Selenium dioxide mediated benzylic sp^3 C–H oxidation in acetic acid: synthesis of lophine derivatives from α-methylene ketones via a domino multicomponent reaction

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Electronic Supplementary Information

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General information

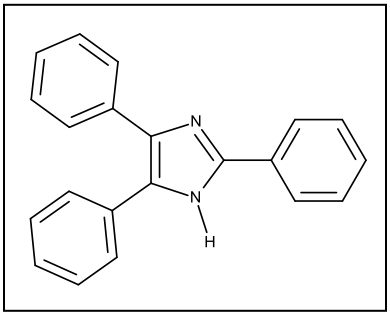
All ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker Avance 400 operating at either 400 or 100 MHz using DMSO-d_6 as an internal standard. Chemical shifts are expressed in parts per million (ppm) relative to the residual solvent and coupling constants (*J* values) were recorded in Hz. High-resolution electron-spray ionization (ESI) mass spectra were recorded on a time-of-flight (TOF) micromass spectrometer. 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. All reagents that were used are commercially available.

Equipment used for reflux

A specially designed cylindrical tube was charged with the reagents and fitted with a condenser. The mixture was heated to 180 ^{o}C and the reaction system monitored. During the course of the reaction (maximum 3 hours) vigorous reflux was observed but no tangible loss of solvent was observed.

Synthetic Procedures and Characterization of Lophine Derivatives

2,4,5-Triphenylimidazole (3a)[1,5,6]



2-Phenylacetophenone (98.12 mg, 0.5 mmol), selenium dioxide (55.48 mg, 0.5 mmol), ammonium acetate (385.4 mg, 5.0 mmol), and benzaldehyde (51.02 µl, 0.5 mmol) were mixed in an elongated tube (equipped with a reflux condenser) with 5.00 ml glacial acetic acid and stirred for 3 hours at 180 ^{o}C. After cooling, the reaction mixture was added drop-wise into a 25% ammonia solution at 0 ^{o}C to form a white precipitate which was then filtered and dried in oven at 50 ^{o}C for 4 hours to afford 2,4,5-triphenylimidazole as a white solid (123.30 mg, 83%). Mp 271 – 273 ^{o}C.

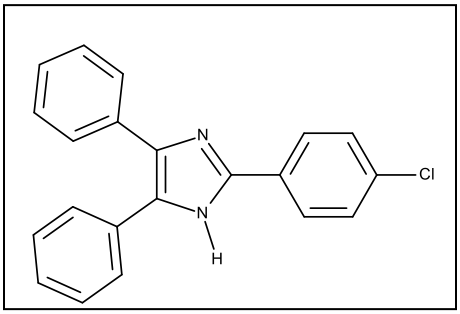
IR (KBr, cm^{-1}): 3426, 2855, 1600, 1488;

^{1}H NMR (400 MHz, DMSO-D_6) δ 12.72 (s, 1H), 8.12 (d, *J* = 7.84 Hz, 2H), 7.62 (d, *J* = 8.08 Hz, 2H), 7.55 (d, *J* = 7.25 Hz, 2H), 7.51 – 7.44 (m, 4H), 7.39 (t, *J* = 7.46 Hz, 2H), 7.32 (t, *J* = 7.46 Hz, 2H) 7.24 (t, *J* = 7.66 Hz, 1H);

^{13}C (100 MHz, DMSO-D_6) δ 146.0, 137.7, 135.7, 131.6, 130.9, 129.1, 128.9, 128.7, 128.7, 127.2, 127.6, 126.9, 125.7;

HRMS (ESI) calcd for $C_{21}H_{16}N_2$ $[M + H]^+$ 297.1392, found 297.1388.

2-(4-Chlorophenyl)-4,5-diphenylimidazole (3b)[1]



Prepared by the procedure given for **3a**, except using 4-chlorobenzaldehyde (70.28 mg, 0.5 mmol), and obtained as a white solid (127.10 mg, 77%).

Mp 258 – 261 ^{o}C.

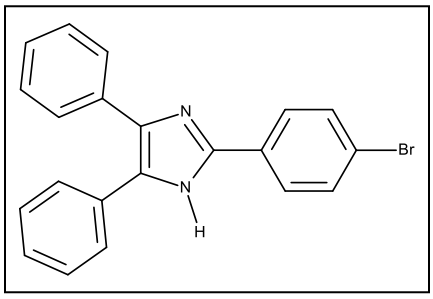
IR (KBr, cm^{-1}): 3423, 3059, 1602, 1324;

^{1}H NMR (400 MHz, DMSO-D_6) δ 12.78 (s, 1H), 8.12 (d, J = 8.57 Hz, 2H), 7.56 (d, J = 8.57 Hz, 6H), 7.38 (s, 6H);

^{13}C (100 MHz, DMSO-D_6) δ 144.9, 133.3, 129.9, 129.8, 129.7, 129.3, 129.2, 129.0, 128.8, 128.3, 127.6, 127.3;

HRMS (ESI) calcd for $C_{21}H_{15}N_2Cl$ $[M + H]^+$ 331.1002, found 331.1009.

2-(4-Bromophenyl)-4,5-diphenylimidazole (3c)[1]



Prepared by the procedure given for **3a**, except using 4-bromobenzaldehyde (92.51 mg, 0.5 mmol), and obtained as a white solid (137.20, 73%).

Mp 255 – 258 ^{o}C.

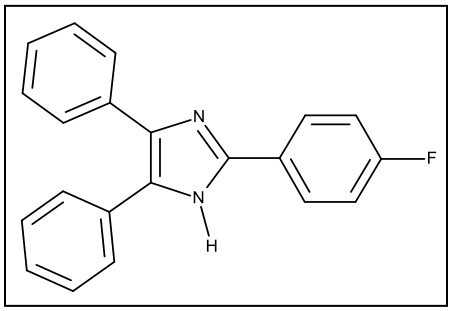
IR (KBr, cm^{-1}): 3408, 3060, 1601, 1323;

^{1}H NMR (400 MHz, DMSO-D_6) δ 12.80 (s, 1H), 8.06 (d, J = 8.42 Hz, 2H), 7.70 (d, J = 8.61 Hz, 2H), 7.55 (d, J = 7.14 Hz, 4H), 7.38 (s, 6H);

^{13}C (100 MHz, DMSO-D_6) δ 144.9, 137.8, 135.5, 132.1, 131.5, 130.0, 129.8, 129.4, 128.9, 127.6, 121.9;

HRMS (ESI) calcd for $C_{21}H_{15}BrN_2$ $[M + H]^+$ 375.0497, found 375.0500.

2-(4-Fluorophenyl)-4,5-diphenyl-1*H*-imidazole (d)[2,3]



Prepared by the procedure given for **3a**, except using 4-fluorobenzaldehyde (53.63 µl, 0.5 mmol), and obtained as a fawn solid (131.30 mg, 84%).

Mp 249 – 253 ^{o}C.

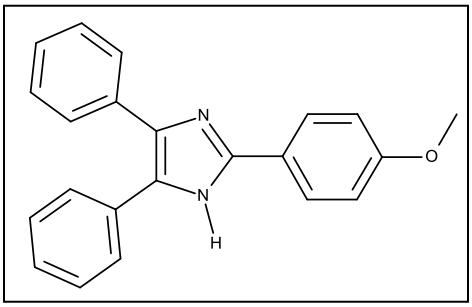
IR (KBr, cm^{-1}): 3408, 3029, 1608, 1492, 1221, 1159, 1131, 694;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.70 (s, 1H), 8.14 (q, *J* = 5.59 Hz, 2H), 7.55 (d, *J* = 6.88 Hz, 4H), 7.36 – 7.31 (m, 8H);

^{13}C (100 MHz, DMSO-D$_6$) δ 163.8, 161.4, 145.2, 137.6, 135.6, 131.5, 129.1, 128.9, 128.7, 128.3, 127.8, 127.8, 127.5, 127.5, 127.0, 116.2, 116.0;

HRMS (ESI) calcd for $C_{21}H_{15}FN_2$ $[M + H]^+$ 315.1298, found 315.1307.

2-(4-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (3e)[1,2]



Prepared by the procedure given for **3a**, except using 4-methoxybenzaldehyde (60.83 µl, 0.5 mmol), and obtained as a pale-white solid (141.30 mg, 87%).

Mp 226 – 229 ^{o}C.

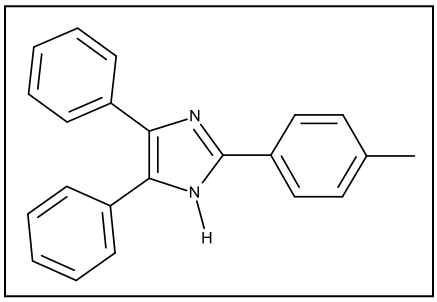
IR (KBr, cm^{-1}): 3400, 3027, 1613, 1492, 1174;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.52 (s, 1H), 8.03 (d, *J* = 9.13 Hz, 2H), 7.56 – 7.51 (d, *J* = 13.52, 4H) 7.44 – 7.23 (m, 6H), 7.07 (d, *J* = 8.73 Hz, 2H), 3.83 (s, 3H);

^{13}C (100 MHz, DMSO-D$_6$) δ 159.9, 146.1, 137.3, 135.8, 131.7, 129.1, 128.8, 128.6, 128.1, 128.1, 127.5, 127.2, 126.9, 123.7, 114.6, 55.7;

HRMS (ESI) calcd for $C_{22}H_{18}N_2O$ $[M + H]^+$ 327.1497, found 327.1494.

4,5-Diphenyl-2-(*p*-tolyl)-1*H*-imidazole (3f)[1,3]



Prepared by the procedure given for **3a**, except using *p*-tolualdehyde (58.95 µl, 0.5 mmol), and obtained as a pale-white solid (129.00 mg, 83%).

Mp 230 – 232 ^{o}C.

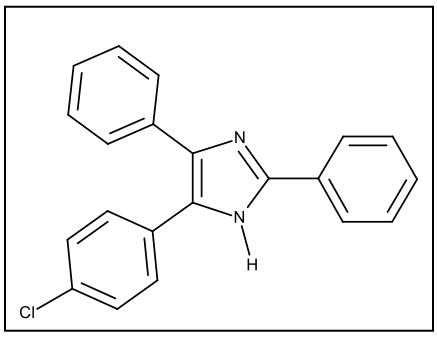
IR (KBr, cm^{-1}): 3394, 3029, 1603, 1486, 1321;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.48 (s, 1H), 7.74 (t, *J* = 4.23 Hz, 1H), 7.59 – 7.52 (q, *J* = 7.40 Hz, 4H), 7.46 – 7.42 (t, *J* = 7.16 Hz, 2H), 7.34 – 7.29 (m, 6H), 7.24 – 7.22 (d, *J* = 7.24 Hz, 1H), 2.66 (s, 3H);

^{13}C (100 MHz, DMSO-D$_6$) δ 146.6, 137.1, 136.8, 135.9, 131.7, 131.6, 130.5, 129.2, 129.1, 128.8, 128.7, 128.6, 128.1, 127.9, 127.5, 126.9, 126.2, 21.6;

HRMS (ESI) calcd for $C_{22}H_{18}N_2$ $[M + H]^+$ 311.1548, found 311.1554.

5-(4-Chlorophenyl)-2,4-diphenyl-1*H*-imidazole (3g)[4]



Prepared by the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol), and obtained as a white solid (130.20 mg, 82%).

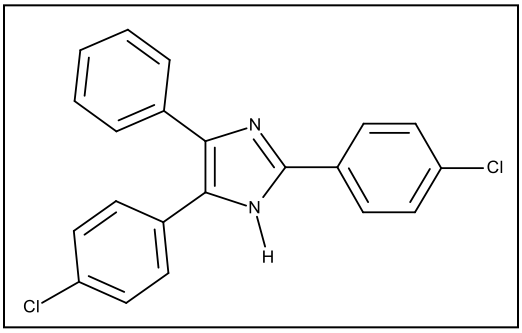
Mp 241 – 245 ^{o}C..

IR (KBr, cm^{-1}): 3415, 3055, 1600, 1322; ^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.73 (s, 1H), 8.10 (d, J = 7.79 Hz, 2H), 7.55 – 7.38, (m, 12H);

^{13}C (100 MHz, DMSO-D$_6$) δ 146.4, 146.2, 138.2, 136.3, 135.5, 134.5, 132.7, 131.5, 131.3, 130.7, 130.5, 130.3, 129.2, 129.1, 129.0, 128.8, 128.7, 128.5, 127.7, 127.4, 127.2, 125.7;

HRMS (ESI) calcd for $C_{21}H_{15}ClN_2$ $[M + H]^+$ 331.1002, found 331.1007.

2,5-Bis(4-chlorophenyl)-4-phenyl-1*H*-imidazole (3h)



Prepared by the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-chlorobenzaldehyde (70.30 mg, 0.5 mmol), and obtained as a white solid (135.60 mg, 74%).

Mp 249 – 252 ^{o}C.

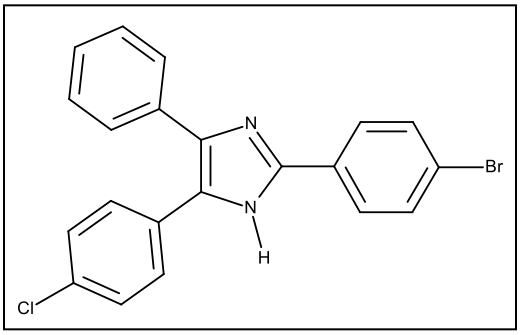
IR (KBr, cm^{-1}): 3419, 3063, 1600, 1311;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.83 (s, 1H), 8.10 (d, J = 8.66 Hz, 2H), 7.56 – 7.37 (m, 11H);

^{13}C (100 MHz, DMSO-D$_6$) δ 145.2, 133.4, 129.5, 129.3, 129.1, 128.8, 127.4;

HRMS (ESI) calcd for $C_{21}H_{14}N_2Cl_2$ $[M + H]^+$ 365.0612, found 365.0615.

2-(4-Bromophenyl)-5-(4-chlorophenyl)-4-phenyl-1*H*-imidazole (3i)



Prepared by the procedure given for **3a**, using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-bromobenzaldehyde (92.50 mg, 0.5 mmol), and obtained as a white solid (135.60 mg, 87%).

Mp 248 – 250 ^{o}C.

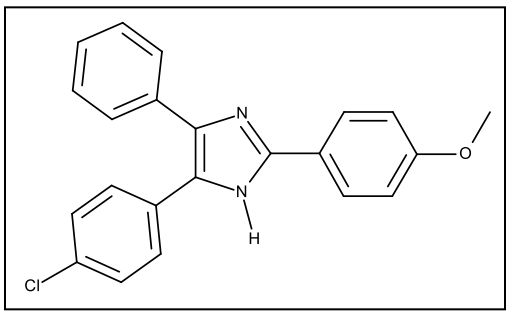
IR (KBr, cm^{-1}): 3416, 3064, 1600, 1323;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.84 (s, 1H), 8.04 (d, *J* = 9.00 Hz, 2H), 7.69 (d, *J* = 8.53 Hz, 2H), 7.54 – 7.36 (m, 9H);

^{13}C (100 MHz, DMSO-D$_6$) δ 145.2, 132.2, 129.9, 129.1, 128.9, 127.6, 122.0;

HRMS (ESI) calcd for $C_{21}H_{14}BrClN_2$ $[M + H]^+$ 409.0107, found 409.0117.

5-(4-Chlorophenyl)-2-(4-methoxyphenyl)-4-phenyl-1*H*-imidazole (3j)



Prepared by the procedure given for **3a**, except using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-methoxybenzaldehyde (60.83 µl, 0.5 mmol), and obtained as a white solid (109.80 mg, 61%).

Mp 233 – 237 ^{o}C.

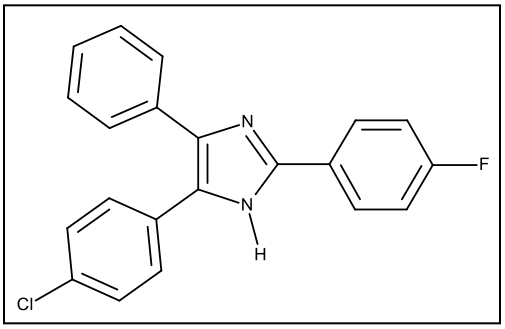
IR (KBr, cm^{-1}): 3408, 3060, 2289, 1601, 1220;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.58 (s, 1H), 8.01 (d, *J* = 8.21 Hz, 2H), 7.57 – 7.44 (m, 6H), 7.41 – 7.27 (m, 3H), 7.07 (d, *J* = 8.21 Hz, 2H), 3.83 (s, 3H);

^{13}C (100 MHz, DMSO-D$_6$) δ 160.0, 146.3, 137.9, 135.9, 135.6, 134.7, 132.5, 131.5, 131.3, 130.3, 129.2, 129.0, 128.9, 128.7, 128.3, 127.8, 127.3, 123.5, 114.6, 55.7;

HRMS (ESI) calcd for $C_{22}H_{17}ClN_2O$ $[M + H]^+$ 361.1108, found 361.1097.

5-(4-Chlorophenyl)-2-(4-fluorophenyl)-4-phenyl-1*H*-imidazole (3k)



Prepared by the procedure given for **3a**, except using 4-chloro-2-phenylacetophenone (115.30 mg, 0.5 mmol) and 4-fluorobenzaldehyde (53.63 µl, 0.5 mmol), and obtained as a cream-white solid (132.40 mg, 76%).

Mp 245 – 249 ^{o}C.

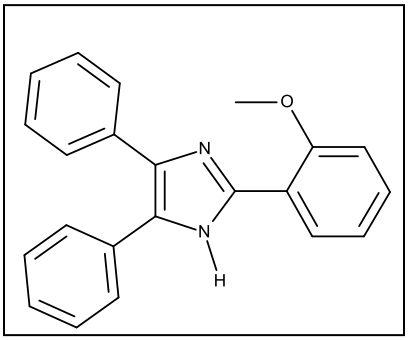
IR (KBr, cm^{-1}): 3431, 3062, 1488, 1222, 1159, 1129, 698;

^{1}H NMR (400 MHz, DMSO-D_6) δ 12.75 (s, 1H), 8.13 (q, J = 5.58 Hz, 2H), 7.56 – 7.52 (t, J = 8.30 Hz, 4H), 7.39 – 7.31 (m, 7H);

^{13}C (100 MHz, DMSO-D_6) δ 163.9, 161.4, 145.4, 129.0, 127.9, 127.8, 127.4, 127.3, 116.2, 116.0;

HRMS (ESI) cacld for $C_{21}H_{14}ClFN_2$ $[M + H]^+$ 349.0912, found 349.0912.

2-(2-Methoxyphenyl)-4,5-diphenyl-1*H*-imidazole (3l)[5]



Prepared by the procedure given for **3a**, except using 2-methoxybenzaldehyde (60.40 µl, 0.5 mmol), and obtained as a white solid (145.20 mg, 88%).

Mp 230 – 233 ^{o}C.

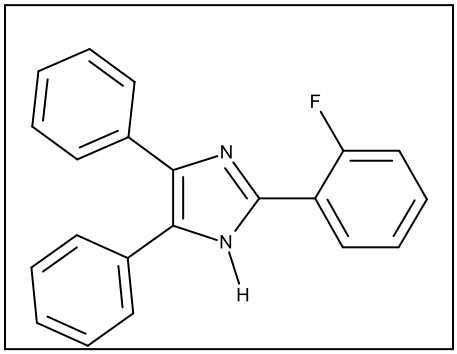
IR (KBr, cm^{-1}): 3429, 3032, 2940, 1601, 1481;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 11.89 (s, 1H), 8.07 (d, *J* = 5.91 Hz, 2H), 7.55 (d, *J* = 7.14 Hz, 2H), 7.48 (d, *J* = 7.14 Hz, 2H), 7.44 – 7.37 (m, 4H), 7.31 (t, *J* = 7.45 Hz, 2H), 7.22 – 7.18 (m, 2H), 7.08 (t, *J* = 7.45 Hz, 1H), 3.93 (s, 3H);

^{13}C (100 MHz, DMSO-D$_6$) δ 156.5, 143.7, 136.9, 135.8, 131.7, 130.2, 129.3, 129.1, 129.0, 128.6, 128.1, 127.9, 127.6, 126.9, 121.1, 119.4, 112.1, 56.0;

HRMS (ESI) calcd for $C_{22}H_{18}N_2O$ $[M + H]^-$ 325.1341, found 325.1345.

2-(2-Fluorophenyl)-4,5-diphenyl-1*H*-imidazole (3m)[2]



Prepared by the procedure given for **3a**, except using 2-fluorobenzaldehyde (52.69 µl, 0.5 mmol), and obtained as a white solid (130.80 mg, 83%).

Mp 238 – 241 ^{o}C.

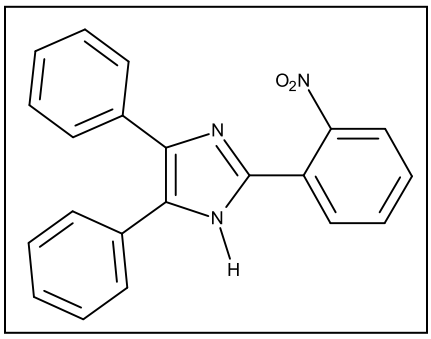
IR (KBr, cm^{-1}): 3453, 3058, 1602, 1484, 1220, 1101, 695;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.55 (s, 1H), 8.02 (t, J = 7.80 Hz, 1H), 7.54 – 7.33, (m, 13H);

^{13}C (100 MHz, DMSO-D$_6$) δ 160.6, 158.1, 141.3, 141.3, 131.4, 130.9, 130.8, 130.1, 130.1, 129.6, 129.0, 128.3, 127.6, 127.1, 125.1, 125.1, 119.2, 119.1, 116.8, 116.6;

HRMS (ESI) calcd for $C_{21}H_{15}FN_2$ $[M + Na]^+$ 337.1117, found 337.1114.

2-(2-Nitrophenyl)-4,5-diphenyl-1*H*-imidazole (3n)[2,5]



Prepared by the procedure given for **3a**, except using 2-nitrobenzaldehyde (75.60 mg, 0.5 mmol), and obtained as a yellow solid (142.00 mg, 83%).

Mp 229 – 232 ^{o}C.

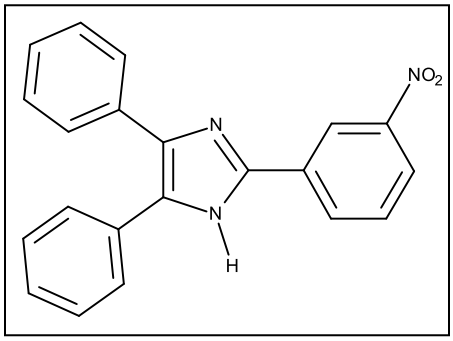
IR (KBr, cm^{-1}): 3397, 3031, 1601, 1524, 1351, 1143, 693;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 12.95 (s, 1H), 8.00 (d, *J* = 8.51 Hz, 1H), 7.93 (d, *J* = 7.09 Hz, 1H), 7.79 (t, *J* = 7.44 Hz, 1H), 7.65 (t, *J* = 7.80 Hz, 1H), 7.51 (d, *J* = 7.80 Hz, 4H), 7.39 – 7.25 (m, 6h);

^{13}C (100 MHz, DMSO-D$_6$) δ 148.8, 141.5, 132.6, 130.3, 129.9, 129.4, 129.2, 128.9, 128.8, 127.6, 124.5, 123.9;

HRMS (ESI) calcd for $C_{21}H_{15}N_3O$ $[M + H]^+$ 342.1243, found 342.1248.

