SUPPORTING INFORMATION

Intramolecular difunctionalization of alkynes involving cascade radical 1,4-heteroaryl migrations and formal total synthesis of a β-imidazolyl-γ-lactam alkaloid: isoanantine

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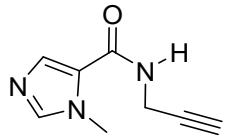
This supplementary material includes:

General procedure A (Preparation of amides)

To a solution of heteroaryl acid **1a**–**1i** (1.0 equiv), coupling reagent (1.5–2 equiv), and DMAP (0.5 equiv) in CH_2Cl_2 (1–20 mL) was added dropwise propargyl amine (1.1 equiv). The reaction mixture was stirred at room temperature for 16-20 h. The resulting mixture was diluted with DCM, washed with saturated $NaHCO_{3\ (aq)}$ and brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated to give a crude product, which was purified by column chromatography (EtOAc/ hexanes = 3/7～pure EtOAc or MeOH/ CH_2Cl_2 = 1/22) to give the desired product **2a**–**2i**.

1-Methyl-*N*-(prop-2-yn-1-yl)-1H-imidazole-5-carboxamide (1a)



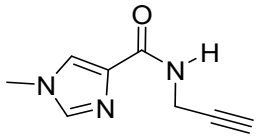
1a

1-Methyl-1H-imidazole-5-carboxylic acid (882.8 mg, 7.0 mmol), EDC (1.5 equiv), DMAP (0.5 equiv), propargyl amine (1.1 equiv) in DMF (7mL) were treated according to method A (with the reaction time of 20 h, and the eluent is MeOH/ CH_2Cl_2 = 1/22) to give **1a** (843.6 mg, 74%), as a white solid.

mp 110.8–112.3 °C; IR (neat) 3282 (NH), 3230, 3281, 3113, 3111, 3038, 1655 (C=O), 1557, 1520, 1496, 1468, 1467, 1419, 1413, 1382, 1360, 1307, 1261, 1235, 1161, 1134, 1048, 989, 931, 837, 834, 800, 761 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.60 (br s, 1H, amide N-H), 7.55 (s, 1H), 7.50 (s, 1H), 4.14 (dd, J = 5.4, 2.4 Hz, 2H), 3.90 (s, 3H, NCH_3), 2.22 (t, J = 2.3 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2 (C), 141.7 (CH), 132.1 (CH), 125.9 (C), 79.7 (C, C≡C-H), 71.4 (CH, C≡C-H), 34.1 (CH_3), 28.7

(CH_2); HRMS (ESI^+): m/z calcd. for $C_8H_9N_3O$ $[M+H]^+$: 164.0824. Found: 164.0825.

1-Methyl-*N*-(prop-2-yn-1-yl)-1H-imidazole-4-carboxamide (1b)

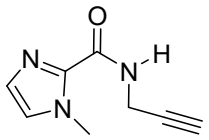


1b

1-Methyl-1H-imidazole-4-carboxylic acid (504.4 mg, 4.0 mmol), EDC (2 equiv), HOBt (0.1 equiv), propargyl amine (1.1 equiv) in DMF (10 mL) were treated according to method A (with the reaction time of 18 h, and the eluent is EtOAc + 0.1% Et_3N) to give **1b** (480.4 mg, 74%), as a white solid.

mp 155.7–157.1 °C; IR (neat) 3381 (NH), 3213, 3149, 3121, 2951, 2114, 1641 (C=O), 1572, 1529, 1493, 1435, 1413, 1361, 1341, 1306, 1251, 1233, 1149, 1052, 955, 922, 845, 796, 774, 749, 706 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, J = 1.5 Hz, 1H), 7.35 (d, J = 0.9 Hz, 1H), 7.31-7.21 (br s, 1H, amide N-H), 4.18 (dd, J = 5.7, 2.7 Hz, 2H), 3.71 (s, 3H), 2.21 (t, J = 2.3 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.1 (C), 137.4 (CH), 137.0 (C), 123.2 (CH), 79.7 (C, C≡C-H), 71.3 (CH, C≡C-H), 33.8 (CH_3), 28.6 (CH_2); HRMS (ESI^+): m/z calcd. for $C_8H_9N_3O$ $[M+H]^+$: 164.0824. Found: 164.0820.

1-Methyl-*N*-(prop-2-yn-1-yl)-1H-imidazole-2-carboxamide (1c)

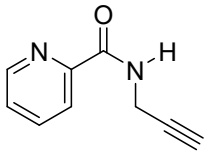


1c

1-Methyl-1H-imidazole-2-carboxylic acid (126.1 mg, 1.0 mmol), DIC (2 equiv), HOBt (0.1 equiv), propargyl amine (1.1 equiv) in DMF (4 mL) were treated according to method A (with the reaction time of 16 h, and the eluent is EtOAc/ hexanes = 1/1 + 0.1% Et_3N) to give **1c** (56.5 mg, 35%), as a white solid.

mp 137.4–141.4 °C; IR (neat) 3341 (NH), 3282, 3115, 2974, 1668 (C=O), 1543, 1506, 1476, 1422, 1354, 1292, 1277, 1153, 1091, 993, 926, 915, 789 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.78 (br s, 1H, amide N-H), 7.00 (d, J = 0.6 Hz, 1H), 6.95 (s, 1H), 4.14 (dd, J = 5.7, 2.7 Hz, 2H), 4.03 (s, 3H), 2.22 (t, J = 2.6 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.9 (C), 138.6 (C), 127.8 (CH), 125.6 (CH), 79.3 (C, C≡C-H), 71.5 (CH, C≡C-H), 35.6 (CH_3), 28.6 (CH_2); HRMS (ESI^+): m/z calcd. for $C_8H_9N_3O$ $[M+H]^+$: 164.0824. Found: 164.0820.

N-(Prop-2-yn-1-yl)picolinamide (1d)



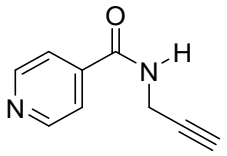
1d

Picolinic acid (246.2 mg, 2.0 mmol), EDC (1.5 equiv), DMAP (0.1 equiv), propargyl amine (1.1 equiv) in THF (20 mL) were treated according to method A (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 3/7) to give **1d** (169.3 mg, 53%), as a white solid.

mp 78.5–79.8 °C; IR (neat) 3393 (NH), 3341, 3302, 30578, 2968, 2917, 2115, 1668

(C=O), 1589, 1570, 1519, 1465, 1434, 1352, 1289, 1265, 1239, 1163, 1115, 1088, 1051, 1002, 918, 819, 738 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.50 (d, *J* = 4.8 Hz, 1H), 8.24 (br s, 1H, amide N-H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.79 (td, *J* = 7.8, 1.5 Hz, 1H), 7.38 (ddd, *J* = 7.5, 4.8, 0.9 Hz, 1H), 4.22 (dd, *J* = 5.7, 2.7 Hz, 2H), 2.24 (t, *J* = 2.6 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.0 (C), 149.4 (C), 148.2 (CH), 137.4 (CH), 126.4 (CH), 122.3 (CH), 79.4 (C, C≡C-H), 71.6 (CH, C≡C-H), 29.1 (CH_2); HRMS (ESI^+): m/z calcd. for $C_9H_8N_2O$ $[M+H]^+$: 161.0715. Found: 161.0714.

_N_-(Prop-2-yn-1-yl)isonicotinamide (1e)

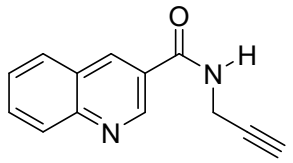


1e

Isonicotinic acid (123.11 mg, 1.0 mmol), EDC (2 equiv), DMAP (0.5 equiv), propargyl amine (1.1 equiv) in DCM (5 mL) were treated according to method A (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 75/25) to give **1e** (125.11 mg, 78%), as a white solid.

mp 142.0–145.1 °C; IR (neat) 3325 (NH), 1648 (C=O), 1556, 1538, 1488, 1417, 1359, 1311, 1261, 1091, 991, 923, 848, 753 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.77 (dd, *J* = 4.5, 1.5 Hz, 2H), 7.64 (dd, *J* = 4.5, 1.5 Hz, 2H), 6.51 (br s, 1H, amide N-H), 4.27 (dd, *J* = 5.3, 2.6 Hz, 2H), 2.32 (t, *J* = 2.4 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.2 (C), 150.6 (CH×2), 140.9 (C), 121.0 (CH×2), 78.8 (C, C≡C-H), 72.4 (CH, C≡C-H), 29.9 (CH_2); HRMS (ESI^+): m/z calcd. for $C_9H_8N_2O$ $[M+H]^+$: 161.0715. Found: 161.0712.

_N_-(Prop-2-yn-1-yl)quinoline-3-carboxamide (1g)

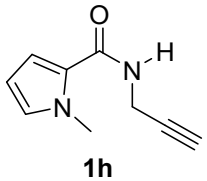


1g

3-Quinoline carboxylic acid (519.5 mg, 3.0 mmol), EDC (1.5 equiv), DMAP (0.5 equiv), propargyl amine (1.1 equiv) in DCM (12 mL) were treated according to method A (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 6/4, 75/25 and 9/1) to give **1g** (563.8 mg, 89%), as a white solid.

mp 120.2–123.5 °C; IR (neat) 3467 (NH), 3307, 3228, 3055, 2929, 2556, 2406, 2114, 1985, 1675 (C=O), 1636, 1619, 1601, 1547, 1504, 1473, 1431, 1410, 1383, 1343, 1304, 1261, 1230, 1207, 1154, 1129, 1045, 1019, 1009, 969, 932, 875, 866, 790, 766, 753, 726, 713 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.30 (s, 1H), 8.64 (s, 1H), 8.17 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.82 (t, *J* = 7.7 Hz, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 6.68 (br s, 1H, amide N-H), 4.34 (dd, *J* = 5.1, 2.7 Hz, 2H), 2.33 (t, *J* = 2.6 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, CD_3OD) δ 166.1 (C), 148.4 (C), 148.3 (CH), 136.4 (CH), 131.6 (CH), 129.0 (CH), 127.6 (CH), 127.0 (C), 126.7 (C), 79.1 (C, C≡C-H), 71.0 (CH, C≡C-H), 28.7 (CH_2); HRMS (ESI^+): m/z calcd. for $C_{13}H_{11}N_2O$ $[M+H]^+$: 211.0871. Found: 211.0874.

1-Methyl-_N_-(prop-2-yn-1-yl)-1H-pyrrole-2-carboxamide (1h)

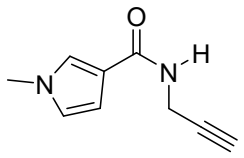


1h

1-Methyl-1H-pyrrole-2-carboxylic acid (250.3 mg, 2.0 mmol), EDC (1.5 equiv), DMAP (0.5 equiv), propargyl amine (1.1 equiv) were treated according to method A (with the reaction time of 17 h, and the eluent is EtOAc/ hexanes = 35/65) to give **1h** (286.5 mg, 88%), as a white solid.

mp 120.8–123.9 °C; IR (neat) 3255 (NH), 3113, 2939, 1635 (C=O), 1543, 1516, 1480, 1464, 1441, 1411, 1387, 1350, 1326, 1272, 1261, 1158, 1095, 1070, 980, 913, 876, 819, 793, 760, 746, 730 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.72 (t, *J* = 1.8 Hz, 1H), 6.58 (dd, *J* = 4.1, 1.7 Hz, 1H), 6.20-6.06 (br s, amide N-H overlapped with one dd at 6.07, *J* = 4.1, 2.6 Hz, 2H), 4.15 (t, *J* = 2.1 Hz, 2H), 3.93 (s, 3H), 2.24 (t, *J* = 2.6 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.5 (C), 128.3 (CH), 125.0 (C), 112.1 (CH), 107.3 (CH), 79.9 (C, C≡C-H), 71.4 (CH, C≡C-H), 36.8 (CH_3), 28.9 (CH_2); HRMS (ESI^+): m/z calcd. for $C_9H_{10}N_2O$ $[M+H]^+$: 163.0871. Found: 163.0866.

1-Methyl-*N*-(prop-2-yn-1-yl)-1H-pyrrole-3-carboxamide (1i)



1i

1-Methyl-1H-pyrrole-3-carboxylic acid (125.1 mg, 1.0 mmol), EDC (1.5 equiv), DMAP (0.5 equiv), propargyl amine (1.1 equiv) in DCM (5 mL) were treated according to method A (with the reaction time of 16 h, and the eluent is EtOAc/ hexanes = 7/3) to give **1i** (139.7 mg, 86%), as a white solid.

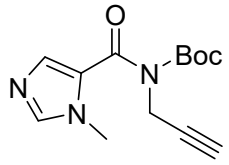
mp 107.9–109.5 °C; IR (neat) 3287 (NH), 2929, 1626 (C=O), 1557, 1523, 1507, 1420, 1390, 1341, 1270, 1238, 1152, 1070, 1011, 942, 912, 854, 815, 790, 761, 720 cm^{-1}; ^1H

NMR (300 MHz, $CDCl_3$) δ 7.19 (d, *J* = 1.5 Hz, 1H), 6.55 (t, *J* = 2.6 Hz, 1H), 6.37 (dd, *J* = 2.6, 2.0 Hz, 1H), 6.07 (br s, 1H), 4.18 (dd, *J* = 5.3, 2.6 Hz, 2H), 3.65 (d, *J* = 1.8 Hz, 3H), 2.23 (t, *J* = 2.6 Hz, 1H, C≡C-H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.3 (C), 124.9 (CH), 122.7(CH), 119.1 (C), 107.2 (CH), 80.3 (C, C≡C-H), 71.3 (CH, C≡C-H), 36.5 (CH_3), 29.0 (CH_2); HRMS (ESI^+): m/z calcd. for $C_9H_{10}N_2O$ $[M+H]^+$: 163.0871. Found: 163.0872.

General procedure B (Preparation of radical precursors)

To a solution of amide **1a**–**1i** (1 equiv) and DMAP (0.2 equiv) in CH_2Cl_2 (1–20 mL) was added Et_3N (1.5–2 equiv) and $(Boc)_2O$ (1.5–2 equiv). The reaction mixture was stirred at room temperature for 16–18 h. The resulting mixture was diluted with DCM, washed with saturated $NaHCO_{3\,(aq)}$ and brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated to give a crude product, which was purified by column chromatography (EtOAc/ hexanes = 15/85–9/1) to give the desired product **2a**–**2i**.

***tert*-Butyl (1-methyl-1H-imidazole-5-carbonyl)(prop-2-yn-1-yl)carbamate (2a)**



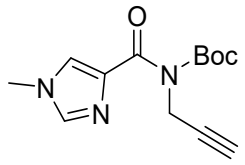
2a

Compound **1a** (0.78 g, 4.77 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 8/2) to give **2a** (1.05 g, 84%), as a white solid.

mp 175.8–177.1 °C; IR (neat) 3282, 3120, 2981, 2123, 1733 (C=O), 1674 (C=O), 1537, 1492, 1477, 1420, 1394, 1370, 1355, 1351, 1334, 1273, 1242, 1240, 1223, 1148, 976,

942, 922, 898, 849, 798, 766, 751 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.81 (br s, 1H), 7.50 (s, 1H), 4.47 (d, *J* = 2.1 Hz, 2H), 4.01 (s, 3H), 2.27 (t, *J* = 2.4 Hz, 1H), 1.45 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.9 (C), 152.5 (C), 141.8 (CH), 136.3 (CH), 127.6 (C), 83.9 (C), 78.9 (C, C≡C-H), 71.2 (C, C≡C-H), 35.0 (CH_2), 33.5 (CH_3), 27.7 (CH_3 ×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+H]^+$: 264.1348. Found 264.1352.

tert-Butyl (1-methyl-1H-imidazole-4-carbonyl)(prop-2-yn-1-yl) carbamate (2b)

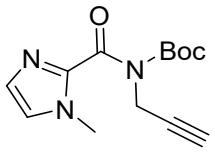


2b

Compound **1b** (0.78 g, 4.77 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 17 h, and the eluent is EtOAc/ hexanes = 8/2) to give **2b** (269.5 g, 83%), as a white solid.

mp 124.2–127.1 °C; IR (neat) 3444, 3265, 2981, 2124, 1726 (C=O), 1652 (C=O), 1548, 1476, 1458, 1425, 1395, 1370, 1334, 1290, 1259, 1216, 1152, 1050, 1020, 941, 896, 850, 798, 767 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45 (d, *J* = 1.2 Hz, 1H), 7.354 (s, 1H), 4.45 (d, *J* = 2.4 Hz, 2H), 3.69 (s, 3H), 2.17 (t, *J* = 2.7 Hz, 1H, C≡C-H), 1.37 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5 (C), 152.7 (C), 138.2 (C), 137.2 (CH), 125.0 (CH), 83.1 (C), 79.4 (CH, C≡C-H), 70.8 (C, C≡C-H), 35.0 (CH_2), 33.8 (CH_3), 27.6 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+Na]^+$: 286.1168. Found 286.1161.

tert-Butyl (1-methyl-1H-imidazole-2-carbonyl)(prop-2-yn-1-yl) carbamate (2c)

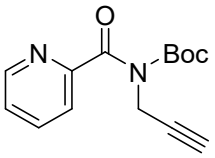


2c

Compound **1c** (0.14 g, 0.84 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (2 equiv), and Et_3N (2 equiv) were treated according to method B (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 35/65) to give **2c** (0.16 g, 72%), as a white solid.

mp 103.4–105.2 °C; IR (neat) 3279, 2979, 1736 (C=O), 1676 (C=O), 1476, 1432, 1369, 1341, 1317, 1285, 1235, 1150, 982, 942, 920, 899, 848, 770 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.01 (d, *J* = 0.9 Hz, 1H), 6.93 (s, 1H), 4.45 (d, *J* = 2.4 Hz, 2H), 3.81 (s, 3H), 2.20 (t, *J* = 2.4 Hz, 1H, C≡C-H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.9 (C), 152.1 (C), 140.9 (C), 128.4 (CH), 124.3 (CH), 83.6 (C), 78.6 (CH, C≡C-H), 71.2 (C, C≡C-H), 34.7 (CH_2), 34.2 (CH_3), 27.5 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+H]^+$: 264.1348. Found 264.1342.

***tert*-Butyl picolinoyl(prop-2-yn-1-yl)carbamate (2d)**



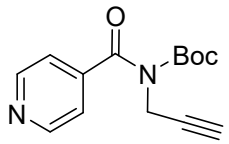
2d

Compound **1d** (0.33 g, 2.05 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 16 h, and the eluent is EtOAc/ hexanes = 2/8) to give **2d** (0.17 g, 90%), as a white solid.

mp 47.1–51.0 °C; IR (neat) 3278, 3057, 2982, 2936, 1746 (C=O), 1683 (C=O), 1588, 1570, 1469, 1458, 1439, 1418, 1380, 1370, 1346, 1291, 1256, 1236, 1153, 1093, 1047, 996, 984, 943, 907, 849, 818, 775, 749, 727 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.54 (d, *J* = 4.8 Hz, 1H), 7.78 (td, *J* = 7.5, 1.5 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.37 (ddd,

J = 7.5, 4.8, 1.1 Hz, 1H), 4.56 (d, *J* = 2.4 Hz, 2H), 2.22 (t, *J* = 2.4 Hz, 1H, C≡C–H), 1.18 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7 (C), 154.1 (C), 152.0 (C), 148.3 (CH), 136.9 (CH), 125.4 (CH), 122.9 (CH), 83.8 (C, O-C(CH_3)$_3$), 78.7 (C, C≡C–H), 71.1 (CH, C≡C–H), 34.7 (CH_2), 27.4 (CH_3×3); HRMS (ESI$^+$): m/z calcd. for $C_{14}H_{15}N_2O_3$ $[M+Na]^+$: 283.1059. Found 283.1052.

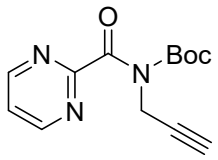
tert-Butyl isonicotinoyl(prop-2-yn-1-yl)carbamate (2e)



2e

Compound **1e** (0.13 g, 0.78 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 16 h, and the eluent is EtOAc/ hexanes = 35/65) to give **2e** (0.18 g, 89%), as a white solid.
mp 106.8–109.7°C; IR (neat) 3411, 3200, 2984, 2937, 2118, 1739 (C=O), 1681 (C=O), 1600, 1558, 1494, 1475, 1456, 1412, 1395, 1380, 1370, 1340, 1322, 1238, 1212, 1149, 1090, 1066, 982, 964, 946, 906, 845, 808, 769, 750, 727 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.64 (dd, *J* = 4.8, 1.4 Hz, 2H), 7.30 (dd, *J* = 4.5, 1.8 Hz, 2H), 4.50 (d, *J* = 2.4 Hz, 2H), 2.22 (t, *J* = 2.4 Hz, 1H), 1.17 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.2 (C), 151.4 (C), 149.9 (CH×2), 144.7 (C), 120.8 (CH×2), 84.9 (C), 78.5 (C, C≡C-H), 71.4 (C, C≡C-H), 34.5 (CH_2), 27.4 (CH_3×3); HRMS (ESI$^+$): m/z calcd. for $C_{14}H_{16}N_2O_3$ $[M+H]^+$: 261.1239. Found 261.1234.

tert-Butylprop-2-yn-1-yl(pyrimidine-2-carbonyl)carbamate (2f)



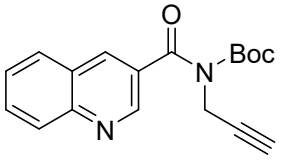
2f

Pyrimidine-2-carboxylic acid (744.6 mg, 6.0 mmol), DIC (2 equiv), HOBt (2 equiv), propargyl amine (1.1 equiv) in DMF (20 mL) were treated according to method A (with the reaction time of 17 h, and the eluent is MeOH/ CH_2Cl_2 = 1/20 + 0.1 %NEt_3) to give **1f** (532.5 mg, 55%, crude), as a white solid, which was directly used for the next step without further purification.

Compound **1f** (0.33 g, 2.05 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 4/6) to give **2f** (0.14 g, 25%), as a white solid.

mp 79.8–81.3°C; IR (neat) 3280, 2983, 1744 (C=O), 1693 (C=O), 1565, 1478, 1440, 1419, 1371, 1349, 1275, 1252, 1190, 1153, 1004, 985, 945, 908, 834, 811, 749, 709 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.79 (d, *J* = 4.8 Hz, 2H), 7.36 (t, *J* = 5.0 Hz 1H), 4.62 (d, *J* = 2.4 Hz, 2H), 2.24 (t, *J* = 2.6 Hz, 1H, C≡C–H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1 (C), 162.5 (C), 157.1 (CH×2), 151.2 (C), 121.3 (CH), 84.5 (C, O-C$(CH_3)_3$), 78.3 (C, C≡C–H), 71.3 (CH, C≡C–H), 33.7 (CH_2), 27.6 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{15}N_3O_3$ $[M+H]^+$: 262.1192. Found 262.1187.

tert-Butyl prop-2-yn-1-yl(quinoline-3-carbonyl)carbamate (2g)

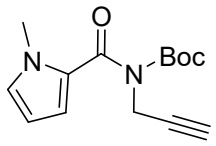


2g

Compound **1g** (0.25 g, 1.2 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.75 equiv) were treated according to method B (with the reaction time of 17 h, and

the eluent is EtOAc/ hexanes = 35/65) to give **2g** (0.35 g, 94%), as a white solid.
mp 120.2–123.5 °C; IR (neat) 3290, 2980, 1737 (C=O), 1678 (C=O), 1620, 1601, 1571, 1497, 1460, 1421, 1370, 1322, 1261, 1225, 1199, 1149, 1023, 946, 847, 789, 763 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.96 (d, *J* = 2.1 Hz, 1H), 8.38 (d, *J* = 1.8 Hz, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.86 (d, *J* = 8.1 Hz, 1H), 7.77 (t, *J* = 7.7 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 1H), 4.61 (d, *J* = 2.4 Hz, 2H), 2.27 (t, *J* = 2.4 Hz, 1H, C≡C–H), 1.19 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1 (C), 152.0 (C), 148.5 (C), 148.3 (CH), 136.1 (CH), 131.4 (C), 130.0 (C), 129.3 (CH), 128.6 (CH), 127.6 (CH), 126.8 (C), 84.7 (C, O-C(CH_3)$_3$), 78.8 (C, C≡C–H), 71.3 (CH, C≡C–H), 35.0 (CH_2), 27.5 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{18}H_{18}N_2O_3$ $[M+H]^+$: 311.1396. Found 311.1396.

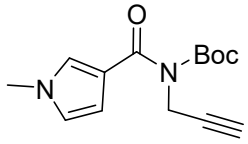
***tert*-Butyl(1-methyl-1H-pyrrole-2-carbonyl)(prop-2-yn-1-yl)carbamate (2h)**



2h

Compound **1h** (0.27 g, 1.67 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.6 equiv) were treated according to method B (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 15/85) to give **2h** (0.22 g, 85%), as a colorless liquid.
IR (neat) 3287, 2929, 1626 (C=O), 1557, 1523, 1507, 1420, 1390, 1341, 1271, 1238, 1152, 1070, 1011, 942, 911, 854, 815, 790, 761, 720 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.75 (d, *J* = 1.5 Hz, 1H), 6.57 (dd, *J* = 3.9, 1.8 Hz, 1H), 6.05 (dd, *J* = 2.6, 1.4 Hz, 1H), 4.37 (d, *J* = 2.1 Hz, 2H), 3.80 (s, 3H), 2.20 (t, *J* = 2.4 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.2 (C), 153.1 (C), 129.4 (CH), 127.6 (C), 117.8 (CH), 107.9 (CH), 82.7 (C), 79.3 (C, C≡C-H), 71.0 (C, C≡C-H), 36.0 (CH_3), 35.0 (CH_2), 27.7 (CH_3 ×3); HRMS (ESI^+): m/z calcd. for $C_{14}H_{18}N_2O_3$ $[M+Na]^+$: 285.1215. Found 285.1212.

***tert*-Butyl(1-methyl-1H-pyrrole-3-carbonyl)(prop-2-yn-1-yl) carbamate (2i)**



2i

Compound **1i** (0.11 g, 0.69 mmol), DMAP (0.2 equiv), $(Boc)_2O$ (1.5 equiv), and Et_3N (1.5 equiv) were treated according to method B (with the reaction time of 18 h, and the eluent is EtOAc/ hexanes = 25/75) to give **2i** (0.15 g, 85%), as a colorless liquid.
IR (neat) 3284, 2979, 1721 (C=O), 1666 (C=O), 1535, 1478, 1423, 1394, 1369, 1347, 1330, 1304, 1259, 1241, 1207, 1149, 1080, 1015, 939, 887, 852, 763, 750 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.16 (s, 1H), 6.49 (t, *J* = 2.6 Hz, 1H), 6.36 (dd, *J* = 2.4, 1.7 Hz, 1H), 4.41 (d, *J* = 2.4 Hz, 2H), 3.63 (s, 3H), 2.17 (t, *J* = 2.3 Hz, 1H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6 (C), 153.1 (C), 127.4 (CH), 122.1 (CH), 120.3 (C), 110.6 (CH), 82.8 (C), 79.9 (C, C≡C-H), 70.5 (C, C≡C-H), 36.6 (CH_3), 35.2 (CH_2), 27.8 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{14}H_{18}N_2O_3$ $[M+Na]^+$: 285.1215. Found 285.1219.

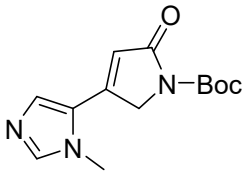
General procedure C (Synthesis of γ-lactams using Bu_3SnH)

To a refluxed solution of compound **2a**–**2h** (1.0 equiv) in toluene (10 mL) at 110 °C (an oil bath) was dropwise added a solution of AIBN (0.6 equiv) and Bu_3SnH (0.6–1.5 equiv) in toluene (6–10 mL) over 2–6 h via a syringe pump. The reaction solution was stirred at the same temperature for another 2–18 h. The reaction mixture was concentrated to give a crude product, which was purified by flash chromatography (10 wt % KF in silica gel) to give the desired compound **3a–3h**.

General procedure D (Synthesis of γ-lactams using TTMSS)

To a refluxed solution of compound **2a**, **2h** (1.0 equiv) in toluene (10 mL) at 110 °C (an oil bath) was dropwise added a solution of AIBN (0.6 equiv) and TTMSS (1.0–1.5 equiv) in toluene (6–10 mL) over 2–6 h via a syringe pump. The reaction solution was stirred at the same temperature for another 2–18 h. The reaction mixture was concentrated to give a crude product, which was purified by flash chromatography to give the desired compound **3a**, **3h**.

***tert*-Butyl 4-(1-methyl-1H-imidazol-5-yl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxylate (3a)**



3a

Bu_3SnH as the reagent

Compound **2a** (96.40 mg, 0.37 mmol), AIBN (0.5 equiv) and Bu_3SnH (0.6 equiv) was treated according to method C (with the reaction time of 4 h, and the eluent MeOH/EtOAc = 1/7 was used) to give **3a** (69.80 mg, 72%), as a white solid.

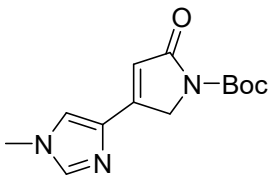
TTMSS as the reagent

Compound **2a** (0.07 g, 0.25 mmol), AIBN (0.6 equiv) and TTMSS (1.1 equiv) was treated according to method D (with the reaction time of 4 h, and the eluent MeOH/EtOAc = 1/7 was used) to give **3a** (0.03 g, 46%), as a white solid.

mp 172.7–176.3 °C; IR (neat) 3111, 2979, 2981, 1767 (C=O), 1724 (C=O), 1627, 1561, 1534, 1452, 1394, 1359, 1308, 1285, 1262, 1235, 1157, 1134, 1095, 1079, 928, 838, 761, 749 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.41 (s, 1H), 6.14 (s, 1H, C=C–H), 4.65 (d, J = 1.2 Hz, 2H, NCH_2), 3.81 (s, 3H, NCH_3), 1.58 (s, 9H); ^{13}C NMR

(75 MHz, $CDCl_3$) δ 169.0 (C), 149.4 (C), 144.1 (C), 142.9 (CH), 133.1 (CH), 125.1 (C), 116.0 (CH), 83.3 (C), 51.3 (CH_2), 34.1 (CH_3), 28.1 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+H]^+$: 264.1348. Found 264.1354.

tert-Butyl-4-(1-methyl-1H-imidazol-4-yl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxylate (3b)

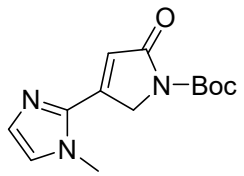


3b

Compound **2b** (52.86 mg, 0.20 mmol), AIBN (0.5 equiv) and Bu_3SnH (0.8 equiv) was treated according to method C (with the reaction time of 1 h, and the eluent MeOH/EtOAc = 1/7 + 0.1 % NEt_3 was used) to give **3b** (16 mg, 30%, crude), as a white solid.

mp 137.0–140.9 °C; IR (neat) 3351, 1761 (C=O), 1723 (C=O), 1667, 1637, 1544, 1434, 1371, 1344, 1299, 1258, 1163, 1082, 834, 772 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.28 (s, 1H), 6.22 (s, 1H, C=C–H), 4.64 (d, J = 0.9 Hz, 2H, NCH_2), 3.74 (s, 3H), 1.54 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1 (C), 150.8 (C), 149.7 (C), 139.2 (CH), 135.1 (C), 120.8 (CH), 116.8 (CH), 82.6 (C), 50.9 (CH_2), 33.8 (CH_3), 28.2 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+H]^+$: 264.1348. Found 264.1353.

tert-Butyl 4-(1-methyl-1H-imidazol-2-yl)-2-oxo-2,5-dihydro-1H-pyrrole-1-carboxylate (3c)

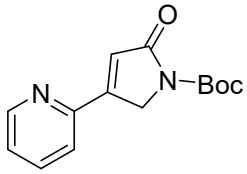


3c

Compound **2c** (0.05 g, 0.20 mmol), AIBN (0.5 equiv) and Bu_3SnH (0.6 equiv) was treated according to method C (with the reaction time of 2 h, and the eluent is EtOAc for chromatography was used) to give **3c** (0.02 g, 37%), as a white solid.

mp 169.1–174.6 °C; IR (neat) 3276, 3116, 2979, 2934, 1767 (C=O), 1735 (C=O), 1677, 1626, 1538, 1536, 1476, 1464, 1460, 1421, 1394, 1368, 1357, 1316, 1285, 1260, 1223, 1150, 1083, 1082, 1057, 976, 942, 924, 898, 848, 737, 707 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.17 (s, 1H), 7.07 (s, 1H), 6.25 (s, 1H, C=C–H), 4.82 (s, 2H, NCH_2), 3.84 (s, 3H), 1.54 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.5 (C), 149.1 (C), 145.3 (C), 139.6 (C), 130.0 (CH), 125.9 (CH), 119.2 (CH), 83.1 (C), 52.2 (CH_2), 35.4 (CH_3), 28.1 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{17}N_3O_3$ $[M+H]^+$: 264.1348. Found 264.1348.

tert-Butyl-2-oxo-4-(pyridin-2-yl)-2,5-dihydro-1H-pyrrole-1-carboxylate (3d)



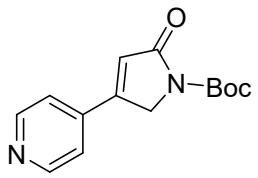
3d

Compound **2d** (57.20 mg, 0.20 mmol), AIBN (0.5 equiv) and Bu_3SnH (1.25 equiv) was treated according to method C (with the reaction time of 6h, and the eluent EtOAc/Hexanes = 55/45 was used) to give **3d** (15.60 mg, 27%), as a yellow solid.

mp 97.4–99.0 °C; IR (neat) 2980, 2933, 2932, 1771 (C=O), 1729 (C=O), 1625, 1587, 1568, 1468, 1440, 1394, 1369, 1340, 1258, 1157, 1087, 992, 877, 874, 850, 786, 749

cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67 (dd, *J* = 4.8, 0.6 Hz, 1H), 7.79 (td, *J* = 7.8, 1.8 Hz, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.34 (ddd, *J* = 7.2, 4.5, 1.1 Hz, 1H), 6.61 (s, 1H, C=C–H), 4.82 (d, *J* = 1.5 Hz, 2H), 1.58 (s, 9H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.4 (C), 156.4 (C), 150.1 (CH), 149.5 (C), 136.8 (CH), 125.0 (CH), 122.6 (CH), 121.9 (CH), 83.0 (C, O-$C(CH_3)_3$), 77.2 (C), 51.2 (CH_2), 28.2 (CH_3×3); HRMS (ESI$^+$): m/z calcd. for $C_{14}H_{16}N_2O_3$ $[M+H]^+$: 261.1239. Found 261.1240.

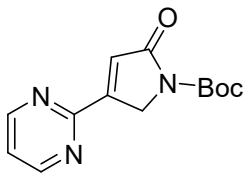
***tert*-Butyl- 2-oxo-4-(pyridin-4-yl)-2,5-dihydro-1H-pyrrole-1-carboxylate (3e)**



3e

Compound **2e** (0.05 g, 0.20 mmol), AIBN (0.6 equiv) and Bu_3SnH (1.2 equiv) was treated according to method C (with the reaction time of 4h, and the eluent MeOH/CH_2Cl_2 = 1/18 was used) to give **3e** (16.50 mg, 32%), as a white solid.
mp 158.2–161.0 °C; IR (neat) 3438, 3092, 3038, 2981, 2932, 1779 (C=O), 1727 (C=O), 1695, 1596, 1555, 1444, 1413, 1392, 1368, 1349, 1327, 1260, 1201, 1186, 1156, 1099, 1013, 889, 871, 823, 780, 768, 737 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.74 (br s, 2H), 7.39 (d, *J* = 5.7 Hz, 2H), 6.59 (s, 1H, C=C–H), 4.69 (d, *J* = 1.2 Hz, 2H), 1.58 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.1 (C), 153.4 (C), 151.0 (CH×2), 149.5 (C), 137.9 (C), 123.7 (CH×2), 120.0 (CH), 83.6 (C, O-$C(CH_3)_3$), 50.7 (CH_2), 28.1 (CH_3×3); HRMS (ESI$^+$): m/z calcd. for $C_{14}H_{16}N_2O_3$ $[M+H]^+$: 261.1239. Found 261.1234.

***tert*-Butyl-2-oxo-4-(pyrimidin-2-yl)-2,5-dihydro-1H-pyrrole-1-carboxylate (3f)**

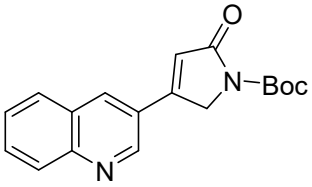


3f

Compound **2f** (0.05 g, 0.18 mmol), AIBN (0.5 equiv) and Bu_3SnH (1.2 equiv) was treated according to method C (with the reaction time of 12h, and the eluent EtOAc/Hexanes = 6/4 was used) to give **3f** (11.50 mg, 24%), as a yellow solid.

mp 157.7–161.3 °C; IR (neat) 3444, 3122, 3075, 3002, 2977, 2927, 1770 (C=O), 1697 (C=O), 1634, 1561, 1478, 1450, 1441, 1422, 1390, 1387, 1349, 1297, 1271, 1242, 1211, 1179, 1107, 1071, 989, 890, 880, 862, 843, 758, 738 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.81 (d, *J* = 4.8 Hz, 2H), 7.30 (t, *J* = 5.0 Hz, 1H), 7.00 (t, *J* = 1.7 Hz, 1H, C=C–H), 4.79 (d, *J* = 1.8 Hz, 2H), 1.57 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.0 (C), 159.7 (C), 157.4 (CH), 157.0 (CH), 155.3 (C), 149.5 (C), 127.5 (CH), 121.0 (CH), 83.1 (C, O-$C(CH_3)_3$), 51.1 (CH_2), 28.2 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{13}H_{15}N_3O_3$ $[M+H]^+$: 262.1192. Found 262.1185.

***tert*-Butyl 2-oxo-4-(quinolin-3-yl)-2,5-dihydro-1H-pyrrole-1-carboxylate (3g)**



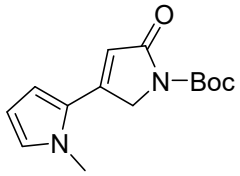
3g

Compound **2g** (62.05 mg, 0.20 mmol), AIBN (0.6 equiv) and Bu_3SnH (1.25 equiv) was treated according to method C (with the reaction time of 4h, and the eluent EtOAc/Hexanes = 25/75, 1/1 was used) to give **3g** (7.0 mg, 11%), as a yellow solid.

mp 163.8–166.6 °C; IR (neat) 3061, 2979, 2930, 1770 (C=O), 1727 (C=O), 1622, 1571, 1541, 1496, 1476, 1456, 1393, 1357, 1316, 1258, 1159, 1094, 1079, 1025, 984, 961, 859, 811, 788, 756, 732 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.13 (br s, 1H), 8.28 (s,

1H), 8.18 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 8.1 Hz, 1H), 7.83 (t, J = 7.5 Hz, 1H), 7.65 (t, J = 7.5 Hz, 1H), 6.66 (s, 1H, C=C–H), 4.84 (d, J = 0.9 Hz, 2H), 1.61 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.6 (C), 153.0 (C), 149.6 (C), 148.9 (C), 147.7 (CH), 133.2 (CH), 131.3 (CH), 129.6 (CH), 128.5 (CH), 127.9 (CH), 127.2 (C), 124.0 (C), 121.3 (C), 83.4 (C, O-C$(CH_3)_3$), 50.9 (CH_2), 28.2 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{18}H_{18}N_2O_3$ $[M+H]^+$: 311.1395. Found 311.1393.

tert-Butyl 1-methyl-5'-oxo-2',5'-dihydro-1H,1'H-[2,3'-bipyrrole]-1'-carboxylate (3h)



3h

Bu_3SnH as the reagent

Compound **2h** (0.05 g, 0.20 mmol), AIBN (0.6 equiv) and Bu_3SnH (1.1 equiv) was treated according to method C (with the reaction time of 4h, and the eluent EtOAc/Hexanes = 1/1 was used) to give **3h** (31.9 mg, 61%), as a yellow solid.

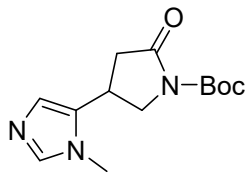
TTMSS as the reagent

Compound **2h** (0.05 g, 0.18 mmol), AIBN (0.6 equiv) and TTMSS (1.0 equiv) was treated according to method D (with the reaction time of 4 h, and the eluent EtOAc/Hexanes = 1/1 was used) to give **3h** (2.8 mg, 6%), as a white solid.

mp 121.0–123.8 °C; IR (neat) 3108, 2934, 2979, 1765(C=O), 1723 (C=O), 1609, 1522, 1477, 1450, 1420, 1394, 1355, 1330, 1290, 1256, 1216, 1160, 1100, 1062, 1039, 981, 890, 864, 846, 833, 774, 733 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.82 (s, 1H), 6.53 (dd, J = 3.9, 1.5 Hz, 1H), 6.17 (dd, J = 3.6, 2.7 Hz, 1H), 5.98 (s, 1H, C=C–H), 4.60 (d, J = 0.9 Hz, 2H, NCH_2), 3.76 (s, 3H,), 1.55 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.0

(C), 149.7 (C), 146.7 (C), 129.6 (CH), 125.2 (C), 113.8 (CH), 113.3 (CH), 109.0 (CH), 82.8 (C), 51.7 (CH_2), 36.9 (CH_3), 28.2 (CH_3×3); HRMS (ESI^+): m/z calcd. for $C_{14}H_{18}N_2O_3$ $[M+Na]^+$: 285.1215. Found 264.1221.

***tert*-Butyl 4-(1-methyl-1H-imidazol-5-yl)-2-oxopyrrolidine-1-carboxylate (4)**

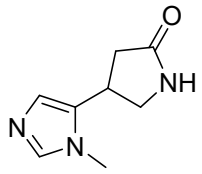


4

To a solution of compound **3a** (290.90 mg, 1.10 mmol) in MeOH (11 mL) was added AcOH (0.13 mL) and Pd/C (72.70 mg, 25 wt.%). The air inside of the reaction vessel was removed and replaced with H_2 gas. The reaction mixture was hydrogenated at room temperature under 70 psi for 2 days. After TLC analysis indicated that the starting material had been consumed completely, the reaction mixture was added K_2CO_3, and filtered by a pad of celite. The filtrate was concentrated to give a white solid **3a** (287.5 mg, 98%), which was directly used for the next step without further purification.

mp 114.7–118.1 °C; IR (neat) 3422, 2982, 1777, 1714 (C=O), 1642 (C=O), 1564, 1508, 1478, 1370, 1307, 1259, 1154, 1116, 1091, 1024, 924, 835, 779, 750 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.36 (br s, 1H), 6.80 (br s, 1H), 4.04 (dd, *J* = 10.5, 7.8 Hz, 1H), 3.64–3.58 (m, 1H), 3.53 (s, 1H), 3.52–3.38 (m, 1H), 2.80 (dd, *J* = 17.1, 8.1 Hz, 1H), 2.53 (dd, *J* = 17.0, 9.2 Hz), 1.43 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.8 (C), 150.0 (C), 139.0 (CH), 131.2 (C), 125.5 (CH), 83.3 (C), 50.9 (CH_2), 38.9 (CH_2), 31.6 (CH_3), 28.0 (CH_3×3), 26.9 (CH); HRMS (ESI^+): m/z calcd. for $C_{13}H_{19}N_3O_3$ $[M+H]^+$: 266.1505. Found 266.1504.

