Electronic Supplementary Information

Copper-catalyzed aerobic benzylic sp^3 C-H oxidation mediated synthesis of 2,4,5-trisubstituted imidazoles via a domino multi-component reaction

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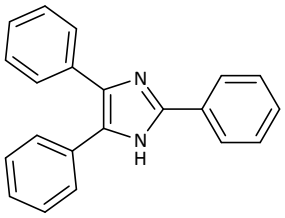
I. General Information

All reagents were purchased without further purification. All ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on a Bruker Advance III spectrometer operating at either 400 or 500 MHz. Chemical shifts (δ) were reported in ppm using the Dimethyl Sulfoxide-d_6 (DMSO-d_6) residual peak (δ 2.50) for ^{1}H NMR. Chemical shifts of ^{13}C NMR were reported relative to DMSO-d_6 (δ 39.51). The following abbreviations were used to describe peak splitting patterns when appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, J, were reported in Hertz unit (Hz). High-resolution electron-spray ionization (ESI) mass spectra were recorded on a time-of-flight (TOF) micromass spectrometer. Infra-Red (IR) spectra were recorded on Perkin Elmer FTIR Spectrometer. Absorption maxima are expressed in wavenumbers (cm^{-1}). Melting points were determined using Kofler hot-stage melting apparatus.

II. Synthetic Procedures and Characterization of Imidazole Derivatives

2,4,5-Triphenyl-1*H*-imidazole (3a, $C_{21}H_{16}N_2$)[1, 2]



2-Phenylacetaphenone (98.12 mg, 0.5 mmol), benzaldehyde (51.02 µL, 0.5 mmol), ammonium acetate (385.4 mg, 5.0 mmol) and catalytic copper chloride dihydrate (5 mol%) were mixed in a round bottomed flask with 1 mL Dimethylformamide (DMF). An oxygen balloon was attached and the mixture was heated at 50 °C for 24 hours. After cooling, ice water was added to the reaction mixture to form a white precipitate which was filtered and dried in an oven at 50 °C for 2 hours. The crude product was recrystallized from 9:1 acetone:water to afford 2, 4, 5-triphenyl-1*H*-imidazole as a white solid (129.20 mg, 87%). Mp 270-271 °C;

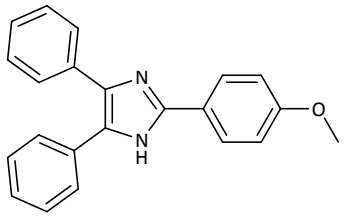
υ_{max} (neat, cm^{-1}): 3419 (N-H), 3039 (C-H), 1600 (C=N), 1461 (C=C), 1488, 1128 (C-N);

^{1}H NMR (400 MHz, DMSO-D_6): 12.71(s, 1H), 8.14-8.12 (d, *J* = 7.20 Hz, 2H,), 7.61-7.59 (d, *J* = 7.24 Hz, 2H), 7.54-7.53 (d, *J* = 7.12 Hz, 2H), 7.50-7.42 (m, 4H), 7.40-7.35 (m, 2H), 7.33-7.29 (t, *J* = 7.14 Hz, 2H), 7.24-7.23 (m, 1H);

^{13}C (100 MHz, DMSO-D_6): 145.5, 137.1, 135.2, 131.1, 130.3, 128.6, 128.6, 128.4, 128.2, 128.1, 127.7, 127.1, 126.5, 125.2;

ESI-MS (*m/z*): 297.1432 (100) $[M+H]^+$, 298.1463 (24).

2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (3b, $C_{22}H_{18}N_2O$)[2]



Prepared according to the procedure given for **3a**, using 4-methoxybenzaldehyde (60.84 µL, 0.5 mmol) obtained as a white solid (97.75 mg, 60%). Mp 230-233 °C;

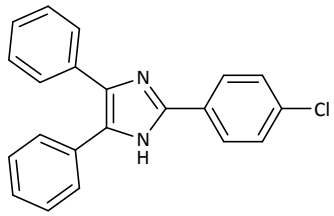
υ_{max} (neat, cm^{-1}): 3411 (N-H), 3024 (C-H), 2959, 1613 (C=N), 1492 (C=C), 1245 (C-N), 1175 (C-O-C);

^{1}H NMR (400 MHz, DMSO-D_6): 12.51 (s, 1H), 8.05-8.03 (d, *J* = 8.80 Hz, 2H), 7.58-7.56 (d, *J* = 7.40 Hz, 2H), 7.52-7.50 (d, *J* = 7.32 Hz, 2H), 7.45-7.41 (t, *J* = 7.46 Hz, 2H), 7.37-7.34 (t, *J* = 7.24 Hz, 1H), 7.32-7.28 (t, *J* = 7.50 Hz, 2H), 7.23-7.19 (t, *J* = 7.26 Hz, 1H), 7.06-7.04 (d, *J* = 8.84 Hz, 2H), 3.82 (s, 3H);

^{13}C (100 MHz, DMSO-D_6): 159.4, 145.6, 136.8, 135.3, 131.2, 128.6, 128.3, 128.1, 127.6, 127.6, 127.1, 126.7, 126.4, 123.1, 114.1, 55.5;

ESI-MS (*m/z*): 325.1070 (100) $[M-H]^+$, 326.1105 [M].

2-(4-Chlorophenyl)-4,5-diphenyl-1*H*-imidazole (3c, $C_{21}H_{15}ClN_2$)[3,4]



Prepared according to the procedure given for **3a**, using 4-chlorobenzaldehyde (70.29 mg, 0.5 mmol) obtained as a white solid (121.21 mg, 73%). Mp 263-264 °C;

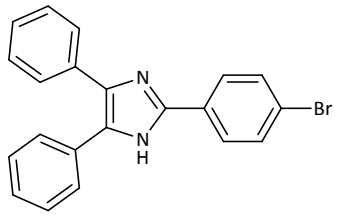
υ_{max} (neat, cm^{-1}): 3408 (N-H), 3059 (C-H), 3027, 1602 (C=N), 1485 (C=C), 1091 (C-N), 764 (C-Cl), 693;

^{1}H NMR (400 MHz, DMSO-D_6): 12.77 (s, 1H), 8.13-8.11 (d, J = 8.56 Hz, 2H), 7.55-7.53 (m, 6H), 7.36 (m, 6H);

^{13}C (100 MHz, DMSO-D_6): 144.5, 137.4, 135.1, 132.8, 131.0, 129.3, 128.8, 128.7, 128.5, 128.2, 127.9, 127.2, 126.9, 126.6;

ESI-MS (*m/z*): 331.1175 (100) $[M+H]^+$, 332.1209 (25), 333.1129 (41).

2-(4-Bromophenyl)-4,5-diphenyl-1*H*-imidazole (3d, $C_{21}H_{15}BrN_2$)[3,4]



Prepared according to the procedure given for **3a**, using 4-bromobenzaldehyde (92.15 mg, 0.5 mmol) obtained as a white solid (142.15 mg, 76%). Mp 250-252 °C;

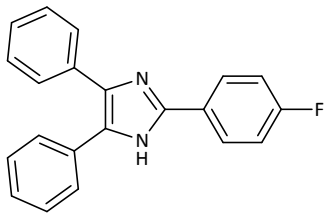
υ_{max} (neat, cm^{-1}): 3415 (N-H), 3057 (C-H), 2835, 1601 (C=N), 1482 (C=C), 1126 (C-N), 604 (C-Br);

^{1}H NMR (400 MHz, DMSO-D_6): 12.77 (s, 1H), 8.06-8.04 (d, *J* = 8.60 Hz, 2H), 7.69-7.67 (d, *J* = 8.60 Hz, 2H), 7.54-7.52 (m, 4H), 7.36 (m, 6H);

^{13}C (100 MHz, DMSO-D_6): 144.5, 137.4, 135.0, 131.7, 130.9, 129.6, 128.7, 128.4, 128.2, 127.9, 127.1, 126.6, 121.4;

ESI-MS (*m/z*): 375.0674 (100) $[M+H]^+$, 376.0711 (24), 377.0663 (97), 378.0696 (23).

2-(4-Fluorophenyl)-4,5-diphenyl-1*H*-imidazole (3e, $C_{21}H_{15}FN_2$)[3]



Prepared according to the procedure given for **3a**, using 4-fluorobenzaldehyde (53.63 µL, 0.5 mmol) obtained as a white solid (126.24 mg, 80%). Mp 189-190 °C;

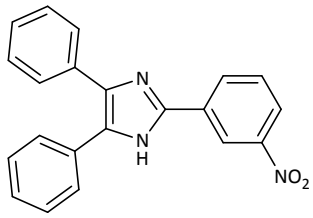
υ_{max} (neat, cm^{-1}): 3423 (N-H), 3028 (C-H), 2794, 1608 (C=N), 1492 (C=C), 1220 (C-N), 1159 (C-F);

^{1}H NMR (400 MHz, DMSO-D_6): 12.72 (s, 1H), 8.16-8.12 (dd, *J* = 8.66 Hz, 5.50 Hz, 2H), 7.57-7.55 (d, *J* = 7.32 Hz, 2H), 7.52-7.50 (d, *J* = 7.24 Hz, 2H), 7.46-7.42 (t, *J* = 7.42 Hz, 2H), 7.39-7.37 (d, *J* = 7.16 Hz, 1H), 7.35-7.28 (m, 4H), 7.24-7.20 (t, *J* = 7.22 Hz, 1H),;

^{13}C (100 MHz, DMSO-D_6): 163.4, 160.9, 144.7, 137.1, 135.1, 131.1, 128.7, 128.4, 128.3, 128.2, 127.8, 127.4, 127.3, 127.1, 127.1, 127.0, 126.6, 115.8, 115.6;

ESI-MS (*m/z*): 315.1533 (100) $[M+H]^+$, 316.1569 (24).

2-(3-Nitrophenyl)-4,5-diphenyl-1*H*-imidazole (3f, $C_{21}H_{15}N_3O_2$)[3,4]



Prepared according to the procedure given for **3a**, using 3-nitrobenzaldehyde (75.56 mg, 0.5 mmol) obtained as a yellow solid (129.61 mg, 76%). Mp 315-317 °C;

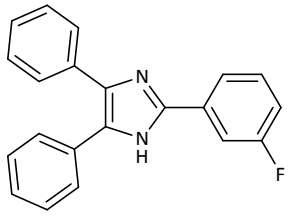
υ_{max} (neat, cm^{-1}): 3396 (N-H), 3057 (C-H), 2860, 1601 (C=N), 1521 (N-O), 1347, 1480 (C=C), 1416, 1252 (C-N), 1071j;

^{1}H NMR (400 MHz, DMSO-D_6): 13.09 (s, 1H), 8.96 (s, 1H), 8.53-8.51 (d, *J* = 7.76 Hz, 1H), 8.22-8.20 (d, *J* = 8.08 Hz, 1H), 7.80-7.76 (t, *J* = 7.98 Hz, 1H), 7.56-7.54 (d, *J* = 7.20 Hz, 4H), 7.38 (m, 6H);

^{13}C (100 MHz, DMSO-D_6): 148.4, 143.4, 137.7, 134.8, 131.8, 131.2, 130.7, 130.4, 129.2, 128.7, 128.4, 128.3, 128.1, 127.2, 126.8, 122.6, 119.4;

ESI-MS (*m/z*): 364.1623 (100) $[M+Na]^+$, 365.1659 (25).

2-(3-Fluorophenyl)-4,5-diphenyl-1*H*-imidazole (3g, $C_{21}H_{15}FN_2$)[5]



Prepared according to the procedure given for **3a**, using 3-fluorobenzaldehyde (53.04 µL, 0.5 mmol) obtained as a white solid (111.48 mg, 71%). Mp 284-285 °C;

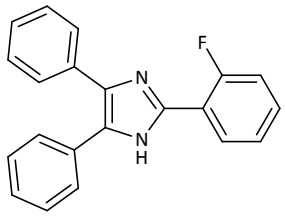
υ_{max} (neat, cm^{-1}): 3434 (N-H), 3056 (C-H), 2970, 1618 (C=N), 1588 (C=C), 1483, 1220 (C-N), 1072 (C-F);

^{1}H NMR (400 MHz, DMSO-D_6): 12.79 (s, 1H), 7.97-7.95 (d, *J* = 7.84 Hz, 1H), 7.92-7.89 (d, *J* = 10.23 Hz, 1H), 7.58-7.49 (m, 5H), 7.45 (m, 2H), 7.40-7.37 (m, 1H), 7.31 (m, 2H), 7.25-7.18 (m, 2H);

^{13}C (100 MHz, DMSO-D_6): 163.7, 161.3, 144.3, 144.2, 137.4, 134.9, 132.7, 132.6, 130.9, 130.8, 130.7, 128.6, 128.4, 128.2, 127.9, 127.1, 126.6, 121.2, 121.2, 114.9, 114.7, 111.8, 111.5;

ESI-MS (*m/z*): 337.1488 (100) $[M+Na]^+$, 338.1528 (24), 315.1644 (25).

2-(2-Fluorophenyl)-4,5-diphenyl-1*H*-imidazole (3h, $C_{21}H_{15}FN_2$)[5]



Prepared according to the procedure given for **3a**, using 2-fluorobenzaldehyde (52.68 µL, 0.5 mmol) obtained as a white solid (80.52 mg, 51%). Mp 238-240 °C;

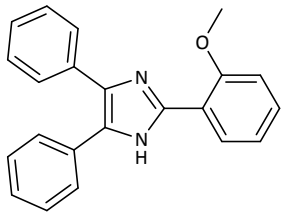
υ_{max} (neat, cm^{-1}): 3457 (N-H), 3058 (C-H), 1602 (C=N), 1484 (C=C), 1470, 1441, 1253 (C-N), 1101;

^{1}H NMR (400 MHz, DMSO-D_6): 12.53 (s, 1H), 8.03-7.99 (t, *J* = 7.70 Hz, 1H), 7.53-7.49 (m, 4H), 7.47-7.44 (t, *J* = 6.60 Hz, 1H), 7.38-7.32 (m, 8H);

^{13}C (100 MHz, DMSO-D_6): 160.1, 157.6, 140.9, 140.9, 137.3, 135.0, 130.9, 130.5, 130.4, 129.7, 129.7, 128.6, 128.6, 128.2, 127.9, 127.2, 126.6, 124.7, 124.7, 118.8, 118.6, 116.4, 116.2;

ESI-MS (*m/z*): 337.1273 (100) $[M+Na]^+$, 338.1314 (24).

2-(2-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (3i, $C_{22}H_{18}N_2O$)[2]



Prepared according to the procedure given for **3a**, using 2-methoxybenzaldehyde (60.40 µL, 0.5 mmol) obtained as a white solid (70.63 mg, 43%). Mp 225-226 °C;

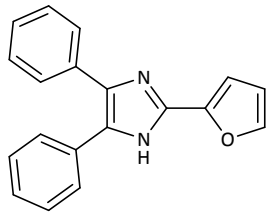
υ_{max} (neat, cm^{-1}): 3431 (N-H), 3186 (C-H), 3065, 2840, 1601 (C=N), 1585, 1481 (C=C), 1470, 1251 (C-N), 1100 (C-O-C);

^{1}H NMR (400 MHz, DMSO-D_6): 11.87 (s, 1H), 8.06-8.04 (dd, *J* = 7.72 Hz, 1.24 Hz, 1H), 7.54-7.52 (d, *J* = 7.52 Hz, 2H), 7.49-7.47 (m, 2H), 7.44-7.41 (t, *J* = 7.42 Hz, 2H), 7.38-7.35 (m, 2H), 7.31-7.27 (t, *J* = 7.27 Hz, 2H), 7.23-7.19 (m, 1H), 7.17-7.15 (d, *J* = 8.32 Hz, 1H), 7.09-7.05 (t, *J* = 7.46 Hz, 1H), 3.92 (s, 3H);

^{13}C (100 MHz, DMSO-D_6): 156.0, 143.2, 136.4, 135.3, 131.2, 129.7, 128.8, 128.6, 128.5, 128.1, 127.6, 127.4, 127.1, 126.4, 120.6, 118.9, 111.6; 55.6;

ESI-MS (*m/z*): 325.1070 (100) $[M+H]^+$, 326.1105 (26).

2-(2-Furan-2-yl)-4,5-diphenyl-1*H*-imidazole (3j, $C_{19}H_{14}N_2O$)[6]



Prepared according to the procedure given for **3a**, using furfural (41.42 μL, 0.5 mmol) obtained as a brown solid (62.21 mg, 32%). Mp 228-230 °C;

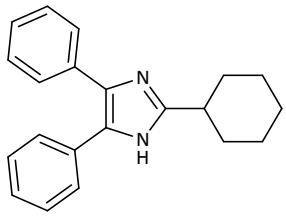
υ_{max} (neat, cm^{-1}): 3396 (N-H), 3025 (C-H), 1602 (C=N), 1445 (C=C), 1014 (C-N);

^{1}H NMR (400 MHz, DMSO-D_6): 12.80 (s, 1H), 7.80 (d, *J* = 1.04 Hz, 1H), 7.53-7.47 (m, 4H), 7.44-7.40 (t, *J* = 7.36 Hz, 2H), 7.38-7.34 (t, *J* = 7.12 Hz, 1H), 7.31-7.28 (t, *J* = 7.44 Hz, 2H), 7.24-7.20 (t, *J* = 7.24 Hz, 1H), 6.98-6.97 (d, *J* = 3.08 Hz, 1H), 6.65-6.64 (dd, *J* = 3.36 Hz, 1.76 Hz, 1H);

^{13}C (100 MHz, DMSO-D_6): 145.7, 143.0, 138.5, 136.9, 134.9, 130.8, 128.6, 128.3, 128.1, 127.8, 127.5, 127.1, 126.6, 111.8, 107.4;

ESI-MS (*m/z*): 309.0824 (100) $[M+Na]^+$, 310.0858 (22), 287.1017 (53).

2-(Cyclohexyl)-4,5-diphenyl-1*H*-imidazole (3k, $C_{21}H_{22}N_2$)[2]



Prepared according to the procedure given for **3a**, using cyclohexanecarboxaldehyde (60.57 µL, 0.5 mmol) obtained as a brown solid (39.26 mg, 26%). Mp 243-244 °C;

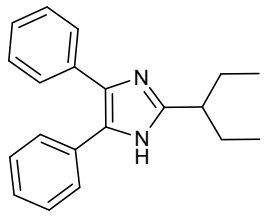
υ_{max} (neat, cm^{-1}): 3392 (N-H), 3031, 2929, 2848 (C-H), 1603 (C=N), 1448 (C=C), 1426, 1182 (C-N);

^{1}H NMR (400 MHz, DMSO-D_6): 11.91 (s, 1H), 7.49-7.47 (d, J = 7.40 Hz, 2H), 7.41-7.35 (m, 4H), 7.30-7.23 (m, 3H), 7.18-7.14 (m, 1H), 2.74-2.67 (m, 1H), 1.99-1.95 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.67 (m, 1H), 1.65-1.55 (m, 2H), 1.41-1.20 (m, 3H);

^{13}C (100 MHz, DMSO-D_6): 152.3, 135.8, 134.9, 131.6, 128.5, 127.9, 127.8, 127.1, 126.9, 125.9, 125.7, 37.2, 31.5, 25.7, 25.6;

ESI-MS (*m/z*): 303.2005 (100) $[M+H]^+$, 304.2094 (25).

2-(pentyl-3-yl)-4,5-diphenyl-1*H*-imidazole (3l, $C_{20}H_{22}N_2$)[7]



Prepared according to the procedure given for **3a**, using 2-ethylbutyraldehyde (61.52 µL, 0.5 mmol) obtained as a white solid (30.86 mg, 21%). Mp 241-243 °C;

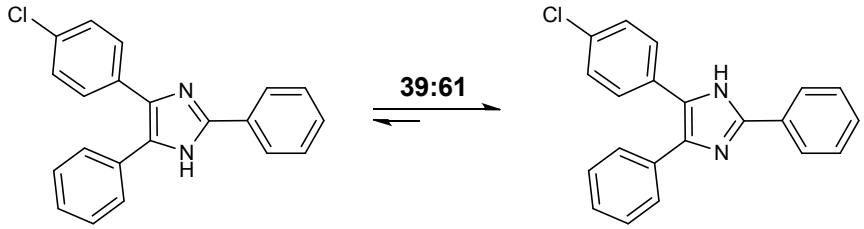
υ_{max} (neat, cm^{-1}): 3140(N-H), 3066, 3031, 2872, 29259, 2928 (C-H), 1062 (C=N), 1449 (C=C), 1428, 1188 (C-N);

^{1}H NMR (400 MHz, DMSO-D_6): 11.96 (s, 1H), 7.50-7.48 (d, *J* = 7.48 Hz, 2H), 7.42-7.35 (m, 4H), 7.31-7.23 (m, 3H), 7.18-7.14 (m, 1H), 2.62-2.55 (m, 1H), 1.77-1.63 (m, 4H), 0.85-0.81 (t, *J* = 7.28, 6H);

^{13}C (100 MHz, DMSO-D_6): 151.3, 135.8, 135.2, 131.7, 129.6, 129.5, 128.6, 128.1, 127.8, 127.1, 127.0, 126.1, 125.7, 42.3, 26.8, 12.1;

ESI-MS (*m/z*): 291.2007 (100) $[M+H]^+$, 292.2039 (23).

