Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary Information

Rhodium-catalyzed cascade C−H activation/annulation/1,6-acyl migration: Direct construction of free N−H indoles under mild conditions

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General remarks

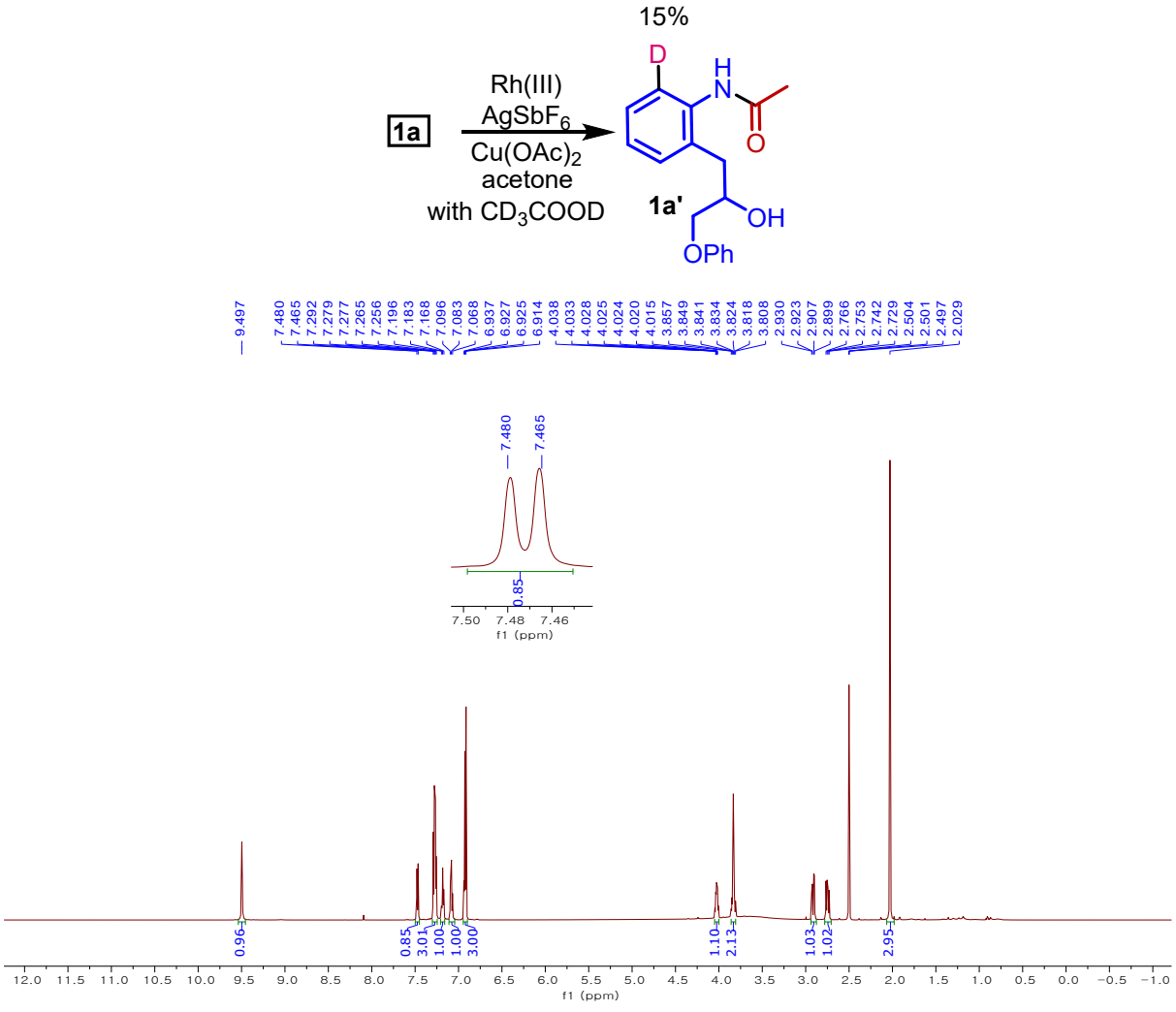
All experiments were carried out under an open atmosphere. Unless otherwise noted, solvents were purchased from commercial suppliers and used without further purification. The substituted acetanilides were synthesized according to the reported procedure.[1] The alkyne coupling partners were purchased from Sigma Aldrich and Alfa aesar. Merck precoated silica gel plates (Art. 5554) treated with a fluorescent indicator were used for analytical thin-layer chromatography (TLC). Column chromatography was performed using silica gel 9385 (Merck) and ethyl acetate/hexane (1:9) were used as the solvents. Melting points are uncorrected and were determined using Fisher-Johns Melting Point Apparatus. ^{1}H NMR and ^{13}C NMR spectra were recorded on VNS (600 and 150 MHz) spectrometer at the core research support center for natural products and medical materials of Yeungnam University. The NMR spectra recorded in $CDCl_3$ using δ = 7.24 and 77.00 ppm as the solvent chemical shifts. All chemical shifts (δ) are expressed in units of ppm and J values are given in Hz. Multiplicities are abbreviated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances, and dd = doublet of doublets. Infrared (IR) spectra were recorded on a PerkinElmer Spectrum Two^{TM} IR spectrometer with frequencies expressed in cm^{-1}, and high-resolution mass spectrometry (HRMS) was carried out using Thermo Fisher Q exactive orbitrap mass spectrometer at the core research support center for natural products and medical materials of Yeungnam University. The crystal structure of the crystal was determined by single-crystal diffraction methods at the Korea Basic Science Institute (KBSI, Western Seoul Center, Korea).

General procedure for the synthesis of 3, 4, 7, and 8.

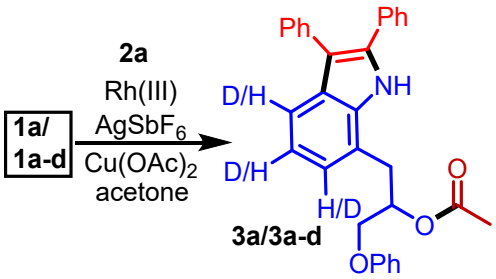
A mixture of acetanilide **1** (1 mmol), alkyne **2** (1 mmol), $[RhCp^*Cl_2]_2$ (1 mol%), $AgSbF_6$ (10 mol%), and $Cu(OAc)_2$ (30 mol%) was stirred in acetone (4 mL) at 40 °C for described time. When the reaction was complete as indicated by TLC, the volatiles was removed in vacuo and the residue was purified by silica gel column chromatography (EtOAc/Hexane= 1:9) to obtain the desired products.

Control experiments

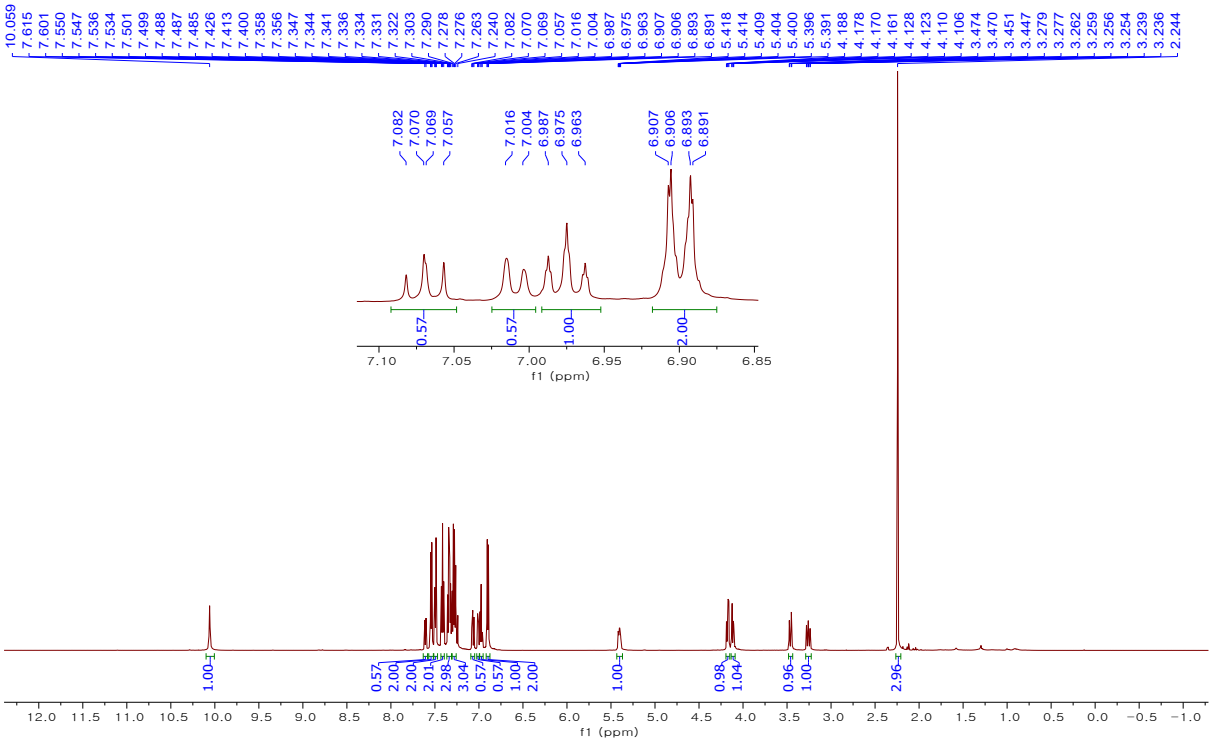
i) Deuterium exchange:



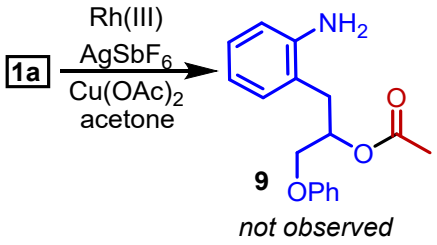
ii) KIE experiment:



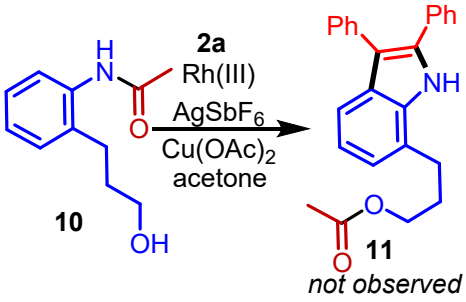
In two different sets of reactions, the reaction of **1a** or **1a-*d*** with **2a** was carried out at standard reaction condition for 8h. After that, both reaction mixtures were combined, and the solvent was removed under reduced pressure. The residue was purified by silica gel chromatography (EtOAc/Hexanes) to afford the product. The KIE was calculated from ^{1}H NMR analysis.



iii) Acyl migration experiment:



iv) Reaction of extended alcohol:



Study of fluorescent probes

Stock solution preparation for spectral detection

Stock solutions of the chloride salts of Ag^{+}, Ba^{2+}, Ca^{2+}, Cd^{2+}, Ce^{3+}, Cu^{2+}, Co^{2+}, Fe^{3+}, Hg^{2+}, Mn^{2+}, Na^{+}, Ni^{2+}, Pb^{2+}, Sn^{2+}, Sr^{2+}, Ti^{3+}, and Zn^{2+} in 10 mM aqueous solutions were prepared. A stock solution of **3a**, **3f**, and **3j** (5 mM) was prepared in Ethanol-water, 1:1 (v/v) HEPES buffer solution (10 mM, pH = 7.4). The working solutions of **3a**, **3f**, and **3j** were freshly prepared by diluting the highly concentrated stock solution to the desired concentration before spectroscopic measurements.

UV–vis and fluorescence spectral studies

All experiments were carried out in Ethanol-water, 1:1 (v/v) HEPES buffer solution (10 mM, pH = 7.4). In all of the spectroscopy experiments, the spectral data were recorded 5 min after the addition of the ions. To investigate the metal ion selectivity, the test samples were prepared

by placing 25 μL of the cation stock solution in 25 μL of the **3a** solution (5 mM) with 1.95 mL of Ethanol-water, 1:1 (v/v) HEPES buffer solution (10 mM, pH = 7.4). For the fluorescence measurements, excitation was provided at 310 nm, and the emission was collected from 320 to 650 nm.

Spectroscopic responses of chemosensor

The spectroscopic properties of receptors **3a**, **3f**, and **3j** (5 mM) in Ethanol-water, 1:1 (v/v) HEPES buffer solution (10 mM, pH = 7.4) was first assessed using the UV–vis and fluorescence spectra (Fig. S1). As shown in Fig. S1a, receptors **3a**, **3f**, and **3j** presented two major absorption bands at 250 and 310 nm corresponding to the π-π* and n-π* transitions. On the other hand, the fluorescence responses of receptors **3a**, **3f**, and **3j** have subsequently investigated fluorescence at 408 nm, when excited at 310 nm as shown in Fig. S1b. In this regard, we have studied the highest fluorescent ability to representative example receptor **3a**; indoles bearing acyl and N-H coordination sites have been utilized as potent fluorescent probes for the selective sensing of metal ions.[2] We firstly studied the UV-Visible and fluorescence spectral response selectivity experiment of receptor **3a** toward 17 common metal ions (Ag^{+}, Ba^{2+}, Ca^{2+}, Cd^{2+}, Ce^{3+}, Cu^{2+}, Co^{2+}, Fe^{3+}, Hg^{2+}, Mn^{2+}, Na^{+}, Ni^{2+}, Pb^{2+}, Sn^{2+}, Sr^{2+}, Ti^{3+}, and Zn^{2+}) in 10 mM aqueous solutions (Fig. 1a,b). The absorption peak became broad with absorption intensity at 310 nm increased only with the presence of Fe^{3+} ions (Fig. 1a). Moreover, upon the addition of various metal ions to the receptor **3a** remarkably displayed excellent "turn-off" response toward only Fe^{3+} ions with significant wavelength shift at 418 nm (λ_{ex} = 310 nm) and the other co-existing metal ions did not induce any major changes in fluorescence emission nature (Fig. 1b).[3] All of these indicating that **3a** coordinates with Fe^{3+} ions to form the complex (**3a**-Fe^{3+}) (Fig. 1c).

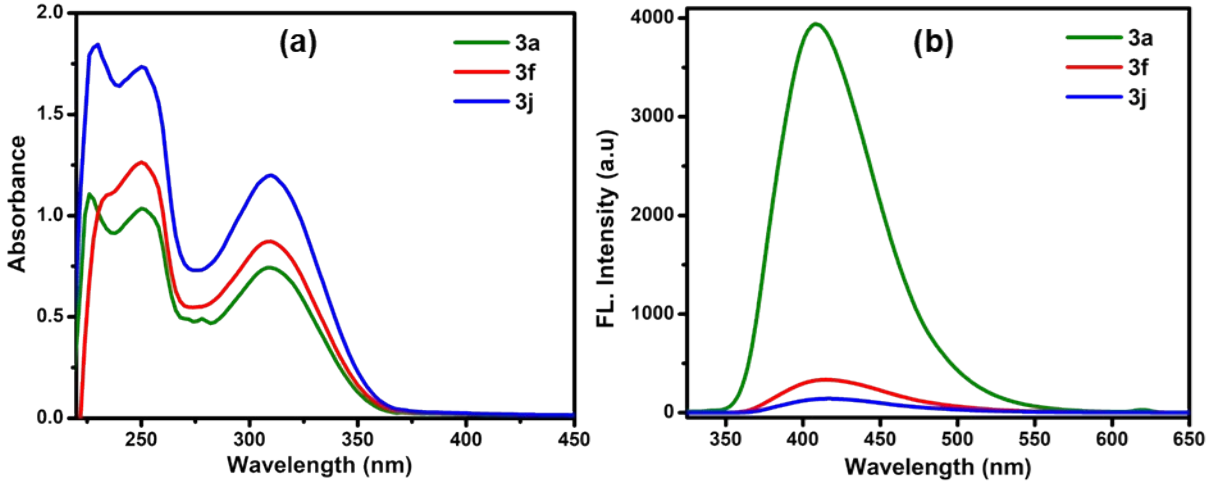


Fig. S1. (a) UV–Vis absorption and (b) fluorescence emission spectra changes of the receptor **3a**, **3f**, and **3j** (5 mM) in Ethanol-water, 1:1 (v/v) HEPES buffer solution (10 mM, pH = 7.4).

The relationship between the fluorescence quenching (F_0/F) and the concentration of the Fe^{3+} quencher, as shown in Fig. 1d, is consistent with the Stern–Volmer relationship.[4] There was a good linear correlation between the emission intensity and Fe^{3+} ions concentrations in the range (0–5.5 mM) with correlation coefficients ($R^2 = 0.9077$). The detection limit of Fe^{3+} ions was obtained by plotting a graph between the relative emission intensity at 408/418 nm as a function of the Fe^{3+} concentration. Based on LOD = K × SD/S, the limit of detection of **3a** for Fe^{3+} was 2.8 μM (Fig. S2).[4]

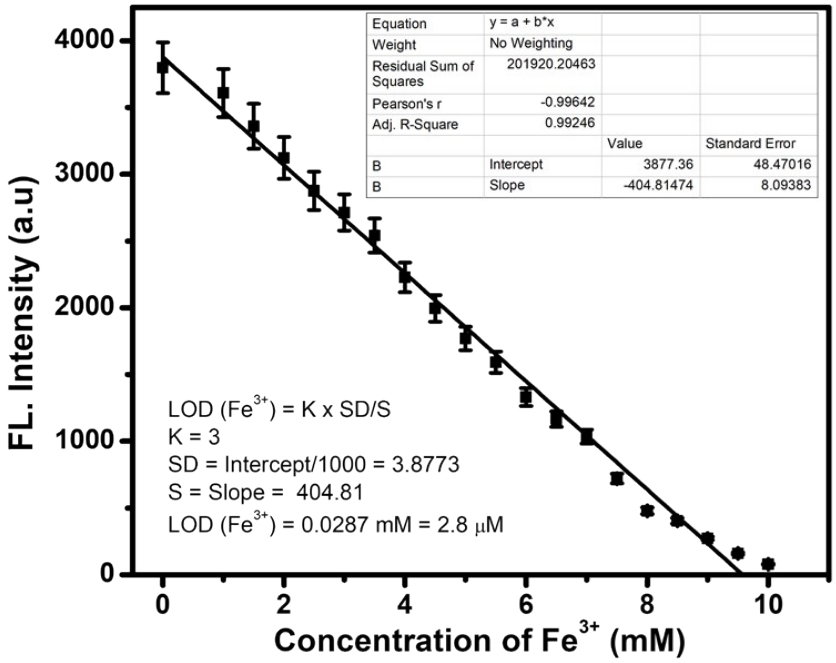


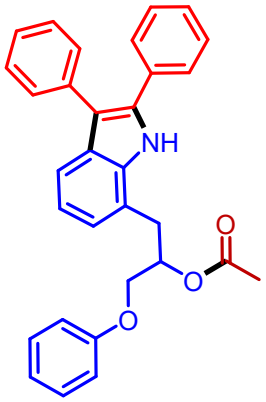
Fig. S2. Calibration curve of **3a** in the presence of Fe^{3+} ions using the monitored emission wavelength at 408 nm (**3a**-Fe^{3+}) complex systems. The detection limit (LOD) was determined from the following equation: LOD = K × SD/S, where K = 3; SD is the standard deviation of the blank solution; S is the slope of the calibration curve.

References

1. R. S. Thombal, T. Feoktistova, G. A. González-Montiel, P. H.-Y. Cheong and Y. R. Lee, *Chem. Sci*., 2020, **11**, 7260-7265.
2. S.K. Sahoo and G. Crisponi. *Molecules*, 2019, **24**, 3267.
3. A. Kuwar, R. Patil, A. Singh, S.K.Sahoo, J. Marek, and N. Singh, *J. Mat. Chem. C.*, 2015, **3**, 453-460.
4. S. Mohandoss, R. Atchudan, T. N. J. I. Edison, K. Mishra, R. J. I. Tamargo, S. Palanisamy, K. Yelithao, S. You, and Y. R. Lee, *Sens. & Actuat. B: Chem.*, 2020, **306**, 127581.

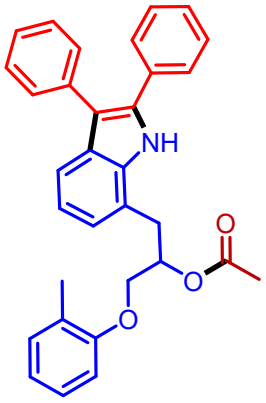
Characterization data of synthesized compounds

1-(2,3-Diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (3a)



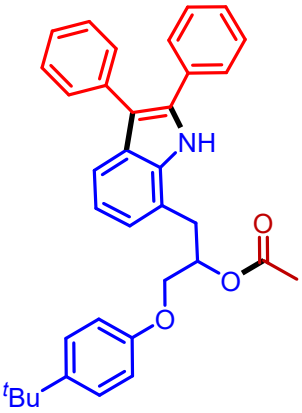
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 86% (398 mg); mp 94-96 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.09 (1H, s), 7.64 (1H, d, J = 8.0 Hz), 7.58 (2H, d, J = 8.3 Hz), 7.53 (2H d, J = 8.1 Hz), 7.45 – 7.42 (2H, m), 7.38 – 7.34 (3H, m), 7.34 – 7.24 (3H, m), 7.10 (1H, t, J = 8.2 Hz), 7.04 (1H, d, J = 7.1 Hz), 7.00 (1H, t, J = 7.5 Hz), 6.92 (2H, d, J = 8.1 Hz), 5.44 – 5.41 (1H, m), 4.21 – 4.18 (1H, m), 4.13 (1H, d, J = 9.9 Hz), 3.48 (1H, d, J = 13.7 Hz), 3.28 (1H, dd, J = 13.8, 10.8 Hz), 2.26 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.4, 158.3, 135.4, 135.3, 134.3, 132.6, 130.2, 129.4, 128.9, 128.5, 128.5, 127.9, 127.4, 126.1, 123.4, 121.2, 120.3, 118.9, 118.8, 114.9, 114.6, 73.5, 67.1, 33.5, 21.3; IR (ATR) 3308, 2995, 1769, 1244, 745 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{28}NO_3$ 462.2063; Found 462.2060.

1-(2,3-Diphenyl-1*H*-indol-7-yl)-3-(o-tolyloxy)propan-2-yl acetate (3b)



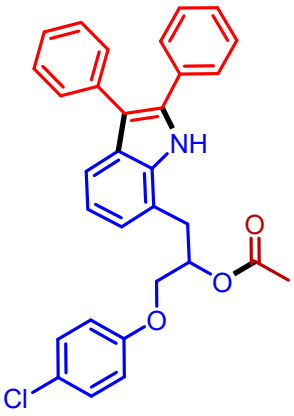
The title compound was prepared according to the general procedure. The product was obtained as a brown solid. Yield: 85% (405 mg); mp 142-144 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.06 (1H, s), 7.61 (1H, d, J = 8.0 Hz), 7.54 (2H, d, J = 8.7 Hz), 7.49 (2H, d, J = 8.1 Hz), 7.40 (1H, d, J = 9 Hz), 7.35 – 7.27 (4H, m), 7.16 (1H, d, J = 7.4 Hz), 7.12 (1H, t, J = 8.0 Hz), 7.07 (1H, t, J = 8.2 Hz), 7.01 (1H, d, J = 7.0 Hz), 6.89 (1H, t, J = 7.4 Hz), 6.75 (1H, d, J = 8.2 Hz), 5.42 – 5.39 (1H, m), 4.20 – 4.09 (2H, m), 3.46 (1H, d, J = 13.6 Hz), 3.29 (1H, dd, J = 12.8, 10.3 Hz), 2.28 (3H, s), 2.23 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.4, 156.4, 135.3, 135.2, 134.3, 132.6, 130.7, 130.2, 128.9, 128.6, 128.5, 127.9, 127.4, 126.8, 126.7, 126.1, 123.4, 120.9, 120.3, 118.9, 118.8, 114.9, 111.2, 73.6, 67.2, 33.5, 21.3, 16.2; IR (ATR) 3327, 2994, 1769, 1241, 744 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{32}H_{30}NO_3$ 476.2220; Found 476.2218.

1-(4-(*tert*-Butyl)phenoxy)-3-(2,3-diphenyl-1*H*-indol-7-yl)propan-2-yl acetate (3c)



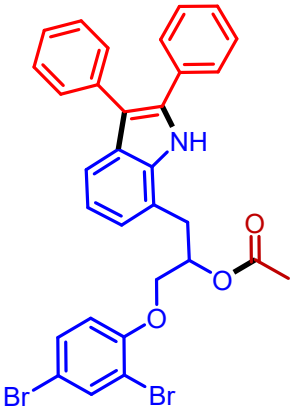
The title compound was prepared according to the general procedure. The product was obtained as a brown solid. Yield: 84% (435 mg); mp 128-130 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.02 (1H, s), 7.59 (1H, d, J = 8.0 Hz), 7.52 (2H, d, J = 7.3 Hz), 7.47 (2H, d, J = 8.4 Hz), 7.40 (2H, t, J = 7.5 Hz), 7.33 – 7.24 (6H, m), 7.05 (1H, t, J = 7.4 Hz), 7.00 (1H, d, J = 6.7 Hz), 6.82 (2H, d, J = 8.9 Hz), 5.39 – 5.35 (1H, m), 4.16 – 4.14 (1H, m), 4.08 (1H, dd, J = 10.8, 2.4 Hz), 3.44 (1H, d, J = 13.9 Hz), 3.25 (1H, dd, J = 13.8, 10.7 Hz), 2.22 (3H, s), 1.29 (9H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.3, 156.0, 143.9, 135.4, 135.3, 134.3, 132.6, 130.2, 128.9, 128.5, 128.5, 127.9, 127.4, 126.2, 126.1, 123.4, 120.2, 118.9, 118.8, 114.9, 114.1, 73.5, 67.2, 34.0, 33.5, 31.4, 21.4; IR (ATR) 3305, 2994, 1769, 1243, 1056, 698 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{35}H_{36}NO_3$ 518.2689; Found 518.2690.

1-(4-Chlorophenoxy)-3-(2,3-diphenyl-1*H*-indol-7-yl)propan-2-yl acetate (3d)



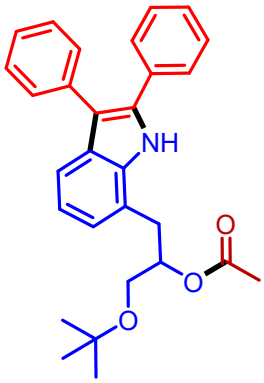
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 79% (392 mg); mp 150-152 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.04 (1H, s), 7.63 (1H, d, J = 8.0 Hz), 7.56 (2H, d, J = 7.6 Hz), 7.51 (2H, d, J = 7.6 Hz), 7.43 (2H, t, J = 7.6 Hz), 7.37-7.30 (4H, m), 7.22 (2H, d, J = 8.6 Hz), 7.08 (1H, t, J = 7.5 Hz), 7.00 (1H, d, J = 7.0 Hz), 6.81 (2H, d, J = 8.7 Hz), 5.41-5.37 (1H, m), 4.13 (1H, dd, J = 10.6, 5.8 Hz), 4.07 (1H, dd, J = 10.6, 2.4 Hz), 3.46 (1H, d, J = 13.8 Hz), 3.22 (1H, dd, J = 13.8, 10.8 Hz), 2.25 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.3, 156.9, 135.3, 135.2, 134.3, 132.5, 130.2, 129.3, 129.0, 128.5, 128.5, 127.9, 127.4, 126.2, 126.1, 123.3, 120.3, 118.9, 118.7, 115.8, 115.0, 73.3, 67.5, 33.5, 21.3; IR (ATR) 3353, 2995, 1769, 1489, 1238, 752 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{27}ClNO_3$ 496.1674; Found 496.1674.

