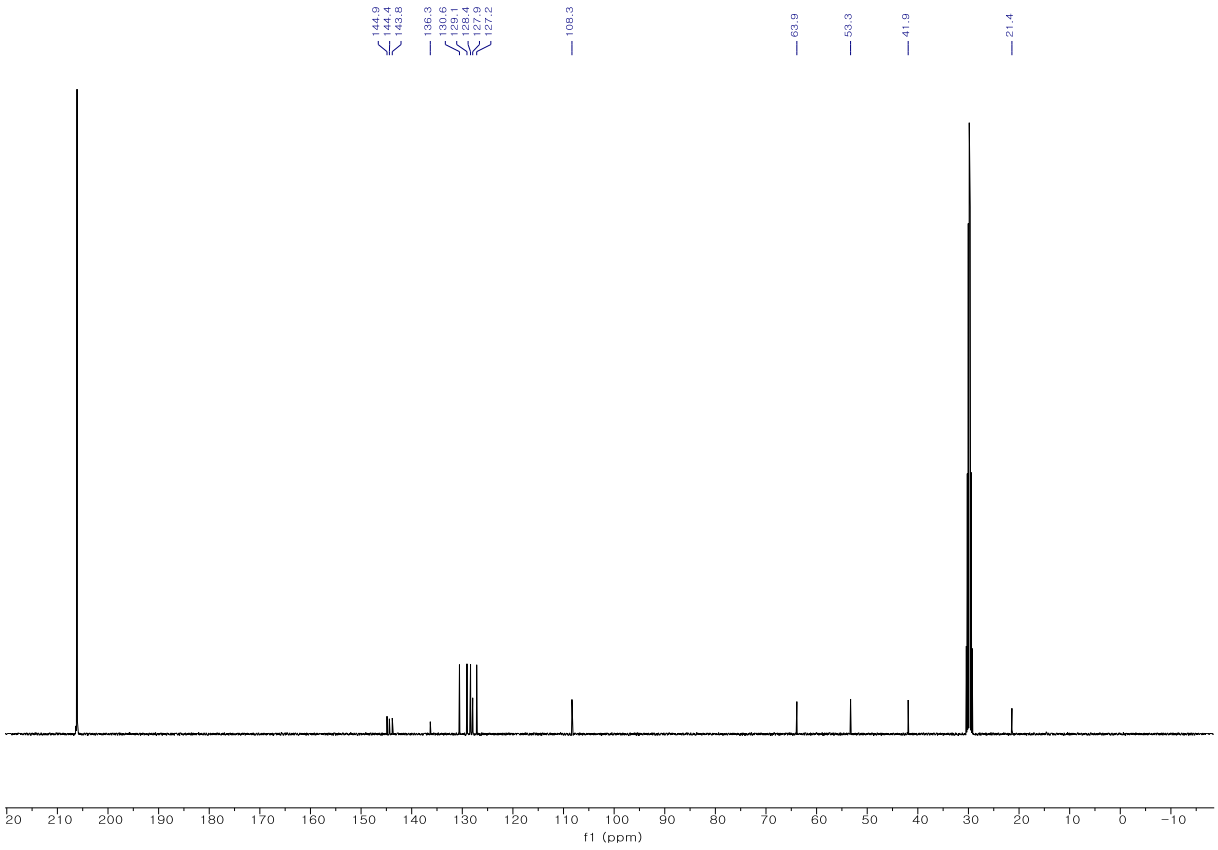
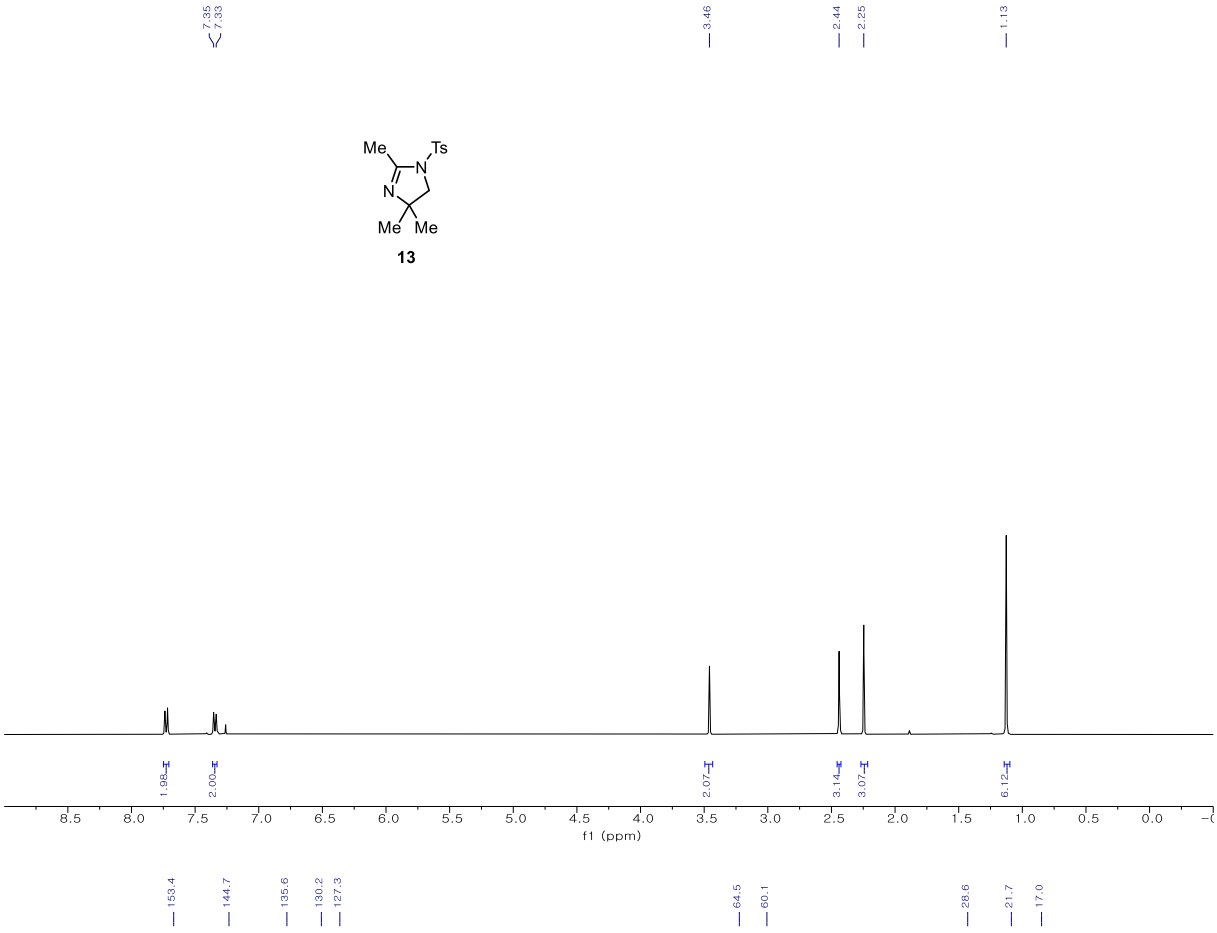
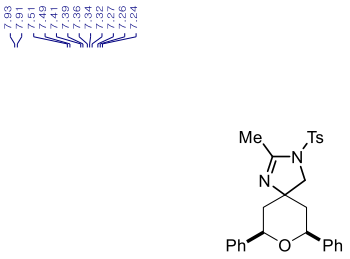
2.03
5.03
2.01
1.01
1.97
1.01
4.19
f1 (ppm)


144.9
144.4
143.8
136.3
130.6
129.1
128.4
127.9
127.2
108.3
63.9
53.3
41.9
21.4
f1 (ppm)


7.35
7.33
3.46
2.44
2.25
1.13
Me
Ts
N
N
Me
Me
13
1.98
2.00
2.07
3.14
3.07
6.12
f1 (ppm)
153.4
144.7
135.6
130.2
127.3
64.5
60.1
28.6
21.7
17.0
f1 (ppm)