4-(4-Chlorophenyl)-2,5-diphenyl-1*H*-imidazole : 5-(4-Chlorophenyl)-2,4-diphenyl-1*H*-imidazole (3o, $C_{21}H_{15}ClN_2$)[2]



Prepared according to the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) obtained as a white solid (111.20 mg, 67%). Mp 241-243 °C;

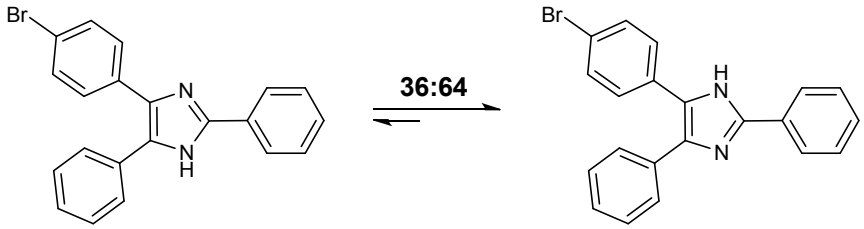
υ_{max} (neat, cm^{-1}): 3413 (N-H), 3054 (C-H), 1601 (C=N), 1501 (C=C), 1486, 1461, 1406, 1093 (C-N), 768 (C-Cl), 692;

^{1}H NMR (400 MHz, DMSO-D_6): 12.75-12.72 (s, 1H), 8.10-8.08 (d, *J* = 7.56 Hz, 2H), 7.58-7.44 (m, 8H), 7.41-7.23 (m, 4H);

^{13}C (100 MHz, DMSO-D_6): 145.8, 145.6, 137.7, 135.7, 134.9, 133.9, 132.2, 130.9, 130.8, 130.2, 130.1, 129.9, 129.8, 128.7, 128.6, 128.5, 128.5, 128.3, 128.2, 128.2, 127.9, 127.2, 126.9, 126.7, 125.2, 125.2;

ESI-MS (*m/z*): calcd for $C_{21}H_{15}ClN_2$ 330.0924, found 331.1006 $[M+H]^+$.

4-(4-Bromophenyl)-2,5-diphenyl-1*H*-imidazole : 5-(4-Bromophenyl)-2,4-diphenyl-1*H*-imidazole (3p, $C_{21}H_{15}BrN_2$)[2]



Prepared according to the procedure given for **3a**, using 2-(4-bromophenyl)acetophenone (137.60 mg, 0.5 mmol) obtained as a white solid (135.10 mg, 72%). Mp 253-255 °C;

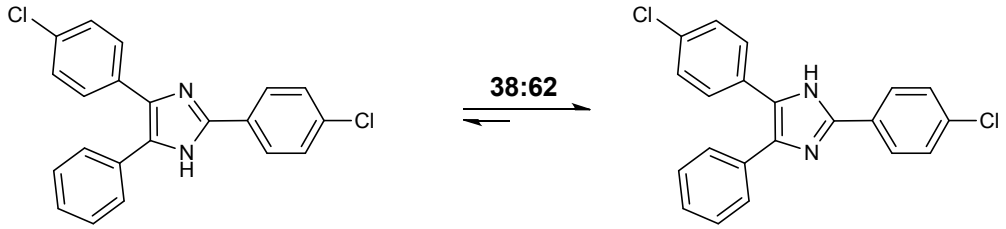
υ_{max} (neat, cm^{-1}): 3408 (N-H), 3054 (C-H), 2780, 1598 (C=N), 1539, 1483 (C=C), 1403, 1010 (C-N), 604 (C-Br), 542;

^1H NMR (400 MHz, DMSO-D_6): 12.74-12.71 (s, 1H), 8.09-8.07 (d, 2H), 7.64-7.25 (m, 12H);

^{13}C (100 MHz, DMSO-D_6): 145.9, 145.8, 137.8, 135.9, 134.9, 134.4, 131.6, 131.2, 130.8, 130.3, 130.2, 128.9, 128.8, 128.7, 128.6, 128.4, 128.4, 128.1, 127.3, 126.9, 126.8, 125.3, 120.8, 119.5;

ESI-MS (*m/z*): calcd for $C_{21}H_{15}BrN_2$ 374.0419, found 374.0492 $[M]^+$.

2,4-Bis-(4-chlorophenyl)-5-phenyl-1*H*-imidazole : 2,5-Bis-(4-chlorophenyl)-4-phenyl-1*H*-imidazole (3q, $C_{21}H_{14}Cl_2N_2$)



Prepared according to the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-chlorobenzaldehyde (70.29 mg, 0.5 mmol) obtained as a white solid (117.35 mg, 64%). Mp 250-252 °C;

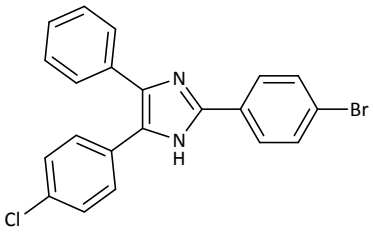
υ_{max} (neat, cm^{-1}): 3419 (N-H), 3062 (C-H), 2827, 1600 (C=N), 1500, 1479 (C=C), 1446, 1088 (C-N), 606 (C-Cl), 557, 480;

^{1}H NMR (400 MHz, DMSO-D_6): 12.82-12.80 (s, 1H), 8.10-8.08 (d, *J* = 8.56 Hz, 2H), 7.56-7.24 (m, 11H);

^{13}C (100 MHz, DMSO-D_6): 144.7, 144.6, 137.9, 135.9, 1348, 133.8, 132.9, 132.3, 131.0, 130.6, 129.9, 129.7, 129.0, 128.9, 128.7, 128.7, 128.5, 128.5, 128.3, 128.2, 128.0, 127.2, 127.2, 126.9, 126.8;

ESI-MS (*m/z*): calcd for $C_{21}H_{14}Cl_2N_2$ 364.0534, found 365.0616 $[M+H]^+$.

2-(4-Bromophenyl)-5-(4-chlorophenyl)-4-phenyl-1*H*-imidazole (3r, $C_{21}H_{14}BrClN_2$)



Prepared according to the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-bromobenzaldehyde (92.15 mg, 0.5 mmol) obtained as a white solid (139.16 mg, 68%). Mp 249-250 °C;

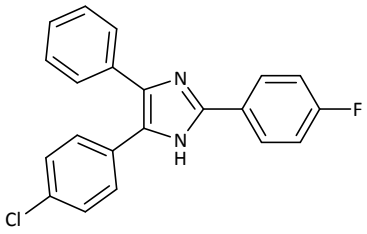
υ_{max} (neat, cm^{-1}): 3411 (N-H), 3064 (C-H), 2632, 1600 (C=N), 1500, 1479 (C=C), 1446, 1090 (C-N), 1068, 1008, 968, 773 (C-Cl), 728, 680, 526 (C-Br);

^{1}H NMR (400 MHz, DMSO-D_6): 12.82 (s, 1H), 8.04-8.02 (d, J = 8.52 Hz, 2H), 7.69-7.67 (d, J = 8.52 Hz, 2H), 7.55-7.50 (t, J = 8.38 Hz, 4H), 7.41 (m, 5H);

^{13}C (100 MHz, DMSO-D_6): 144.8, 131.7, 129.4, 128.9, 128.5, 128.2, 127.2, 125.3, 121.6

ESI-MS (*m/z*): calcd for $C_{21}H_{14}ClBrN_2$ 408.0029, found 409.0110 $[M+H]^+$.

5-(4-Chlorophenyl)-2-(4-fluorophenyl)-4-phenyl-1*H*-imidazole (3s, $C_{21}H_{14}ClFN_2$)



Prepared according to the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-fluorobenzaldehyde (53.63 µL, 0.5 mmol) obtained as a white solid (90.50, 52%). Mp 246-249 °C;

υ_{max} (neat, cm^{-1}): 3435 (N-H), 3067 (C-H), 2949, 2786, 1606 (C=N), 1503, 1488 (C=C), 1447, 1222 (C-F), 1091 (C-N), 774 (C-Cl), 698, 623;

^{1}H NMR (400 MHz, DMSO-D_6): 12.77 (s, 1H), 8.15-8.12 (dd, 2H), 7.57-7.52 (t, 4H), 7.41-7.31 (m, 7H);

^{13}C (100 MHz, DMSO-D_6): 163.5, 161.6, 145.4, 138.0, 136.2, 134.9, 134.1, 132.6, 131.5, 130.9, 130.2, 129.9, 129.0, 128.7, 128.5, 128.4, 127.8, 127.8, 127.6, 127.3, 127.2, 127.0, 126.9, 116.1, 115.9;

ESI-MS (*m/z*): calcd for $C_{21}H_{14}ClFN_2$ 348.0830, found 349.0907 $[M+H]^+$.

III. References

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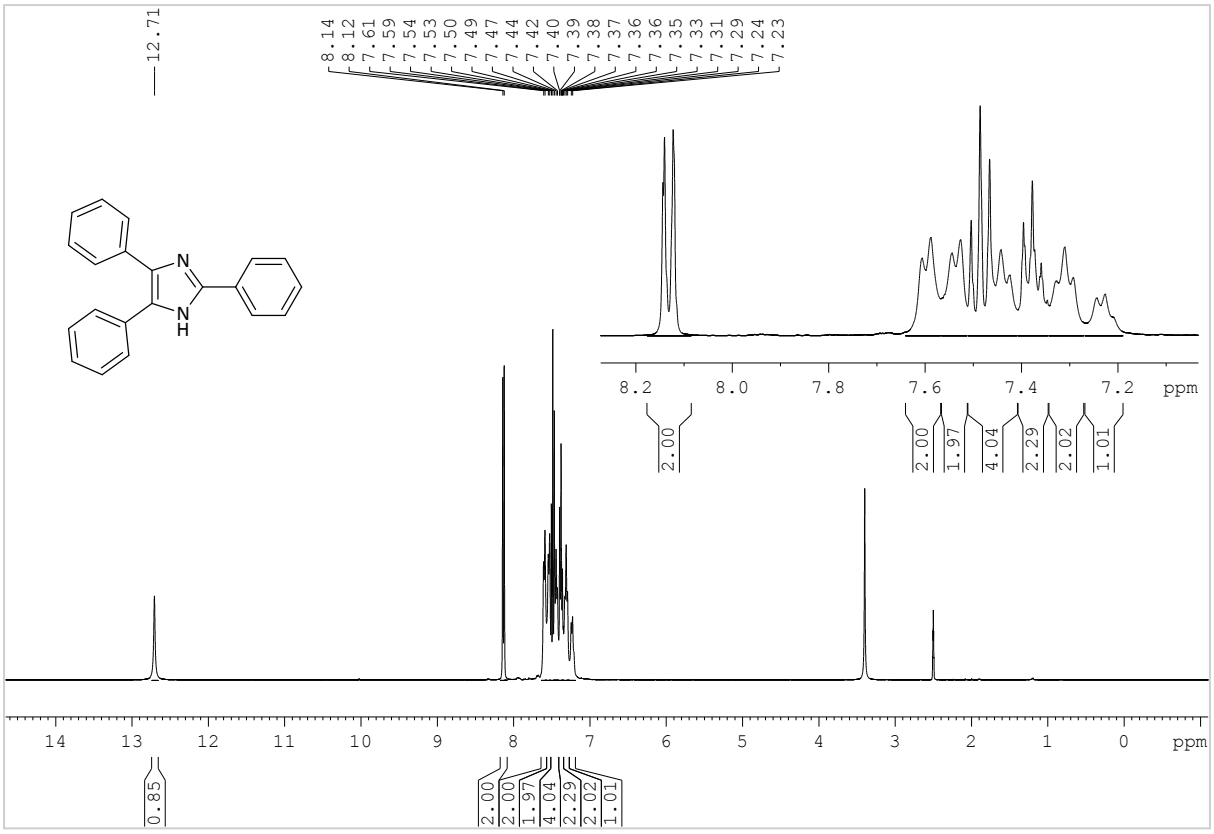
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8.12
7.61
7.59
7.54
7.53
7.50
7.49
7.47
7.44
7.42
7.40
7.39
7.38
7.37
7.36
7.36
7.35
7.33
7.31
7.29
7.24
7.23
N
N
H
8.2
8.0
7.8
7.6
7.4
7.2
ppm
2.00
2.00
1.97
4.04
2.29
2.02
1.01
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
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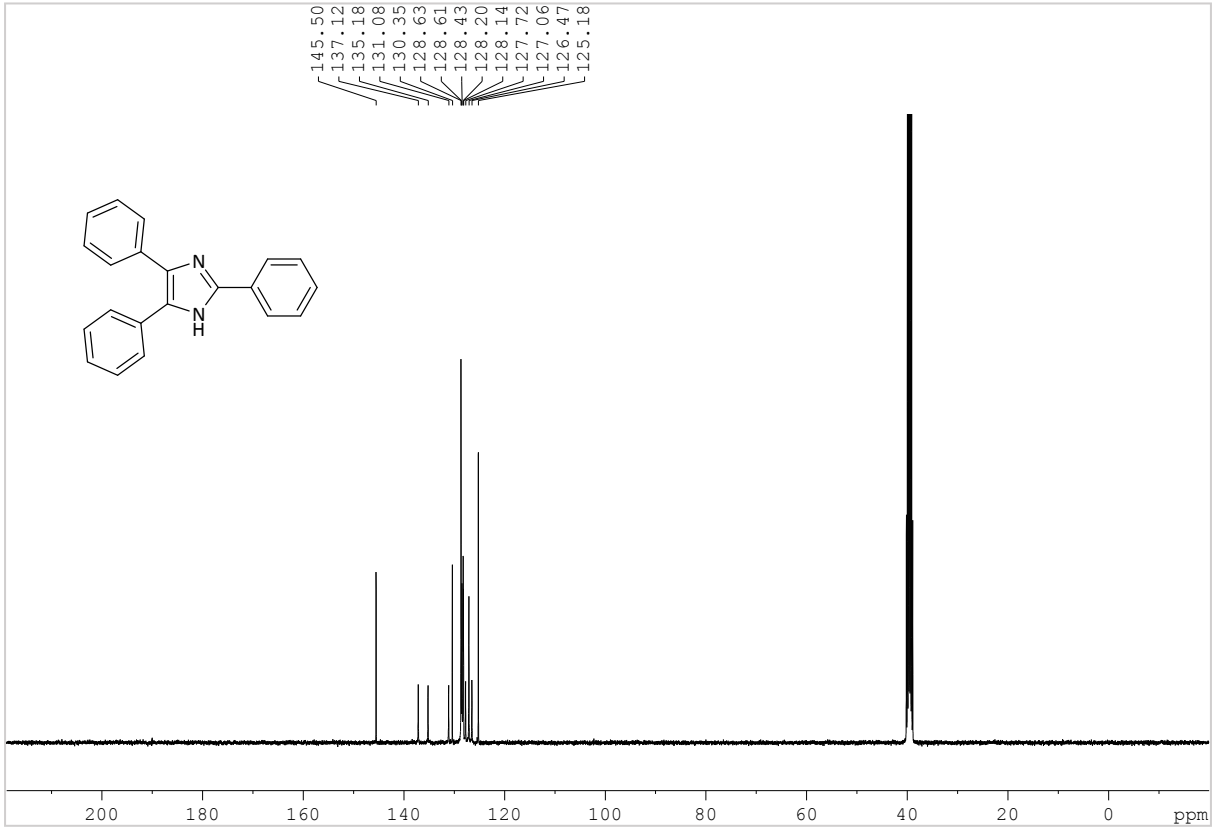
2.00
2.00
1.97
4.04
2.29
2.02
1.01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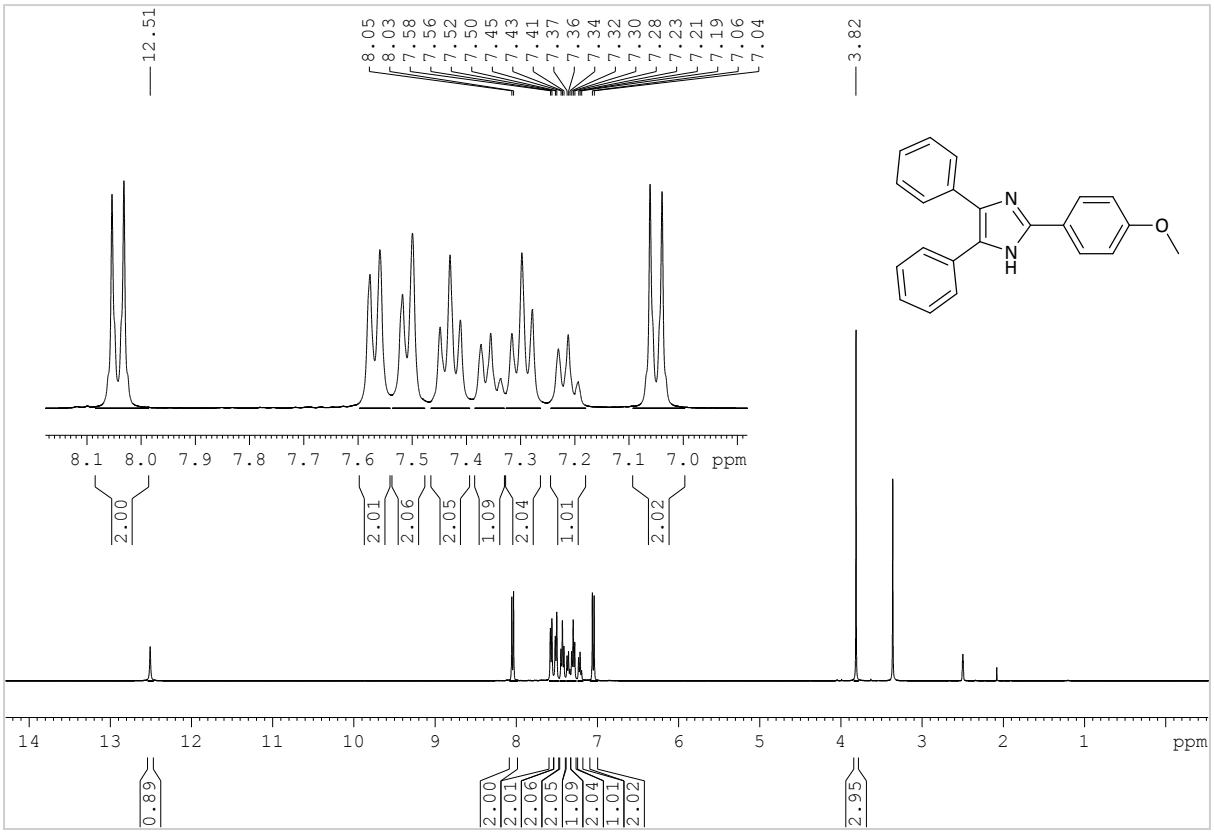
137.12
135.18
131.08
130.35
128.63
128.61
128.43
128.20
128.14
127.72
127.06
126.47
125.18
N
N
H
200
180
160
140
120
100
80
60
40
20
0
ppm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8.03
7.58
7.56
7.52
7.50
7.45
7.43
7.41
7.37
7.36
7.34
7.32
7.30
7.28
7.23
7.21
7.19
7.06
7.04
3.82
N
N
H
O
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 ppm
2.00
2.01
2.06
2.05
1.09
2.04
1.01
2.02
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
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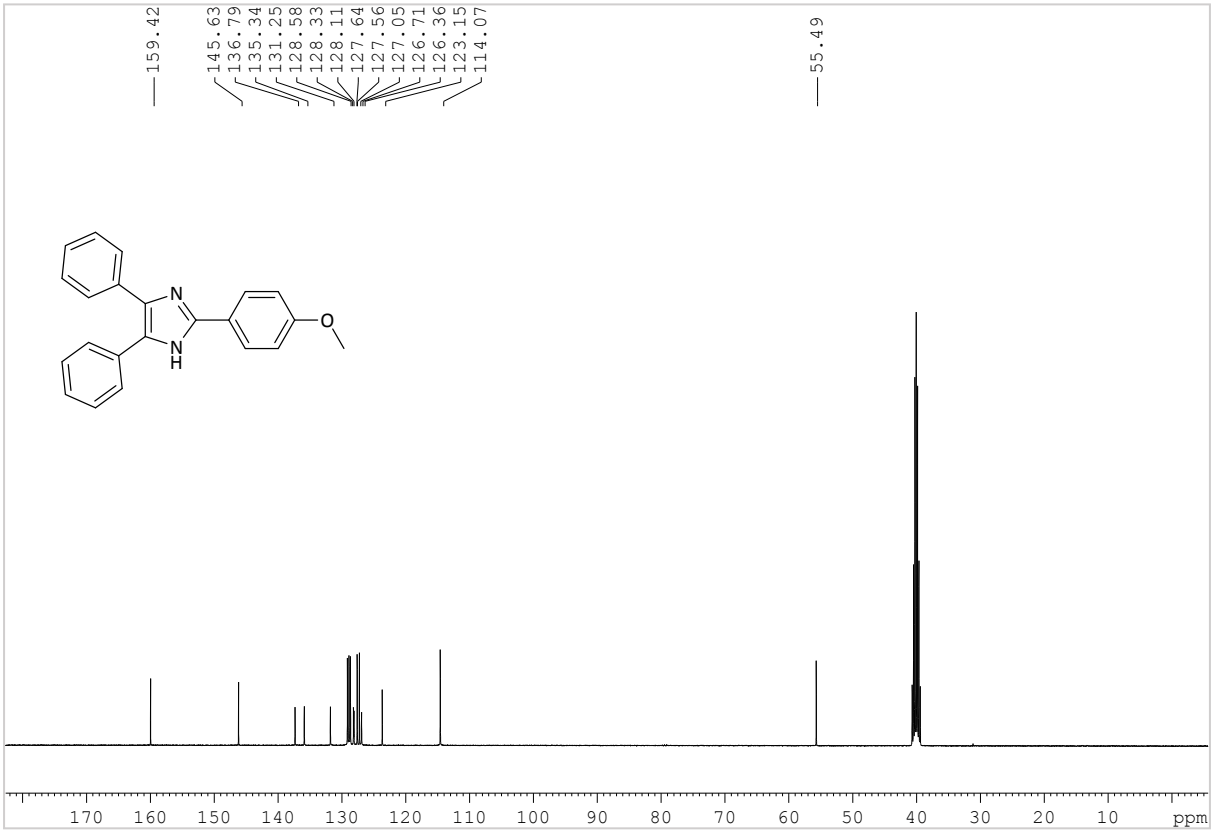
2.00
2.01
2.06
2.05
1.09
2.04
1.01
2.02
2.95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145.63
136.79
135.34
131.25
128.58
128.33
128.11
127.64
127.56
127.05
126.71
126.36
123.15
114.07
55.49
N
N
H
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


