Electronic Supplementary Material (ESI) for ChemComm.

Iron Catalyzed Cross-Couplings of Azetidines: Application to an Improved Formal Synthesis of a Pharmacologically Active Molecule

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Supporting Information

General Information

All reagents were obtained from commercial suppliers unless otherwise stated. Organic solvents were routinely dried and/or distilled prior to use and stored over molecular sieves under argon. Solvents for chromatography were technical grade and distilled prior to use. Thin layer chromatography (TLC) was carried out on Merck aluminum support plates Silicagel 60 F_{254}. Visualization was achieved under a UV mineral light. Column chromatography was performed using silicagel Merck 60 (particle size 0.2-0.063 mm). Proton NMR (^{1}H NMR) spectra were recorded on Varian 400 MHz and 600 MHz spectrometers at 400 and 600 MHz. Carbon NMR (^{13}C) spectra were similarly recorded at 100 or 150 MHz, using a broadband decoupled mode. ^{19}F measurements were performed at 376 and 282 MHz respectively on Varian 400 MHz and Mercury 300 MHz spectrometers. Proton and carbon NMR chemical shifts (δ) are reported in parts per million (ppm) relative to residual proton signals in $CDCl_3$ (δ = 7.26, 77.16). Fluorine NMR chemical shifts (δ) are reported in parts per million (ppm) relative to $CFCl_3$ (δ = 0.00). Coupling constants (*J*) are reported in Hertz (Hz) and refer to apparent multiplicities. The following abbreviations are used for the multiplicities: s: singlet, d: doublet, t: triplet, q: quartet, qu: quintet, m: multiplet, br: broad. Mass spectra (EI-MS, 70 eV) were conducted on a Finnigan SSQ 7000 spectrometer. HRMS were recorded on a Thermo Scientific LTQ Orbitrap XL spectrometer. IR spectra were recorded on a Jasco FT/IR-420 spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Chemical yields refer to pure isolated substances unless otherwise stated.

Experimental Procedures

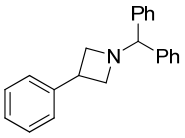
Unless otherwise noted, all Grignard reagents were prepared using this general procedure:

General Procedure A:

Preparation of the Grignard reagent: To oven-dried Mg turnings (5 equiv) in a two-neck flask was added a small amount of THF and a small amount of the desired bromide/iodide. Vigorous stirring and gentle warming caused the reaction to initiate and a dark grey-like colour formed. The rest of the THF followed by the substrate was added slowly and then the mixture was heated to reflux for 2 hours and allowed to cool before use. Only the desired volume of Grignard reagent was drawn up into a syringe and used in a seperate reaction vessel.

Iron-catalyzed coupling: To a screw-cap test tube was added the substrate and iron catalyst. The tube was evacuated and refilled with a N_2 atmosphere three times before the addition of THF (1 mL per 0.1 mmol) and then cooled to -20 ˚C. Meanwhile, to an oven-dried test tube was added TMEDA and the freshly-prepared Grignard, mixed and then drawn back into the syringe. The addition of this solution was performed dropwise over 2 hours (10 min intervals) and the reaction was then stirred at room temperature for 10 mins before being quenched with aqueous NH_4Cl. The aqueous was extracted with Et_2O (×3), washed with brine, dried (Na_2SO_4) and concentrated to yield the crude product. The pure product was isolated by flash column chromatography.

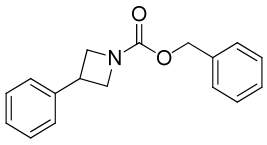
1-Benzhydryl-3-phenylazetidine 2a



2a

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (175 mg, 0.50 mmol, 1 equiv), $Fe(acac)_3$ (18 mg, 0.05 mmol, 10 mol%), TMEDA (174 mg, 1.5 mmol, 3 equiv) and phenylmagnesium bromide (1.83 mL, 1.5 mmol, 3 equiv, 0.82 M solution in THF) gave after column chromatography on silica gel eluting with 50:1 pentane:Et_2O the title compound (100 mg, 0.333 mmol, 67 %) as a colourless solid; m.p. 65 °C; ν_{max} (neat)/cm^{-1} 3844, 3377, 3027, 2931, 2817, 2318, 2062, 1953, 1890, 1735, 1662, 1453, 1365, 1269, 1076, 1027, 984, 911, 809; δ_H (600 MHz, $CDCl_3$) 7.47 (d, *J* = 7.4 Hz, 4H, C*H*$_{arom}$), 7.31 (td, *J* = 14.9, 7.9 Hz, 7H, C*H*$_{arom}$), 7.24 – 7.16 (m, 4H, C*H*$_{arom}$), 4.43 (s, 1H, C*H*), 3.79 – 3.70 (m, 1H, C*H*), 3.68 (dd, *J* = 15.2, 8.5 Hz, 2H, C*H*$_2$), 3.18 (m, *J* = 6.5 Hz, 2H, C*H*$_2$); δ_C (151 MHz, $CDCl_3$) 132.3, 130.0 (2 × C_q, Ar), 128.4 (2 × *C*H, Ar), 127.5 (2 × *C*H, Ar), 127.1, 126.90 (2 × *C*H, Ar), 126.3, 78.2, 60.6, 34.9; *m/z* (EI) 299 (M^+, 36), 167 (100), 152 (16), 104 (35), 91 (16).

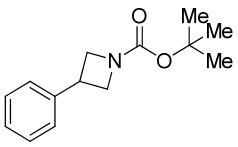
Benzyl 3-phenylazetidine-1-carboxylate 2b



2b

As for general procedure A, reaction of benzyl 3-iodoazetidine-1-carboxylate (159 mg, 0.50 mmol, 1.0 equiv), $Fe(acac)_3$ (17.7 mg, 0.050 mmol, 10 mol%), TMEDA (174 mg, 1.5 mmol, 3 equiv) and phenylmagnesium bromide (1.79 mL, 1.5 mmol, 3 equiv, 0.84 M solution in THF) gave after chromatography on silica gel eluting with 50:1 pentane:Et_2O the title compound (86 mg, 0.320 mmol, 64%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3846, 3427, 3033, 2956, 2889, 2671, 2325, 2085, 1886, 1702, 1415, 1349, 1261, 1121, 1010, 911, 749, 696; δ_H (400 MHz, $CDCl_3$) 7.44 – 7.20 (m, 10H, CH_{arom}), 5.14 (s, 2H, CH_2), 4.41 (t, *J* = 8.7 Hz, 2H, CH_2), 4.07 (dd, *J* = 8.7, 6.1 Hz, 2H, CH_2), 3.84 – 3.70 (m, 1H, C*H*); δ_C (101 MHz, $CDCl_3$) 156.5, 141.7, 136.5, 128.8, 128.5 (2 x *C*H, Ar), 128.0 (2 x *C*H, Ar), 128.0 (2 x *C*H, Ar), 127.1 (2 x *C*H, Ar), 126.7, 66.6, 58.6 (2 x *C*H_2) 33.9; *m/z* (CI) 268 ([M+H]$^+$), 314 (34), 358 (38), 401 (55), 467 (89), 501 (100), 536 (31).

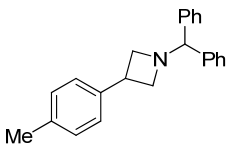
***tert*-Butyl 3-phenylazetidine-1-carboxylate 2c**



2c

As for general procedure A, reaction of *tert*-butyl 3-iodoazetidine-1-carboxylate (142 mg, 0.50 mmol, 1 equiv), $Fe(acac)_3$ (17.7 mg, 0.050 mmol, 10 mol%), TMEDA (174 mg, 1.5 mmol, 3 equiv) and phenylmagnesium bromide (1.79 mL, 1.5 mmol, 3 equiv, 0.84 M solution in THF) gave after column chromatography on silica gel eluting with 50:1 pentan:Et_2O the title compound (71 mg, 0.302 mmol, 60%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3833, 3395, 2968, 2888, 2298, 2072, 1910, 1697, 1598, 1390, 1258, 1012, 907, 856, 752, 697; δ_H (400 MHz, $CDCl_3$) 7.39 – 7.18 (m, 5H, CH_{arom}), 4.31 (m, *J* = 8.7 Hz, 2H, CH_2), 3.97 (dd, *J* = 8.6, 6.1 Hz, 2H, CH_2), 3.78 – 3.65 (m, 1H, C*H*), 1.45 (s, 9H, CH_3); δ_C (101 MHz, $CDCl_3$) 156.4, 142.2, 128.8 (2 x *C*H, Ar), 126.9, 126.7 (2 x *C*H, Ar) 79.5, 33.1, 28.4 (3 x *C*H_3); *m/z* (CI) 234 ([M+H]$^+$, 6), 467 (100).

1-benzhydryl-3-p-tolylazetidine 2d

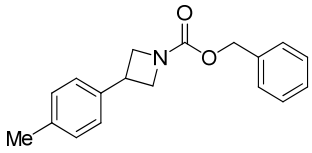


2d

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and p-tolylmagnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (54 mg, 0.173 mmol, 60%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3026, 2940, 2829, 2323, 2250, 2084, 1893, 1740, 1587, 1495, 1453, 1355, 1270, 1207, 1071, 1027, 907, 811, 727; δ_H (400 MHz, $CDCl_3$) 7.44 (d, *J* = 7.3 Hz, 4H, CH_{arom}), 7.26 (dd, *J* = 13.1, 5.4 Hz, 4H, CH_{arom}), 7.18 (dd, *J* = 7.7, 5.8 Hz, 4H, CH_{arom}), 7.12 (d, *J* = 7.9 Hz, 2H, CH_{arom}), 4.40 (s, 1H, C*H*), 3.72 – 3.58 (m, 3H, CH, CH_2), 3.13 (t, *J* = 9.8 Hz, 2H, CH_2), 2.32

(s, 3H. CH_3); δ_C (101 MHz, $CDCl_3$) 142.2, 140.0, 135.8, 129.0, 128.4, 127.5, 127.1, 126.8, 78.2, 60.8, 34.5, 21.0; *m/z* (EI) 313 (M^+, 45), 118 (100), 167 (87), 196 (52), 152 (49), 92 (46).

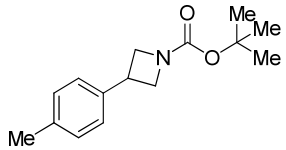
Benzyl 3-(*p*-tolyl)azetidine-1-carboxylate 2e



2e

As for general procedure A, reaction of benzyl 3-iodoazetidine-1-carboxylate (100 mg, 0.315 mmol, 1 equiv), $Fe(acac)_3$ (11.1 mg, 0.0315 mmol 10 mol%), TMEDA (110 mg, 0.946 mmol, 3 equiv) and *p*-tolylmagnesium bromide (1.05 mL, 0.946 mmol, 3 equiv, 0.9 M solution in THF) gave after chromatography on silica gel eluting with 50:1-20:1 pentane:Et_2O the title compound (93 mg, 0.331 mmol, 66%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3417, 2952, 2890, 2197, 2062, 1898, 1704, 1415, 1348, 1121, 982, 917, 812, 748; δ_H (600 MHz, $CDCl_3$) 7.42 – 7.30 (m, 5H, CH_{arom}), 7.18 (dd, *J* = 22.8, 8.1 Hz, 4H, CH_{arom}), 5.13 (s, 2H, CH_2), 4.40 (t, *J* = 8.7 Hz, 2H, CH_2), 4.04 (dd, *J* = 8.4, 6.2 Hz, 2H, CH_2), 3.80 – 3.72 (m, 1H, CH), 2.34 (s, 3H, CH_3); δ_C (151 MHz, $CDCl_3$) 156.5, 138.8, 136.7, 136.7, 129.4 (2 x *C*H, Ar), 128.5 (2 x *C*H, Ar), 128.0, 128.0 (2 x *C*H, Ar), 126.6 (2 x *C*H, Ar), 66.6, 58.2 (3 x *C*H_2) 33.6, 21.0; *m/z* (CI) 282 ($[M+H]^+$, 18), 328.3 (58), 372.3 (88), 563.4 (100).

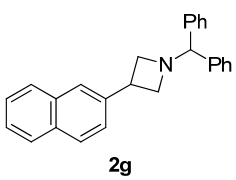
***tert*-Butyl 3-(*p*-tolyl)azetidine-1-carboxylate 2f**



2f

As for general procedure A, reaction of *tert*-butyl 3-iodoazetidine-1-carboxylate (100 mg, 0.353 mmol, 1 equiv), $Fe(acac)_3$ (12.5 mg, 0.0353 mmol, 10 mol%), TMEDA (123 mg, 1.06 mmol, 3 equiv) and *p*-tolylmagnesium bromide (1.18 mL, 1.06 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 50:1 pentan:Et_2O the title compound (82 mg, 0.332 mmol, 66 %) as a colourless oil; ν_{max} (neat)/cm^{-1} 3841, 3373, 2965, 2718, 2319, 2103, 1897, 1698, 1388, 1248, 1131, 968, 907, 856, 809, 774; δ_H (600 MHz, $CDCl_3$) 7.18 (dd, *J* = 25.5, 8.1 Hz, 4H, CH_{arom}), 4.31 (t, *J* = 8.7 Hz, 2H, CH_2), 3.96 (dd, *J* = 8.2, 6.5 Hz, 2H, CH_2), 3.75 – 3.64 (m, 1H, C*H*), 2.34 (s, 3H, CH_3), 1.48 (s, 9H, 3 x CH_3); δ_C (151 MHz, $CDCl_3$) 156.4, 139.0, 136.6, 129.4 (2 x *C*H, Ar), 126.4 (2 x *C*H, Ar), 79.2, 58.0 (2 x *C*H_2), 33.2, 28.1 (3 x *C*H_3), 20.7; *m/z* (CI) 248 ($[M+H]^+$, 2), 395 (25), 495 (100).

1-Benzhydryl-3-(naphthalen-2-yl)azetidine 2g

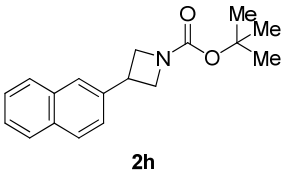


2g

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and naphthalen-2-ylmagnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound

(64 mg, 0.183 mmol, 64%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3894, 3697, 3458, 3028, 2945, 2826, 2318, 2079, 1891, 1740, 1597, 1492, 1451, 1364, 1211, 1282, 1211, 1066, 919, 826, 742, 698; δ_H (600 MHz, $CDCl_3$) 7.80 (dd, *J* = 14.0, 8.1 Hz, 3H, C*H*$_{arom}$), 7.67 (s, 1H, C*H*$_{arom}$), 7.52 – 7.39 (m, 7H, C*H*$_{arom}$), 7.30 (t, *J* = 7.6 Hz, 4H, C*H*$_{arom}$), 7.21 (t, *J* = 7.3 Hz, 2H, C*H*$_{arom}$), 4.46 (s, 1H, C*H*), 3.95 – 3.83 (m, 1H, C*H*), 3.74 (br. s, 2H, C*H*$_2$), 3.27 (br. s, 2H, C*H*$_2$); δ_C (151 MHz, $CDCl_3$) 142.2, 140.5, 133.4, 132.2, 128.5 (4 x *C*H, Ar), 128.1, 127.5 (2 x *C*H, Ar), 127.1 (5 x *C*H, Ar), 126.1, 125.5 125.4, 125.1, 78.3, 60.5 (2 x *C*H$_2$), 34.9; *m/z* (EI) 349 (M$^+$, 37), 167.1 (73), 154.1 (100).

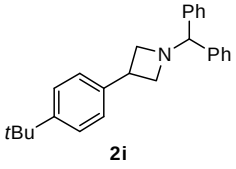
tert-Butyl 3-(naphthalen-2-yl)azetidine-1-carboxylate 2h



2h

As for general procedure A, reaction of *tert*-butyl 3-iodoazetidine-1-carboxylate (100 mg, 0.353 mmol, 1 equiv), $Fe(acac)_3$ (12.5 mg, 0.0353 mmol, 10 mol%), TMEDA (123 mg, 1.06 mmol, 3 equiv) and naphthalen-2-ylmagnesium bromide (1.18 mL, 1.06 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 50:1-20:1 pentane:Et_2O the title compound (64 mg, 0.183 mmol, 64%) as a colourless solid; m.p. 71 °C; ν_{max} (neat)/cm^{-1} 3832, 3361, 2963, 2482, 2281, 2064, 1928, 1686, 1546, 1483, 1383, 1249, 1128, 1015, 946, 818, 749; δ_H (600 MHz, $CDCl_3$) 7.86 – 7.76 (m, 3H, C*H*$_{arom}$), 7.71 (s, 1H, C*H*$_{arom}$), 7.47 (qd, *J* = 9.7, 3.5 Hz, 3H, C*H*$_{arom}$), 4.40 (m, *J* = 8.7 Hz, 2H, C*H*$_2$), 4.13 – 4.04 (m, 2H, C*H*$_2$), 3.90 (tt, *J* = 8.7, 6.0 Hz, 1H, C*H*), 1.49 (s, 9H, 3 x C*H*$_3$); δ_C (151 MHz, $CDCl_3$) 156.5, 139.5, 133.3, 132.4, 128.7, 127.6 (2 x *C*H, Ar), 126.3, 125.8 125.4, 124.8, 79.6, 58.5 (2 x CH_2), 33.7, 28.4 (3 x *C*H$_3$); *m/z* (CI) 284 ([M+H]$^+$, 8), 467 (40), 567 (100).

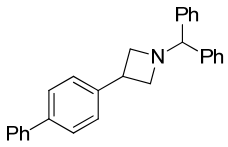
1-Benzhydryl-3-(4-tert-butylphenyl)azetidine 2i



2i

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (70 mg, 0.20 mmol, 1 equiv), $Fe(acac)_3$ (7 mg, 0.020 mmol, 10 mol%), TMEDA (70 mg, 0.60 mmol, 3 equiv) and (4-tert-butylphenyl)magnesium bromide (0.7 mL, 0.60 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (40 mg, 0.113 mmol, 56%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3060, 2957, 2324, 2100, 1908, 1681, 1601, 1490, 1454, 1357, 1269, 1197, 1072, 1022, 917, 829, 744, 700; δ_H (600 MHz, $CDCl_3$) 7.46-7.47 (3H, m, C*H*$_{arom}$), 7.36-7.37 (2H, m, C*H*$_{arom}$), 7.25-7.31 (7H, m, C*H*$_{arom}$), 7.19-7.22 (2H, m, C*H*$_{arom}$), 4.42 (1H, s, C*H*), 3.68-3.72 (1H, m, C*H*), 3.64-3.68 (2H, m, C*H*$_2$), 3.17 (2H, app. t, *J* = 7.0, C*H*$_2$), 1.33 (9H, s, 3 × C*H*$_3$); δ_C (151 MHz, $CDCl_3$) 149.3, 142.4, 140.1, 128.6, 127.7, 127.2, 126.8, 125.4, 78.4, 60.8, 34.6, 31.5, 31.2; *m/z* (EI) 355 (M$^+$, 100), 145 (88), 160 (79), 167 (44).

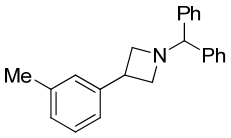
1-Benzhydryl-3-(biphenyl-4-yl)azetidine 2j



2j

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (85 mg, 0.24 mmol, 1 equiv), $Fe(acac)_3$ (9 mg, 0.02 mmol, 10 mol%), TMEDA (85 mg, 0.73 mmol, 3 equiv) and biphenyl-4-ylmagnesium bromide (0.8 mL, 0.73 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (29 mg, 0.077 mmol, 32%) as a white solid; m.p. 84 °C; ν_{max} (neat)/cm^{-1} 3026, 2933, 2803, 1681, 1599, 1486, 1450, 1267, 1196, 1152, 1074, 1027, 819, 750, 696; δ_H (600 MHz, $CDCl_3$) 7.55-7.60 (4H, m, C*H*$_{arom}$), 7.39-7.48 (8H, m, C*H*$_{arom}$), 7.29-7.36 (5H, m, C*H*$_{arom}$), 7.19-7.22 (2H, m, C*H*$_{arom}$), 4.44 (1H, s, C*H*Ph_2), 3.73-3.78 (1H, m, CHAr), 3.68 (2H, app. t, *J* = 7.3, NC*H*$_2$), 3.20 (2H, app. t, *J* = 7.3, NC*H*$_2$); δ_C (151 MHz, $CDCl_3$) 142.3 (×2), 141.1, 139.4, 128.9, 128.6 (×2), 127.7, 127.5, 127.28, 127.2, 127.2, 78.4, 60.8, 34.7; *m/z* (CI) 376 ($[M+H]^+$, 2), 165 (100), 180 (98).

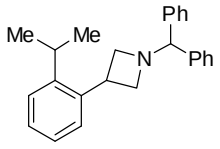
1-Benzhydryl-3-m-tolylazetidine 2k



2k

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.03 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and m-tolylmagnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (36 mg, 0.115 mmol, 40%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3027, 2943, 2826, 2326, 2107, 1937, 1599, 1487, 1451, 1345, 1274, 1204, 1147, 1073, 1027, 927, 781, 744, 698; δ_H (600 MHz, $CDCl_3$) 7.46 (4H, d, *J* = 7.5, C*H*$_{arom}$), 7.28-7.30 (4H, m, C*H*$_{arom}$), 7.19-7.23 (3H, m, C*H*$_{arom}$), 7.03-7.11 (3H, m, C*H*$_{arom}$), 4.42 (1H, s, C*H*), 3.65-3.73 (3H, m, C*H* + C*H*$_2$), 3.14-3.16 (2H, m, C*H*$_2$), 2.35 (3H, s, C*H*$_3$); δ_C (151 MHz, $CDCl_3$) 143.1, 142.3, 138.1, 128.6, 128.5, 127.8, 127.7, 127.2, 127.2, 124.0, 78.4, 60.8, 34.9, 21.6; *m/z* (EI) 313 ($[M]^+$, 100), 167 (98).

1-Benzhydryl-3-(2-isopropylphenyl)azetidine 2l

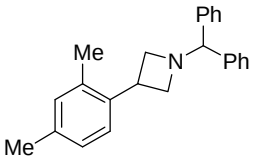


2l

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (85 mg, 0.24 mmol, 1 equiv), $Fe(acac)_3$ (9 mg, 0.02 mmol, 10 mol%), TMEDA (85 mg, 0.73 mmol, 3 equiv) and (2-isopropylphenyl)magnesium bromide (0.8 mL, 0.73 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (80 mg, 0.235 mmol, 96%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3061, 3027, 2959, 2322, 2086, 1918, 1597, 1486, 1451, 1350, 1270, 1201, 1150, 1075, 1031, 912, 811, 750, 699; δ_H (600 MHz, $CDCl_3$) 7.46 (4H, d, *J* = 8.1, C*H*$_{arom}$), 7.29-7.32 (4H, m, C*H*$_{arom}$), 7.25-7.27 (2H, m, C*H*$_{arom}$), 7.17-7.24 (4H, m, C*H*$_{arom}$), 4.37

(1H, s, C*H*Ph_2), 3.98-4.02 (1H, m, C*H*Ar), 3.72 (2H, dd, *J* = 11.3, 4.8, CH_2), 3.15 (2H, dd, *J* = 11.3, 4.8, CH_2), 2.99 (1H, sept., *J* = 6.9, $CH(CH_3)_2$), 1.17 (6H, d, *J* = 6.9, 2 × C*H*$_3$); δ_C (151 MHz, $CDCl_3$) 146.8, 142.2, 138.9, 128.5, 127.5, 127.1, 126.6, 126.2, 125.7, 125.1, 78.5, 60.2, 32.3, 28.9, 23.9; *m/z* (EI) 341 ($[M]^+$, 17), 167 (100), 131 (70).

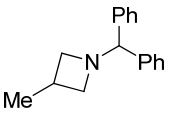
1-Benzhydryl-3-(2,4-dimethylphenyl)azetidine 2m



2m

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (70 mg, 0.20 mmol, 1 equiv), $Fe(acac)_3$ (7 mg, 0.02 mmol, 10 mol%), TMEDA (70 mg, 0.60 mmol, 3 equiv) and (2,4-dimethylphenyl)magnesium bromide (0.7 mL, 0.60 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (45 mg, 0.138 mmol, 69%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3025, 2940, 2831, 1663, 1601, 1452, 1350, 1269, 1196, 1138, 1069, 907, 814, 704; δ_H (600 MHz, $CDCl_3$) 7.46 (4H, dd, *J* = 10.0, 3.1, C*H*$_{arom}$), 7.31 (4H, dd, *J* = 10.0, 5.5, C*H*$_{arom}$), 7.20-7.23 (2H, m, C*H*$_{arom}$), 7.10 (1H, d, *J* = 7.8, C*H*$_{arom}$), 7.01 (1H, d, *J* = 7.8, C*H*$_{arom}$), 6.95 (1H, s, C*H*$_{arom}$), 4.35 (1H, s, C*H*Ph_2), 3.86 (1H, p, *J* = 8.0, C*H*Ar), 3.73 (2H, dd, *J* = 10.7, 4.5, CH_2), 3.10 (2H, app. t, *J* = 8.0, CH_2), 2.31 (3H, s, CH_3), 2.14 (3H, s, CH_3); δ_C (151 MHz, $CDCl_3$) 142.3, 137.7, 136.0, 135.9, 131.0, 128.6, 127.7, 127.2, 126.6, 125.9, 78.7, 59.8, 32.8, 21.0, 19.6; *m/z* (EI) 327 ($[M]^+$, 47), 132 (100), 167 (74).

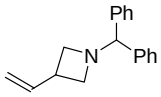
1-Benzhydryl-3-methylazetidine 2n



2n

As for general procedure A, the reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and methylmagnesium bromide (0.43 mL, 0.86 mmol, 3 equiv, 2 M solution in THF from commercial sources) gave after chromatography on silica gel eluting with 50:1-20:1 pentane:Et_2O the title compound (43 mg, 0.182 mmol, 64%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3856, 3028, 2945, 2818, 2324, 2080, 1951, 1890, 1738, 1595, 1454, 1356, 1272, 1203, 1052, 915, 813, 742, 690; δ_H (400 MHz, $CDCl_3$) 7.38 (dd, *J* = 8.1, 1.0 Hz, 4H, C*H*$_{arom}$), 7.24 (dd, *J* = 8.3, 6.8 Hz, 4H, C*H*$_{arom}$), 7.20 – 7.10 (m, 2H, C*H*$_{arom}$), 4.29 (s, 1H, C*H*), 3.36 (dd, *J* = 7.4 Hz, 2H, CH_2), 2.64 (dd, *J* = 7.3 Hz, 2H, CH_2), 2.53 (tt, *J* = 14.0, 7.0 Hz, 1H, C*H*), 1.12 (d, *J* = 6.6 Hz, 3H, CH_3); δ_C (151 MHz, $CDCl_3$) 142.2, 128.3 (2 x *C*H, Ar), 127.5 (2 x *C*H, Ar), 126.9, 78.5, 61.4 (2 x *C*H_2), 25.1, 19.4; *m/z* (EI) 237 ($[M]^+$, 54), 167 (100), 160 (82), 91 (19).

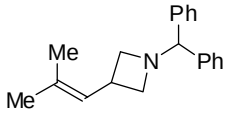
1-Benzhydryl-3-vinylazetidine 2o



2o

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and vinylmagnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 1 M solution in THF from commerical sources) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (50 mg, 0.211 mmol, 74%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3073, 3027, 2945, 2814, 1642, 1596, 1490, 1451, 1342, 1303, 1267, 1200, 1126, 1064, 1027, 988, 916, 819, 747, 698, 663; δ_H (600 MHz, $CDCl_3$) 7.40 (2H, d, *J* = 7.3, C*H*$_{arom}$), 7.24-7.27 (2H, m, C*H*$_{arom}$), 7.16-7.19 (1H, m, C*H*$_{arom}$), 5.96 (1H, ddd, *J* = 17.4, 10.3, 7.4, H_2C=C*H*), 4.97-5.02 (2H, m, *H*$_2$C=CH), 3.42 (2H, app. t, *J* = 7.1, NC*H*$_2$), 3.10-3.16 (1H, m, C*H*), 2.87 (2H, app. t, *J* = 7.1, NC*H*$_2$); δ_C (151 MHz, $CDCl_3$) 142.3, 140.1, 128.5, 127.6, 127.2, 114.8, 78.5, 59.5, 33.9; *m/z* (EI) 249 (M^+, 7), 167 (100).

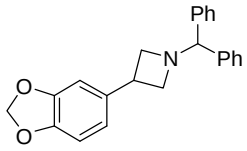
1-Benzhydryl-3-(2-methylprop-1-enyl)azetidine 2p



2p

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and (2-methylprop-1-enyl)magnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 1 M solution in THF from commerical sources) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (29 mg, 0.105 mmol, 37%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3065, 2958, 2813, 1595, 1486, 1449, 1268, 1186, 1072, 1028, 833, 746, 698; δ_H (400 MHz, $CDCl_3$) 7.39-7.41 (2H, m, C*H*$_{arom}$), 7.24-7.28 (2H, m, C*H*$_{arom}$), 7.15-7.19 (1H, m, C*H*$_{arom}$), 5.25-5.28 (1H, m, C*H*=C(CH$_3$)$_2$), 4.29 (1H, s, C*H*), 3.43-3.47 (2H, m, NC*H*$_2$), 3.28-3.36 (1H, m, C*H*), 2.75 (2H, dd, *J* = 10.6 4.6, NC*H*$_2$), 1.67 (3H, s, C*H*$_3$), 1.55 (3H, s, C*H*$_3$); δ_C (101 MHz, $CDCl_3$) 142.5, 133.0, 128.5, 127.7, 127.1, 127.0, 78.6, 60.9, 29.3, 25.7, 18.2; *m/z* (EI) 277 (M^+, 35), 167 (100).

1-Benzhydryl-3-(benzo[d][1,3]dioxol-5-yl)azetidine 2q

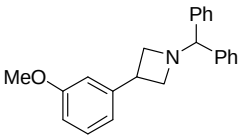


2q

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (48 mg, 0.14 mmol, 1 equiv), $Fe(acac)_3$ (5 mg, 0.014 mmol, 10 mol%), TMEDA (48 mg, 0.41 mmol, 3 equiv) and benzo[d][1,3]dioxol-5-ylmagnesium bromide (0.5 mL, 0.41 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (40 mg, 0.117 mmol, 85%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3062, 2947, 2892, 2828, 2775, 2329, 2108, 1604, 1487, 1446, 1242, 1187, 1122, 1075, 1036, 932, 859, 806, 745, 701; δ_H (600 MHz, $CDCl_3$) 7.42 (4H, dd, *J* = 8.01, 0.9, C*H*$_{arom}$), 7.24-7.27 (4H, m, C*H*$_{arom}$), 7.16-7.18 (2H, m, C*H*$_{arom}$), 6.89 (1H, d, *J* =

1.6, C*H*$_{arom}$), 6.72 (1H, d, *J* = 8.0, C*H*$_{arom}$), 6.69 (1H, dd, *J* = 8.0, 1.6, C*H*$_{arom}$), 5.92 (2H, s, C*H*$_2$OAr), 4.38 (1H, s, C*H*Ph$_2$), 3.57 – 3.60 (3H, m, C*H* + NC*H*$_2$), 3.07-3.08 (2H, m, NC*H*$_2$); δ_C (151 MHz, $CDCl_3$) 147.9, 146.1, 142.3, 137.3, 128.6, 127.7, 127.2, 120.1, 108.2, 107.5, 101.0, 78.4, 61.1, 34.9; *m/z* (EI) 343 (M^+, 5), 148 (100).

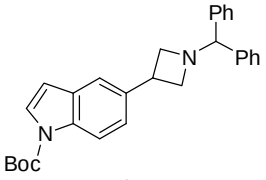
1-Benzhydryl-3-(3-methoxyphenyl)azetidine 2r



2r

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), Fe(acac)$_3$ (10 mg, 0.03 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and (3-methoxyphenyl)magnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (61 mg, 0.185 mmol, 65%) as a colourless oil; ν_{max} (neat)/cm^{-1} 3059, 3027, 2945, 2829, 2325, 2090, 1942, 1594, 1486, 1452, 1346, 1263, 1154, 1042, 909, 864, 780, 740, 698; δ_H (600 MHz, $CDCl_3$) 7.43 (4H, d, *J* = 7.2, C*H*$_{arom}$), 7.26 (4H, t, *J* = 8.0, C*H*$_{arom}$), 7.21 (1H, t, *J* = 8.0, CH$_{arom}$), 7.17 (2H, dt, *J* = 8.5, 1.1, C*H*$_{arom}$), 6.84-6.87 (1H, m, C*H*$_{arom}$), 6.74 (2H, dd, *J* = 8.0, 2.3, C*H*$_{arom}$), 4.38 (1H, s, C*H*), 3.79 (3H, s, OC*H*$_3$), 3.64-3.68 (1H, m, C*H*), 3.60-3.63 (2H, m, C*H*$_2$), 3.13 (2H, app. t, *J* = 6.8, CH$_2$); δ_C (151 MHz, $CDCl_3$) 159.8, 144.9, 142.3, 129.5, 128.6, 127.7, 127.2, 119.5, 113.0, 111.5, 78.4, 60.7, 55.3, 35.0; *m/z* (EI) 329 ($[M]^+$, 68), 133 (100), 167 (44).

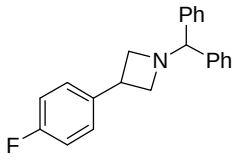
***tert*-Butyl 5-(1-benzhydrylazetidin-3-yl)-1*H*-indole-1-carboxylate 2s**



2s

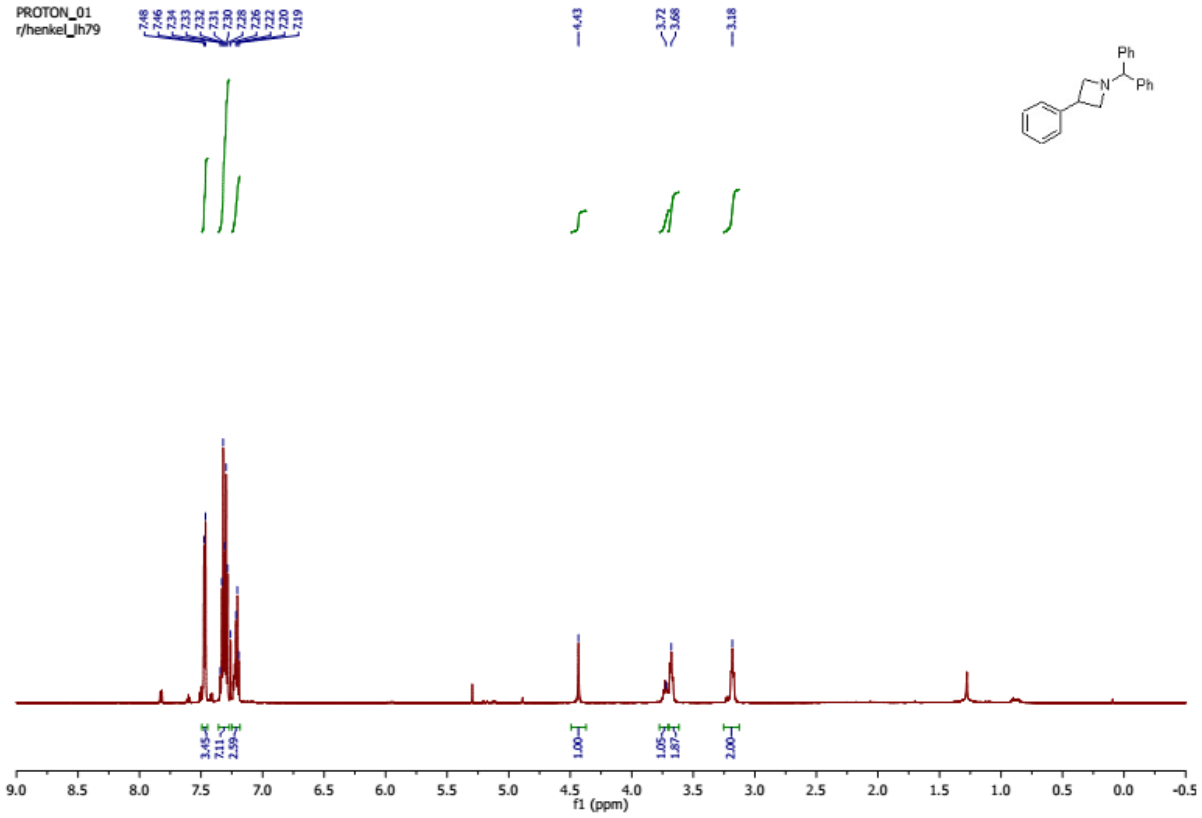
As for general procedure A, the reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), Fe(acac)$_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) 1-(*tert*-butoxycarbonyl)-1*H*-indol-5-yl)magnesium bromide gave after chromatography on silica gel eluting with 50:1-20:1 pentane:Et_2O the title compound (50.8 mg, 0.149 mmol, 52%) as a yellow oil; ν_{max} (neat)/cm^{-1} 3858, 3441, 3063, 3023, 2878, 2834, 2325, 2105, 1948, 1878, 1813, 1732, 1595, 1535, 1483, 1449, 1371, 1338, 1249, 1154, 1119, 1077, 1024, 928, 881, 853, 783, 695; δ_H (600 MHz, $CDCl_3$) 8.18 (s, 1H, C*H*$_{arom}$), 7.59 (d, *J* = 7.7 Hz, 1H, C*H*$_{arom}$), 7.41 (d, *J* = 7.3 Hz, 4H, C*H*$_{arom}$), 7.34 (t, *J* = 8.1 Hz, 1H, C*H*$_{arom}$), 7.29 (t, *J* = 7.6 Hz, 4H, C*H*$_{arom}$), 7.25 (t, *J* = 7.5 Hz, 1H, C*H*$_{arom}$), 7.21 (t, *J* = 7.3 Hz, 2H, C*H*$_{arom}$), 6.59 (d, *J* = 3.7 Hz, 1H, C*H*$_{arom}$), 4.53 (s, 1H, C*H*), 4.45 (p, *J* = 6.7 Hz, 1H, C*H*), 3.86 (dd, *J* = 9.3, 7.1 Hz, 2H, C*H*$_2$), 3.53 (dd, *J* = 8.6, 7.4 Hz, 2H, C*H*$_2$), 1.70 (s, 9H, 3 x C*H*$_3$); δ_C (151 MHz, $CDCl_3$) 149.7 (*C*$_q$), 141.6 (2 x *C*$_q$), 128.5 (3 x *C*H, Ar), 127.3 (3 x *C*H, Ar) 125.8 (3 x *C*H, Ar), 124.1, 122.6, 120.9, 115.1, 106.4, 106.5, 77.9, 65.4 (2 x *C*H$_2$), 28.2 (3 x *C*H$_3$), 4.8;

1-Benzhydryl-3-(4-fluorophenyl)azetidine 5



5

As for general procedure A, reaction of 1-benzhydryl-3-iodoazetidine (100 mg, 0.29 mmol, 1 equiv), $Fe(acac)_3$ (10 mg, 0.029 mmol, 10 mol%), TMEDA (100 mg, 0.86 mmol, 3 equiv) and (4-fluorophenyl)magnesium bromide (1.0 mL, 0.86 mmol, 3 equiv, 0.9 M solution in THF) gave after column chromatography on silica gel eluting with 10:1 hexane:Et_2O the title compound (55 mg, 0.17 mmol, 61%) as a yellow oil which contained small amounts of the bi-aryl dimer; ν_{max} (neat)/cm^{-1} 3031, 2938, 2802, 1600, 1495, 1223, 1155, 1083, 1024, 915, 817, 746, 701; δ_H (400 MHz, $CDCl_3$) 3.17 (2H, app. dt, *J* = 10.0, 5.7, 2 × C*H*$_a$H$_b$N), 3.62-3.79 (3H, m, 2 × CH$_a$*H*$_b$N + C*H*Ar), 4.44 (1H, app. d, *J* = 12.3, C*H*Ph_2), 6.99-7.56 (14H, m, ArC*H*); δ_C (100 MHz, $CDCl_3$) 34.5 (d, *J* = 31), 60.9 (d, *J* = 14), 78.4 (d, *J* = 3), 115.3 (d, *J* = 28), 115.7 (d, *J* = 21), 127.2 (d, *J* = 13), 127.6 (d, *J* = 4), 127.7, 128.6, 137.8 (d, *J* = 128), 162.5 (d, *J* = 246); δ_F (376 MHz, $CDCl_3$) -116.0; *m/z* (EI) 317 (M^+, 8), 167 (100), 198 (90);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7.30
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7.22
7.20
7.19
4.43
3.72
3.68
3.18
Ph
N
Ph
3.45
7.11
2.59
1.00
1.05
1.87
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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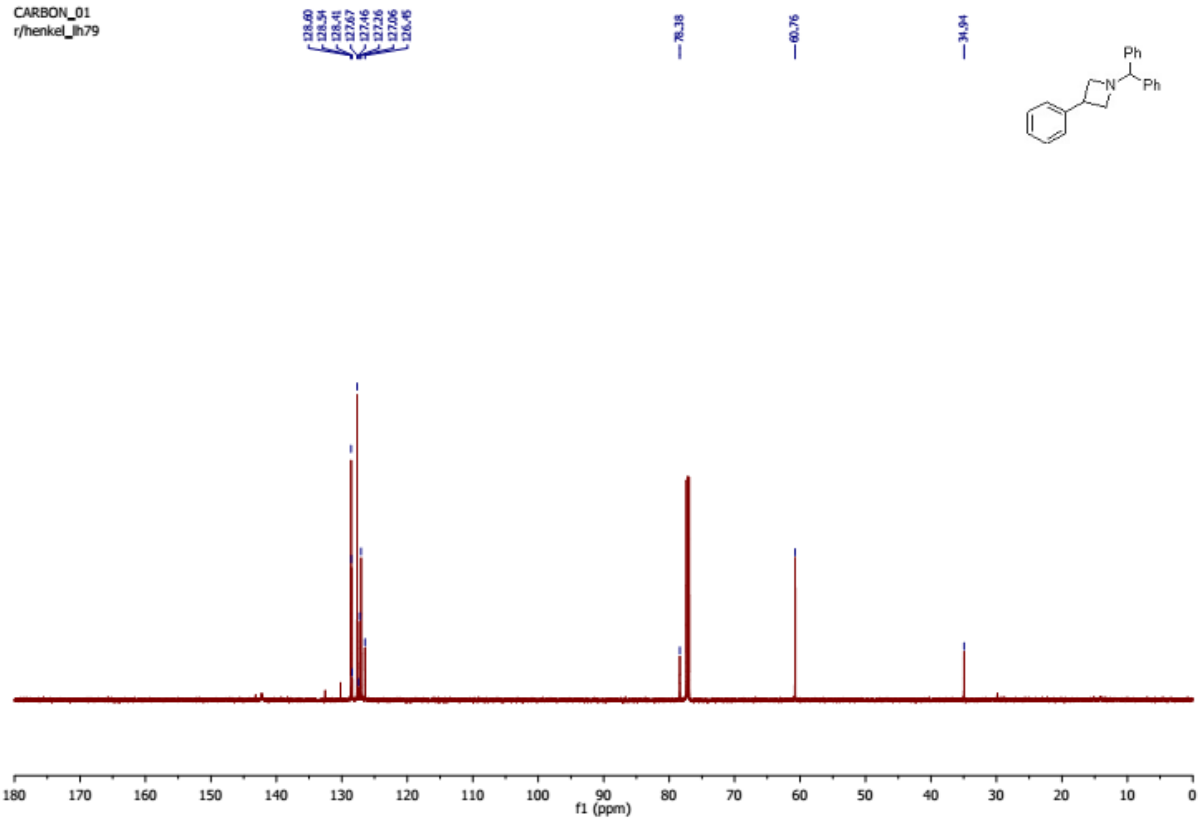
-0.5
f1 (ppm)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N
Ph
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110
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90
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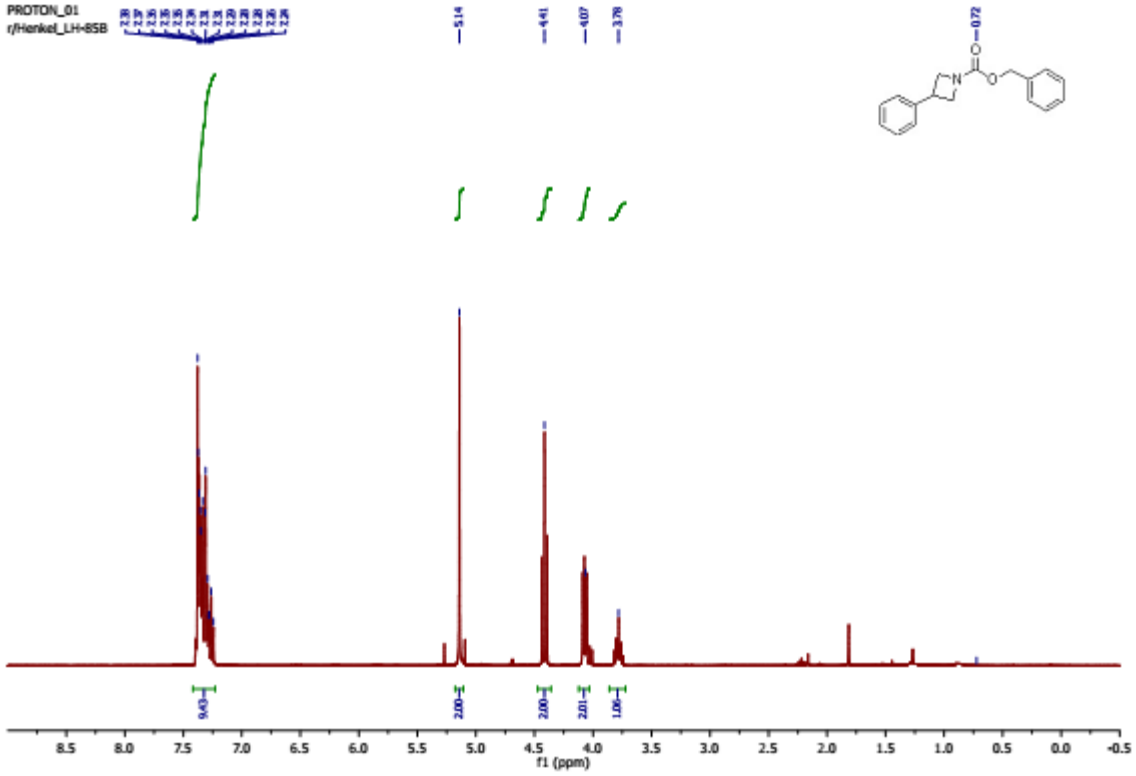
50
40
30
20
10
0
f1 (ppm)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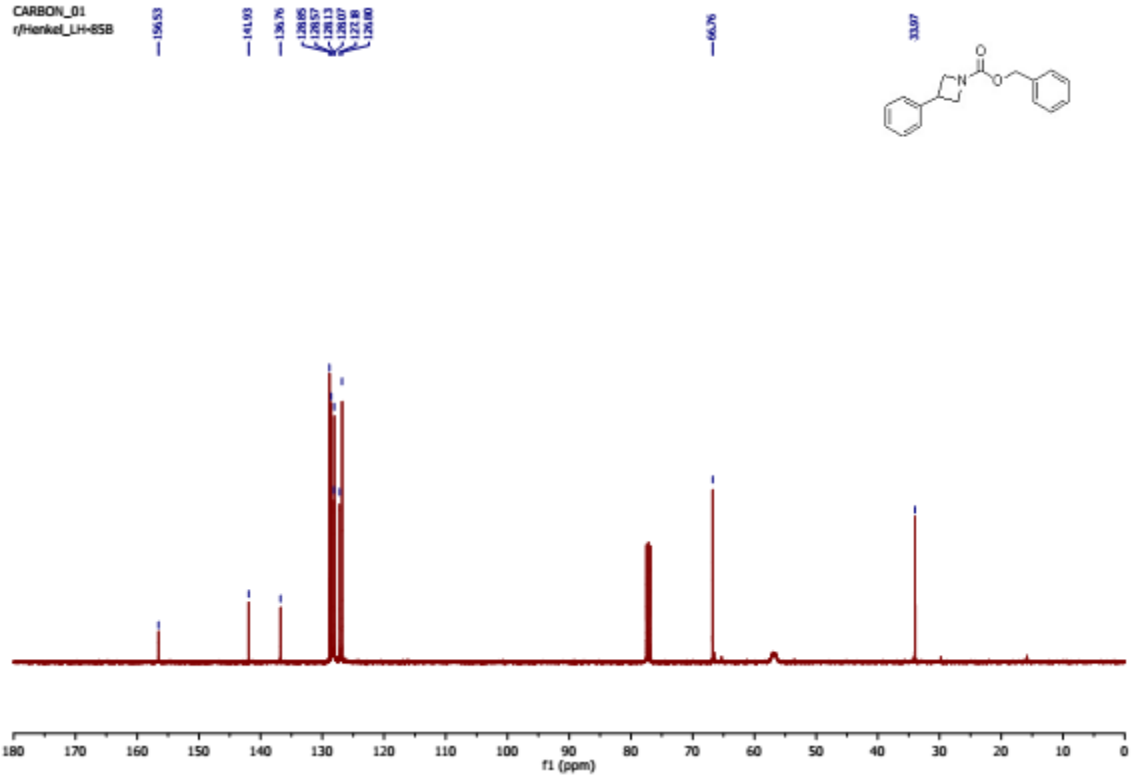
1.06
f1 (ppm)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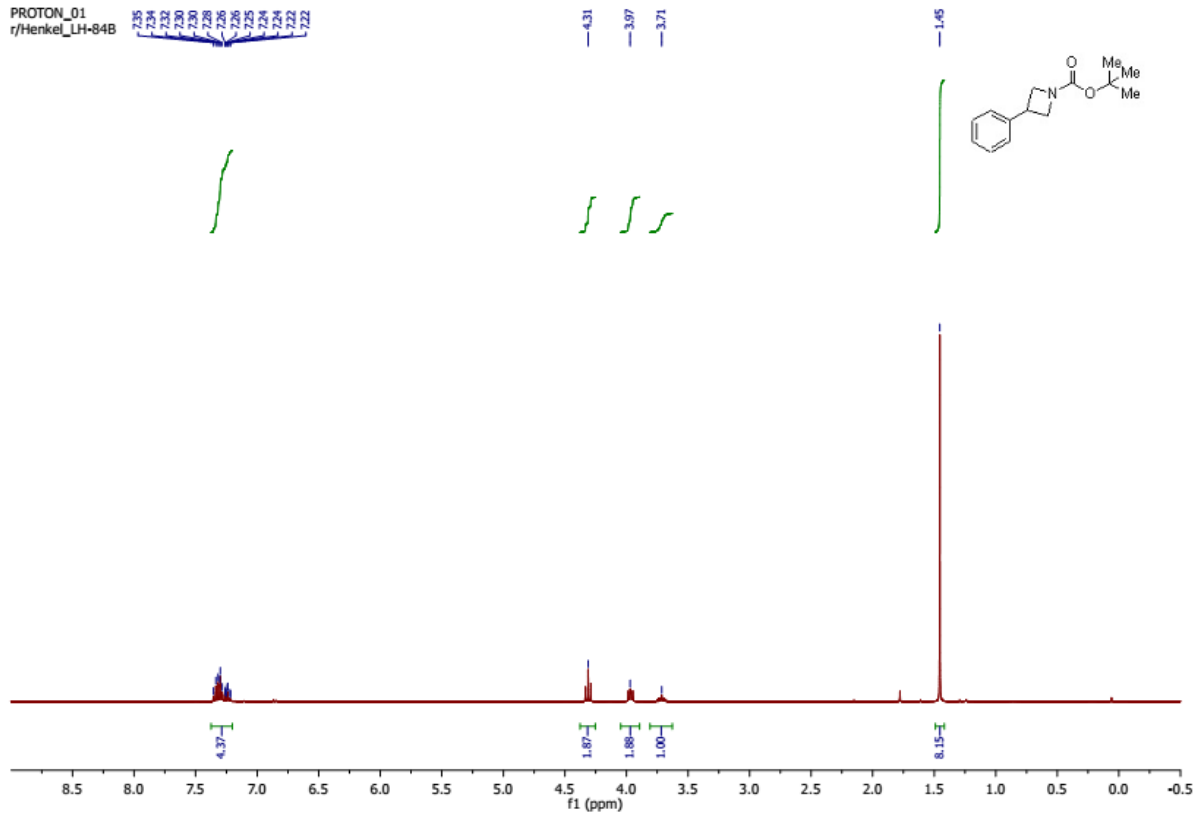
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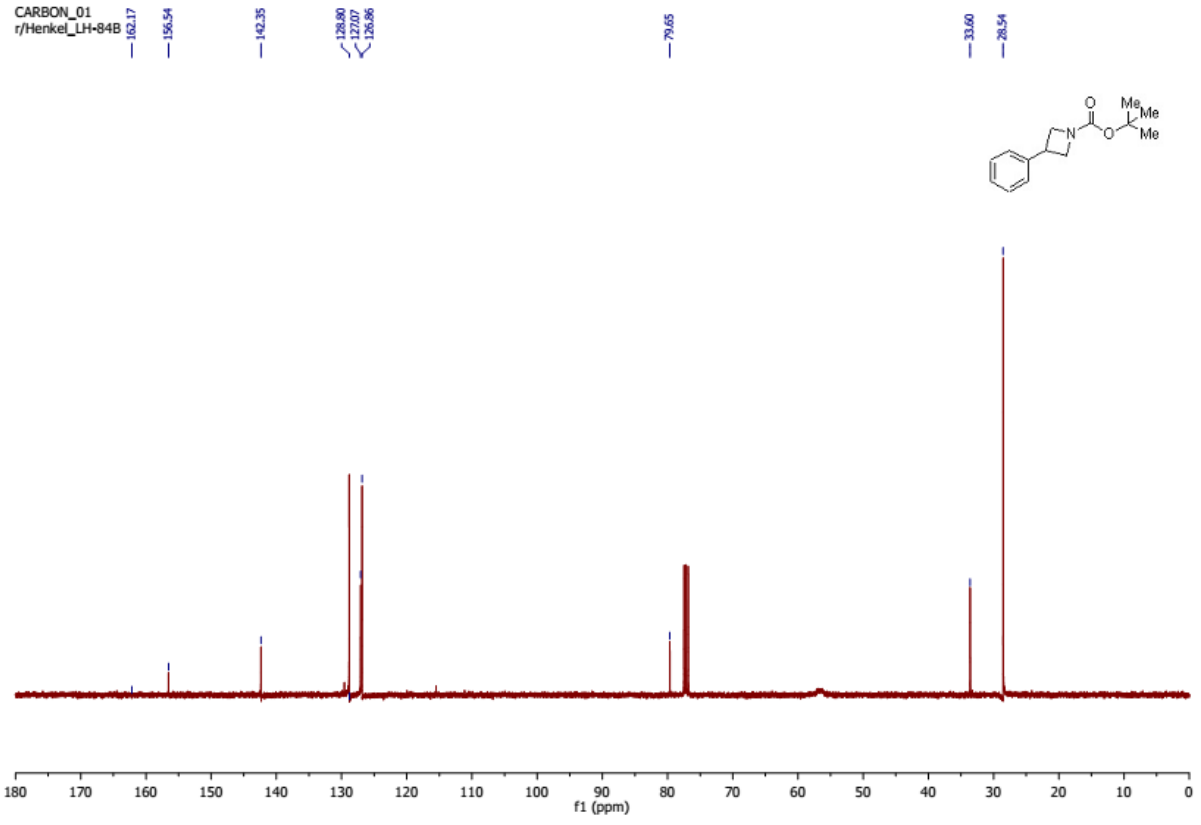
f1 (ppm)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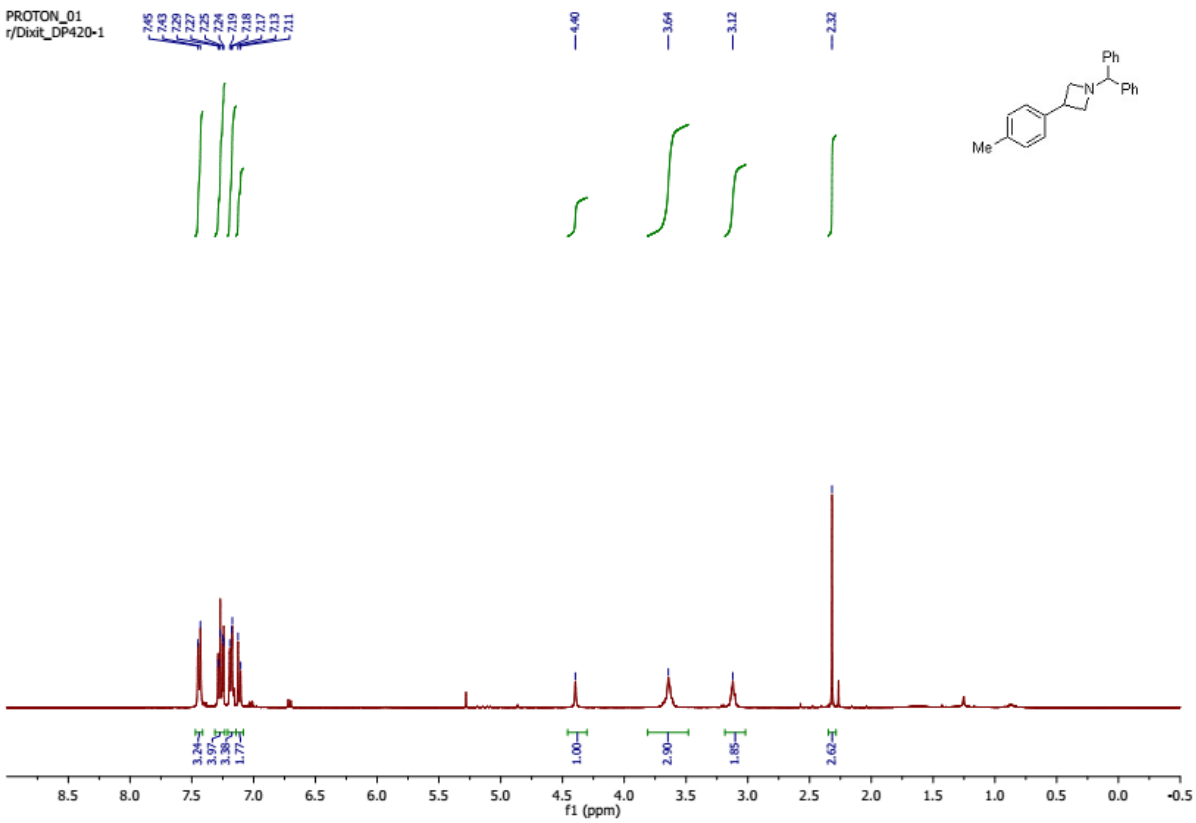
f1 (ppm)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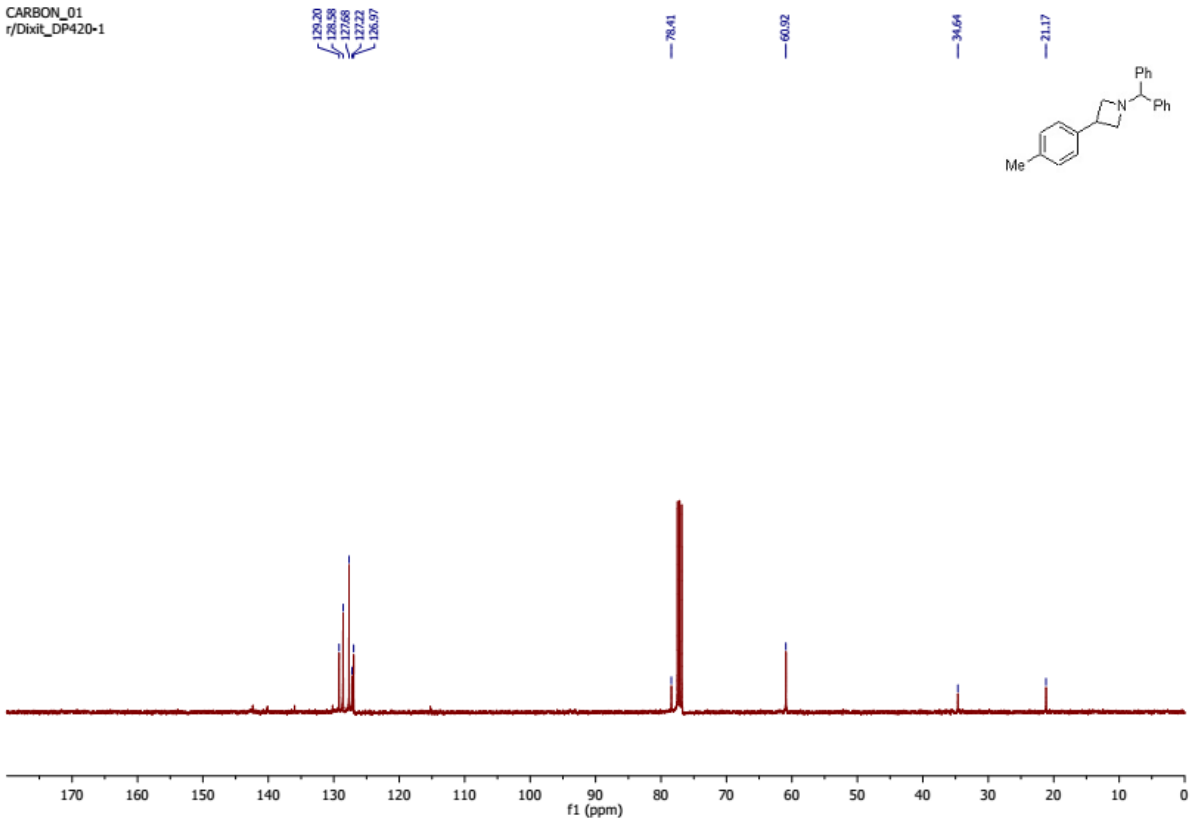
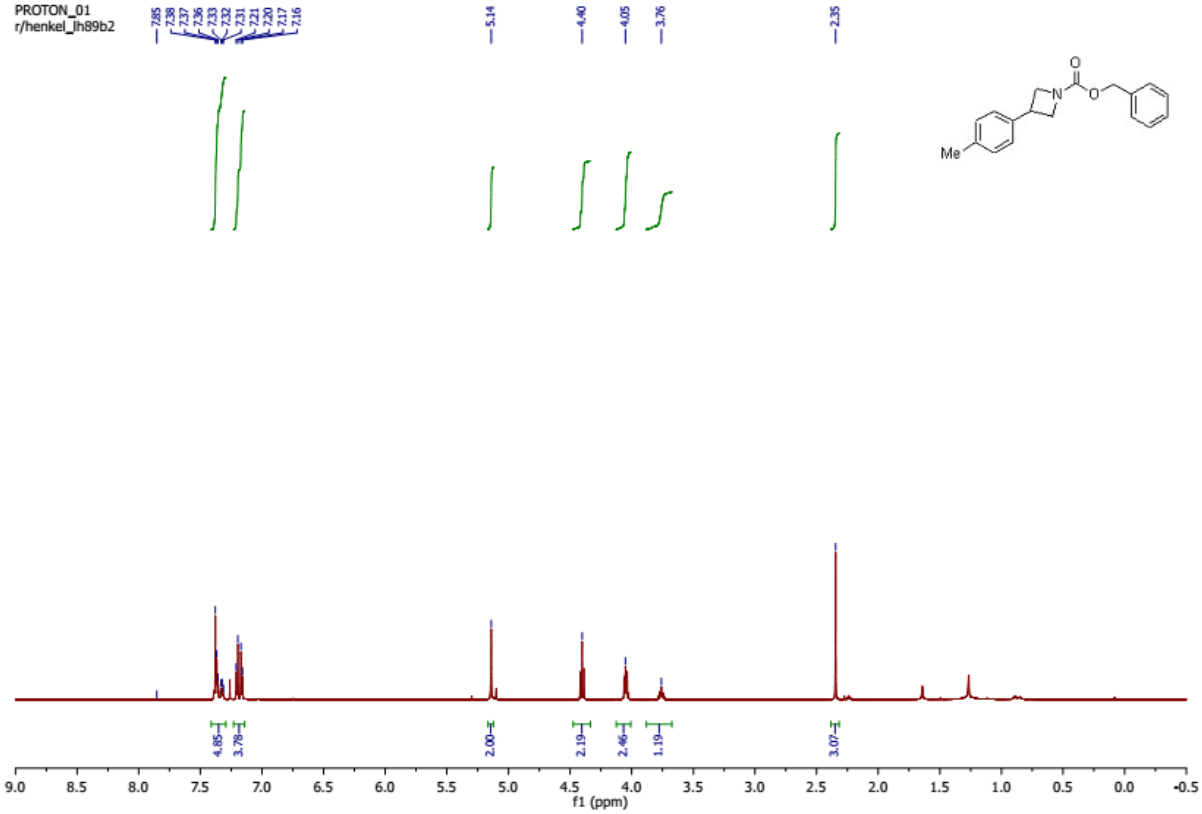
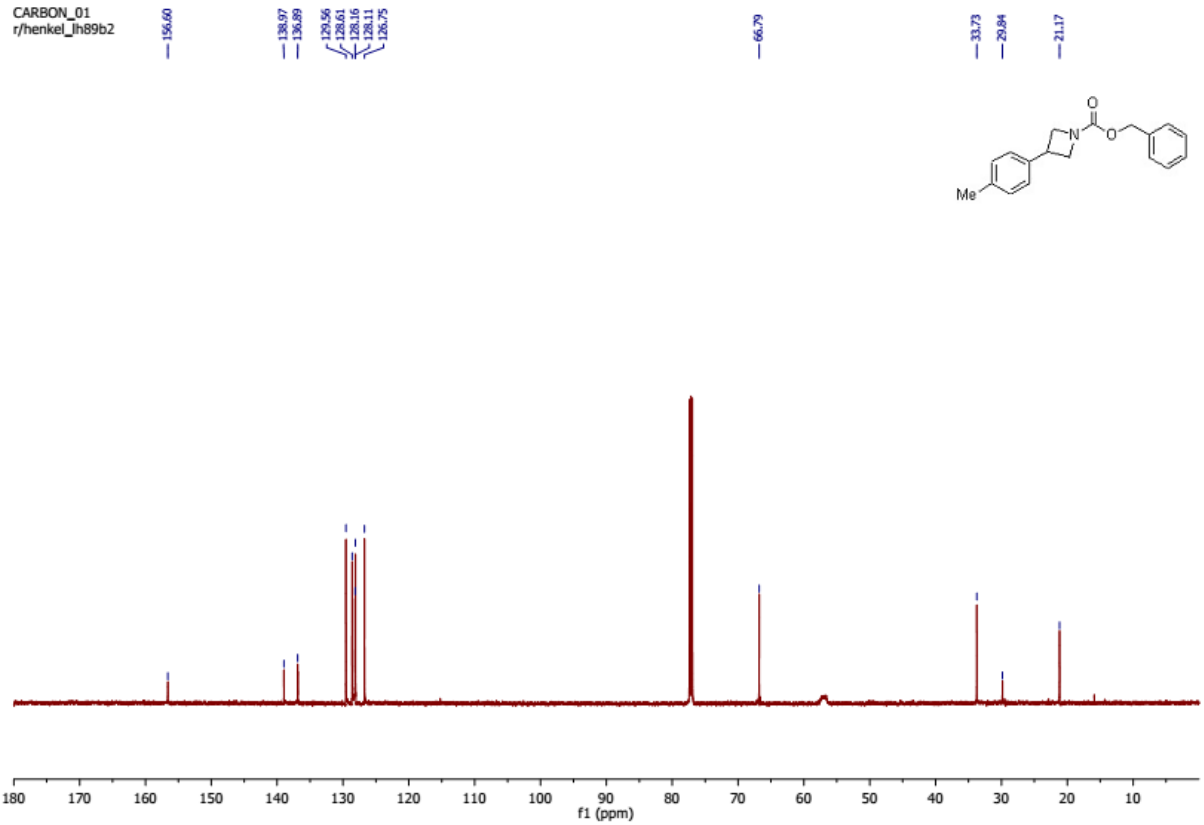
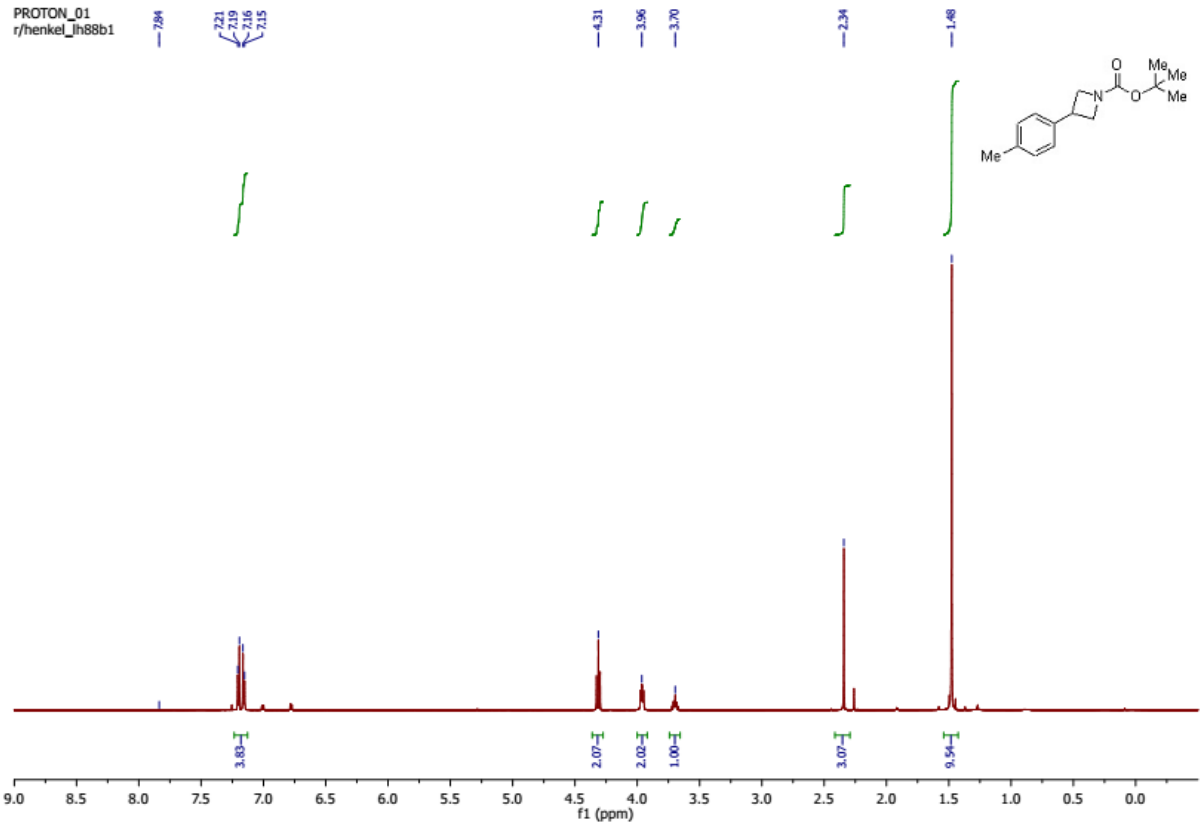
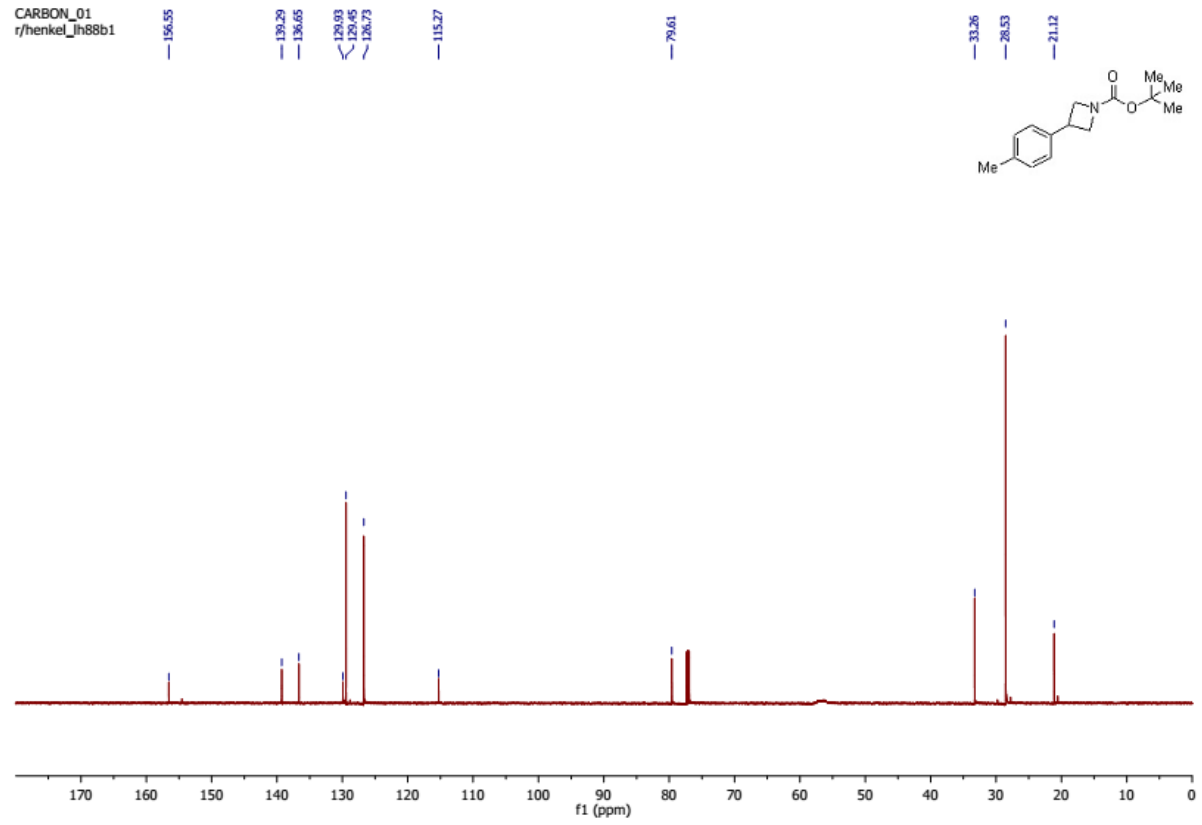
2.32
Ph
Ph
N
Me
3.24
3.97
3.38
1.77
1.00
2.90
1.85
2.62
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

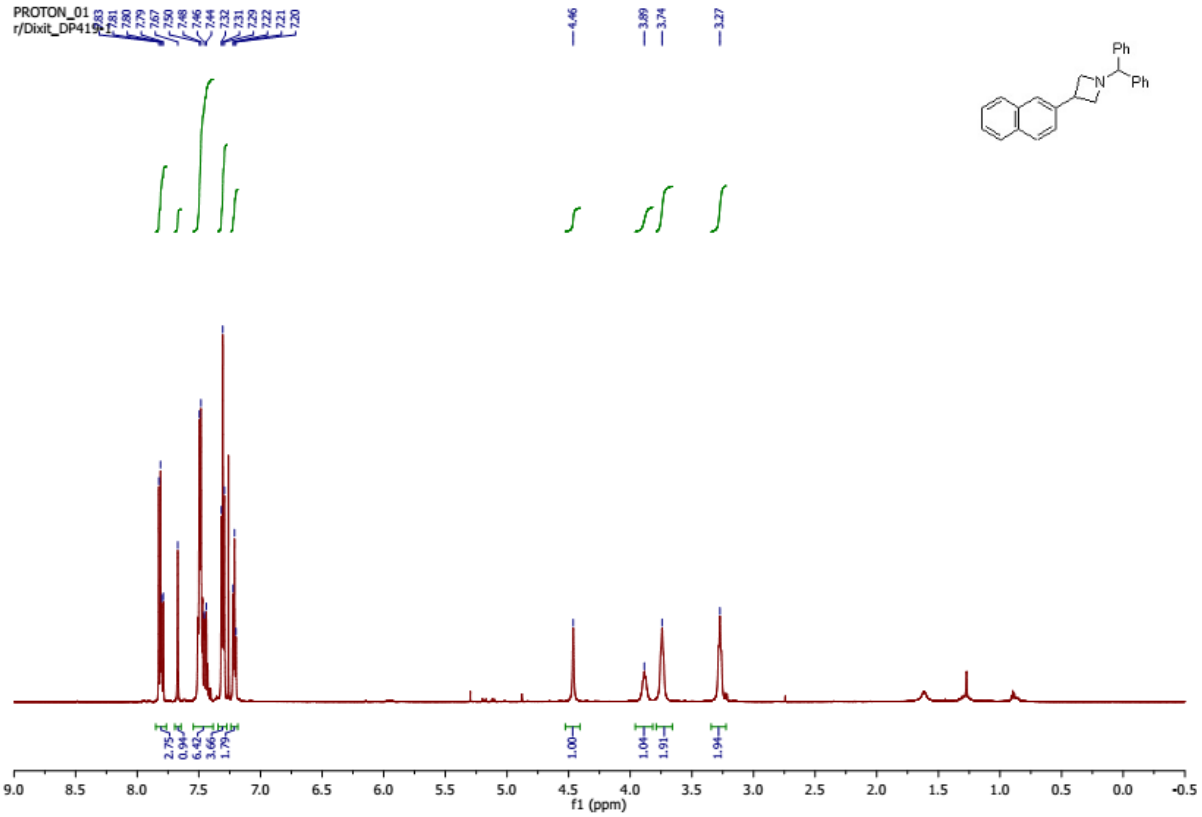

CARBON_01
r/Dixit_DP420-1
129.20
128.58
127.68
127.22
126.97
78.41
60.92
34.64
21.17
Ph
Ph
N
Me
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

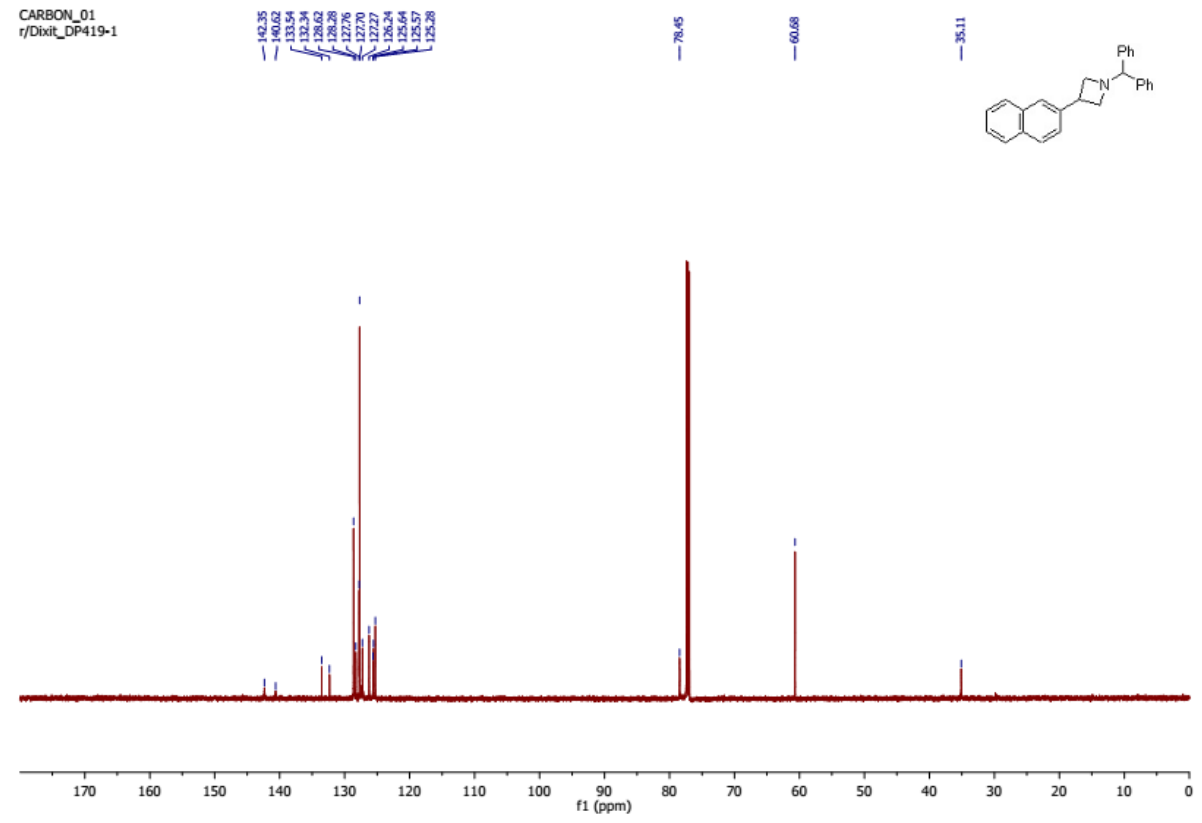

PROTON_01
r/henkel_lh89b2
7.85
7.38
7.37
7.36
7.33
7.32
7.31
7.21
7.20
7.17
7.16
5.14
4.40
4.05
3.76
2.35
Me
4.85
3.78
2.00
2.19
2.46
1.19
3.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

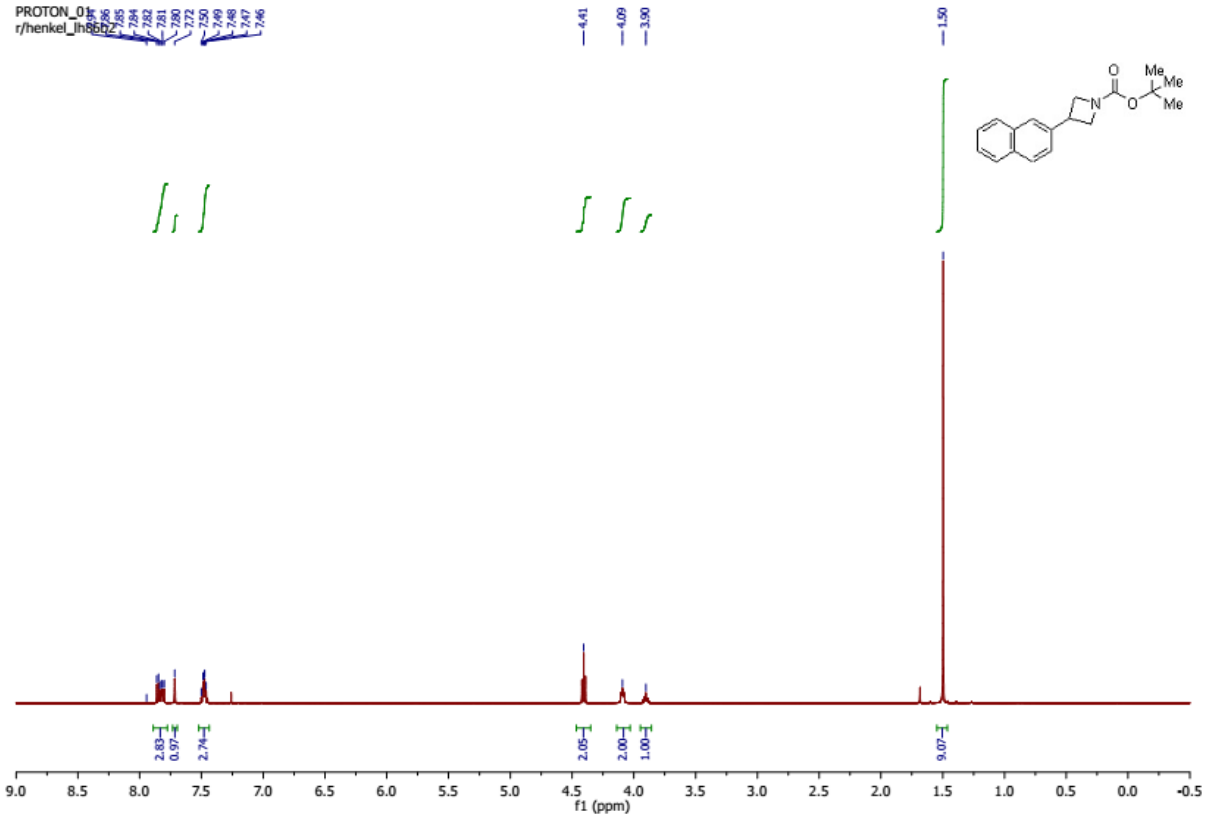

CARBON_01
r/henkel_lh89b2
156.60
138.97
136.89
129.56
128.61
128.16
128.11
126.75
66.79
33.73
29.84
21.17
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

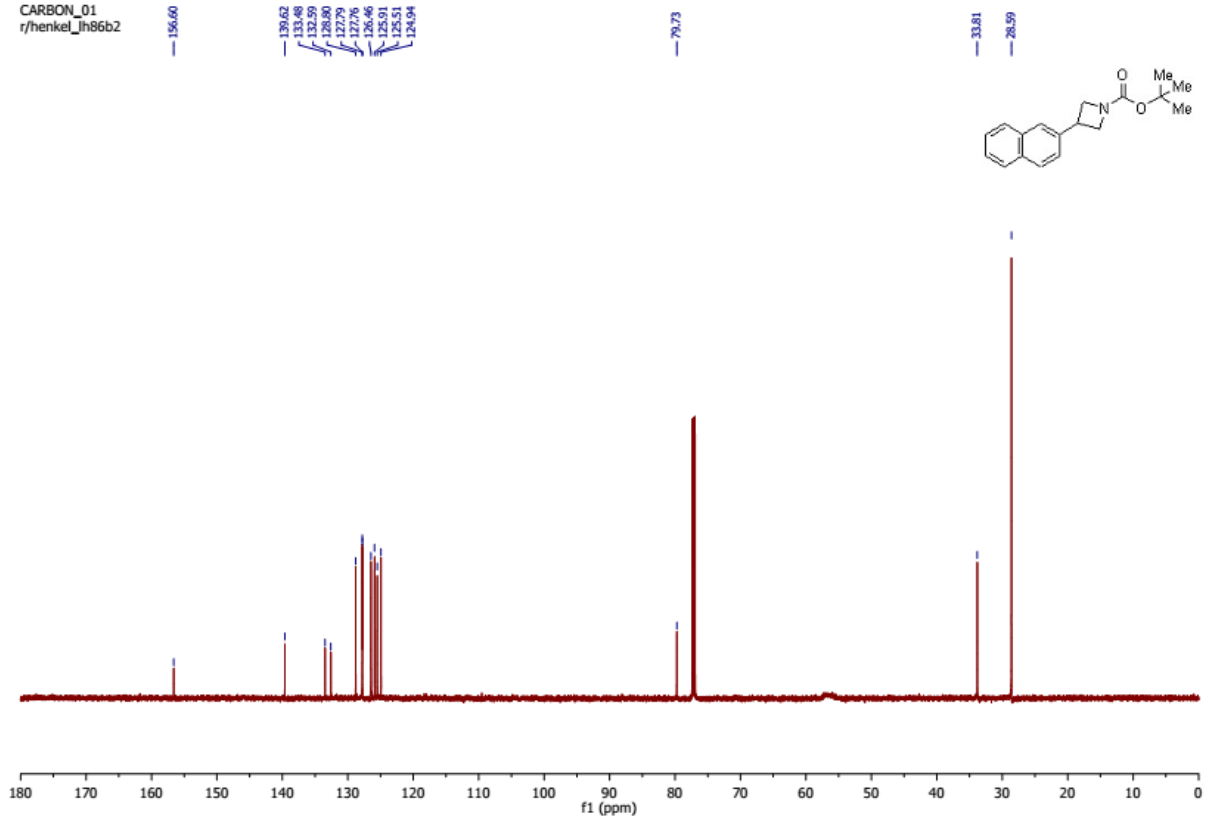

PROTON_01
r/henkel_lh88b1
7.84
7.21
7.19
7.16
7.15
4.31
3.96
3.70
2.34
1.48
3.83
2.07
2.02
1.00
3.07
9.54
f1 (ppm)

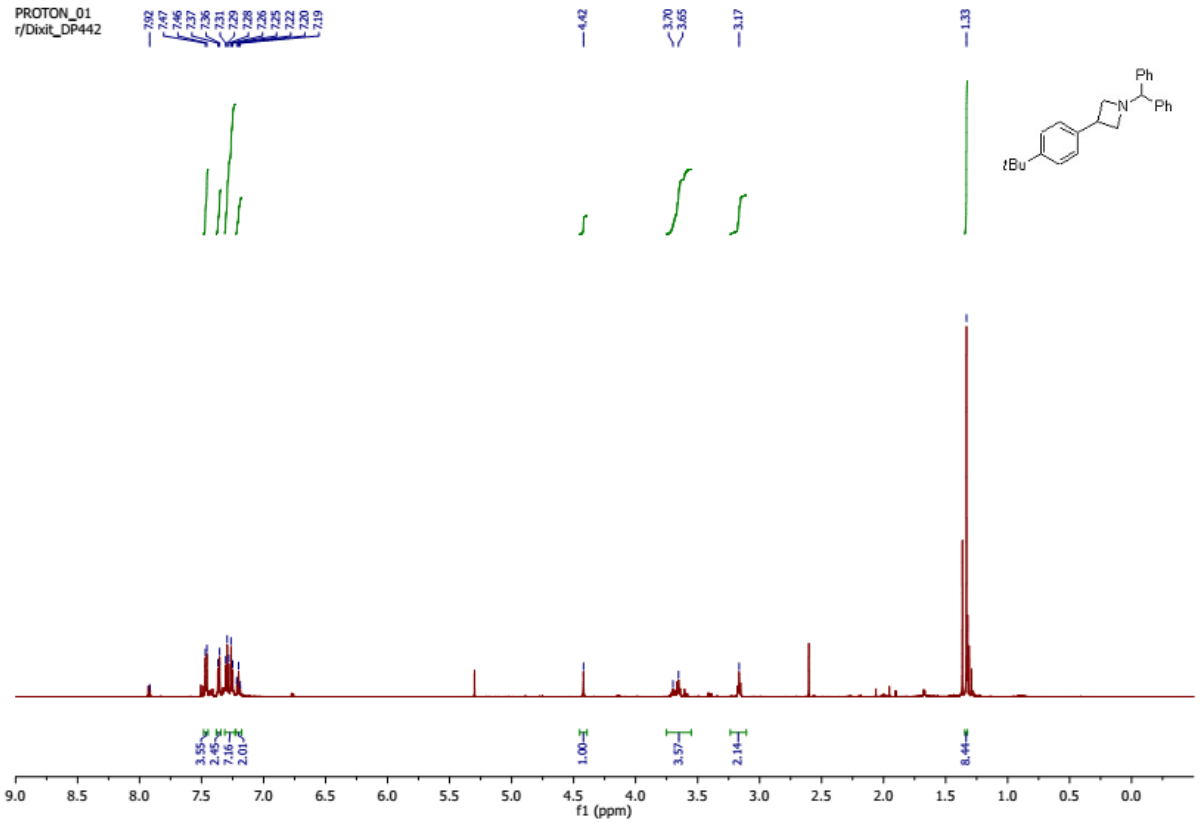

CARBON_01
r/henkel_lh88b1
156.55
139.29
136.65
129.93
129.45
126.73
115.27
79.61
33.26
28.53
21.12
f1 (ppm)

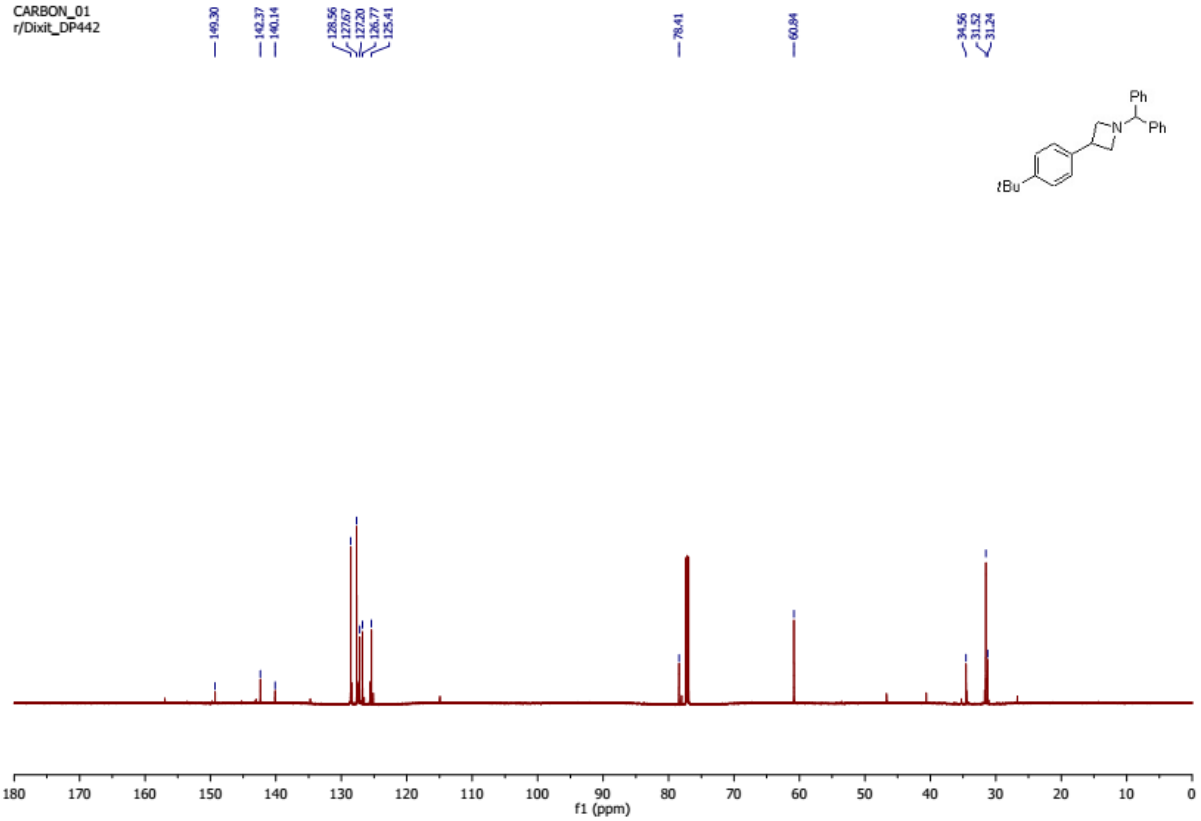

PROTON_01
r/Dixit_DP419-1
7.83
7.81
7.80
7.79
7.67
7.50
7.48
7.46
7.44
7.32
7.31
7.29
7.22
7.21
7.20
4.46
3.89
3.74
3.27
Ph
N
Ph
2.75
0.94
6.42
3.66
1.79
1.00
1.04
1.91
1.94
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

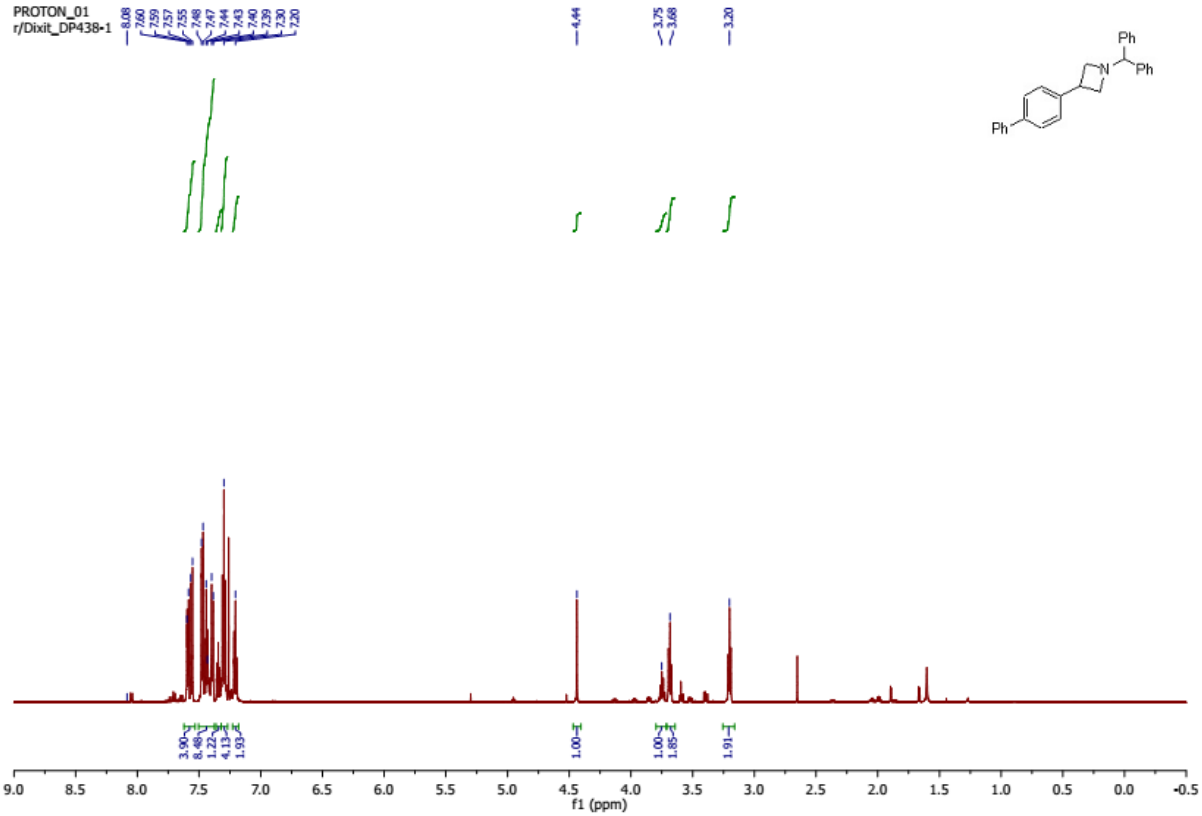

CARBON_01
r/Dixit_DP419-1
142.35
140.62
133.54
132.34
128.62
128.28
127.76
127.70
127.27
126.24
125.64
125.57
125.28
78.45
60.68
35.11
Ph
N
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

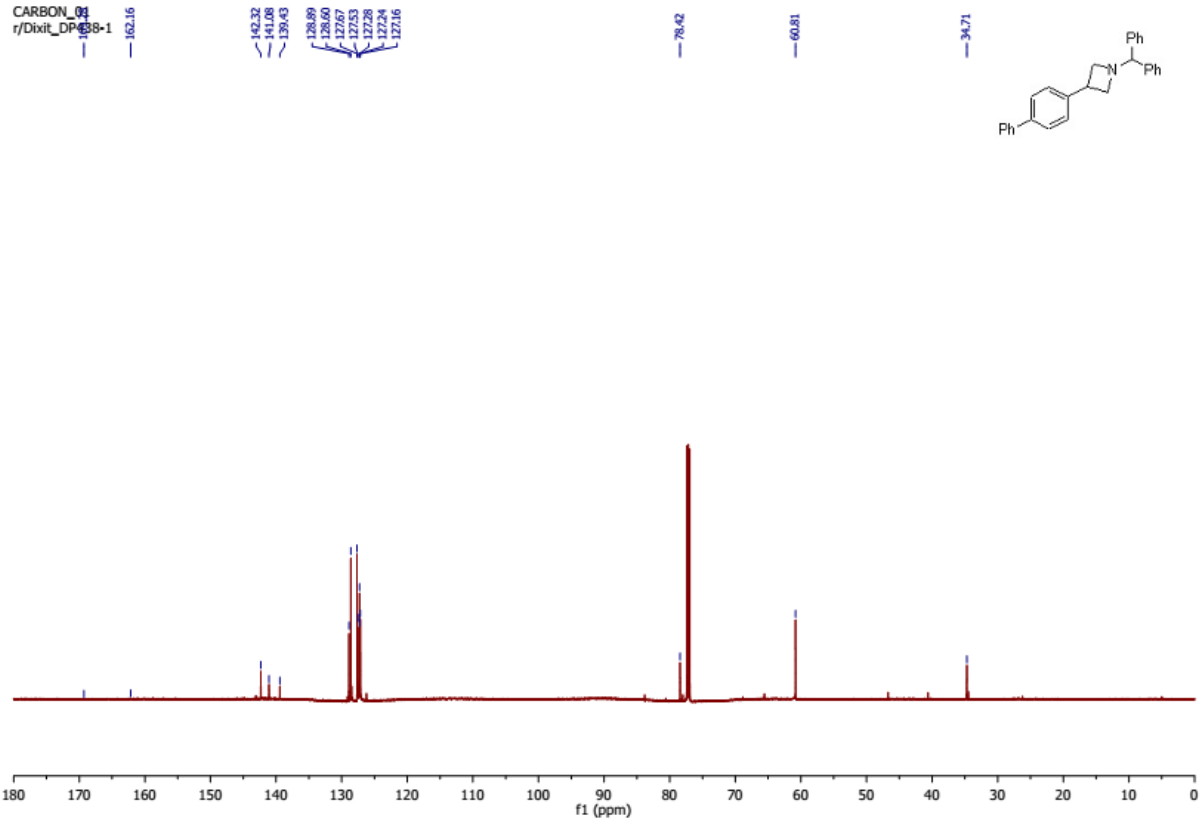

PROTON_01
r/henkel_lh86b2
7.96
7.85
7.84
7.82
7.81
7.80
7.72
7.50
7.49
7.48
7.47
7.46
4.41
4.09
3.90
1.50
Me
Me
Me
O
O
N
2.83
0.97
2.74
2.05
2.00
1.00
9.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

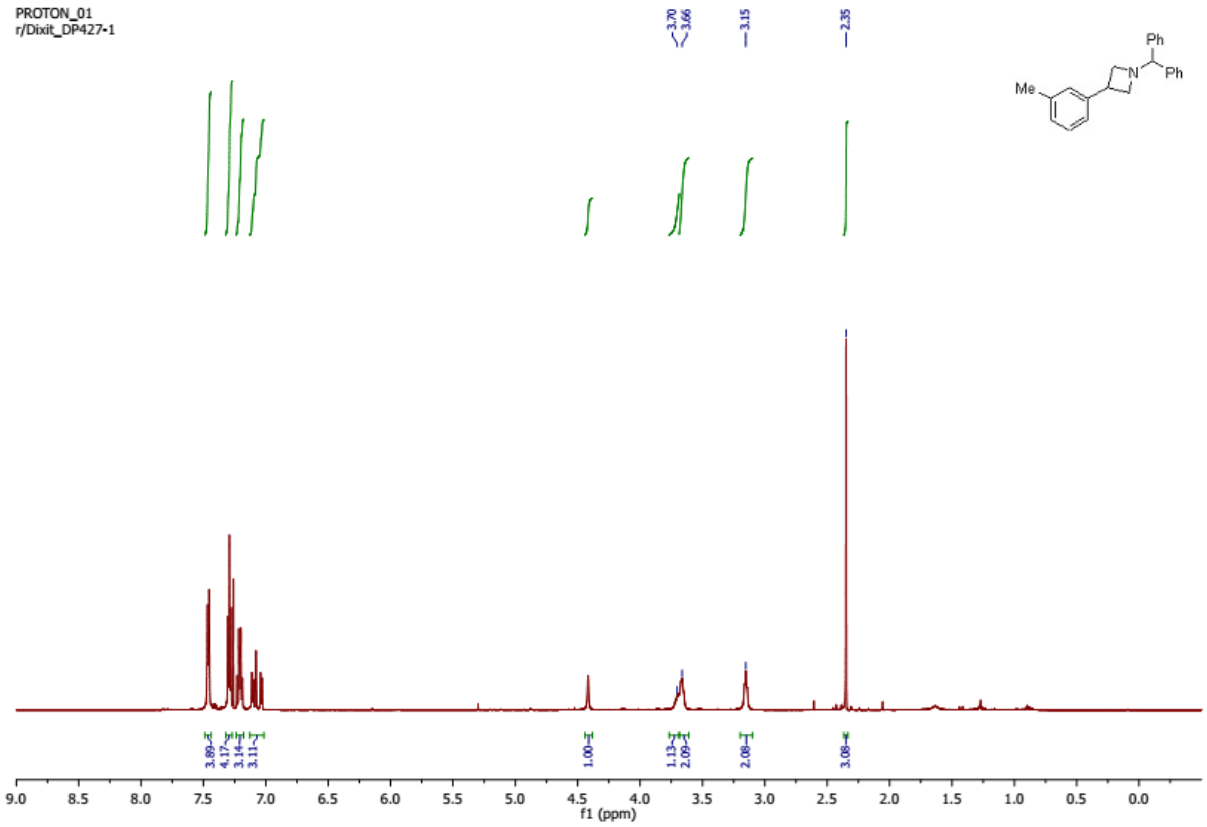

CARBON_01
r/henkel_lh86b2
156.60
139.62
133.48
132.59
128.80
127.79
127.76
126.46
125.91
125.51
124.94
79.73
33.81
28.59
Me
Me
Me
O
O
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

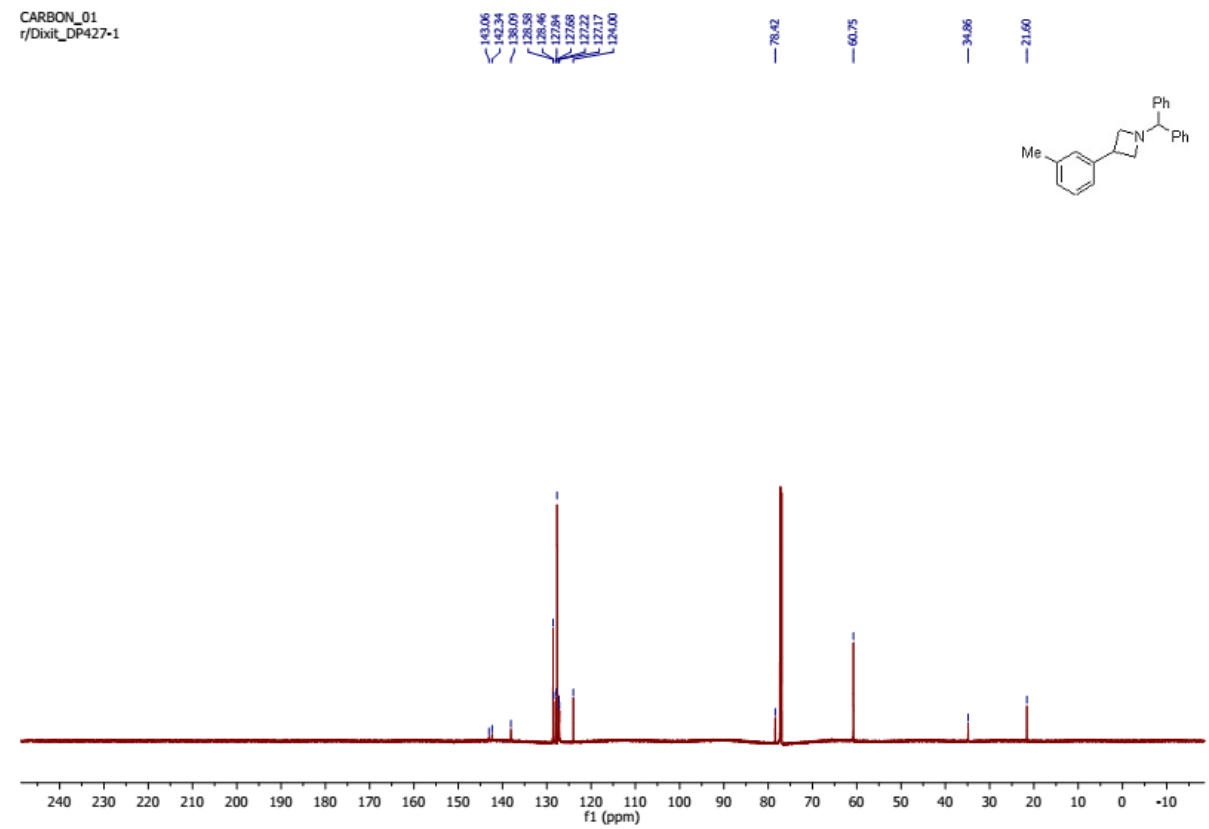

PROTON_01
r/Dixit_DP442
7.92
7.47
7.46
7.37
7.36
7.31
7.29
7.28
7.26
7.25
7.22
7.20
7.19
4.42
3.70
3.65
3.17
1.33
Ph
Ph
N
tBu
3.55
2.45
7.16
2.01
1.00
3.57
2.14
8.44
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

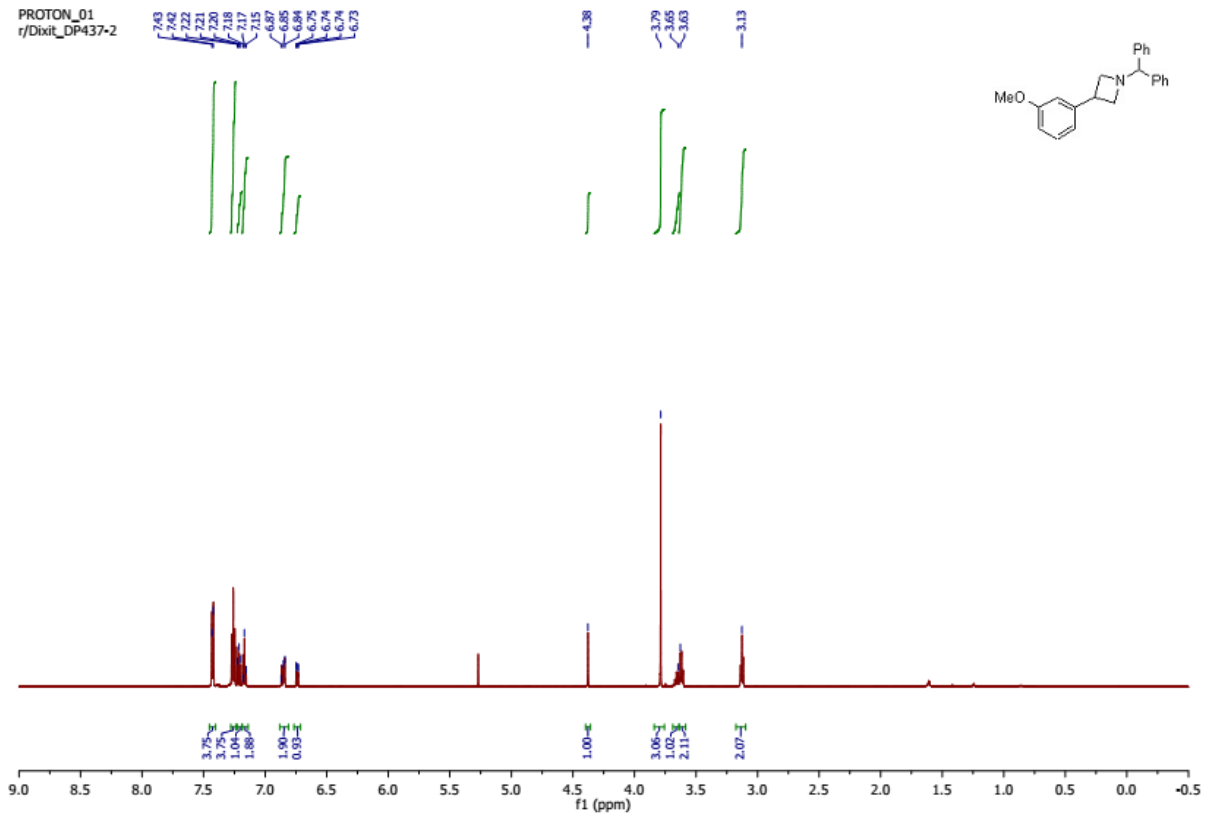

CARBON_01
r/Dixit_DP442
149.30
142.37
140.14
128.56
127.67
127.20
126.77
125.41
78.41
60.84
34.56
31.52
31.24
Ph
Ph
N
tBu
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

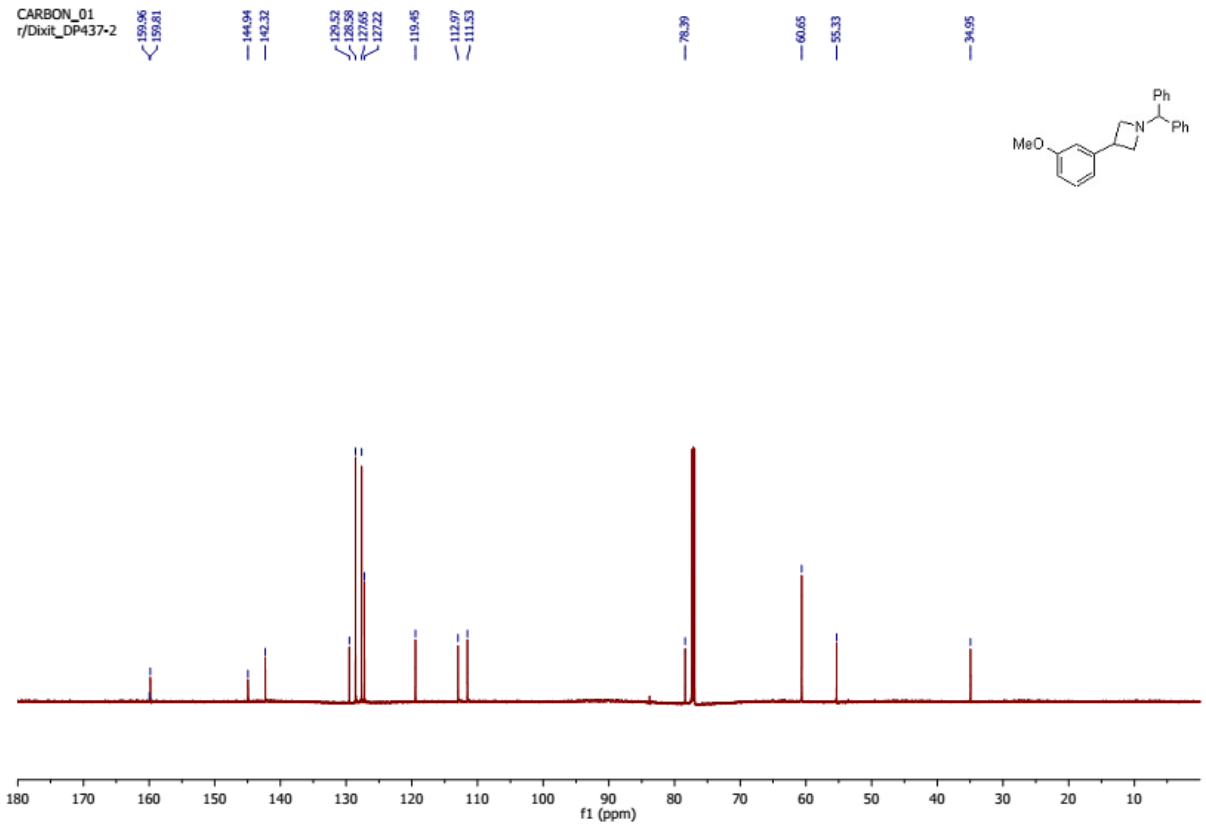

PROTON_01
r/Dixit_DP438-1
8.08
7.60
7.59
7.57
7.55
7.48
7.47
7.44
7.43
7.40
7.39
7.30
7.20
4.44
3.75
3.68
3.20
Ph
Ph
N
Ph
3.90
8.48
1.22
4.13
1.93
1.00
1.00
1.85
1.91
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

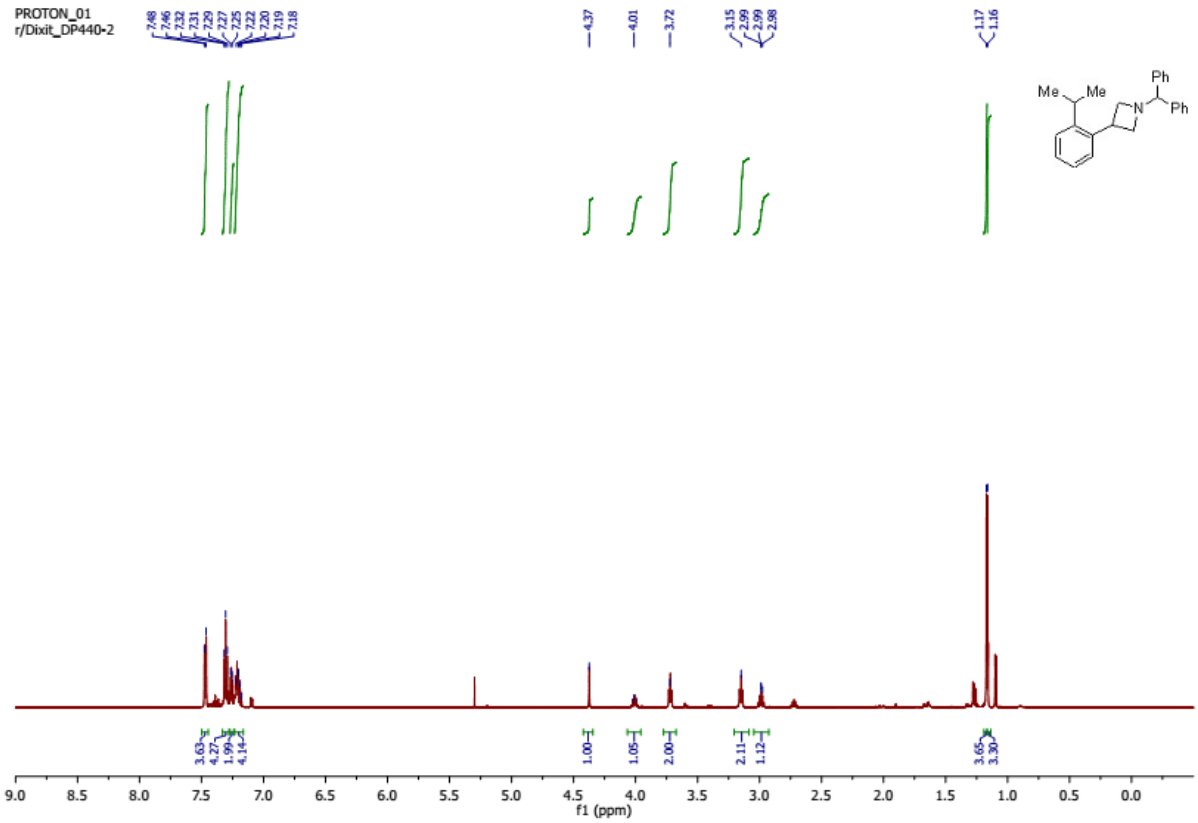

CARBON_01
r/Dixit_DP438-1
162.16
142.32
141.08
139.43
128.89
128.60
127.67
127.53
127.28
127.24
127.16
78.42
60.81
34.71
Ph
Ph
N
Ph
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

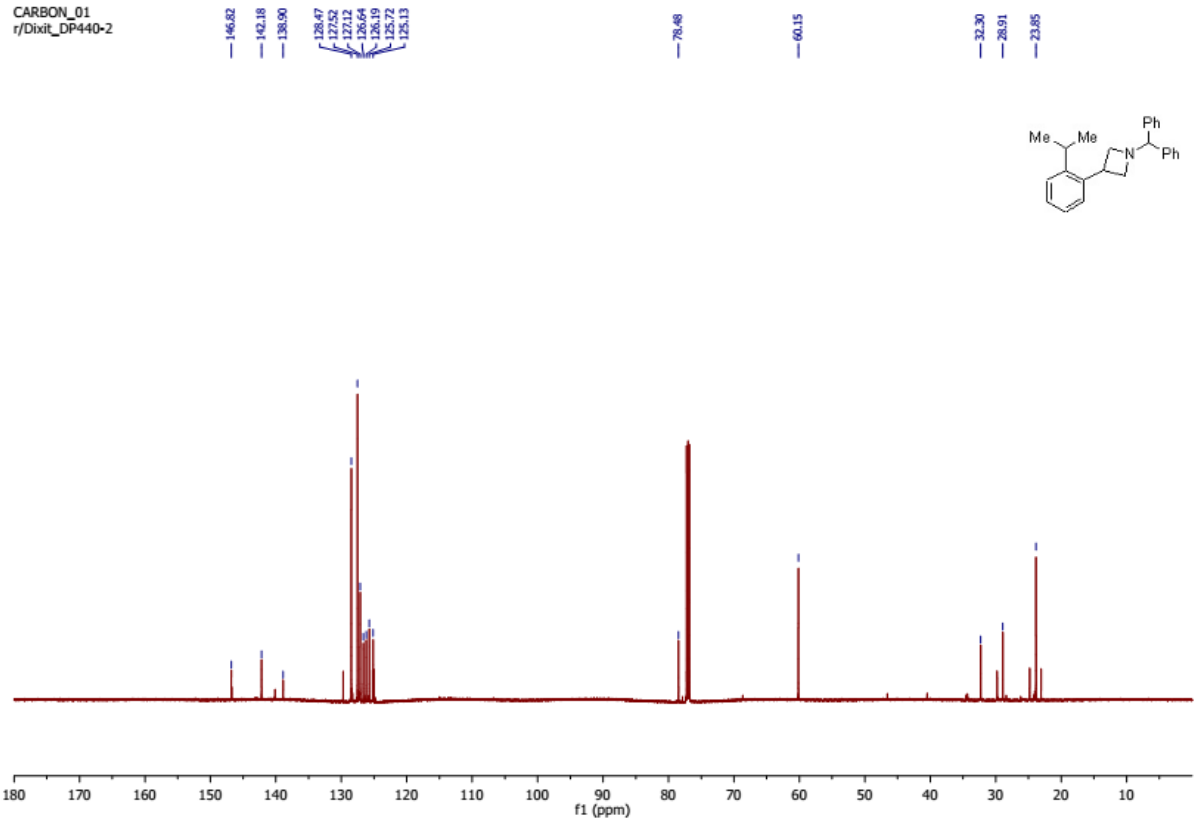

PROTON_01
r/Dixit_DP427-1
3.70
3.66
3.15
2.35
Ph
Ph
N
Me
3.89
4.17
3.14
3.11
1.00
1.13
2.09
2.08
3.08
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

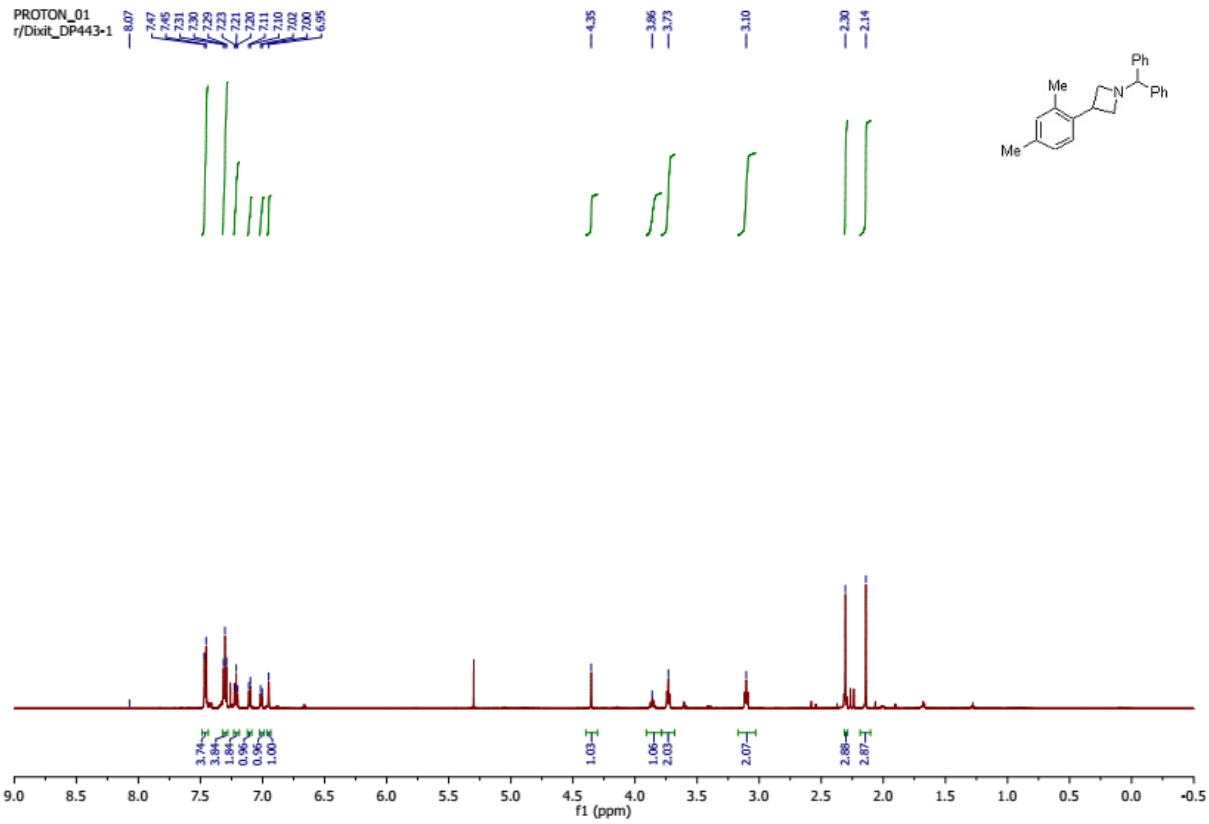

CARBON_01
r/Dixit_DP427-1
143.06
142.34
138.09
128.58
128.46
127.84
127.68
127.22
127.17
124.00
78.42
60.75
34.86
21.60
Ph
Ph
N
Me
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

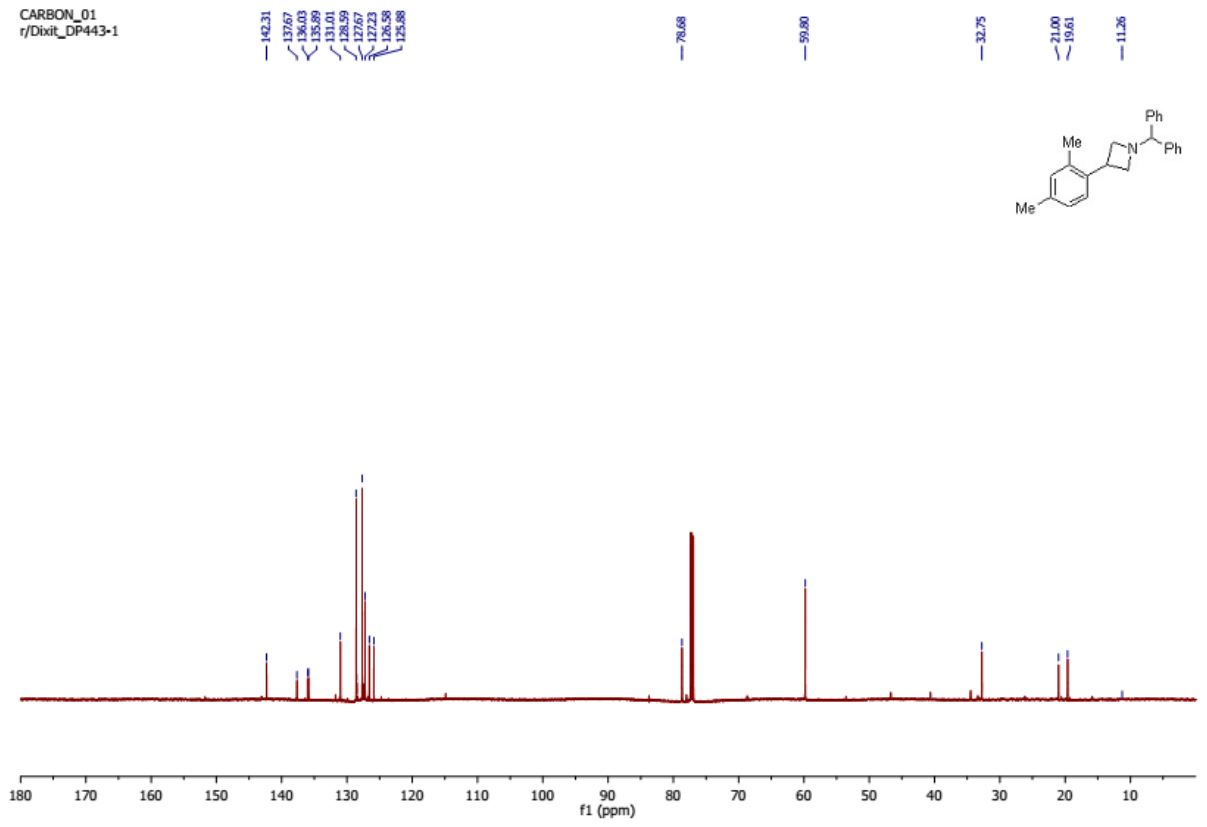

PROTON_01
r/Dixit_DP437-2
7.43
7.42
7.22
7.21
7.20
7.18
7.17
7.15
6.87
6.85
6.84
6.75
6.74
6.74
6.73
4.38
3.79
3.65
3.63
3.13
Ph
Ph
N
MeO
3.75
3.75
1.04
1.88
1.90
0.93
1.00
3.06
1.02
2.11
2.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

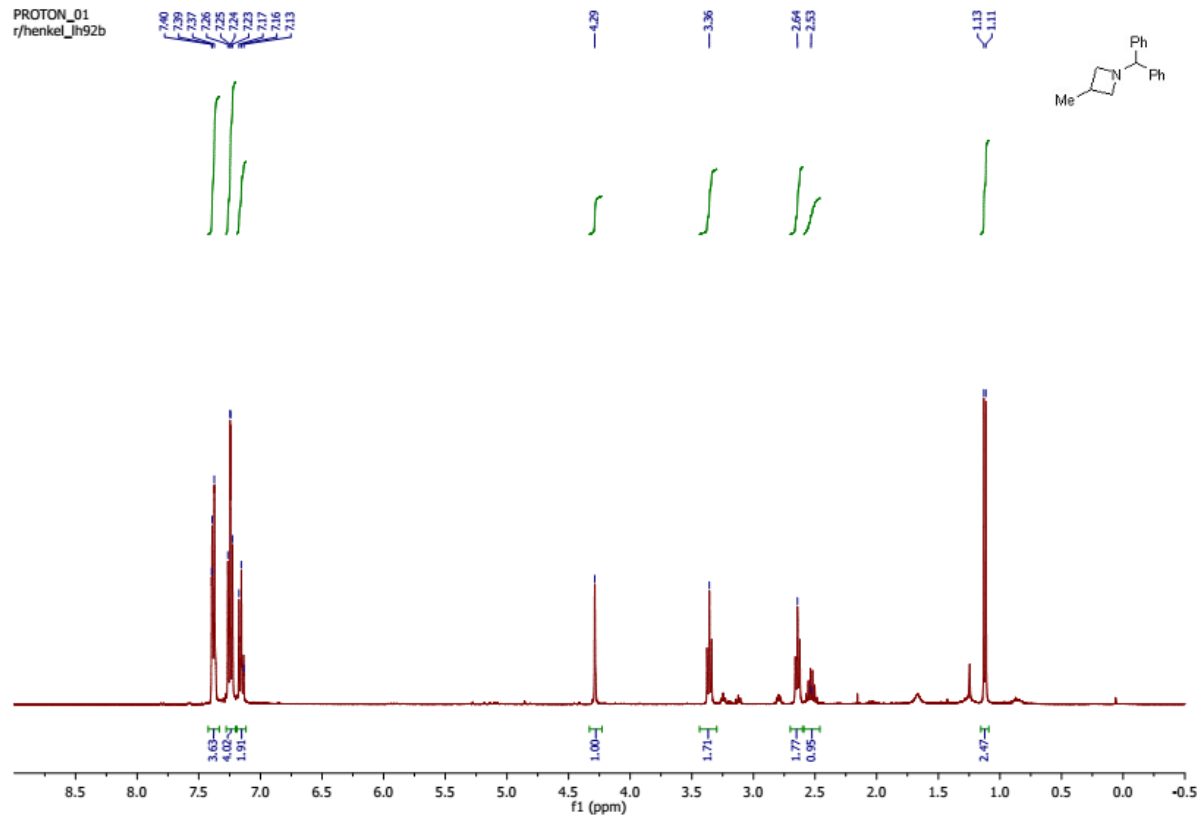

CARBON_01
r/Dixit_DP437-2
159.96
159.81
144.94
142.32
129.52
128.58
127.65
127.22
119.45
112.97
111.53
78.39
60.65
55.33
34.95
Ph
Ph
N
MeO
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

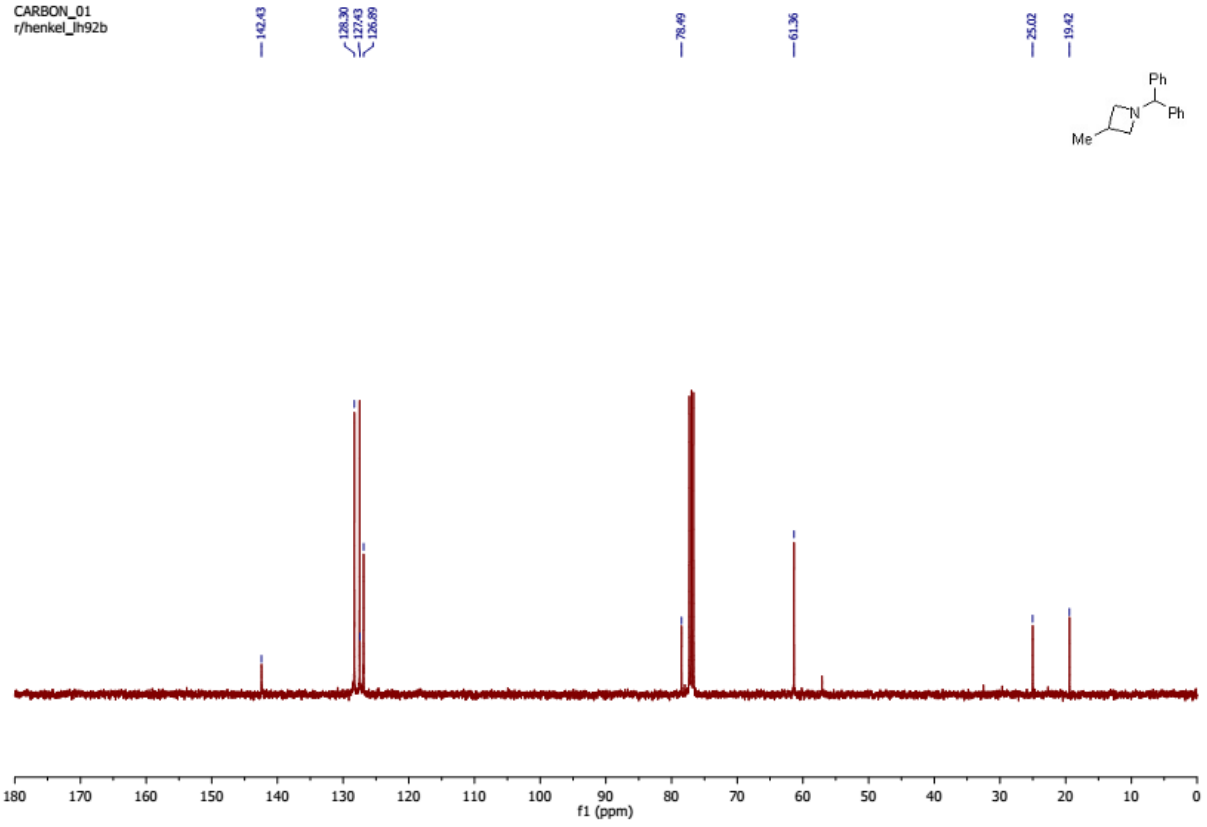

PROTON_01
r/Dixit_DP440-2
7.48
7.46
7.32
7.31
7.29
7.27
7.25
7.22
7.20
7.19
7.18
4.37
4.01
3.72
3.15
2.99
2.99
2.98
1.17
1.16
Me
Me
Ph
Ph
N
3.63
4.27
1.99
4.14
1.00
1.05
2.00
2.11
1.12
3.65
3.30
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

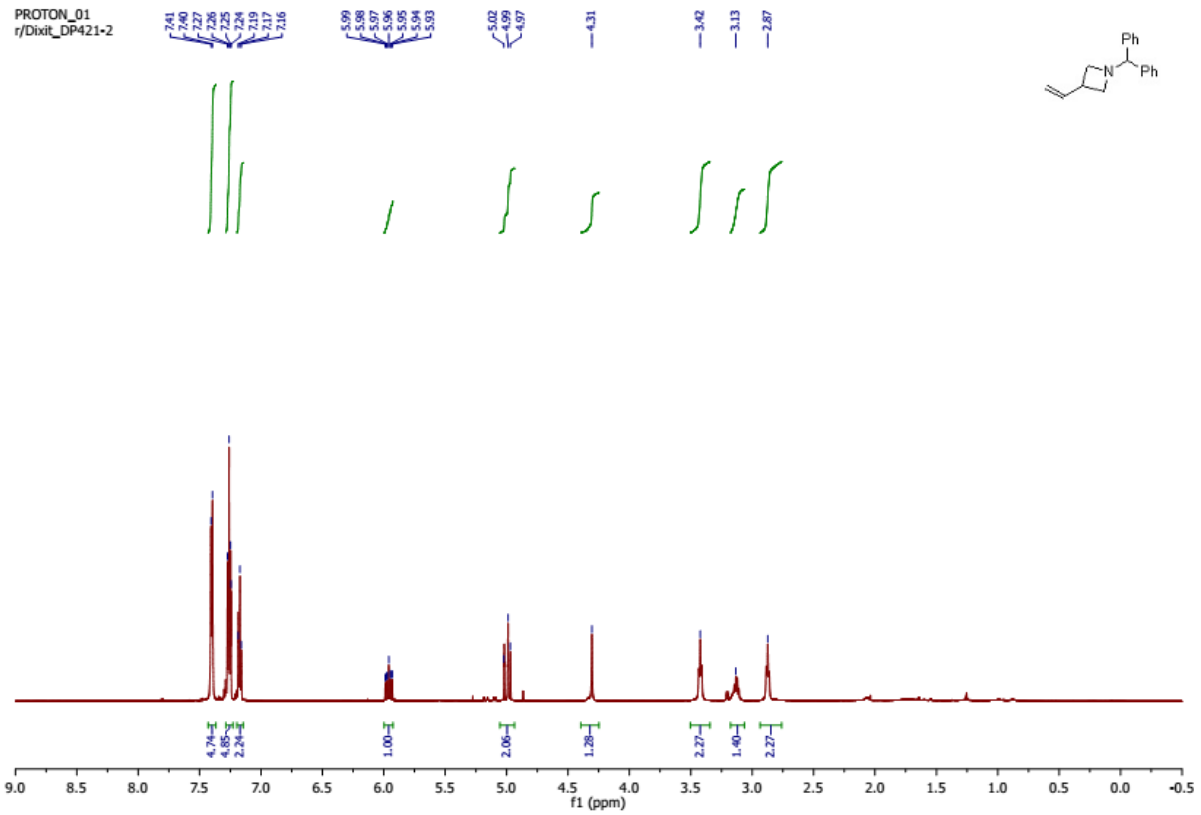

CARBON_01
r/Dixit_DP440-2
146.82
142.18
138.90
128.47
127.52
127.12
126.64
126.19
125.72
125.13
78.48
60.15
32.30
28.91
23.85
Me
Me
Ph
Ph
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

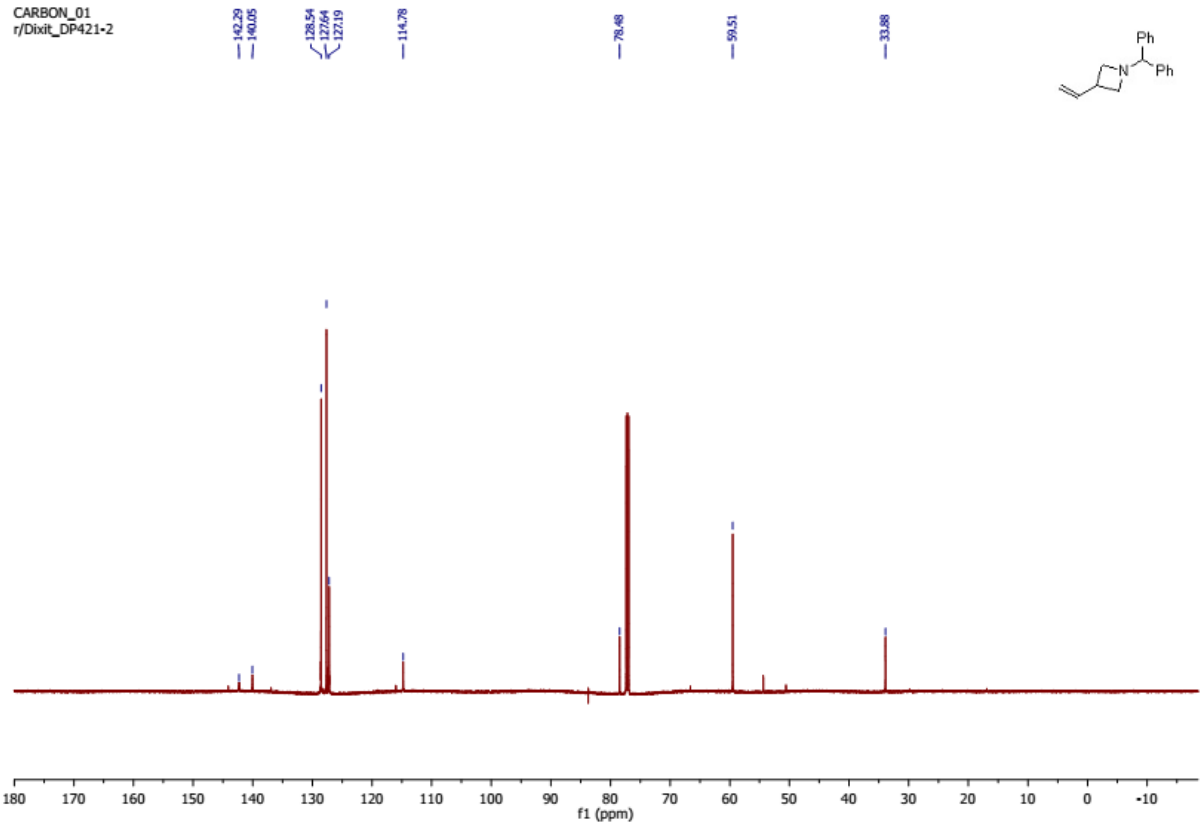

PROTON_01
r/Dixit_DP443-1
8.07
7.47
7.45
7.31
7.30
7.29
7.23
7.21
7.20
7.11
7.10
7.02
7.00
6.95
4.35
3.86
3.73
3.10
2.30
2.14
Ph
Me
N
Ph
Me
3.74
3.84
1.84
0.96
0.96
1.00
1.03
1.06
2.03
2.07
2.88
2.87
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

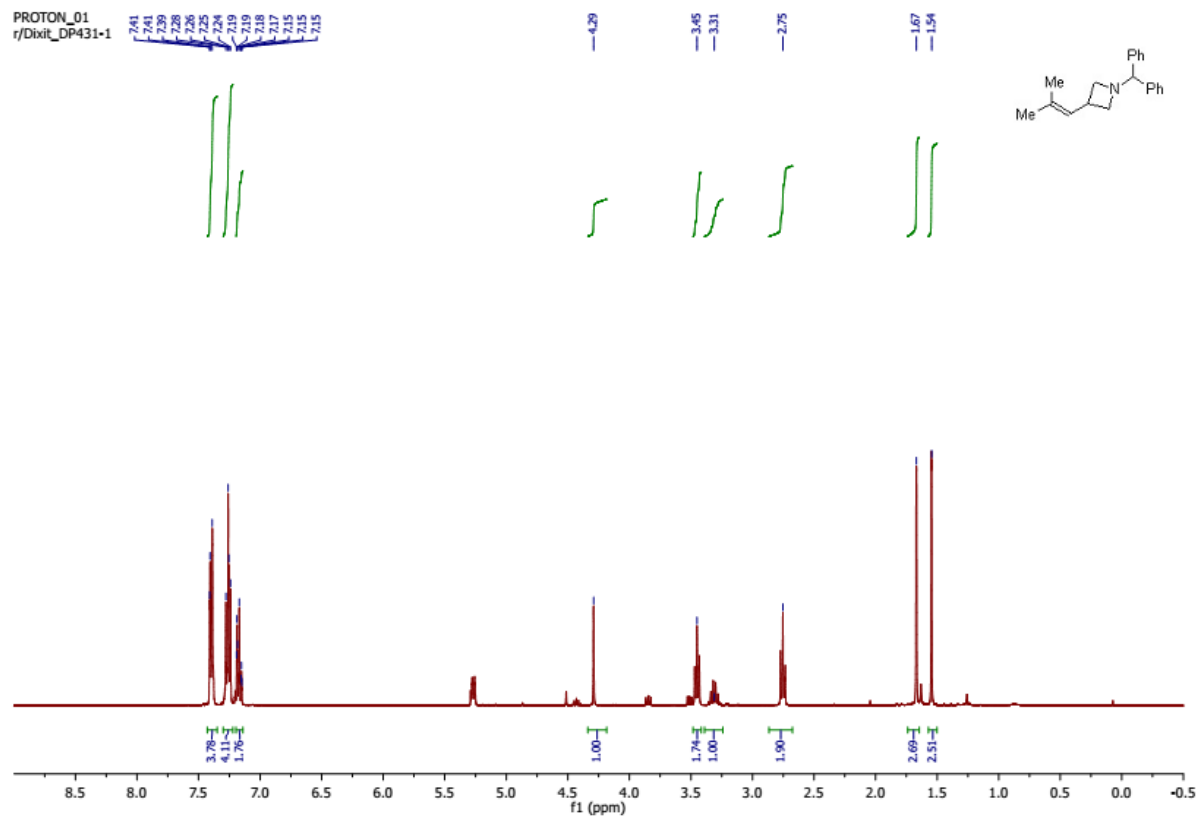

CARBON_01
r/Dixit_DP443-1
142.31
137.67
136.03
135.89
131.01
128.59
127.67
127.23
126.58
125.88
78.68
59.80
32.75
21.00
19.61
11.26
Ph
Me
N
Ph
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

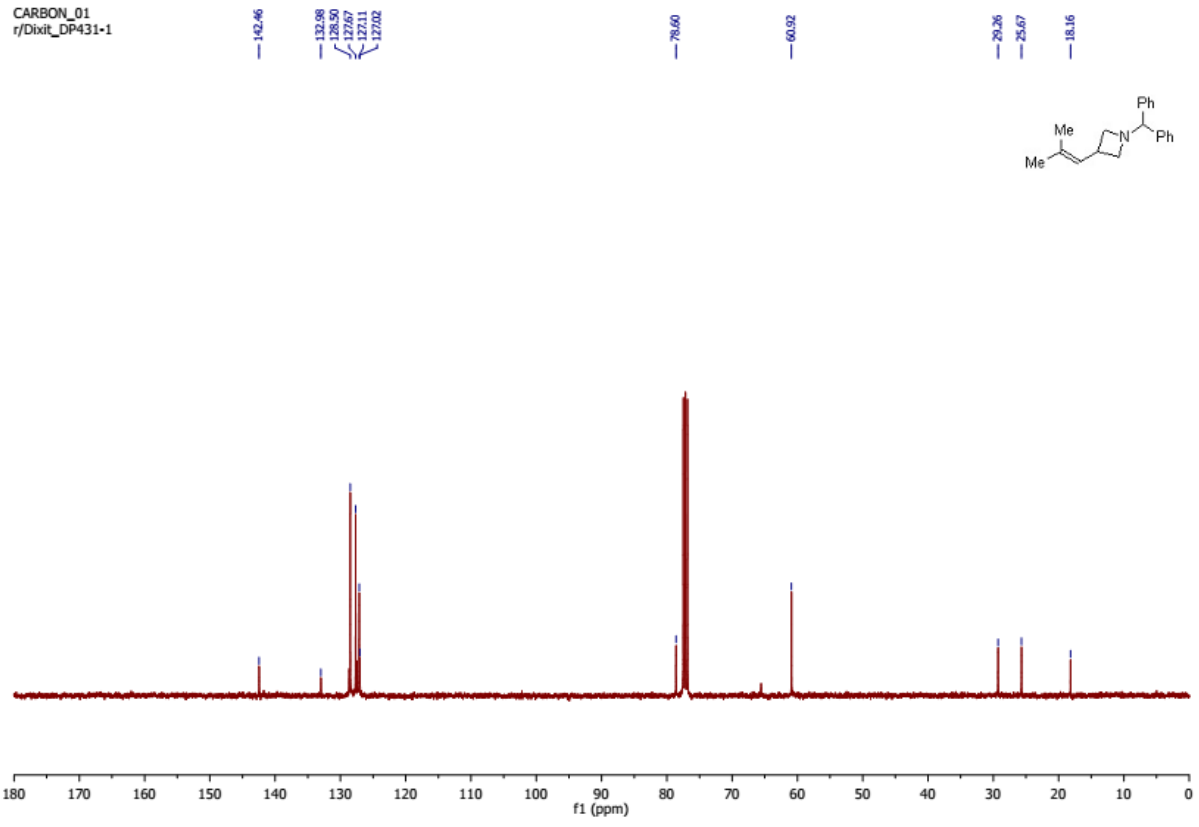

PROTON_01
r/henkel_lh92b
7.40
7.39
7.37
7.26
7.25
7.24
7.23
7.17
7.16
7.13
4.29
3.36
2.64
2.53
1.13
1.11
Ph
Ph
N
Me
3.63
4.02
1.91
1.00
1.71
1.77
0.95
2.47
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

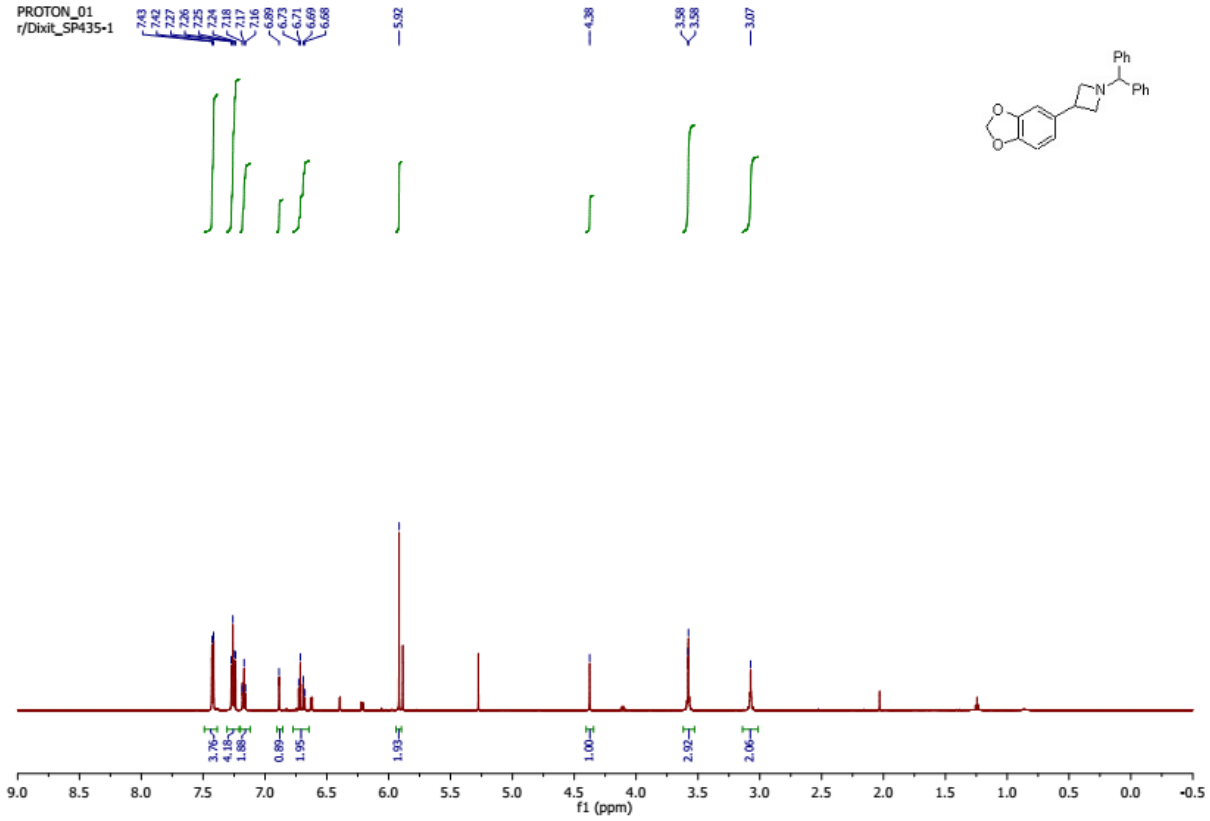

CARBON_01
r/henkel_lh92b
142.43
128.30
127.43
126.89
78.49
61.36
25.02
19.42
Ph
Ph
N
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

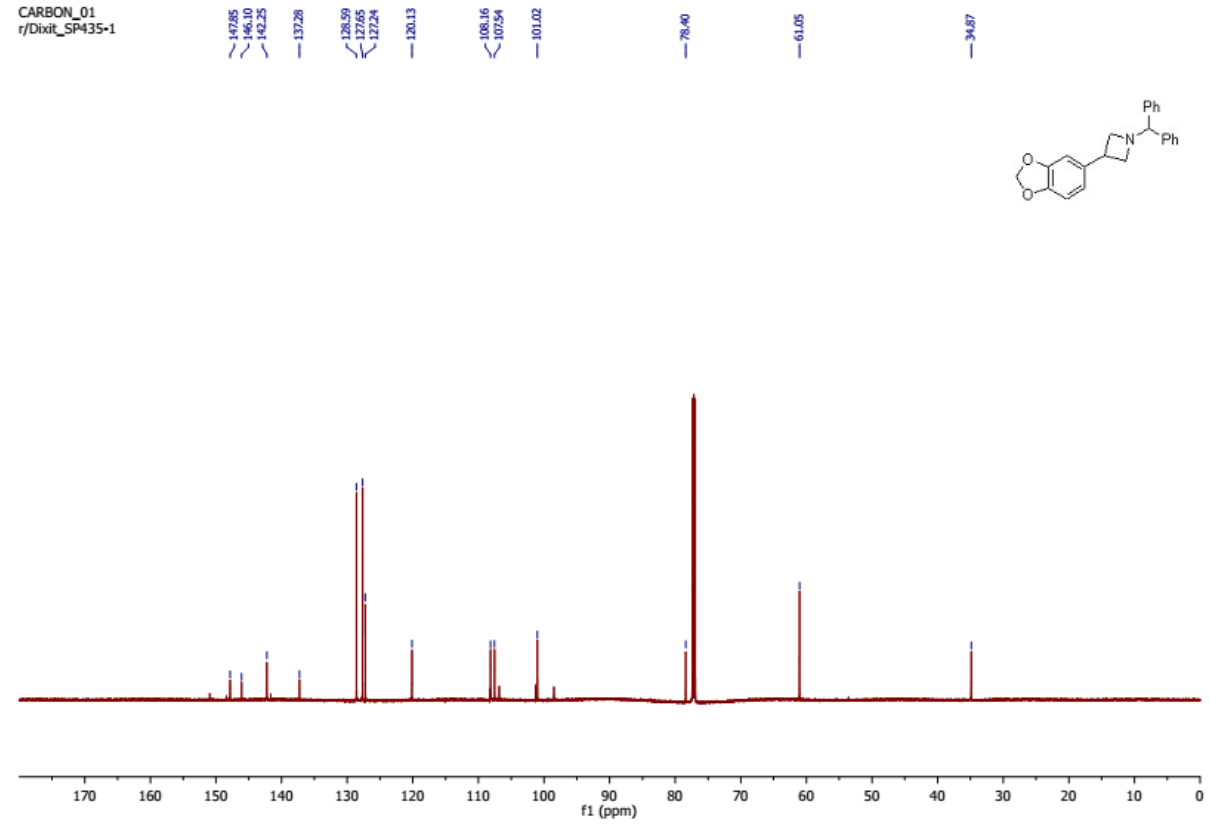

PROTON_01
r/Dixit_DP421-2
7.41
7.40
7.27
7.26
7.25
7.24
7.19
7.17
7.16
5.99
5.98
5.97
5.96
5.95
5.94
5.93
5.02
4.99
4.97
4.31
3.42
3.13
2.87
Ph
Ph
N
4.74
4.85
2.24
1.00
2.06
1.28
2.27
1.40
2.27
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

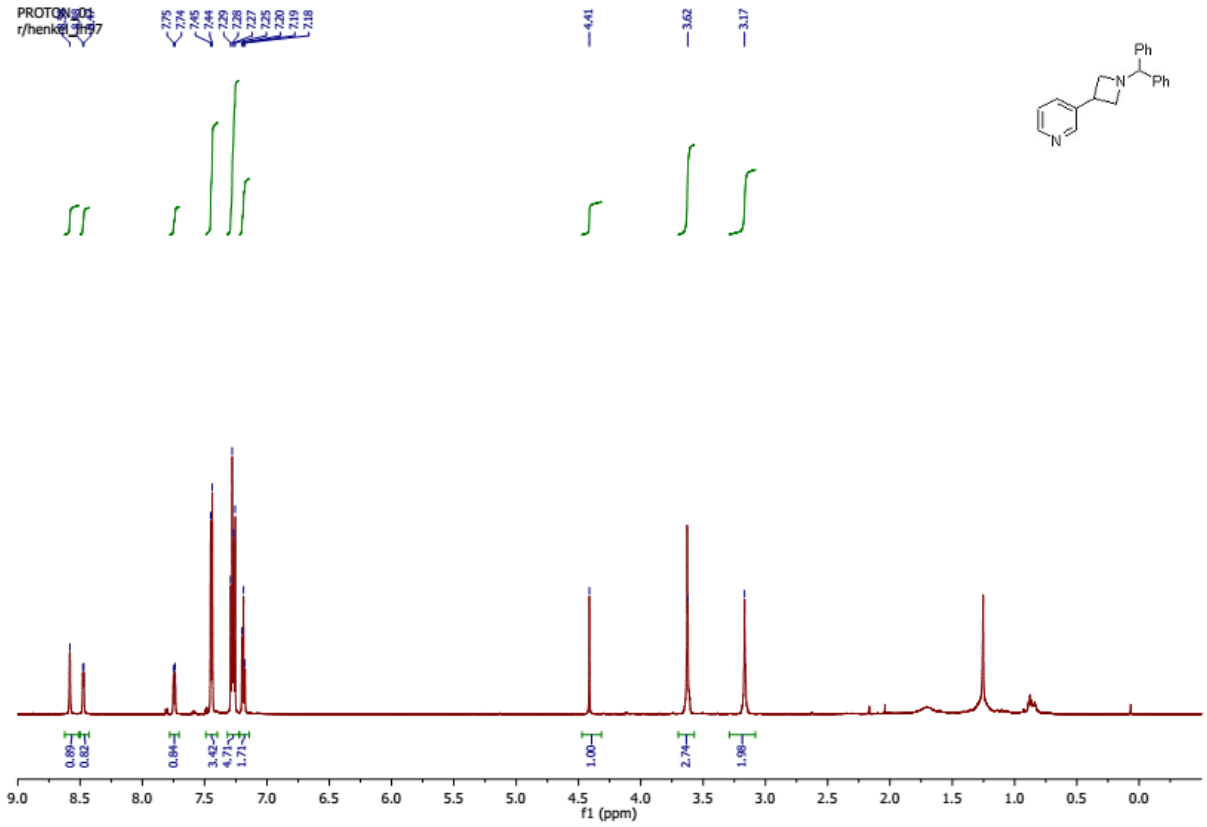

CARBON_01
r/Dixit_DP421-2
142.29
140.05
128.54
127.64
127.19
114.78
78.48
59.51
33.88
Ph
Ph
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

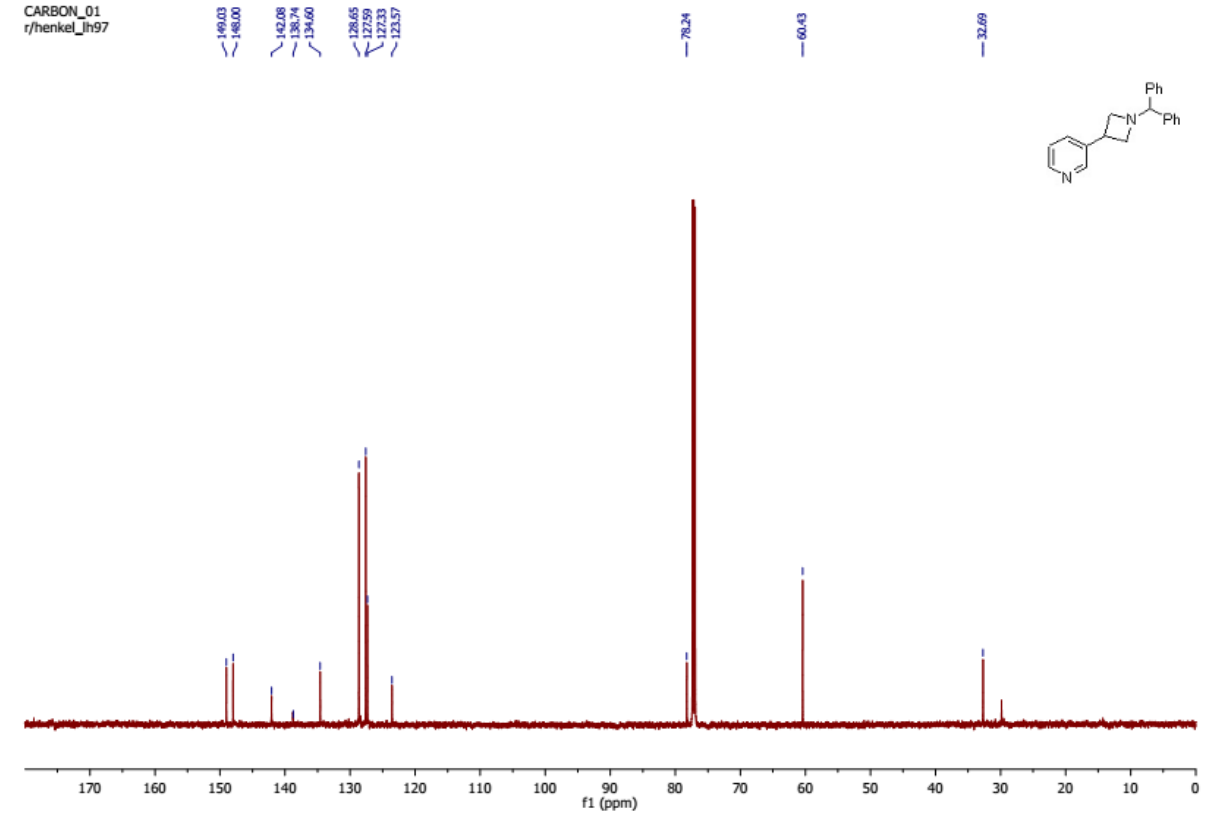

PROTON_01
r/Dixit_DP431-1
7.41
7.41
7.39
7.28
7.26
7.25
7.24
7.19
7.18
7.17
7.15
7.15
4.29
3.45
3.31
2.75
1.67
1.54
Ph
Me
N
Ph
Me
3.78
4.11
1.76
1.00
1.74
1.00
1.90
2.69
2.51
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

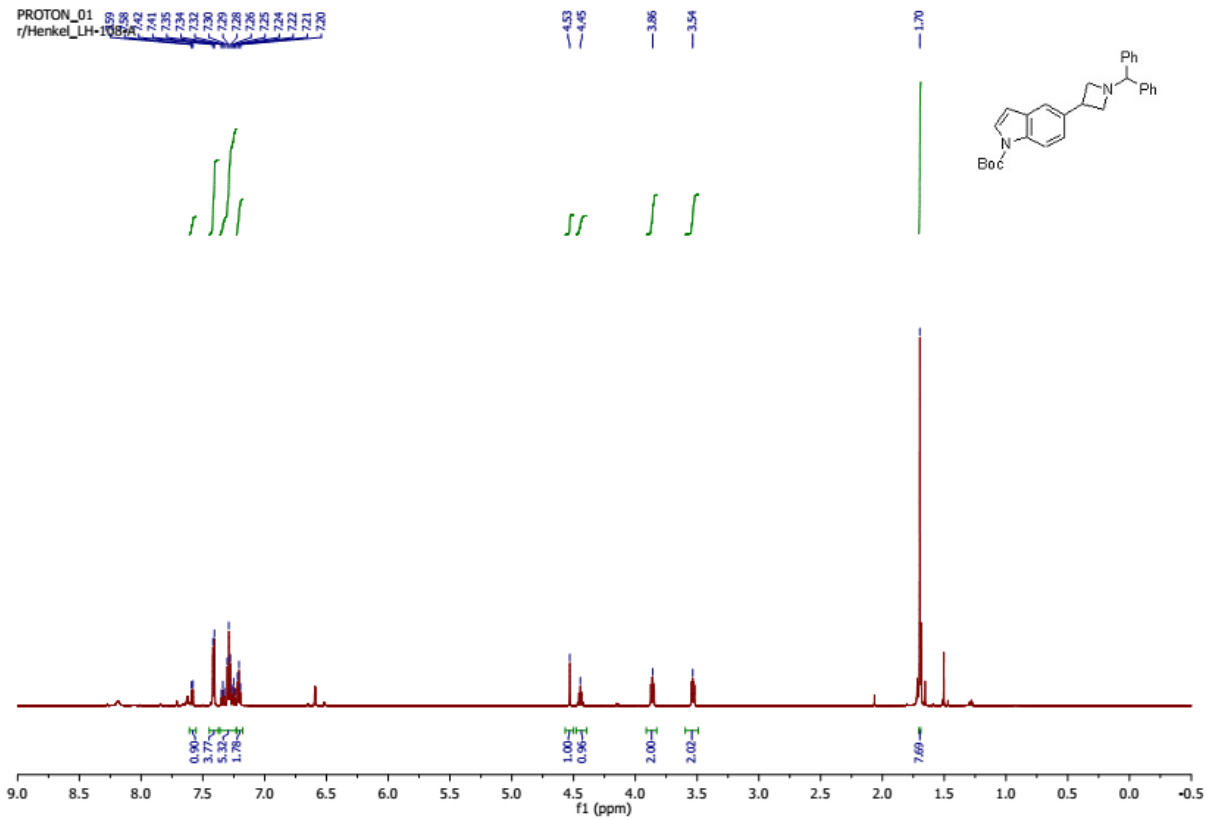

CARBON_01
r/Dixit_DP431-1
142.46
132.98
128.50
127.67
127.11
127.02
78.60
60.92
29.26
25.67
18.16
Ph
Me
N
Ph
Me
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

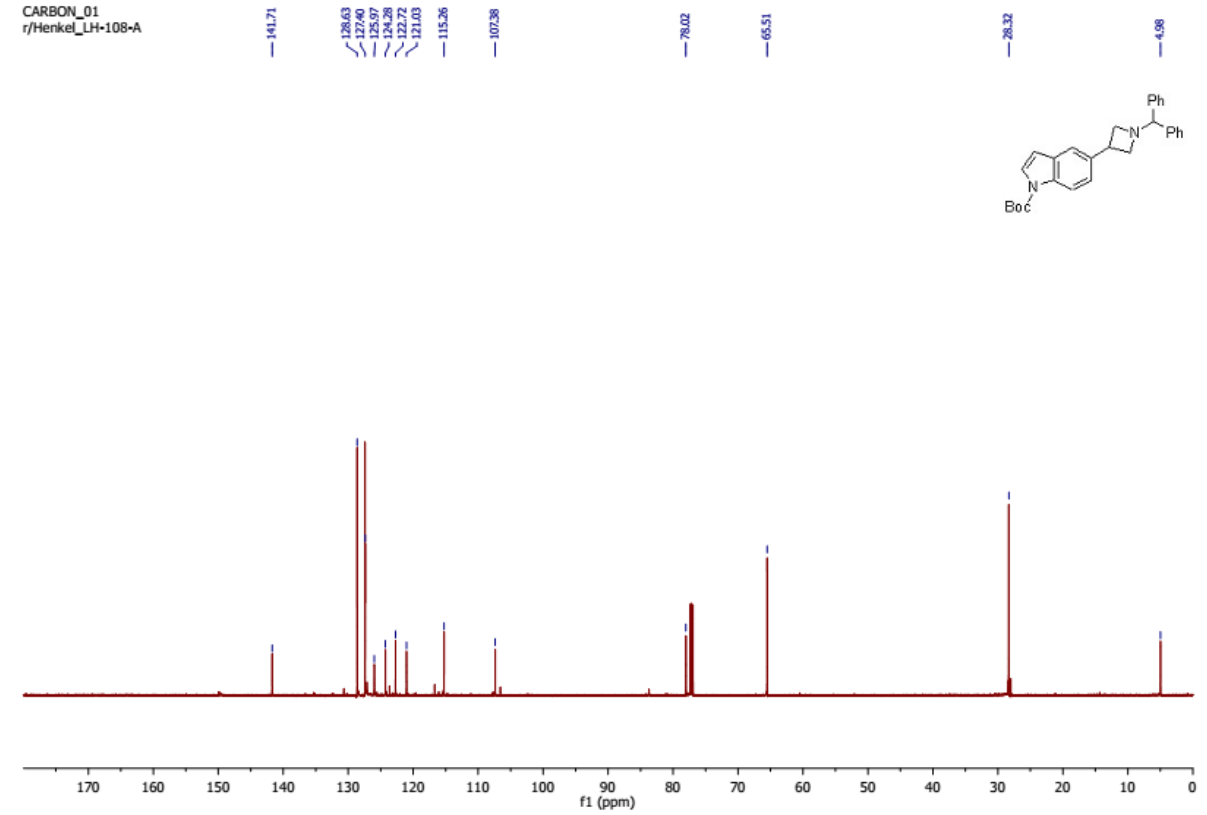

PROTON_01
r/Dixit_SP435-1
7.43
7.42
7.27
7.26
7.25
7.24
7.18
7.17
7.16
6.89
6.73
6.71
6.69
6.68
5.92
4.38
3.58
3.58
3.07
Ph
Ph
N
O
O
3.76
4.18
1.88
0.89
1.95
1.93
1.00
2.92
2.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

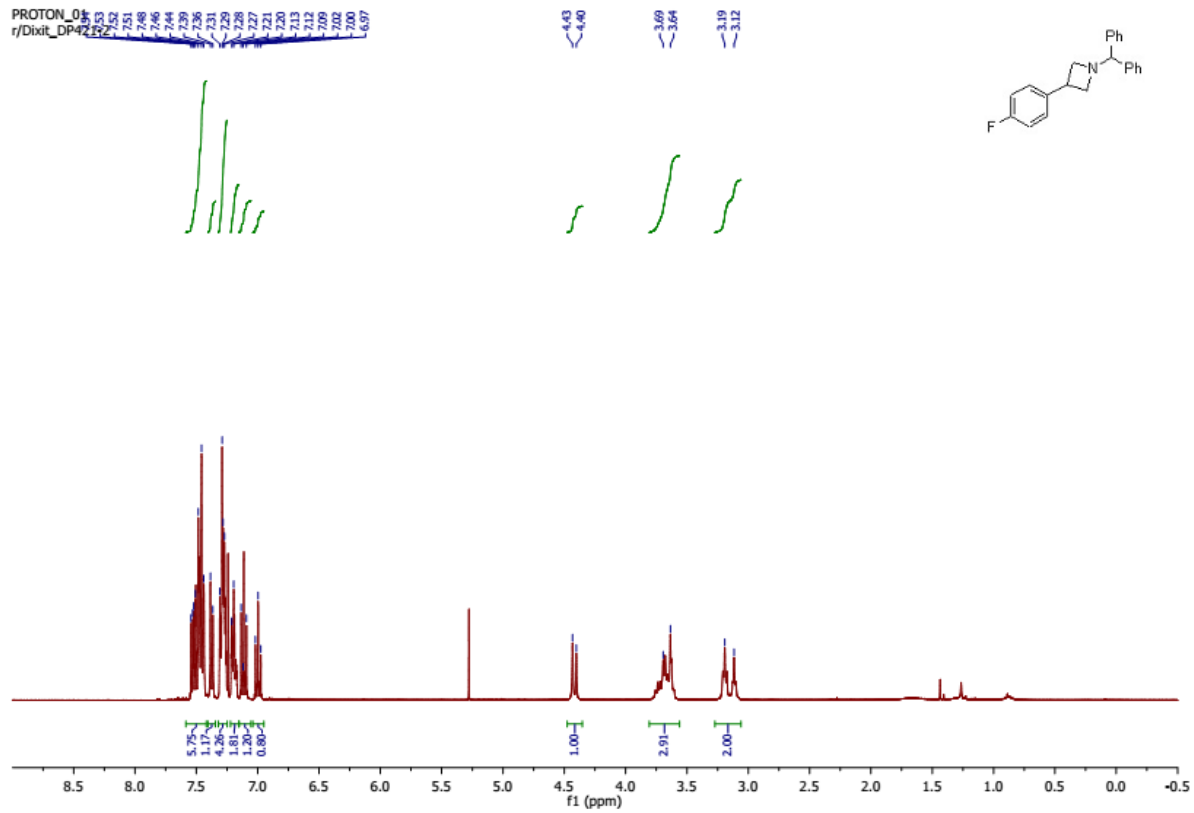

CARBON_01
r/Dixit_SP435-1
147.85
146.10
142.25
137.28
128.59
127.65
127.24
120.13
108.16
107.54
101.02
78.40
61.05
34.87
Ph
Ph
N
O
O
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

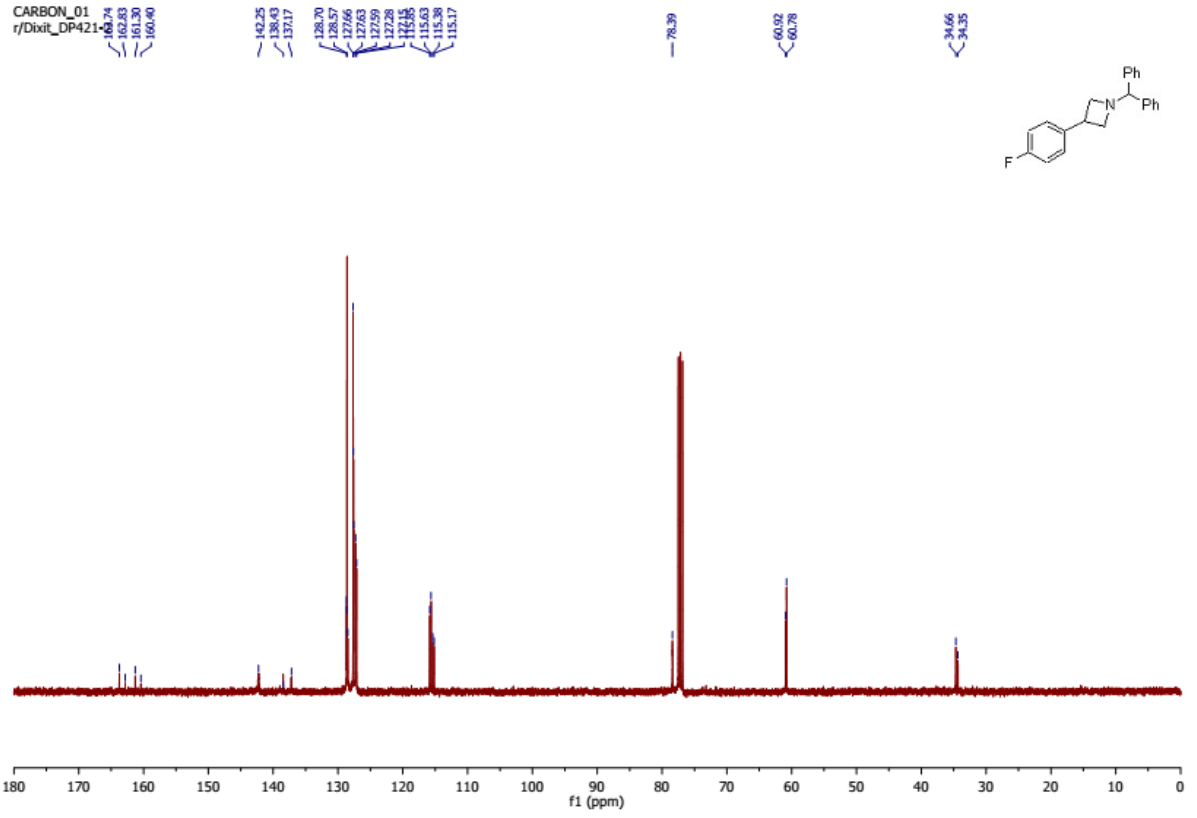

PROTON_01
r/henkel_lh97
7.75
7.74
7.45
7.44
7.29
7.28
7.27
7.25
7.20
7.19
7.18
4.41
3.62
3.17
Ph
Ph
N
N
0.89
0.82
0.84
3.42
4.71
1.71
1.00
2.74
1.98
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


CARBON_01
r/henkel_lh97
149.03
148.00
142.08
136.74
134.60
128.65
127.59
127.33
123.57
78.24
60.43
32.69
Ph
Ph
N
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


PROTON_01
r/Henkel_LH-108-A
7.59
7.58
7.42
7.41
7.35
7.34
7.32
7.30
7.29
7.28
7.26
7.25
7.24
7.22
7.21
7.20
4.53
4.45
3.86
3.54
1.70
Ph
Ph
N
N
Boc
0.90
3.77
5.32
1.78
1.00
0.96
2.00
2.02
7.69
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


CARBON_01
r/Henkel_LH-108-A
141.71
128.63
127.40
125.97
124.28
122.72
121.03
115.26
107.38
78.02
65.51
28.32
4.98
Ph
Ph
N
N
Boc
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


PROTON_01
r/Dixit_DP421-2
4.63
4.60
3.69
3.64
3.19
3.12
Ph
Ph
N
F
5.75
1.17
4.26
1.81
1.20
0.80
1.00
2.91
2.00
f1 (ppm)


CARBON_01
r/Dixit_DP421-2
142.25
138.43
137.17
128.70
128.57
127.66
127.63
127.59
127.28
115.63
115.38
115.17
78.39
60.92
60.78
34.66
34.35
Ph
Ph
N
F
f1 (ppm)