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 [{RuCl$_2$(p-cymene)}$_2$] (5.0 mol %), aromatic or heteroaromatic amide 1 (100 mg), Cu(OAc)$_2$·H$_2$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 ClCH$_2$CH$_2$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$_2$Cl$_2$, 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 [{RuCl2(p-cymene)}2] (5.0 mol %), N-benzyl-4-methoxybenzamide (1a) (100 mg), Cu(OAc)2.H2O (2.20 equiv) and AgSbF6 (20 mol %) was evacuated and purged with nitrogen gas three times (AgSbF6 was taken inside the glove box). To the tube were then added ClCH2CH2Cl (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 CH2Cl2, 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 [{RuCl2(p-cymene)}2] (50 mg), benzamide 1a (1.10 equiv), Cu(OAc)2.H2O (1.20 equiv) and AgSbF6 (4.0 equiv) was evacuated and purged with nitrogen gas three times (AgSbF6 was taken inside the glove box). To the tube were then added ClCH2CH2Cl (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**

![HRMS Spectrum](image)

**Chemical Formula:** $\text{[C}_{25}\text{H}_{27}\text{NO}_{2}\text{Ru}]\text{H}$

**Exact Mass:** 476.1164

**Found:** 476.1176
Note: In $^{13}$C NMR, we think C-Ru peak comes at $\delta$ 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 [{RuCl$_2$(p-cymene)}$_2$] (5.0 mol %), Cu(OAc)$_2$·H$_2$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), ClCH$_2$CH$_2$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$_2$Cl$_2$, 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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{19}$H$_{19}$NO$_3$)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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 [(C$_{18}$H$_{17}$NO$_3$)H] (M+H) 296.1287, measured 296.1291.

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

![Image](image1)

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.

$^1$H 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 [(C$_{18}$H$_{23}$NO$_3$)H] (M+H) 302.1756, measured 302.1758.

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

![Image](image2)

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

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 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).

\(^13\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 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 [(C\(_{13}\)H\(_{15}\)NO\(_3\))Na] (M+Na) 256.0950, measured 256.0946.

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

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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{19}$H$_{19}$NO$_2$)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{18}$H$_{17}$NO$_2$)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); **Ii** 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.

**\(^1\)H NMR (400 MHz, CDCl\(_3\))**: \( \delta \) 7.81 (dd, \( J = 8.4, 1.2 \text{ Hz, 1 H} \)), 7.72 (s, 1 H), 7.59 (d, \( J = 8.0 \text{ Hz, 1 H} \)), 7.31 – 7.22 (m, 3 H), 7.21 – 7.19 (m, 2 H), 4.95 (t, \( J = 6 \text{ Hz, 1 H} \)), 4.84 (d, \( J = 15.4 \text{ Hz, 1 H} \)), 4.56 (d, \( J = 15.4 \text{ Hz, 1 H} \)), 2.87 (dd, \( J = 18, 5.6 \text{ Hz, 1 H} \)), 2.64 (dd, \( J = 18, 6.6 \text{ Hz, 1 H} \)), 1.91 (s, 3 H).

**\(^{13}\)C NMR (100 MHz, CDCl\(_3\))**: \( \delta \) 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 \([\text{C}_{18}\text{H}_{16}\text{INO}_2\text{H}] \) (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); **Im** was taken in 100 mg; yield is 59% (73 mg).

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

**\(^1\)H NMR (400 MHz, CDCl\(_3\))**: \( \delta \) 7.72 (d, \( J = 8.0 \text{ Hz, 1 H} \)), 7.59 (dd, \( J = 8, 1.4 \text{ Hz, 1 H} \)), 7.51 (d, \( J = 1.4 \text{ Hz, 1 H} \)), 7.30 – 7.24 (m, 3 H), 7.22 – 7.17 (m, 2 H), 4.96 (t, \( J = 6 \text{ Hz, 1 H} \)), 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).

\(^{13}\text{C NMR (100 MHz, CDCl}_3\): \(\delta\) 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}] (M+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.

\(^1\text{H NMR (400 MHz, CDCl}_3\): \(\delta\) 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).

\(^{13}\text{C NMR (100 MHz, CDCl}_3\): \(\delta\) 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}] (M+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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{18}$H$_{16}$FNO$_2$)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{19}$H$_{16}$F$_3$NO$_2$)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) \( \tilde{\nu} (\text{cm}^{-1}) \): 1691, 1530, 1409, 1344, 1267, 1163, 1087, and 732.

\(^1\)H NMR (400 MHz, CDCl\(_3\)):\( \delta \) 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).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)):\( \delta \) 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 [(C\(_{18}\)H\(_{16}\)N\(_2\)O\(_4\))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) \( \tilde{\nu} (\text{cm}^{-1}) \): 1683, 1607, 1486, 1406, 1362, 1265, 1084, and 729.

\(^1\)H NMR (400 MHz, CDCl\(_3\)):\( \delta \) 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$_3$): $\delta$ 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 [(C$_{19}$H$_{19}$NO$_3$)H] (M+H) 310.1443, measured 310.1449.

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

![Structure of 3sa](image)

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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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$_3$): $\delta$ 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 [(C$_{19}$H$_{19}$NO$_2$)H] (M+H) 294.1494, measured 294.1490.

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

![Structure of 3ta](image)

Brown oil; eluent (30% ethyl acetate in hexanes) It 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.
\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 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).

\(^13\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 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 [(C\(_{18}\)H\(_{16}\)BrNO\(_2\)](M+H) 358.0443, measured 358.0448.

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

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

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.

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 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).

\(^13\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) 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.


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

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

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 205.8, 168.4, 140.6, 137.1, 135.3, 133.1, 129.5, 128.8, 128.2, 128.0, 127.8, 126.6, 124.3, 121.6, 55.5, 47.4, 45.0, 30.5.