12.77
8.13
8.11
7.55
7.53
7.36
N
N
H
Cl
8.2
8.0
7.8
7.6
7.4
7.2
ppm
2.00
6.22
6.46
14
13
12
11
10
9
8
7
6
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4
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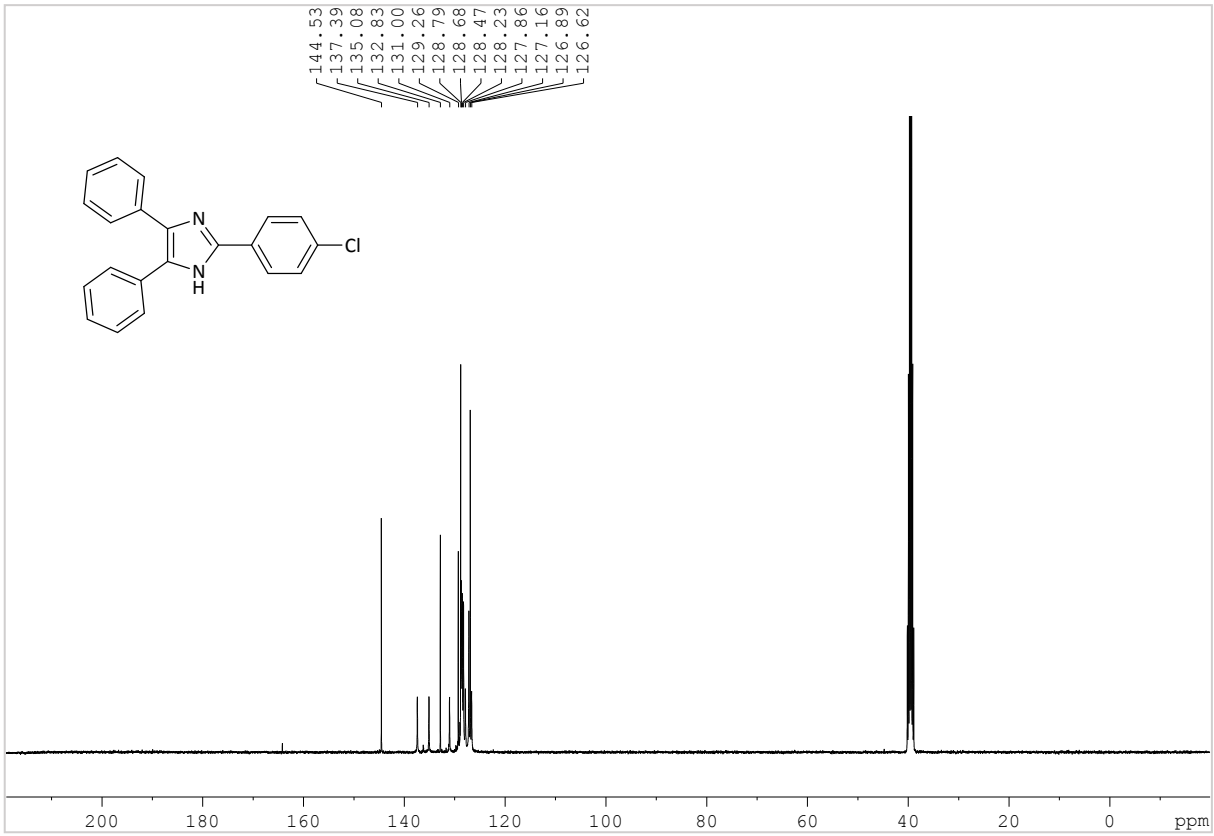
2
1
0
ppm
0.85
2.00
6.22
6.46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128.68
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128.23
127.86
127.16
126.89
126.62
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N
H
Cl
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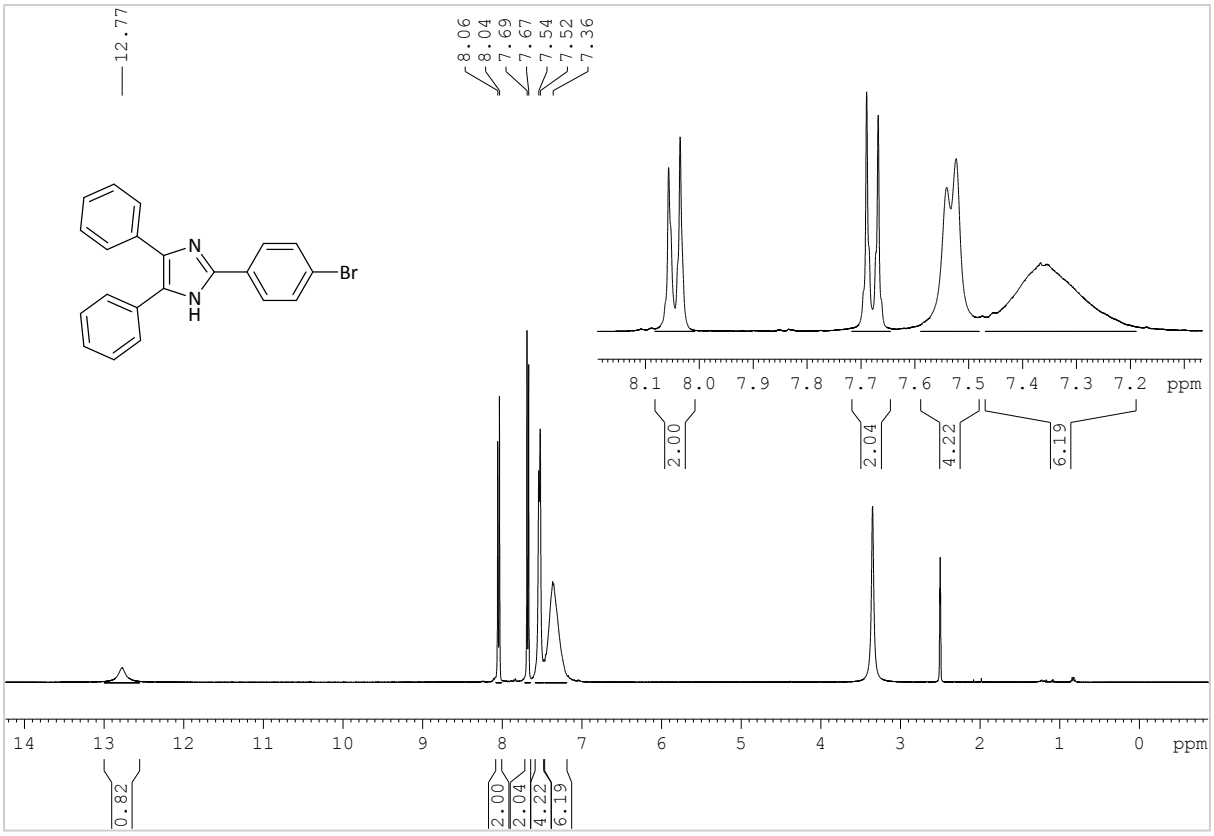
140
120
100
80
60
40
20
0
ppm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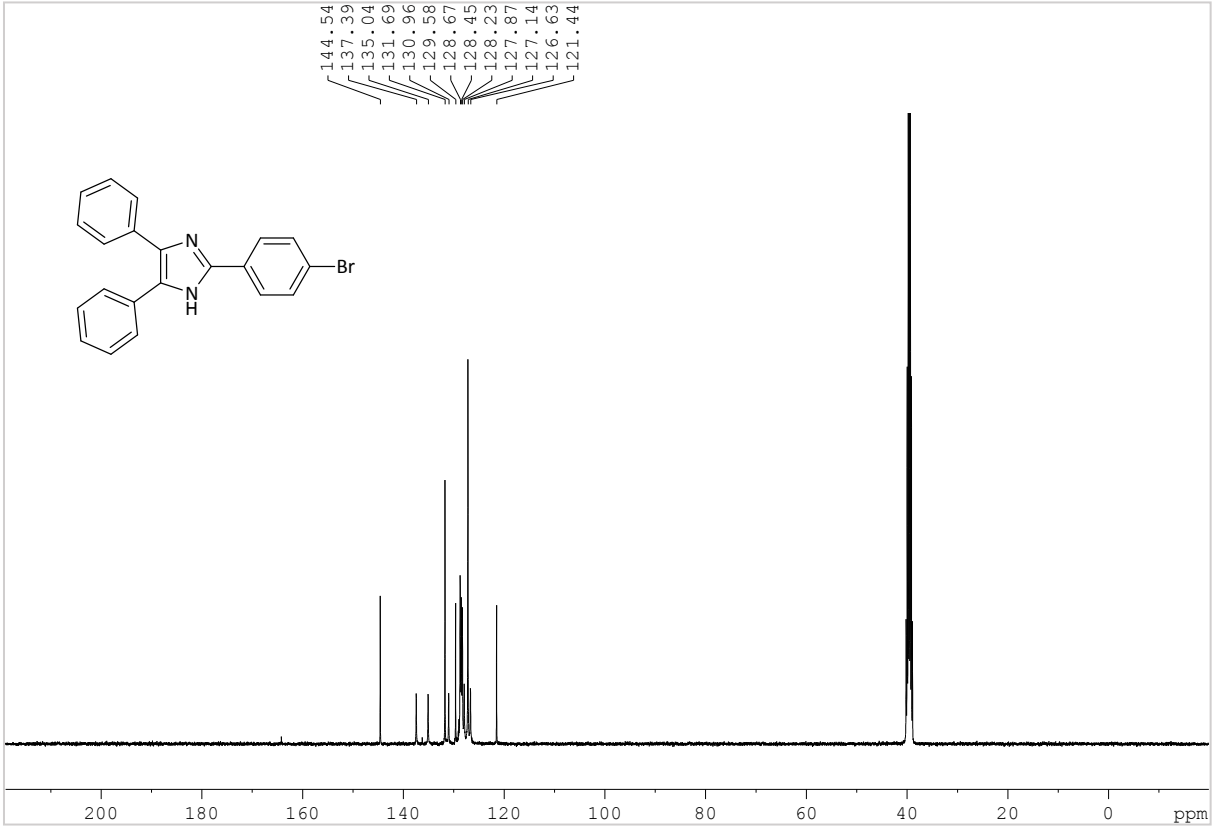
8.04
7.69
7.67
7.54
7.52
7.36
N
N
H
Br
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
2.00
2.04
4.22
6.19
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.82
2.00
2.04
4.22
6.19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130.96
129.58
128.67
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128.23
127.87
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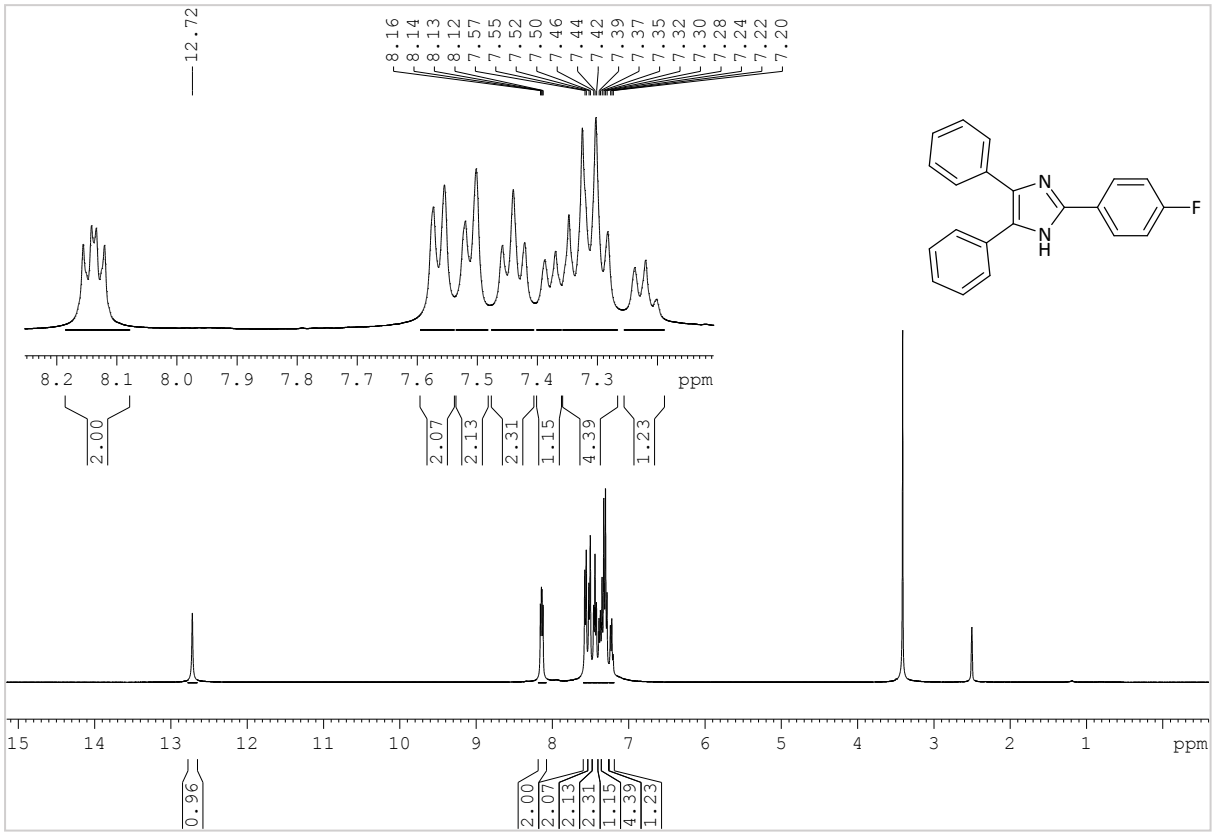
126.63
121.44
N
N
H
Br
200 180 160 140 120 100 80 60 40 20 0 ppm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8.14
8.13
8.12
7.57
7.55
7.52
7.50
7.46
7.44
7.42
7.39
7.37
7.35
7.32
7.30
7.28
7.24
7.22
7.20
N
N
H
F
ppm
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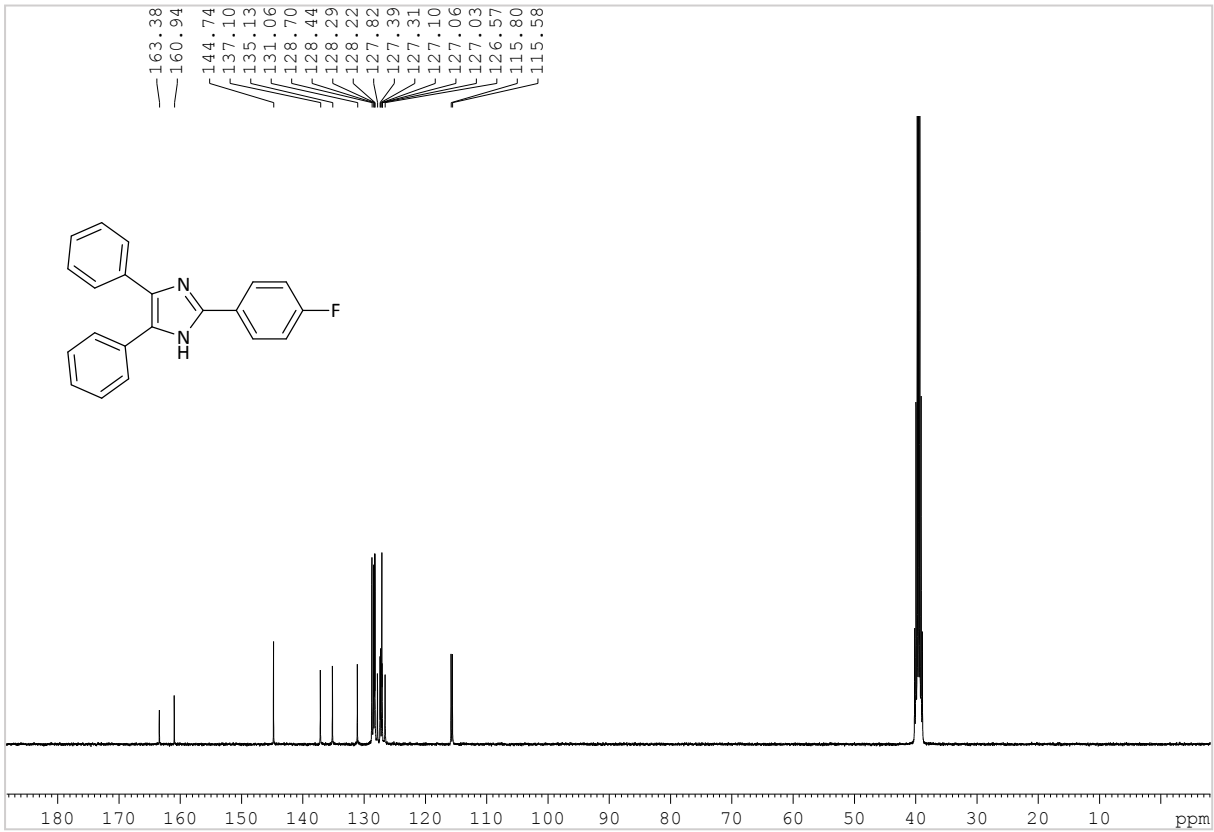
2.07
2.13
2.31
1.15
4.39
1.23
0.96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137.10
135.13
131.06
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128.44
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128.22
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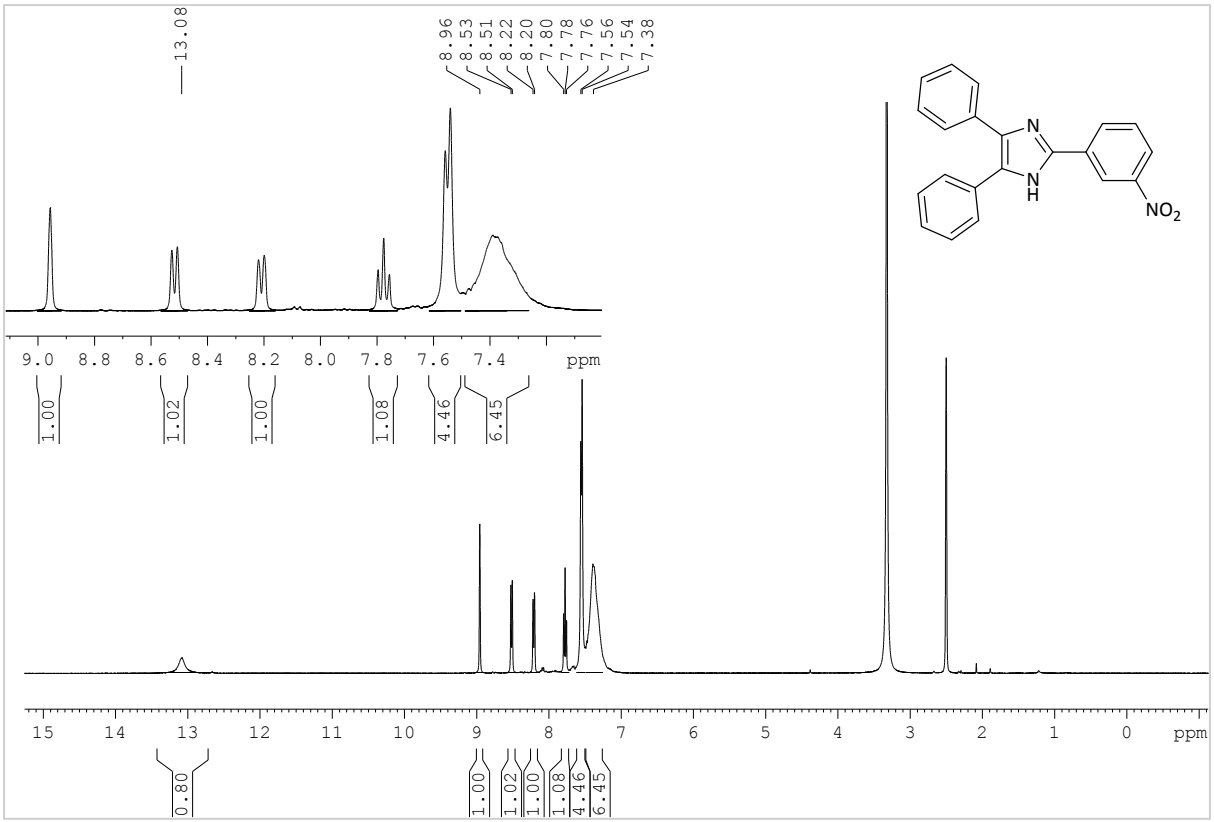
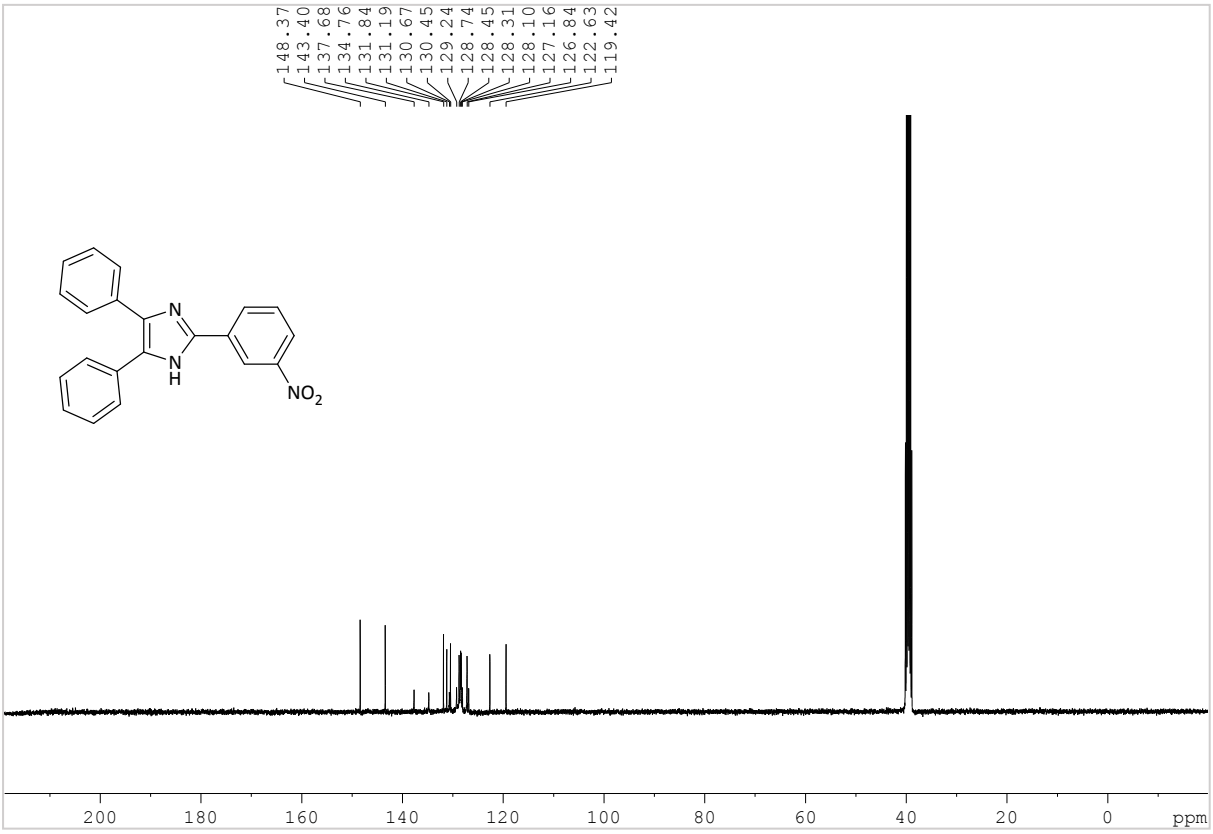
115.80
115.58
N
N
H
F
ppm


