Supplementary Information

C–H amidation of 2-aryl azlactones under iridium(III) catalysis: access to chiral amino acids

Min Seo Park,‡ Eunjae Chung,‡ Neeraj Kumar Mishra, Sang Hoon Han, Sangil Han, Saegun Kim* and In Su Kim*

School of Pharmacy, Sungkyunkwan University, Suwon 16419, Republic of Korea

* Corresponding authors. E-mail: ksk6696@skku.edu (S.Kim), insukim@skku.edu (I.S.Kim)

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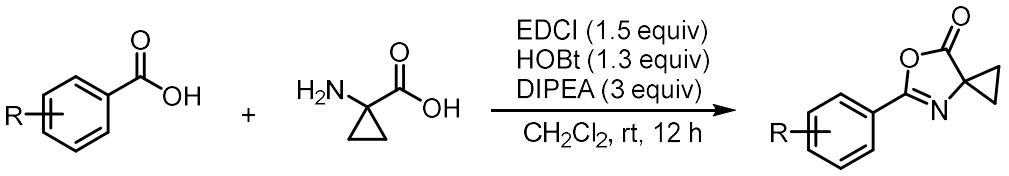
General methods

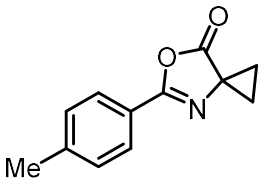
Commercially available reagents were used without additional purification, unless otherwise stated. Sealed tubes (13 × 100 mm^2) were purchased from Fischer Scientific and dried in oven for overnight and cooled at room temperature prior to use. Thin layer chromatography was carried out using plates coated with Kieselgel 60 F_{254} (Merck). For flash column chromatography, E. Merck Kieselgel 60 (230–400 mesh) was used. Nuclear magnetic resonance spectra (^1H and ^{13}C NMR) were recorded on a Bruker Unity 400 spectrometers and 700 spectrometers in $CDCl_3$ solution and chemical shifts are reported as parts per million (ppm). Resonance patterns are reported with the notations s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). Coupling constants (*J*) are reported in hertz (Hz). IR spectra were recorded on a JASCO FT/IR-4600 spectrophotometer and are reported as cm^{-1}. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-600 spectrometer. Enantiomeric excess (ee %) was determined by analytical liquid chromatography (HPLC) with DAICEL CHIRALPAK® AD-H (4.6 × 250 mm) in comparison with racemic samples.

General procedure and characterization data for the synthesis of 2-aryl azlactones (1a–1t and 5a–5e)



To an oven-dried sealed tube charged with benzoic acid (10 mmol), 1-aminocyclopropane-1-carboxylic acid (11 mmol, 1.1 equiv), HOBt (13 mmol, 1.3 equiv), EDCI (15 mmol, 1.5 equiv), and DIPEA (30 mmol, 3 equiv) was added in DCM (50 mL) under air. The reaction mixture was allowed to stir for 12 h at room temperature. The reaction mixture was diluted with DCM (50 mL) and poured into saturated NaCl solution. Extractive workup with DCM (2 x 50 mL). The combined organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography (*n*-hexanes/EtOAc = 30:1 to 15:1) to afford 2-phenyloxazol-5(4*H*)-ones (**1a–1t** and **5a–5e**).

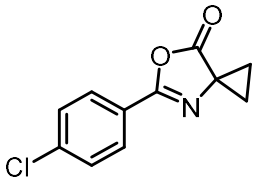
5-(*p*-Tolyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1a)



1a

1.61 g (80%); white solid; mp = 101.1–103.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 1.88–1.82 (m, 2H), 1.81–1.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.8, 162.0, 143.1, 129.7, 127.3, 123.4, 48.3, 21.8, 19.7; IR (KBr) υ 3093, 2923, 1808, 1635, 1573, 1511, 1446, 1411, 1319, 1268, 1180, 1103, 1029, 1006, 971, 917, 863, 829 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{12}H_{11}NO_2$ $[M]^+$ 201.0790, found 201.0787.

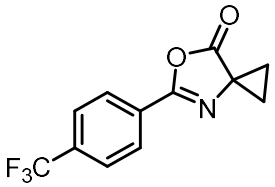
5-(4-Chlorophenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1b)



1b

1.32 g (60%); white solid; mp = 121.3–123.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 1.90–1.85 (m, 2H), 1.83–1.78 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 161.0, 138.7, 129.3, 128.69, 124.7, 48.5, 19.9; IR (KBr) υ 3104, 3085, 3062, 2927, 1801, 1731, 1631, 1596, 1565, 1488, 1403, 1311, 1268, 1087, 1033, 1002, 968, 914, 860, 840 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_8ClNO_2$ $[M]^+$ 221.0244, found 221.0240.

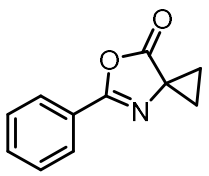
5-(4-(Trifluoromethyl)phenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1c)



1c

1.45 g (57%); white solid; mp = 110.5–112.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.8 Hz, 2H), 7.77 (d, *J* = 8.8 Hz, 2H), 1.97–1.92 (m, 2H), 1.90–1.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 160.7, 133.9 (q, $J_{C\text{-}F}$ = 32.5 Hz), 129.5, 127.7, 125.5 (q, $J_{C\text{-}F}$ = 4.0 Hz), 124.7 (q, $J_{C\text{-}F}$ = 270.9 Hz), 48.7, 20.2; IR (KBr) υ 2927, 2854, 1808, 1635, 1577, 1515, 1438, 1411, 1322, 1272, 1160, 1122, 1099, 1068, 1006, 971, 914, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{12}H_8F_3NO_2$ $[M]^+$ 255.0507, found 255.0509.

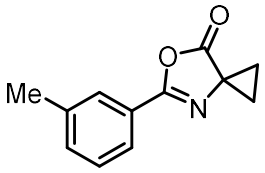
5-Phenyl-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1d)



1d

599.0 mg (32%); white solid; mp = 152.0–154.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.2 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 7.2 Hz, 2H), 1.90–1.84 (m, 2H), 1.83–1.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 161.9, 132.5, 128.9, 127.4, 126.3, 48.4, 19.8; IR (KBr) υ 2923, 2854, 1789, 1631, 1577, 1492, 1450, 1322, 1268, 1180, 1103, 1033, 1006, 971, 921, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_9NO_2$ $[M]^+$ 187.0633, found 187.0634.

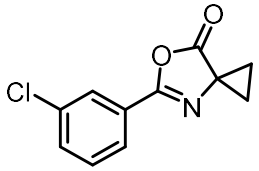
5-(*m*-Tolyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1e)



1e

1.25 g (62%); white solid; mp = 110.9–112.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.77–7.75 (m, 1H), 7.37–7.36 (m, 2H), 2.41 (s, 3H), 1.90–1.84 (m, 2H), 1.83–1.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 162.1, 138.8, 133.3, 128.8, 127.8, 126.1, 124.6, 48.3, 21.4, 19.7; IR (KBr) υ 3008, 2927, 2854, 1793, 1724, 1627, 1577, 1481, 1457, 1430, 1303, 1276, 1199, 1110, 1037, 971, 921, 887 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{12}H_{11}NO_2$ $[M]^+$ 201.0790, found 201.0788.

5-(3-Chlorophenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1f)

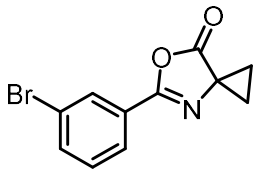


1f

1.06 g (48%); white solid; mp = 113.8–116.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 1.92–1.87 (m, 2H), 1.85–1.80 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 160.7, 135.2, 132.5, 130.3, 127.9, 127.4, 125.4, 48.5, 20.1; IR (KBr) υ 2927, 2854, 1793, 1724, 1627, 1573, 1481, 1461, 1427, 1303, 1276,

1199, 1110, 1033, 971, 921, 890 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_8ClNO_2$ $[M]^+$ 221.0244, found 221.0240.

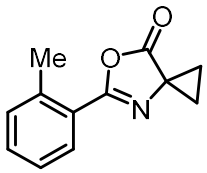
5-(3-Bromophenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1g)



1g

1.45 g (43%); white solid; mp = 111.4–114.1 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.13 (s, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.36 (t, *J* = 8.0 Hz, 1H), 1.92–1.86 (m, 2H), 1.85–1.790 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 160.6, 135.4, 130.5, 130.3, 128.1, 125.9, 123.0, 48.5, 20.1; IR (KBr) υ 2927, 2854, 1808, 1635, 1562, 1473, 1423, 1322, 1303, 1272, 1195, 1168, 1114, 1025, 971, 917, 871 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_8BrNO_2$ $[M]^+$ 264.9738, found 264.9735.

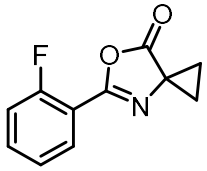
5-(*o*-Tolyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1h)



1h

1.33 g (63%); white solid; mp = 116.6–118.1 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.31–7.28 (m, 2H), 2.62 (s, 3H), 1.90–1.84 (m, 2H), 1.83–1.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.8, 162.0, 139.3, 131.9, 131.6, 129.5, 126.1, 125.1, 48.5, 22.1, 19.9; IR (KBr) υ 3097, 3023, 2985, 2927, 1789, 1623, 1569, 1488, 1457, 1427, 1322, 1261, 1095, 998, 979, 925, 860 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{12}H_{11}NO_2$ $[M]^+$ 201.0790, found 201.0790.

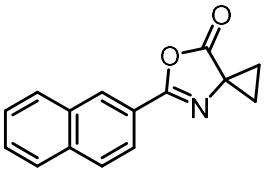
5-(2-Fluorophenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1i)



1i

656.6 mg (32%); white solid; mp = 134.2–135.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (td, *J* = 7.6, 2.0 Hz, 1H), 7.55–7.49 (m, 1H), 7.27–7.18 (m, 2H), 1.96–1.89 (m, 2H), 1.87–1.79 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 161.1 (d, $J_{C\text{-}F}$ = 258.8 Hz), 158.5 (d, $J_{C\text{-}F}$ = 5.8 Hz), 134.0 (d, $J_{C\text{-}F}$ = 8.7 Hz), 130.2, 124.5 (d, $J_{C\text{-}F}$ = 3.6 Hz), 117.1 (d, $J_{C\text{-}F}$ = 21.1 Hz), 114.7 (d, $J_{C\text{-}F}$ = 9.8 Hz), 48.4, 20.2; IR (KBr) υ 3100, 3008, 2927, 1793, 1631, 1577, 1492, 1454, 1423, 1319, 1272, 1230, 1095, 1006, 975, 925, 863, 821 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_8FNO_2$ $[M]^+$ 205.0539, found 205.0538.

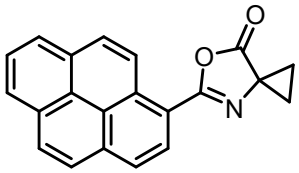
5-(Naphthalen-2-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1j)



1j

1.35 g (57%); white solid; mp = 160.1–161.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.02 (d, *J* = 8.4 Hz, 1H), 7.95–7.87 (m, 3H), 7.61–7.54 (m, 2H), 1.94–1.88 (m, 2H), 1.87–1.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 162.0, 135.2, 132.8, 129.2, 128.9, 128.5, 128.3, 128.0, 127.1, 123.5, 123.1, 48.6, 19.9; IR (KBr) υ 3085, 3058, 3035, 3008, 1793, 1627, 1592, 1508, 1450, 1423, 1357, 1322, 1276, 1234, 1095, 1018, 968, 944, 921, 890, 829 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{15}H_{11}NO_2$ $[M]^+$ 237.0790, found 237.0785.

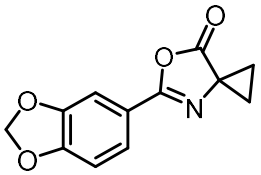
5-(Pyren-1-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1k)



1k

1.03 g (33%); yellow solid; mp = 241.7–243.3 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 9.43 (d, *J* = 9.8 Hz, 1H), 8.60 (d, *J* = 8.4 Hz, 1H), 8.28 (t, *J* = 8.4 Hz, 2H), 8.25–8.18 (m, 3H), 8.10–8.06 (m, 2H), 2.08–2.06 (m, 2H), 1.94–1.92 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 178.6, 162.3, 134.1, 131.2, 130.6, 130.3, 129.8, 129.0, 127.4, 127.3, 126.7, 126.6, 126.5, 125.0, 124.9, 124.6, 124.3, 118.8, 48.9, 20.2; IR (KBr) υ 3043, 2919, 2854, 1793, 1608, 1538, 1511, 1461, 1384, 1322, 1253, 1238, 1199, 1091, 1064, 998, 971, 917, 840 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{13}NO_2$ $[M]^+$ 311.0946, found 311.0941.

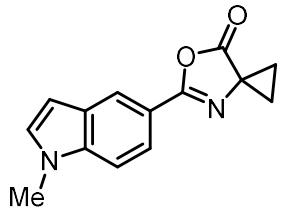
5-(Benzo[*d*][1,3]dioxol-5-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1l)



1l

0.95 g (41%); white solid; mp = 143.3–145.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 1H), 7.40 (s, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 6.05 (s, 2H), 1.85–1.80 (m, 2H), 1.78–1.73 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 161.3, 151.3, 148.3, 122.9, 120.2, 108.6, 107.2, 102.0, 48.3, 19.6; IR (KBr) υ 3104, 3012, 2904, 1801, 1639, 1492, 1450, 1365, 1326, 1299, 1265, 1230, 1087, 1037, 1014, 975, 906, 844, 817 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{12}H_9NO_4$ $[M]^+$ 231.0532, found 231.0530.

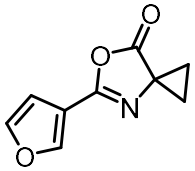
5-(1-Methyl-1*H*-indol-5-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1m)



1m

1.37 g (57%); white solid; mp = 173.3–174.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (s, 1H), 7.87 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.38 (d, *J* = 8.8 Hz, 1H), 7.13 (d, *J* = 3.2 Hz, 1H), 6.58 (d, *J* = 3.2 Hz, 1H), 3.83 (s, 3H), 1.88–1.82 (m, 2H), 1.81–1.74 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.2, 163.0, 138.7, 130.5, 128.3, 121.5, 120.6, 117.3, 109.7, 102.6, 48.2, 33.2, 19.4; IR (KBr) υ 3097, 3004, 2923, 1789, 1708, 1627, 1565, 1488, 1454, 1423, 1319, 1272, 1241, 1180, 1149, 1087, 1010, 975, 917, 890, 848, 802 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{14}H_{12}N_2O_2$ $[M]^+$ 240.0899, found 240.0899.

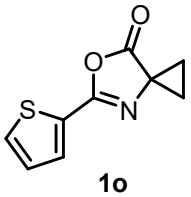
5-(Furan-3-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1n)



1n

545.4 mg (31%); white solid; mp = 120.1–121.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01–7.98 (m, 1H), 7.50 (t, *J* = 2.0 Hz, 1H), 6.80 (dd, *J* = 2.4, 0.8 Hz, 1H), 1.85–1.79 (m, 2H), 1.78–1.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 157.1, 145.3, 144.6, 114.9, 108.2, 47.7, 19.5; IR (KBr) υ 3151, 3116, 2927, 1797, 1654, 1511, 1400, 1319, 1272, 1160, 1130, 1052, 975, 937, 917, 871, 829 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{15}NO_2$ $[M]^+$ 177.0426, found 177.0426.

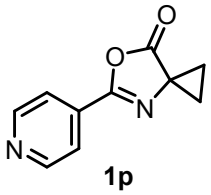
5-(Thiophen-2-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1o)



1o

0.48 g (25%); white solid; mp = 99.6–101.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.56 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.15 (dd, *J* = 5.2, 4.0 Hz, 1H), 1.89–1.83 (m, 2H), 1.82–1.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 157.8, 131.1, 131.0, 128.9, 128.1, 48.2, 19.7; IR (KBr) υ 3120, 3077, 1797, 1623, 1515, 1419, 1365, 1303, 1272, 1226, 1091, 1045, 964, 914, 848 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_9H_7NO_2S$ $[M]^+$ 193.0197, found 193.0198.

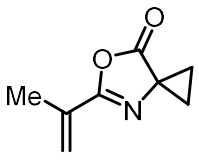
5-(Pyridin-4-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1p)



1p

0.54 g (28%); white solid; mp = 190.5–192.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J* = 4.8 Hz, 2H), 7.82 (d, *J* = 6.0 Hz, 2H), 1.99–1.93 (m, 2H), 1.91–1.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 160.2, 150.6, 133.7, 120.8, 48.9, 20.6; IR (KBr) υ 3097, 1801, 1646, 1592, 1558, 1538, 1415, 1400, 1330, 1229, 1272, 1110, 1087, 1018, 971, 921, 867, 836 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{10}H_8N_2O_2$ $[M]^+$ 188.0586, found 188.0585.

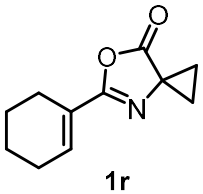
5-(Prop-1-en-2-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1q)



1q

0.44 g (30%); white solid; mp = 86.6–89.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.97–5.96 (m, 1H), 5.63–5.62 (m, 1H), 2.03 (s, 3H), 1.83–1.77 (m, 2H), 1.76–1.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 162.7, 131.3, 123.5, 48.5, 19.7, 17.8; IR (KBr) υ 3093, 3008, 2935, 1797, 1735, 1643, 1592, 1454, 1342, 1319, 1276, 1157, 1060, 968, 929, 867 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_8H_9NO_2$ $[M]^+$ 151.0633, found 151.0634.

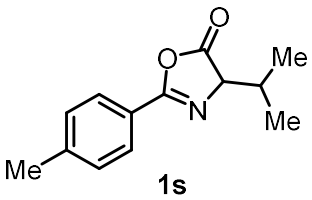
5-(Cyclohex-1-en-1-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (1r)



1r

0.54 g (28%); white solid; mp = 57.2–58.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81–6.78 (m, 1H), 2.33–2.29 (m, 2H), 2.27–2.22 (m, 2H), 1.77–1.74 (m, 2H), 1.72–1.62 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.7, 162.6, 137.5, 125.6, 48.0, 25.8, 23.6, 21.8, 21.6, 19.3; IR (KBr) υ 3097, 3012, 2935, 2681, 1805, 1650, 1604, 1438, 1388, 1322, 1272, 1095, 1076, 998, 975, 921, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_{13}NO_2$ $[M]^+$ 191.0946, found 191.0945.

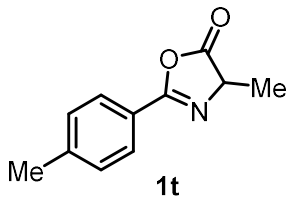
4-Isopropyl-2-(*p*-tolyl)oxazol-5(4*H*)-one (1s)



1s

1.02 g (47%); white solid; mp = 51.1–53.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.30 (d, *J* = 4.4 Hz, 1H), 2.45 (s, 3H), 2.42–2.36 (m, 1H), 1.16 (d, *J* = 6.8 Hz, 3H), 1.04 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 161.9, 143.6, 129.6, 128.0, 123.2, 70.7, 31.4, 21.8, 18.8, 17.6; IR (KBr) υ 2965, 21927, 1877, 1820, 1650, 1616, 1511, 1461, 1411, 1322, 1295, 1245, 1180, 1145, 1041, 1014, 944, 883, 829 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{13}H_{15}NO_2$ $[M]^+$ 217.1103, found 217.1103.

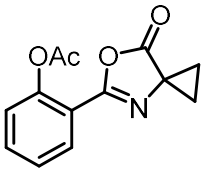
4-Methyl-2-(*p*-tolyl)oxazol-5(4*H*)-one (1t)



1t

0.96 g (51%); white solid; mp = 77.8–80.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.43 (d, *J* = 7.6 Hz, 1H), 2.42 (s, 3H), 1.58 (d, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.3, 161.7, 143.6, 129.6, 127.9, 123.2, 61.1, 21.8, 17.1; IR (KBr) υ 3035, 2985, 2935, 1816, 1754, 1650, 1612, 1511, 1446, 1411, 1315, 1257, 1157, 1106, 1045, 995, 910, 875, 825 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{11}H_{11}NO_2$ $[M]^+$ 189.0790, found 189.0787.

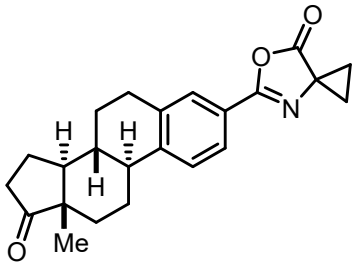
2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl acetate (5a)



5a

1.35 g (55%); white solid; mp = 96.8–97.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.56 (td, *J* = 8.0, 1.6 Hz, 1H), 7.37 (td, *J* = 8.0, 1.6 Hz, 1H), 7.16 (dd, *J* = 8.0, 1.2 Hz, 1H), 2.29 (s, 3H), 1.86–1.83 (m, 2H), 1.81–1.78 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 169.7, 159.0, 149.6, 133.4, 130.1, 126.5, 124.2, 119.4, 48.3, 21.2, 20.1; IR (KBr) υ 3016, 2935, 1808, 1766, 1635, 1492, 1450, 1369, 1315, 1268, 1191, 1087, 1006, 975, 914, 863, 821 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{13}H_{11}NO_4$ $[M]^+$ 245.0688, found 245.0687.

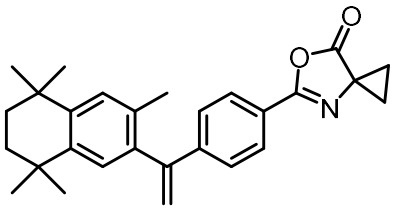
5-((8*R*,9*S*,13*S*,14*S*)-13-Methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta [*a*]phenanthren-3-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (5b)



5b

2.21 g (61%); white solid; mp = 258.9–261.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 2.98–2.94 (m, 2H), 2.55–2.33 (m, 3H), 2.20–1.98 (m, 4H), 1.88–1.75 (m, 4H), 1.65–1.49 (m, 6H), 0.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.5, 178.7, 162.8, 144.8, 137.4, 127.8, 126.0, 124.7, 123.7, 50.6, 48.3, 48.0, 44.8, 37.9, 35.9, 31.6, 29.3, 26.3, 25.7, 21.7, 19.7, 13.9; IR (KBr) υ 2931, 2873, 1805, 1731, 1631, 1604, 1569, 1496, 1415, 1373, 1330, 1311, 1265, 1218, 1172, 1087, 1037, 1010, 971, 887, 848 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{23}H_{25}NO_3$ $[M]^+$ 363.1834, found 363.1834.

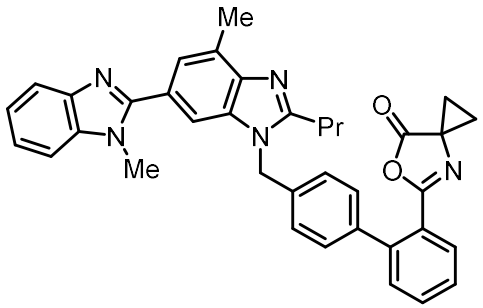
5-(4-(1-(3,5,5,8,8-Pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)vinyl)phenyl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (5c)



5c

2.32 g (56%); white solid; mp = 148.1–149.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 7.13 (s, 1H), 7.08 (s, 1H), 5.82 (s, 1H), 5.32 (s, 1H), 1.95 (s, 3H), 1.90–1.84 (m, 2H), 1.83–1.76 (m, 2H), 1.70 (s, 4H), 1.30 (s, 6H), 1.27 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.7, 161.8, 149.1, 145.2, 144.5, 142.5, 138.0, 132.8, 128.3, 128.2, 127.4, 127.1, 125.0, 117.0, 48.4, 35.3, 35.3, 34.1, 34.0, 32.1, 32.0, 20.1, 19.8; IR (KBr) υ 2958, 2923, 2861, 1808, 1743, 1635, 1500, 1457, 1407, 1365, 1319, 1268, 1234, 1091, 1029, 1002, 971, 910, 860 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{28}H_{31}NO_2$ $[M]^+$ 413.2355, found 413.2351.

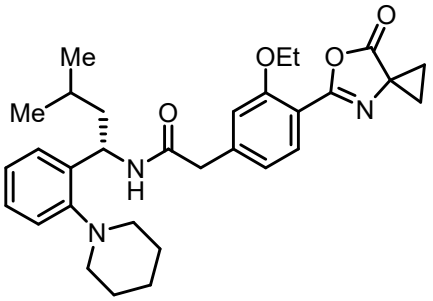
5-(4'-((1,7'-Dimethyl-2'-propyl-1*H*,3'*H*-[2,5'-bibenzo[d]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-yl)-6-oxa-4-azaspiro[2.4]hept-4-en-7-one (5d)



5d

2.49 mg (43%); white solid; mp = 95.9–98.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84–7.81 (m, 2H), 7.55–7.51 (m, 2H), 7.45–7.41 (m, 2H), 7.38–7.29 (m, 4H), 7.27–7.25 (m, 2H), 7.08 (d, *J* = 8.0 Hz, 2H), 5.46 (s, 2H), 3.83 (s, 3H), 2.94 (t, *J* = 8.0 Hz, 2H), 2.77 (s, 3H), 1.92–1.83 (m, 2H), 1.61–1.48 (m, 4H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 162.5, 156.7, 154.5, 141.6, 140.4, 136.6, 135.2, 131.7, 131.2, 131.1, 130.0, 129.7, 129.6, 129.4, 127.8, 126.1, 125.6, 123.9, 122.8, 122.7, 119.4, 109.7, 109.3, 47.9, 47.2, 32.0, 30.0, 22.0, 19.3, 17.0, 14.2; IR (KBr) υ 3043, 2962, 2873, 1805, 1739, 1639, 1511, 1450, 1407, 1319, 1272, 1157, 1091, 1056, 1002, 971, 914, 860 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{37}H_{33}N_5O_2$ $[M]^+$ 579.2634, found 579.2629.

(*S*)-2-(3-Ethoxy-4-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)-*N*-(3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)acetamide (5e)



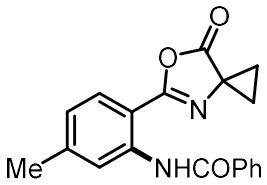
5e

2.12 g (41%); brown solid; mp = 149.7–150.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.0 Hz, 1H), 7.23–7.17 (m, 2H), 7.08–7.03 (m, 2H), 6.90–6.88 (m, 2H), 6.73 (d, *J* = 8.8 Hz, 1H), 5.37 (q, *J* = 8.4 Hz, 1H), 4.10–4.01 (m, 2H), 3.55 (s, 2H), 2.92 (s, 2H), 2.62 (s, 2H), 1.90–1.83 (m, 2H), 1.82–1.75 (m, 2H), 1.73–1.70 (m, 2H), 1.62–1.50 (m, 2H), 1.40 (t, *J* = 6.8 Hz, 4H), 0.91 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.0, 168.7, 160.6, 158.4, 152.6, 141.3, 138.7, 131.1, 128.1, 127.8, 125.2, 123.0, 121.3, 114.7, 114.0, 64.9, 50.0, 48.2, 46.8, 44.4, 26.9, 25.4, 24.2, 22.9, 22.6, 19.8, 14.6; IR (KBr) υ 3293, 2931, 2865, 2800, 1801, 1639, 1535, 1492, 1430, 1384, 1303, 1268, 1253, 1168, 1103, 1037, 1002, 975, 917, 860 cm^{-1}; HRMS (quadrupole, FAB) calcd for $C_{31}H_{40}N_3O_4$ $[M+H]^+$ 518.3019, found 518.3019.

General procedure and characterization data for the amidation of 2-aryl azlactones using acyl azides (3a–3t, 4b–4k, and 6a–6e)

To an oven-dried sealed tube charged with 2-aryl azlactones (0.2 mmol, 100 mol %), $[IrCp^*Cl_2]_2$ (4.0 mg, 0.005 mmol, 2.5 mol %), $AgNTf_2$ (7.8 mg, 0.02 mmol, 10 mol %), LiOAc (4.0 mg, 0.06 mmol, 30 mol %), and acyl azides (0.3 mmol, 150 mol %) was added DCE (1 mL) under air at room temperature. The reaction mixture was allowed to stir at 50 °C for 24 h. The reaction mixture was cooled to room temperature, diluted with EtOAc (3 mL) and concentrated in vacuo. The residue was purified by flash column chromatography (*n*-hexane:EtOAc) to afford the corresponding products **3a–3t**, **4b–4k**, and **6a–6e**.

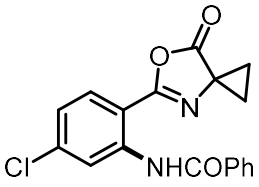
***N*-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3a)**



3a

54.6 mg (85%); white solid; mp = 140.7–141.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.92 (s, 1H), 8.82 (s, 1H), 7.93 (d, *J* = 7.2 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 1H), 2.45 (s, 3H), 1.86 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 166.0, 162.7, 145.2, 140.2, 135.2, 132.1, 128.8, 128.7, 127.5, 124.1, 120.8, 109.3, 47.6, 22.4, 20.0; IR (KBr) υ 3232, 3104, 2923, 1812, 1677, 1619, 1577, 1542, 1500, 1446, 1415, 1326, 1268, 1191, 1157, 1106, 1079, 1006, 979, 925, 890, 856, 821 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{16}N_2O_3$ $[M]^+$ 320.1161, found 320.1163.

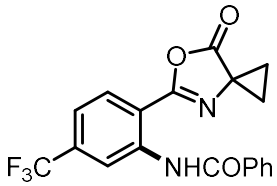
***N*-(5-Chloro-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3b)**



3b

62.8 mg (92%); white solid; mp = 215.2–216.8 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 11.98 (s, 1H), 9.06 (d, *J* = 2.1 Hz, 1H), 7.90 (d, *J* = 7.0 Hz, 2H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.55 (t, *J* = 7.0 Hz, 1H), 7.46 (t, *J* = 8.4 Hz, 2H), 7.14 (dd, *J* = 8.4, 2.1 Hz, 1H), 1.90–1.85 (m, 2H), 1.93–1.88 (m, 4H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 175.8, 166.0, 162.1, 141.0, 140.2, 134.7, 132.3, 129.8, 128.8, 127.5, 123.3, 120.4, 110.1, 47.7, 20.2; IR (KBr) υ 3178, 3116, 3085, 2923, 1816, 1677, 1616, 1577, 1531, 1496, 1411, 1322, 1276, 1245, 1103, 1076, 1006, 975, 921, 852 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{13}ClN_2O_3$ $[M]^+$ 340.0615, found 340.0615.

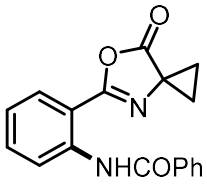
***N*-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)-5-(trifluoromethyl)phenyl)benzamide (3c)**



3c

60.7 mg (81%); white solid; mp = 185.4–187.7 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 12.04 (s, 1H), 9.35 (s, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 7.0 Hz, 2H), 7.56 (t, *J* = 7.0 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 7.7 Hz, 1H), 1.98–1.96 (m, 2H), 1.95–1.94 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 175.5, 166.1, 161.9, 140.6, 135.2 (q, $J_{C\text{-}F}$ = 3.9 Hz), 134.6, 132.5, 129.5, 128.8, 127.5, 123.4 (q, $J_{C\text{-}F}$ = 270.9 Hz), 119.4 (q, $J_{C\text{-}F}$ = 3.9 Hz), 117.4 (q, $J_{C\text{-}F}$ = 3.9 Hz), 114.2, 47.9, 20.5; IR (KBr) υ 3131, 3062, 2923, 2854, 1816, 1670, 1612, 1581, 1550, 1504, 1423, 1322, 1280, 1226, 1172, 1133, 1072, 1002, 917, 894, 833 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{13}F_3N_2O_3$ $[M]^+$ 374.0878, found 374.0879.

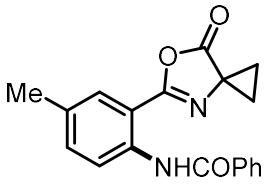
***N*-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3d)**



3d

47.8 mg (78%); white solid; mp = 165.9–167.1 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 11.98 (s, 1H), 8.99 (d, *J* = 8.4 Hz, 1H), 7.96–7.84 (m, 3H), 7.60 (t, *J* = 8.4 Hz, 1H), 7.56 (t, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 8.4 Hz, 2H), 7.20 (t, *J* = 7.7 Hz, 1H), 1.93–1.91 (m, 2H), 1.90–1.88 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 176.3, 166.1, 162.7, 140.3, 135.2, 134.0, 132.1, 129.0, 128.7, 127.5, 123.1, 120.4, 111.9, 47.8, 20.1; IR (KBr) υ 3255, 2923, 2854, 1816, 1677, 1616, 1585, 1546, 1500, 1450, 1299, 1268, 1168, 1106, 1083, 1006, 925, 871 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{14}N_2O_3$ $[M]^+$ 306.1004, found 306.1002.

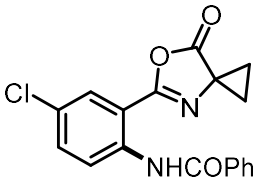
***N*-(4-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3e)**



3e

55.8 mg (87%); white solid; mp = 206.5–207.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.65 (s, 1H), 8.66 (d, *J* = 8.8 Hz, 1H), 7.74–7.71 (m, 2H), 7.52 (s, 1H), 7.35–7.31 (m, 1H), 7.26 (t, *J* = 7.6 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 1H), 2.18 (s, 3H), 1.70–1.68 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 165.7, 162.6, 137.9, 135.2, 134.6, 132.7, 131.9, 129.0, 128.6, 127.4, 120.3, 111.6, 47.7, 20.8, 20.1; IR (KBr) υ 3112, 2919, 2854, 1808, 1731, 1677, 1612, 1538, 1500, 1454, 1407, 1303, 1268, 1203, 1079, 1033, 975, 917, 840 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{16}N_2O_3$ $[M]^+$ 320.1161, found 320.1160.

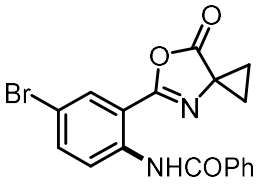
***N*-(4-Chloro-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3f)**



3f

53.2 mg (78%); white solid; mp = 205.8–206.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.89 (s, 1H), 8.96 (d, *J* = 8.8 Hz, 1H), 7.92–7.90 (m, 3H), 7.58–7.45 (m, 4H), 1.96–1.89 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.7, 165.9, 161.7, 138.8, 134.8, 133.7, 132.3, 128.8, 128.5, 128.2, 127.5, 121.8, 113.0, 47.9, 20.4; IR (KBr) υ 3170, 3106, 3023, 2911, 1809, 1657, 1601, 1595, 1566, 1480, 1401, 1392, 1302, 1216, 1183, 1176, 1096, 978, 925, 849 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{13}ClN_2O_3$ $[M]^+$ 340.0615, found 340.0612.

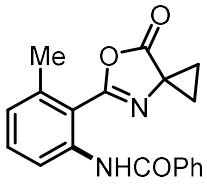
***N*-(4-Bromo-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3g)**



3g

54.8 mg (71%); white solid; mp = 242.3–243.8 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 11.89 (s, 1H), 8.91 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 2.1 Hz, 1H), 7.91 (d, *J* = 7.0 Hz, 2H), 7.67 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.56 (t, *J* = 7.0 Hz, 1H), 7.47 (t, *J* = 8.4 Hz, 2H), 1.95–1.90 (m, 4H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 175.6, 166.0, 161.7, 139.3, 136.6, 134.9, 132.3, 131.4, 128.8, 127.5, 122.0, 115.5, 113.4, 47.9, 20.3; IR (KBr) υ 3112, 2923, 2854, 1816, 1727, 1681, 1612, 1573, 1527, 1496, 1388, 1295, 1257, 1099, 1076, 1014, 975, 921, 879, 840 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{13}BrN_2O_3$ $[M]^+$ 384.0110, found 384.0108.

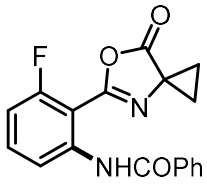
***N*-(3-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3h)**



3h

58.4 mg (91%); white solid; mp = 164.4–165.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.52 (s, 1H), 8.62 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 7.2 Hz, 2H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.44–7.37 (m, 3H), 6.98 (d, *J* = 7.2 Hz, 1H) 2.56 (s, 3H), 1.84 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 165.6, 163.1, 139.7, 139.6, 135.3, 132.6, 132.0, 128.7, 127.3, 126.9, 118.9, 113.3, 46.8, 23.2, 20.1; IR (KBr) υ 3070, 2981, 2931, 1812, 1724, 1670, 1600, 1550, 1492, 1454, 1326, 1292, 1253, 1184, 1114, 1091, 1033, 1002, 983, 921, 867 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{16}N_2O_3$ $[M]^+$ 320.1161, found 320.1161.

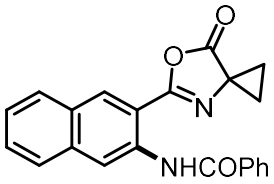
***N*-(3-Fluoro-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (3i)**



3i

41.6 mg (64%); white solid; mp = 176.0–178.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.96 (s, 1H), 8.75 (d, *J* = 8.8 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 2H), 7.57–7.46 (m, 4H), 6.95–6.91 (m, 1H), 1.93–1.90 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.3, 166.0, 161.6 (d, $J_{C\text{-}F}$ = 258.1 Hz), 160.9, 141.1, 135.0, 134.6 (d, $J_{C\text{-}F}$ = 10.5 Hz), 132.3, 128.8, 127.5, 116.3 (d, $J_{C\text{-}F}$ = 3.6 Hz), 111.1 (d, $J_{C\text{-}F}$ = 22.1 Hz), 102.2, 46.6, 20.2; IR (KBr) υ 3247, 3097, 2927, 1812, 1731, 1677, 1608, 1581, 1550, 1465, 1299, 1268, 1184, 1110, 1076, 1006, 979, 925, 867 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{13}FN_2O_3$ $[M]^+$ 324.0910, found 324.0906.

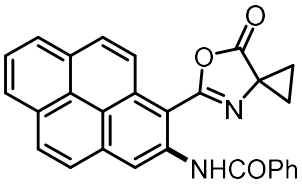
***N*-(3-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)naphthalen-2-yl)benzamide (3j)**



3j

56.3 mg (79%); white solid; mp = 210.6–211.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.9 (s, 1H), 9.43 (s, 1H), 7.98 (d, *J* = 7.6 Hz, 2H), 7.88 (dd, *J* = 13.6, 8.4 Hz, 2H), 7.62–7.54 (m, 2H), 7.51–7.44 (m, 3H), 1.94 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.1, 166.0, 162.6, 136.1, 135.4, 135.2, 132.0, 131.2, 129.4, 128.9, 128.7, 128.7, 128.0, 127.4, 126.0, 117.7, 112.5, 48.0, 20.2; IR (KBr) υ 3262, 3104, 3058, 2923, 1816, 1677, 1631, 1604, 1550, 1484, 1442, 1361, 1334, 1307, 1272, 1076, 991, 921, 894, 752 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{22}H_{16}N_2O_3$ $[M]^+$ 356.1161, found 356.1157.

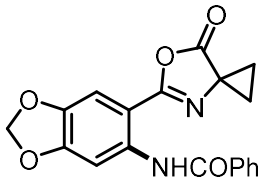
_N_-(1-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)pyren-2-yl)benzamide (3k)



3k

44.0 mg (51%); yellow solid; mp = 181.4–182.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.77 (s, 1H), 9.56 (s, 1H), 8.62 (d, *J* = 9.6 Hz, 1H), 8.20–7.96 (m, 8H), 7.61–7.51 (m, 3H), 2.00–1.98 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 165.8, 163.2, 137.0, 135.4, 134.8, 132.3, 132.2, 131.0, 130.7, 130.5, 129.7, 128.8, 127.6, 127.4, 126.9, 126.6, 126.2, 124.8, 123.9, 121.4, 117.4, 107.7, 47.3, 20.4; IR (KBr) υ 3050, 2923, 2854, 1816, 1724, 1681, 1596, 1558, 1469, 1376, 1330, 1299, 1268, 1184, 1095, 1072, 1002, 921, 879, 829 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{28}H_{18}N_2O_3$ $[M]^+$ 430.1317, found 430.1314.

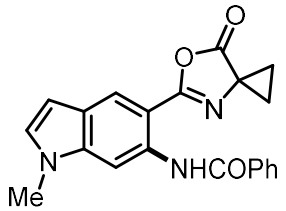
_N_-(6-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)benzo[*d*][1,3]dioxol-5-yl)benzamide (3l)



3l

53.3 mg (76%); white solid; mp = 174.1–176.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.76 (s, 1H), 7.95 (d, *J* = 7.6 Hz, 2H), 7.56–7.44 (m, 4H), 6.76 (d, *J* = 8.4 Hz, 1H), 6.14 (s, 2H), 1.79–1.70 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 164.0, 161.7, 152.5, 141.1, 133.6, 132.3, 128.7, 127.7, 124.4, 121.5, 110.7, 105.1, 102.4, 47.7, 19.8; IR (KBr) υ 3282, 3062, 2904, 1812, 1689, 1635, 1600, 1527, 1465, 1303, 1268, 1160, 1114, 1049, 1025, 979, 925, 875 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{14}N_2O_5$ $[M]^+$ 350.0903, found 350.0901.

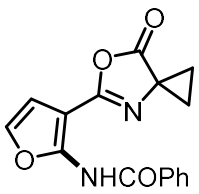
_N_-(1-Methyl-5-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)-1_H_-indol-6-yl)benzamide (3m)



3m

36.7 mg (51%); white solid; mp = 233.5–235.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.21 (s, 1H), 9.04 (s, 1H), 8.24 (s, 1H), 7.99 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.09 (d, *J* = 3.0 Hz, 1H), 6.54 (d, *J* = 3.0 Hz, 1H), 3.82 (s, 3H), 1.87–1.84 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.8, 165.9, 163.5, 139.2, 134.6, 131.8, 130.9, 128.6, 127.4, 123.9, 123.1, 120.5, 105.7, 102.6, 101.1, 47.6, 33.2, 19.6; IR (KBr) υ 3239, 3104, 2923, 1808, 1735, 1700, 1666, 1604, 1535, 1461, 1376, 1353, 1303, 1268, 1180, 1079, 979, 921, 860 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{17}N_3O_3$ $[M]^+$ 359.1270, found 359.1270.

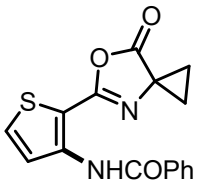
_N_-(3-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)furan-2-yl)benzamide (3n)



3n

16.1 mg (38%); white solid; mp = 153.8–155.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (s, 1H), 8.06 (s, 1H), 7.50–7.46 (m, 2H), 7.30 (t, *J* = 8.0 Hz. 2H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.72 (s, 1H) 1.66–1.61 (m, 2H), 1.14–1.09 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 164.8, 164.8, 145.9, 144.3, 138.0, 129.0, 124.5, 121.7, 120.2, 108.4, 36.6, 16.3; IR (KBr) υ 3293, 2923, 2854, 1646, 1596, 1504, 1438, 1326, 1238, 1168, 1079, 1018, 941, 875, 825 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{18}N_2O_5$ $[M]^+$ 296.0797, found 296.0796.

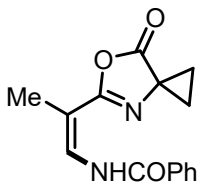
_N_-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)thiophen-3-yl)benzamide (3o)



3o

28.8 mg (46%); white solid; mp = 209.6–211.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.13 (s, 1H), 8.34 (d, *J* = 5.2 Hz, 1H), 7.93 (d, *J* = 7.2Hz, 2H), 7.60–7.47 (m, 4H), 1.85 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.3, 164.5, 159.2, 143.1, 134.0, 132.4, 130.7, 128.9, 127.5, 122.9, 106.9, 47.5, 19.9; IR (KBr) υ 3729, 3698, 3625, 3598, 2919, 1805, 1673, 1608, 1581, 1407, 1292, 1234, 1087, 971, 890 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{16}H_{12}N_2O_3S$ $[M]^+$ 312.0569, found 312.0566.

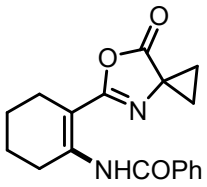
(*Z*)-*N*-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)prop-1-en-1-yl)benzamide (3q)



3q

27.5 mg (51%); white solid; mp = 148.6–151.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.39 (d, *J* = 11.2Hz, 1H), 7.84 (dd, *J* = 8.4, 1.2Hz, 2H), 7.62–7.59 (m, 1H), 7.56 (dt, *J* = 7.2, 1.6Hz, 1H), 7.50–7.46 (m, 2H), 2.03 (d, *J* = 1.2Hz, 3H), 1.83–1.80 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.3, 164.5, 164.2, 132.9, 132.7, 132.4, 128.9, 127.6, 101.1, 47.4, 20.0, 15.6; IR (KBr) υ 3243, 3066, 2923, 2854, 1805, 1677, 1646, 1577, 1527, 1488, 1446, 1357, 1322, 1280, 1241, 1203, 1160, 1052, 979, 925, 890, 852 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{15}H_{14}N_2O_3$ $[M]^+$ 270.1004, found 270.1001.

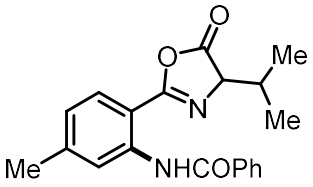
***N*-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)cyclohexan-1-yl)benzamide (3r)**



3r

27.2 mg (44%); white solid; mp = 164.9–166.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.15 (s, 1H), 7.87 (d, *J* = 7.2 Hz, 2H), 7.53 (dt, *J* = 7.2, 1.6 Hz, 1H), 7.43 (t, *J* = 8.0 Hz, 2H), 3.19–3.15 (m, 2H), 2.48–2.44 (m, 2H), 1.80–1.77 (m, 2H), 1.75–1.68 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 165.6, 164.3, 149.4, 135.1, 132.1, 128.6, 127.8, 101.2, 47.1, 28.6, 24.0, 21.9, 21.6, 19.6; IR (KBr) υ 2935, 2857, 1805, 1681, 1631, 1581, 1531, 1492, 1268, 1184, 1160, 1079, 1029, 991, 925, 898, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{18}N_2O_3$ $[M]^+$ 310.1317, found 310.1315.

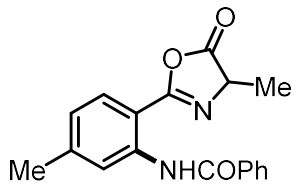
***N*-(2-(4-Isopropyl-5-oxo-4,5-dihydrooxazol-2-yl)-5-methylphenyl)benzamide (3s)**



3s

43.8 mg (65%); white solid; mp = 102.6–105.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.30 (s, 1H), 8.88 (s, 1H), 8.05 (d, *J* = 8.8 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 1H), 4.42 (d, *J* = 4.8 Hz, 1H), 2.47–2.40 (m, 4H), 1.21 (d, *J* = 6.8 Hz, 3H), 0.98 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.7, 166.2, 163.1, 145.6, 140.9, 135.1, 132.1, 129.7, 128.7, 127.7, 124.0, 120.9, 108.7, 70.3, 31.5, 22.4, 19.5, 17.5; IR (KBr) υ 3235, 2965, 2923, 1828, 1681, 1627, 1581, 1542, 1450, 1419, 1334, 1280, 1180, 1130, 1095, 1033, 944, 871, 821 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{20}H_{20}N_2O_3$ $[M]^+$ 336.1474, found 336.1469.

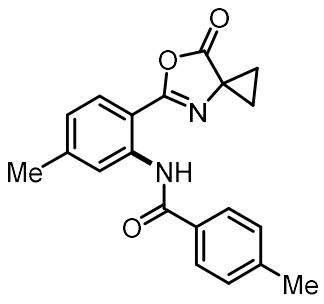
***N*-(5-Methyl-2-(4-methyl-5-oxo-4,5-dihydrooxazol-2-yl)phenyl)benzamide (3t)**



3t

26.0 mg (42%); white solid; mp = 103.1–105.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.23 (s, 1H), 8.84 (s, 1H), 8.02 (d, *J* = 7.6 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.55–7.46 (m, 3H), 6.98 (d, *J* = 8.0 Hz, 1H), 4.57 (q, *J* = 7.6 Hz, 1H), 2.44 (s, 3H), 1.63 (d, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 166.1, 162.8, 145.6, 140.8, 135.1, 132.1, 129.6, 128.8, 127.7, 124.0, 120.9, 108.8, 60.4, 22.4, 17.2; IR (KBr) υ 3232, 3112, 2927, 2857, 1824, 1681, 1627, 1581, 1542, 1446, 1419, 1322, 1276, 1172, 1145, 1110, 998, 917, 890, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{16}N_2O_3$ $[M]^+$ 308.1161, found 308.1160.

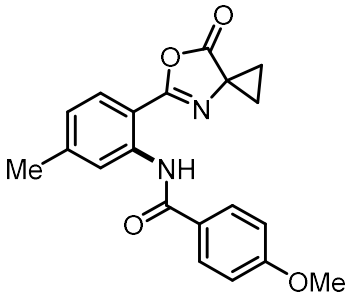
4-Methyl-*N*-(5-methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (4b)



4b

56.9 mg (85%); white solid; mp = 225.6–227.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.85 (s, 1H), 11.80 (s, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.24–7.22 (m, 2H), 6.95 (d, *J* = 8.0 Hz, 1H), 2.42 (s, 3H), 2.39 (s, 3H), 1.85–1.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 165.9, 162.6, 145.1, 142.6, 140.3, 132.4, 129.3, 128.8, 127.5, 123.9, 120.7, 109.2, 47.6, 22.3, 21.6, 19.9; IR (KBr) υ 3259, 2919, 2861, 1812, 1735, 1666, 1619, 1577, 1546, 1511, 1415, 1322, 1272, 1187, 1087, 1006, 975, 921, 887, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{20}H_{18}N_2O_3$ $[M]^+$ 334.1317, found 334.1318.

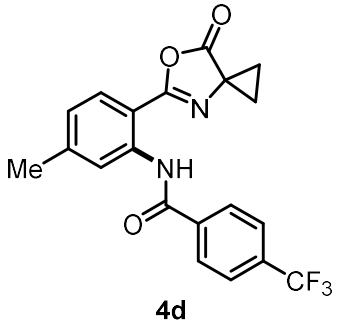
4-Methoxy-*N*-(5-methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (4c)



4c

60.3 mg (86%); white solid; mp = 225.6–227.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.83 (s, 1H), 8.82 (s, 1H), 7.91 (d, *J* = 8.8 Hz, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.00–6.94 (m, 3H), 3.88 (s, 3H), 2.45 (s, 3H), 1.89–1.87 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 165.6, 162.7, 162.7, 145.1, 140.4, 129.4, 128.8, 127.5, 123.8, 120.7, 113.9, 109.2, 55.6, 47.7, 22.4, 19.9; IR (KBr) υ 3262, 2923, 2854, 1805, 1666, 1616, 1581, 1511, 1415, 1322, 1257, 1184, 1087, 1006, 975, 925, 887, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{20}H_{18}N_2O_4$ $[M]^+$ 350.1267, found 350.1268.

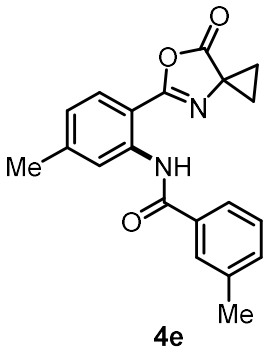
***N*-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)-4-(trifluoromethyl) benzamide (4d)**



4d

49.8 mg (64%); white solid; mp = 178.3–180.2 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.03 (s, 1H), 8.80 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.05 (d, *J* = 8.8 Hz, 1H), 2.47 (s, 3H), 1.93–1.84 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.2, 164.5, 162.8, 145.3, 139.7, 138.5, 133.7 (q, $J_{C\text{-}F}$ = 32.5 Hz), 129.0, 127.9, 125.8 (d, $J_{C\text{-}F}$ = 3.6 Hz), 124.6, 123.7 (d, $J_{C\text{-}F}$ = 271.2 Hz), 120.9, 109.5, 47.6, 22.4, 20.0; IR (KBr) υ 3228, 3112, 2923, 2857, 1808, 1685, 1619, 1581, 1546, 1446, 1419, 1326, 1295, 1272, 1160, 1110, 1064, 1006, 975, 921, 894, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{20}H_{15}F_3N_2O_3$ $[M]^+$ 388.1035, found 388.1032.

3-Methyl-*N*-(5-methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (4e)

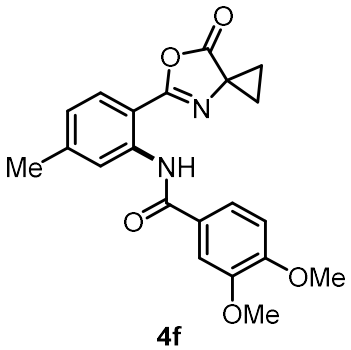


4e

53.5 mg (80%); white solid; mp = 182.7–184.3 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.89 (s, 1H), 8.83 (s, 1H), 7.79–7.73 (m, 3H), 7.32 (d, *J* = 4.8 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 1H), 2.44 (s, 3H), 2.41 (s, 3H), 1.87 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 166.0, 162.6, 145.1, 140.2, 138.3, 135.1, 132.7, 128.8, 128.6, 128.0, 124.8, 124.0, 120.7, 109.2, 47.6, 22.3, 21.5, 19.9; IR (KBr) υ 3100, 2919, 2857, 1808, 1673, 1619, 1577, 1546, 1419, 1326, 1292, 1268, 1083, 1037,

1006, 979, 921, 890, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{20}H_{18}N_2O_3$ $[M]^+$ 334.1317, found 334.1318.

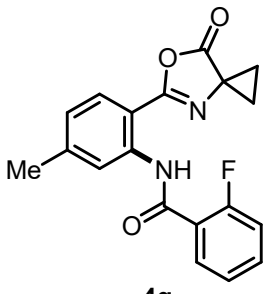
3,4-Dimethoxy-*N*-(5-methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl) benzamide (4f)



4f

63.2 mg (83%); white solid; mp = 194.4–196.5 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.81 (s, 1H), 8.81 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 2.0 Hz, 1H), 7.47 (d, *J* = 8.4, 2.0 Hz, 1H), 7.00 (d, *J* = 7.2 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 3.96 (s, 6H), 2.46 (s, 3H), 1.90–1.87 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 165.7, 162.8, 152.4, 149.3, 145.1, 140.3, 128.9, 127.9, 123.9, 120.8, 120.0, 111.6, 110.2, 109.3, 56.3, 56.2, 47.7, 22.4, 19.9; IR (KBr) υ 3270, 2927, 2846, 1805, 1670, 1619, 1581, 1515, 1446, 1415, 1311, 1265, 1226, 1176, 1087, 1014, 983, 925, 867, 821 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{20}N_2O_5$ $[M]^+$ 380.1372, found 380.1368.

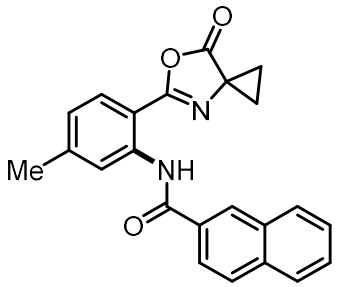
2-Fluoro-*N*-(5-methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (4g)



4g

43.2 mg (63%); white solid; mp = 183.2–185.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.92 (s, 1H), 8.80 (s, 1H), 8.02 (td, *J* = 8.0, 2.0 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.53–7.48 (m, 1H), 7.28 (td, *J* = 8,0, 1.2Hz, 1H), 7.16–7.11 (m, 1H), 7.04–7.01 (m, 1H), 2.45 (s, 3H), 1.85–1.82 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 162.6 (d, $J_{C\text{-}F}$ = 2.2Hz), 161.5 (d, $J_{C\text{-}F}$ = 52.7Hz), 158.8, 144.7, 139.6, 133.6 (d, $J_{C\text{-}F}$ = 8.8Hz), 131.9 (d, $J_{C\text{-}F}$ = 2.3Hz), 128.8, 124.8 (d, $J_{C\text{-}F}$ = 3.6Hz), 124.5, 121.8, 116.3 (d, $J_{C\text{-}F}$ = 23.3Hz), 109.9, 47.8, 22.3, 19.9; IR (KBr) υ 3231, 3081, 1814, 1679, 1649, 1632, 1619, 1582, 1544, 1454, 1415, 1391, 1300, 1267, 1009, 980, 925, 860, 819, 753 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{15}FN_2O_3$ $[M]^+$ 338.1067, found 338.1065.

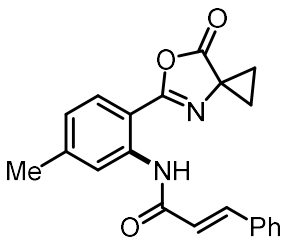
_N_-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)-2-naphthamide (4h)



4h

54.1 mg (73%); white solid; mp = 219.6–220.6 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.13 (s, 1H), 8.90 (s, 1H), 8.46 (s, 1H), 8.03 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.94–7.83 (m, 3H), 7.62–7.56 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 1H), 2.49 (s, 3H), 1.94–1.92 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 166.1, 162.8, 145.3, 140.3, 132.8, 132.5, 129.0, 128.6, 128.6, 128.4, 128.2, 128.1, 128.0, 127.1, 124.1, 124.0, 120.8, 109.3, 47.7, 22.4, 20.0; IR (KBr) 3262, 3054, 2923, 2854, 1808, 1739, 1670, 1619, 1581, 1546, 1419, 1299, 1268, 1230, 1203, 1130, 1079, 1006, 979, 925, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{23}H_{18}N_2O_3$ $[M]^+$ 370.1317, found 370.1314.

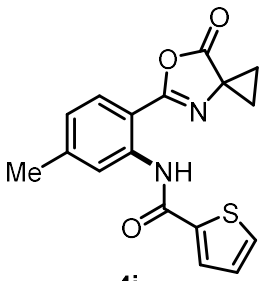
_N_-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)cinnamamide (4i)



4i

53.4 mg (77%); white solid; mp = 177.3–179.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.33 (s, 1H), 8.74 (s, 1H), 7.78 (d, *J* = 4.8 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.52–7.51 (m, 2H), 7.42–7.38 (m, 3H), 6.98 (d, *J* = 4.8 Hz, 1H), 6.43 (d, *J* = 8.8 Hz, 1H), 2.44 (s, 3H), 1.94–1.91 (m, 2H), 1.90–1.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 164.7, 162.5, 145.0, 141.9, 140.1, 134.7, 130.2, 129.0, 128.8, 128.0, 124.0, 122.1, 120.9, 109.0, 47.7, 22.3, 20.1; IR (KBr) υ 3235, 3104, 2923, 1808, 1677, 1619, 1577, 1542, 1446, 1419, 1326, 1261, 1191, 1145, 1091, 1006, 975, 921, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{18}N_2O_3$ $[M]^+$ 346.1317, found 346.1316.

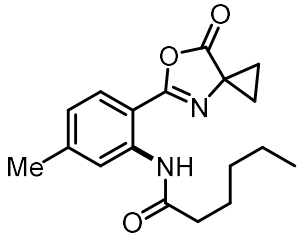
***N*-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)thiophene-2-carboxamide (4j)**



4j

42.5 mg (65%); white solid; mp = 162.5–164.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.88 (s, 1H), 8.72 (s, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.61–7.54 (m, 2H), 7.11 (dd, *J* = 4.0, 5.2 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 2.43 (s, 3H), 1.95–1.86 (m, 4H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 176.5, 162.6, 160.5, 145.1, 140.4, 139.9, 131.2, 128.9, 128.8, 127.8, 124.1, 120.8, 109.1, 47.7, 22.3, 20.0; IR (KBr) υ 3089, 2923, 1812, 1716, 1662, 1619, 1577, 1546, 1415, 1353, 1292, 1276, 1168, 1099, 1037, 1010, 979, 925, 875, 836 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{17}H_{14}N_2O_3S$ $[M]^+$ 326.0725, found 326.0726.

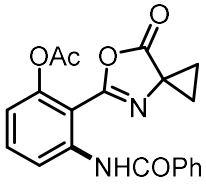
***N*-(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)hexanamide (4k)**



4k

15.1 mg (24%); yellow solid; mp = 74.1–76.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.0 (s, 1H), 8.62 (s, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 2.41 (s, 3H), 2.36 (t, *J* = 8.0 Hz, 2H) 1.84 (brs, 4H), 1.75–1.67 (m, 2H), 1.38–1.32 (m, 4H), 0.91 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 172.4, 162.4, 144.9, 140.0, 128.7, 123.8, 120.7, 108.8, 47.7, 38.7, 31.4, 25.3, 22.5, 22.3, 20.0, 14.0; IR (KBr) υ 3262, 2954, 2923, 2857, 1816, 1700, 1623, 1581, 1538, 1446, 1419, 1326, 1265, 1187, 1091, 1006, 979, 921, 863, 817 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{18}H_{22}N_2O_3$ $[M]^+$ 314.1630, found 314.1632.

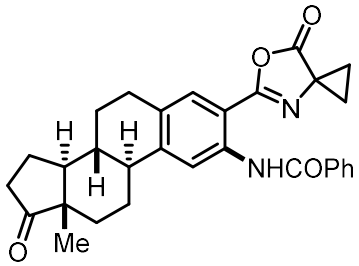
3-Benzamido-2-(4-methyl-5-oxo-4,5-dihydrooxazol-2-yl)phenyl acetate (6a)



6a

60.5 mg (83%); white solid; mp = 180.0–181.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.95 (s, 1H), 8.79 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 6.8 Hz, 2H), 7.53–7.46 (m, 2H), 7.40 (t, *J* = 8.0 Hz, 2H), 6.82 (d, *J* = 8.0 Hz, 1H), 2.32 (s, 3H), 1.82 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 169.9, 165.9, 160.9, 150.0, 141.2, 135.1, 133.9, 132.2, 128.7, 127.4, 118.7, 118.6, 106.8, 46.4, 21.1, 20.3; IR (KBr) υ 3274, 3085, 1927, 1816, 1770, 1685, 1608, 1581, 1542, 1496, 1461, 1369, 1299, 1265, 1195, 1118, 1076, 1010, 979, 921, 871 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{19}H_{16}N_2O_5$ $[M]^+$ 364.1059, found 352.1058.

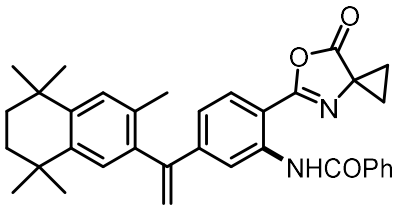
***N*-((8*R*,9*S*,13*S*,14*S*)-13-Methyl-17-oxo-3-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-2-yl)benzamide (6b)**



6b

50.2 mg (52%); white solid; mp = 258.6–260.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.74 (s, 1H), 8.93 (s, 1H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.56 (s, 1H), 7.50–7.38 (m, 3H), 2.87–2.83 (m, 2H), 2.52–2.43 (m, 2H), 2.31–2.25 (m, 1H), 2.12–1.96 (m, 4H), 1.81 (s, 3H), 1.64–1.55 (m, 3H), 1.50–1.41 (m, 3H), 1.19 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 165.8, 162.4, 146.5, 137.9, 135.1, 132.0, 131.5, 128.9, 128.7, 127.4, 119.9, 117.4, 109.6, 50.6, 48.0, 47.6, 45.1, 37.8, 35.9, 31.5, 28.7, 26.3, 25.7, 21.6, 20.0, 13.9; IR (KBr) υ 3278, 3058, 2923, 2854, 1801, 1739, 1677, 1612, 1585, 1527, 1504, 1454, 1407, 1326, 1299, 1272, 1191, 1110, 1076, 1006, 983, 929, 898, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{30}H_{30}N_2O_4$ $[M]^+$ 482.2206, found 482.2204.

***N*-(2-(7-Oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)-5-(1-(3,5,5,8,8-pentamethyl-5,6,7,8-tetrahydronaphthalen-2-yl)vinyl)phenyl)benzamide (6c)**

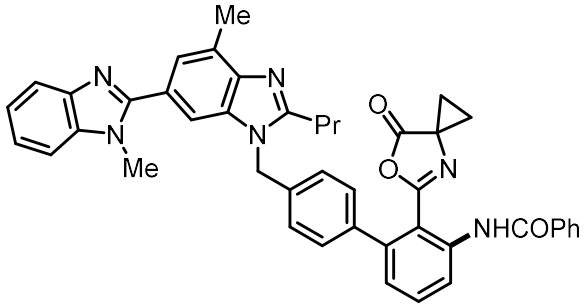


6c

86.3 mg (81%); white solid; mp = 195.4–196.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.83 (s, 1H), 9.12 (s, 1H), 7.82 (d, *J* = 7.2 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 1H), 7.33 (t, *J* = 7.2 Hz, 2H), 7.03 (s, 1H), 6.96 (s, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 5.84 (s, 1H), 5.25 (s, 1H), 1.87 (s, 3H), 1.75 (s, 4H), 1.57 (s, 4H), 1.127–1.16 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$)

δ 176.3, 166.0, 162.5, 149.0, 146.8, 144.4, 142.4, 140.4, 137.9, 135.2, 132.8, 132.0, 128.8, 128.7, 128.2, 128.1, 127.5, 122.1, 118.2, 117.7, 110.7, 47.7, 35.4, 35.3, 34.1, 34.0, 32.1, 32.0, 20.1, 20.0; IR (KBr) υ 3270, 2958, 2923, 2861, 1816, 1685, 1616, 1573, 1535, 1419, 1326, 1268, 1191, 1106, 1076, 1006, 979, 917, 856 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{35}H_{36}N_2O_3$ $[M]^+$ 532.2726, found 532.2728.

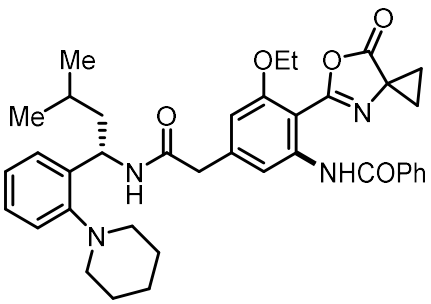
_N_-(4'-((1,7'-Dimethyl-2'-propyl-1_H_,3'_H_-[2,5'-bibenzo[_d_]imidazol]-3'-yl)methyl)-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)-[1,1'-biphenyl]-3-yl)benzamide (6d)



6d

100.7 mg (72%); white solid; mp = 133.3–135.9 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (brs, 1H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.70–7.68 (m, 1H), 7.51–7.46 (m, 1H), 7.38 (td, *J* = 7.6, 1.6 Hz, 1H), 7.29–7.25 (m, 5H), 7.22–7.16 (m, 4H), 7.07 (t, *J* = 7.6 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 6.90 (t, *J* = 7.6 Hz, 1H), 5.35 (s, 2H), 3.55 (s, 3H), 2.87 (t, *J* = 8.0 Hz, 2H), 2.80 (s, 3H), 1.84–1.79 (m, 2H), 1.63–1.60 (m, 2H), 1.49–1.46 (m, 2H), 1.00 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 166.9, 162.8, 158.0, 152.5, 144.2, 141.4, 140.6, 137.8, 134.8, 134.7, 131.8, 131.0, 130.1, 129.4, 129.0, 128.7, 127.9, 127.5, 126.0, 125.6, 124.6, 124.5, 124.3, 124.1, 120.6, 118.0, 110.6, 109.7, 47.9, 47.2, 31.6, 29.9, 21.8, 19.4, 14.3, 14.1; IR (KBr) υ 3054, 2958, 2927, 2857, 1805, 1735, 1662, 1596, 1531, 1504, 1442, 1396, 1319, 1268, 1091, 1060, 1002, 971, 914, 860 cm^{-1}; HRMS (quadrupole, FAB) calcd for $C_{44}H_{38}N_6O_3$ $[M+H]^+$ 699.3078, found 699.3080.

(_S_)-_N_-(3-Ethoxy-5-(2-((3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)amino)-2-oxoethyl)-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)benzamide (6e)



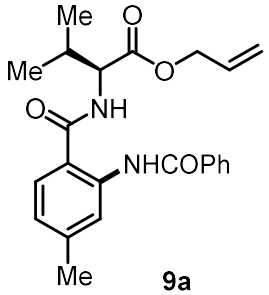
6e

43.4 mg (34%); yellow sticky solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.47 (s, 1H), 8.30 (s, 1H), 7.81 (d, *J* = 7.6 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.12 (s, 2H), 7.06 (d, *J* = 7.6 Hz, 1H), 7.01–6.99 (m, 1H), 6.77 (d, *J* = 8.8 Hz, 1H), 6.64 (s, 1H), 5.32 (q, *J* = 7.6 Hz, 1H), 4.02–3.91 (m, 2H), 3.52 (s, 2H), 2.89 (s, 2H), 2.58 (s, 2H), 2.10 (s, 4H), 1.78 (s, 4H), 1.67 (s, 2H), 1.60–1.53 (m, 3H), 1.34 (t, *J* = 6.8 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 168.7, 165.6, 162.4, 159.3, 152.6, 142.0, 140.5, 138.9, 135.2, 132.2, 128.7, 127.9, 127.8, 127.3, 125.2, 122.9, 113.9, 108.7, 102.6, 65.2, 50.0, 46.8, 46.7, 44.8, 31.0, 26.8, 25.4, 24.3, 22.9, 22.6, 19.8, 14.7; IR (KBr) υ 3289, 2931, 2861, 1812, 1677, 1604, 1573, 1538, 1496, 1438, 1388, 1280, 1141, 1106, 1064, 1006, 979, 921, 863 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{38}H_{44}N_4O_5$ $[M]^+$ 636.3312, found 636.3311.

General procedure and characterization data for the transformation into chiral amino acids (9a and 9b) and racemic amino acid (9c)

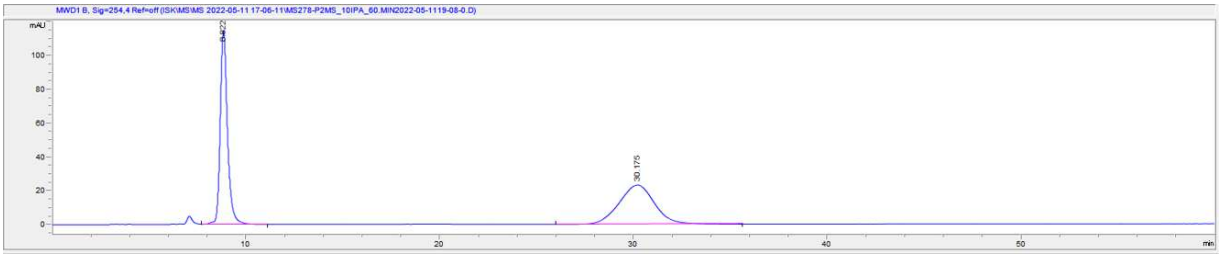
To an oven-dried sealed tube charged with 2-amidobenzoyl azlactone **3s** (0.2 mmol, 100 mol %), urea catalyst **7** (0.01 mmol, 5 mol %) was added DCE (1 mL) followed by allyl alcohol (**8**) (0.3 mmol, 150 mol %) under air at room temperature. The reaction mixture was allowed to stir at room temperature for 48 h. The reaction mixture was diluted with EtOAc (3 mL) and concentrated in vacuo. The residue was purified by flash column chromatography (*n*-hexane:EtOAc) to afford the corresponding compound **9a**. Enantiomeric excess (ee %) of **9a** was determined by analytical liquid chromatography (HPLC) with DAICEL CHIRALPAK® AD-H (4.6 × 250 mm) in comparison with racemic samples.

Allyl (2-benzamido-4-methylbenzoyl)-*L*-valinate (9a)

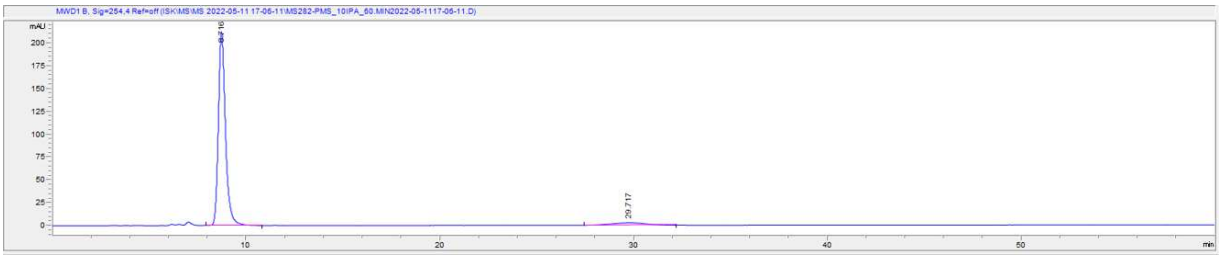


9a

51.3 mg (65%); white solid; mp = 105.0–107.7 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.05 (s, 1H), 8.69 (s, 1H), 8.01 (d, *J* = 6.8, 1.6 Hz, 1H), 7.53–7.48 (m, 4H), 6.93 (d, *J* = 8.4 Hz, 1H), 6.76 (d, *J* = 8.4 Hz, 1H), 5.96–5.87 (m, 1H), 5.35 (d, *J* = 17.2 Hz, 1H), 5.26 (d, *J* = 10.4 Hz, 1H), 4.79 (dd, *J* = 4.8, 8.4 Hz, 1H), 4.72–4.62 (m, 2H), 2.42 (s, 3H), 2.35–2.27 (m, 1H), 1.01 (d, *J* = 8.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 169.1, 165.6, 144.1, 140.2, 135.0, 131.8, 131.5, 128.8, 127.4, 126.7, 123.8, 122.0, 119.3, 117.3, 66.1, 57.4, 31.7, 22.0, 19.1, 18.0; IR (KBr) υ 3313, 3066, 2965, 2931, 1739, 1673, 1643, 1604, 1581, 1523, 1446, 1376, 1288, 1187, 1149, 991, 929 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{23}H_{26}N_2O_4$ $[M]^+$ 394.1893, found 394.1893; HPLC (Chiralcel AD-H column, *n*-hexanes:*i*-PrOH = 90:10, 1.0 mL/min, 254 nm), t_{major} = 8.8 min, t_{minor} = 29.7 min; ee = 89%.

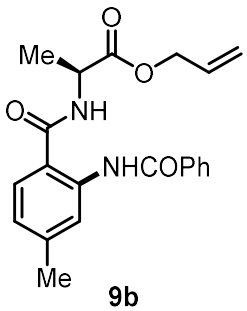


#	Time	Type	Area	Height	Width	Area%	Symmetry
1	8.822	VB	2980.4	114.4	0.392	50.240	0.764
2	30.175	BB	2951.9	23.1	1.9683	49.760	1.02



#	Time	Type	Area	Height	Width	Area%	Symmetry
1	8.716	BB	5362	211.7	0.3815	94.302	0.73
2	29.717	BB	324	2.7	1.424	5.698	1.002

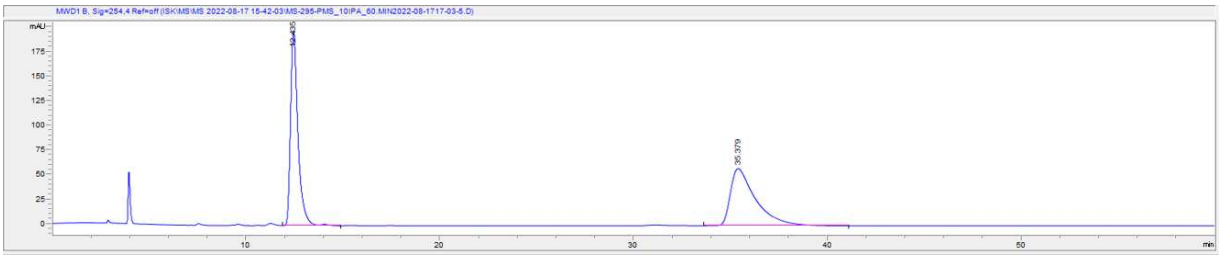
Allyl (2-benzamido-4-methylbenzoyl)-*L*-alaninate (9b)



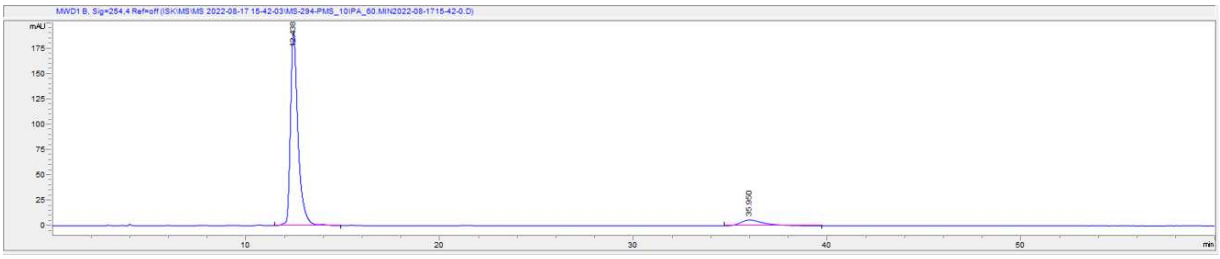
9b

70 mg (96%); white solid; mp = 108.9–111.1 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.13 (s, 1H), 8.70 (s, 1H), 8.02 (d, *J* = 6.4 Hz, 2H), 7.54–7.47 (m, 4H), 6.94–6.84 (m, 2H), 5.96–5.87 (m, 1H), 5.37–5.24 (m, 2H), 4.84–4.67 (m, 3H), 2.42 (s, 3H), 1.55 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 168.8, 165.6, 144.2, 140.4, 135.0, 131.9, 131.4, 128.8, 127.5, 126.8, 123.8,

122.0, 119.1, 116.9, 66.3, 48.6, 22.0, 18.7; IR (KBr) υ 3324, 3066, 2927, 1743, 1670, 1643, 1604, 1581, 1527, 1446, 1380, 1295, 1203, 1172, 1118, 1033, 979, 929, 902 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{21}H_{22}N_2O_4$ $[M]^+$ 366.1580, found 366.1577; HPLC (Chiralcel AD-H column, *n*-hexanes:*i*-PrOH = 90:10, 1 mL/min, 254 nm), t_{major} = 12.4 min, t_{minor} = 35.9 min; ee = 83%.

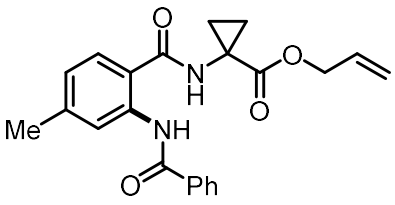


#	Time	Type	Area	Height	Width	Area%	Symmetry
1	12.435	BV R	5240	196.8	0.3966	50.198	0.602
2	35.379	BB	5198.7	57.9	1.2789	49.802	0.41



#	Time	Type	Area	Height	Width	Area%	Symmetry
1	12.438	BV R	5175.7	191.1	0.4019	91.648	0.618
2	35.95	BB	471.7	5.7	1.0085	8.352	0.555

Allyl 1-(2-benzamido-4-methylbenzamido)cyclopropane-1-carboxylate (9c)



9c

23.3 mg (31%); white solid; mp = 162.2–163.4 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.95 (s, 1H), 8.67 (s, 1H), 8.02 (dd, J = 8.4, 1.6Hz, 2H), 7.56–7.47 (m, 3H), 7.41 (d, *J* = 8.0Hz, 1H), 6.91 (d, *J* = 8.0Hz, 1H), 6.78 (s, 1H), 5.83–5.73 (m, 1H) 5.21 (dd, *J* = 17.2, 2.4Hz, 1H), 5.07 (dd, *J* = 10.4, 1.6Hz, 1H), 4.57 (dt, *J* = 5.6, 1.6Hz, 2H), 2.42 (s, 3H), 1.74–1.70 (m, 2H), 1.31–1.28 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.8, 170.8, 165.7, 144.3, 140.2, 134.9, 131.9, 131.7, 128.8, 127.5, 126.7, 123.8, 122.2, 118.4, 117.4, 66.1, 34.2, 22.1, 17.9; IR (KBr) υ 3324, 2923, 2854, 1727, 1650, 1608, 1581, 1527, 1442, 1322, 1284, 1168, 1033, 979, 933 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{22}H_{22}N_2O_4$ $[M]^+$ 378.1580, found 378.1581.

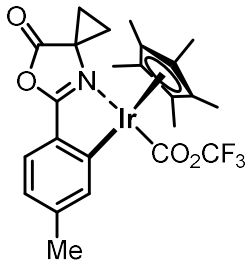
Experimental procedure of gram-scale reaction of 1a

To an oven-dried sealed tube charged with 2-aryl azlactone **1a** (1 g, 5.0 mmol, 100 mol %), $[IrCp^*Cl_2]_2$ (99.6 mg, 0.125 mmol, 2.5 mol %), $AgNTf_2$ (194.0 mg, 0.5 mmol, 10 mol %), LiOAc (99.0 mg, 1.5 mmol, 30 mol %), and acyl azide **2a** (1.1 g, 7.5 mmol, 150 mol %) was added DCE (25 mL) under air at room temperature. The reaction mixture was allowed to stir at 50 °C for 24 h. The reaction mixture was cooled to room temperature, diluted with EtOAc (45 mL) and concentrated in vacuo. The residue was purified by flash column chromatography (*n*-hexane:EtOAc = 10:1 to 8:1) to afford 1.15 g of **3a** in 72% yield.

General procedure and characterization data for the formation of iridacycle-1a (CCDC 2162876)

To an oven-dried sealed tube charged with 2-phenyl azlactone (**1a**) (0.4 mmol, 100 mol %), $[IrCp^*Cl_2]_2$ (159.4 mg, 0.2 mmol, 50 mol %), Li_2CO_3 (14.8 mg, 0.2 mmol, 50 mol %) and AgTFA (44.2 mg, 0.2 mmol, 50 mol %) was added DCE (10 mL) under Ar at room temperature. The reaction mixture was allowed to stir at 40 °C for 24 h. The reaction mixture was cooled to room temperature, diluted with EtOAc (3 mL) and concentrated in vacuo. The resulting powder was crystalized with (*n*-hexanes/EtOAc) at room temperature to afford **iridacycle-1a** (82.1 mg) in 32% yield.

(5-Methyl-2-(7-oxo-6-oxa-4-azaspiro[2.4]hept-4-en-5-yl)phenyl)(1,2,3,4,5-pentamethylcyclopenta-2,4-dien-1-yl)((trifluoromethoxy)carbonyl)iridium (iridacylce-1a)



iridacycle-1a

82.1 mg (32%); yellow solid; mp = 203.2–205.0 °C; ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.43 (d, J = 7.7 Hz, 1H), 6.93 (d, J = 7.7 Hz, 1H), 2.46 (s, 3H), 2.21–2.18 (m, 1H), 1.99–1.95 (m, 1H), 1.89–1.85 (m, 1H), 1.77–1.75 (m, 1H), 1.72 (s, 15H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 178.1, 174.7, 164.1, 162.8 (d, $J_{C\text{-}F}$ = 35.8 Hz), 143.8, 136.8, 129.4, 126.3, 124.4, 114.4 (d, $J_{C\text{-}F}$ = 289.2 Hz), 87.3, 48.5, 22.4, 18.0, 17.8, 9.9; IR (KBr) υ 2989, 2919, 1847, 1824, 1704, 1612, 1581, 1538, 1461, 1376, 1326, 1276, 1187, 1137, 1033, 1010, 921, 879, 836 cm^{-1}; HRMS (quadrupole, EI) calcd for $C_{24}H_{25}F_3IrNO_4$ $[M]^+$ 641.1365, found 641.1362.

X-ray crystallographic data of compound 3a (CCDC 2154665)

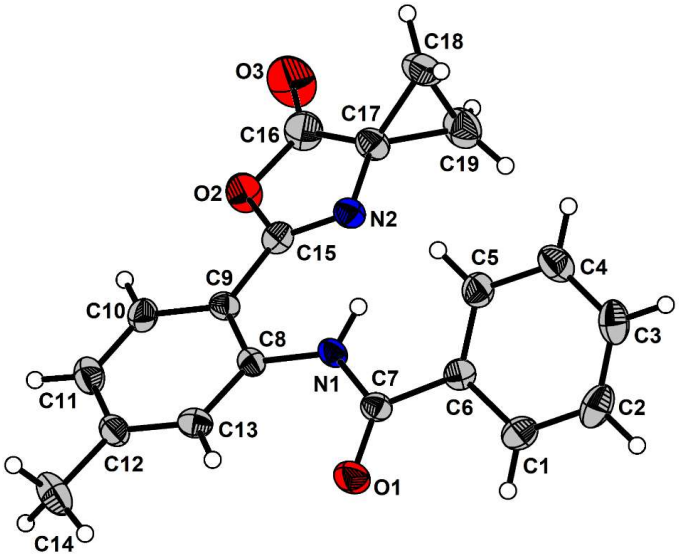
Detailed experimental description for the crystal measurement of compound 3a

Crystals grew as colorless blocks in dichloromethane by slow evaporation from pentane. The crystal structures of compound **3a** were determined by standard crystallographic methods. A colorless plate-like-shaped crystal (0.020 x 0.100 x 0.160 mm^3) was used for single-crystal X-ray diffraction. The data were collected at 296 (2) K using a Bruker D8 Venture equipped with IμS micro-focus sealed tube Mo Kα (λ= 0.71073 Å) and a PHOTON III M14 detector in Western Seoul Center of Korea Basic Science Institute. Data collection and integration were performed with SMART APEX3 software package (SAINT). Absorption correction was performed by multi-scan method implemented in SADABS. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXTL program package (version 6.14). All the non-hydrogen atoms were refined anisotropically, and hydrogen atoms were added to their geometrically ideal positions.

Details of crystal data, data collection and structure refinement are listed in Table S1. Further details of the individual structures can be obtained from the Cambridge Crystallographic Data Centre by quoting **CCDC 2154665**.

Sample preparation (vapor diffusion)

Compound **3a** (3.8 mg) was dissolved with 1 mL of CH_2Cl_2 in opened inner vessel, and pentane (5 mL) as an anti-solvent has been employed in closed outer vessel. After vapor diffusion for 9 days, the single crystals of compound **3a** were obtained.



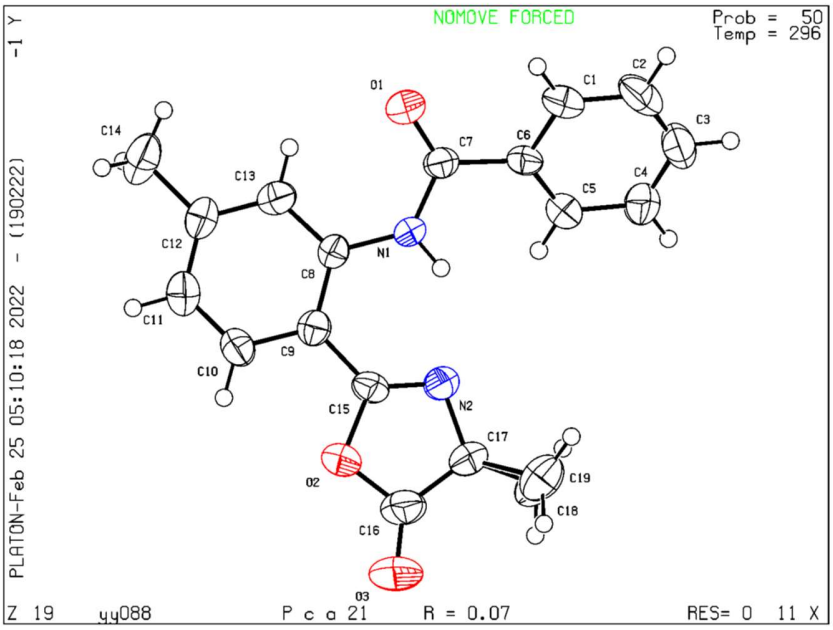


Table S1: Data collection details for 3a.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	60.586	18.54	-174.46	-156.00	54.74	1.00	206	10.00	0.71073	50	30.0	n/a
Omega	60.586	27.81	-165.19	0.00	54.74	1.00	206	10.00	0.71073	50	30.0	n/a
Omega	60.586	18.54	-174.46	102.00	54.74	1.00	206	10.00	0.71073	50	30.0	n/a
Omega	60.586	18.54	-174.46	-54.00	54.74	1.00	206	10.00	0.71073	50	30.0	n/a
Omega	60.586	18.54	-174.46	153.00	54.74	1.00	206	10.00	0.71073	50	30.0	n/a
Phi	60.586	18.54	31.54	0.00	54.74	1.00	360	10.00	0.71073	50	30.0	n/a
Phi	60.586	0.00	0.00	0.00	54.74	360.00	1	108.00	0.71073	50	30.0	n/a

A total of 1391 frames were collected. The total exposure time was 3.89 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using an orthorhombic unit cell yielded a total of 56517 reflections to a maximum θ angle of 28.29° (0.75 Å resolution), of which 4026 were independent (average redundancy 14.038, completeness = 99.9%, R_{int} = 12.73%, R_{sig} = 6.37%) and 2506 (62.25%) were greater than $2\sigma(F^2)$. The final cell constants of a = 11.6867(10) Å, b = 19.3211(17) Å, c = 7.1481(6) Å, volume = 1614.0(2) $Å^3$, are based upon the refinement of the XYZ-centroids of 5067 reflections above 20 σ(I) with 5.471° < 2θ < 43.62°. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.910. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.9860 and 0.9980.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P c a 21, with Z = 4 for the formula unit, $C_{19}H_{16}N_2O_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 218 variables converged at R1 = 6.90%, for the observed data and wR2 = 13.72% for all data. The goodness-of-fit was 1.029. The largest peak in the final difference electron density synthesis was 0.146 $e^-/Å^3$ and the largest hole was -0.182 $e^-/Å^3$ with an RMS deviation of 0.036 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.318 g/cm^3 and F(000), 672 e^-.

Table S2. Sample and crystal data for 3a.

Chemical formula	$C_{19}H_{16}N_2O_3$	
Formula weight	320.34 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal size	0.020 x 0.100 x 0.160 mm	
Crystal habit	colorless plate	
Crystal system	orthorhombic	
Space group	P c a 2_1	
Unit cell dimensions	a = 11.6867(10) Å	α = 90°
	b = 19.3211(17) Å	β = 90°
	c = 7.1481(6) Å	γ = 90°
Volume	1614.0(2) $Å^3$	
Z	4	
Density (calculated)	1.318 g/cm^3	

Absorption coefficient	0.091 mm^{-1}
F(000)	672

Table S3. Data collection and structure refinement for 3a.

Theta range for data collection	2.04 to 28.29°	
Index ranges	-15≤h≤15, -25≤k≤25, -9≤l≤9	
Reflections collected	56517	
Independent reflections	4026 [R(int) = 0.1273]	
Coverage of independent reflections	99.9%	
Absorption correction	Multi-Scan	
Max. and min. transmission	0.9980 and 0.9860	
Structure solution technique	direct methods	
Structure solution program	SHELXT 2018/2 (Sheldrick, 2018)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2018/3 (Sheldrick, 2018)	
Function minimized	$\Sigma\ w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	4026 / 1 / 218	
Goodness-of-fit on F^2	1.029	
Final R indices	2506 data; $I>2\sigma(I)$	$R_1 = 0.0690$, $wR_2 = 0.1191$
	all data	$R_1 = 0.1231$, $wR_2 = 0.1372$
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0432P)^2+0.6033P]$ where $P=(F_o^2+2F_c^2)/3$	
Absolute structure parameter	-0.7(10)	
Largest diff. peak and hole	0.146 and -0.182 $eÅ^{-3}$	
R.M.S. deviation from mean	0.036 $eÅ^{-3}$	

Table S4. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$) for 3a.

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

	x/a	y/b	z/c	U(eq)
C1	0.4714(4)	0.9408(2)	0.6842(7)	0.0548(12)
C2	0.5797(5)	0.9703(2)	0.6785(8)	0.0653(15)

C3	0.6602(4)	0.9456(2)	0.5581(8)	0.0612(14)
C4	0.6360(4)	0.8914(2)	0.4430(9)	0.0591(12)
C5	0.5272(3)	0.8613(2)	0.4475(8)	0.0504(11)
C6	0.4452(3)	0.88554(19)	0.5682(6)	0.0389(9)
C7	0.3258(3)	0.8577(2)	0.5745(6)	0.0388(9)
C8	0.2141(3)	0.75301(19)	0.4885(5)	0.0336(9)
C9	0.2233(3)	0.68135(19)	0.4564(6)	0.0384(9)
C10	0.1227(3)	0.6430(2)	0.4321(7)	0.0456(10)
C11	0.0180(3)	0.6743(2)	0.4337(7)	0.0512(11)
C12	0.0077(3)	0.7449(2)	0.4622(6)	0.0452(10)
C13	0.1060(3)	0.7833(2)	0.4894(6)	0.0445(10)
C14	0.8935(3)	0.7803(3)	0.4534(9)	0.0708(14)
C15	0.3334(3)	0.64644(18)	0.4434(6)	0.0391(9)
C16	0.4394(4)	0.5515(2)	0.4153(9)	0.0636(13)
C17	0.5096(3)	0.6134(2)	0.4288(7)	0.0477(10)
C18	0.6211(4)	0.6186(3)	0.3209(7)	0.0650(15)
C19	0.6239(5)	0.6114(3)	0.5258(7)	0.0669(15)
N1	0.3144(3)	0.79171(16)	0.5121(5)	0.0383(8)
N2	0.4338(3)	0.67144(15)	0.4436(5)	0.0418(8)
O1	0.2469(3)	0.89217(14)	0.6347(5)	0.0599(9)
O2	0.3269(2)	0.57426(13)	0.4279(6)	0.0601(9)
O3	0.4609(3)	0.49159(16)	0.3988(9)	0.1021(15)

Table S5. Bond lengths (Å) for 3a.

C1-C6	1.387(6)	C1-C2	1.389(6)
C1-H1	0.93	C2-C3	1.361(7)
C2-H2	0.93	C3-C4	1.362(7)
C3-H3	0.93	C4-C5	1.399(5)
C4-H4	0.93	C5-C6	1.372(6)
C5-H5	0.93	C6-C7	1.496(5)
C7-O1	1.216(5)	C7-N1	1.358(5)
C8-C13	1.393(5)	C8-N1	1.400(5)
C8-C9	1.408(5)	C9-C10	1.401(5)
C9-C15	1.456(5)	C10-C11	1.365(5)
C10-H10	0.93	C11-C12	1.385(6)
C11-H11	0.93	C12-C13	1.380(6)
C12-C14	1.501(6)	C13-H13	0.93

C14-H14A	0.96	C14-H14B	0.96
C14-H14C	0.96	C15-N2	1.268(4)
C15-O2	1.401(4)	C16-O3	1.191(5)
C16-O2	1.389(5)	C16-C17	1.454(6)
C17-N2	1.433(4)	C17-C19	1.505(7)
C17-C18	1.518(7)	C18-C19	1.472(6)
C18-H18A	0.97	C18-H18B	0.97
C19-H19A	0.97	C19-H19B	0.97
N1-H1A	0.86		

Table S6. Bond angles (°) for 3a.

C6-C1-C2	120.0(4)	C6-C1-H1	120.0
C2-C1-H1	120.0	C3-C2-C1	120.3(4)
C3-C2-H2	119.9	C1-C2-H2	119.9
C2-C3-C4	120.6(4)	C2-C3-H3	119.7
C4-C3-H3	119.7	C3-C4-C5	119.6(5)
C3-C4-H4	120.2	C5-C4-H4	120.2
C6-C5-C4	120.5(4)	C6-C5-H5	119.8
C4-C5-H5	119.8	C5-C6-C1	119.0(4)
C5-C6-C7	123.2(4)	C1-C6-C7	117.7(4)
O1-C7-N1	123.8(4)	O1-C7-C6	121.4(4)
N1-C7-C6	114.8(3)	C13-C8-N1	122.3(3)
C13-C8-C9	118.9(3)	N1-C8-C9	118.8(3)
C10-C9-C8	118.5(3)	C10-C9-C15	119.3(3)
C8-C9-C15	122.2(3)	C11-C10-C9	121.1(4)
C11-C10-H10	119.5	C9-C10-H10	119.5
C10-C11-C12	121.1(4)	C10-C11-H11	119.5
C12-C11-H11	119.5	C13-C12-C11	118.5(4)
C13-C12-C14	120.1(4)	C11-C12-C14	121.3(4)
C12-C13-C8	121.9(4)	C12-C13-H13	119.0
C8-C13-H13	119.0	C12-C14-H14A	109.5
C12-C14-H14B	109.5	H14A-C14-H14B	109.5
C12-C14-H14C	109.5	H14A-C14-H14C	109.5
H14B-C14-H14C	109.5	N2-C15-O2	115.5(3)
N2-C15-C9	129.9(3)	O2-C15-C9	114.7(3)
O3-C16-O2	120.9(4)	O3-C16-C17	133.4(5)
O2-C16-C17	105.7(3)	N2-C17-C16	107.4(3)

N2-C17-C19	122.4(4)	C16-C17-C19	120.7(4)
N2-C17-C18	121.1(4)	C16-C17-C18	120.3(4)
C19-C17-C18	58.3(3)	C19-C18-C17	60.4(4)
C19-C18-H18A	117.7	C17-C18-H18A	117.7
C19-C18-H18B	117.7	C17-C18-H18B	117.7
H18A-C18-H18B	114.8	C18-C19-C17	61.3(4)
C18-C19-H19A	117.6	C17-C19-H19A	117.6
C18-C19-H19B	117.6	C17-C19-H19B	117.6
H19A-C19-H19B	114.7	C7-N1-C8	128.6(3)
C7-N1-H1A	115.7	C8-N1-H1A	115.7
C15-N2-C17	105.9(3)	C16-O2-C15	105.5(3)

Table S7. Anisotropic atomic displacement parameters (Å^2) for 3a.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.062(3)	0.037(2)	0.065(3)	-0.013(2)	0.001(2)	0.000(2)
C2	0.081(4)	0.043(3)	0.072(4)	-0.011(3)	-0.017(3)	-0.017(3)
C3	0.050(3)	0.054(3)	0.080(4)	0.008(3)	-0.013(3)	-0.017(2)
C4	0.042(2)	0.057(3)	0.078(3)	0.002(3)	0.011(3)	-0.004(2)
C5	0.047(2)	0.039(2)	0.065(3)	-0.006(2)	-0.003(3)	-0.0038(19)
C6	0.044(2)	0.0260(19)	0.046(2)	0.003(2)	-0.001(2)	0.0019(17)
C7	0.039(2)	0.034(2)	0.043(2)	-0.0006(19)	0.0017(19)	0.0050(19)
C8	0.031(2)	0.038(2)	0.032(2)	0.0045(17)	0.0008(15)	0.0005(16)
C9	0.037(2)	0.043(2)	0.035(2)	0.001(2)	0.0049(19)	-0.0008(17)
C10	0.045(2)	0.043(2)	0.049(3)	-0.004(2)	-0.002(2)	-0.0086(18)
C11	0.040(2)	0.061(3)	0.053(3)	0.005(3)	-0.001(2)	-0.009(2)
C12	0.033(2)	0.061(3)	0.041(2)	0.002(2)	0.0018(19)	0.0003(19)
C13	0.046(2)	0.041(2)	0.046(3)	0.001(2)	0.005(2)	0.007(2)
C14	0.038(2)	0.091(4)	0.083(4)	0.014(4)	-0.002(3)	0.005(2)
C15	0.048(2)	0.0288(19)	0.040(2)	0.003(2)	-0.003(2)	0.0003(17)
C16	0.064(3)	0.045(3)	0.082(4)	-0.004(3)	-0.011(3)	0.015(2)
C17	0.043(2)	0.044(2)	0.056(3)	0.001(2)	-0.001(2)	0.0112(18)
C18	0.050(3)	0.076(4)	0.068(4)	-0.003(3)	0.010(3)	0.017(3)
C19	0.054(3)	0.078(4)	0.069(4)	0.008(3)	-0.008(3)	0.015(3)

N1	0.0302(17)	0.0316(17)	0.053(2)	-0.0027(16)	0.0011(15)	0.0053(14)
N2	0.0389(18)	0.0393(17)	0.047(2)	0.0000(18)	0.0024(18)	0.0041(15)
O1	0.0471(17)	0.0476(16)	0.085(3)	-0.0167(17)	0.0066(17)	0.0067(17)
O2	0.0548(19)	0.0325(14)	0.093(2)	-0.0034(19)	-0.004(2)	0.0004(13)
O3	0.091(3)	0.0406(18)	0.174(4)	-0.004(3)	-0.011(3)	0.0209(18)

Table S8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for 3a.

	x/a	y/b	z/c	U(eq)
H1	0.4165	0.9581	0.7659	0.066
H2	0.5974	1.0071	0.7573	0.078
H3	0.7322	0.9660	0.5544	0.073
H4	0.6916	0.8744	0.3620	0.071
H5	0.5103	0.8246	0.3680	0.061
H10	0.1273	0.5954	0.4146	0.055
H11	-0.0474	0.6478	0.4154	0.061
H13	0.0998	0.8307	0.5089	0.053
H14A	-0.1609	0.7549	0.5270	0.106
H14B	-0.1321	0.7821	0.3259	0.106
H14C	-0.0997	0.8265	0.5018	0.106
H18A	0.6441	0.5790	0.2464	0.078
H18B	0.6413	0.6632	0.2681	0.078
H19A	0.6461	0.6516	0.5985	0.08
H19B	0.6489	0.5674	0.5768	0.08
H1A	0.3770	0.7709	0.4833	0.046

X-ray crystallographic data of iridacycle-1a (CCDC 2162876)

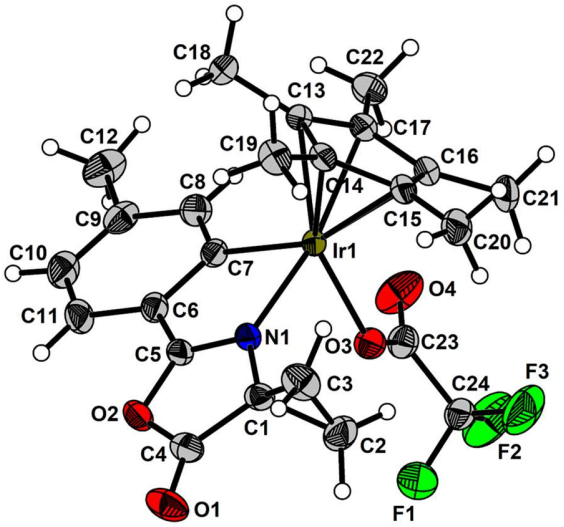
Detailed experimental description for the crystal measurement of iridacycle-1a

Crystals grew as light pink rods in dichloromethane by slow evaporation from pentane. The crystal structures of **iridacycle-1a** were determined by standard crystallographic methods. A colorless plate-like-shaped crystal (0.020 x 0.080 x 0.250 mm^3) was used for single-crystal X-ray diffraction. The data were collected at 296 (2) K using a Bruker D8 Venture equipped with IμS micro-focus sealed tube Mo Kα (λ = 0.71073 Å) and a PHOTON III M14 detector in Western Seoul Center of Korea Basic Science Institute. Data collection and integration were performed with SMART APEX3 software package (SAINT). Absorption correction was performed by multi-scan method implemented in SADABS. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXTL program package (version 6.14). All the non-hydrogen atoms were refined anisotropically, and hydrogen atoms were added to their geometrically ideal positions.