Me
Ts
N
N
Ph
O
Ph

14

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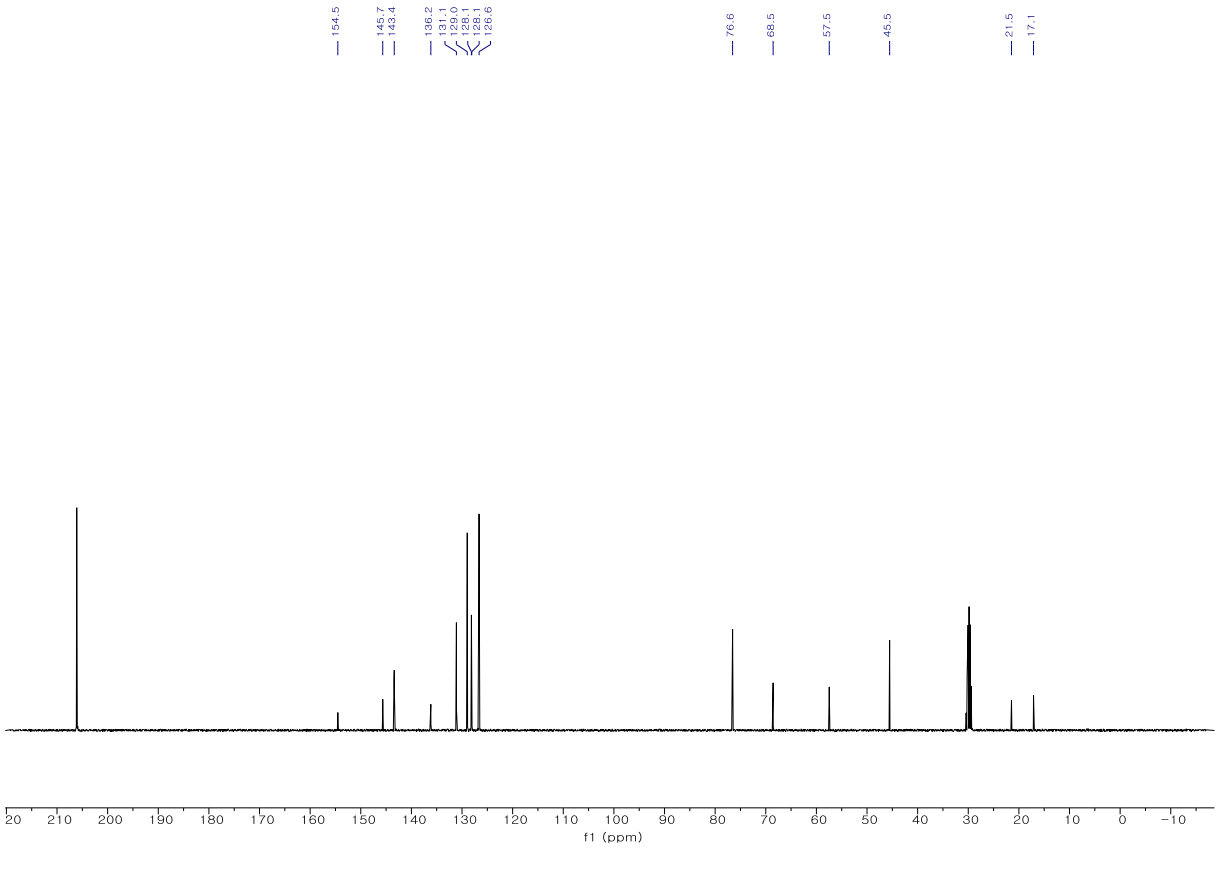
7.39
7.36
7.34
7.32
7.27
7.26
7.24
4.60
4.57
3.99
2.40
2.25
1.76
1.73
1.70
1.40
1.36
1.98
2.00
4.00
4.05
1.98
2.01
2.00
3.00
2.98
2.02
1.99
f1 (ppm)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128.1
126.6
76.6
68.5
57.5
45.5
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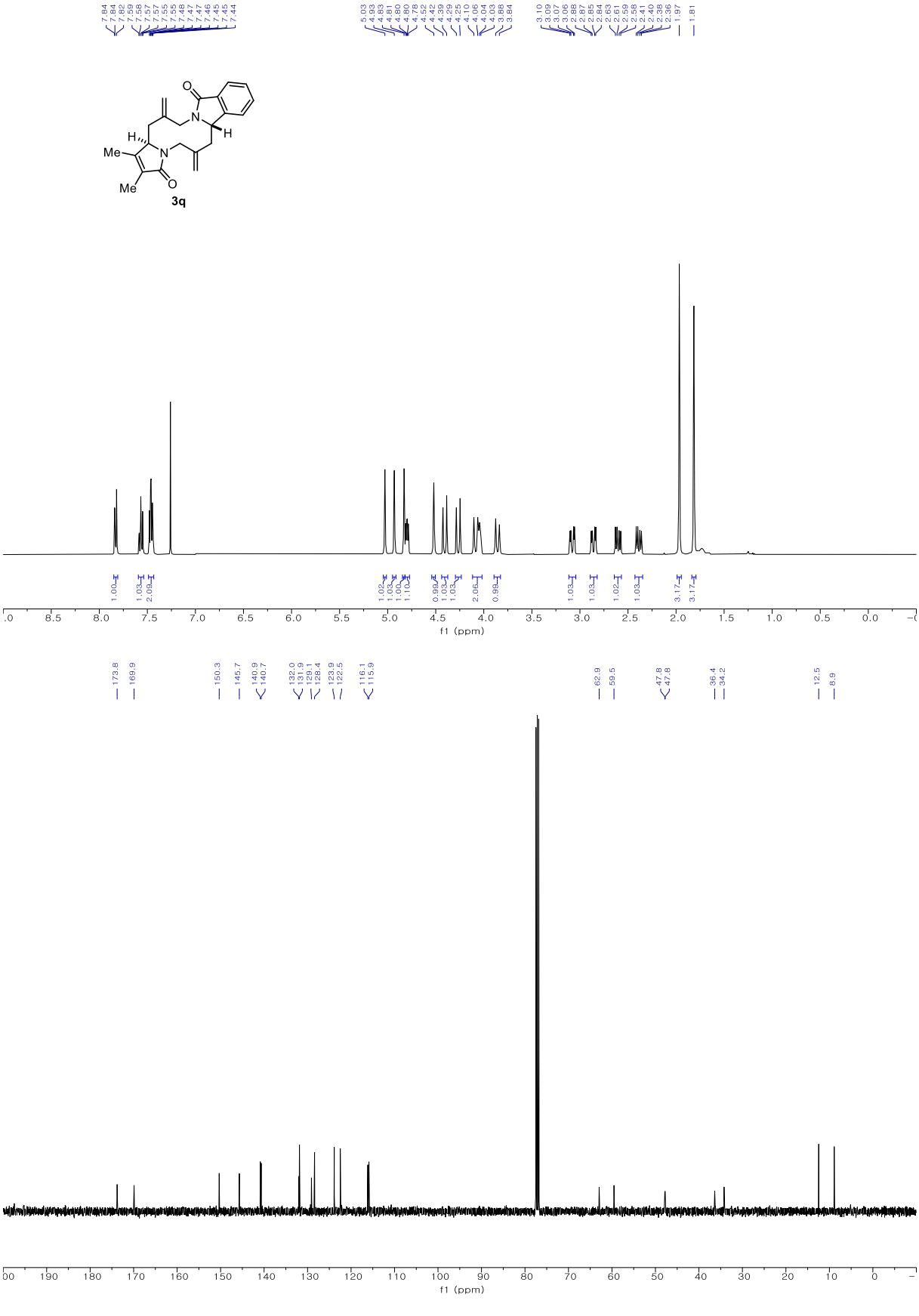
17.1
f1 (ppm)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7.57
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7.55
7.48
7.47
7.47
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7.45
7.45
7.44
5.03
4.93
4.83
4.81
4.80
4.80
4.78
4.52
4.42
4.39
4.29
4.25
4.10
4.06
4.04
4.03
3.88
3.84
3.10
3.09
3.07
3.06
2.88
2.87
2.85
2.84
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H
H
Me
N
Me
O
O
3q
1.00
1.03
2.09
1.02
1.03
1.00
1.10
0.98
1.03
2.06
0.98
1.03
1.03
1.02
1.03
3.17
3.17
f1 (ppm)
173.8
169.9
150.3
145.7
140.9
140.7
132.0
131.9
129.1
128.4
123.9
122.5
116.1
115.9
62.9
59.5
47.8
47.8
36.4
34.2
12.5
8.9
f1 (ppm)

3. X-ray Crystallographic Data

1) Crystallographic Data for **3a**

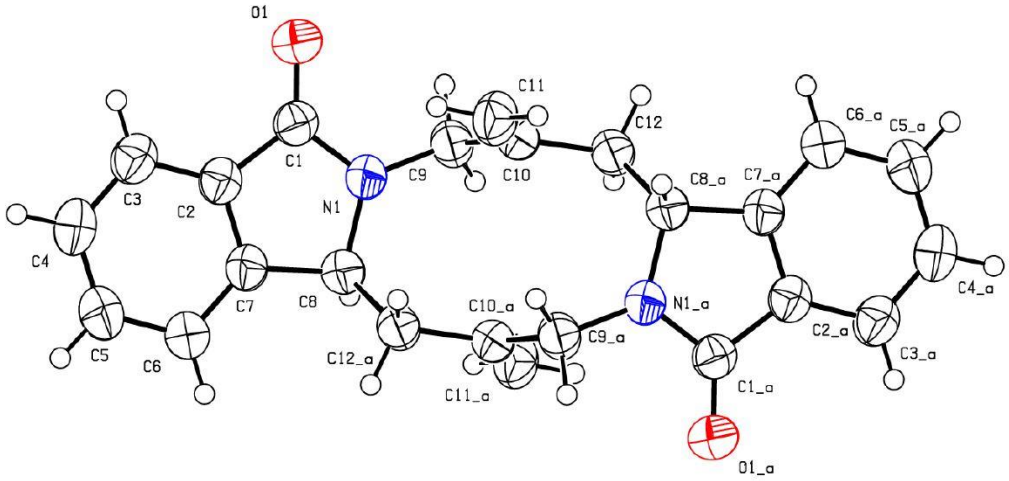


Table 1. Crystal data and structure refinement for kt3_a.

Identification code	kt3_a	
Empirical formula	$C_{24}H_{22}N_2O_2$	
Formula weight	370.43	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 7.1626(14) Å	a = 90°.
	b = 17.647(4) Å	b = 102.13(3)°.
	c = 7.7716(16) Å	c = 90°.
Volume	960.4(3) $Å^3$	
Z	2	
Density (calculated)	1.281 Mg/m^3	
Absorption coefficient	0.082 mm^{-1}	
F(000)	392	
Crystal size	? x ? x ? mm^{-3}	
Theta range for data collection	3.130 to 25.999°.	

Index ranges	-8<=h<=8, -21<=k<=21, -9<=l<=9
Reflections collected	8181
Independent reflections	1885 [R(int) = 0.0513]
Completeness to theta = 25.242°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.5783
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1885 / 0 / 127
Goodness-of-fit on F^2	1.076
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1227
R indices (all data)	R1 = 0.0819, wR2 = 0.1448
Extinction coefficient	n/a
Largest diff. peak and hole	0.242 and -0.174 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for kt3_a.

U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8320(2)	5826(1)	6247(2)	63(1)
N(1)	5681(2)	5424(1)	7243(2)	48(1)
C(1)	6619(3)	5872(1)	6290(2)	47(1)
C(2)	5195(3)	6400(1)	5314(2)	47(1)
C(3)	5415(3)	6970(1)	4144(3)	58(1)
C(4)	3812(4)	7376(1)	3367(3)	69(1)
C(5)	2057(4)	7209(1)	3730(3)	73(1)
C(6)	1832(3)	6639(1)	4886(3)	63(1)
C(7)	3432(3)	6235(1)	5680(3)	48(1)
C(8)	3606(3)	5574(1)	6930(3)	48(1)
C(9)	6601(3)	4792(1)	8290(3)	52(1)
C(10)	7198(3)	4946(1)	10231(3)	48(1)
C(11)	7829(3)	5615(1)	10835(3)	63(1)
C(12)	7151(3)	4268(1)	11418(2)	52(1)

Table 3. Bond lengths [Å] and angles [°] for kt3_a.

O(1)-C(1)	1.228(2)
N(1)-C(1)	1.354(2)
N(1)-C(9)	1.455(2)
N(1)-C(8)	1.479(2)
C(1)-C(2)	1.469(3)
C(2)-C(7)	1.382(3)
C(2)-C(3)	1.386(3)
C(3)-C(4)	1.381(3)
C(3)-H(3)	0.93
C(4)-C(5)	1.377(3)
C(4)-H(4)	0.93
C(5)-C(6)	1.380(3)
C(5)-H(5)	0.93
C(6)-C(7)	1.380(3)
C(6)-H(6)	0.93
C(7)-C(8)	1.506(3)
C(8)-C(12)#1	1.521(3)
C(8)-H(8)	0.98
C(9)-C(10)	1.504(3)
C(9)-H(9A)	0.97
C(9)-H(9B)	0.97
C(10)-C(11)	1.316(3)
C(10)-C(12)	1.515(3)
C(11)-H(11A)	0.93
C(11)-H(11B)	0.93

C(12)-H(12A)	0.97
C(12)-H(12B)	0.97
C(1)-N(1)-C(9)	122.37(17)
C(1)-N(1)-C(8)	113.74(16)
C(9)-N(1)-C(8)	123.41(16)
O(1)-C(1)-N(1)	125.78(19)
O(1)-C(1)-C(2)	127.81(18)
N(1)-C(1)-C(2)	106.39(16)
C(7)-C(2)-C(3)	121.49(19)
C(7)-C(2)-C(1)	108.99(17)
C(3)-C(2)-C(1)	129.49(19)
C(4)-C(3)-C(2)	117.7(2)
C(4)-C(3)-H(3)	121.1
C(2)-C(3)-H(3)	121.1
C(5)-C(4)-C(3)	120.6(2)
C(5)-C(4)-H(4)	119.7
C(3)-C(4)-H(4)	119.7
C(4)-C(5)-C(6)	121.8(2)
C(4)-C(5)-H(5)	119.1
C(6)-C(5)-H(5)	119.1
C(7)-C(6)-C(5)	117.9(2)
C(7)-C(6)-H(6)	121.1
C(5)-C(6)-H(6)	121.1
C(6)-C(7)-C(2)	120.53(19)
C(6)-C(7)-C(8)	129.52(19)
C(2)-C(7)-C(8)	109.91(16)
N(1)-C(8)-C(7)	100.86(15)

N(1)-C(8)-C(12)#1	114.73(16)
C(7)-C(8)-C(12)#1	114.02(17)
N(1)-C(8)-H(8)	109
C(7)-C(8)-H(8)	109
C(12)#1-C(8)-H(8)	109
N(1)-C(9)-C(10)	114.99(17)
N(1)-C(9)-H(9A)	108.5
C(10)-C(9)-H(9A)	108.5
N(1)-C(9)-H(9B)	108.5
C(10)-C(9)-H(9B)	108.5
H(9A)-C(9)-H(9B)	107.5
C(11)-C(10)-C(9)	121.63(19)
C(11)-C(10)-C(12)	122.75(18)
C(9)-C(10)-C(12)	115.49(17)
C(10)-C(11)-H(11A)	120
C(10)-C(11)-H(11B)	120
H(11A)-C(11)-H(11B)	120
C(10)-C(12)-C(8)#1	115.30(17)
C(10)-C(12)-H(12A)	108.4
C(8)#1-C(12)-H(12A)	108.4
C(10)-C(12)-H(12B)	108.4
C(8)#1-C(12)-H(12B)	108.4
H(12A)-C(12)-H(12B)	107.5