**HRMS (ESI):** calc. for [(C$_{22}$H$_{19}$NO$_2$)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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, 30.5.

**HRMS (ESI):** calc. for [(C$_{16}$H$_{15}$NO$_2$S)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{20}$H$_{21}$NO$_3$)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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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$_3$): $\delta$ 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 [(C$_{21}$H$_{23}$NO$_3$)H] (M+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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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$_3$): $\delta$ 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 [(C$_{22}$H$_{25}$NO$_3$)H] (M+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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{25}$H$_{23}$NO$_3$)H] (M+H) 386.1756, measured 386.1765.

2-(2-Benzyl-6-methoxy-3-oxoisoiindol-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.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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 \text{ Hz}, 1 \text{ H}), 4.85 - 4.76 (\text{m}, 1 \text{ H}), 4.36 (d, \( J = 15.4 \text{ Hz}, 1 \text{ H}), 3.83 (s, 3 \text{ H}), 2.94 (dd, \( J = 17.8, 4.6 \text{ Hz}, 1 \text{ H}), 2.77 (dd, \( J = 17.8, 6.4, 1 \text{ H}).

\(^{13}\text{C NMR (100 MHz, CDCl}_3\)): \( \delta \) 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+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), \(1\)w was taken in 100 mg; yield is 47 % (62 mg).

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

\(^1\text{H NMR (400 MHz, CDCl}_3\)): \( \delta \) 8.30 (d, \( J = 16.6 \text{ Hz}, 1 \text{ H}), 7.40 - 7.25 (\text{m}, 7 \text{ H}), 6.49 (d, \( J = 16.6 \text{ Hz}, 1 \text{ H}), 6.35 (s, 1 \text{ H}), 4.60 (d, \( J = 5.6 \text{ Hz}, 2 \text{ H}), 2.35 (s, 3 \text{ H}).

\(^{13}\text{C NMR (50 MHz, CDCl}_3\)): \( \delta \) 199.6, 161.9, 139.9, 137.6, 136.2, 135.4, 130.4, 128.9, 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+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 %), \(\text{Cu(OAc)}_2\text{H}_2\text{O} \) (2.20 eq) and \(\text{AgSbF}_6 \) (20 mol %) was evacuated and purged with nitrogen gas three times (\(\text{AgSbF}_6 \) 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 \(\text{CH}_2\text{Cl}_2\),
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.

\textit{N,N-Diethyl-4-methoxy-2-(3-oxobutyl)benzamide (16).}

![Chemical structure](image)

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

\textbf{IR (ATR) }\tilde{\nu} (\text{cm}^{-1}): 1712, 1611, 1463, 1430, 1243, 1088, 905 and 819.

\textbf{\textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): }\delta 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).

\textbf{\textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}): }\delta 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.

\textbf{HRMS (ESI): }calc. for [(C\textsubscript{16}H\textsubscript{23}NO\textsubscript{3})H] (M+H) 278.1756, measured 278.1758.

\textit{N-Benzyl-4-methoxy-2-(3-oxobutyl)benzamide (18).}

![Chemical structure](image)

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

\textbf{IR (ATR) }\tilde{\nu} (\text{cm}^{-1}): 1705, 1627, 1419, 1306, 1247, 1166, 1054 and 735.
$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 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).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 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 [(C$_{19}$H$_{21}$NO$_3$)H] (M+H) 312.1600, measured 312.1594.
$^1$H and $^{13}$C NMR Spectra of compound 3aa.
$^1$H and $^{13}$C NMR Spectra of compound 3ba.
$^1$H and $^{13}$C NMR Spectra of compound 3ca.
$^1$H and $^{13}$C NMR Spectra of compound 3da.
$^1$H and $^{13}$C NMR Spectra of compounds 3ea+3ea’.
$^1$H and $^{13}$C NMR Spectra of compound 3ja.
$^1$H and $^{13}$C NMR Spectra of compound 3ka.
$^1$H and $^{13}$C NMR Spectra of compound 3la.
$^1$H and $^{13}$C NMR Spectra of compound 3ma.
$^1$H and $^{13}$C NMR Spectra of compound 3na.
$^1$H and $^{13}$C NMR Spectra of compound 3oa.
$^1\text{H}$ and $^{13}\text{C}$ NMR Spectra of compound 3pa.
$^1$H and $^{13}$C NMR Spectra of compound 3qa.
$^1$H and $^{13}$C NMR Spectra of compound 3ra.
$^1$H and $^{13}$C NMR Spectra of compound 3sa.
$^1$H and $^{13}$C NMR Spectra of compound 3ta.
$^{1}$H and $^{13}$C NMR Spectra of compound 3ua.
$^1$H and $^{13}$C NMR Spectra of compound 3va.
$^1$H and $^{13}$C NMR Spectra of compound 3wa.
$^1$H and $^{13}$C NMR Spectra of compound 3ab.
$^1$H and $^{13}$C NMR Spectra of compound 3ac.
$^1$H and $^{13}$C NMR Spectra of compound 3ad.
$^1$H and $^{13}$C NMR Spectra of compound 3ae.
\(^1H\) and \(^{13}C\) NMR Spectra of compound 3af.

\[\text{Chemical Shift (ppm)}\]

\[\begin{array}{c}
192.13 \quad 168.91 \\
143.92 \quad 128.00 \\
127.66 \quad 124.59 \\
115.13 \\
107.71 \\
77.41 \\
76.78 \quad 55.77 \\
54.34 \\
49.48
\end{array}\]
$^{1}H$ and $^{13}C$ NMR Spectra of compound 16.
$^1$H and $^{13}$C NMR Spectra of compound 18.