2-(3-Nitrophenyl)-4,5-diphenyl-1*H*-imidazole (3o)[5]



Prepared by the procedure given for **3a**, except using 2-nitrobenzaldehyde (75.60 mg, 0.5 mmol), and obtained as a yellow solid (87.40 mg, 51%).

Mp 281 – 285 ^{o}C.

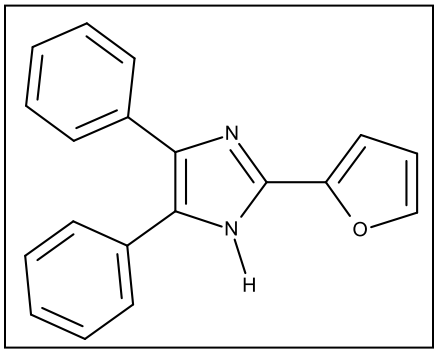
IR (KBr, cm^{-1}): 3394, 3057, 1520, 1347;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 13.08 (s, 1H), 8.97 (s, 1H), 8.51 (d, *J* = 8.01 Hz, 1H), 8.20 (d, *J* = 8.35 Hz, 1H), 7.77 (t, *J* = 8.02 Hz, 1H), 7.55 (d, *J* = 7.47 Hz, 4H), 7.40 (brs, 6H);

^{13}C (100 MHz, DMSO-D$_6$) δ 148.8, 143.9, 132.3, 131.6, 130.8, 129.4, 128.9, 128.7, 128.2, 123.0, 119.9;

HRMS (ESI) calcd for $C_{21}H_{15}N_3O$ $[M + Na]^+$ 364.1062, found 364.1071.

2-(2-Furyl)-4,5-diphenyl-1*H*-imidazole (3p)[3]



Prepared by the procedure given for 3a, except using furfural (41.42 µl, 0.5 mmol), and obtained as a brown solid (95.30 mg, 67%).

Mp 237 – 239 ^{o}C.

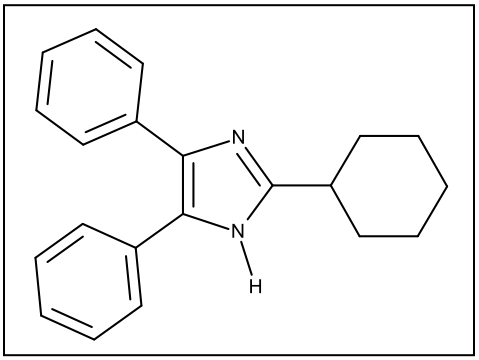
IR (KBr, cm^{-1}): 3023, 1601, 1500, 1485, 739, 695;

^{1}H NMR (400 MHz, DMSO-D_6) δ 12.81 (s, 1H), 8.06 (d, 2H), 7.81, (s, 1H), 7.54 – 7.48 (dd, *J* = 7.16 Hz, 4H), 7.43 (t, *J* = 7.78 Hz, 2H), 7.37 (t, *J* = 7.37 Hz, 1H), 7.31 (t, *J* = 7.37 Hz, 2H), 7.23 (t, *J* = 7.37 Hz, 1H), 6.98 (d, *J* = 3.41 Hz, 1H), 6.66 (s, 1H);

^{13}C (100 MHz, DMSO-D_6) δ 146.2, 143.5, 139.1, 137.5, 135.4, 131.3, 129.1, 128.8, 128.7, 128.3, 128.0, 127.6, 127.1, 112.3, 107.9;

HRMS (ESI) calcd for $C_{21}H_{15}N_3O$ $[M + Na]^+$ 309.1861, found 309.1007.

2-Cyclohexyl-4,5-diphenyl-1*H*-imidazole (3q)[4]



2-phenylacetophenone (98.12 mg, 0.5 mmol) and selenium dioxide (55.48 mg, 0.5 mmol) were reacted under glacial acetic acid (5.00 ml) in an elongated tube equipped with a reflux condenser and a stirrer bar magnet for 3 hours at 180 ^{o}C. Subsequently, cyclohexane carboxaldehyde (60.57 µl, 0.5 mmol) and ammonium acetate (385.4 mg, mmol, 5.0 mmol) were directly added into the reaction mixture and allowed to react for another 3 hours at 180 ^{o}C. After cooling, the ultimate reaction mixture was added dropwise into a 25% ammonia solution at 0 ^{o}C to form a white precipitate which was then filtered and dried in oven at 50 ^{o}C for 4 hours to afford 2-cyclohexyl-4,5-diphenyl-1*H*-imidazole as a brown solid (133.30 mg, 83%).

Mp 243 – 245 ^{o}C.

IR (KBr, cm^{-1}): 3031, 1603;

^{1}H NMR (400 MHz, DMSO-D$_6$) δ 11.92 (s, 1H), 7.50 (d, *J* = 7.94 Hz, 2H), 7.42 (q, *J* = 7.19 Hz, 4H), 7.26 (m, 3H), 7.17 (t, *J* = 6.95 Hz, 1H), 2.71 (m, 1H), 1.97 (d, *J* = 11.61 Hz, 2H), 1.79 (d, *J* = 12.44 Hz, 2H), 1.68 – 1.56 (m, 3H), 1.42 – 1.23 (m, 3H);

^{13}C (100 MHz, DMSO-D$_6$) δ 152.9, 136.3, 135.5, 132.1, 129.0, 128.5, 128.3, 127.6, 127.5, 126.5, 126.2, 37.7, 32.0, 26.2;

HRMS (ESI) calcd for $C_{21}H_{15}N_3O$ $[M + H]^+$ 303.1861, found 303.1869.