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y+1, -z+2

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for kt3_a.
The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U11	U22	U33	U23	U13	U12
O(1)	49(1)	76(1)	65(1)	7(1)	15(1)	-1(1)
N(1)	49(1)	52(1)	44(1)	7(1)	12(1)	3(1)
C(1)	48(1)	54(1)	40(1)	-4(1)	8(1)	-4(1)
C(2)	52(1)	47(1)	39(1)	-3(1)	7(1)	-5(1)
C(3)	62(1)	56(1)	56(1)	3(1)	14(1)	-9(1)
C(4)	80(2)	62(2)	66(1)	20(1)	18(1)	0(1)
C(5)	71(2)	75(2)	73(2)	25(1)	15(1)	16(1)
C(6)	55(1)	72(2)	62(1)	18(1)	13(1)	6(1)
C(7)	52(1)	50(1)	42(1)	4(1)	9(1)	0(1)
C(8)	47(1)	52(1)	45(1)	3(1)	9(1)	1(1)
C(9)	55(1)	54(1)	45(1)	5(1)	11(1)	10(1)
C(10)	44(1)	54(1)	46(1)	5(1)	10(1)	4(1)
C(11)	69(1)	71(2)	46(1)	6(1)	7(1)	-8(1)
C(12)	52(1)	56(1)	47(1)	5(1)	10(1)	8(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for kt3_a.

	x	y	z	U(eq)
H(3)	6602	7074	3892	69
H(4)	3918	7767	2590	83
H(5)	995	7487	3182	88
H(6)	641	6530	5123	75
H(8)	2918	5140	6313	58
H(9A)	7722	4646	7852	62
H(9B)	5731	4364	8118	62
H(11A)	7914	6007	10056	75
H(11B)	8190	5696	12042	75
H(12A)	8436	4068	11766	62
H(12B)	6366	3878	10744	62

Table 6. Torsion angles [°] for kt3_a.

C(9)-N(1)-C(1)-O(1)	-3.2(3)
C(8)-N(1)-C(1)-O(1)	-175.60(19)
C(9)-N(1)-C(1)-C(2)	175.53(16)
C(8)-N(1)-C(1)-C(2)	3.2(2)
O(1)-C(1)-C(2)-C(7)	177.1(2)
N(1)-C(1)-C(2)-C(7)	-1.6(2)
O(1)-C(1)-C(2)-C(3)	-0.8(4)
N(1)-C(1)-C(2)-C(3)	-179.5(2)
C(7)-C(2)-C(3)-C(4)	0.8(3)
C(1)-C(2)-C(3)-C(4)	178.44(19)
C(2)-C(3)-C(4)-C(5)	-1.0(3)
C(3)-C(4)-C(5)-C(6)	0.6(4)
C(4)-C(5)-C(6)-C(7)	0.0(4)
C(5)-C(6)-C(7)-C(2)	-0.2(3)
C(5)-C(6)-C(7)-C(8)	-177.6(2)
C(3)-C(2)-C(7)-C(6)	-0.2(3)
C(1)-C(2)-C(7)-C(6)	-178.29(18)
C(3)-C(2)-C(7)-C(8)	177.69(18)
C(1)-C(2)-C(7)-C(8)	-0.4(2)
C(1)-N(1)-C(8)-C(7)	-3.3(2)
C(9)-N(1)-C(8)-C(7)	-175.55(17)
C(1)-N(1)-C(8)-C(12)#1	-126.28(19)
C(9)-N(1)-C(8)-C(12)#1	61.4(2)
C(6)-C(7)-C(8)-N(1)	179.7(2)
C(2)-C(7)-C(8)-N(1)	2.1(2)

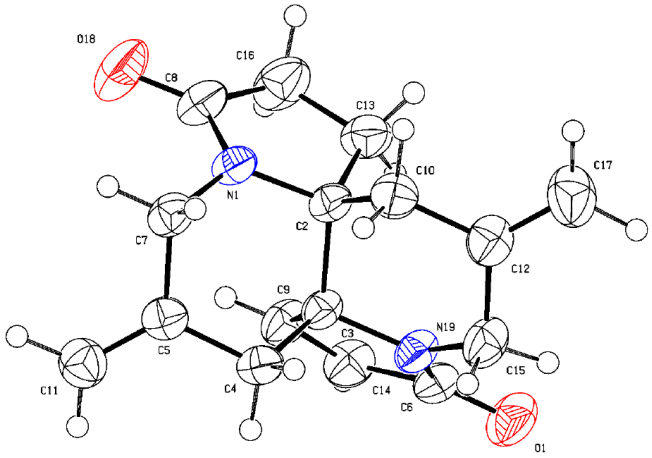
C(6)-C(7)-C(8)-C(12)#1	-56.8(3)
C(2)-C(7)-C(8)-C(12)#1	125.57(18)
C(1)-N(1)-C(9)-C(10)	101.1(2)
C(8)-N(1)-C(9)-C(10)	-87.3(2)
N(1)-C(9)-C(10)-C(11)	-35.4(3)
N(1)-C(9)-C(10)-C(12)	148.67(17)
C(11)-C(10)-C(12)-C(8)#1	44.7(3)
C(9)-C(10)-C(12)-C(8)#1	-139.39(18)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+2

Table 7. Hydrogen bonds for kt3_a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

2) Crystallographic Data for **4**



A. Crystal Data

Empirical Formula	$C_{16}H_{18}N_2O_2$
Formula Weight	270.33
Crystal Color, Habit	yellow, chunk
Crystal Dimensions	0.300 X 0.200 X 0.200 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 10.3324(8) Å b = 10.1087(6) Å c = 13.894(1) Å β = 109.438(3)° V = 1368.5(2) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.312 g/cm^3
F_{000}	576.00
μ(MoKα)	0.874 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	23.0°C
Detector Aperture	280 x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0°
Exposure Rate	90.0 sec./°
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0°
Exposure Rate	90.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
2θ max	54.9°
No. of Reflections Measured	Total: 12866 Unique: 3115 (R_{int} = 0.0295)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.762 - 0.983)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(F_o^2) + (0.0731 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (Max(F_o^2,0) + 2F_c^2)/3$
$2\theta_{max}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3115
No. Variables	205
Reflection/Parameter Ratio	15.20
Residuals: R1 (I>2.00σ(I))	0.0400
Residuals: R (All reflections)	0.0698
Residuals: wR2 (All reflections)	0.1359
Goodness of Fit Indicator	1.145
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.19 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.20 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	Beq
O1	0.2783(2)	0.0362(2)	0.64344(9)	4.68(3)
O2	0.1934(2)	-0.0909(2)	0.09421(9)	5.62(4)
N1	0.2201(2)	0.1237(1)	0.48149(9)	3.04(3)
N2	0.2010(2)	0.0712(1)	0.21075(9)	2.94(3)
C1	0.2681(2)	0.1012(2)	0.3195(1)	2.80(3)
C2	0.1616(2)	0.0803(2)	0.3761(1)	2.69(3)
C3	0.0271(2)	0.1548(2)	0.3204(1)	3.26(3)
C4	-0.0292(2)	0.1195(2)	0.2087(1)	3.09(3)
C5	0.0719(2)	0.1362(2)	0.1526(1)	3.28(3)
C6	0.2441(2)	-0.0422(2)	0.1792(2)	3.79(4)
C7	0.3608(3)	-0.0964(2)	0.2667(2)	5.03(5)
C8	0.3878(2)	0.0025(2)	0.3511(2)	3.87(4)
C9	0.3203(2)	0.2450(2)	0.3359(2)	3.50(3)
C10	0.3789(2)	0.2784(2)	0.4473(2)	3.65(4)
C11	0.2824(2)	0.2542(2)	0.5063(2)	3.80(4)
C12	0.2300(2)	0.0252(2)	0.5501(1)	3.26(3)
C13	0.1726(2)	-0.0950(2)	0.4888(2)	3.65(4)
C14	0.1331(2)	-0.0631(2)	0.3902(2)	3.46(4)
C15	-0.1554(2)	0.0756(2)	0.1633(2)	3.92(4)
C16	0.5040(3)	0.3251(2)	0.4916(2)	5.19(5)

$B_{eq} = 8/3\ \pi^2\ (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	Biso
H3A	-0.0421	0.1326	0.3507	1.00
H3B	0.0434	0.2495	0.3253	1.00
H5A	0.0904	0.2292	0.1456	1.00
H5B	0.0375	0.0959	0.0855	1.00
H7A	0.3359	-0.1809	0.2885	1.00
H7B	0.4419	-0.1071	0.2470	1.00
H8A	0.4745	0.0464	0.3599	1.00
H8B	0.3928	-0.043	0.4137	1.00
H9A	0.3921	0.2579	0.3062	1.00
H9B	0.2455	0.3058	0.3047	1.00
H11A	0.2093	0.3193	0.4889	1.00
H11B	0.3313	0.2570	0.5792	1.00
H13	0.1653	-0.1801	0.5154	1.000
H14	0.0922	-0.123	0.3359	1.000
H15A	-0.1844	0.0537	0.0944	1.00
H15B	-0.2151	0.0665	0.2003	1.00
H16A	0.5614	0.3392	0.4533	1.00
H16B	0.5346	0.3441	0.5610	1.00

Table 3. Anisotropic displacement parameters

atom	U11	U22	U33	U12	U13	U23
O1	0.075(1)	0.0710(8)	0.0331(7)	-0.0046(7)	0.0204(6)	0.0020(6)
O2	0.096(2)	0.0678(8)	0.0441(8)	0.0202(8)	0.0153(8)	-0.0169(6)
N1	0.0502(8)	0.0368(7)	0.0320(7)	-0.0052(6)	0.0182(6)	-0.0026(5)
N2	0.0425(8)	0.0408(7)	0.0314(7)	0.0062(6)	0.0166(6)	0.0006(5)
C1	0.0403(9)	0.0384(8)	0.0301(8)	0.0024(6)	0.0150(7)	0.0014(6)
C2	0.0427(9)	0.0331(7)	0.0292(8)	-0.0031(6)	0.0158(7)	-0.0010(6)
C3	0.0428(9)	0.0461(8)	0.0401(9)	0.0020(7)	0.0205(7)	-0.0008(7)
C4	0.0415(9)	0.0361(8)	0.0408(9)	0.0060(7)	0.0149(7)	0.0034(6)
C5	0.048(1)	0.0449(8)	0.0323(8)	0.0072(7)	0.0137(7)	0.0041(6)
C6	0.061(2)	0.0488(9)	0.039(1)	0.0085(8)	0.0221(9)	-0.0036(7)
C7	0.073(2)	0.066(2)	0.051(2)	0.029(1)	0.018(1)	-0.0029(8)
C8	0.048(1)	0.058(1)	0.044(1)	0.0119(8)	0.0188(8)	0.0043(7)
C9	0.049(1)	0.0444(9)	0.0428(9)	-0.0075(7)	0.0195(8)	0.0044(7)
C10	0.057(1)	0.0360(8)	0.046(1)	-0.0083(7)	0.0173(8)	-0.0006(6)
C11	0.064(2)	0.0394(8)	0.0428(9)	-0.0094(8)	0.0195(9)	-0.0094(7)
C12	0.047(1)	0.0492(9)	0.0324(9)	-0.0009(7)	0.0192(7)	0.0025(6)
C13	0.060(1)	0.0391(8)	0.044(1)	-0.0043(8)	0.0240(8)	0.0066(7)
C14	0.059(1)	0.0373(8)	0.0390(9)	-0.0072(7)	0.0222(8)	-0.0025(7)
C15	0.047(1)	0.052(1)	0.048(1)	0.0004(8)	0.0131(9)	0.0033(8)
C16	0.066(2)	0.068(2)	0.058(2)	-0.024(1)	0.014(1)	-0.000(1)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O1	C12	1.2294(19)	O2	C6	1.224(2)
N1	C2	1.4542(18)	N1	C11	1.458(2)
N1	C12	1.359(2)	N2	C1	1.4697(18)
N2	C5	1.4650(19)	N2	C6	1.355(3)
C1	C2	1.565(3)	C1	C8	1.535(3)
C1	C9	1.540(2)	C2	C3	1.543(2)
C2	C14	1.504(2)	C3	C4	1.507(2)
C4	C5	1.506(3)	C4	C15	1.322(3)
C6	C7	1.502(3)	C7	C8	1.495(3)
C9	C10	1.501(3)	C10	C11	1.506(3)
C10	C16	1.320(3)	C12	C13	1.489(2)
C13	C14	1.332(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C3	H3A	0.97	C3	H3B	0.97
C5	H5A	0.97	C5	H5B	0.97
C7	H7A	0.97	C7	H7B	0.97
C8	H8A	0.97	C8	H8B	0.97
C9	H9A	0.97	C9	H9B	0.97
C11	H11A	0.97	C11	H11B	0.97
C13	H13	0.9500(17)	C14	H14	0.9500(15)
C15	H15A	0.93	C15	H15B	0.93
C16	H16A	0.93	C16	H16B	0.93