13.08
8.96
8.53
8.51
8.22
8.20
7.80
7.78
7.76
7.56
7.54
7.38
N
N
H
NO2
9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 ppm
1.00 1.02 1.00 1.08 4.46 6.45
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.80 1.00 1.02 1.00 1.08 4.46 6.45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N
H
NO2
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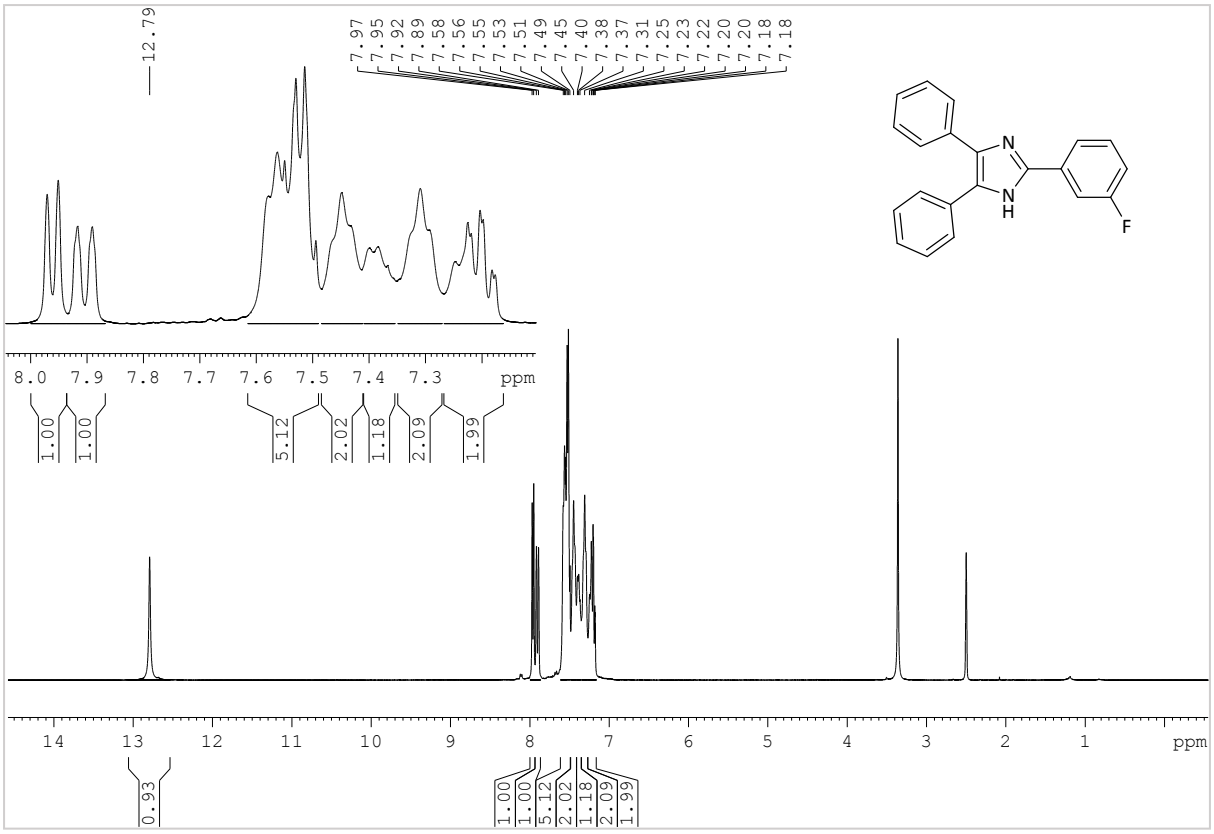
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7.55
7.53
7.51
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7.45
7.40
7.38
7.37
7.31
7.25
7.23
7.22
7.20
7.18
7.18
N
N
H
F
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
1.00 1.00 5.12 2.02 1.18 2.09 1.99
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
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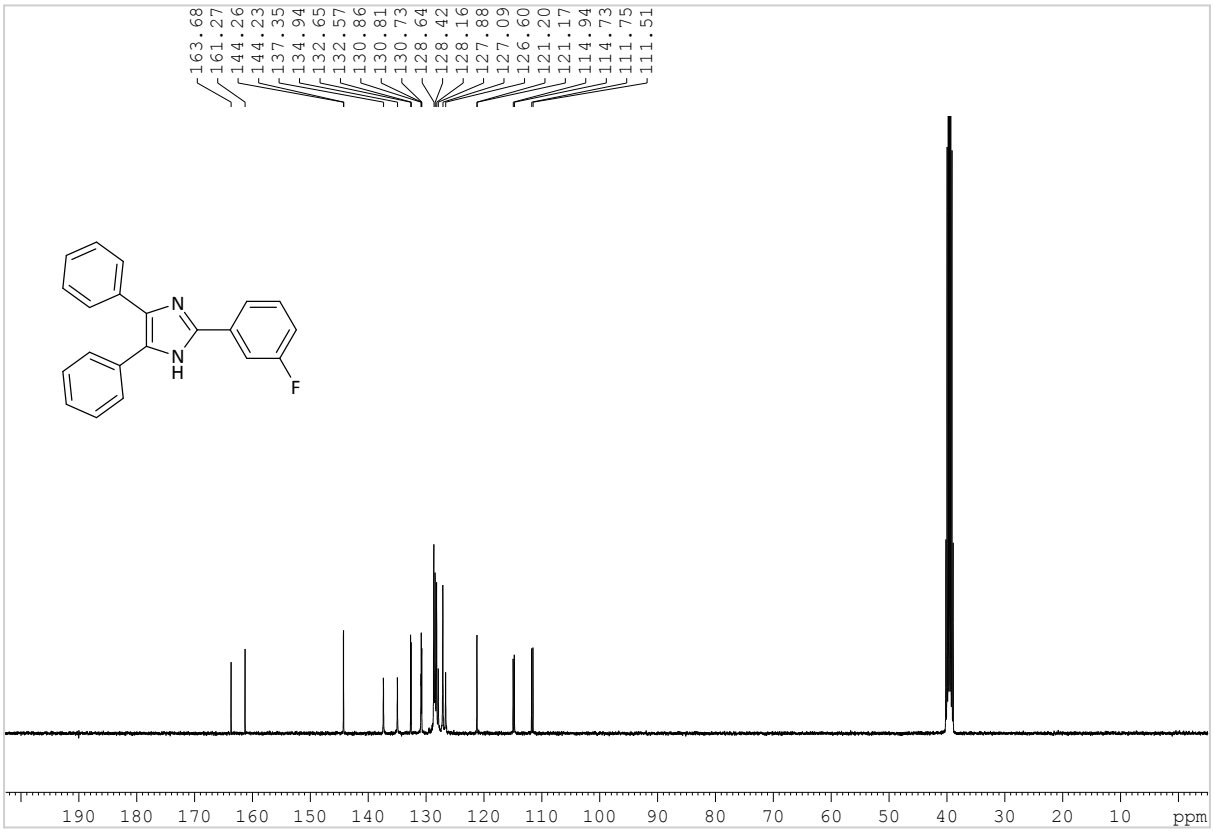
1.00 1.00 5.12 2.02 1.18 2.09 1.99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127.88
127.09
126.60
121.20
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114.73
111.75
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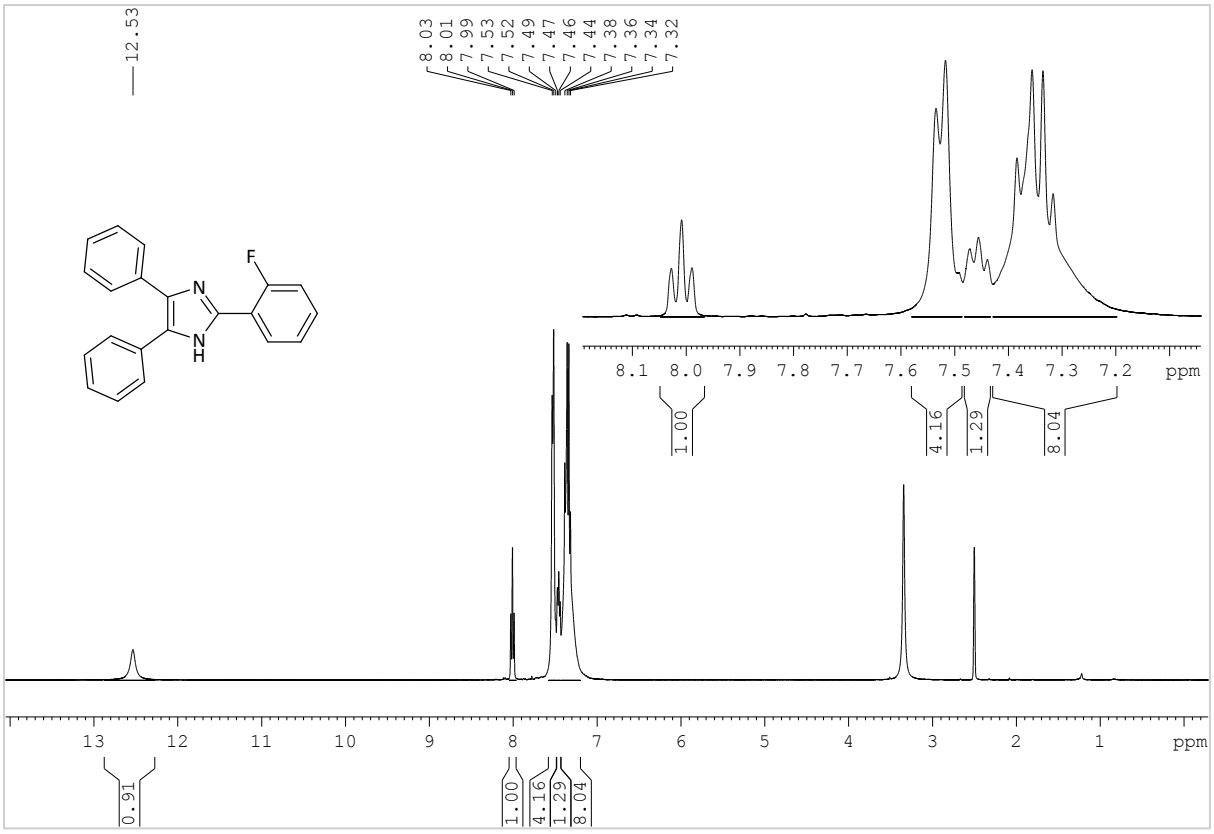
N
N
H
F
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.52
7.49
7.47
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7.44
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7.36
7.34
7.32
F
N
N
H
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
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4.16
1.29
8.04
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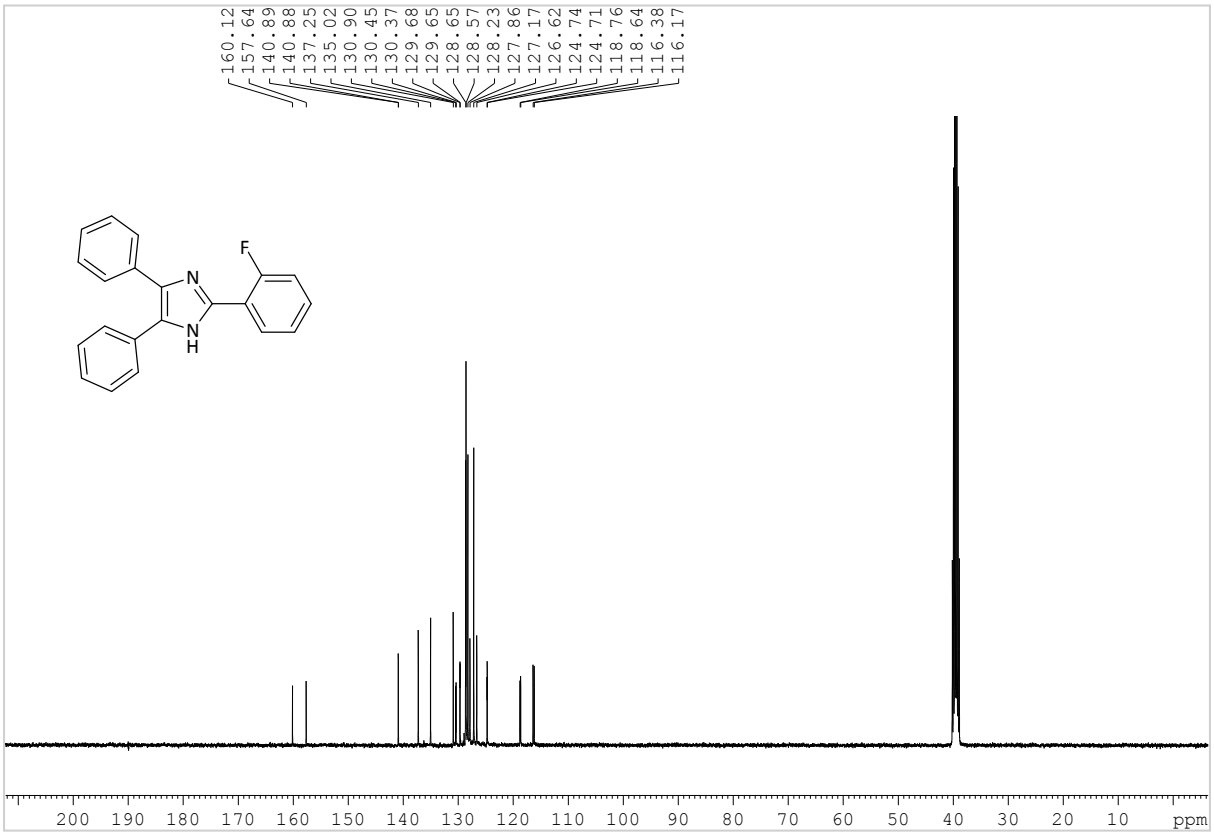
4.16
1.29
8.04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128.23
127.86
127.17
126.62
124.74