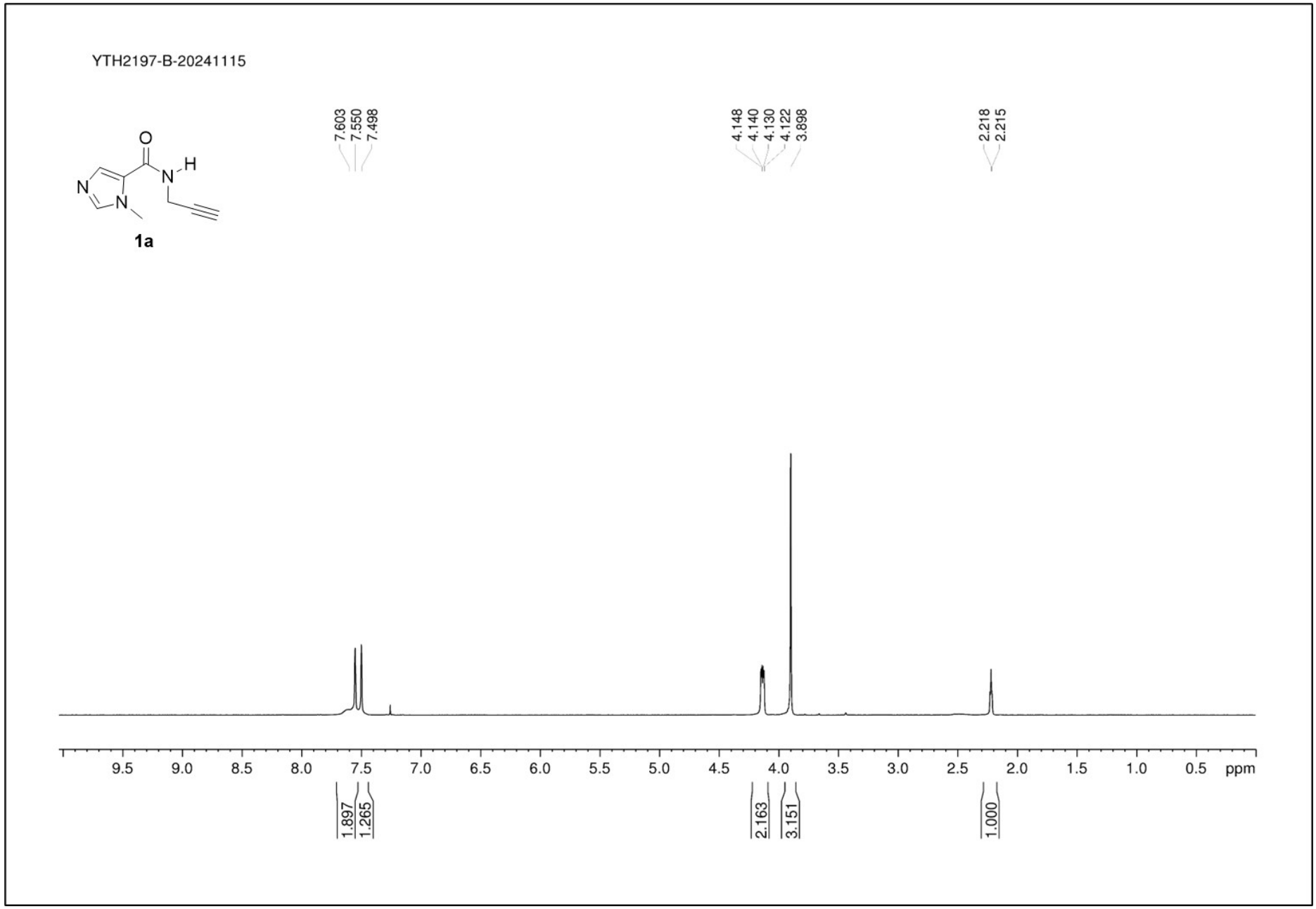
4-(1-Methyl-1H-imidazol-5-yl)pyrrolidin-2-one (5)



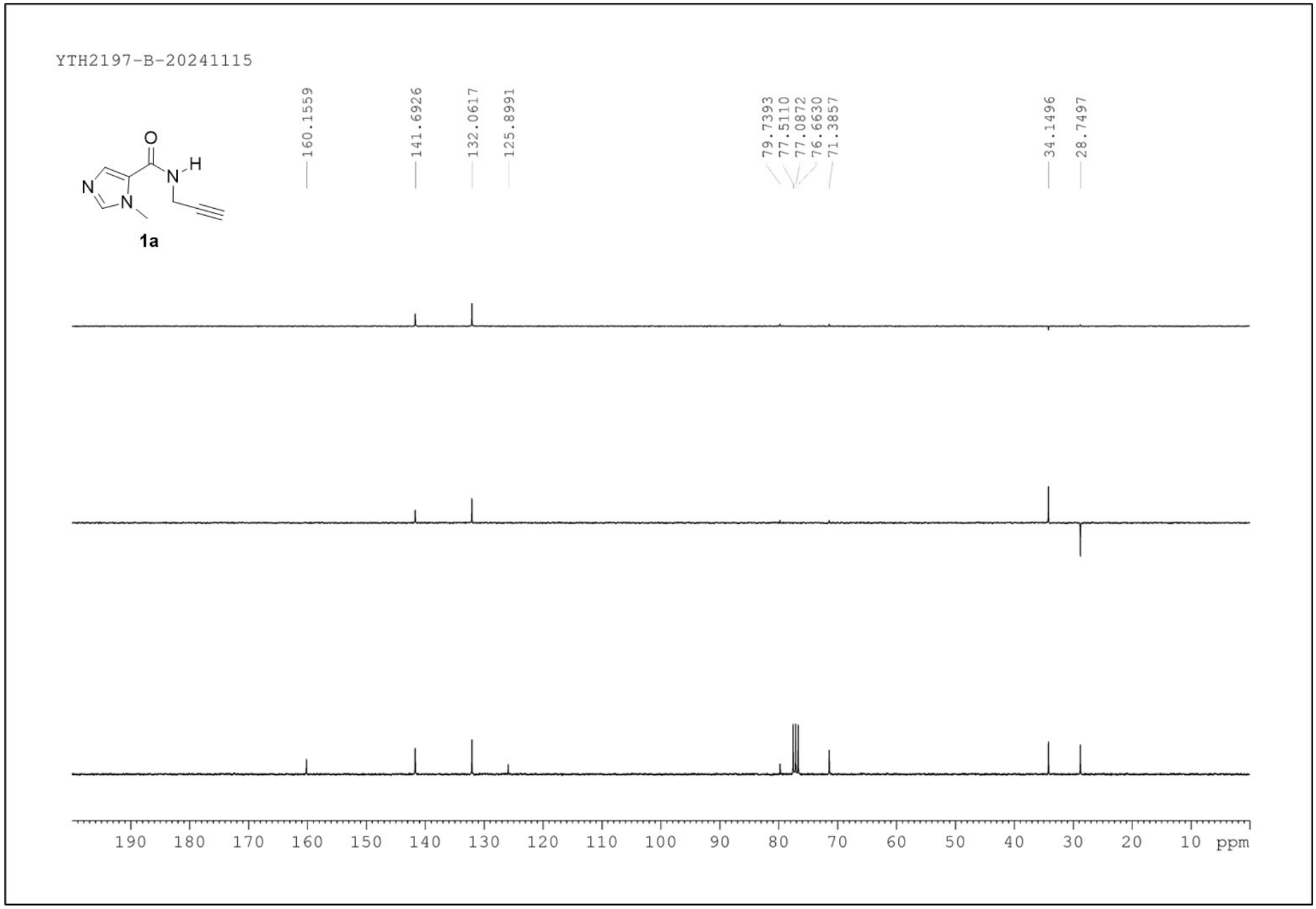
5

To a solution of compound **4** (0.02 g, 0.08 mmol) in THF (0.45 mL) was added to a solution of Na_2CO_3 (0.01 g, 0.09 mmol) in H_2O (0.3 mL) at room temperature for overnight (16 hrs). After TLC analysis indicated that the starting material was not consumed completely, the reaction temperature was warmed up to 50 ^oC for another 17 hrs. The residual of H_2O was removed by azeotropic evaporation 3 times with toluene (1 mL×3). The resulting mixture was filtered and washed with CH_2Cl_2, then concentrated to give the crude product, which was purified by flash chromatography (with the eluent MeOH/CH_2Cl_2 = 1/5 was used) to give **5** (6.0 mg, 48%), as a transparent liquid.

IR (neat) 3406 (NH), 2151, 1773, 1679 (C=O), 1564, 1510, 1451, 1425, 1369, 1335, 1299, 1273, 1236, 1160, 1115, 1082, 1048, 948, 905, 810 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.42 (s, 1H), 6.88 (s, 1H), 6.32 (br s, 1H, amide N-H), 3.79–3.71 (m, 1H), 3.68–3.56 (m, overlapped with one s at 3.59, N-CH_3, 4H), 3.48–3.43 (dd, *J* = 8.1, 7.8 Hz, 1H), 2.72 (dd, *J* = 16.7, 8.6 Hz, 1H), 2.43 (dd, *J* = 13.5, 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 176.5 (C), 138.7 (CH), 133.1 (C), 125.7 (CH), 47.2 (CH_2), 36.4 (CH_2), 31.6 (CH), 30.7 (CH_3); HRMS (ESI^+): m/z calcd. for $C_8H_{11}N_3O$ $[M+H]^+$: 166.0980. Found 166.0980.



^{1}H NMR (300 MHz, $CDCl_3$) of **1a**



$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1a**

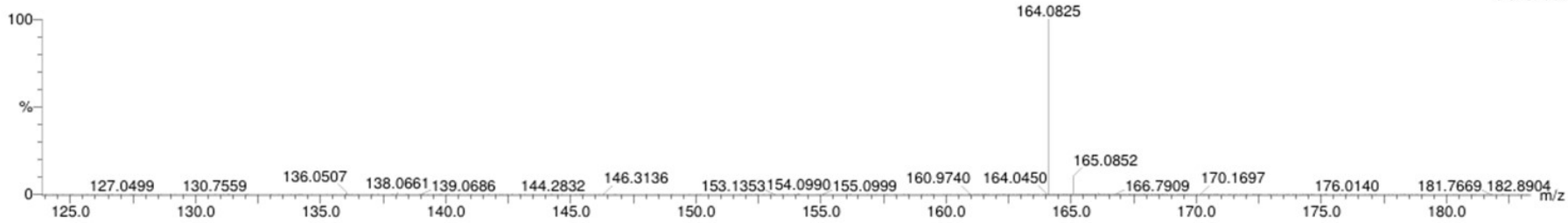
Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
111 formula(e) evaluated with 35 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-20
YTH3032B
250919FJU21 33 (0.340) Cm (31:35-(18:23+55:60))

1: TOF MS ES+
3.02e+005



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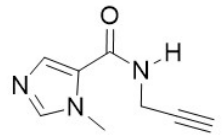
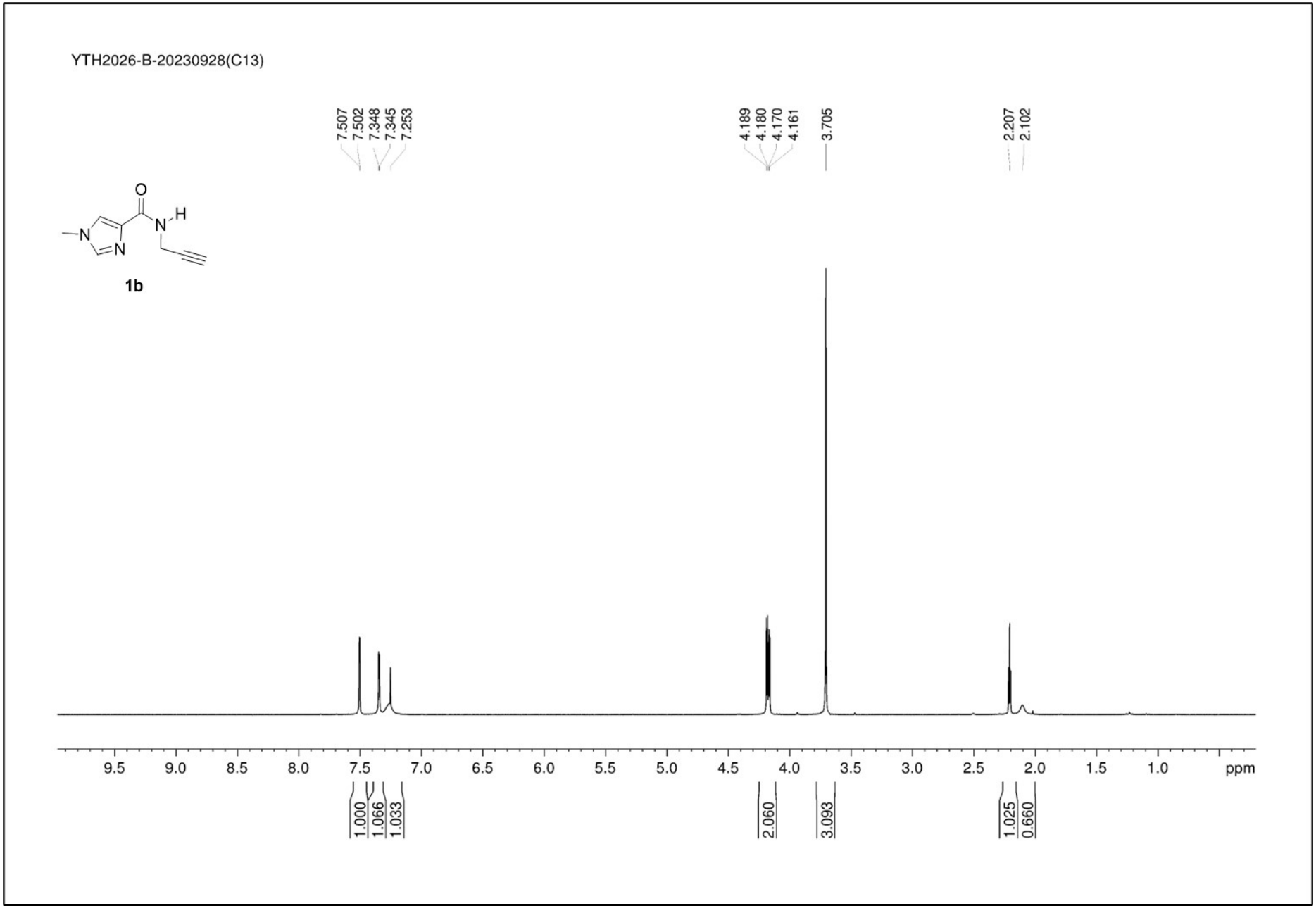
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
164.0825	100.00	164.0824	0.1	0.6	5.5	C8 H10 N3 O
		164.0784	4.1	25.0	1.5	C3 H10 N5 O3
		164.0770	5.5	33.5	-3.5	C2 H14 N O7
		164.0883	-5.8	-35.3	-3.5	C H14 N3 O6
		164.0896	-7.1	-43.3	1.5	C2 H10 N7 O2
		164.0923	-9.8	-59.7	0.5	C6 H14 N O4
		164.0712	11.3	68.9	5.5	C9 H10 N O2
		164.0671	15.4	93.9	1.5	C4 H10 N3 O4
		164.1008	-18.3	-111.5	1.5	C H10 N9 O
		164.1035	-21.0	-128.0	0.5	C5 H14 N3 O3
		164.1075	-25.0	-152.4	4.5	C10 H14 N O
		164.0572	25.3	154.2	6.5	C6 H6 N5 O
		164.0559	26.6	162.1	1.5	C5 H10 N O5
		164.0532	29.3	178.6	2.5	C H6 N7 O3
		164.1134	-30.9	-188.3	-4.5	C3 H18 N O6
		164.1147	-32.2	-196.2	0.5	C4 H14 N5 O2
		164.0460	36.5	222.4	6.5	C7 H6 N3 O2
		164.0420	40.5	246.8	2.5	C2 H6 N5 O4
		164.0406	41.9	255.4	-2.5	C H10 N O8
		164.1246	-42.1	-256.6	-4.5	C2 H18 N3 O5
		164.1260	-43.5	-265.1	0.5	C3 H14 N7 O
		164.1287	-46.2	-281.6	-0.5	C7 H18 N O3
		164.0348	47.7	290.7	6.5	C8 H6 N O3
		164.0321	50.4	307.2	7.5	C4 H2 N7 O
		164.0307	51.8	315.7	2.5	C3 H6 N3 O5

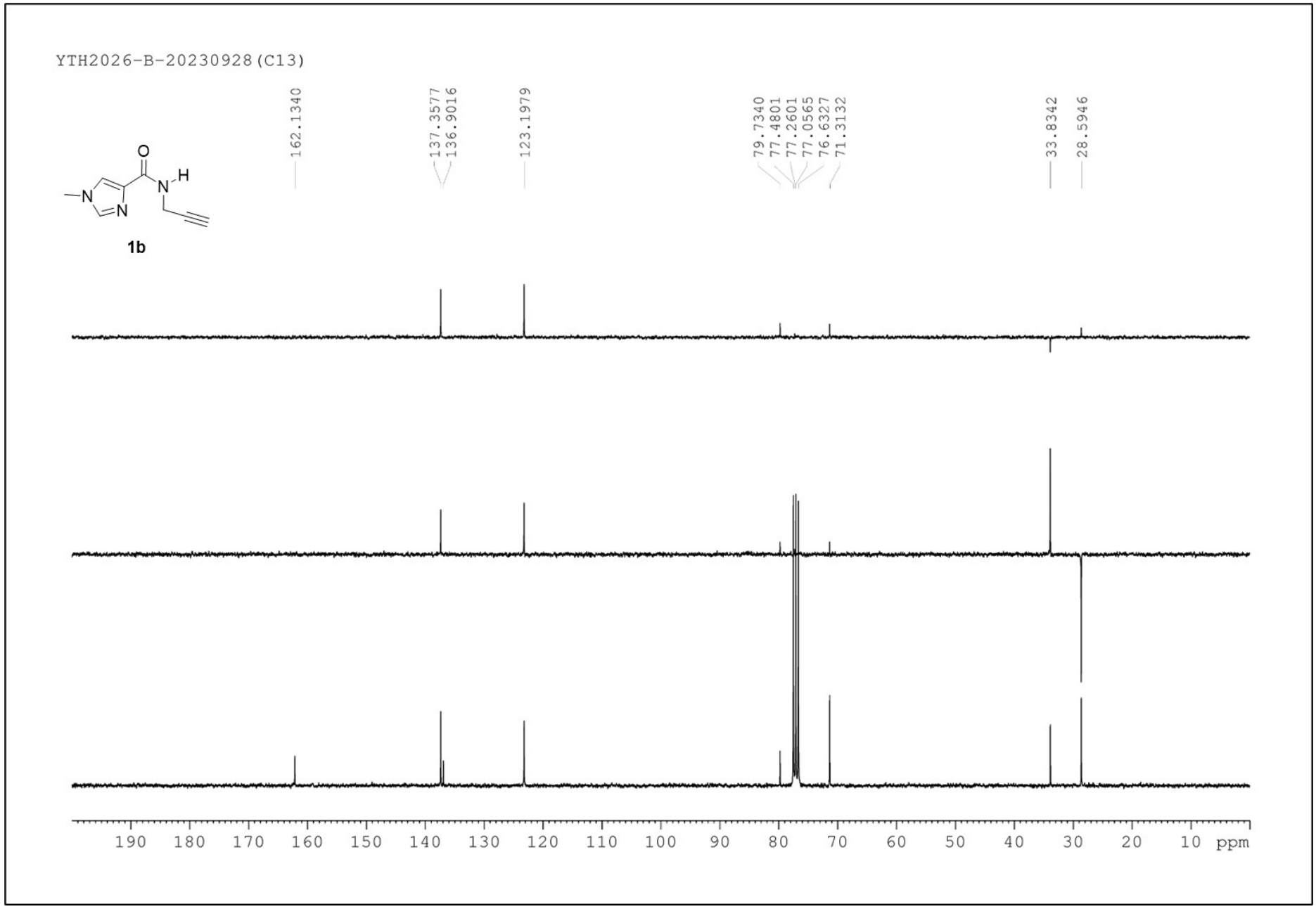


1a

HRMS (ESI$^+$) of **1a**



^{1}H NMR (300 MHz, $CDCl_3$) of **1b**



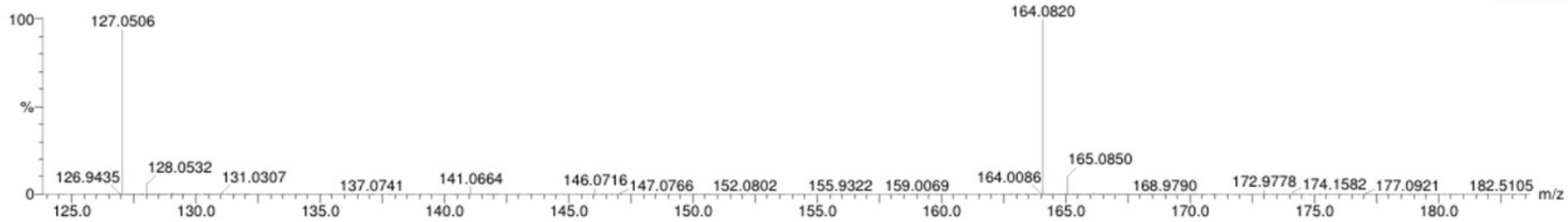
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1b**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
111 formula(e) evaluated with 35 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2026B
250919FJU27 37 (0.394) Cm (32:39-(15:21+59:65))

1: TOF MS ES+
1.39e+005



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Maximum:		10.0	500.0	100.0	
Mass	Calc. Mass	mDa	PPM	DBE	Formula
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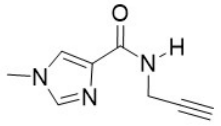
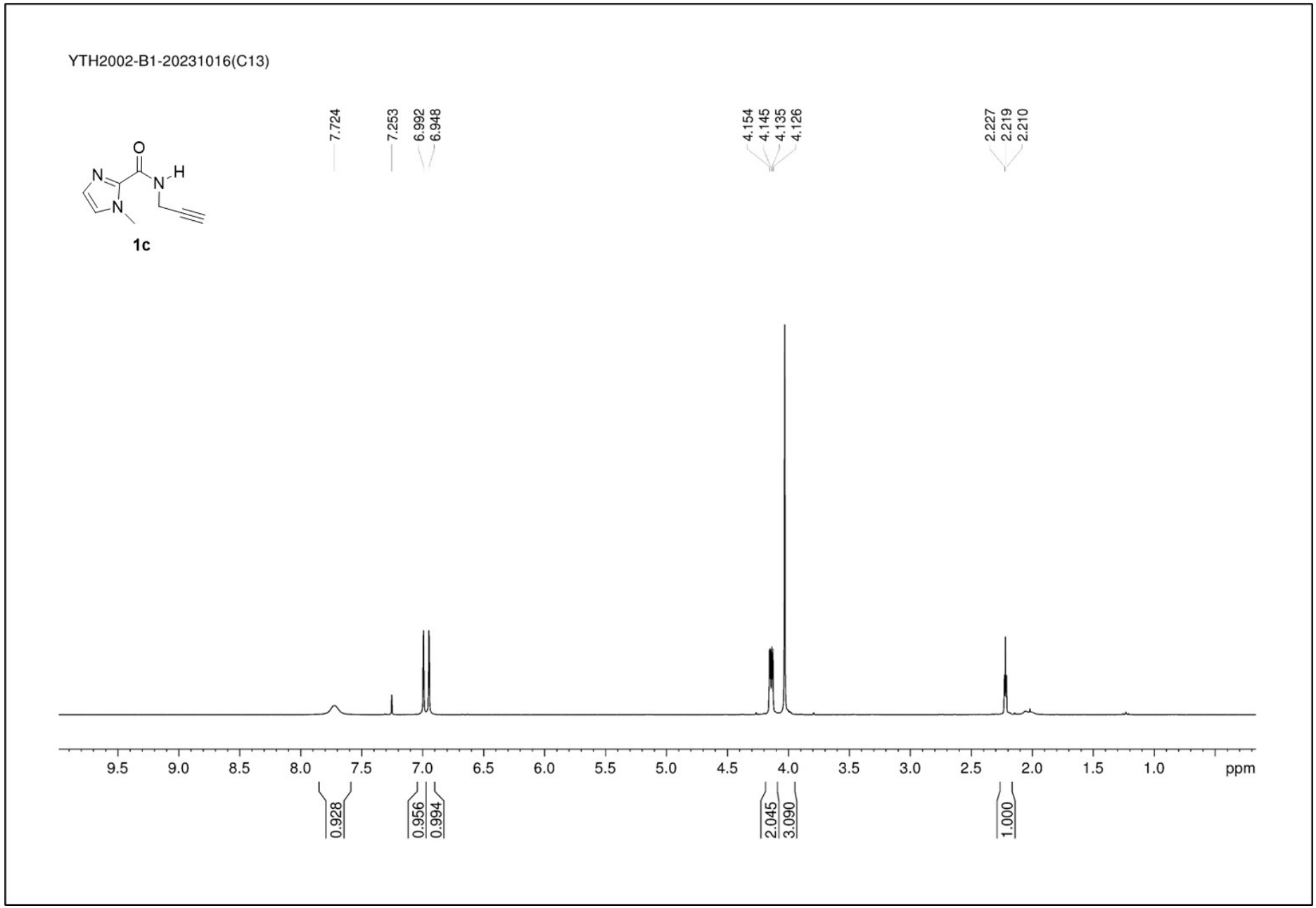
	164.0784	3.6	21.9	1.5	C3 H10 N5 O3
	164.0770	5.0	30.5	-3.5	C2 H14 N O7
	164.0883	-6.3	-38.4	-3.5	C H14 N3 O6
	164.0896	-7.6	-46.3	1.5	C2 H10 N7 O2
	164.0923	-10.3	-62.8	0.5	C6 H14 N O4
	164.0712	10.8	65.8	5.5	C9 H10 N O2
	164.0671	14.9	90.8	1.5	C4 H10 N3 O4
	164.1008	-18.8	-114.6	1.5	C H10 N9 O
	164.1035	-21.5	-131.0	0.5	C5 H14 N3 O3
	164.0572	24.8	151.1	6.5	C6 H6 N5 O
	164.1075	-25.5	-155.4	4.5	C10 H14 N O
	164.0559	26.1	159.1	1.5	C5 H10 N O5
	164.0532	28.8	175.5	2.5	C H6 N7 O3
	164.1134	-31.4	-191.4	-4.5	C3 H18 N O6
	164.1147	-32.7	-199.3	0.5	C4 H14 N5 O2
	164.0460	36.0	219.4	6.5	C7 H6 N3 O2
	164.0420	40.0	243.8	2.5	C2 H6 N5 O4
	164.0406	41.4	252.3	-2.5	C H10 N O8
	164.1246	-42.6	-259.6	-4.5	C2 H18 N3 O5
	164.1260	-44.0	-268.2	0.5	C3 H14 N7 O
	164.1287	-46.7	-284.6	-0.5	C7 H18 N O3
	164.0348	47.2	287.7	6.5	C8 H6 N O3
	164.0321	49.9	304.1	7.5	C4 H2 N7 O
	164.0307	51.3	312.6	2.5	C3 H6 N3 O5

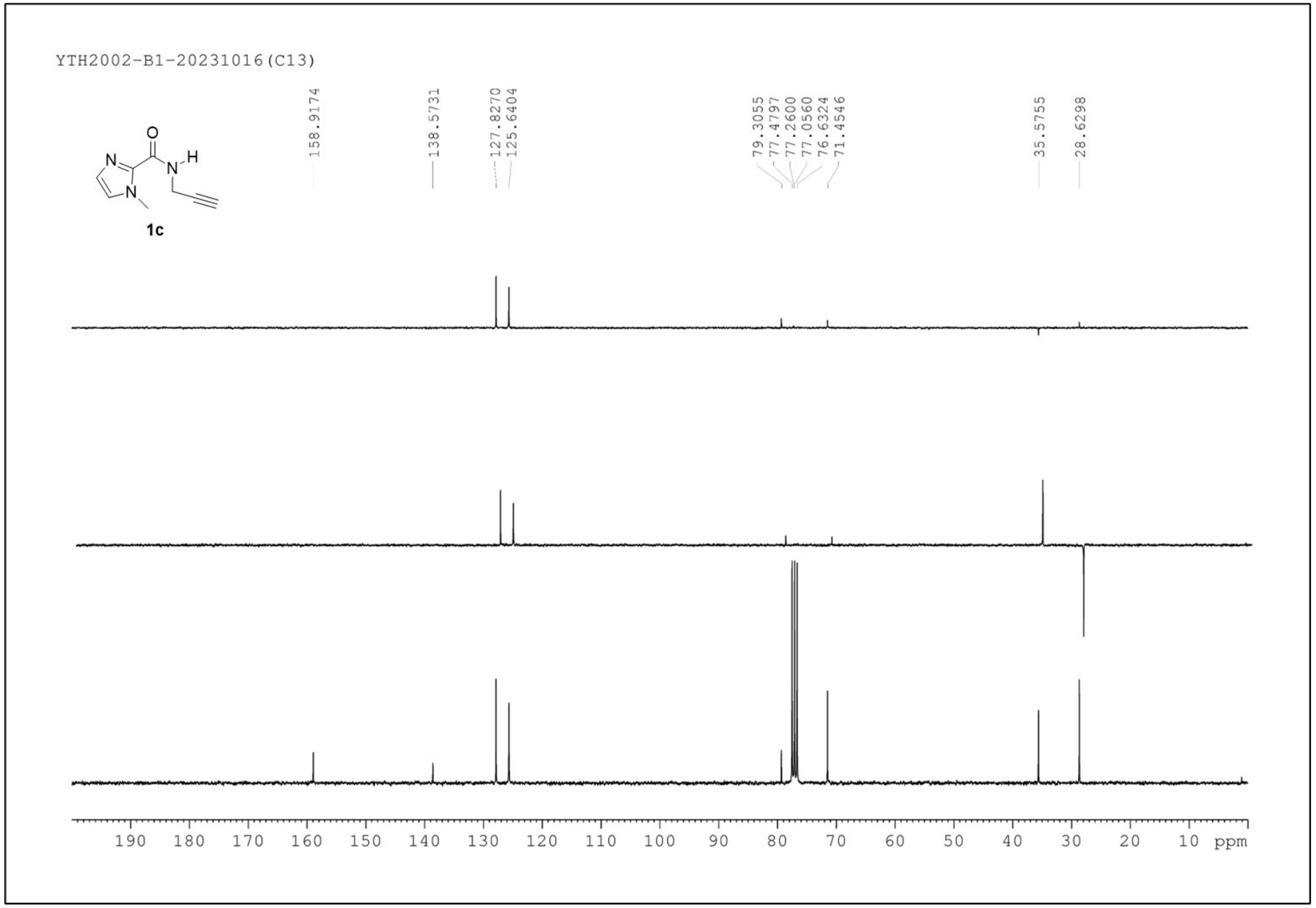


1b

HRMS (ESI$^+$) of **1b**



^{1}H NMR (300 MHz, $CDCl_3$) of **1c** (trace of impurity)



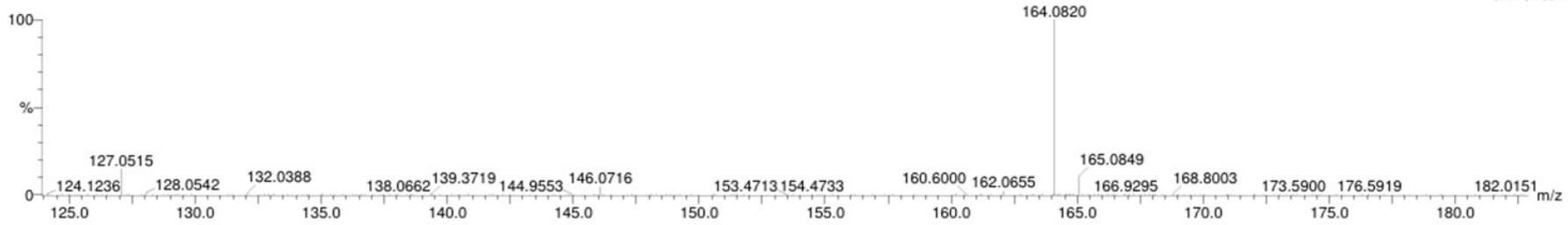
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1c**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

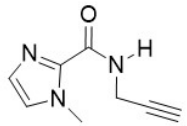
Monoisotopic Mass, Even Electron Ions
111 formula(e) evaluated with 35 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2002B1
250919FJU31 48 (0.488) Cm (46:50-(29:36+72:79))

1: TOF MS ES+
3.27e+004



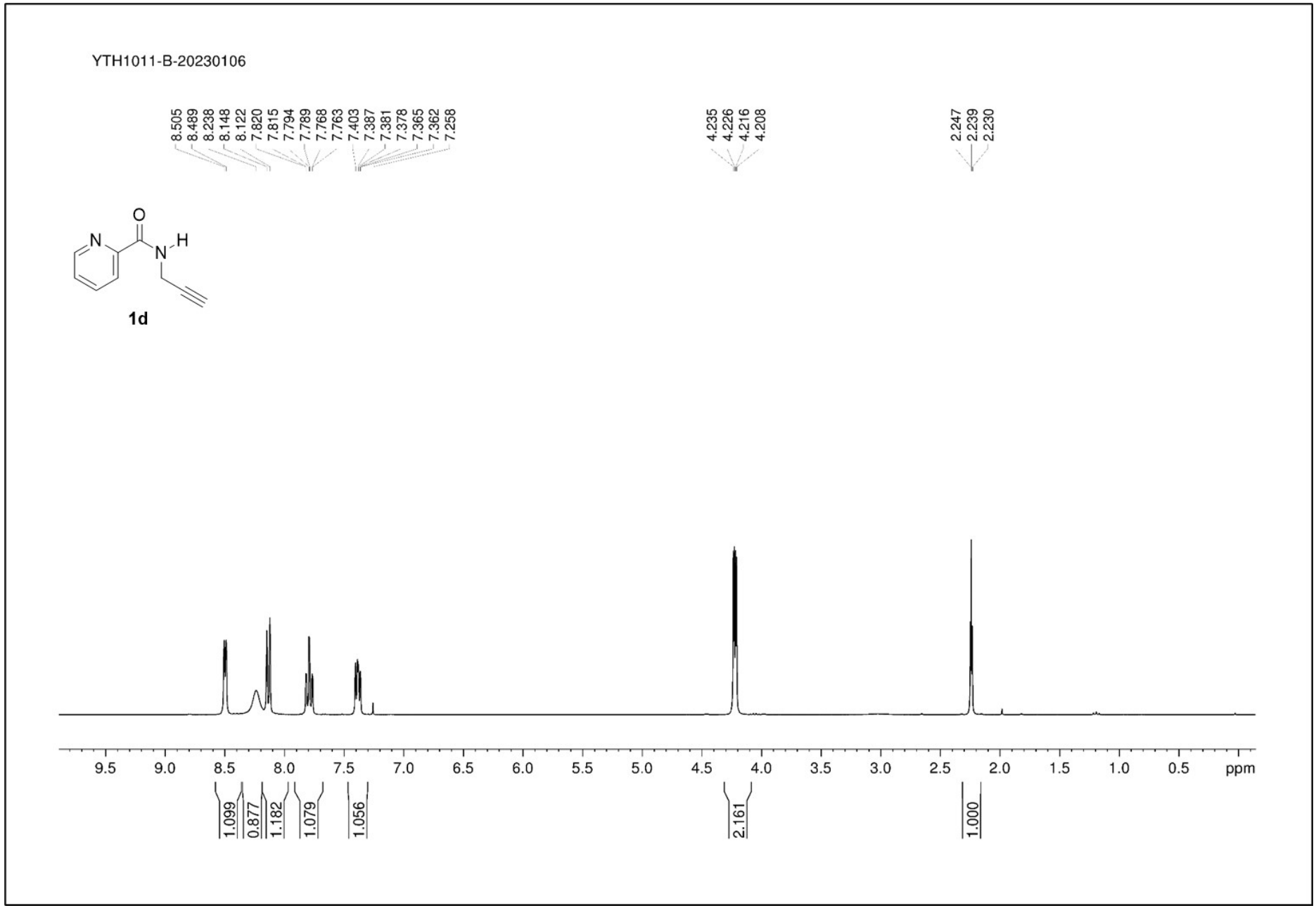
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
164.0820	100.00	164.0824	-0.4	-2.4	5.5	C8 H10 N3 O
		164.0784	3.6	21.9	1.5	C3 H10 N5 O3
		164.0770	5.0	30.5	-3.5	C2 H14 N O7
		164.0883	-6.3	-38.4	-3.5	C H14 N3 O6
		164.0896	-7.6	-46.3	1.5	C2 H10 N7 O2
		164.0923	-10.3	-62.8	0.5	C6 H14 N O4
		164.0712	10.8	65.8	5.5	C9 H10 N O2
		164.0671	14.9	90.8	1.5	C4 H10 N3 O4
		164.1008	-18.8	-114.6	1.5	C H10 N9 O
		164.1035	-21.5	-131.0	0.5	C5 H14 N3 O3
		164.0572	24.8	151.1	6.5	C6 H6 N5 O
		164.1075	-25.5	-155.4	4.5	C10 H14 N O
		164.0559	26.1	159.1	1.5	C5 H10 N O5
		164.0532	28.8	175.5	2.5	C H6 N7 O3
		164.1134	-31.4	-191.4	-4.5	C3 H18 N O6
		164.1147	-32.7	-199.3	0.5	C4 H14 N5 O2
		164.0460	36.0	219.4	6.5	C7 H6 N3 O2
		164.0420	40.0	243.8	2.5	C2 H6 N5 O4
		164.0406	41.4	252.3	-2.5	C H10 N O8
		164.1246	-42.6	-259.6	-4.5	C2 H18 N3 O5
		164.1260	-44.0	-268.2	0.5	C3 H14 N7 O
		164.1287	-46.7	-284.6	-0.5	C7 H18 N O3
		164.0348	47.2	287.7	6.5	C8 H6 N O3
		164.0321	49.9	304.1	7.5	C4 H2 N7 O
		164.0307	51.3	312.6	2.5	C3 H6 N3 O5

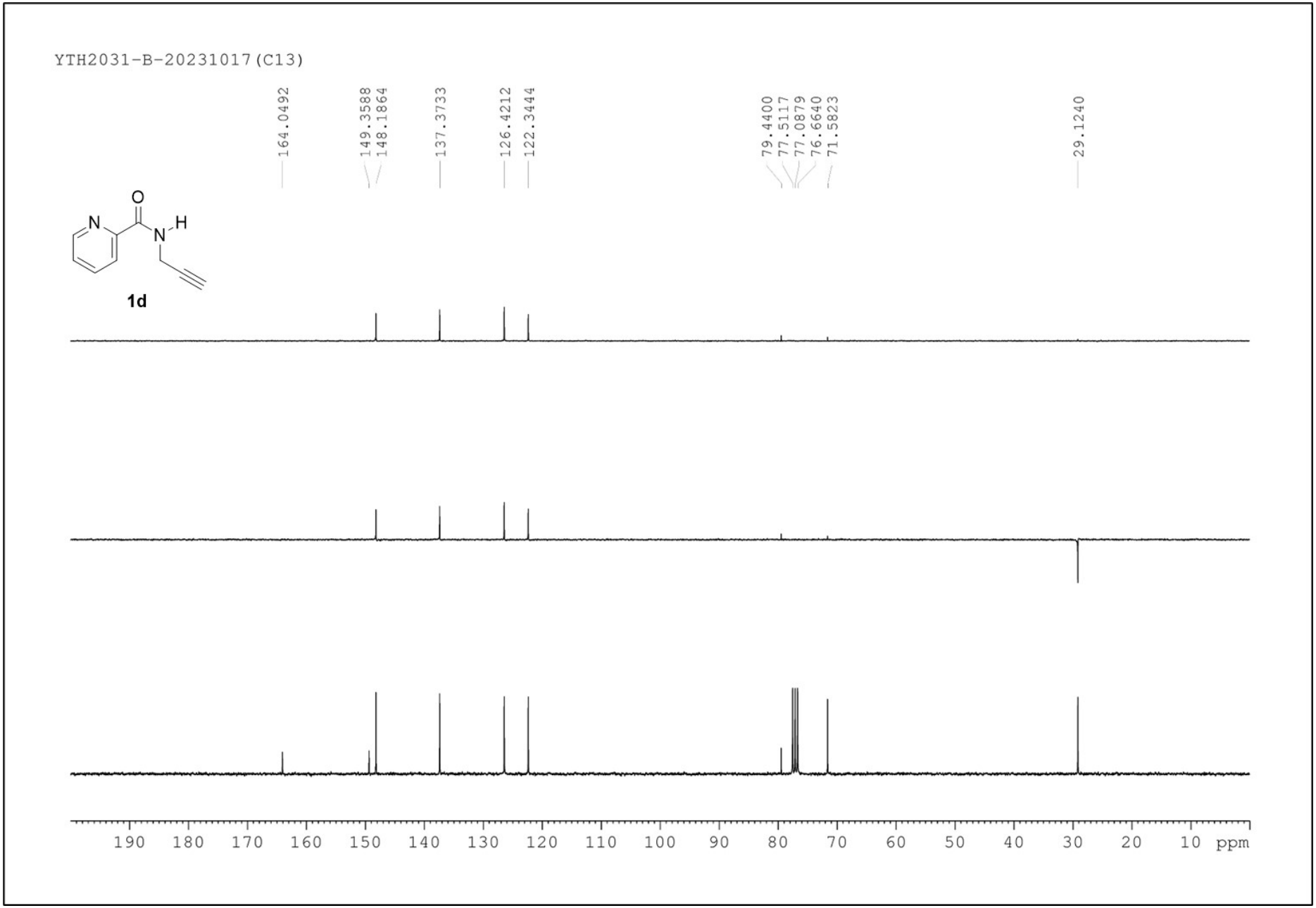


1c

HRMS (ESI$^+$) of **1c**



^{1}H NMR (300 MHz, $CDCl_3$) of **1d**



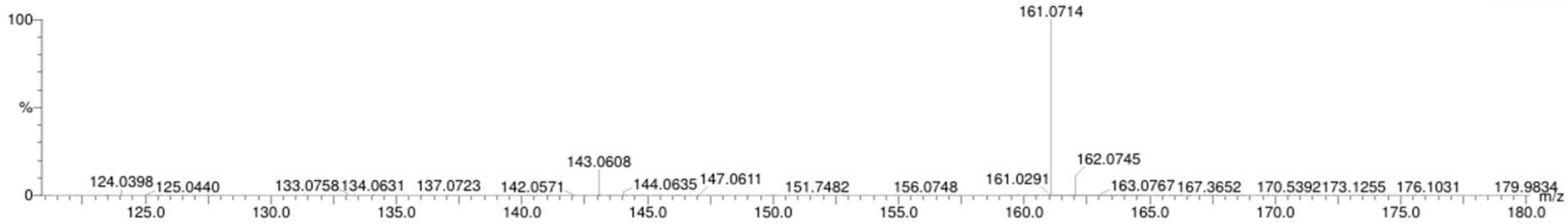
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1d**

Elemental Composition Report

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

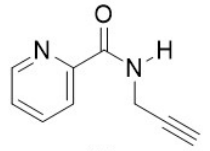
Monoisotopic Mass, Even Electron Ions
113 formula(e) evaluated with 28 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3177B
260108FJU01-3 184 (1.812) Cm (182:187-(167:175+212:219))

1: TOF MS ES+
2.23e+005



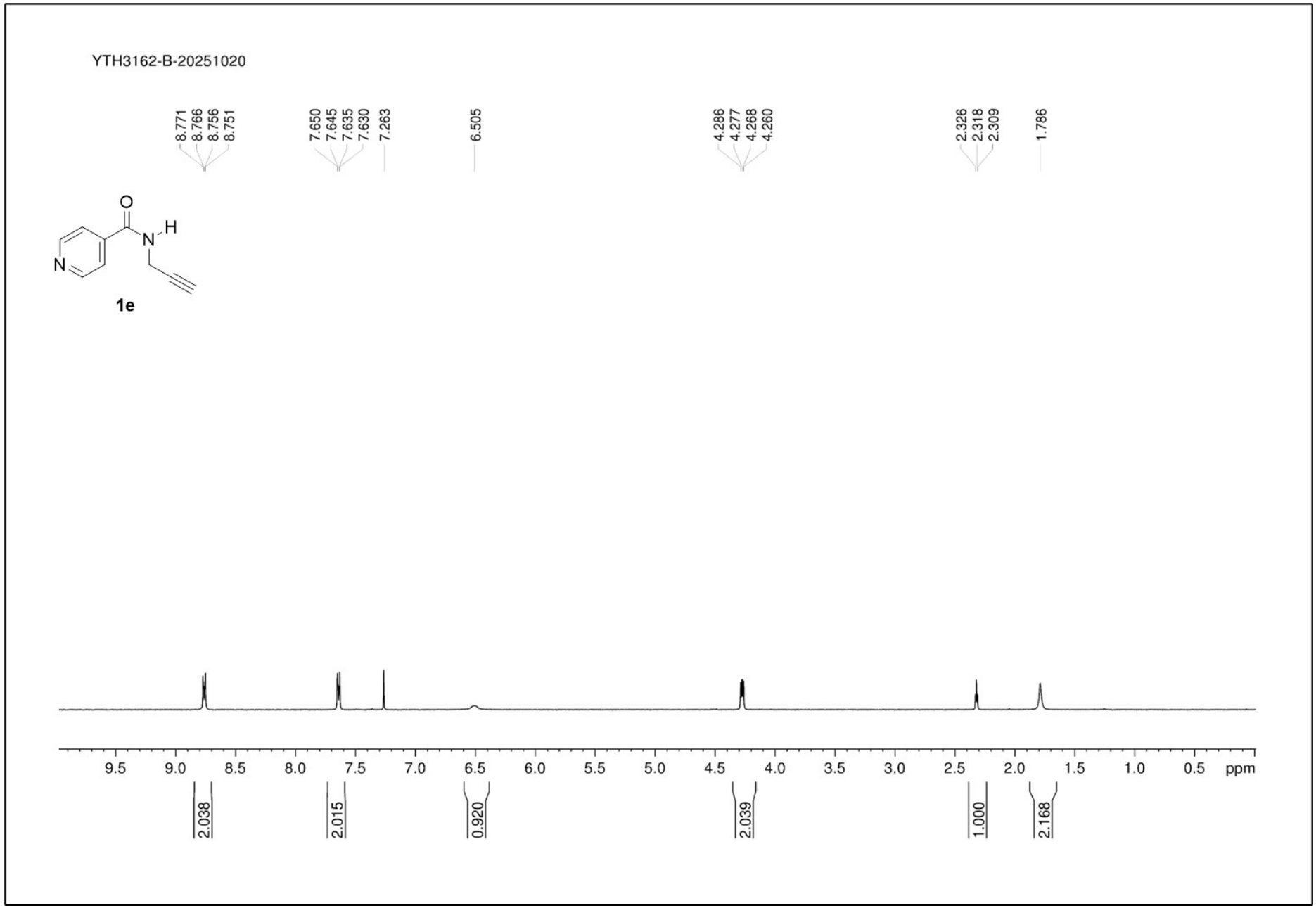
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
161.0714	100.00	161.0715	-0.1	-0.6	6.5	C9 H9 N2 O
		161.0675	3.9	24.2	2.5	C4 H9 N4 O3
		161.0774	-6.0	-37.3	-2.5	C2 H13 N2 O6
		161.0787	-7.3	-45.3	2.5	C3 H9 N6 O2
		161.0562	15.2	94.4	2.5	C5 H9 N2 O4
		161.0886	-17.2	-106.8	-2.5	C H13 N4 O5
		161.0535	17.9	111.1	3.5	C H5 N8 O2
		161.0899	-18.5	-114.9	2.5	C2 H9 N8 O
		161.0926	-21.2	-131.6	1.5	C6 H13 N2 O3
		161.0463	25.1	155.8	7.5	C7 H5 N4 O
		161.0423	29.1	180.7	3.5	C2 H5 N6 O3
		161.0410	30.4	188.7	-1.5	C H9 N2 O7
		161.1039	-32.5	-201.8	1.5	C5 H13 N4 O2
		161.0351	36.3	225.4	7.5	C8 H5 N2 O2
		161.0311	40.3	250.2	3.5	C3 H5 N4 O4
		161.1137	-42.3	-262.6	-3.5	C3 H17 N2 O5
		161.1151	-43.7	-271.3	1.5	C4 H13 N6 O
		161.0212	50.2	311.7	8.5	C5 H N6 O
		161.0198	51.6	320.4	3.5	C4 H5 N2 O5
		161.1250	-53.6	-332.8	-3.5	C2 H17 N4 O4
		161.1290	-57.6	-357.6	0.5	C7 H17 N2 O2
		161.0100	61.4	381.2	8.5	C6 H N4 O2
		161.1362	-64.8	-402.3	-3.5	C H17 N6 O3
		161.0059	65.5	406.7	4.5	C H N6 O4
		161.1402	-68.8	-427.1	0.5	C6 H17 N4 O