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	N1	C11	120.98(13)	C2	N1	C12	113.21(11)
C11	N1	C12	124.96(12)	C1	N2	C5	120.39(13)
C1	N2	C6	114.46(12)	C5	N2	C6	122.59(12)
N2	C1	C2	108.54(12)	N2	C1	C8	102.68(13)
N2	C1	C9	111.65(12)	C2	C1	C8	113.76(13)
C2	C1	C9	108.90(13)	C8	C1	C9	111.20(13)
N1	C2	C1	109.77(12)	N1	C2	C3	111.15(13)
N1	C2	C14	101.18(11)	C1	C2	C3	110.39(12)
C1	C2	C14	113.37(14)	C3	C2	C14	110.68(13)
C2	C3	C4	112.05(14)	C3	C4	C5	114.17(13)
C3	C4	C15	123.52(18)	C5	C4	C15	122.30(16)
N2	C5	C4	108.98(13)	O2	C6	N2	124.80(15)
O2	C6	C7	127.23(17)	N2	C6	C7	107.96(14)
C6	C7	C8	106.34(16)	C1	C8	C7	106.74(13)
C1	C9	C10	111.45(13)	C9	C10	C11	114.36(14)
C9	C10	C16	123.9(2)	C11	C10	C16	121.78(17)
N1	C11	C10	109.77(14)	O1	C12	N1	125.67(14)
O1	C12	C13	128.46(15)	N1	C12	C13	105.86(13)
C12	C13	C14	108.60(14)	C2	C14	C13	111.14(13)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C3	H3A	109.9	C2	C3	H3B	109.9
C4	C3	H3A	107.8	C4	C3	H3B	107.8
H3A	C3	H3B	109.4	N2	C5	H5A	108.4
N2	C5	H5B	108.4	C4	C5	H5A	110.8
C4	C5	H5B	110.8	H5A	C5	H5B	109.4
C6	C7	H7A	110.4	C6	C7	H7B	110.4
C8	C7	H7A	110.1	C8	C7	H7B	110.1
H7A	C7	H7B	109.5	C1	C8	H8A	111.2
C1	C8	H8B	111.2	C7	C8	H8A	109.1
C7	C8	H8B	109.1	H8A	C8	H8B	109.4
C1	C9	H9A	110.1	C1	C9	H9B	110.1
C10	C9	H9A	107.8	C10	C9	H9B	107.8
H9A	C9	H9B	109.4	N1	C11	H11A	108
N1	C11	H11B	108	C10	C11	H11A	110.8
C10	C11	H11B	110.8	H11A	C11	H11B	109.4
C12	C13	H13	125.70(16)	C14	C13	H13	125.70(16)
C2	C14	H14	124.43(15)	C13	C14	H14	124.43(16)
C4	C15	H15A	120	C4	C15	H15B	120
H15A	C15	H15B	120	C10	C16	H16A	120
C10	C16	H16B	120	H16A	C16	H16B	120

Table 8. Torsion Angles(º)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C2	N1	C11	C10	-47.56(18)	C11	N1	C2	C1	51.07(18)
C11	N1	C2	C3	-71.34(19)	C11	N1	C2	C14	171.11(14)
C2	N1	C12	O1	178.51(16)	C2	N1	C12	C13	-0.92(19)
C12	N1	C2	C1	-118.81(14)	C12	N1	C2	C3	118.78(14)
C12	N1	C2	C14	1.23(18)	C11	N1	C12	O1	9.1(3)
C11	N1	C12	C13	-170.32(15)	C12	N1	C11	C10	121.07(17)
C1	N2	C5	C4	-52.01(16)	C5	N2	C1	C2	53.49(16)
C5	N2	C1	C8	174.22(12)	C5	N2	C1	C9	-66.56(18)
C1	N2	C6	O2	173.44(16)	C1	N2	C6	C7	-5.5(2)
C6	N2	C1	C2	-108.84(15)	C6	N2	C1	C8	11.89(18)
C6	N2	C1	C9	131.12(14)	C5	N2	C6	O2	11.6(3)
C5	N2	C6	C7	-167.34(13)	C6	N2	C5	C4	108.83(17)
N2	C1	C2	N1	-173.49(9)	N2	C1	C2	C3	-50.62(13)
N2	C1	C2	C14	74.19(12)	N2	C1	C8	C7	-13.30(17)
N2	C1	C9	C10	175.53(13)	C2	C1	C8	C7	103.77(14)
C8	C1	C2	N1	72.89(14)	C8	C1	C2	C3	-164.24(10)
C8	C1	C2	C14	-39.43(14)	C2	C1	C9	C10	55.69(15)
C9	C1	C2	N1	-51.74(13)	C9	C1	C2	C3	71.13(12)
C9	C1	C2	C14	-164.06(10)	C8	C1	C9	C10	-70.43(18)
C9	C1	C8	C7	-132.84(14)	N1	C2	C3	C4	175.26(12)
N1	C2	C14	C13	-1.12(18)	C1	C2	C3	C4	53.21(15)
C1	C2	C14	C13	116.33(14)	C3	C2	C14	C13	-119.01(14)
C14	C2	C3	C4	-73.13(17)	C2	C3	C4	C5	-53.50(16)

C2	C3	C4	C15	125.44(15)	C3	C4	C5	N2	49.25(16)
C15	C4	C5	N2	-129.69(15)	O2	C6	C7	C8	177.42(19)
N2	C6	C7	C8	-3.7(2)	C6	C7	C8	C1	10.8(2)
C1	C9	C10	C11	-55.02(18)	C1	C9	C10	C16	125.00(15)
C9	C10	C11	N1	47.28(16)	C16	C10	C11	N1	-132.74(16)
O1	C12	C13	C14	-179.26(18)	N1	C12	C13	C14	0.1(2)
C12	C13	C14	C2	0.6(3)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	C2	3.5314(18)	O1	C11	2.923(2)
O1	C14	3.482(2)	O2	C1	3.5439(19)
O2	C5	2.861(3)	O2	C8	3.591(2)
N1	C8	3.142(3)	N1	C9	2.839(3)
N1	C16	3.534(3)	N2	C3	2.843(3)
N2	C14	3.116(3)	N2	C15	3.518(3)
C1	C4	2.949(2)	C1	C11	2.982(3)
C1	C12	3.442(3)	C1	C13	3.461(3)
C1	C16	3.592(3)	C2	C5	2.984(2)
C2	C6	3.361(3)	C2	C7	3.438(3)
C2	C10	2.924(3)	C3	C9	3.102(3)
C3	C11	3.179(2)	C3	C12	3.442(2)
C3	C13	3.430(2)	C4	C6	3.405(3)
C4	C14	3.124(2)	C5	C9	3.151(2)
C6	C9	3.556(3)	C6	C14	3.492(3)
C7	C14	3.359(4)	C8	C10	3.107(3)
C8	C13	3.522(3)	C8	C14	2.935(3)
C10	C12	3.524(3)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H11A	3.507	O1	H11B	2.531
O1	H13	2.8148(12)	O2	H5A	3.552
O2	H5B	2.458	O2	H7A	2.764
O2	H7B	2.738	N1	H3A	2.716
N1	H3B	2.65	N1	H8B	2.833
N1	H9B	3.149	N1	H13	3.1866(12)
N1	H14	3.2065(12)	N2	H3B	3.187
N2	H7A	2.933	N2	H7B	2.978
N2	H8A	2.906	N2	H8B	3.081
N2	H9A	2.734	N2	H9B	2.672
N2	H14	3.0721(14)	C1	H3A	3.39
C1	H3B	2.787	C1	H5A	2.816
C1	H5B	3.331	C1	H7A	3.002
C1	H7B	3.143	C1	H11A	3.423
C1	H14	2.9608(16)	C2	H5A	3.39
C2	H8A	3.333	C2	H8B	2.589
C2	H9A	3.374	C2	H9B	2.74
C2	H11A	2.833	C2	H11B	3.307
C2	H13	3.2599(14)	C3	H5A	2.819
C3	H5B	3.353	C3	H9B	2.792
C3	H11A	2.976	C3	H14	2.8793(16)
C3	H15A	3.333	C3	H15B	2.658
C4	H9B	3.295	C4	H14	3.0384(14)
C5	H3A	3.336	C5	H3B	2.762

C5	H9A	3.509	C5	H9B	2.845
C5	H15A	2.634	C5	H15B	3.322
C6	H5A	3.126	C6	H5B	2.52
C6	H8A	2.965	C6	H8B	3.106
C6	H9A	3.586	C6	H14	3.185(2)
C7	H14	3.236(3)	C8	H9A	2.66
C8	H9B	3.368	C8	H14	3.248(2)
C9	H3B	2.816	C9	H5A	2.912
C9	H8A	2.515	C9	H8B	3.108
C9	H11A	2.832	C9	H11B	3.346
C9	H16A	2.658	C9	H16B	3.329
C10	H3B	3.326	C10	H8A	2.958
C10	H8B	3.291	C11	H3A	3.55
C11	H3B	2.879	C11	H8B	3.599
C11	H9A	3.334	C11	H9B	2.748
C11	H16A	3.316	C11	H16B	2.622
C12	H3A	3.403	C12	H8B	3.003
C12	H11A	3.081	C12	H11B	2.543
C12	H14	3.2114(15)	C13	H3A	3.326
C13	H8B	2.849	C14	H3A	2.613
C14	H3B	3.331	C14	H7A	3.126
C14	H8B	2.601	C15	H3A	2.54
C15	H3B	3.049	C15	H5A	3.056
C15	H5B	2.572	C15	H14	3.4985(18)
C16	H8A	3.319	C16	H9A	2.54
C16	H9B	3.049	C16	H11A	3.032
C16	H11B	2.566	H3A	H11A	3.268

