Supplementary Information

Enantioselective [3 + 2] annulation via C–H activation between cyclic *N*-acyl ketimines and 1,3-dienes catalyzed by iridium/chiral diene complexes

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Contents of Supplementary Information:

1. General

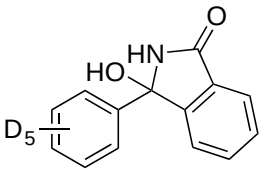
All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen. NMR spectra were recorded on a JEOL JNM LA-500 spectrometer (500 MHz for ^{1}H, 125 MHz for ^{13}C). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) and DMSO-d_6 (δ 2.49) for ^{1}H NMR, and $CDCl_3$ (δ 77.00) and DMSO-d_6 (δ 39.50) for ^{13}C NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: m, multiplet: br, broad. High-resolution mass spectra were obtained with a Bruker micrOTOF spectrometer. Optical rotations were measured on a JASCO P-2200 polarimeter. Preparative thin-layer chromatography was performed with Silica Gel 60 PF_{254} (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque. Preparative recycling gel permeation chromatography was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as eluent.

2. Materials

Toluene was purified by passing through a neutral alumina column under N_2. Iridium complexes, $[IrCl(cod)]_2$, [1] $[IrCl(coe)_2]_2$, [2] $[IrCl((S,S)\text{-Fc-tfb*})]_2$, [3] $[IrCl((S,S)\text{-Ph-tfb*})]_2$,[3] and $[IrCl((S,S)\text{-Me-tfb*})]_2$[3] were prepared according to the reported procedures. $NaBAr^F_4$ was prepared according to the reported procedures.[4]

3. Preparation of 3-aryl-3-hydroxyisoindolin-1-ones 1

Compounds **1a** (CAS: 6637-53-2),[5] **1b** (CAS: 39127-18-9),[5] **1c** (CAS: 92553-10-1),[5] **1e** (CAS: 956-92-3),[5] **1f** (CAS: 87028-37-3),[5] **1h** (CAS: 39127-19-0),[5] **1i** (CAS: 87028-38-4),[5] **1j** (CAS: 22874-54-0),[5] **1l**,[5] and **8** (CAS: 23132-29-8) were prepared according to the reported procedures. Compounds **1a-*d*$_5$**, **1d**, **1g**, and **1k** were prepared according to the procedure for **1a**.

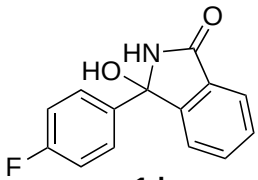


1a-*d*$_5$

Compound 1a-*d*$_5$. To a solution of phthalimide (CAS: 85-41-6) (736 mg, 5.0 mmol) in CH_2Cl_2 (13 mL) at 0 °C was slowly added C_6D_5MgBr solution prepared from C_6D_5Br (1.6 mL, 15 mmol) and Mg (401 mg, 16.5 mmol) in THF (15 mL), and the mixture was stirred at room temperature overnight. Saturated NH_4Cl solution was added and the mixture was extracted with

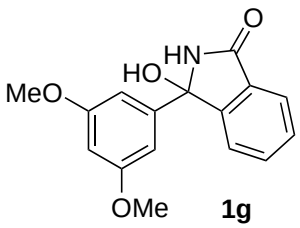
1 J. L. Herde, J. C. Lambert and C. V. Senoff, *Inorg. Synth.*, 1974, **15**, 18.
2 R. Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.*, 1985, **23**, 126.
3 (*a*) T. Nishimuara, Y. Ichikawa, T. Hayashi, N. Onishi, M. Shirotsuki and T. Masuda, *Organometallics*, 2009, **28**, 4890; (*b*) T. Nishimura, Y. Yasuhara, T. Sawano and T. Hayahi, *J. Am. Chem. Soc.*, 2010, **132**, 7872; (*c*) T. Nishimura, Y. Yasuhara, M. Nagaosa and T. Hayashi, *Tetrahedron: Asymmetry*, 2008, **19**, 1778.
4 M. Brookhart, B. Grant and A. F. Volpe, Jr., *Organometallics*, 1992, **11**, 3920.
5 T. Nishimura, A. Noishiki, Y. Ebe and T. Hayashi, *Angew. Chem., Int. Ed.*, 2013, **52**, 1777.

CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The solid residue was recrystallized from hot CH_2Cl_2 and hexane to give **1a-*d*$_5$** (1.09 g, 4.7 mmol, 95% yield). ^{1}H NMR (CD_3OD) δ 7.26–7.38 (m, 4H), 7.46–7.58 (m, 4H), 7.75 (d, *J* = 7.5 Hz, 1H) ^{1}H NMR (DMSO-*d*$_6$) δ 6.87 (s, 1H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.63 (d, *J* = 7.4 Hz, 1H), 9.21 (s, 1H). HRMS (ESI) calcd for $C_{14}H_6D_5NNaO_2$ $(M+Na)^+$ 253.0996, found 253.0997.



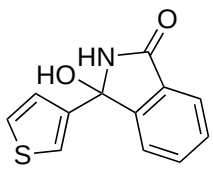
1d

Compound 1d. This compound was prepared by the reaction of phthalimide with 4-fluorophenylmagnesium bromide (3 equiv). The solid residue obtained after work-up was recrystallized from hot EtOAc and hexane to give **1d** (78% yield). ^{1}H NMR (DMSO-*d*$_6$) δ 6.96 (s, 1H), 7.16 (t, *J* = 8.9 Hz, 2H), 7.31 (d, *J* = 7.4 Hz, 1H), 7.44–7.57 (m, 4H), 7.78 (d, *J* = 7.4 Hz, 1H), 9.25 (s, 1H); ^{13}C NMR (DMSO-*d*$_6$) δ 87.0, 114.9 (d, $J_{F\text{-}C}$ = 22 Hz), 122.6, 122.7, 127.7 (d, $J_{F\text{-}C}$ = 8 Hz), 129.0, 130.5, 132.4, 138.4 (d, $J_{F\text{-}C}$ = 3 Hz), 150.6, 161.7 (d, $J_{F\text{-}C}$ = 244 Hz), 168.3. HRMS (ESI) calcd for $C_{14}H_{10}FNNaO_2$ $(M+Na)^+$ 266.0588, found 266.0589.



1g

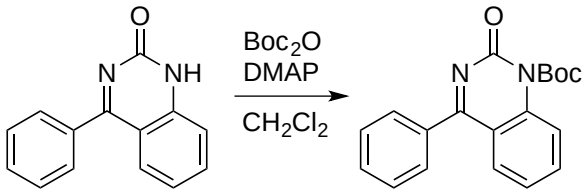
Compound 1g. This compound was prepared by the reaction of phthalimide with 3,5-dimethoxyphenylmagnesium bromide (2.5 equiv). The solid residue obtained after work-up was washed with Et_2O and dried under vacuum to give **1g** (83% yield). ^{1}H NMR ($CDCl_3$) δ 3.75 (s, 6H), 6.40 (t, *J* = 2.3 Hz, 1H), 6.71 (d, *J* = 2.3 Hz, 2H), 6.73 (br s, 2H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.67 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 55.4, 88.2, 100.5, 103.7, 122.7, 123.6, 129.3, 129.4, 133.1, 142.4, 149.7, 160.9, 170.0. HRMS (ESI) calcd for $C_{16}H_{15}NNaO_4$ $(M+Na)^+$ 308.0893, found 308.0891.



1k

Compound 1k. To a solution of 3-bromothiophene (1.9 mL, 20 mmol) in hexane (30 mL)

was added dropwise BuLi (1.6 M in hexane, 13 mL, 21 mmol) at –50 °C.[6] After completion of the addition, THF (3 mL) was slowly added to the mixture, and it was stirred for 15 min. The mixture was diluted with hexane (10 mL) at –50 °C, and $MgBr_2$ (20 mmol) in THF (60 mL) prepared from 1,2-dibromoethane and Mg was added to the mixture at 0 °C. After the mixture was stirred for 1 h, phthalimide (1.03 g, 7.0 mmol) was added and the mixture was stirred at room temperature overnight. Saturated NH_4Cl solution was added and the mixture was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (3:1). The solid product was recrystallized from hot EtOAc and hexane to give **1k** (923 mg, 4.0 mmol, 57% yield). ^{1}H NMR (DMSO-d_6) δ 6.81 (s, 1H), 7.03 (dd, J = 4.9, 1.4 Hz, 1H), 7.45 (d, J = 7.5 Hz, 1H), 7.46–7.51 (m, 3H), 7.56 (d, J = 7.4, 1.0 Hz, 1H), 7.63 (d, J = 7.4 Hz, 1H), 9.27 (s, 1H); ^{13}C NMR (DMSO-d_6) δ 85.8, 121.8, 122.5, 122.7, 126.2, 126.6, 129.0, 130.5, 132.3, 144.0, 150.1, 167.9. HRMS (ESI) calcd for $C_{12}H_9NNaO_2S$ $(M+Na)^+$ 254.0246, found 254.0251.



s1 **6**

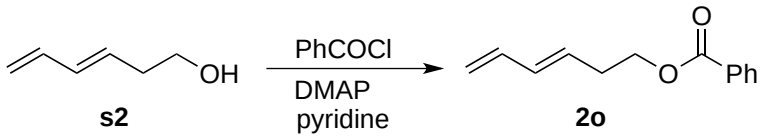
Compound 6. To a solution of **s1** (CAS: 23441-75-0; 112.7 mg, 0.50 mmol) and Boc_2O (220 μL, 0.80 mmol) in CH_2Cl_2 (1.0 mL) was added 4-dimethylaminopyridine (12.2 mg, 0.10 mmol) at 0 °C. The mixture was stirred at room temperature for 24 h, and the solvent was removed on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (2:1). The solid product was recrystallized from CH_2Cl_2 and hexane to give **6** (101.4 mg, 0.31 mmol, 63% yield). ^{1}H NMR ($CDCl_3$) δ 1.71 (s, 9H), 7.26 (td, J = 7.7, 1.0 Hz, 1H), 7.29 (d, J = 8.4 Hz, 1H), 7.51–7.60 (m, 3H), 7.70–7.74 (m, 3H), 7.84 (dd, J = 8.2, 1.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 27.6, 87.1, 114.0, 114.9, 123.1, 128.4, 129.6, 129.9, 130.9, 135.3, 136.2, 140.9, 150.2, 152.6, 176.3. HRMS (ESI) calcd for $C_{19}H_{18}N_2NaO_3$ $(M+Na)^+$ 345.1210, found 345.1212.

4. Preparation of 1,3-dienes 2

Isoprene (**2m**), 1,3-cycloheptadiene (**2v**), 2,3-dimethyl-1,3-butadiene (**2w**), and ethyl sorbate (**2x**) were purchased from commercial suppliers and used as received. 1,3-Dienes **2n** (CAS: 58396-45-5),[7] **2p** (CAS: 16939-57-4),[8] **2q** (CAS: 13369-23-8),[9] **2r** (CAS: 1056894-74-6),[10] **2s**

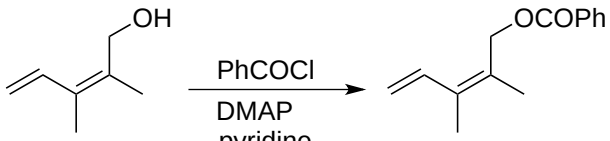
6 X. Wu, N.-A. Chen, L. Zhu and R. D. Rieke, *Tetrahedron Lett.*, 1994, **35**, 3673.
7 J. Y. Wu, B. Moreau and T. Ritter, *J. Am. Chem. Soc.*, 2009, **131**, 12915.

(CAS: 221219-66-5),[11] and **2t** (CAS: 1421371-63-2)[12] were prepared according to the reported procedures. The procedures for the synthesis of **2o** and **2u** are shown below.



s2 **2o**

Compound 2o: To a solution of **s2**[13] (CAS: 73670-87-8; 491 mg, 5.0 mmol), 4-dimethylaminopyridine (61 mg, 0.50 mmol) and pyridine (0.81 mL, 10 mmol) in CH_2Cl_2 (10 mL) was added dropwise benzoyl chloride (0.87 mL, 7.5 mmol) at 0 °C, and the mixture was stirred at room temperature for 1 h. Saturated NH_4Cl solution was added and the mixture was extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (1:20) to give **2o** (806 mg, 4.0 mmol, 80% yield). ^{1}H NMR ($CDCl_3$) δ 2.57 (q, *J* = 6.8 Hz, 2H), 4.37 (t, *J* = 6.8 Hz, 2H), 5.03 (d, *J* = 10.3 Hz, 1H), 5.14 (d, *J* = 16.9 Hz, 1H), 5.75 (dt, *J* = 15.3, 6.8 Hz, 1H), 6.19 (dd, *J* = 15.3, 10.3 Hz, 1H), 6.33 (dt, *J* = 16.9, 10.3 Hz, 1H), 7.40–7.47 (m, 2H), 7.53–7.58 (m, 1H), 8.01–8.06 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 32.0, 64.1, 116.1, 128.3, 129.5, 129.7, 130.3, 132.9, 133.5, 136.8, 166.5. HRMS (ESI) calcd for $C_{13}H_{14}NaO_2$ $(M+Na)^+$ 225.0886, found 225.0891.



s3 **2u**

Compound 2u: To a solution of **s3**[14] (CAS: 1193304-96-9; 390 mg, 3.5 mmol), 4-dimethylaminopyridine (42.8 mg, 0.35 mmol) and pyridine (0.56 mL, 7.0 mmol) in CH_2Cl_2 (35 mL) was added dropwise benzoyl chloride (620 μL, 5.3 mmol) at 0 °C, and the mixture was stirred at 0 °C for 3 h. Saturated NH_4Cl solution was added and the mixture was extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (1:20) to give **2u** (679.4 mg, 3.14 mmol, 90% yield). ^{1}H NMR ($CDCl_3$) δ 1.87 (s, 3H), 1.93 (s, 3H), 5.00 (s, 2H), 5.13 (d, *J* = 11.0 Hz, 1H), 5.28 (d, *J* = 17.1 Hz, 1H), 6.96 (dd, *J* = 17.1, 11.0 Hz, 1H), 7.43 (t, *J* = 7.9 Hz, 2H), 7.53–7.57 (m, 1H), 8.03–8.07 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 18.0, 64.9, 114.1, 128.3, 128.6, 129.6, 130.3, 132.8, 133.1, 134.5, 166.7. HRMS

8 H. Lebel and V. Paquet, *J. Am. Chem. Soc.*, 2004, **126**, 320.
9 J. Rodriguez and B. Waegell, *Synthesis*, 1988, 534.
10 A. P. Marcus, A. S. Lee, R. L. Davis, D. J. Tantillo and R. Sarpong, *Angew. Chem., Int. Ed.*, 2008, **47**, 6379.
11 T. Nishimura, Y. Yasuhara and T. Hayashi, *Angew. Chem., Int. Ed.*, 2006, **45**, 5164.
12 T. Nishimura, Y. Ebe and T. Hayashi, *J. Am. Chem. Soc.*, 2013, **135**, 2092.
13 M. Kimura, A. Ezoe, M. Mori and Y. Tamaru, *J. Am. Chem. Soc.*, 2005, **127**, 201.
14 R. Schobert and B. Barnickel, *Synthesis*, 2009, 2778.

(ESI) calcd for $C_{14}H_{16}NaO_2$ $(M+Na)^+$ 239.1043, found 239.1044.

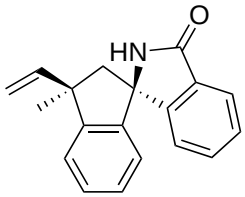
5. A general procedure for Ir-catalyzed asymmetric annulation of 3-hydroxy-3-phenylisoindolin-1-one (1a) with isoprene (2m) (Table 1)

3-Hydroxy-3-phenylisoindolin-1-one (**1a**: 45.0 mg, 0.20 mmol), $NaBAr^F_4$ (18.4 mg calculated as the dihydrate, 0.020 mmol, 10 mol %), and an iridium complex (0.010 mmol of Ir, 5 mol % of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL), isoprene (**2m**: 30 μL, 0.30 mmol), and a base (0.010 mmol, 5 mol %) were added successively. The Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was passed through a short column of alumina with EtOAc as eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/$CHCl_3$ (1:5) to give **3am**.

6. General procedures for Ir-catalyzed asymmetric annulation of 3-aryl-3-hydroxyisoindolin-1-ones 1 with 1,3-dienes 2 (Scheme 2 and Scheme 3)

3-Aryl-3-hydroxyisoindolin-1-one **1** (0.20 mmol), $NaBAr^F_4$ (18.4 mg calculated as the dihydrate, 0.020 mmol, 10 mol%), and $[IrCl((S,S)\text{-Me-tfb*})]_2$ (4.8 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL), 1,3-diene **2** (0.30 mmol), and a solution of DABCO (0.25 M in toluene, 40 μL, 0.010 mmol, 5 mol%) were added successively. The Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was passed through a short column of alumina with EtOAc as eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel to give **3**. In the reaction using 10 mol% of the iridium catalyst (eq 1), 10 mol% of DABCO and 20 mol% of $NaBAr^F_4$ were used.

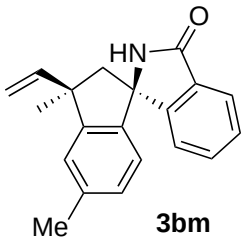
7. Characterization of the products



3am

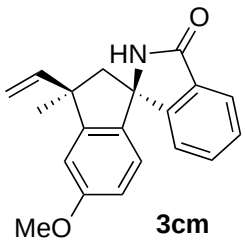
Compound 3am. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.2 min (major), t_2 = 8.7 min (minor); $[\alpha]^{20}_D$ +100 (*c* 1.00, $CHCl_3$) for 91% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.57 (s, 3H), 2.49 (d, *J* = 13.5 Hz, 1H), 2.66 (d, *J* = 13.5 Hz, 1H), 4.62 (dd, *J* = 17.2, 1.1 Hz, 1H), 5.05 (dd, *J* = 10.4, 1.1 Hz, 1H), 6.21 (dd, *J* = 17.2, 10.4 Hz,

1H), 6.43 (br s, 1H), 6.69 (d, *J* = 7.5 Hz, 1H), 7.19 (td, *J* = 7.5, 1.2 Hz, 1H), 7.24–7.30 (m, 2H), 7.39 (td, *J* = 7.5, 1.1 Hz, 1H), 7.51 (td, *J* = 7.5, 1.0 Hz, 1H), 7.57 (td, *J* = 7.5, 1.2 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.5, 49.9, 53.8, 70.0, 113.4, 122.4, 123.5, 124.0, 124.2, 128.1, 128.4, 129.1, 131.7, 132.4, 143.1, 147.8, 148.2, 150.5, 168.7. HRMS (ESI) calcd for $C_{19}H_{17}NNaO$ $(M+Na)^+$ 298.1202, found 298.1200.



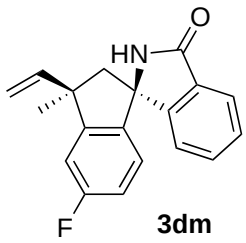
3bm

Compound 3bm. A solution of $CHCl_3$/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 6.9 min (major), t_2 = 9.2 min (minor); $[\alpha]^{20}_D$ +103 (*c* 0.78, $CHCl_3$) for 97% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.56 (s, 3H), 2.40 (s, 3H), 2.47 (d, *J* = 13.5 Hz, 1H), 2.64 (d, *J* = 13.5 Hz, 1H), 4.65 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.04 (dd, *J* = 10.3, 1.2 Hz, 1H), 6.19 (dd, *J* = 17.2, 10.3 Hz, 1H), 6.41 (br s, 1H), 6.58 (d, *J* = 7.8 Hz, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 7.07 (s, 1H), 7.23–7.26 (m, 1H), 7.50 (td, *J* = 7.5, 1.0 Hz, 1H), 7.56 (td, *J* = 7.5, 1.2 Hz, 1H), 7.86–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 21.5, 26.5, 49.6, 54.0, 69.8, 113.2, 122.4, 123.4, 123.7, 124.6, 128.3, 129.0, 131.7, 132.3, 139.1, 140.1, 147.8, 148.4, 150.7, 168.7. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1351.



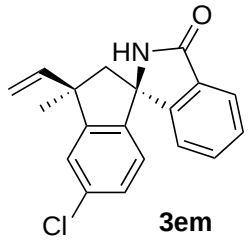
3cm

Compound 3cm. A solution of hexane/$CHCl_3$/EtOAc (1:1:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.3 min (major), t_2 = 10.3 min (minor); $[\alpha]^{20}_D$ +101 (*c* 1.08, $CHCl_3$) for 98% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.55 (s, 3H), 2.48 (d, *J* = 13.4 Hz, 1H), 2.67 (d, *J* = 13.4 Hz, 1H), 3.83 (s, 3H), 4.69 (dd, *J* = 17.3, 1.1 Hz, 1H), 5.06 (dd, *J* = 10.3, 1.1 Hz, 1H), 6.21 (dd, *J* = 17.3, 10.3 Hz, 1H), 6.41 (br s, 1H), 6.61 (d, *J* = 8.3 Hz, 1H), 6.72 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.77 (d, *J* = 2.4 Hz, 1H), 7.24–7.26 (m, 1H), 7.50 (td, *J* = 7.4, 0.9 Hz, 1H), 7.56 (td, *J* = 7.5, 1.2 Hz, 1H), 7.85–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.4, 49.7, 54.4, 55.4, 69.6, 109.1, 113.3, 114.2, 122.3, 123.4, 124.9, 128.3, 131.7, 132.3, 134.9, 147.6, 150.0, 150.7, 160.7, 168.6. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1303.



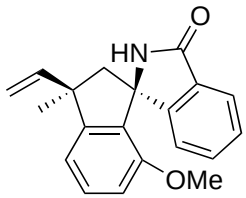
3dm

Compound 3dm. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column × 2, hexane/chloroform/ethanol = 6:2:1, flow 0.4 mL/min, 254 nm, t_1 = 23.0 min (major), t_2 = 26.3 min (minor); $[\alpha]^{20}_D$ +103 (*c* 0.92, $CHCl_3$) for 97% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.56 (s, 3H), 2.50 (d, *J* = 13.6 Hz, 1H), 2.69 (d, *J* = 13.6 Hz, 1H), 4.65 (dd, *J* = 17.3, 0.9 Hz, 1H), 5.09 (dd, *J* = 10.4, 0.9 Hz, 1 H), 6.18 (dd, *J* = 17.3, 10.4 Hz, 1 H), 6.42 (br s, 1H), 6.66 (dd, *J* = 8.5, 5.1 Hz, 1H), 6.88 (td, *J* = 8.5, 2.4 Hz, 1H), 6.95 (dd, *J* = 8.5, 2.4 Hz, 1H), 7.23–7.26 (m, 1H), 7.52 (td, *J* = 7.5, 0.9 Hz, 1 H), 7.58 (td, *J* = 7.5, 1.2 Hz, 1H), 7.87–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.3, 49.7 (d, J_{F-C} = 2 Hz), 54.2, 69.4, 111.1 (d, J_{F-C} = 22 Hz), 113.6, 115.5 (d, J_{F-C} = 23 Hz), 122.3, 123.6, 125.6 (d, J_{F-C} = 9 Hz), 128.6, 131.6, 132.5, 138.6 (d, J_{F-C} = 3 Hz), 147.2, 150.3, 150.7 (d, J_{F-C} = 8 Hz), 163.7 (d, J_{F-C} = 248 Hz), 168.6. HRMS (ESI) calcd for $C_{19}H_{16}FNNaO$ $(M+Na)^+$ 316.1108, found 316.1108.



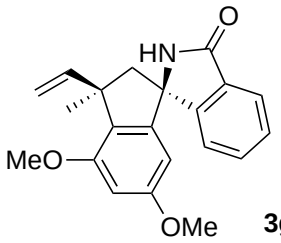
3em

Compound 3em. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.5 min (major), t_2 = 8.7 min (minor); $[\alpha]^{20}_D$ +87 (*c* 0.73, $CHCl_3$) for 92% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.56 (s, 3H), 2.49 (d, *J* = 13.6 Hz, 1H), 2.67 (d, *J* = 13.6 Hz, 1H), 4.65 (d, *J* = 17.3 Hz, 1H), 5.09 (d, *J* = 10.4 Hz, 1H), 6.18 (dd, *J* = 17.3, 10.4 Hz, 1H), 6.41 (br s, 1H), 6.63 (d, *J* = 8.2 Hz, 1H), 7.17 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 2.0 Hz, 1H), 7.52 (td, *J* = 7.5, 0.9 Hz, 1H), 7.58 (td, *J* = 7.5, 1.2 Hz, 1H), 7.88 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.3, 49.9, 53.9, 69.4, 113.8, 122.3, 123.6, 124.5, 125.3, 128.5, 128.6, 131.6, 132.5, 135.1, 141.7, 147.1, 150.1, 150.2, 168.6. HRMS (ESI) calcd for $C_{19}H_{16}ClNNaO$ $(M+Na)^+$ 332.0813, found 332.0810.



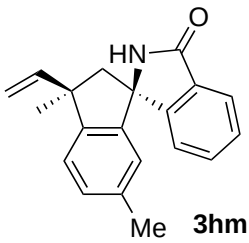
3fm

Compound 3fm. A solution of hexane/$CHCl_3$/EtOAc (1:1:2) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 6.9 min (major), t_2 = 8.3 min (minor); $[\alpha]^{20}_D$ +10 (*c* 1.01, $CHCl_3$) for 96% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.56 (s, 3H), 2.43 (d, *J* = 13.7 Hz, 1H), 2.62 (d, *J* = 13.7 Hz, 1H), 2.99 (s, 3H), 4.62 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.02 (dd, *J* = 10.4, 1.2 Hz, 1H), 6.15 (dd, *J* = 17.2, 10.4 Hz, 1H), 6.25 (br s, 1H), 6.65 (d, *J* = 8.2 Hz, 1H), 6.87 (dd, *J* = 7.9, 0.7 Hz, 1H), 7.22–7.25 (m, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 7.44 (td, *J* = 7.5, 0.9 Hz, 1H), 7.51 (td, *J* = 7.5, 1.1 Hz, 1H), 7.84–7.87 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.3, 49.9, 54.2, 55.0, 69.0, 109.9, 112.9, 116.1, 121.5, 123.2, 127.6, 128.7, 130.9, 131.55, 131.63, 147.7, 150.6, 151.5, 156.3, 169.4. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1307.



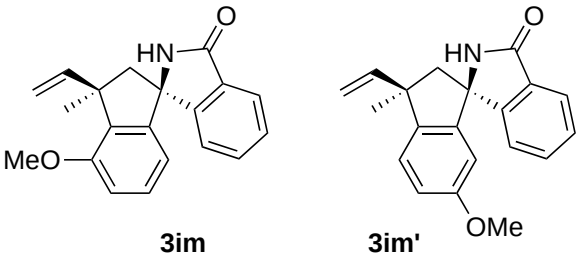
3gm

Compound 3gm. A solution of hexane/$CHCl_3$/EtOAc (1:1:2) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column × 2, hexane/chloroform/ethanol = 6:2:1, flow 0.4 mL/min, 254 nm, t_1 = 20.7 min (major), t_2 = 22.9 min (minor); $[\alpha]^{20}_D$ +36 (*c* 1.04, $CHCl_3$) for 96% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.64 (s, 3H), 2.44 (d, *J* = 13.6 Hz, 1H), 2.62 (d, *J* = 13.6 Hz, 1H), 3.60 (s, 3H), 3.82 (s, 3H), 4.71 (dd, *J* = 17.3, 1.2 Hz, 1H), 5.01 (dd, *J* = 10.3, 1.2 Hz, 1H), 5.71 (d, *J* = 2.1 Hz, 1H), 6.19 (dd, *J* = 17.3, 10.3 Hz, 1H), 6.39 (br s, 1H), 6.43 (d, *J* = 2.1 Hz, 1H), 7.30 (d, *J* = 7.5 Hz, 1H), 7.51 (td, *J* = 7.5, 0.8 Hz, 1 H), 7.58 (td, *J* = 7.5, 1.1 Hz, 1H), 7.88 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.2, 49.9, 54.0, 55.2, 55.4, 70.5, 98.8, 99.9, 112.0, 122.5, 123.5, 127.7, 128.4, 131.7, 132.4, 145.4, 146.2, 150.3, 157.6, 161.5, 168.7. HRMS (ESI) calcd for $C_{21}H_{21}NNaO_3$ $(M+Na)^+$ 358.1414, found 358.1407.



3hm

Compound 3hm. A solution of $CHCl_3$/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 30.4 min (major), t_2 = 35.1 min (minor); $[\alpha]^{20}_D$ +94 (*c* 1.00, $CHCl_3$) for 90% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.55 (s, 3H), 2.23 (s, 3H), 2.47 (d, *J* = 13.4 Hz, 1H), 2.65 (d, *J* = 13.4 Hz, 1H), 4.63 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.03 (dd, *J* = 10.4, 1.2 Hz, 1H), 6.19 (dd, *J* = 17.2, 10.4 Hz, 1H), 6.41 (br s, 1H), 6.48 (s, 1H), 7.15–7.20 (m, 2H), 7.25–7.28 (m, 1H), 7.51 (td, *J* = 7.5, 0.9 Hz, 1H), 7.58 (td, *J* = 7.5, 1.2 Hz, 1H), 7.87–7.91 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 21.1, 26.6, 49.5, 53.9, 70.0, 113.2, 122.5, 123.5, 123.9, 124.3, 128.4, 130.0, 131.8, 132.3, 138.0, 143.1, 145.3, 147.9, 150.6, 168.7. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1356.

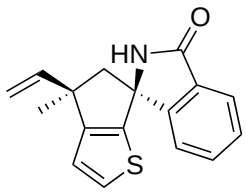


3im **3im'**

Compound 3im. A solution of hexane/$CHCl_3$/EtOAc (1:1:2) was used as an eluent for preparative TLC to give a mitxture of **3im** and **3im'**. The mixture was subjected to preparative TLC on silica gel eluted ten times with hexane/EtOAc (3:1) to give **3im** and **3im'**. The ee was measured by HPLC (Chiralpak IC column, hexane/chloroform/ethanol = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 34.7 min (minor), t_2 = 36.5 min (major); $[\alpha]^{20}_D$ +125 (*c* 0.97, $CHCl_3$) for 98% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.68 (s, 3H), 2.46 (d, *J* = 13.7 Hz, 1H), 2.64 (d, *J* = 13.7 Hz, 1H), 3.86 (s, 3H), 4.71 (dd, *J* = 17.3, 1.2 Hz, 1H), 5.03 (dd, *J* = 10.4, 1.2 Hz, 1H), 6.22 (dd, *J* = 17.3, 10.3 Hz, 1H), 6.26 (d, *J* = 7.6 Hz, 1H), 6.40 (br s, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.8 Hz, 1H), 7.27–7.30 (m, 1H), 7.50 (td, *J* = 7.5, 0.8 Hz, 1H), 7.57 (td, *J* = 7.5, 1.0 Hz, 1H), 7.86–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.1, 50.6, 53.7, 55.3, 70.2, 111.2, 112.3, 116.1, 122.4, 123.5, 128.4, 129.8, 131.7, 132.4, 135.1, 145.0, 145.9, 150.5, 157.0, 168.7. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1308.

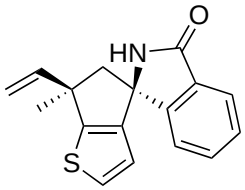
Compound 3im'. The ee was measured by HPLC (Chiralpak IC column, hexane/chloroform/ethanol = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 43.3 min (major), t_2 = 48.3 min (minor); $[\alpha]^{20}_D$ +85 (*c* 1.21, $CHCl_3$) for 83% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.54 (s, 3H), 2.47 (d, *J* = 13.4 Hz, 1H), 2.66 (d, *J* = 13.4 Hz, 1H), 3.65 (s, 3H), 4.63 (dd, *J* = 17.2, 1.1 Hz, 1H),

5.03 (dd, *J* = 10.4, 1.1 Hz, 1H), 6.16 (d, *J* = 2.4 Hz, 1H), 6.19 (dd, *J* = 17.3, 10.4 Hz, 1H), 6.43 (br s, 1H), 6.93 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 7.26–7.29 (m, 1H), 7.51 (td, *J* = 7.5, 0.8 Hz, 1H), 7.58 (td, *J* = 7.5, 1.2 Hz, 1H), 7.87–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.7, 49.3, 54.3, 55.4, 70.0, 108.1, 113.3, 116.1, 122.5, 123.6, 125.0, 128.5, 131.8, 132.4, 140.3, 144.4, 148.2, 150.4, 159.9, 168.7. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1310.



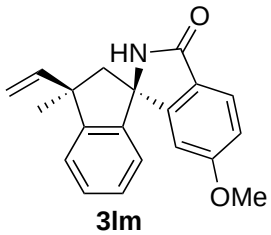
3jm

Compound 3jm. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.8 min (major), t_2 = 8.9 min (minor); $[\alpha]^{20}_D$ +95 (*c* 0.98, $CHCl_3$) for 99% ee (4*R*,6*S*). ^{1}H NMR ($CDCl_3$) δ 1.54 (s, 3H), 2.69 (d, *J* = 13.4 Hz, 1H), 3.00 (d, *J* = 13.4 Hz, 1H), 4.74 (dd, *J* = 17.2, 1.0 Hz, 1H), 5.05 (dd, *J* = 10.3, 1.0 Hz, 1H), 6.17 (dd, *J* = 17.2, 10.3 Hz, 1H), 6.39 (br s, 1H), 6.85 (d, *J* = 5.0 Hz, 1H), 7.38–7.41 (m, 2H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.60 (td, *J* = 7.5, 0.9 Hz, 1H), 7.85 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.8, 47.7, 59.4, 67.9, 113.0, 121.3, 122.2, 123.6, 128.7, 131.1, 132.0, 132.5, 141.9, 146.9, 150.0, 154.2, 168.4. HRMS (ESI) calcd for $C_{17}H_{15}NNaOS$ $(M+Na)^+$ 304.0767, found 304.0773.



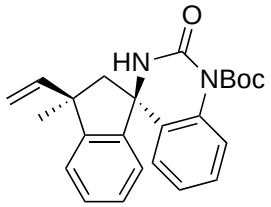
3km

Compound 3km. A solution of hexane/$CHCl_3$/EtOAc (1:1:2) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column × 2, hexane/chloroform/ethanol = 6:2:1, flow 0.4 mL/min, 254 nm, t_1 = 23.4 min (major), t_2 = 37.0 min (minor); $[\alpha]^{20}_D$ +68 (*c* 0.99, $CHCl_3$) for 97% ee (4*S*,6*R*). ^{1}H NMR ($CDCl_3$) δ 1.59, (s, 3H), 2.71 (d, *J* = 13.5 Hz, 1H), 3.00 (d, *J* = 13.5 Hz, 1H), 4.89 (dd, *J* = 17.1, 0.9 Hz, 1H), 5.10 (dd, *J* = 10.3, 0.9 Hz, 1H), 6.20 (dd, *J* = 17.1, 10.3 Hz, 1H), 6.32 (br s, 1H), 6.37 (d, *J* = 5.0 Hz, 1H), 7.24 (d, *J* = 5.0 Hz, 1H), 7.31–7.34 (m, 1H), 7.50 (td, *J* = 7.5, 0.8 Hz, 1H), 7.57 (td, *J* = 7.5, 1.0 Hz, 1H), 7.84–7.88 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 27.9, 48.4, 59.1, 67.3, 113.0, 120.3, 122.2, 123.6, 128.5, 130.2, 131.2, 132.4, 144.8, 146.8, 150.1, 151.8, 168.7. HRMS (ESI) calcd for $C_{17}H_{15}NNaOS$ $(M+Na)^+$ 304.0767, found 304.0765.



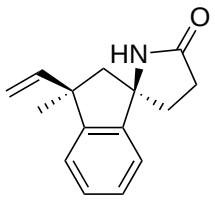
3lm

Compound 3lm. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.2 min (major), t_2 = 8.7 min (minor); $[\alpha]^{20}_D$ +157 (*c* 1.04, $CHCl_3$) for 85% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.57 (s, 3H), 2.48 (d, *J* = 13.5 Hz, 1H), 2.61 (d, *J* = 13.5 Hz, 1H), 3.82 (s, 3H), 4.60 (dd, *J* = 17.2, 1.1 Hz, 1H), 5.04 (dd, *J* = 10.3, 1.1 Hz, 1H), 6.19 (dd, *J* = 17.2, 10.4 Hz, 1H), 6.30 (br s, 1H), 6.68 (d, *J* = 2.3 Hz, 1H), 6.74 (d, *J* = 7.7, 1.2 Hz, 1H), 7.02 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.21 (td, *J* = 7.6, 1.1 Hz, 1H), 7.27–7.30 (m, 1H), 7.39 (td, *J* = 7.6, 1.1 Hz, 1H), 7.79 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 26.4, 49.8, 54.0, 55.6, 69.6, 106.9, 113.3, 115.1, 124.0, 124.1, 124.3, 124.9, 128.1, 129.0, 143.2, 147.7, 148.1, 152.9. 163.4, 168.5. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1302.



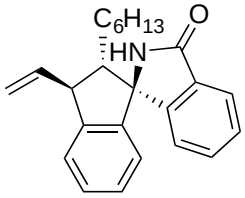
7

Compound 7. A solution of $CHCl_3$/EtOAc (6:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 6.1 min (major), t_2 = 6.7 min (minor); $[\alpha]^{20}_D$ +55 (*c* 0.95, $CHCl_3$) for 90% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.43 (s, 3H), 1.62 (s, 9H), 2.36 (d, *J* = 13.7 Hz, 1H), 2.76 (d, *J* = 13.7 Hz, 1H), 4.82 (dd, *J* = 17.3, 0.6 Hz, 1H), 5.05 (dd, *J* = 10.4, 0.6 Hz, 1H), 5.39 (br s, 1H), 6.14 (dd, *J* = 17.3, 10.4 Hz, 1H), 6.77 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.03 (td, *J* = 7.6, 1.0 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.25–7.29 (m, 2H), 7.34 (td, *J* = 7.6, 1.1 Hz, 1H), 7.43–7.46 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 26.8, 27.8, 49.0, 56.7, 66.8, 84.0, 112.5, 119.5, 124.29, 124.33, 124.9, 125.5, 127.7, 128.3, 129.6, 131.7, 135.6, 143.0, 147.2, 149.0, 151.1, 151.4. HRMS (ESI) calcd for $C_{24}H_{26}N_2NaO_3$ $(M+Na)^+$ 413.1836, found 413.1838.



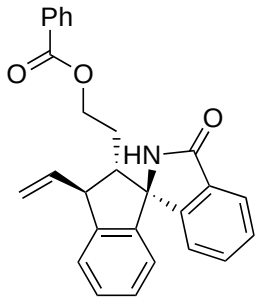
9

Compound 9. A solution of hexane/EtOAc (1:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 8.5 min (major), t_2 = 12.5 min (minor); $[\alpha]^{20}_D$ +73 (*c* 0.96, $CHCl_3$) for 98% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.42 (s, 3H), 2.22 (d, *J* = 13.3 Hz, 1H), 2.30–2.38 (m, 1H), 2.35 (d, *J* = 13.3 Hz, 1H), 2.45–2.64 (m, 3H), 4.78 (dd, *J* = 17.3, 0.8 Hz, 1H), 5.03 (dd, *J* = 10.7, 0.8 Hz, 1H), 5.72 (br s, 1H), 6.05 (dd, *J* = 17.3, 10.7 Hz, 1H), 7.14–7.17 (m, 1H), 7.30–7.35 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 26.6, 30.4, 36.0, 48.4, 54.7, 67.6, 112.6, 122.9, 123.9, 127.9, 128.7, 145.4, 146.8, 147.9, 176.5. HRMS (ESI) calcd for $C_{15}H_{17}NNaO$ $(M+Na)^+$ 250.1202, found 250.1201.



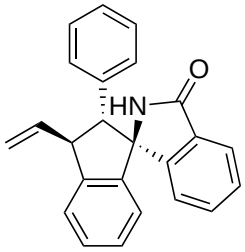
3an

Compound 3an. A solution of hexane/EtOAc (3:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 6.0 min (major), t_2 = 9.7 min (minor); $[\alpha]^{20}_D$ –228 (*c* 0.69, $CHCl_3$) for >99.5% ee (1*R*,2*S*,3*S*). ^{1}H NMR ($CDCl_3$) δ 0.78 (t, *J* = 7.2 Hz, 3H), 1.00–1.40 (m, 10H), 2.46–2.54 (m, 1H), 3.73 (t, *J* = 9.5 Hz, 1H), 5.30 (dd, *J* = 10.0, 1.7 Hz, 1H), 5.35 (dd, *J* = 16.9, 1.6 Hz, 1H), 5.81 (ddd, *J* = 17.0, 9.9, 9.0 Hz, 1H), 6.33 (br s, 1H), 6.95–7.00 (m, 2H), 7.18 (t, *J* = 7.4 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.30 (td, *J* = 7.4, 1.0 Hz, 1H), 7.40–7.46 (m, 2H), 7.86–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 22.3, 27.5, 29.1, 29.3, 31.2, 54.4, 56.0, 73.7, 117.6, 122.7, 123.1, 123.9, 125.0, 127.8, 128.1, 128.5, 130.9, 131.7, 139.9, 143.5, 143.6, 148.9, 170.8. HRMS (ESI) calcd for $C_{24}H_{27}NNaO$ $(M+Na)^+$ 368.1985, found 368.1975.



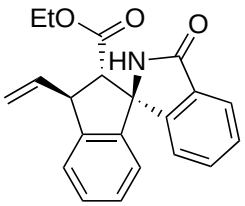
3ao

Compound 3ao. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.8 min (major), t_2 = 11.0 min (minor); $[\alpha]^{25}_D$ –263 (*c* 1.04, $CHCl_3$) for >99.5% ee (1*R*,2*S*,3*S*). ^{1}H NMR ($CDCl_3$) δ 1.62–1.70 (m, 1H), 1.72–1.80 (m, 1H), 2.77 (ddd, *J* = 9.7, 8.5, 5.6 Hz, 1H), 3.87 (t, *J* = 9.7 Hz, 1H), 4.20 (dt, *J* = 11.0, 7.3 Hz, 1H), 4.32 (ddd, *J* = 11.0, 7.4, 5.9 Hz, 1H), 5.32 (dd, *J* = 10.0, 1.3 Hz, 1H), 5.43 (dd, *J* = 16.1, 1.3 Hz, 1H), 5.82 (ddd, *J* = 16.1, 10.0, 9.7 Hz, 1H), 6.49 (br s, 1H), 7.00–7.04 (m, 2H), 7.20 (t, *J* = 7.4 Hz, 1H), 7.25 (d, *J* = 6.7 Hz, 1H), 7.32 (t, *J* = 7.0 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.43–7.48 (m, 2H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.87–7.92 (m, 1H), 7.95 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 28.3, 52.7, 53.8, 62.7, 73.5, 118.4, 122.7, 123.0, 124.2, 125.1, 128.0, 128.2, 128.5, 128.7, 129.4, 130.1, 130.7, 132.1, 132.7, 139.3, 143.1, 143.2, 148.5, 166.1, 170.8. HRMS (ESI) calcd for $C_{27}H_{23}NNaO_3$ $(M+Na)^+$ 432.1570, found 432.1561.



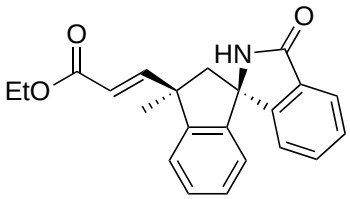
3ap

Compound 3ap. A solution of $CHCl_3$/EtOAc (5:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column × 2, hexane/chloroform/ethanol = 6:2:1, flow 0.4 mL/min, 254 nm, t_1 = 22.3 min (major), t_2 = 48.3 min (minor); $[\alpha]^{20}_D$ –345 (*c* 0.96, $CHCl_3$) for >99.5% ee (1*R*,2*R*,3*S*). ^{1}H NMR ($CDCl_3$) δ 3.85 (d, *J* = 10.4 Hz, 1H), 4.59 (dd, *J* = 10.4 Hz, 1H), 5.24 (dd, *J* = 10.0, 1.4 Hz, 1H), 5.33 (dd, *J* = 17.1, 1.4 Hz, 1H), 5.89 (ddd, *J* = 17.1, 10.0, 8.2 Hz, 1H), 6.90 (br s, 1H), 7.00 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.02–7.10 (m, 4H), 7.13 (d, *J* = 7.8 Hz, 2H), 7.21–7.28 (m, 2H), 7.31 (td, *J* = 7.6, 1.3 Hz, 1H), 7.36–7.42 (m, 2H), 7.57 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 50.0, 62.8, 74.7, 118.5, 123.20, 123.21, 123.4, 125.1, 127.0, 127.8, 128.0, 128.1, 128.4, 128.8, 130.6, 131.4, 135.1, 137.9, 142.3, 143.3, 148.4, 171.3. HRMS (ESI) calcd for $C_{24}H_{19}NNaO$ $(M+Na)^+$ 360.1359, found 360.1351.



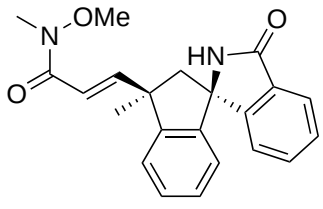
3aq

Compound 3aq. A solution of hexane/$CHCl_3$/EtOAc (1:1:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 8.1 min (major), t_2 = 14.5 min (minor); $[\alpha]^{20}_D$ –169 (*c* 0.52, $CHCl_3$) for 99% ee (1*R*,2*S*,3*S*). ^{1}H NMR ($CDCl_3$) δ 0.76 (t, *J* = 7.1 Hz, 3H), 3.50 (d, *J* = 9.1 Hz, 1H), 3.71 (q, *J* = 7.1 Hz, 2H), 4.61 (t, *J* = 9.1 Hz, 1H), 5.33 (dd, *J* = 10.0, 0.9 Hz, 1H), 5.47 (dd, *J* = 17.0, 0.9 Hz, 1H), 5.87 (ddd, *J* = 17.0, 10.0, 9.1 Hz, 1H), 6.48 (br s, 1H), 6.98–7.04 (m, 2H), 7.23 (t, *J* = 7.3 Hz, 1H), 7.30–7.38 (m, 2H), 7.39–7.46 (m, 2H), 7.82–7.86 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 13.4, 48.6, 60.6, 60.8, 72.6, 118.6, 122.9, 123.1, 123.3, 125.1, 128.2, 128.7, 129.2, 130.6, 132.0, 137.8, 141.3, 142.4, 147.8, 169.5, 170.8. HRMS (ESI) calcd for $C_{21}H_{19}NNaO_3$ $(M+Na)^+$ 356.1257, found 356.1257.



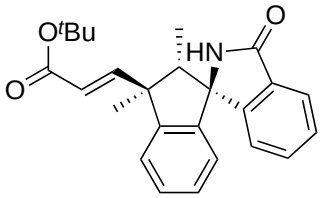
3ar

Compound 3ar. A solution of hexane/$CHCl_3$/EtOAc (1:1:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 11.3 min (minor), t_2 = 15.3 min (major); $[\alpha]^{20}_D$ +38 (*c* 0.99, $CHCl_3$) for 85% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.29 (t, *J* = 7.1 Hz, 3H), 1.65 (s, 3H), 2.60 (d, *J* = 14.0 Hz, 1H), 2.75 (d, *J* = 14.0 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 5.45 (d, *J* = 15.7 Hz, 1H), 6.21 (br s, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 7.20–7.30 (m, 4H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.58 (t, *J* = 7.3 Hz, 1H), 7.89 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.1, 26.4, 49.0, 53.2, 60.5, 69.9, 119.8, 122.3, 123.6, 124.0, 124.3, 128.5, 128.6, 129.5, 131.4, 132.5, 142.8, 147.0, 150.6, 155.7, 166.2, 168.9. HRMS (ESI) calcd for $C_{22}H_{21}NNaO_3$ $(M+Na)^+$ 370.1414, found 370.1410.



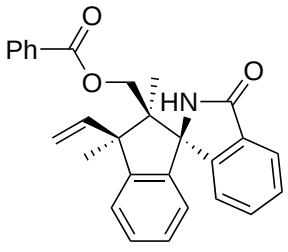
3as

Compound 3as. A solution of $CHCl_3$/EtOAc (3:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 19.0 min (minor), t_2 = 31.3 min (major); $[\alpha]^{20}_D$ +8 (*c* 1.01, $CHCl_3$) for 95% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.66 (s, 3H), 2.65 (d, *J* = 14.0 Hz, 1H), 2.75 (d, *J* = 14.0 Hz, 1H), 3.21 (s, 3H), 3.57 (s, 3H), 6.01 (d, *J* = 15.6 Hz, 1H), 6.31 (s, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 7.20–7.25 (m, 3H), 7.30 (d, *J* = 7.7 Hz, 1H), 7.40 (td, *J* = 7.5, 0.9 Hz, 1H), 7.51 (td, *J* = 7.5, 0.8 Hz, 1H), 7.57 (td, *J* = 7.5, 1.1 Hz, 1H), 7.87–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.4, 32.3, 49.1, 52.9, 61.5, 69.9, 117.3, 122.3, 123.5, 123.8, 124.2, 128.45, 128.51, 129.4, 131.5, 132.4, 142.8, 147.7, 150.7, 154.0, 166.4, 168.8. HRMS (ESI) calcd for $C_{22}H_{22}N_2NaO_3$ $(M+Na)^+$ 385.1523, found 385.1521.



3at

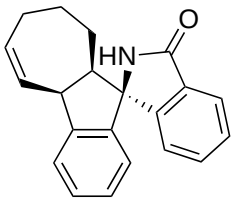
Compound 3at. A solution of $CHCl_3$/EtOAc (6:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 14.6 min (minor), t_2 = 29.3 min (major); $[\alpha]^{20}_D$ –113 (*c* 1.03, $CHCl_3$) for 90% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 0.72 (d, *J* = 7.3 Hz, 3H), 1.50 (s, 9H), 1.51 (s, 3H), 2.81 (q, *J* = 7.3 Hz, 1H), 5.86 (d, *J* = 15.7 Hz, 1H), 6.25 (br s, 1H), 6.91–6.95 (m, 1H), 7.01 (d, *J* = 15.8 Hz, 1H), 7.02–7.08 (m, 1H), 7.19–7.23 (m, 2H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.43–7.49 (m, 2H), 7.87–7.93 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 9.5, 22.6, 28.0, 49.9, 52.4, 74.9, 80.5, 121.0, 123.91, 123.94, 124.4, 124.8, 128.20, 128.23, 129.2, 131.5, 131.6, 141.6, 148.4, 148.5, 154.0, 165.9, 169.8. HRMS (ESI) calcd for $C_{25}H_{27}NNaO_3$ $(M+Na)^+$ 412.1883, found 412.1878.