Details of crystal data, data collection and structure refinement are listed in Table S9. Further details of the individual structures can be obtained from the Cambridge Crystallographic Data Centre by quoting **CCDC 2162876**.

Sample preparation (vapor diffusion)

Iridacycle-1a (3.3 mg) was dissolved with 1 mL of CH_2Cl_2 in opened inner vessel, and pentane (4 mL) as an anti-solvent has been employed in closed outer vessel. After vapor diffusion for 6 days, the single crystals of **iridacycle-1a** were obtained.



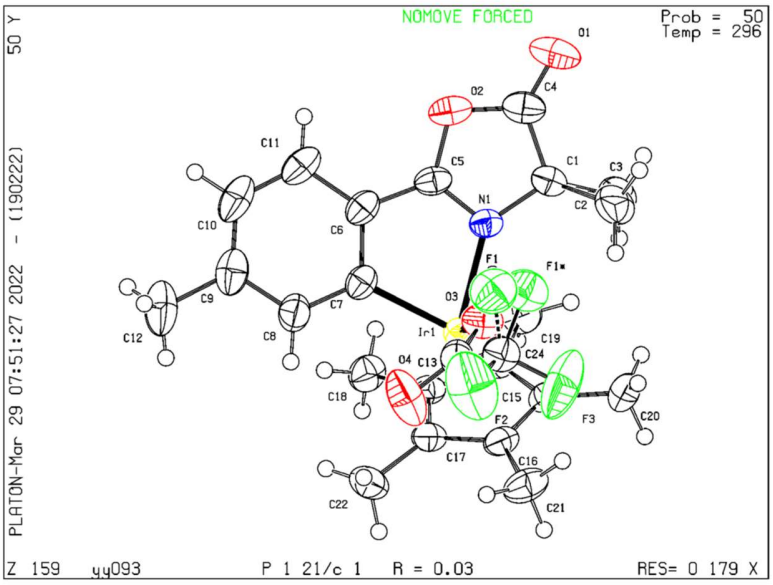


Table S9: Data collection details for iridacycle-1a.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	60.501	18.54	-174.46	-156.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	27.81	-165.19	0.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	18.54	-174.46	102.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	18.54	-174.46	-54.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	18.54	-174.46	153.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	18.54	-174.46	-105.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Omega	60.501	18.54	-174.46	51.00	54.74	0.50	412	10.00	0.71073	50	30.0	n/a
Phi	60.501	18.54	-174.46	0.00	54.74	0.50	720	10.00	0.71073	50	30.0	n/a
Phi	60.501	0.00	0.00	0.00	54.74	360.00	1	108.00	0.71073	50	30.0	n/a

A total of 3605 frames were collected. The total exposure time was 10.04 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 107010 reflections to a maximum θ angle of 28.45° (0.75 Å resolution), of which 5722 were independent (average redundancy 18.702, completeness = 99.0%, R_{int} = 4.63%, R_{sig} = 1.63%) and 5480 (95.77%) were greater than $2\sigma(F^2)$. The final cell constants of a = 8.971(2) Å, b = 27.011(6) Å, c = 9.490(2) Å, β = 93.878(7)°, volume = 2294.3(9) $Å^3$, are based upon the refinement of the XYZ-centroids of 9688 reflections above 20 σ(I) with 5.459° < 2θ < 56.89°. Data were corrected for absorption effects using the Multi-Scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.482. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.3210 and 0.8920.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/c 1, with Z = 4 for the formula unit, $C_{24}H_{25}F_3IrNO_4$. The final anisotropic full-matrix least-squares refinement on F^2 with 314 variables converged at R1 = 2.61%, for the observed data and wR2 = 5.60% for all data. The goodness-of-fit was 1.178. The largest peak in the final difference electron density synthesis was 0.903 $e^-/Å^3$ and the largest hole was -1.297 $e^-/Å^3$ with an RMS deviation of 0.087 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.855 g/cm^3 and F(000), 1248 e^-.

Table S10. Sample and crystal data for iridacycle-1a.

Chemical formula	$C_{24}H_{25}F_3IrNO_4$	
Formula weight	640.65 g/mol	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal size	0.020 x 0.080 x 0.250 mm	
Crystal habit	light pink rod	
Crystal system	monoclinic	
Space group	P 2_1/c	
Unit cell dimensions	a = 8.971(2) Å	α = 90°
	b = 27.011(6) Å	β = 93.878(7)°
	c = 9.490(2) Å	γ = 90°
Volume	2294.3(9) $Å^3$	
Z	4	
Density (calculated)	1.855 g/cm^3	

Absorption coefficient	5.874 mm^{-1}
F(000)	1248

Table S11. Data collection and structure refinement for iridacycle-1a.

Theta range for data collection	2.28 to 28.45°	
Index ranges	-11≤h≤11, -36≤k≤35, -12≤l≤12	
Reflections collected	107010	
Independent reflections	5722 [R(int) = 0.0463]	
Coverage of independent reflections	99.0%	
Absorption correction	Multi-Scan	
Max. and min. transmission	0.8920 and 0.3210	
Structure solution technique	direct methods	
Structure solution program	SHELXT 2018/2 (Sheldrick, 2018)	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2018/3 (Sheldrick, 2018)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	5722 / 14 / 314	
Goodness-of-fit on F^2	1.178	
Δ/σ_{max}	0.003	
Final R indices	5480 data; I>2σ(I)	R_1 = 0.0261, wR_2 = 0.0553
	all data	R_1 = 0.0278, wR_2 = 0.0560
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0143P)^2+4.2571P]$ where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.903 and -1.297 $eÅ^{-3}$	
R.M.S. deviation from mean	0.087 $eÅ^{-3}$	

Table S12. Atomic coordinates and equivalent isotropic atomic displacement parameters ($Å^2$) for iridacycle-1a.

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

	x/a	y/b	z/c	U(eq)
C1	0.4610(4)	0.68736(13)	0.1033(4)	0.0397(7)
C2	0.4418(6)	0.65458(18)	0.9756(5)	0.0654(12)

C3	0.3160(5)	0.66054(18)	0.0670(5)	0.0626(11)
C4	0.4672(4)	0.74139(14)	0.0872(4)	0.0476(9)
C5	0.6206(4)	0.71735(12)	0.2655(4)	0.0379(7)
C6	0.7369(4)	0.71807(13)	0.3752(4)	0.0405(7)
C7	0.7816(4)	0.67003(13)	0.4166(4)	0.0381(7)
C8	0.9013(4)	0.66756(15)	0.5179(4)	0.0486(9)
C9	0.9668(4)	0.70996(17)	0.5804(4)	0.0521(10)
C10	0.9140(5)	0.75632(17)	0.5394(5)	0.0609(12)
C11	0.8011(5)	0.76123(15)	0.4354(5)	0.0531(10)
C12	0.0944(5)	0.7047(2)	0.6917(5)	0.0707(14)
C13	0.5850(4)	0.58401(12)	0.5216(3)	0.0360(7)
C14	0.4596(4)	0.58260(12)	0.4194(4)	0.0371(7)
C15	0.4937(4)	0.54889(12)	0.3080(4)	0.0400(7)
C16	0.6401(4)	0.53120(12)	0.3404(4)	0.0410(8)
C17	0.6990(4)	0.55256(12)	0.4714(4)	0.0396(7)
C18	0.5884(5)	0.61084(14)	0.6595(4)	0.0497(9)
C19	0.3139(4)	0.60842(15)	0.4356(5)	0.0514(9)
C20	0.3899(5)	0.53129(16)	0.1874(5)	0.0587(11)
C21	0.7221(5)	0.49543(15)	0.2531(5)	0.0583(11)
C22	0.8436(5)	0.53827(16)	0.5495(5)	0.0539(10)
C23	0.9071(5)	0.59470(15)	0.1416(4)	0.0477(8)
C24	0.9478(4)	0.58554(12)	0.9891(4)	0.0465(8)
F1	0.9702(18)	0.6279(2)	0.9255(12)	0.073(3)
F1*	0.9055(12)	0.6203(3)	0.8975(6)	0.070(2)
F2	0.0866(4)	0.57198(18)	0.9794(4)	0.1145(14)
F3	0.8704(5)	0.55000(15)	0.9292(4)	0.1172(15)
N1	0.5621(3)	0.67613(10)	0.2223(3)	0.0356(6)
O1	0.4040(4)	0.76866(12)	0.0049(4)	0.0675(9)
O2	0.5674(3)	0.75834(9)	0.1941(3)	0.0493(6)
O3	0.7807(3)	0.61273(10)	0.1458(3)	0.0478(6)
O4	0.9977(4)	0.58236(17)	0.2357(4)	0.0820(11)
Ir1	0.65405(2)	0.61300(2)	0.32700(2)	0.03164(4)

Table S13. Bond lengths (Å) for iridacycle-1a.

C1-N1	1.432(4)	C1-C4	1.469(5)
C1-C2	1.502(5)	C1-C3	1.508(6)
C2-C3	1.478(7)	C2-H2A	0.97

C2-H2B	0.97	C3-H3A	0.97
C3-H3B	0.97	C4-O1	1.189(5)
C4-O2	1.387(5)	C5-N1	1.286(4)
C5-O2	1.367(4)	C5-C6	1.424(5)
C6-C11	1.404(5)	C6-C7	1.406(5)
C7-C8	1.394(5)	C7-Ir1	2.067(3)
C8-C9	1.401(5)	C8-H8	0.93
C9-C10	1.385(7)	C9-C12	1.511(6)
C10-C11	1.373(6)	C10-H10	0.93
C11-H11	0.93	C12-H12A	0.96
C12-H12B	0.96	C12-H12C	0.96
C13-C17	1.435(5)	C13-C14	1.436(5)
C13-C18	1.494(5)	C13-Ir1	2.136(3)
C14-C15	1.443(5)	C14-C19	1.499(5)
C14-Ir1	2.166(3)	C15-C16	1.412(5)
C15-C20	1.502(5)	C15-Ir1	2.251(3)
C16-C17	1.438(5)	C16-C21	1.498(5)
C16-Ir1	2.217(3)	C17-C22	1.501(5)
C17-Ir1	2.152(3)	C18-H18A	0.96
C18-H18B	0.96	C18-H18C	0.96
C19-H19A	0.96	C19-H19B	0.96
C19-H19C	0.96	C20-H20A	0.96
C20-H20B	0.96	C20-H20C	0.96
C21-H21A	0.96	C21-H21B	0.96
C21-H21C	0.96	C22-H22A	0.96
C22-H22B	0.96	C22-H22C	0.96
C23-O4	1.213(5)	C23-O3	1.237(5)
C23-C24	1.536(5)	C24-F3	1.294(5)
C24-F2	1.307(5)	C24-F1	1.316(2)
C24-F1*	1.318(2)	N1-Ir1	2.113(3)
O3-Ir1	2.124(3)		

Table S14. Bond angles (°) for iridacycle-1a.

N1-C1-C4	105.4(3)	N1-C1-C2	122.9(3)
C4-C1-C2	120.3(3)	N1-C1-C3	124.7(3)
C4-C1-C3	119.5(3)	C2-C1-C3	58.8(3)
C3-C2-C1	60.8(3)	C3-C2-H2A	117.7

C1-C2-H2A	117.7	C3-C2-H2B	117.7
C1-C2-H2B	117.7	H2A-C2-H2B	114.8
C2-C3-C1	60.4(3)	C2-C3-H3A	117.7
C1-C3-H3A	117.7	C2-C3-H3B	117.7
C1-C3-H3B	117.7	H3A-C3-H3B	114.9
O1-C4-O2	122.2(4)	O1-C4-C1	131.6(4)
O2-C4-C1	106.2(3)	N1-C5-O2	115.0(3)
N1-C5-C6	120.5(3)	O2-C5-C6	124.4(3)
C11-C6-C7	123.5(4)	C11-C6-C5	124.7(4)
C7-C6-C5	111.9(3)	C8-C7-C6	115.4(3)
C8-C7-Ir1	128.9(3)	C6-C7-Ir1	115.7(3)
C7-C8-C9	122.3(4)	C7-C8-H8	118.8
C9-C8-H8	118.8	C10-C9-C8	119.6(4)
C10-C9-C12	120.7(4)	C8-C9-C12	119.7(4)
C11-C10-C9	120.7(4)	C11-C10-H10	119.6
C9-C10-H10	119.6	C10-C11-C6	118.3(4)
C10-C11-H11	120.8	C6-C11-H11	120.8
C9-C12-H12A	109.5	C9-C12-H12B	109.5
H12A-C12-H12B	109.5	C9-C12-H12C	109.5
H12A-C12-H12C	109.5	H12B-C12-H12C	109.5
C17-C13-C14	107.7(3)	C17-C13-C18	127.2(3)
C14-C13-C18	125.0(3)	C17-C13-Ir1	71.04(19)
C14-C13-Ir1	71.66(19)	C18-C13-Ir1	126.1(2)
C13-C14-C15	108.5(3)	C13-C14-C19	124.6(3)
C15-C14-C19	126.7(3)	C13-C14-Ir1	69.36(19)
C15-C14-Ir1	74.1(2)	C19-C14-Ir1	126.7(2)
C16-C15-C14	107.1(3)	C16-C15-C20	125.3(3)
C14-C15-C20	127.3(4)	C16-C15-Ir1	70.3(2)
C14-C15-Ir1	67.79(18)	C20-C15-Ir1	131.6(3)
C15-C16-C17	109.6(3)	C15-C16-C21	125.6(3)
C17-C16-C21	124.9(4)	C15-C16-Ir1	72.87(19)
C17-C16-Ir1	68.34(19)	C21-C16-Ir1	125.4(3)
C13-C17-C16	107.2(3)	C13-C17-C22	126.9(3)
C16-C17-C22	125.3(3)	C13-C17-Ir1	69.85(18)
C16-C17-Ir1	73.3(2)	C22-C17-Ir1	128.9(3)
C13-C18-H18A	109.5	C13-C18-H18B	109.5
H18A-C18-H18B	109.5	C13-C18-H18C	109.5
H18A-C18-H18C	109.5	H18B-C18-H18C	109.5

C14-C19-H19A	109.5	C14-C19-H19B	109.5
H19A-C19-H19B	109.5	C14-C19-H19C	109.5
H19A-C19-H19C	109.5	H19B-C19-H19C	109.5
C15-C20-H20A	109.5	C15-C20-H20B	109.5
H20A-C20-H20B	109.5	C15-C20-H20C	109.5
H20A-C20-H20C	109.5	H20B-C20-H20C	109.5
C16-C21-H21A	109.5	C16-C21-H21B	109.5
H21A-C21-H21B	109.5	C16-C21-H21C	109.5
H21A-C21-H21C	109.5	H21B-C21-H21C	109.5
C17-C22-H22A	109.5	C17-C22-H22B	109.5
H22A-C22-H22B	109.5	C17-C22-H22C	109.5
H22A-C22-H22C	109.5	H22B-C22-H22C	109.5
O4-C23-O3	130.8(4)	O4-C23-C24	117.3(4)
O3-C23-C24	111.8(3)	F3-C24-F2	104.3(4)
F3-C24-F1	122.6(8)	F2-C24-F1	92.1(7)
F3-C24-F1*	96.7(6)	F2-C24-F1*	112.7(5)
F3-C24-C23	112.2(3)	F2-C24-C23	113.6(3)
F1-C24-C23	110.2(5)	F1*-C24-C23	115.5(4)
C5-N1-C1	106.9(3)	C5-N1-Ir1	114.3(2)
C1-N1-Ir1	138.4(2)	C5-O2-C4	106.4(3)
C23-O3-Ir1	124.7(3)	C7-Ir1-N1	77.27(13)
C7-Ir1-O3	91.36(12)	N1-Ir1-O3	80.53(10)
C7-Ir1-C13	95.89(13)	N1-Ir1-C13	125.14(12)
O3-Ir1-C13	154.28(11)	C7-Ir1-C17	103.23(14)
N1-Ir1-C17	164.16(12)	O3-Ir1-C17	115.18(12)
C13-Ir1-C17	39.11(12)	C7-Ir1-C14	123.69(13)
N1-Ir1-C14	101.34(12)	O3-Ir1-C14	144.66(12)
C13-Ir1-C14	38.97(13)	C17-Ir1-C14	64.91(13)
C7-Ir1-C16	138.66(14)	N1-Ir1-C16	144.05(12)
O3-Ir1-C16	94.51(12)	C13-Ir1-C16	64.16(13)
C17-Ir1-C16	38.39(13)	C14-Ir1-C16	63.18(13)
C7-Ir1-C15	159.95(13)	N1-Ir1-C15	110.85(12)
O3-Ir1-C15	107.85(12)	C13-Ir1-C15	64.28(13)
C17-Ir1-C15	63.80(13)	C14-Ir1-C15	38.09(13)
C16-Ir1-C15	36.84(13)		

Table S15. Anisotropic atomic displacement parameters (Å^2) for iridacycle-1a.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0411(18)	0.0399(18)	0.0383(17)	0.0055(14)	0.0048(14)	0.0079(14)
C2	0.088(3)	0.064(3)	0.042(2)	-0.004(2)	-0.009(2)	0.017(3)
C3	0.055(2)	0.060(3)	0.070(3)	0.009(2)	-0.012(2)	0.000(2)
C4	0.045(2)	0.045(2)	0.054(2)	0.0105(17)	0.0168(17)	0.0103(16)
C5	0.0459(19)	0.0306(16)	0.0388(17)	0.0030(13)	0.0143(14)	0.0033(13)
C6	0.0427(18)	0.0370(17)	0.0431(18)	-0.0054(14)	0.0118(15)	-0.0080(14)
C7	0.0409(18)	0.0376(17)	0.0365(16)	-0.0048(13)	0.0082(13)	-0.0062(14)
C8	0.051(2)	0.049(2)	0.045(2)	-0.0019(16)	-0.0032(16)	-0.0111(17)
C9	0.046(2)	0.070(3)	0.0416(19)	-0.0082(18)	0.0105(16)	-0.0210(19)
C10	0.067(3)	0.057(3)	0.060(3)	-0.016(2)	0.014(2)	-0.031(2)
C11	0.061(2)	0.0380(19)	0.062(2)	-0.0071(17)	0.016(2)	-0.0161(17)
C12	0.058(3)	0.102(4)	0.052(2)	-0.009(2)	0.000(2)	-0.033(3)
C13	0.0435(18)	0.0320(15)	0.0331(15)	0.0020(12)	0.0072(13)	0.0033(13)
C14	0.0384(17)	0.0297(15)	0.0436(18)	0.0021(13)	0.0046(14)	-0.0013(13)
C15	0.0478(19)	0.0301(16)	0.0418(18)	-0.0017(13)	0.0009(15)	-0.0043(14)
C16	0.053(2)	0.0283(15)	0.0419(18)	-0.0006(13)	0.0062(15)	0.0001(14)
C17	0.0459(19)	0.0325(16)	0.0405(17)	0.0034(13)	0.0037(14)	0.0052(14)
C18	0.066(3)	0.047(2)	0.0372(18)	-0.0048(16)	0.0091(17)	-0.0015(18)
C19	0.041(2)	0.047(2)	0.066(3)	0.0004(18)	0.0088(18)	0.0002(16)
C20	0.072(3)	0.045(2)	0.057(2)	-0.0088(18)	-0.011(2)	-0.013(2)
C21	0.077(3)	0.037(2)	0.063(3)	-0.0115(18)	0.020(2)	0.0079(19)
C22	0.054(2)	0.051(2)	0.055(2)	0.0091(18)	-0.0045(18)	0.0117(18)
C23	0.047(2)	0.050(2)	0.046(2)	-0.0052(17)	-0.0016(16)	-0.0014(17)

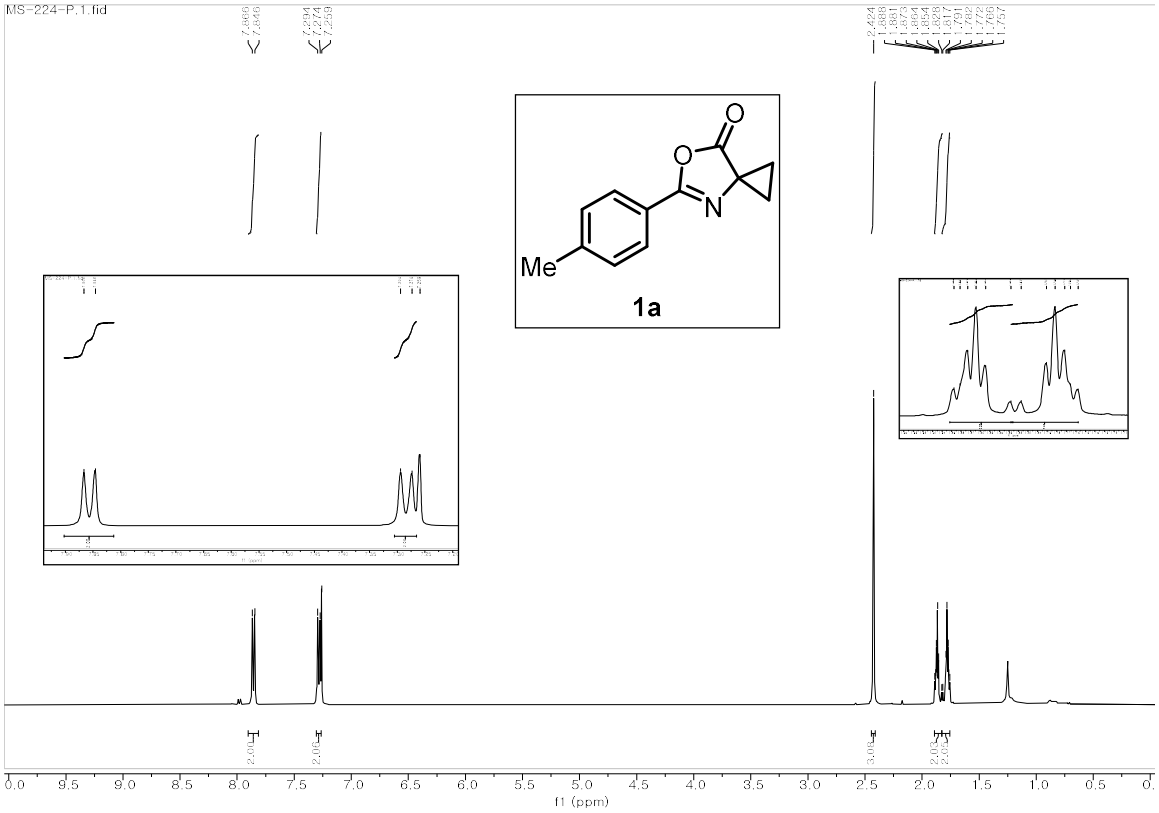
C24	0.047(2)	0.048(2)	0.0454(19)	-0.0022(16)	0.0086(16)	0.0048(17)
F1	0.076(4)	0.073(4)	0.073(4)	0.0049(18)	0.0130(19)	-0.0064(18)
F1*	0.073(3)	0.072(3)	0.064(3)	0.0065(16)	0.0056(17)	0.0078(17)
F2	0.067(2)	0.184(4)	0.093(2)	-0.034(3)	0.0129(18)	0.037(2)
F3	0.151(4)	0.127(3)	0.075(2)	-0.049(2)	0.018(2)	-0.066(3)
N1	0.0399(15)	0.0304(13)	0.0368(14)	0.0006(11)	0.0056(11)	0.0038(11)
O1	0.0643(19)	0.0584(18)	0.080(2)	0.0307(16)	0.0086(16)	0.0199(15)
O2	0.0592(17)	0.0315(12)	0.0591(16)	0.0071(11)	0.0170(13)	0.0040(11)
O3	0.0485(15)	0.0476(15)	0.0489(14)	0.0019(12)	0.0148(12)	0.0087(12)
O4	0.0548(19)	0.135(4)	0.0550(19)	-0.009(2)	-0.0089(15)	0.021(2)
Ir1	0.03502(7)	0.02800(6)	0.03190(7)	-0.00241(5)	0.00222(4)	0.00109(5)

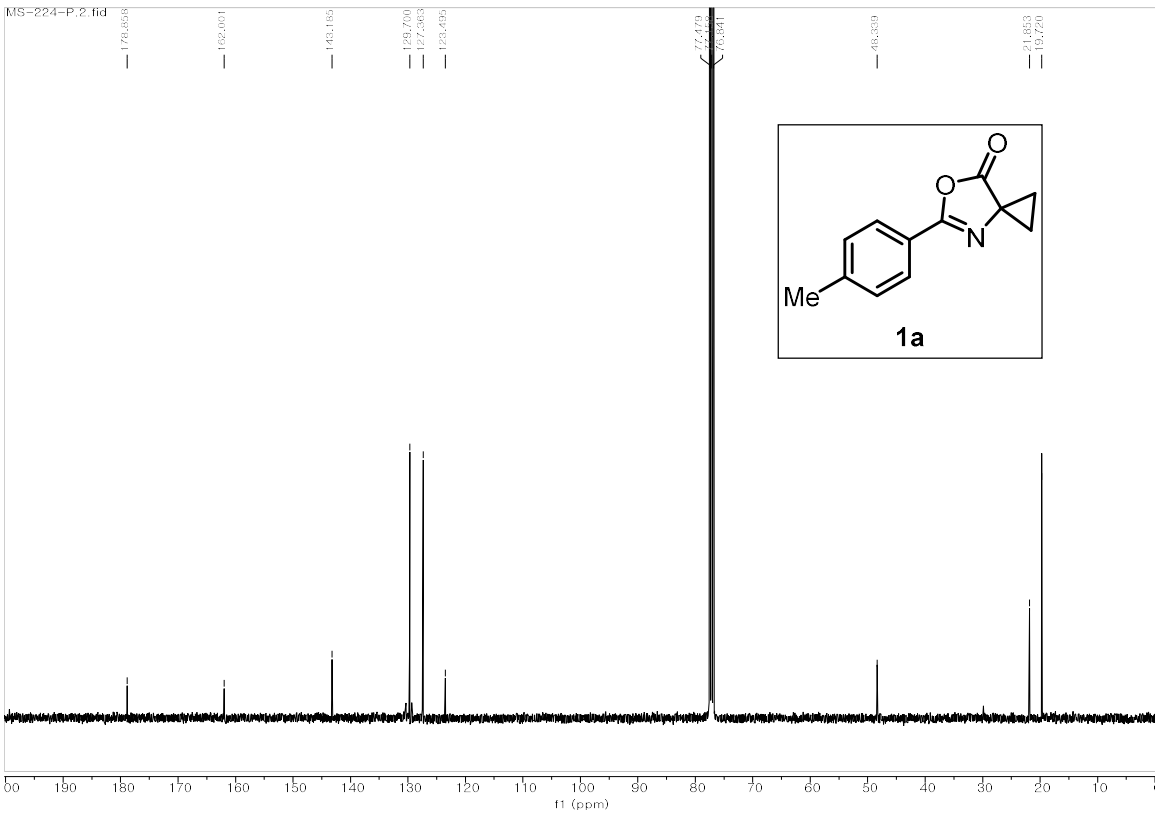
Table S16. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å^2) for iridacycle-1a.

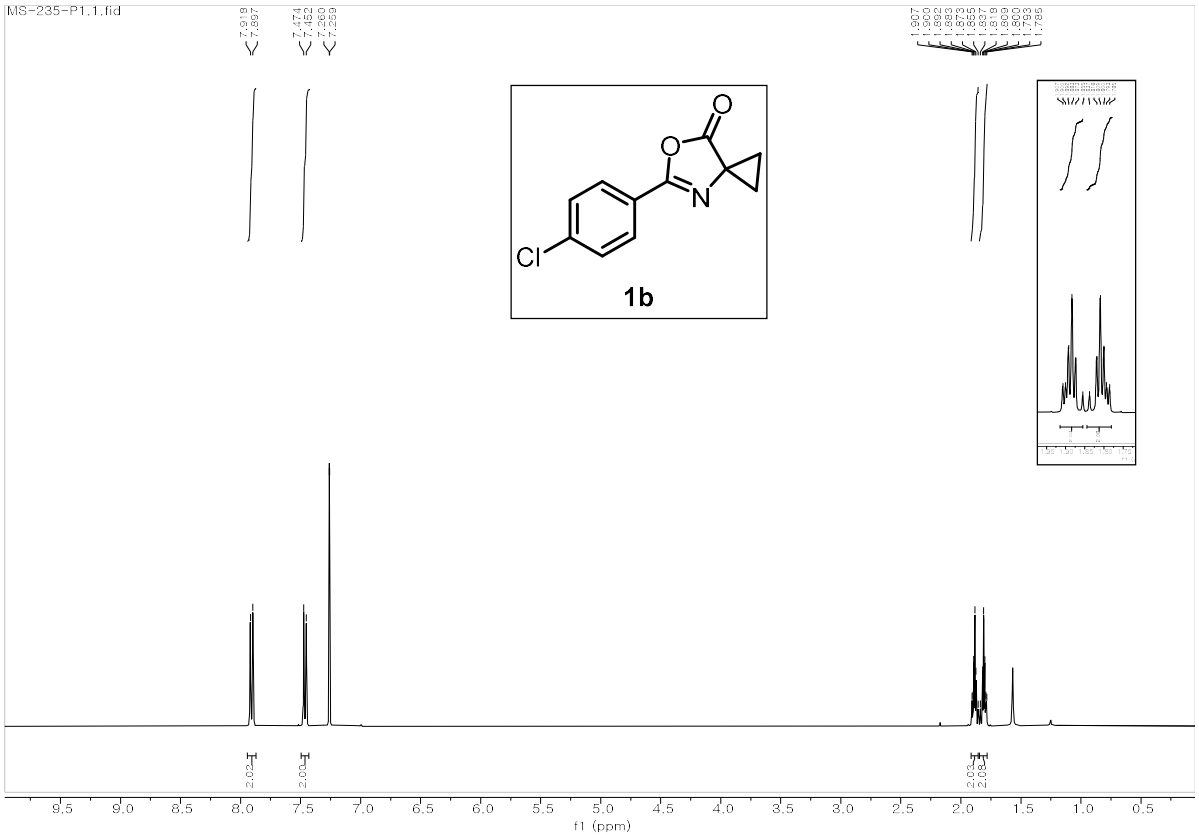
	x/a	y/b	z/c	U(eq)
H2A	0.4335	0.6704	-0.1164	0.078
H2B	0.4940	0.6231	-0.0215	0.078
H3A	0.2916	0.6327	0.1259	0.075
H3B	0.2310	0.6800	0.0309	0.075
H8	0.9389	0.6367	0.5451	0.058
H10	0.9555	0.7844	0.5829	0.073
H11	0.7679	0.7924	0.4054	0.064
H12A	1.1870	0.7116	0.6506	0.106
H12B	1.0963	0.6715	0.7281	0.106
H12C	1.0810	0.7276	0.7672	0.106
H18A	0.5624	0.5884	0.7324	0.074
H18B	0.5179	0.6376	0.6528	0.074
H18C	0.6869	0.6237	0.6819	0.074
H19A	0.2658	0.5942	0.5134	0.077
H19B	0.2507	0.6046	0.3505	0.077
H19C	0.3318	0.6430	0.4533	0.077
H20A	0.4395	0.5333	0.1011	0.088
H20B	0.3022	0.5518	0.1804	0.088
H20C	0.3617	0.4976	0.2038	0.088

H21A	0.6938	0.4622	0.2754	0.087
H21B	0.8277	0.4994	0.2730	0.087
H21C	0.6973	0.5017	0.1547	0.087
H22A	0.8601	0.5588	0.6316	0.081
H22B	0.9241	0.5426	0.4890	0.081
H22C	0.8391	0.5042	0.5779	0.081

^{1}H, ^{13}C and ^{19}F NMR spectra of all compounds



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Cl
1b
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2.03
2.08
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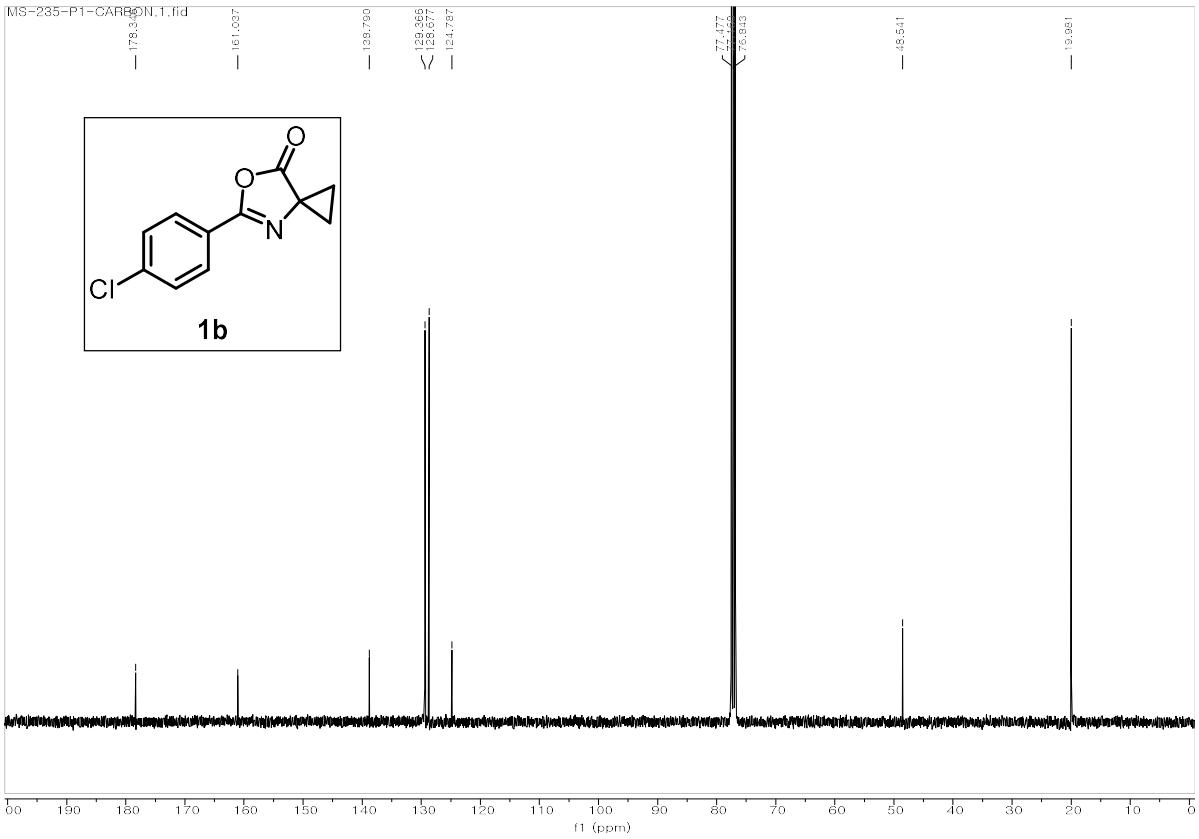
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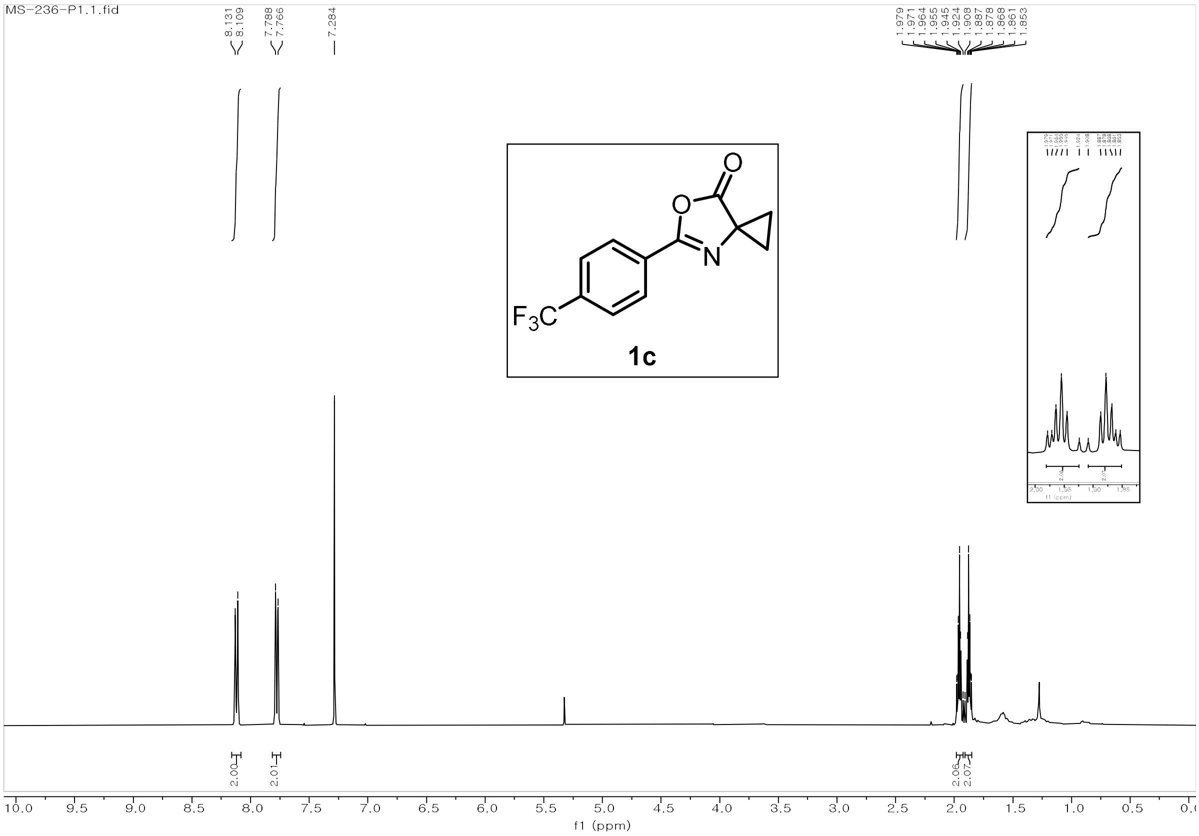
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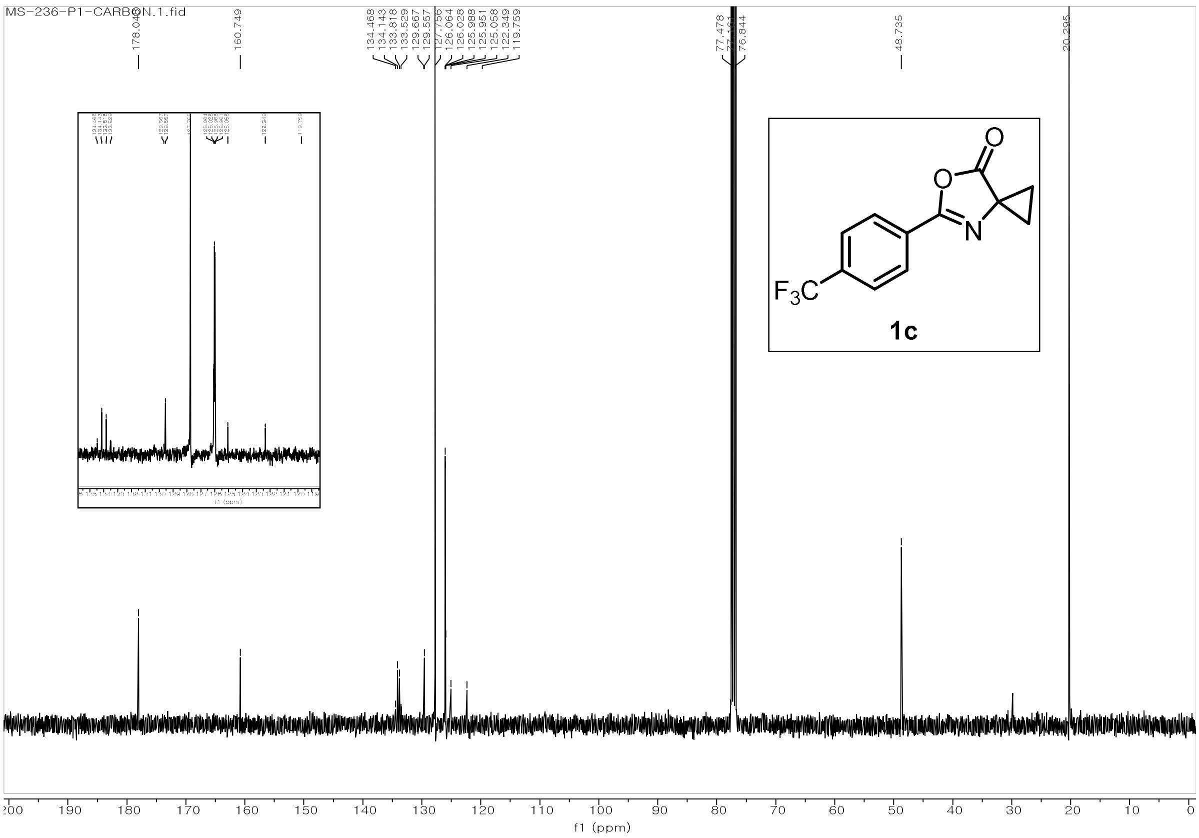
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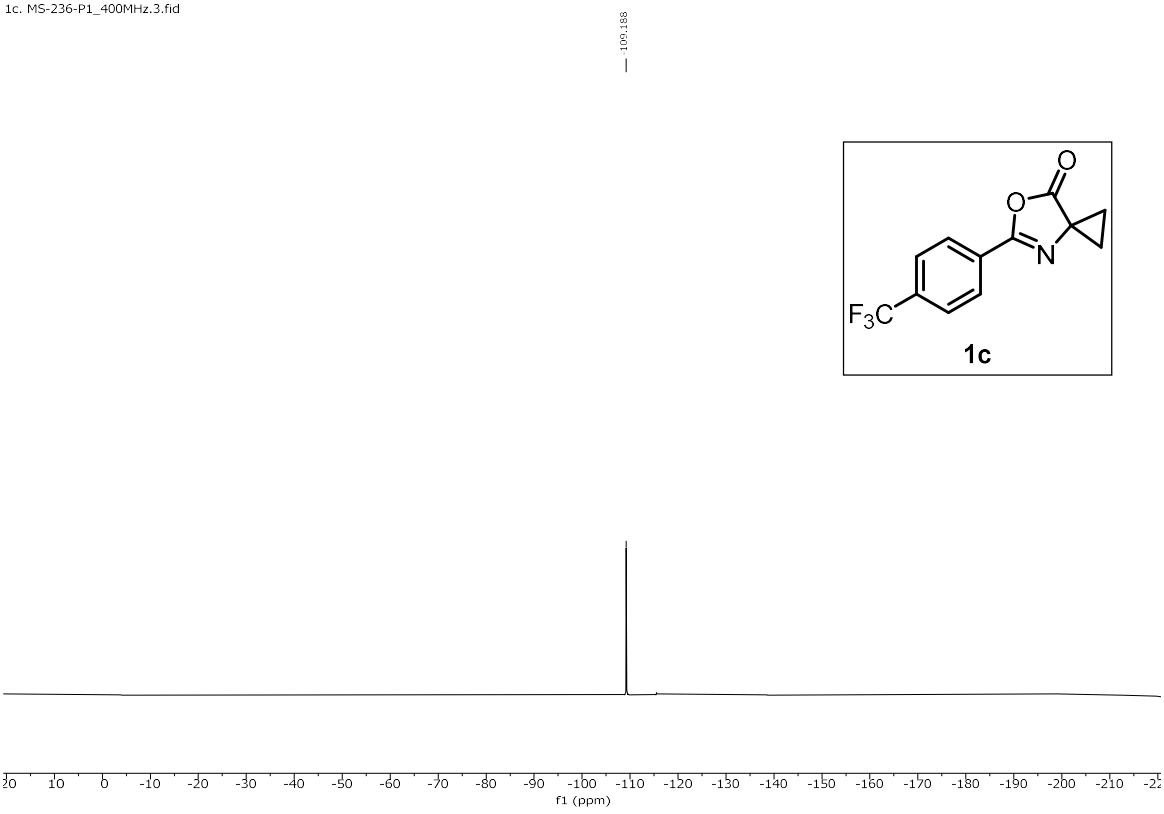

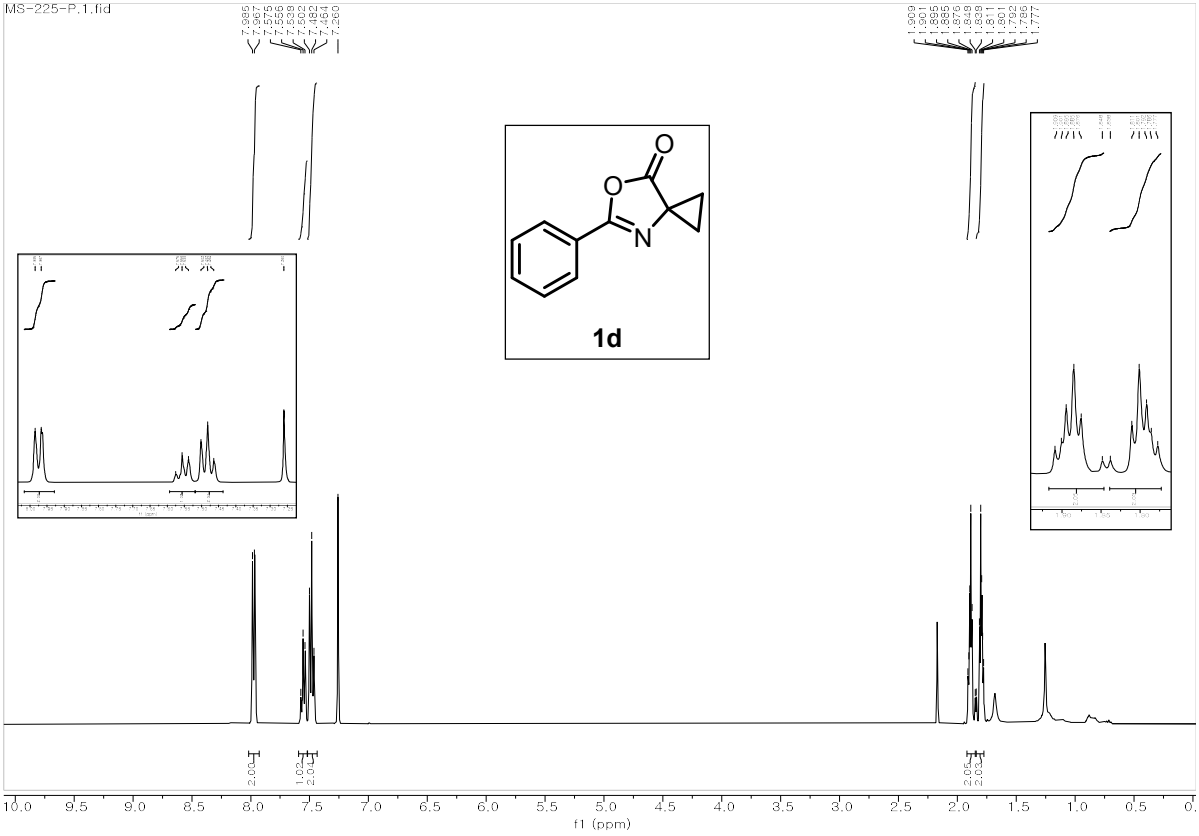
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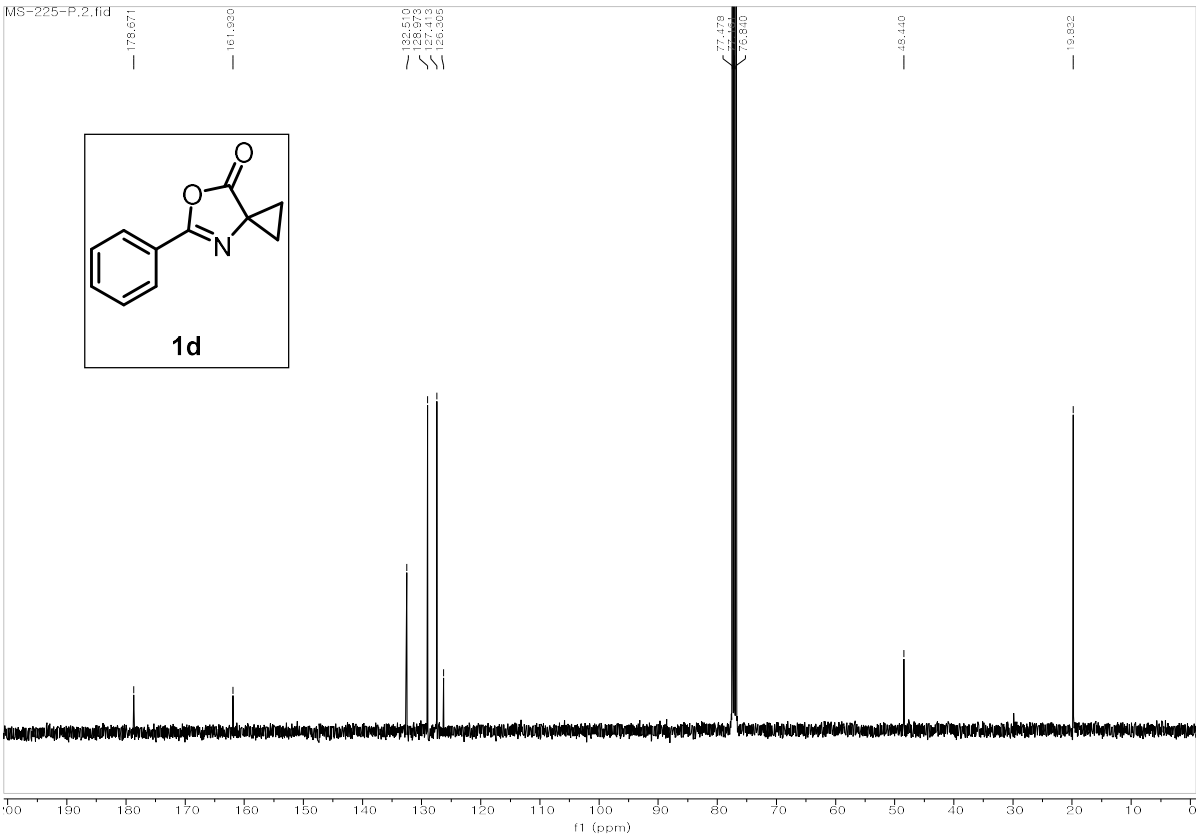
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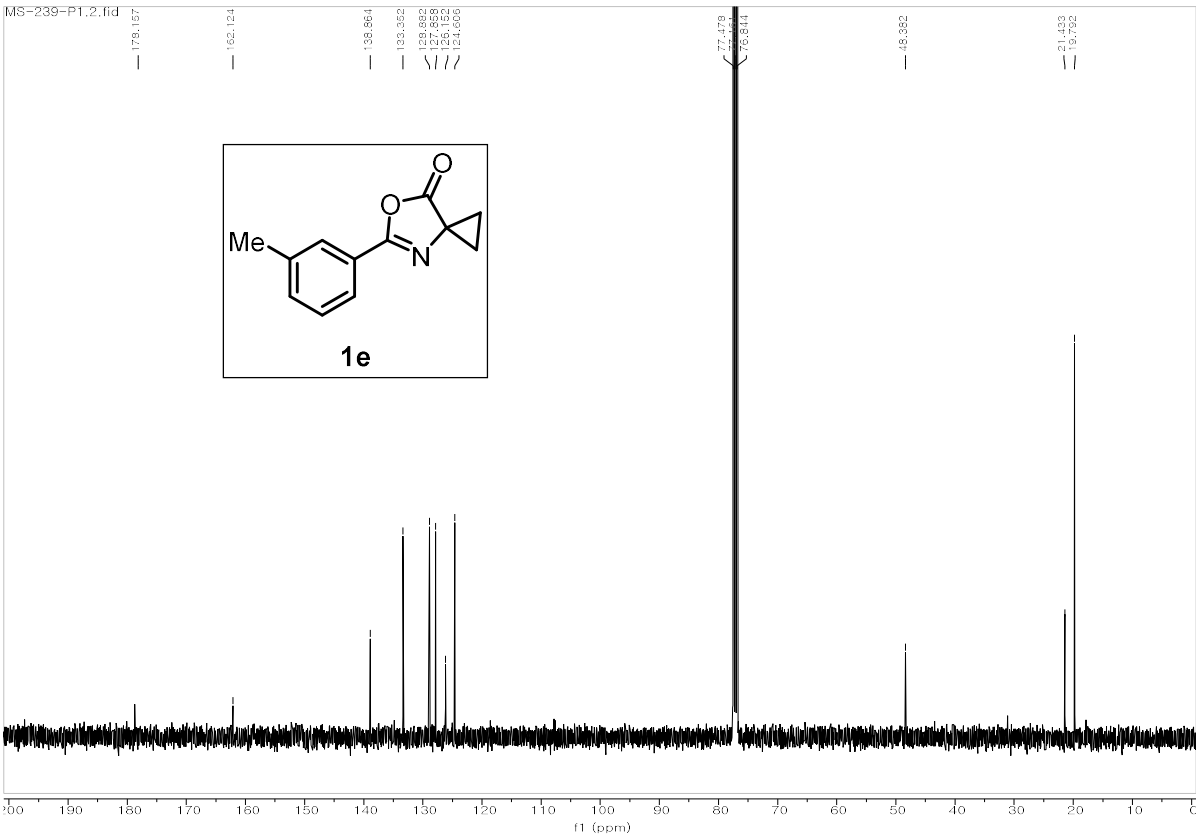
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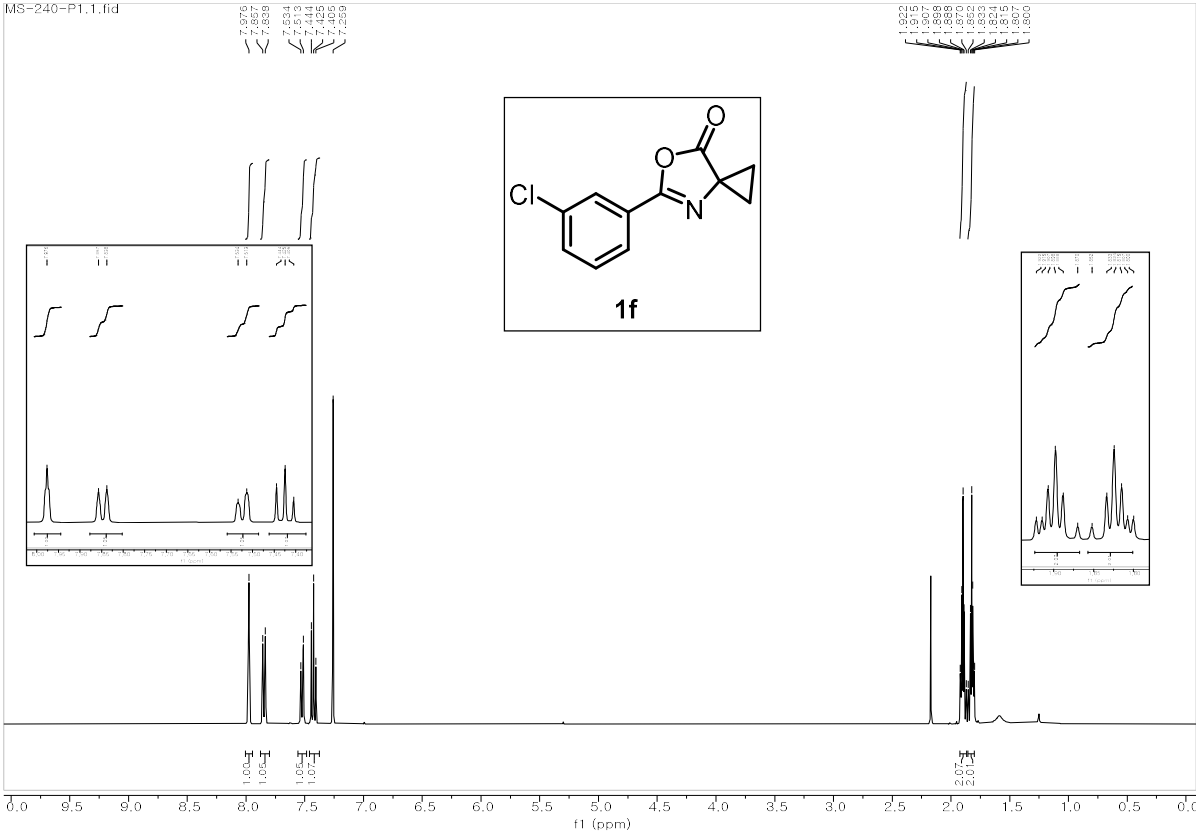
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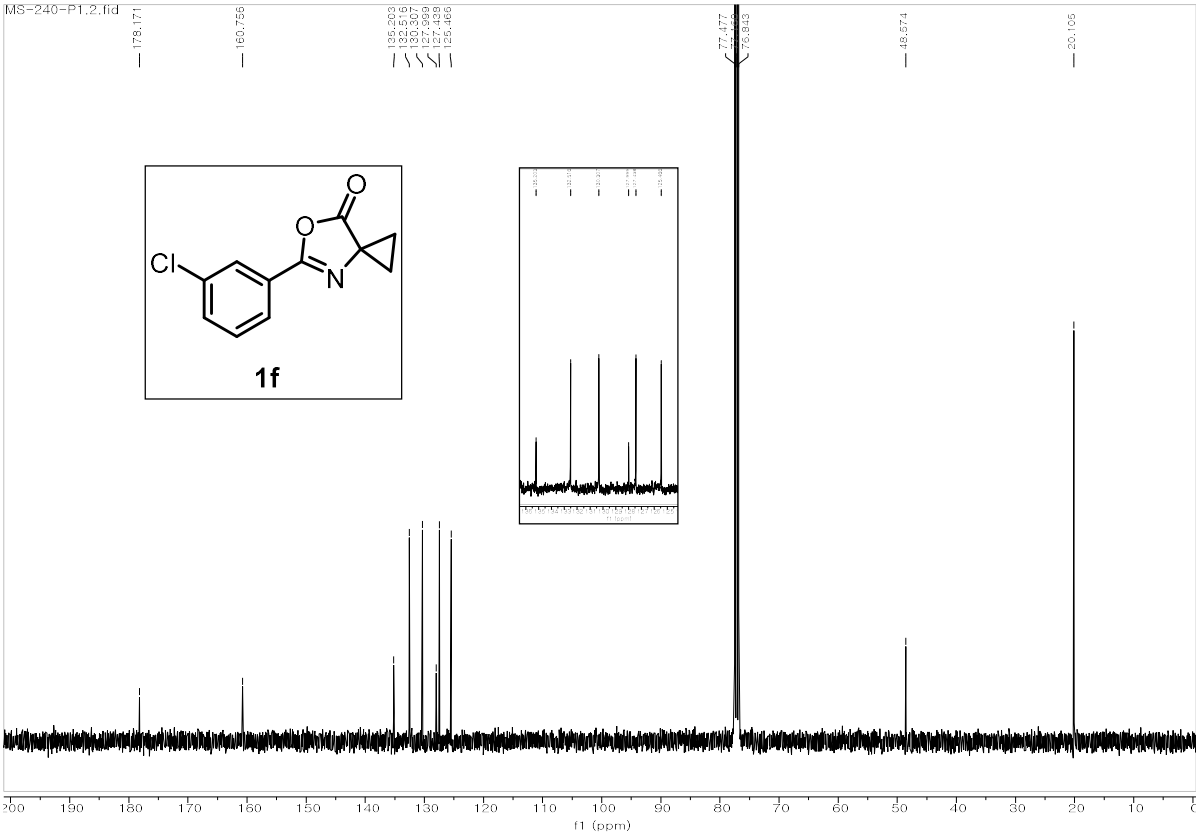
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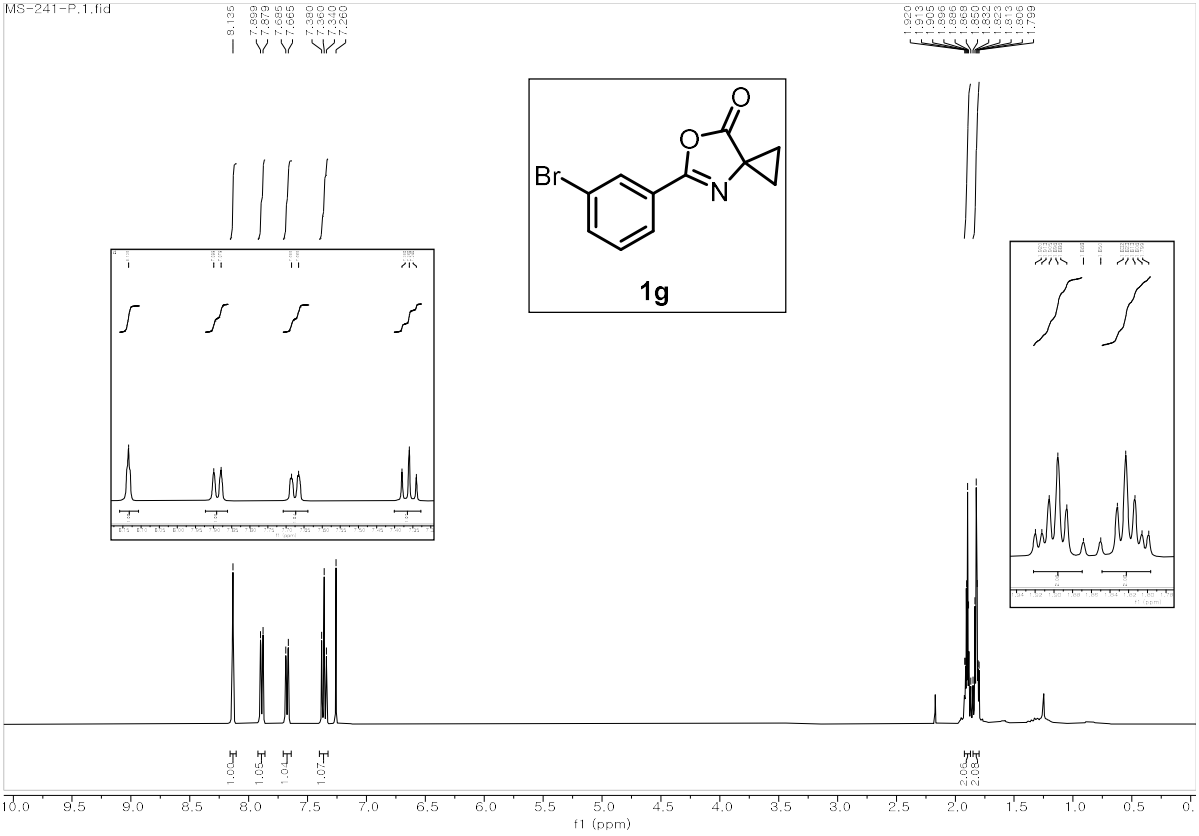
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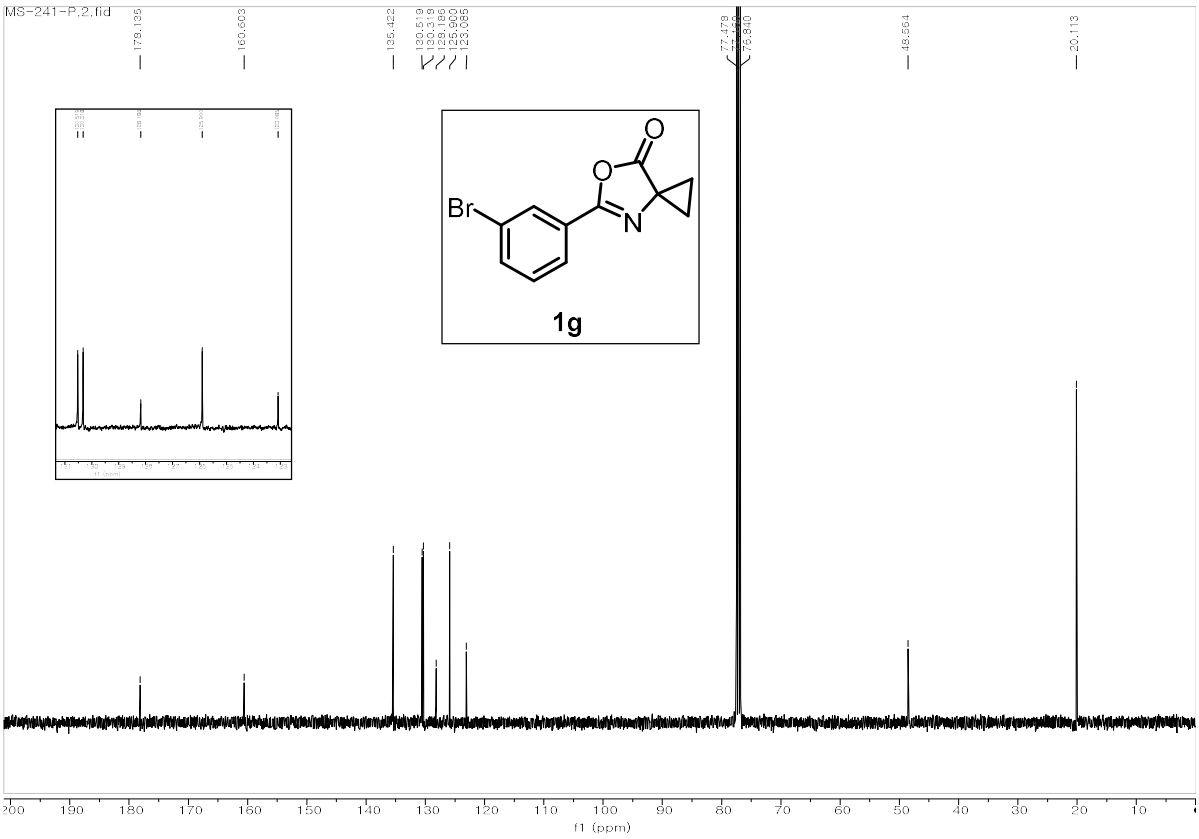
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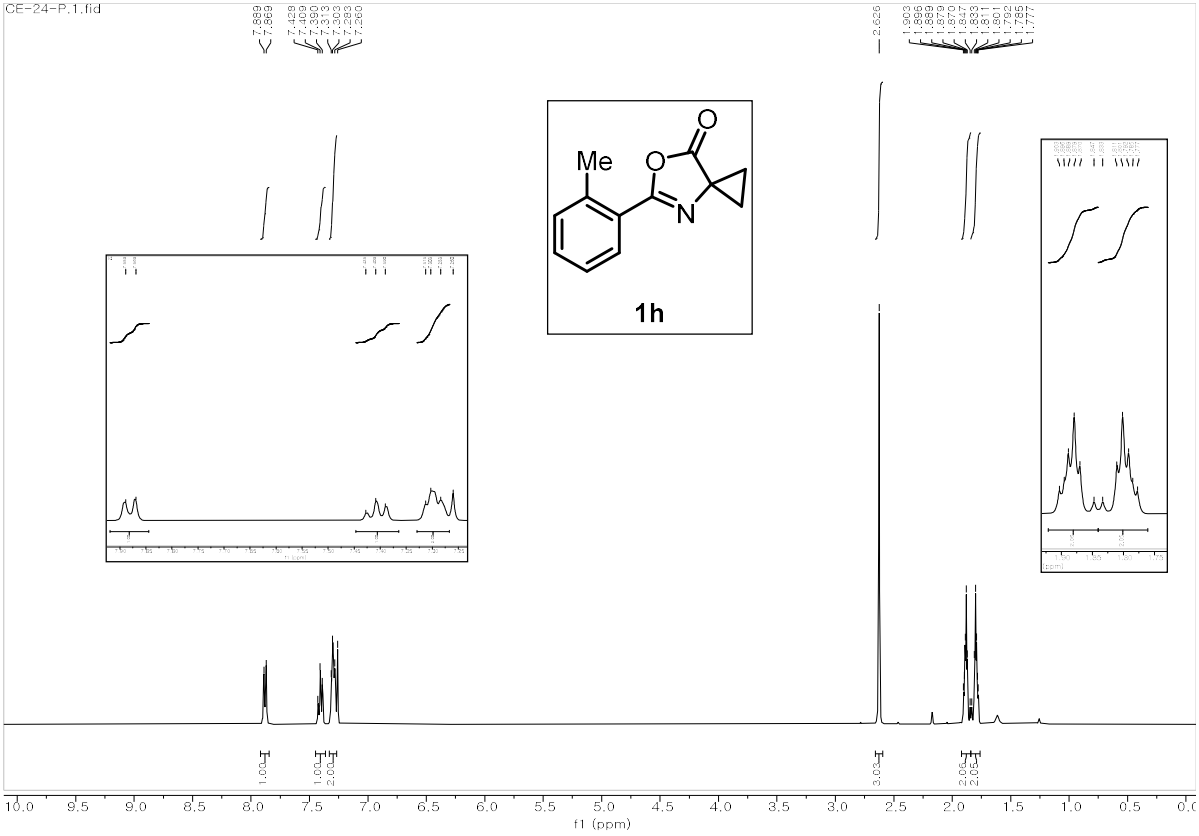
1g
f1 (ppm)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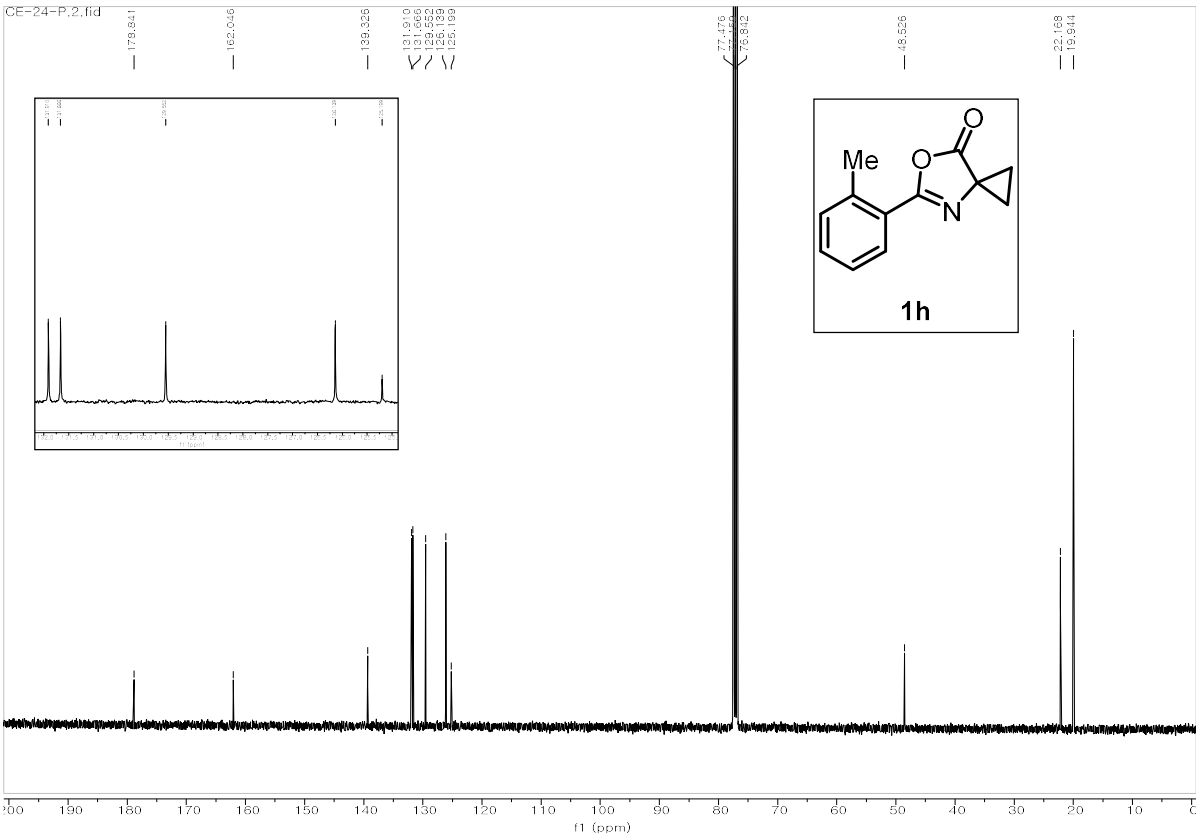
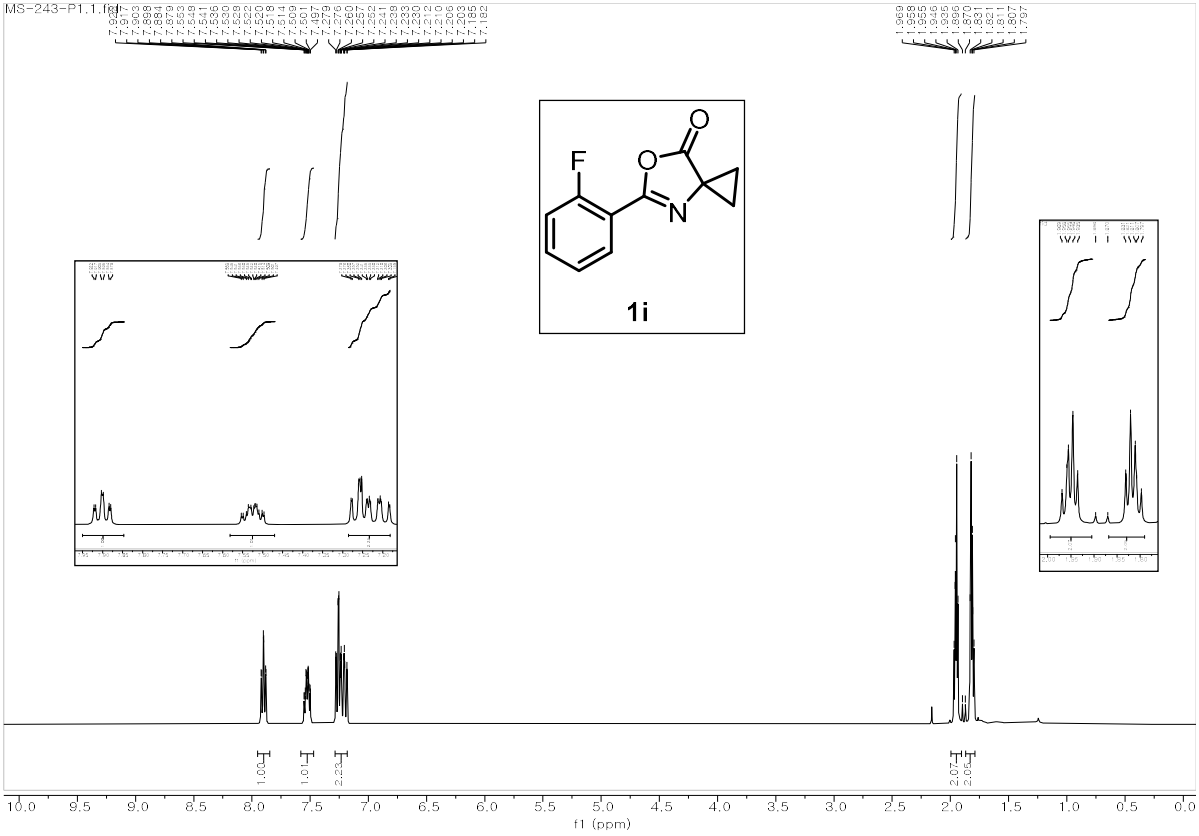
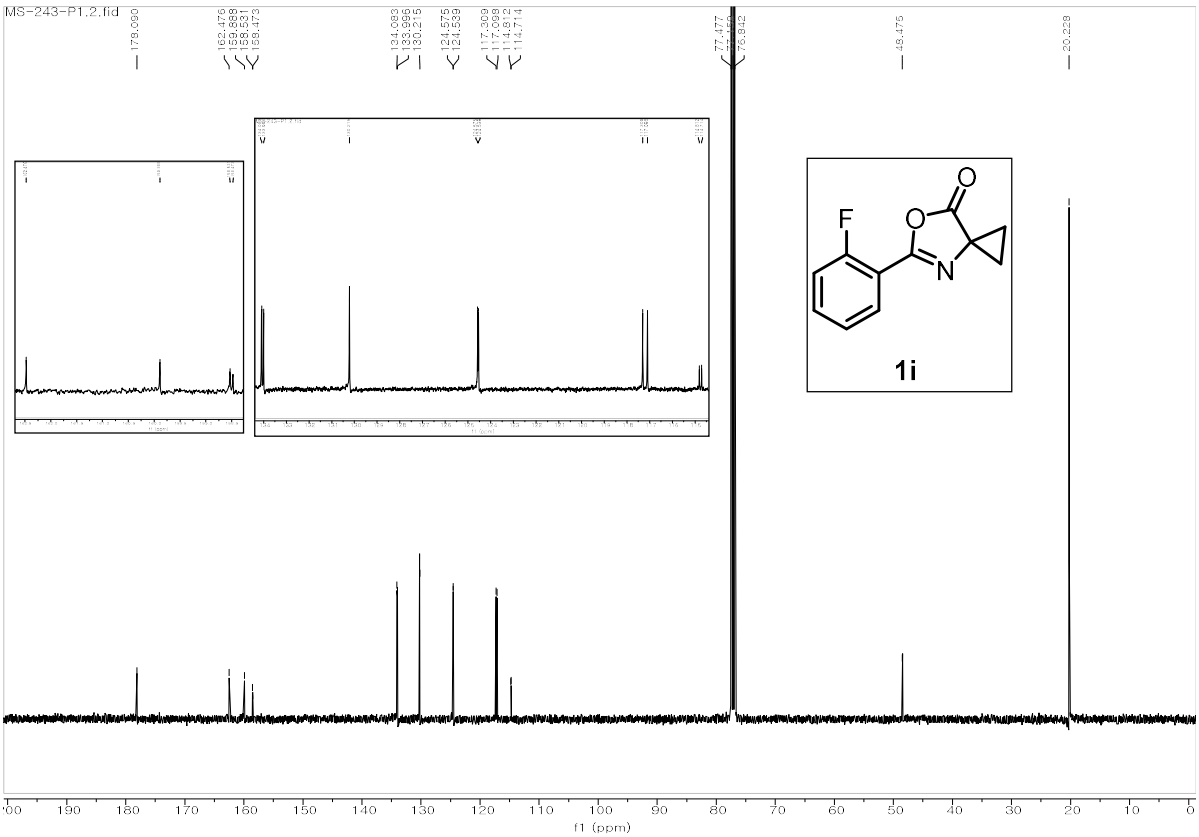
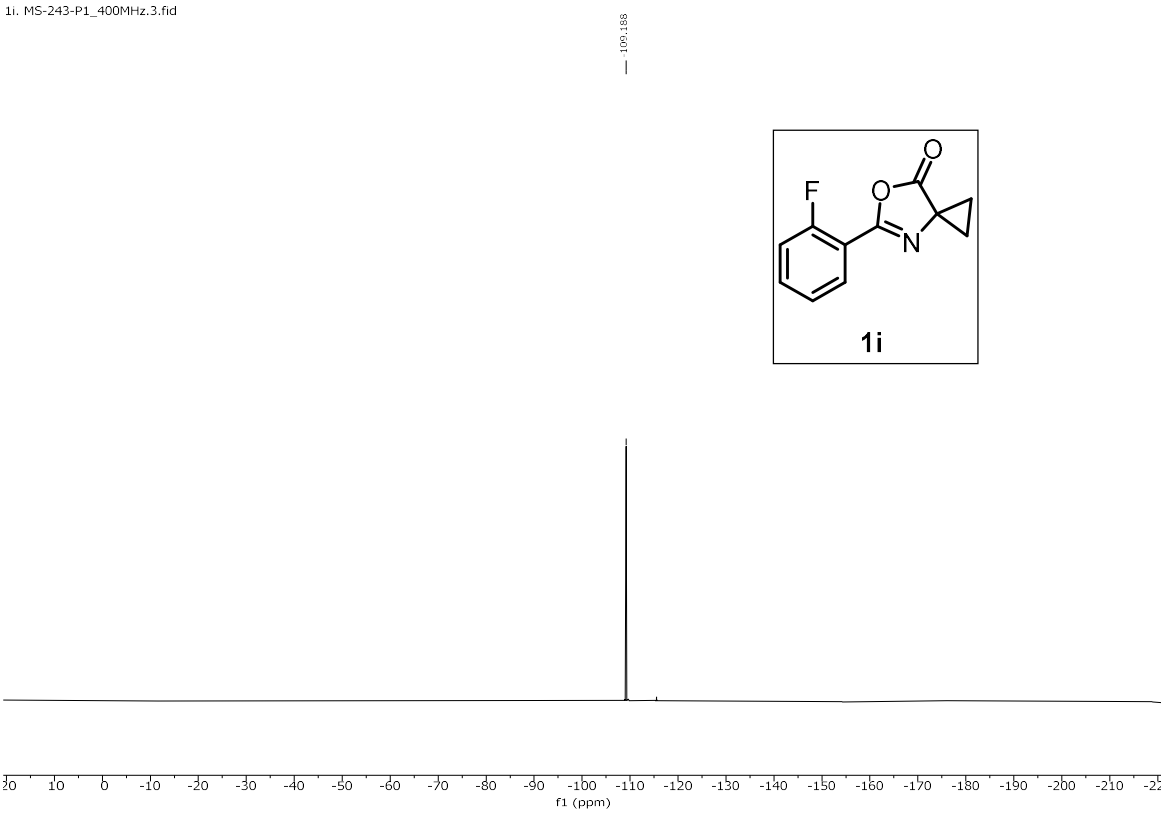
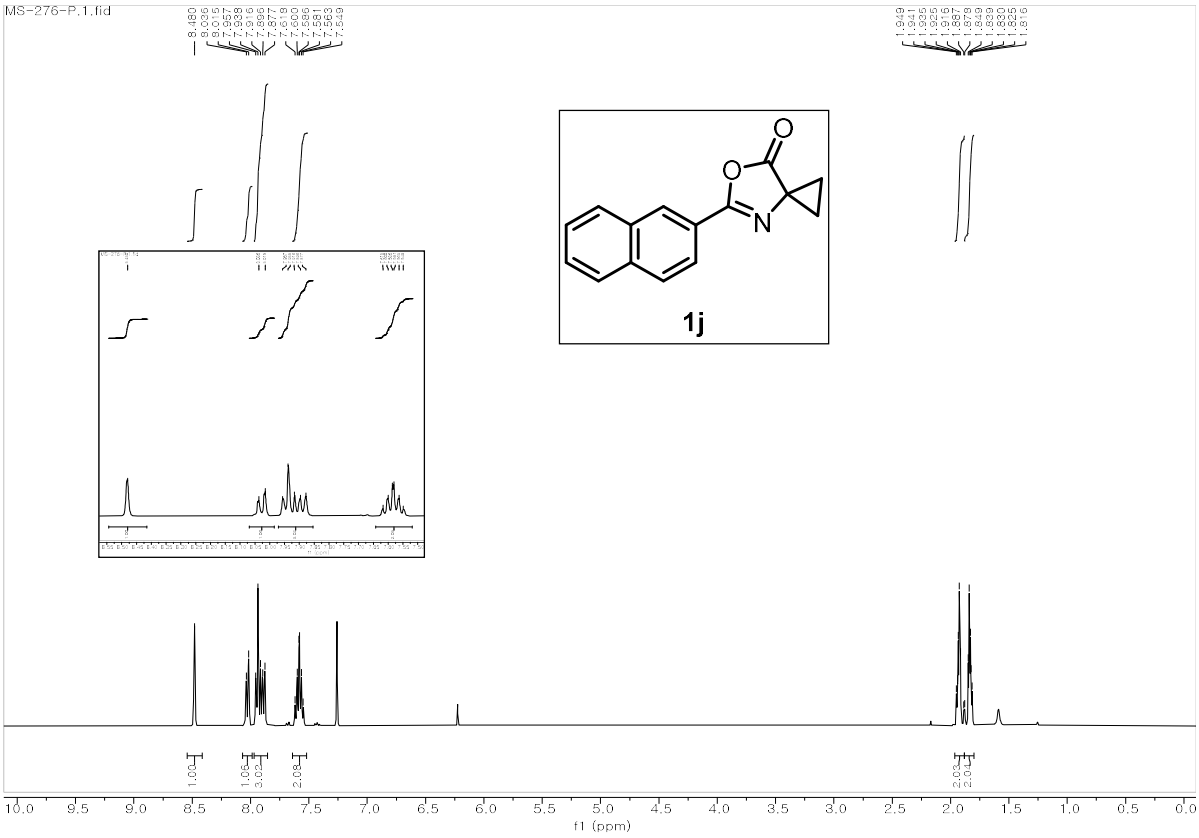
1.0
0.5
f1 (ppm)

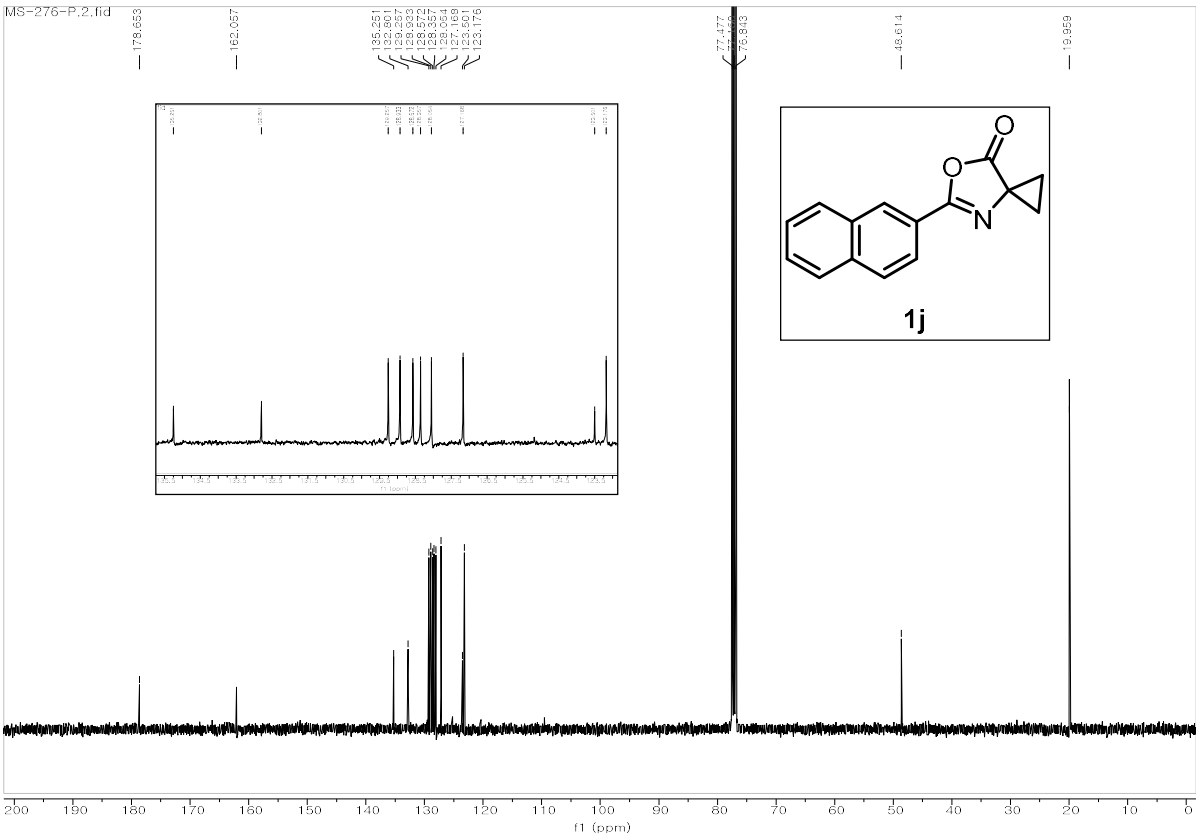

CE-24-P.2.fid
178.841
162.046
139.326
131.910
131.666
129.552
126.139
125.199
77.476
77.158
76.842
48.526
22.168
19.944
Me
O
O
N
1h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

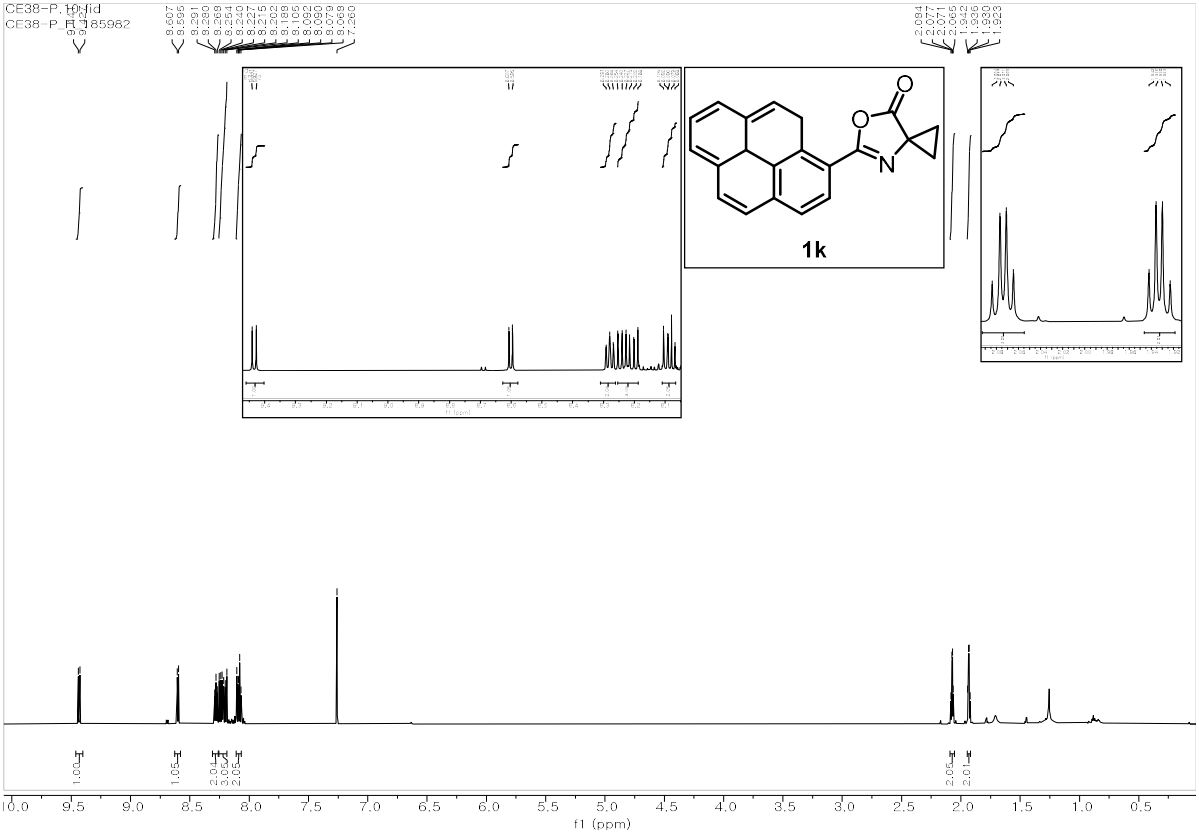

MS-243-P1.1.fid
F
O
O
N
1i
1.00
1.01
2.23
2.07
2.05
f1 (ppm)

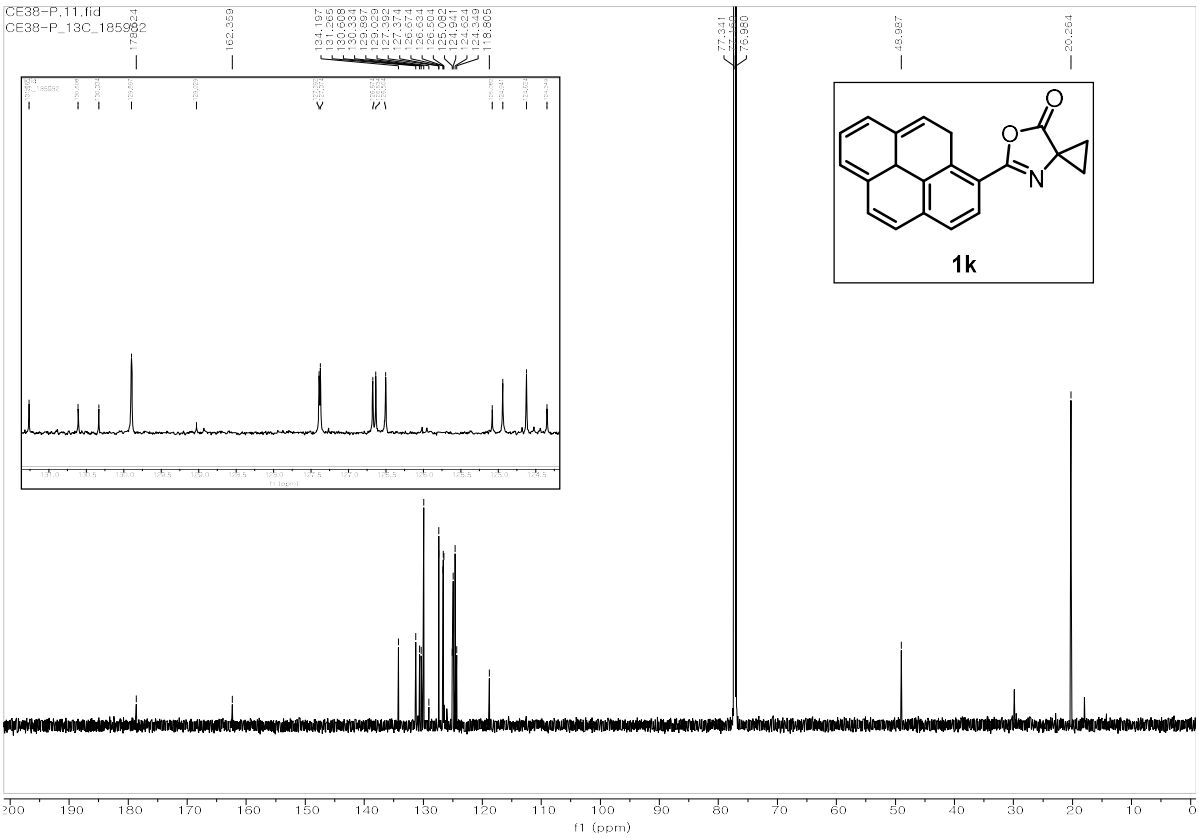

MS-243-P1.2.fid
179.090
162.476
159.888
158.531
158.473
134.088
133.986
130.215
124.575
124.539
117.309
117.099
114.912
114.714
77.477
76.842
48.475
20.228
F
O
O
N
1i
f1 (ppm)

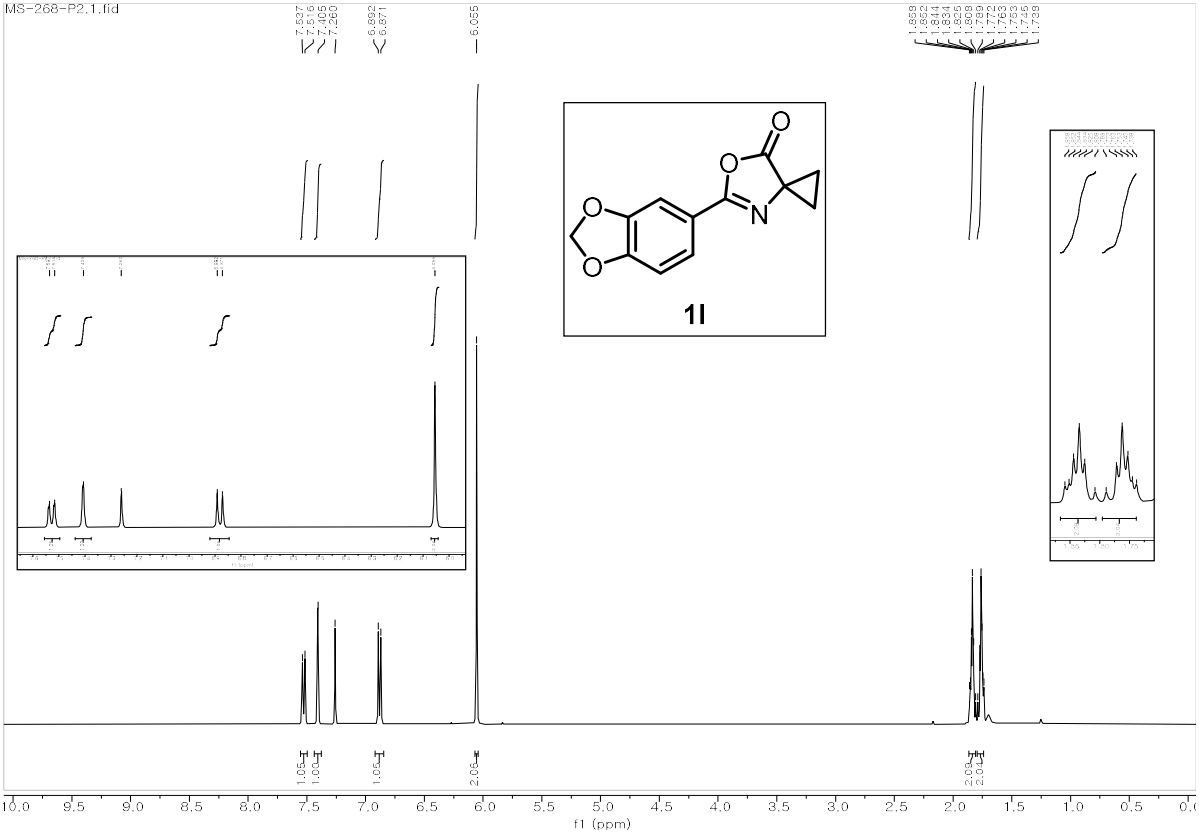

1i. MS-243-P1_400MHz.3.fid
109.188
F
O
O
N
1i
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

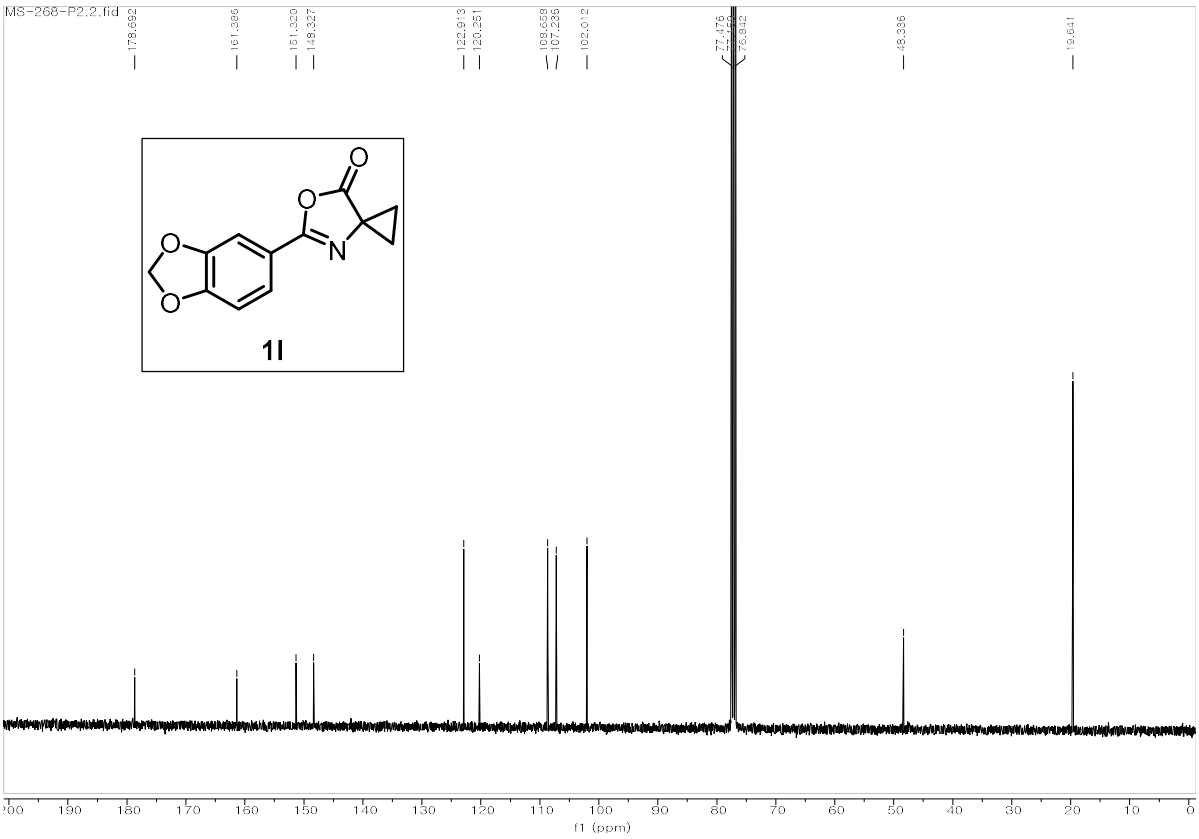

MS-276-P.1.fid
1j
f1 (ppm)

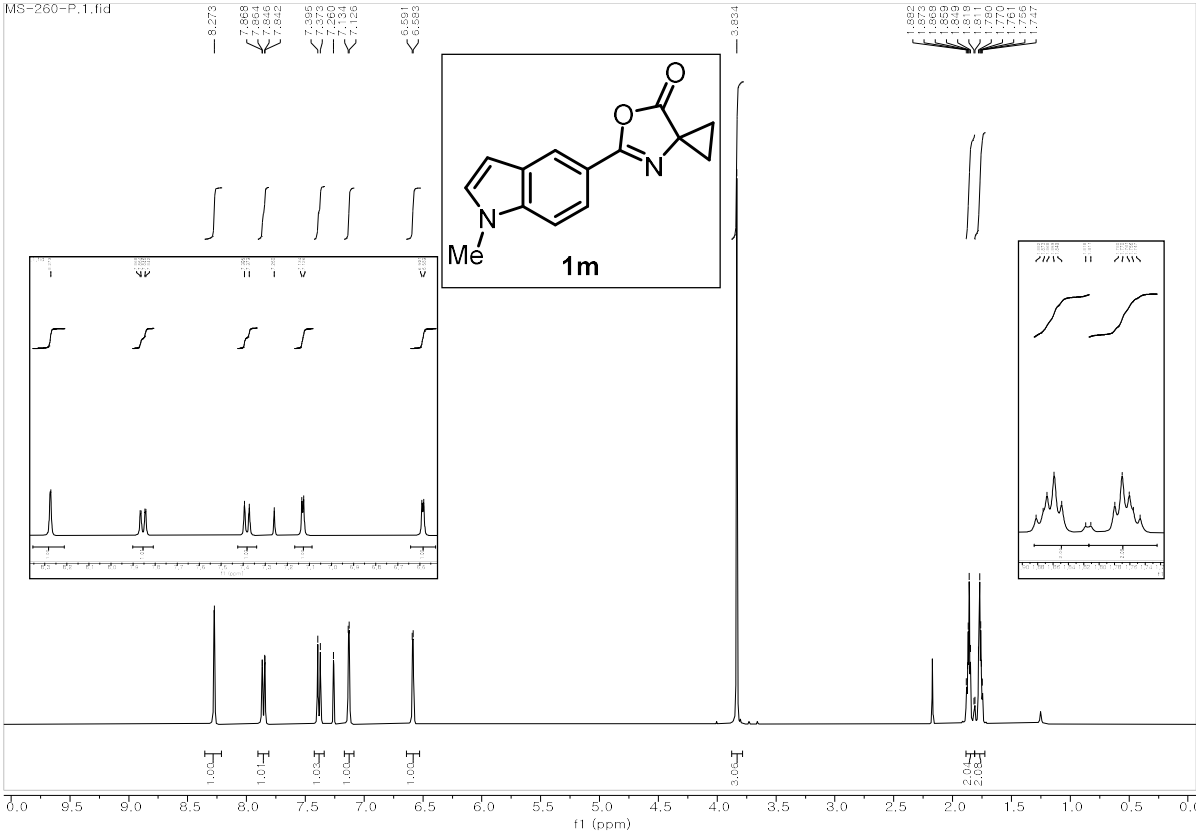

MS-276-P.2.fid
1j
f1 (ppm)

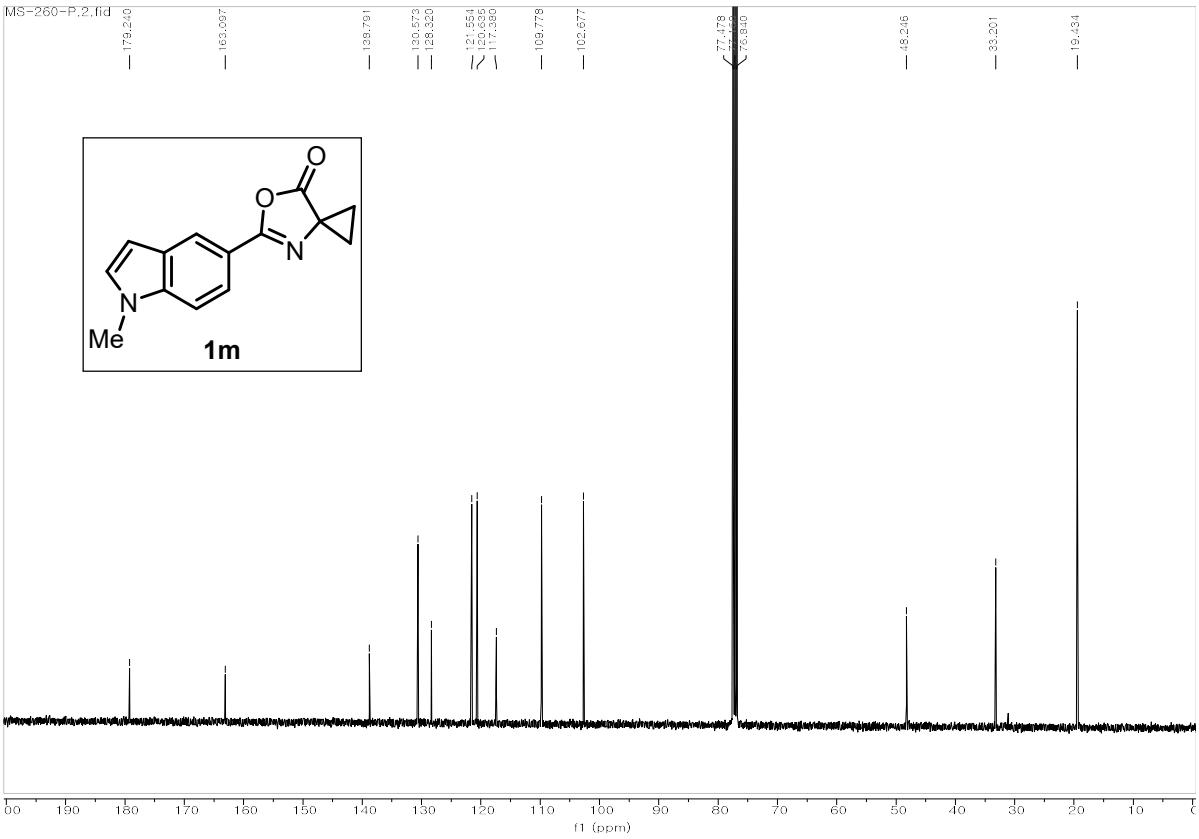

CE38-P.10.fid
CE38-P_H_185962
1k
f1 (ppm)

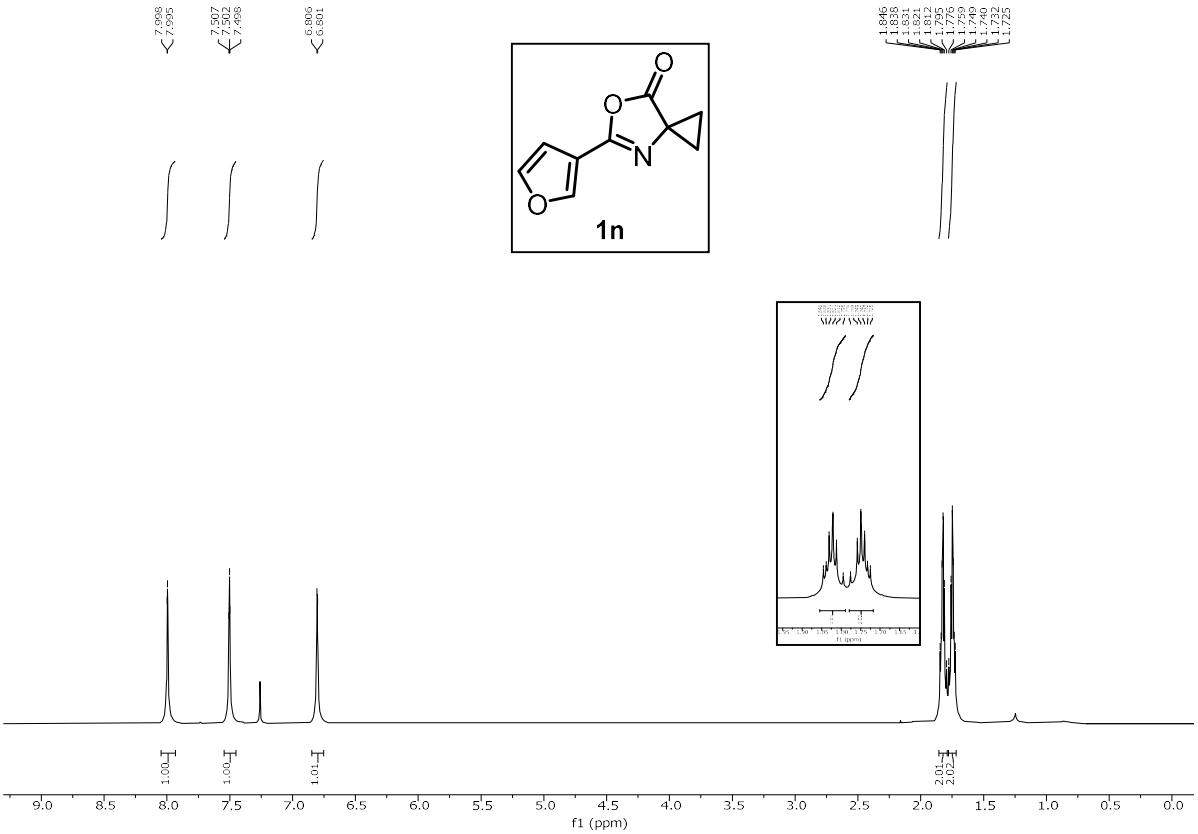

CE38-P.11.fid
CE38-P_13C_185962
1k
f1 (ppm)


MS-268-P2.1.fid
7.637
7.616
7.605
7.260
6.892
6.871
6.055
1.859
1.852
1.844
1.834
1.825
1.808
1.798
1.772
1.763
1.753
1.745
1.738
1l
f1 (ppm)

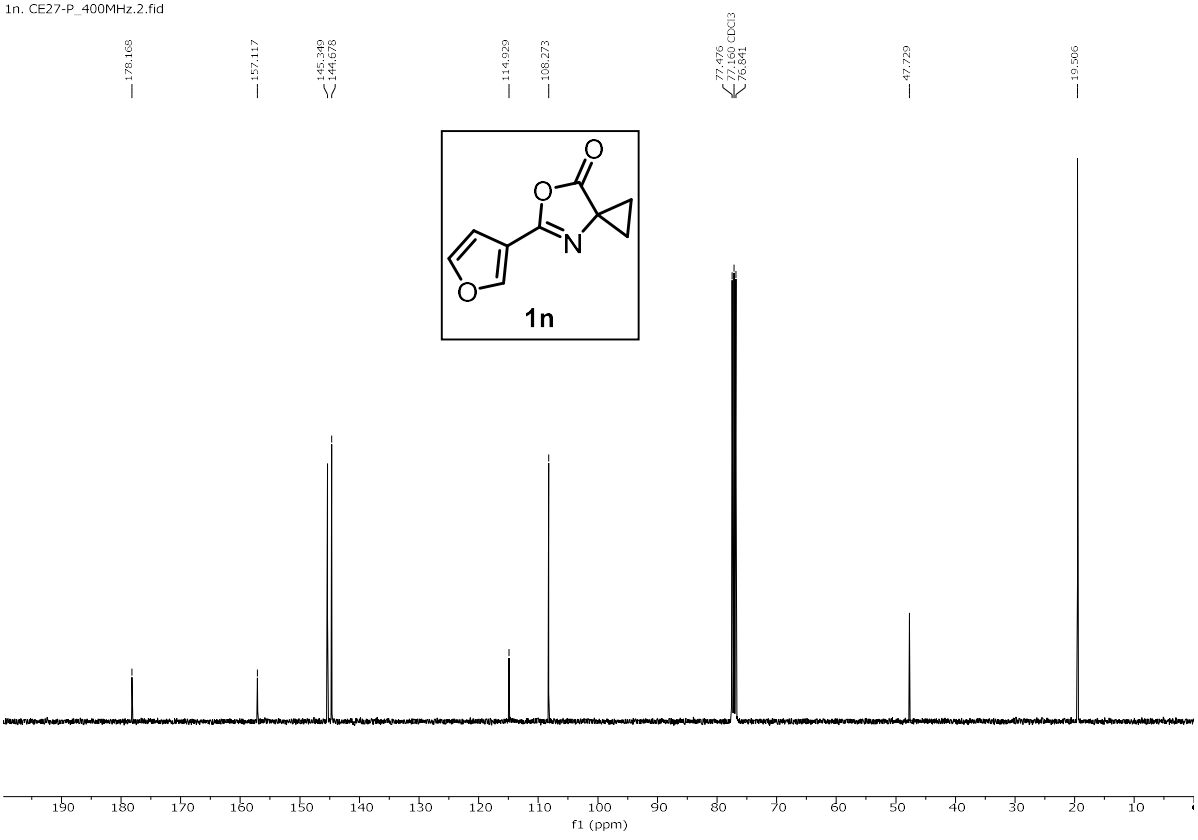

MS-268-P2.2.fid
178.692
161.386
151.320
148.327
122.913
120.251
109.659
107.236
102.012
77.476
76.842
48.336
19.641
1l
f1 (ppm)

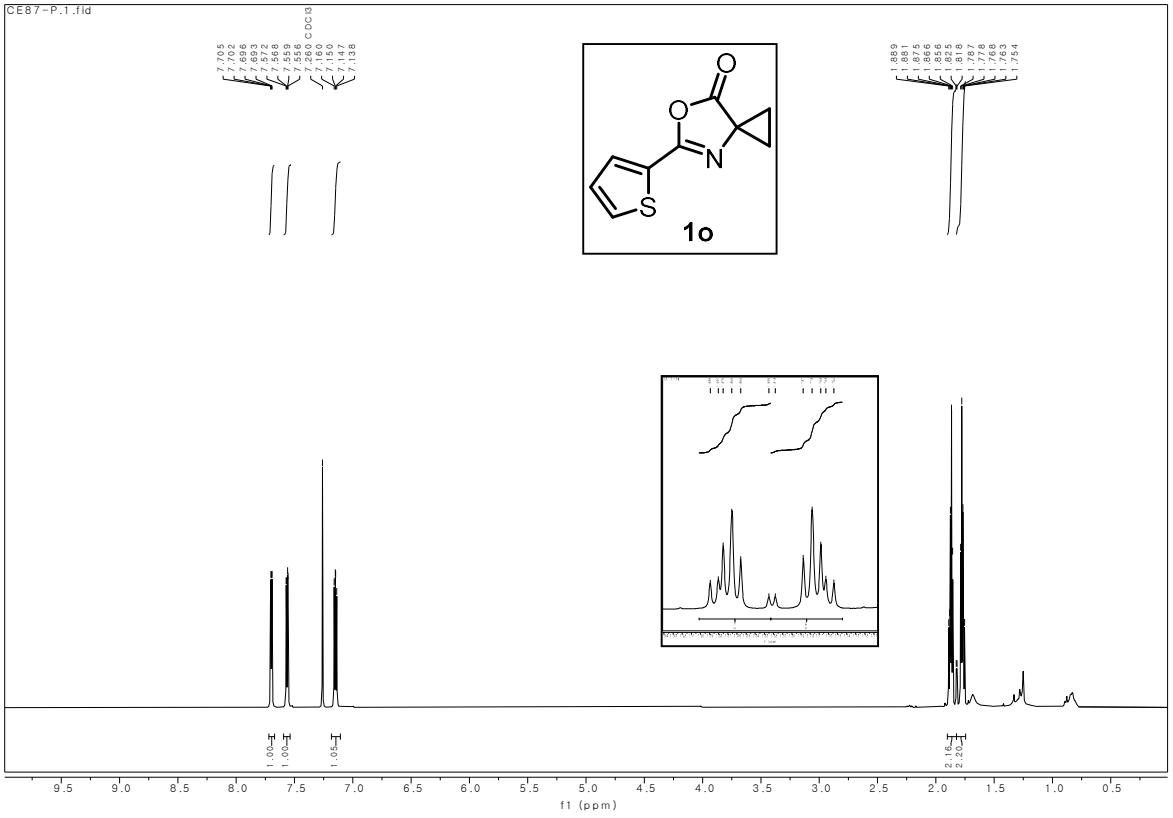

MS-260-P.1.fid
8.273
7.868
7.864
7.846
7.842
7.395
7.373
7.260
7.134
7.126
6.591
6.583
3.834
1.882
1.873
1.868
1.859
1.849
1.839
1.811
1.780
1.770
1.761
1.756
1.747
1m
f1 (ppm)

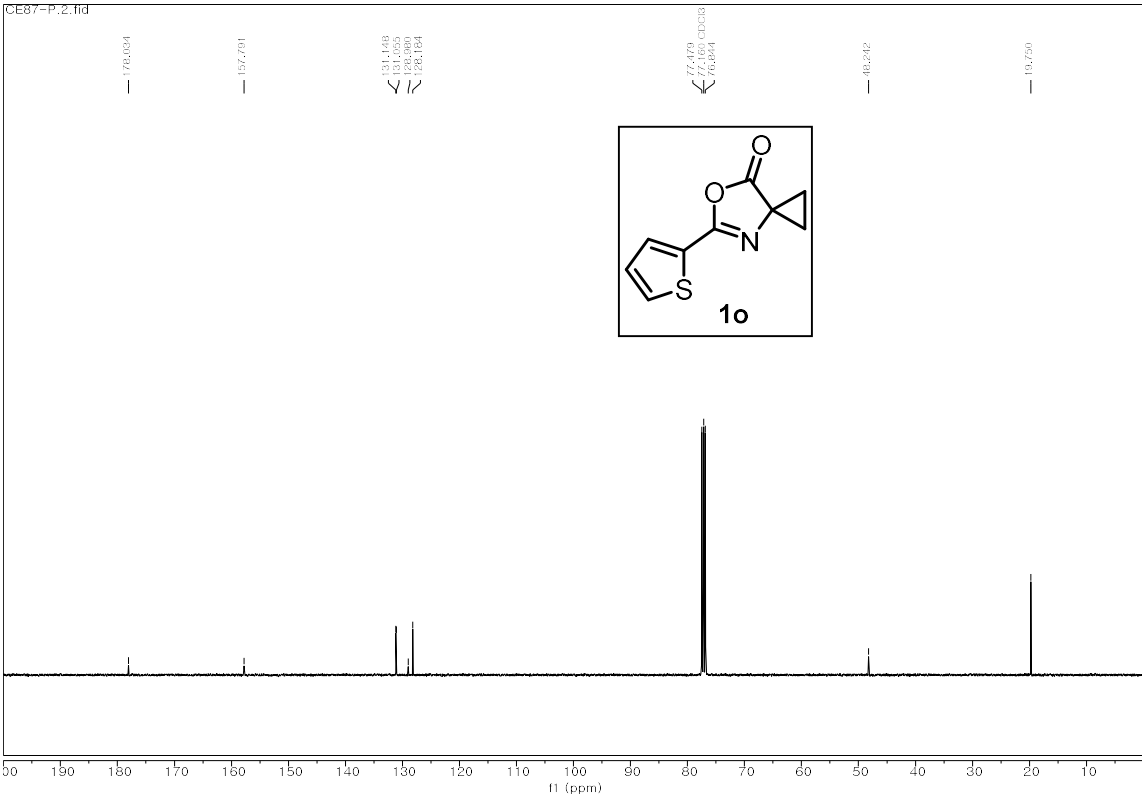

MS-260-P.2.fid
179.240
163.087
138.791
130.573
128.320
121.554
120.635
117.380
109.778
102.677
77.478
76.848
48.246
33.201
19.434
1m
f1 (ppm)

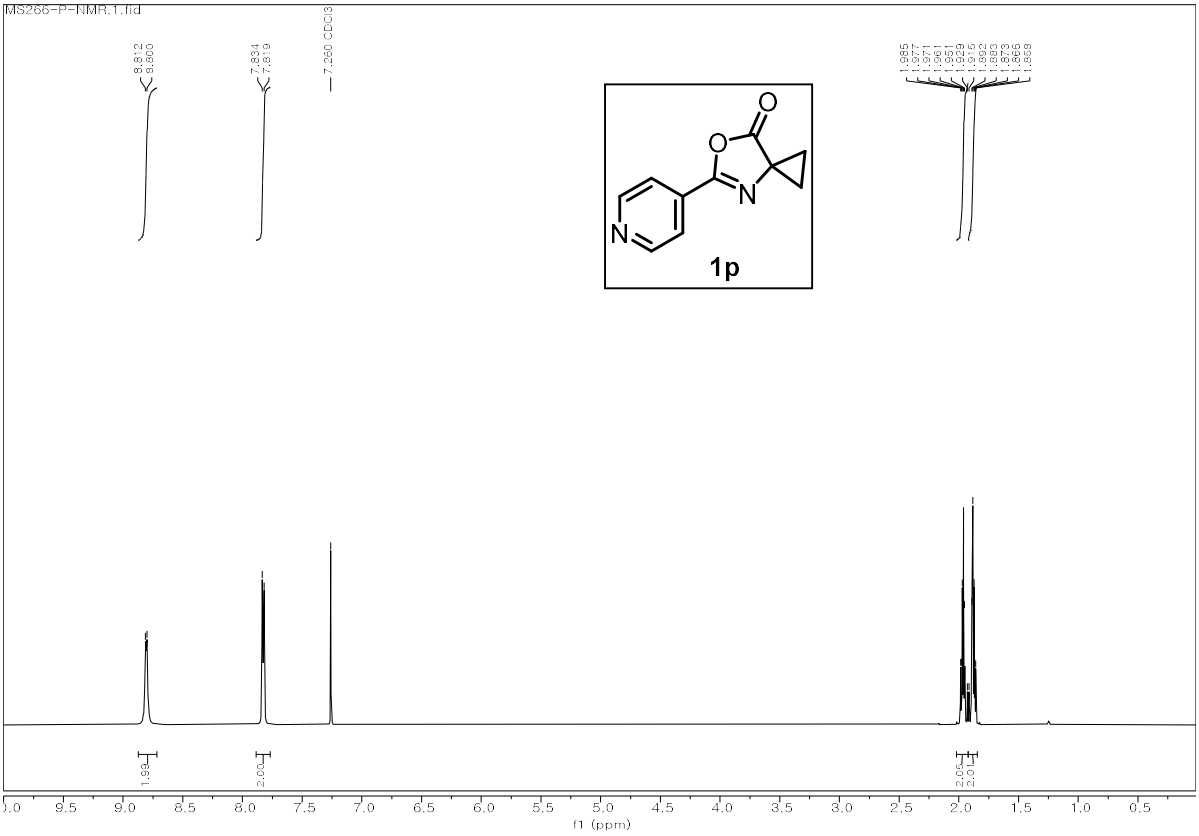


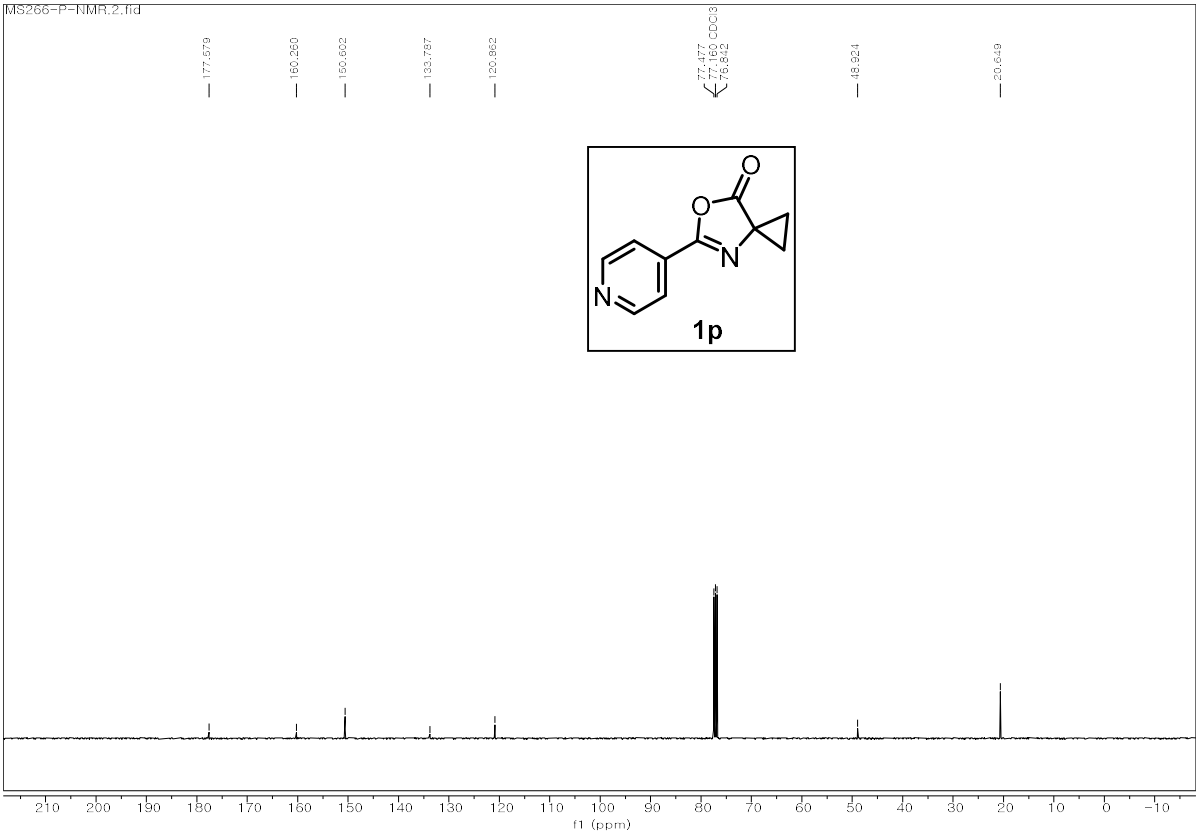
1n. CE27-P_400MHz.2.fid

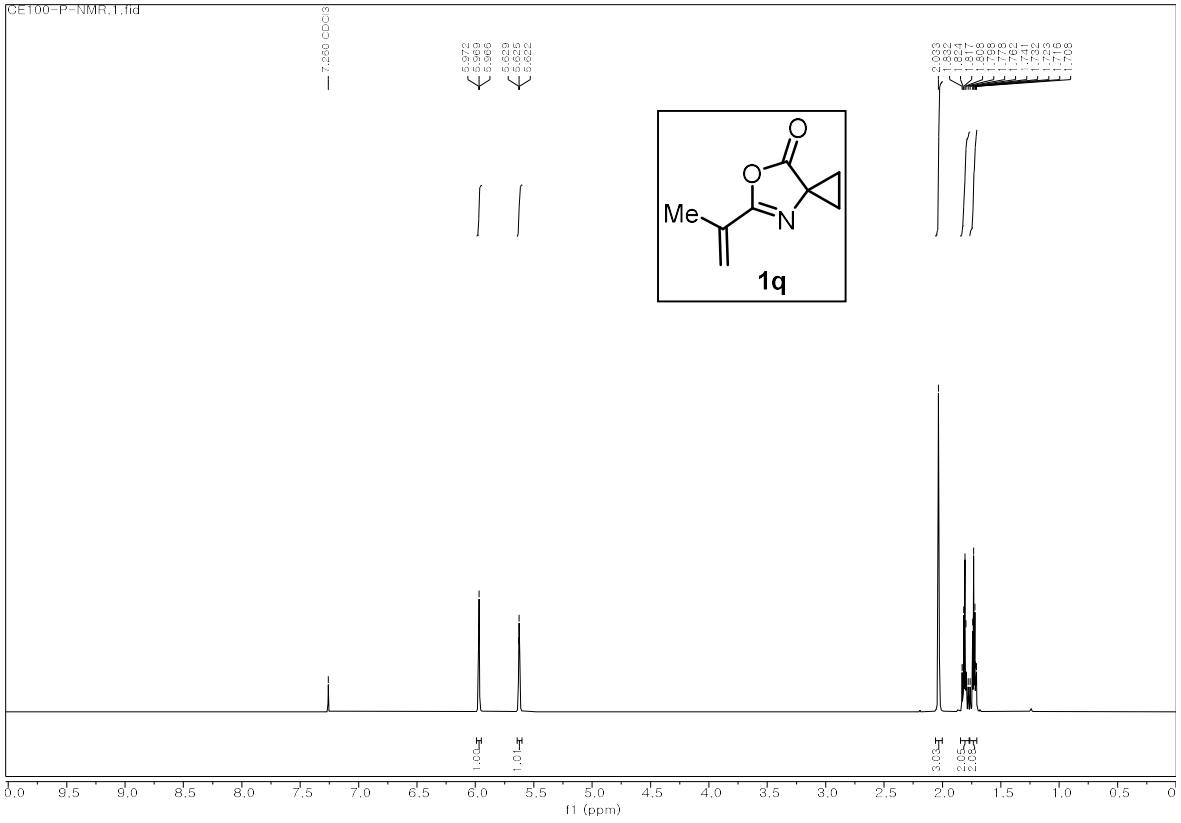


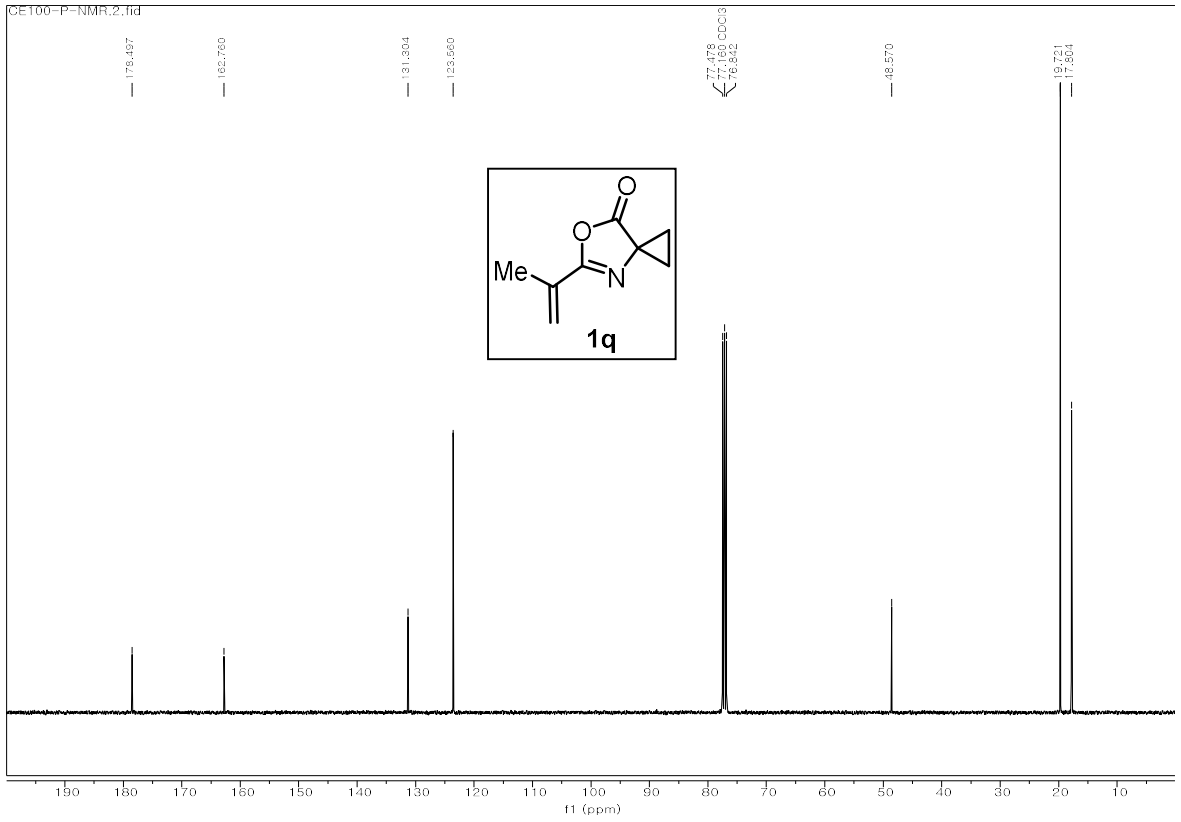

CE87-P.1.fid
1o
f1 (ppm)

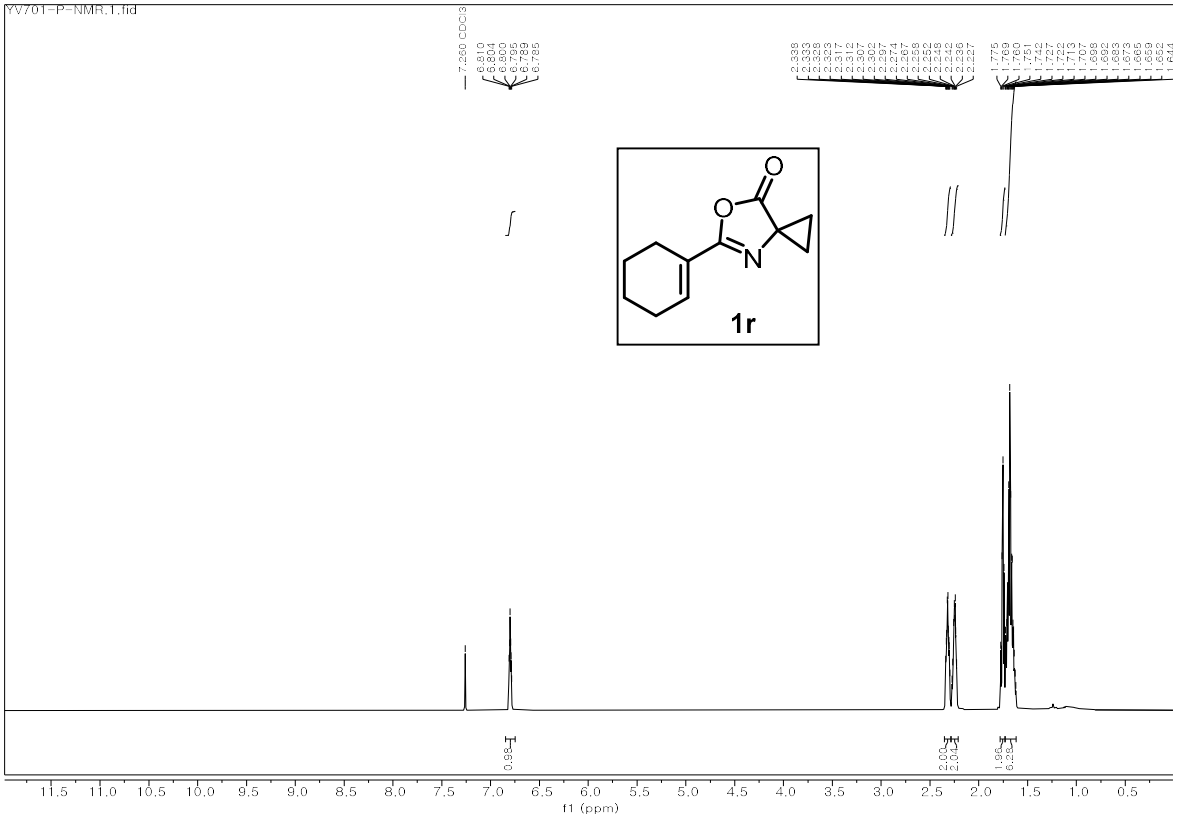

CE87-P.2.fid
1o
f1 (ppm)

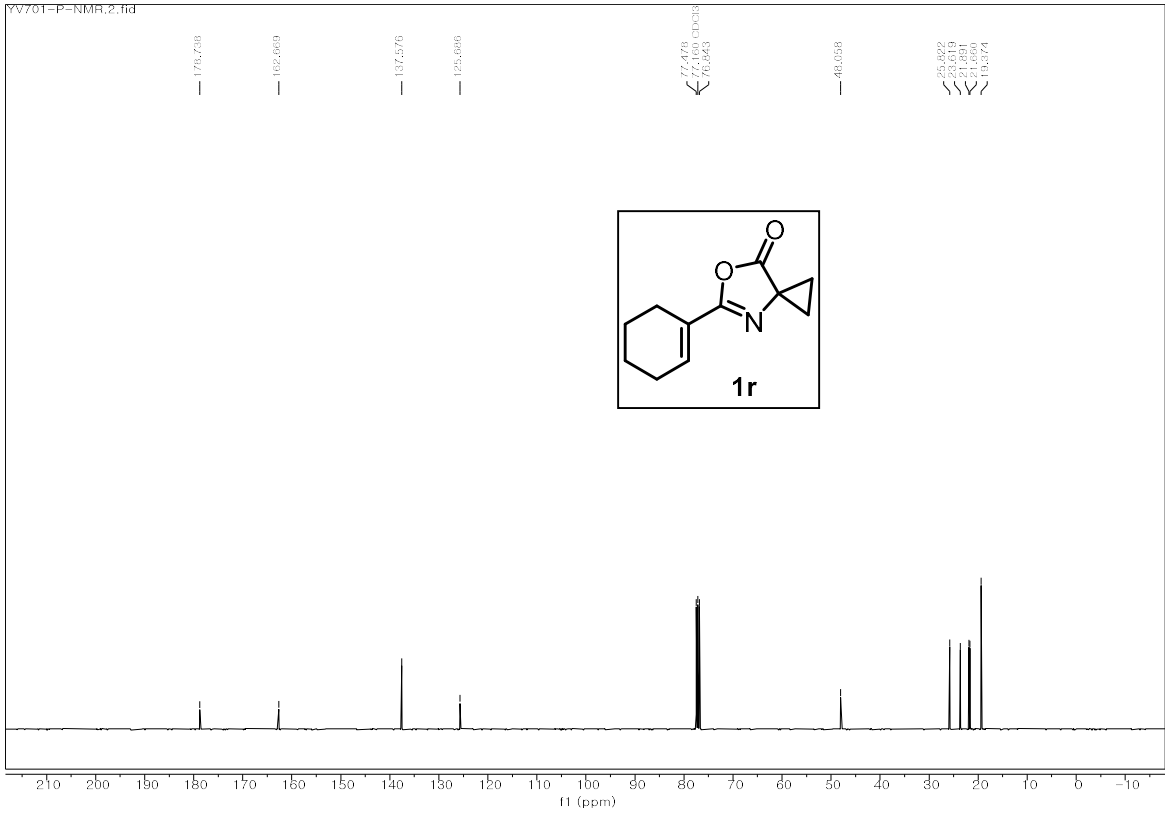

MS266-P-NMR.1.fid
8.812
8.800
7.834
7.819
7.260 CDCl3
1.985
1.977
1.971
1.961
1.951
1.929
1.915
1.892
1.883
1.873
1.866
1.859
O
O
N
N
1p
1.99
2.00
2.05
2.01
f1 (ppm)

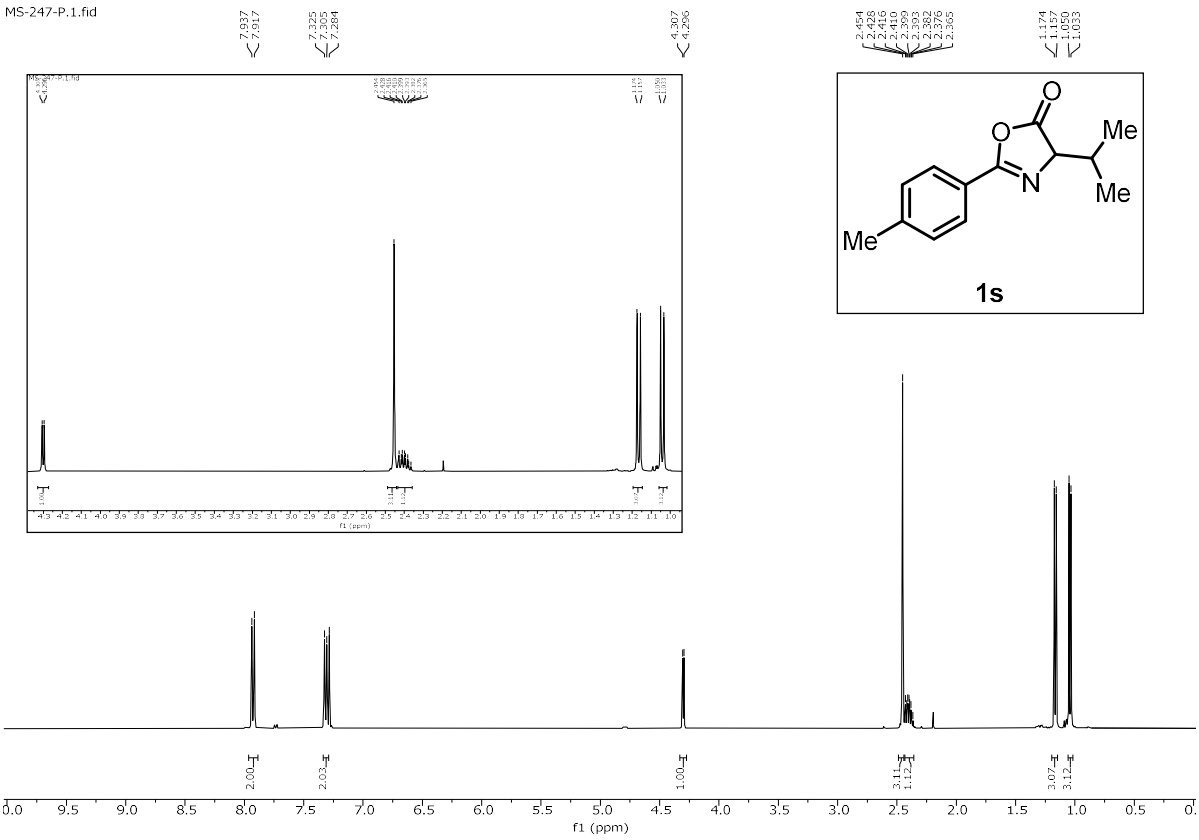

MS266-P-NMR.2.fid
177.579
160.260
150.602
133.787
120.862
77.417
77.160 CDCl3
76.842
48.924
20.649
O
O
N
N
1p
f1 (ppm)

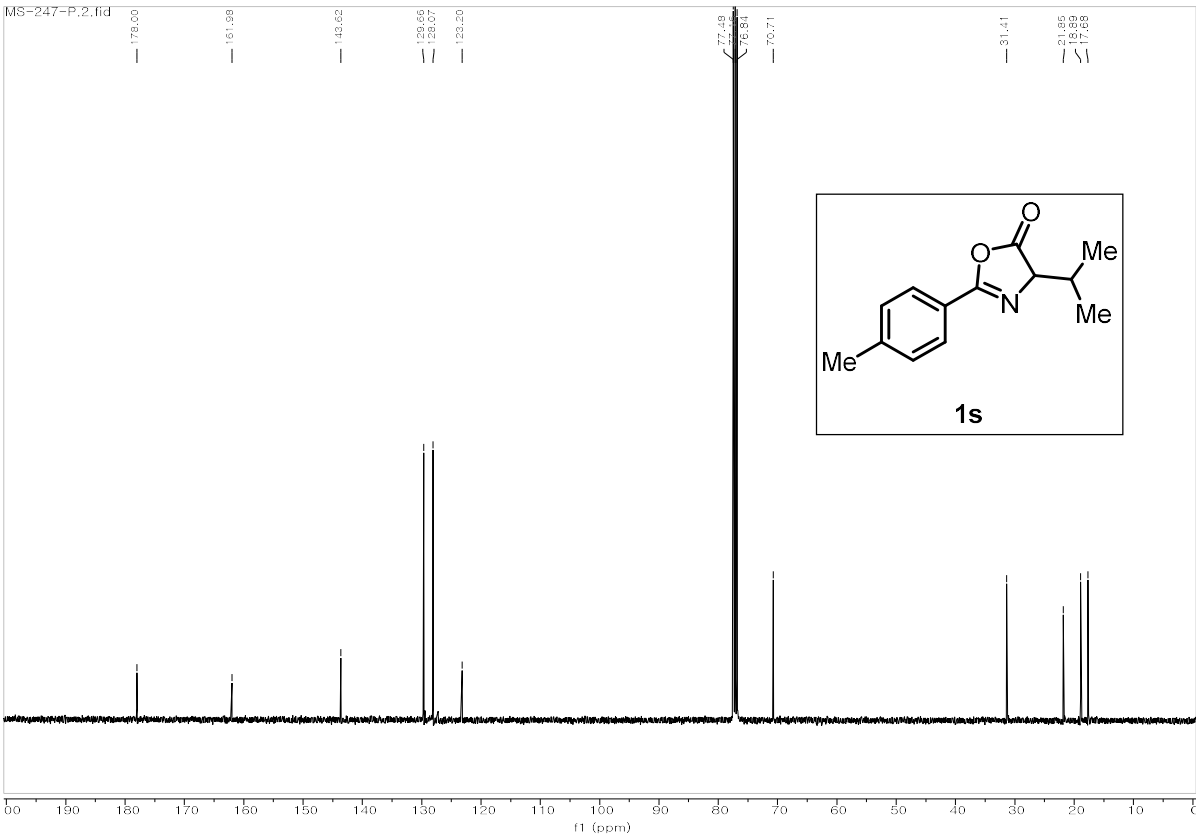

CE100-P-NMR.1.fid
7.260 CDCl3
5.972
5.969
5.966
5.629
5.625
5.622
2.033
1.882
1.844
1.817
1.808
1.798
1.788
1.782
1.741
1.732
1.723
1.718
1.708
Me
O
O
N
1q
1.00
1.01
3.03
2.05
2.08
f1 (ppm)

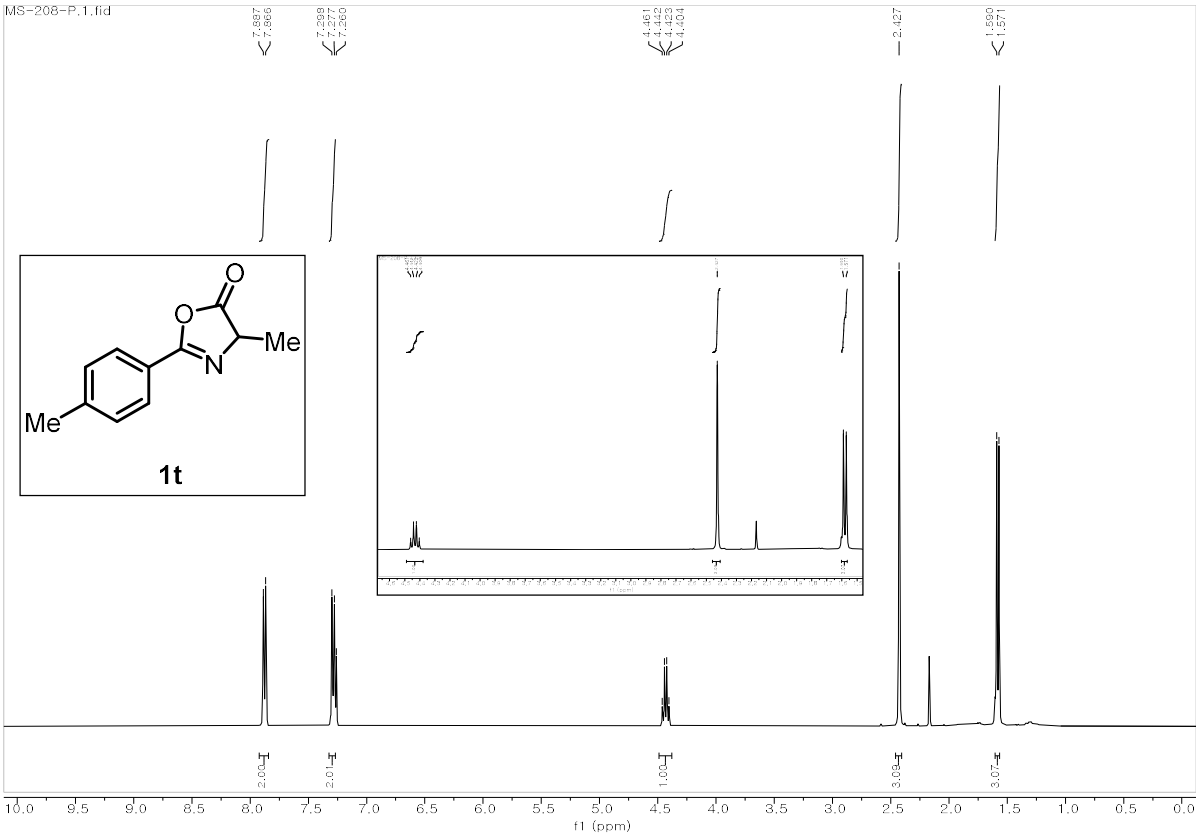

CE100-P-NMR.2.fid
178.497
162.760
131.304
123.560
77.478
77.160 CDCl3
76.842
48.570
19.751
17.864
Me
O
O
N
1q
f1 (ppm)

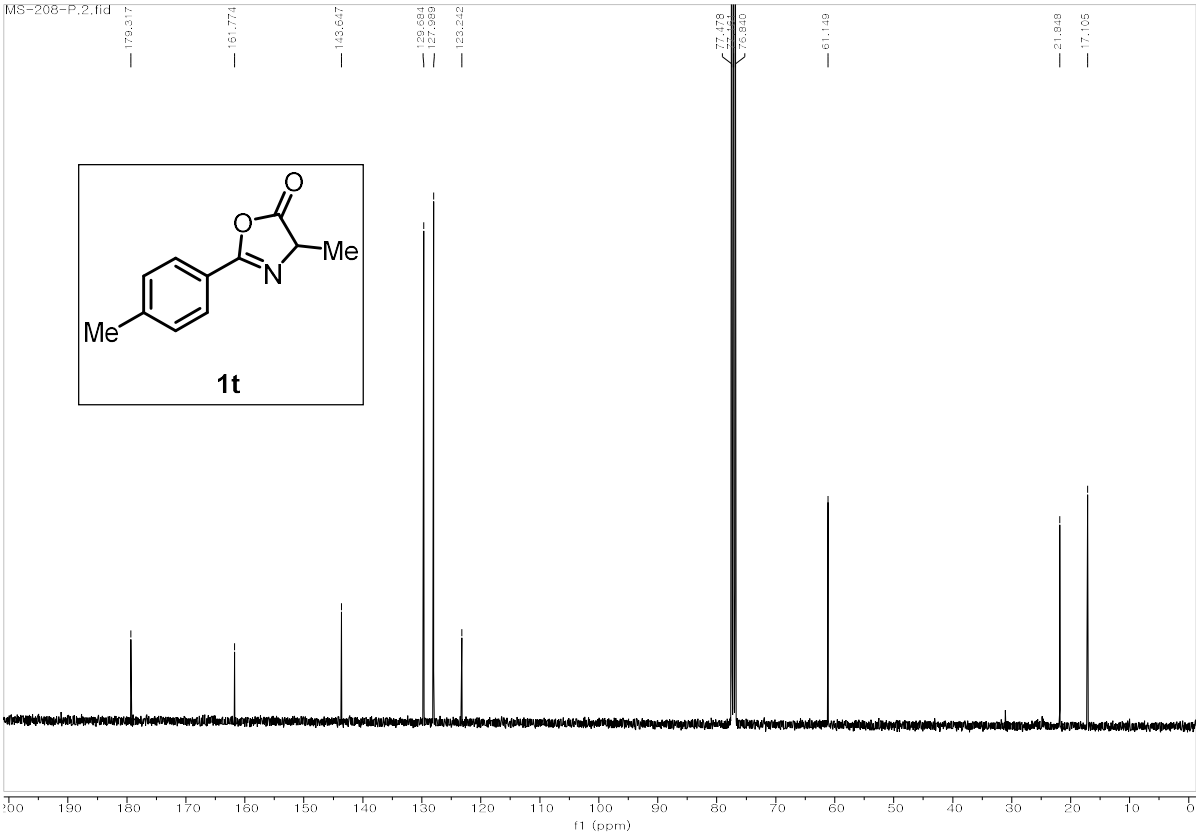

YV701-P-NMR.1.fid
1r
f1 (ppm)

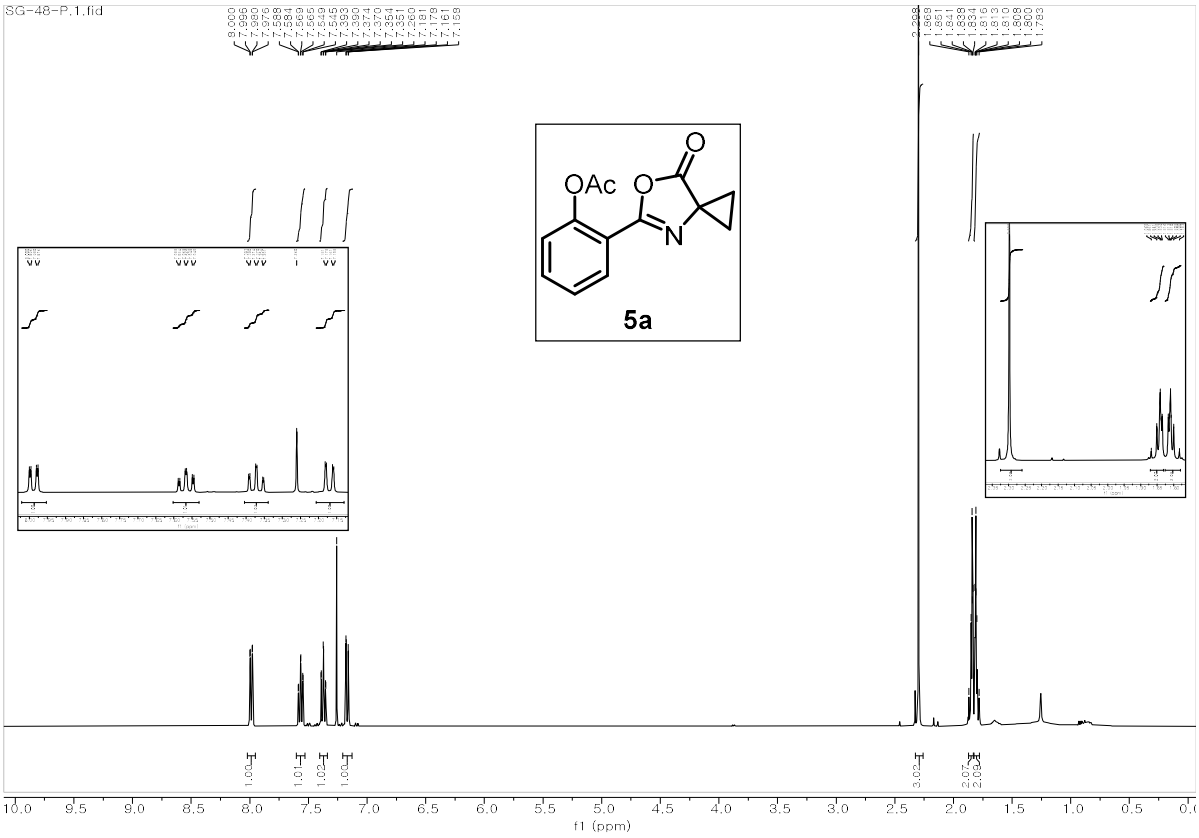

YV701-P-NMR.2.fid
1r
f1 (ppm)

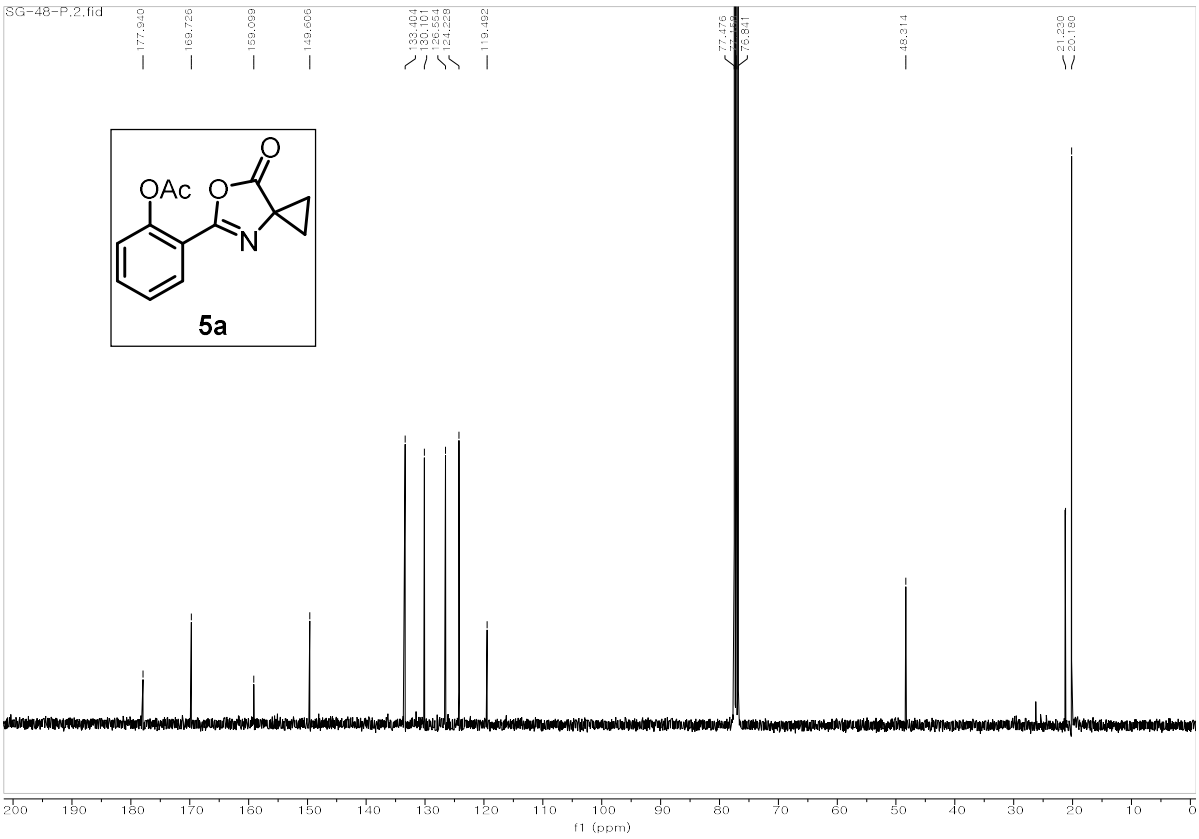

MS-247-P.1.fid
7.937
7.917
7.325
7.305
7.284
4.307
4.296
2.454
2.428
2.416
2.410
2.399
2.393
2.382
2.376
2.365
1.174
1.157
1.050
1.033
O
O
N
Me
Me
Me
1s
2.00
2.03
1.00
3.11
1.12
3.07
3.12
f1 (ppm)

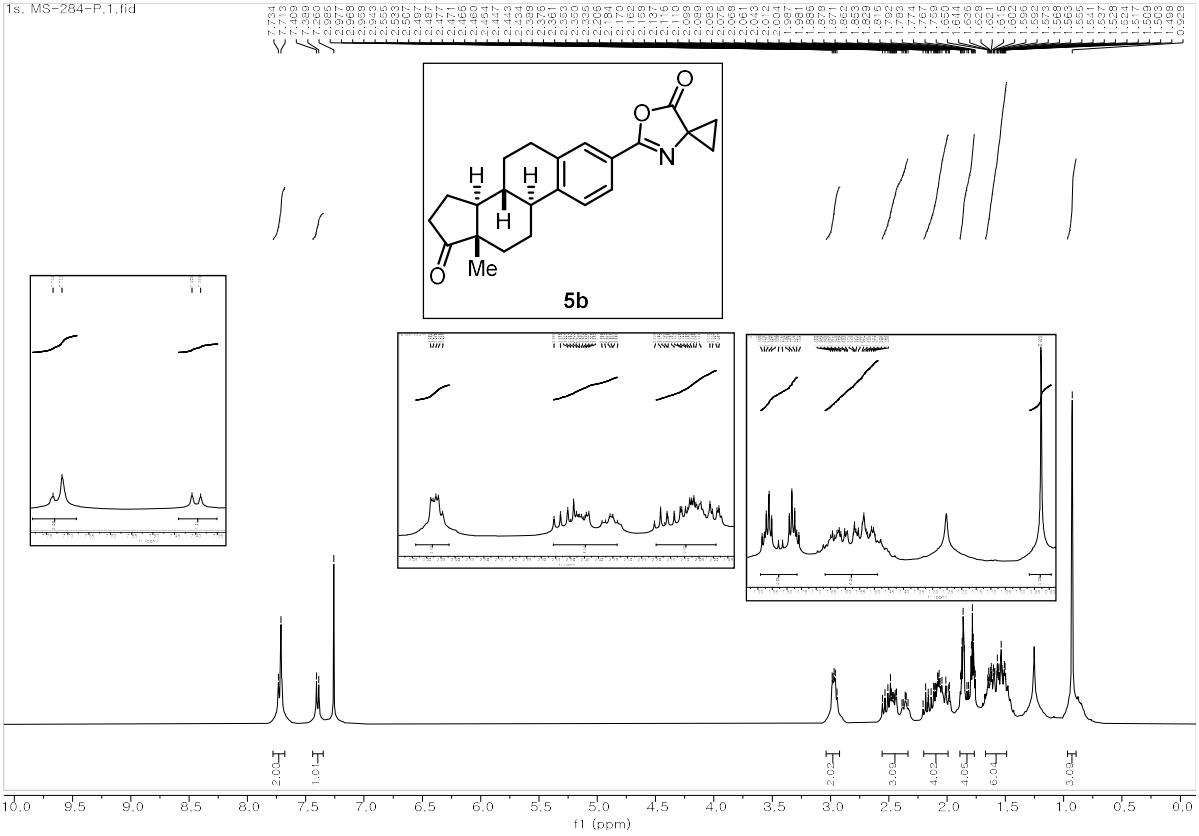

MS-247-P.2.fid
178.00
161.98
143.62
129.66
128.07
123.20
77.48
77.16
76.84
70.71
31.41
21.85
18.89
17.68
O
O
N
Me
Me
Me
1s
f1 (ppm)

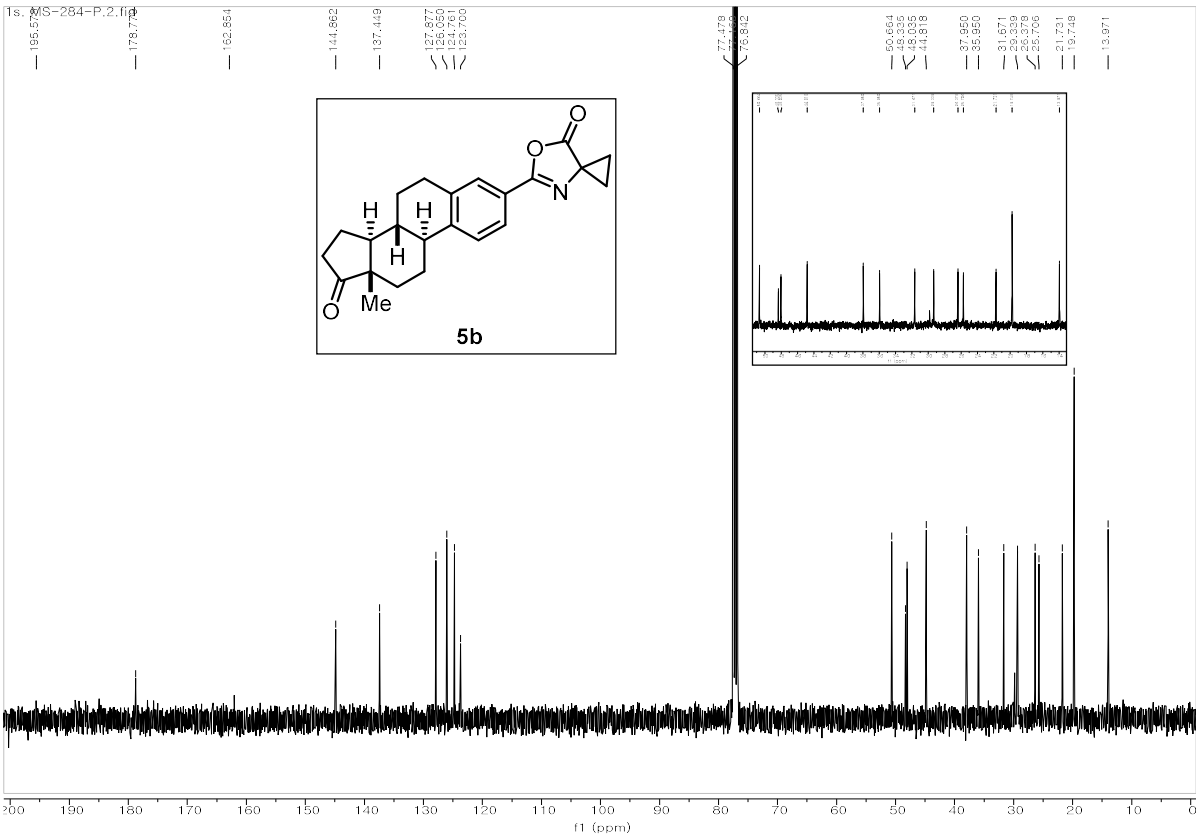

MS-208-P.1.fid
7.887
7.866
7.299
7.277
7.260
4.461
4.442
4.423
4.404
2.427
1.590
1.571
O
O
N
Me
Me
1t
2.00
2.01
1.00
3.09
3.07
f1 (ppm)

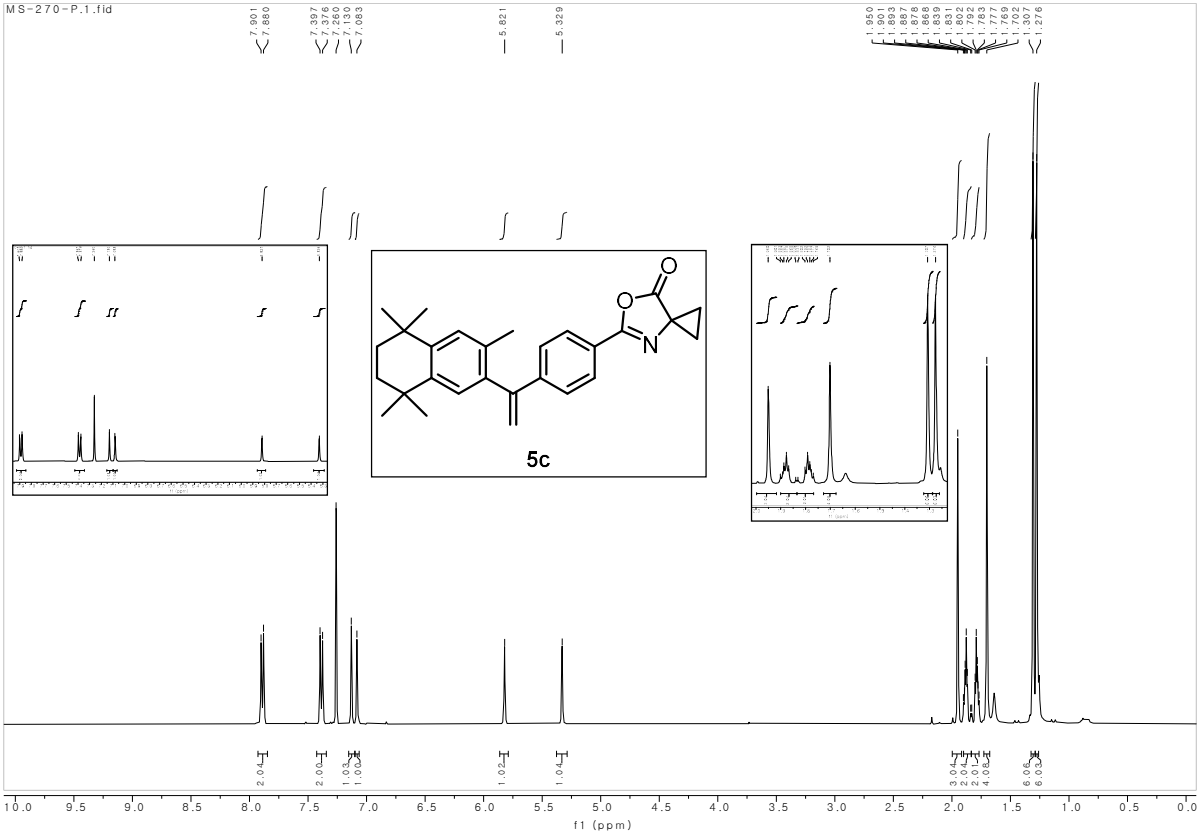

MS-208-P.2.fid
179.317
161.774
143.647
129.684
127.989
123.242
77.478
76.849
61.149
21.848
17.105
O
O
N
Me
Me
1t
f1 (ppm)

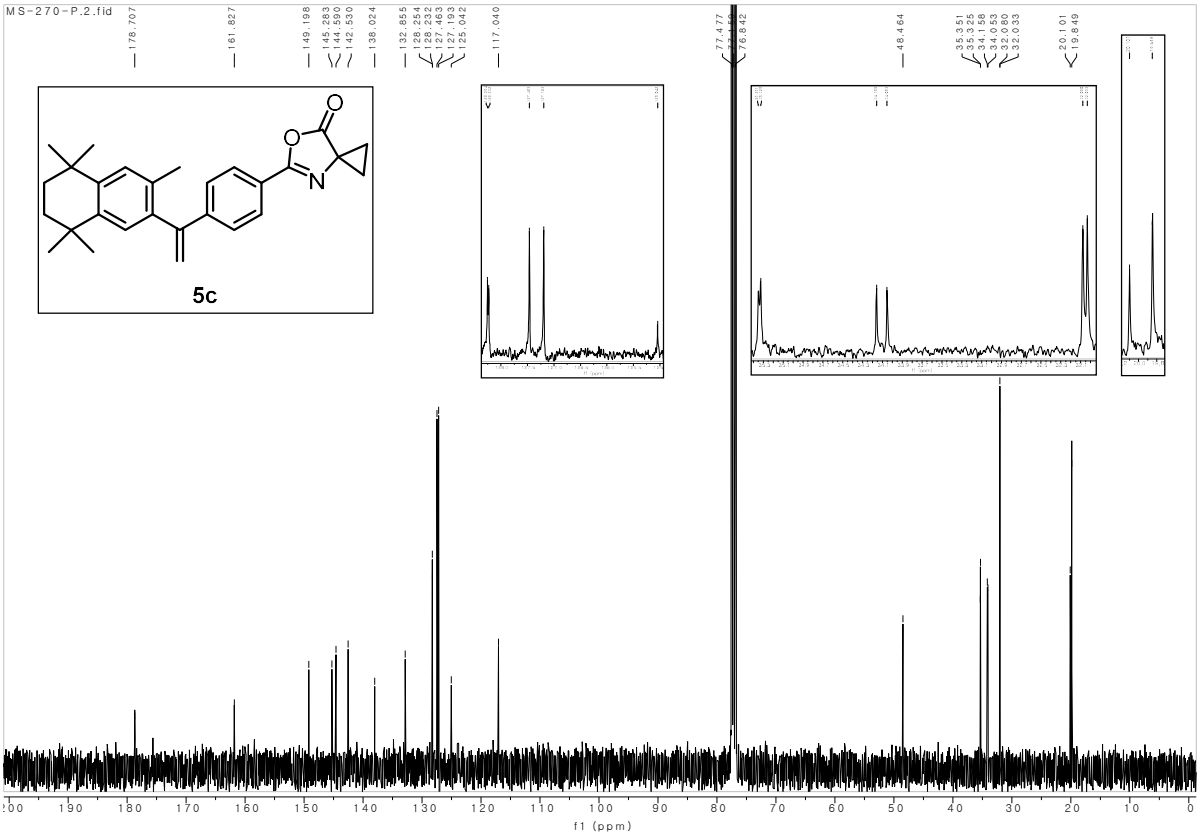

SG-48-P.1.fid
OAc
O
O
N
5a
f1 (ppm)

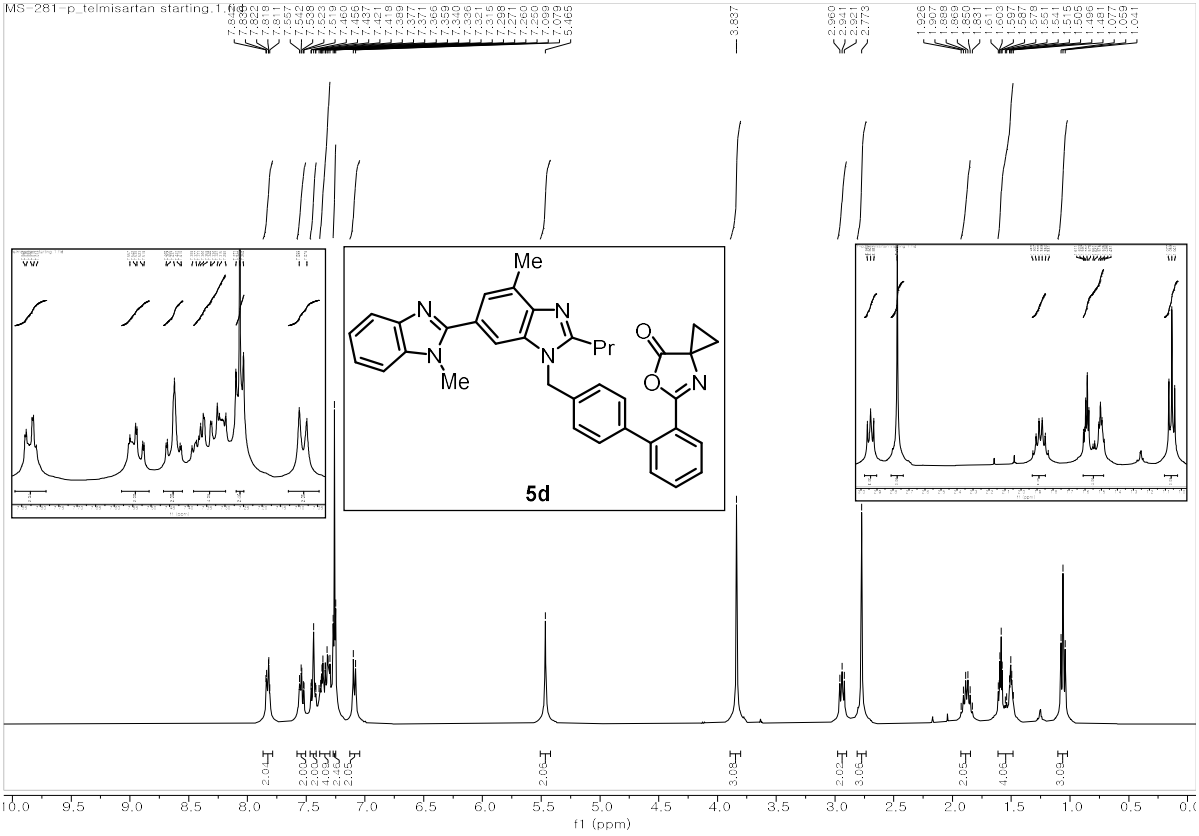

SG-48-P.2.fid
OAc
O
O
N
5a
f1 (ppm)

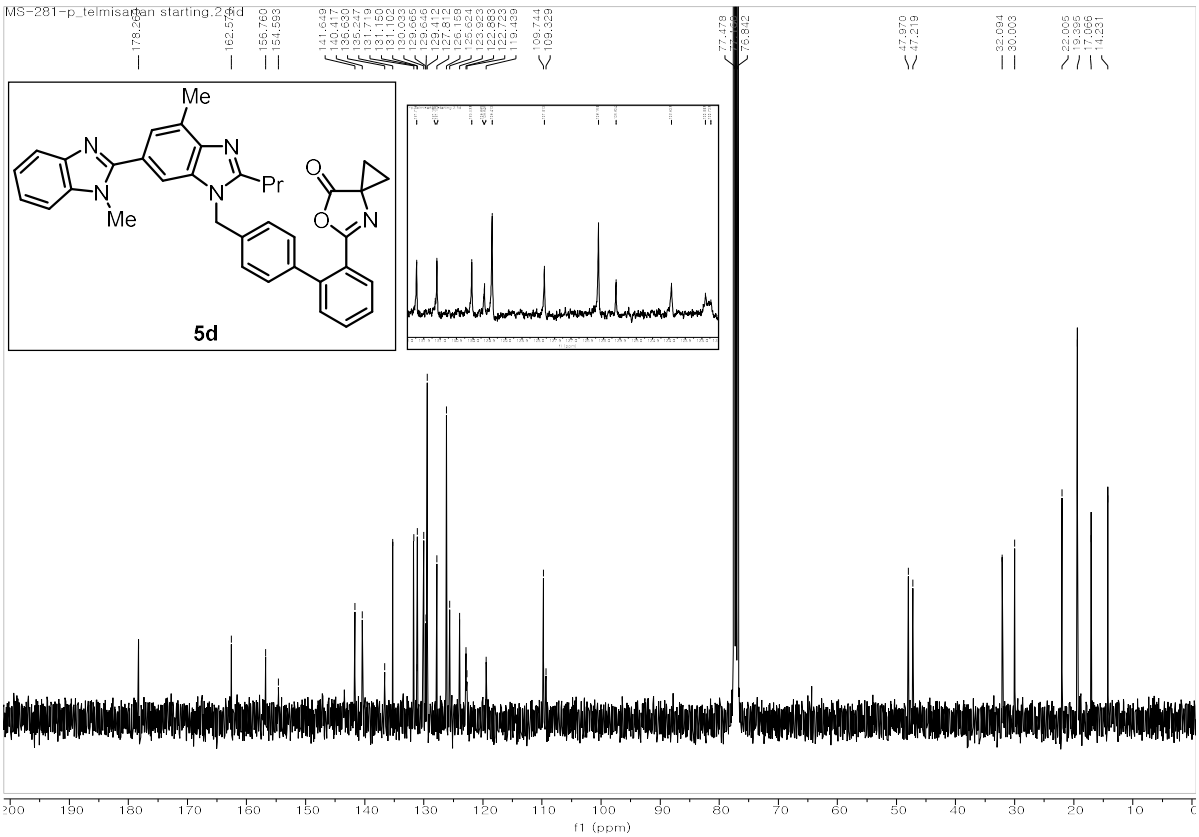

1s, MS-284-P.1.fid
5b
f1 (ppm)

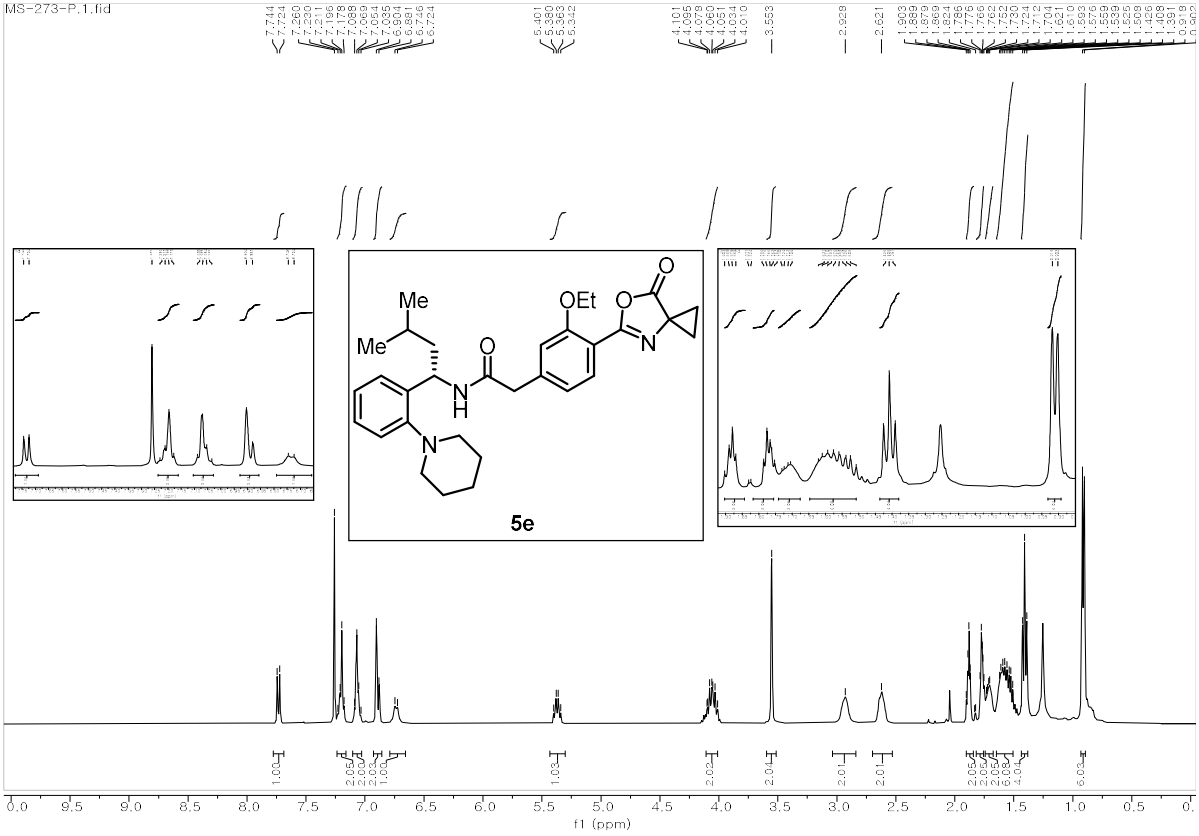

1s, MS-284-P.2.fid
5b
f1 (ppm)

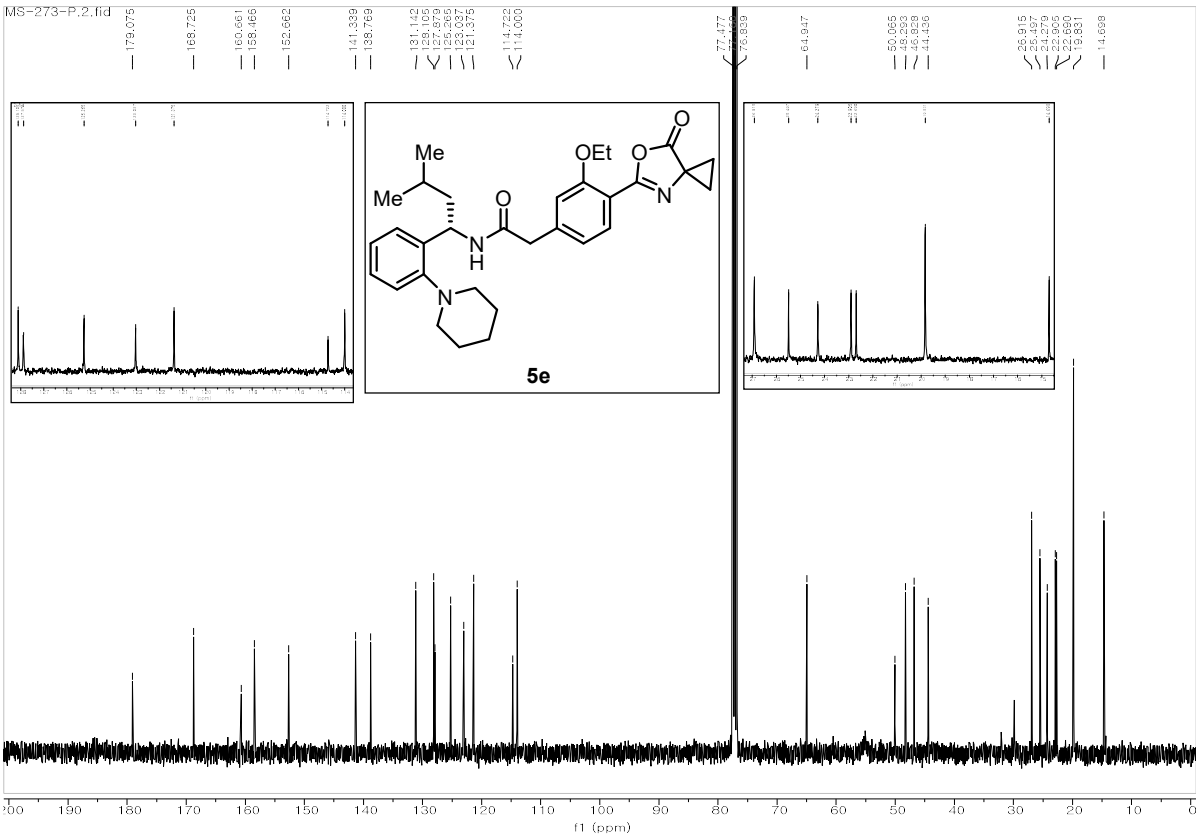

MS-270-P.1.fid
5c
f1 (ppm)

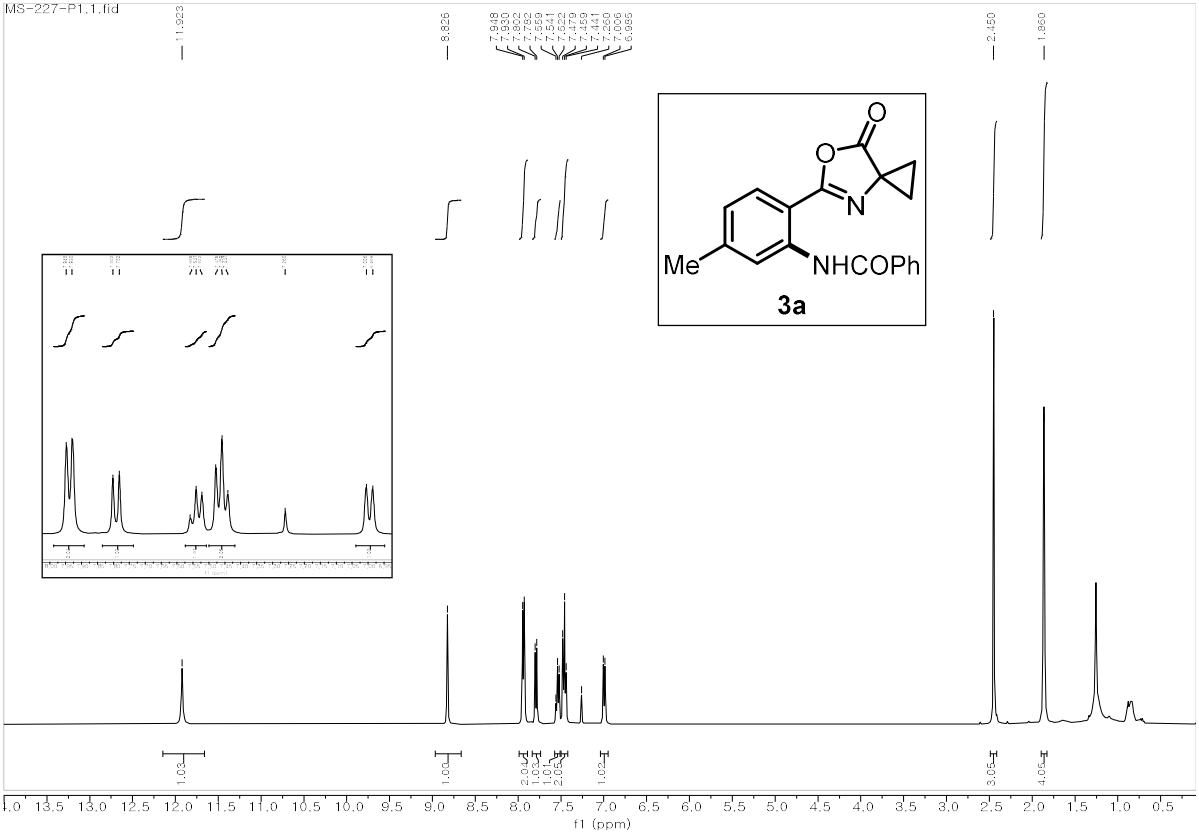

MS-270-P.2.fid
5c
f1 (ppm)

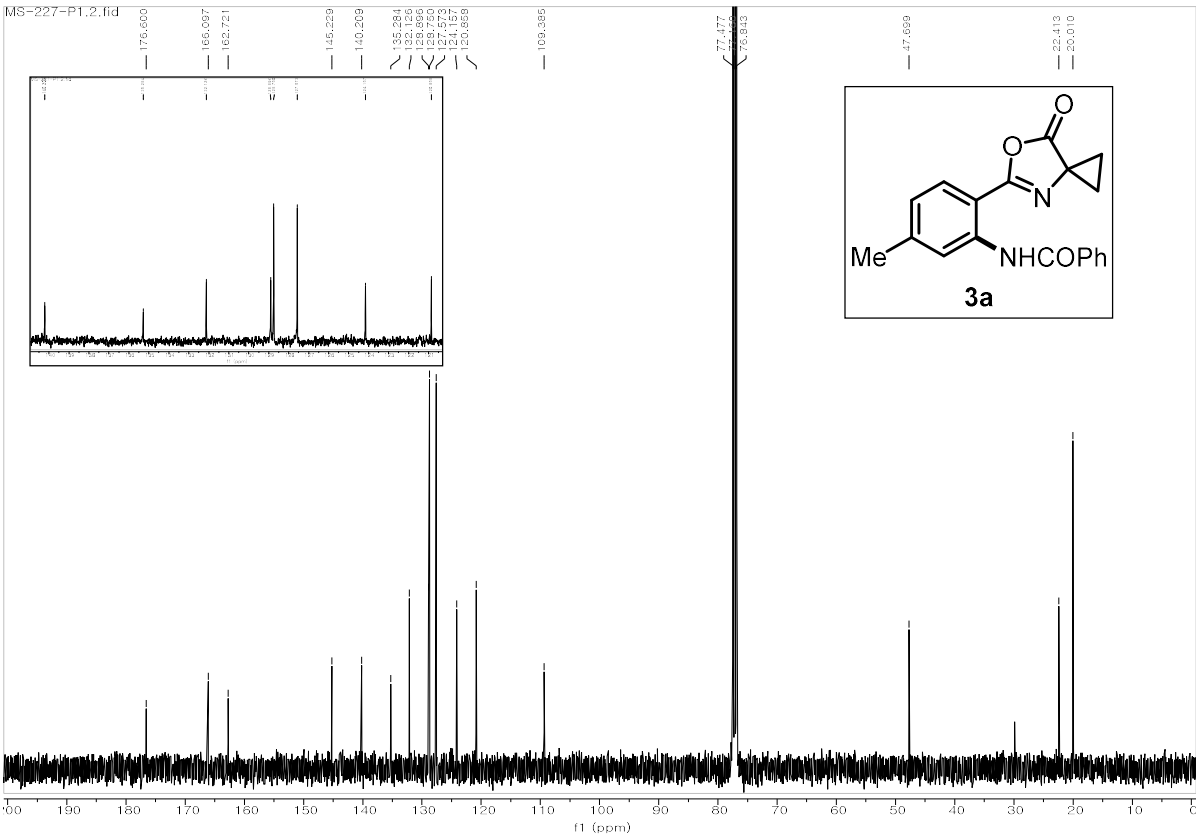

MS-281-p_telmisartan starting.1.fid
Me
N
N
Me
N
Pr
N
O
O
N
5d
f1 (ppm)

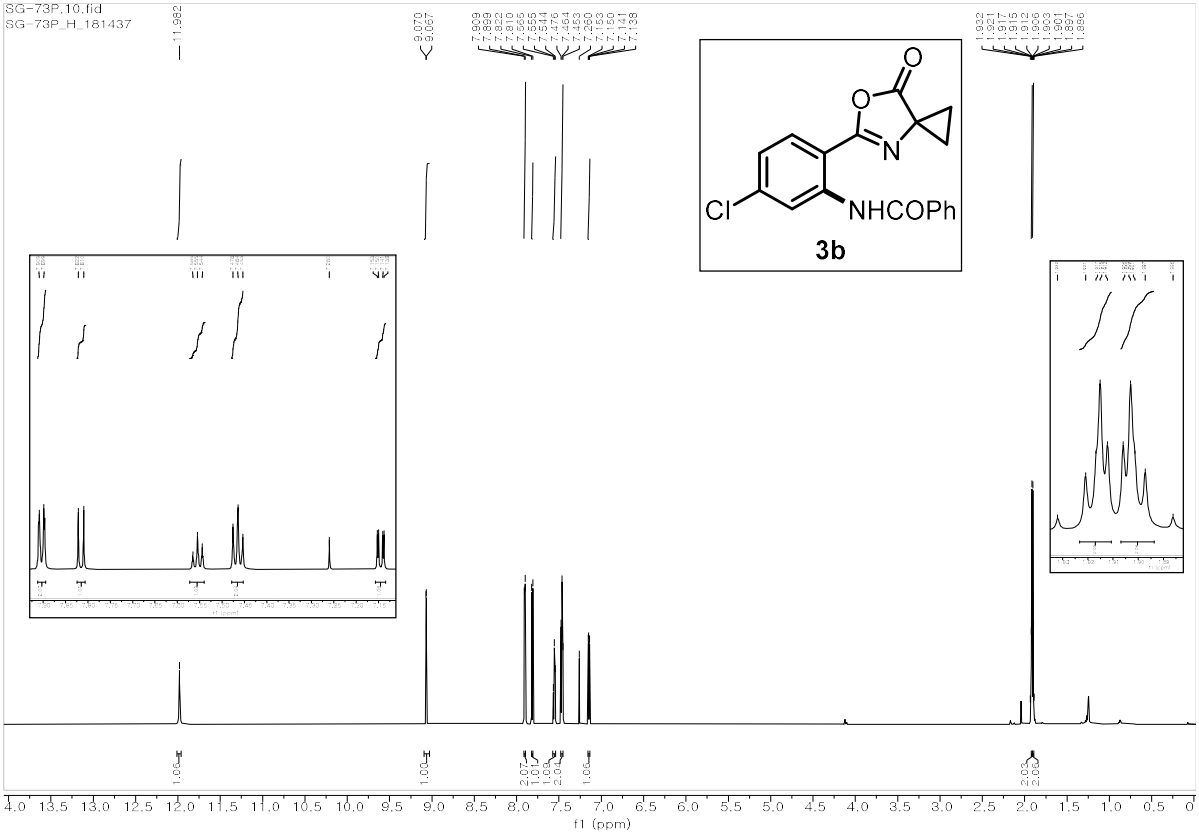

MS-281-p_telmisartan starting.2.fid
Me
N
N
Me
N
Pr
N
O
O
N
5d
f1 (ppm)

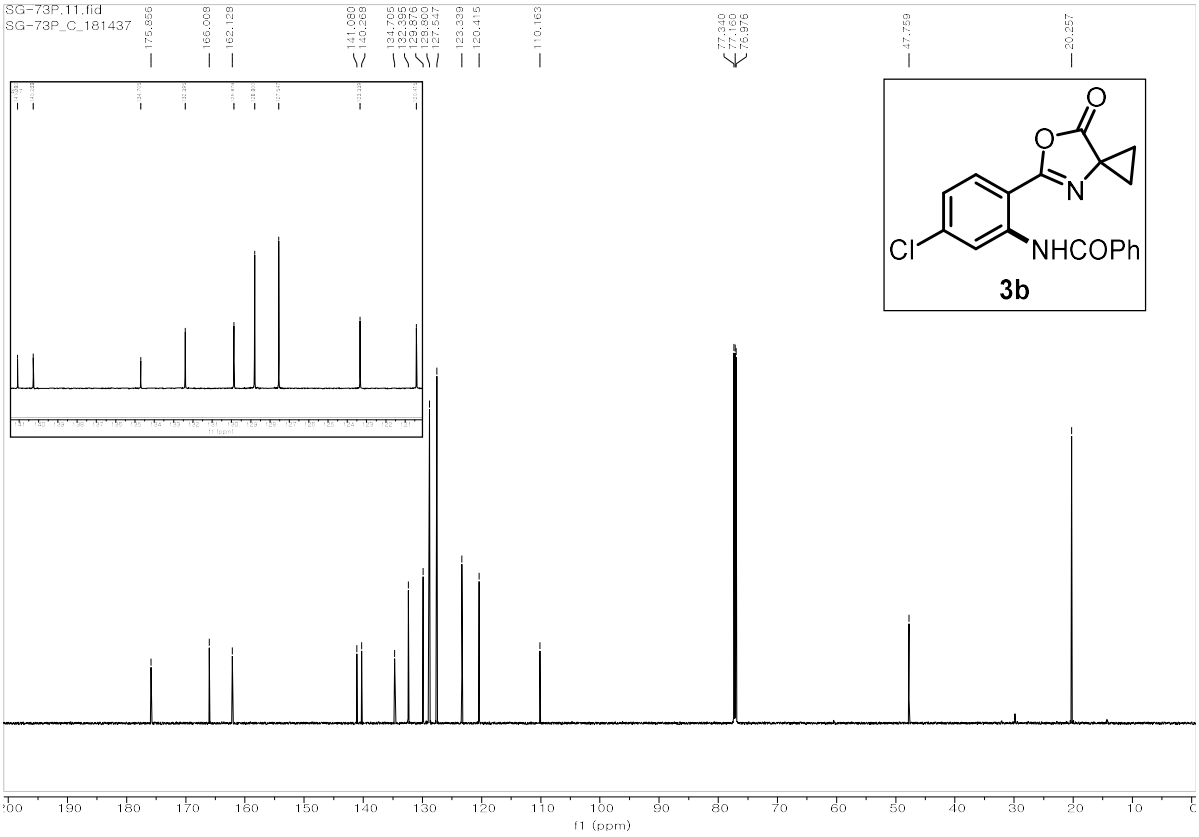

MS-273-P.1.fid
Me
Me
OEt
O
O
N
H
N
O
O
N
5e
f1 (ppm)

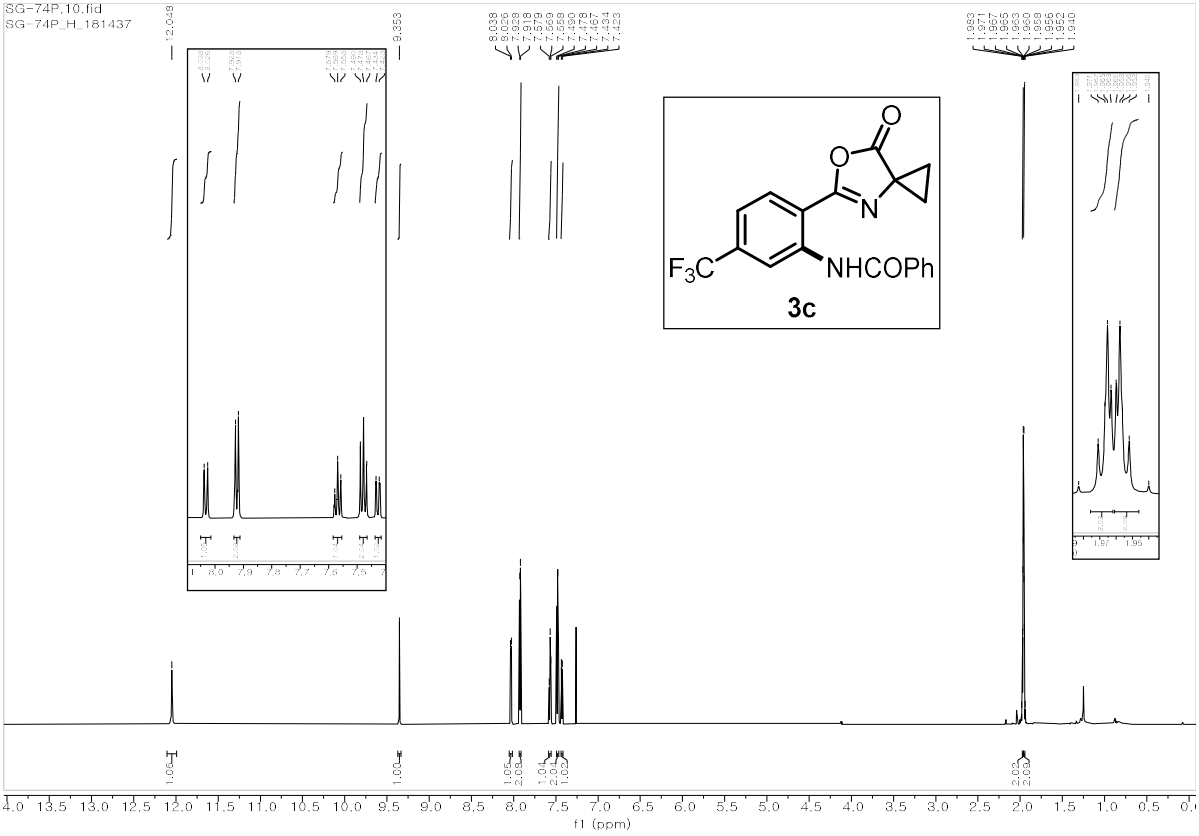

MS-273-P.2.fid
Me
Me
OEt
O
O
N
H
N
O
O
N
5e
f1 (ppm)

