

Electronic Supplementary Material (ESI) for RSC Advances This journal is © The Royal Society of Chemistry 2016

Supporting Information

***n*-Bu₄Ni/TBHP-catalyzed C–N bond formation via cross- dehydrogenative coupling of 1*H*-1,2,4-triazoles (N–H) and methylarenes (C_{sp³}–H).**

Habtamu Abebe, Siddaiah Vidavalur, Venkateswara Rao Battula

Department of Organic Chemistry and FDW, College of Science and Technology, Andhra University,
Visakhapatnam, Andhra Pradesh, India

sidduchem@gmail.com

1. General Considerations.....	S2
2. General procedures for the synthesis of 1-benzyl-1<i>H</i>-1,2,4-triazoles	S2
3. Characterization data for 1-benzyl-1<i>H</i>-1,2,4-triazoles	S3
4. General procedures for the synthesis of 1-alkyl-1<i>H</i>-1,2,4-triazoles.....	S10
5. Characterization data for 1-alkyl-1<i>H</i>-1,2,4-triazoles	S10
6. ¹H and ¹³C NMR spectra.....	S12
7. References	S31

1. General considerations

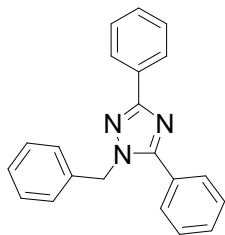
All Chemicals were purchased from Sigma Aldrich and AVRA Synthesis Private Limited Company and used as received without special purification unless stated otherwise. All 1*H*-1,2,4-triazoles were prepared according to the literature [S. Ueda, H. Nagasawa, *J. Am. Chem. Soc.*, 2009, **131**, 15080] procedure. Reactions were monitored by thin layer chromatography (TLC) carried out on aluminum plates coated with silica gel (Silica gel 60 F254) using ethyl acetate and n-hexane as mobile phase. Chromatogram was visualized using UV light (254 nm). The products were purified using column chromatography on silica gel (100-200 mesh). Melting points were determined in open capillary tube and are not corrected. ¹H and ¹³C NMR spectra were recorded on 400 MHz spectrometer using TMS as internal standard. The chemical shifts are reported in (δ) ppm relative to tetramethylsilane (TMS) as internal reference. Data are reported as follows: chemical shift and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). The coupling constant J is given in Hz.

2. General procedure for the synthesis of 1-benzyl-1*H*-1,2,4-triazoles

An oven-dried 50 mL round bottom flask equipped with a magnetic stir bar was charged with methylarene (**1**) (6 mmol), 1*H*-1,2,4-triazole (**2**), (0.3 mmol), *n*-Bu₄NI (20 mol %) and aqueous solution of TBHP (70% In H₂O, 0.9 mmol, 3 equiv.) at room temperature. The reaction mixture was then refluxed at 85 °C for 10 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was admixed with ethyl acetate and transferred in to a separating funnel. The ethyl acetate layer was sequentially washed with 5% solution of sodium bicarbonate and brine solution. The combined organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated in vacuum. The resulting residue was purified over a column of silica gel using ethyl acetate in hexane as eluents to afford the title product **3 or 4**.

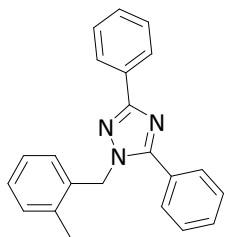
3. Characterization data for 1-benzyl-1*H*-1,2,4-triazoles

1-Benzyl-3, 5-diphenyl-1*H*-1, 2, 4-triazole (**3a**, Table 2)¹



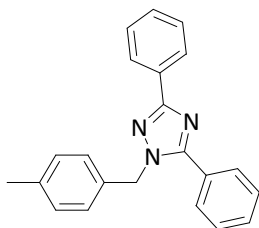
White solid, 90% (37.24 mg), mp 97-99 °C (lit. m.p. 98.5-99.5 °C). ^1H NMR (300 MHz, CDCl_3) : δ 8.21 (d, $J = 7.2$ Hz, 2H), 7.62-7.51 (m, 2H), 7.48-7.13 (m, 11H), 5.39 (s, 2H) ppm; ^{13}C NMR (CDCl_3 , 75 MHz) δ : 161.4, 156.0, 135.9, 130.9, 130.0, 129.0, 128.70, 128.67, 128.6, 128.4, 127.9, 127.8, 126.6, 126.3, 52.6 ppm; LCMS $m/z = 311.145$ $[\text{M}+\text{H}]^+$. Anal. Calcd. for $\text{C}_{21}\text{H}_{17}\text{N}_3$: C, 81.00; H, 5.50; N, 13.49, Found: C, 80.95; H, 5.57.

1-(2-Methylbenzyl)-3,5-diphenyl-1H-1,2,4-triazole (3b, Table 2).



White solid, 83% (31.45 mg), mp 98-100 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.22-8.20 (t, 2H), 7.63-7.65 (d, $J = 8.0$ Hz, 2H), 7.49-7.41 (m, 6H), 7.23-7.16 (t, 3H), 6.91-6.89 (d, 1H, $J = 7.6$ Hz), 5.45 (s, 2H), 2.30 (s, 3H) ppm; ^{13}C NMR (400 MHz, CDCl_3): δ 161.7, 156.3, 134.9, 134.4, 131.1, 130.4, 130.2, 129.2, 128.9, 128.7, 128.6, 128.1, 127.9, 126.6, 126.5, 126.4, 50.8, 19.1 ppm; LCMS: $m/z = 326$ $[\text{M}+\text{H}]^+$. Anal. Calcd. for $\text{C}_{22}\text{H}_{19}\text{N}_3$: C, 81.20; H, 5.89; N, 12.91, Found: 81.16; H, 5.95

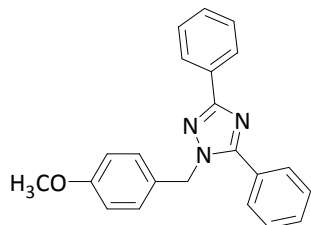
1-(4-Methylbenzyl)-3,5-diphenyl-1H-1,2,4-triazole (3c, Table 2)



White solid, 90% (34.48 mg), mp 98-100 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.21-8.19 (m, 2H), 7.65-7.63 (m, 2H), 7.49-7.40 (m, 6H), 7.17-7.11 (m, 4H), 5.43 (s, 2H), 2.34 (s, 3H) ppm; ^{13}C NMR (400 MHz, CDCl_3): δ 161.6, 156.1, 137.7, 133.1, 131.1, 130.2, 129.6, 129.2, 128.9, 128.8, 128.5, 126.9,

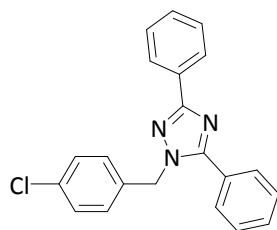
126.5, 52.6, 21.1 ppm; LCMS: $m/z = 326$ $[M+H]^+$. Anal. Calcd. For $C_{22}H_{19}N_3$: C, 81.20; H, 5.89; N, 12.91; Found: C, 81.15; H, 5.94.

1-(4-Methoxybenzyl)-3,5-diphenyl-1H-1,2,4-triazole (3d, Table 2).



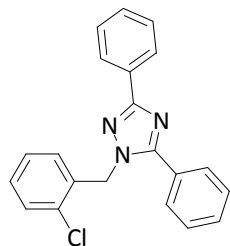
White solid, 70% (31.31 mg), mp 124-126 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.20-8.19 (d, $J = 6.8$ Hz, 2H), 7.64-7.62 (d, $J = 7.6$ Hz, 2H), 7.49-7.34 (m, 6H), 7.17-7.15 (d, $J = 8.8$ Hz, 2H), 6.88-6.85 (d, $J = 8.4$ Hz, 2H), 5.38 (s, 2H), 3.79 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.5, 159.3, 156.0, 131.1, 130.2, 129.2, 128.9, 128.5, 128.4, 128.2, 128.1, 126.5, 114.3, 55.3, 52.3 ppm; LCMS: $m/z = 342$ $[M+H]^+$. Anal. Calcd. For $C_{22}H_{19}N_3O$: C, 77.35; H, 5.61; N, 12.31; O, 4.36; Found: C, 77.30; H, 5.65. N, 12.27.

1-(4-Chlorobenzyl)-3,5-diphenyl-1H-1,2,4-triazole (3e, Table 2).



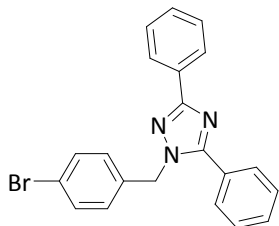
White solid, 79% (32.82 mg), mp 123-125 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.20-8.18 (d, $J = 8.0$ Hz, 2H), 7.62-7.60 (d, $J = 7.6$ Hz, 2H), 7.50-7.44 (m, 6H), 7.33-7.26 (dd, 2H), 7.17-7.15 (d, $J = 6.8$ Hz, 2H), 5.43 (s, 2H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.5, 154.1, 130.4, 129.3, 129.1, 128.9, 128.8, 128.6, 128.3, 126.5, 52.1 ppm; LCMS: $m/z = 346$ $[M+H]^+$. Anal. Calcd. For $C_{21}H_{16}ClN_3$: C, 72.93; H, 4.66; Cl, 10.25; N, 12.15; Found: C, 72.89; H, 4.69. N, 12.10.

1-(2-Chlorobenzyl)-3,5-diphenyl-1H-1,2,4-triazole (3f, Table 2).



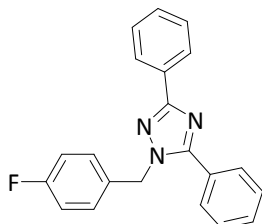
White solid, 73% (30.33 mg), mp 123-125 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.24-8.22 (t, 2H), 7.64-7.51 (dd, *J* = 8.0 Hz, 2H), 7.50-7.42 (m, 4H), 7.32-7.25 (m, 2H), 7.05-7.03 (d, *J* = 8.0 Hz, 1H), 5.60 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 162.0, 156.6, 134.0, 132.1, 130.9, 129.6, 129.4, 129.2, 129.0, 128.6, 127.9, 127.7, 127.5, 126.5, 50.61 ppm; LCMS: *m/z* = 346 [M+H]⁺. Anal. Calcd. For C₂₁H₁₆ClN₃: C, 72.93; H, 4.66; Cl, 10.25; N, 12.15; Found: C, 72.89; H, 4.71; N, 12.18.

1-(4-Bromobenzyl)-3, 5-diphenyl-1*H*-1, 2, 4-triazole (3g, Table 2).



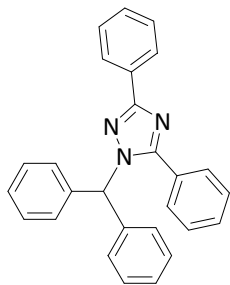
White solid, 81% (35.64 mg), mp 125-127 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.20-8.18 (d, *J* = 7.6 Hz, 2H), 7.61-7.60 (d, *J* = 7.2 Hz, 2H), 7.50-7.41 (m, 8H), 7.11-7.10 (d, *J* = 7.6 Hz, 2H), 5.41 (s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 161.8, 156.2, 135.1, 132.1, 130.9, 130.4, 129.3, 129.0, 128.8, 128.7, 128.6, 127.9, 126.5, 122.1, 52.2 ppm; LCMS: *m/z* = 390 [M+H]⁺. Anal. Calcd. for C₂₁H₁₆BrN₃: C, 64.63; H, 4.13; Br, 20.47; N, 10.77; Found: C, 64.59; H, 4.18; N, 10.72.

1-(4-Fuolorbenzyl)-3, 5-diphenyl-1*H*-1, 2, 4-triazole (3h, Table 2)¹.



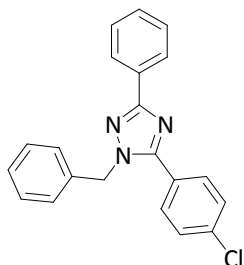
White solid, 75% (34.23 mg), mp 101-103 °C (lit. oil). ¹H NMR (300 MHz, CDCl₃): δ 8.24-8.16 (m, 2H), 7.64-7.52 (m, 2H), 7.50-7.33 (m, 6H), 7.20-7.10 (m, 2H), 7.05-6.92 (m, 2H), 5.37 (s, 2H) ppm; ¹³C NMR (CDCl₃, 75 MHz): δ 162.2 (d, *J* = 245.9 Hz), 161.5, 155.9, 131.6, 130.8, 130.2, 129.2, 128.8, 128.63, 128.61, 128.45, 127.8, 126.3, 115.7, 51.9 ppm; LCMS (*m/z*) 330 [M+H]⁺, Anal. calcd for C₂₁H₁₆N₃F : C, 76.58; H, 4.90; F, 5.77; N, 12.76; Found: C, 76.53; H, 4.96; N, 12.71.

1-Benzhydryl-3,5-diphenyl-1*H*-1,2,4-triazole (3i, Table 2).



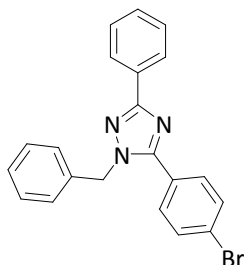
White solid, 75% (29.57 mg), mp 133-135 °C. ^1H NMR (300 MHz, CDCl_3): δ 8.18-8.16 (d, J = 6.8 Hz, 2H), 7.62-7.53 (d, 5H), 7.40-7.32 (m, 14H), 6.72 (s, 1H) ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 161.5, 143.8, 139.3, 131.2, 130.3, 129.2, 129.1, 129.0, 128.6, 128.5, 128.5, 128.4, 128.3, 128.1, 127.6, 126.6, 126.6, 65.8 ppm; LCMS: m/z = 388 $[\text{M}+\text{H}]^+$. Anal. Calcd. for $\text{C}_{27}\text{H}_{21}\text{N}_3$: C, 83.69; H, 5.46; N, 10.48; Found: C, 83.64; H, 5.52.

1-Benzyl-5-(4-chlorophenyl)-3-phenyl-1H-1,2,4-triazole (4a, Table 3)¹.



White solid, 85% (33.15mg), mp 143-145 °C. ^1H NMR (300 MHz, CDCl_3): δ 8.13 (d, J = 8.4 Hz, 2H), 7.65-7.59 (m, 2H), 7.53-7.39 (m, 5H), 7.39-7.28 (m, 3 H), 7.24-7.18 (m, H), 5.45 (s, 2H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz): δ 160.5, 156.1, 135.8, 134.9, 130.2, 129.5, 128.80, 128.78, 128.6, 127.9, 127.70, 127.66, 126.7, 52.6 ppm; MS (EI) (m/z) 347 Anal. Calcd for $\text{C}_{21}\text{H}_{16}\text{ClN}_3$: C, 72.93, H, 4.66, N, 12.15; found: C, 72.88, H, 4.50, N, 12.10.

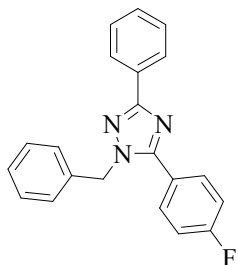
1-Benzyl-5-(4-bromophenyl)-3-phenyl-1H-1,2,4-triazole (4b, Table 3).



Pale yellow solid, 78% (31.15 mg), mp 148-150 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.2-8.18 (t, 1H), 8.08-8.06 (d, J = 8.8 Hz, 1H), 7.61-7.57 (m, 3H), 7.51-7.47 (m, 4H), 7.36-7.34 (m, 2H), 5.46 (s, 2H)

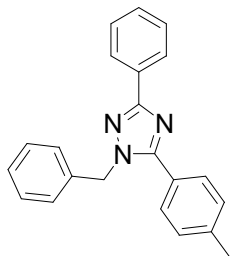
ppm; ^{13}C NMR (100 MHz, CDCl_3) δ 160.8, 156.3, 132.2, 132.2, 131.8, 131.7, 130.4, 130.3, 129.1, 129.0, 128.9, 128.8, 128.6, 128.1, 126.9, 126.7, 126.5, 52.8 ppm; LCMS: $m/z = 391$ [$\text{M}^{+}({}^{81}\text{Br})$]. Anal. Calcd. for $\text{C}_{27}\text{H}_{21}\text{N}_3$: C, 64.63; H, 4.13; Br, 20.47; N, 10.77, Found: C, 64.59; H, 4.19, N, 10.71.

1-Benzyl-5-(4-fluorophenyl)-3-phenyl-1H-1, 2, 4-triazole (4c, Table 3).