3au

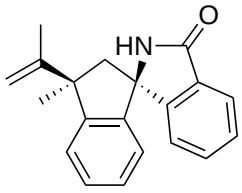
Compound 3au. A solution of hexane/EtOAc (1:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.5 min (minor), t_2 = 8.3 min (major); $[\alpha]^{27}_D$ +193 (*c* 0.97, $CHCl_3$) for 99% ee (1*R*,2*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.36 (s, 3H), 1.55 (s, 3H), 4.39 (d, *J* = 11.2 Hz, 1H), 4.48 (dd, *J* = 17.3, 1.2 Hz, 1H), 4.63 (d, *J* = 11.2 Hz, 1H), 5.21 (dd, *J* = 10.5, 1.2 Hz, 1H), 6.34 (dd,

J = 17.3, 10.5 Hz, 1H), 6.64 (br s, 1H), 6.71 (d, *J* = 7.5 Hz, 1H), 7.20–7.27 (m, 6H), 7.35–7.39 (m, 1H), 7.42 (td, *J* = 7.4, 1.1 Hz, 1H), 7.45 (tt, *J* = 7.4, 1.5 Hz, 1H), 7.57 (td, *J* = 7.4, 1.2 Hz, 1H), 7.60 (td, *J* = 7.4, 1.5 Hz, 1H), 7.84–7.88 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 19.8, 21.8, 55.1, 56.1, 56.8, 66.5, 75.2, 116.2, 124.0, 124.1, 124.9, 125.7, 128.0, 128.29, 128.32, 129.3, 129.4, 131.3, 132.8, 133.0, 142.5, 146.2, 146.7, 147.7, 165.9, 168.2. HRMS (ESI) calcd for $C_{28}H_{25}NNaO_3$ $(M+Na)^+$ 446.1727, found 446.1722.



3av

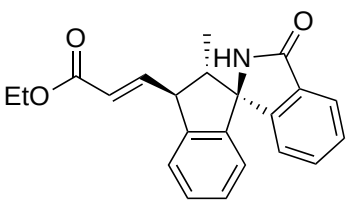
Compound 3av. The crude product was subjected to column chromatography on silica gel with EtOAc/hexane (1:1) and GPC with $CHCl_3$. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.5 min (major), t_2 = 11.3 min (minor); $[\alpha]^{25}_D$ +89 (*c* 1.23, $CHCl_3$) for 97% ee (4b*S*,9a*R*,10*R*). ^{1}H NMR ($CDCl_3$) δ 1.39–1.48 (m, 1H), 1.52–1.61 (m, 1H), 1.63–1.80 (m, 2H), 2.17–2.28 (m, 1H), 2.42–2.51 (m, 1H), 3.20 (ddd, *J* = 11.2, 8.0, 6.9 Hz, 1H), 4.41 (ddd, *J* = 11.2, 5.7, 2.8 Hz, 1H), 5.50–5.55 (m, 1H), 5.60–5.66 (m, 1H), 6.07 (br s, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 7.16 (td, *J* = 7.3, 1.3 Hz, 1H), 7.23–7.27 (m, 1H), 7.34 (td, *J* = 7.3, 0.9 Hz, 1H), 7.35–7.38 (m, 1H), 7.50 (td, *J* = 7.3, 0.7 Hz, 1H), 7.56 (td, *J* = 7.3, 1.1 Hz, 1H), 7.89 (d, *J* = 7.3 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.1, 22.7, 30.1, 44.8, 54.1, 73.2, 122.1, 123.5, 123.9, 124.6, 127.8, 128.5, 129.4, 129.7, 130.2, 132.1, 132.4, 143.1, 147.7, 149.8, 169.2. HRMS (ESI) calcd for $C_{21}H_{19}NNaO$ $(M+Na)^+$ 324.1359, found 324.1358.



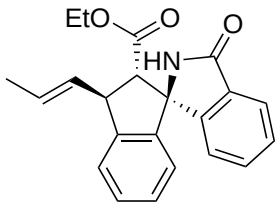
3aw

Compound 3aw. A solution of $CHCl_3$/EtOAc (6:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 6.8 min (major), t_2 = 7.5 min (minor); $[\alpha]^{20}_D$ +78 (*c* 0.96, $CHCl_3$) for 69% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.61 (s, 3H), 1.93 (s, 3H), 2.55 (d, *J* = 13.8 Hz, 1H), 2.62 (d, *J* = 13.8 Hz, 1H), 4.23 (s, 1H), 4.85 (s, 1H), 6.52 (br s, 1H), 6.68 (d, *J* = 7.4 Hz, 1H), 7.18 (t, *J* = 7.4 Hz, 1H), 7.23–7.30 (m, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.88 (d, *J* = 7.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 19.9, 25.5, 52.3, 52.4, 70.0, 113.1, 122.4, 123.5,

123.8, 124.4, 128.0, 128.4, 128.9, 131.8, 132.3, 142.6, 149.8, 150.5, 152.9, 168.7. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1358.



3ax



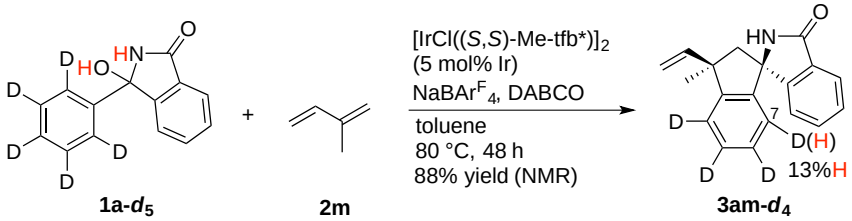
4ax

Compound 3ax. The crude product was subjected to preparative TLC six times with hexane/EtOAc (2:1) to **3ax** and **4ax**. The ee was measured by HPLC (Chiralpak IA column × 2, hexane/chloroform/ethanol = 6:2:1, flow 0.4 mL/min, 254 nm, t_1 = 27.7 min (major), t_2 = 30.7 min (minor); $[\alpha]^{20}_D$ –159 (*c* 0.66, $CHCl_3$) for 76% ee (1*R*,2*S*,3*S*). ^{1}H NMR ($CDCl_3$) δ 0.80 (d, *J* = 6.8 Hz, 3H), 1.33 (t, *J* = 7.1 Hz, 3H), 2.60–2.70 (m, 1H), 3.84 (t, *J* = 9.6 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 6.13 (d, *J* = 15.5 Hz, 1H), 6.41 (br s, 1H), 6.93 (dd, *J* = 15.3, 9.6 Hz, 1H), 6.94 (d, *J* = 6.0 Hz, 1H), 7.05 (d, *J* = 7.3 Hz, 1H), 7.20–7.26 (m, 2H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.40–7.48 (m, 2H), 7.85–7.92 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 11.6, 14.2, 51.6, 52.5, 60.6, 73.6, 122.9, 123.2, 124.2, 124.4, 125.1, 128.4, 128.5, 129.0, 130.7, 132.0, 142.0, 143.4, 147.6, 148.2, 166.0, 170.7. HRMS (ESI) calcd for $C_{22}H_{21}NNaO_3$ $(M+Na)^+$ 370.1414, found 370.1415.

Compound 4ax. The ee was measured by HPLC (Chiralpak IA column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 7.5 min (major), t_2 = 12.9 min (minor); $[\alpha]^{25}_D$ –144 (*c* 0.63, $CHCl_3$) for 99% ee (1*R*,2*S*,3*S*). ^{1}H NMR ($CDCl_3$) δ 0.76 (t, *J* = 7.1 Hz, 3H), 1.79 (dd, *J* = 6.5, 1.6 Hz, 3H), 3.44 (d, *J* = 9.7 Hz, 1H), 3.70 (q, *J* = 7.1 Hz, 2H), 4.56 (dd, *J* = 9.7, 8.4 Hz, 1H), 5.47 (ddq, *J* = 15.1, 8.4, 1.5 Hz, 1H), 5.90 (dq, *J* = 15.1, 9.7, 1.5 Hz, 1H), 6.46 (s, 1H), 6.97 (d, *J* = 7.7 Hz, 1H), 6.99–7.04 (m, 1H), 7.19–7.23 (m, 1H), 7.30 (d, *J* = 7.4 Hz, 1H), 7.32–7.37 (m, 1H), 7.39–7.45 (m, 2H), 7.81–7.86 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 13.5, 18.0, 47.7, 60.6, 61.4, 72.6, 123.0, 123.1, 123.4, 125.3, 128.1, 128.7, 129.2, 129.7, 130.5, 130.8, 132.1, 141.1, 143.3, 147.8, 169.6, 170.6. HRMS (ESI) calcd for $C_{22}H_{21}NNaO_3$ $(M+Na)^+$ 370.1414, found 370.1408.

8. Deuterium-labeling experiments

8-1. The reaction of 1a-*d*$_5$ with 2m

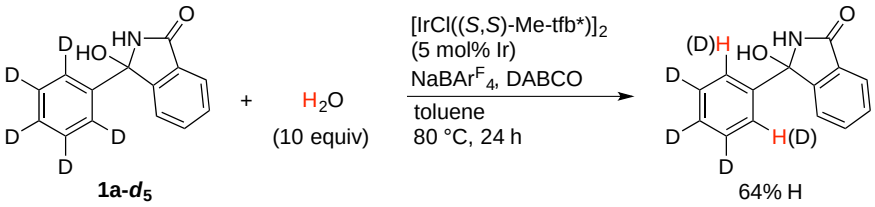


1a-*d*$_5$ **2m** **3am-*d*$_4$**

Compound **1a-*d*$_5$** (46.0 mg, 0.200 mmol), $NaBAr^F_4$ (18.4 mg calculated as the dihydrate,

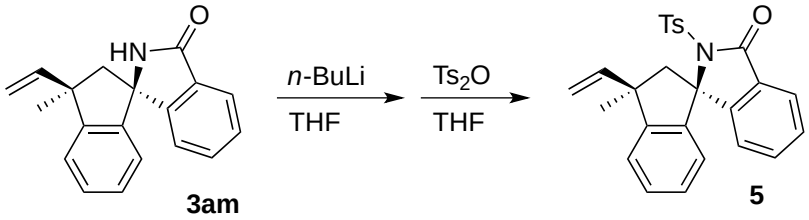
0.020 mmol, 10 mol%), and [IrCl((*S*,*S*)-Me-tfb*)]$_2$ (4.8 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL), isoprene (**2m**: 30 μL, 0.30 mmol), and a solution of DABCO (0.25 M in toluene, 40 μL, 0.010 mmol, 5 mol%) were added successively. The Schlenk tube was capped with a glass stopper and heated at 80 °C for 48 h with stirring. The mixture was passed through a short column of alumina with EtOAc as eluent, and the solvent was removed on a rotary evaporator. A hydrogen content of the annulation product **3am-*d*$_4$** at the C7 position was determined to be 13% by ^{1}H NMR of the crude mixture, and the yield was estimated to be 88% using 1,4-dimethoxybenezene as an internal standard.

8-2. The reaction of 1a-*d*$_5$ with H$_2$O in the absence of 2m



Compound **1a-*d*$_5$** (23.0 mg, 0.100 mmol), NaBAr$^F_4$ (9.2 mg calculated as the dihydrate, 0.010 mmol, 10 mol%), and [IrCl((*S*,*S*)-Me-tfb*)]$_2$ (2.4 mg, 0.0050 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.4 mL), H$_2$O (18 μL, 1.0 mmol), and a solution of DABCO (0.25 M in toluene, 20 μL, 0.0050 mmol, 5 mol%) were added successively. The Schlenk tube was capped with a glass stopper and heated at 80 °C for 24 h with stirring. The mixture was passed through a short column of silica gel with EtOAc as eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (2:1) to give **1a** (31%). A hydrogen content of the recovered **1a** at the *ortho*-position was determined to be 64% by ^{1}H NMR.

9. Transformation of 3am into 5



To a solution of **3am** (44.2 mg, 0.16 mmol, 91% ee) in THF (1.0 mL) was added *n*-BuLi (1.58 M in *n*-hexane, 152 μL, 0.24 mmol) at –78 °C, and the mixture was stirred at –78 °C for 2 h. Ts$_2$O (100 mg, 0.31 mmol) in THF (1.0 mL) was added at –78 °C, then the mixture was stirred at room temperature for 12 h. H$_2$O was added to the mixture and it was extracted with Et$_2$O. The organic layer was washed with brine, dried over Na$_2$SO$_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/CHCl$_3$/EtOAc (3:1:1) to give analytically pure **5** (23.6 mg, 0.0549mmol, 34% yield, 92% ee). The ee was

measured by HPLC (Chiralpak IC column, hexane/chloroform/ethanol = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 9.7 min (minor), t_2 = 10.9 min (major); $[\alpha]^{20}_D$ –80 (*c* 0.50, $CHCl_3$) for 92% ee (1*R*,3*R*). ^{1}H NMR ($CDCl_3$) δ 1.67 (s, 3H), 2.40 (s, 3H), 2.60 (d, *J* = 15.0 Hz, 1H), 3.56 (d, *J* = 15.0 Hz, 1H), 5.18 (dd, *J* = 10.6, 1.1 Hz, 1H), 5.31 (*J* = 17.4, 1.1 Hz, 1H), 6.48–6.52 (m, 1H), 6.61 (dd, *J* = 17.4, 10.6 Hz, 1H), 7.08–7.12 (m, 2H), 7.22–7.28 (m, 3H), 7.35–7.40 (m, 1H), 7.42 (td, *J* = 7.5, 0.8 Hz, 1H), 7.53–7.58 (m, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.79–7.82 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 21.6, 28.3, 49.0, 52.1, 79.2, 111.1, 122.5, 123.9, 124.3, 124.6, 126.9, 127.4, 128.6, 128.96, 128.98, 129.6, 134.7, 136.4, 140.3, 144.9, 146.2, 151.1, 153.0, 166.6. HRMS (ESI) calcd for $C_{26}H_{23}NNaO_3S$ $(M+Na)^+$ 452.1291, found 452.1278.

10. Data for X-ray crystal structure of 5

A colorless crystal of **5** suitable for X-ray crystallographic analysis was obtained by recrystallization from Et_2O/hexane. The ORTEP drawing of **5** is shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 943078). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

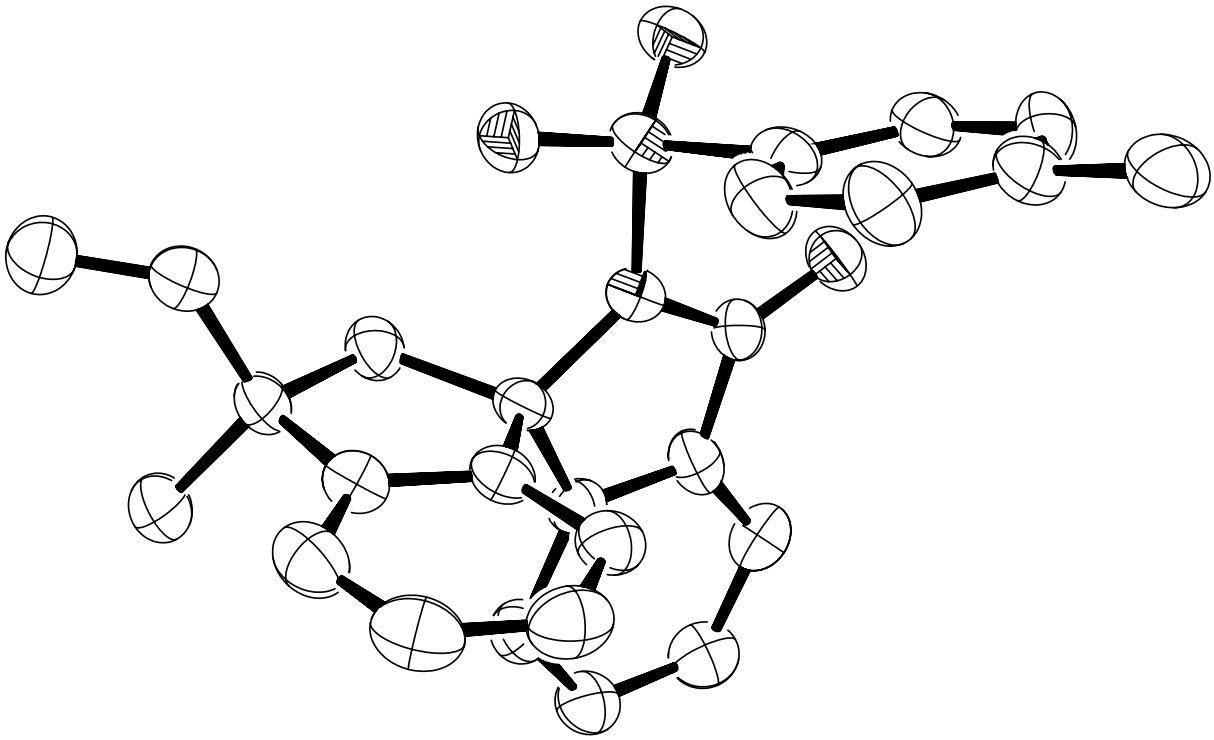


Figure S1. ORTEP illustration of **5** with thermal ellipsoids drawn at 50% probability level (hydrogen atoms are omitted for clarity).

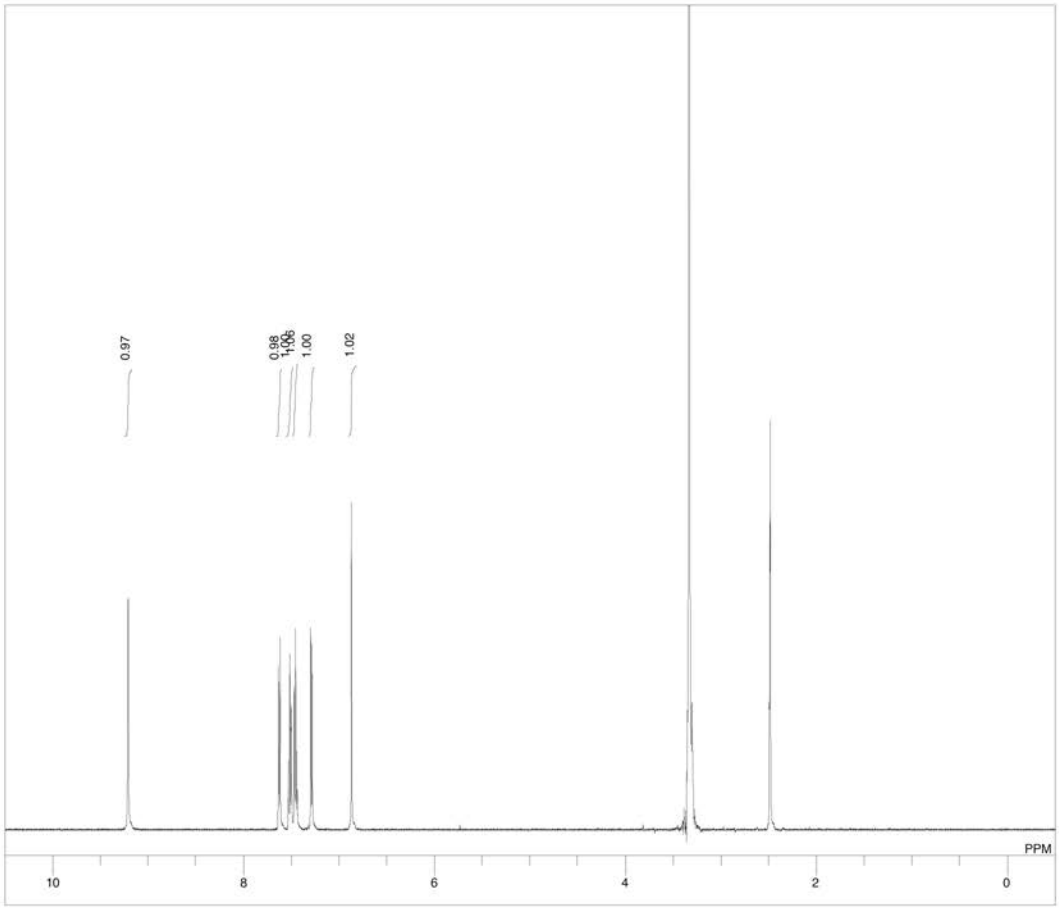
X-Ray data were collected on a Rigaku RAXIS-RAPID imaging plate diffractometer using a graphite monochromater with Cu-*K*α radiation (λ = 1.54187 Å) at 93 K. The structure was solved by direct method (SHELXS-97) and refined with full-matrix least-square technique (SHELXL-97).[15] The data for **5** is summarized in Table S1.

Table S1. Crystal data and structure refinement for **5**.

Empirical formula	$C_{26}H_{23}NO_3S$
Formula weight	429.51
Temperature	93(2) K
Crystal system	Monoclinic
Space group	*P* 2_1 (#4)
Unit cell dimensions	a = 8.6436(3) Å
	b = 12.2137(4) Å
	c = 10.5880(3) Å
	β = 100.721(2)°
Volume	1098.27(6) $Å^3$
Z	2
Density (calculated) [Mg/m^3]	1.299
μ (mm^{-1})	1.531
F(000)	452
Reflections collected	10421
Independent reflections	3740 [*R*(int) = 0.0753]
Completeness to θ (%)	98.4%
Goodness-of-fit	1.004
R_1 [$I>2\sigma(I)$]	0.0592
wR_2 (all data)	0.1877
Flack parameter	0.01(3)
Largest diff. peak and hole [$e^-/Å^{-3}$]	0.390 and – 0.416

15 G. M. Sheldrick, Program for the solution and refinement of crystal structures, University of Göttingen, Göttingen, Germany, 1997.

11. ^{1}H and ^{13}C NMR spectra and chiral HPLC charts



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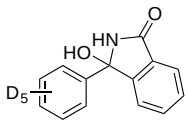
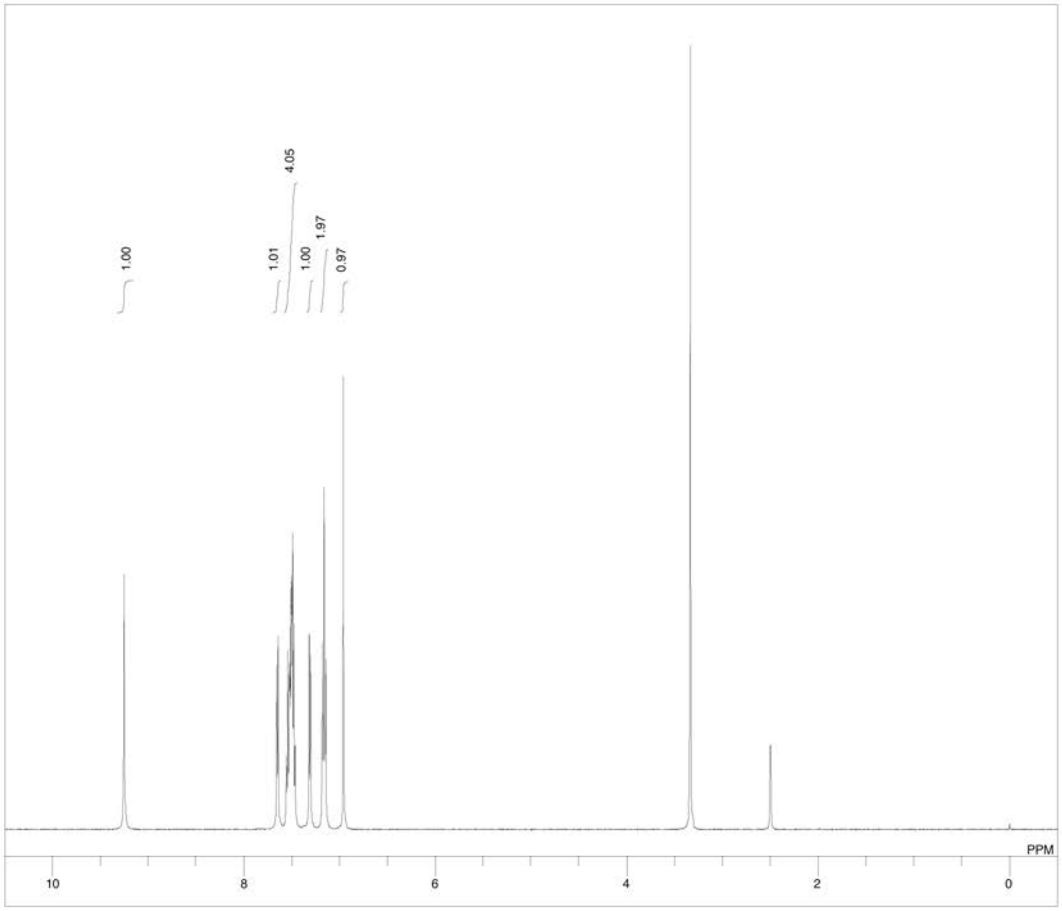


1a-*d*$_5$



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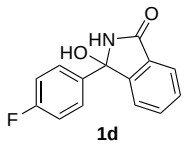

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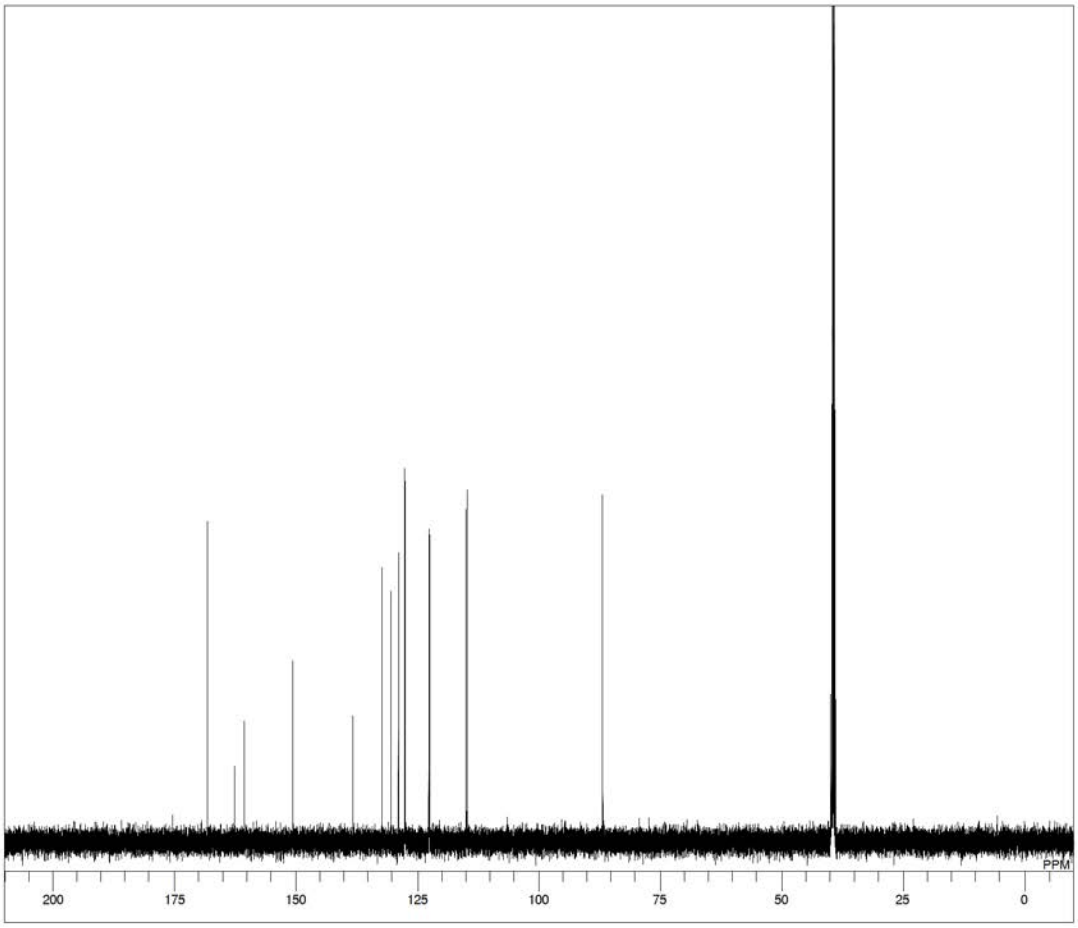

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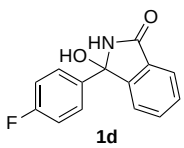

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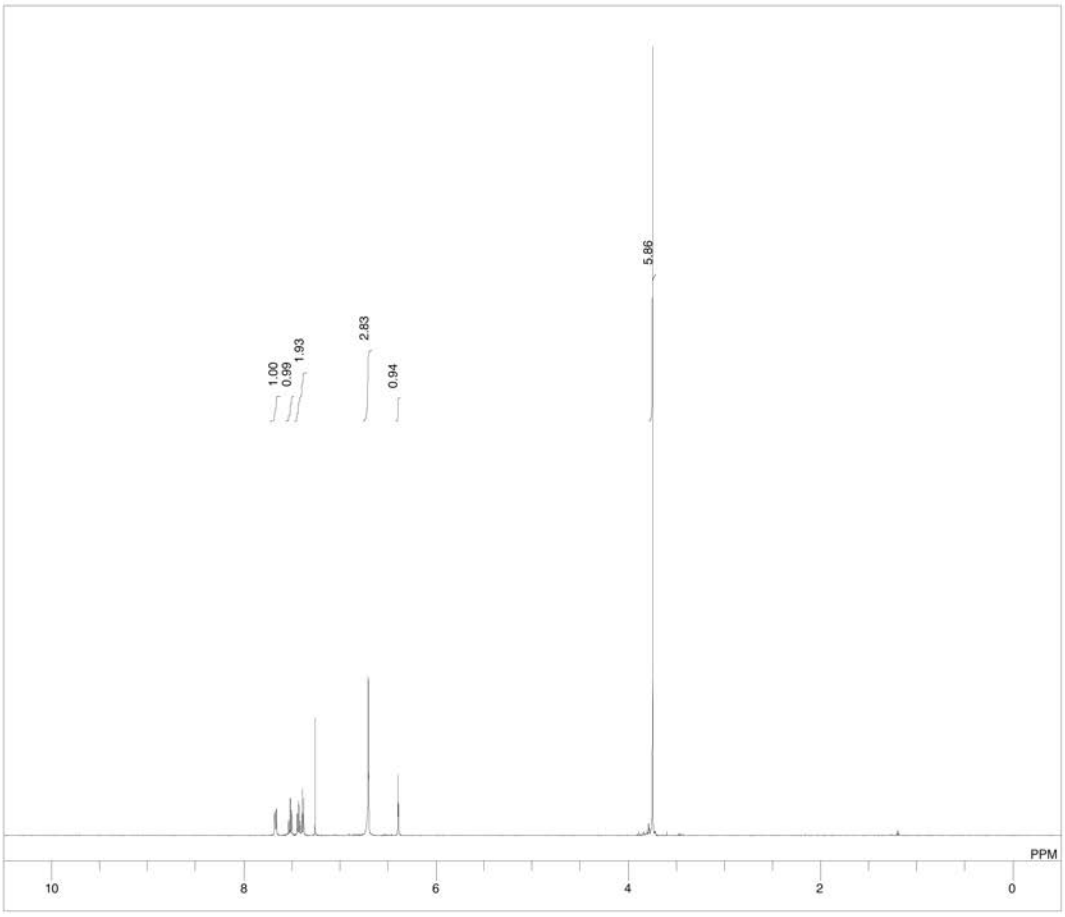

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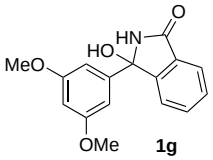

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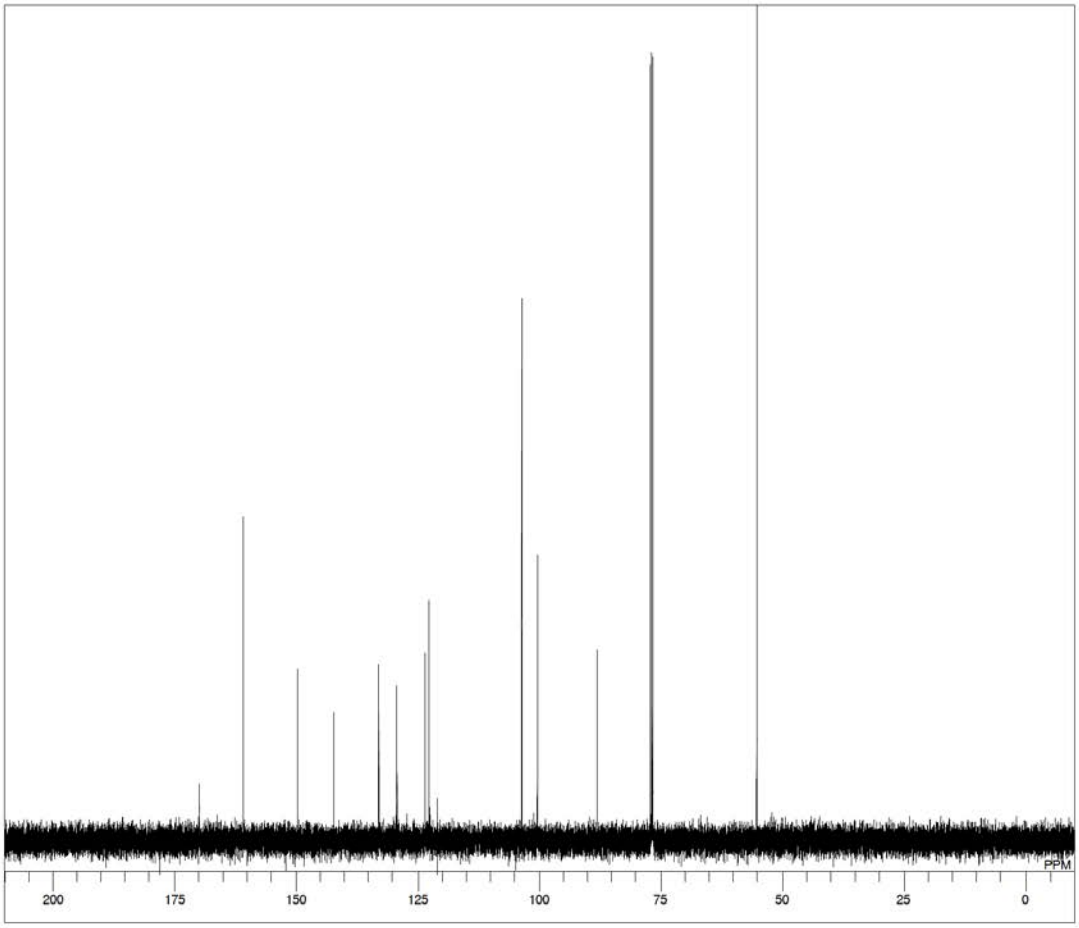

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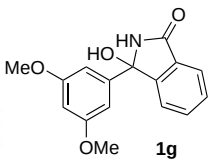

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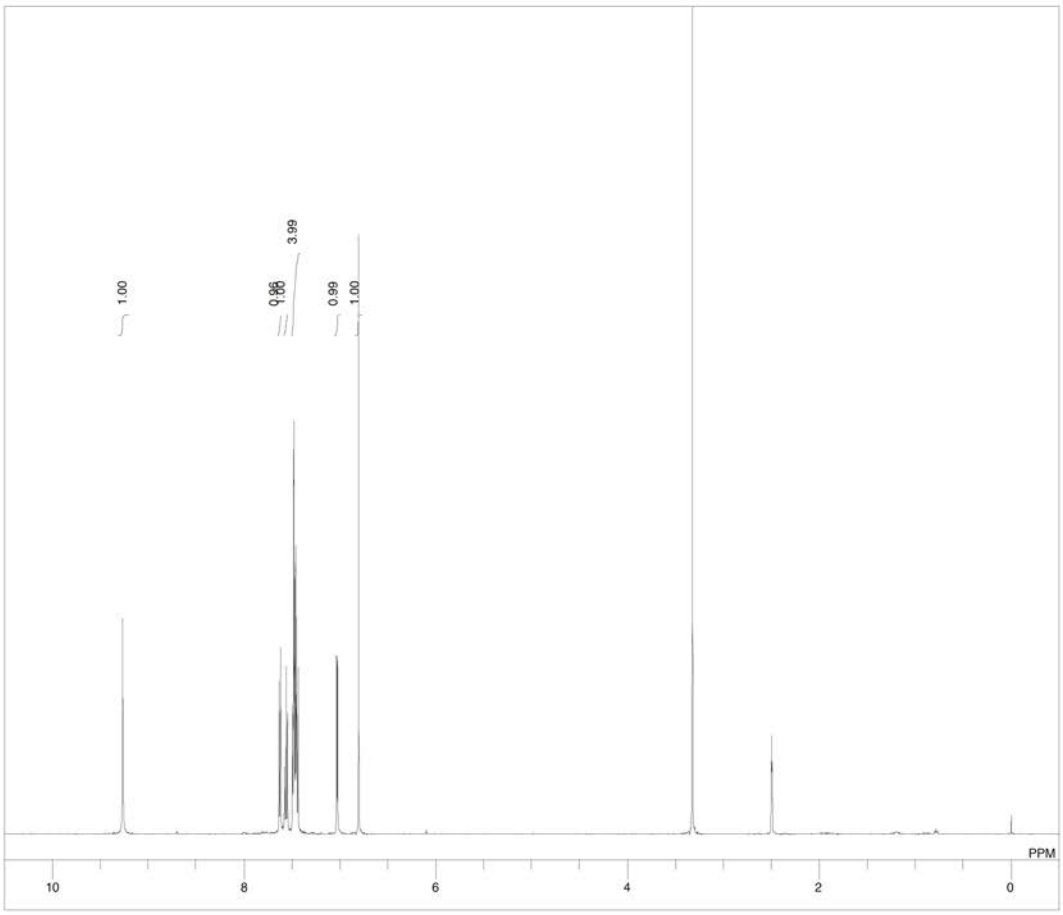

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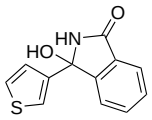

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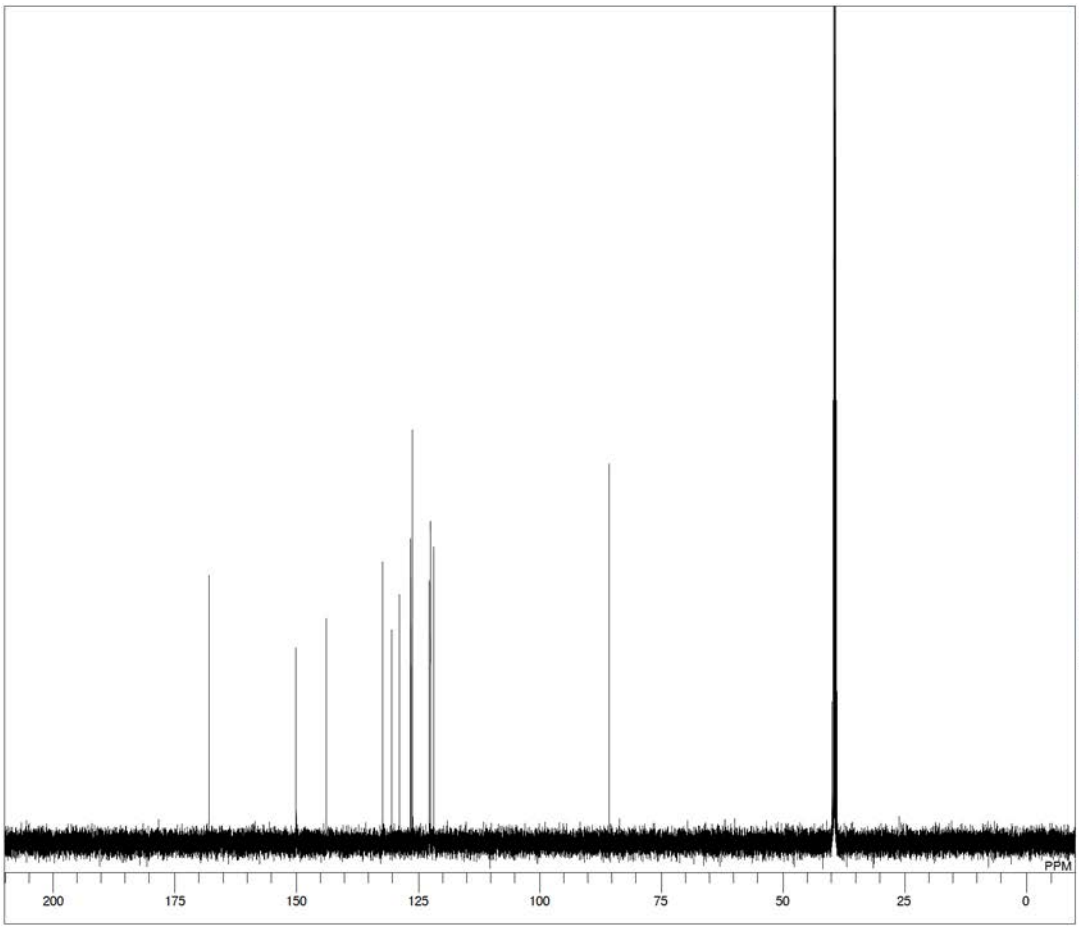

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RGAIN	22
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
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IFR	499.10 MHz
IRSET	0.00 KHz
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IRRPW	50 usec
IRATN	511
DFILE	mn-2-157-carbon-DMSO
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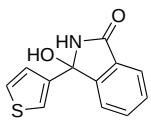
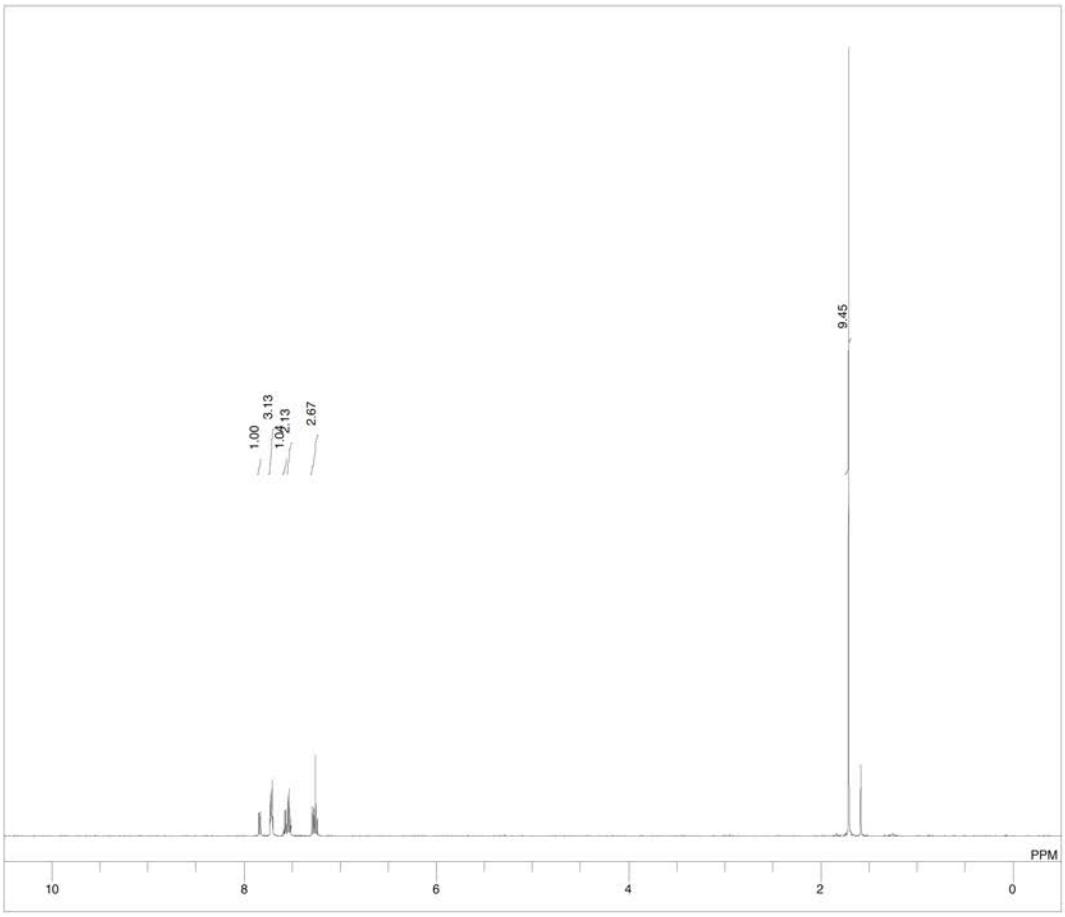
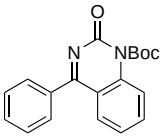


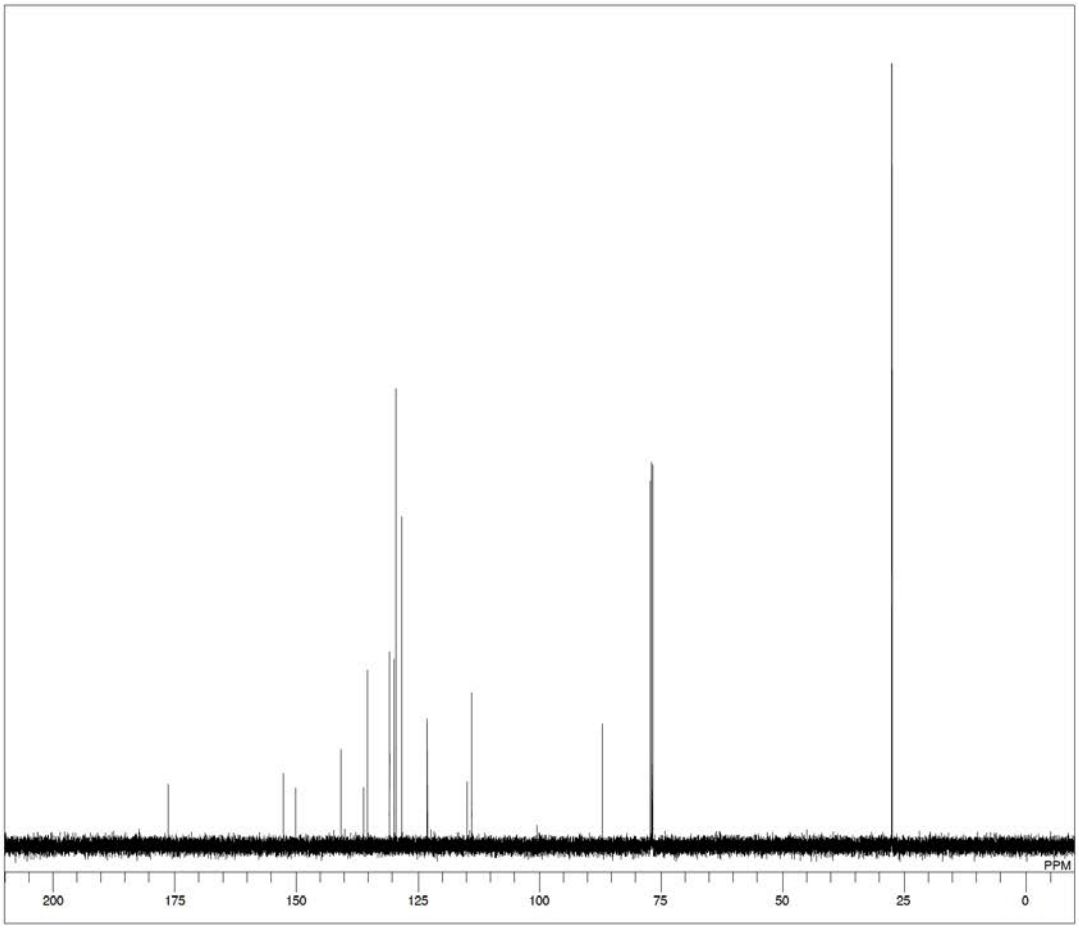
1k



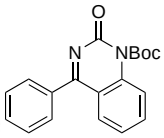
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	17
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-9.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1057
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



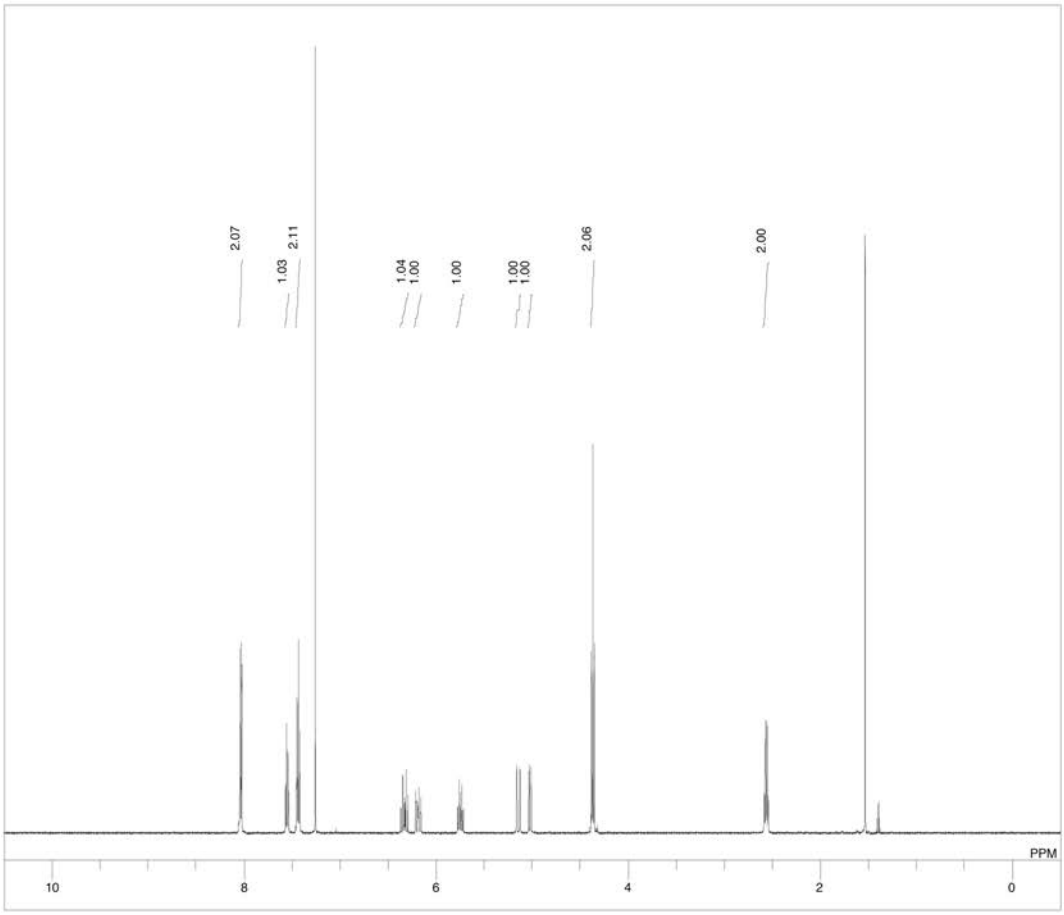
6



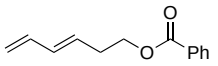
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-9-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	755
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