1-(2,4-Dibromophenoxy)-3-(2,3-diphenyl-1*H*-indol-7-yl)propan-2-yl acetate (3e)



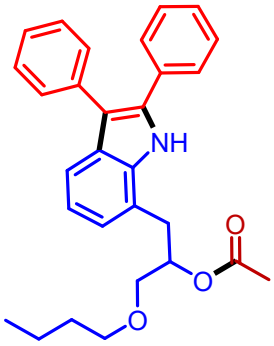
The title compound was prepared according to the general procedure. The product was obtained as a yellow solid. Yield: 77% (480 mg); mp 85-87 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.77 (1H, s), 7.63 (1H, d, J = 2.4 Hz), 7.57 (1H, d, J = 7.9 Hz), 7.47 (2H, d, J = 7.7 Hz), 7.43 (2H, d, J = 7.5 Hz), 7.37 (2H, t, J = 7.7 Hz), 7.32 – 7.26 (5H, m), 7.04 (1H, t, J = 7.5 Hz), 7.00 (1H, d, J = 7.0 Hz), 6.64 (1H, d, J = 8.7 Hz), 5.39 – 5.34 (1H, m), 4.14 (1H, dd, J = 10.6, 2.7 Hz), 4.08 (1H, dd, J = 10.6, 5.3 Hz), 3.43 (1H, dd, J = 13.8, 3.1 Hz), 3.34 (1H, dd, J = 13.8, 10.4 Hz), 2.19 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 154.0, 135.6, 135.2, 134.4, 132.5, 131.2, 131.1, 130.2, 129.0, 128.6, 128.5, 128.0, 127.5, 126.2, 123.4, 120.3, 118.9, 118.6, 115.0, 114.6, 113.7, 113.2, 73.0, 68.4, 33.1, 21.3; IR (ATR) 3330, 2996, 1769, 1244, 696 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{26}Br^{79}Br^{81}NO_3$ 620.0253; Found 620.0250.

1-(*tert*-Butoxy)-3-(2,3-diphenyl-1*H*-indol-7-yl)propan-2-yl acetate (3f)



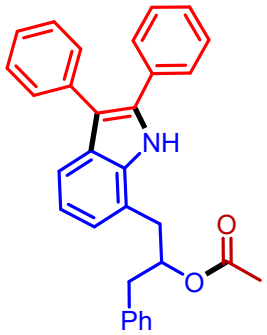
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 80% (354 mg); mp 136-138 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.17 (1H, s), 7.60 (1H, d, J = 9.1 Hz), 7.57 (2H, d, J = 8.4 Hz), 7.49 (2H, d, J = 8.0 Hz), 7.40 – 7.38 (2H, m), 7.35 – 7.33 (2H, m), 7.29 (2H, q, J = 8.6 Hz), 7.09 – 7.06 (1H, m), 7.02 (1H, d, J = 9.6 Hz), 5.12 – 5.10 (1H, m), 3.51 (2H, s), 3.30 (1H, d, J = 13.7 Hz), 3.21 (1H, t, J = 13.1 Hz), 2.19 (3H, s), 1.17 (9H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 135.7, 135.5, 134.3, 132.8, 130.2, 128.7, 128.5, 128.4, 128.2, 127.4, 126.0, 123.5, 120.1, 119.6, 118.4, 114.8, 74.5, 73.6, 61.2, 33.3, 27.4, 21.4; IR (ATR) 3311, 2966, 1769, 1247, 696 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{29}H_{32}NO_3$ 442.2376; Found 442.2375.

1-Butoxy-3-(2,3-diphenyl-1*H*-indol-7-yl)propan-2-yl acetate (3g)



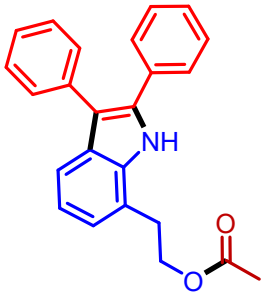
The title compound was prepared according to the general procedure. The product was obtained as a viscous oil. Yield: 82% (360 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.10 (s, 1H), 7.57 – 7.53 (3H, m), 7.47 – 7.45 (2H, m), 7.40 – 7.36 (2H, m), 7.35 – 7.31 (2H, m), 7.31 – 7.25 (2H, m), 7.05 (1H, td, J = 7.6, 2.5 Hz), 6.99 (1H, d, J = 7.0 Hz), 5.17 – 5.14 (1H, m), 3.60 – 3.57 (1H, m), 3.54 (1H, dt, J = 11.2, 2.7 Hz), 3.51 – 3.46 (1H, m), 3.45 – 3.40 (1H, m), 3.30 (1H, d, J = 16.2 Hz), 3.15 (1H, t, J = 12.0 Hz), 2.20 (3H, s), 1.55 (2H, t, J = 6.4 Hz), 1.36 – 1.32 (2H, m), 0.90 (3H, t, J = 7.4 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.2, 135.5, 135.5, 134.3, 132.7, 130.2, 128.8, 128.5, 128.4, 128.0, 127.4, 126.1, 123.5, 120.2, 119.3, 118.5, 114.9, 74.3, 71.5, 69.8, 33.3, 31.6, 21.4, 19.2, 13.8; IR (ATR) 3334, 2955, 1769, 1244, 696 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{29}H_{32}NO_3$ 442.2376; Found 442.2374.

1-(2,3-Diphenyl-1*H*-indol-7-yl)-3-phenylpropan-2-yl acetate (3h)



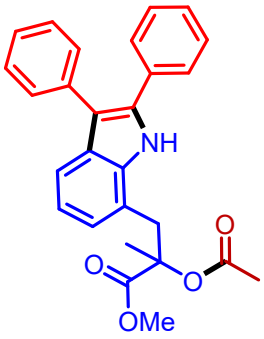
The title compound was prepared according to the general procedure. The product was obtained as a black solid. Yield: 78% (347 mg); mp 125-127 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.00 (1H, s), 7.50 (1H, d, J = 7.9 Hz), 7.46 (2H, dd, J = 8.4, 1.2 Hz), 7.40 (2H, dd, J = 8.0, 1.3 Hz), 7.31 (2H, t, J = 7.7 Hz), 7.24 – 7.21 (3H, m), 7.18 – 7.14 (3H, m), 7.12 – 7.07 (3H, m), 7.01 – 6.98 (1H, m), 6.95 (1H, d, J = 7.0 Hz), 5.14 (1H, m), 3.36 (1H, dd, J = 13.6, 2.3 Hz), 2.96 (1H, dd, J = 14.6, 3.2 Hz), 2.90 – 2.82 (2H, m), 2.02 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.5, 137.4, 135.5, 135.3, 134.2, 132.7, 130.2, 129.4, 128.9, 128.5, 128.5, 128.2, 127.9, 127.4, 126.4, 126.1, 123.3, 120.1, 119.6, 118.6, 114.9, 75.6, 39.0, 37.8, 21.3; IR (ATR) 3359, 2995, 1769, 1244, 696 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{28}NO_2$ 446.2114; Found 446.2111.

2-(2,3-Diphenyl-1*H*-indol-7-yl)ethyl acetate (3i)



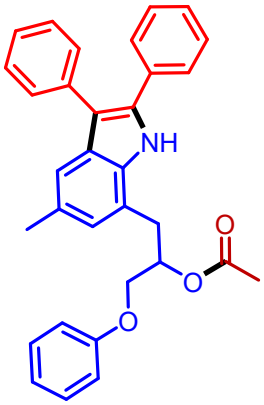
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 87% (309 mg); mp 107-109 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.48 (1H, s), 7.56 (1H, d, J = 7.8 Hz), 7.52 (2H, d, J = 7.1 Hz), 7.46 – 7.44 (2H, m), 7.37 (2H, t, J = 7.7 Hz), 7.33 (2H, t, J = 7.5 Hz), 7.30 – 7.26 (2H, m), 7.06 (1H, t, J = 7.4 Hz), 7.02 (1H, d, J = 7.2 Hz), 4.45 – 4.41 (2H, m), 3.23 (2H, t, J = 7.7 Hz), 2.13 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 135.3, 135.2, 134.1, 132.7, 130.2, 128.9, 128.6, 128.5, 128.0, 127.5, 126.2, 122.8, 120.3, 119.6, 118.6, 115.1, 64.3, 31.8, 21.1; IR (ATR) 3366, 2926, 1712, 1255, 1027, 695 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{24}H_{22}NO_2$ 356.1645; Found 356.1641.

Methyl 2-acetoxy-3-(2,3-diphenyl-1*H*-indol-7-yl)-2-methylpropanoate (3j)



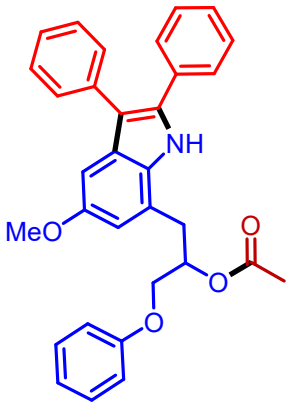
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 75% (320 mg); mp 73-75 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.79 (1H, s), 7.59 (1H, d, J = 7.9 Hz), 7.47 (2H, d, J = 7.0 Hz), 7.45 – 7.43 (2H, m), 7.38 (2H, t, J = 7.6 Hz), 7.34 – 7.31 (2H, m), 7.31 – 7.26 (2H, m), 7.10 – 7.07 (1H, m), 7.01 (1H, d, J = 7.2 Hz), 3.68 (3H, s), 3.58 (1H, d, J = 14.5 Hz), 3.39 (1H, d, J = 14.5 Hz), 1.98 (3H, s), 1.63 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.2, 170.1, 135.8, 135.1, 133.8, 132.5, 130.1, 129.1, 128.7, 128.5, 127.8, 127.6, 126.2, 125.5, 120.2, 119.0, 117.6, 115.1, 81.5, 52.6, 40.5, 21.4, 21.3; IR (ATR) 3330, 2995, 1758, 1245, 697 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{27}H_{26}NO_4$ 428.1856; Found 428.1854.

1-(5-Methyl-2,3-diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (3l)



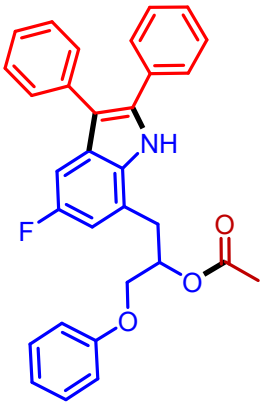
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 78% (371 mg); mp 150-152 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.80 (1H, s), 7.42 – 7.35 (4H, m), 7.32 – 7.29 (2H, m), 7.26 (1H, s), 7.23 – 7.29 (3H, m), 7.18 – 7.15 (3H, m), 6.89 – 6.86 (1H, m), 6.81 (2H, d, J = 5.5 Hz), 6.74 (1H, s), 5.31 – 5.28 (1H, m), 4.09 – 4.06 (1H, m), 4.03 – 4.00 (1H, m), 3.32 (1H, d, J = 13.8 Hz), 3.10 (1H, t, J = 13.9 Hz), 2.30 (3H, s), 2.13 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.3, 158.3, 135.6, 134.4, 133.6, 132.7, 130.2, 129.6, 129.5, 129.2, 128.5, 128.4, 127.9, 127.3, 126.1, 125.1, 121.2, 118.6, 118.3, 114.6, 114.5, 73.5, 67.2, 33.6, 21.4; IR (ATR) 3328, 2996, 1769, 1242, 691 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{32}H_{30}NO_3$ 476.2220; Found 476.2219.

1-(5-Methoxy-2,3-diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (3m)



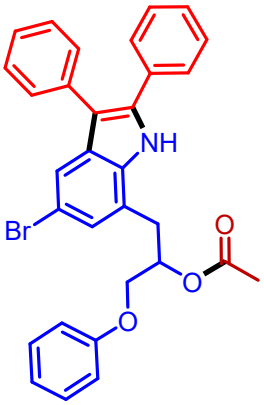
The title compound was prepared according to the general procedure. The product was obtained as a black solid. Yield: 77% (379 mg); mp 142-144 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.78 (1H, s), 7.38 (4H, t, J = 9.5 Hz), 7.33 – 7.28 (2H, m), 7.28 – 7.20 (3H, m), 7.18 (3H, t, J = 7.6 Hz), 6.92 (1H, s), 6.89 – 6.84 (1H, m), 6.79 (2H, d, J = 8.4 Hz), 6.60 (1H, s), 5.33 – 5.25 (1H, m), 4.11 – 3.98 (2H, m), 3.69 (3H, s), 3.32 (1H, d, J = 13.7 Hz), 3.08 (1H, t, J = 12.3 Hz), 2.13 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.3, 158.3, 154.6, 135.5, 135.0, 132.6, 132.2, 130.5, 130.1, 129.5, 129.2, 128.5, 127.8, 127.3, 126.1, 121.2, 119.9, 114.8, 114.6, 113.9, 99.9, 73.4, 67.1, 55.8, 33.5, 21.3; IR (ATR) 3330, 2994, 1769, 1244, 691 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{32}H_{30}NO_4$ 492.2169; Found 492.2165.

1-(5-Fluoro-2,3-diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (3n)



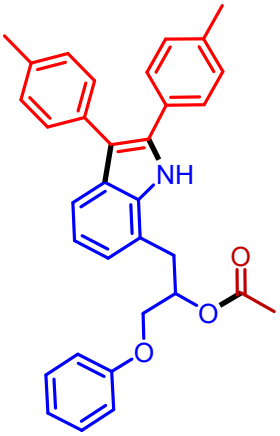
The title compound was prepared according to the general procedure. The product was obtained as a yellow solid. Yield: 72% (308 mg); mp 148-150 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.96 (1H, s), 7.46 (2H, d, J = 8.5 Hz), 7.41 – 7.36 (4H, m), 7.32 – 7.25 (6H, m), 7.20 (1H, d, J = 9.7 Hz), 6.95 (1H, t, J = 7.4 Hz), 6.87 (2H, d, J = 8.1 Hz), 6.76 (1H, d, J = 9.4 Hz), 5.38 – 5.32 (1H, m), 4.14 (1H, dd, J = 10.5, 5.7 Hz), 4.08 (1H, dd, J = 10.7, 2.7 Hz), 3.40 (1H, d, J = 13.9 Hz), 3.19 (1H, dd, J = 13.9, 10.6 Hz), 2.21 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.4, 158.2, 158.1 (d, J = 234 Hz), 135.9, 135.3, 134.9, 132.3, 131.8, 130.0, 129.5, 129.3, 128.6, 128.6, 128.3, 127.9, 127.7, 126.4, 121.4, 120.0 (d, J = 9 Hz), 114.7, 111.6 (d, J = 26 Hz), 103.5 (d, J = 23 Hz), 73.2, 67.1, 33.4, 21.3; IR (ATR) 3382, 1712, 1458, 1237, 693 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{27}FNO_3$ 428.1856; Found 428.1854.

1-(5-Bromo-2,3-diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (3o)



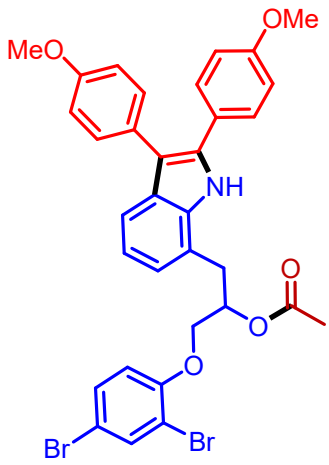
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 73% (395 mg); mp 118-120 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.03 (1H, s), 7.59 (1H, s), 7.40 (2H, d, J = 8.4 Hz), 7.32-7.30 (4H, m), 7.24 (3H, t, J = 7.2 Hz), 7.21 – 7.17 (3H, m), 7.02 (1H, s), 6.89 (1H, t, J = 7.2 Hz), 6.81 (2H, d, J = 8.4 Hz), 5.28-5.25 (1H, m), 4.08 (1H, dd, J = 10.8, 6.0 Hz), 4.02 (1H, dd, J = 10.2, 2.4 Hz), 3.32 (1H, d, J = 13.8 Hz), 3.10 (1H, dd, J = 15.0, 11.4 Hz), 2.15 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.5, 158.2, 135.4, 134.7, 134.0, 132.0, 130.6, 130.1, 129.5, 128.6, 127.9, 127.8, 126.5, 125.8, 121.4, 121.2, 120.7, 114.6, 114.6, 114.6, 113.2, 73.2, 67.2, 33.4, 21.3; IR (ATR) 3307, 2923, 1769, 1239, 694 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{27}BrNO_3$ 540.1168; Found 540.1165.

1-(2,3-Di-*p*-tolyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (4b)



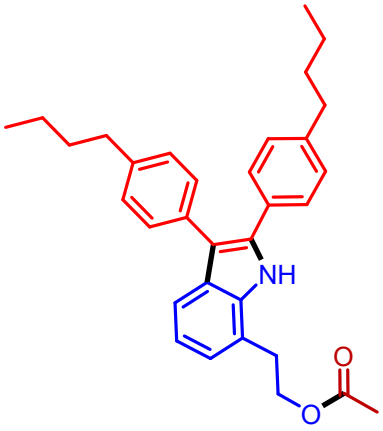
The title compound was prepared according to the general procedure. The product was obtained as a black solid. Yield: 74% (361 mg); mp 130-132 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.97 (1H, s), 7.58 (1H, d, J = 7.9 Hz), 7.44 (2H, d, J = 8.2 Hz), 7.38 (2H, d, J = 8.0 Hz), 7.27 (2H, dd, J = 8.8, 7.3 Hz), 7.22 (2H, d, J = 7.8 Hz), 7.15 (2H, d, J = 7.9 Hz), 7.04 (1H, t, J = 7.5 Hz), 6.99 – 6.95 (2H, m), 6.89 (2H, d, J = 7.5 Hz), 5.40 – 5.38 (1H, m), 4.16 (1H, dd, J = 10.6, 5.7 Hz), 4.10 (1H, dd, J = 10.6, 2.5 Hz), 3.44 (1H, dd, J = 13.9, 2.5 Hz), 3.23 (1H, dd, J = 13.8, 10.7 Hz), 2.42 (3H, s), 2.37 (3H, s), 2.23 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.4, 158.3, 137.1, 135.6, 135.1, 134.3, 132.4, 130.0, 129.8, 129.4, 129.3, 129.2, 129.0, 127.8, 123.1, 121.2, 120.1, 118.7, 118.7, 114.6, 114.4, 73.5, 67.1, 33.5, 21.4, 21.2; IR (ATR) 3334, 2995, 1769, 1241, 750 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{33}H_{32}NO_3$ 490.2376; Found 490.2376.

1-(2,3-bis(4-Methoxyphenyl)-1*H*-indol-7-yl)-3-(2,4-dibromophenoxy)propan-2-yl acetate (4c)



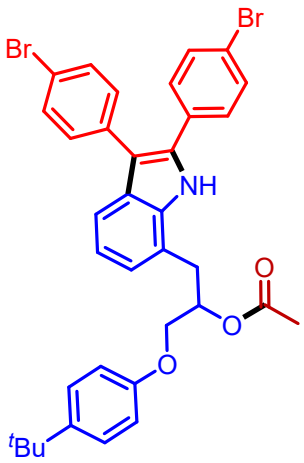
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 75% (509 mg); mp 86-88 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.66 (1H, s), 7.63 (1H, d, J = 2.3 Hz), 7.52 (1H, d, J = 7.8 Hz), 7.41 (2H, d, J = 8.2 Hz), 7.35 (2H, d, J = 8.0 Hz), 7.28 (1H, dd, J = 8.7, 2.3 Hz), 7.02 (1H, t, J = 7.5 Hz), 6.98 – 6.91 (3H, m), 6.86 (2H, d, J = 8.4 Hz), 6.63 (1H, d, J = 8.7 Hz), 5.39 – 5.34 (1H, m), 4.13 (1H, dd, J = 10.6, 2.6 Hz), 4.07 (1H, dd, J = 10.6, 5.3 Hz), 3.84 (3H, s), 3.82 (3H, s), 3.41 (1H, dd, J = 13.8, 2.9 Hz), 3.32 (1H, dd, J = 13.8, 10.4 Hz), 2.20 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 159.0, 158.0, 154.0, 135.5, 135.0, 134.0, 131.2, 129.1, 127.7, 125.1, 123.0, 120.1, 118.6, 118.4, 114.5, 114.0, 113.9, 113.6, 113.2, 73.0, 68.3, 55.2, 33.0, 31.5, 21.3; IR (ATR) 3348, 1717, 1452, 1235, 1029, 832 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{33}H_{30}Br^{79}Br^{81}NO_5$ 680.0464; Found 680.0457.

2-(2,3-Bis(4-butylphenyl)-1*H*-indol-7-yl)ethyl acetate (4d)



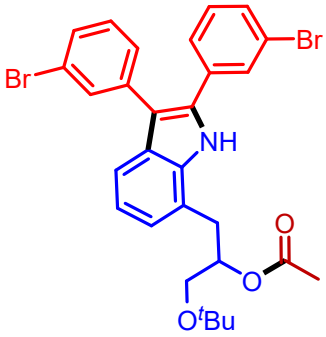
The title compound was prepared according to the general procedure. The product was obtained as a viscous liquid. Yield: 79% (370 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ9.39 (1H, s), 7.56 (1H, d, J = 7.8 Hz), 7.45 (2H, d, J = 8.2 Hz), 7.37 (2H, d, J = 8.0 Hz), 7.20 (2H, d, J = 7.9 Hz), 7.15 (2H, d, J = 8.1 Hz), 7.05 (1H, t, J = 7.5 Hz), 7.01 (1H, d, J = 7.0 Hz), 4.46 – 4.40 (2H, m), 3.23 (2H, t, J = 7.7 Hz), 2.69 – 2.64 (2H, m), 2.61 (2H, t, J = 7.8 Hz), 2.13 (3H, s), 1.70 – 1.64 (2H, m), 1.63 – 1.62 (2H, m), 1.45 – 1.35 (4H, m), 0.97 (3H, t, J = 7.4 Hz), 0.94 (3H, t, J = 7.4 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.1, 142.2, 140.6, 135.1, 134.1, 132.5, 130.1, 130.0, 129.1, 128.6, 128.4, 127.7, 122.6, 120.1, 119.4, 118.6, 114.7, 64.3, 35.4, 35.4, 33.5, 33.4, 31.8, 22.5, 22.4, 21.1, 14.0, 13.9; IR (ATR) 3353, 2927, 1719, 1453, 1236, 745 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{32}H_{38}NO_2$ 468.2897; Found 468.2896.

1-(2,3-Bis(4-bromophenyl)-1*H*-indol-7-yl)-3-(4-(*tert*-butyl)phenoxy)propan-2-yl acetate (4e)



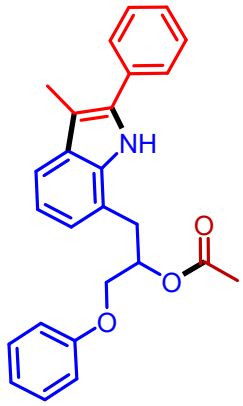
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 72% (487 mg); mp 113-115 ^{o}C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.13 (1H, s), 7.51 (3H, d, J = 8.4 Hz), 7.45 (2H, d, J = 8.5 Hz), 7.34 (2H, d, J = 8.5 Hz), 7.29 (2H, d, J = 8.4 Hz), 7.26 (2H, d, J = 8.7 Hz), 7.06 – 7.02 (1H, m), 7.00 (1H, d, J = 7.1 Hz), 6.79 (2H, d, J = 8.8 Hz), 5.31 – 5.29 (1H, m), 4.12 (1H, dd, J = 10.6, 5.7 Hz,), 4.05 (1H, dd, J = 10.6, 2.4 Hz), 3.39 (1H, dd, J = 13.7, 2.3 Hz), 3.23 (1H, dd, J = 13.8, 10.8 Hz), 2.22 (3H, s), 1.28 (9H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.6, 156.0, 144.1, 135.4, 134.1, 133.3, 131.9, 131.8, 131.7, 131.2, 129.4, 128.5, 126.3, 123.8, 121.8, 120.6, 120.3, 119.1, 118.5, 114.1, 114.0, 73.6, 67.2, 34.0, 33.5, 31.4, 21.4; IR (ATR) 3279, 2953, 1769, 1239, 827 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{35}H_{34}Br^{79}Br^{81}NO_3$ 676.0879; Found 676.0875.

1-(2,3-Bis(3-bromophenyl)-1*H*-indol-7-yl)-3-(*tert*-butoxy)propan-2-yl acetate (4f)



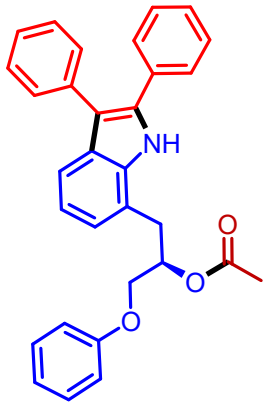
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 71% (425 mg); mp 65-67 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.32 (1H, s), 7.74 (1H, t, J = 1.8 Hz), 7.65 (1H, t, J = 1.8 Hz), 7.55 (1H, d, J = 8.5 Hz), 7.46 – 7.40 (3H, m), 7.35 – 7.31 (1H, m), 7.27 – 7.25 (1H, m), 7.20 (1H, t, J = 7.9 Hz), 7.11 – 7.08 (1H, m), 7.04 (1H, d, J = 7.1 Hz), 5.09 – 5.04 (1H, m), 3.50 (2H, dd, J = 4.3, 1.8 Hz), 3.27 (1H, dd, J = 13.7, 2.8 Hz), 3.20 (1H, dd, J = 13.7, 9.9 Hz), 2.20 (3H, s), 1.17 (9H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.3, 137.3, 135.8, 134.4, 133.0, 132.7, 130.8, 130.6, 130.1, 130.1, 129.3, 128.8, 128.3, 126.9, 124.1, 122.7, 122.5, 120.6, 119.9, 118.3, 114.2, 74.4, 73.8, 61.1, 33.2, 27.4, 21.4; IR (ATR) 3311, 2972, 1769, 1244, 783 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{29}H_{30}Br^{79}Br^{81}NO_3$ 600.0566; Found 600.0568.

