

Synthesis of Isoindolinones via a Ruthenium-Catalyzed Cyclization of *N*-Substituted Benzamides with Allylic Alcohols

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Electronic Supplementary Information (ESI)

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Experimental section

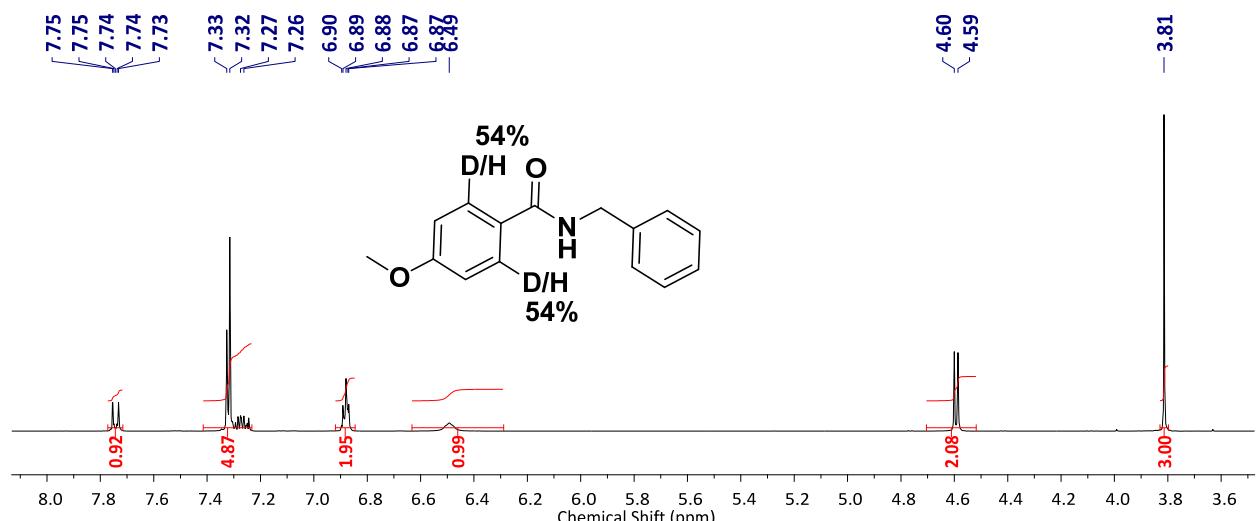
General Procedure for the Cyclization Reaction.

A 15 mL pressure tube with septum containing [$\{\text{RuCl}_2(p\text{-cymene})\}_2$] (5.0 mol %), aromatic or heteroaromatic amide **1** (100 mg), $\text{Cu}(\text{OAc})_2\text{H}_2\text{O}$ (2.20 equiv) and AgSbF_6 (20 mol %) was evacuated and purged with nitrogen gas three times (AgSbF_6 was taken inside the glove box). To the tube were then added allylic alcohol **2** (2.20 equiv) and $\text{ClCH}_2\text{CH}_2\text{Cl}$ (3.0 mL) via syringes after that the reaction mixture was evacuated and purged with nitrogen gas three times. After that, the septum was taken out immediately and a screw cap was used to cover the tube under the nitrogen atmosphere and again the reaction mixture stirred at room temperature for 5 minutes. Then, the reaction mixture was allowed to stir at 110 °C for 16 h. After cooling to the ambient temperature, the reaction mixture was diluted with CH_2Cl_2 , filtered through Celite and the filtrate was concentrated. The crude residue was purified through a silica gel column using hexanes and ethyl acetate as eluent to give pure product **3**.

Mechanistic Studies

Procedure for the Deuteration reaction.

A 15 mL pressure tube with septum containing $\{\text{RuCl}_2(p\text{-cymene})\}_2$ (5.0 mol %), *N*-benzyl-4-methoxybenzamide (**1a**) (100 mg), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (2.20 equiv) and AgSbF_6 (20 mol %) was evacuated and purged with nitrogen gas three times (AgSbF_6 was taken inside the glove box). To the tube were then added $\text{ClCH}_2\text{CH}_2\text{Cl}$ (2.70 mL) and deuterium oxide (0.3 mL) via syringes and again the reaction mixture was evacuated and purged with nitrogen gas three times. After that, the septum was taken out and immediately a screw cap was used to cover the tube under the nitrogen atmosphere. Then, the reaction mixture was allowed to stir at 110 °C for 16 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2 , filtered through Celite and the filtrate was concentrated. The crude residue was purified through a very short silica gel column using hexanes and ethyl acetate as eluent to give duterated *N*-benzyl-4-methoxybenzamide (**d-1a**).

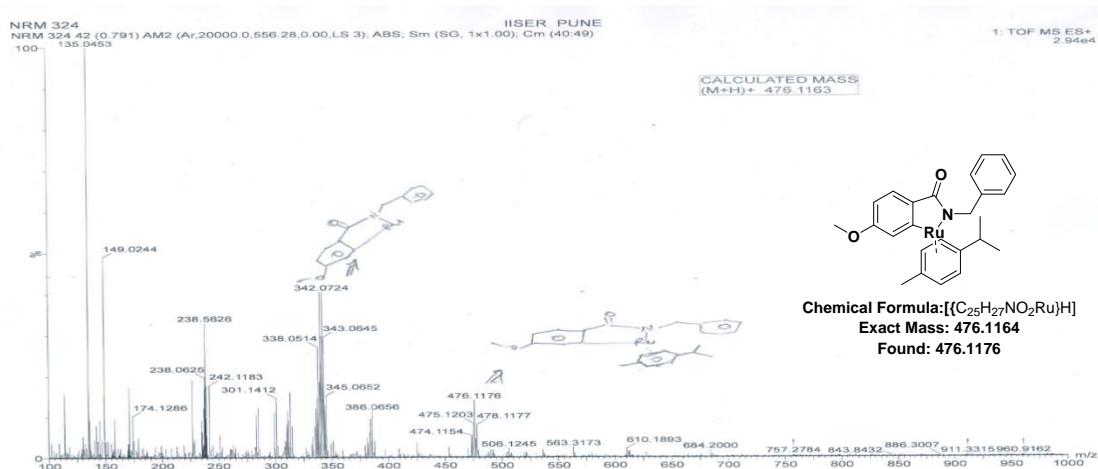


Procedure for the Preparation of a Five-Membered Ruthenacycle **5**.

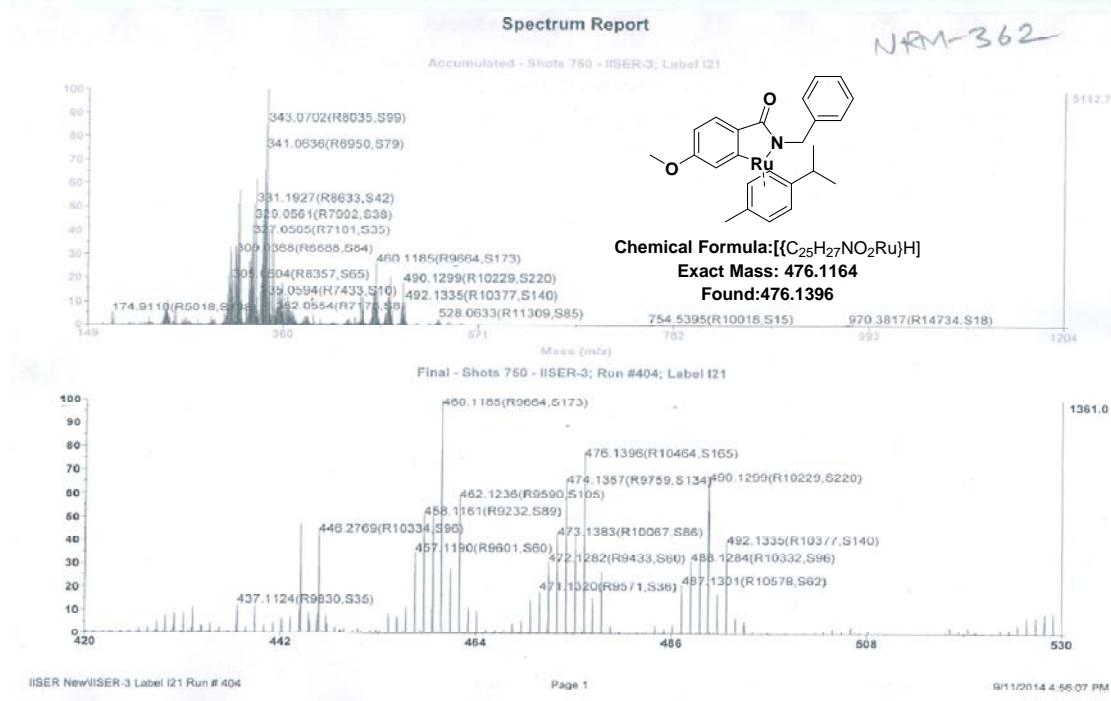
A 15 mL pressure tube with septum containing $\{\text{RuCl}_2(p\text{-cymene})\}_2$ (50 mg), benzamide **1a** (1.10 equiv), $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (1.20 equiv) and AgSbF_6 (4.0 equiv) was evacuated and purged with nitrogen gas three times (AgSbF_6 was taken inside the glove box). To the tube were then added $\text{ClCH}_2\text{CH}_2\text{Cl}$ (2.0 mL) via syringe and again the reaction mixture was evacuated and purged with nitrogen gas three times. After that, the septum was taken out and immediately a screw cap was used to cover the tube under the nitrogen atmosphere. Then, the reaction mixture was allowed to stir at 60 °C for 8 h. After cooling to ambient temperature, the

reaction mixture was diluted with methanol, filtered through Celite and the filtrate was concentrated and taken for further analysis without any further purification.

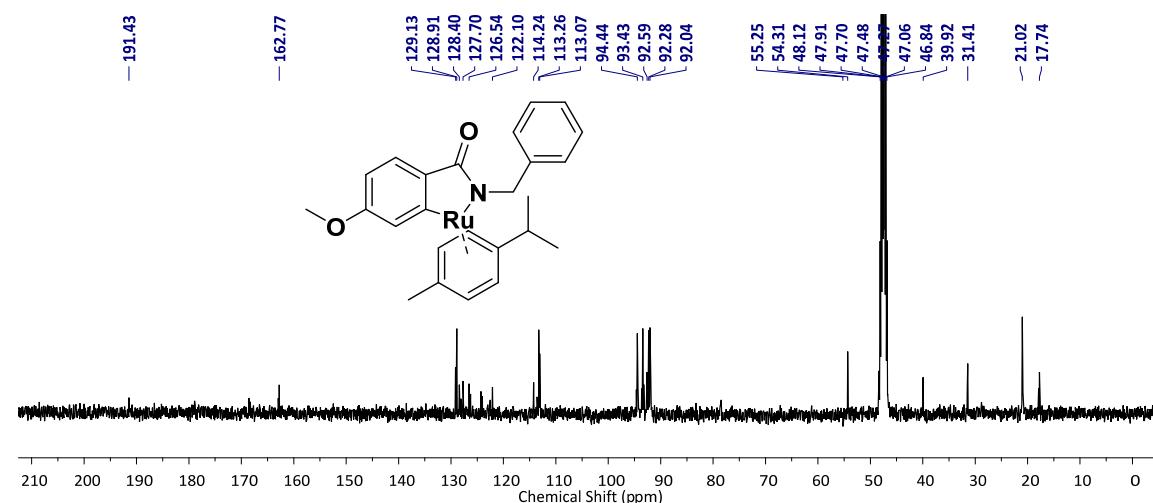
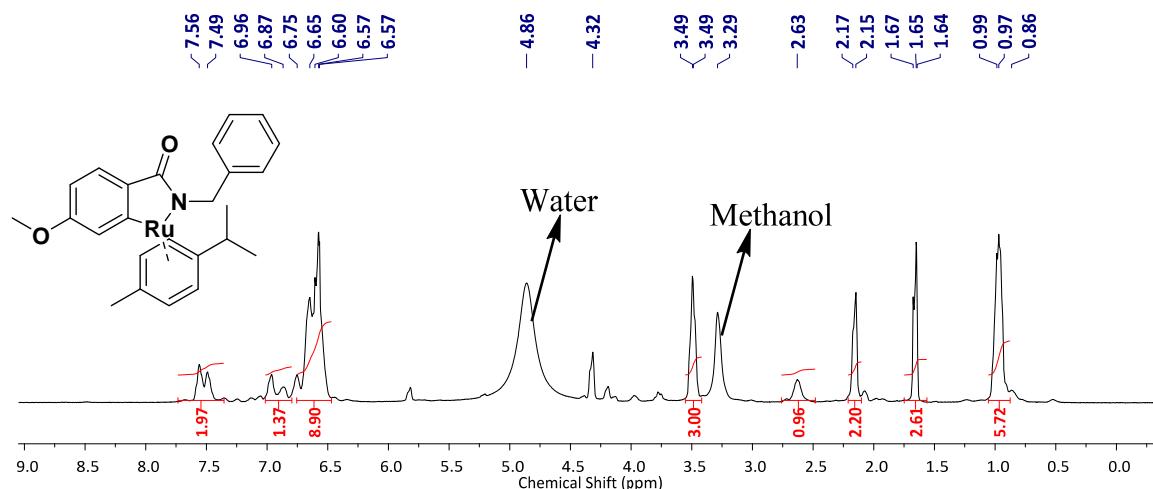
HRMS Data



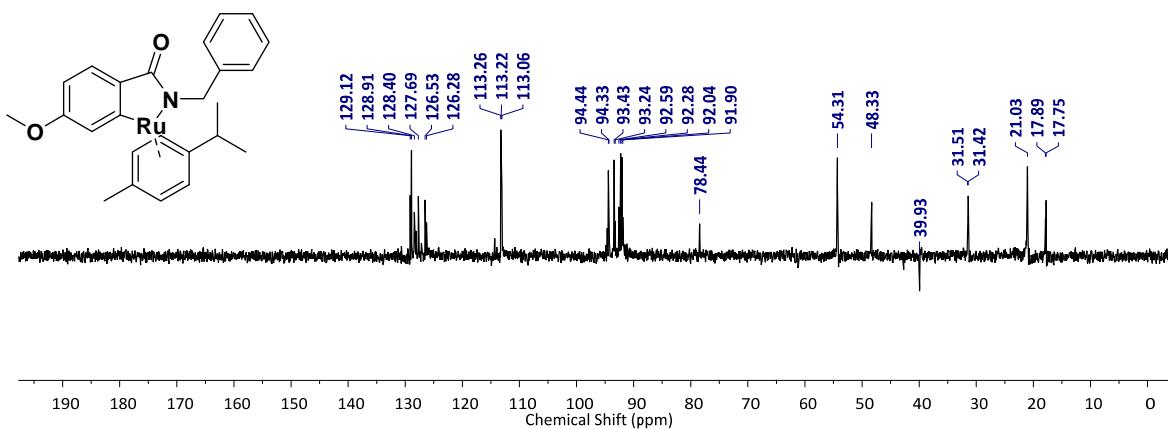
MALDI-TOF



NMR Data

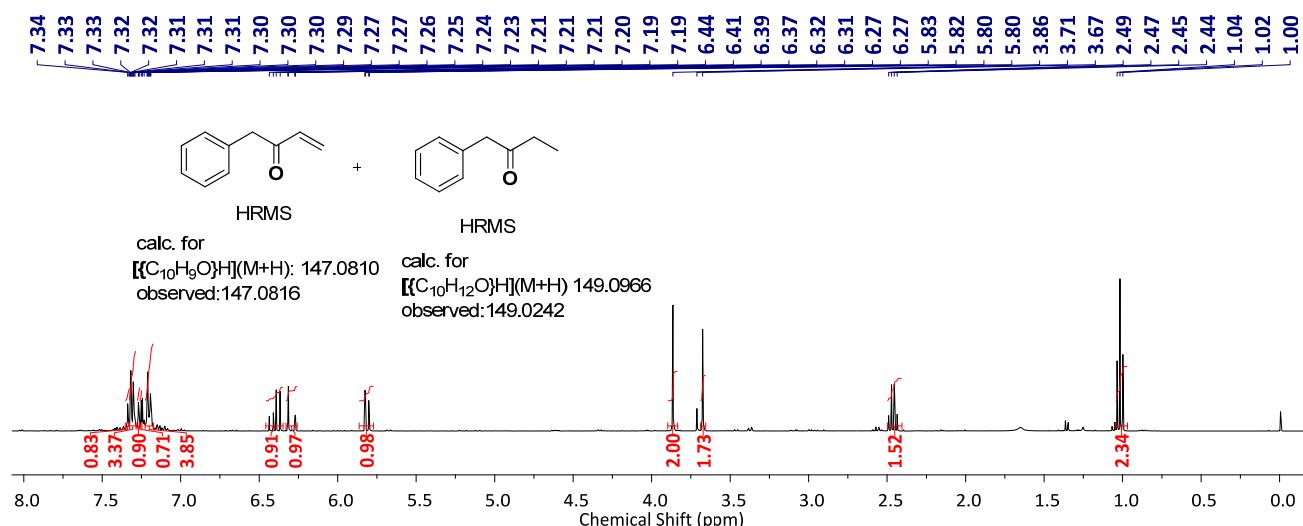


Note: In ^{13}C NMR, we think C-Ru peak comes at δ 191.4 due to the deshielding of C-Ru.



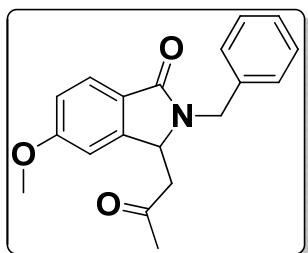
Procedure for the reaction of Phenylbut-3-en-2-ol (2e) with Ru(II) catalyst.

A 15-mL pressure tube with septum containing $\left[\{\text{RuCl}_2(p\text{-cymene})\}_2\right]$ (5.0 mol %), $\text{Cu}(\text{OAc})_2\text{H}_2\text{O}$ (1.20 equiv) and AgSbF_6 (20 mol %) was evacuated and purged with nitrogen gas three times (AgSbF_6 was taken inside the glove box). To the tube were then added 1-phenylbut-3-en-2-ol (100 mg), $\text{ClCH}_2\text{CH}_2\text{Cl}$ (3.0 mL) via syringes and the reaction mixture was evacuated and purged with nitrogen gas three times. After that, the septum was taken out and immediately a screw cap was used to cover the tube under the nitrogen atmosphere and further the reaction mixture stirred at room temperature for 5 minutes. Then, the reaction mixture was allowed to stir at 110 °C for 3 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2 , filtered through Celite and the filtrate was concentrated. The crude residue was purified through a very short silica gel column using hexanes and ethyl acetate as eluent.



Spectral Data of Compounds

2-Benzyl-5-methoxy-3-(2-oxopropyl)isoindolin-1-one (**3aa**).



White semisolid; eluent (40% ethyl acetate in hexanes); **1a** was taken in 100 mg; yield is 72% (92 mg).

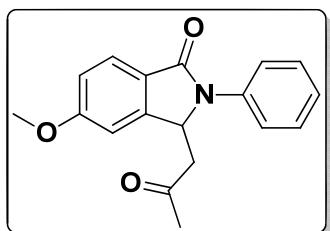
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 2960, 1721, 1515, 1277, 1071 and 743.

¹H NMR (400 MHz, CDCl₃): δ 7.78 (d, *J* = 8.4 Hz, 1 H), 7.31 – 7.25 (m, 2 H), 7.24 – 7.20 (m, 3 H), 6.97 (dd, *J* = 8.4, 2.2 Hz, 1 H), 6.81 (d, *J* = 2.2 Hz, 1 H), 4.93 (t, *J* = 6.0 Hz, 1 H), 4.85 (d, *J* = 15.4 Hz, 1 H), 4.55 (d, *J* = 15.4 Hz, 1 H), 3.81 (s, 3 H), 2.87 (dd, *J* = 17.8, 5.6 Hz, 1 H), 2.64 (dd, *J* = 17.8, 6.6 Hz, 1 H), 1.90 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.8, 168.5, 163.1, 147.9, 137.5, 128.8, 127.9, 127.5, 125.4, 124.3, 114.9, 107.6, 55.7, 55.3, 46.9, 44.7, 30.4.

HRMS (ESI): calc. for [(C₁₉H₁₉NO₃)H] (M+H) 310.1443, measured 310.1443.

5-Methoxy-3-(2-oxopropyl)-2-phenylisoindolin-1-one (**3ba**).



Colorless semisolid; eluent (40 % ethyl acetate in hexanes); **1b** was taken in 100 mg; yield is 59 % (77 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1692, 1614, 1497, 1461, 1373, 1263, 1084, and 729.

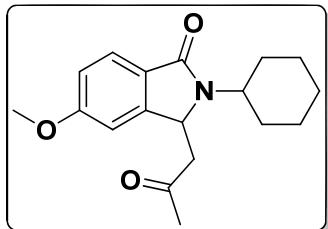
¹H NMR (400 MHz, CDCl₃): δ 7.81 (d, *J* = 8.4 Hz, 1 H), 7.56 – 7.47 (m, 2 H), 7.45 – 7.34 (m, 2 H), 7.23 – 7.16 (m, 1 H), 7.01 (dd, *J* = 8.4, 2.2 Hz, 1 H), 6.94 (d, *J* = 2.2 Hz, 1 H), 5.64

(dd, $J = 9.4, 3.0$ Hz, 1 H), 3.85 (s, 3 H), 3.04 (dd, $J = 18, 3$ Hz, 1 H), 2.61 (dd, $J = 18, 9.4$ Hz, 1 H), 2.09 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 206.5, 166.8, 163.4, 147.4, 136.8, 129.4, 125.6, 125.5, 124.4, 123.2, 115.5, 107.6, 56.1, 55.7, 46.7, 30.8.

HRMS (ESI): calc. for $[(\text{C}_{18}\text{H}_{17}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 296.1287, measured 296.1291.

2-Cyclohexyl-5-methoxy-3-(2-oxopropyl)isoindolin-1-one (3ca).



Colorless semisolid; eluent (35% ethyl acetate in hexanes); **1c** was taken in 100 mg; yield is 46 % (59 mg).

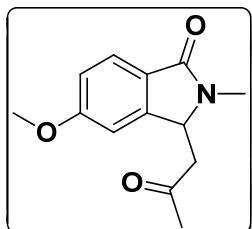
IR (ATR) $\tilde{\nu}$ (cm $^{-1}$): 1708, 1677, 1615, 1515, 1424, 1367, 1262, and 729.

^1H NMR (400 MHz, CDCl_3): δ 7.66 (d, $J = 8.4$ Hz, 1 H), 6.90 (dd, $J = 8.4, 2.2$ Hz, 1 H), 6.78 (d, $J = 2.2$ Hz, 1 H), 5.00 (dd, $J = 9, 3.4$ Hz, 1 H), 3.78 (s, 3 H), 3.71 – 3.65 (m, 1 H), 3.15 (dd, $J = 17.8, 3.4$ Hz, 1 H), 2.65 (dd, $J = 17.8, 9.2$ Hz, 1 H), 2.17 (s, 3 H), 1.94 (dd, $J = 12.4, 3.6$ Hz, 1 H), 1.87 – 1.74 (m, 4 H), 1.76 – 1.69 (m, 2 H), 1.36 – 1.28 (m, 2 H), 1.19 – 1.14 (m, 1 H).

^{13}C NMR (100 MHz, CDCl_3): δ 206.4, 168.3, 162.8, 148.3, 125.1, 124.8, 114.8, 107.5, 55.7, 55.3, 53.8, 48.2, 31.5, 31.1, 30.9, 26.2, 26.1, 25.5.

HRMS (ESI): calc. for $[(\text{C}_{18}\text{H}_{23}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 302.1756, measured 302.1758.

5-Methoxy-2-methyl-3-(2-oxopropyl)isoindolin-1-one (3da).



Yellow semisolid; eluent (55 % ethyl acetate in hexanes); **1d** was taken in 100 mg; yield is 67%, 94 mg).

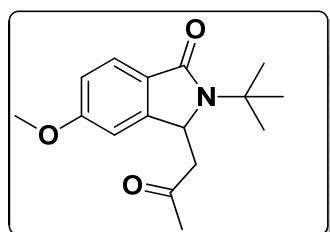
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 2962, 1722, 1463, 1374, 1275, 1124, 1071, and 734.

¹H NMR (400 MHz, CDCl₃): δ 7.69 (d, J = 8.4 Hz, 1 H), 6.93 (dd, J = 8.4, 2.2 Hz, 1 H), 6.85 (d, J = 2 Hz, 1 H), 4.87 (dd, J = 7.4, 5.2 Hz, 1 H), 3.81 (s, 3 H), 3.05 – 2.97 (m, 4 H), 2.71 (dd, J = 17.8, 7.4 Hz, 1 H), 2.21 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 206.0, 168.1, 162.8, 147.6, 124.9, 124.6, 114.8, 107.7, 57.0, 55.7, 46.6, 30.8, 27.7.

HRMS (ESI): calc. for [(C₁₃H₁₅NO₃)Na] (M+Na) 256.0950, measured 256.0946.

2-(*tert*-Butyl)-5-Methoxy-3-(2-oxopropyl)isoindolin-1-one (3ea).



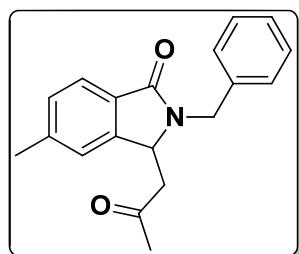
Yellow semisolid; eluent (55 % ethyl acetate in hexanes); **1d** was taken in 100 mg; combined yield is 45% (62 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 2962, 1722, 1463, 1374, 1275, 1124, 1071, and 734.

¹H NMR (400 MHz, CDCl₃): δ 7.62 (d, J = 8.4 Hz, 1 H), 6.89 (dd, J = 8.4, 2.2 Hz, 1 H), 6.75 (d, J = 2.2 Hz, 1 H), 5.17 (dd, J = 9.6, 2.0 Hz, 1 H), 3.79 (s, 3 H), 3.29 (dd, J = 18.2, 2 Hz, 1 H), 2.59 (dd, J = 18.2, 9.6 Hz, 1 H), 2.14 (s, 3 H), 1.53 (s, 9 H).

HRMS (ESI): calc. for [(C₁₆H₂₁NO₃)H] (M+Na) 276.1600 measured 276.1601.

2-Benzyl-5-methyl-3-(2-oxopropyl) isoindolin-1-one (3ja).



White semisolid; eluent (30% ethyl acetate in hexanes); **1j** was taken in 100 mg; yield is 69% (90 mg).

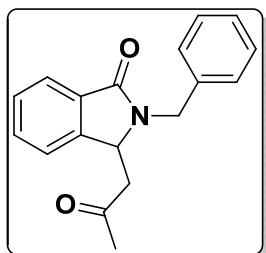
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1681, 1619, 1409, 1360, 1292, 1266, 1156, and 733.

¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 7.8 Hz, 1 H), 7.32 – 7.25 (m, 3 H), 7.22 (m, 3 H), 7.12 (s, 1 H), 4.95 (t, *J* = 6 Hz, 1 H), 4.86 (d, *J* = 15.4 Hz, 1 H), 4.57 (d, *J* = 15.4 Hz, 1 H), 2.85 (dd, *J* = 17.8, 5.8 Hz, 1 H), 2.65 (dd, *J* = 17.8, 6.4 Hz, 1 H), 2.40 (s, 3 H), 1.89 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.7, 168.7, 146.0, 142.6, 137.4, 129.5, 129.2, 128.8, 127.9, 127.5, 123.8, 122.9, 55.4, 46.9, 44.8, 30.4, 22.0.

HRMS (ESI): calc. for [(C₁₉H₁₉NO₂)H] (M+H) 294.1494, measured 294.1498.

2-Benzyl-3-(2-oxopropyl) isoindolin-1-one (3ka).



Colorless semisolid; eluent (35% ethyl acetate in hexanes); **1k** was taken in 100 mg; yield is 60% (71 mg).

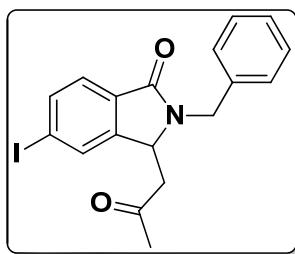
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1684, 1517, 1464, 1408, 1360, 1266, 1154 and 730.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (dd, *J* = 6.6, 1 Hz, 1 H), 7.51 – 7.46 (m, 2 H), 7.35 – 7.27 (m, 4 H), 7.24 – 7.22 (m, 2 H), 5.01 (t, *J* = 6.2 Hz, 1 H), 4.88 (d, *J* = 15.4 Hz, 1 H), 4.60 (d, *J* = 15.2 Hz, 1 H), 2.87 (dd, *J* = 17.8, 5.8 Hz, 1 H), 2.67 (dd, *J* = 17.8, 6.4 Hz, 1 H), 1.89 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.6, 168.6, 145.6, 137.9, 131.9, 131.7, 128.8, 128.7, 127.9, 127.6, 124.0, 122.5, 55.6, 46.8, 44.8, 30.4.

HRMS (ESI): calc. for [(C₁₈H₁₇NO₂)H] (M+H) 280.1338, measured 280.1333.

2-Benzyl-5-iodo-3-(2-oxopropyl) isoindolin-1-one (3la).



Brown solid; eluent (30% ethyl acetate in hexanes); **1l** was taken in 100 mg; yield is 61% (74 mg).

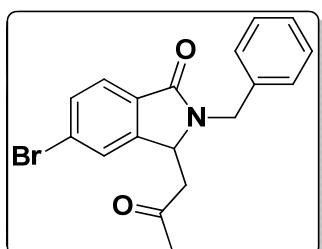
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1684, 1605, 1497, 1411, 1360, 1299, 1163, and 733.

¹H NMR (400 MHz, CDCl₃): δ 7.81 (dd, J = 8.4, 1.2 Hz, 1 H), 7.72 (s, 1 H), 7.59 (d, J = 8.0 Hz, 1 H), 7.31 – 7.22 (m, 3 H), 7.21 – 7.19 (m, 2 H), 4.95 (t, J = 6 Hz, 1 H), 4.84 (d, J = 15.4 Hz, 1 H), 4.56 (d, J = 15.4 Hz, 1 H), 2.87 (dd, J = 18, 5.6 Hz, 1 H), 2.64 (dd, J = 18, 6.6 Hz, 1 H), 1.91 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.2, 167.8, 147.4, 137.8, 136.9, 131.9, 131.3, 128.8, 127.9, 127.7, 125.4, 98.8, 55.1, 46.5, 44.8, 30.3.

HRMS (ESI): calc. for [(C₁₈H₁₆INO₂)H] (M+H) 406.0304, measured 406.0306.

2-Benzyl-5-bromo-3-(2-oxopropyl) isoindolin-1-one (3ma).



Colorless semisolid; eluent (30% ethyl acetate in hexanes); **1m** was taken in 100 mg; yield is 59% (73 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1683, 1608, 1409, 1359, 1163, and 734.

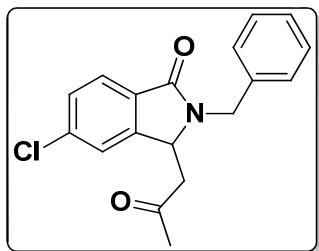
¹H NMR (400 MHz, CDCl₃): δ 7.72 (d, J = 8.0 Hz, 1 H), 7.59 (dd, J = 8, 1.4 Hz, 1 H), 7.51 (d, J = 1.4 Hz, 1 H), 7.30 – 7.24 (m, 3 H), 7.22 – 7.17 (m, 2 H), 4.96 (t, J = 6 Hz, 1 H), 4.85

(d, $J = 15.4$ Hz, 1 H), 4.56 (d, $J = 15.4$ Hz, 1 H), 2.88 (dd, $J = 18, 5.6$ Hz, 1 H), 2.63 (dd, $J = 18, 6.6$ Hz, 1 H), 1.92 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 205.2, 167.7, 147.6, 136.9, 132.0, 130.7, 128.8, 127.9, 127.7, 126.6, 126.1, 125.4, 55.2, 46.5, 44.8, 30.3.

HRMS (ESI): calc. for $[(\text{C}_{18}\text{H}_{16}\text{BrNO}_2)\text{H}] (\text{M}+\text{H})$ 358.0443, measured 358.0450.

2-Benzyl-5-chloro-3-(2-oxopropyl) isoindolin-1-one (3na).



Brown semisolid; eluent (30% ethyl acetate in hexanes); **1n** was taken in 100 mg; yield is 58% (74 mg).

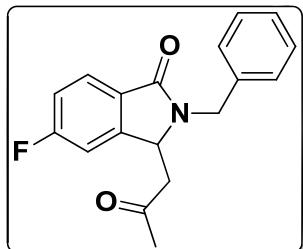
IR (ATR) $\tilde{\nu}$ (cm $^{-1}$): 1683, 1612, 1406, 1359, 1318, 1164, 1073 and 840.

^1H NMR (400 MHz, CDCl_3): δ 7.78 (d, $J = 8.0$ Hz, 1 H), 7.42 (dd, $J = 8.0, 1.6$ Hz, 1 H), 7.34 (m, 1 H), 7.30 – 7.19 (m, 5 H), 4.96 (t, $J = 6.2$ Hz, 1 H), 4.85 (d, $J = 15.4$ Hz, 1 H), 4.56 (d, $J = 15.4$ Hz, 1 H), 2.89 (dd, $J = 18, 5.6$ Hz, 1 H), 2.63 (dd, $J = 18, 6.8$ Hz, 1 H), 1.92 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 205.3, 167.6, 147.3, 138.3, 136.9, 130.2, 129.1, 128.8, 127.9, 127.7, 125.2, 123.2, 55.3, 46.4, 44.8, 30.3.

HRMS (ESI): calc. for $[(\text{C}_{18}\text{H}_{16}\text{ClNO}_2)\text{H}] (\text{M}+\text{H})$ 314.0948, measured 314.0948.

2-Benzyl-5-fluoro-3-(2-oxopropyl) isoindolin-1-one (3oa).



Brown solid; eluent (35% ethyl acetate in hexanes); **1o** was taken in 100 mg; yield is 47% (61 mg).

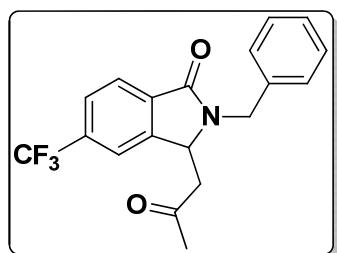
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1684, 1599, 1534, 1486, 1409, 1360, 1269, and 1220.

¹H NMR (400 MHz, CDCl₃): δ 7.84 (dd, J = 8.4, 5.0 Hz, 1 H), 7.29 – 7.24 (m, 3 H), 7.23 – 7.19 (m, 2 H), 7.14 (td, J = 8.6, 2.2 Hz, 1 H), 7.04 (dd, J = 8.4, 2.2 Hz, 1 H), 4.95 (t, J = 6.2 Hz, 1 H), 4.86 (d, J = 15.4 Hz, 1 H), 4.55 (d, J = 15.4 Hz, 1 H), 2.90 (dd, J = 18.0, 5.4 Hz, 1 H), 2.62 (dd, J = 18.0, 6.8 Hz, 1 H), 1.92 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.3, 167.6, 166.5, 164.0, 148.1 and 148.0 (F coupling), 137.0, 128.8, 127.7, 126.0, 125.9, 116.4 and 116.1 (F coupling), 110.4 and 110.1 (F coupling), 55.3, 46.5, 44.8, 30.3.

HRMS (ESI): calc. for [(C₁₈H₁₆FNO₂)H] (M+H) 298.1243, measured 298.1242.

2-Benzyl-3-(2-oxopropyl)-5-(trifluoromethyl)isoindolin-1-one (3pa).



White solid; eluent (35% ethyl acetate in hexanes); **1p** was taken in 100 mg; yield is 54 % (67 mg).

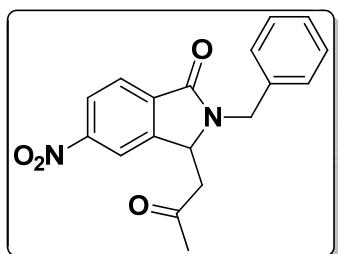
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1693, 1516, 1429, 1362, 1325, 1265, 1166, and 729.

¹H NMR (400 MHz, CDCl₃): δ 7.99 (d, J = 8.2 Hz, 1 H), 7.74 (d, J = 8.2 Hz, 1 H), 7.62 (s, 1 H), 7.33 – 7.19 (m, 5 H), 5.06 (t, J = 6 Hz, 1 H), 4.89 (d, J = 15.4 Hz, 1 H), 4.62 (d, J = 15.4 Hz, 1 H), 2.92 (dd, J = 18.2, 5.6 Hz, 1 H), 2.68 (dd, J = 18.2, 6.6 Hz, 1 H), 1.93 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.1, 167.2, 146.0, 136.7, 135.0, 134.0, 133.7, 128.9, 127.9, 127.8, 125.9 and 125.8 (F coupling), 124.5, 120.0 and 120.0 (F coupling), 55.6, 46.4, 45.0, 30.2.

HRMS (ESI): calc. for [(C₁₉H₁₆F₃NO₂)H] (M+H) 348.1211, measured 348.1220.

2-Benzyl-5-nitro-3-(2-oxopropyl) isoindolin-1-one (3qa).



White solid; eluent (35% ethyl acetate in hexanes); **1q** was taken in 100 mg; yield is 46 % (58 mg).

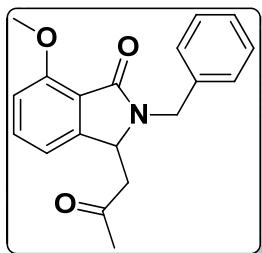
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1691, 1530, 1409, 1344, 1267, 1163, 1087, and 732.

¹H NMR (400 MHz, CDCl₃): δ 8.34 (dd, J = 8.2, 1.8 Hz, 1 H), 8.22 (d, J = 1.8 Hz, 1 H), 8.02 (d, J = 8.2 Hz, 1 H), 7.32 – 7.26 (m, 3 H), 7.23 – 7.21 (m, 2 H), 5.09 (t, J = 6.0 Hz, 1 H), 4.88 (d, J = 15.4 Hz, 1 H), 4.64 (d, J = 15.4 Hz, 1 H), 2.95 (dd, J = 18.4, 5.6 Hz, 1 H), 2.73 (dd, J = 18.4, 6.6 Hz, 1 H), 1.94 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 204.7, 166.4, 150.3, 146.6, 137.1, 136.4, 129.0, 127.9, 125.0, 124.3, 118.5, 55.7, 46.0, 45.2, 30.2.

HRMS (ESI): calc. for [(C₁₈H₁₆N₂O₄)H] (M+H) 325.1188, measured 325.1191.

2-Benzyl-7-methoxy-3-(2-oxopropyl) isoindolin-1-one (3ra).



White solid; eluent (35% ethyl acetate in hexanes) **1r** was taken in 100 mg; yield is 80 % (102 mg).

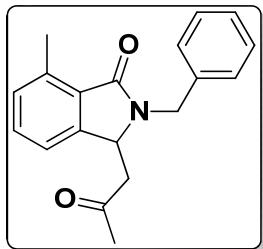
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1683, 1607, 1486, 1406, 1362, 1265, 1084, and 729.

¹H NMR (400 MHz, CDCl₃): δ 7.42 (dd, J = 8.2, 7.4 Hz, 1 H), 7.23 – 7.18 (m, 5 H), 6.87 (dd, J = 6.0, 2.4 Hz, 2 H), 4.92 (t, J = 6.0 Hz, 1 H), 4.75 (d, J = 15.4 Hz, 1 H), 4.55 (d, J = 15.4 Hz, 1 H), 3.94 (s, 3 H), 2.79 (dd, J = 17.8, 6 Hz, 1 H), 2.64 (dd, J = 17.8, 5.8 Hz, 1 H), 1.84 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 205.7, 167.5, 157.4, 148.3, 137.5, 133.6, 128.7, 128.0, 127.4, 118.8, 114.4, 110.5, 55.9, 55.1, 47.0, 44.6, 30.4.

HRMS (ESI): calc. for $[(\text{C}_{19}\text{H}_{19}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 310.1443, measured 310.1449.

2-Benzyl-7-methyl-3-(2-oxopropyl) isoindolin-1-one (3sa).



White solid; eluent (30% ethyl acetate in hexanes) **1s** was taken in 100 mg; yield is 65% (85 mg).

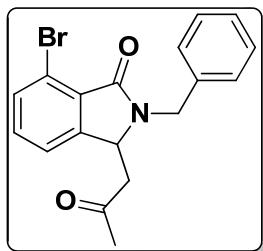
IR (ATR) $\tilde{\nu}$ (cm $^{-1}$): 1687, 1624, 1419, 1364, 1293, 1273, 1181 and 746.

^1H NMR (400 MHz, CDCl_3): δ 7.35 (t, $J = 7.6$ Hz, 1 H), 7.31 – 7.25 (m, 2 H), 7.24 (s, 2 H), 7.23 (d, $J = 2.4$ Hz, 1 H), 7.21 – 7.17 (m, 1 H), 7.12 (d, $J = 7.2$ Hz, 1 H), 4.95 (t, $J = 6.0$ Hz, 1 H), 4.82 (d, $J = 15.4$ Hz, 1 H), 4.59 (d, $J = 15.4$ Hz, 1 H), 2.82 (dd, $J = 17.6, 6.0$ Hz, 1 H), 2.75 (s, 3 H), 2.65 (dd, $J = 17.6, 6.0$ Hz, 1 H), 1.86 (s, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 205.7, 169.3, 146.1, 138.0, 137.5, 131.4, 130.5, 128.7, 128.0, 127.5, 119.7, 55.0, 47.1, 44.6, 30.4, 17.4.

HRMS (ESI): calc. for $[(\text{C}_{19}\text{H}_{19}\text{NO}_2)\text{H}] (\text{M}+\text{H})$ 294.1494, measured 294.1490.

2-Benzyl-7-bromo-3-(2-oxopropyl) isoindolin-1-one (3ta).



Brown oil; eluent (30% ethyl acetate in hexanes) **1t** was taken in 100 mg, yield is 62% (77 mg).

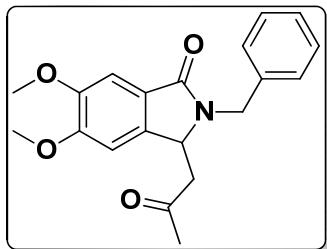
IR (ATR) $\tilde{\nu}$ (cm $^{-1}$): 1693, 1515, 1264, 1409, 1386, 1206, 1084, and 729.

¹H NMR (400 MHz, CDCl₃): δ 7.88 (dd, *J* = 6.6, 1.4 Hz, 1 H), 7.48 (m, 2 H), 7.33 (d, *J* = 7.2 Hz, 1 H), 7.30 – 7.27 (m, 2 H), 7.24 – 7.21 (m, 2 H), 5.01 (t, *J* = 6.2 Hz, 1 H), 4.88 (d, *J* = 15.4 Hz, 1 H), 4.60 (d, *J* = 15.4 Hz, 1 H), 2.86 (dd, *J* = 17.8, 5.8 Hz, 1 H), 2.66 (dd, *J* = 17.8, 6.4 Hz, 1 H), 1.89 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.6, 168.6, 145.6, 137.3, 131.9, 131.7, 128.8, 128.5, 127.9, 127.6, 124.0, 122.5, 55.6, 46.8, 44.8, 30.4.

HRMS (ESI): calc. for [(C₁₈H₁₆BrNO₂)H] (M+H) 358.0443, measured 358.0448.

2-Benzyl-5, 6-dimethoxy-3-(2-oxopropyl)isoindolin-1-one (3ua).



White solid; eluent (35% ethyl acetate in hexanes). **1u** was taken in 100 mg, yield is 53 % (66 mg).

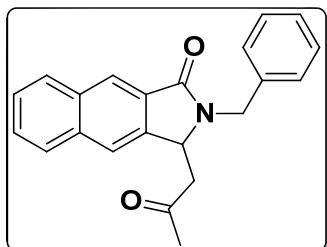
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1682, 1510, 1464, 1426, 1264, 1220, 1084, and 730.

¹H NMR (400 MHz, CDCl₃): δ 7.34 (s, 1 H), 7.31 – 7.25 (m, 2 H), 7.23 – 7.20 (m, 3 H), 6.82 (s, 1 H), 4.96 – 4.83 (m, 2 H), 4.51 (d, *J* = 15.4 Hz, 1 H), 3.93 (s, 3 H), 3.87 (s, 3 H), 2.90 (dd, *J* = 17.8, 5.4 Hz, 1 H), 2.60 (dd, *J* = 17.8, 7.0 Hz, 1 H), 1.94 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 206.0, 168.8, 152.8, 149.9, 139.2, 137.4, 128.7, 127.8, 127.5, 123.9, 105.4, 104.9, 56.3, 55.2, 46.9, 44.7, 30.4.

HRMS (ESI): calc. for [(C₂₀H₂₁NO₄)H] (M+H) 340.1549, measured 340.1551.

2-Benzyl-3-(2-oxopropyl)-2, 3-dihydro-1H-benzo[f]isoindol-1-one (3va).



White waxy solid; eluent (35% ethyl acetate in hexanes) **1v** was taken in 100 mg, yield is 58 % (73 mg).

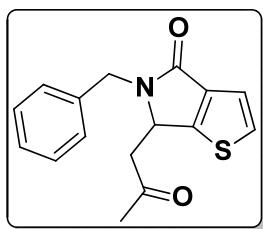
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1685, 1515, 1417, 1364, 1264, 1125, 1084, and 730.

¹H NMR (400 MHz, CDCl₃): δ 8.40 (s, 1 H), 8.00 (dd, J = 6.8, 2 Hz, 1 H), 7.88 – 7.83 (m, 1 H), 7.76 (s, 1 H), 7.54 (m, 2 H), 7.34 – 7.19 (m, 5 H), 5.14 (t, J = 5.8 Hz, 1 H), 4.97 (d, J = 15.4 Hz, 1 H), 4.63 (d, J = 15.4 Hz, 1 H), 2.98 (dd, J = 17.8, 5.4 Hz, 1 H), 2.74 (dd, J = 17.8, 6.8 Hz, 1 H), 1.94 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.8, 168.4, 140.6, 137.1, 135.3, 133.1, 129.5, 128.8, 128.2, 128.0, 127.8, 127.6, 126.6, 124.3, 121.6, 55.5, 47.4, 45.0, 30.5.

HRMS (ESI): calc. for [(C₂₂H₁₉NO₂)H] (M+H) 330.1494, measured 330.1496.

5-Benzyl-6-(2-oxopropyl)-5,6-dihydro-4H-thieno[2,3-c]pyrrol-4-one (3wa).



White semisolid; eluent (35% ethyl acetate in hexanes) 1w was taken in 100 mg; yield is 60 % (79 mg).

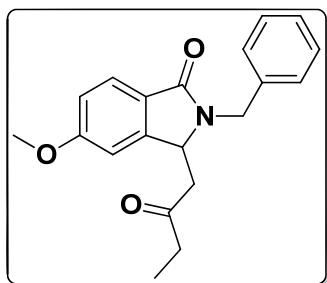
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1681, 1532, 1516, 1397, 1362, 1265, 908 and 730.

¹H NMR (400 MHz, CDCl₃): δ 7.58 (d, J = 4.8 Hz, 1 H), 7.31 – 7.27 (m, 2 H), 7.25 – 7.22 (m, 3 H), 6.91 (d, J = 4.8 Hz, 1 H), 4.94 (d, J = 15.6 Hz, 1 H), 4.80 (dd, J = 8.4, 5.2 Hz, 1 H), 4.43 (d, J = 15.6 Hz, 1 H), 2.95 (dd, J = 17.8, 5.2 Hz, 1 H), 2.54 (dd, J = 17.8, 8.4 Hz, 1 H), 1.98 (s, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.5, 164.5, 155.6, 137.3, 135.1, 134.9, 128.8, 127.8, 127.6, 121.7, 54.8, 45.7, 45.1, 30.4.

HRMS (ESI): calc. for [(C₁₆H₁₅NO₂S)H] (M+H) 286.0902, measured 286.0898.

2-Benzyl-5-methoxy-3-(2-oxobutyl) isoindolin-1-one (3ab).



White solid; eluent (35% ethyl acetate in hexanes) **1a** was taken in 100 mg; yield is 67% (90 mg).

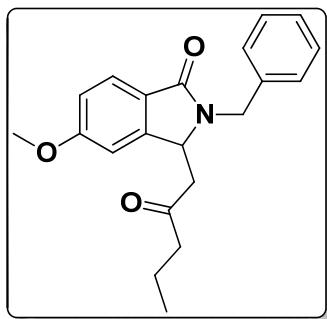
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1676, 1613, 1489, 1405, 1281, 1107, 1027, and 699.

¹H NMR (400 MHz, CDCl₃): δ 7.78 (d, J = 8.4 Hz, 1 H), 7.29 – 7.24 (m, 2 H), 7.22 – 7.19 (m, 3 H), 6.96 (dd, J = 8.4, 2.2 Hz, 1 H), 6.79 (d, J = 2.2 Hz, 1 H), 4.96 (t, J = 6.2 Hz, 1 H), 4.81 (d, J = 15.4 Hz, 1 H), 4.56 (d, J = 15.4 Hz, 1 H), 3.81 (s, 3 H), 2.82 (dd, J = 17.6, 6.0 Hz, 1 H), 2.60 (dd, J = 17.6, 6.4 Hz, 1 H), 2.20 (dq, J = 18, 7.2 Hz, 1 H), 1.97 (dq, J = 18.0, 7.2 Hz, 1 H), 0.94 (t, J = 7.2 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 208.4, 168.5, 163.0, 148.0, 137.5, 128.7, 127.9, 127.5, 125.3, 124.3, 114.9, 107.5, 55.7, 55.5, 45.8, 44.8, 36.4, 7.5.

HRMS (ESI): calc. for [(C₂₀H₂₁NO₃)H] (M+H) 324.1600, measured 324.1598.

2-Benzyl-5-methoxy-3-(2-oxopentyl) isoindolin-1-one (3ac).



White solid; eluent (30% ethyl acetate in hexanes) **1a** was taken in 100 mg; yield is 65% (91 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1676, 1612, 1488, 1403, 1252, 1184, 1027, and 735.

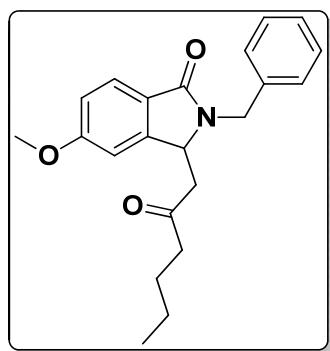
¹H NMR (400 MHz, CDCl₃): δ 7.77 (d, J = 8.4 Hz, 1 H), 7.29 – 7.24 (m, 2 H), 7.23 – 7.17 (m, 3 H), 6.96 (dd, J = 8.4, 2.2 Hz, 1 H), 6.79 (d, J = 2.2 Hz, 1 H), 4.95 (t, J = 6.2 Hz, 1 H),

4.84 (d, $J = 15.4$ Hz, 1 H), 4.53 (d, $J = 15.4$ Hz, 1 H), 3.80 (s, 3 H), 2.83 (dd, $J = 17.6, 5.8$ Hz, 1 H), 2.59 (dd, $J = 17.6, 6.6$ Hz, 1 H), 2.16 (dd, $J = 17.0, 6.6$ Hz, 1 H), 1.96 (dd, $J = 17.2, 6.6$ Hz, 1 H), 1.54 – 1.43 (m, 2 H), 0.82 (t, $J = 7.4$ Hz, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 206.9, 167.3, 161.8, 146.8, 136.3, 127.5, 126.7, 126.3, 124.1, 123.1, 113.8, 106.3, 54.5, 54.2, 44.9, 43.9, 43.6, 15.7, 12.4.

HRMS (ESI): calc. for $[(\text{C}_{21}\text{H}_{23}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 338.1756, measured 338.1754.

2-Benzyl-5-methoxy-3-(2-oxohexyl) isoindolin-1-one (3ad).



White solid; eluent (30 % ethyl acetate in hexanes) 1a was taken in 100 mg, yield is 62 % (90 mg).

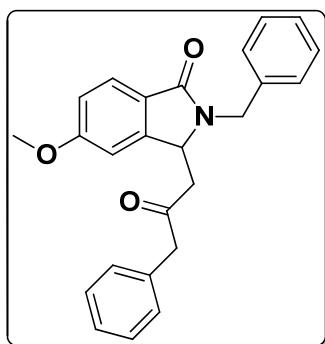
IR (ATR) $\tilde{\nu}$ (cm $^{-1}$): 1683, 1615, 1494, 1408, 1262, 1180, 1084, and 730.

^1H NMR (400 MHz, CDCl_3): δ 7.78 (d, $J = 8.4$ Hz, 1 H), 7.29 – 7.25 (m, 2 H), 7.24 – 7.20 (m, 3 H), 6.97 (dd, $J = 8.4, 2.2$ Hz, 1 H), 6.80 (d, $J = 2.2$ Hz, 1 H), 4.95 (t, $J = 6.2$ Hz, 1 H), 4.85 (d, $J = 15.4$ Hz, 1 H), 4.54 (d, $J = 15.4$ Hz, 1 H), 3.81 (s, 3 H), 2.83 (dd, $J = 17.6, 5.8$ Hz, 1 H), 2.60 (dd, $J = 17.6, 6.6$ Hz, 1 H), 2.18 (dd, $J = 17.0, 6.6$ Hz, 1 H), 1.99 (dd, $J = 17.0, 6.6$ Hz, 1 H), 1.48 – 1.39 (m, 2 H), 1.25 (d, $J = 7.4$ Hz, 1 H), 1.19 (d, $J = 7.6$ Hz, 1 H), 0.84 (t, $J = 7.2$ Hz, 3 H).

^{13}C NMR (100 MHz, CDCl_3): δ 208.2, 168.5, 163.0, 148.0, 137.5, 128.7, 127.9, 127.5, 125.3, 124.3, 114.9, 107.5, 55.7, 55.4, 46.1, 44.8, 43.0, 25.5, 22.2, 13.8.

HRMS (ESI): calc. for $[(\text{C}_{22}\text{H}_{25}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 352.1913, measured 352.1920.

2-Benzyl-5-methoxy-3-(2-oxo-3-phenylpropyl)isoindolin-1-one (3ae).



Colorless semisolid; eluent (35% ethyl acetate in hexanes), **1a** was taken in 100 mg; yield is 65 % (104 mg).

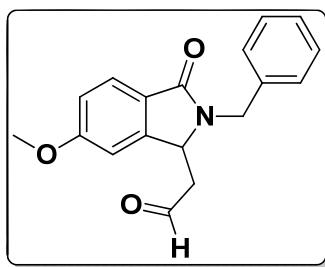
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1683, 1614, 1495, 1451, 1406, 1255, 1045, and 700.

¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, J = 8.4 Hz, 1 H), 7.31 – 7.23 (m, 6 H), 7.18 – 7.16 (m, 2 H), 7.06 – 7.04 (m, 2 H), 6.95 (dd, J = 8.4, 2.2 Hz, 1 H), 6.73 (d, J = 2 Hz, 1 H), 4.89 (t, J = 6 Hz, 1.0 H), 4.82 (d, J = 15.4 Hz, 1 H), 4.44 (d, J = 15.4 Hz, 1 H), 3.78 (s, 3 H), 3.39 (s, 2 H), 2.90 (dd, J = 18, 5.6 Hz, 1 H), 2.63 (dd, J = 18, 6.4 Hz, 1 H).

¹³C NMR (100 MHz, CDCl₃): δ 205.5, 168.5, 163.0, 147.8, 137.4, 133.3, 129.4, 128.9, 128.8, 127.9, 127.5, 127.4, 125.3, 124.3, 115.0, 107.5, 55.7, 55.3, 50.4, 45.3, 44.7.

HRMS (ESI): calc. for [(C₂₅H₂₃NO₃)H] (M+H) 386.1756, measured 386.1765.

2-(2-Benzyl-6-methoxy-3-oxoisoindolin-1-yl) acetaldehyde (3af).



Colorless semisolid; eluent (35% ethyl acetate in hexanes), **1a** was taken in 100 mg; yield is 60% (73 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1678, 1614, 1531, 1494, 1257, 1220, 1029 and 735.

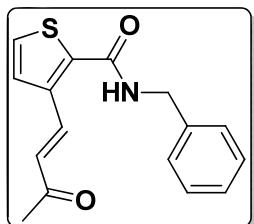
¹H NMR (400 MHz, CDCl₃): δ 9.54 (t, J = 1.2 Hz, 1 H), 7.80 (d, J = 8.4 Hz, 1 H), 7.32 – 7.28 (m, 4 H), 7.23 (s, 1 H), 7.00 (dd, J = 8.4, 2.2 Hz, 1 H), 6.85 (d, J = 2.2 Hz, 1 H), 5.11 (d,

$J = 15.4$ Hz, 1 H), 4.85 – 4.76 (m, 1 H), 4.36 (d, $J = 15.4$ Hz, 1 H), 3.83 (s, 3 H), 2.94 (dd, $J = 17.8, 4.6$ Hz, 1 H), 2.77 (dd, $J = 17.8, 6.4$, 1 H).

^{13}C NMR (100 MHz, CDCl_3): δ 199.1, 168.4, 163.2, 147.0, 136.9, 128.9, 128.0, 127.8, 125.5, 124.3, 115.2, 107.7, 55.7, 54.2, 45.9, 44.5.

HRMS (ESI): calc. for $[(\text{C}_{18}\text{H}_{17}\text{NO}_3)\text{H}] (\text{M}+\text{H})$ 296.1287, measured 296.1290.

(E)-N-Benzyl-3-(3-oxobut-1-en-1-yl)thiophene-2-carboxamide (8).



White semisolid; eluent (35% ethyl acetate in hexanes), **1w** was taken in 100 mg; yield is 47 % (62 mg).

IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1648, 1530, 1425, 1262 and 730.

^1H NMR (400 MHz, CDCl_3): δ 8.30 (d, $J = 16.6$ Hz, 1 H), 7.40 – 7.25 (m, 7 H), 6.49 (d, $J = 16.6$ Hz, 1 H), 6.35 (s, 1 H), 4.60 (d, $J = 5.6$ Hz, 2 H), 2.35 (s, 3 H).

^{13}C NMR (50 MHz, CDCl_3): δ 199.6, 161.9, 139.9, 137.6, 136.2, 135.4, 130.4, 128.9, 127.8, 127.8, 127.1, 126.9, 44.2, 26.5.

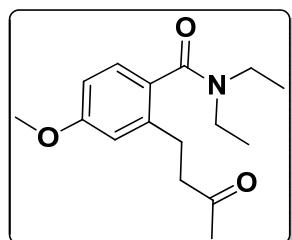
HRMS (ESI): calc. for $[(\text{C}_{16}\text{H}_{15}\text{NO}_2\text{S})\text{H}] (\text{M}+\text{H})$ 286.0902, measured 286.0887.

Procedure for the Reaction of *N,N*-diethyl benzamide **14 with **2a**.**

A 15-mL pressure tube with septum containing $[\{\text{RuCl}_2(p\text{-cymene})\}_2]$ (5.0 mol %), $\text{Cu}(\text{OAc})_2\text{H}_2\text{O}$ (2.20 eq) and AgSbF_6 (20 mol %) was evacuated and purged with nitrogen gas three times (AgSbF_6 was taken inside the glove box). To the tube were then added $\text{ClCH}_2\text{CH}_2\text{Cl}$ (3.0 mL), amide **14** or **1a** (100 mg, 1.0 equiv), acetic acid (2.0 equiv) and allyl alcohol **2** (2.2 equiv) via syringe after that the reaction mixture was evacuated and purged with nitrogen gas three times. After that, the septum was taken out and immediately a screw cap was used to cover the tube under the nitrogen atmosphere and the reaction mixture stirred in room temperature for 5 minutes. Then, the reaction mixture was allowed to stir at 110 °C for 16 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2 ,

filtered through Celite and the filtrate was concentrated. The crude residue was purified through a very short silica gel column using hexanes and ethyl acetate as eluent.

N,N-Diethyl-4-methoxy-2-(3-oxobutyl)benzamide (16).



Yellow liquid: eluent (35% ethyl acetate in hexanes), **14** was taken in 100 mg, combined yield is 69% (920mg).

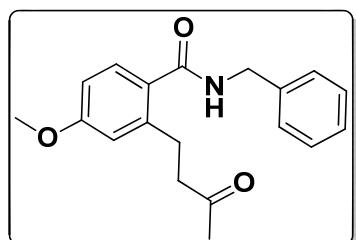
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1712, 1611, 1463, 1430, 1243, 1088, 905 and 819.

¹H NMR (400 MHz, CDCl₃): δ 7.04 (d, J = 9.0 Hz, 1 H), 6.74 – 6.68 (m, 2 H), 3.75 (s, 3 H), 3.56 (s, 2 H), 3.15 – 3.04 (m, 2 H), 2.73 (s, 4 H), 2.07 (s, 3 H), 1.18 (t, J = 7.8 Hz, 3 H), 0.99 (t, J = 7.0 Hz, 3 H).

¹³C NMR (100 MHz, CDCl₃): δ 207.9, 170.6, 159.8, 139.3, 129.4, 127.1, 115.1, 111.5, 55.3, 45.1, 43.0, 38.9, 29.9, 27.5, 14.1, 12.8.

HRMS (ESI): calc. for [(C₁₆H₂₃NO₃)H] (M+H) 278.1756, measured 278.1758.

N-Benzyl-4-methoxy-2-(3-oxobutyl)benzamide (18).



White solid: eluent (35% ethyl acetate in hexanes), **1a** was taken in 100 mg, yield is 69 % (89 mg).

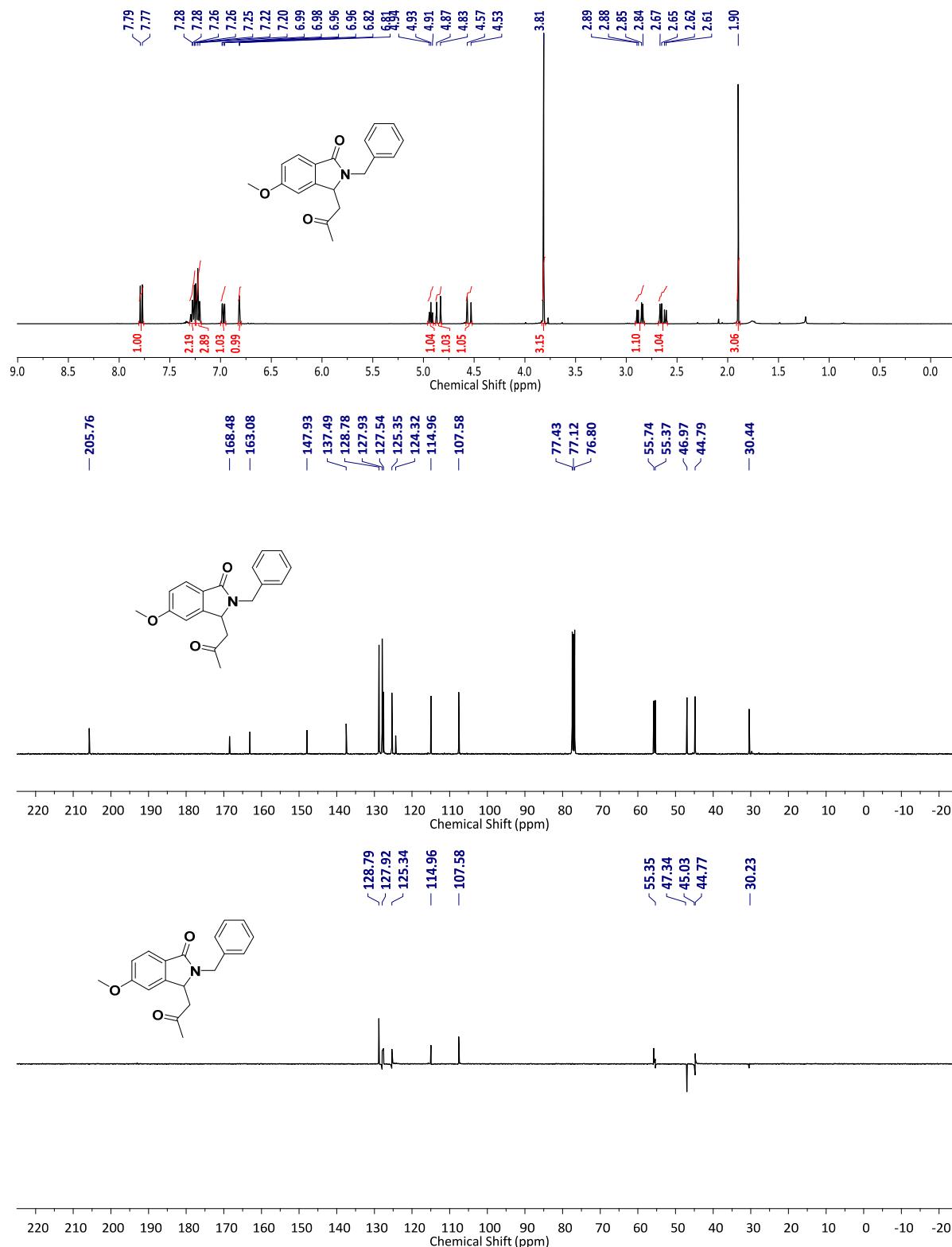
IR (ATR) $\tilde{\nu}$ (cm⁻¹): 1705, 1627, 1419, 1306, 1247, 1166, 1054 and 735.

¹H NMR (400 MHz, CDCl₃): δ 7.36 – 7.31 (m, 5 H), 7.30 – 7.25 (m, 1 H), 6.73 (d, *J* = 2.4 Hz, 1 H), 6.69 (dd, *J* = 8.4, 2.6 Hz, 1 H), 6.55 (s, 1 H), 4.57 (d, *J* = 5.8 Hz, 2 H), 3.77 (s, 3 H), 2.98 (t, *J* = 7.2 Hz, 2 H), 2.84 (t, *J* = 7.2 Hz, 2 H), 2.08 (s, 3 H).

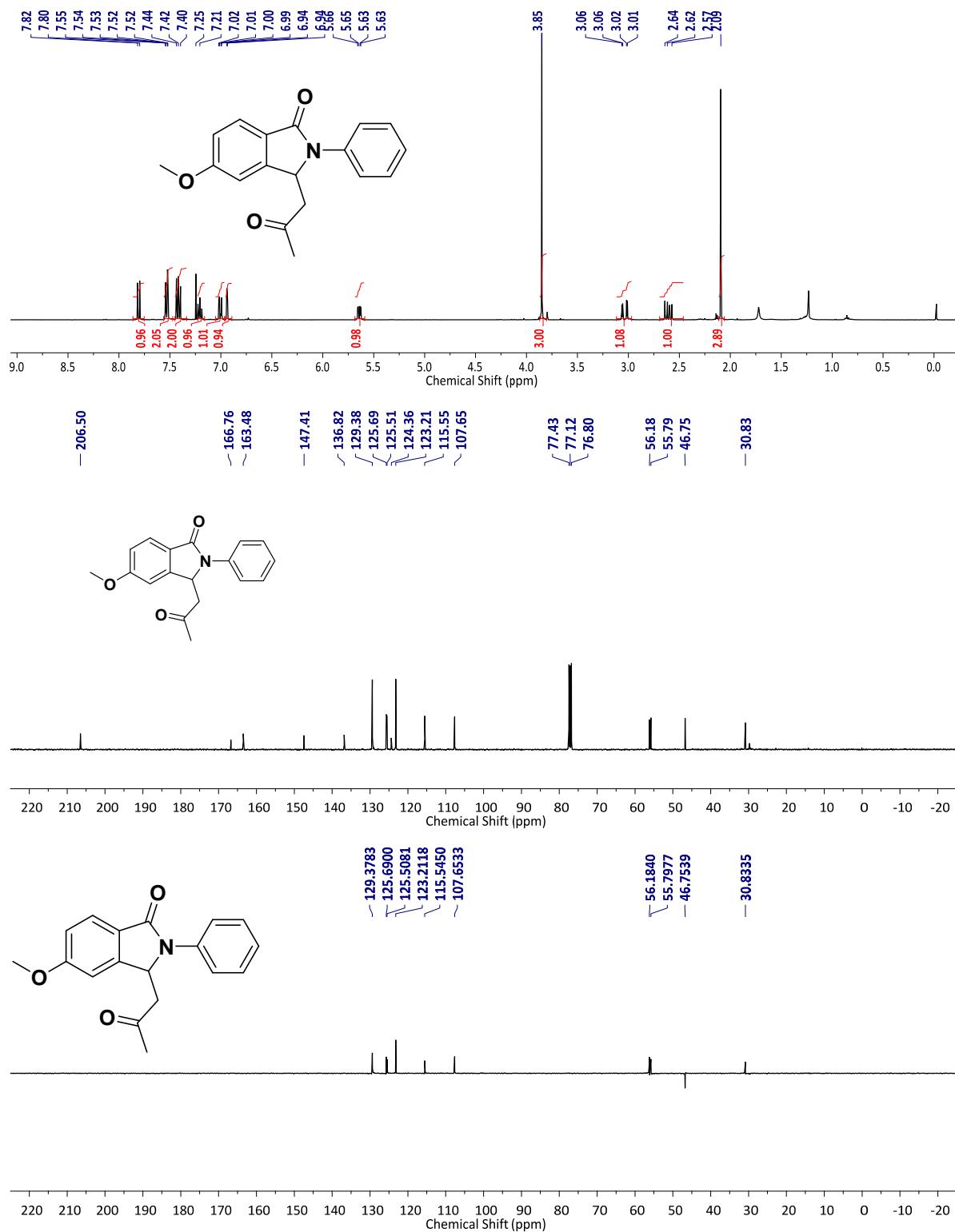
¹³C NMR (100 MHz, CDCl₃): δ 208.6, 169.5, 160.9, 141.9, 138.4, 129.0, 128.8, 128.7, 127.9, 127.6, 115.7, 111.4, 55.4, 45.4, 44.0, 30.0, 27.8.

HRMS (ESI): calc. for [(C₁₉H₂₁NO₃)H] (M+H) 312.1600, measured 312.1594.

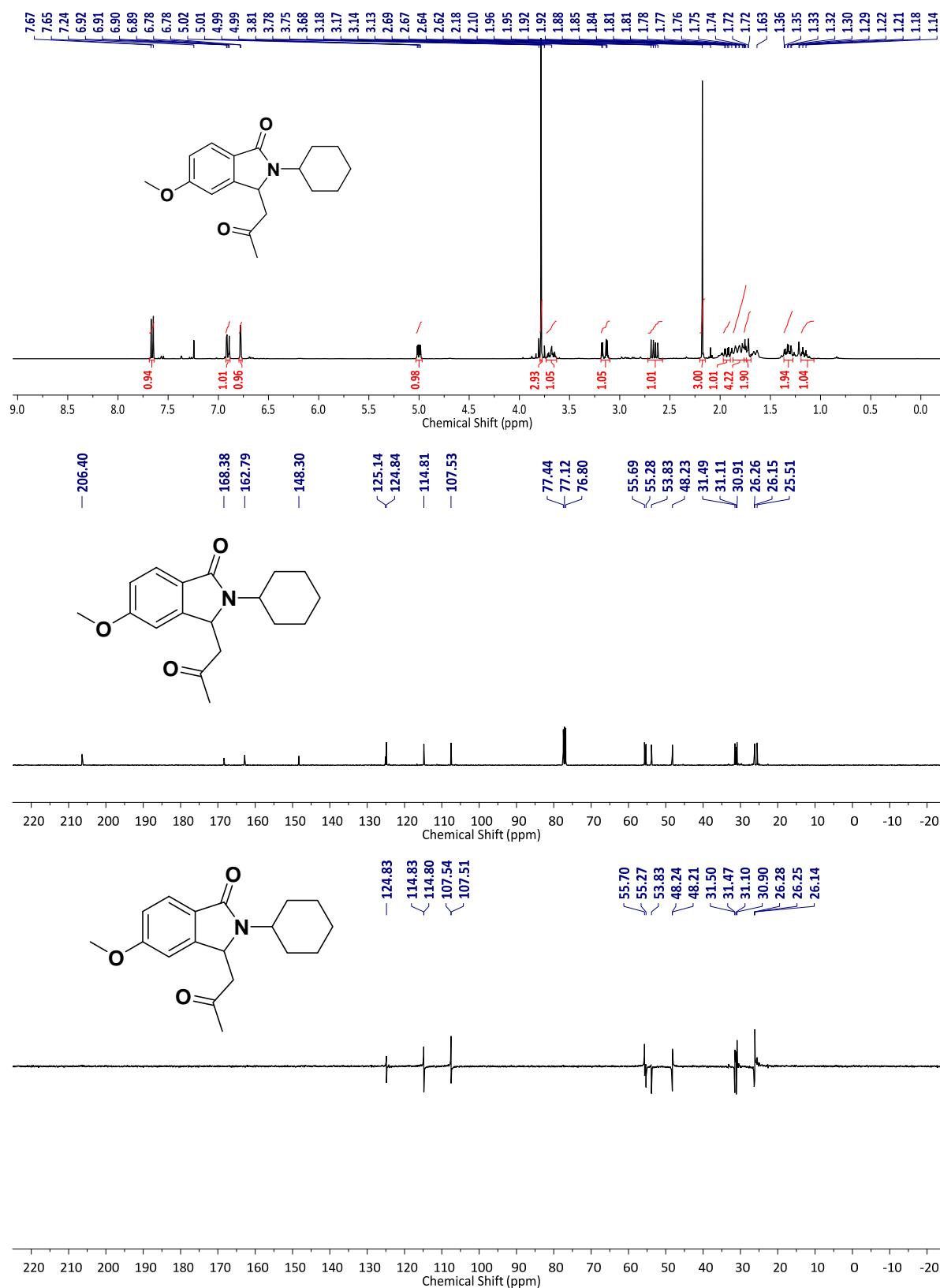
¹H and ¹³C NMR Spectra of compound 3aa.



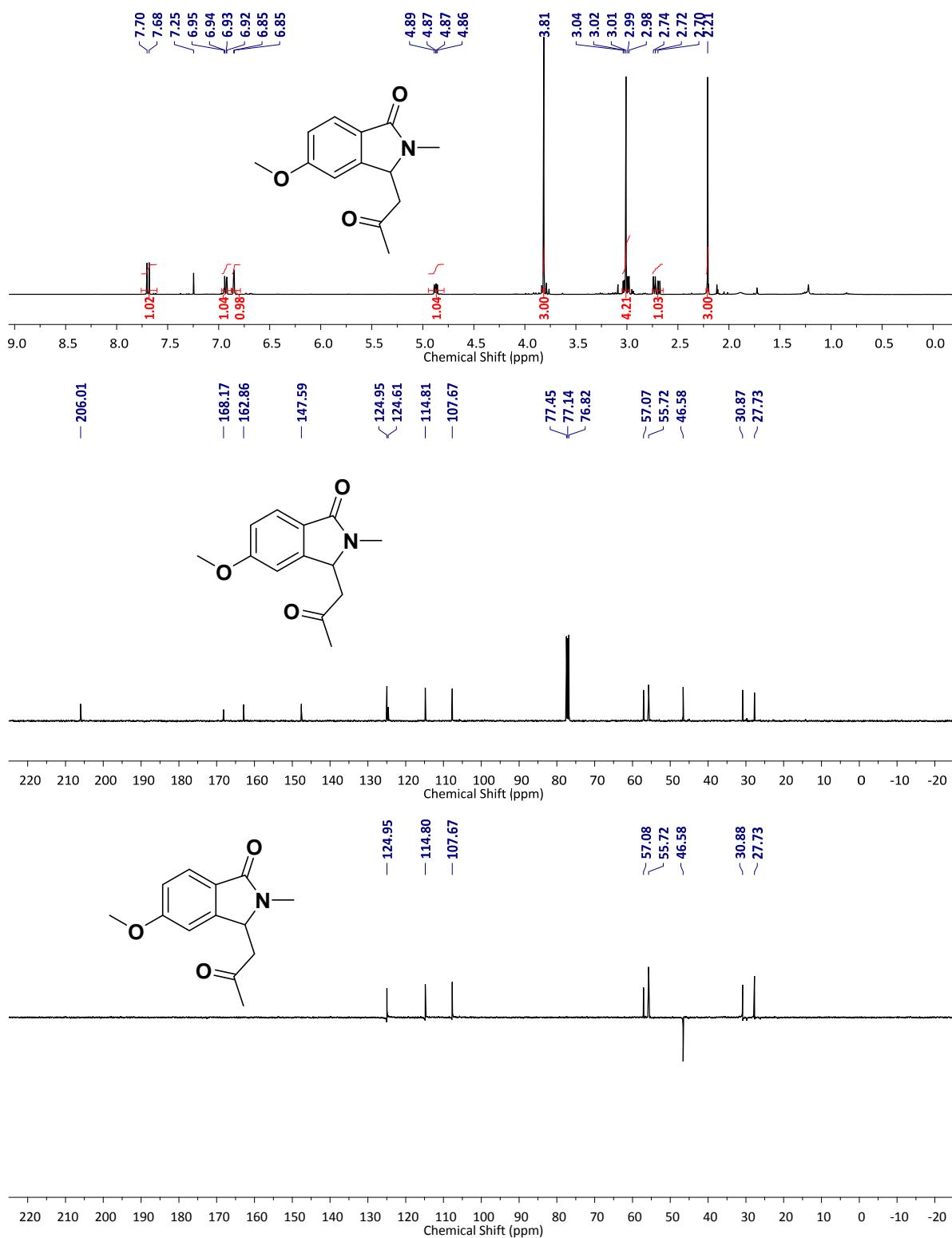
¹H and ¹³C NMR Spectra of compound 3ba.



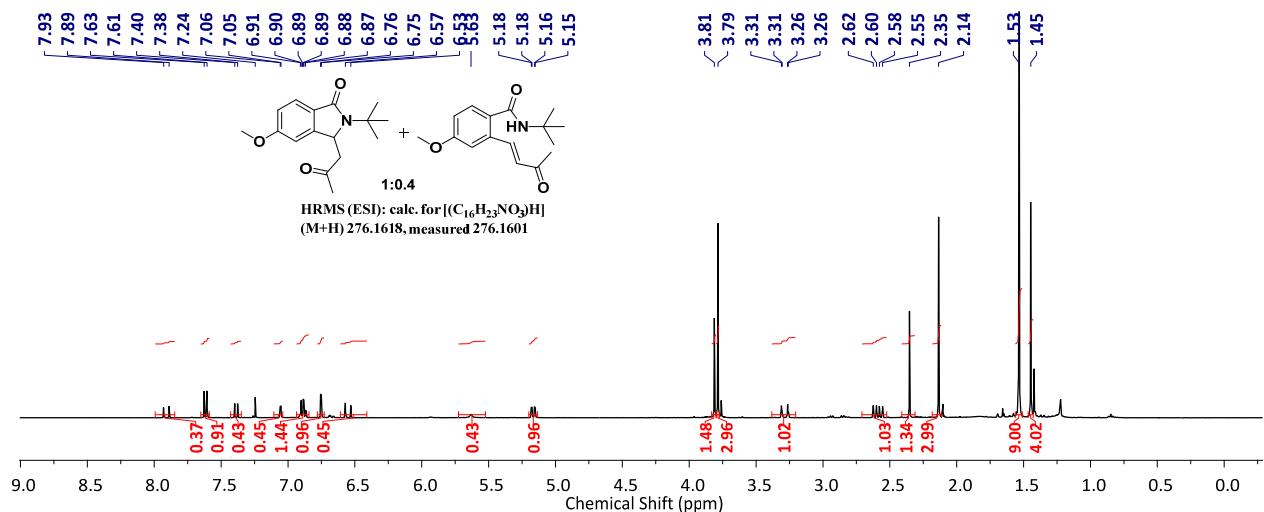
¹H and ¹³C NMR Spectra of compound 3ca.



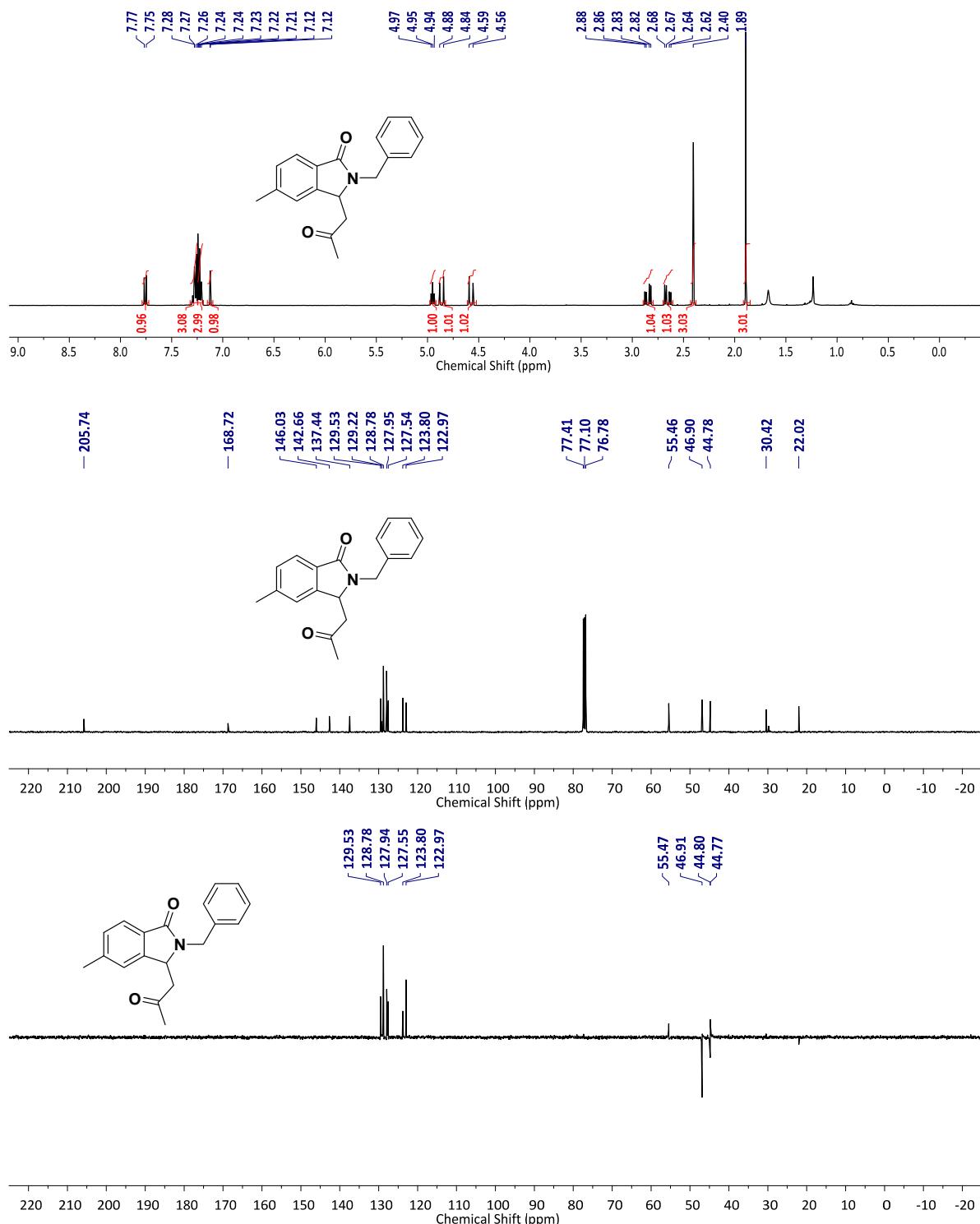
¹H and ¹³C NMR Spectra of compound 3da.



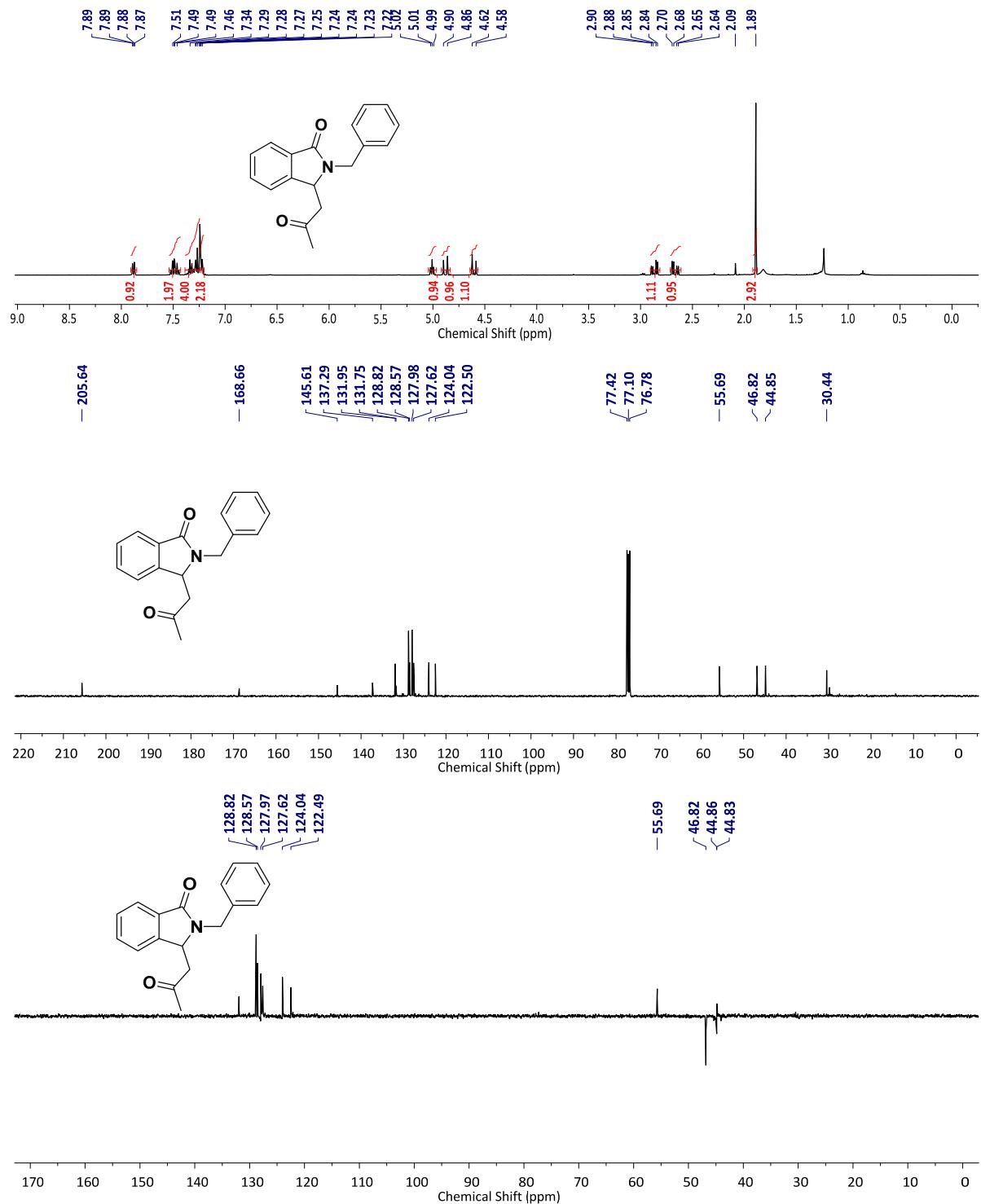
^1H and ^{13}C NMR Spectra of compounds 3ea+3ea'.



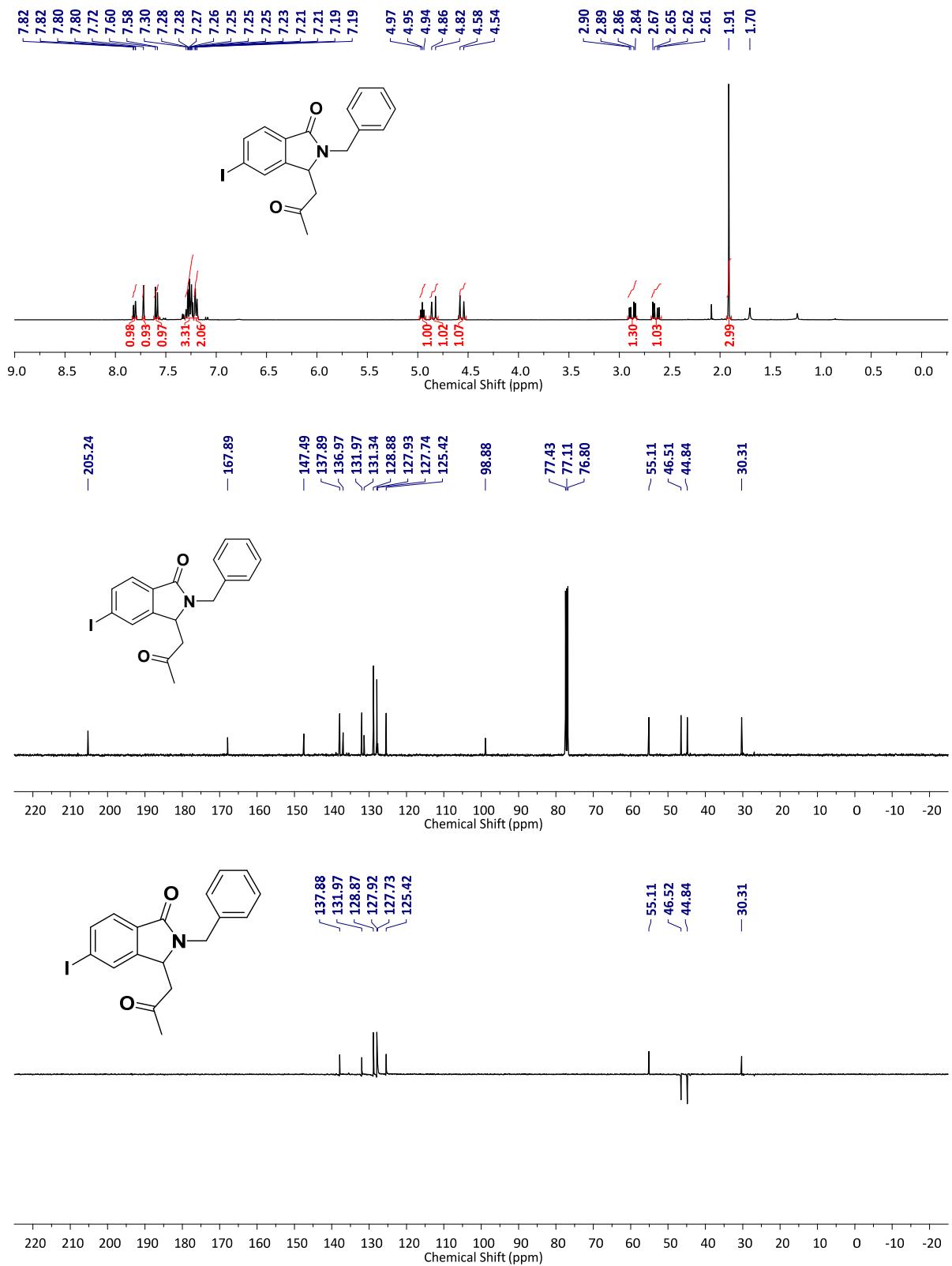
¹H and ¹³C NMR Spectra of compound 3ja.



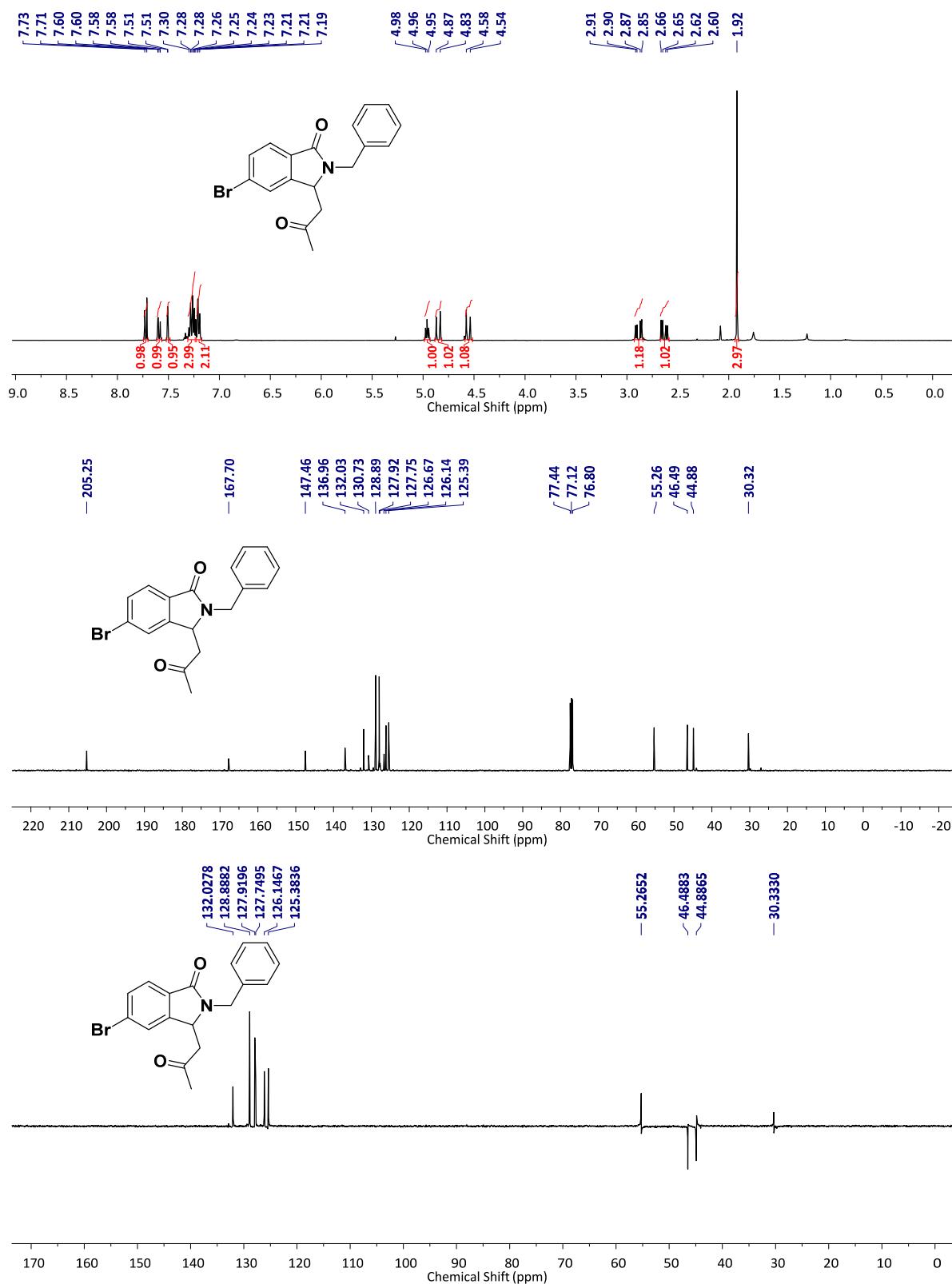
¹H and ¹³C NMR Spectra of compound 3ka.



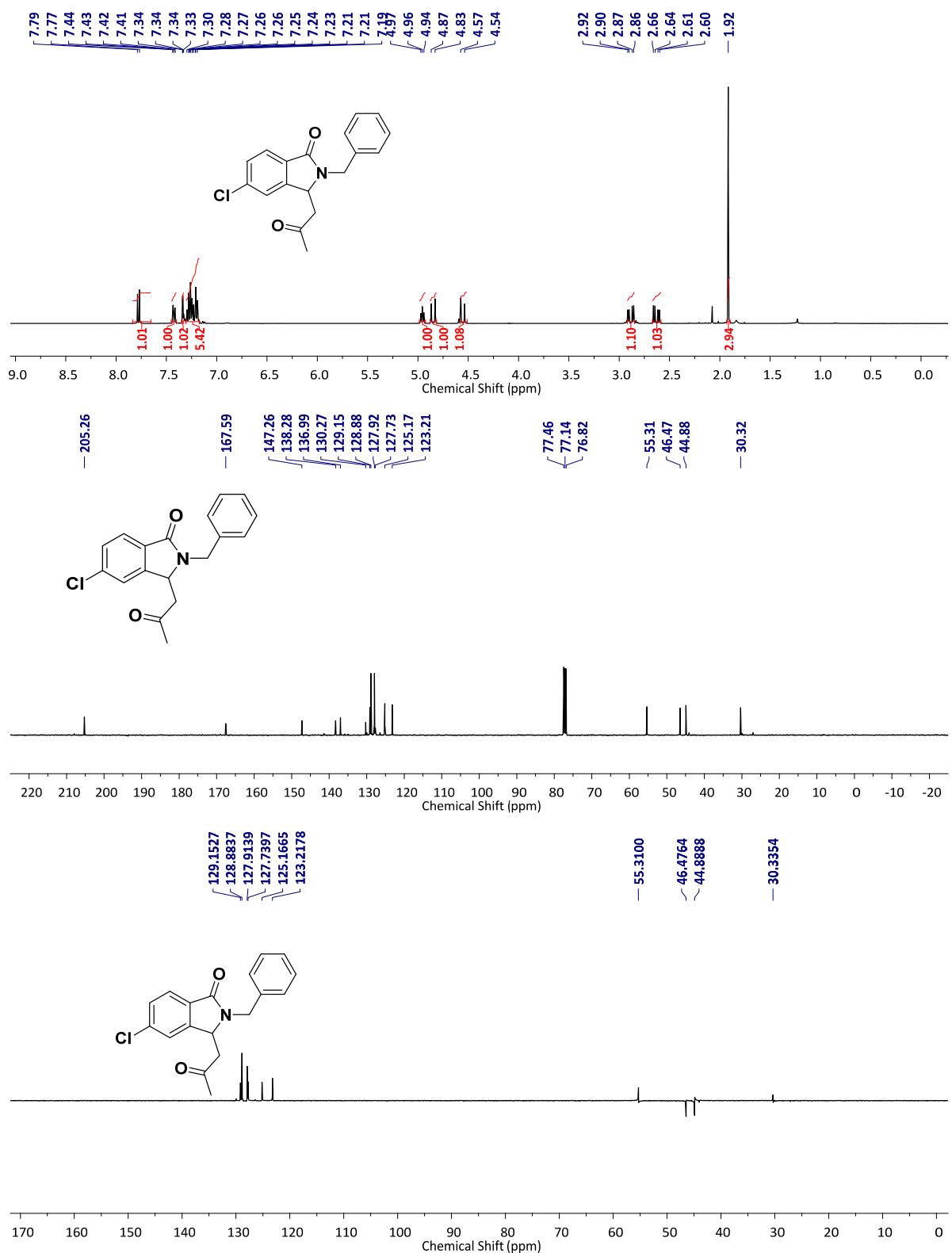
¹H and ¹³C NMR Spectra of compound 3la.



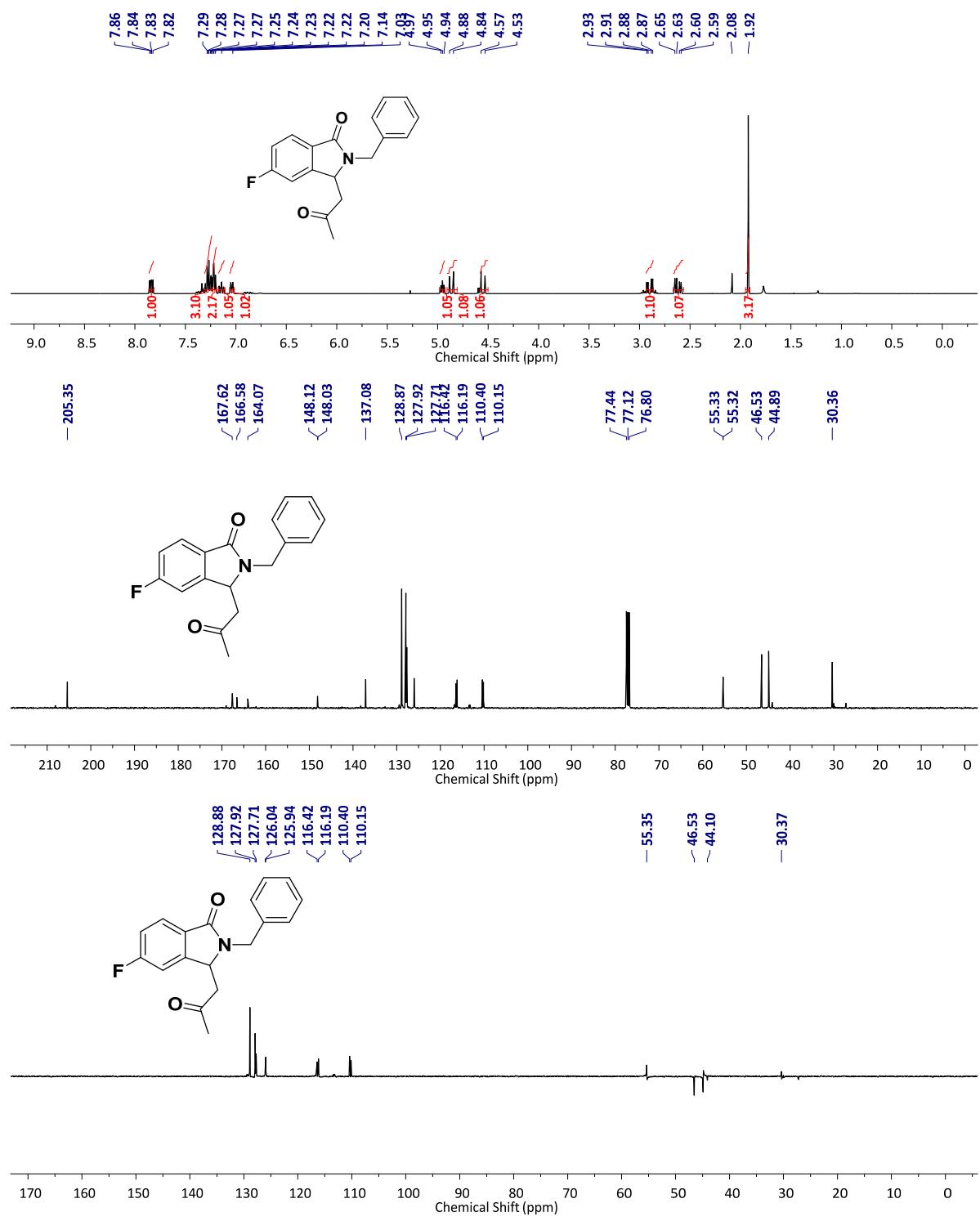
¹H and ¹³C NMR Spectra of compound 3ma.



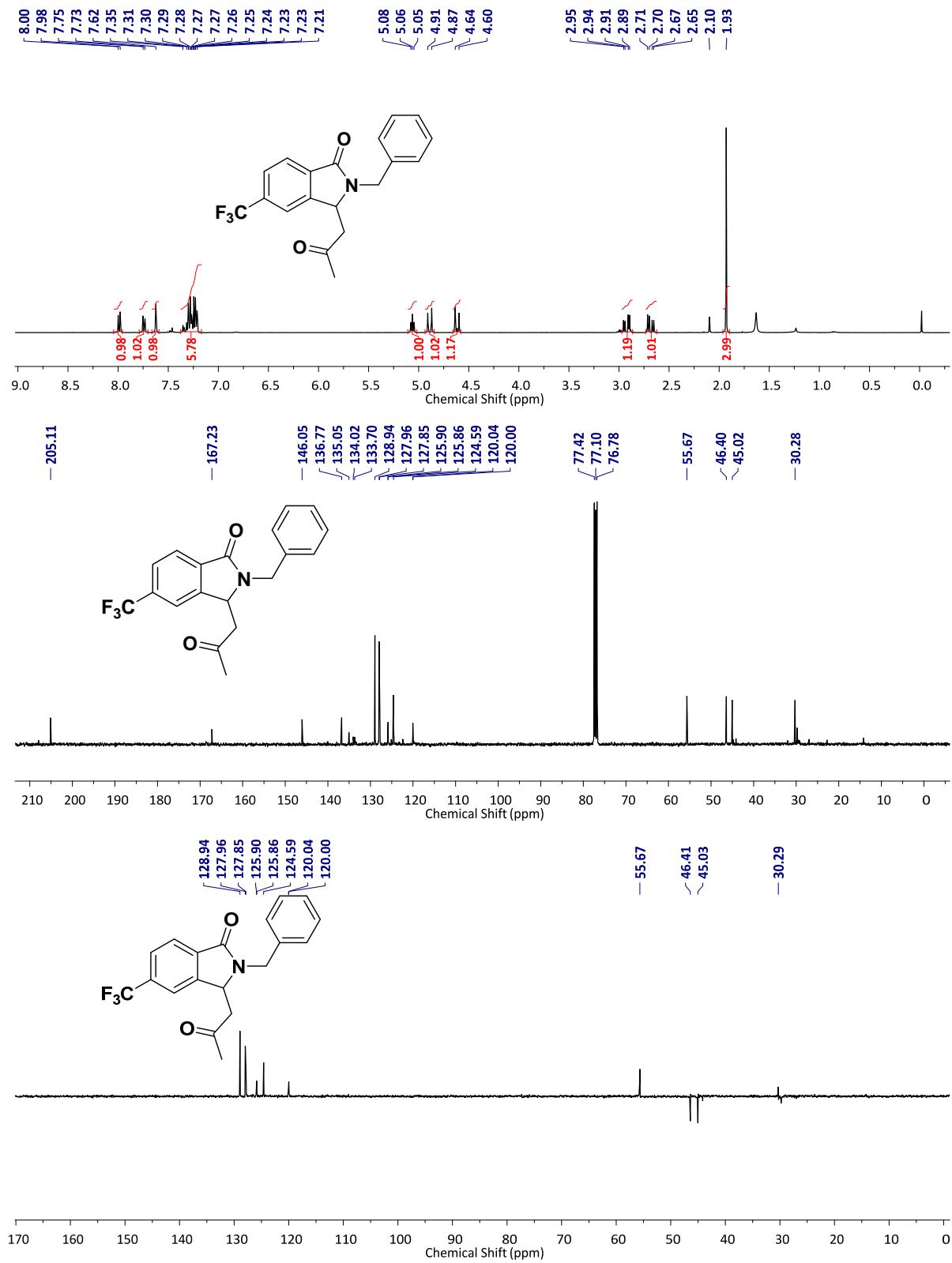
¹H and ¹³C NMR Spectra of compound 3na.



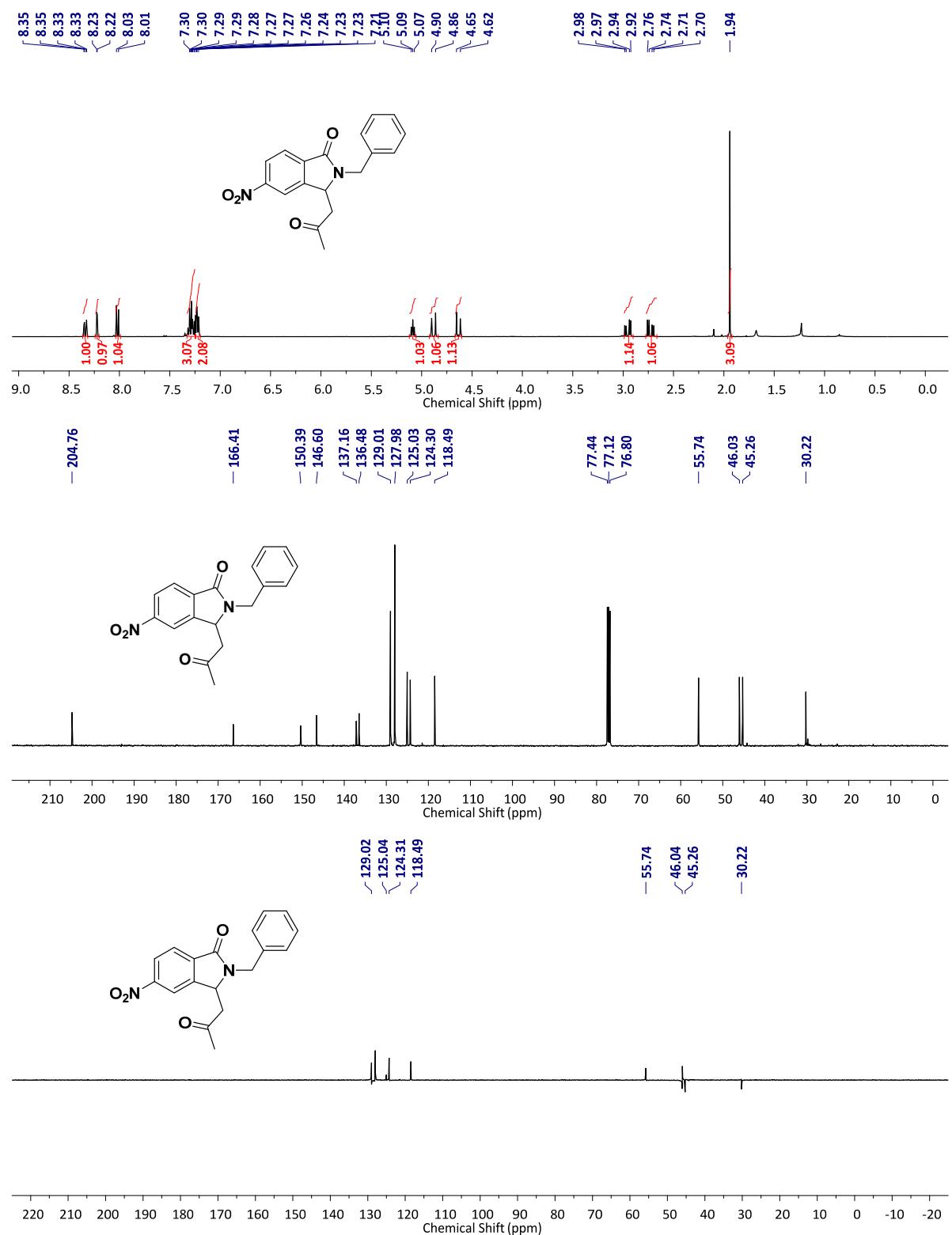
¹H and ¹³C NMR Spectra of compound 3oa.



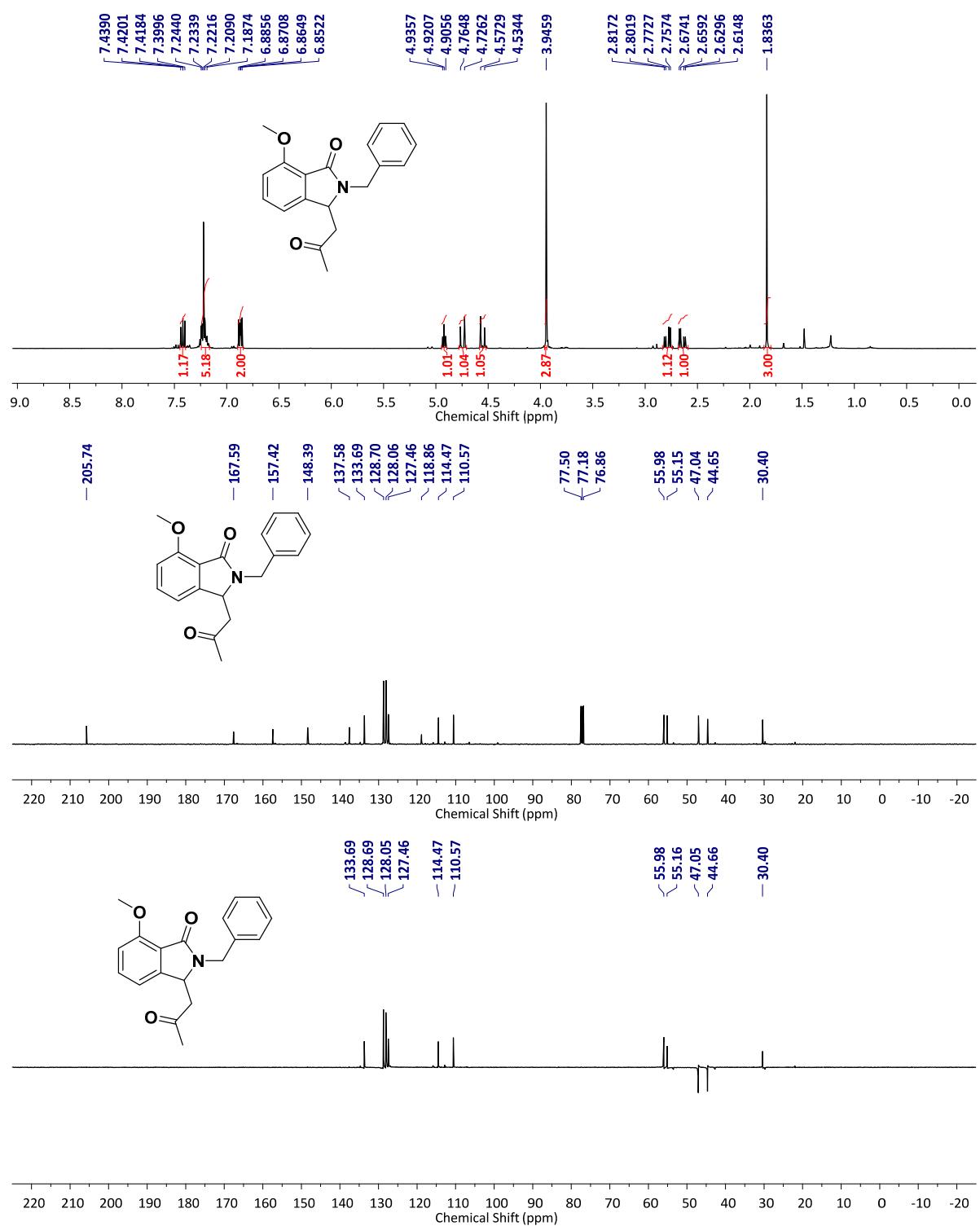
¹H and ¹³C NMR Spectra of compound 3pa.



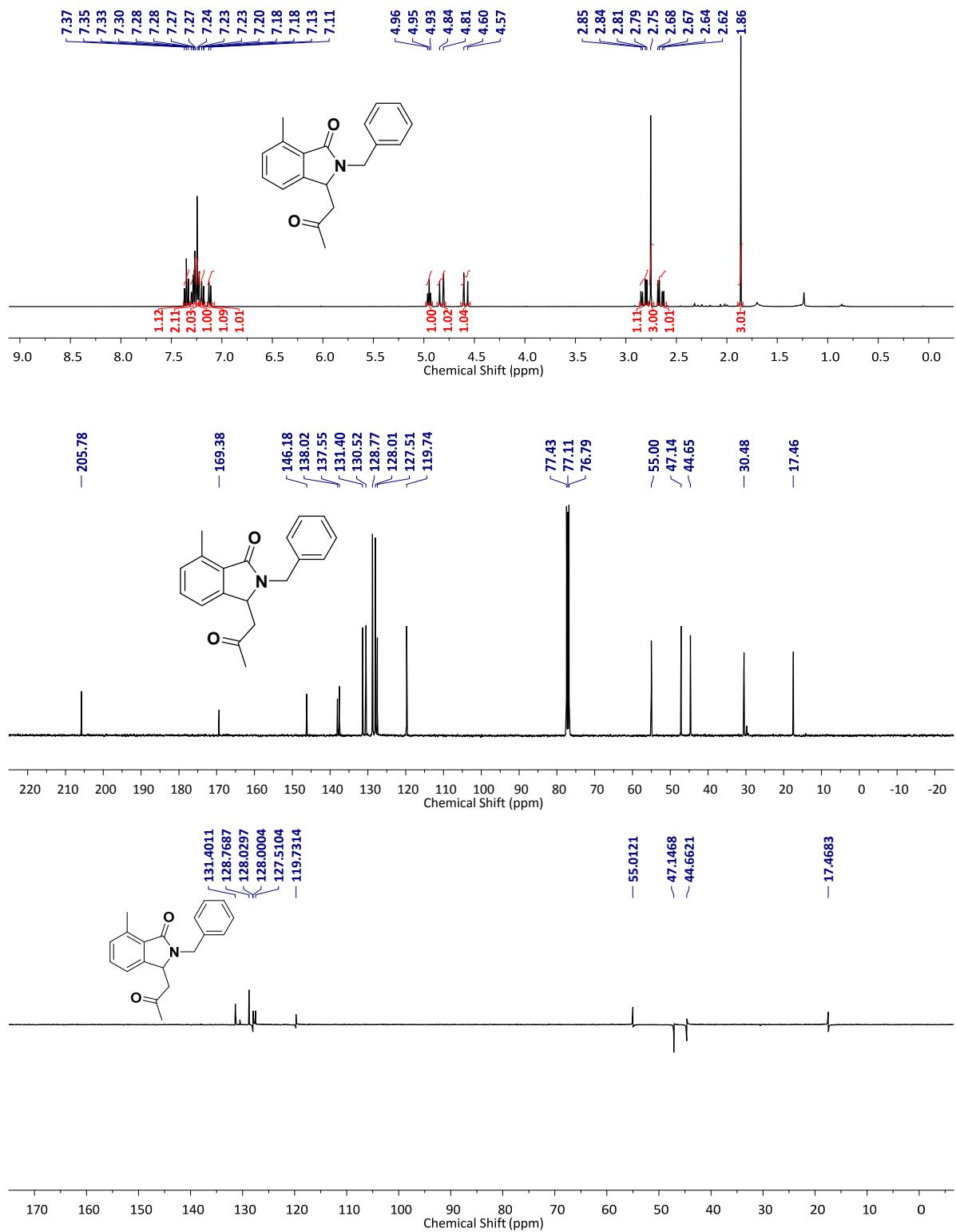
¹H and ¹³C NMR Spectra of compound 3qa.



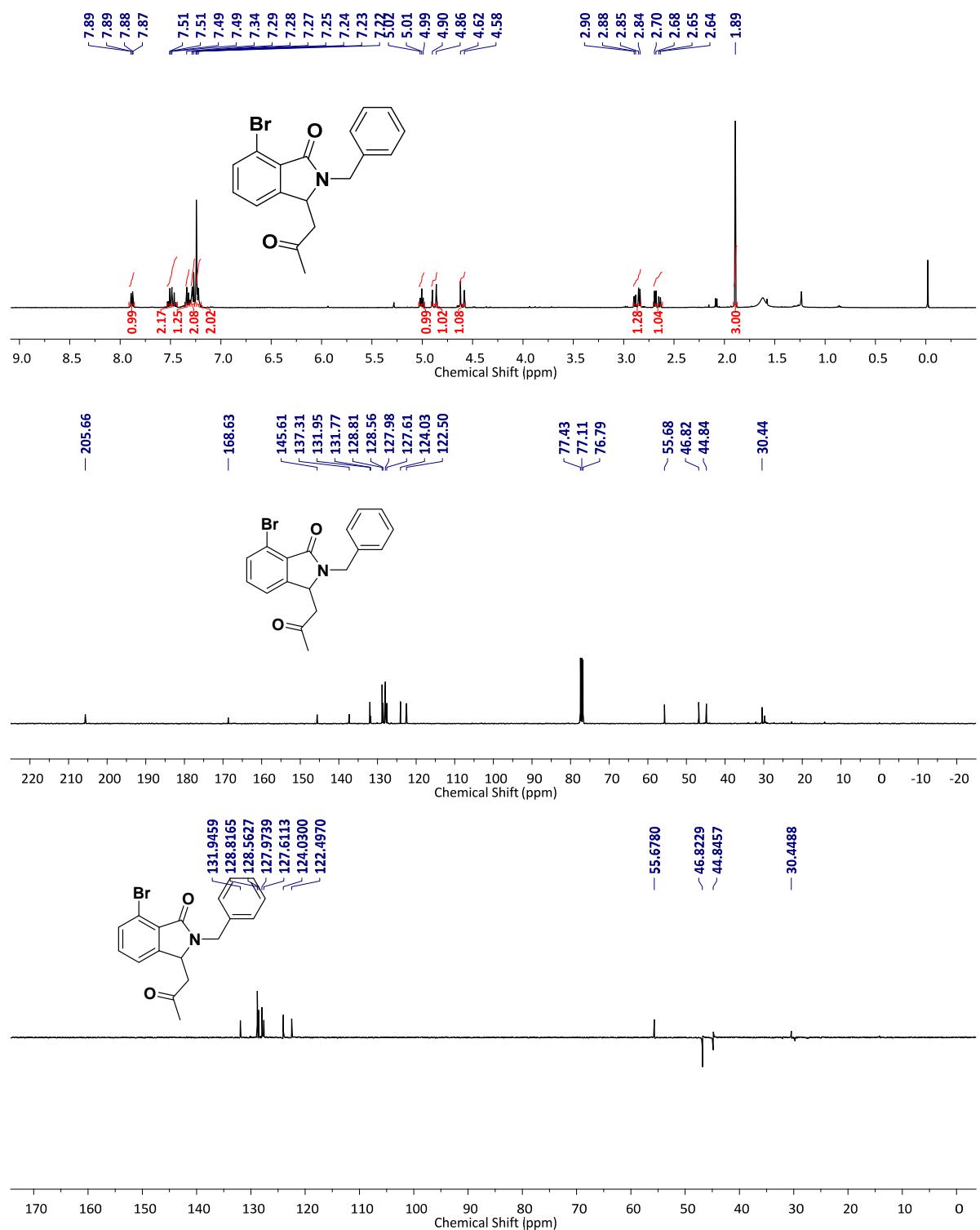
¹H and ¹³C NMR Spectra of compound 3ra.



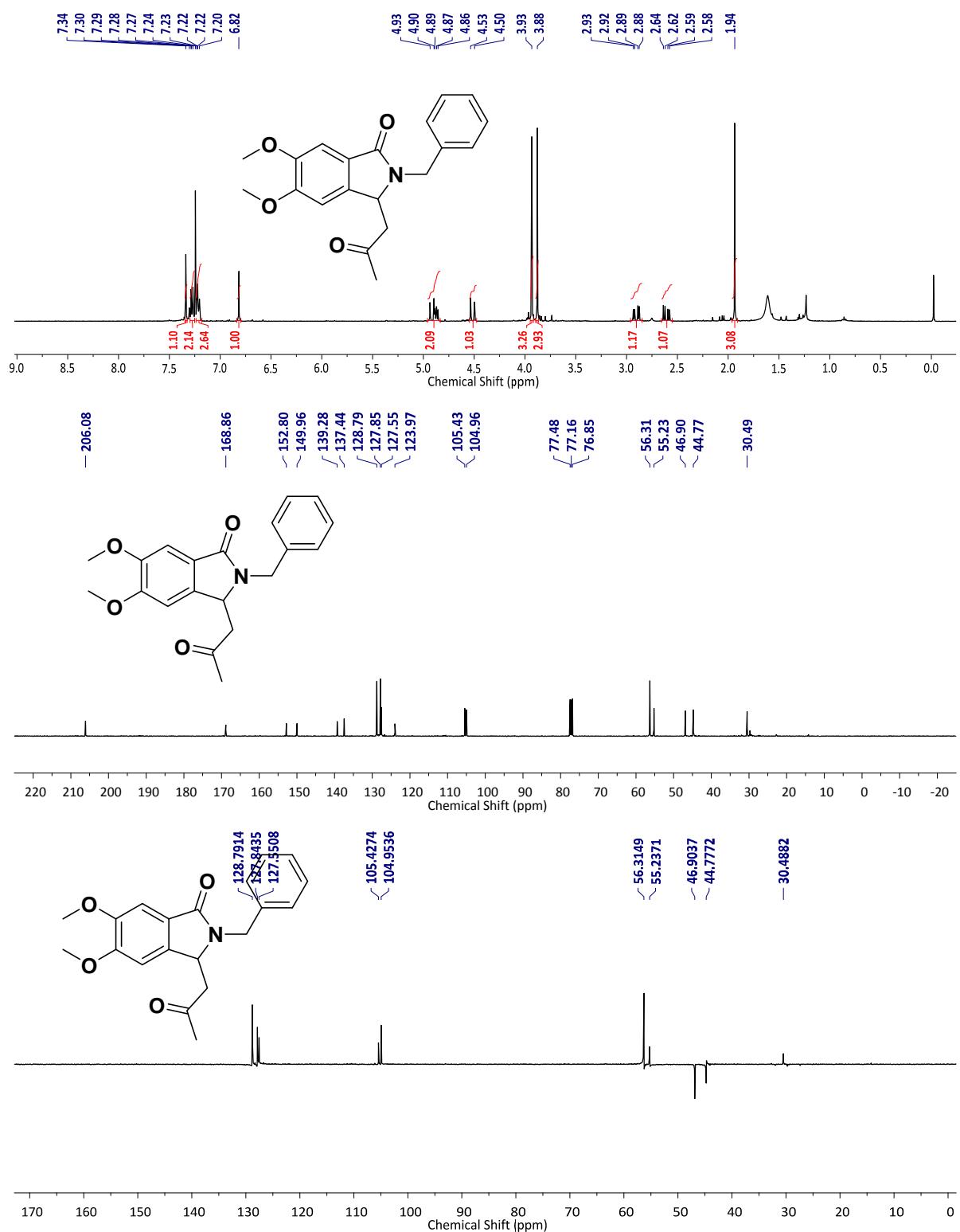
¹H and ¹³C NMR Spectra of compound 3sa.



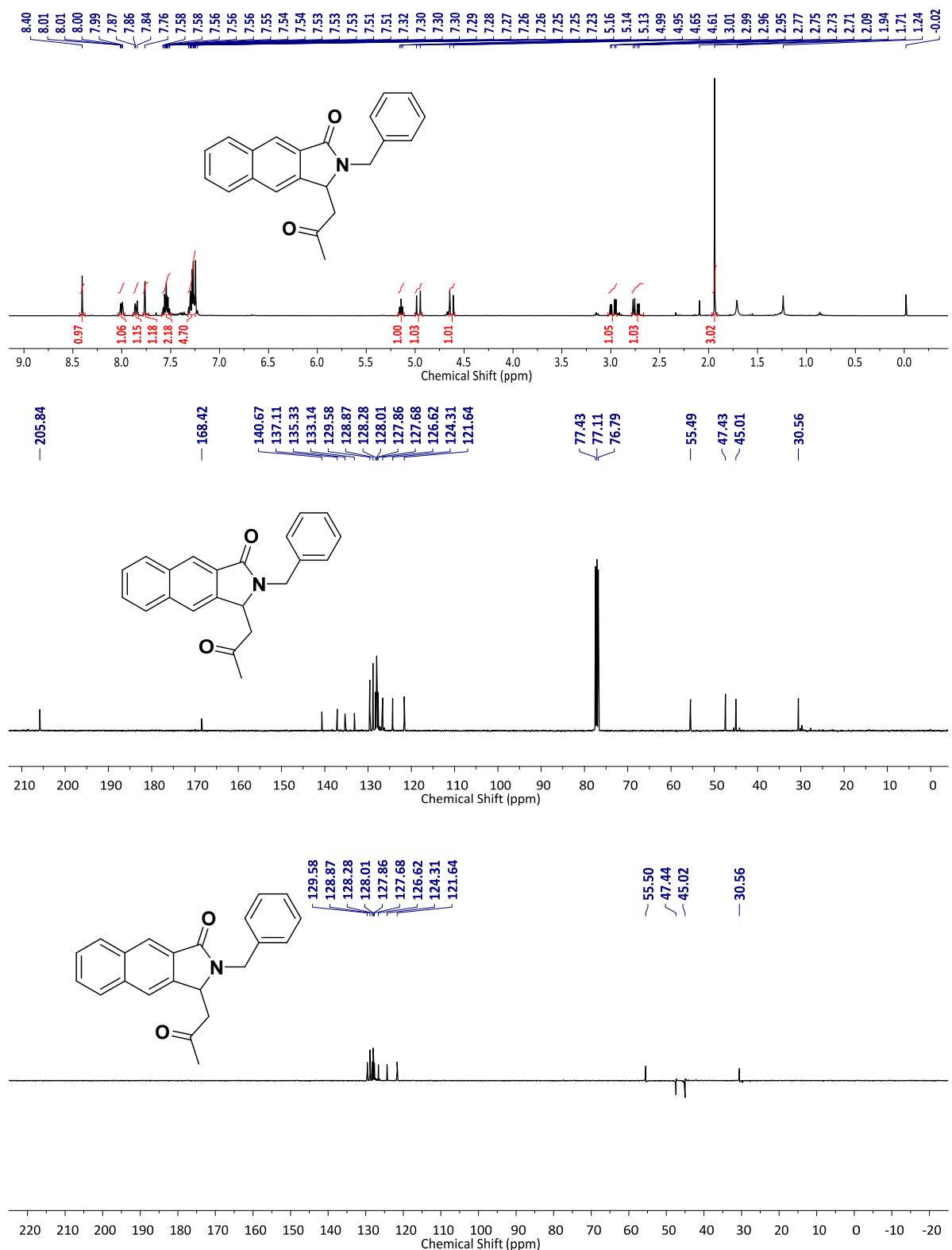
¹H and ¹³C NMR Spectra of compound 3ta.



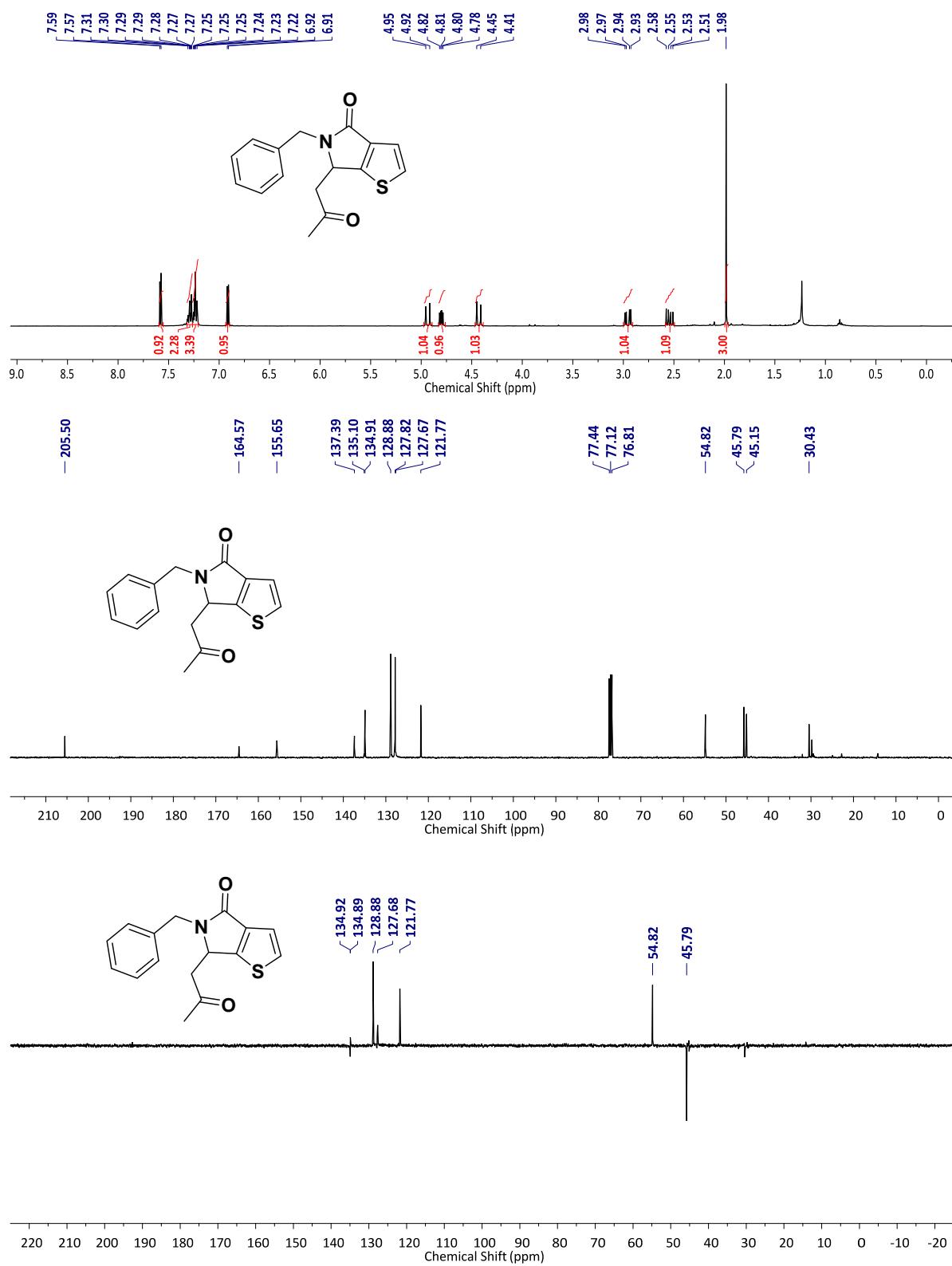
¹H and ¹³C NMR Spectra of compound 3ua.



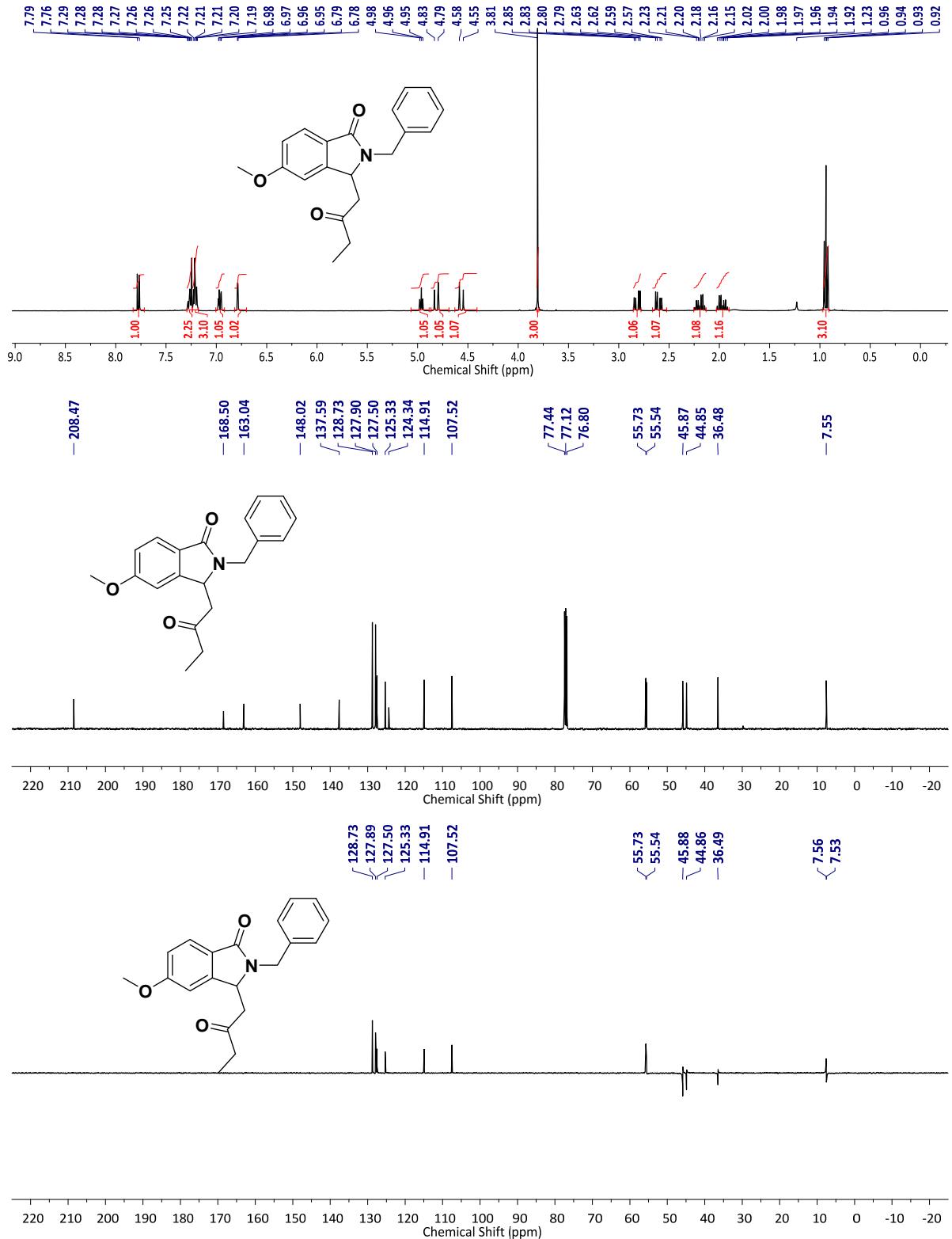
¹H and ¹³C NMR Spectra of compound 3va.



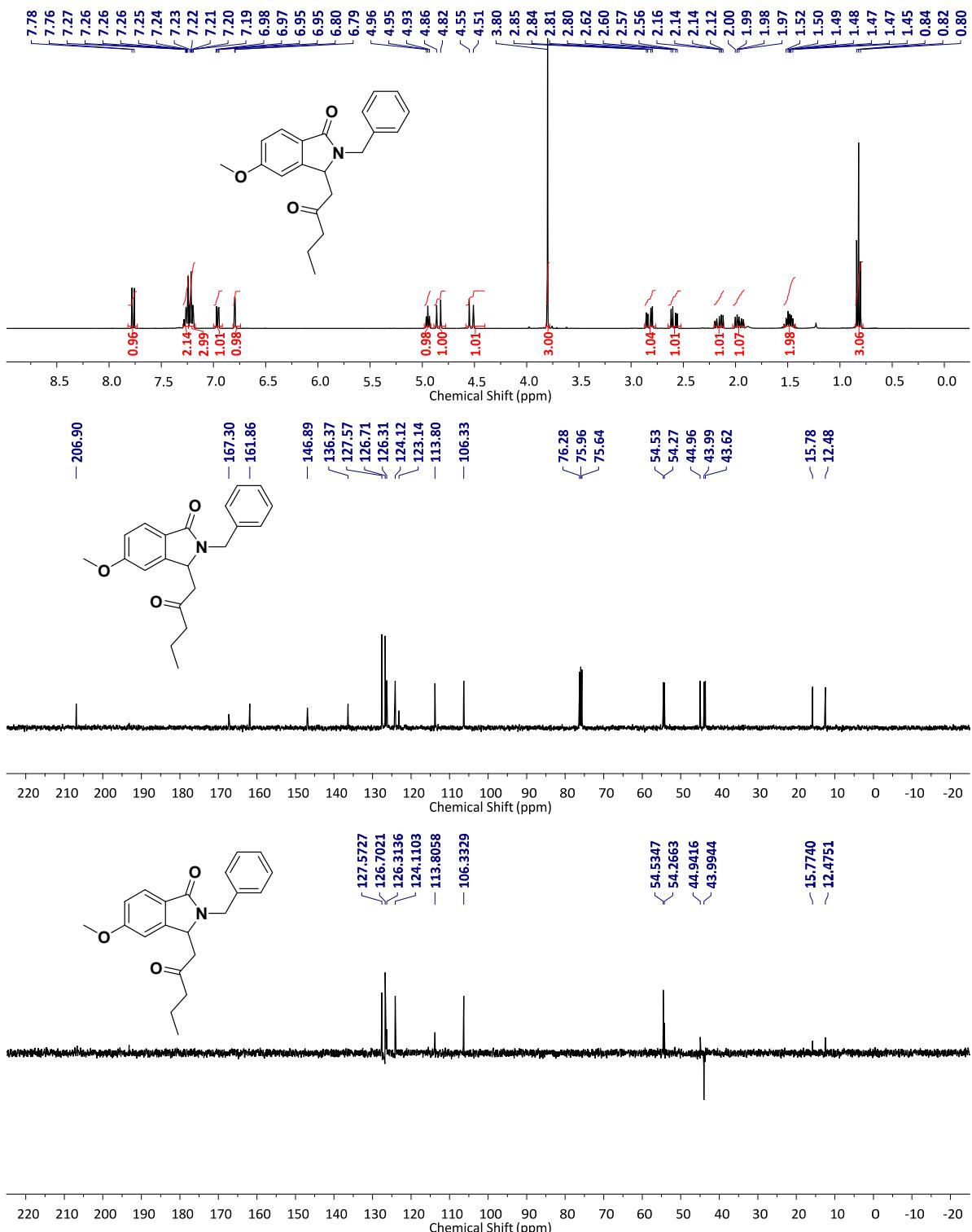
¹H and ¹³C NMR Spectra of compound 3wa.



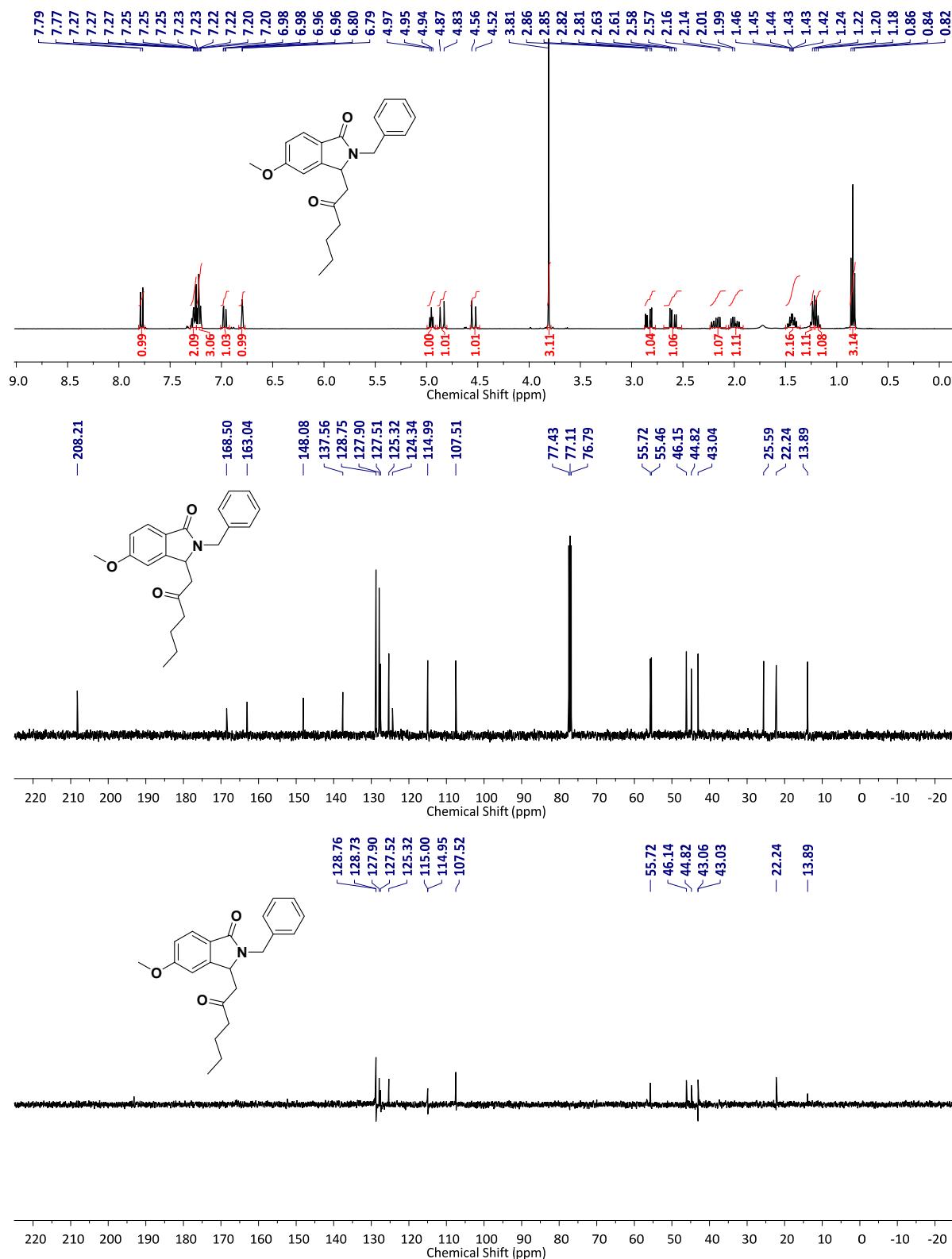
¹H and ¹³C NMR Spectra of compound 3ab.



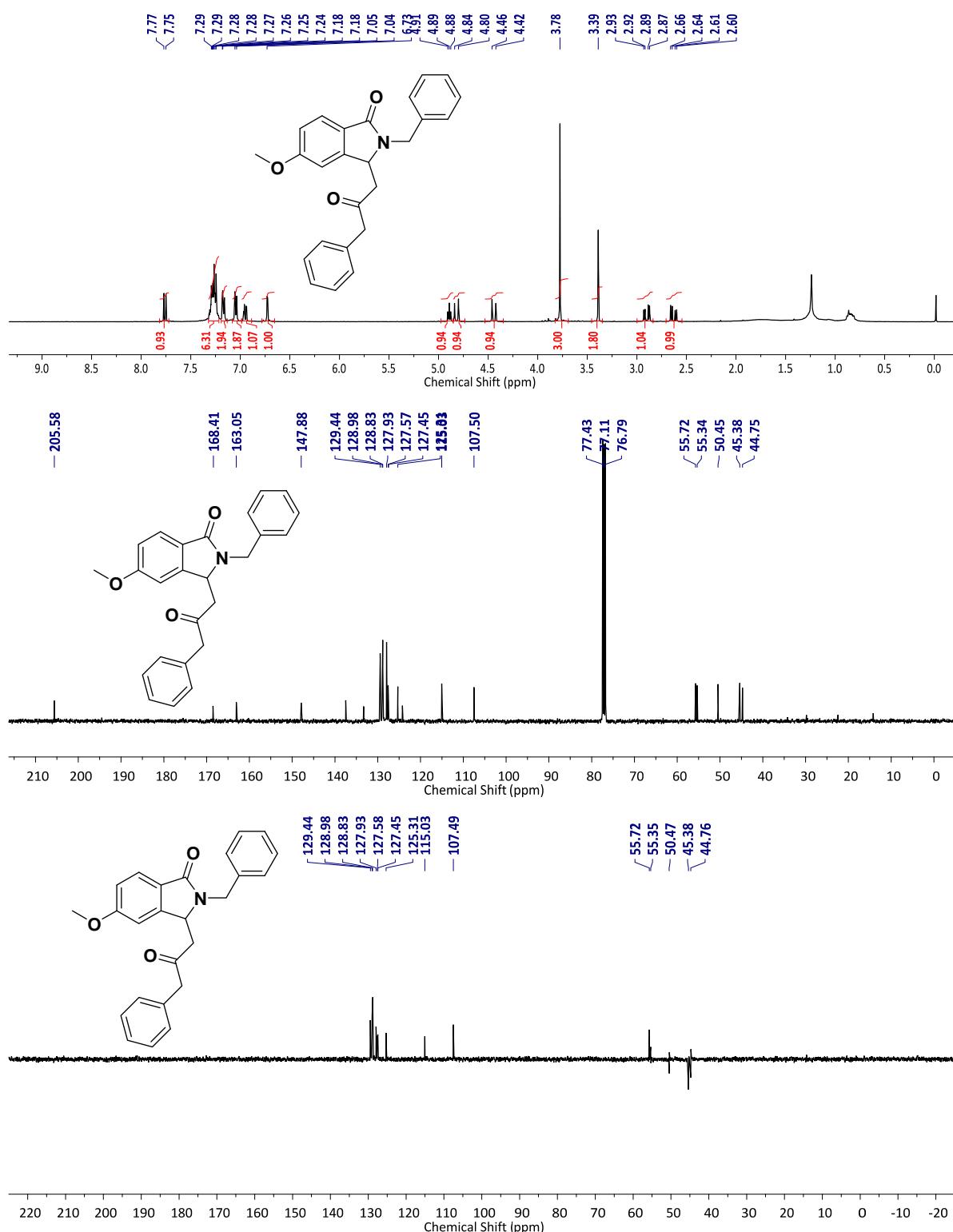
¹H and ¹³C NMR Spectra of compound 3ac.



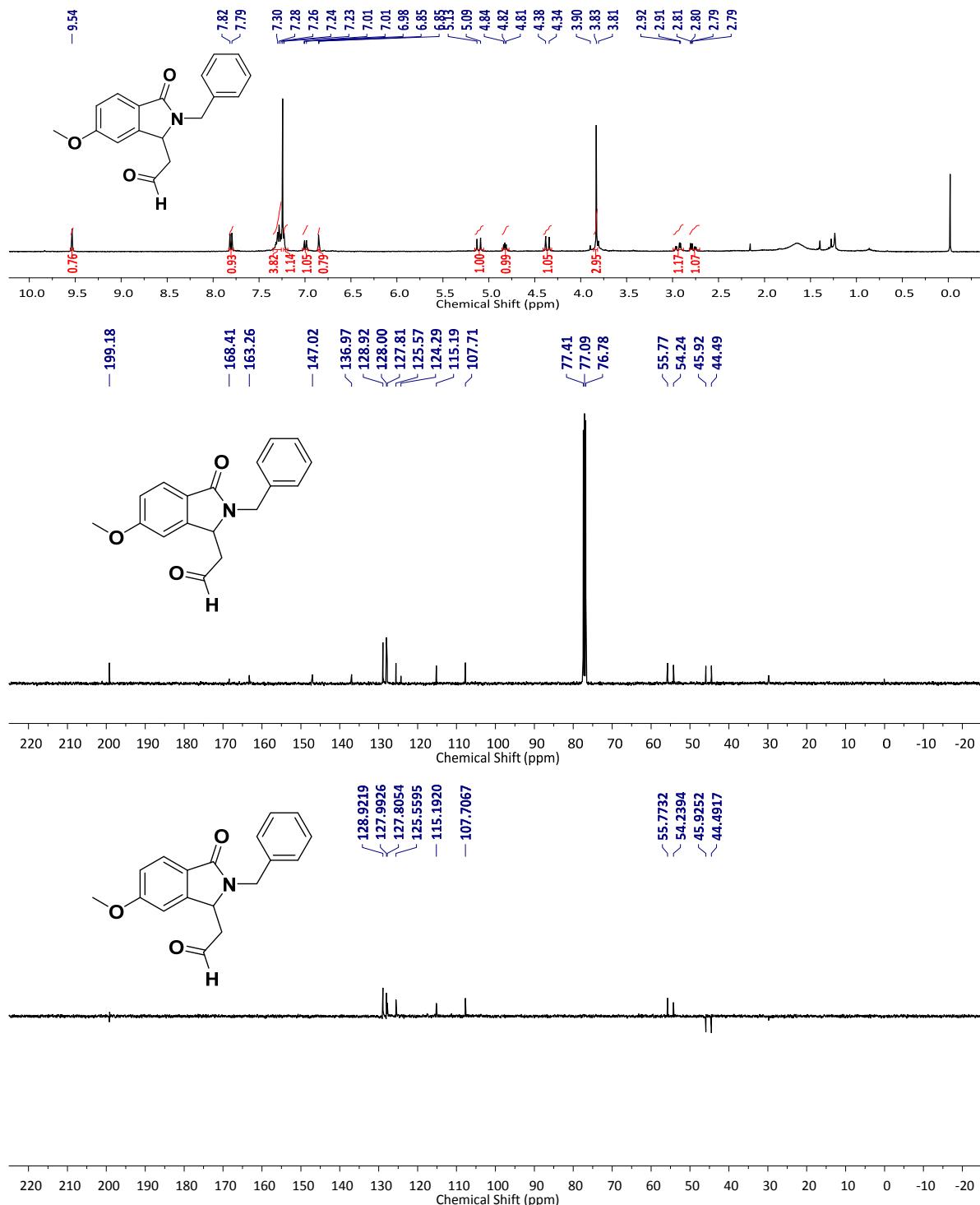
¹H and ¹³C NMR Spectra of compound 3ad.



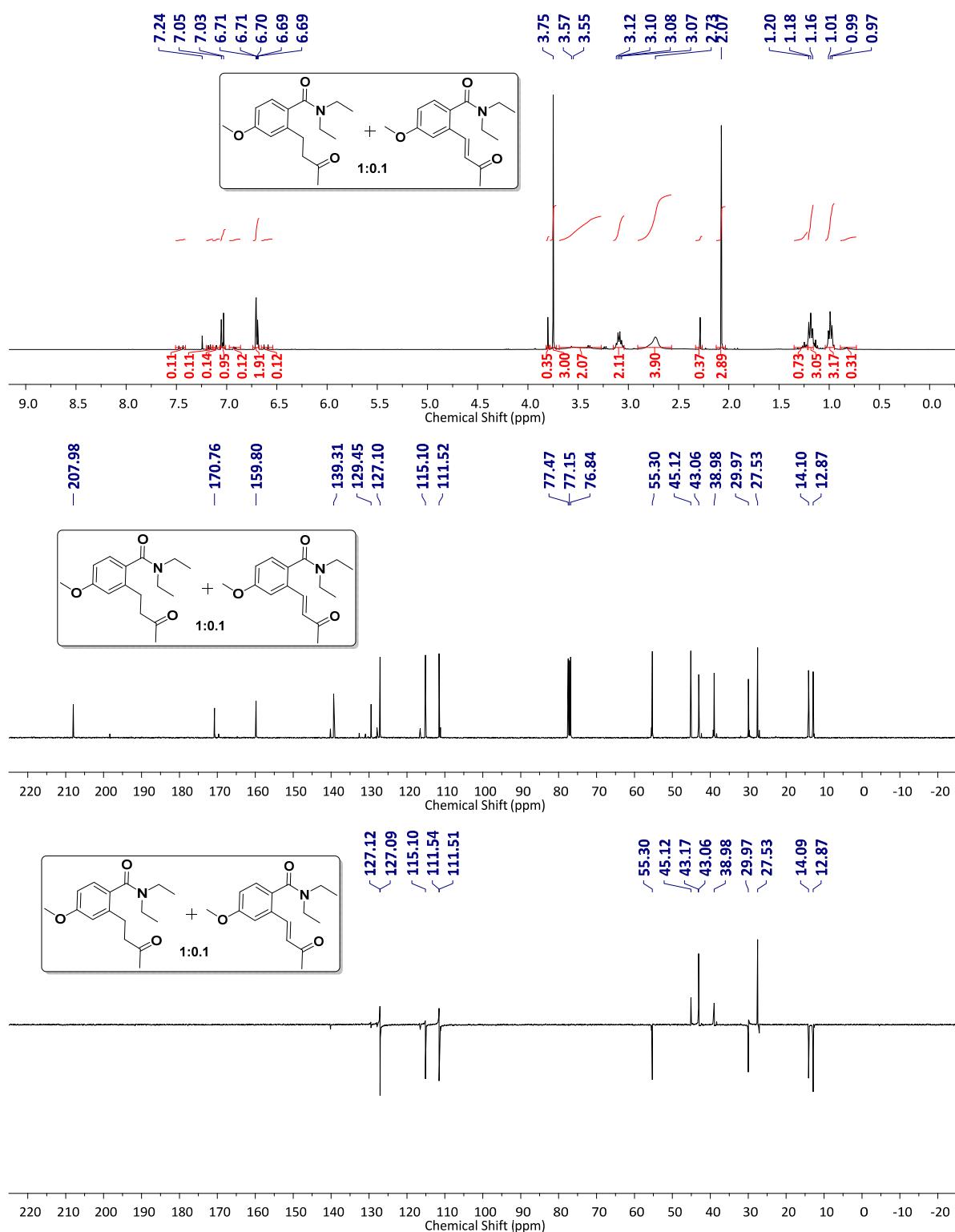
¹H and ¹³C NMR Spectra of compound 3ae.



¹H and ¹³C NMR Spectra of compound 3af.



¹H and ¹³C NMR Spectra of compound 16.



¹H and ¹³C NMR Spectra of compound 18.