References

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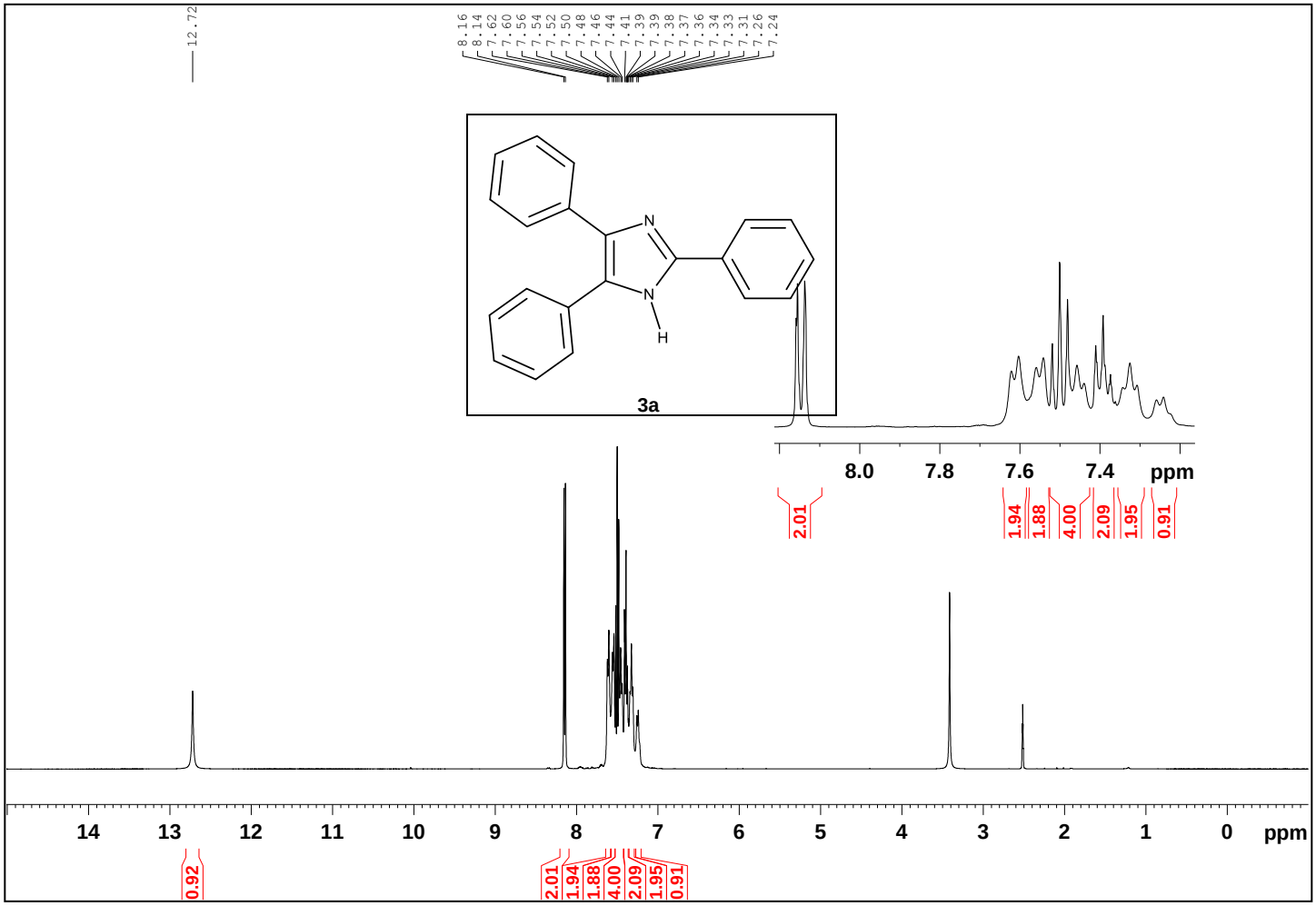
3. C. Yu, M. Lei, W. Su, Y. Xie, *Synthetic Communications* 2007, **37**, 3301.
4. Z. Xie, J. Deng, Z. Qui, J. Li, Q. Zhu, *Chem. Commun.* 2016, **52**, 6467.
5. S. Samai, G. C. Nandi, P. Singh, M. S. Singh, *Tetrahedron* 2009, **65**, 10155.
6. Z. Zarnegar, J. Safari, *RSC Advances* 2014, **4**, 20932.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8.14
7.62
7.60
7.56
7.54
7.52
7.50
7.48
7.46
7.44
7.41
7.39
7.39
7.38
7.37
7.36
7.34
7.33
7.31
7.26
7.24
N
N
H
3a
8.0
7.8
7.6
7.4
ppm
2.01
1.94
1.88
4.00
2.09
1.95
0.91
14
13
12
11
10
9
8
7
6
5
4
3
2
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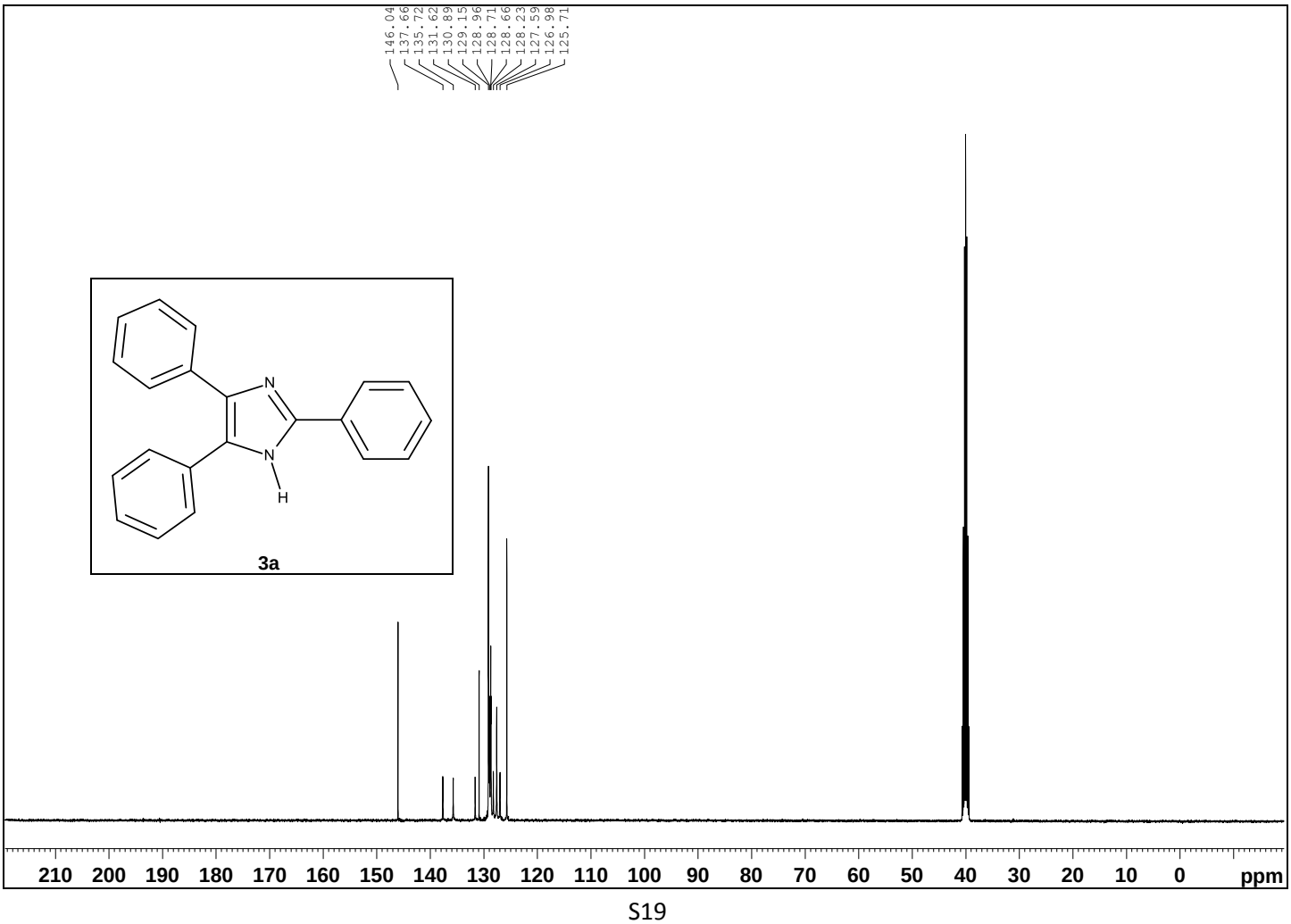
0
ppm
0.92
2.01
1.94
1.88
4.00
2.09
1.95
0.91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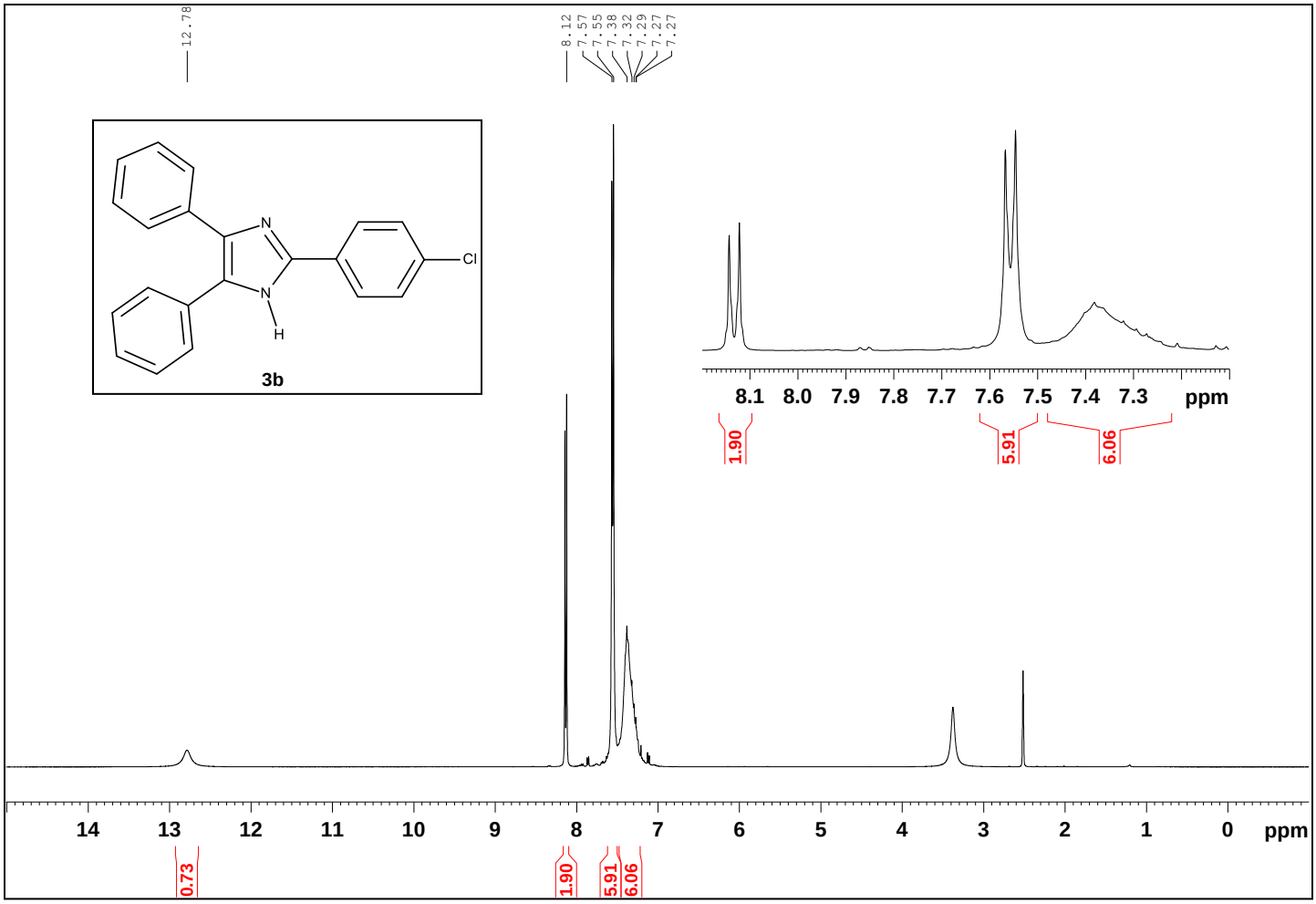
137.66
135.72
131.62
130.89
129.15
128.96
128.71
128.66
128.23
127.59
126.98
125.71
N
N
H
3a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.57
7.55
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7.29
7.27
7.27
N
N
H
Cl
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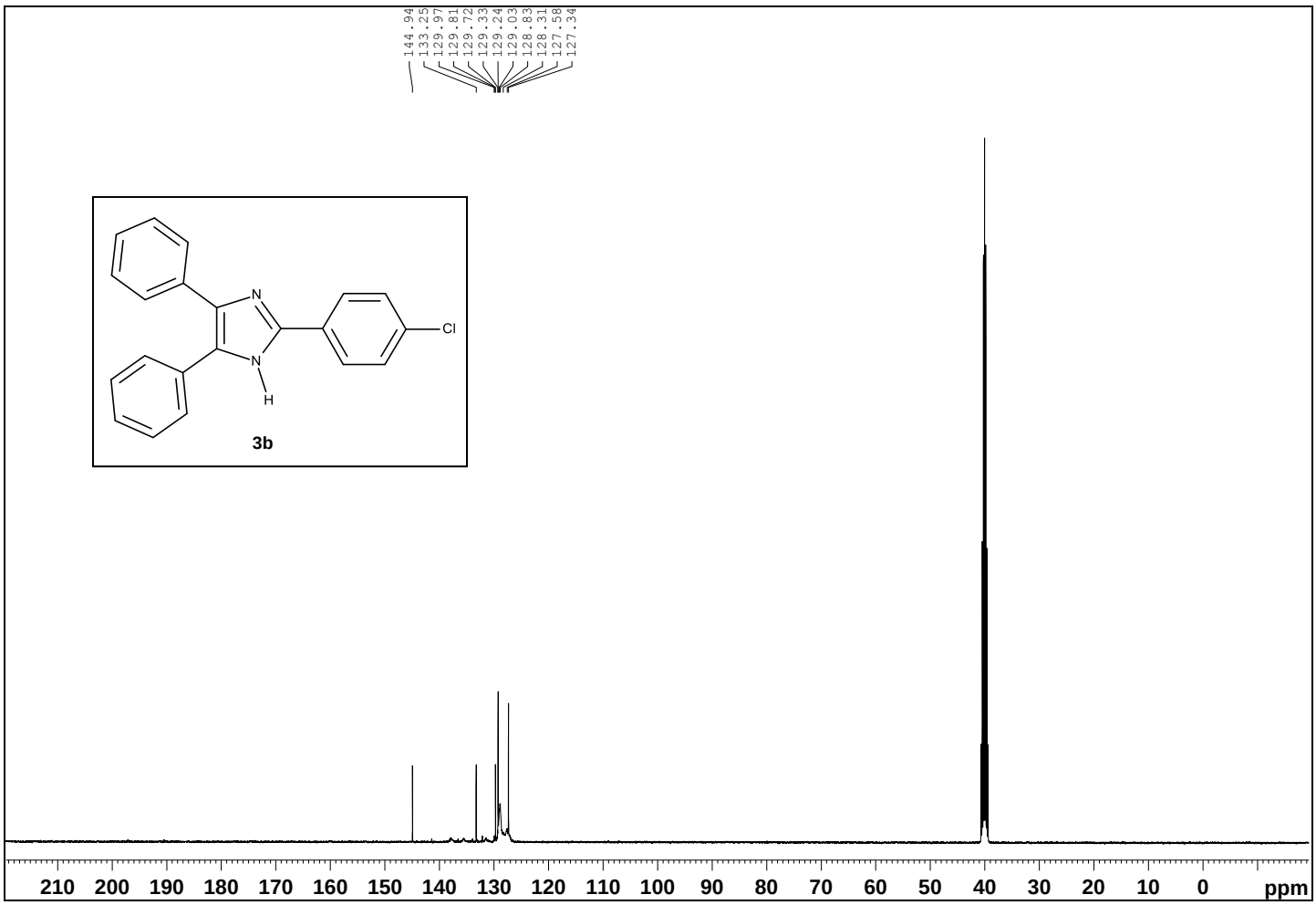
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
1.90
5.91
6.06
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.73
1.90
5.91
6.06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129.03
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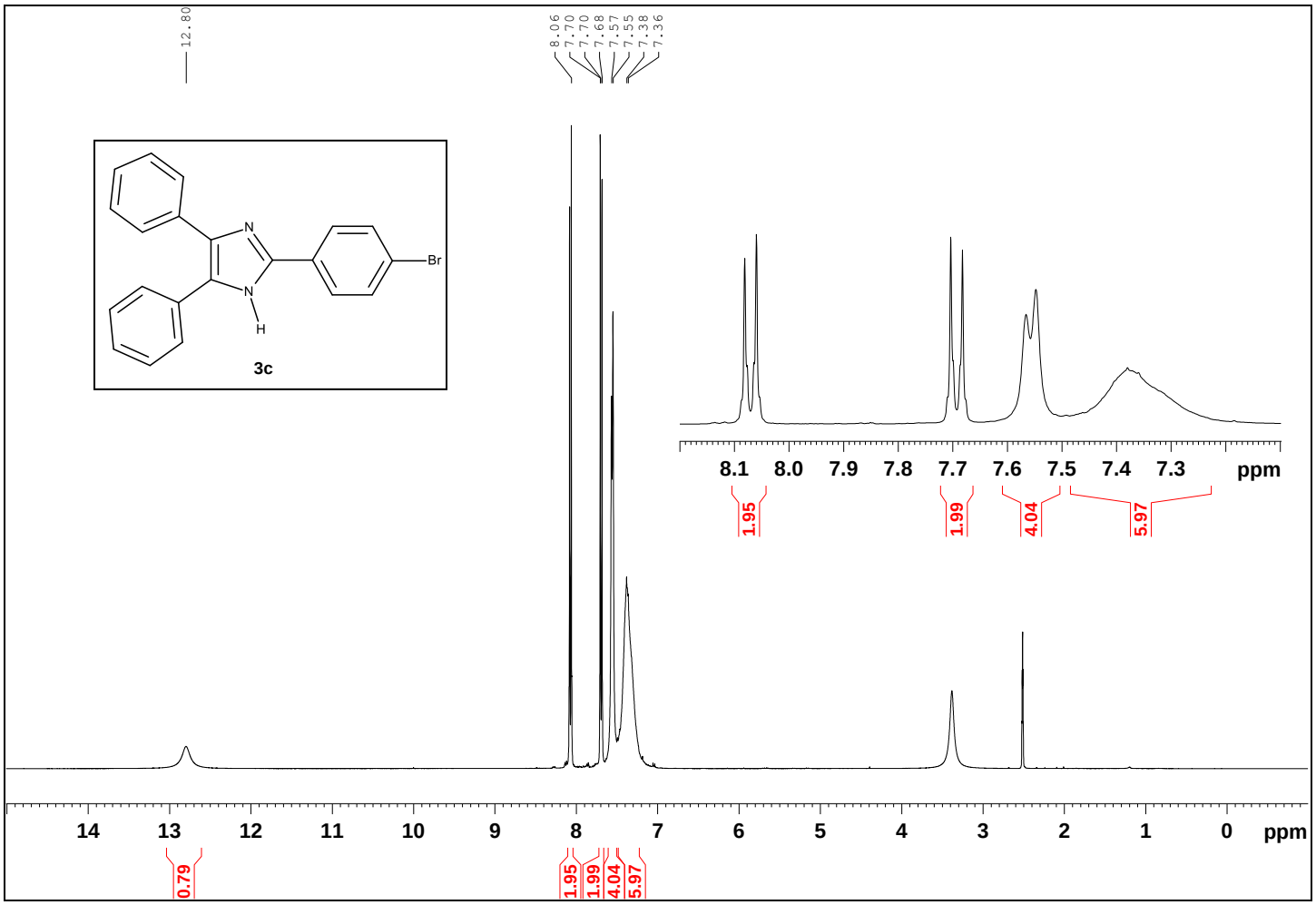
H
Cl
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.06
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7.70
7.68
7.57
7.55
7.38
7.36
N
N
H
Br
3c
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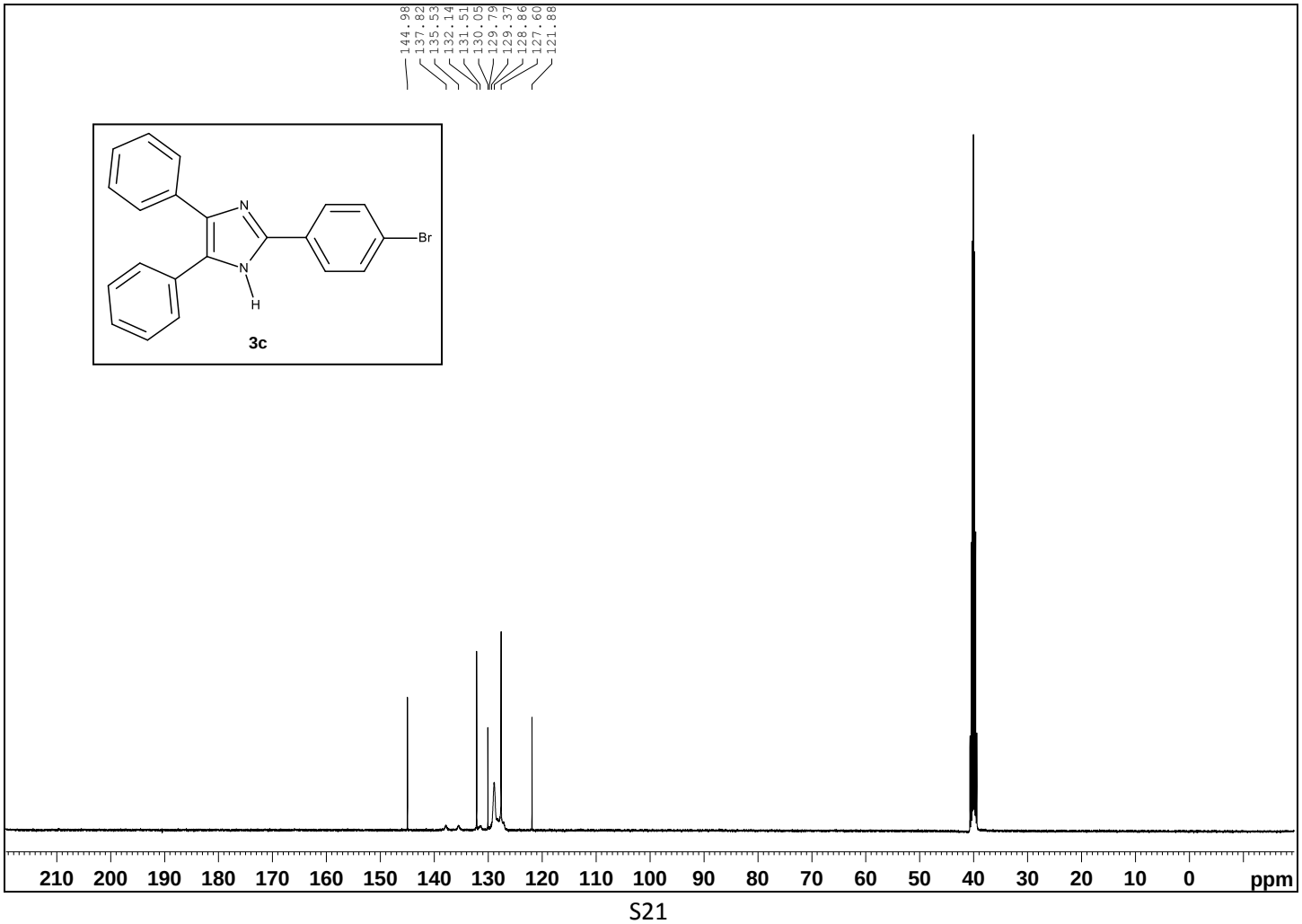
1.95
1.99
4.04
5.97
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.79
1.95
1.99
4.04
5.97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129.79
129.37
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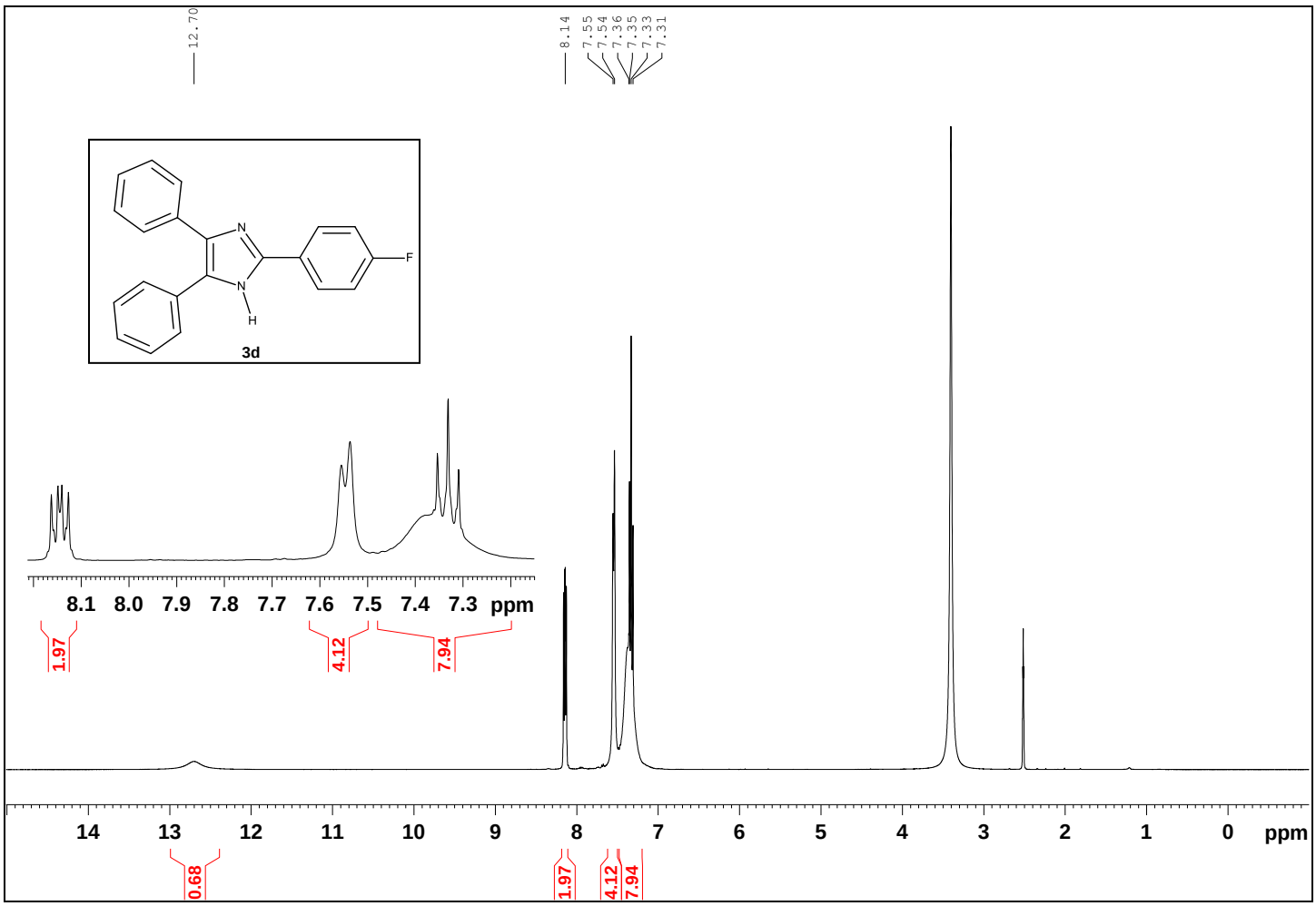
H
Br
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.14
7.55
7.54
7.36
7.35
7.33
7.31
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N
H
F
3d
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4.12
7.94
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
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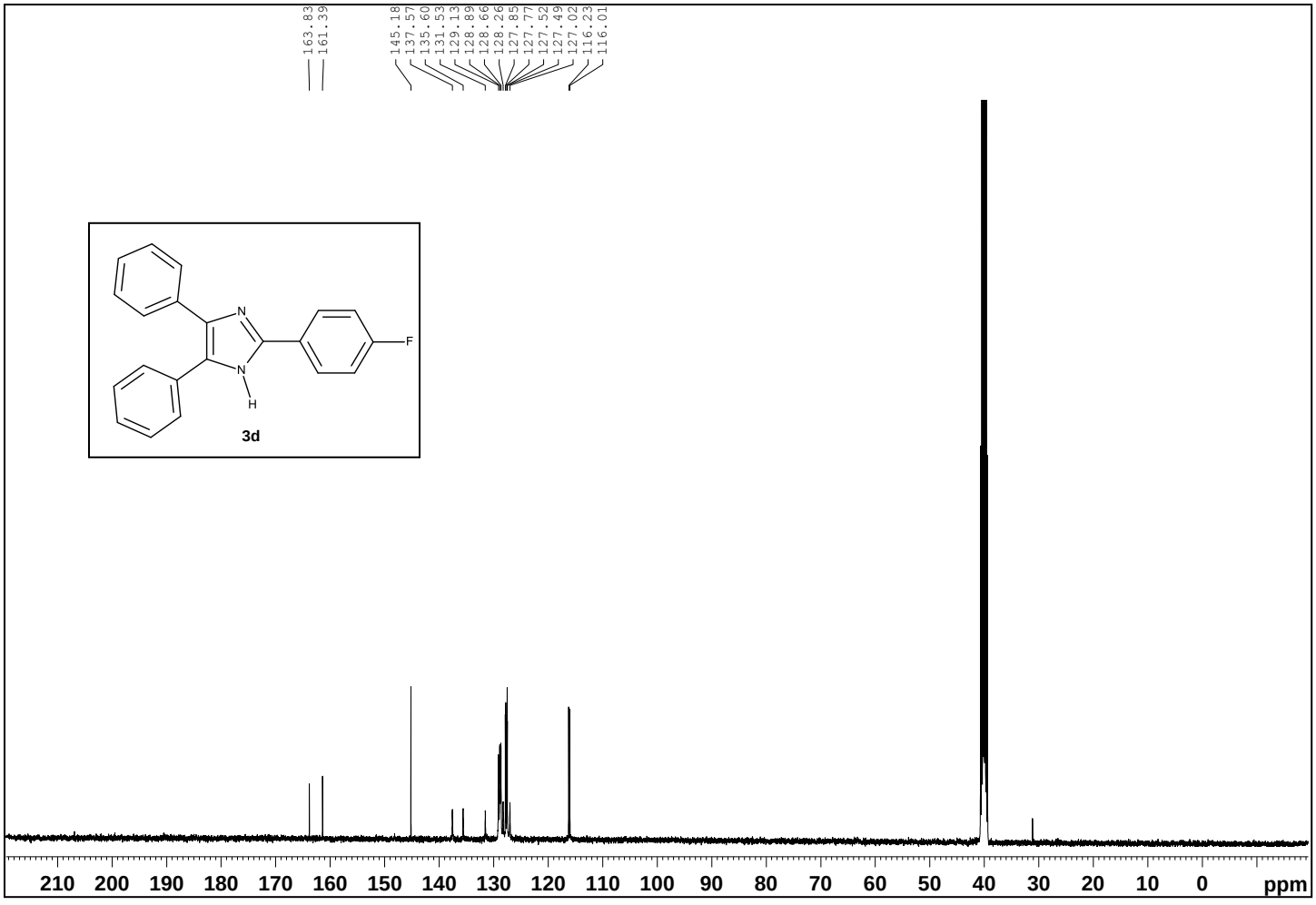
1.97
4.12
7.94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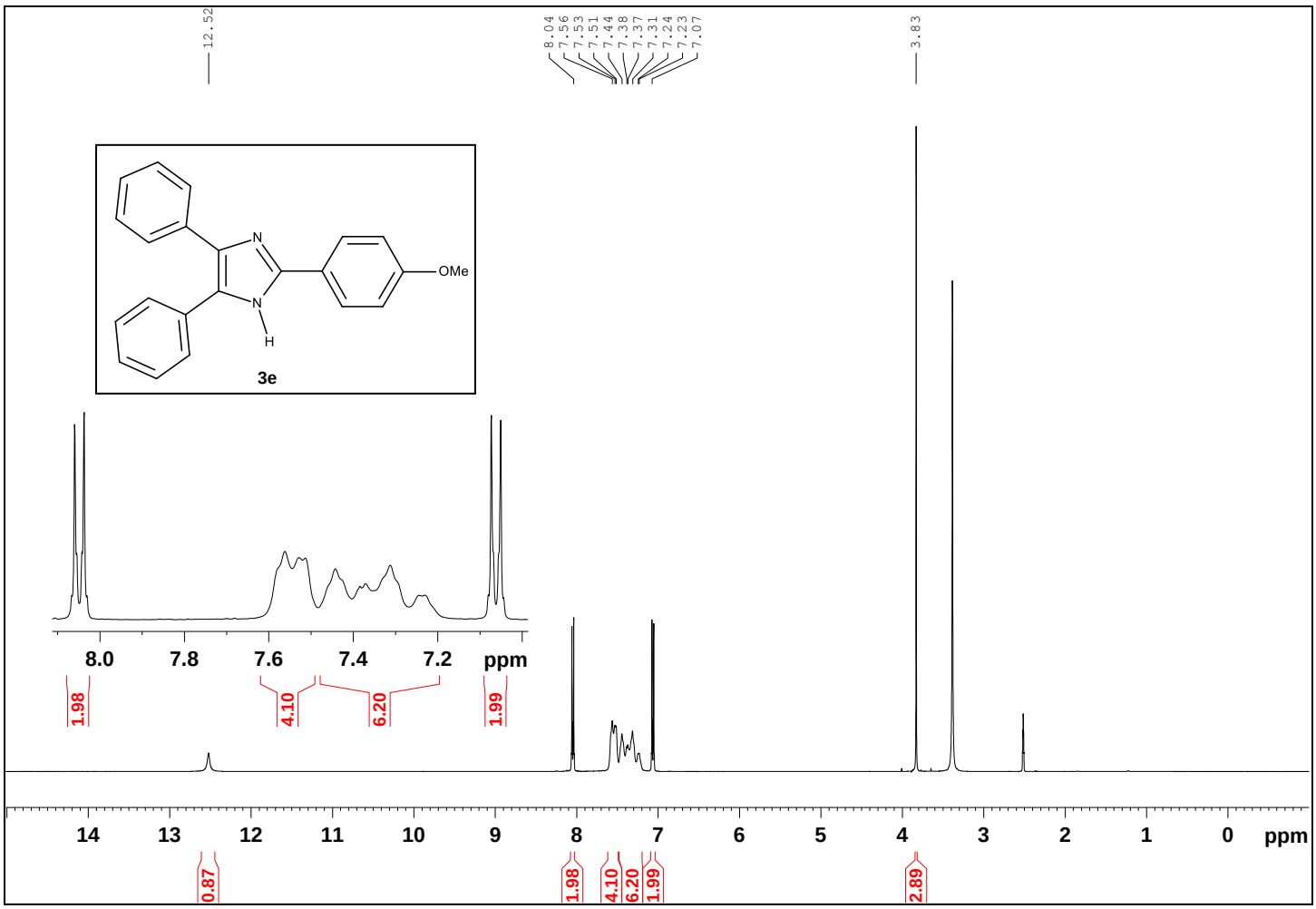
137.57
135.60
131.53
129.13
128.89
128.66
128.26
127.85
127.77
127.52
127.49
127.02
116.23
116.01
N
N
H
F
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.51
7.44
7.38
7.37
7.31
7.24
7.23
7.07
3.83
OMe
N
N
H
3e
8.0
7.8
7.6
7.4
7.2
ppm
1.98
4.10
6.20
1.99
14
13
12
11
10
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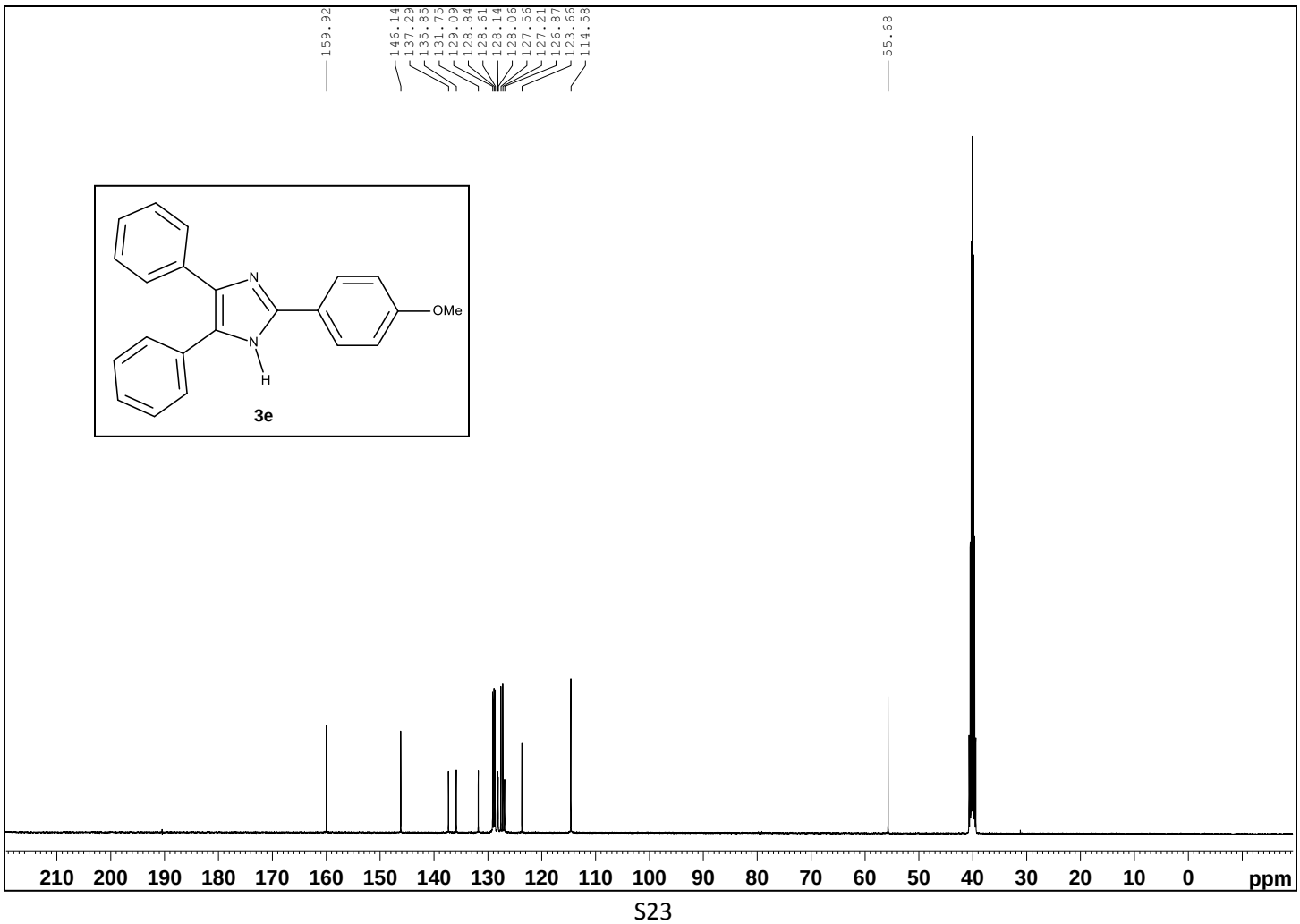
6.20
1.99
2.89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126.87
123.66
114.58
55.68
OMe
N
N
H
3e
210
200
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140
130
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110
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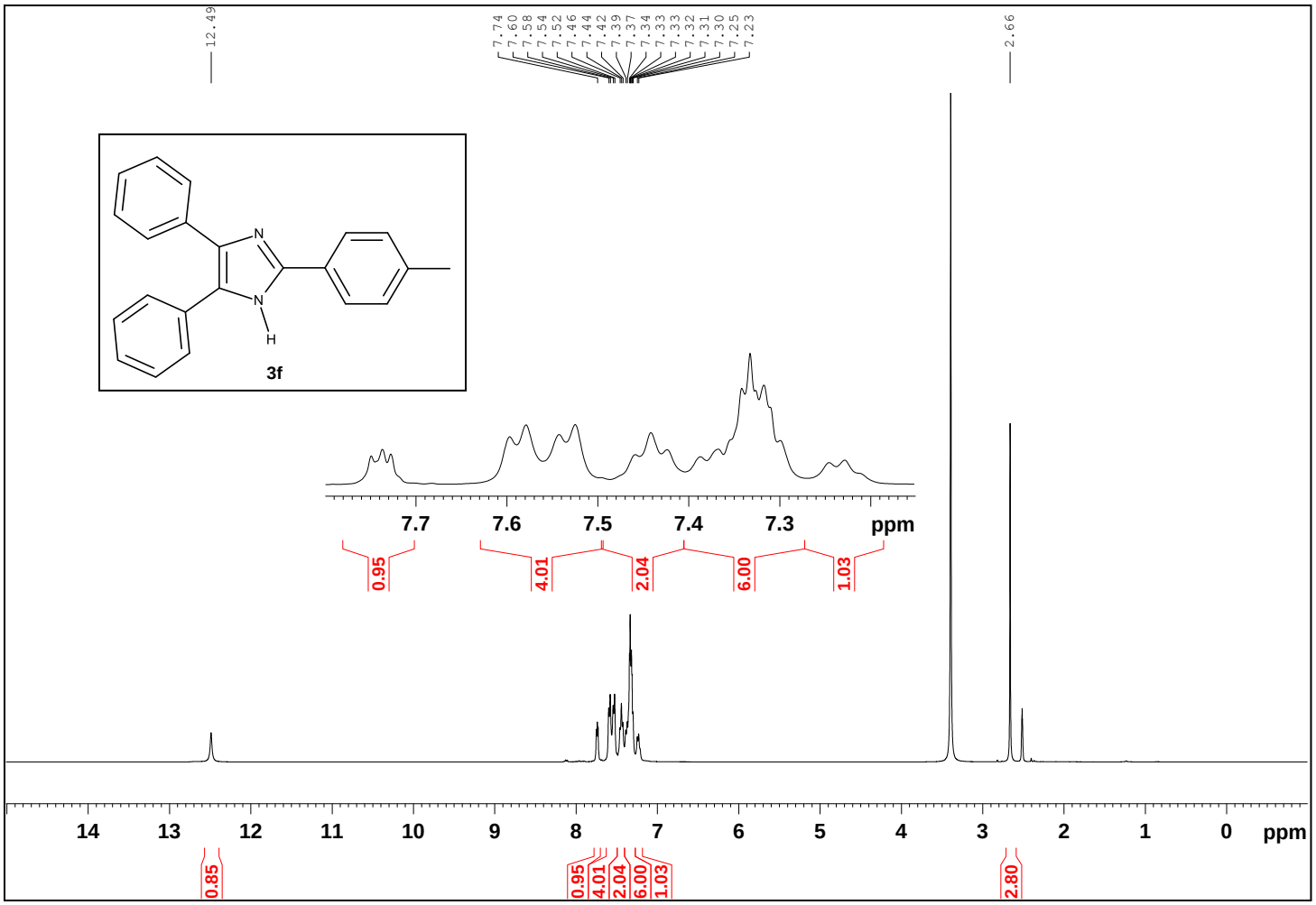
30
20
10
0
ppm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7.30
7.25
7.23
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H
3f
7.7
7.6
7.5
7.4
7.3
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2.04
6.00
1.03
14
13
12
11
10
9
8
7
6
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4.01
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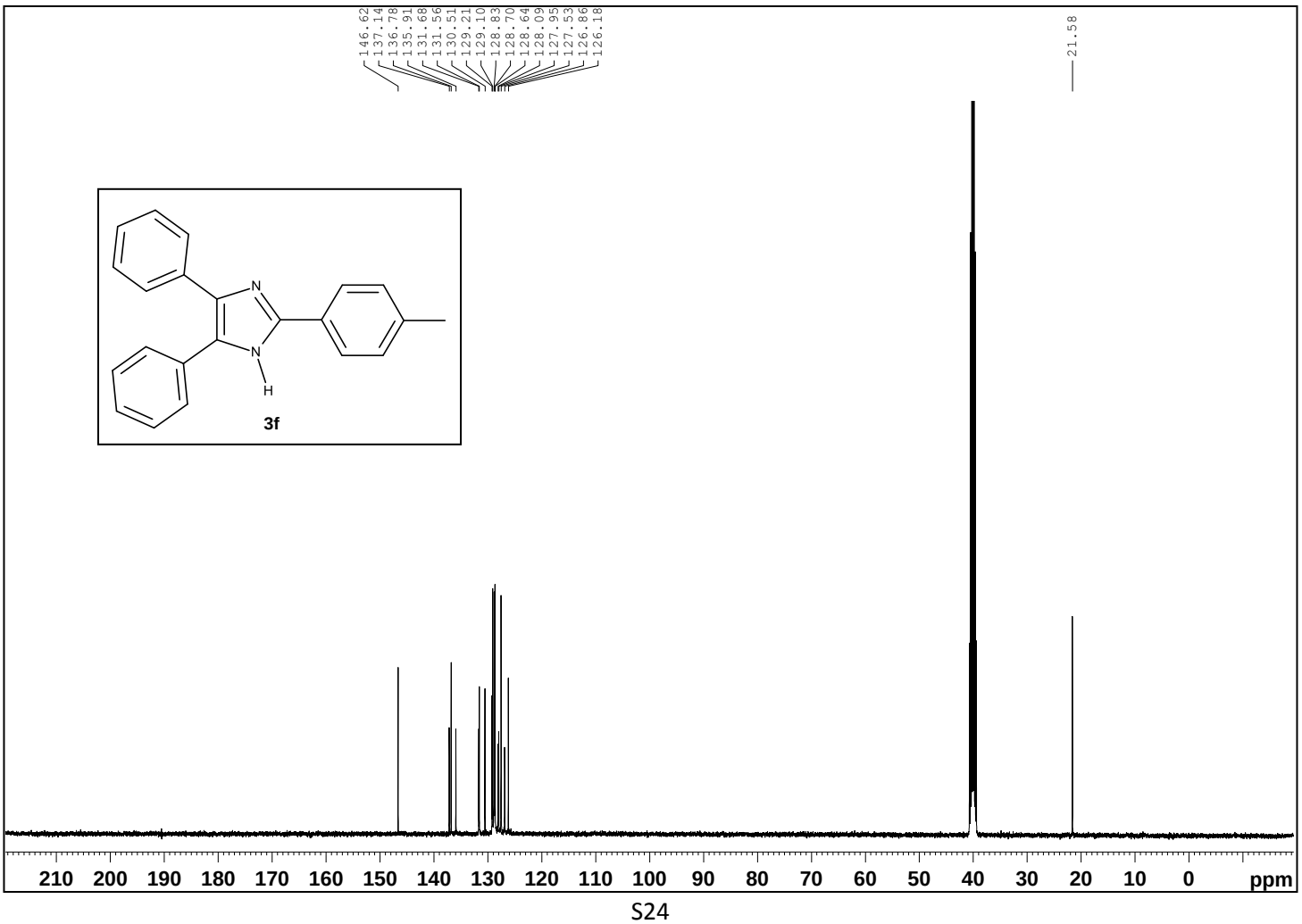
6.00
1.03
2.80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126.18
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N
N
H
3f
210
200
190
180
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20
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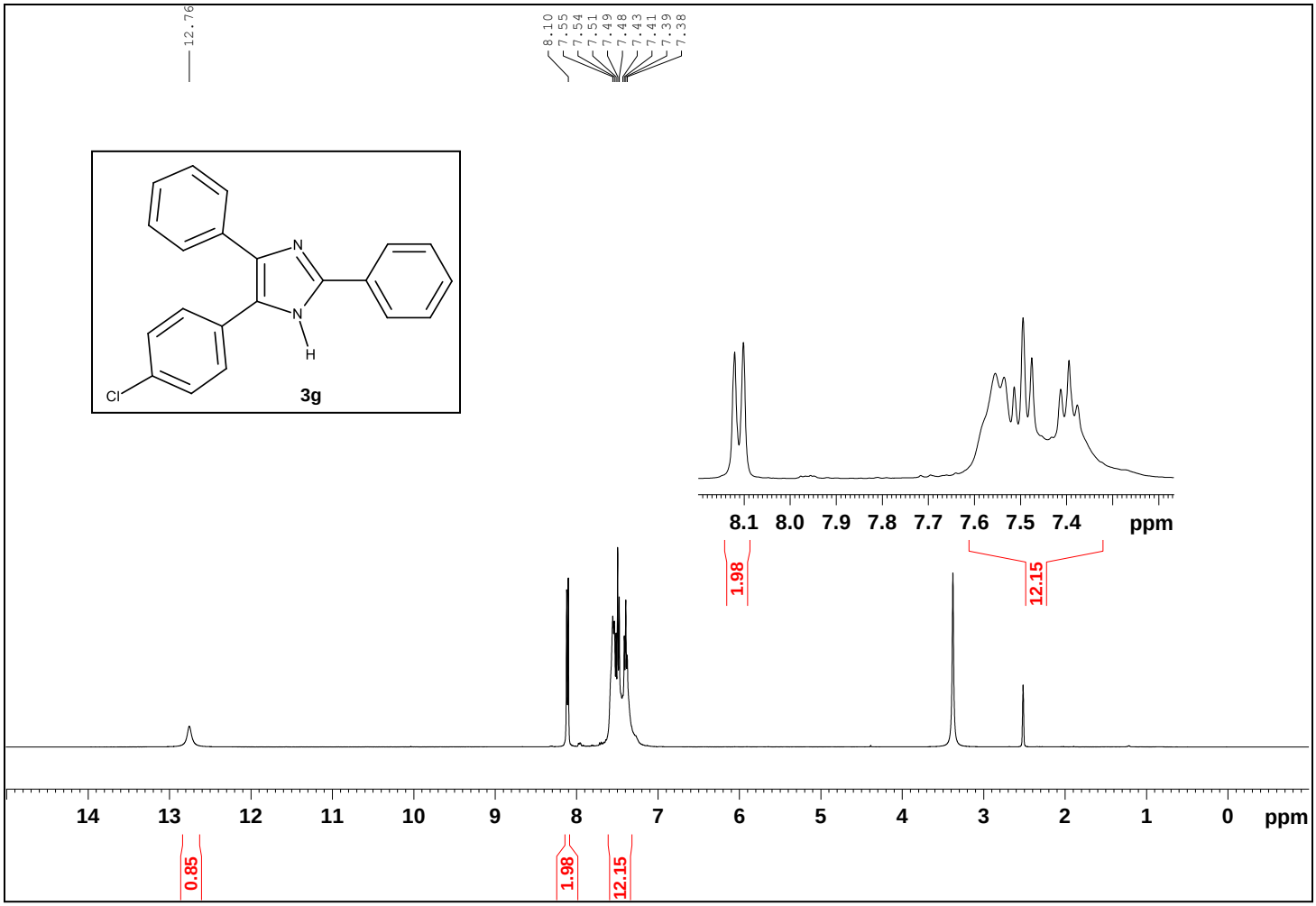
0
ppm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7.39
7.38
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N
H
Cl
3g
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 ppm
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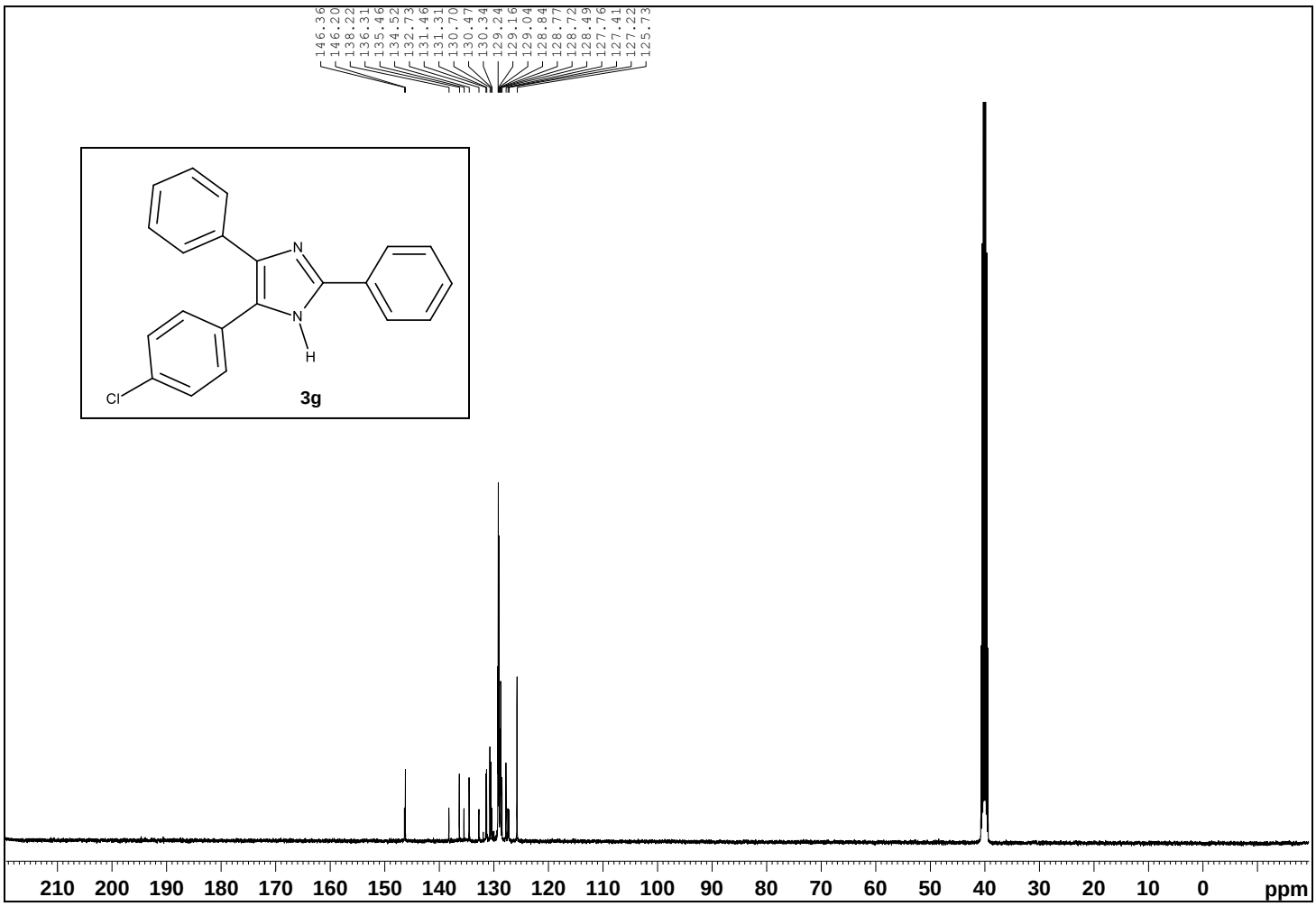
1.98
12.15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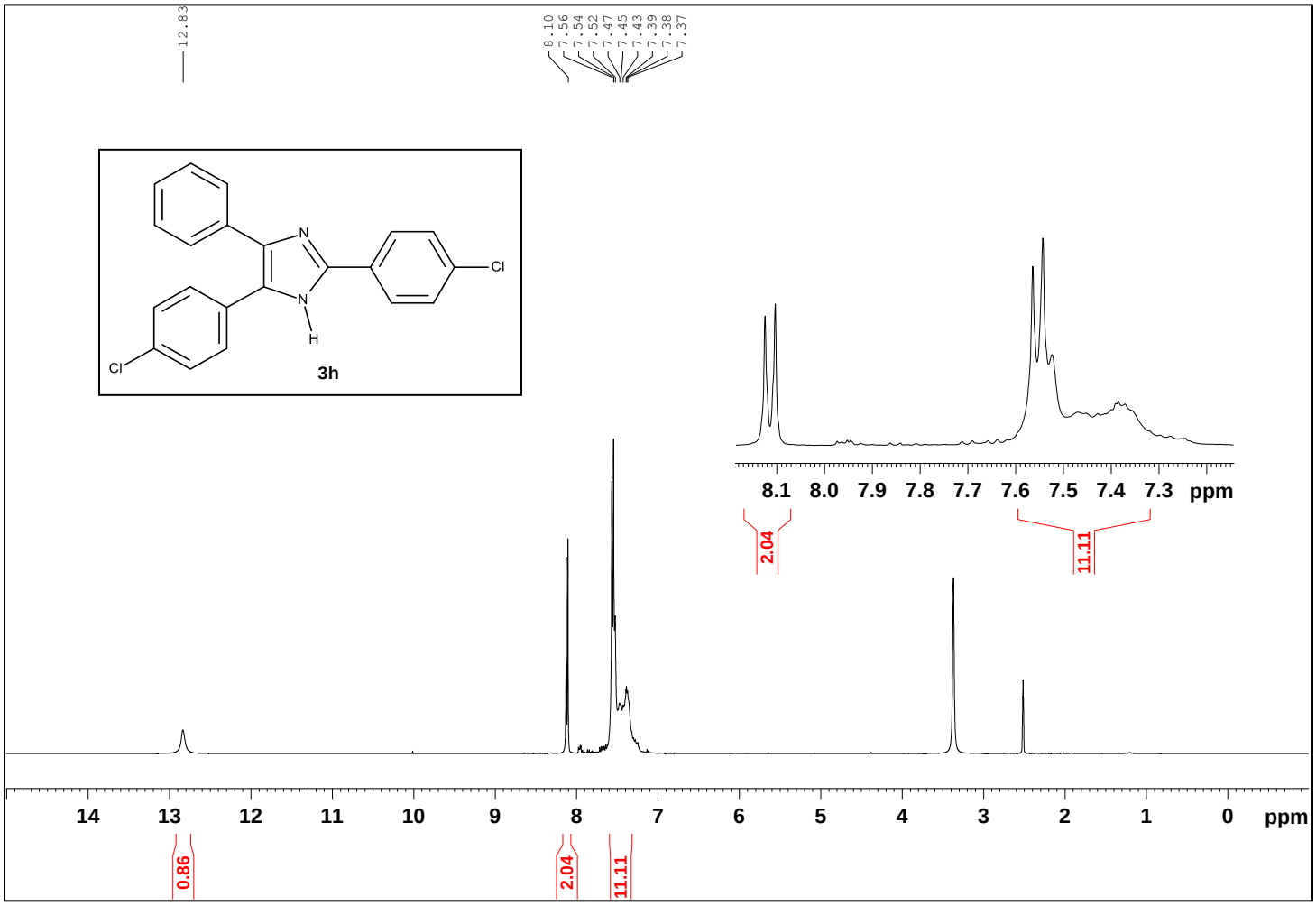
130.70
130.47
130.34
129.24
129.16
129.04
128.84
128.77
128.72
128.49
127.76
127.41
127.22
125.73
N
N
H
Cl
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.56
7.54
7.52
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7.43
7.39
7.38
7.37
N
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H
Cl
Cl
3h
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 ppm
2.04
11.11
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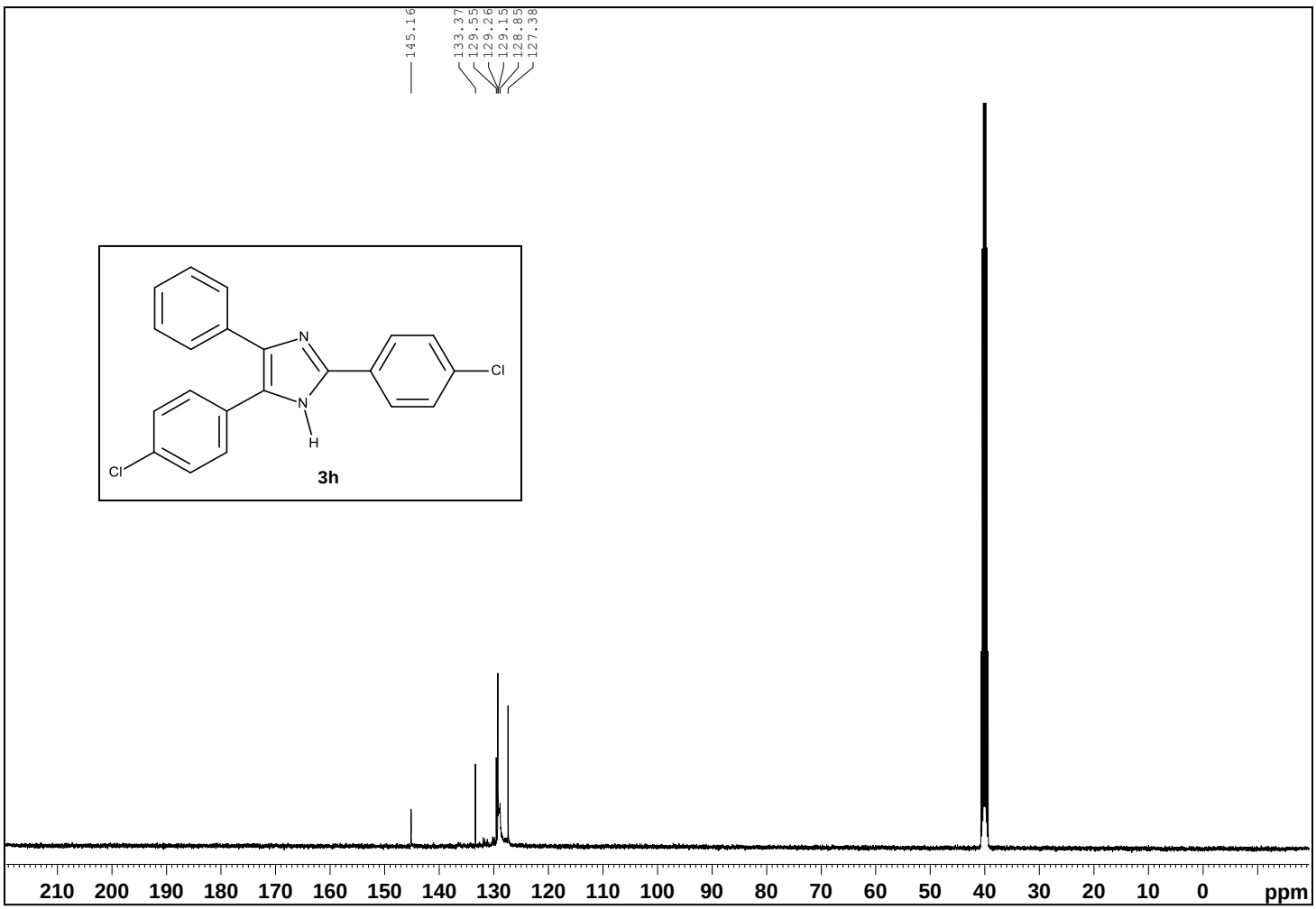
2.04
11.11