1-(3-Methyl-2-phenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (4h)



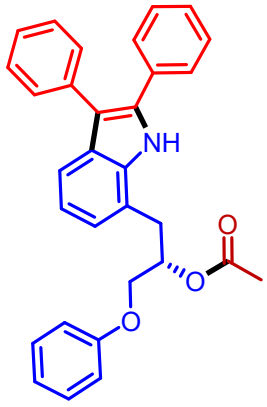
The title compound was prepared according to the general procedure. The product was obtained as a viscous liquid. Yield: 70% (280 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.60 (1H, s), 7.57 (2H, dd, J = 8.0, 1.8 Hz), 7.44 (1H, d, J = 8.0 Hz), 7.39 (2H, t, J = 7.7 Hz), 7.26 (1H, t, J = 7.5 Hz), 7.16 (2H, d, J = 8.1 Hz), 6.96 (1H, t, J = 7.5 Hz), 6.89 – 6.85 (2H, m), 6.78 (2H, d, J = 7.6 Hz), 5.27 – 5.25 (1H, m), 4.03 (1H, dd, J = 10.5, 5.6 Hz), 3.98 (1H, dd, J = 10.6, 2.5 Hz), 3.31 (1H, d, J = 13.8 Hz), 3.12 (1H, dd, J = 13.8, 10.6 Hz), 2.42 (3H, s), 2.12 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.2, 158.3, 135.1, 134.2, 133.3, 130.0, 129.4, 128.7, 127.5, 127.0, 123.0, 121.2, 119.4, 118.6, 118.1, 114.6, 108.5, 73.4, 67.1, 33.5, 21.4, 9.9; IR (ATR) 3330, 2929, 1729, 1455, 1227, 752, 691 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{26}H_{26}NO_3$ 400.1907; Found 400.1907.

(*R*)-1-(2,3-Diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (7)



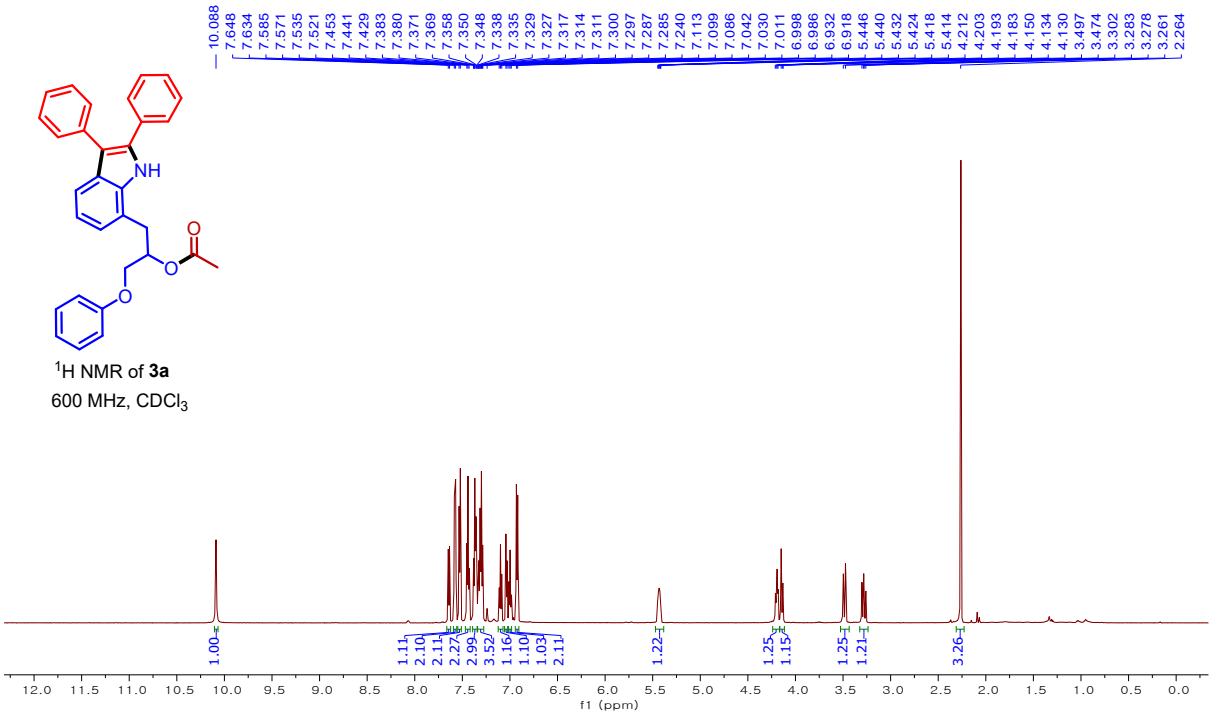
The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 86% (398 mg); mp 94-96 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.98 (1H, s), 7.49 (1H, d, *J* = 8.1 Hz), 7.44-7.35 (4H, m), 7.29 (2H, t, *J* = 7.5 Hz), 7.20-7.13 (6H , m), 6.94 (1H, dt, *J* = 7.8, 5.1 Hz), 6.89-6.84 (2H , m), 6.78 (2H, dd, *J* = 8.4, 2.7 Hz), 5.31-5.25 (1H, m), 4.10 – 3.92 (2H, m), 3.34 (1H, d, *J* = 13.8 Hz), 3.19 – 3.07 (1H, m), 2.13 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) *δ* 172.4, 158.3, 135.3, 135.2, 134.3, 132.5, 130.2, 129.4, 128.9, 128.6, 128.5, 127.9, 127.4, 126.1, 123.4, 121.2, 120.2, 118.9, 118.8, 114.9, 114.5, 73.4, 67.0, 33.5, 21.4; IR (ATR) 3330, 2999, 1765, 1240, 744 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{28}NO_3$ 462.2063; Found 462.2059.

(*S*)-1-(2,3-Diphenyl-1*H*-indol-7-yl)-3-phenoxypropan-2-yl acetate (8)

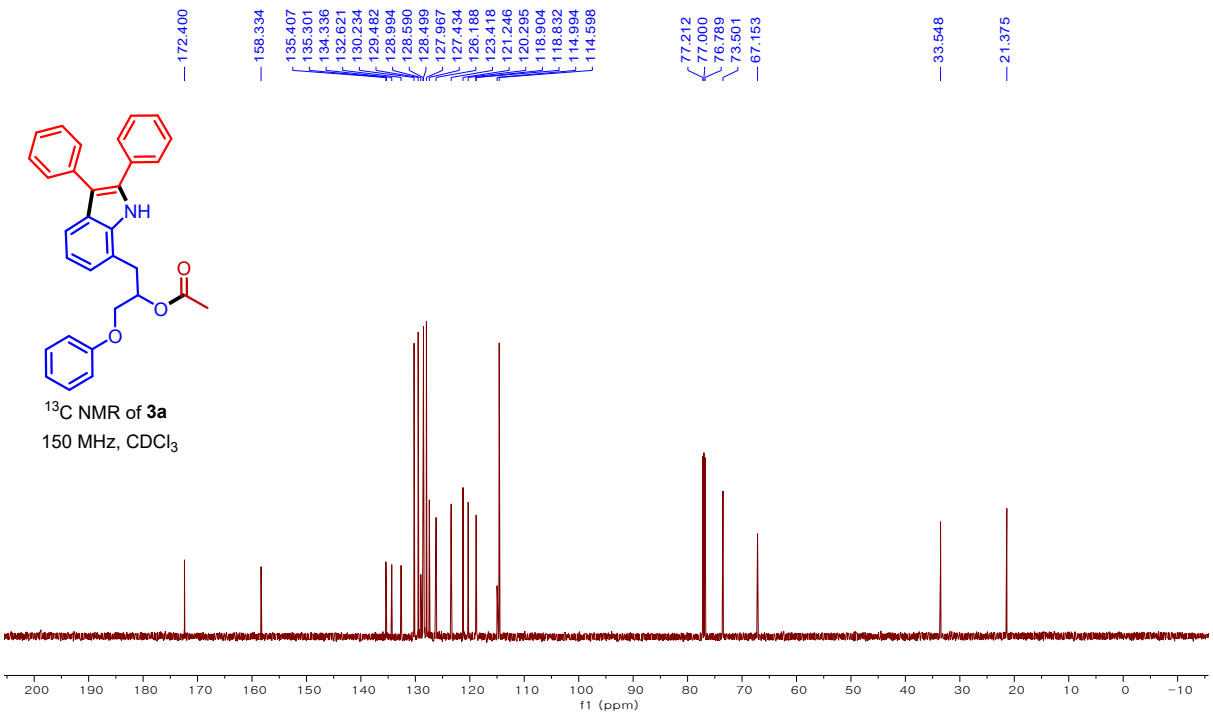


The title compound was prepared according to the general procedure. The product was obtained as a white solid. Yield: 85% (392 mg); mp 94-96 °C. ^{1}H NMR (600 MHz, $CDCl_3$) *δ* 9.97 (1H, s), 7.49 (1H, d, *J* = 7.6 Hz), 7.45 – 7.35 (4H, m), 7.33 – 7.27 (2H, m), 7.25 – 7.12 (6H, m), 7.00 – 6.85 (3H, m), 6.78 (2H, d, *J* = 8.0 Hz), 5.35 – 5.20 (1H, m), 4.12 – 3.95 (2H, m), 3.35 (1H, d, *J* = 13.6 Hz), 3.14 (1H, t, *J* = 12.2 Hz), 2.13 (3H, s); ^{13}C NMR (150 MHz, $CDCl_3$) *δ* 172.4, 158.3, 135.3, 135.2, 134.3, 132.6, 130.2, 129.5, 128.9, 128.6, 128.5, 127.9, 127.4, 126.1, 123.4, 121.2, 120.2, 118.9, 118.8, 114.9, 114.5, 73.5, 67.1, 33.5, 21.4; IR (ATR) 3333, 2996, 1769, 1242, 747 cm^{-1}; HRMS (EI) m/z: [M+H] Calcd for $C_{31}H_{28}NO_3$ 462.2063; Found 462.2060.

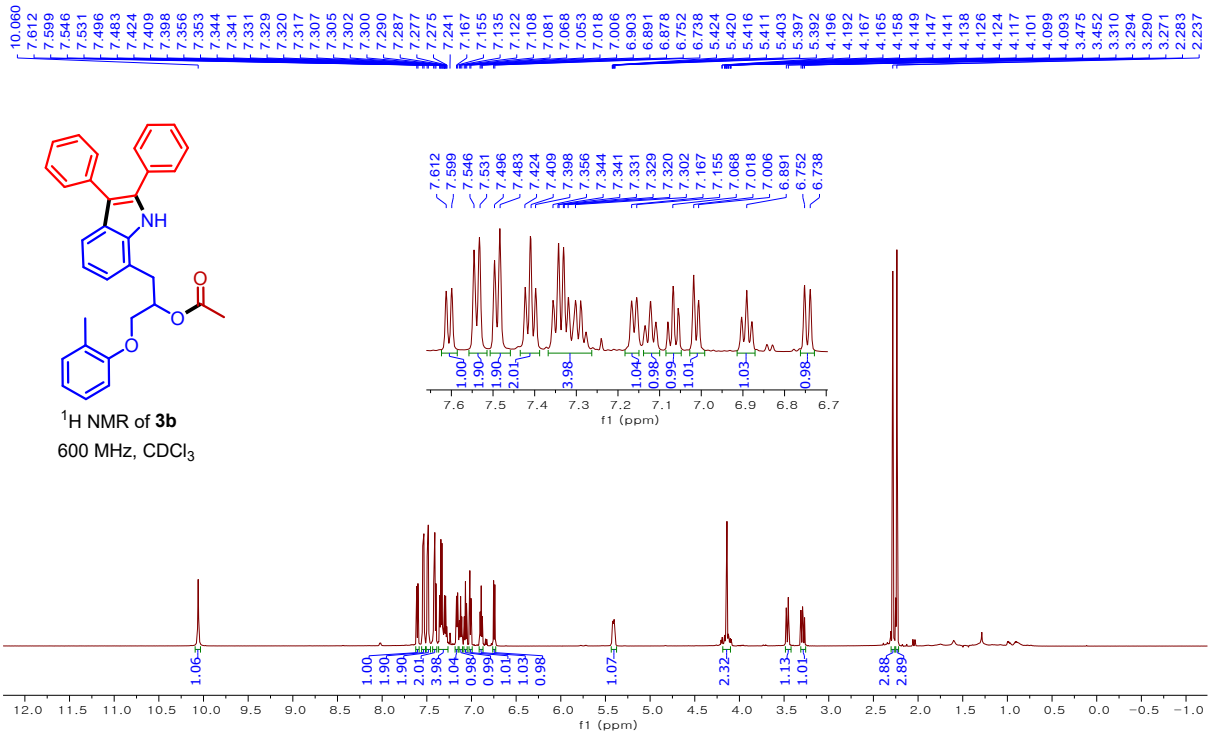
^{1}H NMR and ^{13}C NMR spectra of synthesized compounds

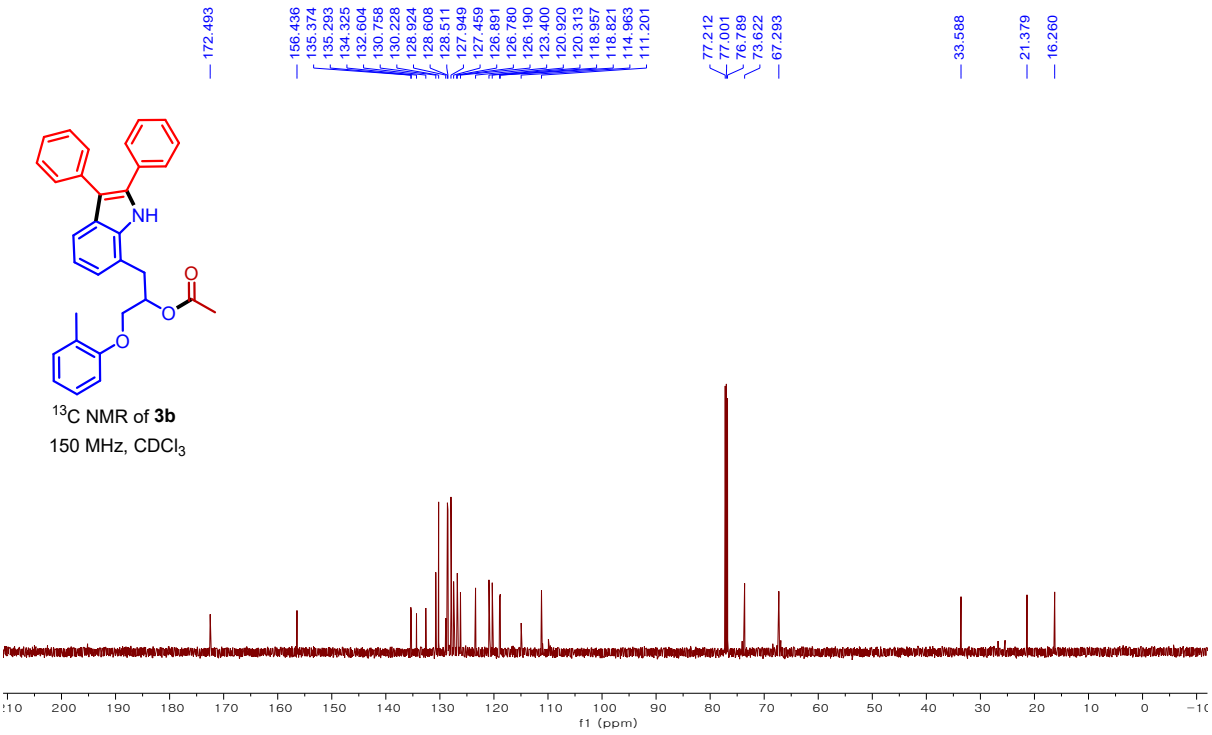


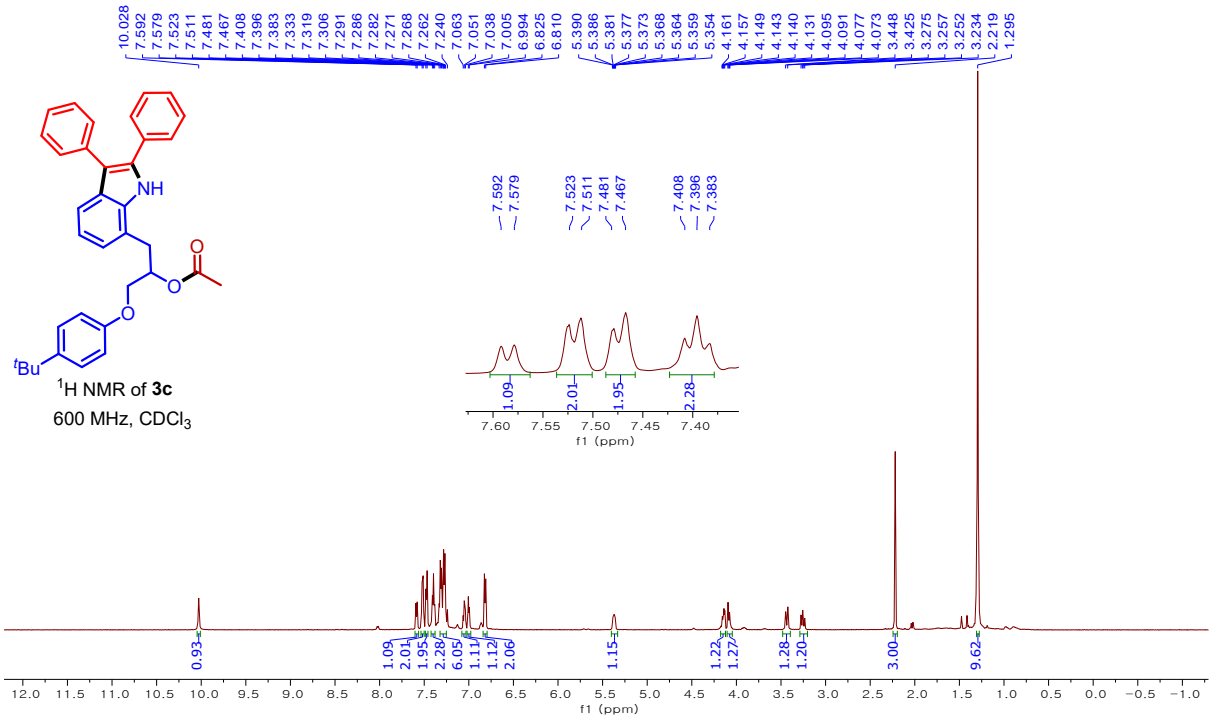
^{1}H NMR of **3a**
600 MHz, $CDCl_3$

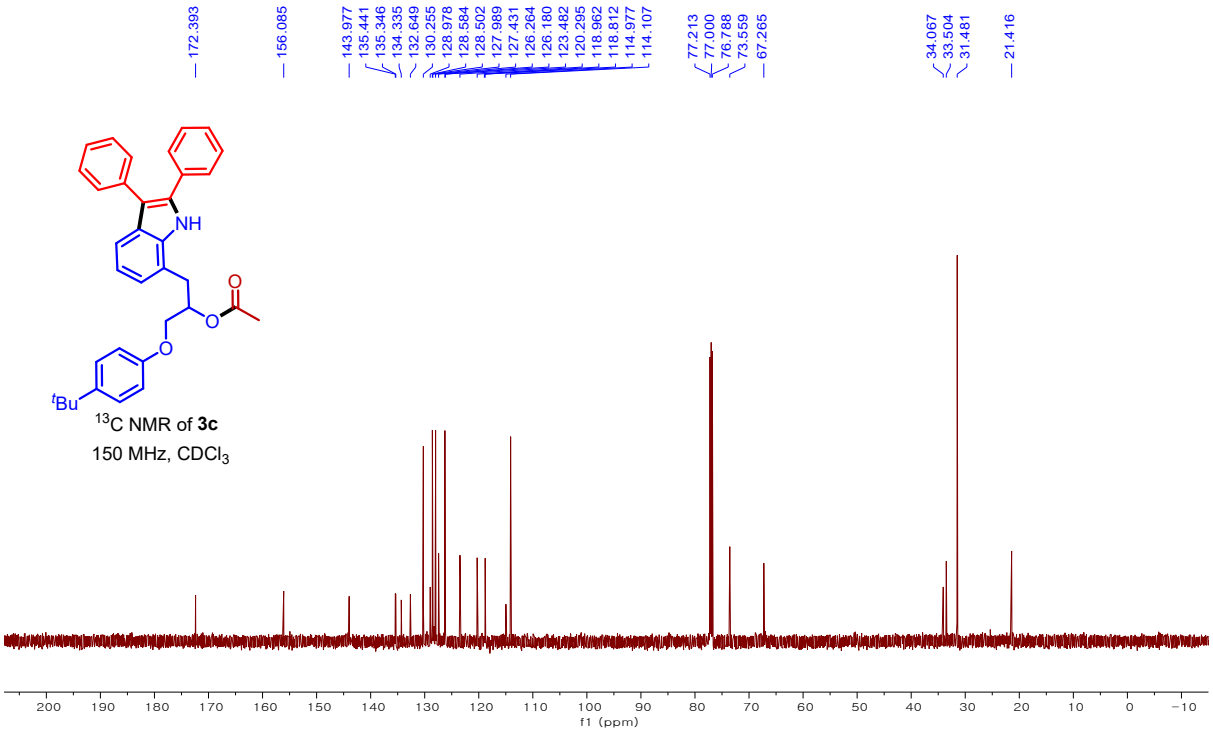


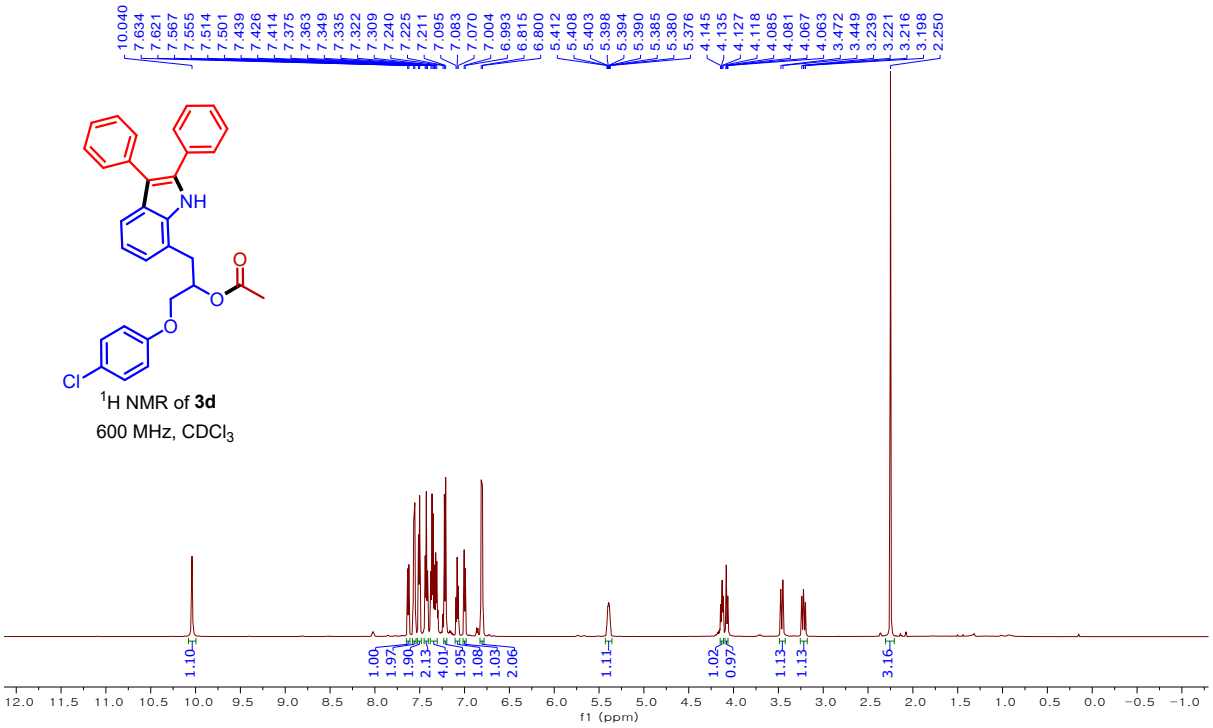
^{13}C NMR of **3a**
150 MHz, $CDCl_3$


NH
O
O
O
1H NMR of 3b
600 MHz, CDCl3
f1 (ppm)

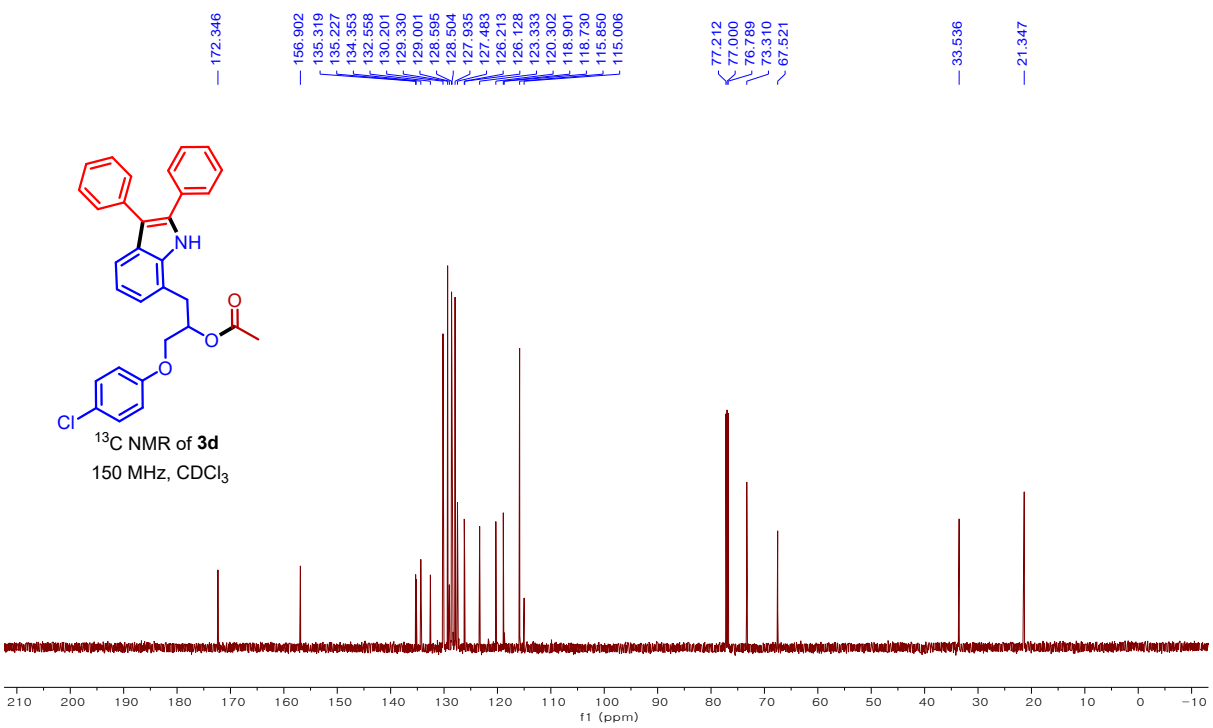

NH
O
O
O
13C NMR of 3b
150 MHz, CDCl3
f1 (ppm)


