Intramolecular Cyclization with Nitrenium Ions Generated by Treatment of *N*-Acylaminophthalimides with Hypervalent Iodine Compounds: Formation of Lactams and Spiro-Fused Lactams

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General Methods. Melting points are uncorrected. ^1H NMR spectra were recorded on 270 and 500 MHz spectrometers, and ^{13}C NMR were recorded on 67.8 and 125.7 MHz spectrometers using CDCl_3 as solvent, unless otherwise noted. Chemical shifts are reported as ppm downfield from tetramethylsilane (TMS) in δ unit. Mass spectra were measured with direct inlet system at 70 eV.

Materials. All solvents were obtained from commercial suppliers and used without further purification. PIFA and HFIP were commercially available. 4-Chlorophenyl iodine(III) bis(trifluoroacetate) was prepared following literature methods.¹⁵ The starting materials **1a-t** were prepared by the reaction of the corresponding acid chlorides or aryl chloroformates with *N*-aminophthalimide in dry pyridine.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-2-phenylacetamide (1a).** White crystals: mp 185-187 °C (EtOAc/*n*-hexane); IR (KBr) 3190, 1805, 1750, 1675, 705 cm^{-1} ; ^1H NMR (270 MHz) δ 3.82 (s, 2H), 7.29-7.45 (m, 6H), 7.74-7.82 (m, 2H), 7.85-7.92 (m, 2H); ^{13}C NMR (67.8 MHz) δ 41.2, 123.8, 127.5, 128.9, 129.3, 129.8, 133.0, 134.5, 164.8, 169.5; EI-MS m/z 280 (M^+ , 7.1), 118 (100), 91 (48.2). Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_3$: C, 68.56; H, 4.32; N, 9.99. Found: C, 68.55; H, 4.16; N, 9.93.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-phenylpropionamide (1b).** White crystals: mp 150-151 °C (EtOAc/*n*-hexane); IR (KBr) 3240, 1740, 1680, 705 cm^{-1} ; ^1H NMR (270 MHz) δ 2.37 (t, J = 7.8 Hz, 2H), 3.08 (t, J = 7.8 Hz, 2H), 7.20-7.38 (m, 6H), 7.77-7.84 (m, 2H), 7.88-7.94 (m, 2H); ^{13}C NMR (67.8 MHz, $\text{DMSO}-d_6$) δ 31.3, 35.3, 124.4, 126.7, 128.9, 129.0, 130.1, 135.9, 141.3, 165.8,

171.5; EI-MS m/z 294 (M^+ , 23.6), 162 (100), 105 (38.5), 91 (40.9). Anal. Calcd for $C_{17}H_{14}N_2O_3$: C, 69.38; H, 4.79; N, 9.52. Found: C, 69.44; H, 4.54; N, 9.38.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-4-phenylbutyramide (1c).** White crystals: mp 118-119 °C (benzene/*n*-hexane); IR (KBr) 3275, 1810, 1755, 1680, 715 cm⁻¹; ¹H NMR (270 MHz) δ 2.09 (quint, *J* = 7.4 Hz, 2H), 2.41 (t, *J* = 7.4 Hz, 2H), 2.76 (t, *J* = 7.4 Hz, 2H), 7.17-7.34 (m, 6H), 7.77-7.83 (m, 2H), 7.88-7.95 (m, 2H); ¹³C NMR (67.8 MHz) δ 26.6, 32.9, 34.7, 123.9, 125.9, 128.3, 128.5, 129.9, 134.6, 141.0, 165.1, 171.3; EI-MS *m/z* 308 (M⁺, 8.1), 204 (60.1), 162 (100), 147 (41.7), 91 (66.4). Anal. Calcd for C₁₈H₁₆N₂O₃: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.08; H, 5.23; N, 9.05.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)biphenyl-2-carboxamide (1d).** White crystals: mp 198-201 °C (EtOAc/*n*-hexane); IR (KBr) 3150, 1790, 1730, 1660, 1120, 705, 695 cm⁻¹; ¹H NMR (270 MHz) δ 7.24 (s, 1H), 7.43-7.63 (m, 8H), 7.74-7.81 (m, 2H), 7.86-7.96 (m, 3H); ¹³C NMR (67.8 MHz) δ 123.8, 127.6, 128.1, 128.8, 129.1, 129.9, 130.0, 130.7, 130.9, 131.5, 134.5, 139.0, 140.8, 164.7, 167.3; EI-MS *m/z* 342 (M⁺, 3.2), 181 (100), 152 (29.3). Anal. Calcd for C₂₁H₁₄N₂O₃: C, 73.68; H, 4.12; N, 8.18. Found: C, 73.75; H, 4.04; N, 8.22.

(1,3-Dioxo-1,3-dihydroisoindol-2-yl)carbamic acid phenyl ester (1e). White crystals; mp 174-177 °C (CHCl₃/*n*-hexane); IR (KBr) 3375, 1805, 1760, 1740, 1220, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆, rotamers) • 7.03 (d, *J* = 7.8 Hz, 0.3H), 7.15-7.35 (m, 2.7H), 7.35-7.55 (m, 2H), 7.88-8.10 (m, 4H), 10.89 (br s, 1H); ¹³C NMR (67.8 MHz, DMSO-*d*₆, rotamers) • 121.2, 121.3, 123.8, 123.8, 125.8, 125.9, 129.0, 129.4, 129.5, 135.3, 150.0, 153.3, 164.7, 164.8; EI-MS *m/z* 282 (M⁺, 1.8), 188 (20.7), 104 (41.1), 94 (100). Anal. Calcd for C₁₅H₁₀N₂O₄: C, 63.83; H, 3.57; N, 9.92. Found: C, 63.64; H, 3.72; N, 9.95.

(1,3-Dioxo-1,3-dihydroisoindol-2-yl)carbamic acid *p*-tolyl ester (1f). White crystals; mp 142-144 °C (EtOAc); IR (KBr) 3210, 1805, 1750, 1730, 1195, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆, rotamers) • 2.31 (s, 3H), 6.90 (d, *J* = 8.2 Hz, 0.3H), 7.09 (d, *J* = 8.4 Hz, 1.7H), 7.17 (d, *J* = 8.2 Hz, 0.3H), 7.23 (d, *J* = 8.4 Hz, 1.7H), 7.90-8.08 (m, 4H), 10.84 (br s, 1H); ¹³C NMR (67.8 MHz, DMSO-*d*₆, rotamers) • 20.4, 120.9, 121.0, 123.7, 129.0, 129.7, 129.8, 135.0, 135.3, 147.8, 153.4, 164.8; EI-MS *m/z* 296 (M⁺, 3.0), 188 (19.7), 108 (100), 107 (38.9), 104 (26.4). Anal. Calcd for C₁₆H₁₂N₂O₄: C, 64.86; H, 4.08; N, 9.45. Found: C, 64.81; H, 3.98; N, 9.45.

(1,3-Dioxo-1,3-dihydroisoindol-2-yl)carbamic acid 4-chlorophenyl ester (1g). White crystals; mp 163-166 °C (EtOH); IR (KBr) 3300, 1805, 1775, 1740, 1230, 885, 705 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆, rotamers) • 7.08 (d, *J* = 8.8 Hz, 0.3H), 7.28 (d, *J* = 8.8 Hz, 1.7H), 7.40-7.57 (m, 2H),

7.90-8.07 (m, 4H), 10.99 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6 , rotamers) • 123.1, 123.3, 123.8, 129.0, 129.4, 129.9, 135.3, 148.7, 153.0, 164.7; EI-MS m/z 318 ($\text{M}^+ + 2$, 0.4) 316 (M^+ , 1.1), 188 (19.4), 128 (100), 104 (35.6); FAB-MS (3-nitrobenzyl alcohol) m/z 317 ($\text{M} + \text{H}$). Anal. Calcd for $\text{C}_{15}\text{H}_9\text{ClN}_2\text{O}_4$: C, 56.89; H, 2.86; N, 8.85. Found: C, 56.72; H, 2.89; N, 8.65.

(1,3-Dioxo-1,3-dihydroisoindol-2-yl)carbamic acid 2-chlorophenyl ester (1h). White crystals; mp 186-189 °C (isopropyl alcohol); IR (KBr) 3260, 1800, 1750, 1685, 1210, 710 cm^{-1} ; ^1H NMR (270 MHz, DMSO- d_6 , rotamers) δ 7.22 (d, $J = 8.0$ Hz, 0.2H), 7.25-7.45 (m, 2.7H), 7.53 (d, $J = 8.0$ Hz, 0.2H), 7.61 (d, $J = 8.0$ Hz, 0.9H) 7.90-8.05 (m, 4H), 11.12 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) δ 123.8, 124.0, 126.0, 127.6, 128.4, 129.0, 130.1, 135.3, 145.9, 152.3, 164.6; EI-MS m/z 318 ($\text{M}^+ + 2$, 0.26), 316 (M^+ , 0.6), 188 (29.1), 128 (100), 104 (40.0); FAB-MS (3-nitrobenzyl alcohol) m/z 317 ($\text{M}^+ + \text{H}$). Anal. Calcd for $\text{C}_{15}\text{H}_9\text{ClN}_2\text{O}_4$: C, 56.89; H, 2.86; N, 8.85. Found: C, 56.73; H, 2.86; N, 8.85.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-2-phenoxyacetamide (1i).** White crystals; mp 213-216 °C (acetone); IR (KBr) 3360, 1800, 1725, 1220 cm^{-1} ; ^1H NMR (270 MHz, DMSO- d_6) • 4.83 (s, 2H), 6.99-7.09 (m, 3H), 7.29-7.40 (m, 2H), 7.91-8.04 (m, 4H), 11.0 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 65.9, 114.8, 121.5, 123.8, 129.4, 129.5, 135.4, 157.5, 164.9, 167.5; EI-MS m/z 296 (M^+ , 21.9), 175 (100), 108 (100), 130 (23.3), 77 (22.9). Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_4$: C, 64.86; H, 4.08; N, 9.46. Found: C, 64.80; H, 4.07; N, 9.45.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-(4-methoxyphenyl)propionamide (1j).** White crystals: mp 142-146 °C (EtOAc/hexane); IR (KBr) 3280, 1810, 1755, 1685, 1250, 710 cm^{-1} ; ^1H NMR (270 MHz) δ 2.69 (t, $J = 7.6$ Hz, 2H), 3.01 (t, $J = 7.6$ Hz, 2H), 3.80 (s, 3H), 6.87 (d, $J = 8.5$ Hz, 2H), 7.19 (d, $J = 8.5$ Hz, 2H), 7.28 (br s, 1H), 7.77-7.84 (m, 2H), 7.87-7.92 (m, 2H); ^{13}C NMR (67.8 MHz) δ 30.2, 35.9, 55.3, 113.9, 123.9, 129.3, 129.8, 132.1, 134.6, 158.0, 165.0, 170.9; EI-MS m/z 324 (M^+ , 23.6), 162 (24.4), 134 (52.2), 121 (100). Anal. Calcd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_4$: C, 66.66; H, 4.97; N, 8.64. Found: C, 66.69; H, 5.00; N, 8.62.

3-(4-Chlorophenyl)-*N*-(1,3-dioxo-1,3-dihydroisoindol-2-yl)propionamide (1k). White crystals: mp 192-195 °C (EtOAc); IR (KBr) 3280, 1800, 1750, 1680, 705 cm^{-1} ; ^1H NMR (270 MHz, DMSO- d_6) • 2.64 (t, $J = 7.5$ Hz, 2H), 2.89 (t, $J = 7.5$ Hz, 2H), 7.27-7.37 (m, 4H), 7.88-7.99 (m, 4H), 10.72 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 29.8, 34.3, 123.6, 128.1, 129.3, 130.1, 130.6, 135.1, 139.5, 165.0, 170.6; EI-MS m/z 330 ($\text{M}^+ + 2$, 3.0), 328 (M^+ , 8.1), 162 (100), 139 (12.0), 125 (39.0),

104 (12.3). Anal. Calcd for $C_{17}H_{13}ClN_2O_3$: C, 62.11; H, 3.99; N, 8.52. Found: C, 62.20; H, 3.80; N, 8.51.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-(4-fluorophenyl)propionamide (1l).** White crystals: mp 171-174 °C (EtOAc/*n*-hexane); IR (KBr) 3250, 1795, 1740, 1675, 705 cm^{-1} ; 1H NMR (270 MHz, DMSO- d_6) • 2.64 (t, $J = 7.6$ Hz, 2H), 2.90 (t, $J = 7.6$ Hz, 2H), 7.11 (t, $J_{H-F} = J_{H-H} = 8.9$ Hz, 2H), 7.31 (dd, $J_{H-H} = 8.7$ Hz, $J_{H-F} = 5.6$ Hz, 2H), 7.89-8.01 (m, 4H), 10.70 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 29.7, 34.6, 114.8 (d, $J_{C-F} = 20.6$ Hz), 123.5, 129.3, 130.0 (d, $J_{C-F} = 7.8$ Hz), 135.1, 136.5 (d, $J_{C-F} = 3.3$ Hz), 160.6 (d, $J_{C-F} = 240.4$ Hz), 164.9, 170.6; EI-MS m/z 312 (M^+ , 11.8), 162 (100), 123 (13.7), 109 (43.6), 104 (9.3). Anal. Calcd for $C_{17}H_{13}FN_2O_3$: C, 65.38; H, 4.20; N, 8.97. Found: C, 65.25; H, 4.13; N, 8.94.

3-(2,4-Difluorophenyl)-*N*-(1,3-dioxo-1,3-dihydroisoindol-2-yl)propionamide (1m). White crystals: mp 179-181.5 °C (toluene); IR (KBr) 3190, 1795, 1740, 1680, 705 cm^{-1} ; 1H NMR (270 MHz, DMSO- d_6) • 2.65 (t, $J = 7.6$ Hz, 2H), 2.91 (t, $J = 7.6$ Hz, 2H), 7.04 (td, $J_{H-H} = 8.5$, $J_{H-F} = 2.6$ Hz, 1H), 7.20 (td, $J_{H-F} = 10.8$, $J_{H-H} = 2.3$ Hz, 1H), 7.41 (dd, $J_{H-F} = 15.5$, $J_{H-H} = 8.5$ Hz, 1H), 7.88-8.03 (m, 4H), 10.74 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 23.3, (d, $J_{C-F} = 2.2$ Hz), 32.9, 103.5 (t, $J_{C-F} = 25.9$ Hz), 111.2 (dd, $J_{C-F} = 20.9$, 3.6 Hz), 123.2 (dd, $J_{C-F} = 15.6$, 3.3 Hz), 123.6, 129.3, 131.5 (dd, $J_{C-F} = 6.4$, 3.1 Hz), 135.1, 160.1 (dd, $J_{C-F} = 245.4$, 12.3 Hz), 160.8 (dd, $J_{C-F} = 243.2$, 12.3 Hz), 164.9, 170.3; EI-MS m/z 330 (M^+ , 5.5), 310 (9.2), 162 (100), 141 (20.2), 127 (49.7). Anal. Calcd for $C_{17}H_{12}F_2N_2O_3$: C, 61.82; H, 3.66; N, 8.48. Found: C, 61.99; H, 3.43; N, 8.35.

3-(3,4-Difluorophenyl)-*N*-(1,3-dioxo-1,3-dihydroisoindol-2-yl)propionamide (1n). White crystals: mp 193-196 °C (EtOH); IR (KBr) 3250, 1790, 1740, 1680, 710 cm^{-1} ; 1H NMR (270 MHz, DMSO- d_6) • 2.66 (t, $J = 7.4$ Hz, 2H), 2.91 (t, $J = 7.4$ Hz, 2H), 7.08-7.18 (m, 1H), 7.28-7.43 (m, 2H), 7.90-8.02 (m, 4H), 10.72 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 29.5, 34.1, 117.0 (d, $J_{C-F} = 16.8$ Hz), 117.1 (d, $J_{C-F} = 16.8$ Hz), 123.6, 125.0 (dd, $J_{C-F} = 6.7$, 3.3 Hz), 129.3, 135.1, 138.2 (dd, $J_{C-F} = 6.1$, 3.9 Hz), 147.8 (dd, $J_{C-F} = 241.1$, 12.3 Hz), 149.0 (dd, $J_{C-F} = 243.7$, 12.8 Hz), 164.9, 170.4; EI-MS m/z 330 (M^+ , 11.6), 162 (100), 141 (16.0), 127 (30.5). Anal. Calcd for $C_{17}H_{12}F_2N_2O_3$: C, 61.82; H, 3.66; N, 8.48. Found: C, 61.82; H, 3.62; N, 8.47.

***N*-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-2-(4-fluorobenzyl)benzamide (1o).** White crystals: mp 197-200 °C (EtOAc); IR (KBr) 3230, 1805, 1750, 1660, 705 cm^{-1} ; 1H NMR (270 MHz, DMSO- d_6) • 4.16 (s, 2H), 7.09 (t, $J_{H-F} = J_{H-H} = 9.0$ Hz, 2H), 7.30-7.44 (m, 4H), 7.49 (t, $J = 7.5$ Hz, 1H), 7.58 (d, $J = 7.4$ Hz, 1H), 7.93-8.10 (m, 4H), 11.29 (br s, 1H); ^{13}C NMR (67.8 MHz, DMSO- d_6) • 36.7, 114.9 (d,

$J_{\text{C-F}} = 20.6$ Hz), 123.7, 126.1, 127.7, 129.3, 130.5 (d, $J_{\text{C-F}} = 7.8$ Hz), 130.8, 132.4, 135.2, 136.9 (d, $J_{\text{C-F}} = 2.4$ Hz), 139.7, 160.5 (d, $J_{\text{C-F}} = 240.4$ Hz), 165.0, 167.9; EI-MS m/z 374 (M^+ , 3.1), 213 (100), 212 (61.4), 183 (17.7). Anal. Calcd for $\text{C}_{22}\text{H}_{15}\text{FN}_2\text{O}_3$: C, 70.58; H, 4.04; N, 7.48. Found: C, 70.45; H, 3.99; N, 7.38.

2-(4-Chlorophenoxy)-N-(1,3-dioxo-1,3-dihydroisoindol-2-yl)acetamide (1p). White crystals: mp 226-228 °C; IR (KBr) 3370, 1795, 1735, 1225 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 4.87 (s, 2H), 7.17 (d, *J* = 8.8 Hz, 2H), 7.39 (d, *J* = 8.8 Hz, 2H), 7.68-8.05 (m, 4H), 11.03 (br s, 1H); ¹³C NMR (67.8 MHz) δ 66.1, 116.6, 123.8, 125.2, 129.2, 129.3, 135.3, 156.2, 164.8, 167.9; EI-MS *m/z* 332 (*M*⁺ + 2, 9.7), 330 (*M*⁺, 28.9), 175 (100), 130 (24.0). Anal. Calcd for C₁₆H₁₁ClN₂O₄: C, 58.11; H, 3.35; N, 8.47. Found: C, 58.01; H, 3.43; N, 8.46.

N-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-*o*-tolylpropionamide (1q). White crystals: mp 173-175 °C (EtOAc/*n*-hexane); IR (KBr) 3270, 1800, 1745, 1690, 710 cm⁻¹; ¹H NMR (270 MHz) δ 2.35 (s, 3H), 2.68 (t, *J* = 7.9 Hz, 2H), 3.06 (t, *J* = 7.9 Hz, 2H), 7.13-7.23 (m, 4H), 7.40 (br s, 1H), 7.75-7.83 (m, 2H), 7.87-7.95 (m, 2H); ¹³C NMR (67.8 MHz) δ 19.4, 24.3, 34.3, 123.9, 126.2, 126.5, 128.6, 129.9, 130.3, 134.6, 135.9, 138.1, 165.0, 170.8; EI-MS *m/z* 308 (*M*⁺, 0.3), 162 (100), 105 (54.8). Anal. Calcd for C₁₈H₁₆N₂O₃: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.08; H, 5.25; N, 9.04.

N-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-*p*-tolylpropionamide (1r). White crystals: mp 173-176 °C (EtOAc/*n*-hexane); IR (KBr) 3290, 2930, 1805, 1755, 1685, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 2.27 (s, 3H), 2.60 (t, *J* = 7.6 Hz, 2H), 2.85 (t, *J* = 7.6 Hz, 2H), 7.13 (q, *J* = 8.0 Hz, 4H), 7.91-8.00 (m, 4H), 10.69 (br s, 1H); ¹³C NMR (67.8 MHz, CDCl₃) δ 21.1, 30.6, 35.8, 123.9, 128.2, 129.2, 129.9, 134.9, 135.8, 136.9, 165.0, 170.8; EI-MS *m/z* 308 (*M*⁺, 23.4), 162 (38.1), 118 (72.3), 105 (100). Anal. Calcd for C₁₈H₁₆N₂O₃: C, 70.12; H, 5.23; N, 9.09. Found: C, 70.00; H, 5.23; N, 9.07.

3-(2-Chlorophenyl)-N-(1,3-dioxo-1,3-dihydroisoindol-2-yl)propionamide (1s). White crystals: mp 187-189 °C (EtOAc); IR (KBr) 3260, 1800, 1750, 1685, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 2.67 (t, *J* = 7.6 Hz, 2H), 3.01 (t, *J* = 7.6 Hz, 2H), 7.22-7.36 (m, 2H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.89-8.04 (m, 4H), 10.76 (br s, 1H); ¹³C NMR (67.8 MHz, DMSO-*d*₆) δ 28.3, 32.5, 123.6, 127.2, 128.0, 129.0, 129.3, 130.5, 132.7, 135.1, 137.6, 164.9, 170.3; EI-MS *m/z* 330 (*M*⁺ + 2, 0.24), 328 (*M*⁺, 0.6), 162 (100), 139 (43.6), 125 (39.8), 103 (20.6). Anal. Calcd for C₁₇H₁₃ClN₂O₃: C, 62.11; H, 3.99; N, 8.52. Found: C, 62.11; H, 3.79; N, 8.51.

N-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-(2-fluorophenyl)propionamide (1t). White crystals: mp 172-175 °C (EtOAc); IR (KBr) 3270, 1800, 1740, 1690, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 2.66 (t, *J* = 7.7 Hz, 2H), 2.93 (t, *J* = 7.7 Hz, 2H), 7.11-7.22 (m, 2H), 7.23-7.32 (m, 1H), 7.32-7.42 (m, 1H), 7.90-8.01 (m, 4H), 10.75 (br s, 1H); ¹³C NMR (67.8 MHz, DMSO-*d*₆) δ 23.7 (d, *J*_{C-F} = 2.8 Hz), 32.9, 114.9 (d, *J*_{C-F} = 21.8 Hz), 123.6, 124.2 (d, *J*_{C-F} = 3.4 Hz), 126.9 (d, *J*_{C-F} = 15.1 Hz), 128.1

(d, $J_{\text{C-F}} = 7.8$ Hz), 129.3, 130.6 (d, $J_{\text{C-F}} = 5.0$ Hz), 135.1, 160.2 (d, $J_{\text{C-F}} = 242.1$ Hz), 164.9, 170.3; EI-MS m/z 312 (M^+ , 9.9), 292 (9.8), 162 (100), 123 (37.0), 109 (39.6). Anal. Calcd for $\text{C}_{17}\text{H}_{13}\text{FN}_2\text{O}_3$: C, 65.38; H, 4.20; N, 8.97. Found: C, 65.26; H, 4.22; N, 8.85.

A Typical Experimental Procedure for Table 1 (Entry 3): PIFA (146 mg, 0.34 mmol) was added to a solution of **1b** (100 mg, 0.340 mmol) in HFIP (5 mL). The mixture was stirred at room temperature for 17 h under an argon atmosphere. After the reaction, the solvent was evaporated and 10% Na_2CO_3 aq. (20 mL) was added to the residue at 0 °C. The mixture was extracted with EtOAc (25 mL x 2). The organic layer was washed with brine, dried over Na_2SO_4 , and concentrated. The crude products were purified by column chromatography, eluting with EtOAc/toluene (1:10), to afford **2b** (73 mg, 73%).

2-(2-Oxo-2,3-dihydroindol-1-yl)isoindole-1,3-dione (2a). White crystals: mp 282-283 °C ($\text{CH}_2\text{Cl}_2/n$ -hexane); IR (KBr) 1805, 1740, 710 cm^{-1} ; ^1H NMR (270 MHz, $\text{DMSO}-d_6$) δ 3.97 (s, 2H), 7.12-7.32 (m, 3H), 7.40 (d, $J = 7.3$ Hz, 1H), 7.98-8.14 (m, 4H); ^{13}C NMR (67.8 MHz) δ 34.1, 107.7, 121.9, 123.9, 124.4, 125.2, 128.2, 130.0, 135.1, 142.0, 163.7, 171.7; EI-MS m/z 278 (M^+ , 100), 249 (20.8), 221 (13.6), 132 (15.1), 104 (31.5), 76 (13.5). Anal. Calcd for $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_3$: C, 69.06; H, 3.62; N, 10.07. Found: C, 68.78; H, 3.53; N, 9.96.

2-(2-Oxo-3,4-dihydro-2H-quinolin-1-yl)isoindole-1,3-dione (2b). White crystals: mp 261-263 °C (EtOAc); IR (KBr) 1795, 1735, 1705, 710 cm^{-1} ; ^1H NMR (270 MHz, $\text{DMSO}-d_6$) δ 2.87 (t, $J = 7.4$ Hz, 2H), 3.08 (t, $J = 7.4$ Hz, 2H), 7.08-7.28 (m, 3H), 7.36 (d, $J = 7.6$ Hz, 1H), 7.98-8.09 (m, 4H); ^{13}C NMR (67.8 MHz) δ 24.9, 31.2, 113.2, 124.2, 124.9, 127.7, 128.1, 130.0, 134.9, 138.9, 164.6, 167.1; EI-MS m/z 292 (M^+ , 100), 145 (19.3), 117 (37.7), 104 (13.1), 76 (9.8). Anal. Calcd for $\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}_3$: C, 69.86; H, 4.14; N, 9.58. Found: C, 69.92; H, 4.11; N, 9.86.

2-(2-Oxo-2,3,4,5-tetrahydrobenzo[*b*]azepin-1-yl)isoindole-1,3-dione (2c). White crystals: mp 246-250 °C (benzene/*n*-hexane); IR (KBr) 1800, 1745, 1695, 715 cm^{-1} ; ^1H NMR (270 MHz) δ 2.27 (quint, $J = 7.2$ Hz, 2H), 2.53 (t, $J = 7.2$ Hz, 2H), 3.20 (t, $J = 7.2$ Hz, 2H), 7.22-7.32 (m, 4H), 7.78-7.84 (m, 2H), 7.89-7.97 (m, 2H); ^{13}C NMR (67.8 MHz) δ 28.1, 29.7, 31.9, 122.8, 124.1, 127.7, 127.8, 128.3, 129.5, 130.0, 134.8, 135.3, 140.4, 165.3, 170.4; EI-MS m/z 306 (M^+ , 72.3), 278 (28.8), 233 (100), 159 (29.6), 132 (48.2). Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_3$: C, 70.58; H, 4.61; N, 9.15. Found: C, 70.80; H, 4.62; N, 9.07.

2-(6-Oxo-6H-phenanthridin-5-yl)isoindole-1,3-dione (2d). White crystals: mp 244-246 °C (EtOAc/*n*-hexane); IR (KBr) 1795, 1745, 1665, 715 cm⁻¹; ¹H NMR (500 MHz) δ 7.25 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.38 (t, *J* = 8.1 Hz, 1H), 7.48 (t, *J* = 8.1 Hz, 1H), 7.62 (t, *J* = 8.1 Hz, 1H), 7.84 (td, *J* = 8.1, 1.6 Hz, 1H), 7.87-7.94 (m, 2H), 8.01-8.08 (m, 2H), 8.31-8.37 (m, 2H), 8.49 (dd, *J* = 8.1, 1.5 Hz, 1H); ¹³C NMR (125.7 MHz) δ 113.3, 119.2, 122.2, 123.5, 123.9, 124.4, 124.7, 128.3, 129.3, 130.0, 130.1, 133.6, 134.1, 135.0, 137.3, 158.5, 164.6; EI-MS *m/z* 340 (M⁺, 100), 296 (92.9), 166 (31.9). Anal. Calcd for C₂₁H₁₂N₂O₃: C, 74.11; H, 3.55; N, 8.23. Found: C, 74.10; H, 3.37; N, 8.28.

Removal of Phthaloyl Group from *N*-Phthalimido-3,4-dihydrocarbostyryl (2b). To **2b** (50 mg, 0.171 mmol) in ethanol (5 mL) was added NH₂NH₂·H₂O (80%) (0.025 mL, 0.428 mmol) at room temperature. After refluxing the reaction mixture for 1 h, ethanol was removed under reduced pressure. To the residue benzene (5 mL) was added and insoluble material was removed by filtration. After concentration of the filtrate, the crude product was purified by column chromatography, eluting EtOAc/CHCl₃ (1:10), to afford *N*-amino-3,4-dihydrocarbostyryl (26 mg, 95%): colorless crystals; mp 141-142 °C (EtOAc/*n*-hexane) (lit.²¹ mp 142-143 °C).

Removal of Amino Group from *N*-Amino-3,4-dihydrocarbostyryl. *N*-Amino-3,4-dihydrocarbostyryl (50 mg, 0.308 mmol) and Raney nickel (150 mg, W-2) were stirred in ethanol (5 mL) under reflux for 30 min. After being cooled, the nickel was removed by filtration and washed with ethanol (5 mL x 2). The combined filtrate was concentrated and the residue was purified by column chromatography, eluting EtOAc/*n*-hexane (1:1), to afford 3,4-dihydrocarbostyryl (32 mg, 91%): colorless crystals; mp 166-167 °C (benzene/*n*-hexane) (lit.²² mp 165-165.5 °C).

A Typical Experimental Procedure for Table 2 (Entry 3): 4-Chlorophenyl iodine(III) bis(trifluoroacetate) (*p*-ClPIFA) (215 mg, 0.462 mmol) was added to a solution of **1e** (100 mg, 0.355 mmol) in TFA (10 mL) at reflux and the reaction mixture was refluxed for 0.5 h under an argon atmosphere. After the reaction, the solvent was evaporated and 10% Na₂CO₃ aq. (20 mL) was added to the residue at 0 °C. The mixture was extracted with EtOAc (25 mL x 2). The organic layer was washed with brine, dried over Na₂SO₄, and concentrated. The crude products were purified by column chromatography, eluting with acetone/toluene (1:40), to afford **2e** (74 mg, 74%).

2-(2-Oxo-benzoxazol-3-yl)isoindole-1,3-dione (2e). White crystals; mp 225-227 °C (toluene); IR (KBr) 1825, 1790, 1750, 715 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 7.26-7.38 (m, 2H), 7.52-7.66 (m, 2H), 8.00-8.20 (m, 4H); ¹³C NMR (67.8 MHz, DMSO-*d*₆) δ 109.7, 110.9, 124.3, 124.4, 124.9, 129.0, 129.1, 135.7, 140.4, 150.5, 162.9; EI-MS *m/z* 280 (M⁺, 100), 236 (4.5), 208 (4.3), 180 (7.0),

152 (7.4), 134 (4.9), 130(5.9). Anal. Calcd for $C_{15}H_8N_2O_4$: C, 64.29; H, 2.88; N, 10.00. Found: C, 64.52; H, 3.00; N, 9.73.

***N*-(1,3-Dioxo-1,3-dihydroisoindole-2-yl)-*N*-(4-iodophenyl)carbamic acid phenyl ester (3e).**

White crystals; mp 146-149 °C (EtOAc); IR (KBr) 1800, 1755, 1740, 710 cm⁻¹; ¹H NMR (270 MHz) • 7.05-7.29 (m, 3H), 7.29-7.55 (m, 4H), 7.65-8.03 (m, 6H); ¹³C NMR (67.8 MHz) • 120.9, 121.3, 124.1, 126.0, 127.1, 129.2, 129.5, 134.8, 138.2, 139.3, 150.2, 151.4, 164.3; EI-MS *m/z* 484 (M⁺, 100), 391 (85.2), 363 (16.2), 264 (58.0), 245 (17.4), 203 (12.1), 104 (11.8). Anal. Calcd for C₂₁H₁₃IN₂O₄: C, 52.09; H, 2.71; N, 5.78. Found: C, 51.90; H, 2.65; N, 5.83.

2-(5-Methyl-2-oxobenzoxazol-3-yl)isoindole-1,3-dione (2f). White crystals; mp 197.5-200 °C (toluene); IR (KBr) 1815, 1785, 1745, 705 cm⁻¹; ¹H NMR (270 MHz) • 2.35 (s, 3H), 6.73 (s, 1H), 7.00 (d, *J* = 8.2 Hz, 1H), 7.18 (d, *J* = 8.2 Hz, 1H), 7.85-7.95 (m, 2H), 7.98-8.08 (m, 2H); ¹³C NMR (67.8 MHz) • 21.5, 108.4, 110.7, 124.4, 124.6, 129.3, 129.6, 134.8, 135.3, 139.4, 151.2, 162.9; EI-MS *m/z* 294 (M⁺, 100), 252 (12.1), 250 (3.8), 148 (22.6), 104 (18.4). Anal. Calcd for C₁₆H₁₀N₂O₄: C, 65.31; H, 3.43; N, 9.52. Found: C, 65.17; H, 3.41; N, 9.48.

2-(5-Chloro-2-oxobenzoxazol-3-yl)isoindole-1,3-dione (2g). White crystals; mp 234-237 °C (toluene); IR (KBr) 1820, 1780, 1750, 705 cm⁻¹; ¹H NMR (270 MHz) • 6.95 (s, 1H), 7.16-7.29 (m, 2H), 7.87-7.95 (m, 2H), 7.98-8.07 (m, 4H); ¹³C NMR (67.8 MHz) • 108.7, 112.0, 124.1, 124.69, 129.4, 130.3, 130.3, 135.5, 139.8, 150.4, 162.6; EI-MS *m/z* 316 (M⁺+2, 33.4), 314 (M⁺, 100), 270 (2.9), 230 (4.0), 214 (3.1), 186 (3.7), 168 (19.4). Anal. Calcd for C₁₅H₇N₂O₄Cl: C, 57.25; H, 2.24; N, 8.90. Found: C, 57.26; H, 2.22; N, 8.86.

2-(7-Chloro-2-oxobenzoxazol-3-yl)isoindole-1,3-dione (2h). White crystals; mp 243-246 °C (toluene); IR (KBr) 1825, 1790, 1750, 705 cm⁻¹; ¹H NMR (270 MHz) • 6.83 (d, *J* = 7.9 Hz, 1H), 7.15 (t, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 7.9 Hz, 1H), 7.77-8.02 (m, 4H); ¹³C NMR (125.7 MHz) • 106.4, 116.9, 124.8, 124.9, 125.3, 129.6, 130.7, 135.6, 138.1, 150.1, 162.8; EI-MS *m/z* 316 (M⁺+2, 33.9), 314 (M⁺, 100), 279 (3.2), 230 (5.5), 168 (13.5). Anal. Calcd for C₁₅H₇N₂O₄Cl: C, 57.25; H, 2.24; N, 8.90. Found: C, 57.01; H, 2.33; N, 8.68.

2-(3-Oxo-2,3-dihydrobenzo[1,4]oxazin-4-yl)isoindole-1,3-dione (2i). White crystals: mp 216-217 °C (EtOAc/*n*-hexane); IR (KBr) 1800, 1750, 1725, 755, 710 cm⁻¹; ¹H NMR (270 MHz) δ 5.06 (s, 2H), 6.97-7.07 (m, 1H), 7.08-7.21 (m, 2H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.93-8.17 (m, 4H); ¹³C NMR (67.8 MHz) δ 67.9, 113.4, 117.4, 123.1, 124.5, 125.3, 128.3, 129.9, 135.2, 144.4, 162.1, 163.9; EI-MS *m/z* 294 (M⁺, 100), 148 (5.3), 119 (40.9). Anal. Calcd for C₁₆H₁₀N₂O₄: C, 65.31; H, 3.43; N, 9.52. Found: C, 65.17; H, 3.38; N, 9.45.

A Typical Experimental Procedure for Table 3 (Entry 5): HTIB (238 mg, 0.606 mmol) was added to a solution of **1m** (100 mg, 0.303 mmol) in TFEA (10 mL). The reaction mixture was stirred at room temperature for 0.5 h under an argon atmosphere. After the reaction, the mixture was evaporated and 10% Na₂CO₃ aq. (20 mL) was added to the residue at 0 °C. The mixture was extracted with EtOAc (25 mL x 2) and the organic layer was washed with brine, dried over Na₂SO₄, and concentrated. The crude products were purified by column chromatography, eluting with EtOAc/toluene (1:3), to afford **4m** (92 mg, 93%).

1-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (4j). White crystals: mp 255-258 °C (EtOAc/*n*-hexane); IR (KBr) 1805, 1750, 1730, 1675, 715 cm⁻¹; ¹H NMR (270 MHz) δ 2.46 (t, *J* = 8.0 Hz, 2H), 2.80 (t, *J* = 8.0 Hz, 2H), 6.27 (dt, *J* = 10.1, 2.7 Hz, 2H), 7.12 (dt, *J* = 10.1, 2.7 Hz, 2H), 7.78-7.84 (m, 2H), 7.85-7.91 (m, 2H); ¹³C NMR (67.8 MHz) δ 21.6, 24.0, 57.5, 119.0, 124.5, 125.2, 129.9, 141.7, 159.4, 166.0, 179.1; EI-MS *m/z* 308 (M⁺, 100), 266 (27.9), 104 (30.4). Anal. Calcd for C₁₇H₁₂N₂O₄: C, 66.23; H, 3.92; N, 9.09. Found: C, 66.00; H, 4.10; N, 9.15.

1-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-6-fluoro-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (4m). White crystals: mp 214-217 °C (EtOAc); IR (KBr) 1805, 1750, 1680, 1655, 710 cm⁻¹; ¹H NMR (270 MHz) δ 2.46-2.82 (m, 3H), 2.88-3.04 (m, 1H), 6.00 (dd, *J*_{H-F} = 12.9, *J*_{H-H} = 1.6 Hz, 1H), 6.24 (dt, *J*_{H-H} = 10.1, *J*_{H-F} = *J*_{H-H} = 1.6 Hz, 1H), 6.90 (t, *J*_{H-F} = *J*_{H-H} = 10.1 Hz, 1H), 7.75-7.95 (m, 4H); ¹³C NMR (67.8 MHz) δ 26.9 (d, *J*_{C-F} = 2.2 Hz), 27.6, 62.4 (d, *J*_{C-F} = 22.3 Hz), 110.8 (d, *J*_{C-F} = 9.5 Hz), 124.1, 124.3, 129.4, 129.5, 130.4 (d, *J*_{C-F} = 1.7 Hz), 134.9, 135.1, 142.7 (d, *J*_{C-F} = 4.5 Hz), 163.9, 164.7, 170.9, 172.2 (d, *J*_{C-F} = 290.0 Hz), 185.8 (d, *J*_{C-F} = 15.6 Hz); EI-MS *m/z* 326 (M⁺, 100), 272 (29.4), 165 (12.9), 138 (29.7), 104 (29.7); Anal. Calcd for C₁₇H₁₁FN₂O₄: C, 62.58; H, 3.40; N, 8.59. Found: C, 62.42; H, 3.47; N, 8.46.

1-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-7-fluoro-1-azaspiro[4.5]deca-6,9-diene-2,8-dione (4n). White crystals: mp 268-270 °C (EtOAc); IR (KBr) 1800, 1740, 1690, 1660, 705 cm⁻¹; ¹H NMR (270 MHz) δ 2.54 (t, *J* = 7.8 Hz, 2H), 2.81 (t, *J* = 7.8 Hz, 2H), 6.28 (dd, *J*_{H-H} = 9.9, *J*_{H-F} = 7.1 Hz, 1H), 6.67 (dd, *J*_{H-F} = 14.7, *J*_{H-H} = 2.9 Hz, 1H), 7.15 (dd, *J*_{H-H} = 9.9, *J*_{H-F} = 2.9 Hz, 1H), 7.78-7.98 (m, 4H); ¹³C NMR (67.8 MHz) δ 26.7, 29.8, 64.5 (d, *J*_{C-F} = 8.4 Hz), 122.8 (d, *J*_{C-F} = 13.9 Hz), 124.2, 124.3, 128.9, 129.0, 129.5, 135.0, 135.0, 147.6 (d, *J*_{C-F} = 2.8 Hz), 153.8 (d, *J*_{C-F} = 269.4 Hz), 164.4, 164.5, 170.5, 177.1 (d, *J*_{C-F} = 22.3 Hz); EI-MS *m/z* 326 (M⁺, 100), 284 (22.0), 165 (17.3), 138 (22.4), 104 (35.4); Anal. Calcd for C₁₇H₁₁FN₂O₄: C, 62.58; H, 3.40; N, 8.59. Found: C, 62.40; H, 3.47; N, 8.61.

1-Oxo-2-phthalimido-3,4-dihydro-2H-isoquinoline-3-spiro-1'-(4'-oxocyclohexa-2',5'-diene)

(4o). White crystals: mp 227-229 °C (EtOAc/*n*-hexane); IR (KBr) 1800, 1745, 1680, 720, 710 cm⁻¹; ¹H NMR (270 MHz) δ 3.51 (s, 2H), 6.24 (d, *J* = 10.5 Hz, 2H), 7.18 (d, *J* = 10.5 Hz, 2H), 7.27 (d, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.60 (dt, *J* = 7.3, 1.4 Hz, 1H), 7.77-7.85 (m, 2H), 7.87-7.94 (m, 2H), 8.15 (d, *J* = 7.3 Hz, 1H); ¹³C NMR (67.8 MHz) δ 39.7, 62.9, 124.2, 126.6, 127.8, 128.1, 129.4, 129.8, 130.0, 134.0, 134.7, 135.0, 146.7, 161.7, 165.5, 184.0; EI-MS *m/z* 370 (M⁺, 37.5), 181 (23.5), 118 (100). Anal. Calcd for C₂₂H₁₄N₂O₄: C, 71.35; H, 3.81; N, 7.56. Found: C, 71.31; H, 3.68; N, 7.48.

3'-(4-Fluorophenyl)-3'H-[2,2']biisoindolyl-1,3,1'-trione (5). White crystals: mp 199-201 °C (EtOAc); IR (KBr) 1800, 1735, 1720, 710 cm⁻¹; ¹H NMR (270 MHz, DMSO-*d*₆) δ 6.15 (s, 1H), 7.18 (t, *J*_{H-F} = *J*_{H-H} = 8.7 Hz, 2H), 7.35 (d, *J* = 7.7 Hz, 1H), 7.45 (dd, *J*_{H-H} = 8.7 Hz, *J*_{H-F} = 5.4 Hz, 2H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.77 (t, *J* = 7.7 Hz, 1H), 7.90-8.07 (m, 5H); ¹³C NMR (67.8 MHz, DMSO-*d*₆) δ 64.0, 115.2 (d, *J*_{C-F} = 21.8 Hz), 123.6, 123.8, 123.9, 125.0, 126.9, 127.9, 128.6, 128.6, 128.7, 128.9, 130.1 (d, *J*_{C-F} = 8.4 Hz), 131.4 (d, *J*_{C-F} = 3.4 Hz), 133.7, 135.4 (d, *J*_{C-F} = 5.6 Hz), 145.1, 161.9 (d, *J*_{C-F} = 244.4 Hz), 163.5, 164.7, 165.6; EI-MS *m/z* 372 (M⁺, 25.9), 226 (100), 225 (45.2), 183 (45.3). Anal. Calcd for C₂₂H₁₃FN₂O₃: C, 70.97; H, 3.52; N, 7.52. Found: C, 70.91; H, 3.48; N, 7.48.

4-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-1-oxa-4-azaspiro[4.5]deca-6,9-diene-3,8-dione (4p).

White crystals: mp 172-174 °C (EtOAc/*n*-hexane); IR (KBr) 1810, 1750, 1680, 1640 cm⁻¹; ¹H NMR (270 MHz) δ 4.66 (s, 2H), 6.27 (d, *J* = 11.8 Hz, 2H), 6.99 (d, *J* = 11.8 Hz, 2H), 7.77-8.01 (m, 4H); ¹³C NMR (67.8 MHz) δ 67.7, 86.7, 124.5, 129.6, 131.5, 135.3, 141.8, 163.8, 166.8, 183.9; EI-MS *m/z* 310 (M⁺, 100), 268 (15.4), 104 (56.7). Anal. Calcd for C₁₆H₁₀N₂O₅: C, 61.94; H, 3.25; N, 9.03. Found: C, 61.92; H, 3.30; N, 9.05.

A Typical Experimental Procedure for Table 4 (Entry 1): PIFA (585 mg, 1.36 mmol) was added to a solution of **1b** (300 mg, 1.36 mmol) in TFEA (20 mL). The reaction mixture was stirred at room temperature for 3 h under an argon atmosphere. After the reaction, the mixture was evaporated and 10% Na₂CO₃ aq. (20 mL) was added to the residue at 0 °C. The mixture was extracted with EtOAc (25 mL x 2) and the organic layer was washed with brine, dried over Na₂SO₄, and concentrated. The crude products were purified by column chromatography, eluting with EtOAc/CHCl₃ (1:10), to afford **2b** (66 mg, 17%) and **6b** (403 mg, 76%).

cis- and trans-2-[2-Oxo-8-(2,2,2-trifluoroethoxy)-1-azaspiro[4.5]deca-6,9-diene-1-yl]isoindole-1,3-dione (6b). White crystals: mp 175-178 °C (EtOAc/*n*-hexane); IR (KBr) 1805, 1750, 1730, 715

cm⁻¹; ¹H NMR (270 MHz) δ 2.24-2.38 (m, 2H), 2.71 (t, J = 9.2 Hz, 2H), 3.32 (q, $J_{\text{H-F}}$ = 8.2 Hz, 1H), 3.68 (q, $J_{\text{H-F}}$ = 8.2 Hz, 1H), 4.43-4.50 (m, 0.5H), 4.53-4.62 (m, 0.5H), 5.92-6.06 (m, 2H), 6.29 (dd, J = 10.1, 1.7 Hz, 2H), 7.76-7.82 (m, 2H), 7.84-7.96 (m, 2H); ¹³C NMR (67.8 MHz) δ 26.9, 27.0, 31.3, 31.9, 61.1, 61.3, 62.2 (q, $J_{\text{C-F}}$ = 34.2 Hz), 63.2 (q, $J_{\text{C-F}}$ = 34.2 Hz), 68.3, 68.7, 123.4 (q, $J_{\text{C-F}}$ = 274.9 Hz), 123.8 (q, $J_{\text{C-F}}$ = 274.9 Hz), 123.9, 124.0, 127.8, 128.2, 129.5, 129.8, 132.7, 133.3, 134.7, 134.8, 164.65, 164.7, 171.0, 171.2; EI-MS m/z 392 (M^+ , 0.5), 293 (4.7), 230 (18.8), 202 (91.6), 189 (100), 162 (33.8); FAB-MS (3-nitrobenzyl alcohol) m/z 393 (M^+ + H). Anal. Calcd for C₁₉H₁₅F₃N₂O₄: C, 58.17; H, 3.85; N, 7.14. Found: C, 58.25; H, 3.70; N, 7.01.

cis- and trans-2-[2-Oxo-8-(2,2,3,3-tetrafluoropropoxy)-1-azaspiro[4.5]deca-6,9-dien-1-yl]isoindole-1,3-dione (6b'). White crystals: mp 154-164 °C (benzene/*n*-hexane); IR (KBr) 1800, 1745, 1725, 710 cm⁻¹; ¹H NMR (270 MHz) δ 2.23-2.38 (m, 2H), 2.71 (t, J = 7.8 Hz, 2H), 3.29 (t, $J_{\text{H-F}}$ = 12.5 Hz, 0.4H), 3.67 (t, $J_{\text{H-F}}$ = 12.5 Hz, 1.6H), 4.43-4.47 (m, 0.8H), 4.54-4.57 (m, 0.2H), 5.62 (t, $J_{\text{H-F}}$ = 53.3 Hz, 0.2H), 5.90 (t, $J_{\text{H-F}}$ = 53.2 Hz, 0.8H), 5.86-6.05 (m, 2H), 6.29 (d, J = 10.1 Hz, 2H), 7.76-7.82 (m, 2H), 7.84-7.90 (m, 2H); ¹³C NMR (67.8 MHz) δ 26.8, 27.0, 31.2, 31.8, 61.2, 61.3, 61.6 ($J_{\text{C-F}}$ = 29.3 Hz), 62.5 (t, $J_{\text{C-F}}$ = 29.3 Hz), 68.0, 68.6, 108.8 (tt, $J_{\text{C-F}}$ = 249.1, 34.0 Hz), 109.1 (tt, $J_{\text{C-F}}$ = 249.1, 34.0 Hz), 114.6 (tt, $J_{\text{C-F}}$ = 249.1, 29.2 Hz), 115.0 (tt, $J_{\text{C-F}}$ = 249.1, 29.2 Hz), 124.0, 124.1, 127.8, 128.3, 129.6, 129.9, 133.1, 133.6, 134.8, 135.0, 164.9, 164.9, 171.3, 171.4; EI-MS m/z 424 (M^+ , 0.9), 234 (91.0), 221 (100); FAB-MS (3-nitrobenzyl alcohol) m/z 425 (M^+ + H). Anal. Calcd for C₂₀H₁₆F₄N₂O₄: C, 56.61; H, 3.80; N, 6.60. Found: C, 56.83; H, 3.52; N, 6.42.

cis- and trans-2-[2-Oxo-9-(2,2,2-trifluoroethoxy)-1-azaspiro[5.5]undeca-7,10-dien-1-yl]isoindole-1,3-dione (6c). White crystals: mp 151-165 °C (EtOAc/*n*-hexane); IR (KBr) 1805, 1725, 1700, 1675, 715 cm⁻¹; ¹H NMR (270 MHz) δ 2.02-2.18 (m, 4H), 2.69 (t, J = 5.8 Hz, 2H), 3.24 (q, $J_{\text{H-F}}$ = 8.6 Hz, 1H), 3.67 (q, $J_{\text{H-F}}$ = 8.6 Hz, 1H), 4.37-4.43 (m, 0.5H), 4.49-4.55 (m, 0.5H), 5.86-5.98 (m, 2H), 6.33-6.42 (m, 2H), 7.72-7.81 (m, 2H), 7.82-7.88 (m, 2H); ¹³C NMR (67.8 MHz) δ 16.7, 17.4, 32.3, 36.5, 37.0, 61.6, 61.9, 62.1 (q, $J_{\text{C-F}}$ = 34.1 Hz), 63.3 (q, $J_{\text{C-F}}$ = 34.1 Hz), 68.0, 69.2, 121.3 (q, $J_{\text{C-F}}$ = 276.2 Hz), 123.7 (q, $J_{\text{C-F}}$ = 276.2 Hz), 123.79, 123.84, 126.6, 127.0, 129.6, 129.8, 133.9, 134.5, 134.6, 134.7, 165.6, 165.7, 167.3, 167.4; EI-MS m/z 406 (M^+ , 0.8), 306 (11.7), 245 (20.6), 233 (14.3), 204 (46.1), 189 (50.0), 162 (100); FAB-MS (3-nitrobenzyl alcohol) m/z 407 (M^+ + H). Anal. Calcd for C₂₀H₁₇F₃N₂O₄: C, 59.12; H, 4.22; N, 6.89. Found: C, 59.03; H, 4.13; N, 6.86.

cis- and trans-2-[6-Methyl-2-oxo-8-(2,2,2-trifluoroethoxy)-1-azaspiro[4.5]deca-6,9-dien-1-yl]isoindole-1,3-dione (6q). Colorless oil: IR (KBr) 1805, 1750, 715 cm^{-1} ; ^1H NMR (270 MHz) δ 2.12 (s, 3H), 2.20-2.50 (m, 2H), 2.57-2.86 (m, 2H), 3.13-3.40 (m, 1H), 3.66 (q, $J_{\text{H-F}} = 8.6$ Hz, 1H), 4.40-4.46 (m, 0.52H), 4.56-4.62 (m, 0.48H), 5.69-5.74 (m, 0.48H), 5.76-5.82 (m, 0.52H), 5.92 (ddd, $J = 9.9, 3.9, 2.2$ Hz, 0.48H), 5.98 (ddd, $J = 9.9, 3.9, 2.2$ Hz, 0.52H), 6.17 (d, $J = 9.9$ Hz, 1H), 7.72-8.00 (m, 4H); ^{13}C NMR (125.7 MHz) δ 18.36, 18.42, 27.6, 27.7, 29.4, 30.1, 61.6 (q, $J_{\text{C-F}} = 35.2$ Hz), 63.2 (q, $J_{\text{C-F}} = 35.2$ Hz), 63.1, 63.4, 69.3, 69.9, 123.6 (q, $J_{\text{C-F}} = 278.3$ Hz), 123.95, 123.97 (q, $J_{\text{C-F}} = 278.3$ Hz), 124.01, 124.8, 125.2, 126.2, 127.6, 128.3, 129.5, 129.6, 129.9, 129.9, 134.2, 134.5, 134.8, 134.9, 134.94, 135.0, 139.5, 140.2, 165.0, 165.1, 165.2, 165.2, 171.5, 171.7; EI-MS m/z 406 (M^+ , 0.6), 244 (29.1), 216 (41.8), 203 (100); FAB-MS (3-nitrobenzyl alcohol) m/z 407 ($\text{M}^+ + \text{H}$); HR-MS (FAB) for $\text{C}_{20}\text{H}_{18}\text{F}_3\text{N}_2\text{O}_4$ calcd 407.1218 ($\text{M}^+ + \text{H}$), found 407.1219.

2-(5-Methyl-2-oxo-3,4-dihydro-2H-quinolin-1-yl)isoindole-1,3-dione (2q). White crystals: mp 282-284 $^{\circ}\text{C}$ (EtOH); IR (KBr) 1795, 1740, 1720, 720 cm^{-1} ; ^1H NMR (270 MHz) δ 2.35 (s, 3H), 2.86-2.95 (m, 2H), 3.05-3.14 (m, 2H), 6.67 (d, $J = 7.9$ Hz, 1H), 6.95 (d, $J = 7.9$ Hz, 1H), 7.07 (t, $J = 7.9$ Hz, 1H), 7.81-7.89 (m, 2H), 7.94-8.01 (m, 2H); ^{13}C NMR (125.7 MHz, DMSO- d_6) δ 19.0, 20.8, 29.9, 11.6, 123.1, 124.1, 125.8, 127.1, 129.5, 135.6, 136.0, 138.7, 164.7, 166.7; EI-MS m/z 306 (M^+ , 100), 305 (37.6), 159 (12.5), 132 (23.6), 131 (21.1). Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_3$: C, 70.58; H, 4.61; N, 9.15. Found: C, 70.42; H, 4.64; N, 9.05.

N-(1,3-Dioxo-1,3-dihydroisoindol-2-yl)-3-[4-(2,2,2-trifluoroethoxymethyl)phenyl]propionamide (6r). White crystals: mp 133-136 $^{\circ}\text{C}$ (benzene/*n*-hexane); IR (KBr) 1800, 1745, 710 cm^{-1} ; ^1H NMR (270 MHz) δ 2.73 (t, $J = 7.6$ Hz, 2H), 3.08 (t, $J = 7.6$ Hz, 2H), 3.57 (q, $J_{\text{H-F}} = 8.7$ Hz, 2H), 4.66 (s, 2H), 7.23 (br s, 1H), 7.28 (d, $J = 8.3$ Hz, 2H), 7.32 (d, $J = 8.3$ Hz, 2H), 7.78-7.84 (m, 2H), 7.88-7.94 (m, 2H); ^{13}C NMR (67.8 MHz) δ 30.7, 31.0, 35.4, 35.8, 67.0 (q, $J_{\text{H-F}} = 102.1$ Hz), 73.8, 123.9, 124.0 (q, $J_{\text{H-F}} = 279.3$ Hz), 128.2, 128.6, 129.8, 134.5, 134.6, 140.1, 164.9, 170.6, 170.8; EI-MS m/z 406 (M^+ , 4.6), 391 (19.6), 306 (59.5), 216 (87.4), 203 (100), 144 (78.0). Anal. Calcd for $\text{C}_{20}\text{H}_{17}\text{F}_3\text{N}_2\text{O}_4$: C, 59.12; H, 4.22; N, 6.89. Found: C, 59.34; H, 4.19; N, 7.03.

2-(7-Methyl-2-oxo-3,4-dihydro-2H-quinolin-1-yl)isoindole-1,3-dione (2r). White crystals: mp 265-266 $^{\circ}\text{C}$ (EtOAc/*n*-hexane); IR (KBr) 1795, 1745, 1720, 725 cm^{-1} ; ^1H NMR (270 MHz) δ 2.25 (s, 3H), 2.86-2.93 (m, 2H), 3.10 (t, $J = 7.1$ Hz, 2H), 6.60 (s, 1H), 6.88 (d, $J = 7.6$ Hz, 1H), 7.12 (d, $J =$

7.6 Hz, 1H), 7.83-7.89 (m, 2H), 7.96-8.02 (m, 2H); ^{13}C NMR (67.8 MHz) δ 21.3, 24.6, 31.3, 113.6, 121.7, 124.1, 124.71, 127.8, 129.9, 134.7, 137.5, 138.5, 164.4, 167.0; EI-MS m/z 306 (M^+ , 100), 159 (18.6), 131 (24.2). Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_3$: C, 70.56; H, 4.61; N, 9.15. Found: C, 70.40; H, 4.64; N, 9.11.

cis- and trans-2-[6-Chloro-2-oxo-8-(2,2,2-trifluoroethoxy)-1-azaspiro[4.5]deca-6,9-dien-1-yl]isoindole-1,3-dione (6s). Colorless oil: IR (KBr) 1805, 1750, 710 cm^{-1} ; ^1H NMR (270 MHz) • 2.25-2.49 (m, 1H), 2.51-2.75 (m, 2H), 2.89-3.07 (m, 1H), 3.19-3.46 (m, 0.84H), 3.71 (q, $J_{\text{H-F}} = 8.6$ Hz, 1.16H), 4.53-4.60 (m, 0.58H), 4.70 (t, $J = 3.8$ Hz 0.42H), 5.95 (ddd, $J = 9.9, 3.6, 1.9$ Hz, 0.42H), 6.02 (ddd, $J = 10.0, 3.7, 2.0$ Hz, 0.58H), 6.12-6.23 (m, 2H), 7.74-7.92 (m, 4H); ^{13}C NMR (125.7 MHz) • 27.5, 27.7, 30.2, 30.8, 62.6 (q, $J_{\text{C-F}} = 35.4$ Hz) 63.7, 63.9 (q, $J_{\text{C-F}} = 35.4$ Hz), 64.0, 70.4, 71.0, 123.2 (q, $J_{\text{C-F}} = 277.3$ Hz), 123.7 (q, $J_{\text{C-F}} = 277.3$ Hz), 124.0, 124.1, 124.2, 126.7, 127.6, 127.7, 128.4, 129.5, 129.6, 129.8, 130.1, 133.1, 133.6, 134.9, 135.00, 135.04, 135.1, 138.3, 139.2, 164.5, 164.6, 165.1, 165.2, 171.3, 171.5; EI-MS m/z 428 ($\text{M}^+ + 2$, 0.7), 426 (M^+ , 2.0), 391 (37.3), 236 (35.6), 223 (100), 162 (82.1); FAB-MS (3-nitrobenzyl alcohol) m/z 427 ($\text{M}^+ + \text{H}$); HR-MS (FAB) for $\text{C}_{19}\text{H}_{15}\text{ClF}_3\text{N}_2\text{O}_4$ calcd 427.0665 ($\text{M}^+ + \text{H}$), found 427.0658.

cis- and trans-2-[6-Fluoro-2-oxo-8-(2,2,2-trifluoroethoxy)-1-azaspiro[4.5]deca-6,9-dien-1-yl]isoindole-1,3-dione (6t). Colorless oil: IR (KBr) 1805, 1740, 710 cm^{-1} ; ^1H NMR (270 MHz) • 2.20-2.75 (m, 3H), 2.75-3.07 (m, 1H), 3.14-3.45 (m, 0.88H), 3.69 (q, $J_{\text{H-F}} = 8.6$ Hz, 1.12H), 4.69-4.77 (m, 0.56H), 4.83-4.90 (m, 0.44H), 5.51-5.69 (m, 1H), 5.56 (d, $J = 14.2$ Hz, 0.44H), 5.62 (d, $J = 14.2$ Hz, 0.56H), 6.16 (m, 2H), 7.74-7.93 (m, 4H); ^{13}C NMR (125.7 MHz) • 27.2 (d, $J_{\text{C-F}} = 2.0$ Hz) 27.4 (d, $J_{\text{C-F}} = 2.0$ Hz), 29.0, 29.7, 61.1 (d, $J_{\text{C-F}} = 24.1$ Hz), 61.4 (d, $J_{\text{C-F}} = 24.1$ Hz), 62.2 (q, $J_{\text{C-F}} = 35.2$ Hz), 63.5 (q, $J_{\text{C-F}} = 35.2$ Hz), 70.9 (d, $J_{\text{C-F}} = 10.3$ Hz), 71.6 (d, $J_{\text{C-F}} = 10.3$ Hz), 103.1 (d, $J_{\text{C-F}} = 14.4$ Hz), 105.9 (d, $J_{\text{C-F}} = 14.4$ Hz), 123.3 (q, $J_{\text{C-F}} = 278.3$ Hz), 123.7 (q, $J_{\text{C-F}} = 278.3$ Hz), 124.10, 124.14, 124.2, 128.87, 128.89, 129.4, 129.45, 129.47, 129.6, 129.7, 129.9, 131.3 (d, $J_{\text{C-F}} = 5.2$ Hz) 131.8 (d, $J_{\text{C-F}} = 5.2$ Hz) 134.9, 135.03, 135.06, 135.1, 159.6 (d, $J_{\text{C-F}} = 268.1$ Hz), 159.9 (d, $J_{\text{C-F}} = 268.1$ Hz), 164.40, 164.41, 165.1, 165.2, 171.4, 171.5; EI-MS m/z 410 (M^+ , 0.5), 390 (5.7), 220 (65.9), 207 (100), 162 (79.3); FAB-MS (3-nitrobenzyl alcohol) m/z 411 ($\text{M}^+ + \text{H}$); HR-MS (FAB) for $\text{C}_{19}\text{H}_{15}\text{F}_4\text{N}_2\text{O}_4$ calcd 411.0966 ($\text{M}^+ + \text{H}$), found 411.0964.

cis and trans-2-[3-Oxo-8-(2,2,2-trifluoroethoxy)-1-oxa-4-azaspiro[4.5]deca-6,9-dien-4-yl]isoindole-1,3-dione (6i). Colorless oil: IR (KBr) 1805, 1745, 720 cm^{-1} ; ^1H NMR (500 MHz) δ 3.35 (q, $J_{\text{H-F}} = 8.6$ Hz, 1H), 3.67 (q, $J_{\text{H-F}} = 8.6$ Hz, 1H), 4.45-4.47 (m, 0.5H), 4.52-4.55 (m, 0.5H), 4.58 (s, 1H), 4.60 (s, 1H), 6.16-6.26 (m, 2H), 6.28-6.36 (m, 2H), 7.79-7.84 (m, 2H) 7.88-7.93 (m, 2H); ^{13}C NMR (125.7 MHz) δ 63.0 (q, $J_{\text{C-F}} = 35.2$ Hz), 63.2 (q, $J_{\text{C-F}} = 35.2$ Hz) 64.9, 64.9, 68.1, 68.6, 86.9, 87.2, 123.3 (q, $J_{\text{C-F}} = 278.3$ Hz), 123.8 (q, $J_{\text{C-F}} = 277.9$ Hz), 124.3, 124.4, 129.5, 129.7, 129.9, 132.7,

132.7, 135.1, 135.2, 164.0, 164.0, 167.1, 167.2; EI-MS m/z 394 (M^+ , 7.8), 247 (78.6), 192 (100), 175 (45.5); HR-MS (EI) for $C_{18}H_{13}F_3N_2O_4$ calcd 394.0777, found 394.0769.