Supporting Information

Metal-free, regioselective, visible light activation of 4CzIPN for the arylation of 2H-indazole derivatives

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Supporting Information

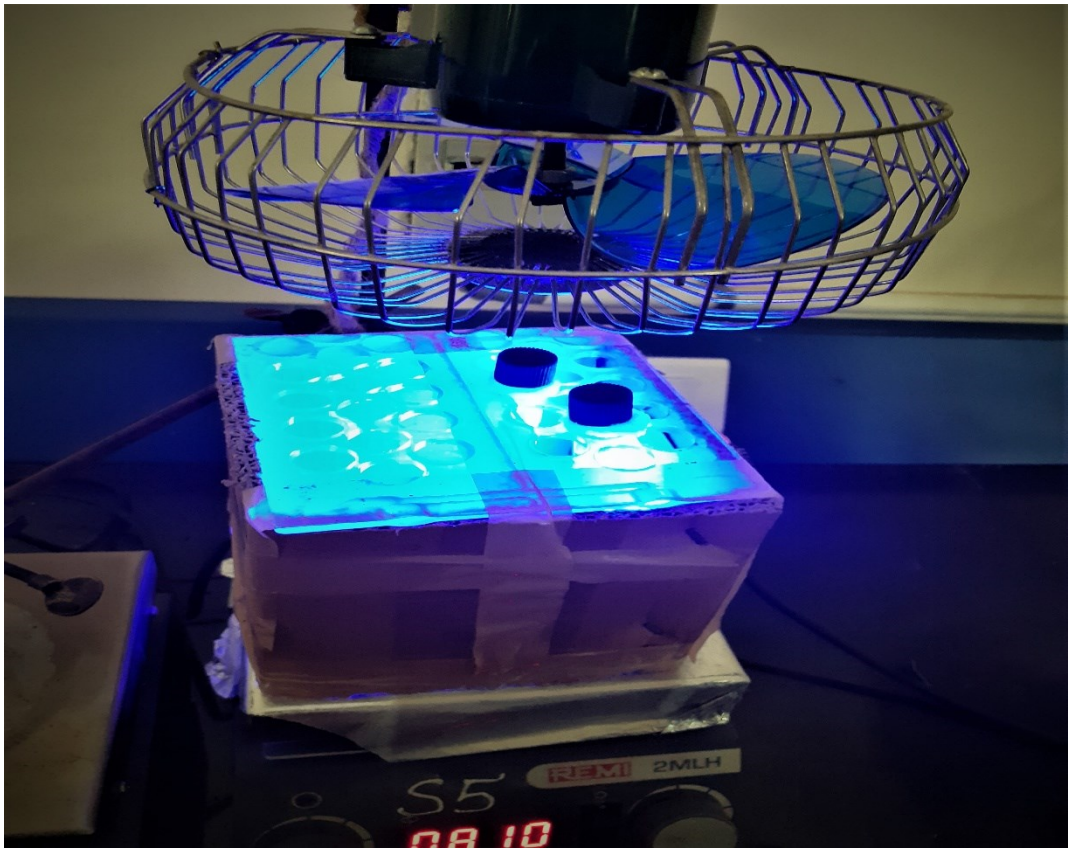
Contents

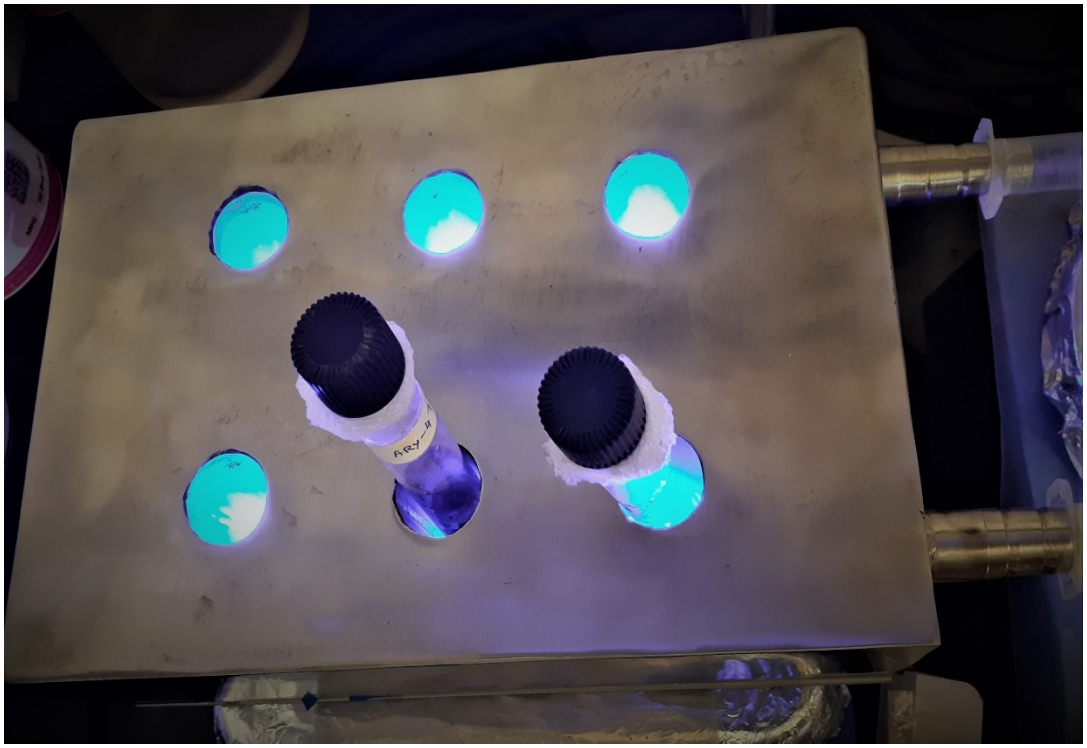
Materials and Methods

General. All reactions dealing with air and moisture-sensitive compounds were carried out in dry reaction vessels under a nitrogen atmosphere. Analytical thin-layer chromatography (TLC) was performed on Merck 60 F254 silica gel plates. ^{1}H nuclear magnetic resonance (NMR) spectra were recorded on Bruker & JEOL (400, 500 & 600 MHz) NMR spectrometers. ^{1}H NMR spectra are reported in parts per million (ppm) downfield from an internal standard tetramethylsilane. Melting points were determined using a capillary melting point apparatus and are uncorrected. Concealed round LED panel for ceiling light purchased from Century creative values Model no.: CL-333 7W blue LED, 6W Green LED and 24W blue LED purchased from sigma aldrich were used without using filters. UV-Vis. spectra were recorded on Evolution 201 thermo scientific spectrometer using cuvettes with 1 cm path length. Photoluminescence spectra were recorded on Ocean optics spectrometer. Borosilicate glass irradiation vessels were used. When the reaction was carried out, the distance from the light source to the irradiation vessel was 5.0 cm. Melting points were determined using a capillary melting point apparatus and are uncorrected.

Materials. Unless otherwise noted, materials were purchased from Avra, TCI, Merck, SRL, and other commercial suppliers and were used as received.

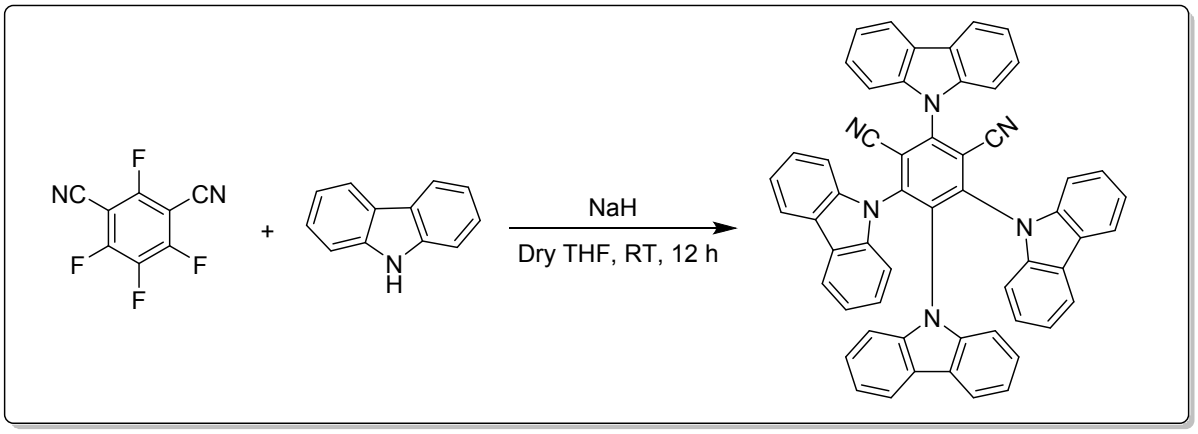
Photo-reactor setup





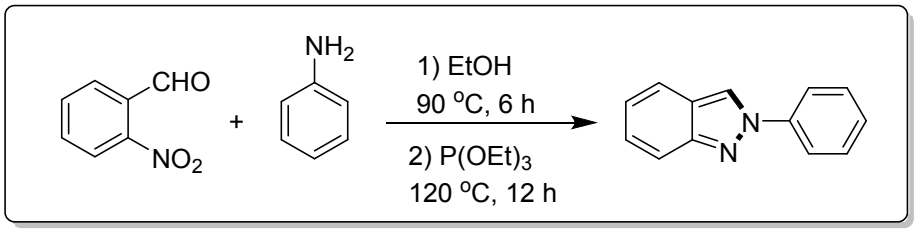
General procedures.

Synthesis of 2,4,5,6-tetra(9H-carbazol-9-yl)isophthalonitrile:



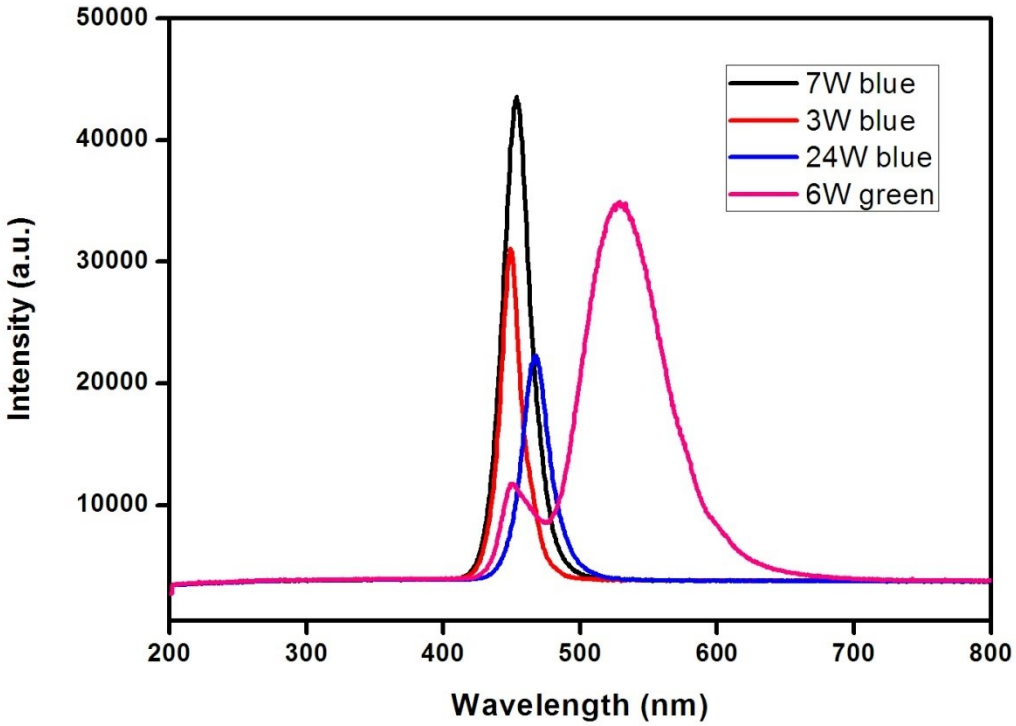
Sodium hydride (washed with hexane) 500 mg (12.5 mmol) 10equiv. pinch wise added to the solution of carbazole 1.05 g (6.25mmol) 5 equiv. in dry THF stirring for 30 minutes then 250 mg (1.25mmol) 1equiv. tetrafluoroisophthalonitrile was added to the mixture. After completion of the reaction the reaction mixture was quenched with water and filtered further purified by column chromatography got yellow solid 96% of yield.

Synthesis of 2-phenyl-2H-indazole:



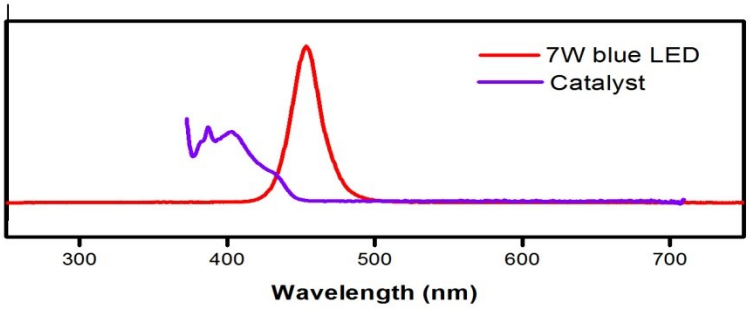
2-Nitrobenzaldehyde (1g, 10.74 mmol) aniline (1.62 g, 10.74 mmol) were dissolved in 10 ml of Ethanol. The reaction mixture was refluxed for 6 hours. After the completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature and the solvent removed by vacuum. Then, 10 equivalents of triethylphosphite was added to the crude product. After 12 h the product extract by water and with ethyl acetate dried. Excess of triethylphosphite was removed by excess of water extracted dried over Na_2SO_4 and evaporated under reduced pressure. The white solid thus obtained was further purified by column chromatography.