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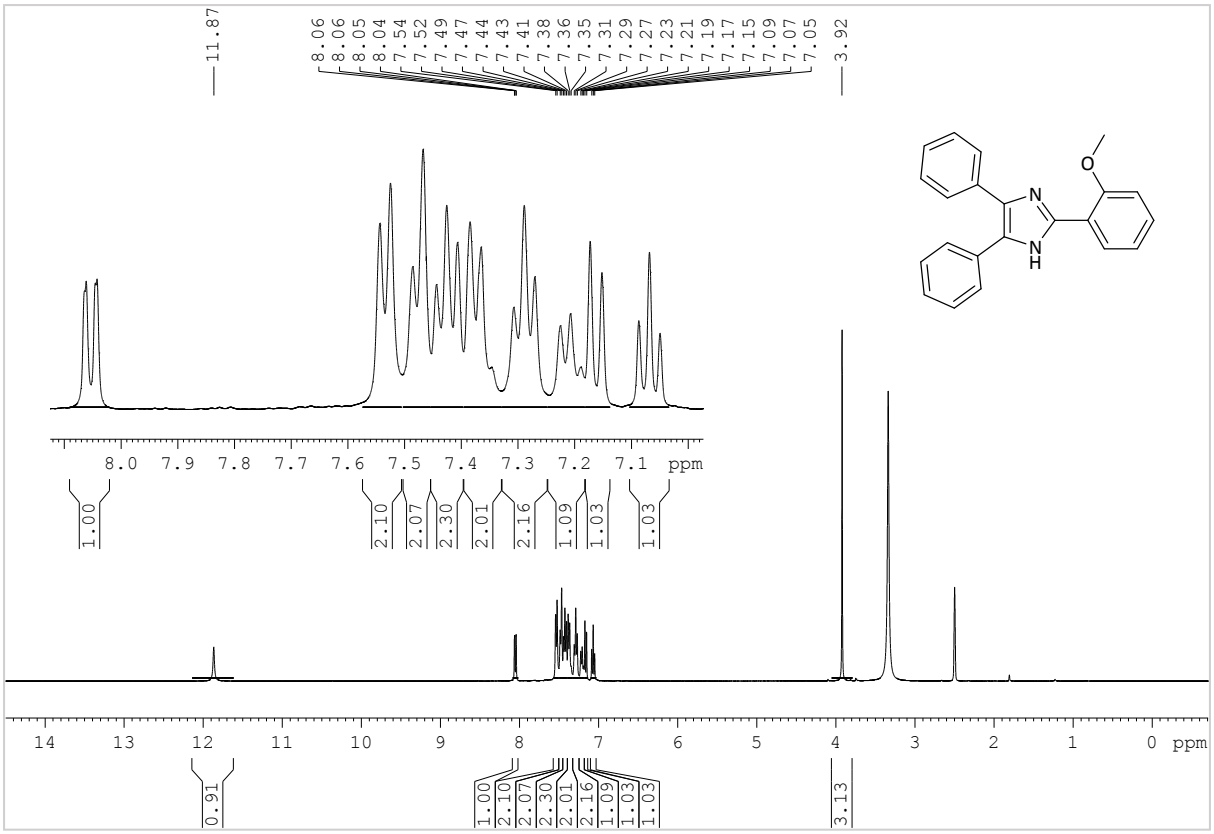
N
N
H
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.52
7.49
7.47
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7.36
7.35
7.31
7.29
7.27
7.23
7.21
7.19
7.17
7.15
7.09
7.07
7.05
3.92
O
N
N
H
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
1.00
2.10
2.07
2.30
2.01
2.16
1.09
1.03
1.03
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
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2.01
2.16
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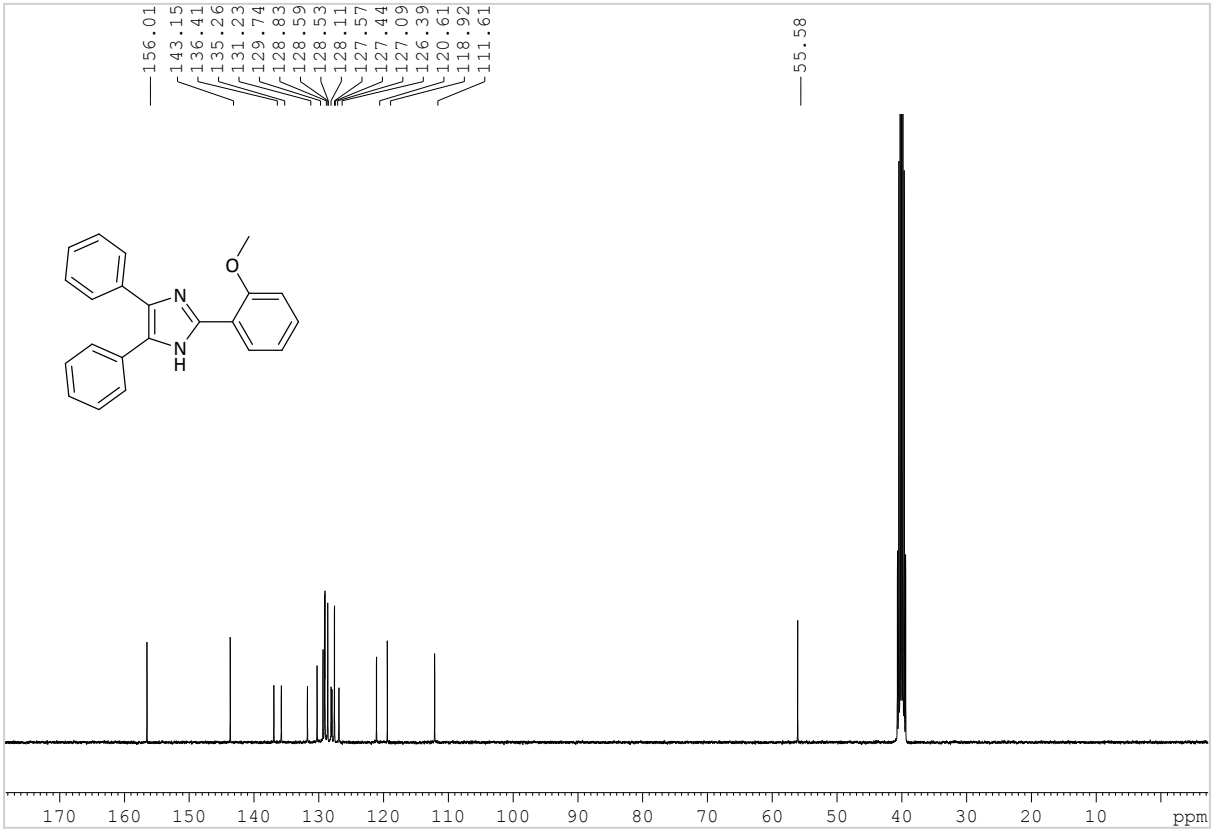
1.03
1.03
3.13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O
N
N
H
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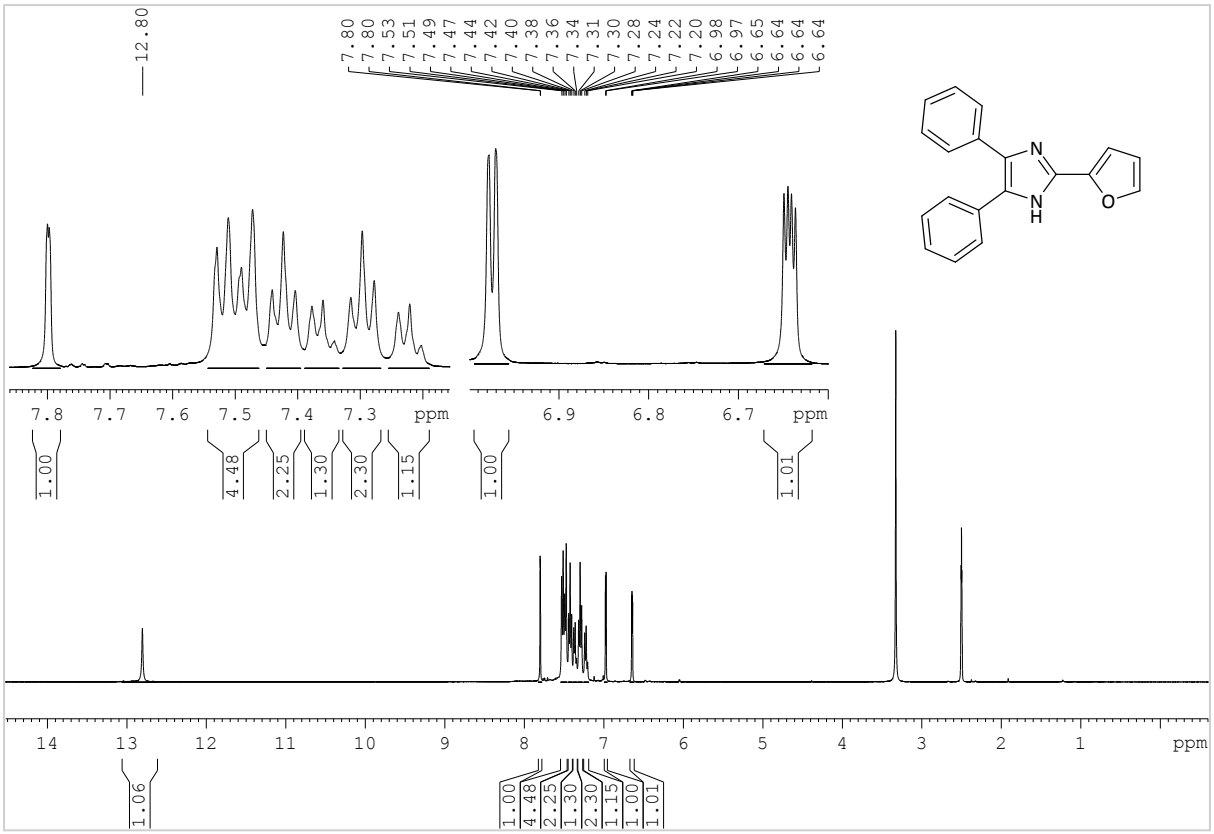
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7.34
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7.24
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6.65
6.64
6.64
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N
N
H
O
7.8
7.7
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1.15
1.00
1.01
14
13
12
11
10
9
8
7
6
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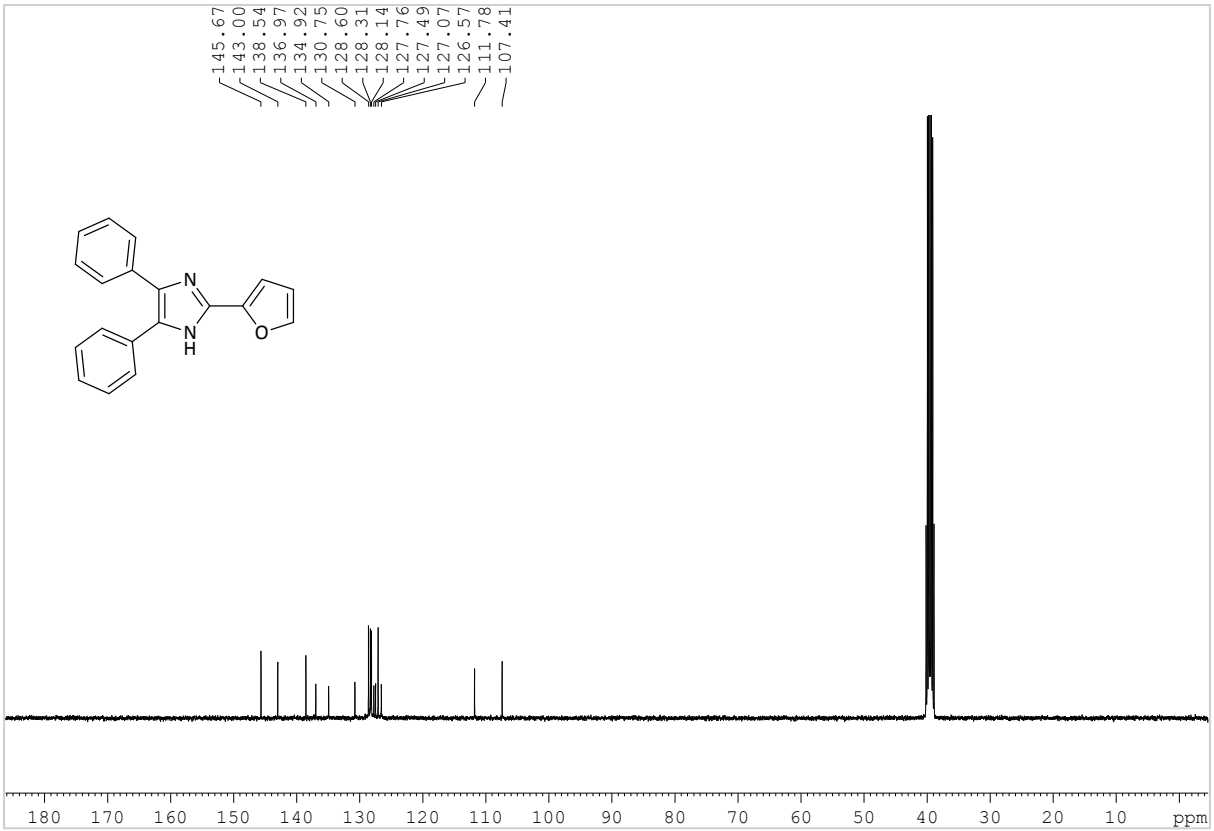
1.01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111.78
107.41
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N
H
O
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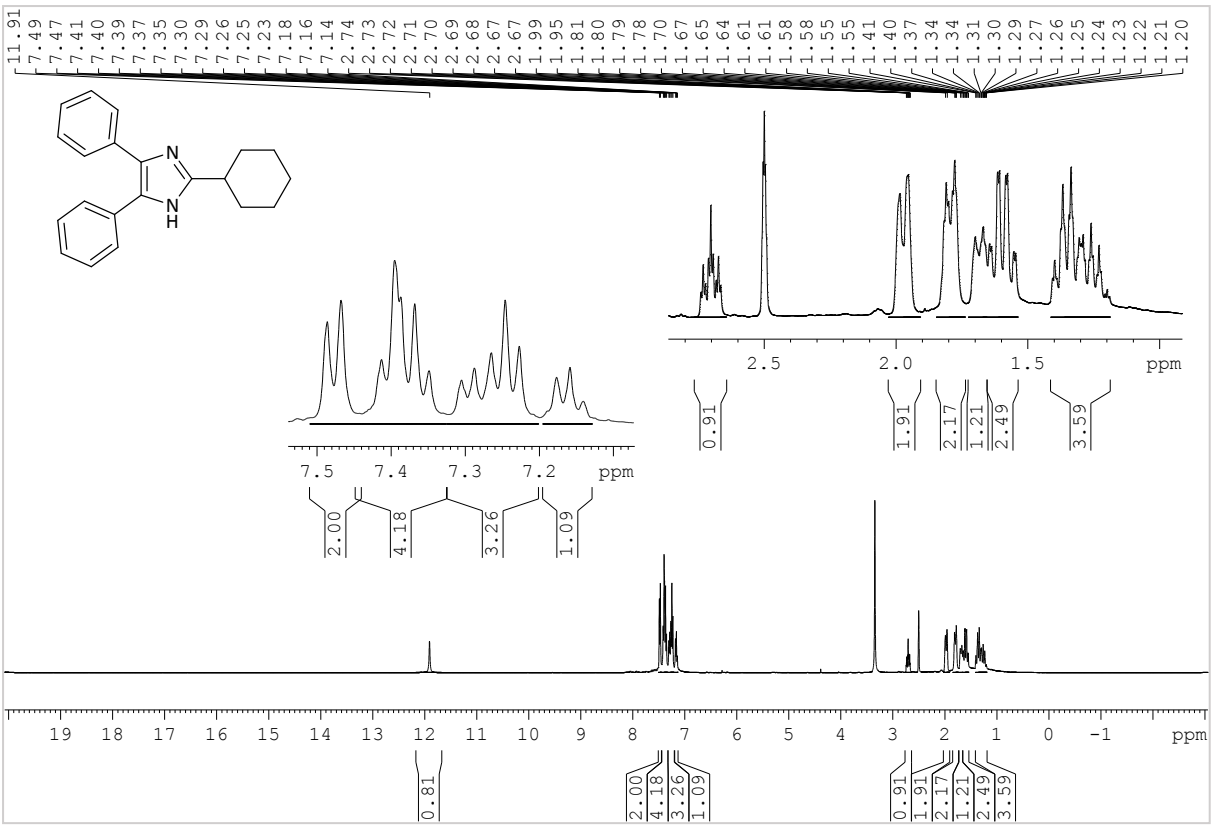
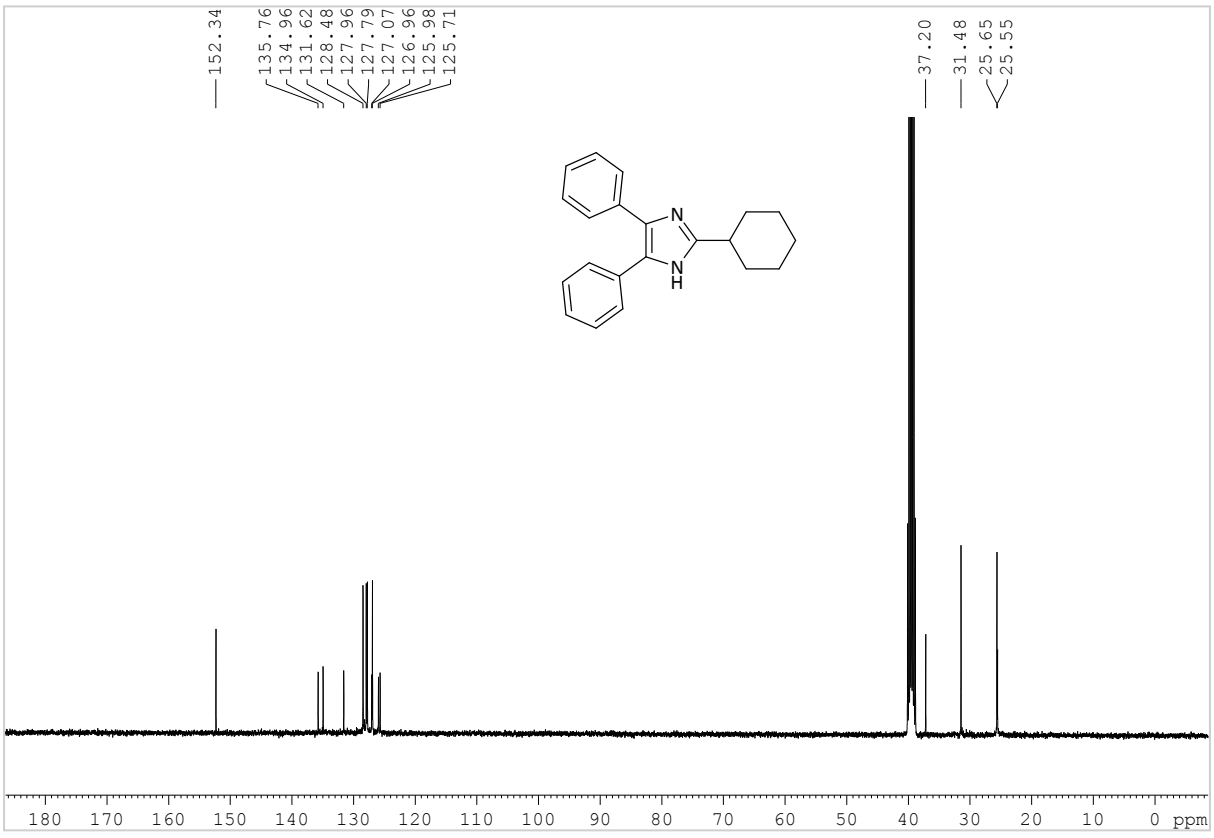
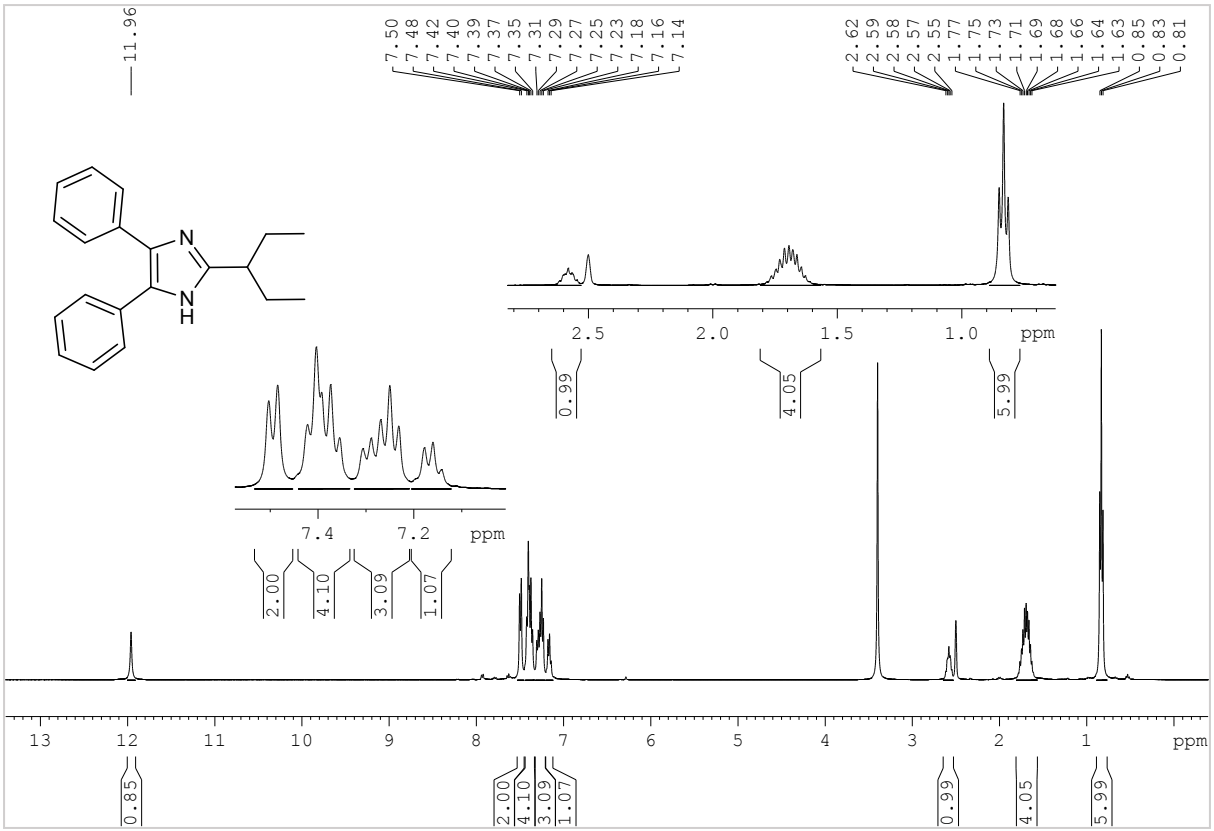
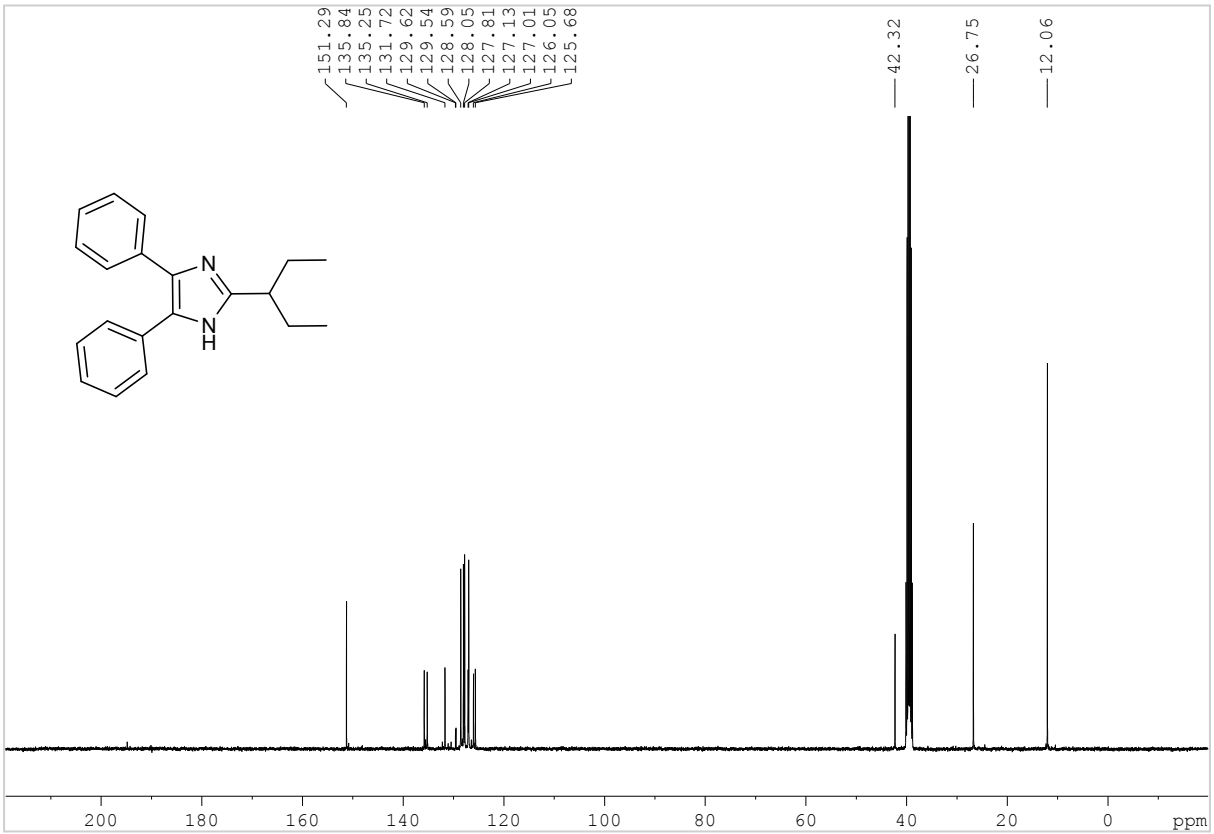
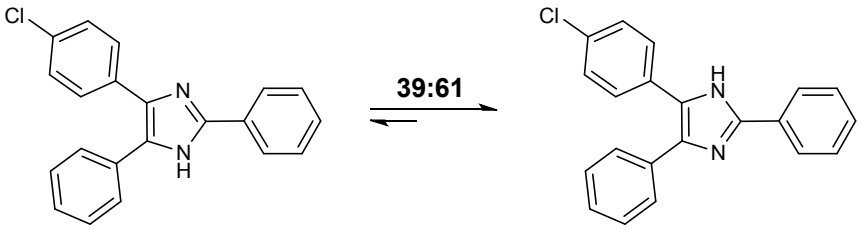
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