H3A	H14	2.973	H3A	H15A	3.464
H3A	H15B	2.35	H3B	H5A	2.706
H3B	H9B	2.273	H3B	H11A	2.451
H3B	H15B	3.238	H5A	H9A	3.185
H5A	H9B	2.386	H5A	H15A	3.22
H5B	H15A	2.375	H5B	H15B	3.495
H7A	H8A	2.714	H7A	H8B	2.153
H7A	H14	2.866	H7B	H8A	2.151
H7B	H8B	2.613	H8A	H9A	2.331
H8A	H9B	3.444	H8A	H16A	3.237
H8B	H9A	3.387	H8B	H13	3.414
H8B	H14	3.038	H9A	H16A	2.353
H9A	H16B	3.464	H9B	H11A	2.71
H9B	H16A	3.247	H11A	H16B	3.181
H11B	H16A	3.488	H11B	H16B	2.367
H13	H14	2.42365(17)			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance
O1	C7[1]	3.568(3)
O1	C9[2]	3.386(2)
O2	C13[4]	3.474(2)
C4	C14[6]	3.515(2)
C8	O1[1]	3.447(3)
C13	O2[8]	3.474(2)
C15	O1[3]	3.517(3)
C16	C16[10]	3.545(3)

atom	atom	distance
O1	C8[1]	3.447(3)
O1	C15[3]	3.517(3)
O2	C15[5]	3.471(3)
C7	O1[1]	3.568(3)
C9	O1[7]	3.386(2)
C14	C4[9]	3.515(2)
C15	O2[5]	3.471(3)

Symmetry Operators

(1)	-X+1,-Y,-Z+1	(2)	X,-Y+1/2,Z+1/2
(3)	-X,-Y,-Z+1	(4)	X,-Y+1/2-1,Z+1/2-1
(5)	-X,-Y,-Z	(6)	-X,Y+1/2,-Z+1/2
(7)	X,-Y+1/2,Z+1/2-1	(8)	X,-Y+1/2-1,Z+1/2
(9)	-X,Y+1/2-1,-Z+1/2	(10)	-X+1,-Y+1,-Z+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H3A[1]	3.001	O1	H5A[2]	3.072
O1	H7B[3]	2.874	O1	H8A[3]	2.701
O1	H9A[2]	3.01	O1	H9B[2]	2.862
O1	H15B[1]	2.676	O2	H3A[4]	3.411
O2	H3B[4]	3.415	O2	H5B[5]	2.823
O2	H11A[6]	3.141	O2	H13[7]	2.5358(13)
O2	H15A[5]	2.618	O2	H16A[8]	2.91
N1	H5A[2]	3.353	N2	H11A[6]	3.304
N2	H11B[6]	3.132	C3	H13[1]	3.4986(19)
C3	H14[9]	3.0814(15)	C4	H13[9]	3.5868(14)
C4	H14[9]	2.7042(15)	C5	H11A[6]	3.09
C5	H11B[6]	3.348	C5	H13[9]	3.3312(14)
C5	H14[9]	3.0008(17)	C6	H11A[6]	3.401
C6	H11B[6]	3.447	C6	H13[7]	3.5334(16)
C6	H16A[8]	3.365	C7	H9A[8]	3.381
C7	H11B[3]	3.579	C7	H16A[8]	3.471
C7	H16B[3]	3.383	C8	H8B[3]	3.324
C9	H7B[10]	3.388	C9	H15B[9]	3.411
C10	H8B[3]	3.454	C10	H15A[9]	3.368
C11	H5A[2]	3.204	C11	H5B[2]	3.431
C11	H15A[9]	3.348	C12	H3A[1]	3.161
C12	H5A[2]	3.359	C12	H8A[3]	2.977
C13	H3A[1]	2.989	C13	H5A[4]	3.264
C13	H8A[3]	3.586	C13	H16A[3]	3.581
C14	H3B[4]	3.5	C14	H5A[4]	3.038
C15	H3B[4]	3.48	C15	H7A[9]	3.289

C15	H9B[4]	2.963	C15	H11A[4]	3.272
C15	H13[9]	3.480(2)	C15	H14[9]	3.1153(18)
C15	H16A[11]	3.484	C15	H16B[11]	3.148
C16	H7A[3]	3.292	C16	H8B[3]	3.174
C16	H15A[12]	3.294	C16	H15B[12]	3.526
C16	H16A[13]	3.592	C16	H16B[13]	3.418
H3A	O1[1]	3.001	H3A	O2[9]	3.411
H3A	C12[1]	3.161	H3A	C13[1]	2.989
H3A	H7A[9]	3.55	H3A	H13[1]	2.623
H3A	H14[9]	3.493	H3B	O2[9]	3.415
H3B	C14[9]	3.5	H3B	C15[9]	3.48
H3B	H14[9]	2.56	H3B	H15A[9]	3.427
H5A	O1[6]	3.072	H5A	N1[6]	3.353
H5A	C11[6]	3.204	H5A	C12[6]	3.359
H5A	C13[9]	3.264	H5A	C14[9]	3.038
H5A	H11A[6]	2.877	H5A	H11B[6]	2.932
H5A	H13[9]	2.983	H5A	H14[9]	2.487
H5B	O2[5]	2.823	H5B	C11[6]	3.431
H5B	H5B[5]	2.963	H5B	H11A[6]	2.698
H5B	H11B[6]	3.408	H5B	H13[9]	3.087
H5B	H14[9]	3.47	H7A	C15[4]	3.289
H7A	C16[3]	3.292	H7A	H3A[4]	3.55
H7A	H9A[8]	3.532	H7A	H11B[3]	3.404
H7A	H15B[4]	2.869	H7A	H16B[3]	2.649
H7B	O1[3]	2.874	H7B	C9[8]	3.388
H7B	H9A[8]	2.489	H7B	H11B[3]	3.139
H7B	H16A[8]	2.825	H7B	H16B[3]	3.532
H8A	O1[3]	2.701	H8A	C12[3]	2.977
H8A	C13[3]	3.586	H8A	H8B[3]	2.984

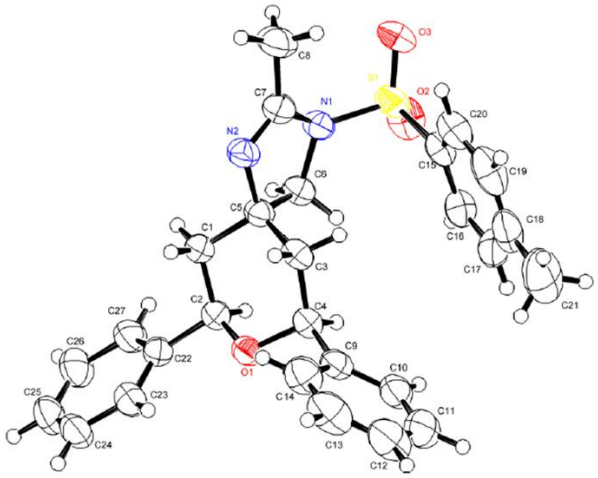
H8B	C8[3]	3.324	H8B	C10[3]	3.454
H8B	C16[3]	3.174	H8B	H8A[3]	2.984
H8B	H8B[3]	2.806	H8B	H11B[3]	3.554
H8B	H16A[3]	3.467	H8B	H16B[3]	3.127
H9A	O1[6]	3.01	H9A	C7[10]	3.381
H9A	H7A[10]	3.532	H9A	H7B[10]	2.489
H9A	H11B[6]	3.008	H9B	O1[6]	2.862
H9B	C15[9]	2.963	H9B	H11B[6]	3.585
H9B	H14[9]	3.46	H9B	H15A[9]	3.039
H9B	H15B[9]	2.652	H11A	O2[2]	3.141
H11A	N2[2]	3.304	H11A	C5[2]	3.09
H11A	C6[2]	3.401	H11A	C15[9]	3.272
H11A	H5A[2]	2.877	H11A	H5B[2]	2.698
H11A	H15A[9]	2.611	H11B	N2[2]	3.132
H11B	C5[2]	3.348	H11B	C6[2]	3.447
H11B	C7[3]	3.579	H11B	H5A[2]	2.932
H11B	H5B[2]	3.408	H11B	H7A[3]	3.404
H11B	H7B[3]	3.139	H11B	H8B[3]	3.554
H11B	H9A[2]	3.008	H11B	H9B[2]	3.585
H13	O2[14]	2.5358(13)	H13	C3[1]	3.4986(19)
H13	C4[4]	3.5868(14)	H13	C5[4]	3.3312(14)
H13	C6[14]	3.5334(16)	H13	C15[4]	3.480(2)
H13	H3A[1]	2.623	H13	H5A[4]	2.983
H13	H5B[4]	3.087	H13	H15A[4]	3.132
H13	H16A[3]	3.152	H14	C3[4]	3.0814(15)
H14	C4[4]	2.7042(15)	H14	C5[4]	3.0008(17)
H14	C15[4]	3.1153(18)	H14	H3A[4]	3.493
H14	H3B[4]	2.56	H14	H5A[4]	2.487
H14	H5B[4]	3.47	H14	H9B[4]	3.46

H14	H15A[4]	3.451	H14	H15B[4]	3.485
H15A	O2[5]	2.618	H15A	C10[4]	3.368
H15A	C11[4]	3.348	H15A	C16[11]	3.294
H15A	H3B[4]	3.427	H15A	H9B[4]	3.039
H15A	H11A[4]	2.611	H15A	H13[9]	3.132
H15A	H14[9]	3.451	H15A	H16A[11]	2.913
H15A	H16B[11]	2.969	H15B	O1[1]	2.676
H15B	C9[4]	3.411	H15B	C16[11]	3.526
H15B	H7A[9]	2.869	H15B	H9B[4]	2.652
H15B	H14[9]	3.485	H15B	H16A[11]	3.57
H15B	H16B[11]	2.814	H16A	O2[10]	2.91
H16A	C6[10]	3.365	H16A	C7[10]	3.471
H16A	C13[3]	3.581	H16A	C15[12]	3.484
H16A	C16[13]	3.592	H16A	H7B[10]	2.825
H16A	H8B[3]	3.467	H16A	H13[3]	3.152
H16A	H15A[12]	2.913	H16A	H15B[12]	3.57
H16A	H16B[13]	3.338	H16B	C7[3]	3.383
H16B	C15[12]	3.148	H16B	C16[13]	3.418
H16B	H7A[3]	2.649	H16B	H7B[3]	3.532
H16B	H8B[3]	3.127	H16B	H15A[12]	2.969
H16B	H15B[12]	2.814	H16B	H16A[13]	3.338
H16B	H16B[13]	3.538			

Symmetry Operators

(1)	-X,-Y,-Z+1	(2)	X,-Y+1/2,Z+1/2
(3)	-X+1,-Y,-Z+1	(4)	-X,Y+1/2-1,-Z+1/2
(5)	-X,-Y,-Z	(6)	X,-Y+1/2,Z+1/2-1
(7)	X,-Y+1/2-1,Z+1/2-1	(8)	-X+1,Y+1/2-1,-Z+1/2
(9)	-X,Y+1/2,-Z+1/2	(10)	-X+1,Y+1/2,-Z+1/2
(11)	X-1,-Y+1/2,Z+1/2-1	(12)	X+1,-Y+1/2,Z+1/2
(13)	-X+1,-Y+1,-Z+1	(14)	X,-Y+1/2-1,Z+1/2