Photo Luminescence of different LEDs

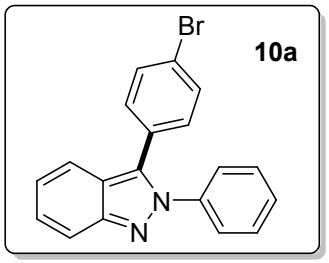


Comparison of 4CzIPN catalyst and 7Wblue LED

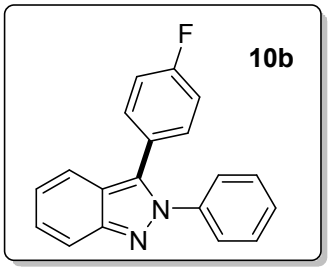
Absortion spectra of 4CzIPN 1M solution in CH_3CN.



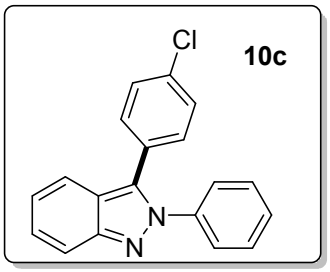
^{1}H NMR data of 2H-Indazoles:



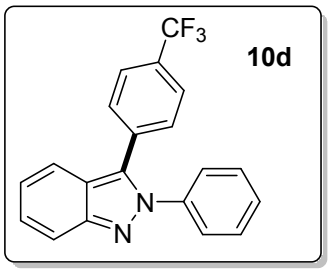
3-(4-bromophenyl)-2-phenyl-2H-indazole[1]: White solid, Yield = 80% (144 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 85 - 87 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 1H), 7.60 (d, *J* = 8.8 Hz, 1H), 7.45 (d, *J* = 7.6 Hz, 2H), 7.34-7.29 (m, 6H), 7.16 (t, *J* = 8.2 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H) ppm.



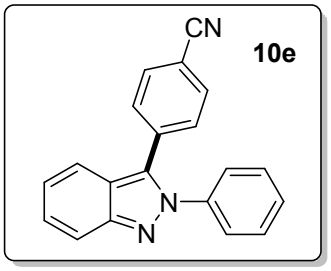
3-(4-fluorophenyl)-2-phenyl-2H-indazole[1]: White solid, Yield = 77% (201mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 91 - 93°C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 9.0 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.42-7.38 (m, 6H), 7.34-7.32 (m, 2H), 7.16-7.14 (m, 1H), 7.09 (t, *J* = 8.4 Hz, 2H) ppm.



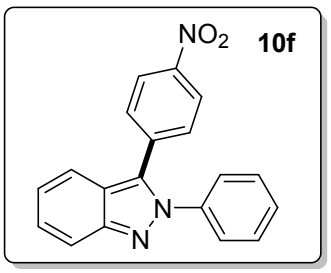
3-(4-chlorophenyl)-2-phenyl-2H-indazole[1]: White solid, Yield = 79% (188 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 117 - 119 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.42-7.37 (m, 8H), 7.30-7.26 (m, 2H), 7.16 (t, *J* = 7.3 Hz, 1H) ppm.



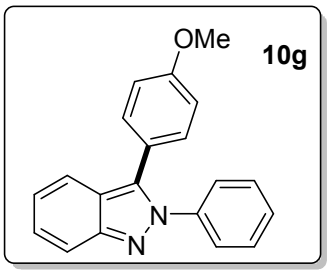
2-phenyl-3-(4-(trifluoromethyl)phenyl)-2H-indazole[4]: White solid, Yield = 78% (163 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 95 - 97 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.4 Hz, 1H), 7.69 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.41-7.37 (m, 6H), 7.20-7.17 (m, 1H) ppm.



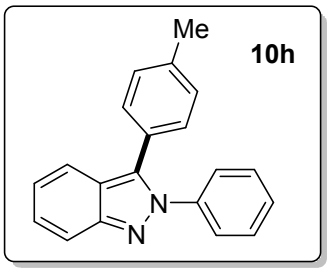
4-(2-phenyl-2H-indazol-3-yl)benzonitrile[2]: Pale yellow solid, Yield = 75% (188 mg), R_f = 0.3 (5% of Ethylacetate in Hexanes), m.p. = 128 - 130 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.4 Hz, 1H), 7.69 (t, *J* = 9.0 Hz, 3H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.43-7.38 (m, 6H), 7.22-7.19 (m, 1H) ppm.



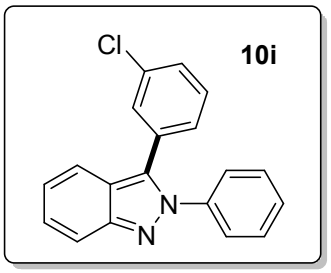
3-(4-nitrophenyl)-2-phenyl-2H-indazole[2]: Yellow solid, Yield = 77% (200 mg), R_f = 0.3 (5% of Ethylacetate in Hexanes), m.p. = 107 - 109°C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 9 Hz, 1H), 7.51 (d, *J* = 9 Hz, 2H) 7.44-7.41 (m, 6H), 7.25-7.21 (m, 1H) ppm.



3-(4-methoxyphenyl)-2-phenyl-2H-indazole[1]: White solid, Yield = 69% (168 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 88 - 90 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 7.9 Hz, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.46-7.44 (m, 2H), 7.41-7.34 (m, 3H), 7.29-7.26 (m, 3H), 7.13 (t, *J* = 7.0 Hz, 1H), 6.92 (d, *J* = 7.3 Hz, 2H), 3.84 (s, 3H) ppm.

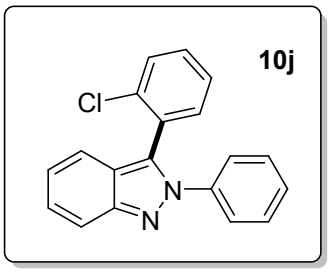


2-phenyl-3-(p-tolyl)-2H-indazole[1]: White solid, Yield = 60% (160 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 82 - 84 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.3 Hz, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.45-7.44 (m, 2H), 7.39-7.37 (m, 4H), 7.25 (d, *J* = 6.7 Hz, 2H), 7.21-7.19 (m, 2H), 7.15-7.11 (m, 1H), 2.39 (s, 3H) ppm.

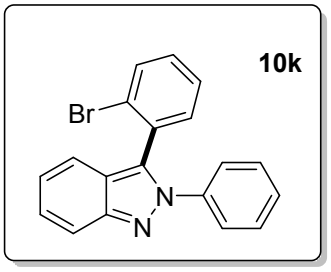


3-(3-chlorophenyl)-2-phenyl-2H-indazole[2]: White solid, Yield = 66% (159 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 59 - 61 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 6.9 Hz,

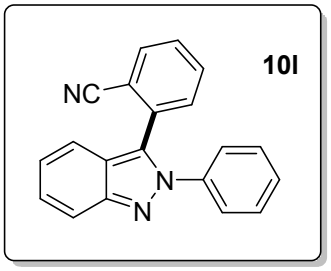
1H), 7.62 (d, *J* = 6.7 Hz, 1H), 7.36-7.18 (m, 9H), 7.11-7.08 (m, 2H) ppm.



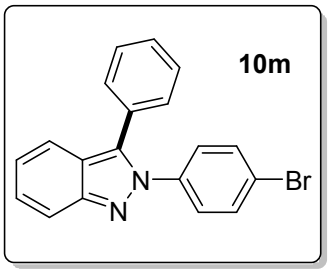
3-(2-chlorophenyl)-2-phenyl-2H-indazole[8]: White solid, Yield = 61% (146 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 51 - 53 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.0 Hz, 1H), 7.39 (t, *J* = 6.8 Hz, 2H), 7.34 (dd, *J* = 7.1, 2.8 Hz, 2H), 7.30-22 (m, 7H) 7.06-7.03 (m, 1H) ppm.



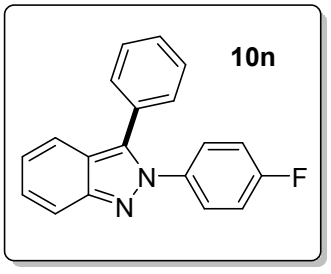
3-(2-bromophenyl)-2-phenyl-2H-indazole[8]: Pale yellow liquid, Yield = 61% (124 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.82 (t, *J* = 8.6 Hz, 1H), 7.77 (dd, *J* = 14, 8.0 Hz, 1H), 7.48-7.43 (m, 10H) 7.17-7.11 (m, 1H) ppm.



2-(2-phenyl-2H-indazol-3-yl)benzonitrile[3]: Pale Yellow solid, Yield = 66% (166 mg), R_f = 0.2 (5% of Ethylacetate in Hexanes), m.p. = 125 - 127 °C**; ^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.82 (d, *J* = 9.0 Hz, 1H), 7.67 (t, *J* = 9 Hz, 3H), 7.46 (d, *J* = 7.8 Hz, 2H), 7.43-7.38 (m, 6H), 7.21-7.19 (m, 1H) ppm.

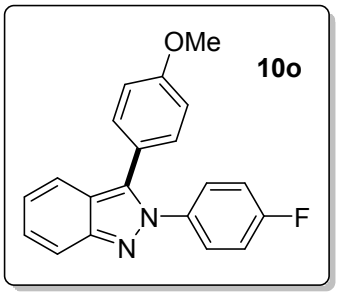


2-(4-bromophenyl)-3-phenyl-2H-indazole[3]: White solid, Yield = 42% (160 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 77 - 79 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (d, *J* = 9.0 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.43-7.34 (m, 10H), 7.15-7.12 (m, 1H) ppm.

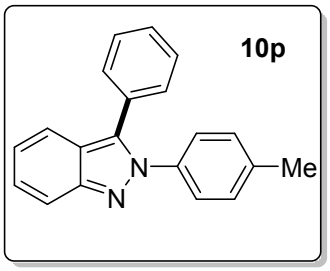


2-(4-fluorophenyl)-3-phenyl-2H-indazole[5]: White solid, Yield = 57% (176 mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. =

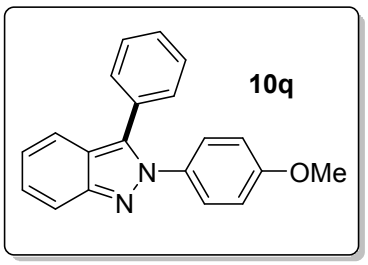
88 - 90 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.42-7.33 (m, 8H), 7.15-7.13 (m, 1H), 7.07 (t, *J* = 8.7 Hz, 3H) ppm.



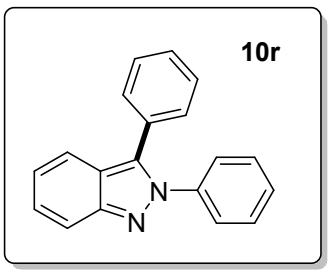
2-(4-fluorophenyl)-3-(4-methoxyphenyl)-2H-indazole[3]: White solid, Yield = 43% 115 mg), R_f = 0.4 (5% of Ethylacetate in Hexanes), m.p. = 90 - 92 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 9.0 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.41 (dd, *J* = 9.0, 4.4 Hz, 2H), 7.36-7.34 (m, 1H), 7.27-7.25 (m, 2H), 7.13-7.11 (m, 1H), 7.08 (t, *J* = 12.0 Hz, 2H), 6.92 (d, *J* = 9.0 Hz, 2H), 3.84 (s, 3H) ppm.



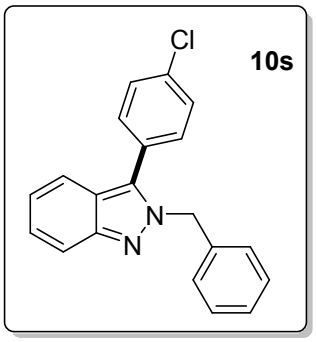
3-phenyl-2-(p-tolyl)-2H-indazole[3]: Semi solid, Yield = 78% (142 mg), R_f = 0.4 (5% of Ethylacetate in Hexanes), **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 9.0 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.39-7.34 (m, 6H), 7.31 (d , *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.14-7.12 (m, 1H), 2.38 (s, 3H) ppm.



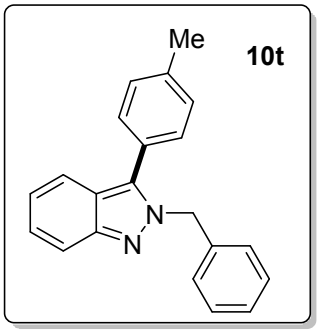
2-(4-methoxyphenyl)-3-phenyl-2H-indazole[3]: White solid, Yield=76% (242 mg), R_f = 0.4 (5% of Ethylacetate in Hexanes), m.p. = 77 - 79 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 9.0 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.38-7.33 (m, 8H), 7.13-7.11 (m, 1H), 6.88 (d, *J* = 9 Hz, 2H), 3.82 (s, 3H) ppm.



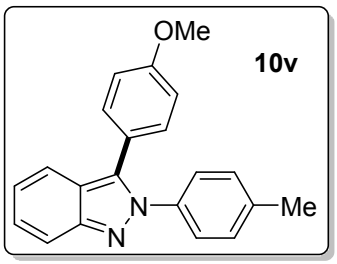
2,3-diphenyl-2H-indazole[1]: White solid, Yield = 68% (188mg), R_f = 0.5 (5% of Ethylacetate in Hexanes), m.p. = 75 - 77 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 9.0 Hz, 1H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.43 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.40-7.35 (m, 9H), 7.15-7.12 (m, 1H) ppm.



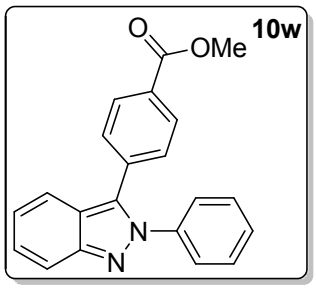
2-benzyl-3-(4-chlorophenyl)-2H-indazole[7]: Yellow solid, Yield = 30% (88 mg), R_f = 0.2 (5% of Ethylacetate in Hexanes), m.p. = 107 - 109 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 9.0 Hz, 1H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.33-7.32 (m, 1H), 7.28-7.24 (m, 3H), 7.11-7.08 (m, 1H), 7.07 (d, *J* = 6.6 Hz, 2H), 5.61 (s, 2H) ppm.