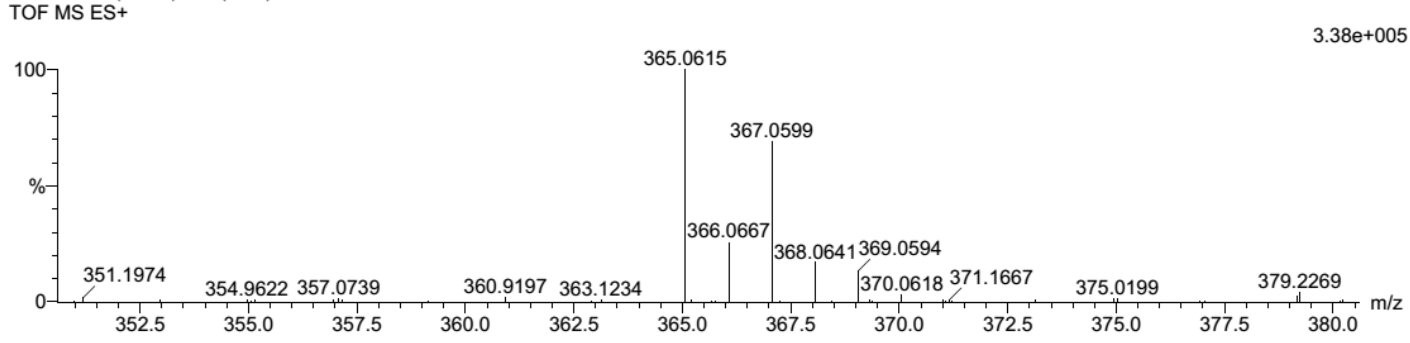

145.16
133.37
129.55
129.26
129.15
128.85
127.38
N
N
H
Cl
Cl
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Elemental Composition Report

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
6 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 20-25 H: 10-15 N: 0-5 Cl: 0-2
217rxn22 9 (0.269) Cm (1:61)
TOF MS ES+



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Maximum:		5.0	5.0	100.0			
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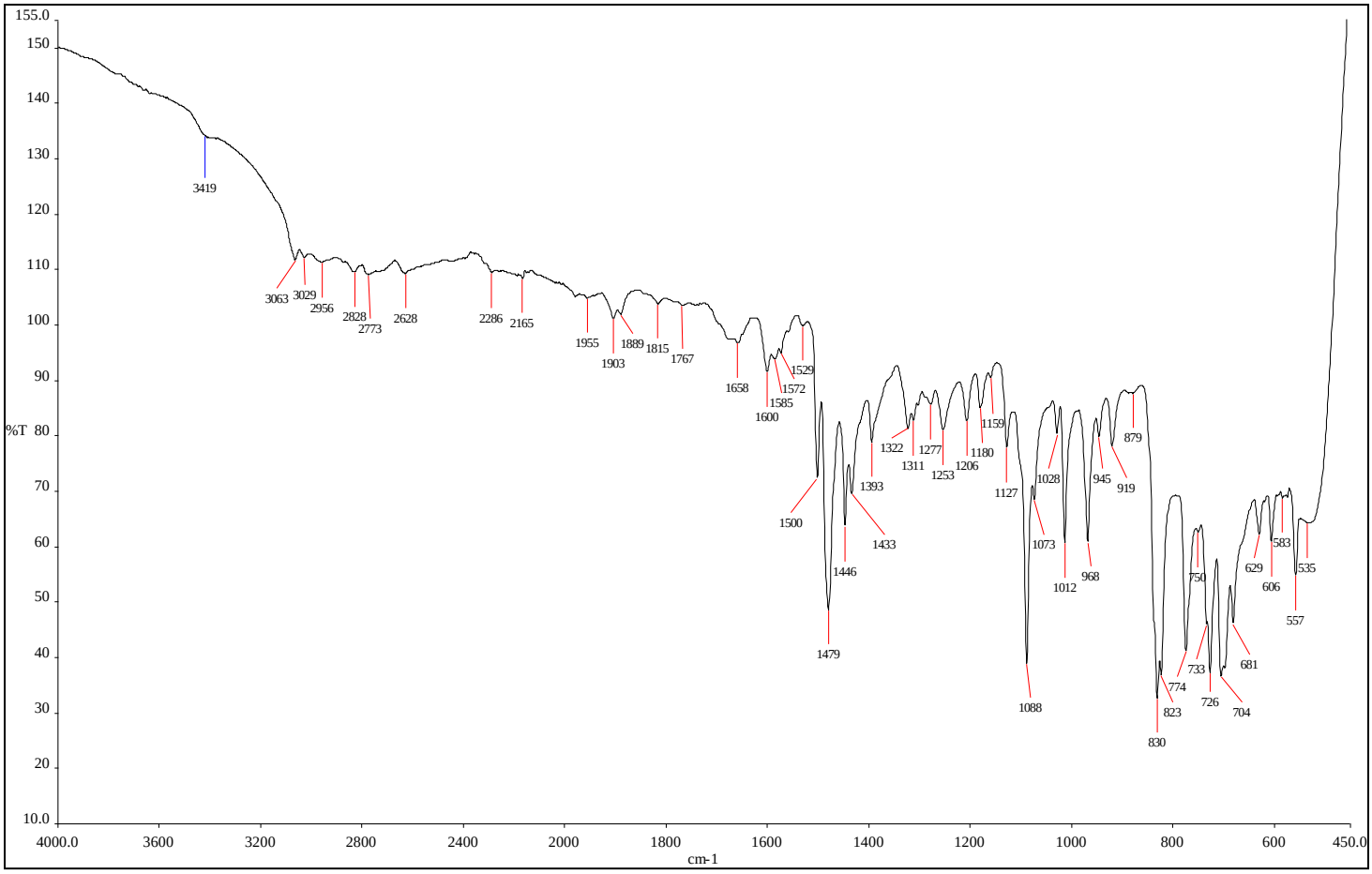

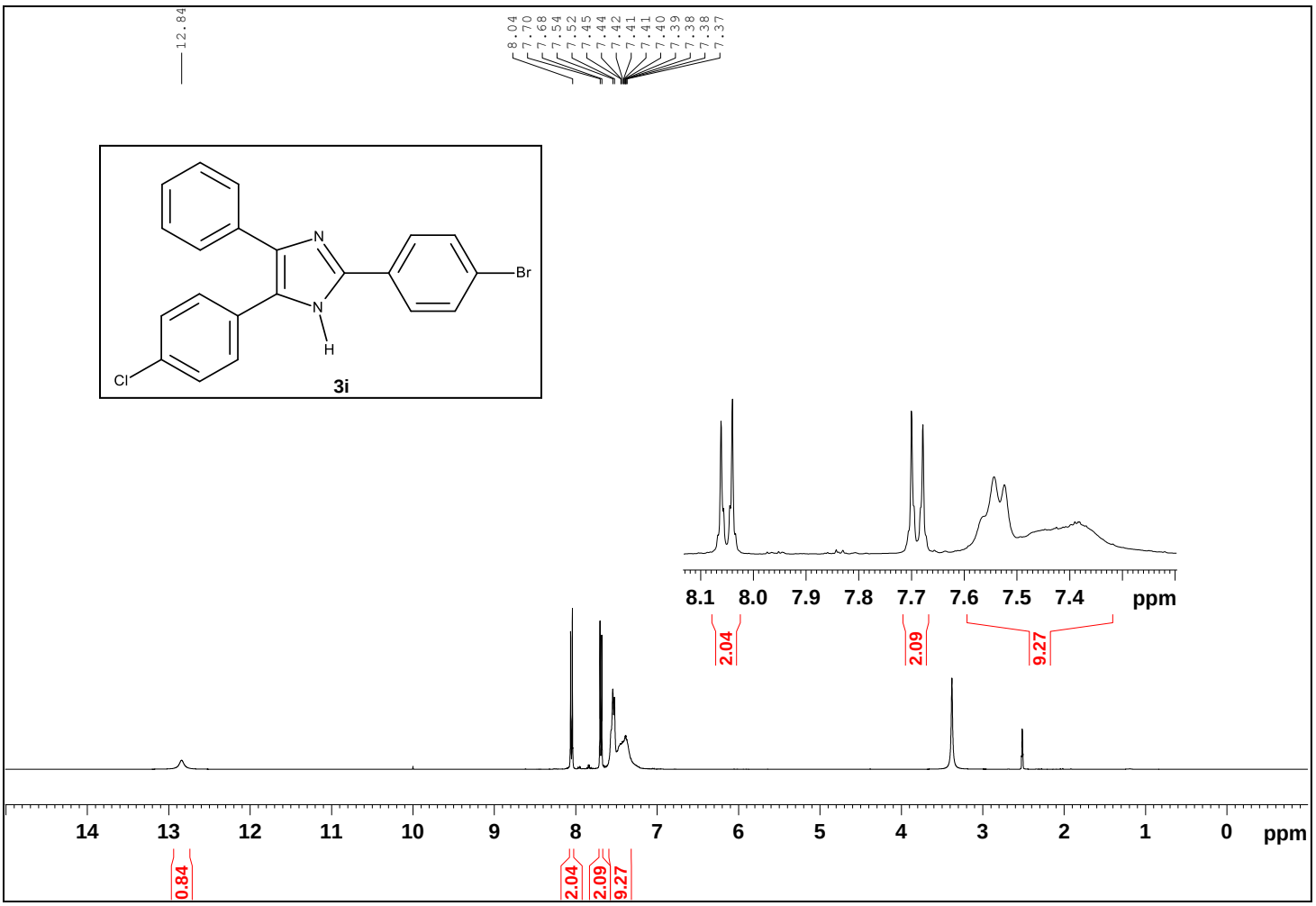
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7.44
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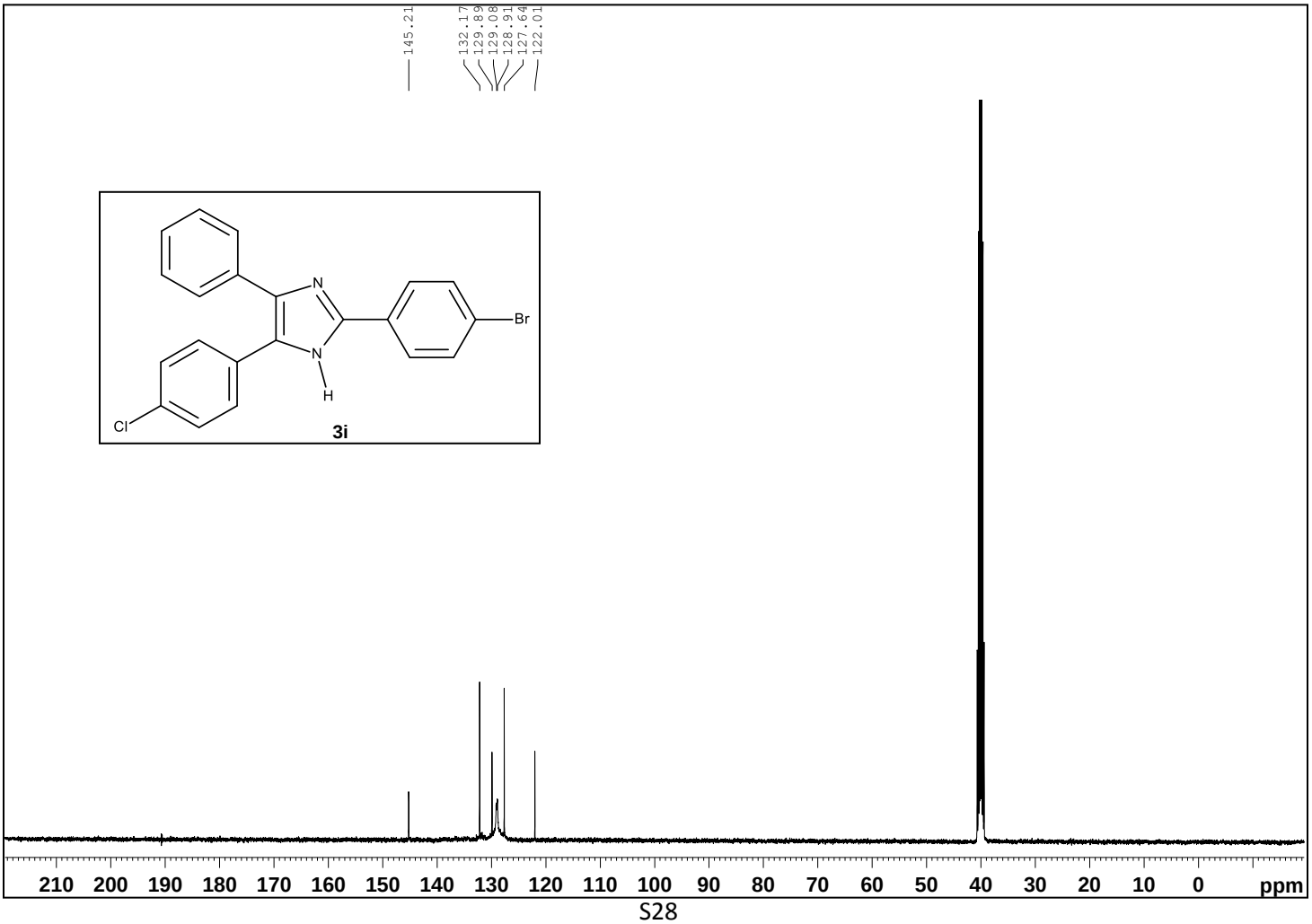
7.41
7.40
7.39
7.38
7.38
7.37
N
Br
N
H
Cl
3i
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
ppm
2.04
2.09
9.27
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.84
2.04
2.09
9.27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127.64
122.01
N
Br
N
H
Cl
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

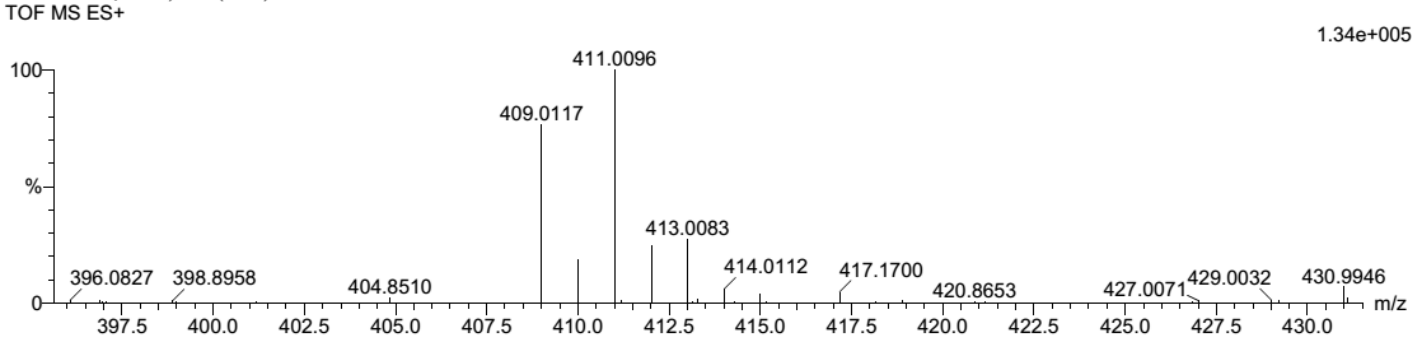
Elemental Composition Report

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
10 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 20-25 H: 10-15 N: 0-5 Cl: 0-1 Br: 0-1

217rxn23 4 (0.101) Cm (1:61)
TOF MS ES+



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Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
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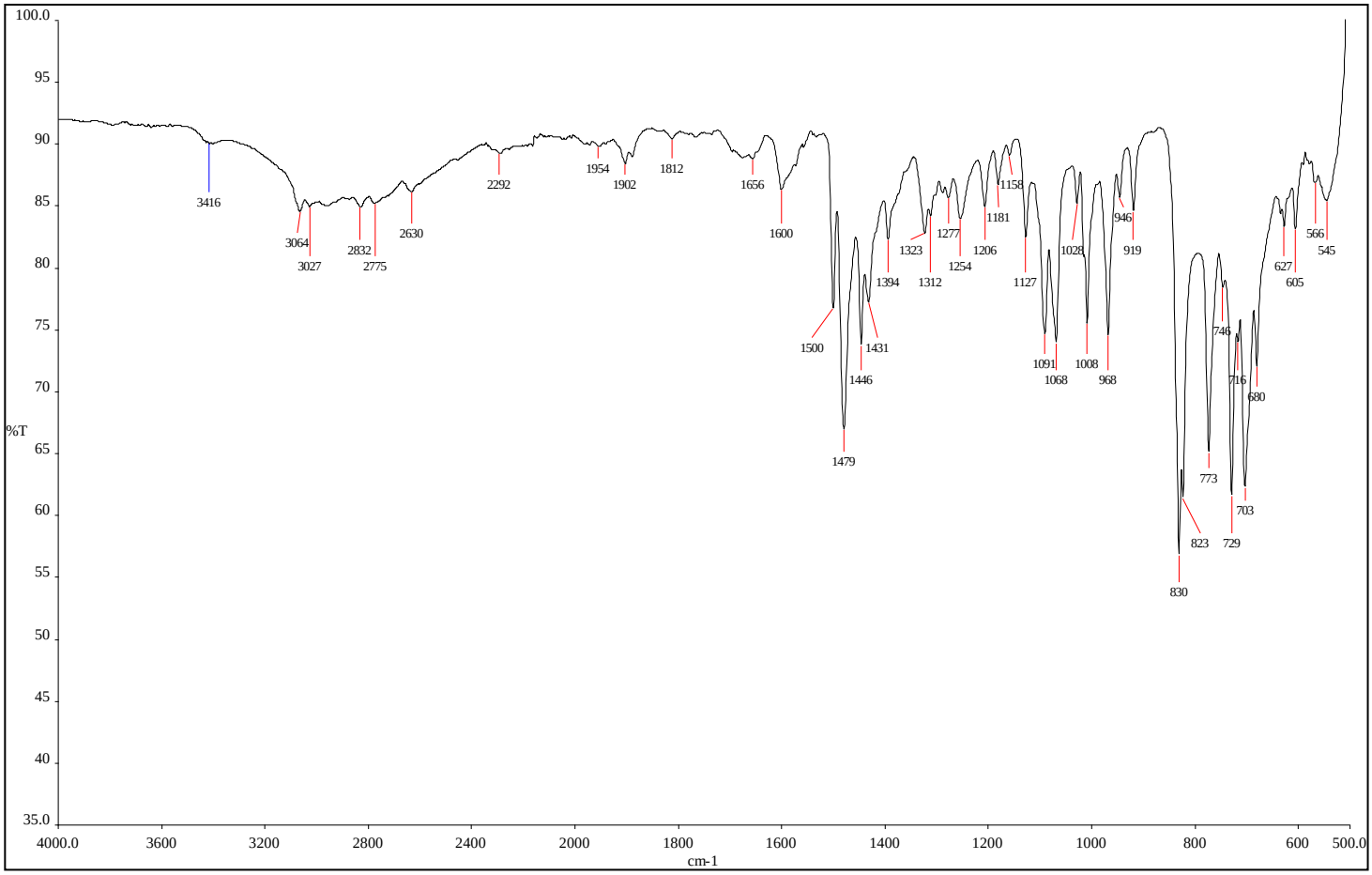

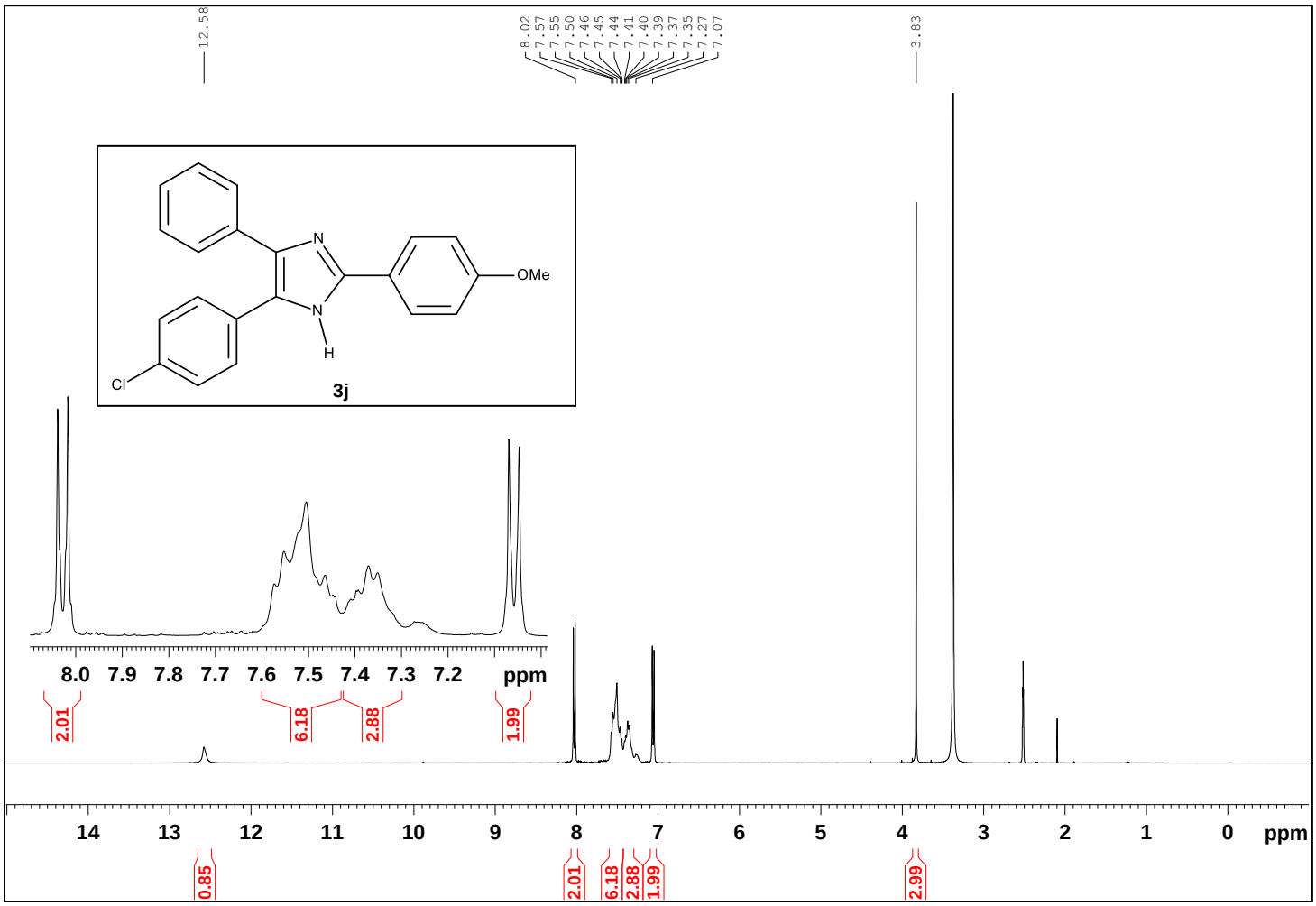
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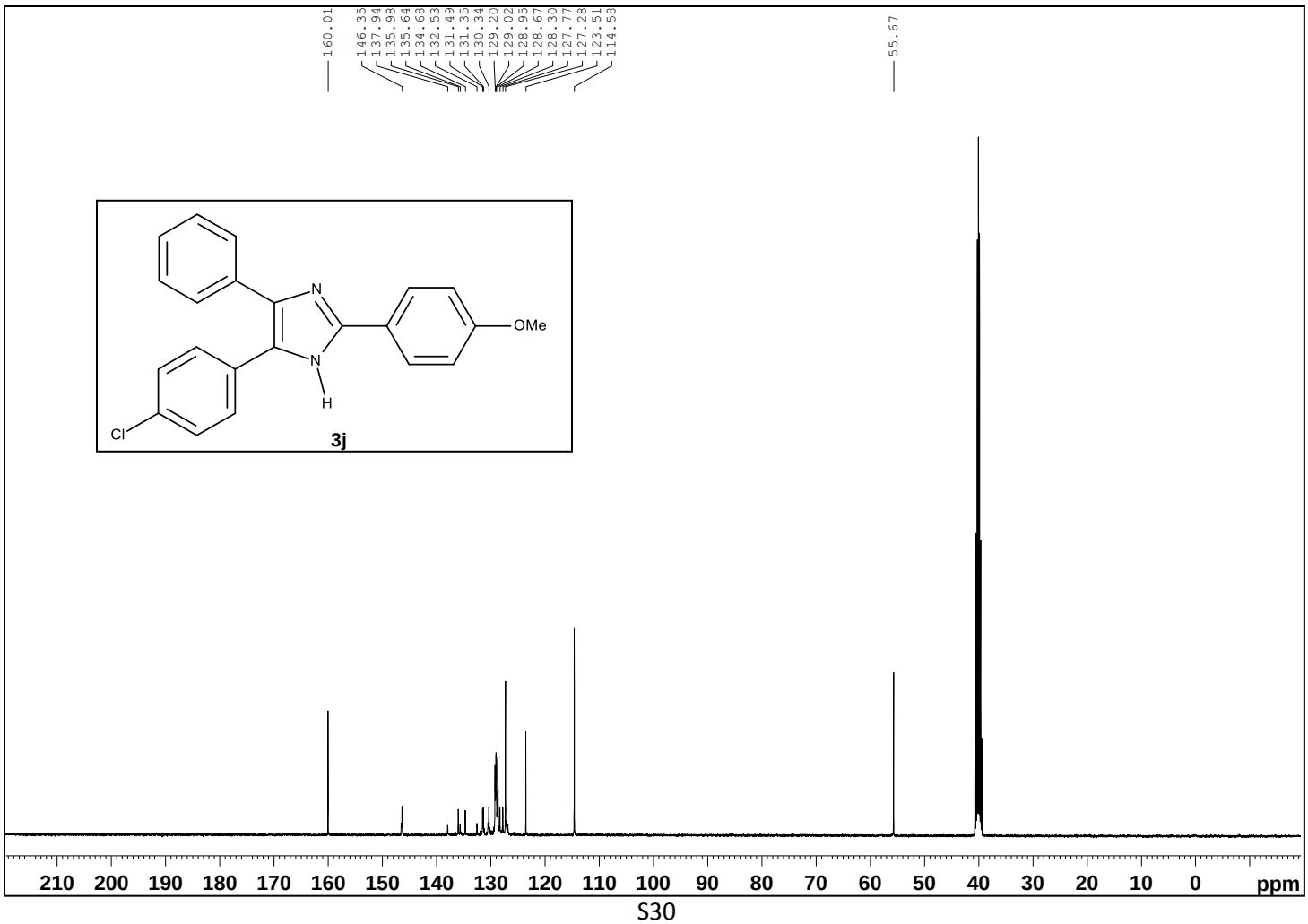
7.44
7.41
7.40
7.39
7.37
7.35
7.27
7.07
3.83
OMe
N
N
H
Cl
3j
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
2.01
6.18
2.88
1.99
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.85
2.01
6.18
2.88
1.99
2.99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128.95
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127.77
127.28
123.51
114.58
55.67
OMe
N
N
H
Cl
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