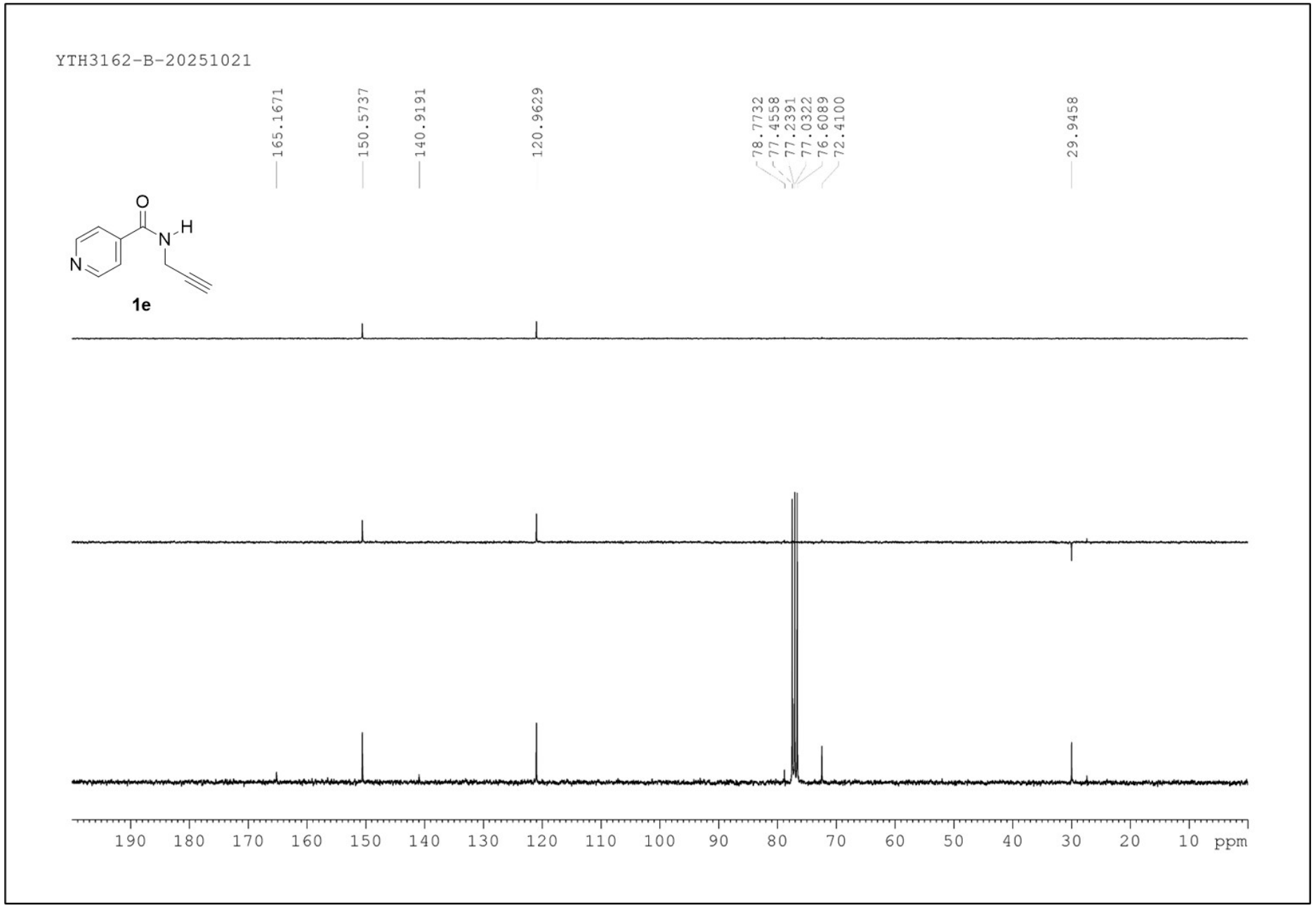


1d

HRMS (ESI$^+$) of **1d**



^{1}H NMR (300 MHz, $CDCl_3$) of **1e**



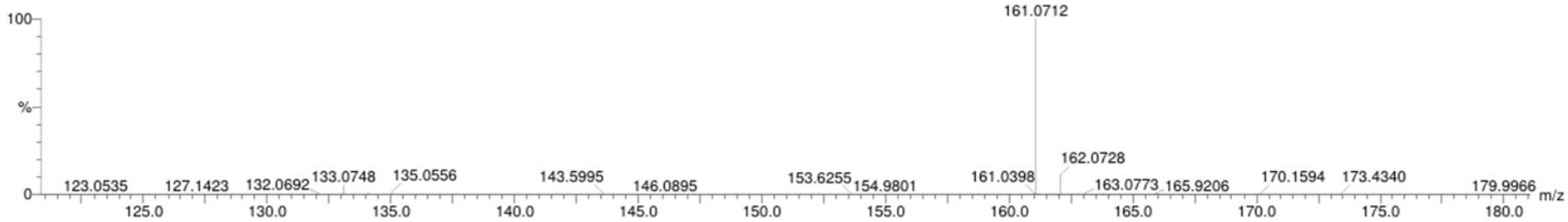
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1e**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

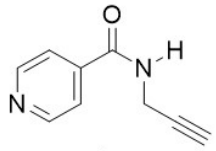
Monoisotopic Mass, Even Electron Ions
113 formula(e) evaluated with 28 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3162B
251211FJU01-3 59 (0.602) Cm (56:62-(37:45+90:97))

1: TOF MS ES+
1.58e+005



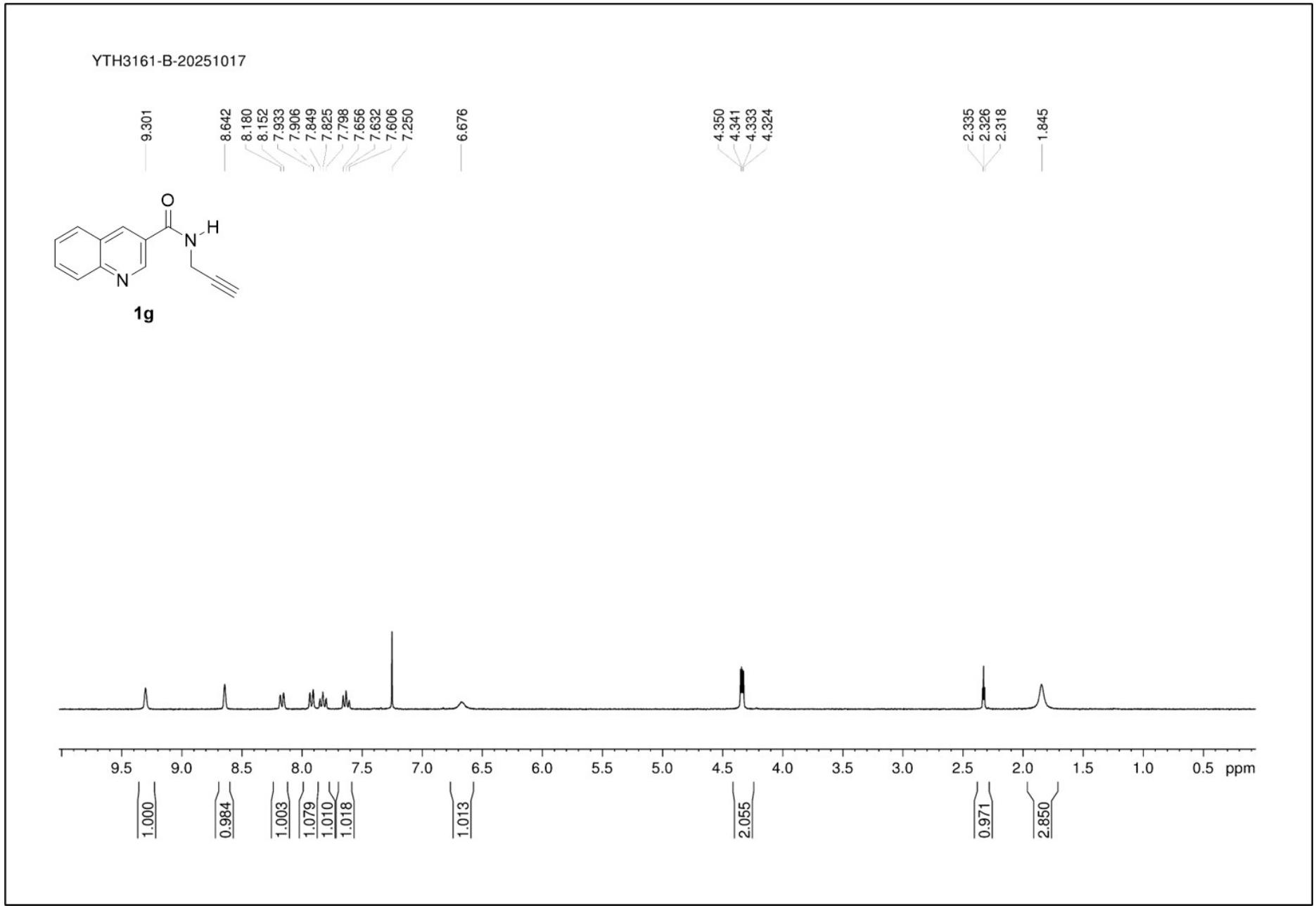
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
161.0712	100.00	161.0715	-0.3	-1.9	6.5	C9 H9 N2 O
		161.0675	3.7	23.0	2.5	C4 H9 N4 O3
		161.0774	-6.2	-38.5	-2.5	C2 H13 N2 O6
		161.0787	-7.5	-46.6	2.5	C3 H9 N6 O2
		161.0562	15.0	93.1	2.5	C5 H9 N2 O4
		161.0886	-17.4	-108.0	-2.5	C H13 N4 O5
		161.0535	17.7	109.9	3.5	C H5 N8 O2
		161.0899	-18.7	-116.1	2.5	C2 H9 N8 O
		161.0926	-21.4	-132.9	1.5	C6 H13 N2 O3
		161.0463	24.9	154.6	7.5	C7 H5 N4 O
		161.0423	28.9	179.4	3.5	C2 H5 N6 O3
		161.0410	30.2	187.5	-1.5	C H9 N2 O7
		161.1039	-32.7	-203.0	1.5	C5 H13 N4 O2
		161.0351	36.1	224.1	7.5	C8 H5 N2 O2
		161.0311	40.1	249.0	3.5	C3 H5 N4 O4
		161.1137	-42.5	-263.9	-3.5	C3 H17 N2 O5
		161.1151	-43.9	-272.6	1.5	C4 H13 N6 O
		161.0212	50.0	310.4	8.5	C5 H N6 O
		161.0198	51.4	319.1	3.5	C4 H5 N2 O5
		161.1250	-53.8	-334.0	-3.5	C2 H17 N4 O4
		161.1290	-57.8	-358.8	0.5	C7 H17 N2 O2
		161.0100	61.2	380.0	8.5	C6 H N4 O2
		161.1362	-65.0	-403.5	-3.5	C H17 N6 O3
		161.0059	65.3	405.4	4.5	C H N6 O4
		161.1402	-69.0	-428.4	0.5	C6 H17 N4 O

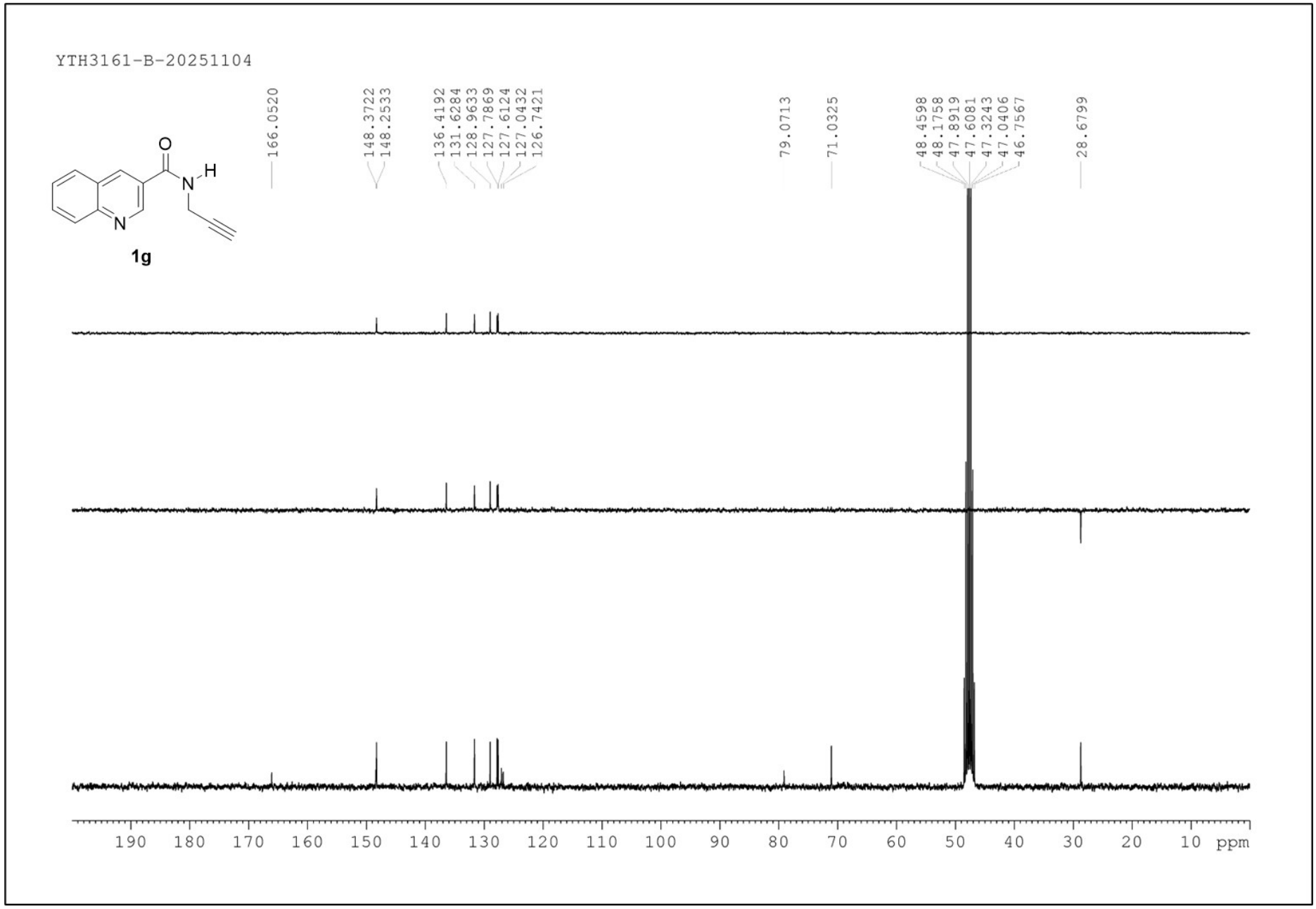


1e

HRMS (ESI$^+$) of **1e**



^{1}H NMR (300 MHz, $CDCl_3$) of **1g**



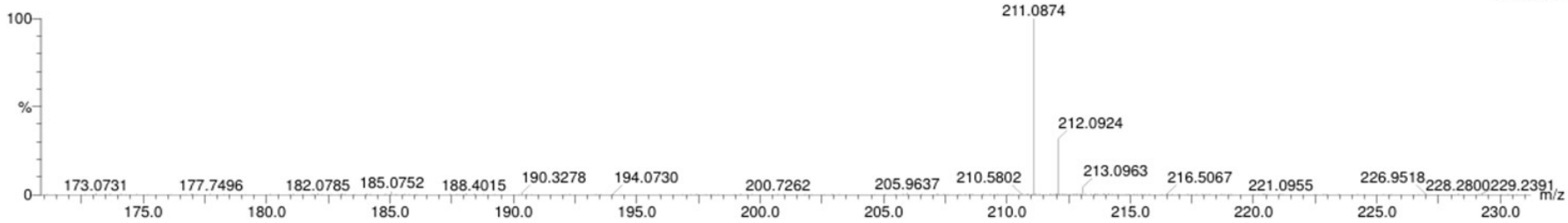
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, CD_3OD) of **1g**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

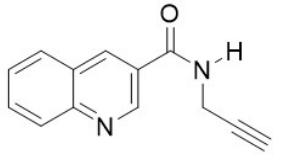
Monoisotopic Mass, Even Electron Ions
218 formula(e) evaluated with 62 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3161B
251210FJU02 155 (1.524) Cm (151:156-(136:144+178:185))

1: TOF MS ES+
4.98e+004



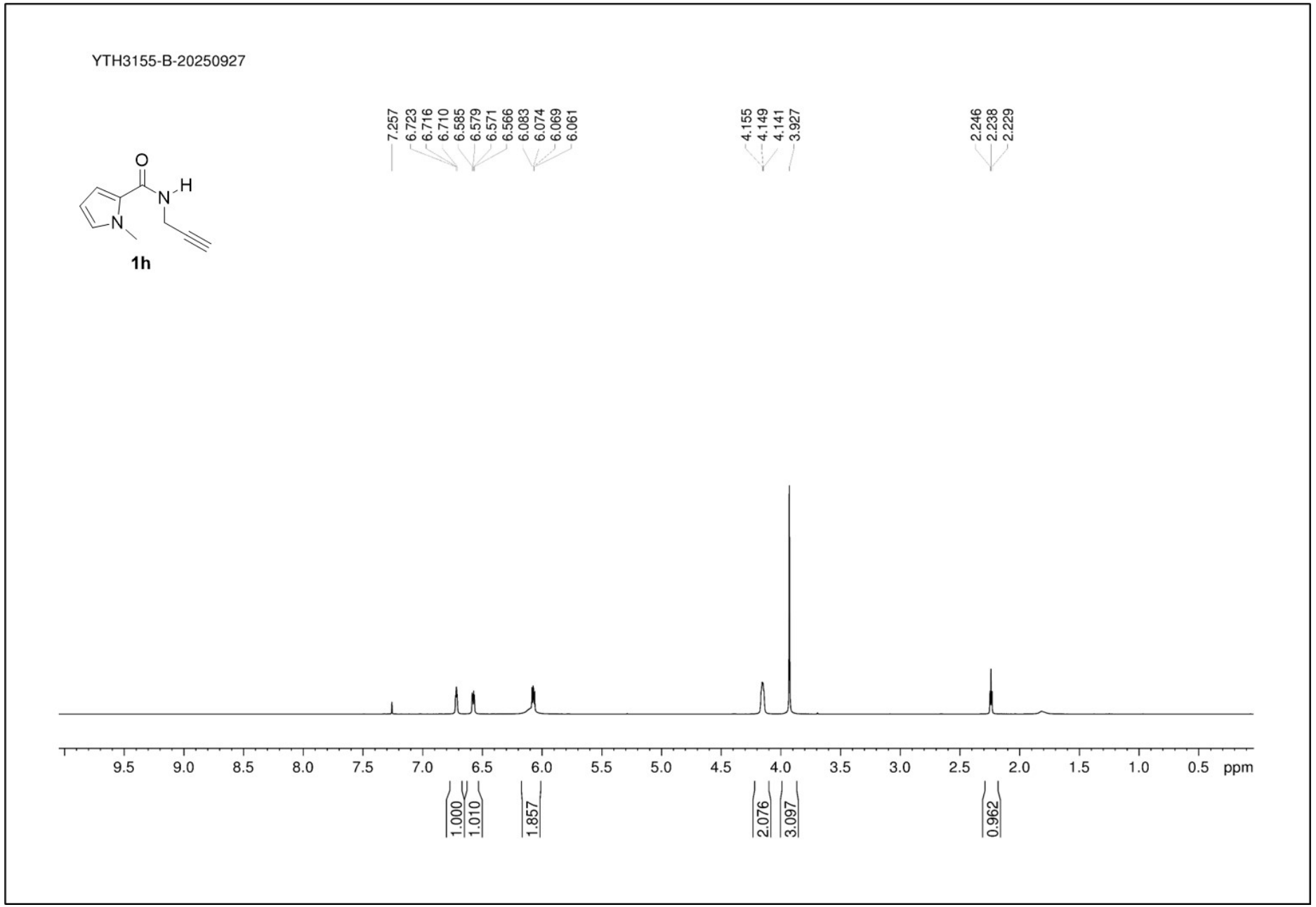
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
211.0874	100.00	211.0871	0.3	1.4	9.5	C13 H11 N2 O
		211.0890	-1.6	-7.6	-3.5	C H15 N4 O8
		211.0903	-2.9	-13.7	1.5	C2 H11 N8 O4
		211.0831	4.3	20.4	5.5	C8 H11 N4 O3
		211.0930	-5.6	-26.5	0.5	C6 H15 N2 O6
		211.0943	-6.9	-32.7	5.5	C7 H11 N6 O2
		211.0804	7.0	33.2	6.5	C4 H7 N10 O
		211.0791	8.3	39.3	1.5	C3 H11 N6 O5
		211.0778	9.6	45.5	-3.5	C2 H15 N2 O9
		211.1016	-14.2	-67.3	1.5	C H11 N10 O3
		211.0719	15.5	73.4	5.5	C9 H11 N2 O4
		211.1042	-16.8	-79.6	0.5	C5 H15 N4 O5
		211.0692	18.2	86.2	6.5	C5 H7 N8 O2
		211.1056	-18.2	-86.2	5.5	C6 H11 N8 O
		211.0679	19.5	92.4	1.5	C4 H11 N4 O6
		211.1083	-20.9	-99.0	4.5	C10 H15 N2 O3
		211.0620	25.4	120.3	10.5	C11 H7 N4 O
		211.1141	-26.7	-126.5	-4.5	C3 H19 N2 O8
		211.1155	-28.1	-133.1	0.5	C4 H15 N6 O4
		211.0580	29.4	139.3	6.5	C6 H7 N6 O3
		211.0566	30.8	145.9	1.5	C5 H11 N2 O7
		211.1195	-32.1	-152.1	4.5	C9 H15 N4 O2
		211.0539	33.5	158.7	2.5	C H7 N8 O5
		211.0508	36.6	173.4	10.5	C12 H7 N2 O2
		211.1254	-38.0	-180.0	-4.5	C2 H19 N4 O7

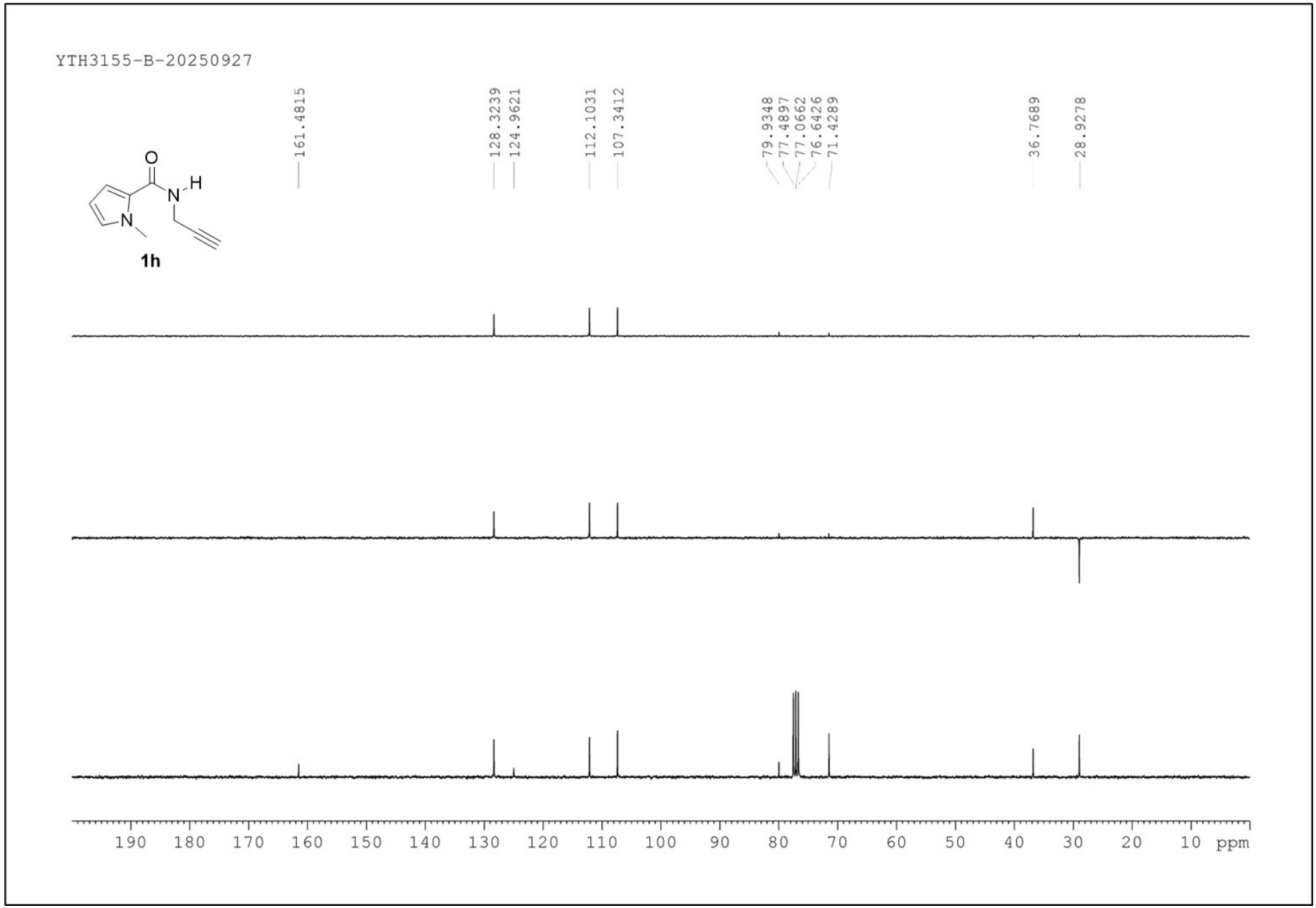


1g

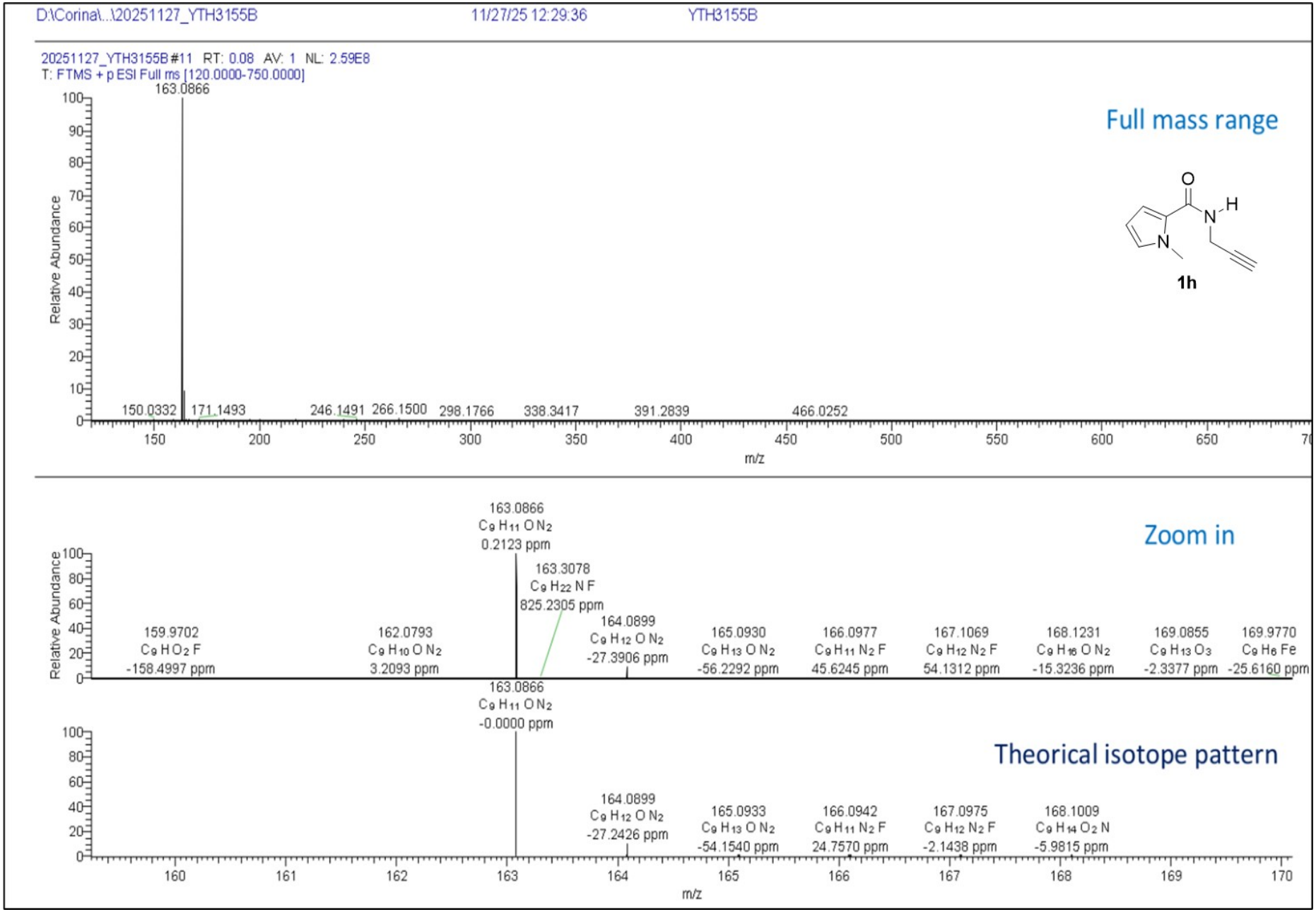
HRMS (ESI$^+$) of **1g**



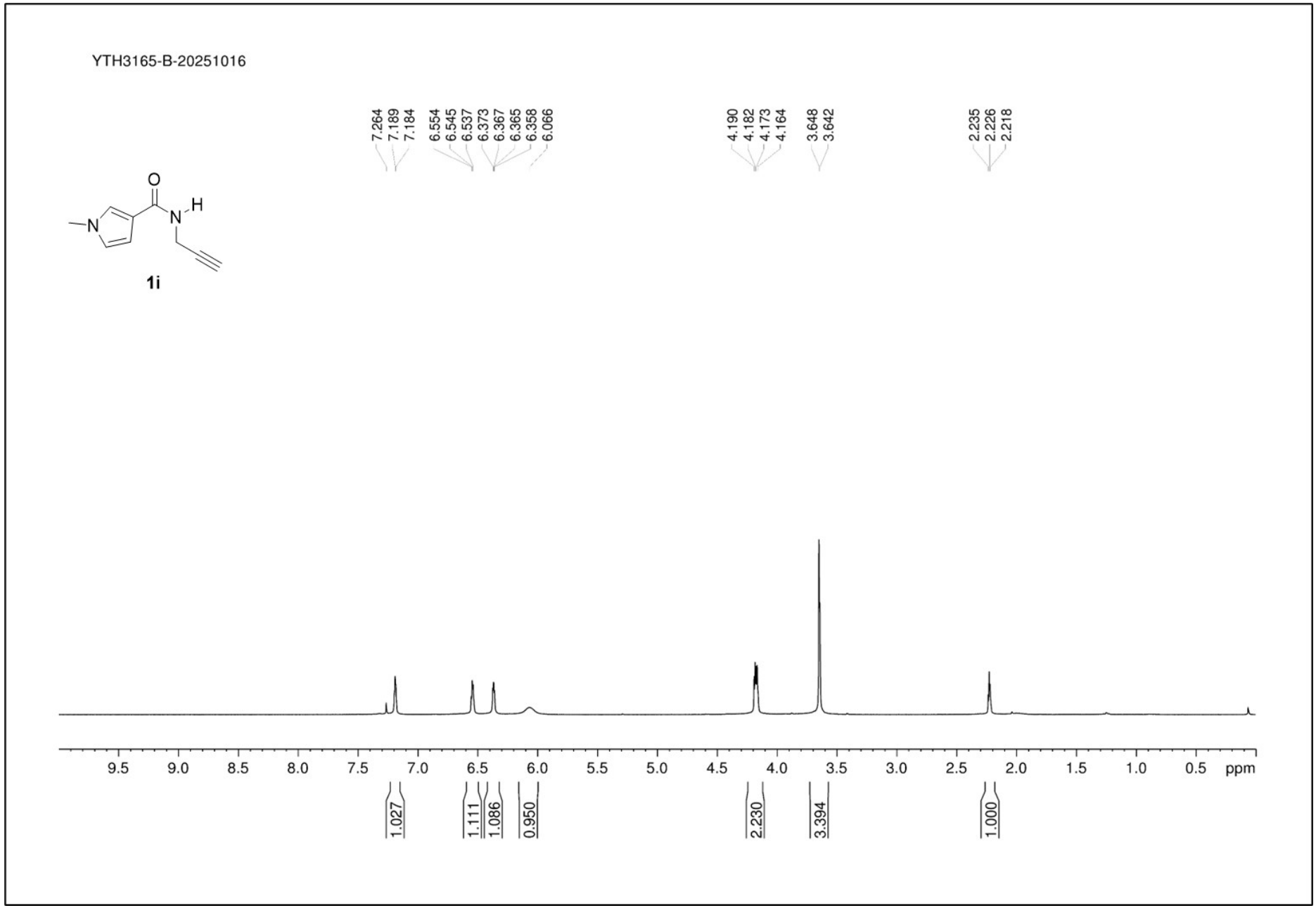
^{1}H NMR (300 MHz, $CDCl_3$) of **1h**



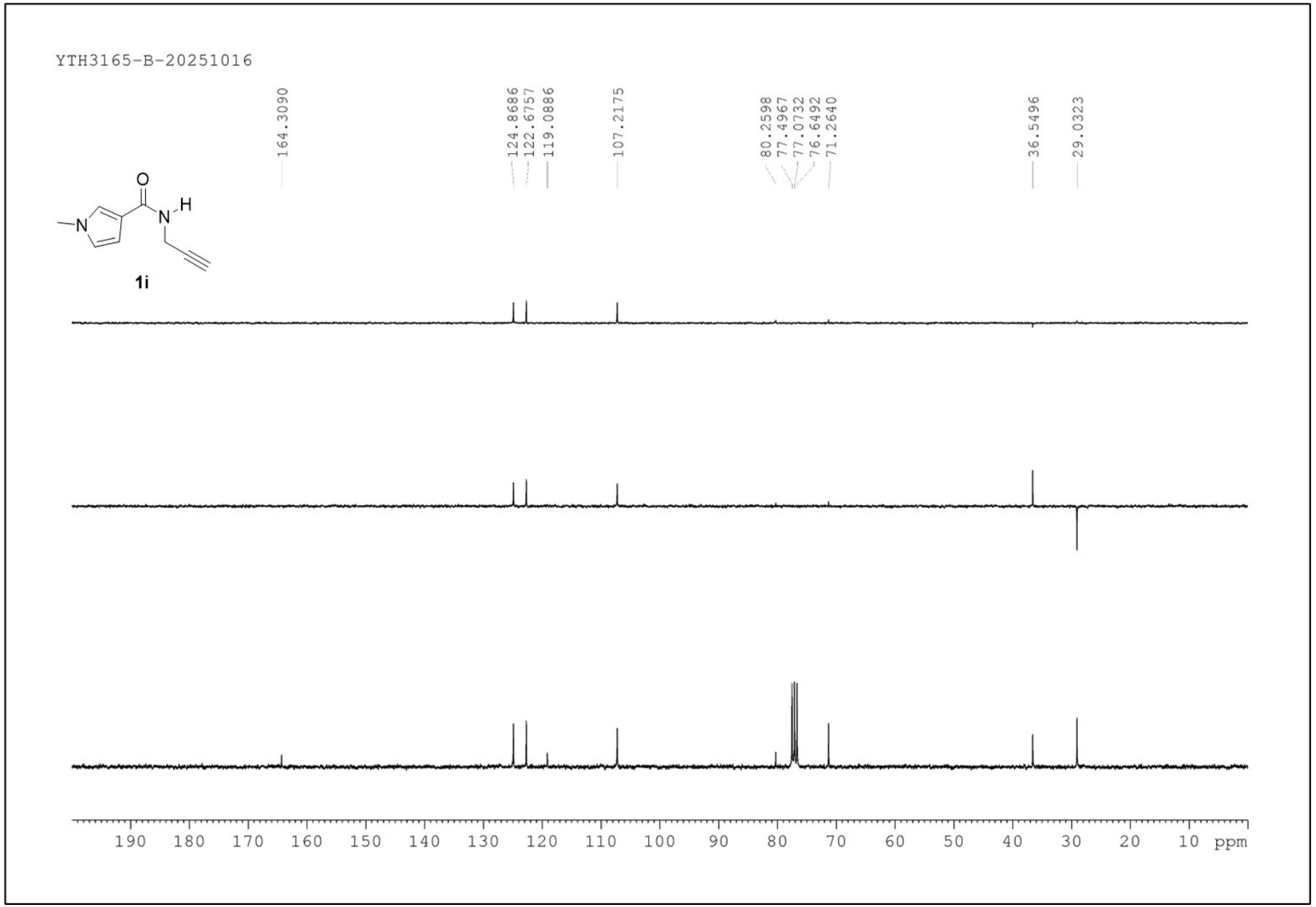
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1h**



HRMS (ESI$^+$) of **1h**



^{1}H NMR (300 MHz, $CDCl_3$) of **1i**



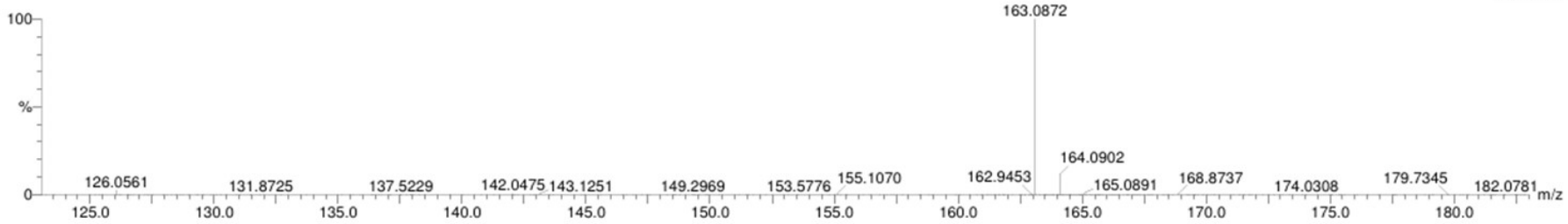
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **1i**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

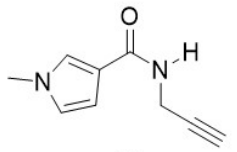
Monoisotopic Mass, Even Electron Ions
115 formula(e) evaluated with 28 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3165B
251210FJU05 90 (0.888) Cm (88:92-(70:76+116:122))

1: TOF MS ES+
1.15e+005



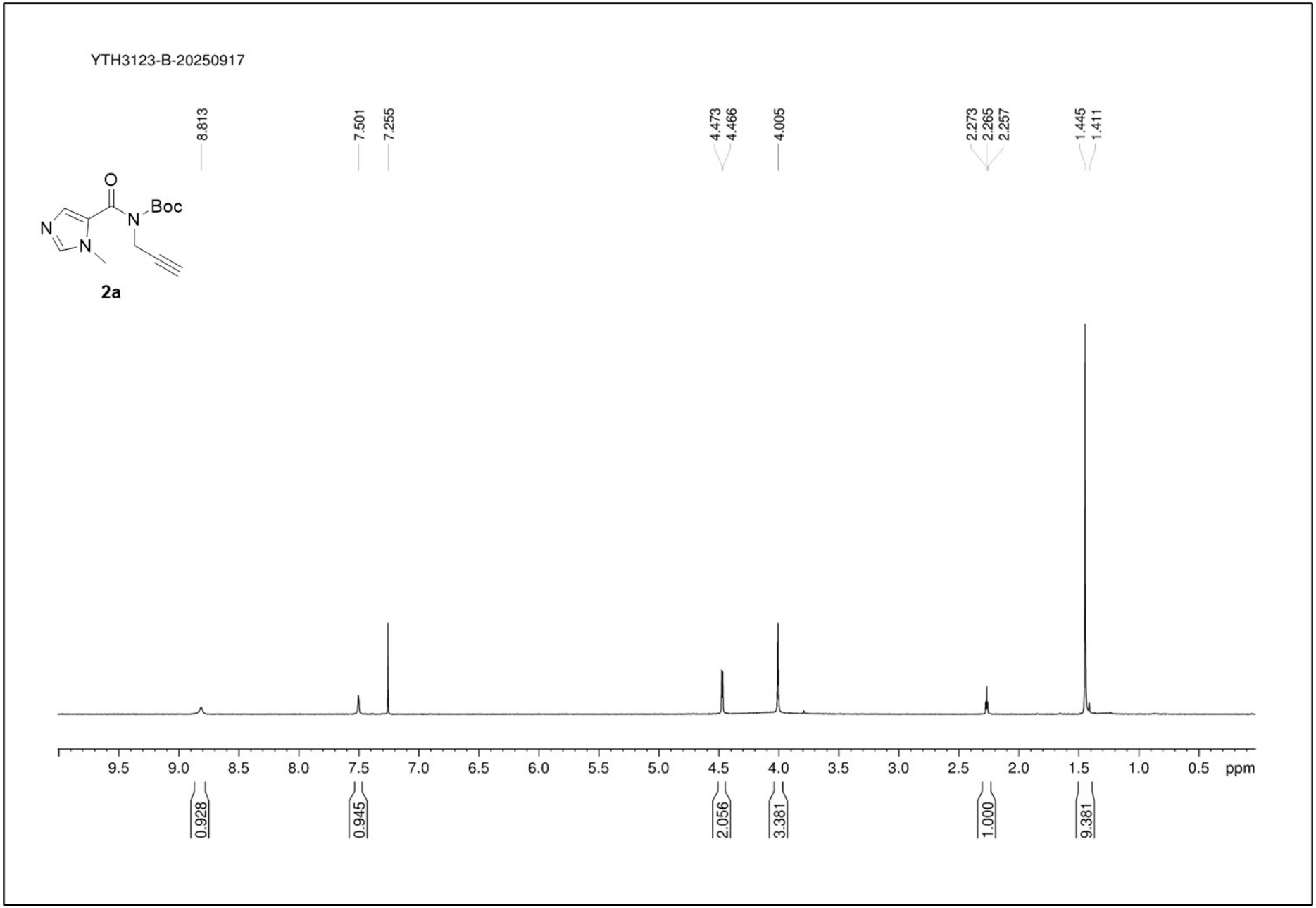
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
163.0872	100.00	163.0871	0.1	0.6	5.5	C9 H11 N2 O
		163.0831	4.1	25.1	1.5	C4 H11 N4 O3
		163.0930	-5.8	-35.6	-3.5	C2 H15 N2 O6
		163.0943	-7.1	-43.5	1.5	C3 H11 N6 O2
		163.0719	15.3	93.8	1.5	C5 H11 N2 O4
		163.1042	-17.0	-104.2	-3.5	C H15 N4 O5
		163.0692	18.0	110.4	2.5	C H7 N8 O2
		163.1056	-18.4	-112.8	1.5	C2 H11 N8 O
		163.1083	-21.1	-129.4	0.5	C6 H15 N2 O3
		163.0620	25.2	154.5	6.5	C7 H7 N4 O
		163.0580	29.2	179.0	2.5	C2 H7 N6 O3
		163.0566	30.6	187.6	-2.5	C H11 N2 O7
		163.1195	-32.3	-198.1	0.5	C5 H15 N4 O2
		163.0508	36.4	223.2	6.5	C8 H7 N2 O2
		163.0467	40.5	248.3	2.5	C3 H7 N4 O4
		163.1294	-42.2	-258.8	-4.5	C3 H19 N2 O5
		163.1307	-43.5	-266.7	0.5	C4 H15 N6 O
		163.0368	50.4	309.0	7.5	C5 H3 N6 O
		163.0355	51.7	317.0	2.5	C4 H7 N2 O5
		163.1406	-53.4	-327.4	-4.5	C2 H19 N4 O4
		163.1447	-57.5	-352.6	-0.5	C7 H19 N2 O2
		163.0256	61.6	377.7	7.5	C6 H3 N4 O2
		163.1519	-64.7	-396.7	-4.5	C H19 N6 O3
		163.0216	65.6	402.2	3.5	C H3 N6 O4
		163.1559	-68.7	-421.2	-0.5	C6 H19 N4 O