NH
O
O
O
tBu
1H NMR of 3c
600 MHz, CDCl3
f1 (ppm)

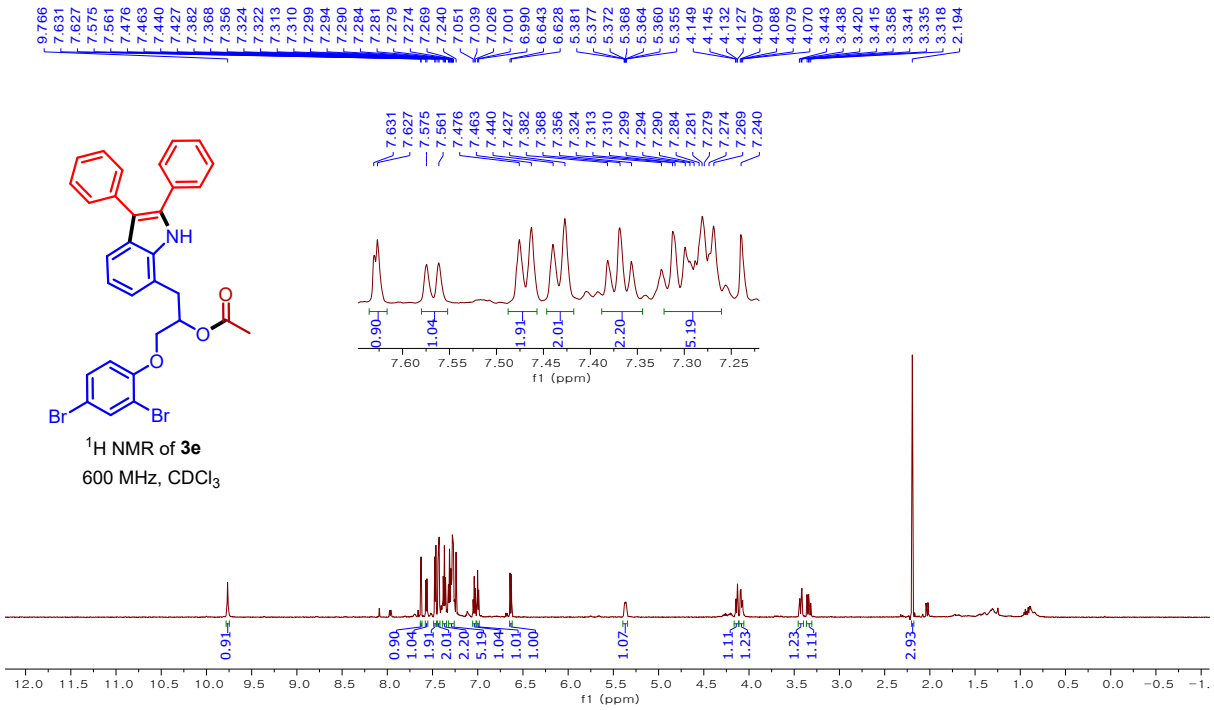

NH
O
O
O
tBu
13C NMR of 3c
150 MHz, CDCl3
f1 (ppm)



