"On water" Catalysis: An expeditious approach for the synthesis of Quaternary Centered 3-hydroxy-3-(nitromethyl)indolin-2-one derivatives

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SUPPORTING INFORMATION

Contents

A. General Information

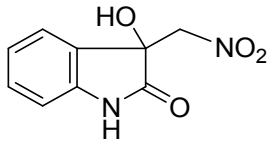
All the substrates including isatin and other chemicals were purchased from Sigma Aldrich, Spectrochem and S. D. Fine chemicals, Pvt. Ltd, India and were used as received without further purification. The N-substituted isatins were synthesized following the literature procedure.[1] The reagents and solvents were commercially available. ACME silica gel was used for column chromatography and was performed on Merck-pre-coated silica gel 60-F_{254} plates and visualized by UV-light. All ^{1}H, ^{13}C NMR spectra were recorded on a Varian-Gemini 200 MHz, Avance-300, Innova-500 MHz Spectrometer using $CDCl_3$ and d_6-DMSO solvents. IR spectra were recorded using a NEXUS 670 FT-IR spectrometer (Nicolet Corporation Ltd, USA) in a KBr pellet. ESI-MS spectra were acquired in positive ionization mode on a LCQ ion trap mass spectrometer (Thermo Fisher, San Jose, CA, USA), equipped with an ESI source. HRMS spectra were determined on QSTAR XL (Applied Bio systems/MDS Scitex, Foster City, USA).

B. Experimental procedure for synthesis of (3a-r)

To the reaction vessel containing isatin (0.5 mmol), nitromethane (2.0 mmol) and water 3 mL was added and the reaction mixture was vigorously stirred at room temperature. The reaction was stirred until orange colour of starting material turns to white precipitate. After the complete consumption of starting material as monitored by thin layer chromatography using ethylacetate and hexane as eluent (3:7) the reaction product was filtered, washed with water and recrystallized from methanol/EtOAc to afford pure products.

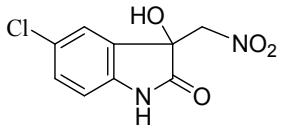
C. Spectroscopic data for all products (3a-r)

3-hydroxy-3-(nitromethyl)indolin-2-one 3a:



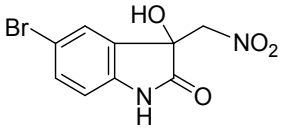
Isolated yield = 99%; IR (KBr) ν 3263, 1734, 1621, 1550, 1468, 1186, 1103, 754 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$+d_6-DMSO): δ 4.81 (d, *J* = 12.6 Hz, 1H), 4.88 (d, *J* = 12.6 Hz, 1H), 6.5 (s, 1H, OH), 6.85 (d, *J* = 7.3 Hz, 1H), 6.95 (t, *J* = 7.3 Hz, 1H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.31 (d, *J* = 7.3 Hz, 1H), 10.3 (s, 1H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): δ 78.2, 95.4, 109.8, 121.5, 124.3, 127.6, 129.8, 142.4, 175.6. ESI MS (m/z): 231 $(M+Na)^+$. HRMS(ESI) calcd for $C_9H_8N_2O_4Na$ $[M+Na]^+$ 231.0375, Found 231.0381.

5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3b:



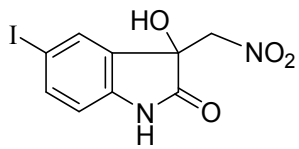
Isolated yield = 90%; IR (KBr) ν 3346, 1713, 1617, 1558, 1479, 1188, 1087, 833 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$+d_6-DMSO): δ 4.89 (s, 2H), 6.74 (s, 1H, OH), 6.85 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 1H), 7.38 (s, 1H), 10.54 (s, 1H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): δ 72.6, 77.8, 111.4, 124.0, 124.8, 125.7, 129.9, 141.4, 175.5. ESI MS(m/z): 264 $(M+Na)^+$. HRMS(ESI) calcd for $C_9H_7N_2O_4ClNa$ $[M+Na]^+$ 264.9990, Found 264.9992.

5-bromo-3-hydroxy-3-(nitromethyl)indolin-2-one 3c:



Isolated yield = 94%; IR (KBr) ν 3394, 1732, 1616, 1549, 1477, 1183, 1088, 827 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.87 (d, *J* = 2.6 Hz, 2H), 6.68 (s, 1H, OH), 6.81 (d, *J* = 8.3 Hz, 1H), 7.36 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.49 (d, *J* = 1.8 Hz, 1H), 10.48 (s, 1H, NH). ^{13}C NMR (75 MHz, d_6-DMSO): δ 72.5, 77.8, 111.9, 113.3, 127.5, 130.4, 132.7, 141.8, 175.4. ESI MS(m/z): 308 $(M+Na)^+$. HRMS(ESI) calcd for $C_9H_7N_2O_4BrNa$ $[M+Na]^+$ 308.9475, found 308.9486.

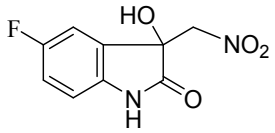
3-hydroxy-5-iodo-3-(nitromethyl)indolin-2-one 3d:



Isolated yield = 94%; IR (KBr) ν 3390, 1730, 1612, 1542, 1474, 1181, 1085, 823 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.87 (s, 2H), 6.69 (s, 1H, OH), 6.71 (d, *J* = 2.0 Hz, 1H), 7.55

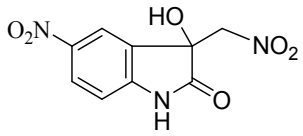
(dd, J = 8.1, 1.7 Hz, 1H), 7.65 (d, J = 1.5 Hz, 1H), 10.53 (s, 1H, NH). ^{13}C NMR (75 MHz, d_6-DMSO): δ 72.4, 77.8, 84.4, 112.4, 130.7, 133.0, 138.6, 142.4, 175.2. ESI MS(m/z): 356$(M+Na)^+$. HRMS(ESI) calcd for $C_9H_7N_2O_4INa$ $[M+Na]^+$ 356.9352, found 356.9348.

5-fluoro-3-hydroxy-3-(nitromethyl)indolin-2-one 3e:



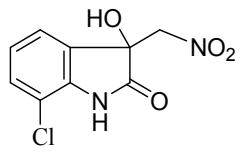
Isolated yield = 91%; IR (KBr) ν 3239, 1720, 1620, 1560, 1490, 1276, 1182, 1072, 824 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.86 (d, J = 1.8 Hz, 2H), 6.68 (s, 1H, OH), 6.84 (dd, J = 8.4, 4.3 Hz, 1H), 6.94 (dd, J = 8.8, 2.4 Hz, 1H), 7.15 (dd, J = 7.7, 2.4 Hz, 1H), 10.37 (s, 1H, NH). ^{13}C NMR (75 MHz, d_6-DMSO): δ 72.8, 78.0, 110.8, 112.7, 116.6, 129.7, 138.7, 159.4, 175.8. ESI MS(m/z): 249 $(M+Na)^+$. HRMS(ESI) calcd for $C_9H_7N_2O_4FNa$ $[M+Na]^+$ 249.0280, found 249.0287.

3-hydroxy-5-nitro-3-(nitromethyl)indolin-2-one 3f:



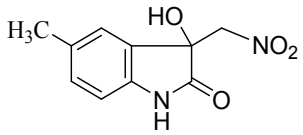
Isolated yield = 90%; IR (KBr) ν 3324, 1744, 1694, 1558, 1534, 1489, 1188, 1384, 1089, 845 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$+d_6-DMSO): δ 4.9 (d, J = 13.5 Hz, 1H), 5.06 (d, J = 13.5 Hz, 1H) 6.85 (s, 1H, OH), 7.0 (d, J = 9.0 Hz, 1H), 8.18 (dd, J = 7.9, 2.2 Hz, 1H), 8.3 (d, J = 2.2 Hz, 1H), 11.0 (s, 1H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): 72.0, 77.3, 110.2, 120.6, 127.3, 129.1, 142.2, 149.0, 176.2. ESI MS(m/z): 276 $(M+Na)^+$.

7-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3g:



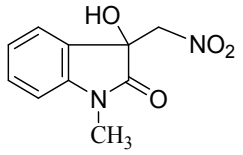
Isolated yield = 91%; IR (KBr) ν 3346, 1713, 1617, 1558, 1479, 1188, 1087, 833 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$+d_6-DMSO): δ 4.8 (q, 2H), 6.71 (s, 1H, OH), 6.92 (d, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.24 (d, *J* = 7.0 Hz, 1H), 10.6 (s, 1H, NH). ^{13}C NMR (100 MHz, d_6-DMSO): 73.2, 78.1, 114.2, 123.3, 129.9, 130.2, 140.3, 175.9. ESI MS(m/z): 264 $(M+Na)^+$. HRMS(ESI) calcd for $C_9H_7N_2O_4ClNa$ $[M+Na]^+$ 264.91410, Found 264.91414.

3-hydroxy-5-methyl-3-(nitromethyl)indolin-2-one 3h:



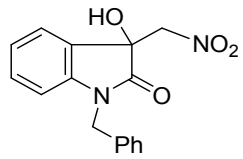
Isolated yield = 80%; IR (KBr) ν 3172, 2924 1737, 1625, 1553, 1490, 1304, 1027, 825 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$+d_6-DMSO): δ 2.3 (s, 3H), 4.76-4.87 (m, 2H), 6.47 (s, 1H, OH), 6.74 (d, *J* = 7.9 Hz, 1H), 7.02 (d, *J* = 7.9 Hz, 1H), 7.13 (s, 1H), 10.2 (s, 1H, NH). ^{13}C NMR (75 MHz, d_6-DMSO): δ 20.5, 72.8, 109.7, 125.1, 127.9, 130.6, 138.7, 140.0, 175. ESI MS(m/z): 245 $(M+Na)^+$. HRMS(ESI) calcd for $C_{10}H_{10}N_2O_4Na$ $[M+Na]^+$ 245.0532, found 245.0538

3-hydroxy-1-methyl-3-(nitromethyl)indolin-2-one 3i:



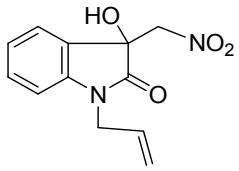
Isolated yield = 88%; IR (KBr) ν 3383, 2926, 1730, 1613, 1553, 1470, 1375, 1096, 756 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$+d_6-DMSO): δ 3.21 (s, 3H), 4.95-4.83 (m, 2H), 6.65 (s, 1H, OH), 6.88 (d, *J* = 7.7 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.4 (d, *J* = 7.3 Hz, 1H). ^{13}C NMR (100 MHz, CDCl3, TMS): δ 25.8, 72.2, 95.4, 108.2, 122.2, 123.8, 126.6, 130.0, 143.6, 173.9. ESI MS(m/z): 223 $(M+1)^+$. HRMS(ESI) calcd for $C_{10}H_{11}N_2O_4$ $[M+1]^+$ 223.0711, Found 223.0718.

1-benzyl-3-hydroxy-3-(nitromethyl)indolin-2-one 3j:



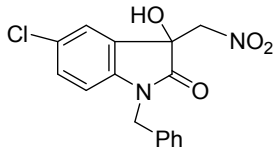
Isolated yield = 92%; IR (KBr) ν 3417, 2927, 1737, 1612, 1553, 1469, 1178, 1030,754 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.82-4.92 (m, 2H), 4.95-5.06 (m, 2H), 6.71(d, *J* = 7.7 Hz, 1H), 6.80 (s, 1H, OH), 7.02 (t, *J* = 7.5 Hz, 1H), 7.23-7.32 (m, 6H), 7.42 (d, *J* = 7.1 Hz, 1H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 43.2, 72.2, 77.7, 109.1, 122.3, 123.8, 126.5, 126.7, 127.0, 128.1, 129.9, 134.8, 142.7, 174.3. ESI MS(m/z): 299 $(M+1)^+$. HRMS(ESI) calcd for $C_{16}H_{15}N_2O_4$ $[M+1]^+$ 299.1045, Found 299.1031.

1-allyl-3-hydroxy-3-(nitromethyl)indolin-2-one 3k:



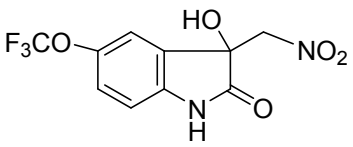
Isolated yield = 74%; IR (KBr) ν 3374, 2925, 1731, 1614, 1554, 1469, 1373, 1186, 1072, 755 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$+d_6-DMSO): δ 4.28-4.34 (m, 2H), 5.02 (m, 2H), 5.20-5.29 (m, 2H), 5.78-5.87 (m, 1H), 6.83 (s, 1H, OH), 6.92 (d, *J* = 7.7 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 7.31 (t, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 7.1 Hz, 1H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 41.5, 72.4, 78.2, 109.5, 116.9, 122.4, 124.3, 127.2, 130.2, 131.4, 143, 174. ESI MS(m/z): 249 $(M+1)^+$. HRMS(ESI) calcd for $C_{12}H_{13}N_2O_4$ $[M+1]^+$ 249.0868, Found 249.0875.

1-benzyl-5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3l:



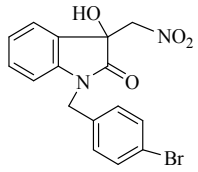
Isolated yield = 88%; IR (KBr) ν 3362, 2925, 1729, 1611, 1555, 1482, 1175, 1078, 753 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.89-5.09 (m, 4H), 6.6 (d, *J* = 7.7 Hz, 1H), 7.09 (s, 1H, OH), 7.19 (d, *J* = 7.7 Hz, 1H), 7.25-7.32 (m, 6H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 42.9, 72.3, 77.6, 111.1, 124.8, 126.8, 127.2, 127.5, 128.5, 129.4, 130.0, 135.5, 142.0, 174.2. ESI MS(m/z): 333 $(M+1)^+$. HRMS(ESI) calcd for $C_{16}H_{14}N_2O_4$ $[M+1]^+$ 333.06378, Found 333.06366.

5-chloro-3-hydroxy-1-methyl-3-(nitromethyl)indolin-2-one 3m:



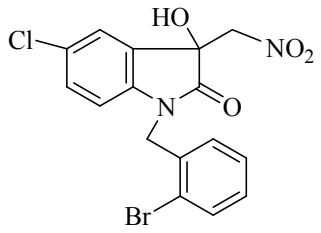
Isolated yield = 96%; IR (KBr) ν 3327, 2933, 1737, 1562, 1491, 1304, 1158, 1069, 889 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.96-4.90 (m, 2H), 6.82 (s, 1H, OH), 6.89-6.92 (d, *J* = 8.3 Hz, 1H), 7.11-7.14 (d, *J* = 8.5 Hz, 1H), 7.30 (s, 1H), 7.46 (s, 1H), 10.41 (s, 1H, NH). ^{13}C NMR (75 MHz, d_6-DMSO): δ 72.6, 78.5, 110.6, 118.2, 121.6, 122.8, 129.0, 141.2, 143.1, 175.5. ESI MS(m/z): 315 $(M+Na)^+$. HRMS(ESI) calcd for $C_{10}H_7O_5N_2F_3Na$ $[M+Na]^+$ 315.01974, Found 315.01993.

1-(4-bromobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3n:



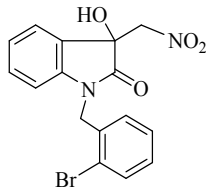
Isolated yield = 85%; IR (KBr) ν 3363, 2925, 1739, 1612, 1468, 1553, 1468, 1175, 1071, 754 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.78-5.11 (m, 4H), 6.71(d, *J* = 7.7 Hz, 1H), 7.06 (t, *J* = 7.5 Hz, 1H), 7.22-7.29 (m, 4H), 7.43-7.46 (m, 3H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 42.2, 72.2, 77.8, 109.1, 111.0, 114.3, 122.3, 124.1, 128.6, 129.1, 129.9, 131.1, 133.7, 138.3, 142.6, 174.2. ESI MS(m/z): 377 $(M+1)^+$.