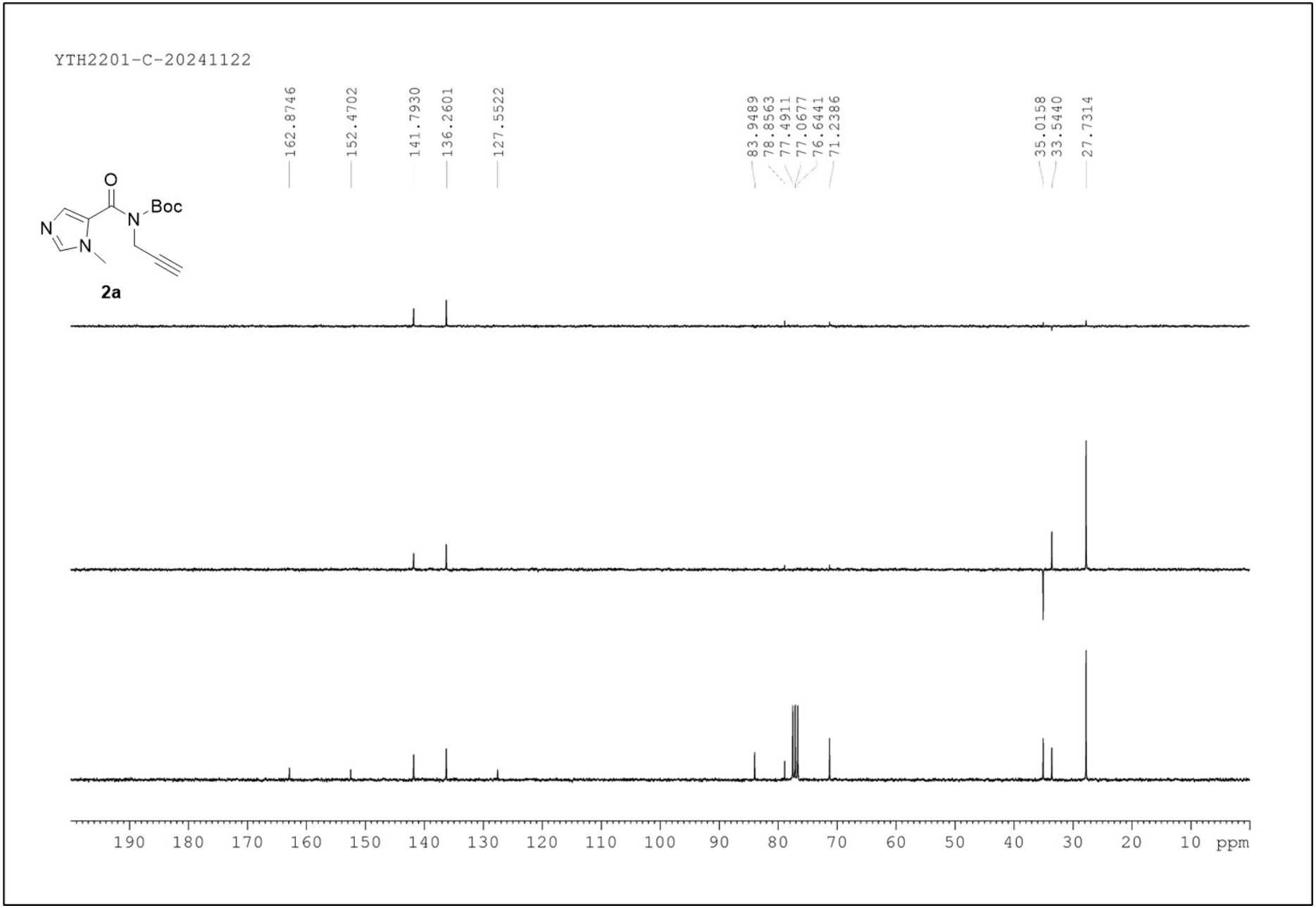


1i

HRMS (ESI$^+$) of **1i**



^{1}H NMR (300 MHz, $CDCl_3$) of **2a**



$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2a**

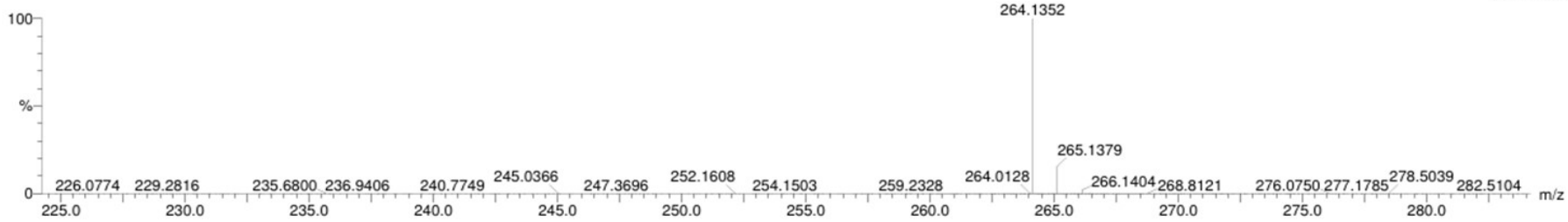
Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
354 formula(e) evaluated with 132 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-20
YTH3035B
250919FJU20 163 (1.613) Cm (163:171-(149:155+192:198))

1: TOF MS ES+
2.70e+005



Minimum:	80.00				-10.0	
Maximum:	100.00		10.0	500.0	100.0	
Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
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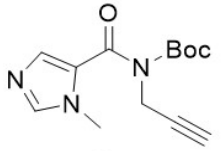
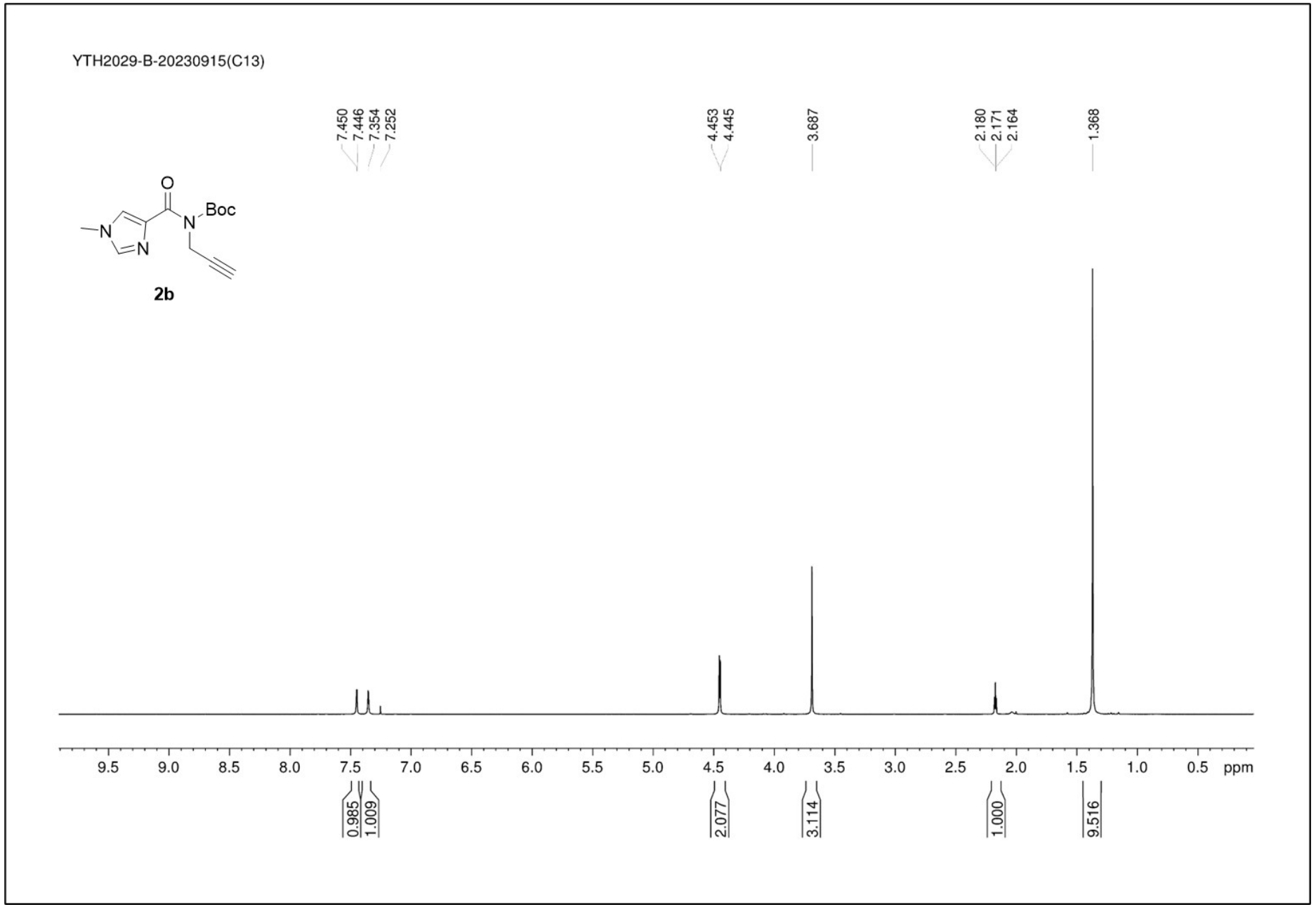
		264.1367	-1.5	-5.7	-6.5	C H22 N5 O10
		264.1380	-2.8	-10.6	-1.5	C2 H18 N9 O6
		264.1321	3.1	11.7	7.5	C9 H14 N9 O
		264.1388	-3.6	-13.6	10.5	C18 H18 N O
		264.1308	4.4	16.7	2.5	C8 H18 N5 O5
		264.1407	-5.5	-20.8	-2.5	C6 H22 N3 O8
		264.1295	5.7	21.6	-2.5	C7 H22 N O9
		264.1420	-6.8	-25.7	2.5	C7 H18 N7 O4
		264.1268	8.4	31.8	-1.5	C3 H18 N7 O7
		264.1447	-9.5	-36.0	1.5	C11 H22 N O6
		264.1254	9.8	37.1	-6.5	C2 H22 N3 O11
		264.1460	-10.8	-40.9	6.5	C12 H18 N5 O2
		264.1236	11.6	43.9	6.5	C14 H18 N O4
		264.1209	14.3	54.1	7.5	C10 H14 N7 O2
		264.1506	-15.4	-58.3	-7.5	C4 H26 N O11
		264.1196	15.6	59.1	2.5	C9 H18 N3 O6
		264.1519	-16.7	-63.2	-2.5	C5 H22 N5 O7
		264.1533	-18.1	-68.5	2.5	C6 H18 N9 O3
		264.1169	18.3	69.3	3.5	C5 H14 N9 O4
		264.1155	19.7	74.6	-1.5	C4 H18 N5 O8
		264.1559	-20.7	-78.4	1.5	C10 H22 N3 O5
		264.1142	21.0	79.5	-6.5	C3 H22 N O12
		264.1137	21.5	81.4	11.5	C16 H14 N3 O
		264.1573	-22.1	-83.7	6.5	C11 H18 N7 O

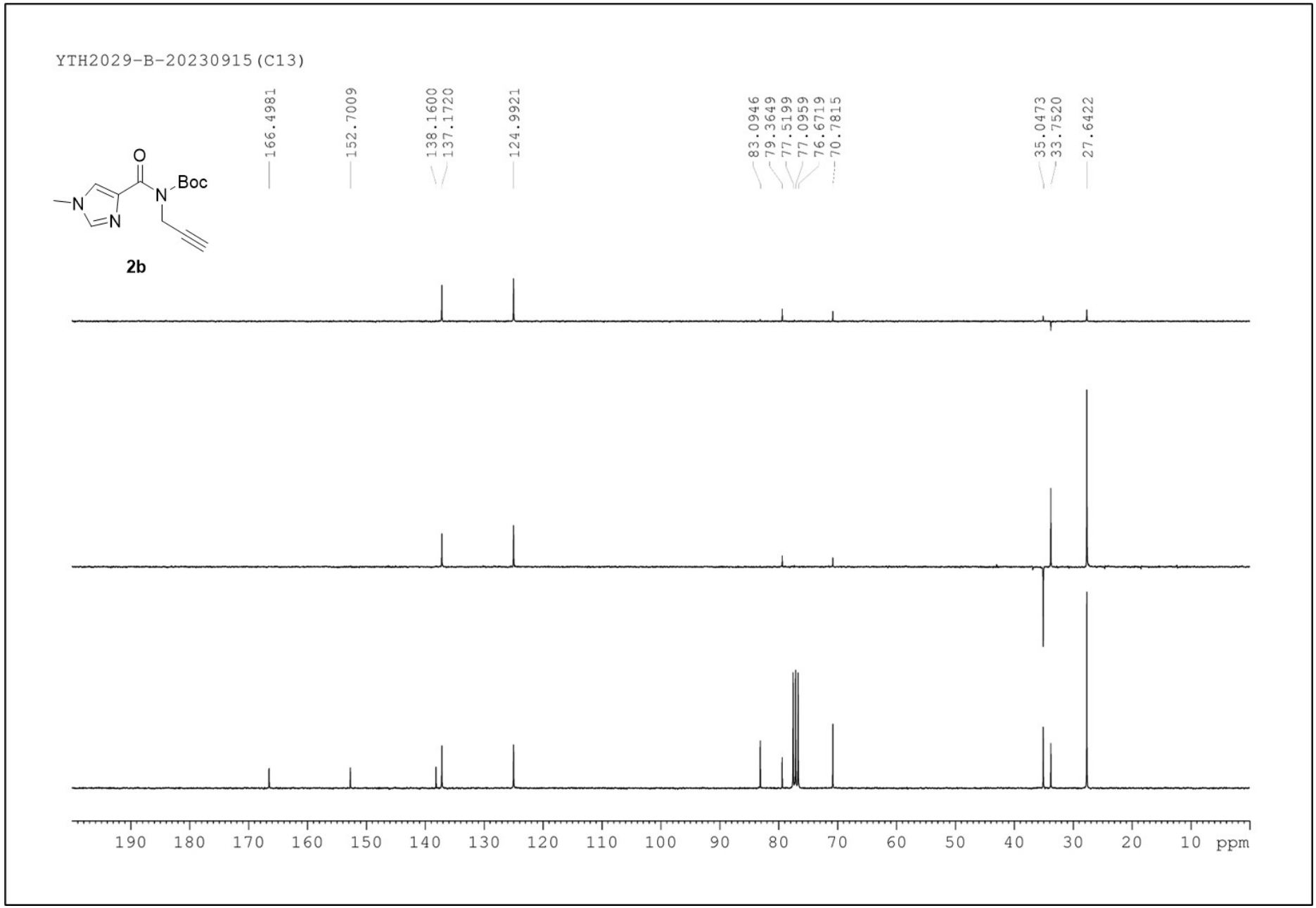


2a

HRMS (ESI$^+$) of **2a**



^{1}H NMR (300 MHz, $CDCl_3$) of **2b**



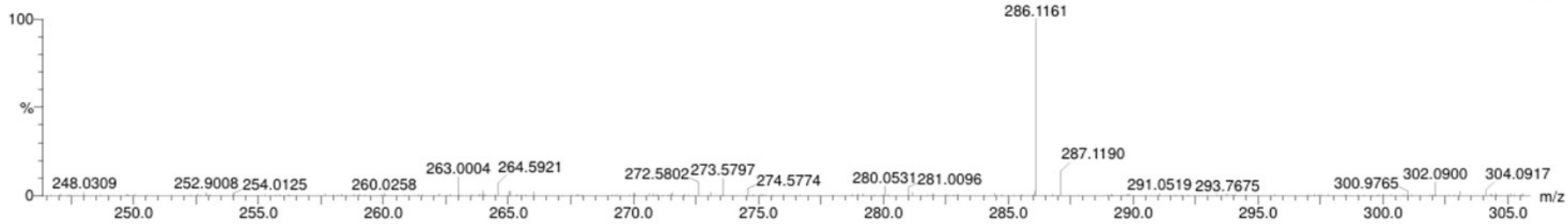
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2b**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

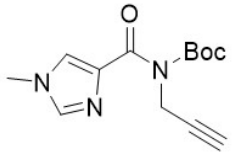
Monoisotopic Mass, Even Electron Ions
697 formula(e) evaluated with 255 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Na: 0-1
YTH2029B
250919FJU28-3 179 (1.750) Cm (178:181-(160:166+207:214))

1: TOF MS ES+
8.98e+003



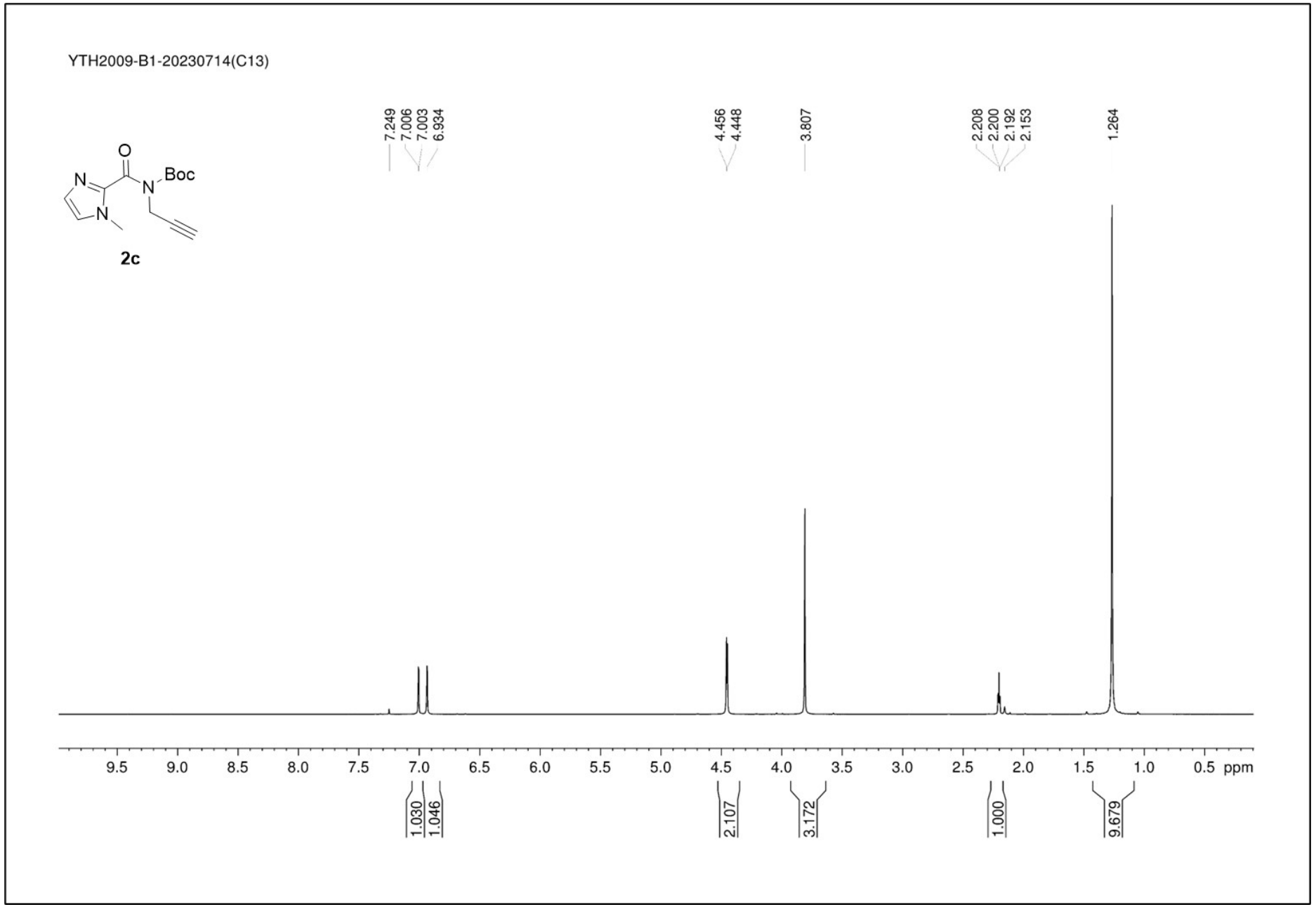
Minimum:	80.00			-10.0	
Maximum:	100.00	10.0	500.0	100.0	

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
286.1161	100.00	286.1168	-0.7	-2.4	6.5	C13 H17 N3 O3 Na
		286.1055	10.6	37.0	6.5	C14 H17 N O4 Na
		286.1141	2.0	7.0	7.5	C9 H13 N9 O Na
		286.1186	-2.5	-8.7	-6.5	C H21 N5 O10 Na
		286.1127	3.4	11.9	2.5	C8 H17 N5 O5 Na
		286.1240	-7.9	-27.6	2.5	C7 H17 N7 O4 Na
		286.1087	7.4	25.9	-1.5	C3 H17 N7 O7 Na
		286.1199	-3.8	-13.3	-1.5	C2 H17 N9 O6 Na
		286.1226	-6.5	-22.7	-2.5	C6 H21 N3 O8 Na
		286.1114	4.7	16.4	-2.5	C7 H21 N O9 Na
		286.1208	-4.7	-16.4	10.5	C18 H17 N O Na
		286.1280	-11.9	-41.6	6.5	C12 H17 N5 O2 Na
		286.1267	-10.6	-37.0	1.5	C11 H21 N O6 Na
		286.1210	-4.9	-17.1	-3.5	C3 H20 N5 O10
		286.1111	5.0	17.5	1.5	C5 H16 N7 O7
		286.1224	-6.3	-22.0	1.5	C4 H16 N9 O6
		286.1232	-7.1	-24.8	13.5	C20 H16 N O
		286.1079	8.2	28.7	9.5	C16 H16 N O4
		286.1192	-3.1	-10.8	9.5	C15 H16 N3 O3
		286.1250	-8.9	-31.1	0.5	C8 H20 N3 O8
		286.1138	2.3	8.0	0.5	C9 H20 N O9
		286.1264	-10.3	-36.0	5.5	C9 H16 N7 O4
		286.1151	1.0	3.5	5.5	C10 H16 N5 O5
		286.1052	10.9	38.1	10.5	C12 H12 N7 O2
		286.1165	-0.4	-1.4	10.5	C11 H12 N9 O

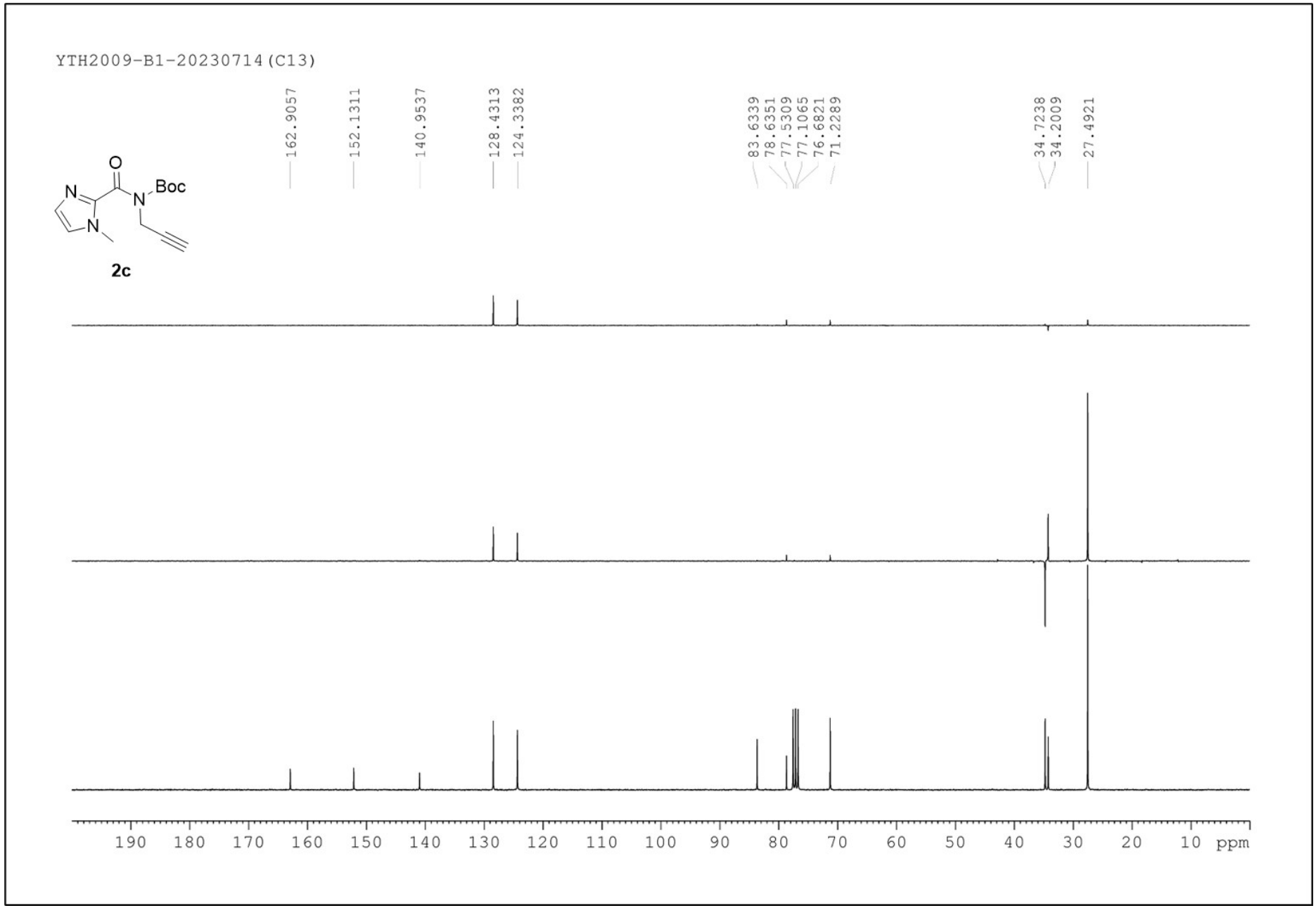


2b

HRMS (ESI$^+$) of **2b**



^{1}H NMR (300 MHz, $CDCl_3$) of **2c**



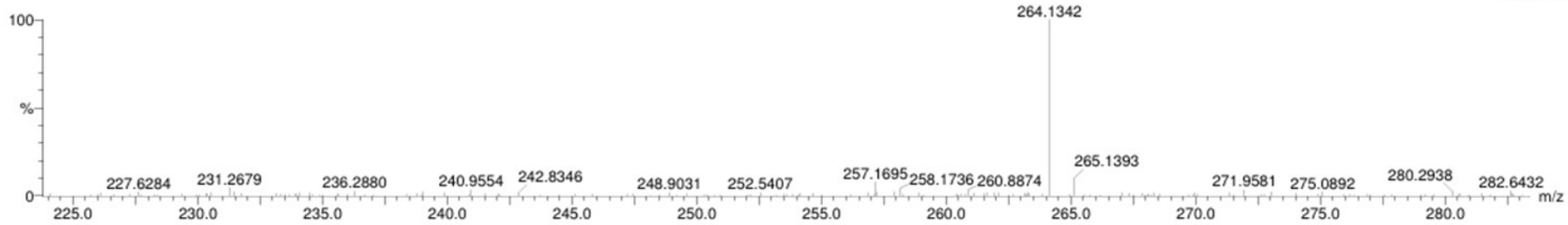
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2c**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

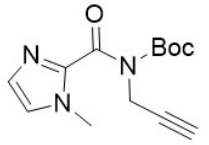
Monoisotopic Mass, Even Electron Ions
323 formula(e) evaluated with 122 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3184B
260106FJU04-3 227 (2.221) Cm (225:231-(210:216+253:260))

1: TOF MS ES+
3.37e+003



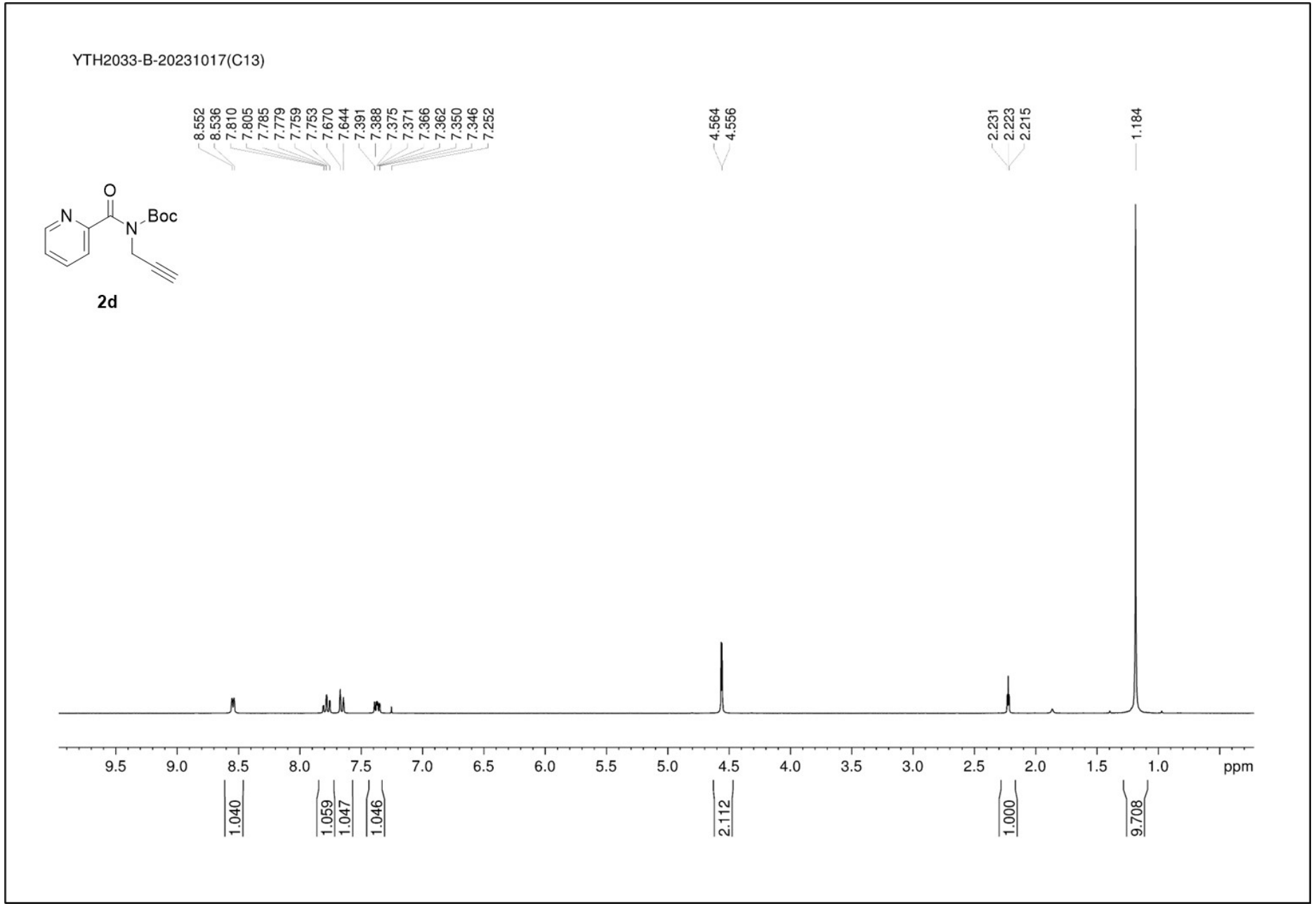
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
264.1342	100.00	264.1348	-0.6	-2.3	6.5	C13 H18 N3 O3
		264.1321	2.1	8.0	7.5	C9 H14 N9 O
		264.1367	-2.5	-9.5	-6.5	C H22 N5 O10
		264.1308	3.4	12.9	2.5	C8 H18 N5 O5
		264.1380	-3.8	-14.4	-1.5	C2 H18 N9 O6
		264.1388	-4.6	-17.4	10.5	C18 H18 N O
		264.1295	4.7	17.8	-2.5	C7 H22 N O9
		264.1407	-6.5	-24.6	-2.5	C6 H22 N3 O8
		264.1268	7.4	28.0	-1.5	C3 H18 N7 O7
		264.1420	-7.8	-29.5	2.5	C7 H18 N7 O4
		264.1447	-10.5	-39.8	1.5	C11 H22 N O6
		264.1236	10.6	40.1	6.5	C14 H18 N O4
		264.1460	-11.8	-44.7	6.5	C12 H18 N5 O2
		264.1209	13.3	50.4	7.5	C10 H14 N7 O2
		264.1196	14.6	55.3	2.5	C9 H18 N3 O6
		264.1169	17.3	65.5	3.5	C5 H14 N9 O4
		264.1519	-17.7	-67.0	-2.5	C5 H22 N5 O7
		264.1155	18.7	70.8	-1.5	C4 H18 N5 O8
		264.1533	-19.1	-72.3	2.5	C6 H18 N9 O3
		264.1137	20.5	77.6	11.5	C16 H14 N3 O
		264.1559	-21.7	-82.2	1.5	C10 H22 N3 O5
		264.1573	-23.1	-87.5	6.5	C11 H18 N7 O
		264.1097	24.5	92.8	7.5	C11 H14 N5 O3
		264.1600	-25.8	-97.7	5.5	C15 H22 N O3
		264.1083	25.9	98.1	2.5	C10 H18 N O7

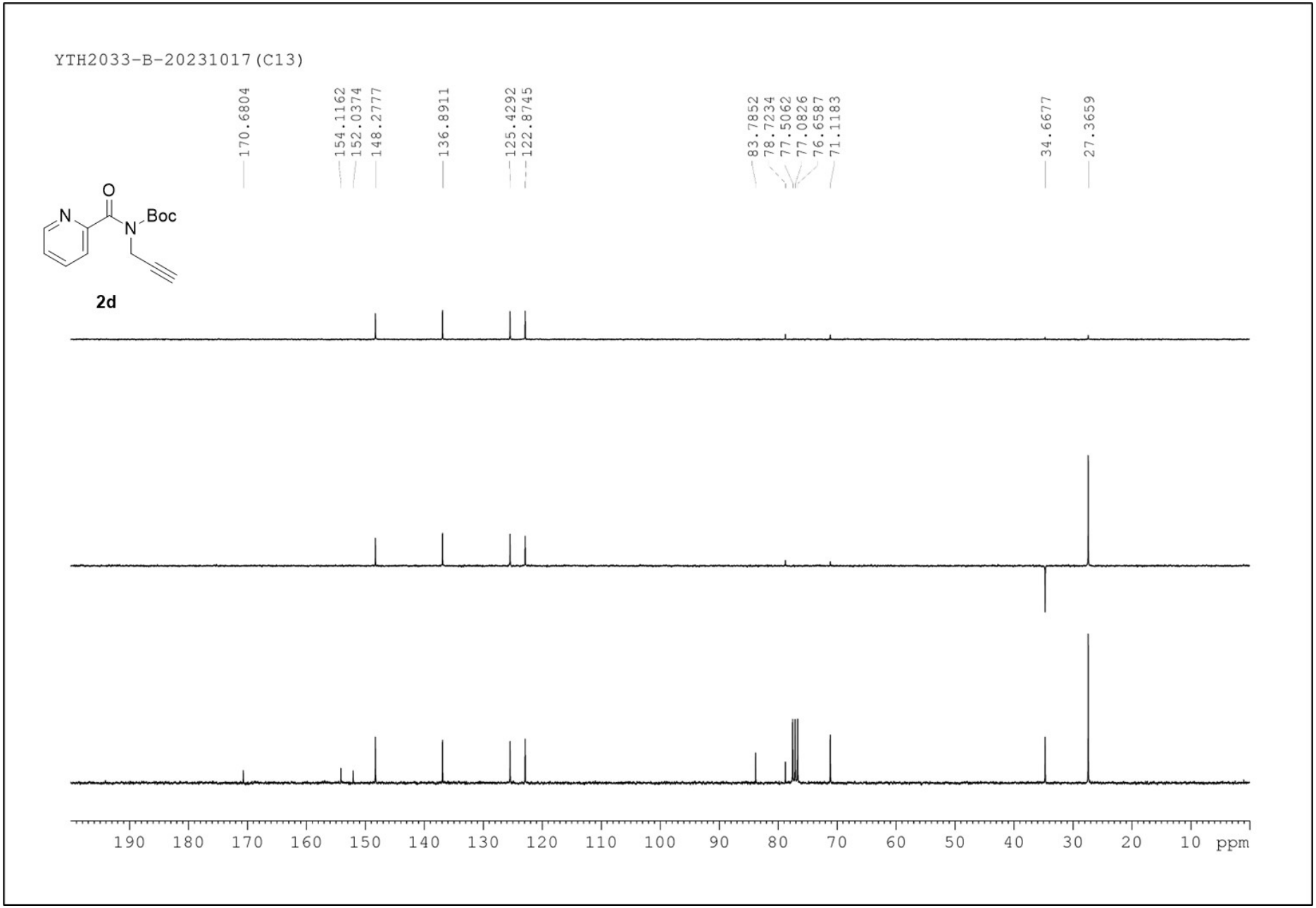


2c

HRMS (ESI$^+$) of **2c**



^{1}H NMR (300 MHz, $CDCl_3$) of **2d**



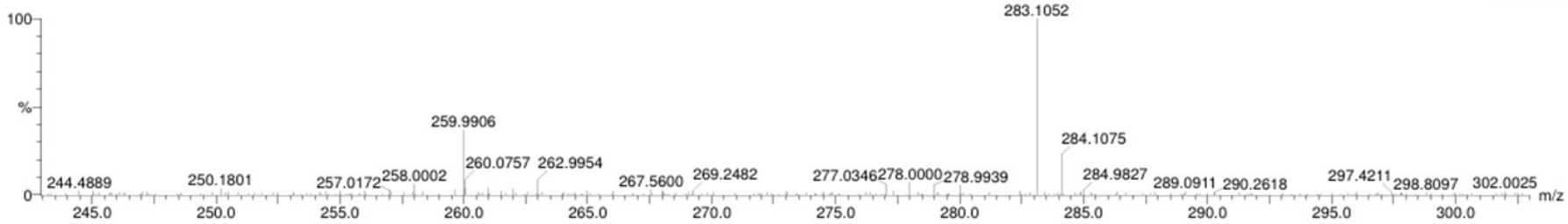
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2d**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

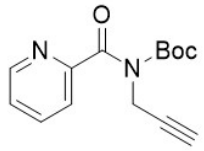
Monoisotopic Mass, Even Electron Ions
683 formula(e) evaluated with 226 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Na: 0-1
YTH3181D
260108FJU02-4 253 (2.483) Cm (252:262-(236:245+293:302))

1: TOF MS ES+
4.29e+003



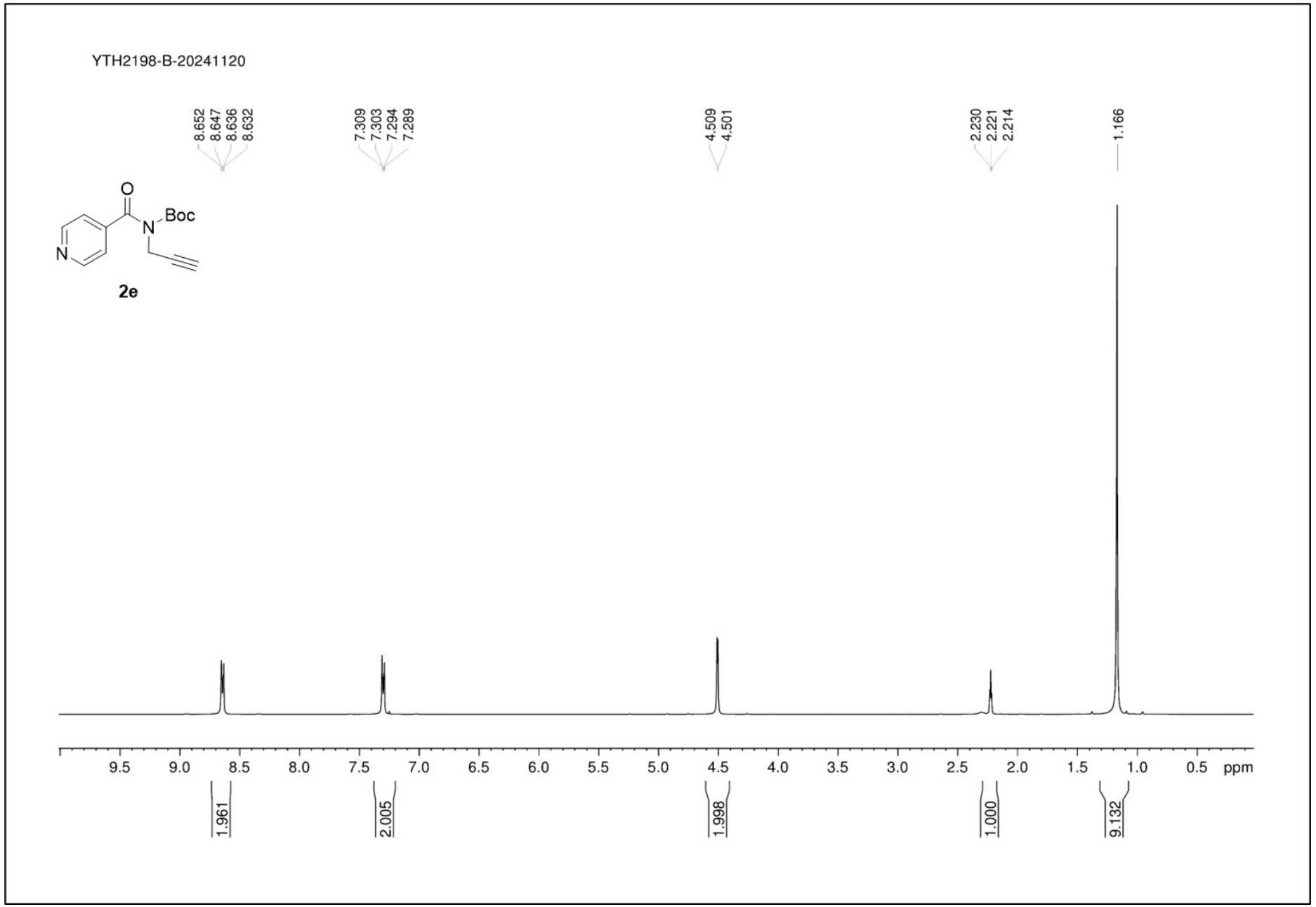
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
283.1052	100.00	283.1059	-0.7	-2.5	7.5	C14 H16 N2 O3 Na
		283.1117	-6.5	-23.0	-1.5	C7 H20 N2 O8 Na
		283.1141	-8.9	-31.4	1.5	C9 H19 N2 O8
		283.1083	-3.1	-10.9	10.5	C16 H15 N2 O3
		283.0930	12.2	43.1	6.5	C12 H15 N2 O6
		283.1018	3.4	12.0	3.5	C9 H16 N4 O5 Na
		283.1077	-2.5	-8.8	-5.5	C2 H20 N4 O10 Na
		283.1171	-11.9	-42.0	7.5	C13 H16 N4 O2 Na
		283.1101	-4.9	-17.3	-2.5	C4 H19 N4 O10
		283.1042	1.0	3.5	6.5	C11 H15 N4 O5
		283.1195	-14.3	-50.5	10.5	C15 H15 N4 O2
		283.0943	10.9	38.5	11.5	C13 H11 N6 O2
		283.1131	-7.9	-27.9	3.5	C8 H16 N6 O4 Na
		283.0978	7.4	26.1	-0.5	C4 H16 N6 O7 Na
		283.1002	5.0	17.7	2.5	C6 H15 N6 O7
		283.1155	-10.3	-36.4	6.5	C10 H15 N6 O4
		283.0919	13.3	47.0	8.5	C11 H12 N6 O2 Na
		283.1189	-13.7	-48.4	-5.5	C H20 N6 O9 Na
		283.0962	9.0	31.8	-1.5	C H15 N8 O9
		283.1032	2.0	7.1	8.5	C10 H12 N8 O Na
		283.1090	-3.8	-13.4	-0.5	C3 H16 N8 O6 Na
		283.1056	-0.4	-1.4	11.5	C12 H11 N8 O
		283.1115	-6.3	-22.3	2.5	C5 H15 N8 O6
		283.0992	6.0	21.2	4.5	C5 H12 N10 O3 Na
		283.1016	3.6	12.7	7.5	C7 H11 N10 O3