^{1}H NMR of **3d**
600 MHz, $CDCl_3$

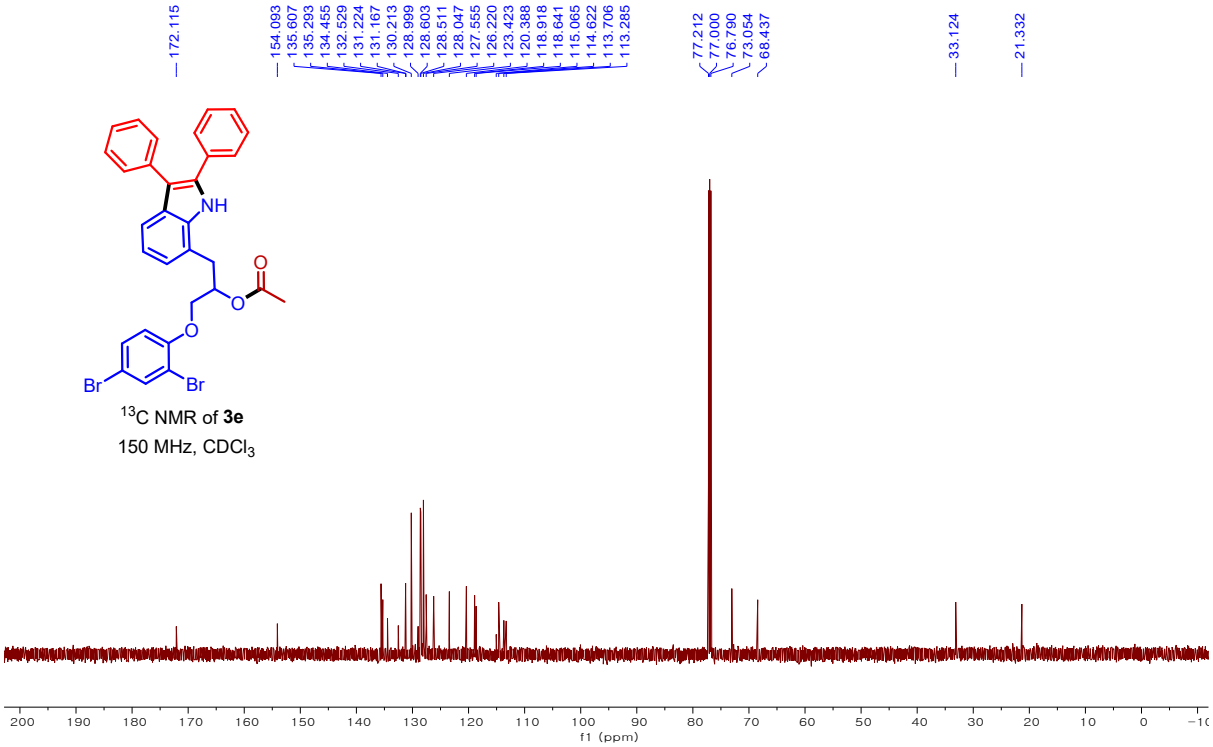


^{13}C NMR of **3d**
150 MHz, $CDCl_3$



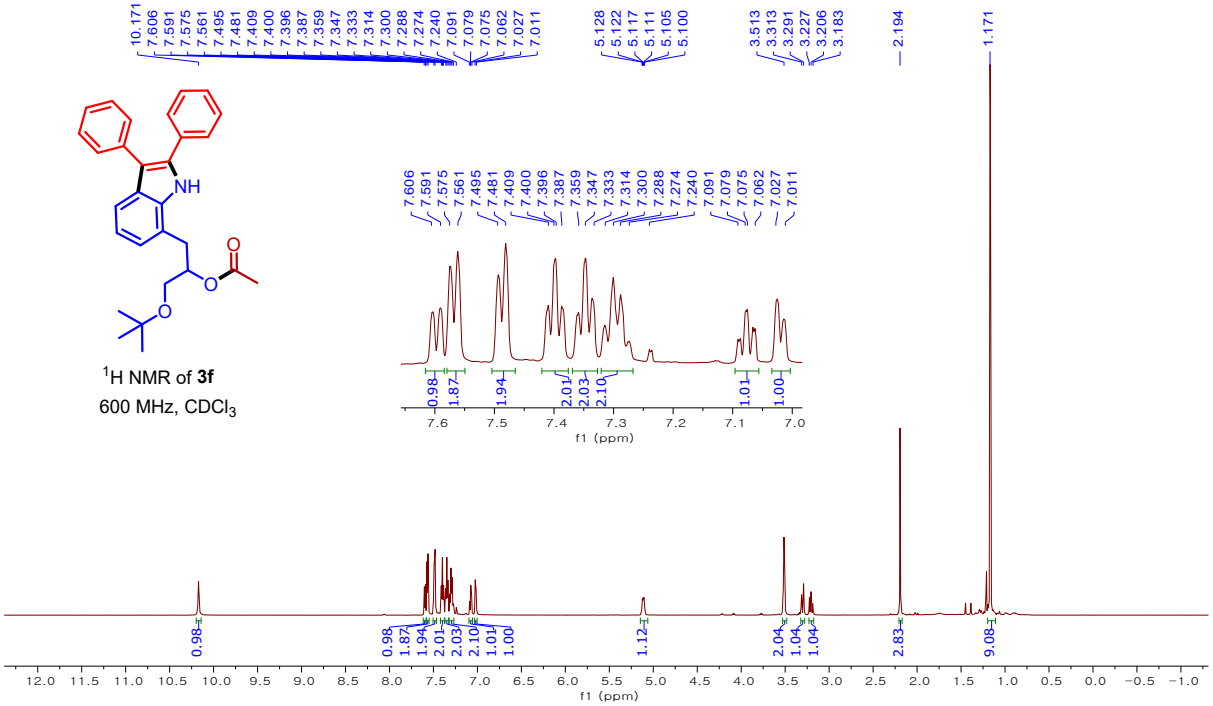
^{1}H NMR of **3e**

600 MHz, $CDCl_3$



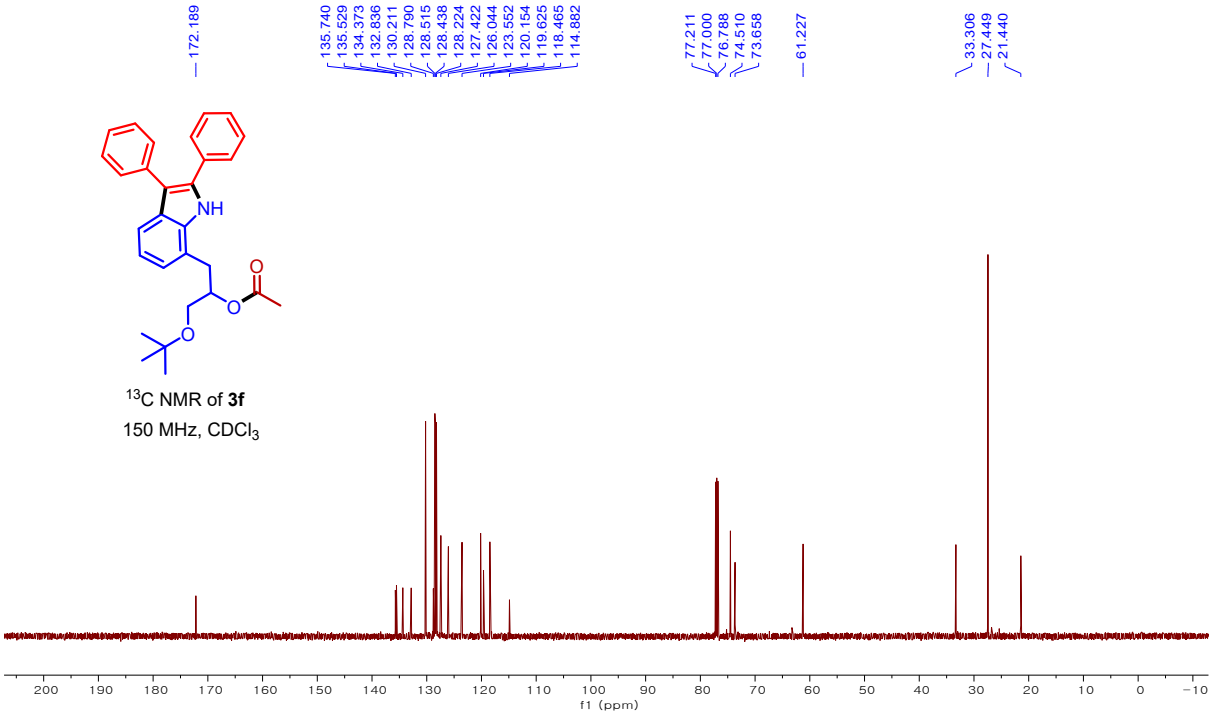
^{13}C NMR of **3e**

150 MHz, $CDCl_3$



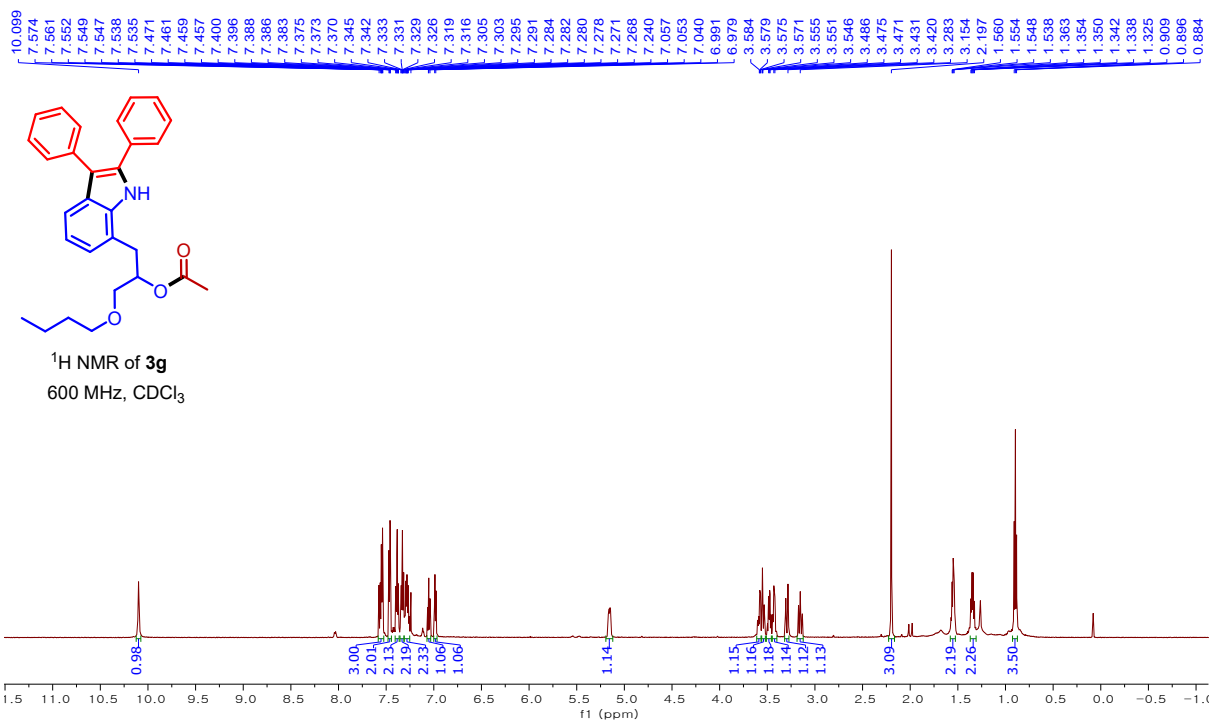
^{1}H NMR of **3f**

600 MHz, $CDCl_3$



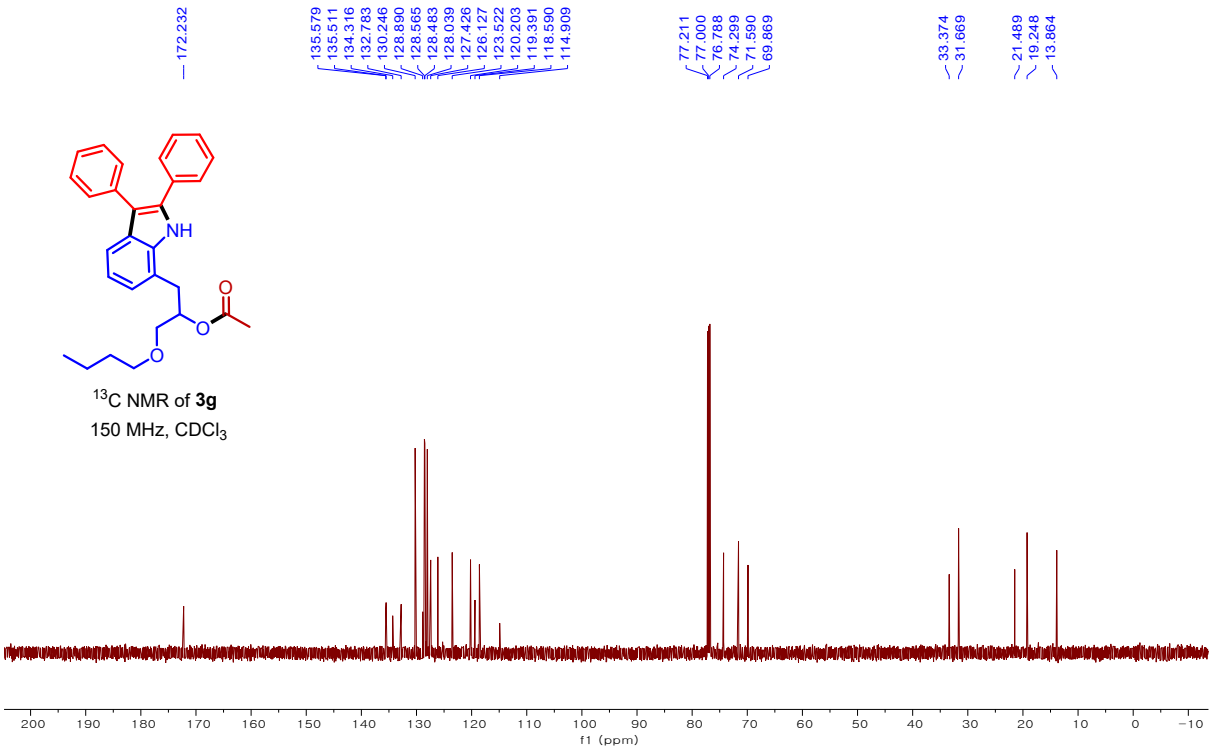
^{13}C NMR of **3f**

150 MHz, $CDCl_3$

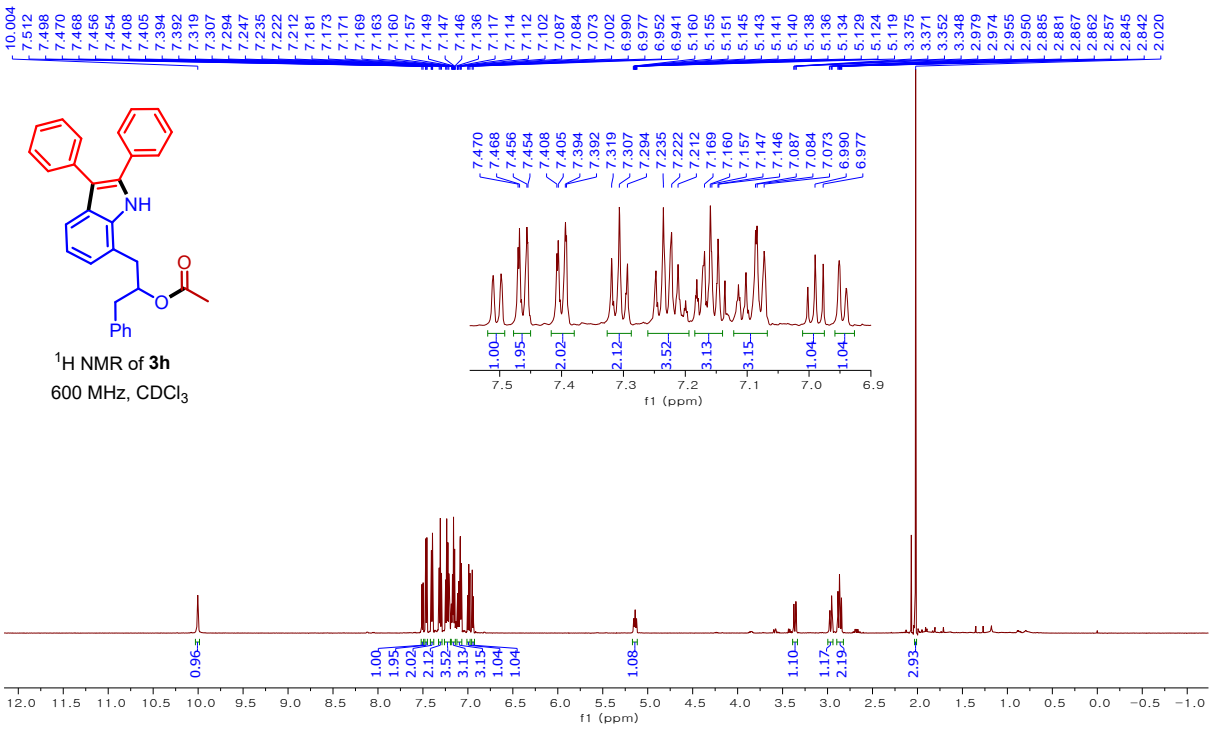


^{1}H NMR of **3g**

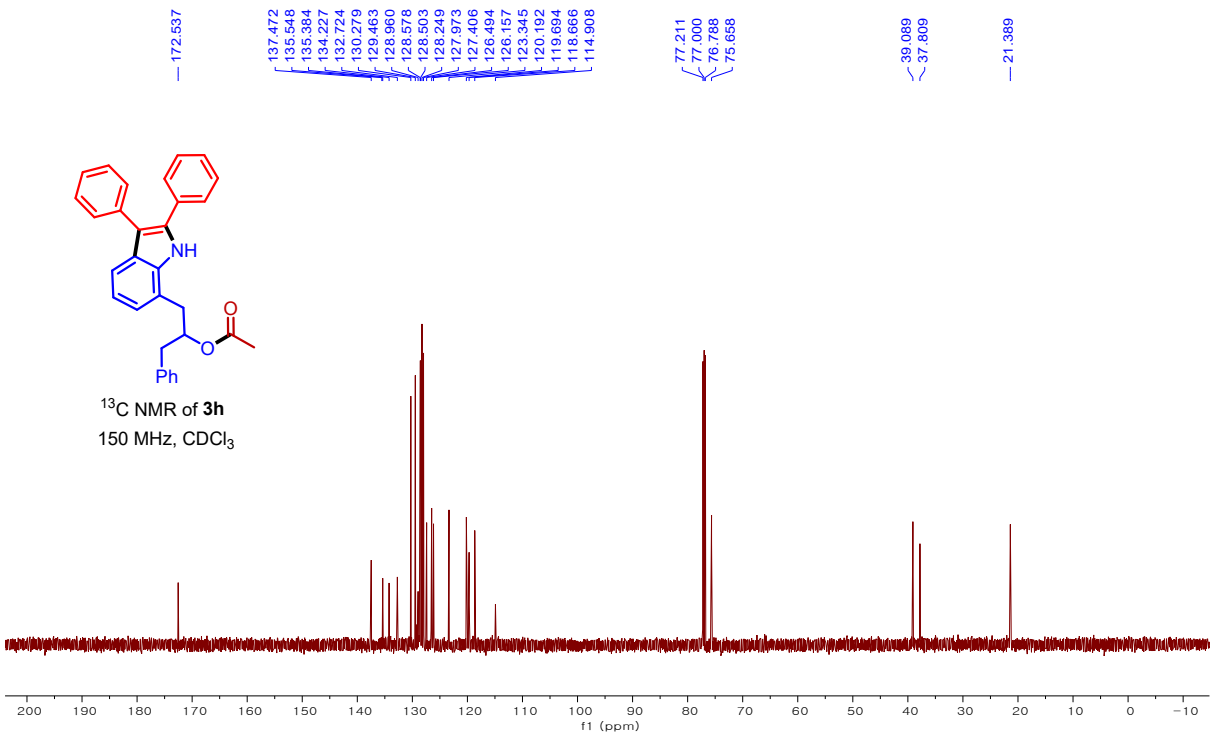
600 MHz, $CDCl_3$



^{13}C NMR of **3g**
150 MHz, $CDCl_3$

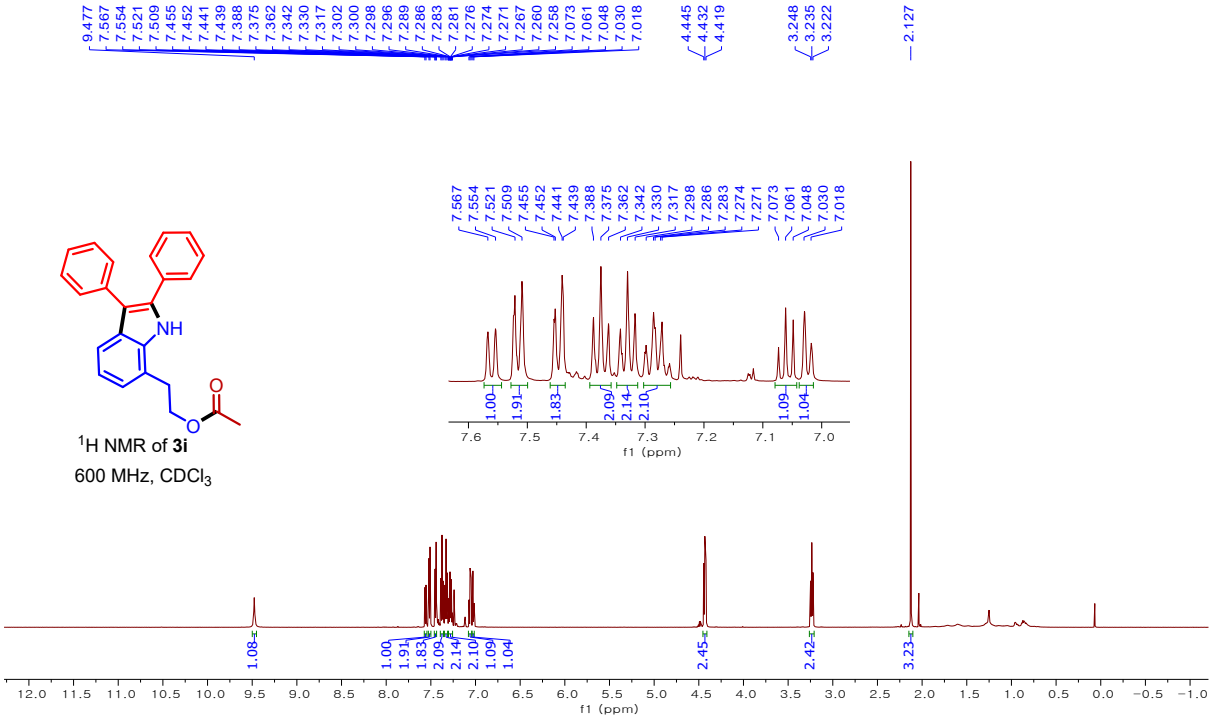


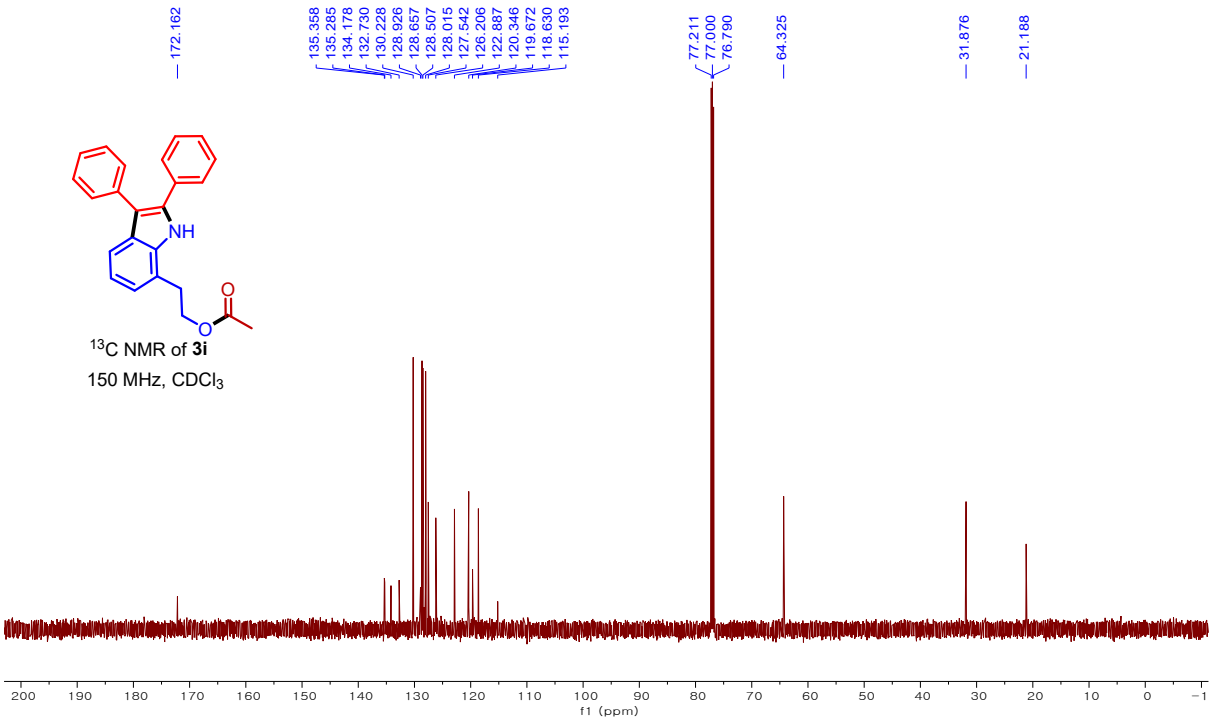
^{1}H NMR of **3h**
600 MHz, $CDCl_3$

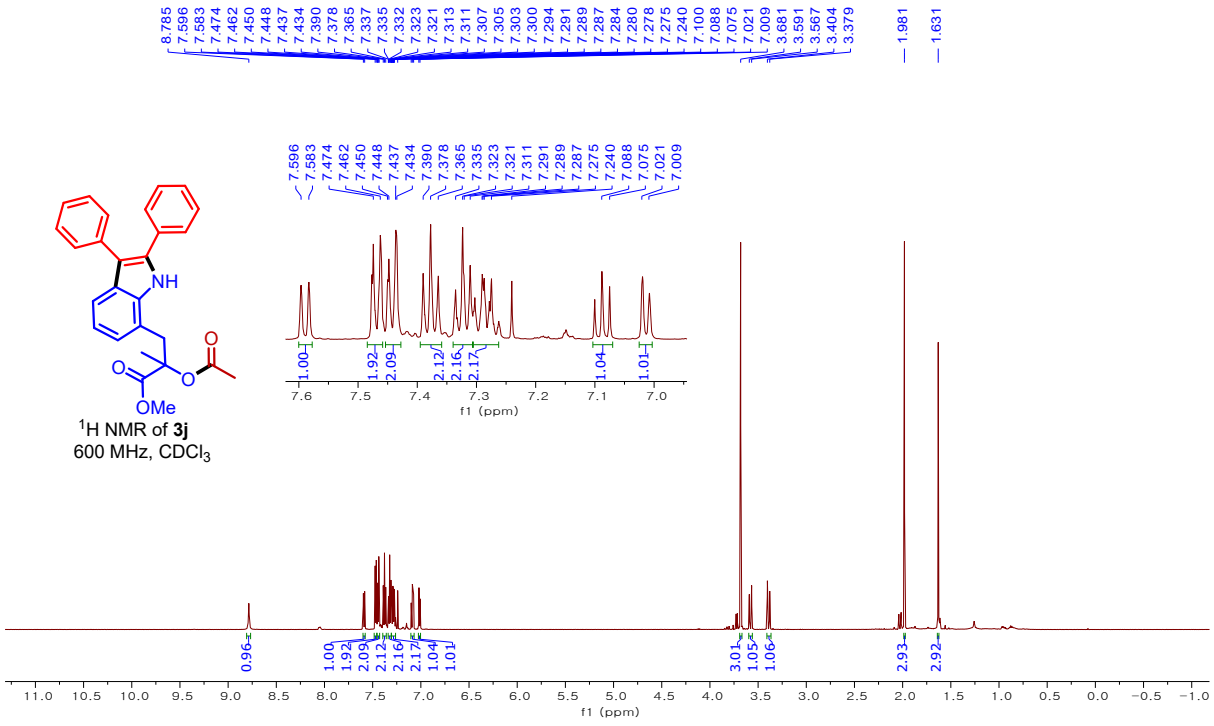


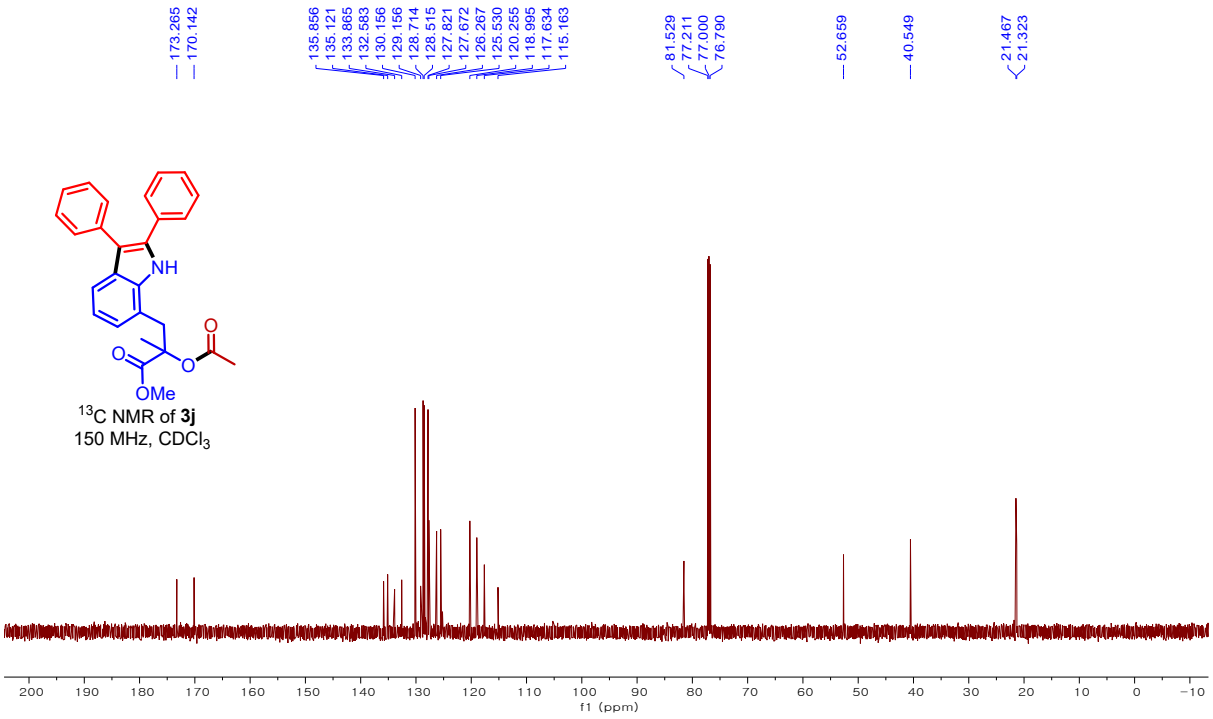
^{13}C NMR of **3h**

150 MHz, $CDCl_3$

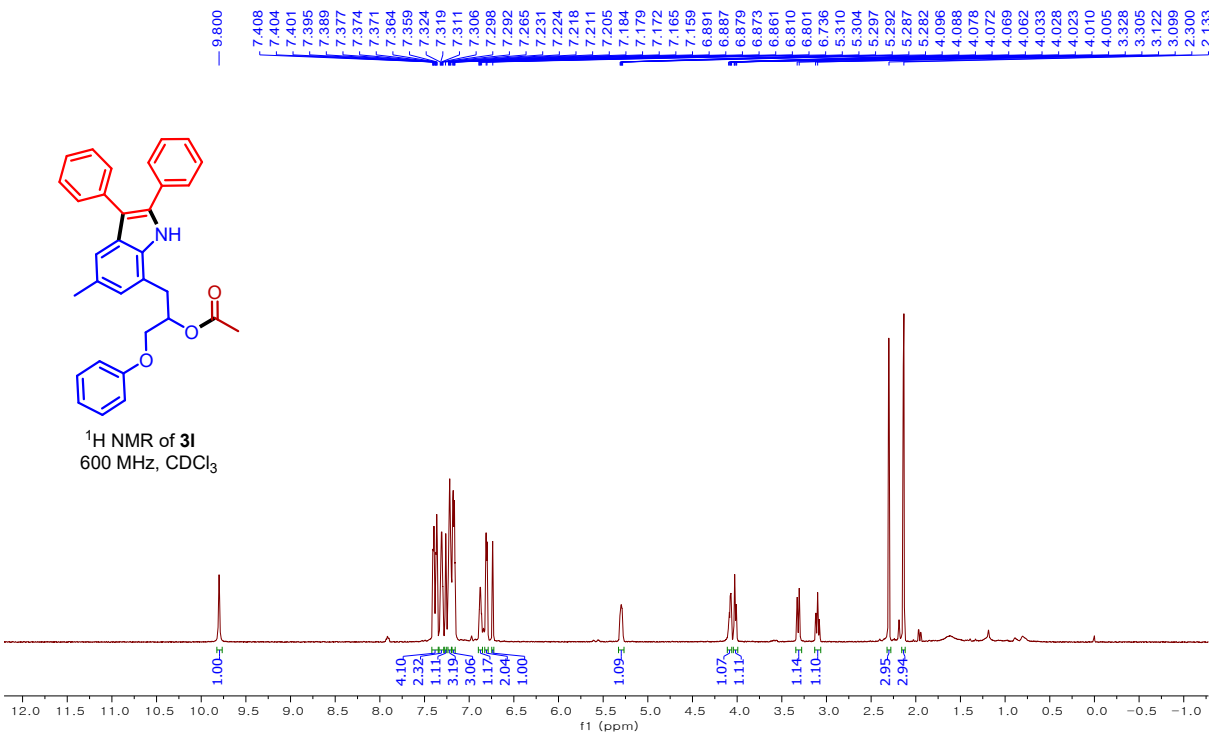

9.477
7.567
7.554
7.521
7.509
7.455
7.452
7.441
7.439
7.388
7.375
7.362
7.342
7.330
7.317
7.302
7.300
7.298
7.296
7.289
7.286
7.283
7.281
7.276
7.274
7.271
7.267
7.260
7.258
7.073
7.061
7.048
7.030
7.018
4.445
4.432
4.419
3.248
3.235
3.222
2.127
NH
O
O
1H NMR of 3i
600 MHz, CDCl3
1.08
1.00
1.91
1.83
2.09
2.14
2.10
1.09
1.04
2.45
2.42
3.23
f1 (ppm)


172.162
135.358
135.285
134.178
132.730
130.228
128.926
128.657
128.507
128.015
127.542
126.206
122.887
120.346
119.672
118.630
115.193
77.211
77.000
76.790
64.325
31.876
21.188
NH
O
O
13C NMR of 3i
150 MHz, CDCl3
f1 (ppm)

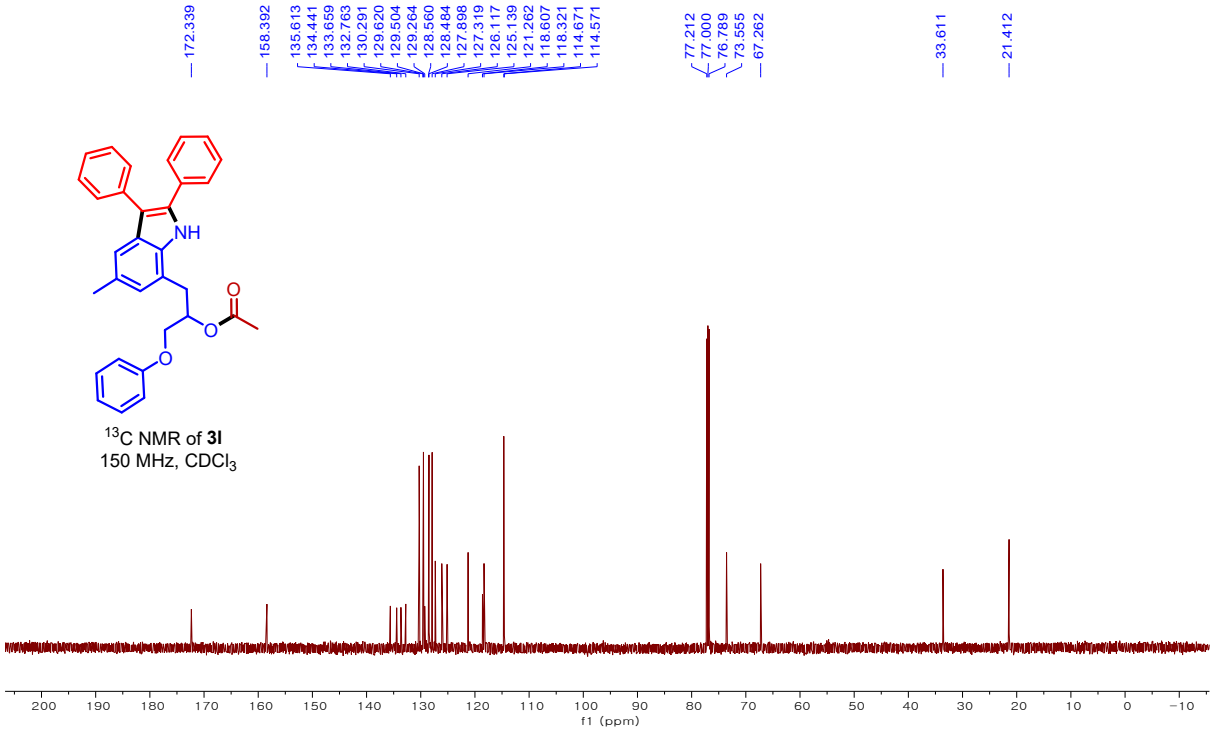

8.785
7.596
7.583
7.474
7.462
7.450
7.448
7.437
7.434
7.390
7.378
7.365
7.337
7.335
7.332
7.323
7.321
7.313
7.311
7.307
7.305
7.303
7.300
7.294
7.291
7.289
7.287
7.284
7.280
7.278
7.275
7.240
7.100
7.088
7.075
7.021
7.009
3.681
3.591
3.567
3.404
3.379
1.981
1.631
NH
O
O
O
OMe
1H NMR of 3j
600 MHz, CDCl3
f1 (ppm)
f1 (ppm)