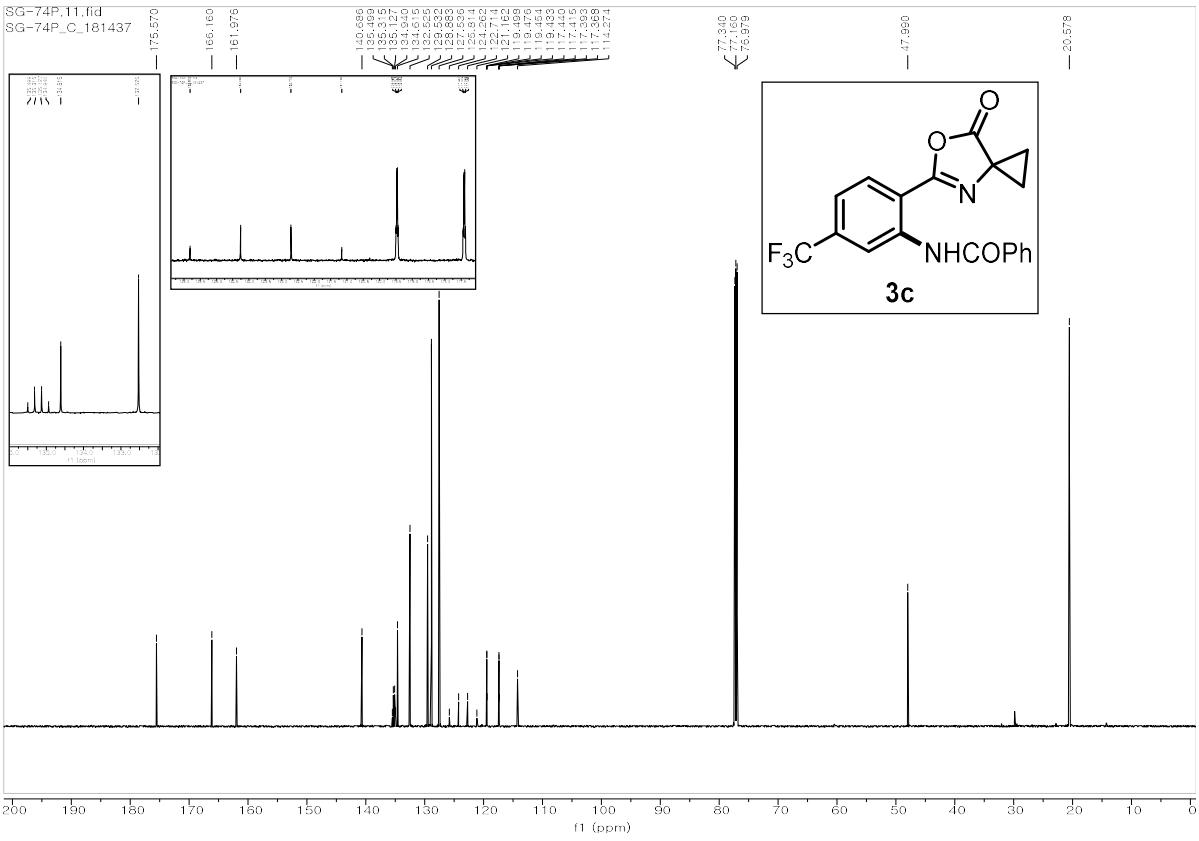

MS-227-P1.1.fid
11.923
8.826
7.948
7.930
7.922
7.782
7.559
7.541
7.523
7.479
7.459
7.441
7.280
7.086
6.886
2.450
1.860
Me
NHCOPh
3a
f1 (ppm)

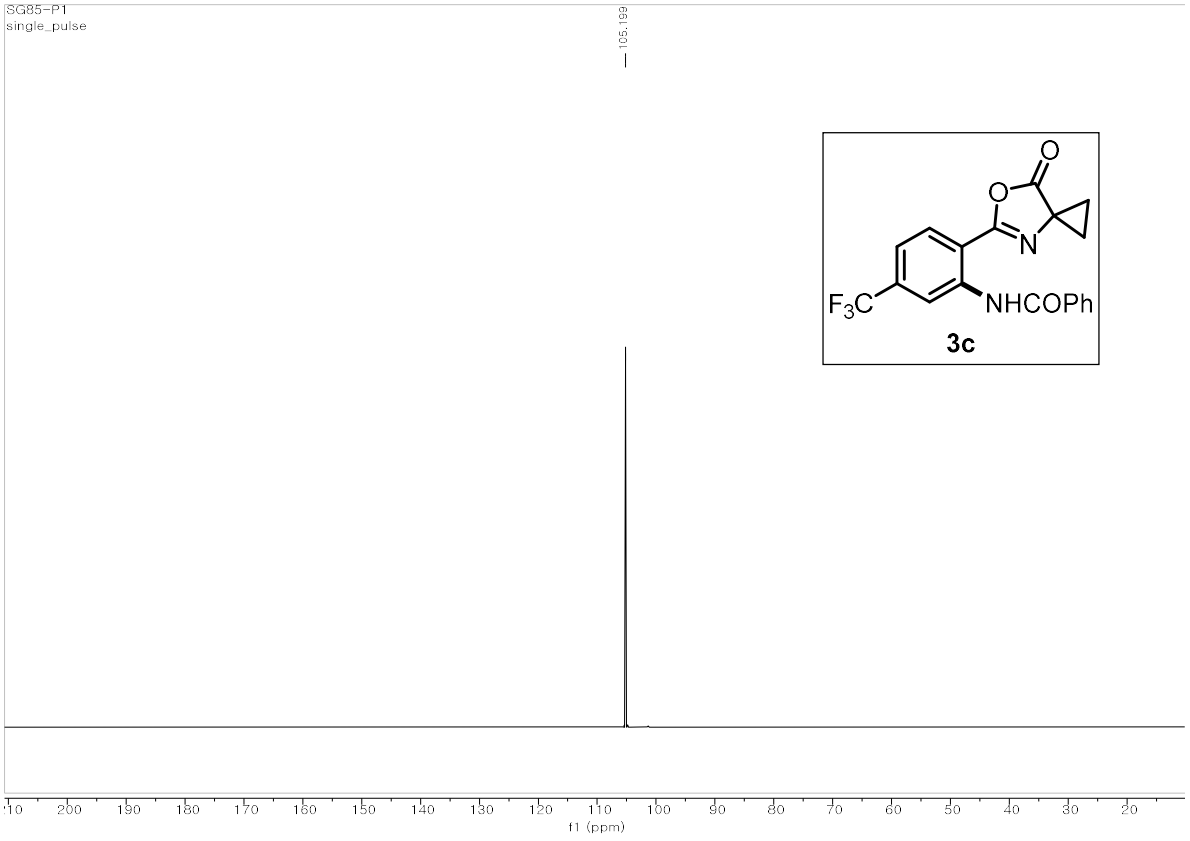

MS-227-P1.2.fid
176.600
166.097
162.721
145.229
140.209
135.284
132.156
129.060
128.750
127.573
124.157
120.859
109.385
77.477
77.163
76.843
47.699
22.413
20.010
Me
NHCOPh
3a
f1 (ppm)

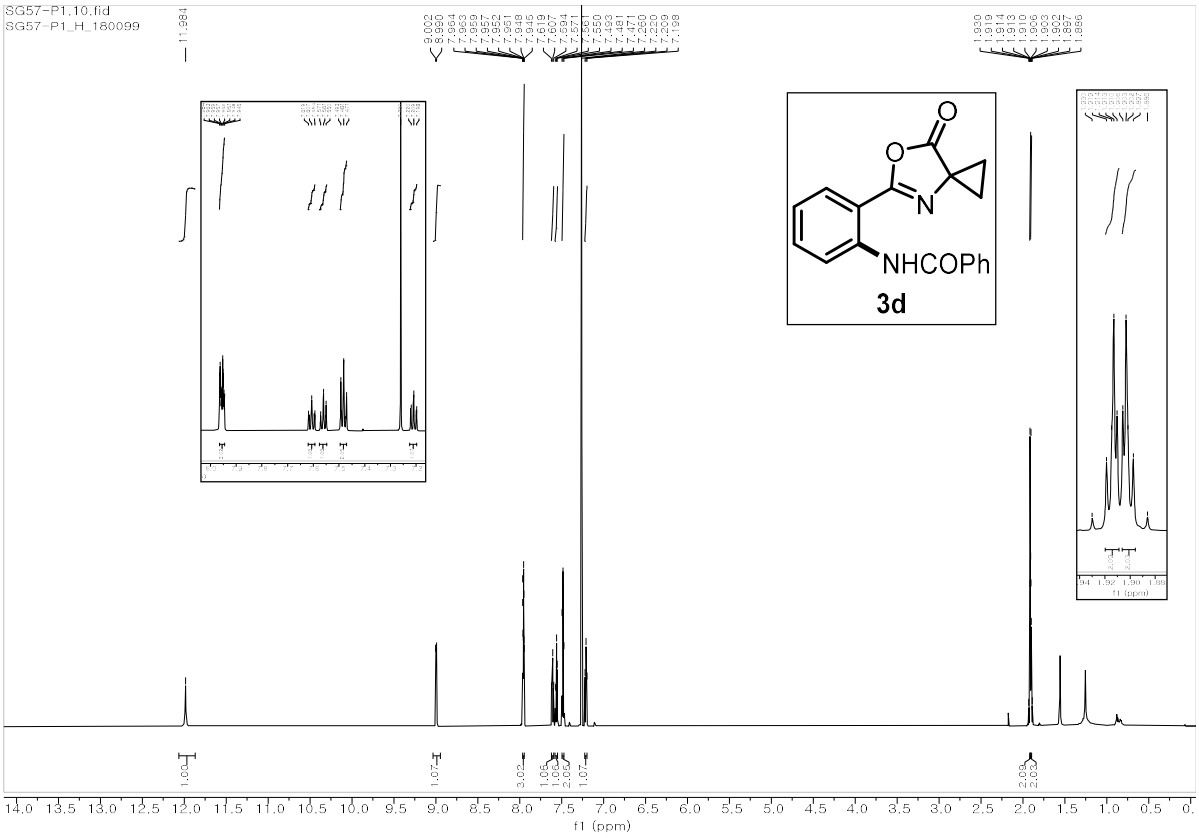

SG-73P.10.fid
SG-73P_H_181437
11.982
9.070
9.067
7.909
7.899
7.822
7.810
7.565
7.555
7.544
7.476
7.464
7.453
7.450
7.153
7.150
7.141
7.138
1.932
1.921
1.917
1.915
1.912
1.906
1.903
1.901
1.897
1.886
Cl
NHCOPh
3b
f1 (ppm)

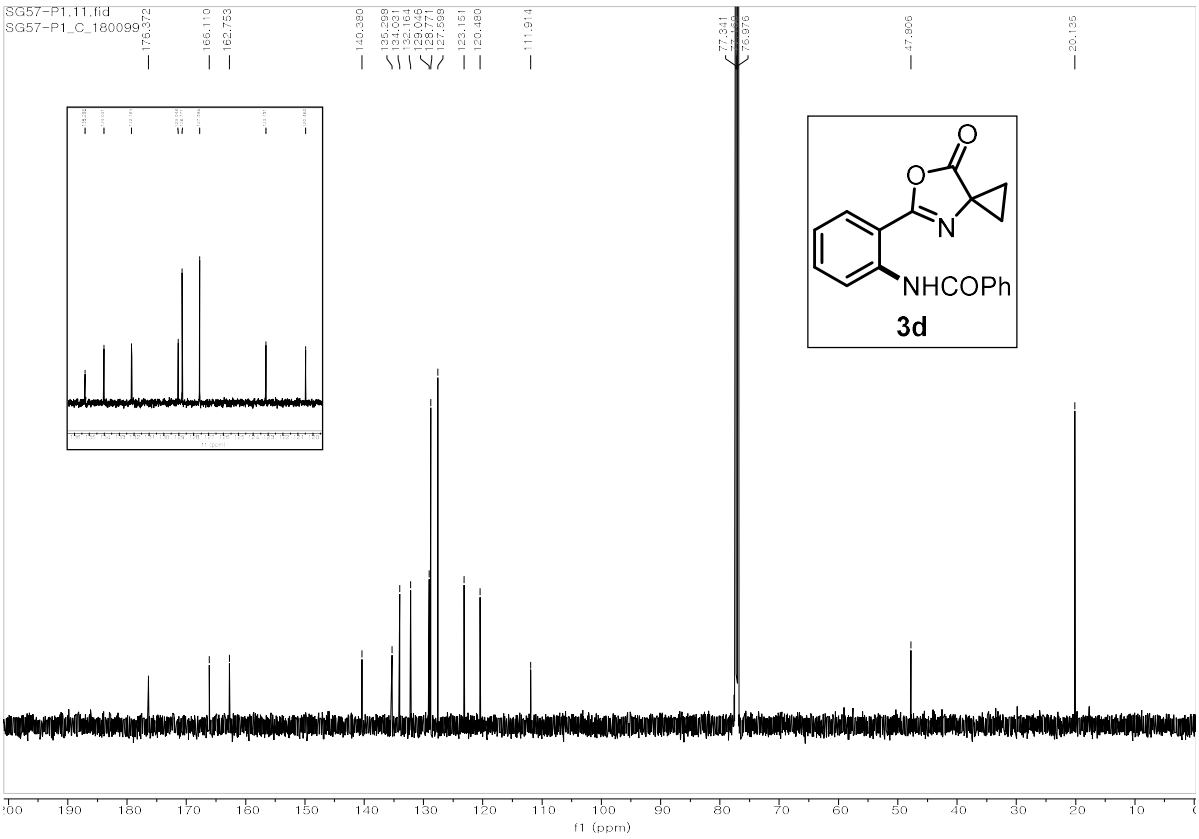

SG-73P.11.fid
SG-73P_C_181437
175.856
166.008
162.129
141.080
140.269
134.705
132.395
129.876
129.800
127.547
123.339
120.415
110.163
77.340
77.160
76.976
47.759
20.257
Cl
NHCOPh
3b
f1 (ppm)

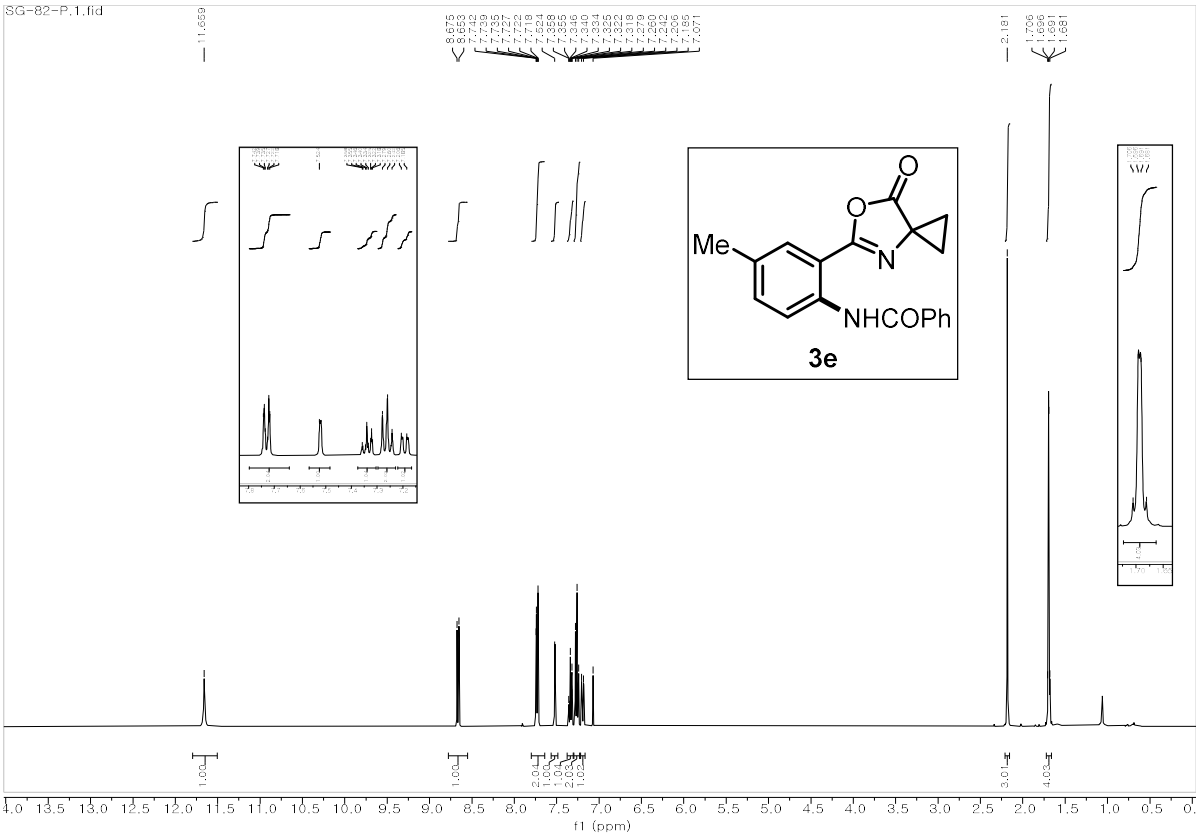

SG-74P_10.fid
SG-74P_H_181437
12.049
9.353
8.038
8.035
7.929
7.918
7.679
7.669
7.668
7.490
7.478
7.467
7.434
7.423
1.983
1.971
1.967
1.965
1.963
1.960
1.958
1.956
1.952
1.940
O
O
N
F3C
NHCOPh
3c
f1 (ppm)

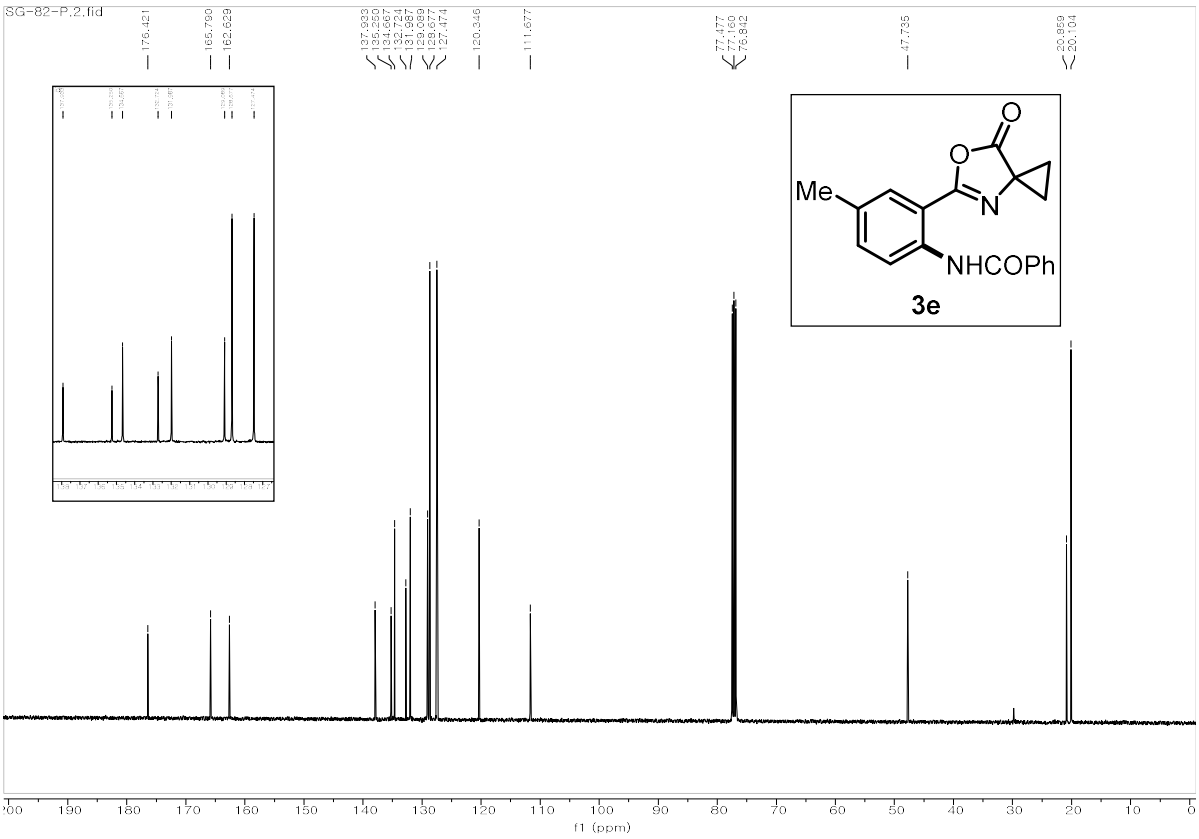

SG-74P_11.fid
SG-74P_C_181437
175.570
166.160
161.976
140.686
135.499
135.315
134.107
134.015
132.625
129.582
127.838
125.814
124.262
121.714
121.150
119.468
119.476
119.464
119.433
117.445
117.415
117.393
117.368
114.274
77.340
77.160
76.979
47.990
20.578
O
O
N
F3C
NHCOPh
3c
f1 (ppm)

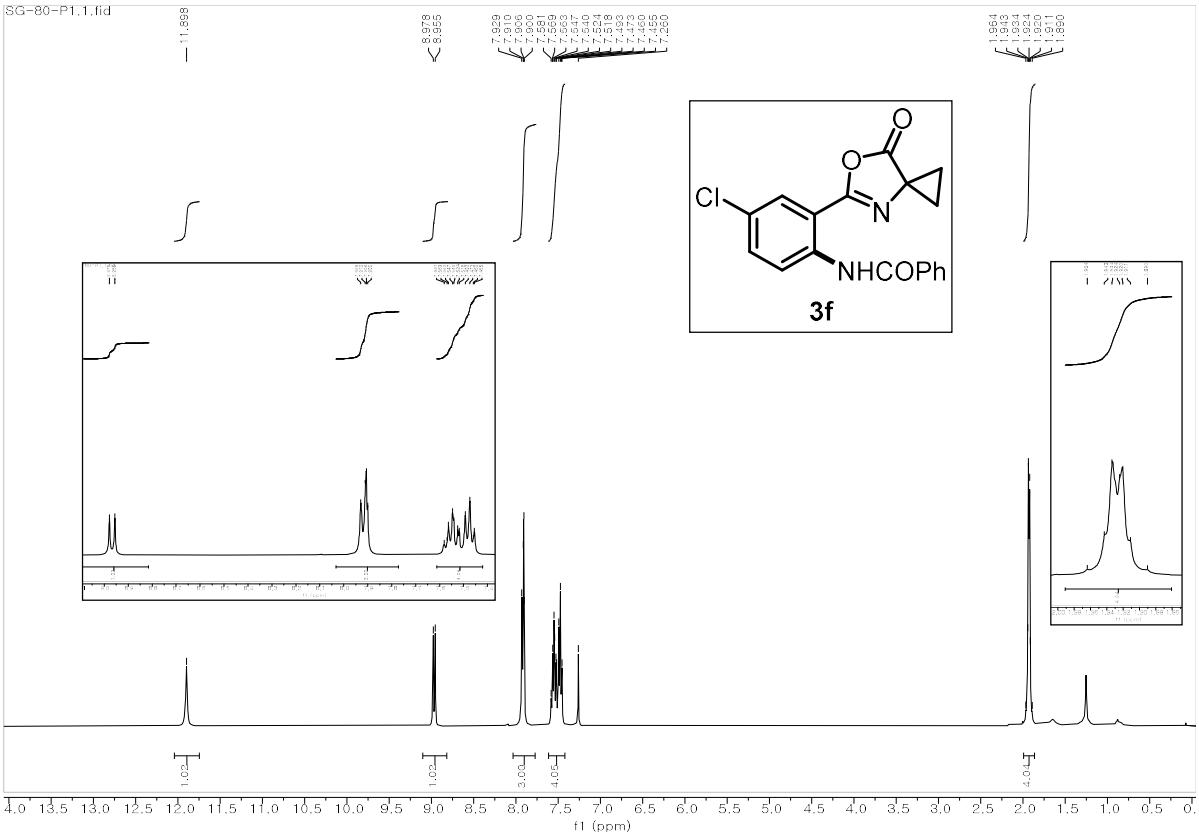

SG85-P1
single_pulse
105.199
O
O
N
F3C
NHCOPh
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

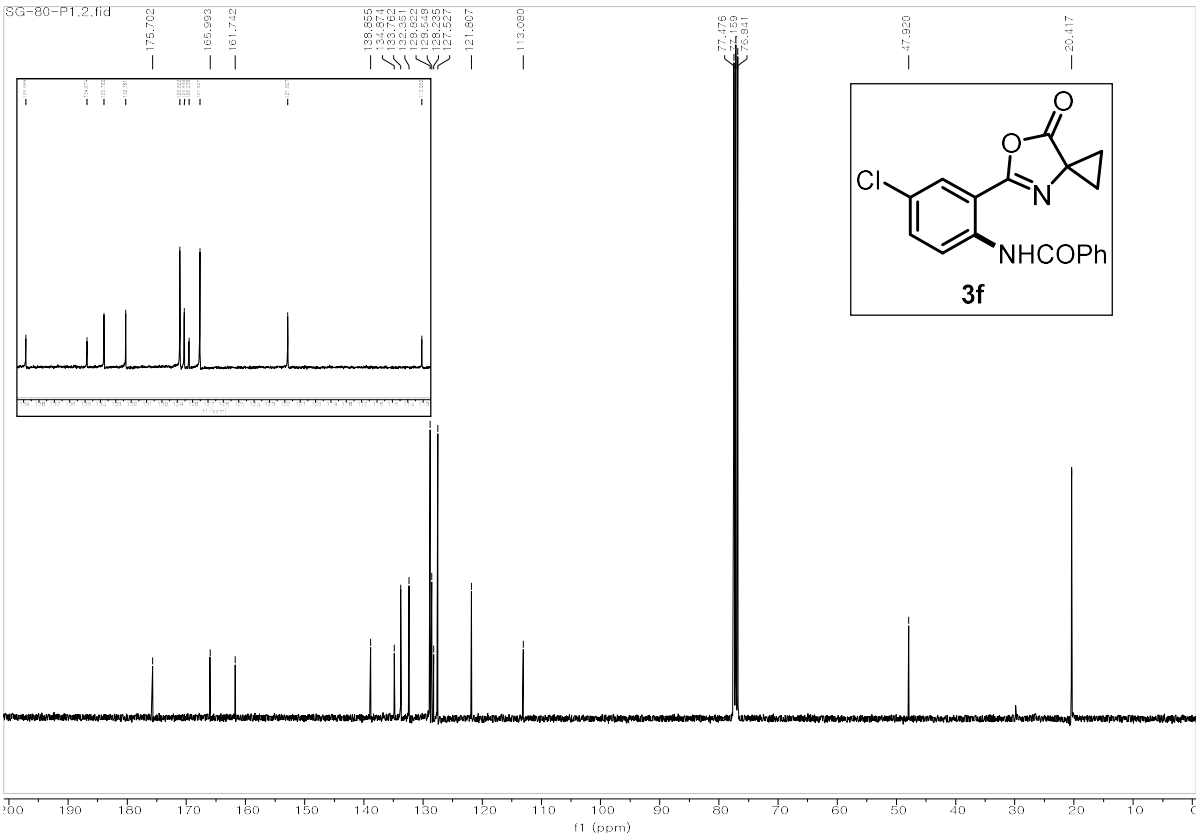

SG57-P1_10.fid
SG57-P1_H_180099
NHCOPh
3d
f1 (ppm)

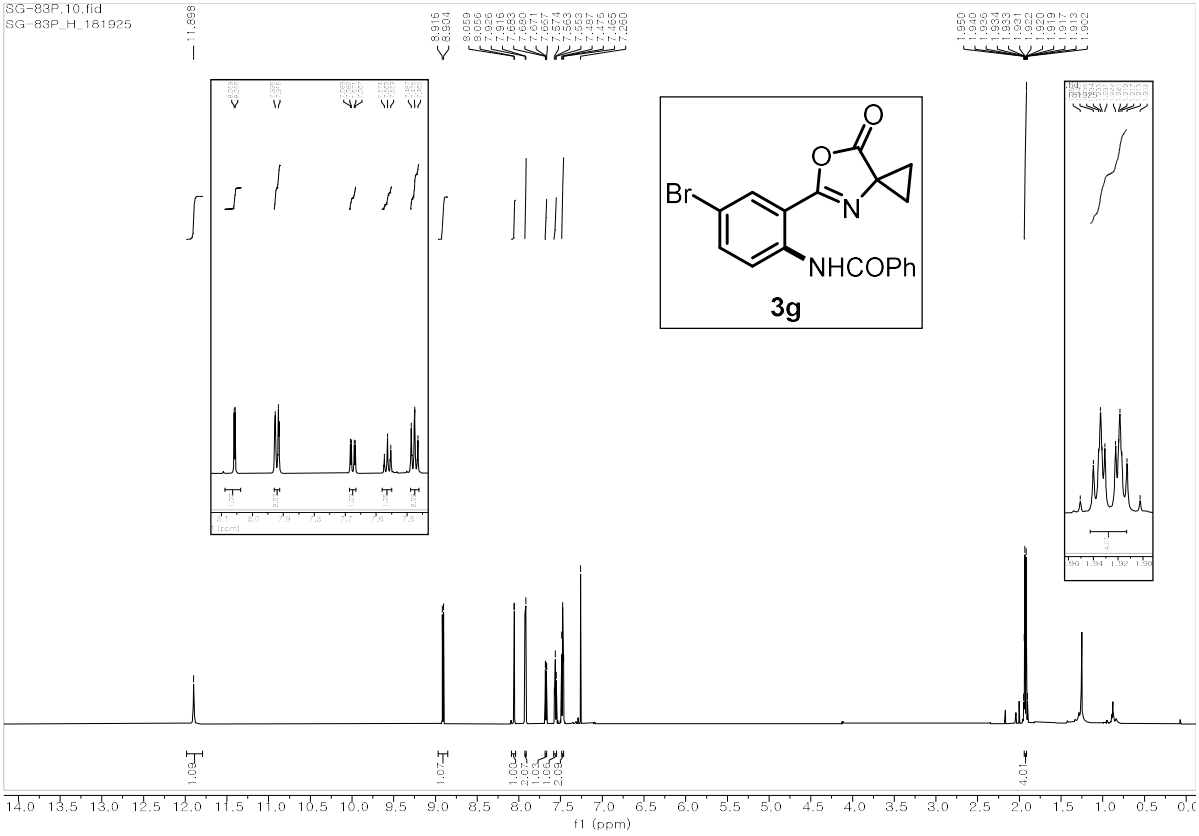

SG57-P1_11.fid
SG57-P1_C_180099
NHCOPh
3d
f1 (ppm)

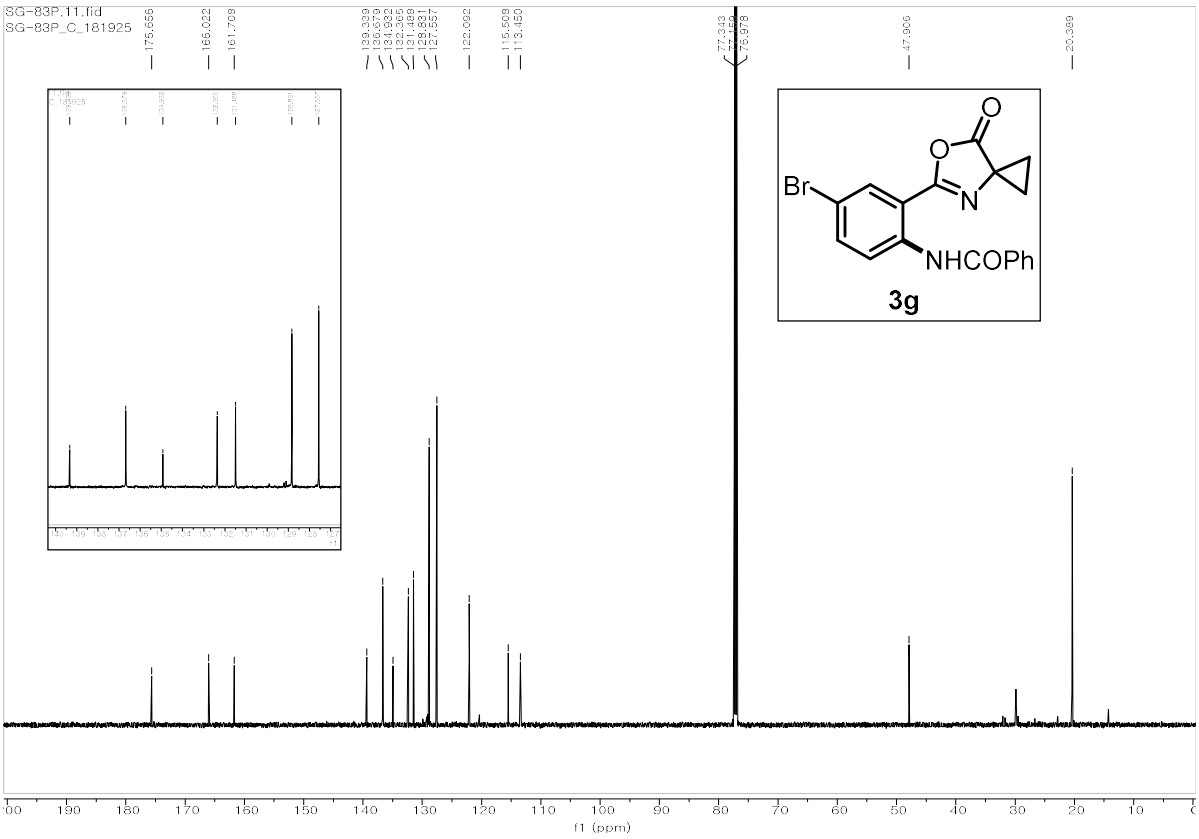

SG-82-P.1.fid
11.659
8.675
8.653
7.742
7.739
7.735
7.727
7.722
7.718
7.624
7.368
7.365
7.346
7.340
7.334
7.325
7.312
7.279
7.260
7.242
7.226
7.196
7.071
2.181
1.706
1.696
1.691
1.681
Me
NHCOPh
3e
f1 (ppm)

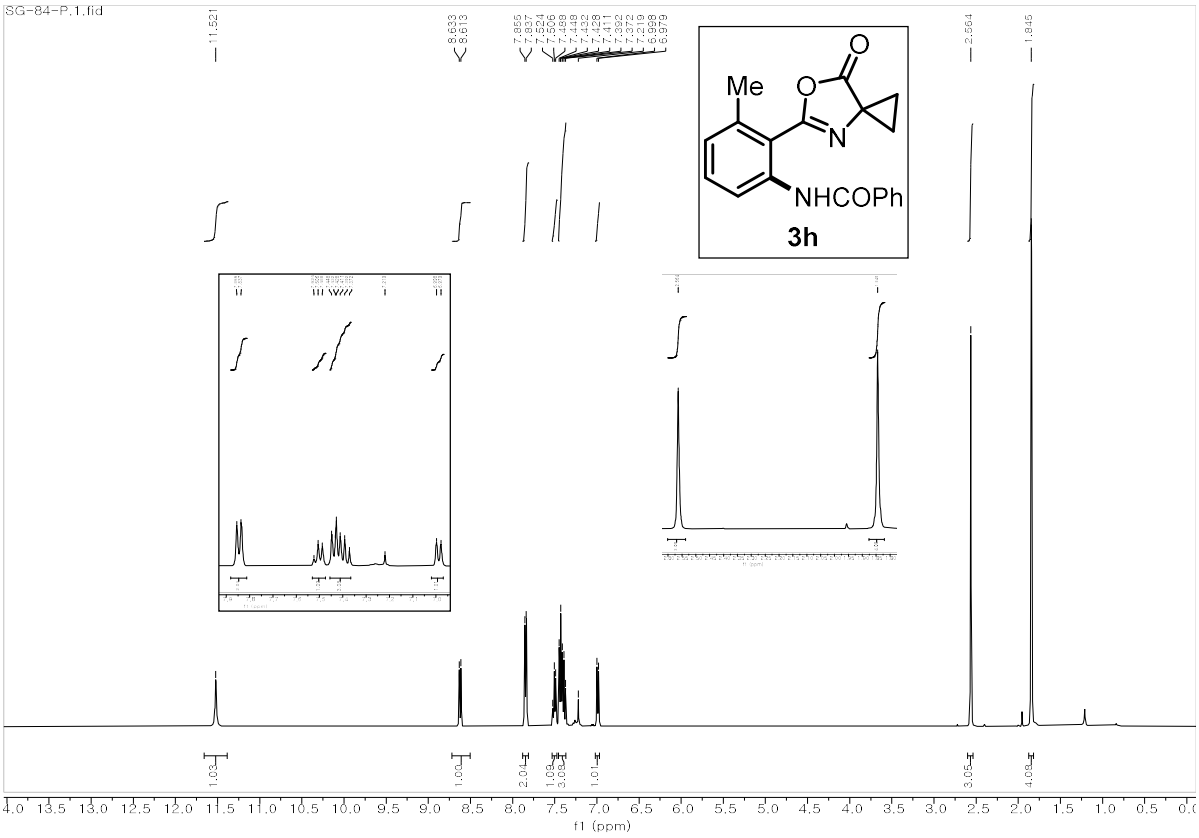

SG-82-P.2.fid
176.421
165.790
162.629
137.933
136.250
134.667
132.724
131.987
129.689
128.677
127.474
120.346
111.677
77.477
77.160
76.842
47.735
20.859
20.104
Me
NHCOPh
3e
f1 (ppm)

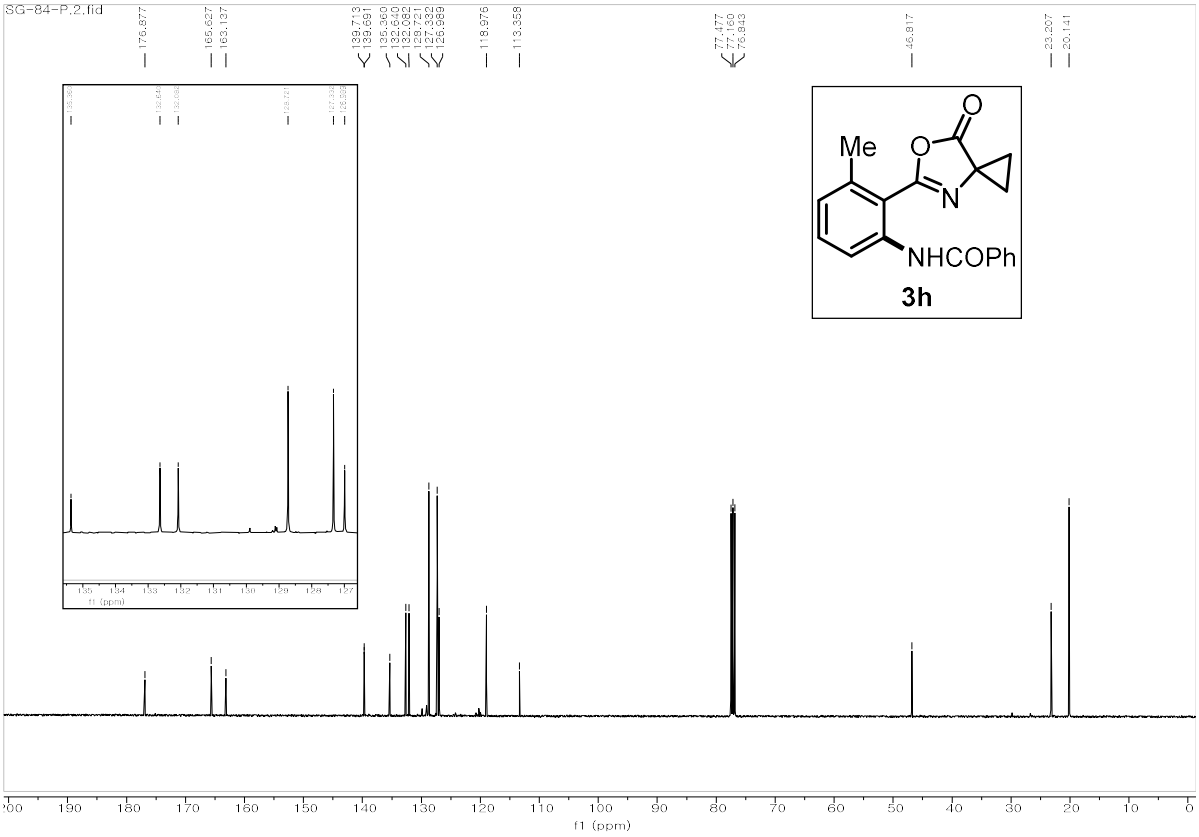

SG-80-P1.1.fid
11.998
8.978
8.955
7.929
7.910
7.906
7.900
7.581
7.569
7.563
7.537
7.540
7.524
7.518
7.493
7.473
7.460
7.455
7.260
1.964
1.943
1.934
1.924
1.911
1.890
1.03
1.02
3.00
4.05
4.04
f1 (ppm)
Cl
O
O
N
NHCOPh
3f

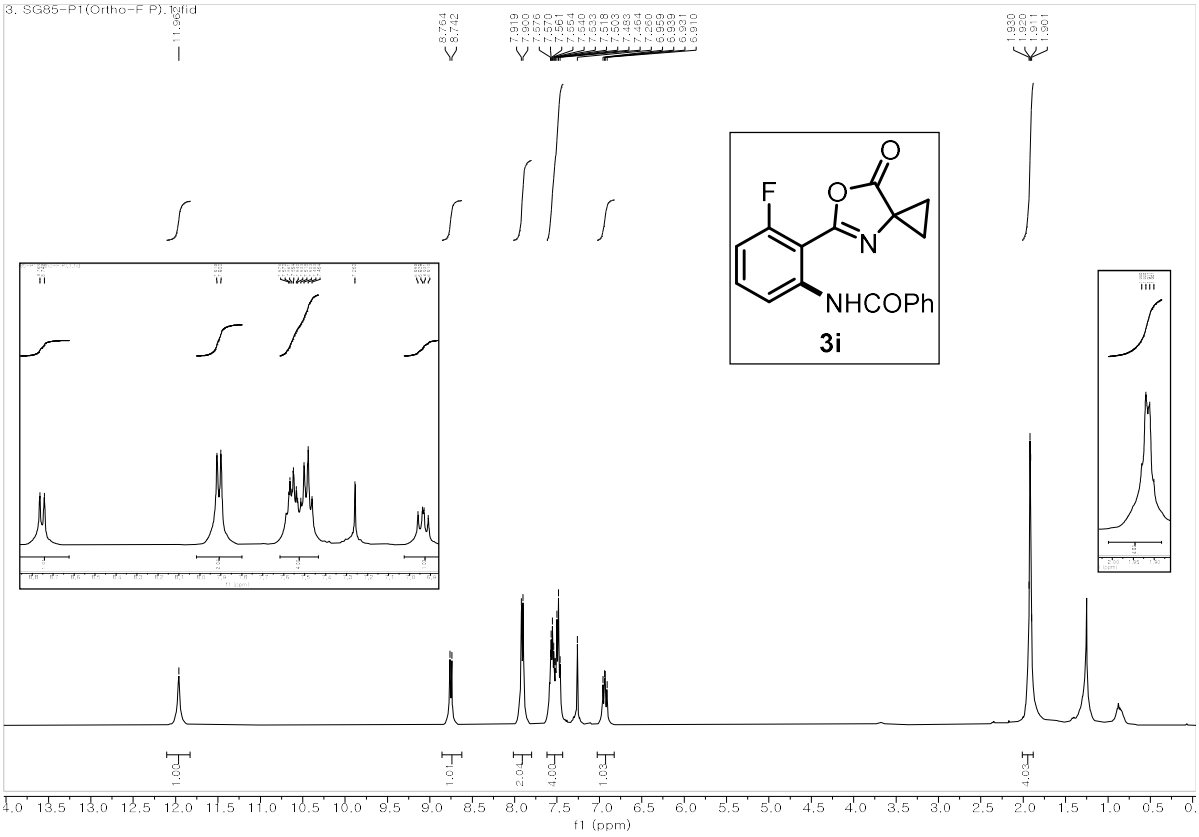

SG-80-P1.2.fid
175.702
165.993
161.742
138.855
134.574
133.571
132.351
129.822
128.549
128.235
127.327
121.807
113.080
77.476
77.159
76.841
47.920
20.417
f1 (ppm)
Cl
O
O
N
NHCOPh
3f

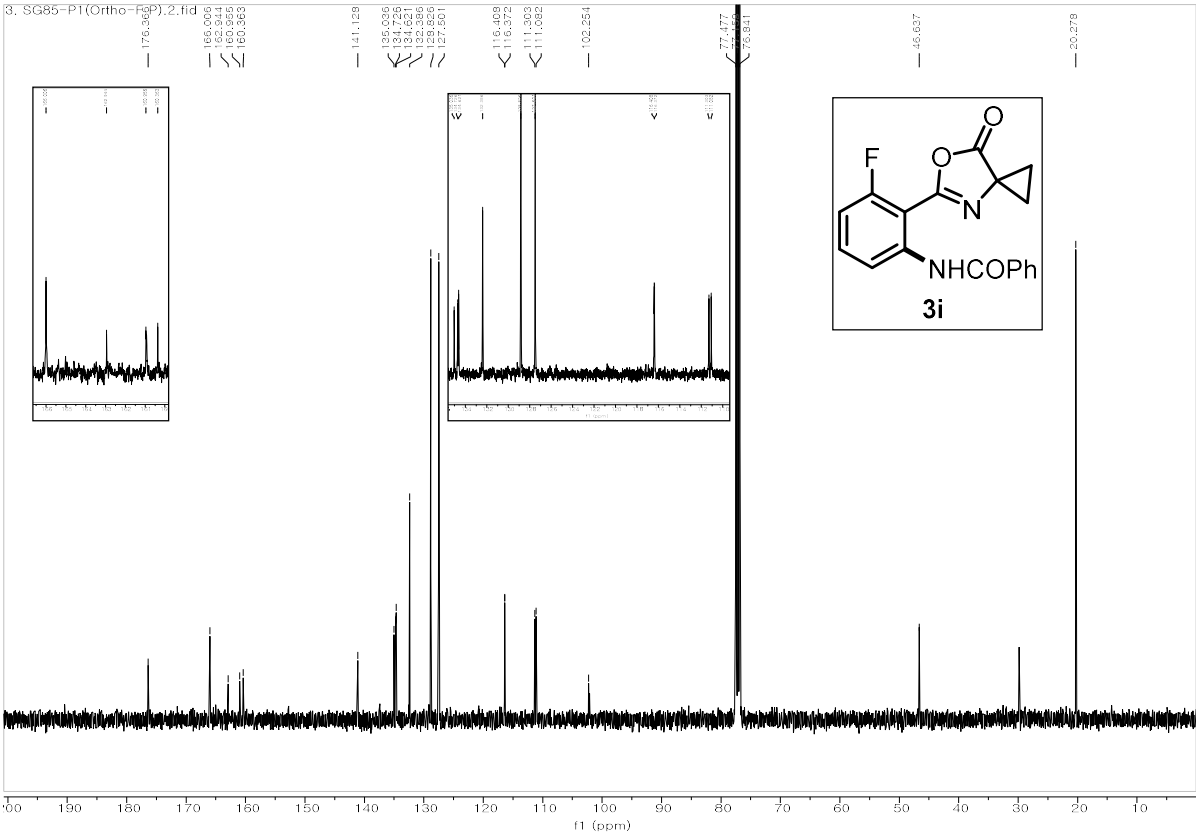

SG-83P.10.fid
SG-83P_H_181925
11.898
8.916
8.904
8.059
8.056
8.038
7.916
7.683
7.680
7.677
7.671
7.574
7.563
7.557
7.475
7.465
7.260
1.950
1.940
1.936
1.934
1.933
1.931
1.922
1.920
1.919
1.917
1.913
1.902
Br
O
O
N
NHCOPh
3g
1.00
1.07
1.00
2.07
1.02
1.05
2.00
4.01
f1 (ppm)

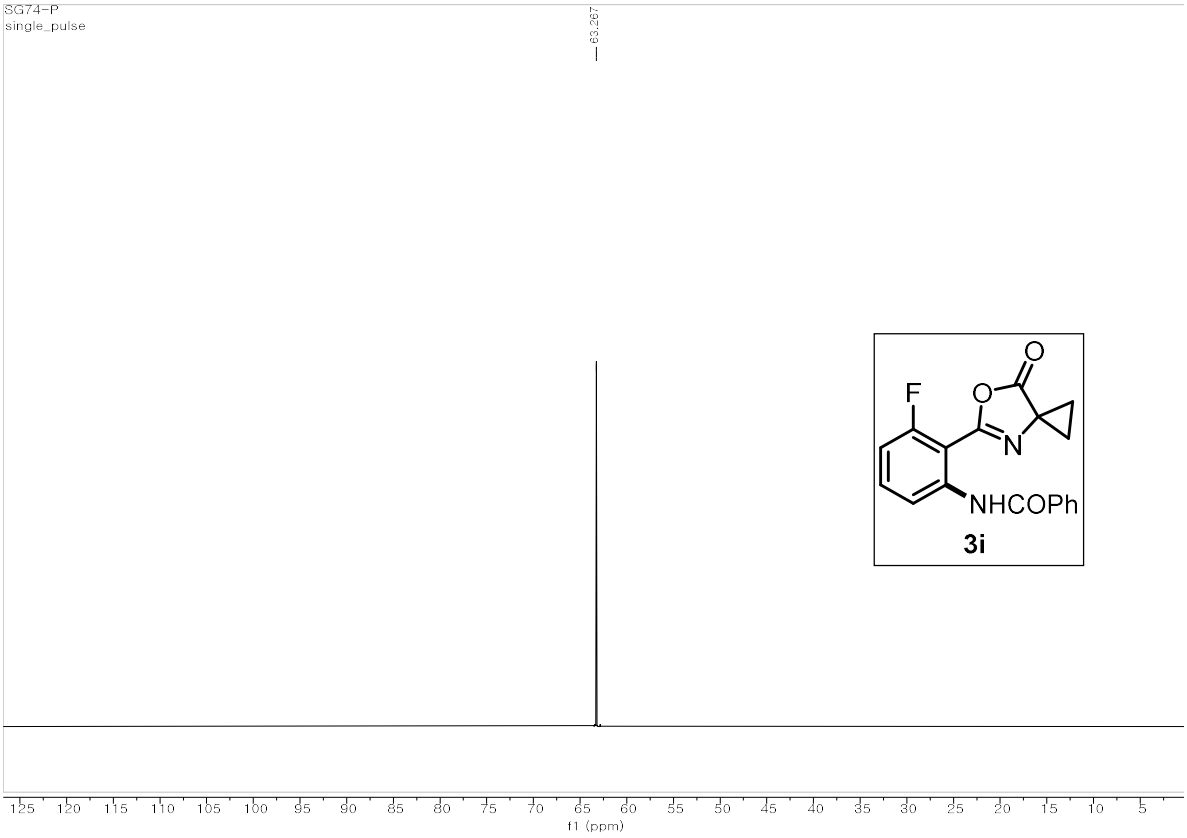

SG-83P.11.fid
SG-83P_C_181925
175.656
166.022
161.709
139.339
136.679
134.982
132.365
131.489
129.831
127.557
122.092
116.508
113.450
77.343
77.156
76.978
47.906
20.389
Br
O
O
N
NHCOPh
3g
f1 (ppm)

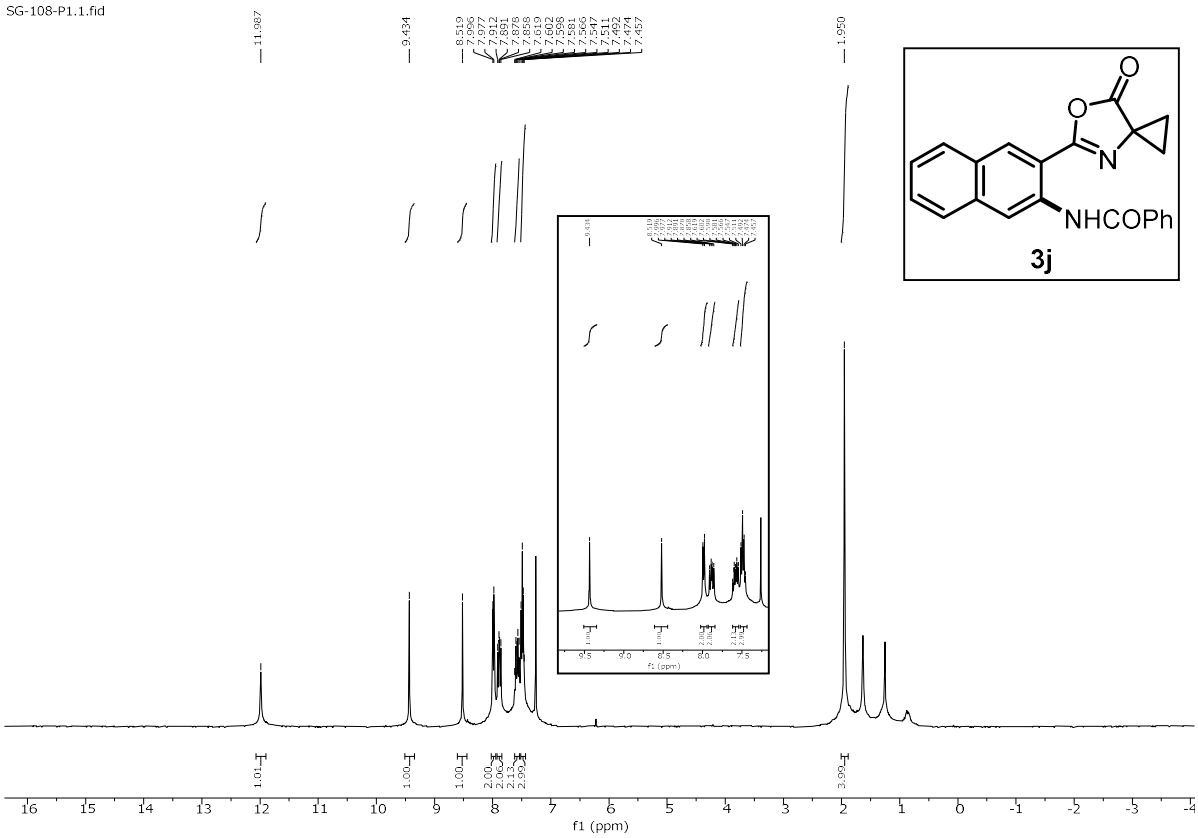

SG-84-P.1.fid
11.521
8.633
8.613
7.855
7.837
7.534
7.506
7.488
7.432
7.411
7.408
7.392
7.372
7.219
6.998
6.979
2.564
1.845
Me
O
O
N
NHCOPh
3h
1.03
1.00
2.04
1.05
3.00
1.01
3.05
4.00
f1 (ppm)


SG-84-P.2.fid
176.877
165.627
163.137
139.713
139.691
135.360
132.640
132.582
128.721
127.332
126.989
118.976
113.358
77.477
77.160
76.843
46.817
23.207
20.141
Me
O
O
N
NHCOPh
3h
f1 (ppm)

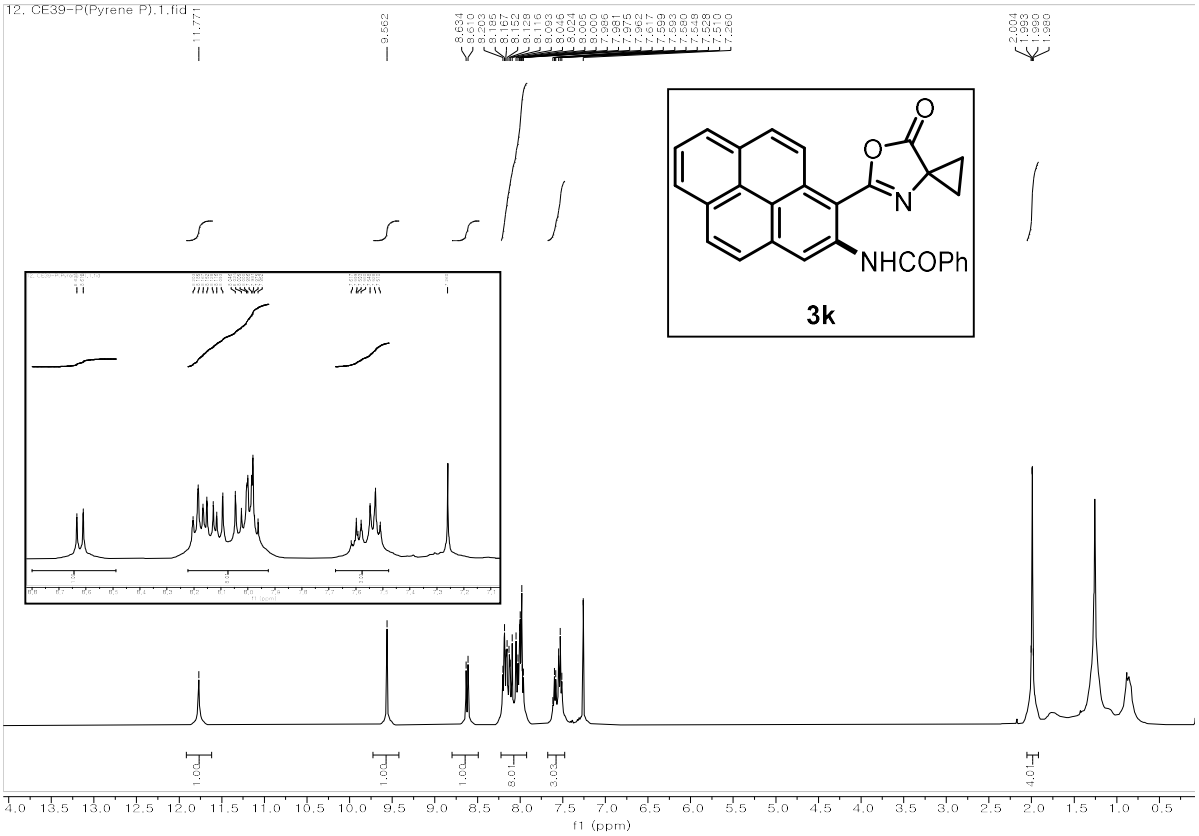

3. SG85-P1(Ortho-F P).1.fid
F
O
O
N
NHCOPh
3i
f1 (ppm)

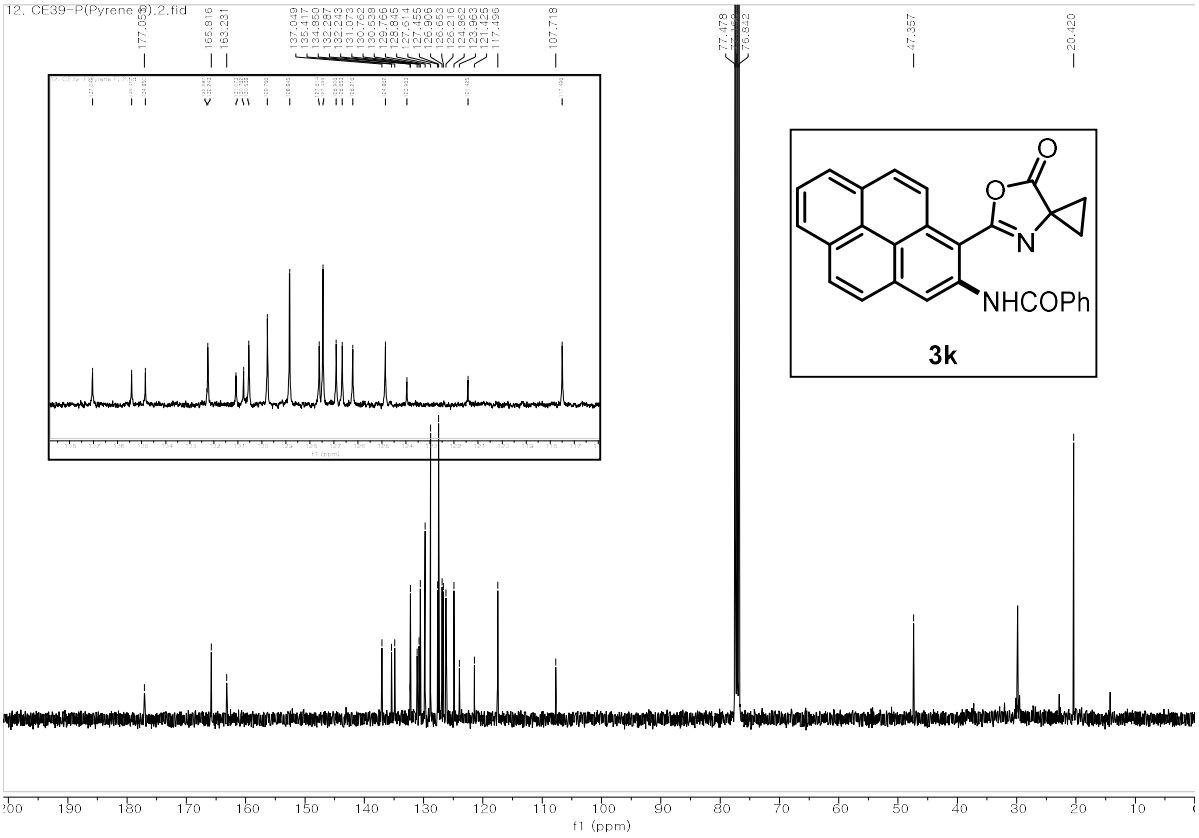

3. SG85-P1(Ortho-F@P).2.fid
F
O
O
N
NHCOPh
3i
f1 (ppm)

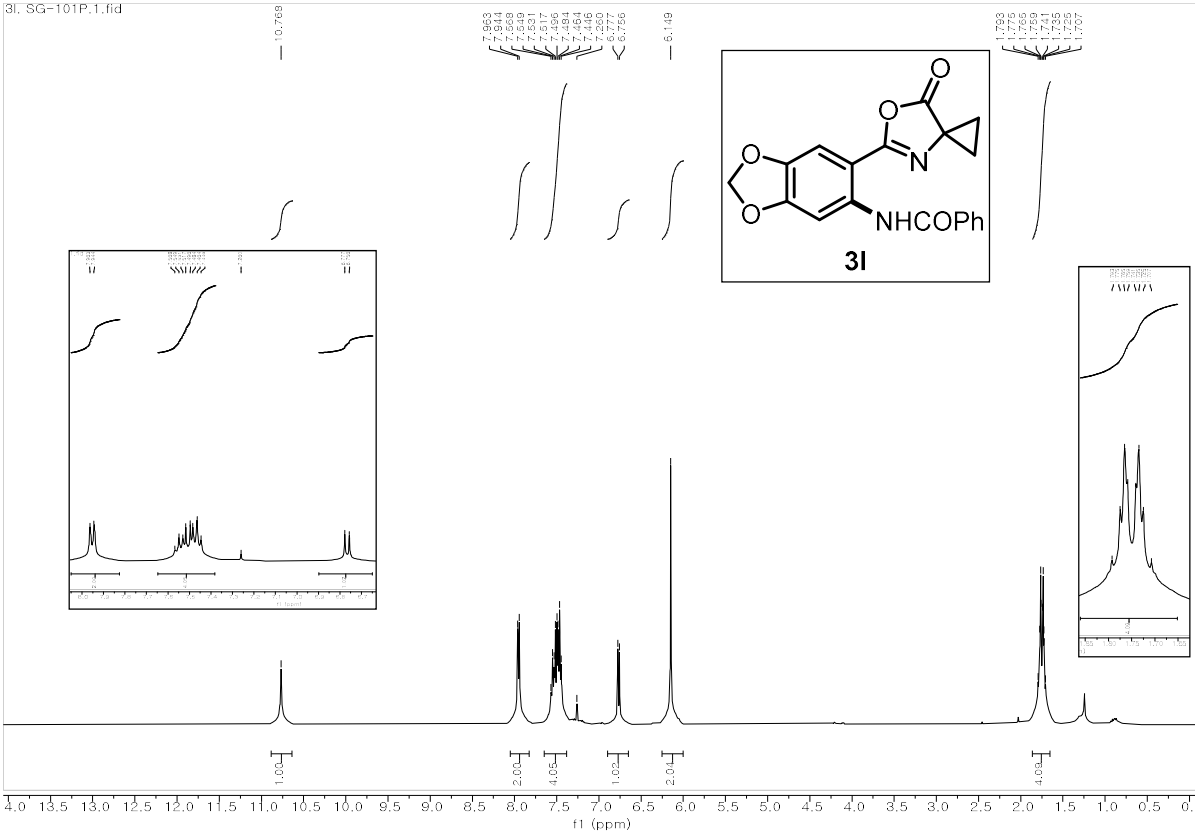

SG74-P
single_pulse
63.267
F
O
O
N
NHCOPh
3i
125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)

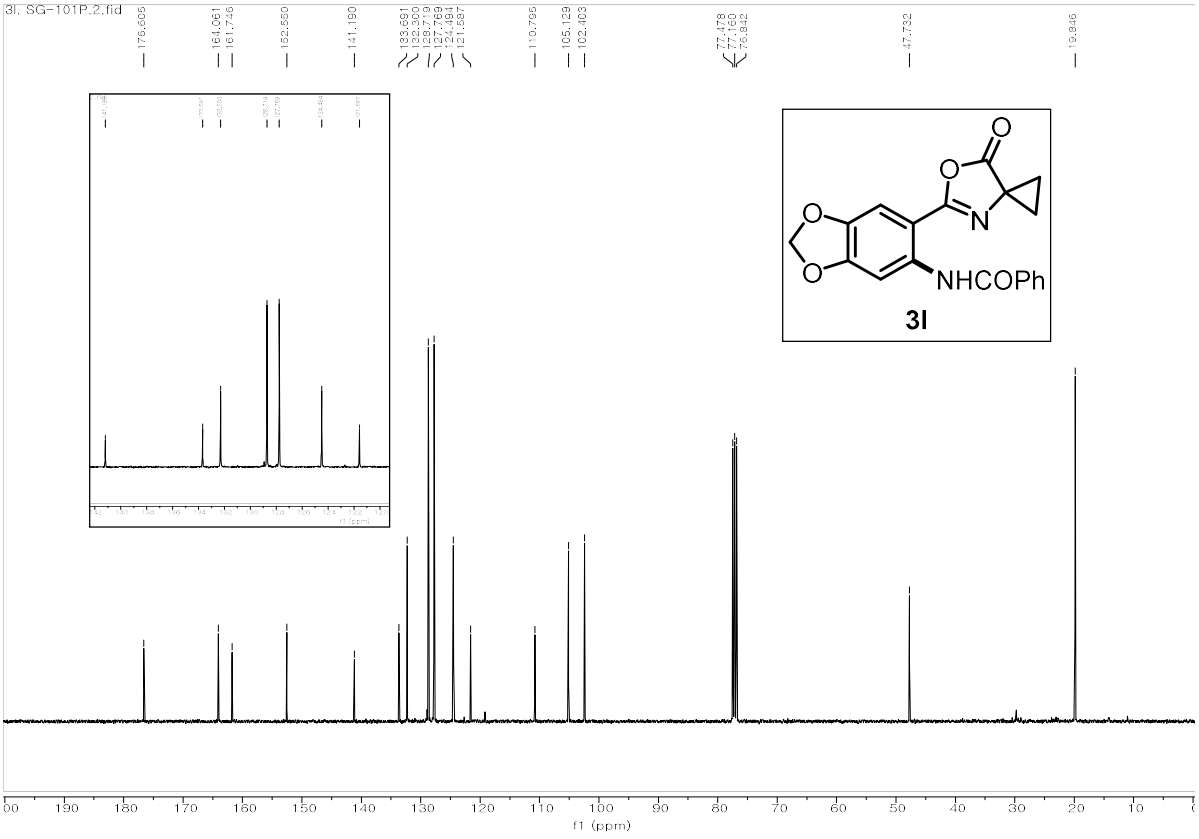

SG-108-P1.1.fid
11.987
9.434
8.519
7.996
7.977
7.912
7.891
7.875
7.858
7.619
7.602
7.588
7.581
7.566
7.547
7.511
7.492
7.474
7.457
1.950
1.01
1.00
1.00
2.00
2.06
2.13
2.99
3.99
f1 (ppm)
NHCOPh
3j

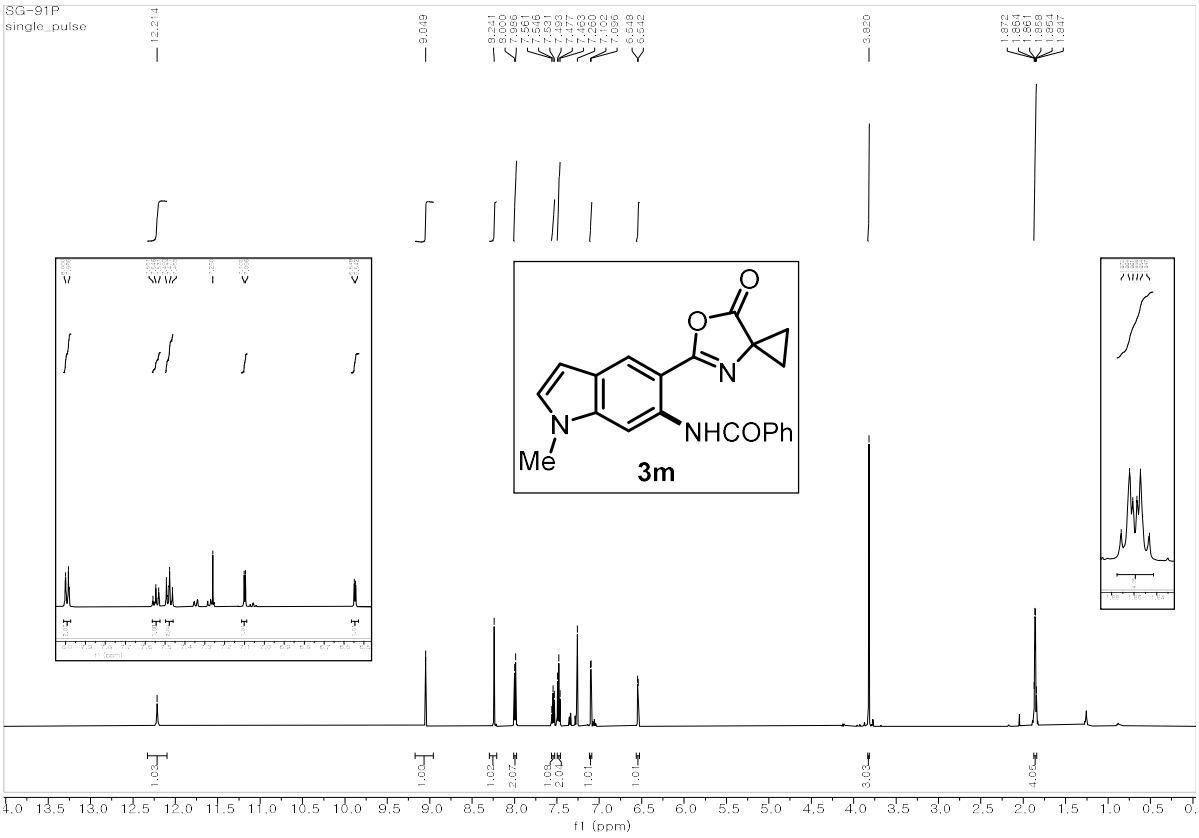

SG-108-P1.2.fid
176.189
166.030
162.677
136.104
135.432
135.228
132.050
131.221
129.461
128.974
128.795
128.764
128.096
127.483
126.021
117.790
112.588
77.478
77.160 CDCl3
76.843
48.018
20.270
f1 (ppm)
NHCOPh
3j

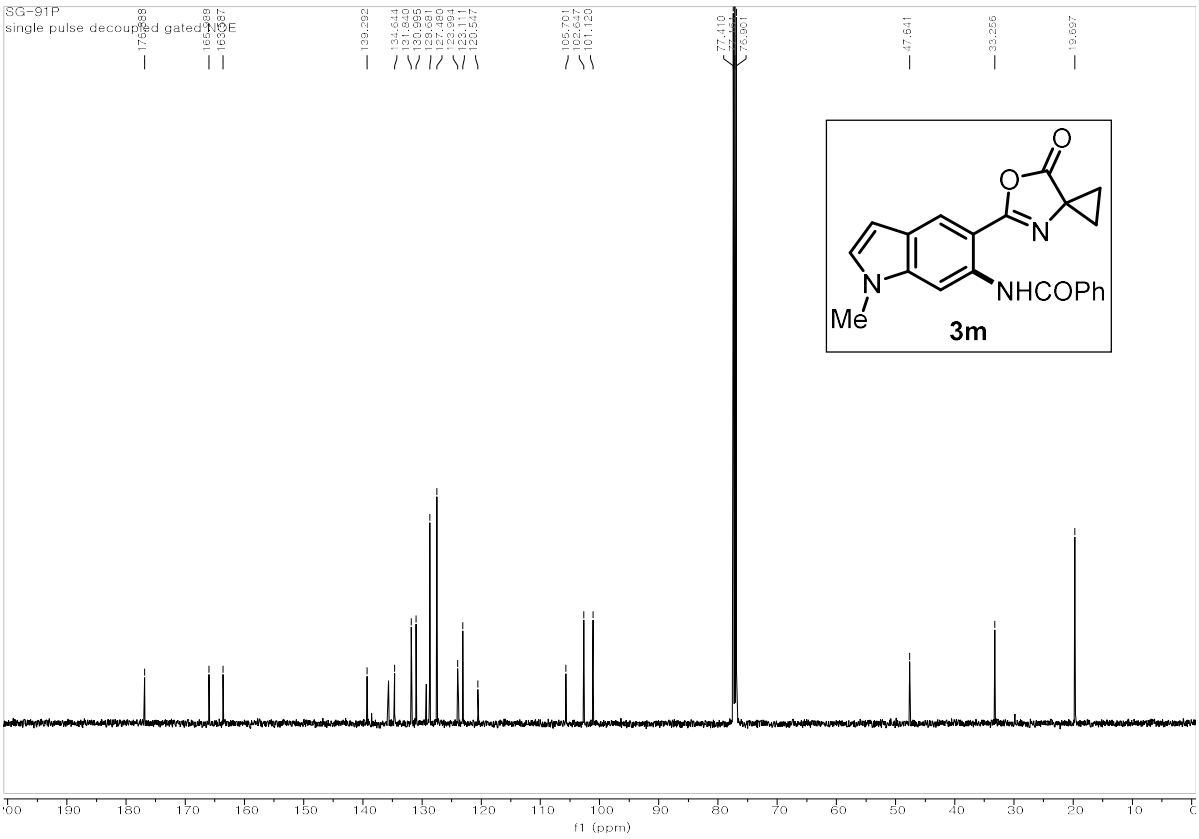

12, CE39-P(Pyrene P).1.fid
11.771
9.562
8.684
8.611
8.203
8.185
8.167
8.152
8.128
8.116
8.093
8.046
8.004
8.000
7.986
7.981
7.975
7.967
7.617
7.599
7.593
7.588
7.548
7.528
7.510
7.260
2.004
1.993
1.990
1.988
NHCOPh
3k
1.00
1.00
1.00
8.01
3.03
4.01
f1 (ppm)

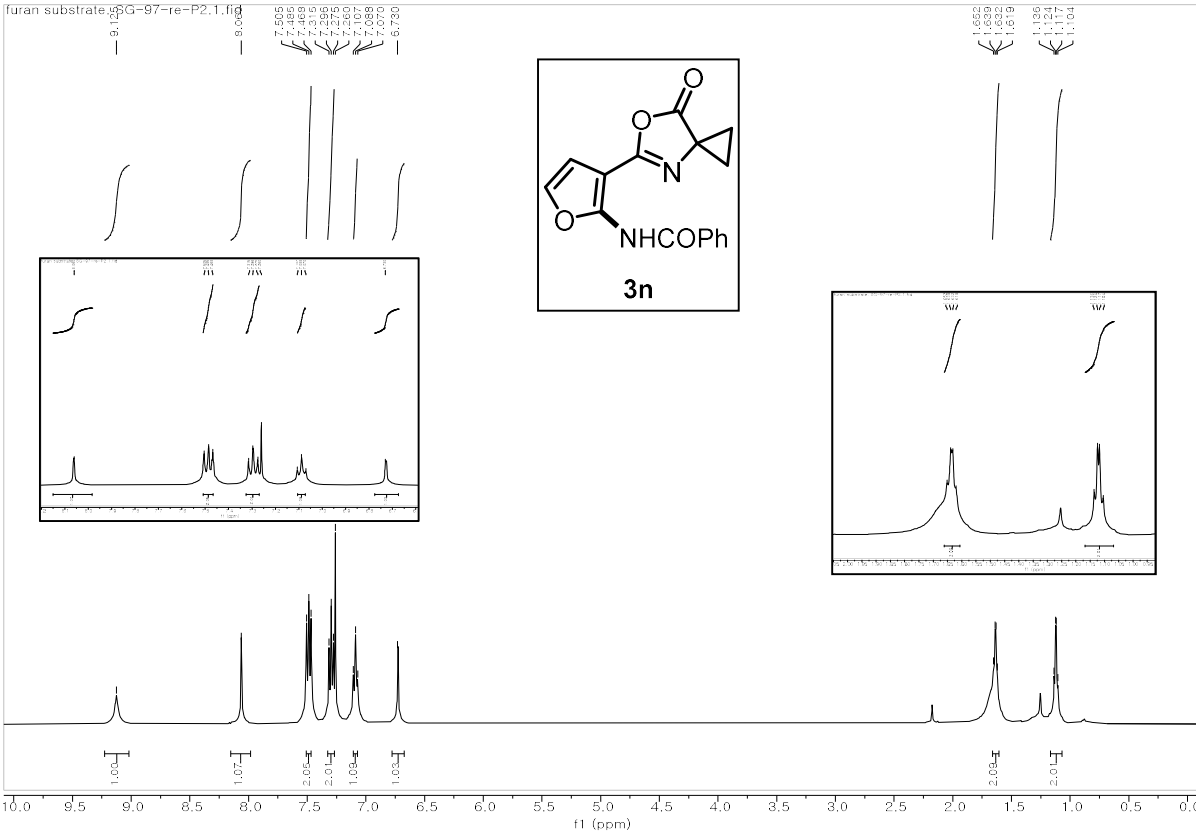

12, CE39-P(Pyrene P).2.fid
177.053
165.916
163.231
137.049
135.417
134.850
132.287
132.243
131.073
130.752
130.539
128.766
128.845
127.614
127.415
126.956
126.653
124.962
124.863
121.425
117.496
107.718
77.478
77.158
76.842
47.357
20.420
NHCOPh
3k
f1 (ppm)

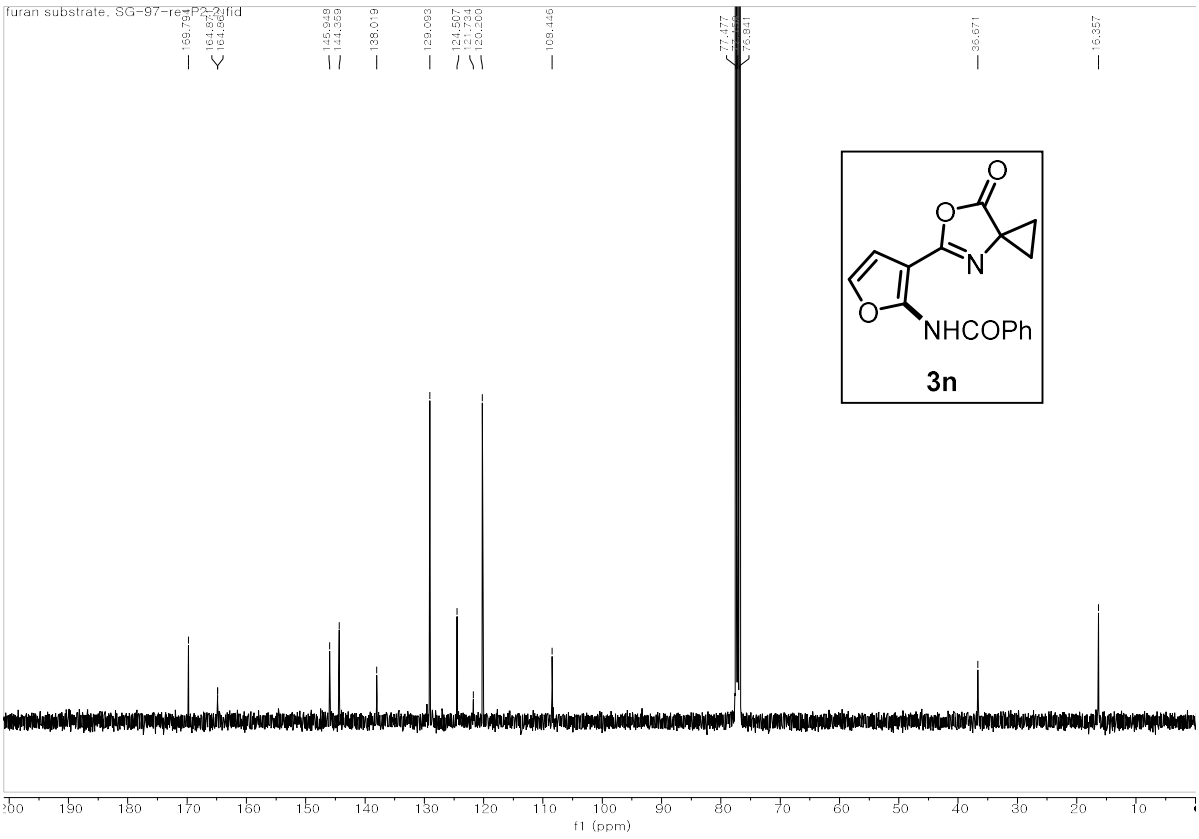

3l. SG-101P.1.fid
10.768
7.963
7.944
7.559
7.549
7.531
7.517
7.496
7.484
7.464
7.446
7.429
6.777
6.756
6.149
1.793
1.775
1.766
1.759
1.741
1.735
1.725
1.707
NHCOPh
3l
f1 (ppm)

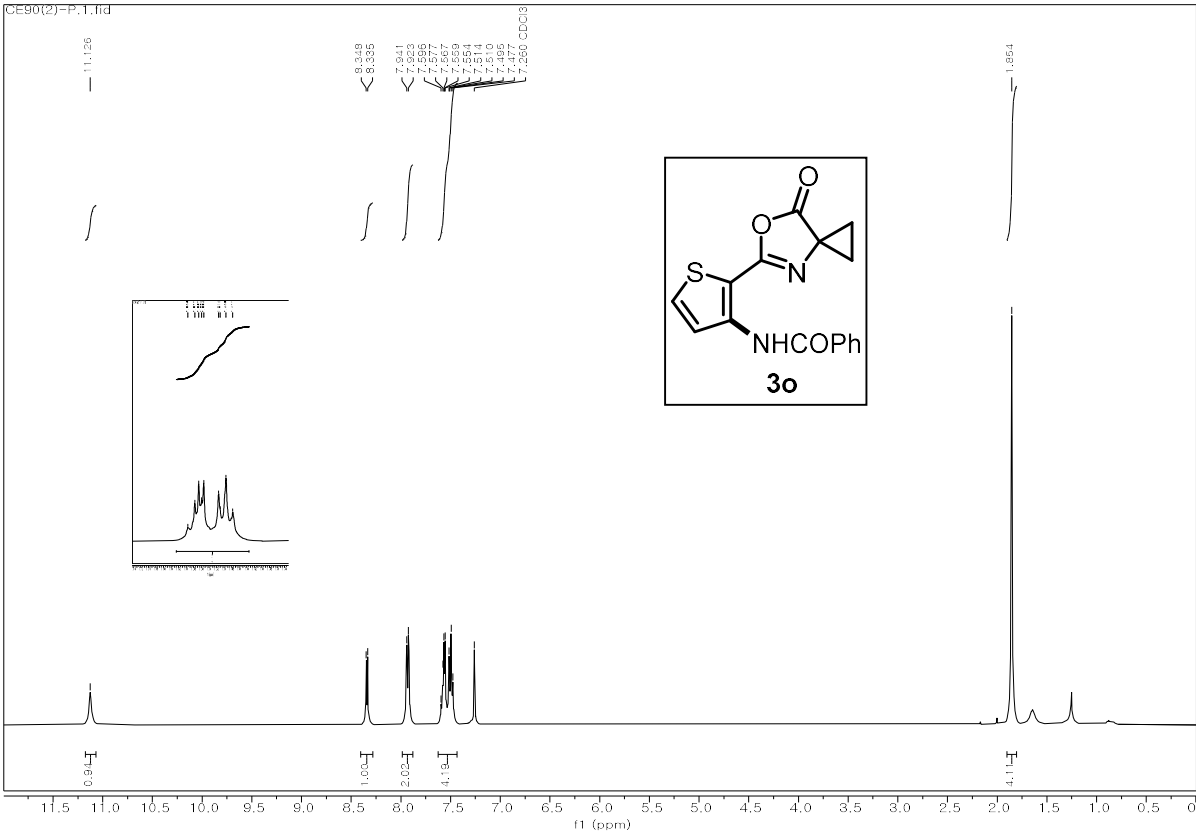

3l. SG-101P.2.fid
176.605
164.061
161.746
152.550
141.190
133.681
132.300
128.719
127.769
124.494
121.587
110.795
105.129
102.403
77.478
77.160
76.842
47.732
19.846
NHCOPh
3l
f1 (ppm)

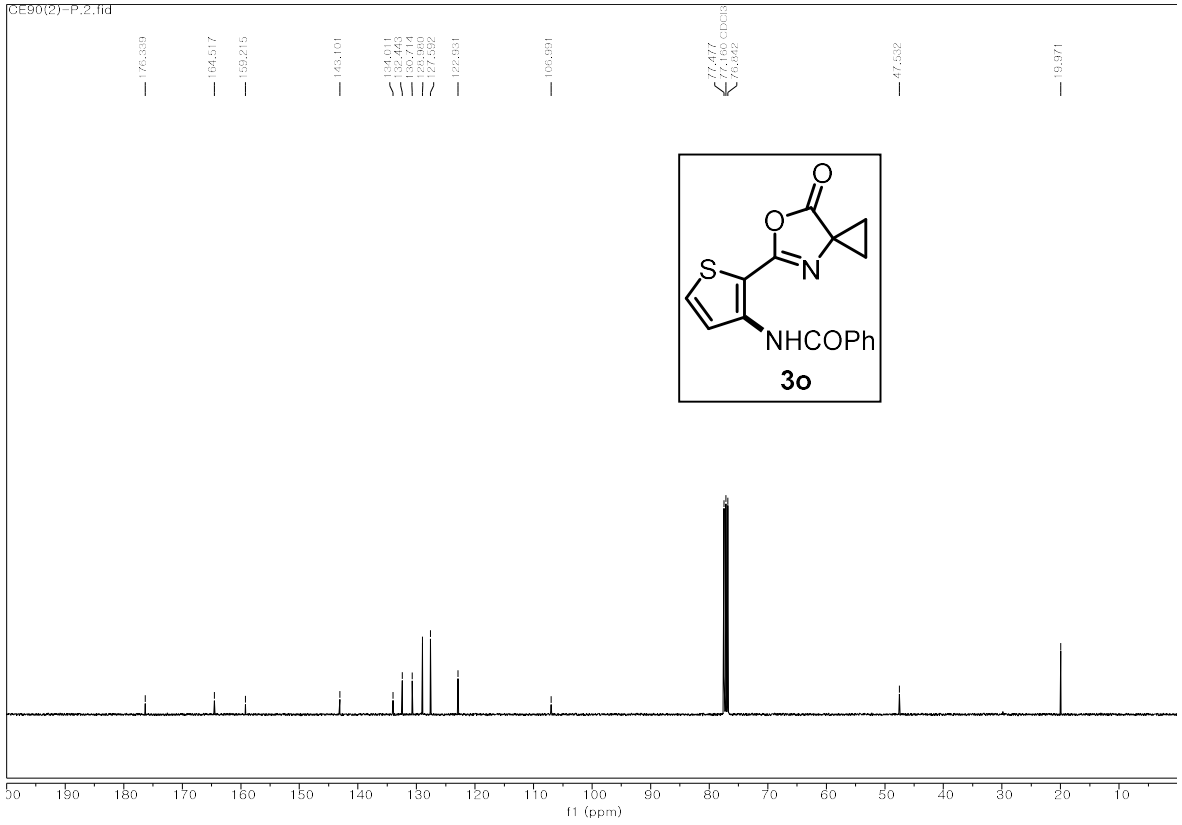

SG-91P
single_pulse
12.214
9.049
8.241
8.000
7.986
7.561
7.546
7.531
7.477
7.463
7.260
7.026
6.549
6.542
3.820
1.872
1.864
1.861
1.853
1.854
1.847
O
O
N
N
Me
NHCOPh
3m
f1 (ppm)

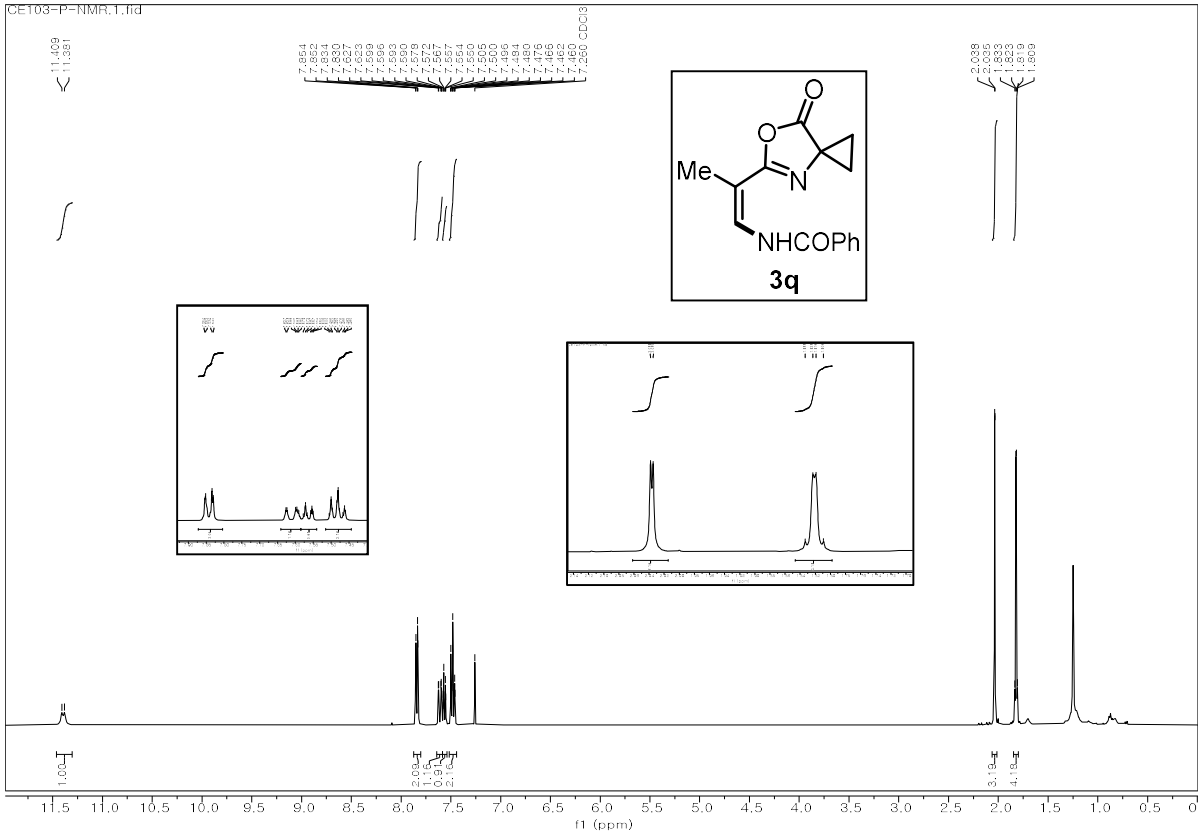

SG-91P
single pulse decoupled gated NOE
176.988
165.389
163.987
139.292
134.644
131.440
130.995
129.681
127.480
123.994
123.111
120.547
105.701
102.647
101.120
77.410
76.991
47.641
33.256
19.697
O
O
N
N
Me
NHCOPh
3m
f1 (ppm)

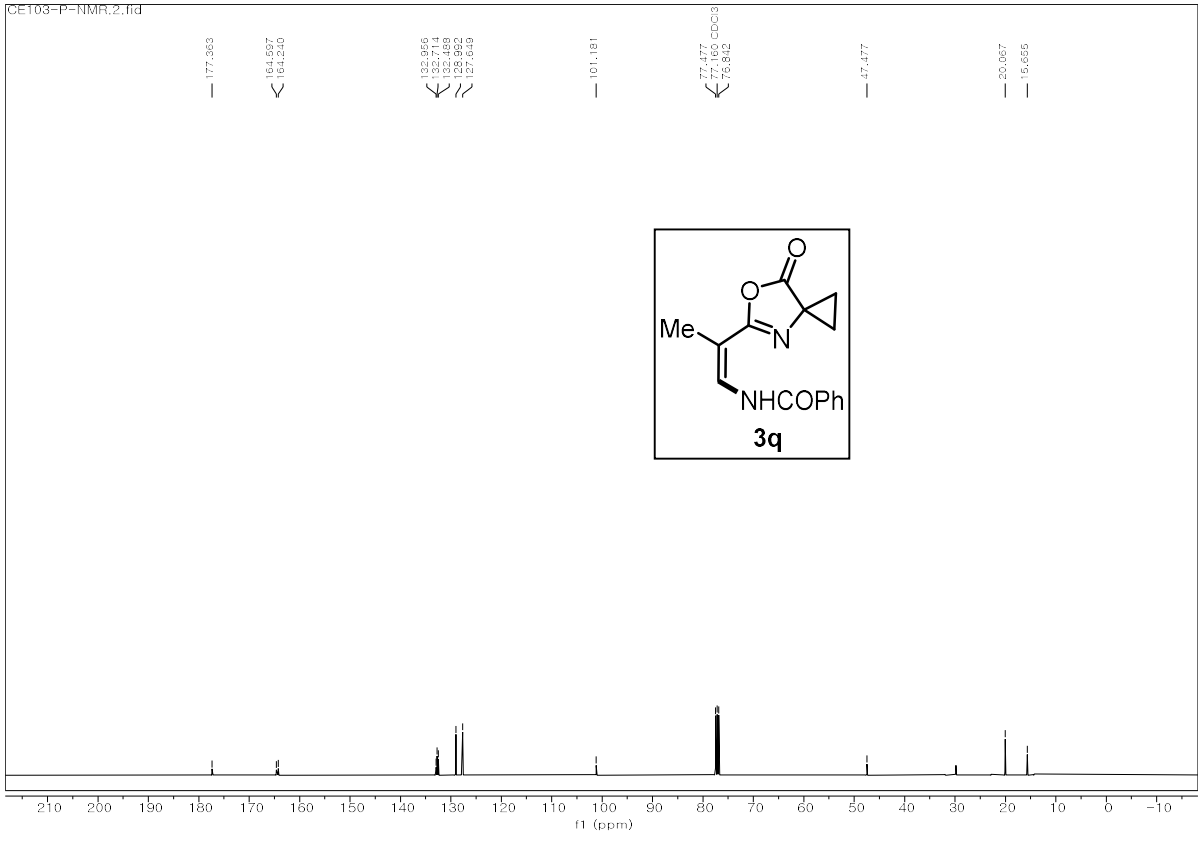

furan substrate, SG-97-re-P2.1.fid
NHCOPh
3n
f1 (ppm)

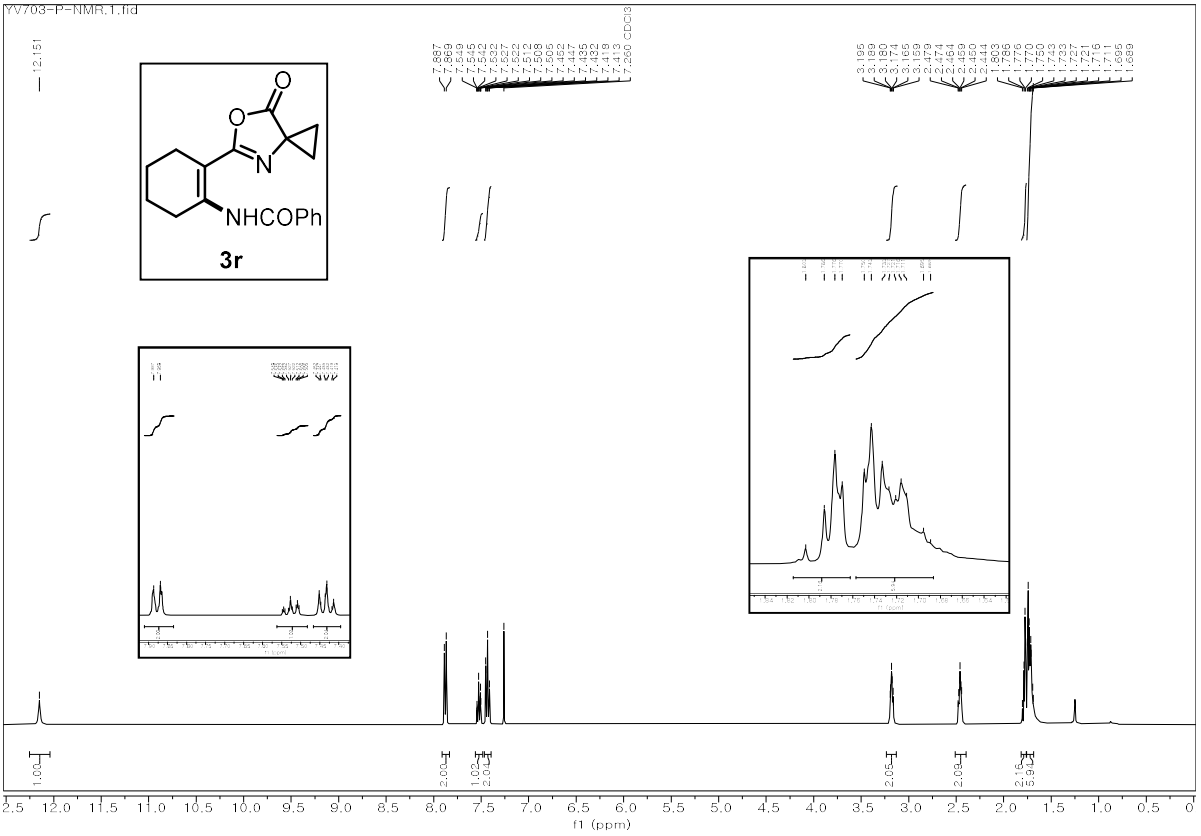

furan substrate, SG-97-re-P3.2.fid
NHCOPh
3n
f1 (ppm)

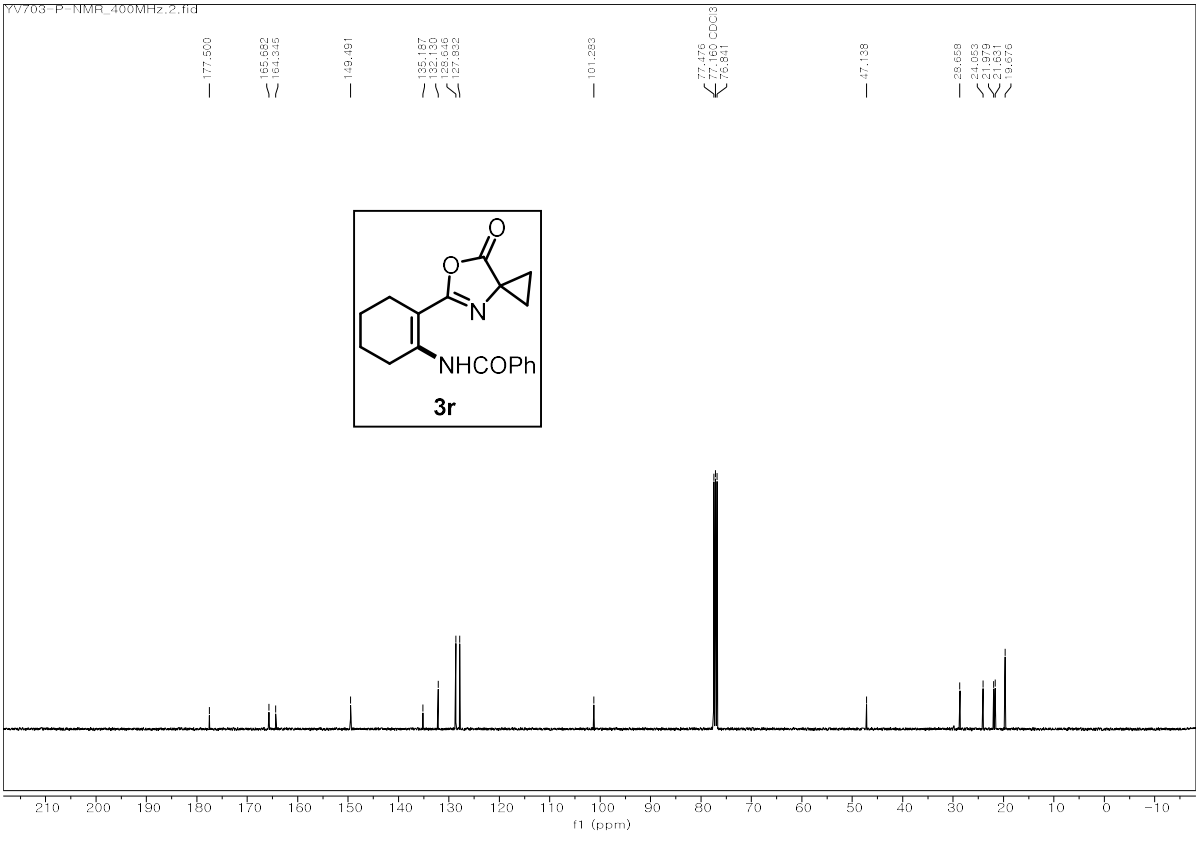

CE90(2)-P.1.fid
11.126
8.348
8.335
7.941
7.923
7.596
7.577
7.567
7.559
7.554
7.514
7.510
7.495
7.477
7.260 CDCl3
1.864
NHCOPh
3o
0.94
1.00
2.02
4.19
4.11
f1 (ppm)


