

## Supporting Information for

# Asymmetric Organocatalytic Formal Double-Arylation of Azomethines for the Synthesis of Highly Enantiomerically Enriched Isoindolines

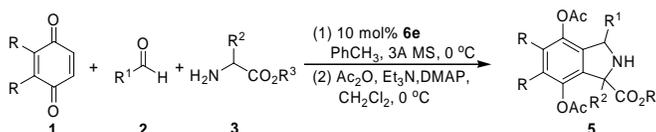
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**General Information:** NMR spectra were recorded on a Bruker-400 MHz spectrometer. HRMS (Bio TOF Q) spectra were recorded on P-SIMS-Gly of Bruker Daltonics Inc. Infrared spectra were recorded on a Nicolet MX-1E FT-IR spectrometer. Optical rotations were measured on Perkin Elmer Model 343 Polarimeter. HPLC analysis was performed on Waters-Breeze (2487 Dual Absorbance Detector and 1525 Binary HPLC Pump). Chiralpak AD, AS, and OD columns were purchased from Daicel Chemical Industries, LTD. X-ray crystallography analysis was performed on an Oxford Diffraction Gemini S Ultra CCD diffractionmeter equipped with mirror Cu K $\alpha$  ( $\lambda = 1.54184 \text{ \AA}$ ) radiation at room temperature. All structures were solved by direct methods and refined by full-matrix least-squares methods with SHELXL-97 programs. Elemental analysis was performed using an Elementar Vario EL III instrument. Solvents for the column chromatography were distilled before use. All starting materials commercially available were used directly. Quinones **1c** and **1d** were prepared according to the methods reported in the literature.<sup>1</sup> Catalyst **6e** was prepared according to previously described procedures<sup>2</sup> and was acidified with 4N HCl before use.

## General Procedure for the Asymmetric Organocatalytic Formal Double-Arylation of Azomethines:



To a solution of an aldehyde **2** (0.24 mmol), the catalyst **6e** (0.02 mmol), and 3Å molecular sieves (300 mg) in PhCH<sub>3</sub> (2.0 mL) was added the amino ester (0.2 mmol). After the mixture was stirred at 25 °C

for 1 hour, the reaction mixture was cooled down to 0 °C and quinone **1** was added. The reaction mixture was stirred for ca. 12 hours until the reaction was complete (monitored by TLC). The reaction mixture was directly charged to a short column chromatography on silica gel (petroleum ether/EtOAc = 5/1) to remove the quinone, giving the crude **4** (when quinone **1b** and **1d** was used, **4** was partially isomerized), which was directly transferred to a test tube. The atmosphere was replaced with argon and CH<sub>2</sub>Cl<sub>2</sub> (2 mL) was added. The test tube was placed in an ice-water bath, and then DMAP (0.2 mmol), Ac<sub>2</sub>O (1.0 mmol) and Et<sub>3</sub>N (1.0 mmol) were added, sequentially. After the consumption of the starting material (monitored by TLC), the reaction mixture was directly charged to column chromatography on silica gel (petroleum ether/ ethyl acetate = 4/1) to give pure product **5**.

**Diethyl 3-(4-nitrophenyl)-4,9-dioxo-2,3,3a,4-tetrahydro-1H-benzo[f]isoindole-1,1(9H,9aH)-dicarboxylate (4a):** Yield: 95%;  $[\alpha]_D^{20} = -57.3$  (c 1.5, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.31-1.40 (m, 6H), 3.31 (s, 1H), 4.25 (dd,  $J_1 = 7.9$  Hz,  $J_2 = 10.5$  Hz, 1H), 4.30-4.48 (m, 5H), 4.98 (d,  $J = 10.5$  Hz, 1H), 7.34 (d,  $J = 8.8$  Hz, 2H), 7.5-7.6 (m, 3H), 7.82 (d,  $J = 8.8$  Hz, 2H), 7.87-7.90 (m, 1H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.06, 14.10, 54.09, 54.84, 61.29, 62.32, 62.83, 75.04, 122.99, 126.28, 126.94, 128.59, 134.47, 134.71, 134.86, 134.92, 146.58, 147.24, 168.36, 170.12, 192.72, 193.55; Enantiomeric excess: 97%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R = 14.69$  min (minor),  $t_R = 23.41$  min (major); IR (KBr): γ 3358, 2957, 2923, 2869, 2852, 2361, 2345, 1757, 1729, 1685, 1591, 1518, 1465, 1345, 1254, 1097, 855, 749 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>24</sub>H<sub>22</sub>N<sub>2</sub>O<sub>8</sub>) requires m/z 466.1376, found m/z 466.1376.

**Diethyl 4,9-diacetoxy-3-(4-nitrophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5a):** Yield: 94%; m.p. 185-187 °C;  $[\alpha]_D^{20} = +88.3$  (c 2.1, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.29-1.33 (m, 6H), 1.93 (s, 3H), 2.45 (s, 3H), 3.97 (s, 1H), 4.13-4.43 (m, 4H), 5.79 (s, 1H), 7.50-7.56 (m, 4H), 7.67-7.74 (m, 2H), 8.17 (d,  $J = 8.5$  Hz, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.02, 14.09, 20.11, 20.94, 62.46, 63.11, 64.96, 76.24, 121.54, 122.92, 123.61, 126.93, 127.08, 127.50, 128.57, 128.90, 129.62, 132.50, 139.29, 141.43, 147.69, 149.21, 167.40, 168.39, 168.73, 169.57; Enantiomeric excess: 97%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_R = 7.23$  min(minor),  $t_R = 21.32$  min(major); IR (KBr): γ 3435, 2959, 2922, 2852, 1755, 1736, 1636, 1608, 1523, 1429, 1348, 1197, 1173, 1093, 860 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>28</sub>H<sub>26</sub>N<sub>2</sub>O<sub>10</sub>+H)<sup>+</sup> requires m/z 551.1666, found m/z 551.1660; Anal. Calcd for

C<sub>28</sub>H<sub>26</sub>N<sub>2</sub>O<sub>10</sub>: C, 61.09; H, 4.76; N, 5.09. Found: C, 60.84; H, 4.82; N, 4.68.

**Diethyl 4,9-diacetoxy-3-(4-bromophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate(5b):**

Yield: 98%; m.p. 187-189 °C;  $[\alpha]_D^{20} = +77.0$  (c 2.4, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.26-1.32 (m, 6H), 1.90 (s, 3H), 2.43 (s, 3H), 3.85 (s, 1H), 4.12-4.40 (m, 4H), 5.63 (s, 1H), 7.20 (d, *J* = 7.1 Hz, 2H), 7.44-7.46 (m, 2H), 7.51-7.53 (m, 2H), 7.71 (d, *J* = 7.1 Hz, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.06, 14.10, 20.08, 20.10, 62.41, 62.96, 65.32, 76.13, 121.64, 122.05, 122.86, 126.87, 127.31, 128.42, 129.00, 130.47, 131.61, 132.94, 139.27, 140.57, 141.25, 167.54, 168.47, 168.83, 169.69; Enantiomeric excess: 95%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 30 °C, 254 nm): t<sub>R</sub> = 7.94 min(major), t<sub>R</sub> = 10.22 min(minor); IR (KBr): γ 3434, 3345, 2983, 2934, 1774, 1727, 1360, 1198, 1174, 1093, 1046, 1012, 766 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>28</sub>H<sub>26</sub>BrNO<sub>8</sub>+H)<sup>+</sup> requires m/z 584.0920, found m/z 584.0916; Anal. Calcd for C<sub>28</sub>H<sub>26</sub>BrNO<sub>8</sub>: C, 57.54; H, 4.48; N, 2.40. Found: C, 57.65; H, 4.62; N, 2.23.

**Diethyl 4,9-diacetoxy-3-(4-chlorophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5c):**

Yield: 98%; m.p. 197-199 °C;  $[\alpha]_D^{20} = +73.3$  (c 2.2, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.27-1.32 (m, 6H), 1.90 (s, 3H), 2.43 (s, 3H), 3.84 (s, 1H), 4.11-4.41 (m, 4H), 5.64 (s, 1H), 7.25-7.31 (m, 4H), 7.49-7.53 (m, 2H), 7.71 (d, *J* = 7.0 Hz, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.08, 14.11, 20.10, 21.00, 62.43, 62.97, 65.29, 76.14, 121.65, 122.88, 126.88, 127.32, 128.44, 128.67, 129.02, 130.14, 133.02, 133.94, 139.30, 140.07, 141.27, 167.54, 168.48, 168.86, 169.71; Enantiomeric excess: 91%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 90/ 10, flow rate 1.0 mL/min, T = 30 °C, 254 nm): t<sub>R</sub> = 11.74 min(major), t<sub>R</sub> = 15.28 min(minor); IR (KBr): γ 3435, 3342, 2984, 2936, 1774, 1727, 1609, 1490, 1431, 1359, 1198, 1174, 1093, 1046, 1016, 767 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>28</sub>H<sub>26</sub>ClNO<sub>8</sub>+H)<sup>+</sup> requires m/z 540.1425, found m/z 540.1424; Anal. Calcd for C<sub>28</sub>H<sub>26</sub>ClNO<sub>8</sub>: C, 62.28; H, 4.85; N, 2.59. Found: C, 62.47; H, 5.02; N, 2.43.

**Diethyl 4,9-diacetoxy-3-(4-cyanophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5d):**

Yield: 95%; m.p. 173-175 °C;  $[\alpha]_D^{20} = +79.7$  (c 2.0, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.25-1.33 (m, 6H), 1.91 (s, 3H), 2.44 (s, 3H), 3.94 (s, 1H), 4.11-4.42 (m, 4H), 5.73 (s, 1H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.51-7.55 (m, 2H), 7.62 (d, *J* = 8.3 Hz, 2H), 7.68-7.72 (m, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.05, 14.11, 20.08, 20.98, 62.47, 63.12, 65.33, 76.23, 111.93, 118.73, 121.59, 122.93, 127.08, 127.50, 128.57, 128.93, 129.57, 132.28, 132.52, 139.30, 141.41, 147.16, 167.39, 168.42,

168.74, 169.60; Enantiomeric excess: 93%, determined by HPLC (Daicel Chirapak AS-H, hexane/isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_R = 9.38$  min(major),  $t_R = 20.82$  min(minor); IR (KBr):  $\gamma$  3434, 2983, 2932, 2228, 1773, 1731, 1608, 1446, 1360, 1198, 1174, 1093, 1047, 883, 768  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{29}\text{H}_{26}\text{N}_2\text{O}_8+\text{H})^+$  requires m/z 531.1767, found m/z 531.1768; Anal. Calcd for  $\text{C}_{29}\text{H}_{26}\text{N}_2\text{O}_8$ : C, 65.65; H, 4.94; N, 5.28. Found: C, 65.34; H, 5.01; N, 5.02.

**Diethyl 4,9-diacetoxy-3-(4-(methoxycarbonyl)phenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5e):** Yield: 91%; m.p. 174-176 °C;  $[\alpha]_D^{20} = +77.2$  (c 2.3,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.26-1.33 (m, 6H), 1.87 (s, 3H), 2.44 (s, 3H), 3.91 (s, 3H), 3.9 (s, 1H), 4.14-4.41 (m, 4H), 5.73 (s, 1H), 7.46 (d,  $J = 7.4\text{Hz}$ , 2H), 7.48-7.55 (m, 2H), 7.69-7.73 (m, 2H), 8.01 (d,  $J = 7.4$  Hz, 2H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.06, 14.09, 20.09, 20.98, 52.16, 62.43, 62.98, 65.56, 76.27, 121.63, 122.88, 126.89, 127.33, 128.46, 128.80, 128.99, 129.82, 129.93, 132.98, 139.30, 141.29, 146.69, 166.86, 167.51, 168.46, 168.84, 169.68; Enantiomeric excess: 95%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_R = 6.65$  min(major),  $t_R = 11.52$  min(minor); IR (KBr):  $\gamma$  3435, 2959, 2922, 2852, 1769, 1732, 1631, 1442, 1360, 1199, 1175, 1095, 771  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{30}\text{H}_{29}\text{NO}_{10}+\text{H})^+$  requires m/z 564.1870, found m/z 564.1868; Anal. Calcd for  $\text{C}_{30}\text{H}_{29}\text{NO}_{10}$ : C, 63.94; H, 5.19; N, 2.49. Found: C, 63.72; H, 5.30; N, 2.25.

**Diethyl 4,9-diacetoxy-3-phenyl-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5f):** Yield: 98%; m.p. 136-138 °C;  $[\alpha]_D^{20} = +157.7$  (c 0.8,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.27-1.32 (m, 6H), 1.82 (s, 3H), 2.43 (s, 3H), 3.9 (s, 1H), 4.13-4.40 (m, 4H), 5.66 (s, 1H), 7.25-7.34 (m, 5H), 7.48-7.52 (m, 2H), 7.70-7.72 (m, 2H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.08, 14.10, 19.97, 21.01, 62.39, 62.87, 66.09, 76.22, 121.70, 122.84, 126.71, 127.16, 127.57, 128.19, 128.35, 128.55, 128.79, 129.06, 133.43, 139.32, 141.17, 141.27, 167.58, 168.51, 168.92, 169.78; Enantiomeric excess: 83%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R = 7.73$  min(minor),  $t_R = 9.79$  min(major); IR (KBr):  $\gamma$  3335, 2982, 2358, 2331, 1759, 1727, 1358, 1239, 1195, 1171, 1094, 1028, 855, 772, 754  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{28}\text{H}_{27}\text{NO}_8+\text{H})^+$  requires m/z 506.1815, found m/z 506.1822; Anal. Calcd for  $\text{C}_{28}\text{H}_{27}\text{NO}_8$ : C, 66.53; H, 5.38; N, 2.77. Found: C, 66.53; H, 5.46; N, 2.55.

**Diethyl 4,9-diacetoxy-3-p-tolyl-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5g):** Yield:

95%; m.p. 173-175 °C;  $[\alpha]_D^{20} = +84.3$  (c 1.6, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.26-1.32 (m, 6H), 1.84 (s, 3H), 2.34 (s, 3H), 2.43 (s, 3H), 3.8 (s, 1H), 4.11-4.40 (m, 4H), 5.62 (s, 1H), 7.13 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 7.48-7.52 (m, 2H), 7.70-7.72 (m, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.09, 20.04, 21.02, 21.26, 62.38, 62.83, 65.85, 76.20, 121.70, 122.83, 126.67, 127.13, 127.64, 128.32, 128.64, 129.08, 129.21, 133.57, 137.86, 138.26, 139.29, 141.14, 167.62, 168.55, 168.95, 169.82; Enantiomeric excess: 86%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 25 °C, 254 nm): *t<sub>R</sub>* = 7.57 min (minor), *t<sub>R</sub>* = 11.56 min (major); IR (KBr): γ 3341, 2985, 2923, 2856, 1774, 1731, 1430, 1359, 1261, 1197, 1173, 1093, 1044, 1022, 765 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>29</sub>H<sub>29</sub>NO<sub>8</sub>+H)<sup>+</sup> requires m/z 520.1971, found m/z 520.1970; Anal. Calcd for C<sub>29</sub>H<sub>29</sub>NO<sub>8</sub>: C, 67.04; H, 5.63; N, 2.70. Found: C, 67.02; H, 5.76; N, 2.45.

**Diethyl 4,9-diacetoxy-3-(3-chlorophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5h):**

Yield: 98%; m.p. 180-182 °C;  $[\alpha]_D^{20} = +57.0$  (c 2.1, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.26-1.33 (m, 6H), 1.95 (s, 3H), 2.44 (s, 3H), 3.9 (s, 1H), 4.11-4.41 (m, 4H), 5.65 (s, 1H), 7.24-7.27 (m, 3H), 7.34 (s, 1H), 7.51-7.53 (m, 2H), 7.70-7.72 (m, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.07, 14.11, 20.08, 20.99, 62.45, 63.00, 65.42, 76.19, 121.66, 122.89, 126.89, 127.19, 127.32, 128.26, 128.46, 128.94, 129.03, 129.82, 132.96, 134.26, 139.35, 141.27, 143.78, 167.56, 168.48, 168.89, 169.72; Enantiomeric excess: 94%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 30 °C, 254 nm): *t<sub>R</sub>* = 7.71 min(minor), *t<sub>R</sub>* = 9.08 min(major); IR (KBr): γ 3439, 3338, 2987, 2928, 1771, 1728, 1439, 1359, 1200, 1176, 1016, 767 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>28</sub>H<sub>26</sub>ClNO<sub>8</sub>+H)<sup>+</sup> requires m/z 540.1425, found m/z 540.1426; Anal. Calcd for C<sub>28</sub>H<sub>26</sub>ClNO<sub>8</sub>: C, 62.28; H, 4.85; N, 2.59. Found: C, 62.44; H, 4.94; N, 2.44.

**Diethyl 4,9-diacetoxy-3-(3-methoxyphenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5i):**

Yield: 98%; m.p. 128-130 °C;  $[\alpha]_D^{20} = +81.6$  (c 2.1, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm): 1.27-1.32 (m, 6H), 1.88 (s, 3H), 2.43 (s, 3H), 3.74 (s, 3H), 3.85 (s, 1H), 4.13-4.40 (m, 4H), 5.64 (s, 1H), 6.84-6.87 (m, 2H), 6.92-6.94 (m, 1H), 7.22-7.24 (m, 1H), 7.48-7.52 (m, 2H), 7.70-7.72 (m, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm): 14.09, 14.12, 20.03, 21.02, 55.30, 62.39, 62.90, 66.05, 76.20, 113.97, 114.10, 121.11, 121.74, 122.86, 126.73, 127.18, 127.52, 128.36, 129.10, 129.46, 133.34, 139.38, 141.18, 142.91, 159.85, 167.66, 168.54, 168.95, 169.81; Enantiomeric excess: 97%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm): *t<sub>R</sub>* = 5.61 min(major), *t<sub>R</sub>* = 7.24 min(minor); IR (KBr): γ 3351, 2982, 2938, 1770, 1732,

1608, 1360, 1240, 1198, 1173, 1044, 766  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{29}\text{H}_{29}\text{NO}_9+\text{H})^+$  requires  $m/z$  536.1921, found  $m/z$  536.1917; Anal. Calcd for  $\text{C}_{29}\text{H}_{29}\text{NO}_9$ : C, 65.04; H, 5.46; N, 2.62. Found: C, 65.04; H, 5.60; N, 2.40.

**Diethyl 4,9-diacetoxy-3-(2,3-dichlorophenyl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5j):** Yield: 96%; m.p. 68-70  $^{\circ}\text{C}$ ;  $[\alpha]_{\text{D}}^{20} = -15.1$  (c 2.2,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.27-1.33 (m, 6H), 2.00 (s, 3H), 2.43 (s, 3H), 3.97 (s, 1H), 4.13-4.42 (m, 4H), 6.37 (s, 1H), 7.10-7.14 (m, 1H), 7.12-7.24 (m, 1H), 7.38-7.40 (m, 1H), 7.48-7.55 (m, 2H), 7.62-7.64 (m, 1H), 7.70-7.73 (m, 1H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.06, 14.10, 19.94, 20.99, 61.77, 62.39, 63.09, 76.23, 121.71, 122.99, 126.89, 127.03, 127.39, 127.81, 128.55, 128.98, 129.53, 129.82, 131.75, 132.80, 139.40, 141.45, 167.58, 168.42, 168.84, 169.62; Enantiomeric excess: 91%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 90/ 10, flow rate 1.0 mL/min, T = 30  $^{\circ}\text{C}$ , 254 nm):  $t_{\text{R}} = 7.83$  min(major),  $t_{\text{R}} = 10.35$  min(minor); IR (KBr):  $\gamma$  3349, 3074, 2982, 2937, 1773, 1735, 1427, 1360, 1241 1197, 1172, 1094, 1046, 764  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{28}\text{H}_{25}\text{Cl}_2\text{NO}_8+\text{H})^+$  requires  $m/z$  574.1035, found  $m/z$  574.1042; Anal. Calcd for  $\text{C}_{28}\text{H}_{25}\text{Cl}_2\text{NO}_8$ : C, 58.55; H, 4.39; N, 2.44. Found: C, 58.58; H, 4.52; N, 2.17.

**Diethyl 4,9-diacetoxy-3-(thiophen-3-yl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarb oxylate (5k):** Yield: 96%; m.p. 140-142  $^{\circ}\text{C}$ ;  $[\alpha]_{\text{D}}^{20} = +62.3$  (c 2.0,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.25-1.31(m, 6H), 1.95 (s, 3H), 2.42 (s, 3H), 3.80 (s, 1H), 4.13-4.38 (m, 4H), 5.80 (s, 1H), 6.93-6.95 (m, 1H), 7.24-7.26 (m, 2H), 7.47-7.53 (m, 2H), 7.70-7.76 (m, 2H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 13.98, 19.94, 20.90, 60.92, 62.35, 62.76, 75.92, 121.61, 122.75, 123.30, 126.24, 126.68, 127.14, 127.23, 127.55, 128.27, 128.95, 132.50, 139.28, 141.13, 142.15, 167.56, 168.42, 168.81, 169.59; Enantiomeric excess: 94%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30  $^{\circ}\text{C}$ , 254 nm):  $t_{\text{R}} = 5.54$  min(minor),  $t_{\text{R}} = 8.13$  min(major); IR (KBr):  $\gamma$  3436, 3343, 2980, 2924, 1773, 1731, 1610, 1429, 1359, 1241, 1198, 1174, 1094, 767  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{26}\text{H}_{25}\text{NO}_8\text{S}+\text{H})^+$  requires  $m/z$  512.1379, found  $m/z$  512.1371; Anal. Calcd for  $\text{C}_{26}\text{H}_{25}\text{NO}_8\text{S}$ : C, 61.05; H, 4.93; N, 2.74. Found: C, 61.11; H, 5.04; N, 2.58.

**Diethyl 4,9-diacetoxy-3-(furan-2-yl)-2,3-dihydro-1H-benzo[f]isoindole-1,1-dicarboxylate (5l):** Yield: 90%; m.p. 116-118  $^{\circ}\text{C}$ ;  $[\alpha]_{\text{D}}^{20} = +78.1$  (c 1.7,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.26-1.31 (m, 6H), 2.14 (s, 3H), 2.42 (s, 3H), 3.81 (s, 1H), 4.16-4.37 (m, 4H), 5.78 (s, 1H), 6.28-6.34

(m, 2H), 7.36-7.37 (m, 1H), 7.51-7.53 (m, 2H), 7.69-7.80 (m, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.02, 20.27, 20.95, 58.88, 62.53, 62.85, 75.97, 108.35, 110.31, 121.69, 122.86, 126.83, 127.26, 128.50, 128.97, 130.76, 139.36, 141.28, 142.84, 153.16, 167.80, 168.44, 168.71, 169.52; Enantiomeric excess: 90%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_{\text{R}} = 8.96$  min(minor),  $t_{\text{R}} = 11.27$  min(major); IR (KBr):  $\gamma$  3348, 2984, 2937, 1771, 1732, 1360, 1241, 1198, 1174, 1095, 1045, 767  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{26}\text{H}_{25}\text{NO}_9+\text{H})^+$  requires m/z 496.1608, found m/z 496.1606; Anal. Calcd for  $\text{C}_{26}\text{H}_{25}\text{NO}_9$ : C, 63.03; H, 5.09; N, 2.83. Found: C, 63.13; H, 5.19; N, 2.63.

**Diethyl 4,7-diacetoxy-3-(4-nitrophenyl)isoindoline-1,1-dicarboxylate (5m):** Yield: 89%; m.p. 122-125 °C;  $[\alpha]_{\text{D}}^{20} = -50.1$  (c 1.8,  $\text{CHCl}_3$ );  $^1\text{H}$ -NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.27-1.33 (m, 6H), 1.80 (s, 3H), 2.28 (s, 3H), 3.95 (d,  $J = 6.1$  Hz, 1H), 4.13-4.39 (m, 4H), 5.70 (d,  $J = 6.1$  Hz, 1H), 7.05 (d,  $J = 8.8$  Hz, 1H), 7.20 (d,  $J = 8.8$  Hz, 1H), 7.48 (d,  $J = 9.1$  Hz, 2H), 8.17 (d,  $J = 9.1$  Hz, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.03, 14.16, 20.30, 21.10, 62.50, 63.12, 65.27, 76.77, 123.67, 124.51, 124.71, 129.38, 130.36, 136.73, 143.03, 144.26, 147.71, 149.44, 167.75, 168.45, 168.84, 169.71; Enantiomeric excess: 94%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_{\text{R}} = 11.15$  min(major),  $t_{\text{R}} = 22.60$  min(minor); IR (KBr):  $\gamma$  3351, 2983, 2938, 1770, 1737, 1598, 1522, 1485, 1369, 1348, 1244, 1184, 1046, 1016, 938, 894, 858, 833, 650  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_{10}+\text{H})^+$  requires m/z 501.1509, found m/z 501.1504; Anal. Calcd for  $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_{10}$ : C, 57.60; H, 4.83; N, 5.60. Found: C, 57.86; H, 4.98; N, 5.26.

**Diethyl 4,7-diacetoxy-3-(4-bromophenyl)isoindoline-1,1-dicarboxylate (5n):** Yield: 86%; m.p. 128-130 °C;  $[\alpha]_{\text{D}}^{20} = -53.2$  (c 1.8,  $\text{CHCl}_3$ );  $^1\text{H}$ -NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.26-1.31 (m, 6H), 1.77 (s, 3H), 2.27 (s, 3H), 3.83 (s, 1H), 4.12-4.37 (m, 4H), 5.54 (s, 1H), 7.05 (d,  $J = 8.7$  Hz, 1H), 7.14-7.18 (m, 3H), 7.44 (d,  $J = 9.0$  Hz, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.03, 14.12, 20.18, 21.11, 62.40, 62.91, 65.55, 76.48, 122.00, 124.25, 124.39, 130.14, 130.47, 131.61, 137.32, 140.90, 143.09, 144.15, 167.93, 168.52, 168.95, 169.82; Enantiomeric excess: 94%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_{\text{R}} = 7.07$  min(minor),  $t_{\text{R}} = 12.39$  min(major); IR (KBr):  $\gamma$  3443, 3355, 2982, 2924, 2852, 1769, 1736, 1486, 1368, 1244, 1184, 1093, 1046, 1013, 892, 859, 651  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{24}\text{H}_{24}\text{BrNO}_8+\text{H})^+$  requires m/z 534.0764, found m/z 534.0757; Anal. Calcd for  $\text{C}_{24}\text{H}_{24}\text{BrNO}_8$ : C, 53.94; H, 4.53; N, 2.62. Found: C, 54.17; H, 4.59; N, 2.45.

**Diethyl 4,7-diacetoxy-3-(4-chlorophenyl)isoindoline-1,1-dicarboxylate (5o):** Yield: 89%; m.p. 113-115 °C;  $[\alpha]_D^{20} = -52.7$  (c 1.7, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 1.26-1.32 (m, 6H), 1.76 (s, 3H), 2.27 (s, 3H), 3.83 (s, 1H), 4.15-4.34 (m, 4H), 5.56 (s, 1H), 7.04 (d,  $J = 8.8$  Hz, 1H), 7.17 (d,  $J = 8.8$  Hz, 1H), 7.22 (d,  $J = 8.4$  Hz, 2H), 7.28 (d,  $J = 8.4$  Hz, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 14.02, 14.11, 20.16, 21.10, 62.38, 62.89, 65.49, 76.47, 124.22, 124.37, 128.64, 129.78, 130.46, 133.85, 137.38, 140.38, 143.09, 144.15, 167.90, 168.50, 168.96, 169.82; Enantiomeric excess: 91%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R = 6.76$  min(major),  $t_R = 8.36$  min(minor); IR (KBr):  $\gamma$  3353, 3072, 2983, 2935, 2896, 1770, 1737, 1485, 1369, 1244, 1180, 1049, 1015, 841, 735, 651, 593 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>24</sub>H<sub>24</sub>ClNO<sub>8</sub>+H)<sup>+</sup> requires m/z 490.1269, found m/z 490.1275; Anal. Calcd for C<sub>24</sub>H<sub>24</sub>ClNO<sub>8</sub>: C, 58.84; H, 4.94; N, 2.86. Found: C, 58.82; H, 4.97; N, 2.50.

**Diethyl 4,7-diacetoxy-3-(3-methoxyphenyl)isoindoline-1,1-dicarboxylate (5p):** Yield: 92%; m.p. 138-140 °C;  $[\alpha]_D^{20} = -49.3$  (c 1.8, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 1.26-1.31 (m, 6H), 1.74 (s, 3H), 2.27 (s, 3H), 3.74 (s, 3H), 3.83 (s, 1H), 4.12-4.37 (m, 4H), 5.55 (s, 1H), 6.81-6.83 (m, 2H), 6.85-6.87 (m, 1H), 7.03 (d,  $J = 8.7$  Hz, 1H), 7.16 (d,  $J = 8.7$  Hz, 1H), 7.21-7.26 (m, 1H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 14.05, 14.12, 20.11, 21.12, 55.29, 62.35, 62.83, 66.26, 76.55, 113.70, 113.90, 120.75, 123.99, 124.37, 129.46, 130.55, 137.76, 143.24, 144.11, 159.84, 168.03, 168.56, 169.08, 169.94; Enantiomeric excess: 94%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_R = 5.84$  min(major),  $t_R = 7.43$  min(minor); IR (KBr):  $\gamma$  3443, 3350, 3074, 2980, 2934, 2851, 1770, 1734, 1609, 1486, 1368, 1245, 1210, 1183, 1047, 896, 738, 599 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>25</sub>H<sub>27</sub>NO<sub>9</sub>+H)<sup>+</sup> requires m/z 486.1764, found m/z 486.1758; Anal. Calcd for C<sub>25</sub>H<sub>27</sub>NO<sub>9</sub>: C, 61.85; H, 5.61; N, 2.89. Found: C, 61.85; H, 5.66; N, 2.64.

**Diethyl 4,7-diacetoxy-5,6-dimethyl-3-(4-nitrophenyl)isoindoline-1,1-dicarboxylate (5q):** Yield: 89%; m.p. 159-161 °C;  $[\alpha]_D^{20} = -14.4$  (c 1.9, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 1.26-1.33 (m, 6H), 1.82 (s, 3H), 1.98 (s, 3H), 2.07 (s, 3H), 2.30 (s, 3H), 3.89 (s, 1H), 4.10-4.40 (m, 4H), 5.65 (s, 1H), 7.47 (d,  $J = 9.2$  Hz, 2H), 8.16 (d,  $J = 9.2$  Hz, 2H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 13.51, 14.06, 14.14, 14.35, 20.04, 20.89, 62.37, 62.99, 65.35, 77.30, 123.61, 127.99, 129.45, 132.82, 133.10, 134.40, 141.90, 143.02, 147.65, 149.85, 167.35, 167.93, 169.11, 169.81; Enantiomeric excess: 94%,

determined by HPLC (Daicel Chirapak IA-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R$  = 6.53 min(minor),  $t_R$  = 21.54 min(major); IR (KBr):  $\gamma$  3434, 3367, 2982, 2926, 1768, 1736, 1521, 1347, 1189, 1086, 857, 702, 592  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_{10}+\text{H})^+$  requires  $m/z$  529.1822, found  $m/z$  529.1827; Anal. Calcd for  $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_{10}$ : C, 59.09; H, 5.34; N, 5.30. Found: C, 59.51; H, 5.52; N, 4.93.

**Diethyl 4,7-diacetoxy-5,6-dichloro-3-(4-nitrophenyl)isoindoline-1,1-dicarboxylate (5r):** Yield: 78%; m.p. 124-126 °C;  $[\alpha]_D^{20} = +5.5$  (c 1.8,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.29-1.34 (m, 6H), 1.88 (s, 3H), 2.33 (s, 3H), 3.93 (d,  $J = 6.0$  Hz, 1H), 4.11-4.42 (m, 4H), 5.70 (d,  $J = 6.0$  Hz, 1H), 7.48 (d,  $J = 9.1$  Hz, 2H), 8.18 (d,  $J = 9.1$  Hz, 2H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 14.03, 14.12, 19.83, 20.64, 62.76, 63.41, 65.42, 77.30, 123.73, 129.19, 129.48, 129.58, 130.12, 136.57, 140.99, 142.43, 147.92, 148.33, 166.17, 166.39, 168.14, 168.93; Enantiomeric excess: 85%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min, T = 30 °C, 254 nm):  $t_R$  = 8.53 min(major),  $t_R$  = 14.53 min(minor); IR (KBr):  $\gamma$  3435, 3357, 2983, 2925, 1782, 1735, 1522, 1429, 1369, 1348, 1170, 1016, 917, 861, 843, 699  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{24}\text{H}_{22}\text{Cl}_2\text{N}_2\text{O}_{10}+\text{H})^+$  requires  $m/z$  569.0730, found  $m/z$  569.0725; Anal. Calcd for  $\text{C}_{24}\text{H}_{22}\text{Cl}_2\text{N}_2\text{O}_{10}$ : C, 50.63; H, 3.89; N, 4.92. Found: C, 50.75; H, 3.97; N, 4.53.

**1-(Methoxycarbonyl)-3-(4-nitrophenyl)-1-phenyl-2,3-dihydro-1H-benzo[f]isoindole-4,9-diyl diacetate (5s):** Yield: 76%;  $[\alpha]_D^{20} = +82.8$  (c 0.9,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.94 (s, 3H), 2.19 (s, 3H), 3.49 (s, 1H), 3.76 (s, 3H), 5.61 (s, 1H), 7.32-7.35 (m, 5H), 7.46 (d,  $J = 8.7$  Hz, 2H), 7.53-7.57 (m, 2H), 7.69-7.77 (m, 2H), 8.21 (d,  $J = 8.7$  Hz, 2H);  $^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 20.13, 20.54, 53.31, 64.89, 77.00, 121.69, 122.59, 123.96, 127.29, 127.52, 128.19, 128.48, 128.83, 129.35, 132.28, 132.94, 139.38, 140.70, 140.75, 147.80, 148.36, 167.59, 168.63, 172.58; Enantiomeric excess: 89%, determined by HPLC (Daicel Chirapak OD-H, hexane/ isopropanol = 85/ 15, flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R$  = 17.56 min(minor),  $t_R$  = 26.96 min(major); IR (KBr):  $\gamma$  3073, 2950, 2359, 2341, 1770, 1732, 1520, 1348, 1196, 1169, 1030, 858, 765, 698  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{30}\text{H}_{24}\text{N}_2\text{O}_8+\text{H})^+$  requires  $m/z$  541.1611, found  $m/z$  541.1614.

**1-(4-Chlorophenyl)-1-(methoxycarbonyl)-3-(4-nitrophenyl)-2,3-dihydro-1H-benzo[f]isoindole-4,9-diyl diacetate (5t):** Yield: 80%;  $[\alpha]_D^{20} = +108.2$  (c 0.9,  $\text{CHCl}_3$ );  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.91 (s, 3H), 2.26 (s, 3H), 3.50 (s, 1H), 3.76 (s, 3H), 5.50 (s, 1H), 7.28-7.34 (m, 4H), 7.47 (d,  $J = 8.8$

Hz, 2H), 7.54-7.61 (m, 2H), 7.71-7.79 (m, 2H), 8.22 (d,  $J = 8.8$  Hz, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 20.50, 20.58, 53.51, 64.95, 76.38, 121.75, 122.50, 124.01, 127.49, 127.68, 128.57, 128.74, 128.91, 128.97, 129.44, 132.26, 133.00, 134.12, 138.99, 139.36, 140.47, 147.79, 147.89, 167.50, 168.69, 172.07; Enantiomeric excess: 95%, determined by HPLC (Daicel Chirapak OD-H, hexane/isopropanol = 85/ 15, flow rate 1.0 mL/min,  $T = 30$  °C, 254 nm):  $t_{\text{R}} = 11.80$  min(minor),  $t_{\text{R}} = 19.34$  min(major); IR (KBr):  $\gamma$  3357, 3074, 2953, 2856, 2360, 2342, 1770, 1734, 1521, 1490, 1348, 1195, 1168, 1091, 1029, 1013, 829, 769,  $591\text{ cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{30}\text{H}_{23}\text{ClN}_2\text{O}_8+\text{H})^+$  requires  $m/z$  575.1221, found  $m/z$  575.1224.

**1-(2-Chlorophenyl)-1-(methoxycarbonyl)-3-(4-nitrophenyl)-2,3-dihydro-1H-benzo[f]isoindole-4,9-diyl diacetate (5u):** Yield: 79%;  $[\alpha]_{\text{D}}^{20} = +127.3$  (c 0.9,  $\text{CHCl}_3$ );  $^1\text{H}$ -NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.90 (s, 3H), 2.28 (s, 3H), 3.81 (s, 3H), 5.43 (s, 1H), 6.94 (d,  $J = 8.0$  Hz, 1H), 7.11-7.16 (m, 1H), 7.26-7.30 (m, 1H), 7.47 (d,  $J = 8.0$  Hz, 1H), 7.57-7.62 (m, 4H), 7.74-7.79 (m, 2H), 8.23 (d,  $J = 8.8$  Hz, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 20.10, 20.66, 53.82, 64.87, 77.30, 121.73, 122.48, 123.92, 126.70, 127.57, 127.88, 128.59, 128.98, 129.11, 129.45, 129.98, 131.03, 133.51, 133.98, 139.57, 141.02, 147.92, 148.34, 167.48, 172.01; Enantiomeric excess: 85%, determined by HPLC (Daicel Chirapak OD-H, hexane/ isopropanol = 90/ 10, flow rate 1.0 mL/min,  $T = 30$  °C, 254 nm):  $t_{\text{R}} = 13.82$  min(minor),  $t_{\text{R}} = 17.58$  min(major); IR (KBr):  $\gamma$  3353, 2972, 2922, 2355, 2330, 1770, 1732, 1520, 1348, 1195, 1169, 1090, 1049, 881,  $760\text{ cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{30}\text{H}_{23}\text{ClN}_2\text{O}_8+\text{H})^+$  requires  $m/z$  575.1221, found  $m/z$  575.1223.

**1-(4-Chlorophenyl)-1-(methoxycarbonyl)-3-(4-nitrophenyl)isoindoline-4,7-diyl diacetate (5v):** Yield: 76%; m.p. 177-179 °C;  $[\alpha]_{\text{D}}^{20} = -21.3$  (c 0.8,  $\text{CHCl}_3$ );  $^1\text{H}$ -NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  (ppm): 1.81 (s, 3H), 2.06 (s, 3H), 3.34 (s, 1H), 3.76 (s, 3H), 5.57 (s, 1H), 7.13 (d,  $J = 8.7$  Hz, 1H), 7.18 (d,  $J = 8.7$  Hz, 1H), 7.24 (d,  $J = 8.8$  Hz, 2H), 7.31 (d,  $J = 8.8$  Hz, 2H), 7.39 (d,  $J = 8.8$  Hz, 2H), 8.19 (d,  $J = 8.8$  Hz, 2H);  $^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  (ppm): 20.27, 20.71, 53.35, 65.27, 76.69, 124.00, 124.24, 124.94, 128.45, 128.59, 128.97, 134.08, 135.26, 136.53, 139.61, 143.30, 143.99, 147.80, 148.40, 167.83, 168.49, 172.48; Enantiomeric excess: 86%, determined by HPLC (Daicel Chirapak OD-H, hexane/ isopropanol = 70/ 30, flow rate 1.0 mL/min,  $T = 25$  °C, 254 nm):  $t_{\text{R}} = 9.04$  min(minor),  $t_{\text{R}} = 11.67$  min(major); IR (KBr):  $\gamma$  3436, 2922, 2852, 1766, 1738, 1520, 1485, 1348, 1178, 1014, 872, 593,  $567\text{ cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{26}\text{H}_{21}\text{ClN}_2\text{O}_8+\text{H})^+$  requires  $m/z$  525.1065, found  $m/z$  525.1072; Anal. Calcd for  $\text{C}_{26}\text{H}_{21}\text{ClN}_2\text{O}_8$ : C, 59.49; H, 4.03; N, 5.34. Found: C, 59.51; H, 4.19; N,

4.93.

**Diethyl 3-(4-chlorophenyl)-4,7-bis(trifluoromethylsulfonyloxy)isoindoline-1,1-dicarboxylate (7):** **4p** (partially isomerized) was transferred to a test tube. The atmosphere was replaced with argon and pyridine (2 mL) was added. The test tube was placed in an ice-water bath, and then Tf<sub>2</sub>O (0.30 mL) was added. The reaction mixture was allowed to warm up to room temperature and stirred overnight. The reaction was quenched with water (10 mL) and the aqueous layer was extracted with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layers were washed with 1 N HCl and saturated NaCl aqueous solution. After removal of the solvent, the residue was purified through column chromatography on a silica gel (eluent: petroleum ether/ethyl acetate = 25/1) to give pure product **7** (97.6 mg, 73%); [ $\alpha$ ]<sub>D</sub><sup>20</sup> = -32.0 (c 1.6, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 1.28-1.33 (m, 6H), 3.93 (d, *J* = 5.3 Hz, 1H), 4.24-4.43 (m, 4H), 5.77 (d, *J* = 5.3 Hz, 1H), 7.21 (d, *J* = 6.6 Hz, 2H), 7.29-7.34 (m, 3H), 7.42 (d, *J* = 9.1 Hz, 1H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 13.80, 13.83, 63.22, 63.56, 65.58, 76.88, 118.35 (q, *J*<sub>C-F</sub> = 319 Hz), 118.40 (q, *J*<sub>C-F</sub> = 318 Hz), 122.24, 124.41, 128.99, 129.67, 132.70, 134.60, 138.71, 140.28, 142.91, 145.01, 167.93, 168.65; Enantiomeric excess: 93%, determined by HPLC (Daicel Chirapak AD-H, hexane/ isopropanol = 90/ 10, flow rate 1.0 mL/min, T = 25 °C, 254 nm): t<sub>R</sub> = 6.42 min(minor), t<sub>R</sub> = 8.80 min(major); IR (KBr):  $\nu$  3427, 2922, 2852, 1740, 1637, 1478, 1430, 1222, 1140, 1091, 930, 880, 602 cm<sup>-1</sup>; HRMS: exact mass calcd for (C<sub>22</sub>H<sub>18</sub>ClF<sub>6</sub>NO<sub>10</sub>S<sub>2</sub>+H)<sup>+</sup> requires m/z 670.0043, found m/z 670.0046.

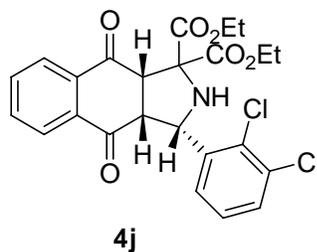
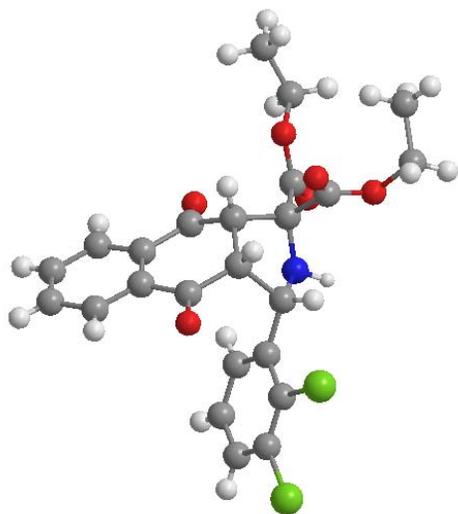
**Diethyl 3-(4-chlorophenyl)isoindoline-1,1-dicarboxylate (8):** To a dry test tube was added **7** (82.9 mg, 0.12 mmol), Pd(OAc)<sub>2</sub> (9.3 mg, 0.024 mmol) and dppp (2.7 mg, 0.012 mmol). The atmosphere was replaced with argon and DMF (1.0 ml) was added. The mixture was stirred and was added with Et<sub>3</sub>N (167  $\mu$ L, 1.2 mmol) and HCOOH (36  $\mu$ L, 0.96 mmol). The reaction was heated to 90 °C for 12 hours. Then water (10 mL) was added to the reaction and extracted with ethyl acetate. The organic phase was combined and dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, the residue was purified through column chromatography on a silica gel (eluent: petroleum ether/ethyl acetate = 20/1) to give **8** (32.1 mg, 69%). [ $\alpha$ ]<sub>D</sub><sup>20</sup> = -113.3 (c 0.4, CHCl<sub>3</sub>); <sup>1</sup>H-NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  (ppm): 1.26-1.33 (m, 6H), 3.72 (s, 1H), 4.23-4.35 (m, 4H), 5.62 (s, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 7.28-7.33 (m, 6H), 7.64 (d, *J* = 7.3 Hz, 1H); <sup>13</sup>C-NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  (ppm): 14.11, 14.13, 62.14, 62.42, 66.12, 76.91, 123.29, 125.63, 127.73, 128.75, 129.23, 129.37, 133.48, 135.95, 142.23, 144.35, 169.83, 170.72; Enantiomeric excess: 93%, determined by HPLC (Daicel Chirapak AS-H, hexane/ isopropanol = 90/ 10,

flow rate 1.0 mL/min, T = 25 °C, 254 nm):  $t_R = 7.43$  min(minor),  $t_R = 15.26$  min(major); IR (KBr):  $\gamma$  3438, 2981, 2923, 2852, 1736, 1459, 1258, 1224, 1123, 1088, 881, 748, 643, 603  $\text{cm}^{-1}$ ; HRMS: exact mass calcd for  $(\text{C}_{20}\text{H}_{20}\text{ClNO}_4+\text{H})^+$  requires m/z 374.1159, found m/z 374.1153.

**Reference:**

- [1]. Ficht, S.; Mulbaier, M.; Giannis, A. *Tetrahedron* **2001**, *57*, 4863-4866; Yu, D.; Mattern, D. L. *Syn. Commun.* **1999**, *29*, 821-825.
- [2]. Itoh, K.; Fuchibe, K.; Akiyama, T. *Angew. Chem. Int. Ed.* **2006**, *45*, 4796–4798.

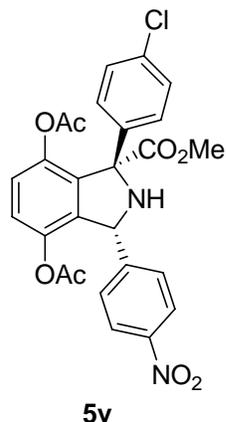
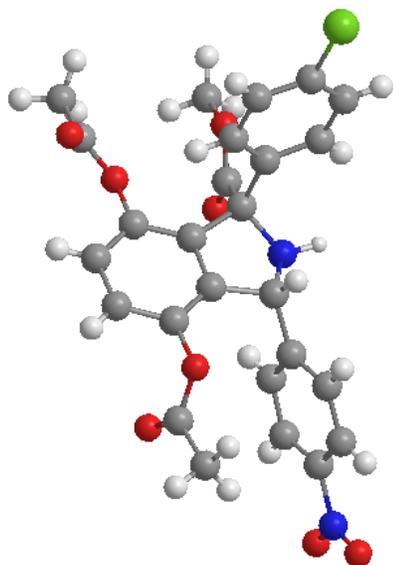
**X-ray single crystal data for 4j (CCDC 704878)**



Chemical formula	C <sub>24</sub> H <sub>21</sub> Cl <sub>2</sub> N O <sub>6</sub>
Formula weight	490.32
Space group	P 21
Z	2
$\alpha$ , Å	8.314(5)
b, Å	22.630(5)
c, Å	12.582(5)
$\alpha$ , °	90
$\beta$ , °	94.055(5)
$\gamma$ , °	90
V, Å <sup>3</sup>	2361.3(18)
T, K	293
$\rho$ , g/cm <sup>3</sup>	1.379

Reflections collected / unique: 29413 / 7332 ( $R_{int} = 0.0201$ ), number of observations [ $I > 2 \sigma(I)$ ] 6900, parameters 603. Final  $R$  indices [ $I > 2 \sigma(I)$ ]:  $R_1=0.0571$ ,  $wR_2=0.1429$ ;  $R$  indices (all data):  $R_1=0.0594$ ,  $wR_2=0.1447$ .

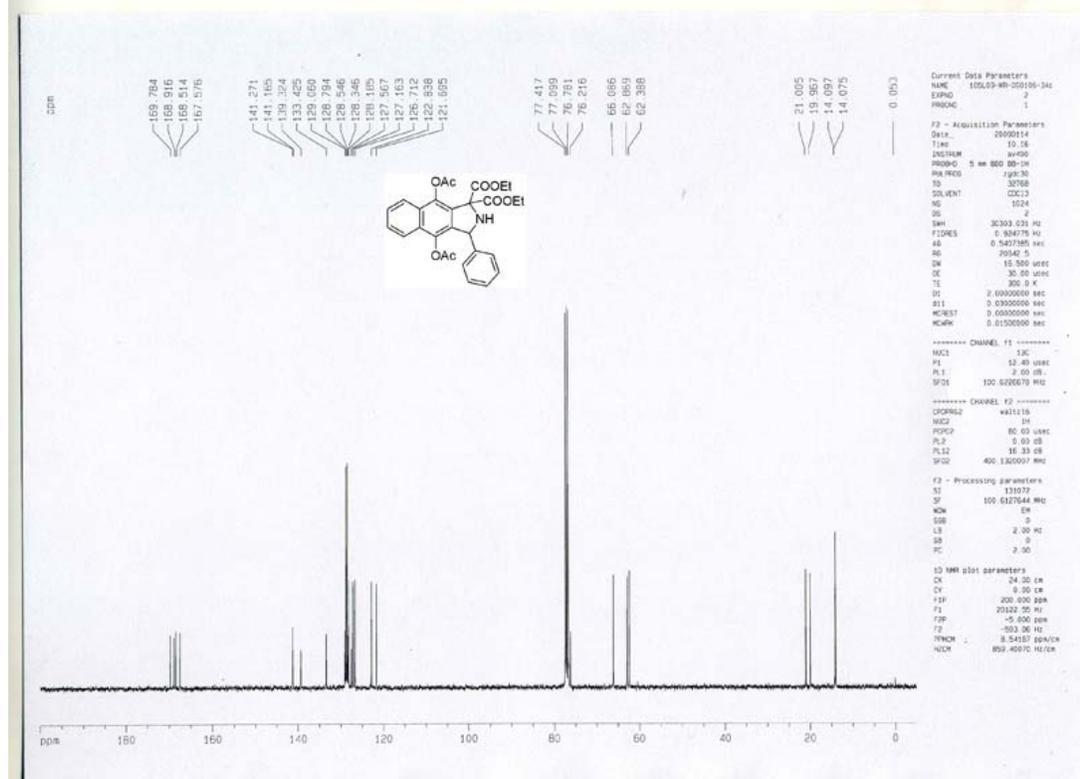
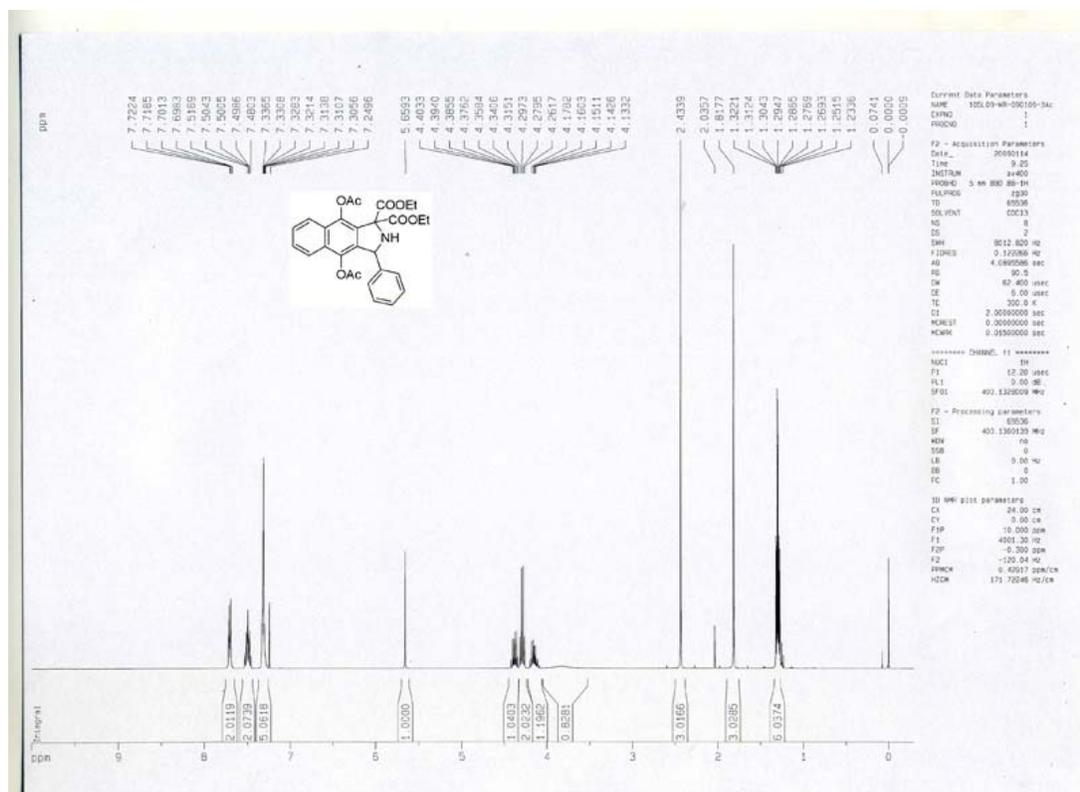
### X-ray single crystal data for 5v (CCDC 742913)

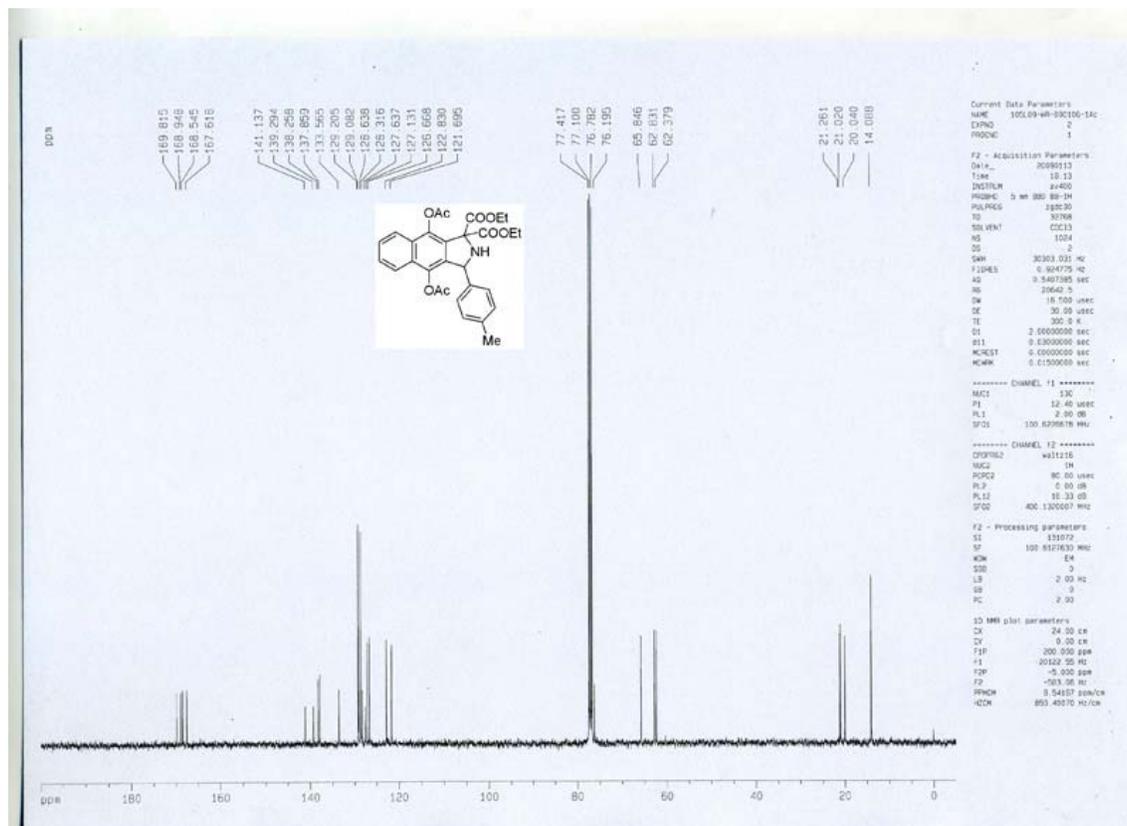
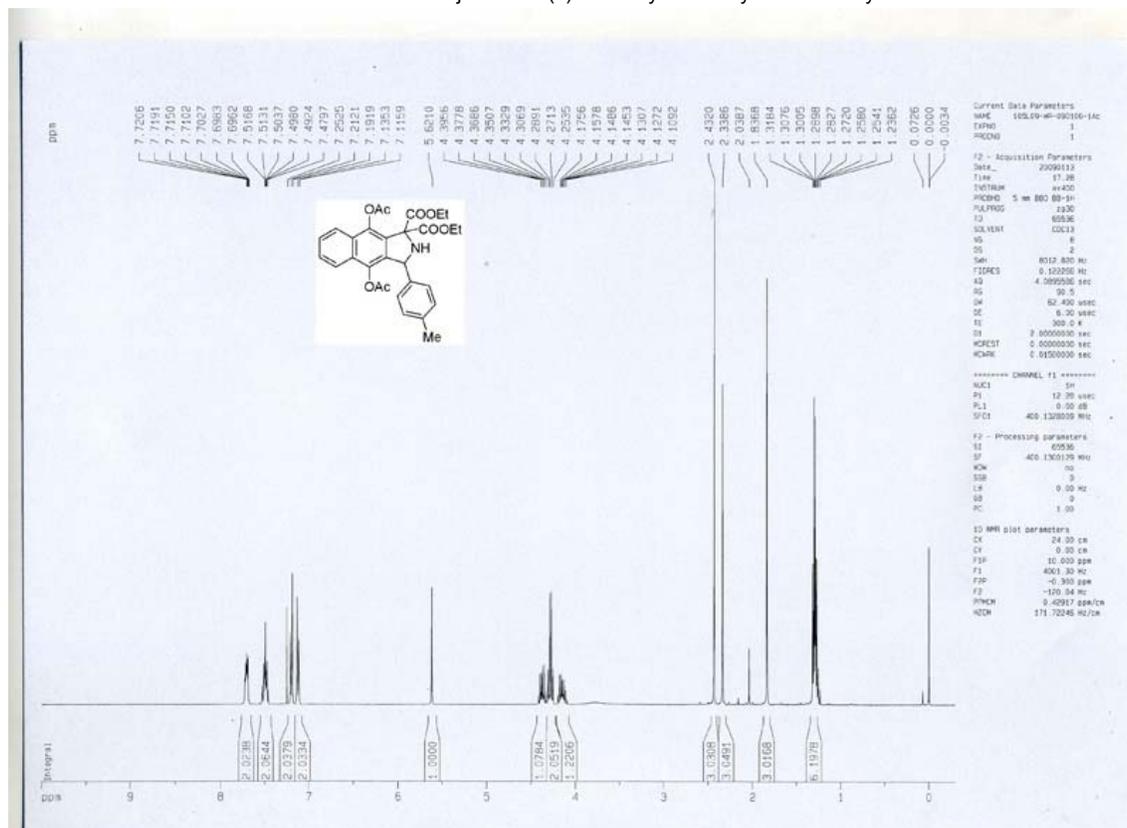


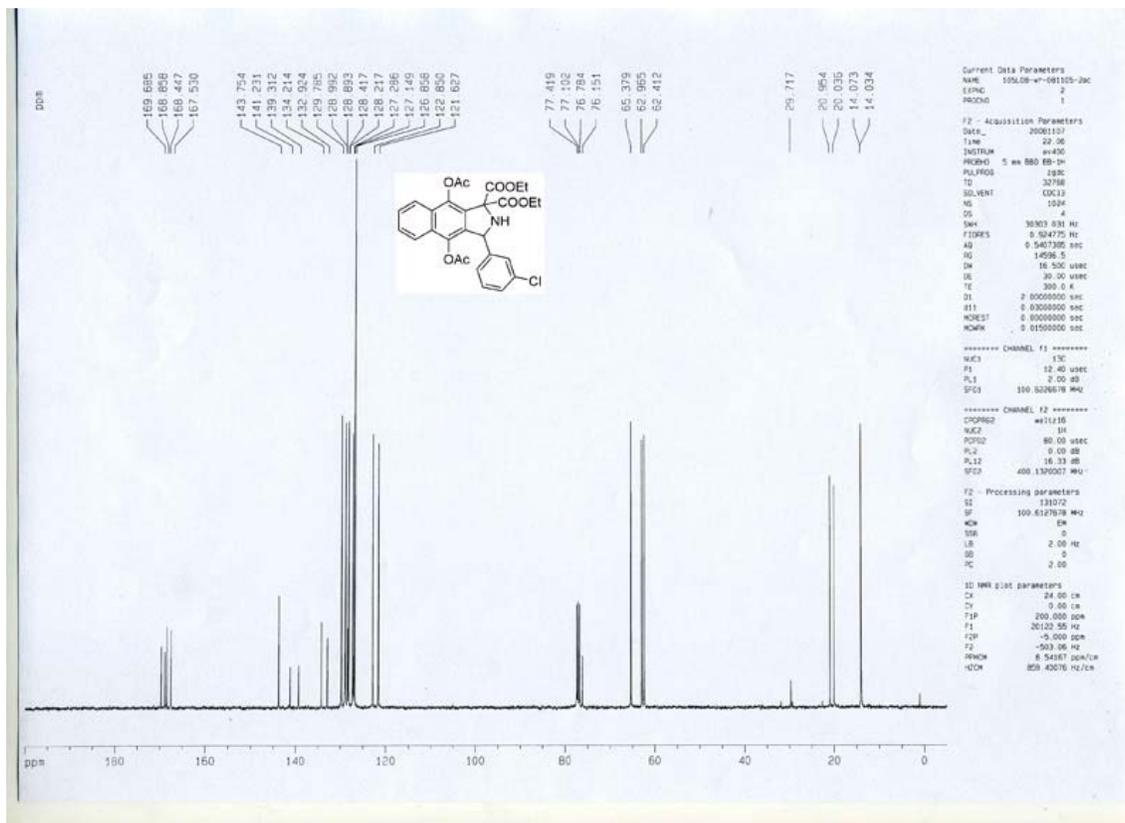
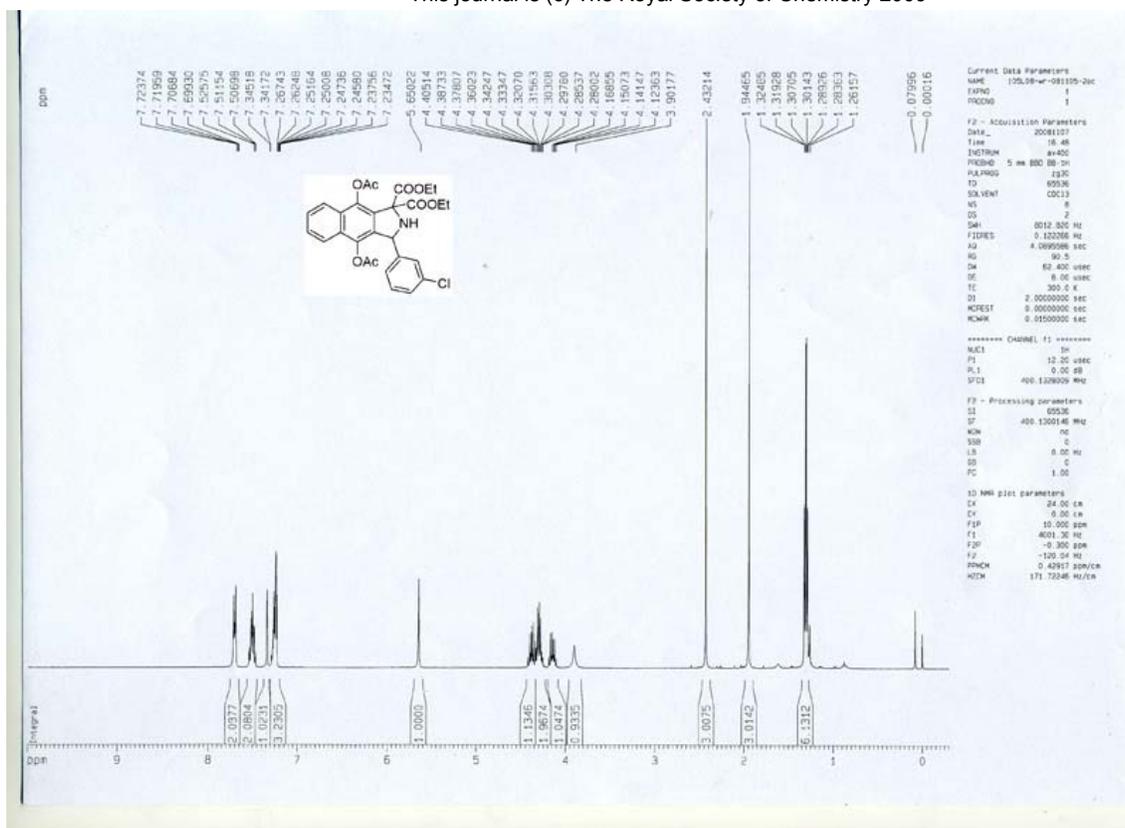
Chemical formula	C <sub>26</sub> H <sub>21</sub> Cl N <sub>2</sub> O <sub>8</sub>
Formula weight	524.90
Space group	P 21
Z	2
$\alpha$ , Å	7.965(5)
b, Å	30.381(5)
c, Å	10.583(5)
$\alpha$ , °	90
$\beta$ , °	101.631(5)
$\gamma$ , °	90
V, Å <sup>3</sup>	2508(2)
T, K	295
$\rho$ , g/cm <sup>3</sup>	1.390

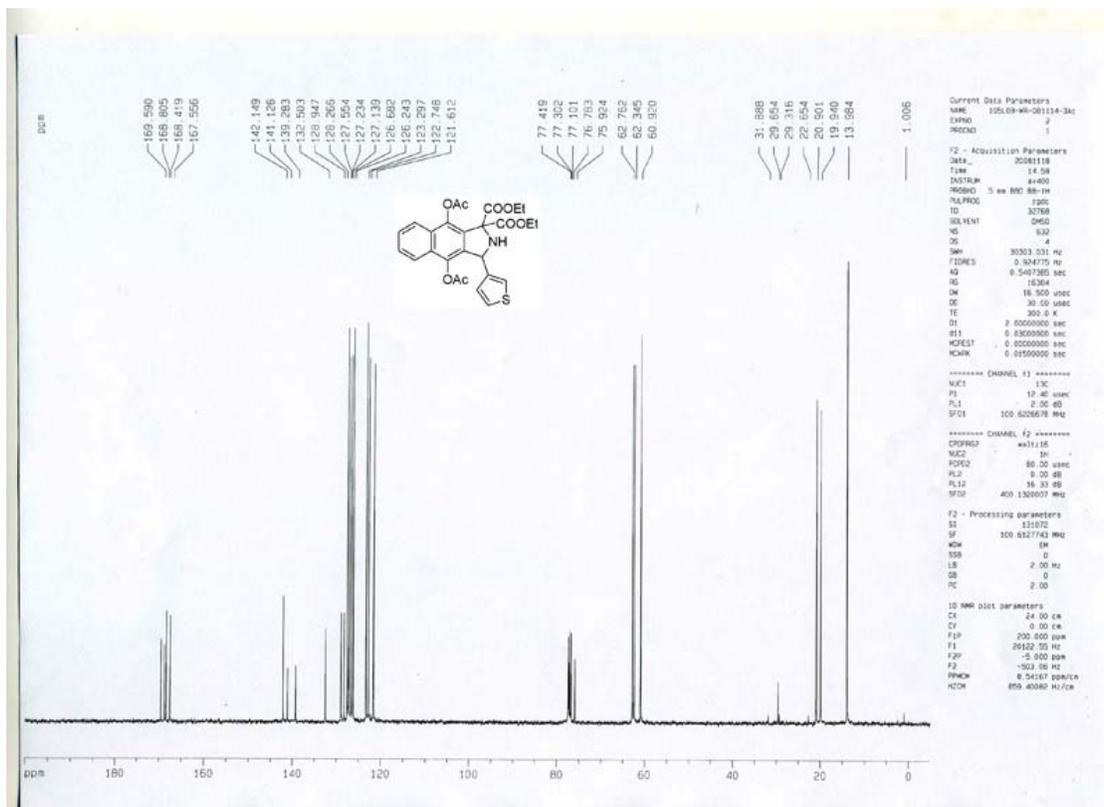
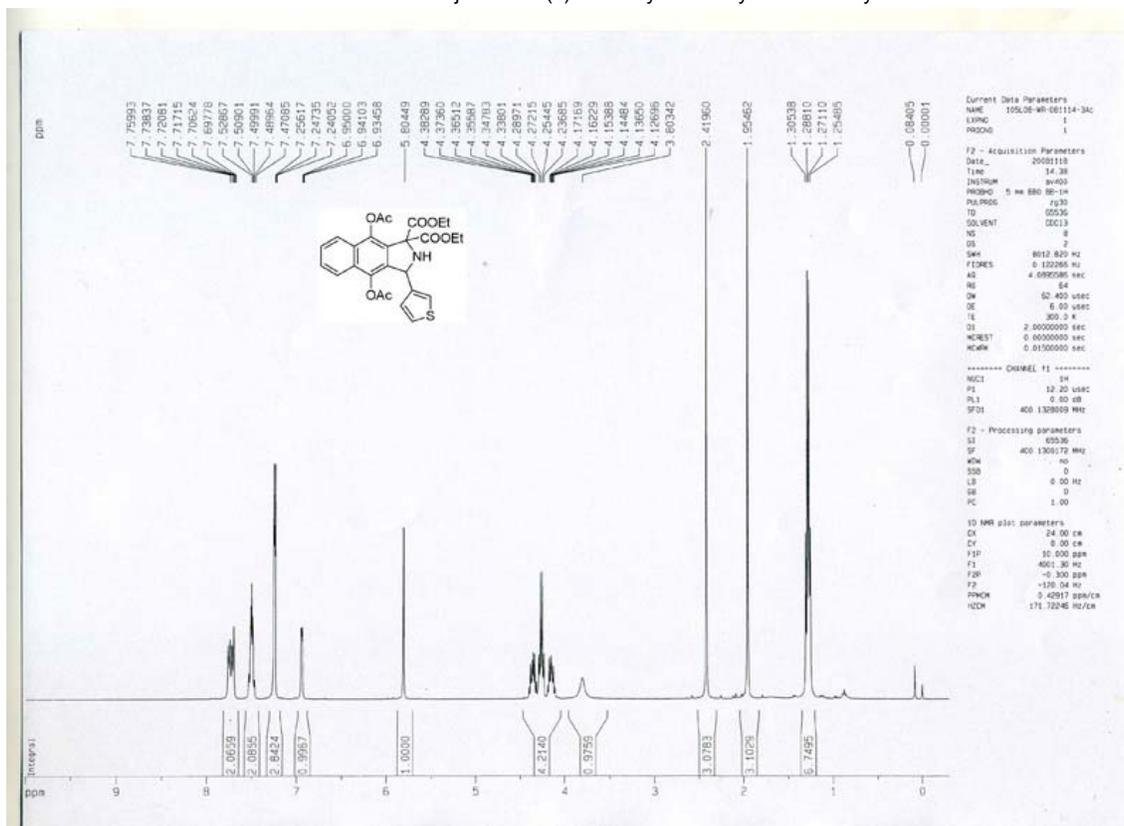
Reflections collected / unique: 14302 / 7313 ( $R_{int} = 0.0289$ ), number of observations [ $I > 2 \sigma(I)$ ] 5182, parameters 676. Final  $R$  indices [ $I > 2 \sigma(I)$ ]:  $R_1=0.0425$ ,  $wR_2=0.0868$ ;  $R$  indices (all data):  $R_1=0.0651$ ,  $wR_2=0.0934$ .

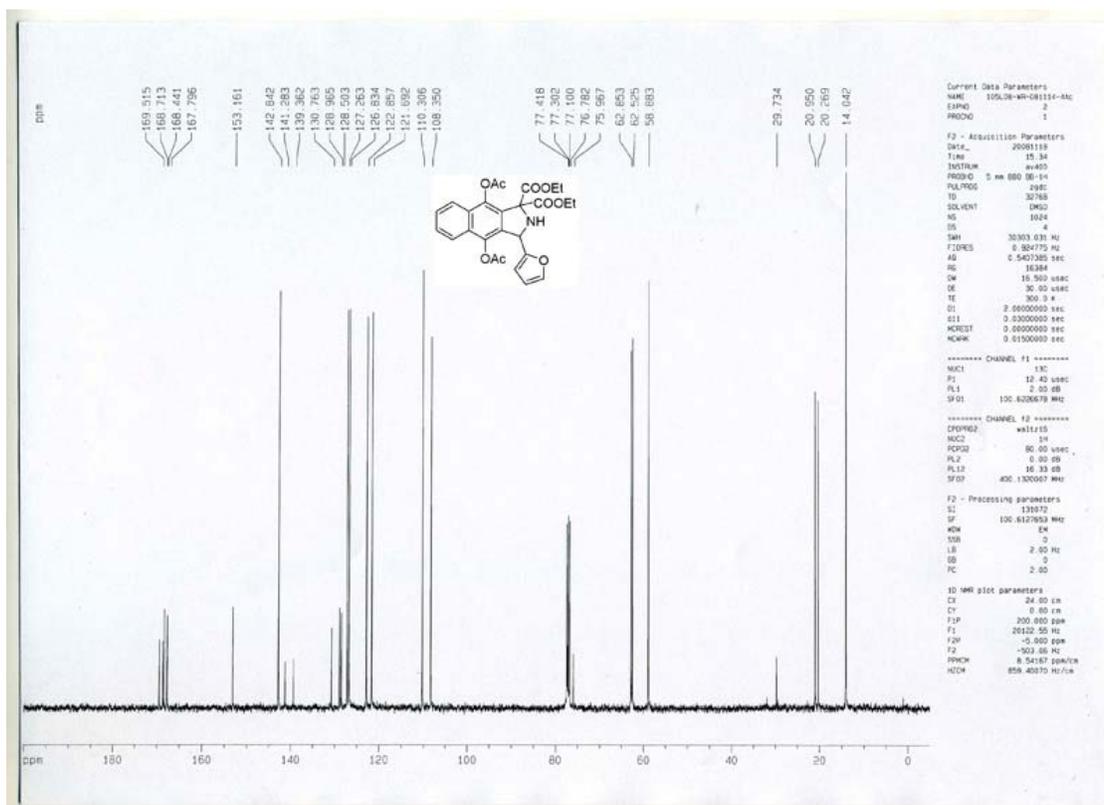
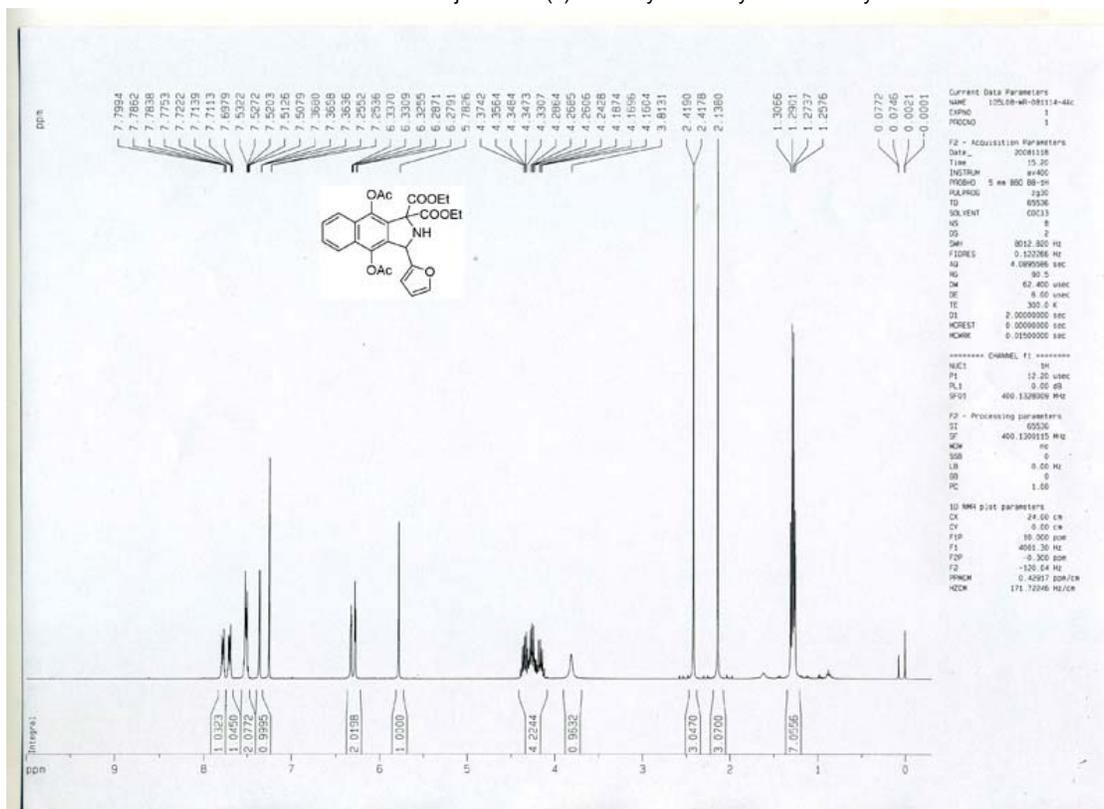
Selected NMR and HPLC

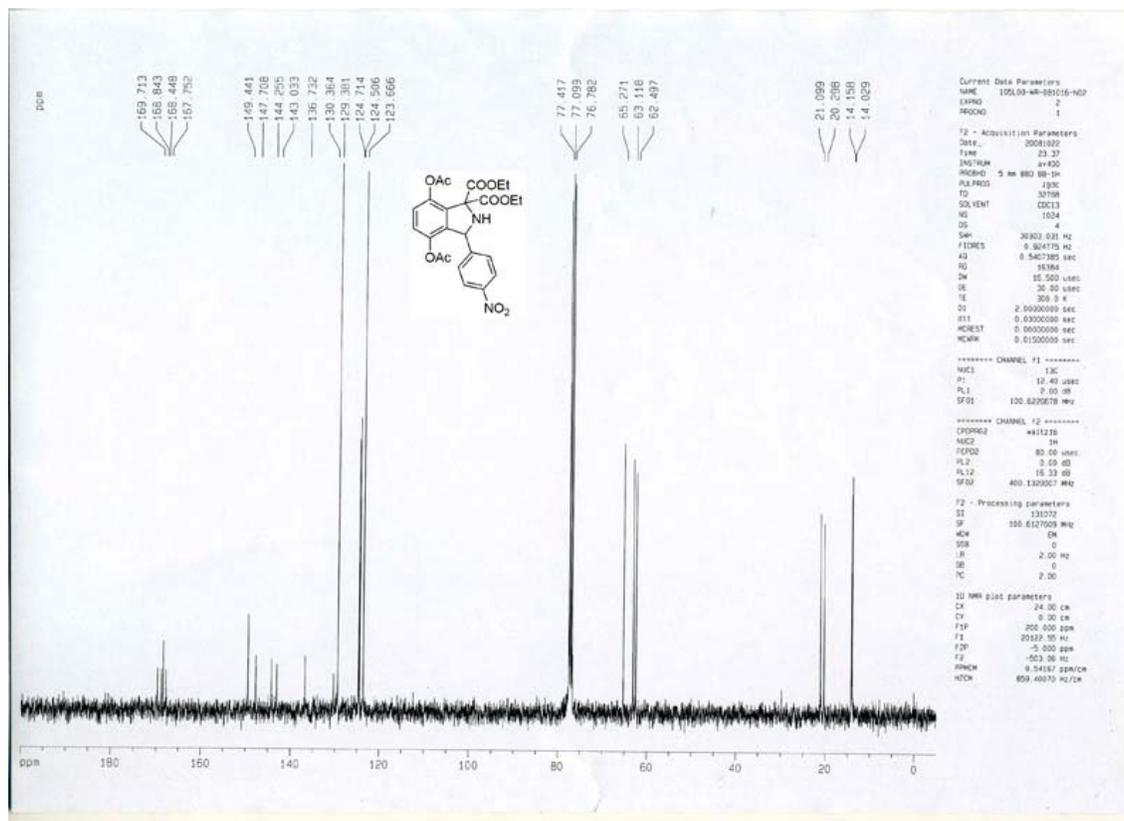
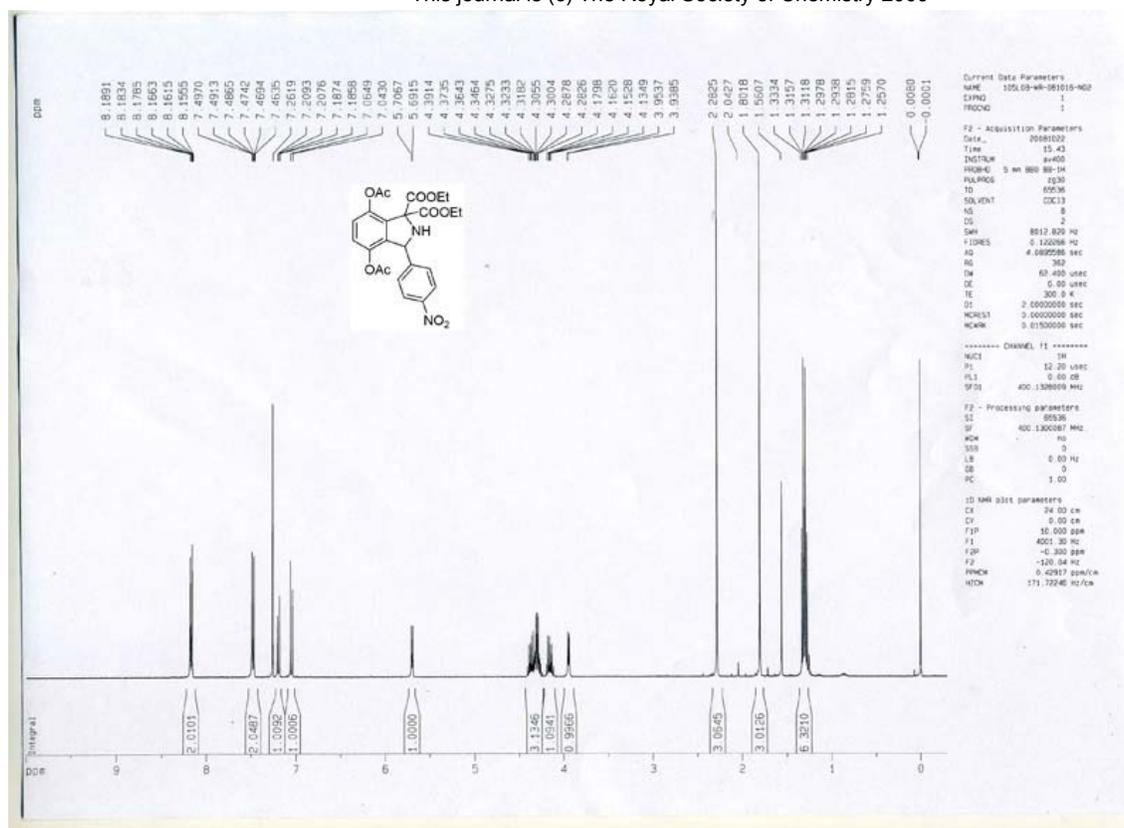


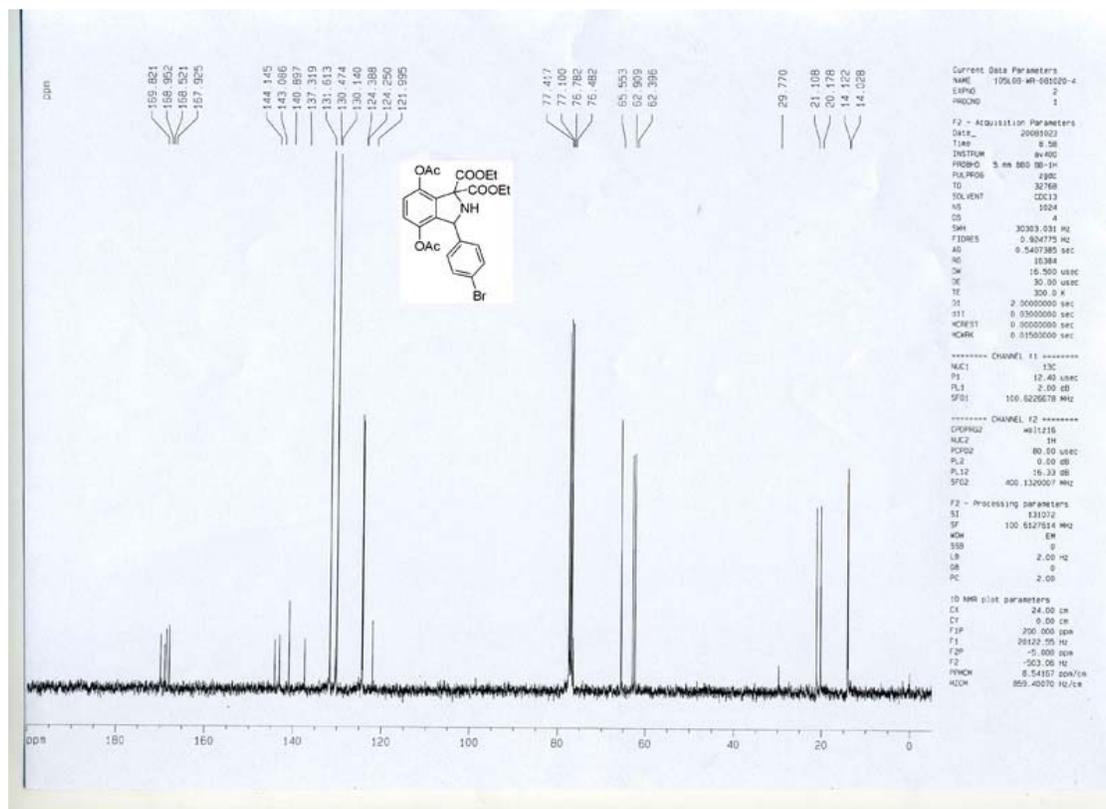
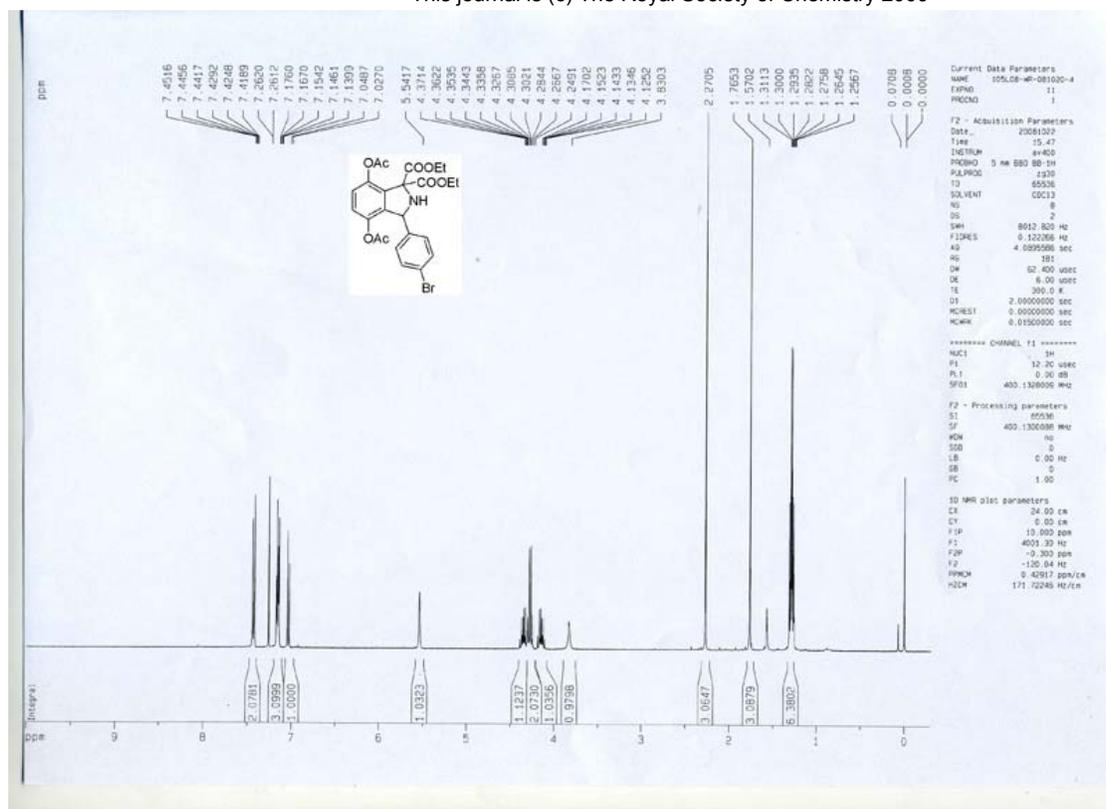


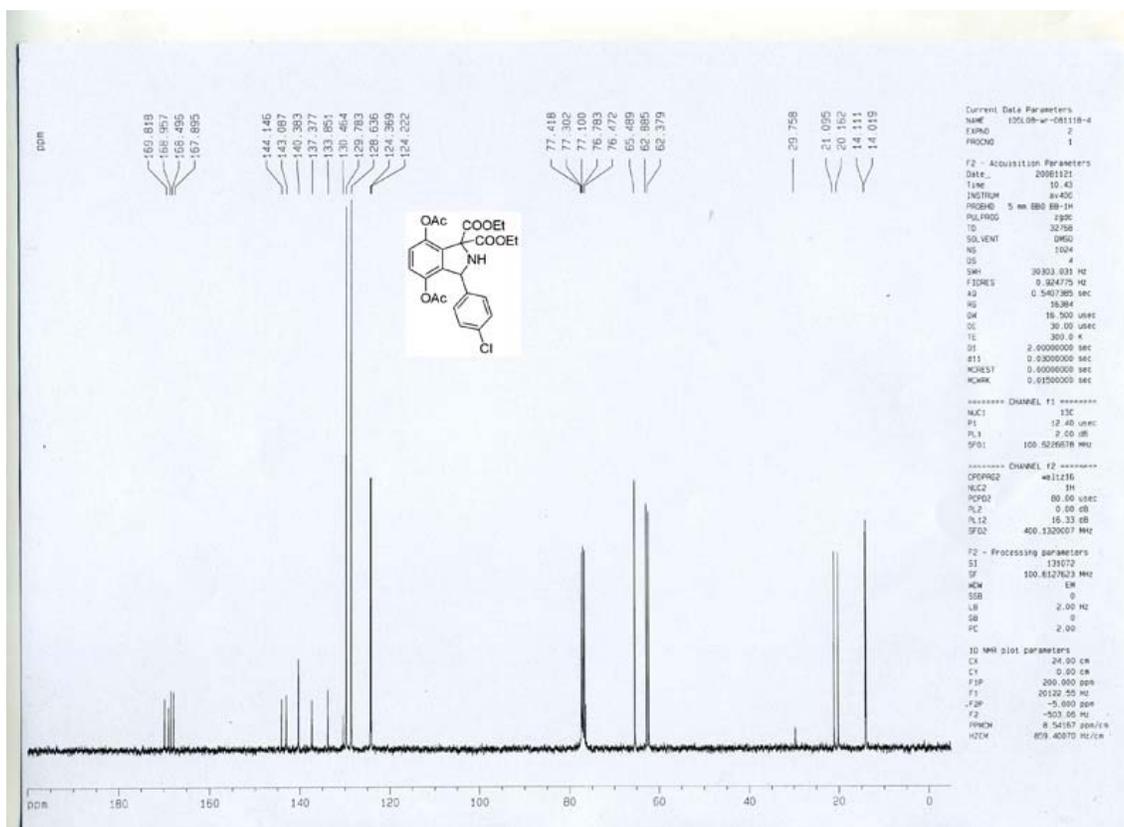
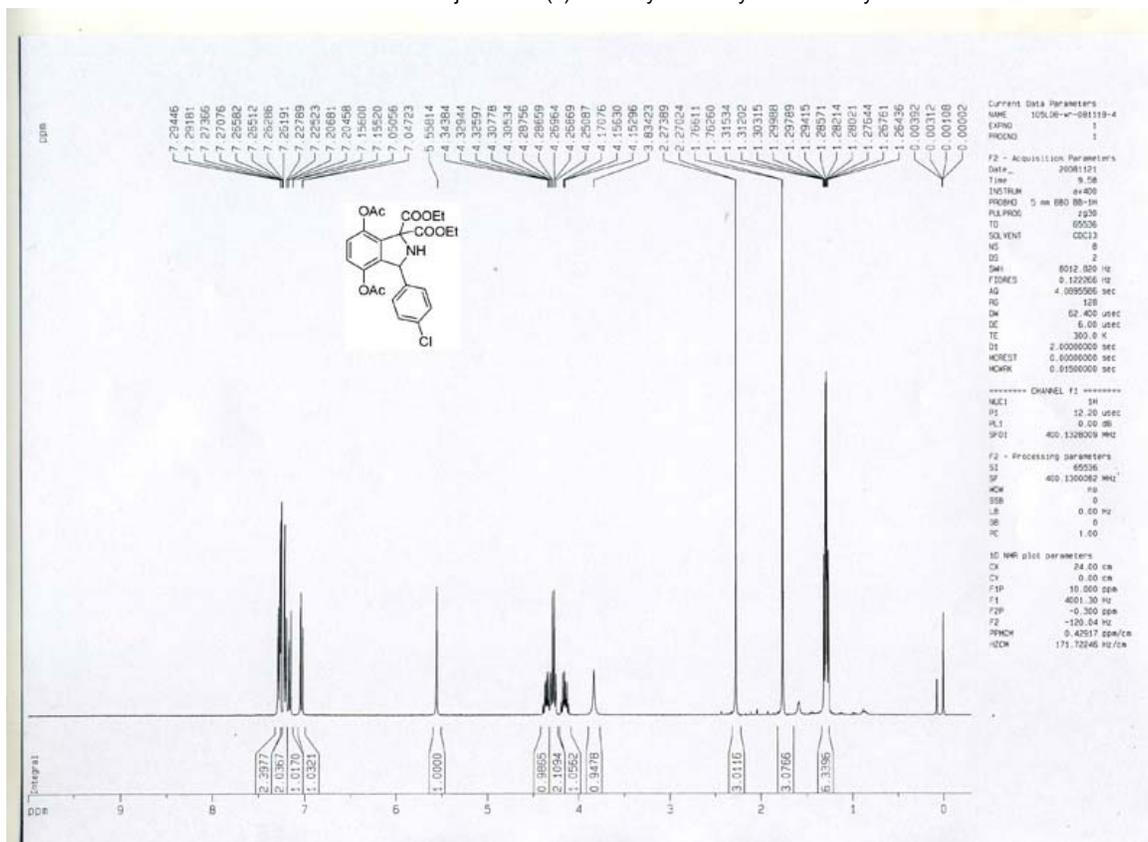


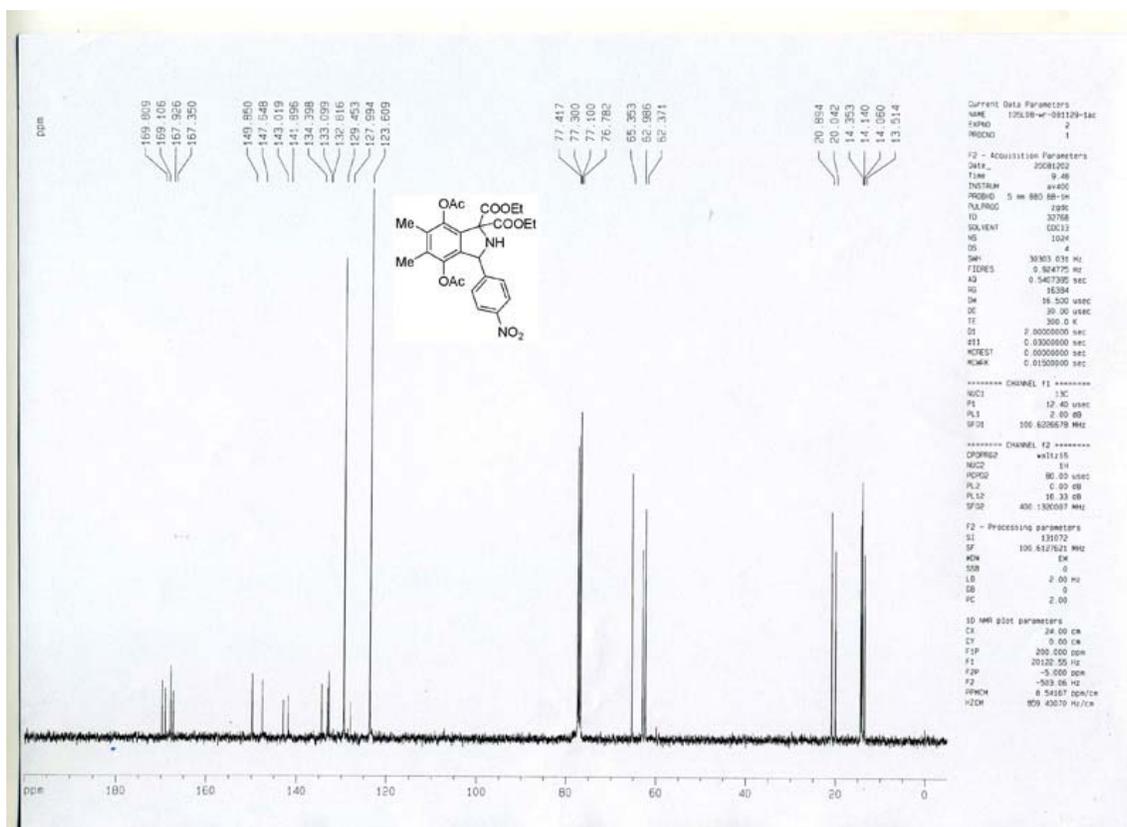
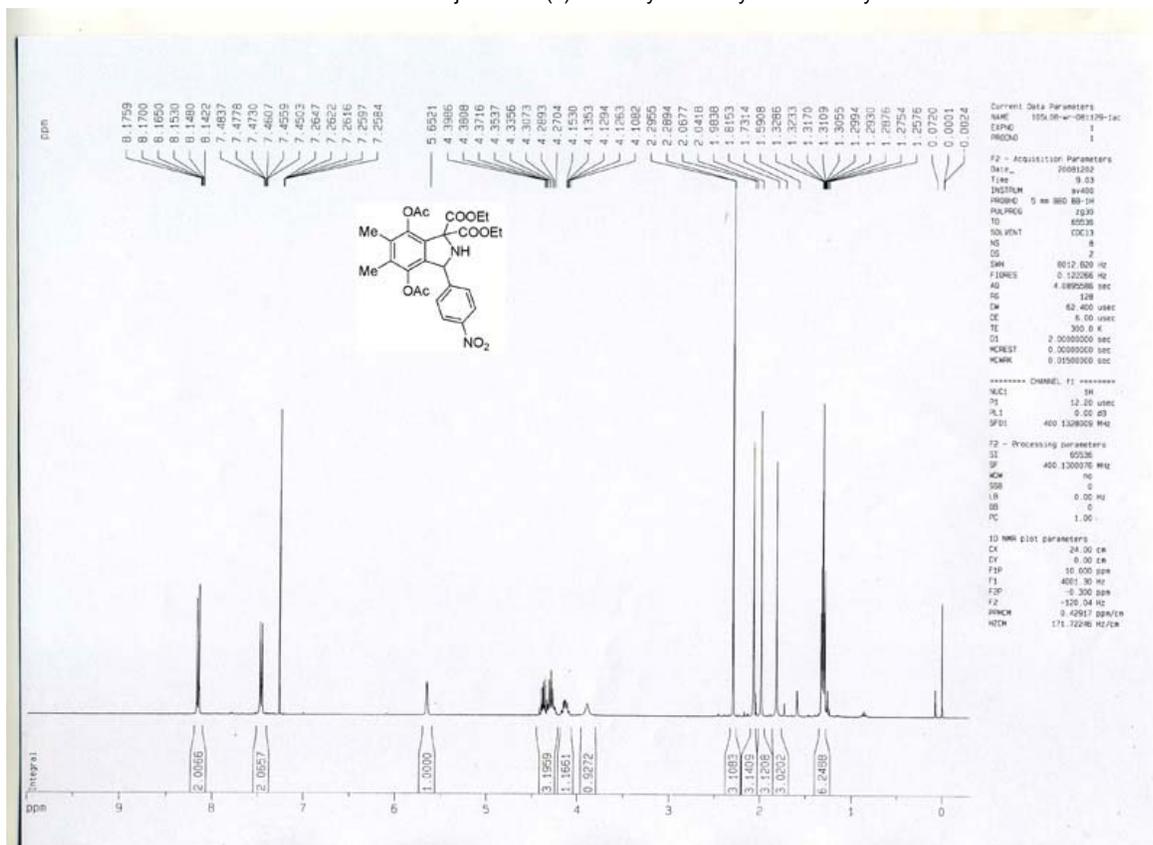


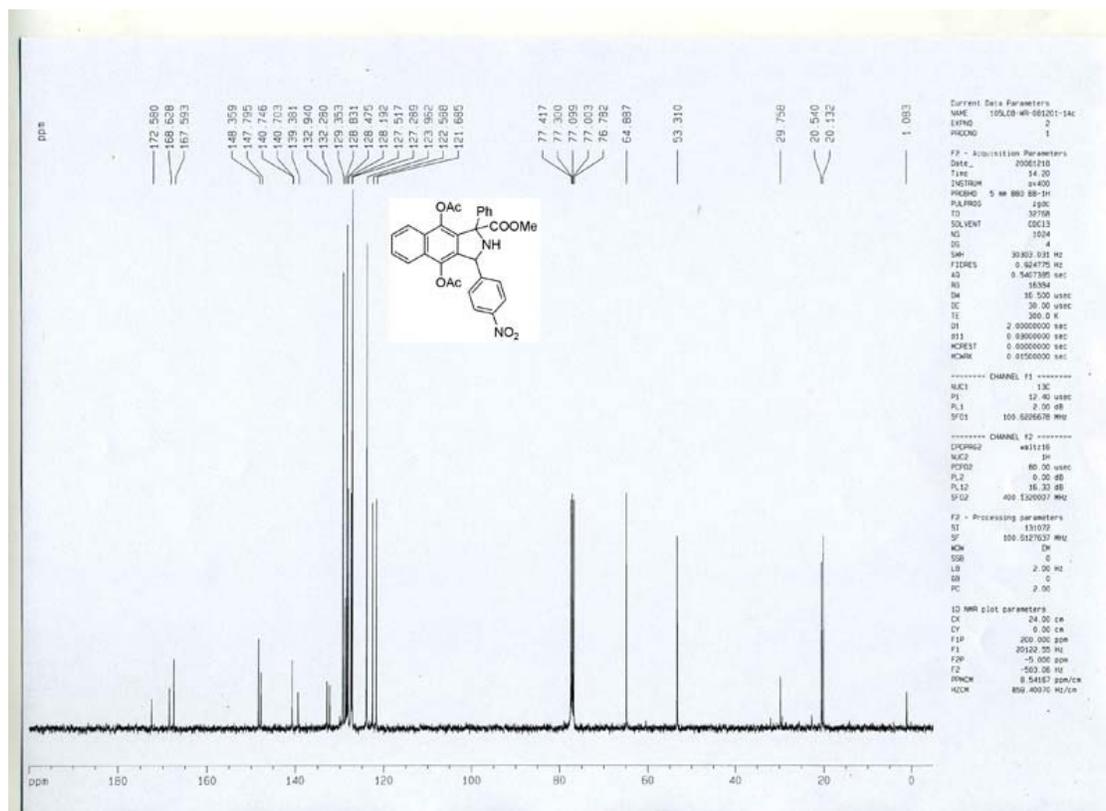
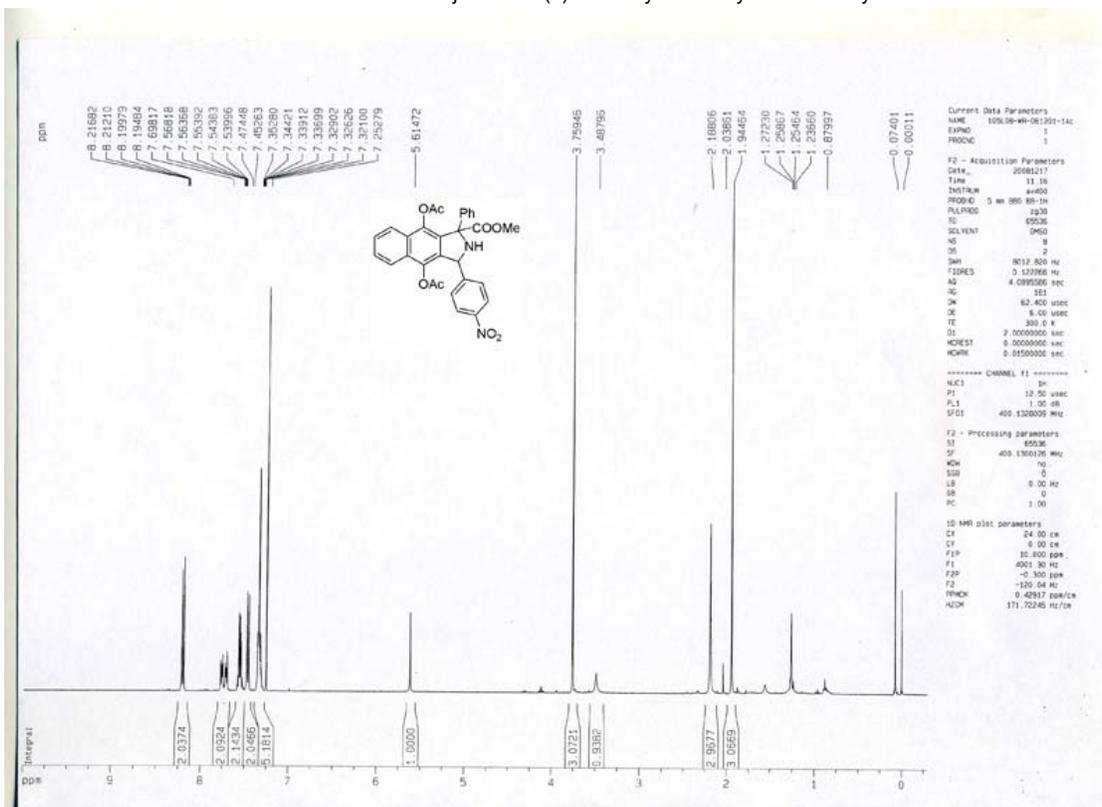


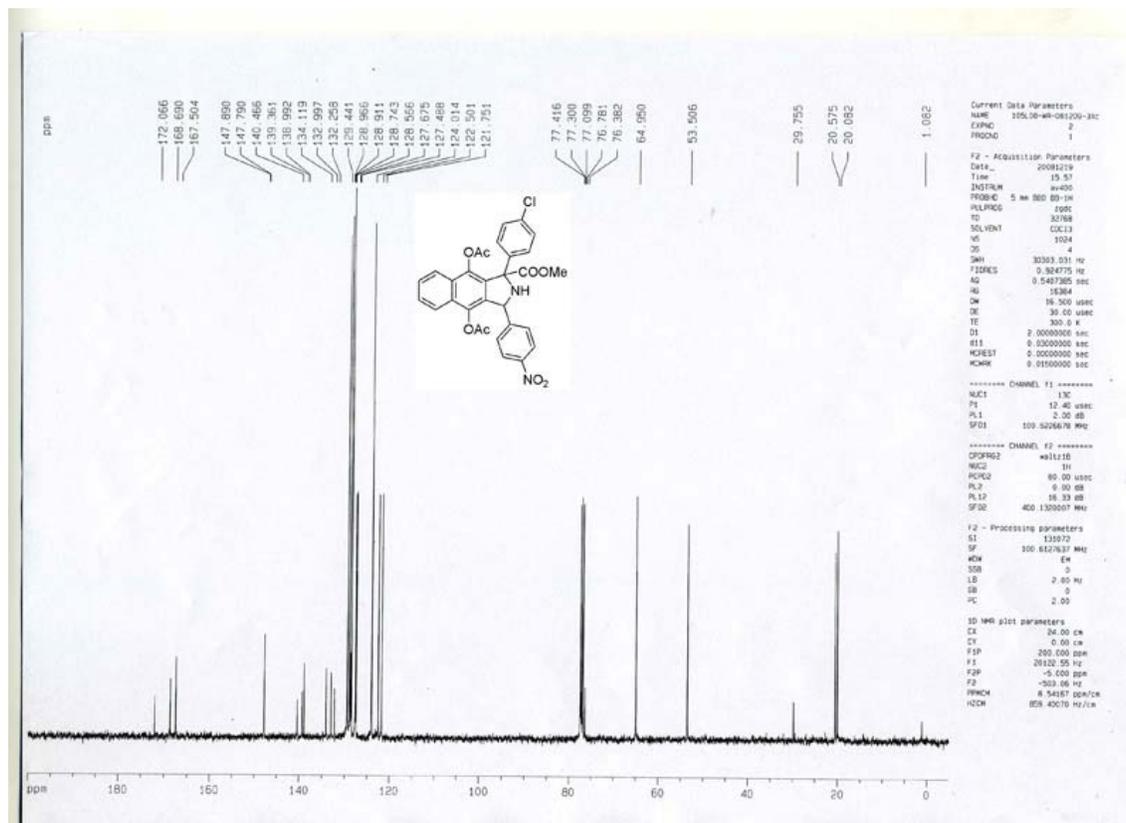
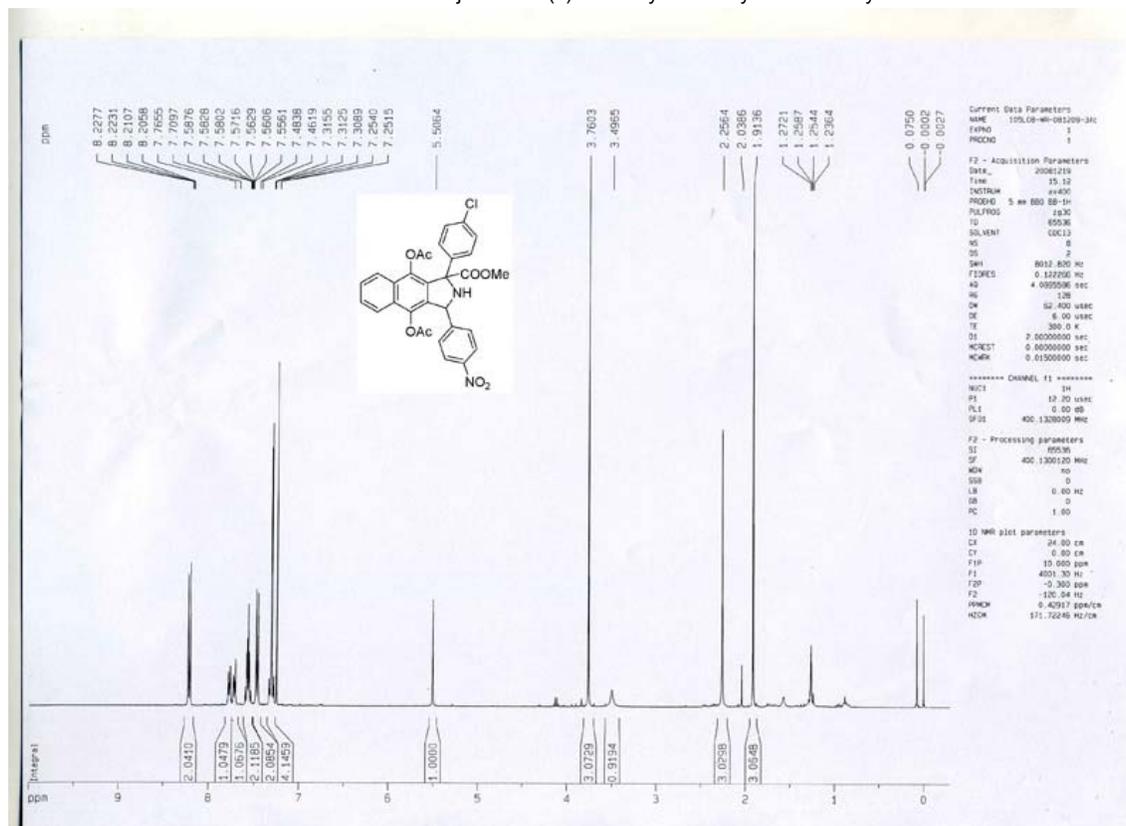


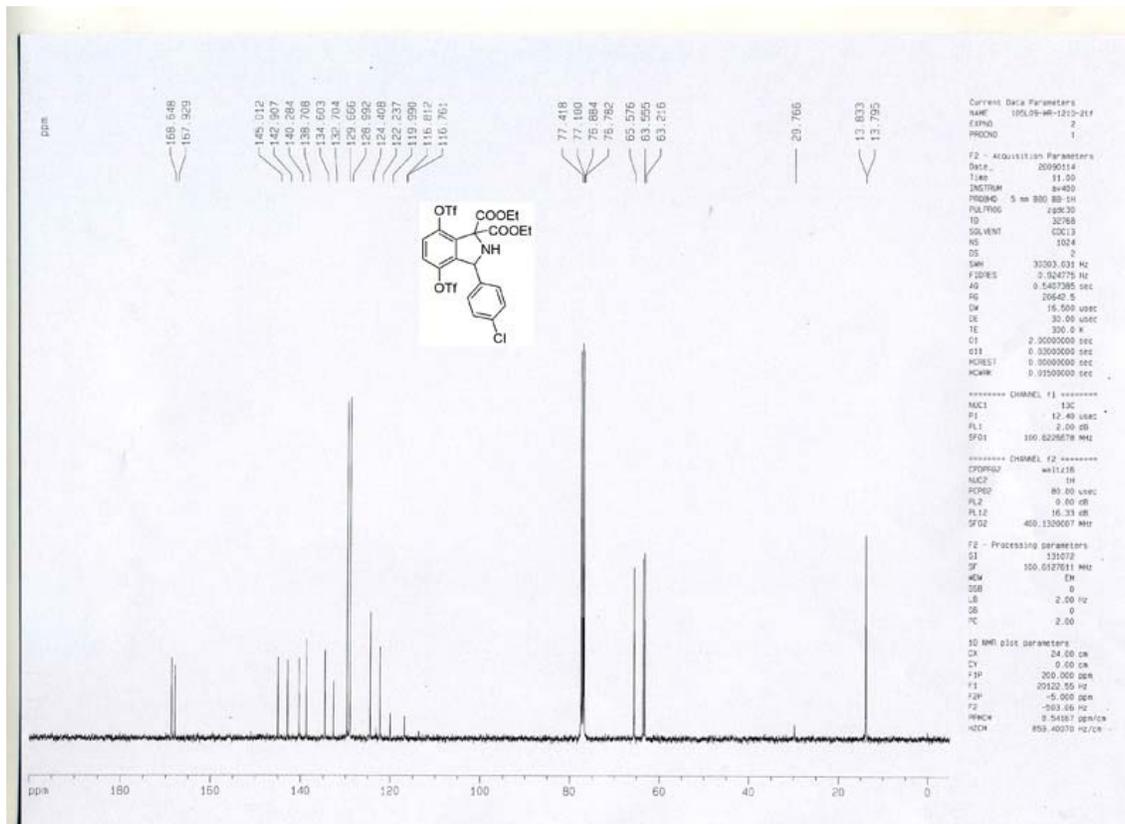
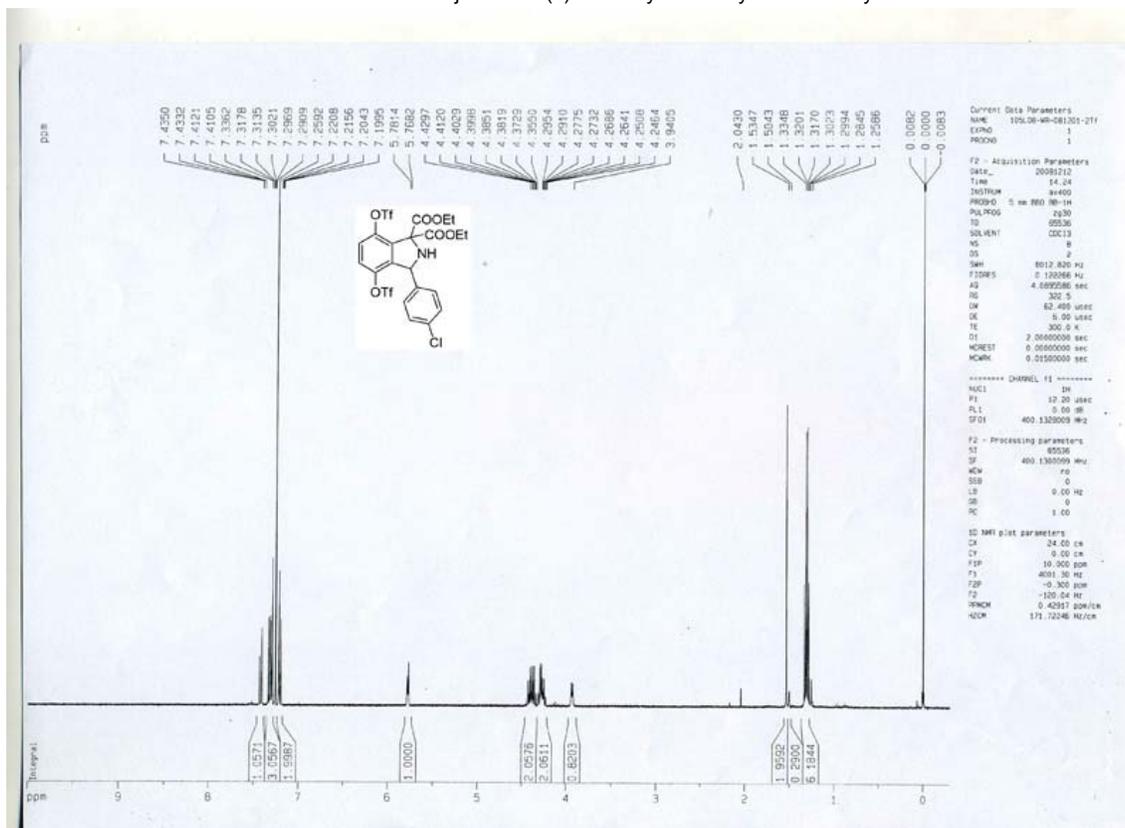


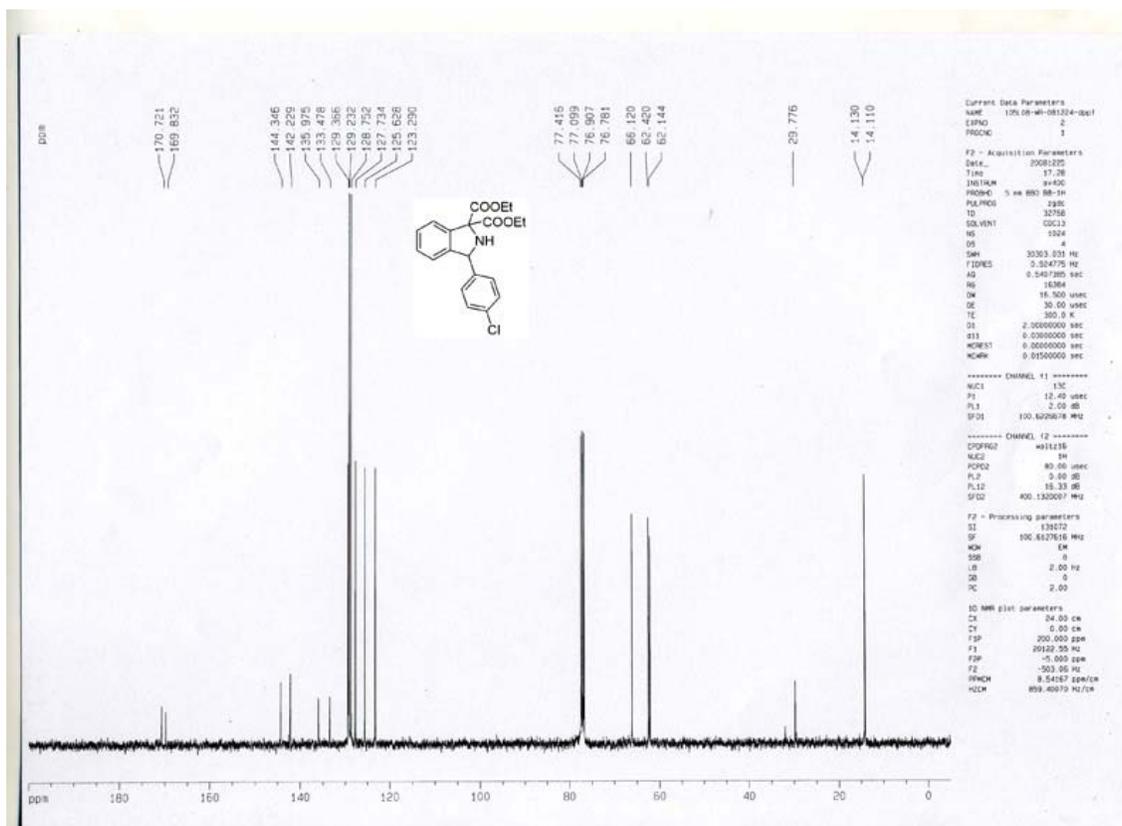
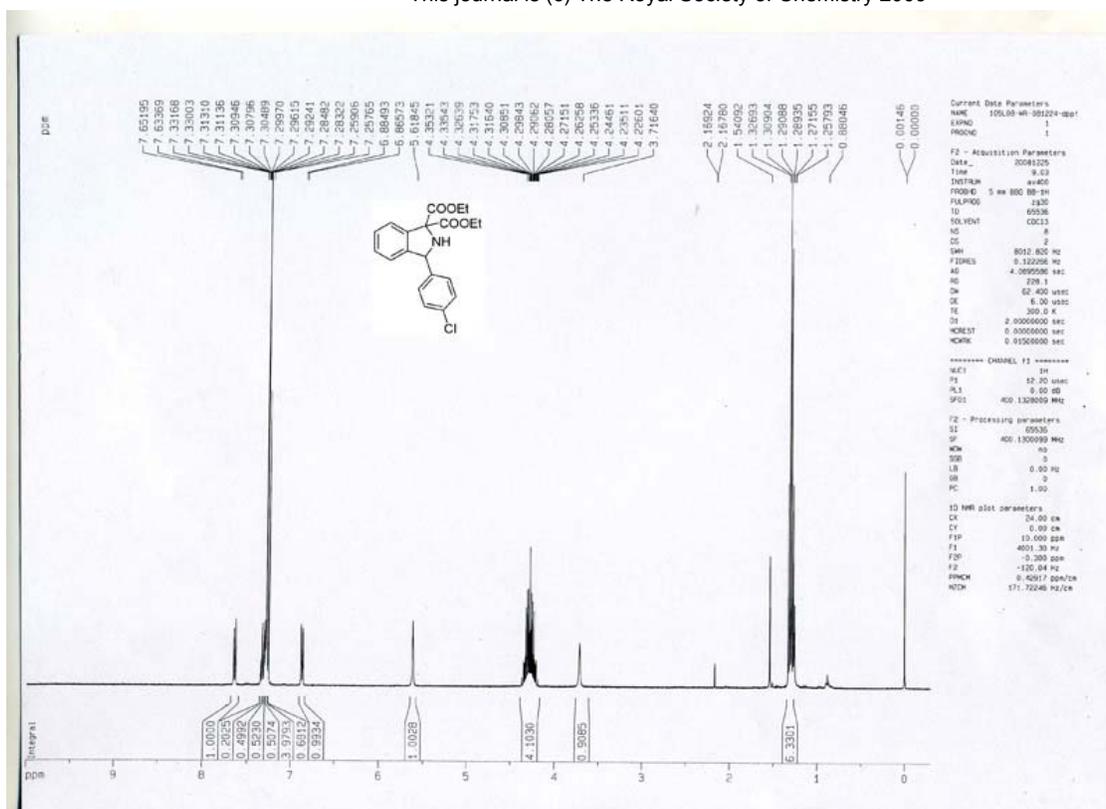












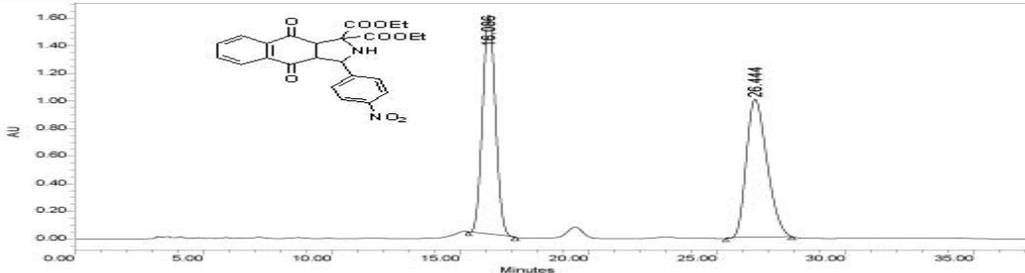
USTC

Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION

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 Sample Type: Unknown  
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 Injection #: 3  
 Injection Volume: 20.00 ul  
 Run Time: 60.00 Minutes  
 Acquired By: System  
 Date Acquired: 4/25/2008 10:12:54 PM  
 Acq. Method: wchao1  
 Date Processed: 4/25/2008 10:50:27 PM  
 Channel Name: 2487Channel 1  
 Sample Set Name:



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
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2	26.444	56593280	50.34	1006880	38.73

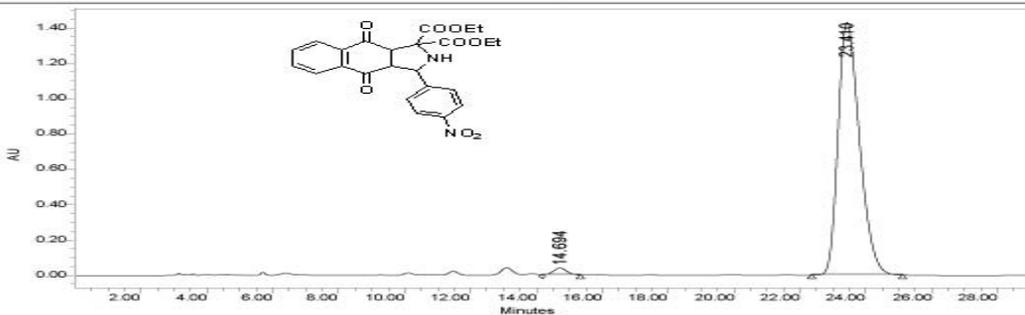
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Project Name: wchao  
 Reported by User: System

*Breeze*

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 Sample Type: Unknown  
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 Injection #: 1  
 Injection Volume: 20.00 ul  
 Run Time: 60.00 Minutes  
 Acquired By: System  
 Date Acquired: 6/19/2008 12:14:50 PM  
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 Date Processed: 6/19/2008 12:45:01 PM  
 Channel Name: 2487Channel 1  
 Sample Set Name:

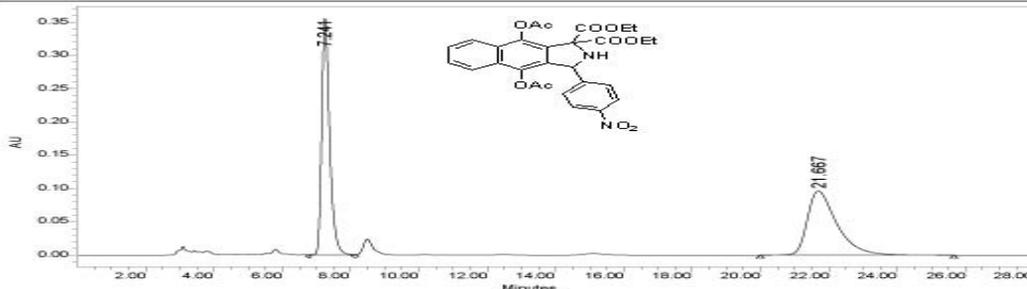


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USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
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Sample Type:	Unknown	Date Acquired:	11/11/2008 6:00:51 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	1	Date Processed:	11/11/2008 6:31:59 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
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Report Method: Untitled

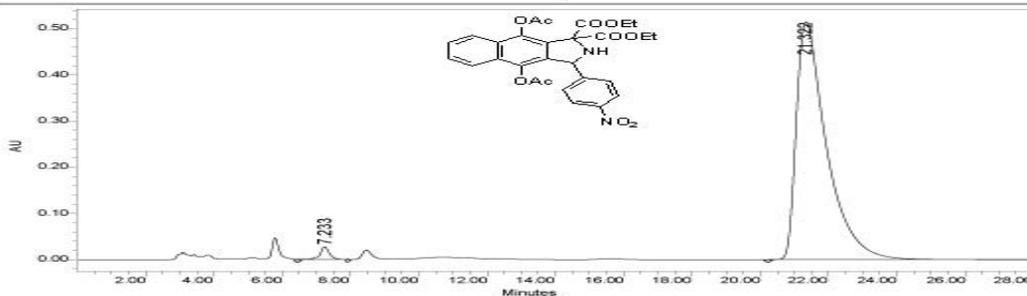
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Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
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Sample Type:	Unknown	Date Acquired:	11/16/2008 3:13:19 AM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	3	Date Processed:	11/16/2008 3:41:51 AM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.233	547058	1.70	27854	5.13
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Printed 11:03:23 PM/15/2009

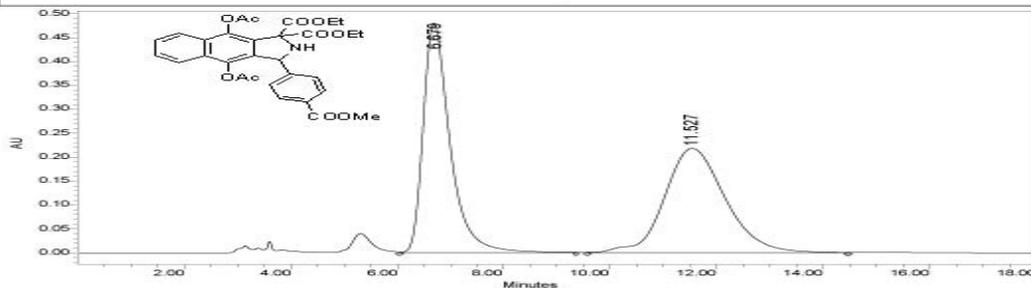
Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION

Sample Name:	wr081119-1-Ac-rac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/20/2008 10:55:10 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	1	Date Processed:	11/20/2008 11:26:09 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.679	16930336	49.36	480512	68.74
2	11.527	17372838	50.64	218554	31.26

Report Method: Untitled

Printed 11:54:49 PM/15/2009

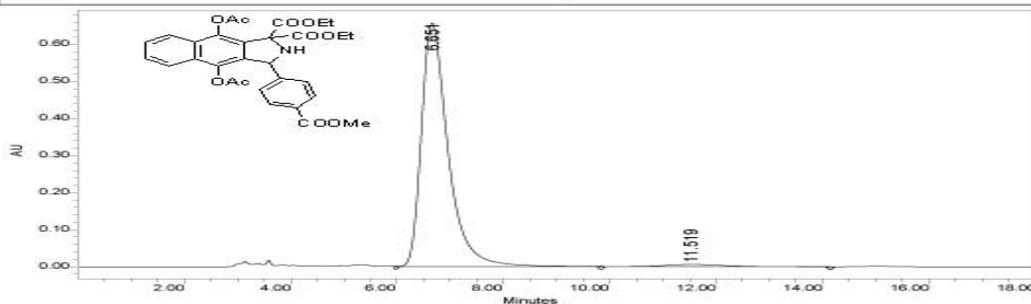
Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION

Sample Name:	wr081119-1-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/20/2008 11:17:24 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	2	Date Processed:	11/23/2008 3:24:30 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.651	22960365	97.74	658124	99.05
2	11.519	530124	2.26	6287	0.95

Report Method: Untitled

Printed 11:51:54 PM/15/2009

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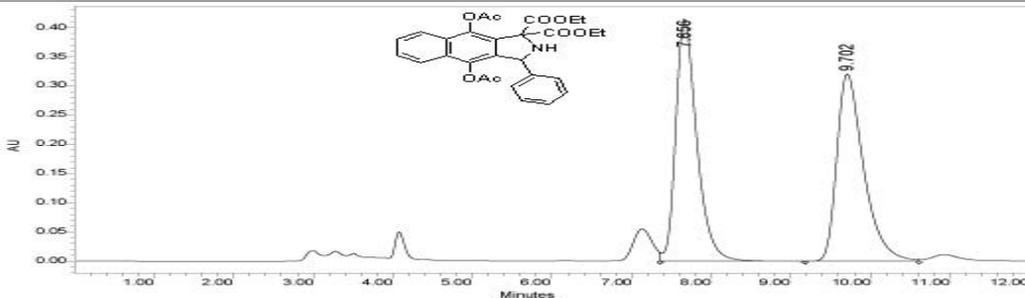
USTC

Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION

Sample Name: wr080805-1-Ac-AD15%  
Sample Type: Unknown  
Vial: 1  
Injection #: 1  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes  
Acquired By: System  
Date Acquired: 1/8/2009 12:37:39 AM  
Acq. Method: wrchao2  
Date Processed: 1/8/2009 1:04:36 AM  
Channel Name: 2487Channel 1  
Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 7.656	7598441	50.28	415506	56.43
2 9.702	7514744	49.72	320844	43.57

Report Method: Untitled

Printed 11:36:54 PM/16/2009

Page: 1 of 1

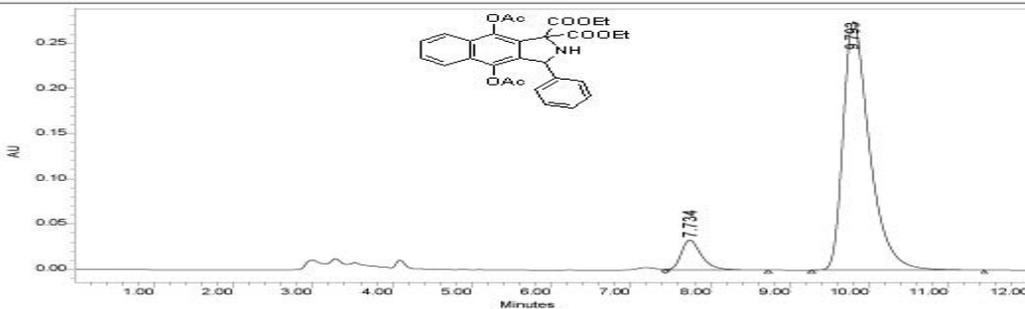
USTC

Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION

Sample Name: wr090106-3-Ac-AD15%  
Sample Type: Unknown  
Vial: 1  
Injection #: 2  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes  
Acquired By: System  
Date Acquired: 1/8/2009 12:51:54 AM  
Acq. Method: wrchao2  
Date Processed: 1/8/2009 1:04:34 AM  
Channel Name: 2487Channel 1  
Sample Set Name:



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 7.734	586896	8.49	33227	10.78
2 9.793	6325995	91.51	274868	89.22

Report Method: Untitled

Printed 11:36:23 PM/16/2009

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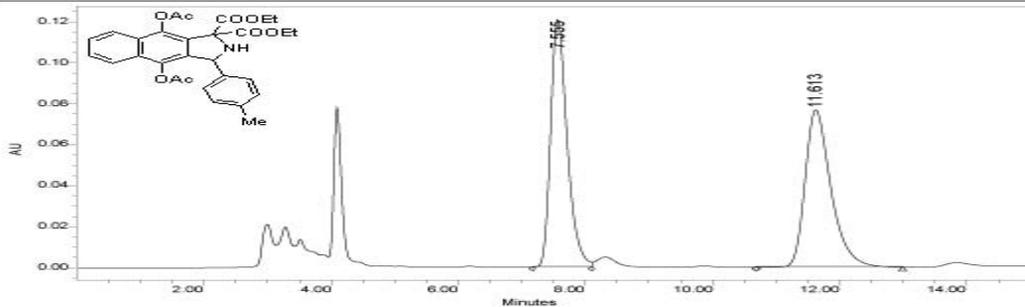
USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

### SAMPLE INFORMATION

Sample Name: wr080711-4-Ac-AD15%  
Sample Type: Unknown  
Vial: 1  
Injection #: 3  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes

Acquired By: System  
Date Acquired: 1/8/2009 1:11:33 AM  
Acq. Method: wrchao2  
Date Processed: 1/16/2009 11:38:40 PM  
Channel Name: 2487Channel 1  
Sample Set Name:



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.555	2186751	50.13	121127	61.18
2	11.613	2175225	49.87	76871	38.82

Report Method: Untitled

Printed 11:39:01 PM/16/2009

Page: 1 of 1

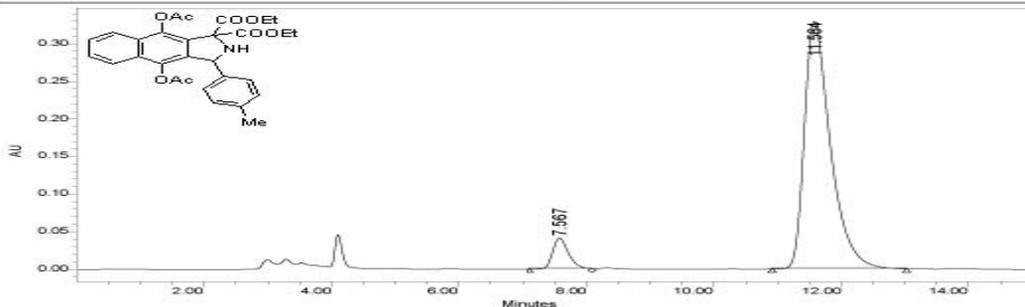
USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

### SAMPLE INFORMATION

Sample Name: wr090106-1-Ac-AD15%  
Sample Type: Unknown  
Vial: 1  
Injection #: 4  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes

Acquired By: System  
Date Acquired: 1/8/2009 1:29:16 AM  
Acq. Method: wrchao2  
Date Processed: 1/8/2009 1:44:35 AM  
Channel Name: 2487Channel 1  
Sample Set Name:



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.567	721473	7.15	41343	11.15
2	11.564	9375272	92.85	329380	88.85

Report Method: Untitled

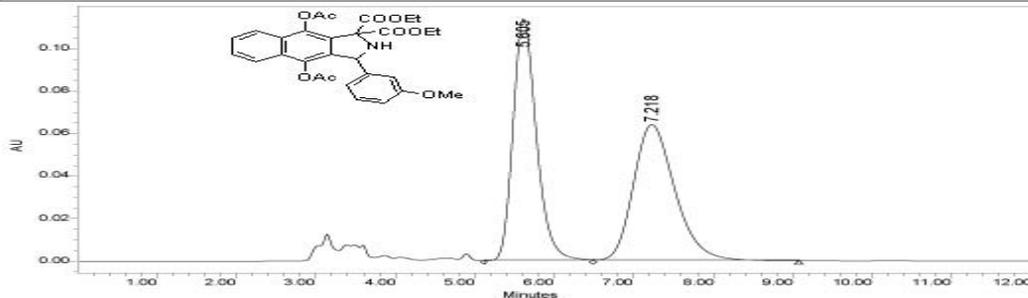
Printed 11:35:55 PM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081111-1-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/13/2008 6:20:02 PM
Vial:	1	Acq. Method:	wchao30%t30
Injection #:	1	Date Processed:	11/13/2008 6:32:28 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	5.605	2338150	50.05	113909	63.99
2	7.218	2333569	49.95	64108	36.01

Report Method: Untitled

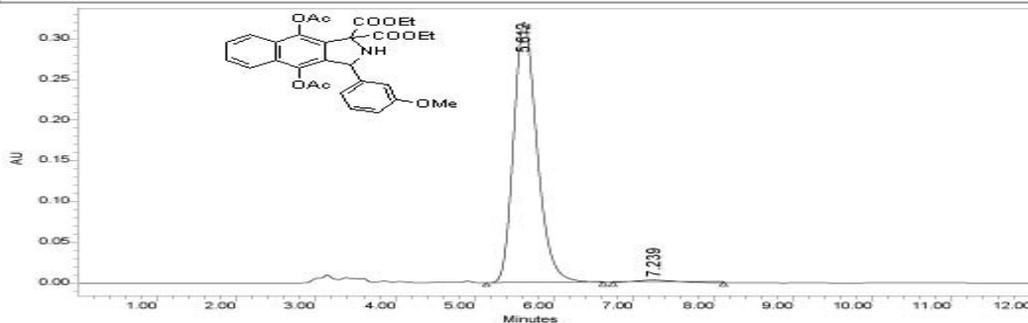
Printed 12:01:55 AM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081111-2-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/13/2008 6:38:43 PM
Vial:	1	Acq. Method:	wchao30%t30
Injection #:	2	Date Processed:	11/13/2008 6:51:50 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	5.612	6541047	98.65	320401	99.14
2	7.239	89655	1.35	2790	0.86

Report Method: Untitled

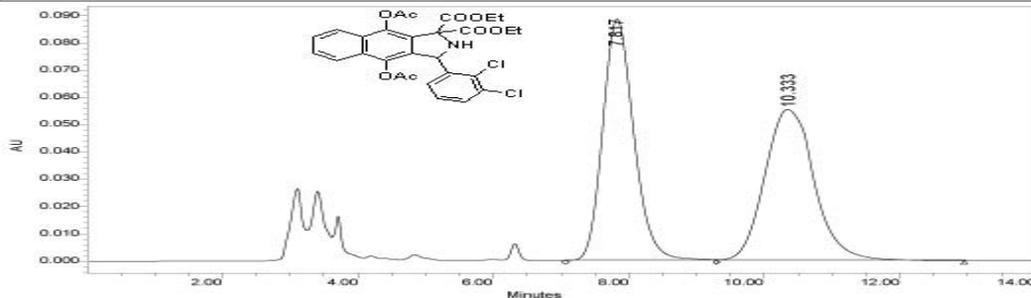
Printed 12:01:05 AM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081129-2-Ac-rac-AS10%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	12/1/2008 12:55:18 AM
Vial:	1	Acq. Method:	wchao10%t30
Injection #:	1	Date Processed:	12/1/2008 1:09:26 AM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.817	2840338	50.07	88667	61.51
2	10.333	2832012	49.93	55494	38.49

Report Method: Untitled

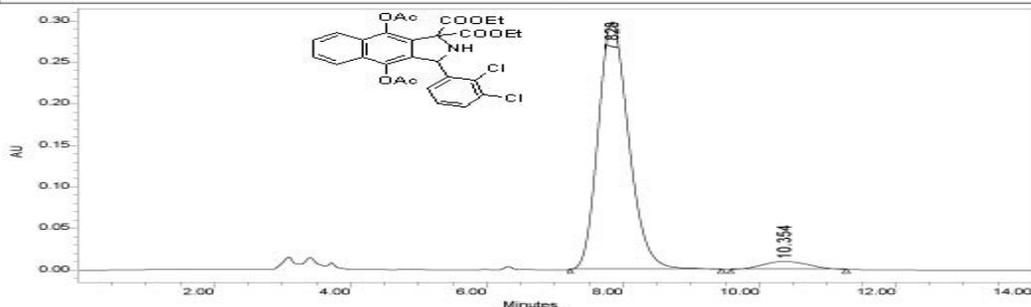
Printed 12:06:47 AM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081129-2-Ac-AS10%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	12/1/2008 1:10:17 AM
Vial:	1	Acq. Method:	wchao10%t30
Injection #:	2	Date Processed:	12/1/2008 1:25:20 AM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.828	9305787	95.41	298097	96.89
2	10.354	447445	4.59	9556	3.11

Report Method: Untitled

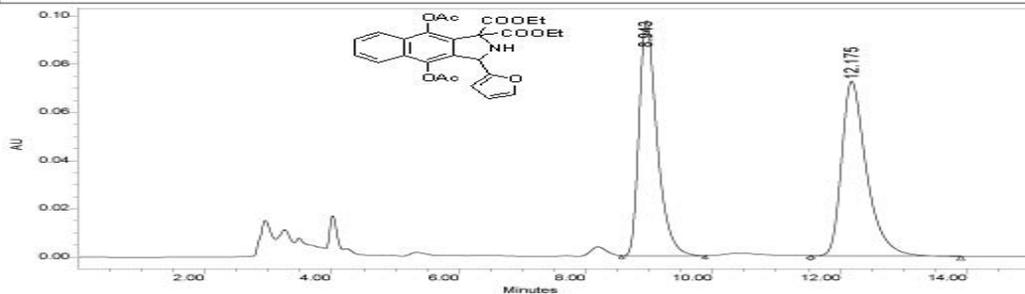
Printed 12:06:13 AM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081114-4-Ac-rac-AD15%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/17/2008 10:40:12 PM
Vial:	1	Acq. Method:	wchao15%430
Injection #:	1	Date Processed:	11/17/2008 10:55:33 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	8.943	1931300	49.22	97791	57.36
2	12.175	1992341	50.78	72709	42.64

Report Method: Untitled

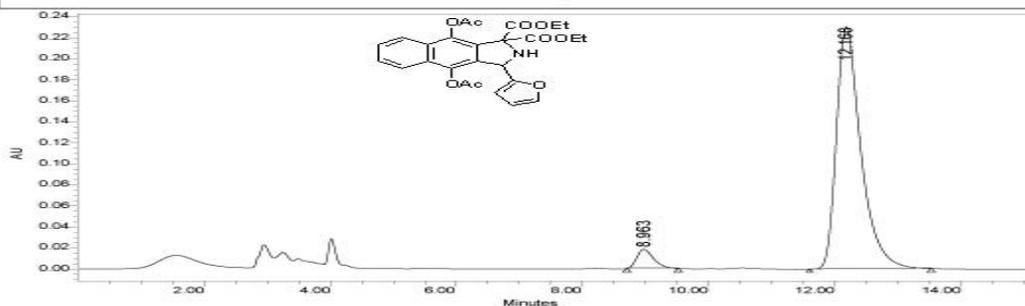
Printed 12:05:16 AM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081114-4-Ac-AD15%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/17/2008 10:59:57 PM
Vial:	1	Acq. Method:	wchao15%430
Injection #:	2	Date Processed:	11/17/2008 11:16:38 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	8.963	336717	5.15	18311	7.36
2	12.168	6205184	94.85	230505	92.64

Report Method: Untitled

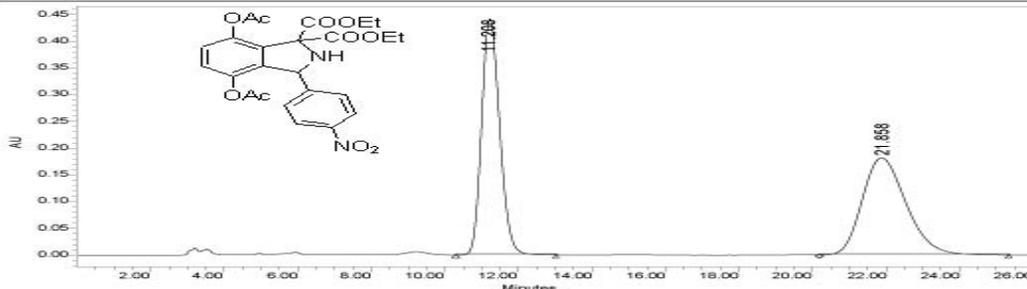
Printed 12:04:38 AM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr080908-1-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	9/17/2008 5:33:16 PM
Vial:	1	Acq. Method:	wchao1
Injection #:	2	Date Processed:	9/17/2008 6:03:55 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	11.208	15040039	50.01	441874	70.86
2	21.858	15031625	49.99	181730	29.14

Report Method: Untitled

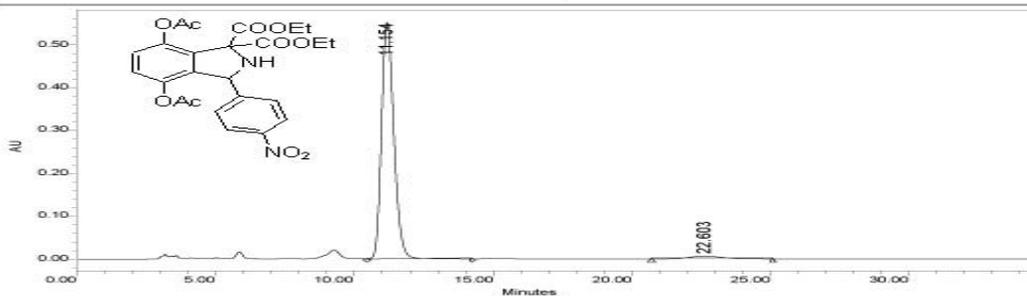
Printed 10:38:38 PM/16/2009

Page: 1 of 1

USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081103-1-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/6/2008 5:03:17 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	1	Date Processed:	11/6/2008 5:38:39 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	11.154	17086579	96.96	551457	98.82
2	22.603	535772	3.04	6591	1.18

Report Method: Untitled

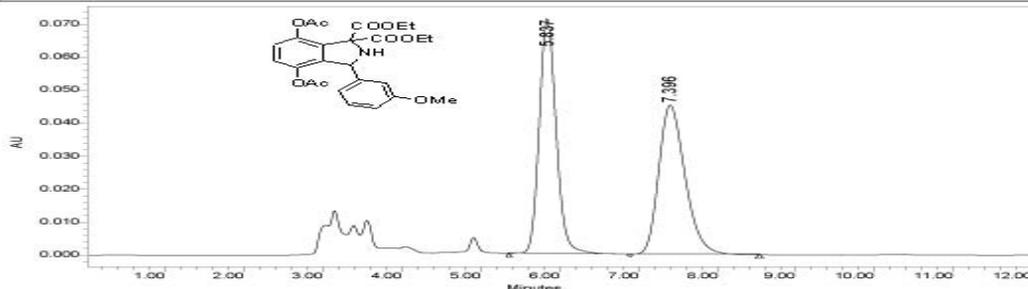
Printed 10:35:57 PM/16/2009

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USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081111-3-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/13/2008 8:08:15 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	6	Date Processed:	11/13/2008 8:21:00 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



RT (min)	Area (V <sup>2</sup> sec)	% Area	Height (V)	% Height
1 5.837	1066837	50.38	71342	61.16
2 7.396	1050816	49.62	45300	38.84

Report Method: Untitled

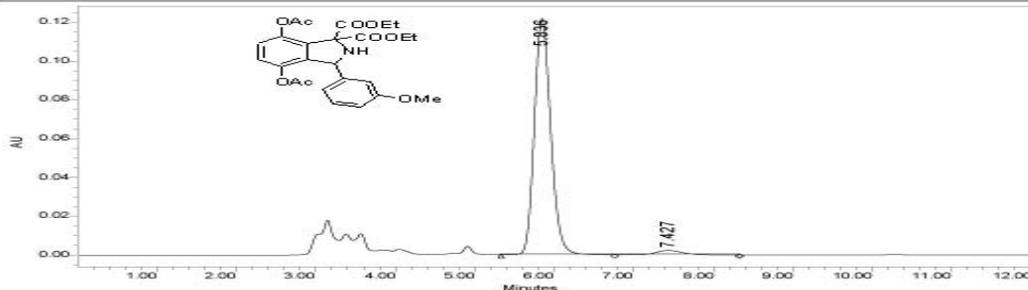
Printed 10:54:25 PM/16/2009

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USTC  
 Project Name: wchao  
 Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081111-4-Ac-AS30%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	11/13/2008 7:48:23 PM
Vial:	1	Acq. Method:	wchao30%430
Injection #:	5	Date Processed:	11/13/2008 8:01:29 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



RT (min)	Area (V <sup>2</sup> sec)	% Area	Height (V)	% Height
1 5.836	1736944	97.01	121715	98.15
2 7.427	53453	2.99	2296	1.85

Report Method: Untitled

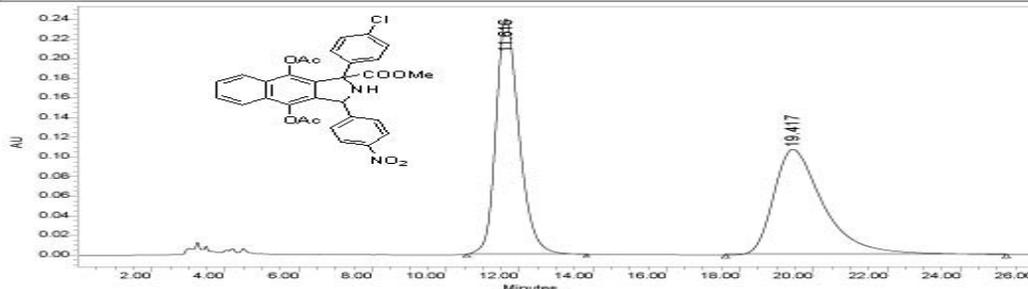
Printed 10:53:44 PM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081201-2-Ac-OD15%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	12/17/2008 3:04:54 PM
Vial:	1	Acq. Method:	wchao15%430
Injection #:	3	Date Processed:	12/17/2008 3:31:24 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 11.616	10444257	50.73	239485	69.04
2 19.417	10143196	49.27	107375	30.96

Report Method: Untitled

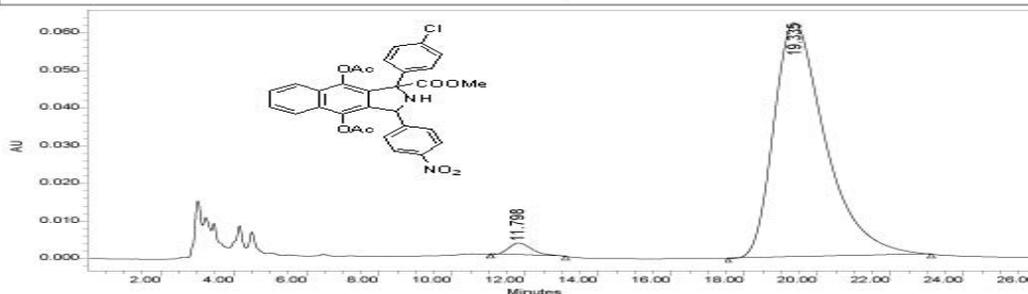
Printed 11:19:05 PM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION			
Sample Name:	wr081209-3-Ac-OD15%	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	12/13/2008 6:50:44 PM
Vial:	1	Acq. Method:	wchao15%430
Injection #:	1	Date Processed:	12/13/2008 7:17:14 PM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	100.00 Minutes	Sample Set Name:	



RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1 11.798	151610	2.41	3249	4.95
2 19.335	6129786	97.59	62373	95.05

Report Method: Untitled

Printed 11:16:50 PM/16/2009

Page: 1 of 1

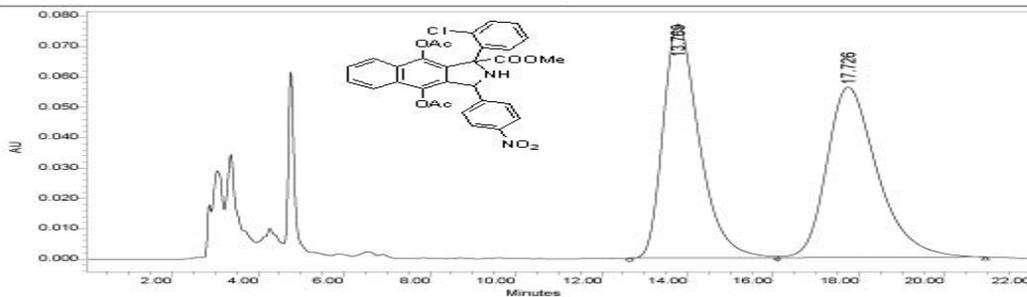
USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

### SAMPLE INFORMATION

Sample Name: wr081206-1-Ac-OD10%  
Sample Type: Unknown  
Vial: 1  
Injection #: 1  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes

Acquired By: System  
Date Acquired: 12/13/2008 5:48:19 PM  
Acq. Method: wchao10%430  
Date Processed: 12/13/2008 6:12:55 PM  
Channel Name: 2487Channel 1  
Sample Set Name:



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	13.769	4512419	49.52	77257	57.89
2	17.726	4600519	50.48	56193	42.11

Report Method: Untitled

Printed 11:23:19 PM/16/2009

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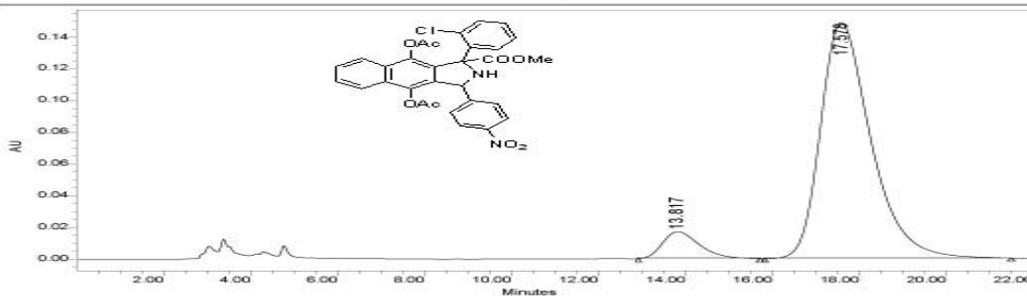
USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

### SAMPLE INFORMATION

Sample Name: wr081208-1-Ac-OD10%  
Sample Type: Unknown  
Vial: 1  
Injection #: 2  
Injection Volume: 20.00 ul  
Run Time: 100.00 Minutes

Acquired By: System  
Date Acquired: 12/13/2008 6:13:36 PM  
Acq. Method: wchao10%430  
Date Processed: 12/13/2008 6:41:14 PM  
Channel Name: 2487Channel 1  
Sample Set Name:



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	13.817	1001451	7.71	17001	10.25
2	17.578	11982708	92.29	148895	89.75

Report Method: Untitled

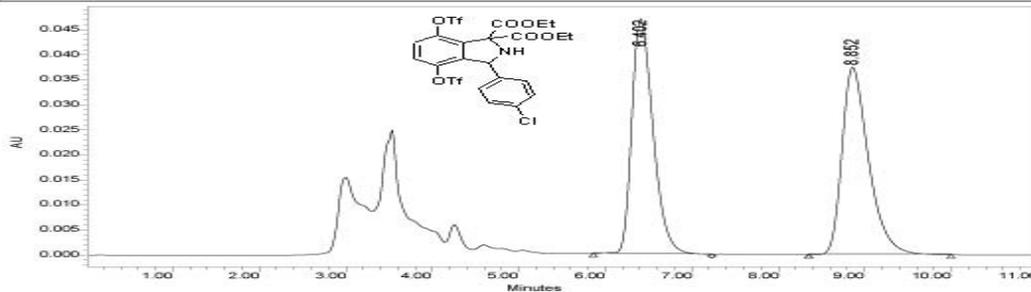
Printed 11:21:53 PM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION				
Sample Name:	wr081227-p-Cl-Tf-rac-AD10%	Acquired By:	System	
Sample Type:	Unknown	Date Acquired:	12/30/2008 9:01:53 PM	
Vial:	1	Acq. Method:	wrchao10%	
Injection #:	1	Date Processed:	12/30/2008 9:13:47 PM	
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1	
Run Time:	100.00 Minutes	Sample Set Name:		



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.402	801519	50.03	46896	55.62
2	8.852	800417	49.97	37412	44.38

Report Method: Untitled

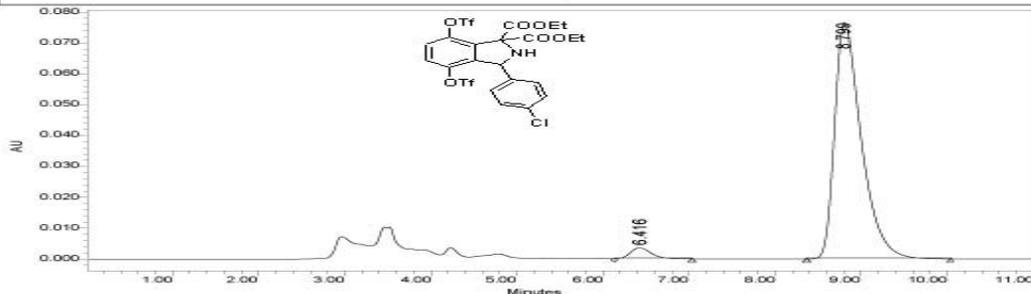
Printed 11:28:57 PM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION				
Sample Name:	wr081230-1-Tf-AD10%	Acquired By:	System	
Sample Type:	Unknown	Date Acquired:	1/1/2009 10:05:58 PM	
Vial:	1	Acq. Method:	wrchao10%	
Injection #:	1	Date Processed:	1/1/2009 10:17:12 PM	
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1	
Run Time:	100.00 Minutes	Sample Set Name:		



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	6.416	60545	3.53	3578	4.46
2	8.799	1652723	96.47	76592	95.54

Report Method: Untitled

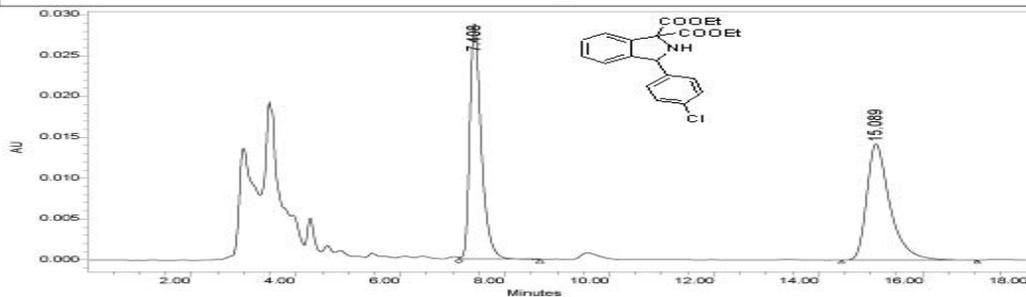
Printed 11:31:35 PM/16/2009

Page: 1 of 1

USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION					
Sample Name:	wr081227-p-Cl-2H-rac-AD10%	Acquired By:	System	Date Acquired:	12/30/2008 10:26:23 PM
Sample Type:	Unknown	Acq. Method:	wrchao10%	Date Processed:	1/16/2009 11:34:02 PM
Vial:	1	Channel Name:	2487Channel 1	Sample Set Name:	
Injection #:	1				
Injection Volume:	20.00 ul				
Run Time:	100.00 Minutes				



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.408	440382	50.27	28965	67.09
2	15.089	435599	49.73	14211	32.91

Report Method: Untitled

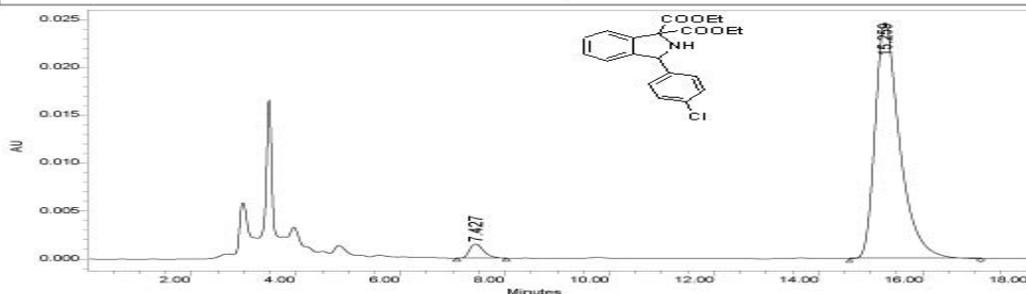
Printed 11:34:56 PM/16/2009

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USTC  
Project Name: wchao  
Reported by User: System

*Breeze*

SAMPLE INFORMATION					
Sample Name:	wr081230-1-2H-AD10%	Acquired By:	System	Date Acquired:	1/15/2009 10:36:35 PM
Sample Type:	Unknown	Acq. Method:	wrchao10%	Date Processed:	1/15/2009 10:55:29 PM
Vial:	1	Channel Name:	2487Channel 1	Sample Set Name:	
Injection #:	1				
Injection Volume:	20.00 ul				
Run Time:	100.00 Minutes				



	RT (min)	Area (V*sec)	% Area	Height (V)	% Height
1	7.427	27872	3.33	1470	5.62
2	15.259	808068	96.67	24681	94.38

Report Method: Untitled

Printed 11:29:34 PM/16/2009

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