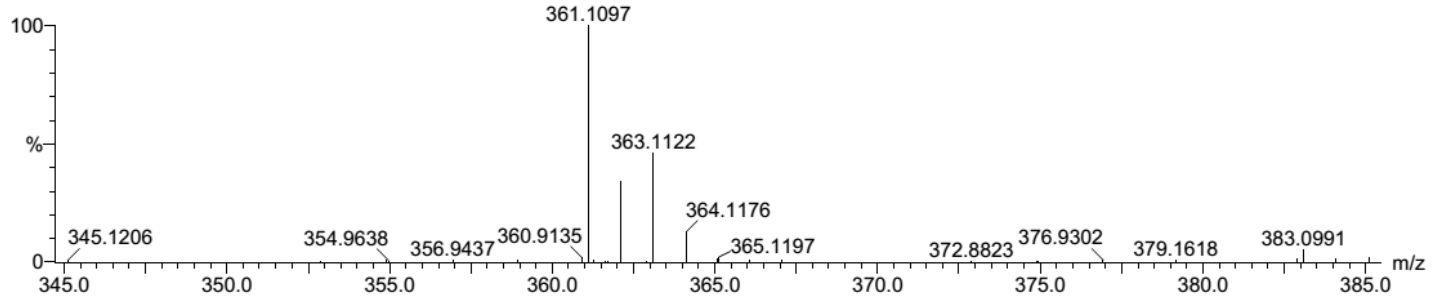
Elemental Composition Report

Single Mass Analysis

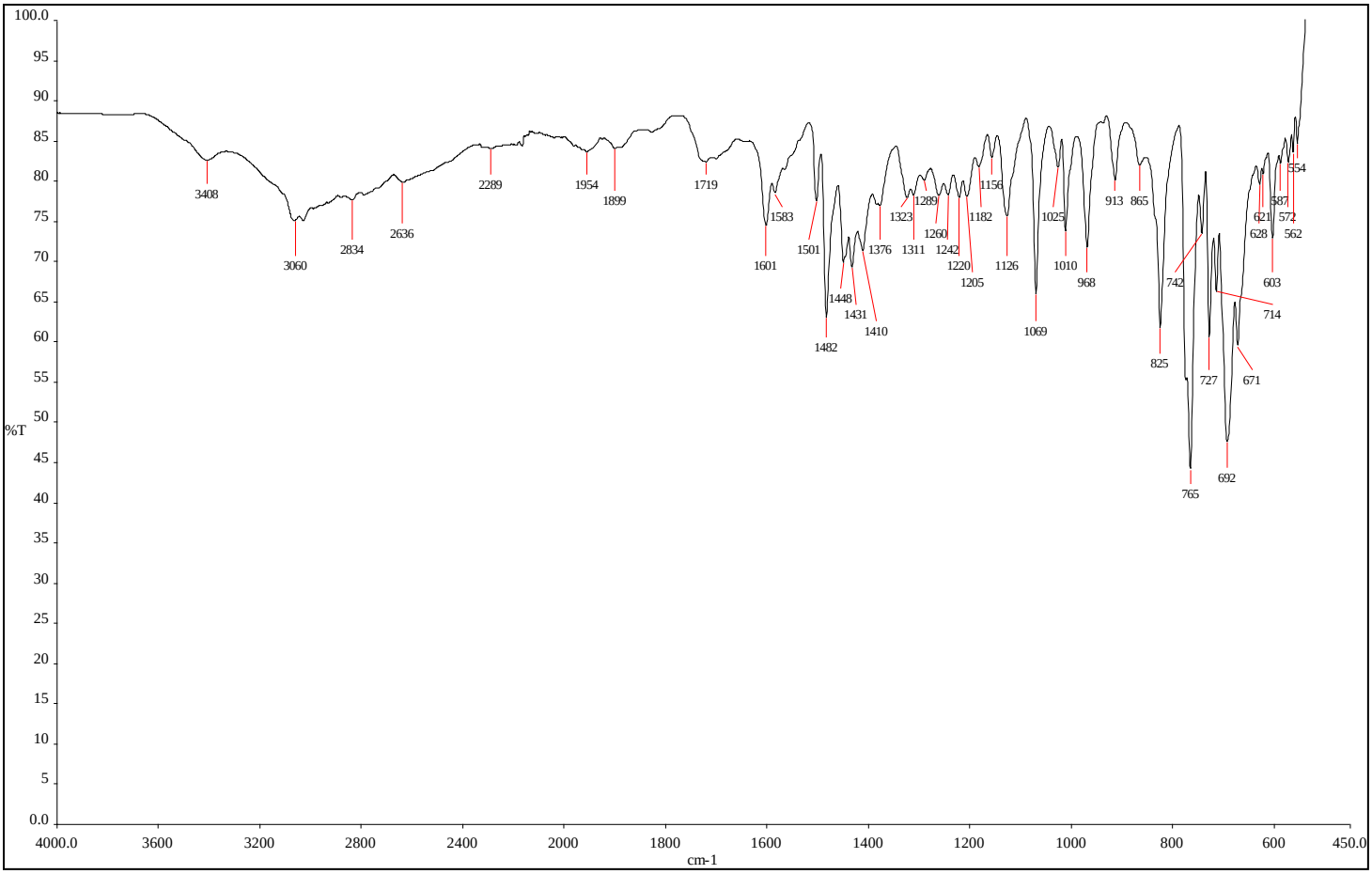
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

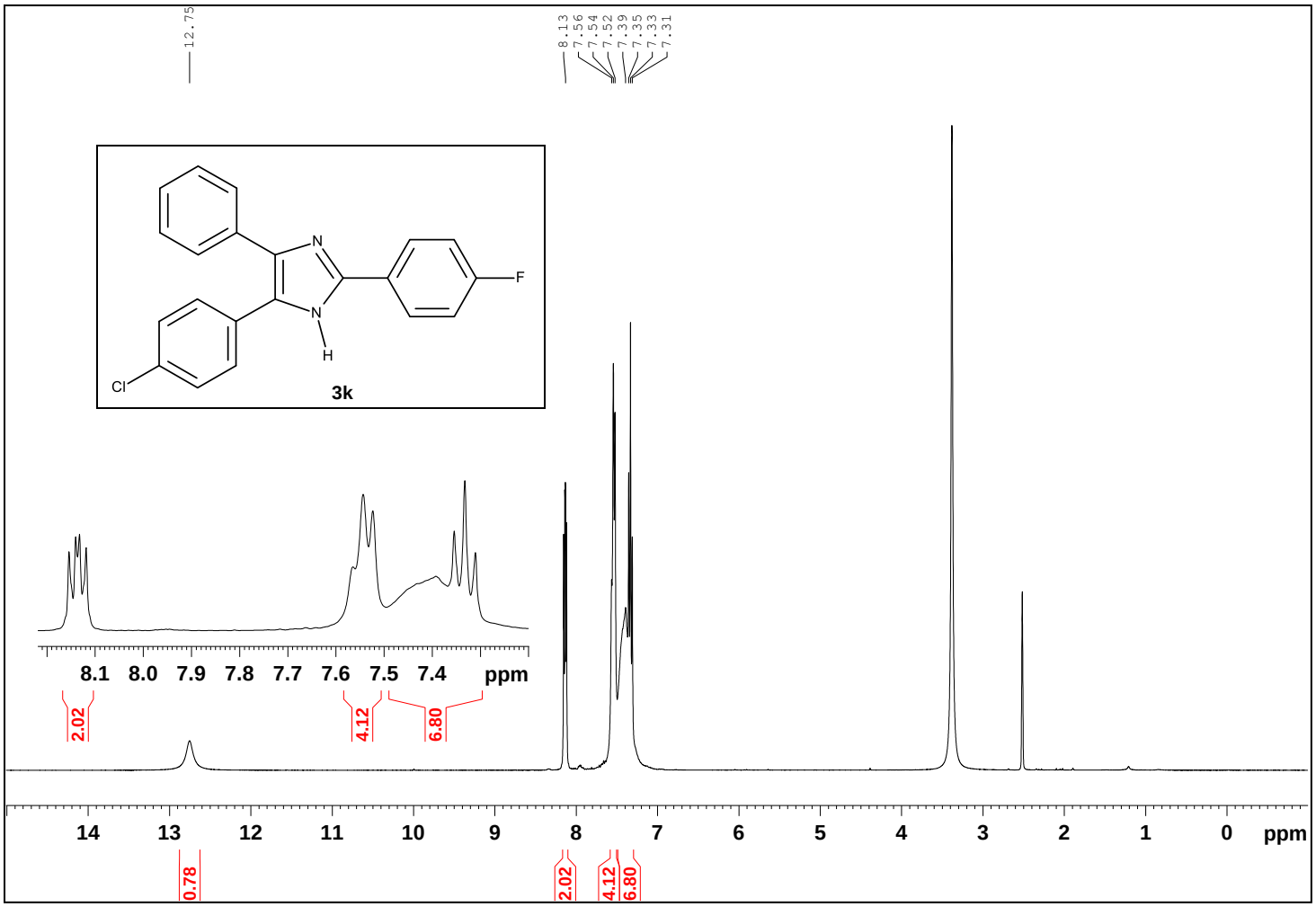
Monoisotopic Mass, Even Electron Ions
29 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 20-25 H: 10-20 N: 0-5 O: 0-5 Cl: 1-1
217rxn37 29 (0.944) Cm (1:61)
TOF MS ES+

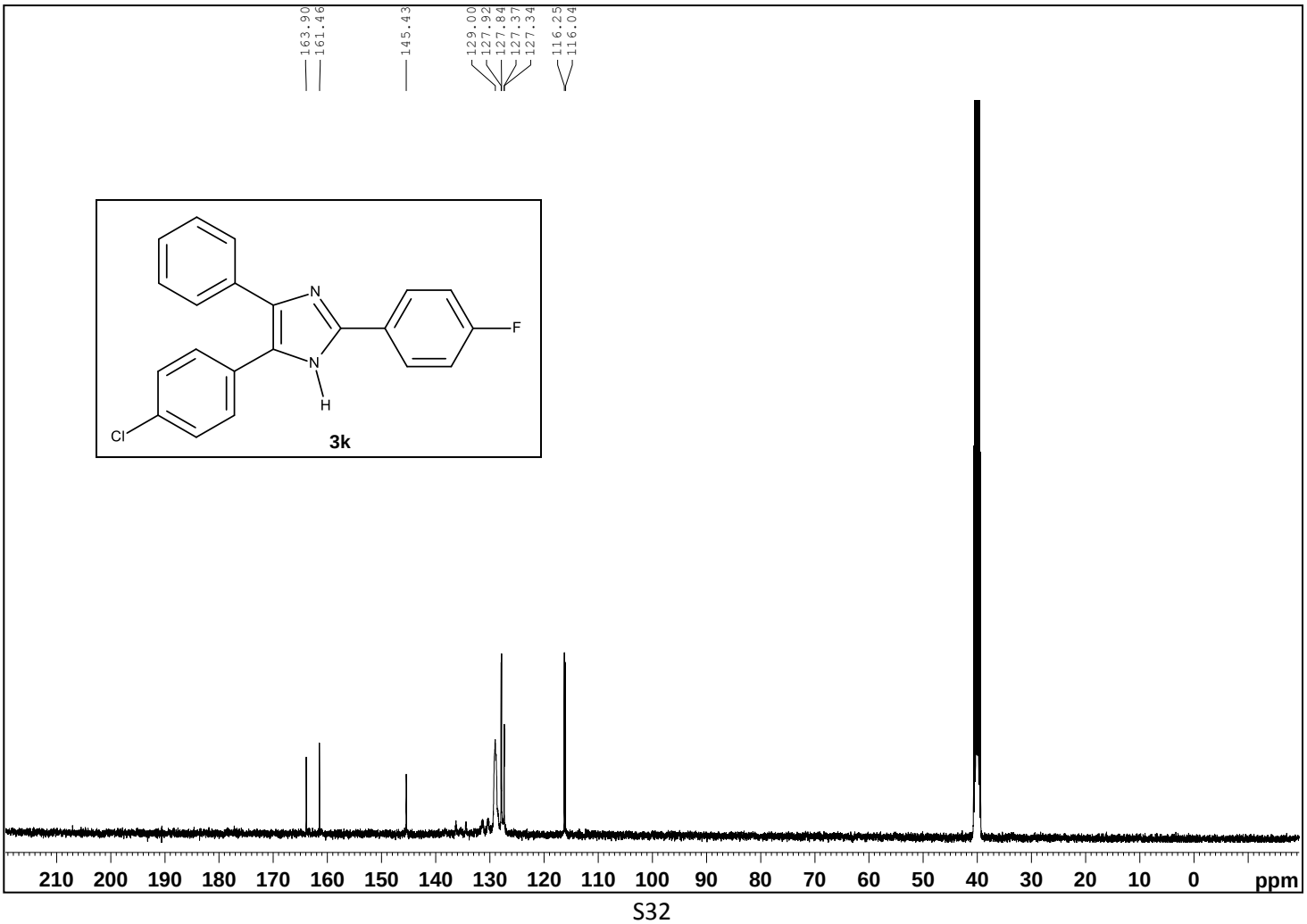
6.76e+005



Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
361.1097	361.1108	-1.1	-3.0	14.5	77.2	0.0	C22 H18 N2 O Cl




12.75
8.13
7.56
7.54
7.52
7.39
7.35
7.33
7.31
N
N
H
F
Cl
3k
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 ppm
2.02
4.12
6.80
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.78
2.02
4.12
6.80

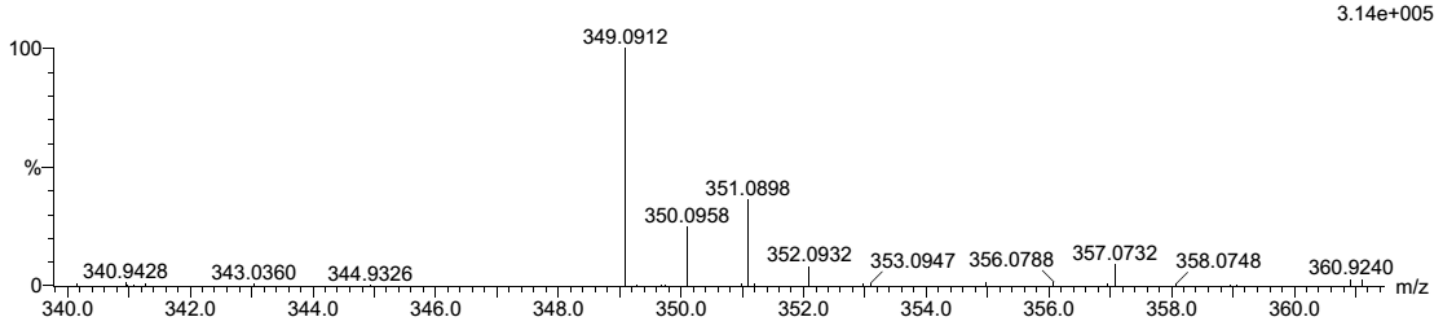

163.90
161.46
145.43
129.00
127.92
127.84
127.37
127.34
116.25
116.04
N
N
H
F
Cl
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Elemental Composition Report

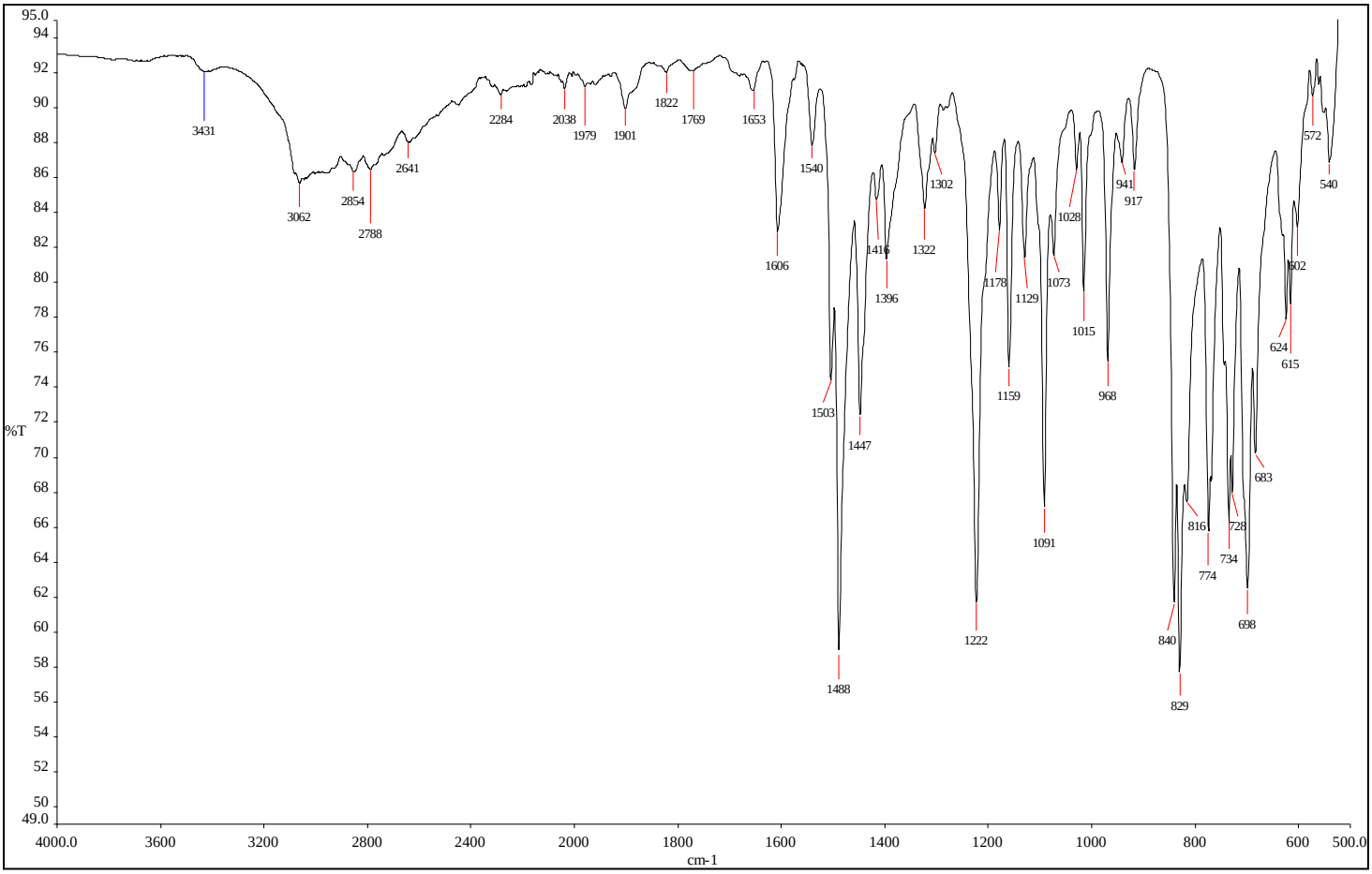
Single Mass Analysis

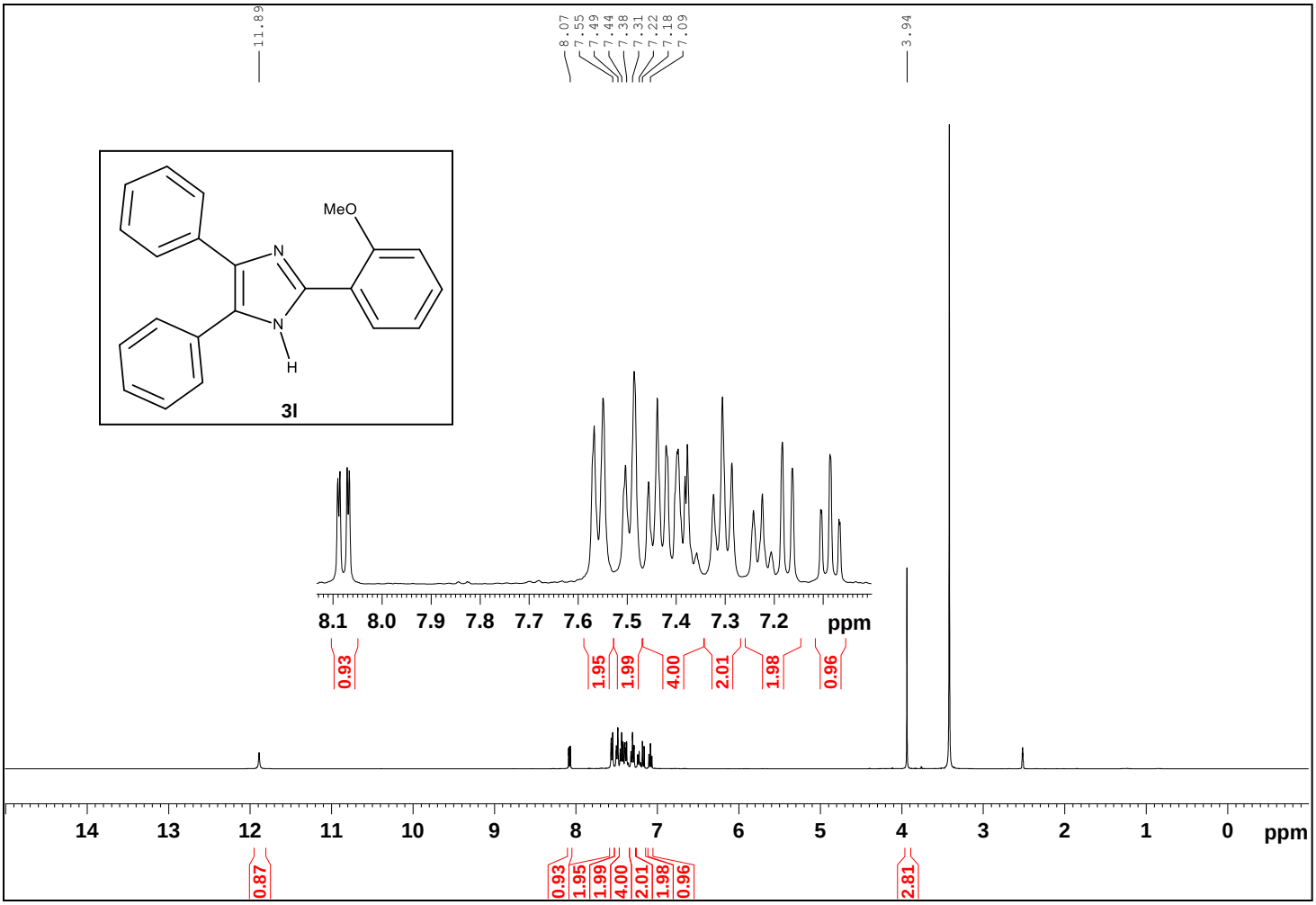
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