CE90(2)-P.2.fid
175.339
164.517
159.215
143.191
134.911
132.443
130.714
129.060
127.592
122.931
105.991
77.477
77.160 CDCl3
76.842
47.532
19.971
NHCOPh
3o
f1 (ppm)

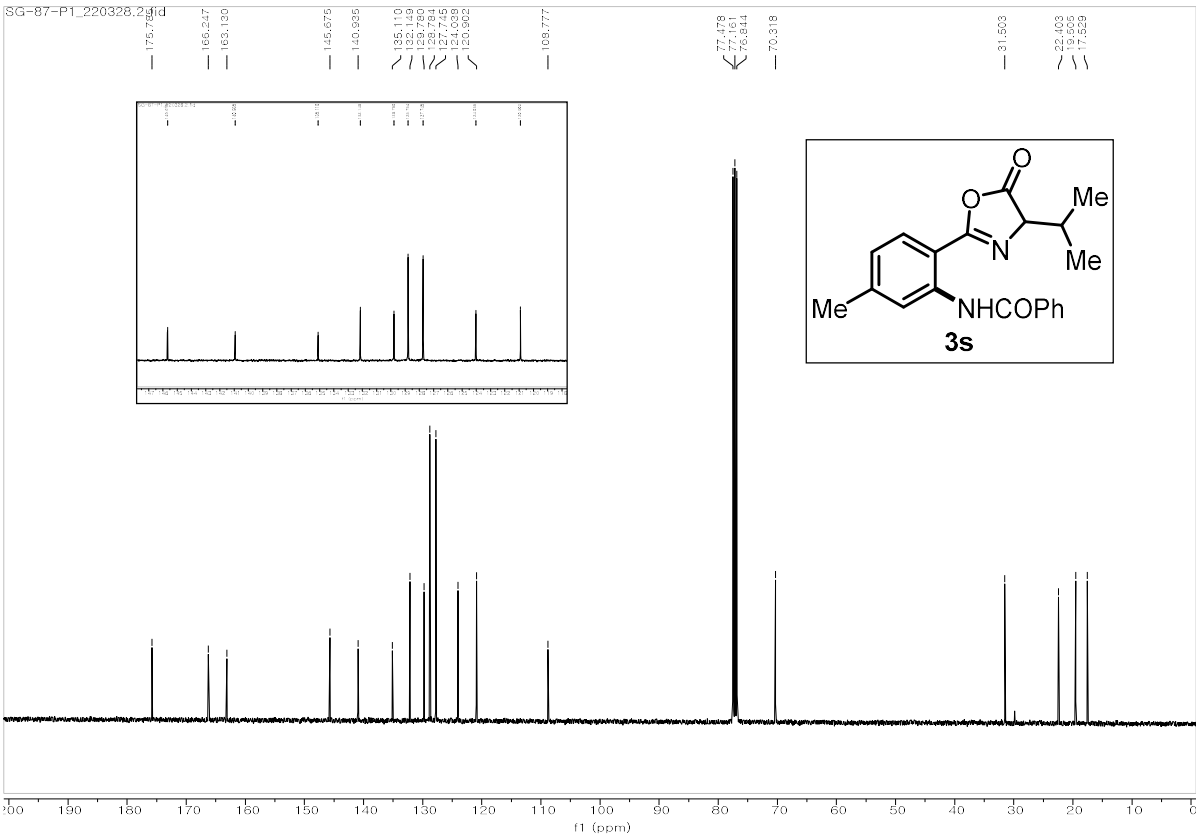

CE103-P-NMR.1.fid
11.409
11.381
7.864
7.862
7.834
7.830
7.627
7.623
7.599
7.596
7.593
7.590
7.579
7.572
7.567
7.564
7.560
7.505
7.500
7.496
7.484
7.480
7.476
7.466
7.460
7.260 CDCl3
2.038
2.035
1.833
1.823
1.819
1.809
Me
O
O
N
NHCOPh
3q
1.00
2.09
1.16
0.91
2.16
3.19
4.18
f1 (ppm)

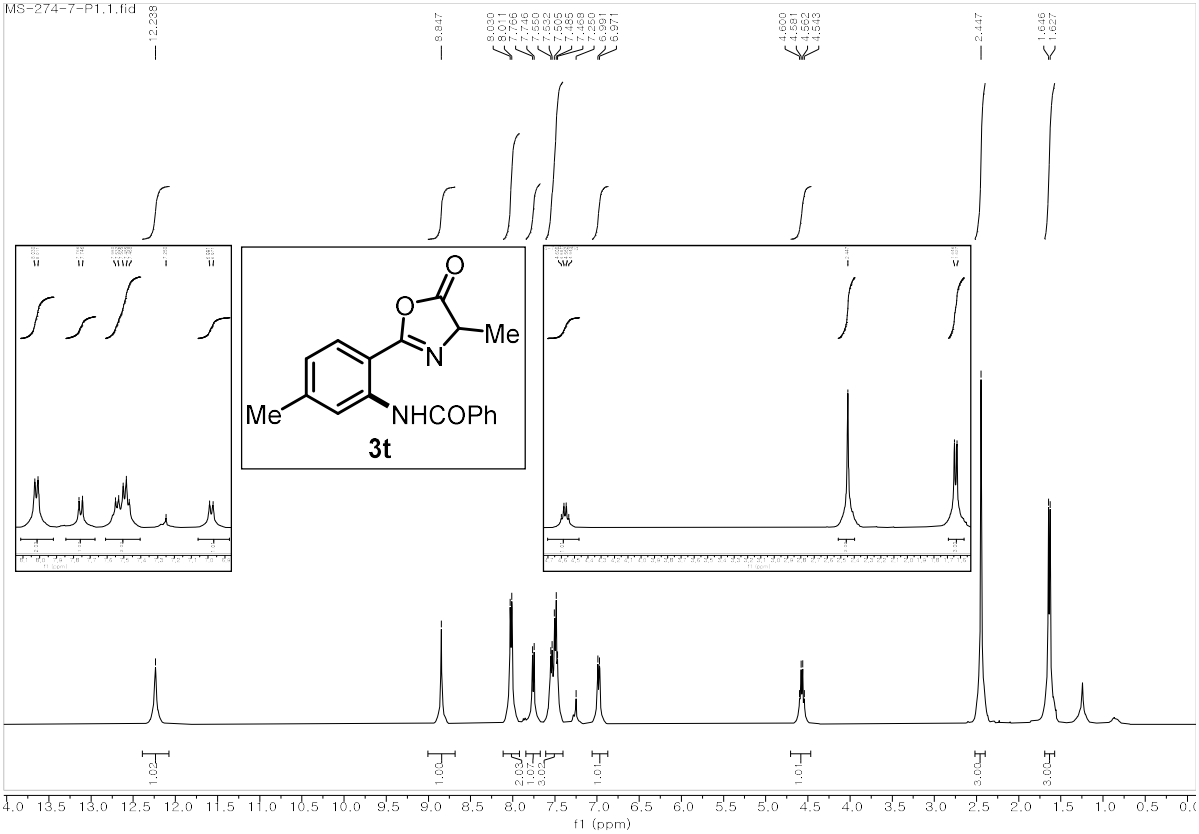

CE103-P-NMR.2.fid
177.363
164.597
164.240
132.956
132.714
132.488
128.992
127.649
101.181
77.477
77.160 CDCl3
76.842
47.477
20.067
15.655
Me
O
O
N
NHCOPh
3q
f1 (ppm)

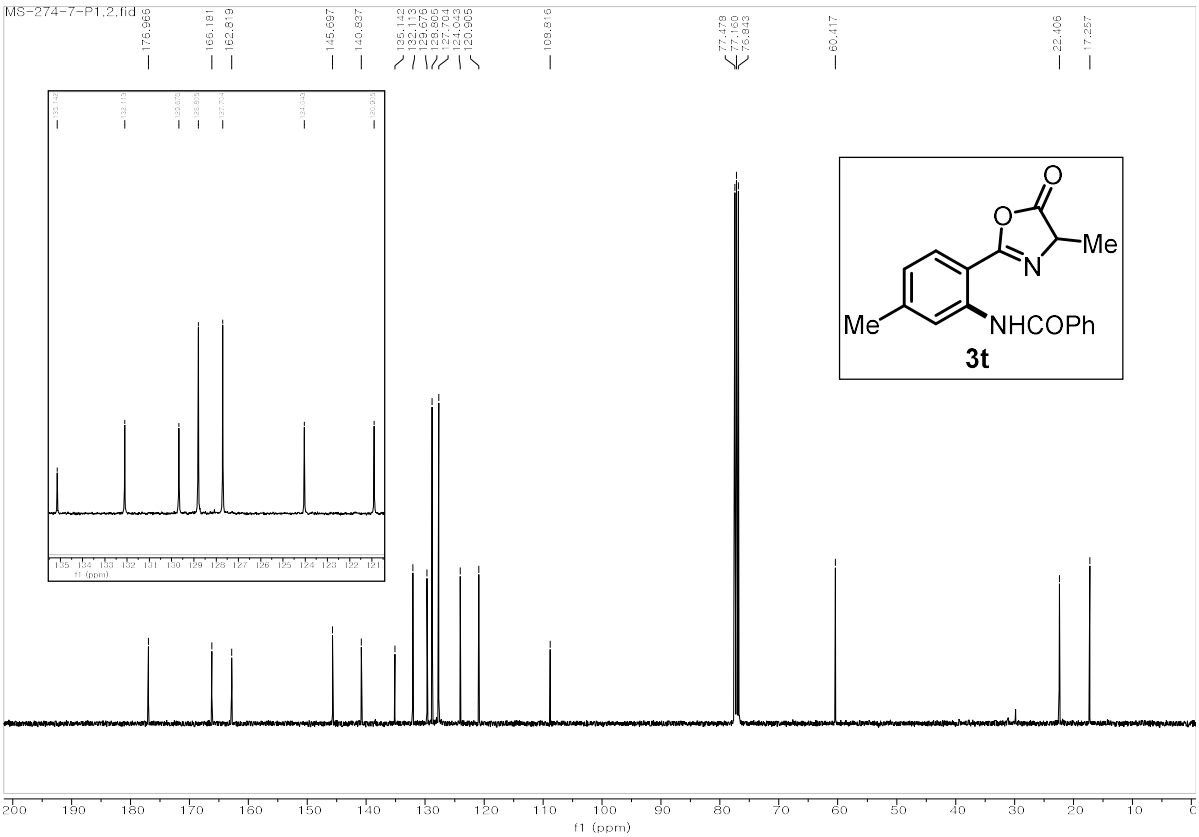

YV703-P-NMR_1.fid
12.151
7.887
7.869
7.549
7.545
7.542
7.532
7.527
7.522
7.512
7.508
7.505
7.492
7.477
7.435
7.432
7.418
7.414
7.260 CDCl3
3.195
3.189
3.180
3.174
3.165
3.159
2.479
2.474
2.464
2.459
2.450
2.444
1.808
1.776
1.770
1.760
1.743
1.737
1.727
1.721
1.716
1.711
1.686
1.680
O
O
N
NHCOPh
3r
1.00
2.00
1.02
2.03
2.05
2.09
2.16
5.94
f1 (ppm)

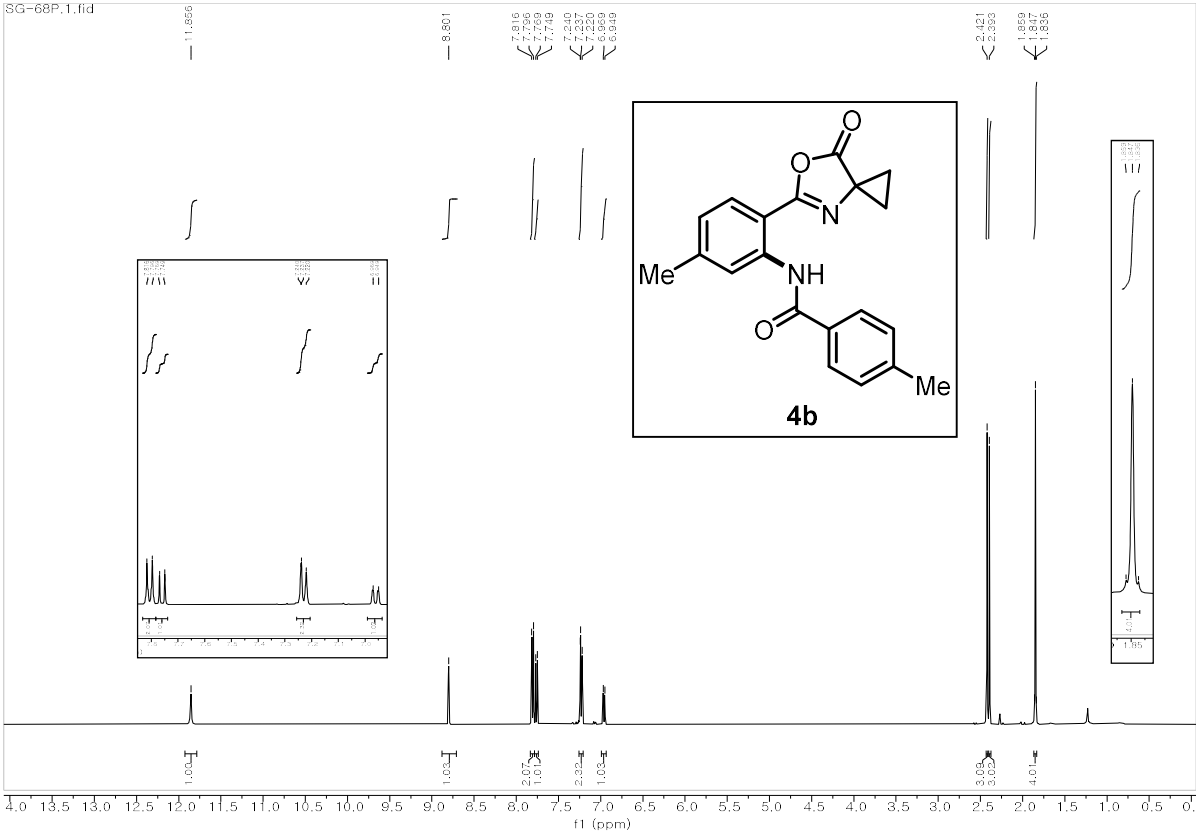

YV703-P-NMR_400MHz_2.fid
177.500
165.682
164.345
149.491
135.187
132.130
128.649
127.932
101.283
77.476
77.160 CDCl3
76.841
47.138
28.658
24.053
21.979
21.631
19.676
O
O
N
NHCOPh
3r
f1 (ppm)

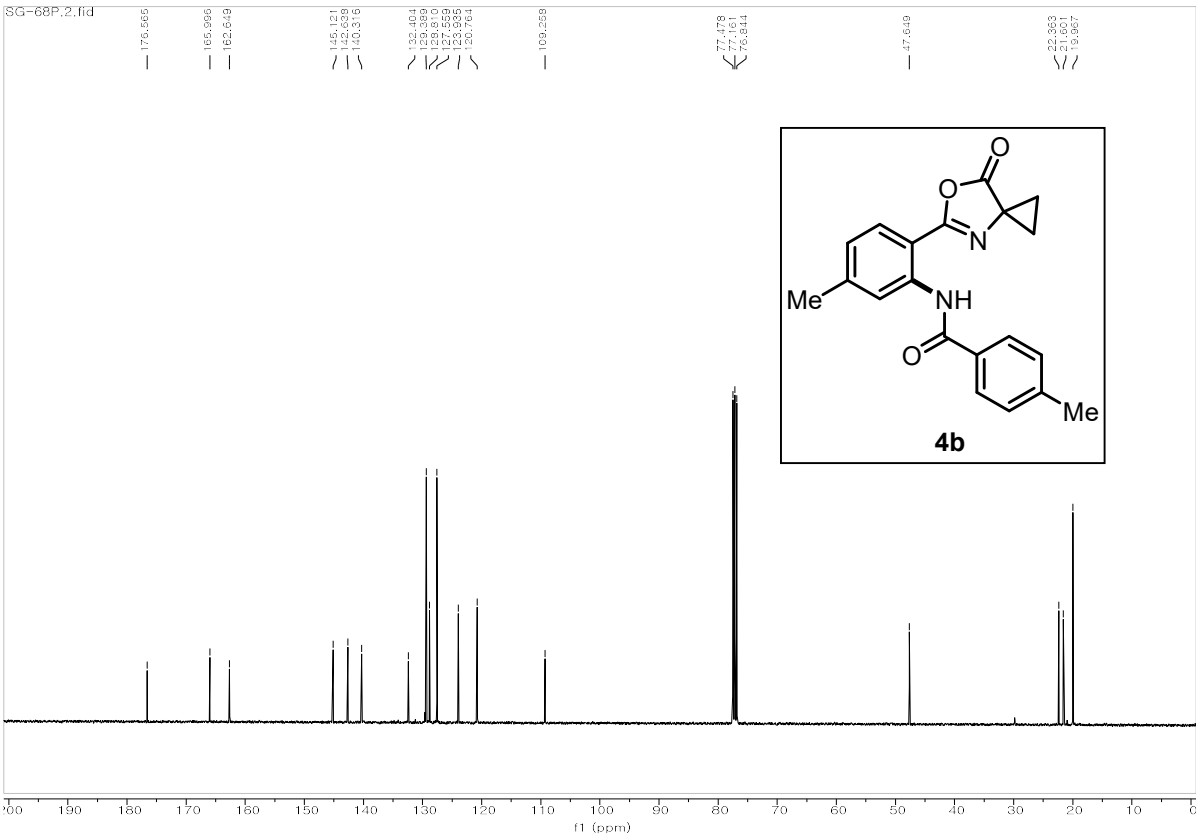

SG-87-P1_220328.1.fid
12.306
9.881
8.065
8.043
7.804
7.779
7.560
7.542
7.502
7.482
7.466
7.260
7.014
6.994
4.428
4.416
2.479
2.477
2.430
2.419
2.413
2.402
1.227
1.210
0.991
0.974
1.00
1.01
2.09
1.02
1.05
2.07
1.04
1.03
4.01
3.04
3.01
f1 (ppm)
O
O
N
Me
Me
Me
NHCOPh
3s

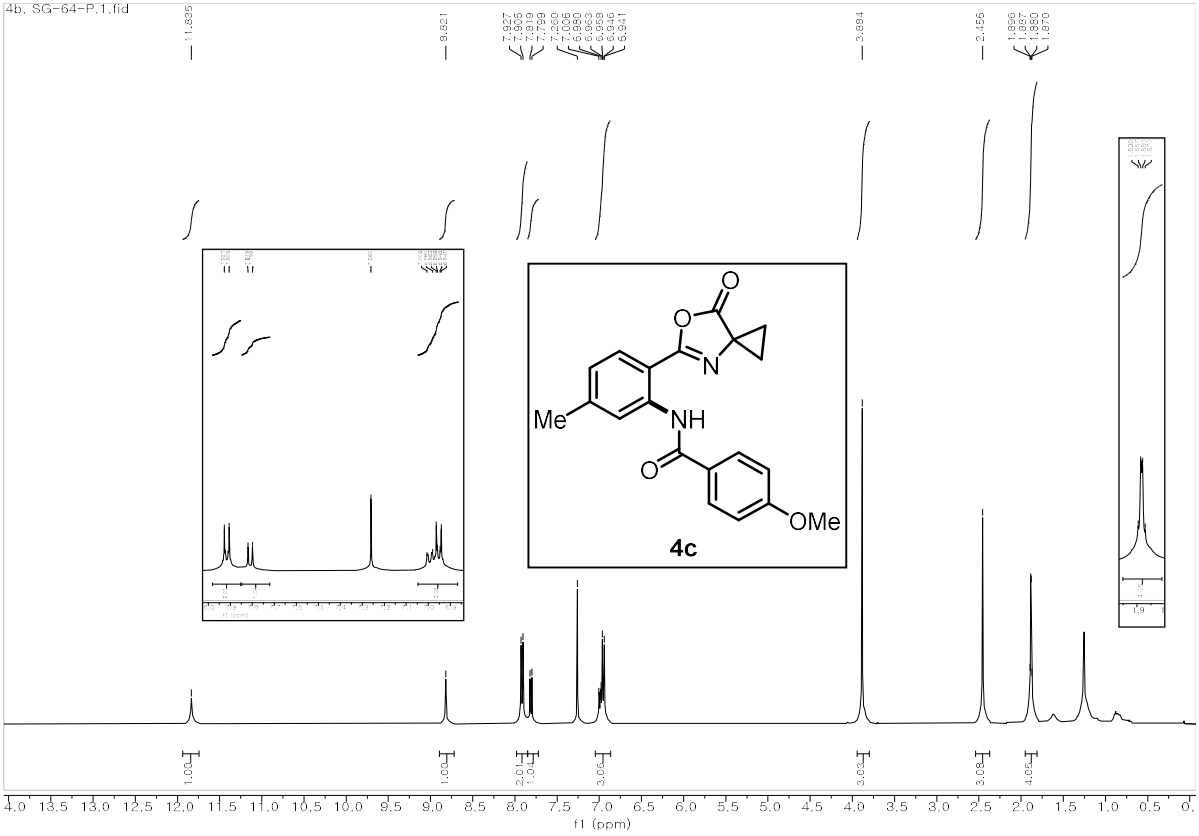

SG-87-P1_220328.2.fid
175.788
166.247
163.130
145.675
140.935
135.110
132.149
129.080
128.784
127.745
124.089
120.902
108.777
77.478
77.161
76.844
70.318
31.503
22.403
19.665
17.829
f1 (ppm)
O
O
N
Me
Me
Me
NHCOPh
3s

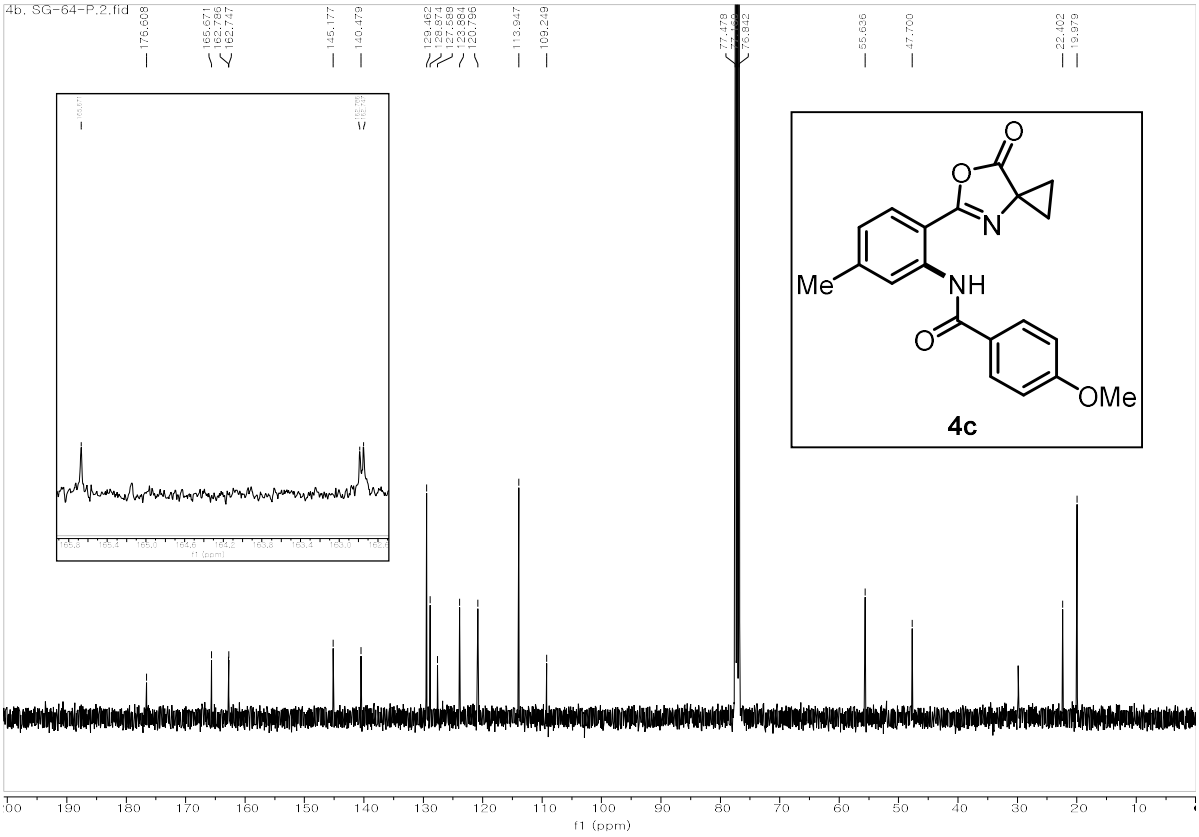

MS-274-7-P1,1.fid
12.238
8.847
8.030
8.011
7.766
7.746
7.650
7.632
7.505
7.485
7.469
7.280
6.991
6.971
4.600
4.581
4.562
4.543
2.447
1.646
1.627
Me
N
O
O
Me
NHCOPh
3t
f1 (ppm)

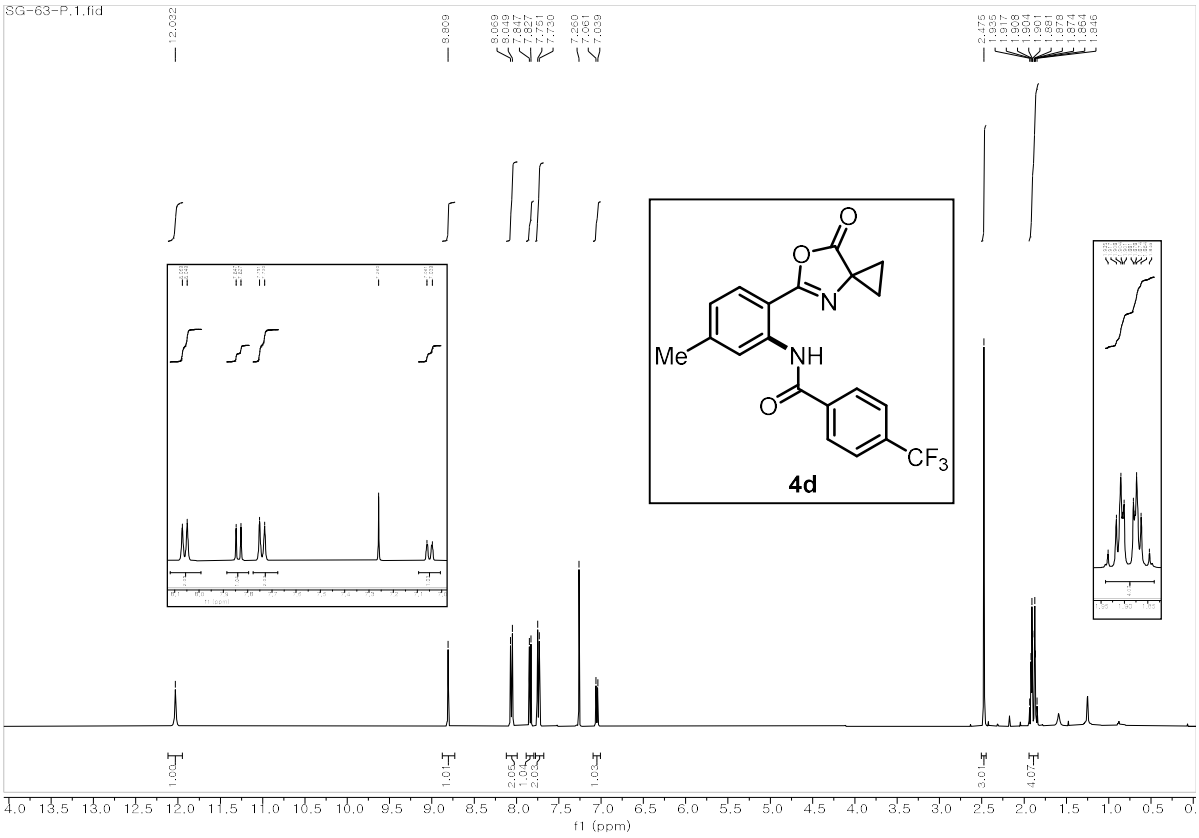

MS-274-7-P1,2.fid
176.966
166.181
162.819
145.697
140.837
135.142
132.113
129.676
129.085
127.704
124.043
120.905
109.816
77.478
77.160
76.843
60.417
22.406
17.257
Me
N
O
O
Me
NHCOPh
3t
f1 (ppm)

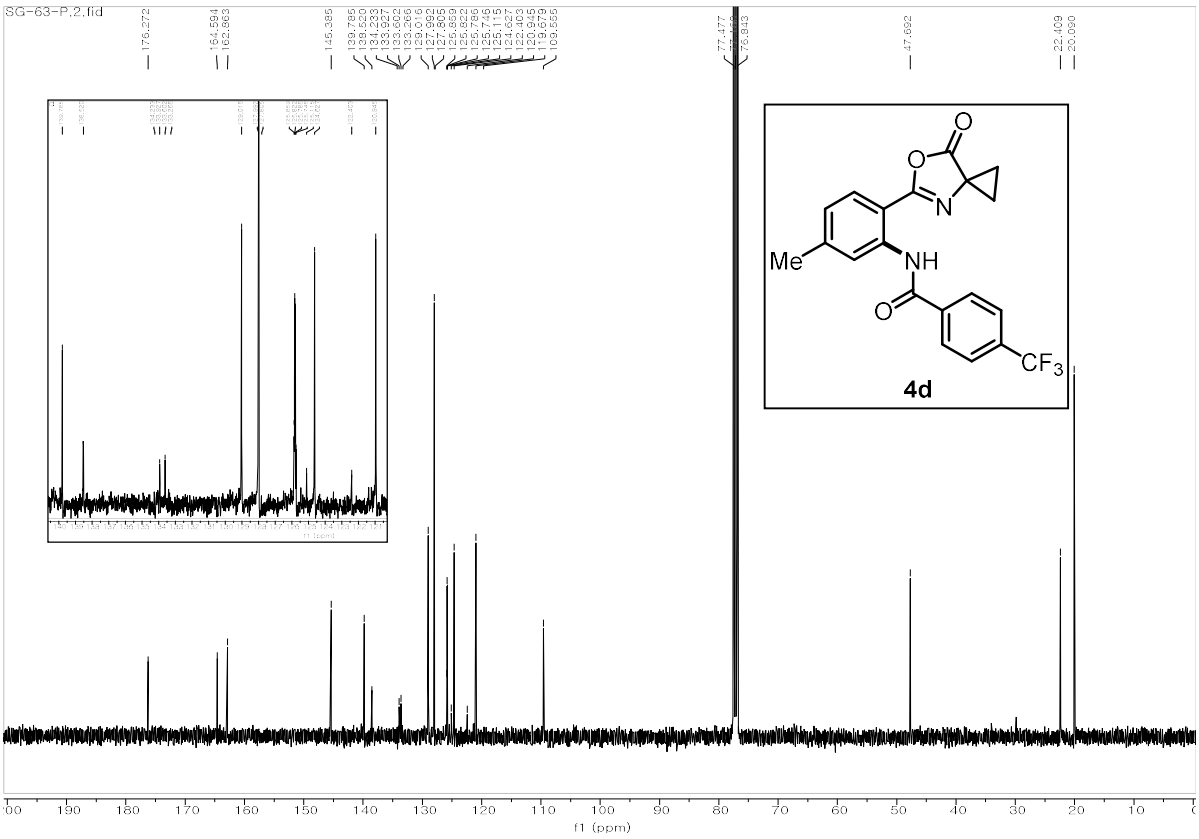

SG-68P.1.fid
11.856
8.801
7.816
7.796
7.769
7.749
7.240
7.237
7.229
7.226
6.949
2.421
2.353
1.859
1.847
1.836
Me
NH
O
N
Me
4b
f1 (ppm)

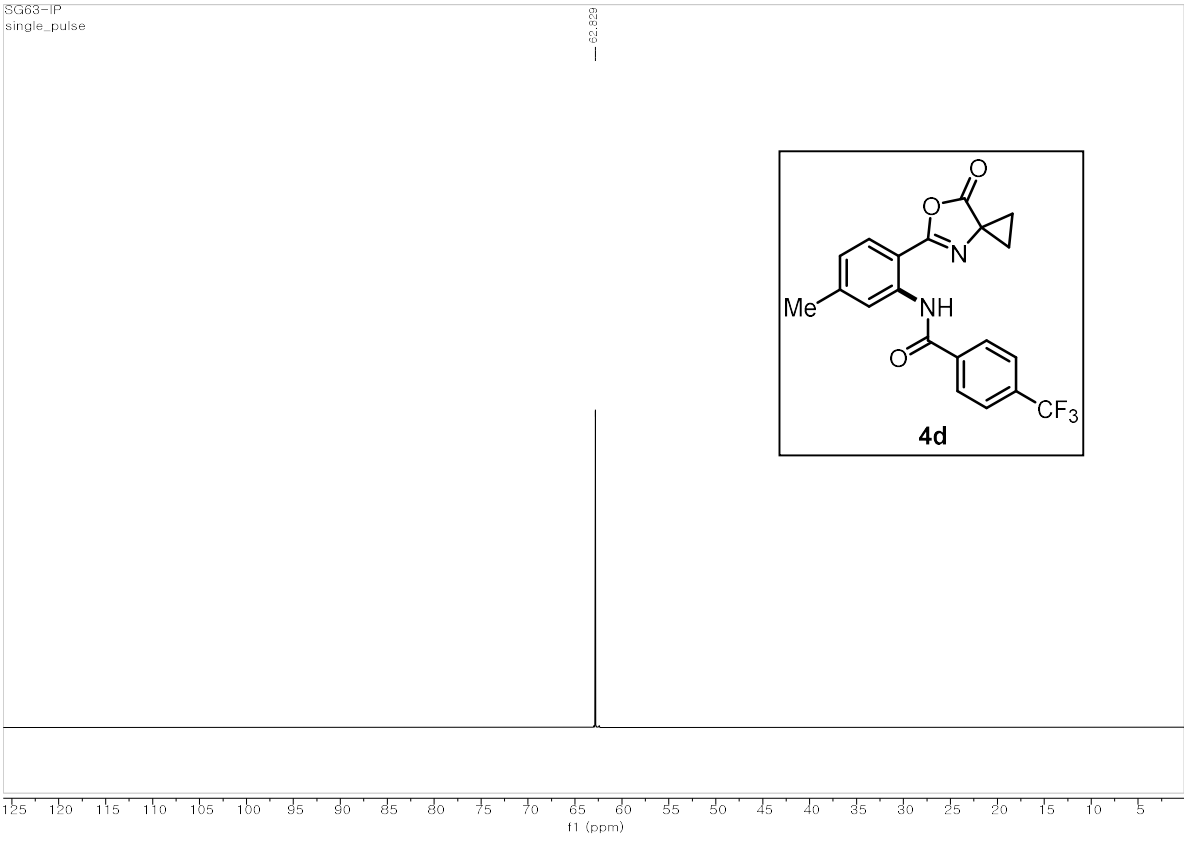

SG-68P.2.fid
176.565
165.996
162.649
145.121
142.639
140.316
132.464
129.389
128.830
127.559
123.935
120.764
109.259
77.478
77.161
76.844
47.649
22.883
21.863
19.967
Me
NH
O
N
Me
4b
f1 (ppm)

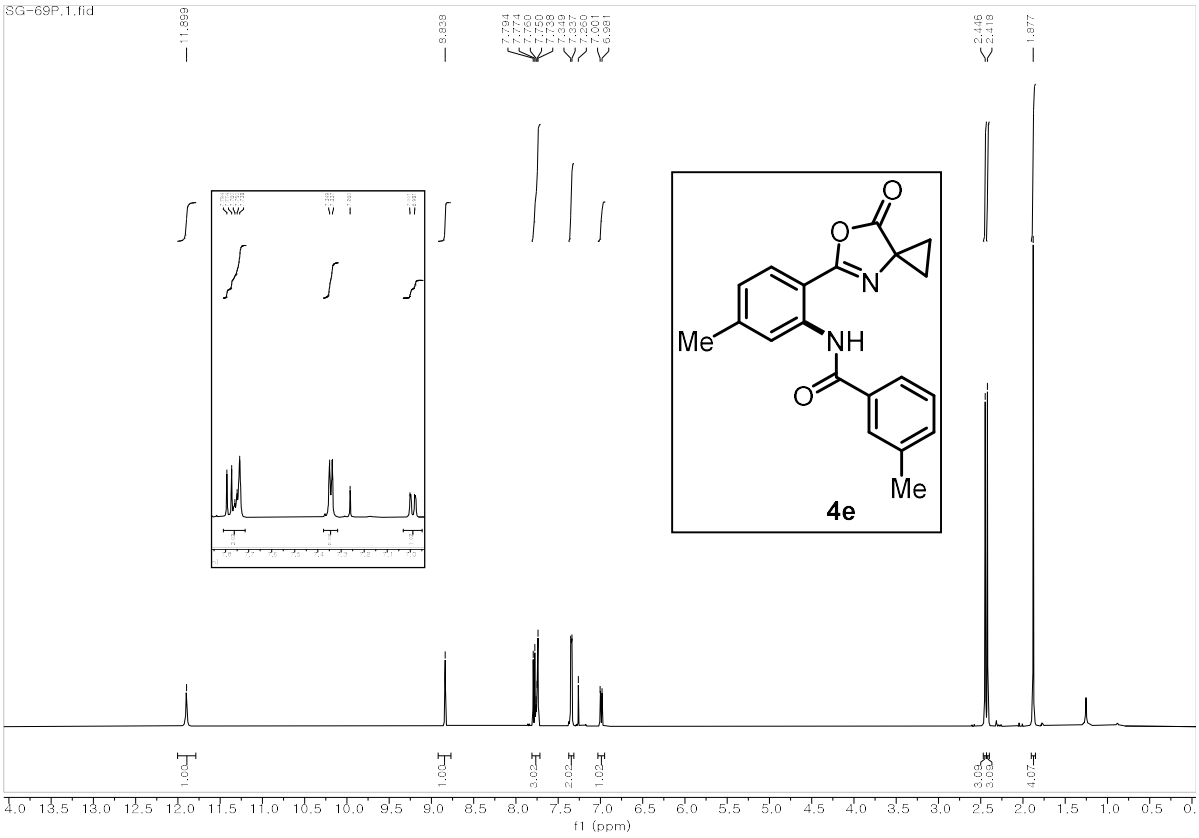

4b, SG-64-P.1.fid
11.835
8.821
7.927
7.915
7.819
7.799
7.260
7.006
6.980
6.963
6.959
6.946
6.941
3.884
2.456
1.896
1.887
1.880
1.870
Me
NH
O
N
OMe
4c
f1 (ppm)

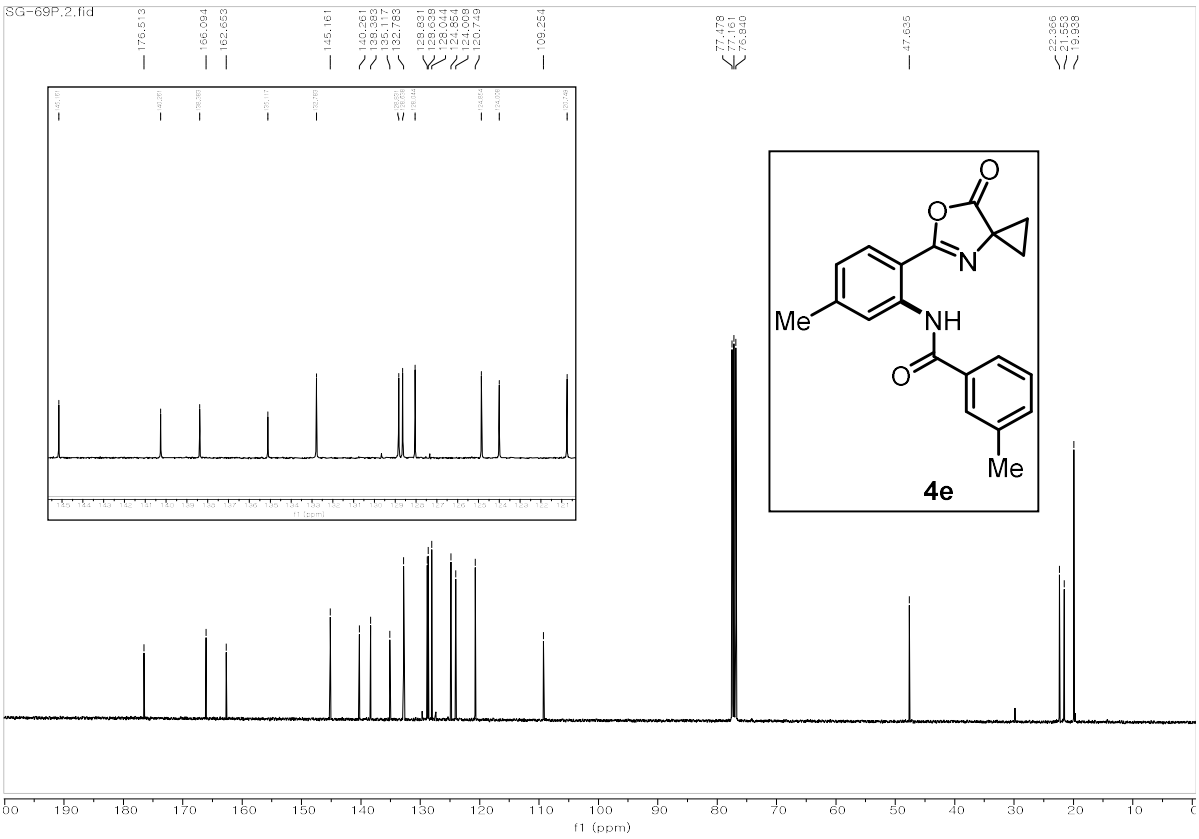

4b, SG-64-P.2.fid
176.608
165.671
162.786
162.747
145.177
140.479
129.462
128.874
123.888
123.444
120.796
113.947
109.249
77.478
77.054
76.842
55.636
47.700
22.402
19.979
Me
NH
O
N
OMe
4c
f1 (ppm)

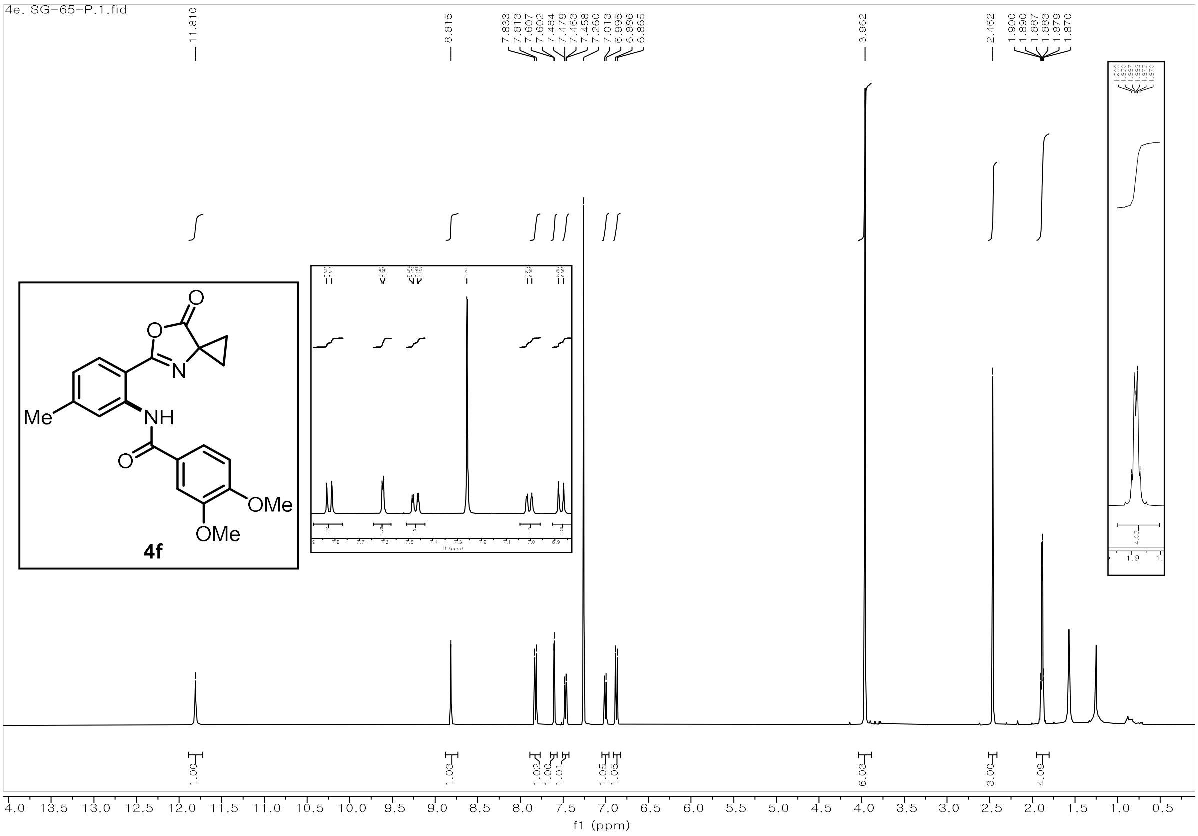

SG-63-P.1.fid
12.032
8.859
8.069
8.049
7.847
7.827
7.751
7.730
7.260
7.061
7.039
2.175
1.935
1.917
1.908
1.904
1.891
1.881
1.879
1.874
1.864
1.846
Me
NH
O
N
CF3
4d
f1 (ppm)

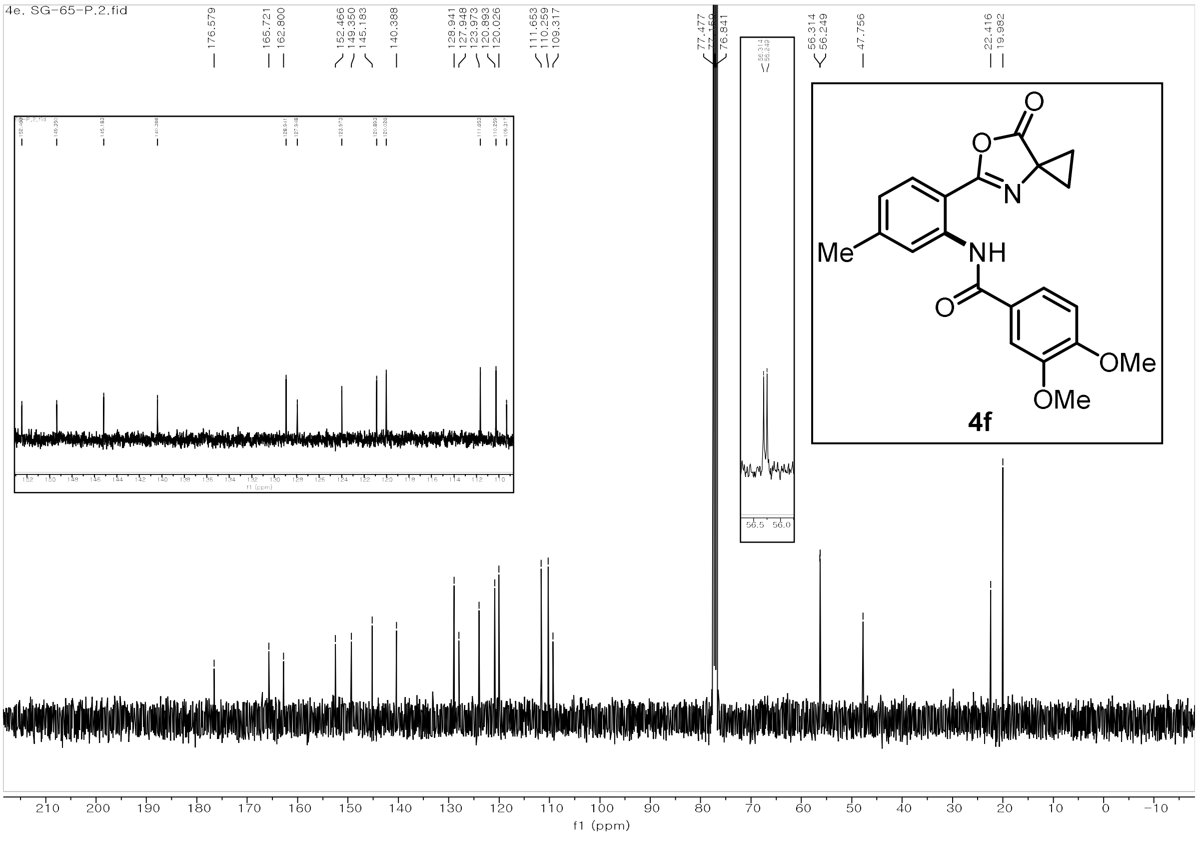

SG-63-P.2.fid
176.272
164.594
162.863
145.385
139.785
139.620
134.233
133.927
133.662
133.076
129.066
127.992
127.805
125.869
125.782
125.746
125.115
124.627
122.943
120.945
119.679
109.595
77.477
77.164
76.843
47.692
22.409
20.099
Me
NH
O
N
CF3
4d
f1 (ppm)

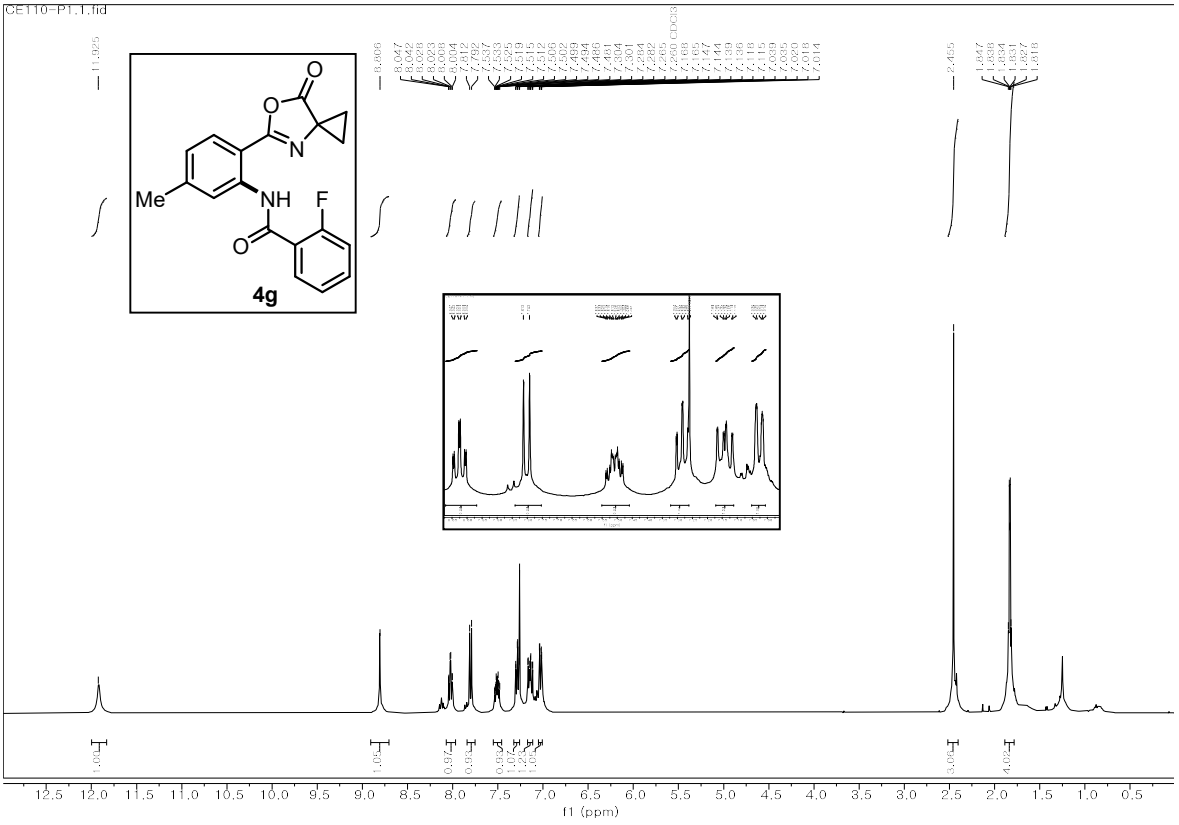

SG63-IP
single_pulse
62.829
O
O
N
Me
NH
O
CF3
4d
125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5
f1 (ppm)

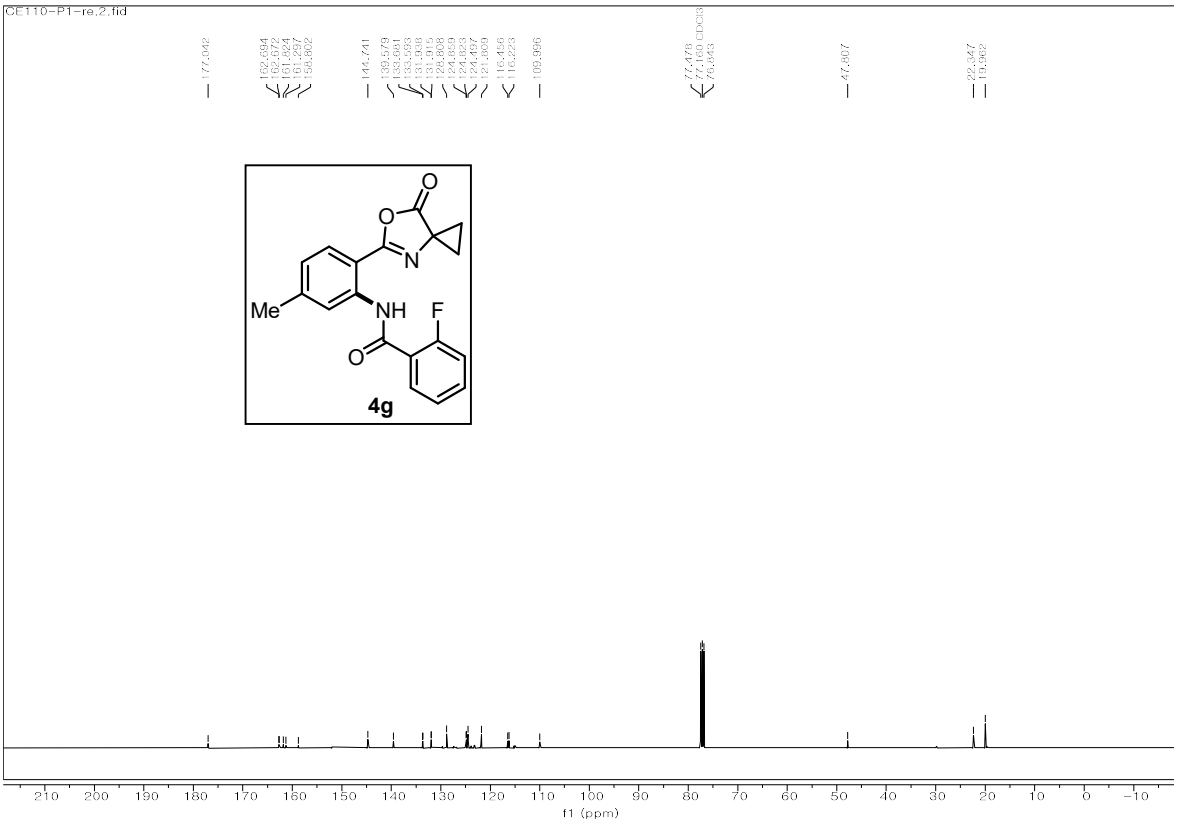

SG-69P.1.fid
11.999
8.839
7.794
7.774
7.760
7.750
7.738
7.349
7.337
7.260
7.001
6.981
2.446
2.418
1.877
Me
NH
O
O
N
Me
4e
f1 (ppm)

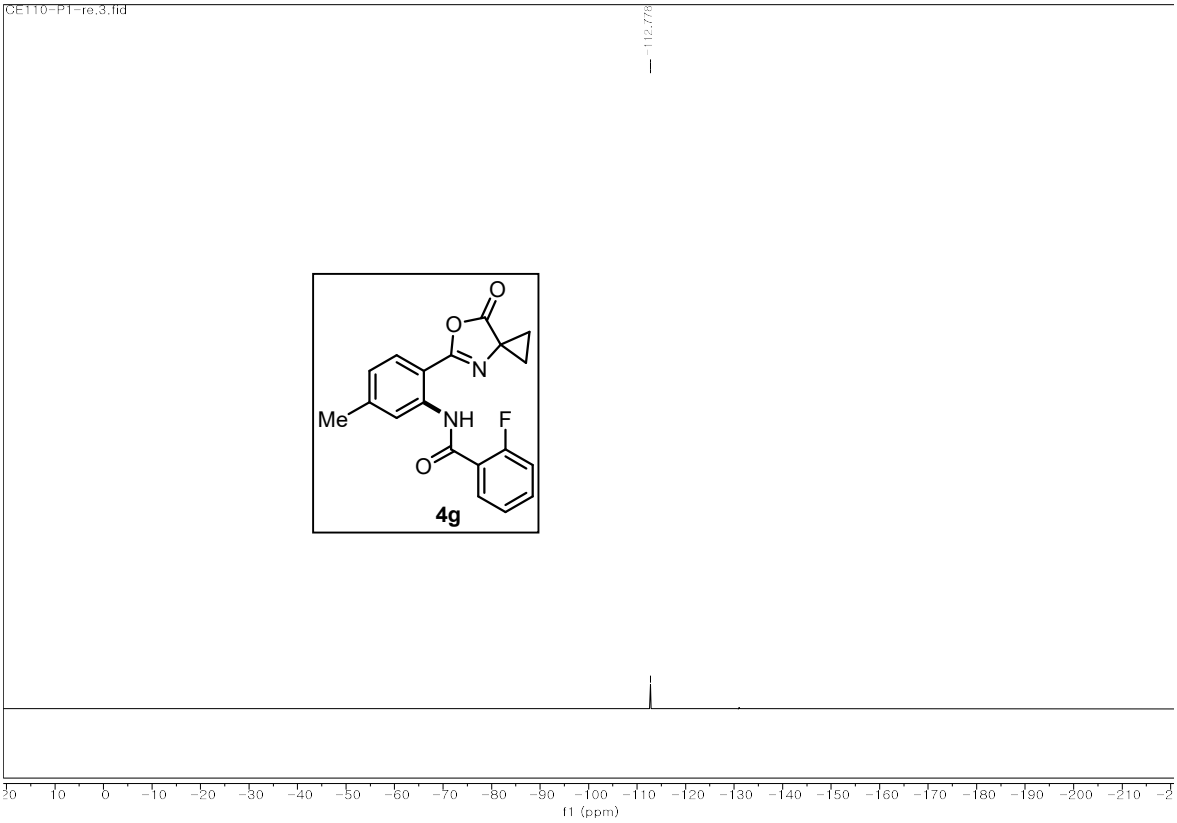

SG-69P.2.fid
176.513
166.094
162.653
145.161
140.261
138.383
135.117
132.783
128.831
128.639
128.044
124.864
124.009
120.749
109.254
77.478
77.161
76.845
47.635
29.666
21.656
19.938
Me
NH
O
O
N
Me
4e
f1 (ppm)

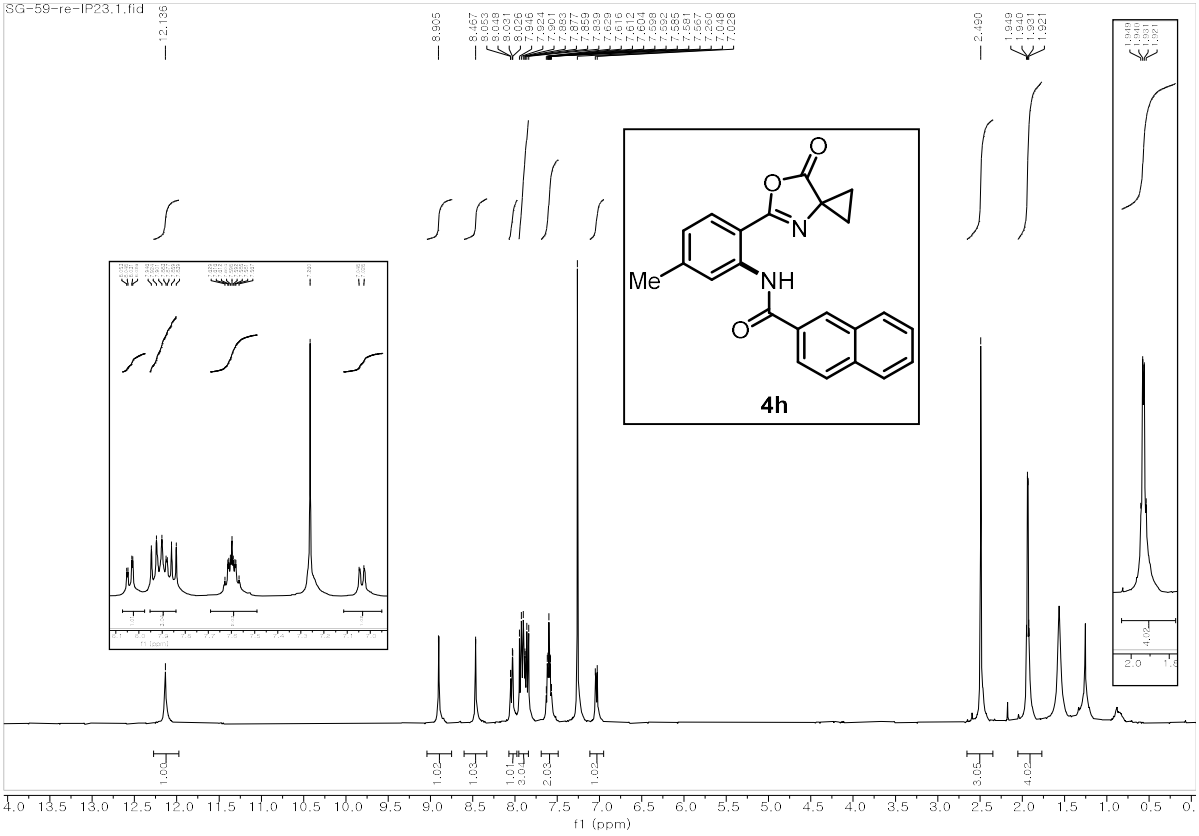

4e. SG-65-P.1.fid
11.810
8.815
7.833
7.813
7.607
7.602
7.484
7.479
7.463
7.458
7.260
7.013
6.995
6.886
6.865
3.962
2.462
1.900
1.890
1.887
1.883
1.879
1.870
Me
NH
O
N
OMe
OMe
4f
1.00
1.03
1.02
1.00
1.01
1.05
1.05
6.03
3.00
4.09
f1 (ppm)

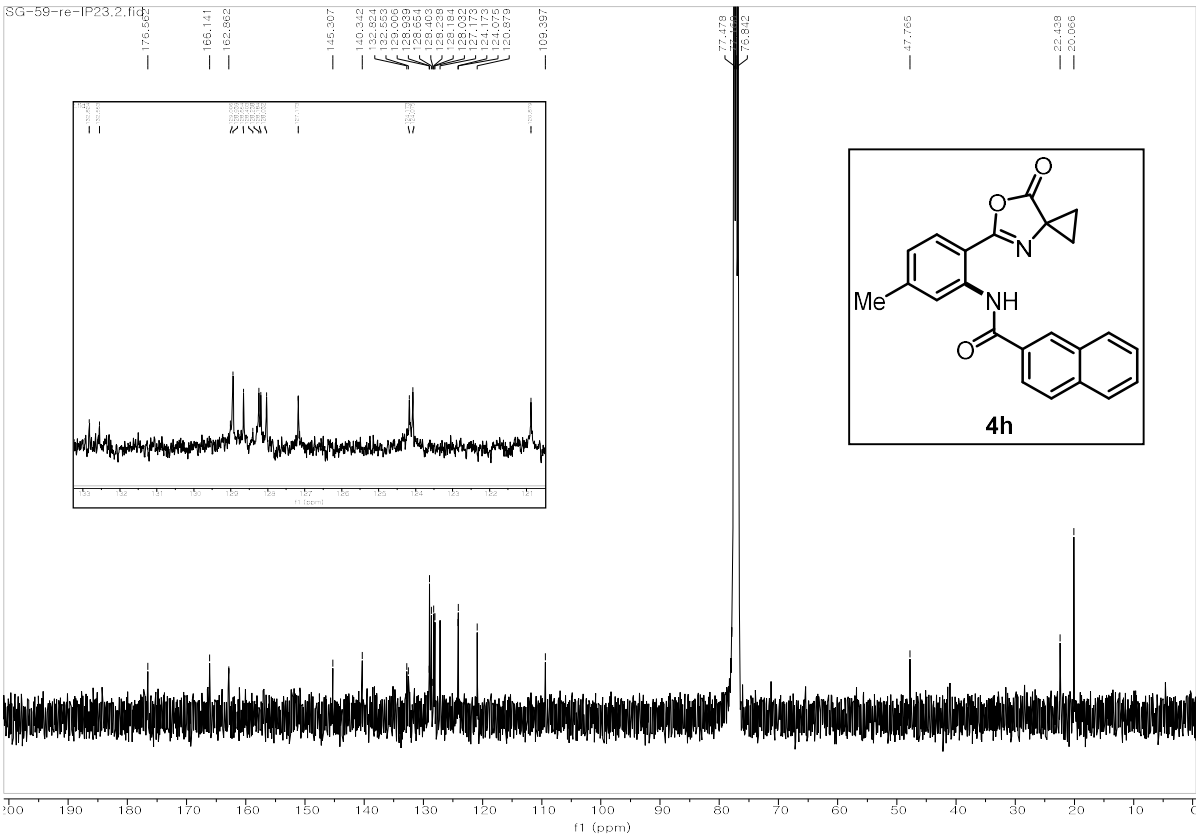

4e. SG-65-P.2.fid
176.579
165.721
162.800
152.466
149.350
145.183
140.388
128.941
127.948
123.973
120.893
120.026
111.653
110.259
109.317
77.477
77.160
76.841
56.314
56.249
47.756
22.416
19.982
Me
NH
O
N
OMe
OMe
4f
f1 (ppm)

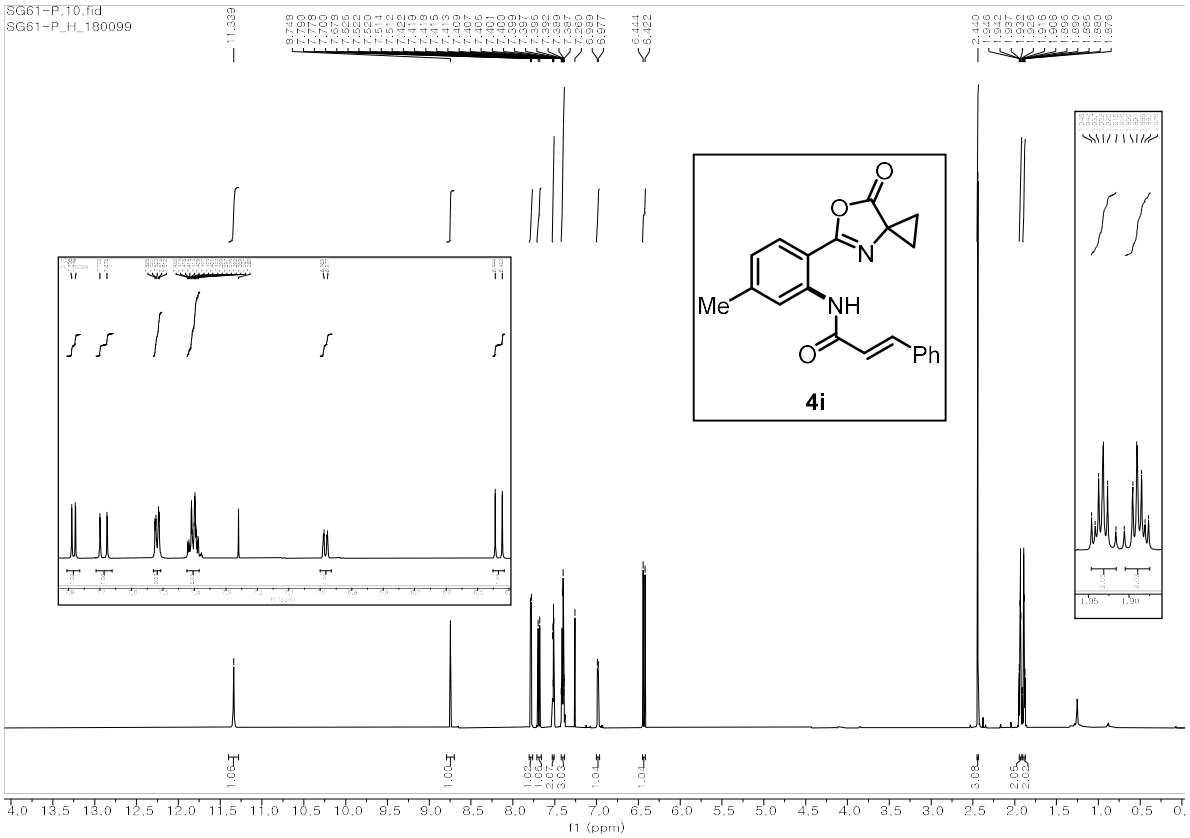

CE110-P1_1.fid
4g
f1 (ppm)

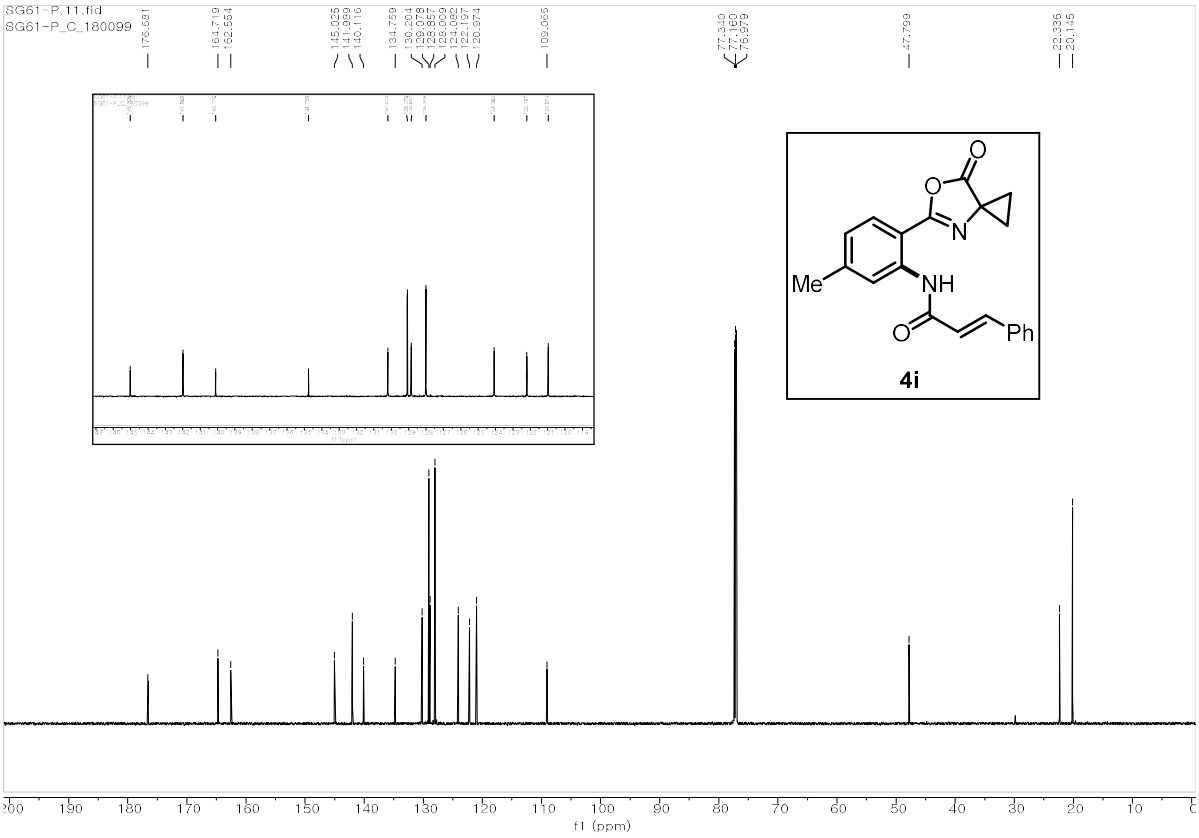

CE110-P1-re_2.fid
4g
f1 (ppm)

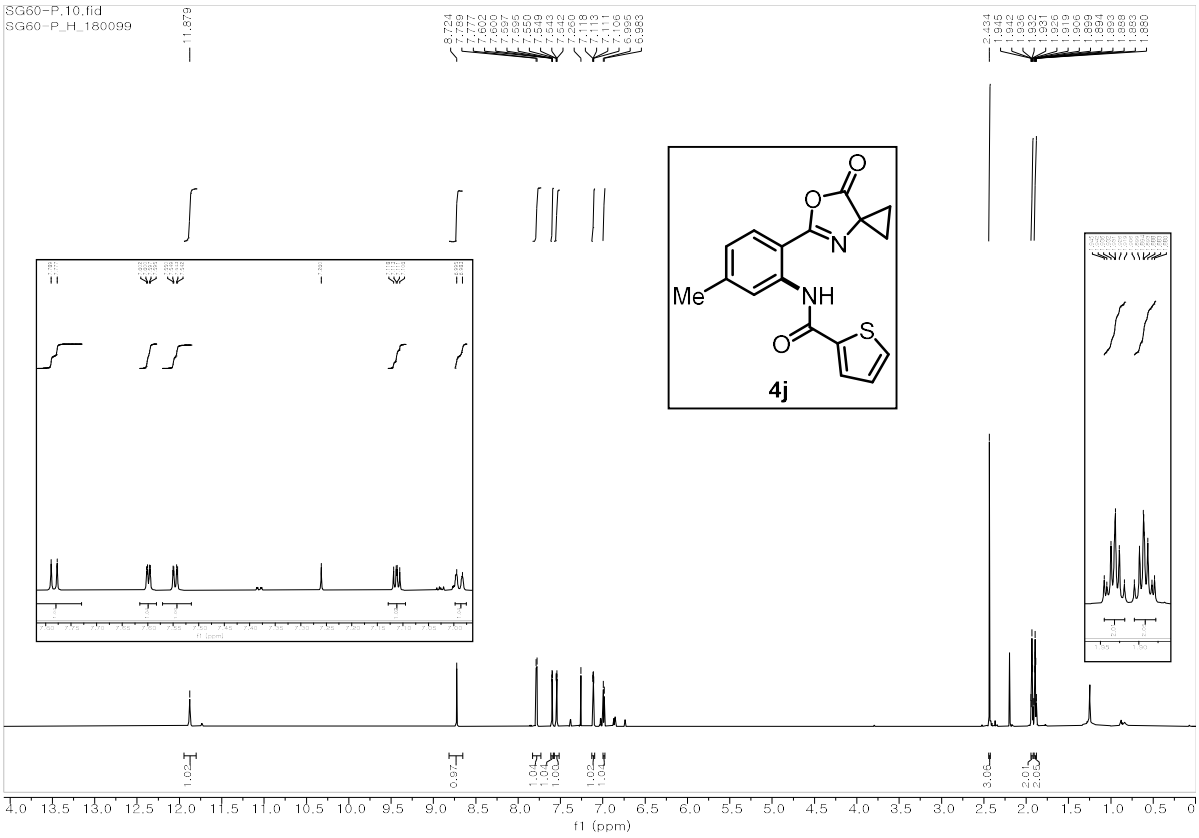

CE110-P1-re.3.fid
O
O
N
Me
NH
F
O
4g
f1 (ppm)

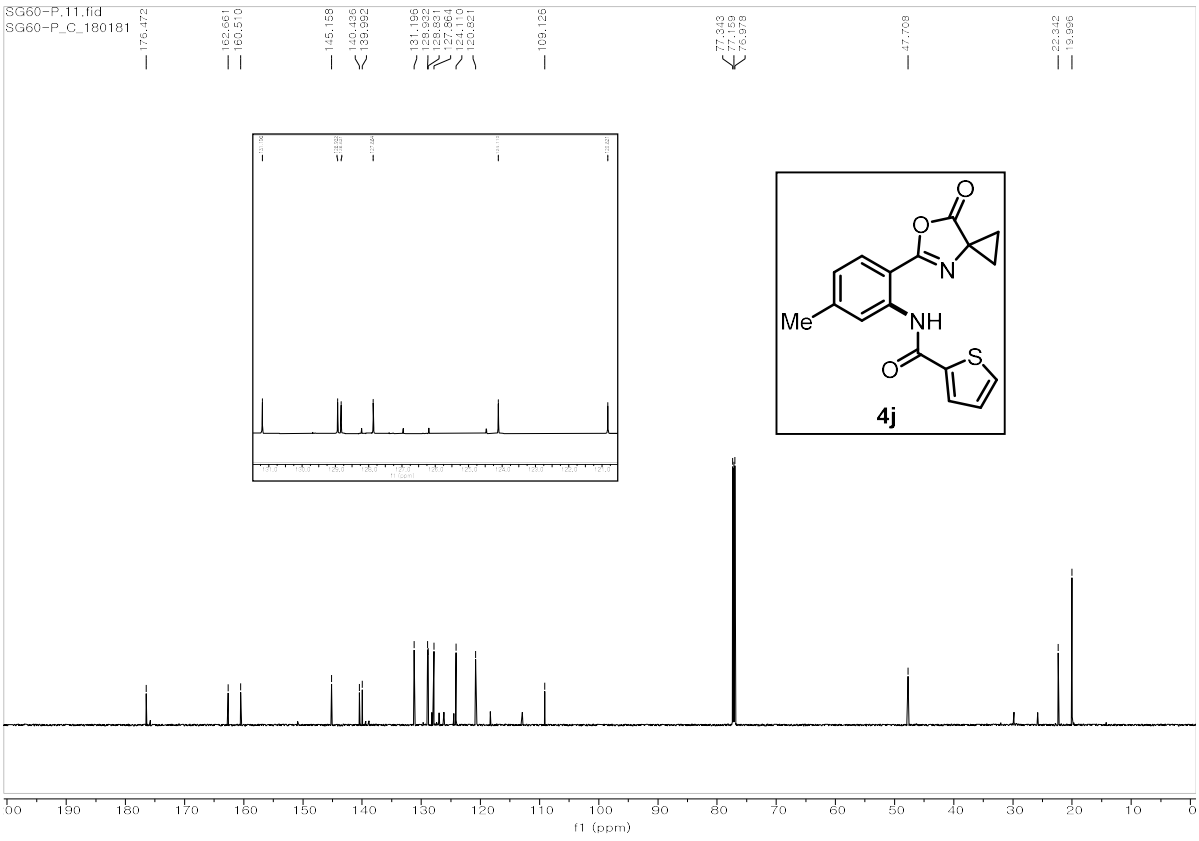

SG-59-re-IP23.1.fid
12.136
8.905
8.457
8.053
8.048
8.031
8.026
7.946
7.924
7.901
7.893
7.877
7.859
7.839
7.623
7.616
7.612
7.604
7.599
7.592
7.585
7.571
7.557
7.260
7.048
7.028
2.490
1.949
1.940
1.931
1.921
1.00
1.02
1.03
1.01
3.04
2.03
1.02
3.05
4.02
Me
NH
O
N
O
O
4h
f1 (ppm)

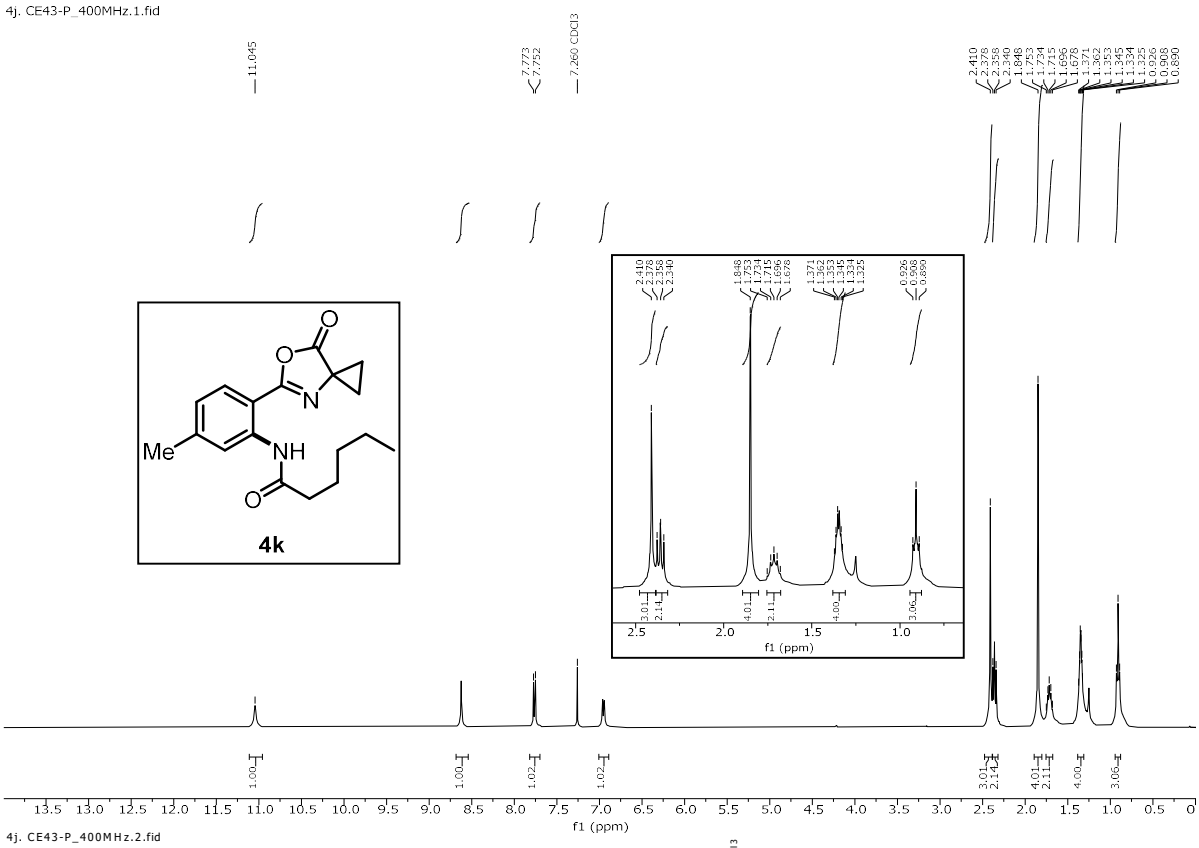

SG-59-re-IP23.2.fid
176.562
166.141
162.962
145.307
140.342
132.824
132.553
129.066
128.869
128.651
128.403
128.239
128.184
128.042
127.173
124.173
124.075
120.879
109.397
77.479
77.164
76.842
47.765
22.438
20.066
Me
NH
O
N
O
O
4h
f1 (ppm)

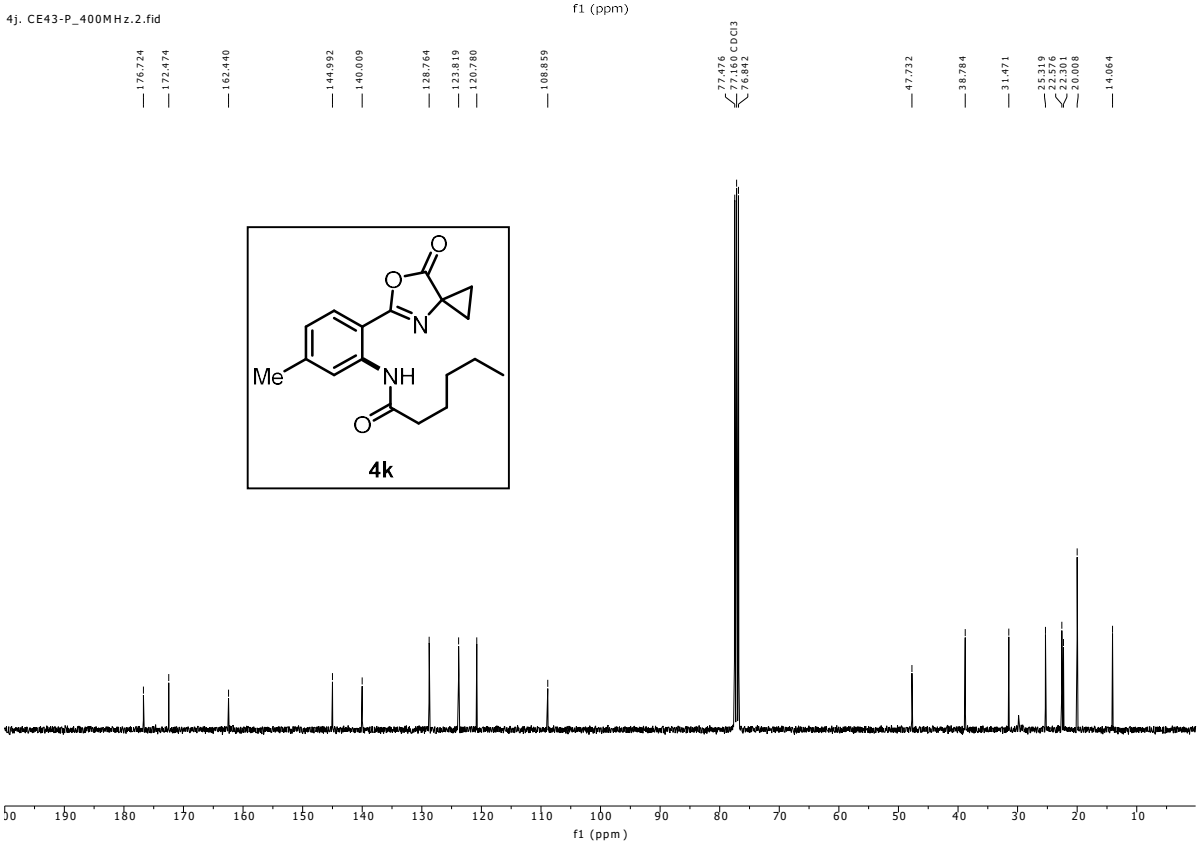

SG61-P_10.fid
SG61-P_H_180099
Me
NH
O
Ph
4i
f1 (ppm)

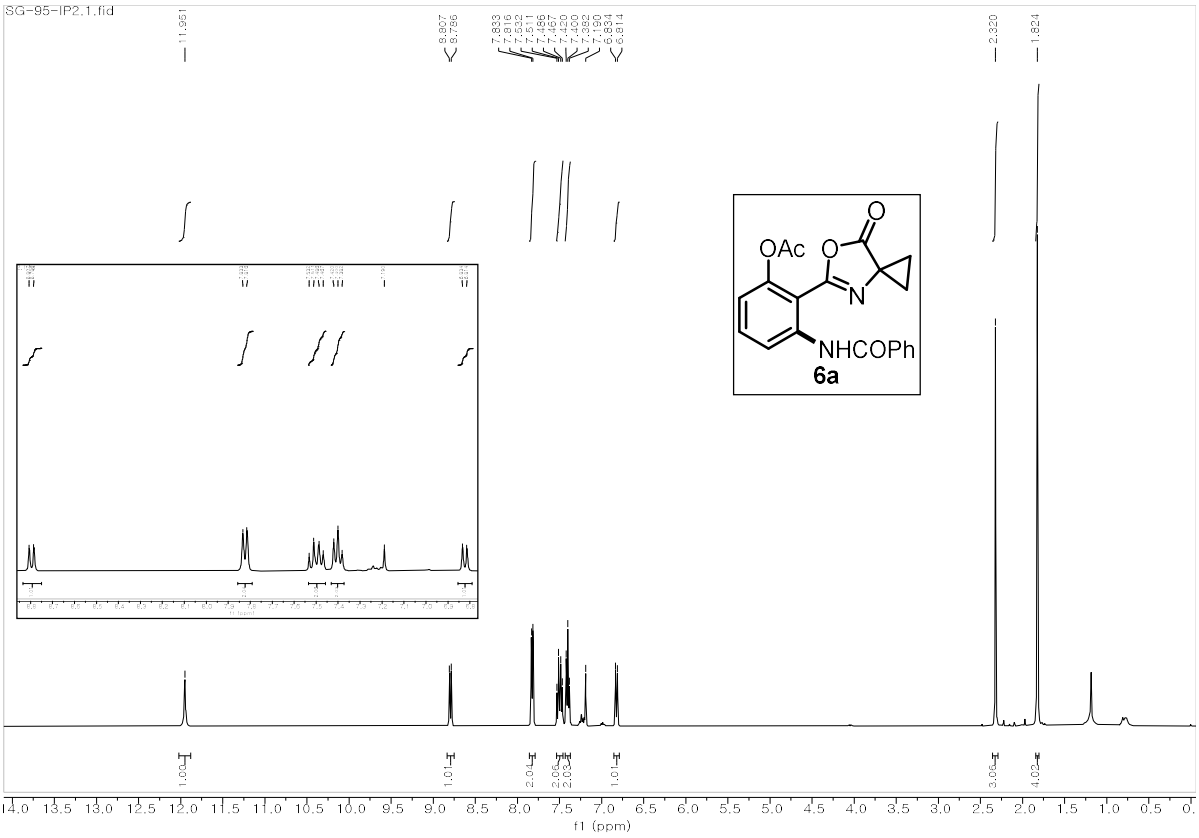

SG61-P_11.fid
SG61-P_C_180099
Me
NH
O
Ph
4i
f1 (ppm)

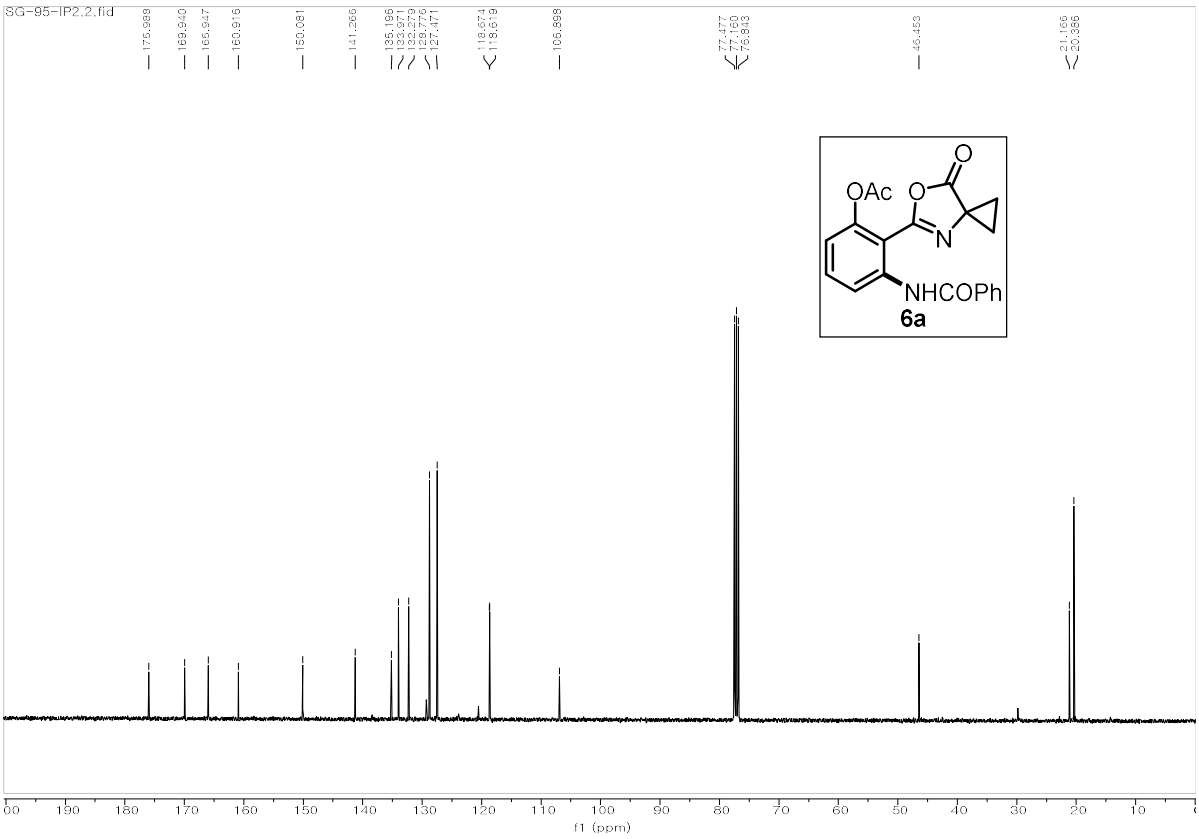

SG60-P.10.fid
SG60-P_H_180099
11.879
8.724
7.759
7.777
7.602
7.600
7.577
7.575
7.550
7.549
7.543
7.542
7.540
7.118
7.113
7.111
7.106
5.995
5.983
2.434
1.945
1.942
1.940
1.932
1.931
1.926
1.919
1.916
1.899
1.894
1.893
1.888
1.883
1.880
1.02
0.97
1.04
1.04
1.03
1.02
1.02
3.06
2.01
2.03
Me
NH
O
S
4j
f1 (ppm)

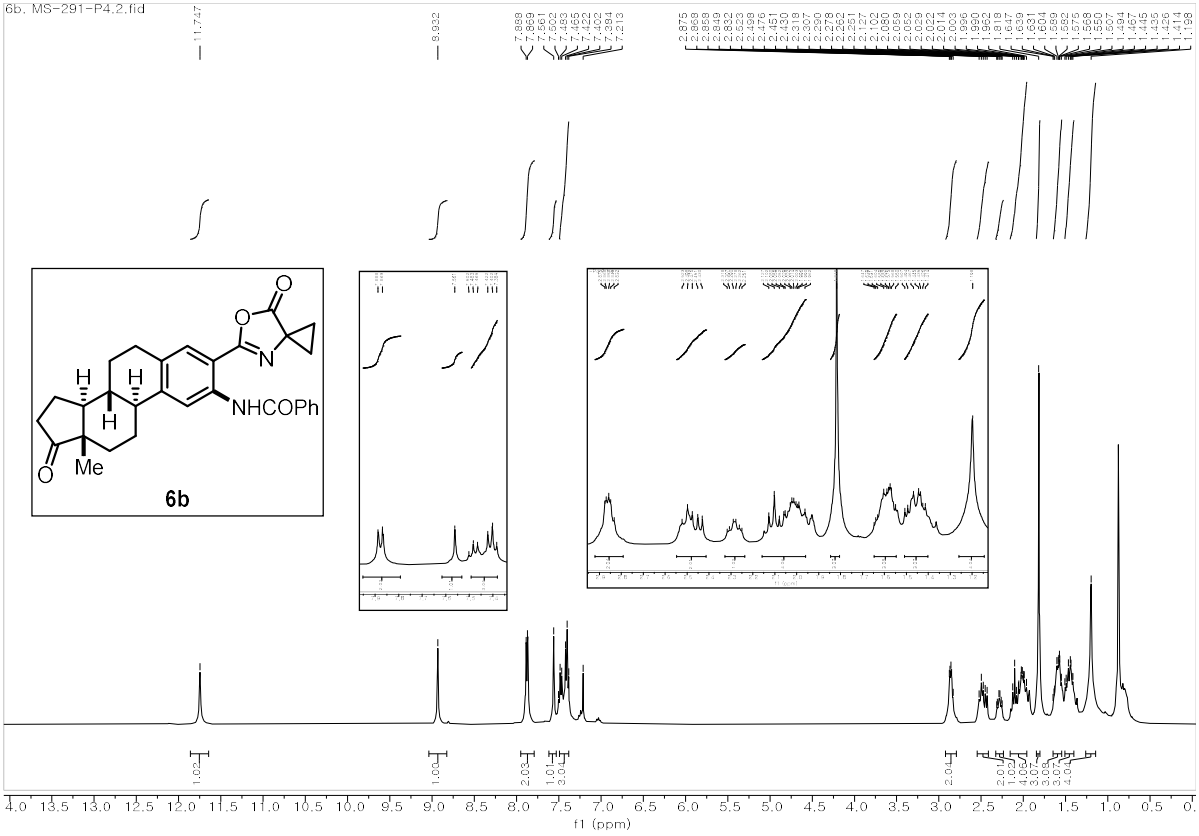

SG60-P.11.fid
SG60-P_C_180181
176.472
162.661
160.510
145.158
140.436
139.992
131.196
128.932
128.681
127.454
124.110
120.821
109.126
77.343
77.159
76.976
47.708
22.342
19.996
Me
NH
O
S
4j
f1 (ppm)

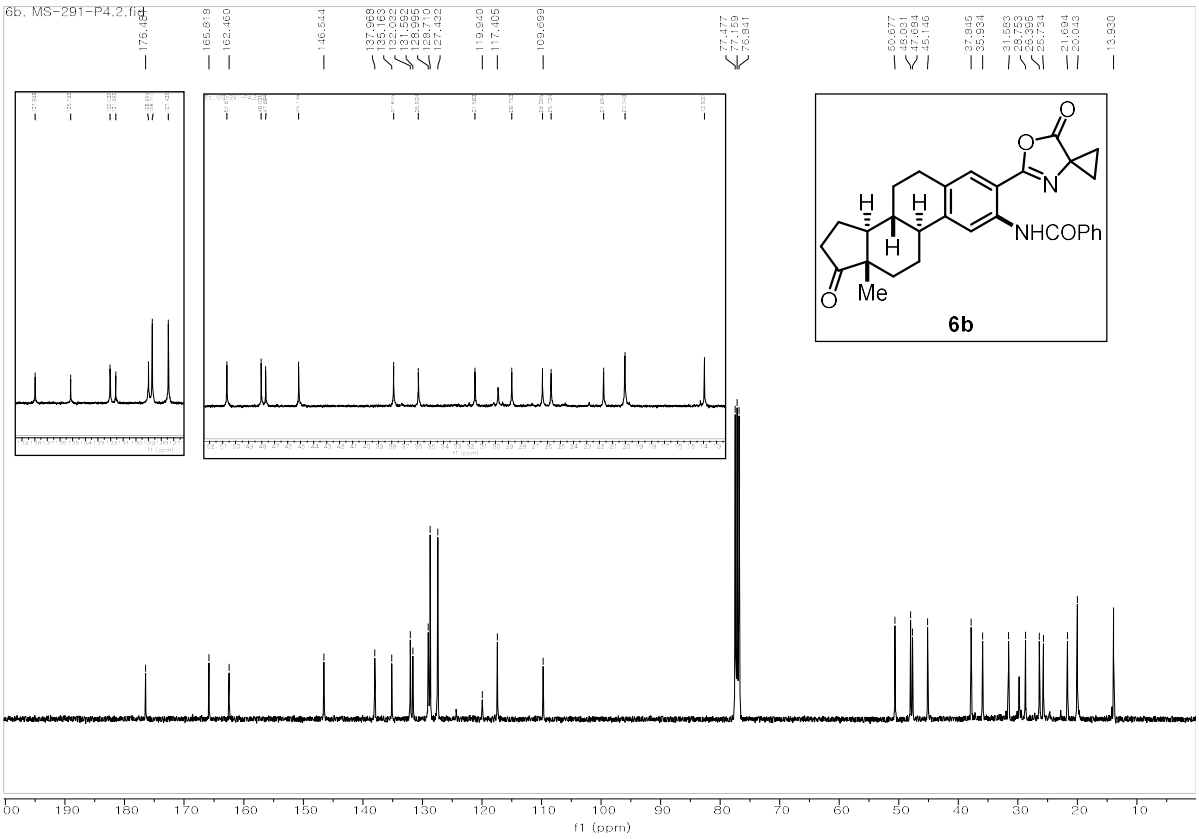

4j. CE43-P_400MHz.1.fid
11.045
7.773
7.752
7.260 CDCl3
2.410
2.378
2.365
2.340
1.848
1.753
1.734
1.715
1.696
1.678
1.371
1.362
1.353
1.345
1.334
1.325
0.926
0.908
0.890
Me
NH
O
N
4k
f1 (ppm)

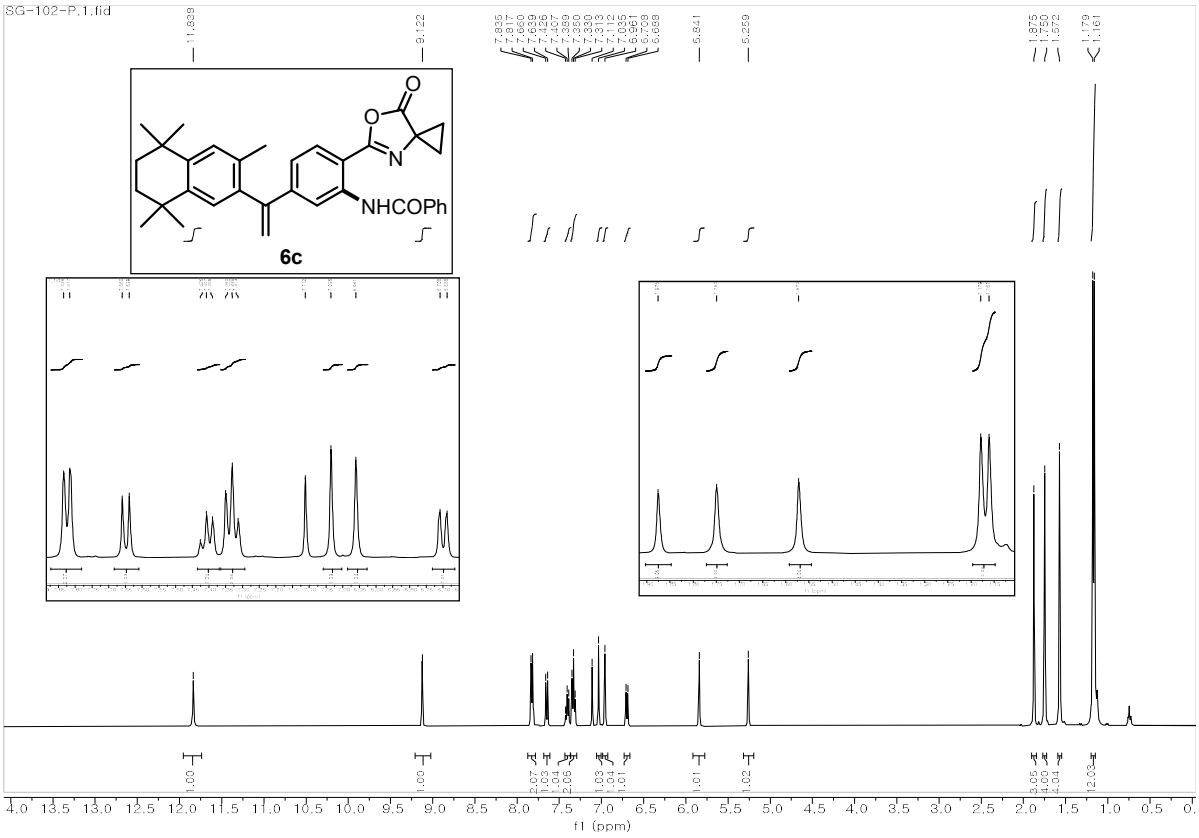

4j. CE43-P_400MHz.2.fid
176.724
172.474
162.440
144.992
140.009
128.764
123.819
120.780
108.859
77.476
77.160 CDCl3
76.842
47.732
38.784
31.471
25.319
22.576
22.301
20.008
14.064
Me
NH
O
N
4k
f1 (ppm)