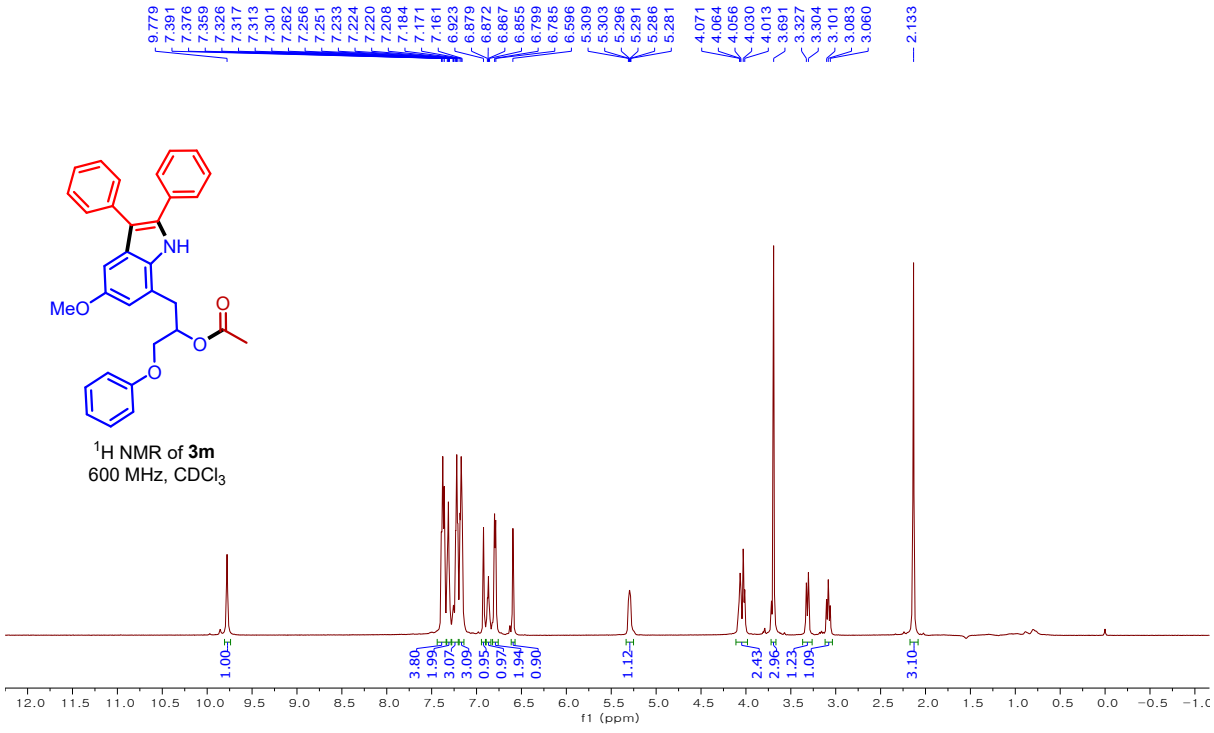
^{13}C NMR of **3j**
150 MHz, $CDCl_3$



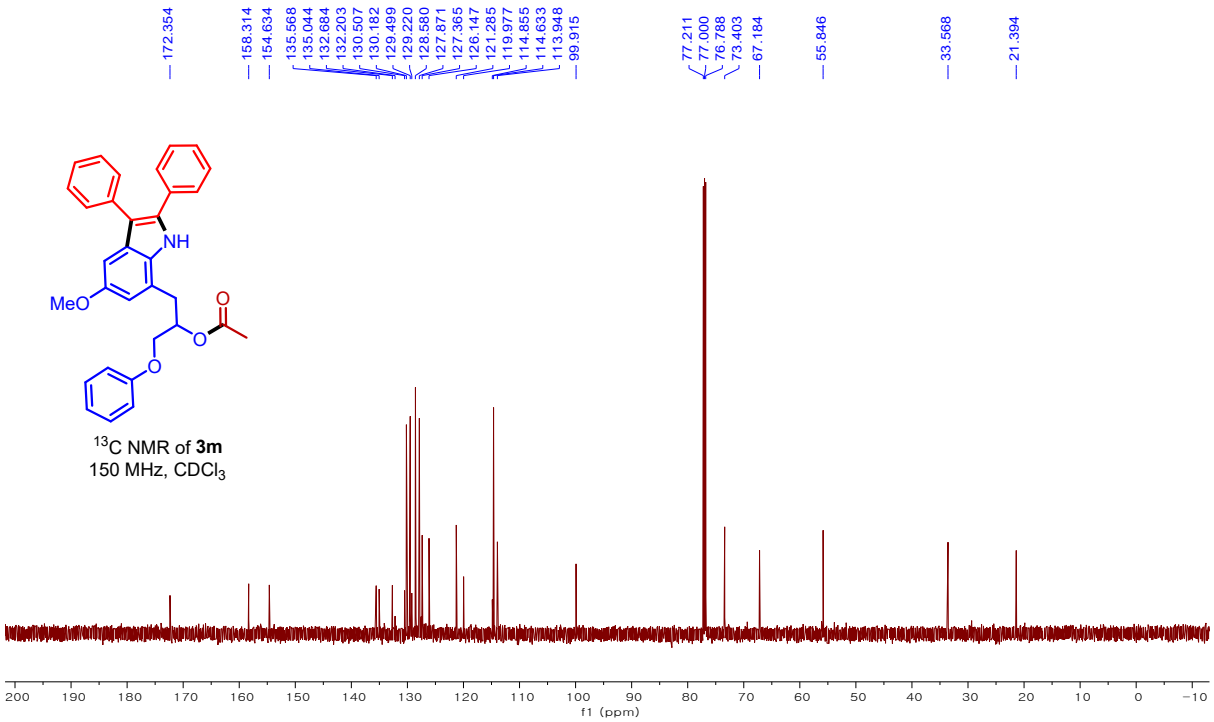
^{1}H NMR of **3l**
600 MHz, $CDCl_3$

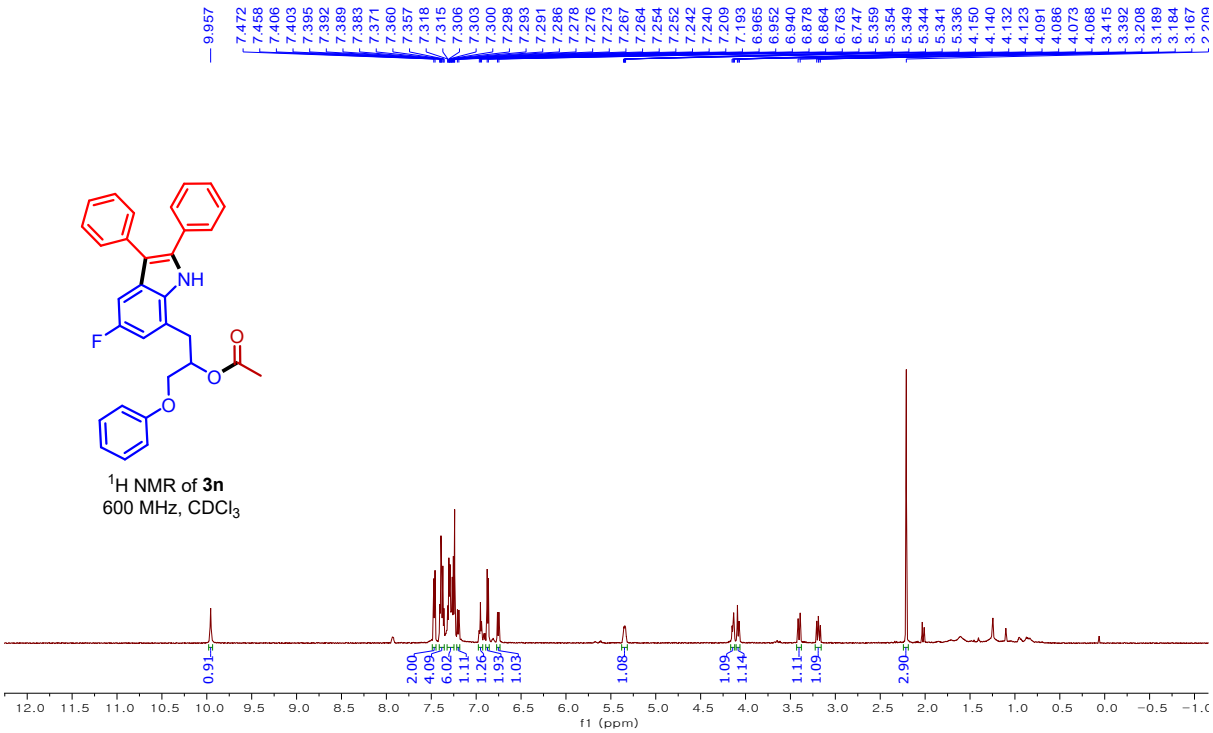


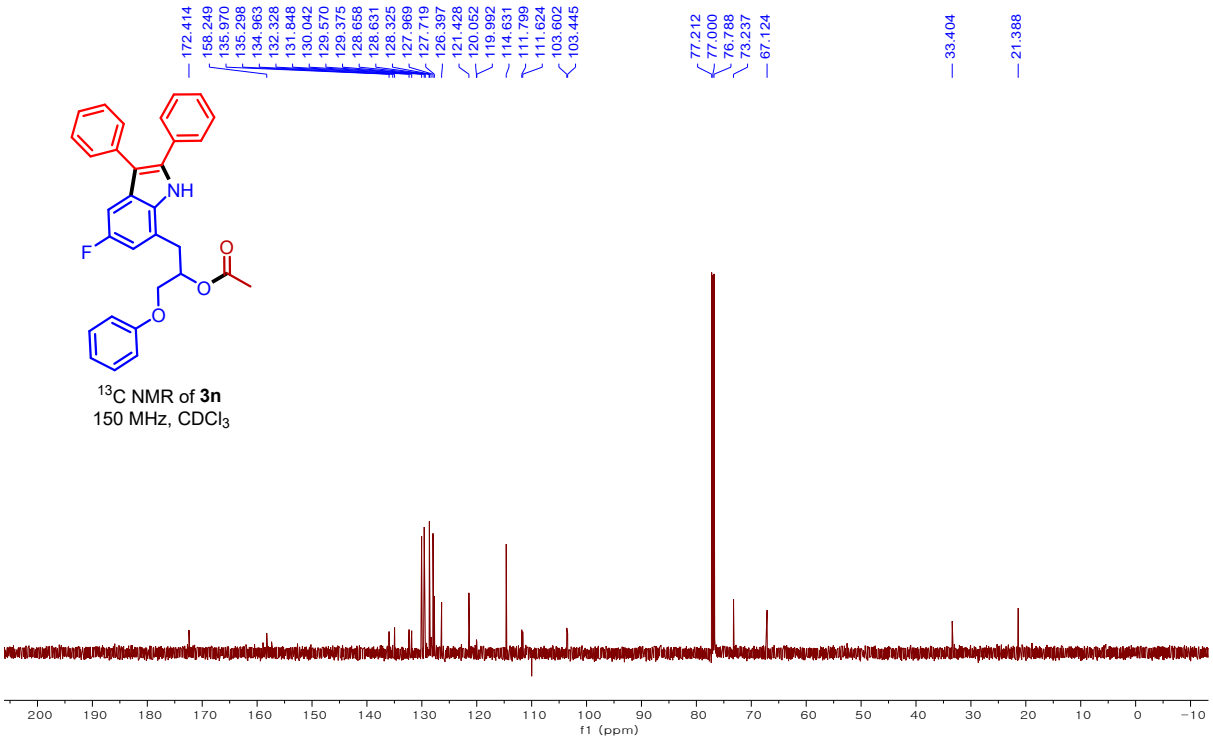
^{13}C NMR of **3l**
150 MHz, $CDCl_3$

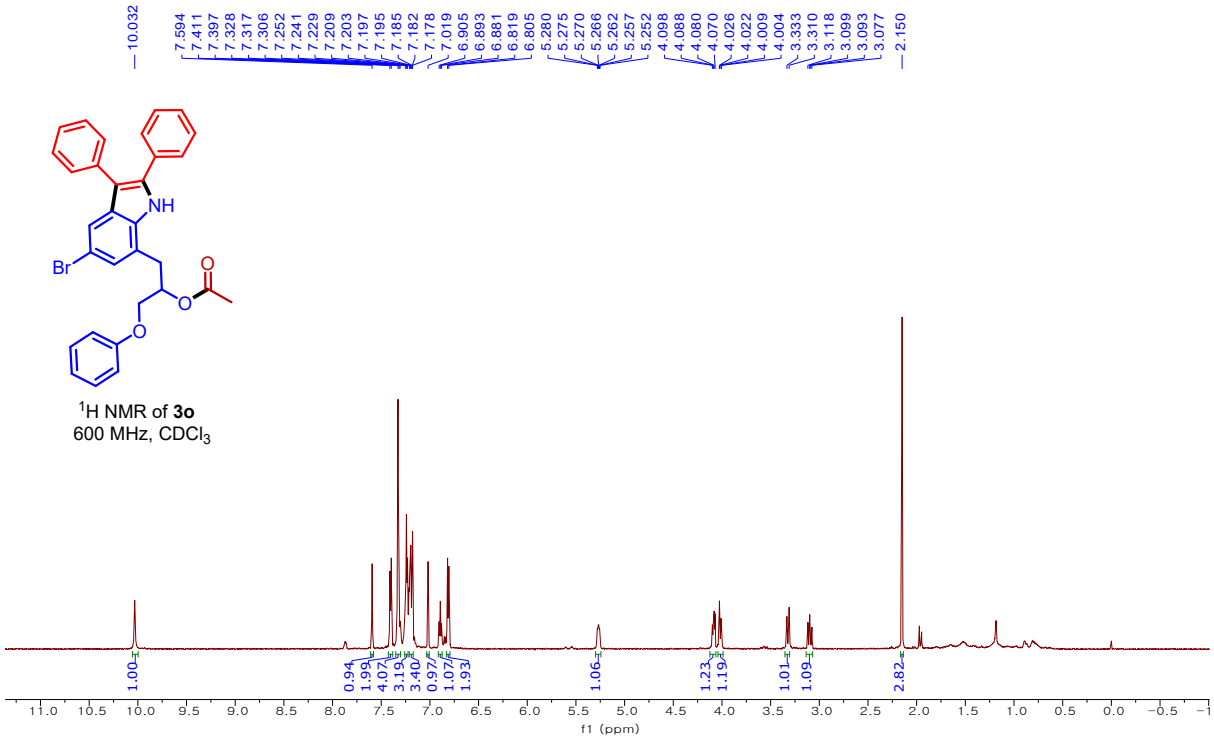


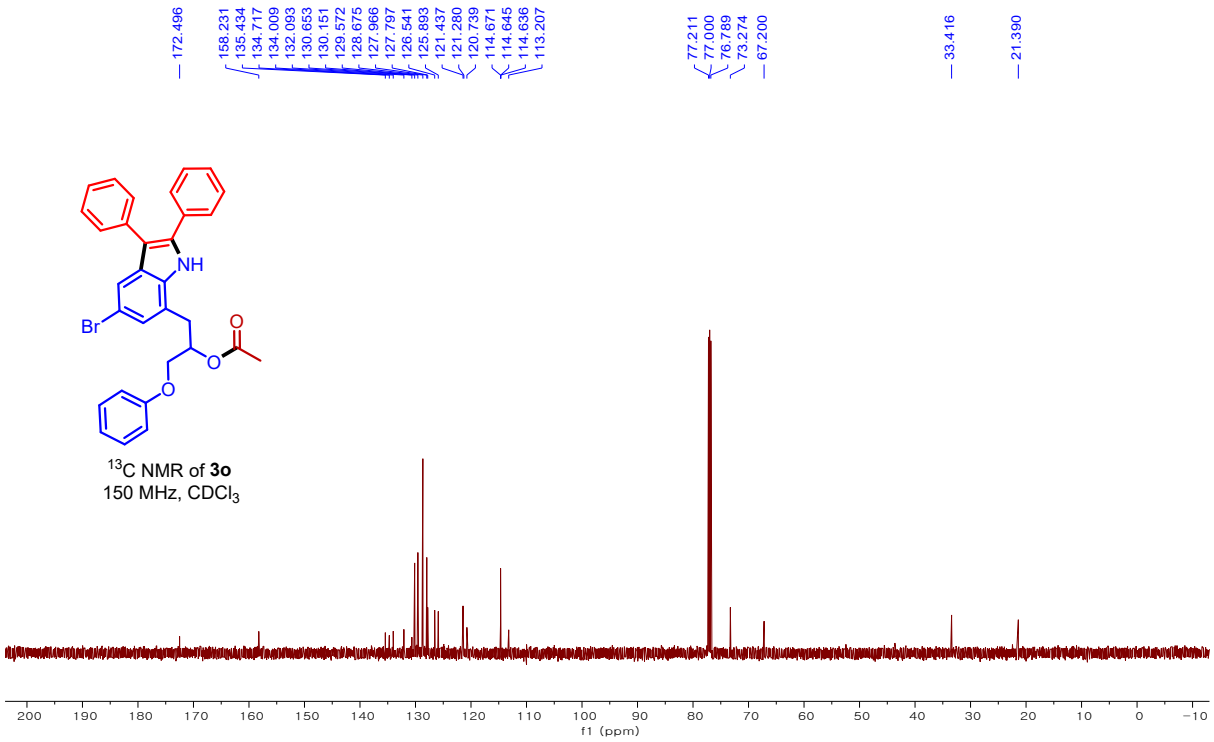
^{1}H NMR of **3m**
600 MHz, $CDCl_3$


172.354
158.314
154.634
135.568
135.044
132.684
132.203
130.507
130.182
129.499
129.220
128.580
127.871
127.365
126.147
121.285
119.977
114.855
114.633
113.948
99.915
77.211
77.000
76.788
73.403
67.184
55.846
33.568
21.394
NH
MeO
O
O
O
^{13}C NMR of **3m**
150 MHz, $CDCl_3$
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

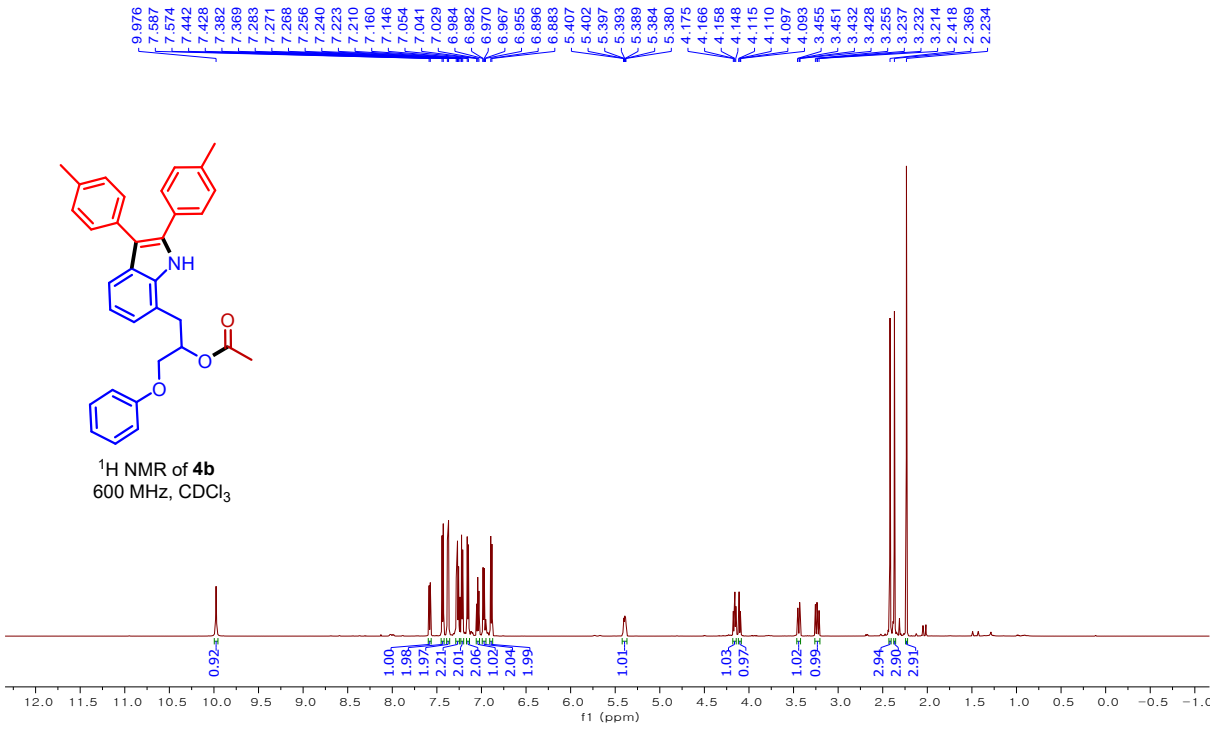

9.957
7.472
7.458
7.406
7.403
7.395
7.392
7.389
7.383
7.371
7.360
7.357
7.318
7.315
7.306
7.303
7.300
7.298
7.293
7.291
7.286
7.278
7.276
7.273
7.267
7.264
7.254
7.252
7.242
7.240
7.209
7.193
6.965
6.952
6.940
6.878
6.864
6.763
6.747
5.359
5.354
5.349
5.344
5.341
5.336
4.150
4.140
4.132
4.123
4.091
4.086
4.073
4.068
3.415
3.392
3.208
3.189
3.184
3.167
NH
F
O
O
O
1H NMR of 3n
600 MHz, CDCl3
0.91
2.00
4.09
6.02
1.11
1.26
1.93
1.03
1.08
1.09
1.14
1.11
1.09
2.90
f1 (ppm)


172.414
158.249
135.970
135.298
134.963
132.328
131.848
130.042
129.570
129.375
128.658
128.631
128.325
127.969
127.719
126.397
121.428
120.052
119.992
114.631
111.799
111.624
103.602
103.445
77.212
77.000
76.788
73.237
67.124
33.404
21.388
NH
F
O
O
O
13C NMR of 3n
150 MHz, CDCl3
f1 (ppm)

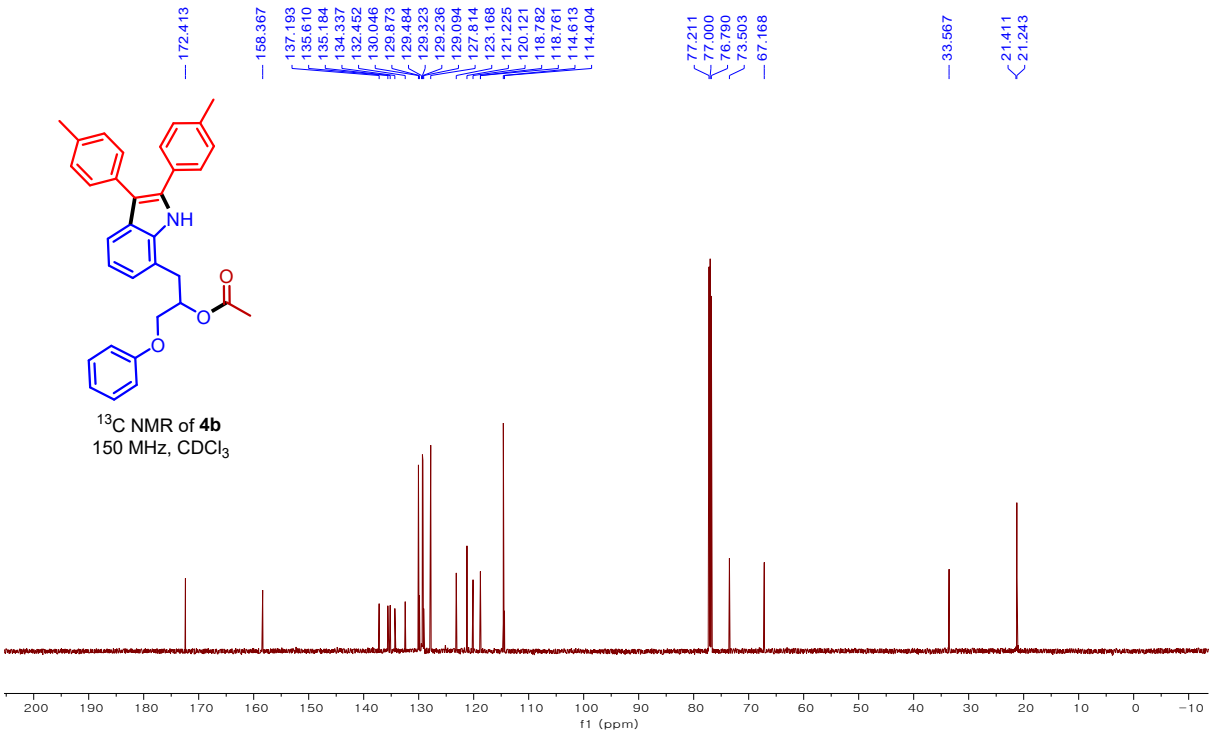

10.032
7.594
7.411
7.397
7.328
7.317
7.306
7.252
7.241
7.229
7.209
7.203
7.197
7.195
7.185
7.182
7.178
7.019
6.905
6.893
6.881
6.819
6.805
5.280
5.275
5.270
5.266
5.262
5.257
5.252
4.098
4.088
4.080
4.070
4.026
4.022
4.009
4.004
3.333
3.310
3.118
3.099
3.093
3.077
2.150
NH
Br
O
O
O
1H NMR of 3o
600 MHz, CDCl3
1.00
0.94
1.99
4.07
3.19
3.40
0.97
1.07
1.93
1.06
1.23
1.19
1.01
1.09
2.82
f1 (ppm)