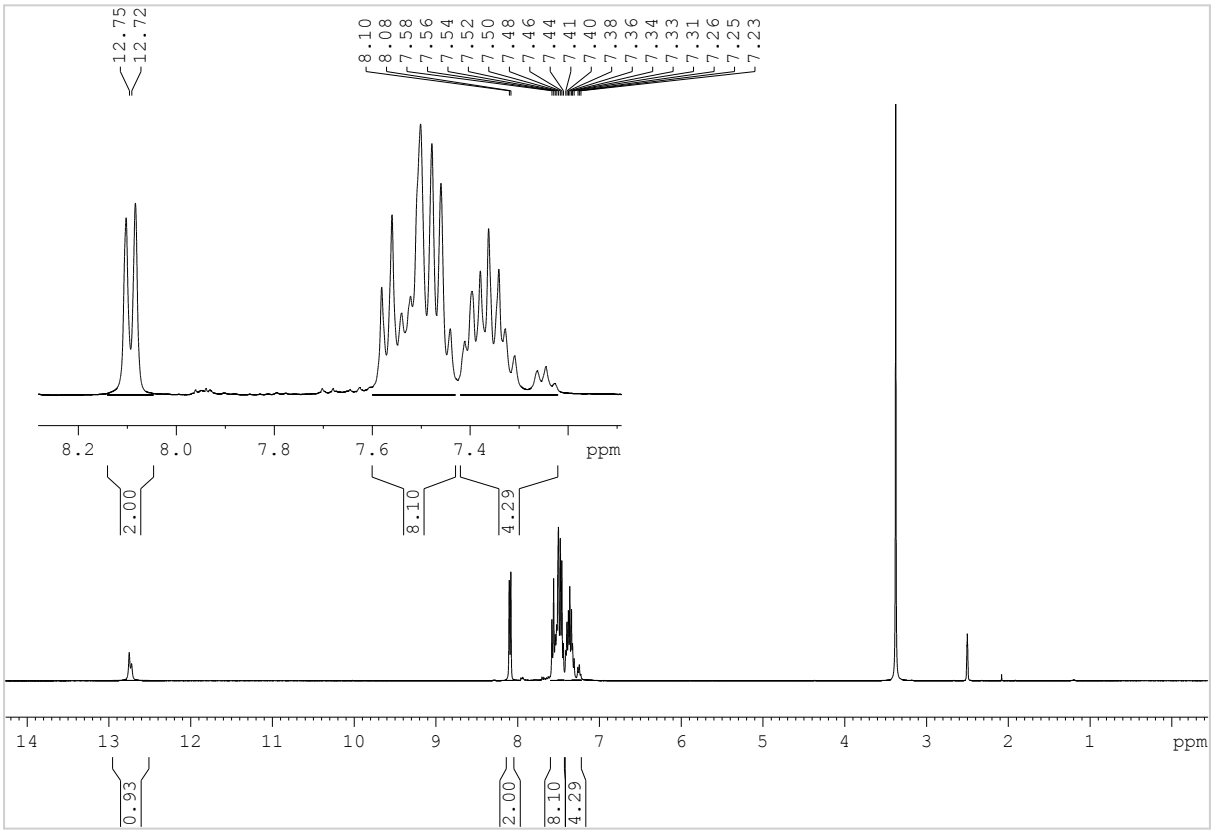


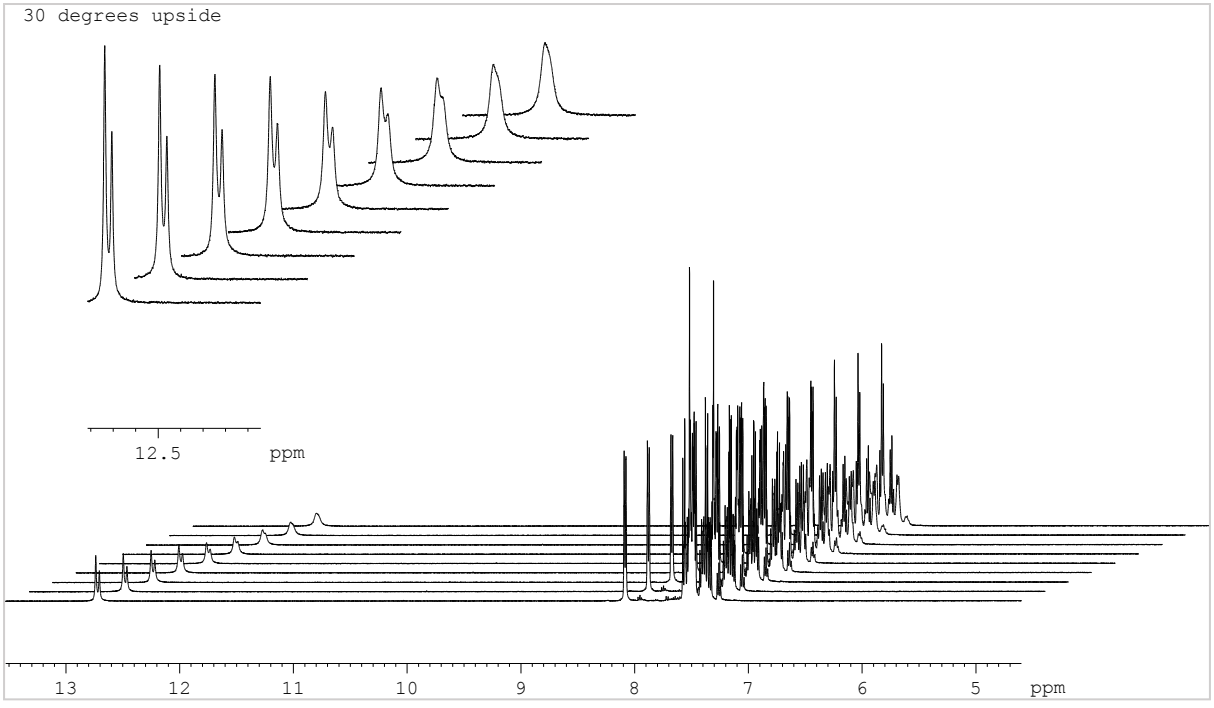


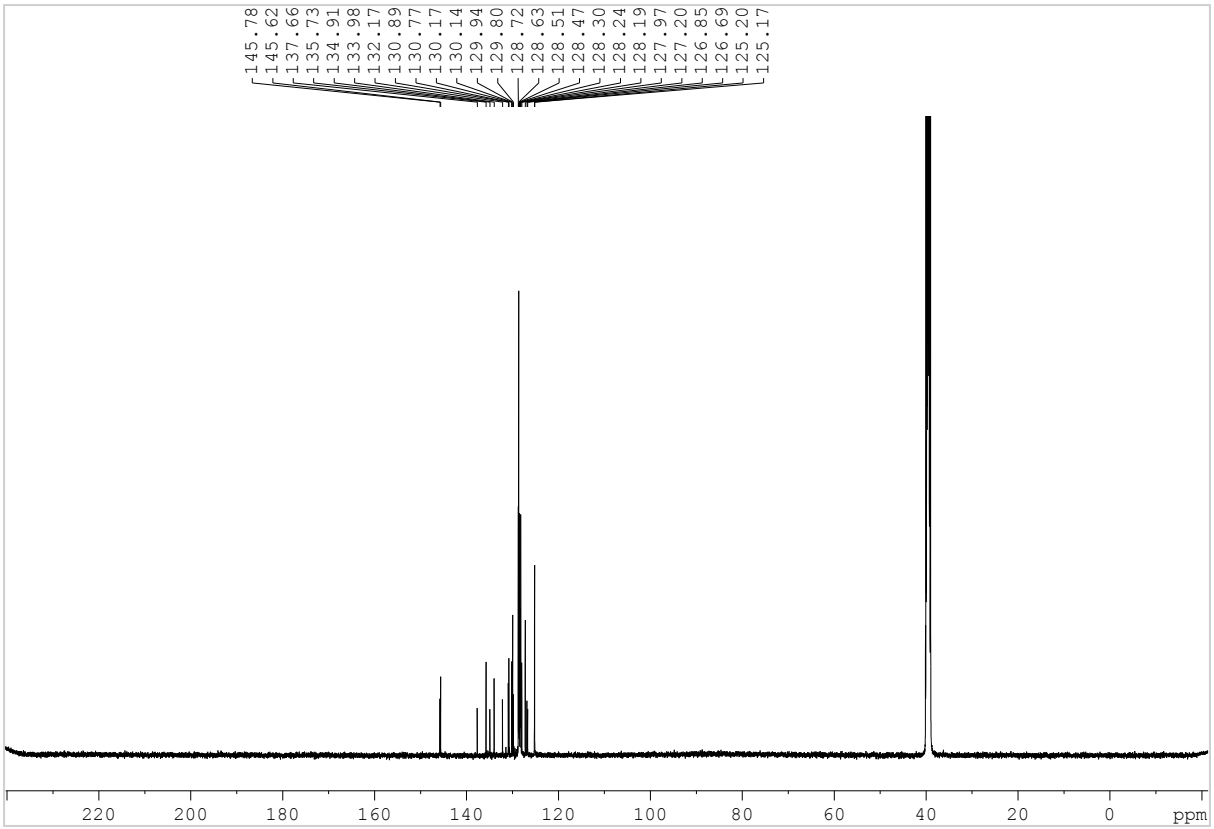

11.96
7.50
7.48
7.42
7.40
7.39
7.37
7.35
7.31
7.29
7.27
7.25
7.23
7.18
7.16
7.14
2.62
2.59
2.58
2.57
2.55
1.77
1.75
1.73
1.71
1.69
1.68
1.66
1.64
1.63
0.85
0.83
0.81
N
N
H
2.5
2.0
1.5
1.0
ppm
0.99
4.05
5.99
7.4
7.2
ppm
2.00
4.10
3.09
1.07
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.85
2.00
4.10
3.09
1.07
0.99
4.05
5.99


151.29
135.84
135.25
131.72
129.62
129.54
128.59
128.05
127.81
127.13
127.01
126.05
125.68
42.32
26.75
12.06
N
N
H
200
180
160
140
120
100
80
60
40
20
0
ppm


Cl
N
N
H
39:61
Cl
H
N
N


12.75
12.72
8.10
8.08
7.58
7.56
7.54
7.52
7.50
7.48
7.46
7.44
7.41
7.40
7.38
7.36
7.34
7.33
7.31
7.26
7.25
7.23
8.2
8.0
7.8
7.6
7.4
ppm
2.00
8.10
4.29
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.93
2.00
8.10
4.29


30 degrees upside
12.5
ppm
13
12
11
10
9
8
7
6
5
ppm


145.78
145.62
137.66
135.73
134.91
133.98
132.17
130.89
130.77
130.17
130.14
129.94
129.80
128.72
128.63
128.51
128.47
128.30
128.24
128.19
127.97
127.20
126.85
126.69
125.20
125.17
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Elemental Composition Report

Single Mass Analysis Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

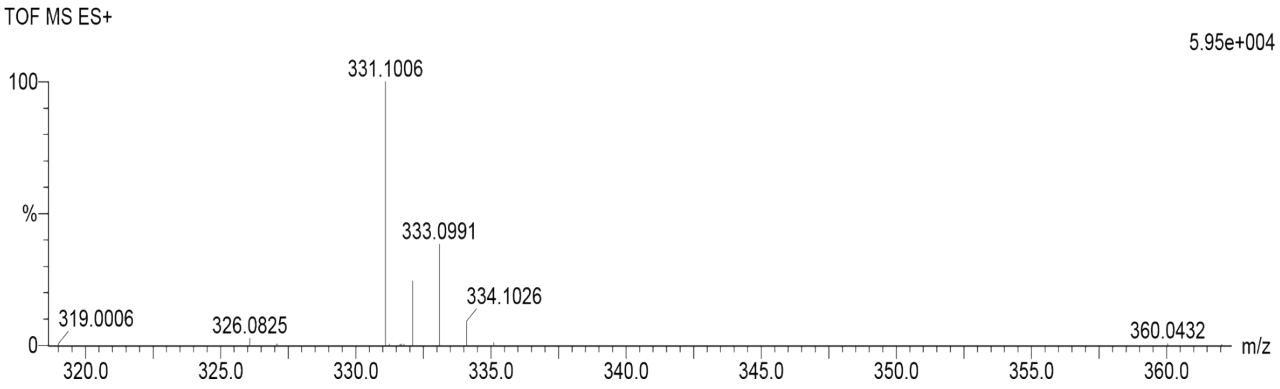
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions

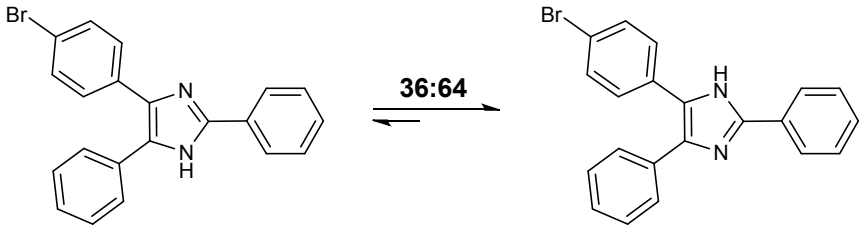
153 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass) Elements Used:

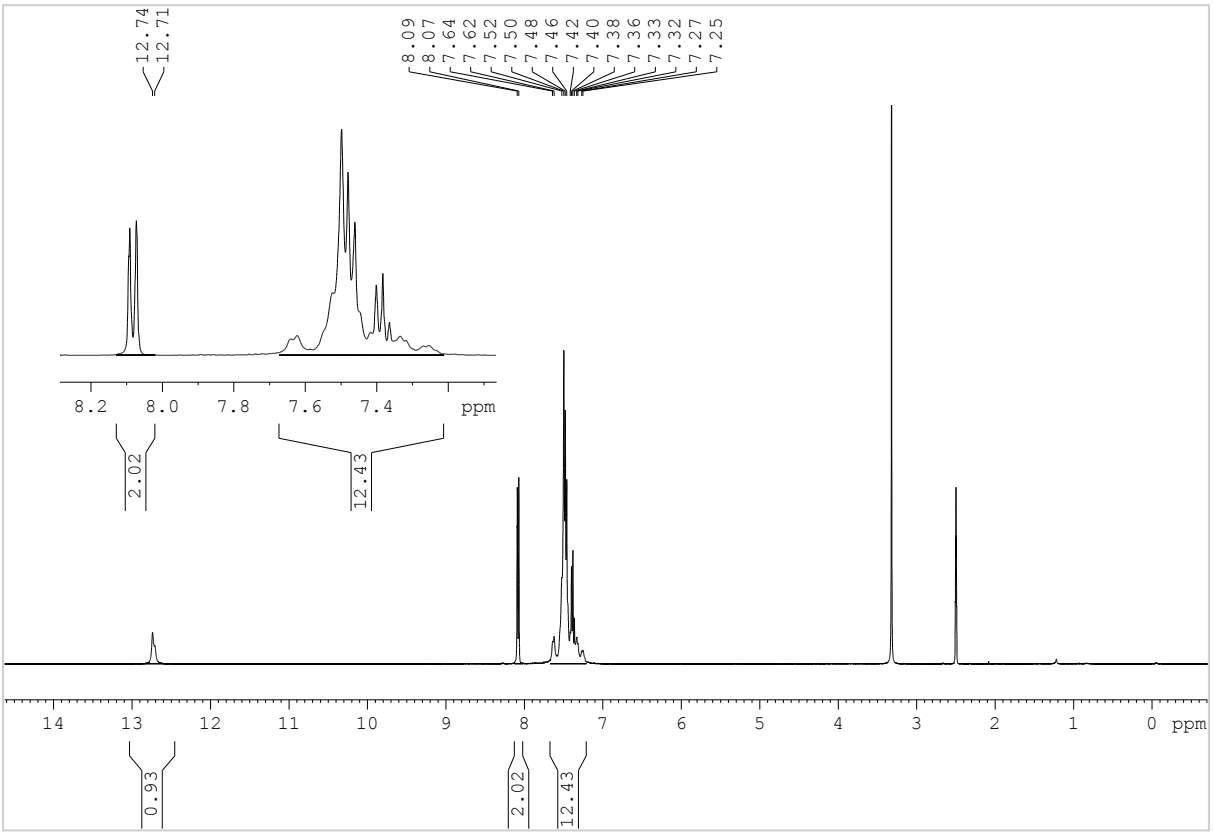
C: 0-50 H: 0-50 N: 0-20 Cl: 0-1

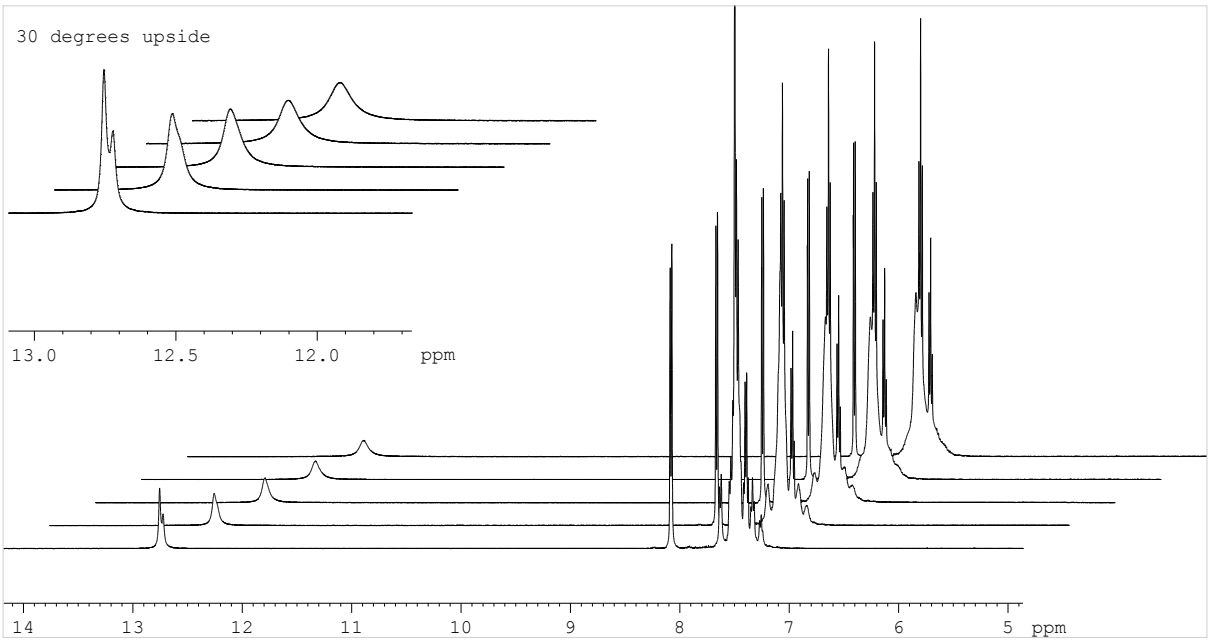
Chloro-H Imidazole 59 (1.957) Cm (1:61)

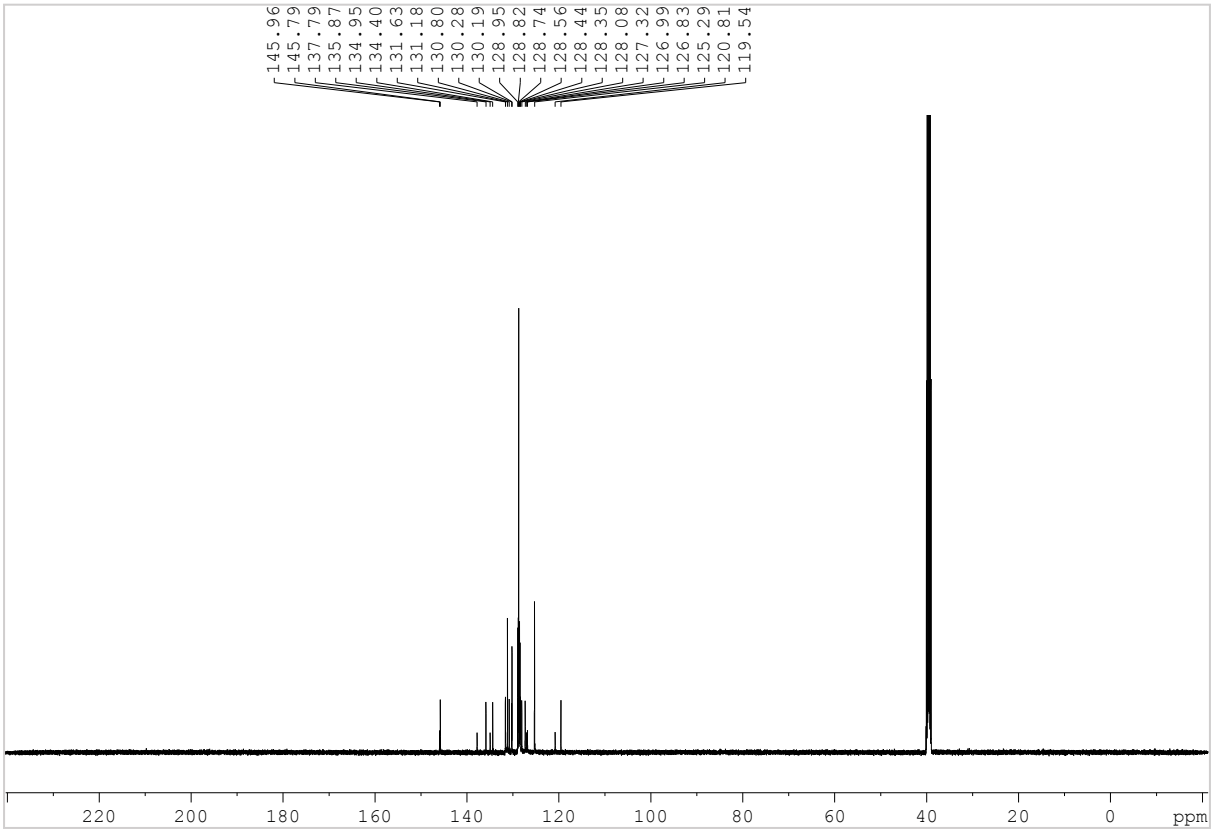


Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
331.1006	331.1002	0.4	1.2	14.5	50.3	0.0	C21 H16 N2 Cl


Br
N
N
H
36:64
Br
H
N
N


12.74
12.71
8.09
8.07
7.64
7.62
7.52
7.50
7.48
7.46
7.42
7.40
7.38
7.36
7.33
7.32
7.27
7.25
8.2
8.0
7.8
7.6
7.4
ppm
2.02
12.43
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.93
2.02
12.43


30 degrees upside
13.0
12.5
12.0
ppm
14
13
12
11
10
9
8
7
6
5 ppm


145.96
145.79
137.79
135.87
134.95
134.40
131.63
131.18
130.80
130.28
130.19
128.95
128.82
128.74
128.56
128.44
128.35
128.08
127.32
126.99
126.83
125.29
120.81
119.54
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Elemental Composition Report
Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 2

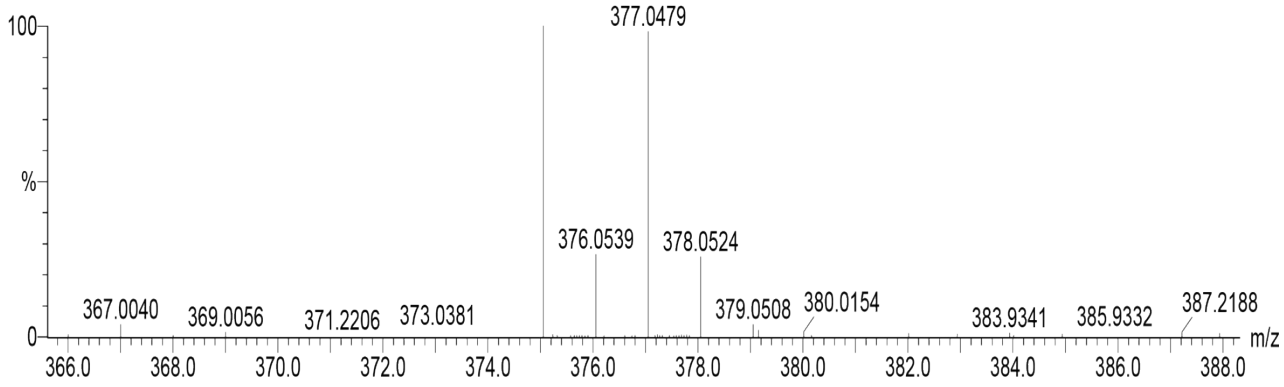
Monoisotopic Mass, Even Electron Ions

7 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass) Elements Used:

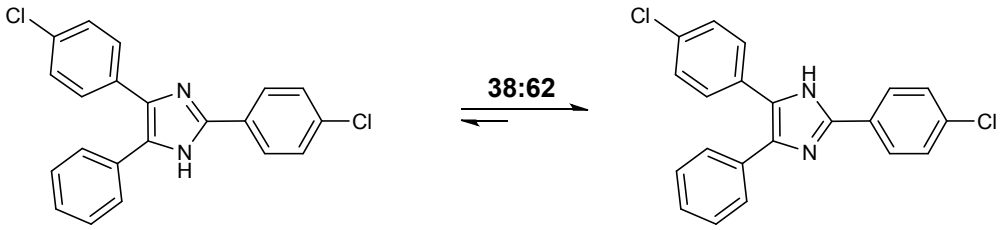
C: 20-25 H: 15-20 N: 0-5 Br: 0-1

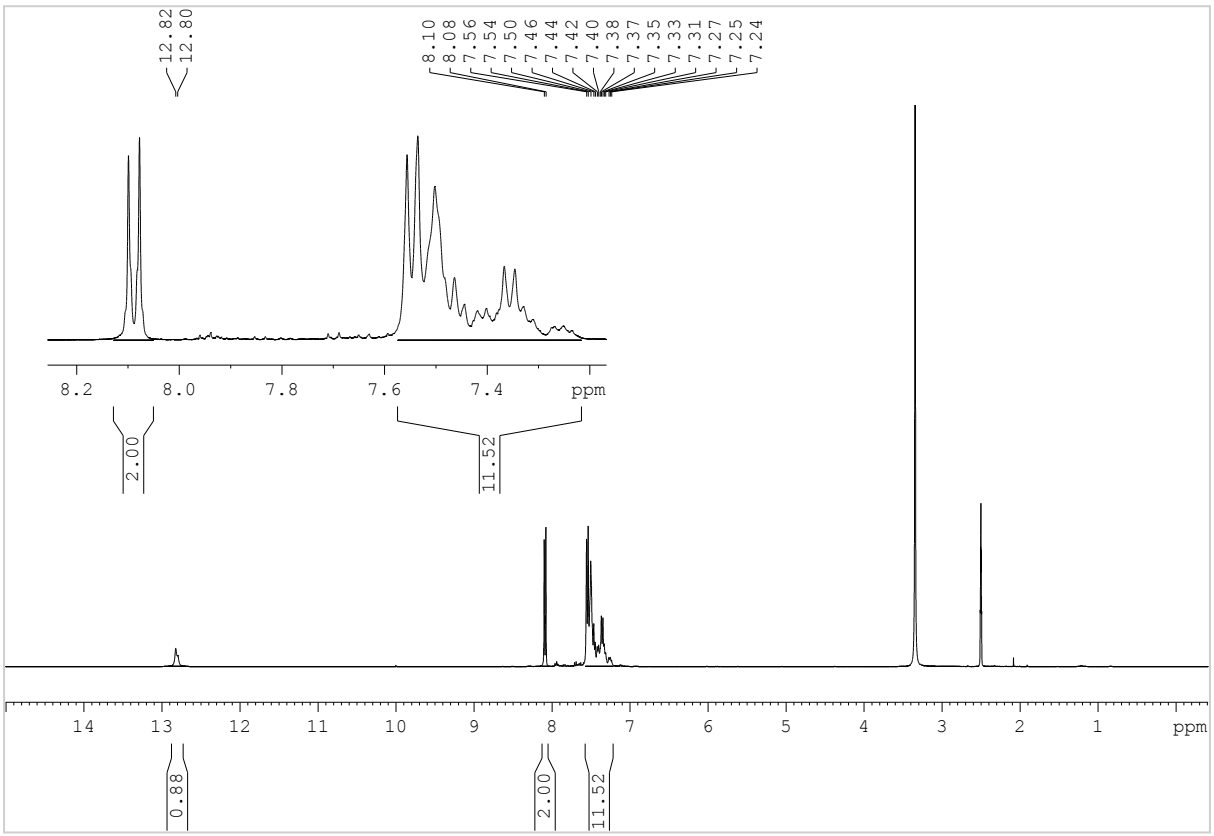
4-Bromo-H-Imid 11 (0.338) Cm (1:61)

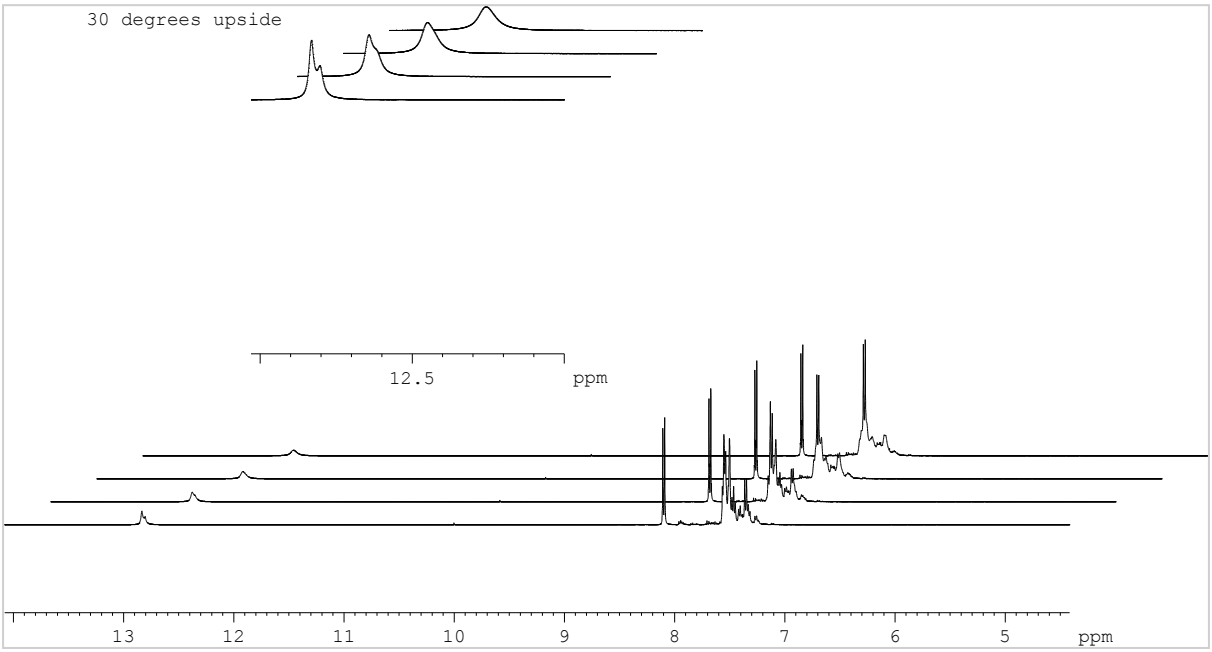
TOF MS ES+ 1.55e+005 375.0492

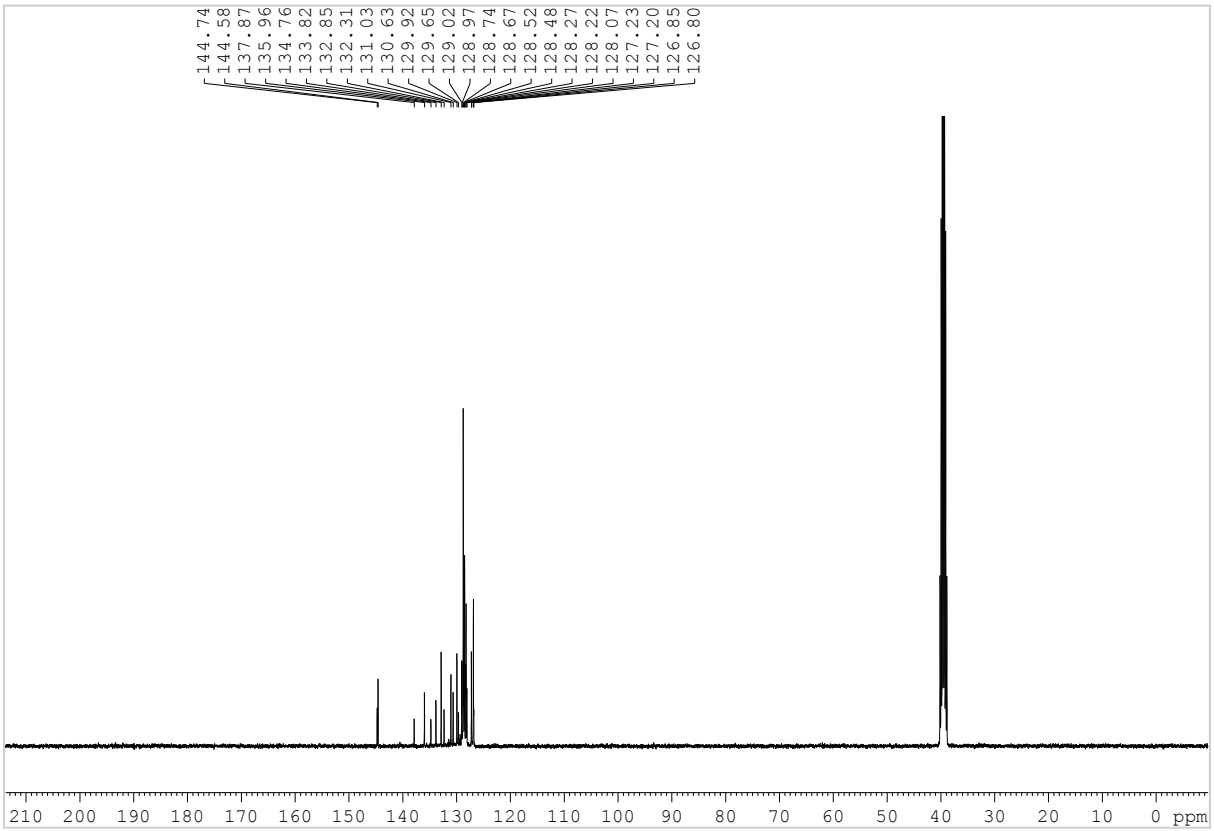


Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT(Norm)	Formula
375.0492	375.0497	-0.5	-1.3	14.5	145.5	0.0	C21 H16 N2 Br