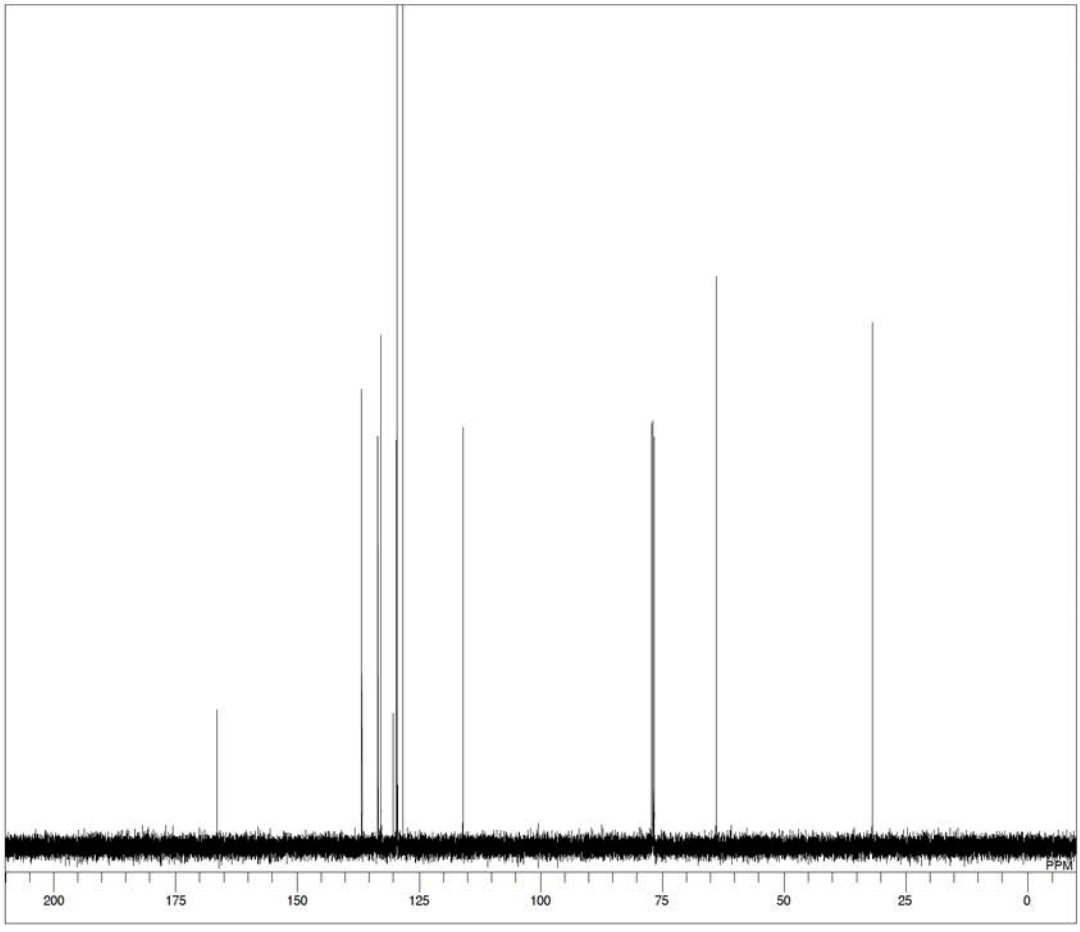
6



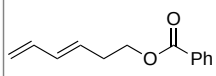
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	22
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	diene.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	860
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



2o



MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	diene-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	640
CSPED	10 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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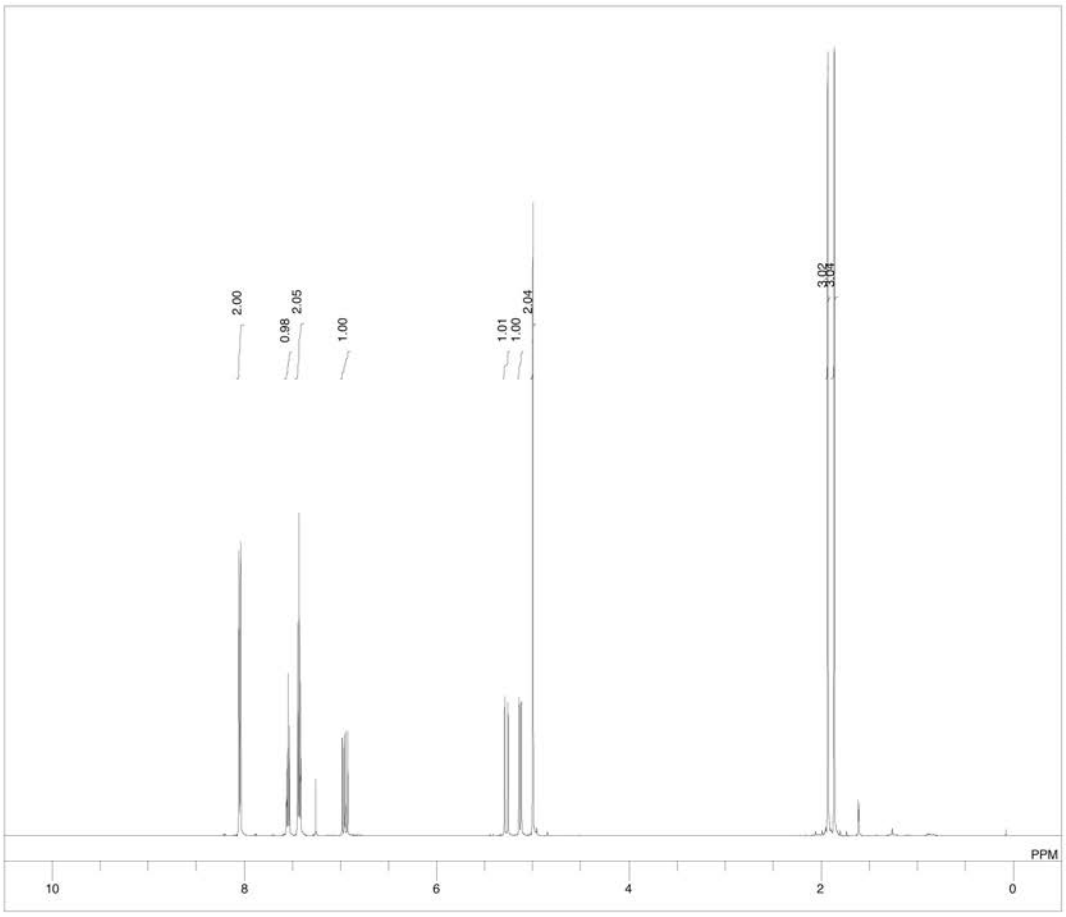
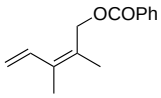
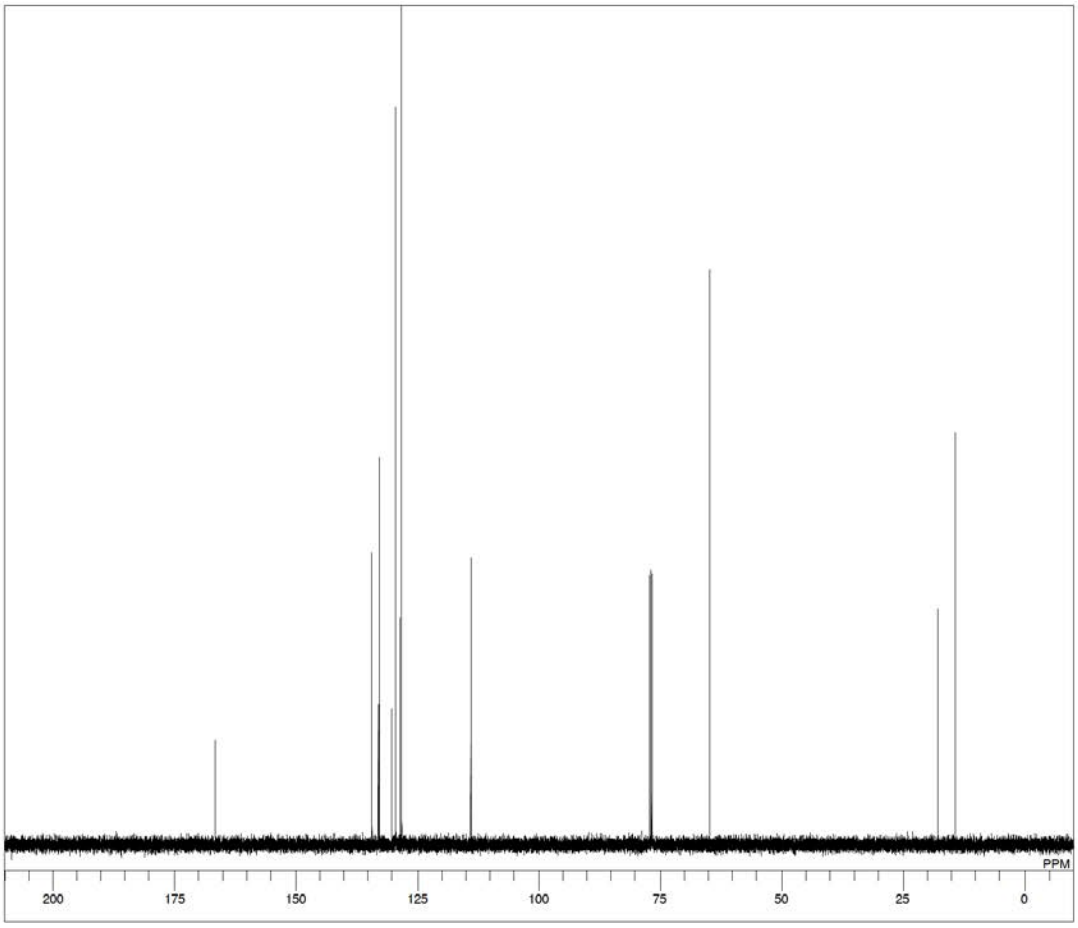


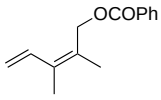
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	12
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-183.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1010
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



2u



MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	25
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-183-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	730
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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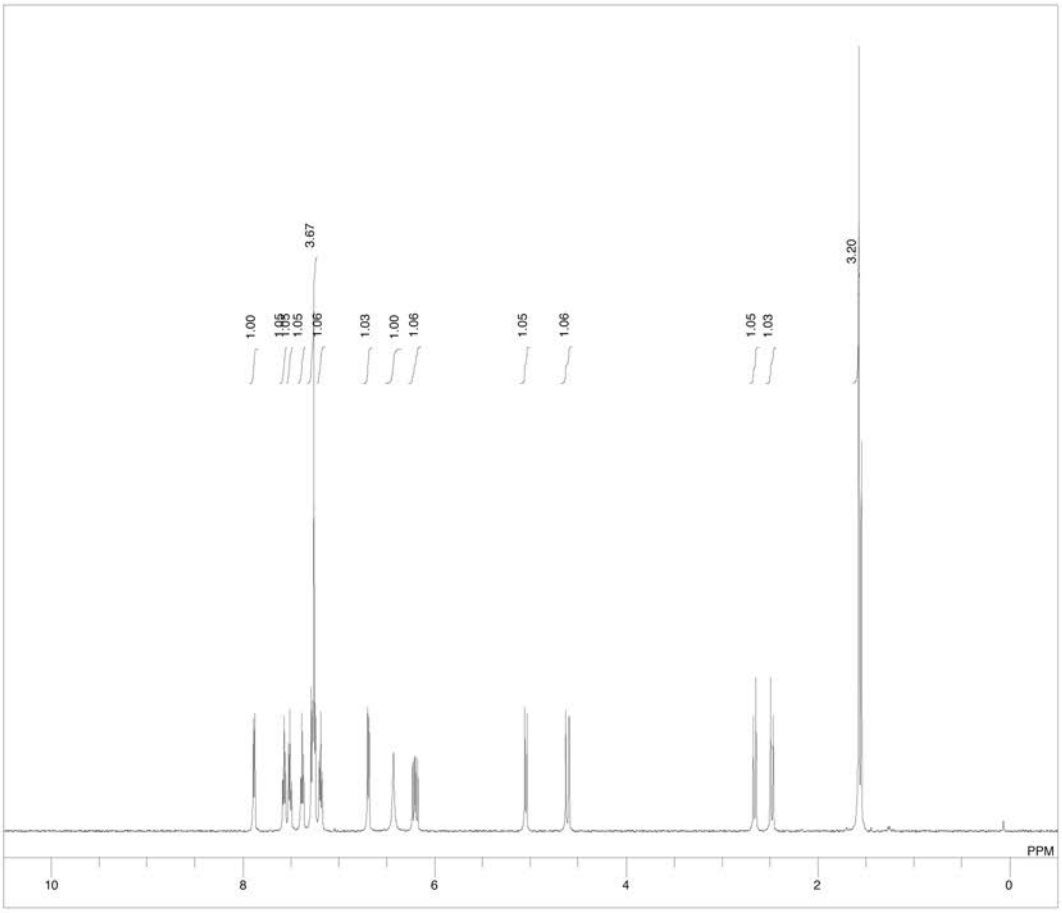
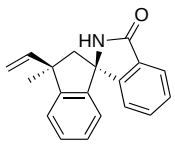
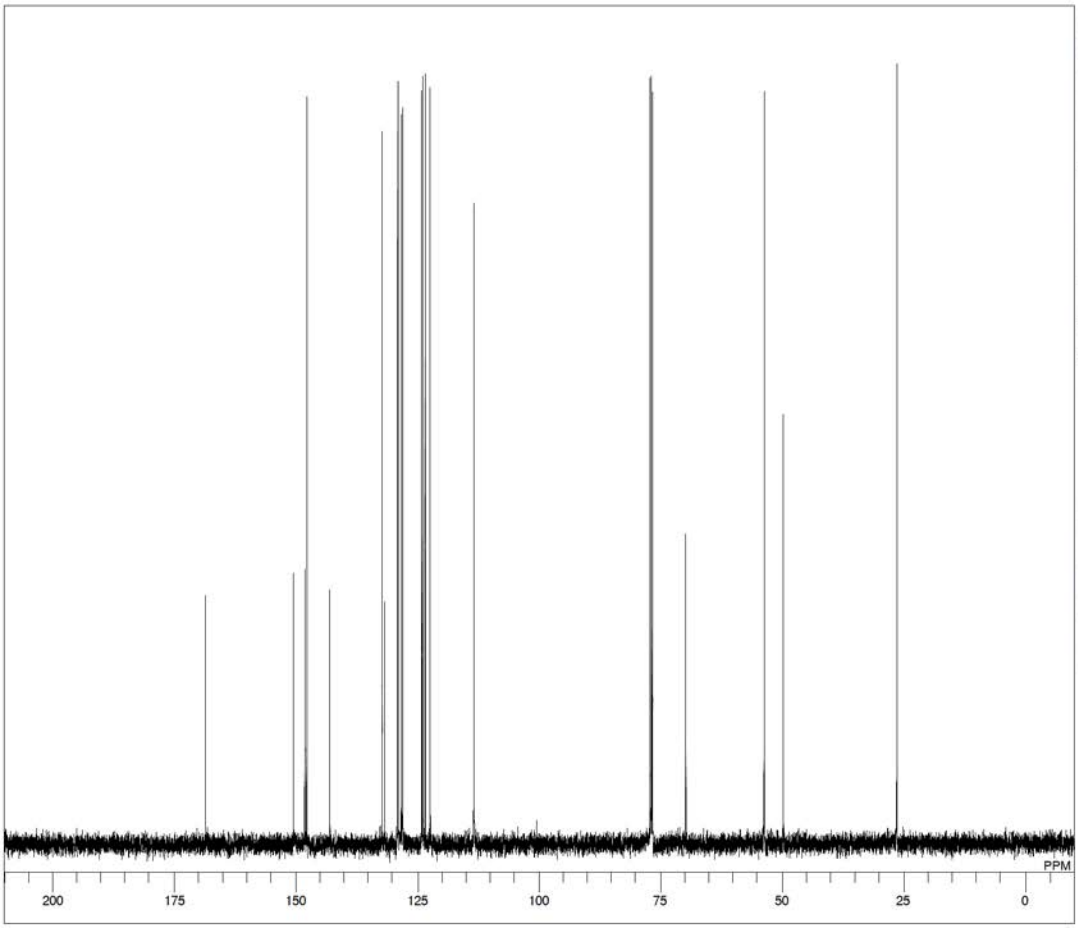


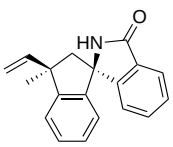
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	23
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-42.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	833
CSPED	15 Hz
FILDC	
FILDF	
SLVNT	CDCL3



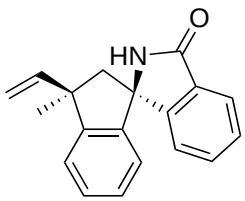
3am



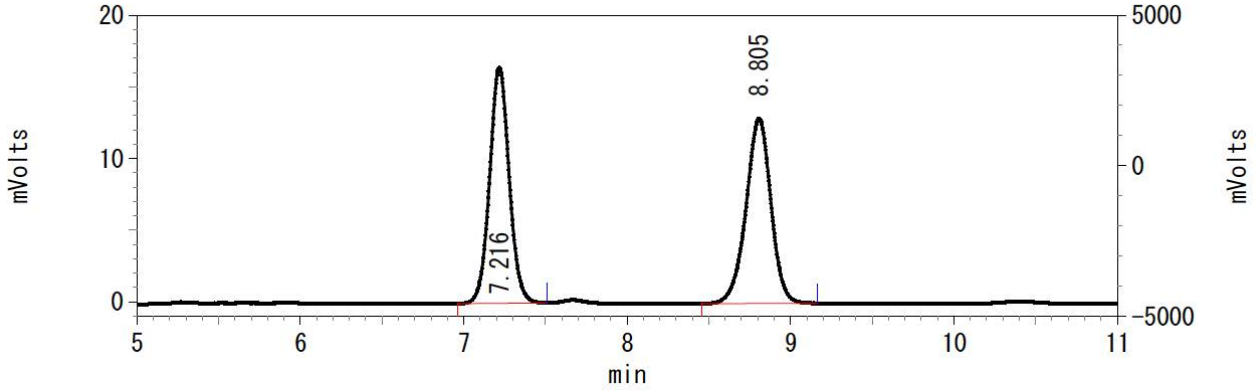
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-42-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	624
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



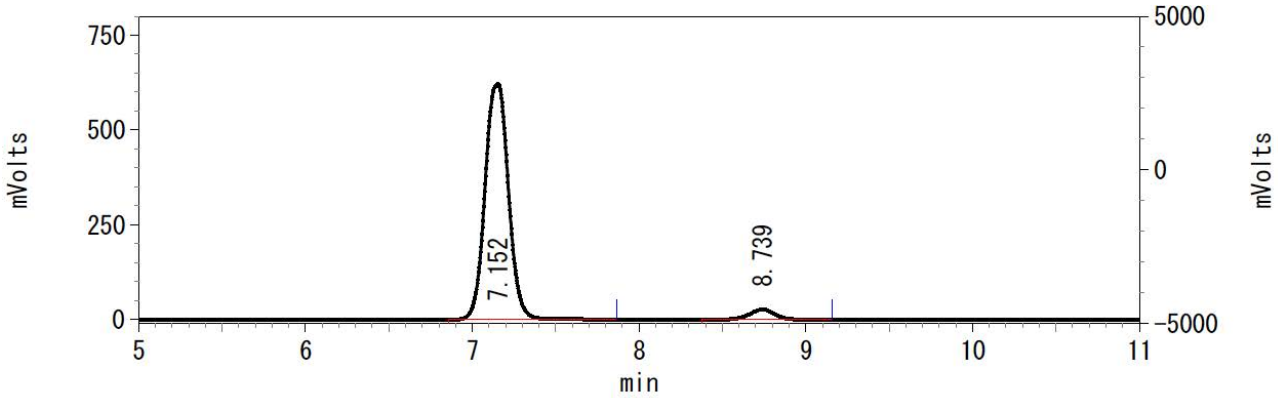
3am



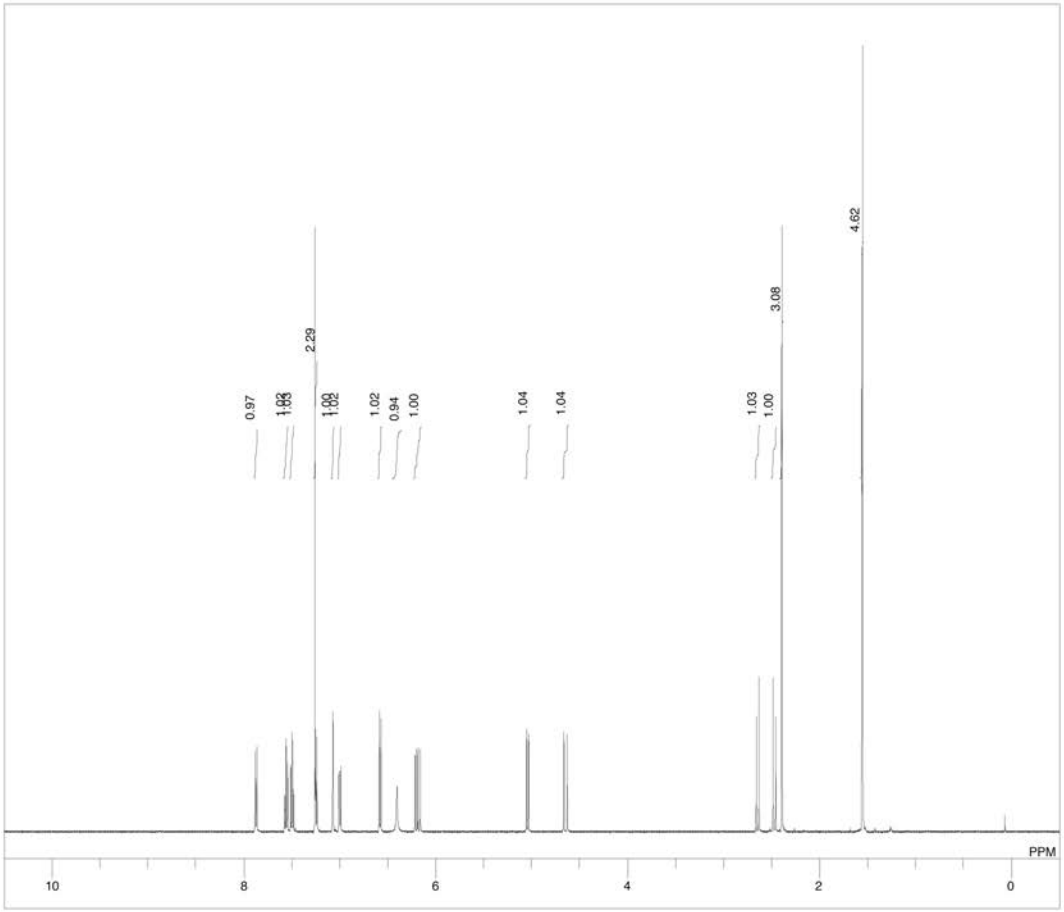
3am



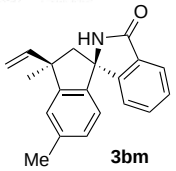
Pk #	Retention Time	Area	Area Percent
1	7.216	137175	50.003
2	8.805	137159	49.997



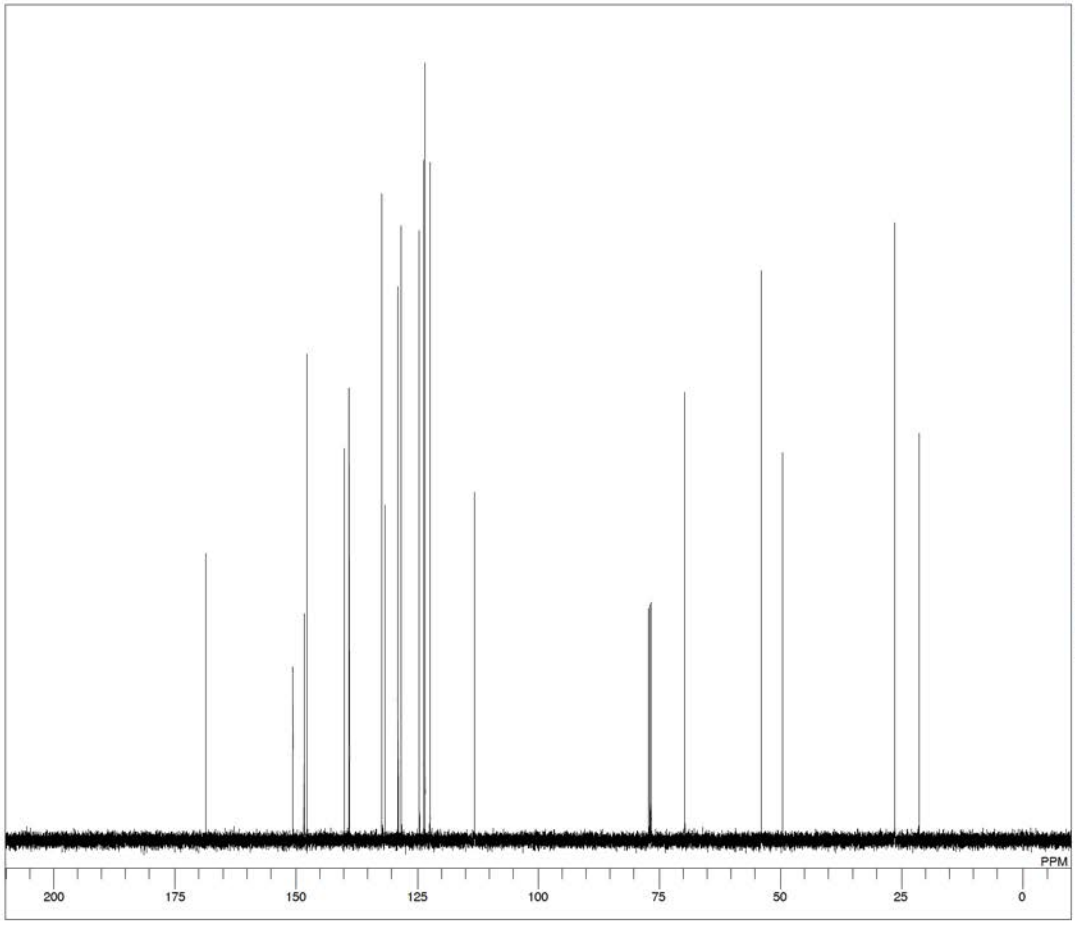
Pk #	Retention Time	Area	Area Percent
1	7.152	6066940	95.381
2	8.739	293790	4.619



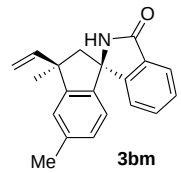
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	21
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-48.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1089
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



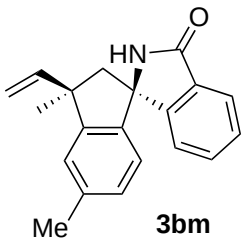
3bm



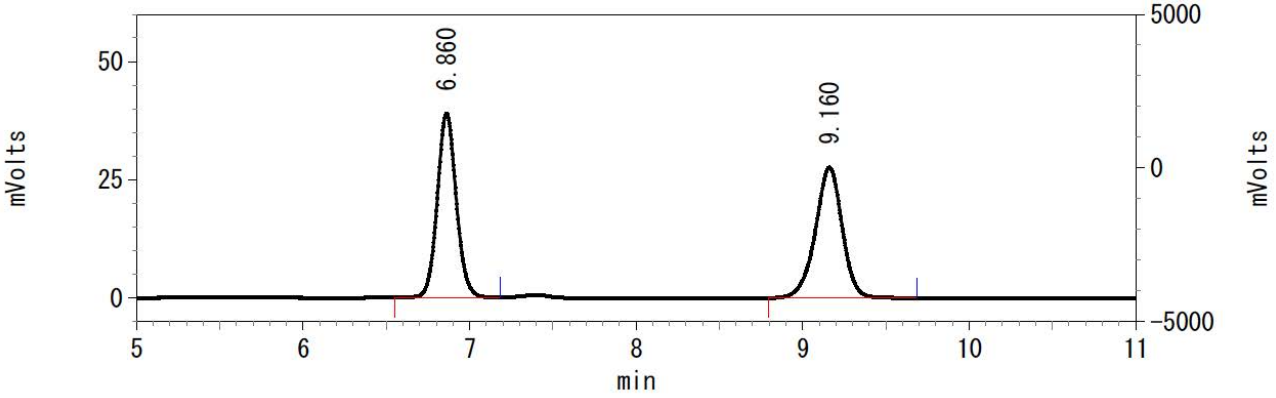
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-48-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	709
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



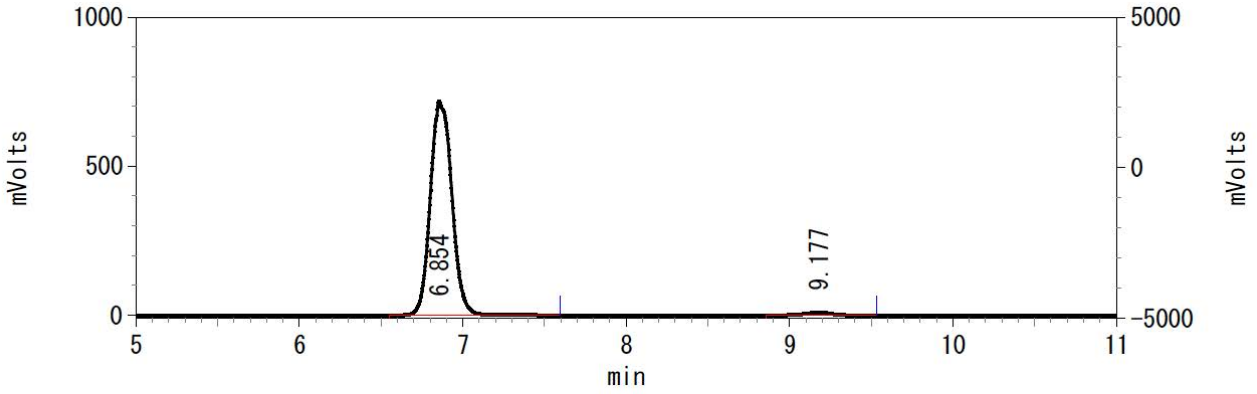
3bm



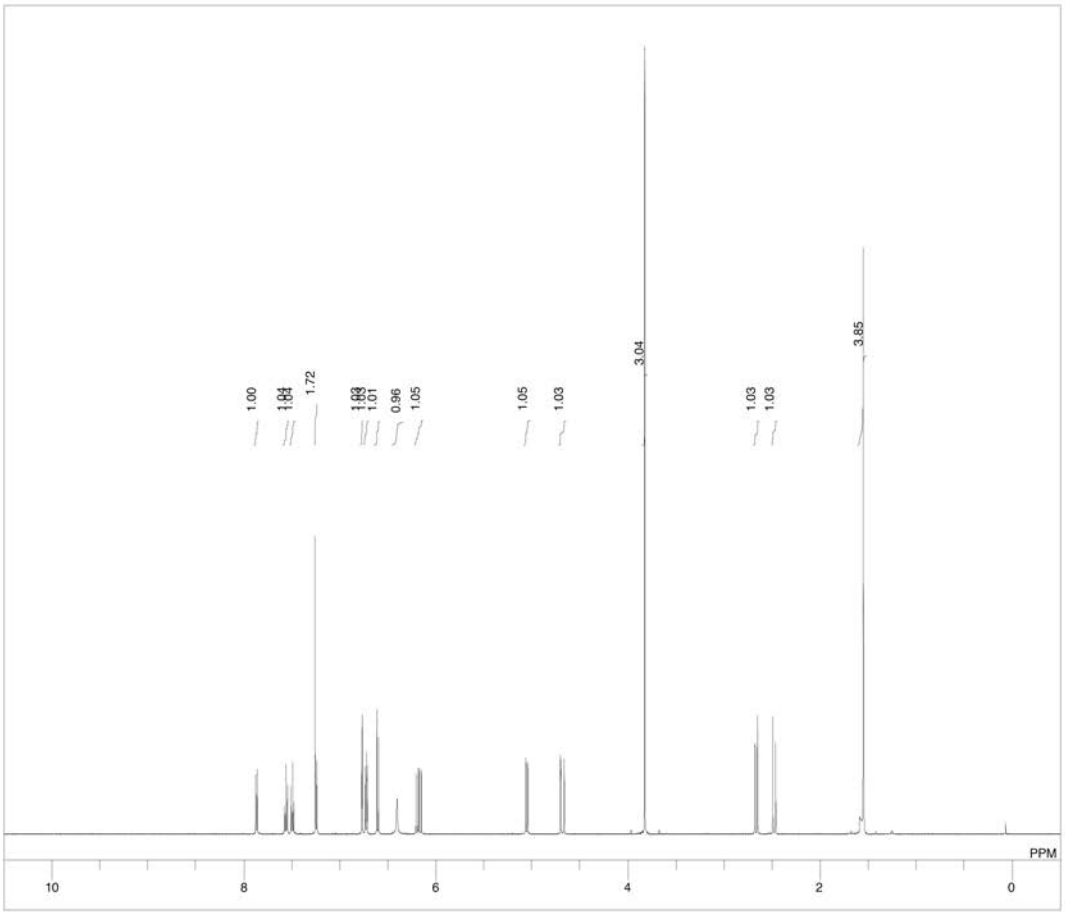
3bm



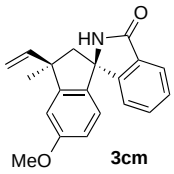
Pk #	Retention Time	Area	Area Percent
1	6.860	309969	49.983
2	9.160	310179	50.017



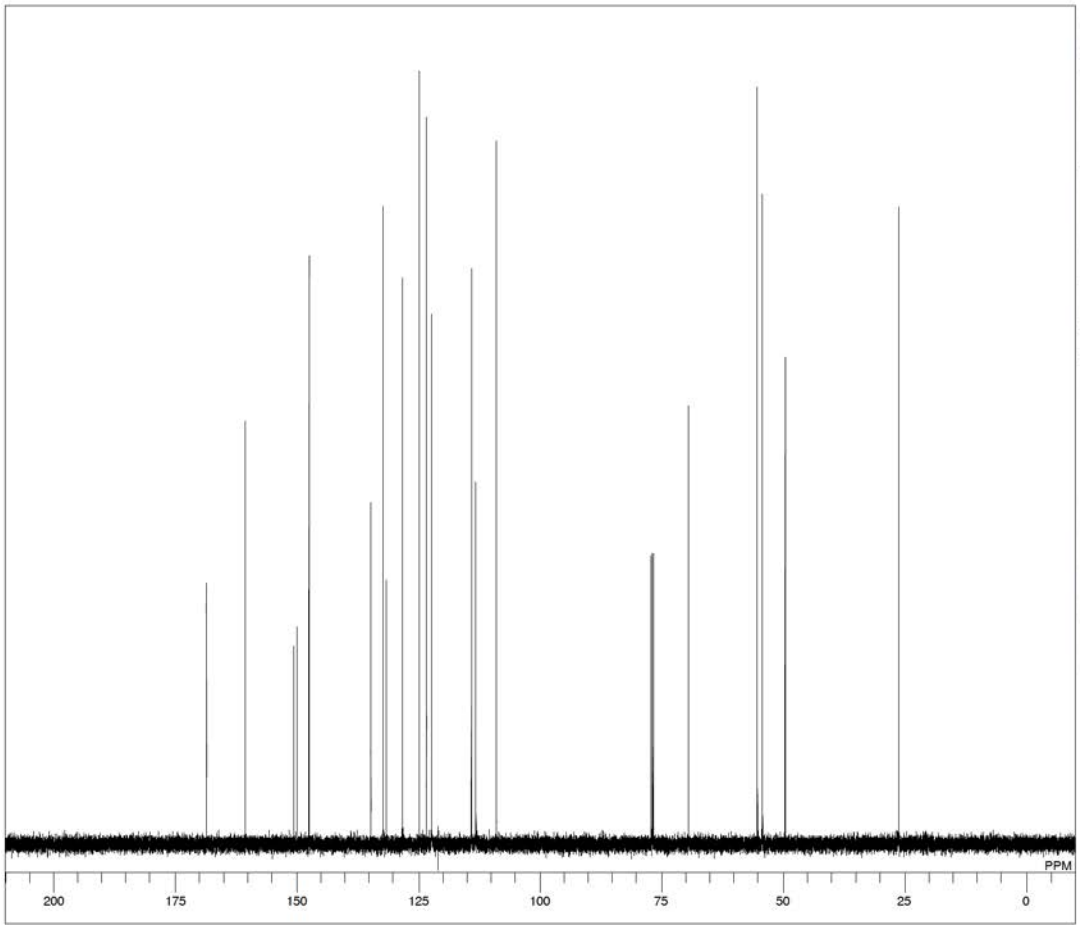
Pk #	Retention Time	Area	Area Percent
1	6.854	6583201	98.395
2	9.177	107366	1.605



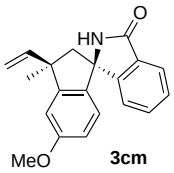
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-50'.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1093
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



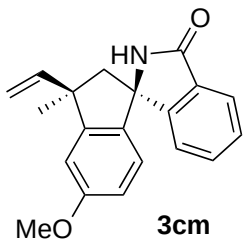
3cm



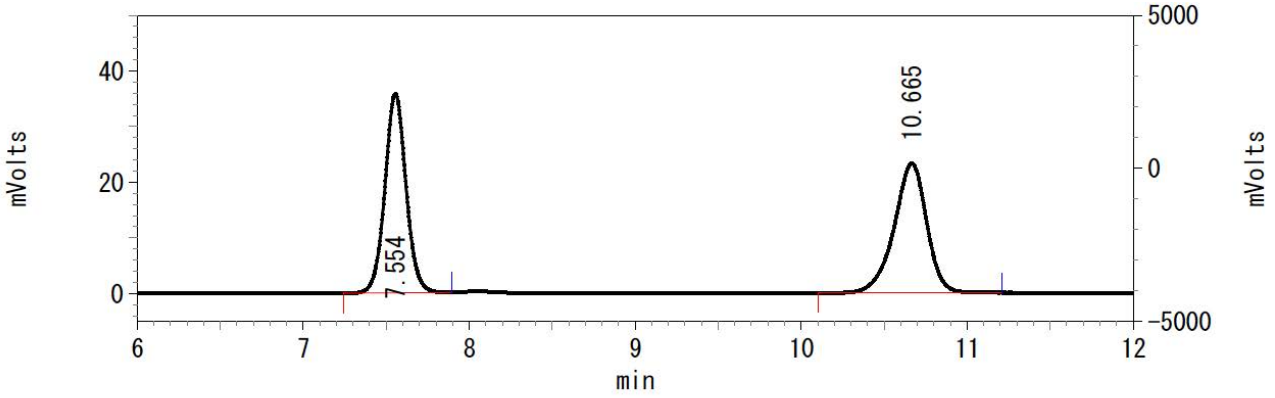
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-50-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	543
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



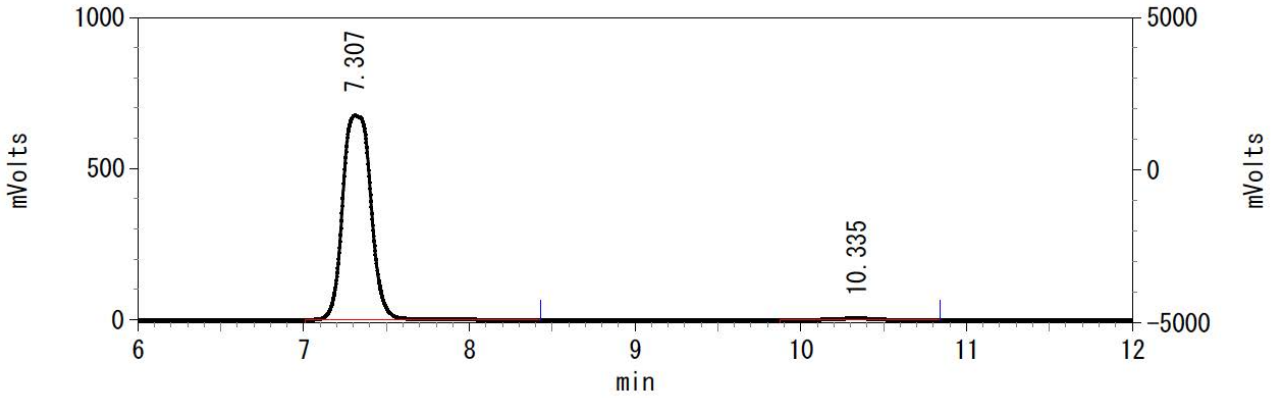
3cm



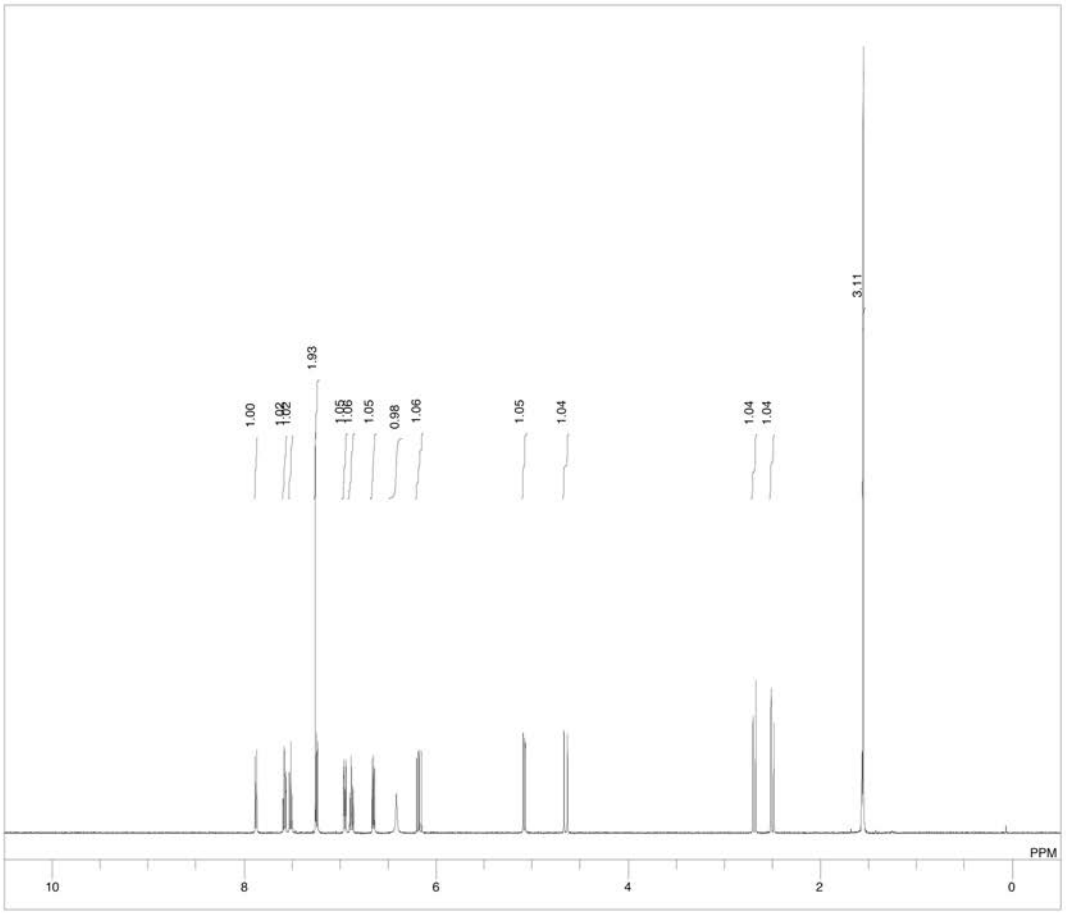
3cm



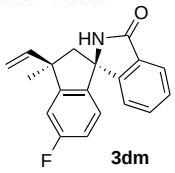
Pk #	Retention Time	Area	Area Percent
1	7.554	316710	49.899
2	10.665	317997	50.101



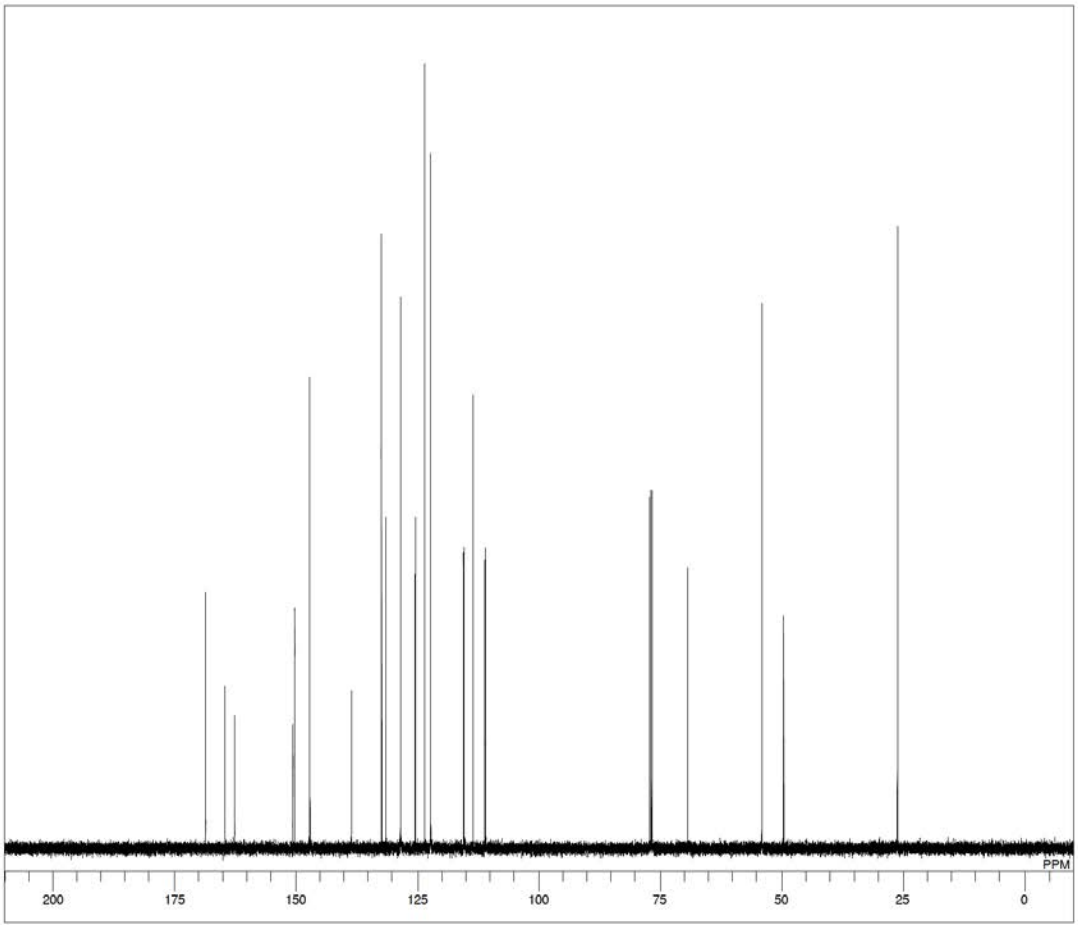
Pk #	Retention Time	Area	Area Percent
1	7.307	7982491	99.150
2	10.335	68434	0.850



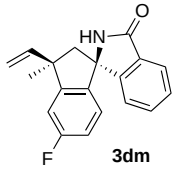
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	21
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-133.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	883
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



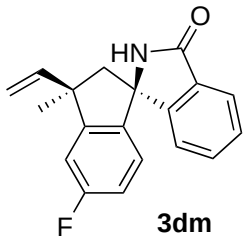
3dm



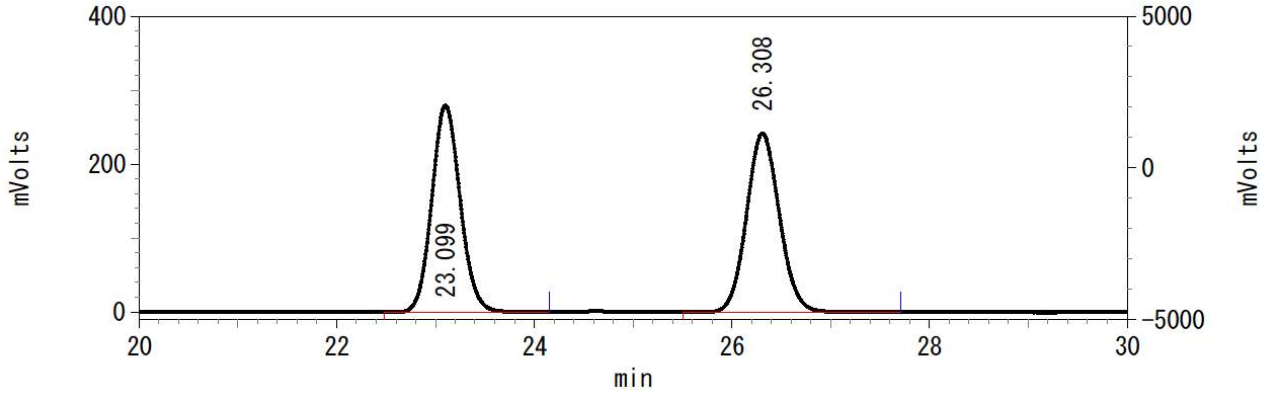
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-133-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	690
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