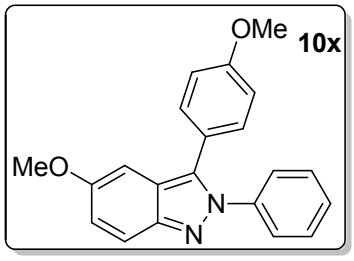
2-benzyl-3-(p-tolyl)-2H-indazole[7]: Yellow solid, Yield = 27% (84 mg), R_f = 0.2 (5% of Ethylacetate in Hexanes), m.p. = 67 - 69 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.73 (d, *J* = 9.0 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.33-7.26 (m, 7H), 7.25-7.24 (m, 1H), 7.08-7.06 (m, 1H), 5.62 (s, 2H), 2.43 (s, 3H) ppm.



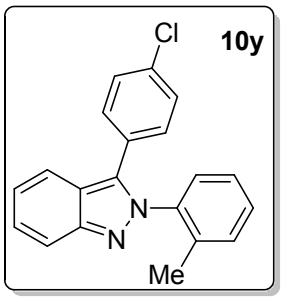
3-(4-methoxyphenyl)-2-(p-tolyl)-2H-indazole[2]: White solid, Yield = 63% 162 mg), R_f = 0.4 (5% of Ethylacetate in Hexanes), m.p. = 120 - 122 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.35-7.33 (m, 1H), 7.31(d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 9.0 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.12-7.09 (m, 1 H), 6.91 (d, *J* = 8.4 Hz, 2H), 3.83(s, 3H), 2.37 (s, 3H) ppm.



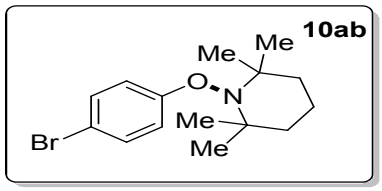
Methyl 4-(2-phenyl-2H-indazol-3-yl)benzoate[2]: White solid, Yield = 73%, R_f = 0.4 (5% of Ethylacetate in Hexanes), m.p. = 129 - 131 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 9 Hz, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.43-7.38 (m, 8H), 7.19-7.16 (m, 1 H), 3.92(s, 3H) ppm.



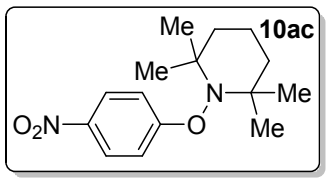
5-methoxy-3-(4-methoxyphenyl)-2-phenyl-2H-indazole: Pale yellow solid, Yield = 65%, R_f = 0.4 (5% of Ethylacetate in Hexanes), m.p. = 120 - 122 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 9.6 Hz, 1H), 7.40 (d, *J* = 7.2 Hz, 2H), 7.37-7.34 (m, 3H), 7.24 (s, 3H), 7.05 (dd, *J* = 9 & 2.4 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 2.4 Hz, 1H), 3.83(s, 3H), 3.81(s, 3H) ppm.



3-(4-chlorophenyl)-2-(o-tolyl)-2H-indazole[2]: Yellow viscous oil, R_f = 0.4 (5% of Ethylacetate in Hexanes); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.80-7.78 (m, 2H), 7.43-7.28 (m, 9H), 7.20-7.17 (m, 1H), 1.92(s, 3H) ppm.



1-(4-bromophenoxy)-2,2,6,6-tetramethylpiperidine[6]: White solid, Yield = 42% (83 mg), R_f = 0.8 (5% of Ethylacetate in Hexanes), m.p. = 73 - 75 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.27 (d, *J* = 9.0 Hz, 2H), 7.05 (d, *J* = 7.8 Hz, 2H), 1.56-1.55 (m, 5H), 1.41-1.38 (m, 1H), 1.2 (s, 6H), 0.97 (s, 6H) ppm.



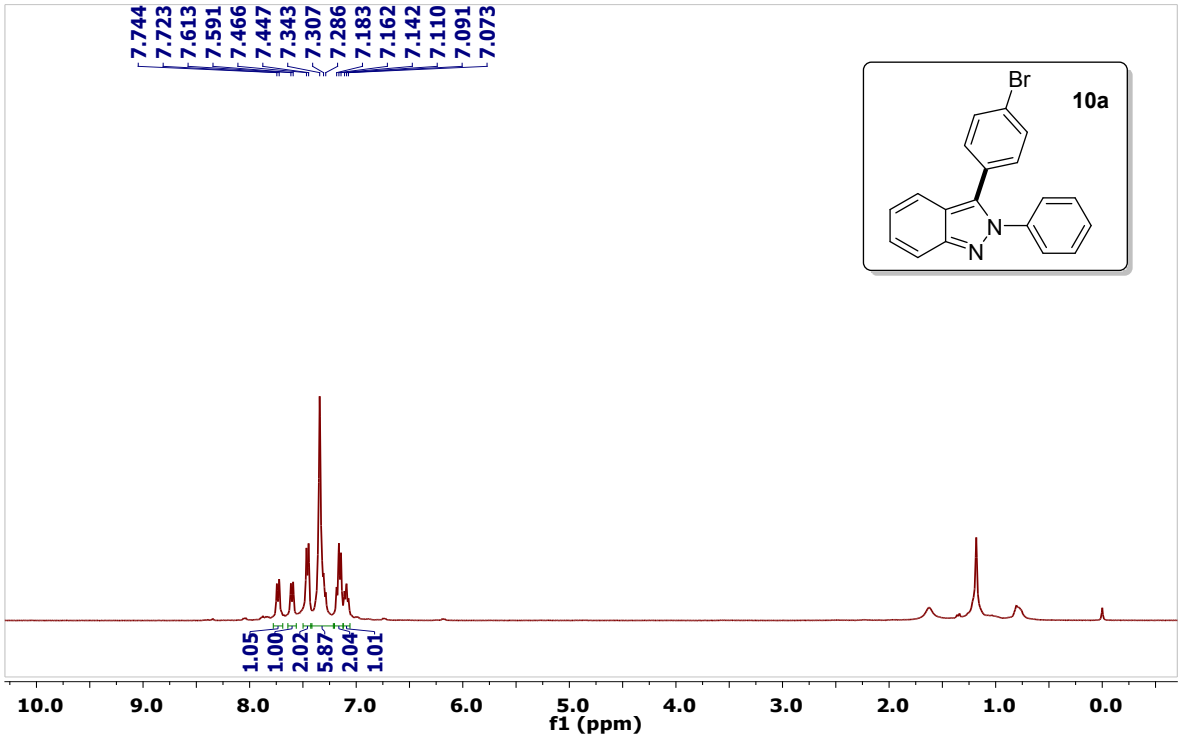
2,2,6,6-tetramethyl-1-(4-nitrophenoxy) piperidine[9]: Pale yellow semisolid; Yield = 44%, R_f = 0.5 (5% of Ethylacetate in Hexanes); ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm) 8.15 (2H, d, J = 9.3 Hz), 1.67-1.43 (m, 7H), 1.25 (s, 7H), 0.99 (s, 6H) ppm.

REFERENCE:

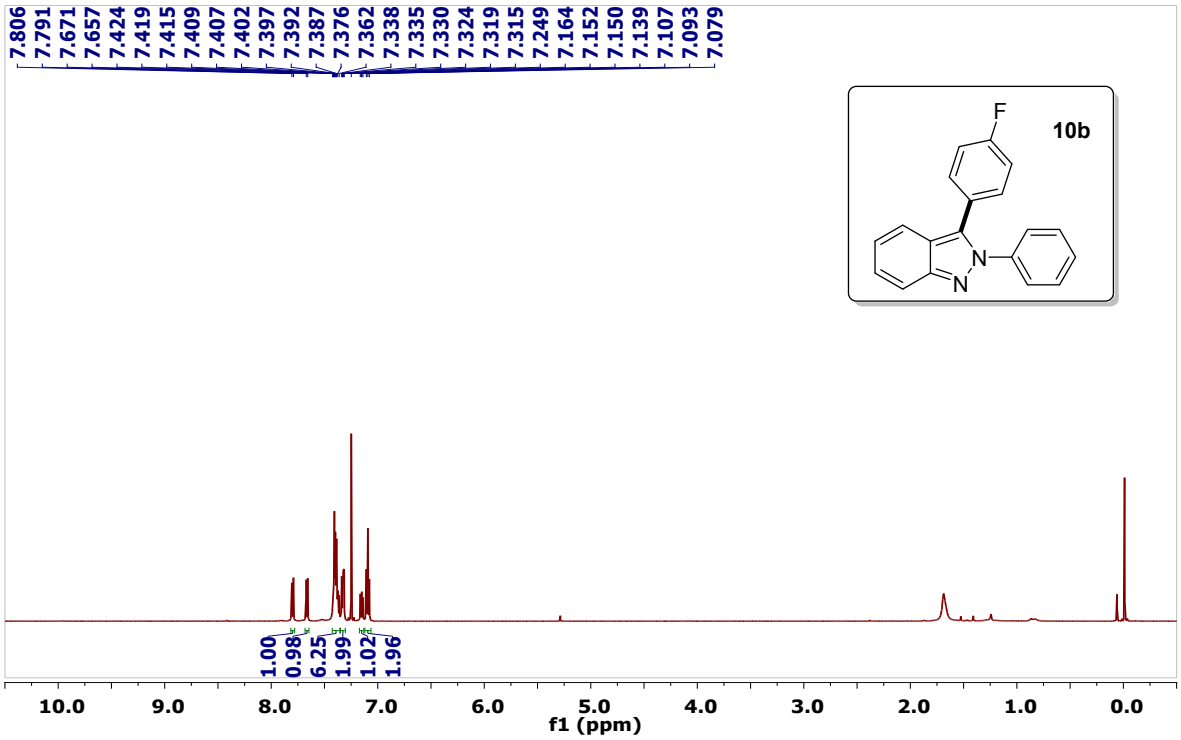
1. C. K. Christopher, A. J. Kimb, and A. Lee, *Org. Biomol. Chem*., 2019, **17**, 9698.
2. S. A. Ohnmacht, A. J. Culshaw, M. F. Greaney *Org. lett*. 2010, **12**, 224.
3. Zhen Long, Yudong Yang, and J. You *Org. Lett*. 2017, **19**, 2781.
4. X. Geng, C. Wang, *Org. let*. 2015, **17**, 2434.
5. S. G. Zhang, C. G. Liang and W. H. Zhang, *Molecules* 2018, **23**, 2783.
6. R. Saritha, B. Annes, S. Saravanan and S. Ramesh*, Org. Biomol. Chem.*, 2020, **18***,* 2510.
7. F. Belkessam, M. Aidene, J. F. Soulé and H. Doucet, *Chem. Cat. chem.,* 2016*,* **9,** 2239.
8. J. Y. Son, H. Kim, Y. Baek, K. Um, G. H. Ko, G. U. Han, P. H. Lee, *Adv. Synth. Catal*., 2018, **360**, 4354.
9. D. P. Hari, P. Schroll, B. König, *J. Am. Chem. Soc*. 2012, **134**, 2958–2961.

^{1}H NMR spectrum of 2H-Indazoles:

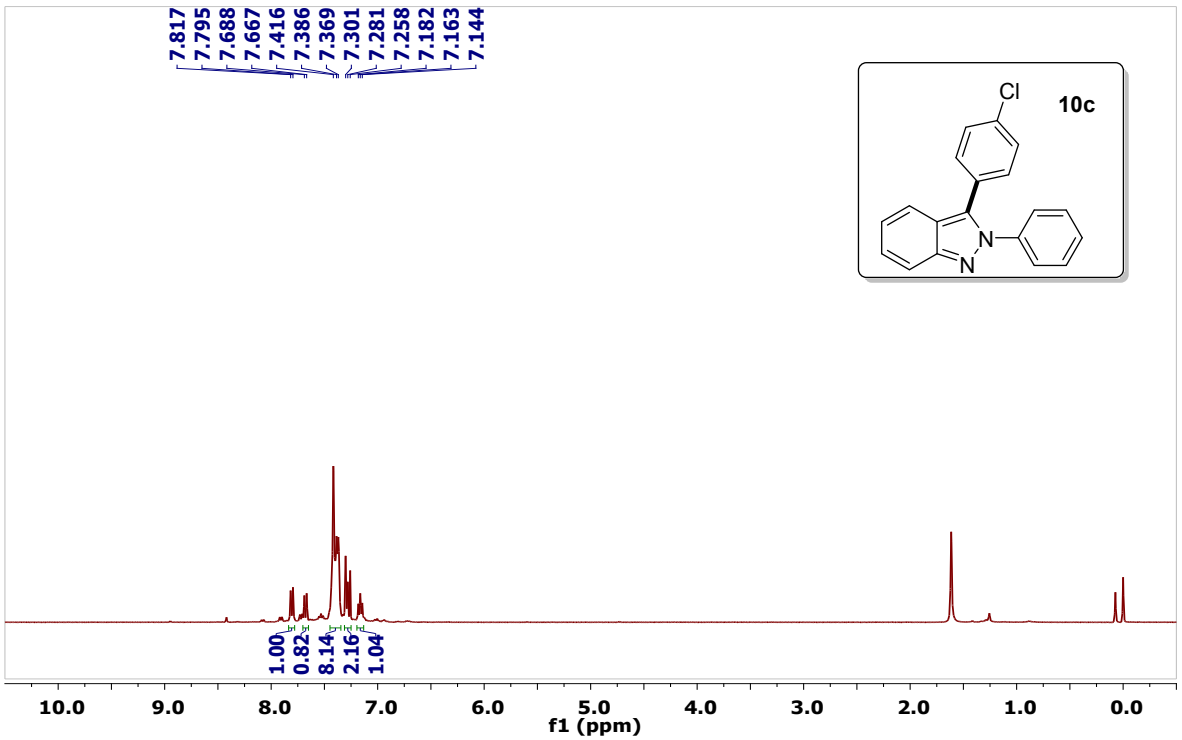
^{1}H NMR Spectrum of 3-(4-bromophenyl)-2-phenyl-2H-indazole