3) Crystallographic Data for **14**



A. Crystal Data

Empirical Formula	$C_{27}H_{28}N_2O_3S$
Formula Weight	460.59
Crystal Color, Habit	colorless, chunk
Crystal Dimensions	0.300 X 0.300 X 0.300 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 18.6366(8) Å b = 12.5881(8) Å c = 10.2431(5) Å β = 94.9576(12)° V = 2394.0(2) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.278 g/cm^3
F_{000}	976.00
μ(MoKα)	1.664 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	23.0°C
Detector Aperture	460.0 x 256.0 mm
Data Images	44 exposures
w oscillation Range (c=45.0, f=90.0)	130.0 - 190.0°
Exposure Rate	90.0 sec./°
w oscillation Range (c=45.0, f=270.0)	0.0 - 160.0°
Exposure Rate	90.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2q_{max}$	54.9°
No. of Reflections Measured	Total: 23098 Unique: 5467 (R_{int} = 0.0329)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.722 - 0.951)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR2008)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(F_o^2) + (0.0548 \cdot P)^2 + 0.3518 \cdot P]$ where $P = (Max(F_o^2,0) + 2F_c^2)/3$
$2\theta_{max}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5467
No. Variables	300
Reflection/Parameter Ratio	18.22
Residuals: R1 (I>2.00σ(I))	0.0421
Residuals: R (All reflections)	0.0680
Residuals: wR2 (All reflections)	0.1229
Goodness of Fit Indicator	1.098
Max Shift/Error in Final Cycle	0.003
Maximum peak in Final Diff. Map	0.19 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.35 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	Beq
S1	0.66242(2)	0.16264(4)	0.48218(4)	3.979(11)
O1	0.84267(6)	0.07308(9)	1.00333(10)	3.61(2)
O2	0.64469(7)	0.26895(10)	0.51618(15)	5.18(3)
O3	0.65465(8)	0.13001(13)	0.34849(12)	5.59(3)
N1	0.74761(7)	0.14622(11)	0.53538(13)	3.58(3)
N2	0.83694(8)	0.02880(12)	0.59641(13)	3.75(3)
C1	0.88963(9)	0.11949(14)	0.79703(15)	3.41(3)
C2	0.87356(8)	0.15746(13)	0.93283(15)	3.26(3)
C3	0.78294(9)	0.00235(13)	0.80069(15)	3.41(3)
C4	0.77470(9)	0.03885(13)	0.94095(16)	3.33(3)
C5	0.82074(8)	0.08321(13)	0.71864(15)	3.21(3)
C6	0.77231(9)	0.17719(13)	0.67128(16)	3.47(3)
C7	0.79332(9)	0.06023(14)	0.50270(16)	3.75(3)
C8	0.79296(12)	0.01914(19)	0.36605(18)	5.37(5)
C9	0.74735(9)	-0.04933(14)	1.02309(16)	3.68(3)
C10	0.68146(10)	-0.04114(16)	1.07433(18)	4.47(4)
C11	0.65647(12)	-0.1235(2)	1.1488(2)	5.97(5)
C12	0.69710(15)	-0.2129(2)	1.1725(3)	6.71(6)
C13	0.76302(15)	-0.22229(18)	1.1211(3)	6.35(5)
C14	0.78801(12)	-0.14070(16)	1.0473(2)	5.00(4)
C15	0.61371(9)	0.07646(13)	0.57602(17)	3.65(3)
C16	0.59336(9)	0.11041(16)	0.69645(19)	4.26(4)

C17	0.56031(10)	0.04056(19)	0.7749(2)	5.15(4)
C18	0.54622(10)	-0.0627(2)	0.7372(3)	5.56(5)
C19	0.56455(11)	-0.09424(16)	0.6153(3)	5.84(5)
C20	0.59919(10)	-0.02622(15)	0.5343(2)	4.71(4)
C21	0.51204(15)	-0.1401(3)	0.8262(3)	9.19(9)
C22	0.94022(9)	0.19682(14)	1.01232(16)	3.43(3)
C23	0.97610(9)	0.13705(15)	1.11101(18)	4.10(3)
C24	1.03779(10)	0.17678(18)	1.1806(2)	4.76(4)
C25	1.06378(10)	0.27461(19)	1.1506(2)	5.26(4)
C26	1.02888(11)	0.33448(18)	1.0521(2)	5.24(4)
C27	0.96745(10)	0.29581(16)	0.98410(18)	4.30(4)

$B_{eq} = 8/3\ \pi^2\ (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1A	0.91135	0.17681	0.75090	4.092
H1B	0.92365	0.06107	0.80551	4.092
H2	0.83873	0.21582	0.92263	3.910
H3A	0.73554	-0.01232	0.75773	4.087
H3B	0.81007	-0.06349	0.80364	4.087
H4	0.74067	0.09831	0.93878	3.996
H6A	0.73203	0.18502	0.72447	4.163
H6B	0.79933	0.24311	0.67309	4.163
H8A	0.74995	-0.02151	0.34482	6.446
H8B	0.83434	-0.02525	0.35907	6.446
H8C	0.79436	0.07773	0.30633	6.446
H10	0.65367	0.01967	1.05898	5.360
H11	0.61192	-0.11763	1.18272	7.160
H12	0.68041	-0.26741	1.22309	8.046
H13	0.79046	-0.28347	1.13630	7.622
H14	0.83254	-0.147	1.01345	5.995
H16	0.60206	0.18004	0.72376	5.117
H17	0.54711	0.06366	0.85569	6.177
H19	0.55339	-0.16285	0.58659	7.013
H20	0.61235	-0.04941	0.45352	5.658
H21A	0.46329	-0.11893	0.83566	11.033
H21B	0.53881	-0.14059	0.91056	11.033

H21C	0.51233	-0.21006	0.78879	11.033
H23	0.95893	0.07012	1.13087	4.925
H24	1.06135	0.13678	1.24756	5.717
H25	1.10517	0.30077	1.19685	6.312
H26	1.04674	0.40084	1.03157	6.283
H27	0.94385	0.33680	0.91813	5.165

Table 3. Anisotropic displacement parameters

atom	U11	U22	U33	U12	U13	U23
S1	0.0508(3)	0.0517(3)	0.0465(3)	0.0027(2)	-0.00809(19)	0.0091(2)
O1	0.0462(6)	0.0510(7)	0.0388(6)	-0.0086(5)	-0.0034(5)	0.0016(5)
O2	0.0667(8)	0.0424(7)	0.0861(10)	0.0080(6)	-0.0029(7)	0.0170(7)
O3	0.0673(9)	0.0994(11)	0.0424(7)	0.0036(8)	-0.0146(6)	0.0056(7)
N1	0.0470(8)	0.0505(9)	0.0375(7)	0.0006(6)	-0.0031(6)	-0.0005(6)
N2	0.0489(8)	0.0531(9)	0.0394(8)	0.0034(6)	-0.0012(6)	-0.0081(6)
C1	0.0410(8)	0.0495(10)	0.0387(9)	0.0003(7)	0.0009(7)	-0.0032(7)
C2	0.0403(8)	0.0429(9)	0.0402(8)	-0.0015(7)	0.0015(7)	-0.0018(7)
C3	0.0440(9)	0.0401(9)	0.0436(9)	-0.0009(7)	-0.0060(7)	-0.0024(7)
C4	0.0411(8)	0.0408(9)	0.0437(9)	-0.0010(7)	-0.0022(7)	0.0015(7)
C5	0.0431(8)	0.0435(9)	0.0346(8)	0.0013(7)	-0.0006(7)	-0.0050(7)
C6	0.0470(9)	0.0435(9)	0.0404(9)	-0.0018(7)	-0.0016(7)	-0.0024(7)
C7	0.0481(9)	0.0525(10)	0.0415(9)	-0.0032(8)	0.0007(8)	-0.0070(8)
C8	0.0710(13)	0.0872(16)	0.0440(11)	0.0090(11)	-0.0058(9)	-0.0163(10)
C9	0.0504(10)	0.0472(10)	0.0404(9)	-0.0096(8)	-0.0064(8)	0.0032(7)
C10	0.0504(10)	0.0650(12)	0.0528(11)	-0.0106(9)	-0.0045(9)	0.0071(9)
C11	0.0614(13)	0.0939(18)	0.0703(14)	-0.0308(12)	-0.0009(11)	0.0161(13)
C12	0.0892(18)	0.0791(17)	0.0817(16)	-0.0396(14)	-0.0201(13)	0.0318(13)
C13	0.0936(18)	0.0560(13)	0.0867(16)	-0.0116(12)	-0.0214(14)	0.0241(12)
C14	0.0678(13)	0.0543(12)	0.0660(13)	0.0005(10)	-0.0041(10)	0.0137(10)
C15	0.0407(8)	0.0431(9)	0.0523(10)	0.0032(7)	-0.0099(8)	0.0024(8)
C16	0.0451(9)	0.0523(11)	0.0640(12)	0.0024(8)	0.0009(9)	-0.0019(9)

C17	0.0439(10)	0.0821(15)	0.0696(13)	-0.0029(10)	0.0047(9)	0.0081(11)
C18	0.0433(10)	0.0767(15)	0.0884(17)	-0.0115(10)	-0.0106(11)	0.0260(13)
C19	0.0607(12)	0.0441(12)	0.112(2)	-0.0095(9)	-0.0254(13)	0.0111(12)
C20	0.0566(11)	0.0504(11)	0.0688(13)	0.0031(9)	-0.0140(9)	-0.0063(9)
C21	0.0786(18)	0.125(2)	0.143(3)	-0.0380(16)	-0.0090(17)	0.063(2)
C22	0.0406(8)	0.0500(10)	0.0395(9)	-0.0004(7)	0.0024(7)	-0.0077(7)
C23	0.0476(10)	0.0537(11)	0.0533(10)	0.0024(8)	-0.0027(8)	-0.0035(8)
C24	0.0486(10)	0.0753(14)	0.0546(11)	0.0099(10)	-0.0102(9)	-0.0070(10)
C25	0.0473(10)	0.0822(16)	0.0681(13)	-0.0120(10)	-0.0075(10)	-0.0141(11)
C26	0.0564(11)	0.0710(14)	0.0698(13)	-0.0212(10)	-0.0044(10)	-0.0028(11)
C27	0.0526(10)	0.0602(12)	0.0496(10)	-0.0103(9)	-0.0023(8)	0.0016(9)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S1	O2	1.4287(14)	S1	O3	1.4250(13)
S1	N1	1.6464(13)	S1	C15	1.7538(18)
O1	C2	1.433(2)	O1	C4	1.4348(19)
N1	C6	1.480(2)	N1	C7	1.435(2)
N2	C5	1.481(2)	N2	C7	1.266(2)
C1	C2	1.525(2)	C1	C5	1.524(2)
C2	C22	1.509(2)	C3	C4	1.529(2)
C3	C5	1.530(2)	C4	C9	1.508(2)
C5	C6	1.541(2)	C7	C8	1.492(3)
C9	C10	1.380(3)	C9	C14	1.388(3)
C10	C11	1.391(3)	C11	C12	1.366(4)
C12	C13	1.383(4)	C13	C14	1.380(3)
C15	C16	1.389(3)	C15	C20	1.381(3)
C16	C17	1.373(3)	C17	C18	1.375(3)
C18	C19	1.381(4)	C18	C21	1.513(4)
C19	C20	1.389(3)	C22	C23	1.385(2)
C22	C27	1.385(3)	C23	C24	1.392(3)
C24	C25	1.368(3)	C25	C26	1.377(3)
C26	C27	1.377(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1A	0.97	C1	H1B	0.97
C2	H2	0.98	C3	H3A	0.97
C3	H3B	0.97	C4	H4	0.98
C6	H6A	0.97	C6	H6B	0.97
C8	H8A	0.96	C8	H8B	0.96
C8	H8C	0.96	C10	H10	0.93
C11	H11	0.93	C12	H12	0.93
C13	H13	0.93	C14	H14	0.93
C16	H16	0.93	C17	H17	0.93
C19	H19	0.93	C20	H20	0.93
C21	H21A	0.96	C21	H21B	0.96
C21	H21C	0.96	C23	H23	0.93
C24	H24	0.93	C25	H25	0.93
C26	H26	0.93	C27	H27	0.93