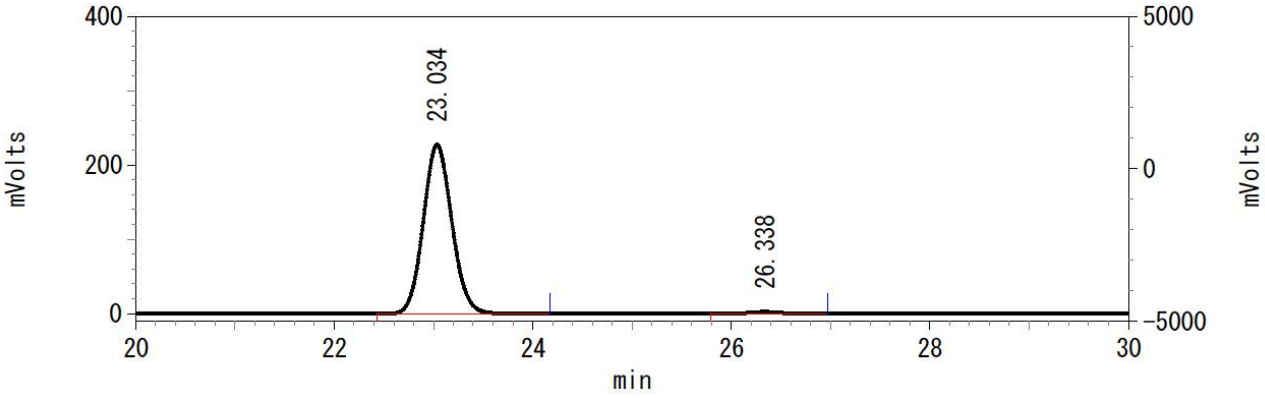
3dm



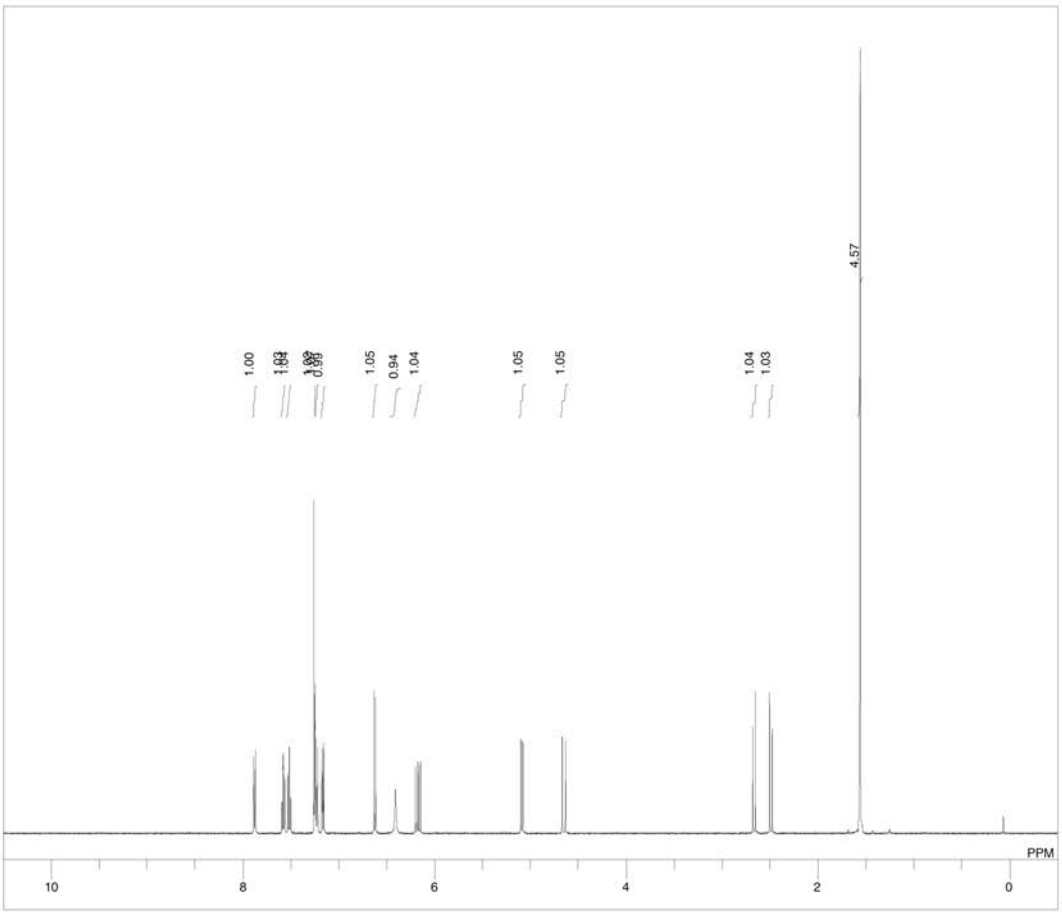
3dm



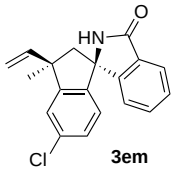
Pk #	Retention Time	Area	Area Percent
1	23.099	5604798	49.913
2	26.308	5624288	50.087



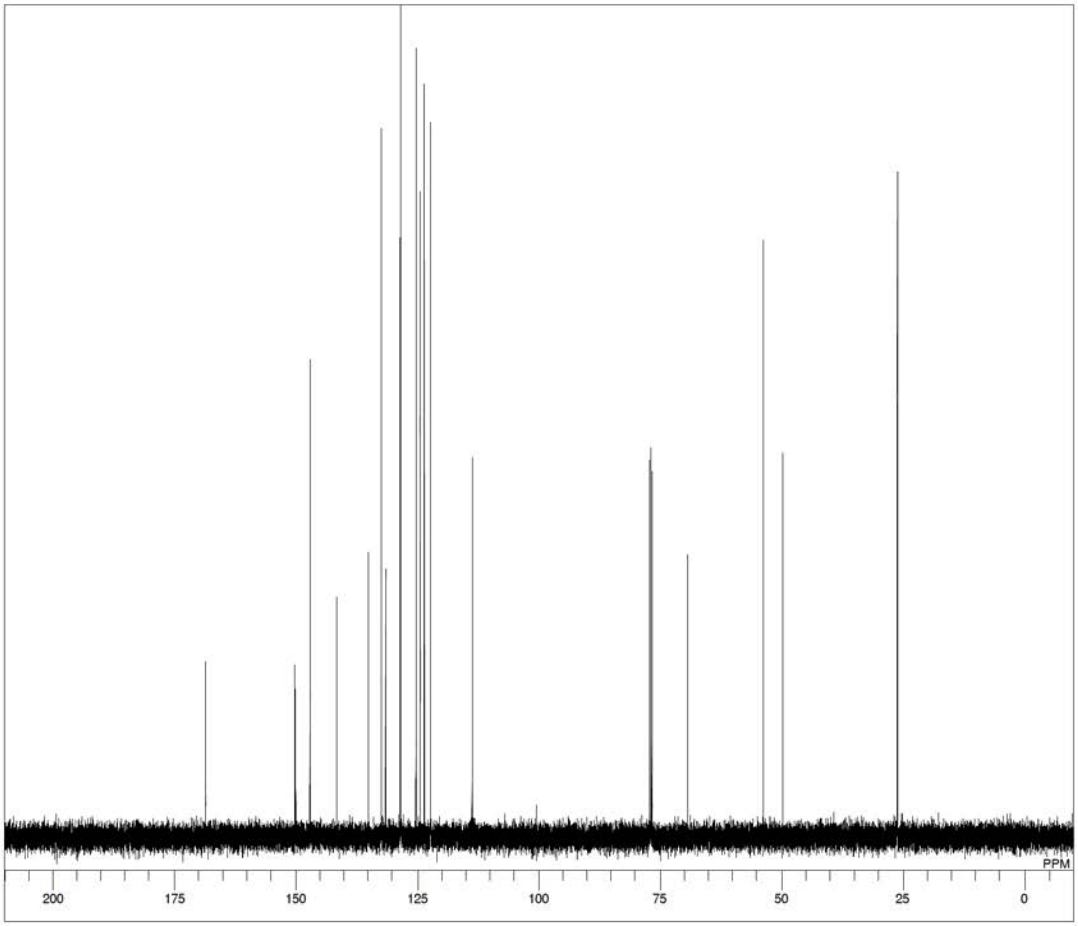
Pk #	Retention Time	Area	Area Percent
1	23.034	4568635	98.602
2	26.338	64767	1.398



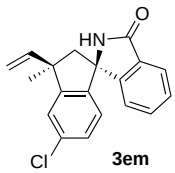
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	21
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-135.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	857
CSPED	10 Hz
FILDC	
FILDF	
SLVNT	CDCL3



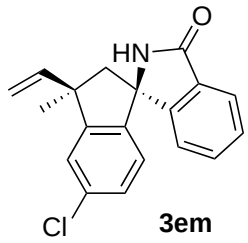
3em



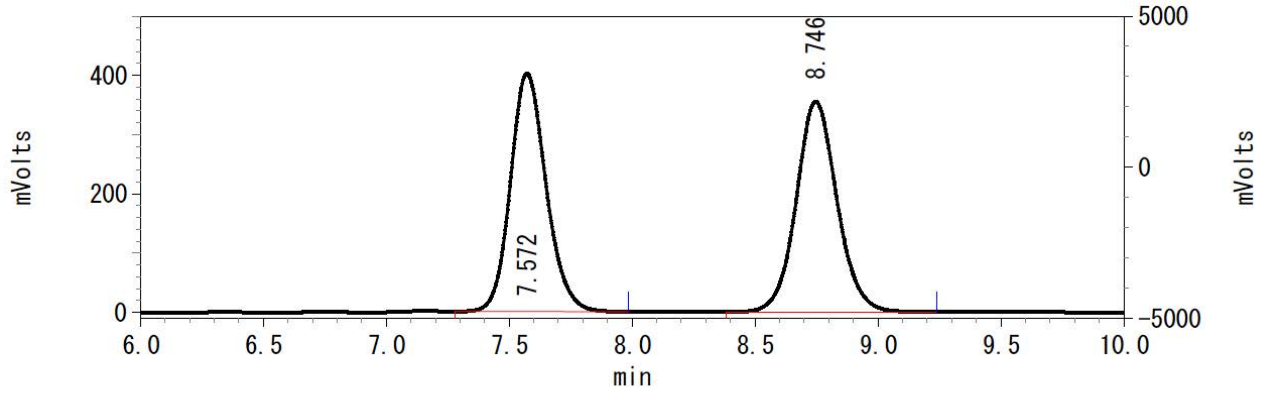
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-135-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	634
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



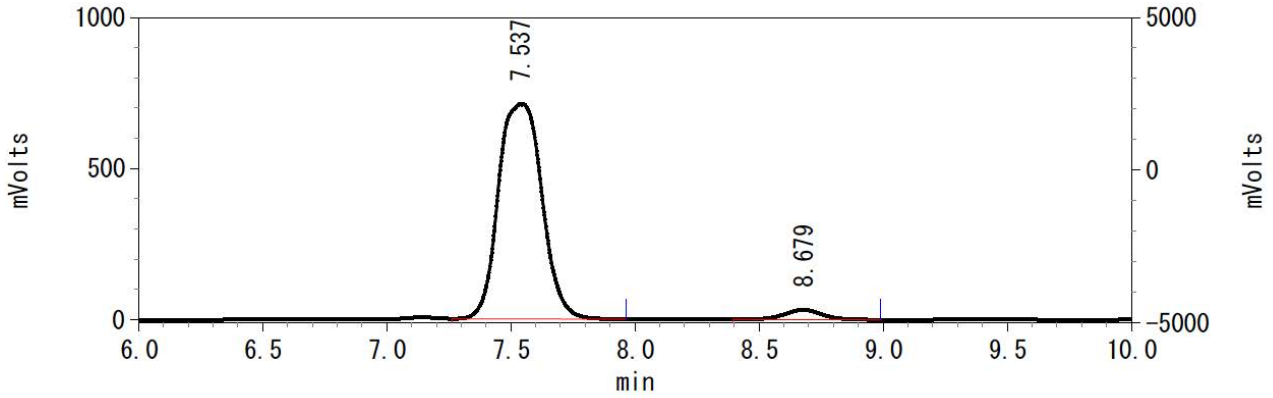
3em



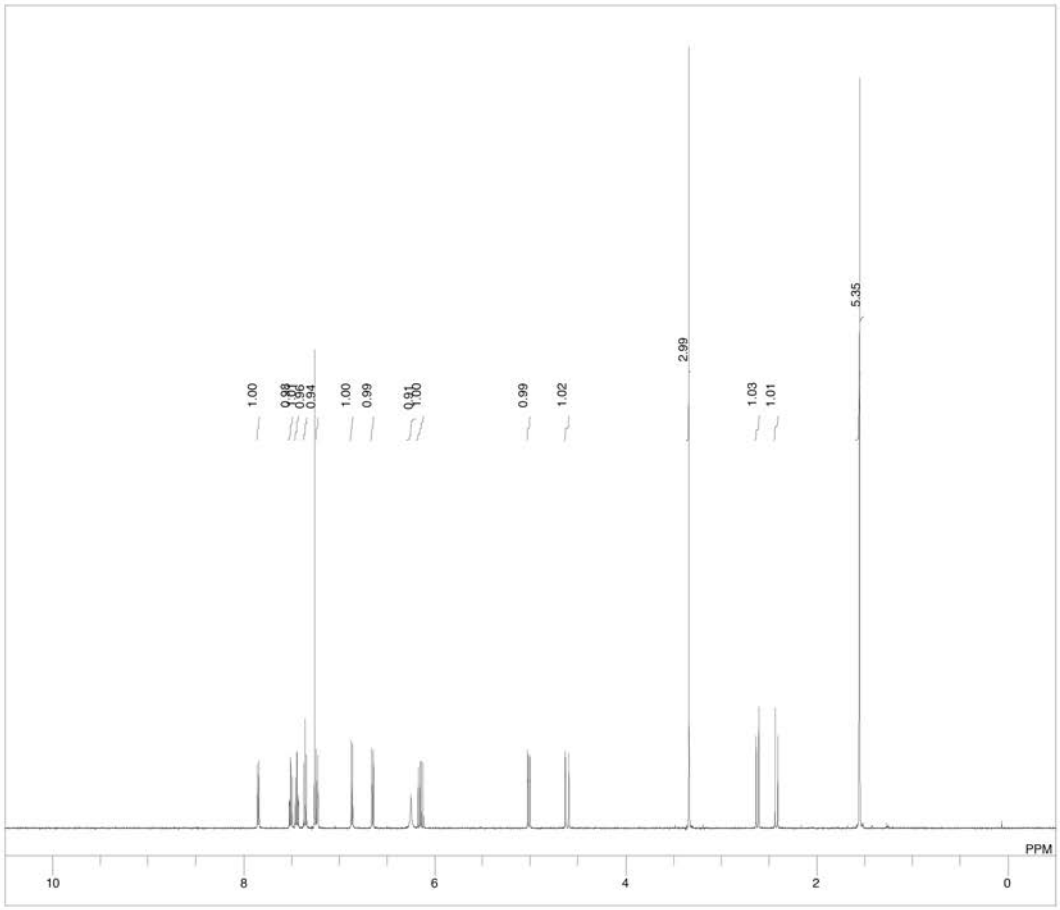
3em



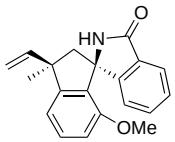
Pk #	Retention Time	Area	Area Percent
1	7.572	4061800	49.692
2	8.746	4112138	50.308



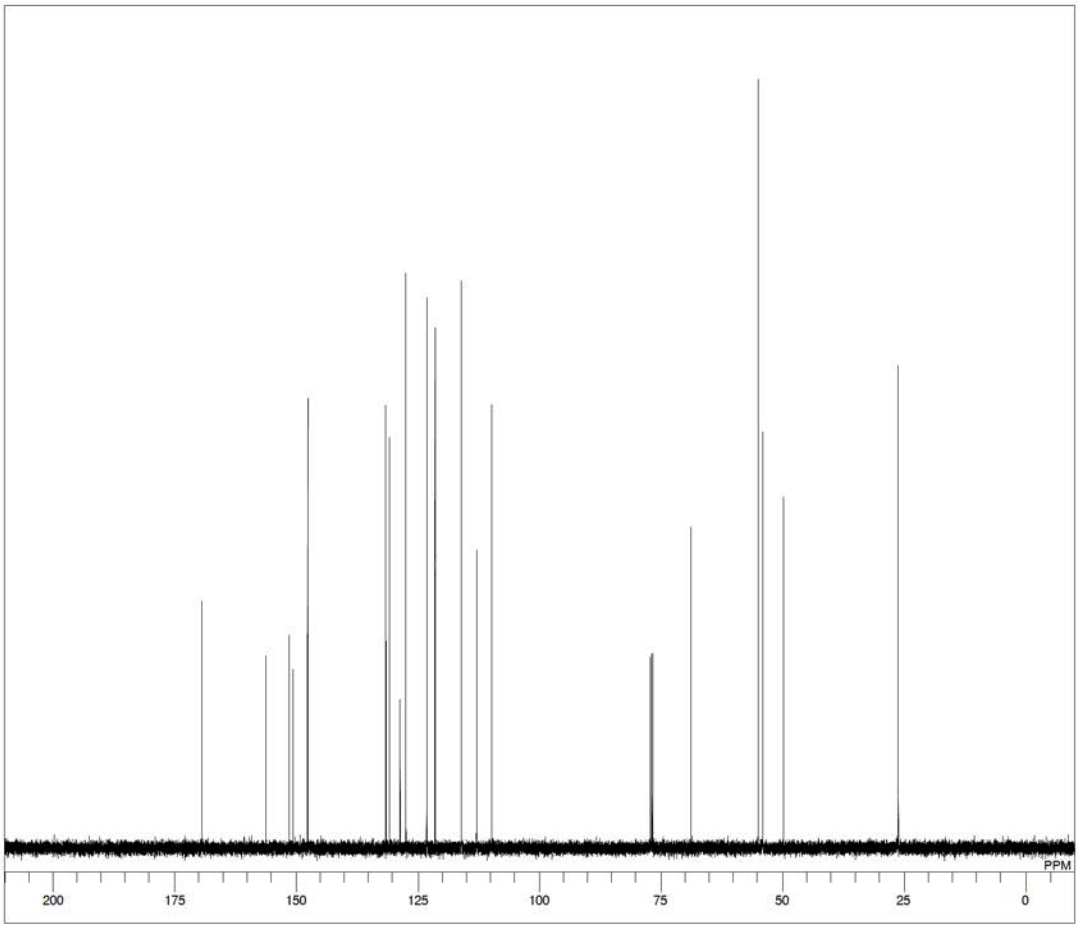
Pk #	Retention Time	Area	Area Percent
1	7.537	8674372	96.221
2	8.679	340701	3.779



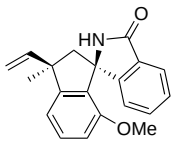
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	22
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-51.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	877
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



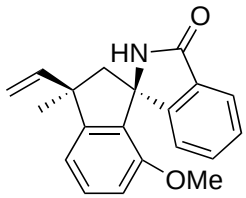
3fm



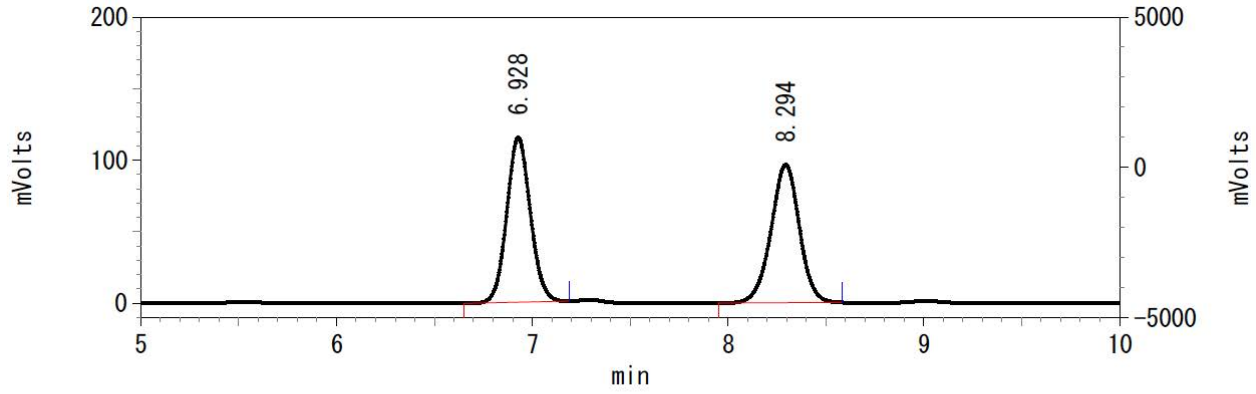
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-51-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	654
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



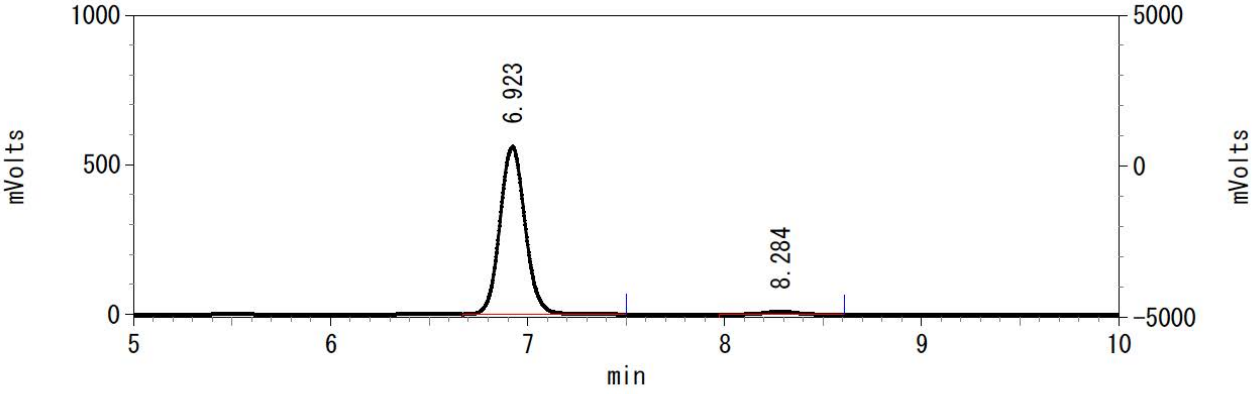
3fm



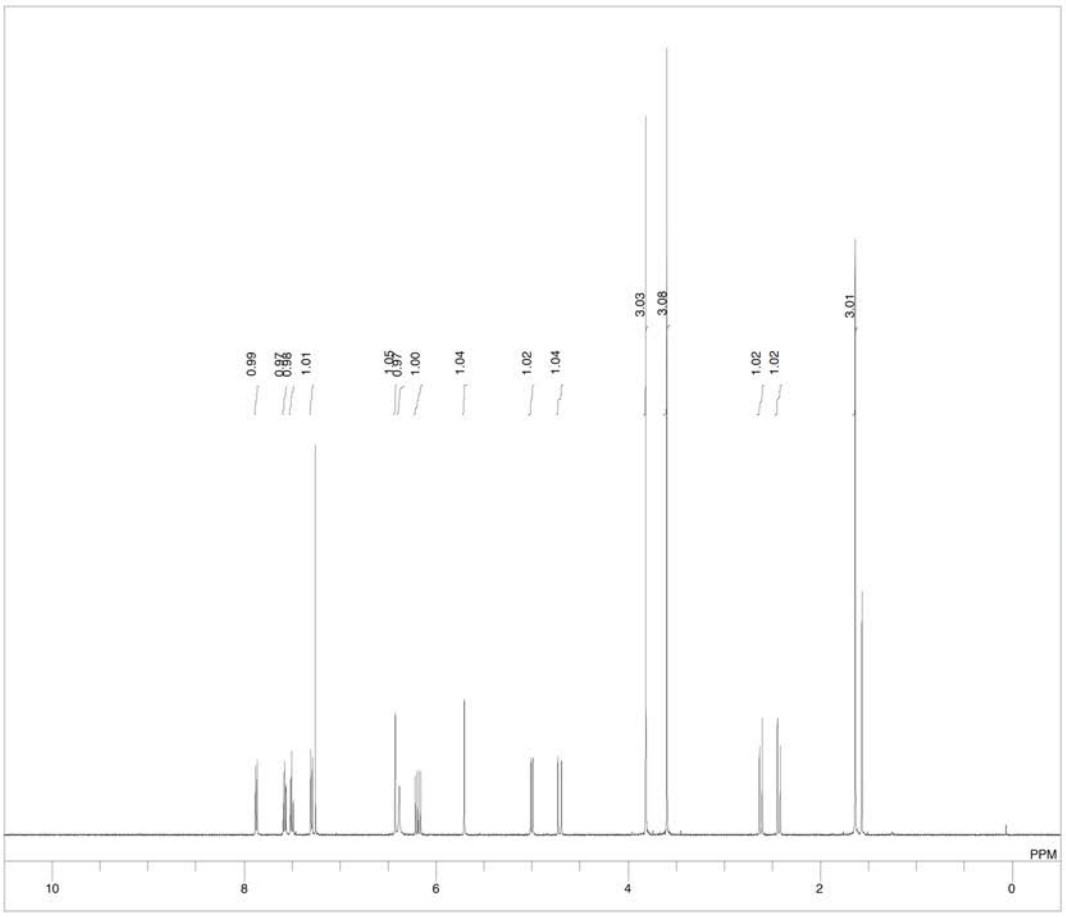
3fm



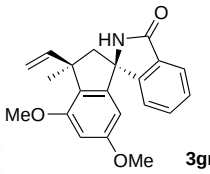
Pk #	Retention Time	Area	Area Percent
1	6.928	965086	49.682
2	8.294	977437	50.318



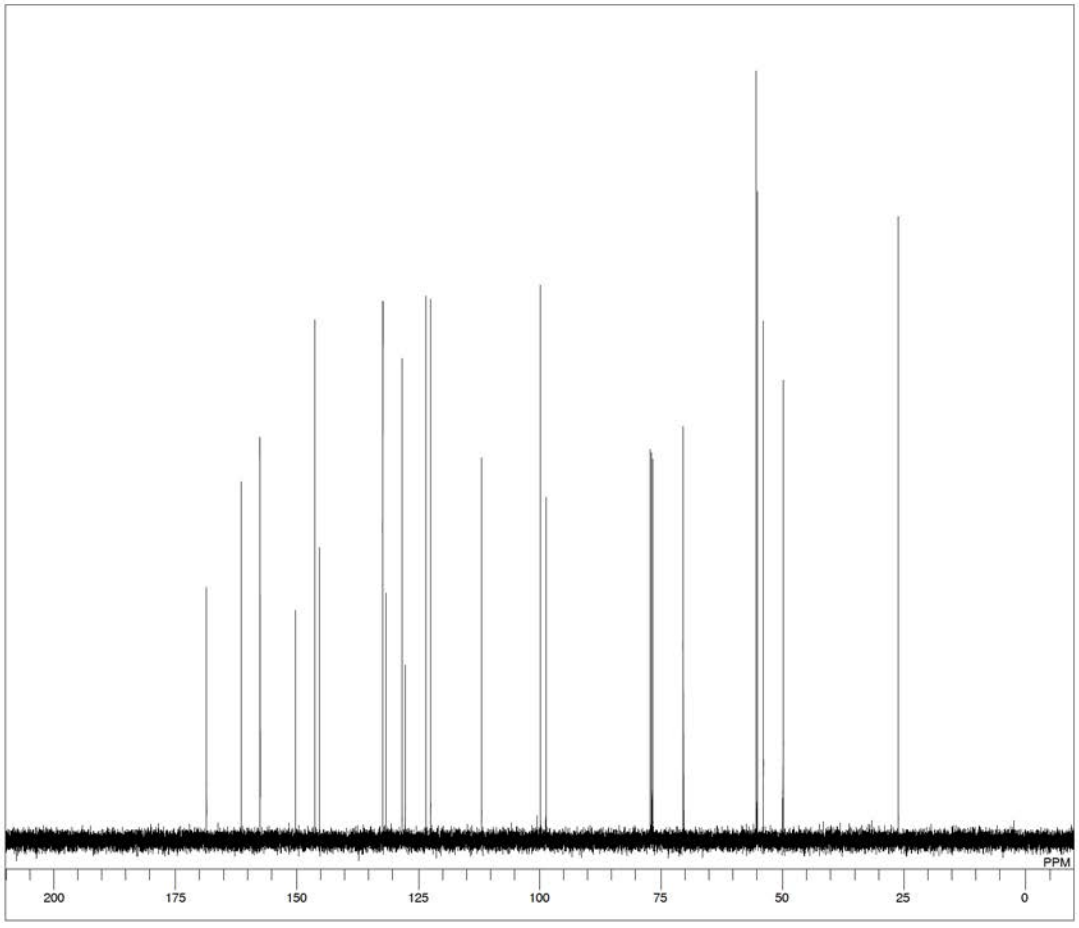
Pk #	Retention Time	Area	Area Percent
1	6.923	4979716	97.985
2	8.284	102393	2.015



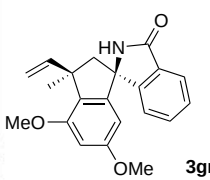
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-147.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1063
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



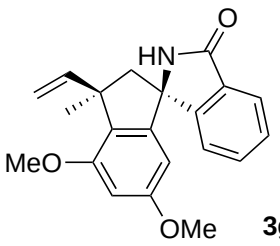
3gm



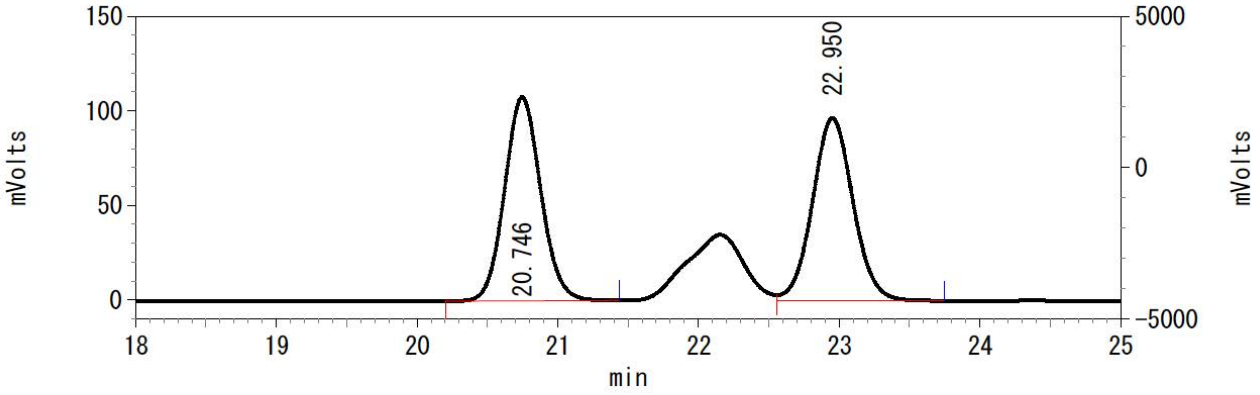
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-147-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	647
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



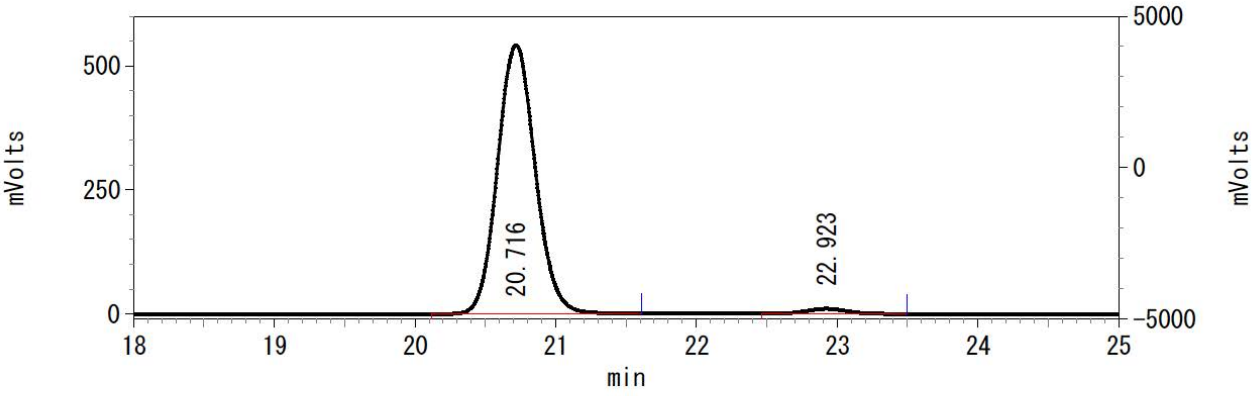
3gm



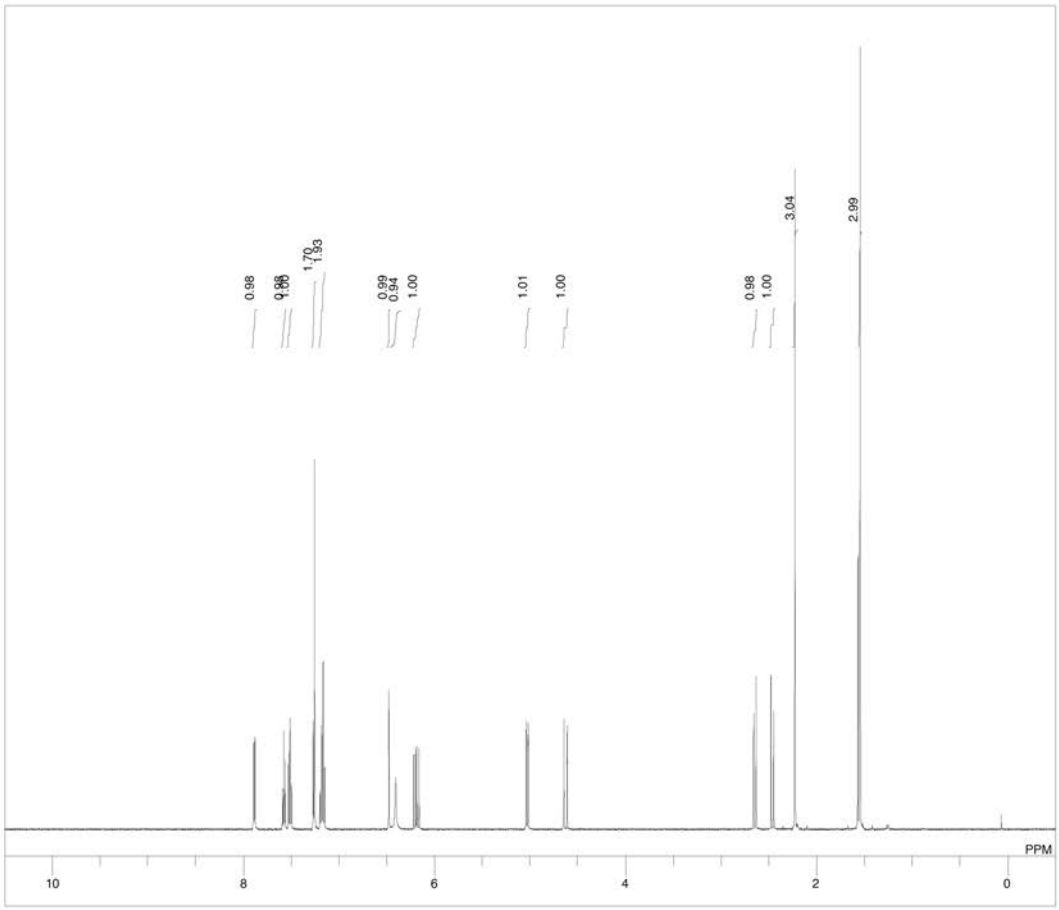
3gm



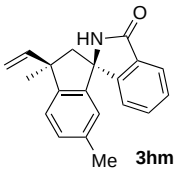
Pk #	Retention Time	Area	Area Percent
1	20.746	1928632	49.848
2	22.950	1940417	50.152



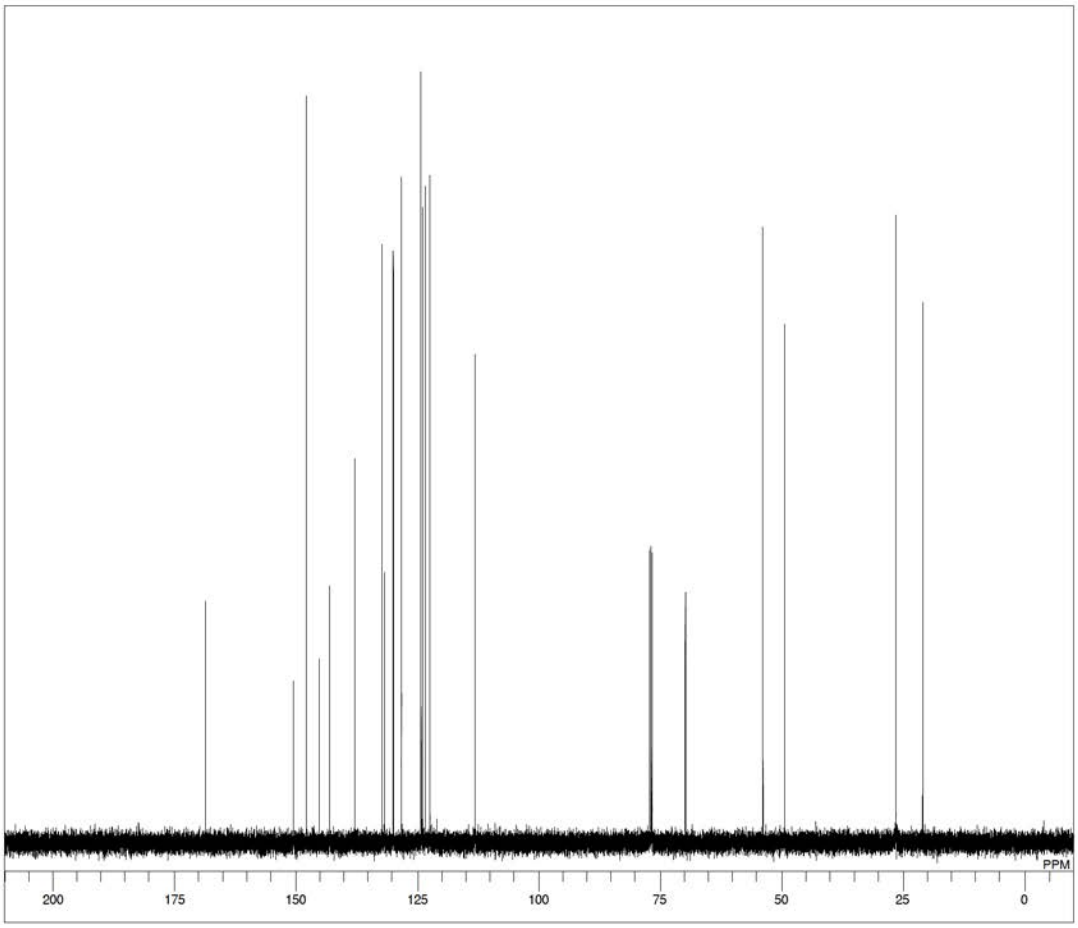
Pk #	Retention Time	Area	Area Percent
1	20.716	10085161	98.081
2	22.923	197364	1.919



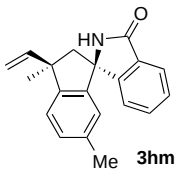
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-49.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1018
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



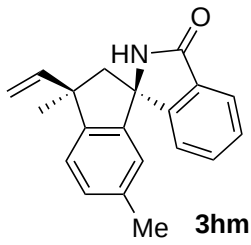
3hm



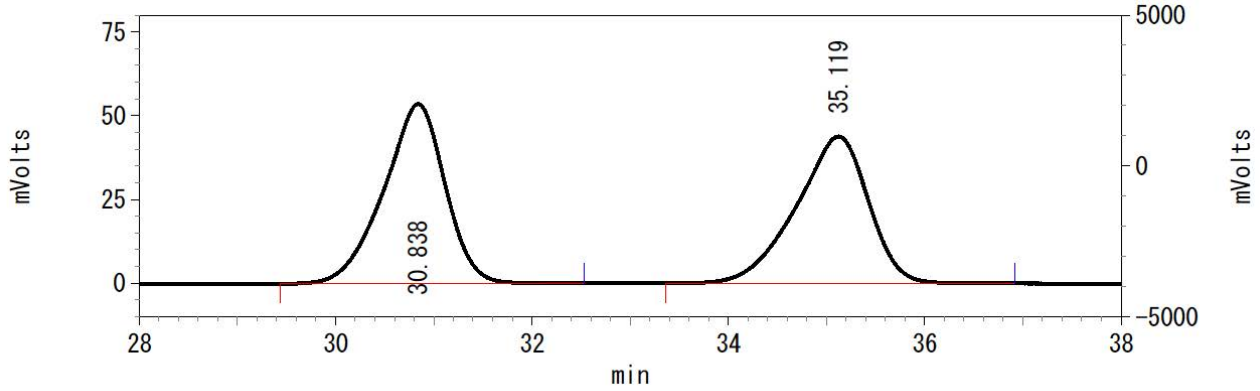
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-49-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	732
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



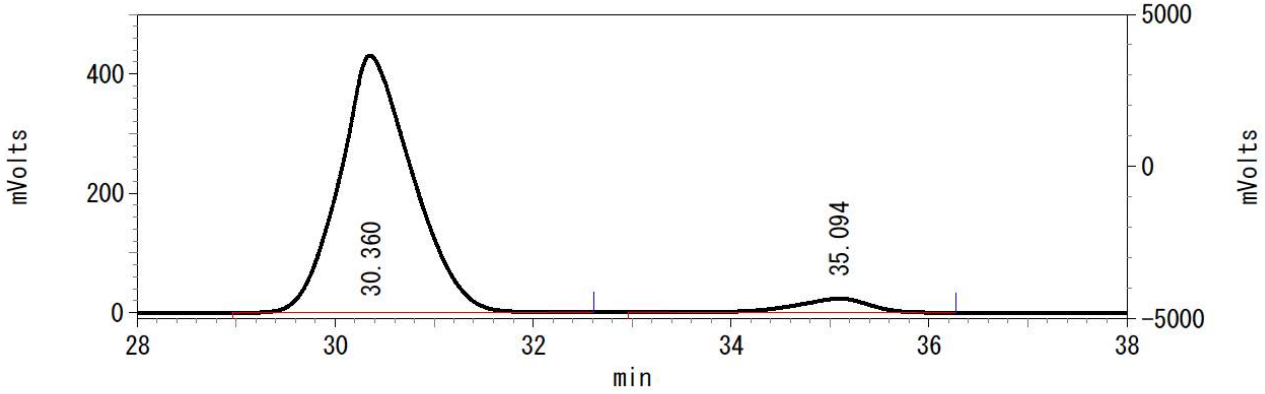
3hm



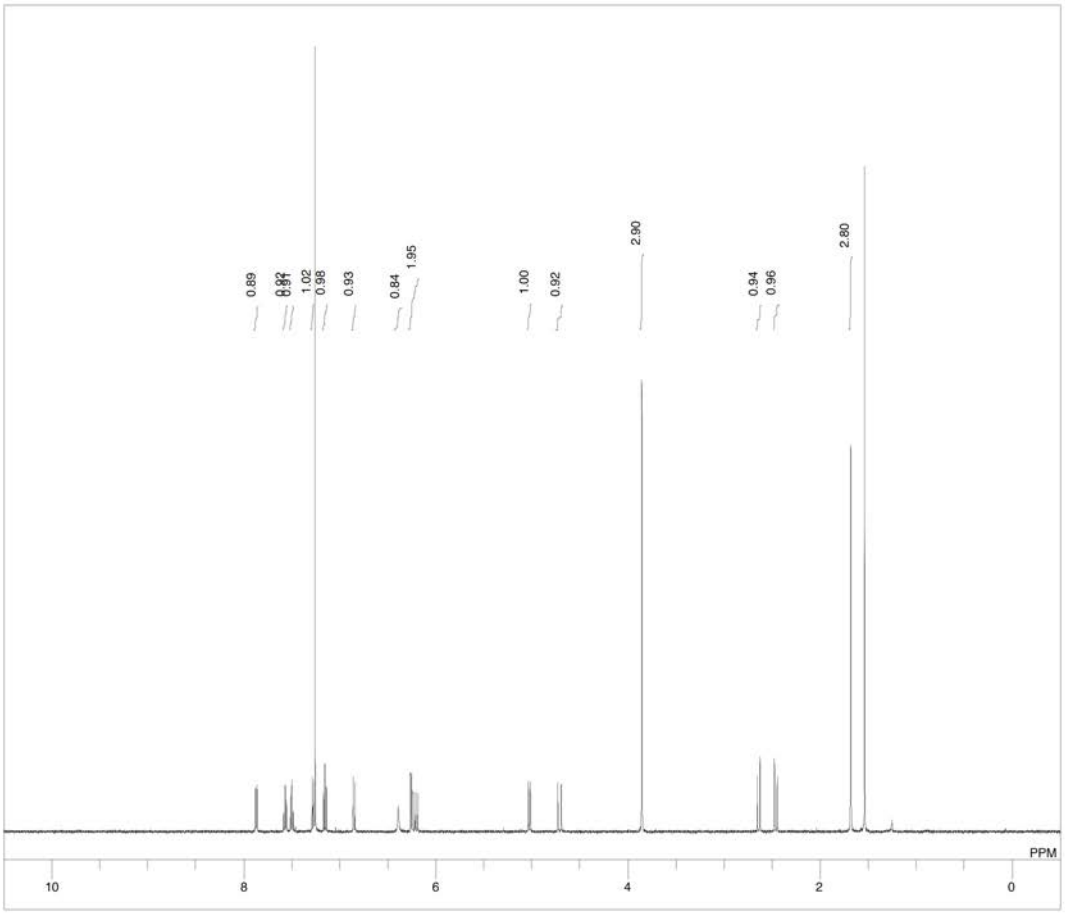
3hm



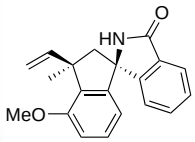
Pk #	Retention Time	Area	Area Percent
1	30.838	2334898	50.932
2	35.119	2249413	49.068



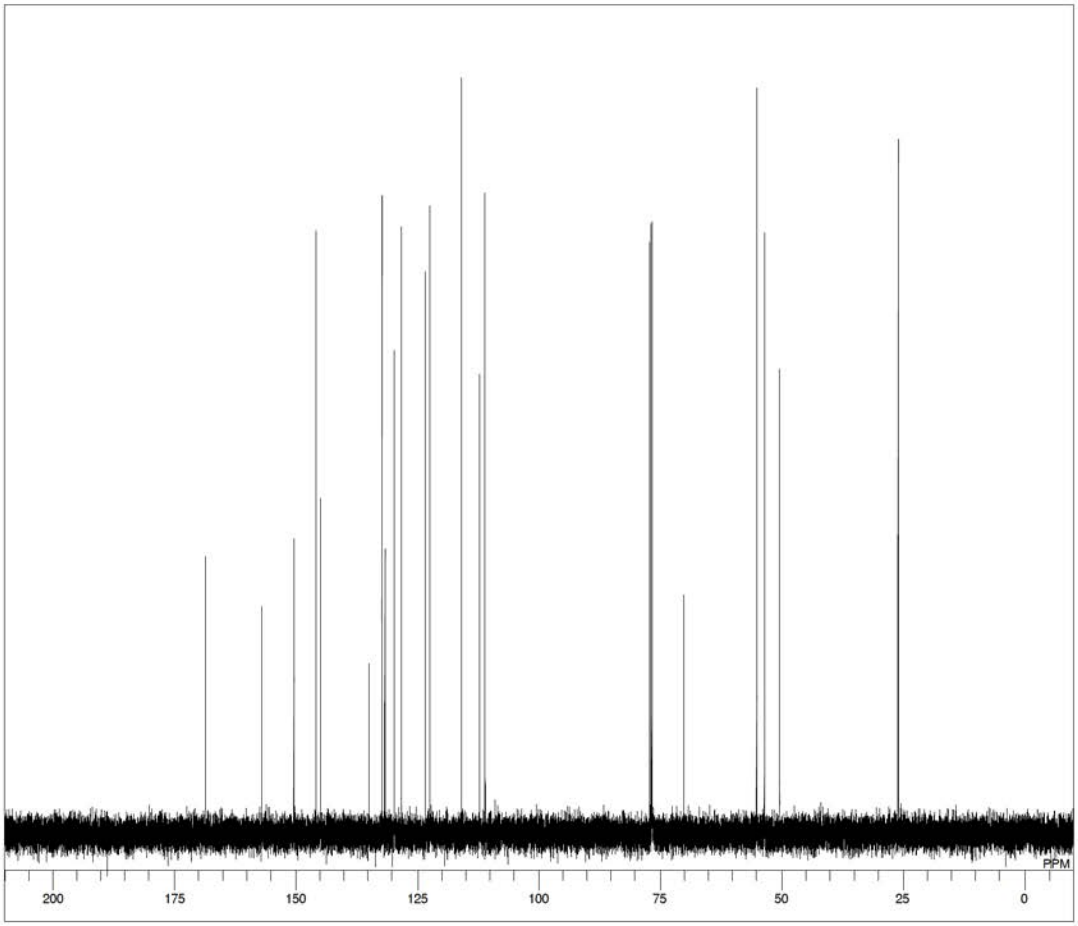
Pk #	Retention Time	Area	Area Percent
1	30.360	22119673	94.813
2	35.094	1210072	5.187