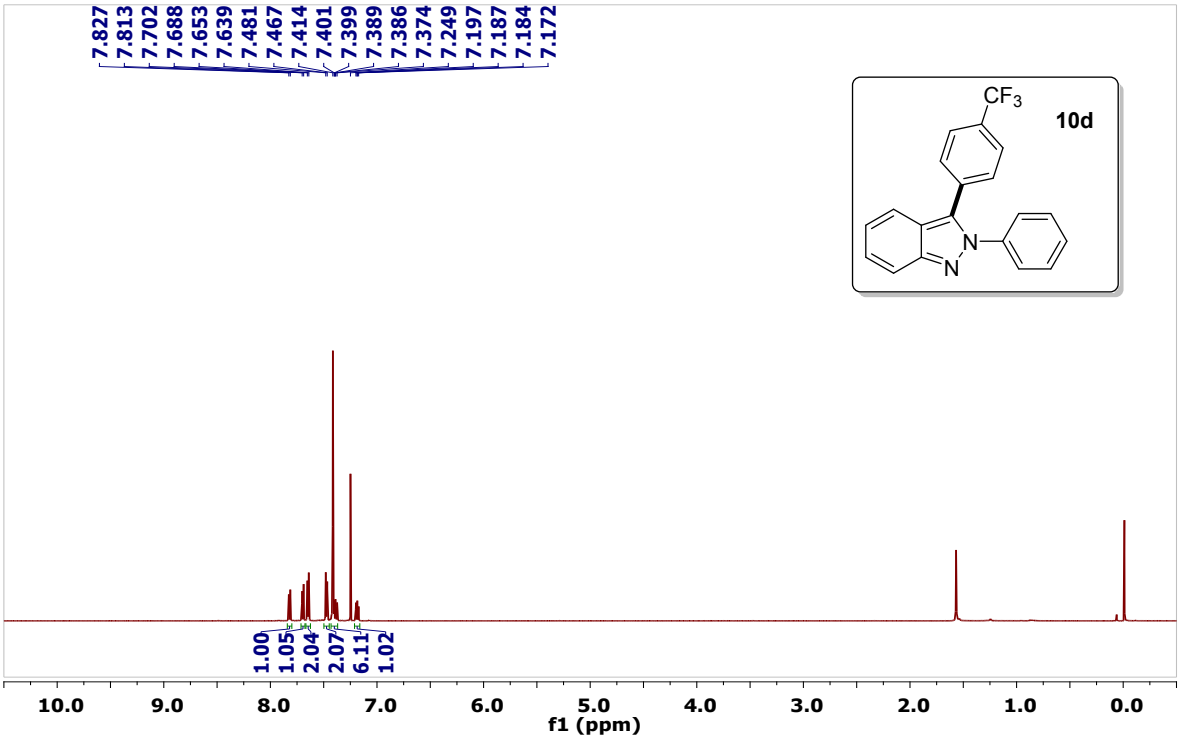
^{1}H NMR Spectrum of 3-(4-fluorophenyl)-2-phenyl-2H-indazole



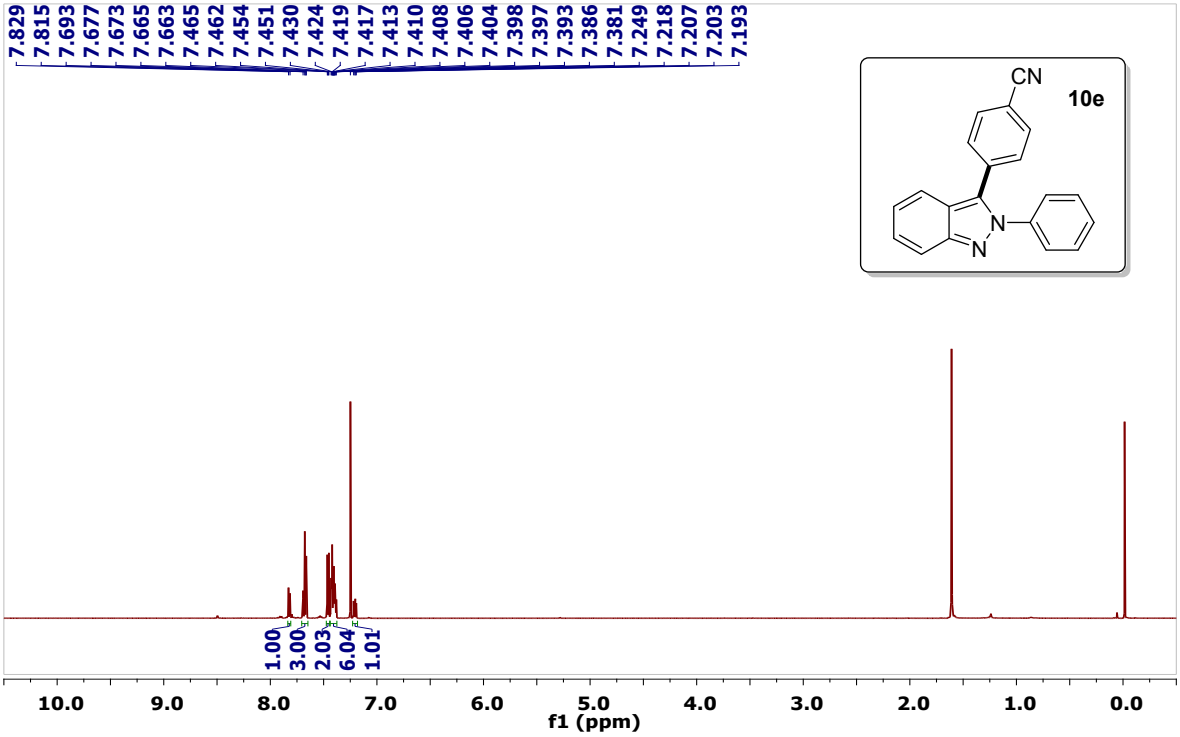
^{1}H NMR Spectrum of 3-(4-chlorophenyl)-2-phenyl-2H-indazole



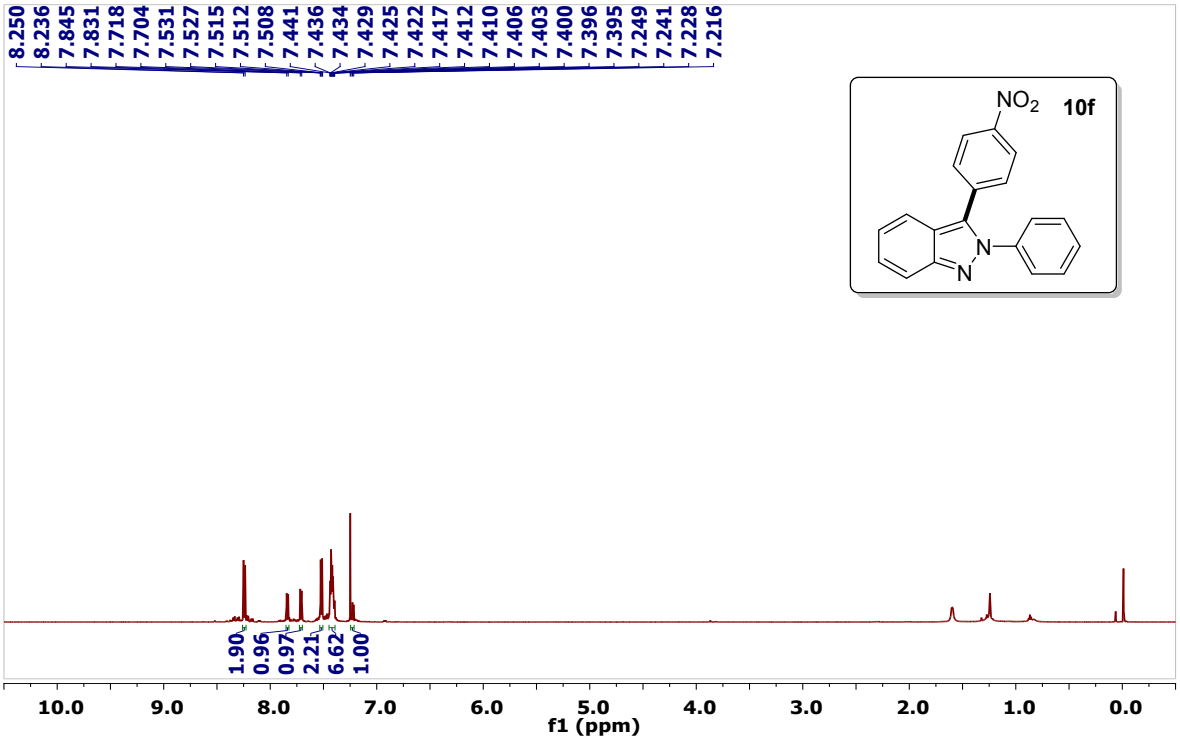
^{1}H NMR Spectrum of 2-phenyl-3-(4-(trifluoromethyl)phenyl)-2H-indazole



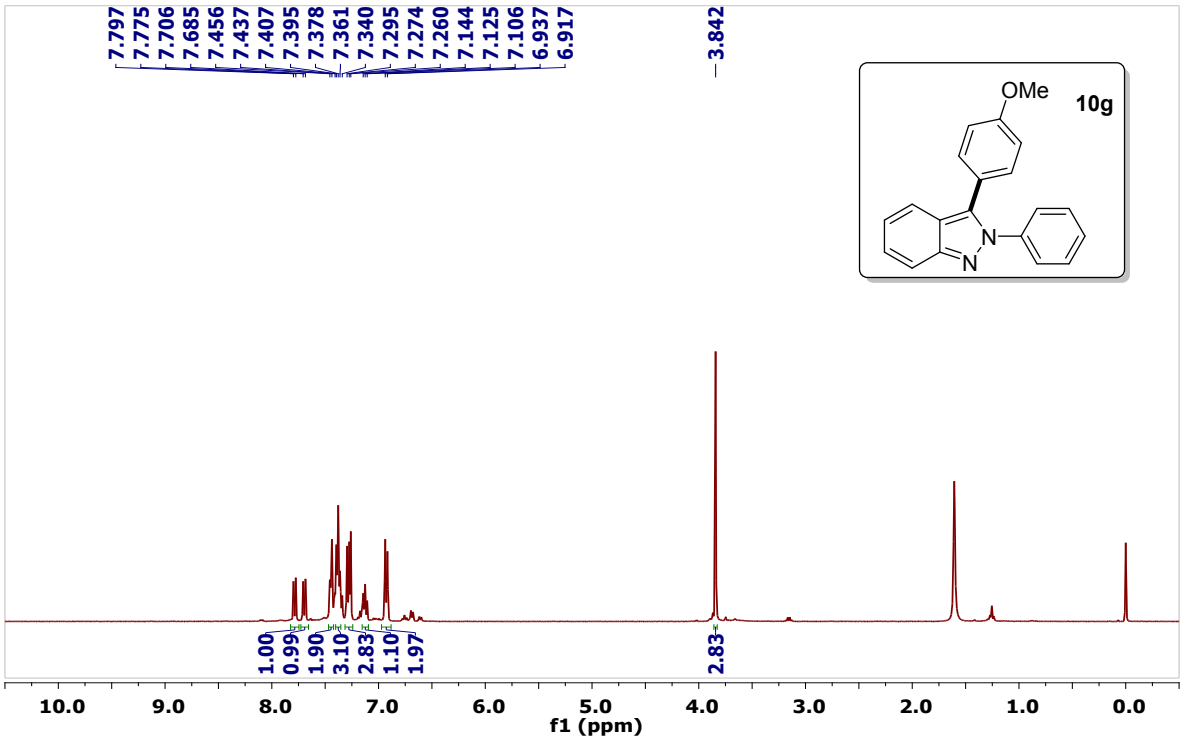
^{1}H NMR Spectrum of 4-(2-phenyl-2H-indazol-3-yl)benzonitrile



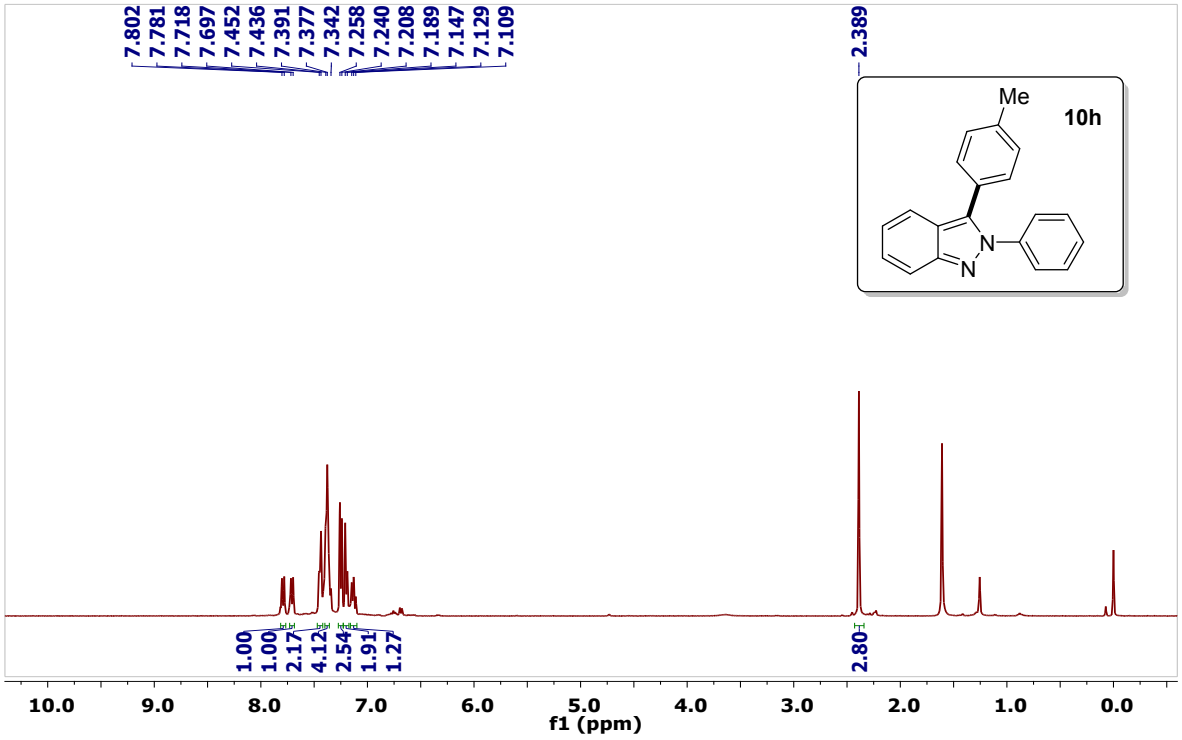
^{1}H NMR Spectrum of 3-(4-nitrophenyl)-2-phenyl-2H-indazole