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O2	S1	O3	119.85(9)	O2	S1	N1	105.88(8)
O2	S1	C15	107.71(8)	O3	S1	N1	107.27(8)
O3	S1	C15	109.51(9)	N1	S1	C15	105.72(8)
C2	O1	C4	112.08(11)	S1	N1	C6	119.39(11)
S1	N1	C7	126.33(11)	C6	N1	C7	105.99(12)
C5	N2	C7	109.38(14)	C2	C1	C5	110.58(13)
O1	C2	C1	110.28(13)	O1	C2	C22	108.62(12)
C1	C2	C22	112.19(13)	C4	C3	C5	113.87(13)
O1	C4	C3	110.63(13)	O1	C4	C9	107.49(12)
C3	C4	C9	111.68(14)	N2	C5	C1	111.10(13)
N2	C5	C3	107.09(13)	N2	C5	C6	104.22(12)
C1	C5	C3	108.47(13)	C1	C5	C6	112.31(13)
C3	C5	C6	113.50(13)	N1	C6	C5	102.71(13)
N1	C7	N2	114.46(15)	N1	C7	C8	121.73(15)
N2	C7	C8	123.60(17)	C4	C9	C10	120.79(16)
C4	C9	C14	120.27(16)	C10	C9	C14	118.94(18)
C9	C10	C11	120.27(19)	C10	C11	C12	120.3(2)
C11	C12	C13	120.0(2)	C12	C13	C14	119.8(2)
C9	C14	C13	120.6(2)	S1	C15	C16	119.37(13)
S1	C15	C20	120.41(15)	C16	C15	C20	120.11(17)
C15	C16	C17	119.59(19)	C16	C17	C18	121.7(2)
C17	C18	C19	118.0(2)	C17	C18	C21	121.3(2)

C19	C18	C21	120.6(2)	C18	C19	C20	121.8(2)
C15	C20	C19	118.7(2)	C2	C22	C23	122.58(16)
C2	C22	C27	118.82(15)	C23	C22	C27	118.59(16)
C22	C23	C24	120.25(18)	C23	C24	C25	120.07(18)
C24	C25	C26	120.23(18)	C25	C26	C27	119.8(2)
C22	C27	C26	121.07(18)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C1	H1A	109.5	C2	C1	H1B	109.5
C5	C1	H1A	109.5	C5	C1	H1B	109.5
H1A	C1	H1B	108.1	O1	C2	H2	108.6
C1	C2	H2	108.6	C22	C2	H2	108.6
C4	C3	H3A	108.8	C4	C3	H3B	108.8
C5	C3	H3A	108.8	C5	C3	H3B	108.8
H3A	C3	H3B	107.7	O1	C4	H4	109
C3	C4	H4	109	C9	C4	H4	109
N1	C6	H6A	111.2	N1	C6	H6B	111.2
C5	C6	H6A	111.2	C5	C6	H6B	111.2
H6A	C6	H6B	109.1	C7	C8	H8A	109.5
C7	C8	H8B	109.5	C7	C8	H8C	109.5
H8A	C8	H8B	109.5	H8A	C8	H8C	109.5
H8B	C8	H8C	109.5	C9	C10	H10	119.9
C11	C10	H10	119.9	C10	C11	H11	119.9
C12	C11	H11	119.9	C11	C12	H12	120
C13	C12	H12	120	C12	C13	H13	120.1
C14	C13	H13	120.1	C9	C14	H14	119.7
C13	C14	H14	119.7	C15	C16	H16	120.2
C17	C16	H16	120.2	C16	C17	H17	119.2
C18	C17	H17	119.1	C18	C19	H19	119.1
C20	C19	H19	119.1	C15	C20	H20	120.6
C19	C20	H20	120.6	C18	C21	H21A	109.5

C18	C21	H21B	109.5	C18	C21	H21C	109.5
H21A	C21	H21B	109.5	H21A	C21	H21C	109.5
H21B	C21	H21C	109.5	C22	C23	H23	119.9
C24	C23	H23	119.9	C23	C24	H24	120
C25	C24	H24	120	C24	C25	H25	119.9
C26	C25	H25	119.9	C25	C26	H26	120.1
C27	C26	H26	120.1	C22	C27	H27	119.5
C26	C27	H27	119.5				

Table 8. Torsion Angles(º)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O2	S1	N1	C6	48.93(12)	O2	S1	N1	C7	-167.29(11)
O2	S1	C15	C16	-26.86(14)	O2	S1	C15	C20	156.78(11)
O3	S1	N1	C6	178.01(10)	O3	S1	N1	C7	-38.21(14)
O3	S1	C15	C16	-158.74(11)	O3	S1	C15	C20	24.90(15)
N1	S1	C15	C16	85.99(12)	N1	S1	C15	C20	-90.37(12)
C15	S1	N1	C6	-65.19(11)	C15	S1	N1	C7	78.59(12)
C2	O1	C4	C3	57.92(15)	C2	O1	C4	C9	-179.91(11)
C4	O1	C2	C1	-62.96(14)	C4	O1	C2	C22	173.72(11)
S1	N1	C6	C5	136.31(10)	S1	N1	C7	N2	-142.56(12)
S1	N1	C7	C8	42.6(2)	C6	N1	C7	N2	5.06(18)
C6	N1	C7	C8	-169.81(13)	C7	N1	C6	C5	-14.01(15)
C5	N2	C7	N1	7.11(19)	C5	N2	C7	C8	-178.13(13)
C7	N2	C5	C1	-136.89(14)	C7	N2	C5	C3	104.83(14)
C7	N2	C5	C6	-15.72(17)	C2	C1	C5	N2	-169.99(12)
C2	C1	C5	C3	-52.55(16)	C2	C1	C5	C6	73.72(16)
C5	C1	C2	O1	60.29(16)	C5	C1	C2	C22	-178.49(12)
O1	C2	C22	C23	19.5(2)	O1	C2	C22	C27	-161.73(12)
C1	C2	C22	C23	-102.71(17)	C1	C2	C22	C27	76.10(18)
C4	C3	C5	N2	169.34(11)	C4	C3	C5	C1	49.36(16)
C4	C3	C5	C6	-76.21(15)	C5	C3	C4	O1	-51.87(17)
C5	C3	C4	C9	-171.56(11)	O1	C4	C9	C10	122.03(14)
O1	C4	C9	C14	-58.57(18)	C3	C4	C9	C10	-116.45(15)
C3	C4	C9	C14	62.95(18)	N2	C5	C6	N1	17.60(14)
C1	C5	C6	N1	137.95(12)	C3	C5	C6	N1	-98.56(14)
C4	C9	C10	C11	179.34(13)	C4	C9	C14	C13	-179.26(14)
C10	C9	C14	C13	0.2(3)	C14	C9	C10	C11	-0.1(3)
C9	C10	C11	C12	0.3(3)	C10	C11	C12	C13	-0.7(3)

C11	C12	C13	C14	0.7(4)	C12	C13	C14	C9	-0.5(3)
S1	C15	C16	C17	-174.83(10)	S1	C15	C20	C19	175.89(11)
C16	C15	C20	C19	-0.4(2)	C20	C15	C16	C17	1.5(2)
C15	C16	C17	C18	-0.4(3)	C16	C17	C18	C19	-1.7(3)
C16	C17	C18	C21	177.93(15)	C17	C18	C19	C20	2.9(3)
C21	C18	C19	C20	-176.79(18)	C18	C19	C20	C15	-1.8(3)
C2	C22	C23	C24	179.41(14)	C2	C22	C27	C26	-178.77(14)
C23	C22	C27	C26	0.1(3)	C27	C22	C23	C24	0.6(3)
C22	C23	C24	C25	-0.8(3)	C23	C24	C25	C26	0.4(3)
C24	C25	C26	C27	0.3(3)	C25	C26	C27	C22	-0.5(3)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	C8	3.331(2)	O1	C5	2.9122(18)
O1	C10	3.464(2)	O1	C14	2.926(2)
O1	C23	2.752(2)	O2	C6	2.977(2)
O2	C16	2.935(2)	O3	C7	3.040(2)
O3	C8	2.923(3)	O3	C20	2.983(3)
N1	C3	3.285(2)	N1	C16	3.465(2)
N1	C20	3.515(2)	C1	C4	2.887(2)
C1	C7	3.454(2)	C1	C23	3.478(2)
C1	C27	3.197(3)	C2	C3	2.846(2)
C2	C6	3.151(2)	C3	C7	3.160(2)
C3	C10	3.557(3)	C3	C14	3.097(3)
C4	C6	3.262(2)	C6	C15	3.286(2)
C6	C16	3.470(2)	C7	C15	3.499(2)
C9	C12	2.776(3)	C10	C13	2.759(3)
C11	C14	2.753(3)	C15	C18	2.781(3)
C16	C19	2.746(3)	C17	C20	2.760(3)
C22	C25	2.776(3)	C23	C26	2.759(3)
C24	C27	2.749(3)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H6A	2.714	S1	H6B	3.24
S1	H8A	3.227	S1	H8C	3.346
S1	H16	2.814	S1	H20	2.835
O1	H1A	3.256	O1	H1B	2.633
O1	H3A	3.256	O1	H3B	2.701
O1	H14	2.779	O1	H23	2.432
O2	H6A	2.778	O2	H6B	3.193
O2	H16	2.588	O3	H8A	2.609
O3	H8C	2.756	O3	H20	2.651
N1	H3A	3.051	N1	H8A	2.878
N1	H8B	3.322	N1	H8C	2.713
N1	H16	3.488	N1	H20	3.572
N2	H1A	2.742	N2	H1B	2.601
N2	H3A	2.666	N2	H3B	2.508
N2	H6A	3.139	N2	H6B	2.912
N2	H8A	2.991	N2	H8B	2.521
N2	H8C	3.071	C1	H3A	3.311
C1	H3B	2.743	C1	H4	3.253
C1	H6A	3.079	C1	H6B	2.549
C1	H27	3.135	C2	H3B	3.258
C2	H4	2.592	C2	H6A	3.265
C2	H6B	3.085	C2	H23	2.701
C2	H27	2.621	C3	H1A	3.32
C3	H1B	2.721	C3	H2	3.104

C3	H6A	2.582	C3	H6B	3.325
C3	H14	2.965	C4	H1B	3.221
C4	H2	2.542	C4	H6A	2.938
C4	H10	2.661	C4	H14	2.655
C5	H2	2.673	C5	H4	2.816
C6	H1A	2.648	C6	H1B	3.363
C6	H2	2.801	C6	H3A	2.655
C6	H3B	3.369	C6	H4	3.02
C6	H16	3.264	C7	H1A	3.534
C7	H3A	3.05	C7	H3B	3.444
C7	H6A	3.063	C7	H6B	2.885
C9	H3A	2.748	C9	H3B	2.628
C9	H11	3.24	C9	H13	3.243
C10	H3A	3.497	C10	H4	2.548
C10	H12	3.231	C10	H14	3.224
C10	H17	3.472	C10	H21B	3.268
C11	H13	3.219	C11	H21B	3.144
C12	H10	3.227	C12	H14	3.228
C13	H11	3.22	C14	H3A	3.444
C14	H3B	2.742	C14	H4	3.301
C14	H10	3.226	C14	H12	3.23
C15	H3A	3.023	C15	H6A	2.909
C15	H17	3.223	C15	H19	3.22
C16	H3A	3.085	C16	H4	3.542
C16	H6A	2.741	C16	H20	3.243
C17	H3A	3.352	C17	H10	3.269
C17	H19	3.202	C17	H21A	2.807

C17	H21B	2.718	C17	H21C	3.286
C18	H3A	3.573	C18	H16	3.235
C18	H20	3.257	C19	H3A	3.542
C19	H17	3.203	C19	H21A	3.081
C19	H21B	3.158	C19	H21C	2.556
C20	H3A	3.274	C20	H8A	3.551
C20	H16	3.239	C21	H17	2.658
C21	H19	2.65	C22	H1A	2.697
C22	H1B	2.718	C22	H24	3.245
C22	H26	3.242	C23	H1B	3.337
C23	H2	3.226	C23	H25	3.231
C23	H27	3.222	C24	H26	3.218
C25	H23	3.228	C25	H27	3.218
C26	H24	3.219	C27	H1A	2.934
C27	H1B	3.534	C27	H2	2.628
C27	H23	3.225	C27	H25	3.221
H1A	H2	2.362	H1A	H6A	3.331
H1A	H6B	2.324	H1A	H27	2.679
H1B	H2	2.84	H1B	H3B	2.633
H1B	H6B	3.452	H1B	H23	3.343
H2	H4	2.368	H2	H6A	2.744
H2	H6B	2.62	H2	H23	3.479
H2	H27	2.485	H3A	H4	2.315
H3A	H6A	2.507	H3A	H6B	3.561
H3A	H10	3.581	H3A	H14	3.491
H3A	H16	3.467	H3B	H4	2.837
H3B	H6A	3.515	H3B	H14	2.397