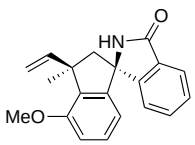
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-90-2-1.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1013
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



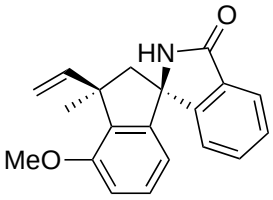
3im



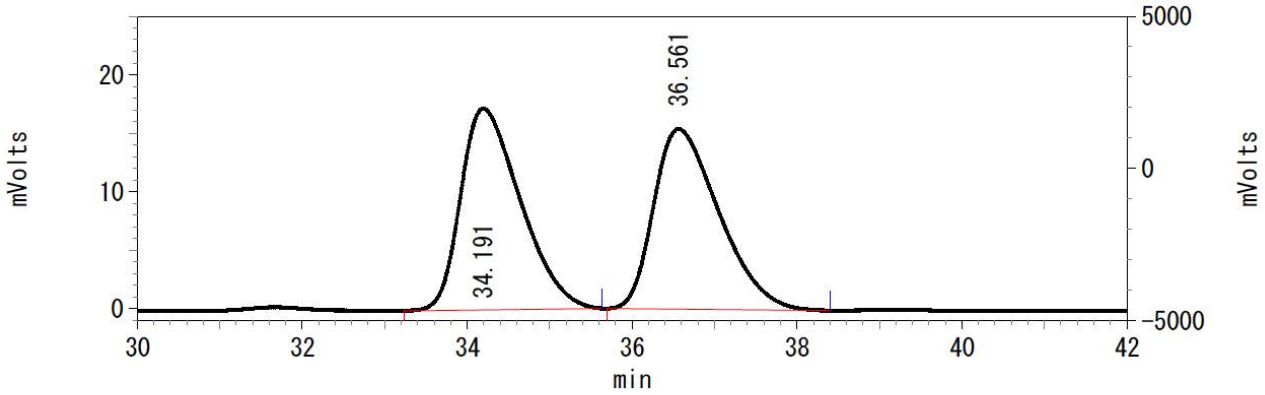
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-90-2-1-carbon'.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	658
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



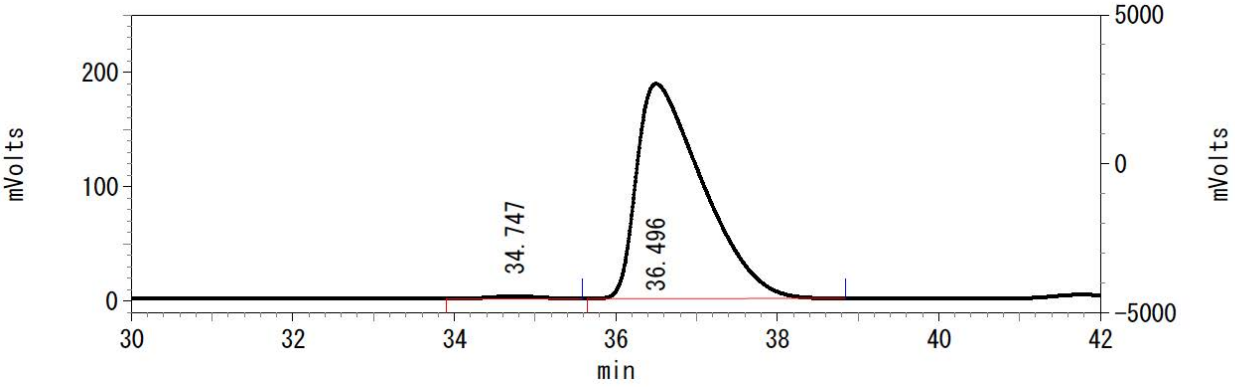
3im



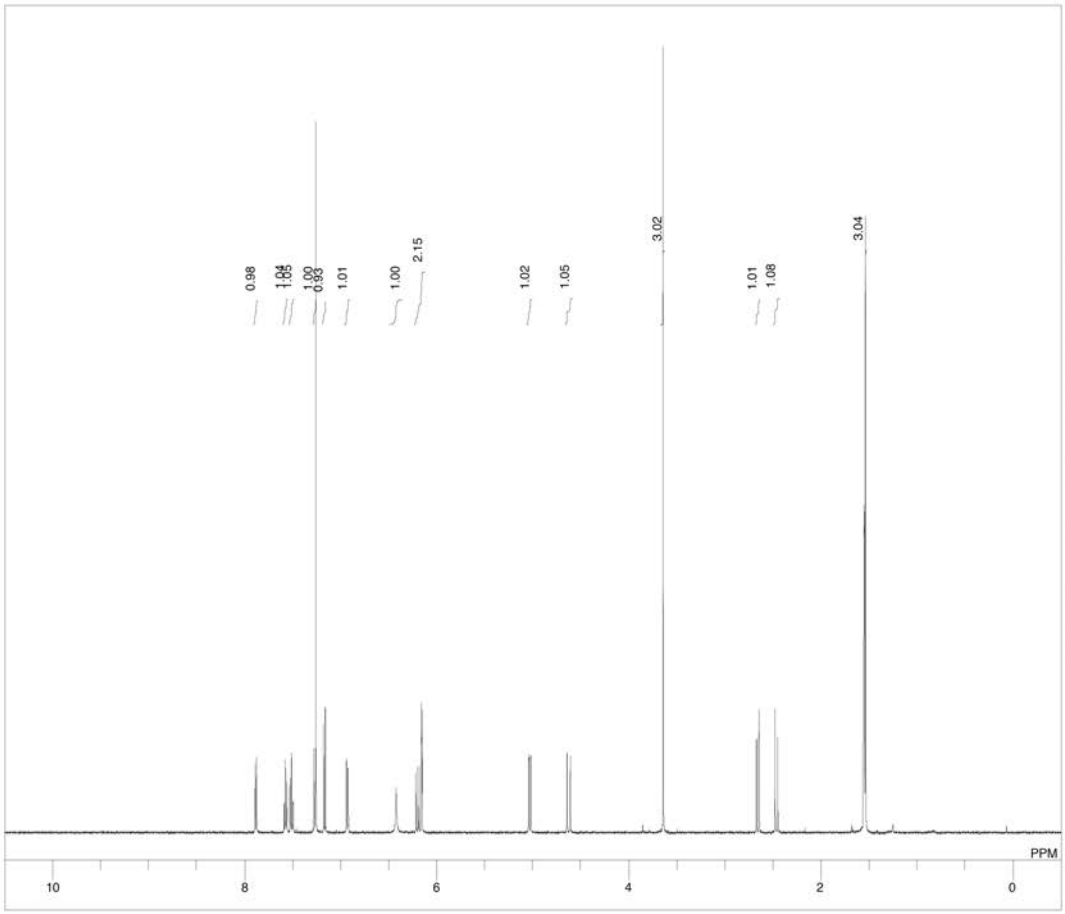
3im



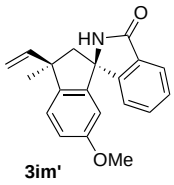
Pk #	Retention Time	Area	Area Percent
1	34.191	864804	50.486
2	36.561	848164	49.514



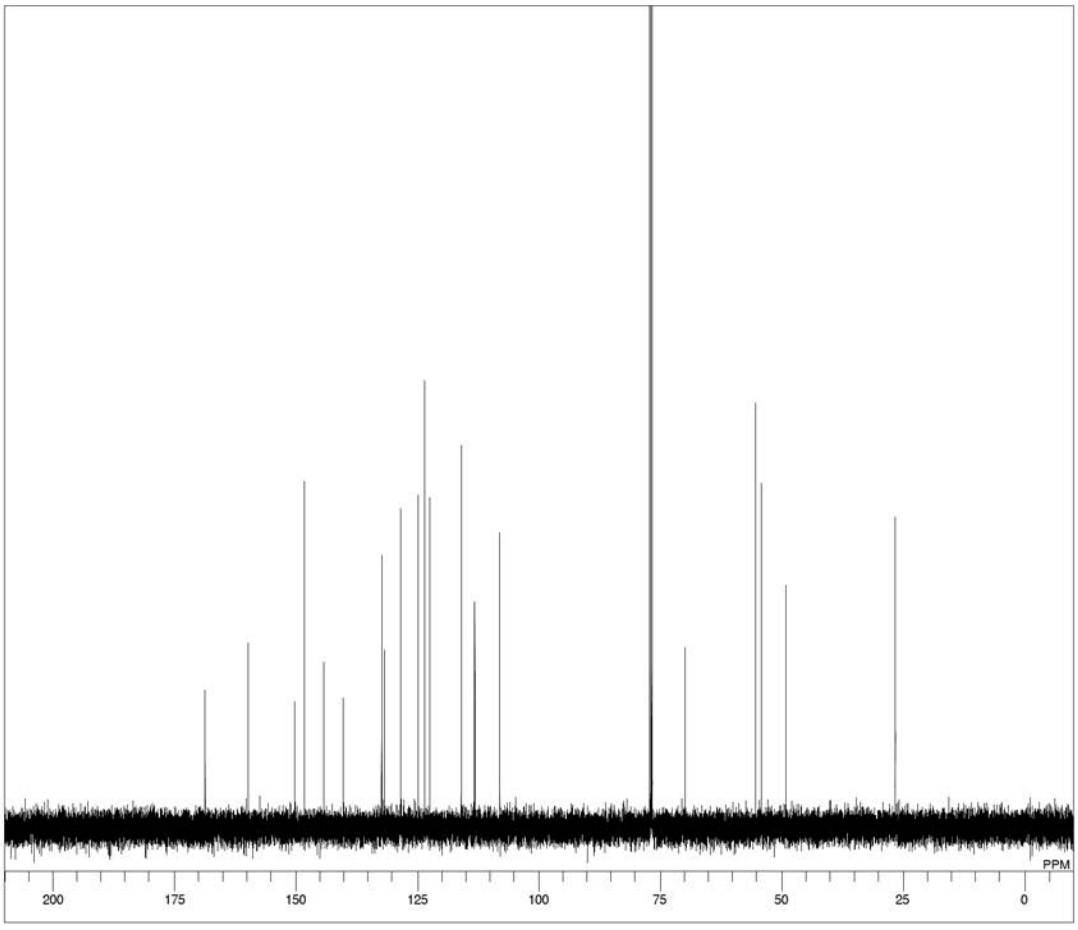
Pk #	Retention Time	Area	Area Percent
1	34.747	89250	0.831
2	36.496	10656098	99.169



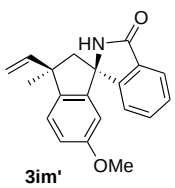
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	22
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-90-2-2.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1083
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



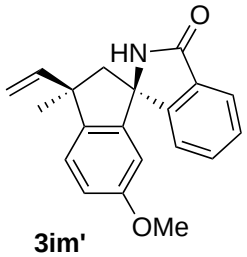
3im'



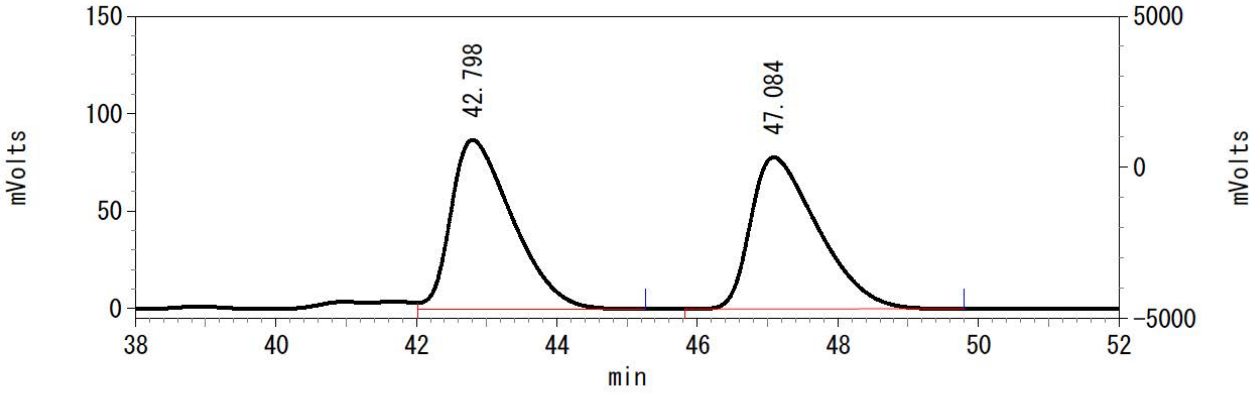
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-90-2-2-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	741
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



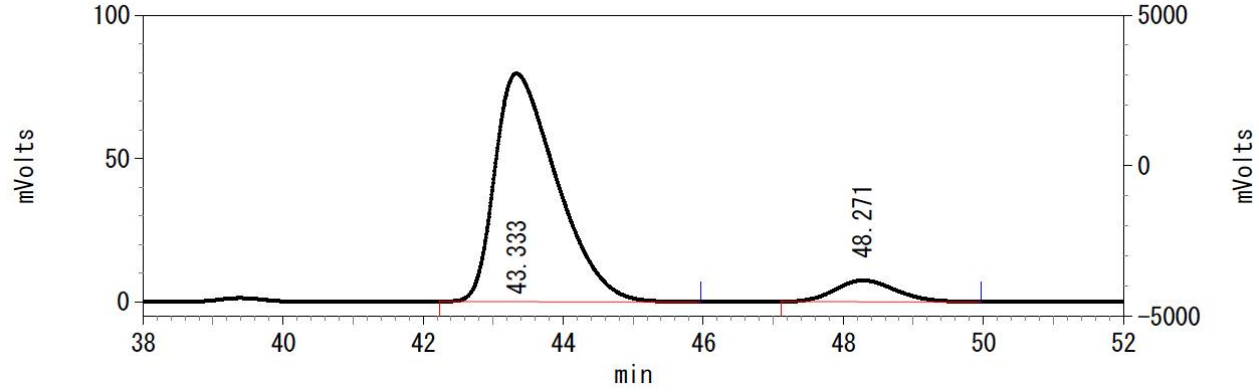
3im'



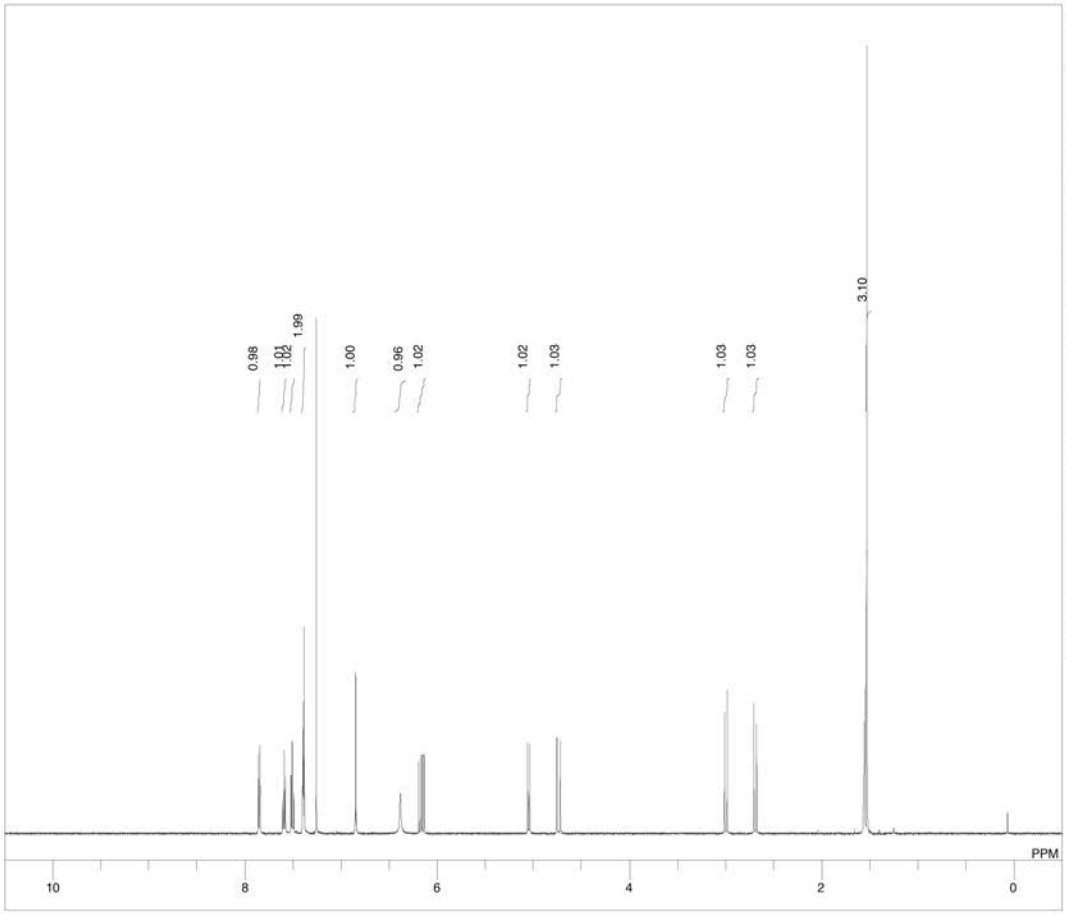
3im'



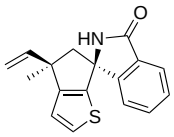
Pk #	Retention Time	Area	Area Percent
1	42.798	5267010	50.428
2	47.084	5177705	49.572



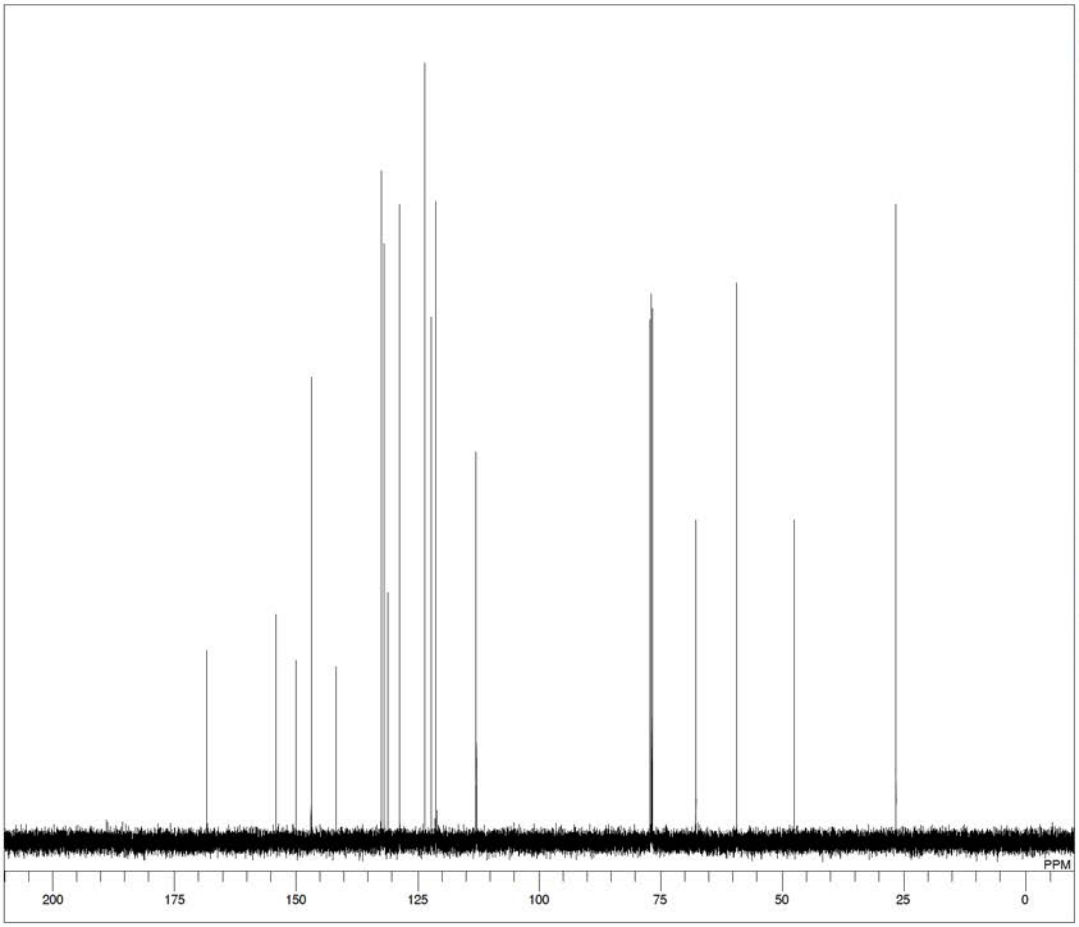
Pk #	Retention Time	Area	Area Percent
1	43.333	4866413	91.494
2	48.271	452421	8.506



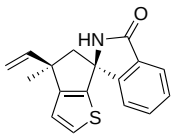
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	22
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-142.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	844
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



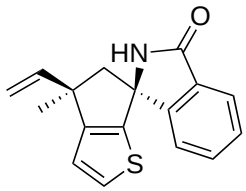
3jm



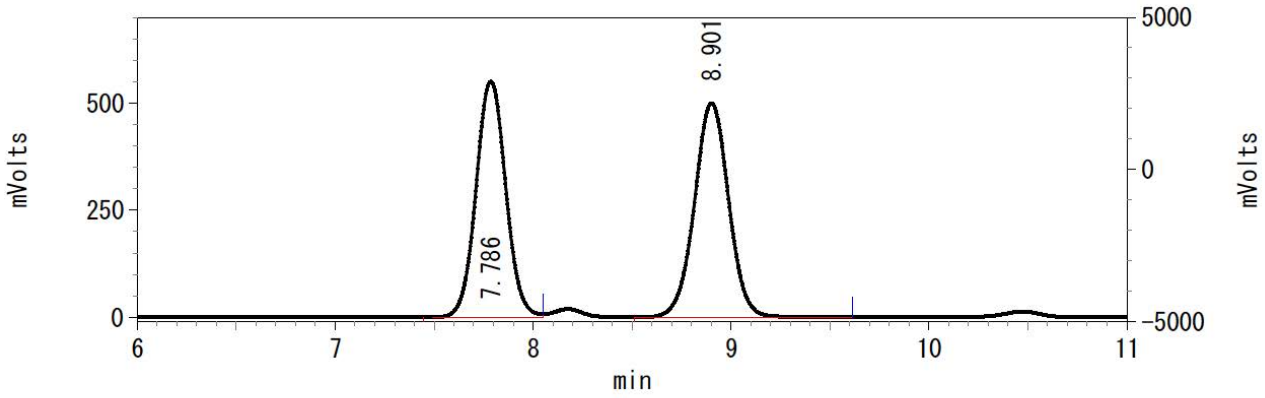
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	25
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-142-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	658
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



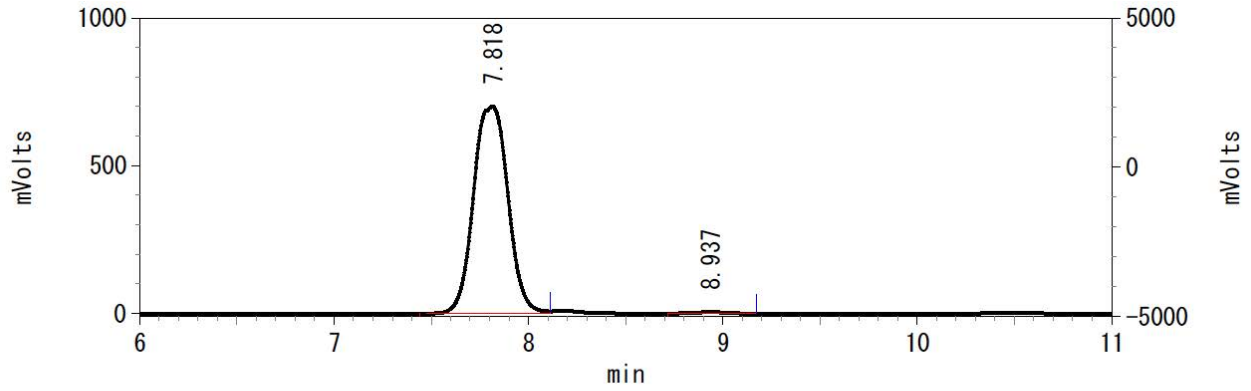
3jm



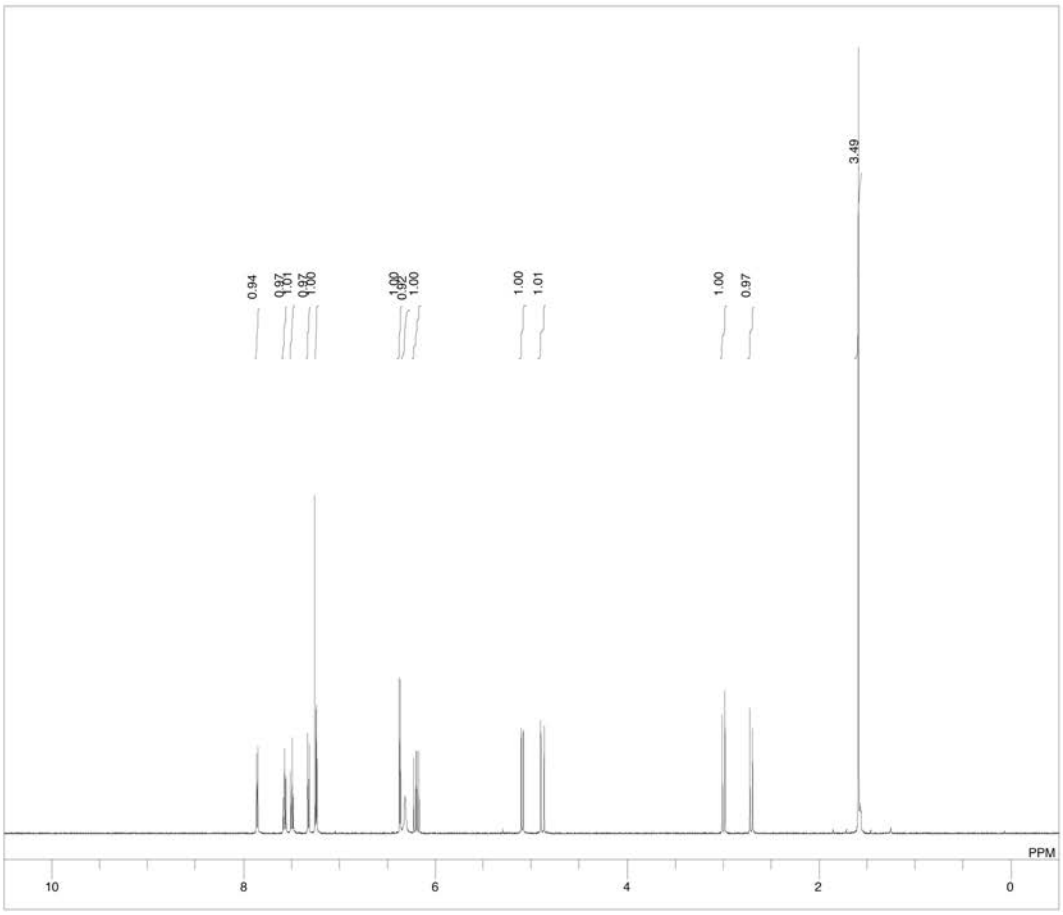
3jm



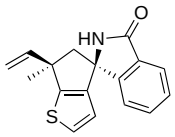
Pk #	Retention Time	Area	Area Percent
1	7.786	5774070	49.053
2	8.901	5997060	50.947



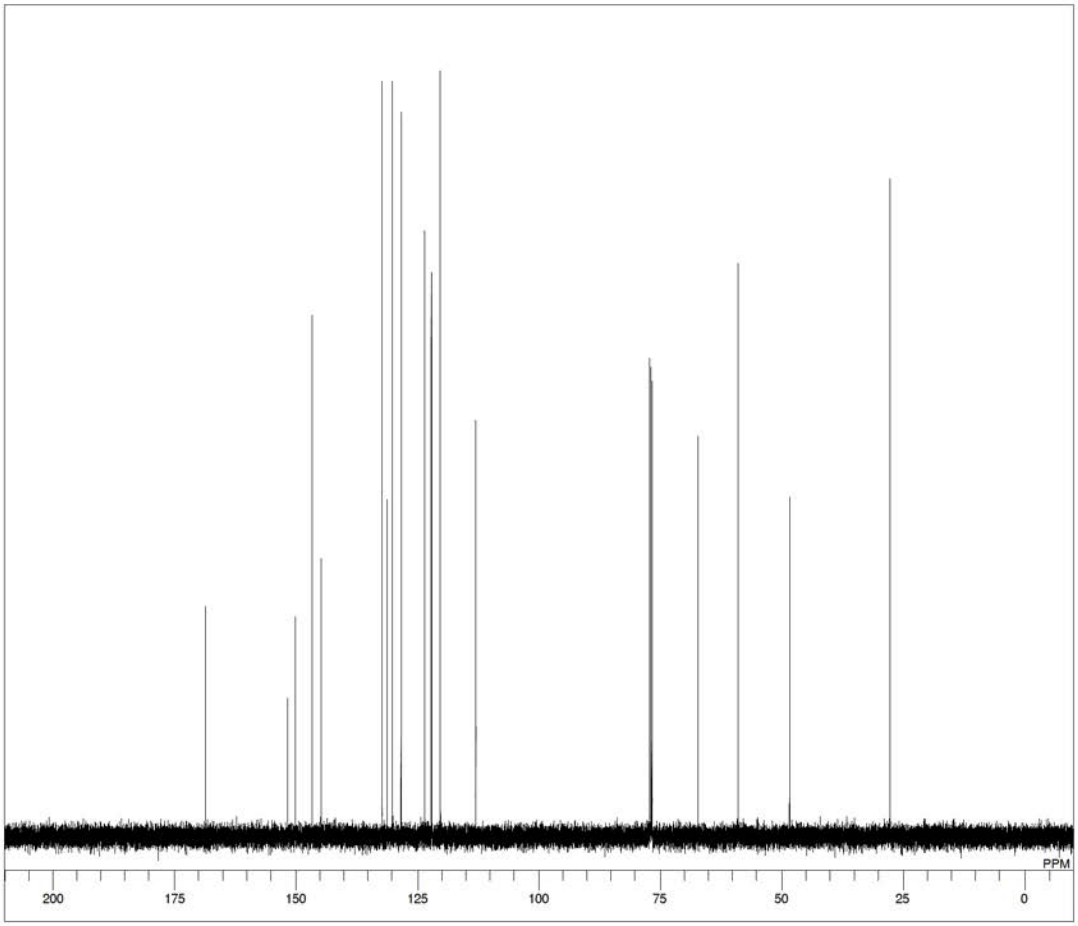
Pk #	Retention Time	Area	Area Percent
1	7.818	8659249	99.460
2	8.937	46980	0.540



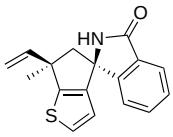
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE mn-2-164.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 1029
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3



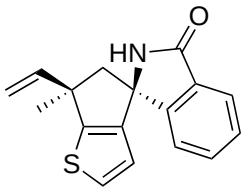
3km



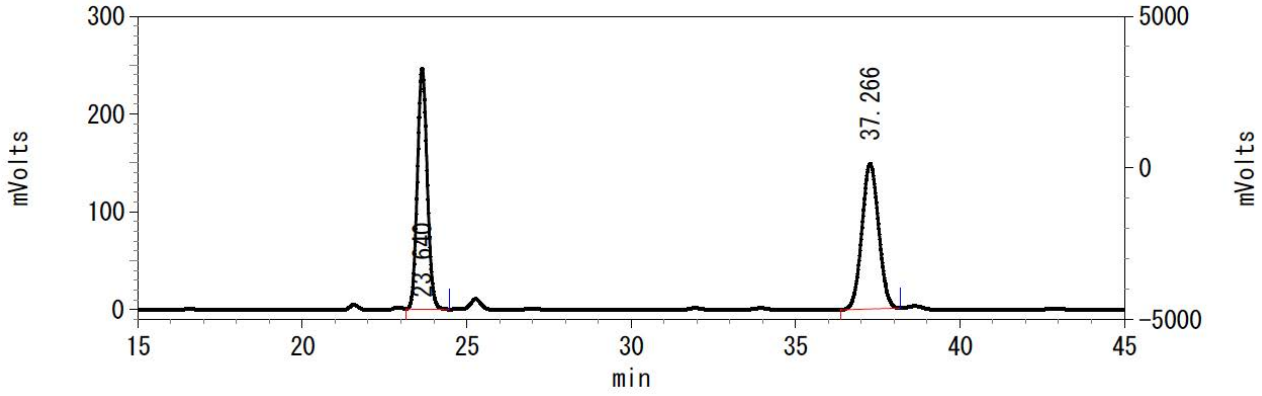
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 720
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE mn-2-164-carbon.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 343
LKSIG 648
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3



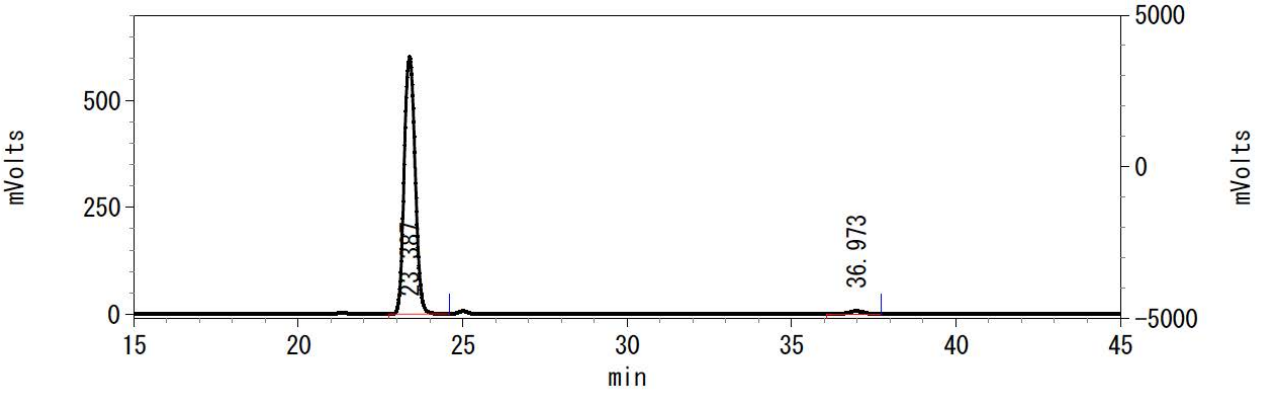
3km



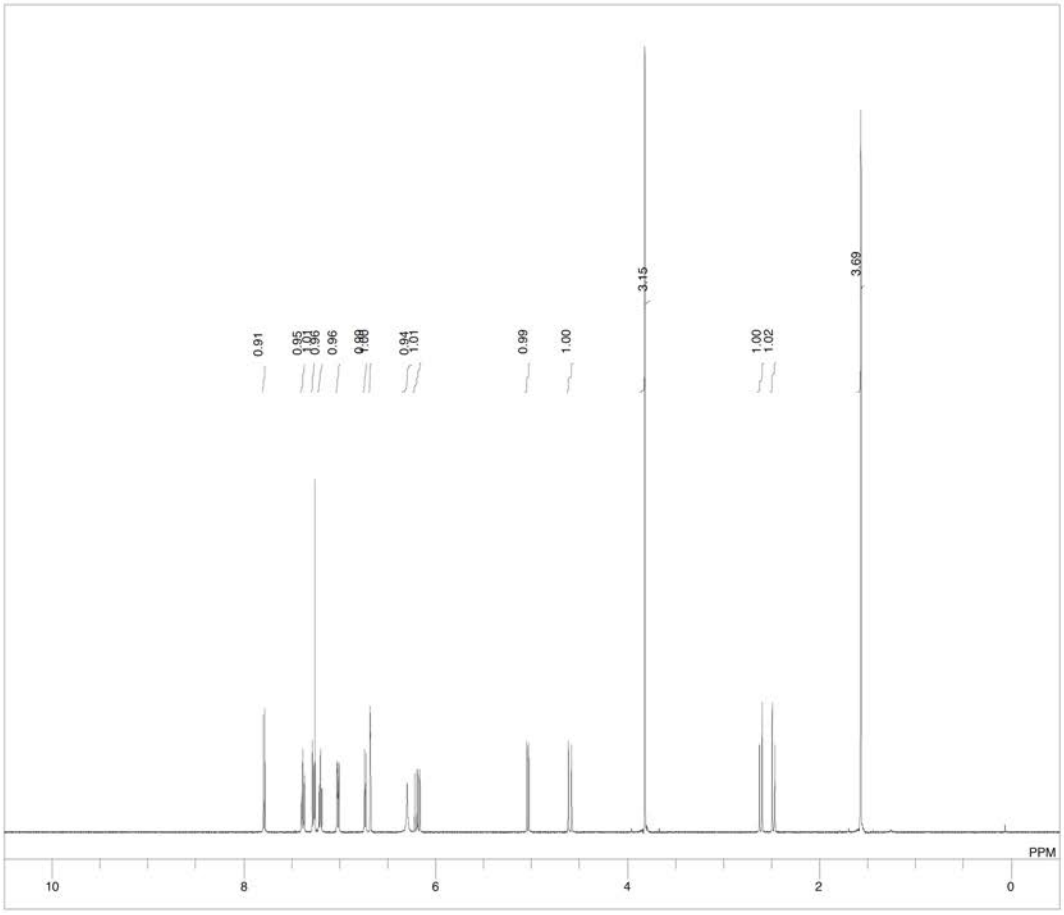
3km



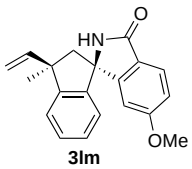
Pk #	Retention Time	Area	Area Percent
1	23.640	5147679	49.426
2	37.266	5267309	50.574



Pk #	Retention Time	Area	Area Percent
1	23.387	13956948	98.261
2	36.973	246972	1.739



MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-58.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1006
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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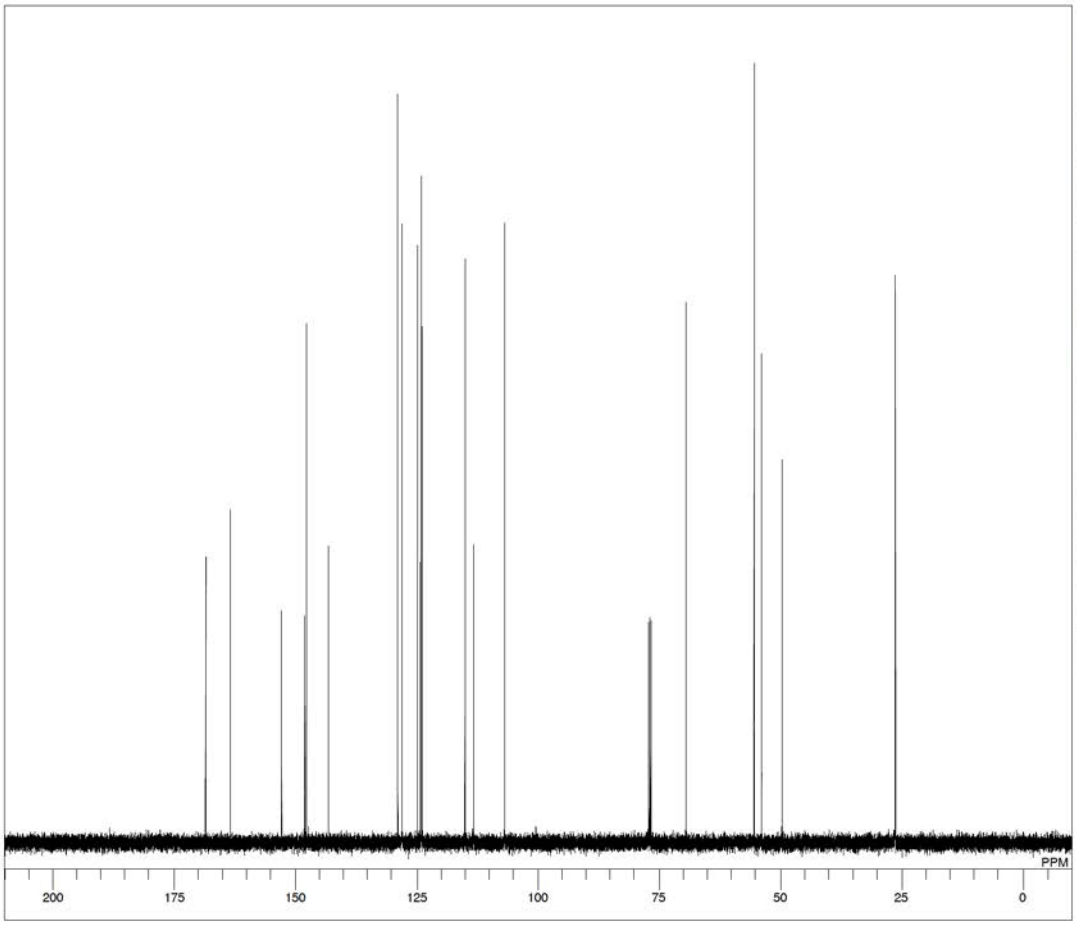
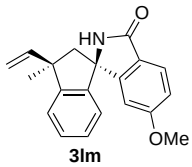


MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-58-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	649
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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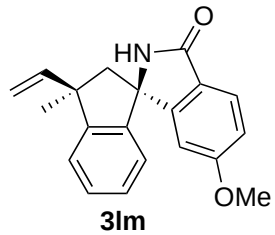
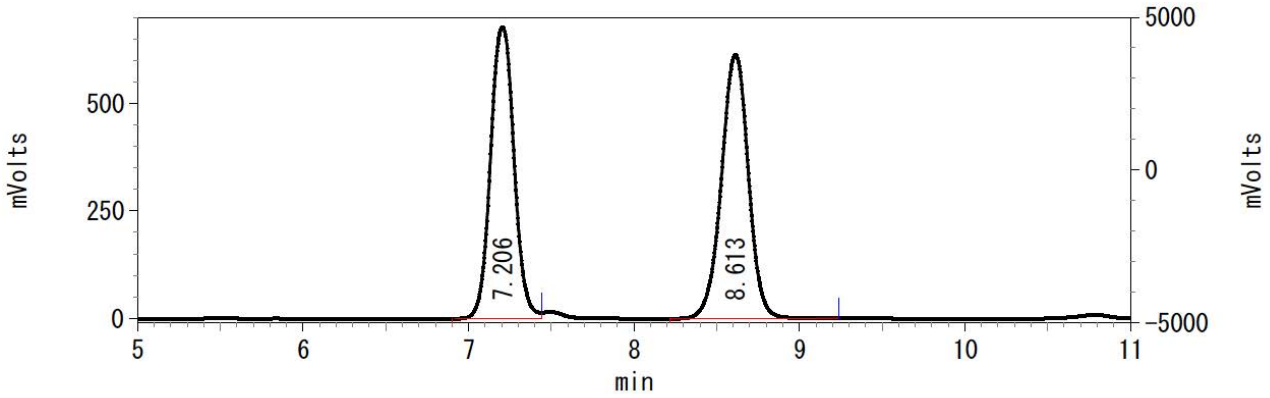
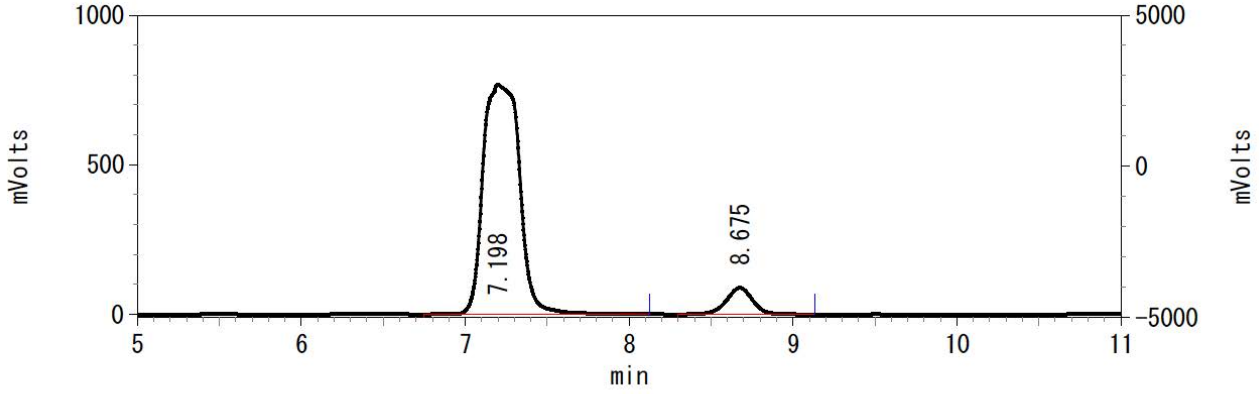


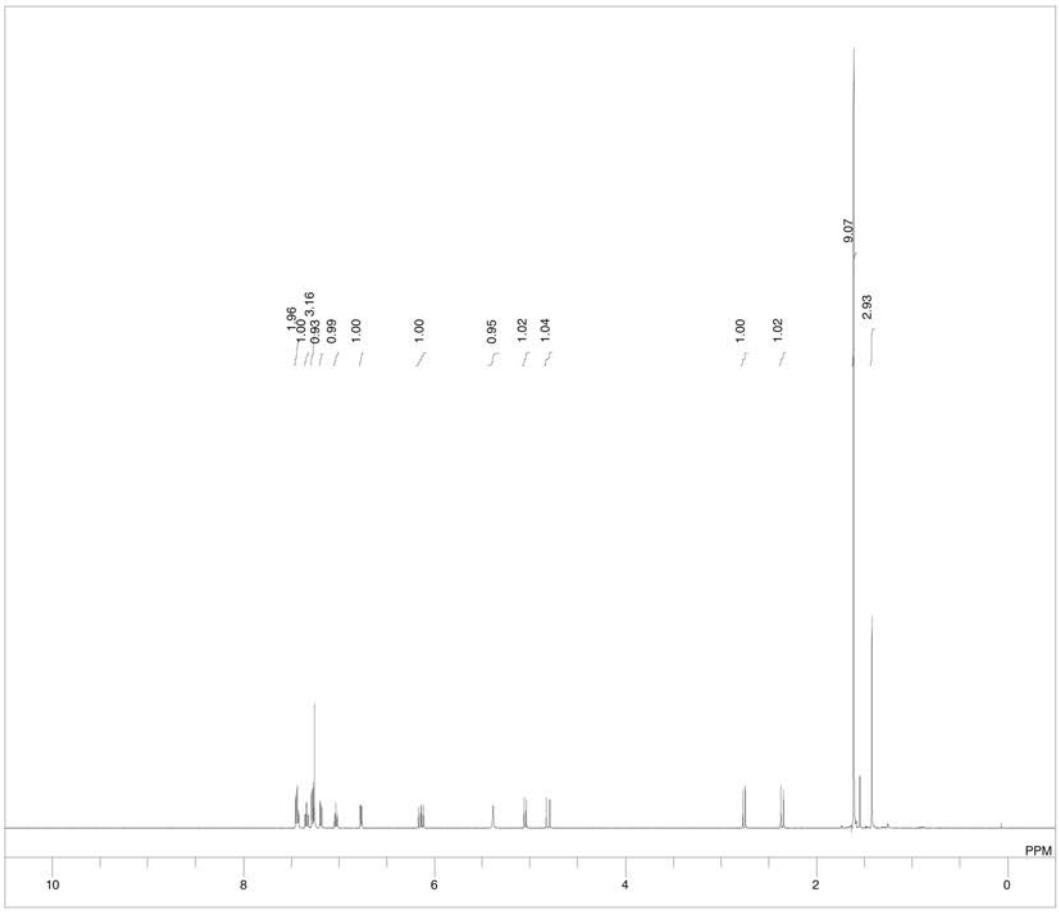
3lm



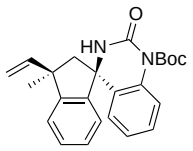
Pk #	Retention Time	Area	Area Percent
1	7.206	6709148	48.989
2	8.613	6986060	51.011



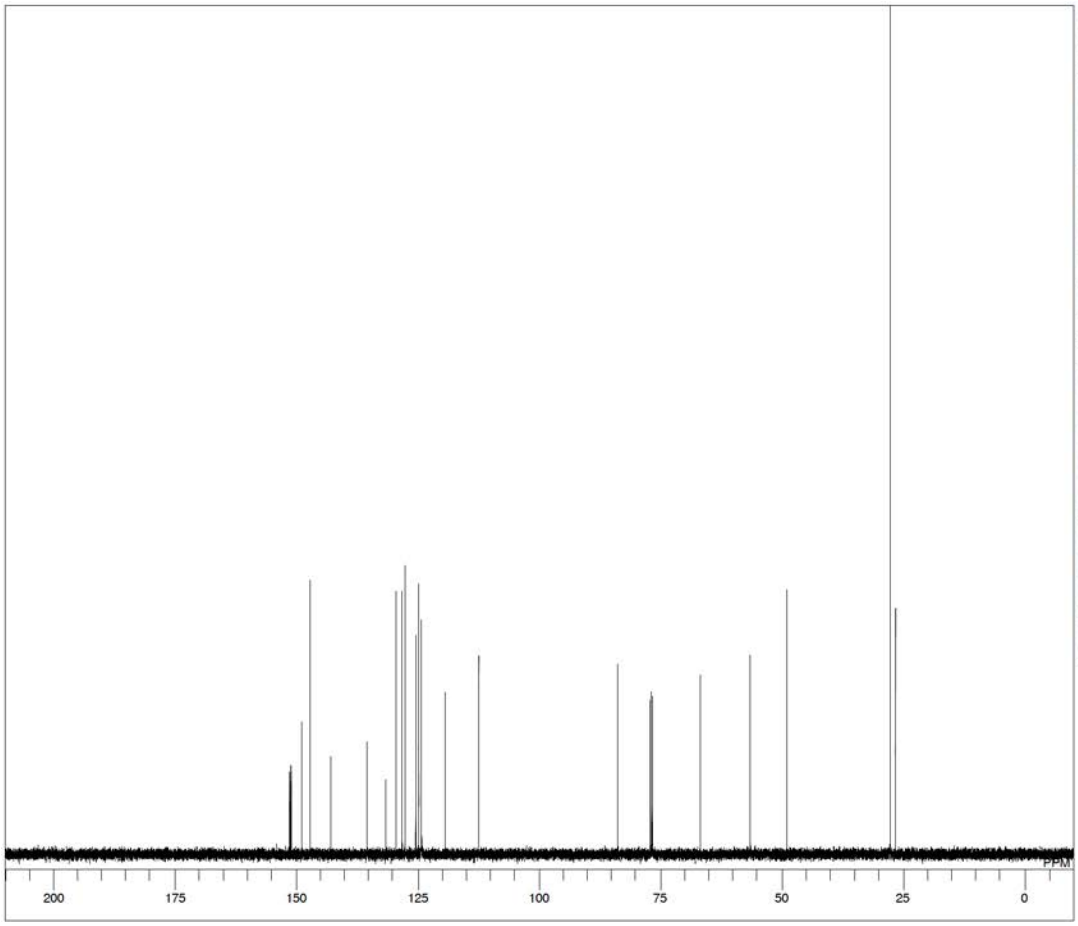
Pk #	Retention Time	Area	Area Percent
1	7.198	11840748	92.359
2	8.675	979565	7.641