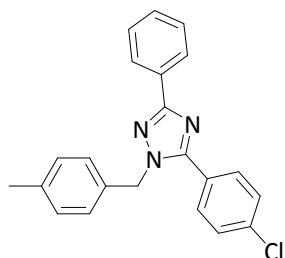
White solid, 72% (28.85 mg), mp 115-117 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.18-8.17 (d, $J = 6.0$ Hz, 2H), 7.6 (d, 2H), 7.49-7.45 (m, 3H), 7.36-7.50 (m, 3H), 7.03-6.97 (m, 2H), 7.14-7.12 (m, 2H), 5.46 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 162.3, 156.3, 147.42, 130.3, 129.0, 128.9, 128.9, 128.8, 128.6, 126.9, 126.8, 126.5, 116.2, 115.6, 115.4, 52.8 ppm; LCMS: $m/z = 330$ [$\text{M}+\text{H}$] $^+$. Anal. Calcd. for $\text{C}_{21}\text{H}_{16}\text{FN}_3$: C, 76.58; H, 4.90; F, 5.77; N, 12.76; Found: C, 76.53; H, 4.95; N, 12.70.

1-Benzyl-3-phenyl-5-tolyl-1H-1, 2, 4-triazole (4d, Table 3)



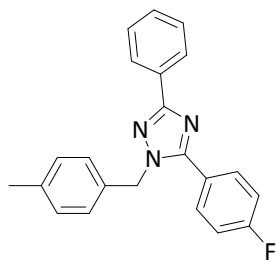
White solid, 85% (28.54 mg), mp 105-107 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.09-8.07 (d, $J = 8.0$ Hz, 1H), 7.63-7.60 (dd, 1H), 7.52-7.50 (d, 1H), 7.47-7.44 (ddd, 1H), 7.36-7.50 (m, 3H), 7.34-7.31 (m, 4H), 7.27-7.20 (m, 4H), 5.45 (s, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 139.1, 136.2, 132.5, 130.2, 129.6, 129.3, 129.2, 129.0, 128.9, 128.7, 128.6, 128.5, 128.3, 128.1, 128.0, 126.7, 126.5, 126.4, 52.7, 21.4 ppm; LCMS: $m/z = 326$ [$\text{M}+\text{H}$] $^+$. Anal. Calcd. For $\text{C}_{22}\text{H}_{19}\text{N}_3$: C, 81.20; H, 5.89; N, 12.91; Found: 81.15; H, 5.94.

1-(4-Methylbenzyl)-5-(4-chlorophenyl)-3-phenyl-1H-1,2,4-triazole(4e, Table 3)



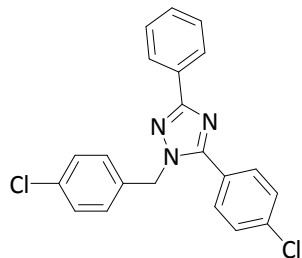
White solid, 71% (**25.68 mg**), mp 117-119 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.18 (s, 2H), 7.62-7.12 (t, 11H), 5.43 (s, 2H), 2.32 (s, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 161.6, 154.2, 130.2, 129.6, 129.2, 128.9, 128.8, 128.5, 126.8, 126.5, 52.6, 21.1 ppm; LCMS (m/z) 363[M+H]⁺. Anal. calcd for C₂₂H₁₈ClN₃: C, 73.43; H, 5.04, Cl, 9.85; N, 11.68; found: C, 73.39; H, 5.09; N, 11.63.

1-(4-Methylbenzyl)-5-(4-fluorophenyl)-3-phenyl-1H-1, 2, 4-triazole (4f, Table 3).



White solid, 79% (25.63 mg, 90%), mp 121-123 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.21-8.16 (dd, *J* = 4.8 Hz, 2H), 7.64-7.60 (dd, 2H), 7.50-7.41 (m, 4H), 7.16-7.10 (m, 5H), 5.43-5.41 (s, 2H), 2.35 (s, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 164.2, 151.7, 131.0, 130.2, 129.6, 129.2, 128.9, 128.5, 126.9, 126.7, 126.5, 116.1, 115.9, 115.6 ppm; LCMS: m/z = 344 [M+H]⁺. Anal. Calcd. For C₂₂H₁₈FN₃: C, 76.95; H, 5.28; F, 5.53; N, 12.24; Found: C, 76.90; H, 5.59; N, 12.19.

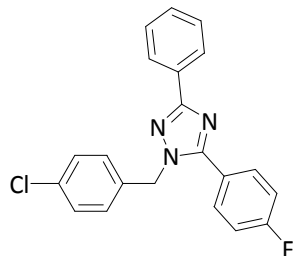
1-(4-Chlorobenzyl)-5-(4-chlorophenyl)-3-phenyl-1H-1,2,4-triazole (4g, Table 3).



White solid, 65% (25.53 mg), mp 120-122 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.13-8.11 (d, *J* = 7.2 Hz, 2H), 7.59 (s, 2H), 7.51 (d, 3H), 7.44-7.42 (d, *J* = 7.2, 2H), 7.34-7.32 (d, *J* = 7.2, 2H) 7.16-7.15(d, *J* = 7.2, 2H), 5.41 (s, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 164.7, 156.6, 156.4, 149.41, 130.5, 129.2,

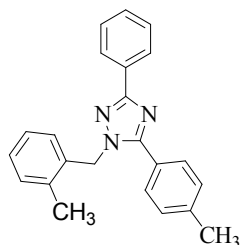
129.0, 128.8, 128.4, 127.8, 52.2 ppm; LCMS: $m/z = 380 [M+H]^+$. Anal. Calcd. For $C_{21}H_{15}Cl_2N_3$ C, 66.33; H, 3.98; Cl, 18.65; N, 11.05; Found: C, 66.29; H, 4.03; N, 10.99.

1-(4-Chlorobenzyl)-5-(4-fluorophenyl)-3-phenyl-1H-1,2,4-triazole (4h, Table 3)



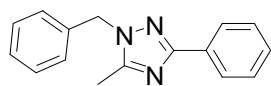
White solid, 77% (25.63 mg), mp 131-133 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.18-8.17 (d, $J = 5.6$ Hz, 2H), 7.59 (d, 2H), 7.50-7.44 (t, 3H), 7.34-7.32 (d, 2H), 7.20-7.14 (t, 3H), 5.41 (s, 2) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.1, 164.8, 161.8, 134.4, 134.4, 130.9, 130.8, 130.4, 130.5, 129.4, 129.3, 129.2, 129.1, 129.1, 129.0, 129.0, 128.8, 128.8, 128.6, 128.6, 128.4, 128.4, 128.2, 126.5, 126.5, 116.3, 116.1, 115.7, 52.2 ppm; LCMS: $m/z = 364 [M+H]^+$. Anal. Calcd. for $C_{21}H_{15}ClFN_3$: C, 69.33; H, 4.16; Cl, 9.74, F, 5.22; N, 11.55; Found: C, 69.28; H, 4.21; N, 11.50.

1-(2-Methylbenzyl)-5-(4-methylphenyl)-3-phenyl-1H-1,2,4-triazole (4i, Table 3)



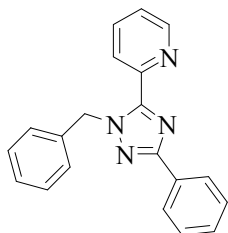
White solid, 71% (22.27 mg), mp 110-112 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.25-8.12 (dd, $J = 7.2$ Hz, 1H), 7.64-7.63 (t, 2H), 7.55-7.50 (m, 5H), 7.49-7.45 (m, 2), 7.30-7.18, (m, 1), 6.91-6.93 (d, $J = 7.6$ Hz, 1H), 5.45 (s, 2H), 2.43 (s, 3H), 2.431 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.8, 156.3, 140.4, 139.2, 134.8, 134.5, 130.4, 130.2, 129.6, 129.3, 128.9, 128.7, 128.6, 128.5, 127.8, 126.6, 126.5, 126.4, 50.77, 21.46, 19.12 ppm; LCMS: $m/z = 340 [M+H]^+$. Anal. Calcd. For $C_{23}H_{21}N_3$: C, 81.38; H, 6.24; N, 12.38; Found: C, 81.32; H, 6.29.

1-Benzyl-5-methyl-3-phenyl-1H-1,2,4-triazole (4j, Table 3)²



White solid, 60% (20.54 mg), mp 70-72 °C . ¹H NMR (400 MHz, CDCl₃): δ 2.45 (s, 3H), 5.40 (s, 2H), 7.24-7.27 (m, 3H), 7.31-7.44 (m, 5H), 7.98-8.00 (m, 2H) ppm. ¹³C NMR (100 MHz, CD₃OD): δ 11.8, 53.1, 127.2, 128.3, 129.2, 129.7, 130.0, 130.4, 131.9, 137.0, 155.0, 161.6 ppm; MS [m/z]: 249(M⁺) . Anal. Calcd. For C₁₆H₁₅N₃: C, 77.08; H, 6.06; N, 16.85; Found: C, 77.01; H, 6.11.

2-(2-Benzyl-5-phenyl-1H-1,2,4-triazol-3-yl)pyridine (4k, Table 3).



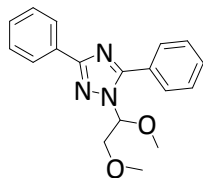
White solid, 67% (24.65 mg), mp 100-102 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.69-8.68 (d, 1H, *J* = 4.0 Hz), 8.35-8.33 (d, 1H, *J* = 8.0), 8.20-8.18 (d, 2H, *J* = 8.0 Hz), 7.86-7.82 (d, 1H), 7.48-7.40 (d, 3H), 7.37-7.24 (m, 9H), 6.12 (s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 161.1, 154.94, 148.8, 137.0, 129.2, 128.5, 128.5, 127.9, 127.6, 126.5, 124.2, 124.0, 54.2 ppm; LCMS: m/z = 313 [M+H]⁺. Anal. Calcd. For C₂₀H₁₆N₄: C, 76.90; H, 5.16; N, 17.94; Found: C, 76.84; H, 5.21.

4. General procedures for the synthesis of 1-alkyl-1H-1,2,4-triazoles

An oven-dried 50 mL round bottom flask equipped with a magnetic stir bar was charged with methylarene (**1**) (6 mmol), 1H-1,2,4-triazole (**2**), (0.3 mmol), *n*-Bu₄NI (20 mol %) and aqueous solution of TBHP (70% In H₂O, 0.9 mmol, 3 equiv.) at room temperature. The reaction mixture was then refluxed at 85 °C for 10 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was admixed with ethyl acetate and transferred in to a separating funnel. The ethyl acetate layer was sequentially washed with 5% solution of sodium bicarbonate and brine solution. The combined organic layer was dried over anhydrous Na₂SO₄, filtered and concentrated in vacuum. The resulting residue was purified over a column of silica gel using ethyl acetate in hexane as eluents to afford the title product **5**.

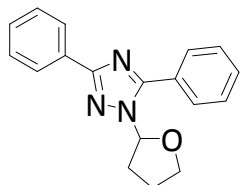
5. Characterization data for 1-alkyl-1H-1,2,4-triazoles

1-(1,2,-Dimethoxyl)-3,5-diphenyl-1H-1,2,4-triazole (6a, Scheme 2)



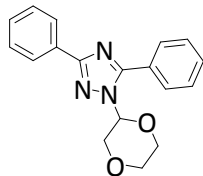
Light yellow oil, 60% (25.44mg). ^1H NMR (400 MHz, CDCl_3): δ 8.22-8.20 (d, J = 6.8 Hz, 2H), 8.00-7.98 (dd, J = 9.2 Hz, 2H), 7.55-7.54 (t, 3H), 7.50-7.43 (m, 4H), 5.62 (s, 1H), 4.00-3.98 (t, 2H), 3.63-3.61 (t, 3H), 3.42 (s, 3H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 161.3, 156.9, 130.8, 130.5, 129.4, 129.1, 129.0, 128.8, 128.6, 128.5, 127.5, 126.6, 126.5, 77.9, 71.43, 68.9, 59.1 ppm; LCMS: m/z = 310 $[\text{M}+\text{H}]^+$. Anal. Calcd. For $\text{C}_{18}\text{H}_{19}\text{N}_3\text{O}_2$: C, 69.88; H, 6.19; N, 13.58; O, 10.34; Found: C, 69.84; H, 6.23.

3,5-Diphenyl-1-(tetrahydrofuran-2-yl)-1H-1,2,4-triazole (6b, Scheme 2)



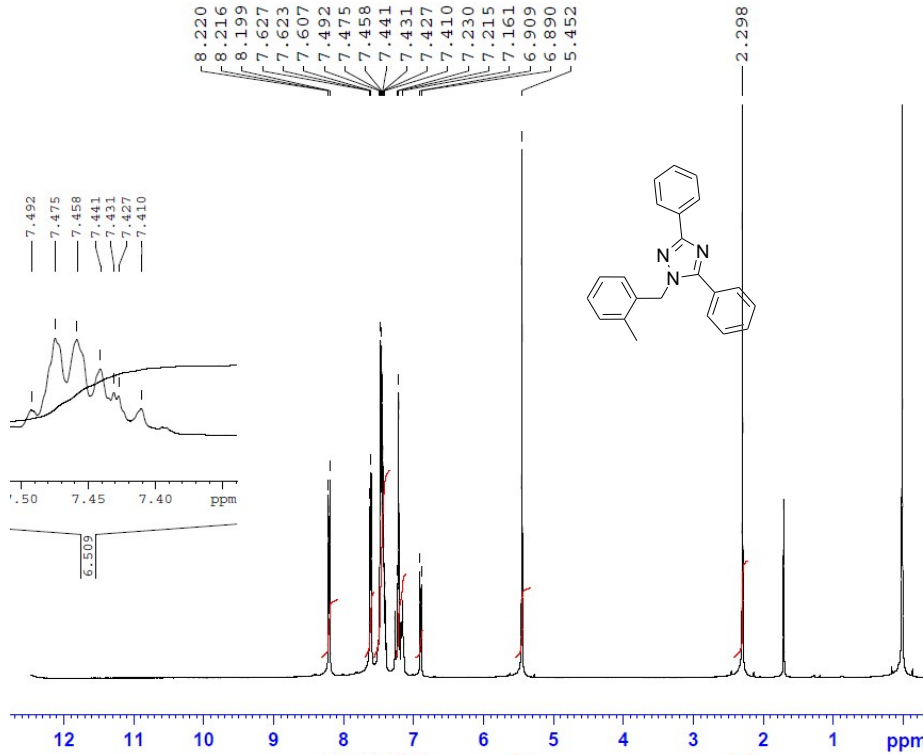
Light yellow oil, 65% (29.07 mg). ^1H NMR (400 MHz, CDCl_3): δ 8.21-8.19 (d, J = 7.2 Hz, 2H), 7.89-7.87 (dd, J = 9.2 Hz, 2H), 7.55-7.54 (t, 3H), 7.48-7.29 (m, 3H), 6.09-6.07 (dd, 1H), 4.34-4.04 (m, 2H), 2.81-2.64 (m, 2H), 2.35-2.14 (m, 2H) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 161.1, 156.4, 131.2, 130.2, 129.9, 129.4, 129.2, 128.8, 128.5, 128.1, 126.6, 126.5, 86.7, 69.5, 31.2, 25.2 ppm; LCMS: m/z = 292 $[\text{M}+\text{H}]^+$. Anal. Calcd. For $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}$: C, 74.20; H, 5.88; N, 14.42; O, 5.49; Found: C, 74.15; H, 5.93.

1-(1,4-Dioxan-2-yl)-3,5-diphenyl-1H-1,2,4-triazole (6c, Scheme 2)



White solid, 73% (28.60 mg), mp 107-109 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.19 (s, 2H), 7.82(d, 2H), 7.57-7.56 (s, 3H), 7.46-7.43 (d, 3H), 5.54-5.52 (t, 1H), 4.52-4.45 (d, 1H), 4.13-3.82 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) ppm; ^{13}C NMR (100 MHz, CDCl_3): δ 152.7, 148.7, 144.6, 130.7, 129.4, 129.0, 128.5, 127.5, 126.7, 80.6, 68.1, 66.6, 65.7 ppm; LCMS: m/z = 308 $[\text{M}+\text{H}]^+$. Anal. Calcd. For $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_2$: C, 70.34; H, 5.58; N, 13.67; O, 10.41; Found: C, 70.26; H, 5.62.

3b.



