

## Supporting Information

Organocatalytic Direct Asymmetric Vinylogous Mannich Reaction of  $\gamma$ -Butenolides with  
Isatins-derived Ketimine

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## General methods

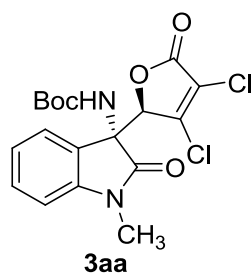
Commercial grade solvent was dried and purified by standard procedures as specified in Purification of Laboratory Chemicals, 4th Ed (Armarego, W. L. F.; Perrin, D. D. Butterworth Heinemann: 1997). NMR spectra were recorded with tetramethylsilane as internal standard.  $^1\text{H}$  NMR spectra were recorded at 300 MHz, and  $^{13}\text{C}$  NMR spectra were recorded at 75 MHz (Bruker Avance). Chemical shifts ( $\delta$ ) were reported in ppm downfield from  $\text{CDCl}_3$  ( $\delta = 7.26$  ppm) for  $^1\text{H}$  NMR and relative to the central  $\text{CDCl}_3$  resonance ( $\delta = 77.0$  ppm) for  $^{13}\text{C}$  NMR spectroscopies. Flash column chromatography was carried out using silica gel eluting with ethyl acetate and petroleum ether. High-resolution mass spectra were obtained with the microTOF-Q mass spectrometer (ESI). Reactions were monitored by TLC and visualized with ultraviolet light. Enantiomeric excess was determined by HPLC analysis on chiralpak AD-H and IC-H columns at 35 °C.

## Materials

All starting materials were obtained from commercial supplier without further purification. The substrates **1**<sup>1</sup> and **2**<sup>2</sup> were prepared according to literature methods. The catalysts **4** were purchased from commercial suppliers or prepared according to literature methods.<sup>3</sup>

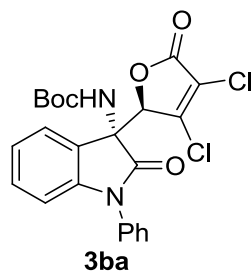
## Procedure for the Direct Asymmetric Vinylogous Mannich Reaction

A mixture of ketimine **1** (0.1 mmol), butenolide **2** (0.2 mmol) and catalyst **4k** (5 mol %) in dry DCM (0.5 mL) was stirred at -30 °C. After the reaction was completed (monitored by TLC), the mixture was directly transferred to a silica gel column and purified by flash column chromatography (hexane/AcOEt = 10/1-4/1) to give **3**.



***tert*-butyl(*R*)-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-1-methyl-2-oxoindolin-3-ylcar**

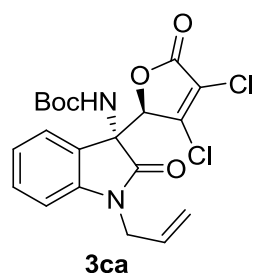
**bamate (3aa)**, white solid, yield 95 %, 77/23 dr, Enantiomeric excess: 90 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 11.0 min,  $t_R$  (major) = 18.5 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37-7.32 (m, 2H), 7.06-7.02 (m, 1H), 6.87-6.83 (m, 1H), 5.47 (s, 1H), 5.52 (s, 1H), 3.32 (s, 3H), 1.30 (s, 9H);  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 163.5, 153.3, 147.5, 147.3, 143.5, 130.6, 128.8, 123.8, 123.5, 108.9, 82.8, 81.2, 63.5, 28.0, 26.5; **HRMS (ESI)** Calcd. for  $\text{C}_{18}\text{H}_{18}\text{Cl}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 435.0490; Found: 435.0485.



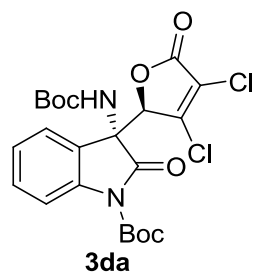
***tert*-butyl(*R*)-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxo-1-phenylindolin-3-ylcar**

**bamate (3ba)**, white solid, yield 94 %, 79/21 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel IC-H column, hexane/*i*-propanol = 85/15, flow rate 1.0 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 16.2 min,  $t_R$  (major) = 17.8 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.56-7.28 (m, 7H), 7.10-7.05 (m, 1H), 6.87-6.80 (m, 1H), 5.81 (s, 1H), 5.60 (s, 1H), 1.35 (s, 9H);  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 163.6, 153.8, 147.7, 143.8, 133.9, 130.4, 129.7, 126.5, 126.1, 124.5, 124.0, 123.9, 123.4, 110.2, 83.0, 80.9, 63.5, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{23}\text{H}_{20}\text{Cl}_2\text{NaN}_2\text{O}_5$

[M+Na]<sup>+</sup>: 497.0647; Found: 497.0648.



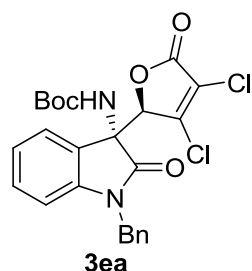
*tert*-butyl(*R*)-1-allyl-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (**3ca**), white solid, yield 97 %, 68/32 dr, Enantiomeric excess: 94 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 7.5 min,  $t_R$  (major) = 15.0 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.38-7.29 (m, 2H), 7.07-7.05 (m, 1H), 6.89-6.82 (m, 1H), 5.83-5.80 (m, 1H), 5.68-5.64 (m, 2H), 5.28-5.22 (m, 2H), 4.47-4.18 (m, 2H), 1.33 (s, 9H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>) δ 170.7, 163.6, 153.5, 147.9, 143.0, 130.7, 130.4, 124.5, 124.1, 123.5, 118.7, 118.2, 109.8, 82.3, 81.2, 63.4, 43.1, 28.1; HRMS (ESI) Calcd. for C<sub>20</sub>H<sub>20</sub>Cl<sub>2</sub>NaN<sub>2</sub>O<sub>5</sub> [M+Na]<sup>+</sup>: 461.0647; Found: 461.0652.



(*R*)-*tert*-butyl-3-(*tert*-butoxycarbonylamino)-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-1-carboxylate (**3da**), white solid, yield 66 %, 75/25 dr, Enantiomeric excess: 83 %, determined by HPLC (Chiralcel IC-H column, hexane/ethanol = 90/10, flow rate 1.0 mL/min, UV detection at 220 nm),  $t_R$  (major) = 9.6 min,  $t_R$  (minor) = 11.7 min. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.89-7.84 (m, 1H), 7.41-7.36 (m, 1H), 7.24-7.12 (m, 2H), 5.78 (s, 1H), 5.44 (s, 1H), 1.63 (s, 9H), 1.26 (s, 9H); <sup>13</sup>C NMR (75MHz, CDCl<sub>3</sub>) δ 170.9, 169.7, 163.2, 153.1, 148.4,

147.0, 139.8, 130.8, 125.2, 124.5, 123.2, 115.8, 85.0, 83.2, 81.0, 63.8, 28.0, 27.8; **HRMS (ESI)**

Calcd. for  $C_{22}H_{24}Cl_2NaN_2O_7$   $[M+Na]^+$ : 521.0858; Found: 521.0863.



**tert-butyl(R)-1-benzyl-3-((R)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcar**

**bamate (3ea)**, white solid, yield 94 %, 67/33 dr, Enantiomeric excess: 96 %, determined by HPLC

(Chiralcel IC-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220

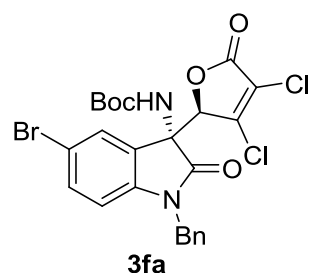
nm),  $t_R$  (minor) = 7.6 min,  $t_R$  (major) = 8.6 min.  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  7.43-7.22 (m, 7H),

7.05-7.03 (m, 1H), 6.78-6.72 (m, 1H), 5.69-5.62 (m, 2H), 5.10-5.03 (m, 1H), 4.79-4.66 (m, 1H),

1.36 (s, 9H);  **$^{13}C$  NMR** (75MHz,  $CDCl_3$ )  $\delta$  172.0, 163.6, 153.8, 148.1, 143.8, 134.8, 130.7, 128.8,

127.8, 127.3, 124.5, 124.2, 123.5, 123.0, 109.9, 82.3, 81.2, 63.4, 44.7, 28.1; **HRMS (ESI)** Calcd.

for  $C_{24}H_{22}Cl_2NaN_2O_5$   $[M+Na]^+$ : 511.0803; Found: 511.0800.



**tert-butyl(R)-1-benzyl-5-bromo-3-((R)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindoli**

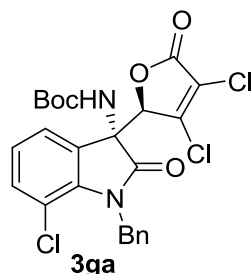
**n-3-ylcarbamate (3fa)**, white solid, yield 69 %, 58/42 dr, Enantiomeric excess: 90 %, determined

by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV

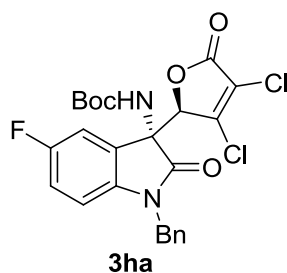
detection at 220 nm),  $t_R$  (minor) = 8.9 min,  $t_R$  (major) = 12.4 min.  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$

7.38-7.30 (m, 7H), 6.63-6.57 (m, 1H), 5.72-5.59 (m, 2H), 5.11-5.00 (m, 1H), 4.78-4.66 (m, 1H),

1.40 (s, 9H);  $^{13}\text{C}$  NMR (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 162.6, 153.8, 148.0, 142.9, 134.4, 133.5, 128.9, 128.0, 127.8, 127.5, 125.9, 123.7, 116.3, 111.4, 81.7, 81.5, 63.4, 44.7, 28.1; HRMS (ESI) Calcd. for  $\text{C}_{24}\text{H}_{21}\text{BrCl}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 590.9888; Found: 590.9903.

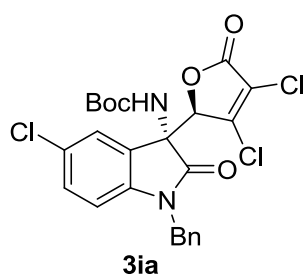


**tert-butyl(R)-1-benzyl-7-chloro-3-((R)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3ga)**, white solid, yield 77 %, 50/50 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel AD-H column, hexane/ethanol = 50/50+0.1% TFA, flow rate 0.7 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.7 min,  $t_R$  (major) = 17.3 min.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.43-7.41 (m, 1H), 7.34-7.29 (m, 3H), 7.25-7.23 (m, 3H), 7.03-6.99 (m, 1H), 5.73-5.55 (m, 2H), 5.44 (s, 1H), 5.22 (s, 1H), 1.37 (s, 9H);  $^{13}\text{C}$  NMR (75MHz,  $\text{CDCl}_3$ )  $\delta$  171.9, 163.4, 153.4, 147.7, 139.2, 136.3, 133.1, 128.5, 127.8, 127.2, 127.0, 126.6, 124.4, 122.7, 116.2, 82.1, 81.6, 62.8, 45.9, 28.1; HRMS (ESI) Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Cl}_3\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 545.0414; Found: 545.0402.

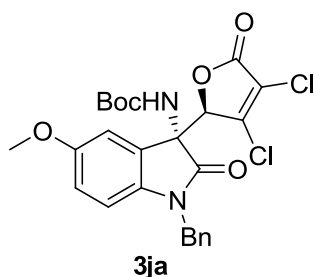


**tert-butyl(R)-1-benzyl-3-((R)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-5-fluoro-2-oxoindolin-3-ylcarbamate (3ha)**, white solid, yield 87 %, 61/39 dr, Enantiomeric excess: 88 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 8.7 min,  $t_R$  (major) = 12.1 min.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$

7.38-7.29 (m, 6H), 6.96-6.92 (m, 1H), 6.69-6.65 (m, 1H), 5.74-5.64 (m, 2H), 5.08-5.01 (m, 1H), 4.79-4.65 (m, 1H), 1.39 (s, 9H);  $^{13}\text{C}$  NMR (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 161.0 ( $J_{\text{C-F}} = 241.8$  Hz), 153.7, 148.1, 146.9, 138.2, 134.2, 128.9, 127.9, 127.3, 123.6, 117.0 ( $J_{\text{C-F}} = 23.4$  Hz), 113.0, 112.7, 110.6 ( $J_{\text{C-F}} = 7.8$  Hz), 81.6, 80.5, 63.5, 44.7, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Cl}_2\text{FNaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 529.0709; Found: 529.0709.

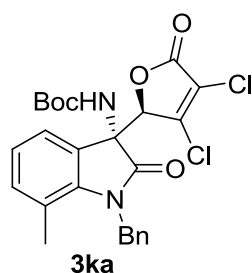


***tert*-butyl(*R*)-1-benzyl-5-chloro-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindole-1-carbamate (3ia)**, white solid, yield 77 %, 60/40 dr, Enantiomeric excess: 90 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_{\text{R}}$  (minor) = 8.6 min,  $t_{\text{R}}$  (major) = 11.7 min.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (s, 1H), 7.35-7.31 (m, 5H), 7.23-7.20 (m, 1H), 6.68-6.61 (m, 1H), 5.73-5.60 (m, 2H), 5.12-5.01 (m, 1H), 4.78-4.60 (m, 1H), 1.40 (s, 9H);  $^{13}\text{C}$  NMR (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 163.3, 153.6, 148.0, 141.5, 134.0, 130.4, 129.0, 128.5, 127.9, 127.5, 127.3, 125.0, 123.6, 110.9, 81.6, 81.5, 63.4, 44.7, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Cl}_3\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 545.0414; Found: 545.0402.



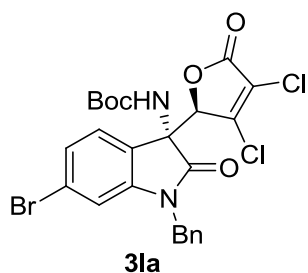
***tert*-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-5-methoxy-2-oxoindole-1-carbamate (3ja)**

**lin-3-ylcarbamate (3ja)**, white solid, yield 95 %, 65/35 dr, Enantiomeric excess: 92 %, determined by HPLC (Chiralcel IC-H column, hexane/*i*-propanol = 50/50, flow rate 1 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.1 min,  $t_R$  (major) = 12.6 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41-7.29 (m, 5H), 7.03-7.00 (m, 1H), 6.77-6.74 (m, 1H), 6.66-6.58 (m, 1H), 5.69-5.66 (m, 2H), 5.12-5.02 (m, 1H), 4.75-4.64 (m, 1H), 3.71 (s, 3H), 1.38 (s, 9H);  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 163.7, 156.4, 153.5, 147.3, 136.1, 134.6, 128.8, 127.7, 127.3, 125.9, 123.4, 115.3, 110.9, 110.6, 82.1, 80.7, 63.6, 55.8, 44.6, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{25}\text{H}_{24}\text{Cl}_2\text{NaN}_2\text{O}_6$   $[\text{M}+\text{Na}]^+$ : 541.0909; Found: 541.0916.

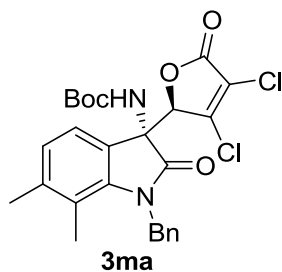


**tert-butyl(R)-1-benzyl-3-((R)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-7-methyl-2-oxoindolin-3-ylcarbamate (3ka)**, white solid, yield 93 %, 64/36 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 8.7 min,  $t_R$  (major) = 17.8 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38-7.22 (m, 6H), 7.03-6.93 (m, 2H), 5.72-5.62 (m, 2H), 5.40 (d,  $J$  = 16.8 Hz, 1H), 4.96 (d,  $J$  = 16.8 Hz, 1H), 2.26 (s, 3H), 1.39 (s, 9H);  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  172.2, 163.7, 153.6, 148.1, 141.0, 136.7, 134.5, 128.8, 127.2, 126.0, 125.8, 123.6, 122.9, 122.0, 120.6, 82.3, 81.0, 62.9, 45.9, 28.1, 18.8; **HRMS (ESI)** Calcd. for  $\text{C}_{25}\text{H}_{24}\text{Cl}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 525.0960; Found: 525.0962.





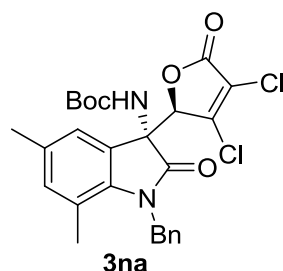
**tert-butyl(*R*)-1-benzyl-6-bromo-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-2-oxindolin-3-ylcarbamate (3la)**, white solid, yield 70 %, 63/37 dr, Enantiomeric excess: 90 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 7.5 min,  $t_R$  (major) = 12.3 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41-7.18 (m, 7H), 6.91-6.85 (m, 1H), 5.71-5.62 (m, 2H), 5.12-5.01 (m, 1H), 4.76-4.64 (m, 1H), 1.39 (s, 9H);  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  172.0, 163.4, 153.7, 147.8, 145.1, 134.0, 128.9, 127.9, 127.6, 127.2, 126.0, 125.4, 124.3, 123.7, 113.2, 82.0, 80.5, 63.0, 44.8, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{21}\text{BrCl}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 590.9879; Found: 590.9895.



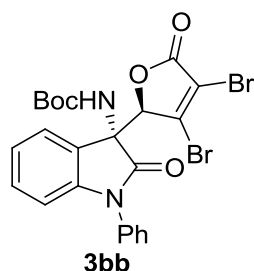
**tert-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-6,7-dimethyl-2-oxindolin-3-ylcarbamate (3ma)**, white solid, yield 56 %, 59/41 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 8.2 min,  $t_R$  (major) = 15.9 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35-7.22 (m, 5H), 7.17-7.01 (m, 1H), 6.89-6.85 (m, 1H), 5.68 (s, 2H), 5.41-5.28 (m, 1H), 5.12-4.93 (m, 1H), 2.20 (s, 3H), 2.10 (s, 3H), 1.39 (s, 9H).  $^{13}\text{C NMR}$  (75MHz,  $\text{CDCl}_3$ )  $\delta$  172.8, 163.8, 153.6, 148.2, 141.2, 137.0, 136.8, 128.8, 127.1, 126.0, 125.4, 124.7, 123.5, 121.4, 119.8,

82.5, 81.1, 62.7, 46.7, 28.2, 21.0, 14.0; **HRMS (ESI)** Calcd. for  $C_{26}H_{26}Cl_2NaN_2O_5$   $[M+Na]^+$ :

539.1116; Found: 539.1113.

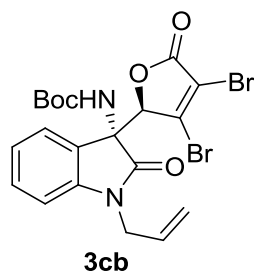


***tert*-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dichloro-5-oxo-2,5-dihydrofuran-2-yl)-5,7-dimethyl-2-oxoin-  
dolin-3-ylcarbamate (3na)**, white solid, yield 92 %, 65/35 dr, Enantiomeric excess: 88 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 7.3 min,  $t_R$  (major) = 15.4 min.  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  7.34-7.30 (m, 3H), 7.27-7.21 (m, 2H), 7.13 (s, 1H), 6.82 (s, 1H), 5.70-5.61 (m, 2H), 5.37 (d,  $J$  = 16.8 Hz, 1H), 4.93 (d,  $J$  = 16.8 Hz, 1H), 2.24 (s 3H), 2.20 (s, 3H), 1.40 (s, 9H).  **$^{13}C$  NMR** (75 MHz,  $CDCl_3$ )  $\delta$  172.0, 163.7, 153.7, 148.2, 138.3, 136.7, 134.9, 133.2, 130.8, 128.8, 127.1, 125.9, 124.3, 122.7, 120.2, 82.2, 81.1, 63.0, 45.8, 28.1, 20.7, 18.5; **HRMS (ESI)** Calcd. for  $C_{26}H_{26}Cl_2NaN_2O_5$   $[M+Na]^+$ : 539.1116; Found: 539.1105.

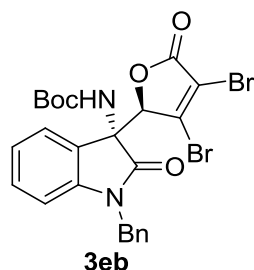


***tert*-butyl(*R*)-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxo-1-phenylindolin-3-ylcarbamate (3bb)**, white solid, yield 93 %, 59/41 dr, Enantiomeric excess: 90 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 16.9 min,  $t_R$  (major) = 21.3 min.  **$^1H$  NMR** (300 MHz,  $CDCl_3$ )  $\delta$  7.54-7.51

(m, 3H), 7.44-7.39 (m, 2H), 7.30-7.28 (m, 2H), 7.10-6.89 (m, 2H), 5.87 (s, 1H), 5.59 (s, 1H), 1.35 (s, 9H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 164.6, 153.4, 145.0, 142.0, 133.9, 130.7, 129.7, 128.2, 126.5, 126.0, 124.1, 123.9, 117.8, 110.1, 85.5, 81.2, 63.5, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{23}\text{H}_{20}\text{Br}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 586.9616; Found: 586.9632.

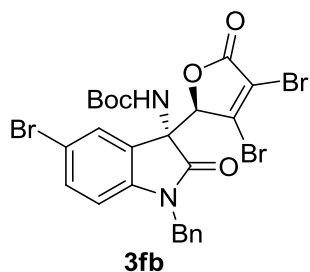


**tert-butyl(R)-1-allyl-3-((R)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3cb)**, white solid, yield 95 %, 68/32 dr, Enantiomeric excess: 92 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 11.6 min,  $t_R$  (major) = 28.1 min.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35-7.28 (m, 2H), 7.06-7.01 (m, 1H), 6.88-6.81 (m, 1H), 5.93-5.72 (m, 1H), 5.70-5.66 (m, 2H), 5.45-5.39 (m, 1H), 5.29-5.25 (m, 1H), 4.46-4.41 (m, 1H), 4.30-4.19 (m, 1H), 1.33 (s, 9H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 164.6, 153.4, 143.0, 142.2, 130.7, 130.3, 124.0, 123.4, 118.6, 118.2, 117.6, 109.8, 84.7, 81.1, 63.4, 43.2, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{20}\text{H}_{20}\text{Br}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 550.9616; Found: 550.9615.

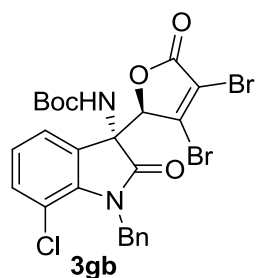


**tert-butyl(R)-1-benzyl-3-((R)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3eb)**, white solid, yield 95 %, 65/35 dr, Enantiomeric excess: 91 %, determined by

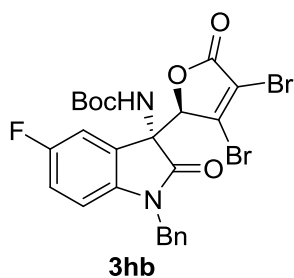
HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 8.3 min,  $t_R$  (major) = 17.7 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45-7.40 (m, 1H), 7.32-7.21 (m, 6H), 7.06-7.01 (m, 1H), 6.76-6.69 (m, 1H), 5.75(dd,  $J = 9.5, 15.4$  Hz, 2H), 5.16 (d,  $J = 15.6$  Hz, 1H), 4.72 (dd,  $J = 10.0, 15.5$  Hz, 1H), 1.36 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.2, 164.6, 153.5, 143.9, 142.3, 134.5, 130.3, 128.7, 127.7, 127.4, 124.1, 123.5, 118.6, 117.6, 109.8, 83.2, 81.2, 63.4, 44.7, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{22}\text{Br}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 600.9773; Found: 600.9786.



***tert*-butyl(*R*)-1-benzyl-5-bromo-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3fb)**, white solid, yield 59 %, 60/40 dr, Enantiomeric excess: 90 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.7 min,  $t_R$  (major) = 13.8 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (s, 1H), 7.36-7.27 (m, 6H), 6.61-.6.56 (m, 1H), 5.72-5.69 (m, 1H), 5.60 (s, 1H), 5.12 (d,  $J = 15.2$  Hz, 1H), 4.71 (dd,  $J = 5.7, 15.7$  Hz, 1H), 1.40 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.1, 164.4, 153.4, 144.3, 141.9, 133.9, 128.8, 128.0, 127.3, 126.4, 125.9, 124.2, 123.8, 117.8, 113.3, 84.5, 81.4, 63.1, 44.8, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Br}_3\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 680.8857; Found: 680.8887.

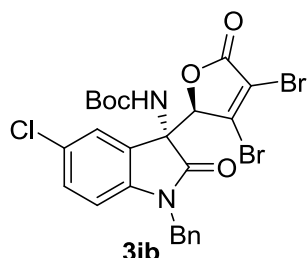


**tert-butyl(*R*)-1-benzyl-7-chloro-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3gb)**, white solid, yield 60 %, 64/36 dr, Enantiomeric excess: 92 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.0 min,  $t_R$  (major) = 29.8 min.  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45-7.43 (m, 1H), 7.34-7.24 (m, 6H), 7.03-6.95 (m, 1H), 5.76-5.75 (m, 1H), 5.51 (d,  $J$  = 16.0 Hz, 1H), 5.36-5.32 (m, 1H), 5.15 (d,  $J$  = 16.0 Hz, 1H), 1.37 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  172.0, 164.4, 153.3, 141.8, 139.1, 136.3, 133.1, 131.6, 128.4, 127.2, 126.6, 124.3, 122.5, 117.9, 116.1, 84.6, 81.5, 62.9, 45.9, 28.1; HRMS (ESI) Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Br}_2\text{ClNaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 634.9383; Found: 634.9390.

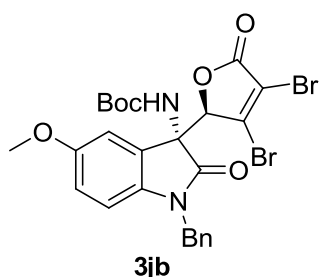


**tert-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-5-fluoro-2-oxoindolin-3-ylcarbamate (3hb)**, white solid, yield 64 %, 64/36 dr, Enantiomeric excess: 89 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.7 min,  $t_R$  (major) = 13.5 min;  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39-7.21 (m, 6H), 6.96-6.93 (m, 1H), 6.65-6.60 (m, 1H), 5.72-5.65 (m, 2H), 5.14 (d,  $J$  = 15.7 Hz, 1H), 4.72 (d,  $J$  = 15.5 Hz, 1H), 1.40 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 164.4, 160.9

( $J_{C-F} = 241.2$  Hz), 153.9, 142.2, 134.5, 128.8, 127.9, 127.4, 118.8, 117.7, 116.9 ( $J_{C-F} = 23.1$  Hz), 113.4, 112.6, 110.7 ( $J_{C-F} = 7.9$  Hz), 84.1, 81.5, 63.6, 44.8, 28.2; **HRMS (ESI)** Calcd. for  $C_{24}H_{21}Br_2FNaN_2O_5$  [ $M+Na$ ] $^+$ : 618.9678; Found: 618.9679.

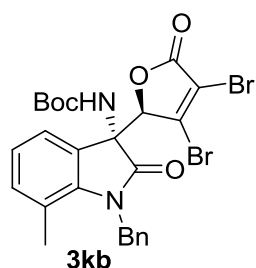


**tert-butyl(R)-1-benzyl-5-chloro-3-((R)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3ib)**, white solid, yield 65 %, 63/37 dr, Enantiomeric excess: 87 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 10.1 min,  $t_R$  (major) = 12.9 min;  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  7.45-7.28 (m, 6H), 7.22-7.19 (m, 1H), 6.66-6.61 (m, 1H), 5.73 (d,  $J = 7.2$  Hz, 2H), 5.13 (d,  $J = 15.6$  Hz, 1H), 4.71 (dd,  $J = 7.2, 15.6$  Hz, 1H), 1.40 (s, 9H);  $^{13}C$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  171.6, 164.3, 153.8, 142.5, 141.5, 134.4, 130.6, 128.9, 128.5, 127.9, 127.5, 126.8, 125.2, 124.9, 110.9, 83.1, 81.6, 63.5, 44.7, 28.2; **HRMS (ESI)** Calcd. for  $C_{24}H_{21}Br_2ClNaN_2O_5$  [ $M+Na$ ] $^+$ : 634.9383; Found: 634.9386.

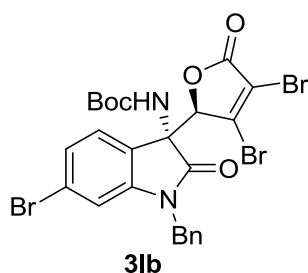


**tert-butyl(R)-1-benzyl-3-((R)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-5-methoxy-2-oxoindolin-3-ylcarbamate (3jb)**, white solid, yield 82 %, 66/34 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min,

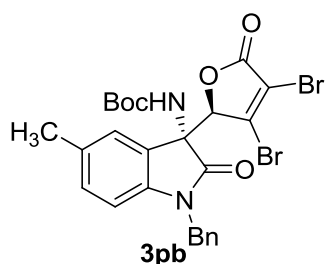
UV detection at 220 nm),  $t_R$  (minor) = 15.8 min,  $t_R$  (major) = 30.1 min;  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.00 (m, 5H), 6.76-6.74 (m, 1H), 6.68-6.64 (m, 1H), 6.61-6.58 (m, 1H), 5.72-5.68 (m, 2H), 5.15 (dd,  $J$  = 6.8, 15.3 Hz, 1H), 4.67 (d,  $J$  = 15.3 Hz, 1H), 3.70 (s, 3H), 1.38 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.9, 164.7, 156.4, 154.0, 142.4, 137.2, 134.9, 130.8, 128.7, 127.7, 126.0, 117.6, 115.4, 111.9, 110.5, 84.6, 81.4, 63.8, 55.8, 44.9, 28.1; **HRMS (ESI)** Calcd. for  $\text{C}_{25}\text{H}_{24}\text{Br}_2\text{NaN}_2\text{O}_6$   $[\text{M}+\text{Na}]^+$ : 630.9878; Found: 630.9890.



**tert-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-7-methyl-2-oxoindolin-3-ylcarbamate (3kb)**, white solid, yield 96 %, 60/40 dr, Enantiomeric excess: 91 %, determined by HPLC (Chiralcel IC-H column, hexane/*i*-propanol = 75/25, flow rate 1.0 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 6.0 min,  $t_R$  (major) = 6.4 min;  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39-7.35 (m, 3H), 7.33-7.27 (m, 3H), 7.01-6.94 (m, 2H), 5.76 (d,  $J$  = 8.6 Hz, 2H), 5.45 (d,  $J$  = 16.8 Hz, 1H), 4.91 (dd,  $J$  = 8.4, 16.9 Hz, 1H), 2.21 (s, 3H), 1.39 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  172.2, 164.6, 153.7, 142.2, 141.4, 136.7, 134.4, 128.8, 127.2, 126.0, 125.6, 123.6, 121.8, 120.5, 117.7, 83.5, 81.1, 62.9, 45.9, 28.1, 18.7; **HRMS (ESI)** Calcd. for  $\text{C}_{25}\text{H}_{24}\text{Br}_2\text{N}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 614.9929; Found: 614.9910.



***tert*-butyl(*R*)-1-benzyl-6-bromo-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-2-oxoindolin-3-ylcarbamate (3lb)**, white solid, yield 71 %, 60/40 dr, Enantiomeric excess: 93 %, determined by HPLC (Chiralcel AD-H column, hexane/ethanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (major) = 17.3 min,  $t_R$  (minor) = 20.1 min;  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.40-7.29 (m, 5H), 7.20-7.16 (m, 2H), 6.88-6.84 (m, 1H), 5.74 (d,  $J = 5.7$  Hz, 1H), 5.60 (s, 1H), 5.12 (d,  $J = 15.7$  Hz, 1H), 4.68 (dd,  $J = 5.4, 15.6$  Hz, 1H), 1.37 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 164.3, 153.5, 142.0, 134.0, 133.3, 131.8, 128.8, 127.9, 127.6, 127.4, 126.1, 117.7, 116.2, 111.4, 83.1, 81.6, 63.4, 44.7, 28.2; **HRMS (ESI)** Calcd. for  $\text{C}_{24}\text{H}_{21}\text{Br}_3\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 678.8878; Found: 678.8866.



***tert*-butyl(*R*)-1-benzyl-3-((*R*)-3,4-dibromo-5-oxo-2,5-dihydrofuran-2-yl)-5-methyl-2-oxoindolin-3-ylcarbamate (3pb)**, white solid, yield 95 %, 66/34 dr, Enantiomeric excess: 88 %, determined by HPLC (Chiralcel AD-H column, hexane/*i*-propanol = 50/50, flow rate 0.6 mL/min, UV detection at 220 nm),  $t_R$  (minor) = 9.8 min,  $t_R$  (major) = 25.5 min;  $^1\text{H NMR}$  (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.40 (m, 1H), 7.35-7.23 (m, 4H), 7.19 (s, 1H), 7.04-7.01 (m, 1H), 6.63-6.57 (m, 1H), 5.71-5.67 (m, 2H), 5.11-5.06 (m, 1H), 4.69-4.63 (m, 1H), 2.25 (s, 3H), 1.38 (s, 9H);  $^{13}\text{C NMR}$  (75 MHz,  $\text{CDCl}_3$ )  $\delta$  171.0, 164.6, 153.5, 142.5, 140.5, 134.6, 133.2, 132.6, 130.6, 128.7, 127.7, 124.9, 124.0, 117.5, 109.6, 84.7, 81.1, 63.6, 44.6, 28.2, 21.0; **HRMS (ESI)** Calcd. for  $\text{C}_{25}\text{H}_{24}\text{Br}_2\text{NaN}_2\text{O}_5$   $[\text{M}+\text{Na}]^+$ : 614.9929; Found: 614.9910.



## X-Ray Crystal Data for Compound 3aa

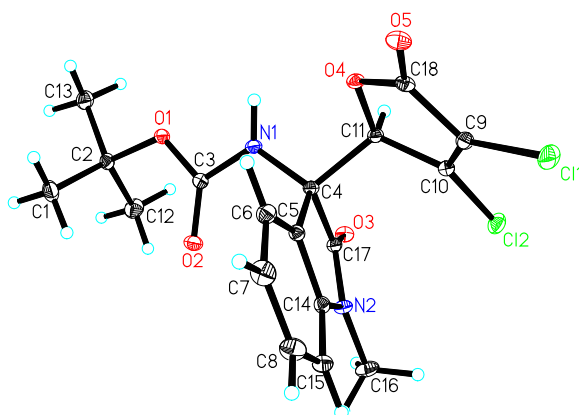


Table 1. Crystal data and structure refinement for **3aa**

Empirical formula	C <sub>18</sub> H <sub>18</sub> Cl <sub>2</sub> N <sub>2</sub> O <sub>5</sub>	
Formula weight	413.24	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Orthorhombic, P 21 21 21	
Unit cell dimensions	a = 8.5564(8) Å	alpha = 90 deg.
	b = 9.1450(8) Å	beta = 90 deg.
	c = 24.531(2) Å	gamma = 90 deg.
Volume	1919.5(3) Å <sup>3</sup>	
Z, Calculated density	4, 1.430 Mg/m <sup>3</sup>	
Absorption coefficient	0.370 mm <sup>-1</sup>	
F(000)	856	
Crystal size	0.38 x 0.22 x 0.10 mm	
Theta range for data collection	1.66 to 30.67 deg.	
Limiting indices	-11<=h<=12, -12<=k<=12, -34<=l<=35	
Reflections collected / unique	20623 / 5509 [R(int) = 0.0456]	
Completeness to theta = 30.67	95.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9639 and 0.8722	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	5509 / 0 / 248	
Goodness-of-fit on F <sup>2</sup>	1.044	
Final R indices [I>2sigma(I)]	R1 = 0.0347, wR2 = 0.0816	
R indices (all data)	R1 = 0.0428, wR2 = 0.0852	
Absolute structure parameter	0.00(4)	
Largest diff. peak and hole	0.339 and -0.298 e.Å <sup>-3</sup>	

Table 2. Atomic coordinates ( x 10<sup>4</sup>) and equivalent isotropic displacement parameters (Å<sup>2</sup> x 10<sup>3</sup>) for **3aa**.

U(eq) is defined as one third of the trace of the orthogonalized

Uij tensor.

	x	y	z	U(eq)
Cl(1)	7593(1)	11290(1)	9785(1)	29(1)
Cl(2)	7888(1)	9024(1)	8684(1)	25(1)
O(1)	1303(1)	10318(1)	7183(1)	17(1)
O(2)	1641(2)	8349(1)	7748(1)	24(1)
O(3)	5338(1)	8088(1)	7629(1)	18(1)
O(4)	4726(1)	12173(1)	8593(1)	16(1)
O(5)	4876(2)	13397(1)	9385(1)	22(1)
N(1)	3018(2)	10445(1)	7857(1)	16(1)
N(2)	4553(2)	7331(1)	8483(1)	16(1)
C(1)	-1337(2)	9279(2)	7150(1)	27(1)
C(2)	138(2)	9582(2)	6829(1)	18(1)
C(3)	1937(2)	9588(2)	7602(1)	17(1)
C(4)	4011(2)	9786(2)	8265(1)	14(1)
C(5)	3259(2)	9363(2)	8803(1)	15(1)
C(6)	2353(2)	10145(2)	9163(1)	20(1)
C(7)	1916(2)	9484(2)	9652(1)	26(1)
C(8)	2399(2)	8067(2)	9773(1)	27(1)
C(9)	6488(2)	11274(2)	9213(1)	18(1)
C(10)	6576(2)	10388(2)	8783(1)	16(1)
C(11)	5390(2)	10858(2)	8363(1)	14(1)
C(12)	844(2)	8205(2)	6581(1)	29(1)
C(13)	-149(2)	10721(2)	6389(1)	24(1)
C(14)	3704(2)	7924(2)	8922(1)	16(1)
C(15)	3305(2)	7257(2)	9408(1)	21(1)
C(16)	5140(2)	5836(2)	8462(1)	25(1)
C(17)	4710(2)	8299(2)	8069(1)	15(1)
C(18)	5299(2)	12397(2)	9105(1)	17(1)

Table 3. Bond lengths [Å] and angles [deg] for **3aa**.

Cl(1)-C(9)	1.6918(16)
Cl(2)-C(10)	1.6952(16)
O(1)-C(3)	1.3401(19)
O(1)-C(2)	1.4827(19)
O(2)-C(3)	1.2153(19)
O(3)-C(17)	1.221(2)
O(4)-C(18)	1.3621(19)
O(4)-C(11)	1.4451(18)
O(5)-C(18)	1.201(2)
N(1)-C(3)	1.365(2)

N(1)-C(4)	1.444(2)
N(1)-H(1)	0.8800
N(2)-C(17)	1.355(2)
N(2)-C(14)	1.407(2)
N(2)-C(16)	1.458(2)
C(1)-C(2)	1.512(2)
C(1)-H(1A)	0.9800
C(1)-H(1B)	0.9800
C(1)-H(1C)	0.9800
C(2)-C(13)	1.521(2)
C(2)-C(12)	1.524(2)
C(4)-C(5)	1.518(2)
C(4)-C(11)	1.553(2)
C(4)-C(17)	1.561(2)
C(5)-C(6)	1.376(2)
C(5)-C(14)	1.401(2)
C(6)-C(7)	1.396(2)
C(6)-H(6)	0.9500
C(7)-C(8)	1.392(3)
C(7)-H(7)	0.9500
C(8)-C(15)	1.398(3)
C(8)-H(8)	0.9500
C(9)-C(10)	1.332(2)
C(9)-C(18)	1.469(2)
C(10)-C(11)	1.510(2)
C(11)-H(11)	1.0000
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-C(15)	1.381(2)
C(15)-H(15)	0.9500
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(3)-O(1)-C(2)	119.61(12)
C(18)-O(4)-C(11)	110.15(12)
C(3)-N(1)-C(4)	118.51(12)
C(3)-N(1)-H(1)	120.7
C(4)-N(1)-H(1)	120.7
C(17)-N(2)-C(14)	111.94(13)
C(17)-N(2)-C(16)	123.44(14)

C(14)-N(2)-C(16)	124.56(14)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
O(1)-C(2)-C(1)	109.91(14)
O(1)-C(2)-C(13)	102.30(12)
C(1)-C(2)-C(13)	111.12(14)
O(1)-C(2)-C(12)	110.02(13)
C(1)-C(2)-C(12)	112.79(16)
C(13)-C(2)-C(12)	110.21(15)
O(2)-C(3)-O(1)	127.36(16)
O(2)-C(3)-N(1)	122.75(15)
O(1)-C(3)-N(1)	109.89(13)
N(1)-C(4)-C(5)	117.33(14)
N(1)-C(4)-C(11)	106.88(12)
C(5)-C(4)-C(11)	110.44(13)
N(1)-C(4)-C(17)	112.03(13)
C(5)-C(4)-C(17)	102.01(12)
C(11)-C(4)-C(17)	107.84(12)
C(6)-C(5)-C(14)	120.49(15)
C(6)-C(5)-C(4)	131.61(14)
C(14)-C(5)-C(4)	107.83(14)
C(5)-C(6)-C(7)	118.55(15)
C(5)-C(6)-H(6)	120.7
C(7)-C(6)-H(6)	120.7
C(8)-C(7)-C(6)	120.44(17)
C(8)-C(7)-H(7)	119.8
C(6)-C(7)-H(7)	119.8
C(7)-C(8)-C(15)	121.45(17)
C(7)-C(8)-H(8)	119.3
C(15)-C(8)-H(8)	119.3
C(10)-C(9)-C(18)	108.74(14)
C(10)-C(9)-Cl(1)	129.02(13)
C(18)-C(9)-Cl(1)	122.08(12)
C(9)-C(10)-C(11)	109.22(14)
C(9)-C(10)-Cl(2)	126.77(13)
C(11)-C(10)-Cl(2)	123.84(12)
O(4)-C(11)-C(10)	103.50(12)
O(4)-C(11)-C(4)	106.67(12)
C(10)-C(11)-C(4)	115.86(12)
O(4)-C(11)-H(11)	110.2

C(10)-C(11)-H(11)	110.2
C(4)-C(11)-H(11)	110.2
C(2)-C(12)-H(12A)	109.5
C(2)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(2)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(2)-C(13)-H(13A)	109.5
C(2)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(2)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(15)-C(14)-C(5)	121.88(16)
C(15)-C(14)-N(2)	128.13(15)
C(5)-C(14)-N(2)	109.99(14)
C(14)-C(15)-C(8)	117.15(16)
C(14)-C(15)-H(15)	121.4
C(8)-C(15)-H(15)	121.4
N(2)-C(16)-H(16A)	109.5
N(2)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
N(2)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
O(3)-C(17)-N(2)	127.09(14)
O(3)-C(17)-C(4)	125.39(13)
N(2)-C(17)-C(4)	107.45(13)
O(5)-C(18)-O(4)	122.29(15)
O(5)-C(18)-C(9)	129.57(16)
O(4)-C(18)-C(9)	108.10(13)

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Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for **3aa**.

The anisotropic displacement factor exponent takes the form:  $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

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	U11	U22	U33	U23	U13	U12
Cl(1)	38(1)	28(1)	21(1)	-1(1)	-14(1)	1(1)
Cl(2)	21(1)	22(1)	30(1)	-3(1)	-5(1)	7(1)
O(1)	18(1)	15(1)	19(1)	2(1)	-5(1)	-2(1)
O(2)	26(1)	18(1)	28(1)	7(1)	-8(1)	-6(1)

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O(3)	23(1)	16(1)	15(1)	-2(1)	2(1)	0(1)
O(4)	20(1)	11(1)	16(1)	-1(1)	-2(1)	1(1)
O(5)	30(1)	17(1)	19(1)	-3(1)	5(1)	-2(1)
N(1)	20(1)	12(1)	18(1)	2(1)	-4(1)	-1(1)
N(2)	21(1)	12(1)	16(1)	2(1)	1(1)	2(1)
C(1)	17(1)	34(1)	31(1)	2(1)	0(1)	-4(1)
C(2)	16(1)	18(1)	20(1)	-3(1)	-4(1)	0(1)
C(3)	16(1)	16(1)	19(1)	2(1)	0(1)	-1(1)
C(4)	16(1)	12(1)	15(1)	1(1)	0(1)	0(1)
C(5)	14(1)	16(1)	14(1)	1(1)	0(1)	-1(1)
C(6)	20(1)	18(1)	23(1)	-1(1)	2(1)	2(1)
C(7)	27(1)	31(1)	22(1)	-4(1)	10(1)	1(1)
C(8)	33(1)	30(1)	18(1)	4(1)	6(1)	-2(1)
C(9)	21(1)	17(1)	15(1)	2(1)	-5(1)	-2(1)
C(10)	16(1)	14(1)	19(1)	2(1)	-2(1)	0(1)
C(11)	16(1)	12(1)	15(1)	0(1)	-2(1)	0(1)
C(12)	30(1)	23(1)	33(1)	-8(1)	-4(1)	5(1)
C(13)	24(1)	26(1)	23(1)	-1(1)	-7(1)	0(1)
C(14)	15(1)	17(1)	15(1)	0(1)	0(1)	-1(1)
C(15)	23(1)	20(1)	20(1)	5(1)	0(1)	-1(1)
C(16)	34(1)	15(1)	28(1)	3(1)	5(1)	8(1)
C(17)	16(1)	13(1)	16(1)	1(1)	-2(1)	0(1)
C(18)	21(1)	14(1)	15(1)	2(1)	1(1)	-4(1)

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for **3aa**.

	x	y	z	U(eq)
H(1)	3108	11378	7775	20
H(1A)	-1716	10190	7314	41
H(1B)	-1114	8567	7438	41
H(1C)	-2139	8885	6905	41
H(6)	2032	11115	9080	25
H(7)	1284	10004	9905	32
H(8)	2106	7641	10111	32
H(11)	5923	11086	8010	17
H(12A)	1008	7475	6868	43
H(12B)	1847	8445	6410	43
H(12C)	130	7811	6305	43
H(13A)	826	10903	6190	37
H(13B)	-510	11632	6557	37
H(13C)	-946	10361	6135	37
H(15)	3633	6289	9490	25

H(16A)	6169	5791	8636	38
H(16B)	5231	5527	8080	38
H(16C)	4415	5186	8653	38

Table 6. Torsion angles [deg] for **3aa**.

C(3)-O(1)-C(2)-C(1)	66.94(18)
C(3)-O(1)-C(2)-C(13)	-174.95(14)
C(3)-O(1)-C(2)-C(12)	-57.84(19)
C(2)-O(1)-C(3)-O(2)	-2.2(3)
C(2)-O(1)-C(3)-N(1)	177.56(13)
C(4)-N(1)-C(3)-O(2)	9.4(2)
C(4)-N(1)-C(3)-O(1)	-170.38(14)
C(3)-N(1)-C(4)-C(5)	-71.05(19)
C(3)-N(1)-C(4)-C(11)	164.36(14)
C(3)-N(1)-C(4)-C(17)	46.44(19)
N(1)-C(4)-C(5)-C(6)	-52.0(2)
C(11)-C(4)-C(5)-C(6)	70.8(2)
C(17)-C(4)-C(5)-C(6)	-174.80(17)
N(1)-C(4)-C(5)-C(14)	131.09(14)
C(11)-C(4)-C(5)-C(14)	-106.13(14)
C(17)-C(4)-C(5)-C(14)	8.30(16)
C(14)-C(5)-C(6)-C(7)	1.3(3)
C(4)-C(5)-C(6)-C(7)	-175.29(17)
C(5)-C(6)-C(7)-C(8)	0.4(3)
C(6)-C(7)-C(8)-C(15)	-1.2(3)
C(18)-C(9)-C(10)-C(11)	-1.65(19)
Cl(1)-C(9)-C(10)-C(11)	-177.10(13)
C(18)-C(9)-C(10)-Cl(2)	173.72(12)
Cl(1)-C(9)-C(10)-Cl(2)	-1.7(3)
C(18)-O(4)-C(11)-C(10)	-5.46(16)
C(18)-O(4)-C(11)-C(4)	117.23(13)
C(9)-C(10)-C(11)-O(4)	4.29(17)
Cl(2)-C(10)-C(11)-O(4)	-171.25(11)
C(9)-C(10)-C(11)-C(4)	-112.09(16)
Cl(2)-C(10)-C(11)-C(4)	72.38(18)
N(1)-C(4)-C(11)-O(4)	64.80(15)
C(5)-C(4)-C(11)-O(4)	-63.88(15)
C(17)-C(4)-C(11)-O(4)	-174.57(11)
N(1)-C(4)-C(11)-C(10)	179.38(13)
C(5)-C(4)-C(11)-C(10)	50.70(18)
C(17)-C(4)-C(11)-C(10)	-59.99(17)
C(6)-C(5)-C(14)-C(15)	-2.4(2)
C(4)-C(5)-C(14)-C(15)	174.92(15)

C(6)-C(5)-C(14)-N(2)	177.05(15)
C(4)-C(5)-C(14)-N(2)	-5.64(18)
C(17)-N(2)-C(14)-C(15)	179.33(17)
C(16)-N(2)-C(14)-C(15)	2.0(3)
C(17)-N(2)-C(14)-C(5)	-0.07(19)
C(16)-N(2)-C(14)-C(5)	-177.43(16)
C(5)-C(14)-C(15)-C(8)	1.6(3)
N(2)-C(14)-C(15)-C(8)	-177.69(17)
C(7)-C(8)-C(15)-C(14)	0.1(3)
C(14)-N(2)-C(17)-O(3)	-177.38(16)
C(16)-N(2)-C(17)-O(3)	0.0(3)
C(14)-N(2)-C(17)-C(4)	5.57(18)
C(16)-N(2)-C(17)-C(4)	-177.03(15)
N(1)-C(4)-C(17)-O(3)	48.2(2)
C(5)-C(4)-C(17)-O(3)	174.52(16)
C(11)-C(4)-C(17)-O(3)	-69.2(2)
N(1)-C(4)-C(17)-N(2)	-134.70(14)
C(5)-C(4)-C(17)-N(2)	-8.37(16)
C(11)-C(4)-C(17)-N(2)	107.96(14)
C(11)-O(4)-C(18)-O(5)	-177.43(15)
C(11)-O(4)-C(18)-C(9)	4.71(17)
C(10)-C(9)-C(18)-O(5)	-179.50(17)
Cl(1)-C(9)-C(18)-O(5)	-3.7(3)
C(10)-C(9)-C(18)-O(4)	-1.84(19)
Cl(1)-C(9)-C(18)-O(4)	173.99(12)

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Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **3aa** [A and deg.].

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D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(3)#1	0.88	2.28	3.0401(18)	144.6

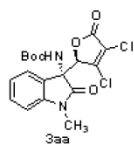
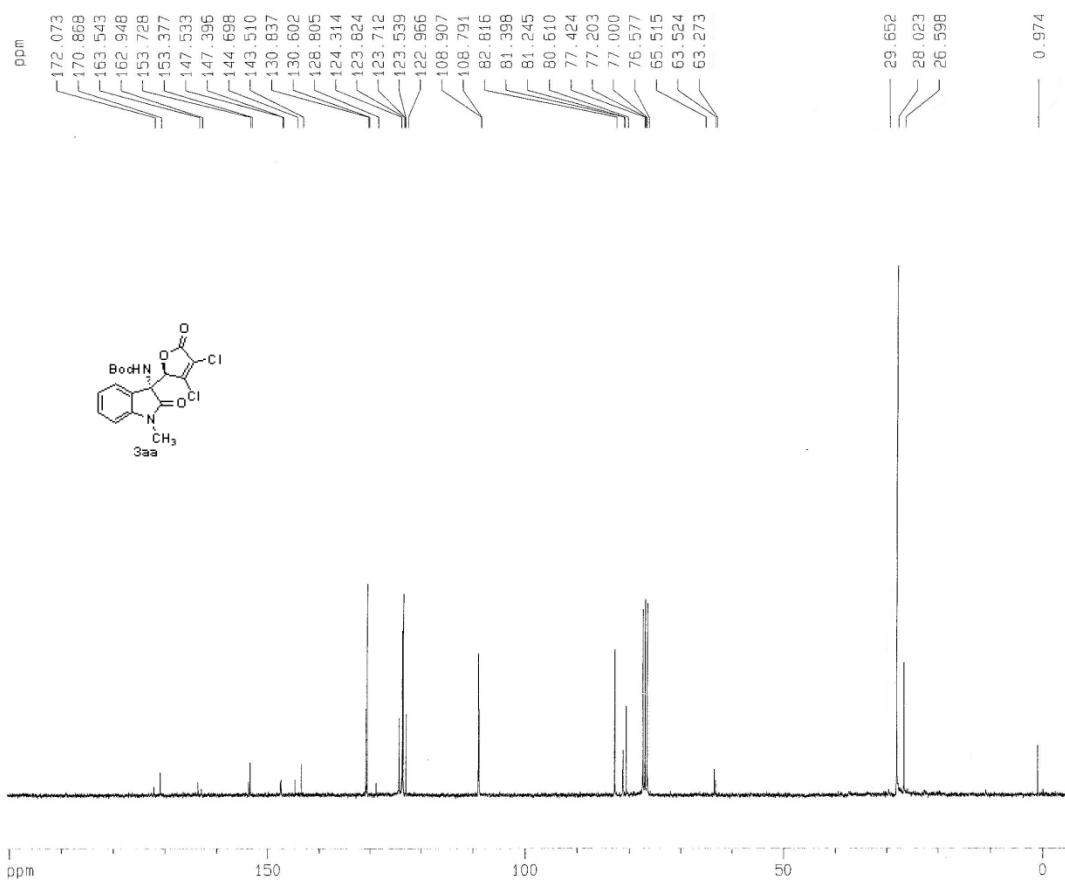
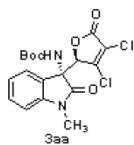
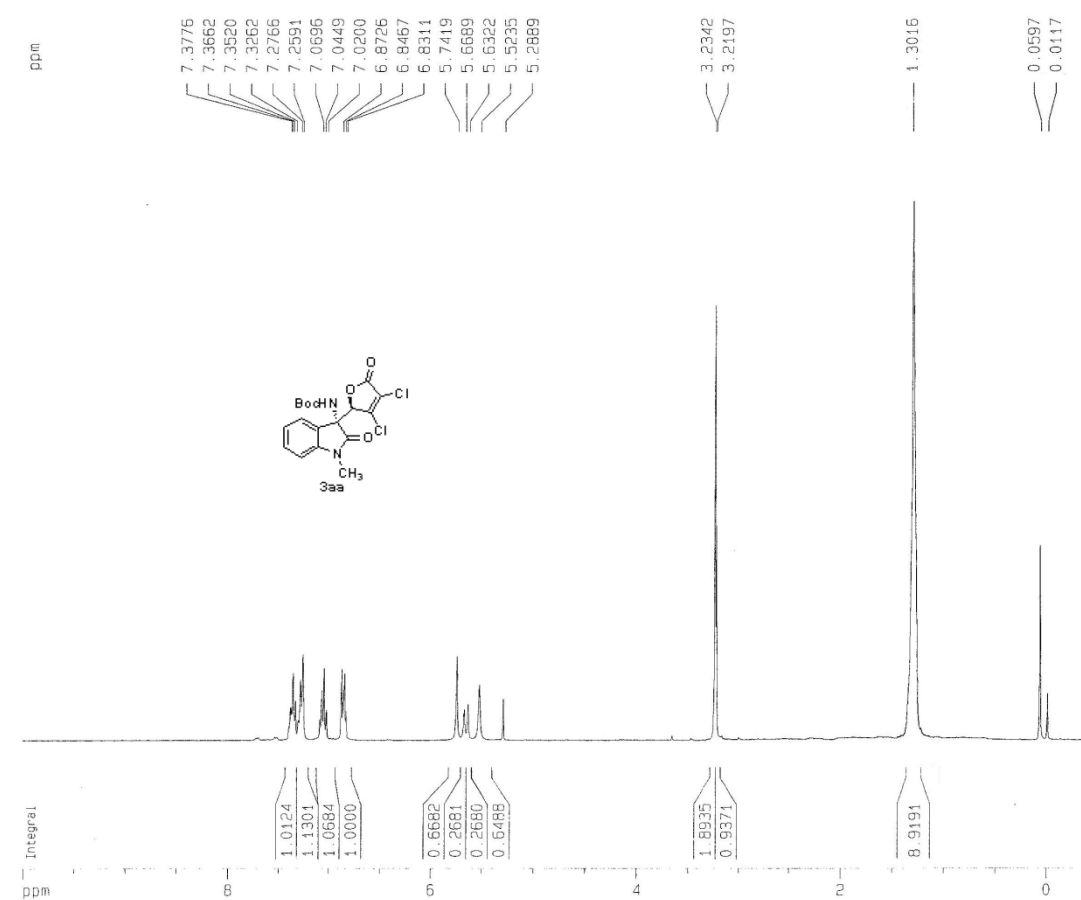
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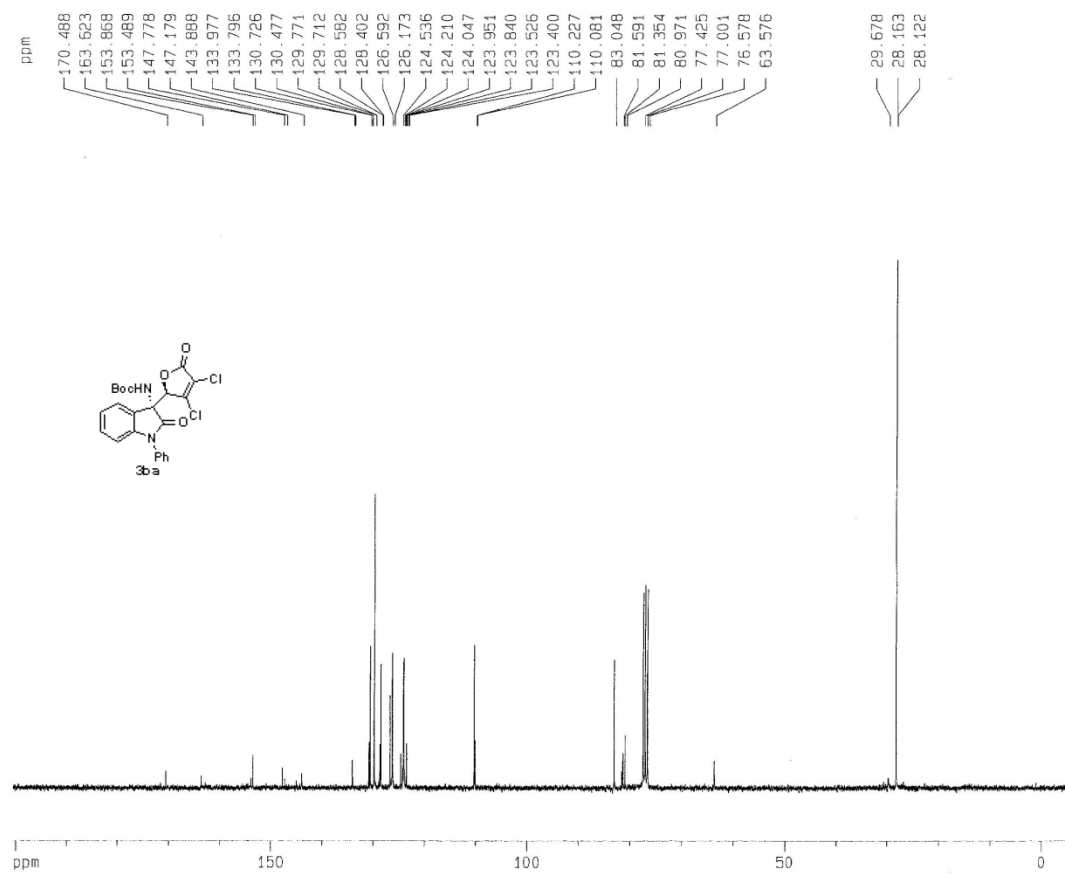
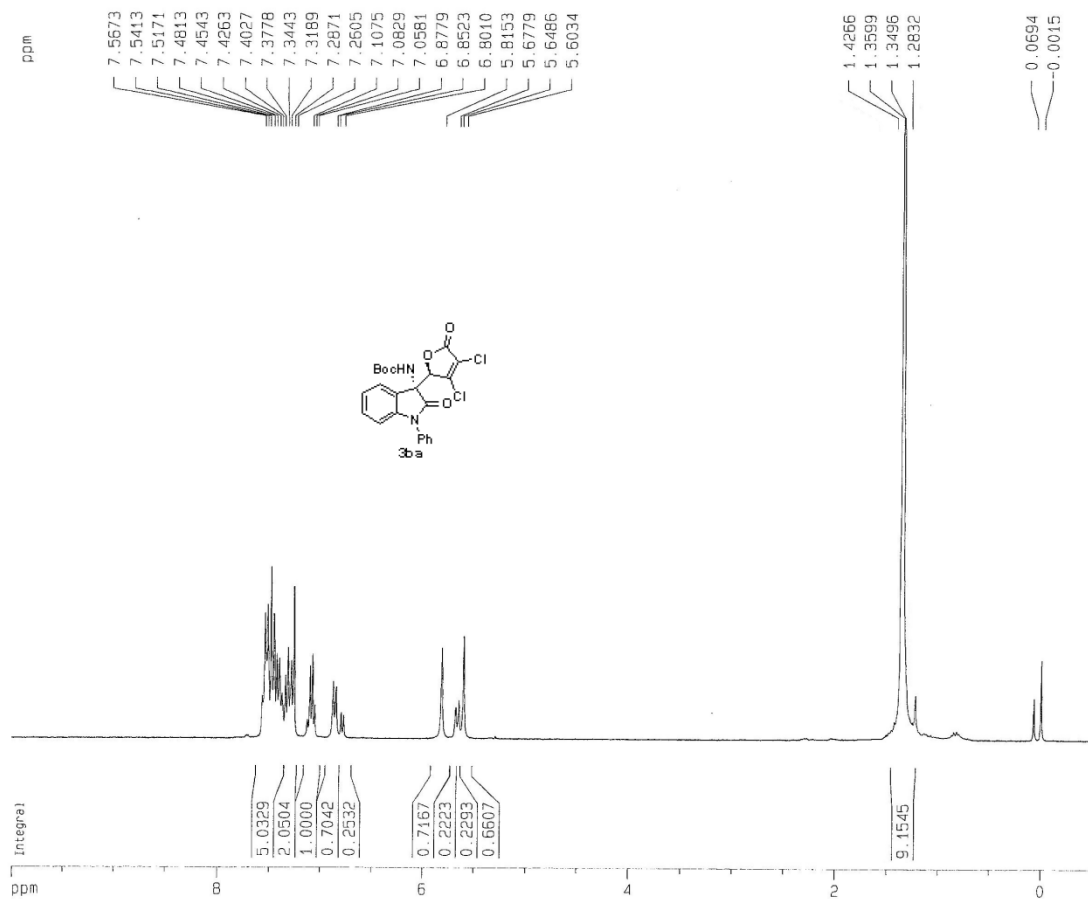
Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y+1/2,-z+3/2

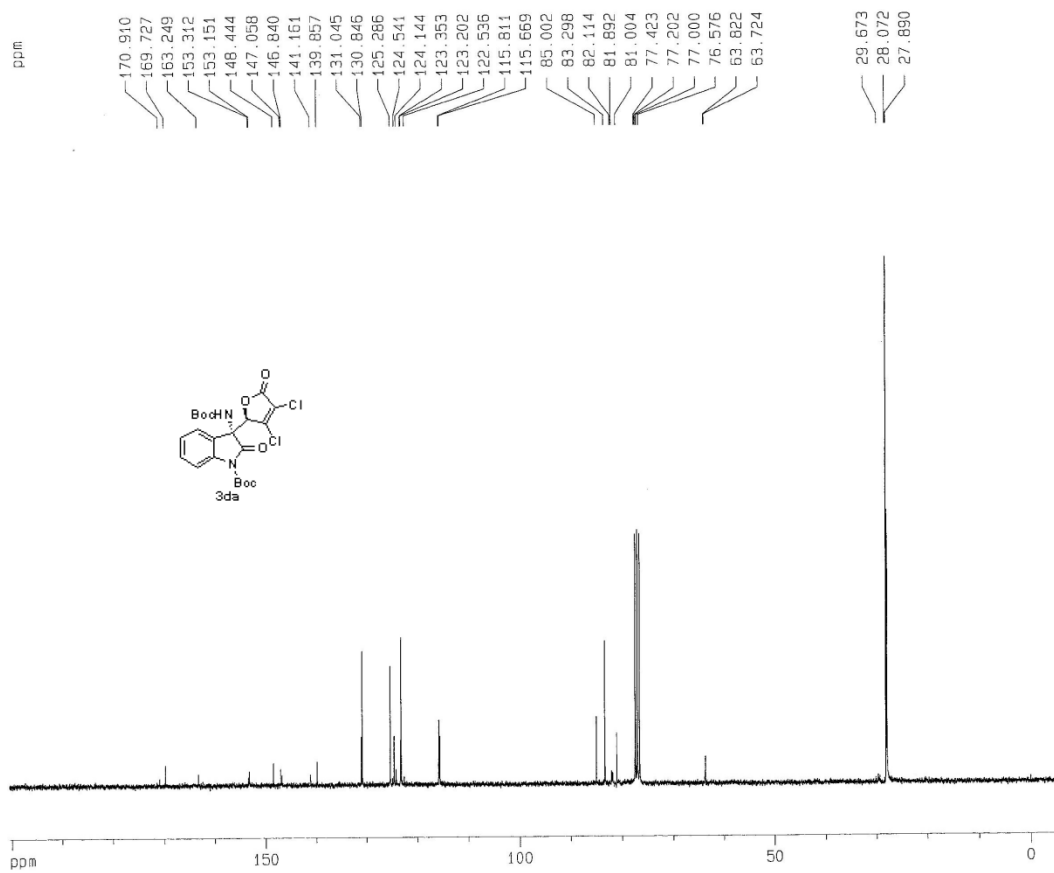
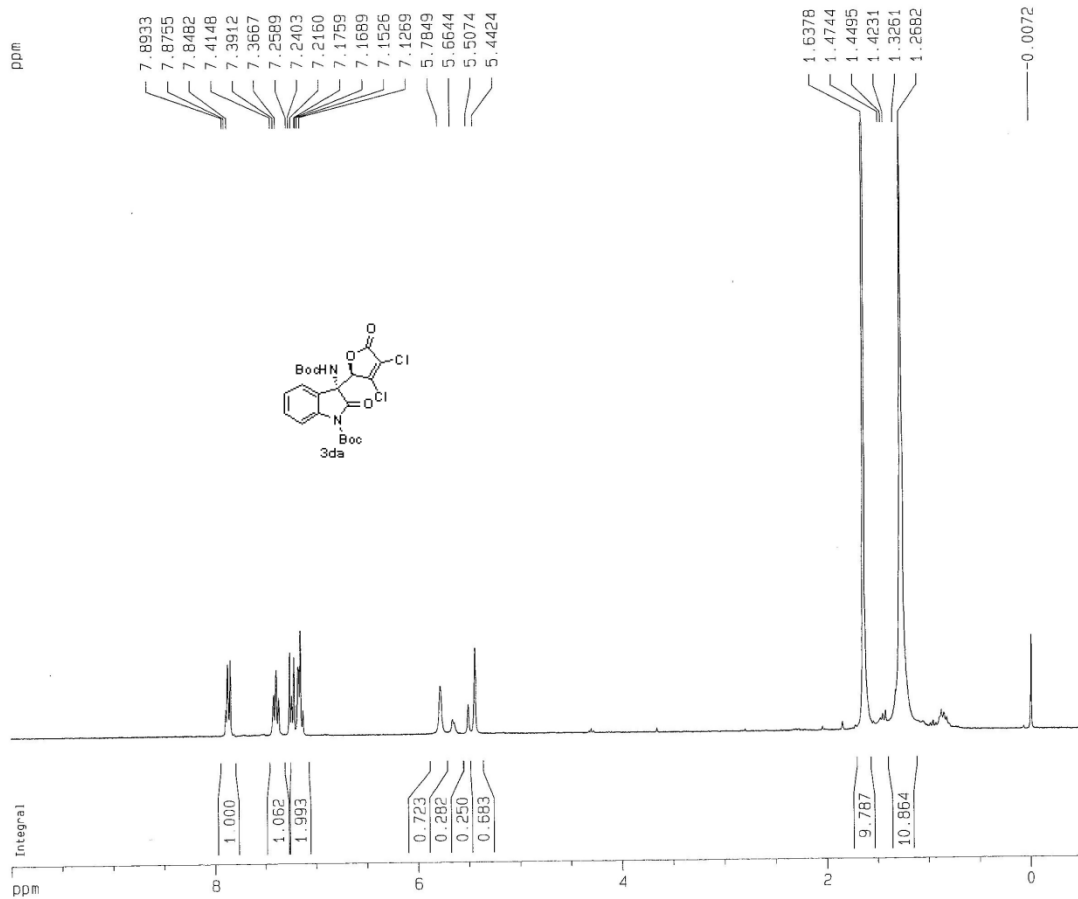


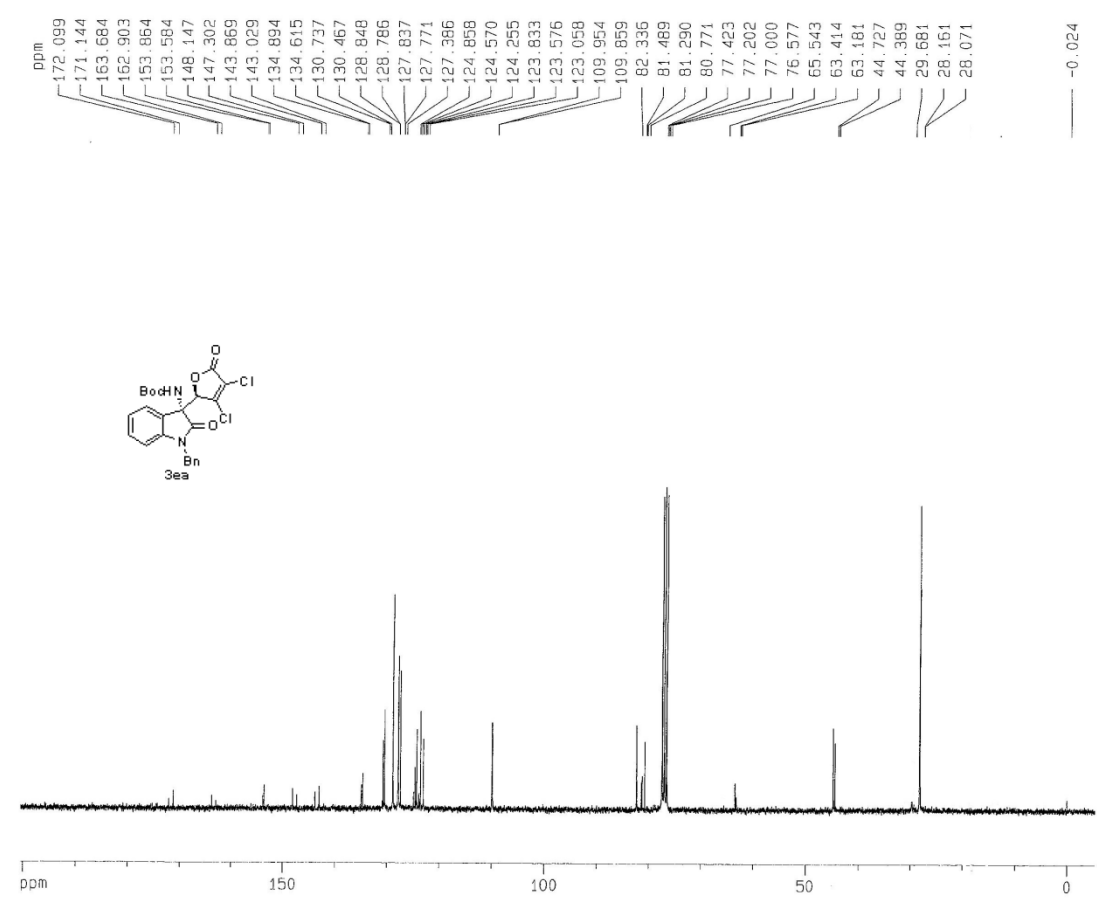
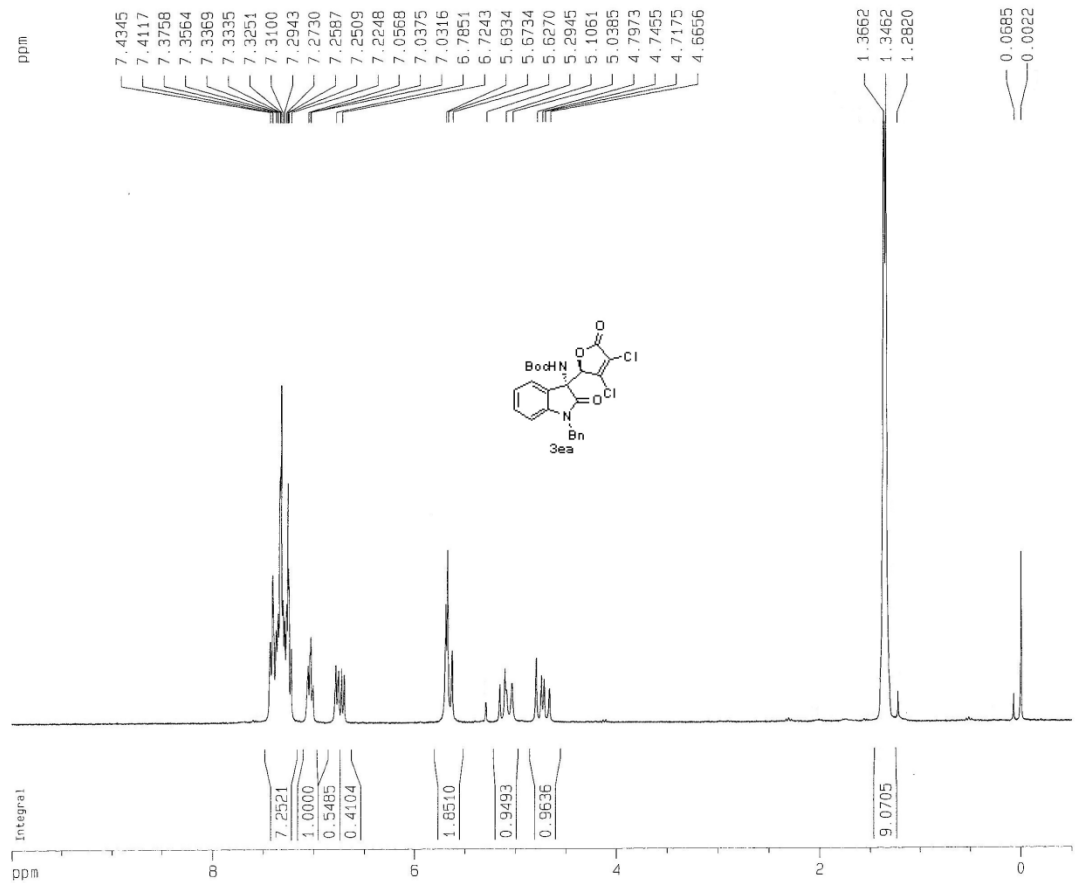
# NMR Spectra of Products

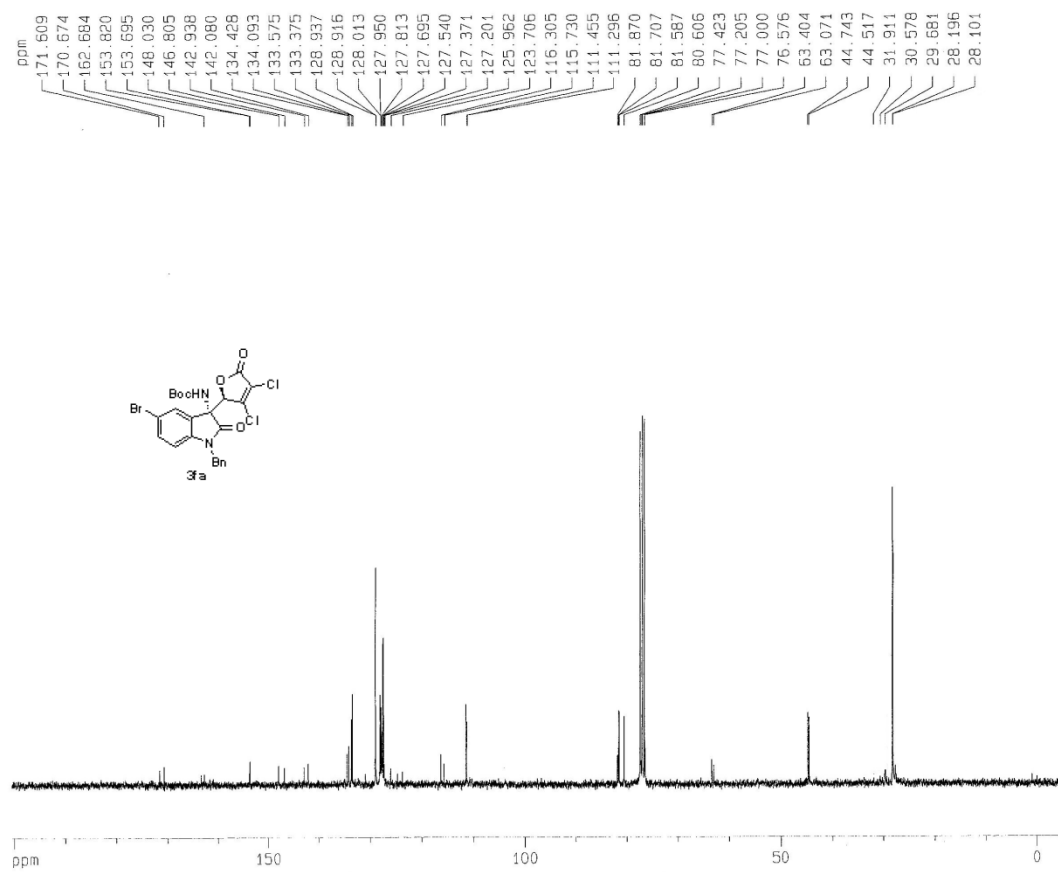
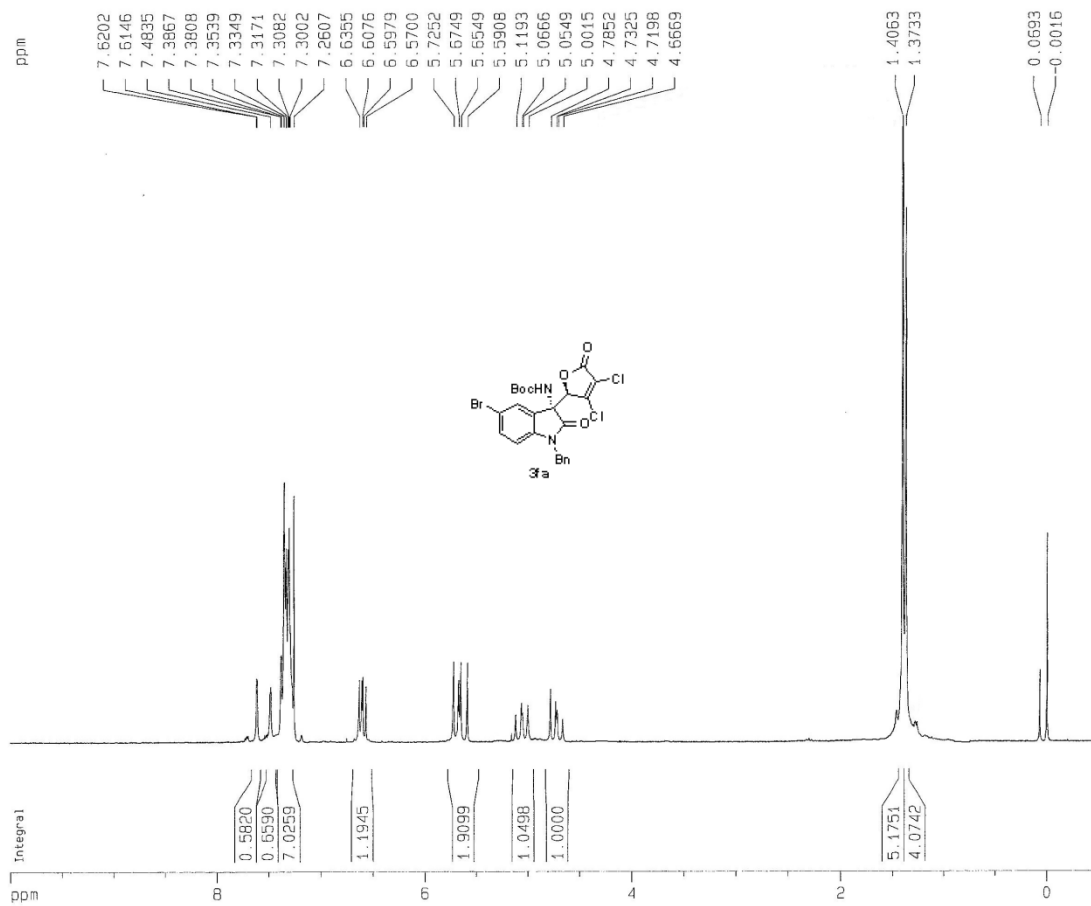


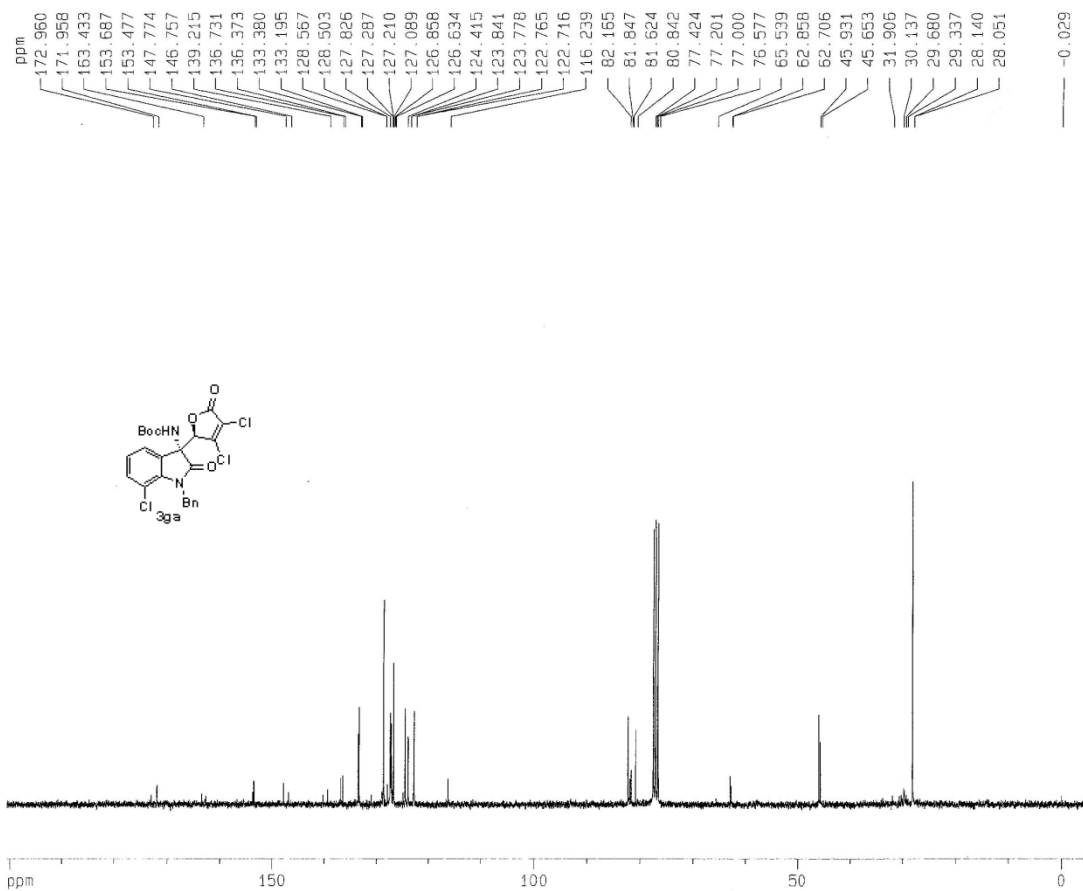
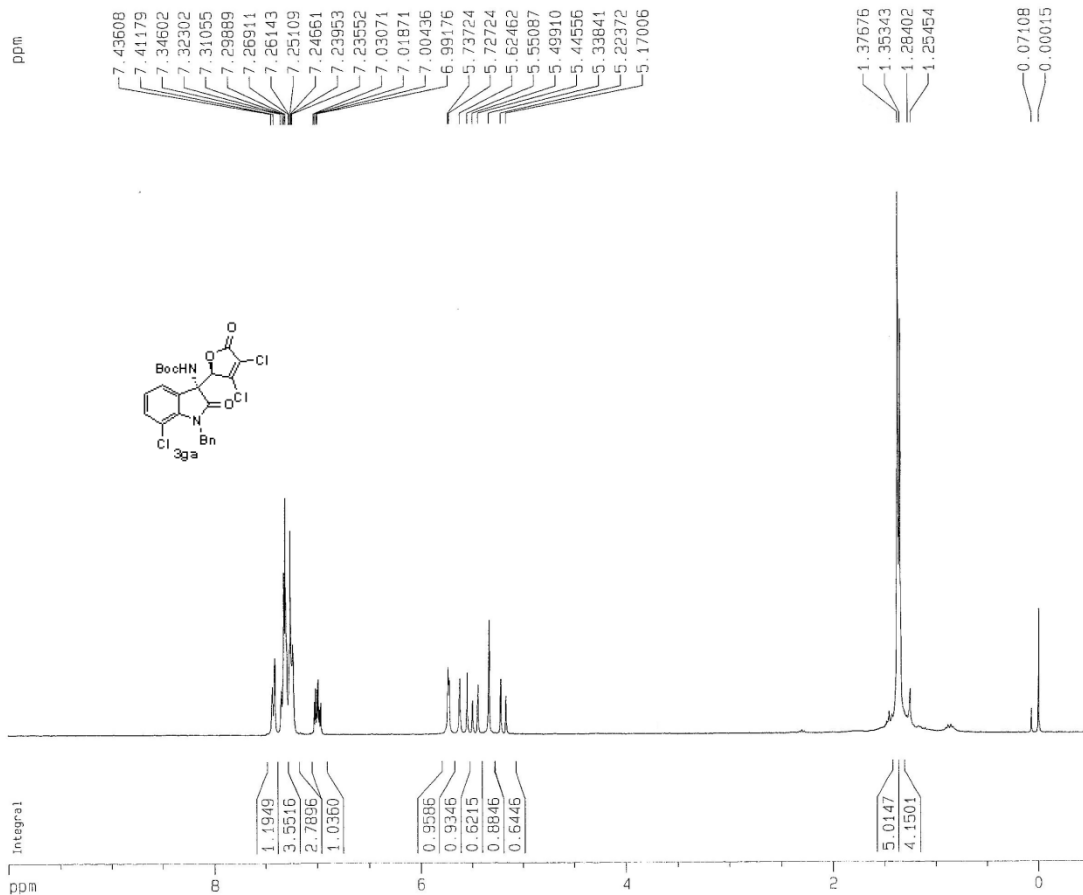


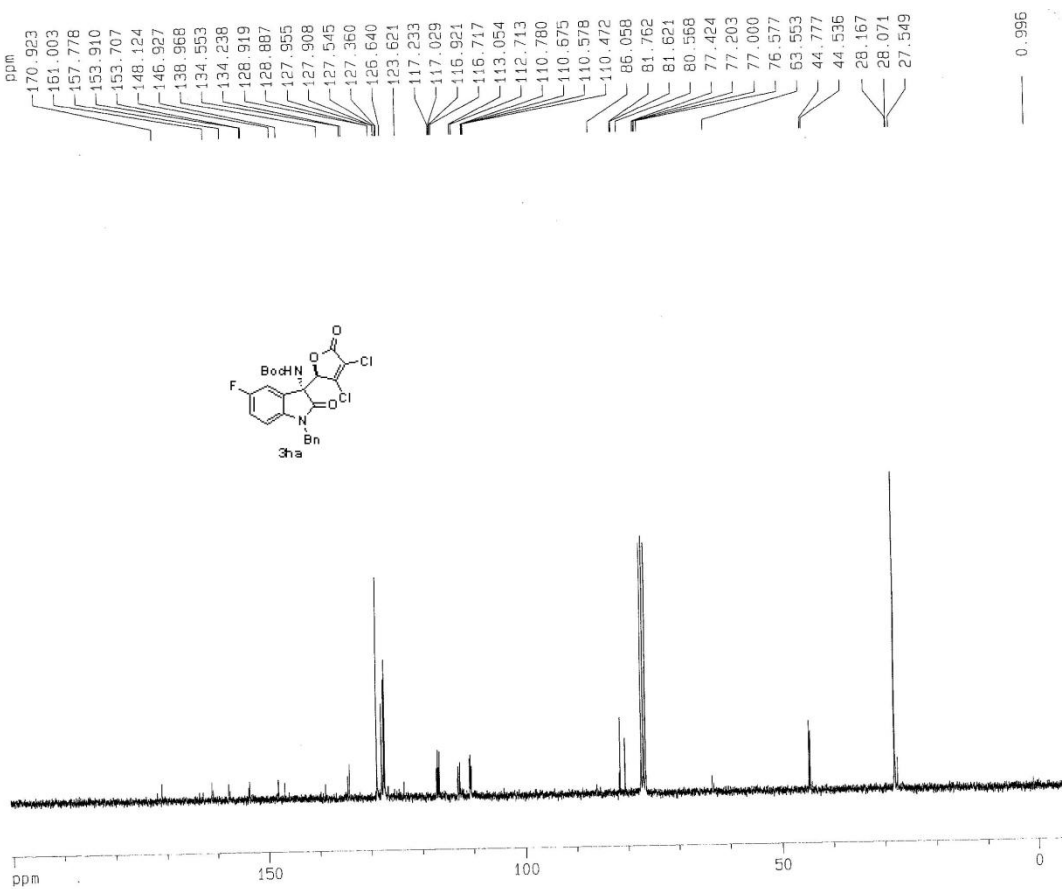
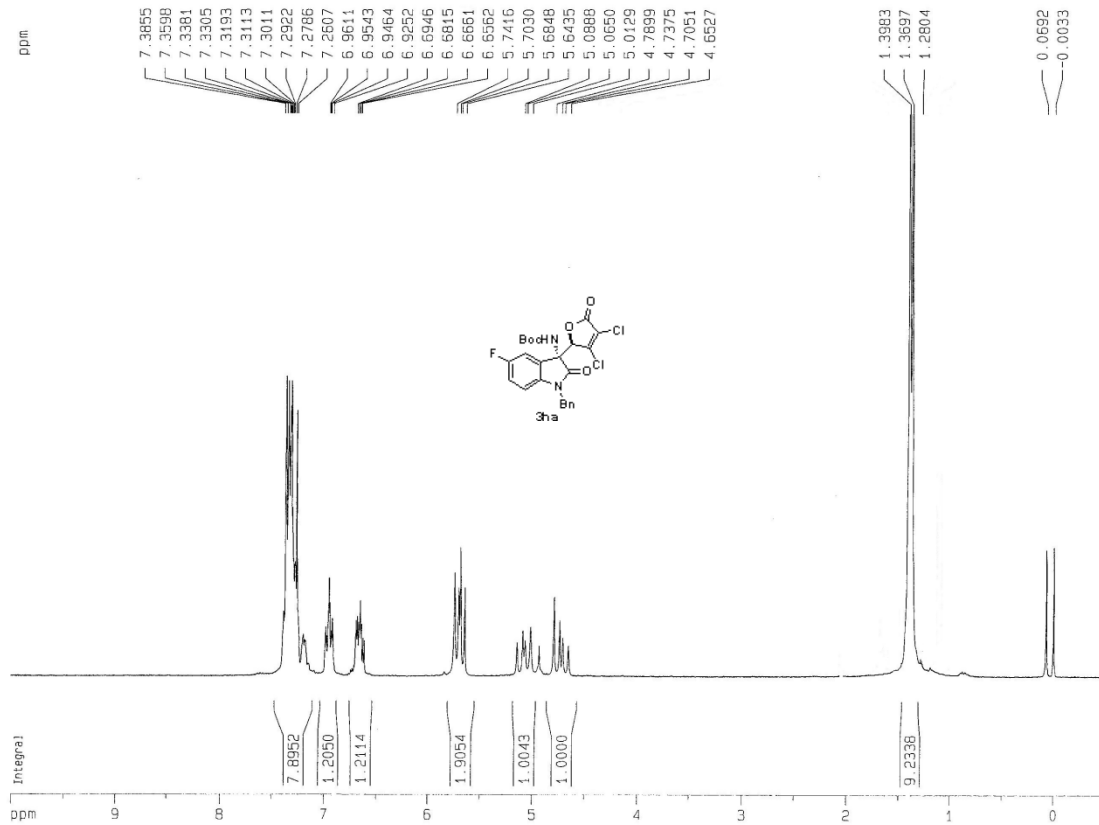




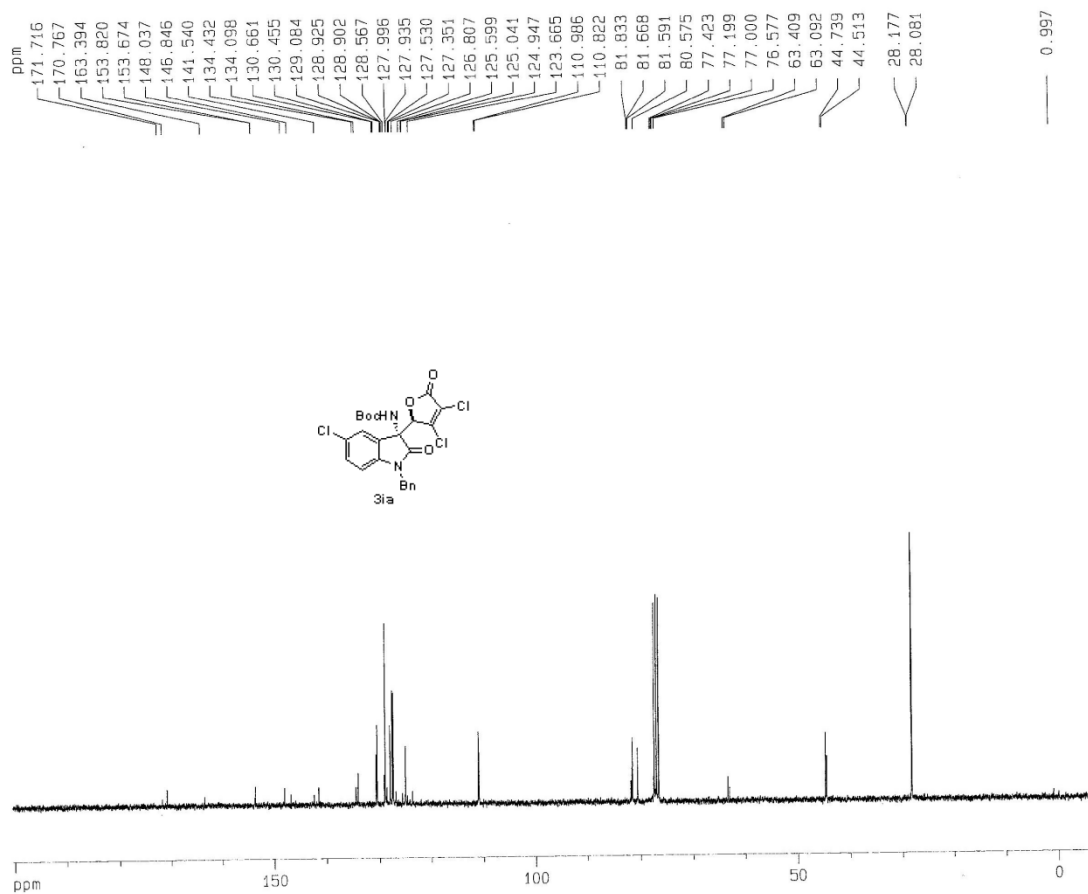
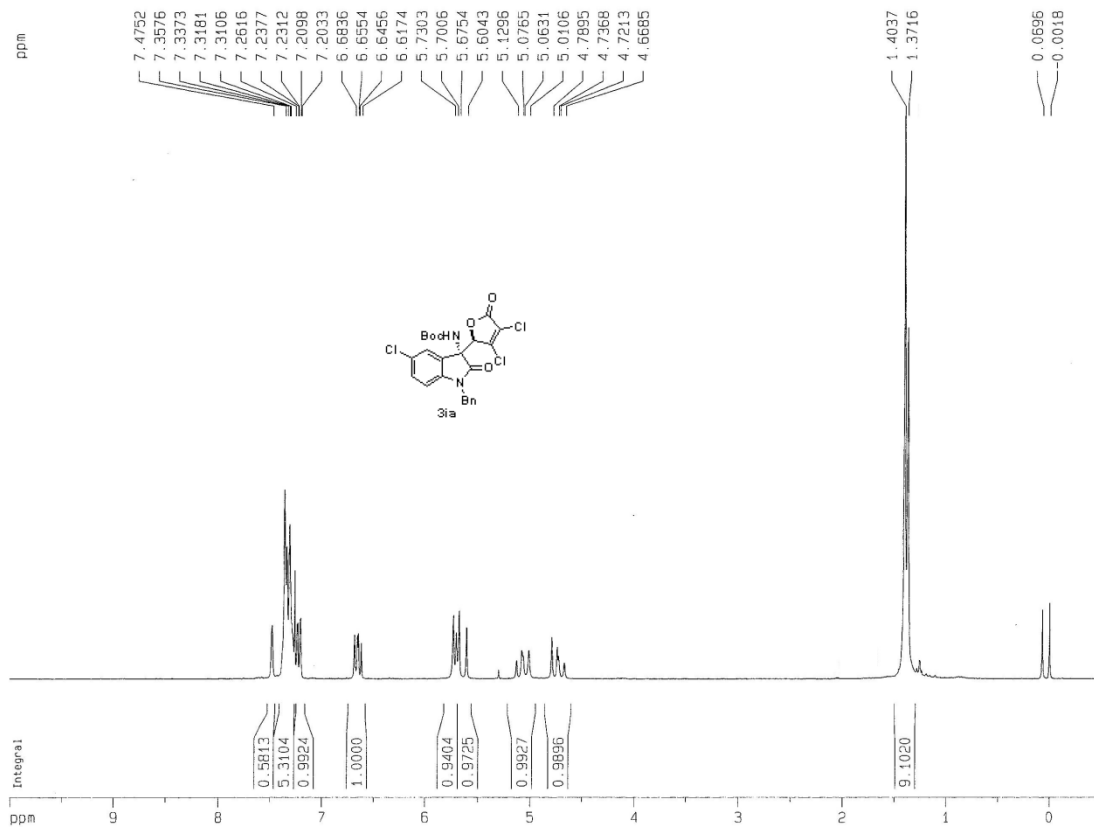




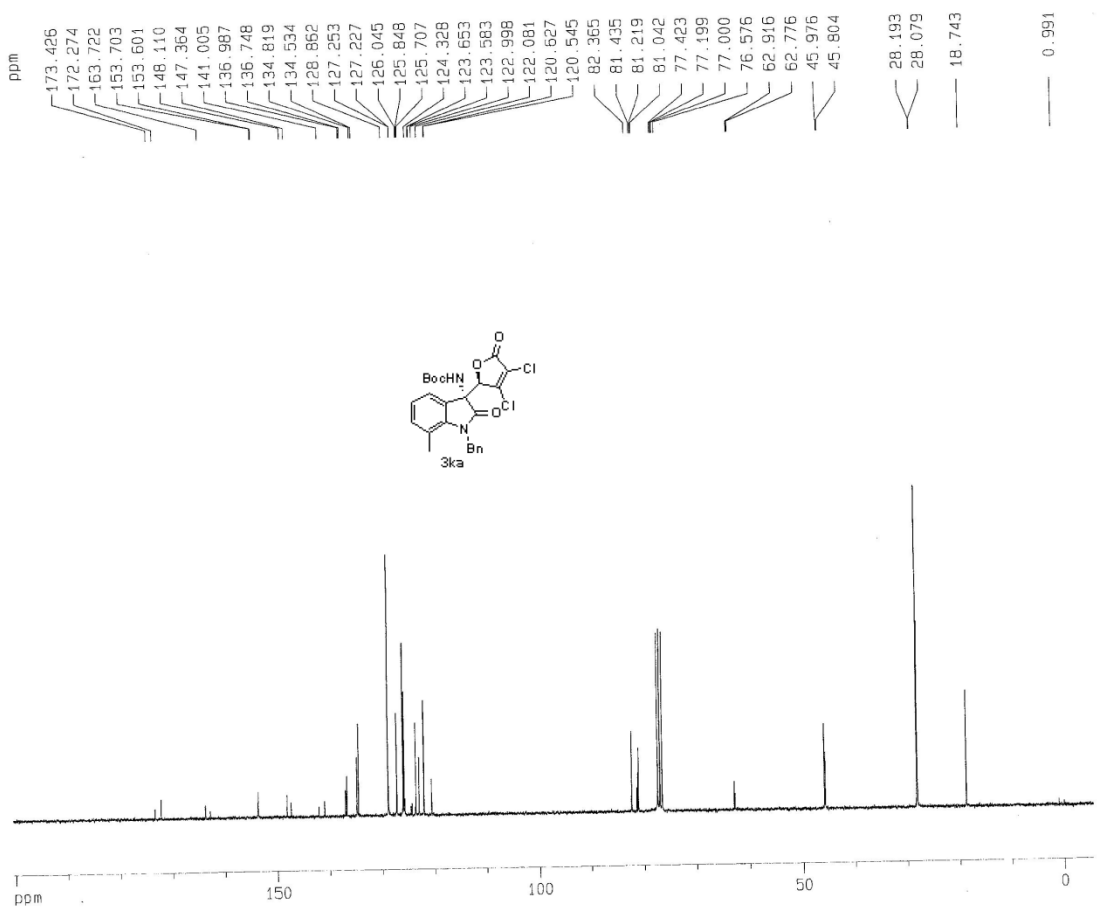
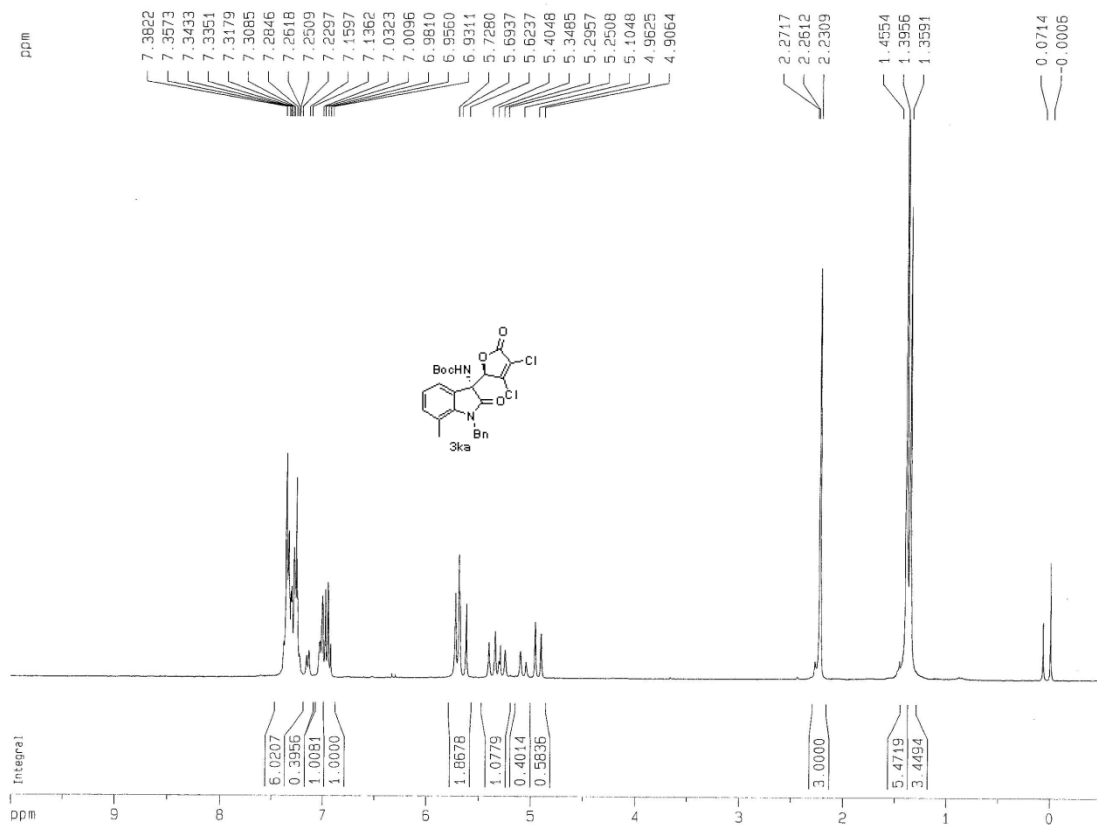


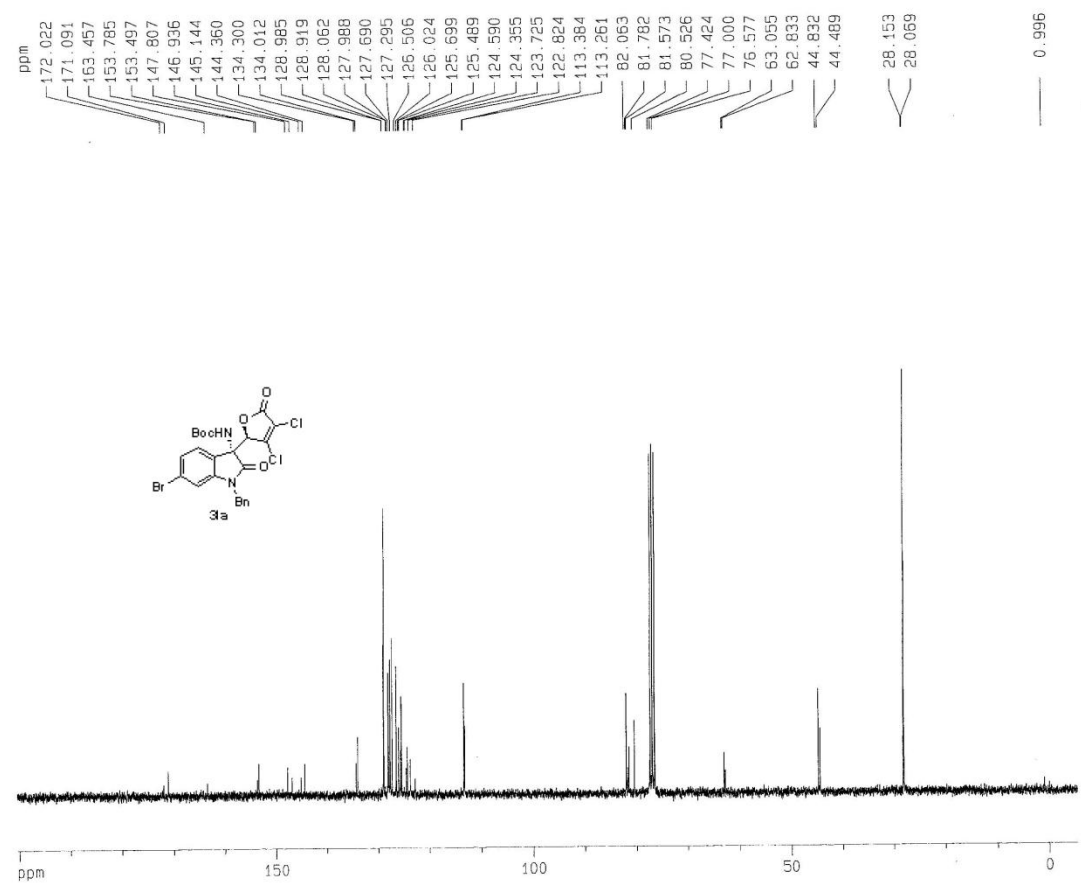
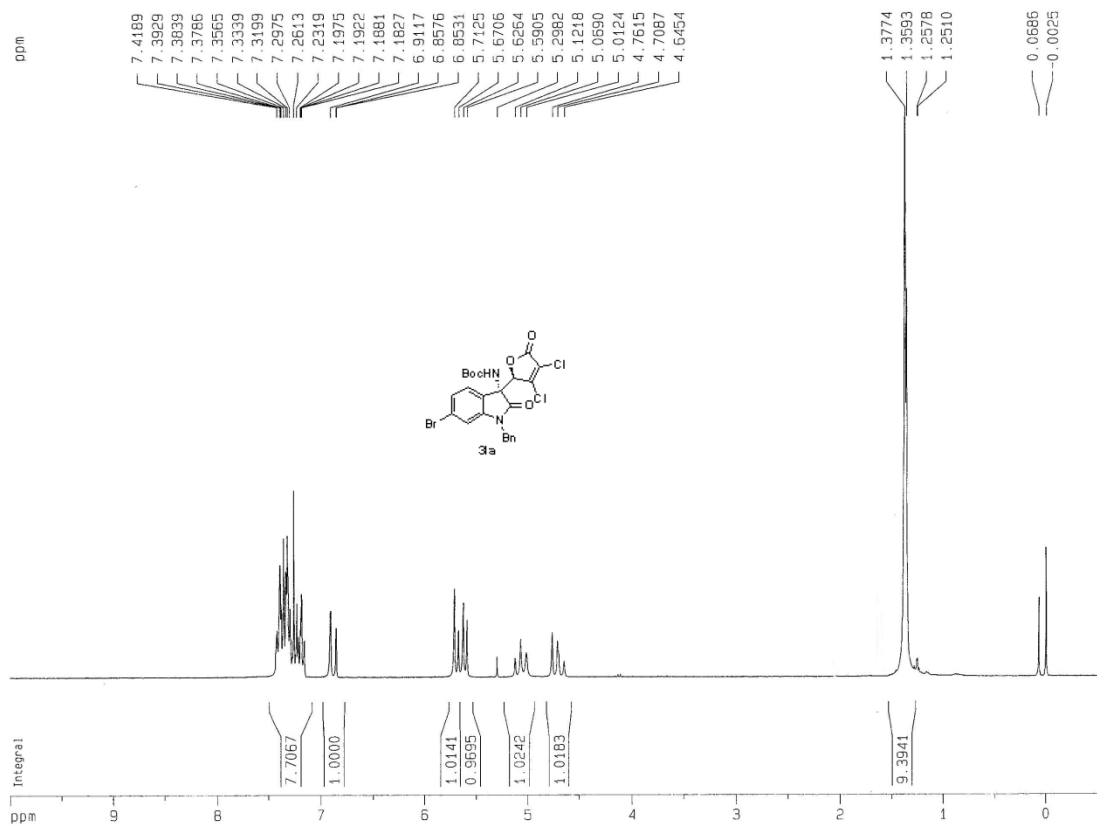


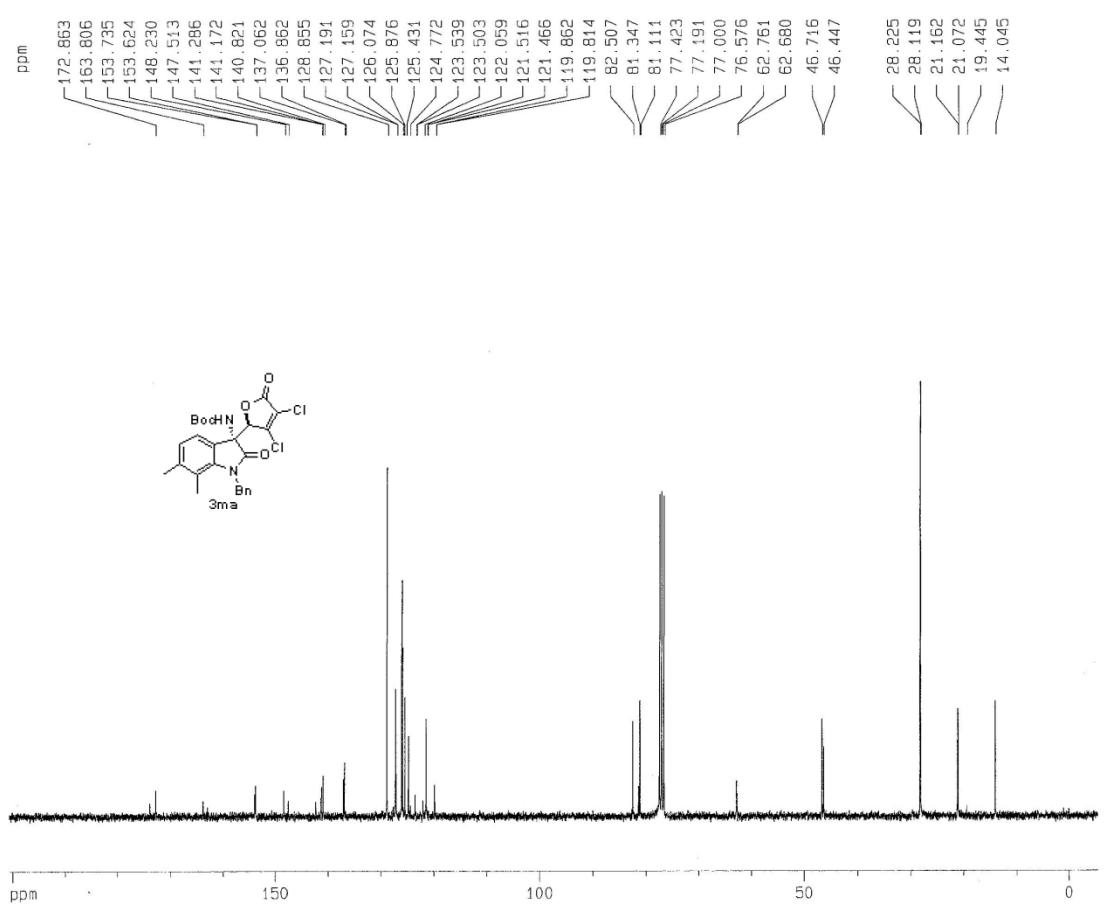
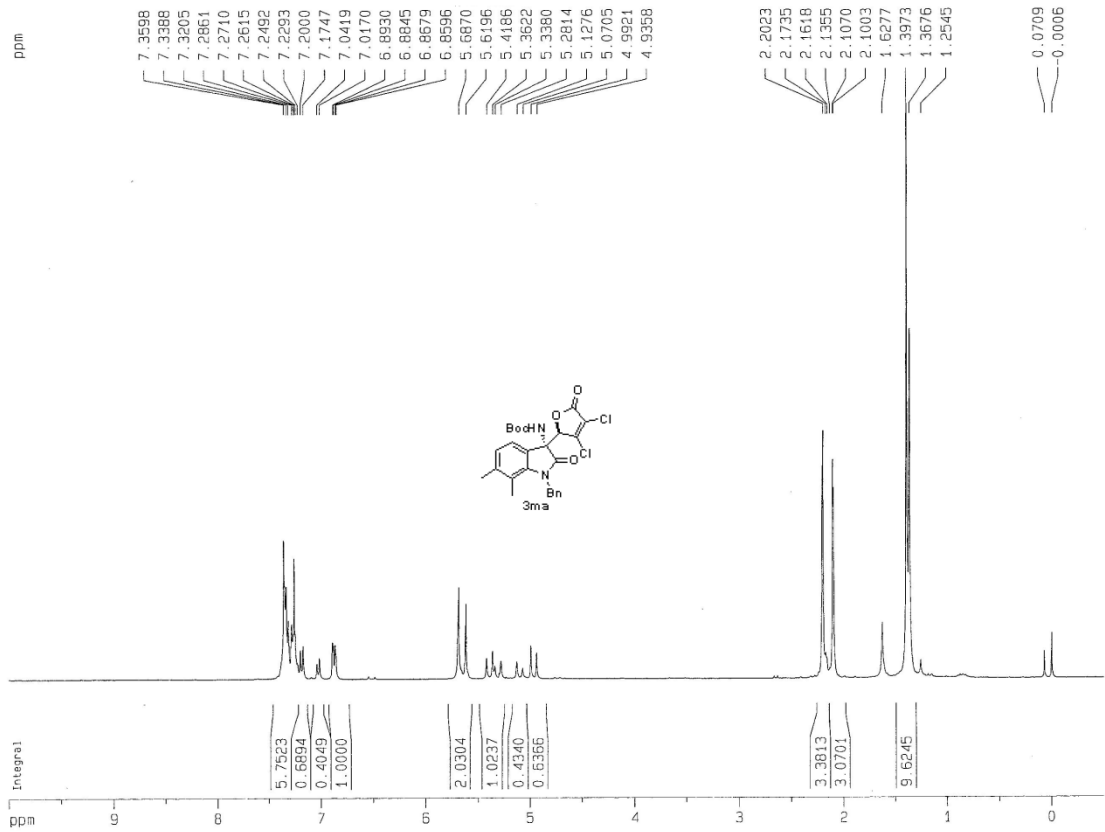


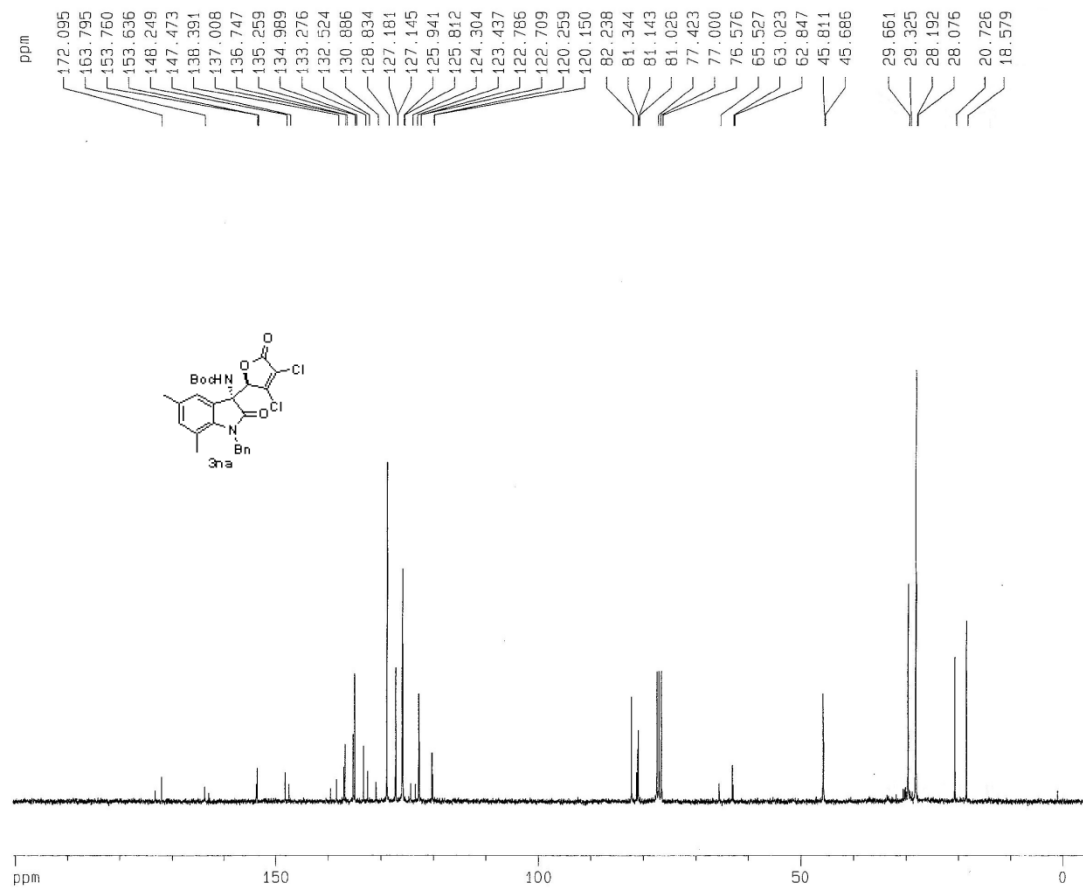
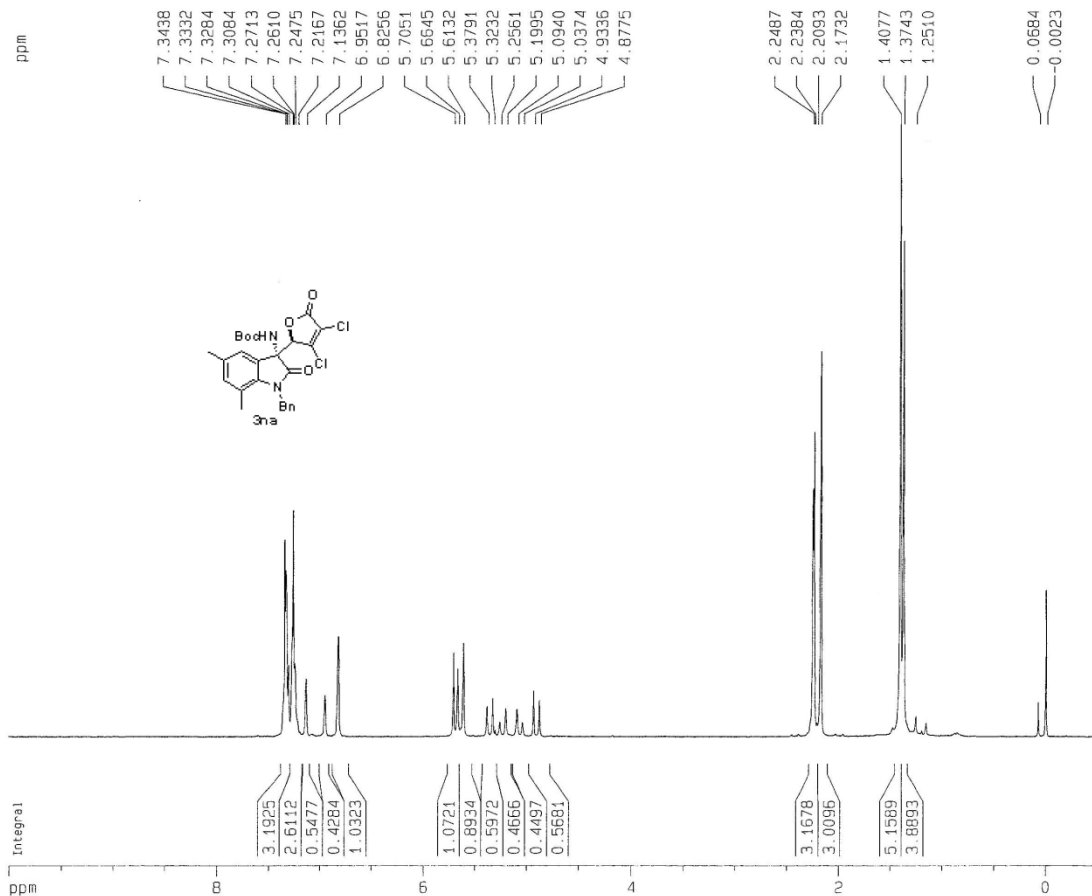


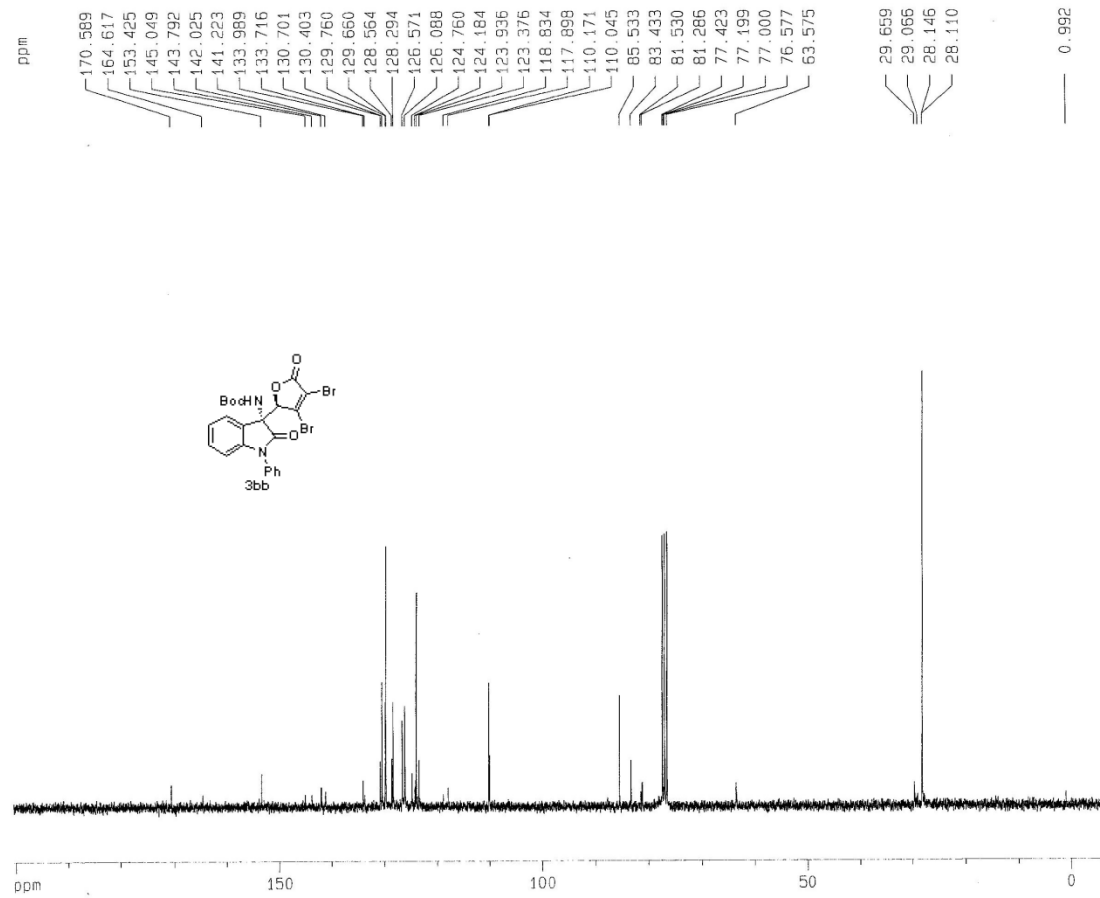
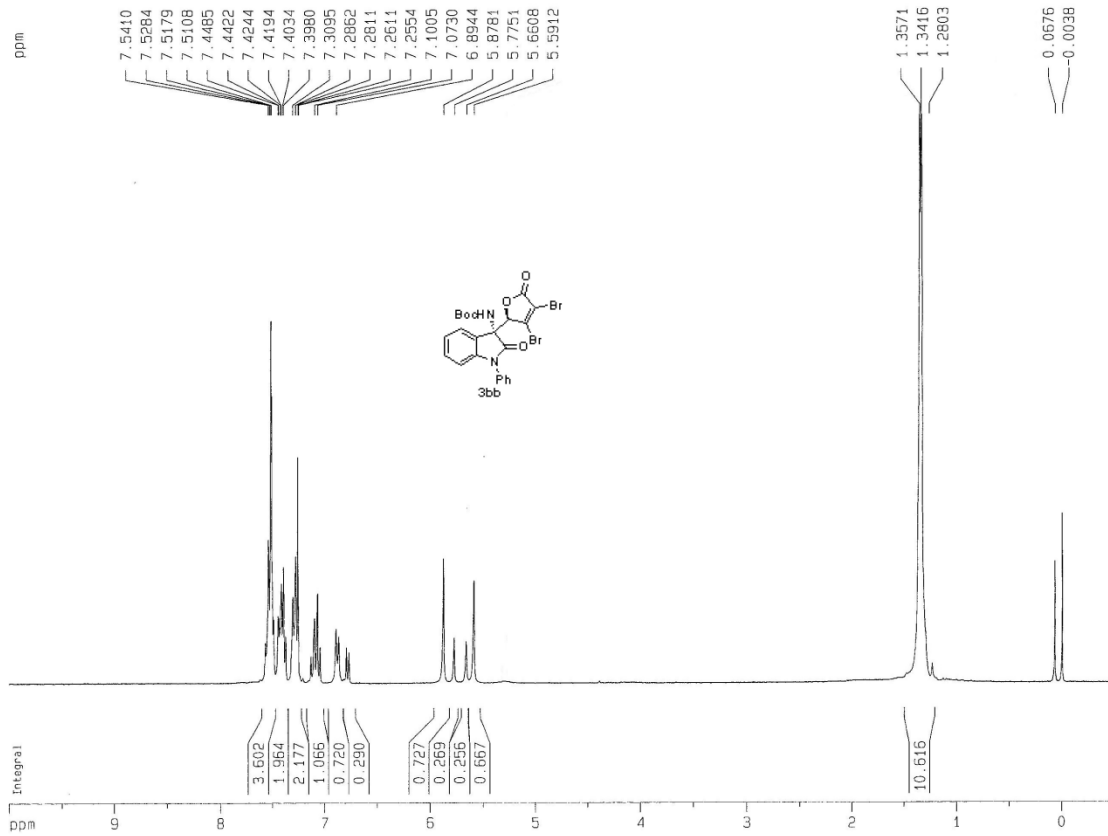


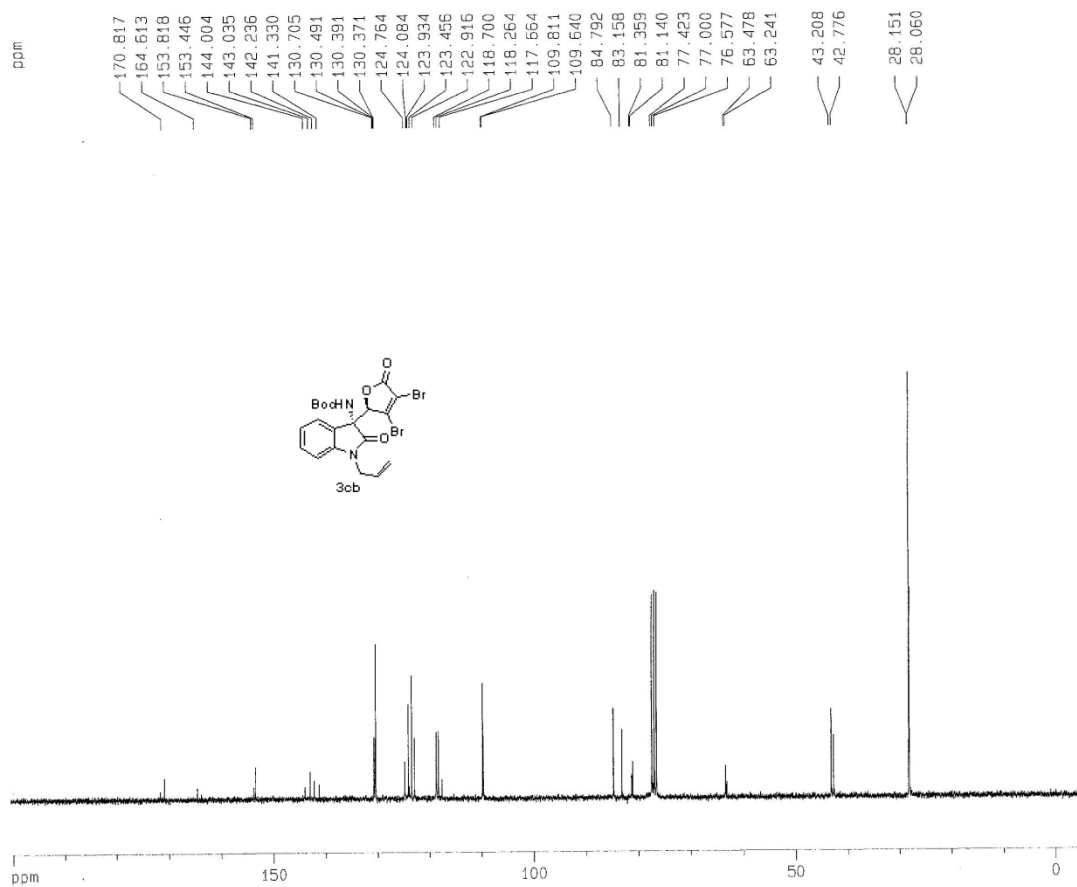
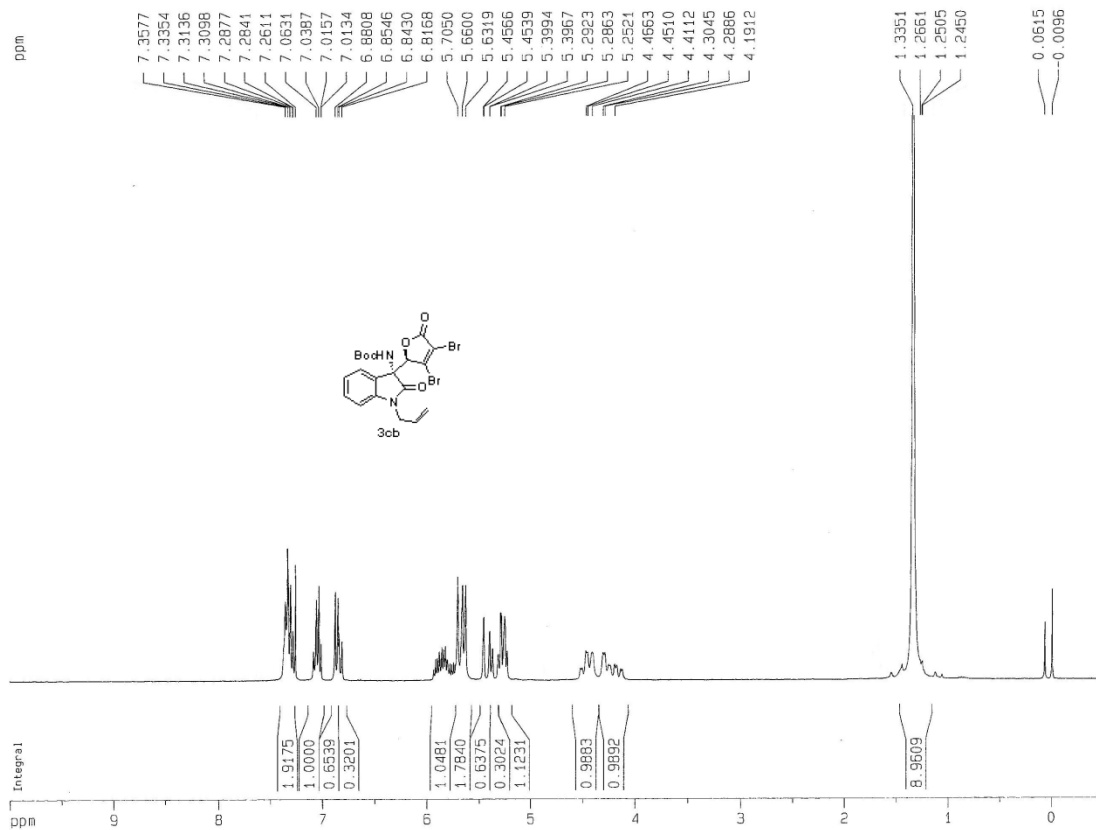




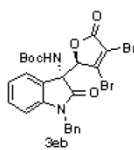
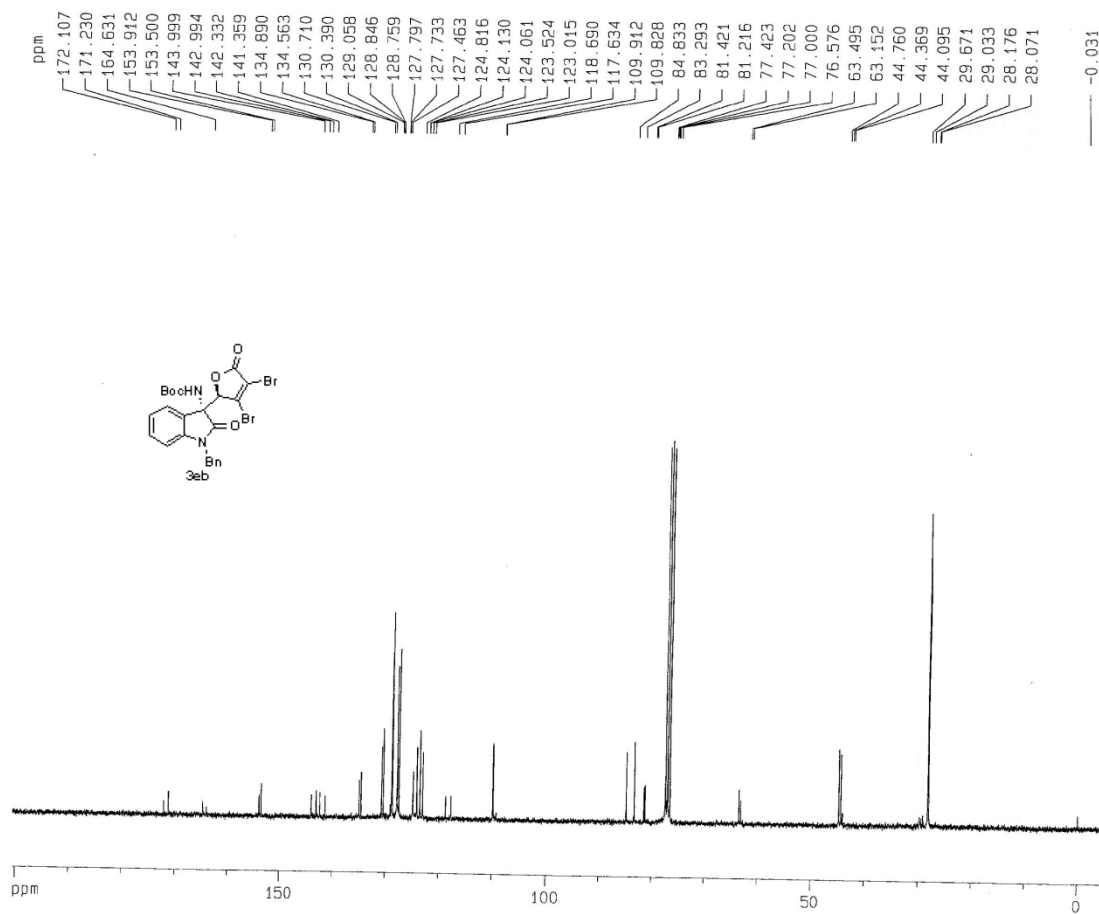
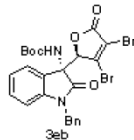
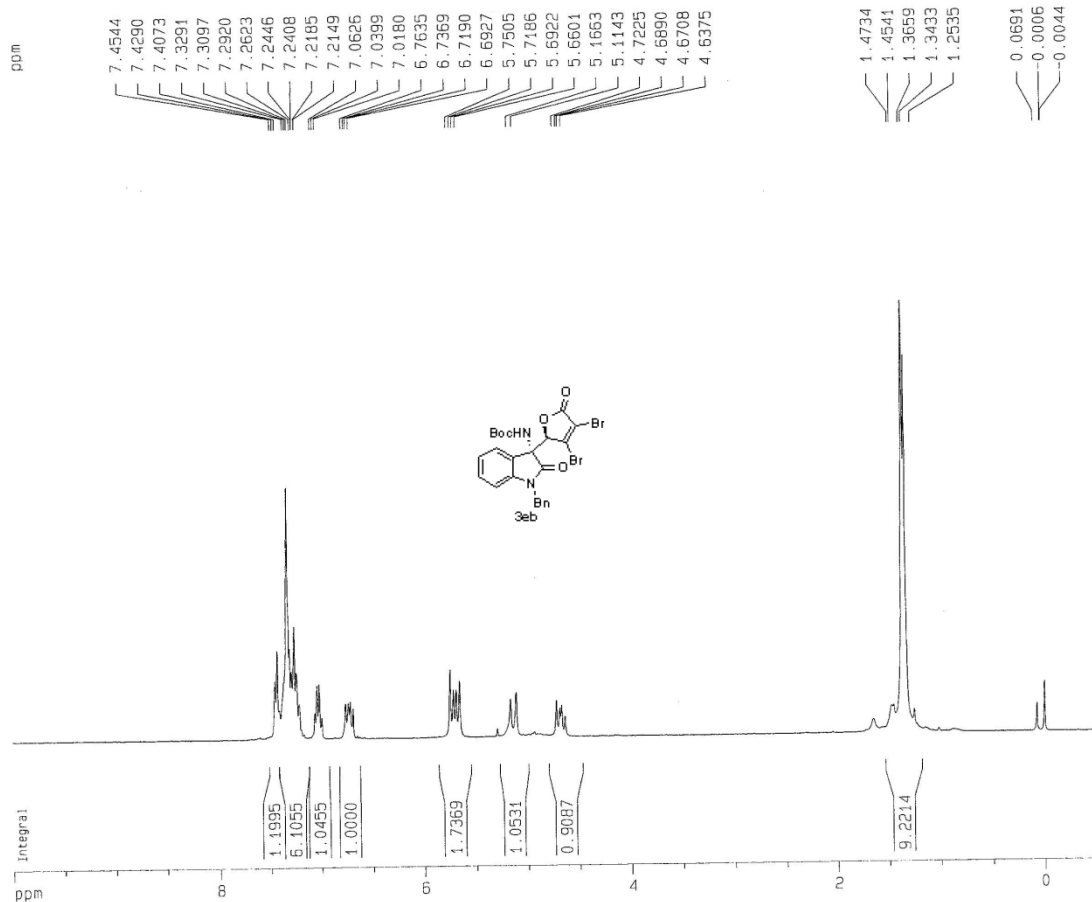


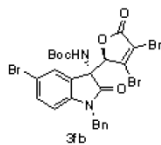
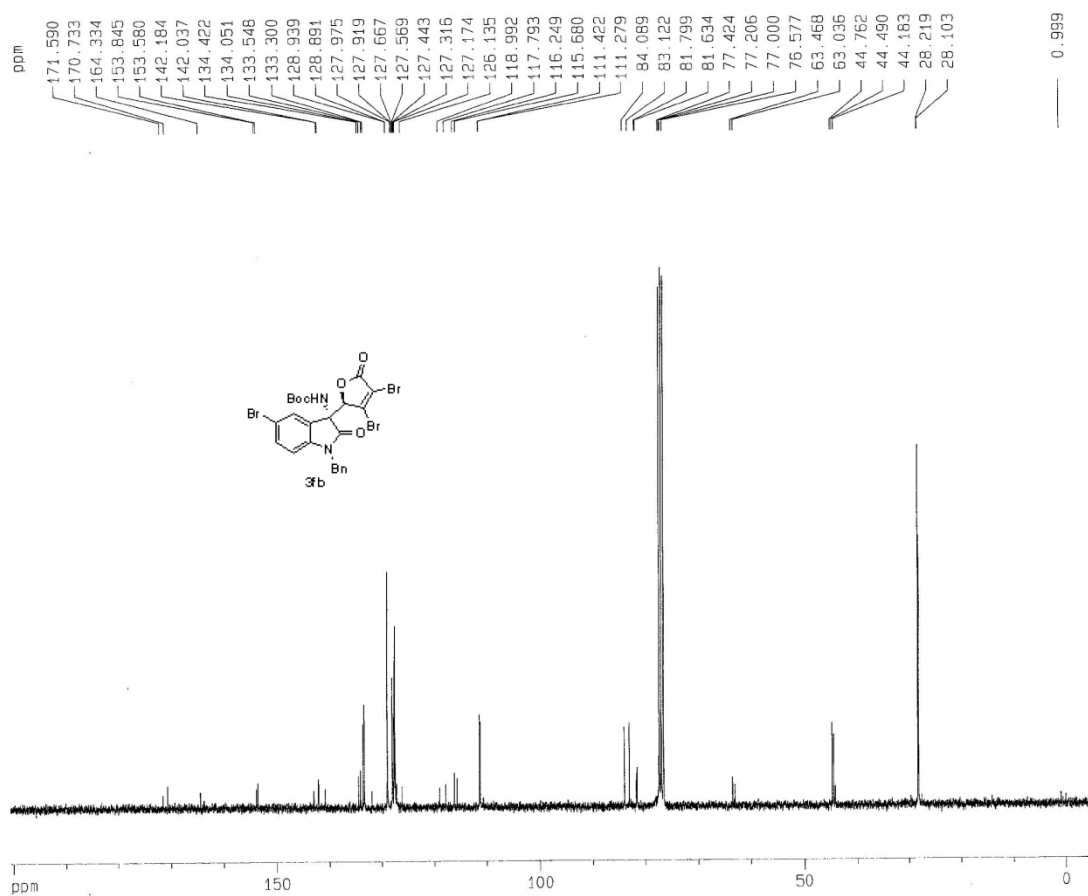
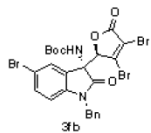
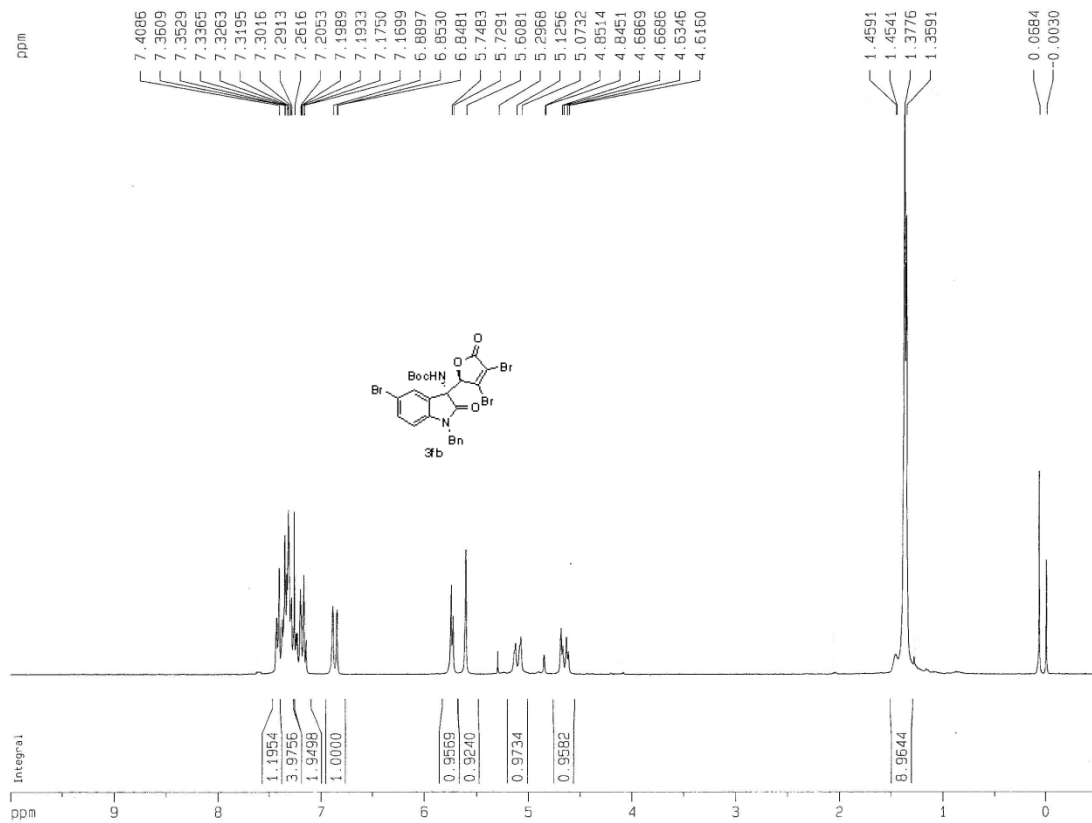


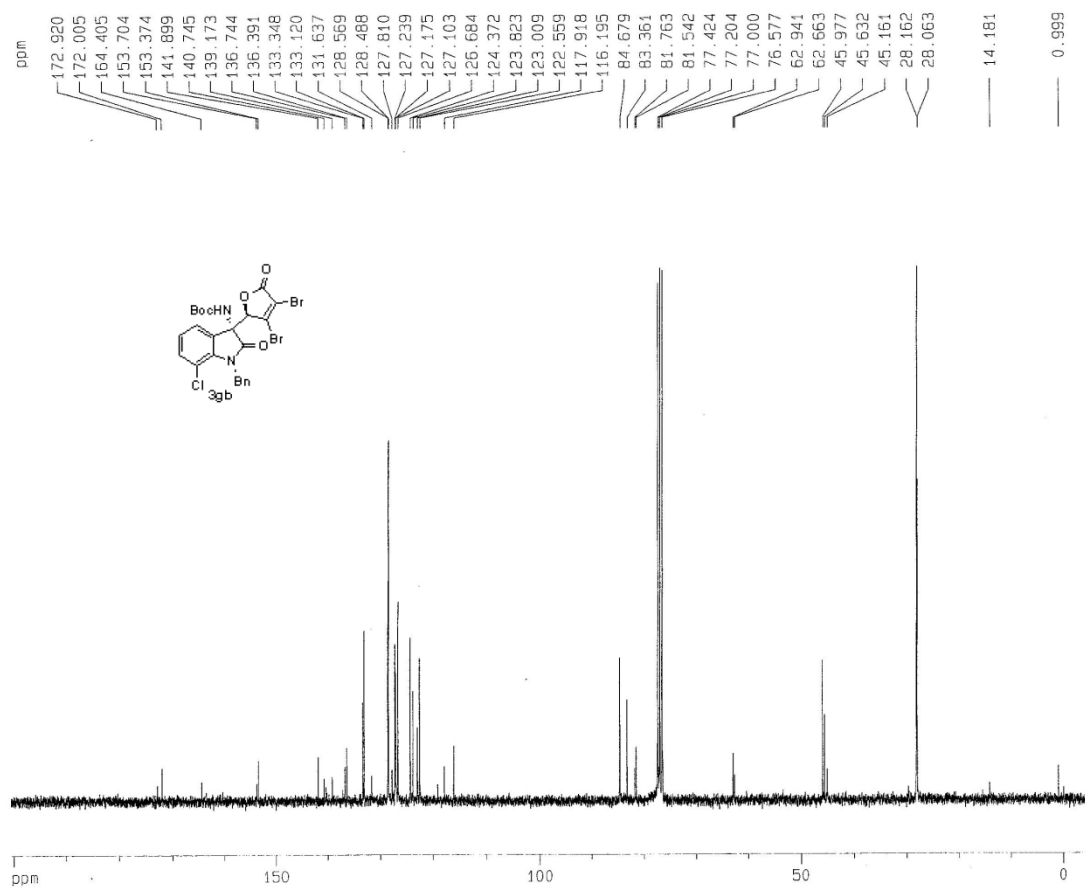
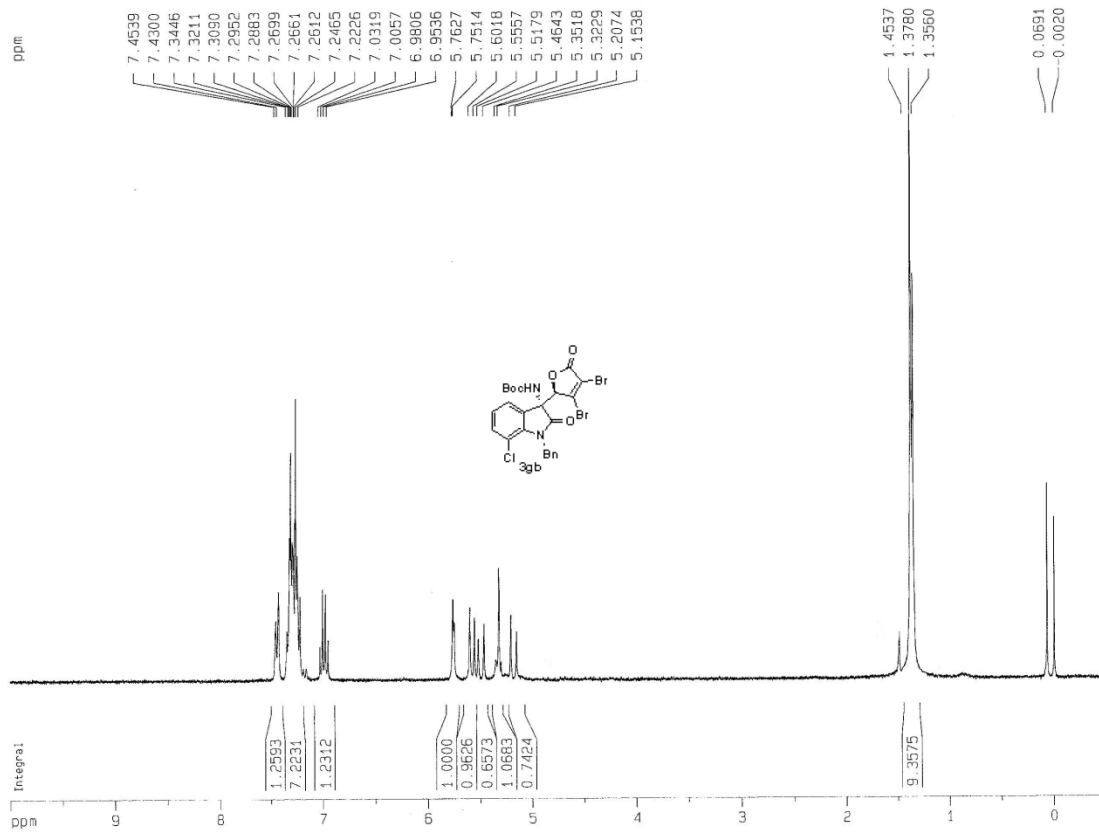


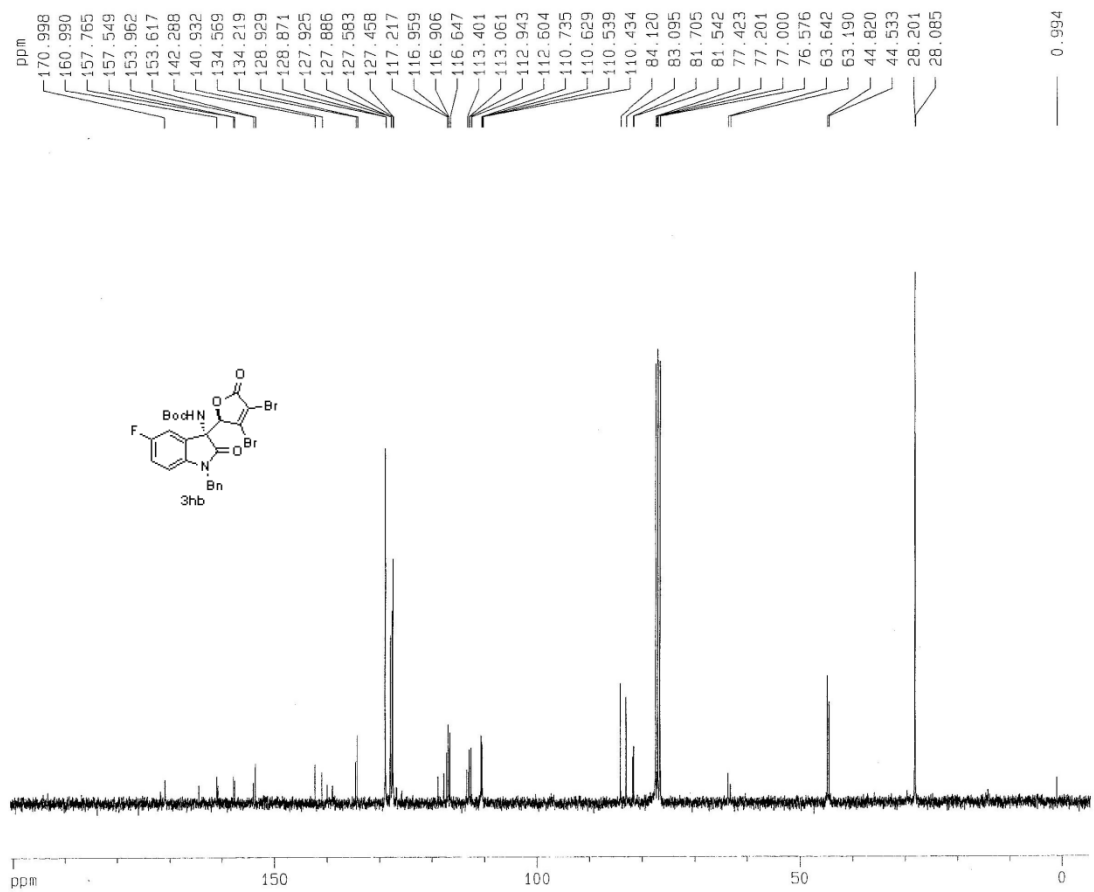
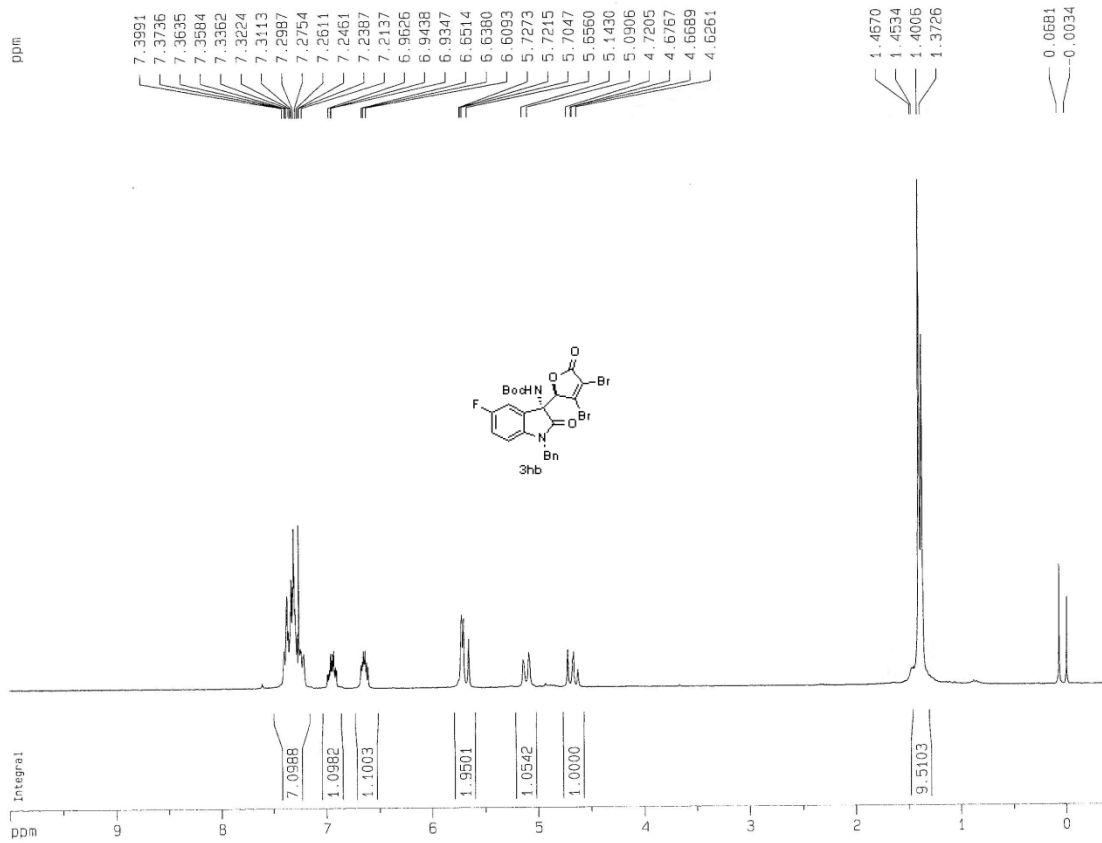


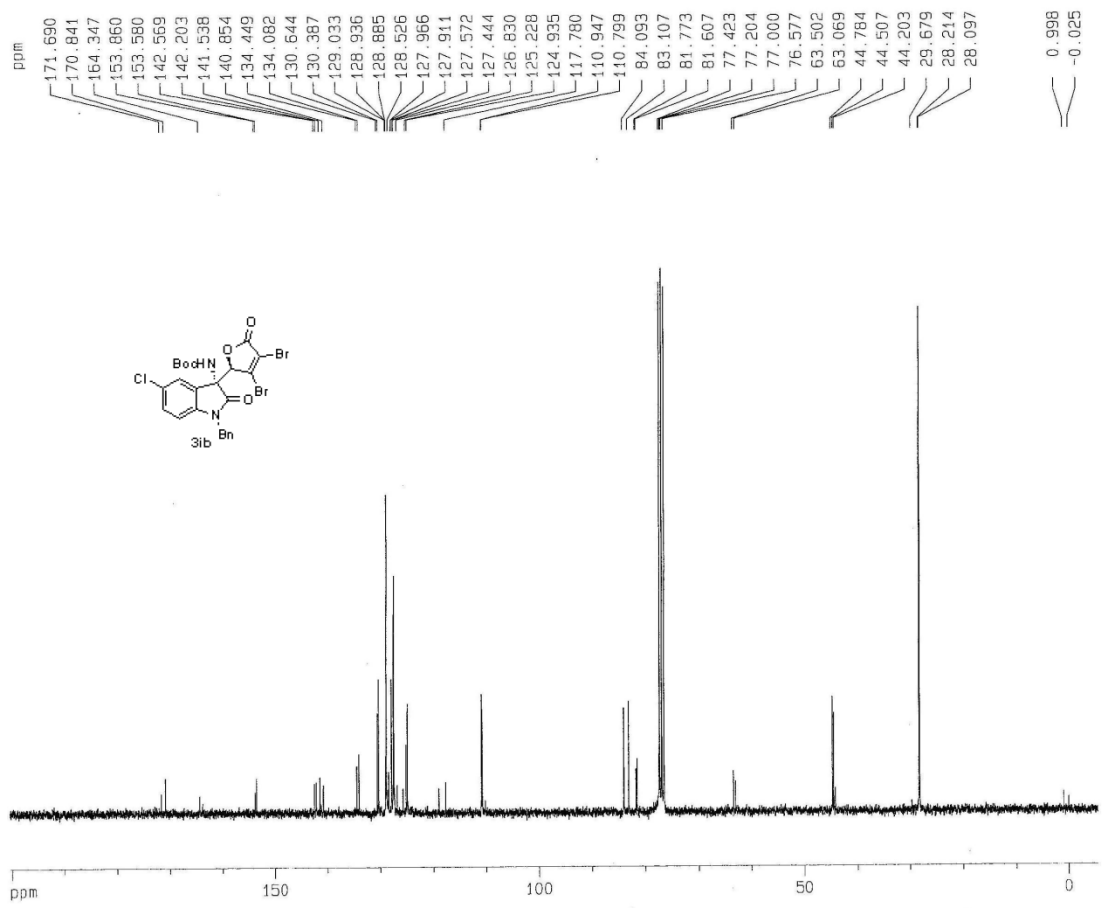
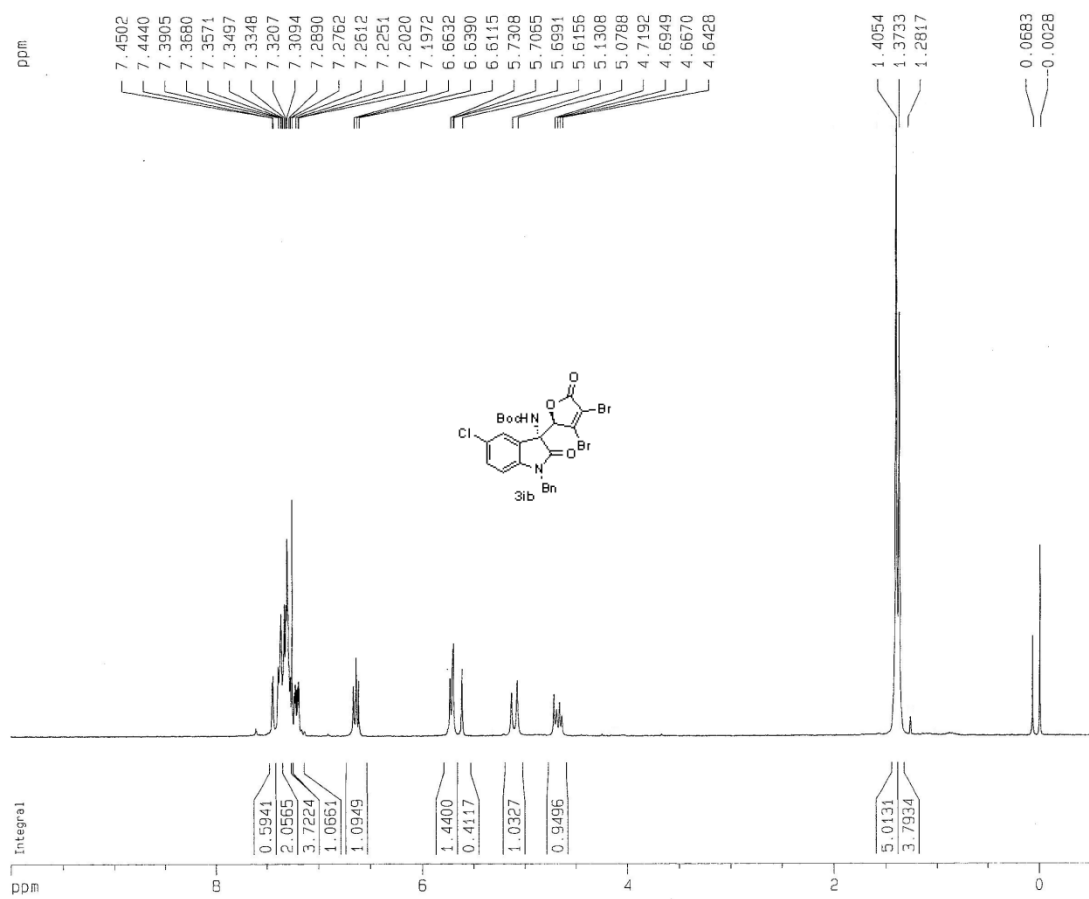


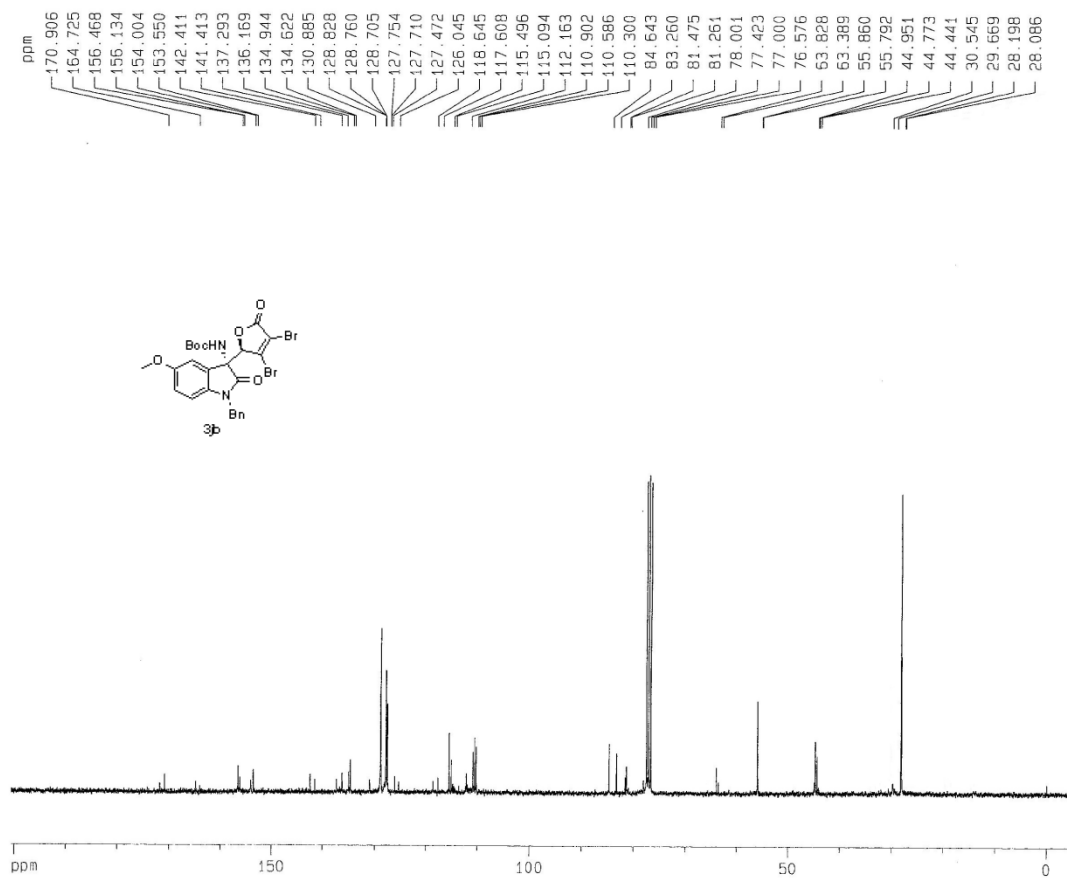
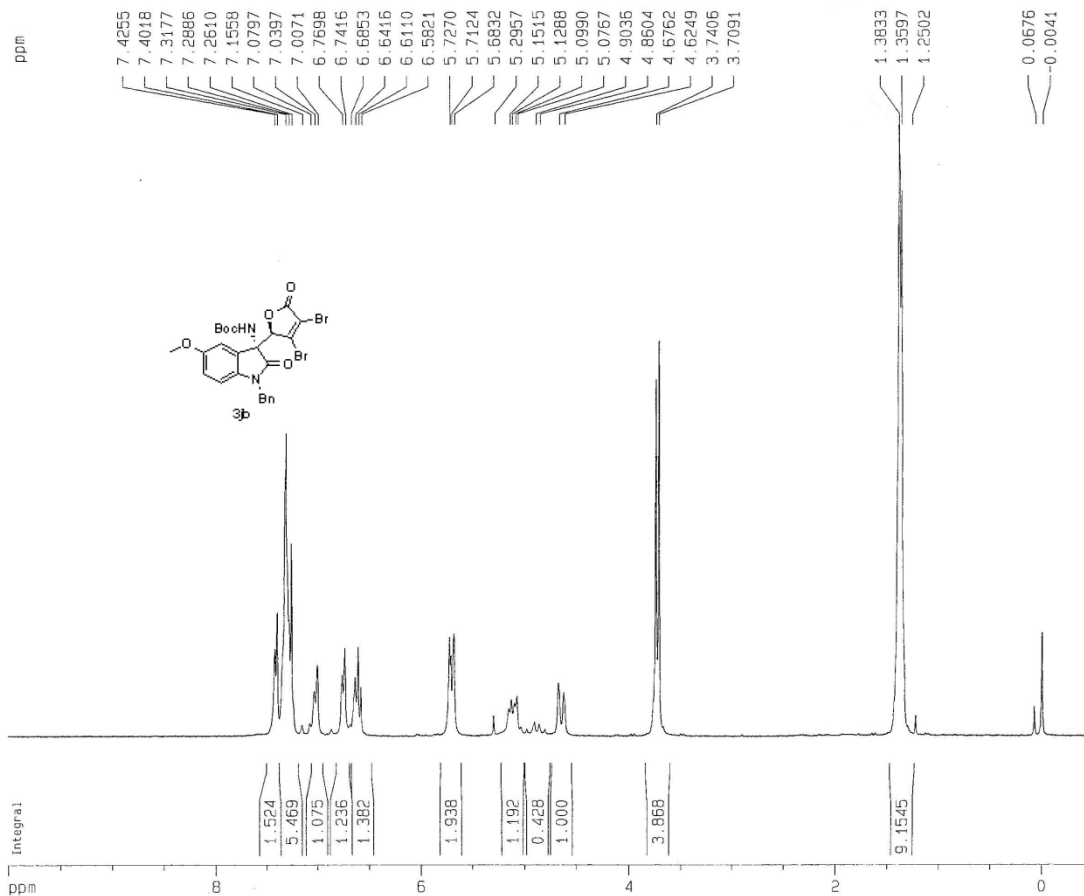


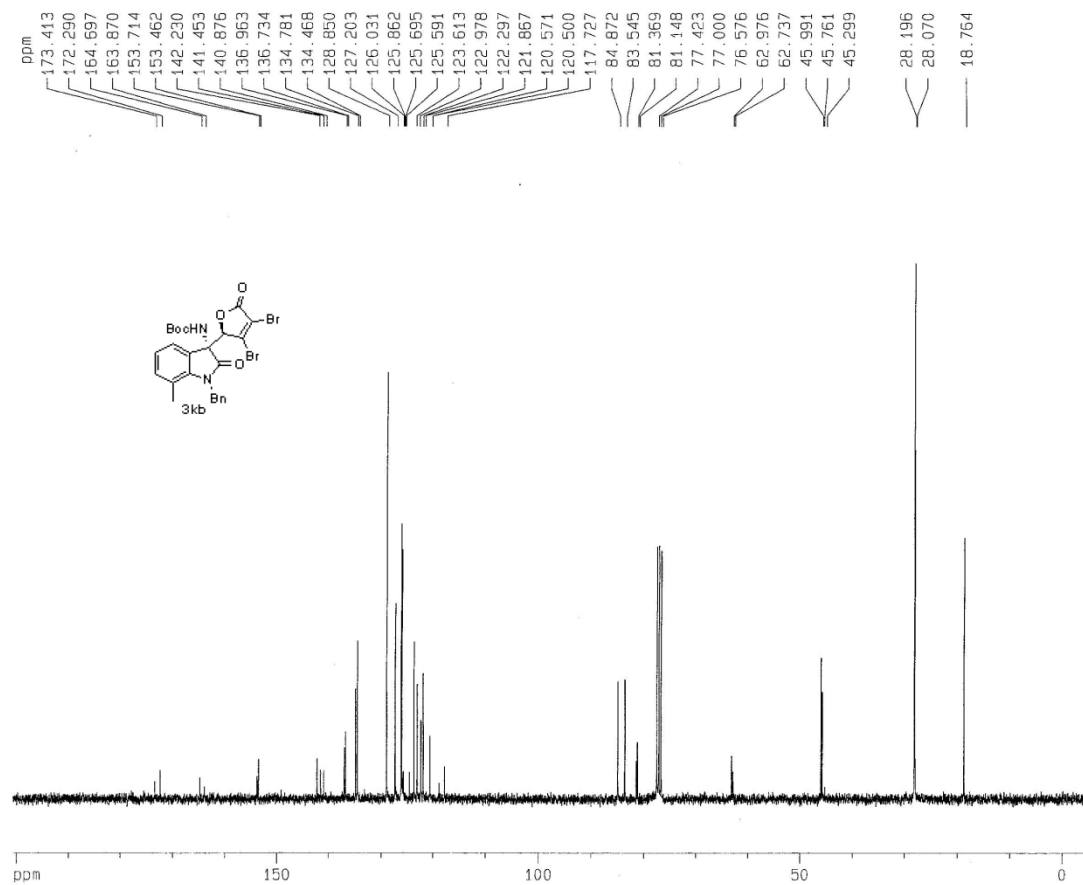
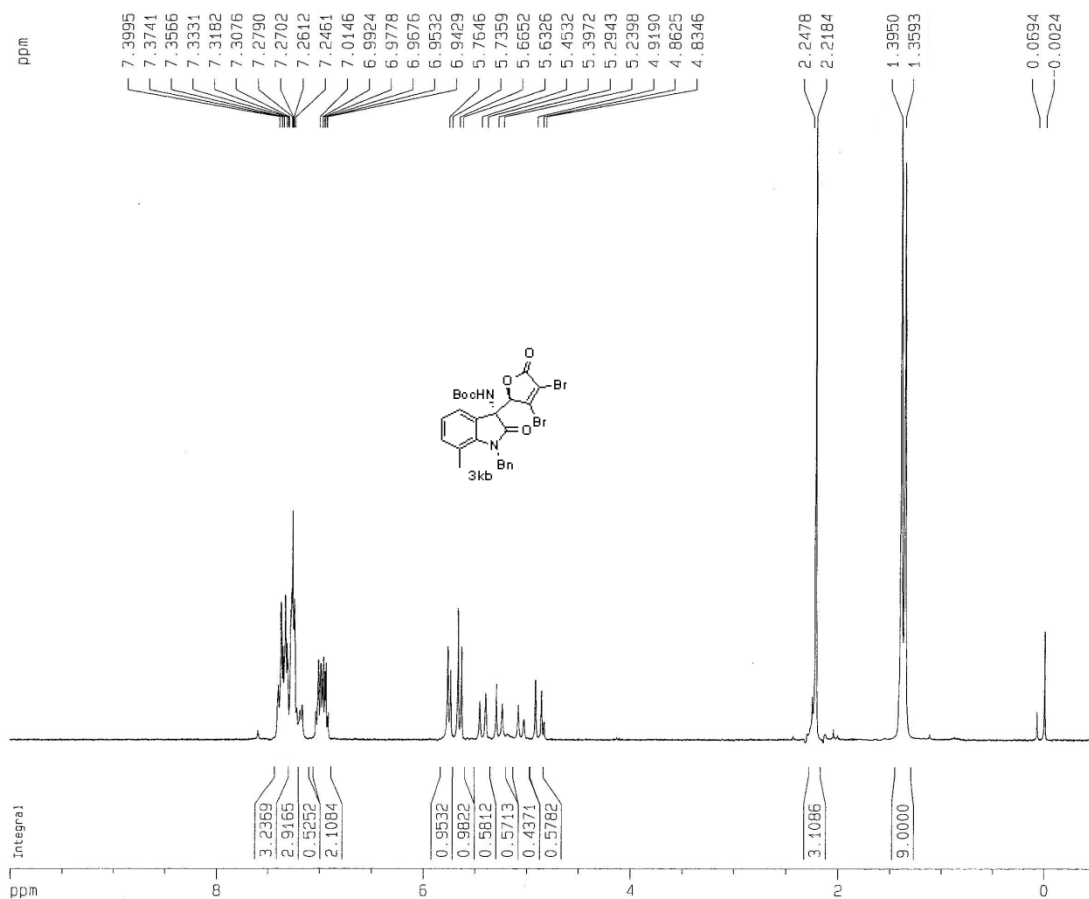


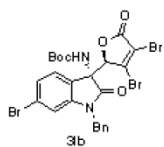
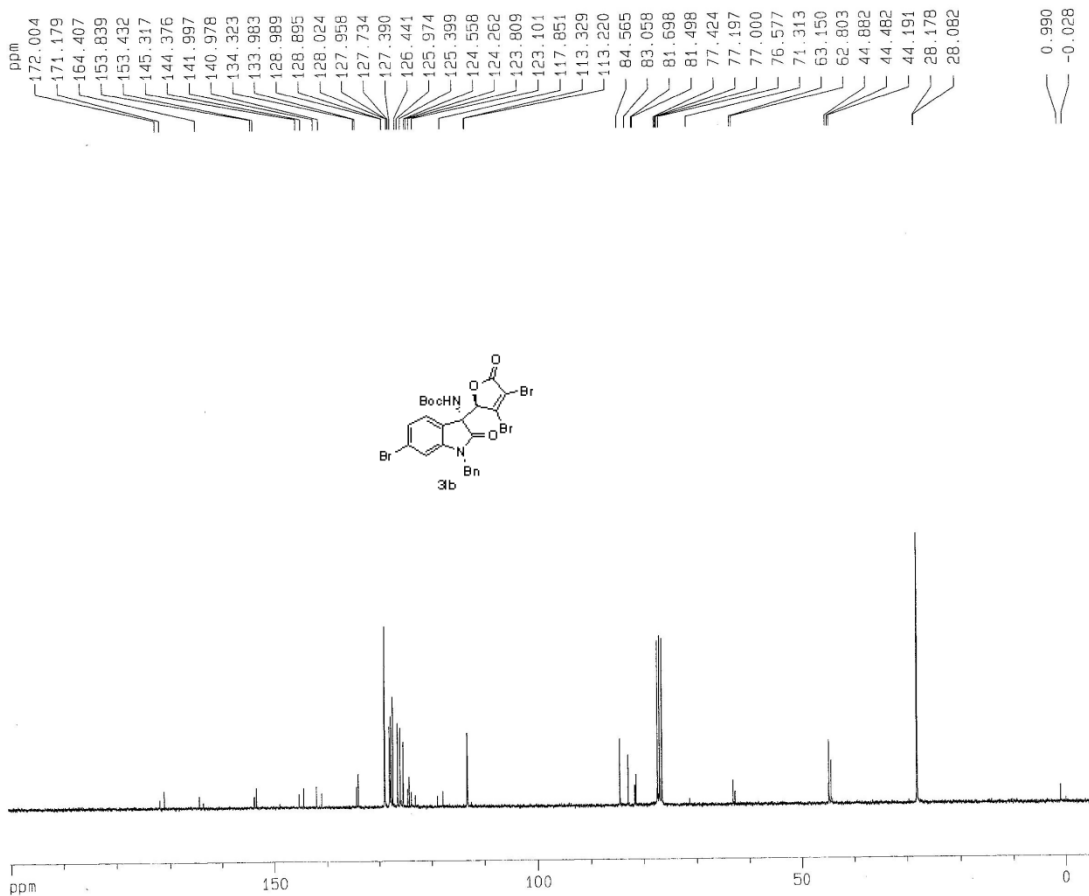
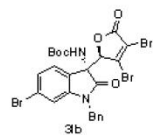
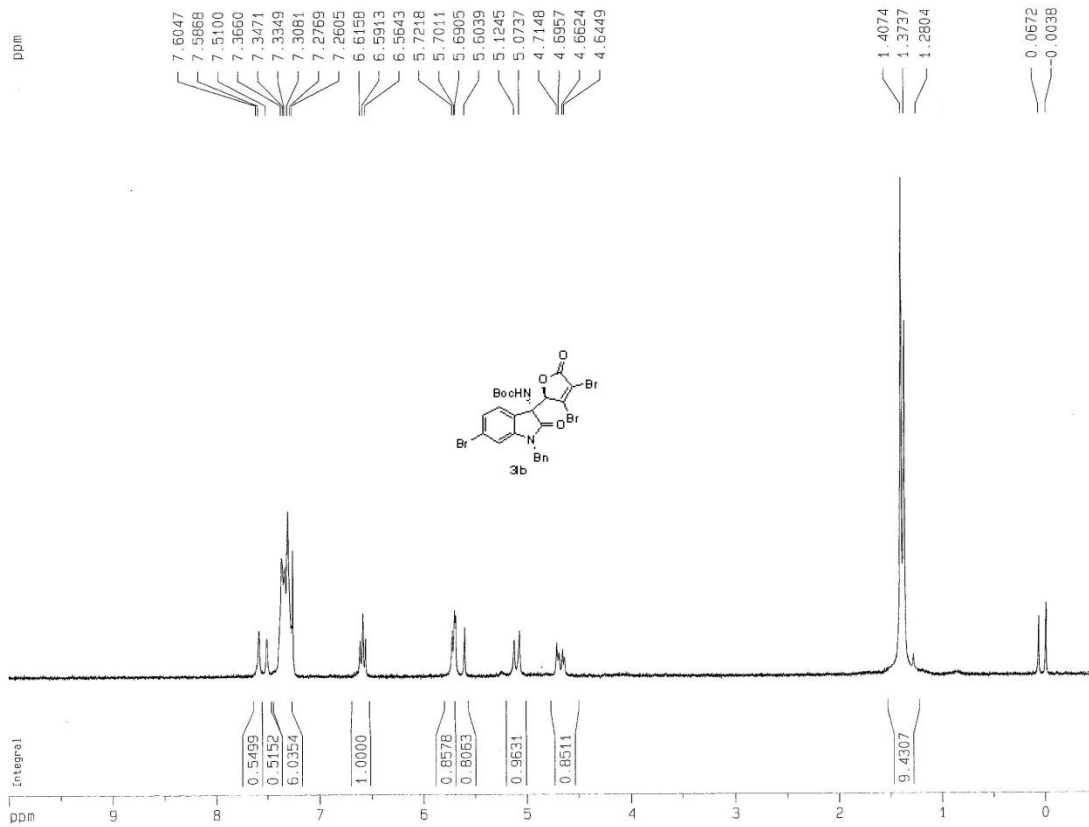




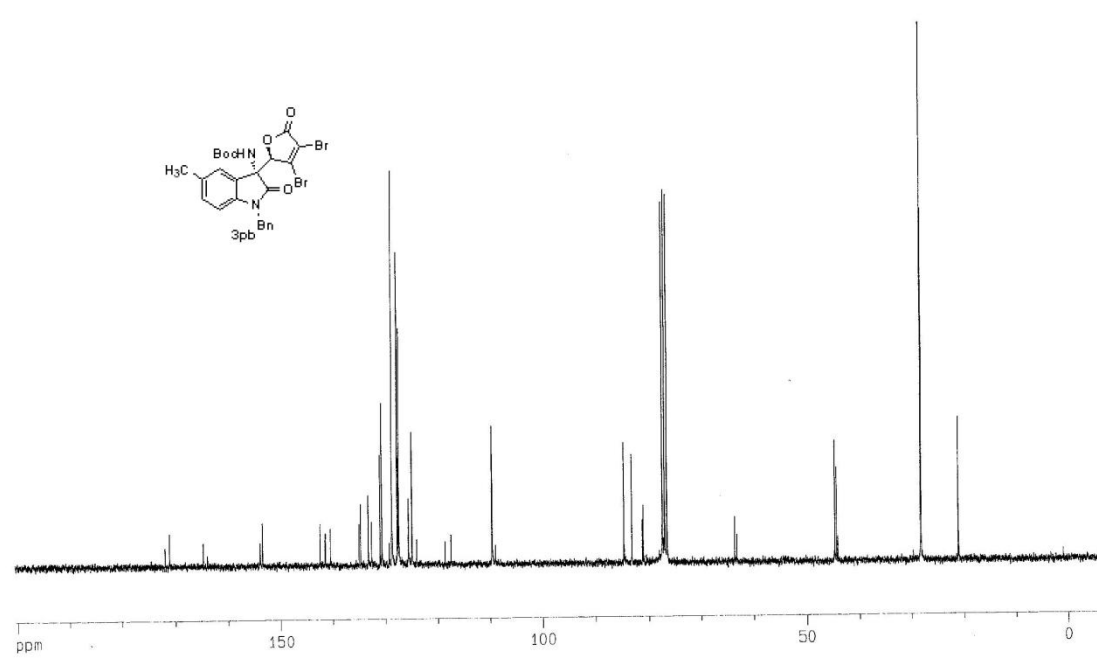
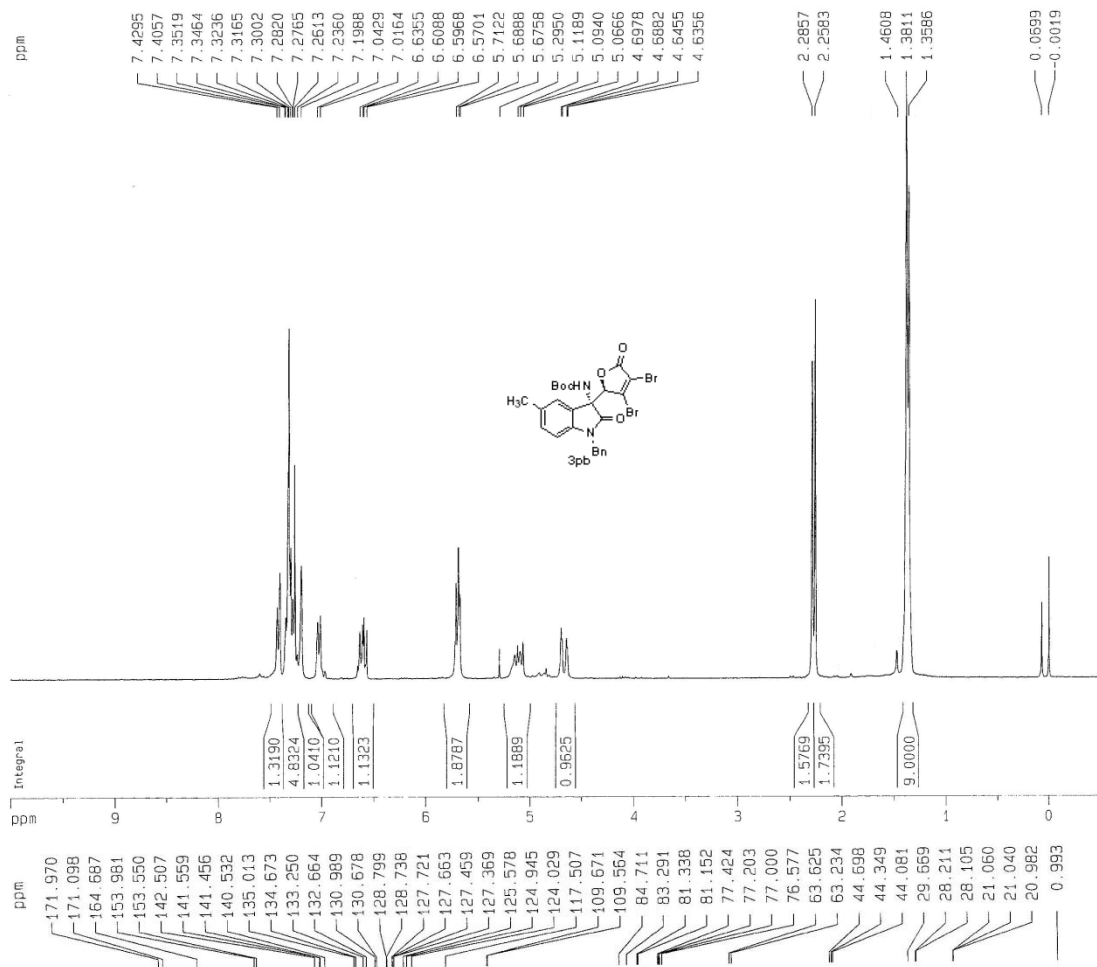




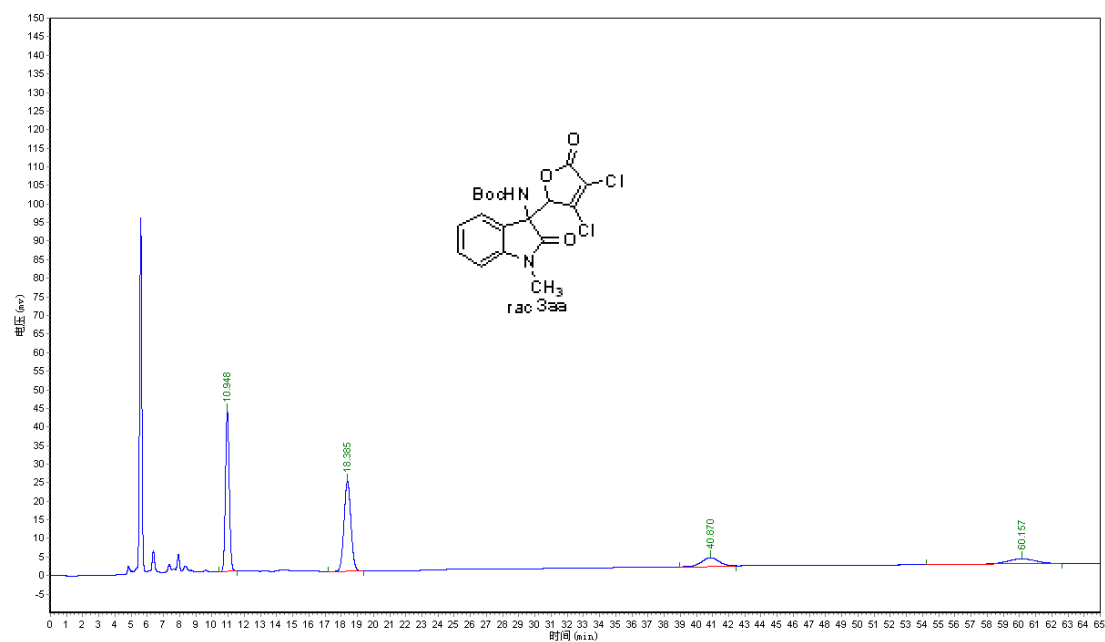




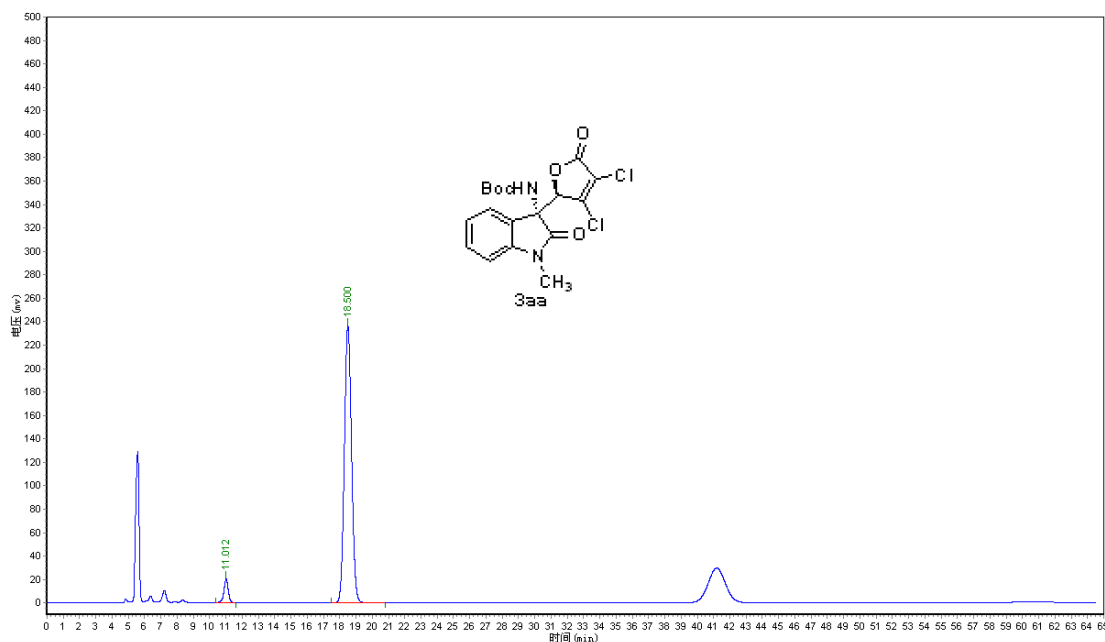




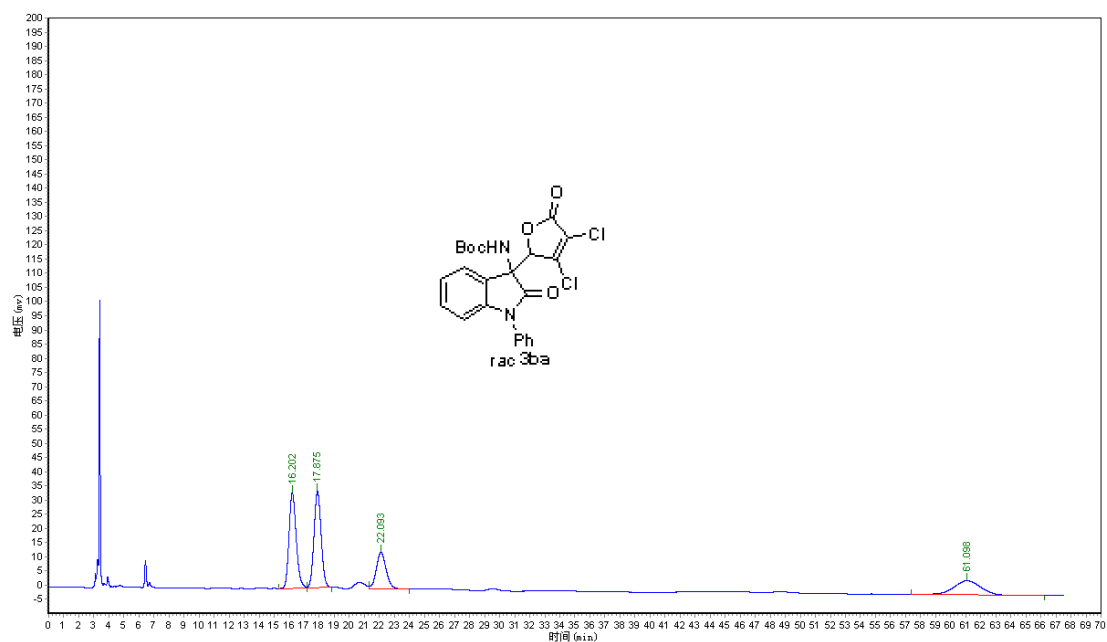
## HPLC Analysis of Products



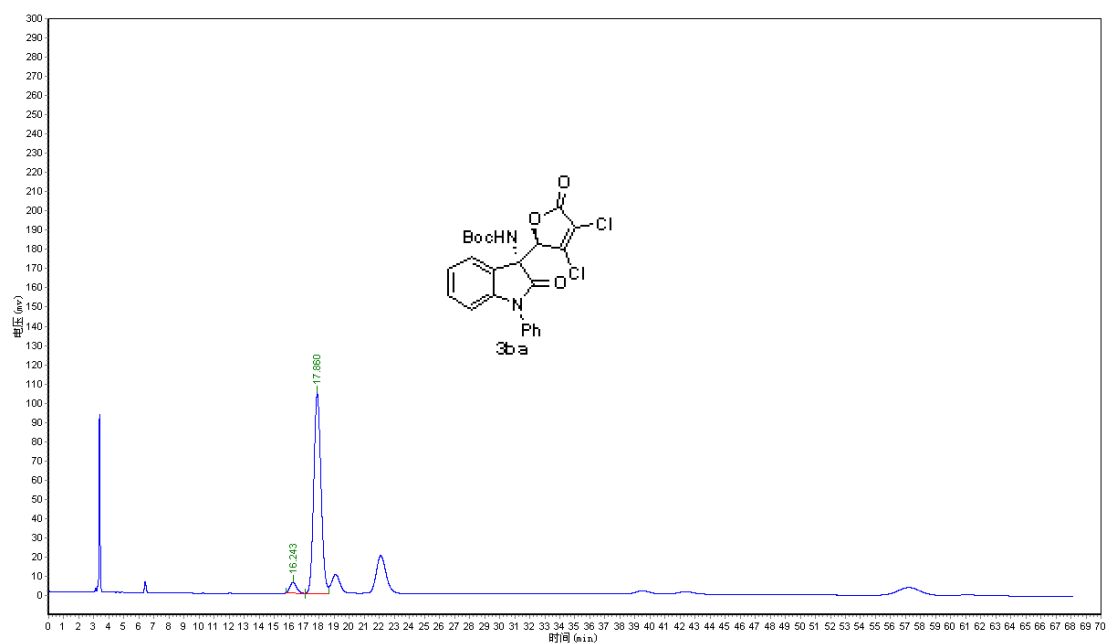
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.948	43186.887	711754.875	40.4866
2	18.385	24190.416	715714.563	40.7118
3	40.870	2274.969	167526.156	9.5293
4	60.157	1331.229	163006.703	9.2723



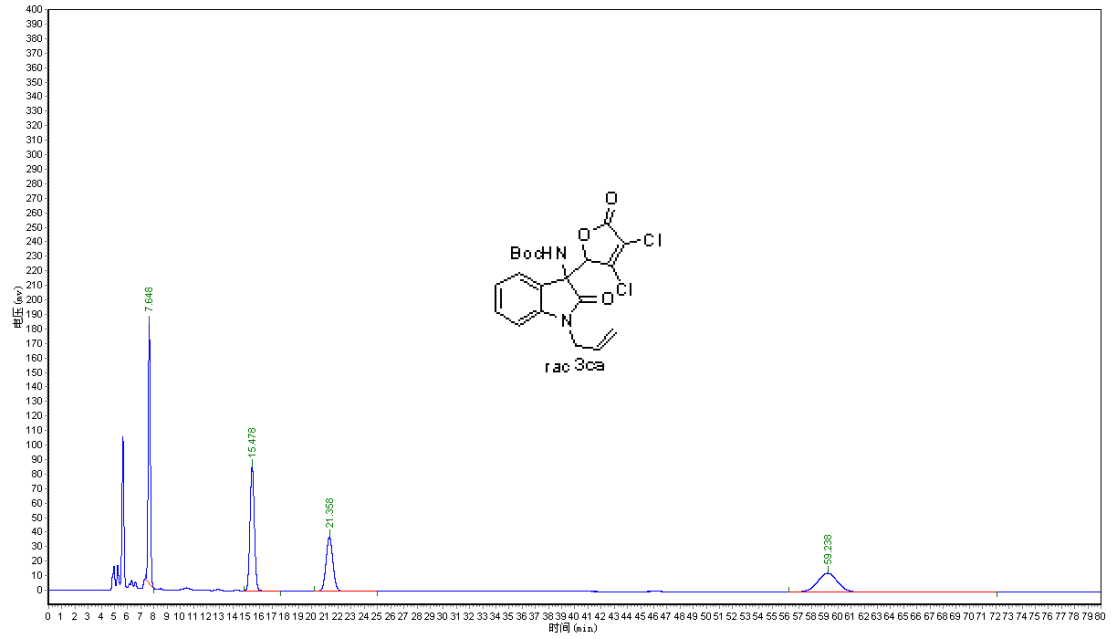
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	11.012	19974.441	373646.688	4.8995
2	18.500	236715.359	7252641.500	95.1005



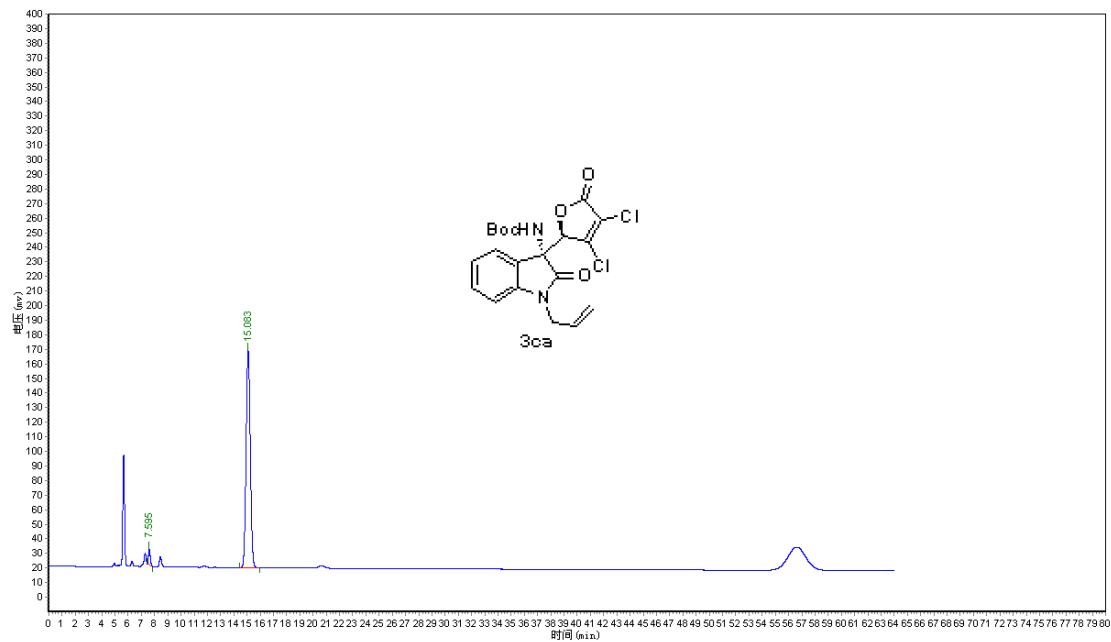
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	16.202	33600.953	1113794.625	32.4771
2	17.875	33878.523	1115014.250	32.5126
3	22.093	12943.305	594039.813	17.3216
4	61.098	4931.216	606631.813	17.6887



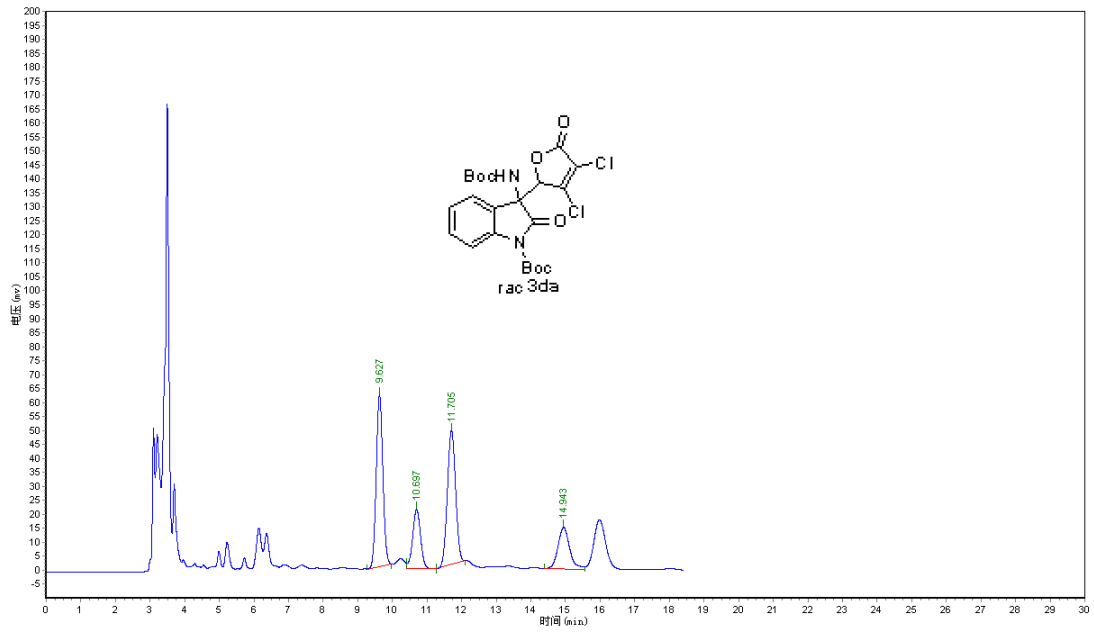
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	16.243	5525.462	173562.406	4.7335
2	17.860	103801.141	3493101.750	95.2665



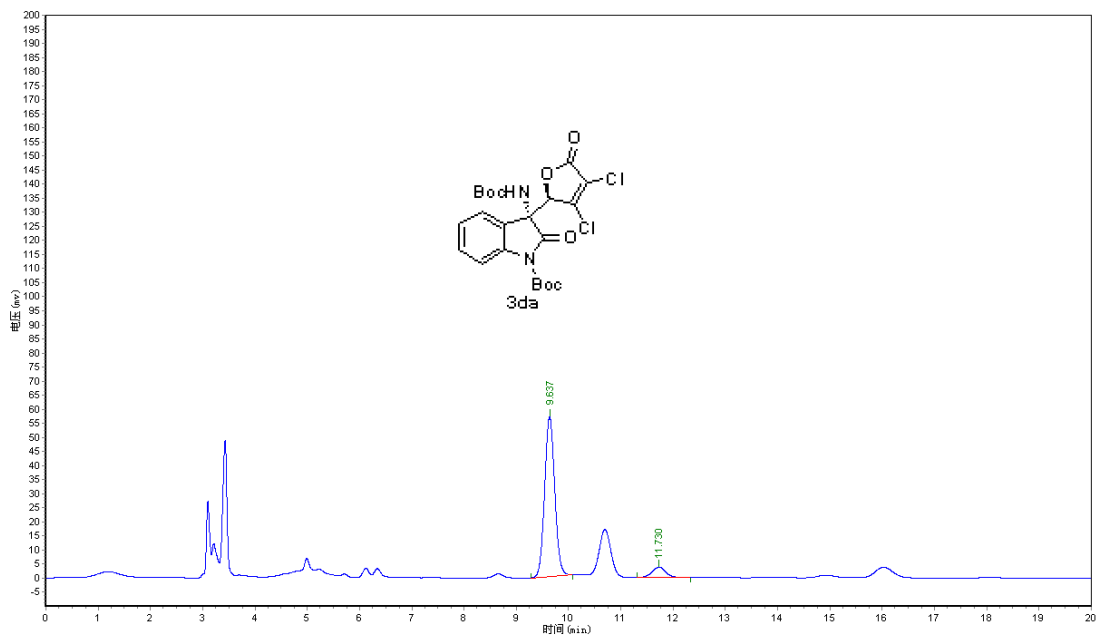
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.648	183144.641	2176273.750	31.7517
2	15.478	85848.430	1994313.750	29.0969
3	21.358	37227.109	1340752.750	19.5615
4	59.238	12830.772	1342702.875	19.5899



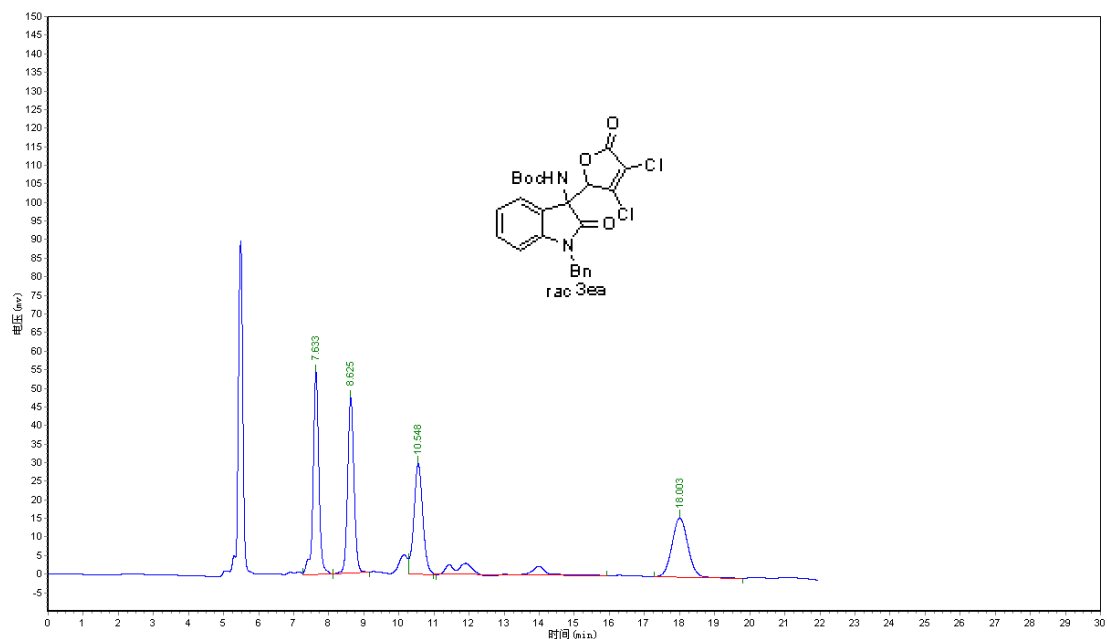
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.595	10553.082	103795.008	3.0559
2	15.083	148866.172	3292759.250	96.9441



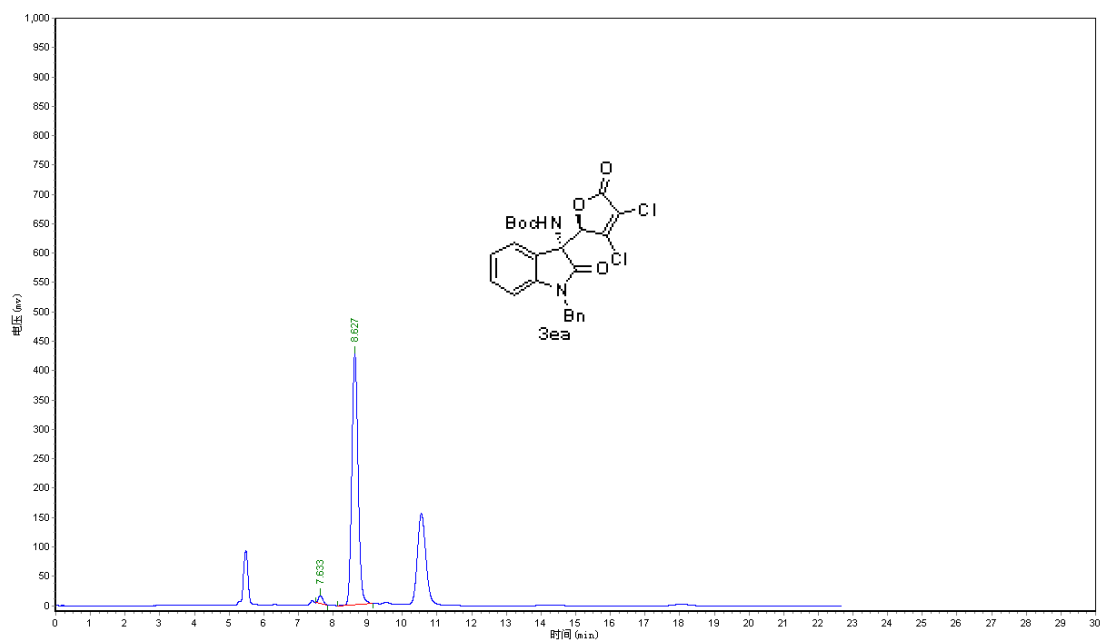
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	9.627	61465.477	817143.938	35.6555
2	10.697	21315.629	341022.094	14.8803
3	11.705	47904.750	790038.188	34.4728
4	14.943	14750.278	343571.406	14.9915



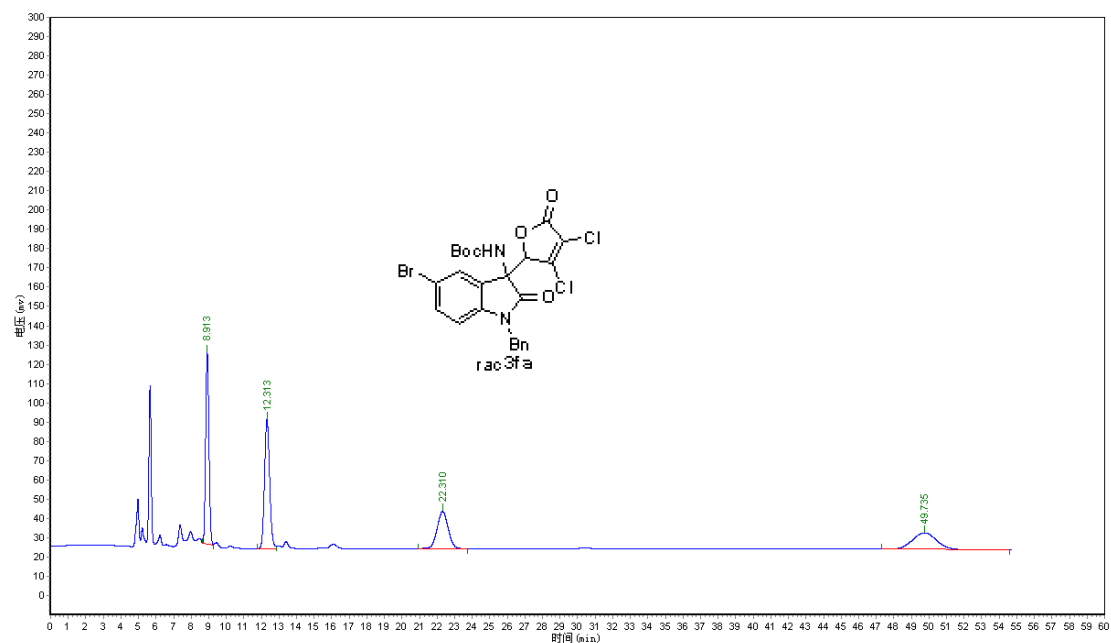
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	9.637	56766.391	761780.313	91.6742
2	11.730	3713.199	69184.102	8.3258



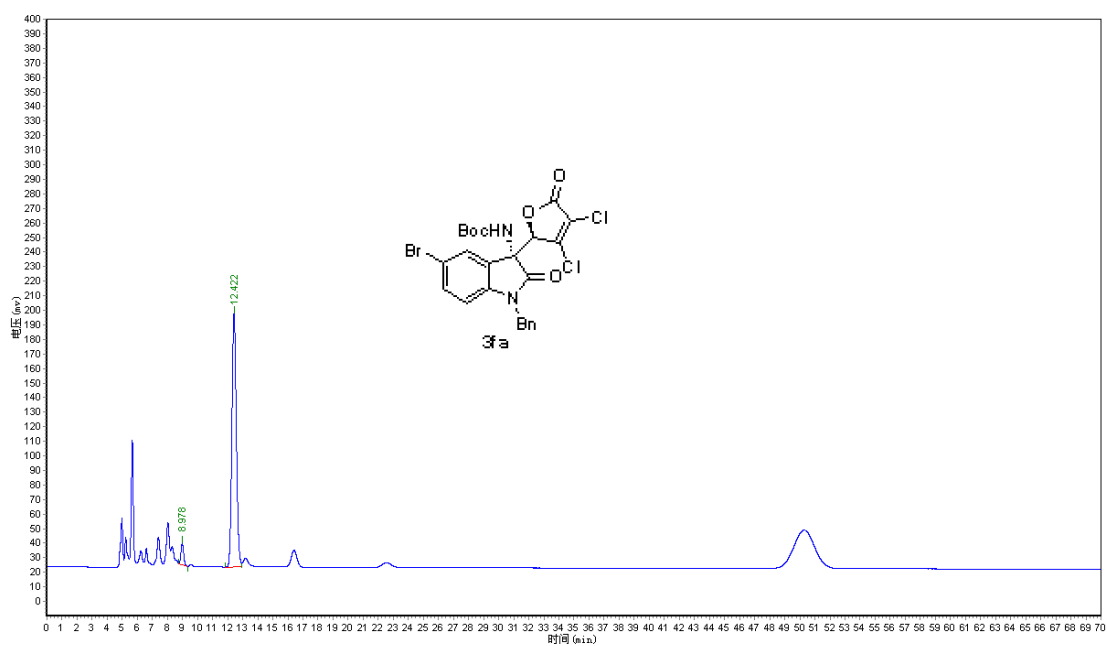
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.633	54341.012	623894.500	28.3394
2	8.625	47158.332	593187.813	26.9446
3	10.548	29747.672	524894.688	23.8425
5	18.003	15919.872	496869.656	22.5695



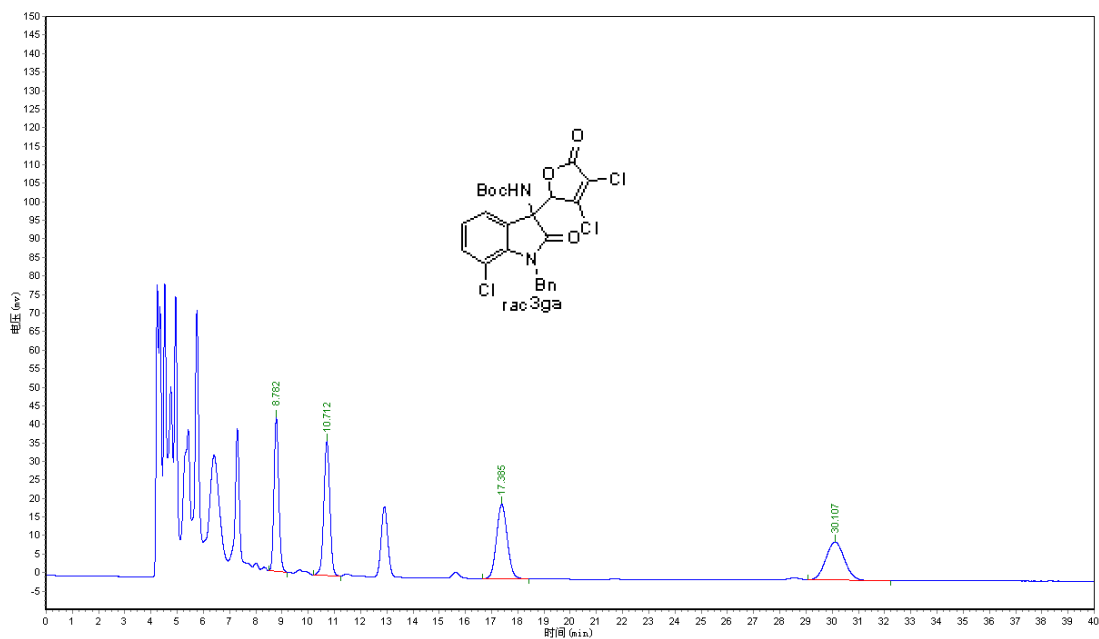
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.633	13640.335	121941.594	2.2217
2	8.627	427458.063	5366768.000	97.7783



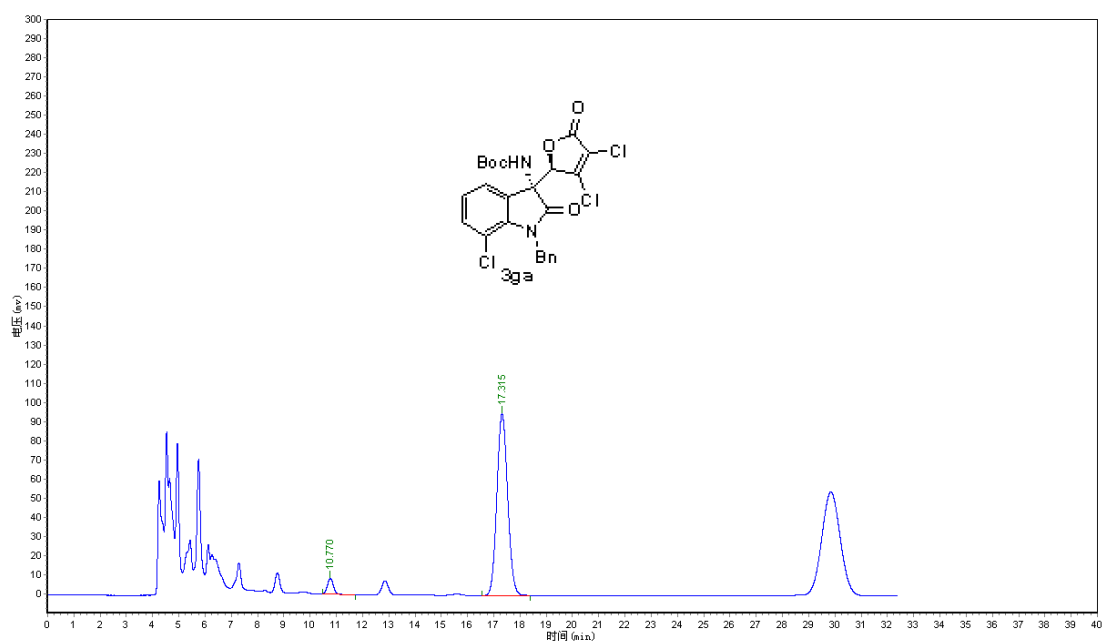
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.913	99327.906	1305766.750	30.2931
2	12.313	66992.805	1361081.125	31.5763
3	22.310	19147.713	823689.000	19.1091
4	49.735	8576.276	819908.625	19.0214



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.978	14583.021	184964.438	4.9049
2	12.422	174090.234	3586045.500	95.0951

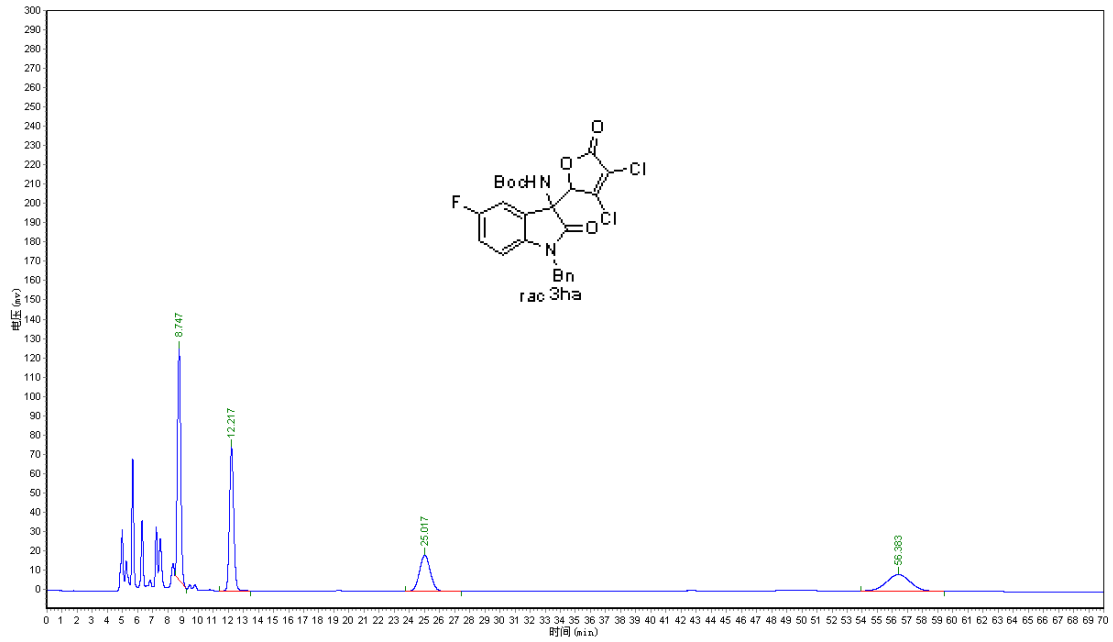


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.782	41439.375	522852.563	24.0422
2	10.712	36174.777	576136.625	26.4923
3	17.385	20045.479	572187.313	26.3107
4	30.107	10279.355	503552.688	23.1547

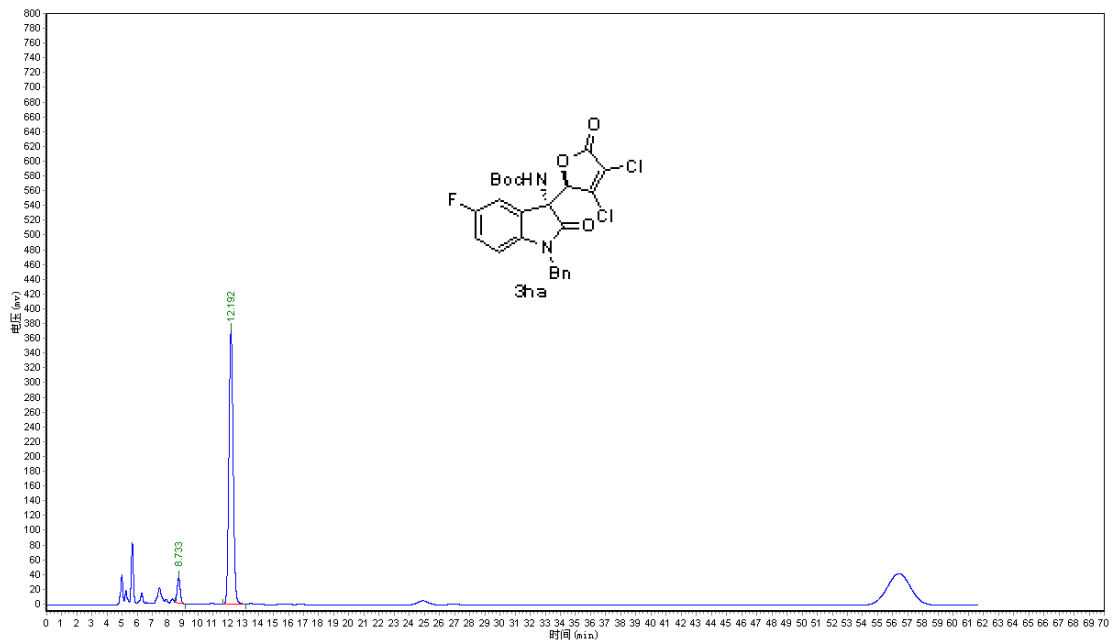


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.770	8179.040	124052.547	4.3794
2	17.315	94831.602	2708588.750	95.6206

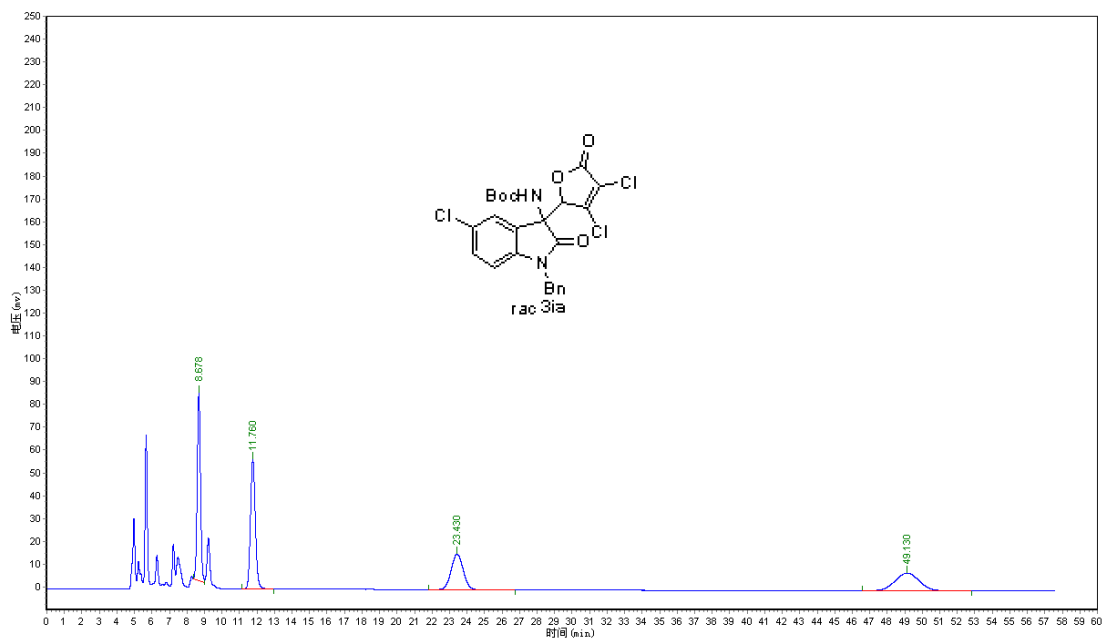




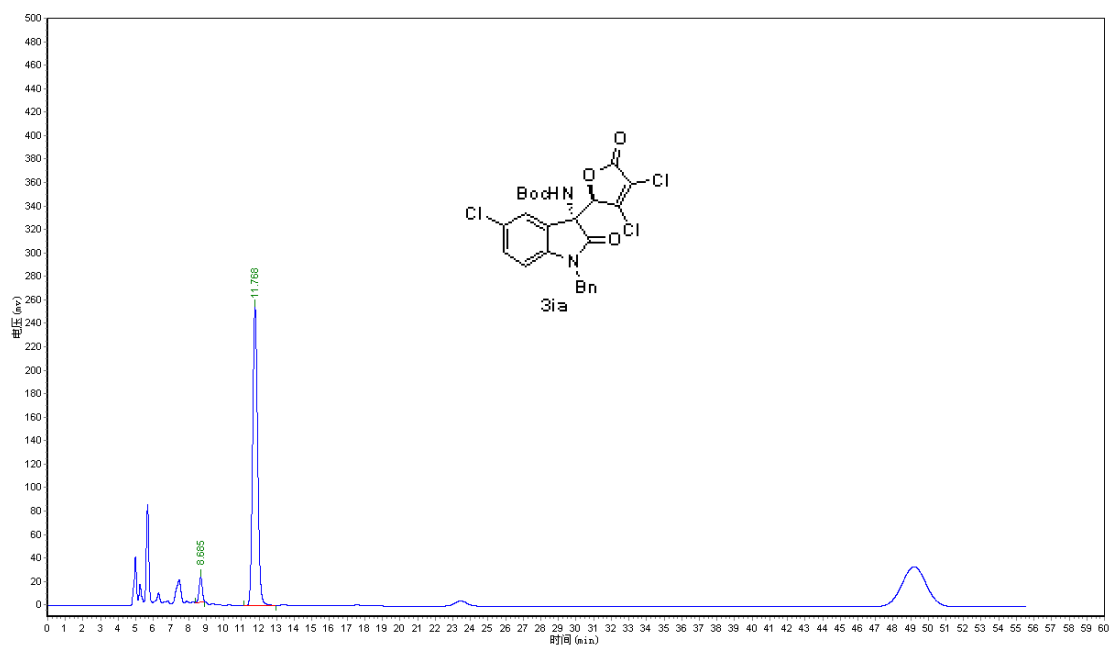
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.747	119987.805	1843084.875	35.8413
2	12.217	74321.070	1452519.875	28.2463
3	25.017	18607.607	919573.813	17.8824
4	56.383	8493.208	927164.813	18.0300



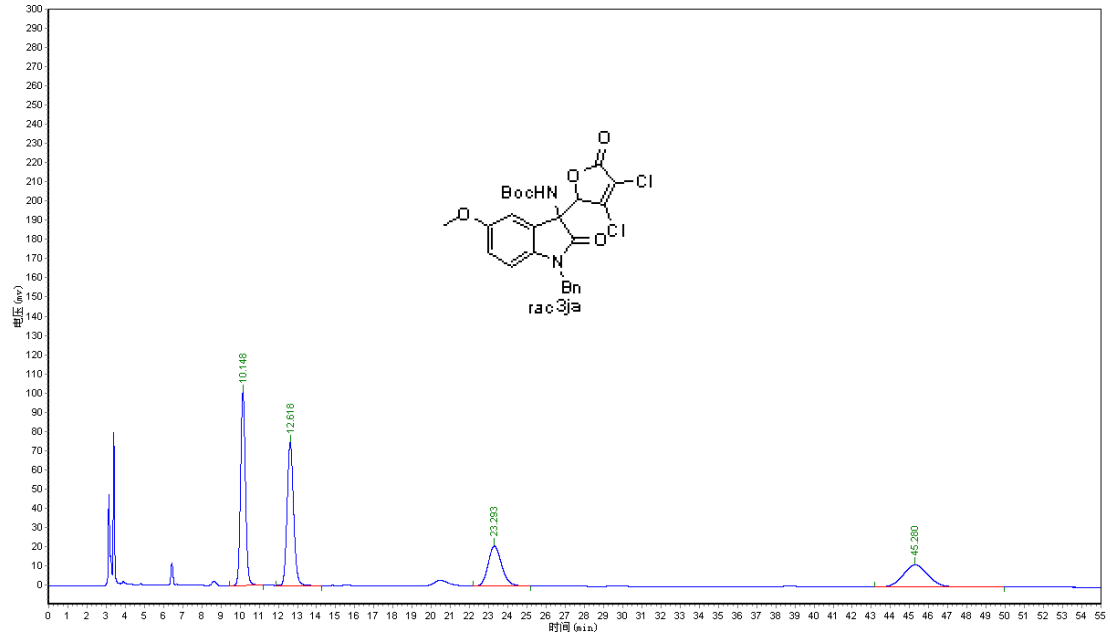
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.733	33342.168	458198.375	5.9596
2	12.192	369725.531	7230147.500	94.0404



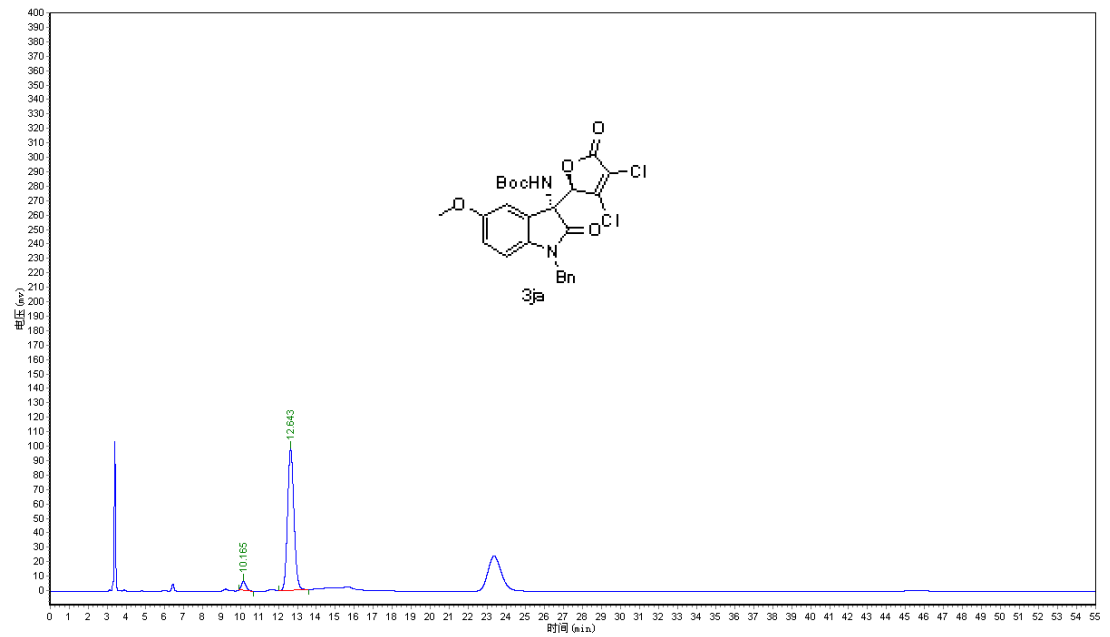
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.678	81817.516	1061358.375	29.0225
2	11.760	56548.359	1100477.875	30.0922
3	23.430	15573.232	745448.875	20.3841
4	49.130	7731.106	749734.000	20.5012



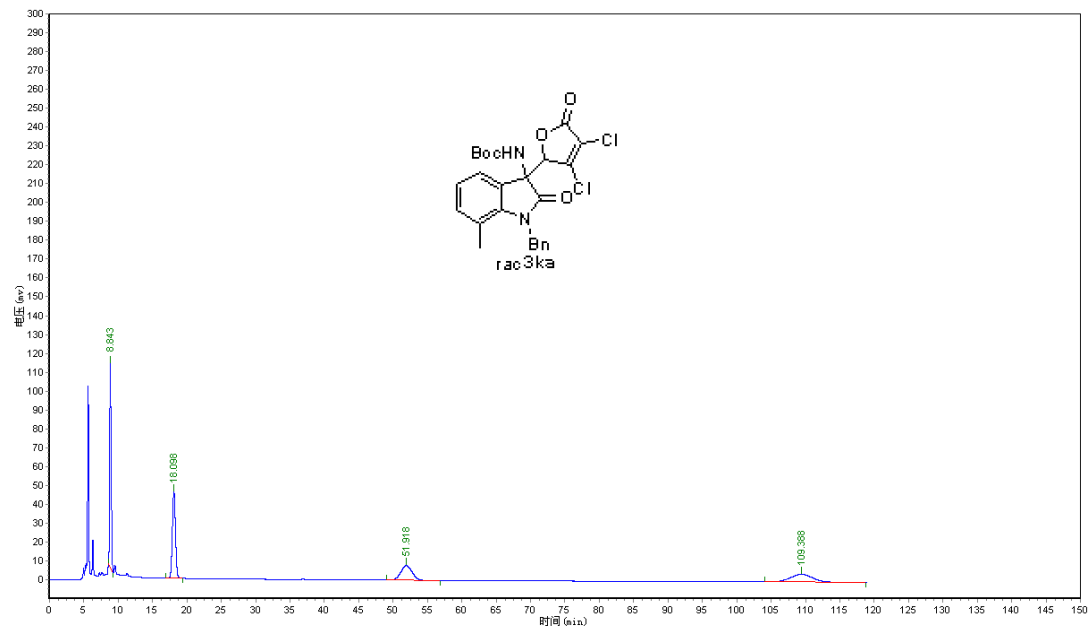
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.685	21536.572	267291.750	5.0843
2	11.768	254040.563	4989902.000	94.9157



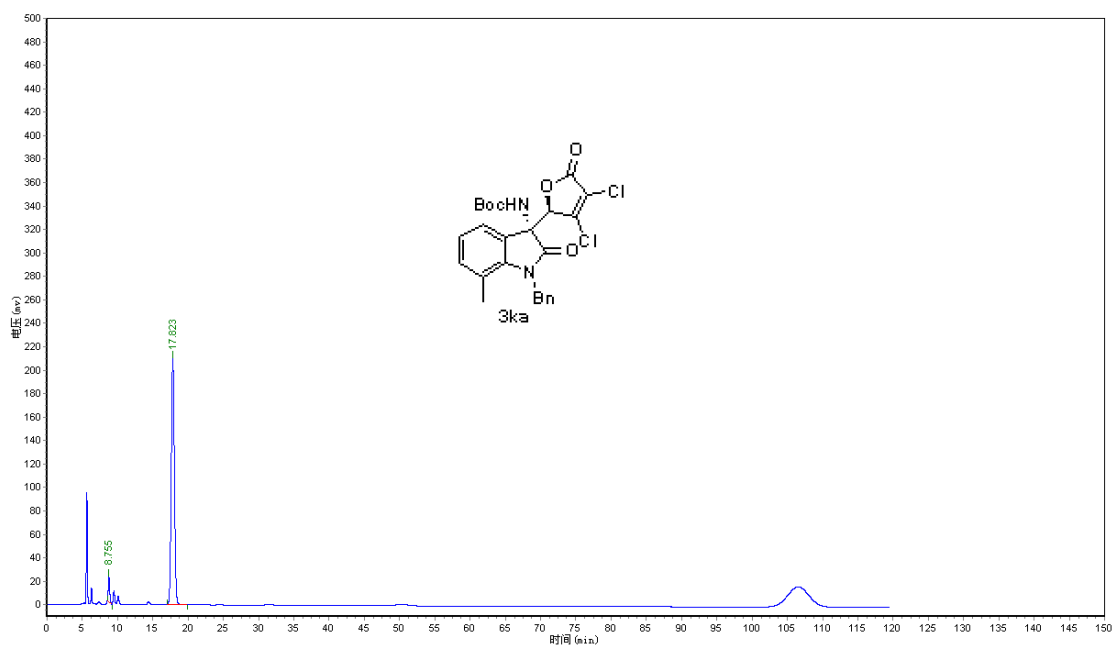
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.148	100755.367	1790111.875	31.9635
2	12.618	74641.328	1760507.875	31.4349
3	23.293	20839.744	1019915.125	18.2112
4	45.280	11431.326	1029952.625	18.3904



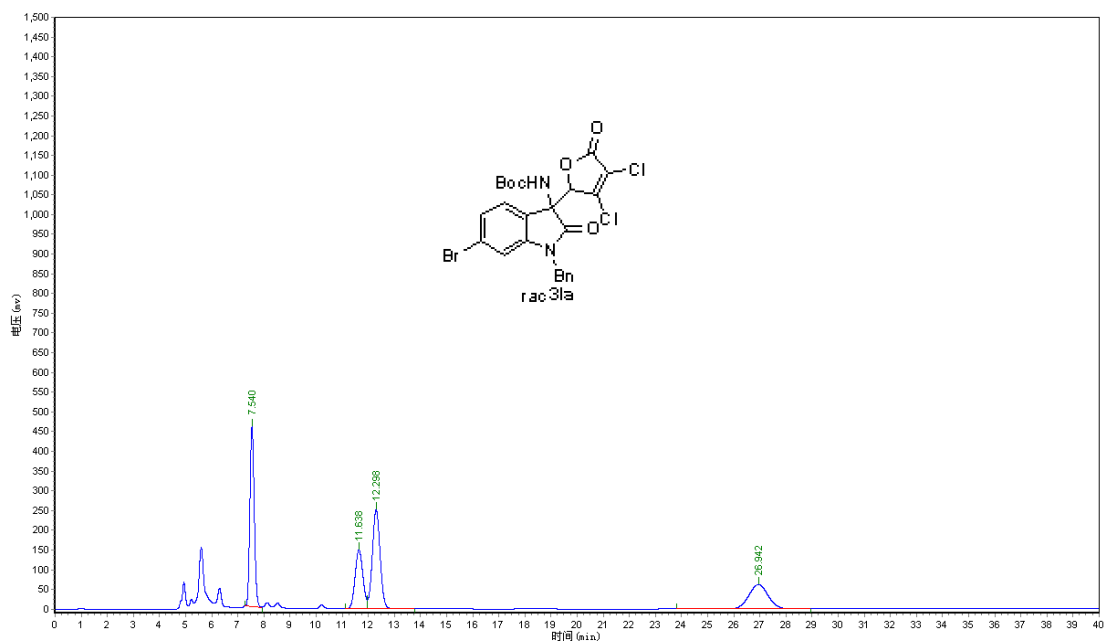
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.165	6051.625	99907.102	4.1814
2	12.643	97895.477	2289425.250	95.8186



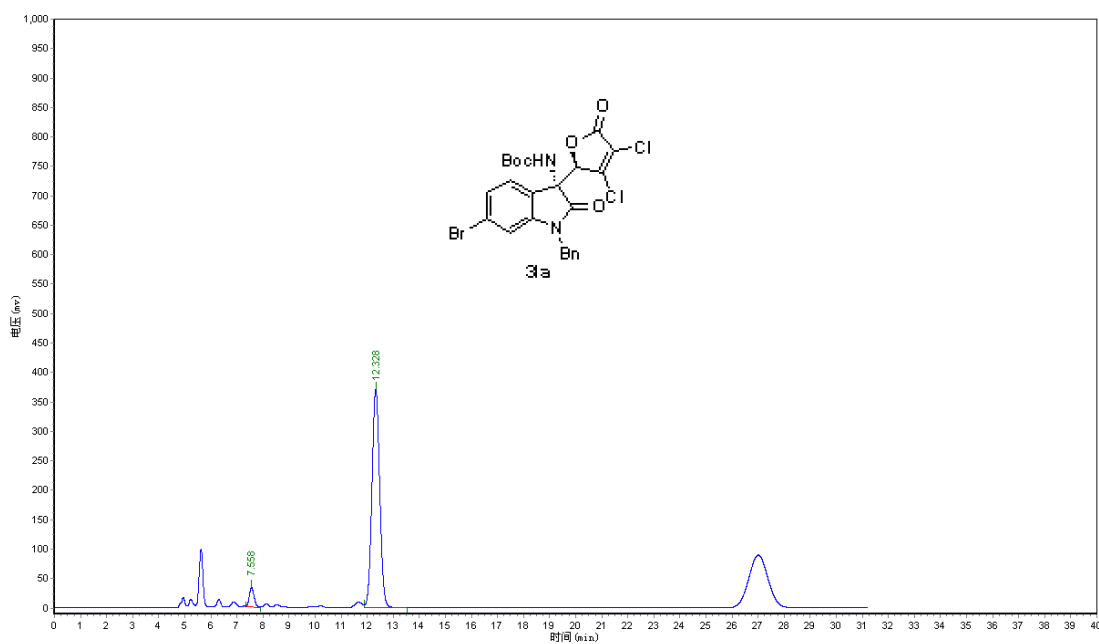
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.843	107775.172	1635492.375	33.4401
2	18.098	46021.691	1466626.375	29.9874
3	51.918	7703.782	885215.500	18.0995
4	109.388	4033.082	903481.750	18.4730



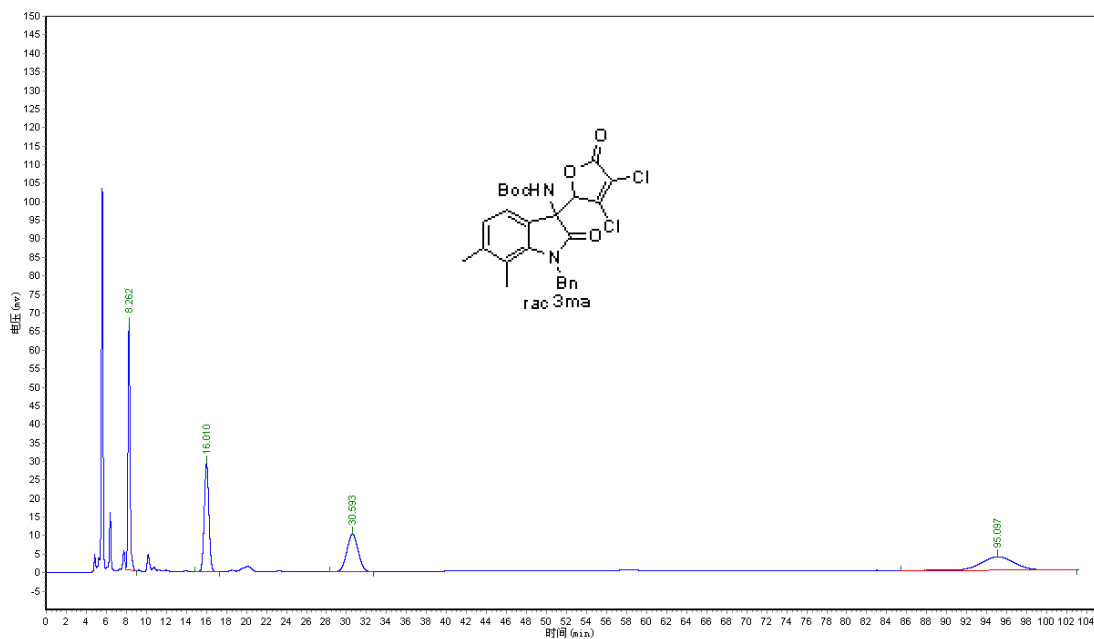
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.755	21003.699	292806.719	4.3242
2	17.823	209524.156	6478598.500	95.6758



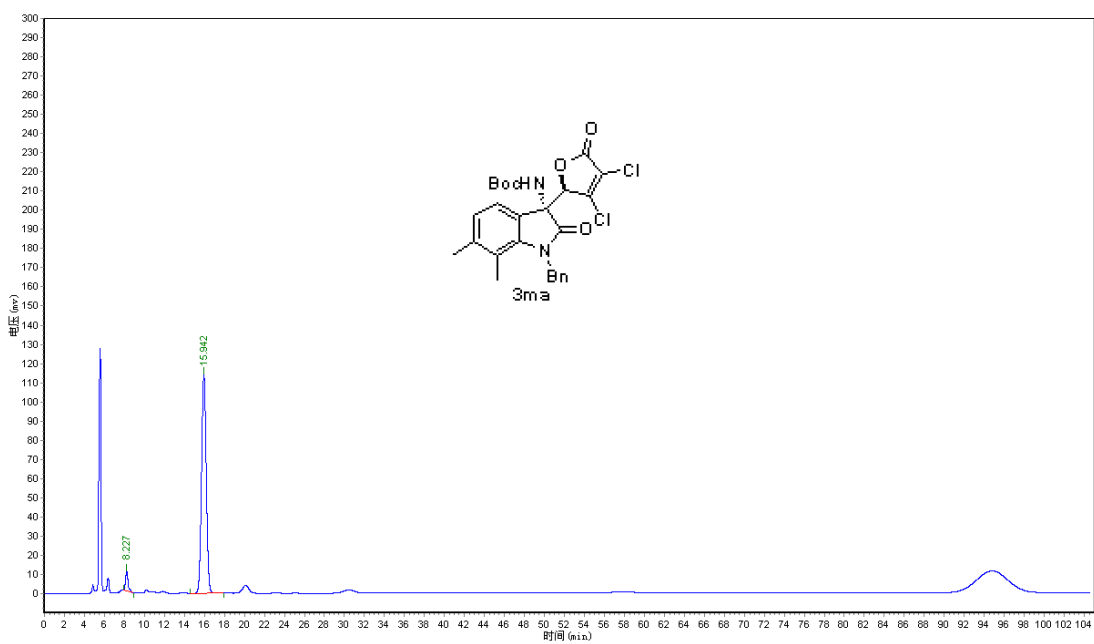
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.540	455120.813	5289743.000	31.5455
2	11.638	149738.891	3108366.250	18.5368
3	12.298	251356.031	5207160.000	31.0530
4	26.942	61417.141	3163343.750	18.8647



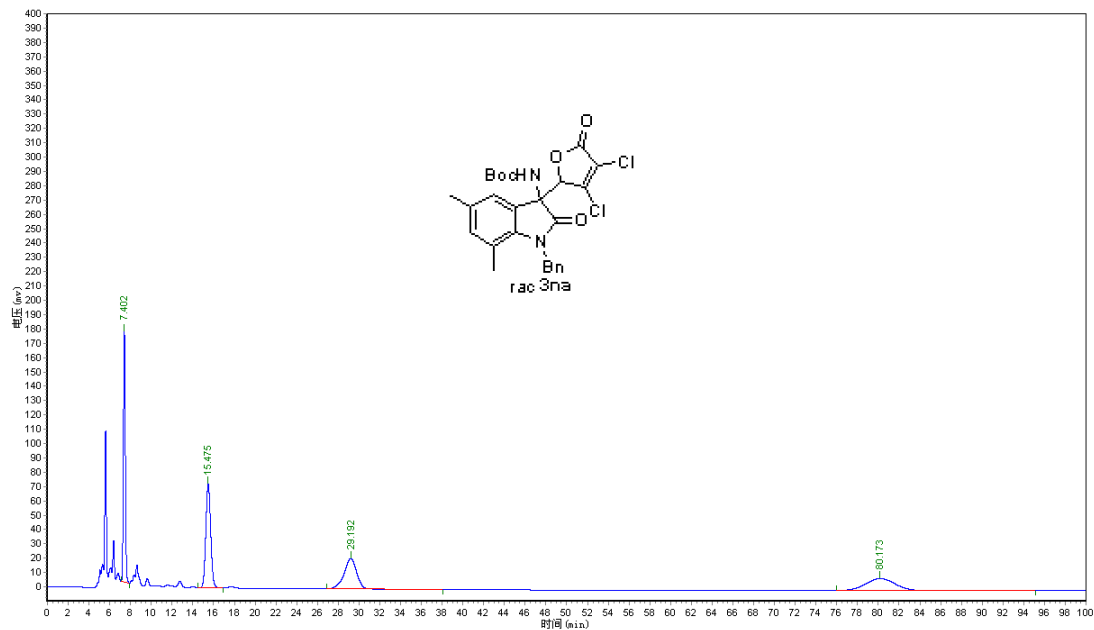
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.558	33462.934	423780.781	5.2207
2	12.328	371222.406	7693605.000	94.7793



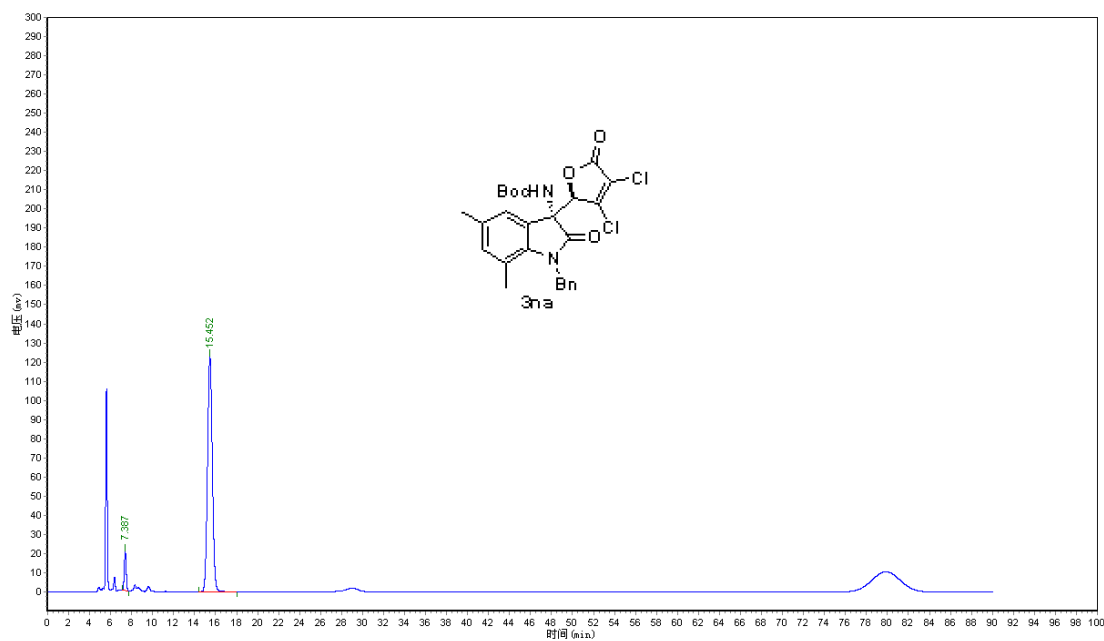
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.262	66198.578	965588.813	27.4582
2	16.010	29247.000	938467.688	26.6869
3	30.593	10093.351	795739.000	22.6282
4	95.097	3577.837	816787.875	23.2267



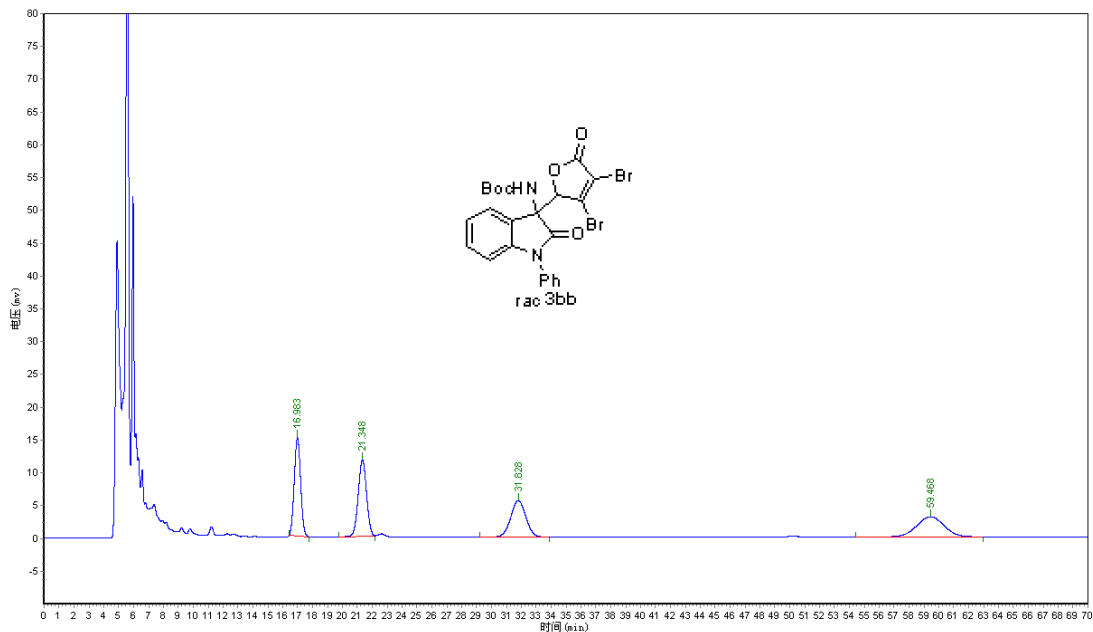
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.227	10234.311	185134.109	4.7166
2	15.942	113977.813	3740024.250	95.2834



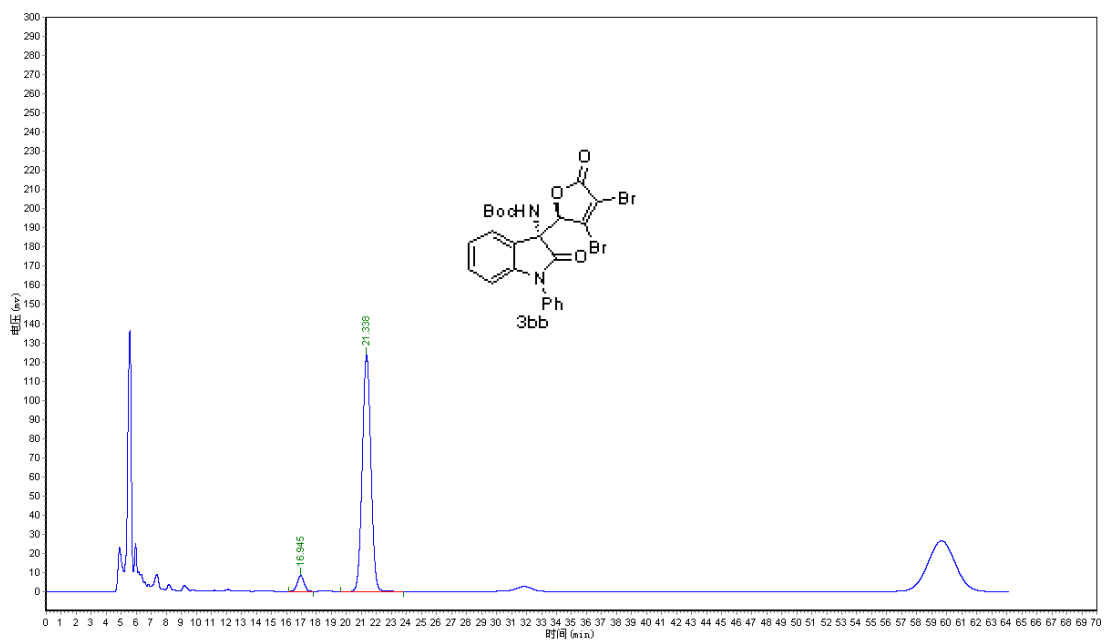
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.402	174674.984	2251189.250	28.2268
2	15.475	72535.984	2368942.500	29.7032
3	29.192	21141.666	1770262.375	22.1966
4	80.173	8230.957	1584979.625	19.8734



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	7.387	20375.834	262114.156	6.2867
2	15.452	122590.844	3907221.750	93.7133

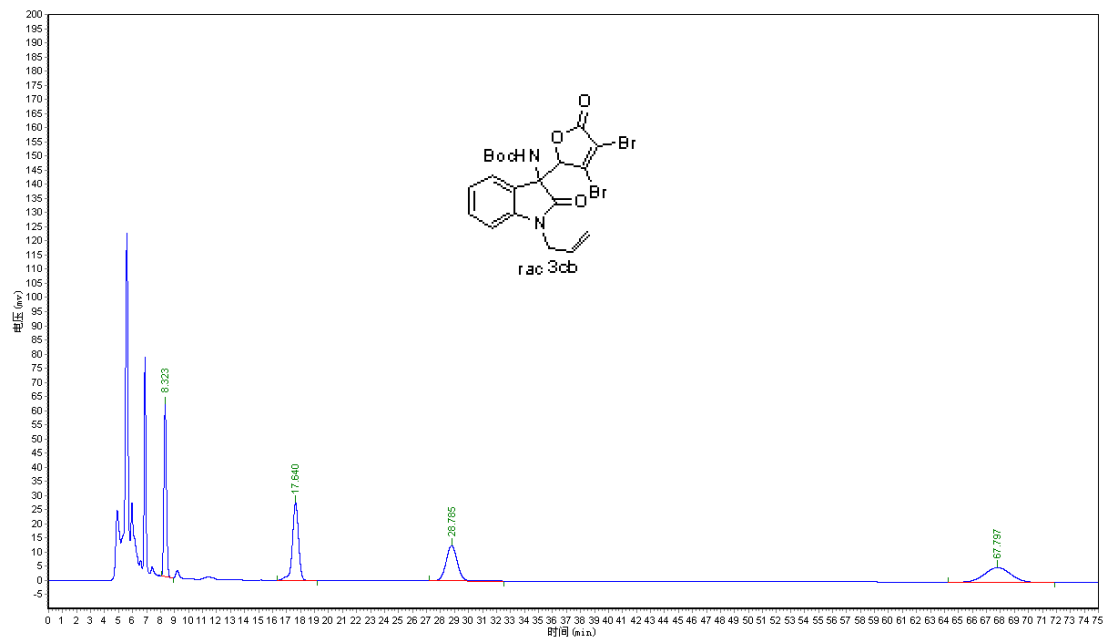


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	16.983	14988.339	443282.938	26.2463
2	21.348	11748.357	466230.594	27.6050
3	31.828	5535.136	391288.188	23.1677
4	59.468	3057.458	388135.000	22.9810

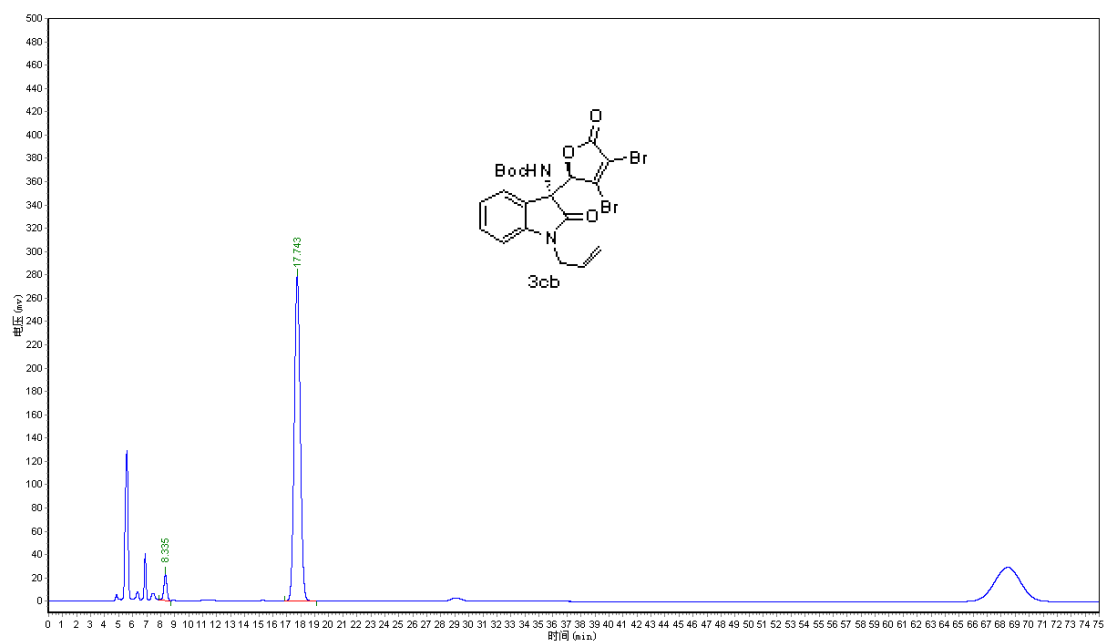


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	16.945	8250.203	259722.203	5.0125
2	21.338	123340.875	4921746.000	94.9875

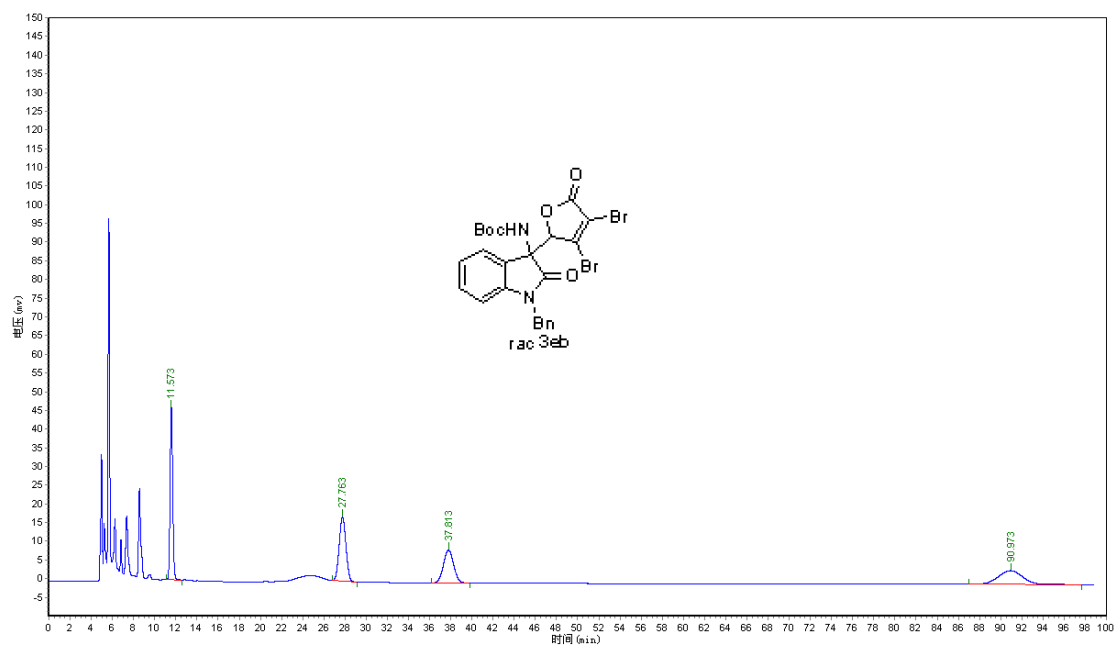




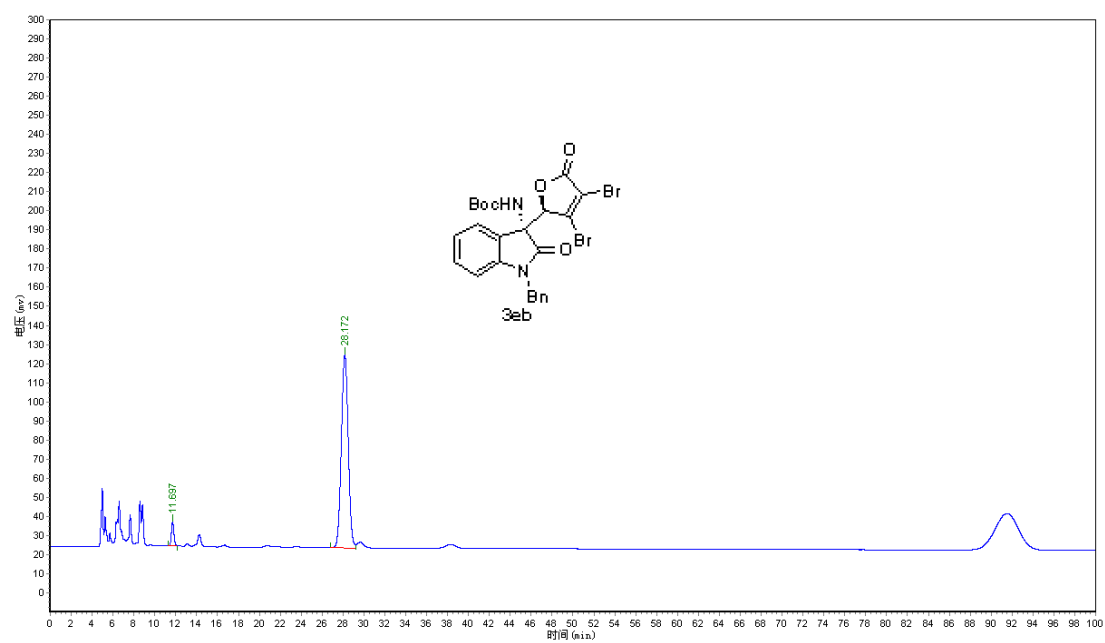
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.323	60729.602	773248.563	26.0862
2	17.640	27461.416	833979.625	28.1350
3	28.785	12409.807	682052.375	23.0096
4	67.797	5184.129	674929.938	22.7693



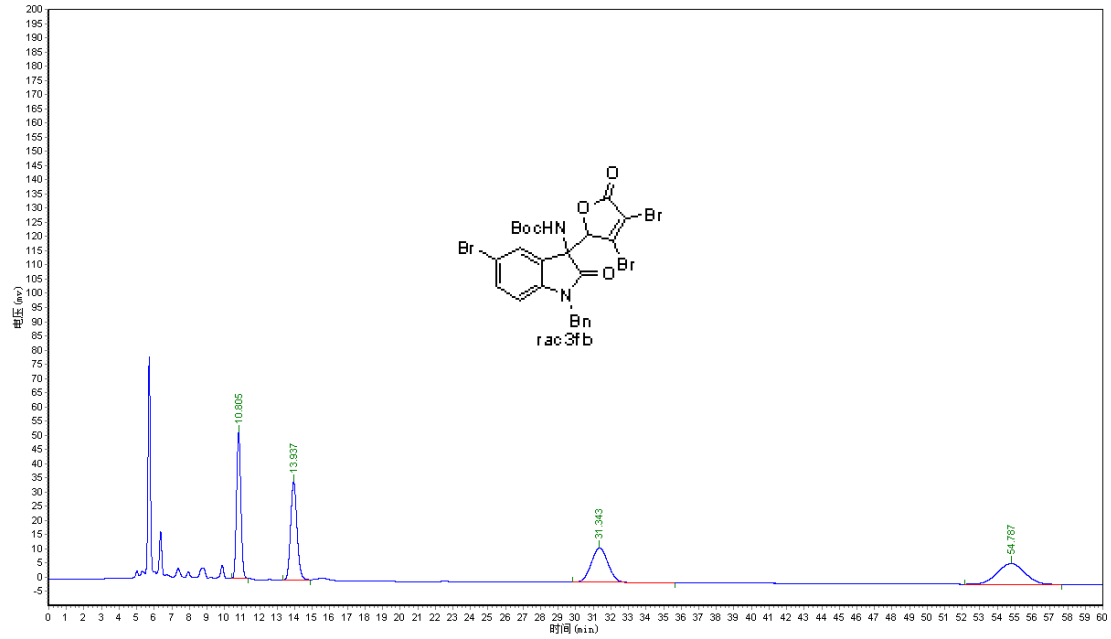
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.335	22374.381	343851.563	3.9716
2	17.743	278624.813	8313929.500	96.0284



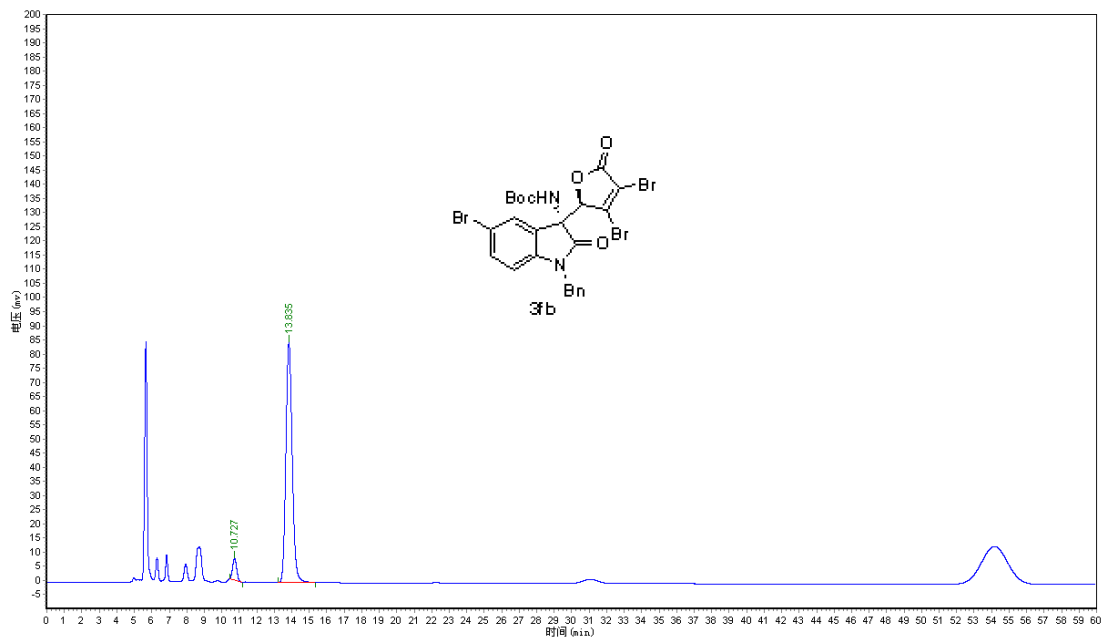
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	11.573	45891.070	791839.813	28.9424
2	27.763	17094.932	761633.313	27.8383
3	37.813	8806.108	585391.375	21.3965
4	90.973	3684.217	597053.750	21.8228



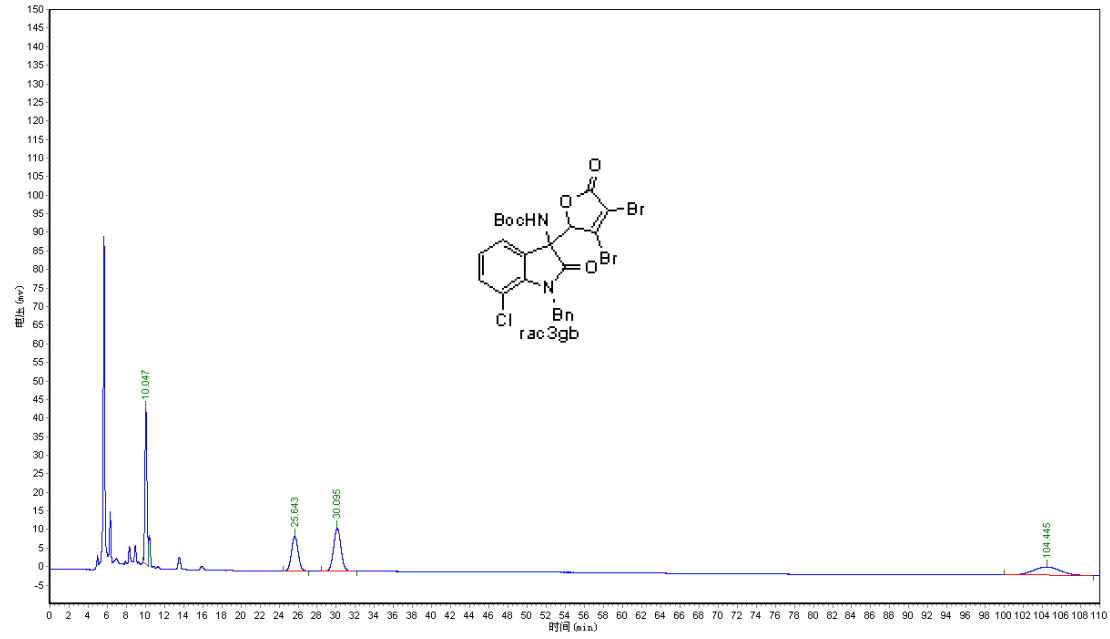
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	11.697	12397.798	206235.922	4.2649
2	28.172	101026.539	4629456.000	95.7351



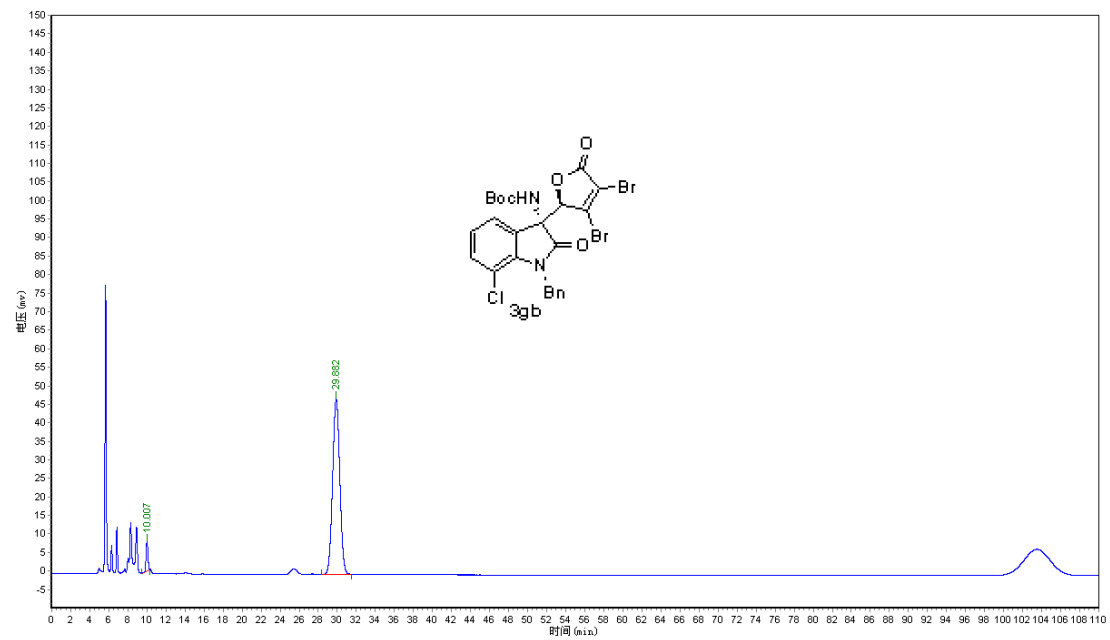
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.805	51428.246	881109.750	26.2670
2	13.937	34580.285	876928.813	26.1423
3	31.343	12107.016	806777.688	24.0511
4	54.787	7281.546	789622.125	23.5396



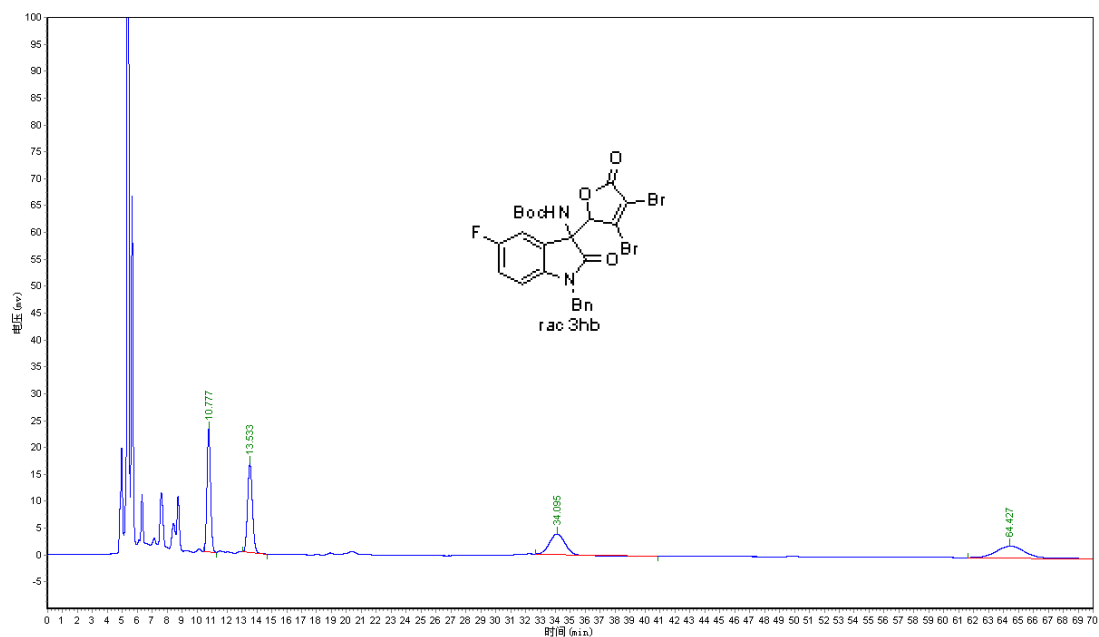
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.727	7568.125	118541.406	5.2823
2	13.835	84612.594	2125563.000	94.7177



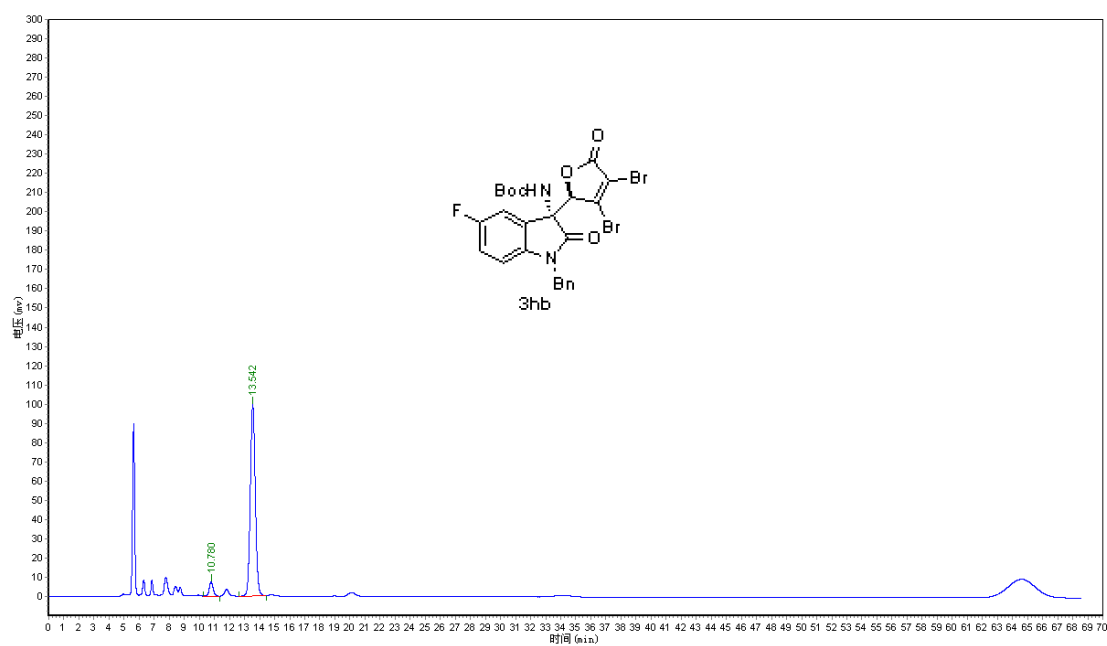
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.047	41847.223	635664.688	29.7358
2	25.643	9306.414	443756.438	20.7585
3	30.095	11551.470	616073.125	28.8193
4	104.445	2210.926	442217.000	20.6865



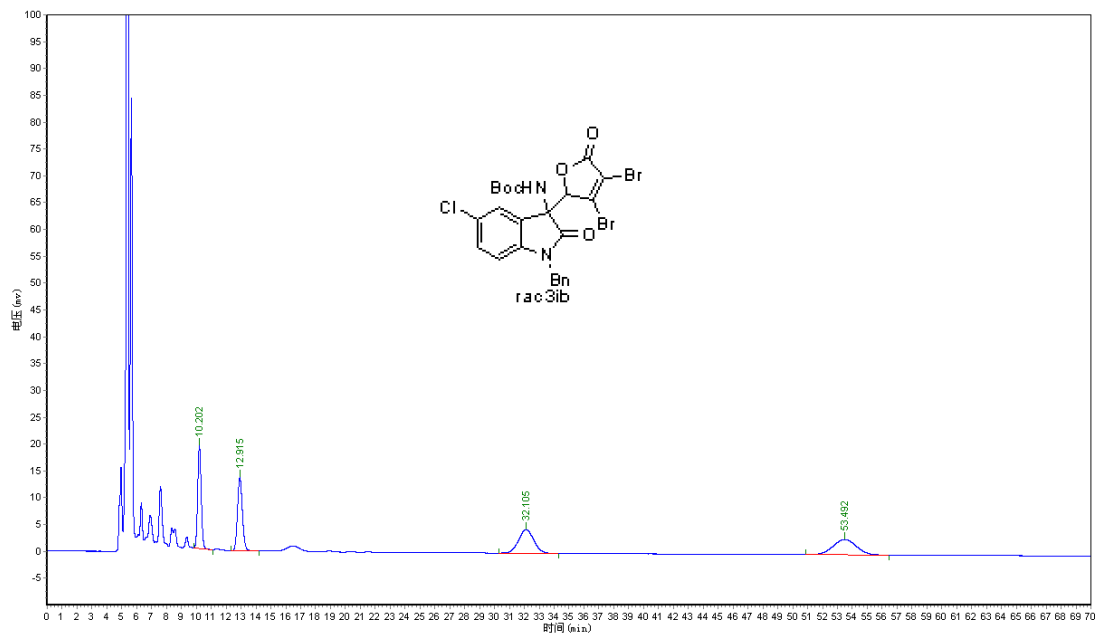
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.007	7981.455	107096.500	4.1231
2	29.882	47312.941	2490374.000	95.8769



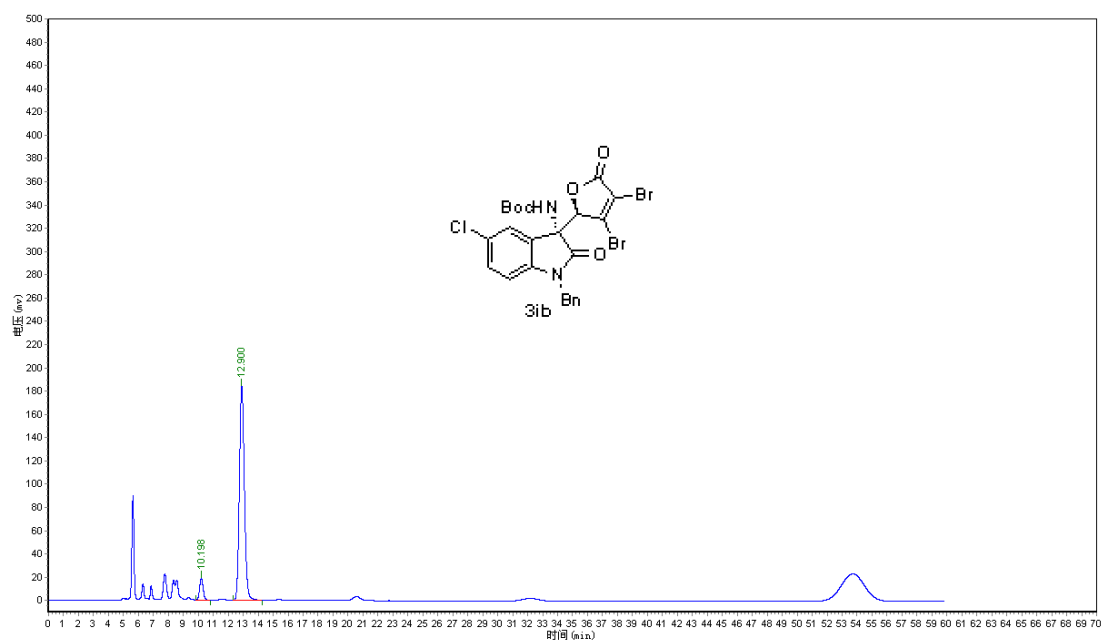
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.777	22769.932	382220.250	29.7858
2	13.533	16447.539	370783.469	28.8945
3	34.095	3796.958	247343.203	19.2750
4	64.427	2177.713	282883.813	22.0447



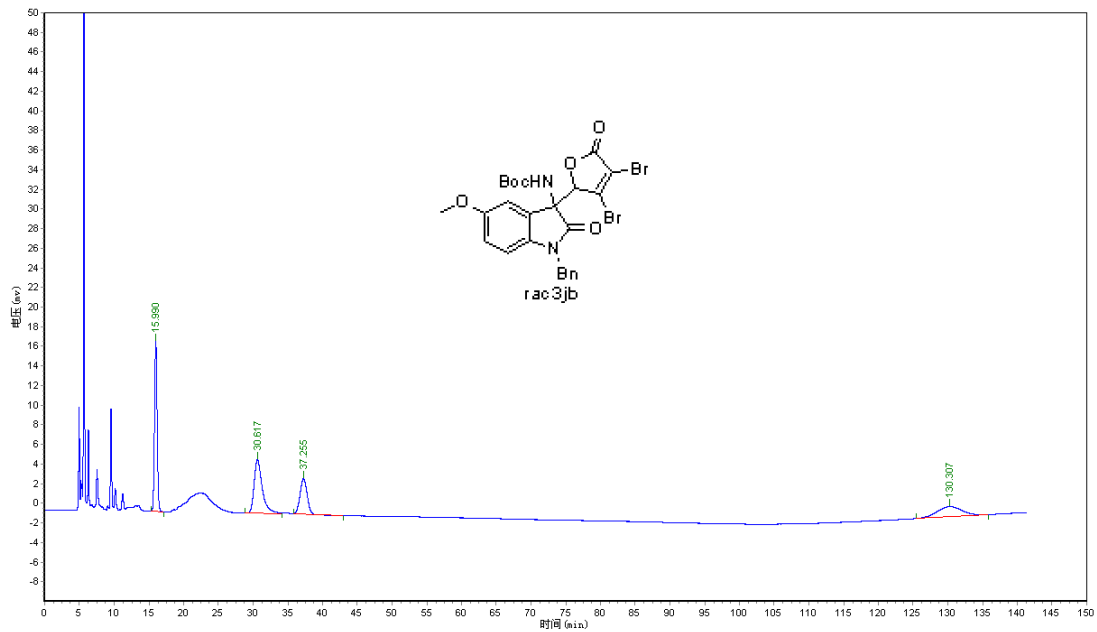
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.780	7374.627	132133.594	5.4149
2	13.542	99420.219	2308045.750	94.5851



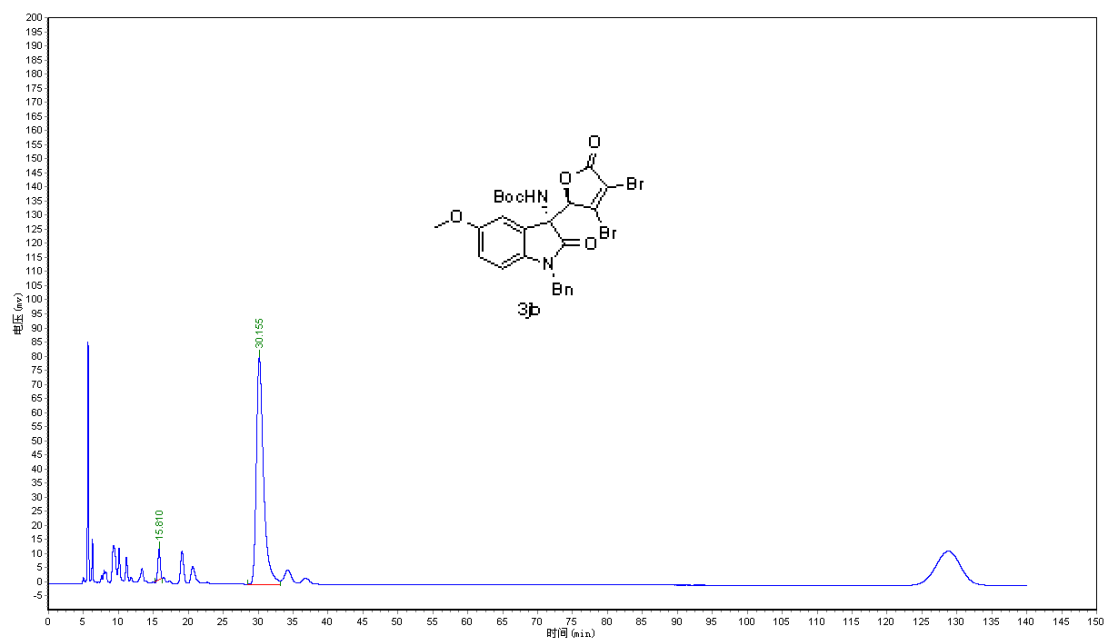
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.202	19115.482	318351.344	25.4320
2	12.915	13626.677	314105.531	25.0928
3	32.105	4368.990	310297.344	24.7885
4	53.492	2848.239	309022.719	24.6867



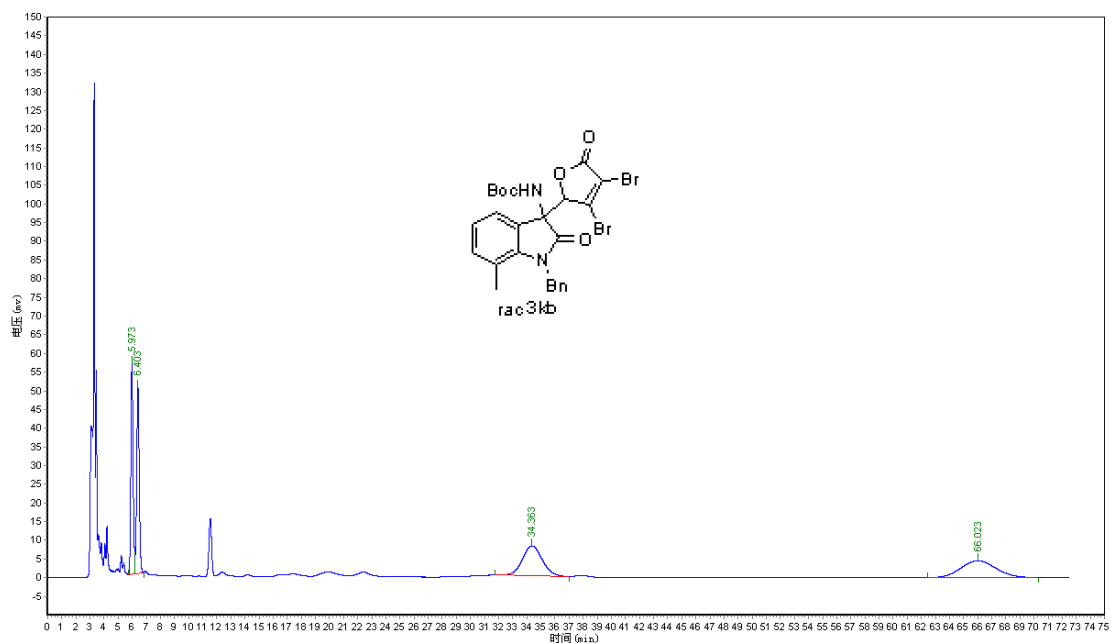
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	10.198	18761.348	307689.375	6.7163
2	12.900	183749.000	4273513.500	93.2837



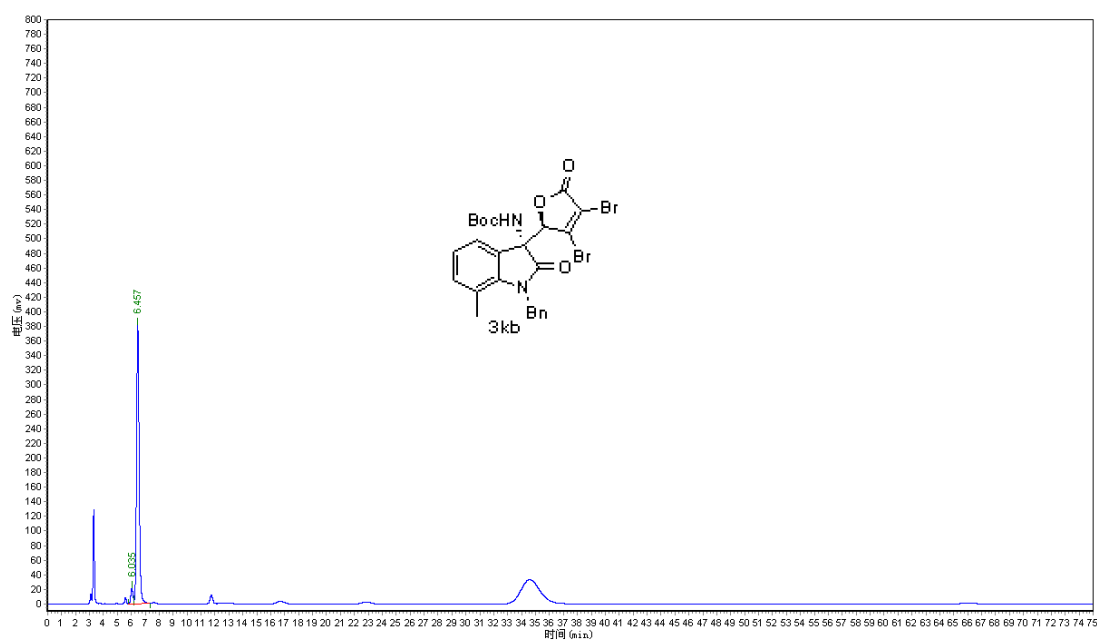
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	15.990	17404.861	459610.719	32.0390
2	30.617	5493.610	456100.531	31.7943
3	37.255	3637.689	258973.391	18.0528
4	130.307	1037.633	259849.500	18.1139



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	15.810	10907.018	262422.906	4.3448
2	30.155	80527.727	5777490.000	95.6552

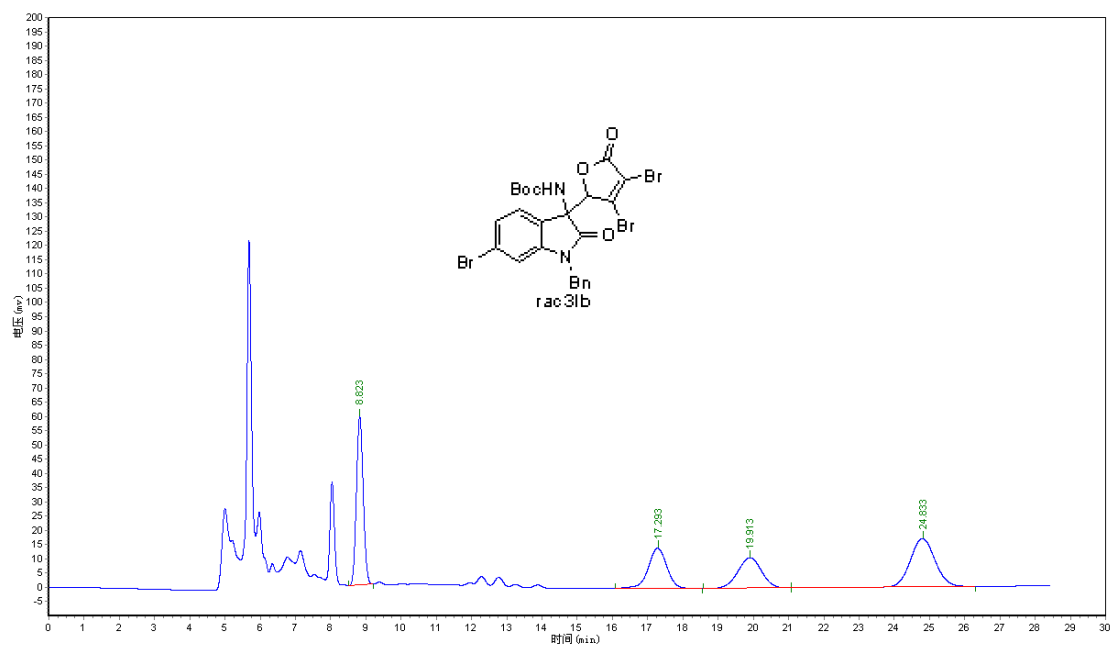


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	5.973	56446.207	628841.875	21.4980
2	6.403	50191.805	637902.063	21.8077
3	34.363	8275.669	873569.938	29.8644
4	66.023	4499.655	784807.063	26.8299

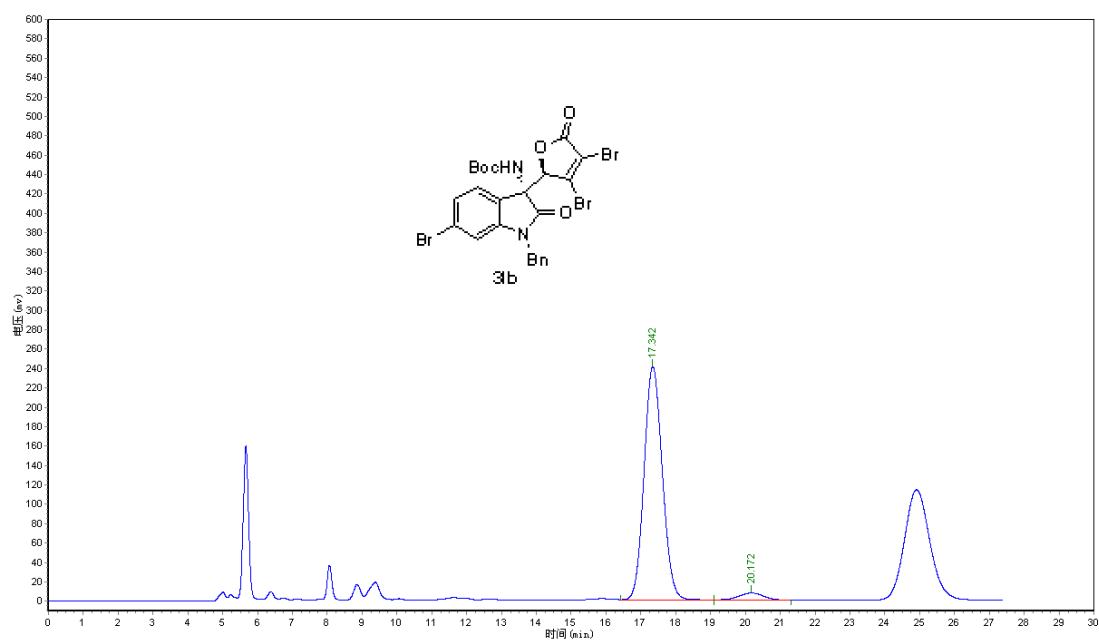


Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	6.035	20915.123	233090.297	4.4687
2	6.457	381159.969	4982919.500	95.5313

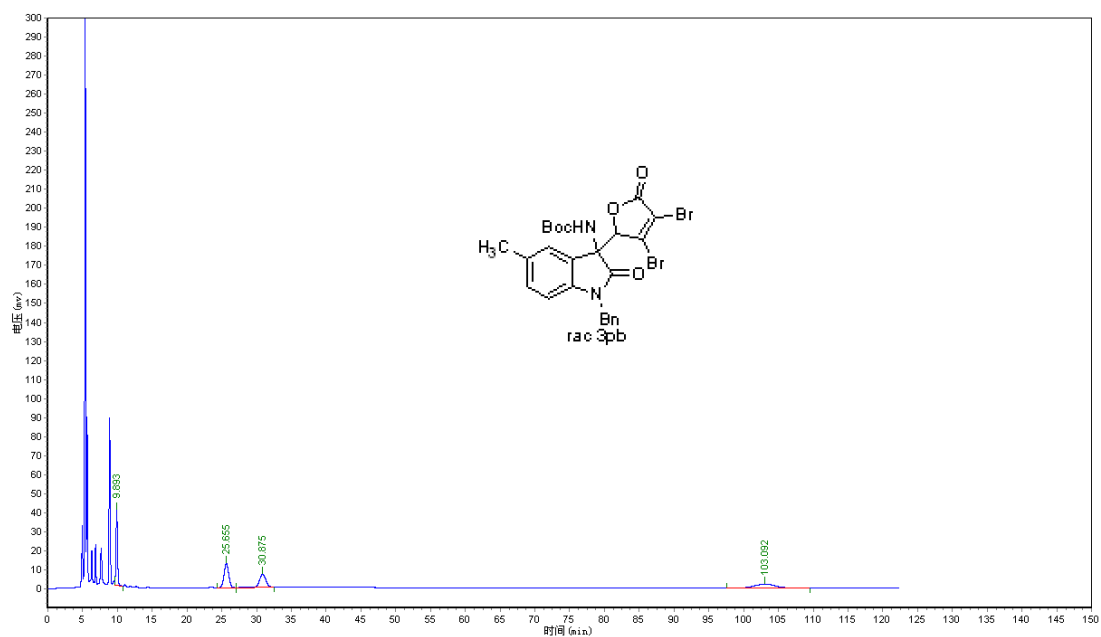




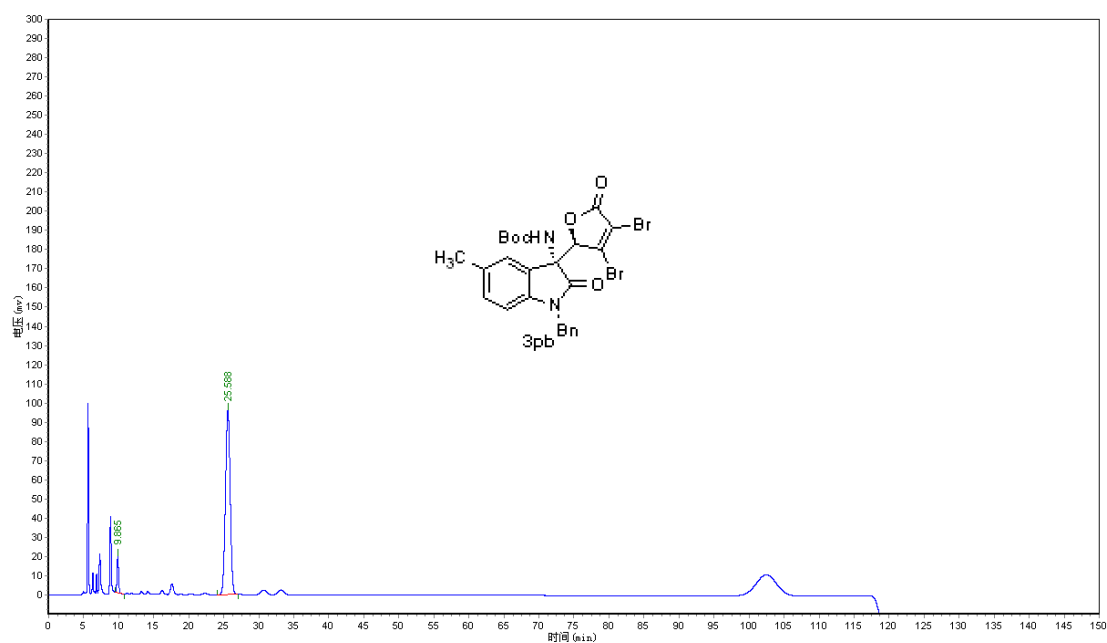
Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	8.823	59255.160	800350.438	30.2949
2	17.293	14154.412	522517.906	19.7784
3	19.913	10648.048	495655.000	18.7616
4	24.833	16890.541	823338.188	31.1651



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	17.342	240486.109	8729720.000	96.2862
2	20.172	7049.230	336708.281	3.7138



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	9.893	39545.578	627360.375	29.6823
2	25.655	12707.087	598752.563	28.3288
3	30.875	6977.250	441629.469	20.8948
4	103.092	2086.019	445841.500	21.0941



Peak	RT(min.)	Height(mV*sec)	Area(mV)	Area(%)
1	9.865	19012.695	295170.750	6.1180
2	25.588	96067.805	4529464.500	93.8820

## References

- 1 W.-J. Wen, D. Wang, J.-C. Feng, P. Li, D.-P. Zhao and R. Wang, *Org. Lett.*, **2012**, *14*, 2512.
- 2 F. Bellina and R. Rossi, *Practical Synthetic Procedures*, **2001**, 1887.
- 3 (a) B. Vákuyla, S. Varga, A. Cs ámpai and T. So ós, *Org. Lett.*, **2005**, *7*, 1967; (b) W. Yang and D.-M. Du, *Org. Lett.*, **2012**, *12*, 5450; (c) H.-M. Li, Y. Wang, L. Tang, F.-H. Wu, X.-F. Liu, C.-Y. Guo, B. M. Foxman and L. Deng, *Angew. Chem. Int. Ed.*, **2004**, *44*, 105.