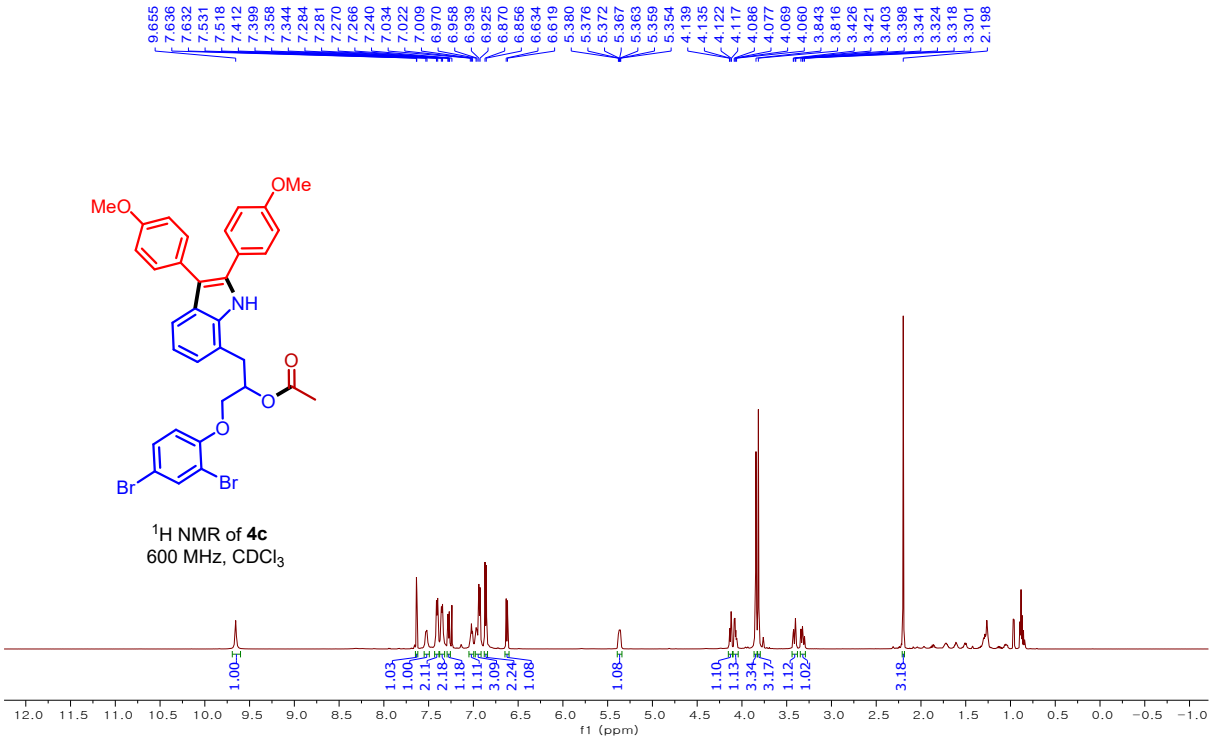
^{13}C NMR of **3o**
150 MHz, $CDCl_3$



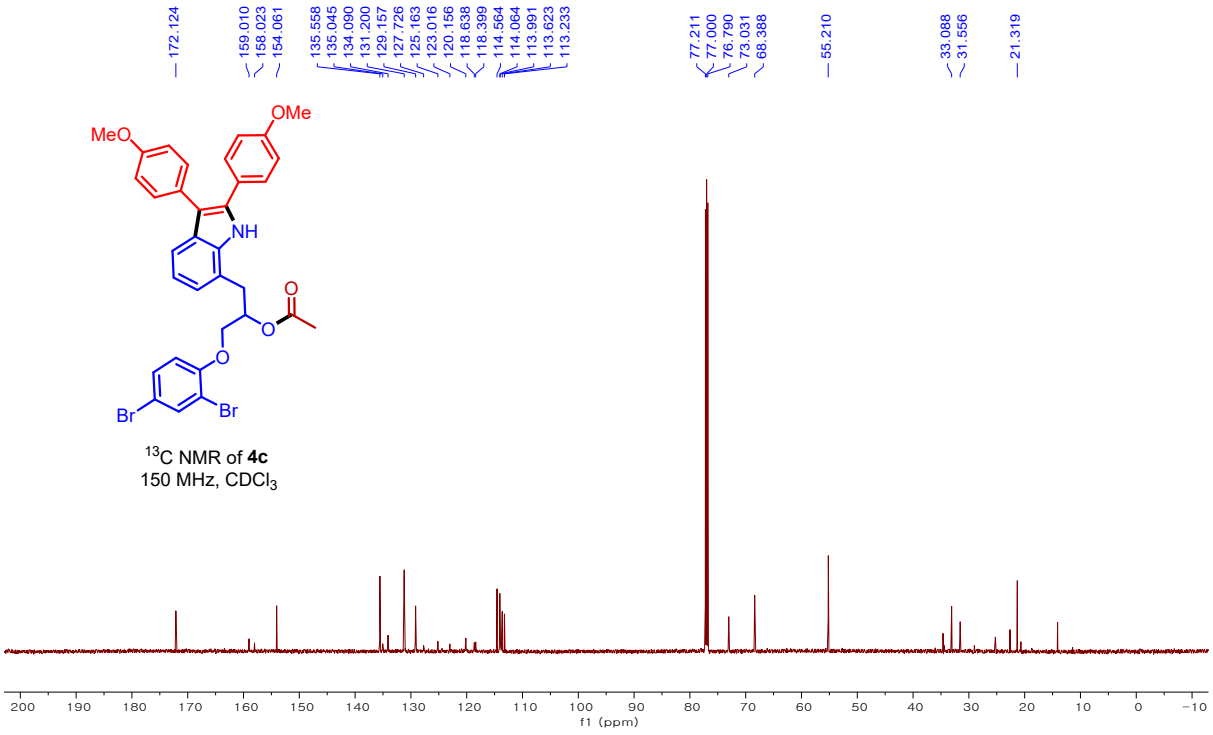
^{1}H NMR of **4b**
600 MHz, $CDCl_3$



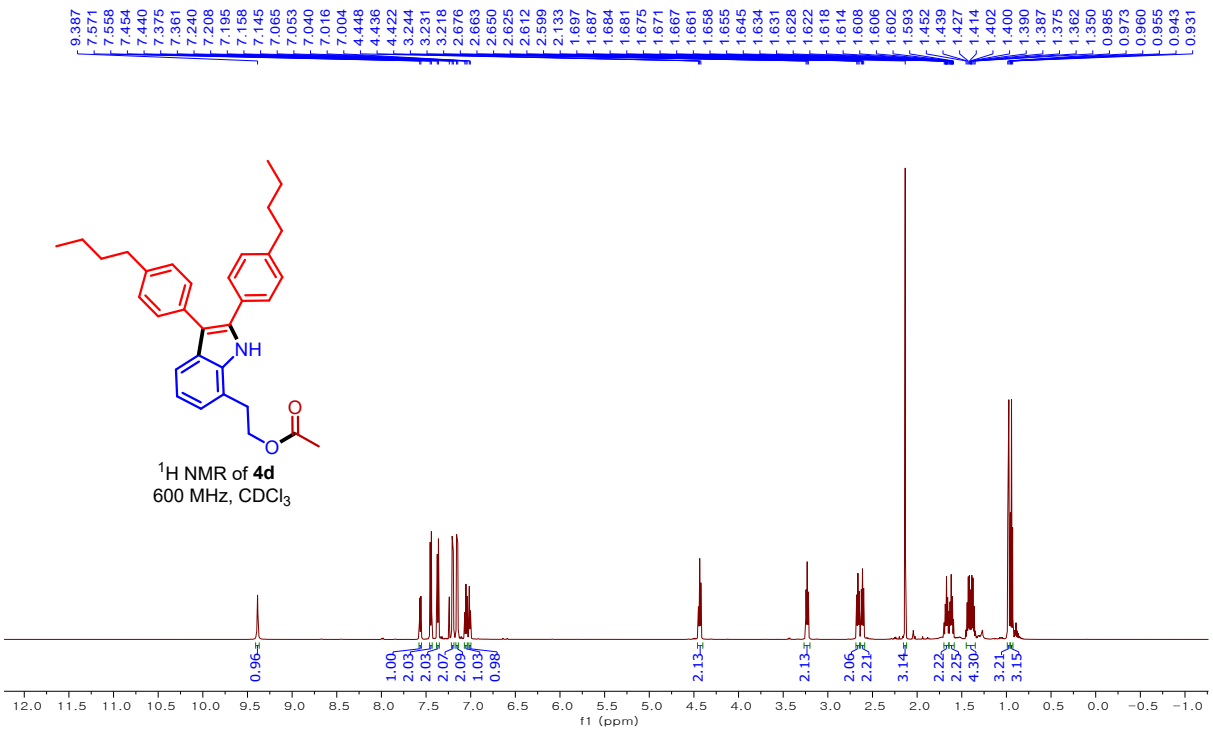
^{13}C NMR of **4b**
150 MHz, $CDCl_3$

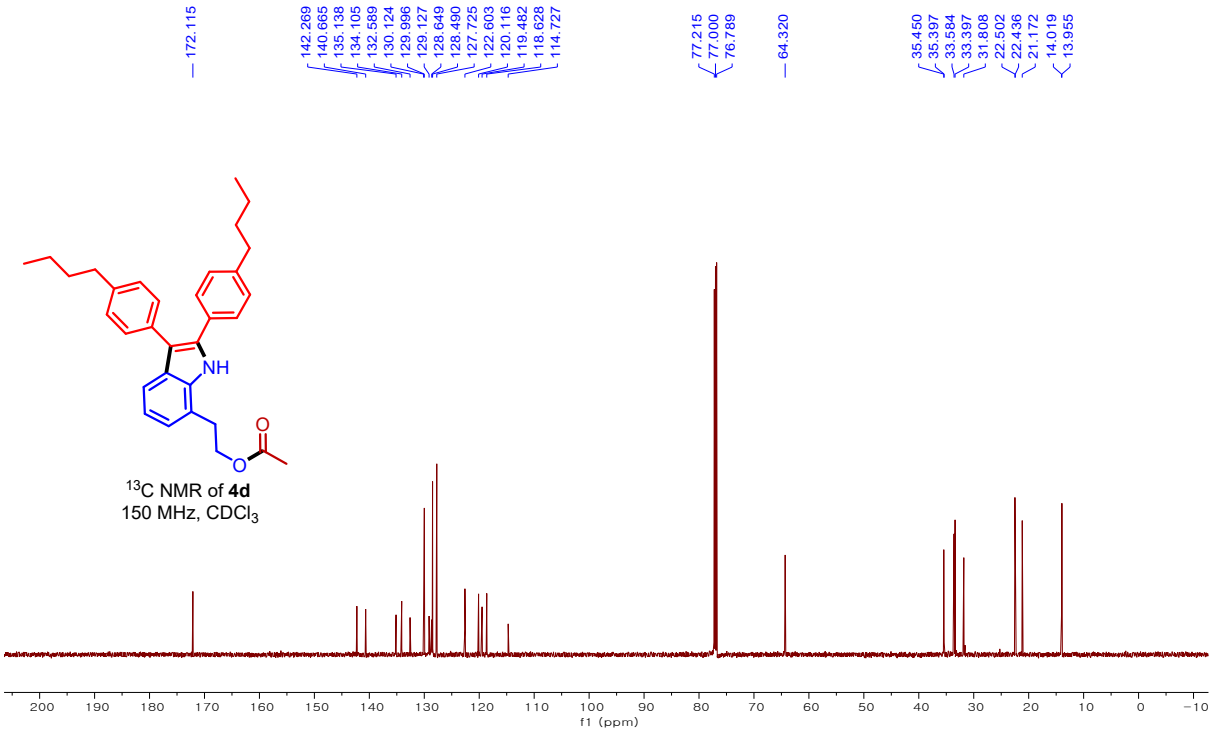


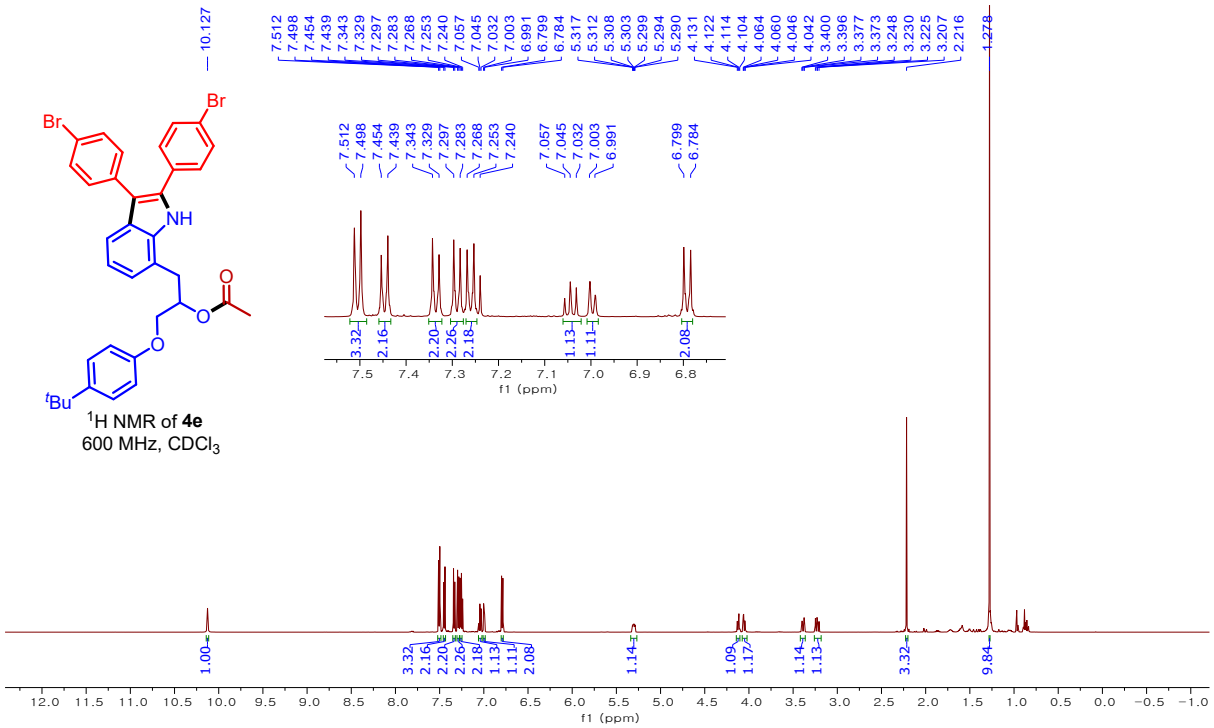
^{1}H NMR of **4c**
600 MHz, $CDCl_3$



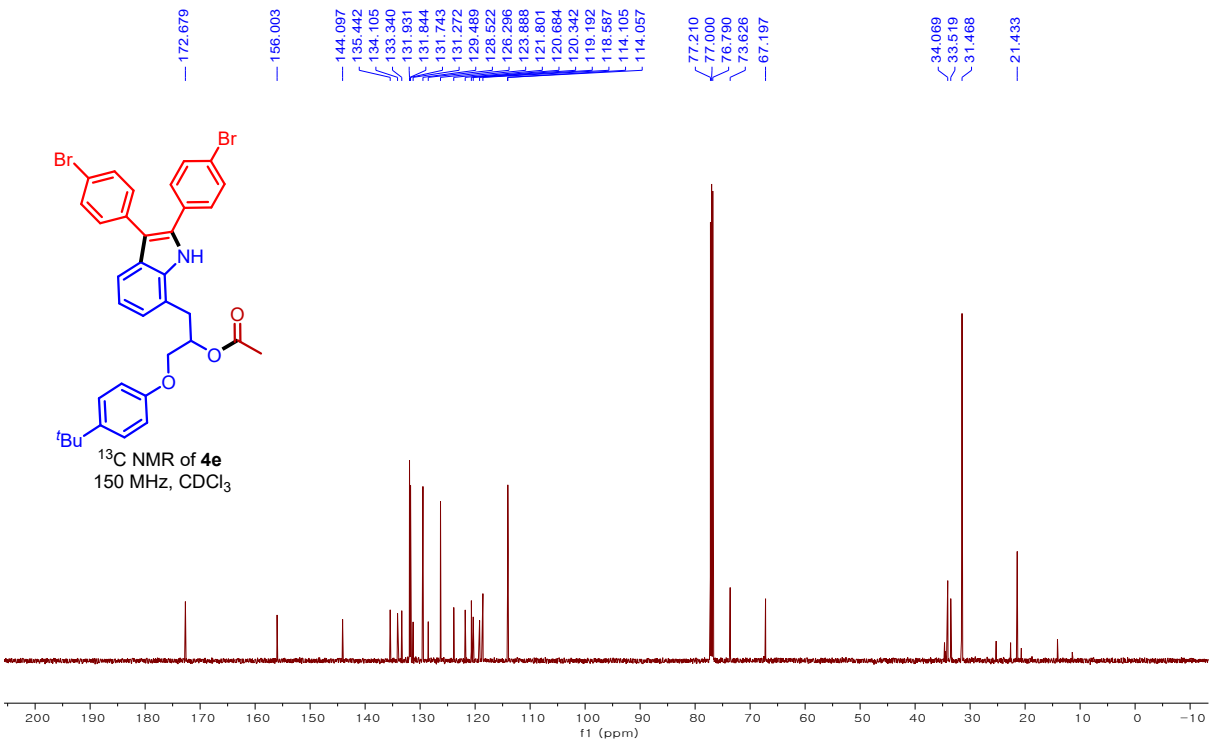
^{13}C NMR of **4c**
150 MHz, $CDCl_3$


NH
O
O
^{1}H NMR of **4d**
600 MHz, $CDCl_3$
f1 (ppm)

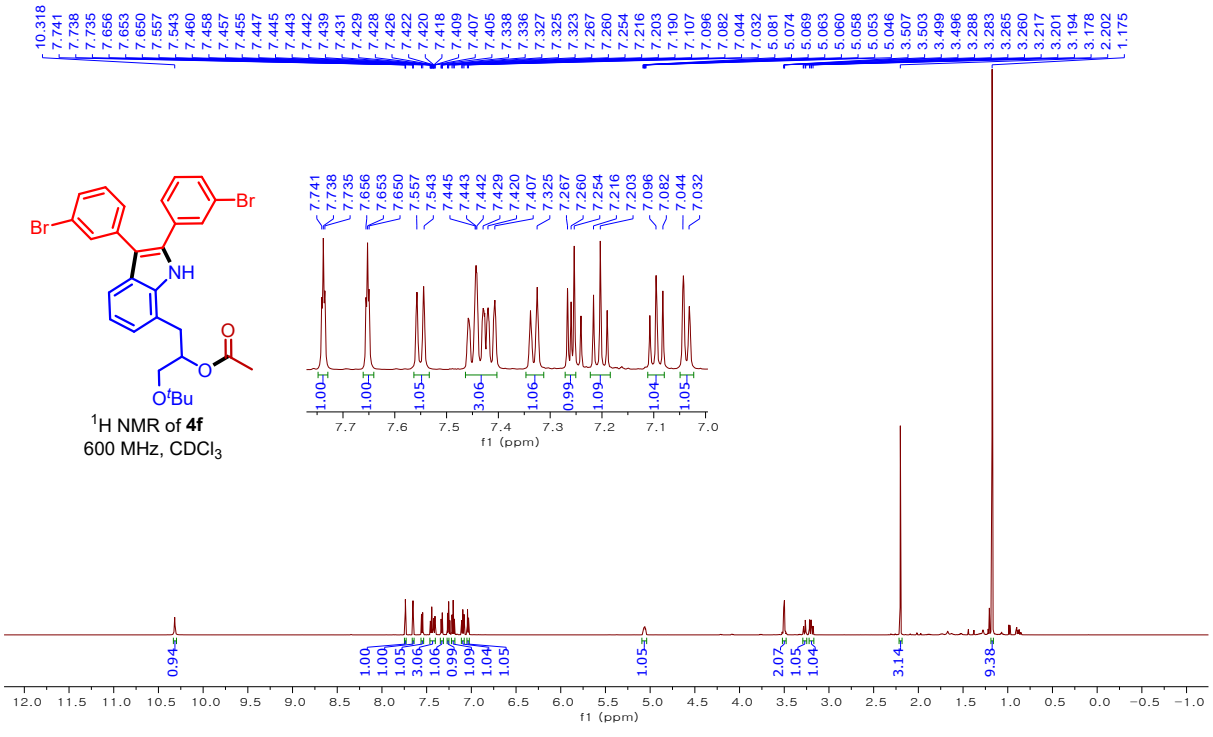

NH
O
O
^{13}C NMR of **4d**
150 MHz, $CDCl_3$
f1 (ppm)



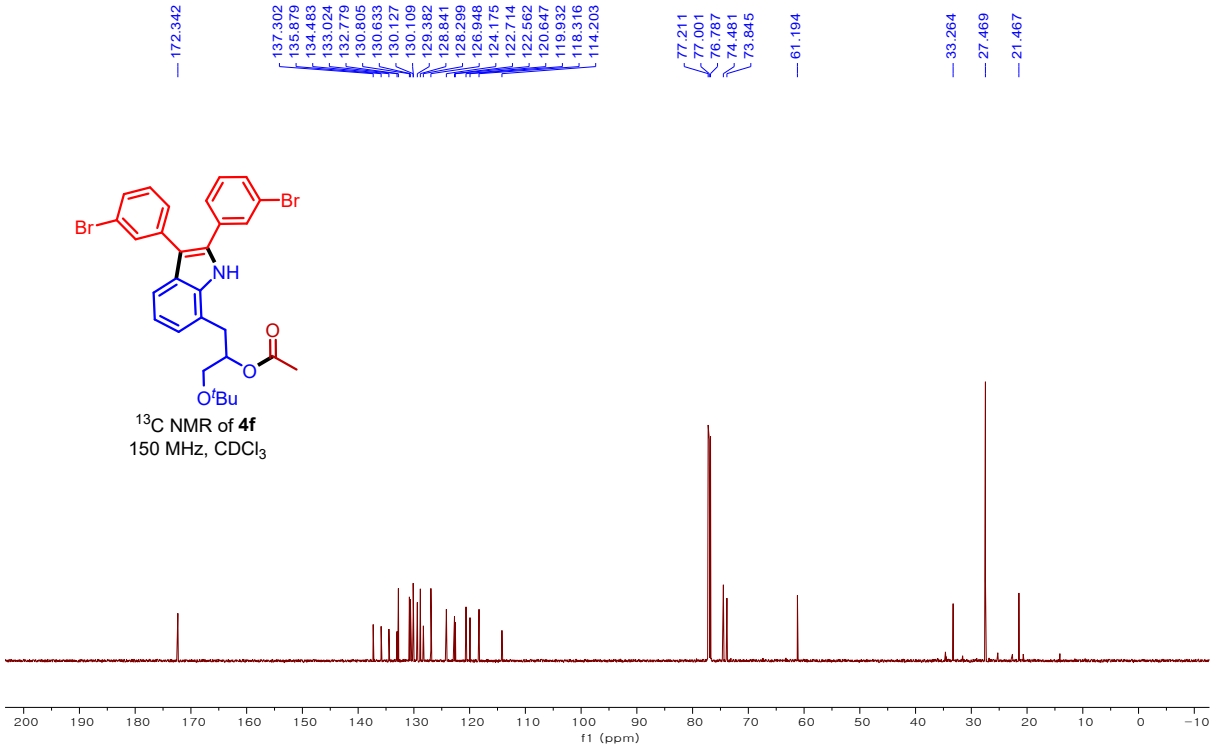
^{1}H NMR of **4e**
600 MHz, $CDCl_3$



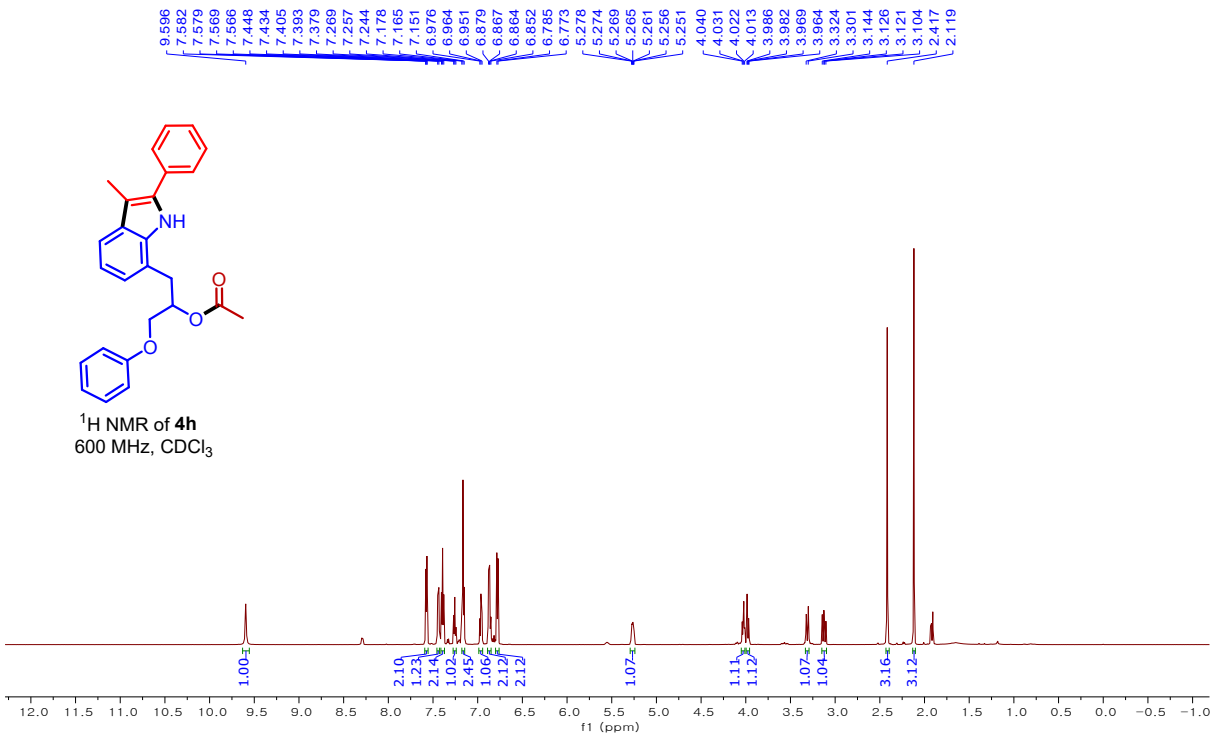
^{13}C NMR of **4e**
150 MHz, $CDCl_3$



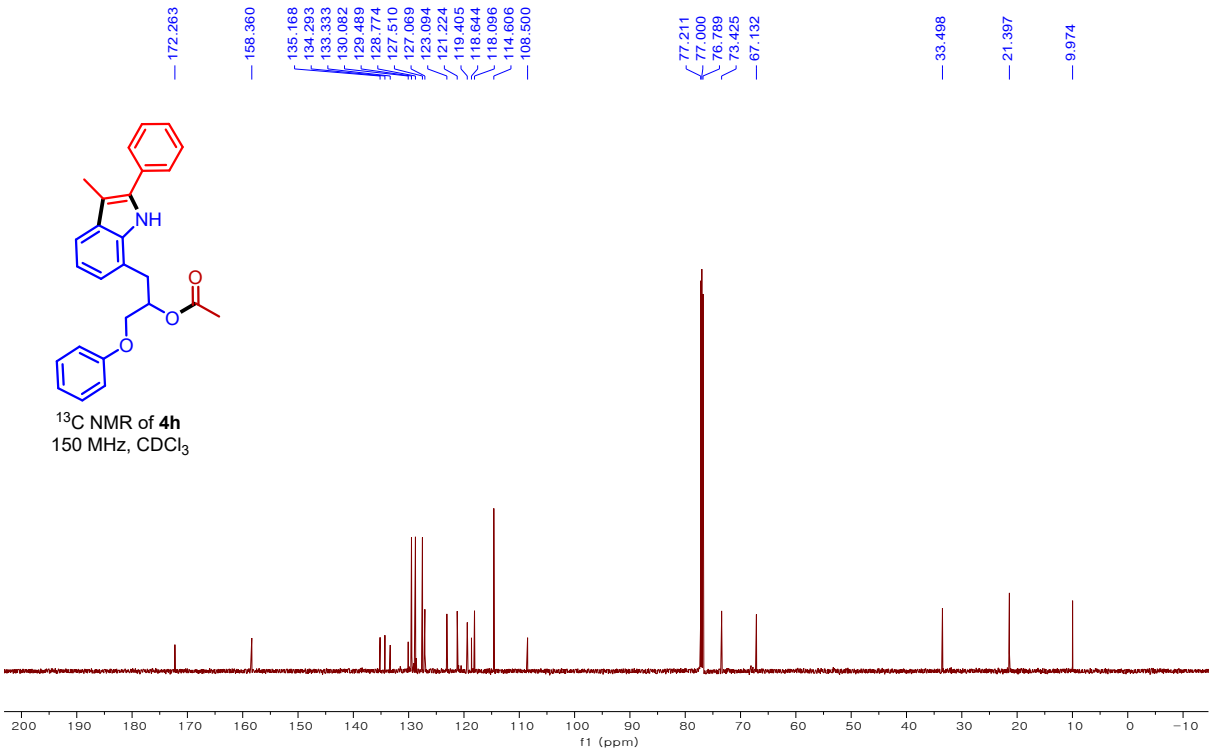
^{1}H NMR of **4f**
600 MHz, $CDCl_3$



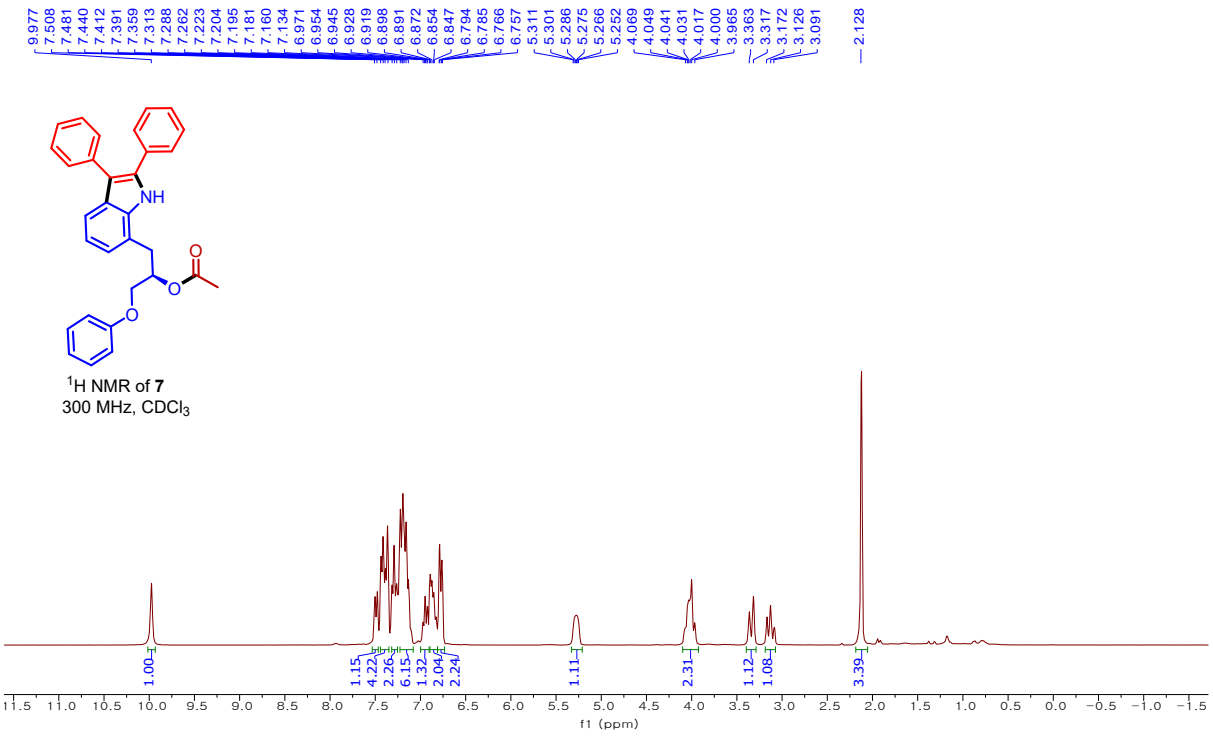
^{13}C NMR of **4f**
150 MHz, $CDCl_3$



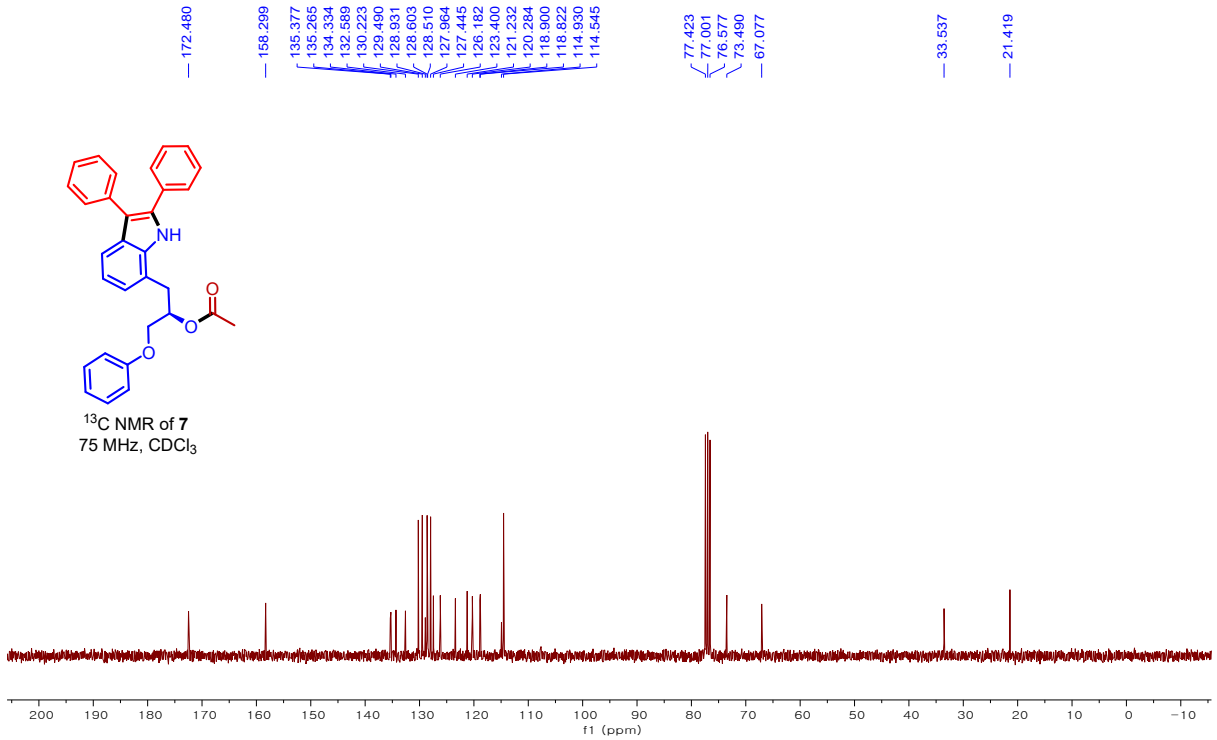
^{1}H NMR of **4h**
600 MHz, $CDCl_3$



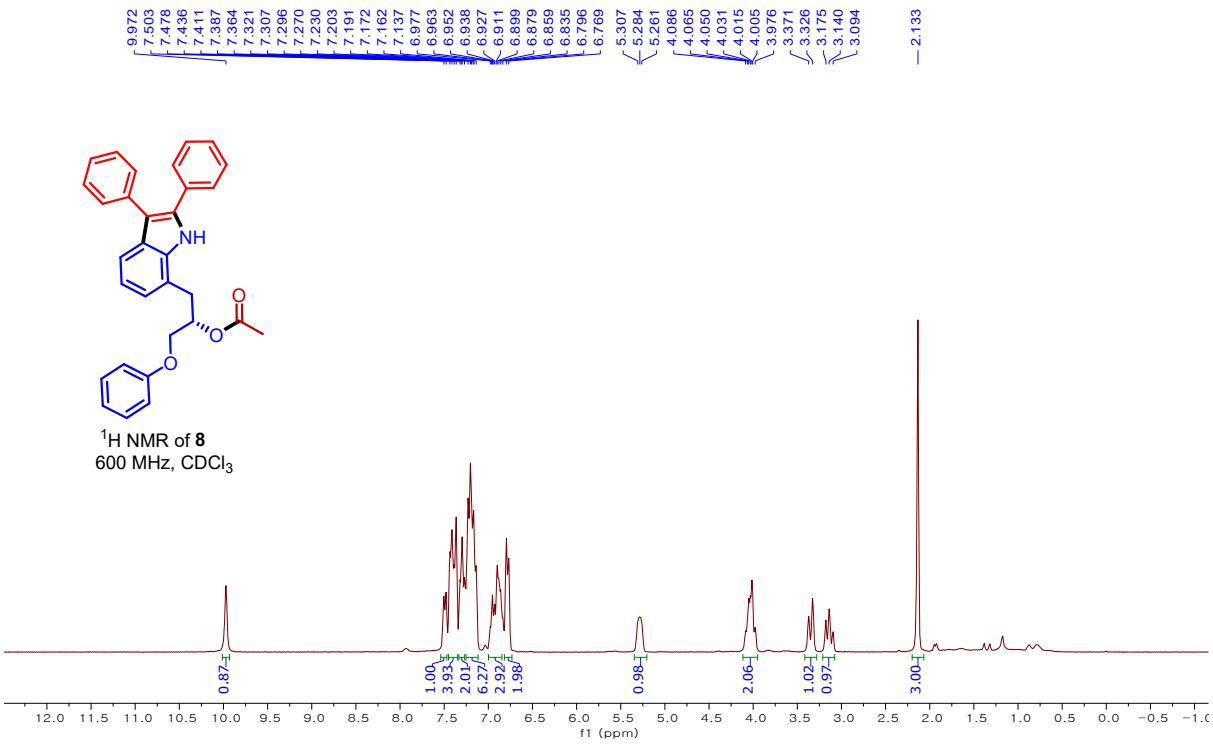
^{13}C NMR of **4h**
150 MHz, $CDCl_3$



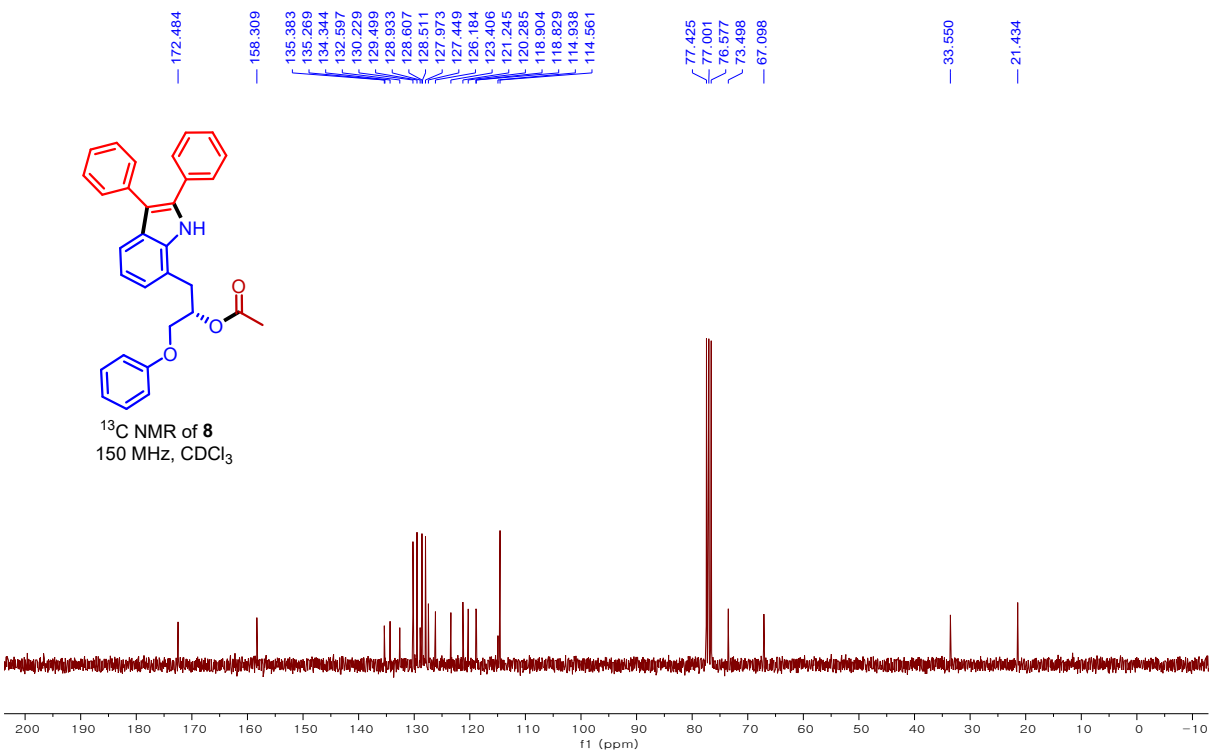
^{1}H NMR of **7**
300 MHz, $CDCl_3$



^{13}C NMR of **7**
75 MHz, $CDCl_3$



^{1}H NMR of **8**
600 MHz, $CDCl_3$



^{13}C NMR of **8**
150 MHz, $CDCl_3$

Crystal refinement data for compound 3a:

The crystal was prepared *via* slow evaporation method using ethyl acetate/hexane as solvents. The crystal structure of the compound **3a** was determined by single-crystal diffraction methods at the Korea Basic Science Institute (KBSI, Western Seoul Center, Korea). Colorless block crystal (0.262 x 0.187 x 0.174 mm^3) was picked up with paraton oil and mounted on a Bruker D8 Venture PHOTON III M14 diffractometer equipped with a graphite-monochromated Mo Kα (λ= 0.71073 Å) radiation source and a nitrogen cold stream (-50 °C). Data collection and integration were performed with SMART APEX3 (Bruker, 2016) and SAINT (Bruker, 2016) [1]. Absorption correction was performed by multi-scan method implemented in SADABS [2]. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXTL [3]. All the non-hydrogen atoms were refined anisotropically, and hydrogen atoms were added to their geometrically ideal positions.