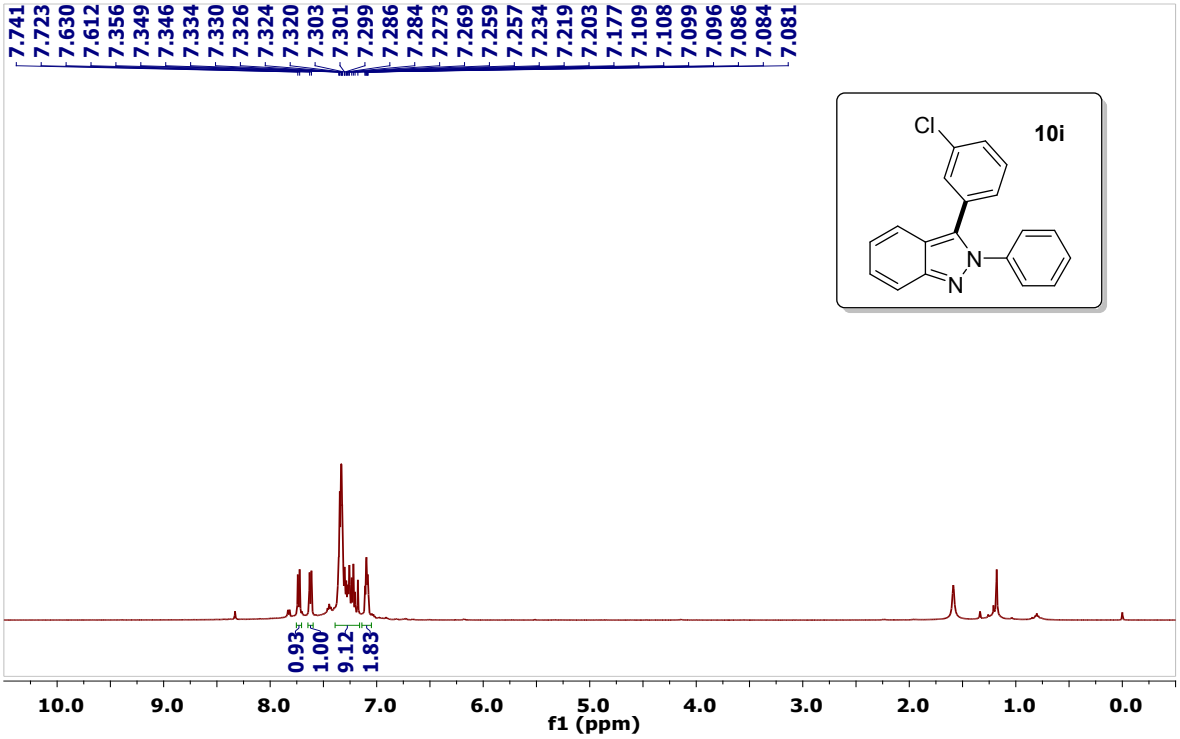
^{1}H NMR Spectrum of 3-(4-methoxyphenyl)-2-phenyl-2H-indazole



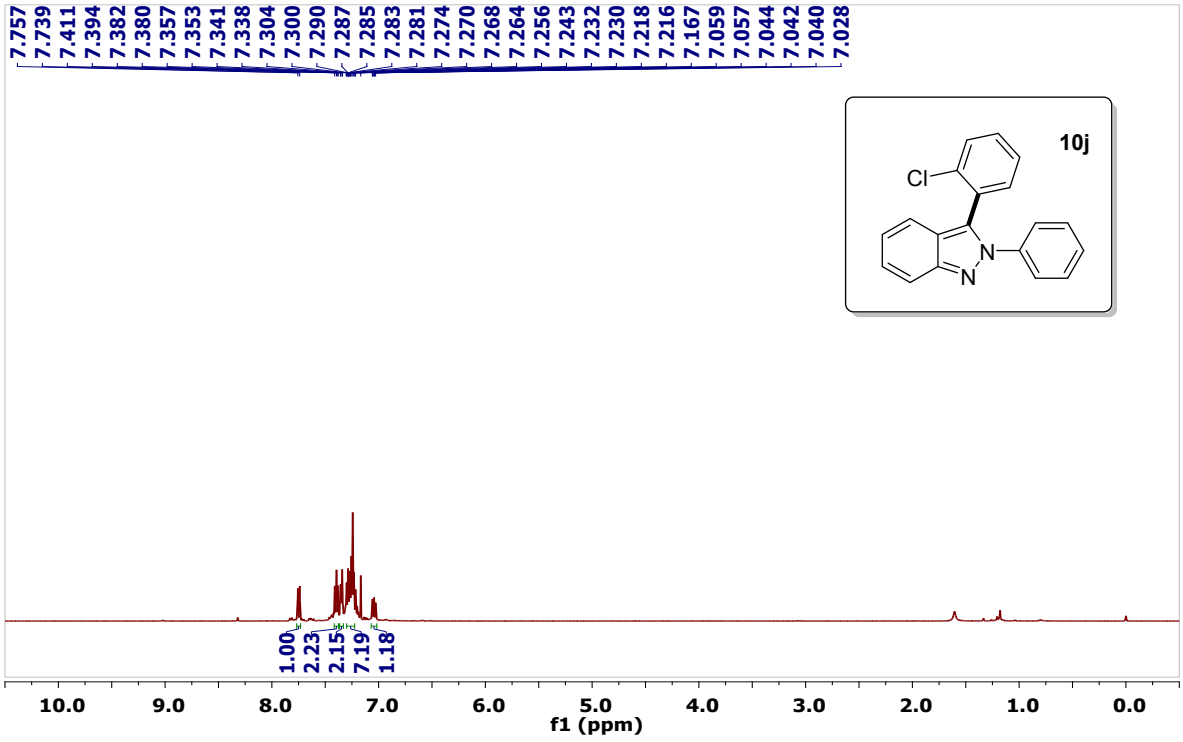
^{1}H NMR Spectrum of 2-phenyl-3-(p-tolyl)-2H-indazole



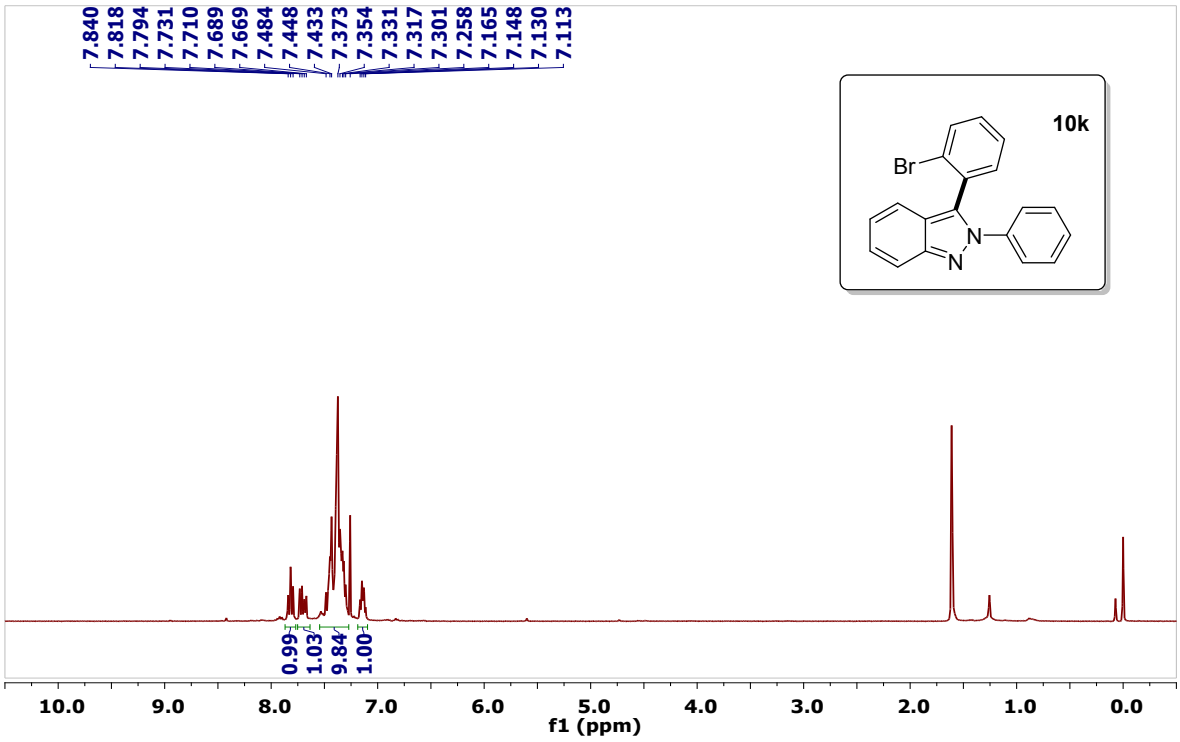
^{1}H NMR Spectrum of 3-(3-chlorophenyl)-2-phenyl-2H-indazole



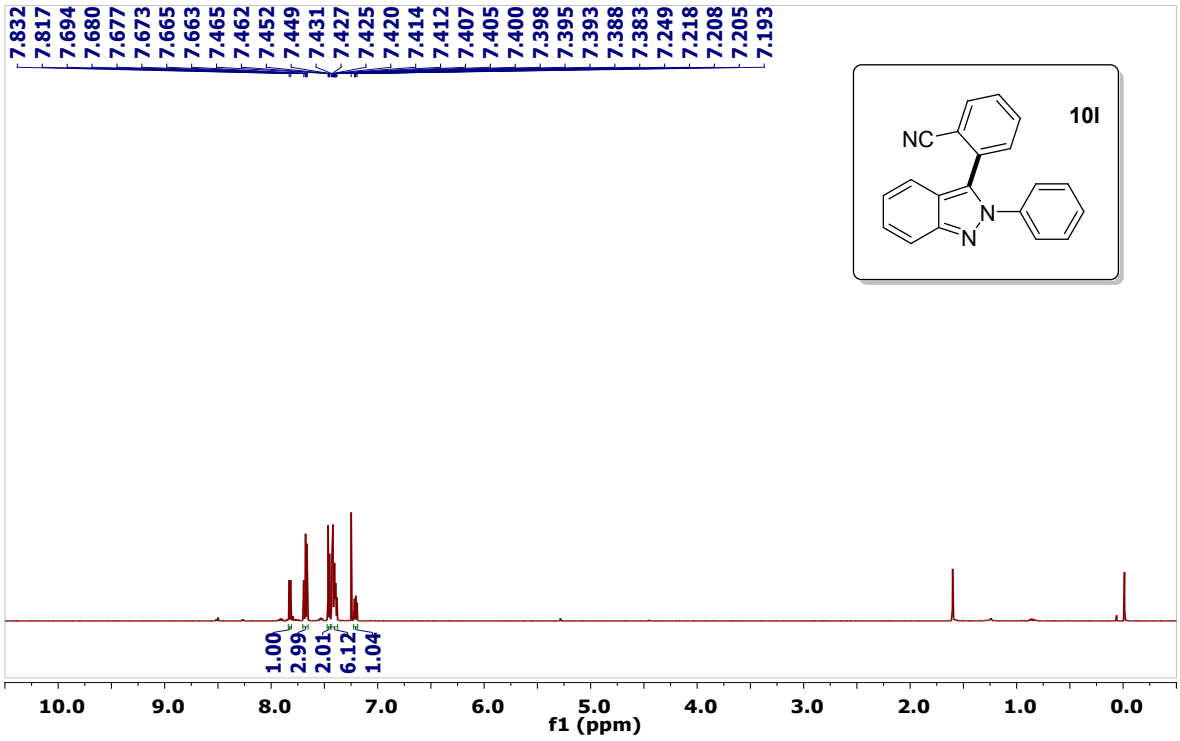
^{1}H NMR Spectrum of 3-(2-chlorophenyl)-2-phenyl-2H-indazole



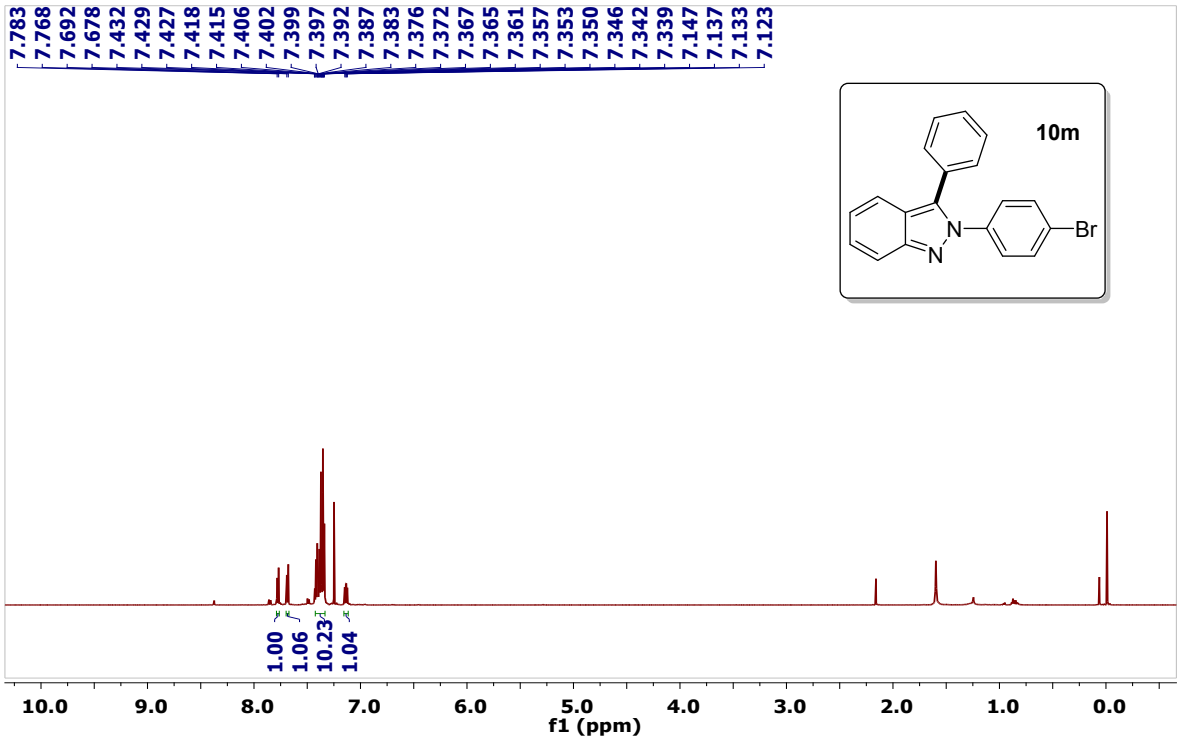
^{1}H NMR Spectrum of 3-(2-bromophenyl)-2-phenyl-2H-indazole