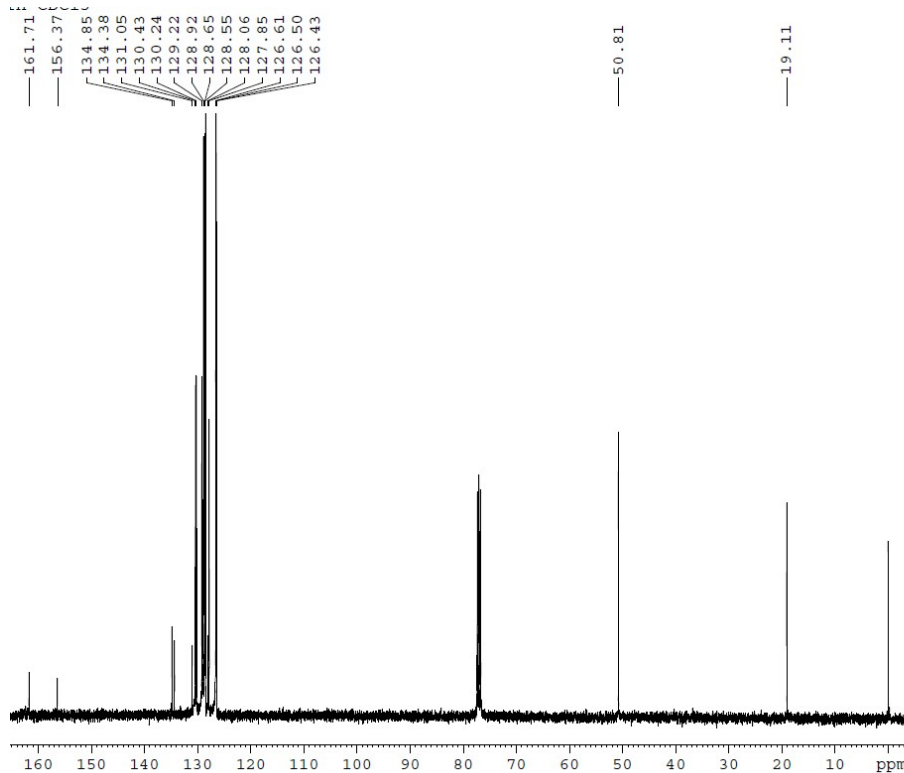
```

Current Data Parameters
NAME          4B
EXPNO         21
PROCNO        1

F2 - Acquisition Parameters
Date_         20151029
Time          15.54
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS           552
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG           162.09
DW           20.800 usec
DE           6.50 usec
TE           298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TDO          1

===== CHANNEL f1 =====
SFO1         400.1324008 MHz
NUC1          1H
P1           14.00 usec
PL1          11.00000000 W

F2 - Processing parameters
SI           65536
SF           400.1300069 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



```

Current Data Parameters
NAME          4B
EXPNO         21
PROCNO        1

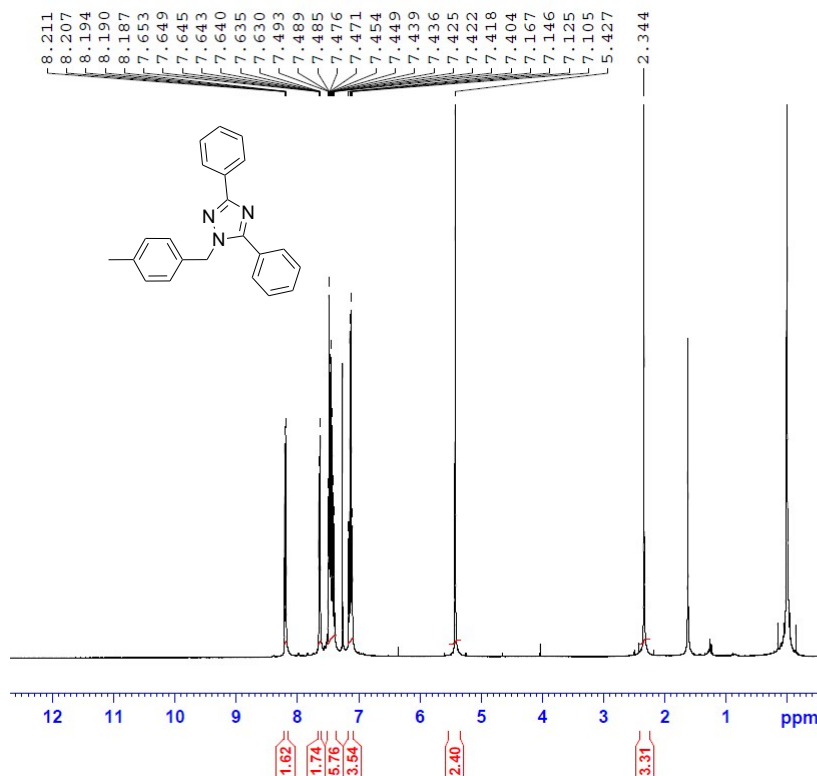
F2 - Acquisition Parameters
Date_         20151029
Time          15.54
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS           552
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631488 sec
RG           162.09
DW           20.800 usec
DE           6.50 usec
TE           298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TDO          1

===== CHANNEL f1 =====
SFO1         100.6228293 MHz
NUC1          13C
P1           9.45 usec
PL1          50.00000000 W

===== CHANNEL f2 =====
SFO2         400.1316005 MHz
NUC2          1H
CPDPRG2       waltz16
PCPD2         90.00 usec
PLW2         11.00000000 W
PLW12        0.27380000 W
PLW13        0.22180000 W

F2 - Processing parameters
SI           32768
SF           100.6127685 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```

3c.



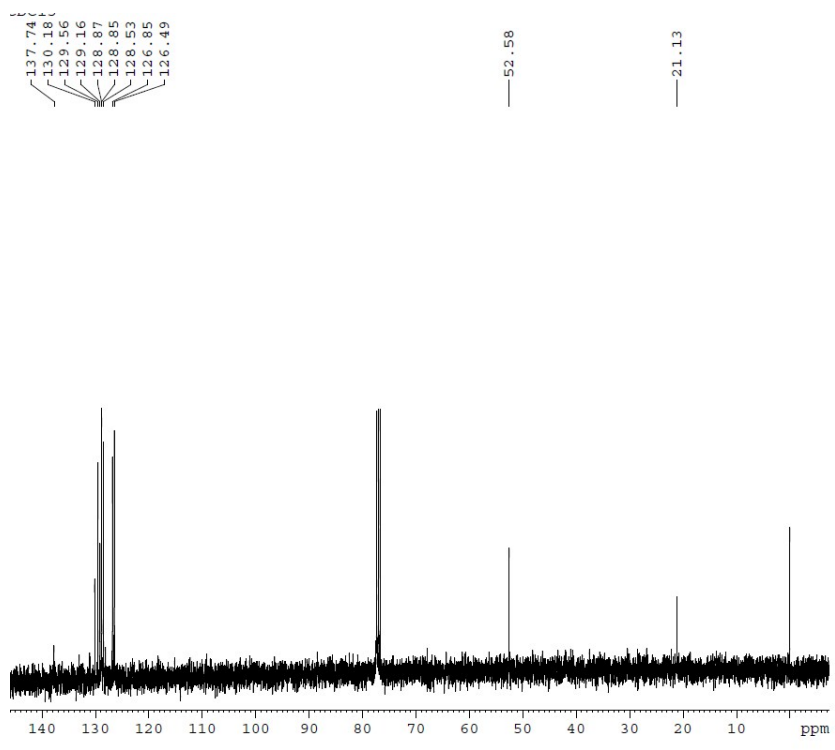
```

Current Data Parameters
NAME      4B
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20151026
Time     18.47
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        32768
SOLVENT  CDCl3
NS        32
DS        0
SWH       6009.615 Hz
FIDRES   0.182299 Hz
AQ        2.7262976 sec
RG         65.04
DW        83.200 usec
DE         6.50 usec
TE        298.2 K
D1        1.50000000 sec
TDO       1

===== CHANNEL f1 =====
SFO1     400.1328009 MHz
NUC1      1H
P1        14.00 usec
PLW1     11.00000000 W

F2 - Processing parameters
SI        65536
SF        400.1300069 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



```

Current Data Parameters
NAME      4B
EXPNO    30
PROCNO   1

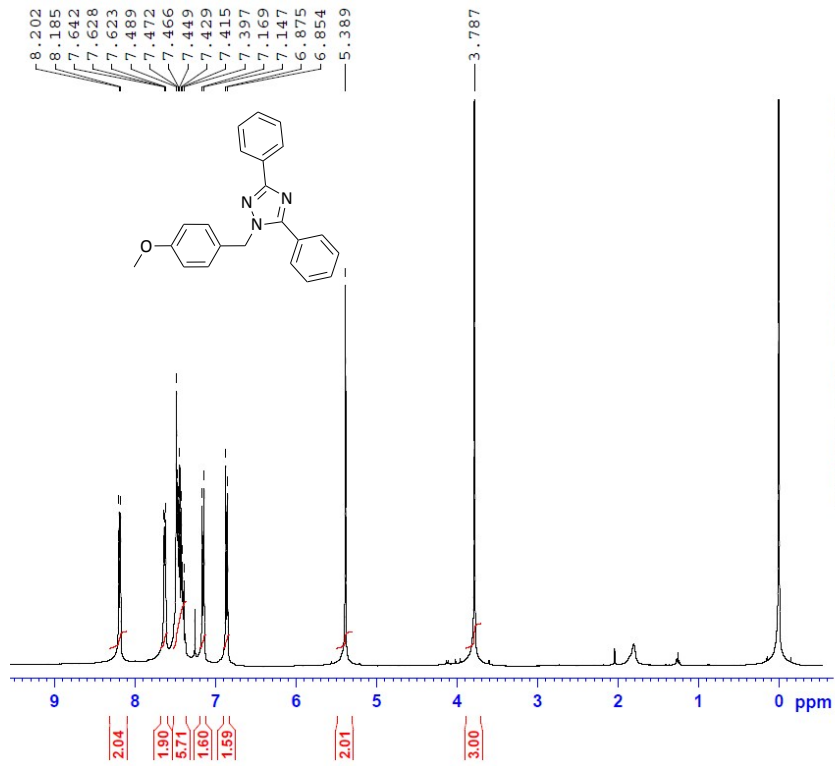
F2 - Acquisition Parameters
Date_    20151030
Time     18.24
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631488 sec
RG         204
DW        20.800 usec
DE         6.50 usec
TE        298.1 K
D1        2.50000000 sec
D11       0.03000000 sec
TDO       1

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1      13C
P1        9.45 usec
PLW1     50.00000000 W

===== CHANNEL f2 =====
SFO2     400.1316009 MHz
NUC2      1H
CPDPRG2  waltz16
PCPD2    90.00 usec
PLW2     11.00000000 W
PLW12    0.27383000 W
PLW13    0.22180000 W

F2 - Processing parameters
SI        32768
SF        100.6127685 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

3d.



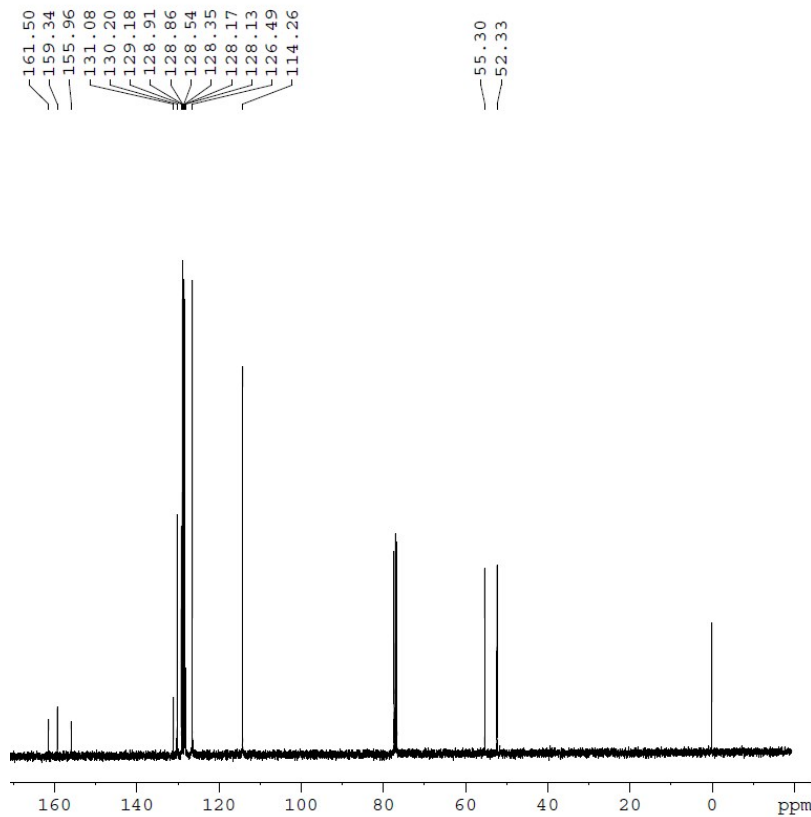
```

Current Data Parameters
NAME      9XX in DMSO
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20160508
Time     12.24
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg
TD        65536
SOLVENT  CDCl3
NS        127
DS        0
SWH       6009.615 Hz
FIDRES   0.091699 Hz
AQ        5.452592 sec
RG        50.82
DW        83.200 usec
DE        6.50 usec
TE        298.2 K
D1        1.5000000 sec
TD0       1

***** CHANNEL f1 *****
SF01    400.1328009 MHz
NUC1     1H
P1       14.20 usec
PLM1    11.0000000 W

F2 - Processing parameters
SI       65536
SF       400.1300105 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



```

Current Data Parameters
NAME      9XX in DMSO
EXPNO    15
PROCNO   1

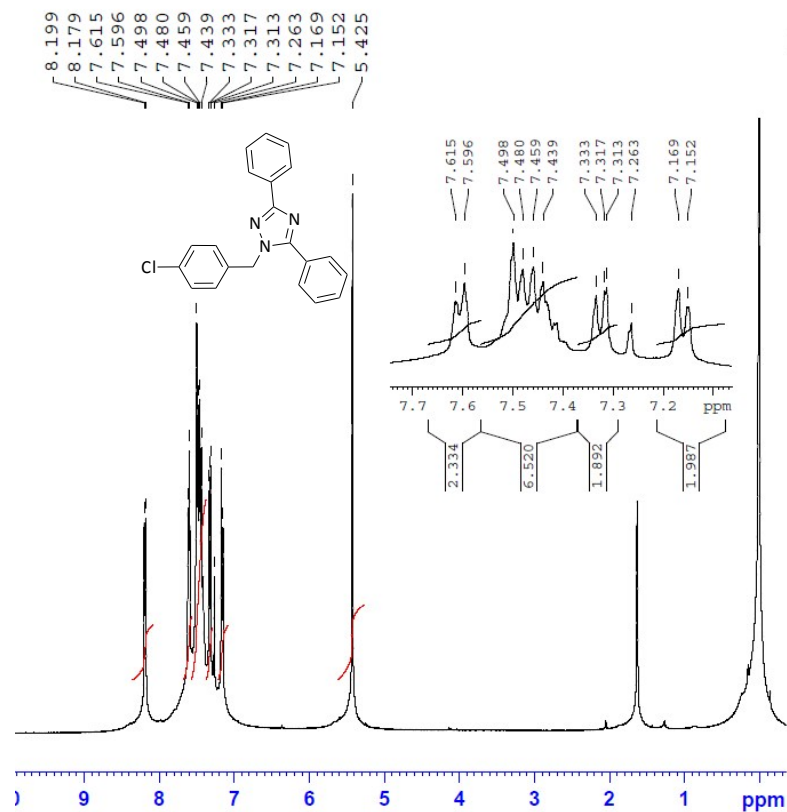
F2 - Acquisition Parameters
Date_    20160508
Time     12.46
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg3
TD        65536
SOLVENT  CDCl3
NS        176
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631488 sec
RG        204
DW        20.800 usec
DE        6.50 usec
TE        298.1 K
D1        2.5000000 sec
D11      0.0300000 sec
TD0       1

----- CHANNEL f1 -----
SF01    100.6228293 MHz
NUC1     13C
P1       9.45 usec
PLM1    50.0000000 W

----- CHANNEL f2 -----
SF02    400.1316005 MHz
NUC2     1H
CFDPRG[2] waltz16
PCPD2    90.00 usec
PLM2    11.0000000 W
PLM12   0.2718300 W
PLM13   0.2218000 W

F2 - Processing parameters
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

3e.



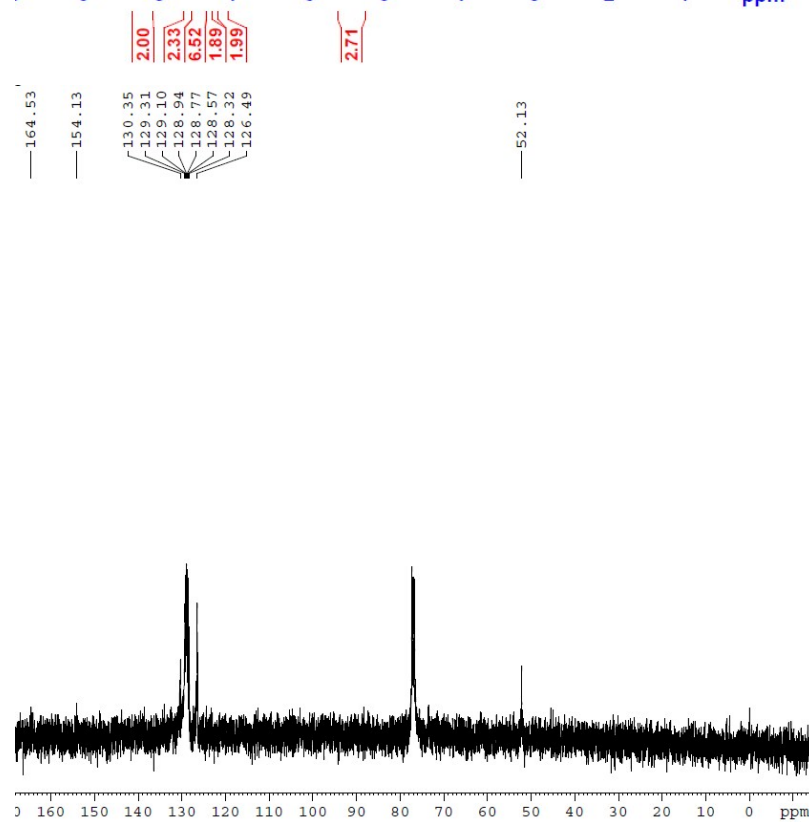
```

Current Data Parameters
NAME      4B
EXPNO    7
PROCNO   1

F2 - Acquisition Parameters
Date      20151026
Time      19.12
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        32768
SOLVENT  CDCl3
NS        32
DS        4
SWH       5197.505 Hz
FIDRES    0.158615 Hz
AQ        3.1529815 sec
RG         65.04
DW         96.200 usec
DE         6.50 usec
TE         298.1 K
D1         1.50000000 sec
TD0        1

----- CHANNEL f1 -----
SP01     400.1324008 MHz
NUC1      1H
P1        14.00 usec
PLW1     11.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300069 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

Current Data Parameters
NAME      4B
EXPNO    27
PROCNO   1

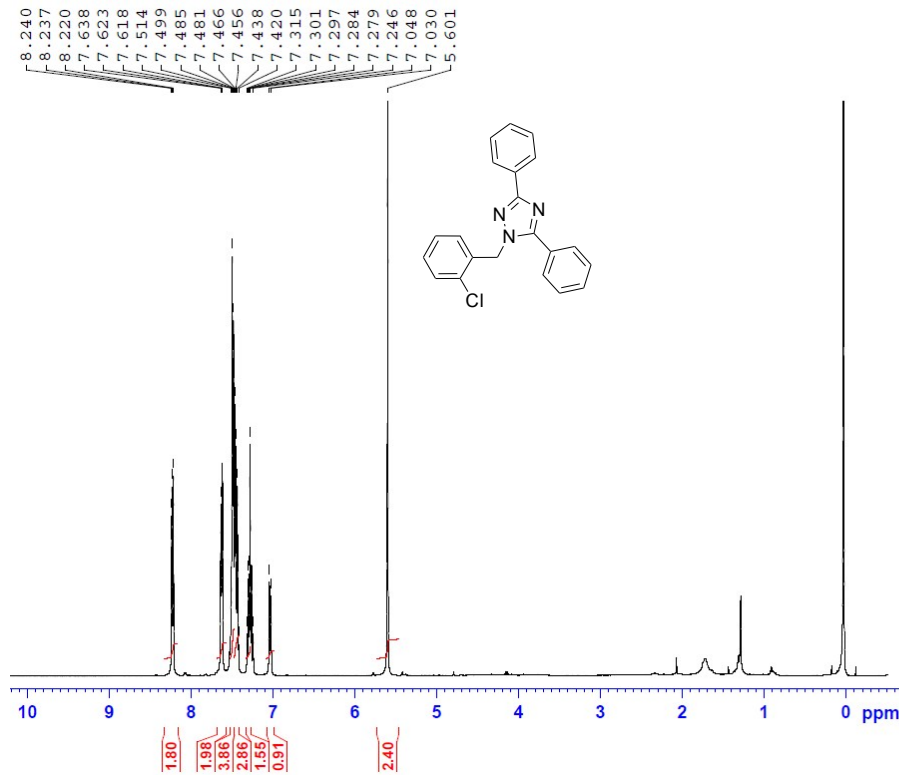
F2 - Acquisition Parameters
Date      20151030
Time      11.31
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        735
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631488 sec
RG         204
DW         20.800 usec
DE         6.50 usec
TE         298.2 K
D1         2.50000000 sec
D11        0.03000000 sec
TD0        1

----- CHANNEL f1 -----
SP01     100.6228293 MHz
NUC1      13C
P1         9.45 usec
PLW1     50.00000000 W

----- CHANNEL f2 -----
SP02     400.1316005 MHz
NUC2      1H
CPDPRG2  waltz16
PCPD2    30.00 usec
PLW2     11.00000000 W
PLW12    0.27383000 W
PLW13    0.22180000 W

F2 - Processing parameters
SI         32768
SF         100.6127685 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

3f.



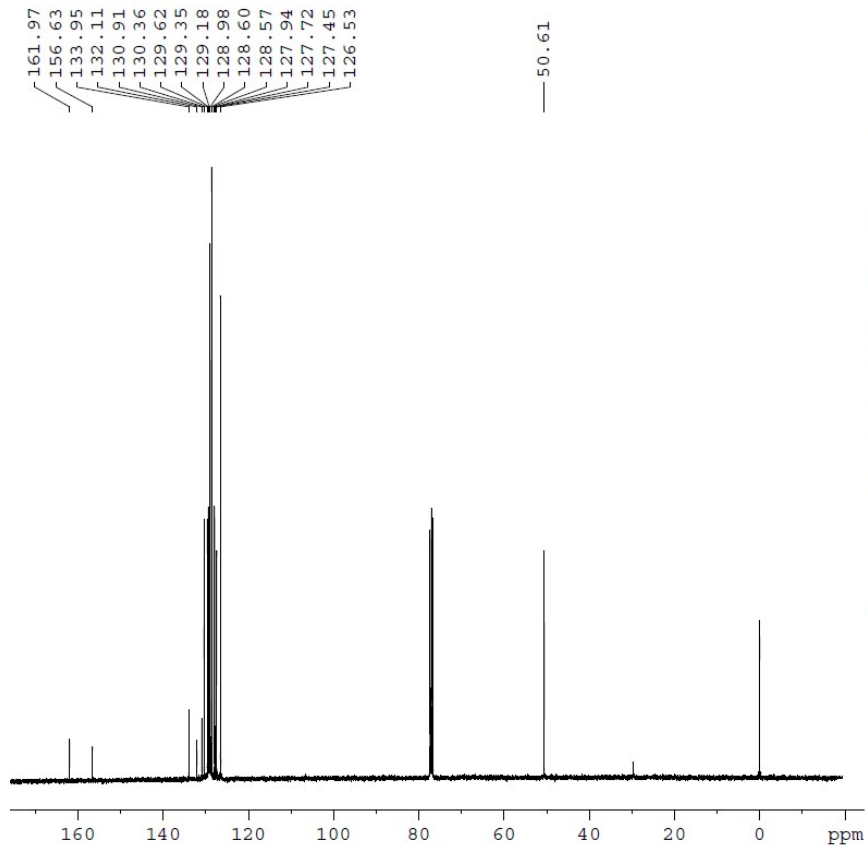
```

Current Data Parameters
NAME      STP-1 in DMSO
EXPNO    13
PROCNO   1

F2 - Acquisition Parameters
Date_    20160419
Time     16.57
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zg
TD        32768
SOLVENT  CDCl3
NS        32
DS        0
SWH       6009.615 Hz
FIDRES   0.183399 Hz
AQ        2.7262976 sec
RG        50.82
DW        83.200 usec
DE        6.50 usec
TE        298.2 K
D1        1.50000000 sec
TDO       1

===== CHANNEL f1 =====
SFO1     400.1328009 MHz
NUC1     1H
P1       14.40 usec
PLW1     11.00000000 W

F2 - Processing parameters
SI       65536
SF       400.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```



```

Current Data Parameters
NAME      STP-1 in DMSO
EXPNO    17
PROCNO   1

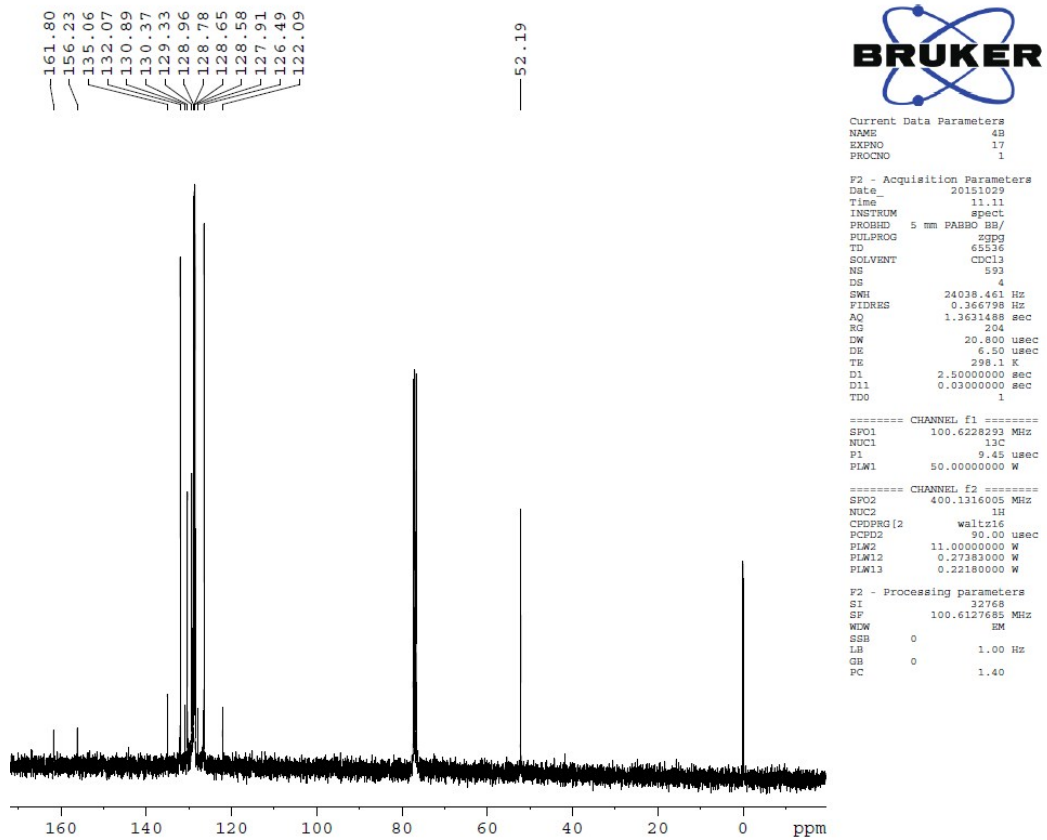
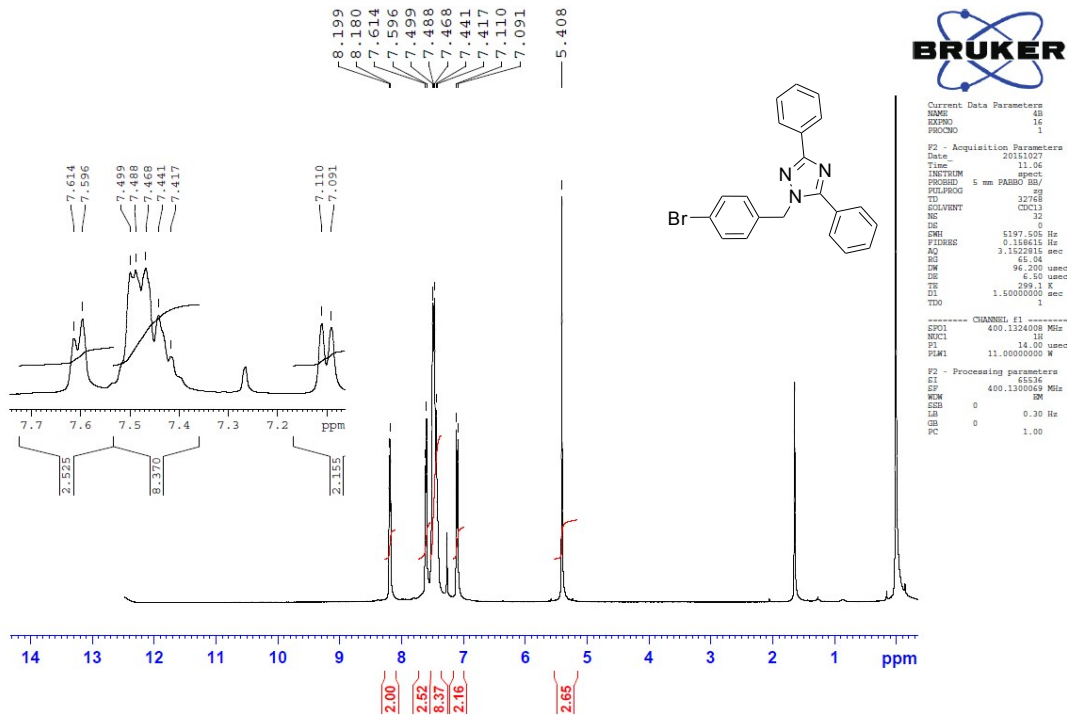
F2 - Acquisition Parameters
Date_    20160420
Time     19.00
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        4
DS        4
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631488 sec
RG        204
DW        20.800 usec
DE        6.50 usec
TE        298.1 K
D1        2.50000000 sec
D11      0.03000000 sec
TDO       1

----- CHANNEL f1 -----
SFO1     100.6228293 MHz
NUC1     13C
P1       9.45 usec
PLW1     50.00000000 W

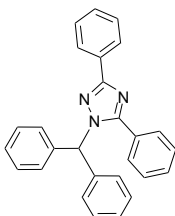
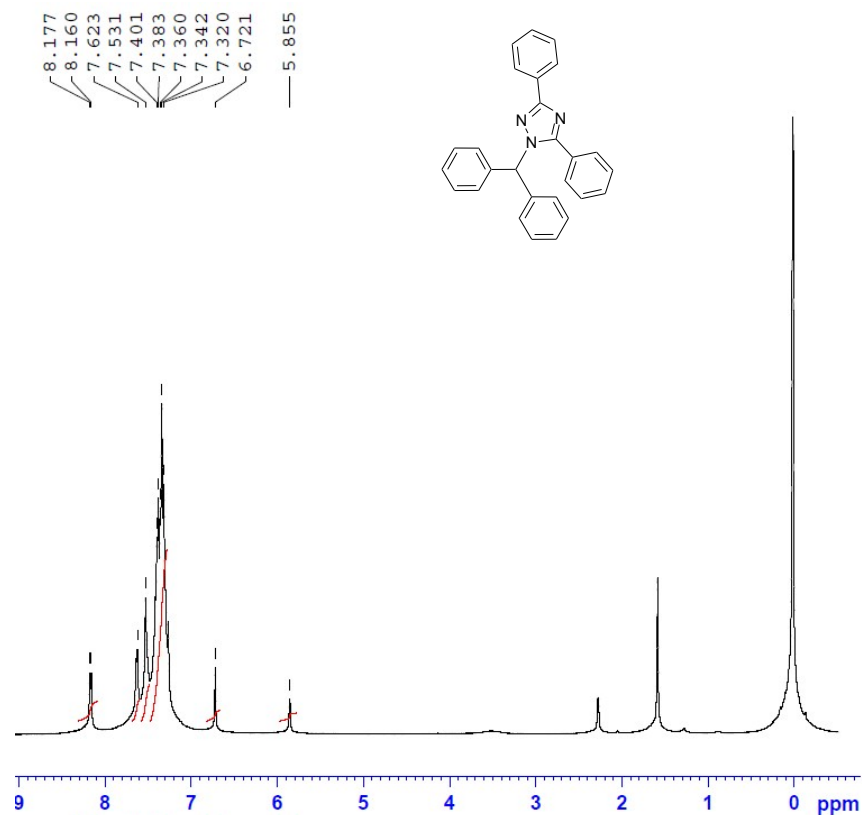
----- CHANNEL f2 -----
SFO2     400.1316005 MHz
NUC2     1H
CPDPRG2  waltz16
PCPD2    90.00 usec
PLW2     11.00000000 W
PLW12    0.27383000 W
PLW13    0.22180000 W

F2 - Processing parameters
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```


3g.



3i.



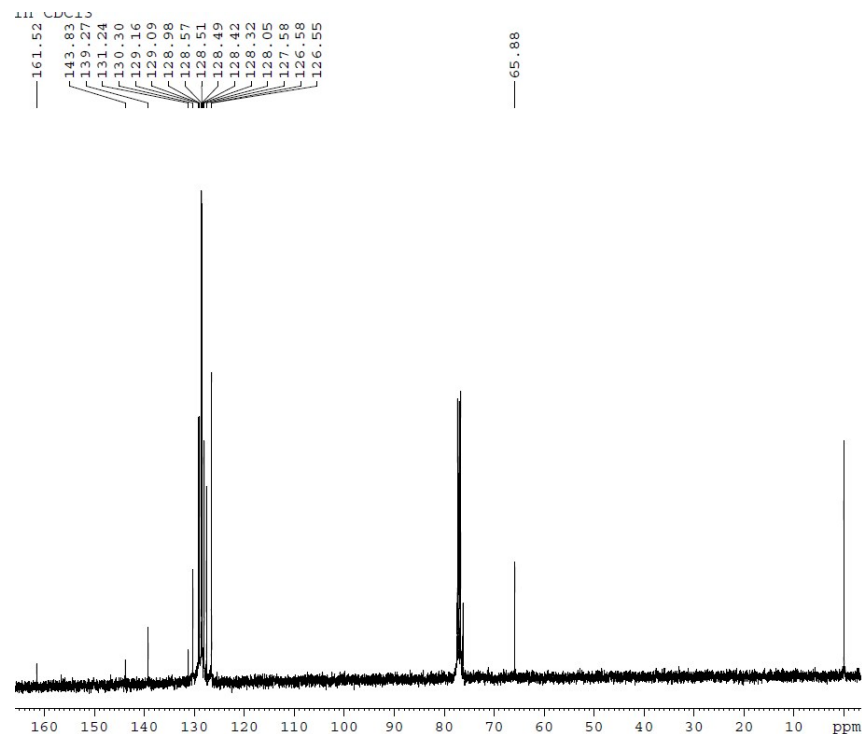
```