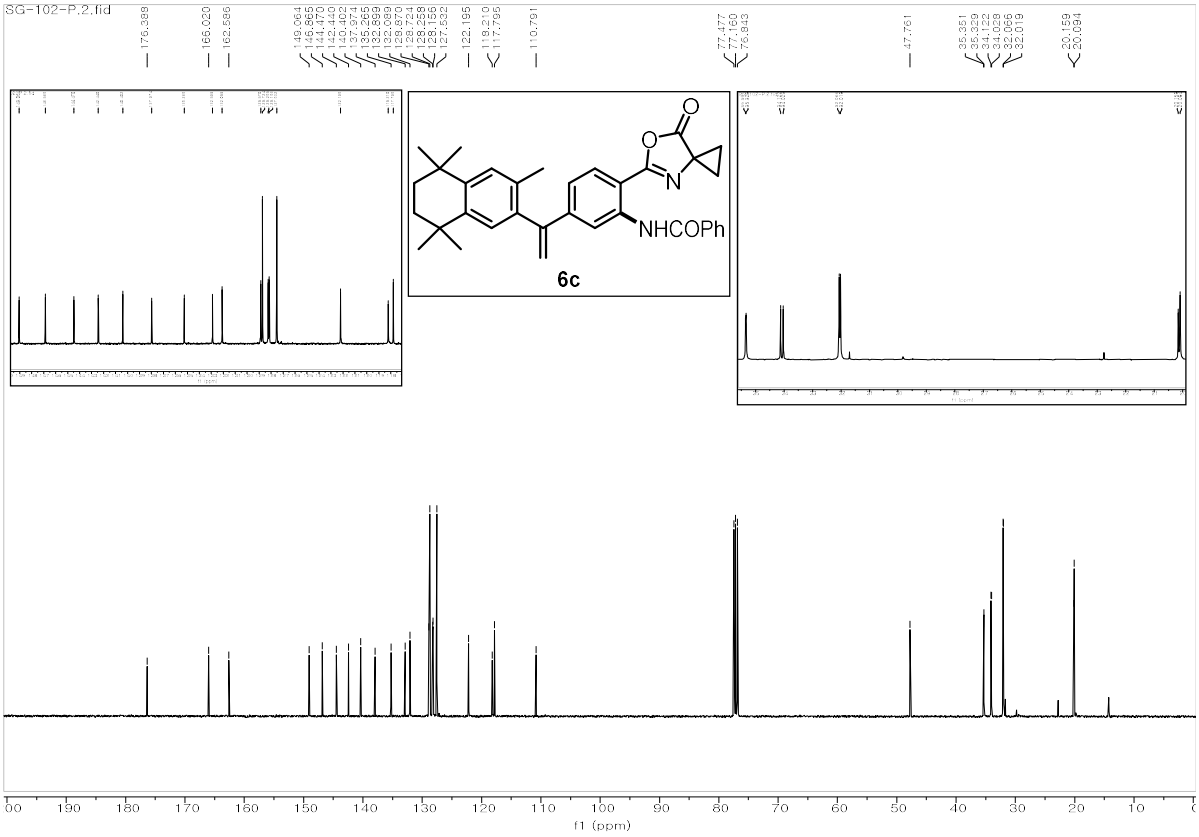

SG-95-IP2.1.fid
11.951
8.807
8.786
7.833
7.816
7.523
7.511
7.486
7.467
7.420
7.400
7.382
7.190
6.834
6.814
2.320
1.824
OAc
O
O
N
NHCOPh
6a
f1 (ppm)

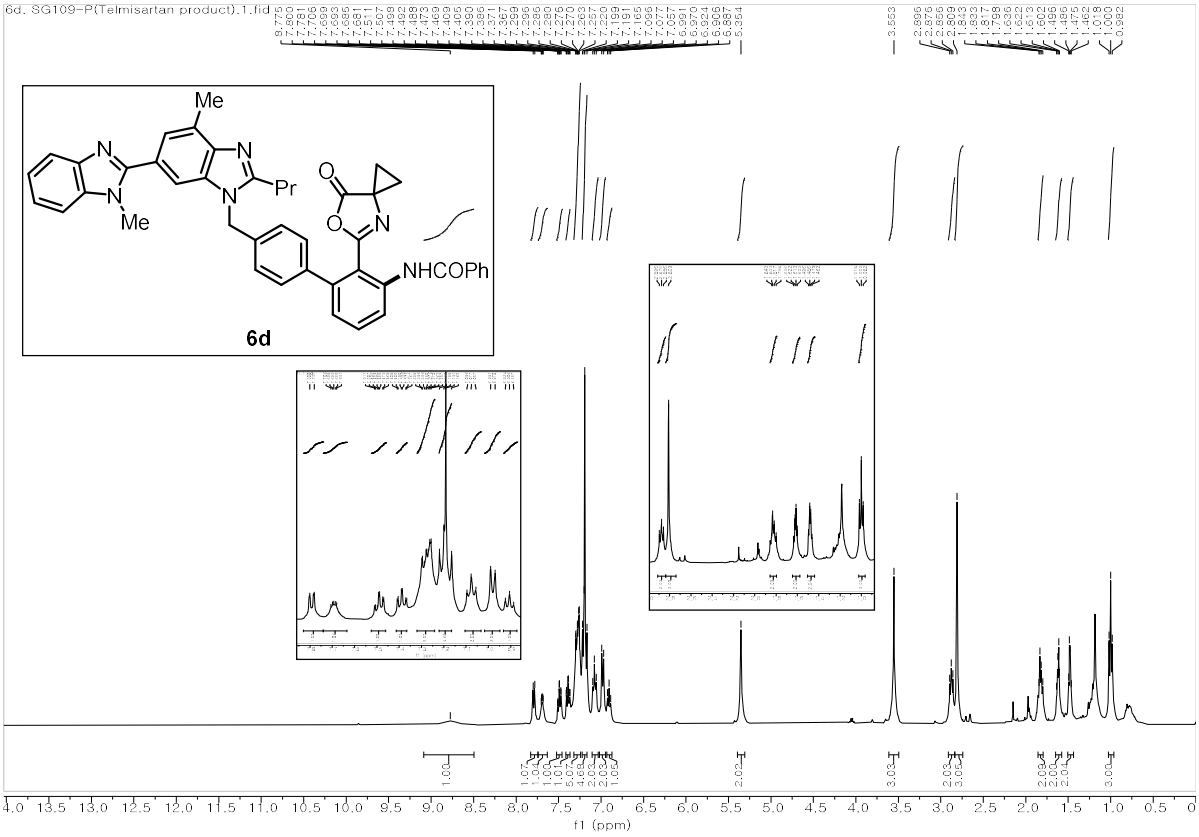

SG-95-IP2.2.fid
175.989
169.940
165.947
160.916
150.081
141.266
135.196
133.971
132.279
129.776
127.471
118.674
118.619
106.898
77.477
77.160
76.843
46.453
21.166
20.386
OAc
O
O
N
NHCOPh
6a
f1 (ppm)

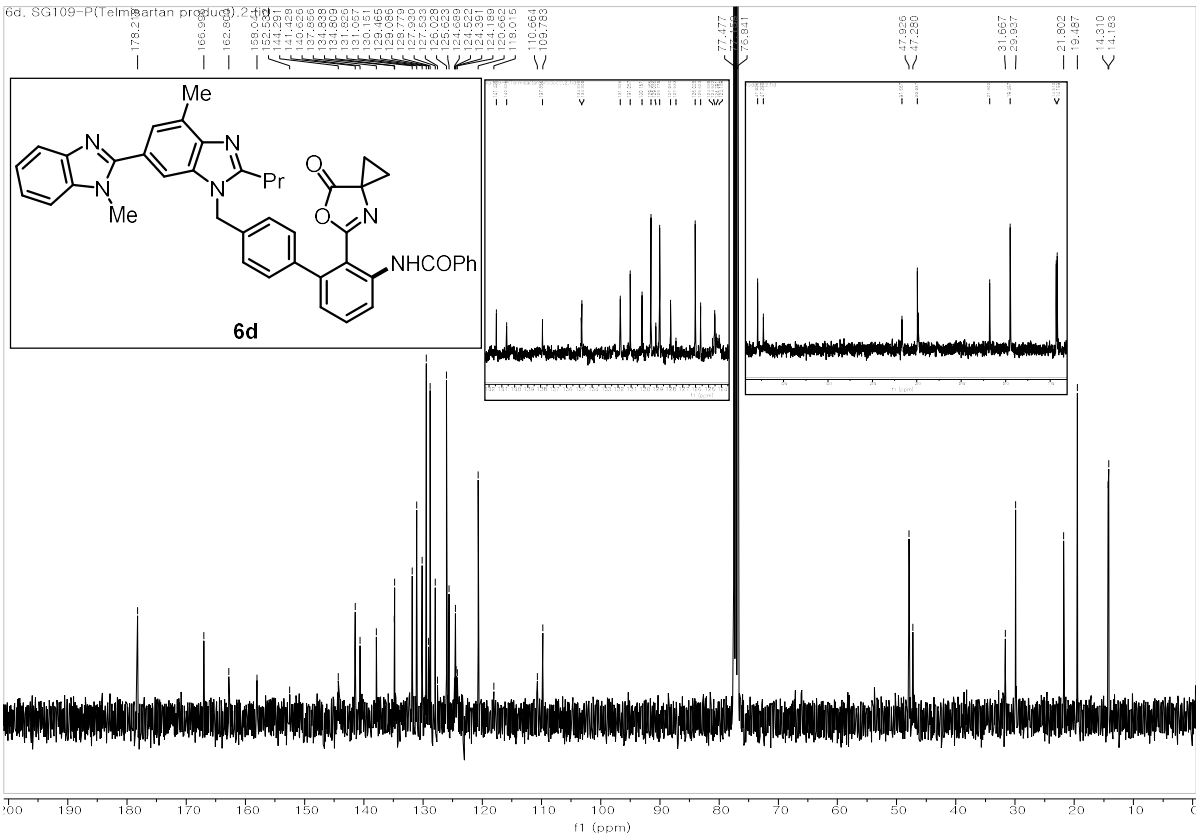

6b. MS-291-P4.2.fid
11.747
8.932
7.888
7.869
7.561
7.552
7.489
7.455
7.422
7.402
7.384
7.213
2.875
2.869
2.849
2.842
2.623
2.499
2.476
2.451
2.318
2.307
2.290
2.278
2.262
2.251
2.127
2.102
2.080
2.069
2.052
2.039
2.022
2.014
2.003
1.996
1.990
1.962
1.819
1.747
1.639
1.631
1.604
1.589
1.582
1.575
1.568
1.550
1.507
1.484
1.467
1.445
1.435
1.426
1.414
1.398
O
O
N
H
H
H
NHCOPh
O
Me
6b
1.02
1.00
2.03
1.01
3.04
2.04
f1 (ppm)

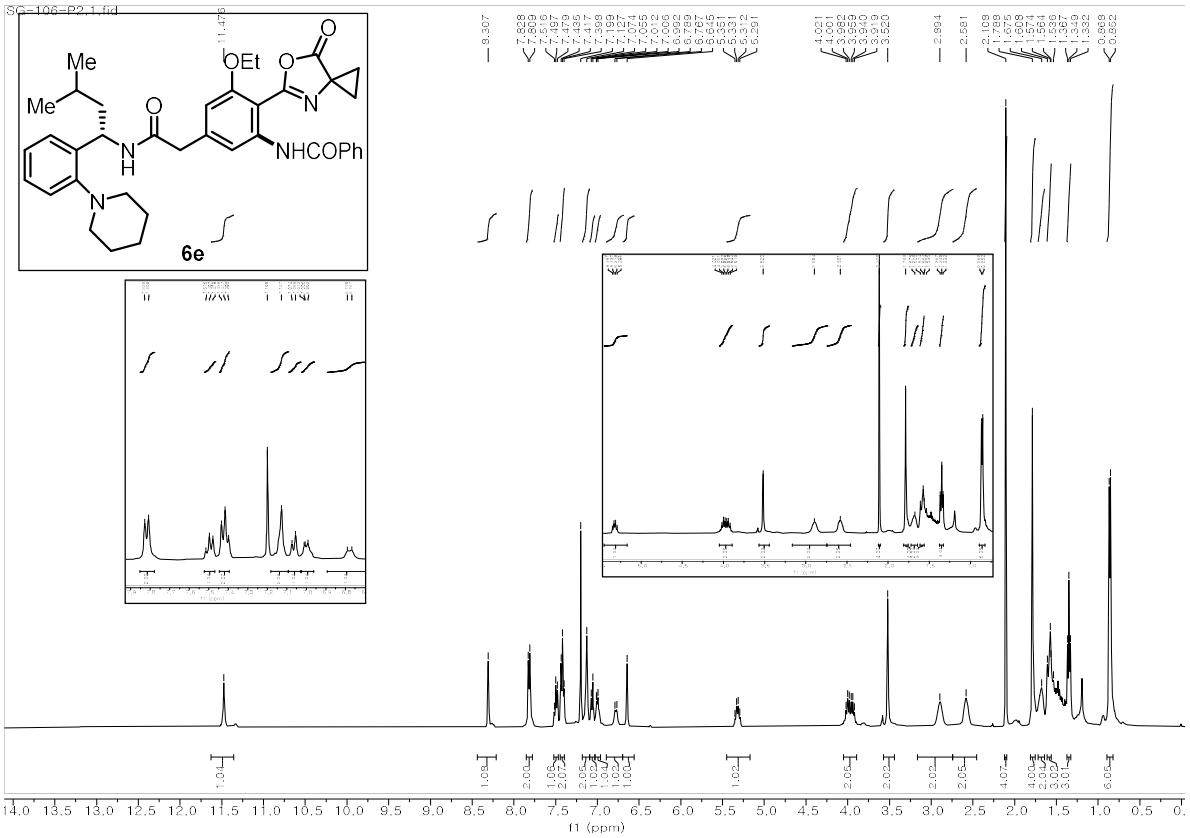

6b. MS-291-P4.2.fid
176.464
165.819
162.460
146.544
137.968
135.163
132.032
131.592
128.995
128.710
127.422
119.940
117.405
109.699
77.477
77.159
76.841
50.677
49.031
47.694
45.146
37.845
35.884
31.583
28.753
26.395
25.734
21.694
20.043
13.930
O
O
N
H
H
H
NHCOPh
O
Me
6b
f1 (ppm)

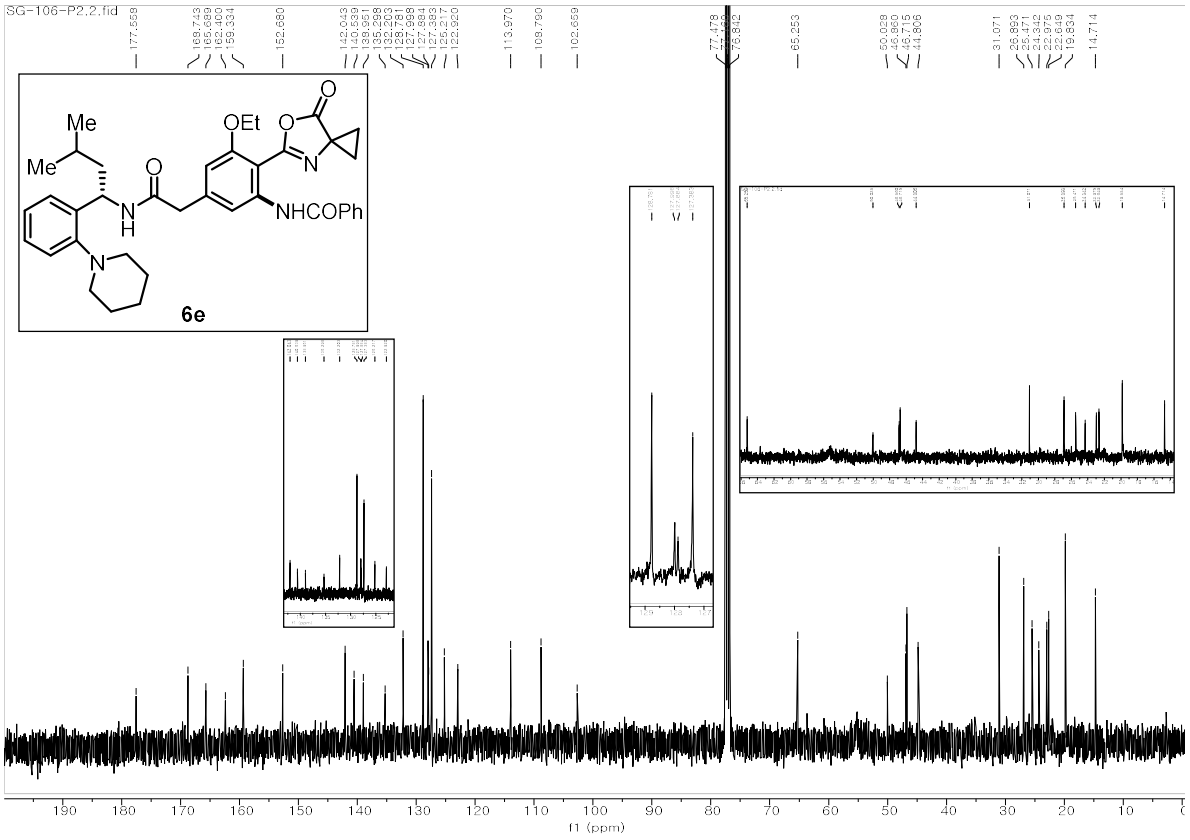

SG-102-P.1.fid
11.839
8.122
7.835
7.817
7.669
7.654
7.426
7.407
7.389
7.380
7.330
7.313
7.112
7.035
6.961
6.758
6.689
5.841
5.259
1.875
1.750
1.572
1.179
1.161
NHCOPh
6c
f1 (ppm)

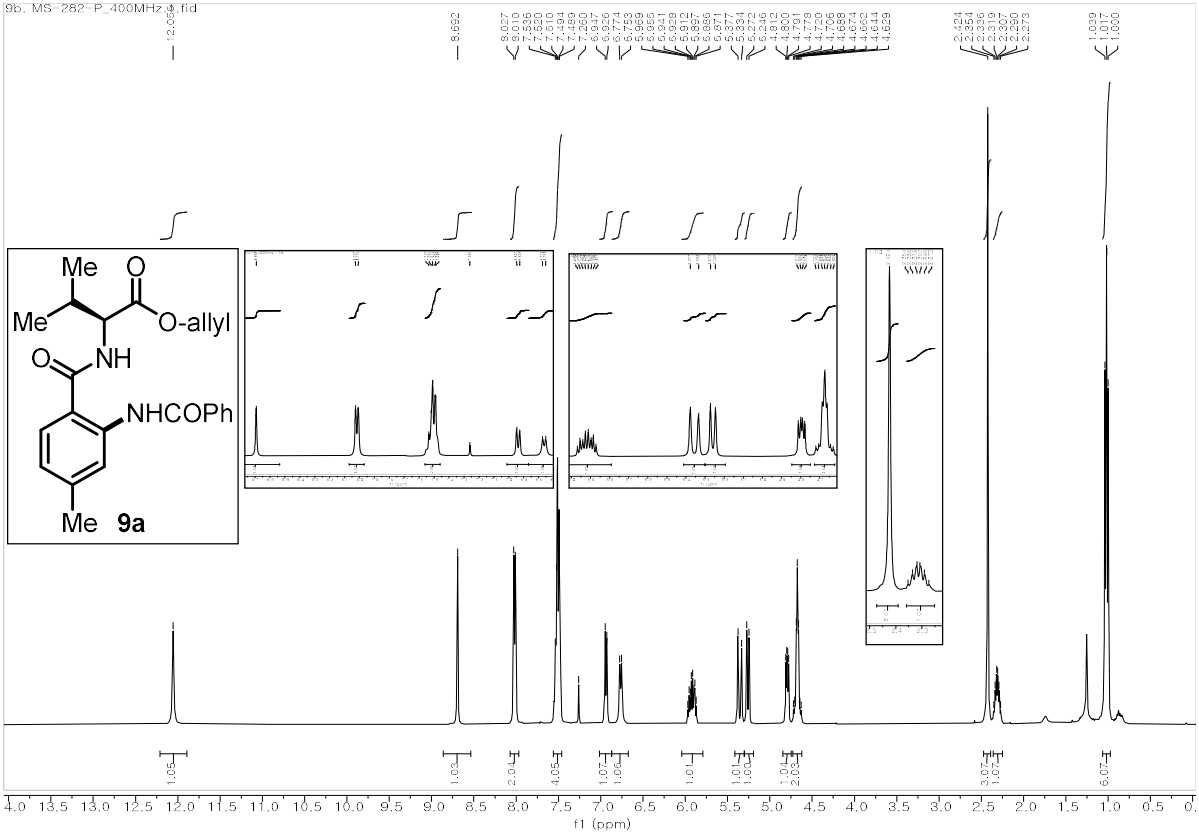

SG-102-P.2.fid
176.389
166.020
162.586
149.064
146.845
144.470
142.440
140.462
137.974
135.845
132.869
132.089
129.870
128.754
128.258
128.156
127.632
122.195
119.210
117.795
110.791
77.477
77.160
76.843
47.761
35.351
35.329
34.122
34.068
32.066
32.019
30.159
20.084
NHCOPh
6c
f1 (ppm)

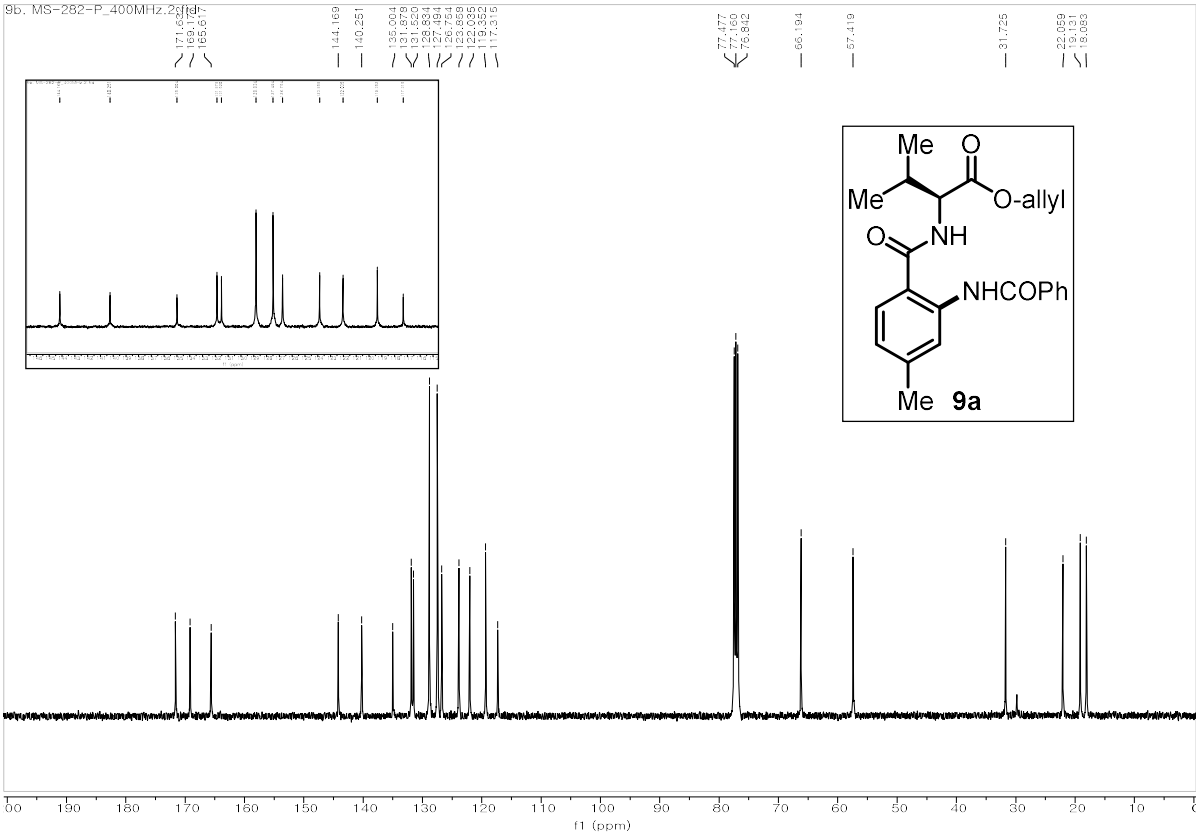

6d. SG109-P(Telmisartan product).1.fid
Me
N
N
Me
N
Pr
O
O
N
NHCOPh
6d
f1 (ppm)

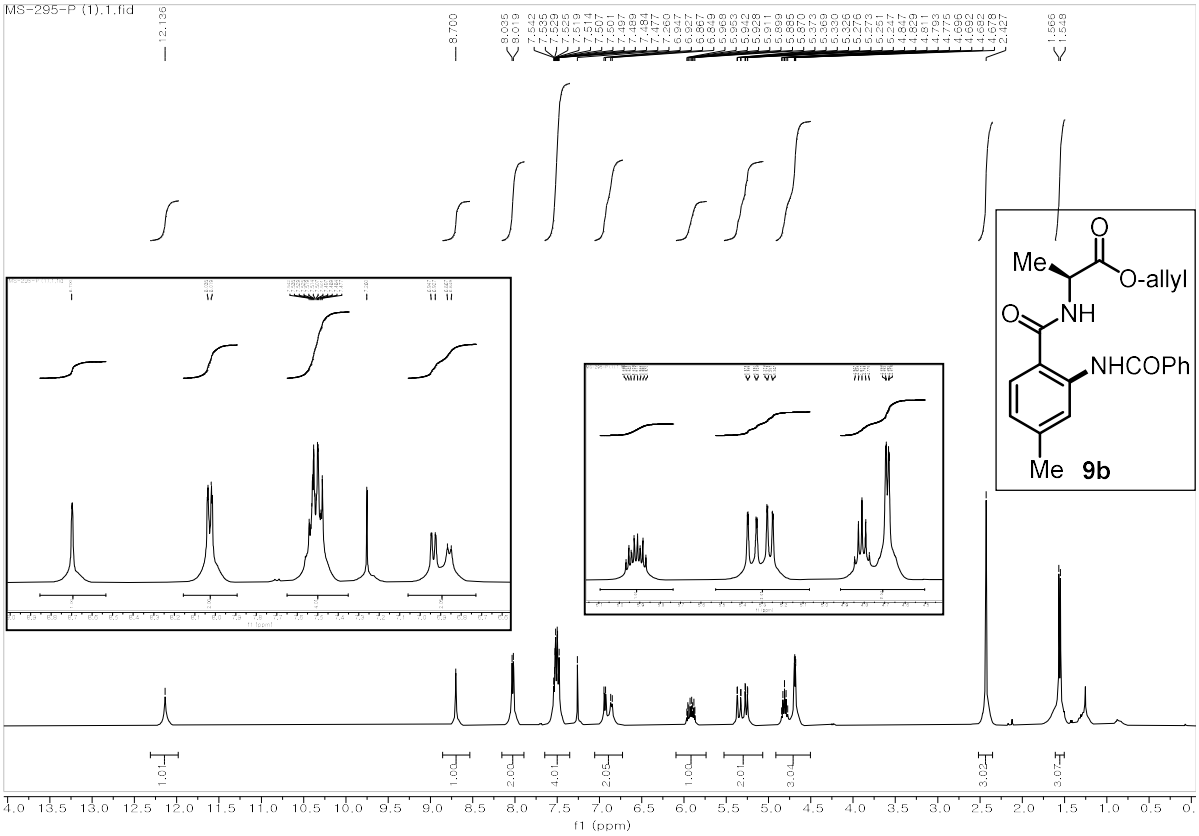

6d. SG109-P(Telmisartan product).2.fid
Me
N
N
Me
N
Pr
O
O
N
NHCOPh
6d
f1 (ppm)

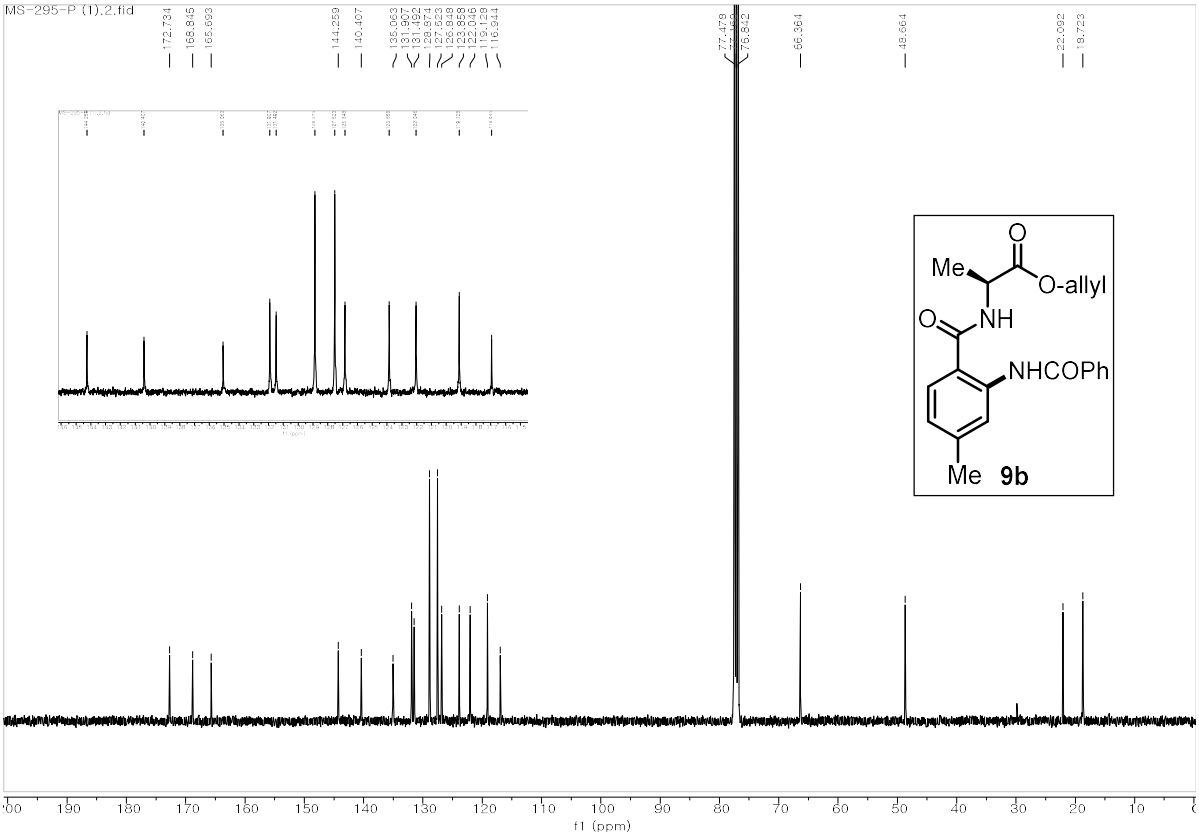

SG-106-P2.1.fid
Me
Me
OEt
O
O
N
O
N
H
NHCOPh
N
6e
f1 (ppm)

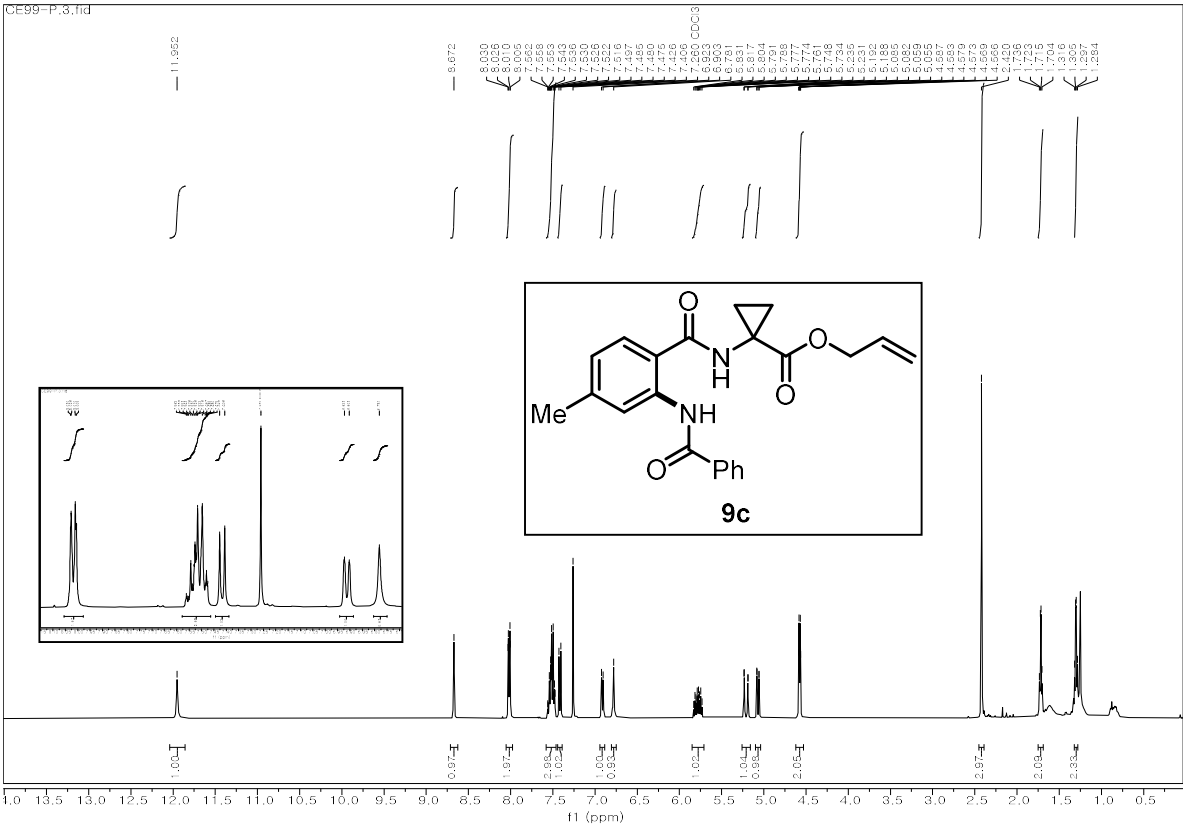

SG-106-P2.2.fid
Me
Me
OEt
O
O
N
O
N
H
NHCOPh
N
6e
f1 (ppm)

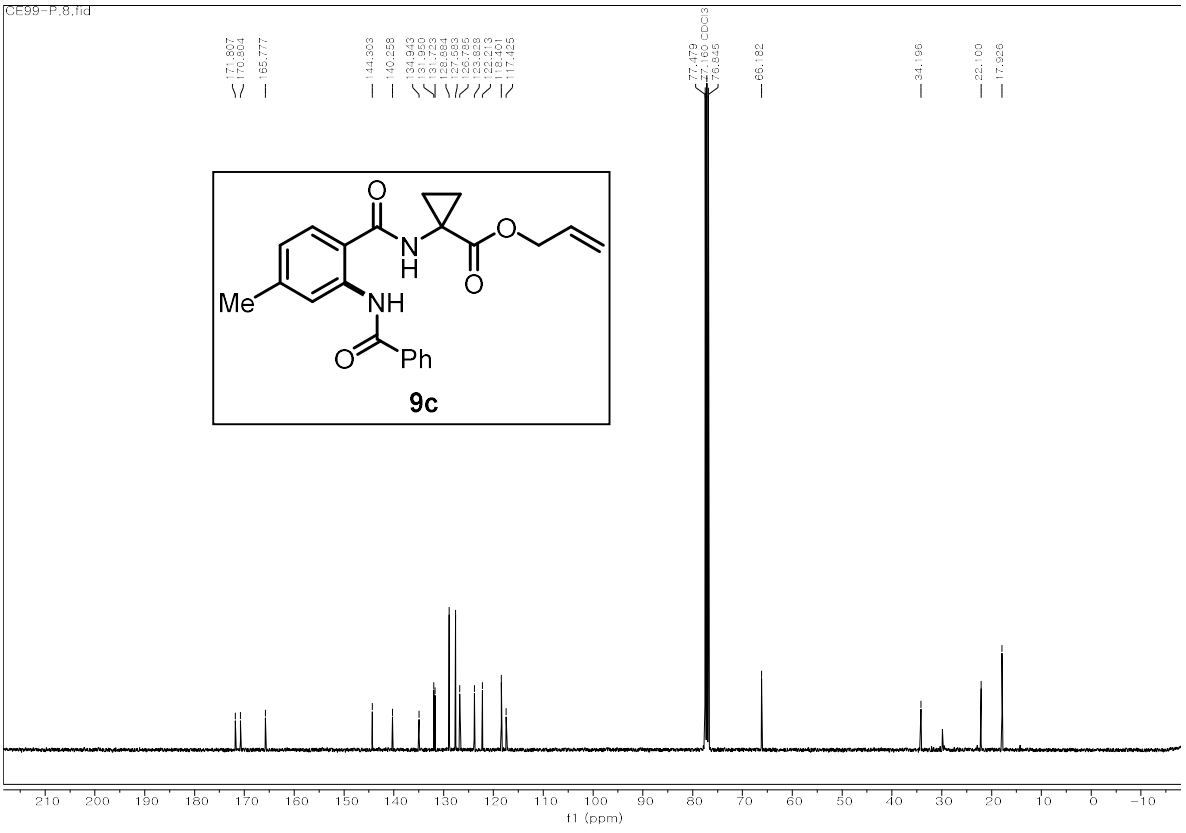

9b. MS-282-P_400MHz.1.fid
Me
O
Me
O-allyl
O
NH
NHCOPh
Me 9a
f1 (ppm)

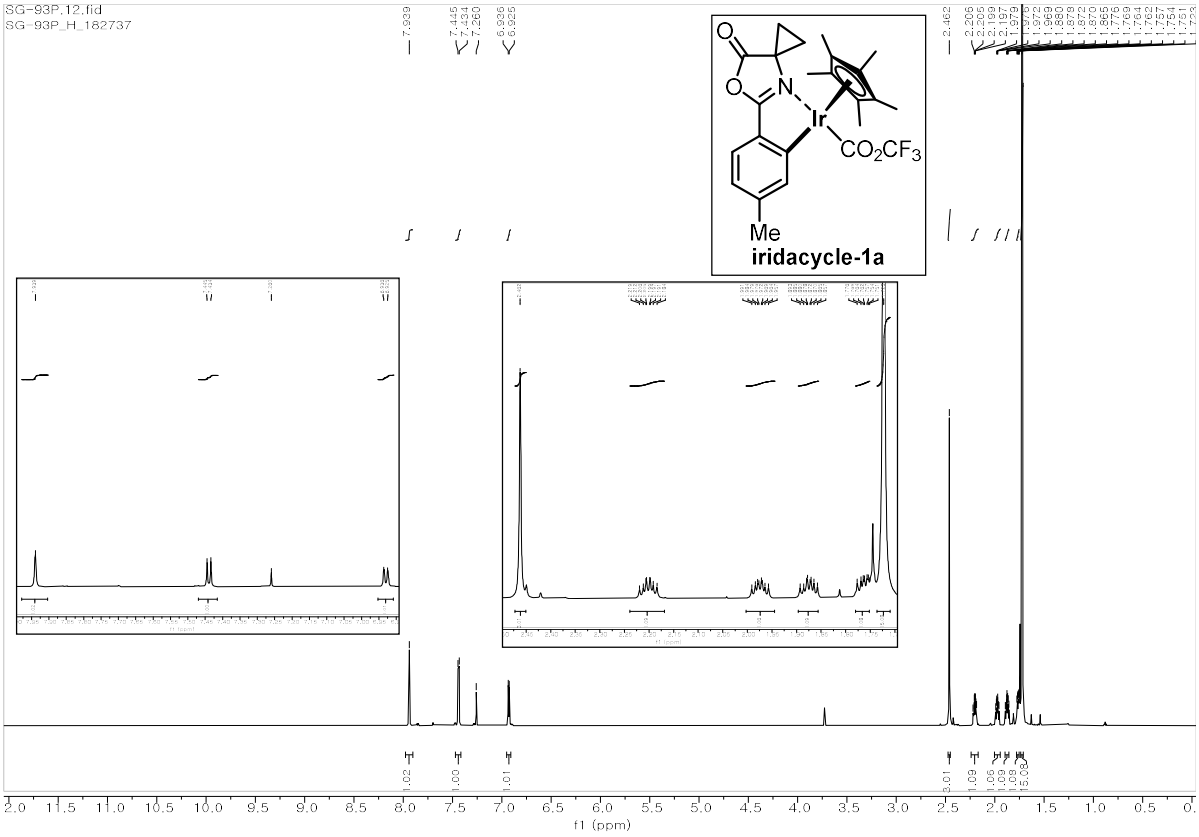

9b. MS-282-P_400MHz.2.fid
Me
O
Me
O-allyl
O
NH
NHCOPh
Me 9a
f1 (ppm)

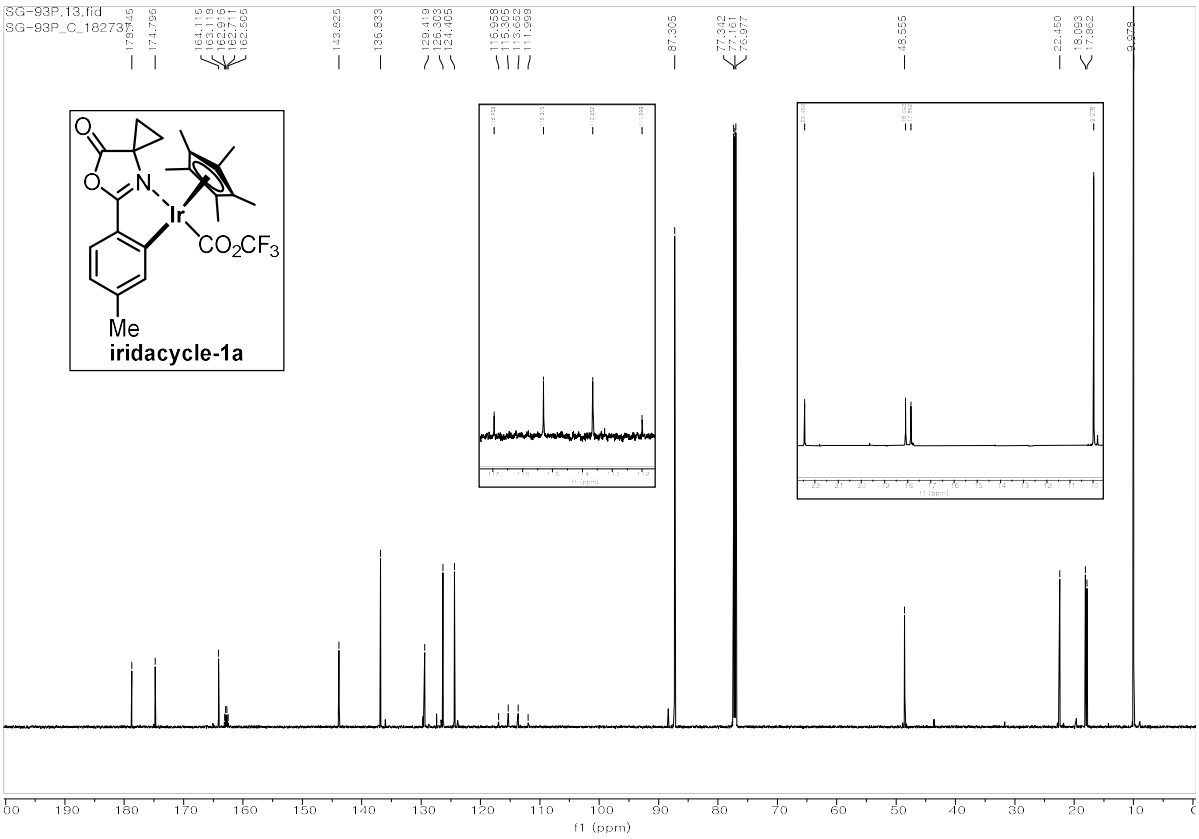

MS-295-P (1).1.fid
Me
O-allyl
NH
NHCOPh
Me 9b
f1 (ppm)

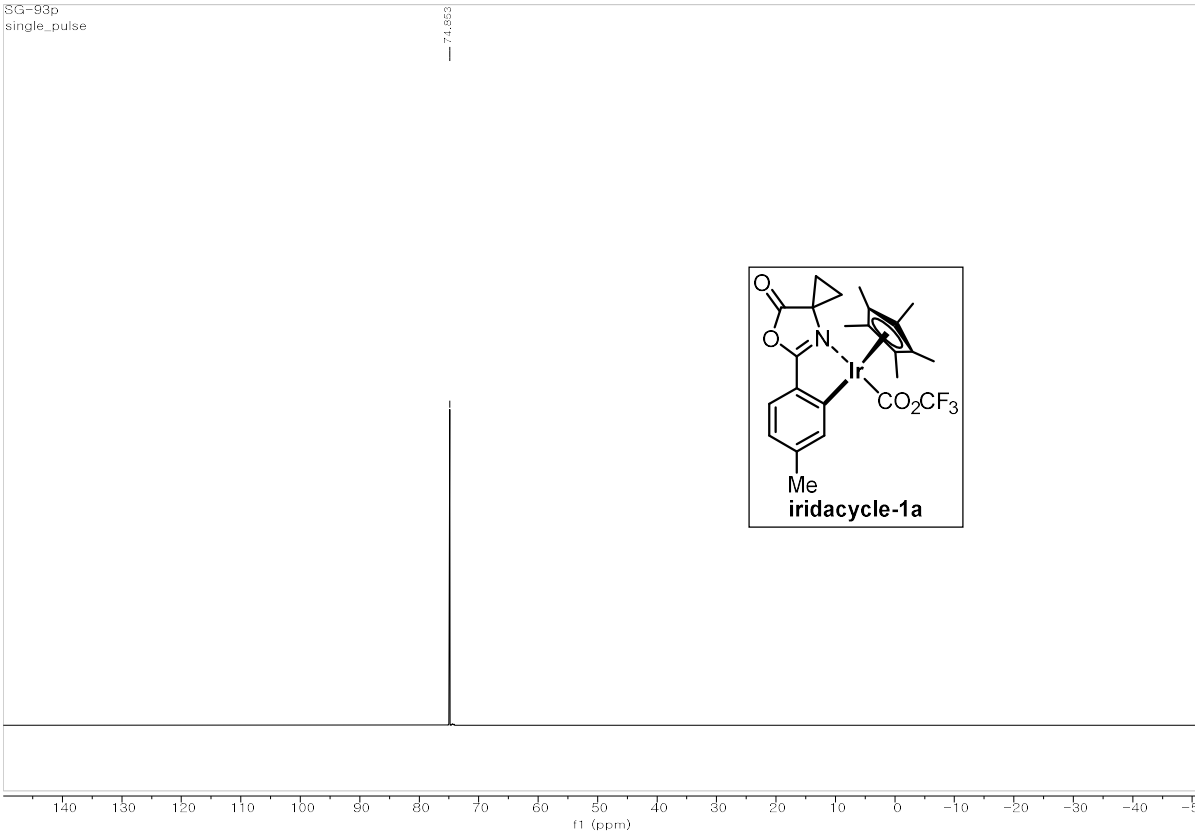

MS-295-P (1).2.fid
Me
O-allyl
NH
NHCOPh
Me 9b
f1 (ppm)


CE99-P.3.fid
11.952
9.672
8.030
8.026
8.010
8.006
7.568
7.563
7.543
7.538
7.526
7.522
7.516
7.497
7.495
7.480
7.475
7.436
7.426
7.280 CDCl3
6.903
6.781
6.831
6.817
6.804
5.791
5.788
5.777
5.774
5.761
5.748
5.234
5.231
5.192
5.188
5.085
5.059
5.055
4.587
4.583
4.573
4.569
4.566
4.430
4.426
1.715
1.704
1.316
1.305
1.297
1.284
Me
NH
O
Ph
9c
1.00
0.97
1.97
2.09
1.03
1.00
0.93
1.02
1.04
0.98
2.05
2.97
2.09
2.33
f1 (ppm)


CE99-P.8.fid
171.807
170.804
165.777
144.303
140.258
134.943
131.950
131.723
128.684
127.683
126.785
123.828
123.213
118.401
117.425
77.479
77.160 CDCl3
76.845
66.182
34.196
22.100
17.926
Me
NH
O
Ph
9c
f1 (ppm)


SG-93P_12.fid
SG-93P_H_182737
iridacycle-1a
f1 (ppm)


SG-93P_13.fid
SG-93P_C_182737
iridacycle-1a
f1 (ppm)


SG-93p
single_pulse
74.853
O
O
N
Ir
CO2CF3
Me
iridacycle-1a
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
f1 (ppm)

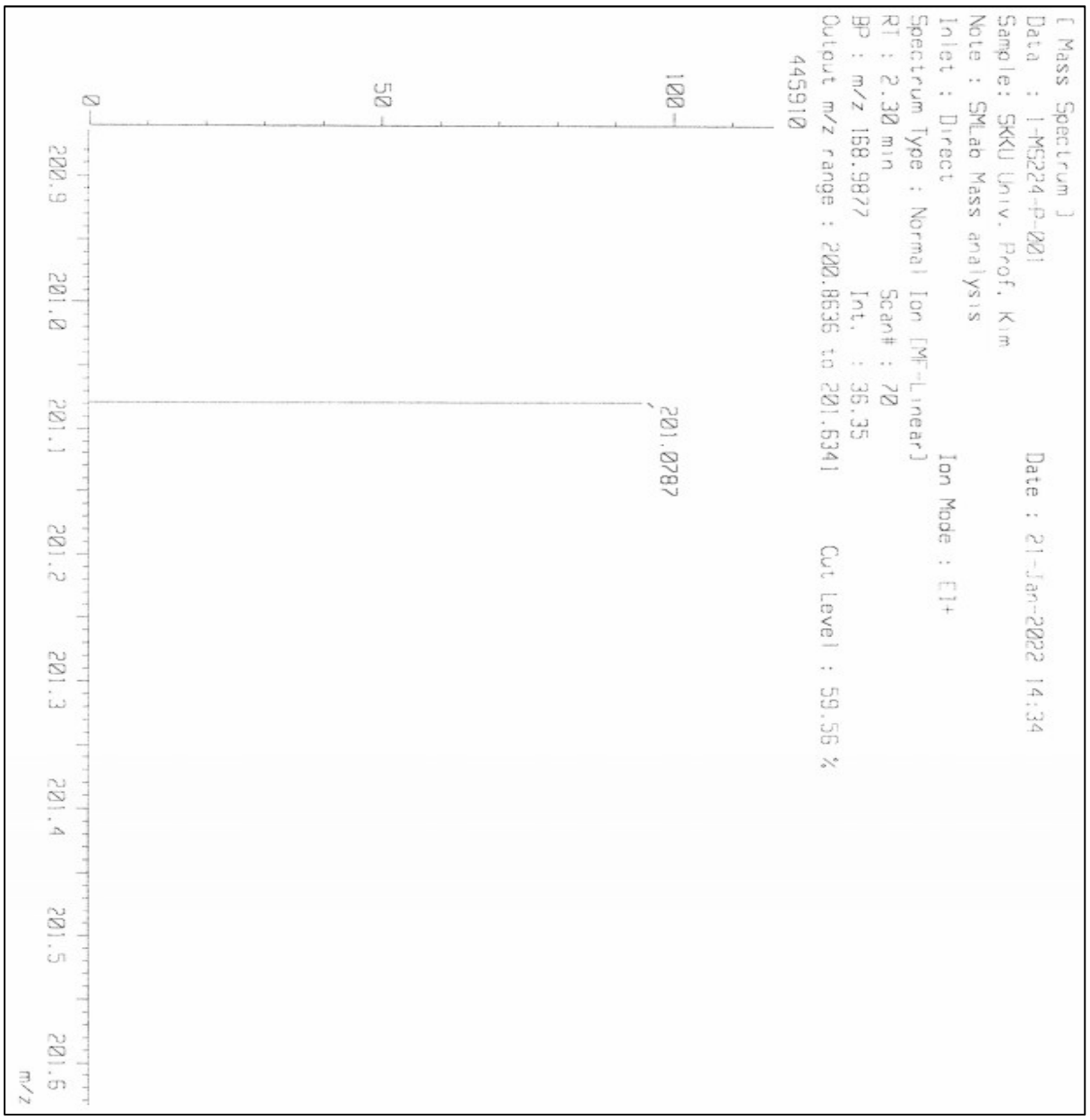
HRMS data of all compounds

[Elemental Composition]
Data : 1-MS224-P-001
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.30 min
Elements : C 13/0, H 12/0, O 3/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 21-Jan-2022 14:34
Ion Mode : EI+
Scan#: 70

1a

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
201.0787	94.6	-1.4 / -0.3	8.0	C 12 H 11 O 2 N



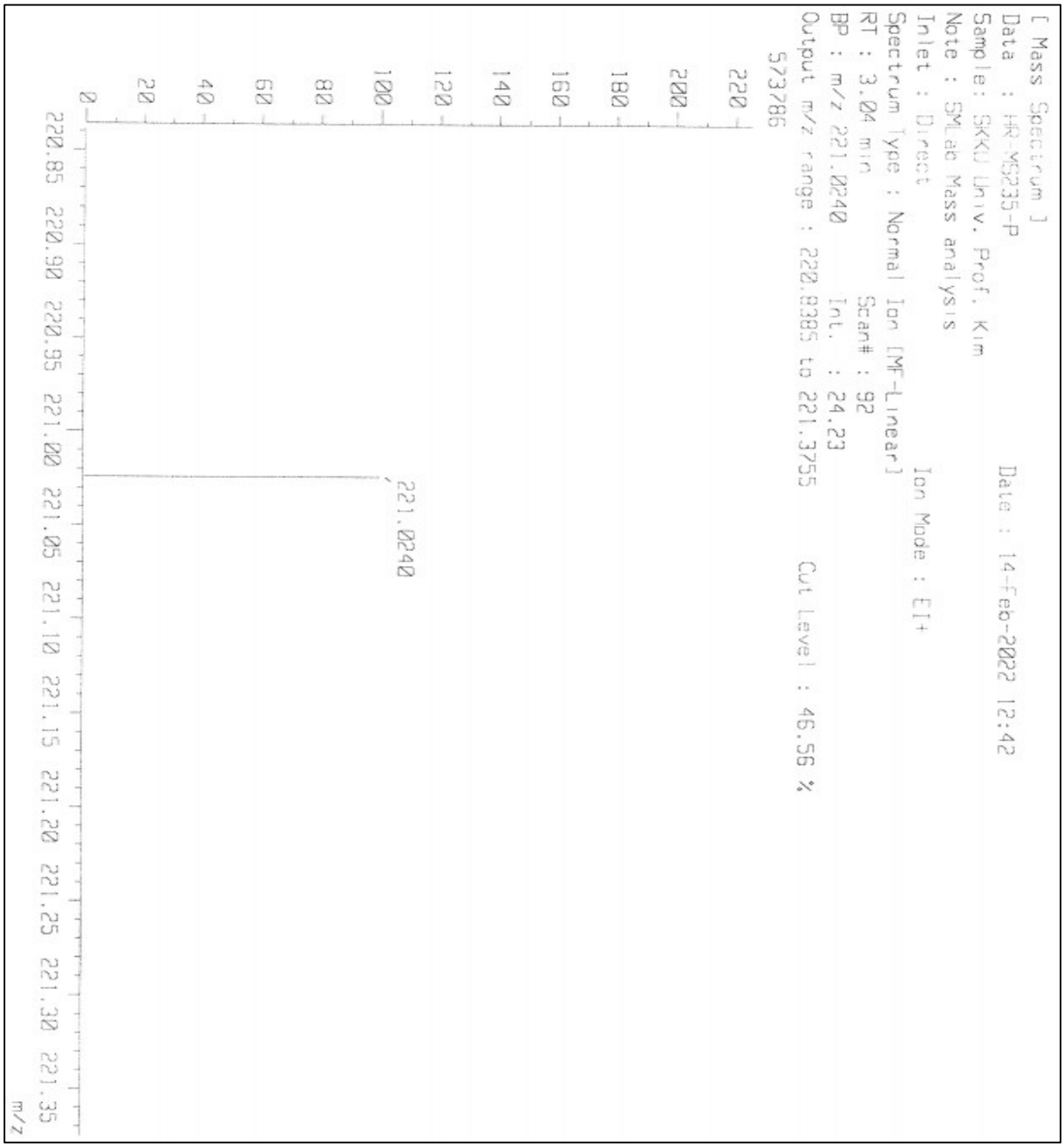
[Elemental Composition]

Page: 1

1b

Data : HR-MS235-P
Date : 14-Feb-2022 12:42
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
Ion Mode : EI+
RT : 3.04 min
Scan#: 92
Elements : C 15/0, H 12/0, O 2/0, N 1/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
221.0240	100.0	-1.5 / -0.3	8.0	C 11 H 8 O 2 N Cl



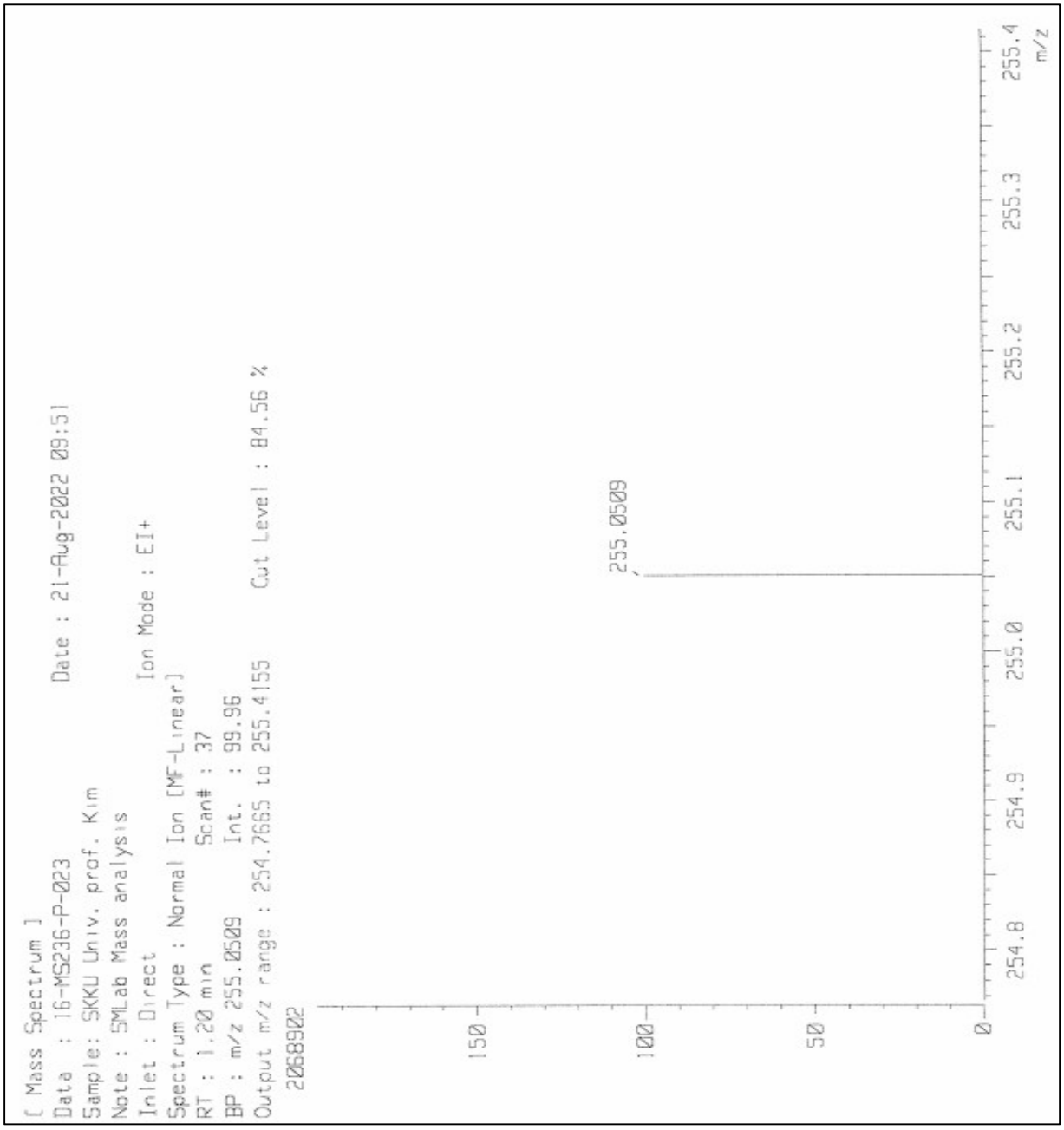
[Elemental Composition]
Data : 16-MS236-P-023
Sample: SKKU Univ. prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.20 min
Elements : C 13/0, H 10/0, O 2/0, N 1/0, F 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 21-Aug-2022 09:51

Ion Mode : EI+
Scan#: 37

1c

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
255.0509	100.0	+0.6 / +0.2	8.0	C 12 H 8 O 2 N F 3

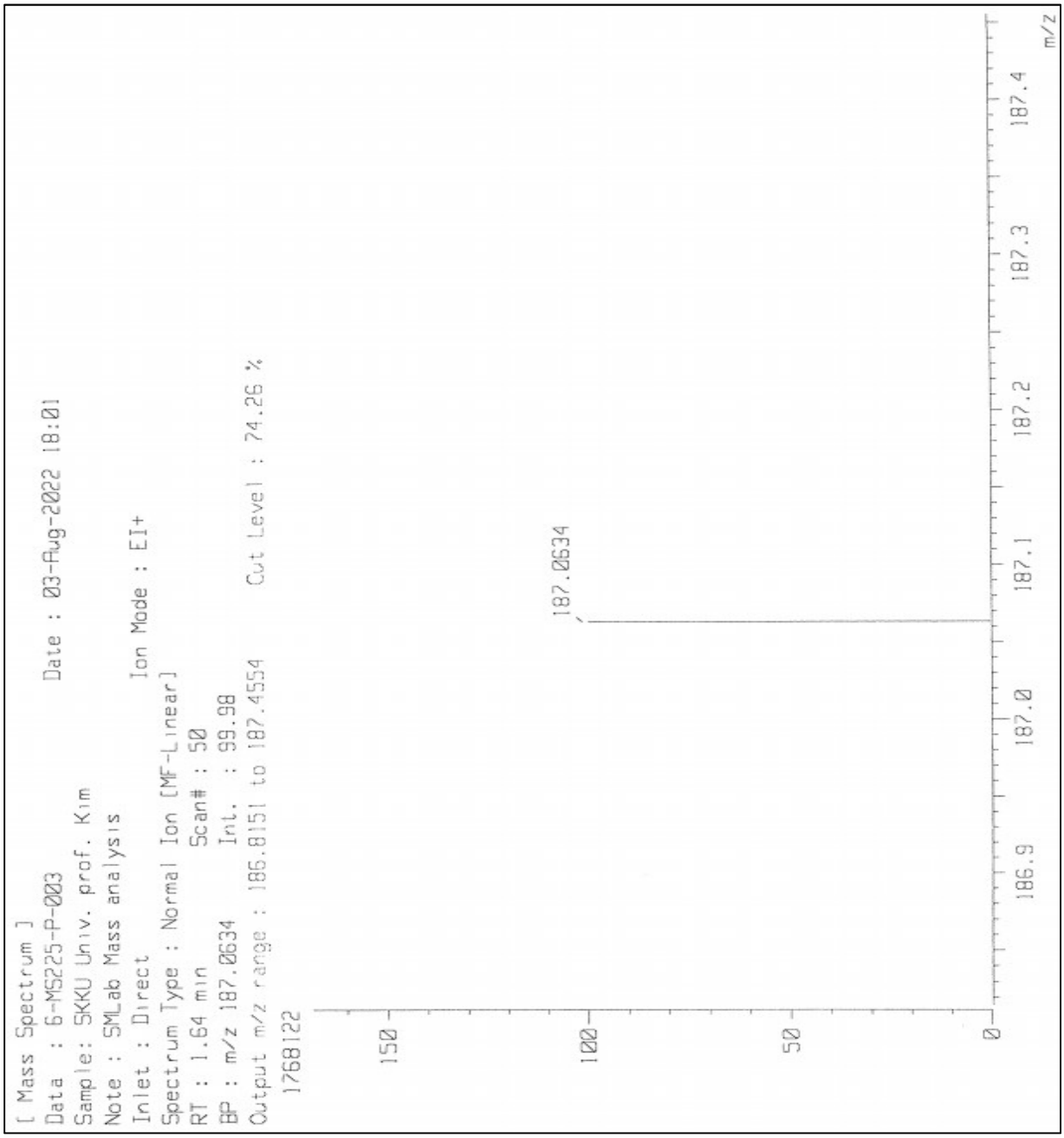


1d

[Elemental Composition]
Data : 6-MS225-P-003
Sample: SKKU Univ. prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.64 min
Elements : C 12/0, H 10/0, O 2/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 03-Aug-2022 18:01
Ion Mode : EI+
Scan#: 50

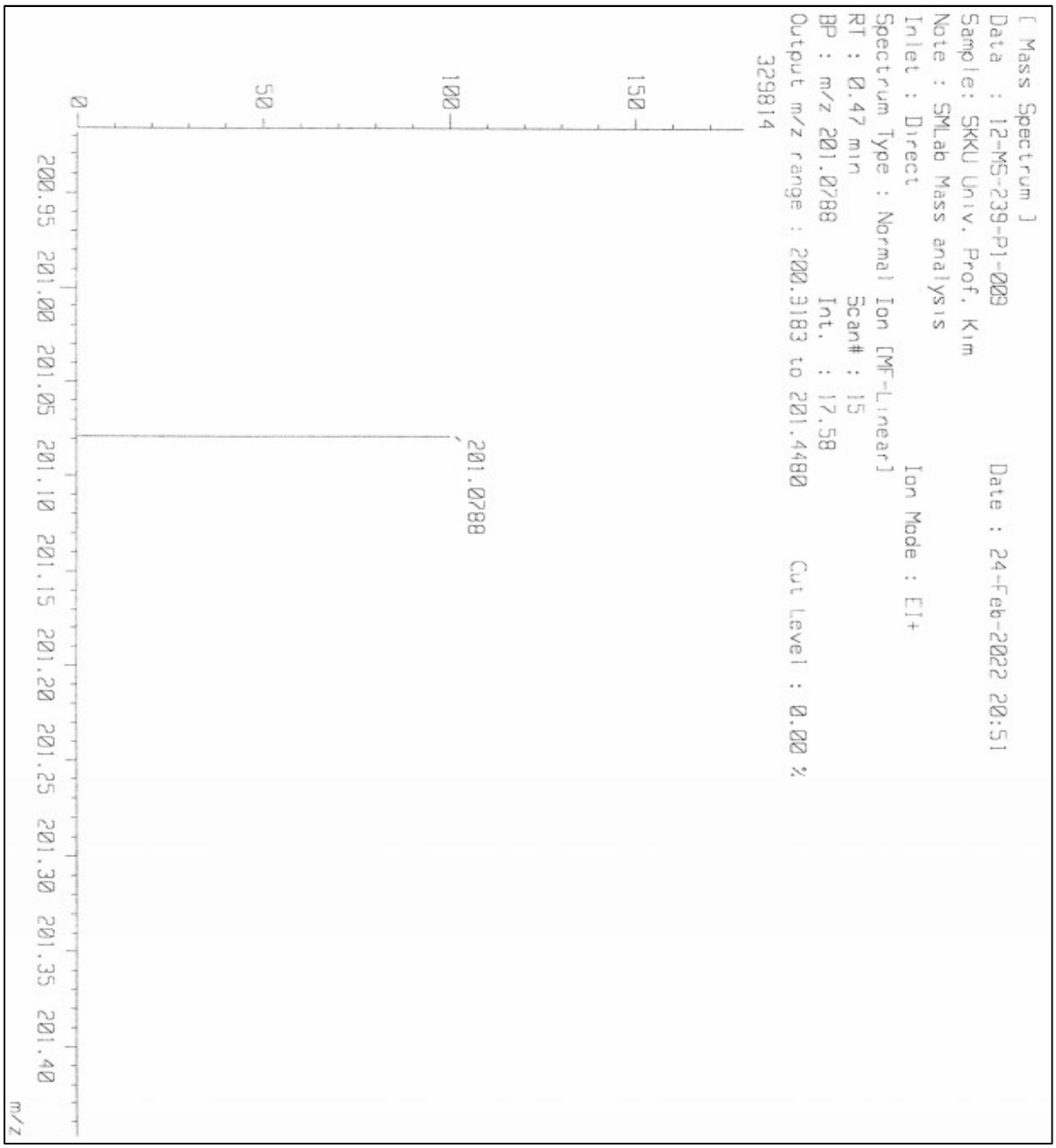
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
187.0634	100.0	+0.5 / +0.1	8.0	C 11 H 9 O 2 N



[Elemental Composition]
Data : 12-MS-239-P1-009 Date : 24-Feb-2022 20:51
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 0.47 min Scan#: 15
Elements : C 12/0, H 12/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
201.0788	100.0	-1.0 / -0.2	8.0	C 12 H 11 O 2 N

1e



[Elemental Composition] Page: 1

Data : 13-MS-240-P1-016 Date : 24-Feb-2022 21:07

1f

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

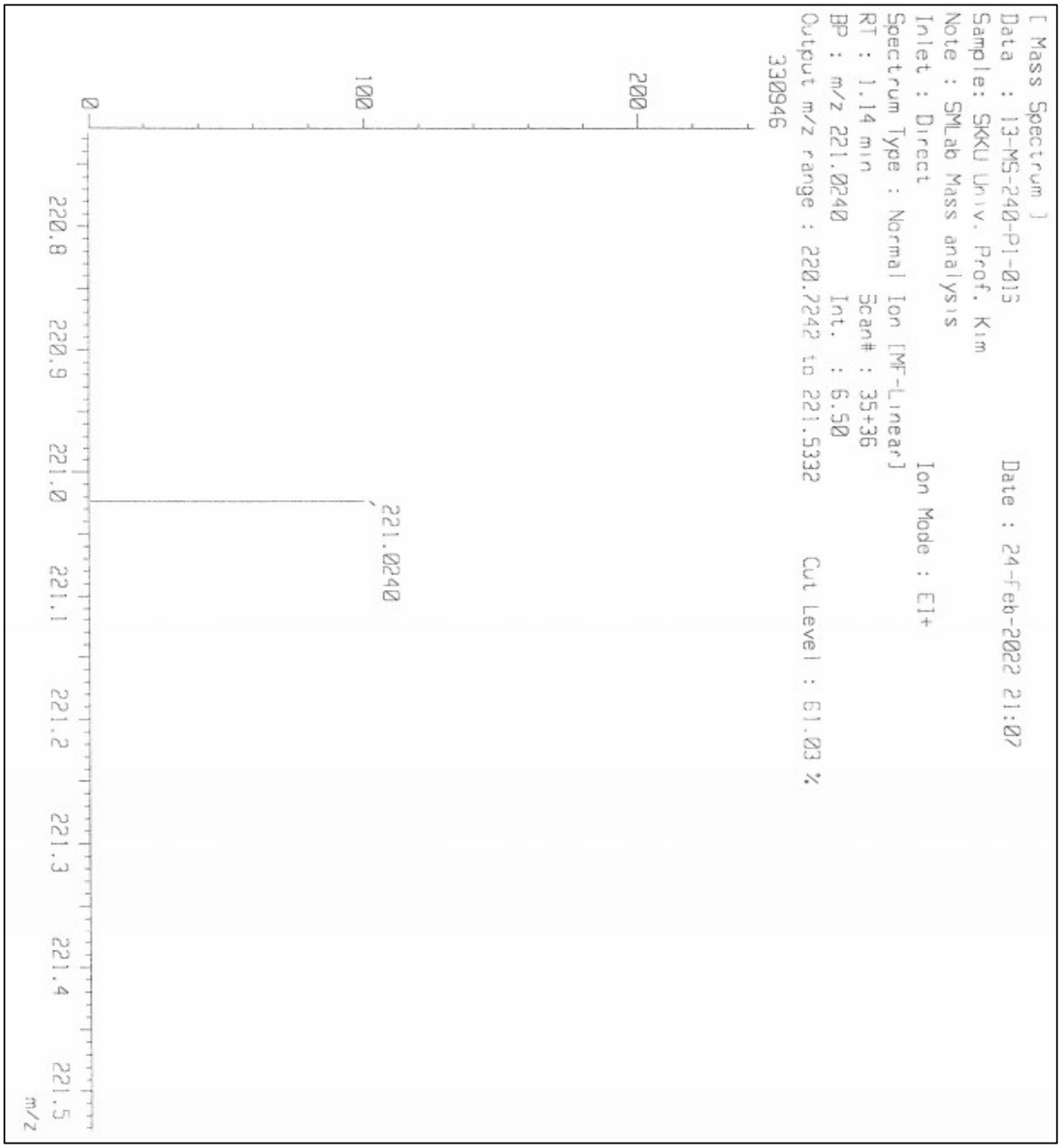
RT : 1.14 min Scan#: 35+36

Elements : C 12/0, H 12/0, O 3/0, N 2/0, Cl 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
221.0240	100.0	-1.5 / -0.3	8.0	C 11 H 8 O 2 N Cl



[Elemental Composition] Page: 1

Data : 14-MS-241-P-013 Date : 24-Feb-2022 20:59

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

1g

Inlet : Direct Ion Mode : EI+

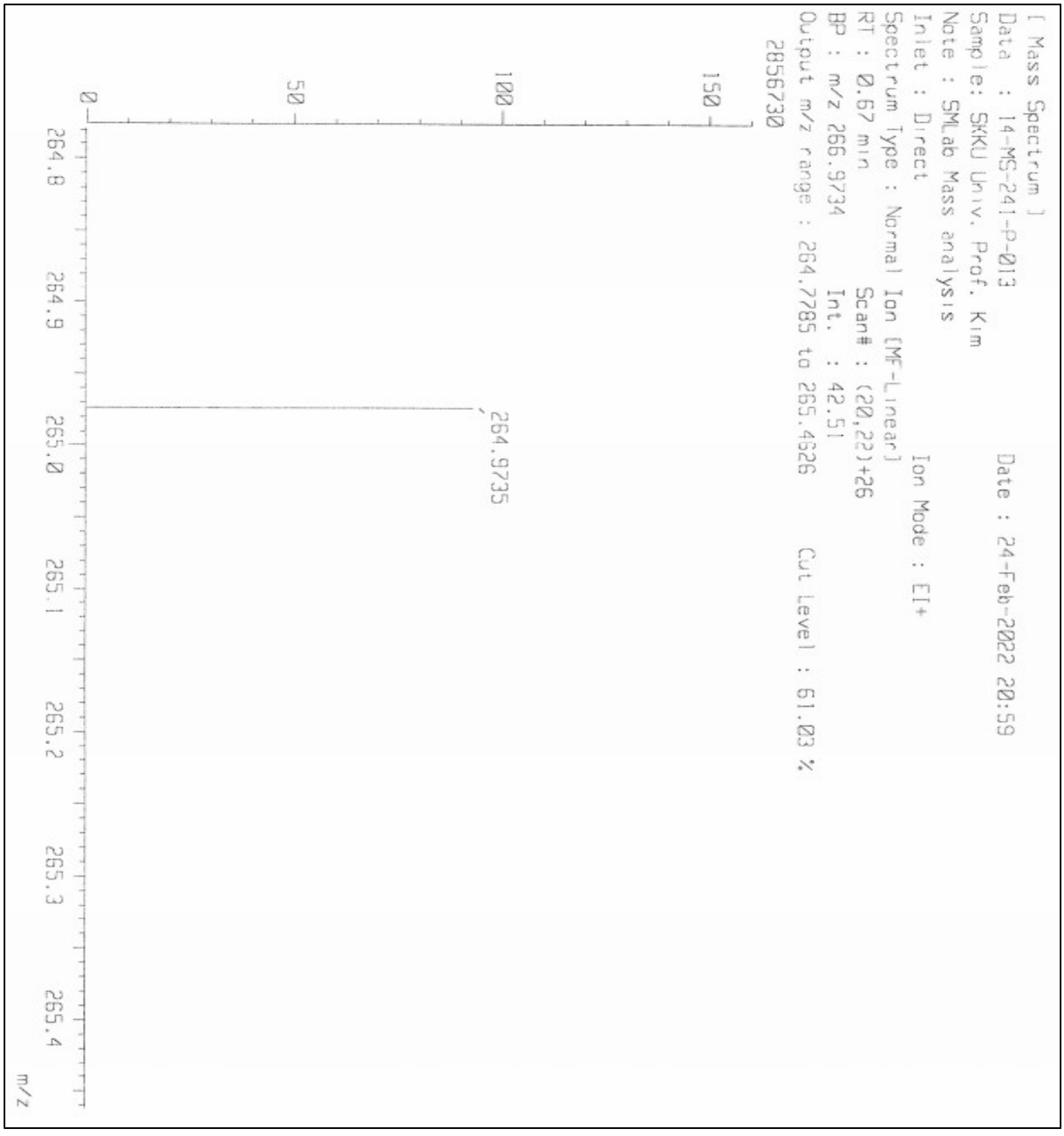
RT : 0.67 min Scan#: (20,22)+26

Elements : C 12/0, H 12/0, O 2/0, N 2/0, Br 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
264.9735	93.2	-1.4 / -0.4	8.0	C 11 H 8 O 2 N Br



[Elemental Composition] Page: 1

Data : 4-CE24-P-006 Date : 11-Mar-2022 17:44

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

1h

Inlet : Direct Ion Mode : EI+

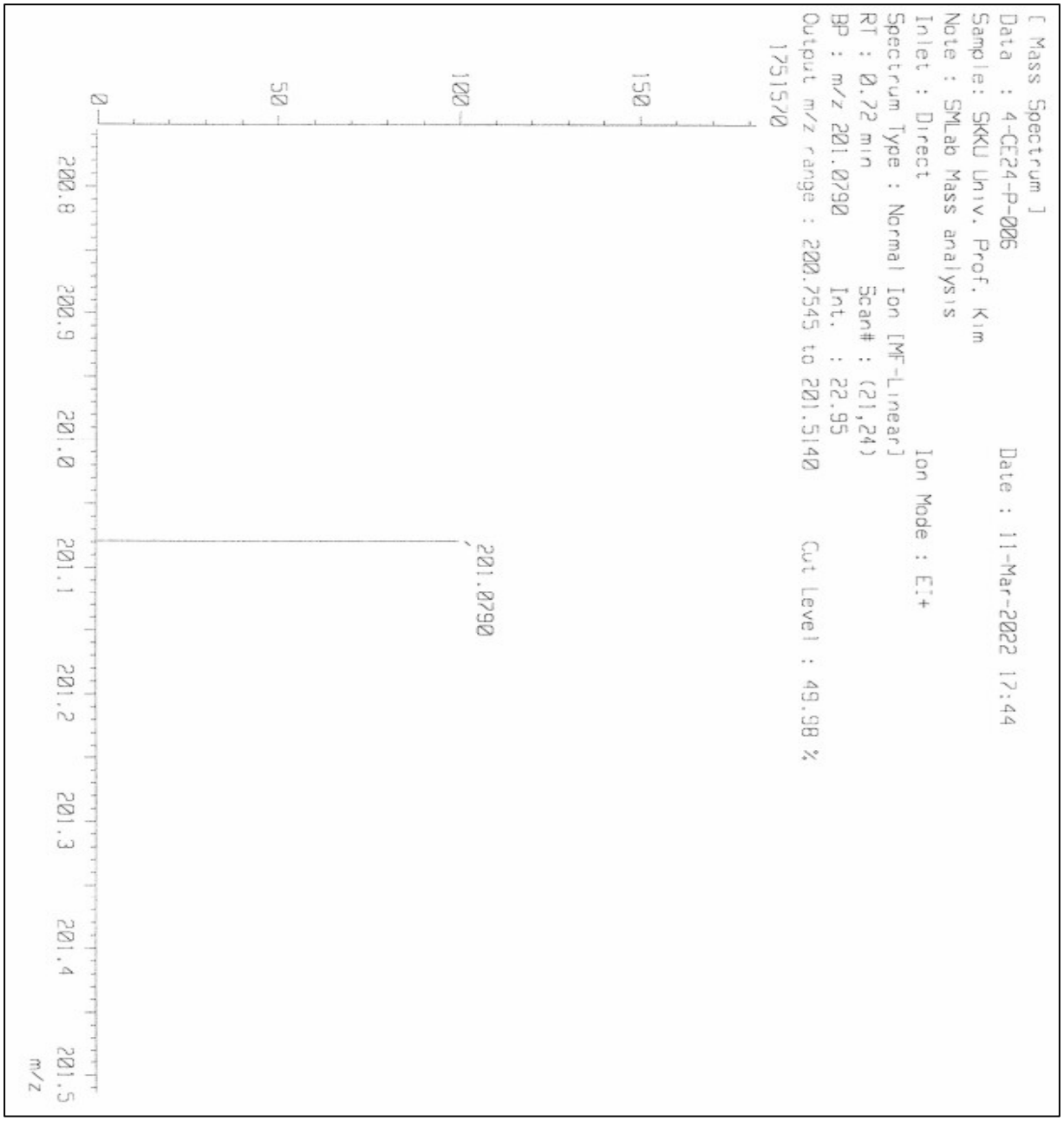
RT : 0.72 min Scan#: (21,24)

Elements : C 13/0, H 12/0, O 3/0, N 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
201.0790	100.0	-0.1 / +0.0	8.0	C 12 H 11 O 2 N



[Elemental Composition] Page: 1

Data : 3-MS243-P-003 Date : 26-Mar-2022 08:24

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

RT : 1.00 min Scan#: (30,32)

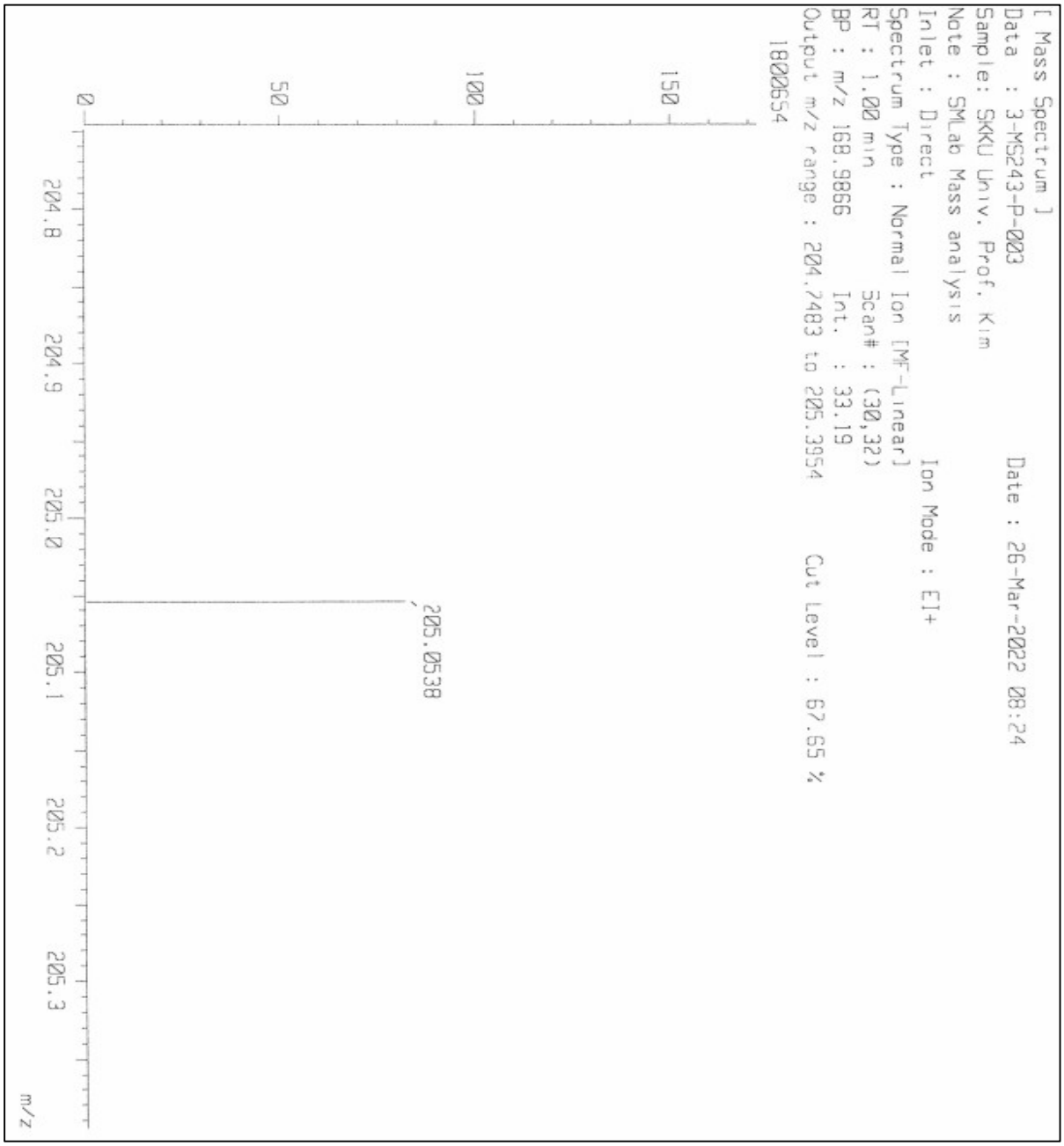
Elements : C 12/0, H 10/0, O 3/0, N 1/0, F 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
205.0538	81.8	-0.7 / -0.1	8.0	C 11 H 8 O 2 N F

1i



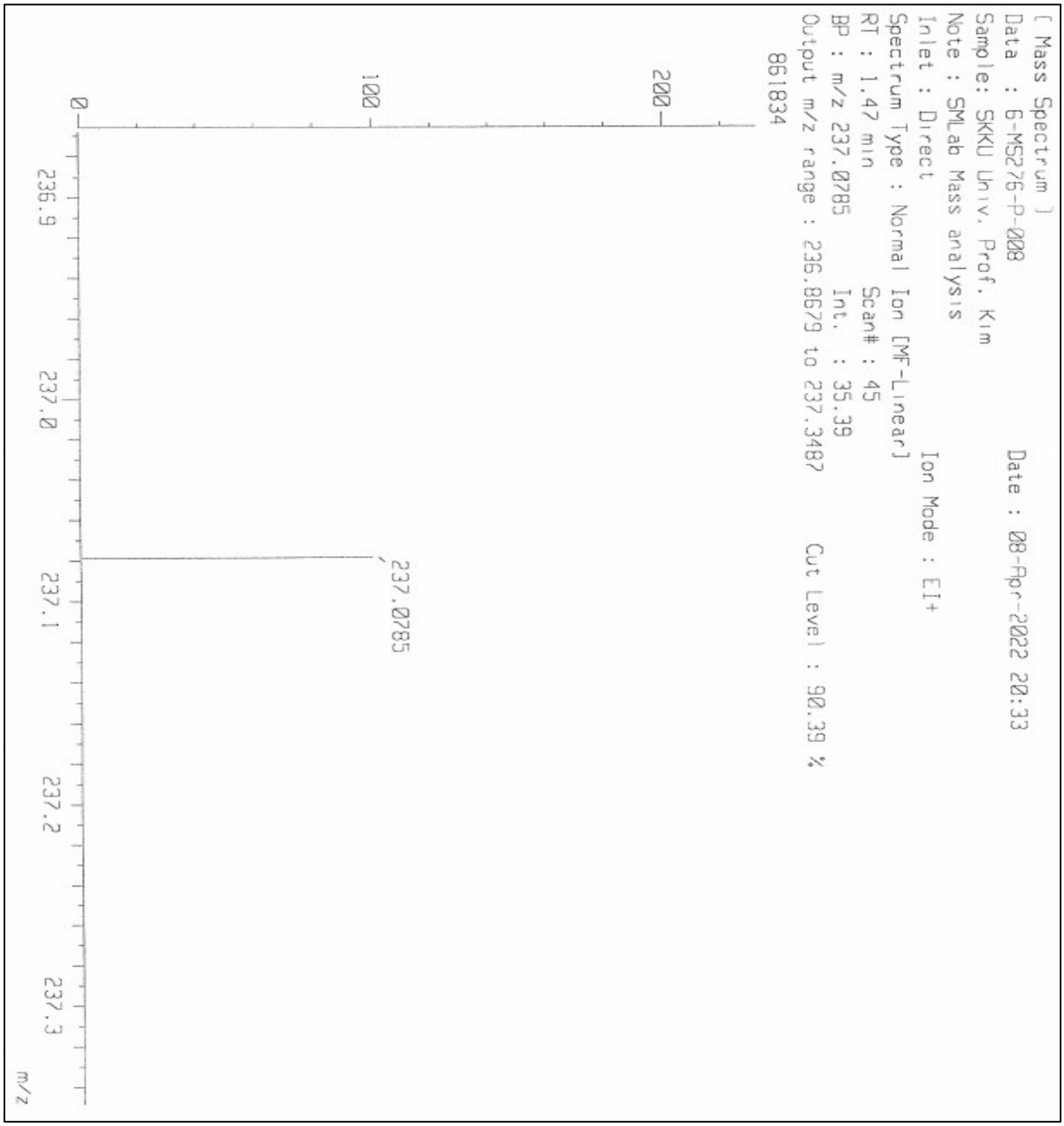
[Elemental Composition]
Data : 6-MS276-P-008
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.47 min
Elements : C 16/0, H 12/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 08-Apr-2022 20:33
Ion Mode : EI+
Scan#: 45

Page: 1

1j

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
237.0785	100.0	-1.9 / -0.4	11.0	C 15 H 11 O 2 N



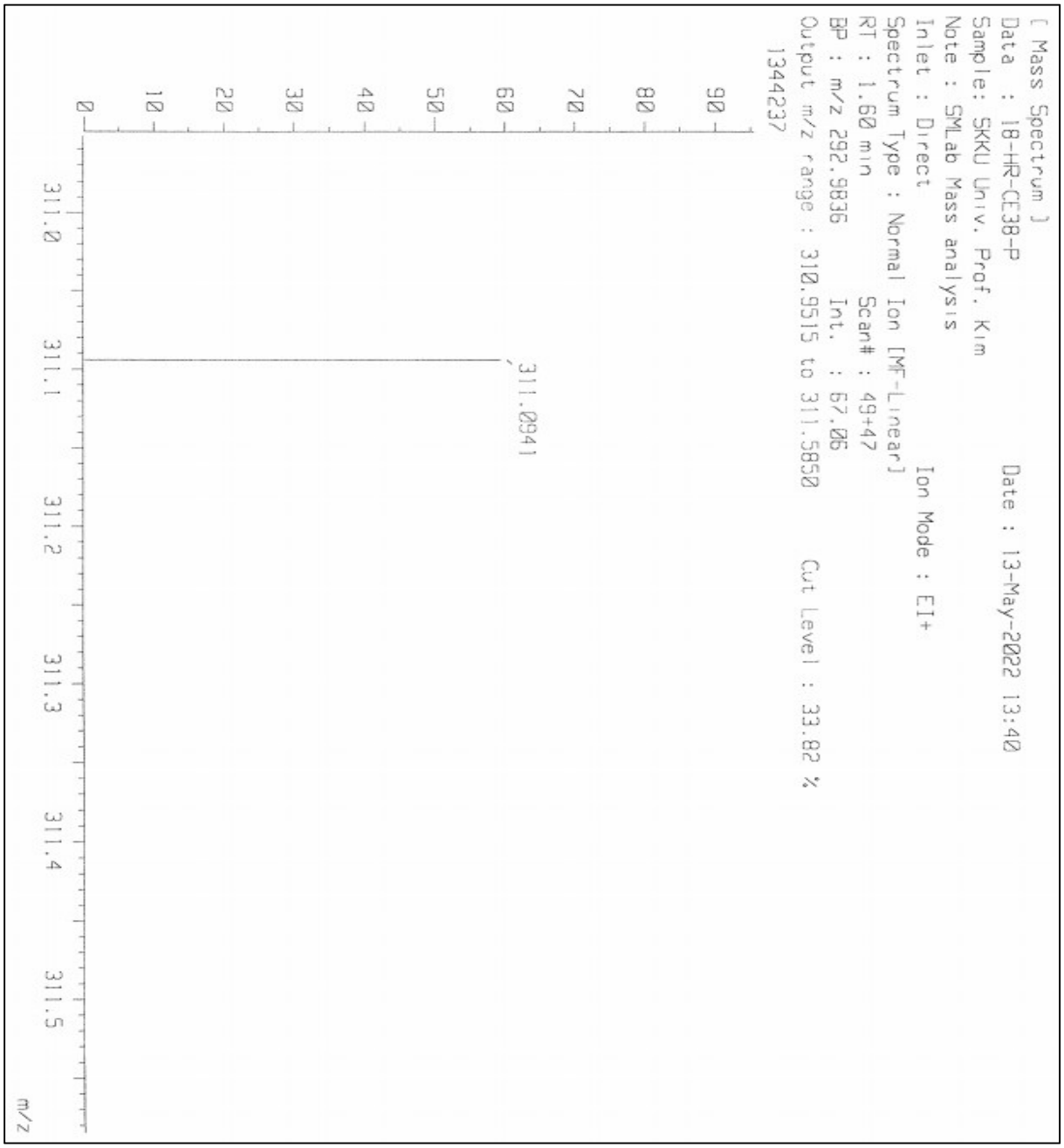
[Elemental Composition]
Page: 1

Data : 18-HR-CE38-P
Date : 13-May-2022 13:40

1k

Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
Ion Mode : EI+
RT : 1.60 min
Scan#: 49+47
Elements : C 22/0, H 15/0, O 2/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
311.0941	59.5	-1.6 / -0.5	16.0	C 21 H 13 O 2 N



[Elemental Composition]
Data : 15-MS268-P2-016
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 0.47 min
Elements : C 14/0, H 10/0, O 5/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

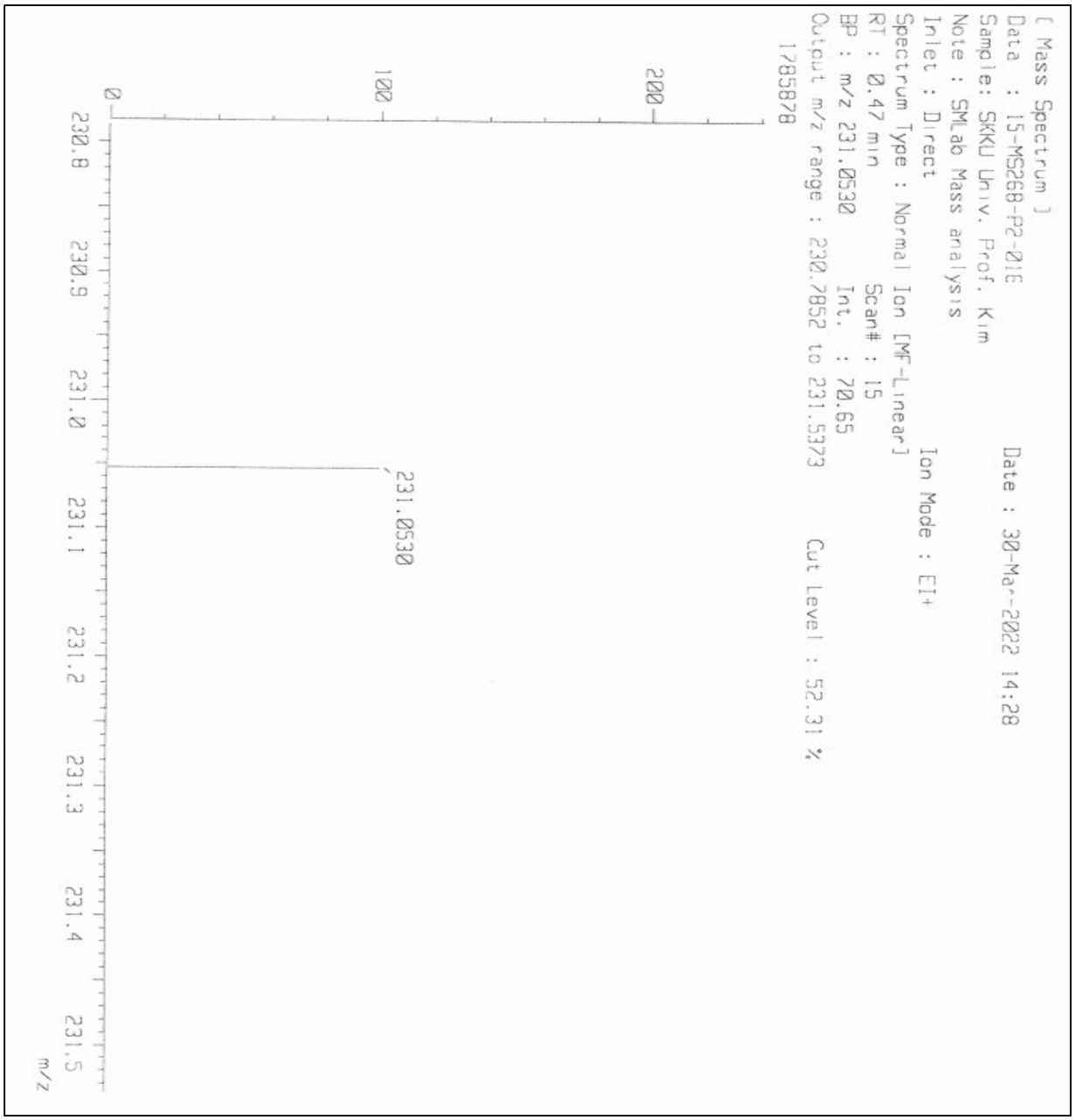
Date : 30-Mar-2022 14:28

Ion Mode : EI+
Scan#: 15

Page: 1

1l

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
231.0530	100.0	-0.5 / -0.1	9.0	C 12 H 9 O 4 N



[Elemental Composition] Page: 1

Data : 7-MS260-P-013 Date : 03-Aug-2022 18:54

1m

Sample: SKKU Univ. prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

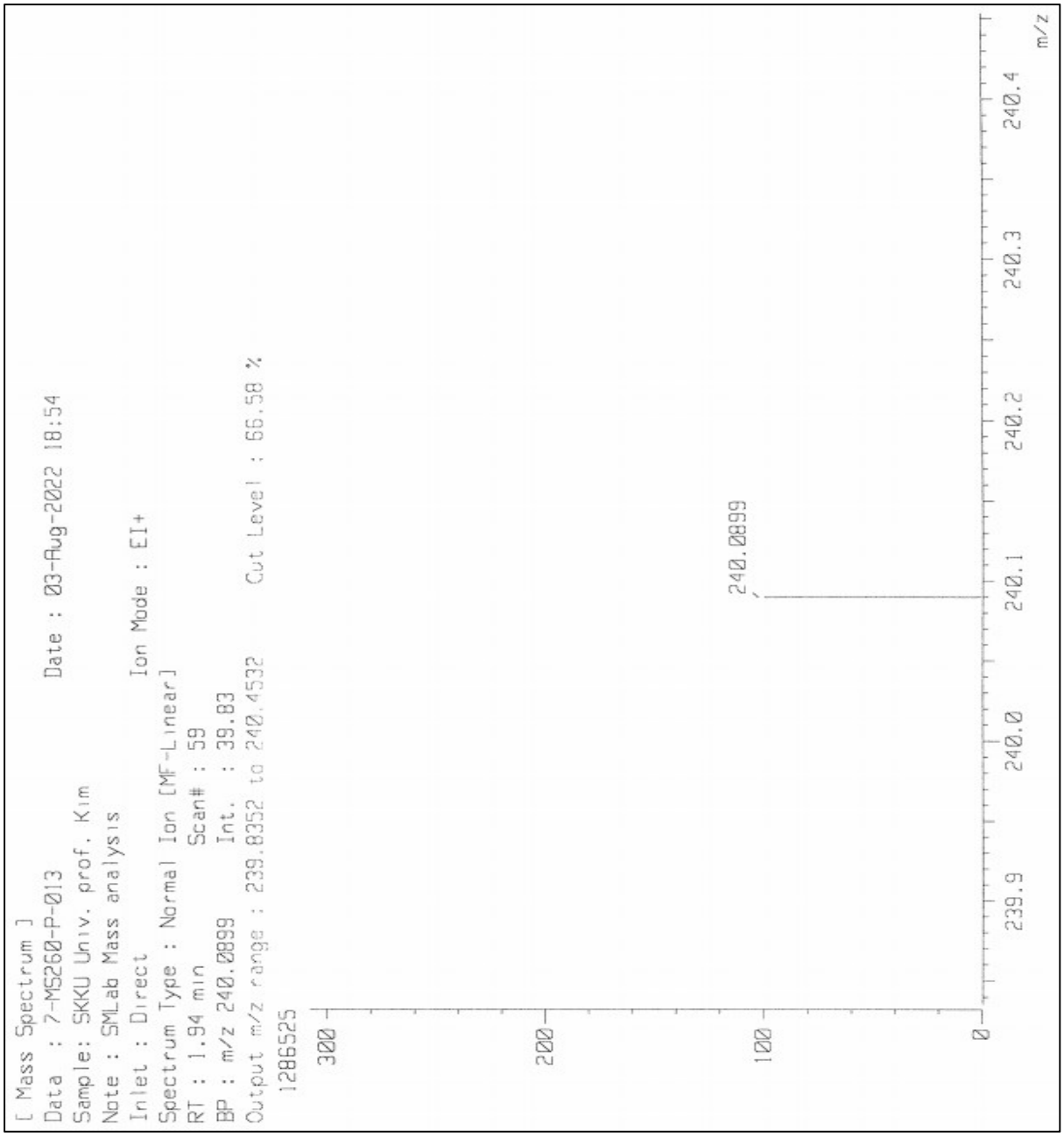
RT : 1.94 min Scan#: 59

Elements : C 16/0, H 15/0, O 2/0, N 2/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
240.0899	100.0	-0.1 / +0.0	10.0	C 14 H 12 O 2 N 2