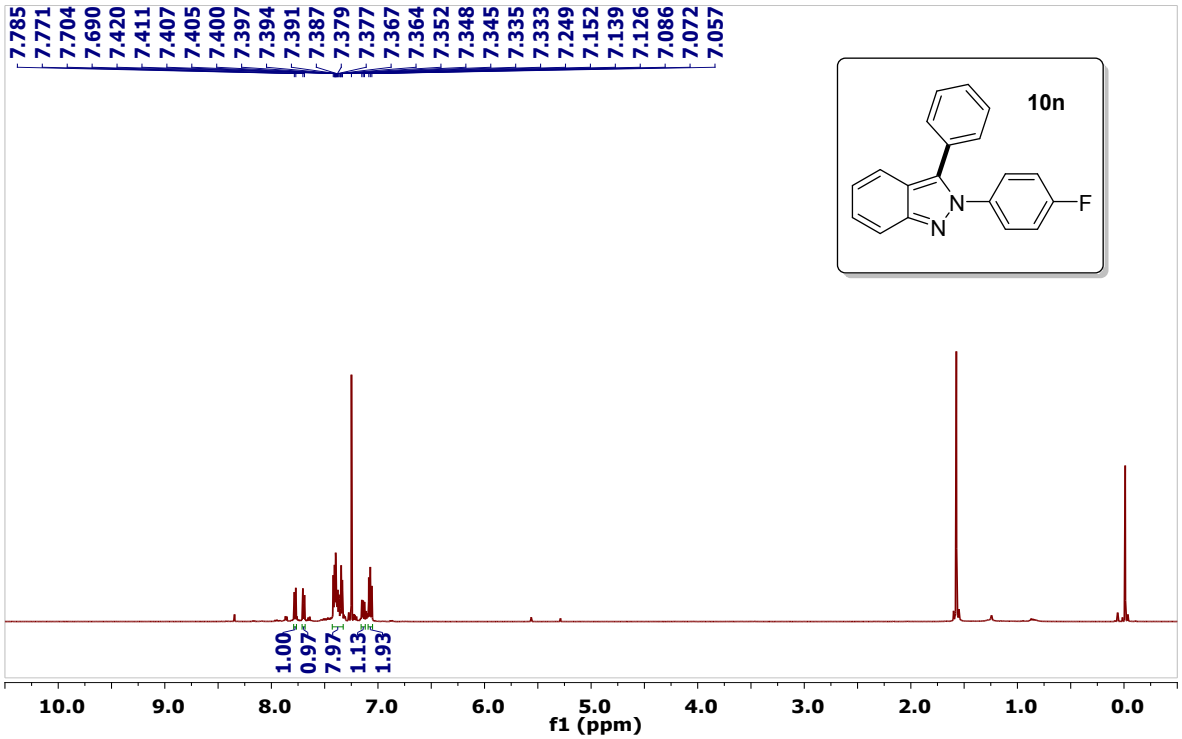
^{1}H NMR Spectrum of 2-(2-phenyl-2H-indazol-3-yl)benzonitrile



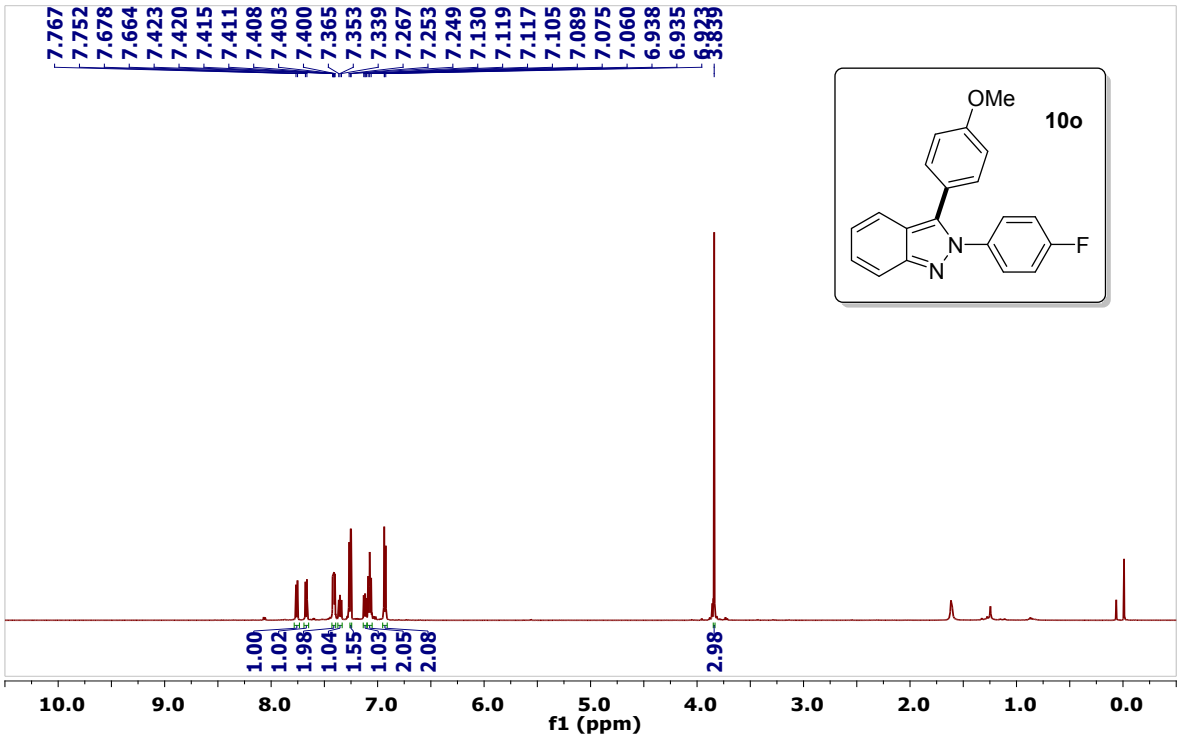
^{1}H NMR Spectrum of 2-(4-bromophenyl)-3-phenyl-2H-indazole



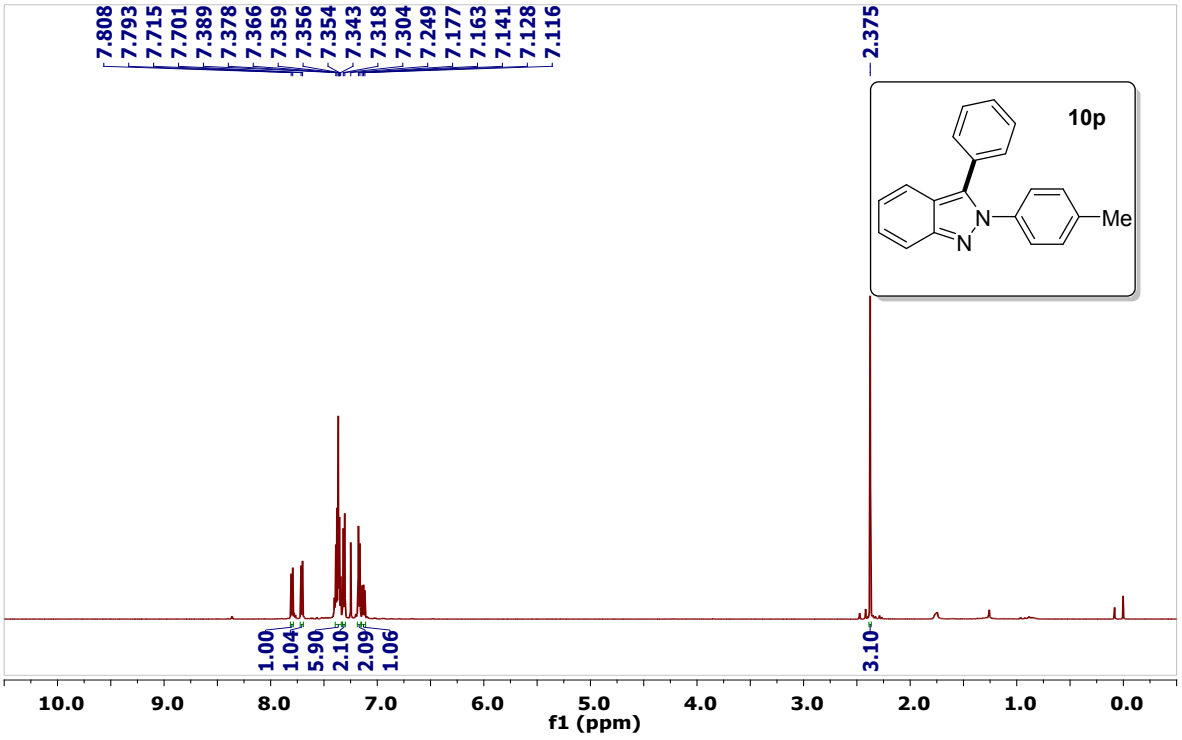
^{1}H NMR Spectrum of 2-(4-fluorophenyl)-3-phenyl-2H-indazole



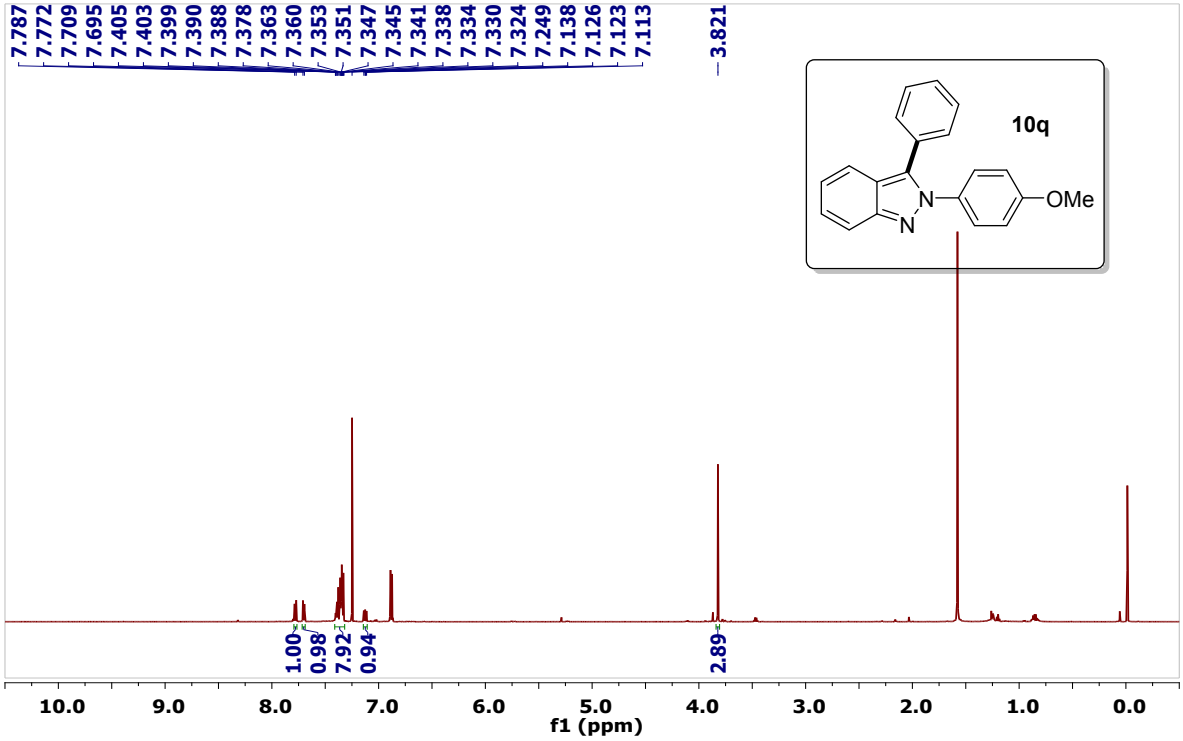
^{1}H NMR Spectrum of 2-(4-fluorophenyl)-3-(4-methoxyphenyl)-2H-indazole