H4	H6A	2.444	H4	H6B	3.527
H4	H10	2.339	H4	H14	3.581
H4	H16	3.406	H6A	H16	2.422
H8A	H20	2.904	H10	H11	2.317
H10	H17	2.806	H10	H21B	3.225
H11	H12	2.294	H11	H21B	3.009
H12	H13	2.314	H13	H14	2.307
H16	H17	2.294	H17	H21A	2.777
H17	H21B	2.639	H17	H21C	3.562
H19	H20	2.316	H19	H21A	3.221
H19	H21B	3.365	H19	H21C	2.346
H23	H24	2.32	H24	H25	2.296
H25	H26	2.307	H26	H27	2.3

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O2	C4[1]	3.555(2)	O2	C10[1]	3.535(2)
O3	C21[2]	3.451(3)	N2	C27[1]	3.549(2)
C4	O2[3]	3.555(2)	C8	C10[4]	3.571(3)
C10	O2[3]	3.535(2)	C10	C8[5]	3.571(3)
C12	C19[6]	3.477(3)	C19	C12[7]	3.477(3)
C21	O3[2]	3.451(3)	C24	C27[3]	3.494(3)
C27	N2[3]	3.549(2)	C27	C24[1]	3.494(3)

Symmetry Operators

(1) X,-Y+1,Z (2) -X+1,-Y,-Z+1
(3) X,-Y+1,Z+1 (4) X,Y,Z-1
(5) X,Y,Z+1 (6) X,-Y,Z+1
(7) X,-Y,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
S1	H4[1]	3.39	S1	H6A[1]	3.595
S1	H16[1]	3.417	O1	H6B[2]	3.045
O1	H8C[3]	3.306	O2	H4[1]	2.62
O2	H6A[1]	3.571	O2	H10[1]	2.7
O2	H16[1]	3.099	O2	H17[1]	3.152
O2	H21A[4]	2.979	O3	H6A[1]	3.071
O3	H10[5]	3.273	O3	H16[1]	2.845
O3	H21A[6]	2.773	O3	H21C[6]	3.453
N1	H2[1]	2.752	N1	H4[1]	3.364
N2	H13[7]	3.243	N2	H24[8]	3.157
N2	H26[9]	3.083	N2	H27[1]	3.286
C1	H24[8]	3.394	C2	H6B[2]	3.182
C3	H12[7]	3.573	C3	H13[7]	3.238
C3	H24[8]	3.461	C5	H24[8]	3.534
C6	H2[1]	3.221	C6	H8C[2]	3.391
C7	H2[1]	3.075	C7	H27[1]	3.274
C8	H2[1]	3.48	C8	H6B[1]	3.594
C8	H25[9]	3.433	C8	H26[9]	3.421
C8	H27[1]	3.35	C9	H8A[3]	3.31
C9	H8C[3]	3.362	C10	H8A[3]	2.959
C10	H8C[3]	3.384	C10	H21A[10]	3.552
C11	H8A[3]	2.847	C11	H19[11]	3.335
C11	H20[3]	3.425	C12	H8A[3]	3.097
C12	H19[11]	3.16	C13	H3B[11]	3.354
C13	H8A[3]	3.434	C13	H25[12]	2.967
C14	H8A[3]	3.524	C14	H8B[3]	3.547

C14	H25[12]	3.236	C15	H19[6]	3.572
C16	H21A[4]	3.574	C16	H21C[4]	3.01
C17	H11[10]	3.417	C17	H21C[4]	3.458
C18	H12[7]	3.303	C19	H12[7]	2.916
C20	H12[7]	3.503	C21	H12[7]	3.59
C21	H16[13]	3.118	C22	H1A[2]	3.003
C22	H6B[2]	3.305	C23	H1A[2]	3.049
C23	H1B[8]	3.188	C23	H27[2]	3.27
C24	H1A[2]	3.124	C24	H1B[8]	3.08
C24	H3B[8]	3.165	C24	H14[8]	3.28
C24	H27[2]	3.123	C25	H1A[2]	3.163
C25	H3B[8]	3.552	C25	H8B[14]	3.161
C25	H13[15]	3.414	C25	H14[8]	3.114
C26	H1A[2]	3.12	C26	H8B[14]	3.169
C26	H24[1]	3.249	C27	H1A[2]	3.029
C27	H24[1]	3.223	H1A	C22[1]	3.003
H1A	C23[1]	3.049	H1A	C24[1]	3.124
H1A	C25[1]	3.163	H1A	C26[1]	3.12
H1A	C27[1]	3.029	H1A	H23[1]	3.555
H1A	H27[1]	3.517	H1B	C23[8]	3.188
H1B	C24[8]	3.08	H1B	H23[8]	2.774
H1B	H24[8]	2.57	H2	N1[2]	2.752
H2	C6[2]	3.221	H2	C7[2]	3.075
H2	C8[2]	3.48	H2	H6B[2]	2.777
H2	H8C[2]	2.947	H3A	H12[7]	2.968
H3A	H13[7]	3.07	H3B	C13[7]	3.354
H3B	C24[8]	3.165	H3B	C25[8]	3.552
H3B	H12[7]	3.272	H3B	H13[7]	2.584
H3B	H24[8]	2.662	H3B	H25[8]	3.379

H4	S1[2]	3.39	H4	O2[2]	2.62
H4	N1[2]	3.364	H4	H6B[2]	3.238
H6A	S1[2]	3.595	H6A	O2[2]	3.571
H6A	O3[2]	3.071	H6A	H8C[2]	3.287
H6B	O1[1]	3.045	H6B	C2[1]	3.182
H6B	C8[2]	3.594	H6B	C22[1]	3.305
H6B	H2[1]	2.777	H6B	H4[1]	3.238
H6B	H8C[2]	2.642	H8A	C9[5]	3.31
H8A	C10[5]	2.959	H8A	C11[5]	2.847
H8A	C12[5]	3.097	H8A	C13[5]	3.434
H8A	C14[5]	3.524	H8A	H10[5]	3.34
H8A	H11[5]	3.178	H8A	H12[5]	3.541
H8A	H25[9]	3.56	H8B	C14[5]	3.547
H8B	C25[9]	3.161	H8B	C26[9]	3.169
H8B	H25[9]	2.551	H8B	H26[9]	2.57
H8B	H27[1]	3.154	H8C	O1[5]	3.306
H8C	C6[1]	3.391	H8C	C9[5]	3.362
H8C	C10[5]	3.384	H8C	H2[1]	2.947
H8C	H6A[1]	3.287	H8C	H6B[1]	2.642
H8C	H10[5]	3.561	H8C	H27[1]	3.111
H10	O2[2]	2.7	H10	O3[3]	3.273
H10	H8A[3]	3.34	H10	H8C[3]	3.561
H10	H21A[10]	2.808	H11	C17[10]	3.417
H11	H8A[3]	3.178	H11	H17[10]	3.033
H11	H19[11]	3.1	H11	H20[3]	2.903
H11	H21A[10]	3.29	H11	H21C[11]	3.111
H12	C3[11]	3.573	H12	C18[11]	3.303
H12	C19[11]	2.916	H12	C20[11]	3.503
H12	C21[11]	3.59	H12	H3A[11]	2.968

H12	H3B[11]	3.272	H12	H8A[3]	3.541
H12	H19[11]	2.785	H12	H21B[11]	3.587
H12	H21C[11]	3.273	H13	N2[11]	3.243
H13	C3[11]	3.238	H13	C25[12]	3.414
H13	H3A[11]	3.07	H13	H3B[11]	2.584
H13	H24[12]	3.079	H13	H25[12]	2.693
H14	C24[8]	3.28	H14	C25[8]	3.114
H14	H24[8]	3.462	H14	H25[12]	3.161
H14	H25[8]	3.189	H16	S1[2]	3.417
H16	O2[2]	3.099	H16	O3[2]	2.845
H16	C21[4]	3.118	H16	H21A[4]	2.851
H16	H21C[4]	2.535	H17	O2[2]	3.152
H17	H11[10]	3.033	H17	H21A[10]	3.259
H17	H21B[10]	3.146	H17	H21C[4]	3.354
H19	C11[7]	3.335	H19	C12[7]	3.16
H19	C15[6]	3.572	H19	H11[7]	3.1
H19	H12[7]	2.785	H19	H21B[7]	3.06
H19	H21C[7]	3.471	H20	C11[5]	3.425
H20	H11[5]	2.903	H21A	O2[13]	2.979
H21A	O3[6]	2.773	H21A	C10[10]	3.552
H21A	C16[13]	3.574	H21A	H10[10]	2.808
H21A	H11[10]	3.29	H21A	H16[13]	2.851
H21A	H17[10]	3.259	H21B	H12[7]	3.587
H21B	H17[10]	3.146	H21B	H19[11]	3.06
H21C	O3[6]	3.453	H21C	C16[13]	3.01
H21C	C17[13]	3.458	H21C	H11[7]	3.111
H21C	H12[7]	3.273	H21C	H16[13]	2.535
H21C	H17[13]	3.354	H21C	H19[11]	3.471
H23	H1A[2]	3.555	H23	H1B[8]	2.774

H23	H27[2]	3.202	H24	N2[8]	3.157
H24	C1[8]	3.394	H24	C3[8]	3.461
H24	C5[8]	3.534	H24	C26[2]	3.249
H24	C27[2]	3.223	H24	H1B[8]	2.57
H24	H3B[8]	2.662	H24	H13[15]	3.079
H24	H14[8]	3.462	H24	H26[2]	2.983
H24	H27[2]	2.936	H25	C8[14]	3.433
H25	C13[15]	2.967	H25	C14[15]	3.236
H25	H3B[8]	3.379	H25	H8A[14]	3.56
H25	H8B[14]	2.551	H25	H13[15]	2.693
H25	H14[15]	3.161	H25	H14[8]	3.189
H26	N2[14]	3.083	H26	C8[14]	3.421
H26	H8B[14]	2.57	H26	H24[1]	2.983
H26	H26[16]	3.081	H26	H27[16]	3.345
H27	N2[2]	3.286	H27	C7[2]	3.274
H27	C8[2]	3.35	H27	C23[1]	3.27
H27	C24[1]	3.123	H27	H1A[2]	3.517
H27	H8B[2]	3.154	H27	H8C[2]	3.111
H27	H23[1]	3.202	H27	H24[1]	2.936
H27	H26[16]	3.345			

Symmetry Operators

(1) X,-Y+1,Z	(2) X,-Y+1,Z+1
(3) X,Y,Z+1	(4) -X+1,Y+1/2,-Z+1/2+1
(5) X,Y,Z-1	(6) -X+1,-Y,-Z+1
(7) X,-Y,Z	(8) -X+2,-Y,-Z+2
(9) -X+2,Y+1/2-1,-Z+1/2+1	(10) -X+1,-Y,-Z+2
(11) X,-Y,Z+1	(12) -X+2,Y+1/2-1,-Z+1/2+2
(13) -X+1,Y+1/2-1,-Z+1/2+1	(14) -X+2,Y+1/2,-Z+1/2+1
(15) -X+2,Y+1/2,-Z+1/2+2	(16) -X+2,-Y+1,-Z+2