[Elemental Composition] Page: 1

Data : 26-CE27-028 Date : 26-Mar-2022 13:03

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

1n

Inlet : Direct Ion Mode : EI+

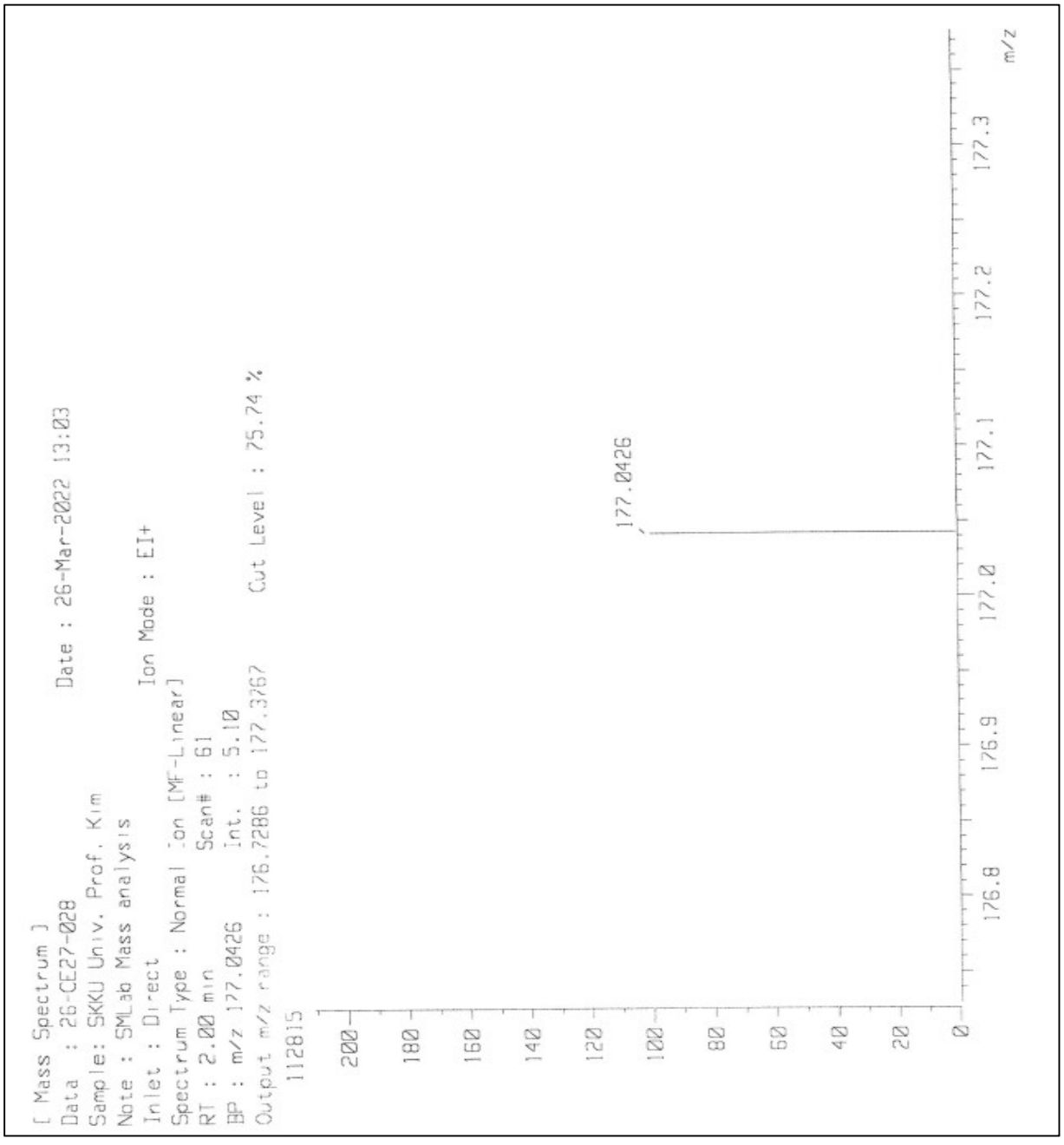
RT : 2.00 min Scan#: 61

Elements : C 10/0, H 10/0, O 3/0, N 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
177.0426	100.0	+0.1 / +0.0	7.0	C 9 H 7 O 3 N



[Elemental Composition]
Data : 15-CE87-P-013
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 0.97 min
Elements : C 10/0, H 10/0, O 2/0, N 1/0, S 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

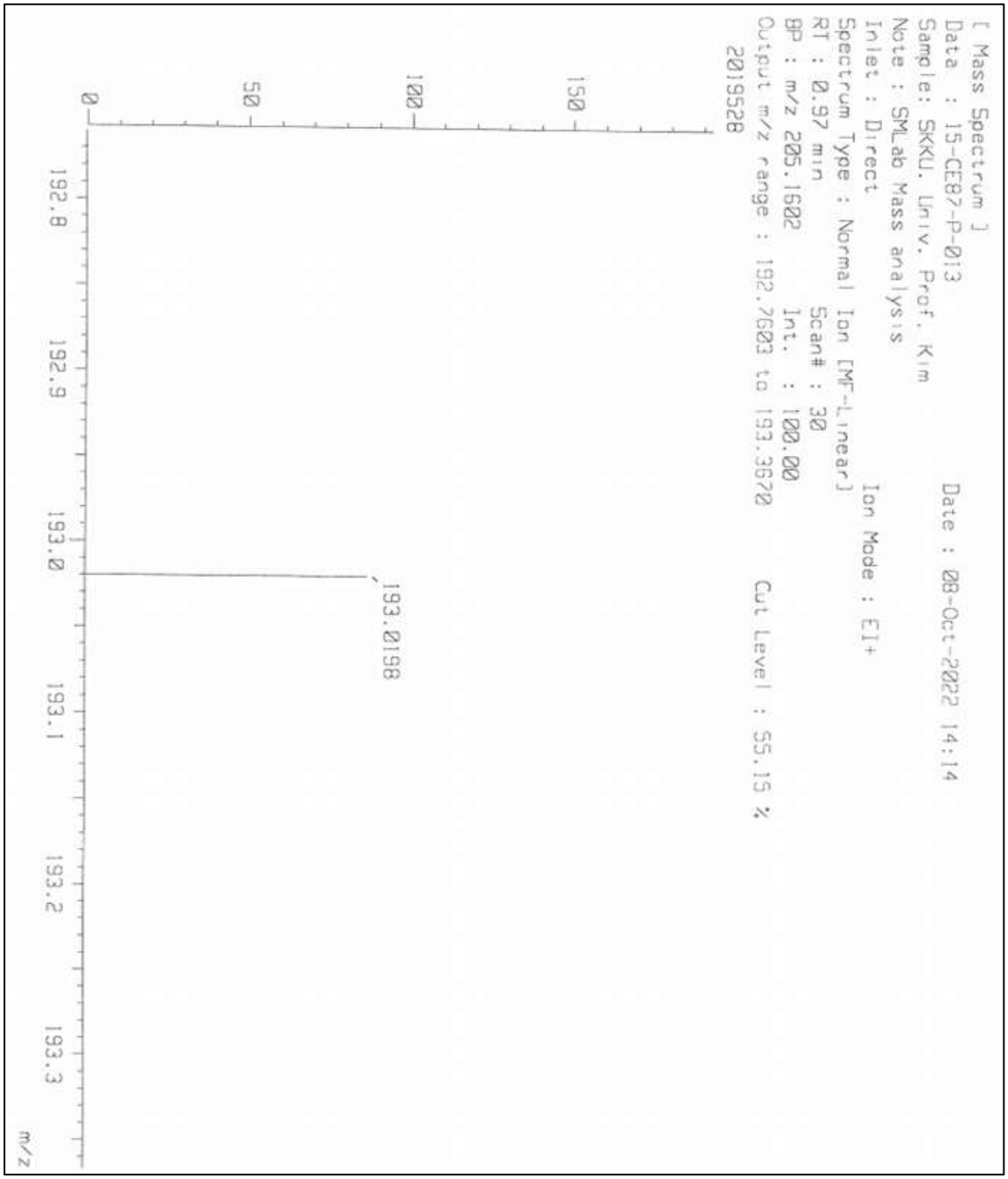
Date : 08-Oct-2022 14:14

Ion Mode : EI+
Scan#: 30

Page: 1

1o

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
193.0198	86.6	+0.4 / +0.1	8.0	C 9 H 7 O 2 N S



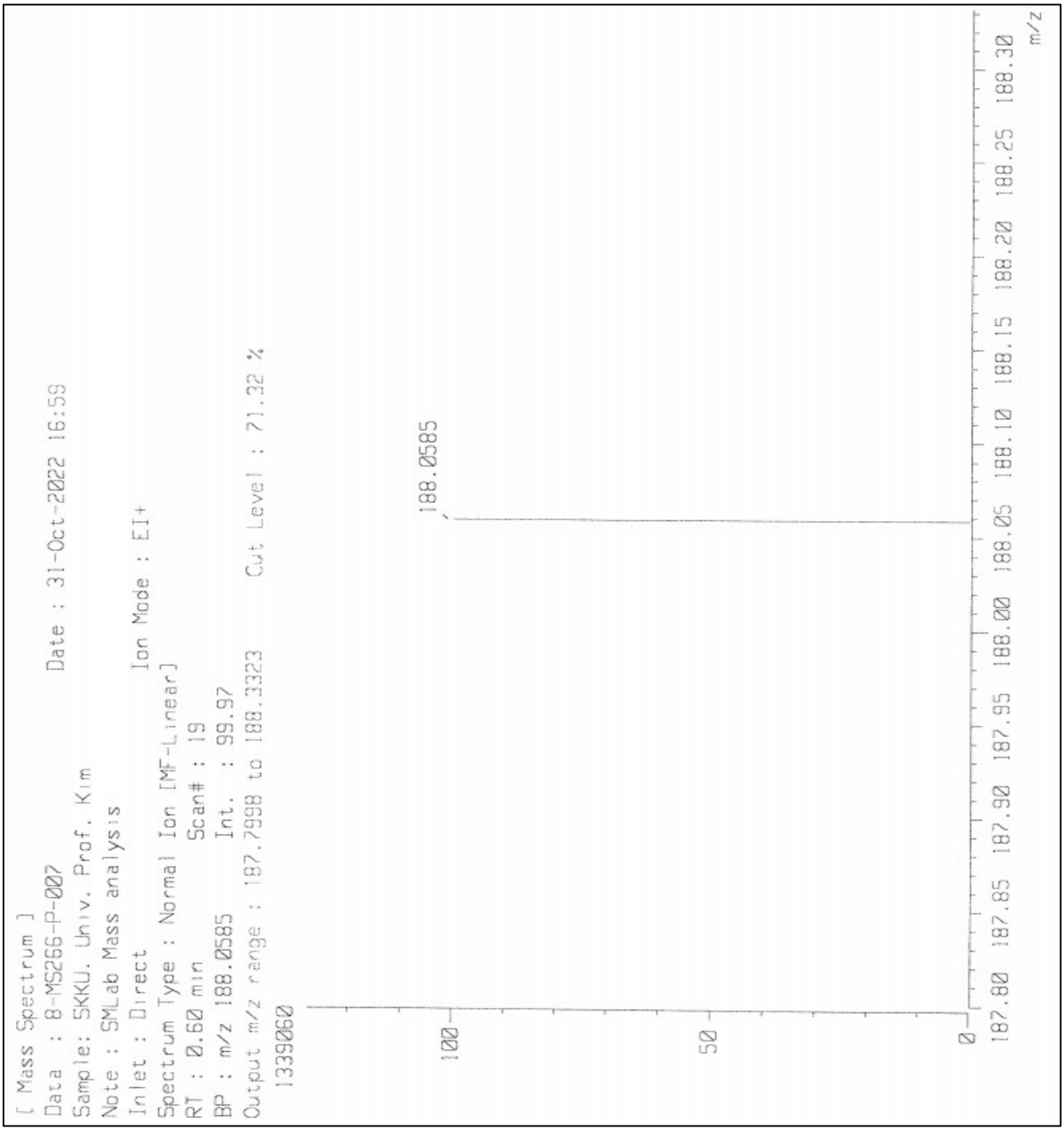
[Elemental Composition]
Data : 8-MS266-P-007
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 0.60 min
Elements : C 12/0, H 10/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

Date : 31-Oct-2022 16:59

Ion Mode : EI+
Scan#: 19

1p

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
188.0585	100.0	-0.2 / +0.0	8.0	C 10 H 8 O 2 N 2

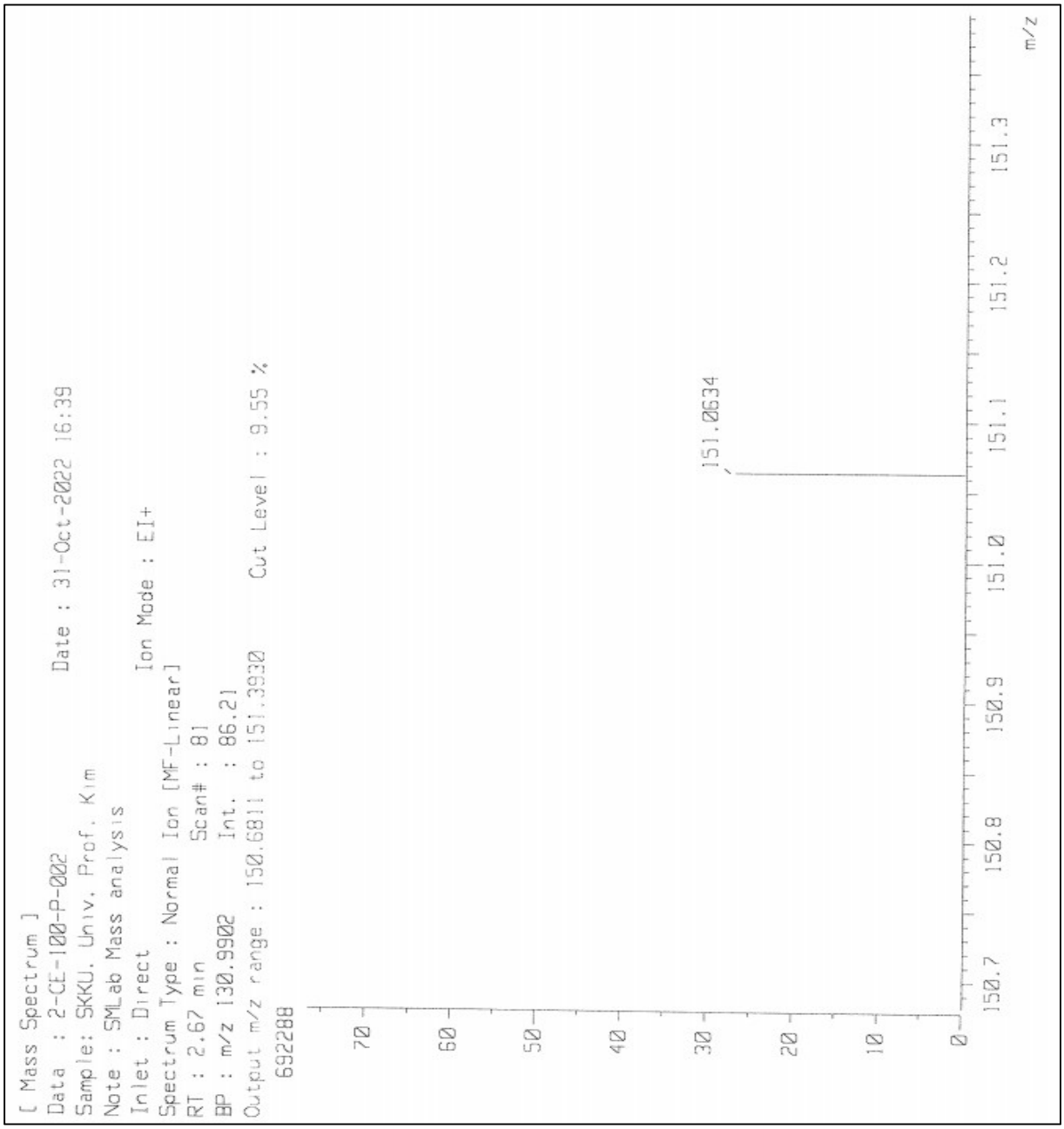


[Elemental Composition]
Data : 2-CE-100-P-002
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.67 min
Elements : C 10/0, H 10/0, O 3/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

Date : 31-Oct-2022 16:39
Ion Mode : EI+
Scan#: 81

1q

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
151.0634	27.0	+0.6 / +0.1	5.0	C 8 H 9 O 2 N



[Elemental Composition]
Data : 6-YV701-P-006
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.80 min
Elements : C 12/0, H 14/0, O 3/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

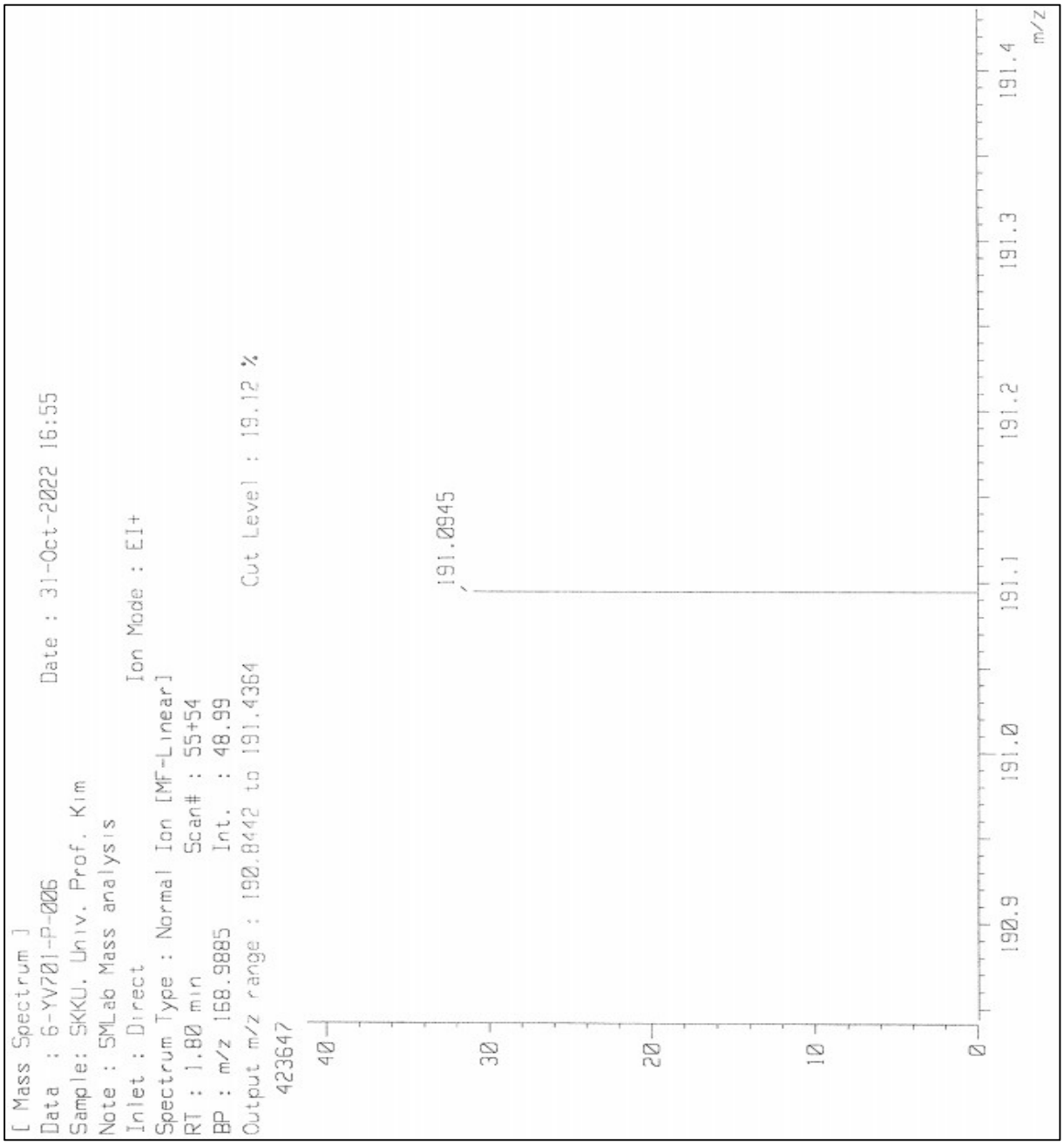
Date : 31-Oct-2022 16:55

Ion Mode : EI+
Scan#: 55+54

Page: 1

1r

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
191.0945	31.0	-0.7 / -0.1	6.0	C 11 H 13 O 2 N



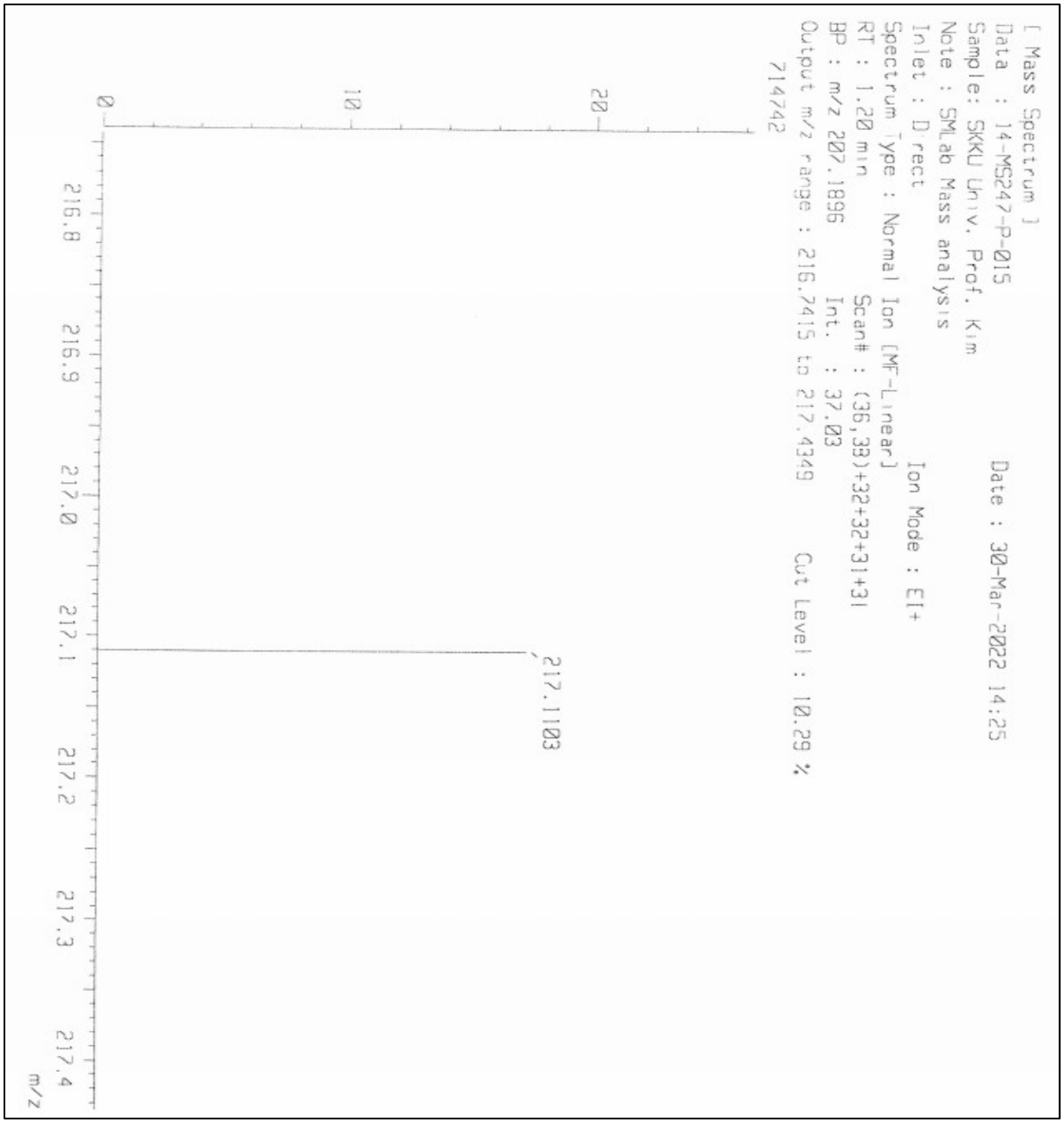
[Elemental Composition]
Data : 14-MS247-P-015
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.20 min
Elements : C 15/0, H 15/0, O 2/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 30-Mar-2022 14:25
Ion Mode : EI+
Scan#: (36,38)+32+32+31+31

Page: 1

1s

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
217.1103	17.3	+0.1 / +0.0	7.0	C 13 H 15 O 2 N



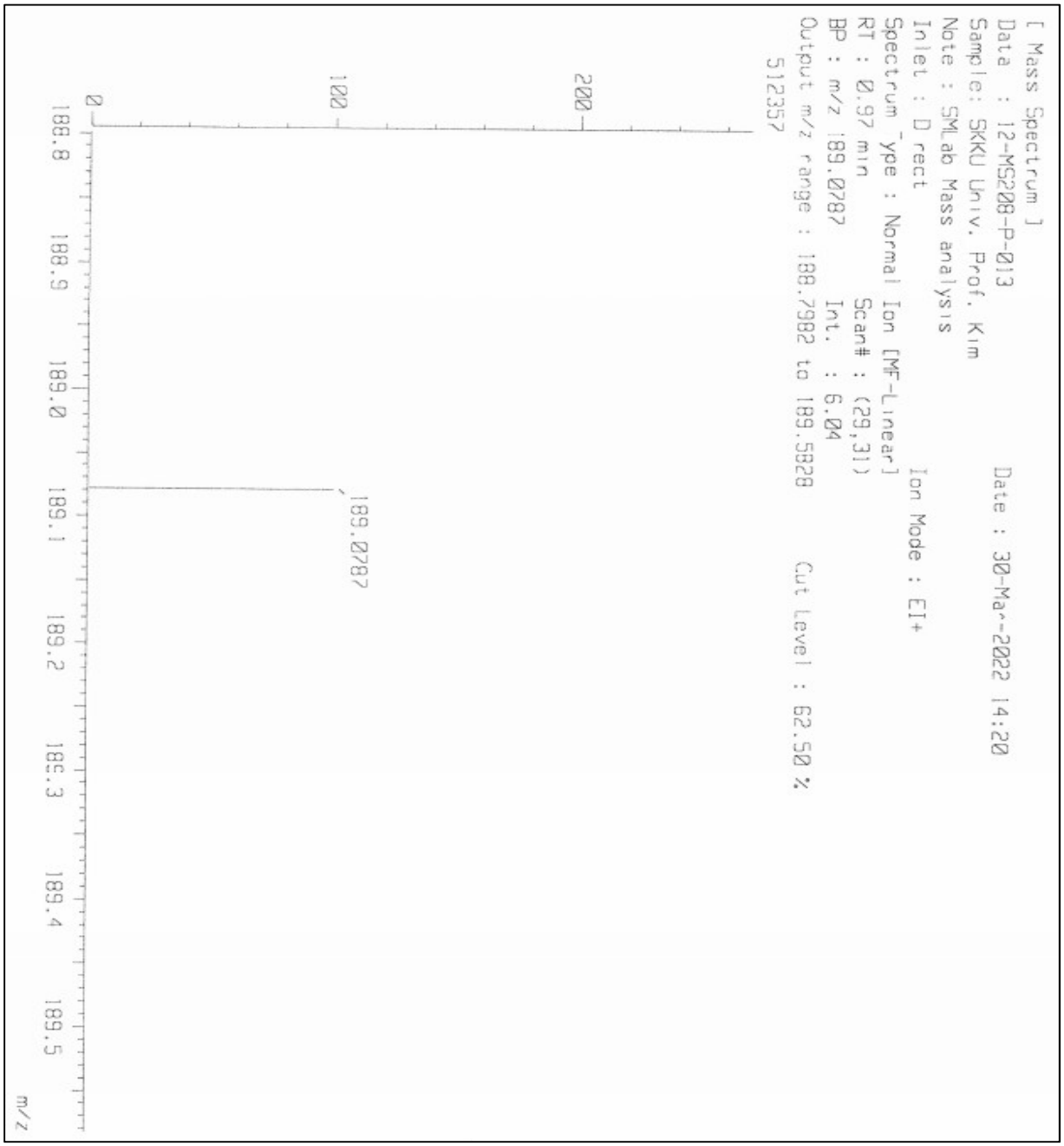
[Elemental Composition]
Data : 12-MS208-P-013
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 0.97 min
Elements : C 12/0, H 12/0, O 2/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 30-Mar-2022 14:20
Ion Mode : EI+
Scan#: (29,31)

Page: 1

1t

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
189.0787	100.0	-1.4 / -0.3	7.0	C 11 H 11 O 2 N



[Elemental Composition] Page: 1

Data : FAB-SG-93-P-003 Date : 25-Mar-2022 16:08

Sample: SKKU Univ. Prof. Kim

Note : SM Lab Research Center

3a

Inlet : Reserv. Ion Mode : FAB+

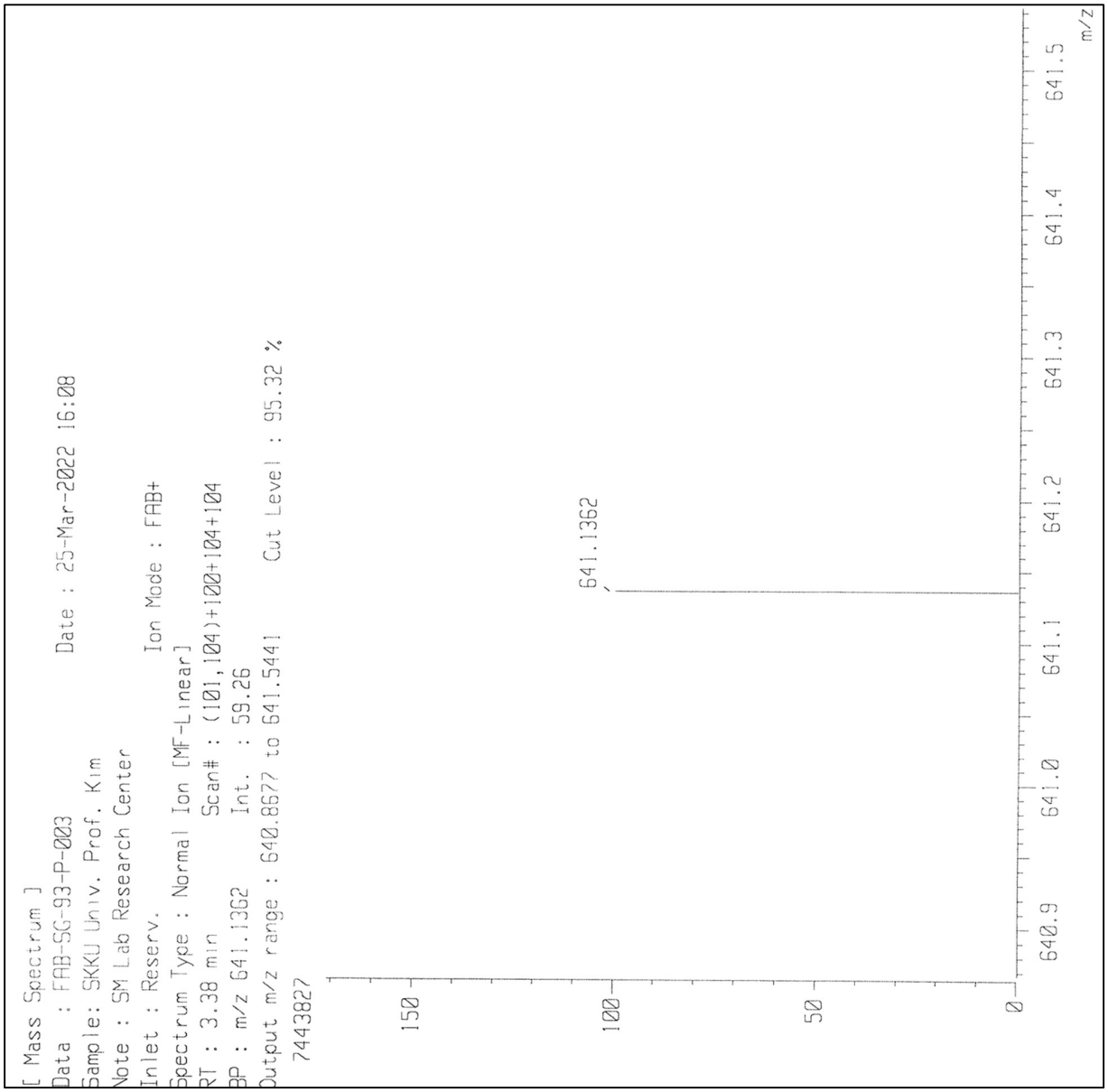
RT : 3.38 min Scan#: (101,104)+100+104+104

Elements : C 25/0, H 25/0, O 5/0, N 1/0, F 3/0, Ir 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 80.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
641.1362	100.0	-0.4 / -0.3	12.0	C 24 H 25 O 4 N F 3 Ir

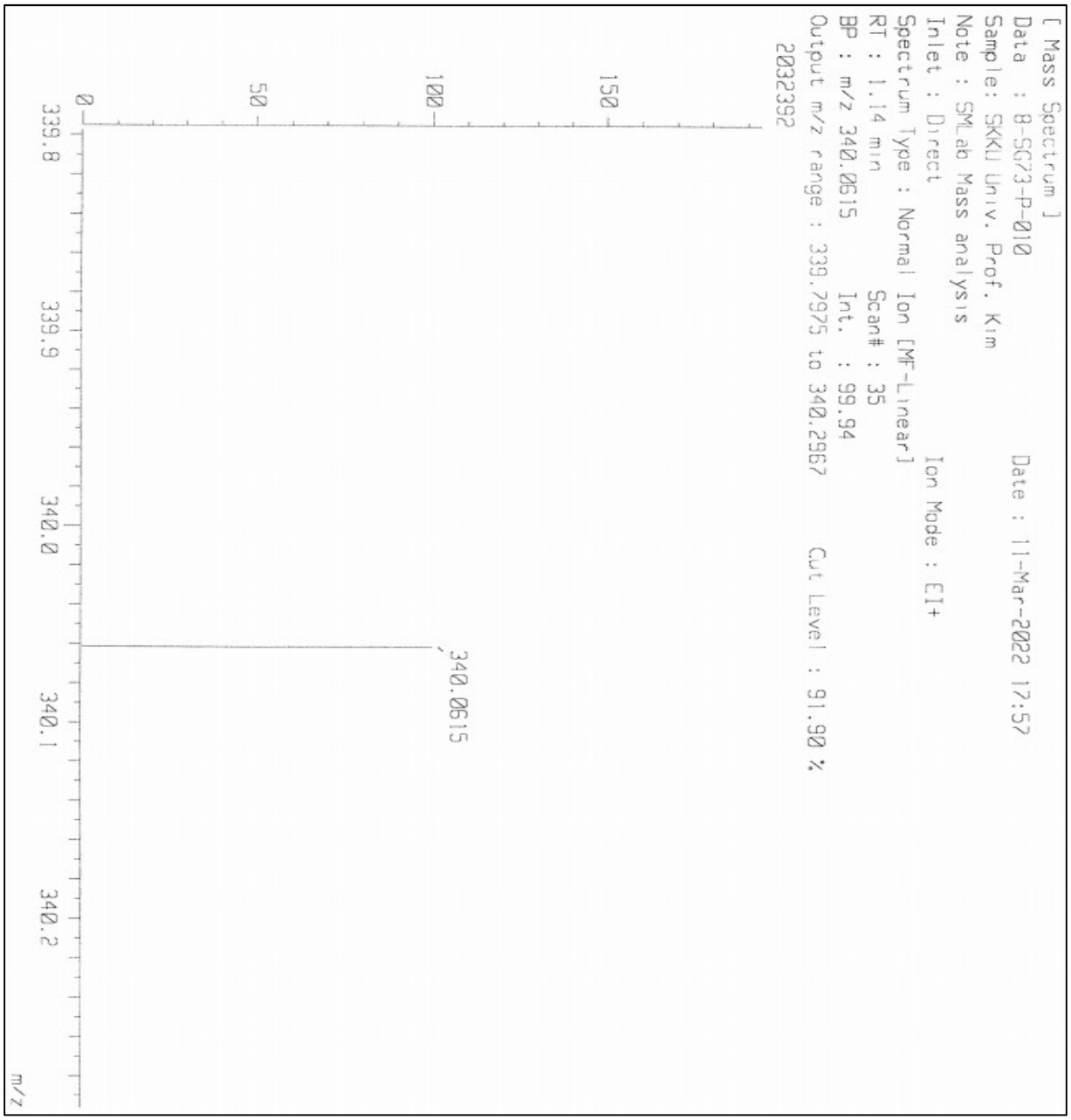


[Elemental Composition]
Data : 8-SG73-P-010
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.14 min
Elements : C 18/0, H 15/0, O 3/0, N 3/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 11-Mar-2022 17:57
Ion Mode : EI+
Scan#: 35

3b

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
340.0615	100.0	+0.1 / +0.0	13.0	C 18 H 13 O 3 N 2 Cl



[Elemental Composition] Page: 1

Data : 9-SG74-P-011 Date : 11-Mar-2022 18:00

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

RT : 0.87 min Scan#: 27

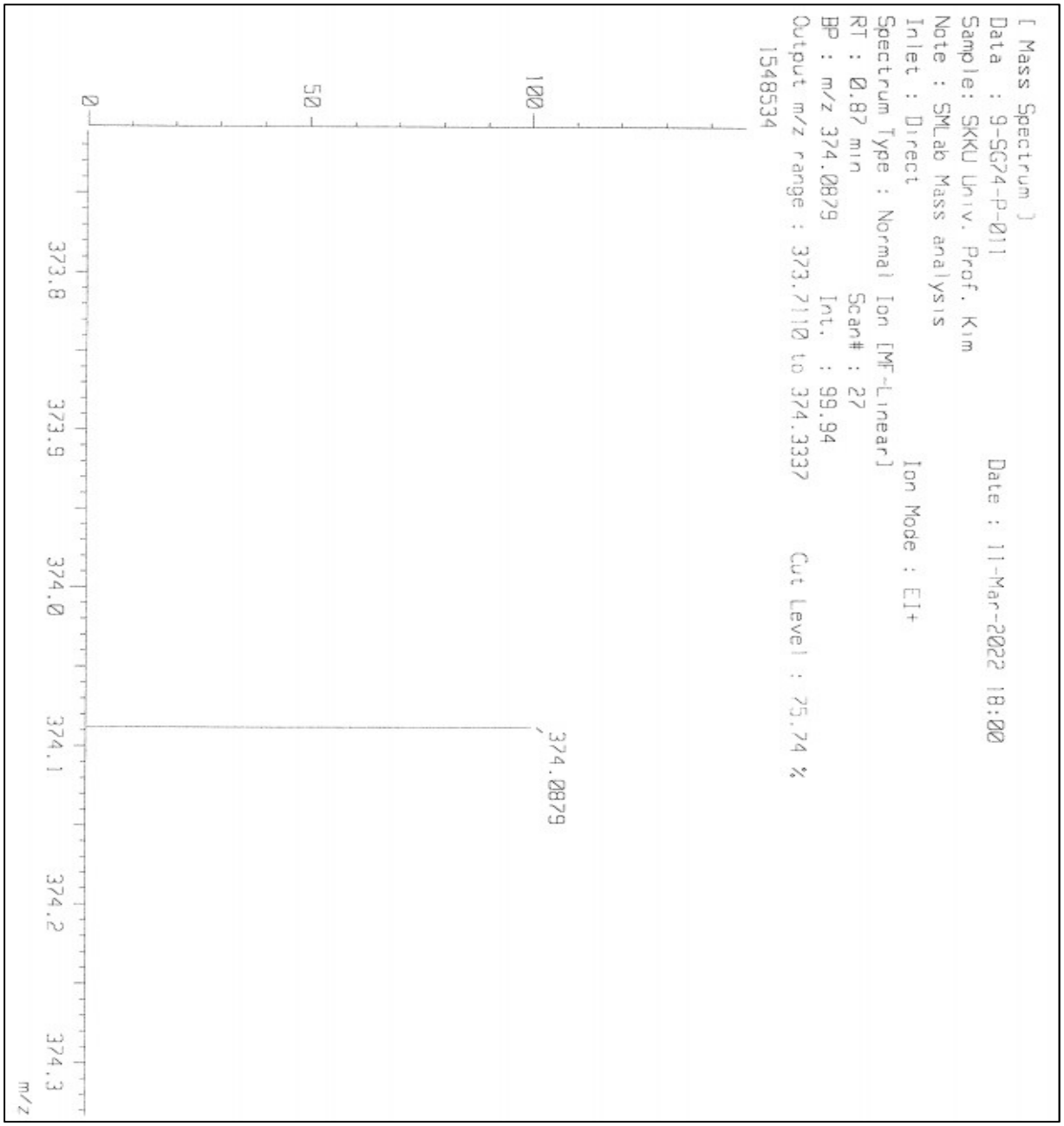
Elements : C 20/0, H 15/0, O 3/0, N 3/0, F 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
374.0879	100.0	+0.3 / +0.1	13.0	C 19 H 13 O 3 N 2 F 3

3c



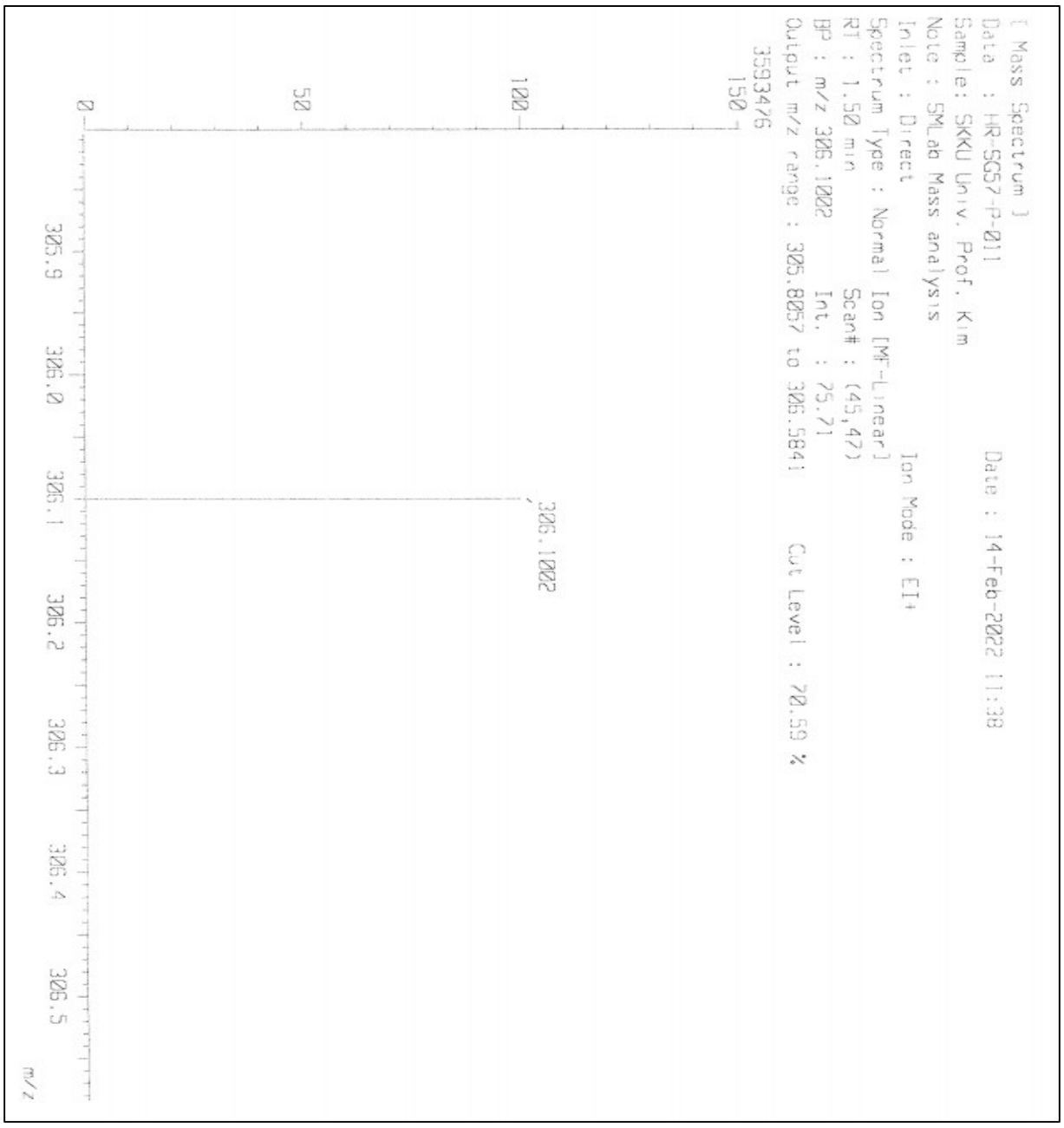
[Elemental Composition]
Data : HR-SG57-P-011
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.50 min
Elements : C 20/0, H 15/0, O 4/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 14-Feb-2022 11:38
Ion Mode : EI+
Scan#: (45,47)

Page: 1

3d

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
306.1002	100.0	-0.7 / -0.2	13.0	C 18 H 14 O 3 N 2



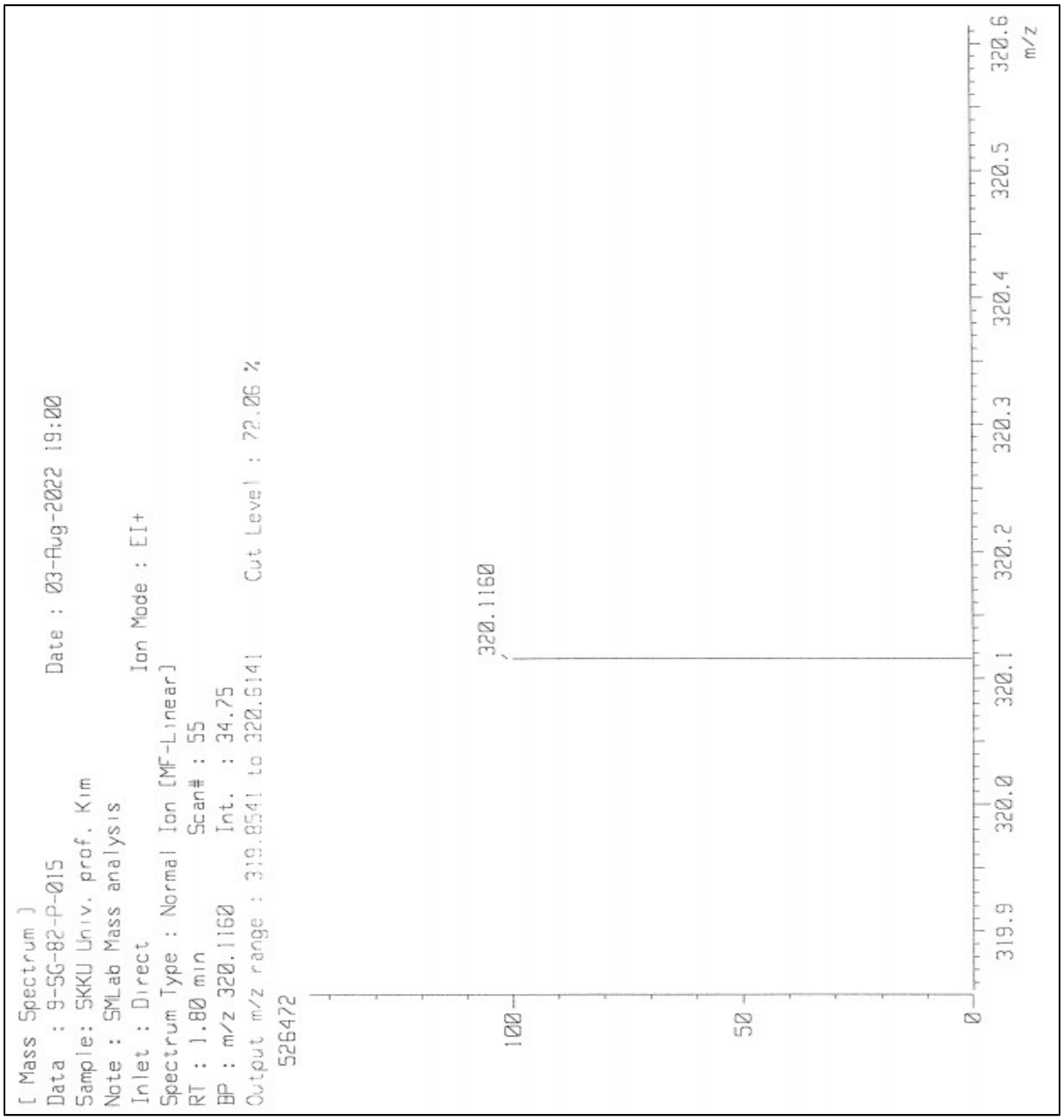
[Elemental Composition]
Data : 9-SG-82-P-015
Sample: SKKU Univ. prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.80 min
Elements : C 20/0, H 17/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 03-Aug-2022 19:00

Ion Mode : EI+
Scan#: 55

3e

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
320.1160	100.0	-0.3 / -0.1	13.0	C 19 H 16 O 3 N 2

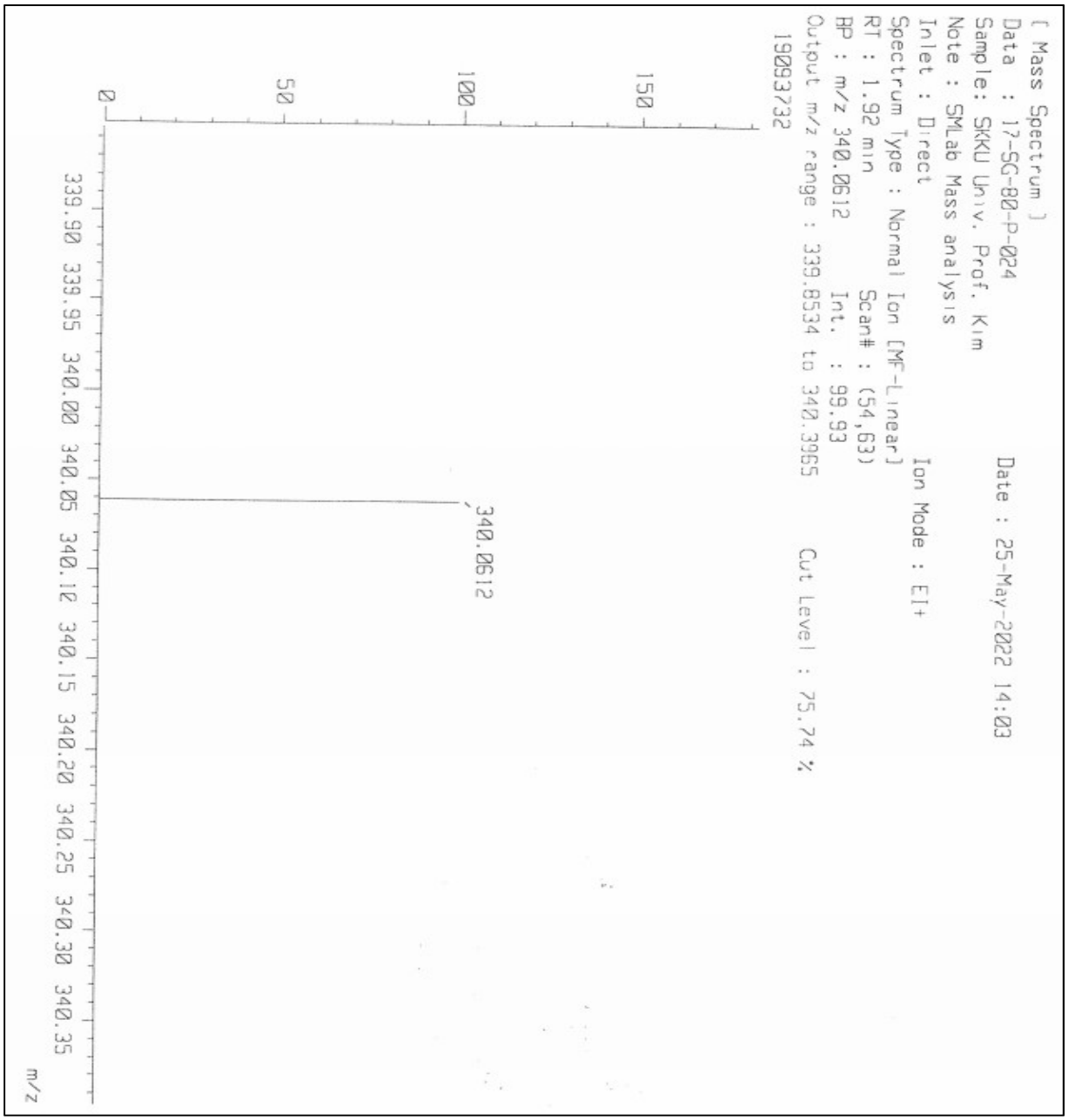


[Elemental Composition]
Data : 17-SG-80-P-024
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.92 min
Elements : C 20/0, H 15/0, O 3/0, N 2/0, Cl 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 25-May-2022 14:03
Ion Mode : EI+
Scan#: (54,63)

3f

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
340.0612	100.0	-0.8 / -0.3	13.0	C 18 H 13 O 3 N 2 Cl

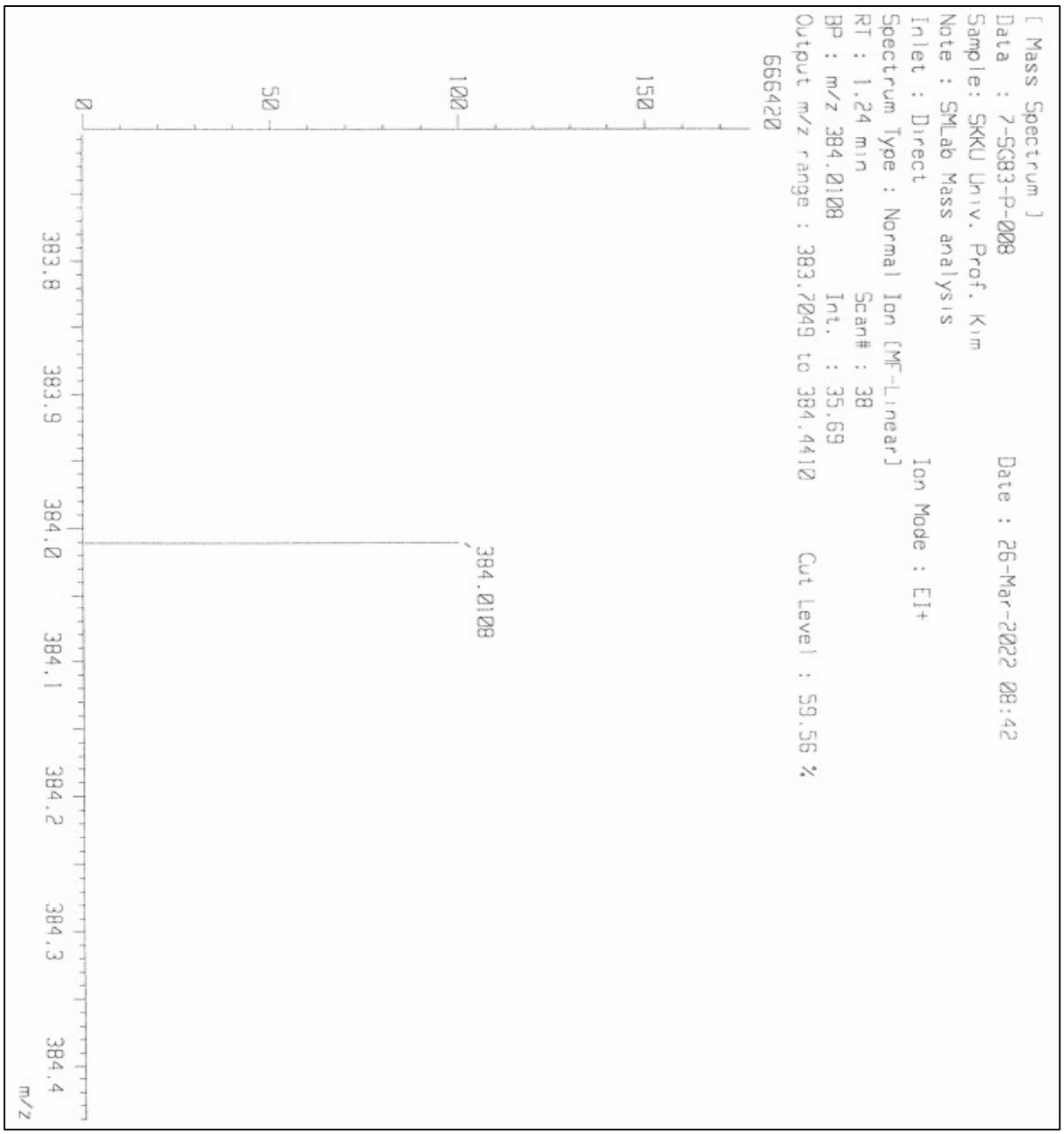


[Elemental Composition]
Data : 7-SG83-P-008
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.24 min
Elements : C 20/0, H 16/0, O 3/0, N 3/0, Br 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 26-Mar-2022 08:42
Ion Mode : EI+
Scan#: 38

3g

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
384.0108	100.0	-0.4 / -0.2	13.0	C 18 H 13 O 3 N 2 Br

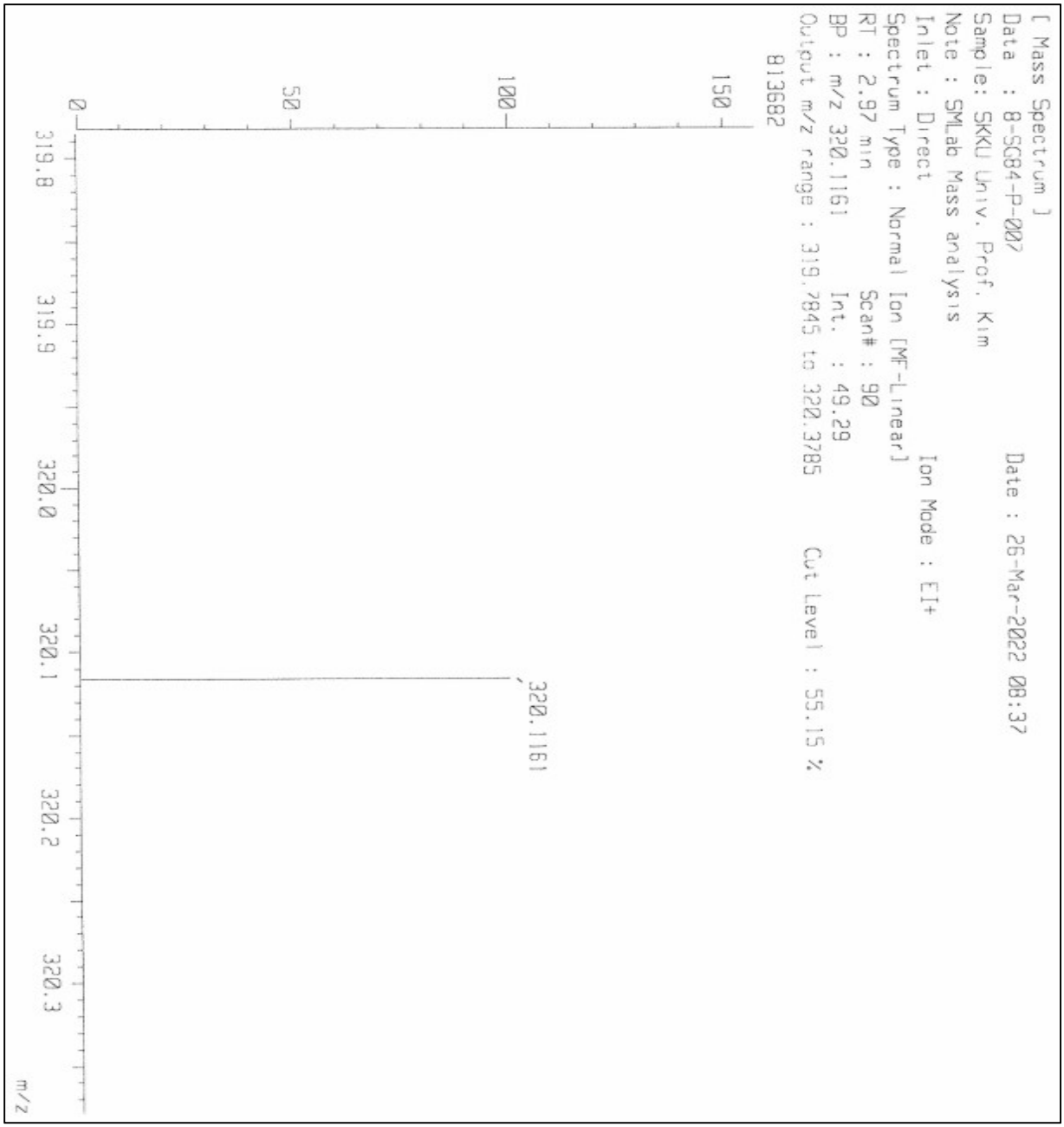


[Elemental Composition]
Data : 8-SG84-P-007
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.97 min
Elements : C 20/0, H 16/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 26-Mar-2022 08:37
Ion Mode : EI+
Scan#: 90

3h

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
320.1161	100.0	+0.2 / +0.1	13.0	C 19 H 16 O 3 N 2



[Elemental Composition] Page: 1

Data : 9-SG85-P-009 Date : 26-Mar-2022 08:44

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

RT : 0.97 min Scan#: (26,34)

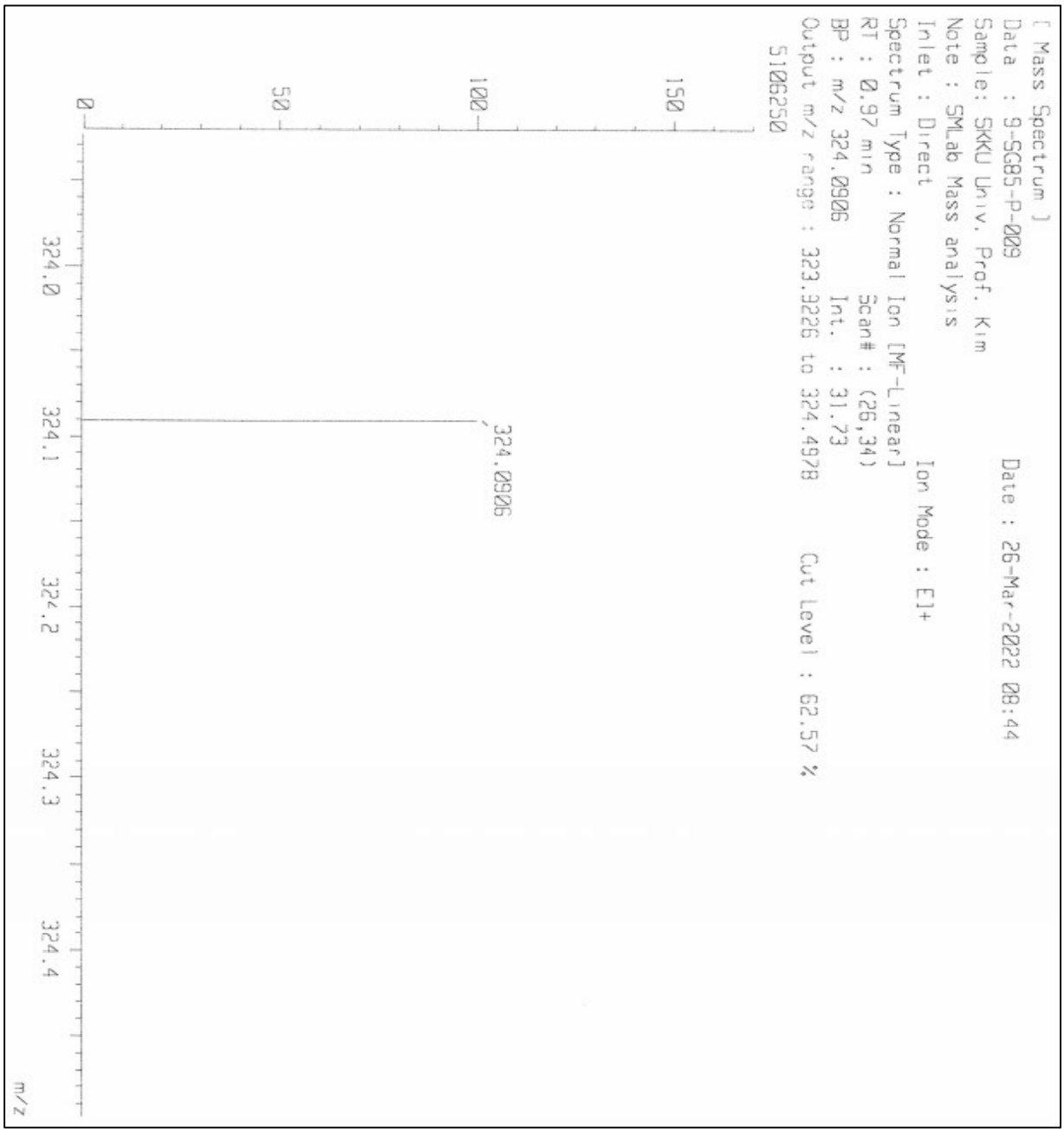
Elements : C 20/0, H 16/0, O 3/0, N 3/0, F 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
324.0906	100.0	-1.3 / -0.4	13.0	C 18 H 13 O 3 N 2 F

3i



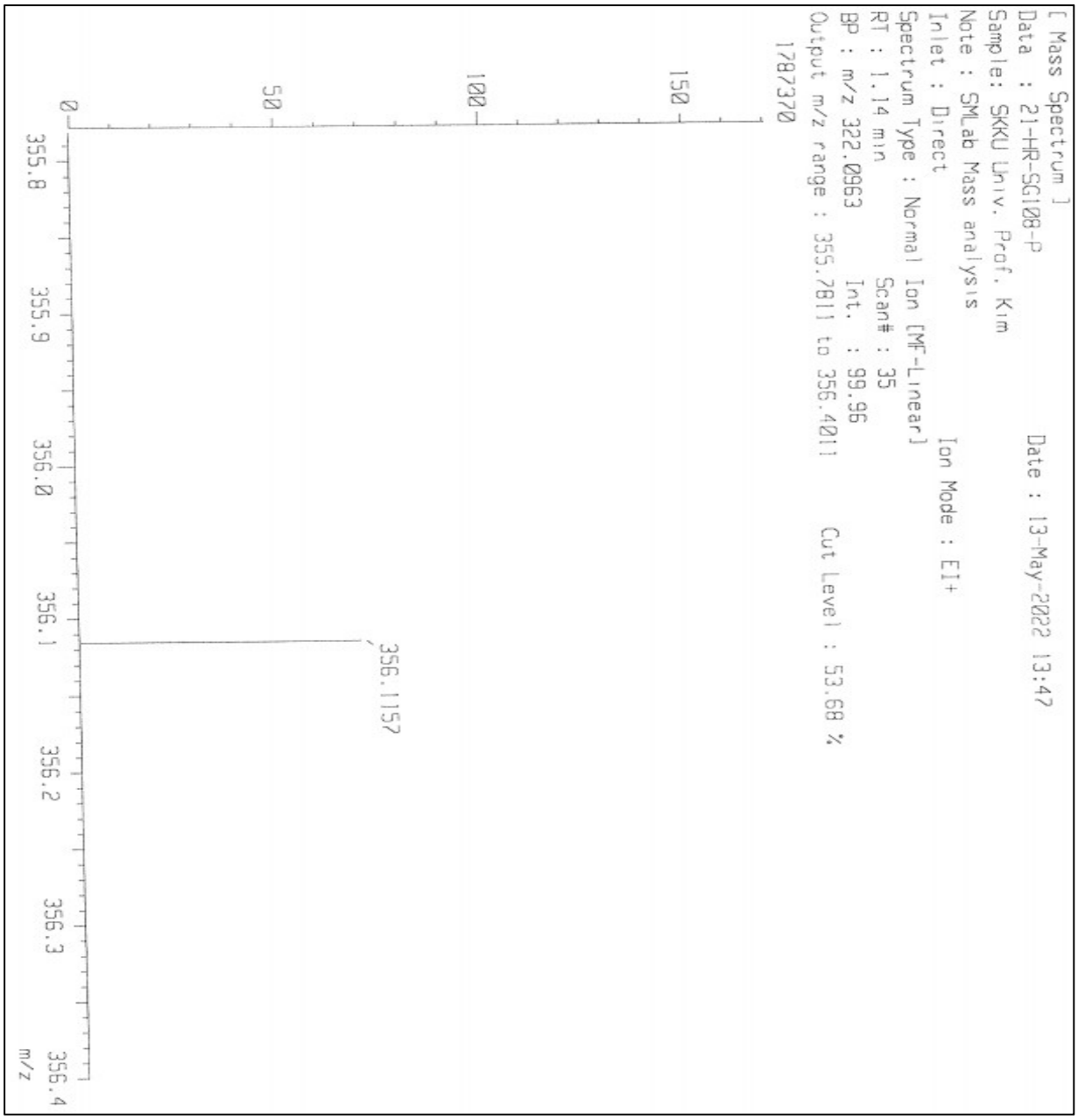
3j

[Elemental Composition]
Data : 21-HR-SG108-P
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.14 min
Elements : C 25/0, H 18/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 13-May-2022 13:47

Ion Mode : EI+
Scan#: 35

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
356.1157	68.9	-1.2 / -0.4	16.0	C 22 H 16 O 3 N 2



[Elemental Composition]
Data : 19-HE-CE39-P
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.07 min
Elements : C 30/0, H 20/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

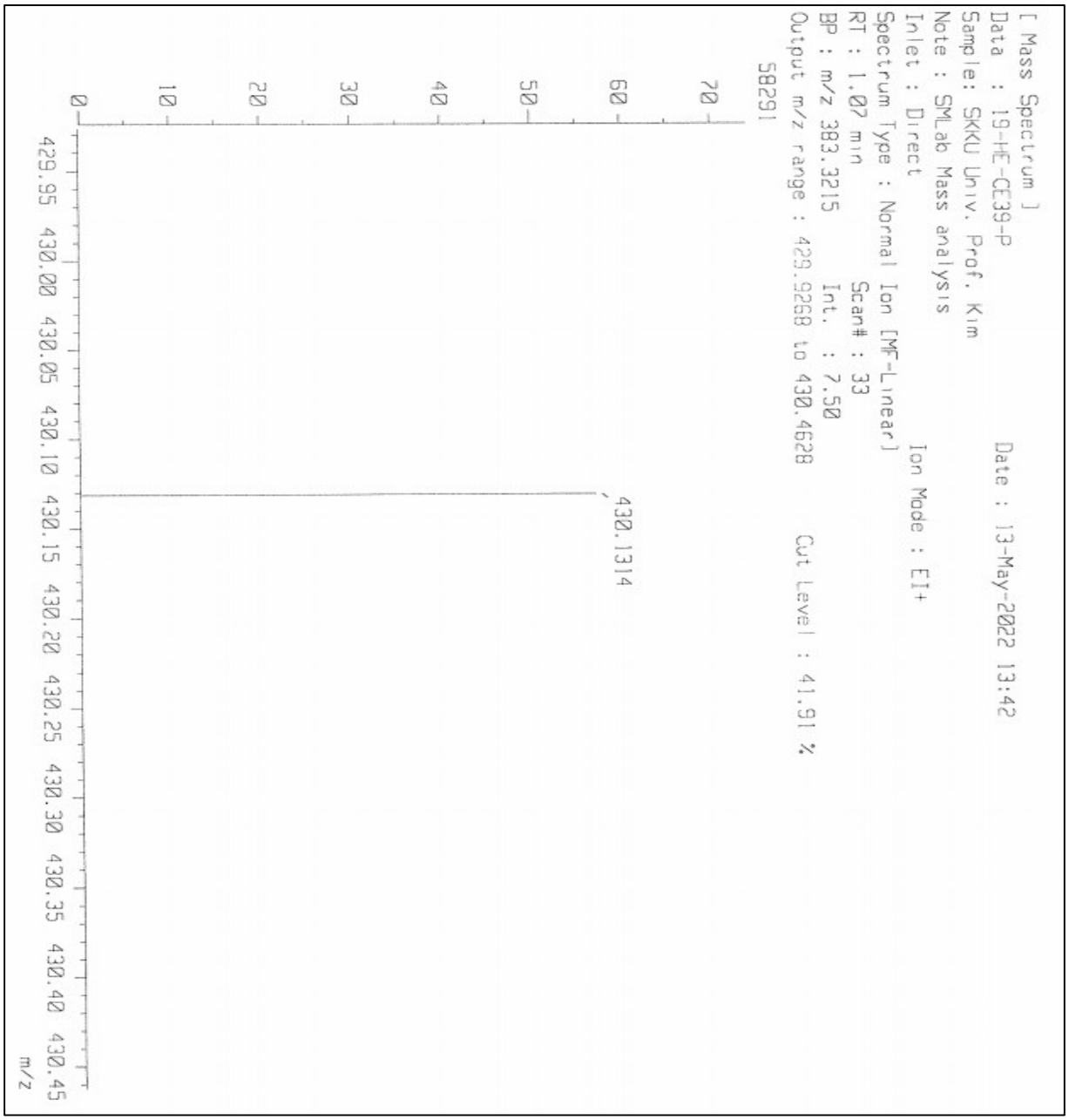
Date : 13-May-2022 13:42

Ion Mode : EI+
Scan#: 33

Page: 1

3k

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
430.1314	57.2	-0.8 / -0.4	21.0	C 28 H 18 O 3 N 2



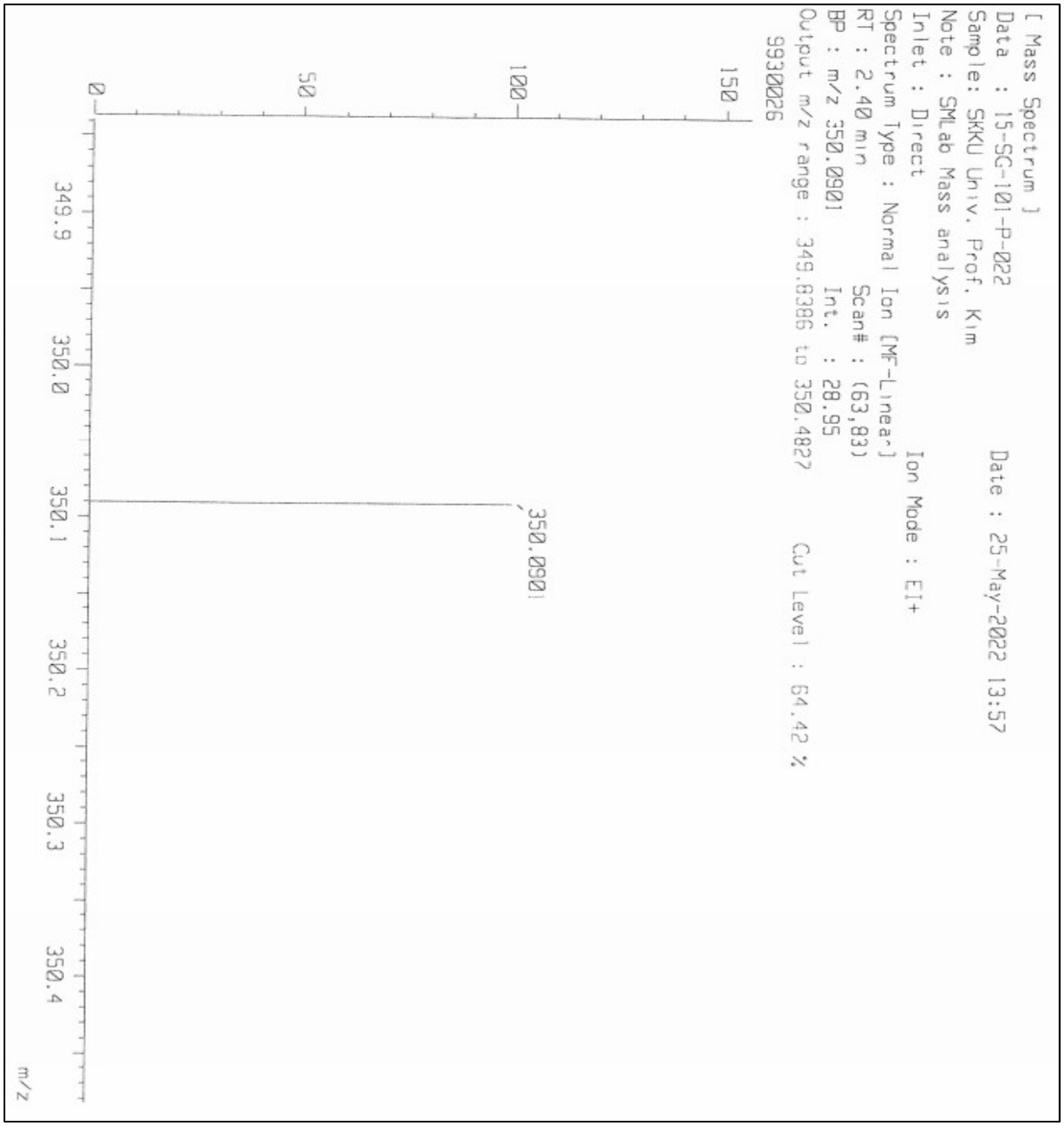
[Elemental Composition]
Data : 15-SG-101-P-022
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.40 min
Elements : C 20/0, H 15/0, O 5/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 25-May-2022 13:57
Ion Mode : EI+
Scan#: (63,83)

Page: 1

3l

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
350.0901	100.0	-0.4 / -0.2	14.0	C 19 H 14 O 5 N 2



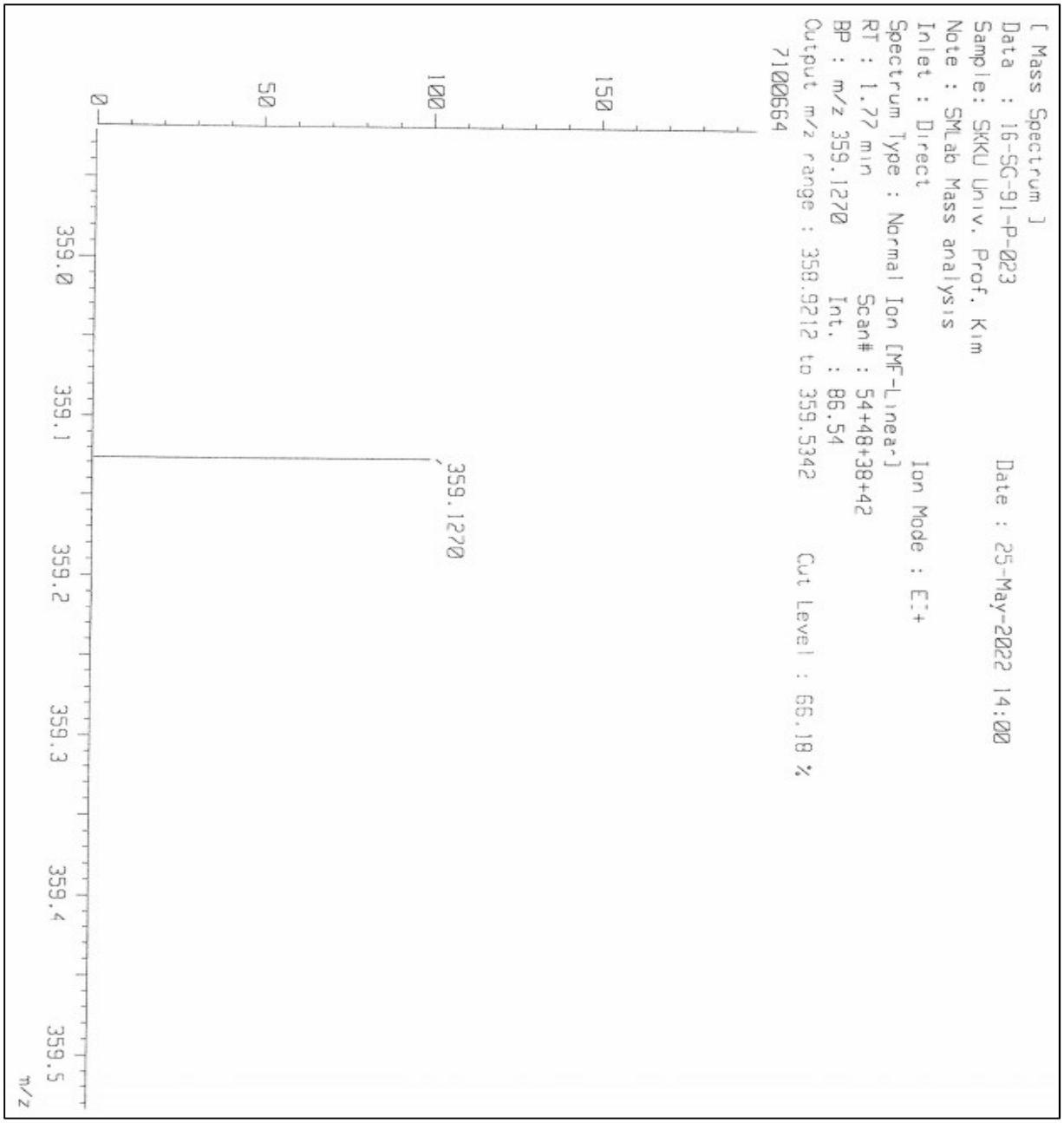
[Elemental Composition]
Data : 16-SG-91-P-023
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.77 min
Elements : C 23/0, H 20/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 25-May-2022 14:00
Ion Mode : EI+
Scan#: 54+48+38+42

Page: 1

3m

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
359.1270	100.0	+0.1 / +0.0	15.0	C 21 H 17 O 3 N 3



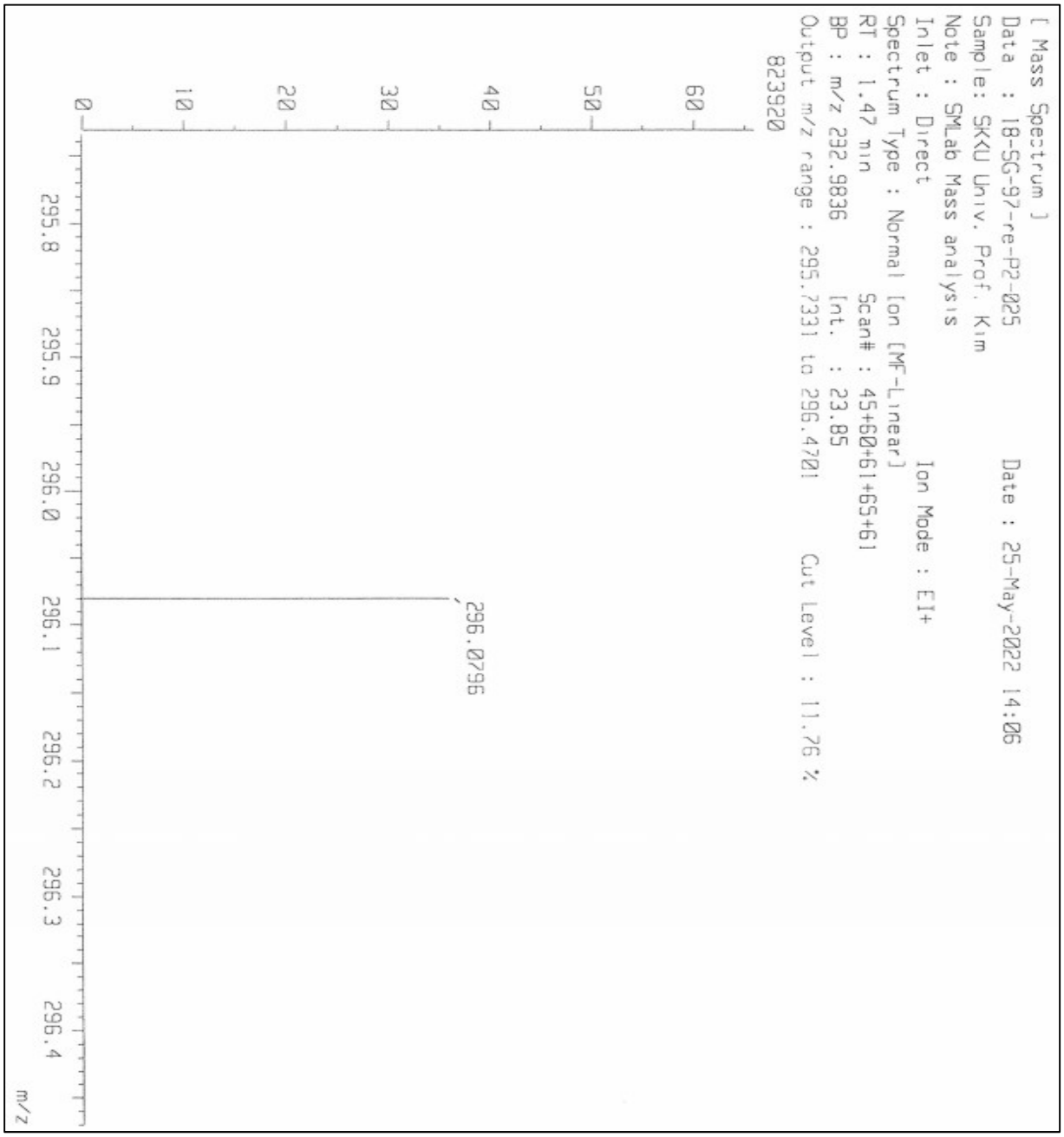
[Elemental Composition]
Data : 18-SG-97-re-P2-025
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.47 min
Elements : C 18/0, H 15/0, O 4/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 25-May-2022 14:06
Ion Mode : EI+
Scan#: 45+60+61+65+61

Page: 1

3n

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
296.0796	35.9	-0.4 / -0.1	12.0	C 16 H 12 O 4 N 2



[Elemental Composition] Page: 1

Data : 18-CE90-P-016 Date : 08-Oct-2022 14:25

Sample: SKKU. Univ. Prof. Kim

Note : SMLab Mass analysis

3o

Inlet : Direct Ion Mode : EI+

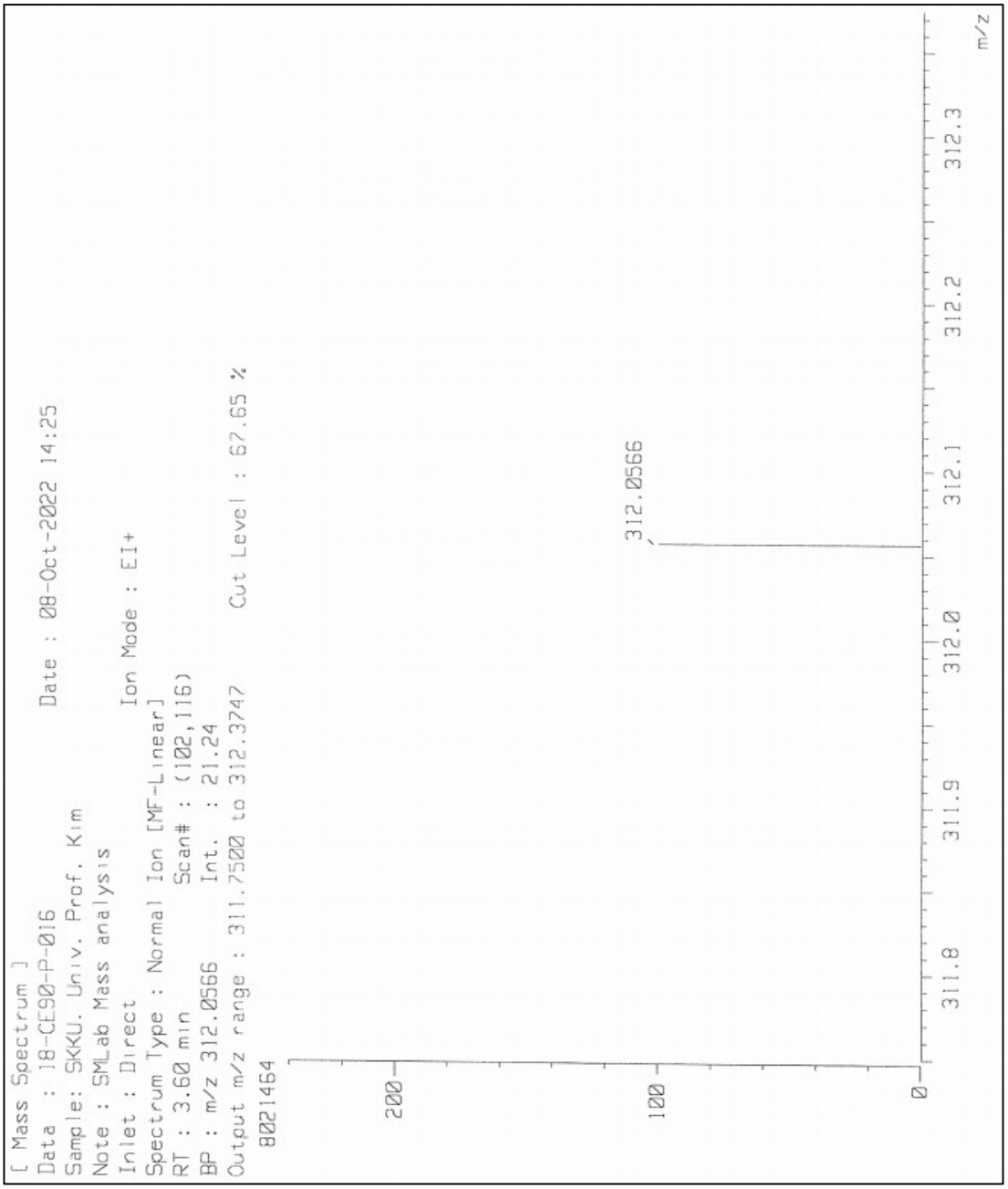
RT : 3.60 min Scan#: (102,116)

Elements : C 17/0, H 14/0, O 3/0, N 2/0, S 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
312.0566	100.0	-0.9 / -0.3	13.0	C 16 H 12 O 3 N 2 S

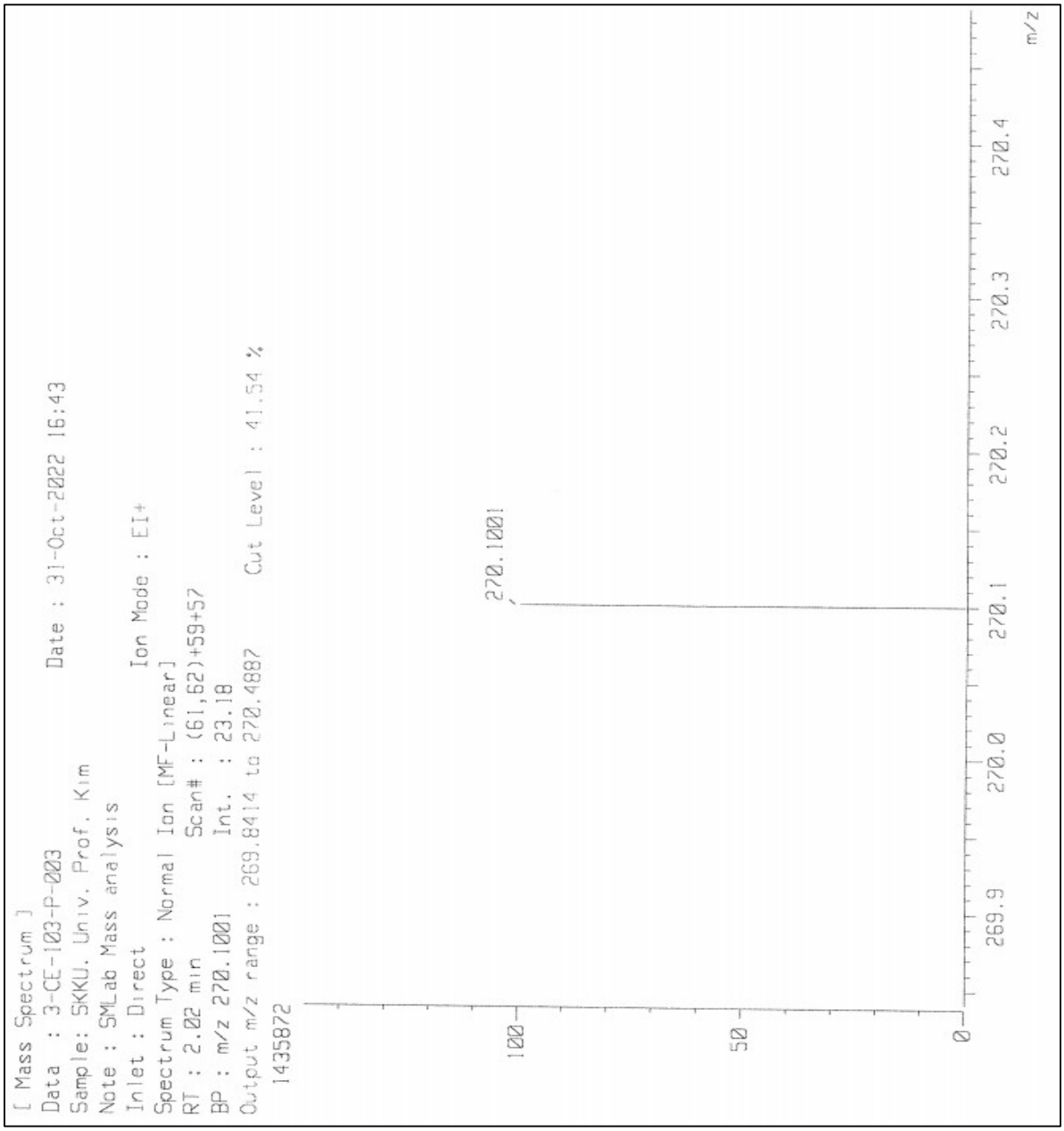


[Elemental Composition]
Data : 3-CE-103-P-003
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.02 min
Elements : C 16/0, H 15/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

Date : 31-Oct-2022 16:43
Ion Mode : EI+
Scan#: (61,62)+59+57

3q

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
270.1001	100.0	-1.3 / -0.3	10.0	C 15 H 14 O 3 N 2



[Elemental Composition] Page: 1

Data : 7-YV703-P-008 Date : 31-Oct-2022 17:02

Sample: SKKU. Univ. Prof. Kim

Note : SMLab Mass analysis

3r

Inlet : Direct Ion Mode : EI+

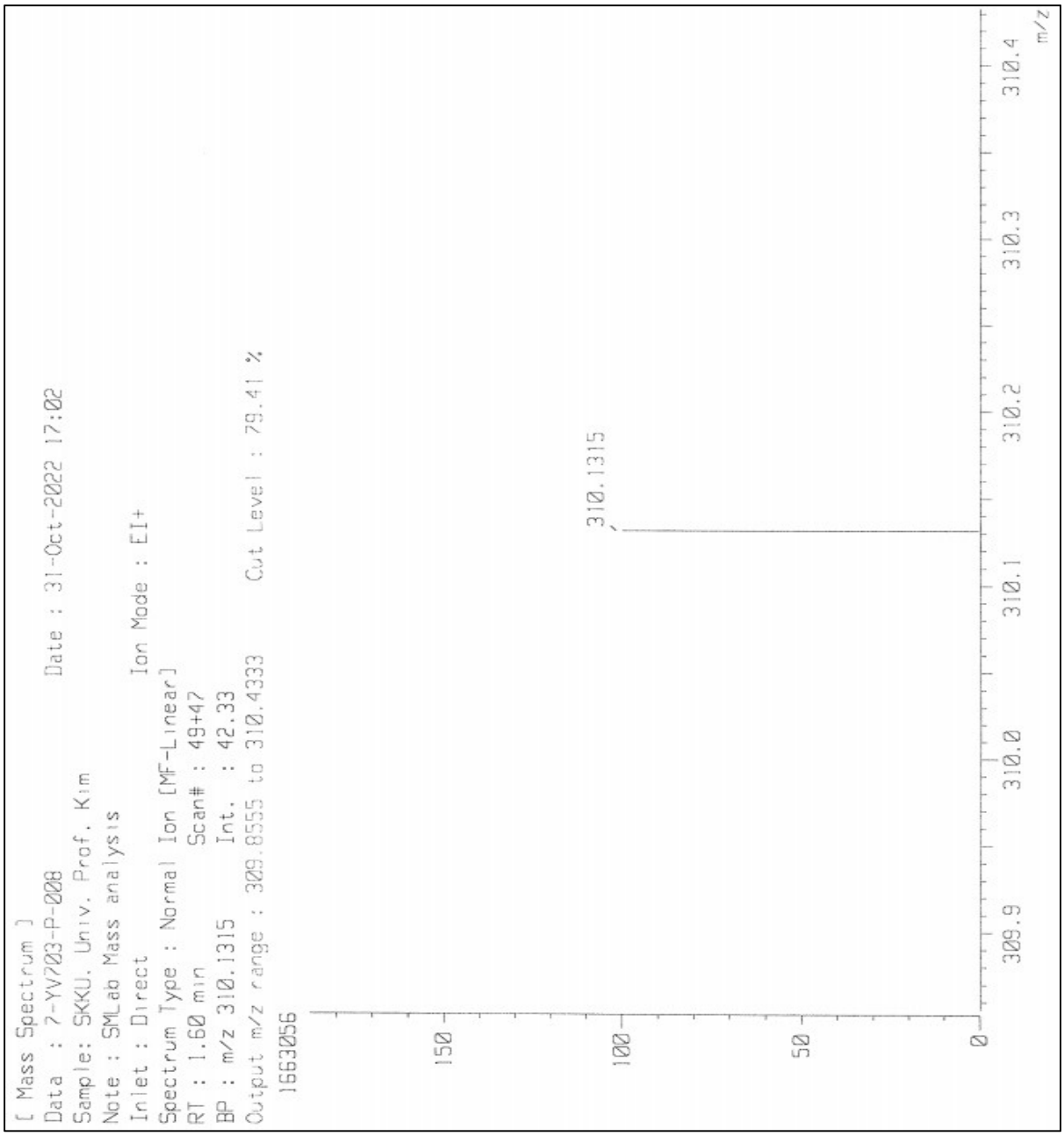
RT : 1.60 min Scan#: 49+47

Elements : C 20/0, H 21/0, O 3/0, N 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
310.1315	100.0	-0.7 / -0.2	11.0	C 18 H 18 O 3 N 2