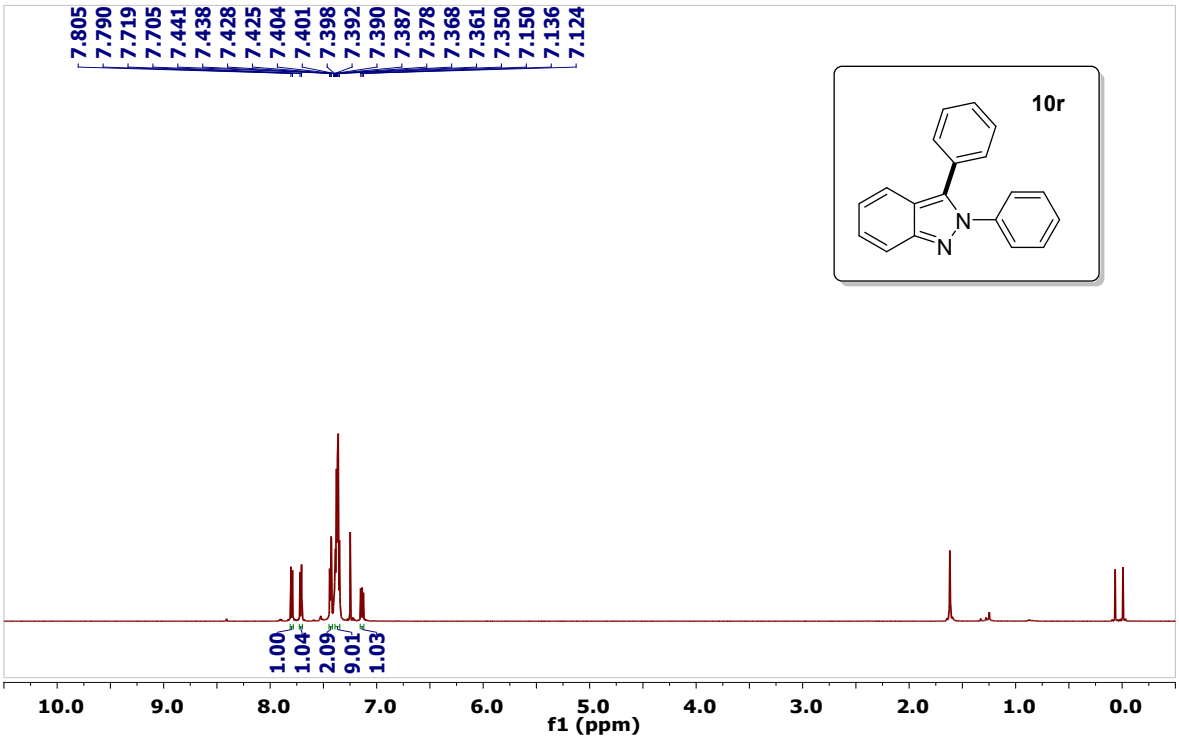
^{1}H NMR Spectrum of 3-phenyl-2-(p-tolyl)-2H-indazole



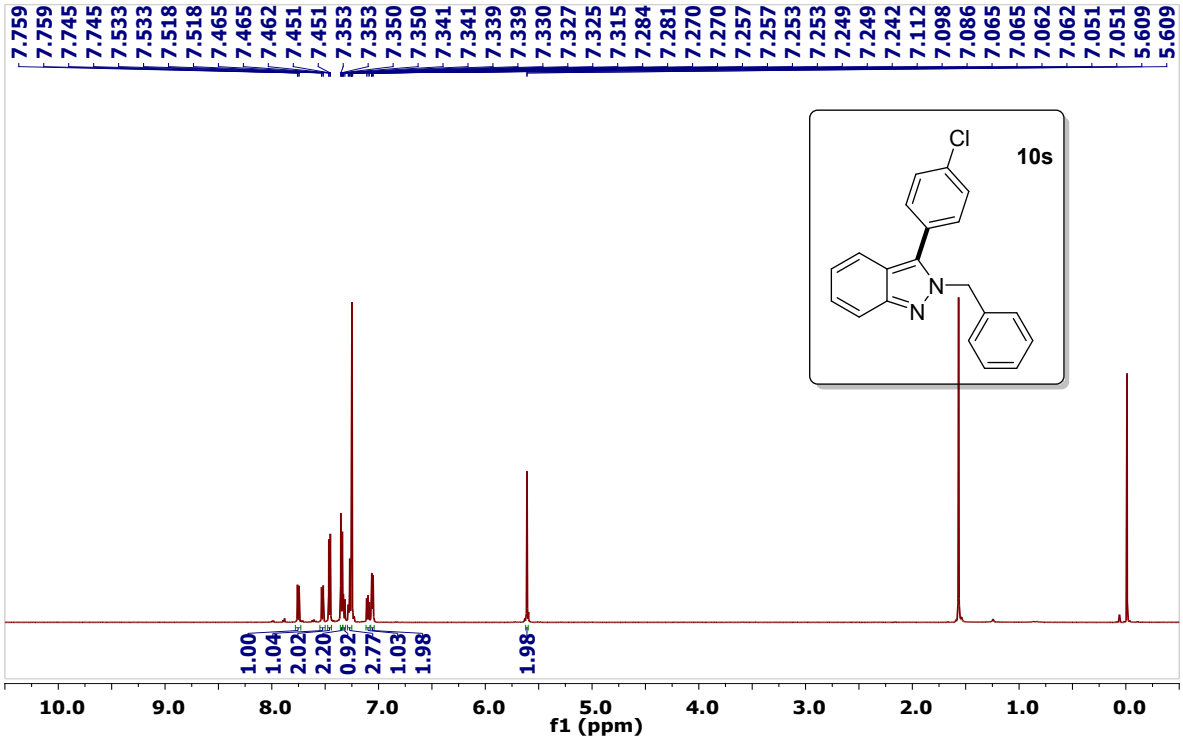
^{1}H NMR Spectrum of 2-(4-methoxyphenyl)-3-phenyl-2H-indazole



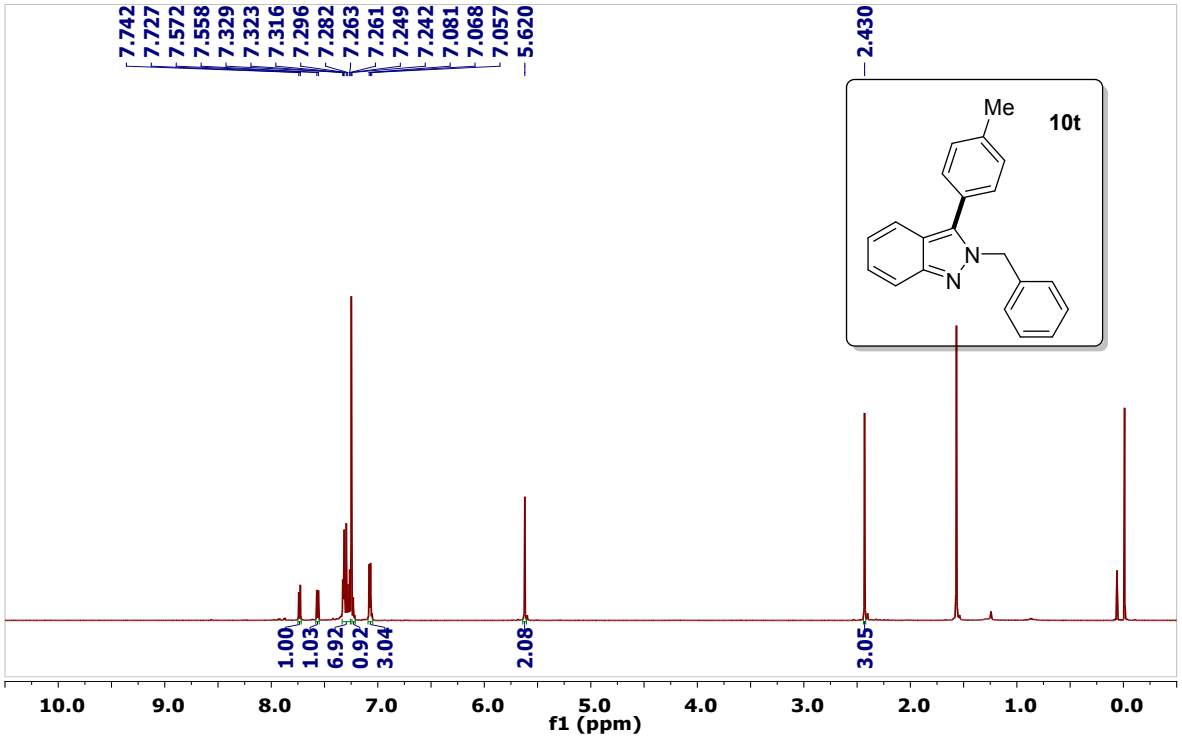
^{1}H NMR Spectrum of 2,3-diphenyl-2H-indazole



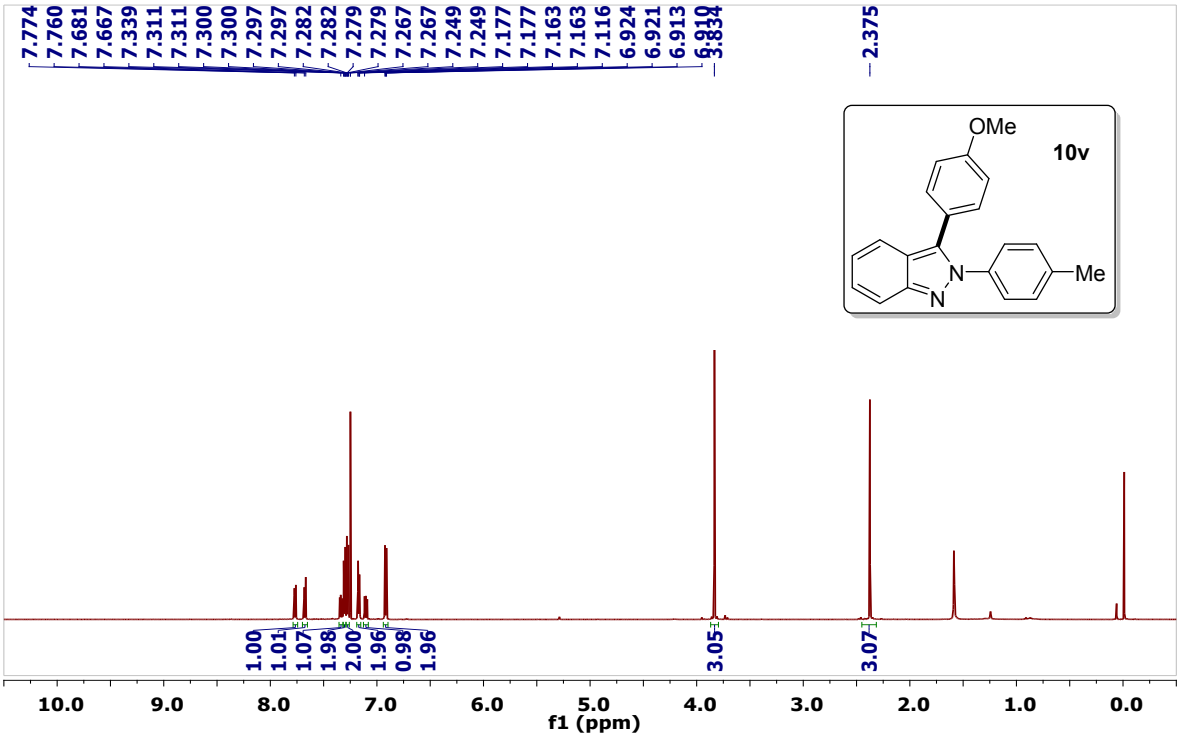
^{1}H NMR Spectrum of 2-benzyl-3-(4-chlorophenyl)-2H-indazole



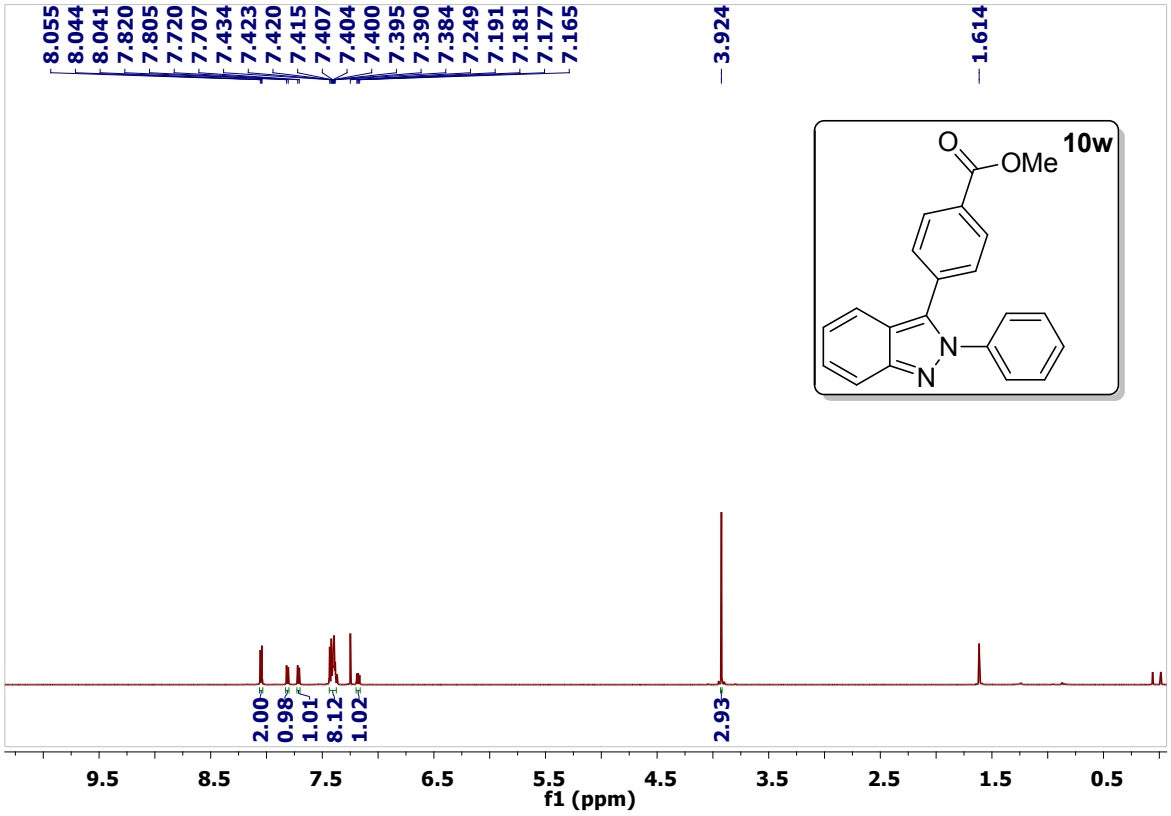
^{1}H NMR Spectrum of 2-benzyl-3-(p-tolyl)-2H-indazole



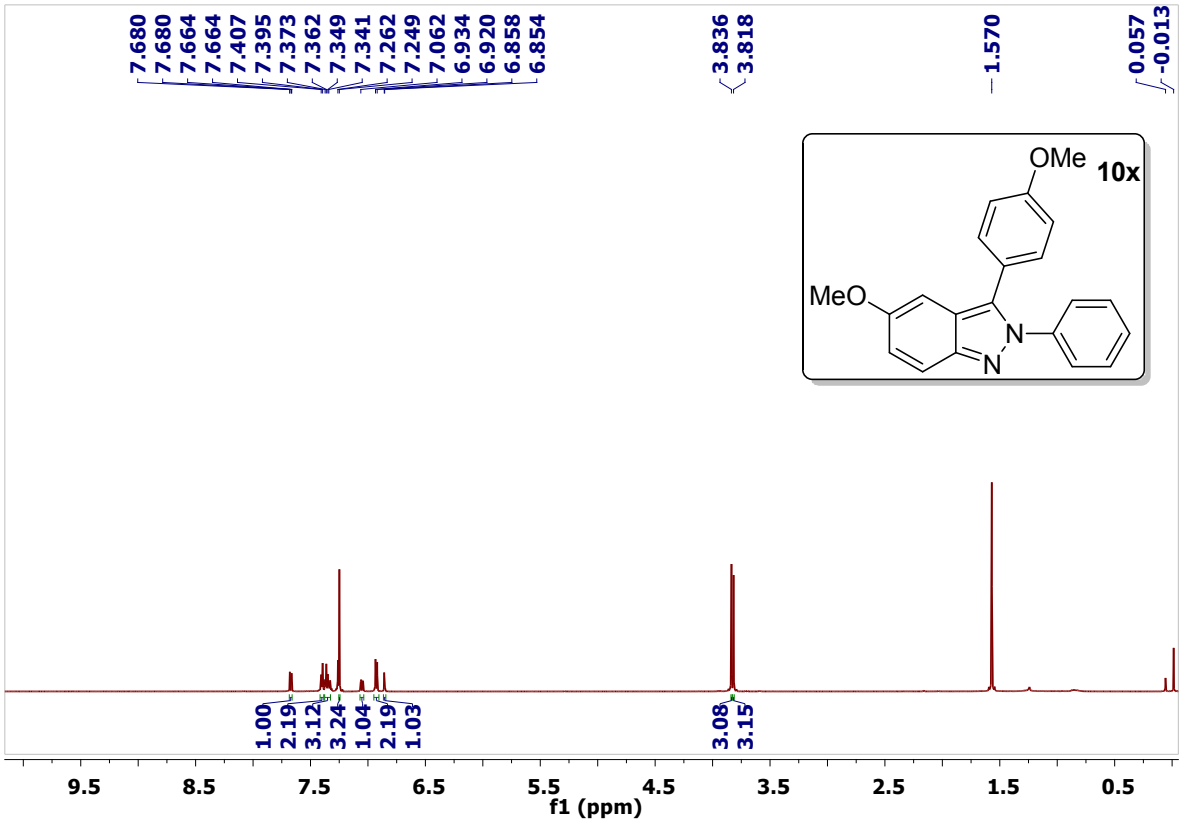
^{1}H NMR Spectrum of 3-(4-methoxyphenyl)-2-(p-tolyl)-2H-indazole



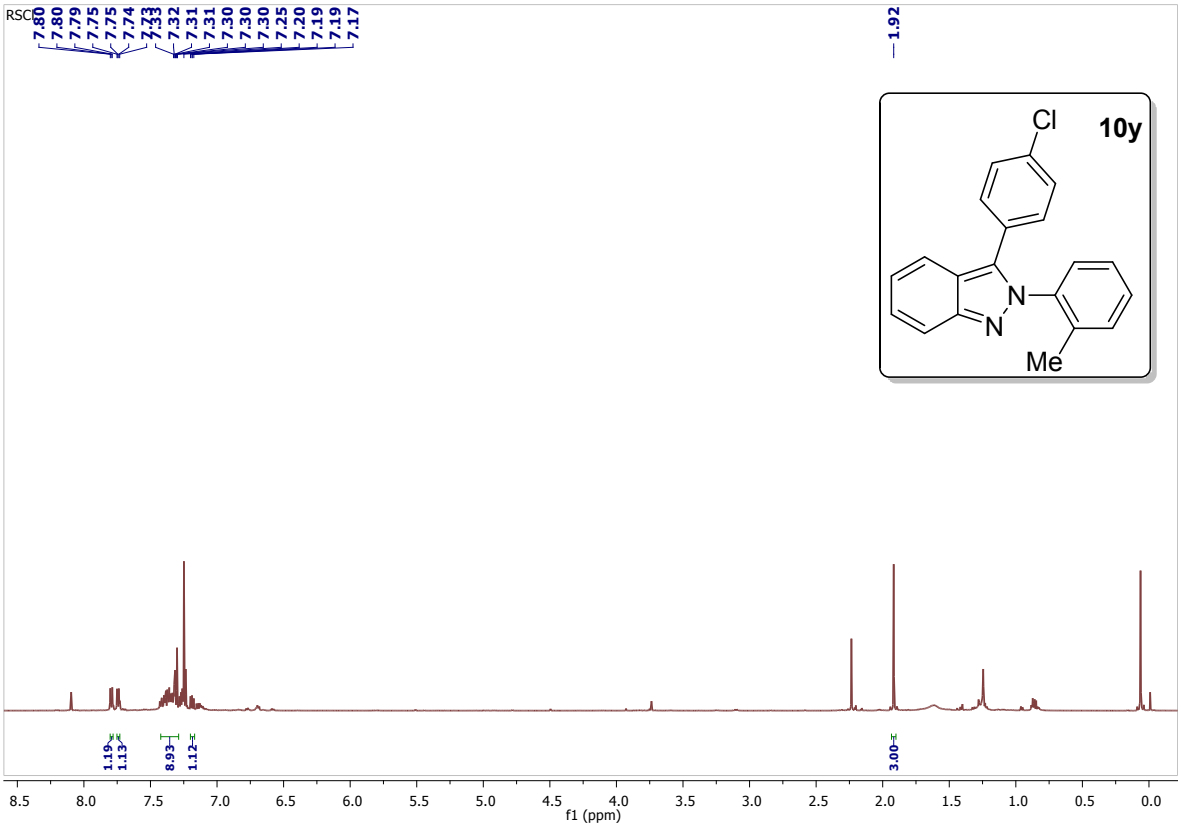
[1]H NMR Spectrum of Methyl 4-(2-phenyl-2H-indazol-3-yl)benzoate



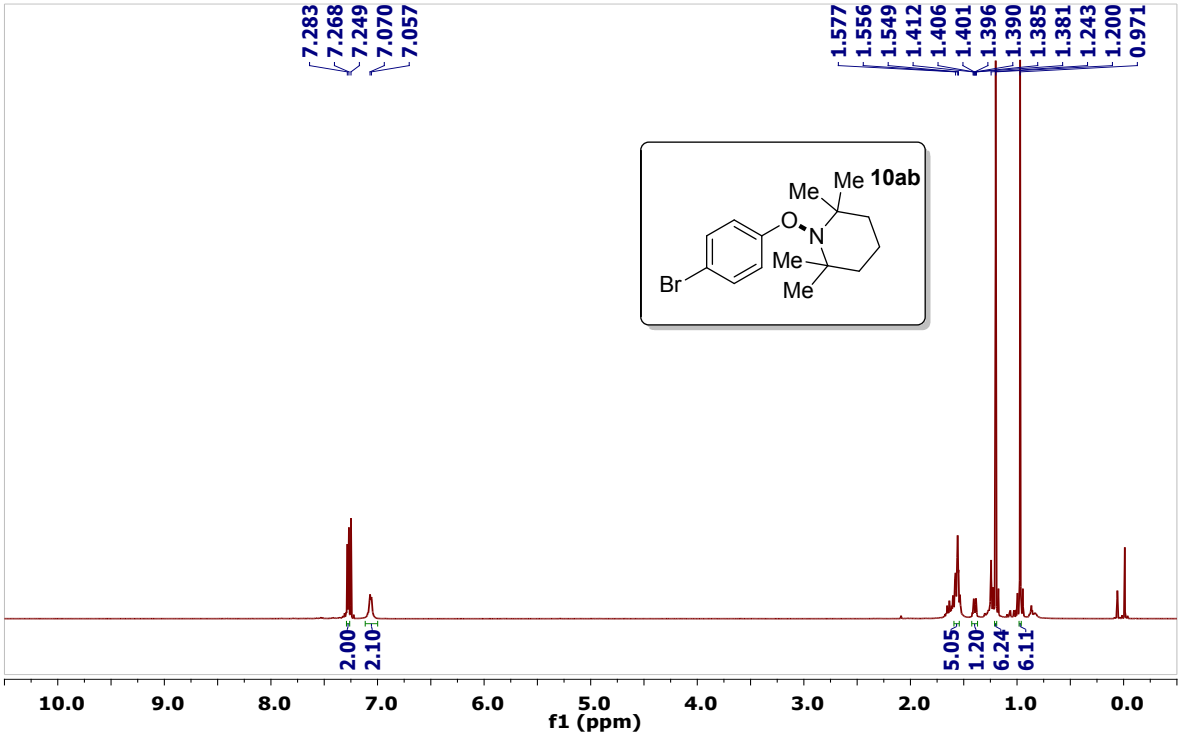
[1]H NMR Spectrum of 5-methoxy-3-(4-methoxyphenyl)-2-phenyl-2H-indazole



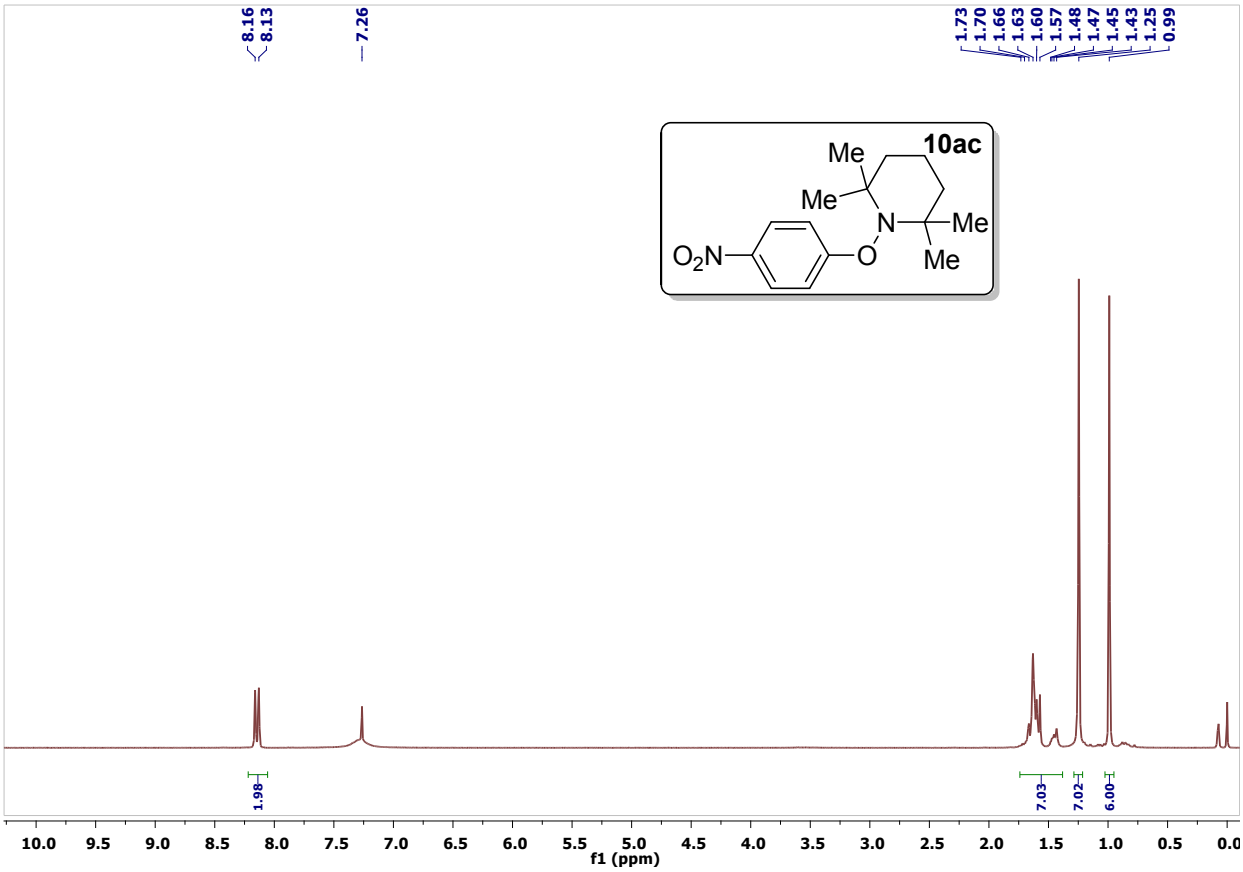
[1]H NMR Spectrum of 3-(4-chlorophenyl)-2-(o-tolyl)-2H-indazole



^{1}H NMR Spectrum of 1-(4-bromophenoxy)-2,2,6,6-tetramethylpiperidine



^{1}H NMR Spectrum of 2,2,6,6-tetramethyl-1-(4-nitrophenoxy) piperidine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8.13
7.26
1.73
1.70
1.66
1.63
1.60
1.57
1.48
1.47
1.45
1.43
1.25
0.99
10ac
Me
Me
N
Me
Me
O2N
O
1.98
7.03
7.02
6.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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5.0
4.5
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3.5
3.0
2.5
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0.0
f1 (ppm)