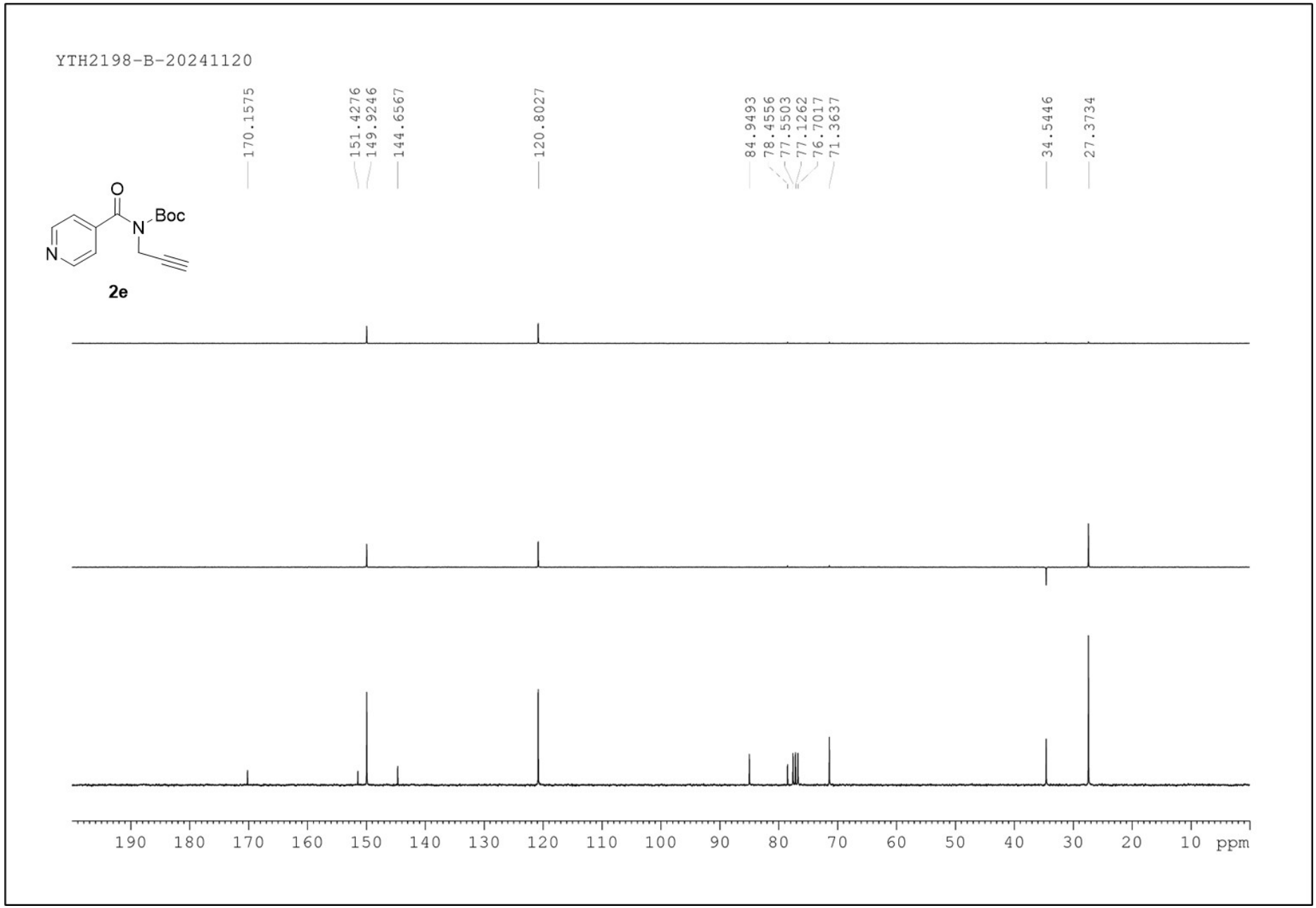


2d

HRMS (ESI$^+$) of **2d**



^{1}H NMR (300 MHz, $CDCl_3$) of **2e**



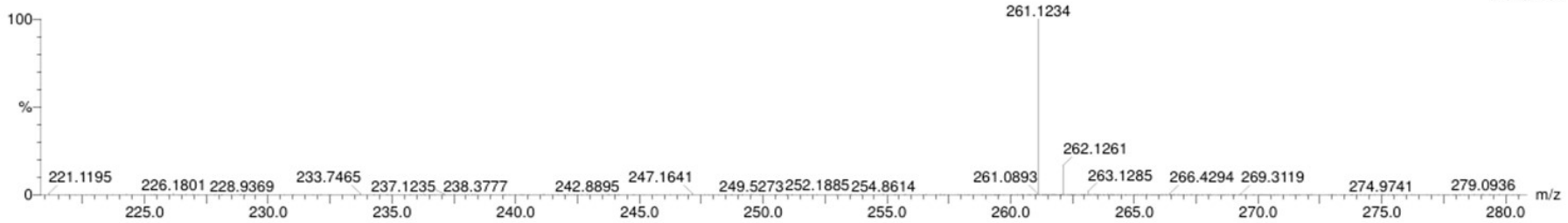
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2e**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

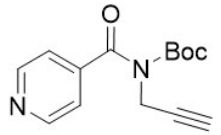
Monoisotopic Mass, Even Electron Ions
326 formula(e) evaluated with 113 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3032B (YTH2198B)
250924FJU40 235 (2.309) Cm (233:240-(217:224+265:270))

1: TOF MS ES+
9.89e+004



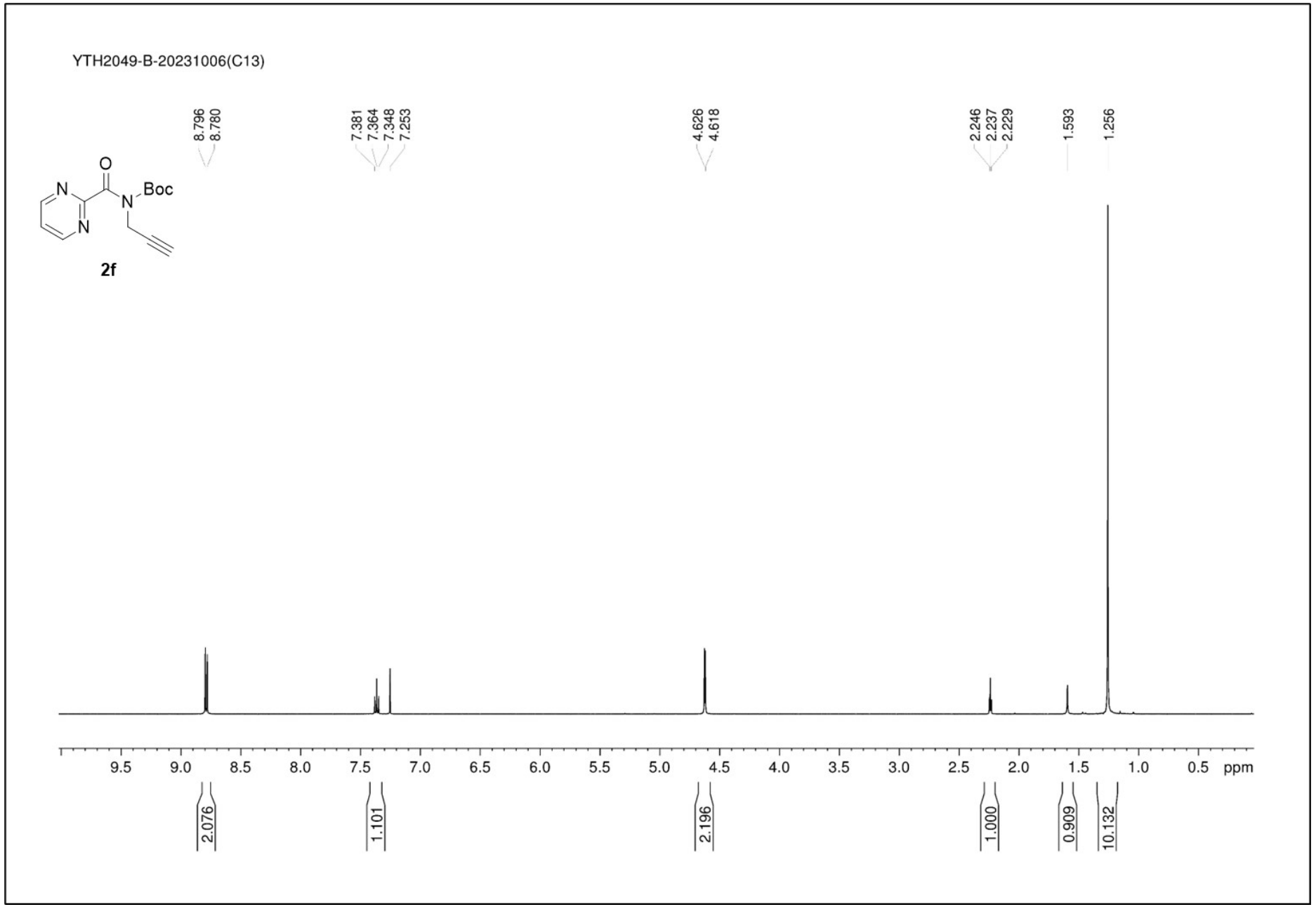
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
261.1234	100.00	261.1239	-0.5	-1.9	7.5	C14 H17 N2 O3
		261.1212	2.2	8.4	8.5	C10 H13 N8 O
		261.1258	-2.4	-9.2	-5.5	C2 H21 N4 O10
		261.1199	3.5	13.4	3.5	C9 H17 N4 O5
		261.1271	-3.7	-14.2	-0.5	C3 H17 N8 O6
		261.1172	6.2	23.7	4.5	C5 H13 N10 O3
		261.1298	-6.4	-24.5	-1.5	C7 H21 N2 O8
		261.1159	7.5	28.7	-0.5	C4 H17 N6 O7
		261.1311	-7.7	-29.5	3.5	C8 H17 N6 O4
		261.1352	-11.8	-45.2	7.5	C13 H17 N4 O2
		261.1100	13.4	51.3	8.5	C11 H13 N6 O2
		261.1370	-13.6	-52.1	-5.5	C H21 N6 O9
		261.1087	14.7	56.3	3.5	C10 H17 N2 O6
		261.1383	-14.9	-57.1	-0.5	C2 H17 N10 O5
		261.1060	17.4	66.6	4.5	C6 H13 N8 O4
		261.1410	-17.6	-67.4	-1.5	C6 H21 N4 O7
		261.1046	18.8	72.0	-0.5	C5 H17 N4 O8
		261.1424	-19.0	-72.8	3.5	C7 H17 N8 O3
		261.1028	20.6	78.9	12.5	C17 H13 N2 O
		261.1020	21.4	82.0	0.5	C H13 N10 O6
		261.1450	-21.6	-82.7	2.5	C11 H21 N2 O5
		261.1464	-23.0	-88.1	7.5	C12 H17 N6 O
		261.0988	24.6	94.2	8.5	C12 H13 N4 O3
		261.0961	27.3	104.5	9.5	C8 H9 N10 O
		261.1509	-27.5	-105.3	-6.5	C4 H25 N2 O10

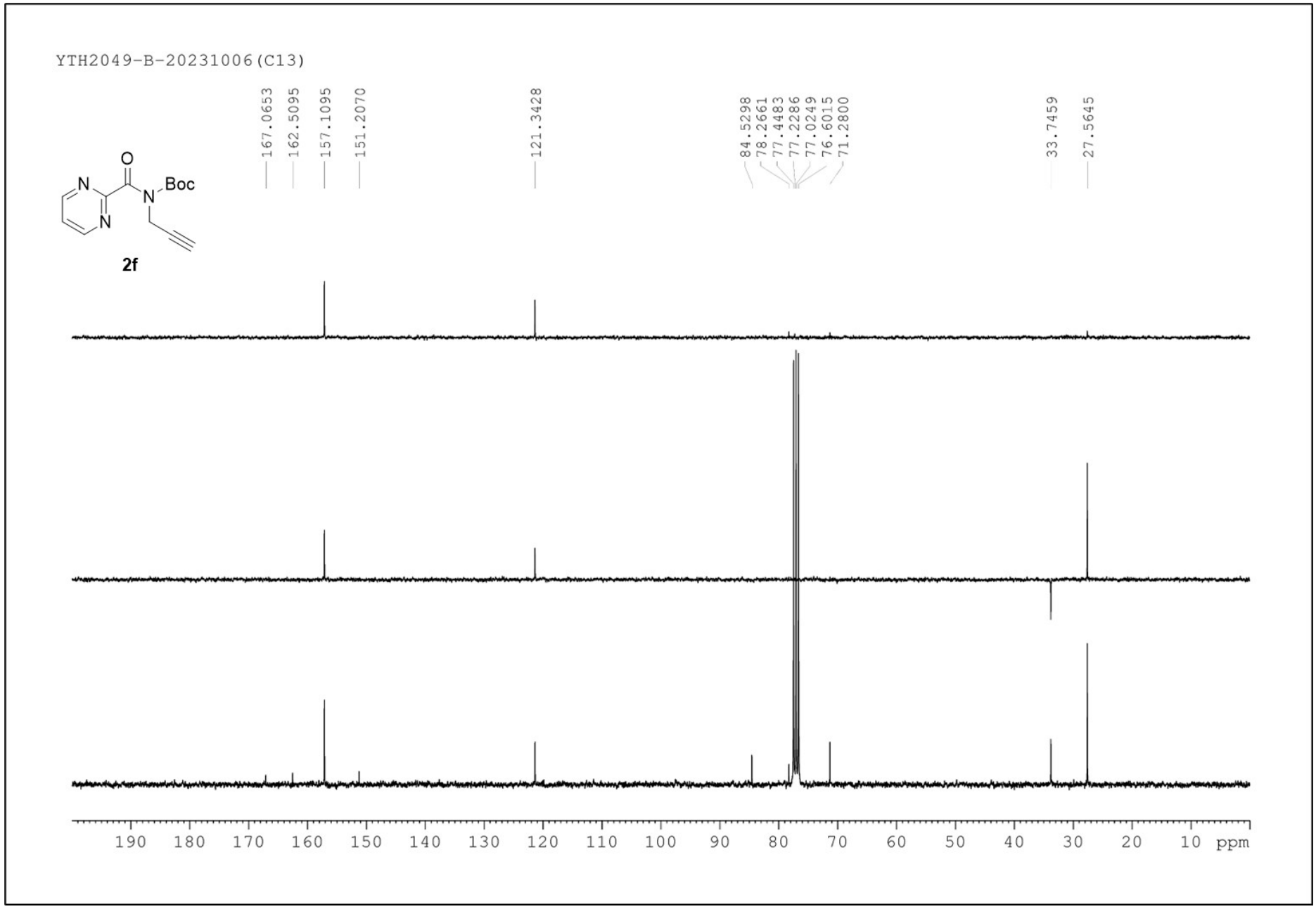


2e

HRMS (ESI$^+$) of **2e**



^{1}H NMR (300 MHz, $CDCl_3$) of **2f** (trace of water)



$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2f**

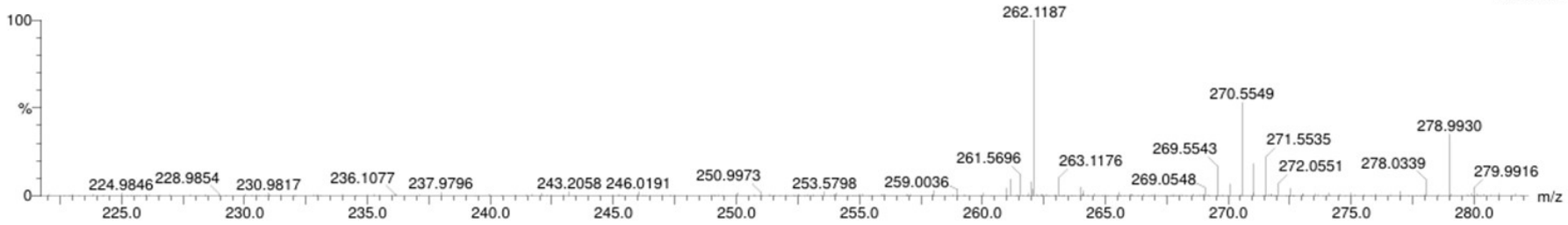
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
319 formula(e) evaluated with 114 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10

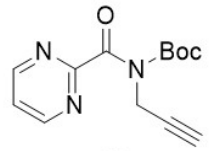
YTH3180B
260106FJU03 240 (2.352) Cm (240:246-(223:230+268:275))

1: TOF MS ES+
1.33e+004



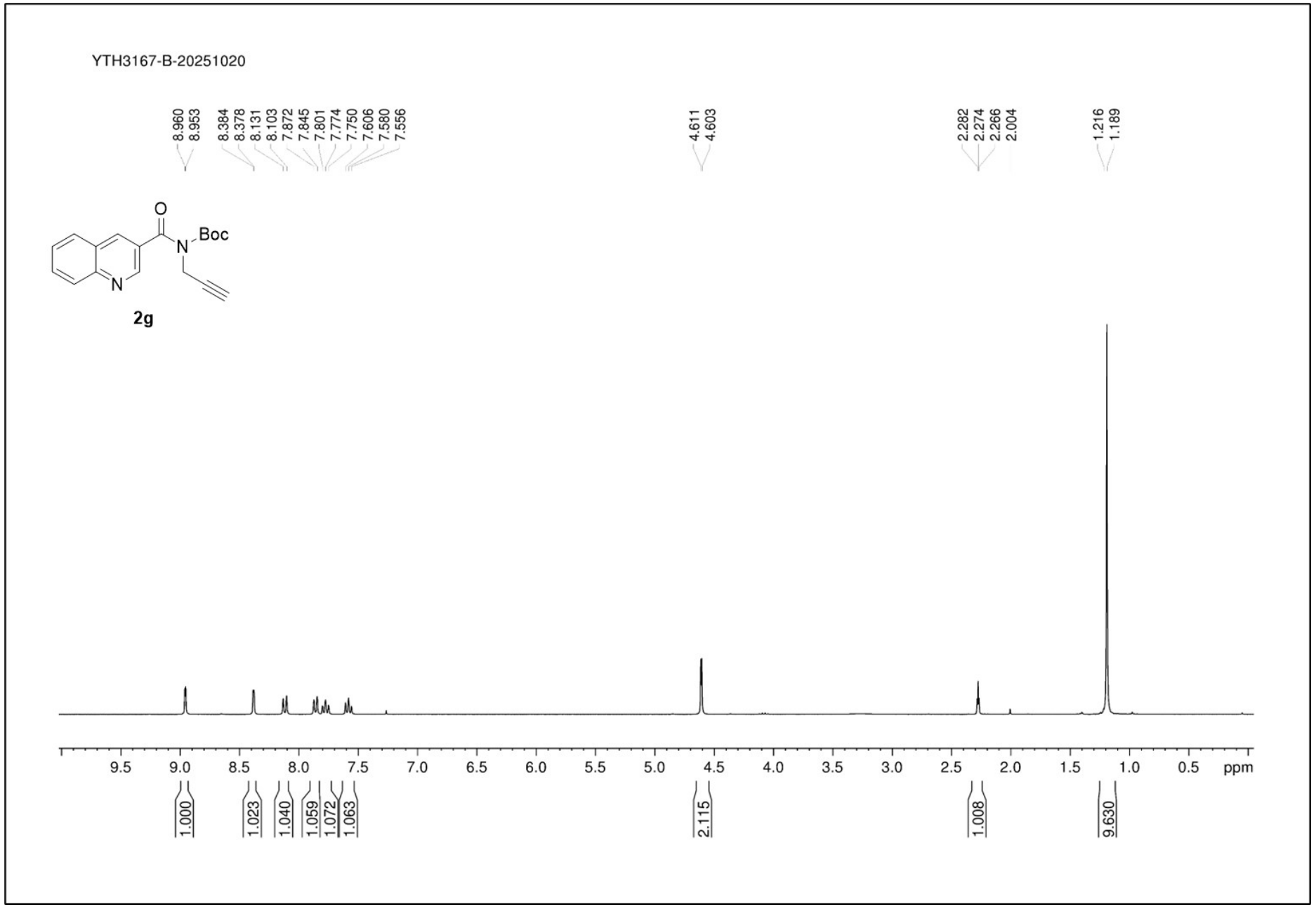
Minimum:	80.00			-10.0	
Maximum:	100.00	10.0	500.0	100.0	

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
262.1187	100.00	262.1192	-0.5	-1.9	7.5	C13 H16 N3 O3
		262.1165	2.2	8.4	8.5	C9 H12 N9 O
		262.1210	-2.3	-8.8	-5.5	C H20 N5 O10
		262.1151	3.6	13.7	3.5	C8 H16 N5 O5
		262.1224	-3.7	-14.1	-0.5	C2 H16 N9 O6
		262.1232	-4.5	-17.2	11.5	C18 H16 N O
		262.1138	4.9	18.7	-1.5	C7 H20 N O9
		262.1250	-6.3	-24.0	-1.5	C6 H20 N3 O8
		262.1111	7.6	29.0	-0.5	C3 H16 N7 O7
		262.1264	-7.7	-29.4	3.5	C7 H16 N7 O4
		262.1291	-10.4	-39.7	2.5	C11 H20 N O6
		262.1079	10.8	41.2	7.5	C14 H16 N O4
		262.1304	-11.7	-44.6	7.5	C12 H16 N5 O2
		262.1052	13.5	51.5	8.5	C10 H12 N7 O2
		262.1039	14.8	56.5	3.5	C9 H16 N3 O6
		262.1012	17.5	66.8	4.5	C5 H12 N9 O4
		262.1363	-17.6	-67.1	-1.5	C5 H20 N5 O7
		262.0999	18.8	71.7	-0.5	C4 H16 N5 O8
		262.1376	-18.9	-72.1	3.5	C6 H16 N9 O3
		262.0980	20.7	79.0	12.5	C16 H12 N3 O
		262.1403	-21.6	-82.4	2.5	C10 H20 N3 O5
		262.1416	-22.9	-87.4	7.5	C11 H16 N7 O
		262.0940	24.7	94.2	8.5	C11 H12 N5 O3
		262.1443	-25.6	-97.7	6.5	C15 H20 N O3
		262.0927	26.0	99.2	3.5	C10 H16 N O7

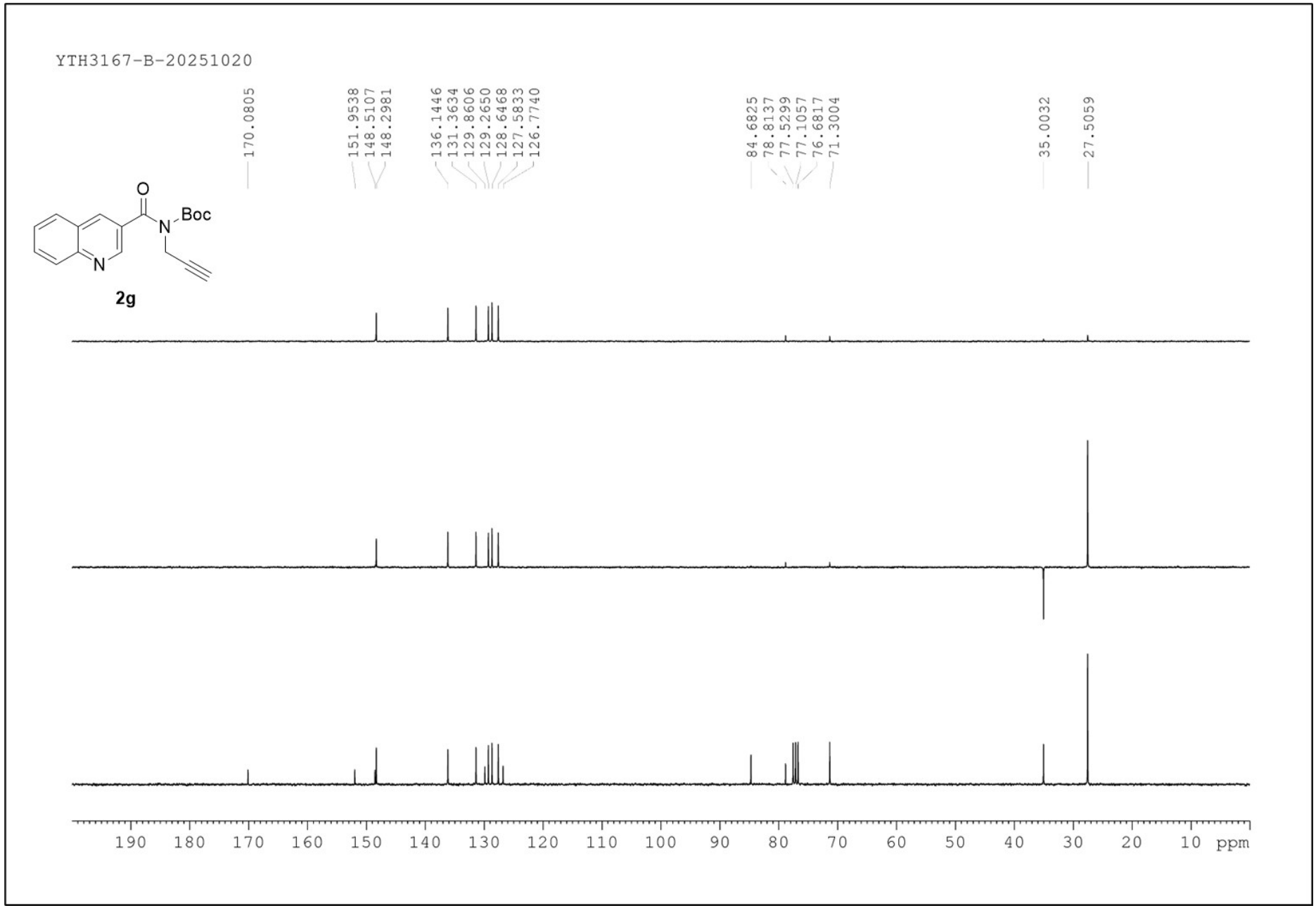


2f

HRMS (ESI$^+$) of **2f**



^{1}H NMR (300 MHz, $CDCl_3$) of **2g**



$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2g**

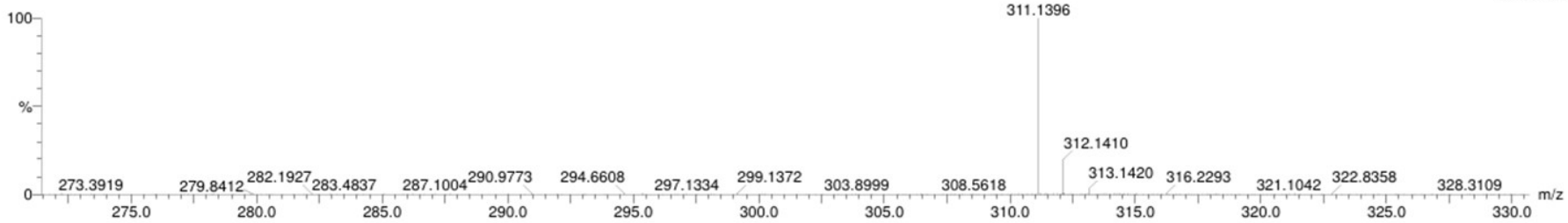
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
418 formula(e) evaluated with 147 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10

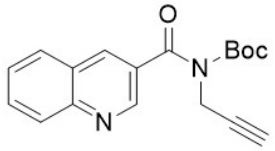
YTH2188B
251211FJU03-3 274 (2.683) Cm (273:276-(259:265+294:301))

1: TOF MS ES+
1.01e+005



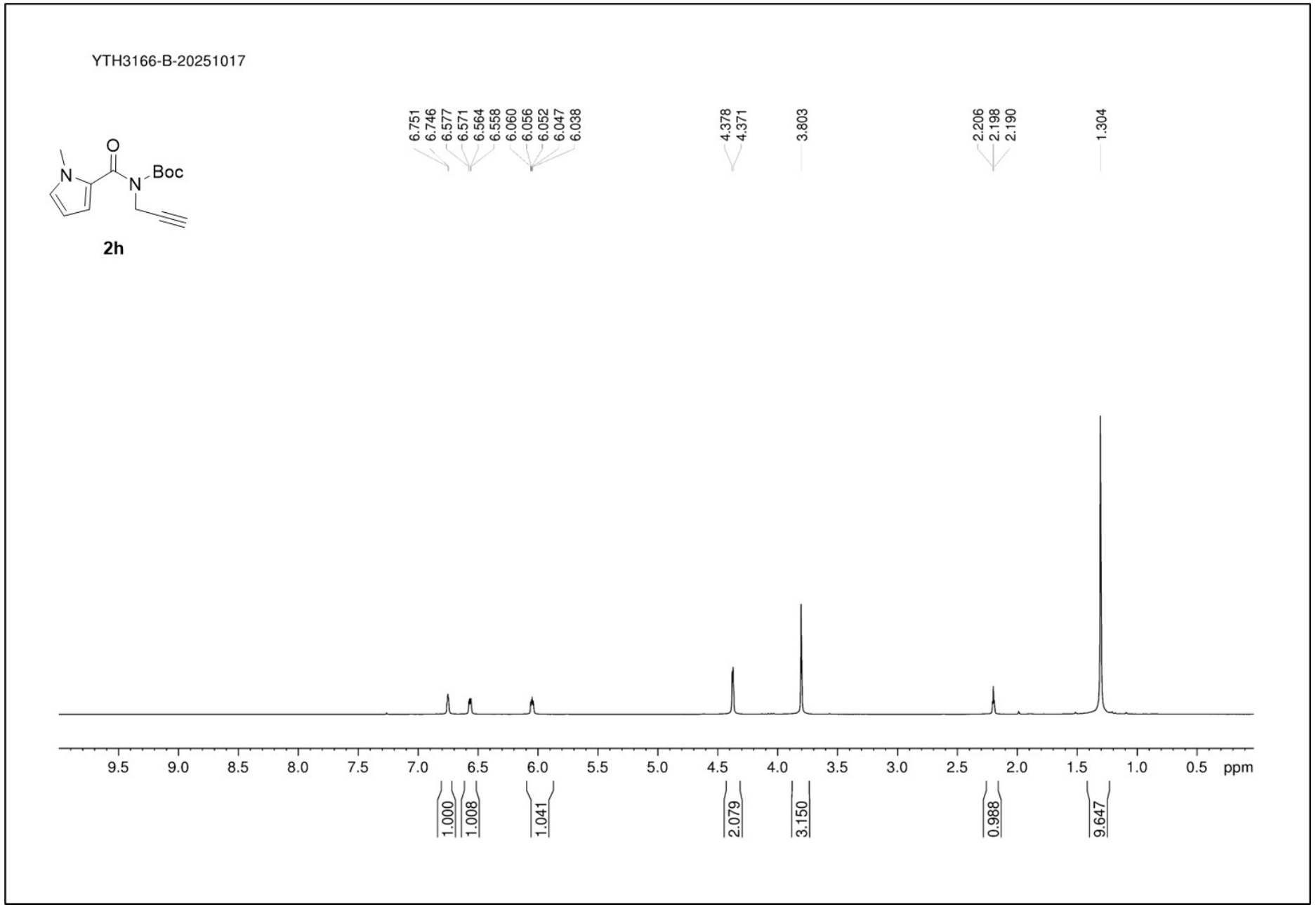
Minimum:	80.00			-10.0	
Maximum:	100.00	10.0	500.0	100.0	

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
311.1396	100.00	311.1396	0.0	0.0	10.5	C18 H19 N2 O3
		311.1387	0.9	2.9	-1.5	C2 H19 N10 O8
		311.1414	-1.8	-5.8	-2.5	C6 H23 N4 O10
		311.1369	2.7	8.7	11.5	C14 H15 N8 O
		311.1428	-3.2	-10.3	2.5	C7 H19 N8 O6
		311.1355	4.1	13.2	6.5	C13 H19 N4 O5
		311.1454	-5.8	-18.6	1.5	C11 H23 N2 O8
		311.1329	6.7	21.5	7.5	C9 H15 N10 O3
		311.1468	-7.2	-23.1	6.5	C12 H19 N6 O4
		311.1315	8.1	26.0	2.5	C8 H19 N6 O7
		311.1508	-11.2	-36.0	10.5	C17 H19 N4 O2
		311.1275	12.1	38.9	-1.5	C3 H19 N8 O9
		311.1527	-13.1	-42.1	-2.5	C5 H23 N6 O9
		311.1256	14.0	45.0	11.5	C15 H15 N6 O2
		311.1540	-14.4	-46.3	2.5	C6 H19 N10 O5
		311.1243	15.3	49.2	6.5	C14 H19 N2 O6
		311.1567	-17.1	-55.0	1.5	C10 H23 N4 O7
		311.1216	18.0	57.9	7.5	C10 H15 N8 O4
		311.1580	-18.4	-59.1	6.5	C11 H19 N8 O3
		311.1203	19.3	62.0	2.5	C9 H19 N4 O8
		311.1607	-21.1	-67.8	5.5	C15 H23 N2 O5
		311.1184	21.2	68.1	15.5	C21 H15 N2 O
		311.1176	22.0	70.7	3.5	C5 H15 N10 O6
		311.1620	-22.4	-72.0	10.5	C16 H19 N6 O
		311.1163	23.3	74.9	-1.5	C4 H19 N6 O10

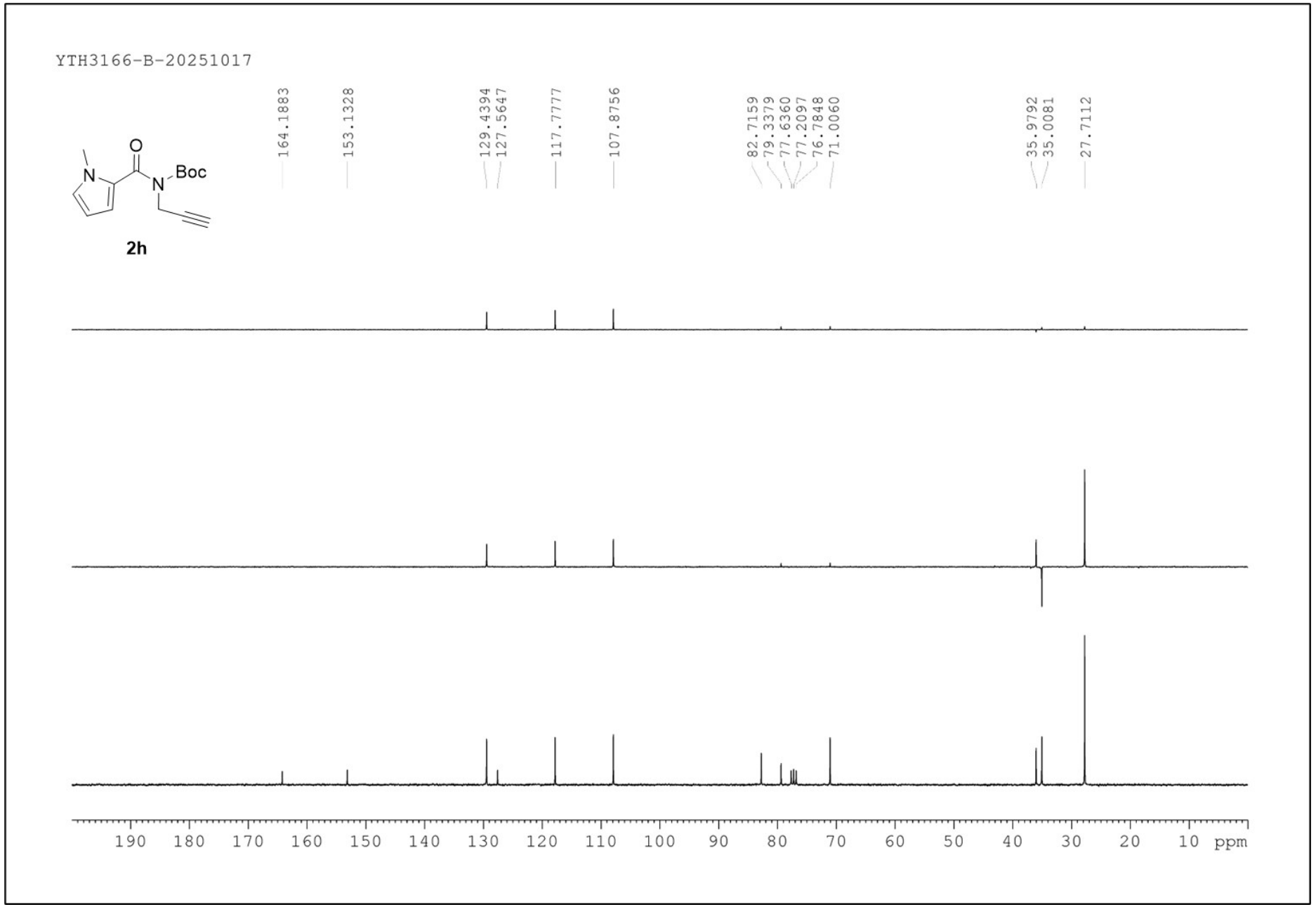


2g

HRMS (ESI$^+$) of **2g**



^{1}H NMR (300 MHz, $CDCl_3$) of **2h**



$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2h**

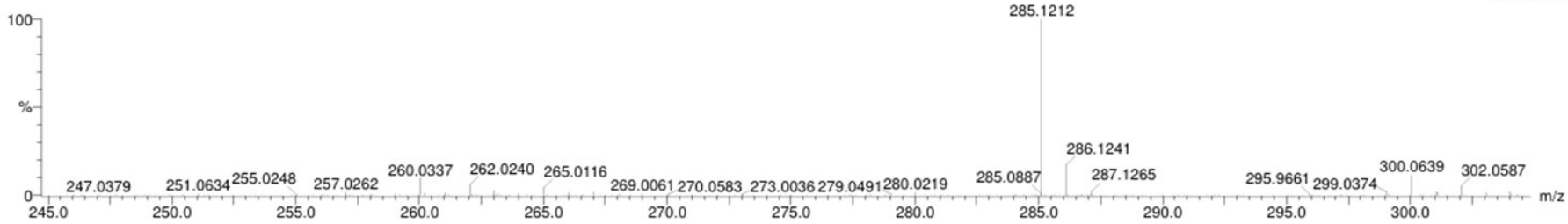
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
691 formula(e) evaluated with 248 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Na: 0-1

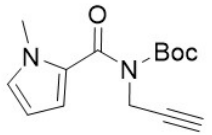
YTH3166B
251112FJU08 280 (2.735) Cm (277:290-(259:268+329:339))

1: TOF MS ES+
5.18e+004



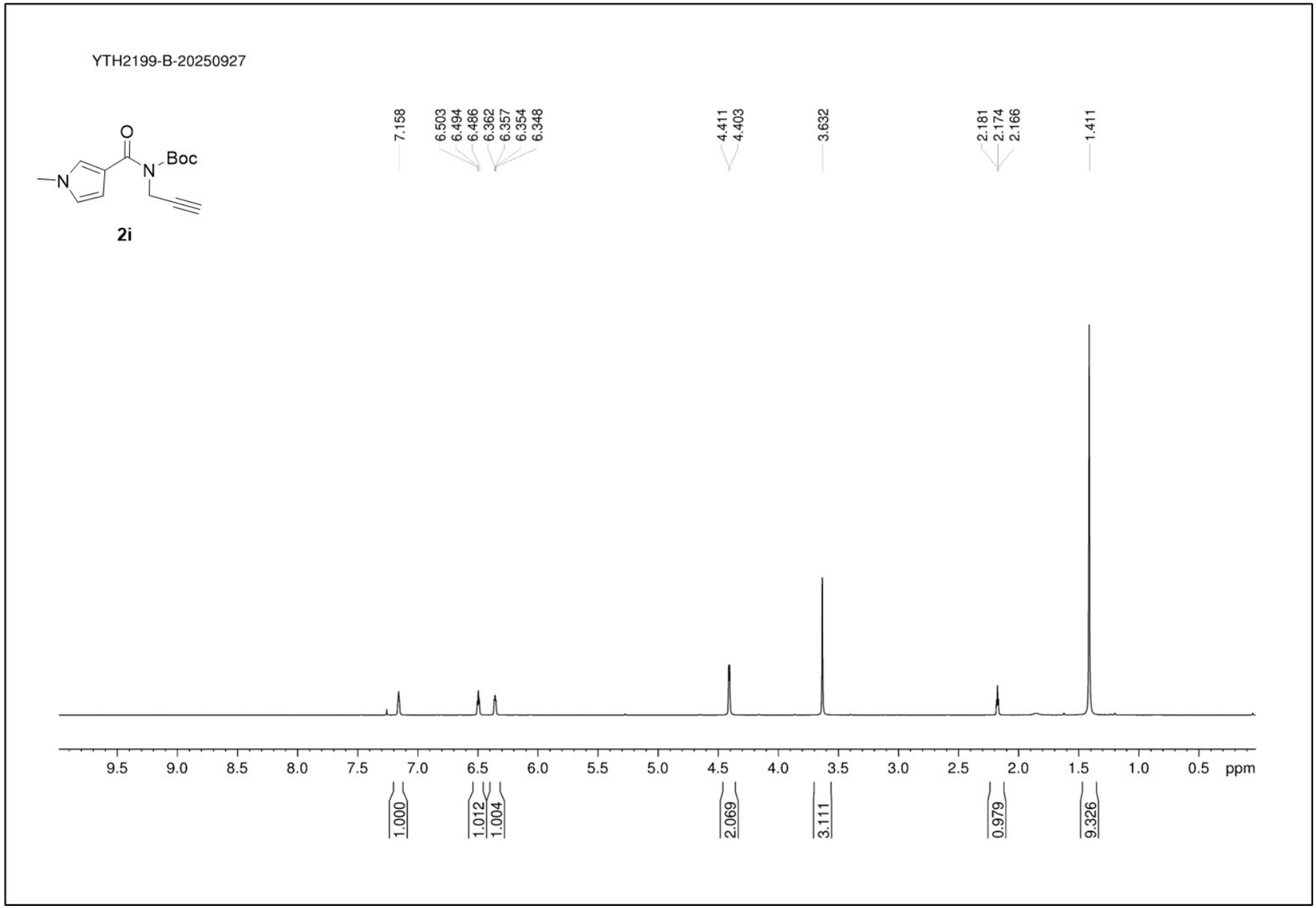
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
285.1212	100.00	285.1215	-0.3	-1.1	6.5	C14 H18 N2 O3 Na
		285.1274	-6.2	-21.7	-2.5	C7 H22 N2 O8 Na
		285.1087	12.5	43.8	5.5	C12 H17 N2 O6
		285.1298	-8.6	-30.2	0.5	C9 H21 N2 O8
		285.1239	-2.7	-9.5	9.5	C16 H17 N2 O3
		285.1327	-11.5	-40.3	6.5	C13 H18 N4 O2 Na
		285.1352	-14.0	-49.1	9.5	C15 H17 N4 O2
		285.1234	-2.2	-7.7	-6.5	C2 H22 N4 O10 Na
		285.1175	3.7	13.0	2.5	C9 H18 N4 O5 Na
		285.1199	1.3	4.6	5.5	C11 H17 N4 O5
		285.1258	-4.6	-16.1	-3.5	C4 H21 N4 O10
		285.1311	-9.9	-34.7	5.5	C10 H17 N6 O4
		285.1287	-7.5	-26.3	2.5	C8 H18 N6 O4 Na
		285.1159	5.3	18.6	1.5	C6 H17 N6 O7
		285.1076	13.6	47.7	7.5	C11 H14 N6 O2 Na
		285.1346	-13.4	-47.0	-6.5	C H22 N6 O9 Na
		285.1100	11.2	39.3	10.5	C13 H13 N6 O2
		285.1135	7.7	27.0	-1.5	C4 H18 N6 O7 Na
		285.1118	9.4	33.0	-2.5	C H17 N8 O9
		285.1188	2.4	8.4	7.5	C10 H14 N8 O Na
		285.1271	-5.9	-20.7	1.5	C5 H17 N8 O6
		285.1247	-3.5	-12.3	-1.5	C3 H18 N8 O6 Na
		285.1212	0.0	0.0	10.5	C12 H13 N8 O
		285.1172	4.0	14.0	6.5	C7 H13 N10 O3
		285.1148	6.4	22.4	3.5	C5 H14 N10 O3 Na

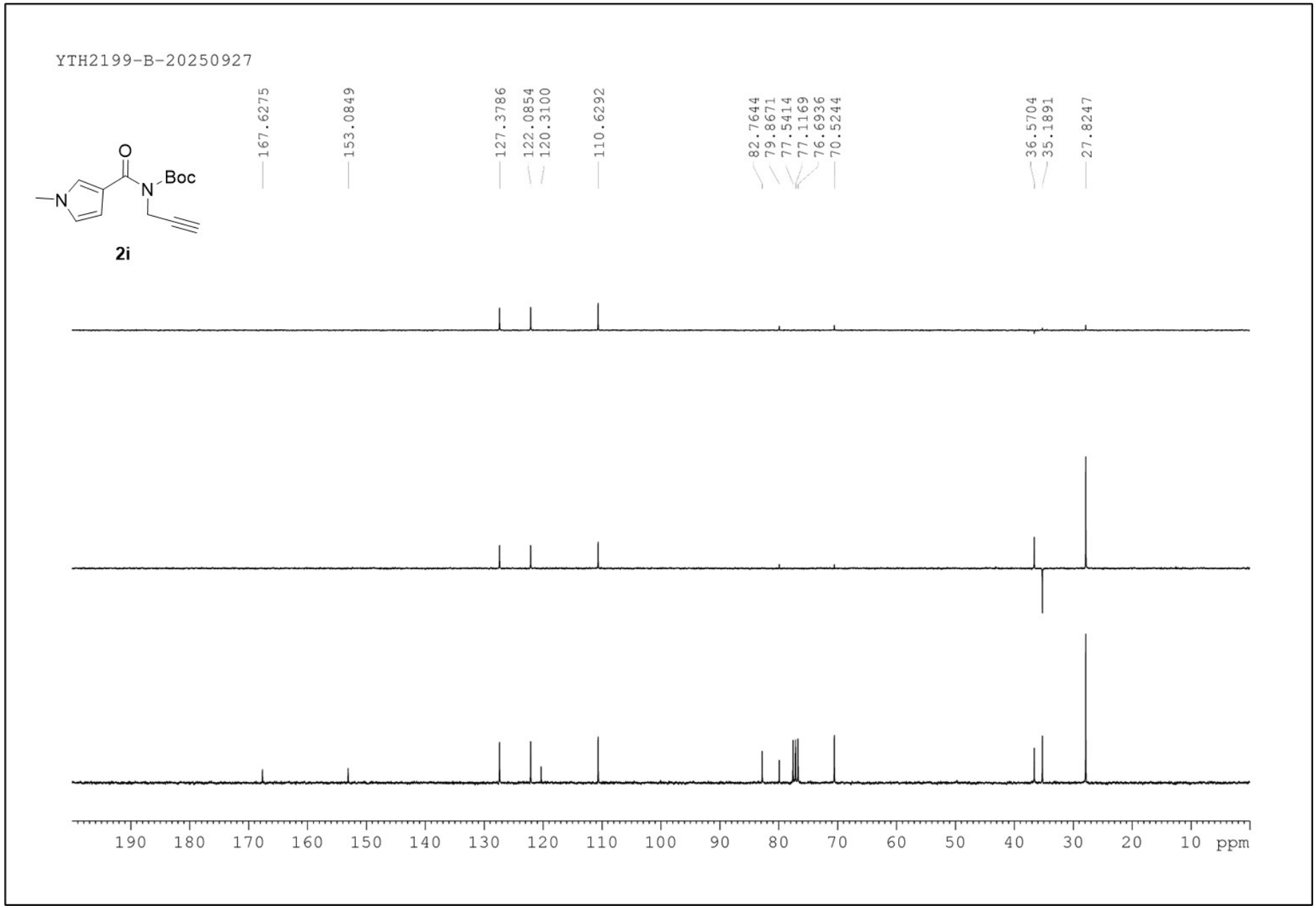


2h

HRMS (ESI$^+$) of **2h**



^{1}H NMR (300 MHz, $CDCl_3$) of **2i**



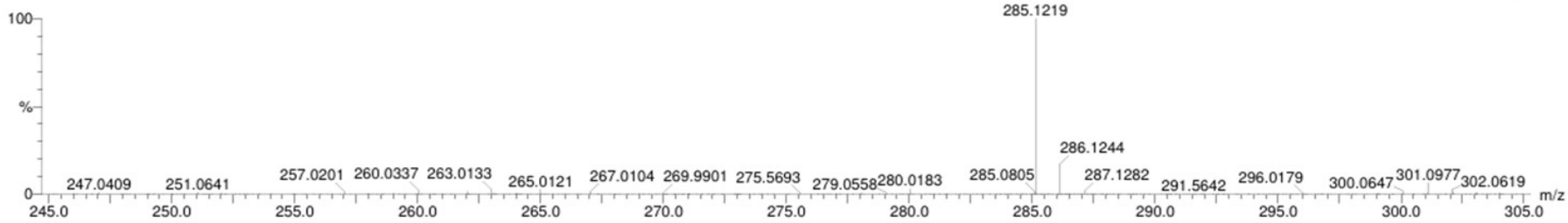
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **2i**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

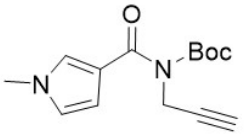
Monoisotopic Mass, Even Electron Ions
691 formula(e) evaluated with 248 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Na: 0-1
YTH2199B
250924FJU46 264 (2.578) Cm (262:273-(246:252+301:307))

1: TOF MS ES+
5.31e+004



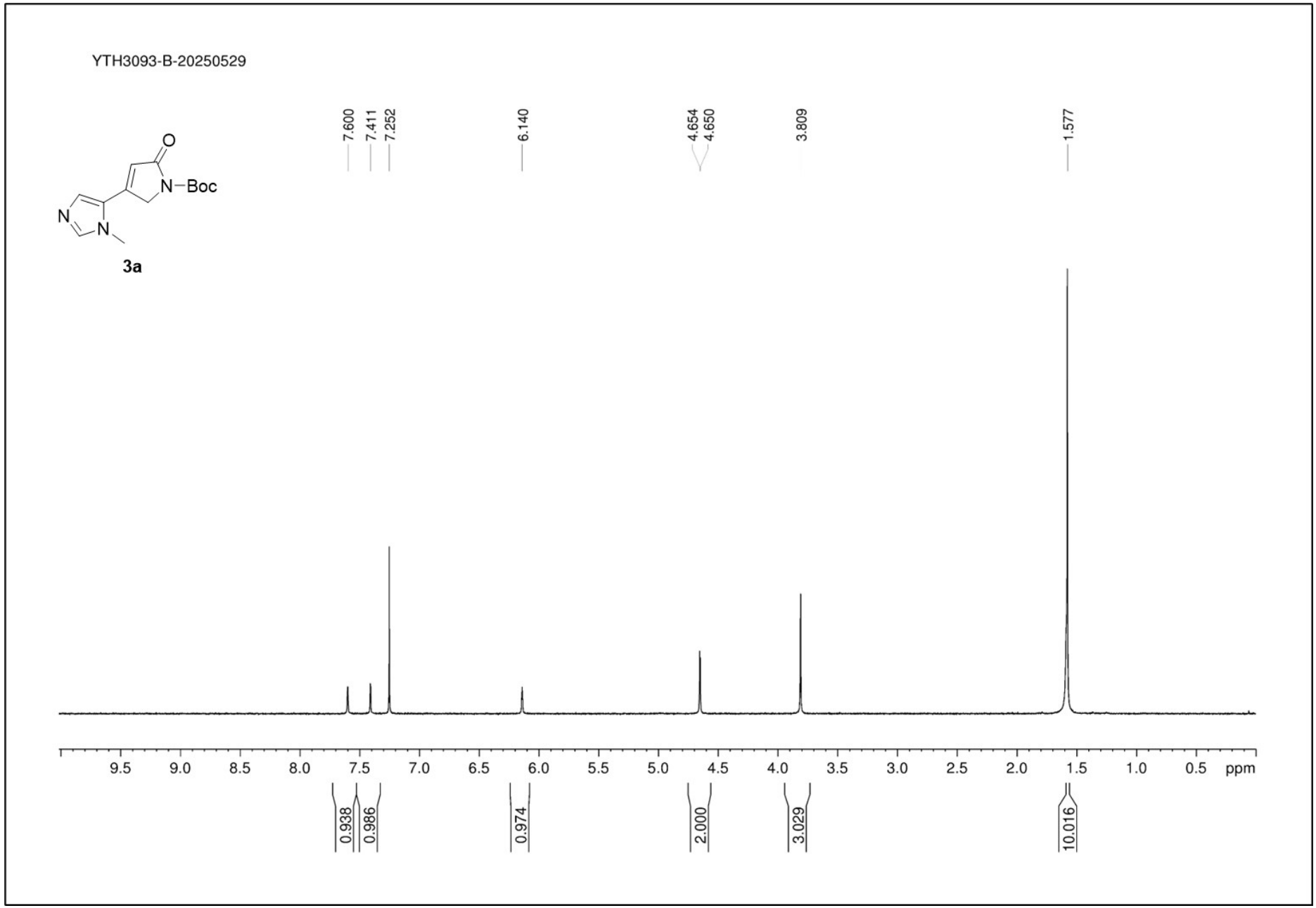
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
285.1219	100.00	285.1215	0.4	1.4	6.5	C14 H18 N2 O3 Na
		285.1212	0.7	2.5	10.5	C12 H13 N8 O
		285.1234	-1.5	-5.3	-6.5	C2 H22 N4 O10 Na
		285.1239	-2.0	-7.0	9.5	C16 H17 N2 O3
		285.1199	2.0	7.0	5.5	C11 H17 N4 O5
		285.1247	-2.8	-9.8	-1.5	C3 H18 N8 O6 Na
		285.1188	3.1	10.9	7.5	C10 H14 N8 O Na
		285.1258	-3.9	-13.7	-3.5	C4 H21 N4 O10
		285.1175	4.4	15.4	2.5	C9 H18 N4 O5 Na
		285.1172	4.7	16.5	6.5	C7 H13 N10 O3
		285.1271	-5.2	-18.2	1.5	C5 H17 N8 O6
		285.1274	-5.5	-19.3	-2.5	C7 H22 N2 O8 Na
		285.1159	6.0	21.0	1.5	C6 H17 N6 O7
		285.1287	-6.8	-23.8	2.5	C8 H18 N6 O4 Na
		285.1148	7.1	24.9	3.5	C5 H14 N10 O3 Na
		285.1298	-7.9	-27.7	0.5	C9 H21 N2 O8
		285.1135	8.4	29.5	-1.5	C4 H18 N6 O7 Na
		285.1311	-9.2	-32.3	5.5	C10 H17 N6 O4
		285.1118	10.1	35.4	-2.5	C H17 N8 O9
		285.1327	-10.8	-37.9	6.5	C13 H18 N4 O2 Na
		285.1100	11.9	41.7	10.5	C13 H13 N6 O2
		285.1346	-12.7	-44.5	-6.5	C H22 N6 O9 Na
		285.1087	13.2	46.3	5.5	C12 H17 N2 O6
		285.1352	-13.3	-46.6	9.5	C15 H17 N4 O2
		285.1359	-14.0	-49.1	-1.5	C2 H18 N10 O5 Na

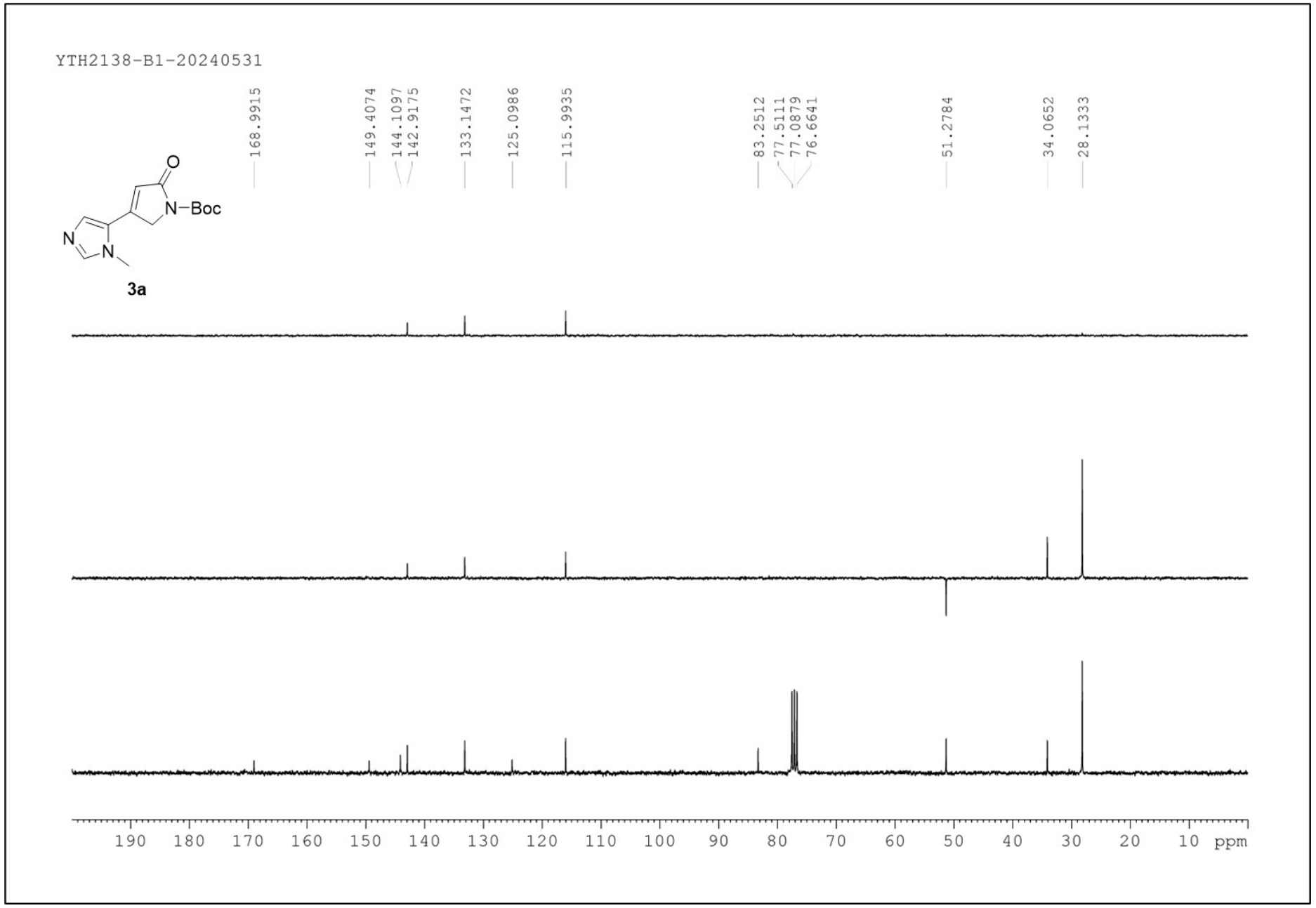


2i

HRMS (ESI$^+$) of **2i**



^{1}H NMR (300 MHz, $CDCl_3$) of **3a** (water peak at around 1.6 ppm)



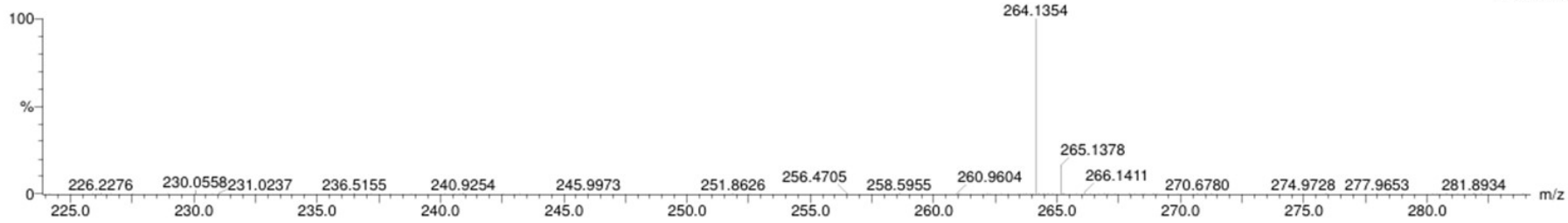
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3a**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

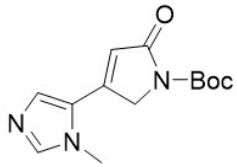
Monoisotopic Mass, Even Electron Ions
323 formula(e) evaluated with 122 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3135B
251112FJU07 165 (1.630) Cm (164:166-(151:158+183:190))

1: TOF MS ES+
1.55e+004



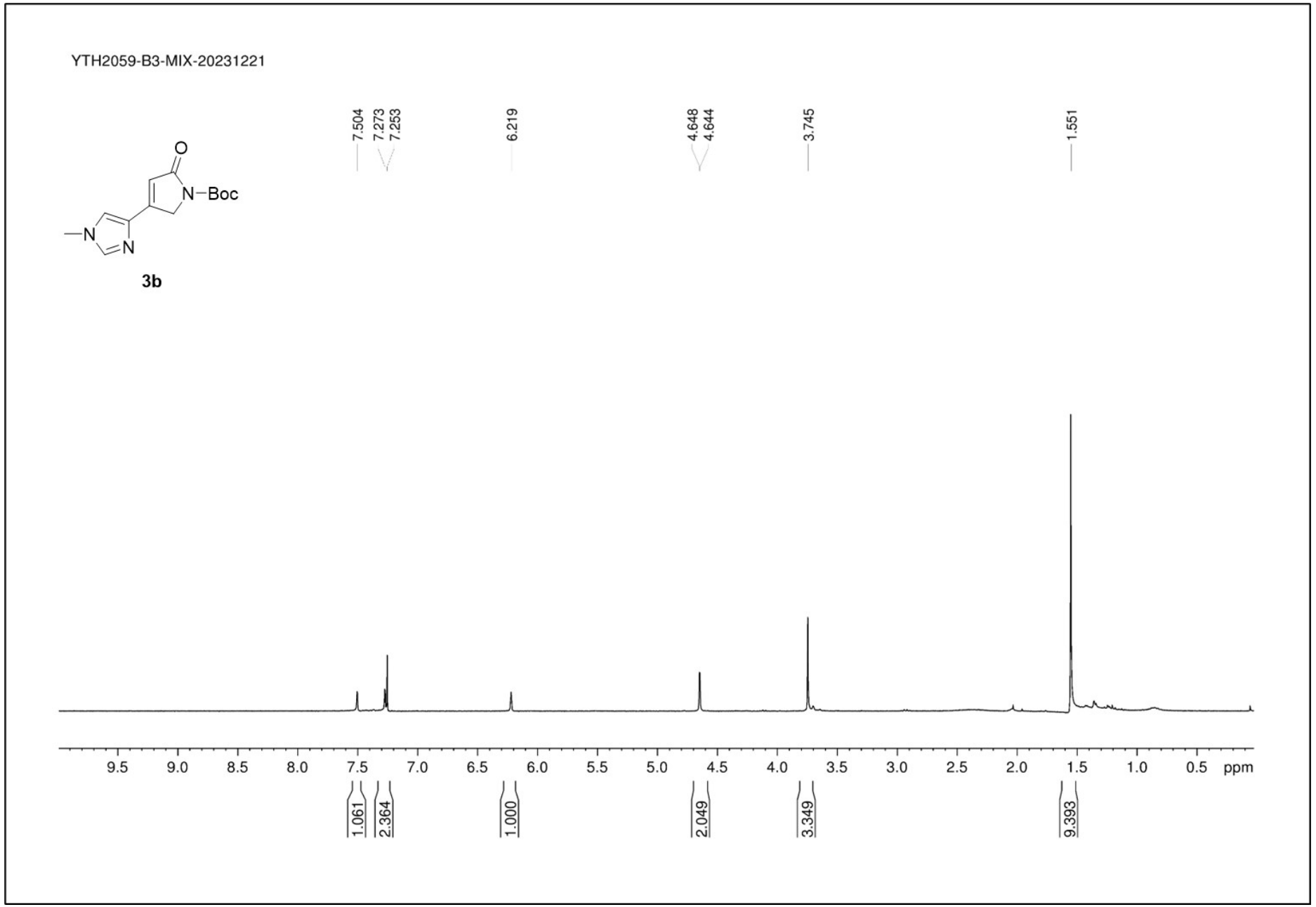
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
264.1354	100.00	264.1348	0.6	2.3	6.5	C13 H18 N3 O3
		264.1367	-1.3	-4.9	-6.5	C H22 N5 O10
		264.1380	-2.6	-9.8	-1.5	C2 H18 N9 O6
		264.1321	3.3	12.5	7.5	C9 H14 N9 O
		264.1388	-3.4	-12.9	10.5	C18 H18 N O
		264.1308	4.6	17.4	2.5	C8 H18 N5 O5
		264.1407	-5.3	-20.1	-2.5	C6 H22 N3 O8
		264.1295	5.9	22.3	-2.5	C7 H22 N O9
		264.1420	-6.6	-25.0	2.5	C7 H18 N7 O4
		264.1268	8.6	32.6	-1.5	C3 H18 N7 O7
		264.1447	-9.3	-35.2	1.5	C11 H22 N O6
		264.1460	-10.6	-40.1	6.5	C12 H18 N5 O2
		264.1236	11.8	44.7	6.5	C14 H18 N O4
		264.1209	14.5	54.9	7.5	C10 H14 N7 O2
		264.1196	15.8	59.8	2.5	C9 H18 N3 O6
		264.1519	-16.5	-62.5	-2.5	C5 H22 N5 O7
		264.1533	-17.9	-67.8	2.5	C6 H18 N9 O3
		264.1169	18.5	70.0	3.5	C5 H14 N9 O4
		264.1155	19.9	75.3	-1.5	C4 H18 N5 O8
		264.1559	-20.5	-77.6	1.5	C10 H22 N3 O5
		264.1137	21.7	82.2	11.5	C16 H14 N3 O
		264.1573	-21.9	-82.9	6.5	C11 H18 N7 O
		264.1600	-24.6	-93.1	5.5	C15 H22 N O3
		264.1097	25.7	97.3	7.5	C11 H14 N5 O3
		264.1618	-26.4	-99.9	-7.5	C3 H26 N3 O10

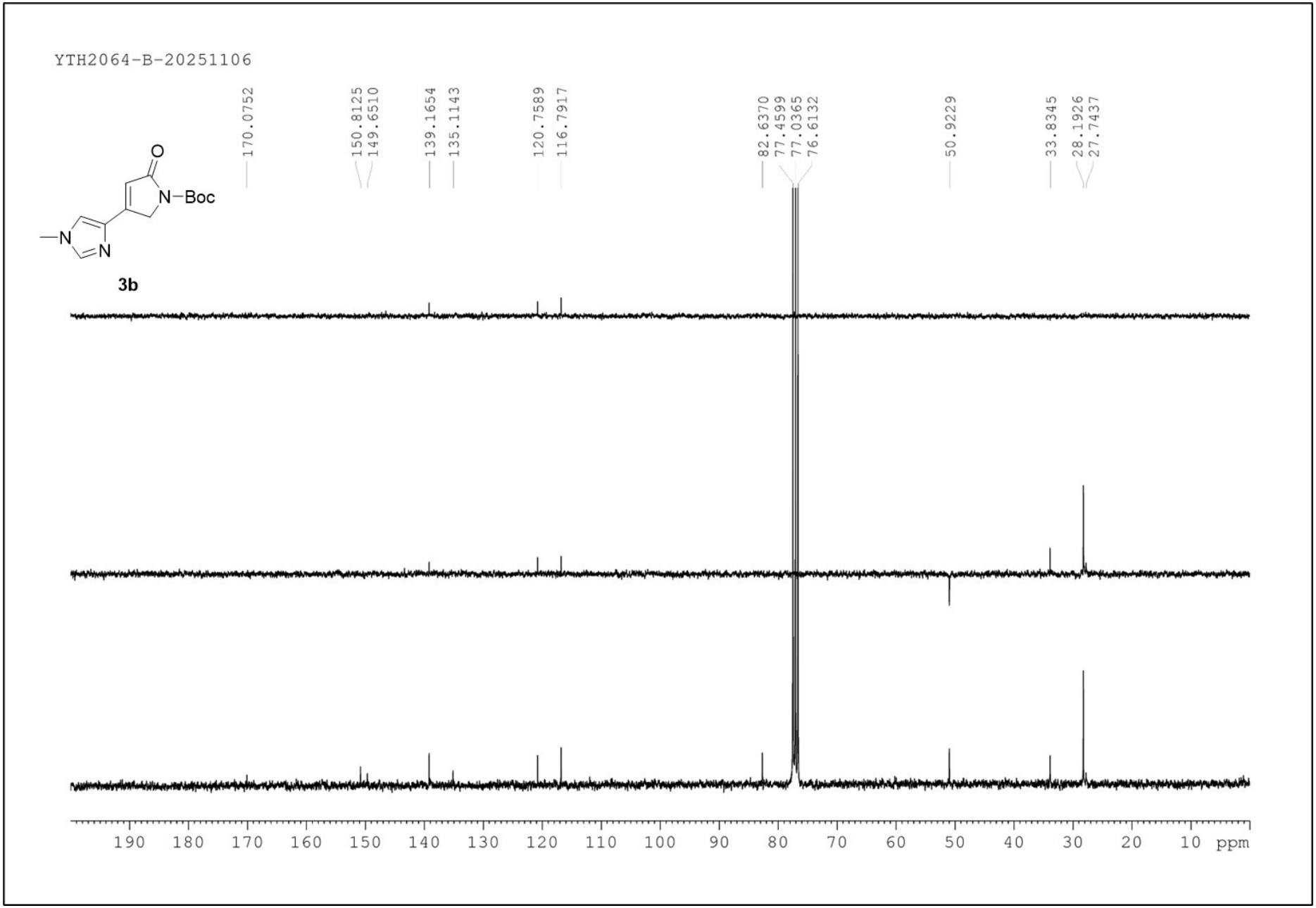


3a

HRMS (ESI$^+$) of **3a**



^{1}H NMR (300 MHz, $CDCl_3$) of **3b** (contaminated with tin residue)



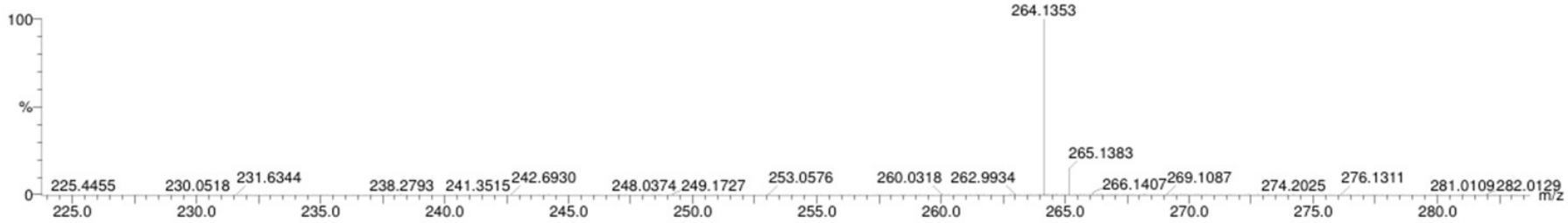
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3b**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

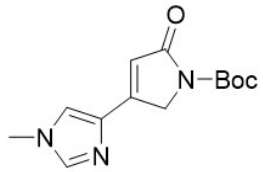
Monoisotopic Mass, Even Electron Ions
323 formula(e) evaluated with 122 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2039B
250919FJU29 109 (1.090) Cm (108:113-(93:99+134:140))

1: TOF MS ES+
2.75e+005



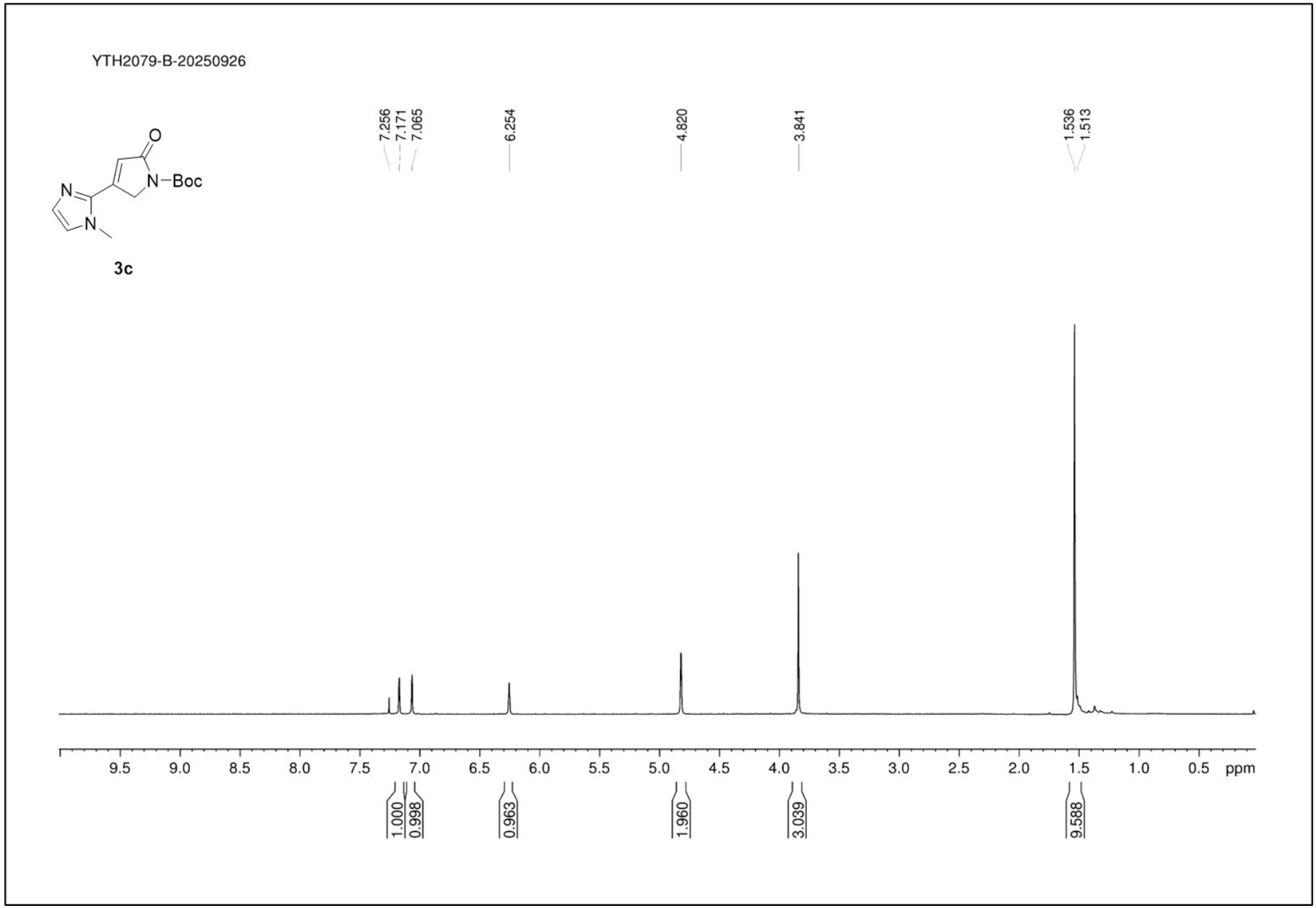
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
264.1353	100.00	264.1348	0.5	1.9	6.5	C13 H18 N3 O3
		264.1367	-1.4	-5.3	-6.5	C H22 N5 O10
		264.1380	-2.7	-10.2	-1.5	C2 H18 N9 O6
		264.1321	3.2	12.1	7.5	C9 H14 N9 O
		264.1388	-3.5	-13.3	10.5	C18 H18 N O
		264.1308	4.5	17.0	2.5	C8 H18 N5 O5
		264.1407	-5.4	-20.4	-2.5	C6 H22 N3 O8
		264.1295	5.8	22.0	-2.5	C7 H22 N O9
		264.1420	-6.7	-25.4	2.5	C7 H18 N7 O4
		264.1268	8.5	32.2	-1.5	C3 H18 N7 O7
		264.1447	-9.4	-35.6	1.5	C11 H22 N O6
		264.1460	-10.7	-40.5	6.5	C12 H18 N5 O2
		264.1236	11.7	44.3	6.5	C14 H18 N O4
		264.1209	14.4	54.5	7.5	C10 H14 N7 O2
		264.1196	15.7	59.4	2.5	C9 H18 N3 O6
		264.1519	-16.6	-62.8	-2.5	C5 H22 N5 O7
		264.1533	-18.0	-68.1	2.5	C6 H18 N9 O3
		264.1169	18.4	69.7	3.5	C5 H14 N9 O4
		264.1155	19.8	75.0	-1.5	C4 H18 N5 O8
		264.1559	-20.6	-78.0	1.5	C10 H22 N3 O5
		264.1137	21.6	81.8	11.5	C16 H14 N3 O
		264.1573	-22.0	-83.3	6.5	C11 H18 N7 O
		264.1600	-24.7	-93.5	5.5	C15 H22 N O3
		264.1097	25.6	96.9	7.5	C11 H14 N5 O3
		264.1618	-26.5	-100.3	-7.5	C3 H26 N3 O10

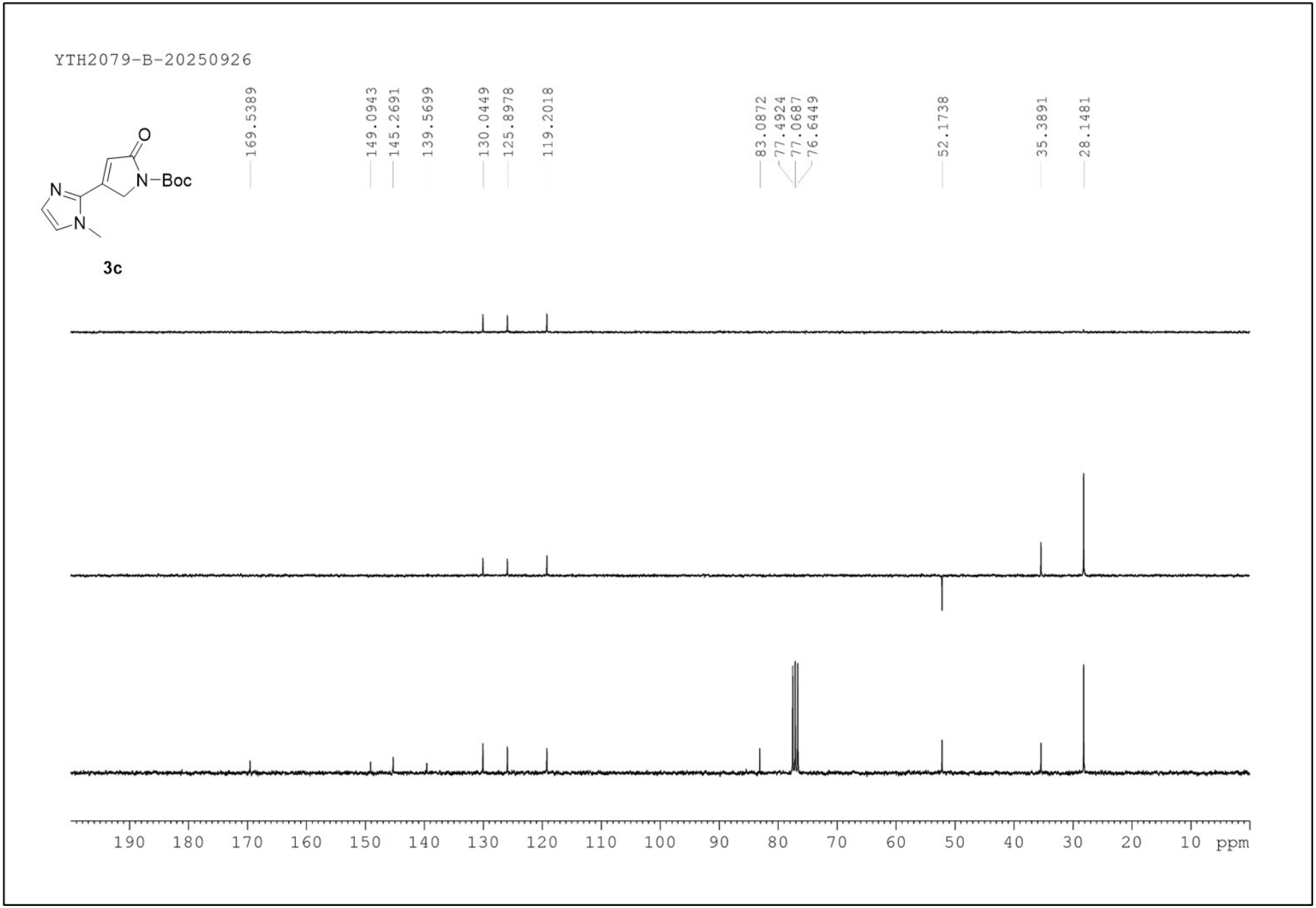


3b

HRMS (ESI$^+$) of **3b**



^{1}H NMR (300 MHz, $CDCl_3$) of **3c** (contaminated with trace of tin residue)



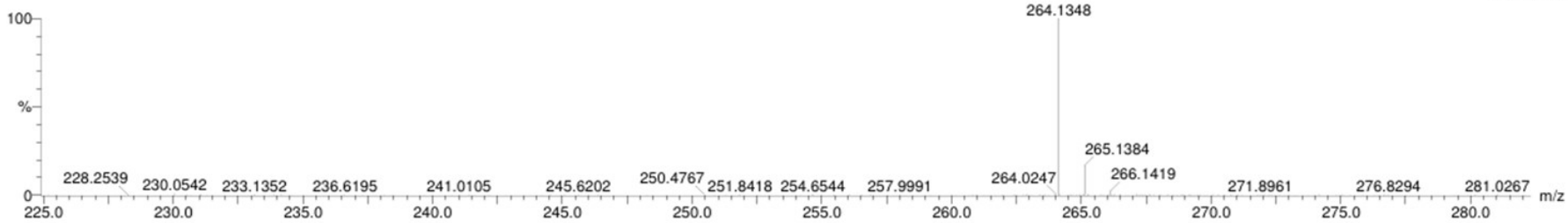
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3c**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

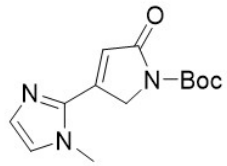
Monoisotopic Mass, Even Electron Ions
323 formula(e) evaluated with 122 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2019B
250924FJU33 187 (1.838) Cm (186:189-(174:180+204:209))

1: TOF MS ES+
5.41e+004



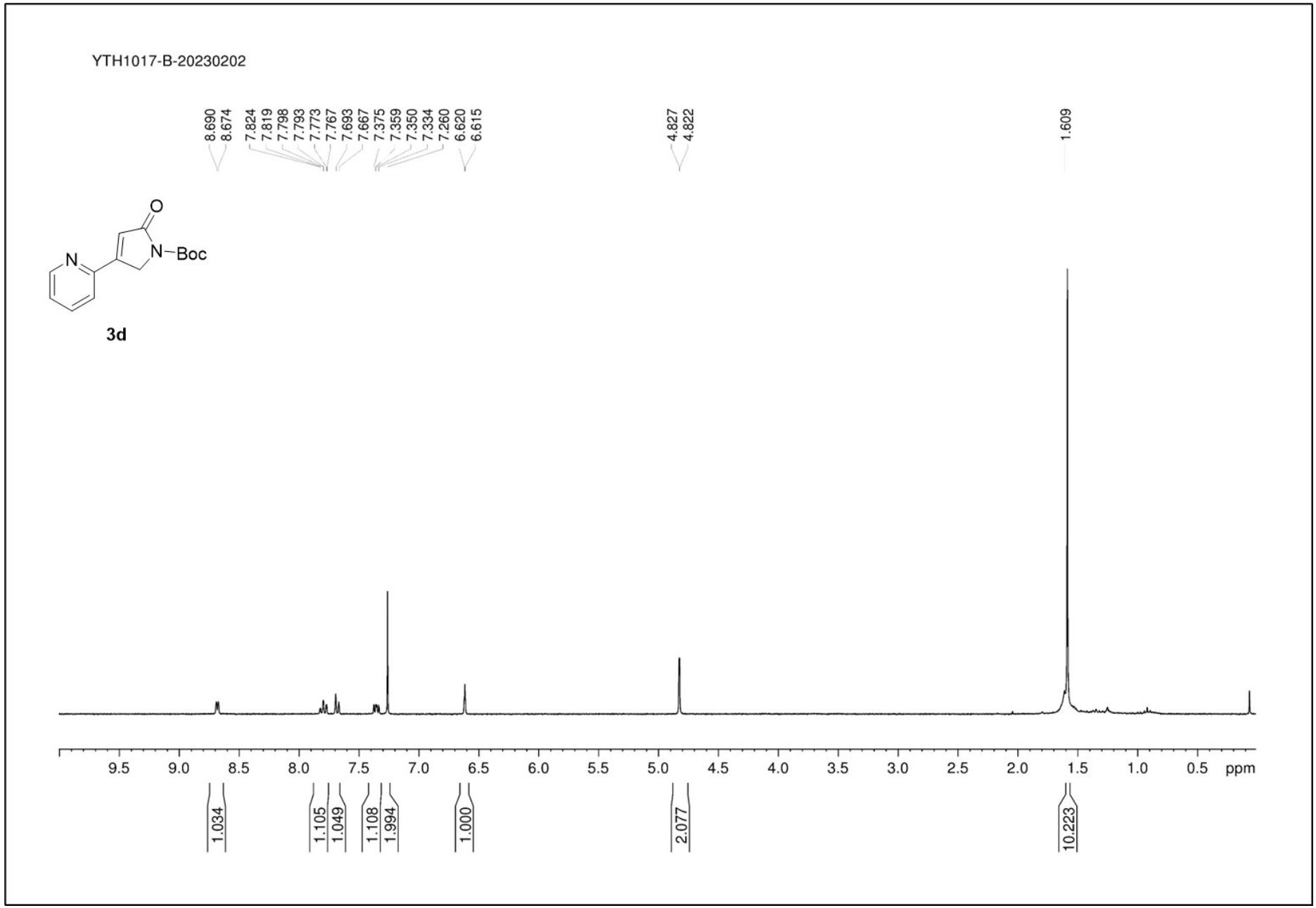
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
264.1348	100.00	264.1348	0.0	0.0	6.5	C13 H18 N3 O3
		264.1367	-1.9	-7.2	-6.5	C H22 N5 O10
		264.1321	2.7	10.2	7.5	C9 H14 N9 O
		264.1380	-3.2	-12.1	-1.5	C2 H18 N9 O6
		264.1388	-4.0	-15.1	10.5	C18 H18 N O
		264.1308	4.0	15.1	2.5	C8 H18 N5 O5
		264.1295	5.3	20.1	-2.5	C7 H22 N O9
		264.1407	-5.9	-22.3	-2.5	C6 H22 N3 O8
		264.1420	-7.2	-27.3	2.5	C7 H18 N7 O4
		264.1268	8.0	30.3	-1.5	C3 H18 N7 O7
		264.1447	-9.9	-37.5	1.5	C11 H22 N O6
		264.1460	-11.2	-42.4	6.5	C12 H18 N5 O2
		264.1236	11.2	42.4	6.5	C14 H18 N O4
		264.1209	13.9	52.6	7.5	C10 H14 N7 O2
		264.1196	15.2	57.5	2.5	C9 H18 N3 O6
		264.1519	-17.1	-64.7	-2.5	C5 H22 N5 O7
		264.1169	17.9	67.8	3.5	C5 H14 N9 O4
		264.1533	-18.5	-70.0	2.5	C6 H18 N9 O3
		264.1155	19.3	73.1	-1.5	C4 H18 N5 O8
		264.1559	-21.1	-79.9	1.5	C10 H22 N3 O5
		264.1137	21.1	79.9	11.5	C16 H14 N3 O
		264.1573	-22.5	-85.2	6.5	C11 H18 N7 O
		264.1097	25.1	95.0	7.5	C11 H14 N5 O3
		264.1600	-25.2	-95.4	5.5	C15 H22 N O3
		264.1083	26.5	100.3	2.5	C10 H18 N O7

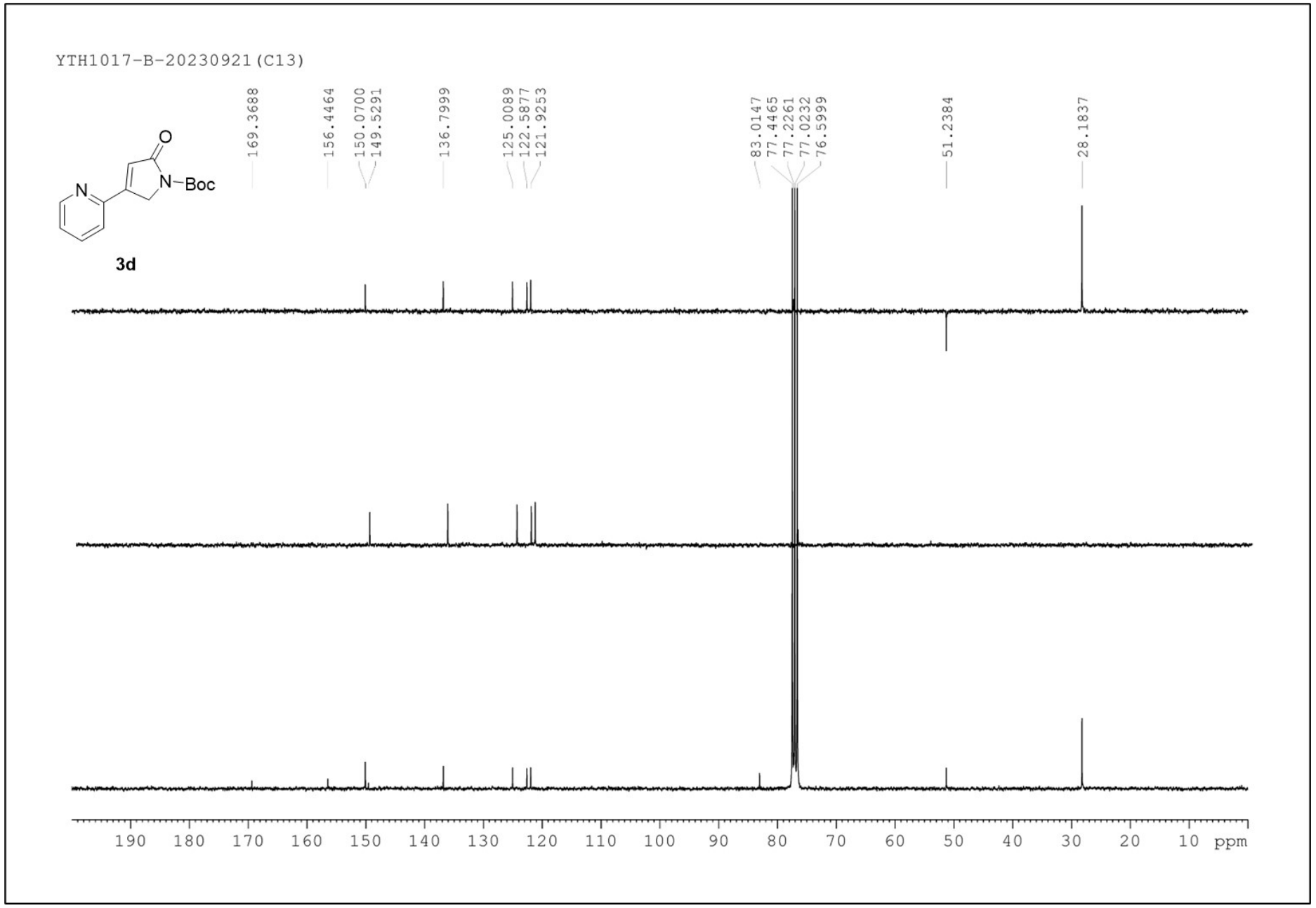


3c

HRMS (ESI$^+$) of **3c**



^{1}H NMR (300 MHz, $CDCl_3$) of **3d** (contaminated with tin residue)