Cl
N
N
H
Cl
38:62
Cl
H
N
N
Cl


12.82
12.80
8.10
8.08
7.56
7.54
7.50
7.46
7.44
7.42
7.40
7.38
7.37
7.35
7.33
7.31
7.27
7.25
7.24
8.2
8.0
7.8
7.6
7.4
ppm
2.00
11.52
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.88
2.00
11.52


30 degrees upside
12.5
ppm
13
12
11
10
9
8
7
6
5
ppm

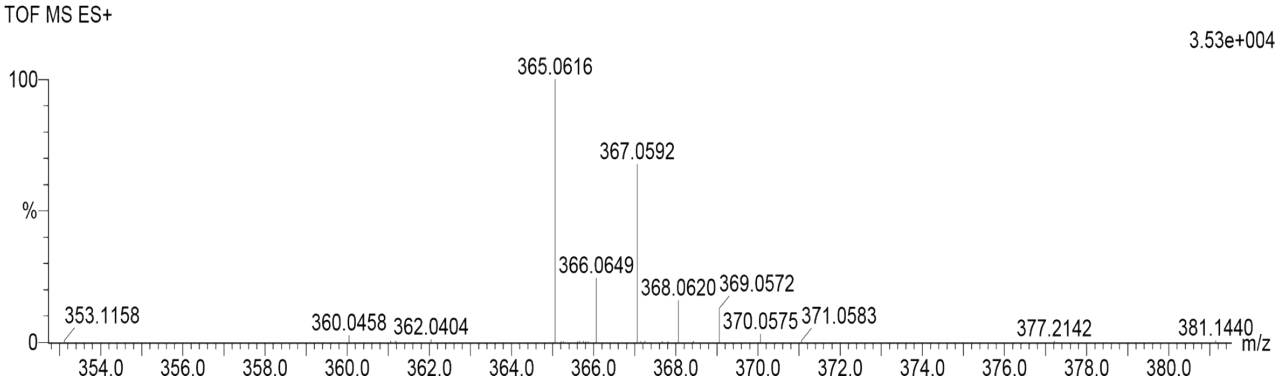

144.74
144.58
137.87
135.96
134.76
133.82
132.85
132.31
131.03
130.63
129.92
129.65
129.02
128.97
128.74
128.67
128.52
128.48
128.27
128.22
128.07
127.23
127.20
126.85
126.80
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Elemental Composition Report
Page 1

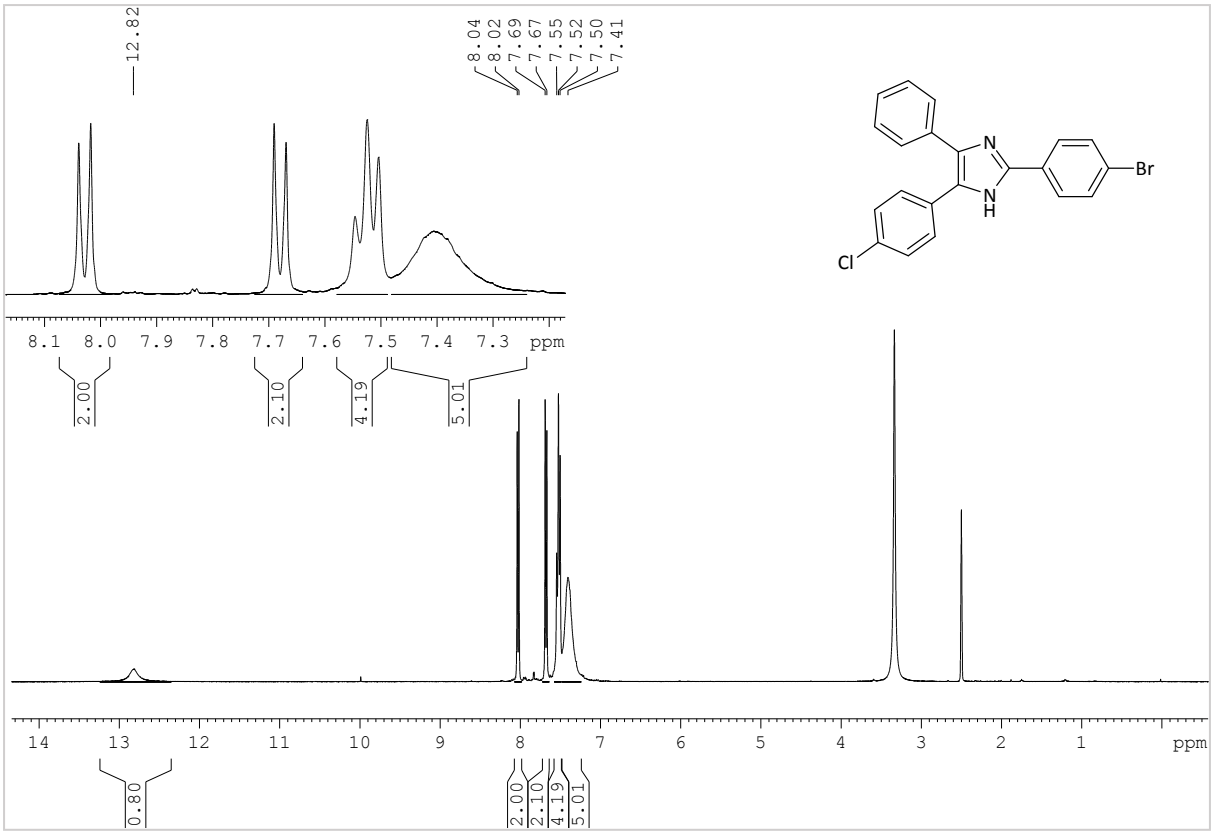
Single Mass Analysis

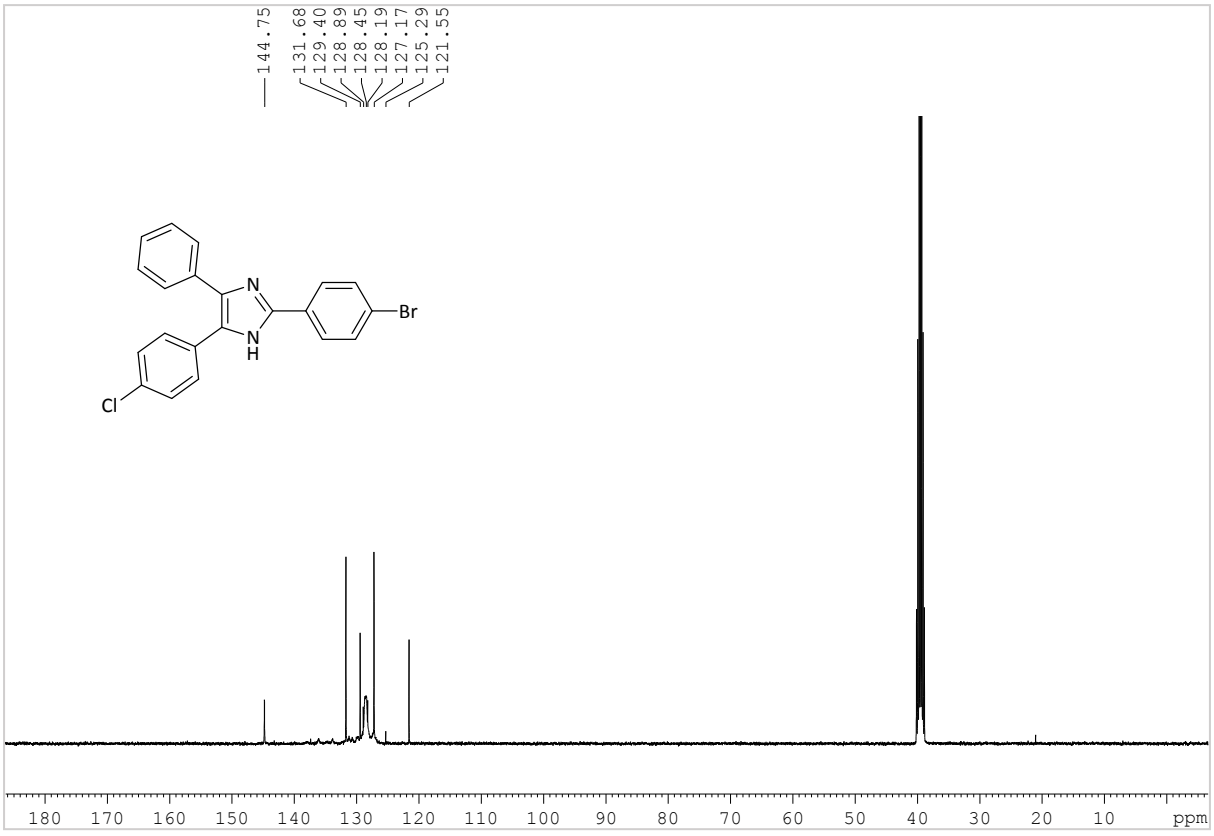
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
238 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass) Elements Used:
C: 0-50 H: 0-50 N: 0-20 Cl: 0-2
Chloro-chloro Imidazole 6 (0.169) Cm (1:61)



Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT(Norm)	Formula
365.0616	365.0612	0.4	1.1	14.5	74.2	0.0	C21 H15 N2 Cl2


12.82
8.04
8.02
7.69
7.67
7.55
7.52
7.50
7.41
N
N
H
Br
Cl
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
2.00
2.10
4.19
5.01
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.80
2.00
2.10
4.19
5.01


144.75
131.68
129.40
128.89
128.45
128.19
127.17
125.29
121.55
N
N
H
Br
Cl
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Elemental Composition Report
Page 1

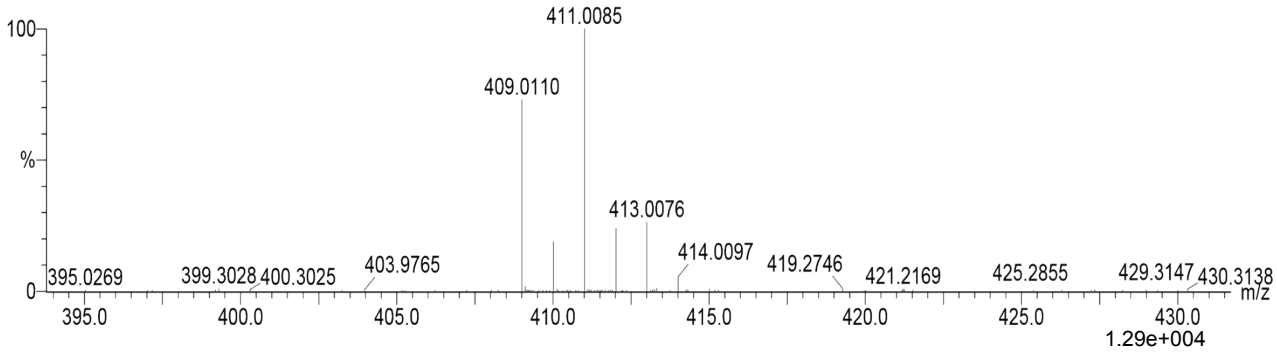
Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

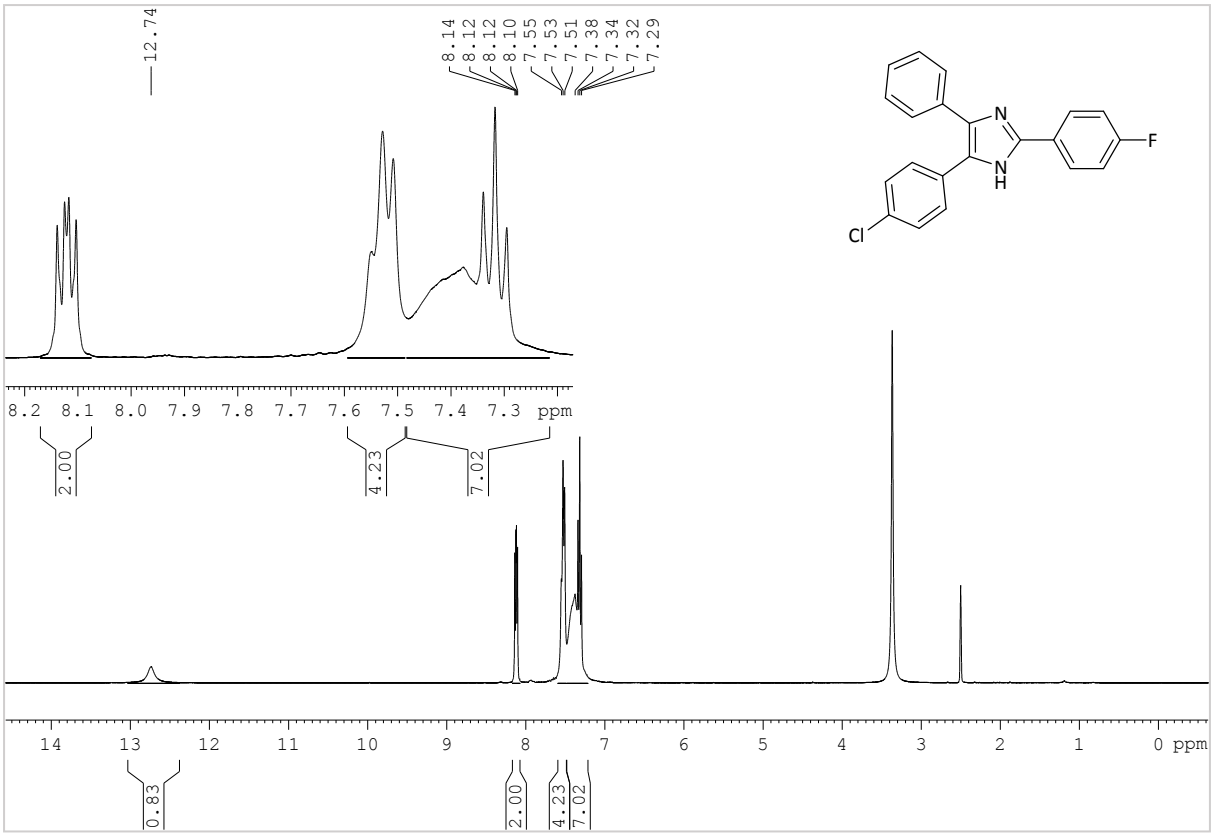
Monoisotopic Mass, Even Electron Ions
330 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass) Elements Used:
C: 0-50 H: 0-50 N: 0-20 Cl: 0-1 Br: 0-1

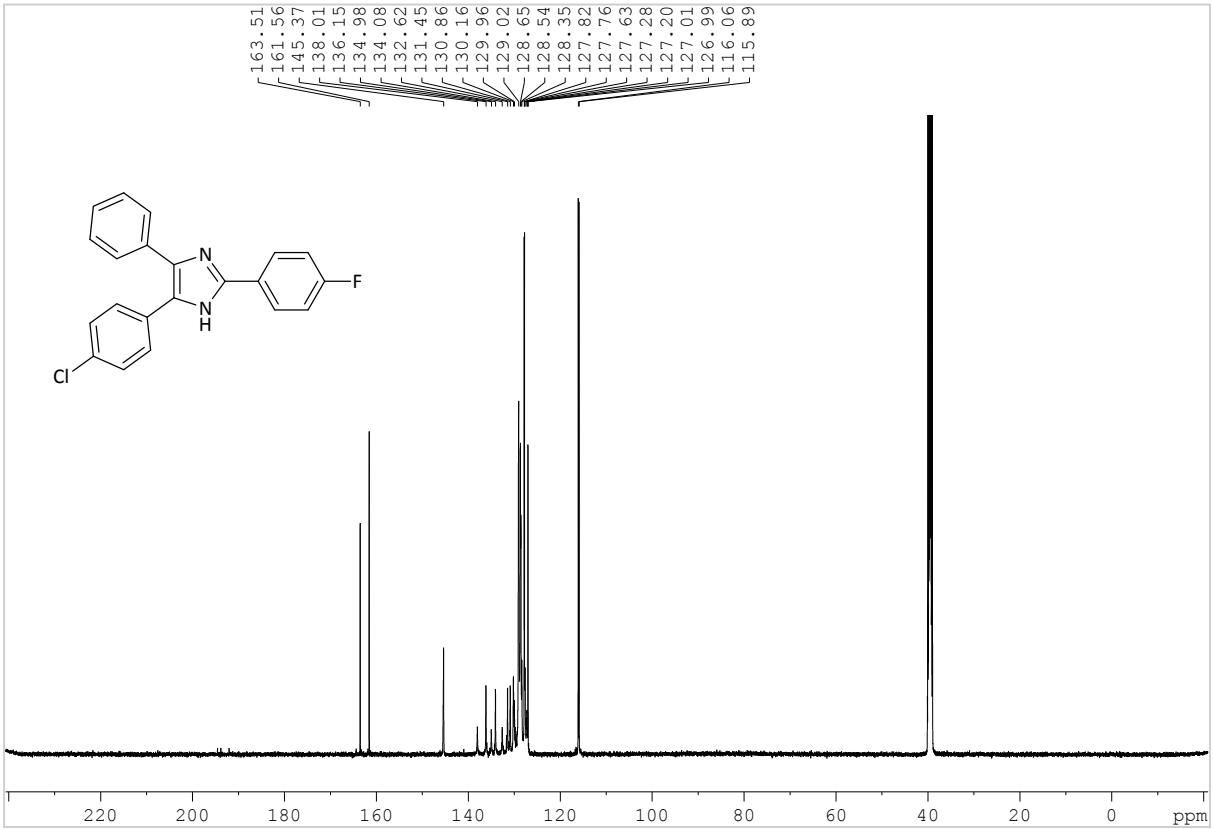
Chloro-bromo Imidazole 7 (0.202) Cm (1:61)

TOF MS ES+



Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT(Norm)	Formula
409.0110	409.0107	0.3	0.7	14.5	129.1	0	C21 H15 N2 Cl Br


12.74
8.14
8.12
8.12
8.10
7.55
7.53
7.51
7.38
7.34
7.32
7.29
N
N
H
F
Cl
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
2.00
4.23
7.02
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.83
2.00
4.23
7.02


163.51
161.56
145.37
138.01
136.15
134.98
134.08
132.62
131.45
130.86
130.16
129.96
129.02
128.65
128.54
128.35
127.82
127.76
127.63
127.28
127.20
127.01
126.99
116.06
115.89
N
N
H
F
Cl
220 200 180 160 140 120 100 80 60 40 20 0 ppm

Elemental Composition Report
Page 1

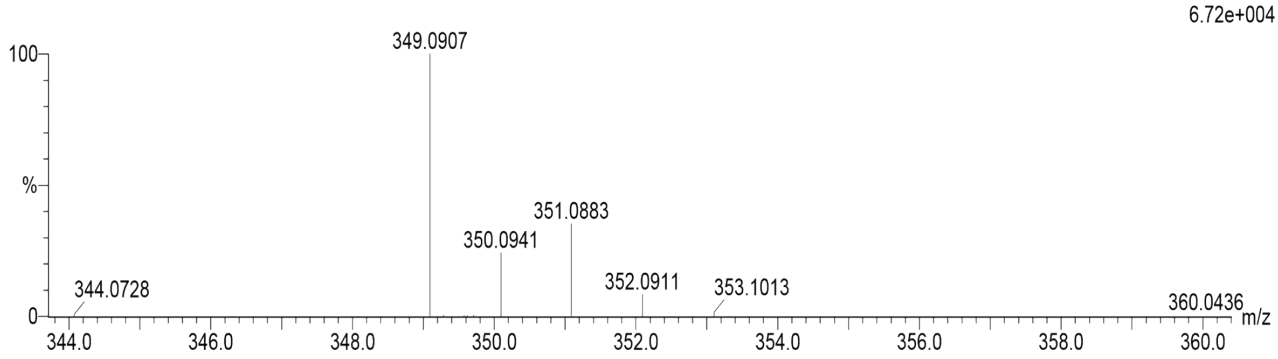
Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
249 formula(e) evaluated with 1 results within limits (up to 20 best isotopic matches for each mass) Elements Used:
C: 10-50 H: 0-50 N: 0-20 Cl: 0-1 F: 0-1

Chloro-fluoro Imidazole 3 (0.068) Cm (1:61)

TOF MS ES+



Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT(Norm)	Formula
349.0907	349.0908	-0.1	-0.3	14.5	50.9	0.0	C21 H15 N2 Cl F