Current Data Parameters
NAME      4B
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20151027
Time      9.25
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zg
TD         32768
SOLVENT   CDCl3
NS         32
DS         0
SWH        5197.505 Hz
FIDRES     0.158615 Hz
AQ         3.1522815 sec
RG         65.04
DW         96.200 usec
DE         6.50 usec
TE         304.1 K
D1         1.50000000 sec
TDO        1

===== CHANNEL f1 =====
SFO1      400.1324008 MHz
NUC1       1H
P1         14.00 usec
PLM1       11.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300069 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

Current Data Parameters
NAME      4B
EXPNO     24
PROCNO    1

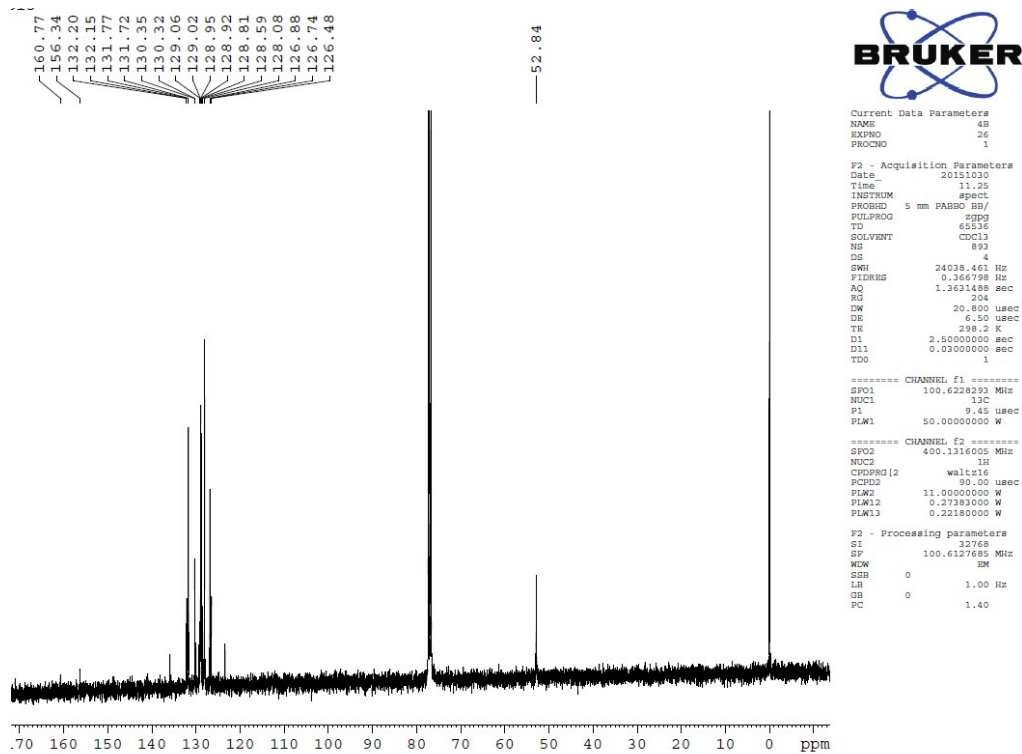
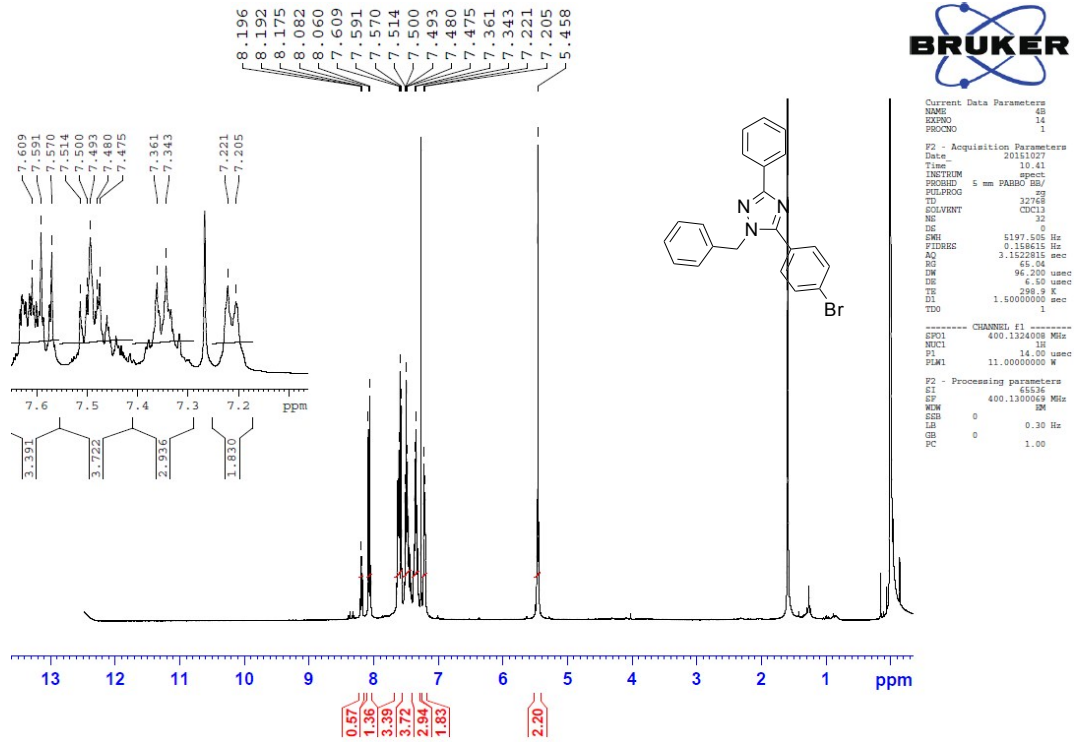
F2 - Acquisition Parameters
Date_     20151029
Time      19.39
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         147.52
DW         20.800 usec
DE         6.50 usec
TE         298.1 K
D1         2.50000000 sec
D11        0.23000000 sec
TDO        1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1       13C
P1         9.45 usec
PLM1       50.00000000 W

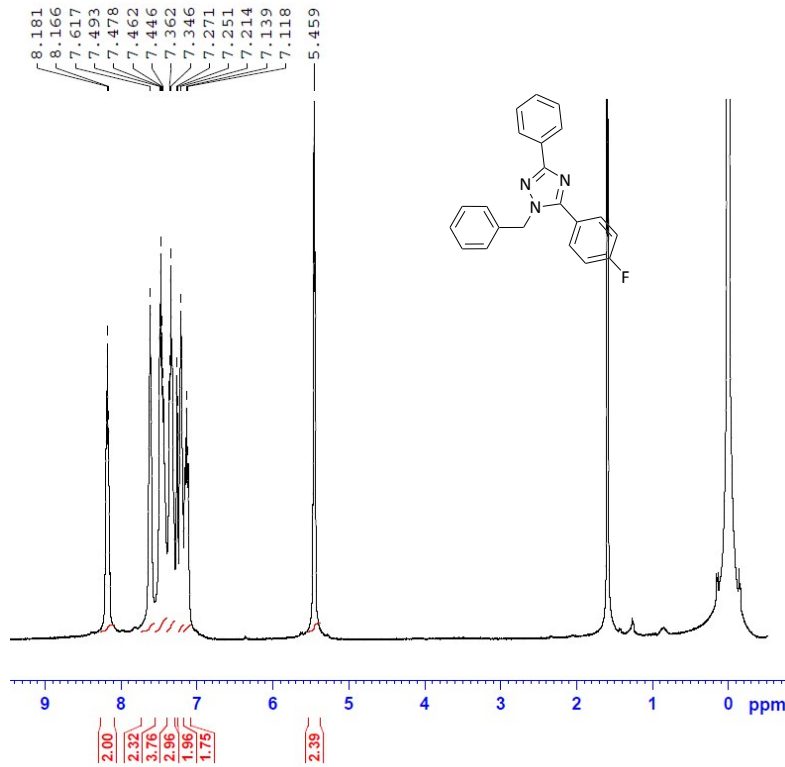
===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
CPDPRG2   waltz16
PCPD2     90.00 usec
PLM2       11.00000000 W
PLM12     0.27380000 W
PLM13     0.22180000 W

F2 - Processing parameters
SI         32768
SF         100.6127685 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

4b.



4c.



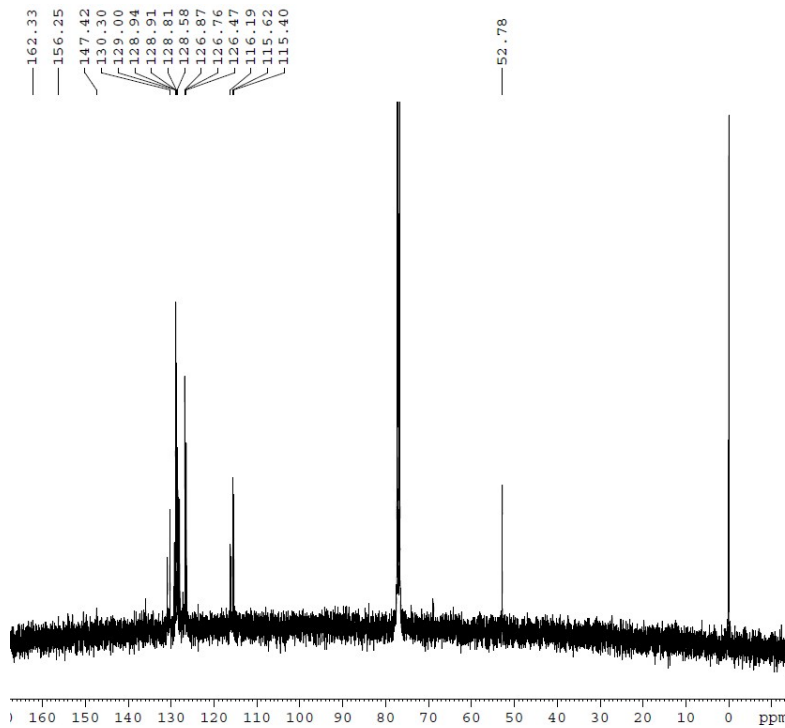
BRUKER

Current Data Parameters
 NAME 4B
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151026
 Time 19.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 26
 DS 0
 SWH 5197.505 Hz
 FIDRES 0.158615 Hz
 AQ 3.1522815 sec
 RG 65.04
 DW 96.200 usec
 DE 6.50 usec
 TE 298.1 K
 D1 1.5000000 sec
 TDO 1

***** CHANNEL f1 *****
 SFO1 400.1324008 MHz
 NUC1 1H
 P1 14.00 usec
 PLW1 11.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300069 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



BRUKER

Current Data Parameters
 NAME 4B
 EXPNO 18
 PROCNO 1

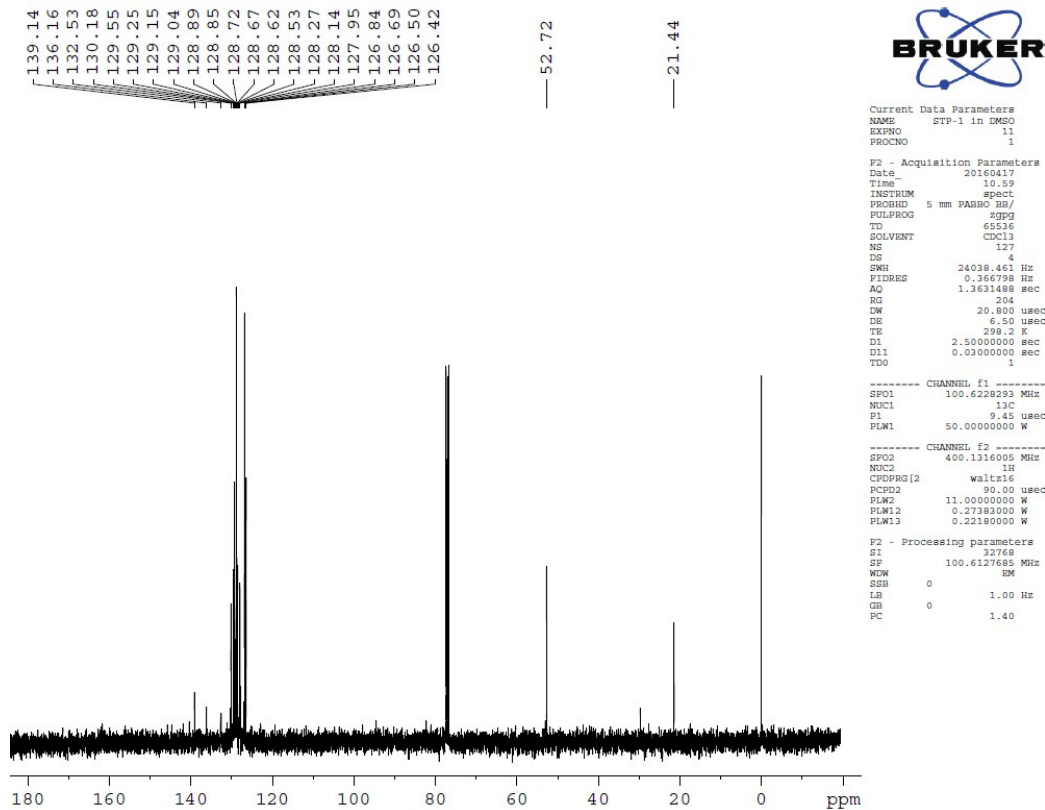
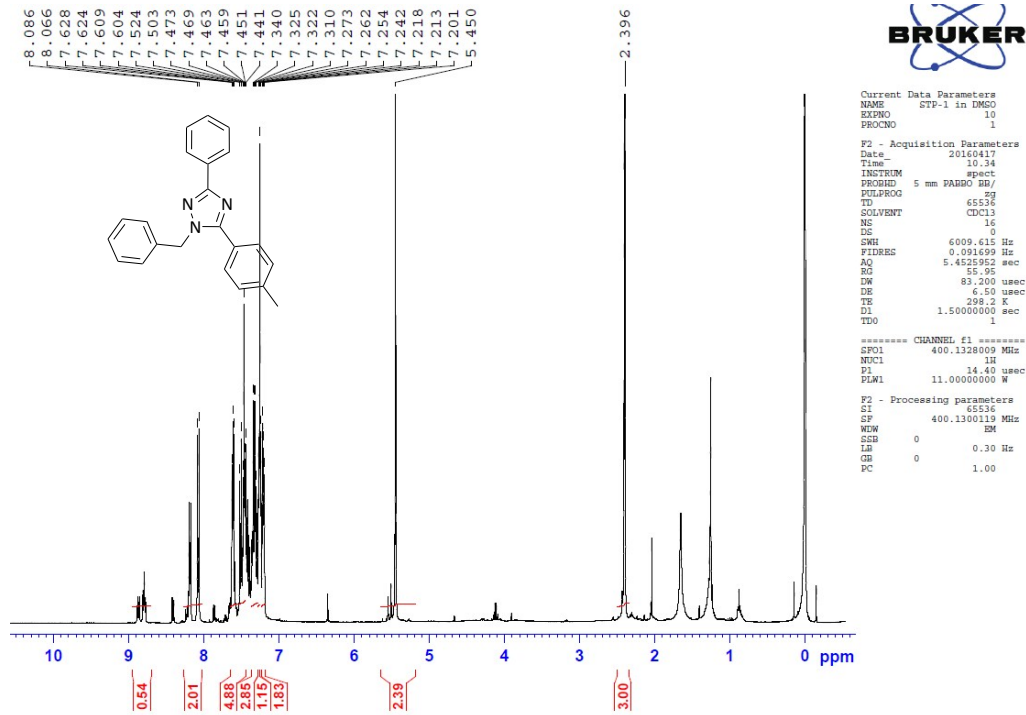
F2 - Acquisition Parameters
 Date_ 20151029
 Time 12.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 701
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 204
 DW 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.5000000 sec
 D11 0.03000000 sec
 TDO 1

***** CHANNEL f1 *****
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 9.45 usec
 PLW1 50.00000000 W

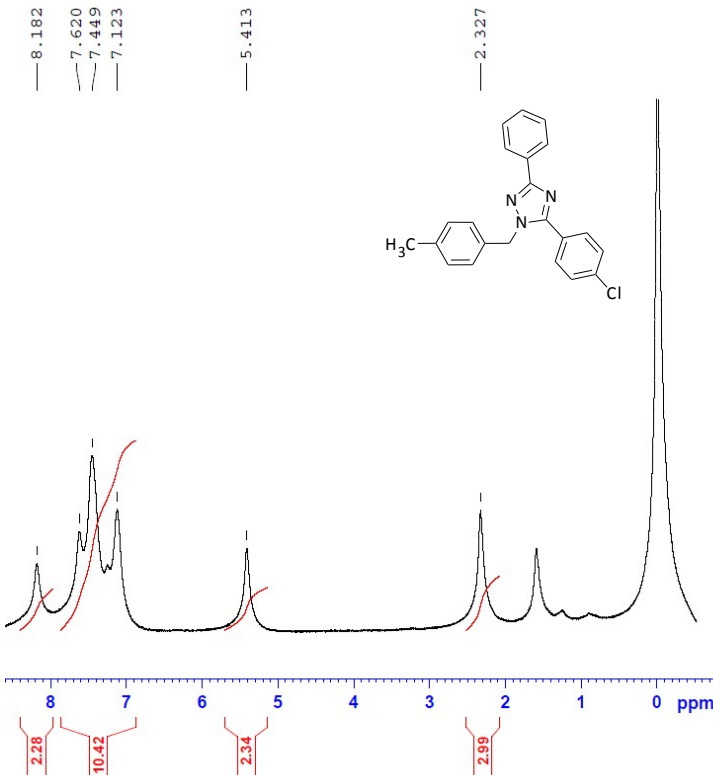
***** CHANNEL f2 *****
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCDPRG2 90.00 usec
 PLW2 11.00000000 W
 PLW12 0.27383000 W
 PLW13 0.22180000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

4d.



4e.



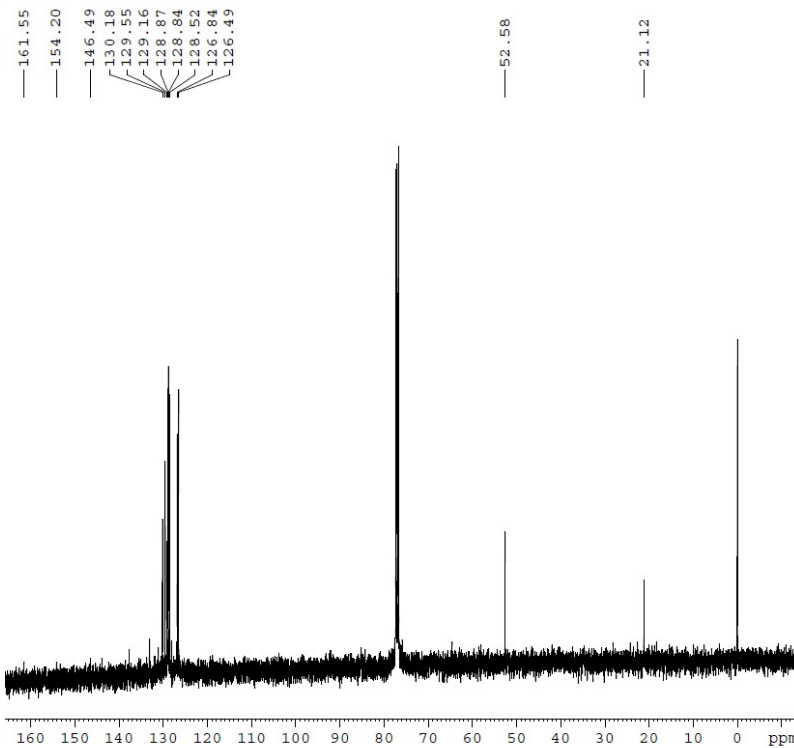
```

Current Data Parameters
NAME          4B
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20151026
Time         18.11
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg
TD           32768
SOLVENT      CDCl3
NS           32
DS           0
SWH          6009.615 Hz
FIDRES       0.183399 Hz
AQ           2.7262976 sec
RG           40.31
DW           83.200 usec
DE           6.50 usec
TE           298.2 K
D1           1.50000000 sec
TDO          1

===== CHANNEL f1 =====
SFO1         400.1328009 MHz
NUC1         1H
P1           14.00 usec
PLW1         11.00000000 W

F2 - Processing parameters
SI           65536
SF           400.1300069 MHz
WDW          EM
SGB          0
LB           0.30 Hz
GB           0
PC           1.00
  
```



```

Current Data Parameters
NAME          4B
EXPNO        29
PROCNO       1

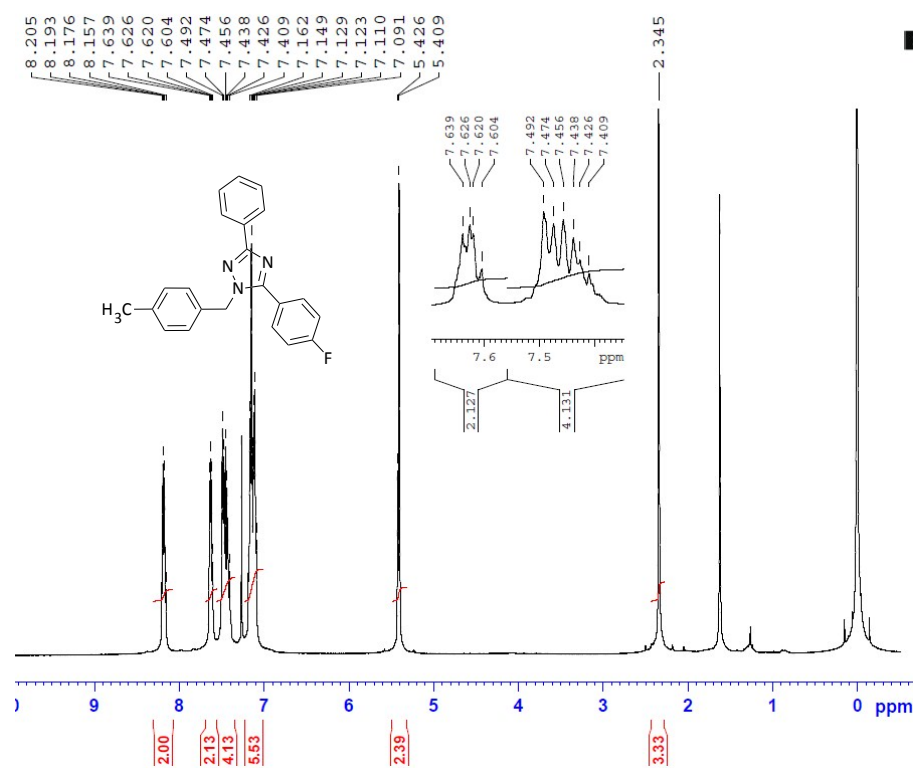
F2 - Acquisition Parameters
Date_        20151000
Time         17.27
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg
TD           65536
SOLVENT      CDCl3
NS           442
DS           4
SWH          24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631488 sec
RG           204
DW           20.800 usec
DE           6.50 usec
TE           298.2 K
D1           2.50000000 sec
D11          0.03000000 sec
TDO          1

===== CHANNEL f1 =====
SFO1         100.6228293 MHz
NUC1         13C
P1           9.45 usec
PLW1         50.00000000 W

===== CHANNEL f2 =====
SFO2         400.1316000 MHz
NUC2         1H
CPDPRG2      waltz16
PCPD2        80.00 usec
PLW2         11.00000000 W
PLW12        0.27383000 W
PLW13        0.22180000 W

F2 - Processing parameters
SI           32768
SF           100.6127685 MHz
WDW          EM
SGB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```

4f.



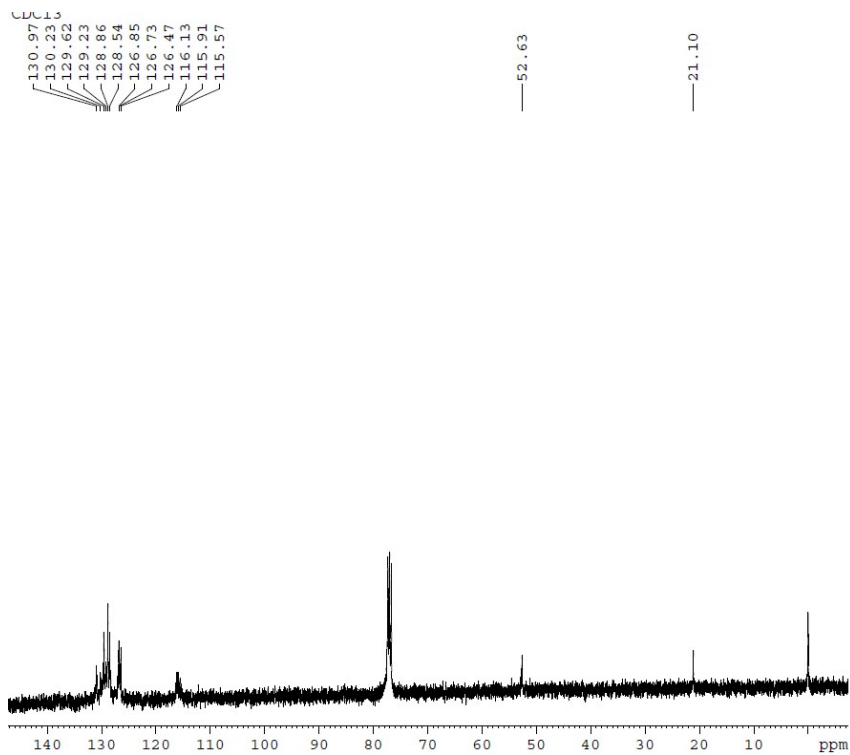
```

Current Data Parameters
NAME      4B
EXFNO    5
PROCNO   1

F2 - Acquisition Parameters
Date_    20151026
Time     18.46
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        2768
SOLVENT  CDCl3
NS        32
DS        0
SWH       5197.505 Hz
FIDRES    0.118615 Hz
AQ        3.1522815 sec
RG         63.34
DM        96.200 usec
DE         6.50 usec
TE        298.2 K
D1        1.50000000 sec
D11       0
TDO       1

===== CHANNEL f1 =====
SFO1     400.1324098 MHz
NUC1     1H
P1        14.00 usec
PLM1     11.00000000 W

F2 - Processing parameters
SI        65536
SF        400.1300049 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```



```

Current Data Parameters
NAME      4B
EXFNO    28
PROCNO   1

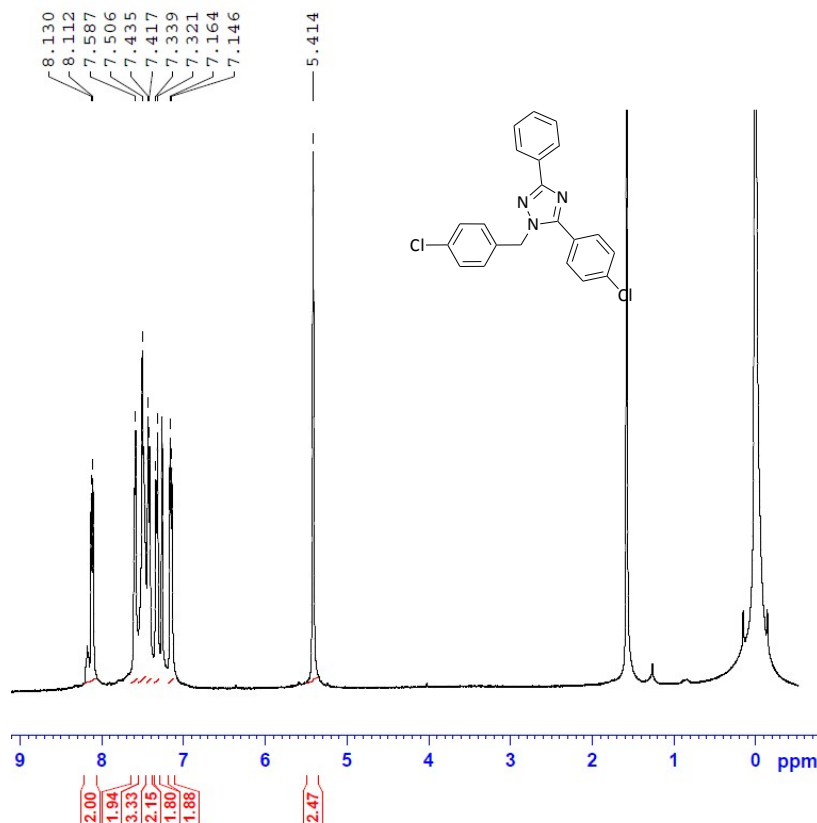
F2 - Acquisition Parameters
Date_    20151030
Time     14.29
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH       24028.461 Hz
FIDRES    0.366798 Hz
AQ        1.3621488 sec
RG         204
DM        20.800 usec
DE         6.50 usec
TE        298.2 K
D1        2.50000000 sec
D11       0.03000000 sec
TDO       1

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1        9.45 usec
PLM1     50.00000000 W

===== CHANNEL f2 =====
SFO2     400.1316005 MHz
NUC2     1H
CPDPRG2  waltz16
PCPD2    90.00 usec
PLM2     11.00000000 W
PLM12    0.27383000 W
PLM13    0.22180000 W

F2 - Processing parameters
SI        32768
SF        100.6127685 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

4g.



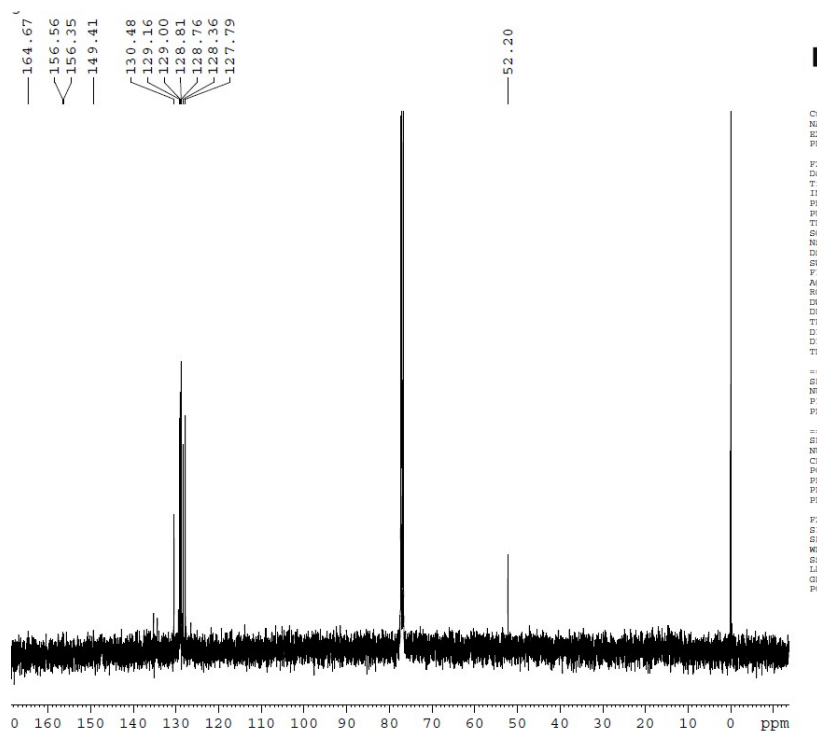
```

Current Data Parameters
NAME          4B
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        20151026
Time         18.18
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zg
TD           32768
SOLVENT      CDCl3
NS           32
DS           0
SWH          6009.615 Hz
FIDRES      0.183399 Hz
AQ          2.7262976 sec
RG          89.63
DW          83.200 usec
DE          6.50 usec
TE          298.2 K
D1          1.5000000 sec
TD0         1

===== CHANNEL f1 =====
SFO1        400.1328009 MHz
NUC1         1H
P1          14.00 usec
PLW1        11.00000000 W

F2 - Processing parameters
SI          65536
SF          400.1300669 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```



```

Current Data Parameters
NAME          4B
EXPNO        22
PROCNO       1

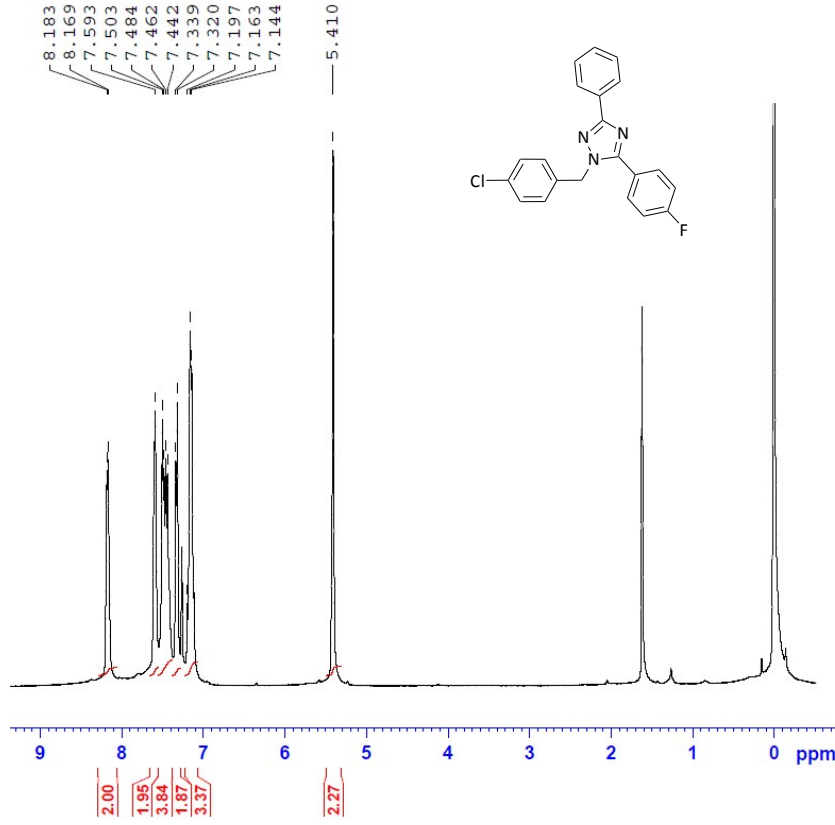
F2 - Acquisition Parameters
Date_        20151029
Time         16.34
INSTRUM      spect
PROBHD       5 mm PABBO BB/
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           585
DS           4
SWH          24038.461 Hz
FIDRES      0.366798 Hz
AQ          1.3631488 sec
RG          162.09
DW          20.800 usec
DE          6.50 usec
TE          298.3 K
D1          2.5000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
SFO1        100.6228293 MHz
NUC1         13C
P1          9.45 usec
PLW1        50.00000000 W

===== CHANNEL f2 =====
SFO2        400.1316005 MHz
NUC2         1H
CPDPRG2      waltz16
PCPD2       90.00 usec
PLW2        11.00000000 W
PLW12       0.27383000 W
PLW13       0.22180000 W

F2 - Processing parameters
SI          32768
SF          100.6127685 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```


4h.



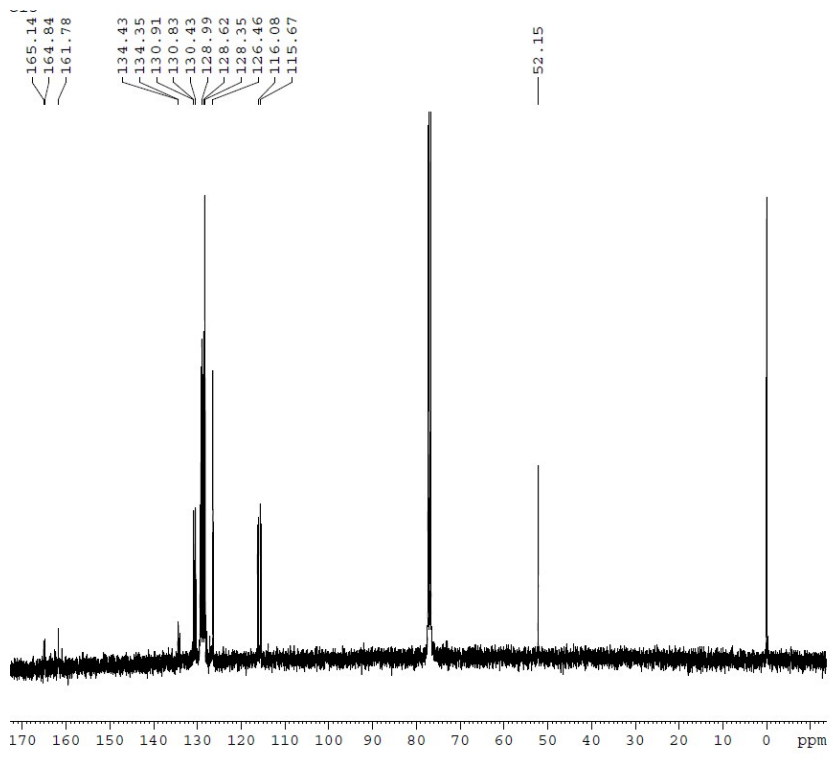
```

Current Data Parameters
NAME          4B
EXPNO        13
PROCNO       1

F2 - Acquisition Parameters
Date_        20151027
Time         10.22
INSTRUM     spect
PROBHD      5 mm PABBO BB/
PULPROG     zg
TD          32768
SOLVENT     CDCl3
NS          32
DS          0
SWH         5197.505 Hz
FIDRES     0.158615 Hz
AQ         3.1522815 sec
RG         65.04
DW         96.200 usec
DE         6.50 usec
TE         299.9 K
D1         1.50000000 sec
TDO        1

===== CHANNEL f1 =====
SFO1      400.1324008 MHz
NUC1       1H
P1        14.00 usec
PLW1      11.00000000 W

F2 - Processing parameters
SI         65536
SF         400.1300069 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



```

Current Data Parameters
NAME          4B
EXPNO        23
PROCNO       1

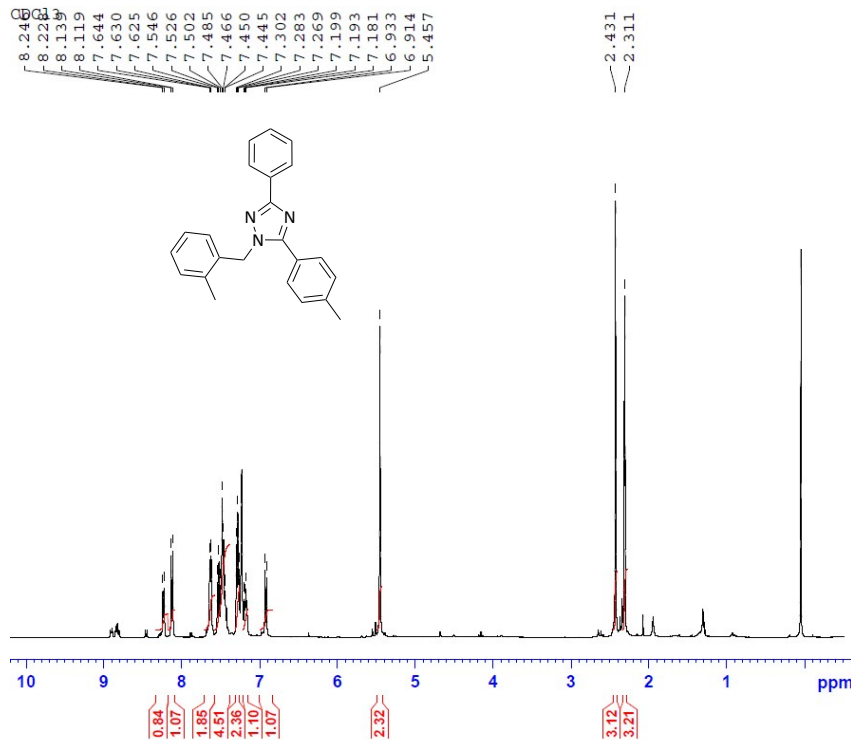
F2 - Acquisition Parameters
Date_        20151029
Time         17.37
INSTRUM     spect
PROBHD      5 mm PABBO BB/
PULPROG     zgpg30
TD          65536
SOLVENT     CDCl3
NS          1024
DS          4
SWH         24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631488 sec
RG         204
DW         20.800 usec
DE         6.50 usec
TE         298.1 K
D1         2.50000000 sec
D11        0.03000000 sec
TDO        3

===== CHANNEL f1 =====
SFO1      100.6282293 MHz
NUC1       13C
P1         9.45 usec
PLW1      50.00000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2       1H
PCPDPRG[2] waltz16
PCPD2     90.00 usec
PLW2     11.00000000 W
PLW12    0.27383000 W
PLW13    0.22180000 W

F2 - Processing parameters
SI         32768
SF         100.6174885 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

4i.



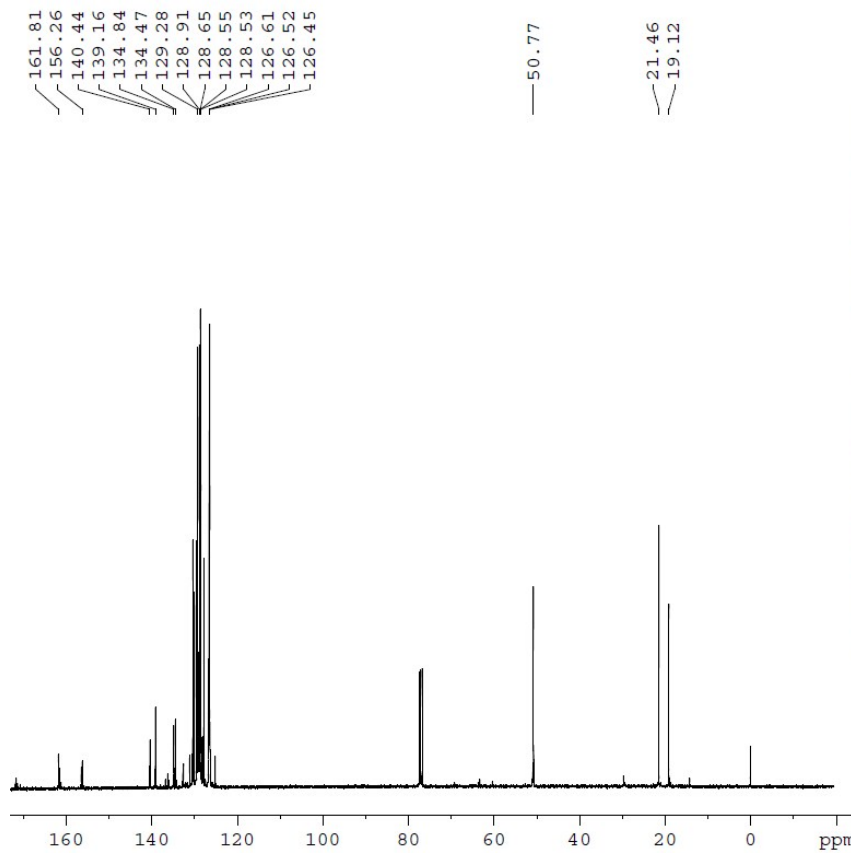
```

Current Data Parameters
NAME      STP-1 in DMSO
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20160419
Time     14.52
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        32768
SOLVENT  CDCl3
NS        32
DS        0
SWH      6009.615 Hz
FIDRES   0.183399 Hz
AQ        2.7262976 sec
RG        25.33
DW        83.200 usec
DE        6.50 usec
TE        298.2 K
D1        1.50000000 sec
TDO       1

===== CHANNEL f1 =====
SFO1    400.1328009 MHz
NUC1     1H
P1       14.40 usec
PLW1     11.00000000 W

F2 - Processing parameters
SI       65536
SF       400.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



```

Current Data Parameters
NAME      STP-1 in DMSO
EXPNO    18
PROCNO   1

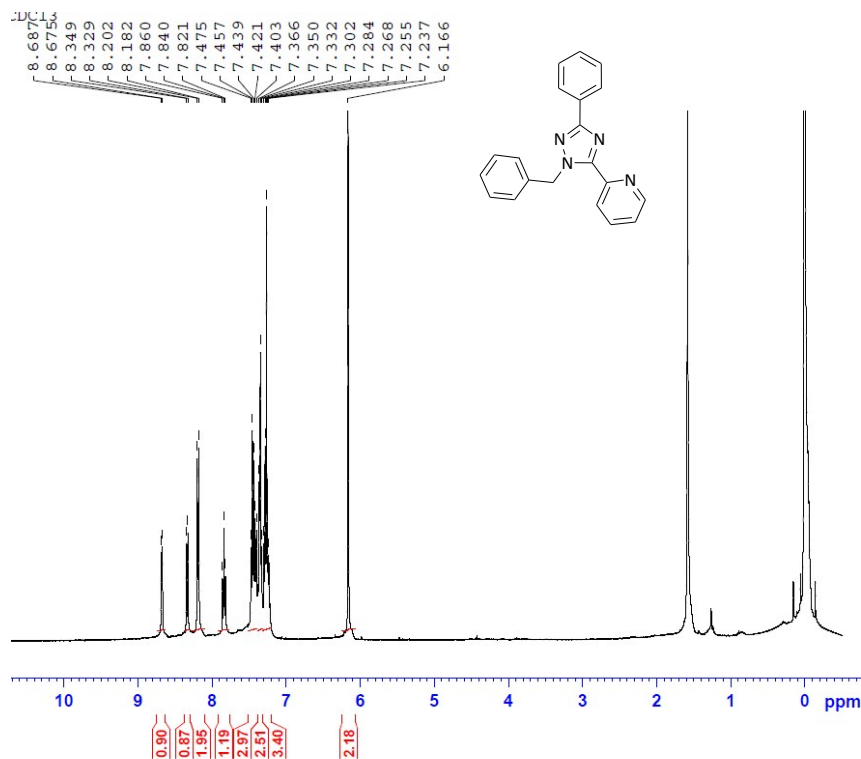
F2 - Acquisition Parameters
Date_    20160421
Time     13.23
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631488 sec
RG        204
DW        20.800 usec
DE        6.50 usec
TE        298.1 K
D1        2.50000000 sec
D11       0.03000000 sec
TDO       1

----- CHANNEL f1 -----
SFO1    100.6228293 MHz
NUC1     13C
P1       9.45 usec
PLW1     50.00000000 W

----- CHANNEL f2 -----
SFO2    400.1316005 MHz
NUC2     1H
CPDPRG2  waltz16
PCPD2    90.00 usec
PLW2     11.00000000 W
PLW12    0.27383000 W
PLW13    0.22180000 W

F2 - Processing parameters
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

4k.

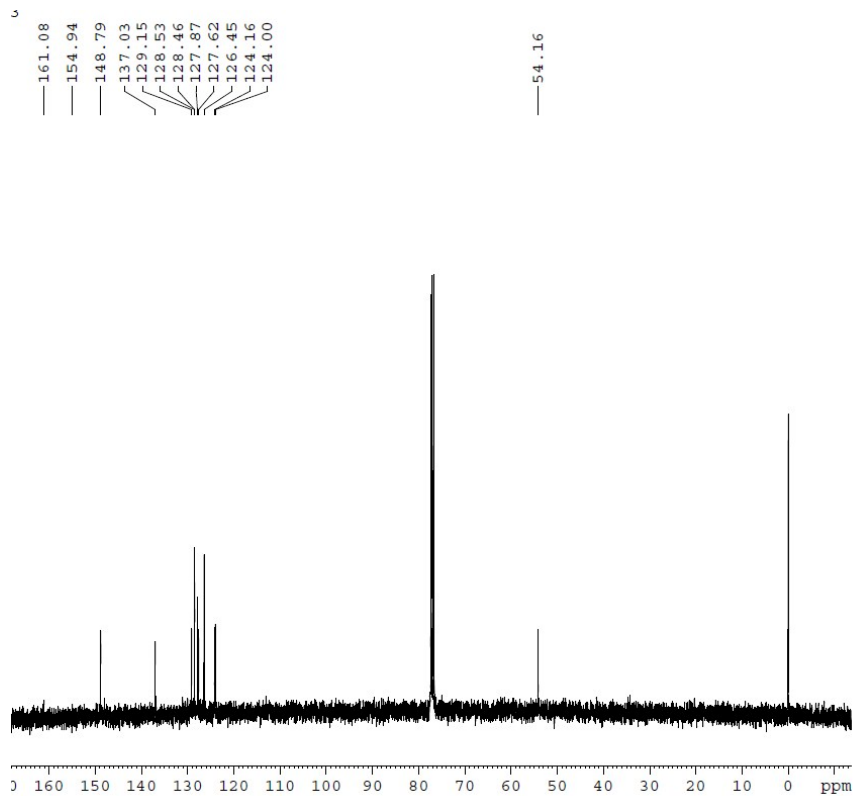


Current Data Parameters
 NAME 4B
 EXPNO 19
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20151027
 Time 10.52
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 5197.505 Hz
 FIDRES 0.158615 Hz
 AQ 3.1522815 sec
 RG 65.04
 DW 96.200 usec
 DE 6.50 usec
 TE 298.2 K
 D1 1.50000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324008 MHz
 NUCL 1H
 P1 14.00 usec
 PLW1 11.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300009 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME 4B
 EXPNO 19
 PROCNO 1

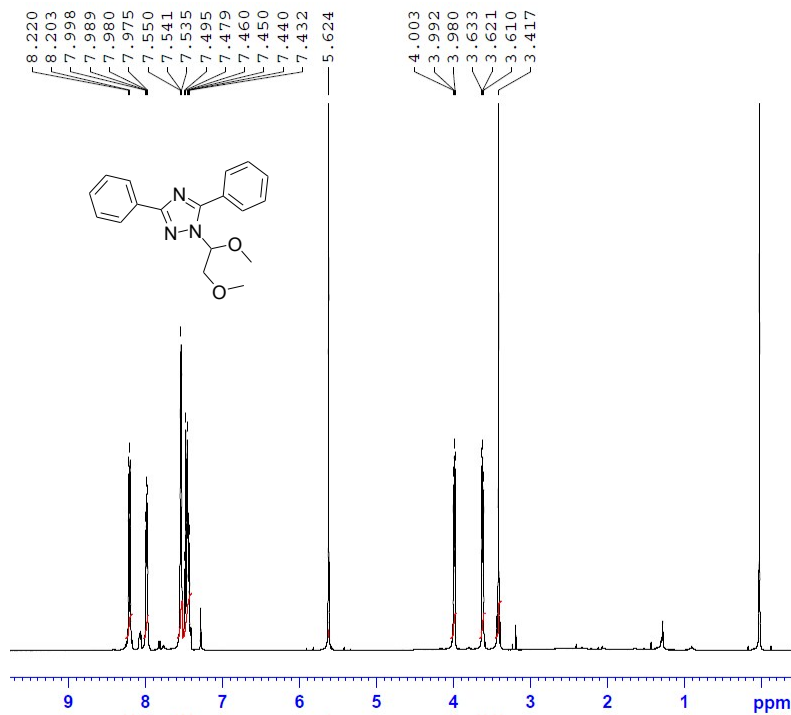
F2 - Acquisition Parameters
 Date_ 20151029
 Time 12.38
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg
 TD 65536
 SOLVENT CDCl3
 NS 473
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 162.09
 DW 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.50000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUCL 13C
 P1 9.45 usec
 PLW1 50.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUCL2 1H
 CPOPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 11.00000000 W
 PLW12 0.27383000 W
 PLW13 0.22180000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW RM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

6a.

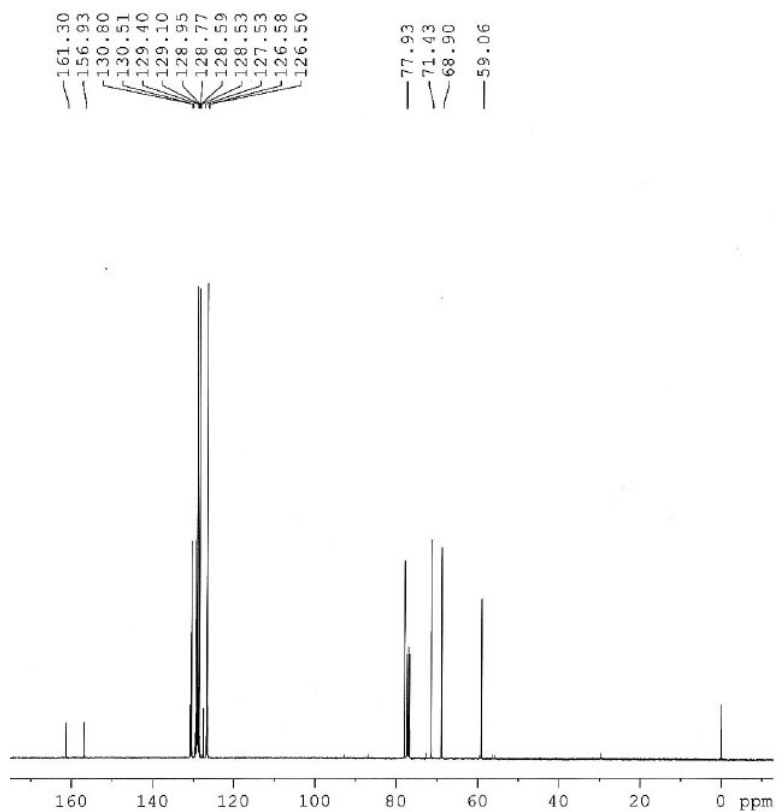


Current Data Parameters
 NAME Saa in CDCl3
 EXPNO 19
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20160531
 Time 16.51
 INSTRUM spect
 PROBEID 5 mm PABBO BB/
 PULPROG zg
 TD 65536
 SCLVENT CDCl3
 NS 16
 DS 0
 SWH 6009.615 Hz
 FIDRES 0.091699 Hz
 AQ 5.4525952 sec
 RG 32.73
 DW 83.200 usec
 DE 6.50 usec
 TE 298.1 K
 D1 1.50000000 sec
 TDO 1

***** CHANNEL f1 *****
 SFO1 400.1328009 MHz
 NUC1 1H
 P1 14.40 usec
 PLW1 11.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME Saa in CDCl3
 EXPNO 14
 PROCNO 1

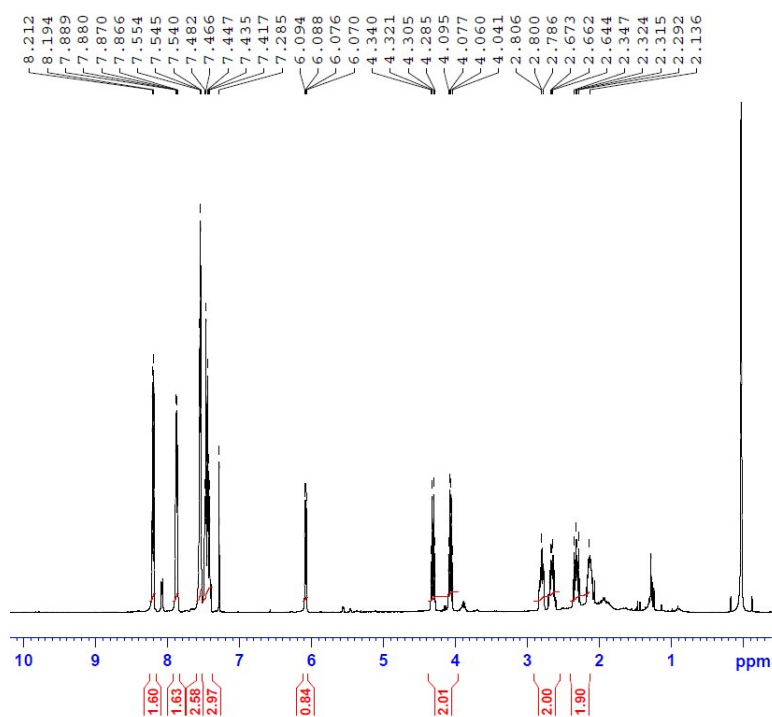
F2 - Acquisition Parameters
 Date_ 20160601
 Time 11.31
 INSTRUM spect
 PROBEID 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SCLVENT CDCl3
 NS 1024
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 204
 DW 20.800 usec
 DE 6.30 usec
 TE 298.0 K
 D1 2.50000000 sec
 D11 0.03000000 sec
 TDO 1

***** CHANNEL f1 *****
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 8.45 usec
 PLW1 50.00000000 W

***** CHANNEL f2 *****
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 FOCF2 90.00 usec
 PLW2 11.00000000 W
 PLW12 0.27383000 W
 PLW13 0.22180000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

6b.



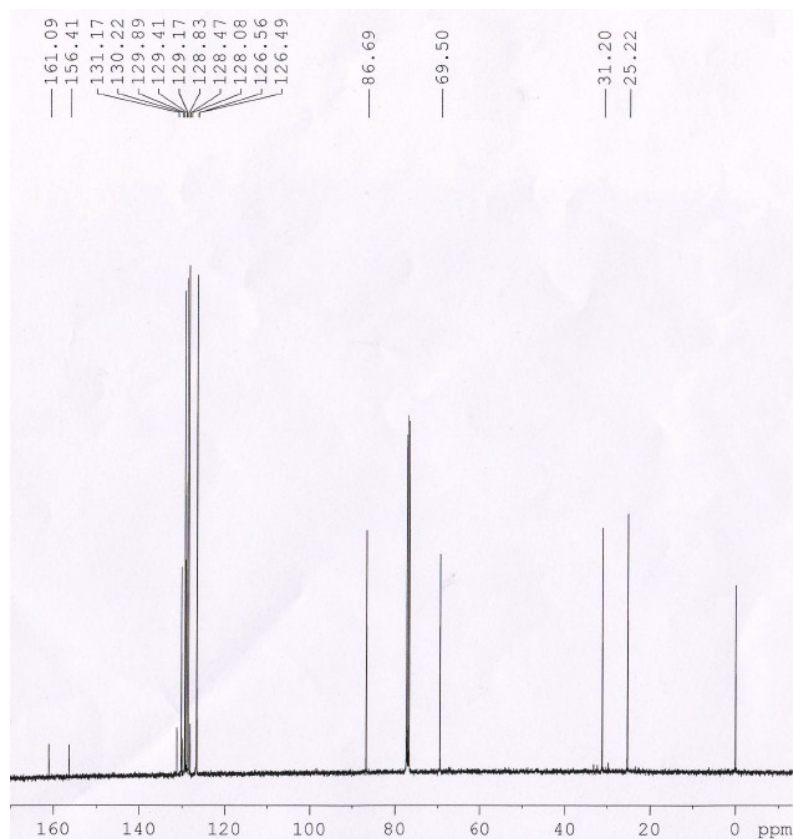
```

Current Data Parameters
NAME      5aa in CDCl3
EXPNO    3
PROCNO   1

F2 - Acquisition Parameters
Date_    20160528
Time     10.39
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        2
DS        2
SWH      6009.615 Hz
FIDRES   0.091699 Hz
AQ        5.4525952 sec
RG        63.04
RW        83.200 usec
DE        6.50 usec
TE        298.2 K
D1        1.50000000 sec
TD0       1

===== CHANNEL f1 =====
SFO1     400.130000 MHz
NUC1     1H
P1       14.40 usec
PLM1     11.00000000 W

F2 - Processing parameters
SI       65536
SF       400.130000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



```

Current Data Parameters
NAME      5aa in CDCl3
EXPNO    16
PROCNO   1

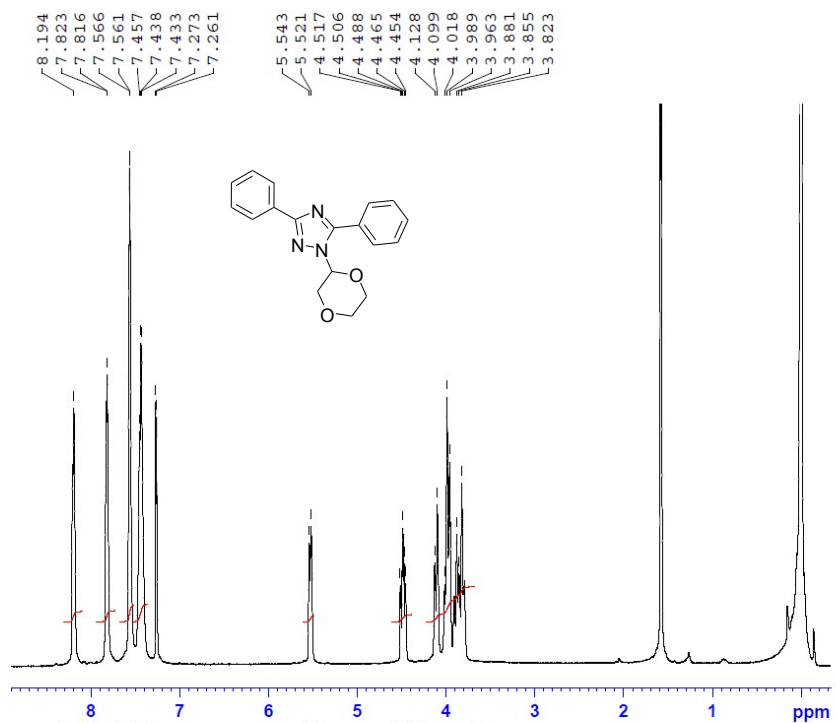
F2 - Acquisition Parameters
Date_    20160531
Time     10.55
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH      24038.461 Hz
FIDRES   0.366795 Hz
AQ        1.3631488 sec
RG        204
RW        20.800 usec
DE        6.50 usec
TE        298.2 K
D1        2.50000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
SFO1     100.6228293 MHz
NUC1     13C
P1       9.45 usec
PLM1     50.00000000 W

===== CHANNEL f2 =====
SFO2     400.1316005 MHz
NUC2     1H
CPDPRG2  waltz16
PCPDZ    90.00 usec
PLM2     11.00000000 W
PLM12    0.27383000 W
PLM13    0.22180000 W

F2 - Processing parameters
SI       32768
SF       100.6127685 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

6c.



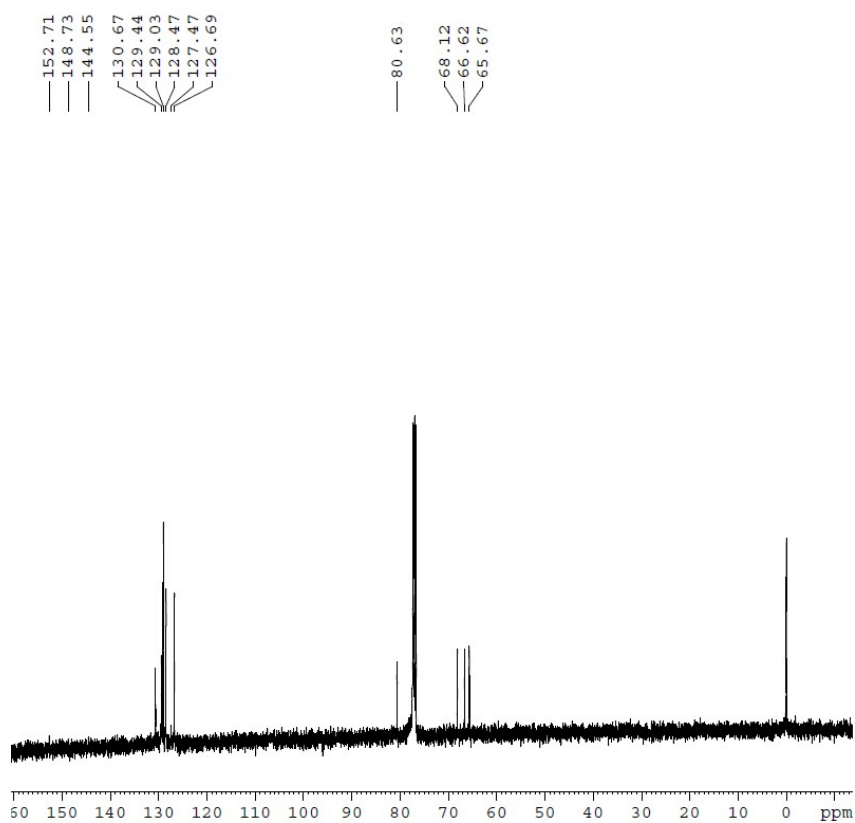
```

Current Data Parameters
NAME      4B
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20151027
Time     9.53
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG  zg
TD       32768
SOLVENT  CDCl3
NS       32
DS       0
SWH      5197.505 Hz
FIDRES   0.158415 Hz
AQ       3.1522815 sec
RG       65.04
SW       56.205 usec
DE       6.50 usec
TE       300.2 K
D1       1.50000000 sec
TDO      1

===== CHANNEL f1 =====
SFO1    400.1324008 MHz
NUC1     1H
P1      14.00 usec
PLW1    11.00000000 W

F2 - Processing parameters
SI      4533
SF      400.1300069 MHz
WDW     EM
SSB     0
LB      0.30 Hz
GB      0
PC      1.00
  
```



```

Current Data Parameters
NAME      4B
EXPNO    20
PROCNO   1

F2 - Acquisition Parameters
Date_    20151029
Time     13.54
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg
TD       65536
SOLVENT  CDCl3
NS       1024
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631488 sec
RG       162.09
DW       20.800 usec
DE       6.50 usec
TE       298.1 K
D1       2.50000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
SFO1    100.6228293 MHz
NUC1     13C
P1      9.45 usec
PLW1    50.00000000 W

===== CHANNEL f2 =====
SFO2    400.1316005 MHz
NUC2     1H
PCPD2   waltz16
PCPD2   90.00 usec
PLW2    11.00000000 W
PLW12   0.27383000 W
PLW13   0.22180000 W

F2 - Processing parameters
SI      32768
SF      100.6127685 MHz
WDW     EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.40
  
```

References

1. J. Kuang, B. Chena, S. Ma , *Org. Chem. Front.*, 2014, **1**, 186
2. Kare B. Jorgensen, R. B. Olsen, P.H.J. Carlsen, *Molecules*, 2001, **6**, 481