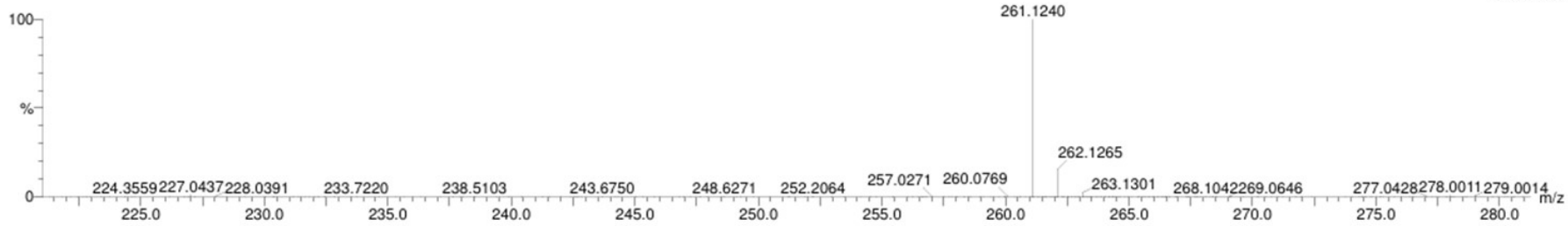
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3d**

Elemental Composition Report **Page 1**

Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

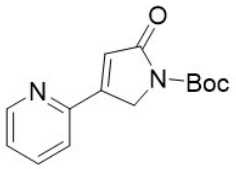
Monoisotopic Mass, Even Electron Ions
326 formula(e) evaluated with 114 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH1017B
250924FJU38 237 (2.326) Cm (236:241-(221:228+258:265))

1: TOF MS ES+
8.69e+004



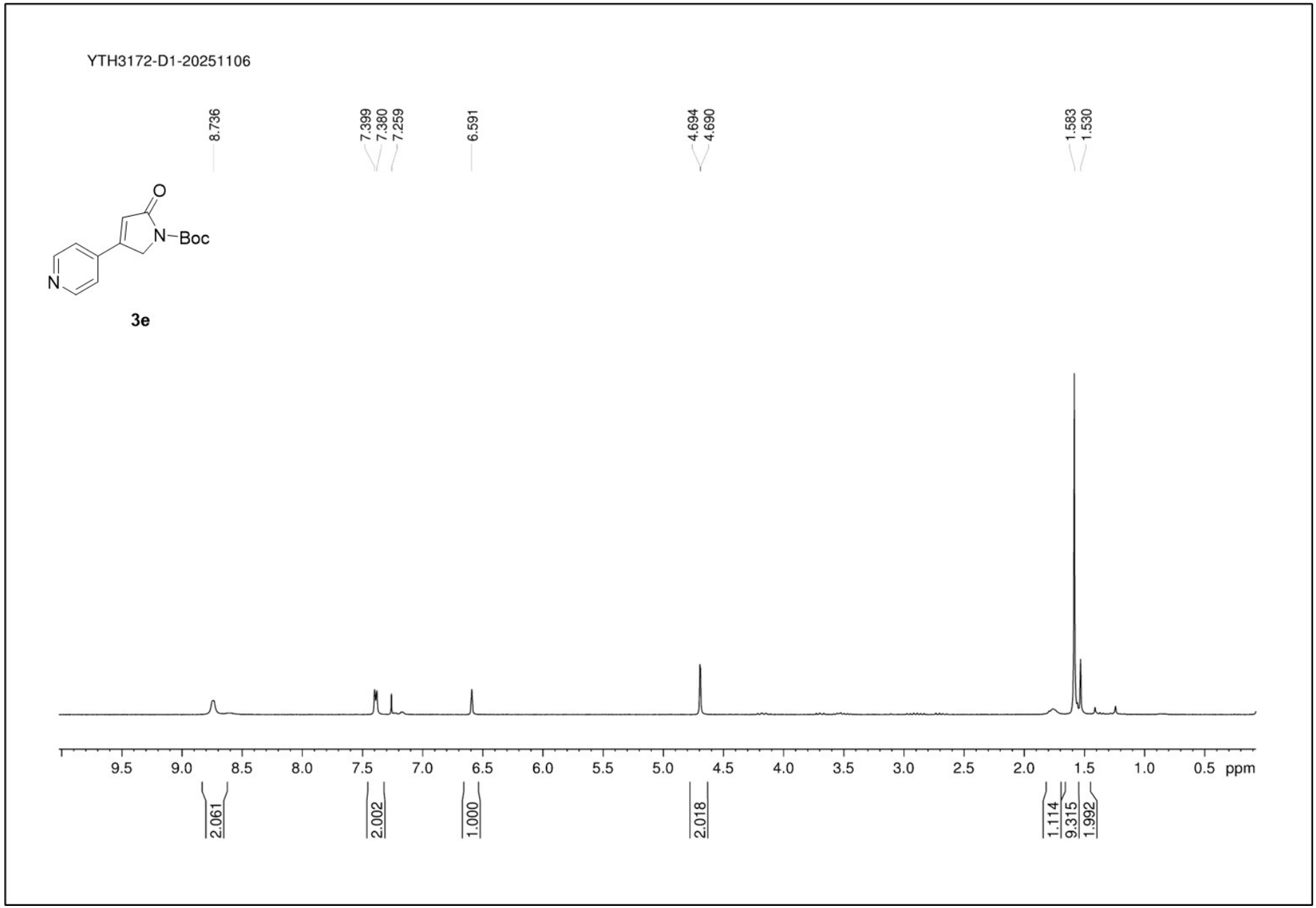
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
261.1240	100.00	261.1239	0.1	0.4	7.5	C14 H17 N2 O3
		261.1258	-1.8	-6.9	-5.5	C2 H21 N4 O10
		261.1212	2.8	10.7	8.5	C10 H13 N8 O
		261.1271	-3.1	-11.9	-0.5	C3 H17 N8 O6
		261.1199	4.1	15.7	3.5	C9 H17 N4 O5
		261.1298	-5.8	-22.2	-1.5	C7 H21 N2 O8
		261.1172	6.8	26.0	4.5	C5 H13 N10 O3
		261.1311	-7.1	-27.2	3.5	C8 H17 N6 O4
		261.1159	8.1	31.0	-0.5	C4 H17 N6 O7
		261.1352	-11.2	-42.9	7.5	C13 H17 N4 O2
		261.1370	-13.0	-49.8	-5.5	C H21 N6 O9
		261.1100	14.0	53.6	8.5	C11 H13 N6 O2
		261.1383	-14.3	-54.8	-0.5	C2 H17 N10 O5
		261.1087	15.3	58.6	3.5	C10 H17 N2 O6
		261.1410	-17.0	-65.1	-1.5	C6 H21 N4 O7
		261.1060	18.0	68.9	4.5	C6 H13 N8 O4
		261.1424	-18.4	-70.5	3.5	C7 H17 N8 O3
		261.1046	19.4	74.3	-0.5	C5 H17 N4 O8
		261.1450	-21.0	-80.4	2.5	C11 H21 N2 O5
		261.1028	21.2	81.2	12.5	C17 H13 N2 O
		261.1020	22.0	84.3	0.5	C H13 N10 O6
		261.1464	-22.4	-85.8	7.5	C12 H17 N6 O
		261.0988	25.2	96.5	8.5	C12 H13 N4 O3
		261.1509	-26.9	-103.0	-6.5	C4 H25 N2 O10
		261.0961	27.9	106.8	9.5	C8 H9 N10 O

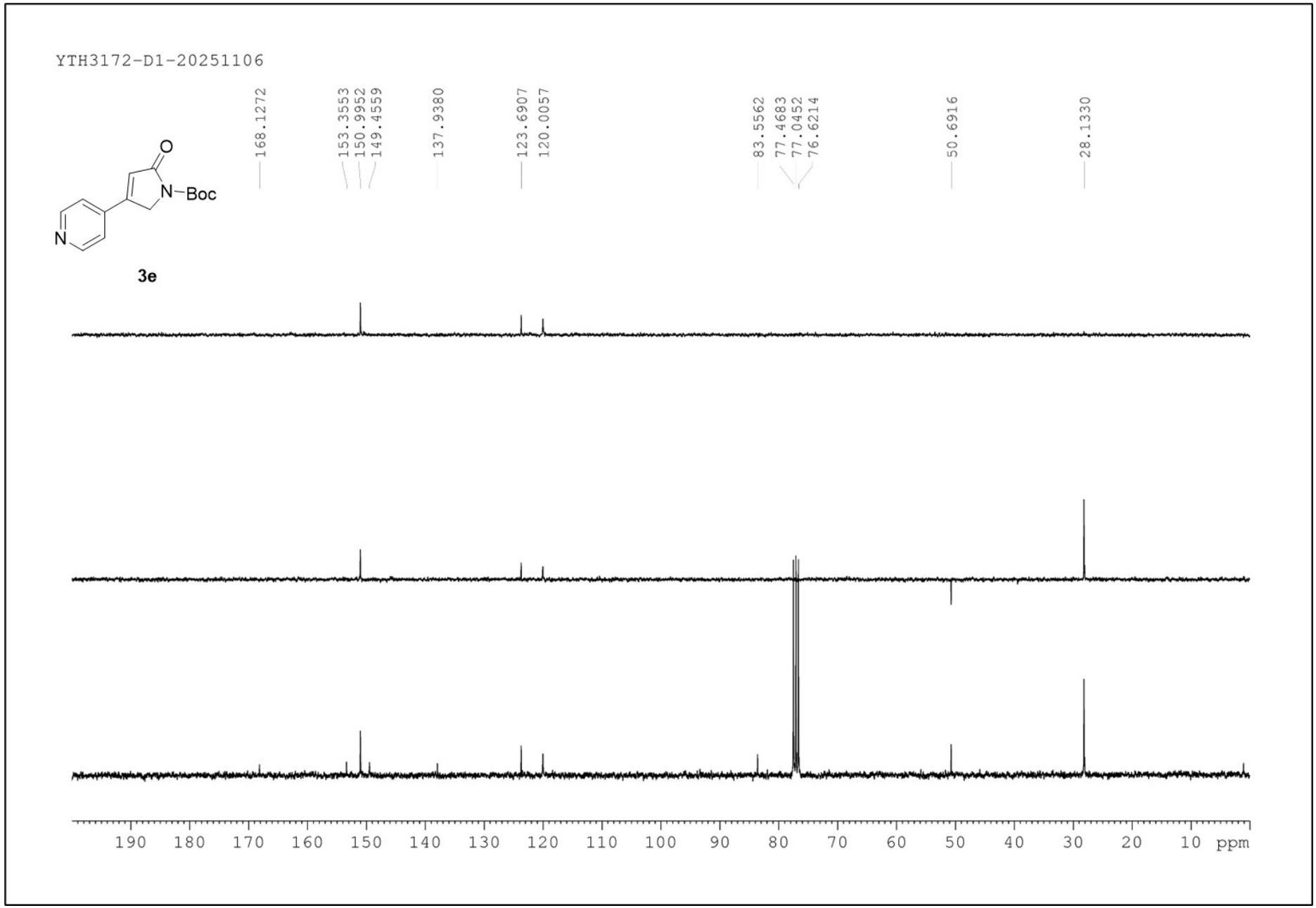


3d

HRMS (ESI$^+$) of **3d**



^{1}H NMR (300 MHz, $CDCl_3$) of **3e** (contaminated with tin residue)



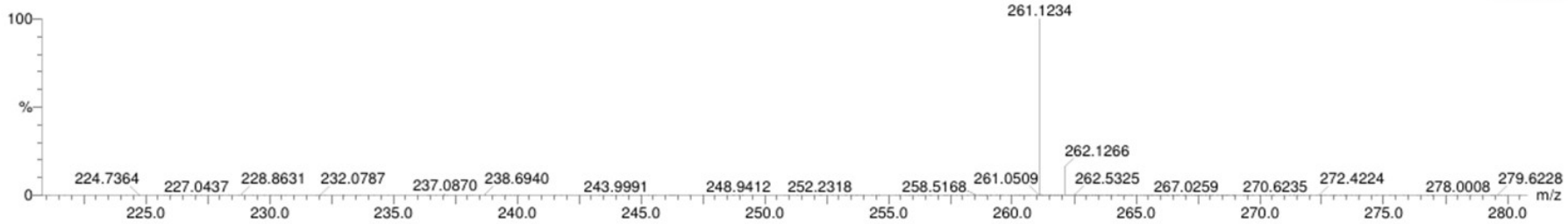
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3e**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

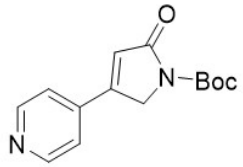
Monoisotopic Mass, Even Electron Ions
326 formula(e) evaluated with 113 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2019B (YTH3055B)
250924FJU41 197 (1.924) Cm (196:198-(179:186+217:225))

1: TOF MS ES+
1.94e+005



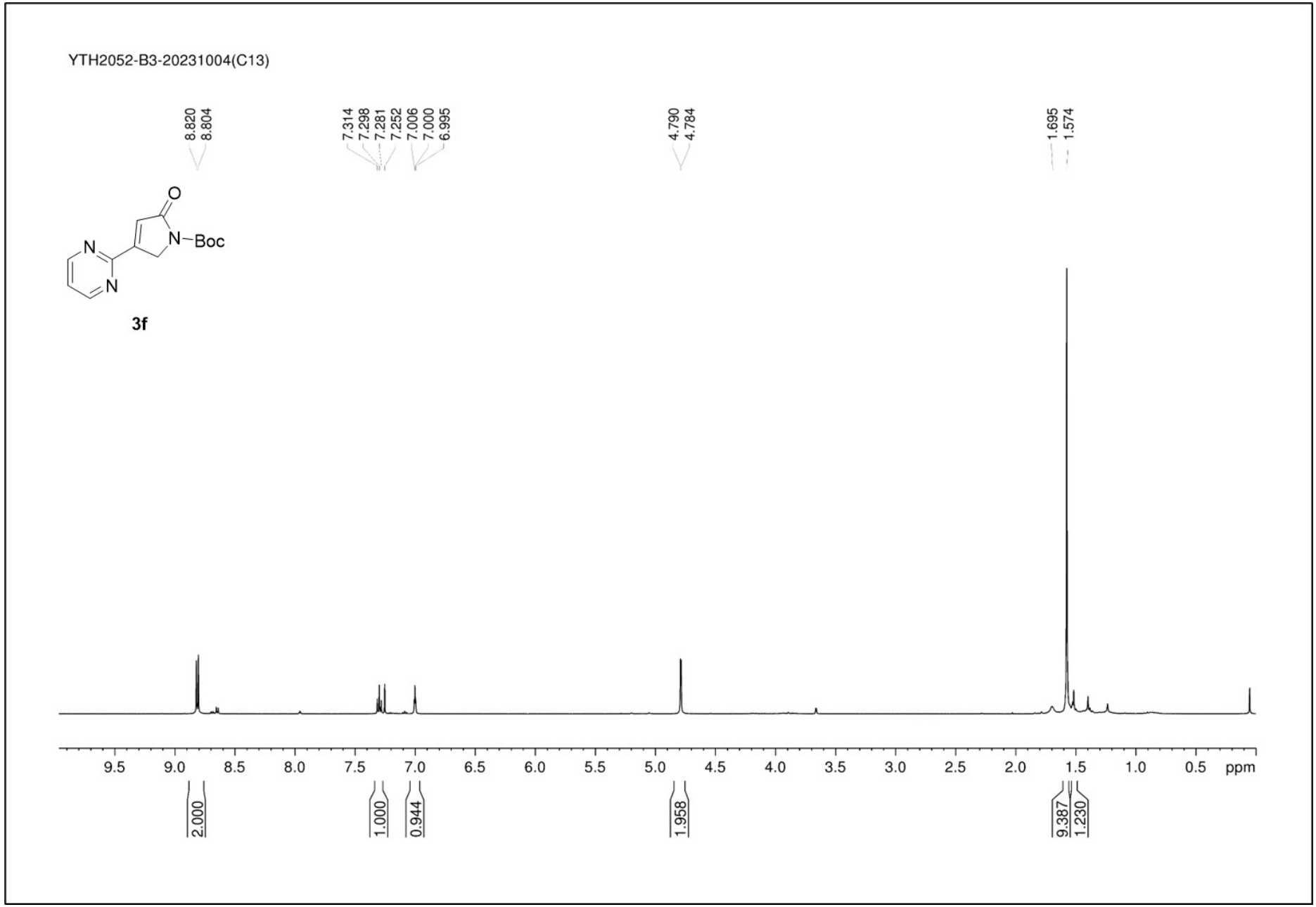
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
261.1234	100.00	261.1239	-0.5	-1.9	7.5	C14 H17 N2 O3
		261.1212	2.2	8.4	8.5	C10 H13 N8 O
		261.1258	-2.4	-9.2	-5.5	C2 H21 N4 O10
		261.1199	3.5	13.4	3.5	C9 H17 N4 O5
		261.1271	-3.7	-14.2	-0.5	C3 H17 N8 O6
		261.1172	6.2	23.7	4.5	C5 H13 N10 O3
		261.1298	-6.4	-24.5	-1.5	C7 H21 N2 O8
		261.1159	7.5	28.7	-0.5	C4 H17 N6 O7
		261.1311	-7.7	-29.5	3.5	C8 H17 N6 O4
		261.1352	-11.8	-45.2	7.5	C13 H17 N4 O2
		261.1100	13.4	51.3	8.5	C11 H13 N6 O2
		261.1370	-13.6	-52.1	-5.5	C H21 N6 O9
		261.1087	14.7	56.3	3.5	C10 H17 N2 O6
		261.1383	-14.9	-57.1	-0.5	C2 H17 N10 O5
		261.1060	17.4	66.6	4.5	C6 H13 N8 O4
		261.1410	-17.6	-67.4	-1.5	C6 H21 N4 O7
		261.1046	18.8	72.0	-0.5	C5 H17 N4 O8
		261.1424	-19.0	-72.8	3.5	C7 H17 N8 O3
		261.1028	20.6	78.9	12.5	C17 H13 N2 O
		261.1020	21.4	82.0	0.5	C H13 N10 O6
		261.1450	-21.6	-82.7	2.5	C11 H21 N2 O5
		261.1464	-23.0	-88.1	7.5	C12 H17 N6 O
		261.0988	24.6	94.2	8.5	C12 H13 N4 O3
		261.0961	27.3	104.5	9.5	C8 H9 N10 O
		261.1509	-27.5	-105.3	-6.5	C4 H25 N2 O10

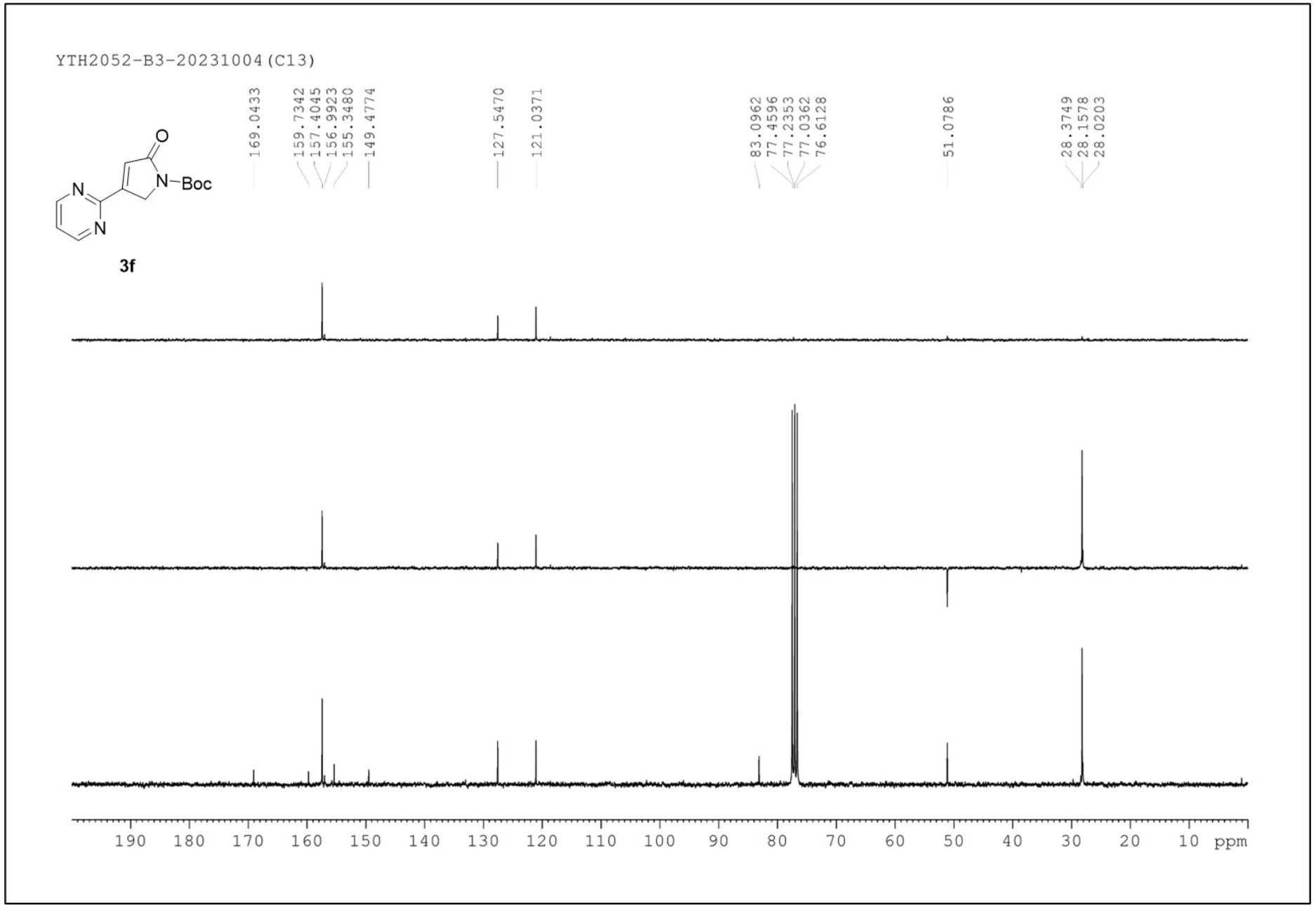


3e

HRMS (ESI$^+$) of **3e**



^{1}H NMR (300 MHz, $CDCl_3$) of **3f** (contaminated with tin residue)



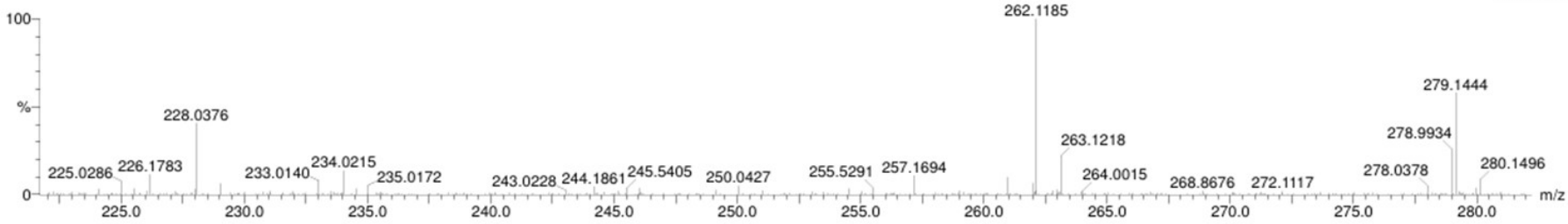
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3f**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

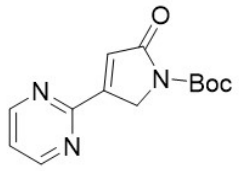
Monoisotopic Mass, Even Electron Ions
319 formula(e) evaluated with 114 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2052B
250924FJU42 223 (2.187) Cm (221:231-(202:210+245:252))

1: TOF MS ES+
1.14e+004



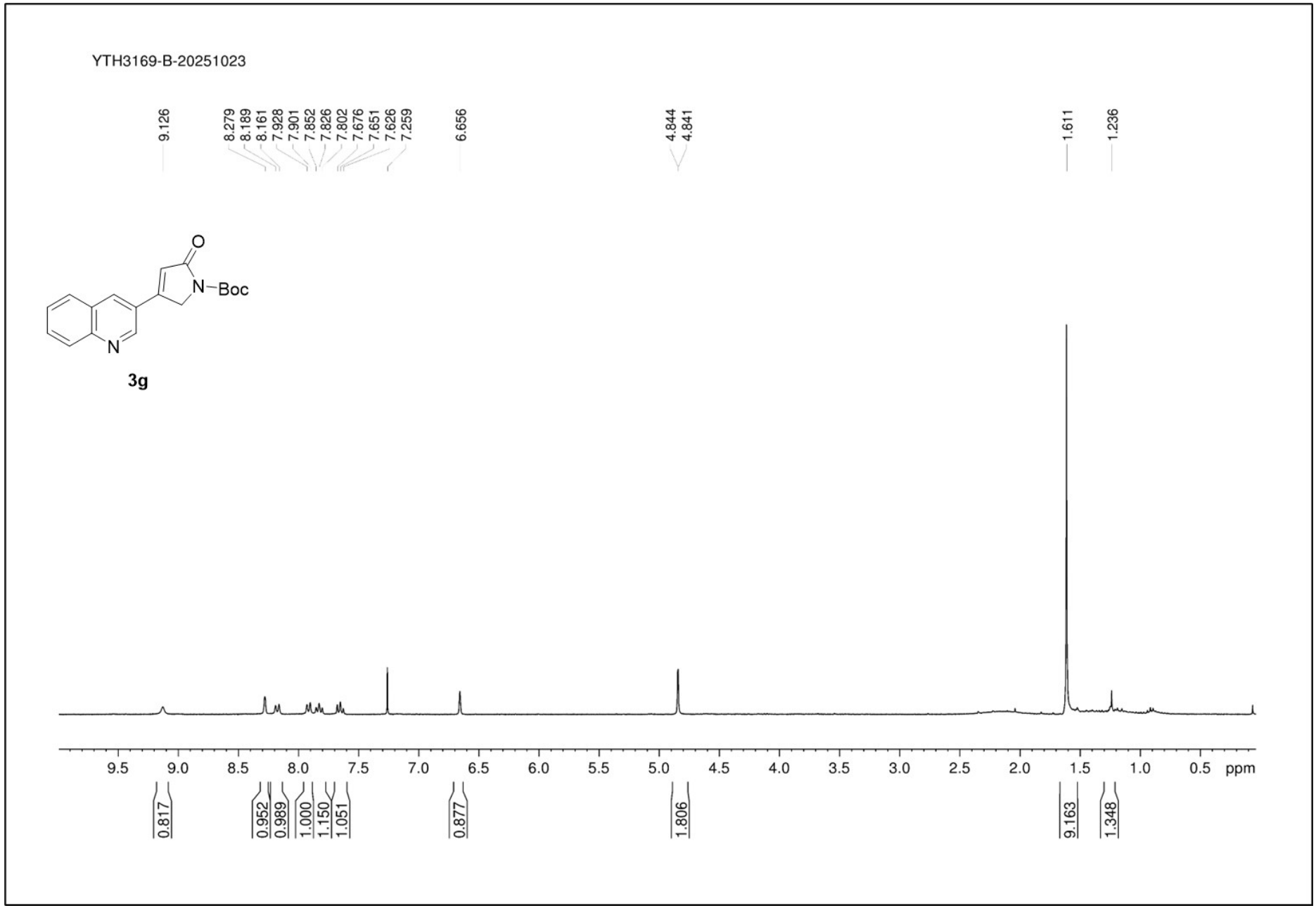
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
262.1185	100.00	262.1192	-0.7	-2.7	7.5	C13 H16 N3 O3
		262.1165	2.0	7.6	8.5	C9 H12 N9 O
		262.1210	-2.5	-9.5	-5.5	C H20 N5 O10
		262.1151	3.4	13.0	3.5	C8 H16 N5 O5
		262.1224	-3.9	-14.9	-0.5	C2 H16 N9 O6
		262.1232	-4.7	-17.9	11.5	C18 H16 N O
		262.1138	4.7	17.9	-1.5	C7 H20 N O9
		262.1250	-6.5	-24.8	-1.5	C6 H20 N3 O8
		262.1111	7.4	28.2	-0.5	C3 H16 N7 O7
		262.1264	-7.9	-30.1	3.5	C7 H16 N7 O4
		262.1079	10.6	40.4	7.5	C14 H16 N O4
		262.1291	-10.6	-40.4	2.5	C11 H20 N O6
		262.1304	-11.9	-45.4	7.5	C12 H16 N5 O2
		262.1052	13.3	50.7	8.5	C10 H12 N7 O2
		262.1039	14.6	55.7	3.5	C9 H16 N3 O6
		262.1012	17.3	66.0	4.5	C5 H12 N9 O4
		262.1363	-17.8	-67.9	-1.5	C5 H20 N5 O7
		262.0999	18.6	71.0	-0.5	C4 H16 N5 O8
		262.1376	-19.1	-72.9	3.5	C6 H16 N9 O3
		262.0980	20.5	78.2	12.5	C16 H12 N3 O
		262.1403	-21.8	-83.2	2.5	C10 H20 N3 O5
		262.1416	-23.1	-88.1	7.5	C11 H16 N7 O
		262.0940	24.5	93.5	8.5	C11 H12 N5 O3
		262.0927	25.8	98.4	3.5	C10 H16 N O7
		262.1443	-25.8	-98.4	6.5	C15 H20 N O3

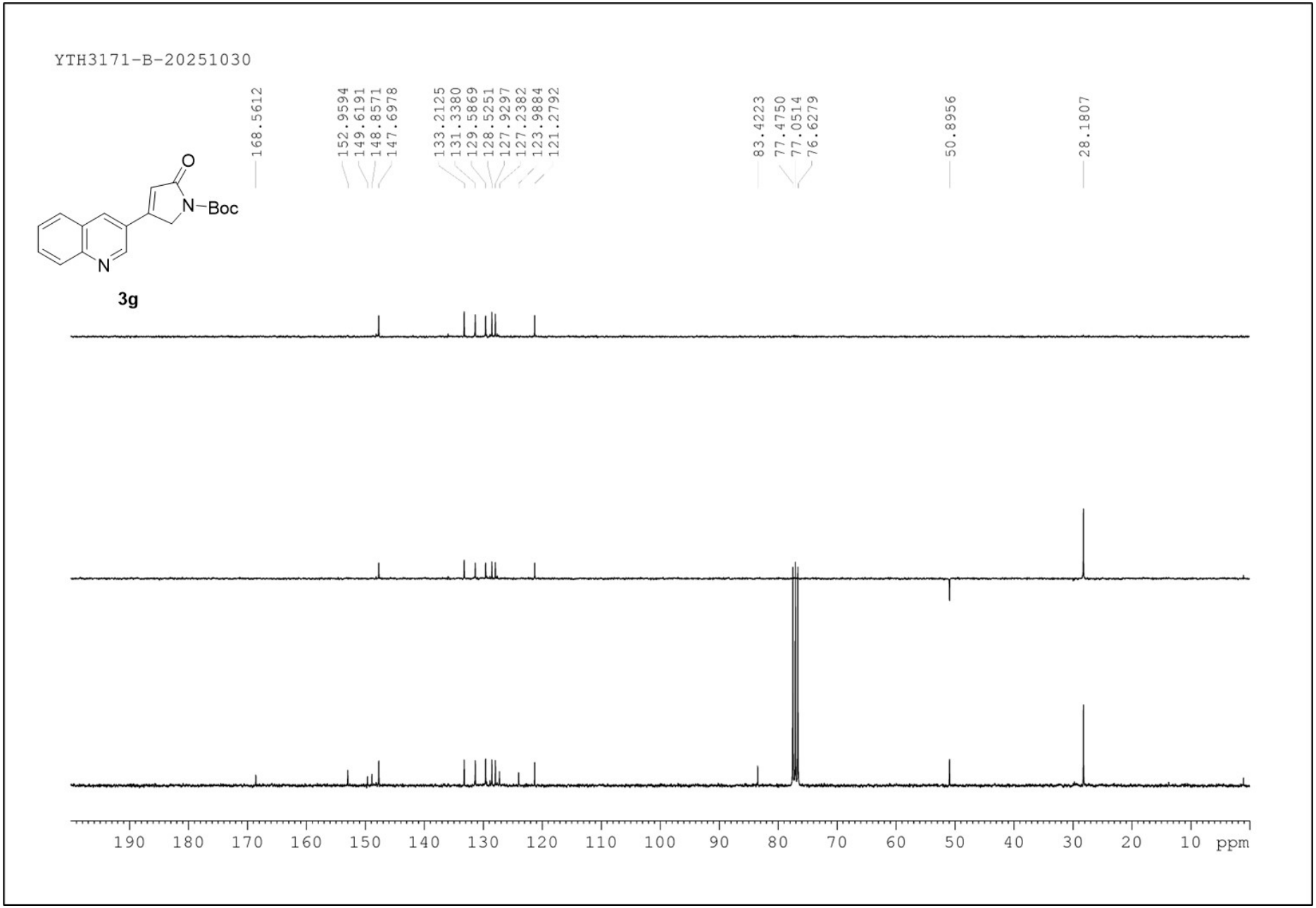


3f

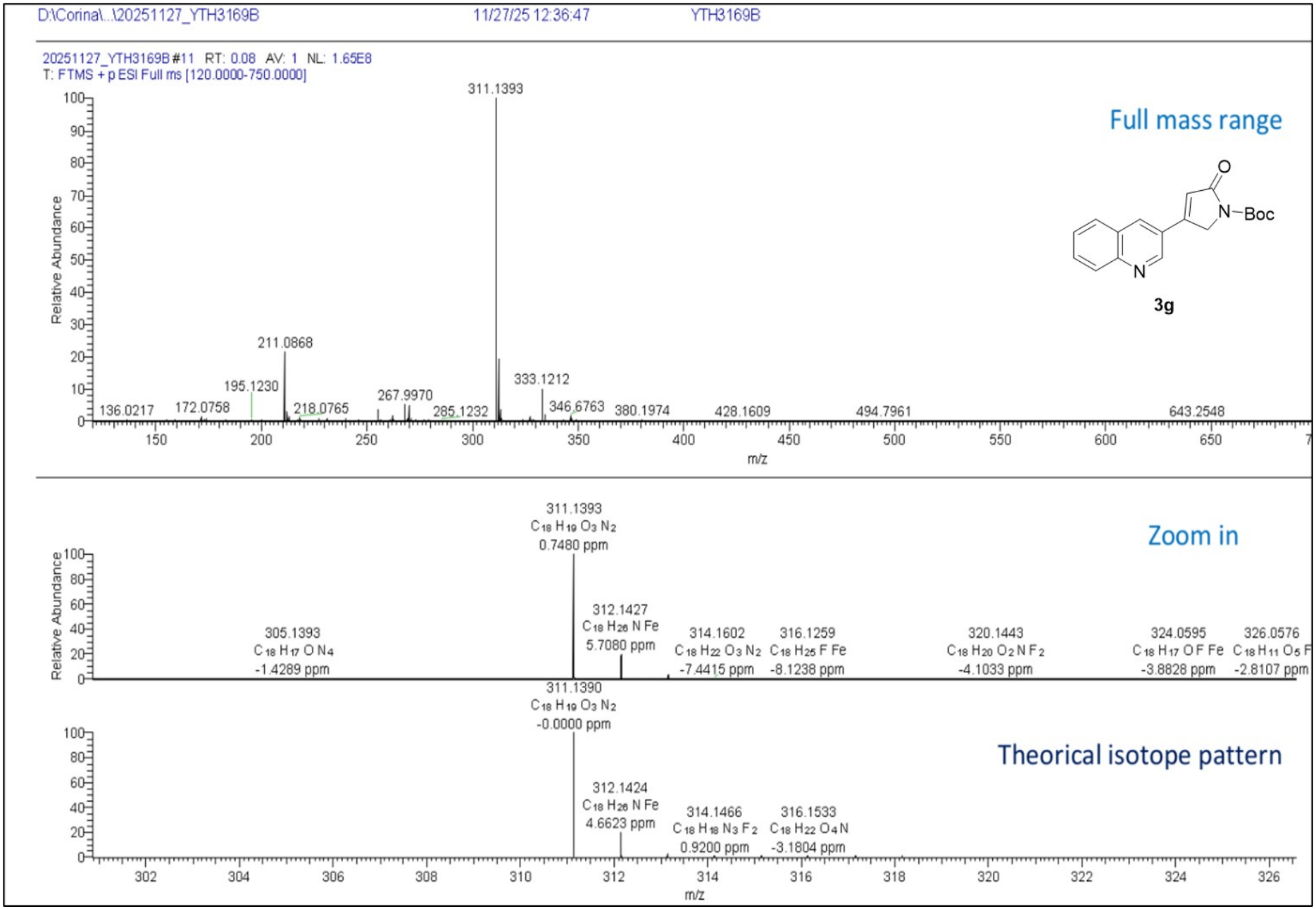
HRMS (ESI$^+$) of **3f**



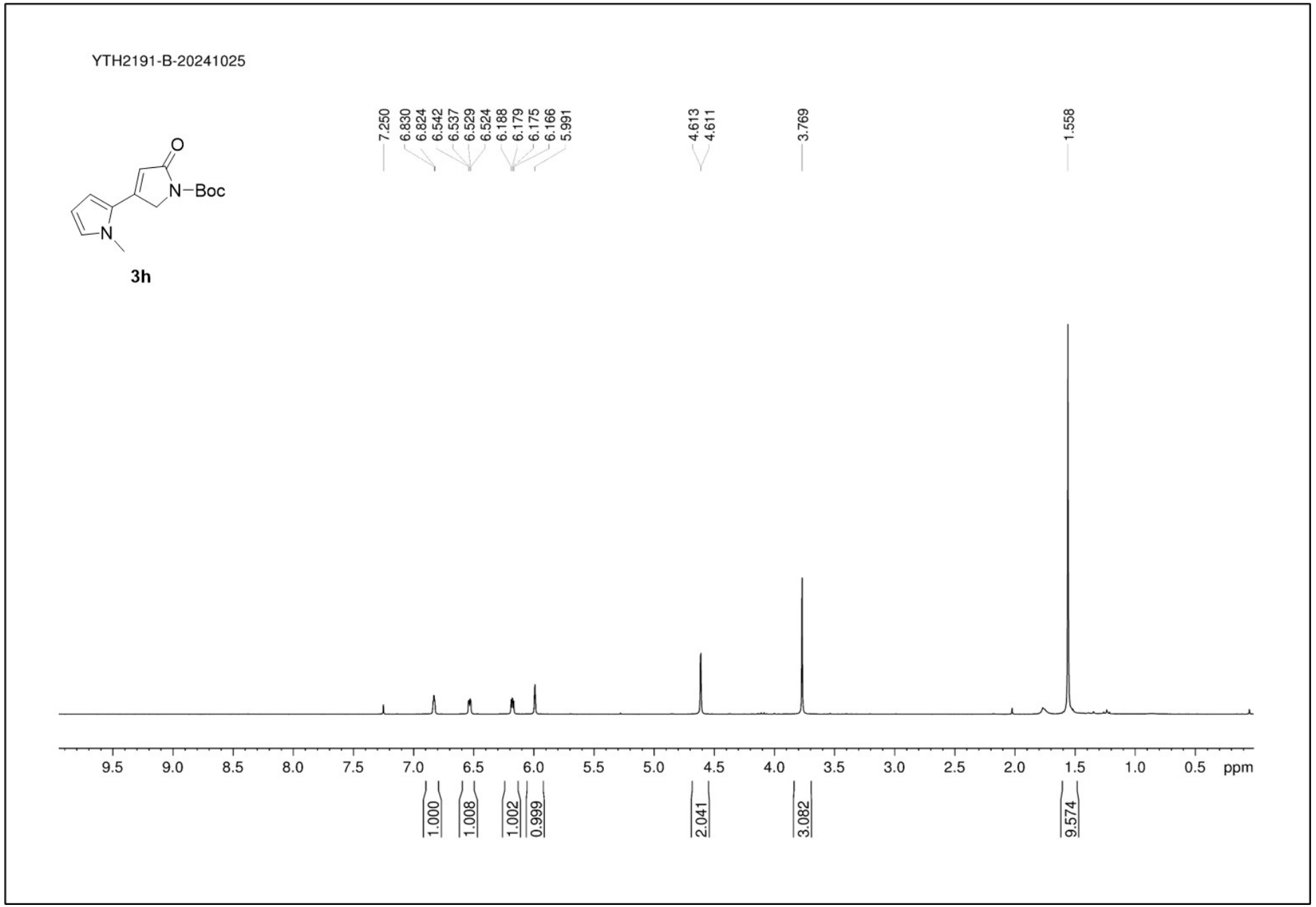
^{1}H NMR (300 MHz, $CDCl_3$) of **3g** (contaminated with tin residue)



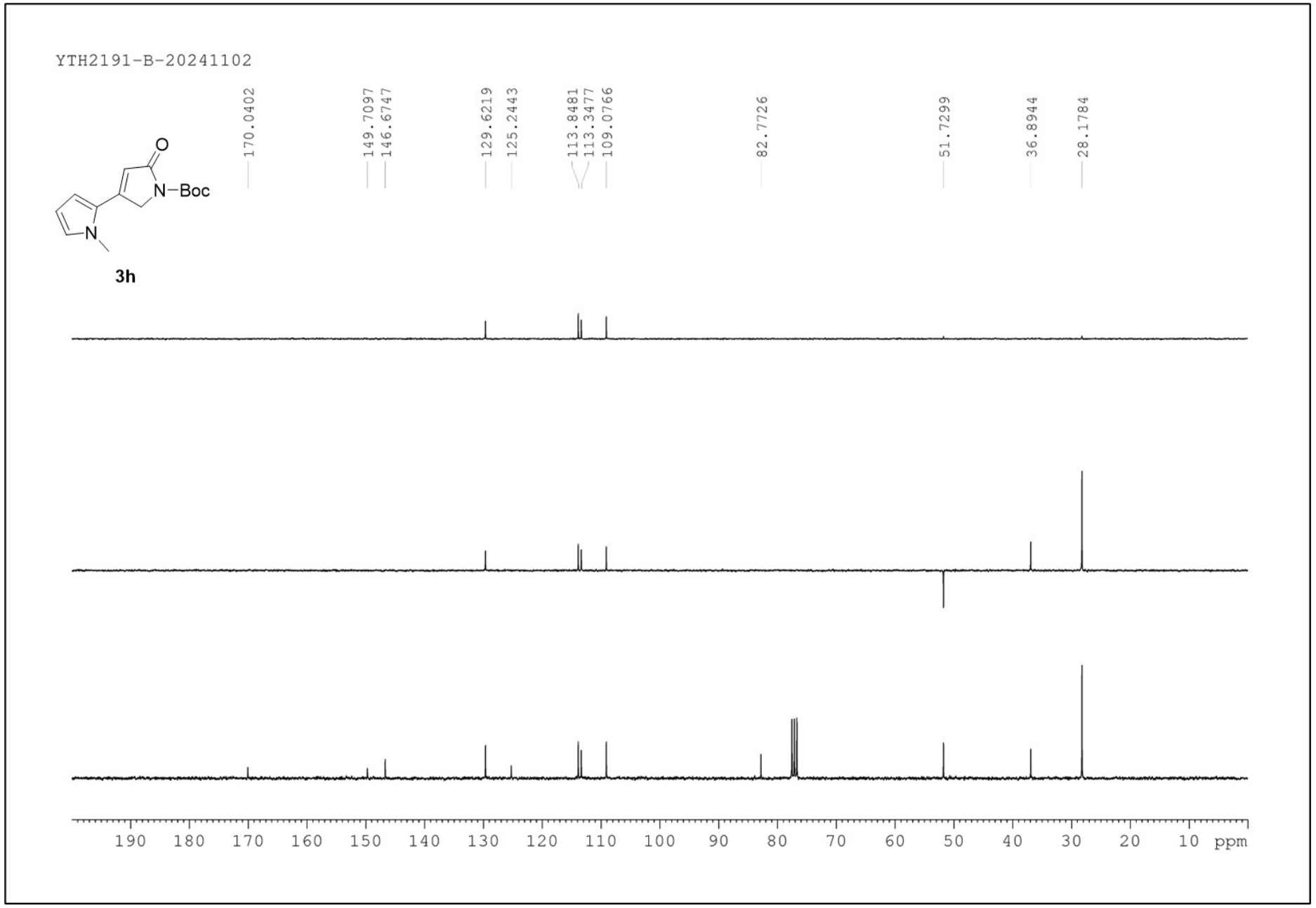
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3g**



HRMS (ESI$^+$) of **3g**



^{1}H NMR (300 MHz, $CDCl_3$) of **3h**



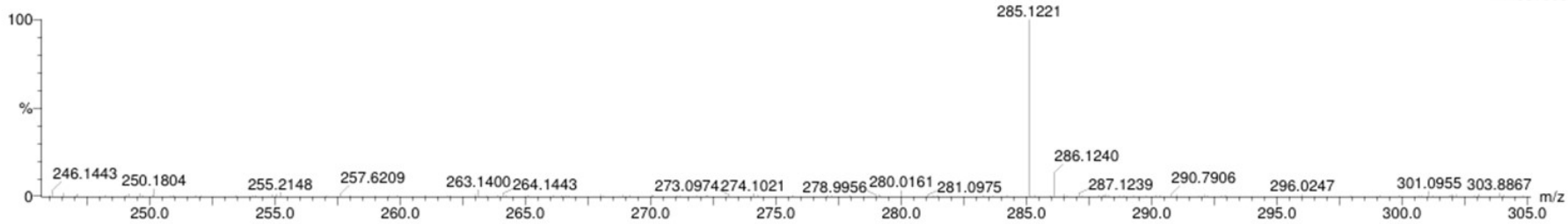
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **3h**

Elemental Composition Report

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

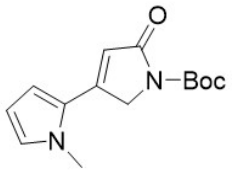
Monoisotopic Mass, Even Electron Ions
691 formula(e) evaluated with 248 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10 Na: 0-1
YTH2203B
251112FJU10 252 (2.455) Cm (250:255-(235:242+277:284))

1: TOF MS ES+
7.45e+003



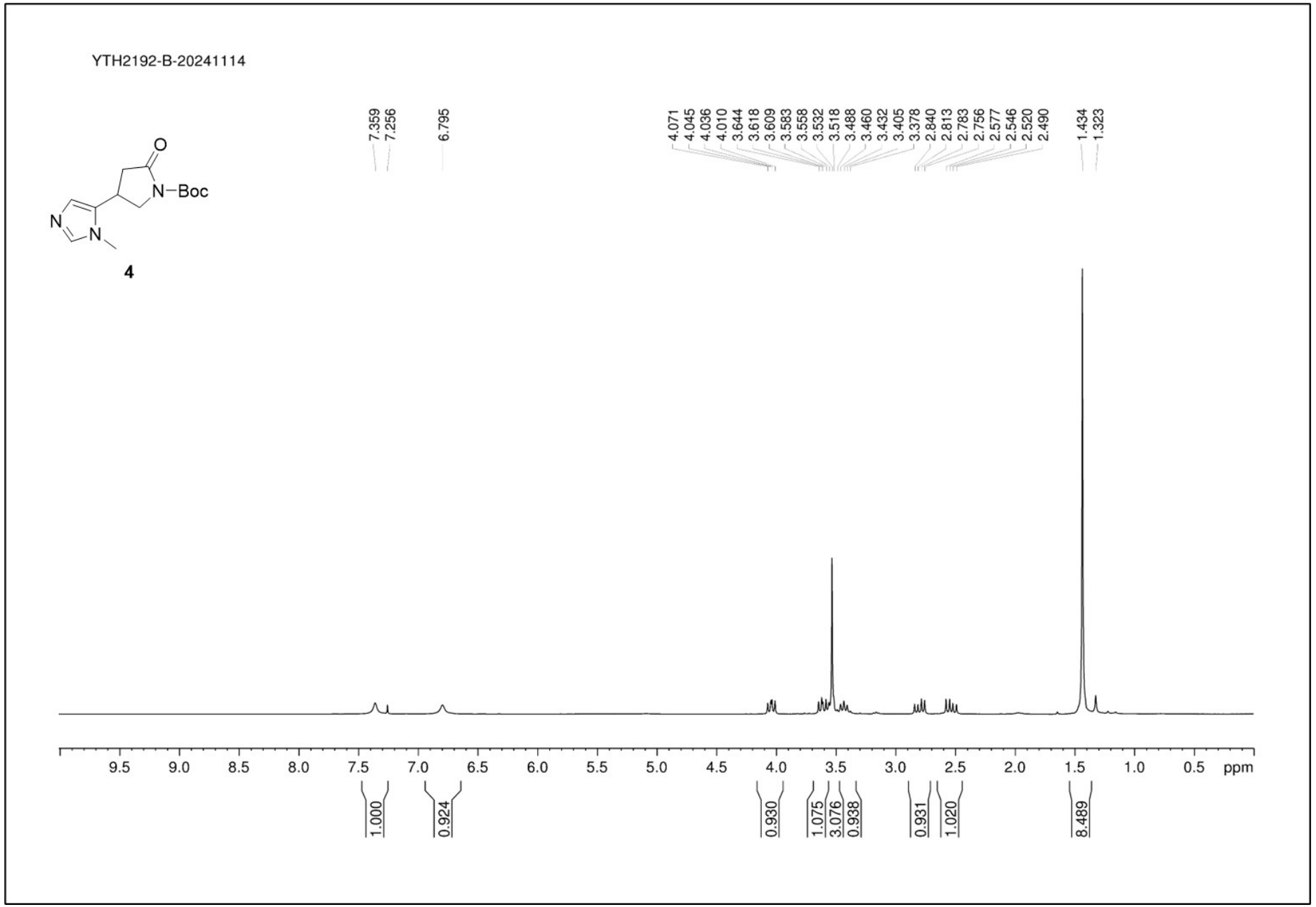
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
285.1221	100.00	285.1215	0.6	2.1	6.5	C14 H18 N2 O3 Na
		285.1212	0.9	3.2	10.5	C12 H13 N8 O
		285.1234	-1.3	-4.6	-6.5	C2 H22 N4 O10 Na
		285.1239	-1.8	-6.3	9.5	C16 H17 N2 O3
		285.1199	2.2	7.7	5.5	C11 H17 N4 O5
		285.1247	-2.6	-9.1	-1.5	C3 H18 N8 O6 Na
		285.1188	3.3	11.6	7.5	C10 H14 N8 O Na
		285.1258	-3.7	-13.0	-3.5	C4 H21 N4 O10
		285.1175	4.6	16.1	2.5	C9 H18 N4 O5 Na
		285.1172	4.9	17.2	6.5	C7 H13 N10 O3
		285.1271	-5.0	-17.5	1.5	C5 H17 N8 O6
		285.1274	-5.3	-18.6	-2.5	C7 H22 N2 O8 Na
		285.1159	6.2	21.7	1.5	C6 H17 N6 O7
		285.1287	-6.6	-23.1	2.5	C8 H18 N6 O4 Na
		285.1148	7.3	25.6	3.5	C5 H14 N10 O3 Na
		285.1298	-7.7	-27.0	0.5	C9 H21 N2 O8
		285.1135	8.6	30.2	-1.5	C4 H18 N6 O7 Na
		285.1311	-9.0	-31.6	5.5	C10 H17 N6 O4
		285.1118	10.3	36.1	-2.5	C H17 N8 O9
		285.1327	-10.6	-37.2	6.5	C13 H18 N4 O2 Na
		285.1100	12.1	42.4	10.5	C13 H13 N6 O2
		285.1346	-12.5	-43.8	-6.5	C H22 N6 O9 Na
		285.1352	-13.1	-45.9	9.5	C15 H17 N4 O2
		285.1087	13.4	47.0	5.5	C12 H17 N2 O6
		285.1359	-13.8	-48.4	-1.5	C2 H18 N10 O5 Na

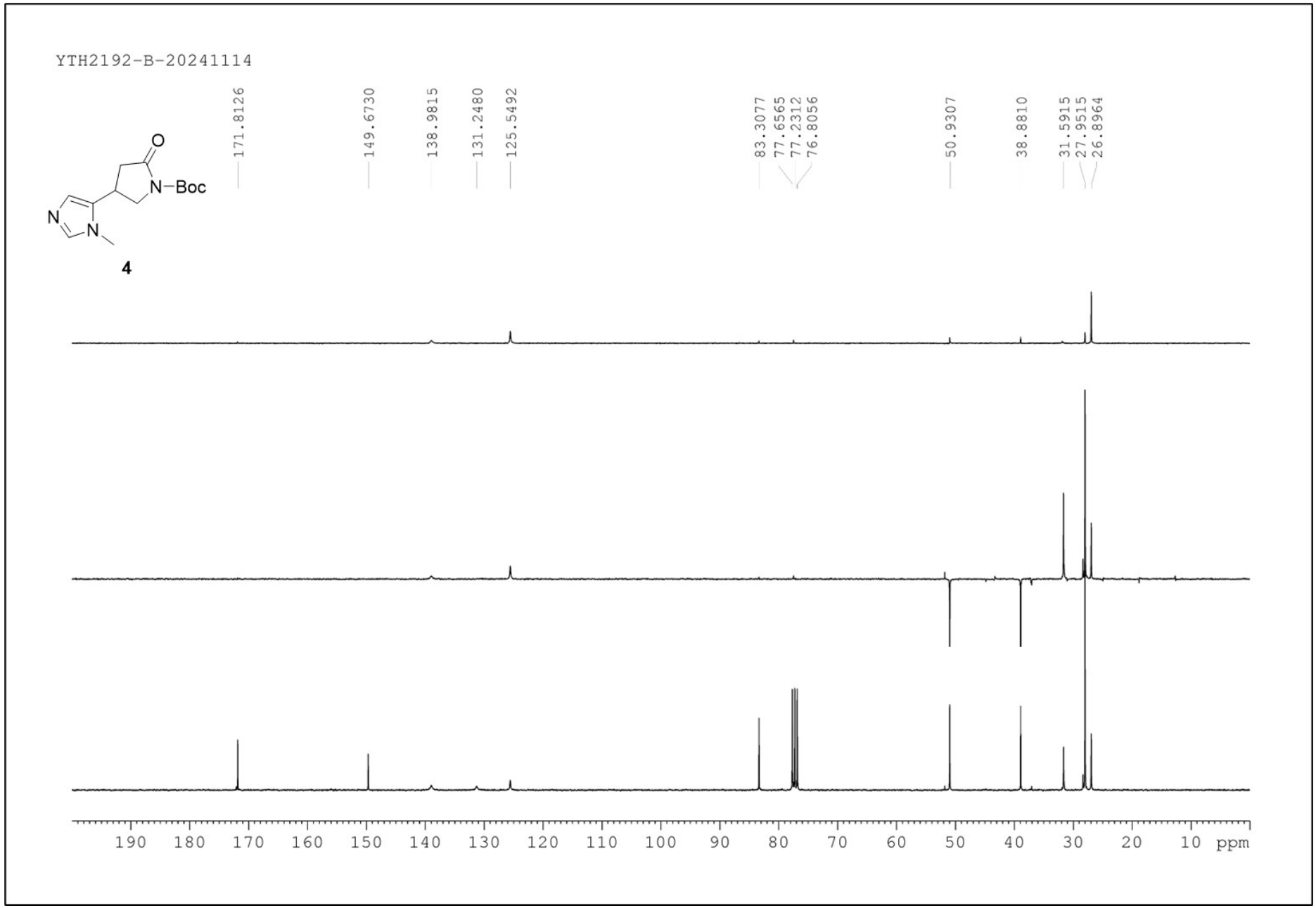


3h

HRMS (ESI$^+$) of **3h**



^{1}H NMR (300 MHz, $CDCl_3$) of **4** (trace of impurity)



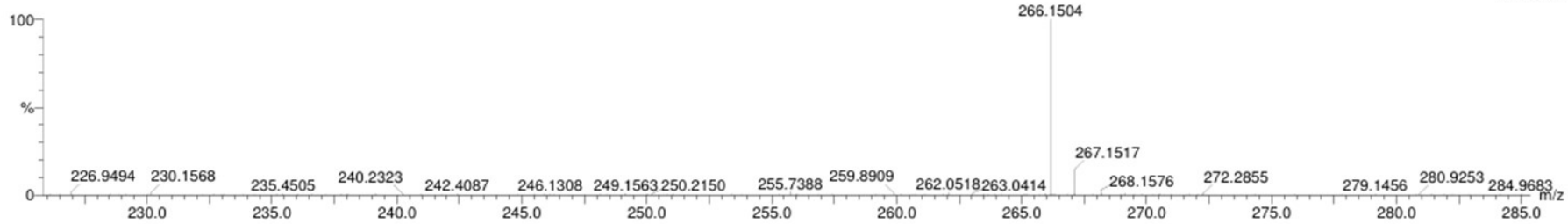
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **4**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

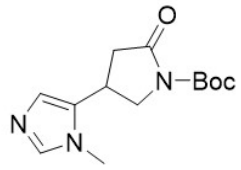
Monoisotopic Mass, Even Electron Ions
329 formula(e) evaluated with 118 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH2149A2
250919FJU24 60 (0.611) Cm (58:62-(42:48+79:85))

1: TOF MS ES+
1.22e+004



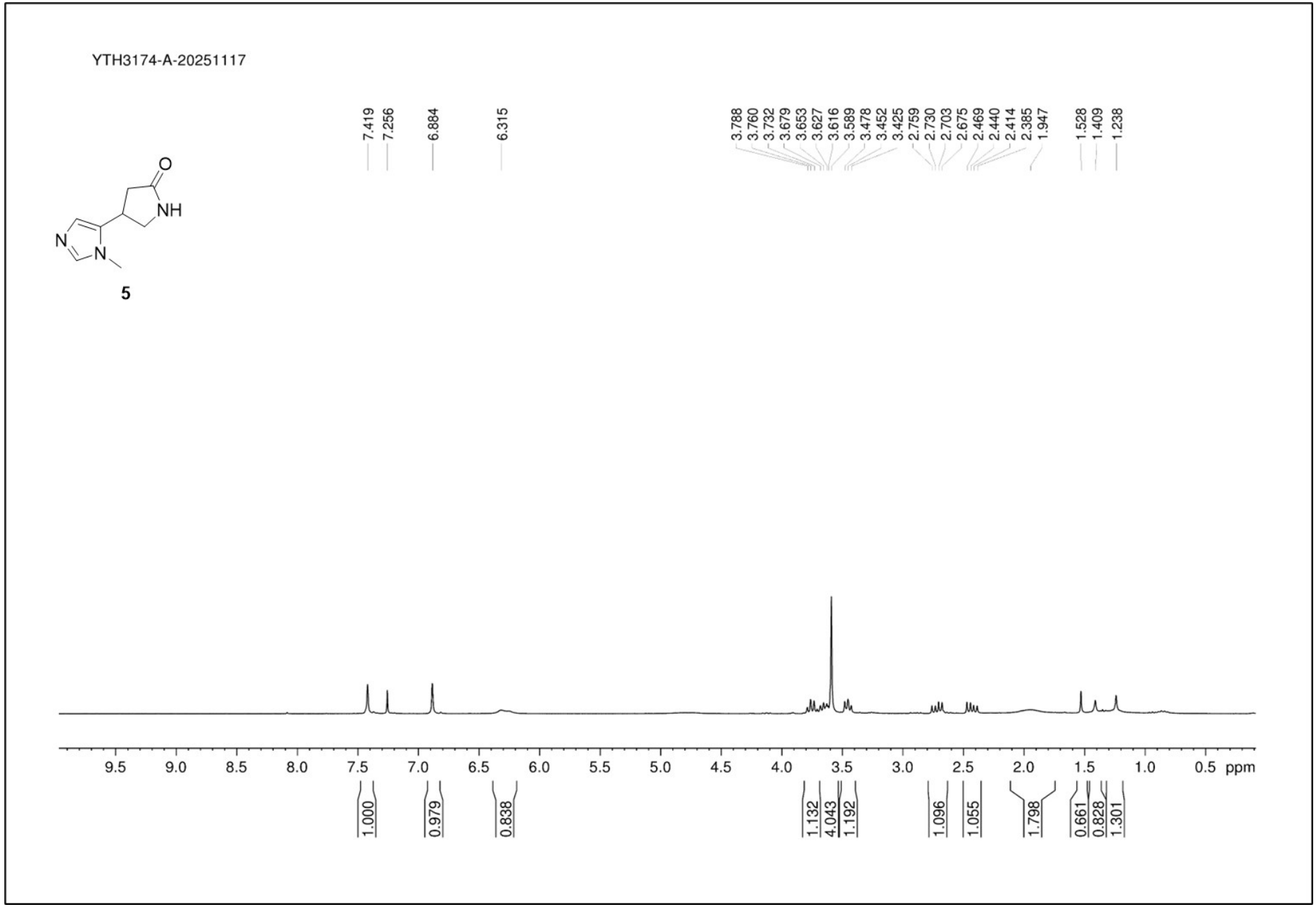
Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
266.1504	100.00	266.1505	-0.1	-0.4	5.5	C13 H20 N3 O3
		266.1523	-1.9	-7.1	-7.5	C H24 N5 O10
		266.1478	2.6	9.8	6.5	C9 H16 N9 O
		266.1537	-3.3	-12.4	-2.5	C2 H20 N9 O6
		266.1464	4.0	15.0	1.5	C8 H20 N5 O5
		266.1545	-4.1	-15.4	9.5	C18 H20 N O
		266.1451	5.3	19.9	-3.5	C7 H24 N O9
		266.1563	-5.9	-22.2	-3.5	C6 H24 N3 O8
		266.1577	-7.3	-27.4	1.5	C7 H20 N7 O4
		266.1424	8.0	30.1	-2.5	C3 H20 N7 O7
		266.1604	-10.0	-37.6	0.5	C11 H24 N O6
		266.1392	11.2	42.1	5.5	C14 H20 N O4
		266.1617	-11.3	-42.5	5.5	C12 H20 N5 O2
		266.1365	13.9	52.2	6.5	C10 H16 N7 O2
		266.1352	15.2	57.1	1.5	C9 H20 N3 O6
		266.1676	-17.2	-64.6	-3.5	C5 H24 N5 O7
		266.1325	17.9	67.3	2.5	C5 H16 N9 O4
		266.1689	-18.5	-69.5	1.5	C6 H20 N9 O3
		266.1312	19.2	72.1	-2.5	C4 H20 N5 O8
		266.1293	21.1	79.3	10.5	C16 H16 N3 O
		266.1716	-21.2	-79.7	0.5	C10 H24 N3 O5
		266.1729	-22.5	-84.5	5.5	C11 H20 N7 O
		266.1253	25.1	94.3	6.5	C11 H16 N5 O3
		266.1756	-25.2	-94.7	4.5	C15 H24 N O3
		266.1240	26.4	99.2	1.5	C10 H20 N O7

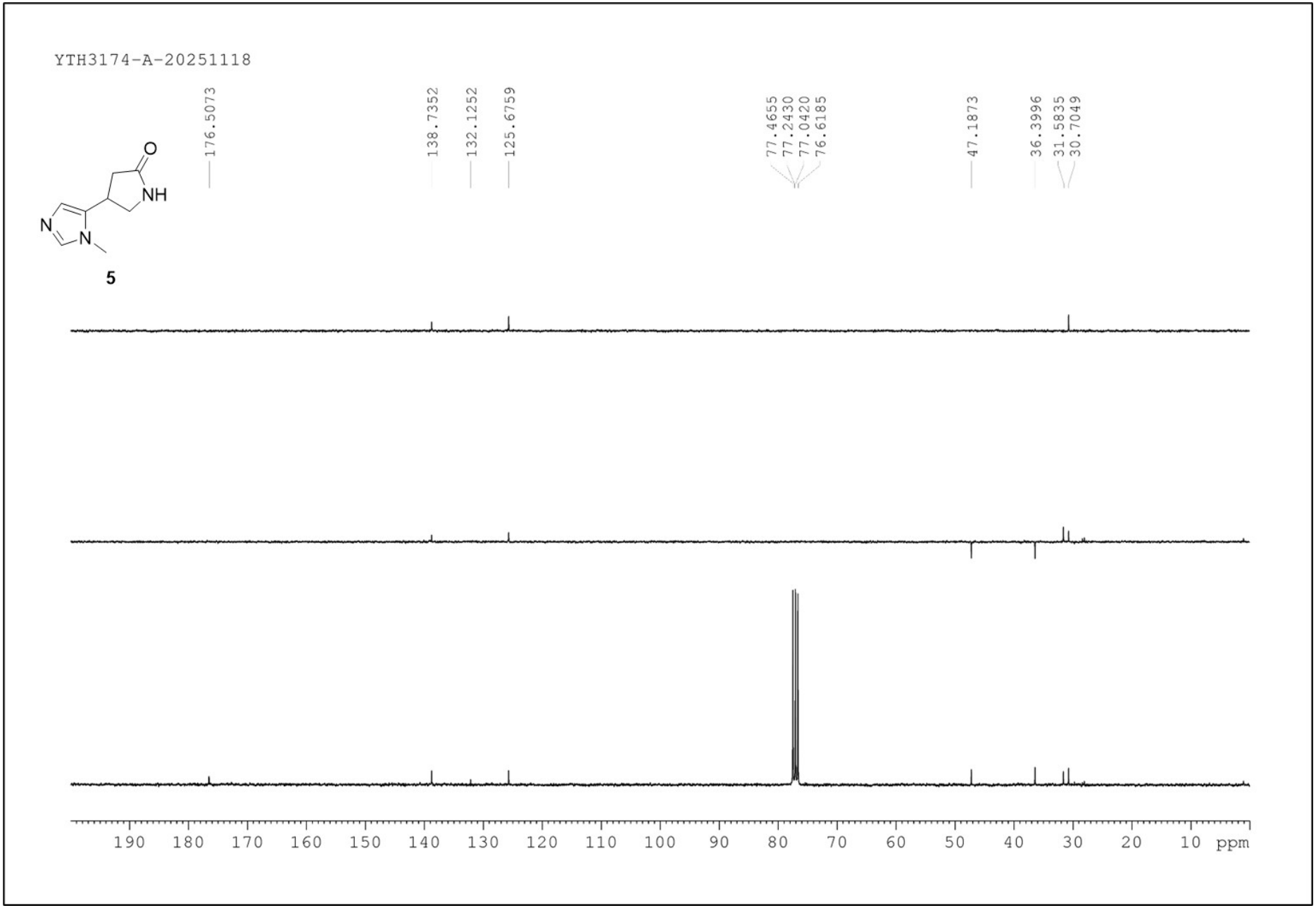


4

HRMS (ESI$^+$) of **4**



^{1}H NMR (300 MHz, $CDCl_3$) of **5** (trace of impurity)



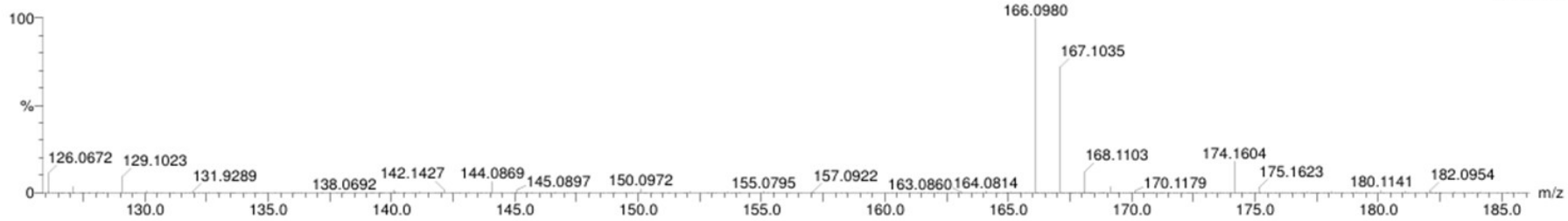
$^{13}C\{^{1}H\}$-DEPT NMR (75 MHz, $CDCl_3$) of **5**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 500.0 PPM / DBE: min = -10.0, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

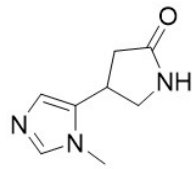
Monoisotopic Mass, Even Electron Ions
111 formula(e) evaluated with 36 results within limits (up to 25 closest results for each mass)
Elements Used:
C: 1-100 H: 1-100 N: 1-10 O: 1-10
YTH3173B
251210FJU04 32 (0.331) Cm (29:34-(12:20+55:63))

1: TOF MS ES+
5.83e+004



Minimum:	80.00			-10.0	
Maximum:	100.00		10.0	500.0	100.0

Mass	RA	Calc. Mass	mDa	PPM	DBE	Formula
166.0980	100.00	166.0980	0.0	0.0	4.5	C8 H12 N3 O
		166.0940	4.0	24.1	0.5	C3 H12 N5 O3
		166.0927	5.3	31.9	-4.5	C2 H16 N O7
		166.1039	-5.9	-35.5	-4.5	C H16 N3 O6
		166.1052	-7.2	-43.3	0.5	C2 H12 N7 O2
		166.1079	-9.9	-59.6	-0.5	C6 H16 N O4
		166.0868	11.2	67.4	4.5	C9 H12 N O2
		166.0828	15.2	91.5	0.5	C4 H12 N3 O4
		166.1165	-18.5	-111.4	0.5	C H12 N9 O
		166.1192	-21.2	-127.6	-0.5	C5 H16 N3 O3
		166.0729	25.1	151.1	5.5	C6 H8 N5 O
		166.1232	-25.2	-151.7	3.5	C10 H16 N O
		166.0715	26.5	159.5	0.5	C5 H12 N O5
		166.0689	29.1	175.2	1.5	C H8 N7 O3
		166.1291	-31.1	-187.2	-5.5	C3 H20 N O6
		166.1304	-32.4	-195.1	-0.5	C4 H16 N5 O2
		166.0617	36.3	218.5	5.5	C7 H8 N3 O2
		166.0576	40.4	243.2	1.5	C2 H8 N5 O4
		166.0563	41.7	251.1	-3.5	C H12 N O8
		166.1403	-42.3	-254.7	-5.5	C2 H20 N3 O5
		166.1416	-43.6	-262.5	-0.5	C3 H16 N7 O
		166.1443	-46.3	-278.8	-1.5	C7 H20 N O3
		166.0504	47.6	286.6	5.5	C8 H8 N O3
		166.0477	50.3	302.8	6.5	C4 H4 N7 O
		166.0464	51.6	310.7	1.5	C3 H8 N3 O5



5

HRMS (ESI$^+$) of **5**

d25314

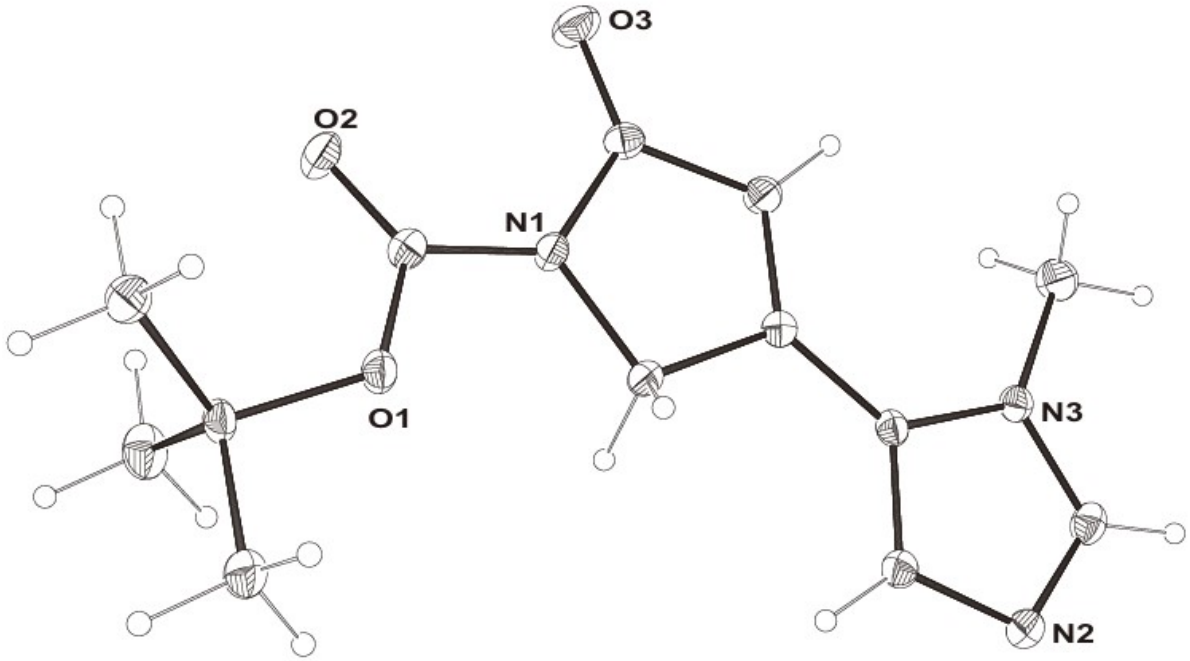


Figure 1. X-ray crystal structure of compound **3a**

Deposition Number 2501597

Crystallographic data were obtained from Dual X-ray Single Crystal Diffractometer, and the ellipsoid contour probability is 30%.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) shelx

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: shelx

Bond precision: C-C = 0.0044 A Wavelength=0.71073

Cell: a=5.8928(6) b=13.3792(15) c=16.7377(18)
alpha=90 beta=90 gamma=90

Temperature: 200 K

	Calculated	Reported
Volume	1319.6(2)	1319.6(2)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C13 H17 N3 O3	C13 H17 N3 O3
Sum formula	C13 H17 N3 O3	C13 H17 N3 O3
Mr	263.30	263.29
Dx,g cm-3	1.325	1.325
Z	4	4
Mu (mm-1)	0.096	0.096
F000	560.0	560.0
F000'	560.26	
h,k,lmax	7,15,19	7,15,19
Nref	2322[1372]	2319
Tmin,Tmax	0.991,0.998	0.928,0.998
Tmin'	0.927	

Correction method= # Reported T Limits: Tmin=0.928 Tmax=0.998

AbsCorr = MULTI-SCAN

Data completeness= 1.69/1.00 Theta(max)= 25.034

R(reflections)= 0.0414(1948) wR2(reflections)= 0.1066(2319)

S = 1.015 Npar= 176

The following ALERTS were generated. Each ALERT has the format **test-name_ALERT_alert-type_alert-level**. Click on the hyperlinks for more details of the test.

Alert level C

ABSTY02_ALERT_1_C An _exptl_absorpt_correction_type has been given without a literature citation. This should be contained in the _exptl_absorpt_process_details field.

Absorption correction given as multi-scan STRVA01_ALERT_4_C

Flack test results are ambiguous.

From the CIF: _refine_ls_abs_structure_Flack 0.500

From the CIF: _refine_ls_abs_structure_Flack_su 1.000 PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00438 Ang.

Alert level G

PLAT032_ALERT_4_G Std. Uncertainty on Flack Parameter Value High . 1.000 Report

PLAT063_ALERT_4_G Crystal Size Possibly too Large for Beam Size .. 0.79 mm

PLAT066_ALERT_1_G Predicted and Reported Tmin&Tmax Range Identical ? Check

PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary .. Please Do !

PLAT909_ALERT_3_G Percentage of I>2sig(I) Data at Theta(Max) Still 56% Note

PLAT910_ALERT_3_G Missing FCF Reflection(s) Below Theta(Min)[Deg]= 2.43 Note

0 1 1,

PLAT916_ALERT_2_G Hooft y and Flack x Parameter Values Differ by . 1.00 Check

PLAT933_ALERT_2_G Number of HKL-OMIT Records in Embedded .res File 1 Note

0 1 1,

PLAT965_ALERT_2_G The SHELXL WEIGHT Optimisation has not Converged Please Check

PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ..... 2.060 Note

Predicted wR2: Based on SigI**2 5.18 or SHELX Weight 10.51

PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 0 Info

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0 **ALERT level A** = Most likely a serious problem - resolve or explain

0 **ALERT level B** = A potentially serious problem, consider carefully

3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight

11 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data

4 ALERT type 2 Indicator that the structure model may be wrong or deficient

3 ALERT type 3 Indicator that the structure quality may be low 3

ALERT type 4 Improvement, methodology, query or suggestion

1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

PLATON version of 26/09/2025; check.def file version of 20/09/2025

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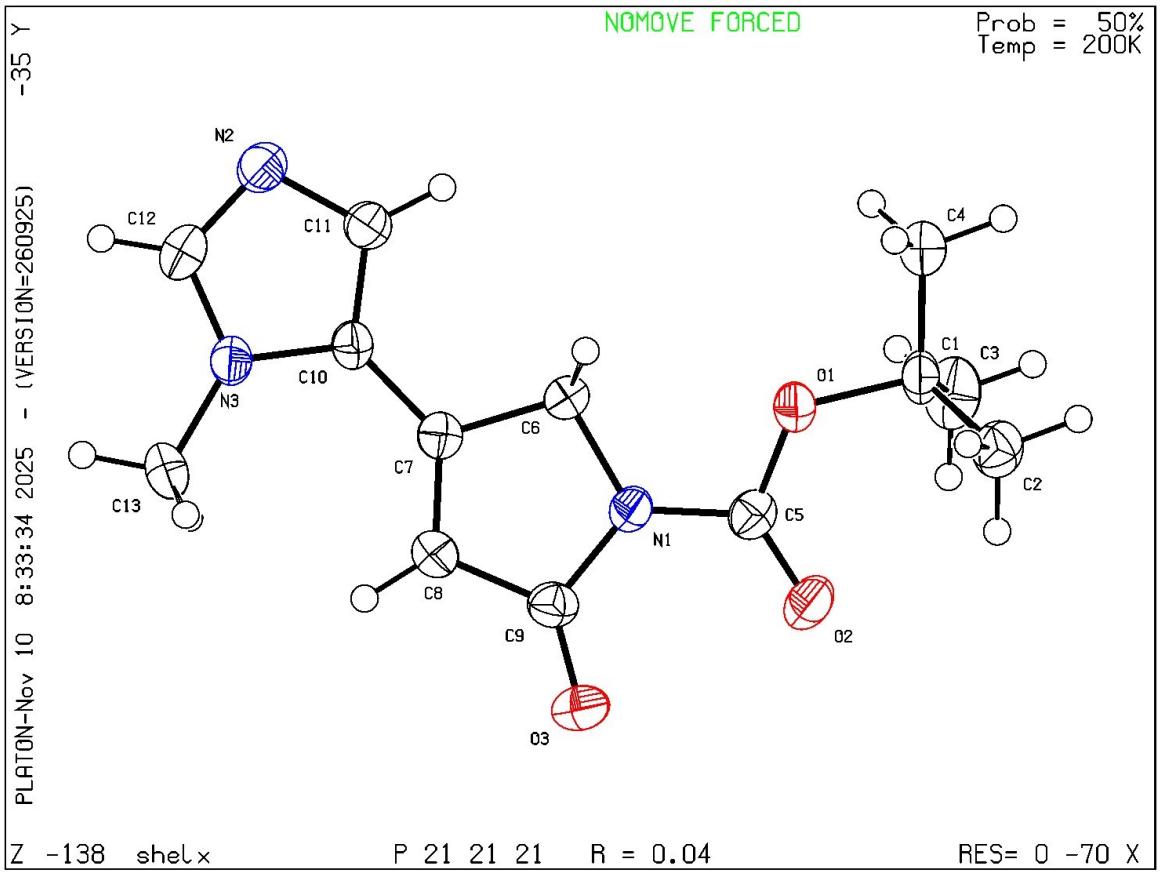
duplicate check

No duplication found

Datablock shelx - ellipsoid plot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Prob = 50%
Temp = 200K
-35 Y
PLATON-Nov 10 8:33:34 2025 - (VERSION=260925)
N2
C12
C11
C10
N3
C13
C6
C7
C8
C9
O3
N1
C5
O2
O1
C4
C1
C3
C2
Z -138 shelx
P 21 21 21
R = 0.04
RES= 0 -70 X

Table 1. Crystal data and structure refinement for d25314.

Identification code	shelx	
Empirical formula	C13 H17 N3 O3	
Formula weight	263.29	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 5.8928(6) Å	α= 90°.
	b = 13.3792(15) Å	β= 90°.
	c = 16.7377(18) Å	γ = 90°.
Volume	1319.6(2) $Å^3$	
Z	4	
Density (calculated)	1.325 Mg/m^3	
Absorption coefficient	0.096 mm^{-1}	
F(000)	560	
Crystal size	0.790 x 0.080 x 0.020 mm^3	
Theta range for data collection	2.434 to 25.034°.	
Index ranges	-5<=h<=7, -15<=k<=13, -19<=l<=18	
Reflections collected	8375	
Independent reflections	2319 [R(int) = 0.0530]	
Completeness to theta = 25.034°	99.9 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2319 / 0 / 176	
Goodness-of-fit on F^2	1.015	
Final R indices [I>2sigma(I)]	R1 = 0.0414, wR2 = 0.0951	
R indices (all data)	R1 = 0.0549, wR2 = 0.1066	
Absolute structure parameter	0.5(10)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.137 and -0.172 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for d25314. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	-1136(5)	4472(2)	5106(2)	32(1)
C(2)	-3697(5)	4526(3)	5105(2)	40(1)
C(3)	-263(7)	3444(3)	4880(2)	50(1)
C(4)	-185(6)	4808(3)	5905(2)	46(1)
C(5)	-562(5)	5221(2)	3771(2)	31(1)
C(6)	2854(5)	6327(2)	3822(2)	30(1)
C(7)	3945(5)	6969(2)	3191(2)	28(1)
C(8)	2723(6)	6904(3)	2511(2)	35(1)
C(9)	798(5)	6229(2)	2612(2)	32(1)
C(10)	5944(5)	7515(2)	3412(2)	28(1)
C(11)	7056(5)	7525(2)	4134(2)	32(1)
C(12)	8935(5)	8492(2)	3377(2)	35(1)
C(13)	6786(7)	8401(3)	2094(2)	49(1)
N(1)	927(4)	5878(2)	3402(2)	29(1)
N(2)	8926(4)	8135(2)	4112(2)	35(1)
N(3)	7198(4)	8153(2)	2928(2)	30(1)
O(1)	-122(4)	5213(2)	4550(1)	35(1)
O(2)	-1973(4)	4730(2)	3428(1)	44(1)
O(3)	-643(4)	6001(2)	2125(1)	49(1)

Table 3. Bond lengths [Å] and angles [°] for d25314.

C(1)-O(1)	1.484(4)
C(1)-C(2)	1.511(4)
C(1)-C(4)	1.517(4)
C(1)-C(3)	1.517(5)
C(2)-H(2A)	0.9800
C(2)-H(2B)	0.9800
C(2)-H(2C)	0.9800
C(3)-H(3A)	0.9800
C(3)-H(3B)	0.9800
C(3)-H(3C)	0.9800
C(4)-H(4A)	0.9800
C(4)-H(4B)	0.9800
C(4)-H(4C)	0.9800
C(5)-O(2)	1.205(4)
C(5)-O(1)	1.330(4)
C(5)-N(1)	1.387(4)
C(6)-N(1)	1.464(4)
C(6)-C(7)	1.505(4)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-C(8)	1.349(4)
C(7)-C(10)	1.434(4)
C(8)-C(9)	1.460(5)
C(8)-H(8)	0.9500
C(9)-O(3)	1.216(4)
C(9)-N(1)	1.405(4)
C(10)-C(11)	1.374(4)
C(10)-N(3)	1.390(4)
C(11)-N(2)	1.372(4)
C(11)-H(11)	0.9500
C(12)-N(2)	1.320(4)
C(12)-N(3)	1.348(4)
C(12)-H(12)	0.9500
C(13)-N(3)	1.455(4)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800

O(1)-C(1)-C(2)	111.7(3)
O(1)-C(1)-C(4)	101.9(2)
C(2)-C(1)-C(4)	110.9(3)
O(1)-C(1)-C(3)	108.2(3)
C(2)-C(1)-C(3)	112.4(3)
C(4)-C(1)-C(3)	111.3(3)
C(1)-C(2)-H(2A)	109.5
C(1)-C(2)-H(2B)	109.5
H(2A)-C(2)-H(2B)	109.5
C(1)-C(2)-H(2C)	109.5
H(2A)-C(2)-H(2C)	109.5
H(2B)-C(2)-H(2C)	109.5
C(1)-C(3)-H(3A)	109.5
C(1)-C(3)-H(3B)	109.5
H(3A)-C(3)-H(3B)	109.5
C(1)-C(3)-H(3C)	109.5
H(3A)-C(3)-H(3C)	109.5
H(3B)-C(3)-H(3C)	109.5
C(1)-C(4)-H(4A)	109.5
C(1)-C(4)-H(4B)	109.5
H(4A)-C(4)-H(4B)	109.5
C(1)-C(4)-H(4C)	109.5
H(4A)-C(4)-H(4C)	109.5
H(4B)-C(4)-H(4C)	109.5
O(2)-C(5)-O(1)	126.7(3)
O(2)-C(5)-N(1)	124.7(3)
O(1)-C(5)-N(1)	108.5(3)
N(1)-C(6)-C(7)	103.2(2)
N(1)-C(6)-H(6A)	111.1
C(7)-C(6)-H(6A)	111.1
N(1)-C(6)-H(6B)	111.1
C(7)-C(6)-H(6B)	111.1
H(6A)-C(6)-H(6B)	109.1
C(8)-C(7)-C(10)	133.6(3)
C(8)-C(7)-C(6)	109.1(3)
C(10)-C(7)-C(6)	117.4(3)
C(7)-C(8)-C(9)	110.9(3)
C(7)-C(8)-H(8)	124.5
C(9)-C(8)-H(8)	124.5
O(3)-C(9)-N(1)	125.8(3)

O(3)-C(9)-C(8)	128.3(3)
N(1)-C(9)-C(8)	105.9(3)
C(11)-C(10)-N(3)	104.6(3)
C(11)-C(10)-C(7)	128.6(3)
N(3)-C(10)-C(7)	126.8(3)
N(2)-C(11)-C(10)	111.4(3)
N(2)-C(11)-H(11)	124.3
C(10)-C(11)-H(11)	124.3
N(2)-C(12)-N(3)	113.2(3)
N(2)-C(12)-H(12)	123.4
N(3)-C(12)-H(12)	123.4
N(3)-C(13)-H(13A)	109.5
N(3)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
N(3)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(5)-N(1)-C(9)	126.6(3)
C(5)-N(1)-C(6)	122.5(2)
C(9)-N(1)-C(6)	110.9(2)
C(12)-N(2)-C(11)	104.1(3)
C(12)-N(3)-C(10)	106.6(3)
C(12)-N(3)-C(13)	125.8(3)
C(10)-N(3)-C(13)	127.6(3)
C(5)-O(1)-C(1)	122.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for d25314. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	29(2)	32(2)	36(2)	8(1)	6(1)	-6(1)
C(2)	30(2)	45(2)	44(2)	3(2)	4(2)	0(2)
C(3)	45(2)	36(2)	70(3)	5(2)	9(2)	6(2)
C(4)	44(2)	56(2)	39(2)	9(2)	0(2)	-17(2)
C(5)	28(2)	31(2)	35(2)	-4(1)	2(1)	-1(2)
C(6)	29(2)	33(2)	29(2)	1(1)	-4(1)	-6(1)
C(7)	27(2)	27(2)	29(2)	-1(1)	2(1)	2(1)
C(8)	35(2)	42(2)	27(2)	3(2)	0(2)	-5(2)
C(9)	31(2)	37(2)	28(2)	-3(1)	-1(2)	2(2)
C(10)	28(2)	25(2)	30(2)	4(1)	2(1)	-1(1)
C(11)	36(2)	29(2)	31(2)	0(2)	-1(1)	-2(1)
C(12)	28(2)	33(2)	45(2)	-2(2)	3(2)	-4(2)
C(13)	47(2)	62(3)	38(2)	19(2)	1(2)	-17(2)
N(1)	29(1)	31(2)	29(1)	-2(1)	1(1)	-7(1)
N(2)	32(2)	35(2)	39(2)	0(1)	-3(1)	-4(1)
N(3)	27(1)	31(2)	32(1)	5(1)	2(1)	-4(1)
O(1)	40(1)	36(1)	30(1)	3(1)	2(1)	-13(1)
O(2)	38(1)	50(2)	44(1)	-5(1)	0(1)	-17(1)
O(3)	44(2)	69(2)	35(1)	-1(1)	-10(1)	-16(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for d25314.

	x	y	z	U(eq)
H(2A)	-4298	4106	5536	60
H(2B)	-4276	4286	4591	60
H(2C)	-4178	5219	5188	60
H(3A)	-751	2956	5281	75
H(3B)	1398	3457	4855	75
H(3C)	-873	3255	4356	75
H(4A)	-753	4368	6328	69
H(4B)	-665	5496	6012	69
H(4C)	1476	4776	5890	69
H(6A)	3917	5807	4015	36
H(6B)	2339	6735	4281	36
H(8)	3070	7250	2031	42
H(11)	6590	7155	4590	38
H(12)	10052	8943	3184	42
H(13A)	7982	8849	1902	74
H(13B)	5310	8732	2044	74
H(13C)	6787	7787	1774	74