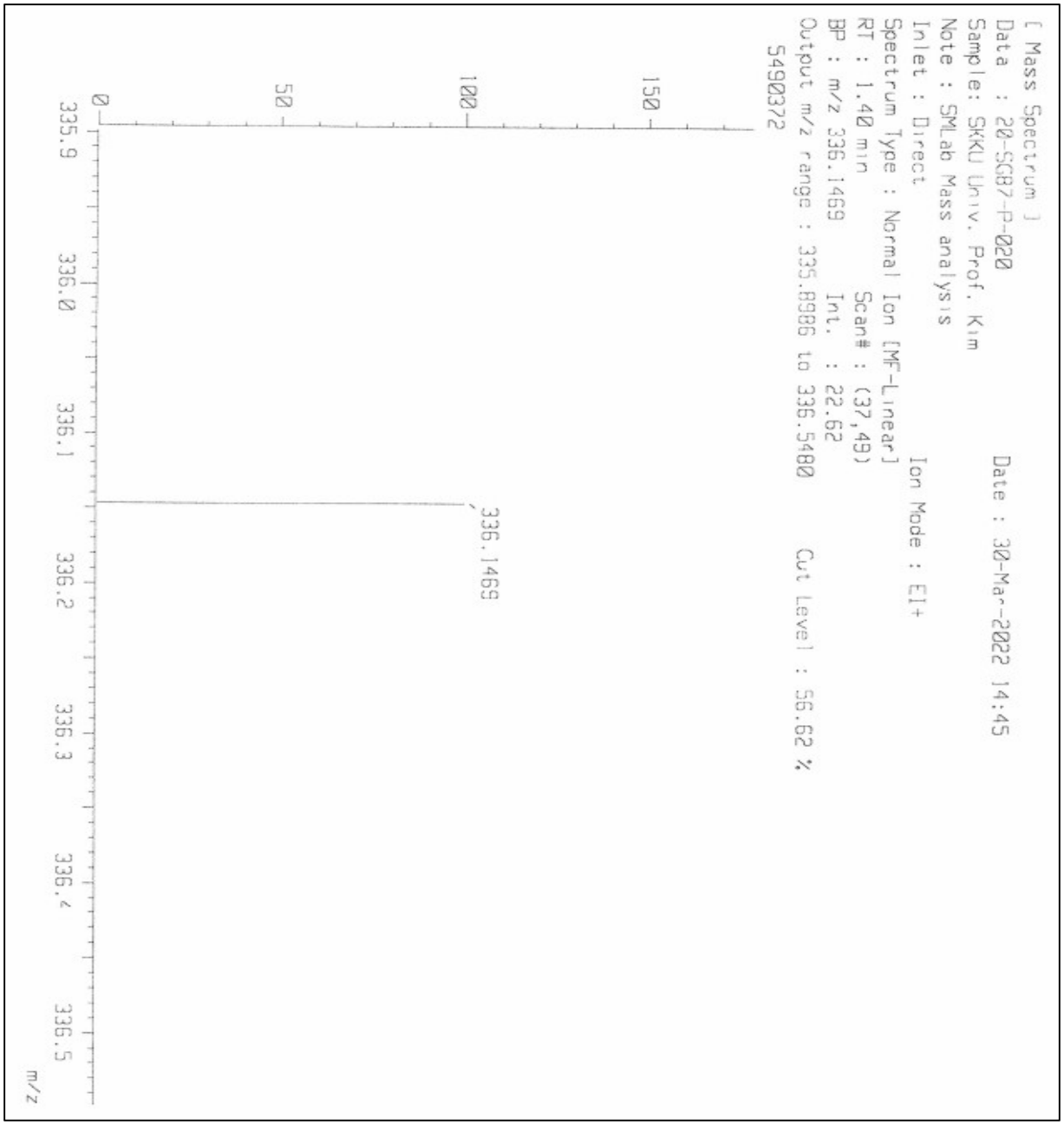
[Elemental Composition]
Data : 20-SG87-P-020
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.40 min
Elements : C 22/0, H 22/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 30-Mar-2022 14:45
Ion Mode : EI+
Scan#: (37,49)

Page: 1

3s

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
336.1469	100.0	-1.3 / -0.5	12.0	C 20 H 20 O 3 N 2



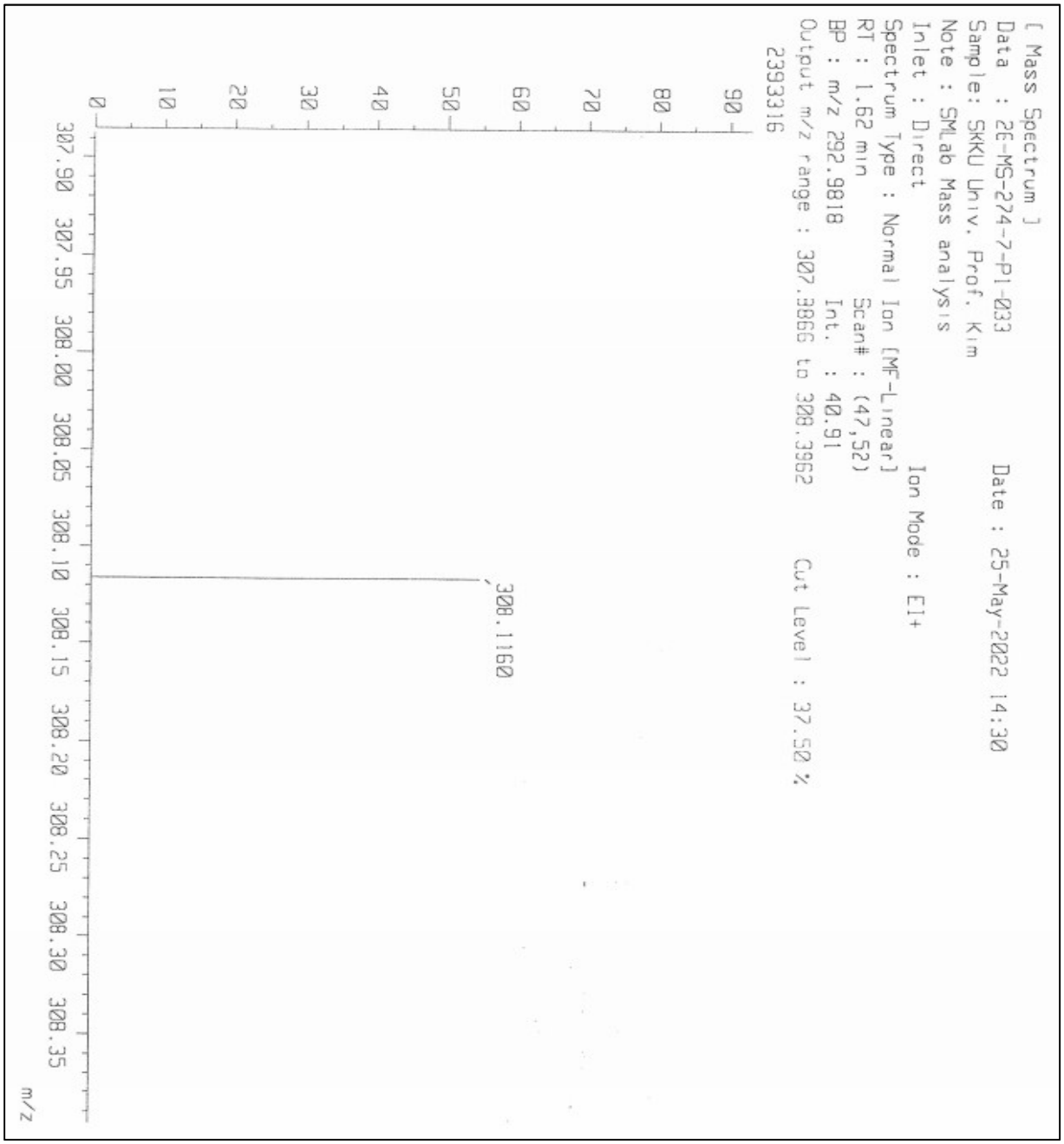
[Elemental Composition]

Page: 1

3t

Data : 26-MS-274-7-P1-033 Date : 25-May-2022 14:30
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 1.62 min Scan#: (47,52)
Elements : C 20/0, H 20/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
308.1160	54.9	-0.4 / -0.1	12.0	C 18 H 16 O 3 N 2



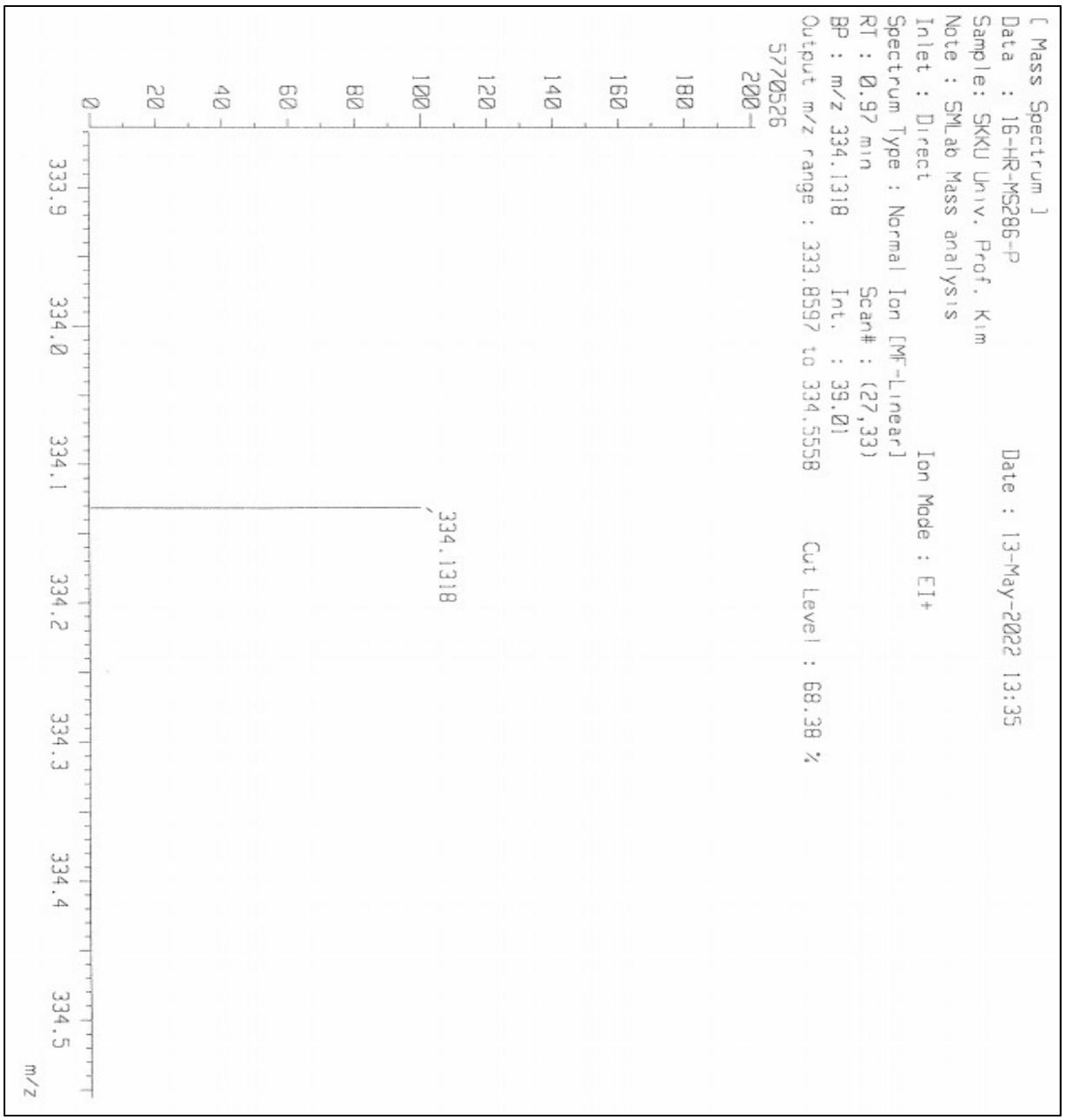
[Elemental Composition]
Data : 16-HR-MS286-P
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 0.97 min
Elements : C 20/0, H 20/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 13-May-2022 13:35
Ion Mode : EI+
Scan#: (27,33)

Page: 1

4b

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
334.1318	100.0	+0.2 / +0.1	13.0	C 20 H 18 O 3 N 2

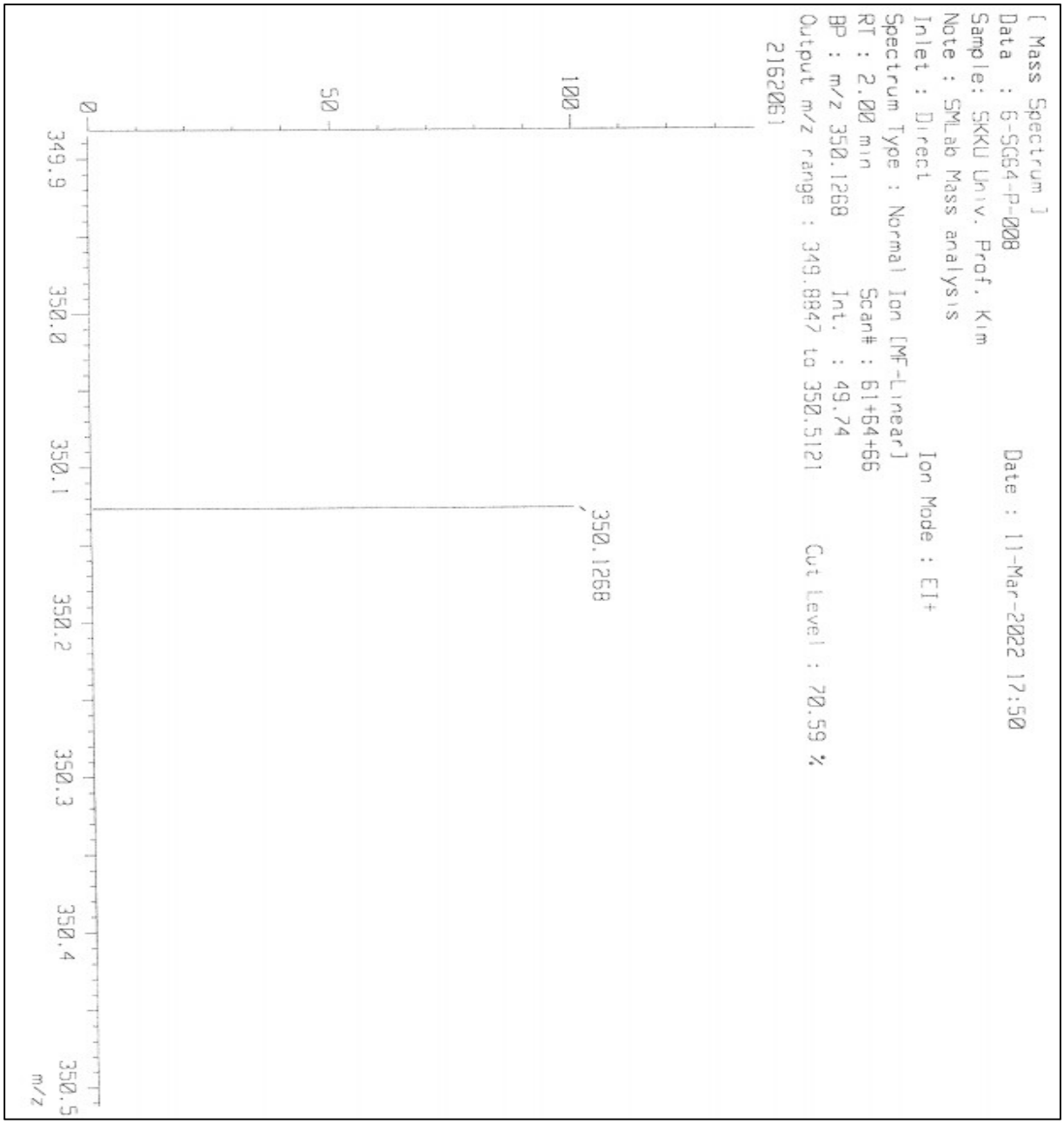


[Elemental Composition]
Data : 6-SG64-P-008
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 2.00 min
Elements : C 22/0, H 20/0, O 4/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 11-Mar-2022 17:50
Ion Mode : EI+
Scan#: 61+64+66

4c

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
350.1268	100.0	+0.4 / +0.1	13.0	C 20 H 18 O 4 N 2



[Elemental Composition] Page: 1

Data : 18-SG63-P-018 Date : 30-Mar-2022 14:35

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

RT : 1.87 min Scan#: (55,59)+51

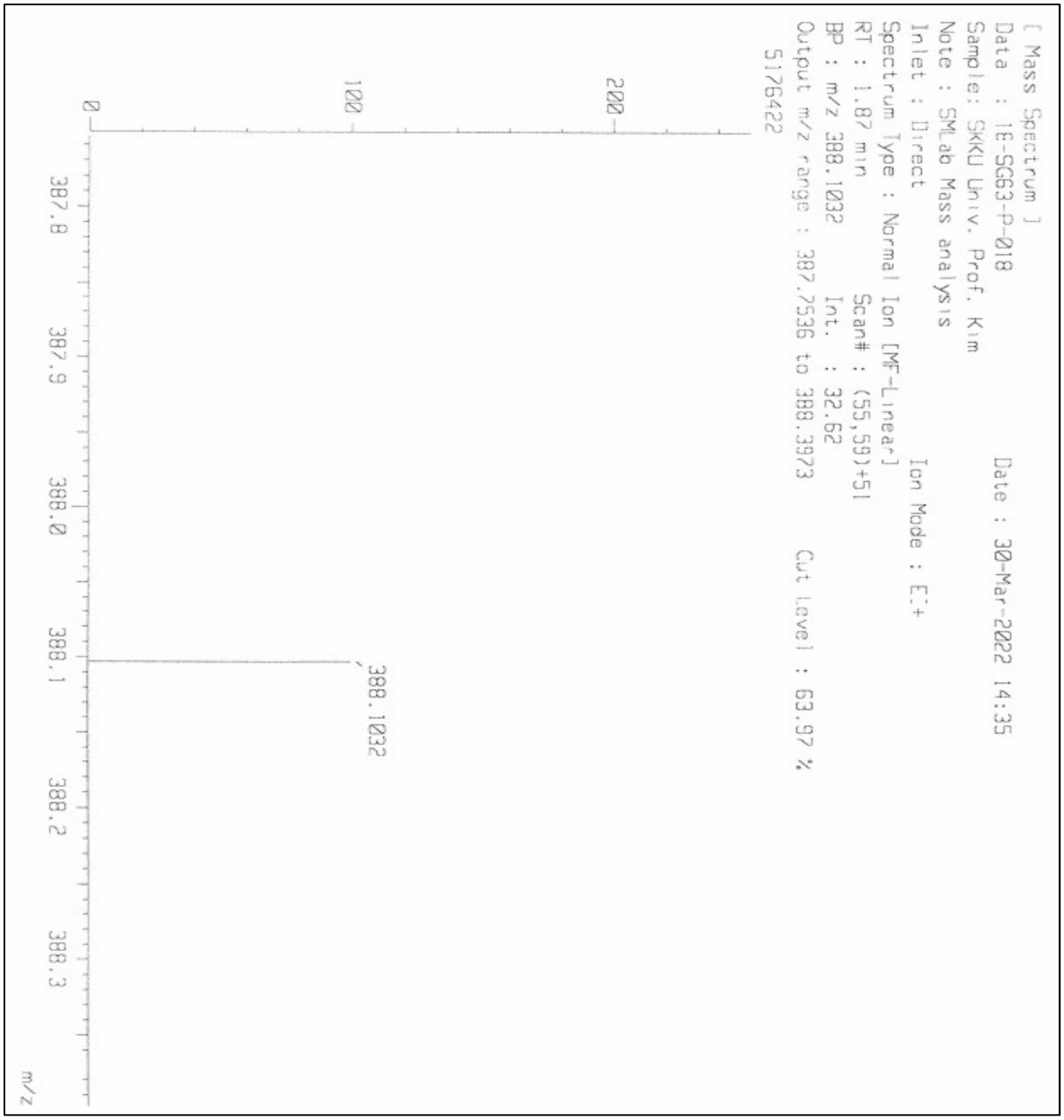
Elements : C 21/0, H 16/0, O 3/0, N 2/0, F 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
388.1032	100.0	-0.8 / -0.3	13.0	C 20 H 15 O 3 N 2 F 3

4d



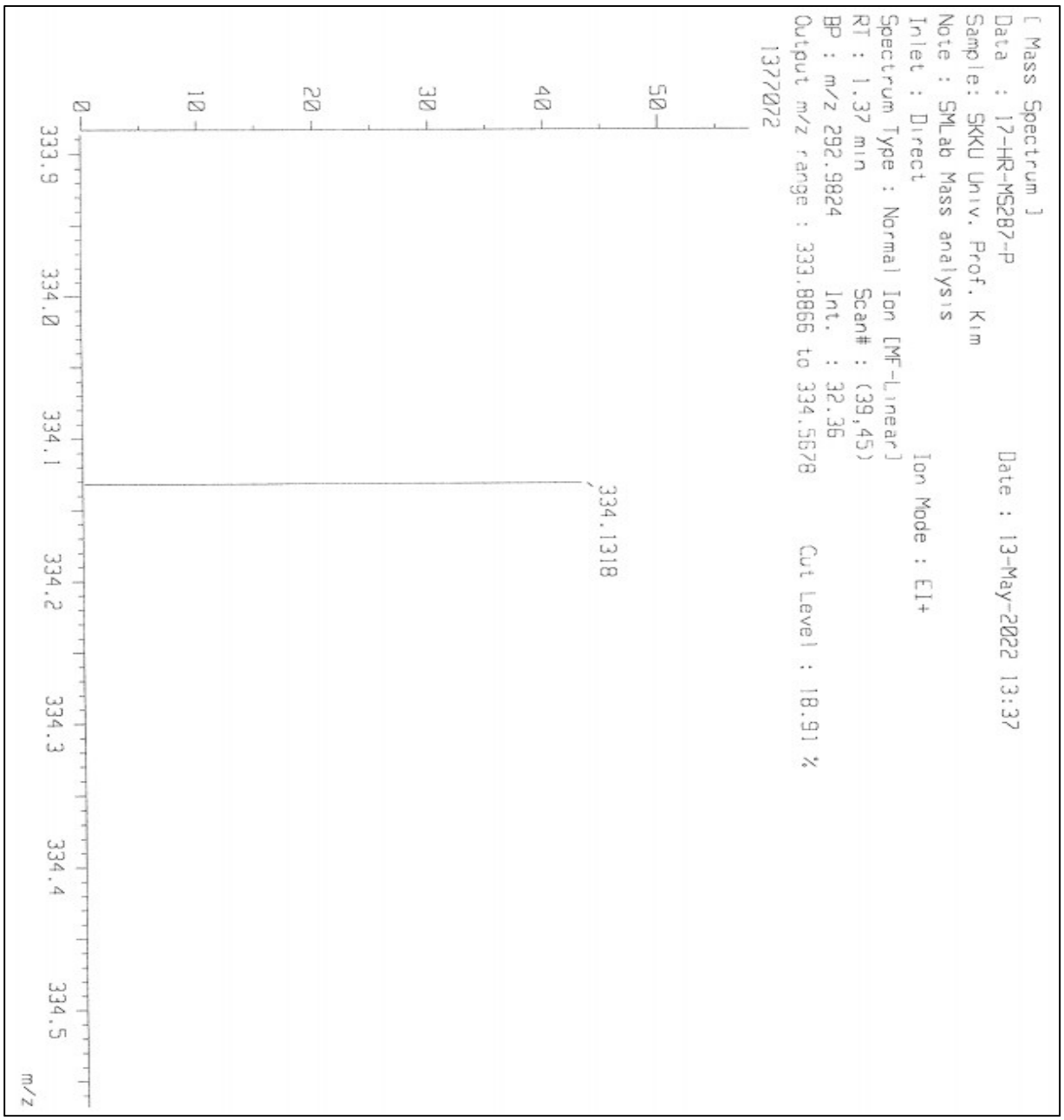
[Elemental Composition]

Page: 1

4e

Data : 17-HR-MS287-P Date : 13-May-2022 13:37
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 1.37 min Scan#: (39,45)
Elements : C 20/0, H 20/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
334.1318	43.2	+0.1 / +0.0	13.0	C 20 H 18 O 3 N 2



[Elemental Composition] Page: 1

Data : 7-SG65-P-009 Date : 11-Mar-2022 17:54

4f

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

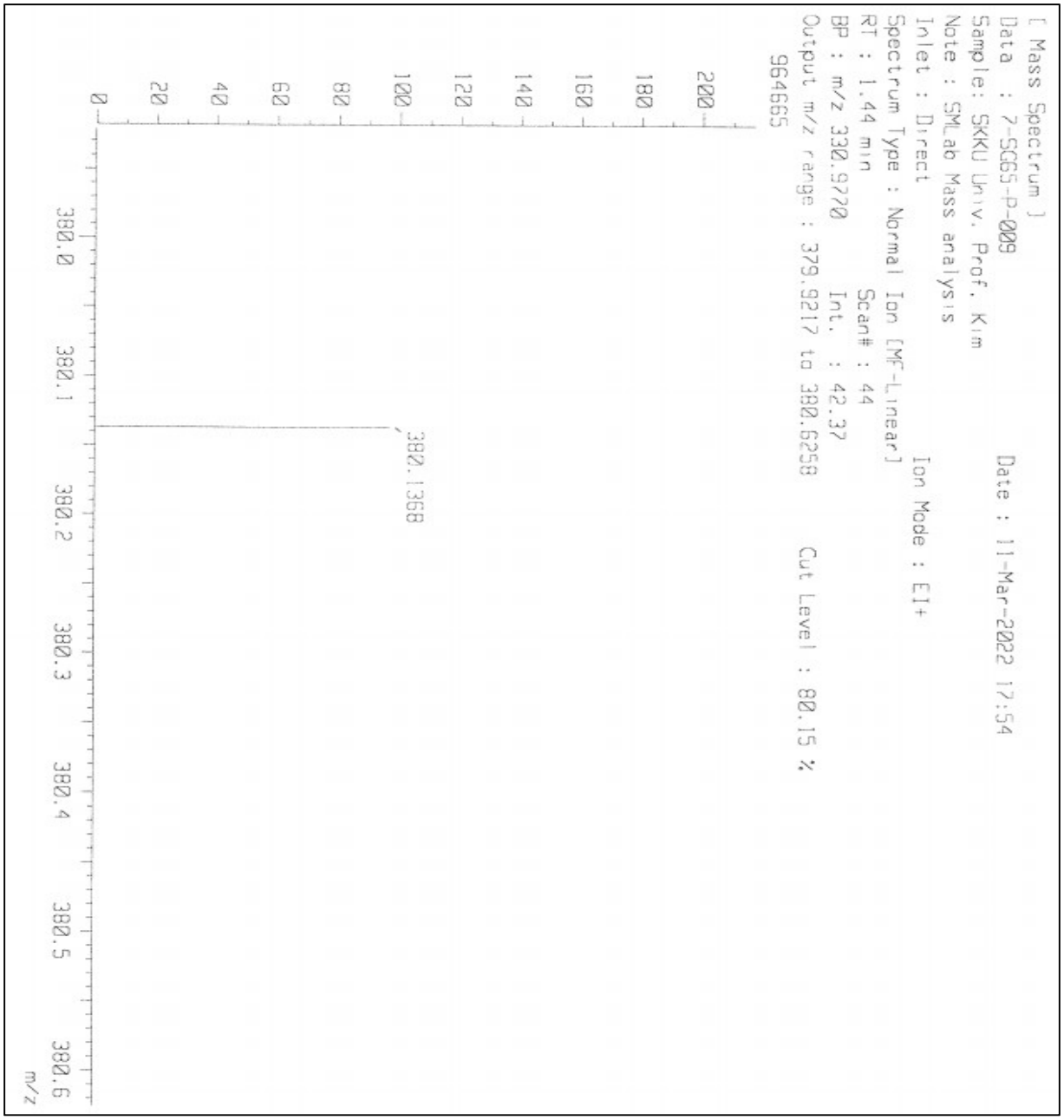
RT : 1.44 min Scan#: 44

Elements : C 22/0, H 22/0, O 6/0, N 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
380.1368	96.9	-1.1 / -0.4	13.0	C 21 H 20 O 5 N 2

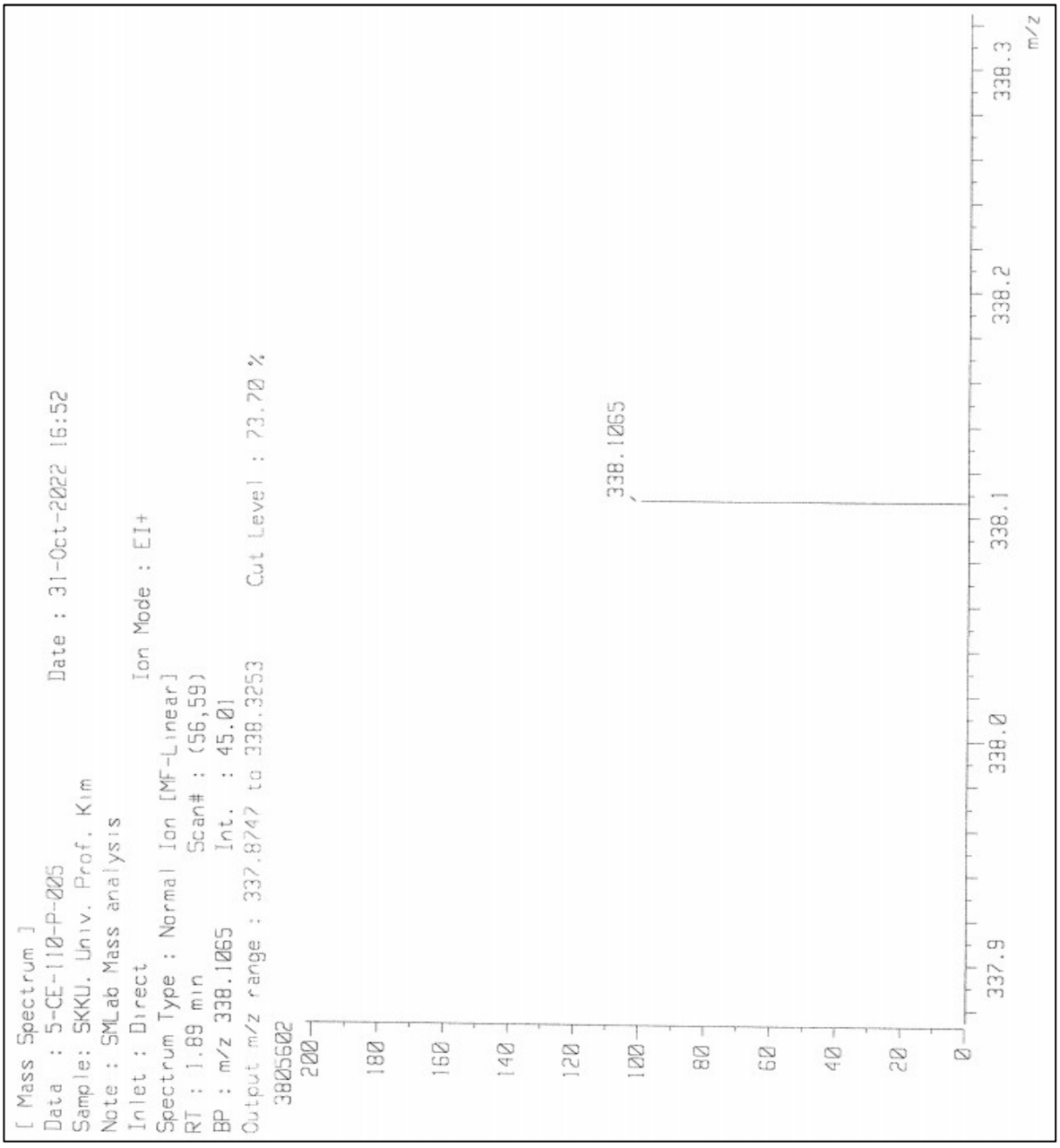


[Elemental Composition]
Data : 5-CE-110-P-005
Sample: SKKU. Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.89 min
Elements : C 20/0, H 21/0, O 3/0, N 3/0, F 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 70.0

Date : 31-Oct-2022 16:52
Ion Mode : EI+
Scan#: (56,59)

4g

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
338.1065	100.0	-0.4 / -0.1	13.0	C 19 H 15 O 3 N 2 F



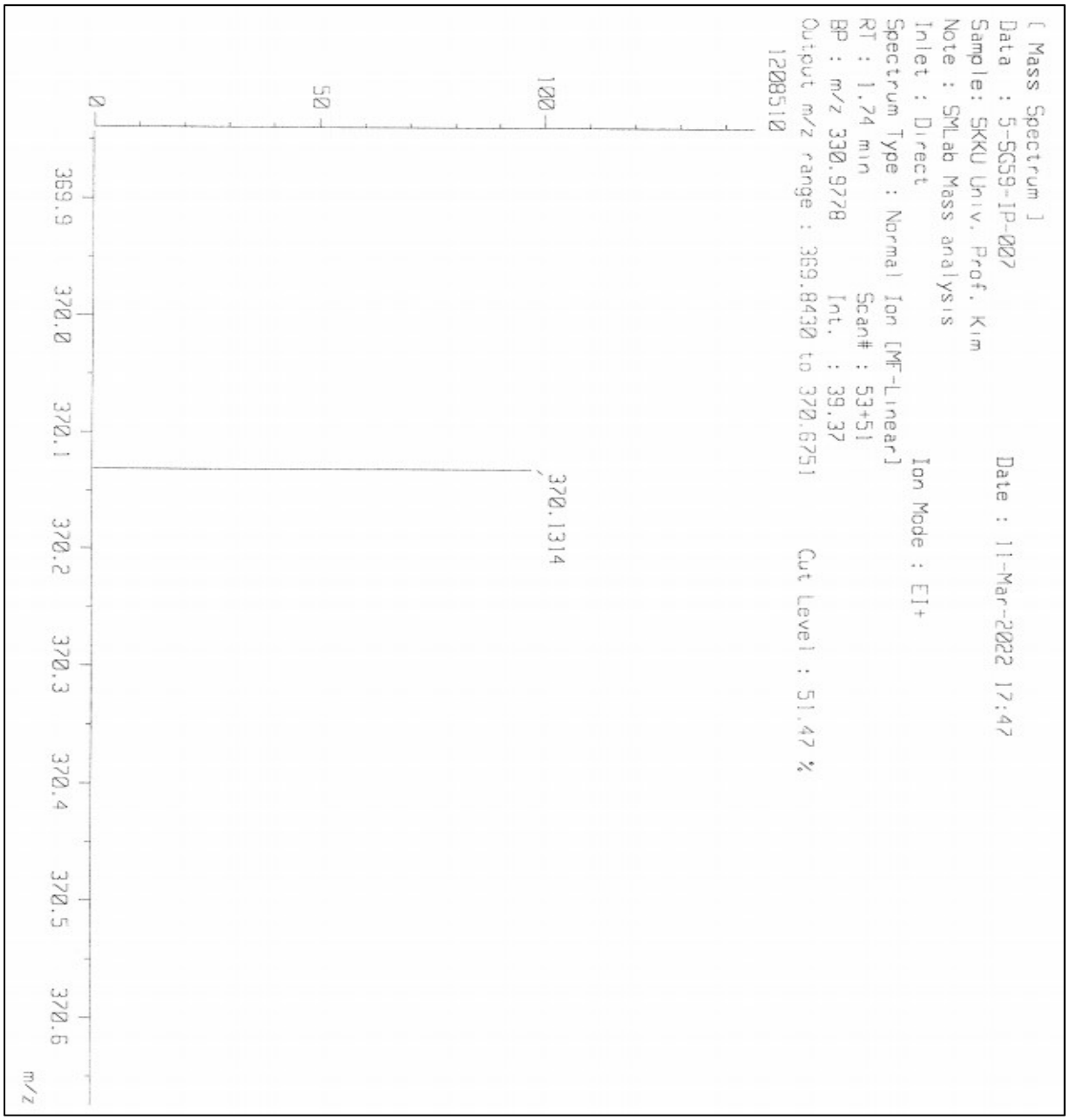
[Elemental Composition]
Data : 5-SG59-IP-007
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.74 min
Elements : C 23/0, H 20/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 11-Mar-2022 17:47
Ion Mode : EI+
Scan#: 53+51

Page: 1

4h

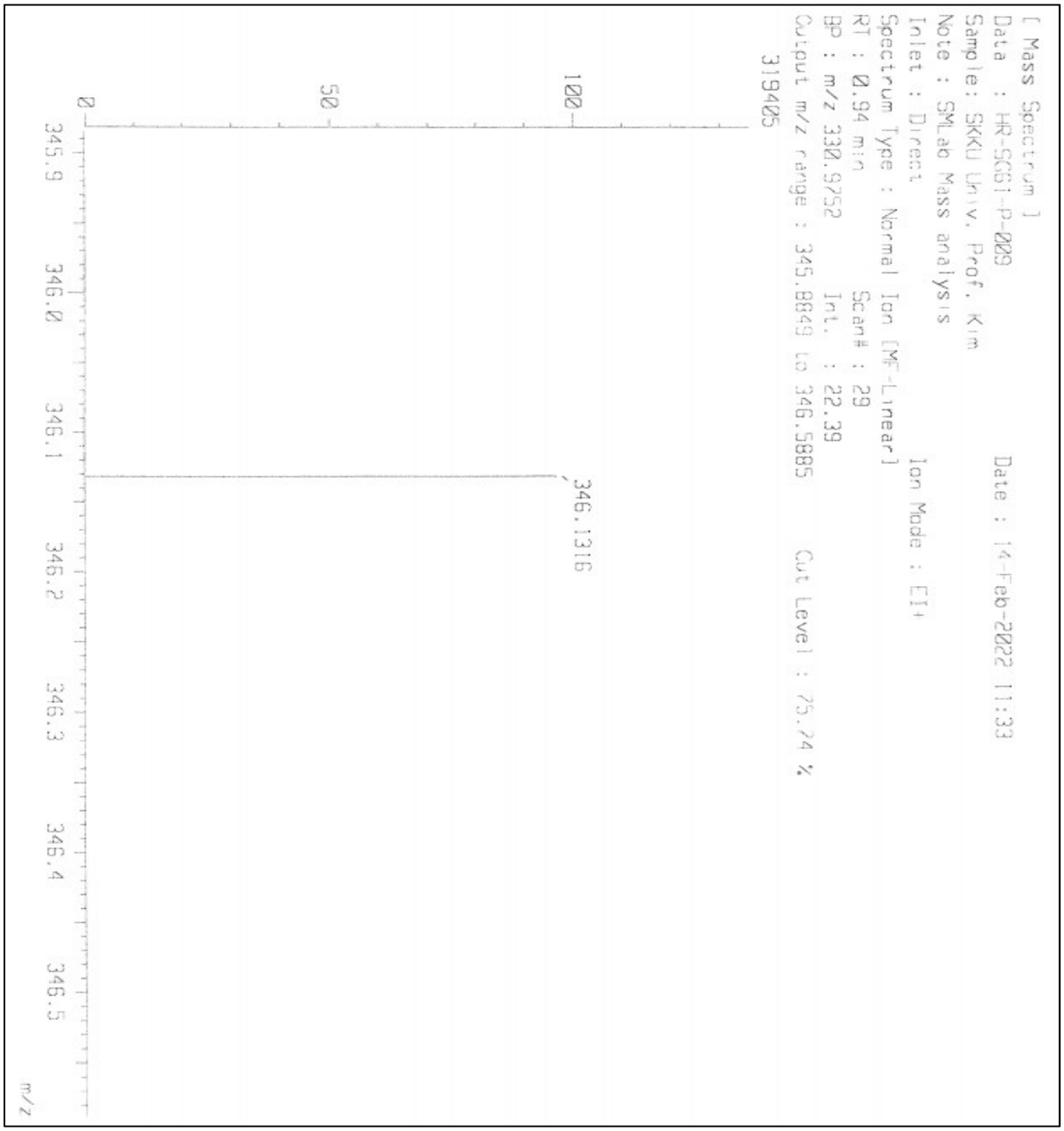
Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
370.1314	97.4	-0.9 / -0.3	16.0	C 23 H 18 O 3 N 2



[Elemental Composition]
Data : HR-SG61-P-009 Date : 14-Feb-2022 11:33
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 0.94 min Scan#: 29
Elements : C 22/0, H 20/0, O 4/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
346.1316	96.7	-0.5 / -0.2	14.0	C 21 H 18 O 3 N 2

4i

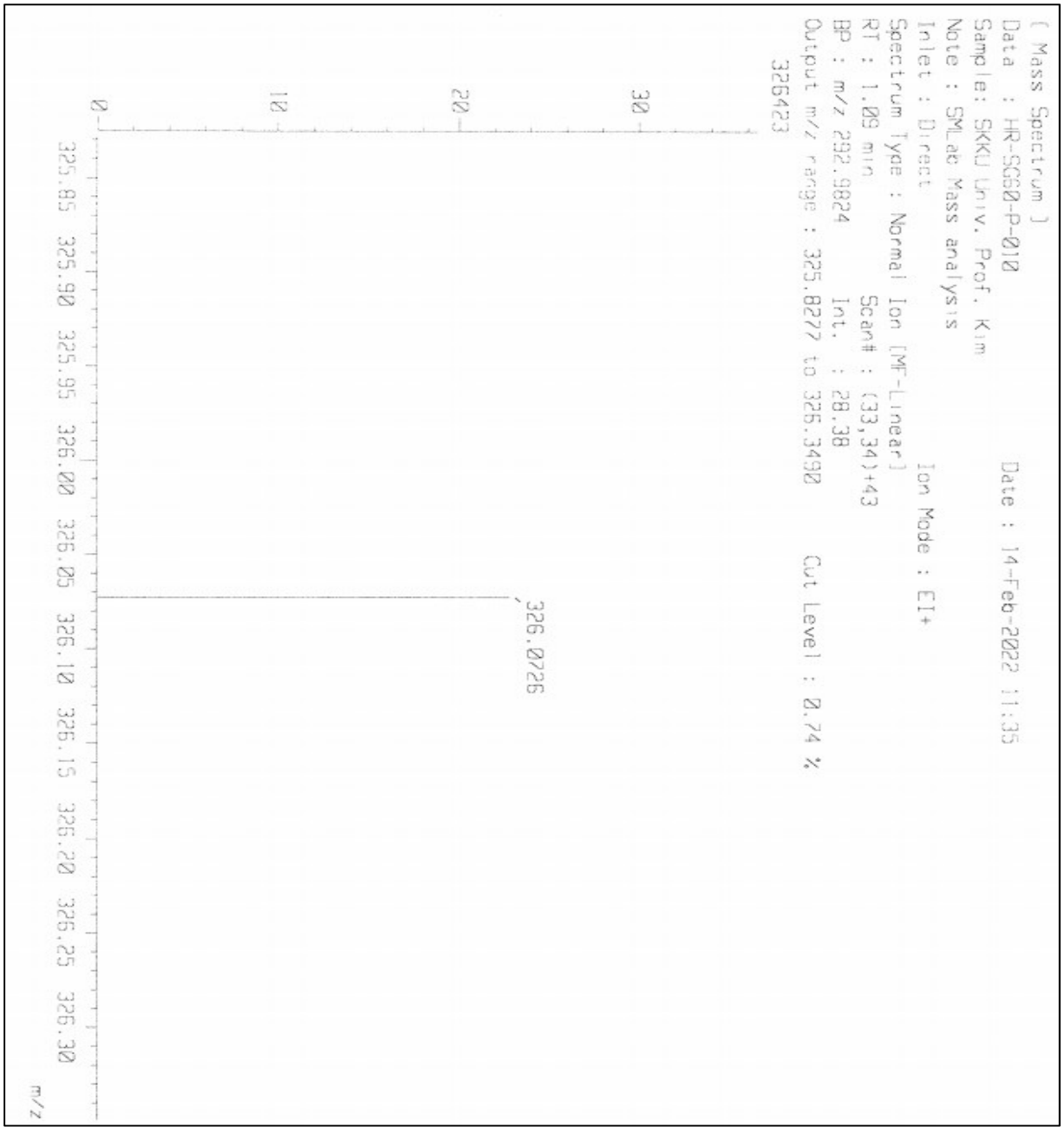


[Elemental Composition]
Data : HR-SG60-P-010
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.09 min
Elements : C 18/0, H 15/0, O 4/0, N 2/0, S 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 14-Feb-2022 11:35
Ion Mode : EI+
Scan#: (33,34)+43

4j

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
326.0726	22.8	+0.2 / +0.1	13.0	C 17 H 14 O 3 N 2 S



Page: 1

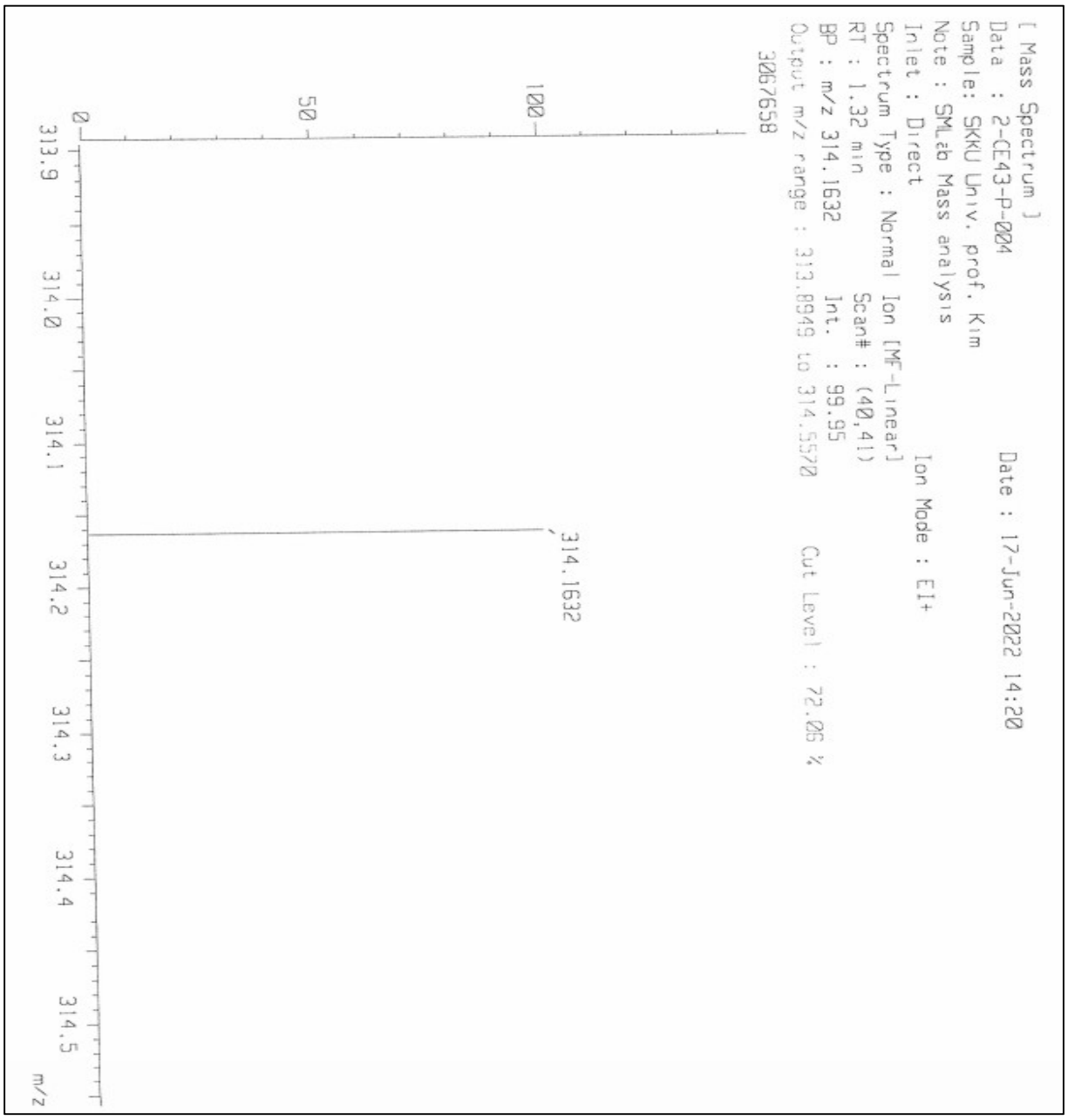
4k

[Elemental Composition]
Data : 2-CE43-P-004
Sample: SKKU Univ. prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.32 min
Elements : C 20/0, H 23/0, O 3/0, N 3/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 17-Jun-2022 14:20

Ion Mode : EI+
Scan#: (40,41)

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
314.1632	100.0	+0.4 / +0.1	9.0	C 18 H 22 O 3 N 2



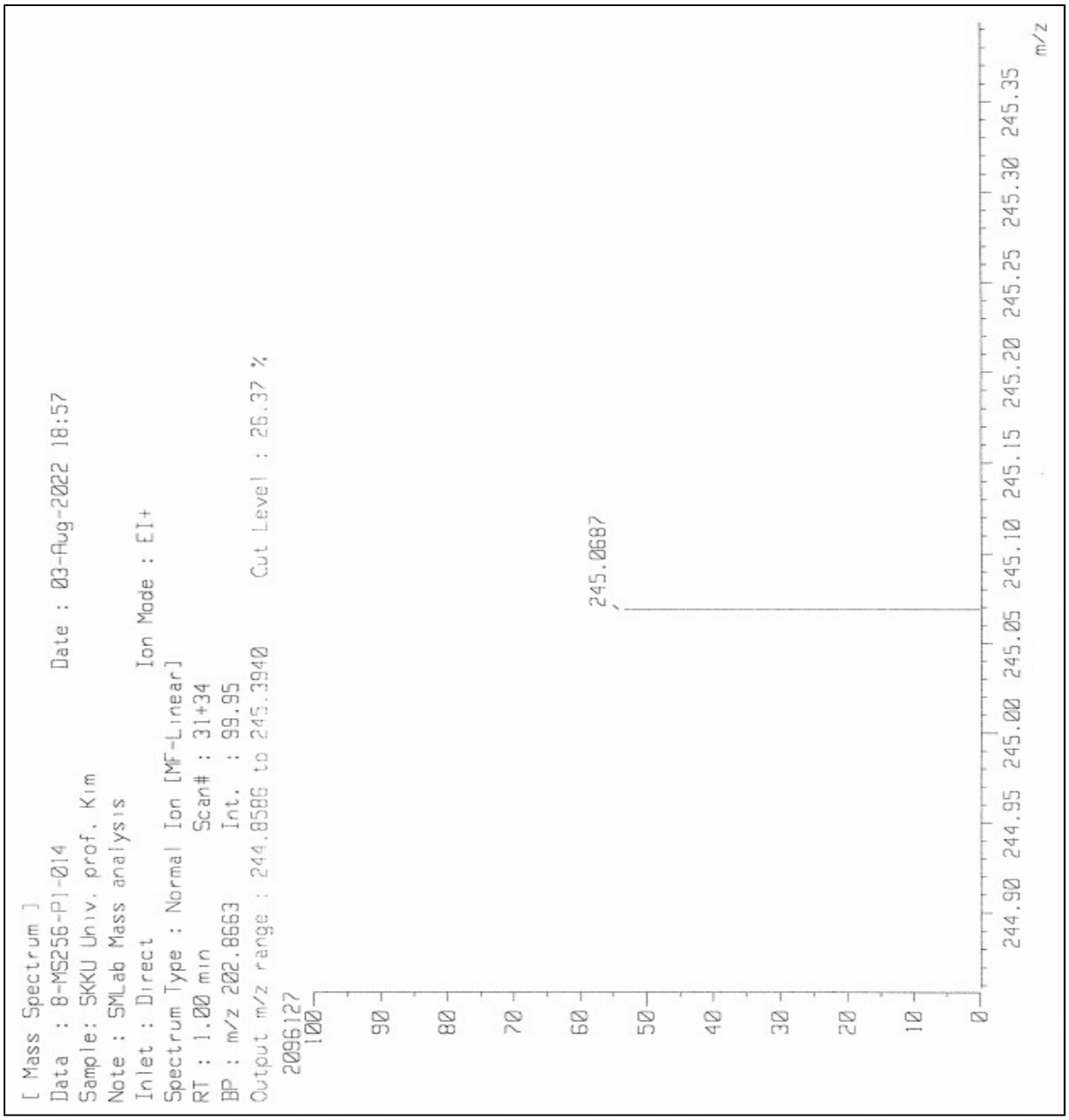
[Elemental Composition]
Data : 8-MS256-P1-014
Sample: SKKU Univ. prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.00 min
Elements : C 16/0, H 15/0, O 4/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 03-Aug-2022 18:57

Ion Mode : EI+
Scan#: 31+34

5a

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
245.0687	53.5	-0.3 / -0.1	9.0	C 13 H 11 O 4 N



[Elemental Composition] Page: 1

Data : 15-HR-MS284-P Date : 13-May-2022 13:32

5b

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

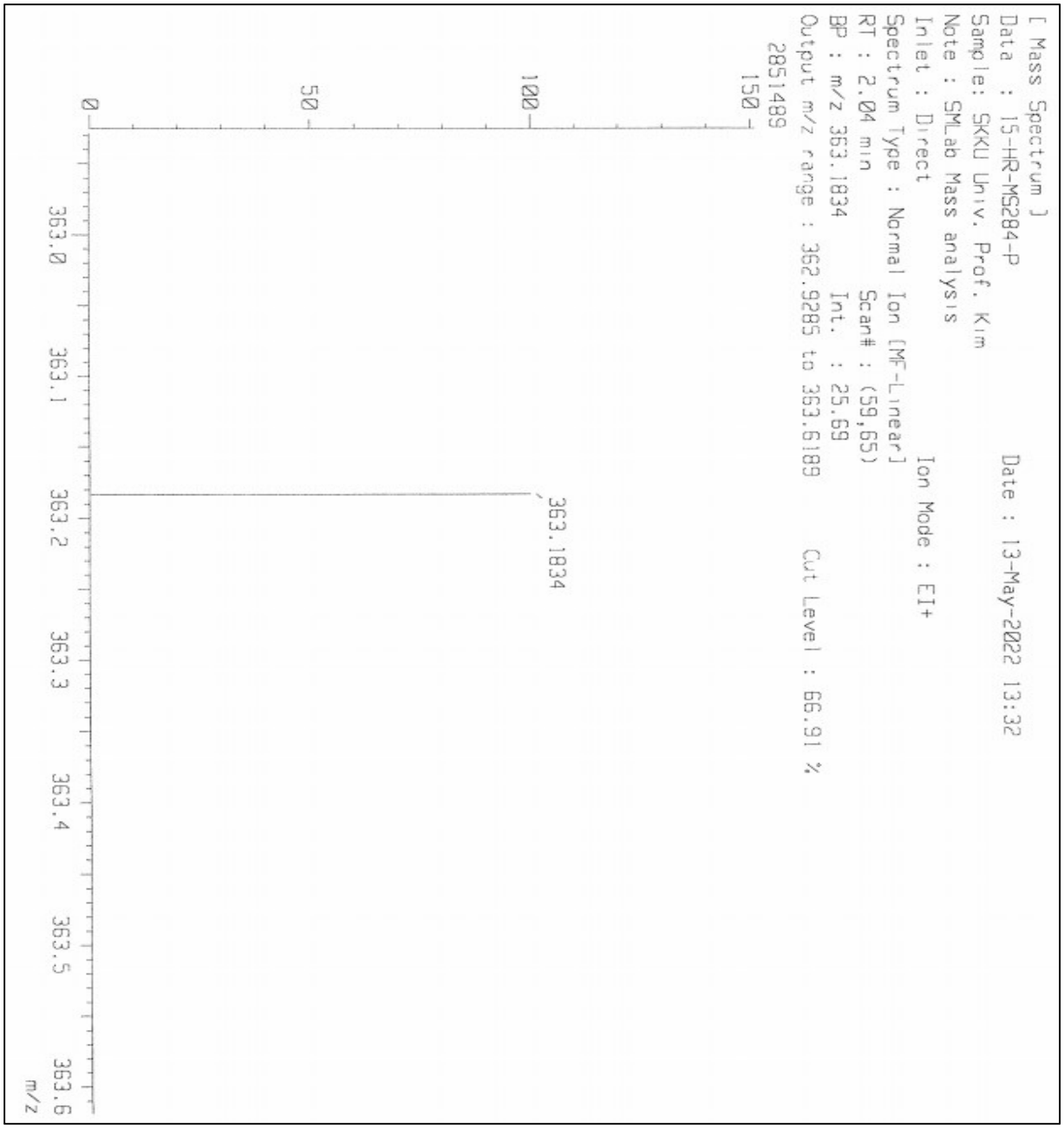
RT : 2.04 min Scan#: (59,65)

Elements : C 25/0, H 28/0, O 3/0, N 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
363.1834	100.0	-0.1 / +0.0	12.0	C 23 H 25 O 3 N

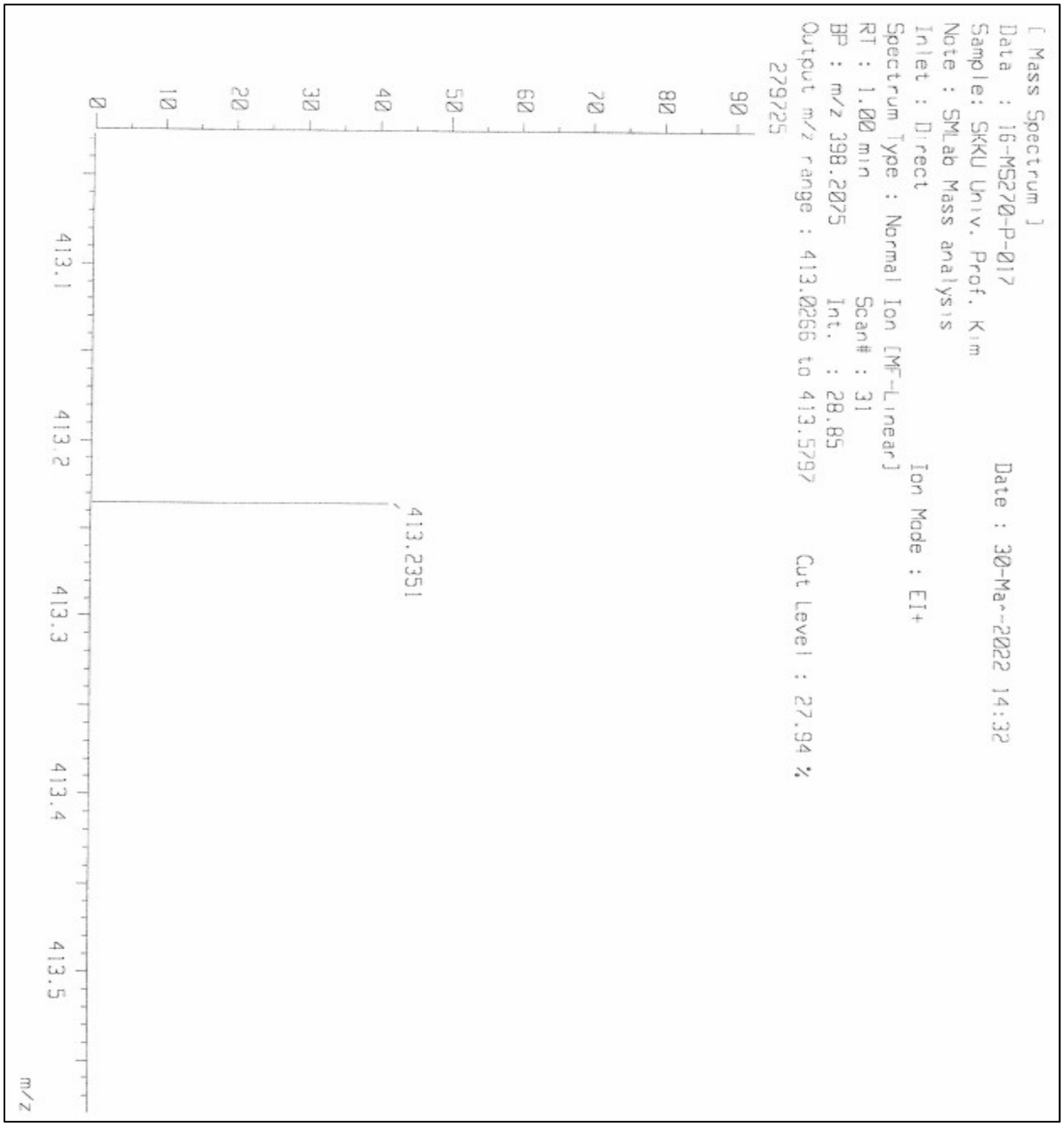


[Elemental Composition]
Data : 16-MS270-P-017
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.00 min
Elements : C 30/0, H 35/0, O 2/0, N 1/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 30-Mar-2022 14:32
Ion Mode : EI+
Scan#: 31

5c

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
413.2351	41.6	-0.8 / -0.3	14.0	C 28 H 31 O 2 N

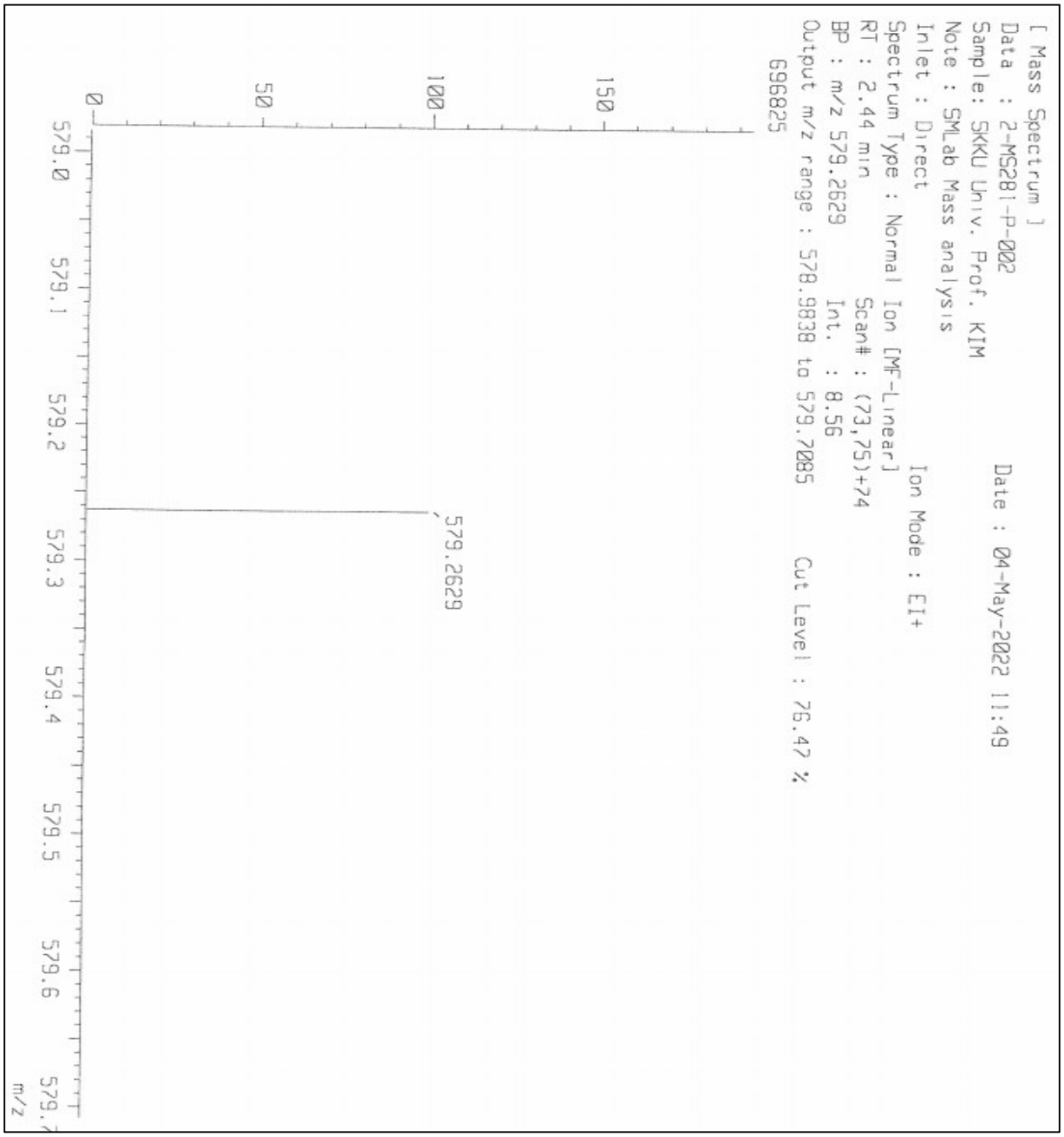


[Elemental Composition]
Data : 2-MS281-P-002 Date : 04-May-2022 11:49
Sample: SKKU Univ. Prof. KIM
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 2.44 min Scan#: (73,75)+74
Elements : C 40/0, H 35/0, O 2/0, N 6/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
579.2629	100.0	-0.9 / -0.5	24.0	C 37 H 33 O 2 N 5

Page: 1

5d



[Elemental Composition] Page: 1

Data : 17-FAB-MS273-P-007 Date : 30-Mar-2022 12:25

Sample: SKKU Univ. Prof. Kim

Note : SM Lab Research Center

5e

Inlet : Reserv. Ion Mode : FAB+

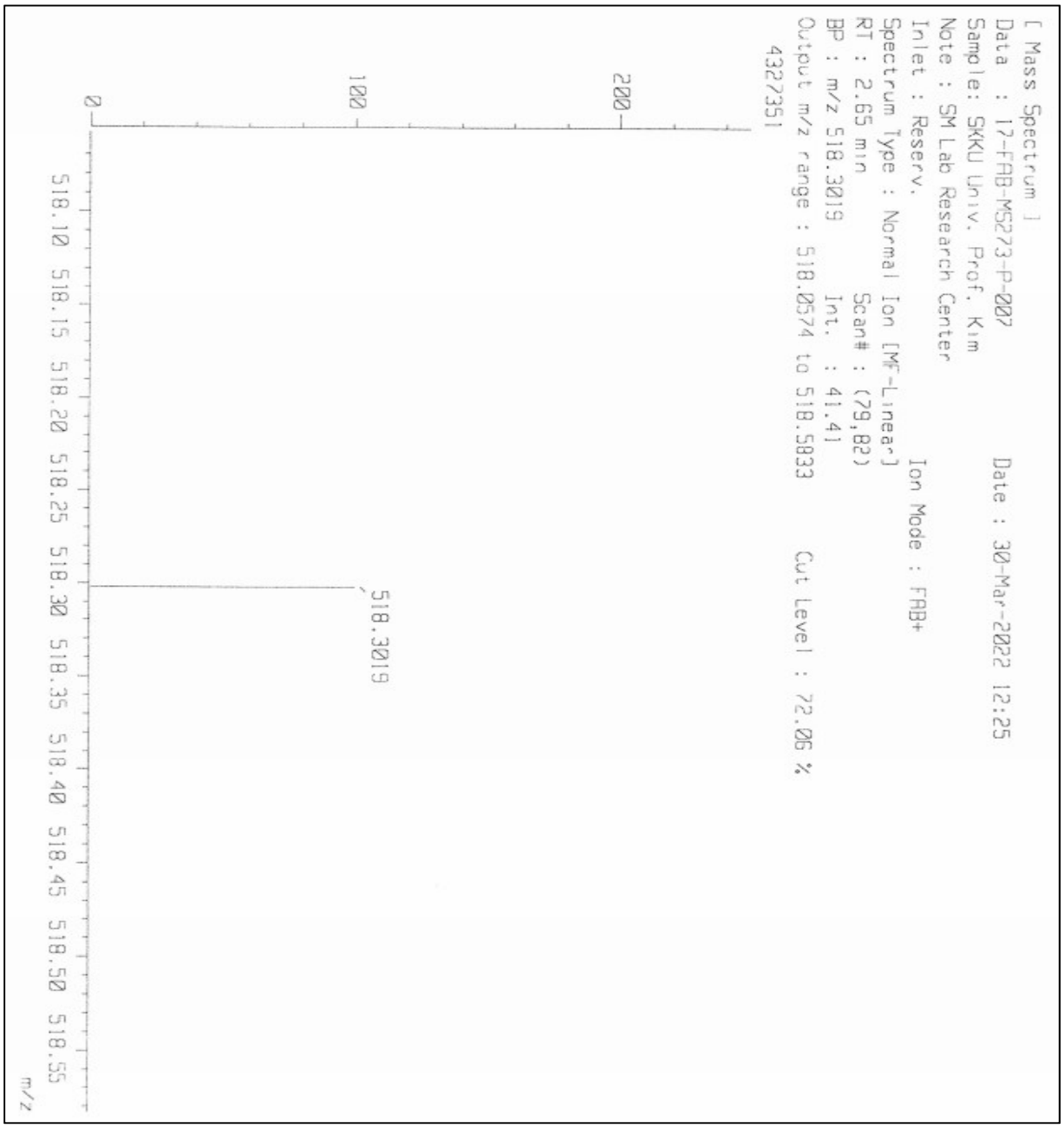
RT : 2.65 min Scan#: (79,82)

Elements : C 32/0, H 40/0, O 5/0, N 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 80.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
518.3019	100.0	+0.0 / +0.0	13.5	C 31 H 40 O 4 N 3

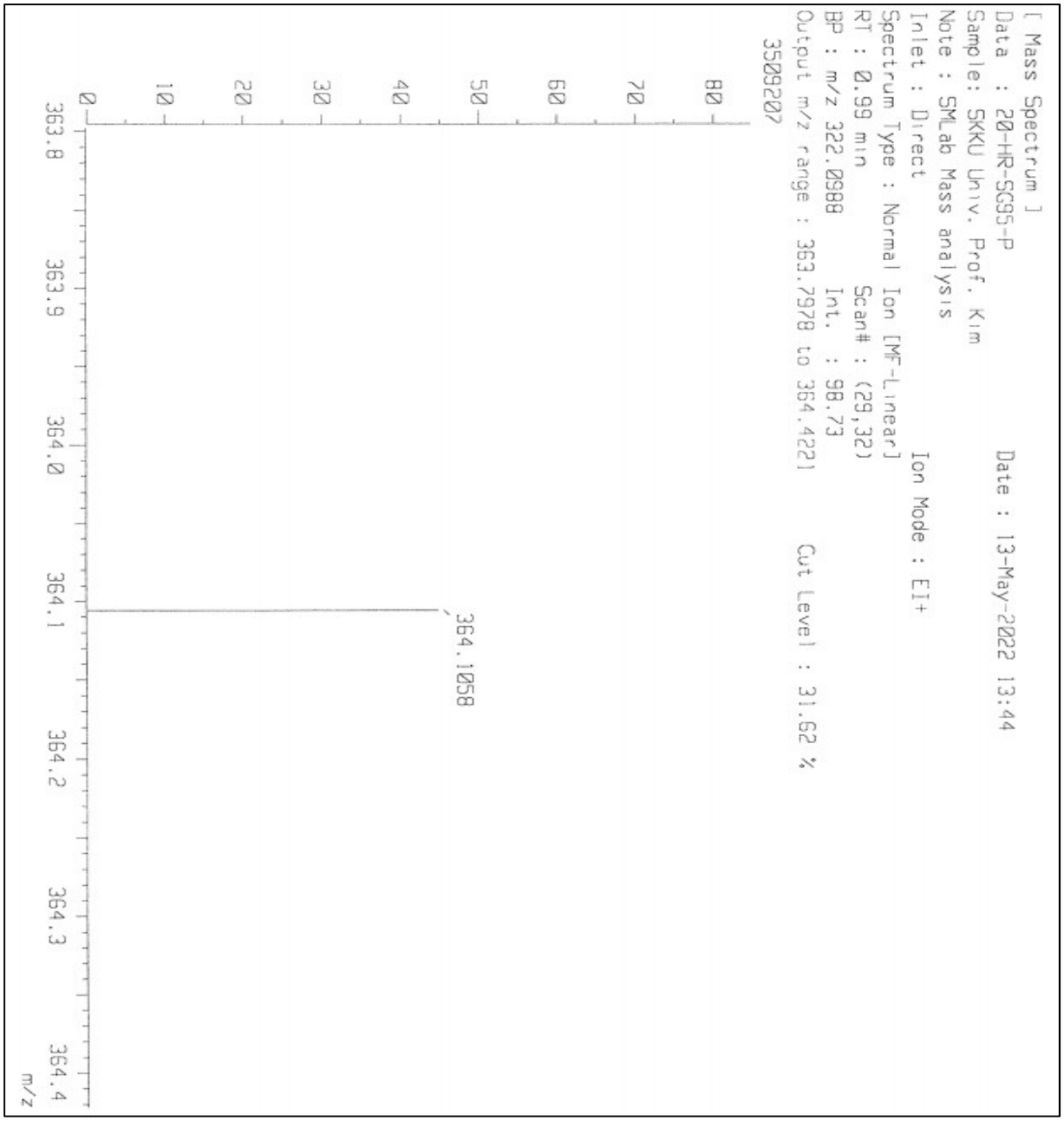


[Elemental Composition]
Page: 1

6a

Data : 20-HR-SG95-P
Date : 13-May-2022 13:44
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
Ion Mode : EI+
RT : 0.99 min
Scan#: (29,32)
Elements : C 20/0, H 18/0, O 5/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
364.1058	44.8	-0.3 / -0.1	14.0	C 20 H 16 O 5 N 2

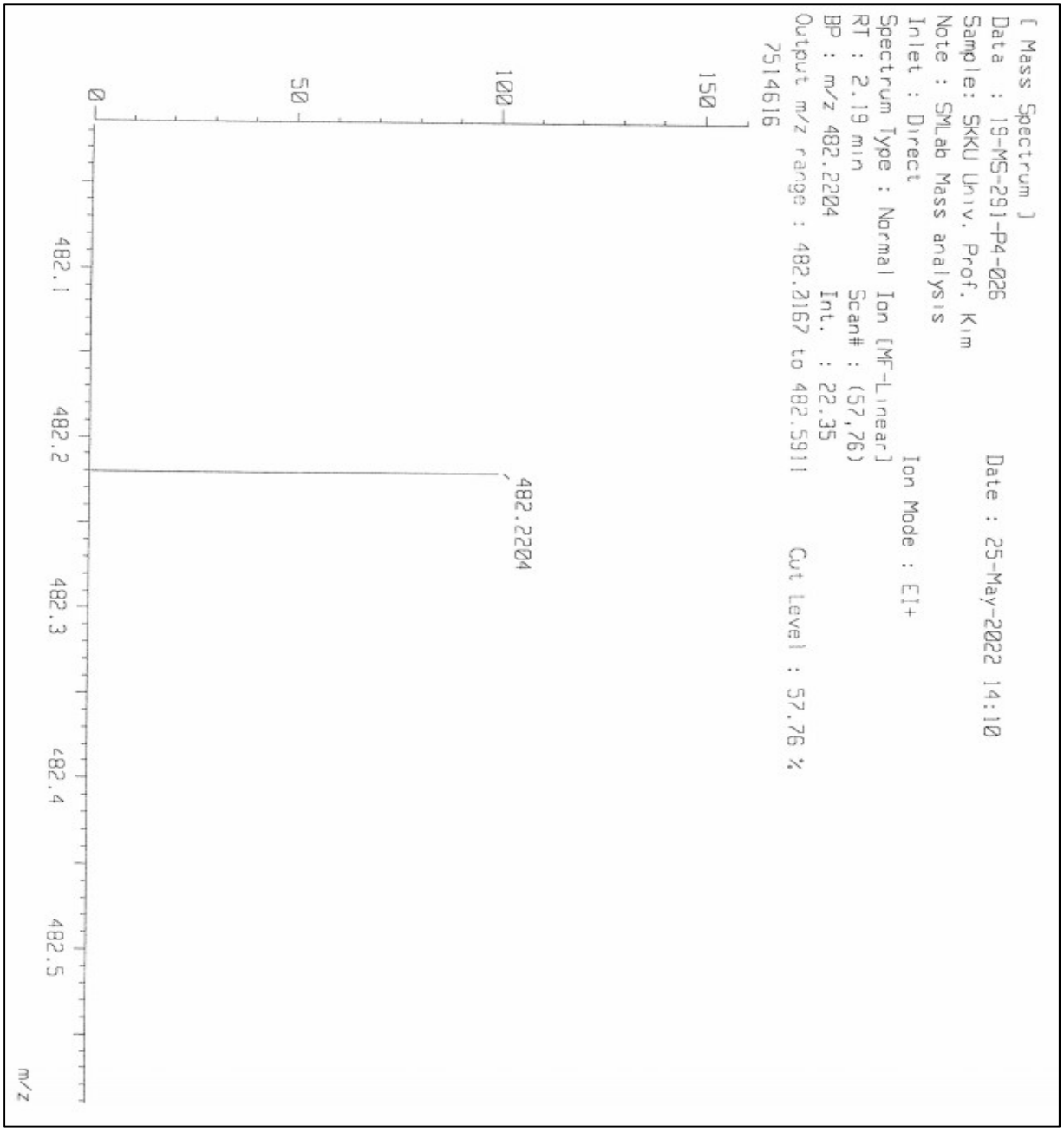


[Elemental Composition] Page: 1

6b

Data : 19-MS-291-P4-026 Date : 25-May-2022 14:10
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 2.19 min Scan#: (57,76)
Elements : C 30/0, H 33/0, O 4/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
482.2204	100.0	-0.3 / -0.2	17.0	C 30 H 30 O 4 N 2



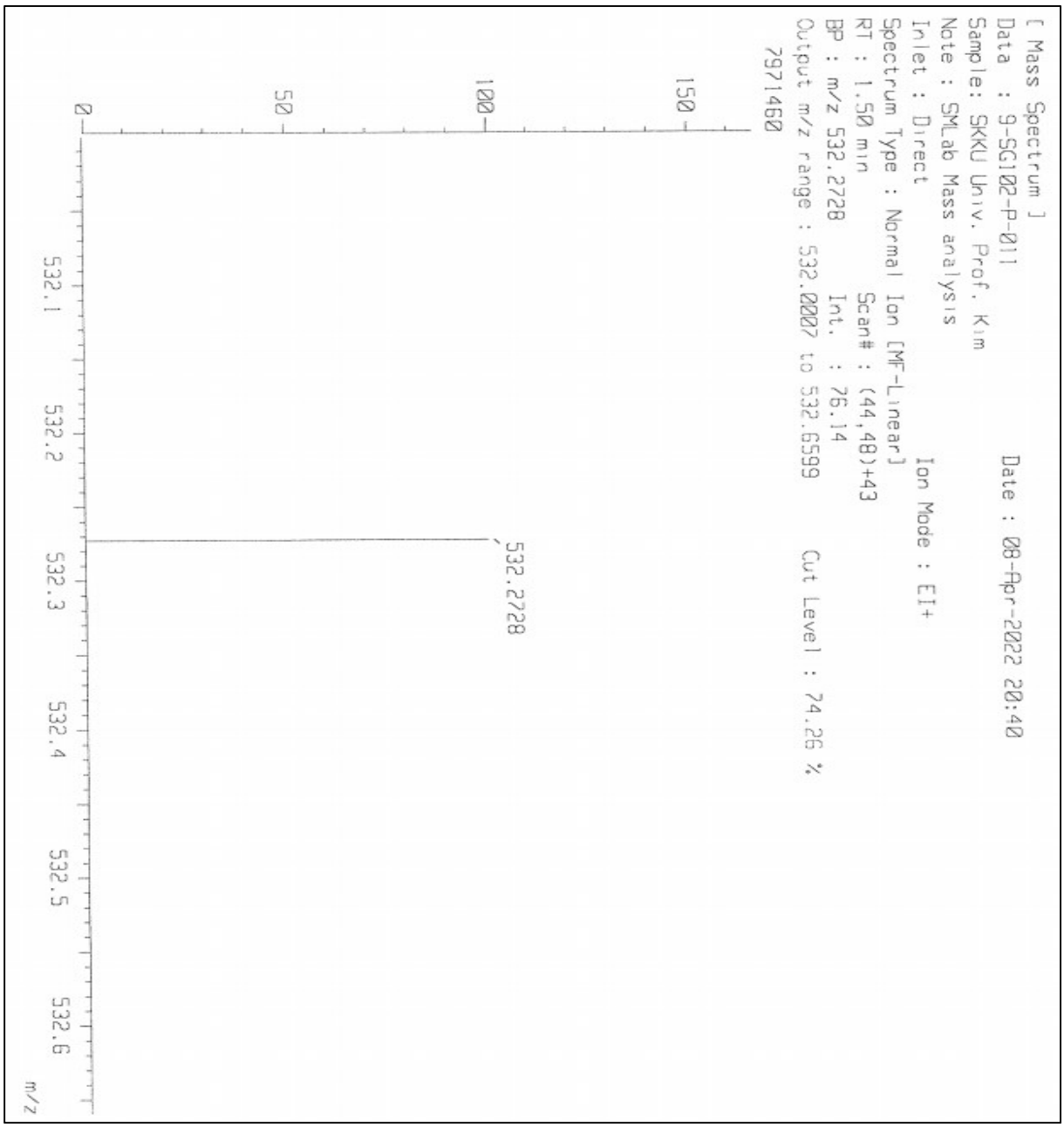
6c

[Elemental Composition]
Data : 9-SG102-P-011
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.50 min
Elements : C 37/0, H 37/0, O 3/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 08-Apr-2022 20:40

Ion Mode : EI+
Scan#: (44,48)+43

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
532.2728	100.0	+0.3 / +0.2	19.0	C 35 H 36 O 3 N 2



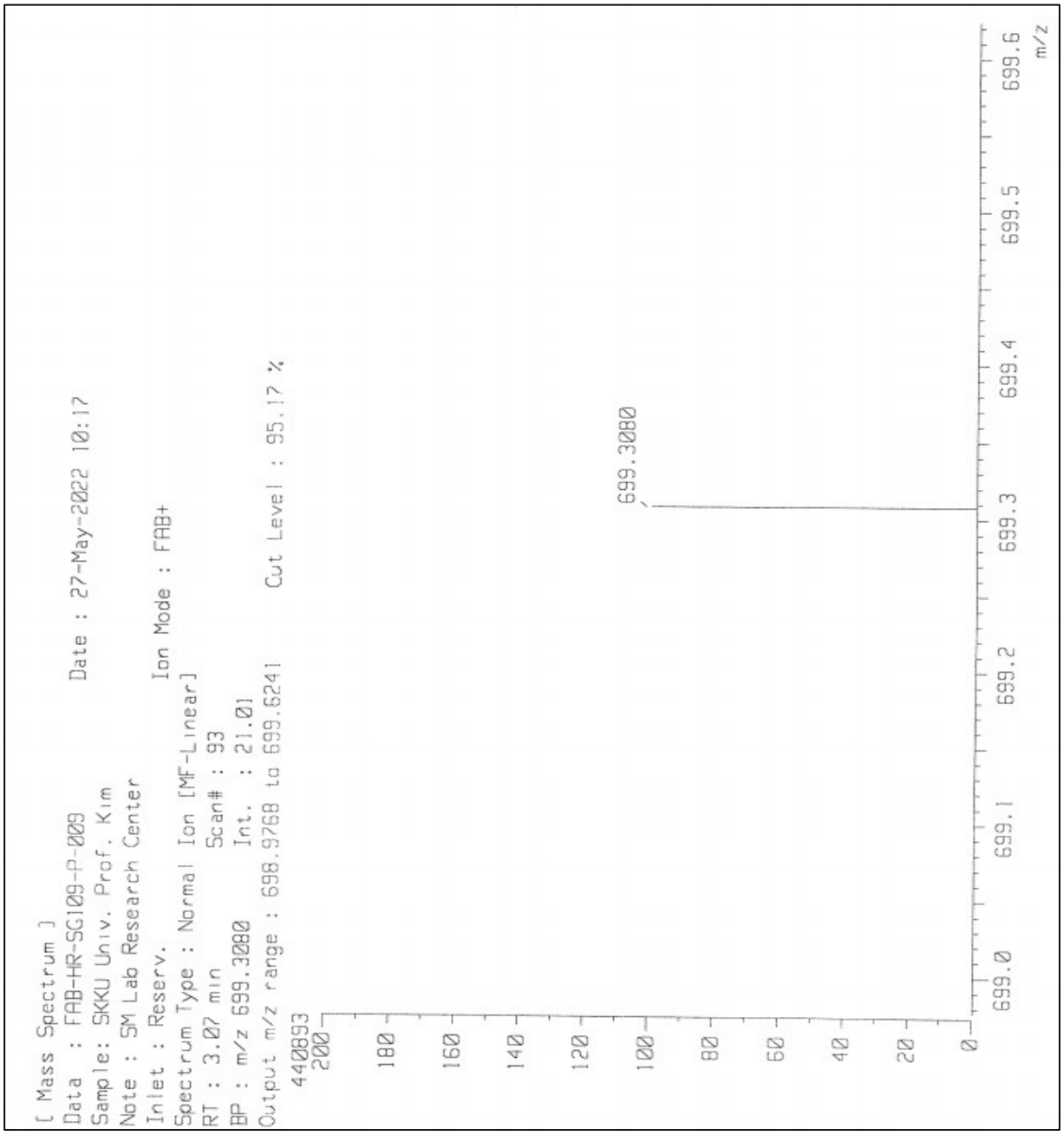
[Elemental Composition] Page: 1

Data : FAB-HR-SG109-P-009 Date : 27-May-2022 10:17

6d

Sample: SKKU Univ. Prof. Kim
Note : SM Lab Research Center
Inlet : Reserv. Ion Mode : FAB+
RT : 3.07 min Scan#: 93
Elements : C 45/0, H 40/0, O 3/0, N 6/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10
Unsaturation (U.S.) : -0.5 - 80.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
699.3080	100.0	-0.5 / -0.4	28.5	C 44 H 39 O 3 N 6

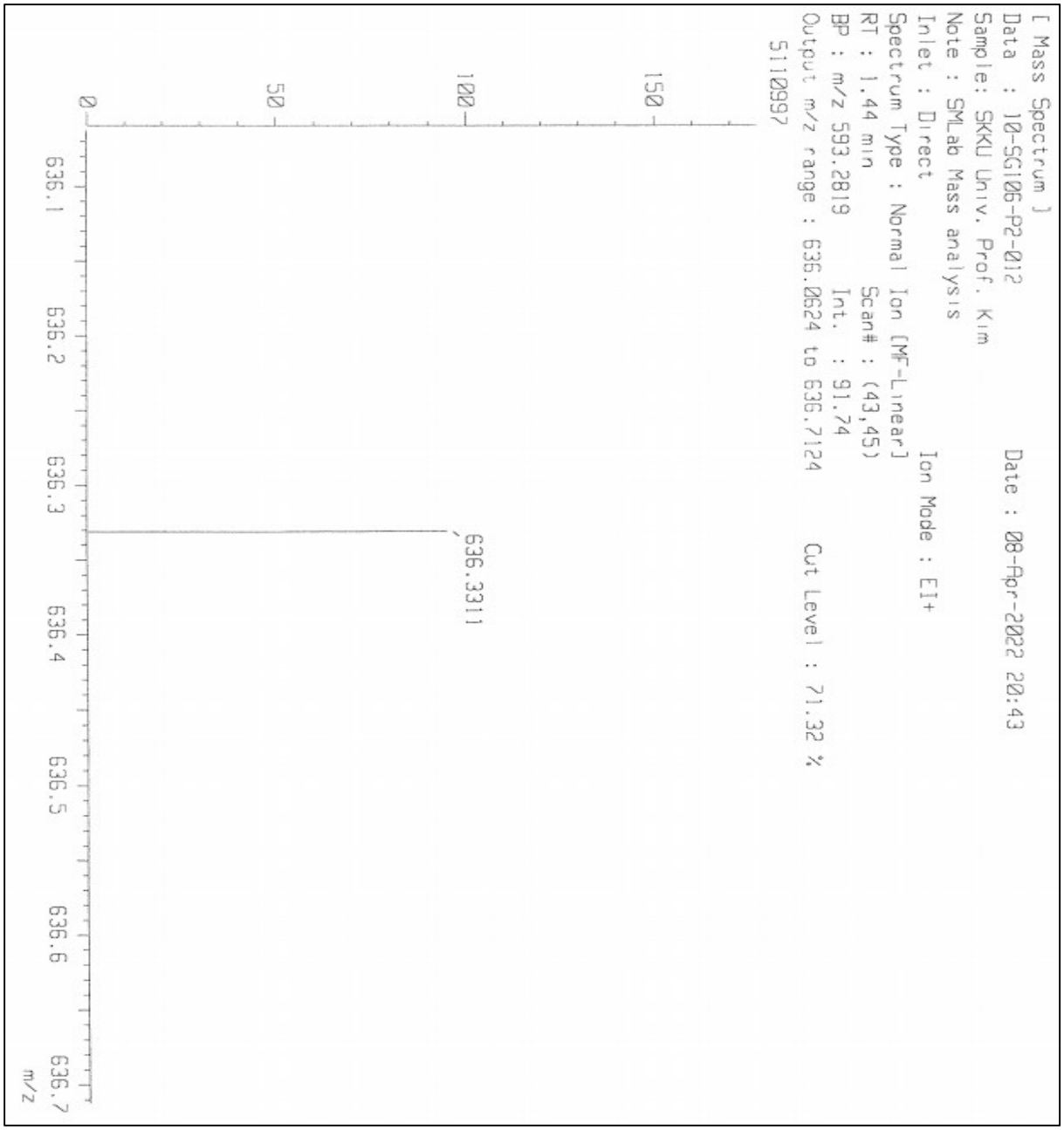


[Elemental Composition]
Data : 10-SG106-P2-012
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct
RT : 1.44 min
Elements : C 40/0, H 45/0, O 5/0, N 5/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Date : 08-Apr-2022 20:43
Ion Mode : EI+
Scan#: (43,45)

6e

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
636.3311	95.1	-0.2 / -0.1	19.0	C 38 H 44 O 5 N 4



[Elemental Composition] Page: 1

Data : 23-MS-282-P-030 Date : 25-May-2022 14:22

Sample: SKKU Univ. Prof. Kim

Note : SMLab Mass analysis

Inlet : Direct Ion Mode : EI+

RT : 1.82 min Scan#: (48,63)

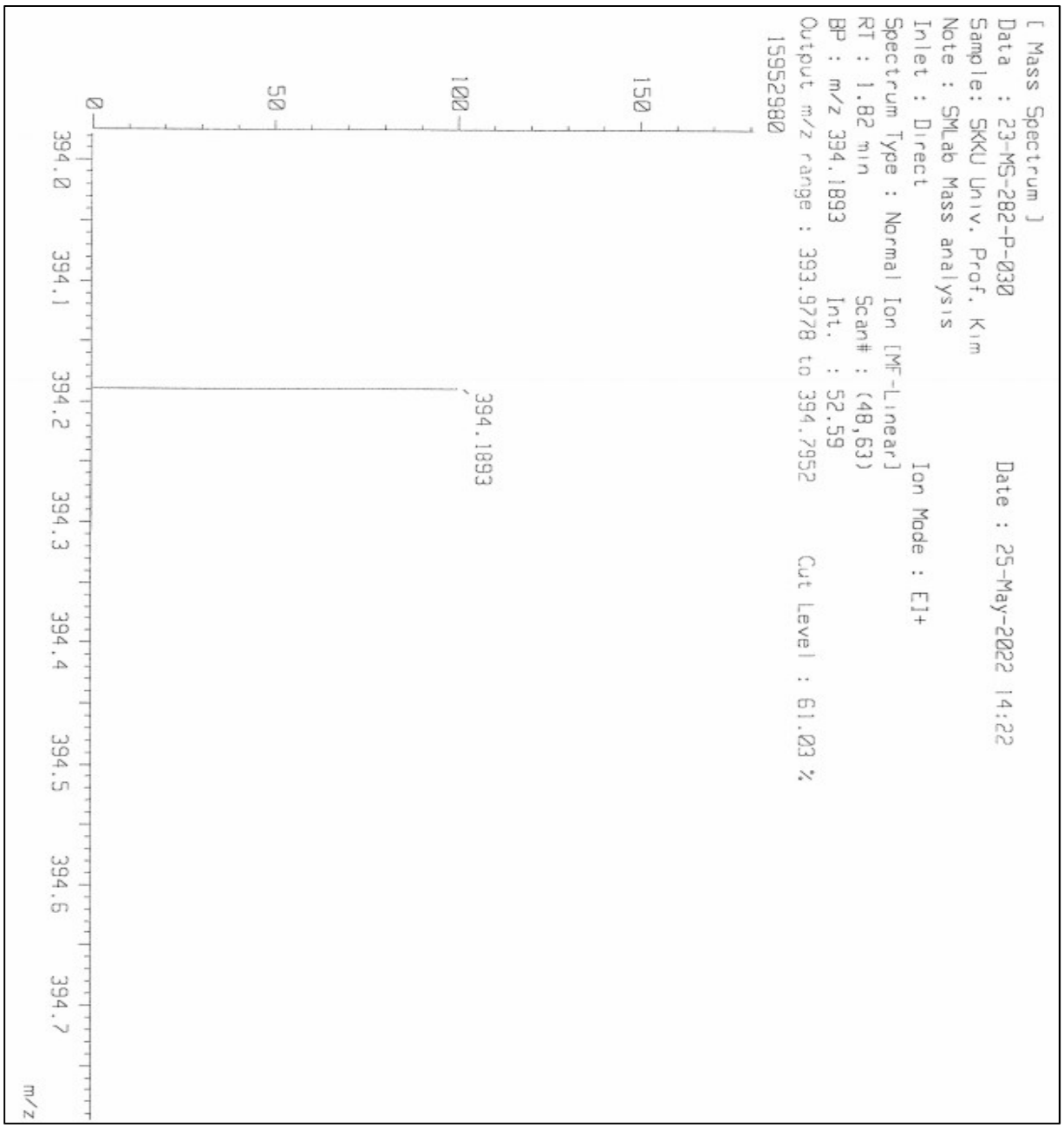
Elements : C 25/0, H 30/0, O 4/0, N 2/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
394.1893	100.0	+0.1 / +0.0	12.0	C 23 H 26 O 4 N 2

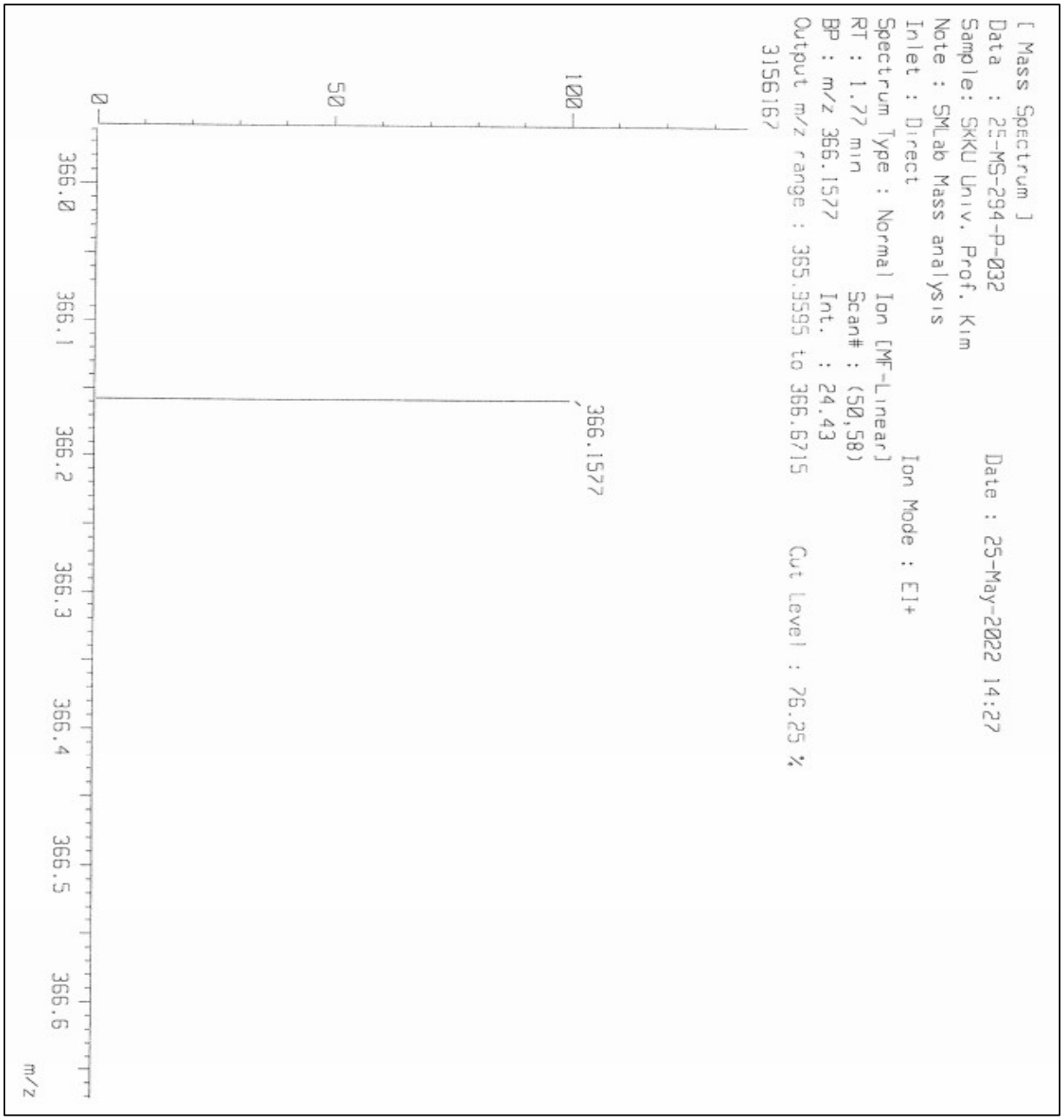
9a



[Elemental Composition]
Data : 25-MS-294-P-032 Date : 25-May-2022 14:27
Sample: SKKU Univ. Prof. Kim
Note : SMLab Mass analysis
Inlet : Direct Ion Mode : EI+
RT : 1.77 min Scan#: (50,58)
Elements : C 23/0, H 25/0, O 4/0, N 2/0
Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 5mmu if m/z > 5
Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
366.1577	100.0	-0.8 / -0.3	12.0	C 21 H 22 O 4 N 2

9b



[Elemental Composition] Page: 1

Data : 4-CE-99-P-004 Date : 31-Oct-2022 16:47

Sample: SKKU. Univ. Prof. Kim

Note : SMLab Mass analysis

9c

Inlet : Direct Ion Mode : EI+

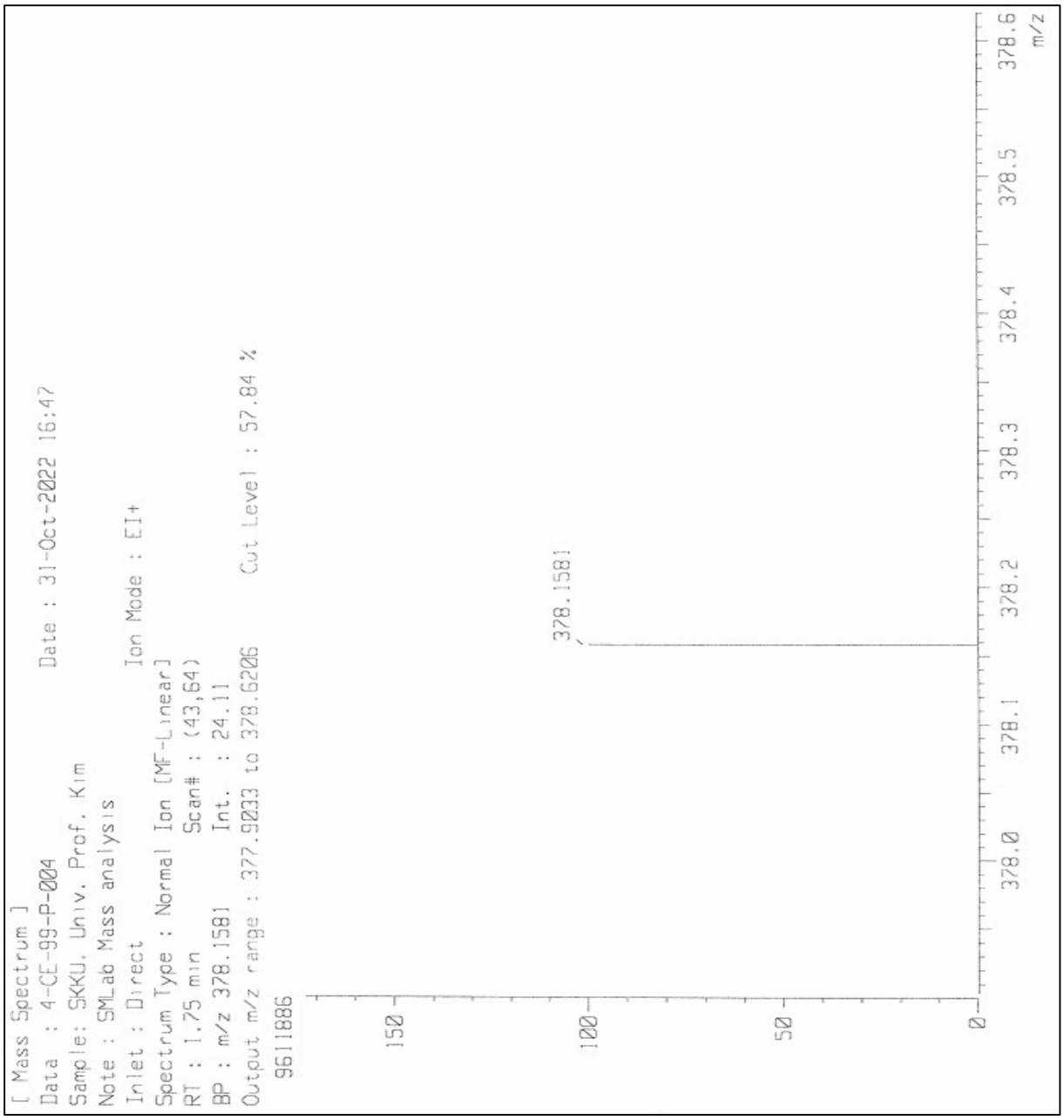
RT : 1.75 min Scan#: (43,64)

Elements : C 22/0, H 23/0, O 4/0, N 3/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 70.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
378.1581	100.0	+0.5 / +0.2	13.0	C 22 H 22 O 4 N 2



[Elemental Composition] Page: 1

Data : FAB-SG-93-P-003 Date : 25-Mar-2022 16:08

Iridacycle-1a

Sample: SKKU Univ. Prof. Kim

Note : SM Lab Research Center

Inlet : Reserv. Ion Mode : FAB+

RT : 3.38 min Scan#: (101,104)+100+104+104

Elements : C 25/0, H 25/0, O 5/0, N 1/0, F 3/0, Ir 1/0

Mass Tolerance : 1000ppm, 3mmu if m/z < 3, 10mmu if m/z > 10

Unsaturation (U.S.) : -0.5 - 80.0

Observed m/z	Int%	Err[ppm / mmu]	U.S.	Composition
641.1362	100.0	-0.4 / -0.3	12.0	C 24 H 25 O 4 N F 3 Ir