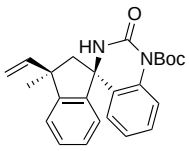
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	19
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-175.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1013
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



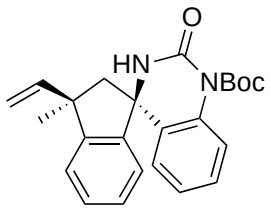
7



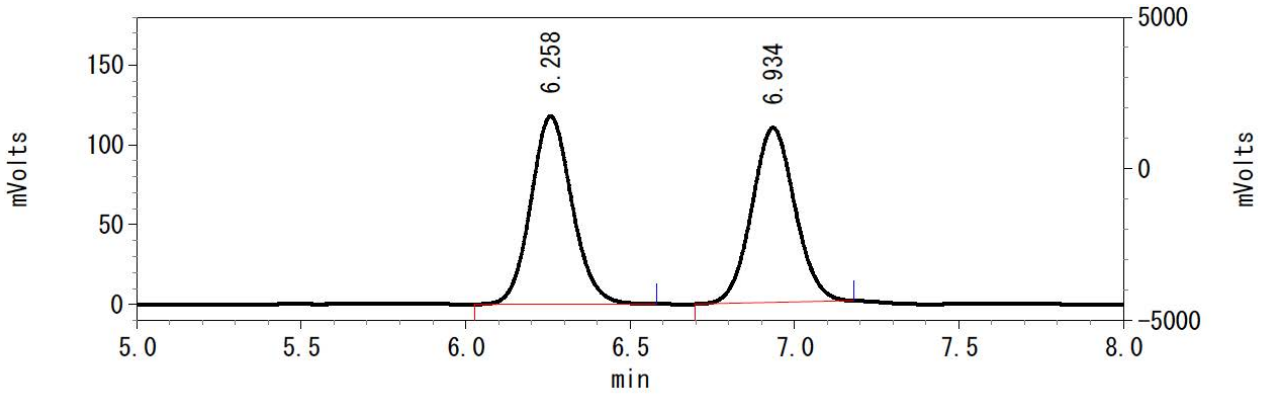
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	23
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-175-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	609
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



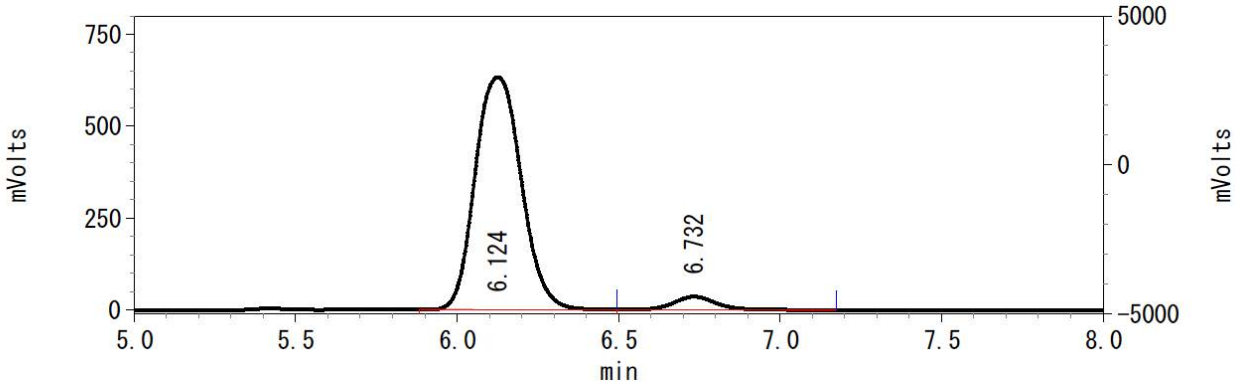
7



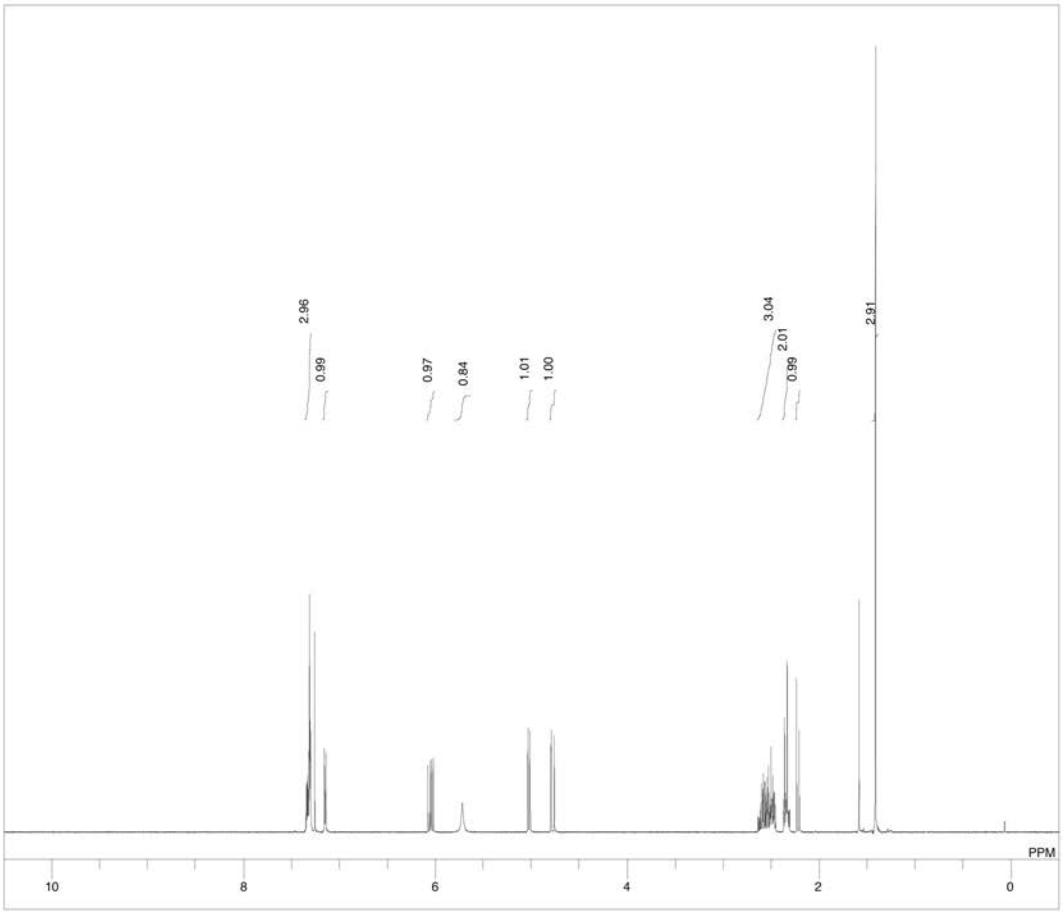
7



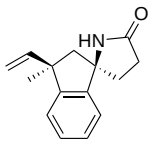
Pk #	Retention Time	Area	Area Percent
1	6.258	1026258	50.648
2	6.934	999999	49.352



Pk #	Retention Time	Area	Area Percent
1	6.124	6295958	94.897
2	6.732	338546	5.103



MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-104.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1014
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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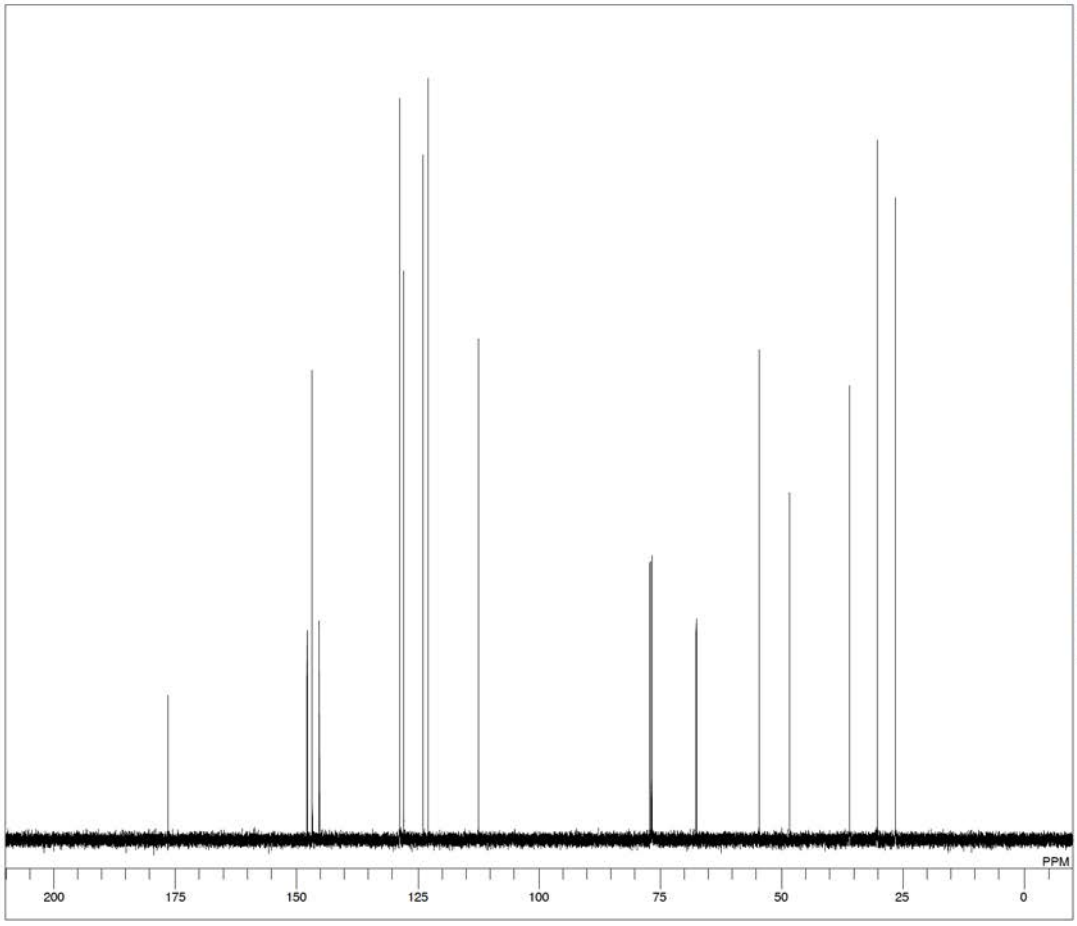
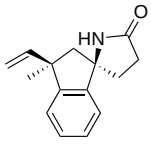
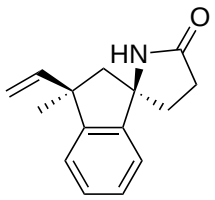


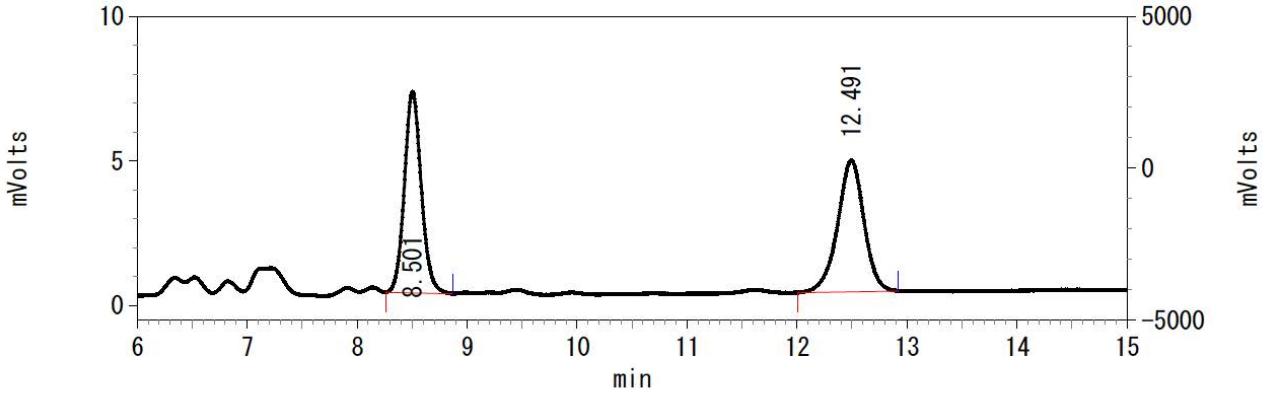
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-104-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	649
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



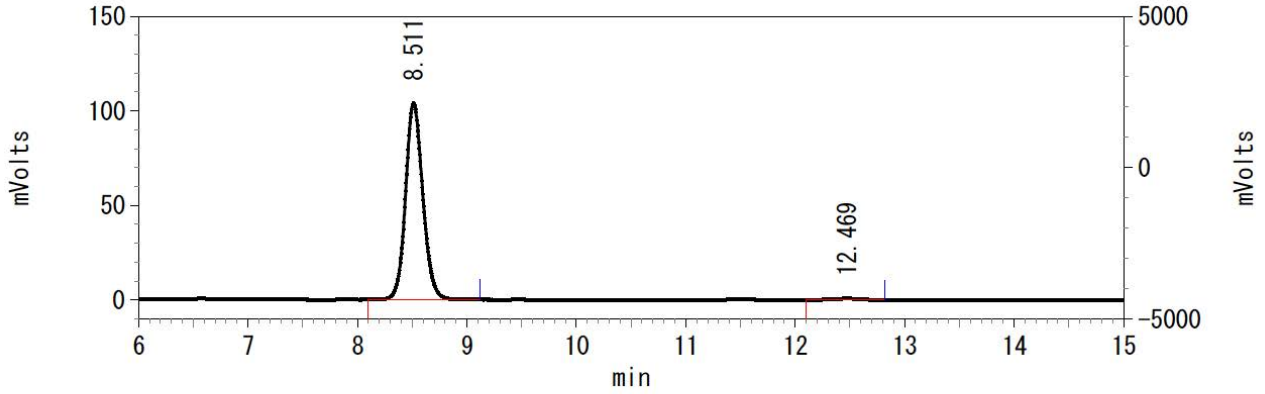
9



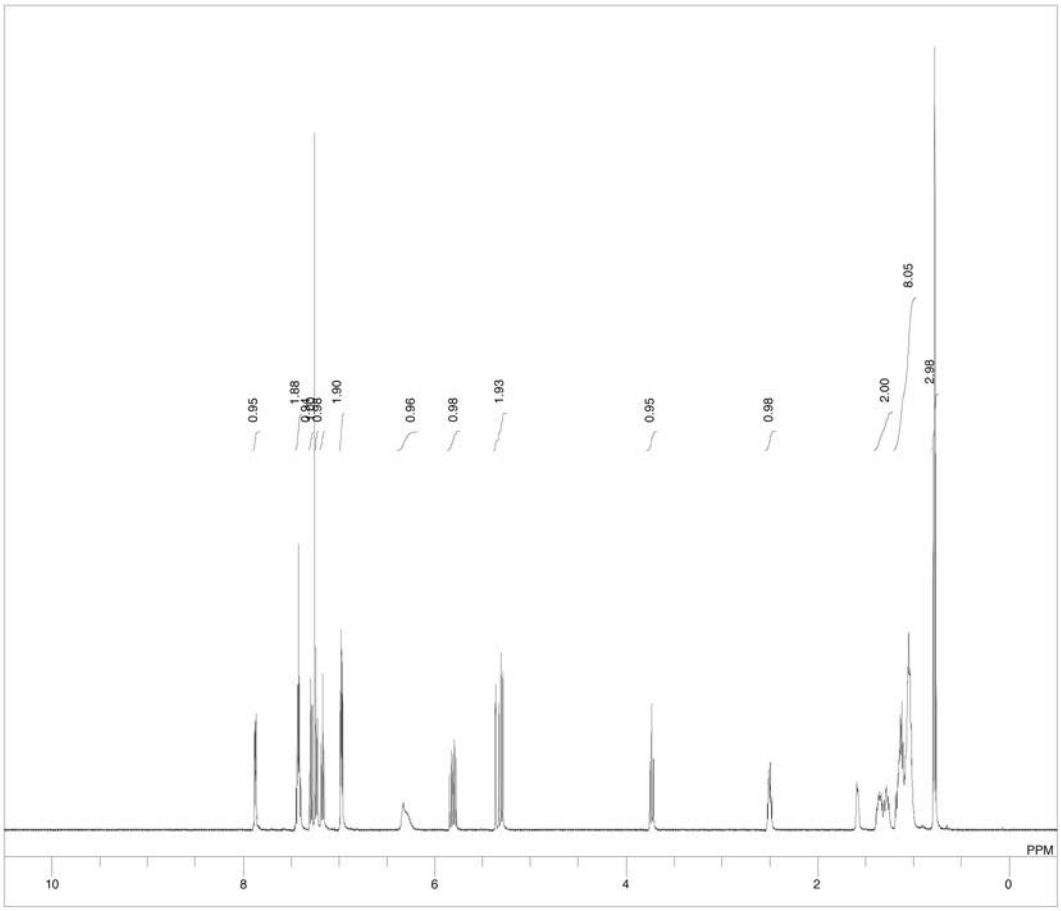
9



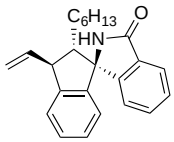
Pk #	Retention Time	Area	Area Percent
1	8.501	74396	49.564
2	12.491	75704	50.436



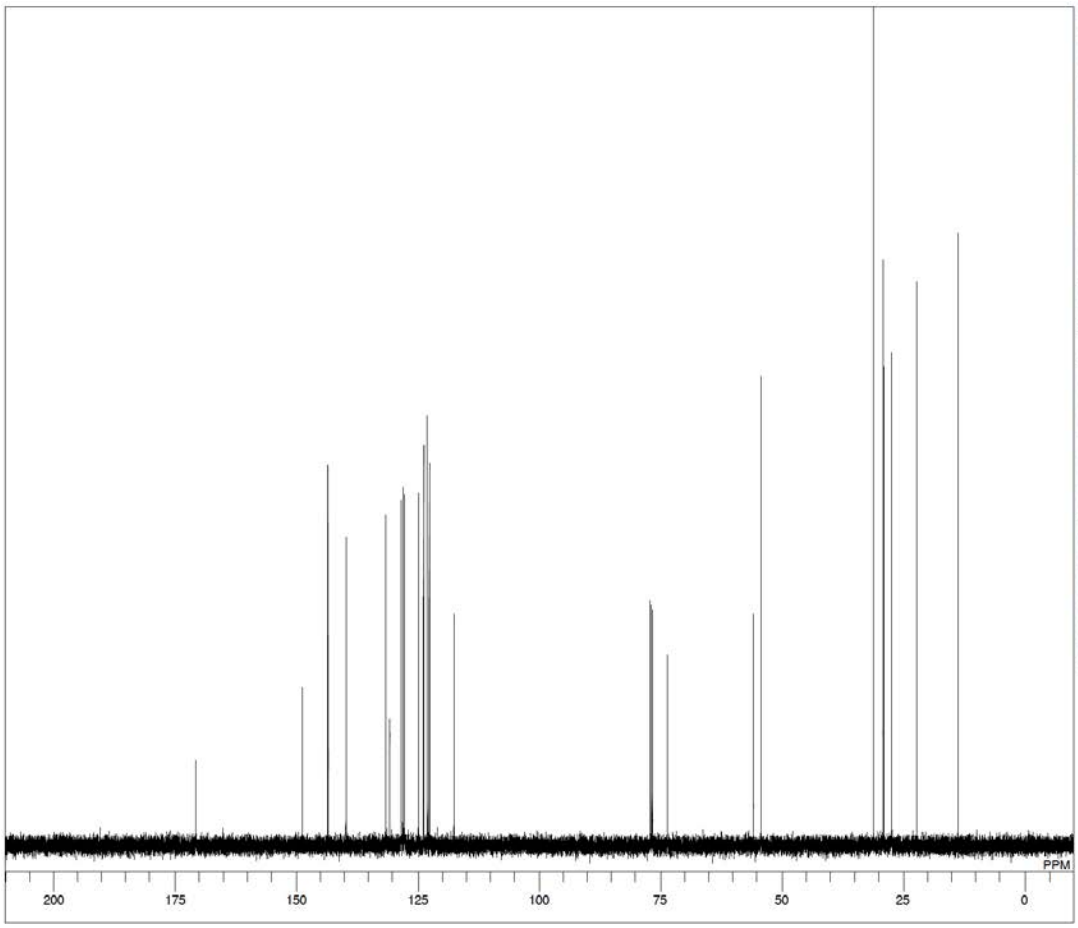
Pk #	Retention Time	Area	Area Percent
1	8.511	1142949	99.028
2	12.469	11223	0.972



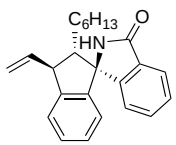
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	17
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-122.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	829
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



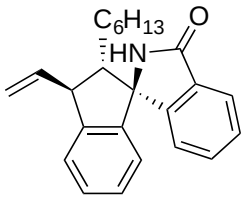
3an



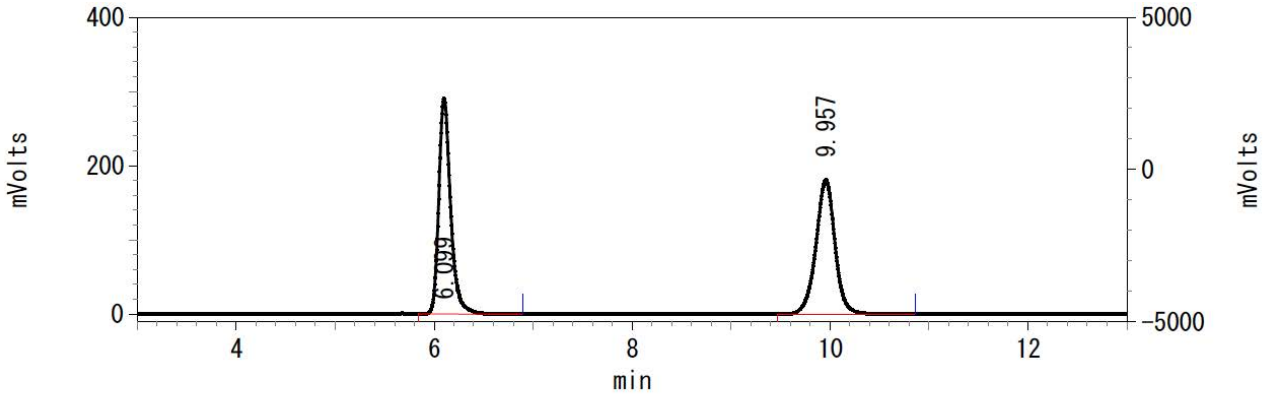
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	25
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-122-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	602
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



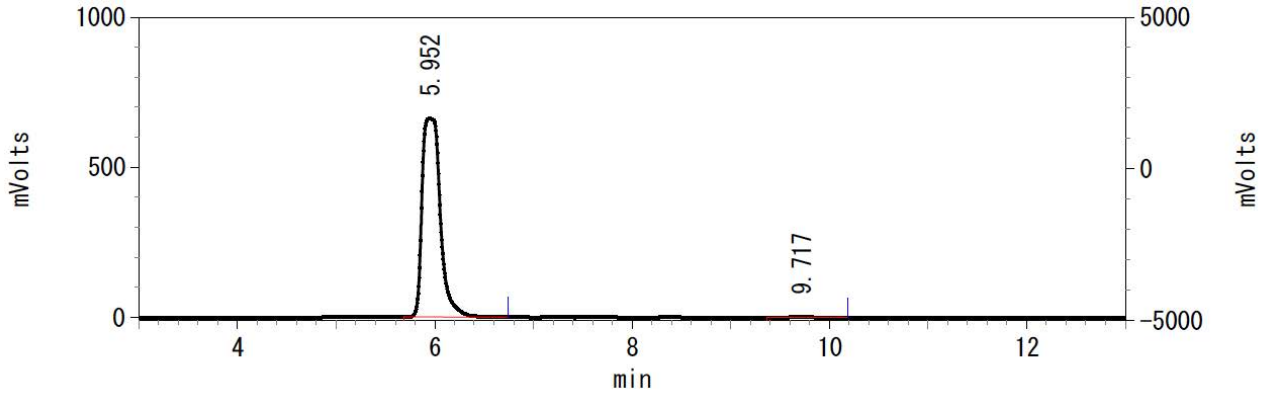
3an



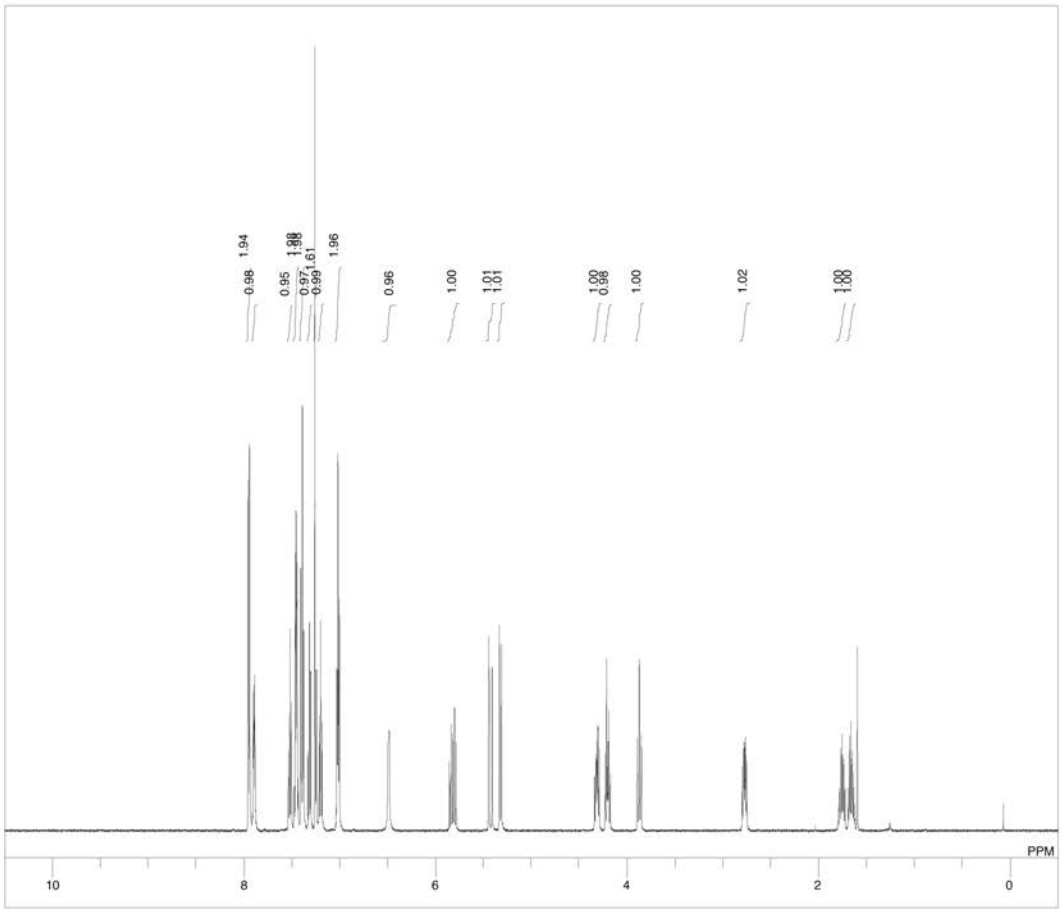
3an



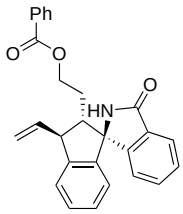
Pk #	Retention Time	Area	Area Percent
1	6.099	2422122	49.918
2	9.957	2430115	50.082



Pk #	Retention Time	Area	Area Percent
1	5.952	8496780	99.787
2	9.717	18172	0.213



MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	17
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-145.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1001
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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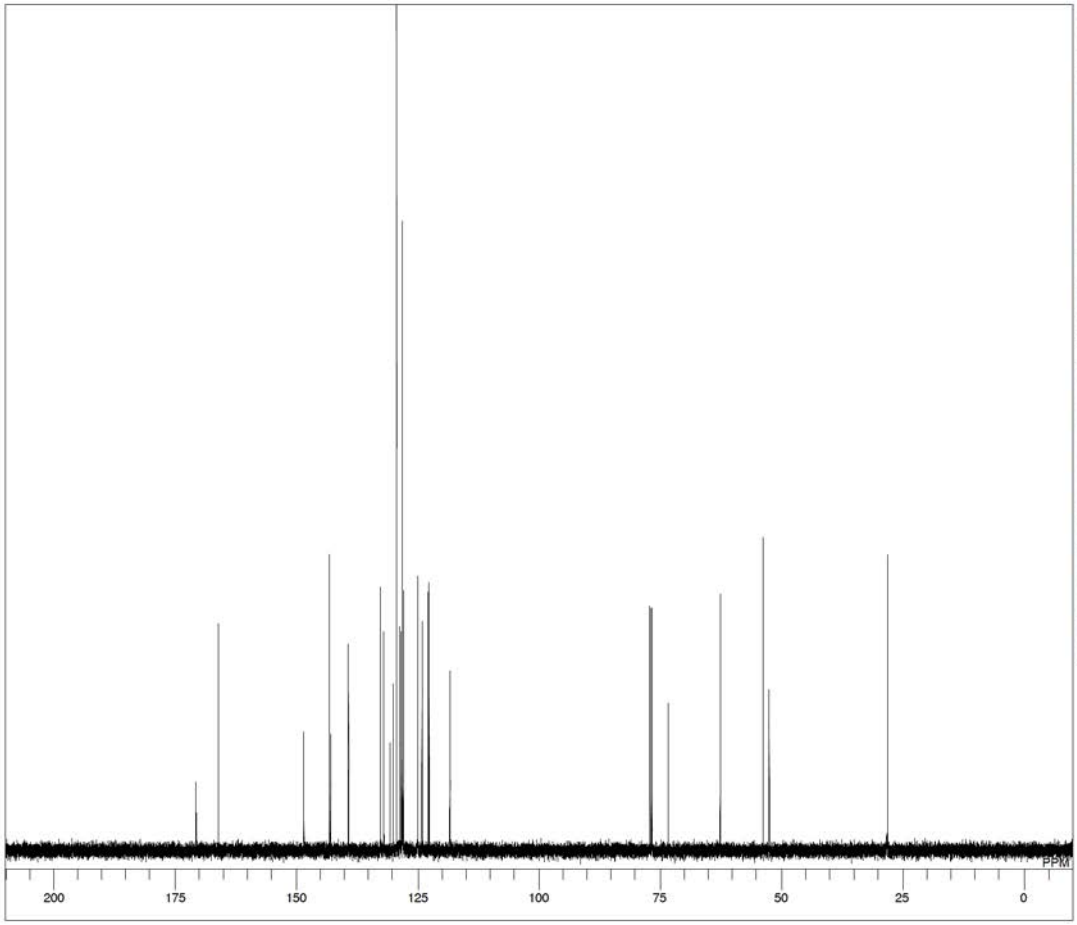
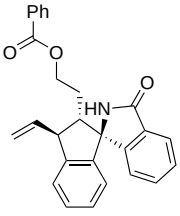


MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-145-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	752
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



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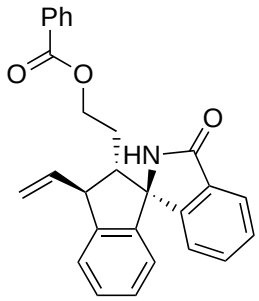
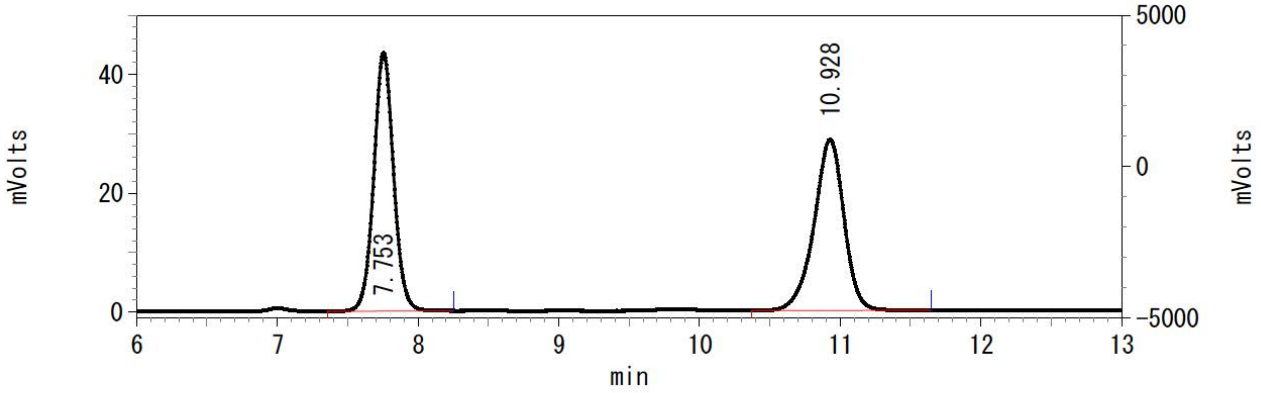
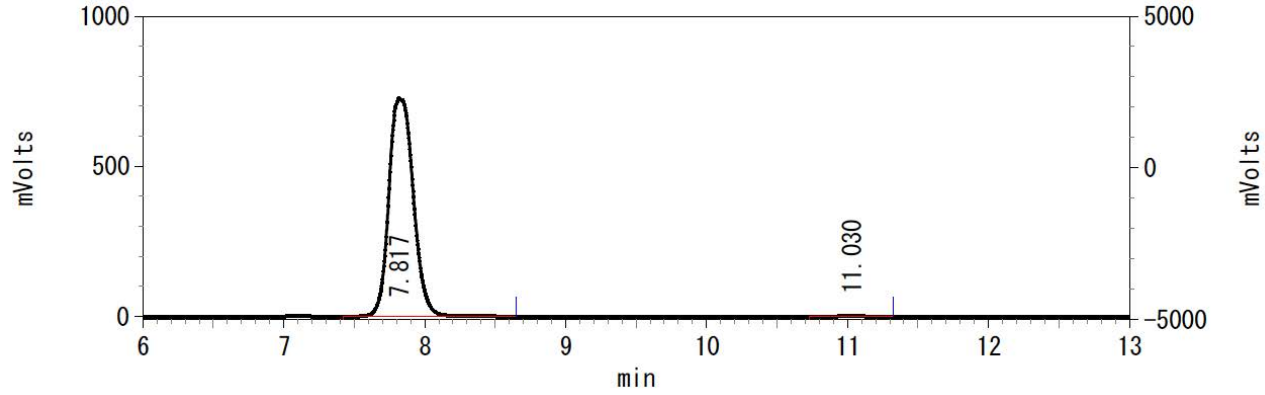


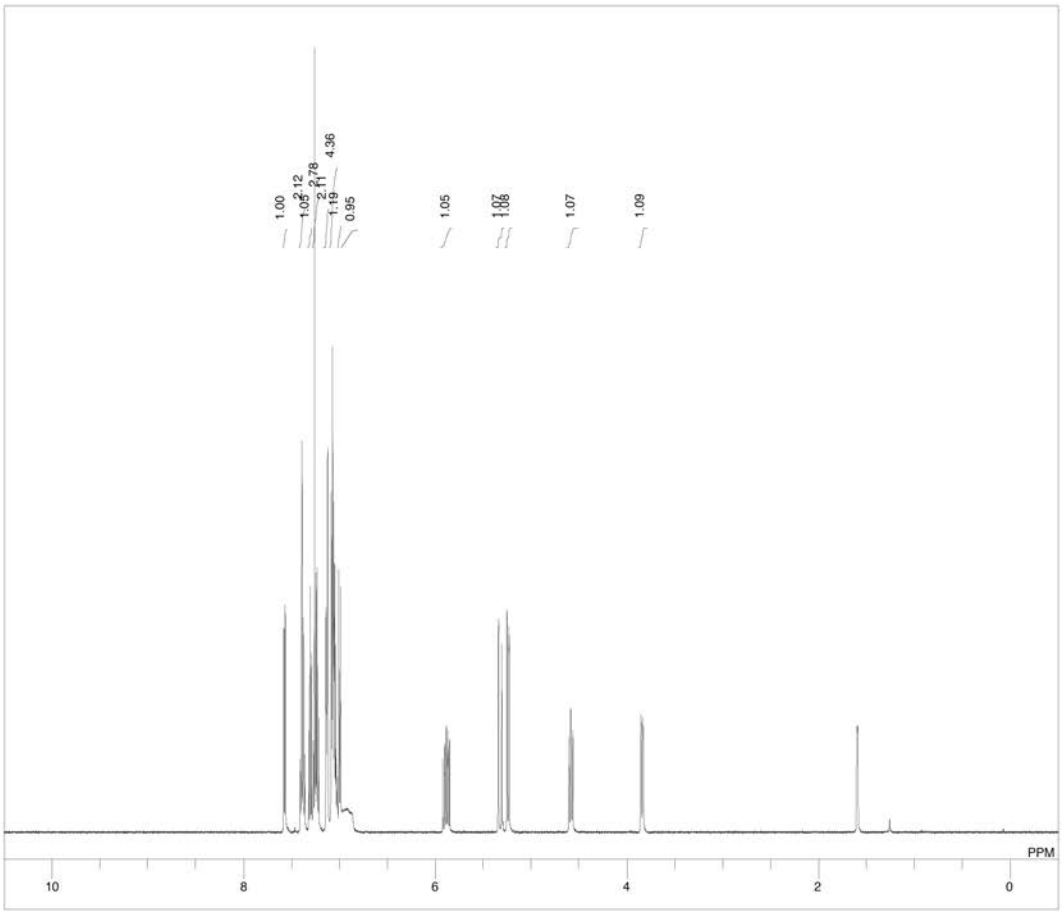
3ao



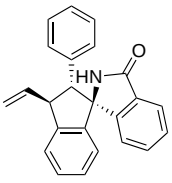
Pk #	Retention Time	Area	Area Percent
1	7.753	426281	49.942
2	10.928	427272	50.058



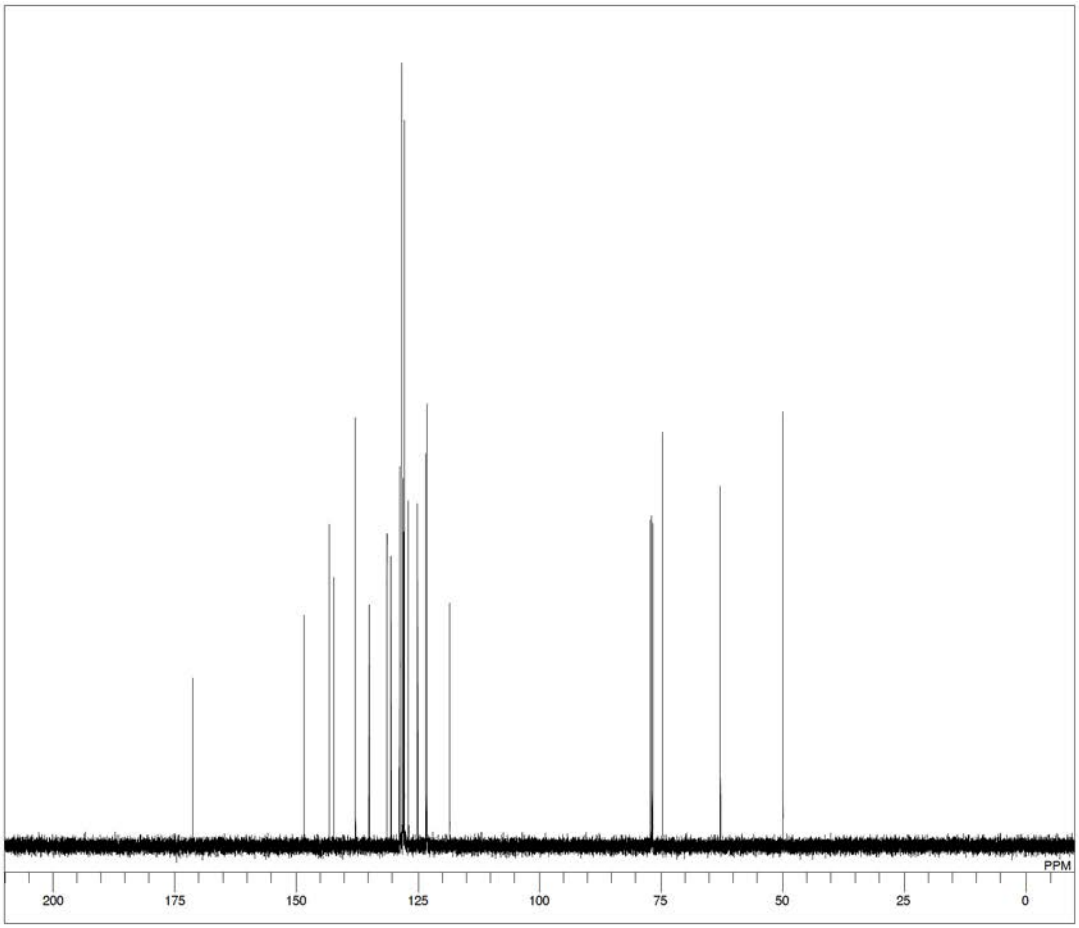
Pk #	Retention Time	Area	Area Percent
1	7.817	8753755	99.805
2	11.030	17098	0.195



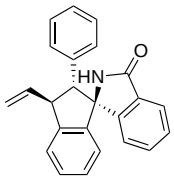
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	18
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-81.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1060
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



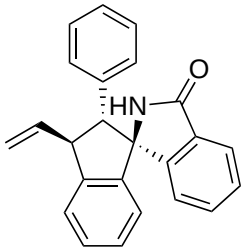
3ap



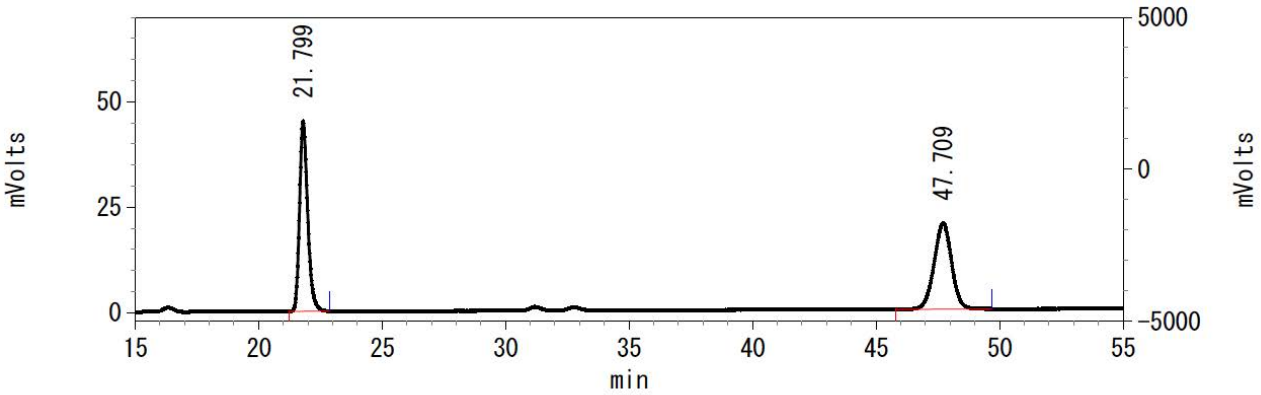
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	23
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-81-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	810
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



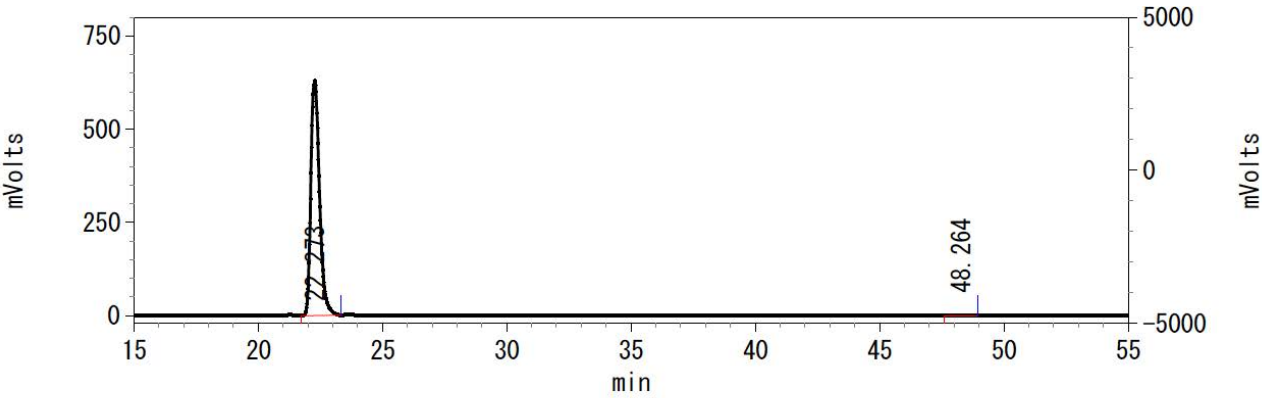
3ap



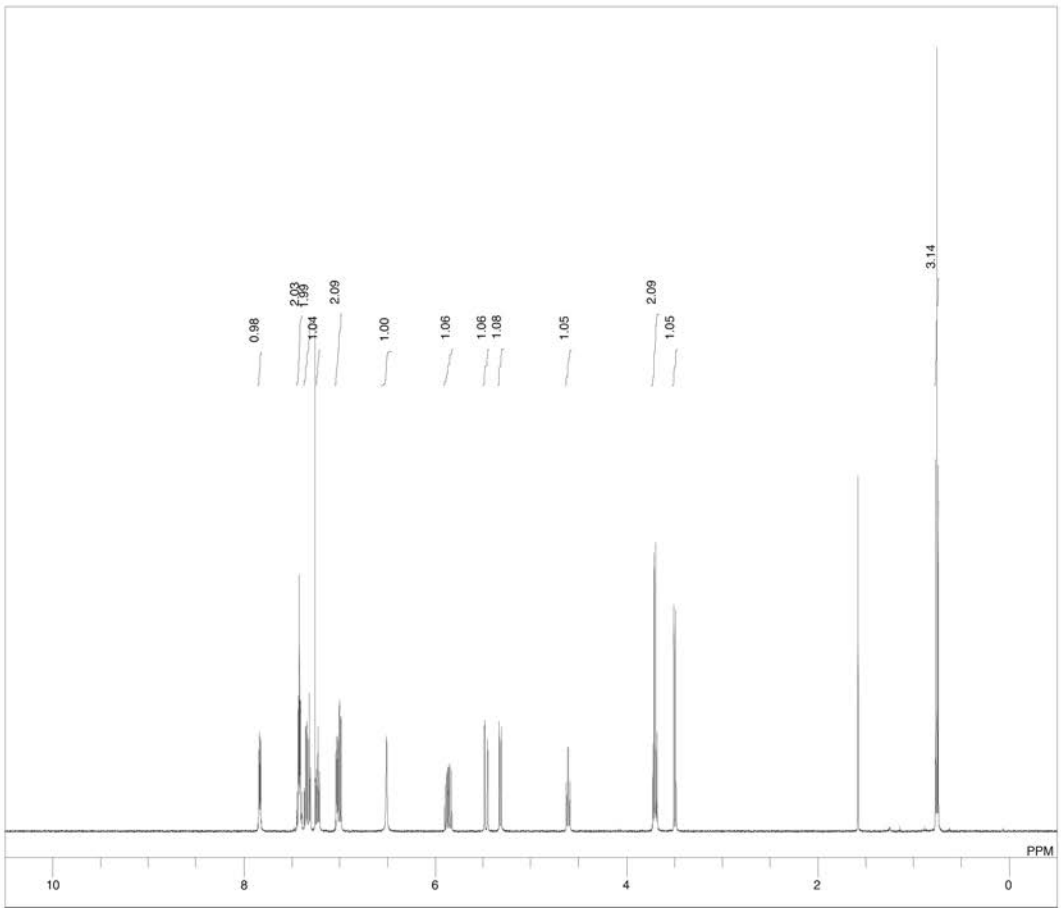
3ap



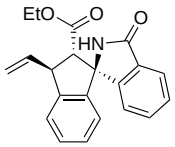
Pk #	Retention Time	Area	Area Percent
1	21.799	1030173	50.688
2	47.709	1002199	49.312



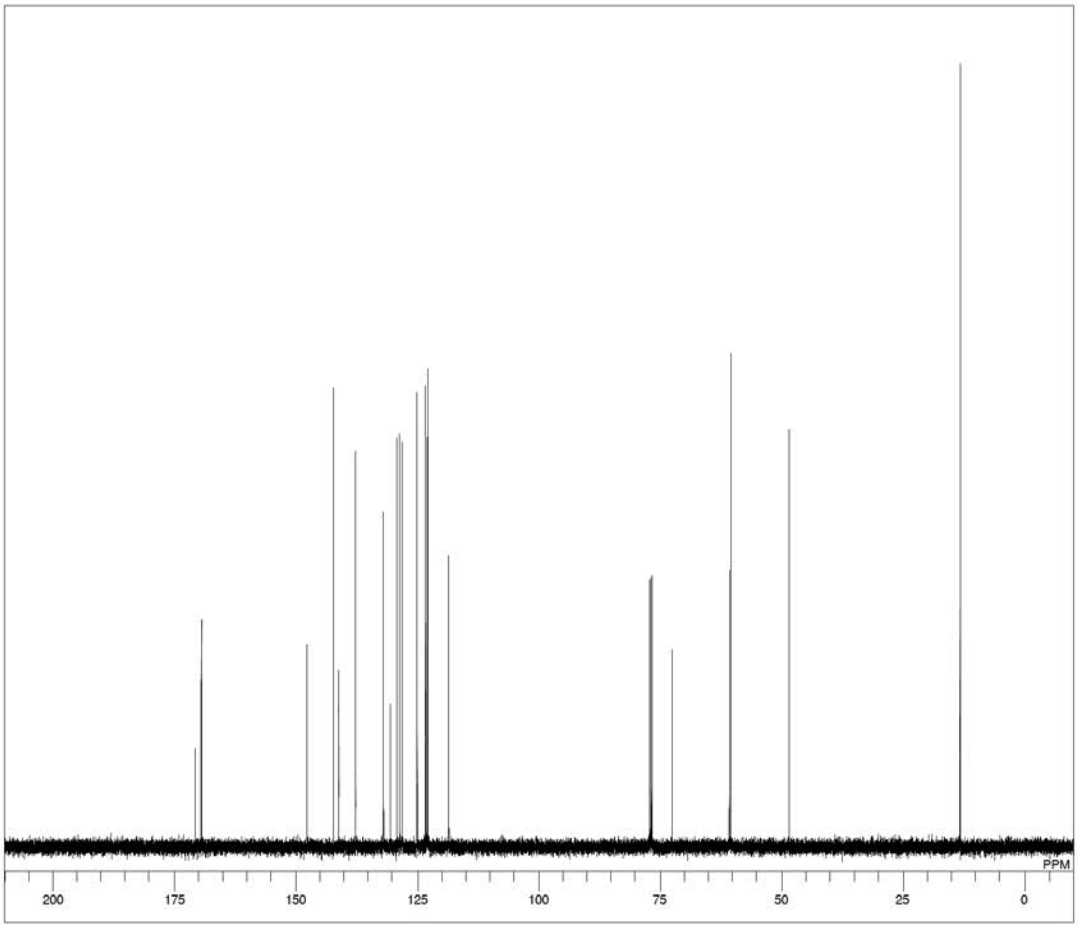
Pk #	Retention Time	Area	Area Percent
1	22.273	15303862	99.895
2	48.264	16160	0.105



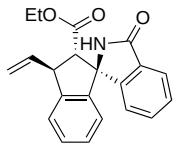
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	20
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-87'.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1061
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



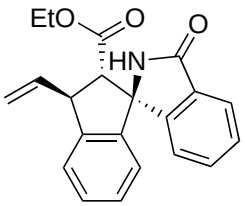
3aq



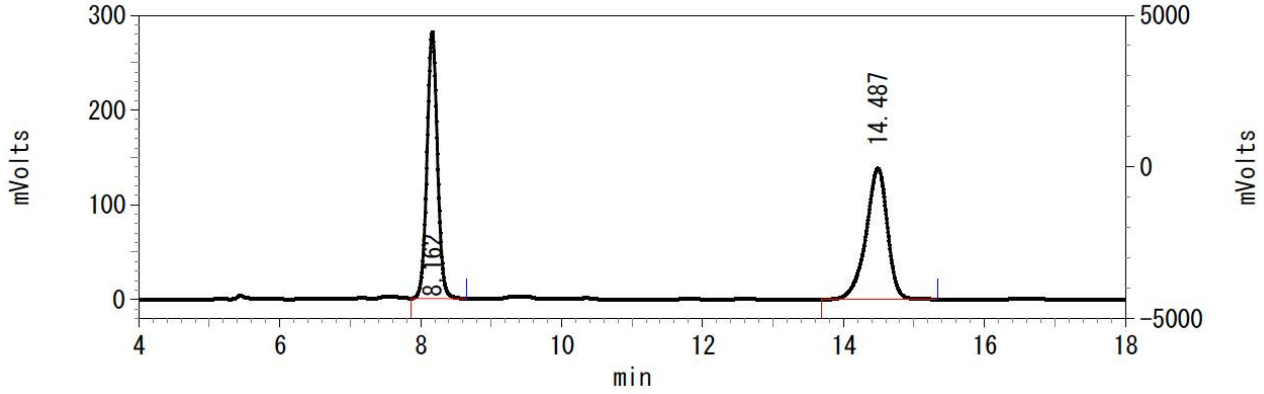
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-87-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	634
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



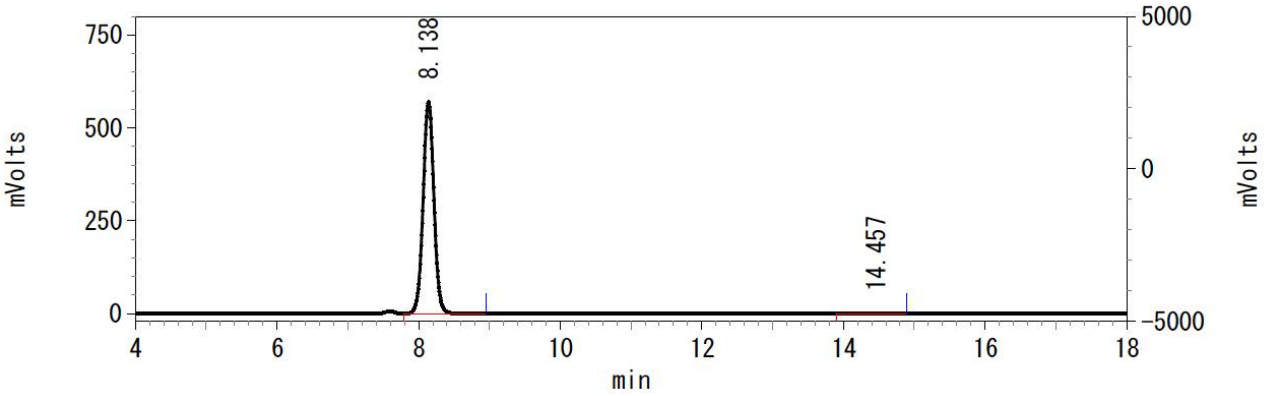
3aq



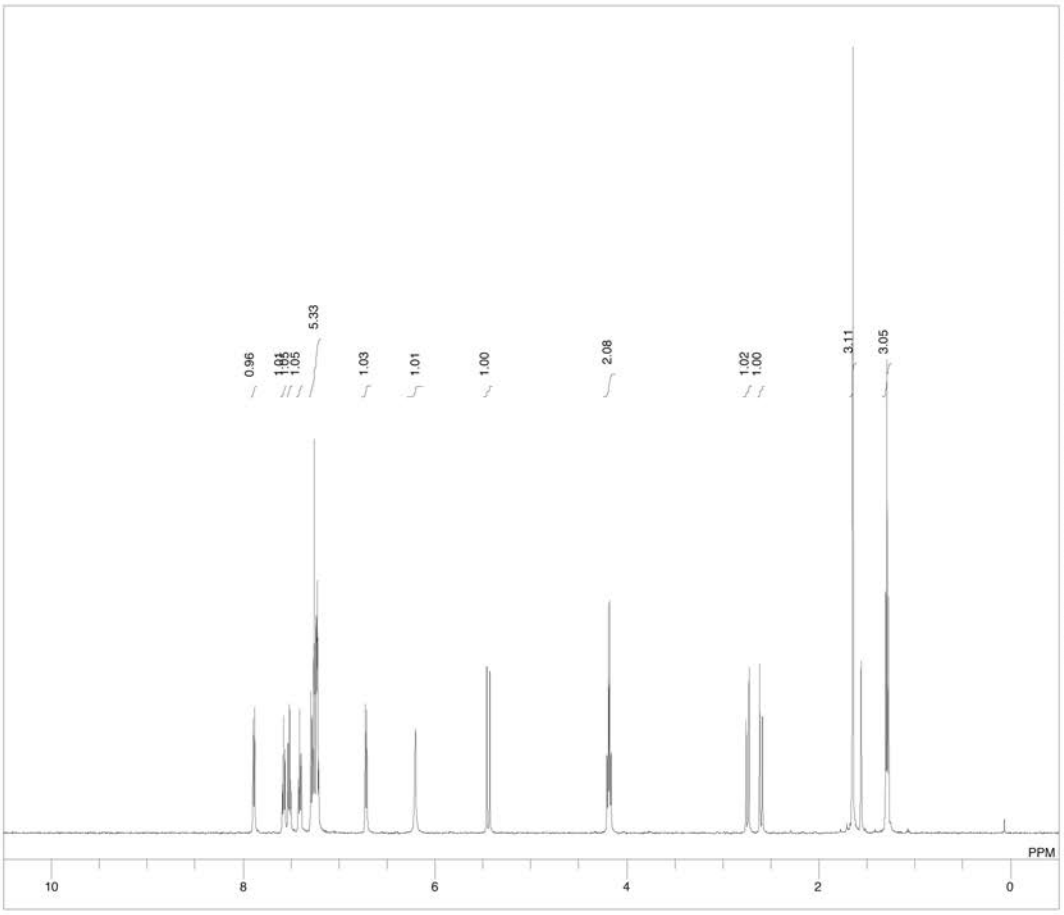
3aq



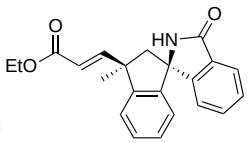
Pk #	Retention Time	Area	Area Percent
1	8.162	2906592	49.893
2	14.487	2919105	50.107



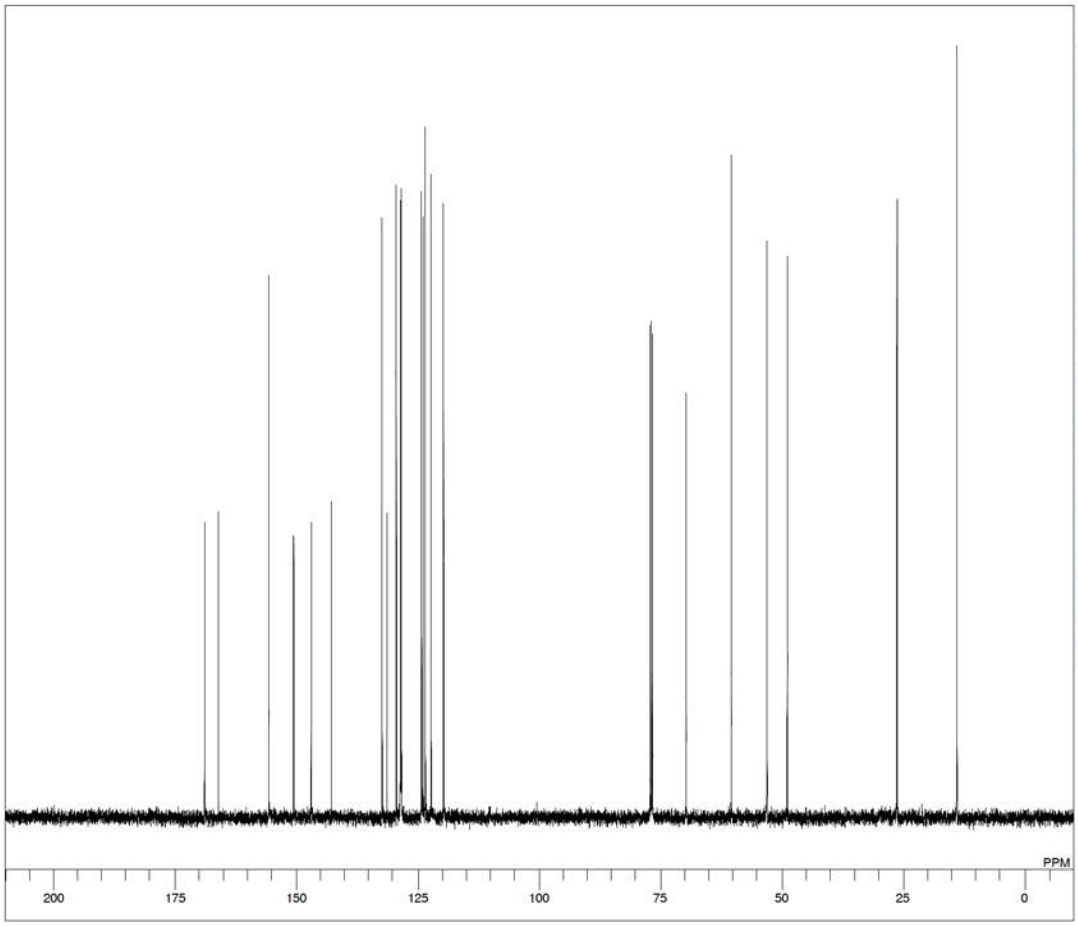
Pk #	Retention Time	Area	Area Percent
1	8.138	6067197	99.685
2	14.457	19143	0.315



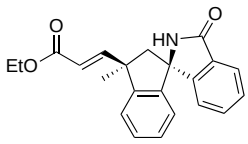
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	21
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-46.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1036
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



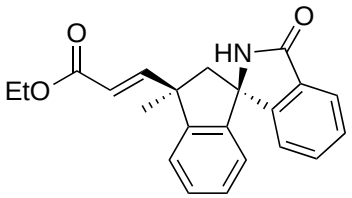
3ar



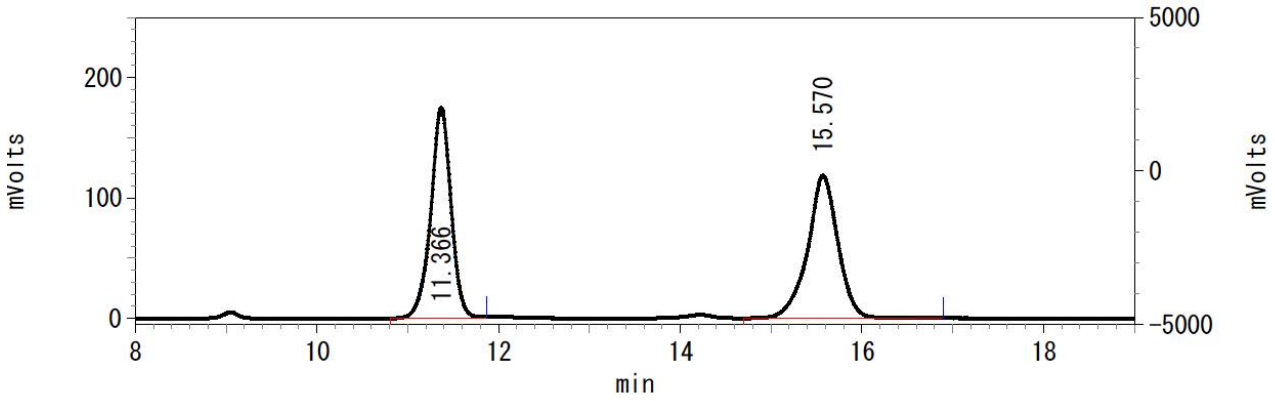
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-46-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	611
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



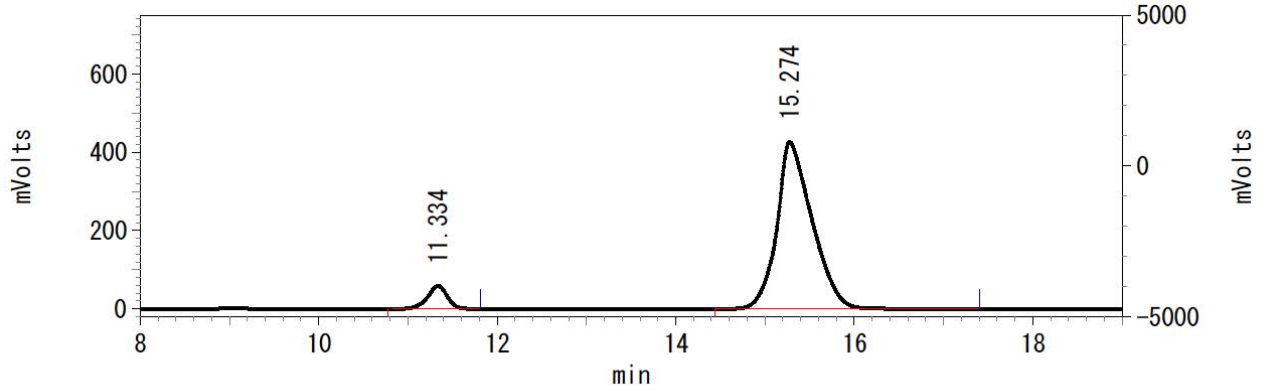
3ar



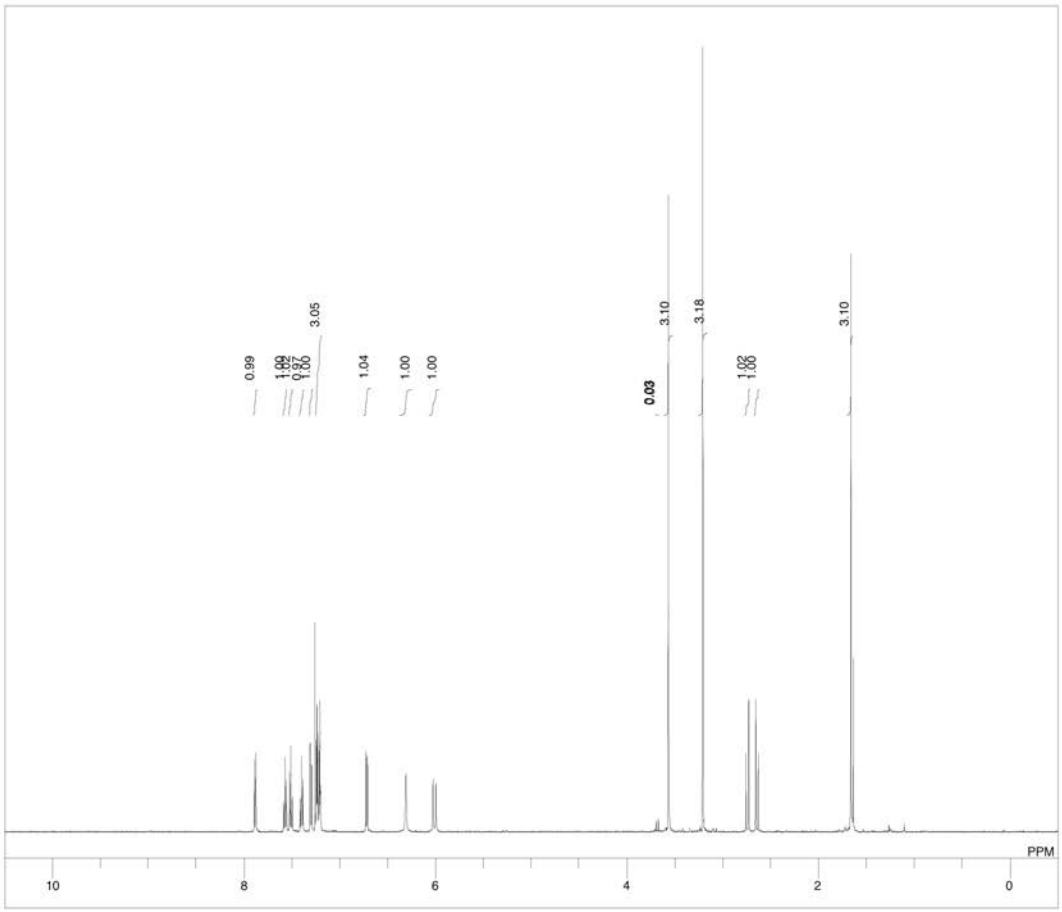
3ar



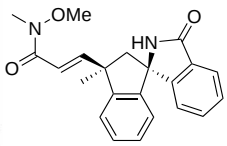
Pk #	Retention Time	Area	Area Percent
1	11.366	2742686	49.977
2	15.570	2745245	50.023



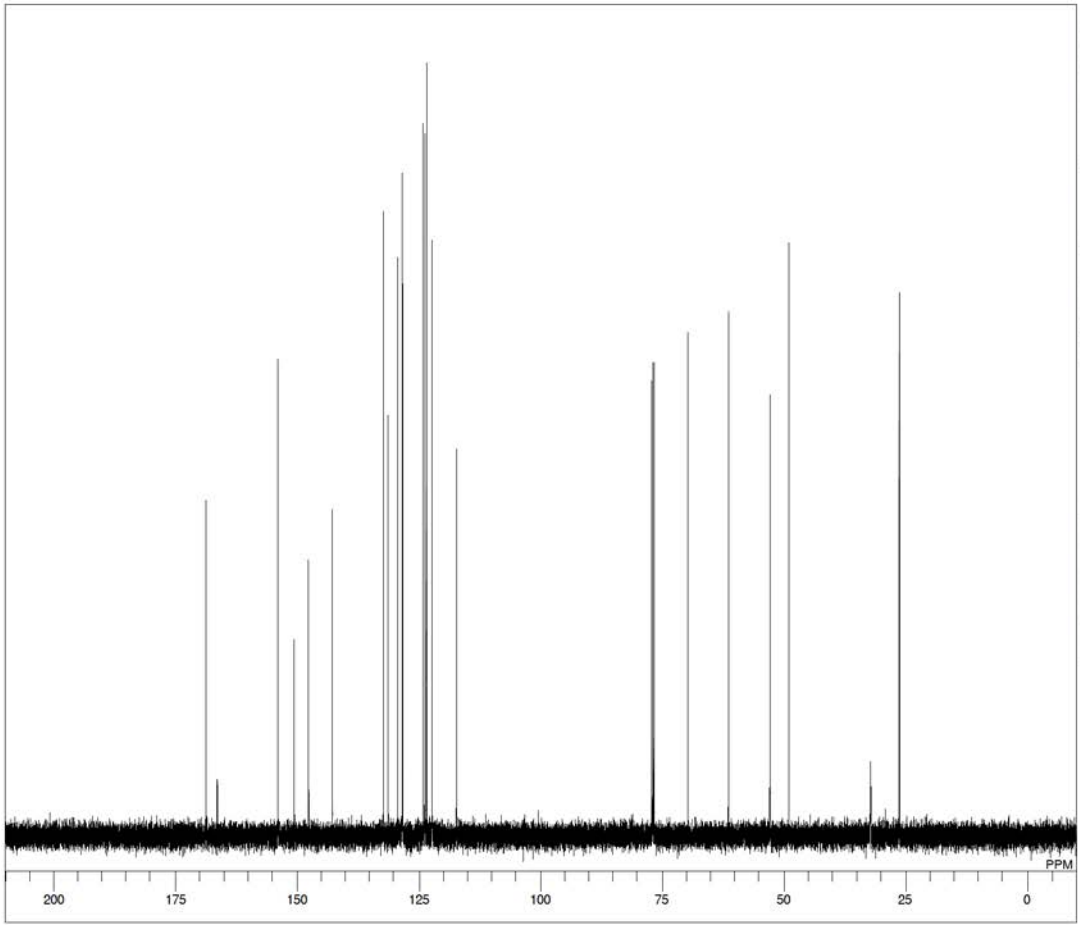
Pk #	Retention Time	Area	Area Percent
1	11.334	927905	7.342
2	15.274	11710332	92.658



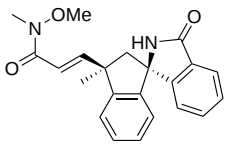
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	17
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-57.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1044
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



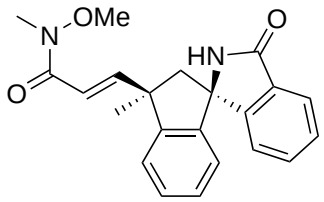
3as



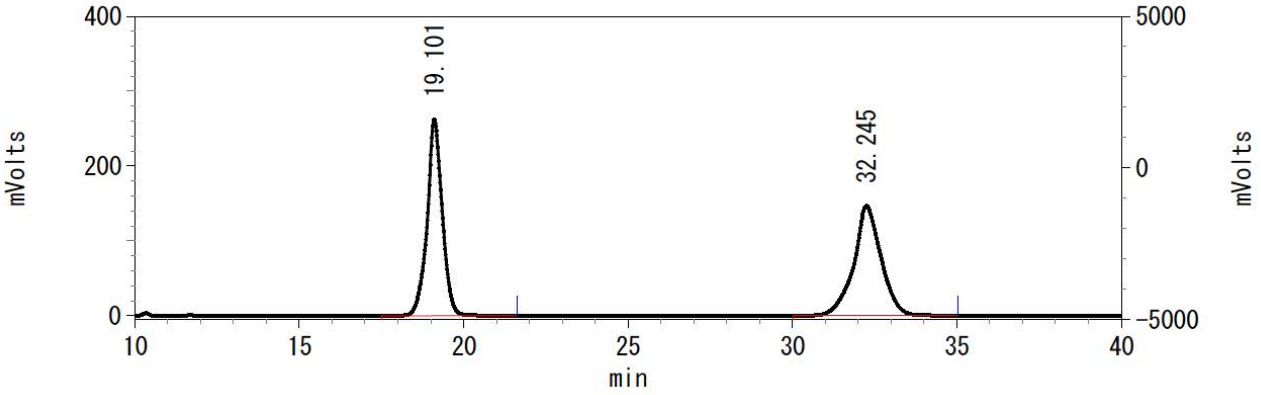
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-57-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	775
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



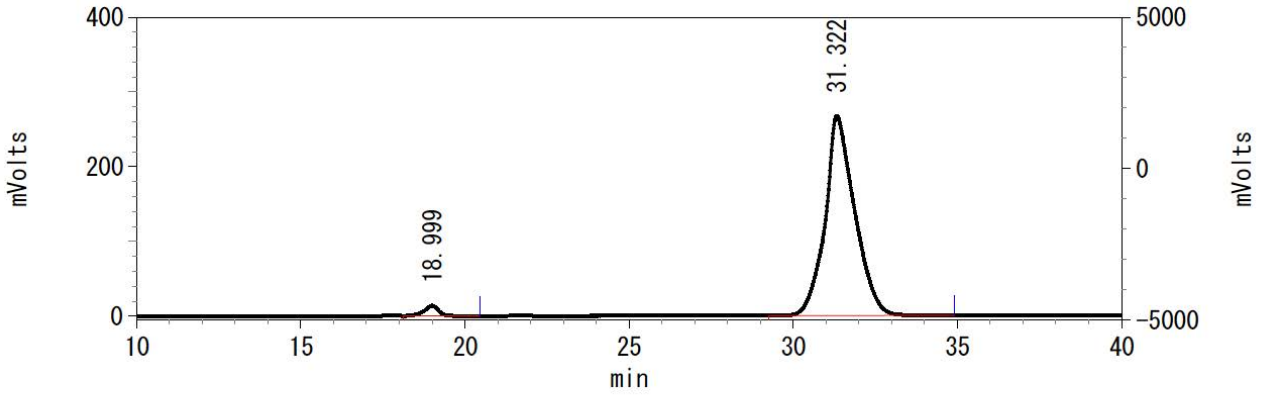
3as



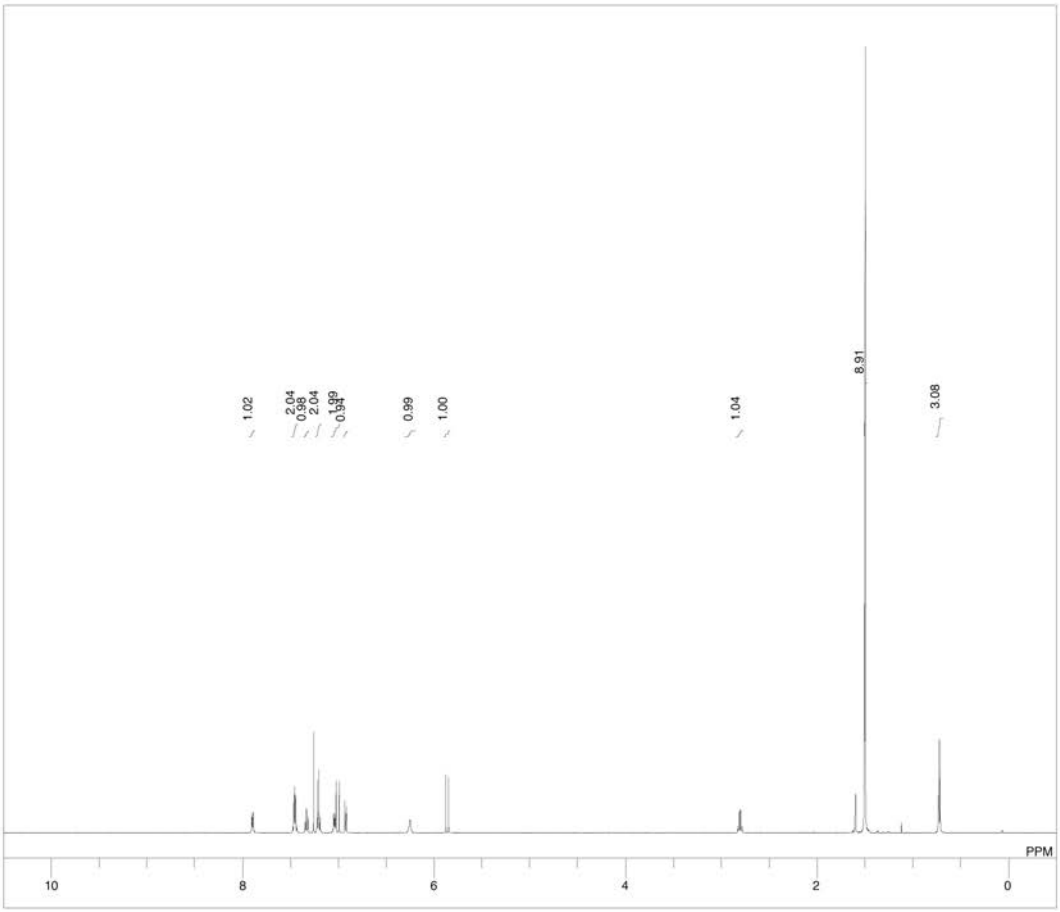
3as



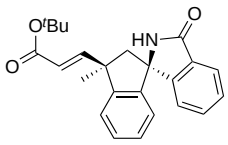
Pk #	Retention Time	Area	Area Percent
1	19.101	8365459	49.691
2	32.245	8469510	50.309



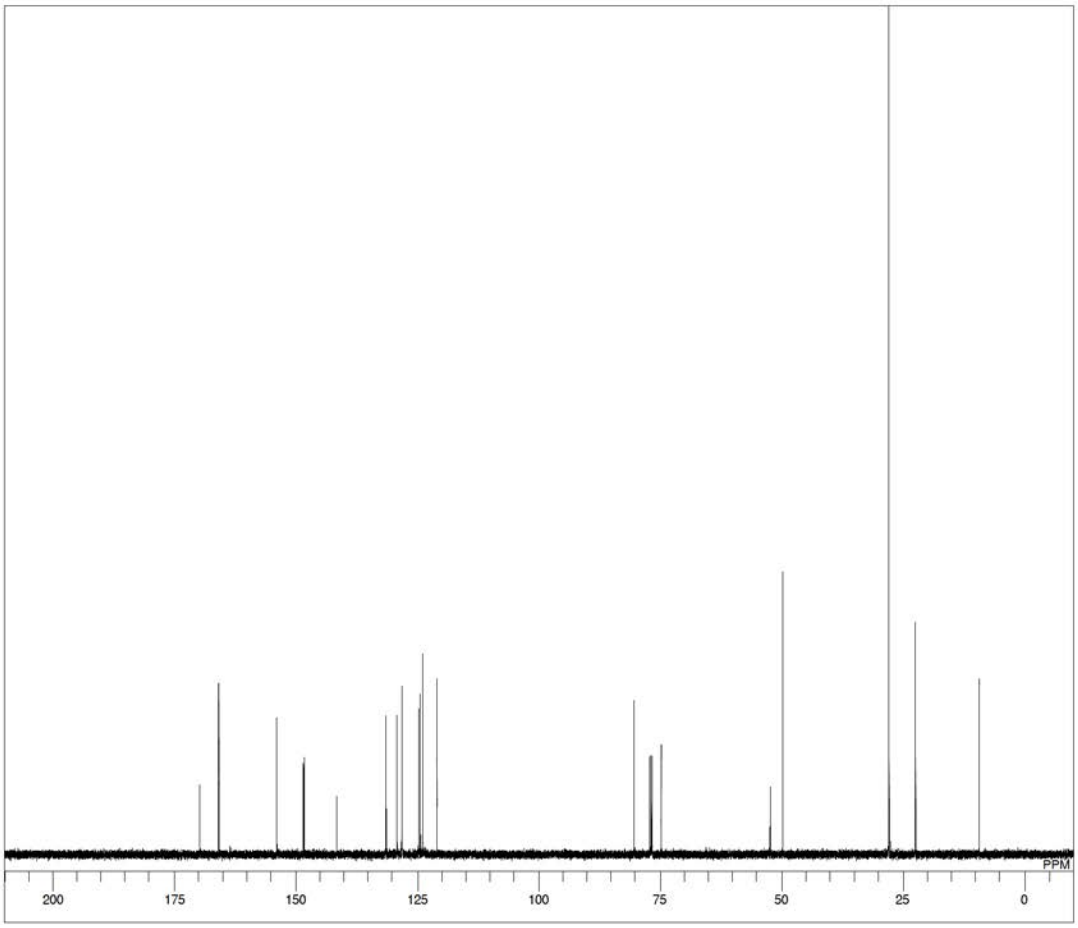
Pk #	Retention Time	Area	Area Percent
1	18.999	407573	2.375
2	31.322	16751867	97.625



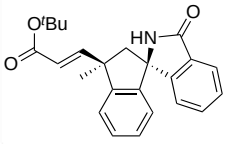
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	18
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-54.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1055
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



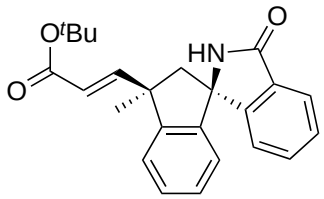
3at



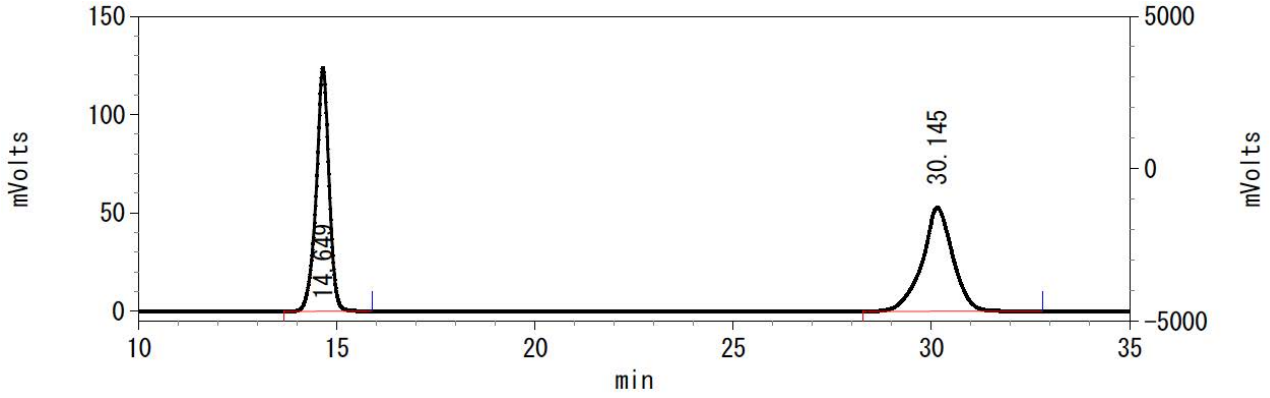
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	25
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-54-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	723
CSPED	10 Hz
FILDC	
FILDF	
SLVNT	CDCL3



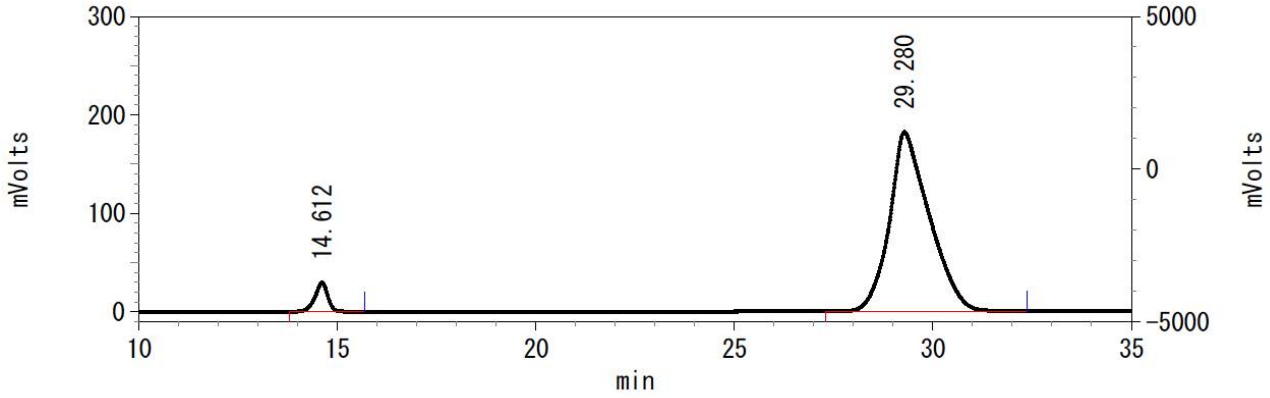
3at



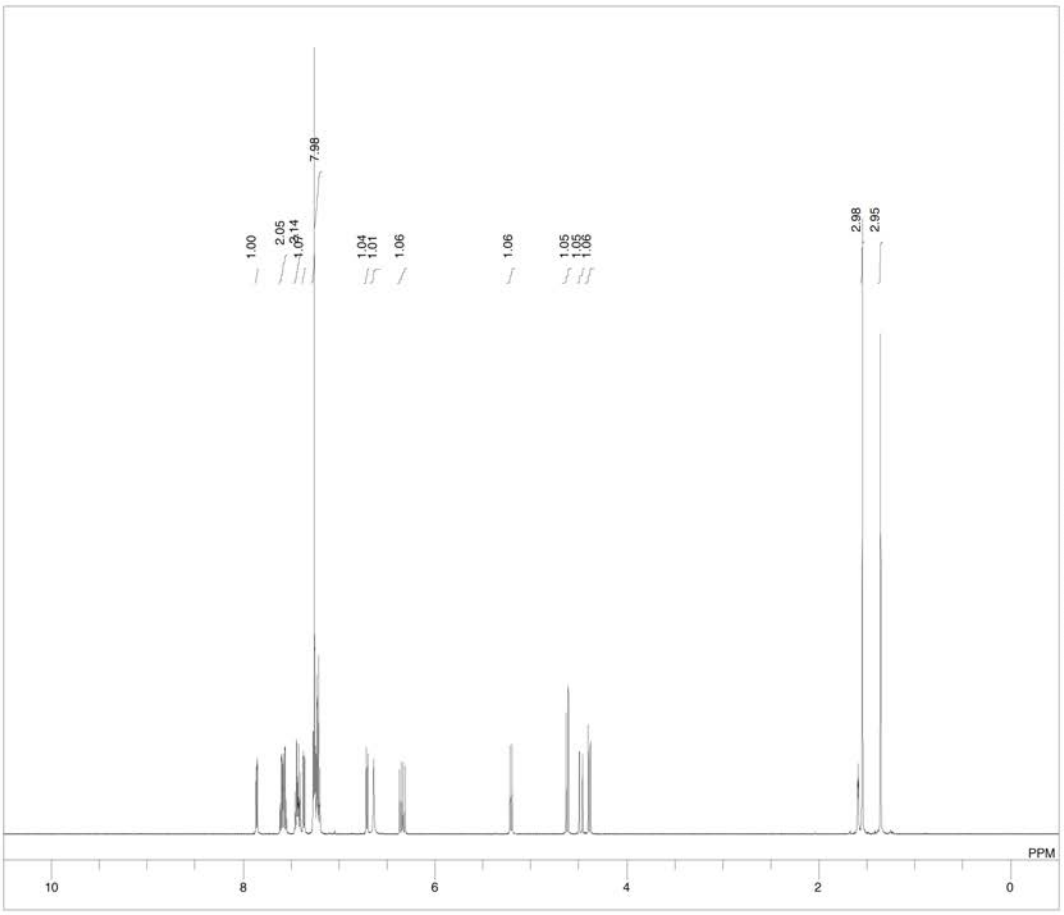
3at



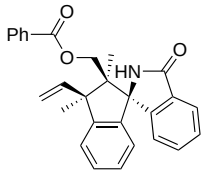
Pk #	Retention Time	Area	Area Percent
1	14.649	2806363	49.700
2	30.145	2840245	50.300



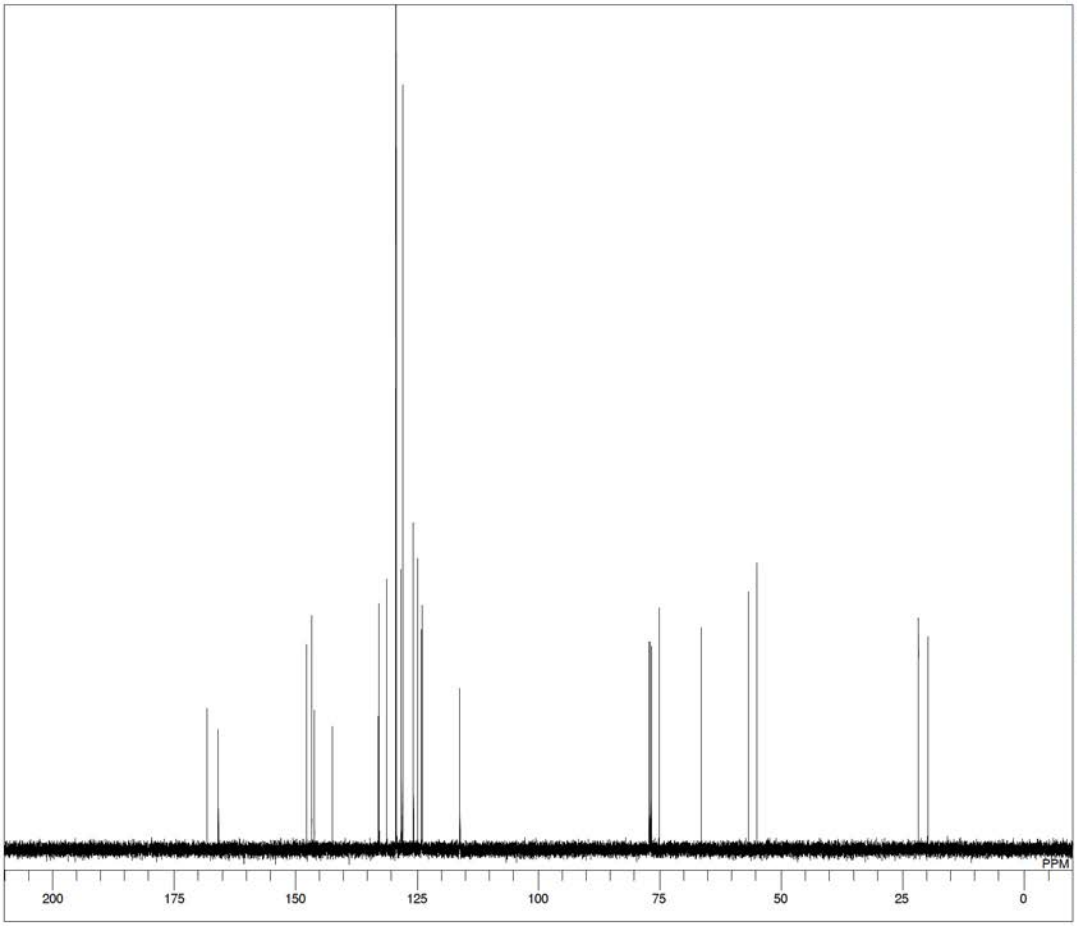
Pk #	Retention Time	Area	Area Percent
1	14.612	669576	5.001
2	29.280	12718674	94.999



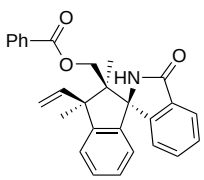
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	17
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-190.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	831
CSPED	12 Hz
FILDC	
FILDF	
SLVNT	CDCL3



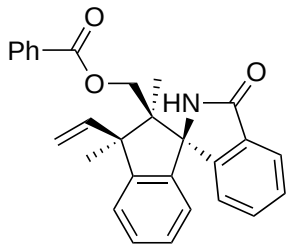
3au



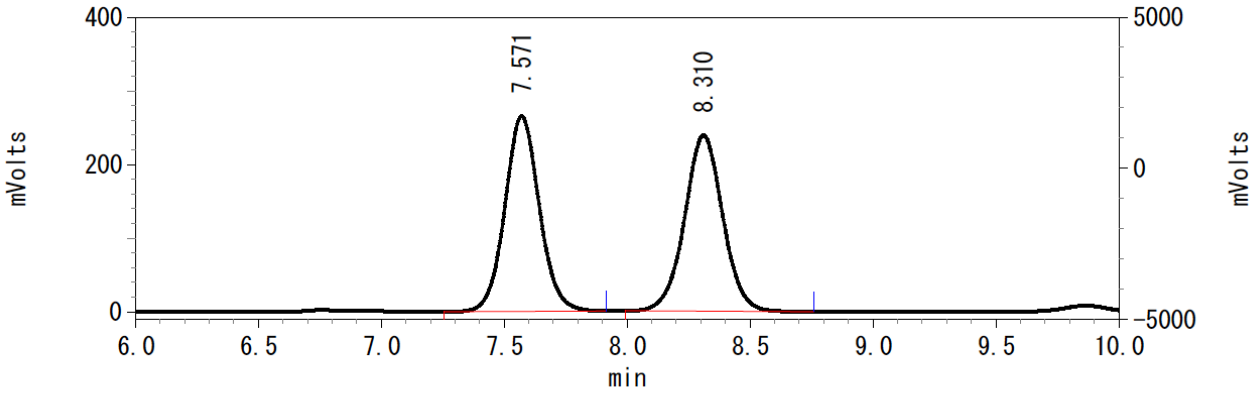
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-190-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	671
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



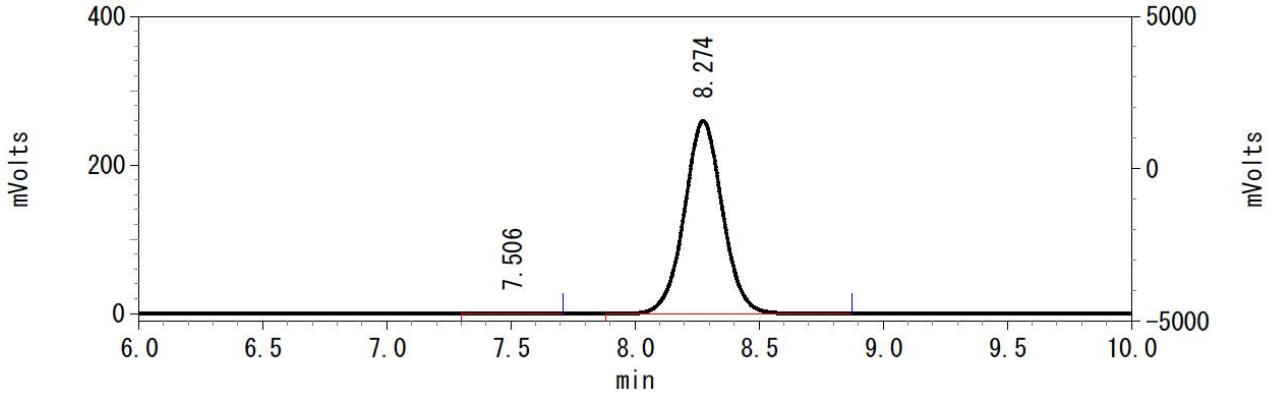
3au



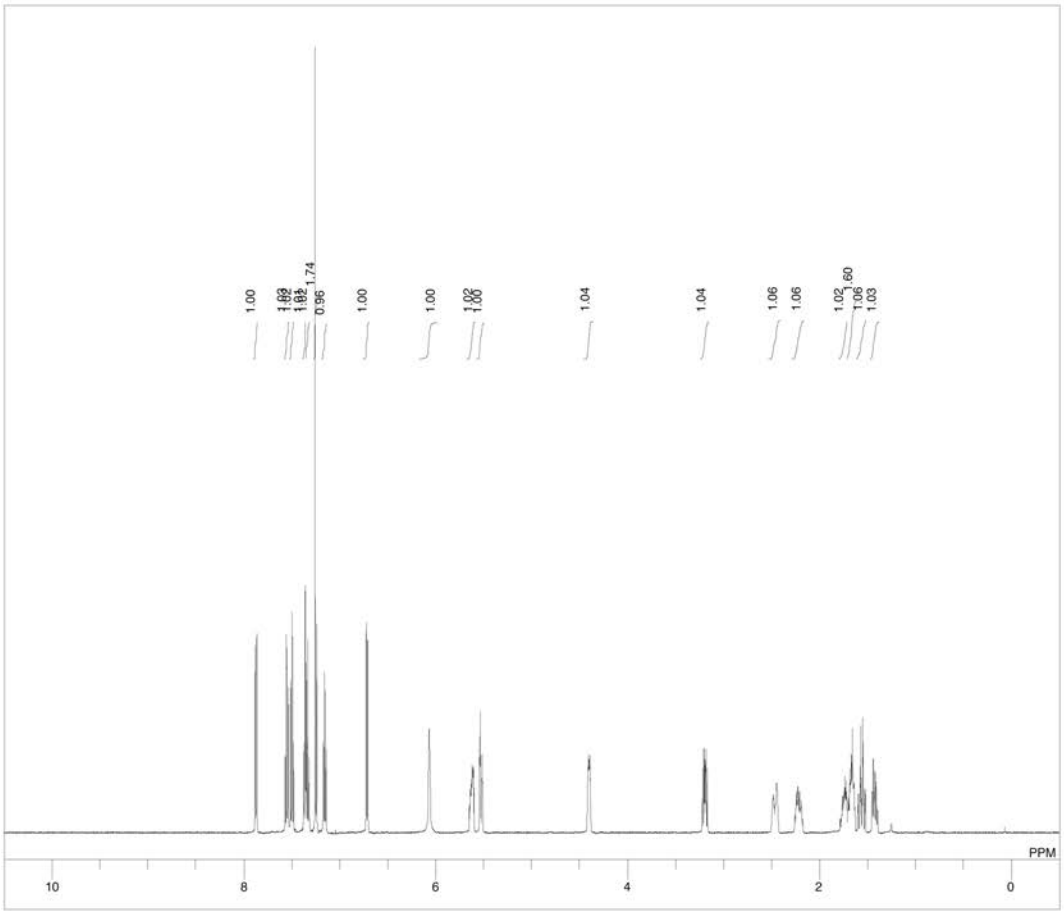
3au



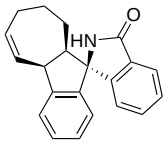
Pk #	Retention Time	Area	Area Percent
1	7.571	2585252	50.008
2	8.310	2584411	49.992



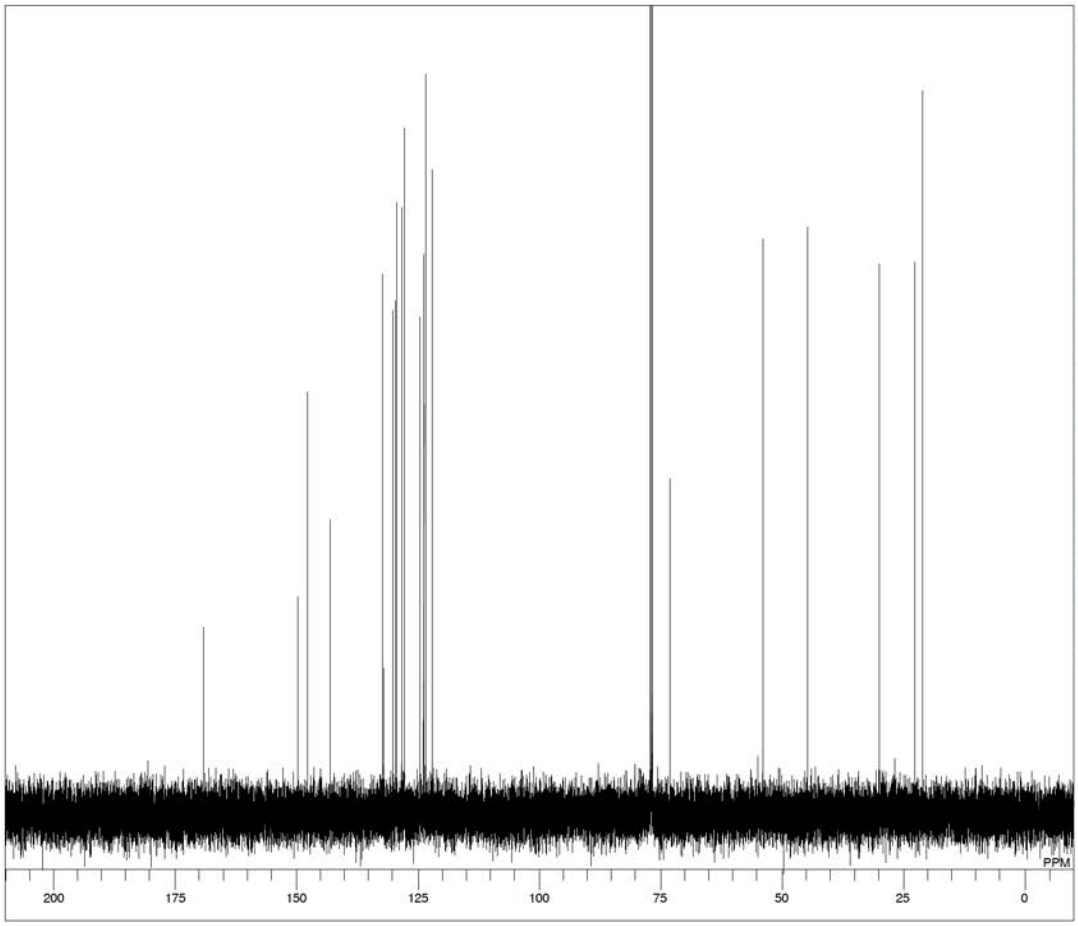
Pk #	Retention Time	Area	Area Percent
1	7.506	11637	0.416
2	8.274	2787508	99.584



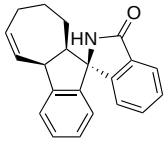
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE mn-2-188.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 1047
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3



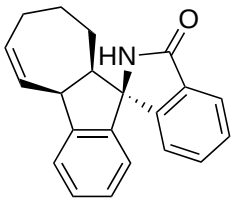
3av



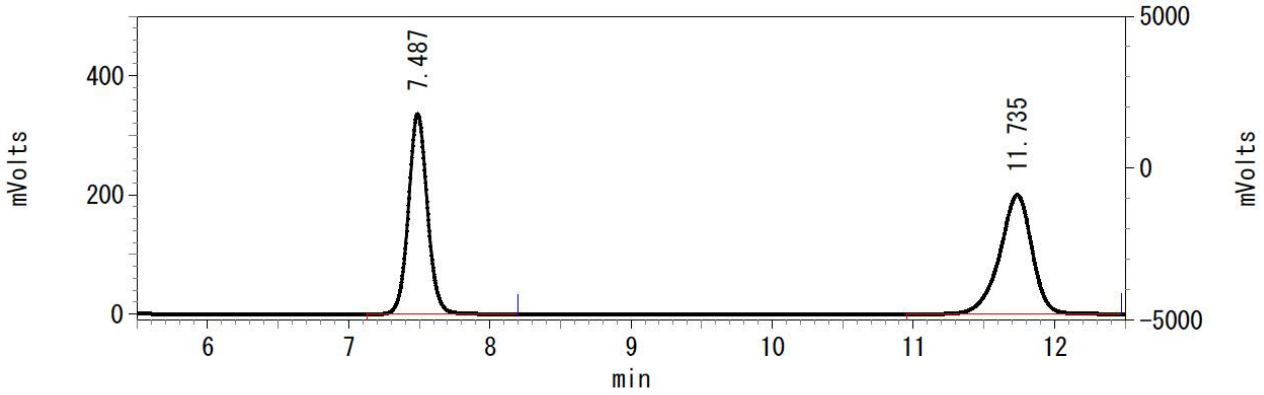
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 720
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE mn-2-188-cabon.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 343
LKSIG 680
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3



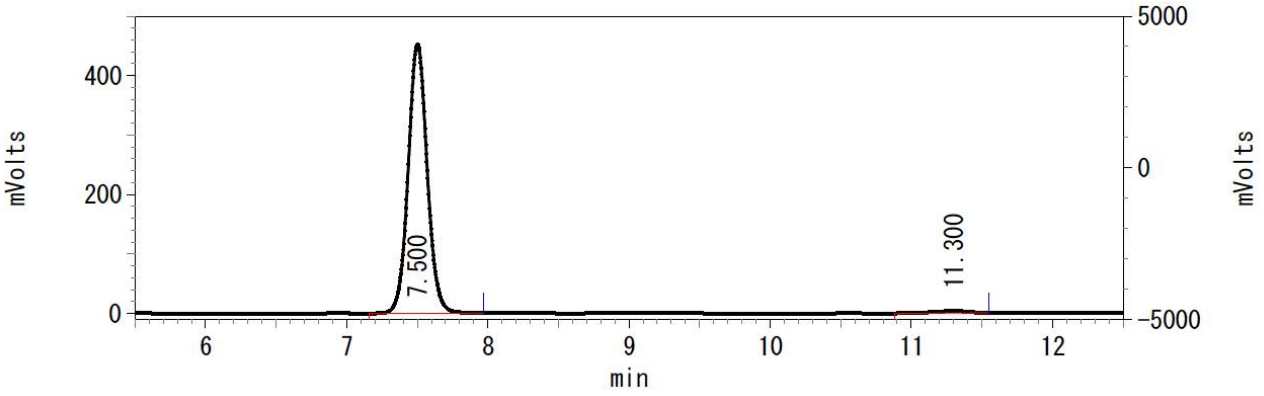
3av



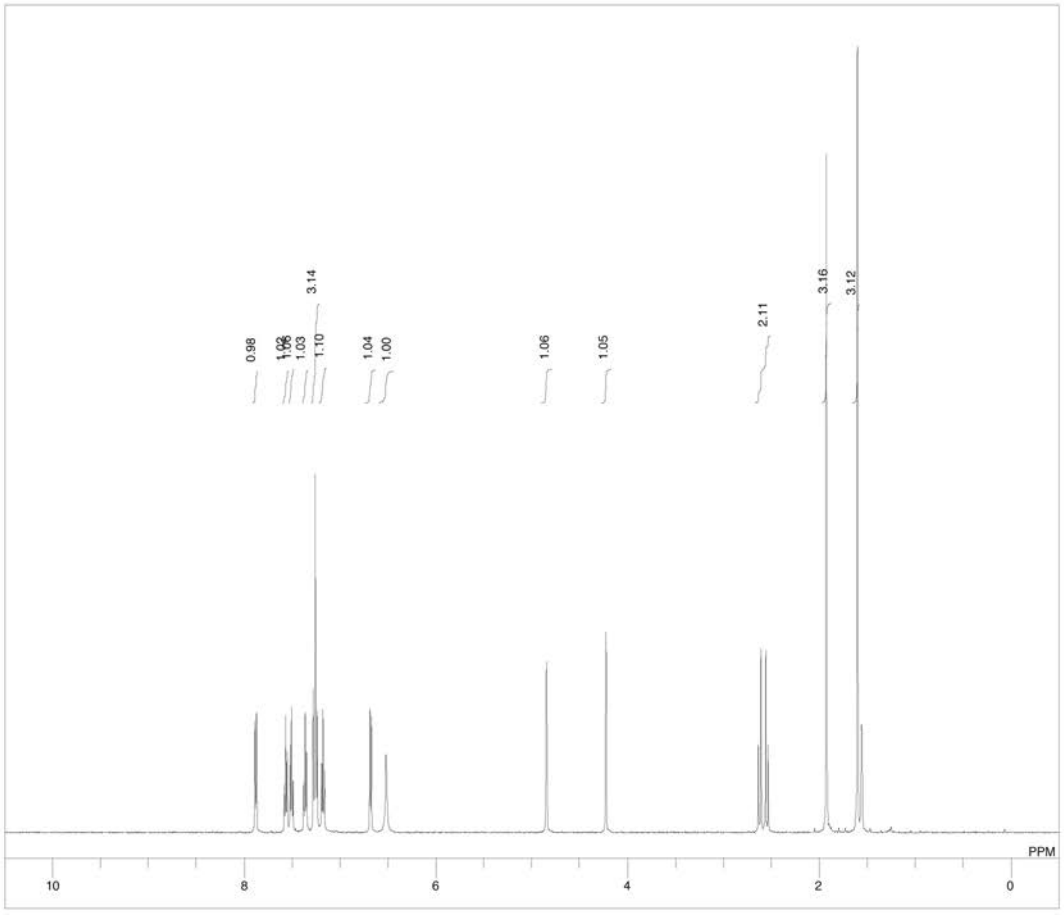
3av



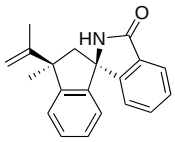
Pk #	Retention Time	Area	Area Percent
1	7.487	3135587	49.677
2	11.735	3176356	50.323



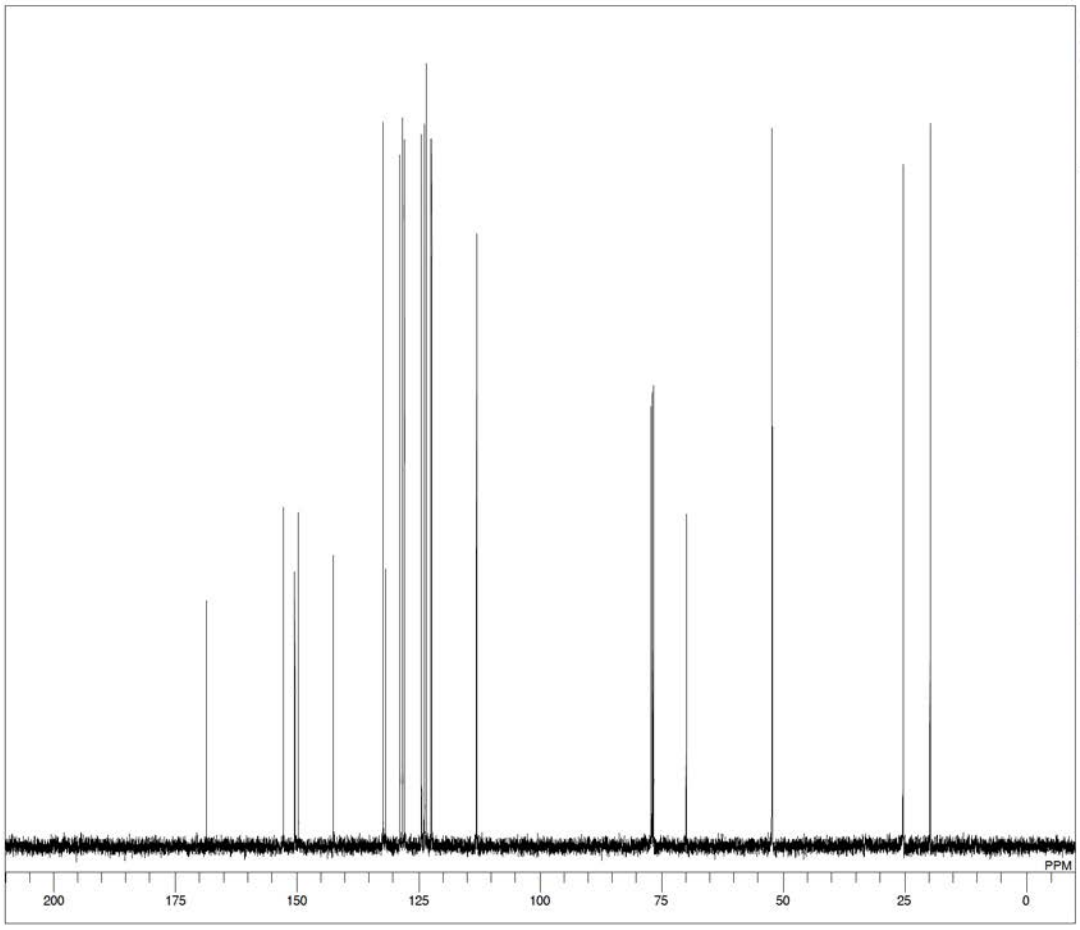
Pk #	Retention Time	Area	Area Percent
1	7.500	4239185	98.617
2	11.300	59456	1.383



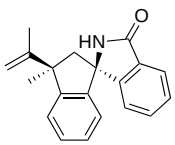
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	19
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-44.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	996
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



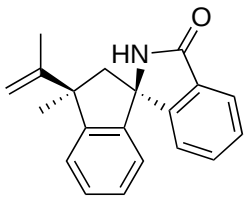
3aw



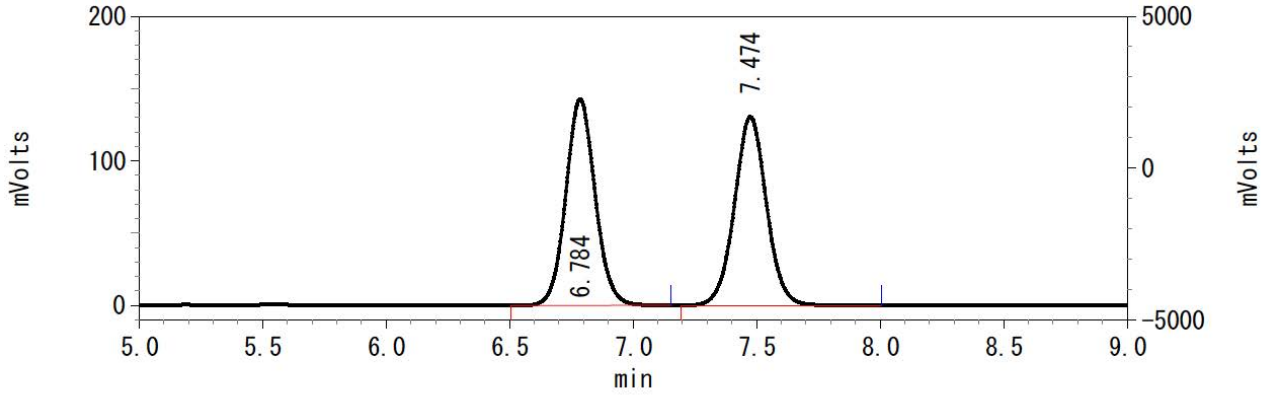
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-44-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	629
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



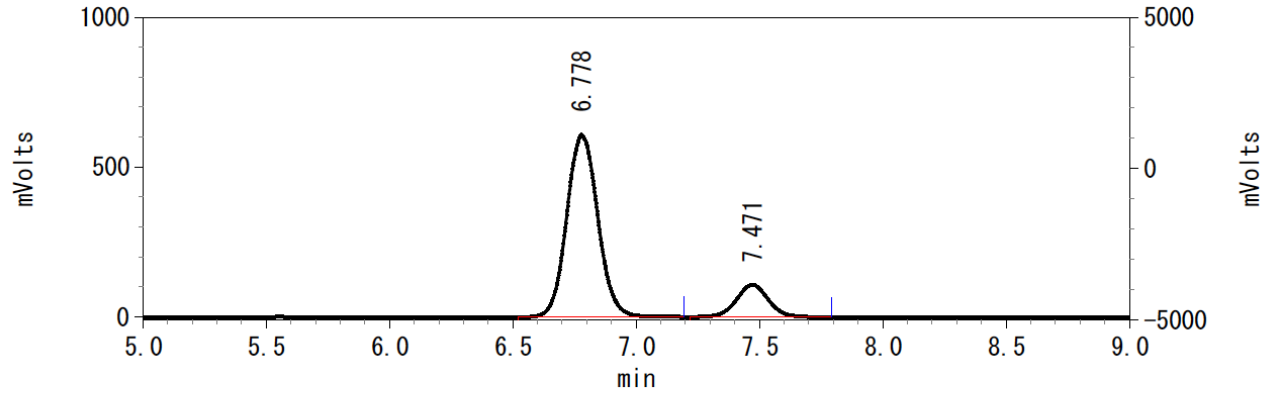
3aw



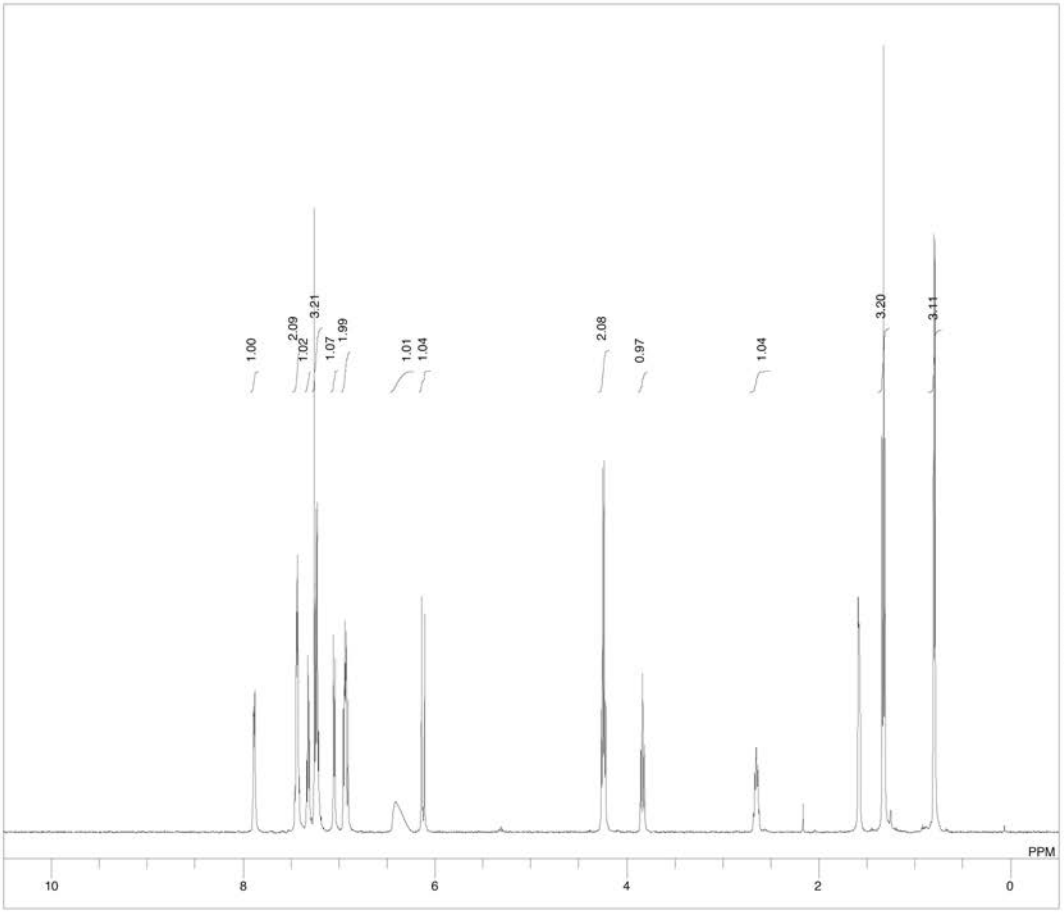
3aw



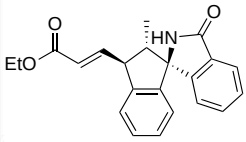
Pk #	Retention Time	Area	Area Percent
1	6.784	1183182	50.062
2	7.474	1180241	49.938



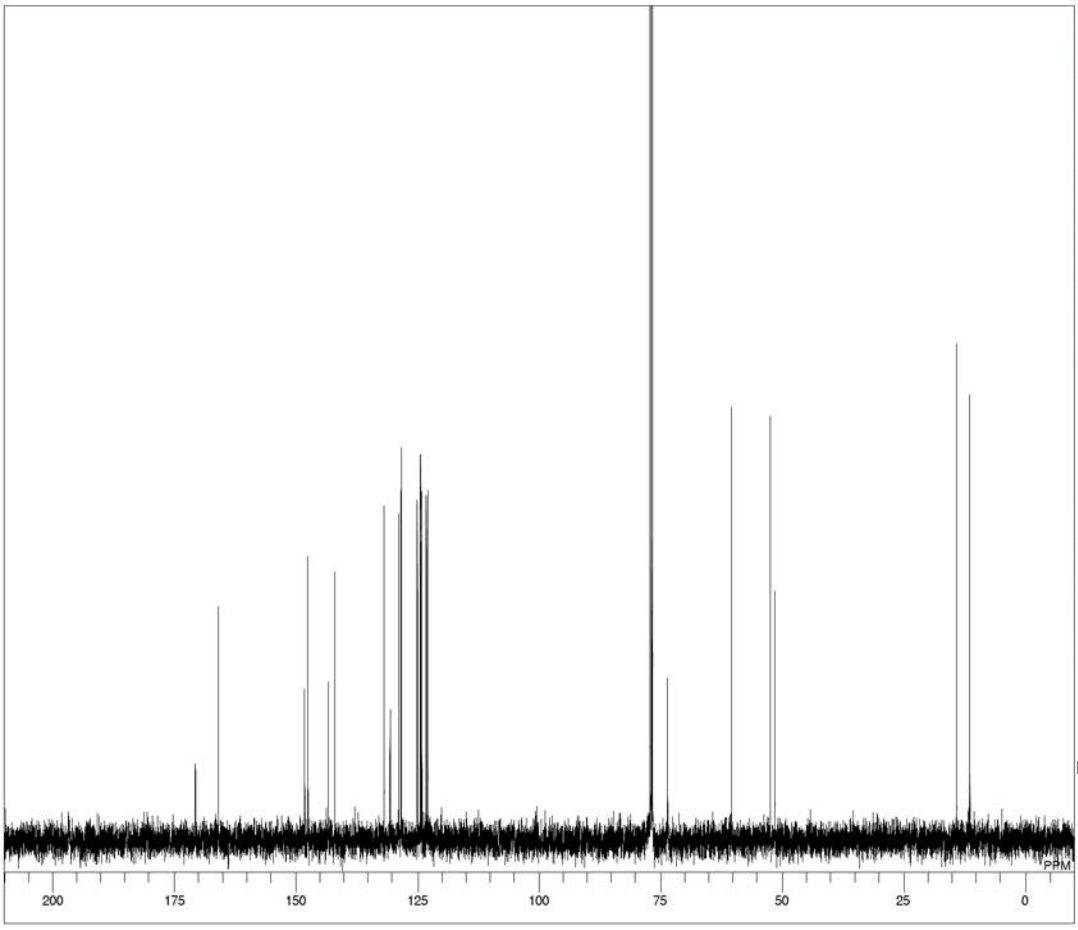
Pk #	Retention Time	Area	Area Percent
1	6.778	5331103	84.705
2	7.471	962609	15.295



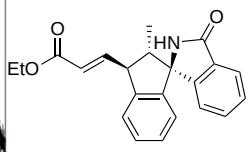
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	19
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-116-2.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1045
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



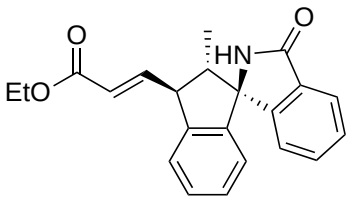
3ax



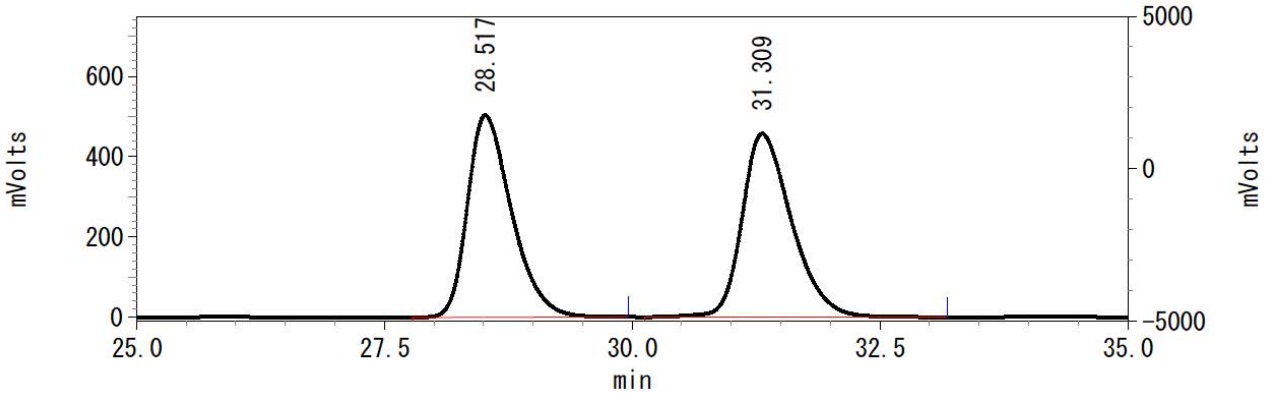
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	1.20 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-116-2-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	688
CSPED	11 Hz
FILDC	
FILDF	
SLVNT	CDCL3



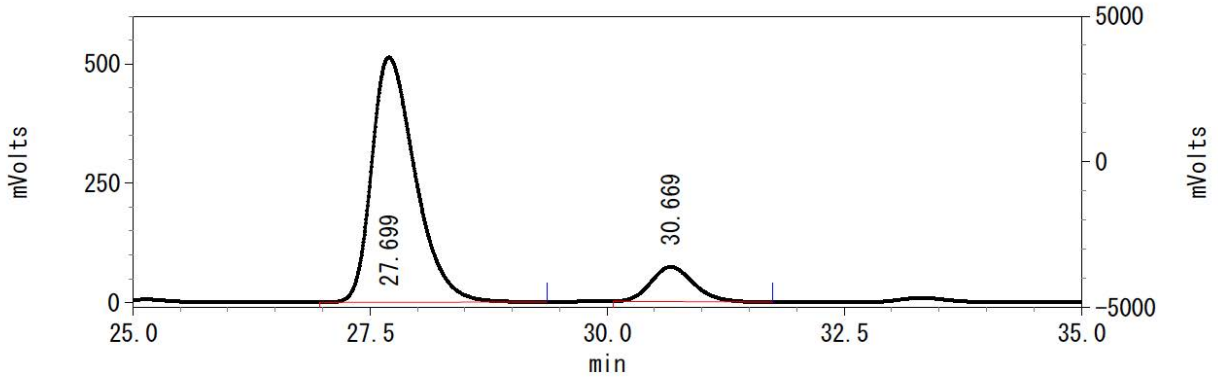
3ax



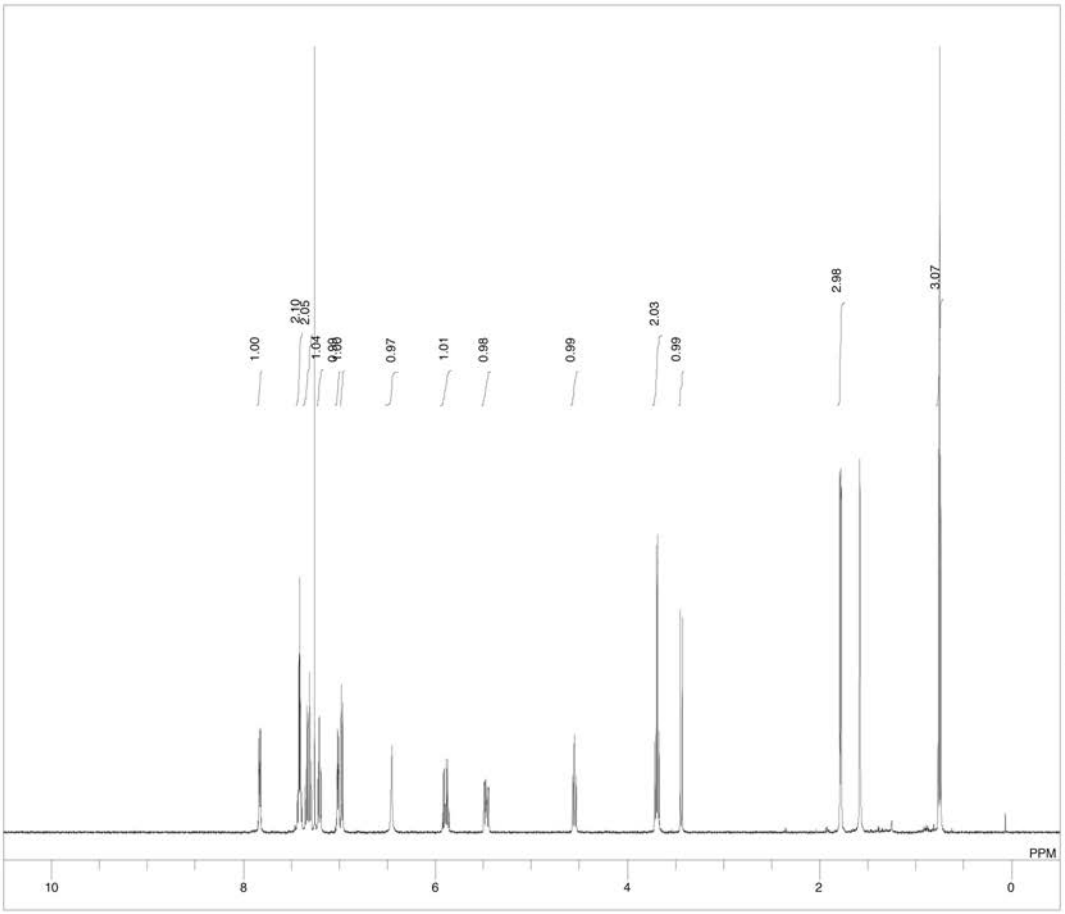
3ax



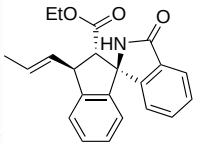
Pk #	Retention Time	Area	Area Percent
1	28.517	15631373	49.490
2	31.309	15953448	50.510



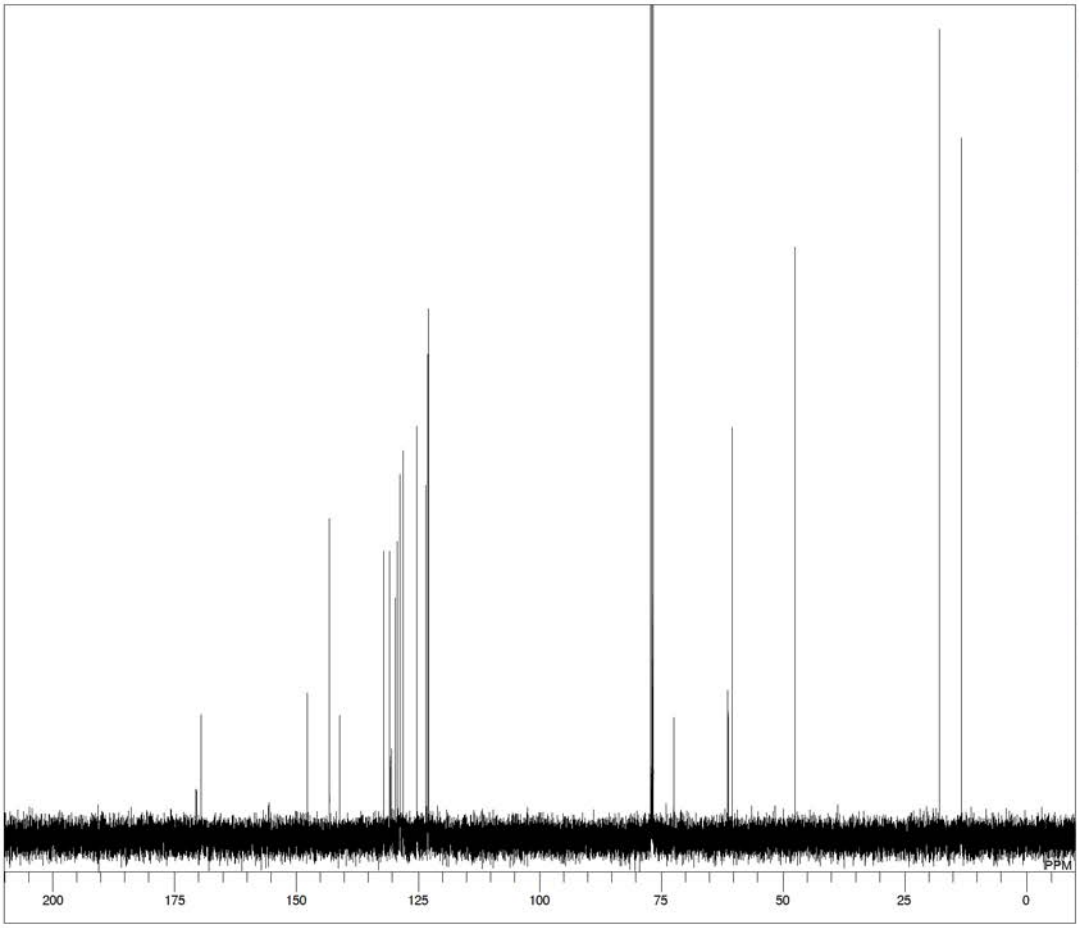
Pk #	Retention Time	Area	Area Percent
1	27.699	16385379	87.818
2	30.669	2272970	12.182



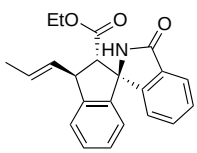
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	19
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-116-1.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1072
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



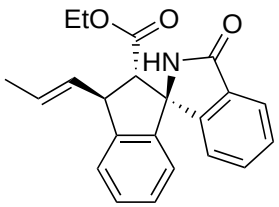
4ax



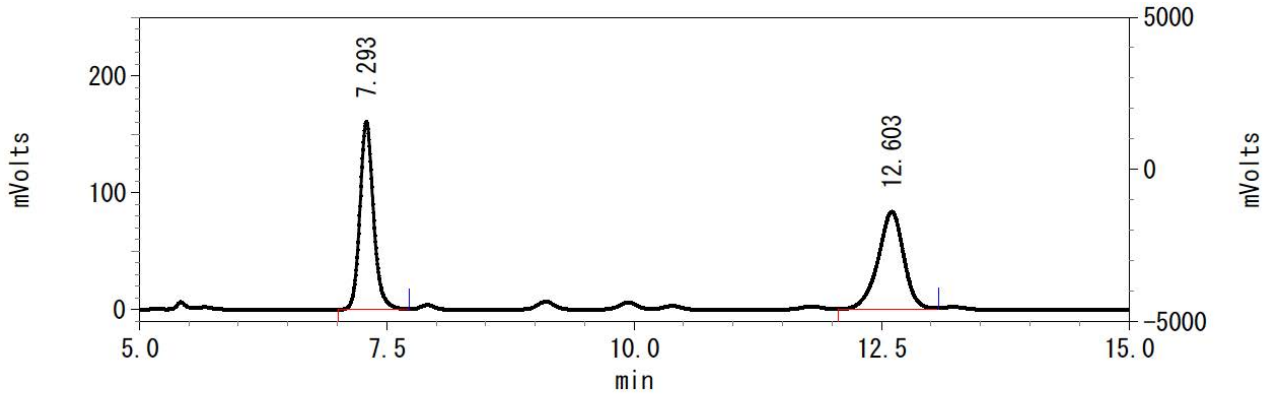
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-116-1-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	710
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



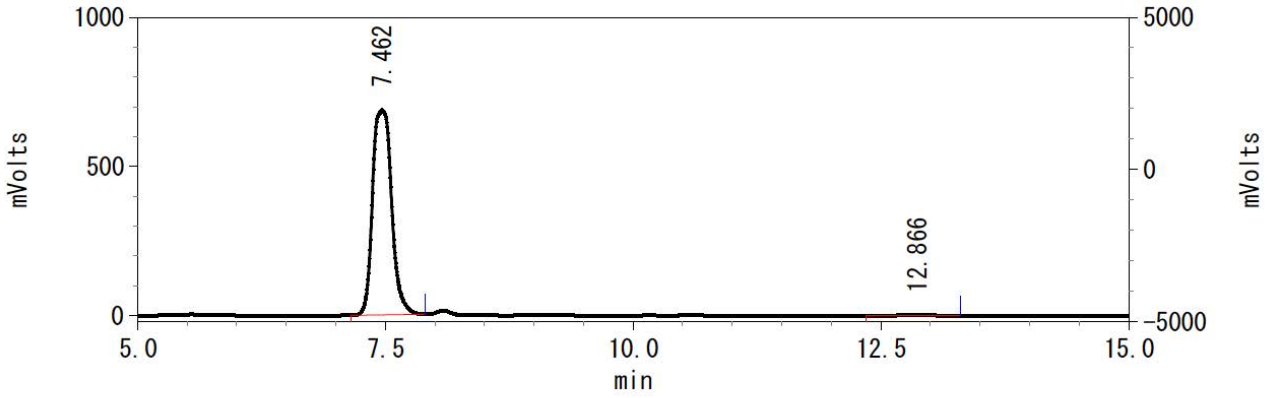
4ax



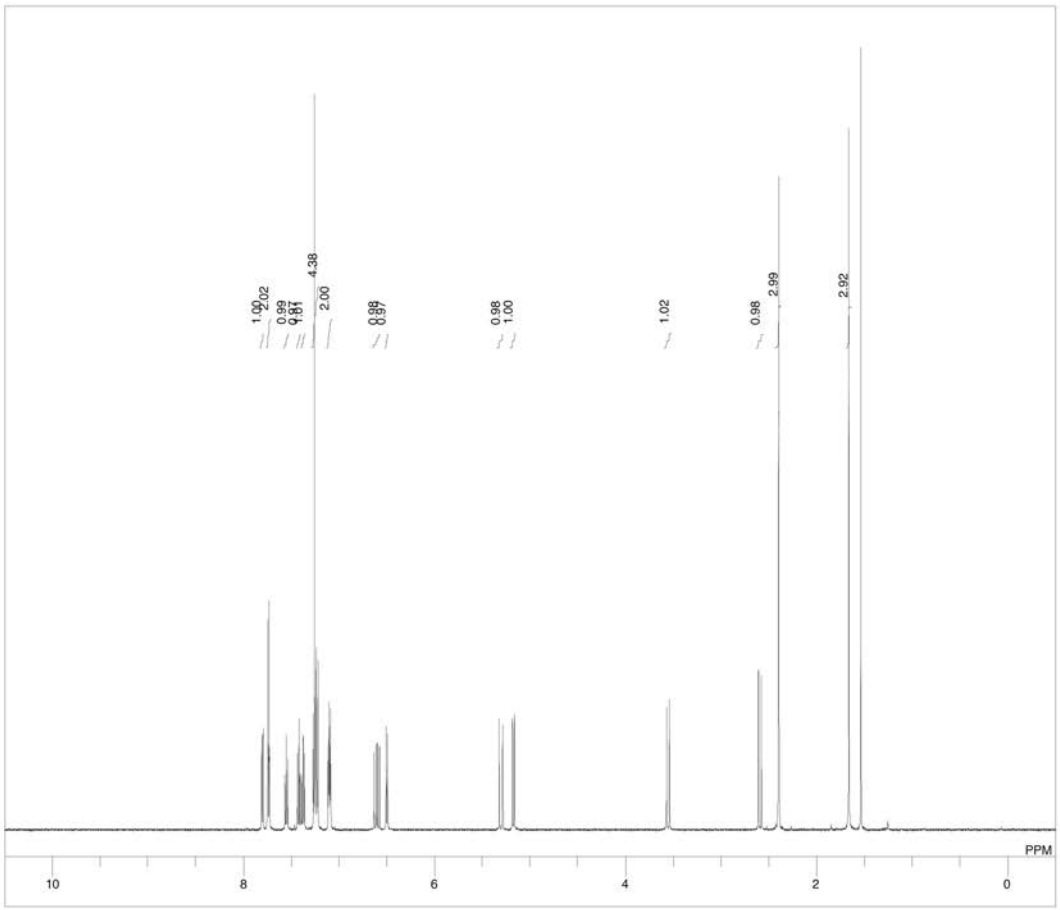
4ax



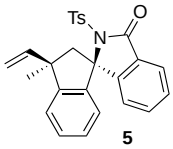
Pk #	Retention Time	Area	Area Percent
1	7.293	1533230	50.352
2	12.603	1511773	49.648



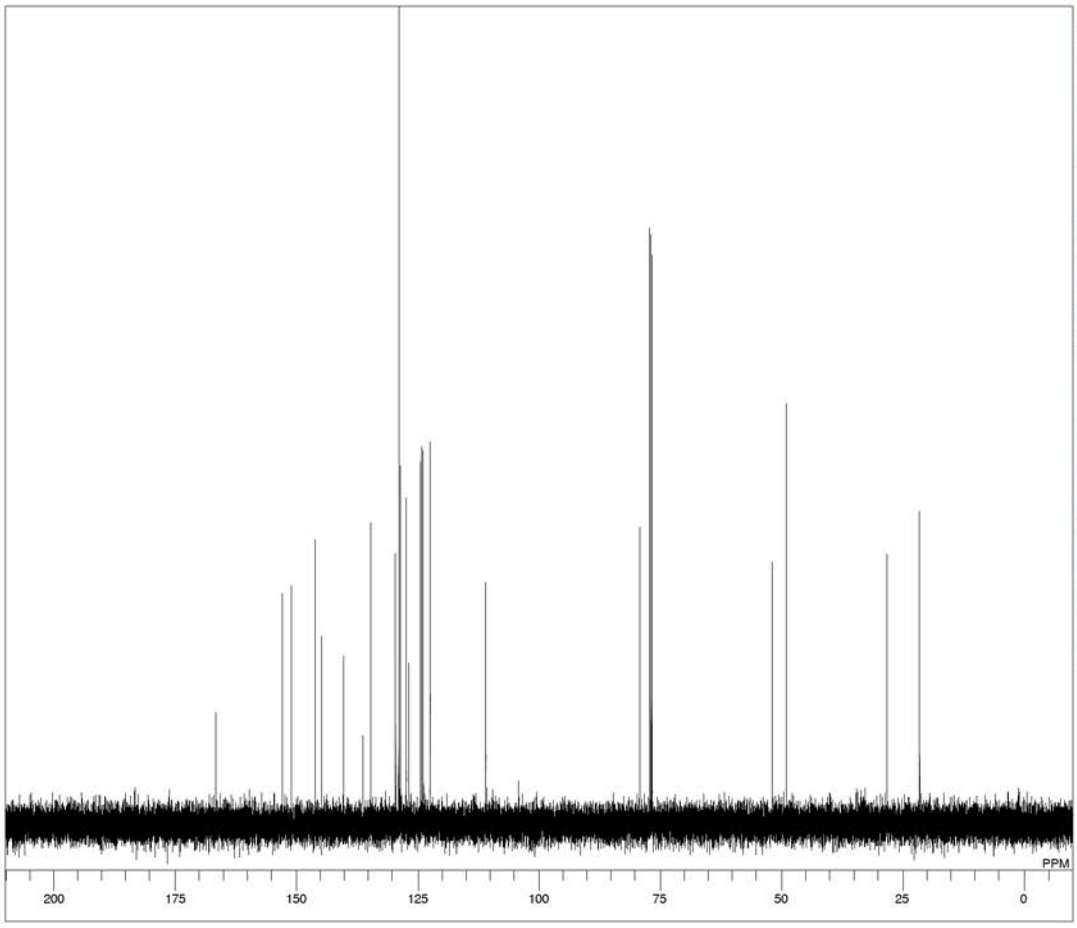
Pk #	Retention Time	Area	Area Percent
1	7.462	9177448	99.396
2	12.866	55751	0.604



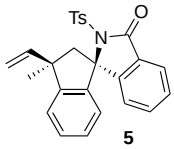
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	21
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-124.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	343
LKSIG	1047
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



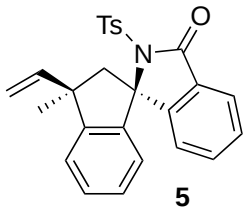
5



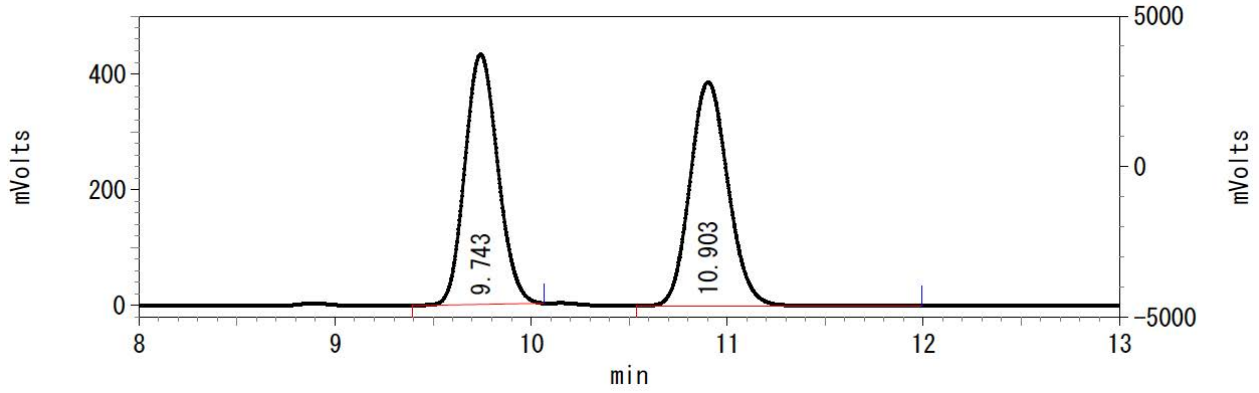
MENUF	13C
OBNUC	13C
OFR	125.40 MHz
OBSET	0.00 KHz
OBFIN	143041.00 Hz
PW1	6.25 usec
DEADT	10.00 usec
PREDL	0.01000 msec
IWT	1000.0000 sec
POINT	65536
SPO	65536
TIMES	720
DUMMY	2
FREQU	33898.30 Hz
FLT	16950 Hz
DELAY	11.80 usec
ACQTM	1.9333 sec
PD	1.0670 sec
ADBIT	16
RGAIN	24
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	bcm
EXPCM	single pulse with bilevel
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-2-124-carbon.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	22
LKPHS	343
LKSIG	691
CSPED	10 Hz
FILDC	
FILDF	
SLVNT	CDCL3



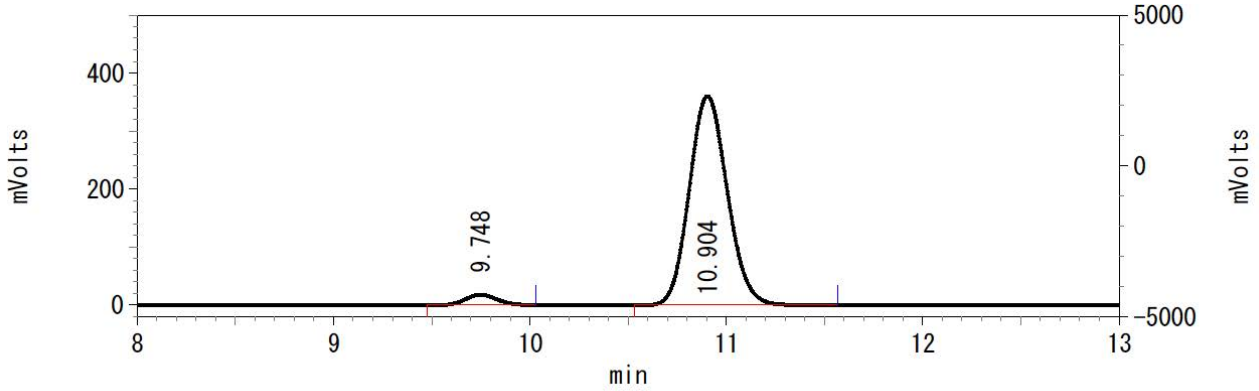
5



5



Pk #	Retention Time	Area	Area Percent
1	9.743	5046621	49.196
2	10.903	5211567	50.804



Pk #	Retention Time	Area	Area Percent
1	9.748	201053	3.980
2	10.904	4851103	96.020