1-(4-bromobenzyl)-5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3o:



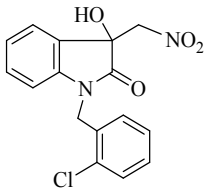
Isolated yield = 88%; IR (KBr) ν 2926, 1738, 1609, 1553, 1478, 1176, 1070,725 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.77-5.12 (m, 4H), 6.68 (d, *J* = 8.3 Hz, 1H), 7.10 (s, 1H, OH), 7.27 (d, *J* = 8.3 Hz, 3H), 7.47 (d, *J* = 9.2 Hz, 3H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 43.0, 72.5, 77.7, 110.9, 121.2, 124.8, 127.8, 128.5, 129.0, 129.3, 130.2, 130.9, 131.6, 134.4, 141.6, 174.4. ESI MS(m/z): 411 $(M+1)^+$.

1-(2-bromobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3p:



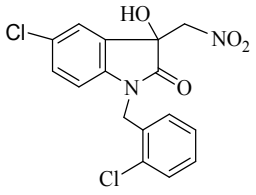
Isolated yield = 85%; IR (KBr) ν 3410, 2925, 1706, 1617, 1555, 1469, 1387, 1177, 1023,756 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.9-5.15 (m, 4H), 6.67 (d, *J* = 7.9 Hz, 1H), 7.01 (s, 1H, OH), 7.09 (t, *J* = 7.5 Hz, 1H), 7.14-7.29 (m, 4H), 7.49 (d, *J* = 7.3 Hz, 1H), 7.62 (m, 1H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 43.4, 72.5, 78.1, 109.3, 122.0, 122.9, 124.6, 127.4, 127.9, 129.4, 130.5, 132.8, 134.1, 137.9, 142.8, 174.6. ESI MS(m/z): 399 $(M+Na)^+$. HRMS(ESI) calcd for $C_{16}H_{13}O_4\ N_2Br\ Na\ [M+Na]^+$ 398.99474, Found 398.99509.

1-(2-chlorobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3q:



Isolated yield = 92%; IR (KBr) ν 3281, 2945, 1703, 1611, 1555, 1469, 1375, 1198, 1052, 759 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.94-5.14 (m, 4H), 6.69 (d, *J* = 7.9 Hz, 1H), 6.99 (s, 1H, OH), 7.09 (t, *J* = 7.5 Hz, 1H), 7.19-7.27 (m, 4H), 7.41-7.49 (dd, *J* = 7.3, 14.1 Hz, 2H), ^{13}C NMR (75 MHz, d_6-DMSO): δ 40.7, 72.3, 77.7, 108.9, 122.5, 124.1, 126.8, 127.3, 128.5, 129.0, 130.1, 131.9, 132.1, 137.8, 142.6, 174.4. ESI MS(m/z): 333 $(M+1)^+$, 355 $(M+Na)^+$. HRMS(ESI) calcd for $C_{16}H_{14}N_2O_4Cl$ $[M+1]^+$ 333.06365, Found 333.06366

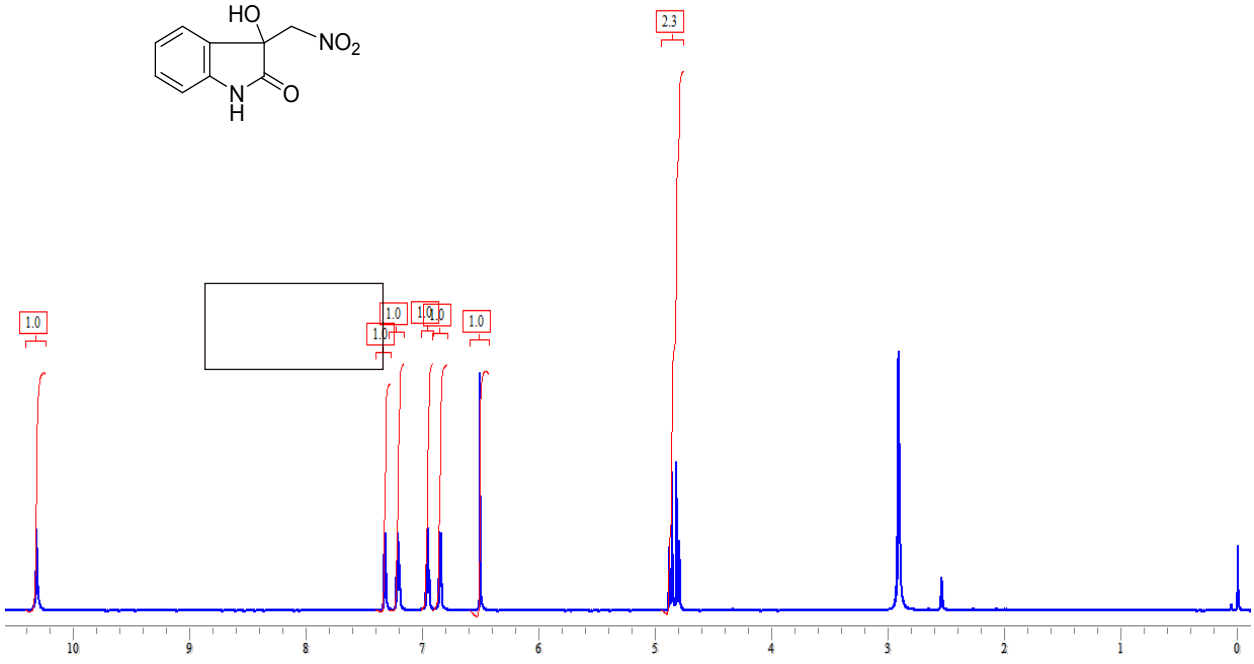
5-chloro-1-(2-chlorobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3r:

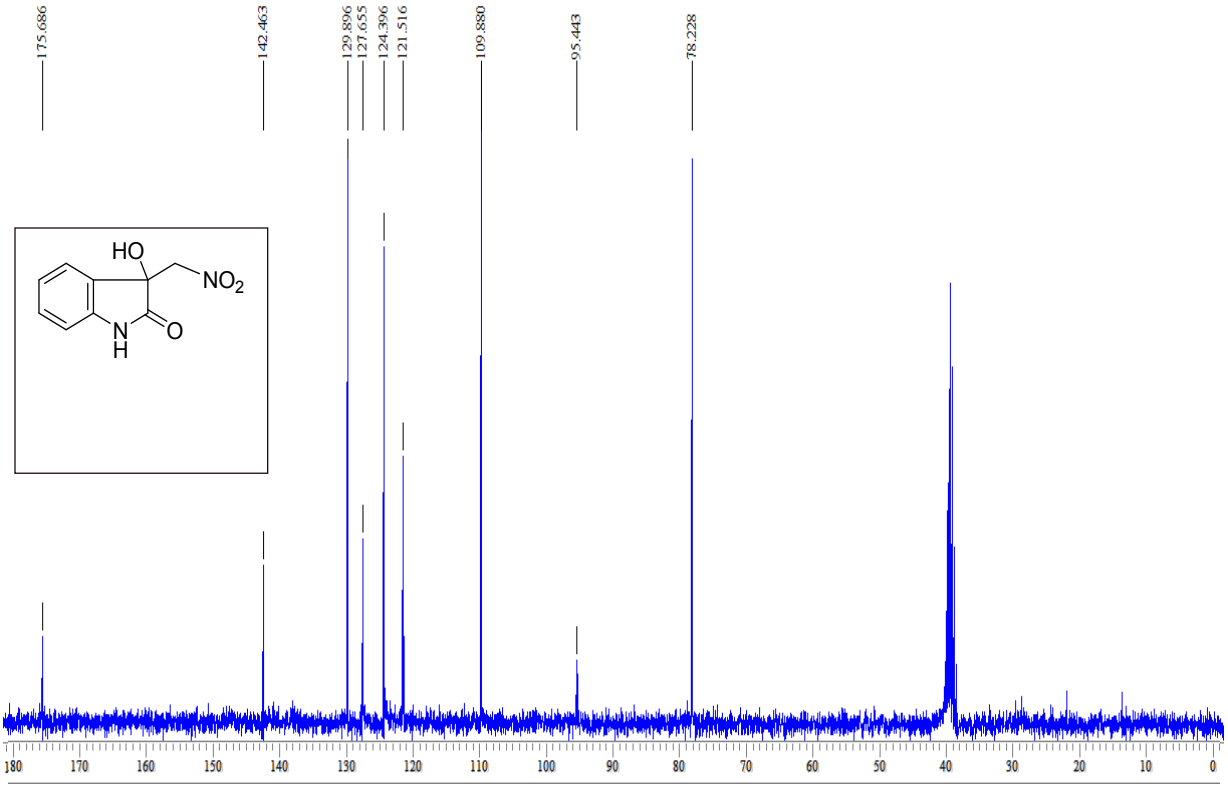


Isolated yield = 91%; IR (KBr) ν 3645, 3226, 1704, 1611, 1555, 1481, 1183, 1073, 742 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$+d_6-DMSO): δ 4.94-5.14 (m, 4H), 6.64 (d, *J* = 8.3 Hz, 1H), 7.13 (s, 1H, OH), 7.20-7.28 (m, 4H), 7.43 (d, *J* = 6.9 Hz, 1H), 7.49 (d, *J* = 2.0 Hz, 1H). ^{13}C NMR (75 MHz, d_6-DMSO): δ 40.9, 72.1, 77.2, 110.1, 124.4, 126.7, 127.3, 127.5, 128.5, 129.0, 129.8, 131.6, 131.9, 137.0, 141.2, 174.0. ESI MS(m/z): 389 $(M+Na)^+$.

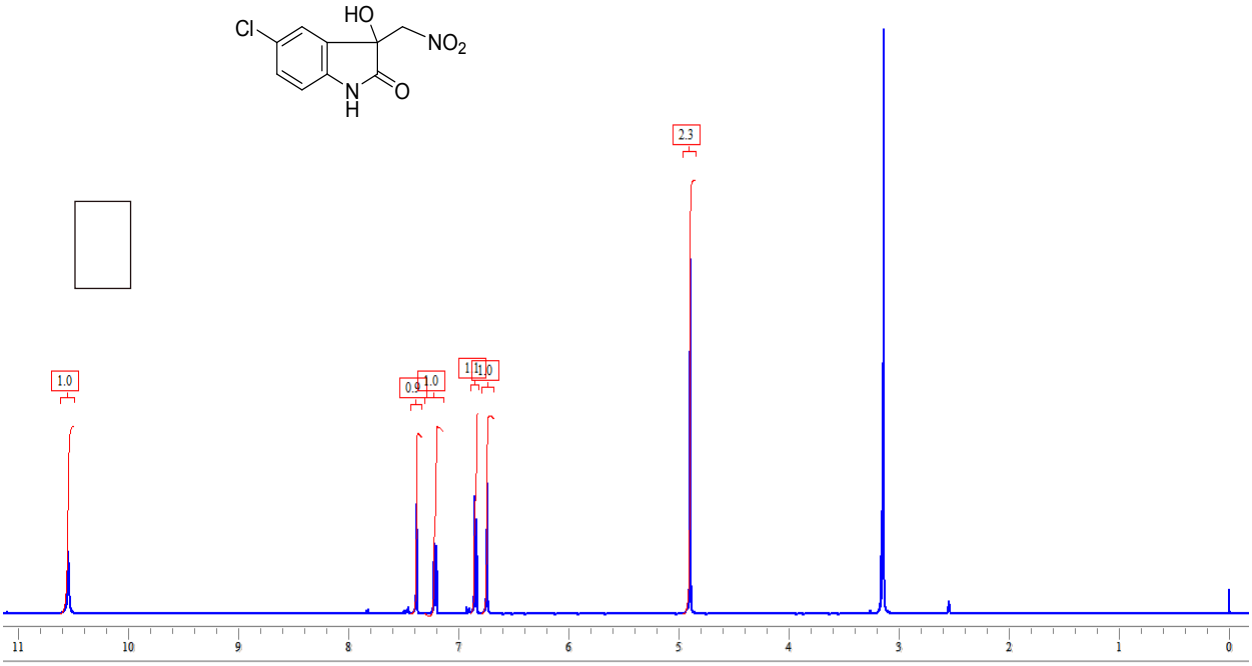
D. ^{1}H NMR and ^{13}C NMR spectra for compounds (3a-r)

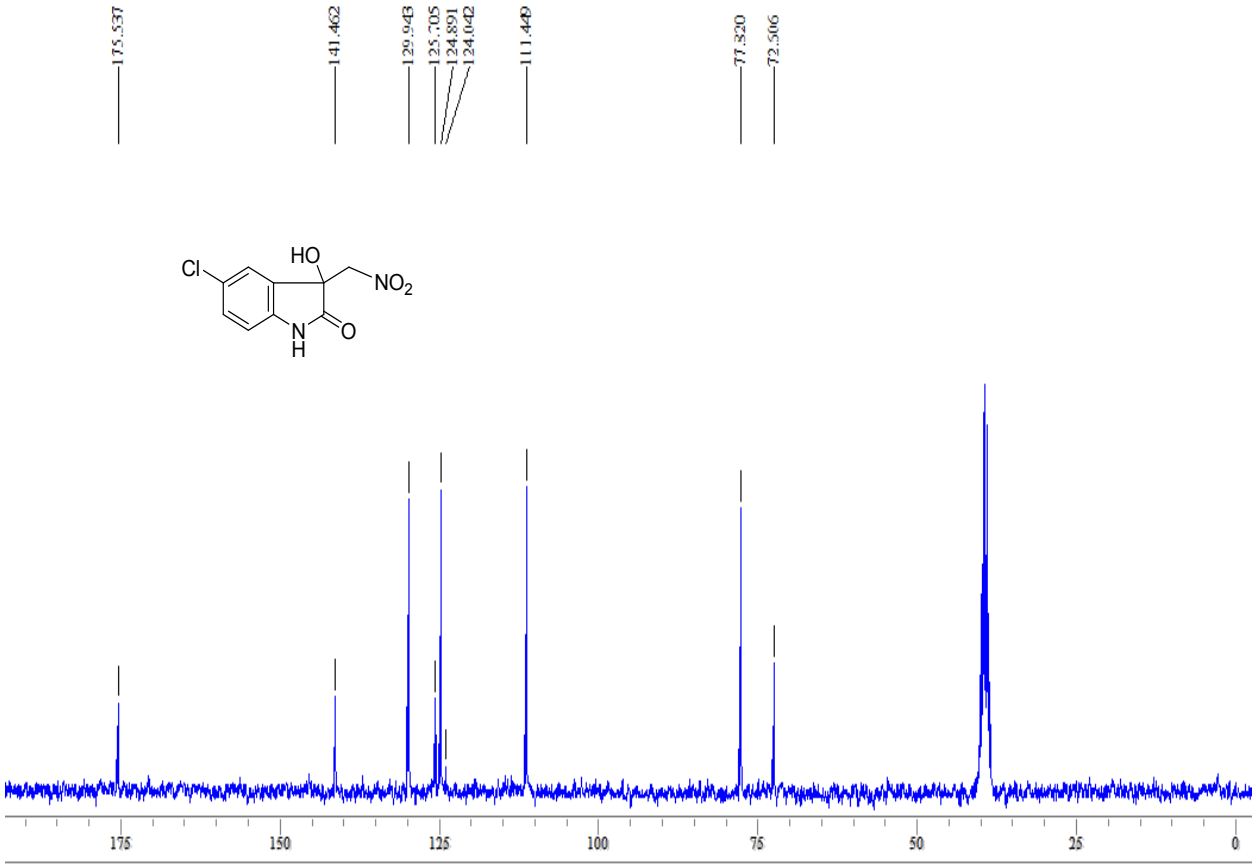
3-hydroxy-3-(nitromethyl)indolin-2-one 3a:



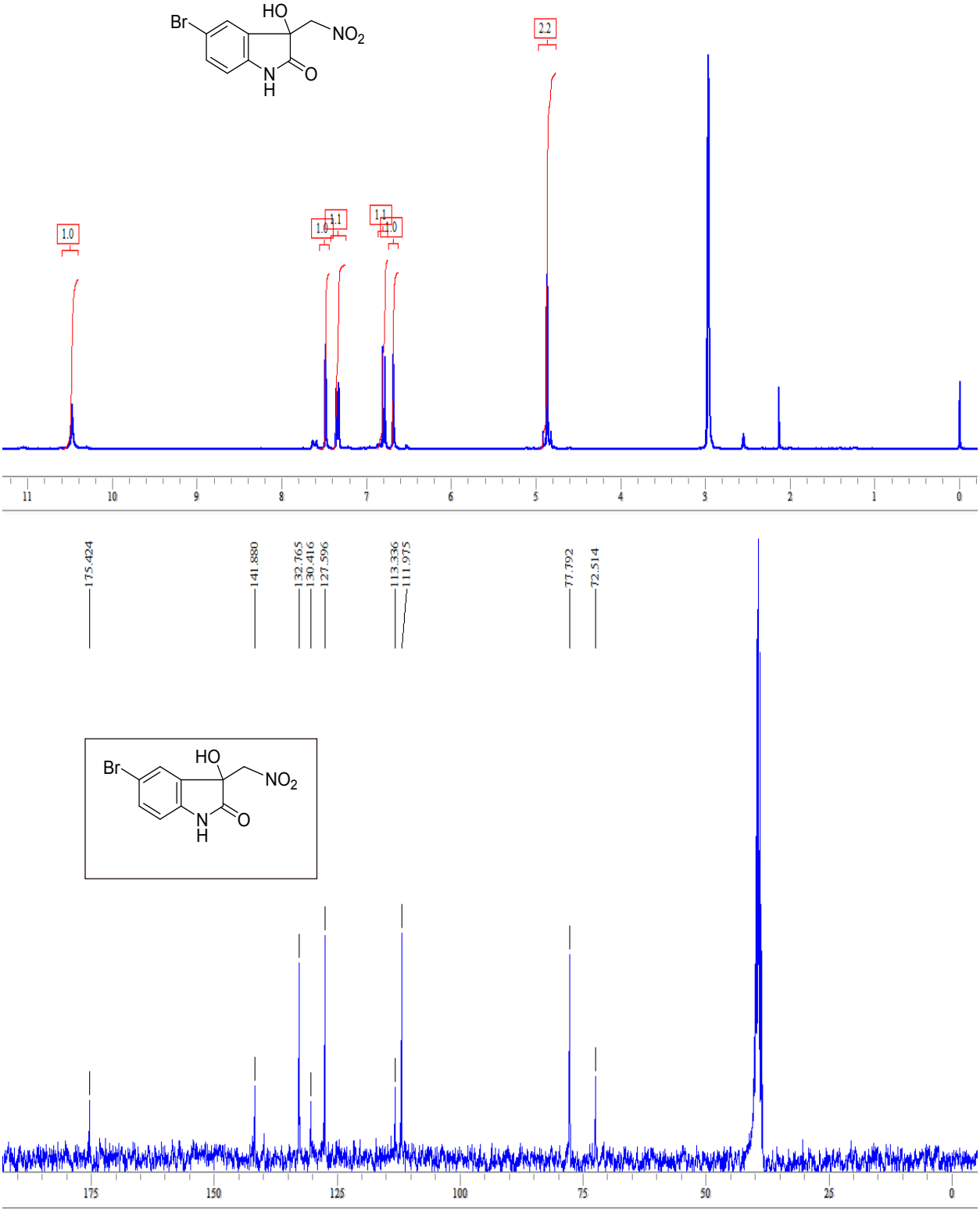


5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3b:

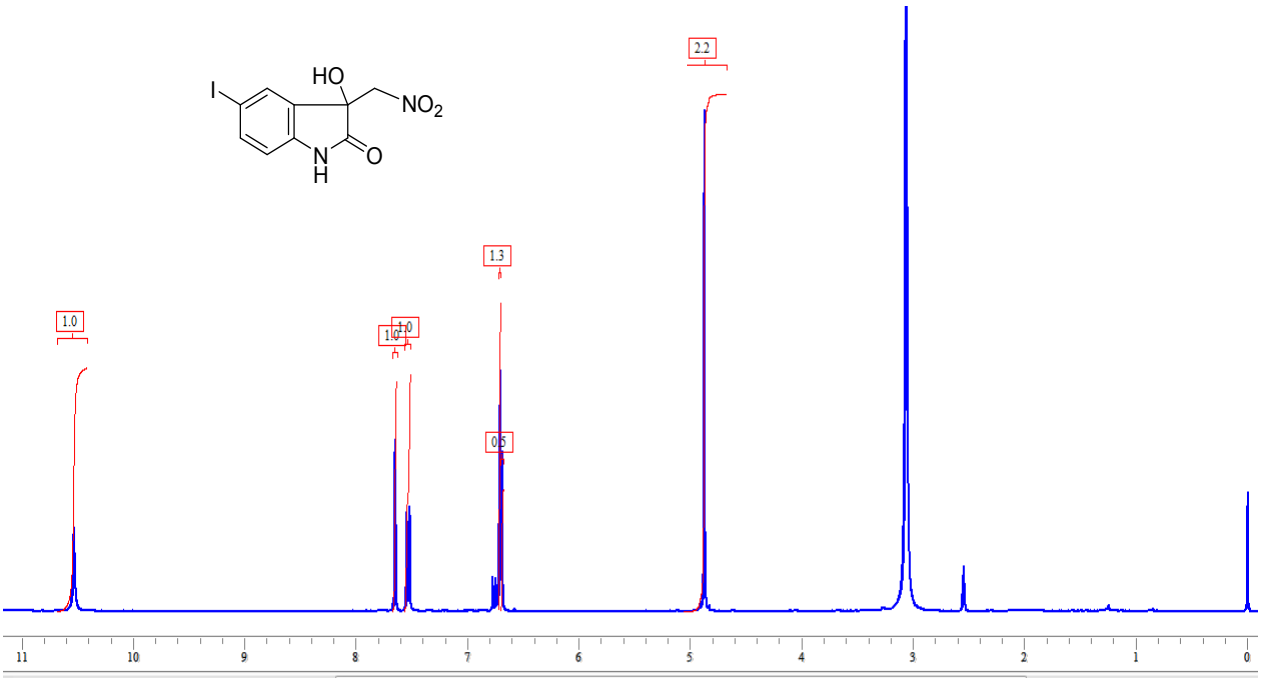


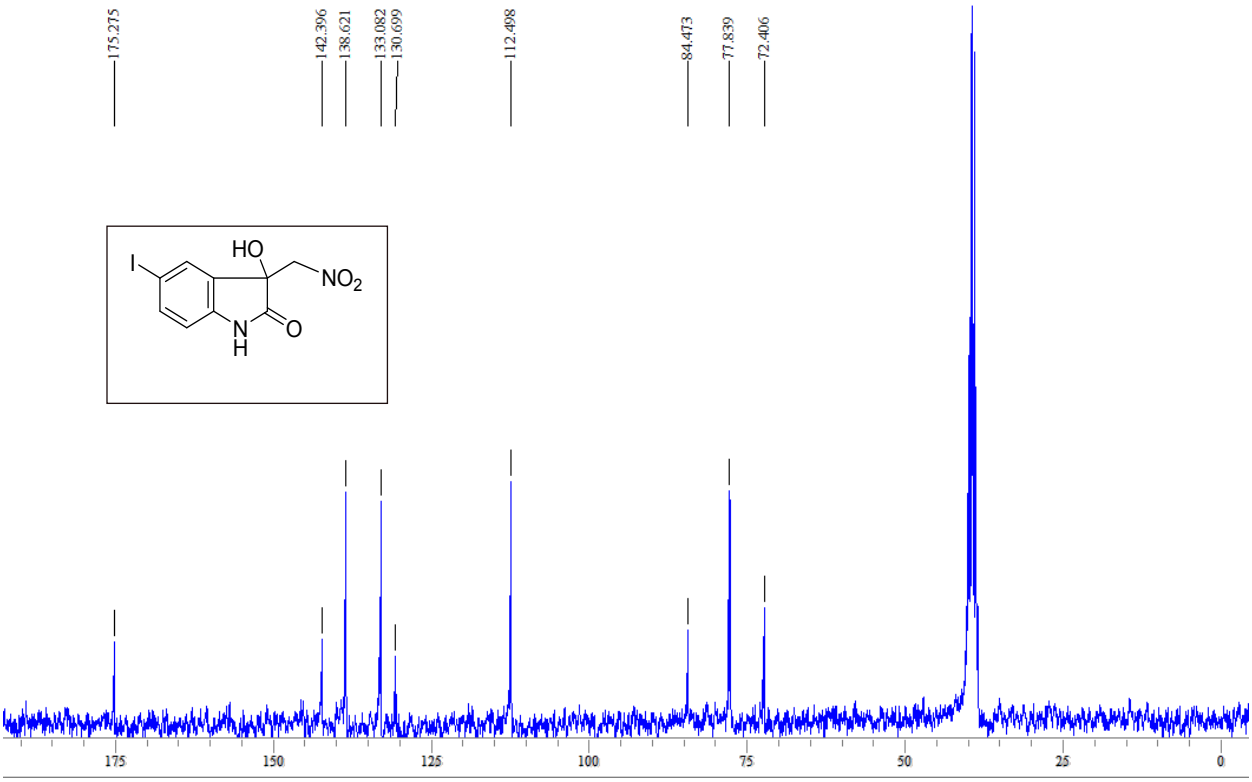


5-bromo-3-hydroxy-3-(nitromethyl)2H-indolin-2-one 3c:

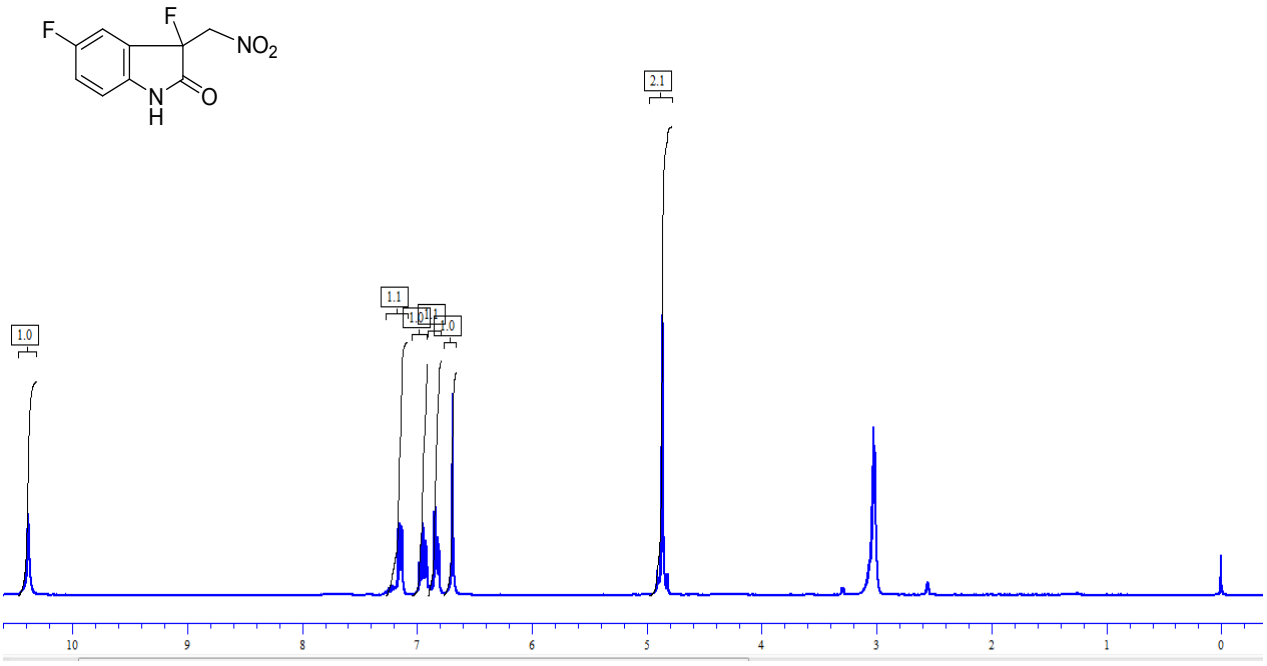


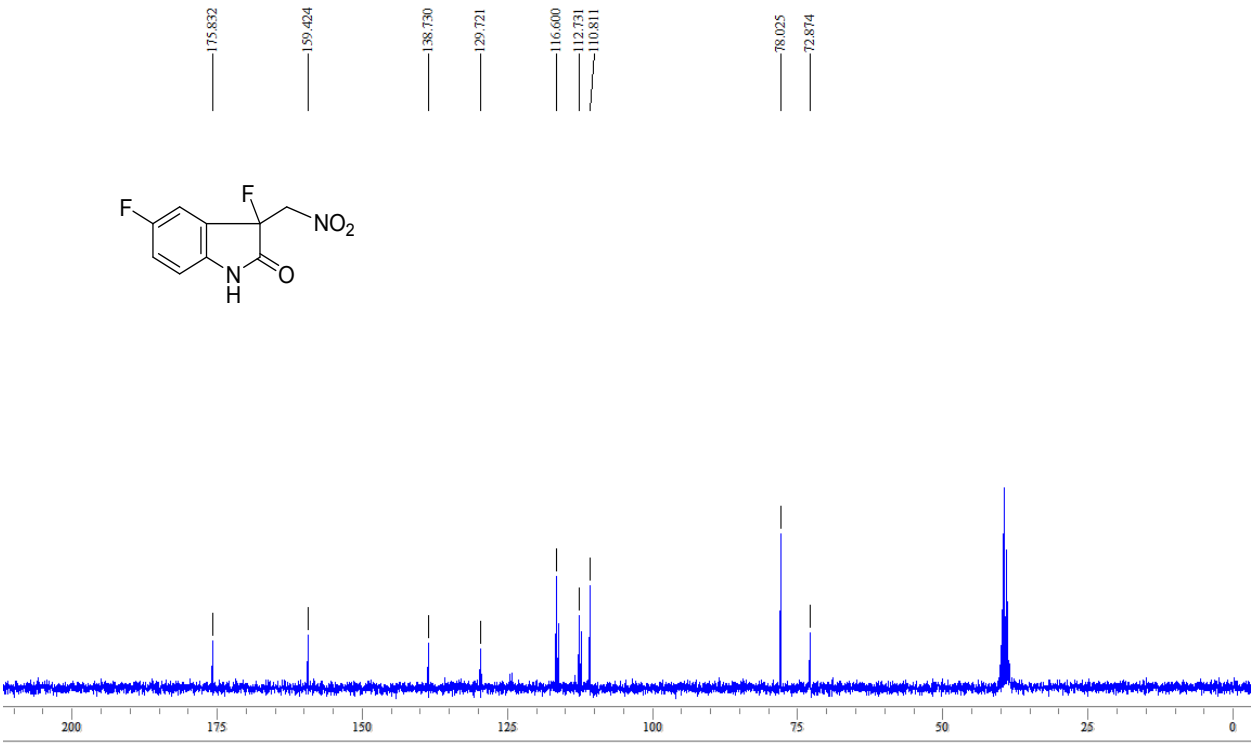
3-hydroxy-5-iodo-3-(nitromethyl)indolin-2-one 3d:



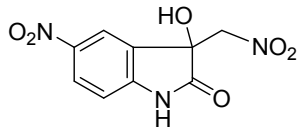


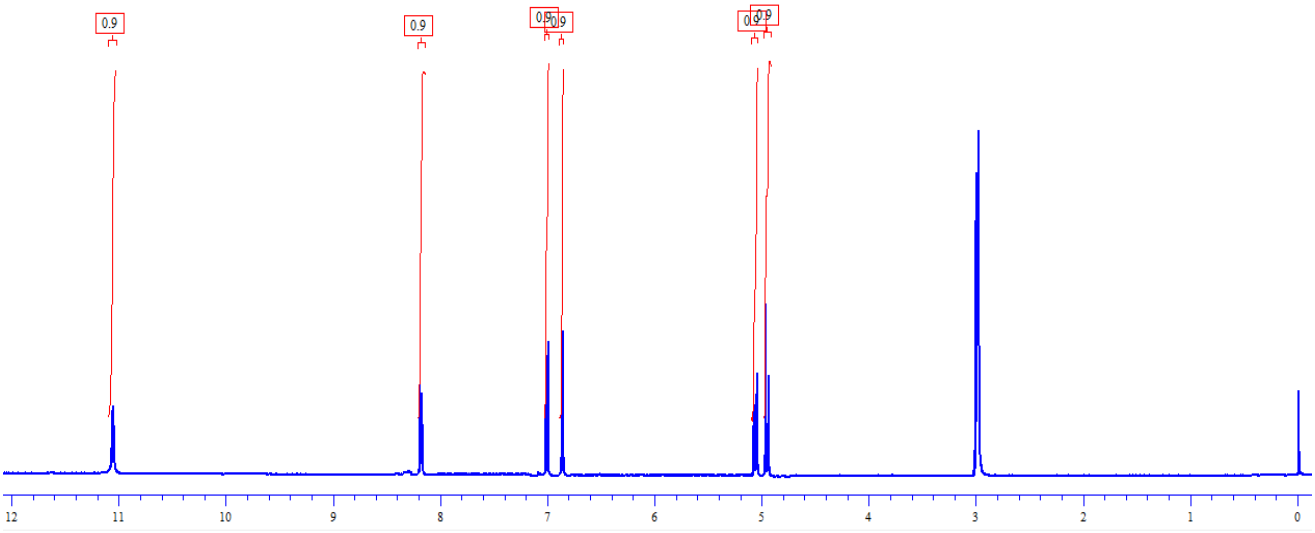
5-fluoro-3-hydroxy-3-(nitromethyl)indolin-2-one 3e:

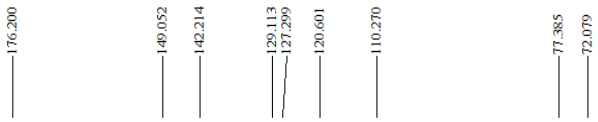


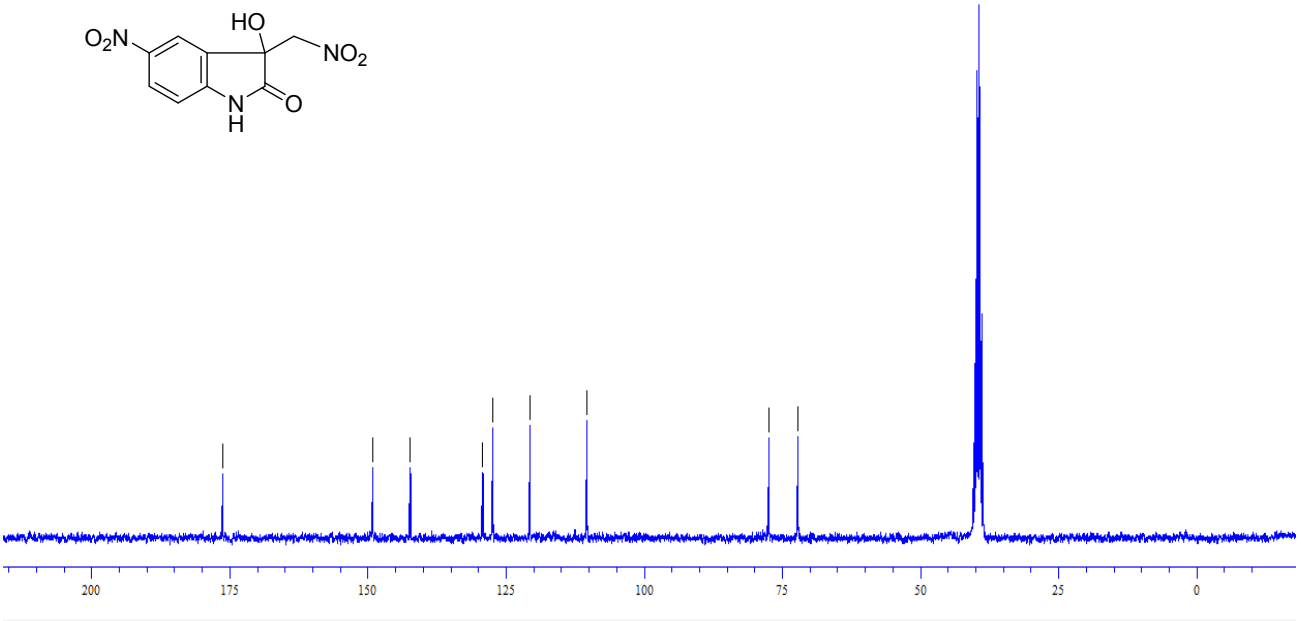


3-hydroxy-5-nitro-3-(nitromethyl)indolin-2-one 3f:

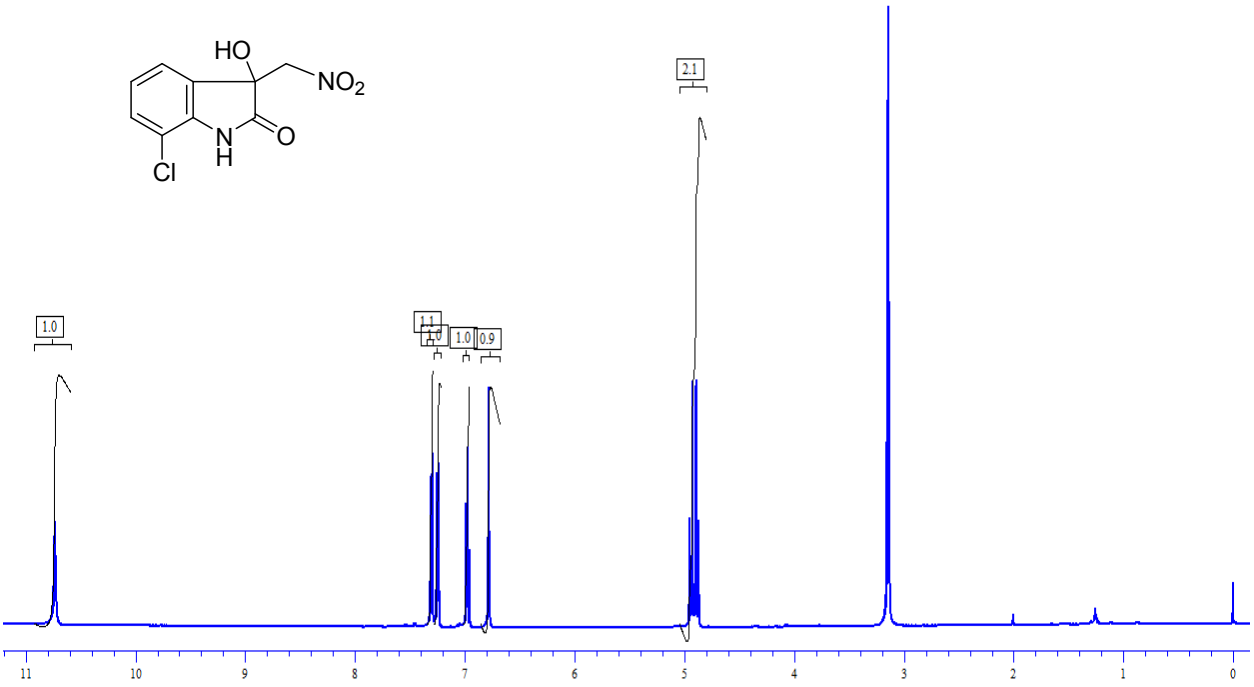


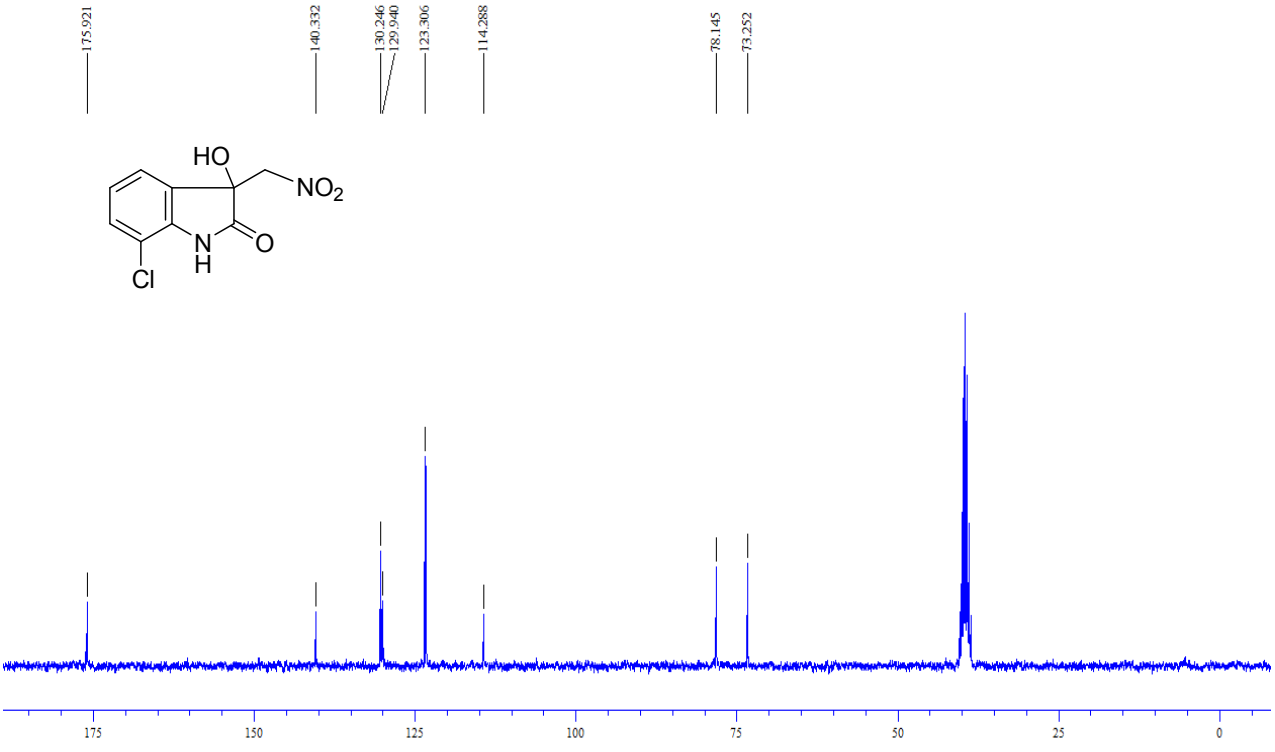




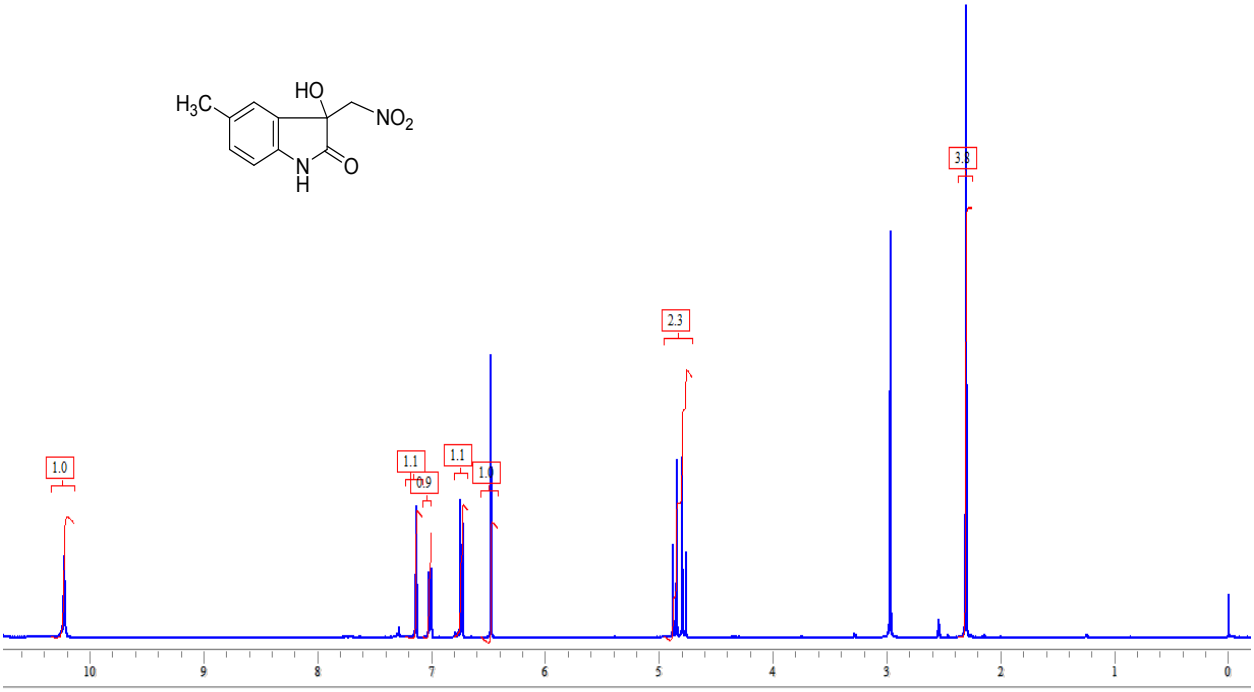


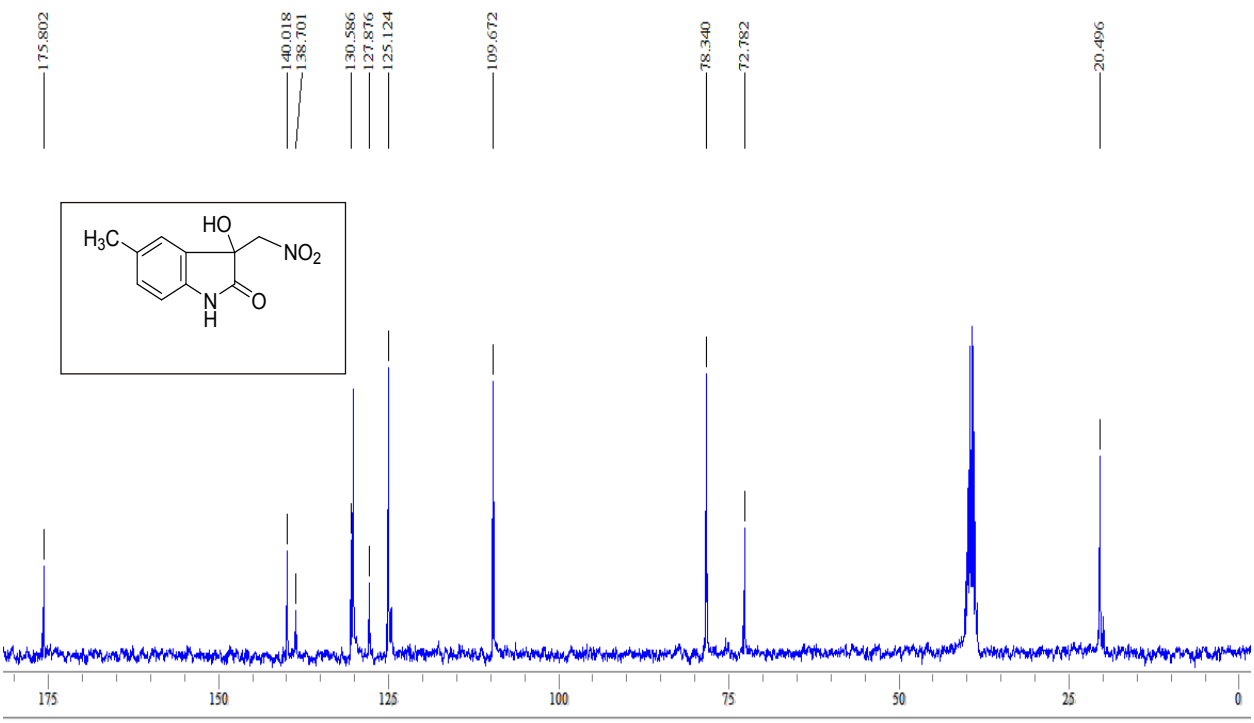
7-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3g:



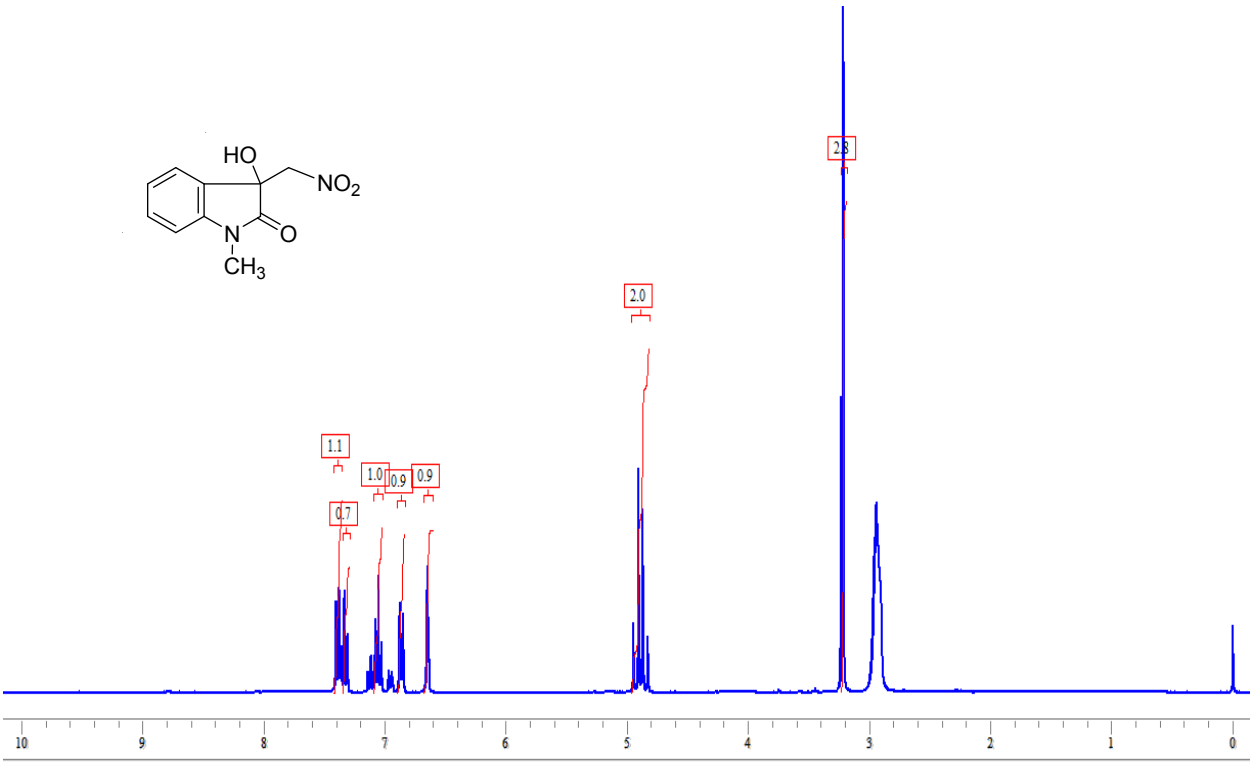


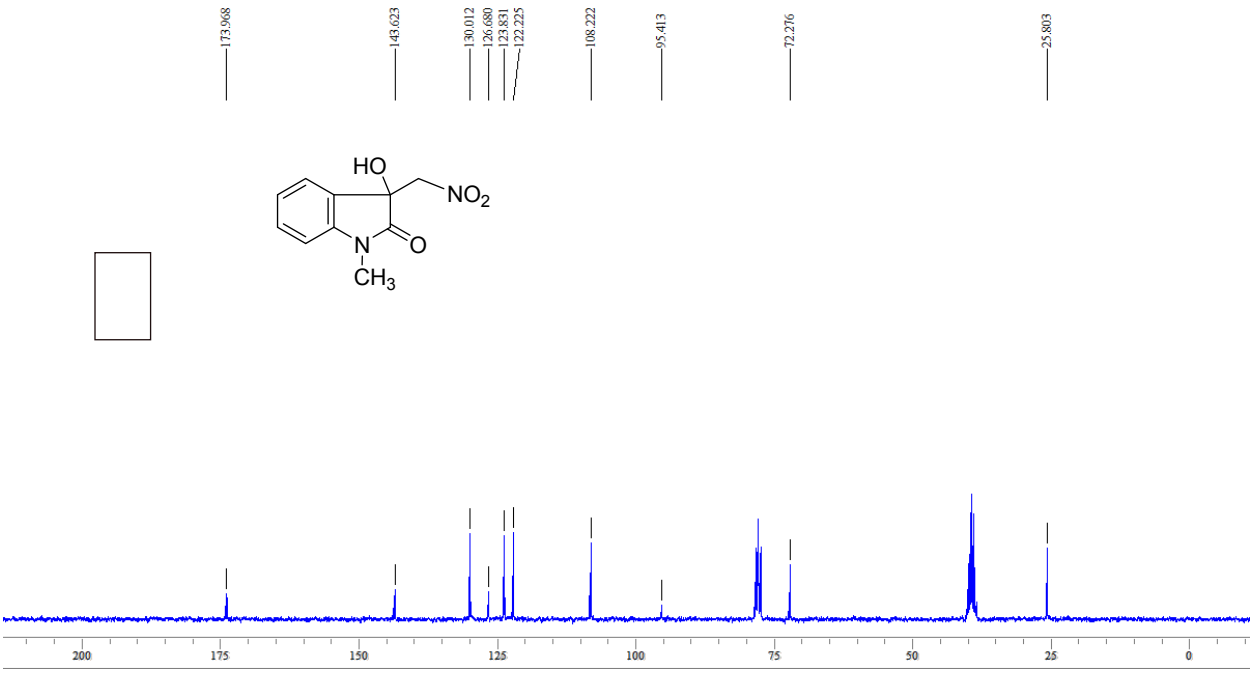
3-hydroxy-5-methyl-3-(nitromethyl)2H-indolin-2-one 3h:



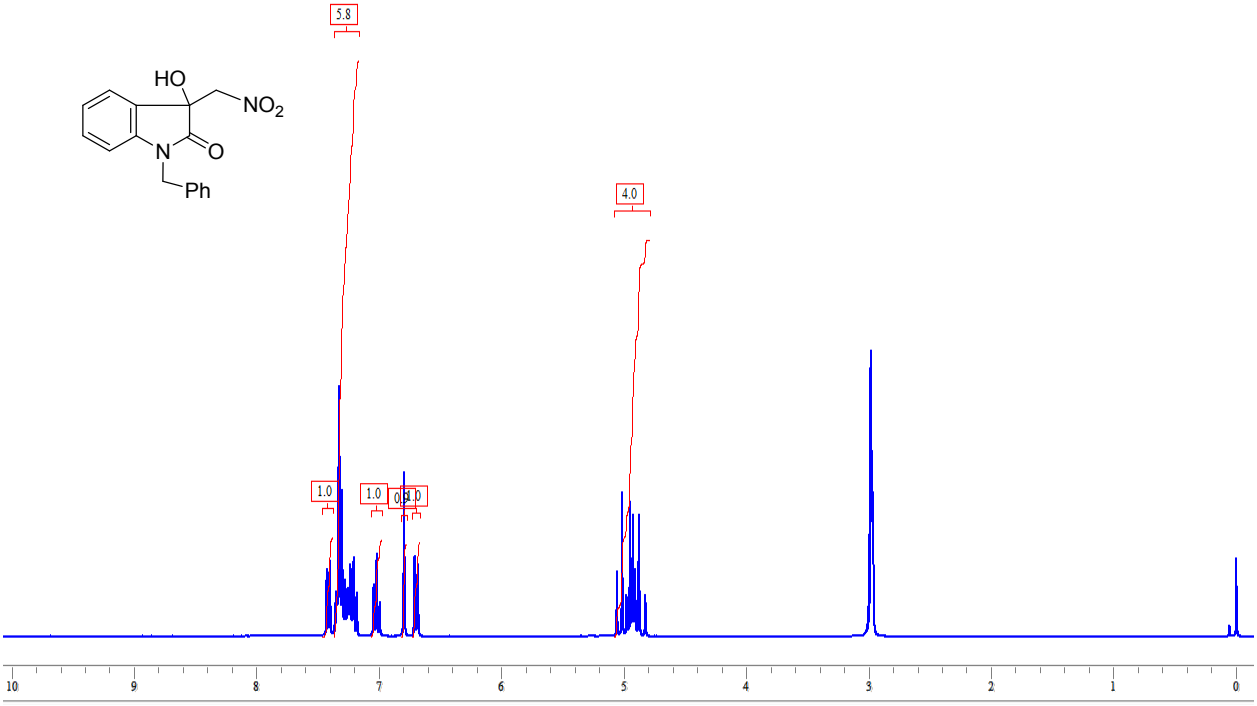


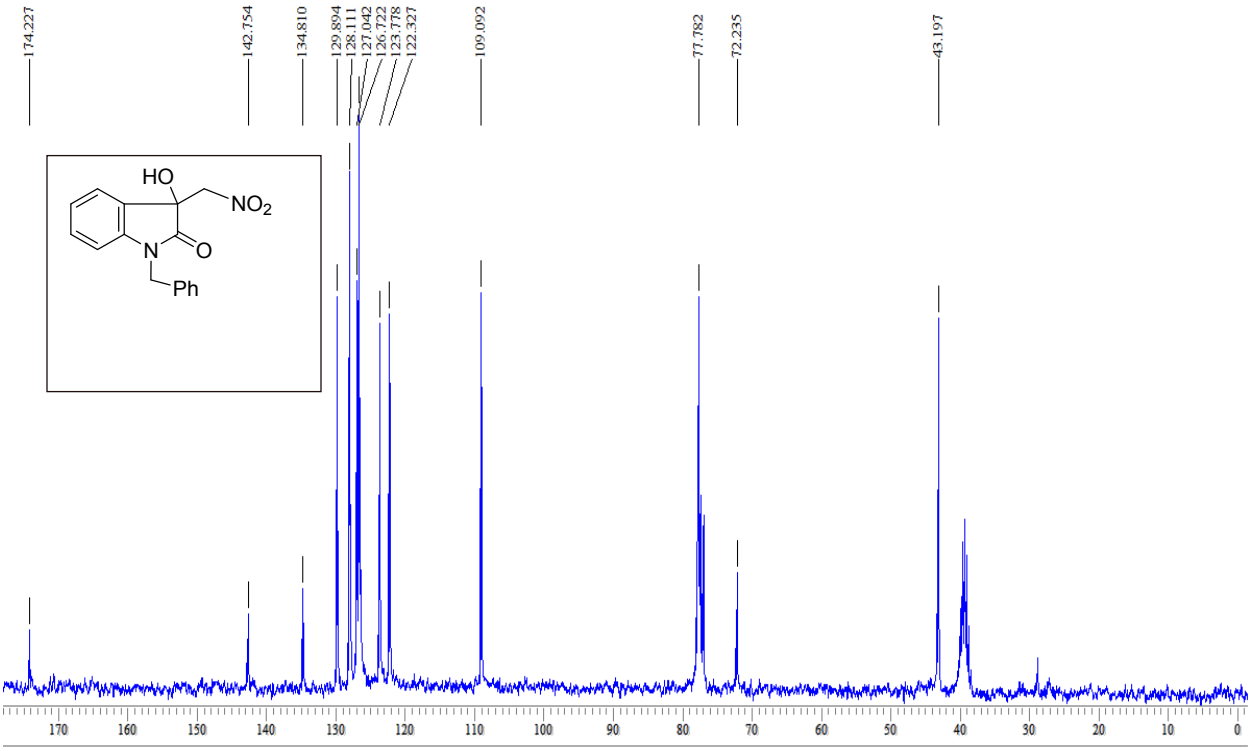
3-hydroxy-1-methyl-3-(nitromethyl)indolin-2-one 3i:



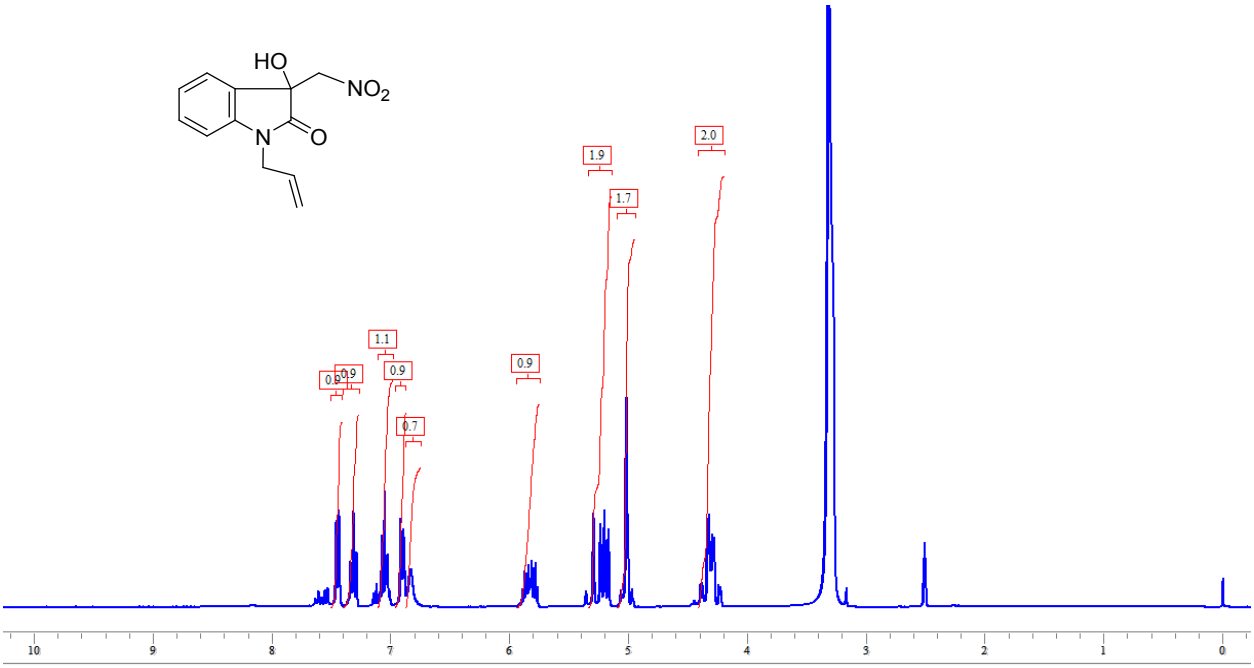


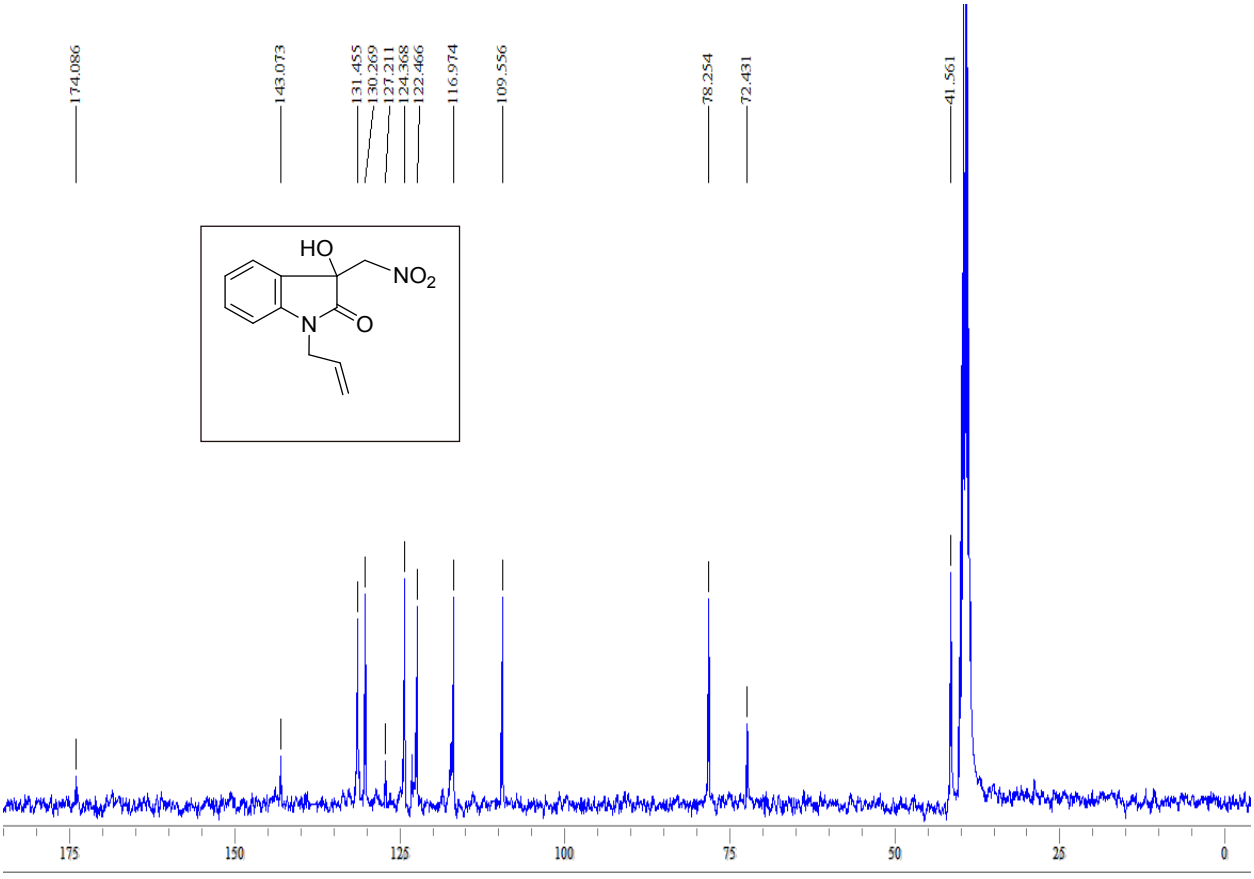
1-benzyl-3-hydroxy-3-(nitromethyl)indolin-2-one 3j:



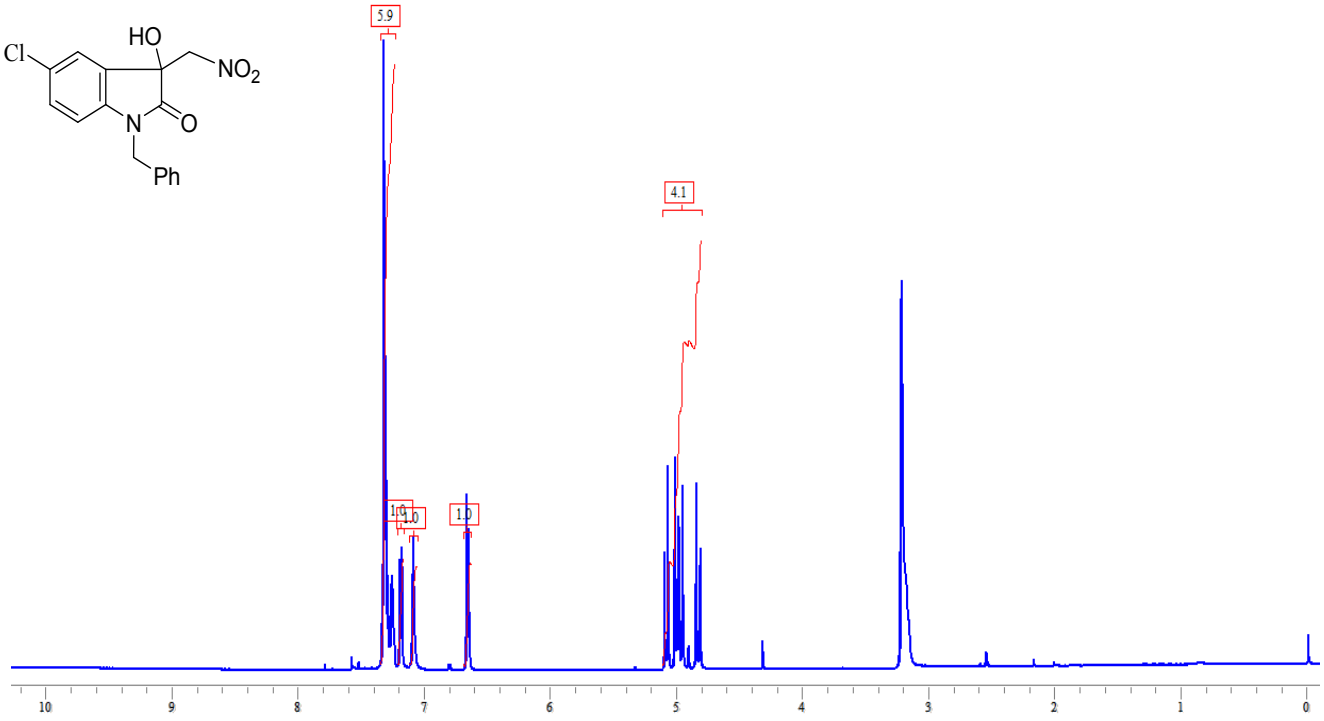


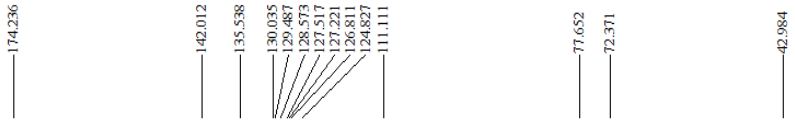
1-allyl-3-hydroxy-3-(nitromethyl)indolin-2-one 3k:

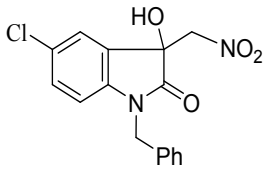

HO
NO2
N
O
2.0
1.9
1.7
1.1
0.9
0.9
0.9
0.9
0.7
10
9
8
7
6
5
4
3
2
1
0

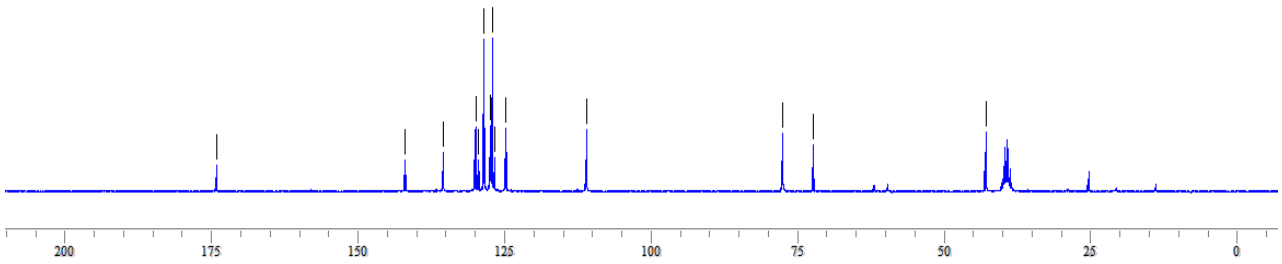

174.086
143.073
131.455
130.269
127.211
124.368
122.466
116.974
109.556
78.254
72.431
41.561
HO
NO2
N
O
175
150
125
100
75
50
25
0

1-benzyl-5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3l:

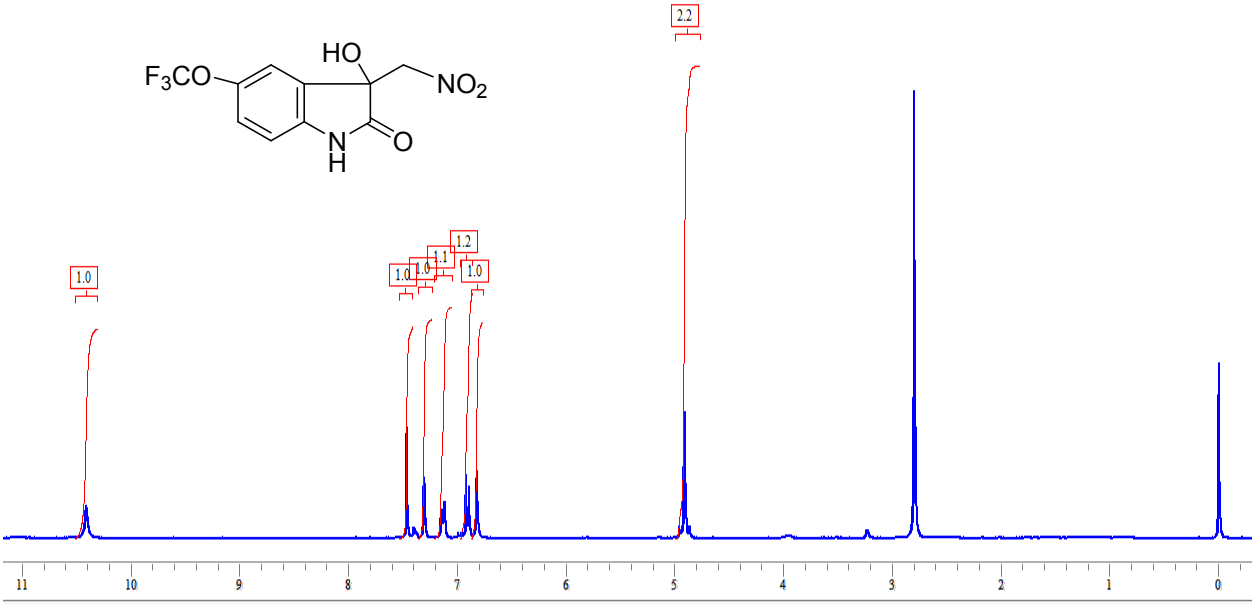


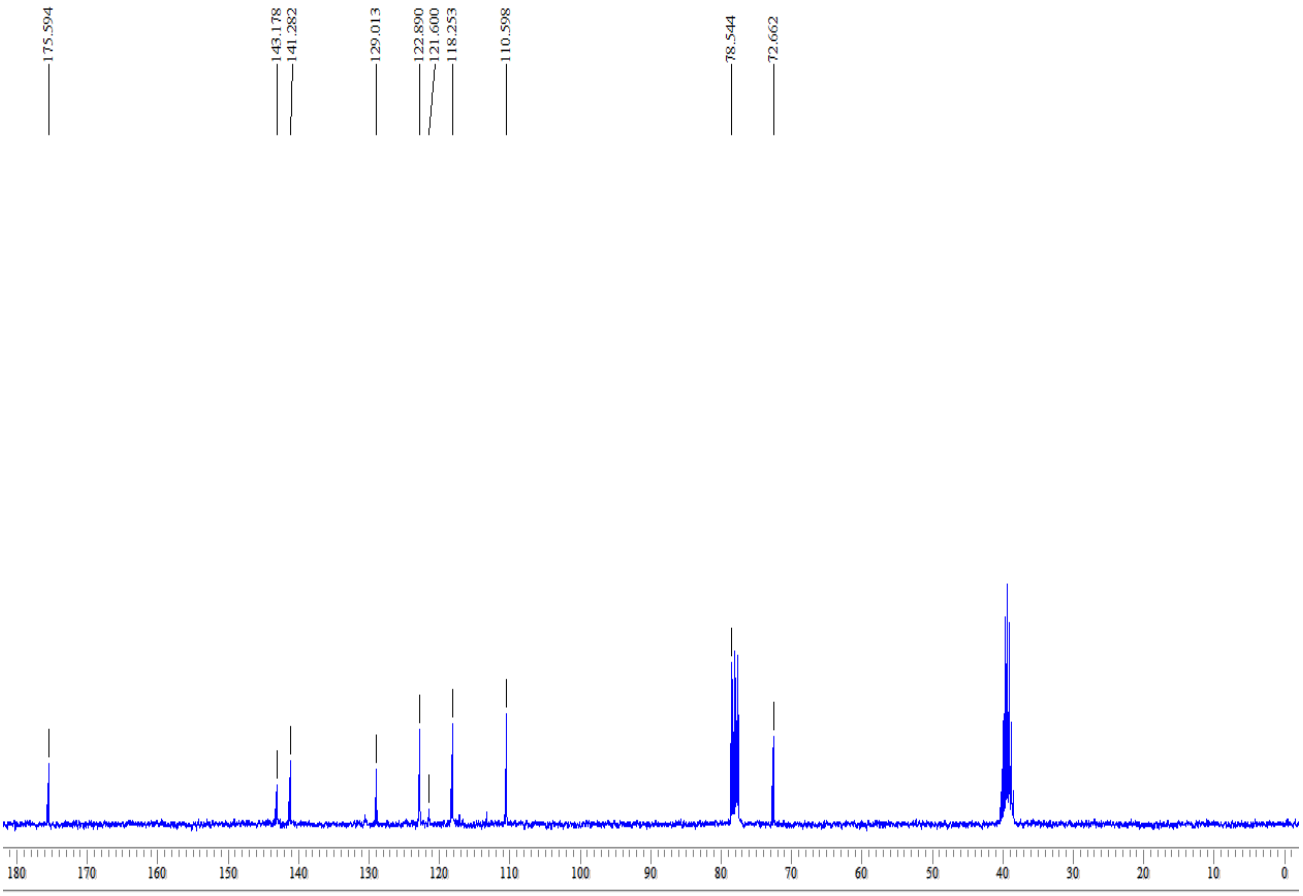




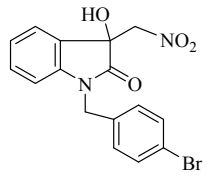


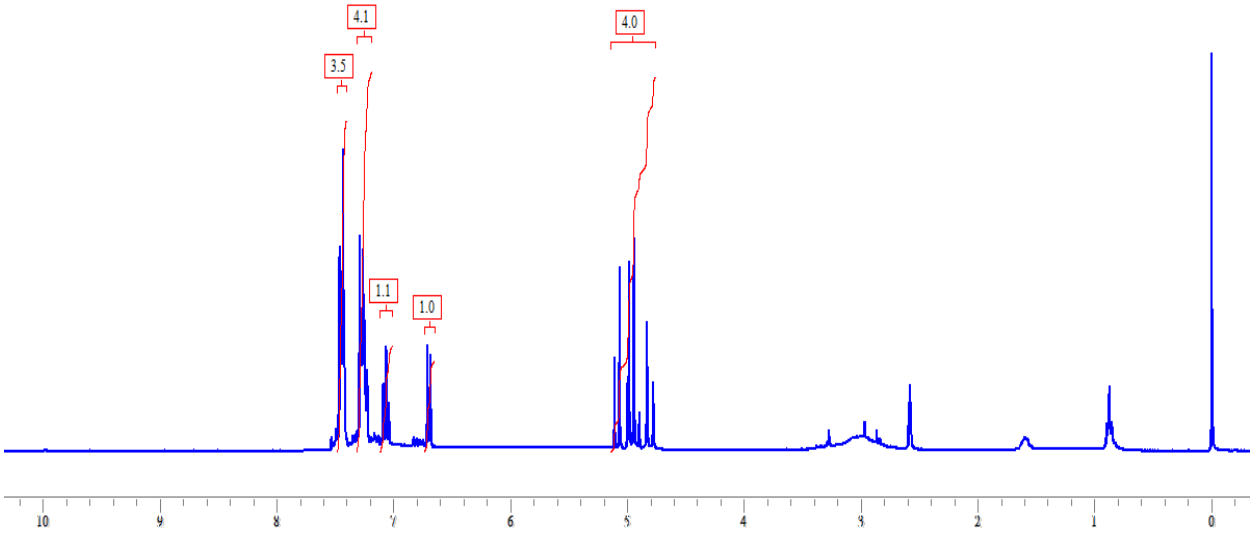
5-chloro-3-hydroxy-1-methyl-3-(nitromethyl)indolin-2-one 3m:

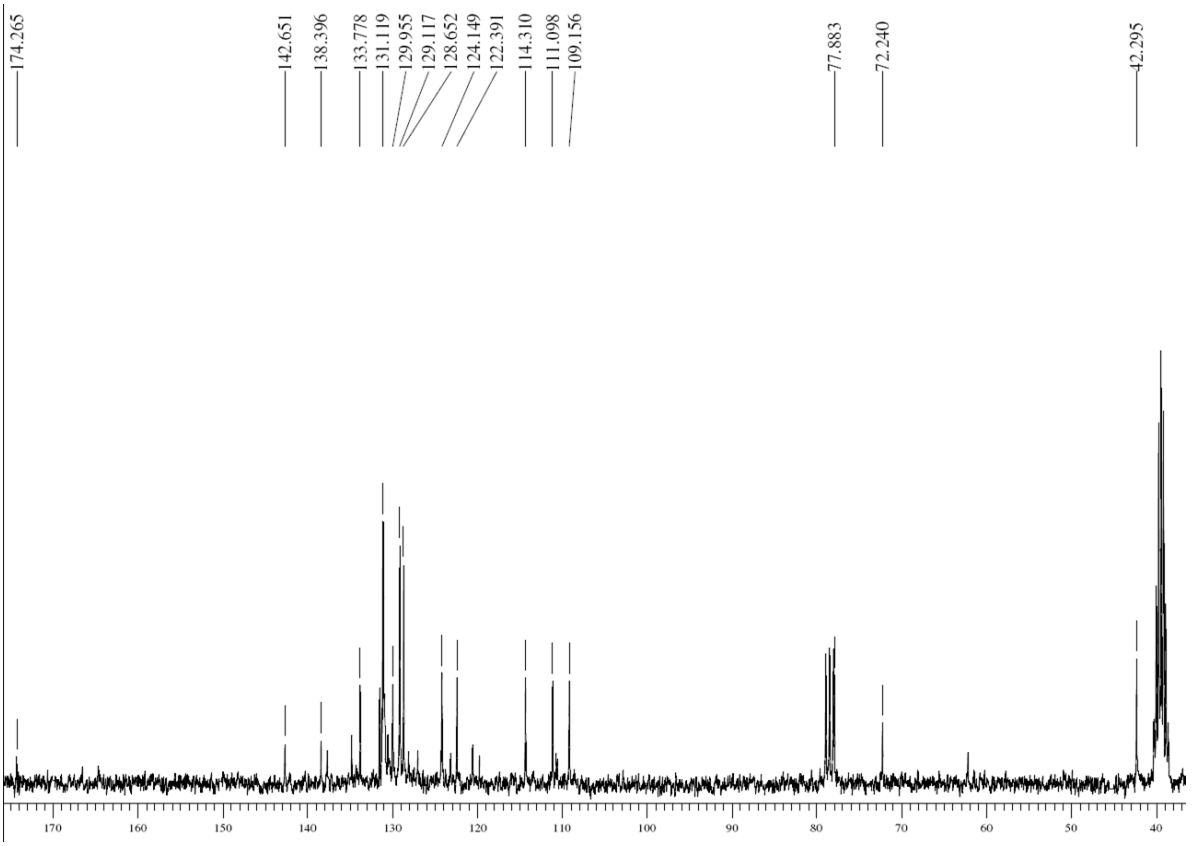




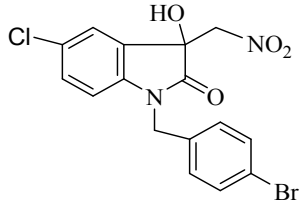
1-(4-bromobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3n:

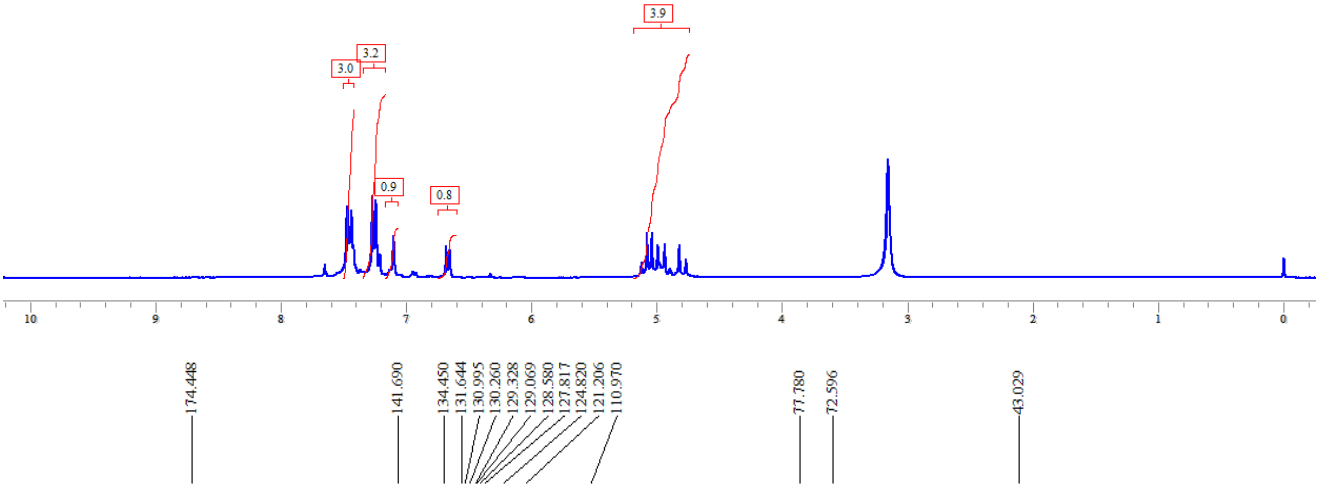


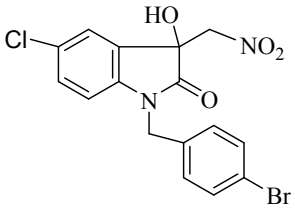


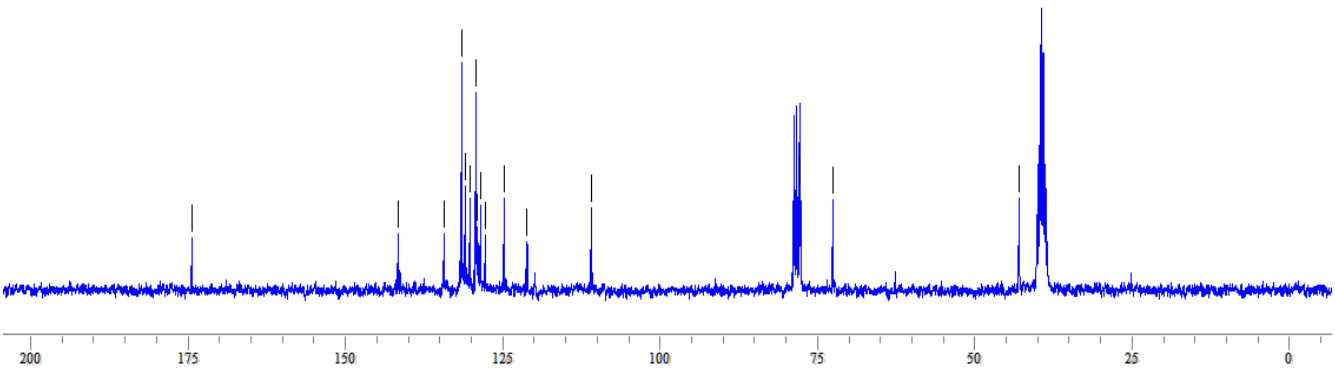


1-(4-bromobenzyl)-5-chloro-3-hydroxy-3-(nitromethyl)indolin-2-one 3o:

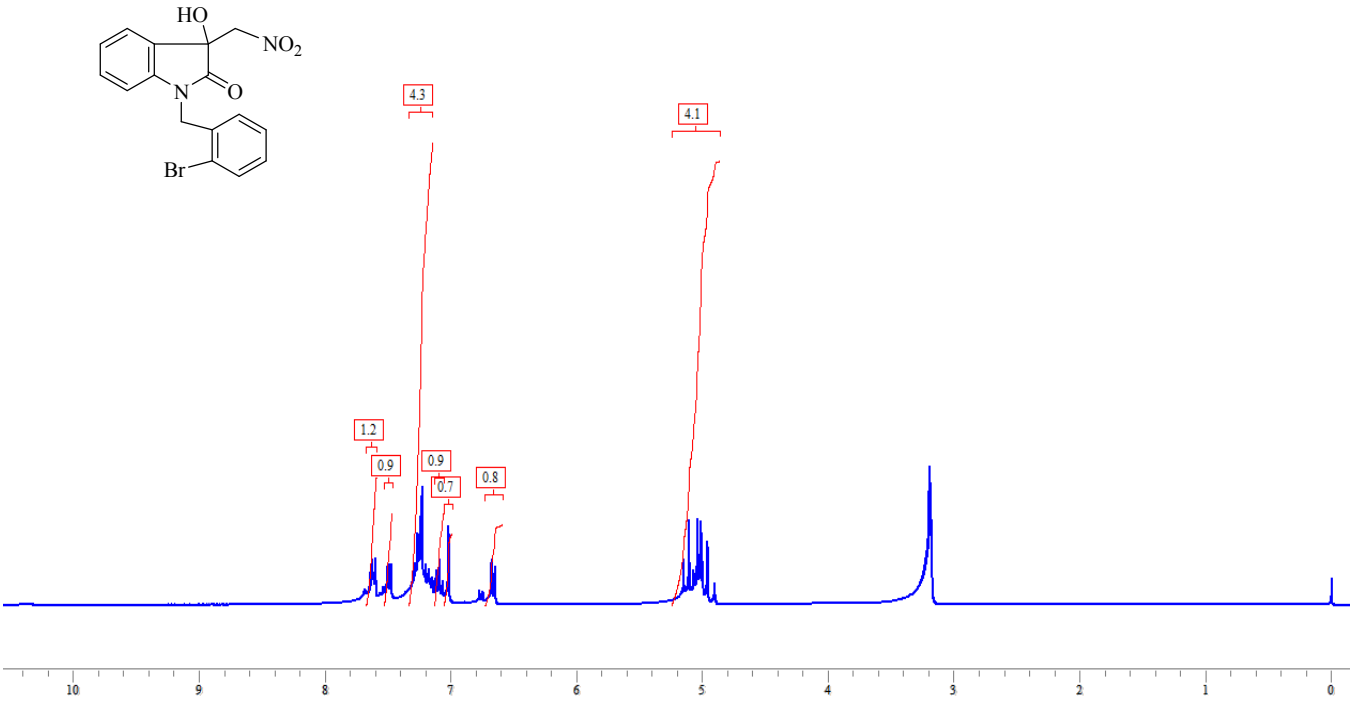


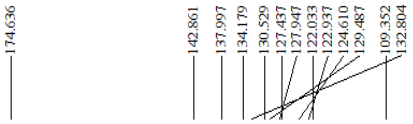


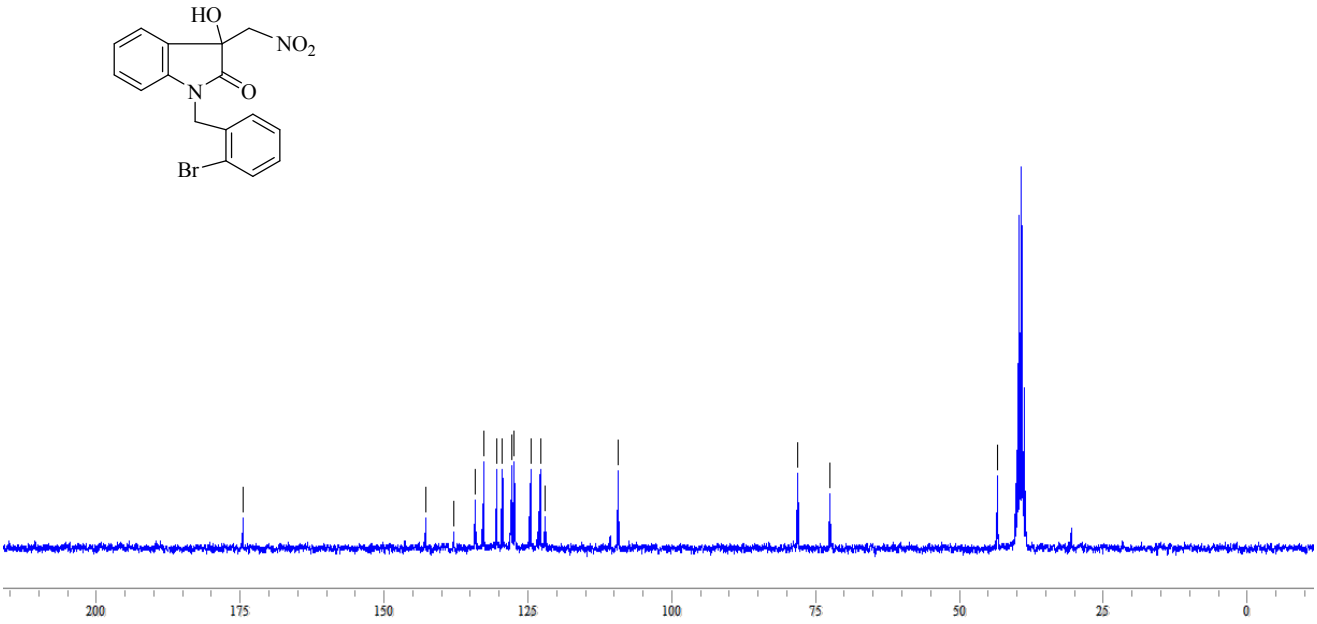




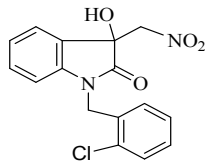
1-(2-bromobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3p:

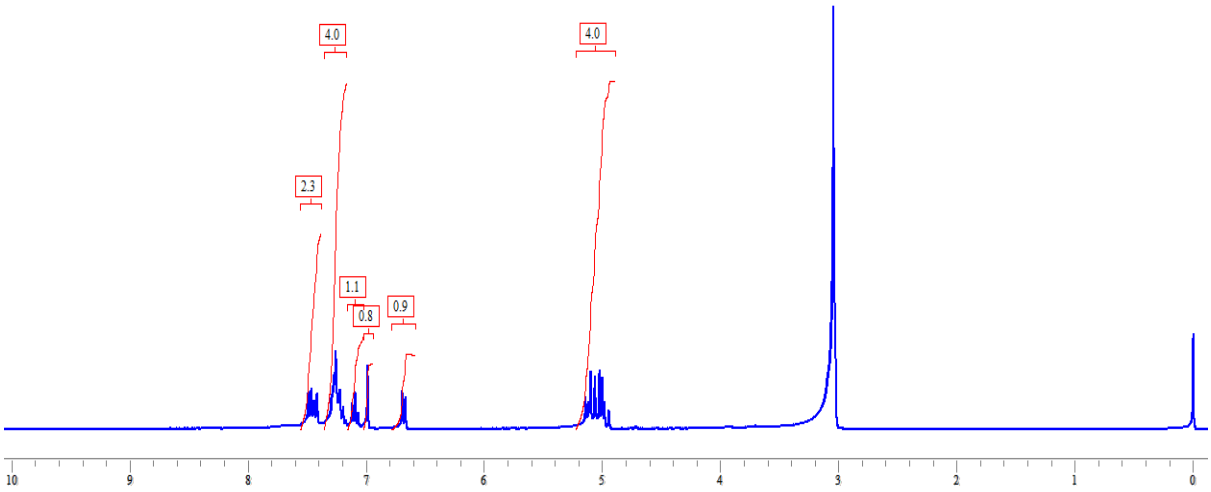


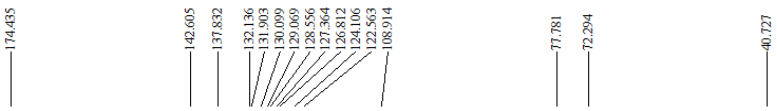


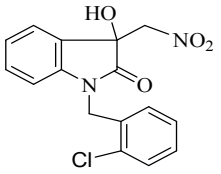


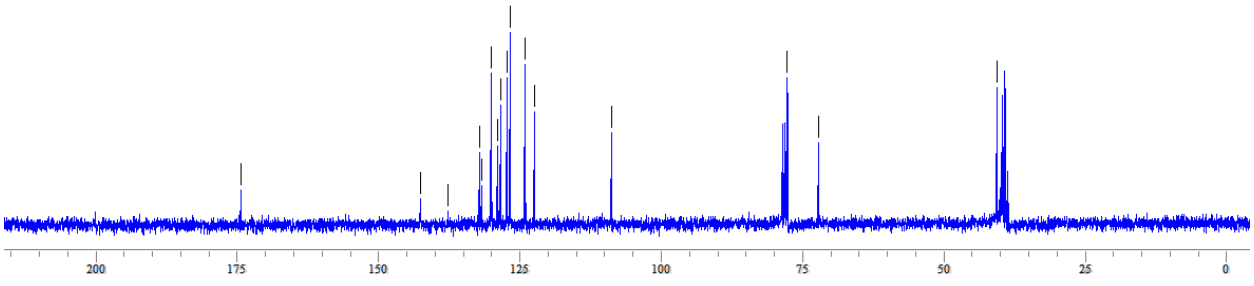
1-(2-chlorobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3q:



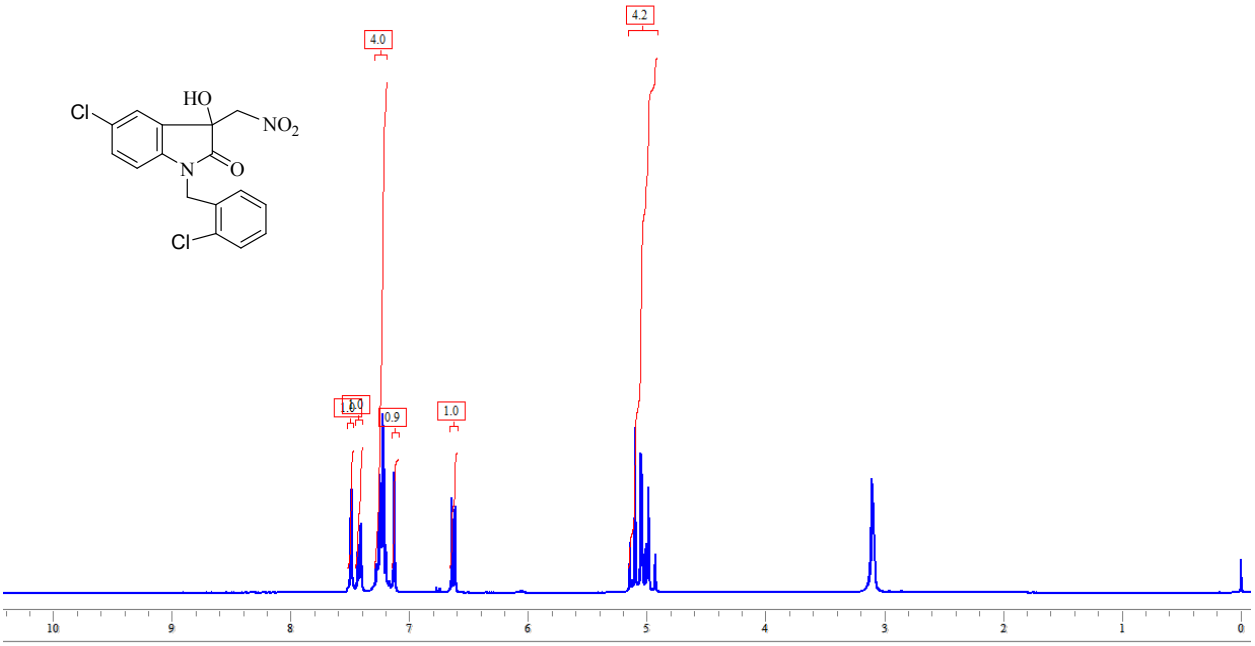


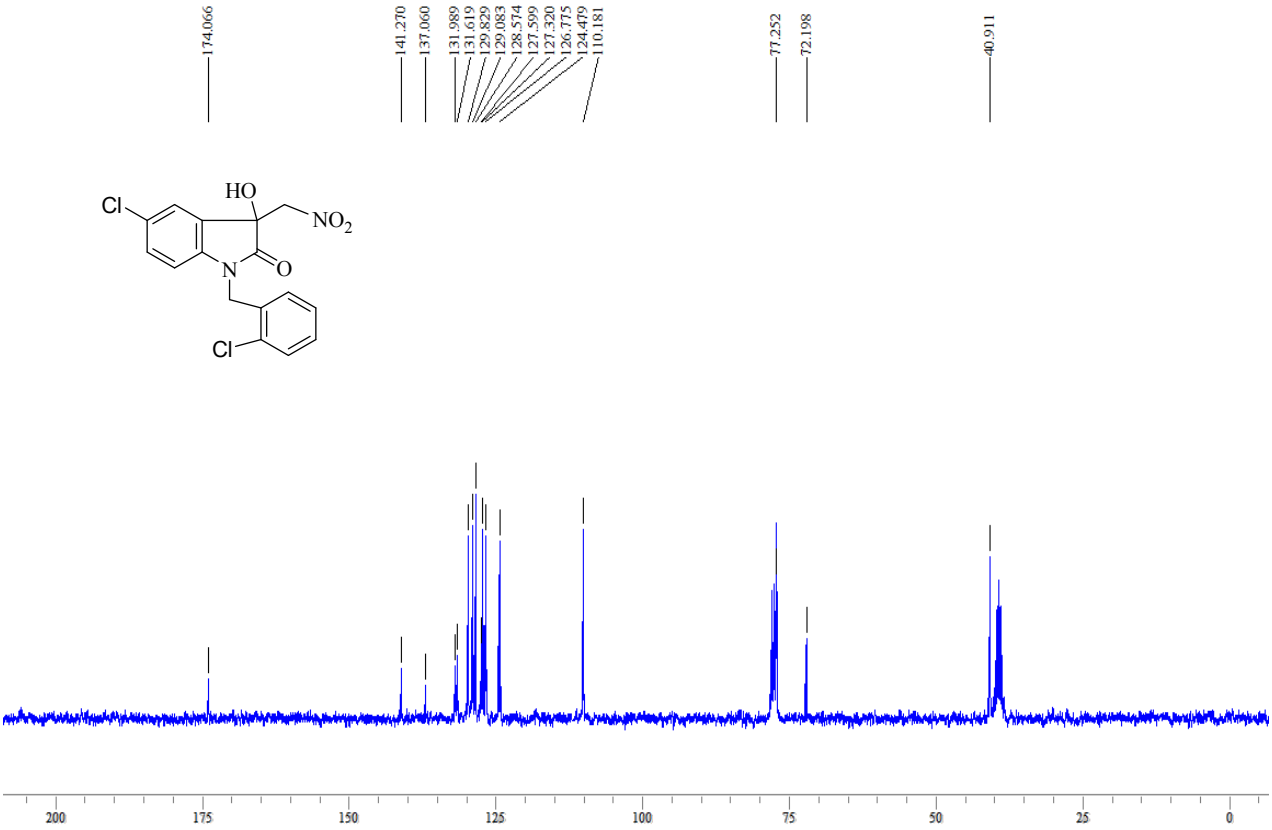






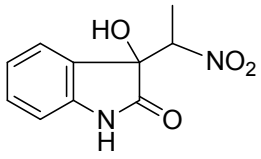
5-chloro-1-(2-chlorobenzyl)-3-hydroxy-3-(nitromethyl)indolin-2-one 3r:





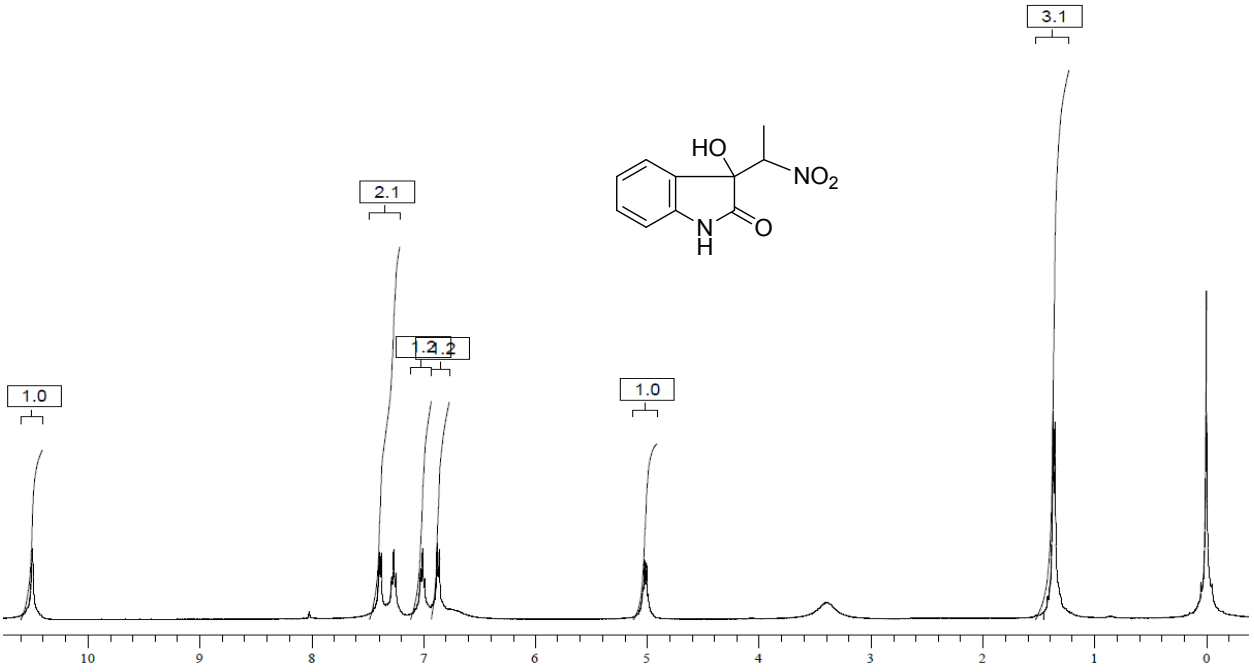
E. ^{1}H NMR and ^{13}C NMR spectra for compound 5

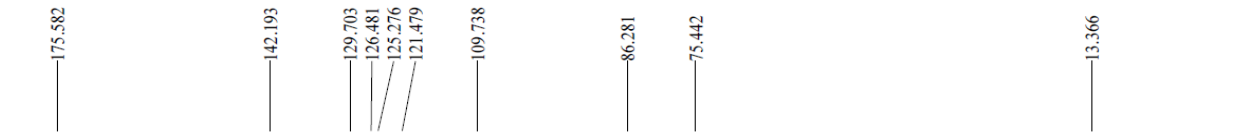
3-hydroxy-3-(1-nitroethyl)indolin-2-one 5:

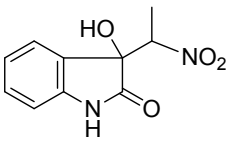


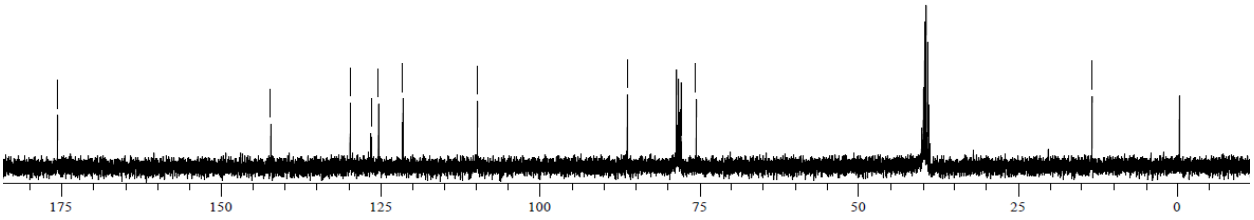
Isolated yield = 90%; diastereomeric ratio (98:2). IR (KBr) ν 3346, 1713, 1617, 1558, 1479, 1188, 1087, 833 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$+d_6-DMSO): δ 1.3 (d, 3H), 5.0 (q, 1H), 6.87-6.85 (d, *J* = 7.5 Hz, 1H), 7.0 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.39-7.37 (d, *J* = 7.3 Hz, 1H), 10.4 (s, 1H, NH) major. ^{13}C NMR (100 MHz, d_6-DMSO): 13.3, 75.4, 86.2, 109.7, 121.4, 125.2, 126.4, 129.7, 142.1, 175.5. ESI MS (m/z): 245 $(M+Na)^+$. HRMS(ESI) calcd for $C_{10}H_{10}N_2O_4Na$ $[M+Na]^+$ 245.05308, Found 245.05328.

3-hydroxy-3-(1-nitroethyl)indolin-2-one 5:

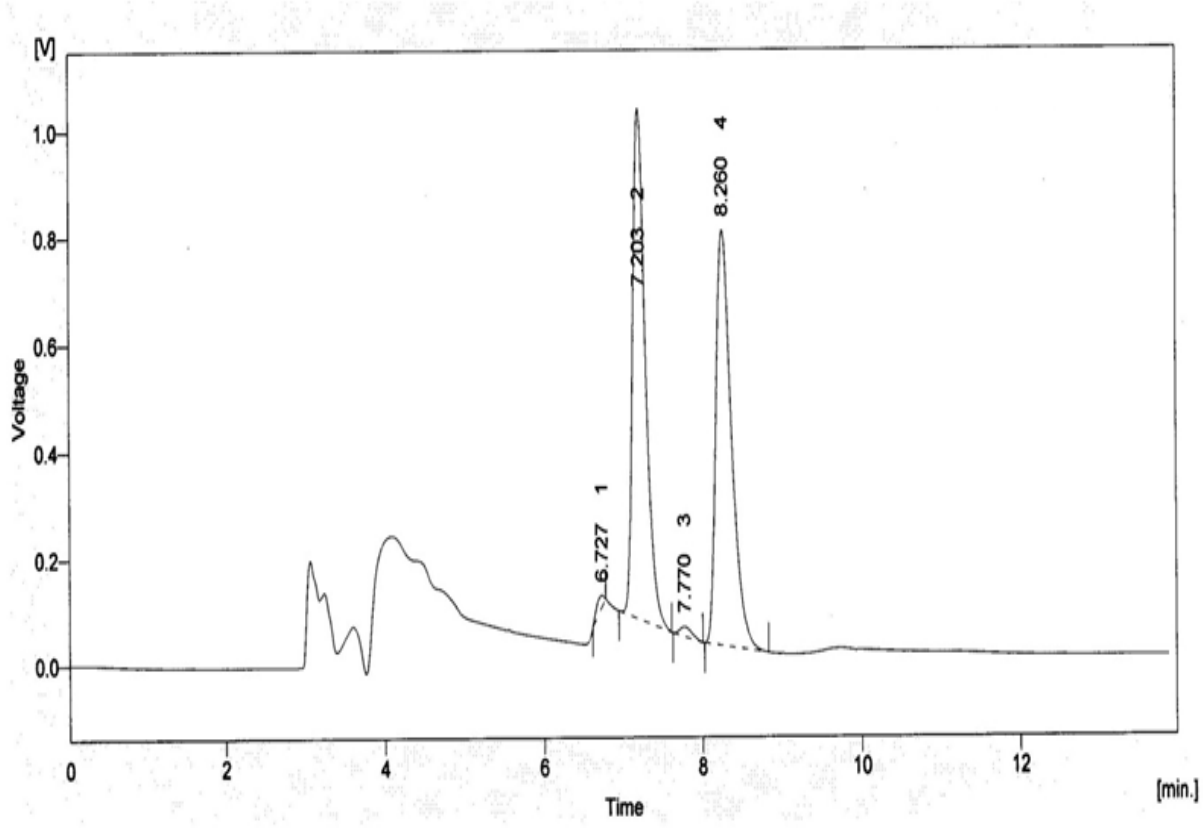








HPLC Chromotogram of 3-hydroxy-3-(1-nitroethyl)indolin-2-one 5:



	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	6.727	57.285	7.477	0.8	1.3	0.10
2	7.203	3567.216	319.032	49.3	53.9	0.17
3	7.770	65.968	6.123	0.9	1.0	0.17
4	8.260	3539.768	259.585	49.0	43.8	0.21
	Total	7230.237	592.218	100.0	100.0	