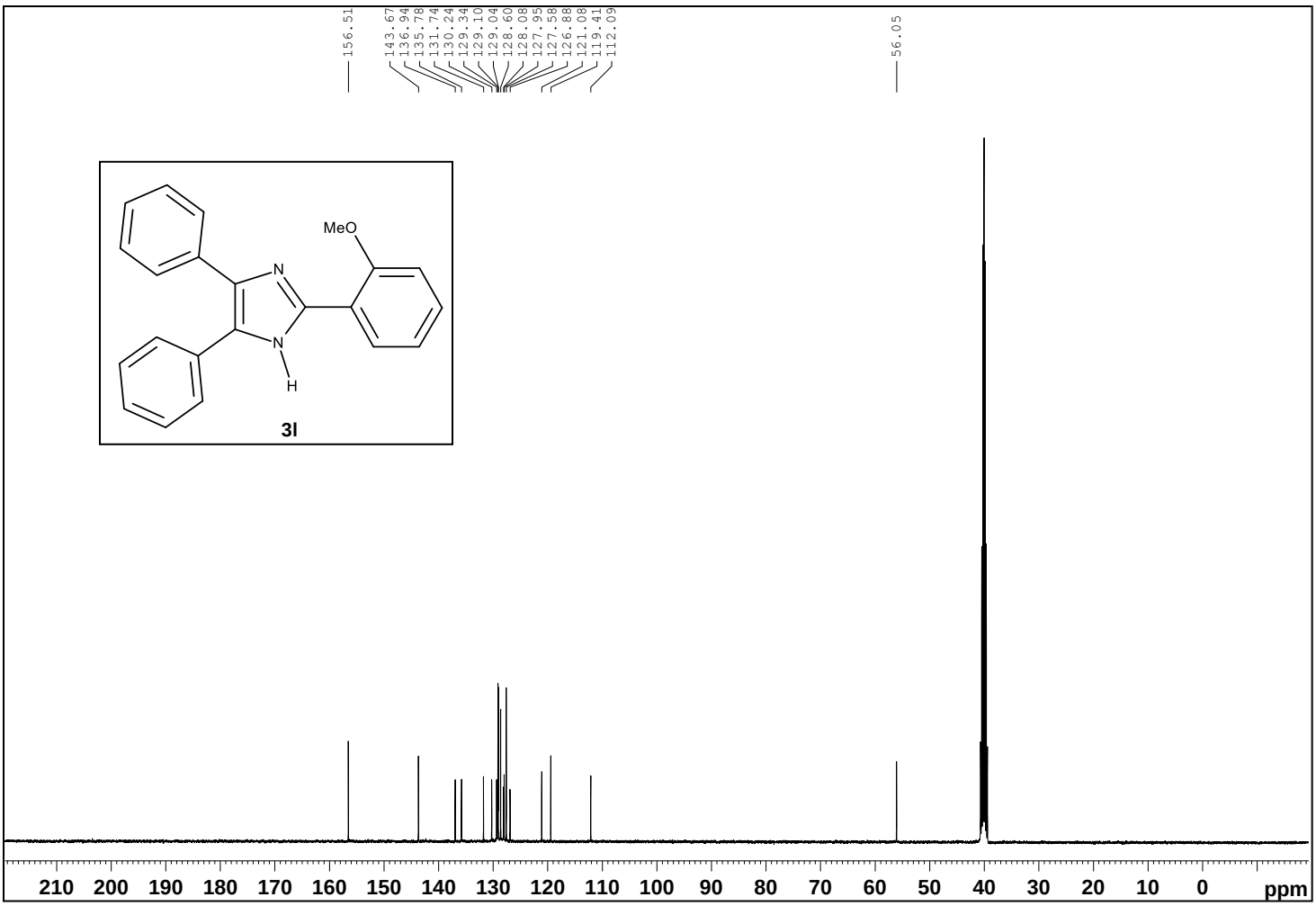
Monoisotopic Mass, Even Electron Ions
10 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)
Elements Used:
C: 20-25 H: 10-15 N: 0-5 F: 0-1 Cl: 0-1
217rxn36 4 (0.135) Cm (1:60)
TOF MS ES+

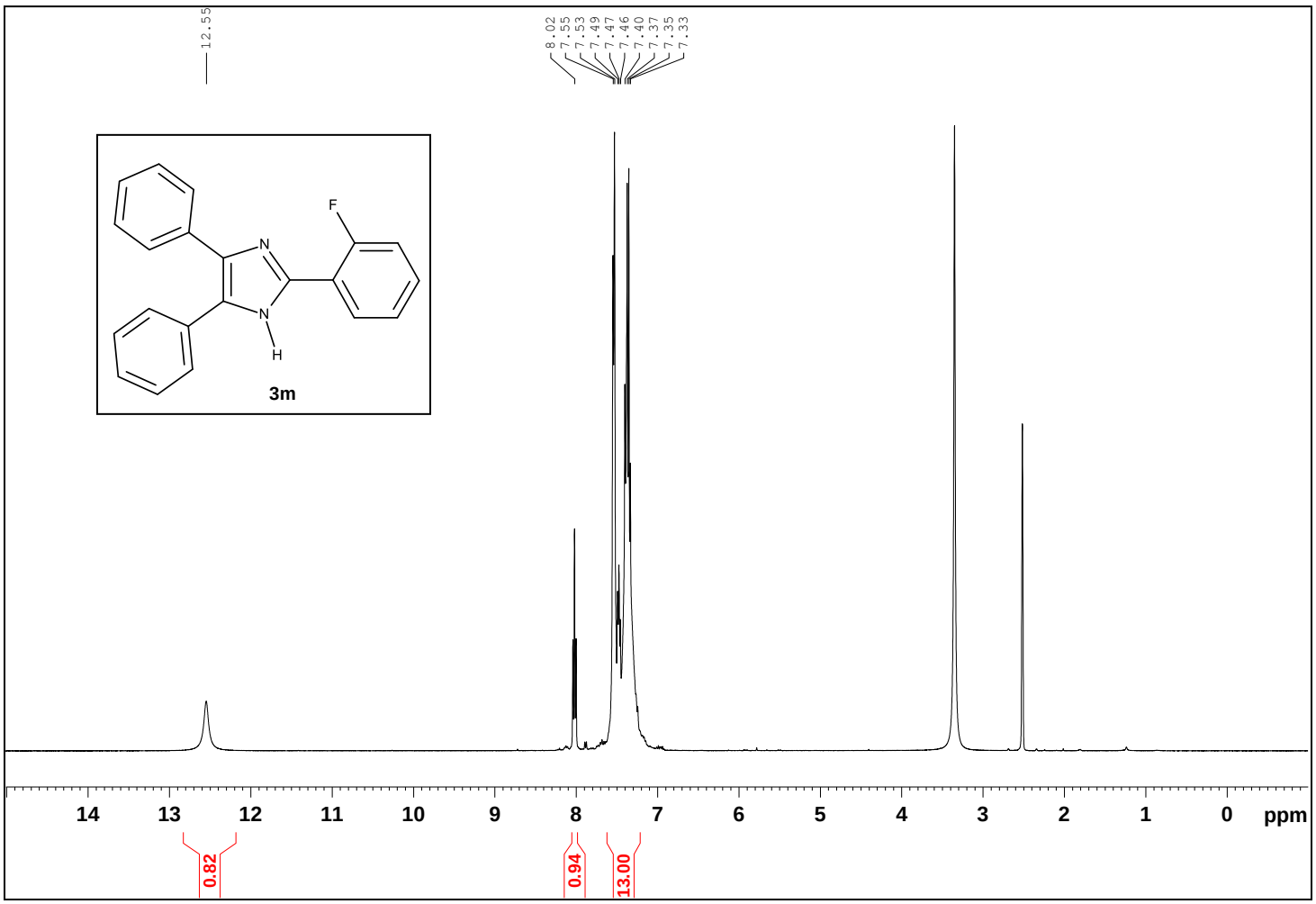


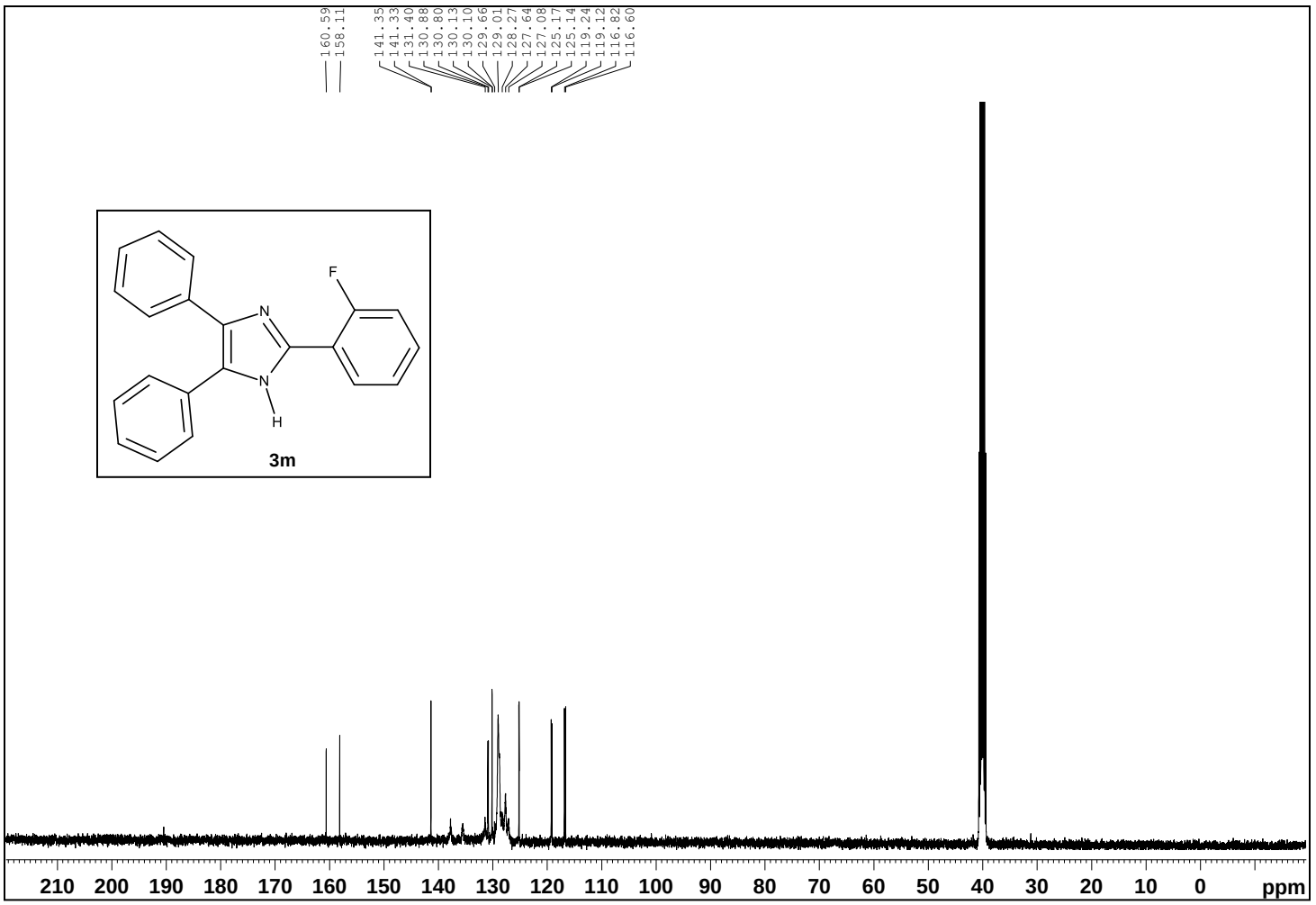
Minimum:				-1.5			
Maximum:		5.0	5.0	100.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
349.0912	349.0908	0.4	1.1	14.5	57.6	0.0	C21 H15 N2 F Cl

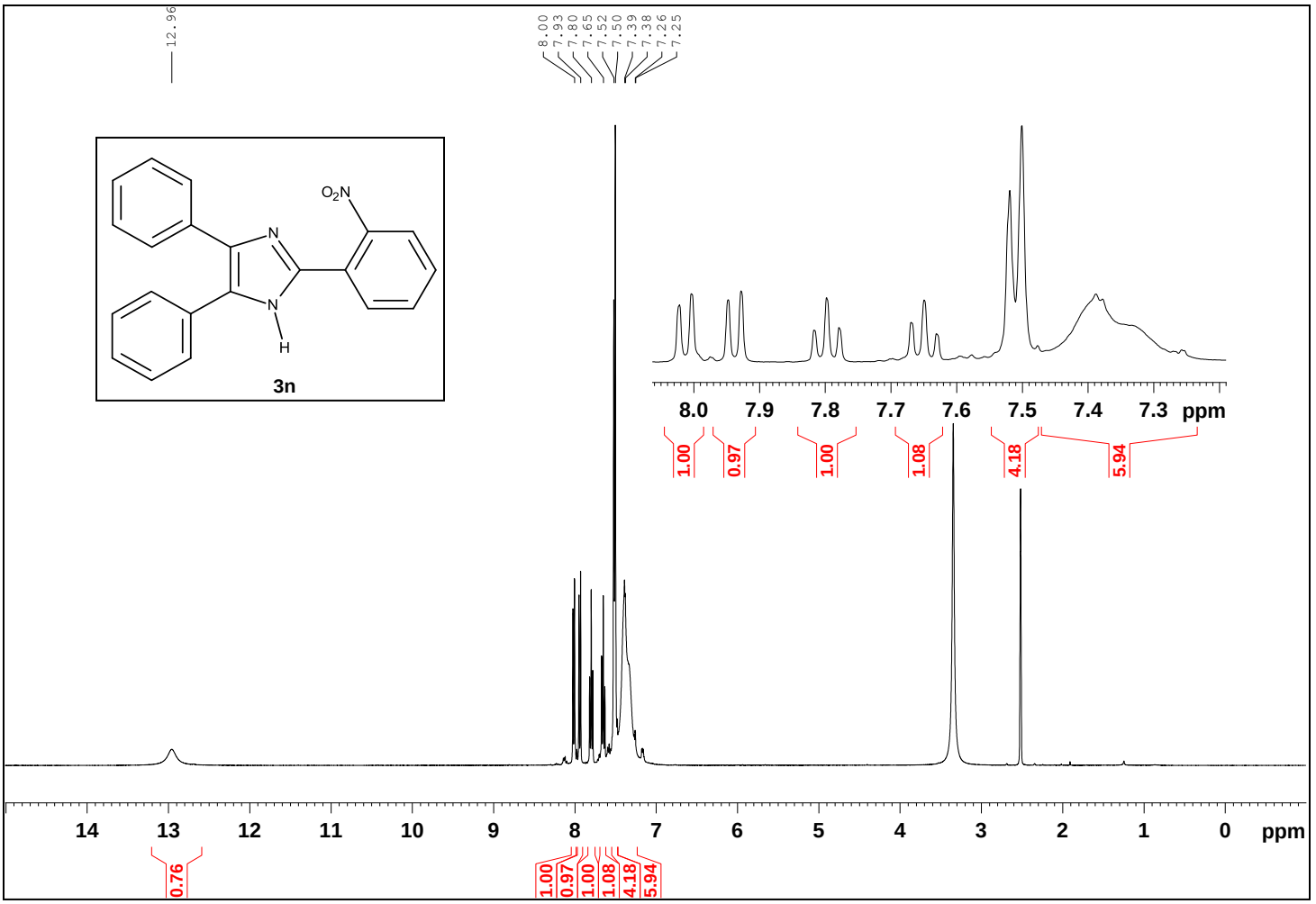


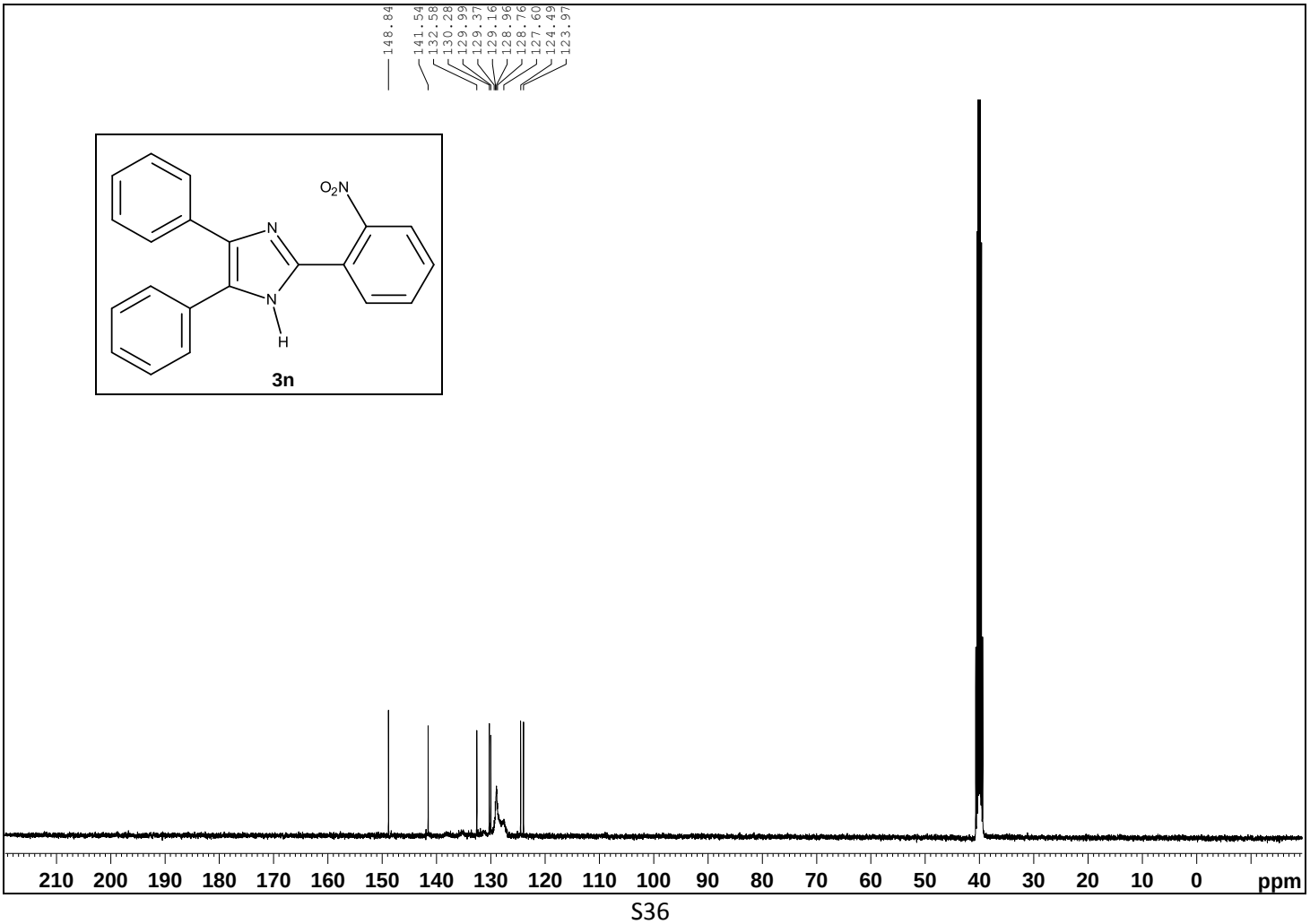

11.89
8.07
7.55
7.49
7.44
7.38
7.31
7.22
7.18
7.09
3.94
MeO
N
N
H
3l
8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
0.93
1.95
1.99
4.00
2.01
1.98
0.96
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.87
0.93
1.95
1.99
4.00
2.01
1.98
0.96
2.81

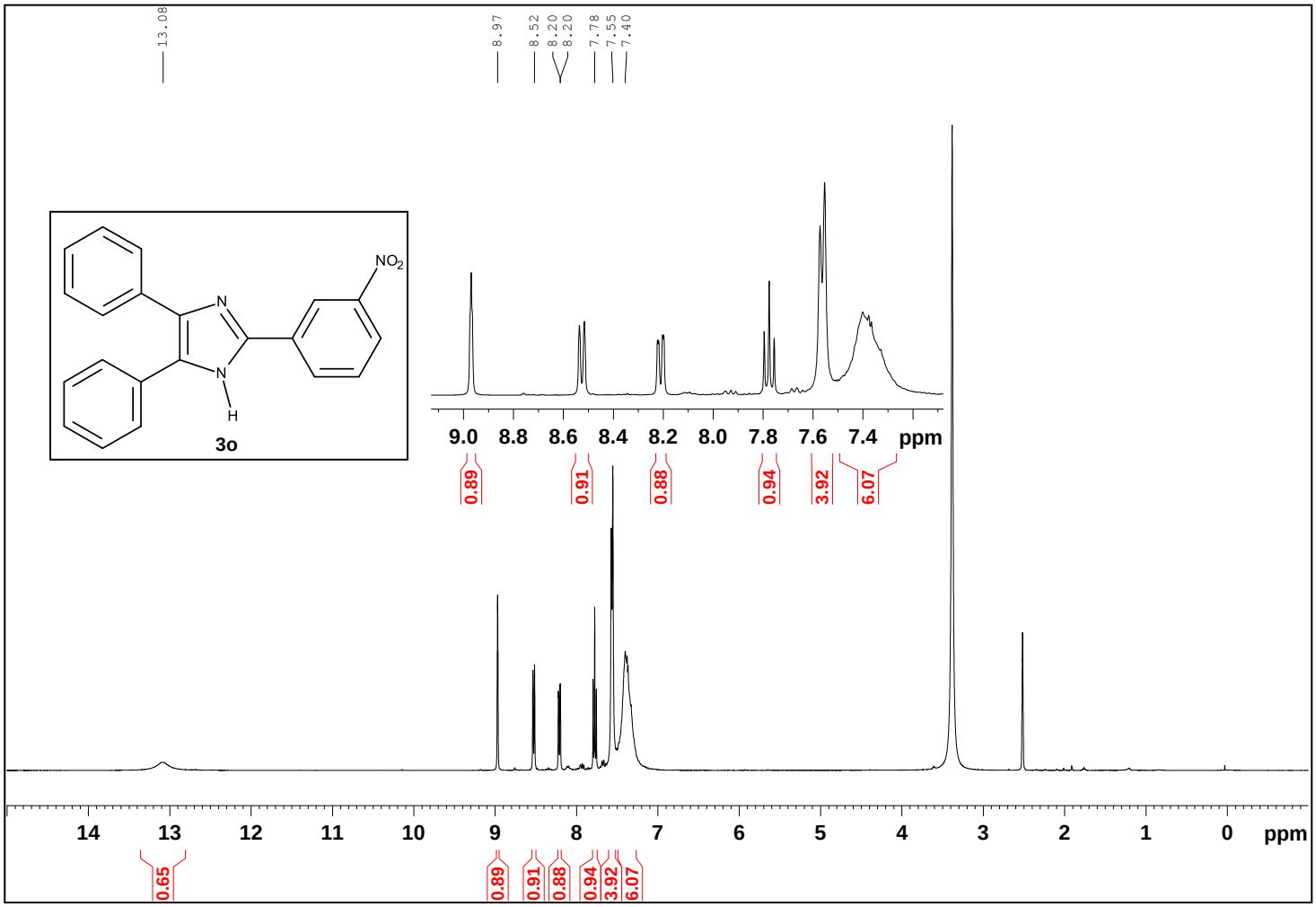

156.51
143.67
136.94
135.78
131.74
130.24
129.34
129.10
129.04
128.60
128.08
127.95
127.58
126.88
121.08
119.41
112.09
56.05
MeO
N
N
H
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

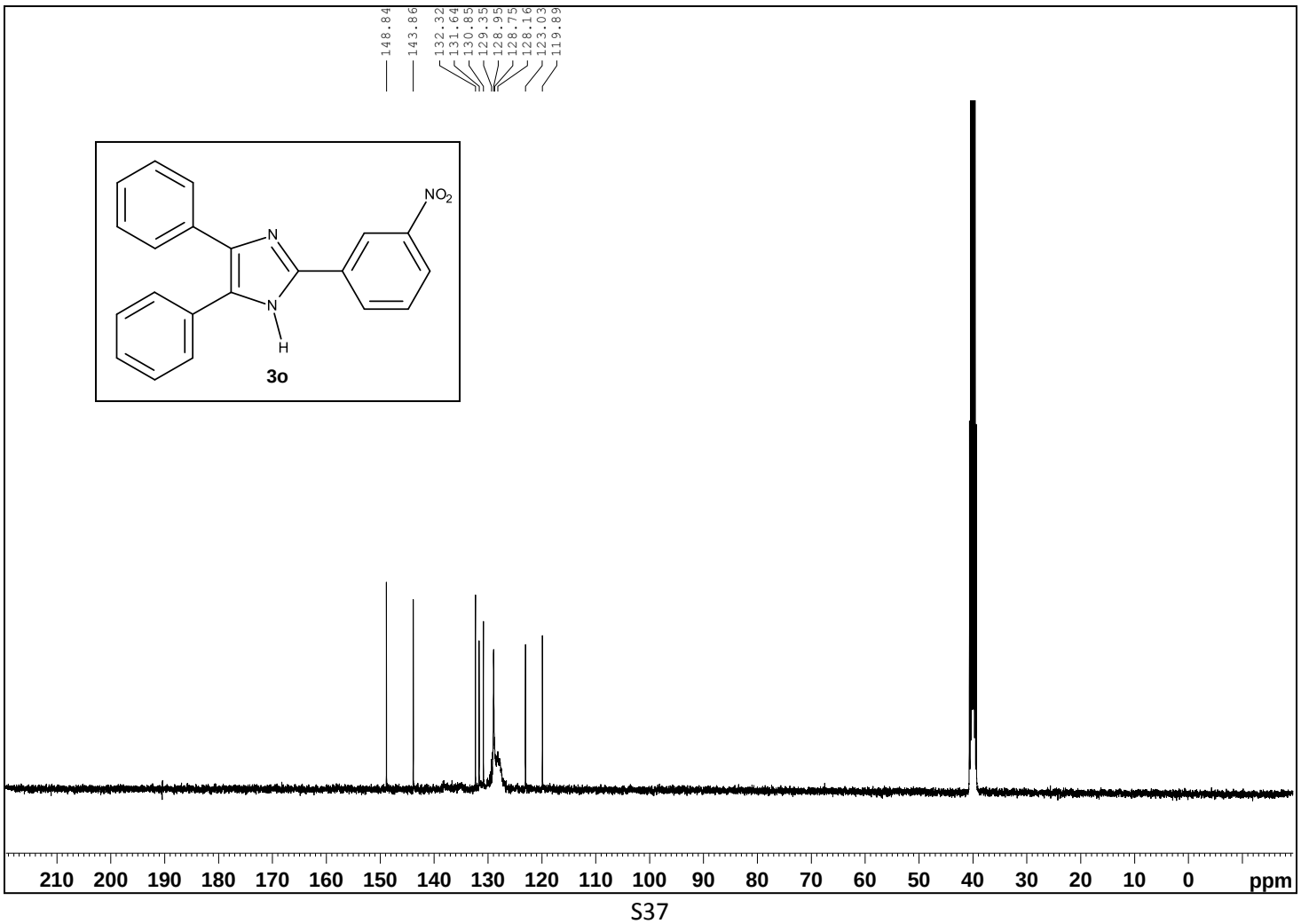

12.55
8.02
7.55
7.53
7.49
7.47
7.46
7.40
7.37
7.35
7.33
F
N
N
H
3m
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.82
0.94
13.00

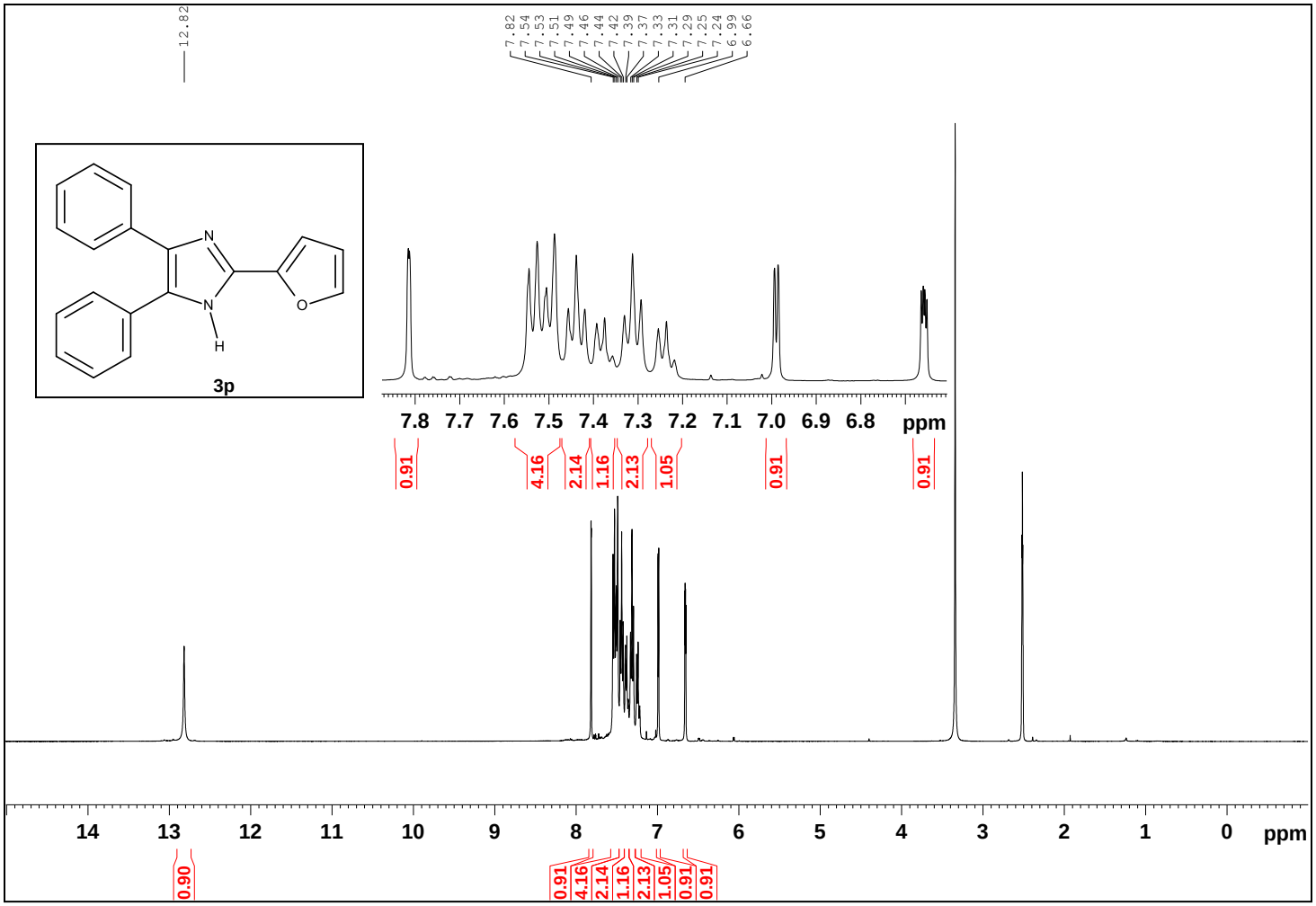

160.59
158.11
141.35
141.33
131.40
130.88
130.80
130.13
130.10
129.66
129.01
128.27
127.64
127.08
125.17
125.14
119.24
119.12
116.82
116.60
F
N
N
H
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

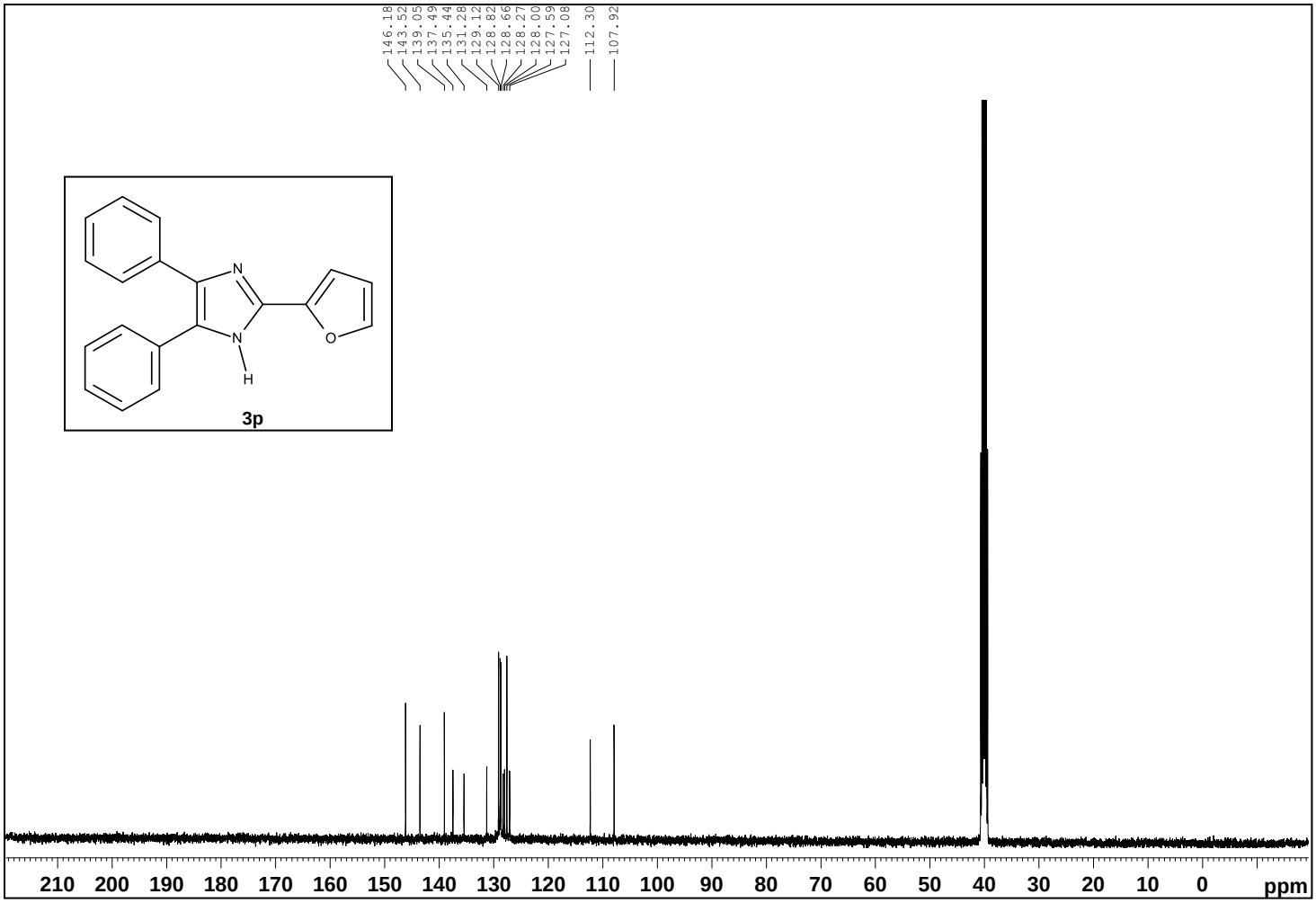

12.96
8.00
7.93
7.80
7.65
7.52
7.50
7.39
7.38
7.26
7.25
O2N
N
N
H
3n
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
ppm
1.00
0.97
1.00
1.08
4.18
5.94
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.76
1.00
0.97
1.00
1.08
4.18
5.94

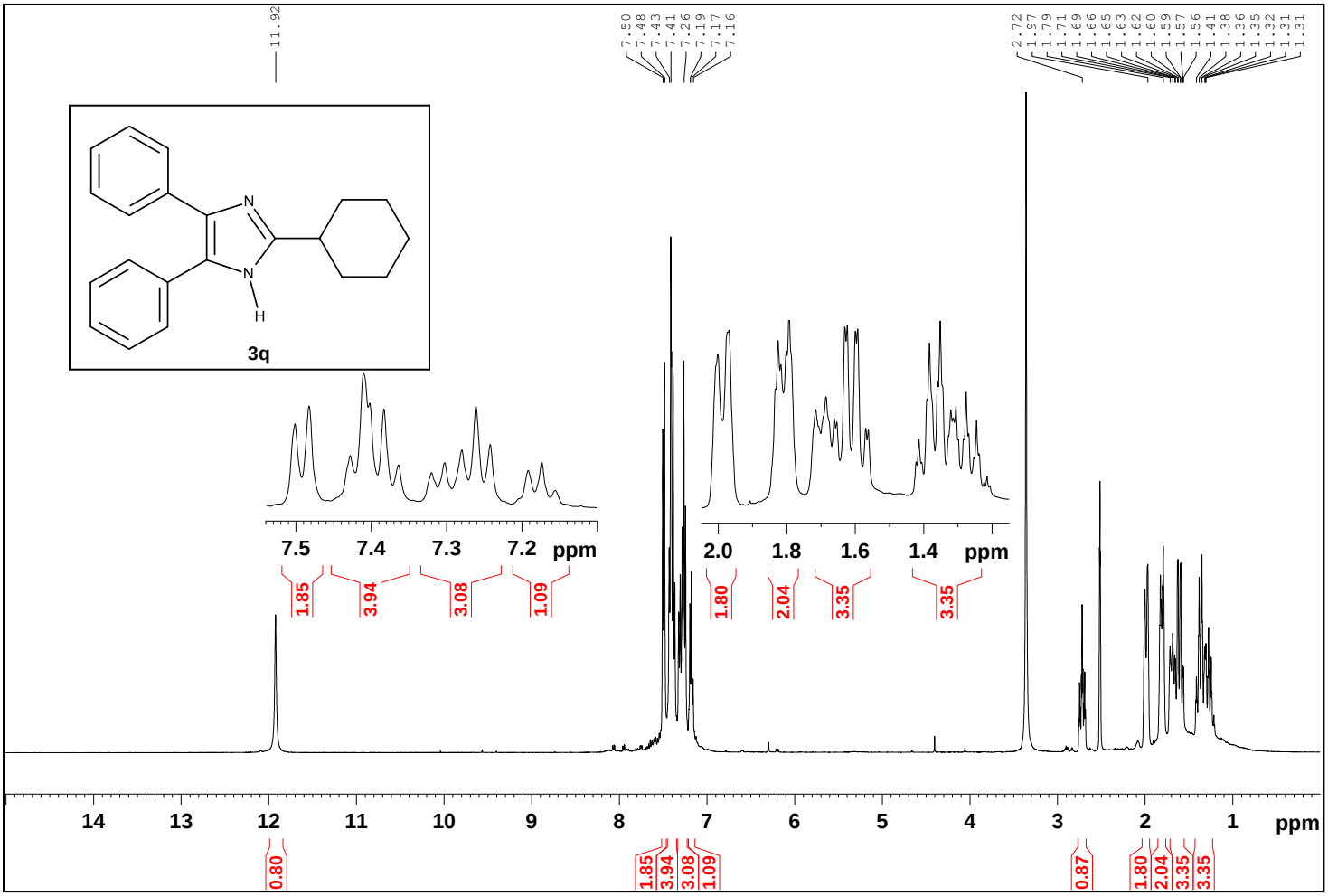

148.84
141.54
132.58
130.28
129.99
129.37
129.16
128.96
128.76
127.60
124.49
123.97
O2N
N
N
H
3n
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

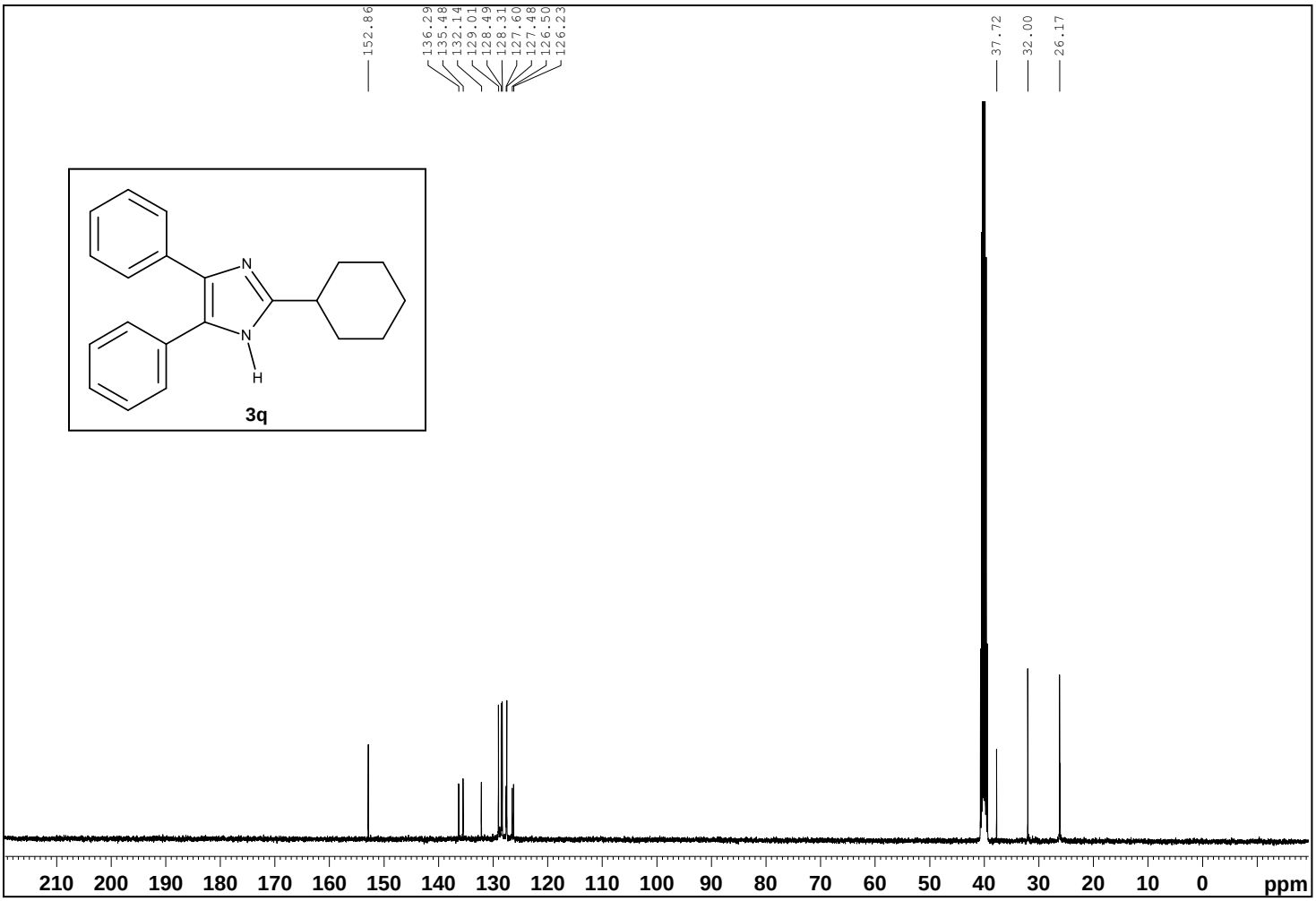

13.08
8.97
8.52
8.20
8.20
7.78
7.55
7.40
NO2
N
N
H
3o
9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 ppm
0.89
0.91
0.88
0.94
3.92
6.07
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.65
0.89
0.91
0.88
0.94
3.92
6.07


148.84
143.86
132.32
131.64
130.85
129.35
128.95
128.75
128.16
123.03
119.89
NO2
N
N
H
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


12.82
7.82
7.54
7.53
7.51
7.49
7.46
7.44
7.42
7.39
7.37
7.33
7.31
7.29
7.25
7.24
6.99
6.66
3p
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
0.91
4.16
2.14
1.16
2.13
1.05
0.91
0.91
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.90
0.91
4.16
2.14
1.16
2.13
1.05
0.91
0.91


146.18
143.52
139.05
137.49
135.44
131.28
129.12
128.82
128.66
128.27
128.00
127.59
127.08
112.30
107.92
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


11.92
7.50
7.48
7.43
7.41
7.26
7.19
7.17
7.16
2.72
1.97
1.79
1.71
1.69
1.66
1.65
1.63
1.62
1.60
1.59
1.57
1.56
1.41
1.38
1.36
1.35
1.32
1.31
1.31
N
N
H
3q
7.5
7.4
7.3
7.2
ppm
1.85
3.94
3.08
1.09
2.0
1.8
1.6
1.4
ppm
1.80
2.04
3.35
3.35
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.80
1.85
3.94
3.08
1.09
0.87
1.80
2.04
3.35
3.35


152.86
136.29
135.48
132.14
129.01
128.49
128.31
127.60
127.48
126.50
126.23
37.72
32.00
26.17
N
N
H
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm