

Supporting Information for Publication

Regioselective One-Pot, Three-Component Synthesis of Substituted 2*H*-Indazoles from 2-Nitroarylaldehyde, Alkyne and Amine Catalyzed by CuBr/Zn(OTf)₂ System

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General experimental methods: Solvents were distilled before use. All starting materials were used as received without further purification unless otherwise indicated. The combined organic layers were dried over Na₂SO₄. Solvents were evaporated under reduced pressure. All yields given refer to isolated yields. ¹H and ¹³C NMR spectra were recorded in CDCl₃ on 600, 400 and 150, 100 MHz NMR spectrometer, respectively using TMS as internal standard. HRMS spectra were recorded using a TOF mass spectrometer. IR spectra were recorded either neat or as film on KBr pellets on a FT-IR spectrometer. Melting points were measured in open capillary tubes and are uncorrected. For thin-layer chromatography (TLC) silica gel GF₂₅₄ was used. Column chromatography was carried out using silica gel 60-120 mesh. X-ray analysis was done on single crystal X-ray diffractometer. All the reactions were performed under nitrogen atmosphere.

General procedure for the Synthesis of 2*H*-Indazoles: To a mixture of CuBr (30 mol%), *o*-nitrobenzaldehyde (0.5 mmol), and Zn(OTf)₂ (10 mol%) in 3 mL of dry toluene, amine (0.55 mmol) and alkyne (1.0mmol) were added by dissolving in 5 mL of toluene and the reaction mixture was refluxed for specified time. After completion of the reaction, the reaction was cooled, the solvent was removed under rotary evaporator, diluted with water and then extracted with ethyl acetate. The organic layer was further washed with brine solution for 2-3 times. The combined organic layers were dried over Na₂SO₄ and concentrated in rotary evaporator. The crude was subjected to column chromatography over silica gel to give the corresponding product.

2-(3,4-Dimethoxyphenethyl)-3-(phenylethynyl)-2*H*-indazole (4a):

Red solid; mp 118-120 °C; R_f (hexane/EtOAc 4:1) 0.52; yield 159 mg, 83%; ¹H NMR (400 MHz, CDCl₃): δ 7.74 (t, *J* = 9.2 Hz, 2 H), 7.53 (s, 2 H), 7.40 (s, 3 H), 7.73 (t, *J* = 7.6 Hz, 1 H), 7.17 (t, *J* = 7.2 Hz, 1 H), 6.74 (s, 2 H), 6.43 (s, 1 H), 4.77 (t, *J* = 6.8 Hz, 2 H), 3.79 (s, 3 H), 3.56 (s, 3 H), 3.27 (t, *J* = 6.8 Hz, 2 H); ¹³C NMR (100 MHz, CDCl₃): δ 149.0, 148.2, 148.0, 131.6, 130.4, 129.2, 128.6, 126.7, 124.2, 122.8, 122.3, 120.9, 120.1, 119.1, 117.9, 112.0, 111.5, 100.3, 77.0, 55.9, 55.6, 53.6, 36.7; IR (KBr, neat) 2925, 2853, 1592, 1516, 1464, 1263, 1238, 1156, 1139, 1028, 751, 690 cm⁻¹; HRMS (ESI) calcd. for C₂₅H₂₃N₂O₂ (M + H)⁺ 383.1754, found 383.1761.

2-(3,4-Dimethoxyphenethyl)-6-nitro-3-(phenylethynyl)-2H-indazole (4b):

Yellow solid; mp 160-162 °C; R_f (hexane/EtOAc 7:3) 0.50; yield 160 mg, 75%; ^1H NMR (400 MHz, CDCl_3): δ 8.75 (s, 1 H), 7.98 (d, $J = 9.2$ Hz, 1 H), 7.84 (d, $J = 9.2$ Hz, 1 H), 7.55 (d, $J = 7.6$ Hz, 2 H), 7.43 (d, $J = 5.6$ Hz, 3 H), 6.75-6.69 (m, 2 H), 6.48 (s, 1 H), 4.84 (t, $J = 6.8$ Hz, 2 H), 3.80 (s, 3 H), 3.60 (s, 3 H), 3.31 (t, $J = 7.2$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 149.2, 148.3, 147.3, 146.3, 131.8, 129.9, 129.8, 128.9, 126.4, 121.7, 121.6, 121.0, 120.7, 116.7, 116.0, 112.0, 111.7, 101.5, 75.7, 56.1, 55.8, 54.4, 36.6; IR (KBr, neat) 2923, 1561, 1529, 1519, 1503, 1346, 1267, 1235, 1159, 1029, 837, 754 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{25}\text{H}_{22}\text{N}_3\text{O}_4$ ($\text{M} + \text{H}$)⁺ 428.1605, found 428.1613.

2-(3,4-Dimethoxyphenethyl)-3-(phenylethynyl)-6-(trifluoromethyl)-2H-indazole (4c):

Pale yellow solid; mp 94-96 °C; R_f (hexane/EtOAc 7:3) 0.55; yield 178 mg, 79%; ^1H NMR (400 MHz, CDCl_3): δ 8.08 (s, 1 H), 7.83 (d, $J = 8.4$ Hz, 1 H), 7.57-7.53 (m, 2 H), 7.42 (s, 3 H), 7.33 (d, $J = 8.4$ Hz, 1 H), 6.75-6.70 (m, 2 H), 6.45 (s, 1 H), 4.81 (t, $J = 6.8$ Hz, 2 H), 3.80 (s, 3 H), 3.58 (s, 3 H), 3.29 (t, $J = 6.8$ Hz, 2 H); ^{13}C NMR (150 MHz, CDCl_3): δ 149.2, 148.2, 146.7, 131.7, 130.1, 129.6, 129.1 (q, $J = 33.0$ Hz), 128.8, 125.2, 124.6 (q, $J = 271.5$ Hz), 122.0, 121.5, 121.0, 120.0, 118.6, 116.5 (q, $J = 4.5$ Hz), 112.0, 111.6, 101.0, 76.2, 56.0, 55.7, 54.0, 36.7; ^{19}F NMR (376 MHz, $\text{C}_6\text{F}_6/\text{CDCl}_3$): δ 99.44. IR (KBr, neat) 2934, 2202, 1516, 1463, 1337, 1253, 1160, 1121, 1045, 808, 755, 688 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{26}\text{H}_{22}\text{F}_3\text{N}_2\text{O}_2$ ($\text{M} + \text{H}$)⁺ 451.1628, found 451.1628.

5-Chloro-2-(3,4-dimethoxyphenethyl)-3-(phenylethynyl)-2H-indazole (4d):

Pale yellow solid; mp 83-85 °C; R_f (hexane/EtOAc 4:1) 0.51; yield 148 mg, 71%; ^1H NMR (400 MHz, CDCl_3): δ 7.71 (s, 1 H), 7.68 (d, $J = 9.2$ Hz, 1 H), 7.54-7.51 (m, 2 H), 7.43-7.39 (m, 3 H), 7.28-7.24 (m, 1 H), 6.75-6.69 (m, 2 H), 6.46 (s, 1 H), 4.75 (t, $J = 7.2$ Hz, 2 H), 3.80 (s, 3 H), 3.59 (s, 3 H), 3.27 (t, $J = 7.2$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 149.5, 148.5, 146.9, 132.0, 130.5, 130.0, 129.8, 129.1, 128.4, 124.9, 122.4, 121.3, 119.8, 119.4, 119.3, 112.3, 112.0, 101.1, 76.7, 56.3, 56.0, 54.1, 37.0; IR (KBr, neat) 2932, 2834, 1515, 1463, 1327, 1263, 1237,

1157, 1028, 804, 756, 690 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{25}\text{H}_{22}\text{ClN}_2\text{O}_2$ ($\text{M} + \text{H}$)⁺ 417.1364, found 417.1366.

5-(Benzyloxy)-2-(3,4-dimethoxyphenethyl)-3-(phenylethynyl)-2H-indazole (4e):

Pale yellow semisolid; R_f (hexane/EtOAc 4:1) 0.51; yield 73 mg, 30%; ^1H NMR (400 MHz, CDCl_3): δ 7.66 (d, $J = 9.2$ Hz, 1 H), 7.56–7.53 (m, 2 H), 7.49 (d, $J = 7.2$ Hz, 2 H), 7.43–7.34 (m, 6 H), 7.11 (d, $J = 8.8$ Hz, 1 H), 7.04 (s, 1 H), 6.75 (s, 2 H), 6.45 (s, 1 H), 5.11 (s, 2 H), 4.73 (t, $J = 7.6$ Hz, 2 H), 3.81 (s, 3 H), 3.58 (s, 3 H), 3.26 (t, $J = 7.2$ Hz, 2 H); ^{13}C NMR (150 MHz, CDCl_3): δ 155.5, 149.2, 148.1, 145.0, 137.1, 131.7, 130.6, 129.2, 128.8, 128.7, 128.2, 127.9, 124.5, 122.6, 121.7, 121.0, 119.5, 118.4, 112.2, 111.6, 100.3, 98.3, 77.4, 70.6, 56.1, 55.7, 53.6, 36.8; IR (KBr, neat) 2925, 2850, 1633, 1516, 1464, 1262, 1236, 1189, 1027, 807, 756 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{32}\text{H}_{29}\text{N}_2\text{O}_3$ ($\text{M} + \text{H}$)⁺ 489.2173, found 489.2171.

2-(3,4-Dimethoxyphenethyl)-3-(phenylethynyl)-2H-indazol-5-yl acetate (4f):

Pale yellow semisolid; R_f (hexane/EtOAc 7:3) 0.54; yield 77 mg, 35%; ^1H NMR (400 MHz, CDCl_3): δ 7.75 (d, $J = 9.2$ Hz, 1 H), 7.54–7.51 (m, 2 H), 7.45–7.38 (m, 4 H), 7.06 (d, $J = 8.8$ Hz, 1 H), 6.76–6.72 (m, 2 H), 6.48 (s, 1 H), 4.77 (t, $J = 7.2$ Hz, 2 H), 3.80 (s, 3 H), 3.61 (s, 3 H), 3.27 (t, $J = 7.6$ Hz, 2 H), 2.34 (s, 3 H); ^{13}C NMR (150 MHz, CDCl_3): δ 170.1, 149.2, 148.2, 146.5, 146.4, 131.7, 130.3, 129.4, 128.8, 123.9, 122.8, 122.3, 121.0, 119.8, 119.3, 112.1, 111.6, 111.2, 100.8, 76.7, 56.1, 55.8, 53.9, 36.8, 21.3; IR (KBr, neat) 2921, 2851, 1757, 1655, 1516, 1465, 1261, 1208, 1158, 1028, 806, 757, 668 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{27}\text{H}_{25}\text{N}_2\text{O}_4$ ($\text{M} + \text{H}$)⁺ 441.1809, found 441.1811.

2-(3,4-Dimethoxyphenethyl)-3-(*p*-tolylethynyl)-2H-indazole (4g):

Red solid; mp 74–76 $^\circ\text{C}$; R_f (hexane/EtOAc 4:1) 0.55; yield 119 mg, 60%; ^1H NMR (400 MHz, CDCl_3): δ 7.72 (t, $J = 7.6$ Hz, 2 H), 7.41 (d, $J = 7.6$ Hz, 2 H), 7.31 (t, $J = 8.0$ Hz, 1 H), 7.19 (d, $J = 8.0$ Hz, 2 H), 7.14 (t, $J = 7.2$ Hz, 1 H), 6.73 (s, 2 H), 6.43 (s, 1 H), 4.75 (t, $J = 7.2$ Hz, 2 H), 3.79 (s, 3 H), 3.55 (s, 3 H), 3.25 (t, $J = 7.2$ Hz, 2 H), 2.39 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 149.1, 148.2, 148.0, 139.6, 131.6, 130.5, 129.5, 126.7, 124.1, 122.7, 121.0, 120.2, 119.4, 119.3,

117.9, 112.0, 111.5, 100.6, 76.4, 56.0, 55.7, 53.6, 36.7, 21.8; IR (KBr, neat) 2925, 2853, 1625, 1515, 1464, 1263, 1238, 1139, 1028, 815, 748 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{26}\text{H}_{25}\text{N}_2\text{O}_2$ ($\text{M} + \text{H}$)⁺ 397.1911, found 397.1915.

3-((4-(*tert*-Butyl)phenyl)ethynyl)-2-(3,4-dimethoxyphenethyl)-2H-indazole (4h):

Pale yellow semisolid; R_f (hexane/EtOAc 4:1) 0.56; yield 125 mg, 57%; ^1H NMR (600 MHz, CDCl_3): δ 7.76-7.71 (m, 2 H), 7.48 (d, $J = 7.8$ Hz, 2 H), 7.42 (d, $J = 7.8$ Hz, 2 H), 7.33 (t, $J = 7.8$ Hz, 1 H), 7.16 (t, $J = 7.2$ Hz, 1 H), 6.75 (s, 2 H), 6.48 (s, 1 H), 4.77 (t, $J = 7.8$ Hz, 2 H), 3.81 (s, 3 H), 3.58 (s, 3 H), 3.28 (t, $J = 7.2$ Hz, 2 H), 1.35 (s, 9 H); ^{13}C NMR (150 MHz, CDCl_3): δ 152.9, 149.2, 148.3, 148.1, 131.5, 130.5, 126.8, 125.8, 124.2, 122.8, 121.0, 120.2, 119.5, 119.5, 118.0, 112.1, 111.6, 100.7, 76.5, 56.1, 55.7, 53.7, 36.8, 35.2, 31.4 (3C); IR (KBr, neat) 2920, 1630, 1516, 1464, 1263, 1028, 747, 600 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{29}\text{H}_{31}\text{N}_2\text{O}_2$ ($\text{M} + \text{H}$)⁺ 439.2380, found 439.2385.

3-((4-Chlorophenyl)ethynyl)-2-(3,4-dimethoxyphenethyl)-2H-indazole (4i):

Colorless solid; mp 112-114 $^\circ\text{C}$; R_f (hexane/EtOAc 4:1) 0.52; yield 129 mg, 62%; ^1H NMR (400 MHz, CDCl_3): δ 7.76 (d, $J = 9.2$ Hz, 1 H), 7.70 (d, $J = 8.4$ Hz, 1 H), 7.44 (d, $J = 8.8$ Hz, 2 H), 7.39-7.32 (m, 3 H), 7.17 (t, $J = 6.8$ Hz, 1 H), 6.75-6.70 (m, 2 H), 6.38 (s, 1 H), 4.76 (t, $J = 7.6$ Hz, 2 H), 3.80 (s, 3 H), 3.55 (s, 3 H), 3.27 (t, $J = 7.6$ Hz, 2 H); ^{13}C NMR (150 MHz, CDCl_3): δ 149.1, 148.4, 148.1, 135.4, 132.8, 130.5, 129.1 (2C), 126.9, 124.4, 123.1, 121.0, 120.9, 120.1, 118.1, 112.1, 111.6, 99.2, 78.1, 56.1, 55.7, 53.8, 36.8; IR (KBr, neat) 2916, 2844, 1633, 1516, 1468, 1263, 1027, 789, 748 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{25}\text{H}_{22}\text{ClN}_2\text{O}_2$ ($\text{M} + \text{H}$)⁺ 417.1364, found 417.1364.

3-((4-Bromophenyl)ethynyl)-2-(3,4-dimethoxyphenethyl)-2H-indazole (4j):

Pale yellow semisolid; mp 104-106 $^\circ\text{C}$; R_f (hexane/EtOAc 4:1) 0.53; yield 150 mg, 65%; ^1H NMR (400 MHz, CDCl_3): δ 7.76 (d, $J = 8.8$ Hz, 1 H), 7.70 (d, $J = 8.0$ Hz, 1 H), 7.53 (d, $J = 8.4$ Hz, 2 H), 7.38-7.31 (m, 3 H), 7.17 (t, $J = 7.2$ Hz, 1 H), 6.75-6.69 (m, 2 H), 6.38 (s, 1 H), 4.76 (t, $J = 7.2$ Hz, 2 H), 3.79 (s, 3 H), 3.55 (s, 3 H), 3.27 (t, $J = 7.6$ Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 149.1, 148.3, 148.1, 132.9, 132.0, 130.4, 126.8, 124.3, 123.6, 123.0, 121.3, 120.9,

120.0, 118.9, 118.1, 112.1, 111.6, 99.2, 78.2, 56.0, 55.7, 53.7, 36.7; IR (KBr, neat) 2932, 1624, 1515, 1463, 1263, 1139, 1010, 821, 747 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{25}\text{H}_{22}\text{BrN}_2\text{O}_2$ (M + H)⁺ 461.0859, found 461.0856.

2-(3,4-Dimethoxyphenethyl)-3-((4-nitrophenyl)ethynyl)-2H-imidazole (4k):

Yellow solid; mp 154-156 °C; R_f (hexane/EtOAc 7:3) 0.55; yield 173 mg, 81%; ¹H NMR (400 MHz, CDCl_3): δ 8.25 (d, $J = 8.4$ Hz, 2 H), 7.79 (d, $J = 8.4$ Hz, 1 H), 7.71 (d, $J = 8.0$ Hz, 1 H), 7.63 (d, $J = 8.8$ Hz, 2 H), 7.37 (t, $J = 8.0$ Hz, 1 H), 7.22 (t, $J = 7.2$ Hz, 1 H), 6.72 (s, 2 H), 6.33 (s, 1 H), 4.79 (t, $J = 6.8$ Hz, 2 H), 3.77 (s, 3 H), 3.53 (s, 3 H), 3.29 (t, $J = 6.8$ Hz, 2 H); ¹³C NMR (100 MHz, CDCl_3): δ 149.1, 148.3, 148.2, 147.5, 133.6, 132.1, 130.3, 129.2, 127.0, 124.7, 123.9, 123.6, 120.9, 119.8, 118.3, 112.1, 111.5, 98.4, 82.2, 56.0, 55.7, 53.9, 36.7; IR (KBr, neat) 2927, 2851, 2202, 1592, 1516, 1463, 1342, 1262, 1238, 1140, 1107, 1028, 855, 748, cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{25}\text{H}_{22}\text{N}_3\text{O}_4$ (M + H)⁺ 428.1605, found 428.1600.

2-Phenethyl-3-(phenylethynyl)-2H-indazole (4l):

Red solid; mp 70-72 °C; R_f (hexane/EtOAc 4:1) 0.60; yield 126 mg, 78%; ¹H NMR (400 MHz, CDCl_3): δ 7.77-7.73 (m, 2 H), 7.58-7.54 (m, 2 H), 7.43-7.40 (m, 3 H), 7.36-7.16 (m, 7 H), 4.80 (t, $J = 8.0$ Hz, 2 H), 3.35 (t, $J = 8.0$ Hz, 2 H); ¹³C NMR (100 MHz, CDCl_3): δ 148.3, 137.9, 131.7, 129.2, 129.0, 128.9, 128.8, 127.0, 126.7, 124.3, 122.9, 122.4, 120.1, 118.9, 118.1, 100.6, 77.1, 53.5, 37.2; IR (KBr, neat) 2926, 1518, 1493, 1464, 1364, 1283, 1076, 747, 689 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{23}\text{H}_{19}\text{N}_2$ (M + H)⁺ 323.1543, found 323.1550.

2-Benzyl-3-(phenylethynyl)-2H-indazole (4m):

Colorless solid; mp 78-80 °C; R_f (hexane/EtOAc 9:1) 0.62; yield 74 mg, 48%; ¹H NMR (400 MHz, CDCl_3): δ 7.76 (t, $J = 7.6$ Hz, 2 H), 7.58-7.54 (m, 2 H), 7.40-7.27 (m, 9 H), 7.19 (t, $J = 7.6$ Hz, 1 H), 5.76 (s, 2 H); ¹³C NMR (100 MHz, CDCl_3): δ 148.4, 136.2, 131.7, 129.3, 129.0, 128.8, 128.7, 128.3, 128.2, 126.8, 124.7, 123.0, 122.4, 120.2, 118.3, 101.0, 77.5, 55.9; IR (KBr, neat) 2952, 2852, 1494, 1455, 1443, 1283, 1172, 1097, 748, 706, 689 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{22}\text{H}_{17}\text{N}_2$ (M + H)⁺ 309.1386, found 309.1396.

2-Allyl-3-(phenylethynyl)-2H-indazole (4n):

Pale yellow semi solid; R_f (hexane/EtOAc 9:1) 0.59; yield 76 mg, 59%; ^1H NMR (400 MHz, CDCl_3): δ 7.77 (t, $J = 8.4$ Hz, 2 H), 7.61-7.58 (m, 2 H), 7.43-7.40 (m, 3 H), 7.35-7.31 (m, 1 H), 7.20-7.16 (m, 1 H), 6.19-6.09 (m, 1 H), 5.34-5.28 (m, 2 H), 5.21 (d, $J = 6.0$ Hz, 2 H); ^{13}C NMR (150 MHz, CDCl_3): δ 148.4, 132.3, 131.7, 129.3, 128.8, 126.8, 124.7, 123.0, 122.5, 120.1, 119.2, 118.9, 118.3, 100.9, 77.2, 54.6; IR (KBr, neat) 2924, 2853, 1529, 1465, 1364, 1218, 1091, 927, 748, 689 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{15}\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 259.1230, found 259.1236.

2-Cyclohexyl-3-(phenylethynyl)-2H-indazole (4o):

Pale yellow semi solid; R_f (hexane/EtOAc 9:1) 0.61; yield 99 mg, 66%; ^1H NMR (400 MHz, CDCl_3): δ 7.77 (d, $J = 8.8$ Hz, 2 H), 7.62-7.58 (m, 2 H), 7.45-7.39 (m, 3 H), 7.31 (t, $J = 8.0$ Hz, 1 H), 7.16 (t, $J = 8.0$ Hz, 1 H), 4.82-4.74 (m, 1 H), 2.22-2.12 (m, 4 H), 2.32-1.95 (m, 2 H), 1.82-1.76 (m, 1 H), 1.57-1.34 (m, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 147.9, 131.6, 129.2, 129.0, 128.8, 126.4, 124.4, 122.7, 120.0, 118.2, 117.7, 100.8, 77.5, 61.4, 33.2, 25.9, 25.5; IR (KBr, neat) 2931, 2854, 1452, 1352, 1280, 1211, 1010, 746, 868 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{21}\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 301.1699, found 301.1696.

2-Isopropyl-3-(phenylethynyl)-2H-indazole (4p):

Pale yellow semi solid; R_f (hexane/ EtOAc 9:1) 0.62; yield 83 mg, 64%; ^1H NMR (600 MHz, CDCl_3): δ 7.80-7.77 (m, 2 H), 7.62 (t, $J = 4.2$ Hz, 2 H), 7.44-7.39 (m, 3 H), 7.33 (t, $J = 7.8$ Hz, 1 H), 7.18 (t, $J = 7.8$ Hz, 1 H), 5.26-5.20 (m, 1 H), 1.71 (d, $J = 7.2$ Hz, 6 H); ^{13}C NMR (150 MHz, CDCl_3): δ 147.9, 131.6, 129.2, 128.8, 126.4, 124.5, 122.7, 122.6, 120.0, 118.2, 117.6, 100.8, 77.3, 53.8, 22.9; IR (KBr, neat) 2923, 1641, 1459, 1347, 1276, 1235, 1082, 746, 689 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{17}\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 261.1386, found 261.1389.

2-Butyl-3-(phenylethynyl)-2H-indazole (4q):

Pale yellow semi solid; R_f (hexane/EtOAc 9:1) 0.64; yield 96 mg, 70%; ^1H NMR (400 MHz, CDCl_3): δ 7.76 (t, $J = 9.6$ Hz, 2 H), 7.62-7.59 (m, 2H), 7.43-7.40 (m, 3 H), 7.33 (t, $J = 7.2$ Hz, 1H), 7.17 (t, $J = 7.2$ Hz, 1 H), 4.60 (t, $J = 7.6$ Hz, 2 H), 2.05 (p, $J = 7.2$ Hz, 2 H), 1.44-1.36 (m, 2

H), 0.98 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 148.1, 131.6, 129.2, 128.8, 126.5, 124.4, 122.8, 122.5, 120.0, 118.8, 118.1, 100.6, 77.4, 51.8, 32.7, 20.1, 13.8; IR (KBr, neat) 2958, 2929, 2871, 2659, 2206, 2107, 1625, 1597, 1492, 1464, 1364, 1284, 1084, 746, 689 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 275.1543, found 275.1552.

2-Butyl-3-(*p*-tolylethynyl)-6-(trifluoromethyl)-2*H*-indazole (4r):

Pale yellow solid; mp 84-86 °C; R_f (hexane/EtOAc 9:1) 0.58; yield 107 mg, 60%; ^1H NMR (600 MHz, CDCl_3): δ 8.06 (s, 1 H), 7.86 (d, $J = 8.4$ Hz, 1 H), 7.50 (d, $J = 7.8$ Hz, 2 H), 7.32 (d, $J = 9.0$ Hz, 1 H), 7.23 (d, $J = 7.8$ Hz, 2 H), 4.62 (t, $J = 7.2$ Hz, 2 H), 2.41 (s, 3 H), 2.05 (p, $J = 7.2$ Hz, 2 H), 1.43-1.38 (m, 2 H), 0.98 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (150 MHz, CDCl_3): δ 146.6, 140.0, 131.7, 129.6, 128.7 (q, $J = 33.0$ Hz), 125.3, 124.7 (q, $J = 270.0$ Hz), 121.5, 119.8, 119.0, 118.4, 116.5 (q, $J = 4.5$ Hz), 101.5, 75.9, 52.2, 32.6, 21.8, 20.0, 13.8; ^{19}F NMR (376 MHz, $\text{C}_6\text{F}_6/\text{CDCl}_3$): δ 99.43; IR (KBr, neat) 2926, 1653, 1455, 1337, 1252, 1164, 1123, 1084, 1045, 809 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{20}\text{F}_3\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 357.1573, found 357.1572.

2-(*tert*-Butyl)-3-(phenylethynyl)-2*H*-indazole (4s):

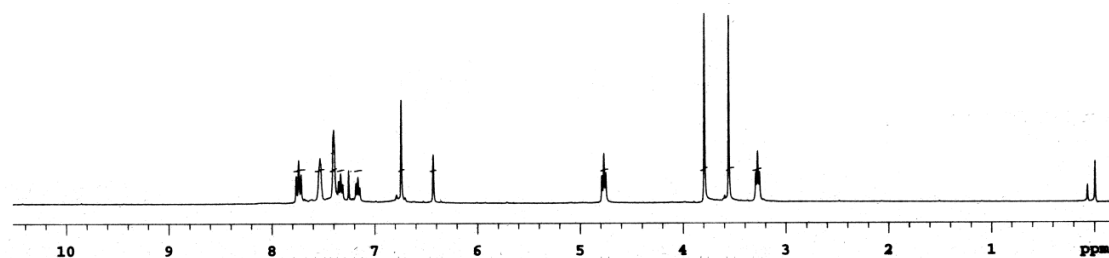
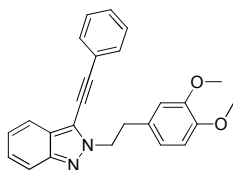
Pale yellow solid; mp 92-94 °C; R_f (hexane/EtOAc 9:1) 0.70; yield 70 mg, 51%; ^1H NMR (400 MHz, CDCl_3): δ 7.77 (d, $J = 8.4$ Hz, 2 H), 7.62-7.58 (m, 2 H), 7.44-7.39 (m, 3 H), 7.33-7.28 (m, 1 H), 7.17 (t, $J = 7.2$ Hz, 1 H), 1.97 (s, 9 H); ^{13}C NMR (100 MHz, CDCl_3): δ 146.3, 131.3, 129.1, 128.8, 128.7, 126.8, 126.3, 123.0, 122.8, 119.8, 118.4, 101.3, 79.5, 63.1, 30.1; IR (KBr, neat) 2925, 1653, 1456, 1369, 1343, 1261, 1205, 1028, 746, 688, 668 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{N}_2$ ($\text{M} + \text{H}$) $^+$ 275.1543, found 275.1544.

2-Octadecyl-3-(phenylethynyl)-2*H*-indazole (4t):

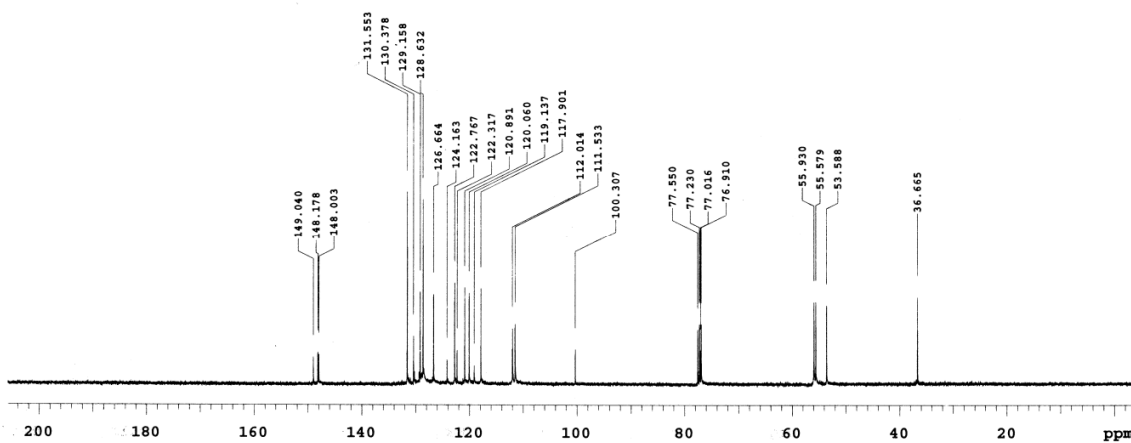
Pale yellow solid; mp 81-83 °C; R_f (hexane/EtOAc 9:1) 0.78; yield 132 mg, 56%; ^1H NMR (400 MHz, CDCl_3): δ 7.75 (t, $J = 9.2$ Hz, 2 H), 7.63-7.58 (m, 2 H), 7.43-7.39 (m, 3 H), 7.32 (t, $J = 8.0$ Hz, 1 H), 7.17 (t, $J = 7.6$ Hz, 1 H), 4.59 (t, $J = 6.8$ Hz, 2 H), 2.09-2.03 (m, 2 H), 1.37-1.21 (m, 30 H), 0.88 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 148.1, 131.6, 129.2, 128.8, 126.5, 124.4, 122.8, 122.6, 120.1, 118.7, 118.1, 100.6, 77.4, 52.1, 32.1, 30.7, 29.9 (4C), 29.8 (3C),

29.82, 29.8, 29.7, 29.6, 29.3, 26.8, 22.9, 14.3; IR (KBr, neat) 2920, 2852, 2213, 1641, 1465, 1368, 1279, 1025, 745, 688 cm^{-1} ; HRMS (ESI) calcd. for $\text{C}_{33}\text{H}_{47}\text{N}_2$ ($\text{M} + \text{H}$)⁺ 471.3734, found 471.3721.

^1H and ^{13}C spectra of compound **4a**

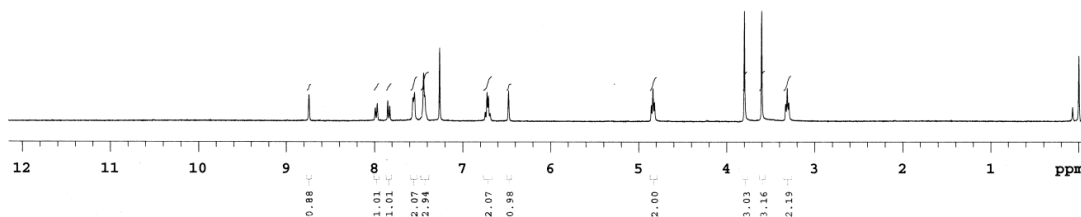
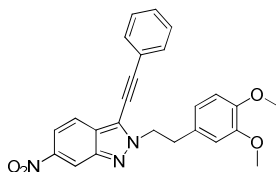


<p>PULSE SEQUENCE: DATA PROCESSING OBSERVE H1, 399.8509634</p> <p>Relax. delay 1.000 sec</p> <p>Pulse 45.0 degrees</p> <p>Acq. time 2.561 sec</p> <p>Width 6396.0 Hz</p> <p>24 repetitions</p> <p>"ITG-NMR"</p>	<p>DATA PROCESSING AU-3112</p> <p>FT size 32768</p> <p>Total time 1 minute</p>	<p>AU-3112</p> <p>Solvent: cdcl3</p> <p>Temp: 25.0 C / 298.1 K</p> <p>Operator: chem</p> <p>File: AU-3112</p> <p>Mercury-400 "ITG-NMR"</p>
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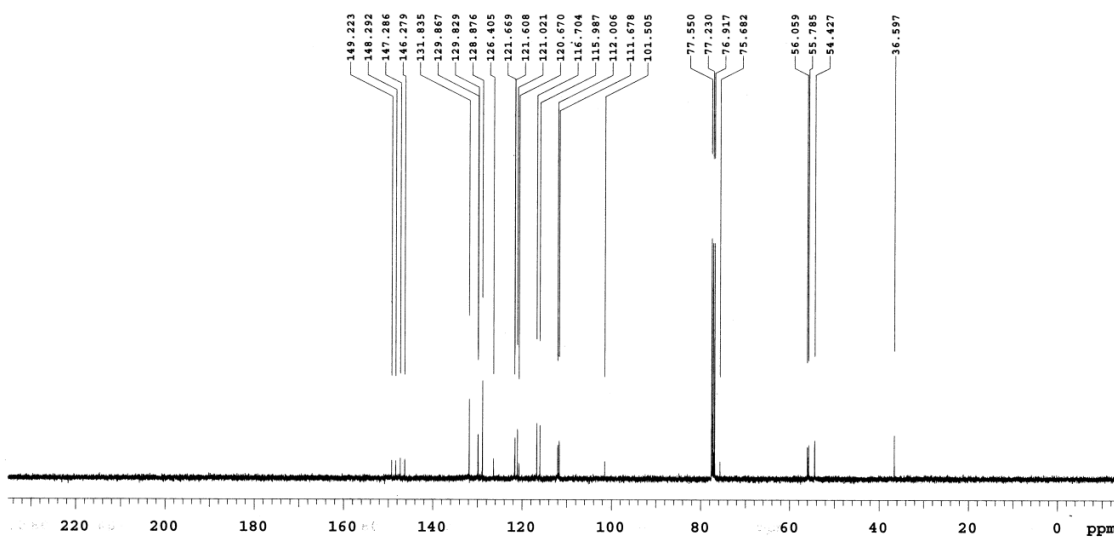


<p>PULSE SEQUENCE</p> <p>Relax. delay 1.000 sec</p> <p>Pulse 45.0 degrees</p> <p>Acq. time 1.304 sec</p> <p>Width 25125.6 Hz</p> <p>1110 repetitions</p>	<p>OBSERVE C13, 100.5425947</p> <p>DECOUPLE H1, 399.8529994</p> <p>Power: 42 dB</p> <p>continuously on</p> <p>WALTZ-16 modulated</p>	<p>DATA PROCESSING</p> <p>Line broadening 0.5 Hz</p> <p>FT size 65536</p> <p>Total time 43 minutes</p>	<p>SP-562-4-f</p> <p>Solvent: cdcl3</p> <p>Temp: 25.0 C / 298.1 K</p> <p>Operator: chem</p> <p>File: AU-1Z-SPH-H1-18-c13-2</p> <p>Mercury-400 "ITG-NMR"</p>
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^1H and ^{13}C spectra of compound **4b**

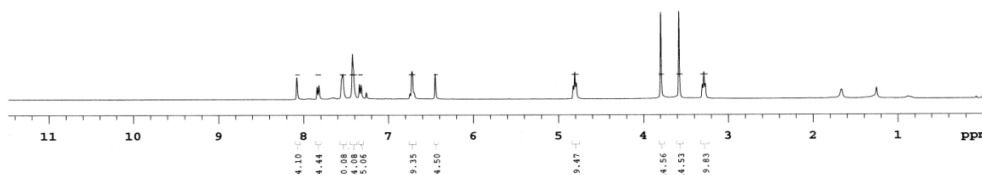
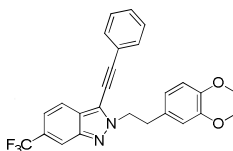


EXPERIMENTAL PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE ^1H 399.8509609 DECOUPLE H1, 399.8529994 Power: 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING FT size 32768 Total time 1 minutes Line broadening 0.5 Hz FT size 65536 Total time 51 minutes	AU-IZ-SP-NO2-H1 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 *IITG-NMR*
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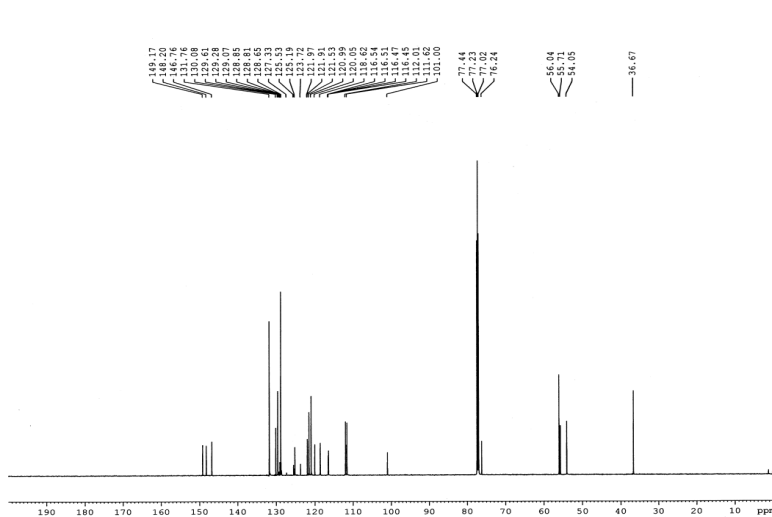
EXPERIMENTAL PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degree Acq. time 1.304 sec Width 25125.6 Hz 1340 repetitions	OBSERVE ^{13}C 100.5425824 DECOUPLE H1, 399.8529994 Power: 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 51 minutes	AU-IZ-SP-NO2-13C Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IZ-SP-NO2-13C Mercury-400 *IITG-NMR*
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¹H and ¹³C spectra of compound 4c



PULSE SEQUENCE	OBSERVE OF H1: 399.8509593	DATA PROCESSING	AU-IZCFPH-H1
Relax. delay 1.000 sec		FT size 32768	
Pulse 45.0 degrees		Total time 1 minutes	
Acq. time 2.561 sec			Solvent: cdcl3
Width 6398.0 Hz			Temp. 25.0 C / 298.1 K
32 repetitions			Operator: chm
			Mercury-400 *IITG-NMR*

AU-IZCF-SP-Ph_13C



```

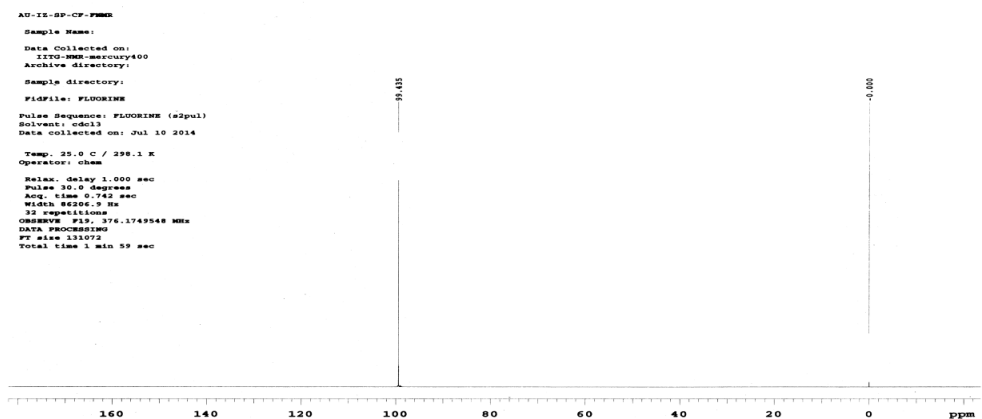
Current Data Parameters
NAME: AU-IZCF-SP-Ph_13C
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20141015
Time: 10.10
INSTRUM: spect
PROBHD: 5 mm PABBO MMV
PULPROG: zgpg30
TD: 32768
SOLVENT: CDCl3
NS: 4000
DS: 2
SWH: 36057.651 Hz
FIDRES: 1.100293 Hz
AQ: 0.452325 sec
RG: 65.24
DM: 13.867 usec
DE: 6.50 usec
TE: 298.4 K
D1: 2.0000000 sec
D11: 0.0300000 sec
TDO: 1

===== CHANNEL f1 =====
SF01: 150.9279571 MHz
NUC1: 13C
P1: 10.50 usec
PLW1: 95.0000000 W

===== CHANNEL f2 =====
SF02: 600.1724007 MHz
NUC2: 1H
CPDPRG2: waltz16
P1P2: 70.00 usec
PLW2: 21.0000000 W
PLW12: 0.6174000 W
PLW13: 0.30239999 W

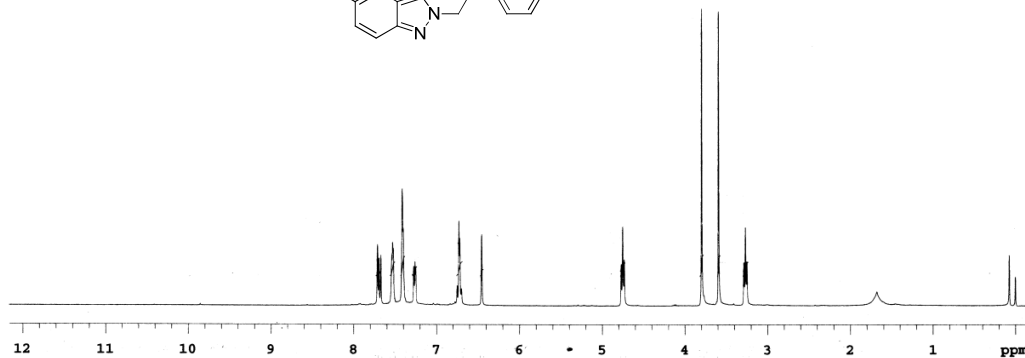
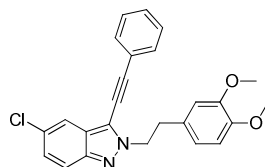
F2 - Processing parameters
SI: 32764
SF: 150.9128370 MHz
WSW: 2K
SBB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40
    
```



```

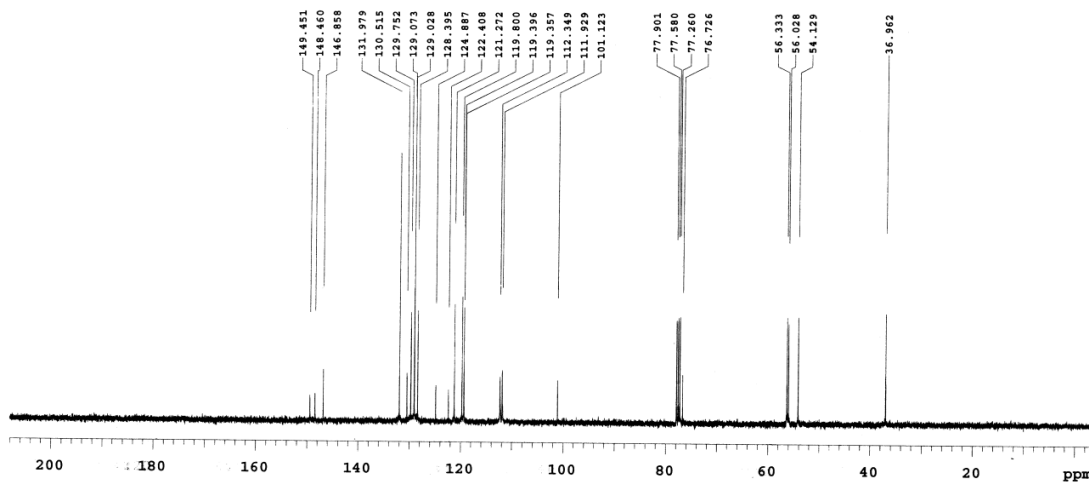
AU-IZ-SP-CF-PHMR
Sample Name:
Data Collected on:
IITG-NMR-mercury400
Archive directory:
Sample directory:
File: FLUORINE
Pulse Sequence: FLUORINE (zgpg1)
Solvent: cdcl3
Data collected on: Jul 10 2014
Temp. 25.0 C / 298.1 K
Operator: chm
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.743 sec
Width 86206.9 Hz
32 repetitions
OBSERVE F19: 376.1749548 MHz
DATA PROCESSING
FT size 131072
Total time 1 min 55 sec
    
```

¹H and ¹³C spectra of compound **4d**



```

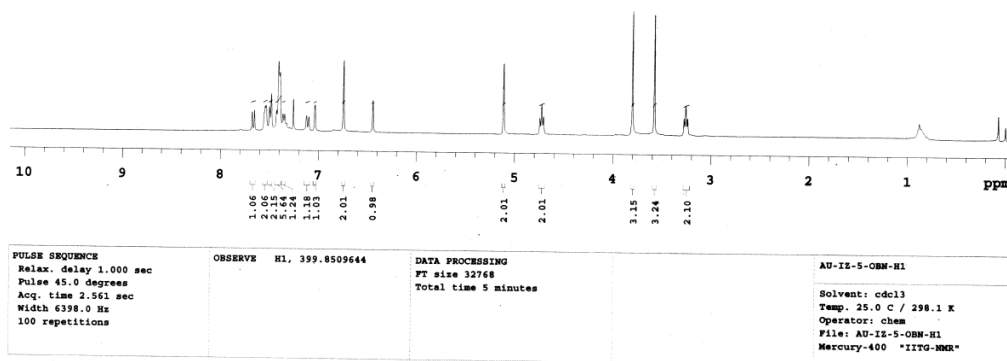
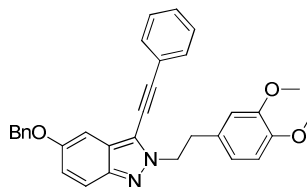
PULSE SEQUENCE      OBSERVE: 311: 399.8509632      DATA PROCESSING      AU-12-SCL-5-1H
Relax. delay 1.000 sec      FT size 32768
Pulse 45.0 degree      Total time: 1 minutes
Acq. time 2.561 sec
Width 6398.0 Hz
32 repetitions
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: chem
File: AU-12-SCL-5-1H
Mercury-400 *1TG-NMR*
    
```



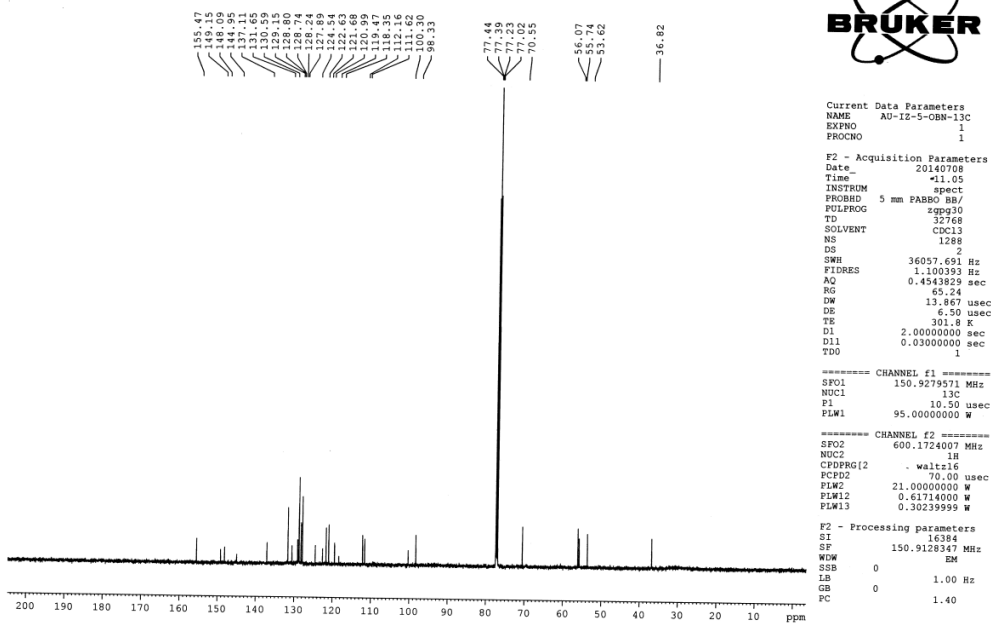
```

PULSE SEQUENCE      OBSERVE: 313: 108.5425541      DATA PROCESSING      EG-73-1H
Relax. delay 1.000 sec      DECOUPLE H1, 399.8529994      Line broadening 0.5 Hz
Pulse 45.0 degree      Power: 42.0 dB      FT size 65536
Acq. time 1.304 sec      continuously on      Total time 18 minutes
Width 25125.6 Hz      WALTZ-16 modulated
470 repetitions
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: chem
Mercury-400 *1TG-NMR*
    
```

^1H and ^{13}C spectra of compound **4e**



AU-IZ-5-OBN-13C



```

Current Data Parameters
NAME      AU-IZ-5-OBN-13C
EXPNO    1
PROCNO   1

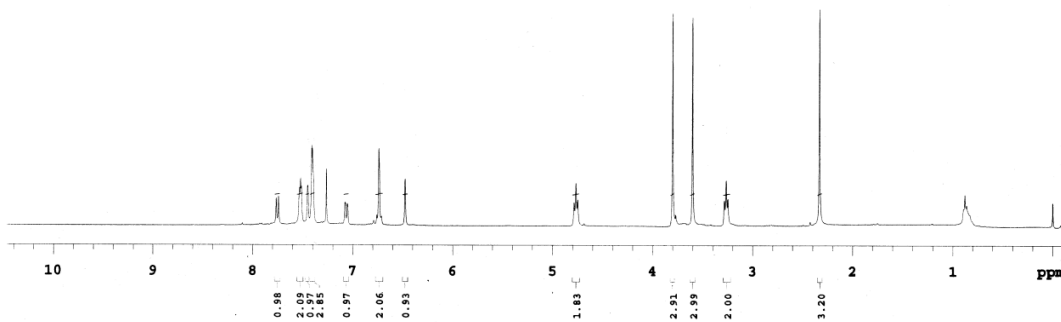
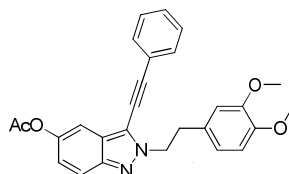
F2 - Acquisition Parameters
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Time     11:05
INSTRUM  spect
PROBHD   5 mm PABBO BB/
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        1288
DS        2
SWH       36057.691 Hz
FIDRES    1.100393 Hz
AQ         0.4543829 sec
RG         65.24
DW         13.867 usec
DE         6.50 usec
TE         301.8 K
D1         2.0000000 sec
D11        0.0300000 sec
TDD        1

===== CHANNEL f1 =====
SF01      150.9279571 MHz
NUC1       13C
P1         10.50 usec
PLW1       95.0000000 W

===== CHANNEL f2 =====
SF02      600.1724007 MHz
NUC2       1H
CPDPRG2   . waltz16
PCPD2     70.00 usec
PLW2      21.0000000 W
PLW12     0.61714000 W
PLW13     0.30239999 W

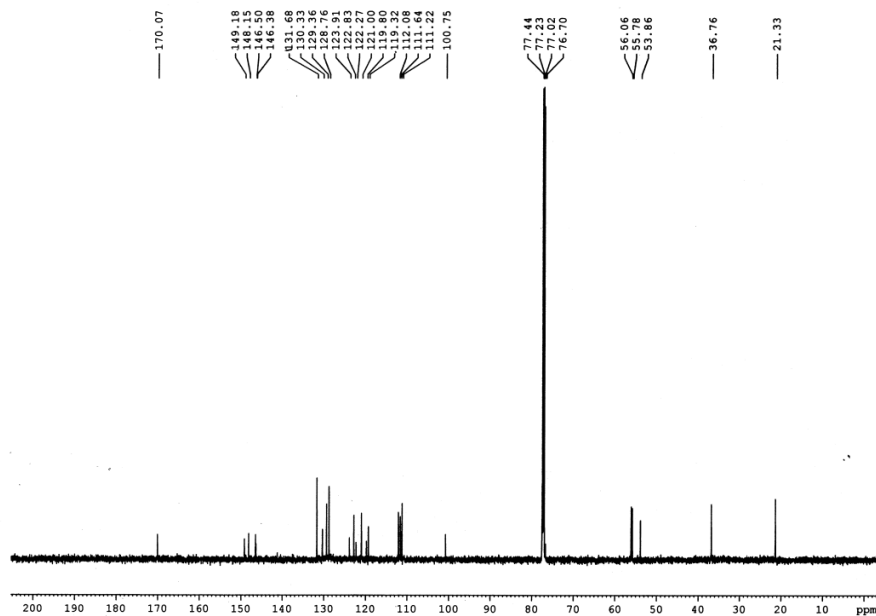
F2 - Processing parameters
SI         16384
SF         150.9128347 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

¹H and ¹³C spectra of compound **4f**



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509625	DATA PROCESSING FT size 32768 Total time 1 minutes	AU-IZ-OAc-5-1H Solvent: cdc13 Temp: 25.0 C / 298.1 K Operator: chem File: AU-IZ-OAc-5-1H Mercury-400 *17G-MMR*
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AU-IZ-5OAc_13C



```

Current Data Parameters
NAME      AU-IZ-5OAc_13C
EXPNO    1
PROCNO   1

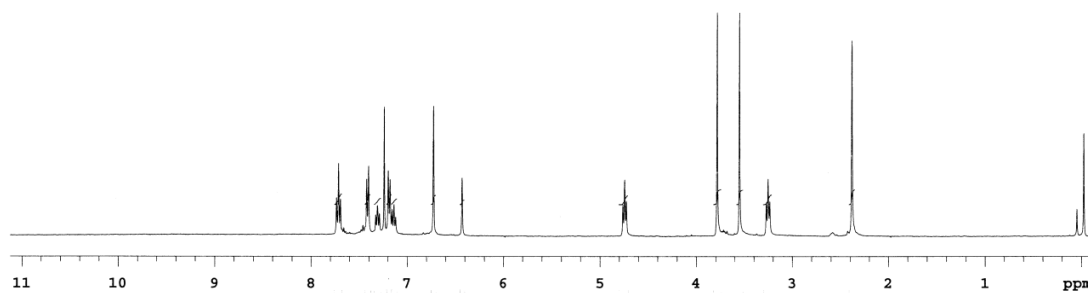
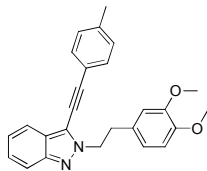
F2 - Acquisition Parameters
Date_    20140708
Time     19.47
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG zgpg30
TD       32768
SOLVENT  CDCl3
NS       417
DS       2
SWH      36057.691 Hz
FIDRES   1.100393 Hz
AQ       0.4543829 sec
RG       65.24
DM       13.867 usec
DE       6.50 usec
TE       301.3 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1

===== CHANNEL f1 =====
SFO1     150.9279571 MHz
NUC1     13C
P1       10.50 usec
PLW1     95.00000000 W

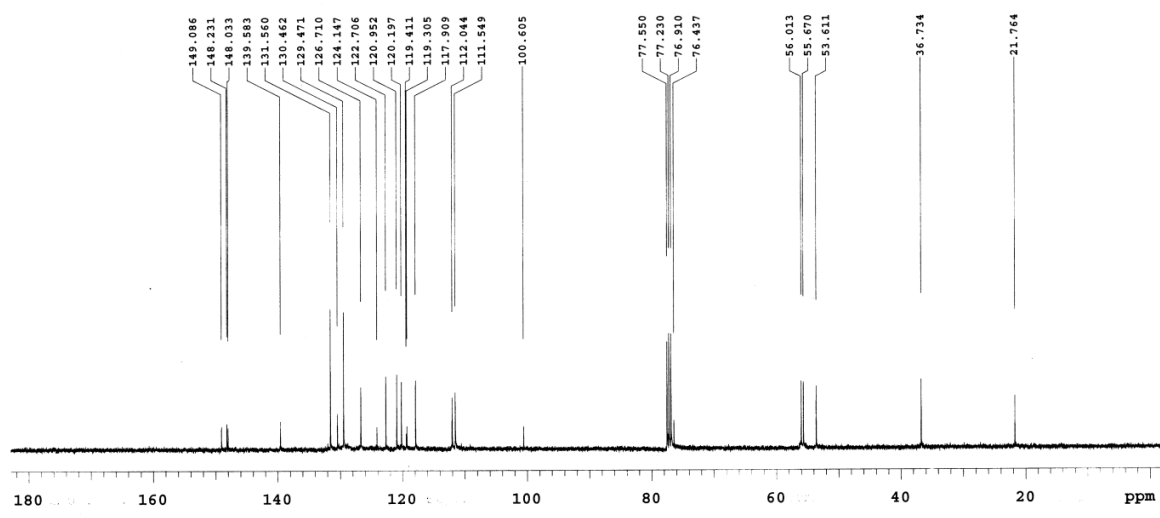
===== CHANNEL f2 =====
SFO2     600.1724007 MHz
NUC2     1H
CPDPRG2  waltz16
PCPD2    70.00 usec
PLW2     21.00000000 W
PLW12    0.61714000 W
PLW13    0.30239999 W

F2 - Processing parameters
SI       16384
SF       150.9128347 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

¹H and ¹³C spectra of compound **4g**



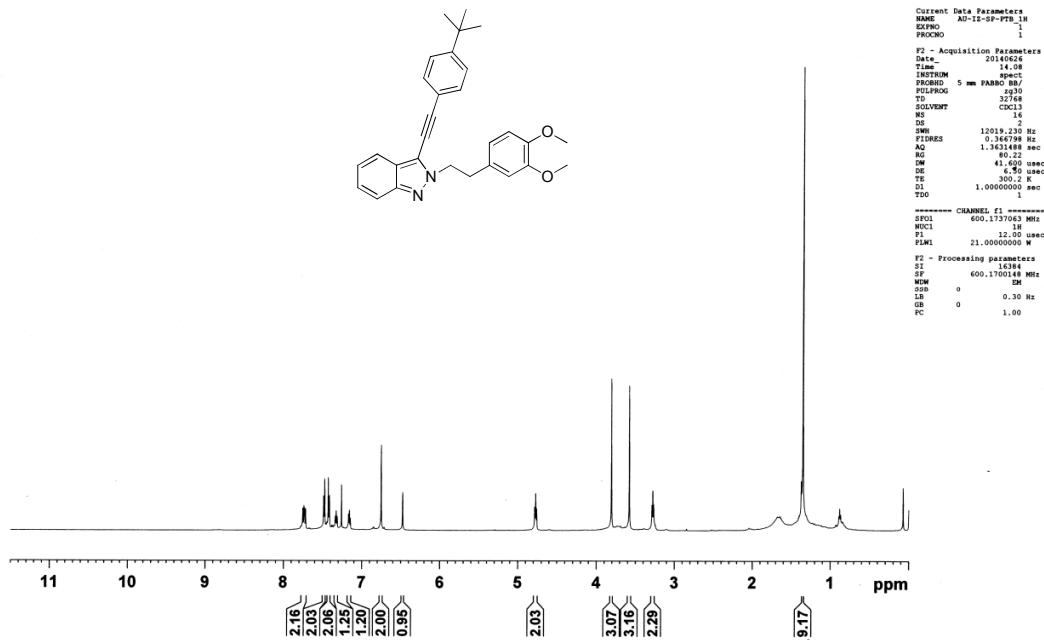
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509695	DATA PROCESSING Line broadening 1.0 Hz FT size 32768 Total time 1 minutes	aks-aa Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: aks-aa Mercury-400 *IITG-NMR*
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PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 25125.6 Hz 1650 repetitions	OBSERVE CDCl3, 100.625855 DECOUPLE H1, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 63 minutes	AU-IQPHMe2-C13 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IQPHMe2-C13 Mercury-400 *IITG-NMR*
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¹H and ¹³C spectra of compound 4h

AU-IZ-SP-PTB_1H



```

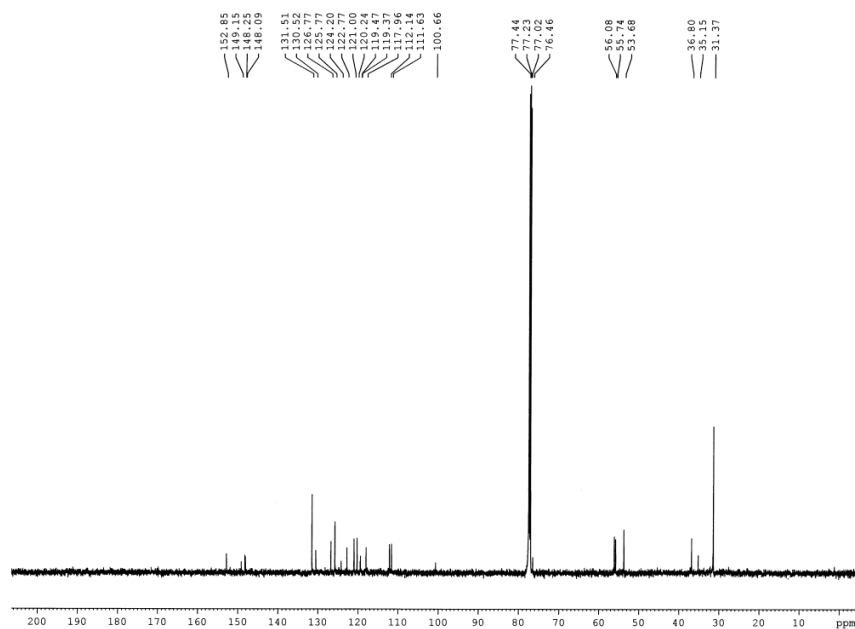
Current Data Parameters
NAME AU-IZ-SP-PTB_1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140626
Time 14.08
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 14
DS 2
SWH 12019.210 Hz
FIDRES 0.362798 Hz
AQ 1.3431488 sec
RG 80.22
MW 41.600 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 600.137063 MHz
NUC1 1H
P1 12.00 usec
PL1 21.00000000 W

F2 - Processing parameters
SI 16384
SF 600.137063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
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AU-IZ-SP-PTB_13C



```

Current Data Parameters
NAME AU-IZ-SP-PTB_13C
EXPNO 1
PROCNO 1

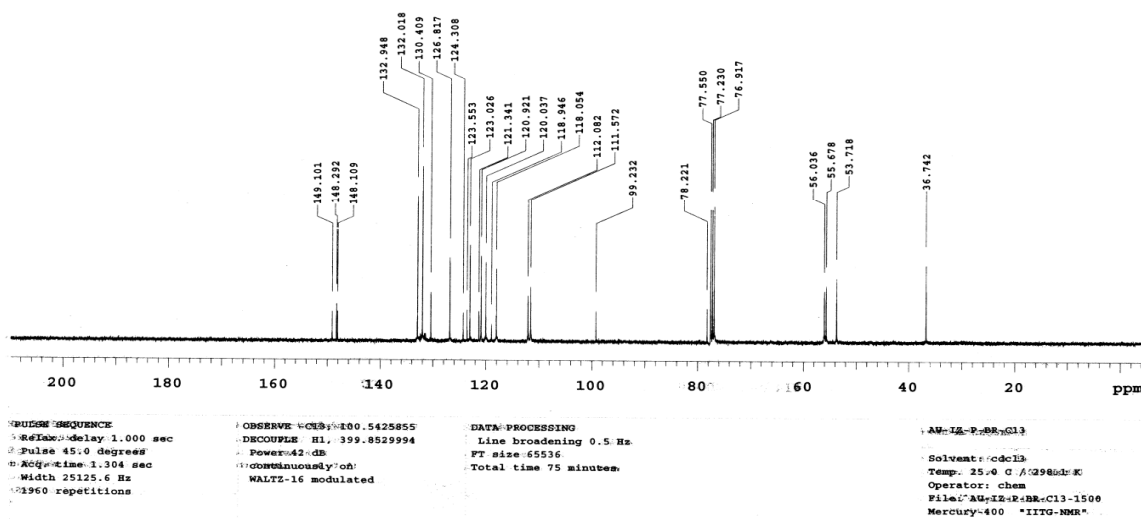
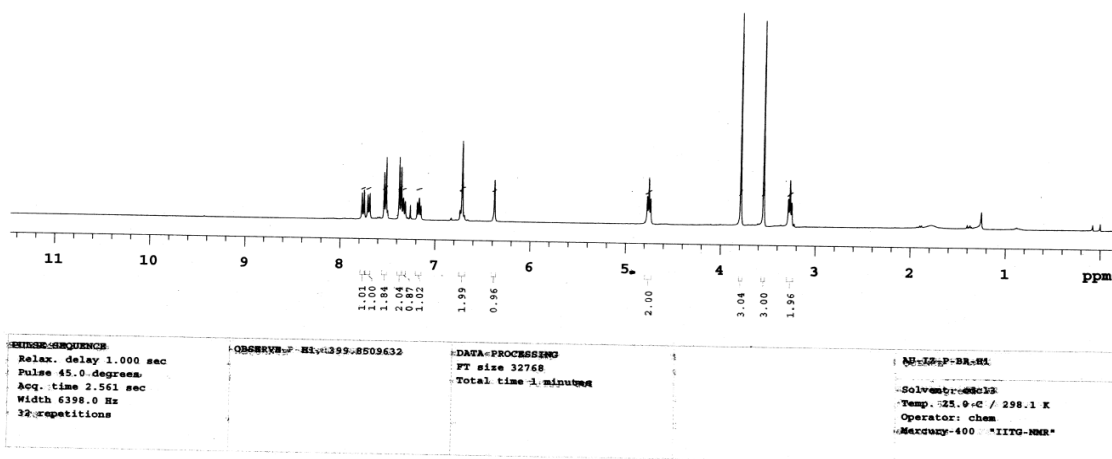
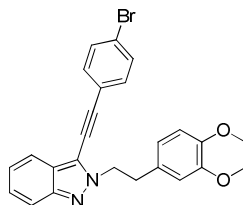
F2 - Acquisition Parameters
Date_ 20140626
Time 14.19
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 562
DS 2
SWH 36057.691 Hz
FIDRES 1.100593 Hz
AQ 0.4543829 sec
RG 65.24
MW 13.867 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
TDO 1

----- CHANNEL f1 -----
SFO1 150.927871 MHz
NUC1 13C
P1 10.50 usec
PL1 95.00000000 W

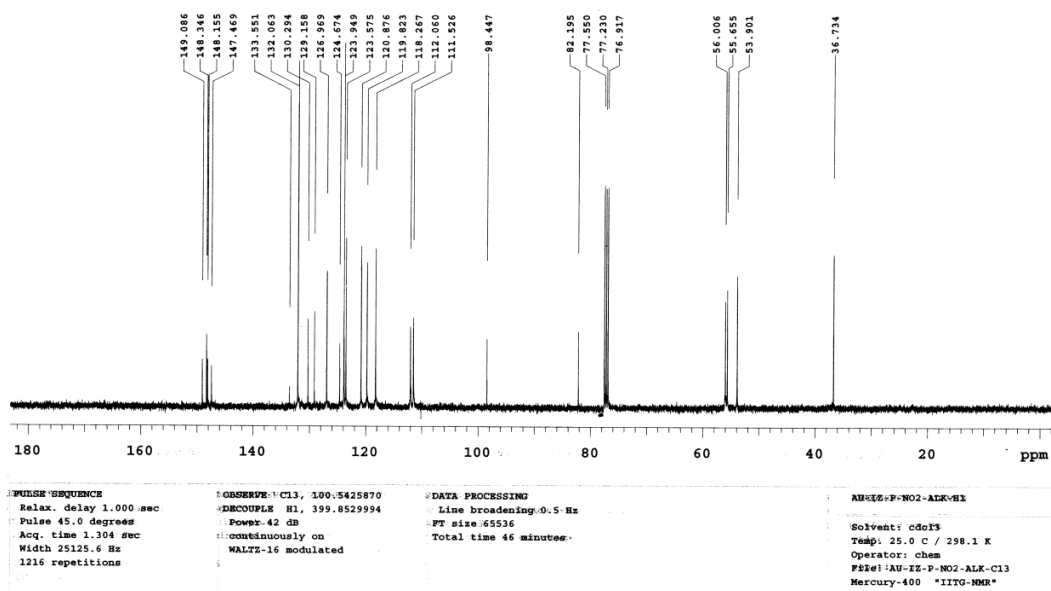
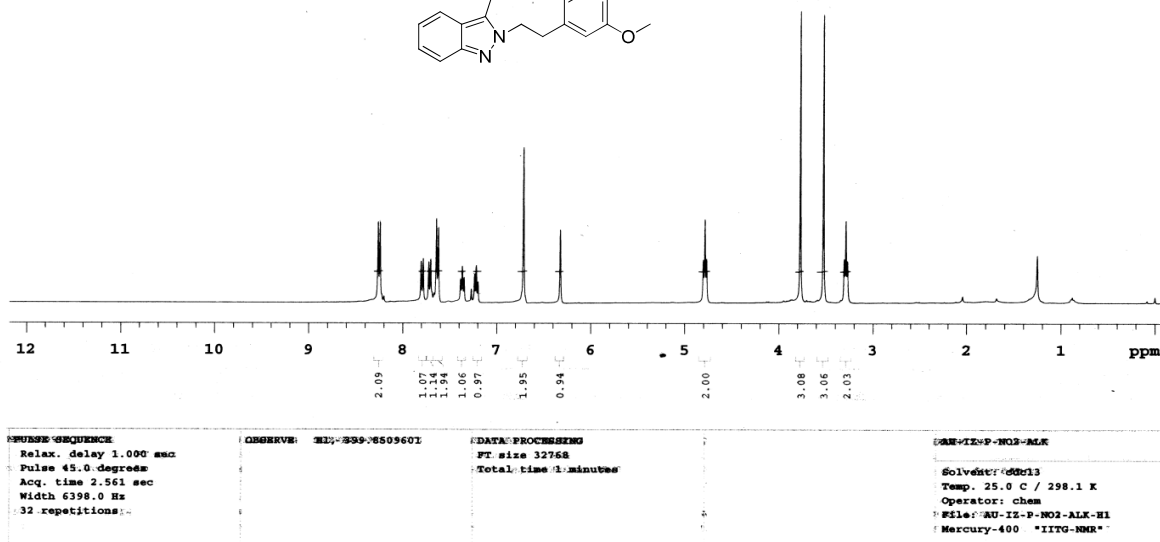
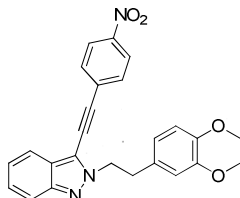
----- CHANNEL f2 -----
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 70.00 usec
PLM2 21.00000000 W
PLM12 0.61714000 W
PLM13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.918341 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
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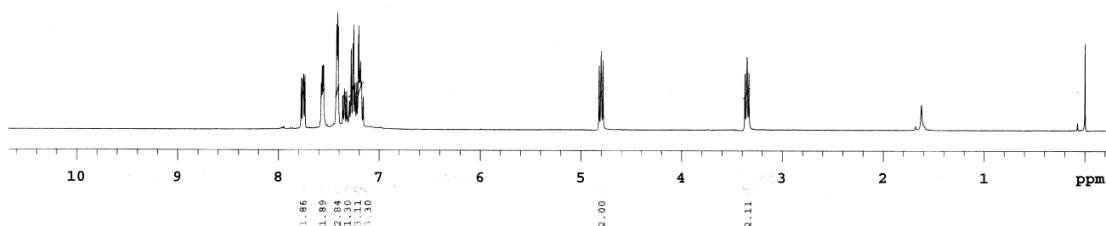
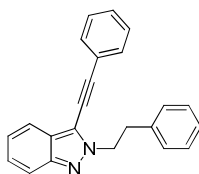

^1H and ^{13}C spectra of compound **4j**



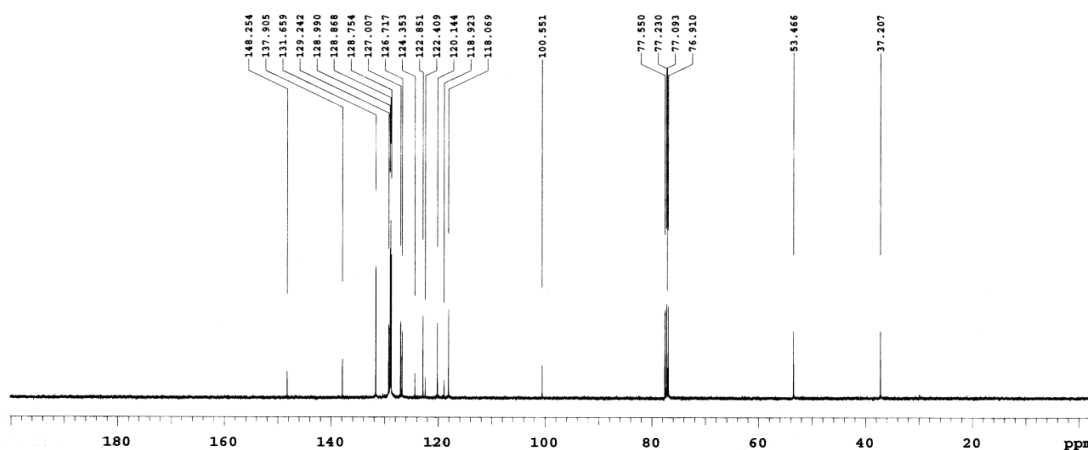
¹H and ¹³C spectra of compound 4k



^1H and ^{13}C spectra of compound **4I**

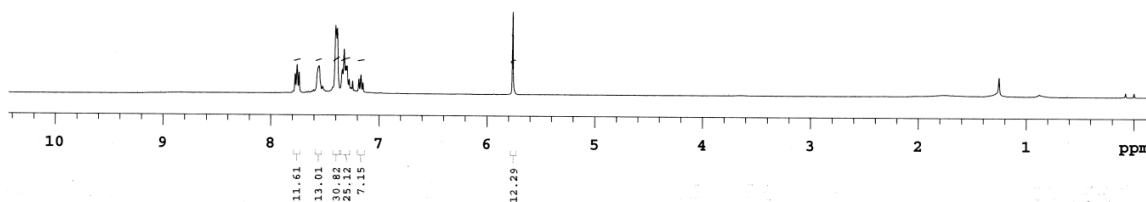
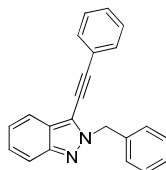


PULSE SEQUENCE	OBSERVE H1, 399.8509632	DATA PROCESSING	NAME si1p1h1
Relax. delay 1.000 sec		FT size 32768	Solvent: cdcl3
Pulse 45.0 degree		Total time 1 minutes	Temp. 25.0 C / 298.1 K
Acq. time 2.561 sec			Operator: chem
Width 6398.0 Hz			File: au1si1p1h1
32 repetitions			Mercury-400 *IITG-NMR*

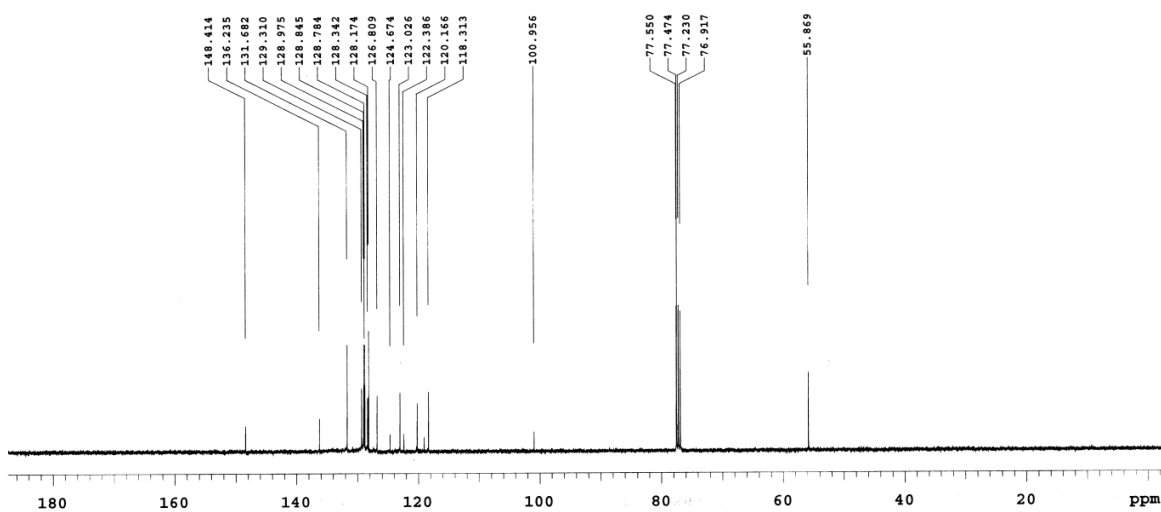


PULSE SEQUENCE	OBSERVE C13, 100.9425870	DATA PROCESSING	NAME SI1p1h1_13C-1
Relax. delay 1.000 sec	DECOUPLE H1, 399.8529994	Line broadening 0.5 Hz	Solvent: cdcl3
Pulse 45.0 degree	Power 42 dB	FT size 65536	Temp. 25.0 C / 298.1 K
Acq. time 1.204 sec	continuously on	Total time 31 minutes	Operator: chem
Width 25125.6 Hz	WALTZ-16 modulated		File: au-SI1p1h1-13C-1
820 repetitions			Mercury-400 *IITG-NMR*

¹H and ¹³C spectra of compound 4m

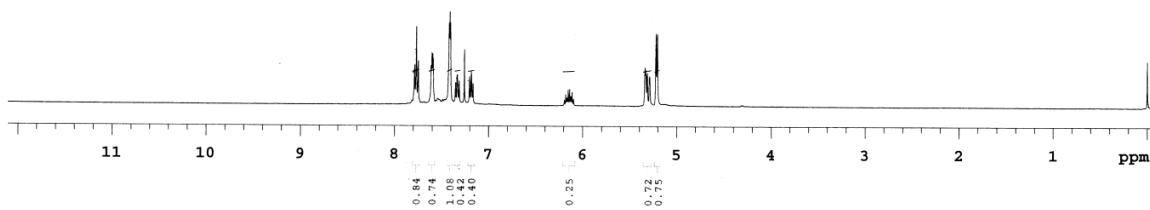
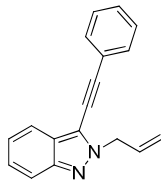


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509675	DATA PROCESSING FT size 32768 Total time 1 minutes	AU-IZ2BNPH-H1 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "IITG-NMR"
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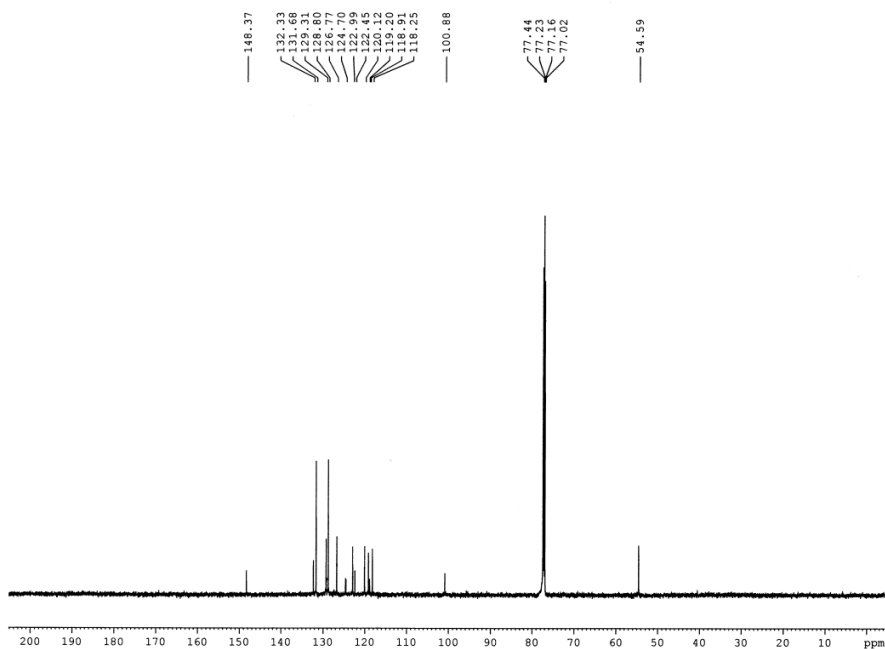
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 25125.6 Hz 1130 repetitions	OBSERVE C13, 100.5425840 DECOUPLE H1, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 43 minutes	AU-IZ-s-bn-c13 Solvent: Cdc13 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IZ-s-bn-c13 Mercury-400 "IITG-NMR"
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^1H and ^{13}C spectra of compound **4n**



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE ^1H , 399.8509636	DATA PROCESSING FT size 32768 Total time 1 minutes	AU-IZ-AL' Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IZ-AL Mercury-400 *IITG-NMR*
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IZ-AL_13C



```

Current Data Parameters
NAME      AU-IZ-AL_13C
EXPNO    1
PROCNO   1

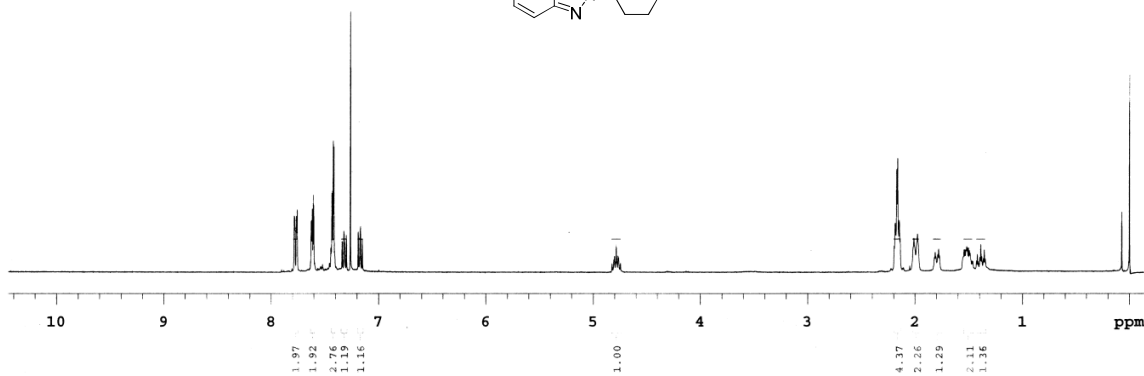
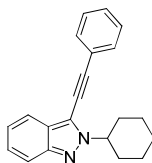
F2 - Acquisition Parameters
Date_    20140401
Time     16.00
INSTRUM spect
PROBHD   5 mm PABBO BB/
PULPROG zgpg30
TD       32768
SOLVENT  CDCl3
NS       716
DS       2
SWH      36057.691 Hz
FIDRES   1.100393 Hz
AQ       0.4543829 sec
RG       65.24
DW       13.867 usec
DE       6.50 usec
TE       298.2 K
D1       2.0000000 sec
D11      0.03000000 sec
TDO      1

----- CHANNEL f1 -----
SFO1    150.9279571 MHz
NUC1     13C
P1       10.50 usec
PLW1     95.0000000 W

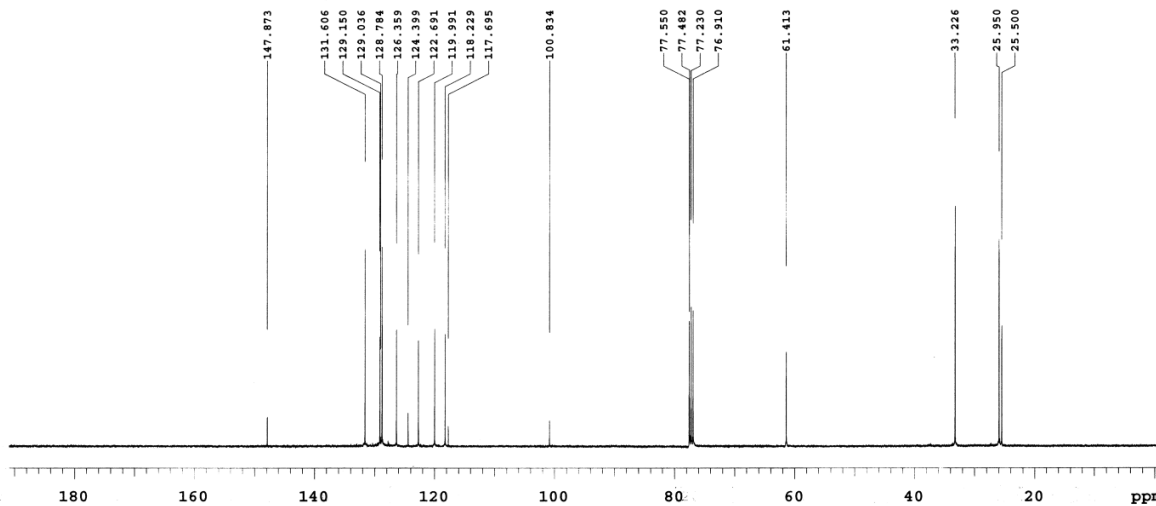
----- CHANNEL f2 -----
SFO2    600.1724007 MHz
NUC2     1H
CPDPRG2 waltz16
PCPD2   70.00 usec
PLW2    21.0000000 W
PLW12   0.61714000 W
PLW13   0.302299999 W

F2 - Processing parameters
SI       16384
SF       150.9128348 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

¹H and ¹³C spectra of compound **4o**



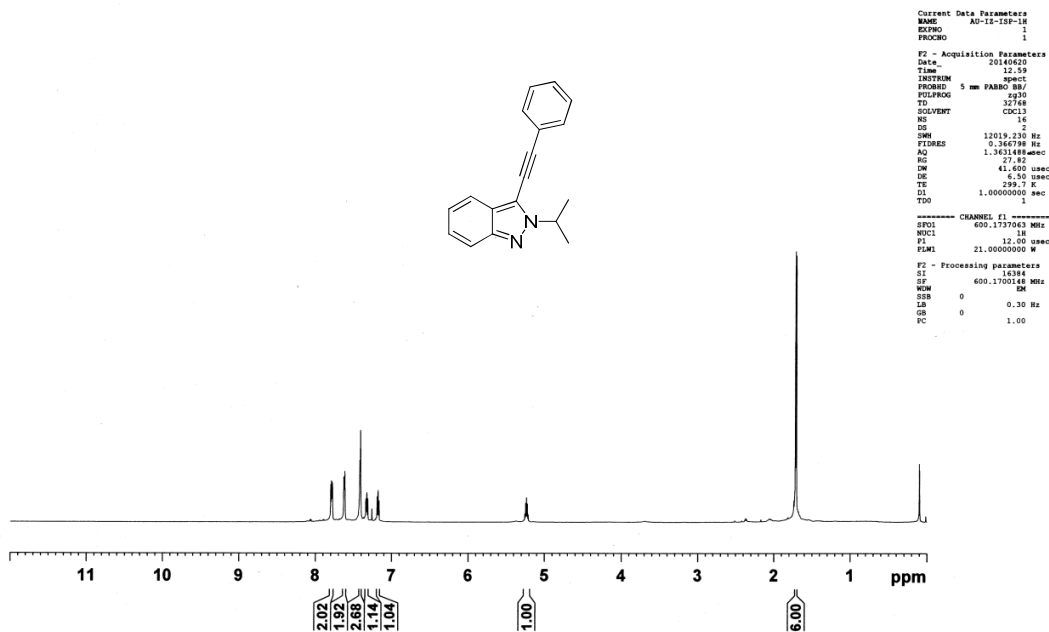
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509609	DATA PROCESSING FT size 32768 Total time 1 minutes	AU-IZCY-HENCE Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IZCY-H1 Mercury-400 *IITG-NMR*
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PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.304 sec Width 25125.6 Hz 5960 repetitions	OBSERVE C13, 100.5425840 DECOUPLE H1, 399.8529994 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 3.8 hours	AU-IZ-CYPH-13C Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AU-IZcypH-13C Mercury-400 *IITG-NMR*
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^1H and ^{13}C spectra of compound **4p**

AU-IZ-ISP-1H



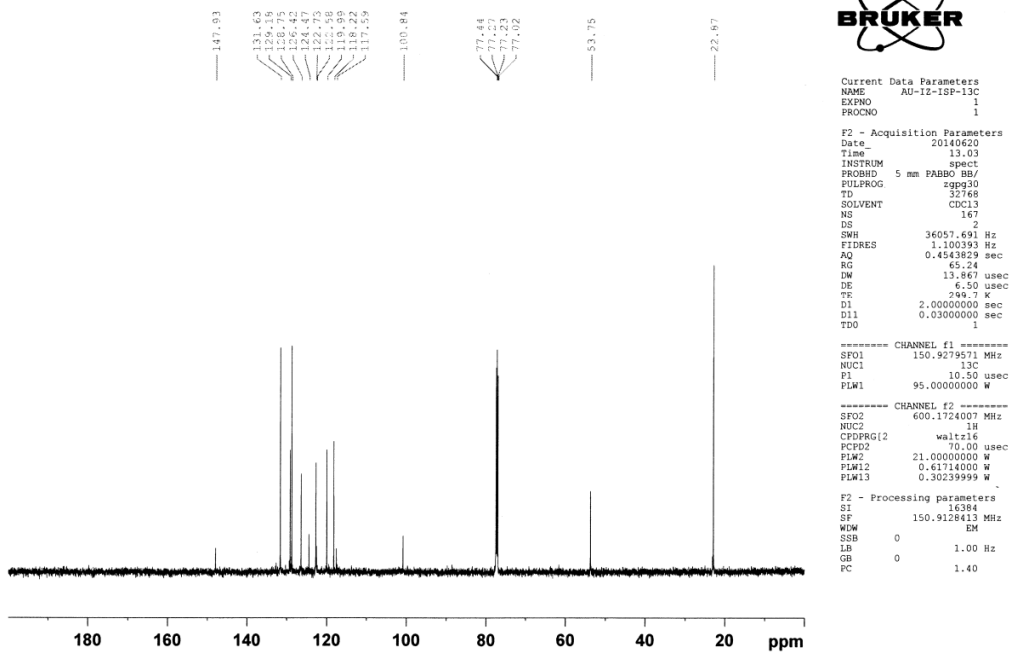
```
Current Data Parameters
NAME      AU-IZ-ISP-1H
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20140620
Time      12.59
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         2
SWH        12019.230 Hz
FIDRES     0.366798 Hz
AQ         1.3631489 sec
RG         27.82
DE         41.600 usec
TE         299.7 K
D1         1.00000000 sec
TD0        1

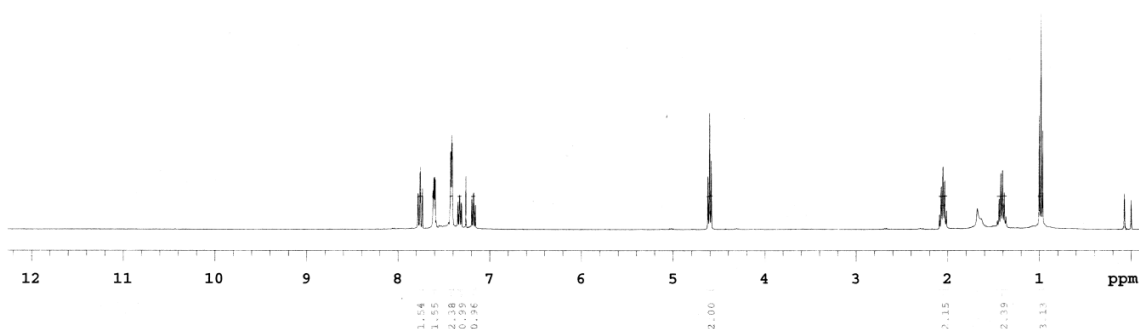
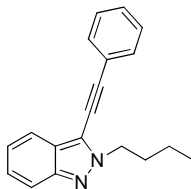
----- CHANNEL f1 -----
SFO1      600.1737943 MHz
NUC1       1H
P1         12.00 usec
PLM1      21.00000000 W

F2 - Processing parameters
SI         16384
SF         600.1700148 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

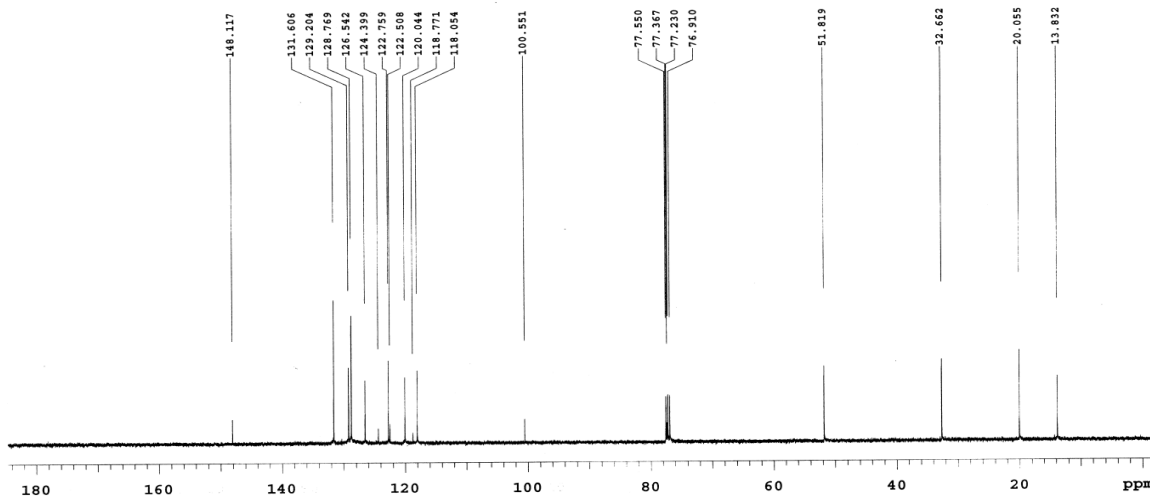
AU-IZ-ISP-13C



¹H and ¹³C spectra of compound 4q



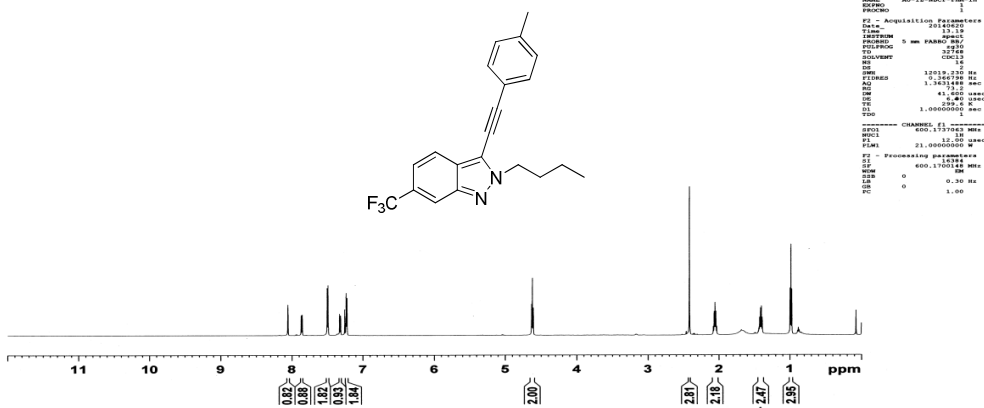
PULSE SEQUENCE	OBSERVE H1, 399.8509613	DATA PROCESSING	SU-2FU
Relax. delay 1.000 sec		FT size 16384	Solvent: cdc13
Pulse 45.0 degrees		Total time 1 minute	Temp. 25.0 C / 298.1 K
Acq. time 0.640 sec			Operator: chem
Width 10000.0 Hz			Mercury-400 "IITG-NMR"
32 repetitions			



PULSE SEQUENCE	OBSERVE GC13, 100.5425855	DATA PROCESSING	AUXIZPHNB-13C
Relax. delay 1.000 sec	DECOUPLE H1, 399.8529994	Line broadening 0.5 Hz	Solvent: cdc13
Pulse 45.0 degrees	Power: 42 dB	FT size 65536	Temp. 25.0 C / 298.1 K
Acq. time 1.304 sec	continuously on	Total time 26 minutes	Operator: chem
Width 25125.6 Hz	WALTZ-16 modulated		File: AU-13PHNB-13C
680 repetitions			Mercury-400 "IITG-NMR"

^1H , ^{13}C and ^{19}F spectra of compound **4r**

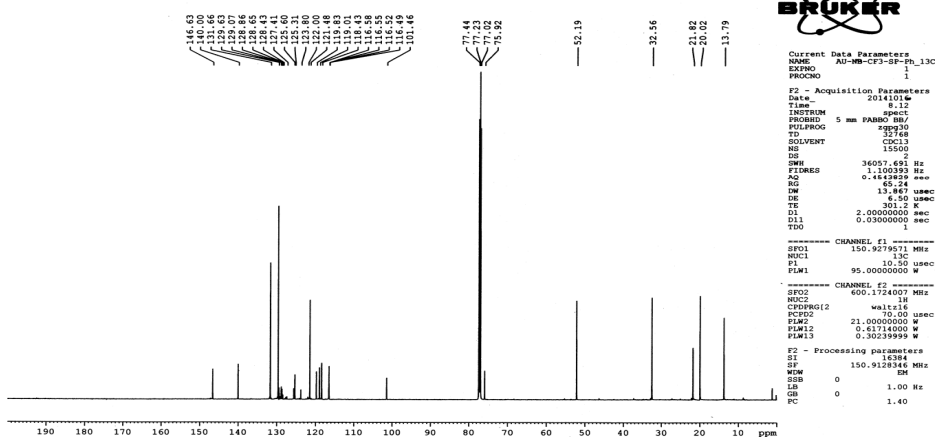
AU-IZ-NBCF-PHM-1H



```

Current Data Parameters
NAME AU-IZ-NBCF-PHM-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140101
Time 13:19
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1550
DS 2
SWH 36051.693 Hz
FIDRES 1.100383 Hz
AQ 0.4443229 sec
RG 327.64
DE 13.86 usec
TE 301.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 0.0000000 W
===== CHANNEL f2 =====
NUC2 1H
CPDPRG2 waltz16
PCPD2 21.0000000 W
PLM2 0.4371400 W
PLM3 0.30239999 W
F2 - Processing parameters
SI 6384
SF 150.912836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
    
```

AU-NB-CF3-SP-Ph_13C



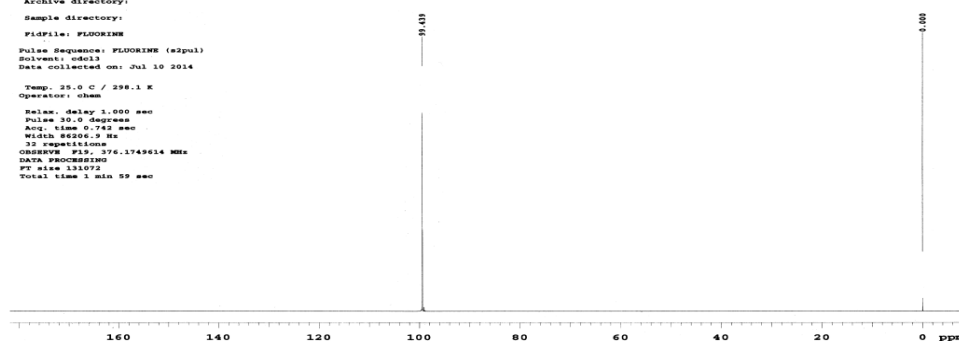
```

Current Data Parameters
NAME AU-NB-CF3-SP-Ph_13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140101
Time 8:12
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1550
DS 2
SWH 36051.693 Hz
FIDRES 1.100383 Hz
AQ 0.4443229 sec
RG 327.64
DE 13.86 usec
TE 301.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PL1 0.0000000 W
===== CHANNEL f2 =====
NUC2 1H
CPDPRG2 waltz16
PCPD2 21.0000000 W
PLM2 0.4371400 W
PLM3 0.30239999 W
F2 - Processing parameters
SI 6384
SF 150.912836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

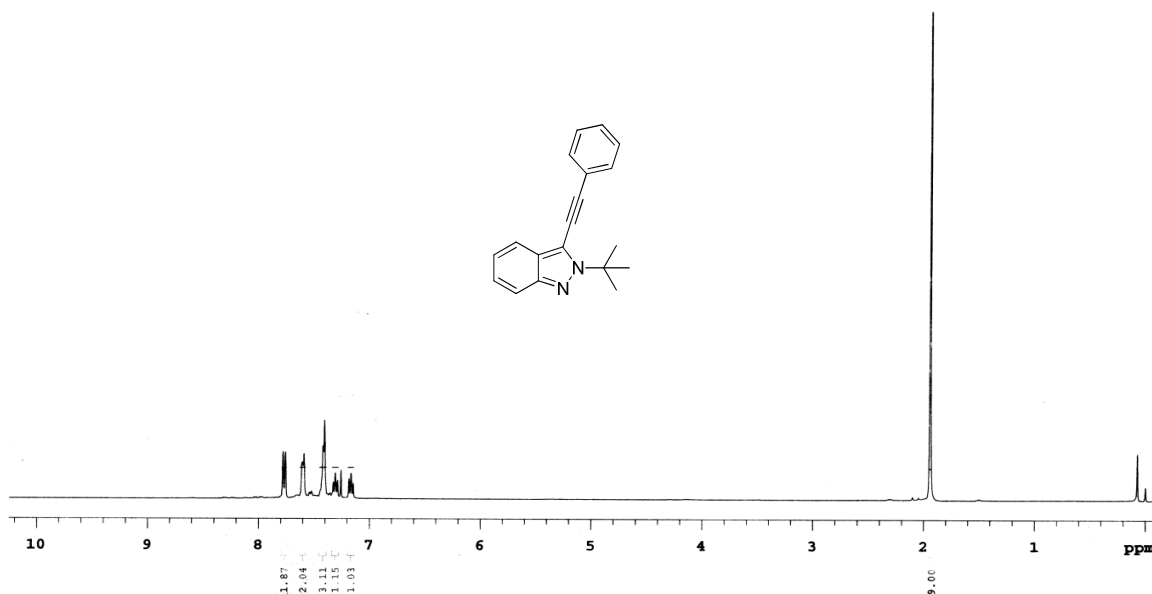
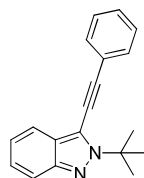
AU-IZ-NB-CF-PHMOR

```

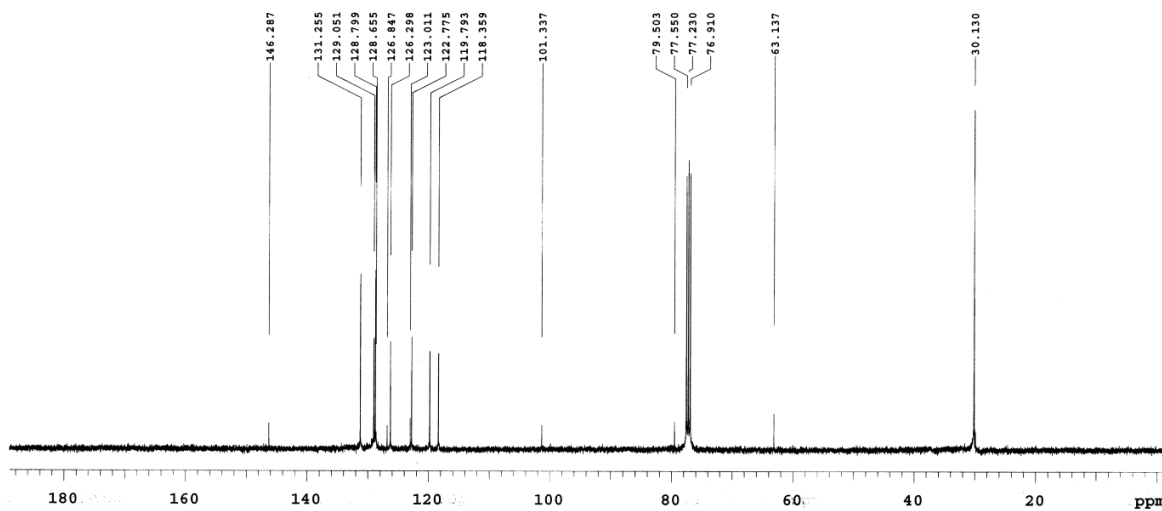
Sample Name:
Data Collected on:
1770 MHz spectscopy00
Archive directory:
Sample directory:
P1dfile: FLDORINE
Pulse Sequence: FLDORINE (s2pul)
Solvent: cdcl3
Data collected on: Jul 10 2014
Temp: 25.0 C / 298.1 K
Operator: oham
Relax. delay: 1.000 sec
Pulse 30.0 degree
Acq. time 0.742 sec
Width 86204.9 Hz
25 equillibrons
OBSERVE F1: 376.1749614 MHz
DATA PROCESSING
PT size 131072
Total time 1 min 59 sec
    
```



¹H and ¹³C spectra of compound 4s

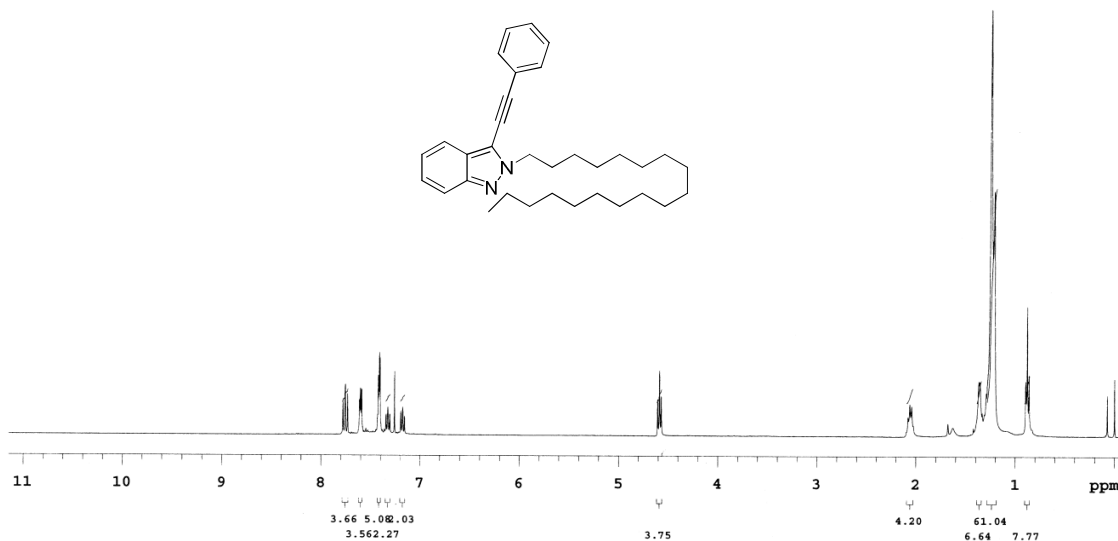
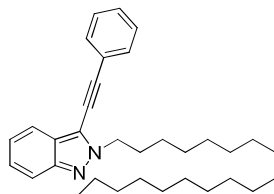


PULSE SEQUENCE Relax: delay 1.000 sec Pulse 45.0 degree# Acq. time 2.561 sec Width 6398.0 Hz 22 repetitions	OBSERVE ¹ H, 399.8529994 DECOUPLE H1, 399.8529994 Power: 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING FT size 32768 Total time 1 minutes	AU-IZCF-PHMR-13C Solvent: cdcl3 Temp: 25.0 C / 298.1 K Operator: chem File: AU-IZCF-PHMR-H11 Mercury-400 *IITG-NMR*
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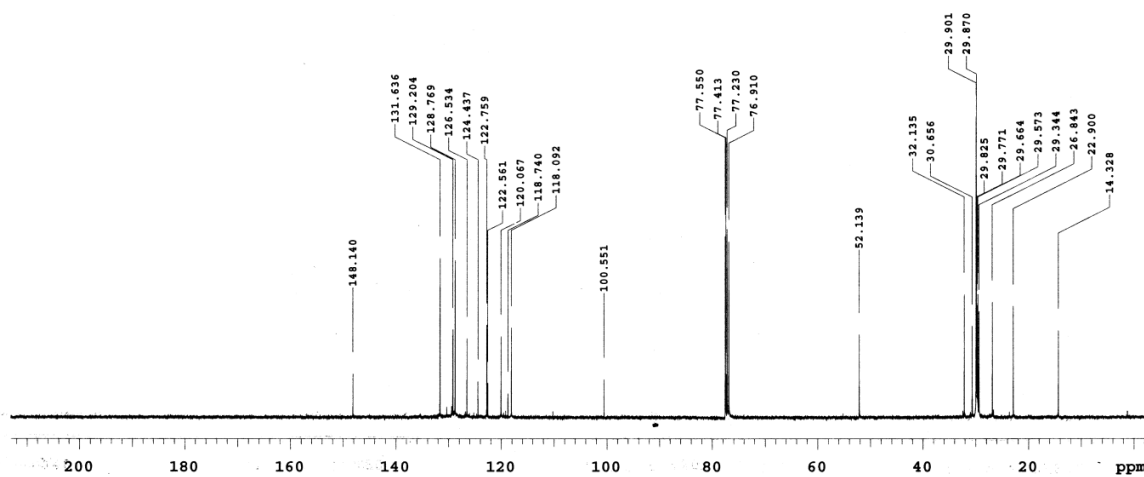


PULSE SEQUENCE Relax: delay 1.000 sec Pulse 45.0 degree# Acq. time 1.304 sec Width 25125.6 Hz 6040 repetitions	OBSERVE ¹³ C, 100.5425824 DECOUPLE H1, 399.8529994 Power: 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 3.9 hours	AU-IZ-TB-Ph_13C Solvent: cdcl3 Temp: 25.0 C / 298.1 K Operator: chem File: AU-IZ-TB-Ph_13C Mercury-400 *IITG-NMR*
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^1H and ^{13}C spectra of compound **4t**



PULSE SEQUENCE	OBSERVE: ^1H 399.8509625	DATA PROCESSING	AU-IZOCTPH-H1
Relax. delay 1.000 sec		FT size 32768	Solvent: cdcl3
Pulse 45.0 degrees		Total time 1 minutes	Temp: 25.0 C / 298.1 K
Acq. time 2.561 sec			Operator: chem
Width 6398.0 Hz			Mercury-400 "IITG-NMR"
32 repetitions			



PULSE SEQUENCE	OBSERVE: ^{13}C 100.5425832	DATA PROCESSING	AU-IZOCTPH-13C
Relax. delay 1.000 sec	DECOUPLE: ^1H 399.8529994	Line broadening 0.5 Hz	Solvent: cdcl3
Pulse 45.0 degrees	Power 42 dB	FT size 65536	Temp: 25.0 C / 298.1 K
Acq. time 1.304 sec	PROBHD: cpdpr131	Total time 86 minutes	Operator: chem
Width 25125.6 Hz	PROBHD: WALTZ-16 modulated		File: AU_17_06_08_13C
2280 repetitions			Mercury-400 "IITG-NMR"

The crystal parameters of compound **4a**

	4a- CCDC1008821
Formula	C ₂₅ H ₂₂ N ₂ O ₂
Formula weight	382.45
<i>T</i> /K	296(2)
Crystal system	Orthorhombic
Space group	P2(1)2(1)2(1)
<i>a</i> /Å	5.1346(2)
<i>b</i> /Å	15.0120(4)
<i>c</i> /Å	26.4598(7)
<i>α</i> ^o	90.00
<i>β</i> ^o	90.00
<i>γ</i> ^o	90.00
<i>V</i> /Å ³	2039.54(11)
<i>Z</i>	4
Abs. Coeff./mm ⁻¹	0.080
Abs. Correction	Multi-scan
GOF on <i>F</i> ²	1.299
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0335 <i>wR</i> 2 = 0.0707
<i>R</i> indices [all data]	<i>R</i> 1 = 0.0371 <i>wR</i> 2 = 0.0719

ORTEP diagram of **4a**