1. SMART, SAINT and SADABS, Bruker AXS Inc., Madison, Wisconsin, USA, 2016.
2. G. M. Sheldrick, SADABS v 2.03, University of Göttingen, Germany, 2002.
3. SHELXTL v 6.10; Bruker AXS, Inc: Madison, Wisconsin, USA, 2000.

Empirical Formula- $C_{31}H_{27}NO_3$, M= 461.53, Triclinic, Space group P-1, a = 10.3312(6) Å, b = 11.7235(6) Å, c = 11.9422(6) Å, V = 1348.11(13) $Å^3$, Z = 2, T = 223(2) K, ρ_{calcd} = 1.137 Mg/m^3, $2\Theta_{max.}$ = 25.24^0, Refinement of 321 parameters on 6716 independent reflections out of 43834 collected reflections (R_{int} = 0.0636) led to R_1 = 0.0911 [I >2σ(I)], wR_2 = 0.1541 (all data) and S = 1.038 with the largest difference peak and hole of 0.271 and -0.259 $e.A^{-3}$ respectively. The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (CCDC 2092174). The data can be obtained free of charge via the Internet at www.ccdc.cam.ac.uk/data_request/cif.

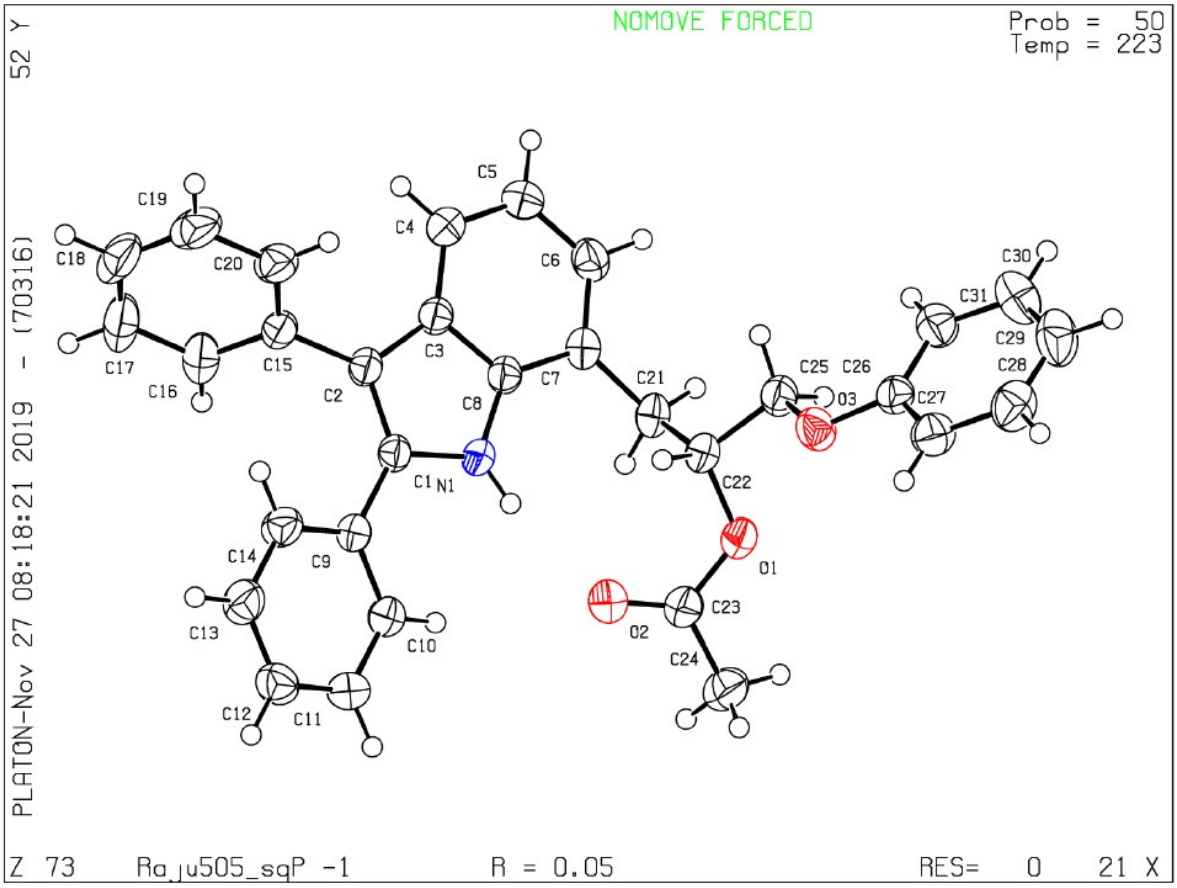


Figure S3. X-ray structure of compound **3a** (ellipsoid contour % probability-50).

Table 1. Crystal data and structure refinement for Raju505_sq.

Identification code	Raju505_sq	
Empirical formula	C31 H27 N O3 [+ solvent]	
Formula weight	461.53	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.3312(6) Å	α= 99.9553(18)°.
	b = 11.7235(6) Å	β= 105.4578(19)°.
	c = 11.9422(6) Å	γ = 97.7123(19)°.
Volume	1348.11(13) $Å^3$	
Z	2	
Density (calculated)	1.137 Mg/m^3	
Absorption coefficient	0.073 mm^{-1}	
F(000)	488	
Crystal size	0.291 x 0.201 x 0.140 mm^3	
Theta range for data collection	1.796 to 28.349°.	
Index ranges	-13<=h<=13, -15<=k<=15, -15<=l<=15	
Reflections collected	43834	
Independent reflections	6716 [R(int) = 0.0636]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.7046	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6716 / 0 / 321	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0549, wR2 = 0.1313	
R indices (all data)	R1 = 0.0911, wR2 = 0.1541	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.271 and -0.259 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Raju505_sq. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	1673(2)	-406(1)	1094(1)	33(1)
C(1)	1705(2)	-1538(2)	1273(2)	32(1)
C(2)	1074(2)	-1700(2)	2137(2)	33(1)
C(3)	672(2)	-607(2)	2521(2)	33(1)
C(4)	-5(2)	-230(2)	3350(2)	39(1)
C(5)	-272(2)	892(2)	3472(2)	43(1)
C(6)	107(2)	1653(2)	2780(2)	40(1)
C(7)	776(2)	1318(2)	1951(2)	34(1)
C(8)	1053(2)	175(2)	1843(2)	32(1)
C(9)	2393(2)	-2313(2)	616(2)	34(1)
C(10)	2705(2)	-2066(2)	-394(2)	40(1)
C(11)	3372(2)	-2791(2)	-992(2)	45(1)
C(12)	3728(2)	-3778(2)	-608(2)	45(1)
C(13)	3437(2)	-4036(2)	391(2)	48(1)
C(14)	2789(2)	-3306(2)	1008(2)	42(1)
C(15)	818(2)	-2770(2)	2608(2)	35(1)
C(16)	33(2)	-3828(2)	1879(2)	48(1)
C(17)	-171(3)	-4820(2)	2348(3)	62(1)
C(18)	380(3)	-4761(2)	3539(3)	69(1)
C(19)	1134(3)	-3720(2)	4275(2)	63(1)
C(20)	1349(2)	-2723(2)	3815(2)	47(1)
C(21)	1138(2)	2092(2)	1153(2)	37(1)
C(22)	2667(2)	2560(2)	1428(2)	36(1)
O(1)	2927(1)	2858(1)	365(1)	41(1)
C(23)	2988(2)	1957(2)	-472(2)	44(1)
O(2)	2907(2)	952(1)	-350(1)	56(1)
C(24)	3146(3)	2347(2)	-1561(2)	58(1)
C(25)	3132(2)	3681(2)	2373(2)	38(1)
O(3)	4583(1)	3987(1)	2684(1)	44(1)
C(26)	5198(2)	5084(2)	3395(2)	38(1)
C(27)	6595(2)	5398(2)	3584(2)	46(1)
C(28)	7290(2)	6494(2)	4262(2)	54(1)

C(29)	6614(3)	7276(2)	4762(2)	61(1)
C(30)	5229(3)	6948(2)	4586(2)	59(1)
C(31)	4510(2)	5852(2)	3906(2)	46(1)

Table 3. Bond lengths [Å] and angles [°] for Raju505_sq.

N(1)-C(8)	1.373(2)
N(1)-C(1)	1.384(2)
N(1)-H(1)	0.85(2)
C(1)-C(2)	1.385(2)
C(1)-C(9)	1.473(2)
C(2)-C(3)	1.437(2)
C(2)-C(15)	1.482(2)
C(3)-C(4)	1.402(3)
C(3)-C(8)	1.407(2)
C(4)-C(5)	1.372(3)
C(4)-H(4)	0.9400
C(5)-C(6)	1.402(3)
C(5)-H(5)	0.9400
C(6)-C(7)	1.384(3)
C(6)-H(6)	0.9400
C(7)-C(8)	1.399(2)
C(7)-C(21)	1.507(2)
C(9)-C(10)	1.395(2)
C(9)-C(14)	1.399(3)
C(10)-C(11)	1.384(3)
C(10)-H(10)	0.9400
C(11)-C(12)	1.376(3)
C(11)-H(11)	0.9400
C(12)-C(13)	1.378(3)
C(12)-H(12)	0.9400
C(13)-C(14)	1.386(3)
C(13)-H(13)	0.9400
C(14)-H(14)	0.9400
C(15)-C(20)	1.388(3)

C(15)-C(16)	1.391(3)
C(16)-C(17)	1.390(3)
C(16)-H(16)	0.9400
C(17)-C(18)	1.369(4)
C(17)-H(17)	0.9400
C(18)-C(19)	1.371(4)
C(18)-H(18)	0.9400
C(19)-C(20)	1.390(3)
C(19)-H(19)	0.9400
C(20)-H(20)	0.9400
C(21)-C(22)	1.528(3)
C(21)-H(21A)	0.9800
C(21)-H(21B)	0.9800
C(22)-O(1)	1.455(2)
C(22)-C(25)	1.504(3)
C(22)-H(22)	0.9900
O(1)-C(23)	1.343(2)
C(23)-O(2)	1.207(2)
C(23)-C(24)	1.492(3)
C(24)-H(24A)	0.9700
C(24)-H(24B)	0.9700
C(24)-H(24C)	0.9700
C(25)-O(3)	1.424(2)
C(25)-H(25A)	0.9800
C(25)-H(25B)	0.9800
O(3)-C(26)	1.377(2)
C(26)-C(31)	1.378(3)
C(26)-C(27)	1.387(3)
C(27)-C(28)	1.380(3)
C(27)-H(27)	0.9400
C(28)-C(29)	1.375(4)
C(28)-H(28)	0.9400
C(29)-C(30)	1.380(4)
C(29)-H(29)	0.9400
C(30)-C(31)	1.387(3)
C(30)-H(30)	0.9400
C(31)-H(31)	0.9400

C(8)-N(1)-C(1)	109.72(15)
C(8)-N(1)-H(1)	125.8(15)
C(1)-N(1)-H(1)	123.9(15)
N(1)-C(1)-C(2)	108.46(15)
N(1)-C(1)-C(9)	119.80(15)
C(2)-C(1)-C(9)	131.69(16)
C(1)-C(2)-C(3)	107.02(15)
C(1)-C(2)-C(15)	128.85(16)
C(3)-C(2)-C(15)	124.12(16)
C(4)-C(3)-C(8)	119.13(16)
C(4)-C(3)-C(2)	133.82(17)
C(8)-C(3)-C(2)	107.03(15)
C(5)-C(4)-C(3)	118.63(18)
C(5)-C(4)-H(4)	120.7
C(3)-C(4)-H(4)	120.7
C(4)-C(5)-C(6)	121.25(18)
C(4)-C(5)-H(5)	119.4
C(6)-C(5)-H(5)	119.4
C(7)-C(6)-C(5)	122.03(17)
C(7)-C(6)-H(6)	119.0
C(5)-C(6)-H(6)	119.0
C(6)-C(7)-C(8)	116.14(16)
C(6)-C(7)-C(21)	123.34(16)
C(8)-C(7)-C(21)	120.45(16)
N(1)-C(8)-C(7)	129.42(16)
N(1)-C(8)-C(3)	107.75(15)
C(7)-C(8)-C(3)	122.82(16)
C(10)-C(9)-C(14)	117.74(17)
C(10)-C(9)-C(1)	121.37(16)
C(14)-C(9)-C(1)	120.86(16)
C(11)-C(10)-C(9)	120.80(18)
C(11)-C(10)-H(10)	119.6
C(9)-C(10)-H(10)	119.6
C(12)-C(11)-C(10)	120.63(19)
C(12)-C(11)-H(11)	119.7
C(10)-C(11)-H(11)	119.7

C(11)-C(12)-C(13)	119.60(19)
C(11)-C(12)-H(12)	120.2
C(13)-C(12)-H(12)	120.2
C(12)-C(13)-C(14)	120.25(19)
C(12)-C(13)-H(13)	119.9
C(14)-C(13)-H(13)	119.9
C(13)-C(14)-C(9)	120.94(18)
C(13)-C(14)-H(14)	119.5
C(9)-C(14)-H(14)	119.5
C(20)-C(15)-C(16)	118.28(18)
C(20)-C(15)-C(2)	119.70(17)
C(16)-C(15)-C(2)	122.01(18)
C(17)-C(16)-C(15)	120.5(2)
C(17)-C(16)-H(16)	119.8
C(15)-C(16)-H(16)	119.8
C(18)-C(17)-C(16)	120.4(2)
C(18)-C(17)-H(17)	119.8
C(16)-C(17)-H(17)	119.8
C(17)-C(18)-C(19)	120.0(2)
C(17)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0
C(18)-C(19)-C(20)	120.2(2)
C(18)-C(19)-H(19)	119.9
C(20)-C(19)-H(19)	119.9
C(15)-C(20)-C(19)	120.7(2)
C(15)-C(20)-H(20)	119.7
C(19)-C(20)-H(20)	119.7
C(7)-C(21)-C(22)	114.71(15)
C(7)-C(21)-H(21A)	108.6
C(22)-C(21)-H(21A)	108.6
C(7)-C(21)-H(21B)	108.6
C(22)-C(21)-H(21B)	108.6
H(21A)-C(21)-H(21B)	107.6
O(1)-C(22)-C(25)	106.26(14)
O(1)-C(22)-C(21)	108.82(15)
C(25)-C(22)-C(21)	111.38(15)
O(1)-C(22)-H(22)	110.1

C(25)-C(22)-H(22)	110.1
C(21)-C(22)-H(22)	110.1
C(23)-O(1)-C(22)	116.51(14)
O(2)-C(23)-O(1)	123.34(18)
O(2)-C(23)-C(24)	124.56(19)
O(1)-C(23)-C(24)	112.10(17)
C(23)-C(24)-H(24A)	109.5
C(23)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(23)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
O(3)-C(25)-C(22)	108.22(15)
O(3)-C(25)-H(25A)	110.1
C(22)-C(25)-H(25A)	110.1
O(3)-C(25)-H(25B)	110.1
C(22)-C(25)-H(25B)	110.1
H(25A)-C(25)-H(25B)	108.4
C(26)-O(3)-C(25)	117.38(14)
O(3)-C(26)-C(31)	123.98(18)
O(3)-C(26)-C(27)	115.80(17)
C(31)-C(26)-C(27)	120.23(19)
C(28)-C(27)-C(26)	119.8(2)
C(28)-C(27)-H(27)	120.1
C(26)-C(27)-H(27)	120.1
C(29)-C(28)-C(27)	120.7(2)
C(29)-C(28)-H(28)	119.7
C(27)-C(28)-H(28)	119.7
C(28)-C(29)-C(30)	119.1(2)
C(28)-C(29)-H(29)	120.4
C(30)-C(29)-H(29)	120.4
C(29)-C(30)-C(31)	121.2(2)
C(29)-C(30)-H(30)	119.4
C(31)-C(30)-H(30)	119.4
C(26)-C(31)-C(30)	119.0(2)
C(26)-C(31)-H(31)	120.5
C(30)-C(31)-H(31)	120.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Raju505_sq. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... \ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	39(1)	27(1)	37(1)	11(1)	15(1)	5(1)
C(1)	35(1)	26(1)	33(1)	8(1)	9(1)	5(1)
C(2)	36(1)	29(1)	35(1)	9(1)	10(1)	4(1)
C(3)	34(1)	30(1)	34(1)	8(1)	10(1)	3(1)
C(4)	42(1)	38(1)	41(1)	11(1)	16(1)	6(1)
C(5)	47(1)	42(1)	45(1)	6(1)	21(1)	11(1)
C(6)	41(1)	32(1)	48(1)	6(1)	14(1)	9(1)
C(7)	32(1)	30(1)	38(1)	9(1)	6(1)	4(1)
C(8)	33(1)	29(1)	33(1)	7(1)	9(1)	3(1)
C(9)	34(1)	30(1)	36(1)	7(1)	11(1)	3(1)
C(10)	51(1)	33(1)	41(1)	12(1)	17(1)	9(1)
C(11)	54(1)	45(1)	41(1)	9(1)	23(1)	8(1)
C(12)	44(1)	43(1)	51(1)	5(1)	19(1)	11(1)
C(13)	50(1)	41(1)	63(1)	20(1)	27(1)	18(1)
C(14)	49(1)	42(1)	48(1)	19(1)	23(1)	17(1)
C(15)	35(1)	32(1)	45(1)	14(1)	18(1)	9(1)
C(16)	43(1)	34(1)	66(1)	13(1)	16(1)	4(1)
C(17)	52(1)	34(1)	111(2)	23(1)	38(2)	7(1)
C(18)	85(2)	50(1)	111(2)	49(2)	68(2)	32(1)
C(19)	91(2)	63(2)	68(2)	41(1)	50(2)	38(1)
C(20)	61(1)	46(1)	47(1)	19(1)	26(1)	19(1)
C(21)	37(1)	30(1)	45(1)	12(1)	9(1)	6(1)
C(22)	41(1)	31(1)	39(1)	12(1)	13(1)	6(1)
O(1)	52(1)	33(1)	41(1)	12(1)	16(1)	3(1)
C(23)	48(1)	39(1)	44(1)	7(1)	17(1)	0(1)
O(2)	80(1)	37(1)	57(1)	9(1)	34(1)	7(1)
C(24)	72(2)	56(1)	45(1)	11(1)	23(1)	-4(1)
C(25)	37(1)	34(1)	45(1)	9(1)	15(1)	4(1)

O(3)	38(1)	38(1)	51(1)	0(1)	14(1)	1(1)
C(26)	43(1)	36(1)	32(1)	8(1)	10(1)	2(1)
C(27)	42(1)	49(1)	43(1)	8(1)	11(1)	3(1)
C(28)	46(1)	55(1)	50(1)	11(1)	3(1)	-6(1)
C(29)	66(2)	43(1)	55(1)	4(1)	-1(1)	-4(1)
C(30)	65(2)	45(1)	57(1)	-2(1)	8(1)	11(1)
C(31)	47(1)	43(1)	46(1)	6(1)	13(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Raju505_sq.

	x	y	z	U(eq)
H(1)	2080(20)	-100(20)	650(20)	49(6)
H(4)	-270	-737	3813	47
H(5)	-718	1157	4030	52
H(6)	-100	2414	2882	48
H(10)	2459	-1400	-673	48
H(11)	3584	-2607	-1666	54
H(12)	4168	-4274	-1026	54
H(13)	3679	-4710	656	57
H(14)	2614	-3481	1698	51
H(16)	-362	-3873	1063	58
H(17)	-690	-5534	1846	74
H(18)	241	-5435	3851	82
H(19)	1507	-3679	5093	76
H(20)	1860	-2010	4327	57
H(21A)	671	2764	1216	45
H(21B)	787	1642	327	45
H(22)	3196	1956	1680	43
H(24A)	2531	1797	-2263	87
H(24B)	4082	2369	-1579	87
H(24C)	2928	3127	-1549	87
H(25A)	2717	4315	2073	46
H(25B)	2853	3567	3075	46

H(27)	7067	4866	3251	55
H(28)	8235	6708	4383	65
H(29)	7089	8024	5218	73
H(30)	4765	7477	4934	71
H(31)	3567	5636	3795	55

Table 6. Torsion angles [°] for Raju505_sq.

C(8)-N(1)-C(1)-C(2)	1.1(2)
C(8)-N(1)-C(1)-C(9)	-176.54(16)
N(1)-C(1)-C(2)-C(3)	-1.5(2)
C(9)-C(1)-C(2)-C(3)	175.71(18)
N(1)-C(1)-C(2)-C(15)	177.98(18)
C(9)-C(1)-C(2)-C(15)	-4.8(3)
C(1)-C(2)-C(3)-C(4)	179.3(2)
C(15)-C(2)-C(3)-C(4)	-0.2(3)
C(1)-C(2)-C(3)-C(8)	1.4(2)
C(15)-C(2)-C(3)-C(8)	-178.12(17)
C(8)-C(3)-C(4)-C(5)	0.0(3)
C(2)-C(3)-C(4)-C(5)	-177.8(2)
C(3)-C(4)-C(5)-C(6)	0.6(3)
C(4)-C(5)-C(6)-C(7)	-0.6(3)
C(5)-C(6)-C(7)-C(8)	0.1(3)
C(5)-C(6)-C(7)-C(21)	176.98(18)
C(1)-N(1)-C(8)-C(7)	-178.60(18)
C(1)-N(1)-C(8)-C(3)	-0.2(2)
C(6)-C(7)-C(8)-N(1)	178.72(18)
C(21)-C(7)-C(8)-N(1)	1.7(3)
C(6)-C(7)-C(8)-C(3)	0.5(3)
C(21)-C(7)-C(8)-C(3)	-176.53(17)
C(4)-C(3)-C(8)-N(1)	-179.07(17)
C(2)-C(3)-C(8)-N(1)	-0.8(2)
C(4)-C(3)-C(8)-C(7)	-0.5(3)
C(2)-C(3)-C(8)-C(7)	177.80(17)
N(1)-C(1)-C(9)-C(10)	-16.8(3)
C(2)-C(1)-C(9)-C(10)	166.26(19)

N(1)-C(1)-C(9)-C(14)	161.43(18)
C(2)-C(1)-C(9)-C(14)	-15.5(3)
C(14)-C(9)-C(10)-C(11)	0.7(3)
C(1)-C(9)-C(10)-C(11)	178.92(18)
C(9)-C(10)-C(11)-C(12)	0.7(3)
C(10)-C(11)-C(12)-C(13)	-1.1(3)
C(11)-C(12)-C(13)-C(14)	0.0(3)
C(12)-C(13)-C(14)-C(9)	1.4(3)
C(10)-C(9)-C(14)-C(13)	-1.7(3)
C(1)-C(9)-C(14)-C(13)	-179.98(19)
C(1)-C(2)-C(15)-C(20)	121.4(2)
C(3)-C(2)-C(15)-C(20)	-59.2(3)
C(1)-C(2)-C(15)-C(16)	-59.8(3)
C(3)-C(2)-C(15)-C(16)	119.7(2)
C(20)-C(15)-C(16)-C(17)	-2.0(3)
C(2)-C(15)-C(16)-C(17)	179.11(19)
C(15)-C(16)-C(17)-C(18)	1.1(3)
C(16)-C(17)-C(18)-C(19)	0.2(4)
C(17)-C(18)-C(19)-C(20)	-0.4(4)
C(16)-C(15)-C(20)-C(19)	1.8(3)
C(2)-C(15)-C(20)-C(19)	-179.29(19)
C(18)-C(19)-C(20)-C(15)	-0.6(3)
C(6)-C(7)-C(21)-C(22)	110.9(2)
C(8)-C(7)-C(21)-C(22)	-72.3(2)
C(7)-C(21)-C(22)-O(1)	156.93(15)
C(7)-C(21)-C(22)-C(25)	-86.26(19)
C(25)-C(22)-O(1)-C(23)	160.38(16)
C(21)-C(22)-O(1)-C(23)	-79.6(2)
C(22)-O(1)-C(23)-O(2)	-3.8(3)
C(22)-O(1)-C(23)-C(24)	175.43(17)
O(1)-C(22)-C(25)-O(3)	-68.52(18)
C(21)-C(22)-C(25)-O(3)	173.12(14)
C(22)-C(25)-O(3)-C(26)	169.79(15)
C(25)-O(3)-C(26)-C(31)	6.1(3)
C(25)-O(3)-C(26)-C(27)	-173.87(17)
O(3)-C(26)-C(27)-C(28)	178.42(18)
C(31)-C(26)-C(27)-C(28)	-1.5(3)

C(26)-C(27)-C(28)-C(29)	0.6(3)
C(27)-C(28)-C(29)-C(30)	0.5(4)
C(28)-C(29)-C(30)-C(31)	-0.7(4)
O(3)-C(26)-C(31)-C(30)	-178.56(19)
C(27)-C(26)-C(31)-C(30)	1.4(3)
C(29)-C(30)-C(31)-C(26)	-0.3(4)

Symmetry transformations used to generate equivalent atoms: