

Organocatalytic Domino Michael–Alkylation Reaction: Highly Enantioselective Construction of Spiro- cyclopentanoneoxindoles and Tetronic acid Scaffolds

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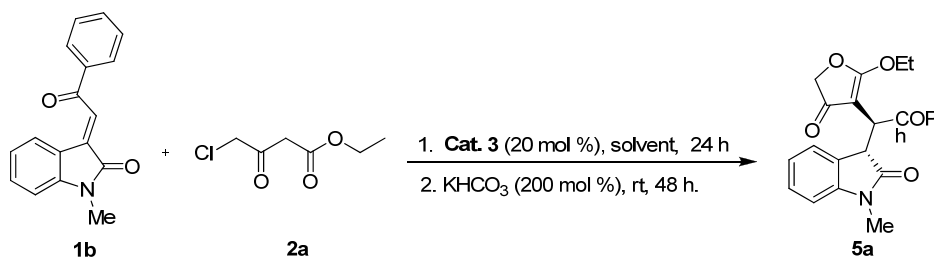
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1. Optimization of the reaction conditions of N-methyl protected methyleneindolinone **1b** with ethyl 4-chloroacetoacetate **2a**

A series of catalysts, solvents and reaction temperature were examined, and the results were summarized in Table 1. Quinine based thiourea catalyst **3d** afforded better results than catalysts **3a-3c**, **3e** with chiral diamine scaffolds (Table 1, entry 4 vs entries 1-3, 5). All solvents provided **5a** as the main product with moderate to good yields and enantioselectivities (Table 1, entries 6-9). In terms of yield and stereoselectivity, chloroform was chosen as the suitable solvent (Table 1, entry 4). Increasing or decreasing reaction temperature allowed slight decreases in stereoselectivities (Table 1, entry 4 vs entries 10, 11). Based on these screenings, a set of optimal reaction conditions were established: 0.2 mmol of **1b**, 0.2 mmol of **2a** and catalyst **3d** (20 mol %) were stirred in chloroform (1.0 mL) for 24 h at room temperature, then 2 equivs of KHCO_3 was added and the crude mixture was stirred for other 48 h at room temperature.

Table 1. Optimization of the reaction conditions ^a



Entry	Cat.	solvent	T (°C)	yield (%) ^b	dr ^c	ee (%) ^d
1	3a	CHCl_3	25	70	1.0:1	51 ^e /49 ^e
2	3b	CHCl_3	25	68	1:1.2	58 ^e /51 ^e
3	3c	CHCl_3	25	65	1.3:1	42/64
4	3d	CHCl_3	25	72	1.2:1	84/>99
5	3e	CHCl_3	25	50	1.5:1	36/55
6	3d	CH_2Cl_2	25	65	1.1:1	86/96
7	3d	PhCH_3	25	70	1:1.6	87/85
8	3d	THF	25	50	1:1.4	46/48
9	3d	EtOAc	25	71	1:1.2	62/62
10	3d	CHCl_3	45	70	1.3:1	82/94
11	3d	CHCl_3	-5	73	1.6:1	84/95

^a Unless otherwise noted, the reaction was performed on a 0.2 mmol scale in 1.0 mL solvent. ^b Isolated yields of mixture of diastereomers. ^c Determined by chiral HPLC

analysis; dr and ee value of the major product **5a**.^d The major diastereomers, determined by chiral HPLC analysis.^e Contrary configuration to **5f**.

2. 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). Racemic products were obtained from corresponding substrates catalyzed by Et₃N or Na₂CO₃ at room temperature. NMR spectra were recorded with tetramethylsilane as the internal standard. ¹H NMR spectra were recorded at 300 MHz, and ¹³C NMR spectra were recorded at 75 MHz (Bruker Avance). Chemical shifts (δ) are reported in ppm downfield from CDCl₃ (δ = 7.26 ppm) for ¹H NMR and relative to the central CDCl₃ resonance (δ = 77.0 ppm) for ¹³C NMR spectroscopy. The following abbreviations were used to describe peak patterns where appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants were reported in Hertz (Hz). Flash column chromatography was carried out using silica gel eluting with ethyl acetate and petroleum ether. Reactions were monitored by TLC and visualized with ultraviolet light. Optical rotations were measured on a Perkin-Elmer 341 polarimeter. The enantiomeric excess (ee) of the products were determined by HPLC using Daicel Chiralpak (AD, AD-H, AY or IC) columns. Owing to the difficulty to isolate the diastereomers, yield and optical rotation below refer to mixture of diastereomers.

3. Preparation of materials and catalysts

Methyleneindolinones **1** were prepared following literature procedures.^{1, 2} Ethyl 4-chloroacetoacetate **2a** was commercially available and purified by silica gel column chromatography. Methyl 4-chloroacetoacetate **2b** was commercially available and used as received. Methyl 4-bromoacetoacetate **2c** was prepared following literature procedures.³ Catalyst **3a** was synthesized according to literature procedure.⁴ Catalyst **3d** was synthesized according to literature procedure.⁵

4. General procedure for asymmetric domino Michael-Alkylation reaction of methyleneindolinones with γ-halogenated-β-ketoesters

Typical procedure for asymmetric Michael-Alkylation domino reaction with

N-Boc protected methyleneindolinones

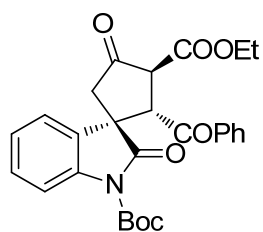
A stirred solution of catalyst **3a** (5 mol %), N-Boc protected methyleneindolinones **1** (0.20 mmol) in THF (1.0 mL) was cooled to -30 °C and γ -halogenated- β - ketoesters **2** (0.2 mmol) was added at the same temperature. The reaction mixture was stirred at -30 °C for 2 days, then KHCO₃ (0.4 mmol) was added. The reaction solution was stirred for 1 day at rt, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford pure products **4**.

Typical procedure for asymmetric Michael-Alkylation domino reaction with N-alkyl protected methyleneindolinones

A solution of catalyst **3d** (20 mol %), N-alkyl protected methyleneindolinones **1** (0.20 mmol) and ethyl 4-chloro-acetoacetate **2a** (0.2 mmol) in CHCl₃ (1.0 mL) was stirred at rt for 1 days, then KHCO₃ (0.4 mmol) was added. The reaction solution was stirred for 2 day at the same temperature, and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford pure products **5**.

5. Characterization Data and HPLC Conditions of Product 4 and 5

1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-benzoyl-2', 3-dioxo-1', 2'-dihydrospiro



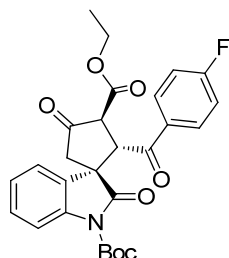
[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (**4a**)

94% yield, $[\alpha]_D^{20} = +151.5$ (c 1.2, CH₂Cl₂); (dr = 7.3:1, >99% ee for the major diastereomer, 90% ee for the minor diastereomer);

HPLC conditions: major diastereomer: Chiralcel IC column, Hexane/EtOH (0.1% Et₂NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 27.20 min (major) and 22.11 min (minor); minor diastereomer: Chiralcel AD column, Hexane/EtOH (0.1% TFA) = 90:10, flow rate 1.0 mL/min, UV detection at 254 nm, retention time: major diastereomer: 7.77 min (major) and 10.37 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.31 (t, *J* = 7.11 Hz, 3H), 1.51 (s, 9H), 2.65 (d, *J* = 18.33 Hz, 1H), 3.24 (d, *J* = 18.33 Hz, 1H), 4.22-4.29 (m, 2H), 4.54 (d, *J* = 11.31 Hz, 1H), 5.12 (d, *J* = 11.31 Hz, 1H), 6.98 (d, *J* = 6.72 Hz, 1H), 7.11 (t, *J* = 7.56 Hz, 1H), 7.20 (d, *J* = 8.40 Hz, 1H), 7.29 (t, *J* = 7.47 Hz, 2H), 7.36 (d, *J* = 7.38 Hz, 2H), 7.46 (d, *J* = 7.92 Hz, 2H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.1, 27.8, 50.0, 55.2, 57.7, 60.4, 62.2, 84.5, 114.1, 123.5, 124.9, 127.4, 128.1, 128.8, 132.7,

133.3, 136.1, 138.4, 147.7, 166.4, 176.1, 196.1, 204.6; HRMS (ESI) Calcd. for $C_{27}H_{27}NNaO_7 [M+Na]^+$: 500.1685; Found: 500.1669.

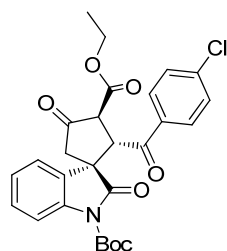
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(4-fluorobenzoyl)-2', 3-dioxo-1', 2'-



dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4b)

92% yield, $[\alpha]_D^{20} = +68.5$ (c 1.2, CH_2Cl_2); (dr = 8.1:1, 96% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 23.84 min (major) and 18.64 min (minor). 1H NMR ($CDCl_3$, 300 MHz) δ 1.30 (t, $J = 7.11$ Hz, 3H), 1.54 (s, 9H), 2.66 (d, $J = 18.33$ Hz, 1H), 3.24 (d, $J = 18.39$ Hz, 1H), 4.23-4.30 (m, 2H), 4.53 (d, $J = 11.31$ Hz, 1H), 5.08 (d, $J = 11.31$ Hz, 1H), 6.95-7.01 (m, 3H), 7.13 (t, $J = 7.50$ Hz, 1H), 7.24 (t, $J = 7.14$ Hz, 1H), 7.41-7.50 (m, 3H); ^{13}C NMR ($CDCl_3$, 75 Hz) δ 14.1, 27.8, 49.8, 55.6, 57.7, 60.4, 62.3, 84.9, 114.9, 115.4, 123.6, 124.8, 127.0, 128.9, 130.1, 132.5, 138.4, 147.7, 164.0, 166.3, 176.1, 194.5, 204.4; HRMS (ESI) Calcd. for $C_{27}H_{26}FNNaO_7 [M+Na]^+$: 518.1591; Found: 518.1586.

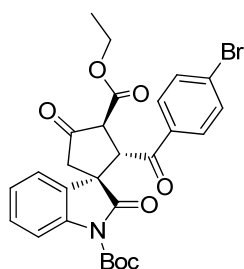
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(4-chlorobenzoyl)-2', 3-dioxo-1',



2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4c)

95% yield, $[\alpha]_D^{20} = +49.0$ (c 1.9, CH_2Cl_2); (dr = 10.2:1, 90% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 24.02 min (major) and 22.84 min (minor). 1H NMR ($CDCl_3$, 300 MHz) δ 1.31 (t, $J = 7.14$ Hz, 3H), 1.55 (s, 9 H), 2.65 (d, $J = 18.33$ Hz, 1H), 3.23 (d, $J = 18.39$ Hz, 1H), 4.23-4.30 (m, 2H), 4.52 (d, $J = 11.31$ Hz, 1H), 5.07 (d, $J = 11.34$ Hz, 1H), 6.98 (d, $J = 7.41$ Hz, 1H), 7.10-7.15 (m, 1H), 7.21-7.35 (m, 5H), 7.49 (d, $J = 8.22$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 Hz) δ 14.1, 27.9, 49.8, 55.6, 57.8, 60.5, 62.3, 84.9, 114.1, 123.6, 124.8, 127.0, 128.5, 128.9, 129.7, 134.4, 138.3, 139.8, 147.6, 166.3, 176.0, 195.0, 204.3; HRMS (ESI) Calcd. for $C_{27}H_{26}ClNNaO_7 [M+Na]^+$: 534.1295; Found: 534.1286.

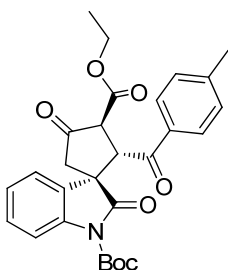
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(4-bromobenzoyl)-2', 3-dioxo-1',



2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4d)

93% yield, $[\alpha]_D^{20} = +46.3$ (c 1.2, CH_2Cl_2); (dr = 7.7:1, 82% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 24.47 min (major) and 19.78 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.32 (t, $J = 7.14$ Hz, 3H), 1.56 (s, 9H), 2.66 (d, $J = 18.36$ Hz, 1H), 3.24 (d, $J = 18.39$ Hz, 1H), 4.23-4.30 (m, 2H), 4.52 (d, $J = 11.31$ Hz, 1H), 5.07 (d, $J = 11.31$ Hz, 1H), 6.97 (d, $J = 7.29$ Hz, 1H), 7.12 (t, $J = 7.47$ Hz, 1H), 7.21-7.26 (m, 3H), 7.43-7.50 (m, 3H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 28.0, 49.9, 55.6, 57.8, 60.5, 62.4, 85.0, 114.9, 121.9, 123.6, 125.0, 127.0, 128.9, 129.8, 131.7, 134.8, 138.4, 147.7, 166.3, 176.0, 195.2, 204.3; HRMS (ESI) Calcd. for $\text{C}_{27}\text{H}_{26}\text{BrNNaO}_7$ $[\text{M}+\text{Na}]^+$: 578.0790; Found: 578.0768.

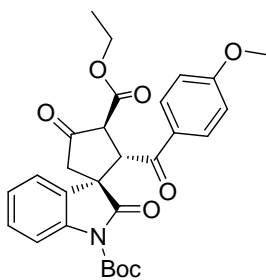
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(4-methylbenzoyl)-2', 3-dioxo-1',



2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4e)

96% yield, $[\alpha]_D^{20} = +99.1$ (c 1.5, CH_2Cl_2); (dr = 7.2:1, 91% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 32.60 min (major) and 28.75 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.31 (t, $J = 7.11$ Hz, 3H), 1.53 (s, 9H), 2.30 (s, 3H), 2.66 (d, $J = 18.30$ Hz, 1H), 3.25 (d, $J = 18.36$ Hz, 1H), 4.22-4.29 (m, 2H), 4.56 (d, $J = 11.31$ Hz, 1H), 5.10 (d, $J = 11.34$ Hz, 1H), 6.99 (d, $J = 6.93$ Hz, 1H), 7.00-7.14 (d, $J = 8.01$ Hz, 3H), 7.19-7.26 (m, 1H), 7.32 (d, $J = 8.16$ Hz, 2H), 7.49 (d, $J = 8.07$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.0, 21.5, 27.8, 49.9, 55.4, 57.4, 60.3, 62.2, 84.4, 114.8, 121.8, 123.6, 124.7, 127.2, 129.0, 133.4, 138.4, 144.1, 147.8, 166.4, 168.7, 176.1, 195.4, 204.8; HRMS (ESI) Calcd. for $\text{C}_{28}\text{H}_{29}\text{NNaO}_7$ $[\text{M}+\text{Na}]^+$: 514.1842; Found: 514.1816.

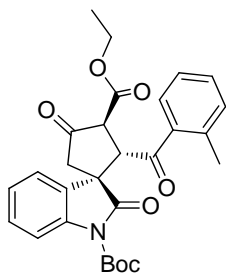
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(4-methoxybenzoyl)-2', 3-dioxo-1', 2'-



dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4f)

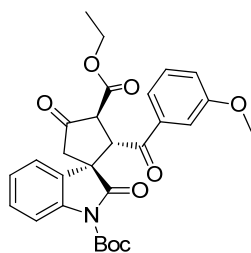
92% yield, $[\alpha]_D^{20} = +71.2$ (c 2.3, CH_2Cl_2); (dr = 12.1:1, 92% ee for the major diastereomer); HPLC conditions: Chiralcel AD column, Hexane/EtOH (0.1% Et_2NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 13.60 min (major) and 17.18 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.30 (t, $J = 7.11$ Hz, 3H), 1.52 (s, 9H), 2.66 (d, $J = 18.30$ Hz, 1H), 3.25 (d, $J = 18.33$ Hz, 1H), 3.79 (s, 3H), 4.21-4.28 (m, 2H), 4.56 (d, $J = 11.34$ Hz, 1H), 5.08 (d, $J = 11.37$ Hz, 1H), 6.78 (d, $J = 8.82$ Hz, 2H), 6.99 (d, $J = 7.47$ Hz, 1H), 7.11 (t, $J = 7.56$ Hz, 1H), 7.22 (t, $J = 7.80$ Hz, 1H), 7.43-7.50 (m, 3H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 27.8, 49.8, 52.1, 55.1, 55.4, 55.4, 62.2, 84.5, 113.5, 114.8, 123.7, 124.6, 127.2, 128.8, 129.7, 130.9, 138.3, 147.8, 163.7, 166.5, 176.3, 193.9, 204.9; HRMS (ESI) Calcd. for $\text{C}_{28}\text{H}_{29}\text{NNaO}_8$ $[\text{M}+\text{Na}]^+$: 530.1791; Found: 530.1761.

1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(2-methylbenzoyl)-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4g)



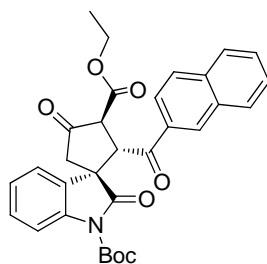
93% yield, $[\alpha]_D^{20} = +57.5$ (c 2.5, CH_2Cl_2); (dr = 3.6:1, 84% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 27.96 min (major) and 17.57 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.34 (t, $J = 7.11$ Hz, 3H), 1.51 (s, 12H), 2.61 (d, $J = 18.27$ Hz, 1H), 3.20 (d, $J = 18.30$ Hz, 1H), 4.25-4.33 (m, 2H), 4.49 (d, $J = 11.64$ Hz, 1H), 5.12 (d, $J = 11.64$ Hz, 1H), 7.11-7.16 (m, 2H), 7.19-7.30 (m, 5H), 7.58 (d, $J = 8.16$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 19.0, 28.0, 50.6, 55.1, 57.4, 59.5, 62.3, 84.4, 115.3, 121.9, 123.4, 124.8, 125.4, 127.8, 129.6, 131.2, 132.0, 135.2, 138.9, 147.7, 166.5, 168.2, 175.5, 198.0, 204.6; HRMS (ESI) Calcd. for $\text{C}_{28}\text{H}_{29}\text{NNaO}_7$ $[\text{M}+\text{Na}]^+$: 514.1842; Found: 514.1823.

1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(3-methoxybenzoyl)-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4h)



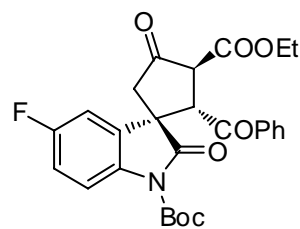
92% yield, $[\alpha]_D^{20} = +105.7$ (c 2.1, CH_2Cl_2); (dr = 12.2:1, >99% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 31.71 min (major) and 22.78 min (minor). ^1H

NMR (CDCl_3 , 300 MHz) δ 1.32 (t, $J = 7.11$ Hz, 3H), 1.53 (s, 9H), 2.65 (d, $J = 18.30$ Hz, 1H), 3.24 (d, $J = 18.33$ Hz, 1H), 3.74 (s, 3H), 4.24-4.31 (m, 2H), 4.54 (d, $J = 11.31$ Hz, 1H), 5.10 (d, $J = 11.31$ Hz, 1H), 6.77 (s, 1H), 6.97-7.01 (m, 3H), 7.05-7.26 (m, 3H), 7.51 (d, $J = 8.16$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 27.8, 50.0, 51.8, 55.2, 58.0, 60.4, 62.3, 84.7, 110.9, 114.1, 114.9, 120.4, 123.5, 124.7, 127.3, 128.7, 129.0, 137.4, 138.7, 147.8, 159.3, 166.4, 176.1, 196.1, 204.6; HRMS (ESI) Calcd. for $\text{C}_{28}\text{H}_{29}\text{NNaO}_8$ $[\text{M}+\text{Na}]^+$: 530.1791; Found: 530.1788.



1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-(naphthalene-2-carbonyl)-2', 3'-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4i)

92% yield, $[\alpha]_D^{20} = +6.9$ (c 1.1, CH_2Cl_2); (dr = 10.1:1, 90% ee for the major diastereomer); HPLC conditions: Chiralcel IC column, Hexane/EtOH (0.1% Et_2NH) = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 53.55 min (major) and 33.11 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.15 (s, 9H), 1.33 (t, $J = 7.11$ Hz, 3H), 2.70 (d, $J = 18.27$ Hz, 1H), 3.30 (d, $J = 18.30$ Hz, 1H), 4.25-4.32 (m, 2H), 4.63 (d, $J = 11.34$ Hz, 1H), 5.31 (d, $J = 11.37$ Hz, 1H), 7.04 (d, $J = 7.38$ Hz, 1H), 7.15-7.27 (m, 4H), 7.40 (d, $J = 8.04$ Hz, 1H), 7.53-7.57 (m, 3H), 7.67 (d, $J = 8.58$ Hz, 1H), 7.78 (d, $J = 8.31$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 27.3, 44.1, 49.9, 55.3, 60.4, 62.3, 84.3, 114.0, 114.9, 123.3, 123.6, 124.9, 126.9, 127.2, 127.5, 128.4, 128.7, 129.4, 130.1, 131.9, 133.3, 135.5, 138.4, 147.5, 166.5, 176.0, 195.8, 204.7; HRMS (ESI) Calcd. for $\text{C}_{31}\text{H}_{29}\text{NNaO}_7$ $[\text{M}+\text{Na}]^+$: 550.1842; Found: 550.1809.

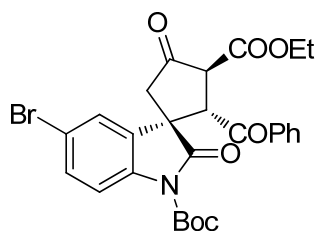


1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-benzoyl-5'-fluoro-2', 3'-dioxo-1',

2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4j)

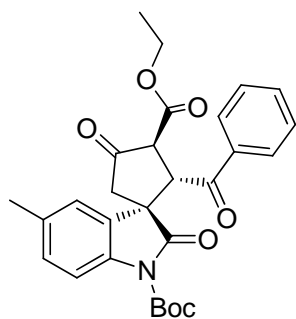
92% yield, $[\alpha]_D^{20} = +81.5$ (c 1.8, CH₂Cl₂); (dr = 7.0:1, 89% ee for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 11.07 min (major) and 40.46 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.32 (t, $J = 7.08$ Hz, 3H), 1.51 (s, 9 H), 2.65 (d, $J = 18.39$ Hz, 1H), 3.25 (d, $J = 18.42$ Hz, 1H), 4.23-4.30 (m, 2H), 4.50 (d, $J = 11.37$ Hz, 1H), 5.12 (d, $J = 11.37$ Hz, 1H), 6.72 (d, $J = 7.50$ Hz, 1H), 7.27 (d, $J = 7.11$ Hz, 1H), 7.32 (d, $J = 7.74$ Hz, 2H), 7.40-7.52 (m, 4H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.1, 28.0, 49.7, 55.4, 57.5, 60.5, 62.3, 84.8, 111.1, 115.9, 116.4, 127.5, 128.0, 128.7, 133.0, 133.5, 135.9, 147.7, 158.1, 166.2, 175.7, 195.8, 204.1; HRMS (ESI) Calcd. for C₂₇H₂₆FNNaO₇ [M+Na]⁺: 518.1591; Found: 518.1579.

1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-benzoyl-5'-bromo-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4k)



90% yield, $[\alpha]_D^{20} = +25.2$ (c 1.5, CH₂Cl₂); (dr = 2.0:1, 55% ee for the major diastereomer); HPLC conditions: Chiralcel AD column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 6.92 min (major) and 8.39 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.17 (t, $J = 7.11$ Hz, 3H), 1.63 (s, 9H), 2.74 (d, $J = 18.12$ Hz, 1H), 3.58 (d, $J = 18.18$ Hz, 1H), 4.12-4.14 (m, 2H), 4.37 (d, $J = 11.76$ Hz, 1H), 5.79 (d, $J = 11.79$ Hz, 1H), 7.25-7.30 (m, 5H), 7.62 (d, $J = 7.20$ Hz, 2H), 7.72 (d, $J = 8.19$ Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.0, 28.0, 44.7, 51.6, 53.6, 56.7, 62.3, 85.1, 113.6, 114.5, 118.1, 127.6, 128.1, 128.9, 130.5, 133.3, 136.2, 142.4, 148.4, 166.8, 176.2, 196.8, 205.5; HRMS (ESI) Calcd. for C₂₇H₂₆BrNNaO₇ [M+Na]⁺: 578.0790; Found: 578.0785.

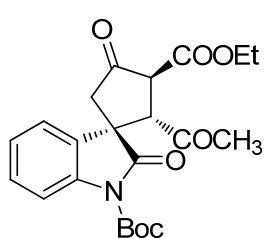
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-benzoyl-5'-methyl-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4l)



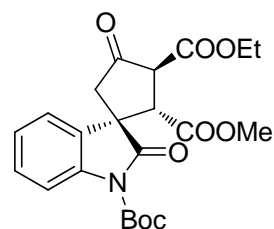
88% yield, $[\alpha]_D^{20} = +35.6$ (c 1.8, CH₂Cl₂); (dr = 2.0:1, 66% ee for the major diastereomer); HPLC conditions: Chiralcel IC

column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 12.95 min (major) and 14.42 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.32 (t, *J* = 7.08 Hz, 3H), 1.51 (s, 9H), 2.30 (s, 3H), 2.67 (d, *J* = 17.88 Hz, 1H), 3.25 (d, *J* = 18.30 Hz, 1H), 4.12-4.18 (m, 2H), 4.27 (d, *J* = 11.52 Hz, 1H), 5.11 (d, *J* = 11.31 Hz, 1H), 6.78 (s, 1H), 7.19 (t, *J* = 7.12 Hz, 1H), 7.18-7.26 (m, 1H), 7.27-7.36 (m, 4H), 7.59 (t, *J* = 8.31 Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 13.9, 21.0, 28.0, 48.6, 51.9, 55.3, 57.6, 62.2, 84.3, 114.7, 115.3, 122.4, 127.2, 128.1, 128.3, 129.8, 133.3, 134.7, 136.2, 147.8, 166.5, 176.1, 196.2, 204.8; HRMS (ESI) Calcd. for C₂₈H₂₉NNaO₇ [M+Na]⁺: 514.1842; Found: 514.1836.

1'-tert-butyl 4-ethyl (1S, 4S, 5S)-5-acetyl-2', 3'-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4m)



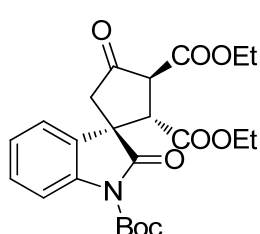
80% yield, [α]_D²⁰ = +30.2 (c 1.6, CH₂Cl₂); (dr = 1.3:1, 63% ee for the major diastereomer); HPLC conditions: Chiralcel AD column, Hexane/*i*-PrOH = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 7.13 min (major) and 10.17 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.32 (t, *J* = 7.14 Hz, 3H), 1.63 (s, 9H), 1.93 (s, 3H), 2.73 (d, *J* = 18.66 Hz, 2H), 4.19-4.36 (m, 4H), 6.94 (d, *J* = 7.50 Hz, 1H), 7.24 (t, *J* = 7.50 Hz, 1H), 7.31-7.37 (m, 1H), 7.89 (t, *J* = 8.04 Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.1, 28.0, 28.9, 49.2, 51.2, 55.8, 60.1, 62.3, 84.7, 115.6, 122.9, 124.9, 127.6, 129.6, 140.3, 148.9, 168.3, 175.9, 201.7, 203.0; HRMS (ESI) Calcd. for C₂₂H₂₅NNaO₇ [M+Na]⁺: 438.1529; Found: 438.1539.



1'-tert-butyl 4-ethyl 5-methyl (1S, 4S, 5S)-2', 3'-dioxo-1', 2'-dihydrospiro [cyclopentane-1, 3'-indole]-1', 4, 5-tricarboxylate (4n)

93% yield, [α]_D²⁰ = +182.8 (c 0.7, CH₂Cl₂); (dr = 1.9:1, 96% ee for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 11.34 min (major) and 12.48 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.35 (t, *J* = 4.26 Hz, 3H), 1.66 (s, 9H), 2.64 (d, *J* = 17.04 Hz, 1H), 3.13 (d, *J*

= 18.12 Hz, 1H), 3.31 (s, 3H), 4.14 (d, $J = 11.01$ Hz, 2H), 4.24-4.35 (m, 2H), 6.99 (d, $J = 7.50$ Hz, 1H), 7.12 (d, $J = 7.56$ Hz, 1H), 7.31-7.37 (m, 1H), 7.89 (t, $J = 8.16$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 28.0, 43.1, 50.0, 51.5, 53.2, 56.4, 62.3, 84.9, 115.4, 122.3, 124.9, 128.2, 139.3, 148.8, 166.2, 167.9, 168.8, 175.6, 204.1; HRMS (ESI) Calcd. for $\text{C}_{22}\text{H}_{25}\text{NNaO}_8$ $[\text{M}+\text{Na}]^+$: 454.1478; Found: 454.1474.

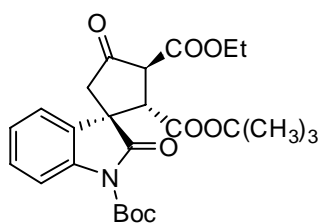


1'-tert-butyl 4, 5-diethyl (1S, 4S, 5S)-2', 3-dioxo-1', 2'-dihydrospiro

[cyclopentane-1, 3'-indole]-1', 4, 5-tricarboxylate (4o)

95% yield, $[\alpha]_{\text{D}}^{20} = +145.2$ (c 0.9, CH_2Cl_2); (dr = 1.6:1, 79% ee

for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et_2NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 10.09 min (major) and 14.33 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 0.84 (t, $J = 7.20$ Hz, 3H), 1.32 (t, $J = 7.14$ Hz, 3H), 1.66 (s, 9H), 2.64 (d, $J = 18.21$ Hz, 1H), 3.12 (d, $J = 18.12$ Hz, 1H), 3.72-3.77 (m, 2H), 4.12 (d, $J = 11.97$ Hz, 1H), 4.24-4.34 (m, 3H), 6.99 (d, $J = 7.47$ Hz, 1H), 7.12 (t, $J = 7.59$ Hz, 1H), 7.34 (t, $J = 7.62$ Hz, 1H), 7.90 (d, $J = 8.22$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 13.3, 14.1, 28.0, 43.3, 50.0, 53.1, 55.6, 61.5, 62.3, 84.9, 115.4, 122.3, 125.0, 129.5, 139.5, 148.8, 166.3, 168.3, 175.6, 177.3, 204.2; HRMS (ESI) Calcd. for $\text{C}_{23}\text{H}_{27}\text{NNaO}_8$ $[\text{M}+\text{Na}]^+$: 468.1634; Found: 468.1633.

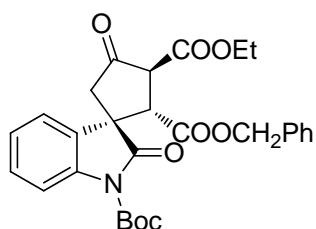


1', 5-di-tert-butyl 4-ethyl (1S, 4S, 5S)-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4, 5-tricarboxylate (4p)

90% yield, $[\alpha]_{\text{D}}^{20} = +106.2$ (c 0.8, CH_2Cl_2); (dr = 1.9:1,

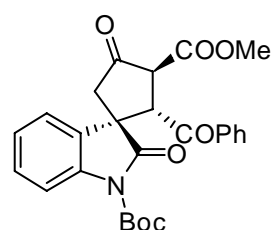
90% ee for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et_2NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: major diastereomer: 7.00 min (major) and 6.21 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.00 (s, 9H), 1.31 (t, $J = 3.12$ Hz, 3H), 1.64 (s, 9H), 2.61 (d, $J = 18.15$ Hz, 1H), 3.09 (d, $J = 18.09$ Hz, 1H), 4.25-4.30 (m, 4H), 7.00 (d, $J = 7.41$ Hz, 1H), 7.12 (t, $J = 7.59$ Hz, 1H), 7.35 (t, $J = 7.14$ Hz, 1H), 7.96 (d, $J = 8.19$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.1, 27.1, 27.9, 50.5, 51.1,

53.6, 55.7, 62.2, 82.6, 84.8, 115.3, 122.3, 125.0, 129.4, 139.6, 148.9, 166.5, 167.8, 175.7, 177.0, 204.4; HRMS (ESI) Calcd. for C₂₅H₃₁NNaO₈ [M+Na]⁺: 496.1947; Found: 496.1950.



5-benzyl 1'-tert-butyl 4-ethyl (1S, 4S, 5S)-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4, 5-tricarboxylate (4q)

91% yield, $[\alpha]_D^{20} = +76.4$ (c 1.0, CH₂Cl₂); (dr = 1.4:1, 81% ee for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 13.16 min (major) and 18.25 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.32 (t, *J* = 7.14 Hz, 3H), 1.58 (s, 9H), 2.60 (d, *J* = 18.12 Hz, 1H), 3.10 (d, *J* = 18.12 Hz, 1H), 4.15-4.29 (m, 2H), 4.40 (d, *J* = 11.85 Hz, 1H), 4.73 (s, 2H), 4.98 (d, *J* = 12.84 Hz, 1H), 6.95 (d, *J* = 8.25 Hz, 2H), 7.20-7.34 (m, 6H), 7.75 (d, *J* = 11.76 Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.0, 27.9, 49.4, 50.4, 52.6, 55.7, 62.3, 67.4, 84.7, 115.5, 121.7, 122.0, 124.7, 127.7, 128.2, 128.5, 129.4, 134.2, 139.2, 148.5, 166.2, 168.4, 175.4, 204.0; HRMS (ESI) Calcd. for C₂₈H₂₉NNaO₈



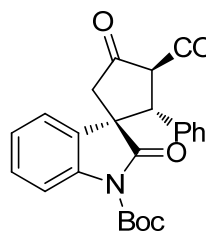
1'-tert-butyl 4-methyl (1S, 4S, 5S)-5-benzoyl-2', 3-dioxo-1', 2'-dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4r)

[M+Na]⁺: 530.1791; Found: 530.1788. 94% yield, $[\alpha]_D^{20} = +70.3$ (c 1.1, CH₂Cl₂); (dr = 6.3:1, 92% ee for the major diastereomer); HPLC conditions: Chiralcel AY column, Hexane/EtOH (0.1% Et₂NH) = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 17.55 min (major) and 44.55 min (minor). ¹H NMR (CDCl₃, 300 MHz) δ 1.52 (s, 9H), 2.66 (d, *J* = 17.37 Hz, 1H), 3.25 (d, *J* = 18.36 Hz, 1H), 3.82 (s, 3H), 4.58 (d, *J* = 11.25 Hz, 1H), 5.13 (d, *J* = 11.25 Hz, 1H), 6.97 (d, *J* = 7.50 Hz, 1H), 7.11 (t, *J* = 7.56 Hz, 1H), 7.20 (d, *J* = 7.89 Hz, 1H), 7.26 (t, *J* = 8.10 Hz, 2H), 7.36 (d, *J* = 7.41 Hz, 2H), 7.46 (t, *J* = 7.77 Hz, 2H); ¹³C NMR (CDCl₃, 75 Hz) δ 27.9, 44.0, 51.8, 53.2, 55.6, 57.4, 84.6, 114.9, 121.9, 123.5, 124.9, 127.4, 128.4, 129.4, 133.5, 136.0,

138.5, 147.7, 166.9, 176.1, 196.1, 204.6; HRMS (ESI) Calcd. for $C_{26}H_{25}NNaO_7$ $[M+Na]^+$: 486.1529; Found: 486.1530.

1'-tert-butyl 4-ethyl (1S, 4S, 5R)-2', 3-dioxo-5-phenyl-1', 2'-

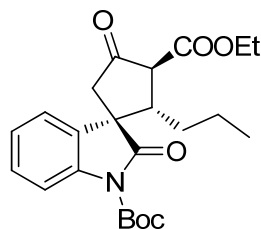
dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4s)



84% yield, $[\alpha]_D^{20} = +25.2$ (c 1.5, CH_2Cl_2); (dr = 2.7:1, 36% ee for the major diastereomer, 41% ee for the minor diastereomer); HPLC conditions: Chiralcel AD column, Hexane/EtOH = 95:5, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 7.20 min (major) and 9.97 min (minor); minor diastereomer: 11.11 min (major) and 13.01 min (minor). 1H NMR ($CDCl_3$, 300 MHz) δ 1.22 (t, $J = 7.17$ Hz, 3H), 1.50 (s, 9H), 2.94 (d, $J = 7.23$ Hz, 2H), 4.09-4.20 (m, 3H), 4.58 (d, $J = 13.41$ Hz, 1H), 6.87 (d, $J = 6.99$ Hz, 3H), 7.07-7.16 (m, 5H), 7.47 (d, $J = 7.25$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 Hz) δ 14.0, 27.9, 48.0, 55.1, 56.6, 57.7, 61.8, 84.2, 115.0, 122.1, 124.4, 127.1, 127.6, 128.0, 128.2, 129.2, 132.9, 140.0, 148.2, 168.2, 176.9, 205.6; HRMS (ESI) Calcd. for $C_{26}H_{27}NNaO_6$ $[M+Na]^+$: 472.1736; Found: 472.1730.

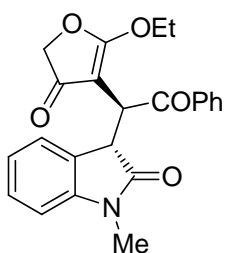
1'-tert-butyl 4-ethyl (1S, 4S, 5S)-2', 3-dioxo-5-propyl-1', 2'-

dihydrospiro[cyclopentane-1, 3'-indole]-1', 4-dicarboxylate (4t)



92% yield, $[\alpha]_D^{20} = +10.2$ (c 1.2, CH_2Cl_2); (dr = 1.3:1, 16% ee for the major diastereomer, 15% ee for the minor diastereomer); HPLC conditions: Chiralcel AD column, Hexane/*i*-PrOH = 90:10, flow rate 1.0 mL/min, UV detection at 220 nm, retention time: major diastereomer: 5.74 min (major) and 6.50 min (minor); minor diastereomer: 4.98 min (major) and 7.09 min (minor). 1H NMR ($CDCl_3$, 300 MHz) δ 0.73 (t, $J = 6.48$ Hz, 3H), 1.07-1.14 (m, 4H), 1.30 (t, $J = 7.11$ Hz, 3H), 1.65 (s, 9H), 2.54 (d, $J = 17.91$ Hz, 1H), 3.14 (d, $J = 17.91$ Hz, 1H), 3.75 (d, $J = 12.00$ Hz, 1H), 4.21-4.28 (m, 3H), 6.97 (d, $J = 7.50$ Hz, 1H), 7.36 (t, $J = 7.68$ Hz, 2H), 7.91 (d, $J = 8.22$ Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 Hz) δ 13.9, 20.7, 28.0, 31.4, 32.7, 48.7, 50.3, 53.6, 58.9, 61.9, 84.9, 115.1, 122.8, 124.8, 128.9, 139.2, 140.2, 148.9, 169.7, 176.5, 207.5; HRMS (ESI) Calcd. for $C_{23}H_{29}NNaO_6$ $[M+Na]^+$: 438.1893; Found: 438.1884.

(3S)-3-[(1S)-1-(2-ethoxy-4-oxo-4, 5-dihydrofuran-3-yl)-2-oxo-2-phenylethyl]-1-m

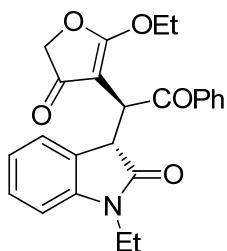


ethyl-2, 3-dihydro-1H-indol-2-one(5a)

72% yield, $[\alpha]_D^{20} = +10.6$ (c 2.2, CH₂Cl₂); (dr = 1.2:1, 84% ee for the major diastereomer, >99% ee for the minor diastereomer); HPLC conditions: Chiralcel IC column, Hexane/i-PrOH = 50:50, flow rate 0.7 mL/min, UV detection at 220 nm, retention time:

major diastereomer: 35.90 min (major) and 63.90 min (minor), minor diastereomer: 29.54 min (major). ¹H NMR (CDCl₃, 300 MHz) δ 1.35 (t, *J* = 7.11 Hz, 3H), 3.16 (s, 3H), 4.14 (m, 1H), 4.40-4.45 (m, 3H), 4.54 (d, *J* = 15.93 Hz, 1H), 4.71 (d, *J* = 15.96 Hz, 1H), 6.78 (d, *J* = 7.95 Hz, 1H), 6.87 (t, *J* = 7.47 Hz, 1H), 7.04 (d, *J* = 7.39 Hz, 1H), 7.26 (t, *J* = 7.82 Hz, 1H), 7.37-7.39 (m, 2H), 7.42-7.50 (m, 1H), 7.90 (d, *J* = 7.14 Hz, 1H), 8.03 (d, *J* = 7.20 Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 14.6, 26.1, 42.2, 45.1, 66.7, 75.0, 89.2, 107.7, 122.1, 124.7, 126.9, 127.9, 128.3, 128.5, 133.2, 135.7, 144.0, 175.6, 181.6, 193.8, 195.1; HRMS (ESI) Calcd. for C₂₃H₂₁NNaO₅ [M+Na]⁺: 414.1317; Found: 414.1316.

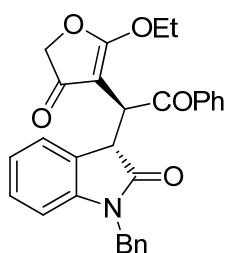
(3S)-3-[(1S)-1-(2-ethoxy-4-oxo-4,5-dihydrofuran-3-yl)-2-oxo-2-phenylethyl]-1-ethyl-2, 3-dihydro-1H-indol-2-one(5b)



70% yield, $[\alpha]_D^{20} = +20.7$ (c 1.8, CH₂Cl₂); (dr = 1.1:1, 96% ee for the major diastereomer, 94% ee for the minor diastereomer); HPLC conditions: Chiralcel IC column, Hexane/i-PrOH = 50:50, flow rate 0.7 mL/min, UV detection at 220 nm, retention time: major diastereomer: major diastereomer: 32.44 min (major) and 44.41 min (minor), minor diastereomer: 28.24 min (major) and 22.54 min (minor).

¹H NMR (CDCl₃, 300 MHz) δ 1.14 (t, *J* = 7.08 Hz, 3H), 1.23 (t, *J* = 6.93 Hz, 3H), 3.70-3.74 (m, 2H), 4.07-4.10 (m, 1H), 4.38-4.40 (m, 3H), 4.58 (d, *J* = 8.10 Hz, 1H), 4.67 (d, *J* = 8.35 Hz, 1H), 6.77 (d, *J* = 7.89 Hz, 1H), 6.82 (t, *J* = 9.51 Hz, 1H), 7.05 (d, *J* = 8.35 Hz, 1H), 7.20 (t, *J* = 7.62 Hz, 1H), 7.39 (d, *J* = 6.48 Hz, 2H), 7.48-7.50 (m, 1H), 7.89 (d, *J* = 7.47 Hz, 1H), 8.02 (d, *J* = 7.53 Hz, 1H); ¹³C NMR (CDCl₃, 75 Hz) δ 12.5, 14.6, 34.5, 42.2, 45.2, 66.6, 75.0, 89.3, 107.9, 121.9, 124.9, 127.2, 127.8, 128.3, 128.8, 133.1, 135.6, 143.1, 175.3, 181.5, 193.9, 195.1; HRMS (ESI) Calcd. for C₂₄H₂₃NNaO₅ [M+Na]⁺: 428.1474; Found: 428.1474.

(3S)-1-benzyl-3-[(1S)-1-(2-ethoxy-4-oxo-4,5-dihydrofuran-3-yl)-2-oxo-2-phenyl

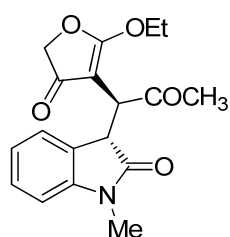


ethyl]-2,3-dihydro-1H-indol-2-one (5c)

78% yield, $[\alpha]_D^{20} = +36.2$ (c 1.6, CH_2Cl_2); (dr = 19.0:1, 99% ee for the major diastereomer, 94% ee for the minor diastereomer); HPLC conditions: Chiralcel IC column, Hexane/*i*-PrOH = 50:50, flow rate 0.7 mL/min, UV detection at 220 nm, retention time:

major diastereomer: 21.36 min (major) and 19.63 min (minor), minor diastereomer: 25.65 min (major) and 29.40 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.36 (t, $J = 7.08$ Hz, 3H), 4.38 (d, $J = 4.05$ Hz, 1H), 4.50-4.69 (m, 4H), 4.84 (s, 2H), 5.20 (d, $J = 4.25$ Hz, 1H), 6.68 (t, $J = 7.02$ Hz, 1H), 6.84 (t, $J = 7.58$ Hz, 1H), 7.10 (d, $J = 6.81$ Hz, 2H), 7.24-7.33 (m, 5H), 7.38-7.43 (m, 2H), 7.92 (d, $J = 7.29$ Hz, 1H), 8.05 (d, $J = 7.32$ Hz, 2H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.6, 29.6, 42.6, 45.2, 66.6, 75.0, 89.3, 108.7, 122.1, 124.8, 125.8, 127.3, 127.8, 128.1, 128.3, 128.5, 128.6, 132.8, 133.1, 135.8, 143.1, 175.8, 181.4, 193.8, 195.0; HRMS (ESI) Calcd. for $\text{C}_{29}\text{H}_{25}\text{NNaO}_5$ $[\text{M}+\text{Na}]^+$: 490.1630; Found: 490.1631.

(3S)-3-[(1S)-1-(2-ethoxy-4-oxo-4,5-dihydrofuran-3-yl)-2-oxopropyl]-1-methyl-2,

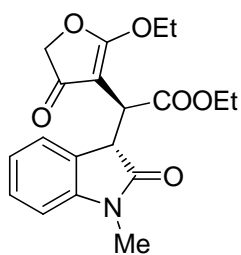


3-dihydro-1H-indol-2-one(5d)

75% yield, $[\alpha]_D^{20} = +15.2$ (c 1.5, CH_2Cl_2); (dr = 1.6:1, 91% ee for the major diastereomer, 89% ee for the minor diastereomer); HPLC conditions: Chiralcel AD-H column, Hexane/*i*-PrOH = 80:20, flow rate 1.0 mL/min, UV detection at 220 nm, retention

time: major diastereomer: 10.30 min (major) and 11.88 min (minor), minor diastereomer: 9.10 min (major) and 13.09 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.35 (t, $J = 7.11$ Hz, 3H), 2.12 (s, 3H), 3.15 (s, 3H), 3.79 (d, $J = 9.45$ Hz, 1H), 4.28-4.33 (m, 2H), 4.44 (d, $J = 4.44$ Hz, 2H), 4.69 (d, $J = 2.97$ Hz, 1H), 6.73 (d, $J = 7.53$ Hz, 1H), 6.94-6.97 (m, 2H), 7.09 (d, $J = 7.35$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.7, 28.7, 29.7, 44.8, 47.9, 53.4, 66.8, 75.2, 107.3, 122.2, 124.5, 125.8, 127.9, 144.6, 176.7, 181.8, 194.4, 204.8; HRMS (ESI) Calcd. for $\text{C}_{18}\text{H}_{19}\text{NNaO}_5$ $[\text{M}+\text{Na}]^+$: 352.1161; Found: 352.1161.

Ethyl (2S)-2-(2-ethoxy-4-oxo-4,5-dihydrofuran-3-yl)-2-[(3S)-1-methyl-2-oxo-2,3-

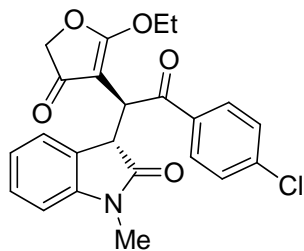


dihydro-1H-indol-3-yl] acetate (5e)

73% yield, $[\alpha]_D^{20} = +23.4$ (c 0.8, CH_2Cl_2); (dr = 1.2:1, >99% ee for the major diastereomer, >99% ee for the minor diastereomer); HPLC conditions: Chiralcel AD-H column, Hexane/i-PrOH = 80:20, flow rate 1.0 mL/min, UV detection at 220 nm, retention

time: major diastereomer: 9.91 min (major), minor diastereomer: 12.73 min (major). ^1H NMR (CDCl_3 , 300 MHz) δ 1.09 (t, $J = 7.14$ Hz, 3H), 1.38 (t, $J = 7.11$ Hz, 3H), 3.16 (s, 3H), 3.99-4.17 (m, 4H), 4.18-4.23 (m, 1H), 4.43-4.46 (m, 2H), 4.62 (s, 1H), 6.72-6.80 (m, 1H), 6.96 (t, $J = 7.53$ Hz, 1H), 7.20-7.32 (m, 2H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 13.8, 14.7, 26.2, 39.2, 44.5, 53.4, 61.0, 66.5, 75.0, 107.7, 122.1, 124.5, 127.2, 128.8, 144.5, 170.2, 175.6, 181.4, 194.1; HRMS (ESI) Calcd. for $\text{C}_{19}\text{H}_{21}\text{NNaO}_6$ $[\text{M}+\text{Na}]^+$: 382.1267; Found: 382.1270.

(3S)-3-[(1S)-2-(4-chlorophenyl)-1-(2-ethoxy-4-oxo-4,5-dihydrofuran-3-yl)-2-oxoethyl]-1-methyl-2,3-dihydro-1H-indol-2-one(5f)



68% yield, $[\alpha]_D^{20} = +67.5$ (c 1.2, CH_2Cl_2); (dr = 1.5:1, >99% ee for the major diastereomer, 96% ee for the minor diastereomer); HPLC conditions: Chiralcel AD-H column, EtOH/Hexane = 30:70, flow rate 1.0 mL/min, UV

detection at 254 nm, retention time: major diastereomer: 20.34 min (major) and 21.69 min (minor), minor diastereomer: 24.79 min (major) and 30.05 min (minor). ^1H NMR (CDCl_3 , 300 MHz) δ 1.36 (t, $J = 7.11$ Hz, 3H), 3.15 (s, 3H), 4.15-4.19 (m, 2H), 4.38-4.43 (m, 2H), 4.71 (d, $J = 16.02$ Hz, 1H), 4.95 (d, $J = 3.87$ Hz, 1H), 6.79 (t, $J = 8.55$ Hz, 1H), 7.01 (d, $J = 7.44$ Hz, 1H), 7.33-7.37 (m, 4H), 7.97 (d, $J = 8.52$ Hz, 2H); ^{13}C NMR (CDCl_3 , 75 Hz) δ 14.6, 26.1, 42.3, 45.1, 66.8, 75.1, 89.0, 107.8, 122.2, 124.7, 126.8, 128.0, 128.4, 128.8, 134.0, 139.7, 144.1, 175.5, 181.7, 193.8, 195.6; HRMS (ESI) Calcd. for $\text{C}_{23}\text{H}_{20}\text{ClNNaO}_5$ $[\text{M}+\text{Na}]^+$: 448.0928; Found: 448.0927.

6. References

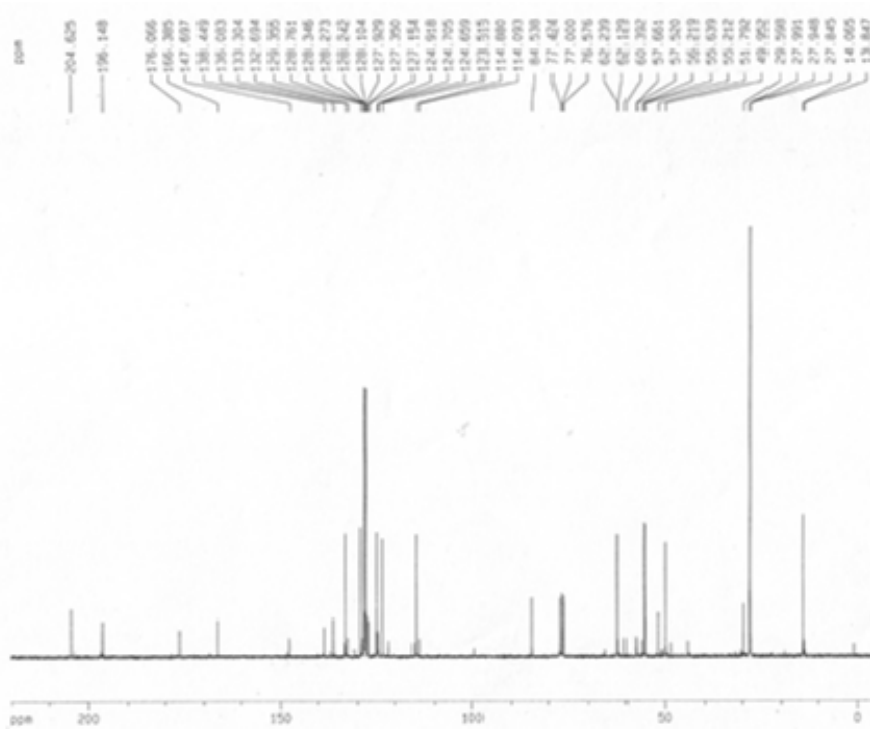
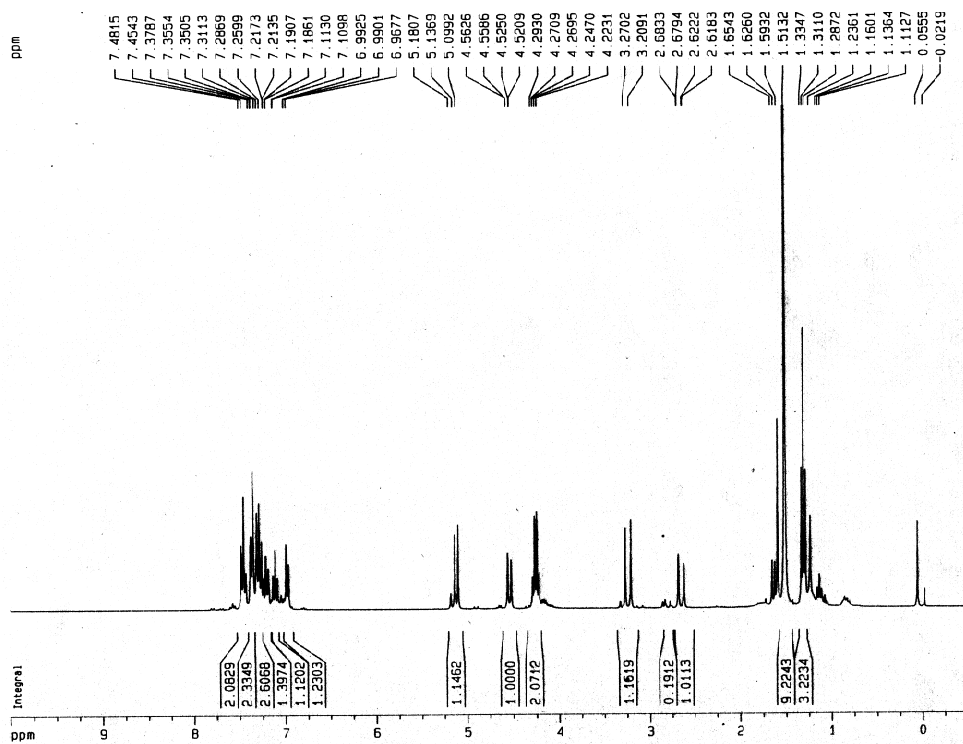
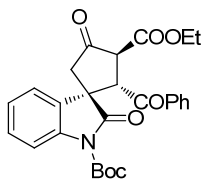
- 1 E. M. Beccalli and A. Marchesini, *Tetrahedron*, 1993, **49**, 4741.
- 2 B. Tan, N. R. Candeias and C. F. Barbas, *J. Am. Chem. Soc.*, 2012, **133**, 4672.
- 3 H. Y. Choi and D. Y. Chi, *Org. Lett.*, 2003, **5**, 411.

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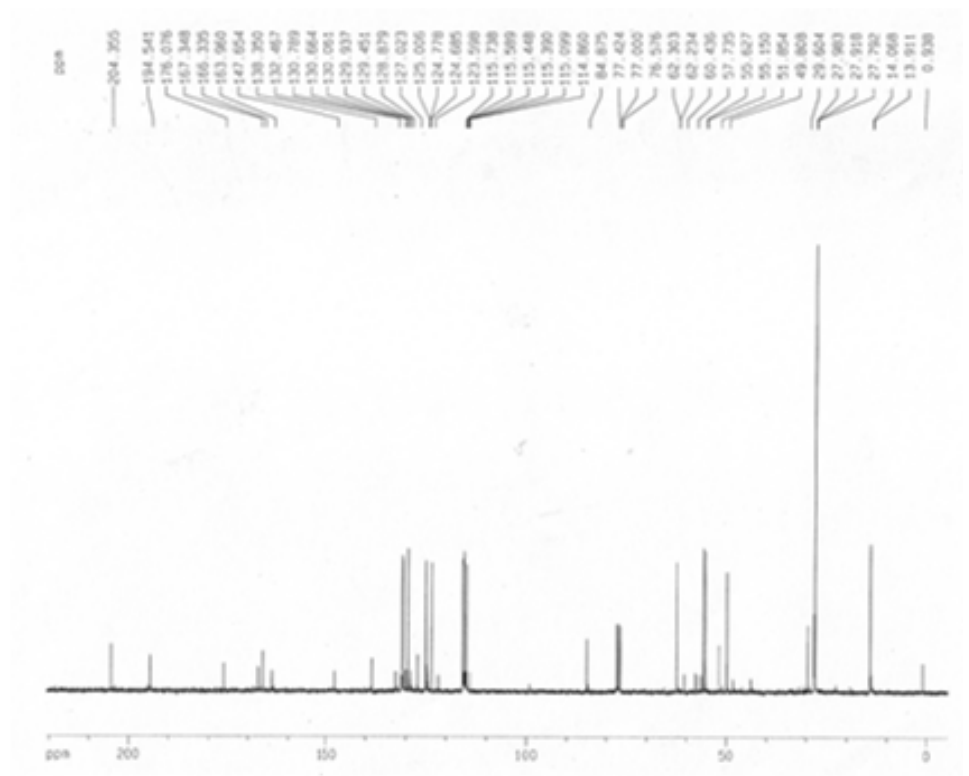
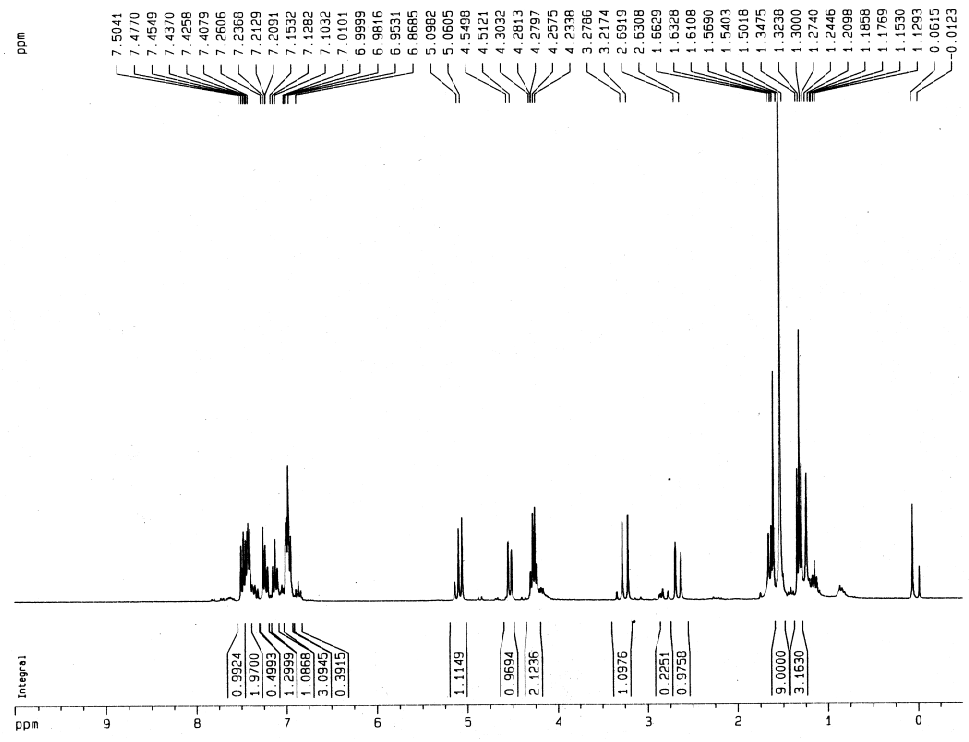
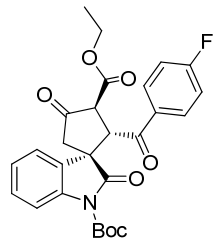
5 (a) B. Vakulya, A. Varga, S. Csámpai and T. Soós, *Org. Lett.*, 2005, **7**, 1967; (b) S. H. McCooey and S. J. Connon, *Angew. Chem. Int. Ed.*, 2005, **44**, 6367.

7. NMR Spectra

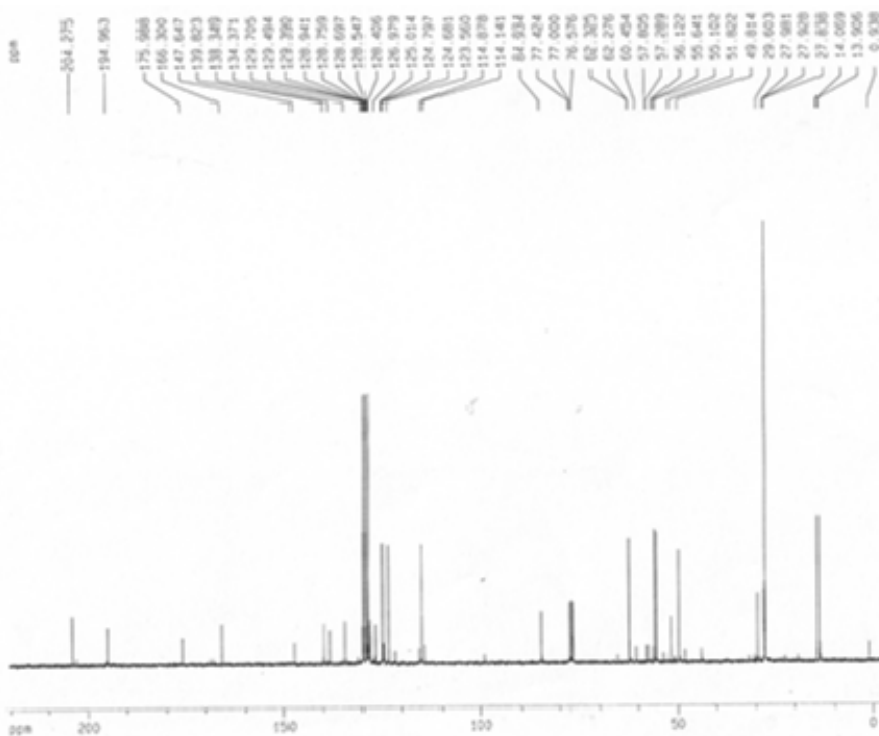
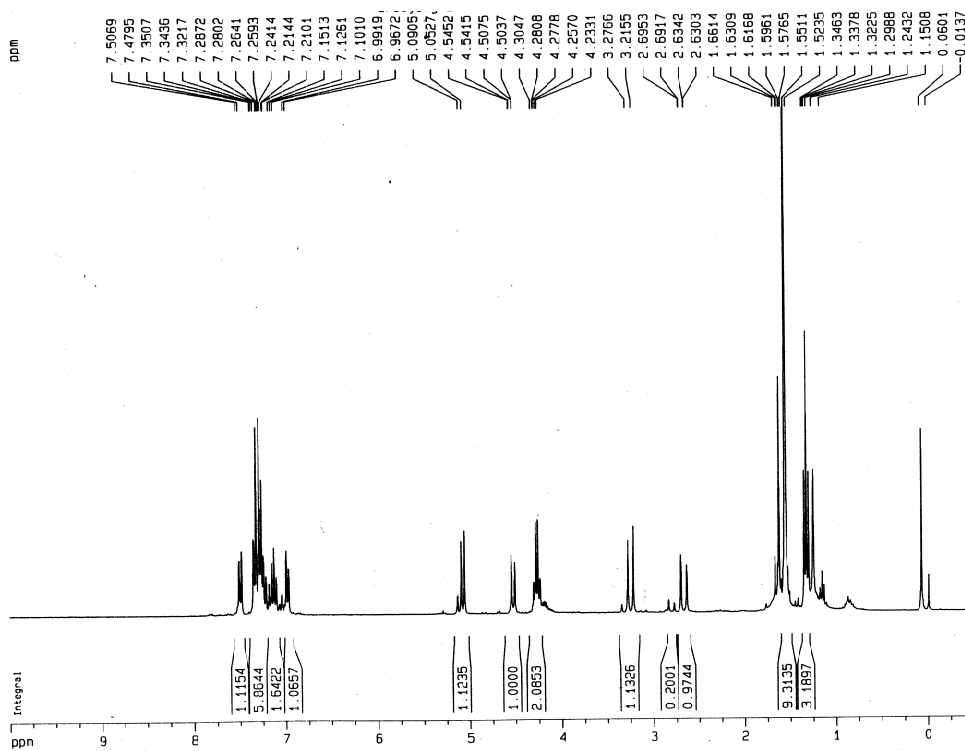
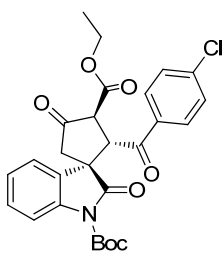
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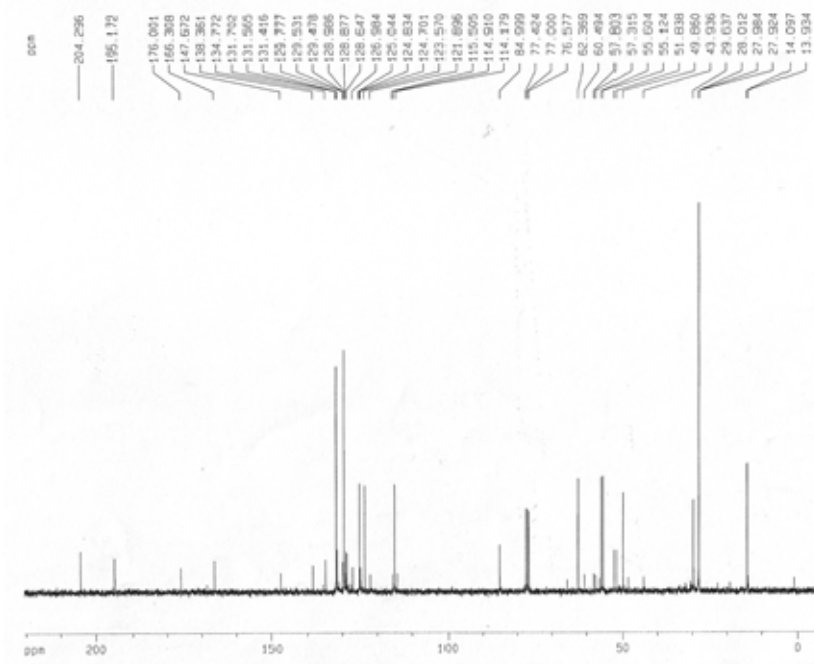
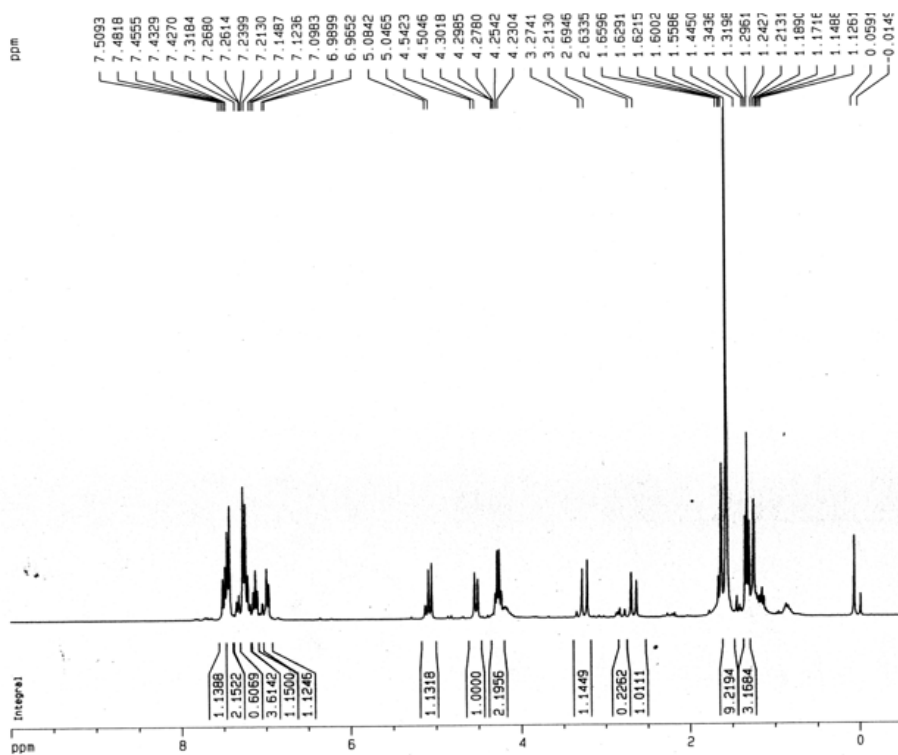
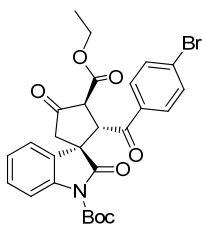
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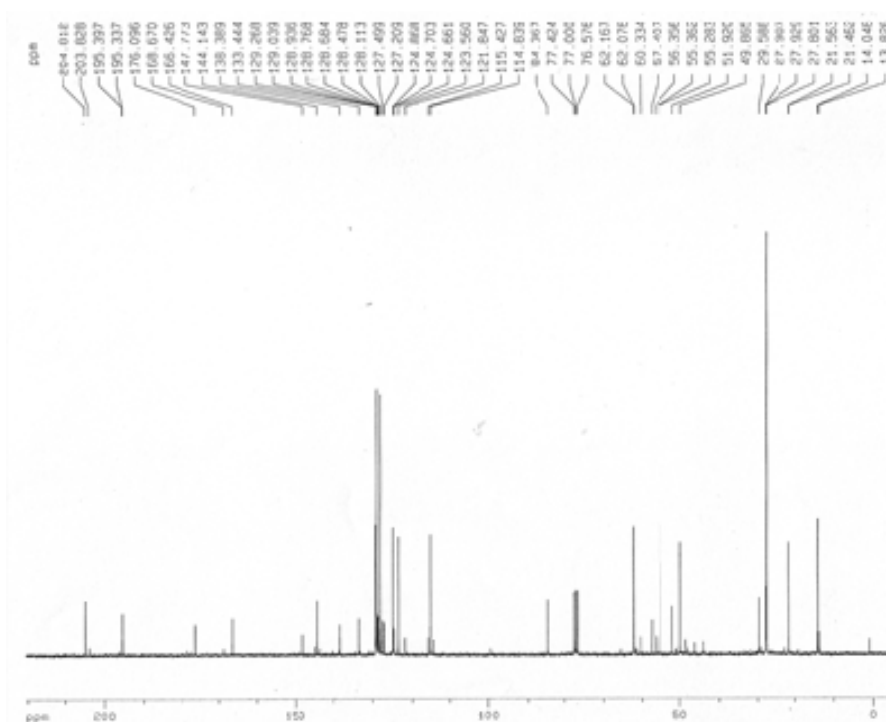
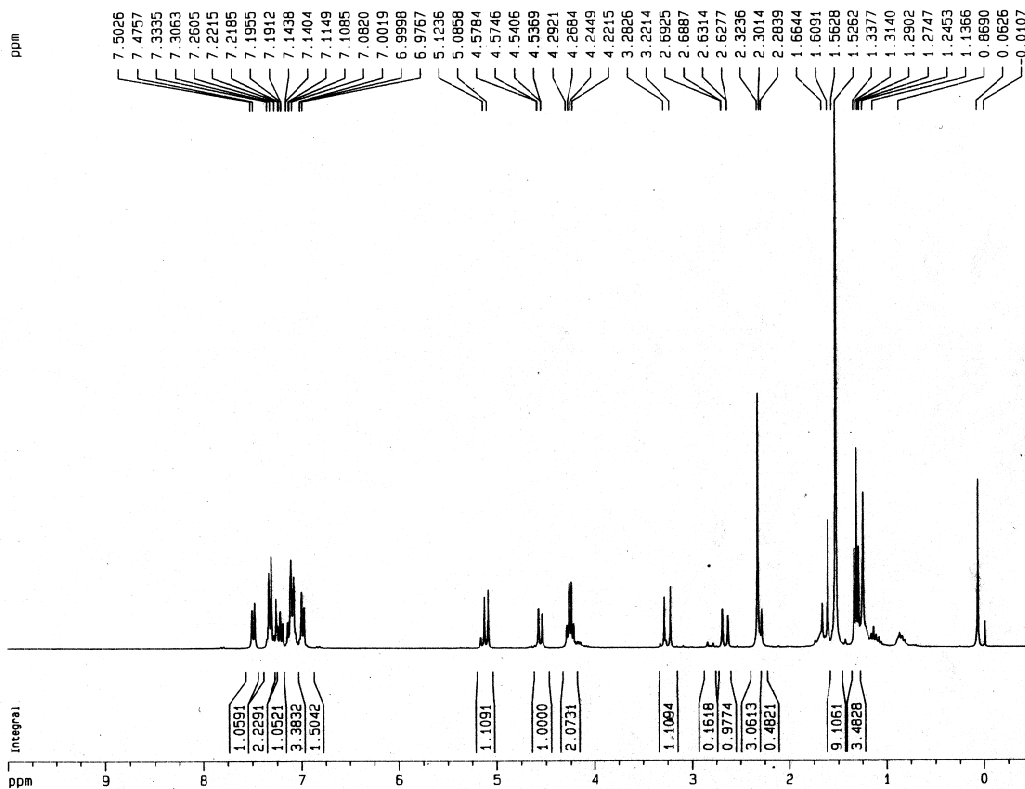
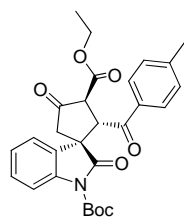
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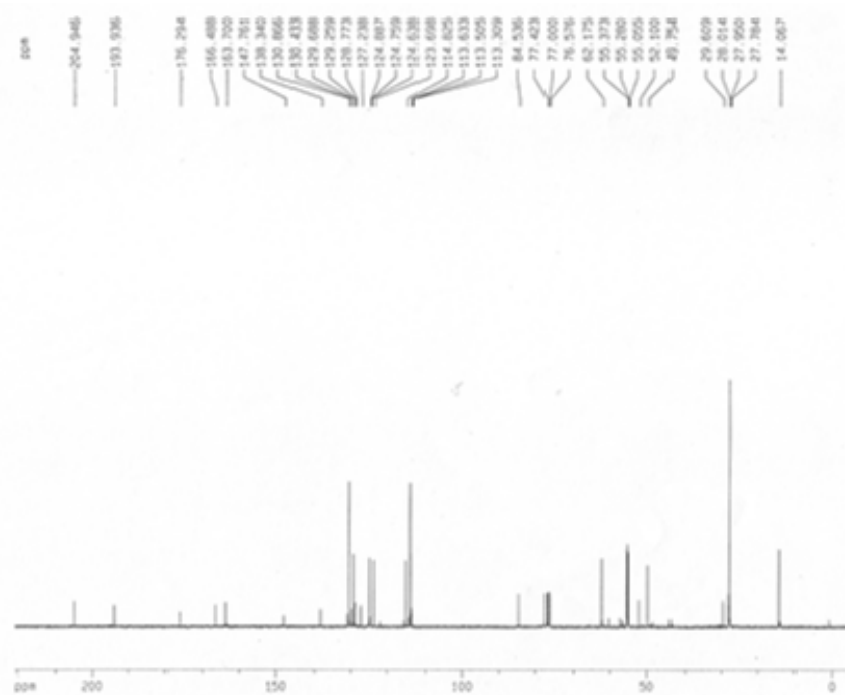
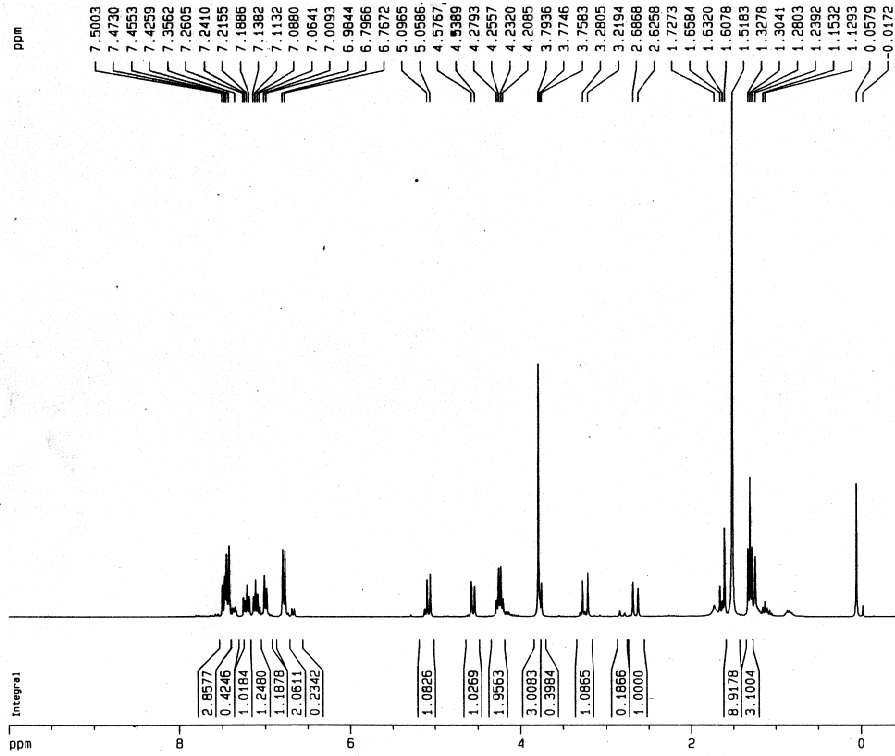
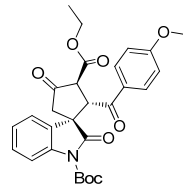
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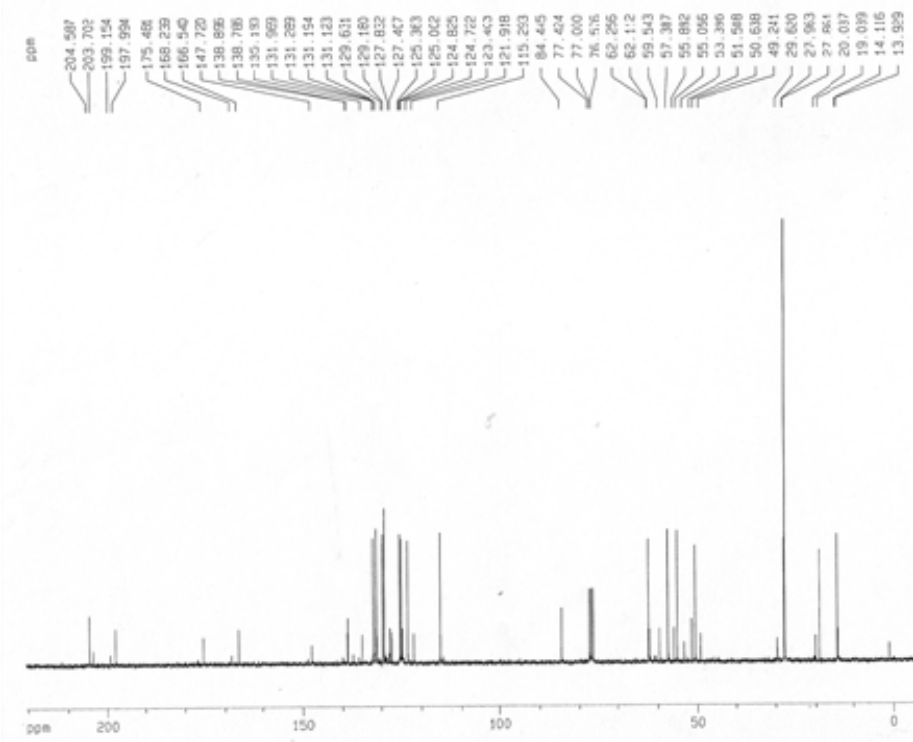
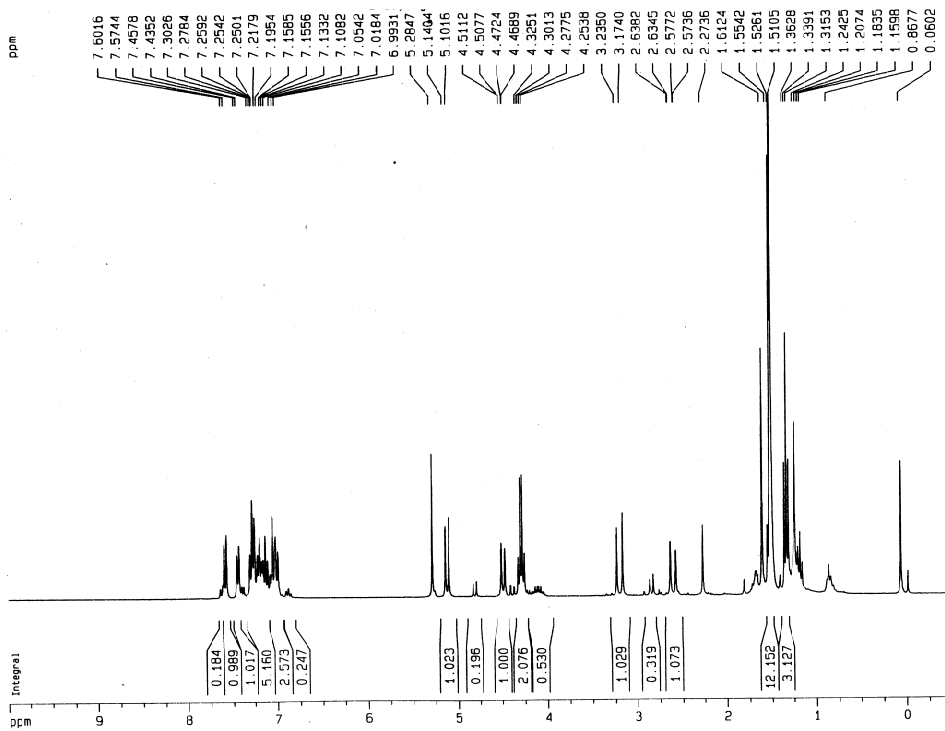
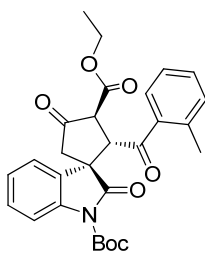
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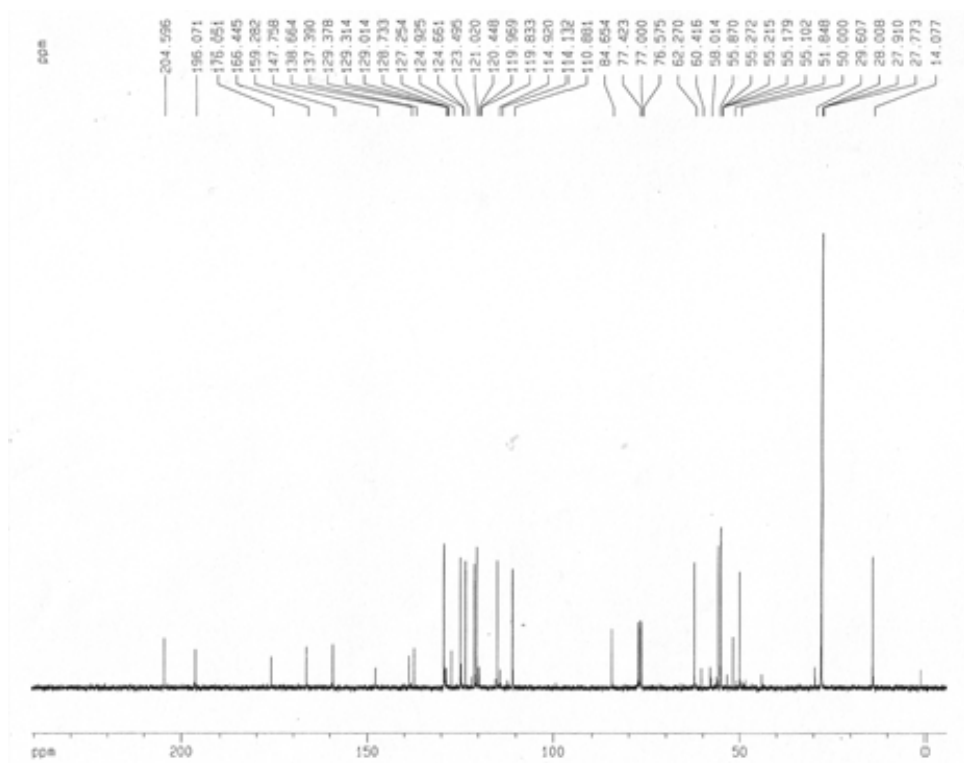
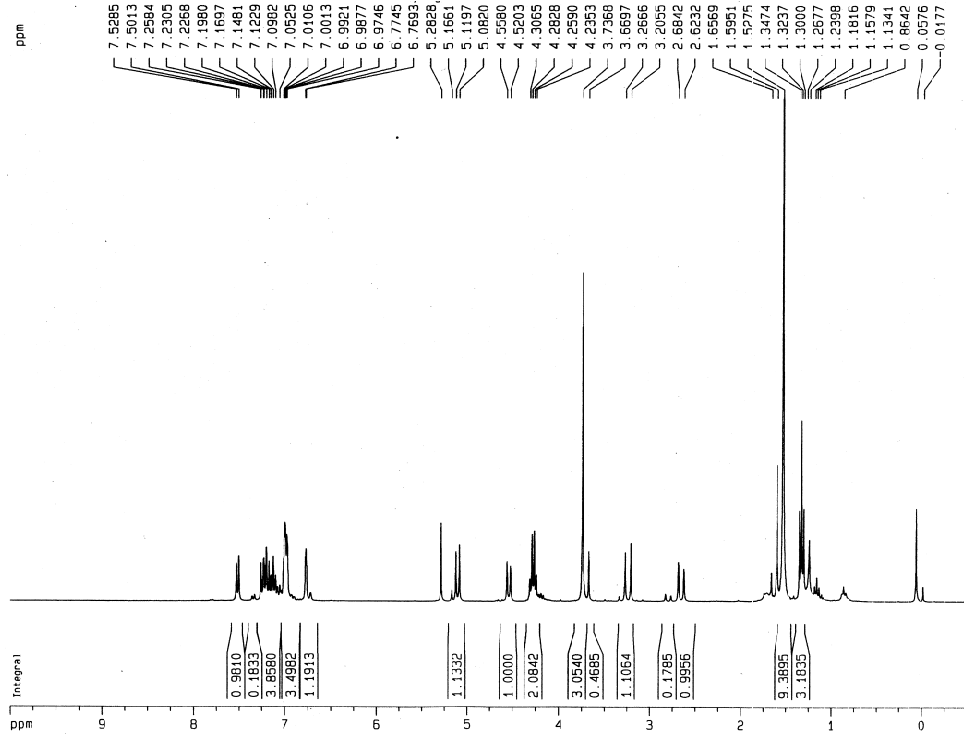
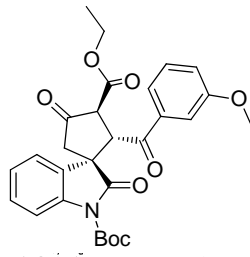
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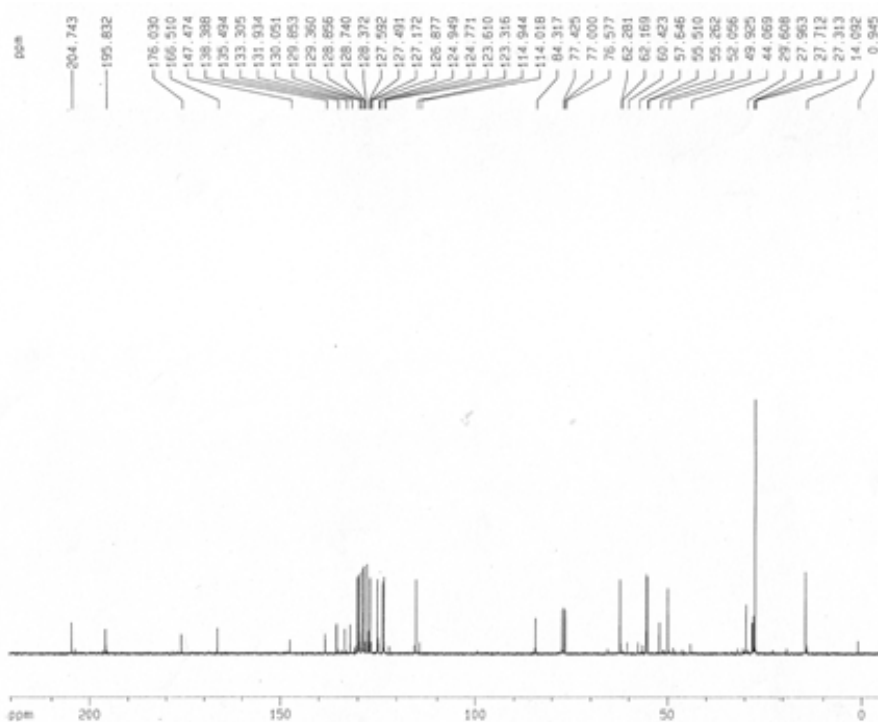
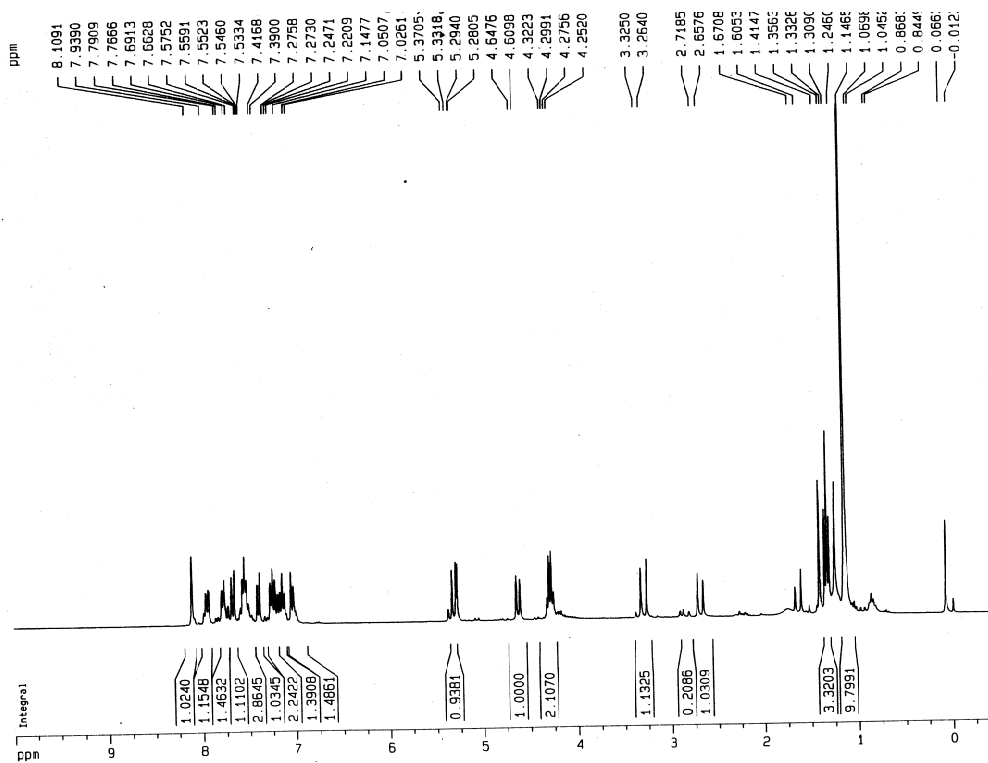
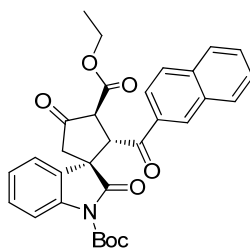
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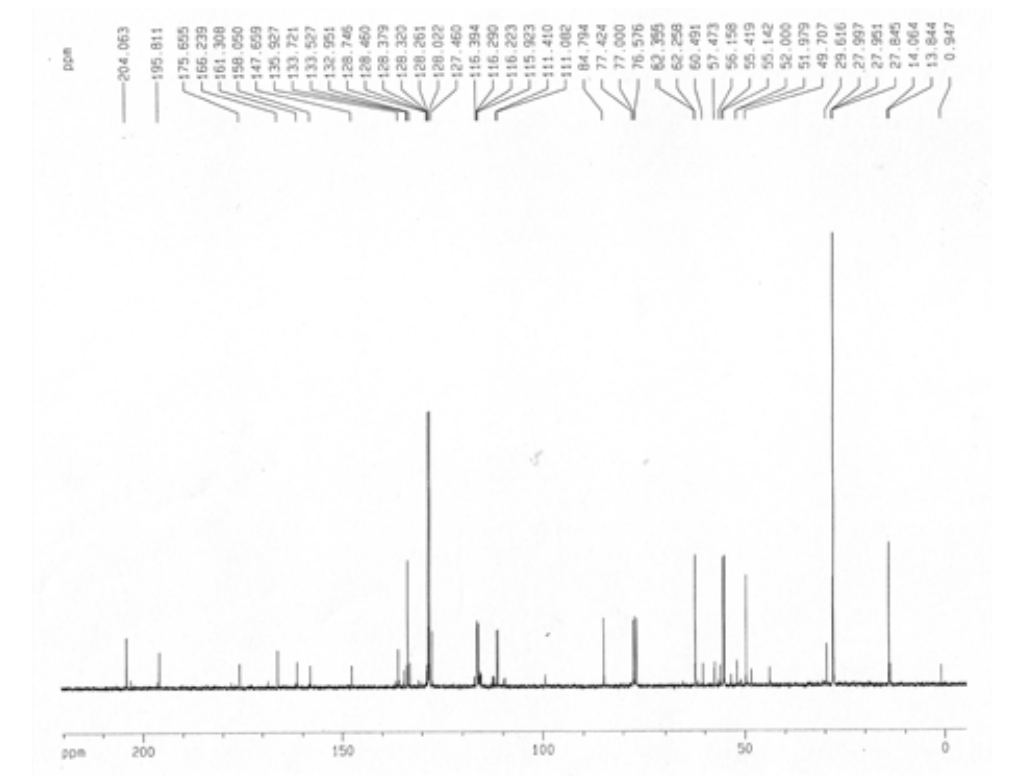
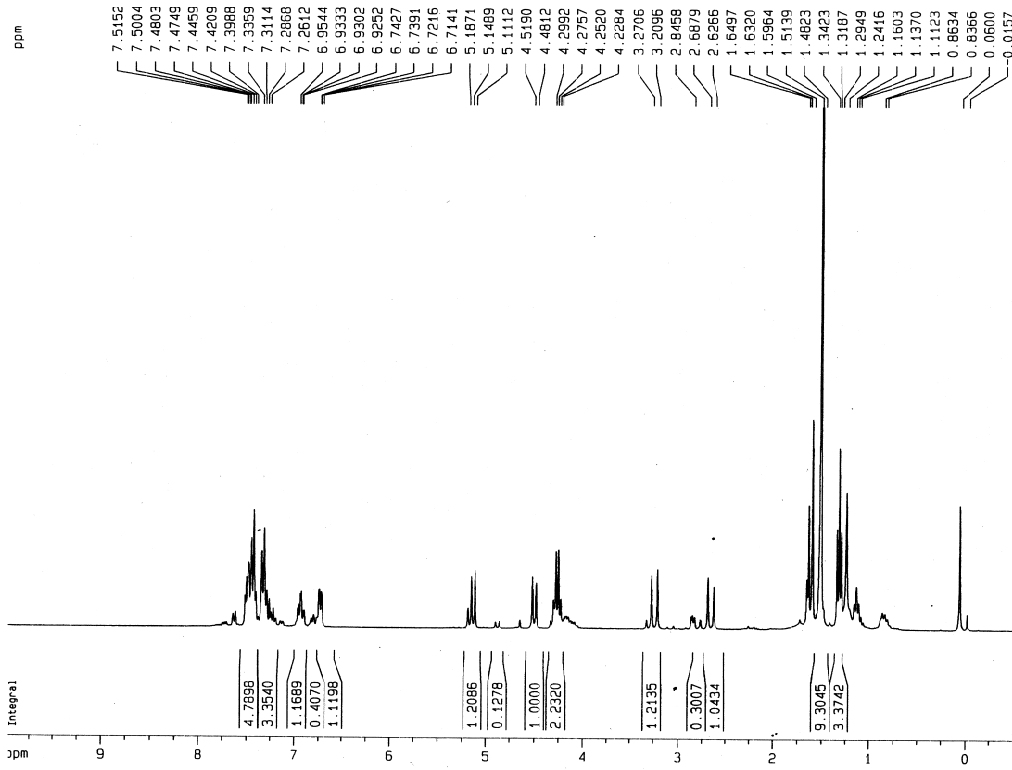
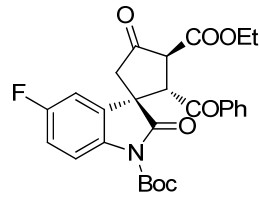
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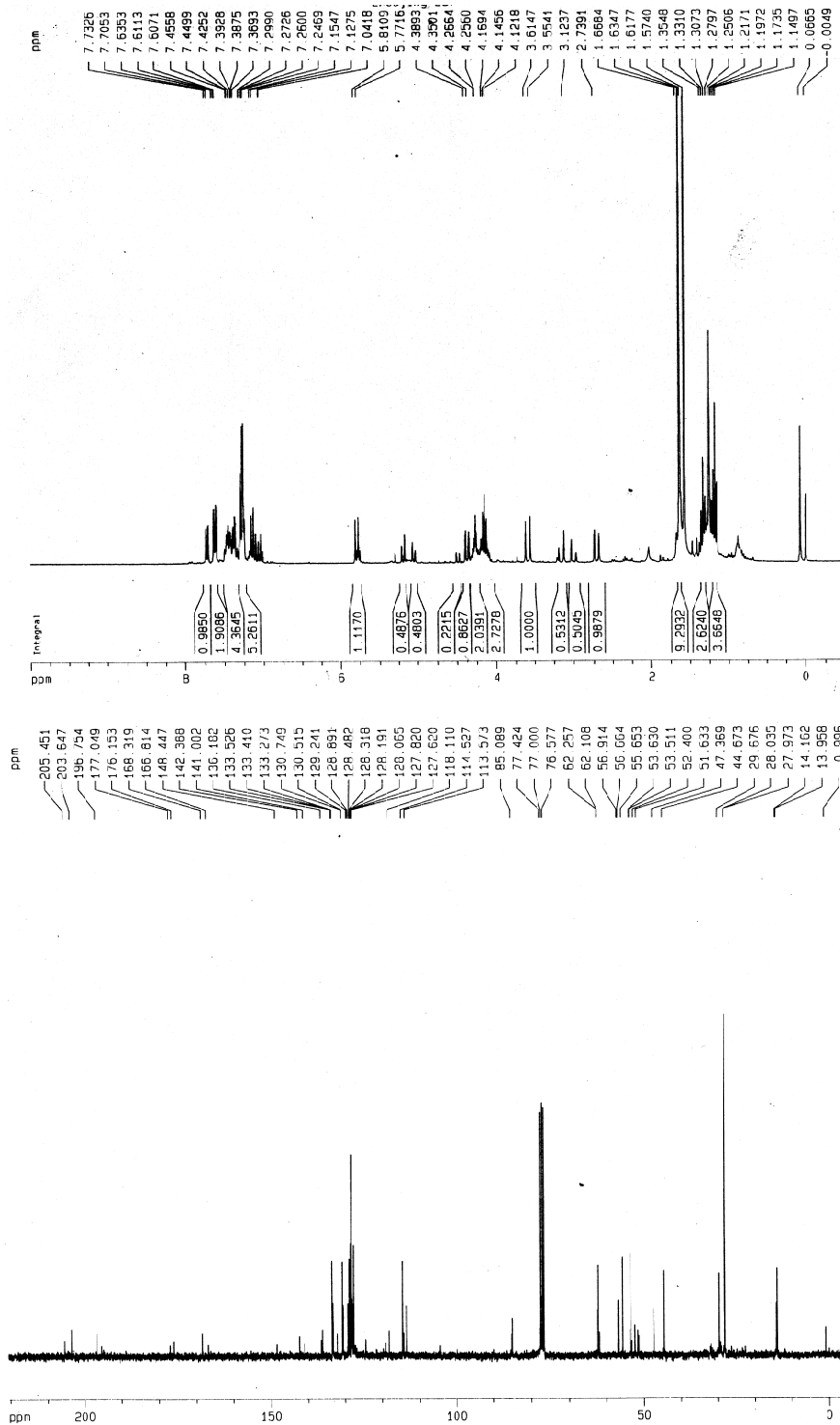
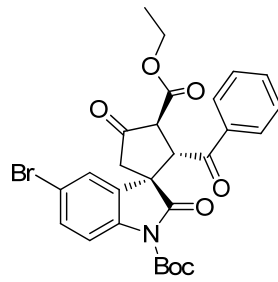
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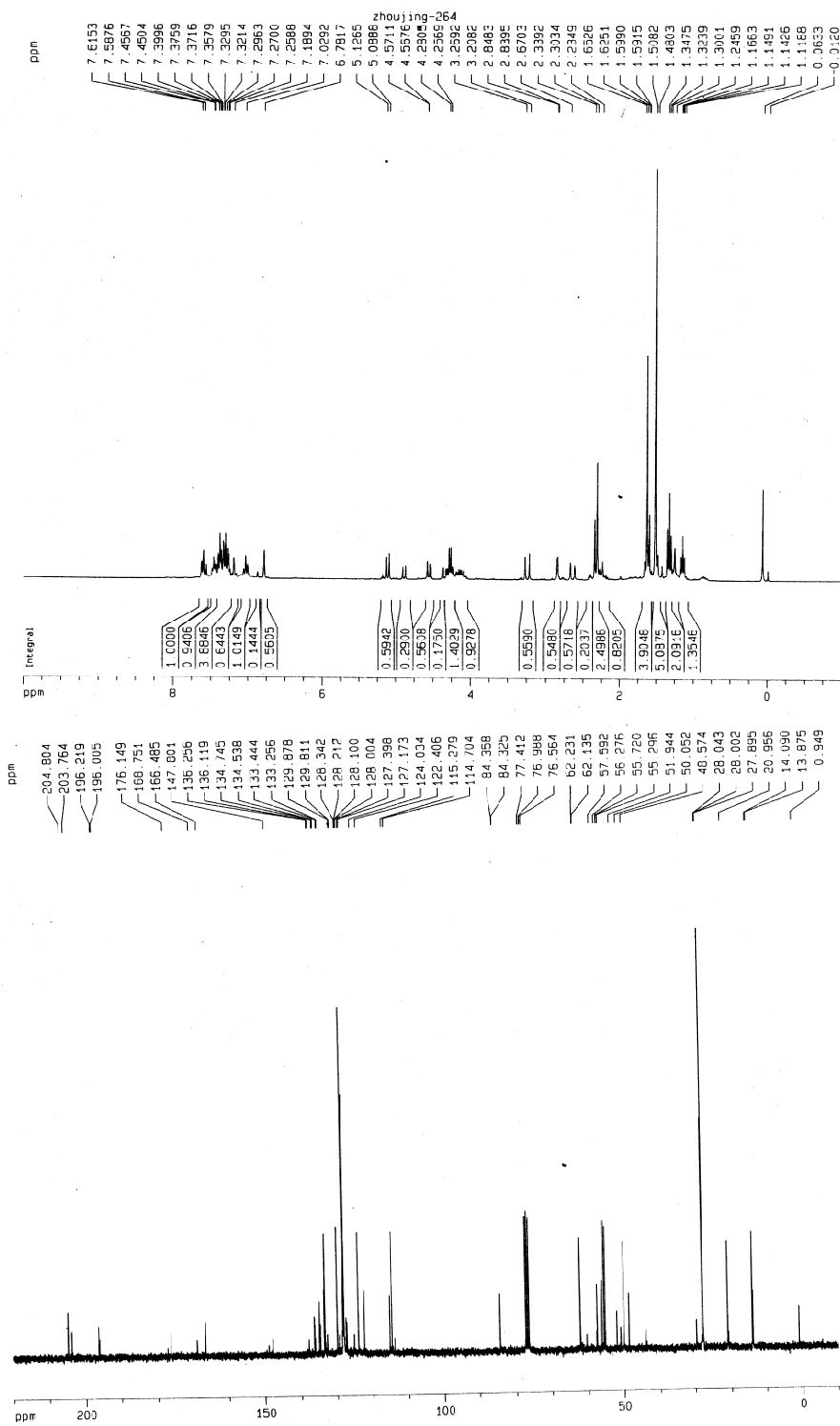
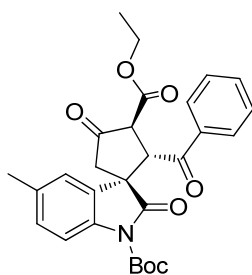
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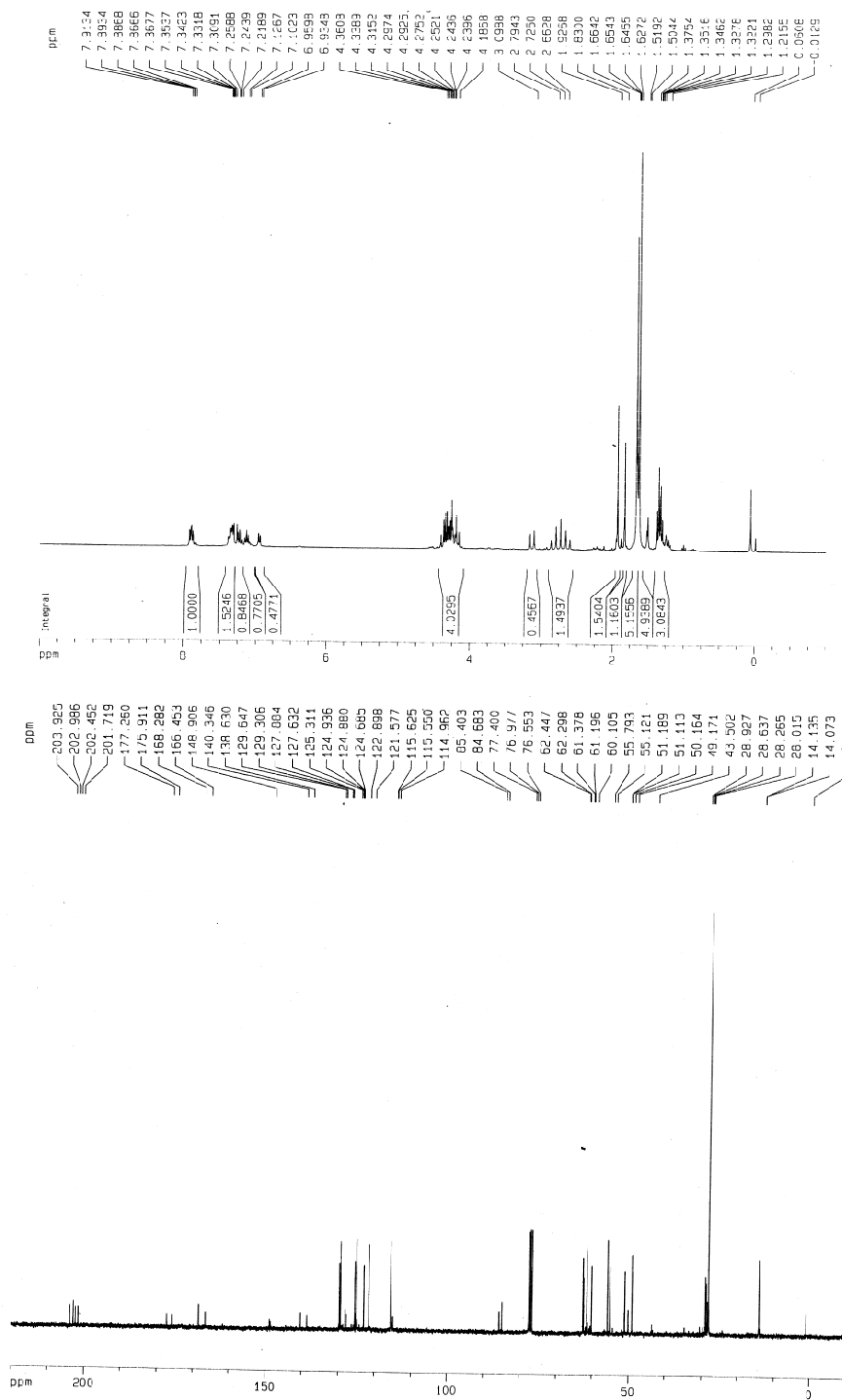
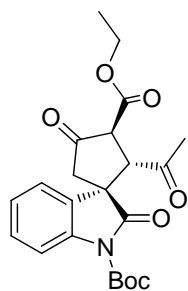
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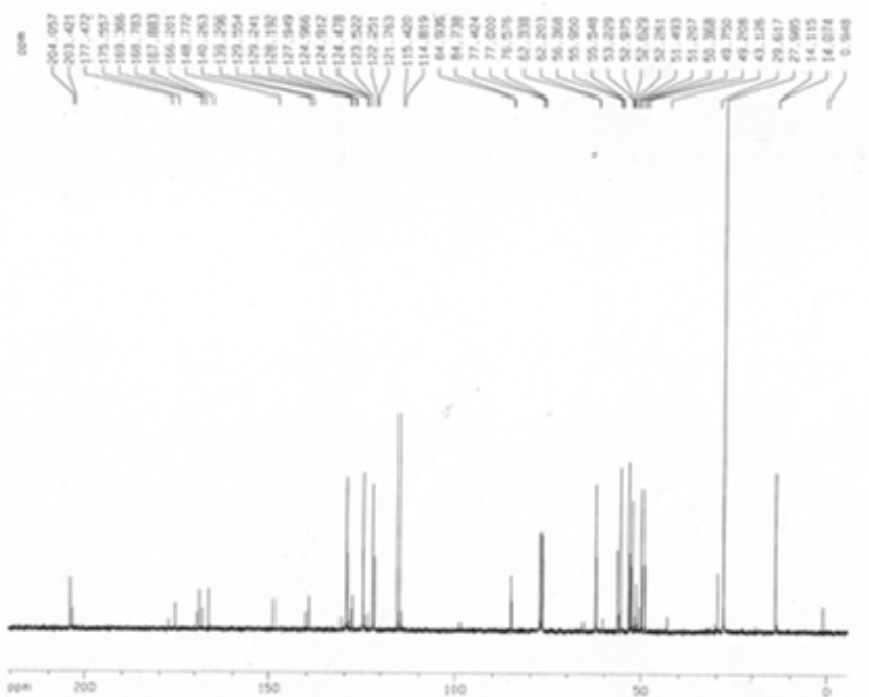
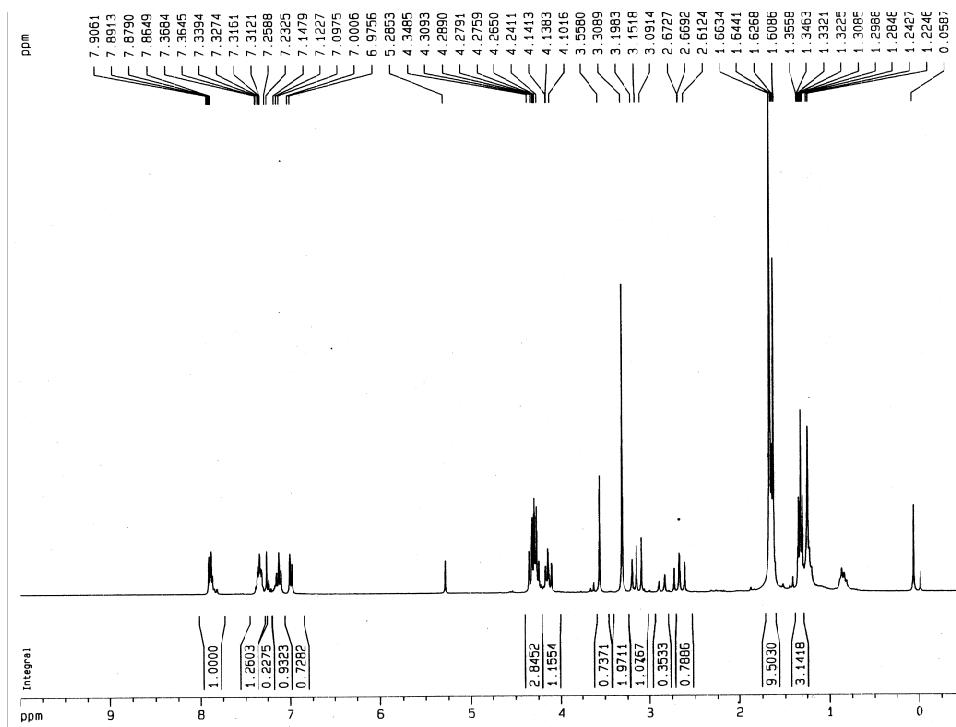
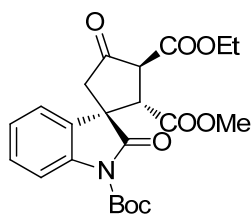
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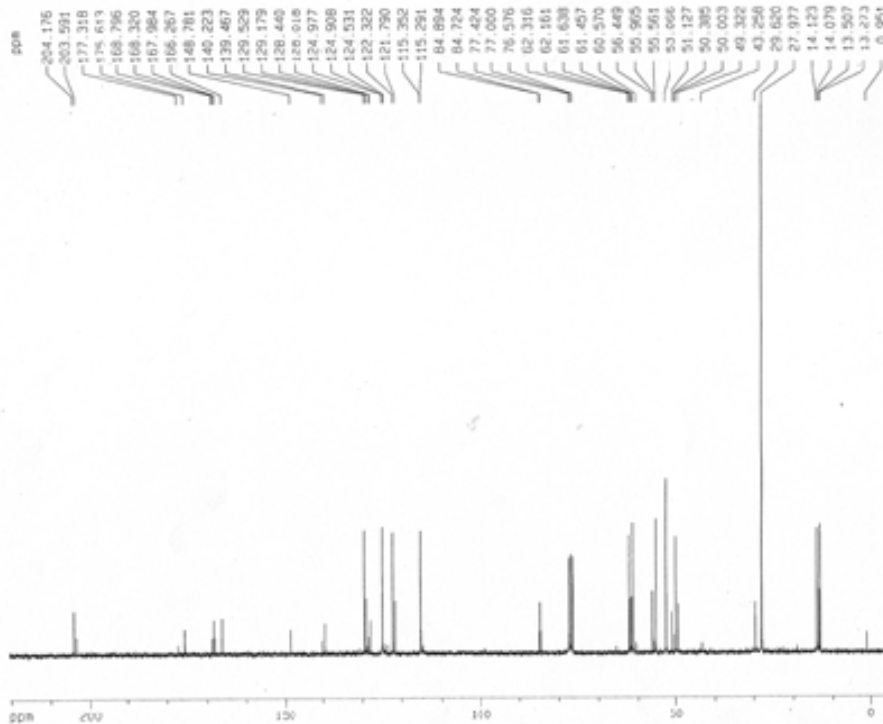
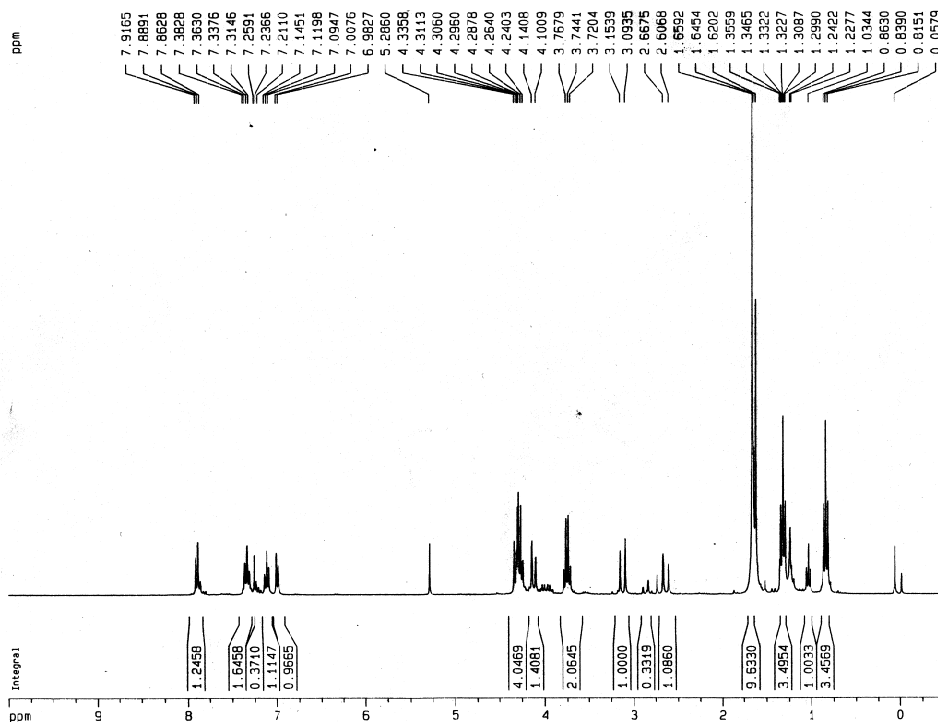
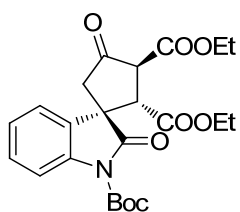
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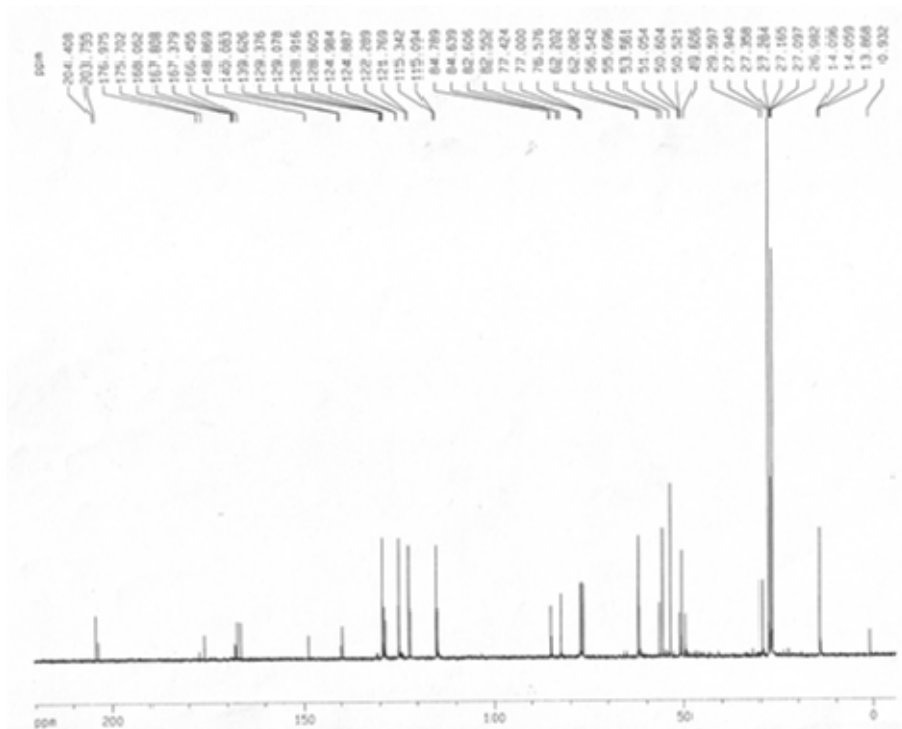
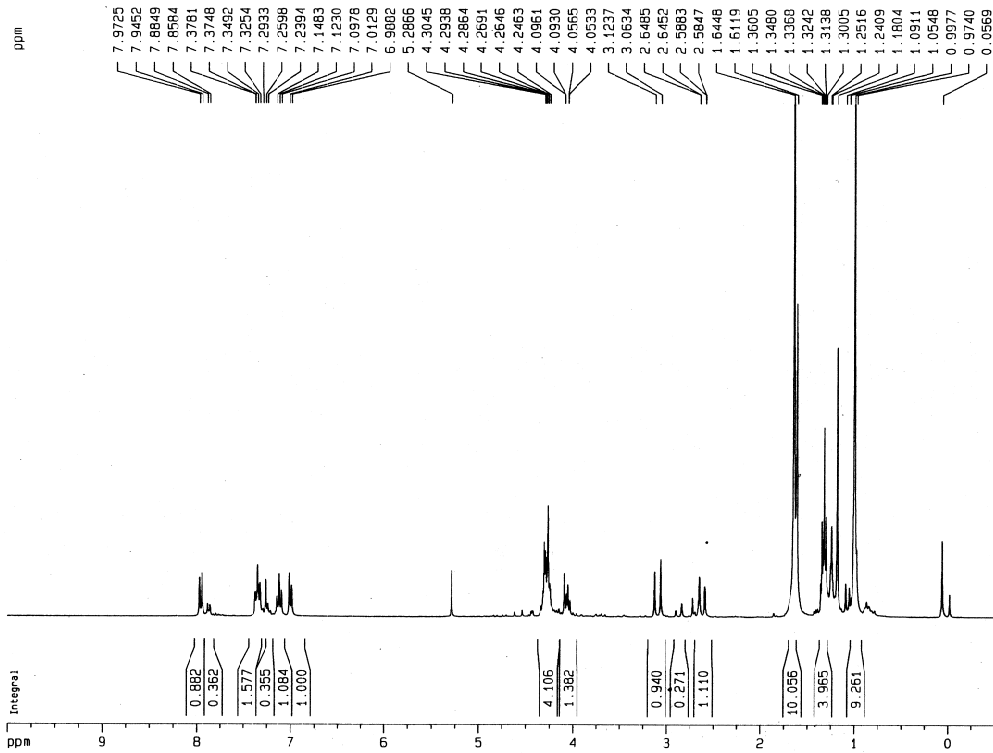
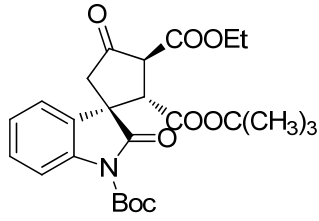
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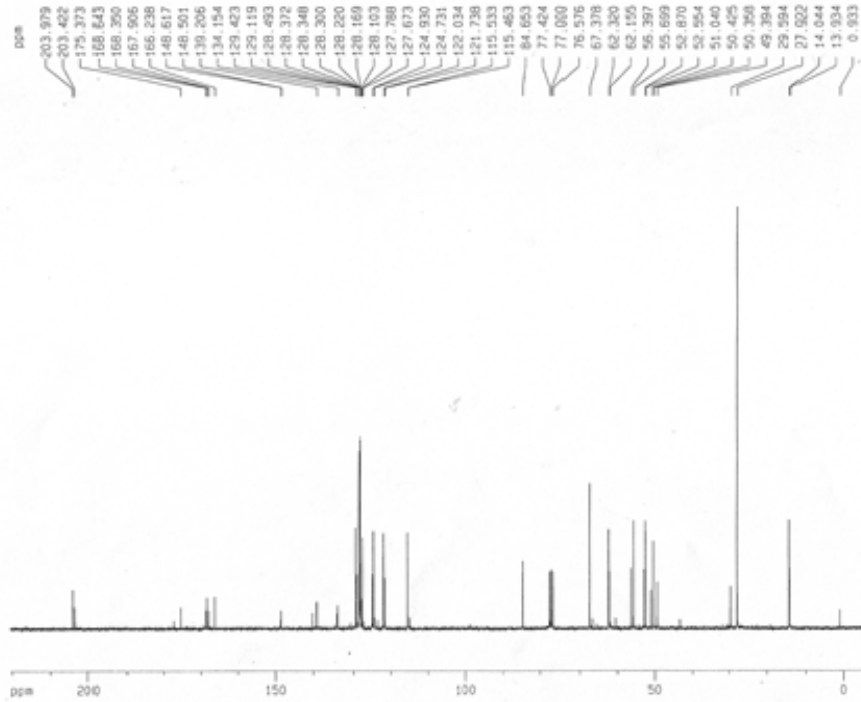
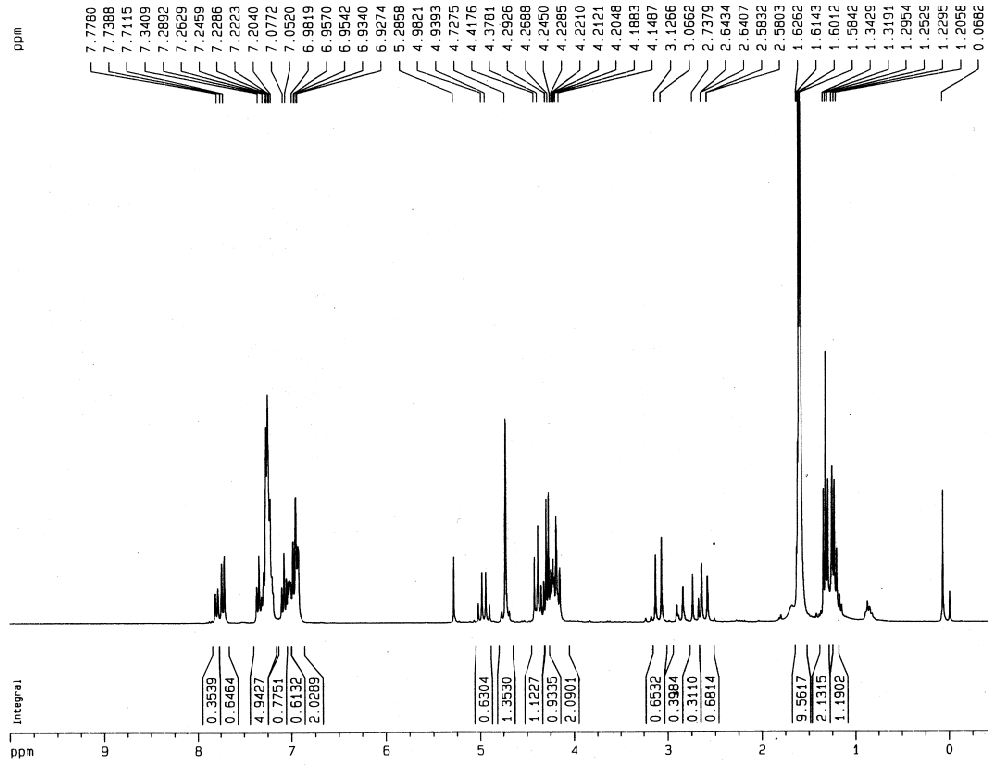
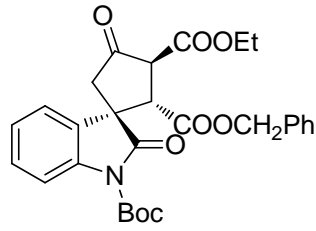
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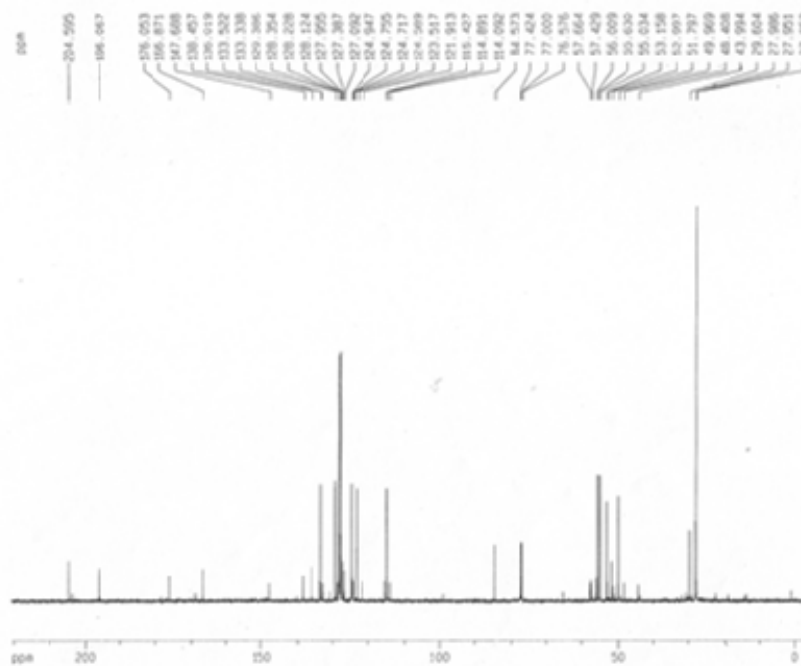
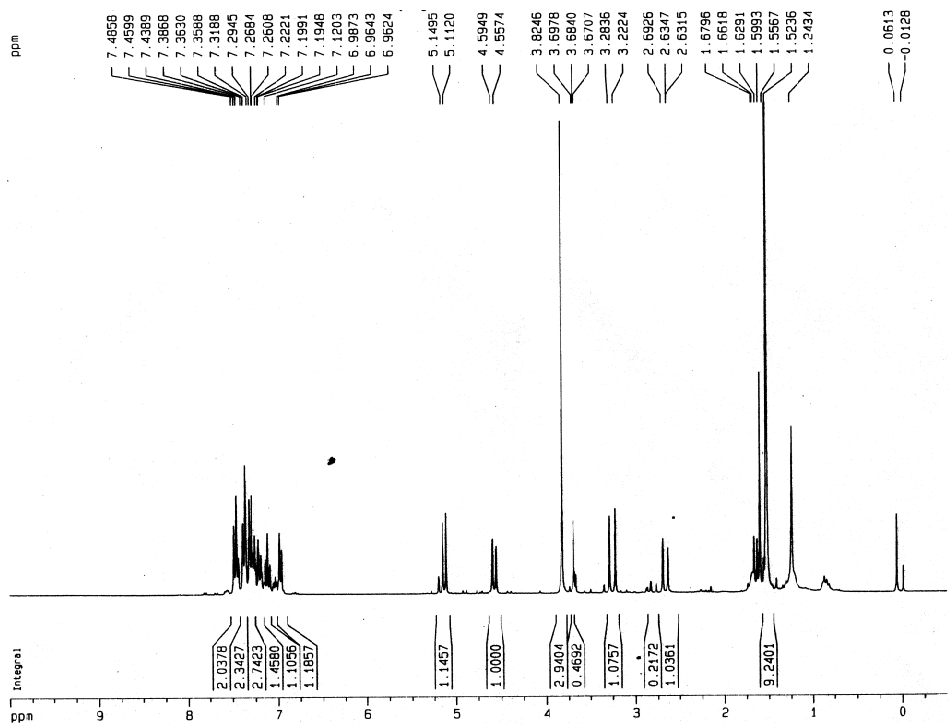
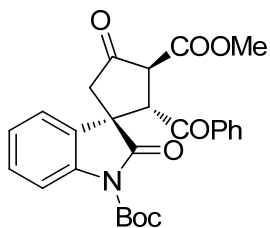
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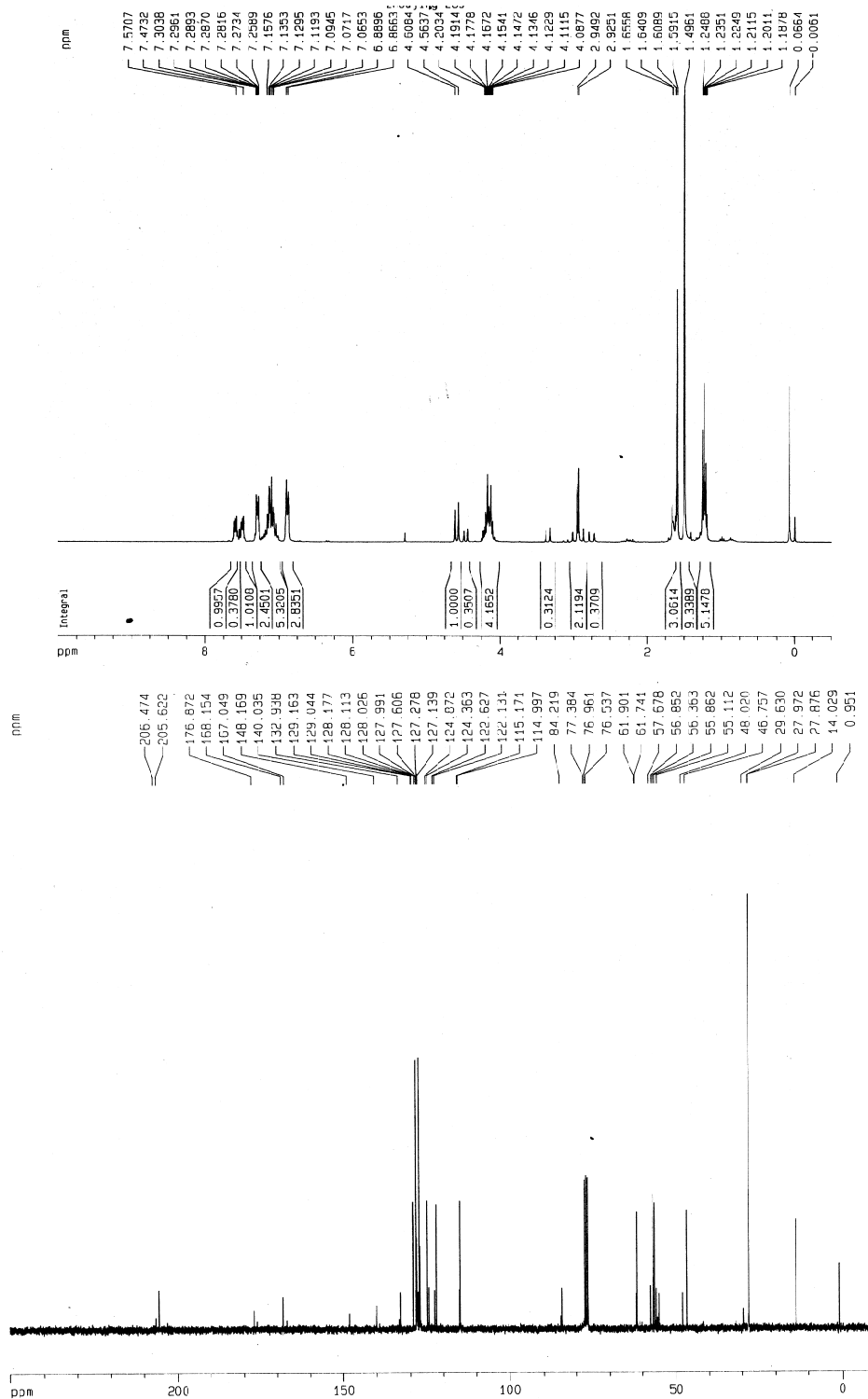
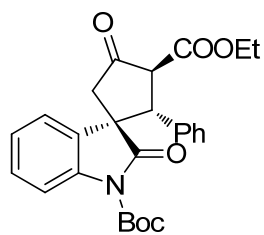
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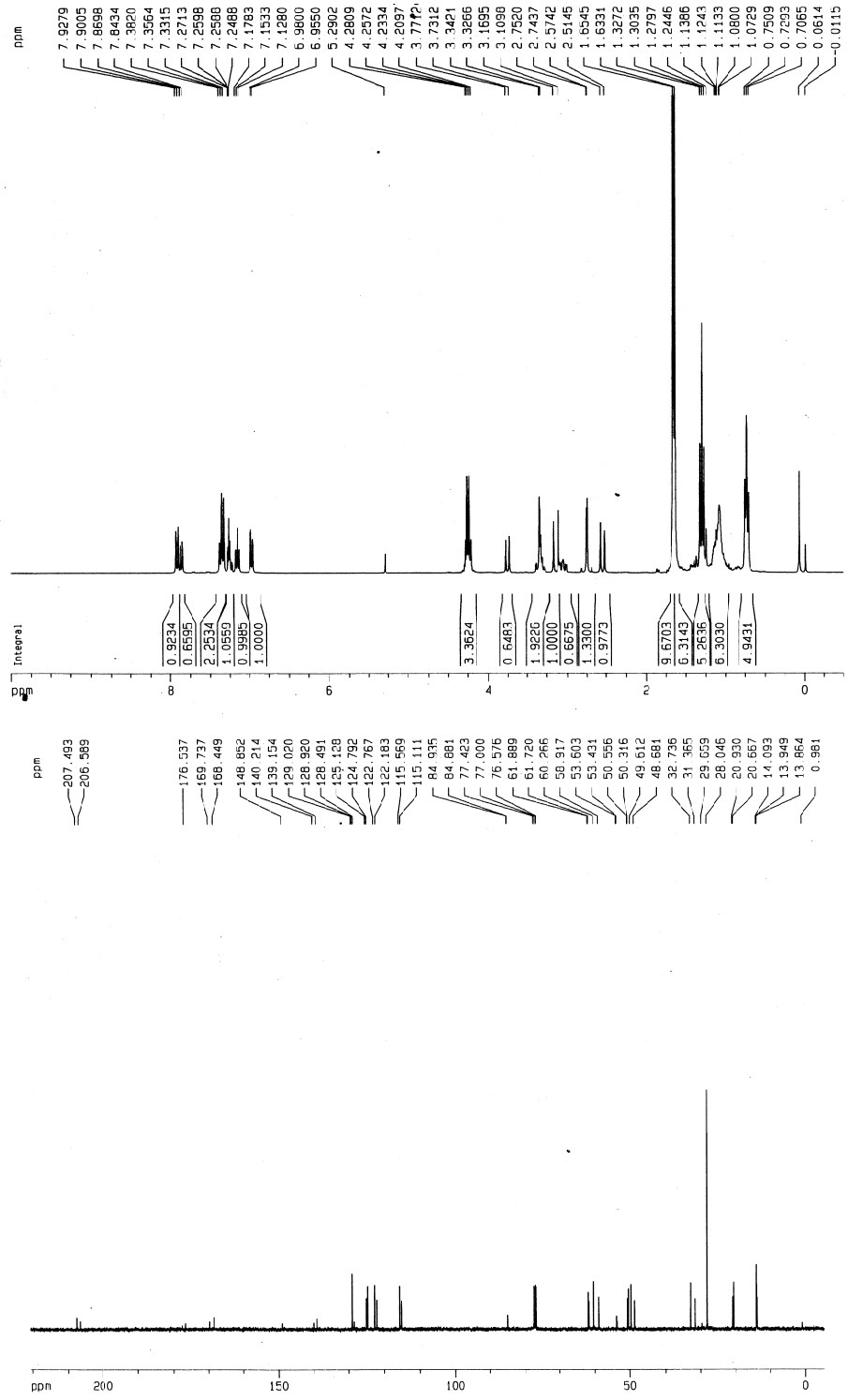
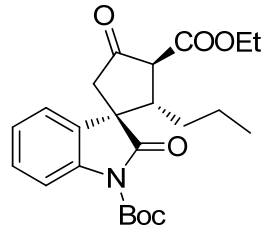
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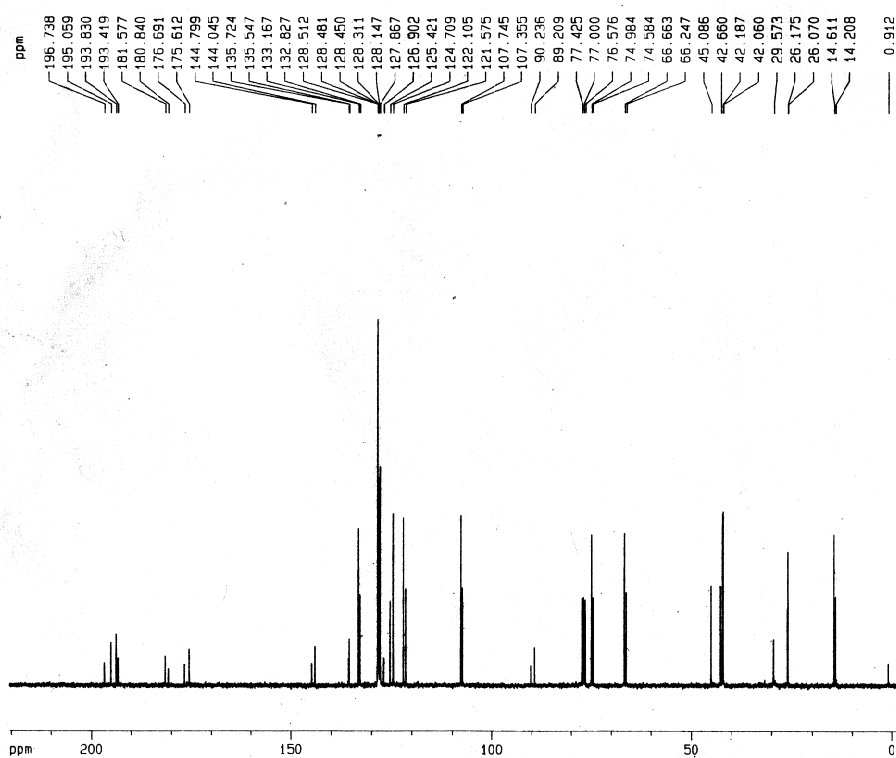
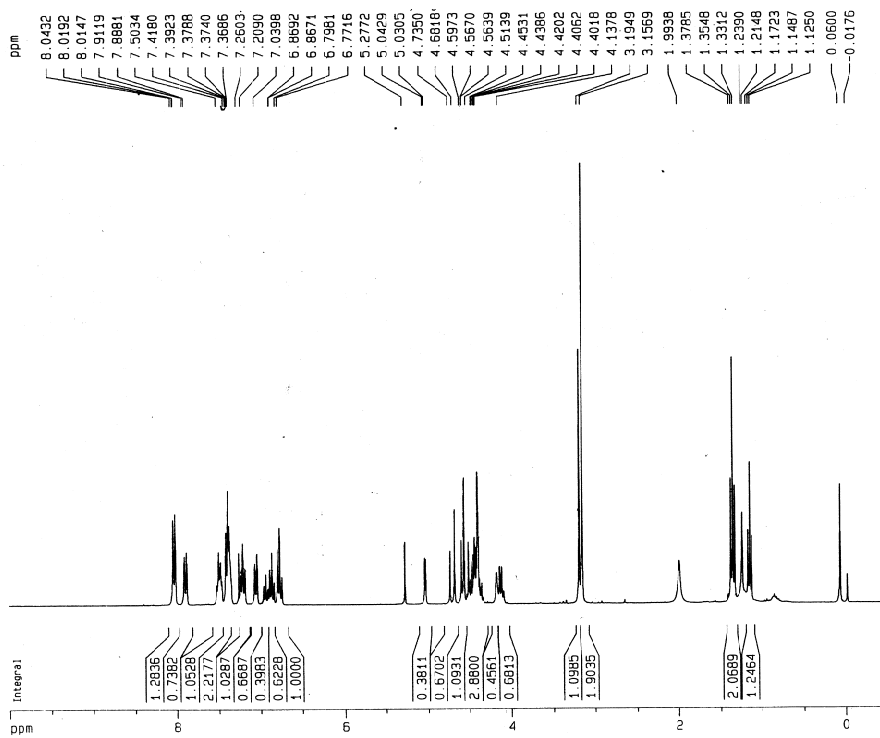
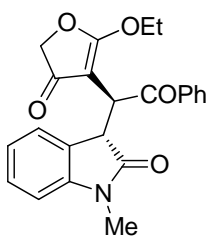
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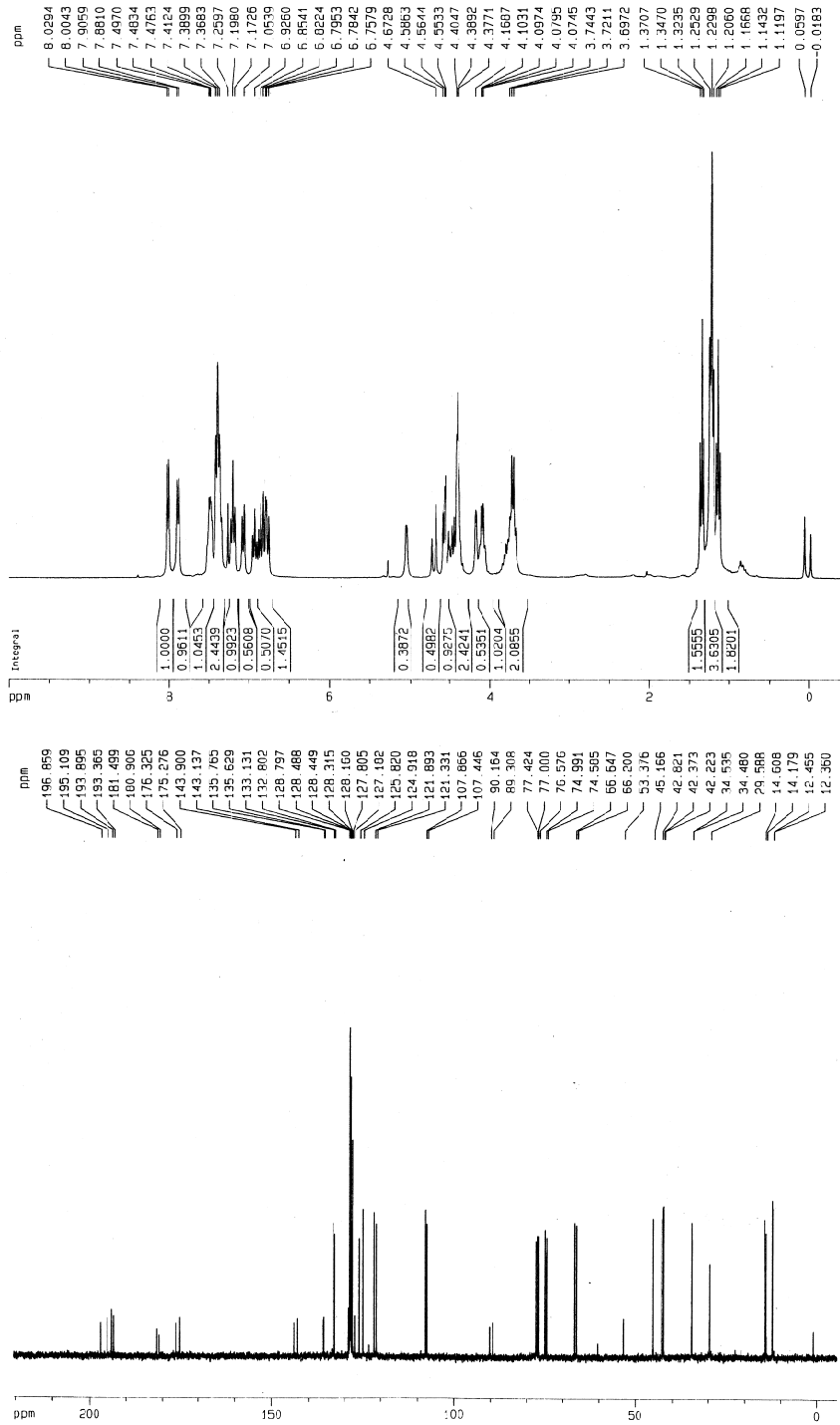
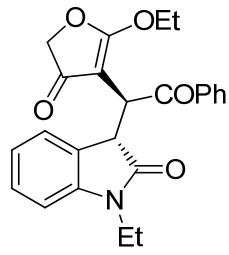
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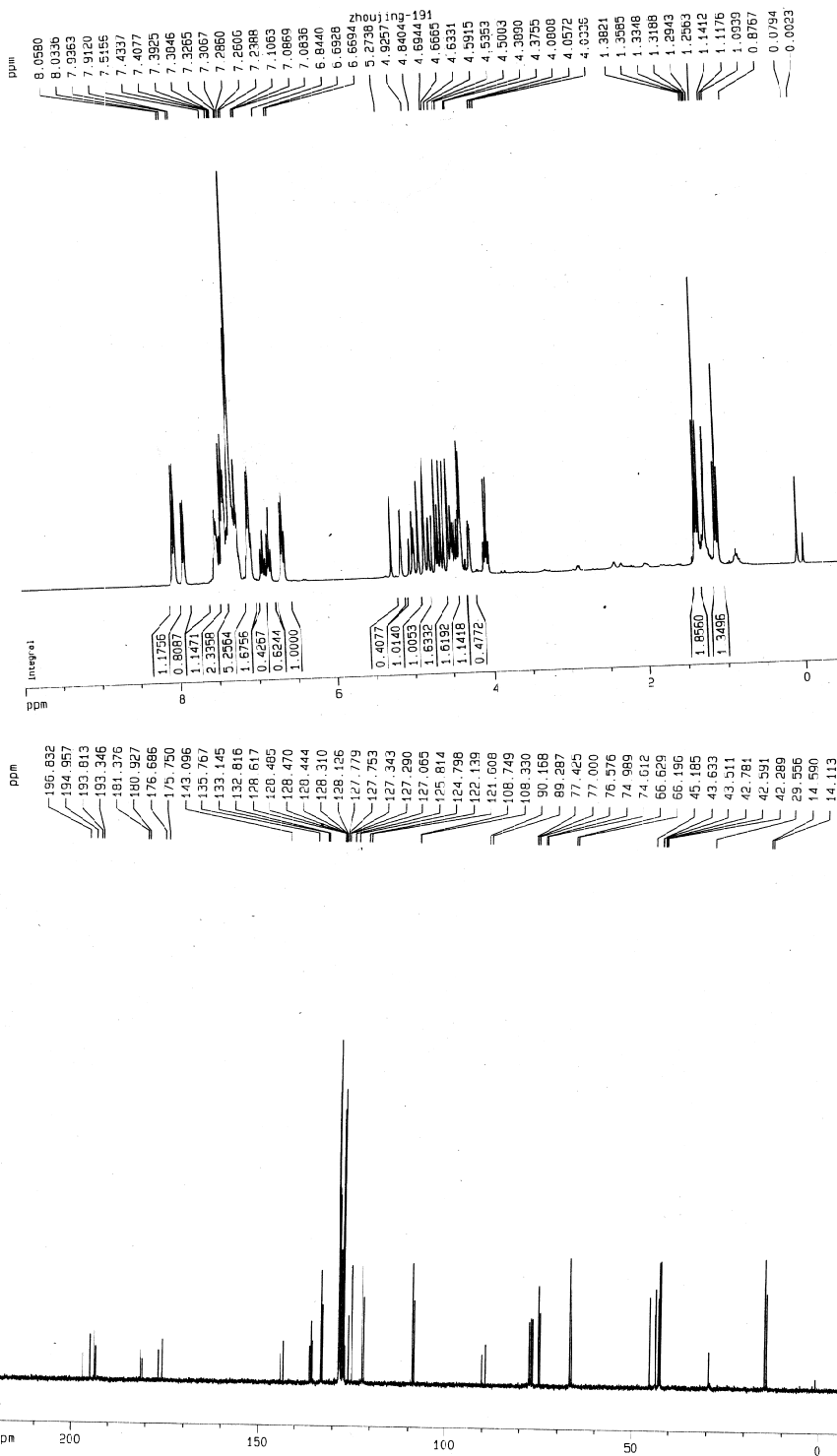
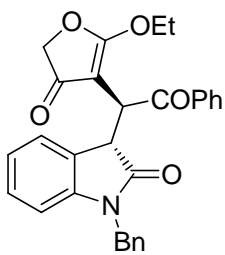
¹H and ¹³C NMR of 5a



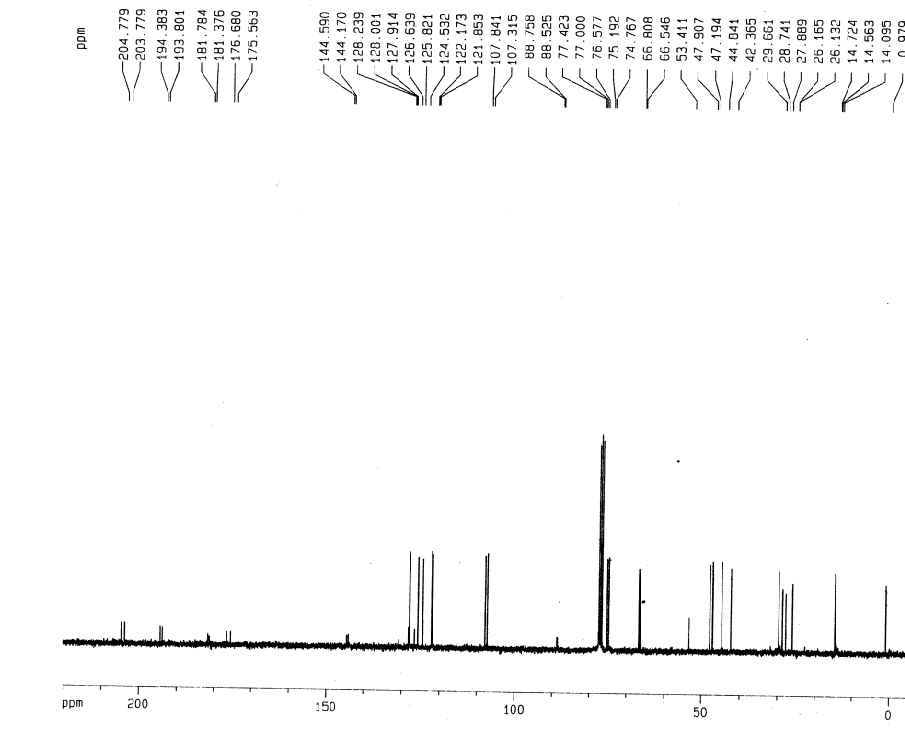
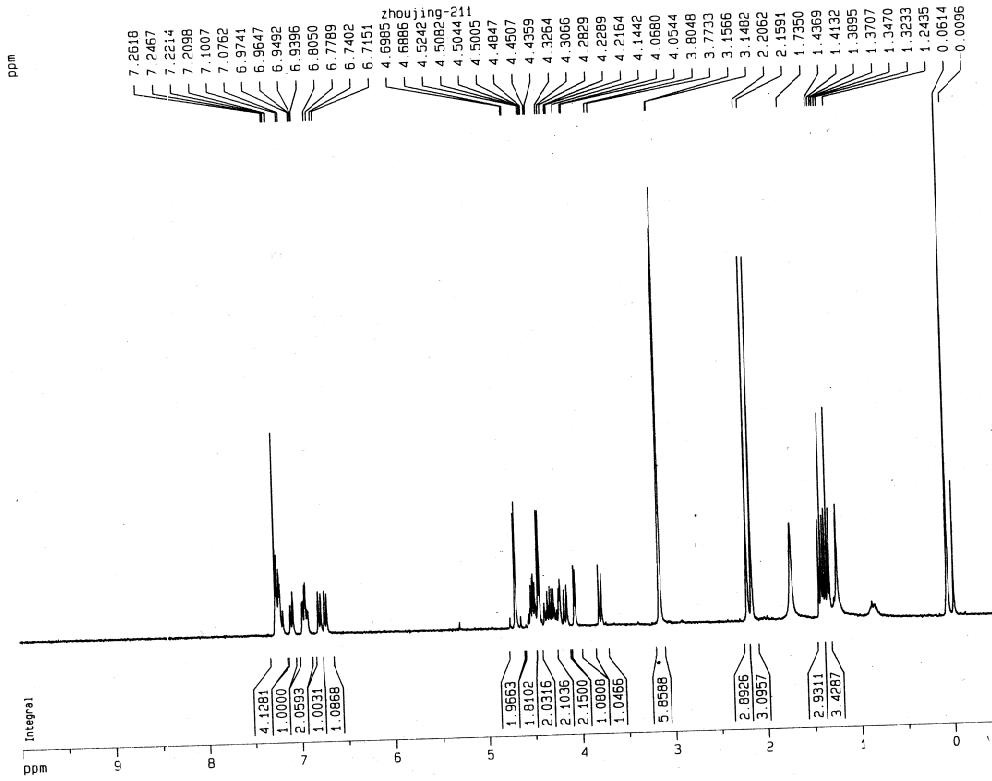
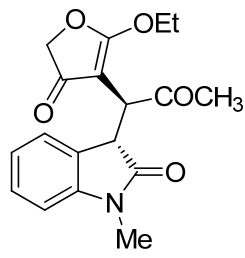
¹H and ¹³C NMR of 5b



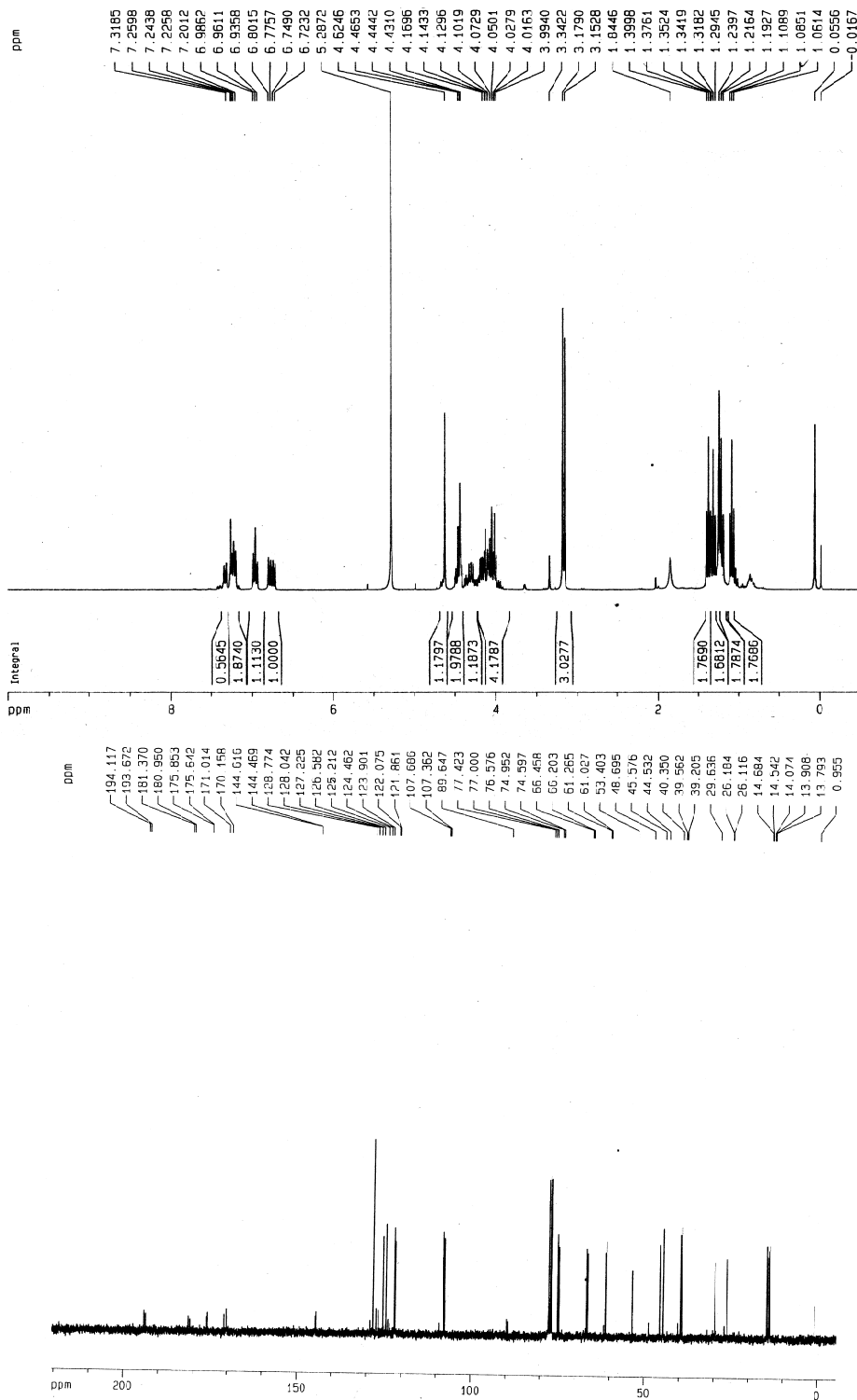
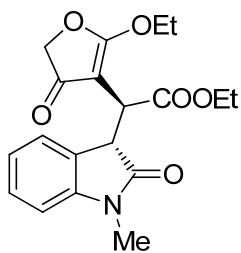
¹H and ¹³C NMR of 5c



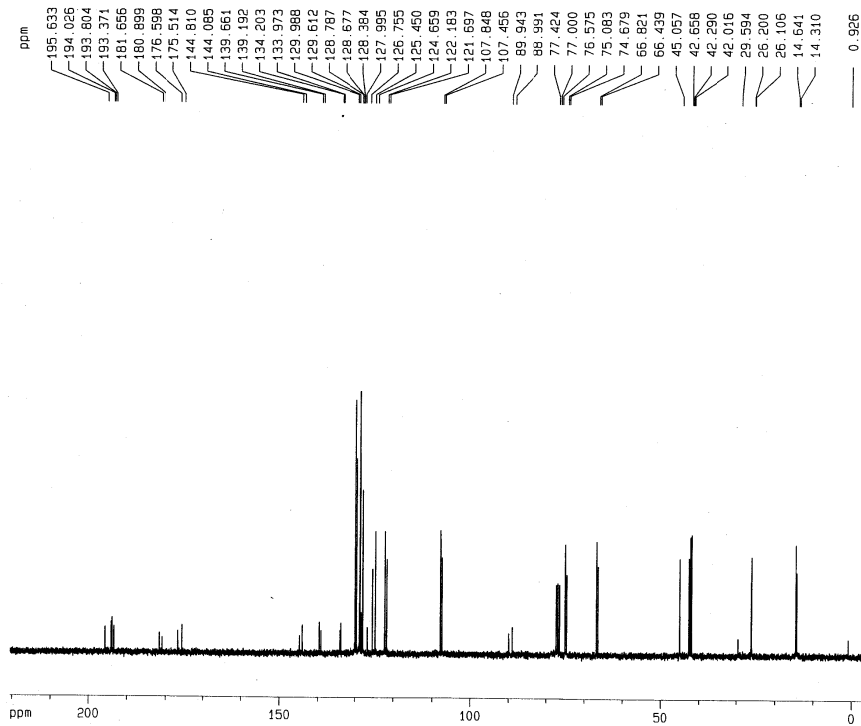
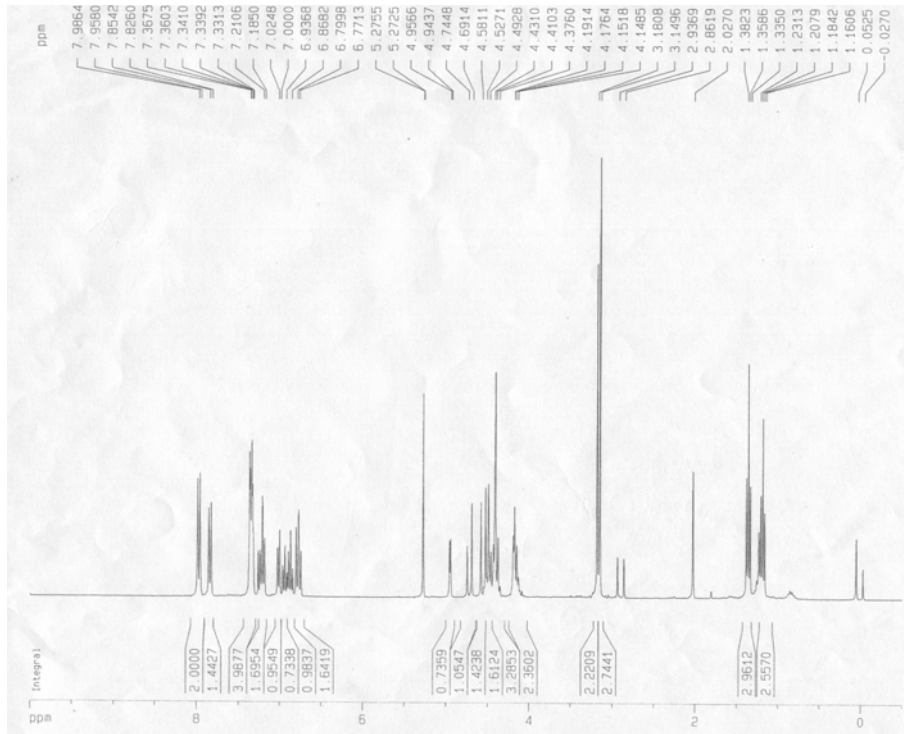
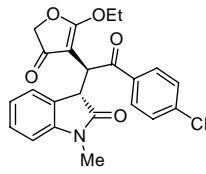
¹H and ¹³C NMR of 5d



¹H and ¹³C NMR of 5e

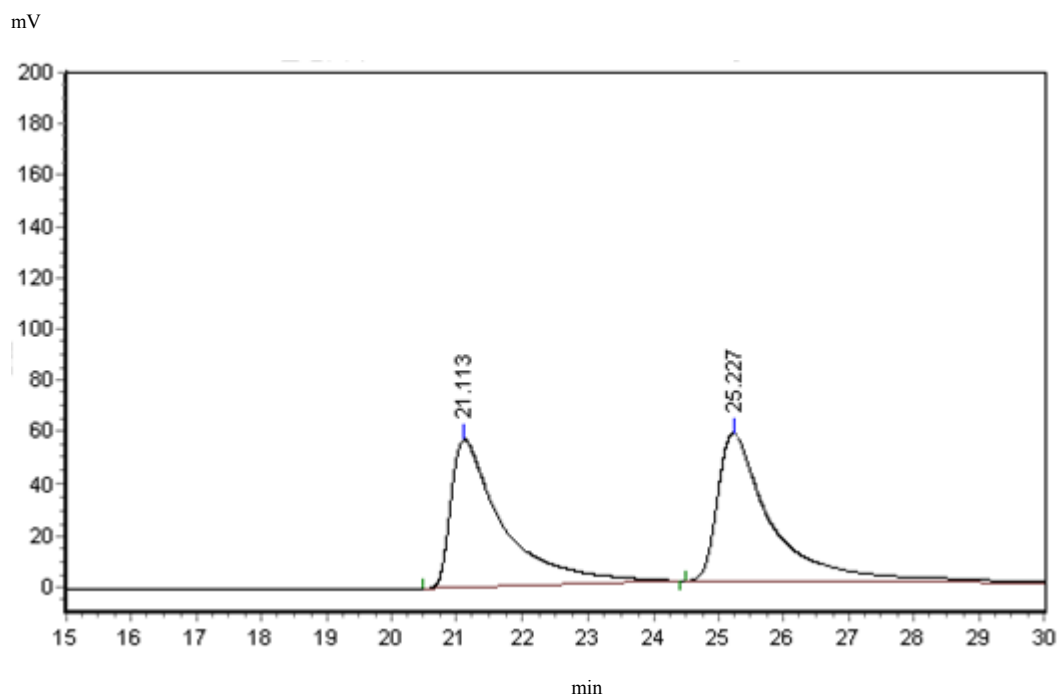


¹H and ¹³C NMR of 5f

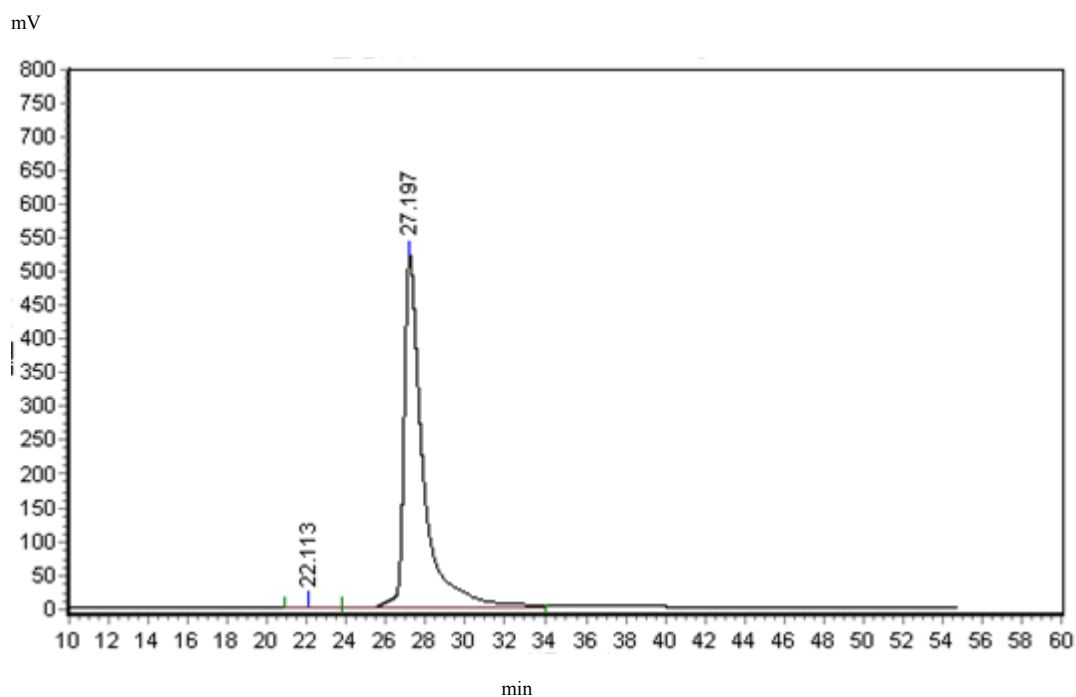


8. HPLC Spectra

HPLC of 4a

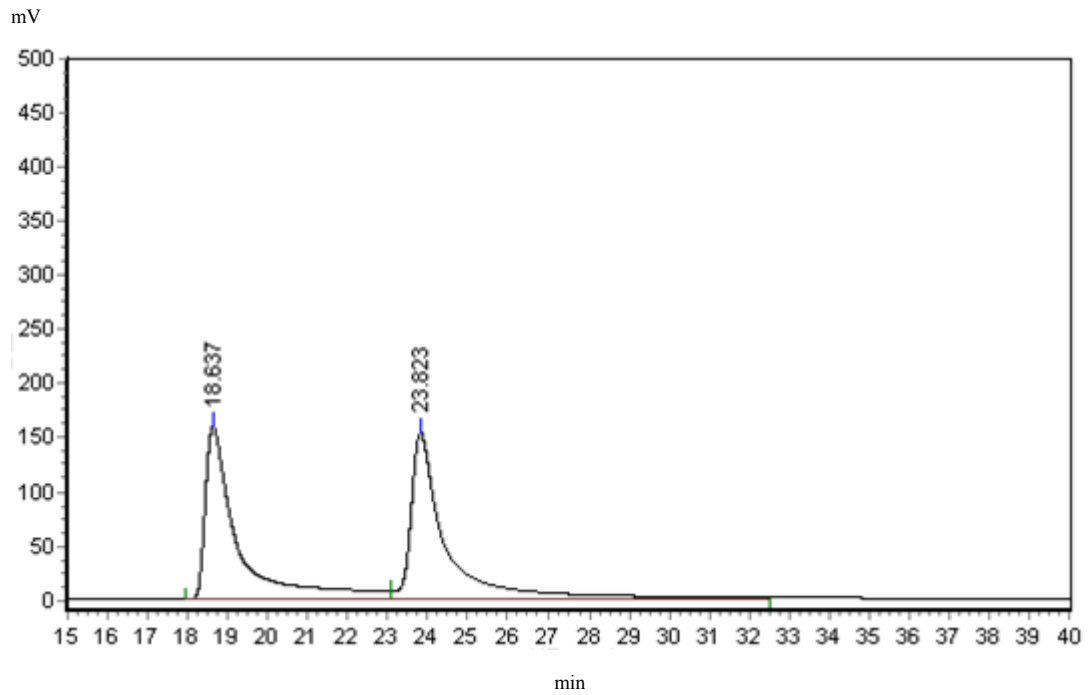


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	21.113	57034.938	3159161.500	48.4768
2	25.227	57097.367	3357692.500	51.5232

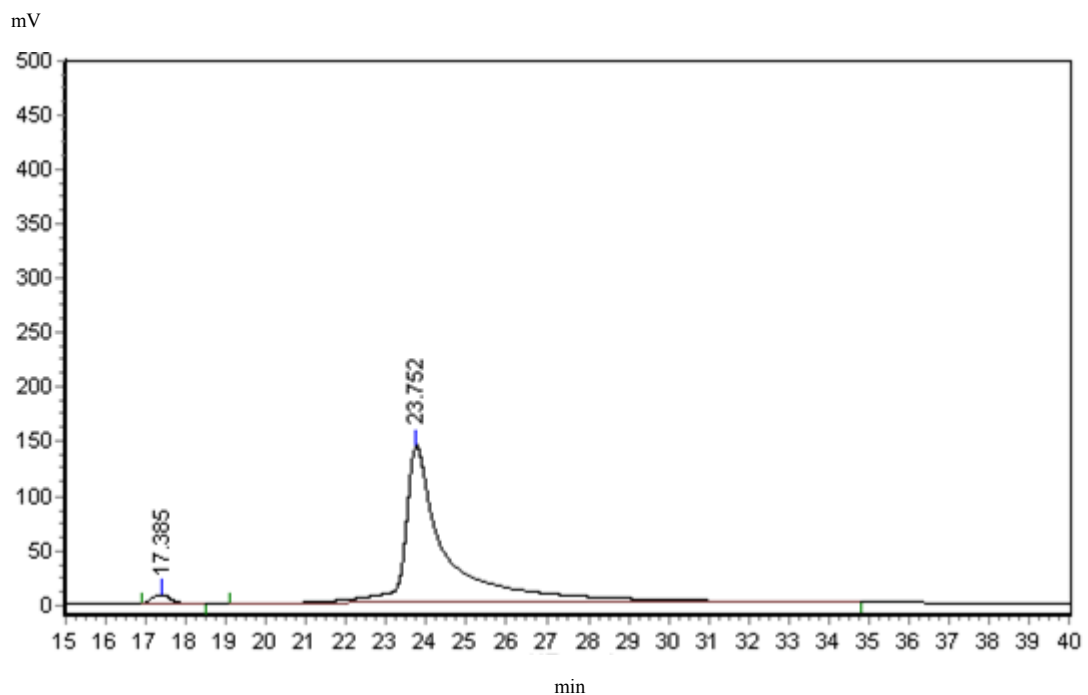


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	22.113	926.785	63574.160	0.1801
2	27.197	522731.063	35235796.000	99.8199

HPLC of 4b

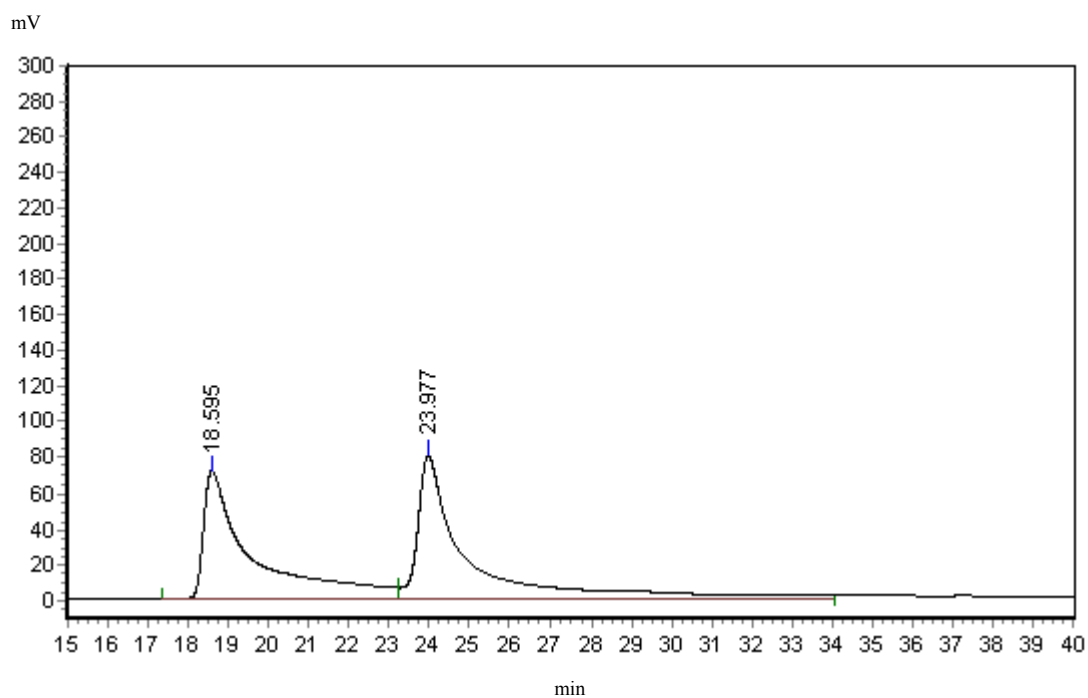


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	18.637	158864.156	9073485.000	48.5972
2	23.823	153290.094	9597323.000	51.4028

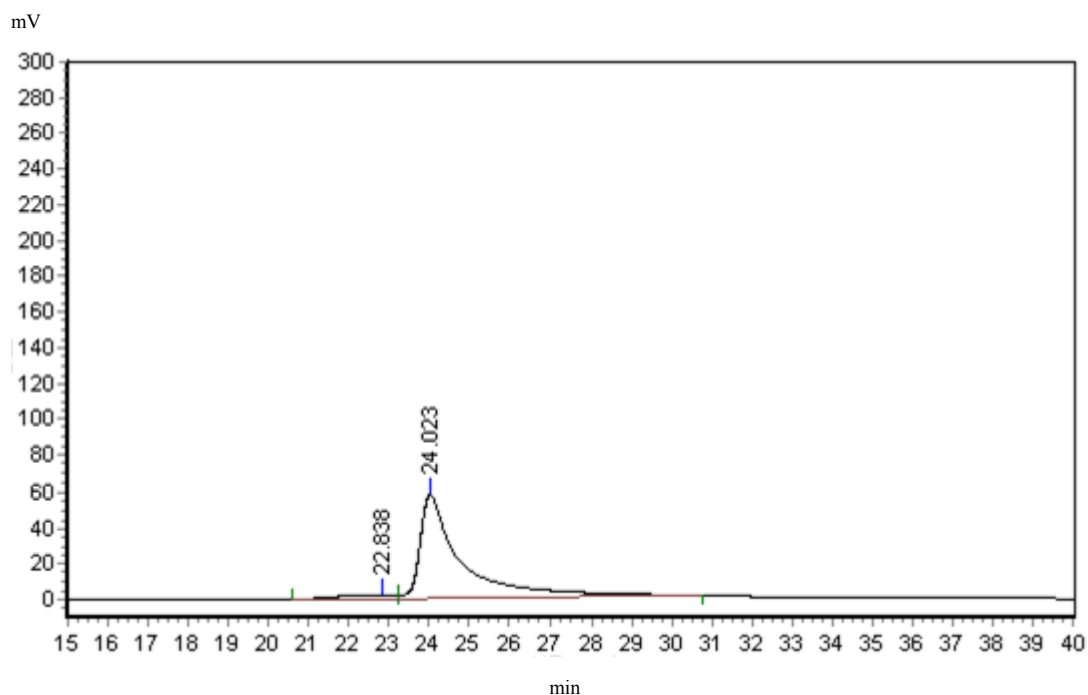


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	17.385	8135.778	261197.547	2.2699
2	23.752	144422.578	11246038.000	97.7301

HPLC of 4c

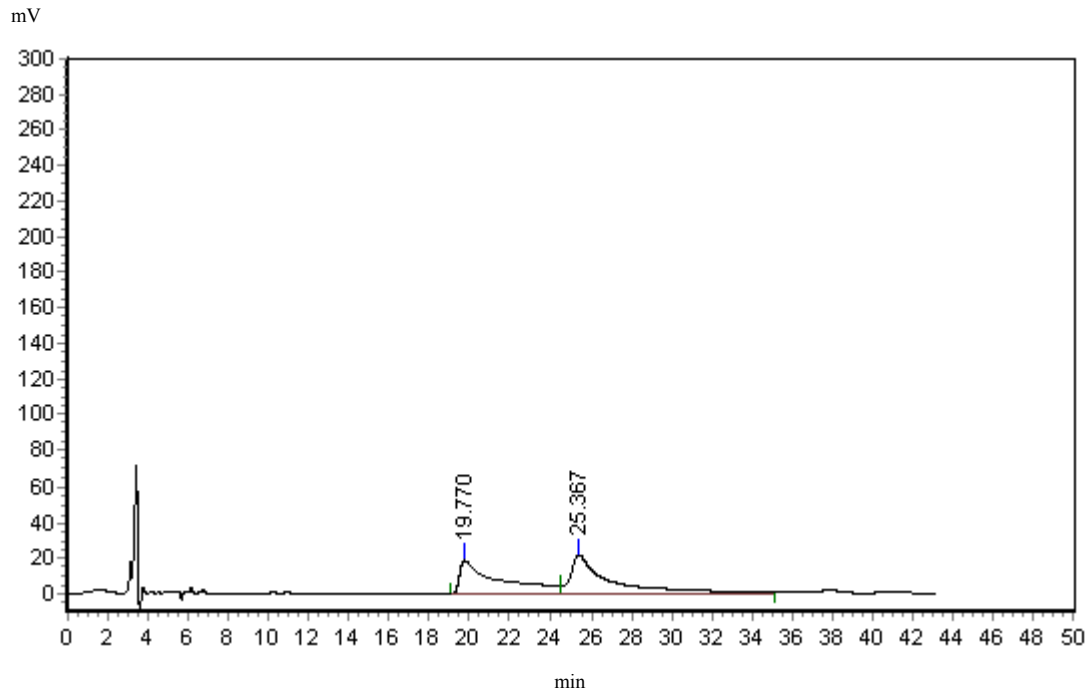


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	18.595	71069.992	6053160.500	47.9130
2	23.977	79654.258	6580487.000	52.0870

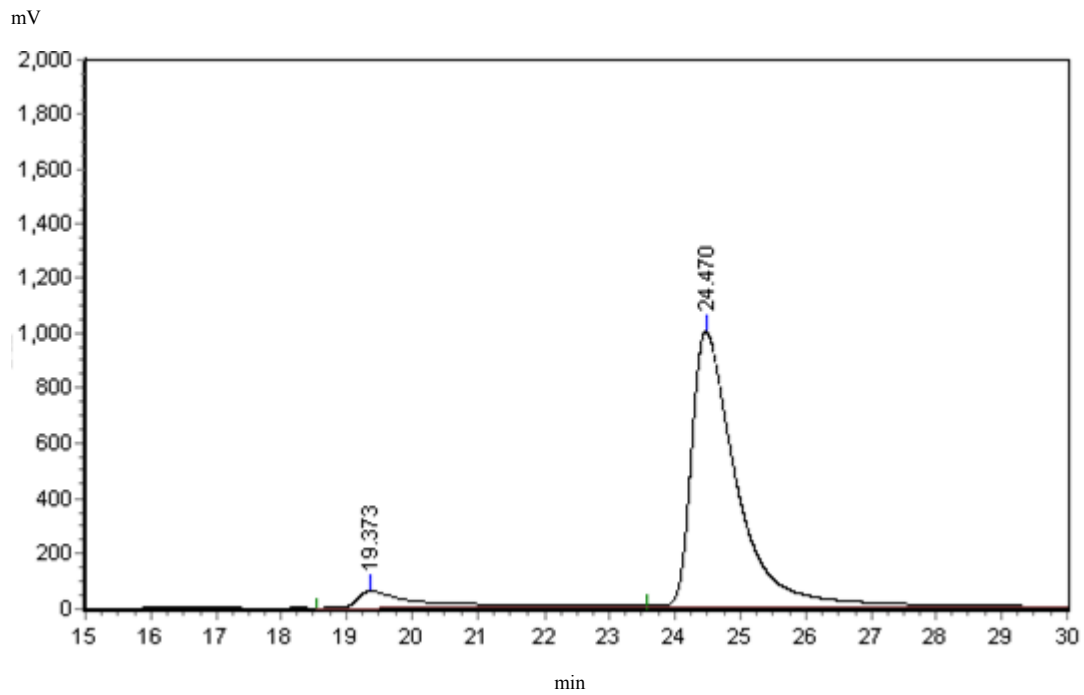


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	22.838	2009.948	213303.047	4.8532
2	24.023	57724.043	4181803.750	95.1468

HPLC of 4d

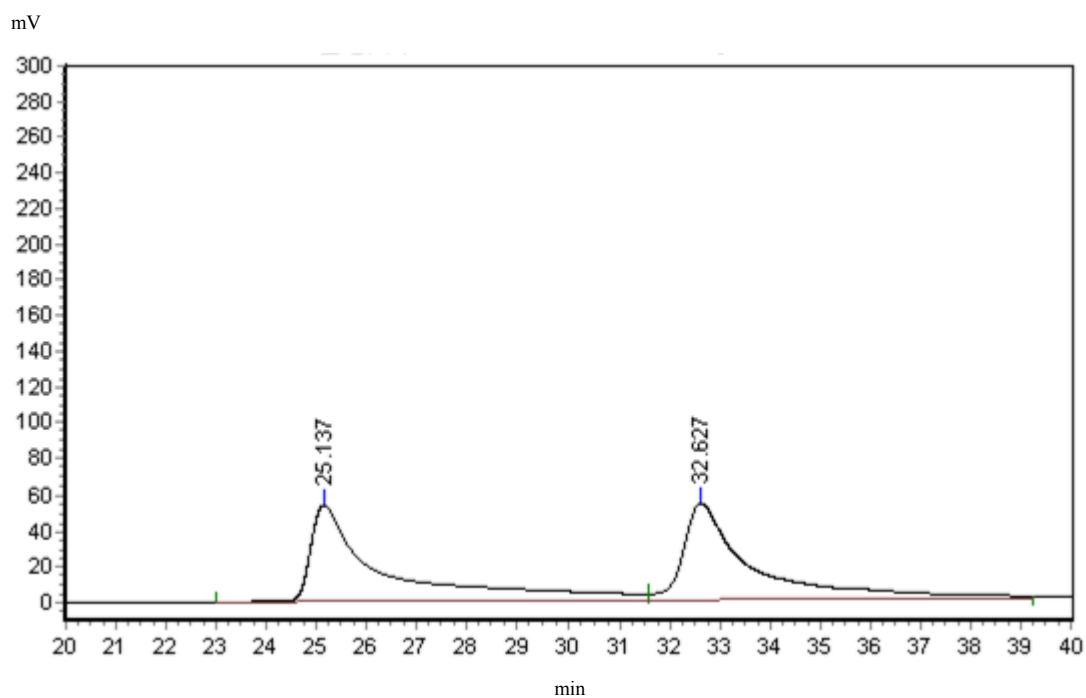


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	19.770	18489.016	2462103.500	45.5696
2	25.367	21754.004	2940848.750	54.4304

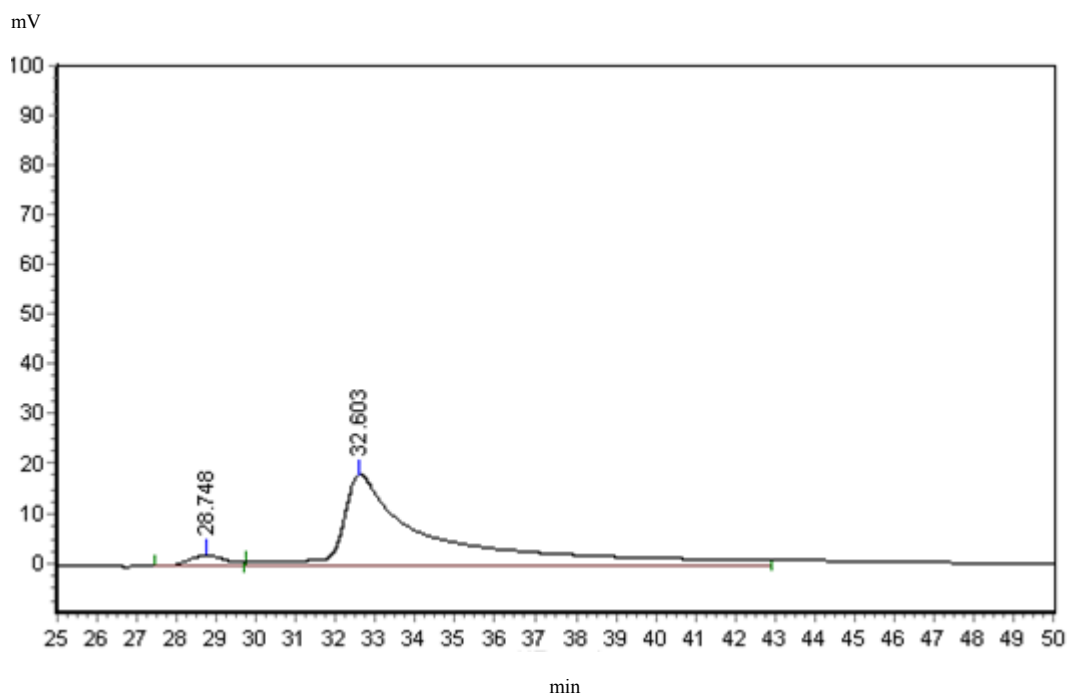


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	19.373	60494.559	5169482.000	9.2006
2	24.470	1005555.000	51017048.000	90.7994

HPLC of 4e

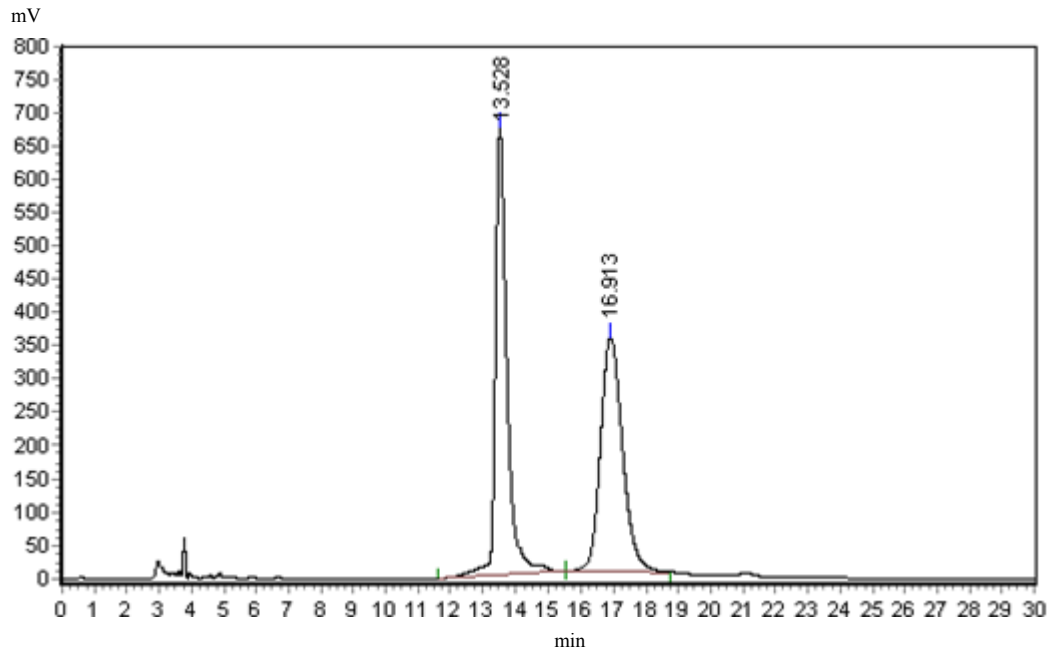


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	18.637	158864.156	9073485.000	48.5972
2	23.823	153290.094	9597323.000	51.4028

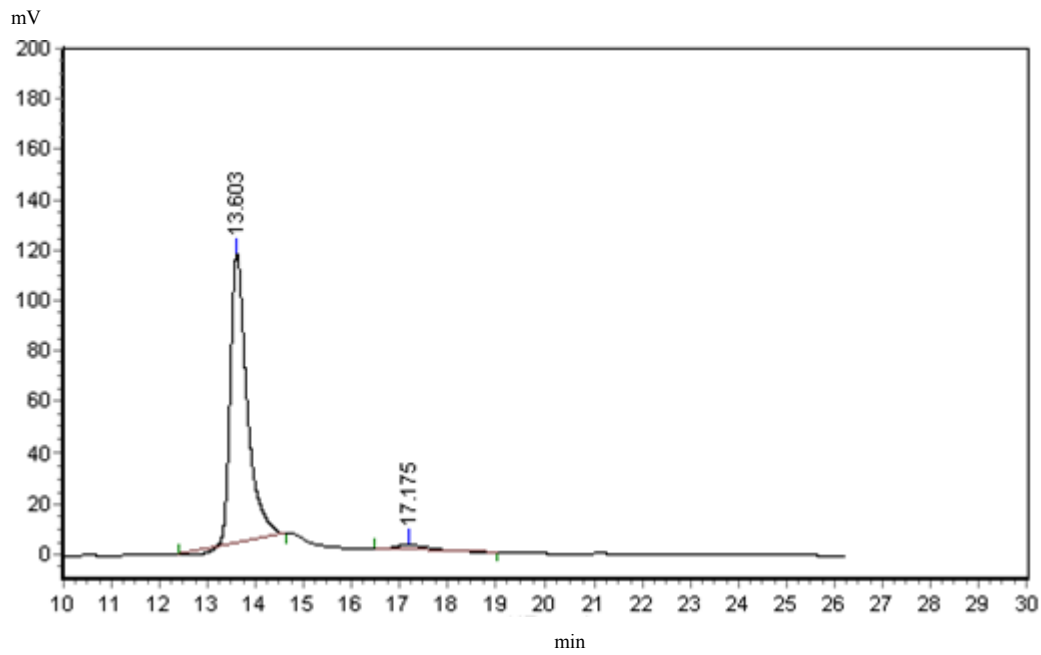


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	28.748	2173.176	143278.578	4.6452
2	32.603	18145.834	2941175.750	95.3548

HPLC of 4f

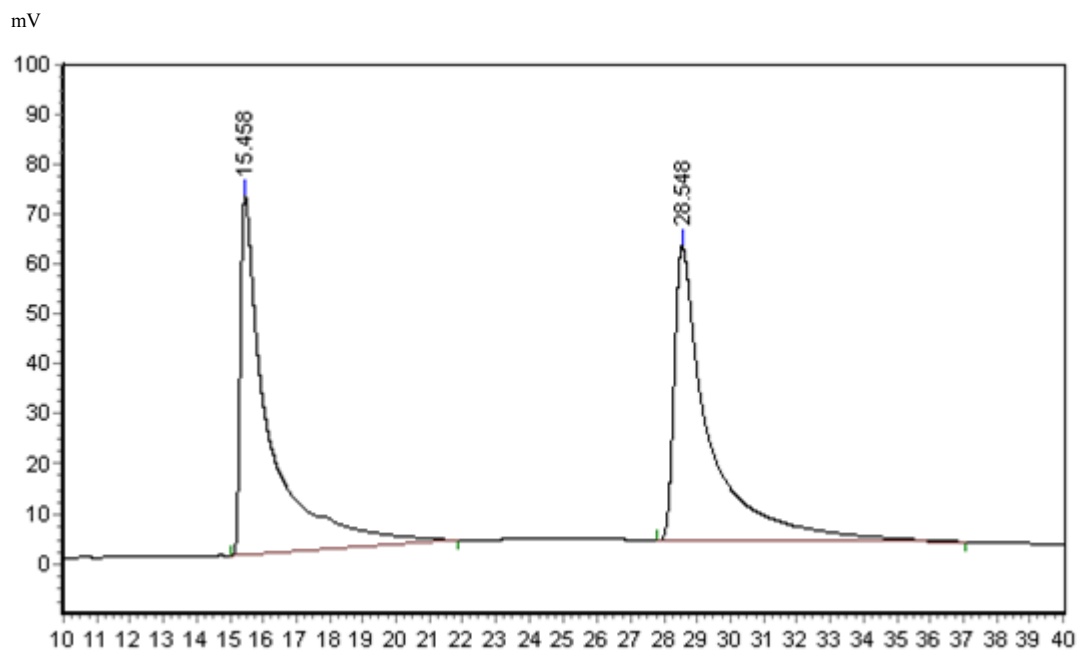


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	13.528	676555.375	15809841.000	48.6206
2	16.913	353296.406	16706911.000	51.3794

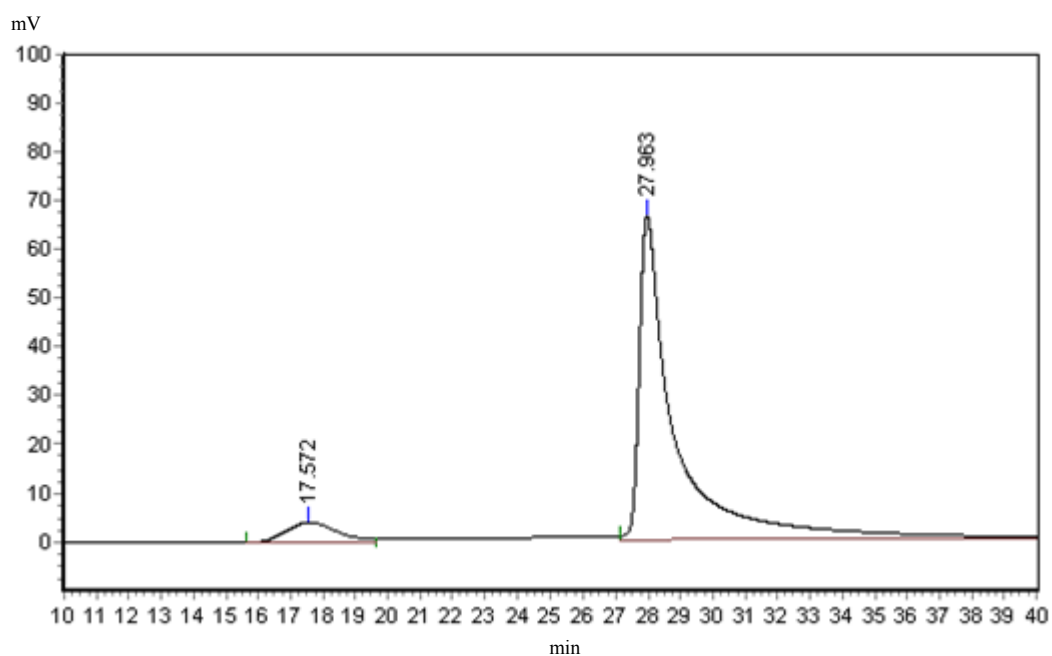


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	13.603	114582.305	2793556.250	96.1340
2	17.175	2092.568	112342.469	3.8660

HPLC of 4g



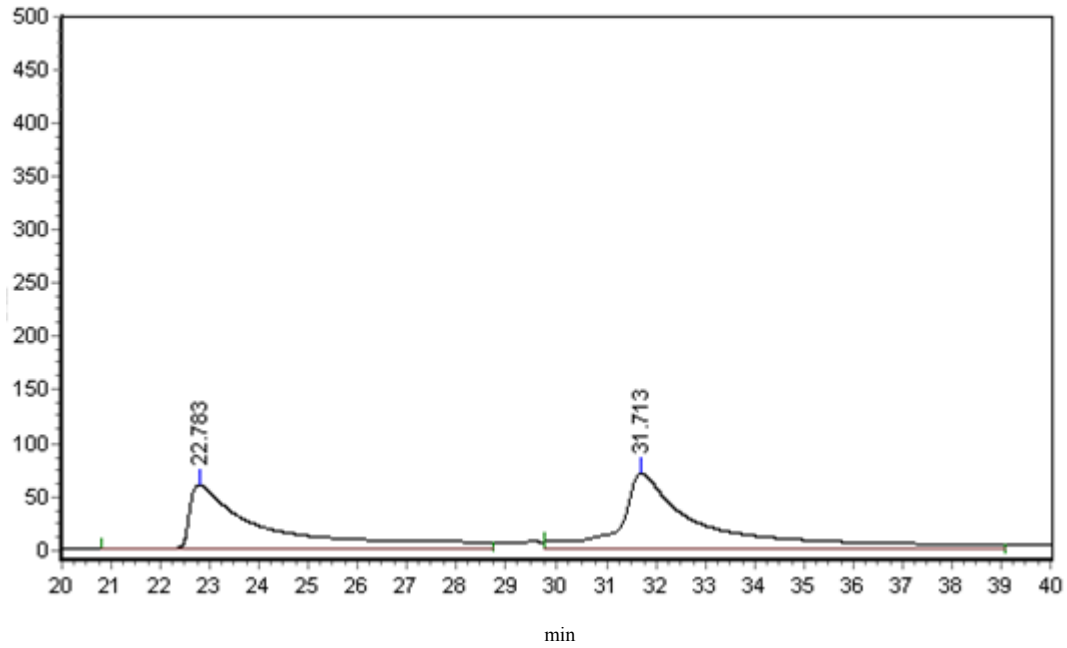
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	15.458	71622.102	4247303.500	49.3414
2	28.548	58938.176	4360694.500	50.6586



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	17.572	4054.498	450936.500	7.8607
2	27.963	66477.234	5285693.000	92.1394

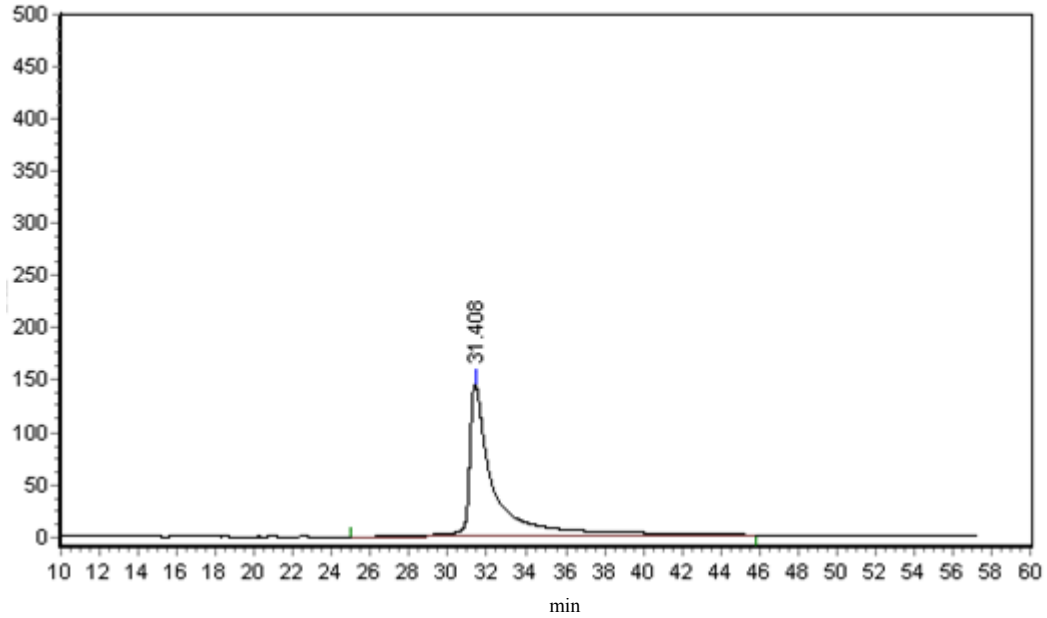
HPLC of 4h

mV



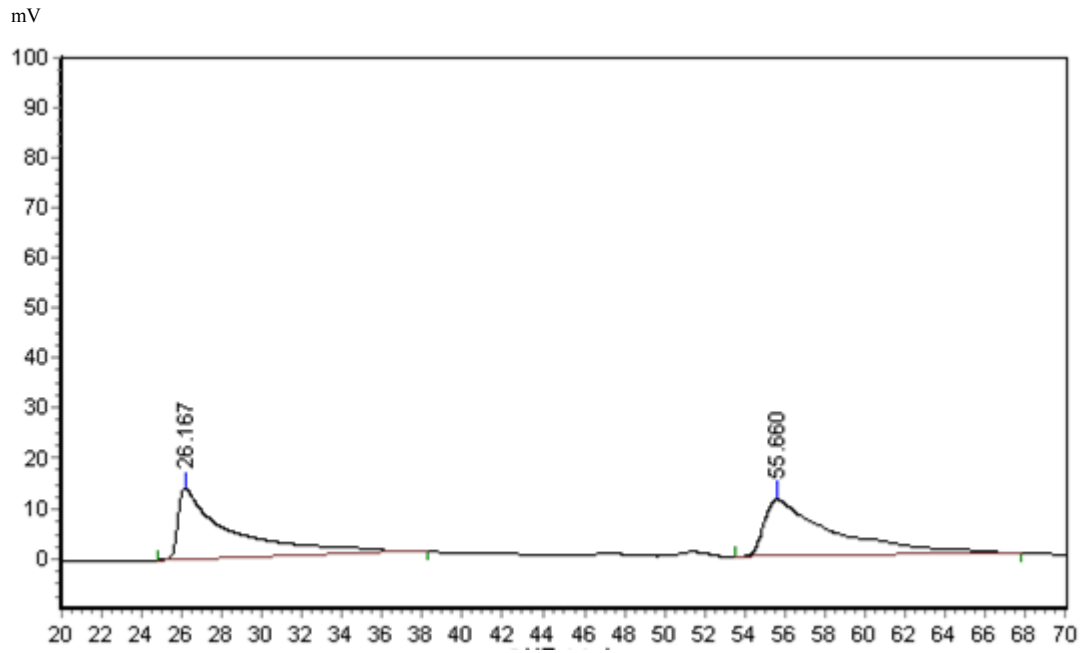
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	22.783	60930.469	6667377.000	43.5111
2	31.713	71760.727	8656026.000	56.4889

mV

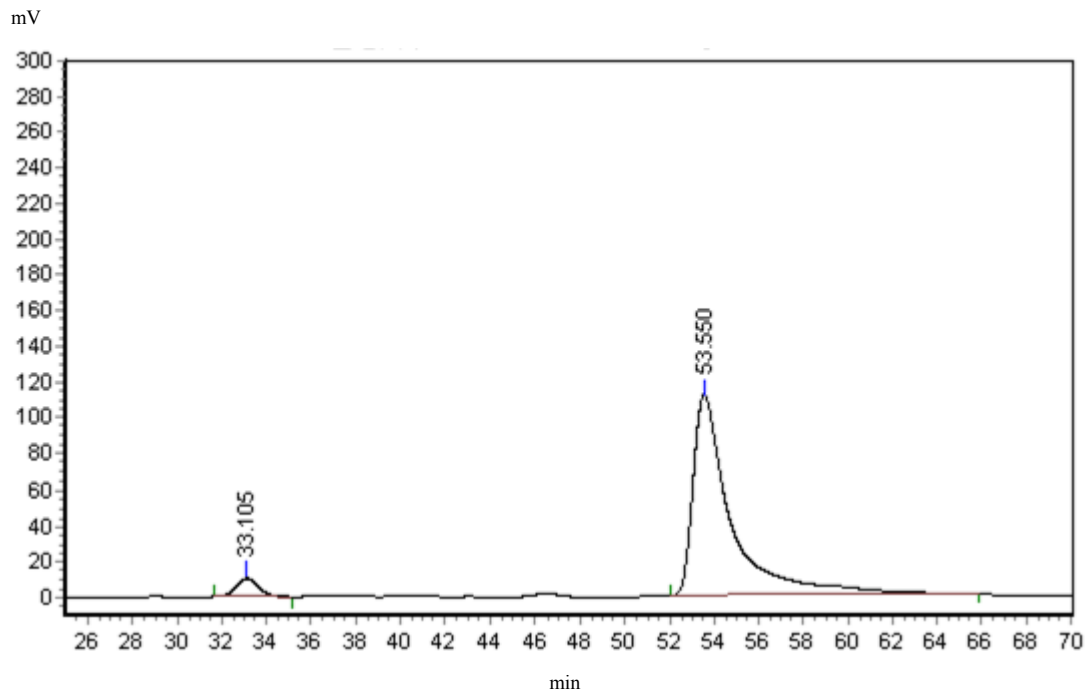


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	31.408	145808.547	13296896.000	100.0000

HPLC of 4i



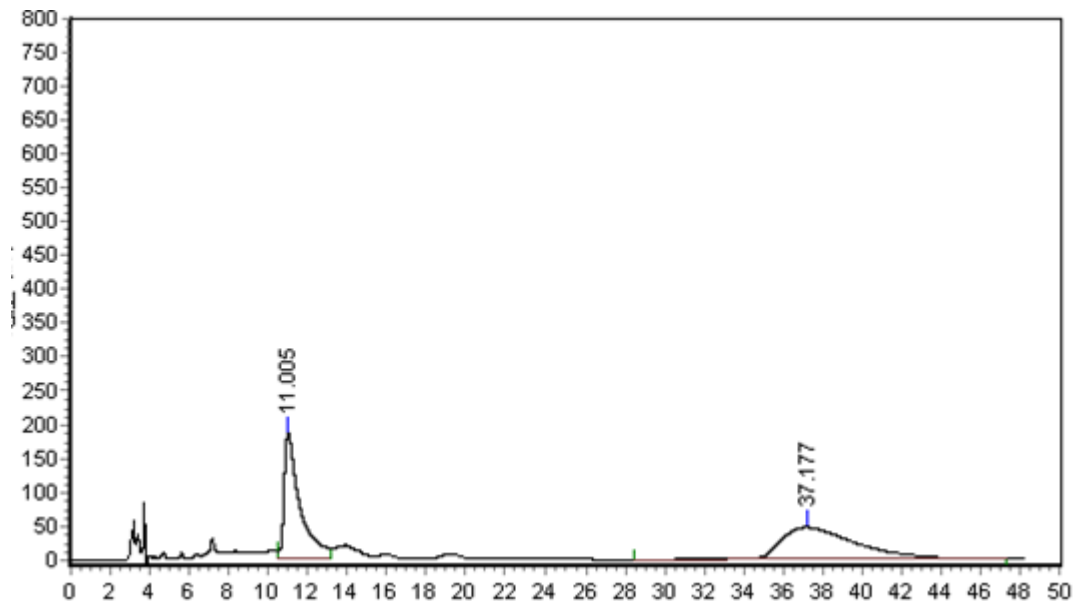
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	26.167	14086.023	2676671.250	49.2470
2	55.660	11337.842	2758529.250	50.7530



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	33.105	10210.298	699896.375	4.8426
2	53.550	112164.961	13752926.000	95.1574

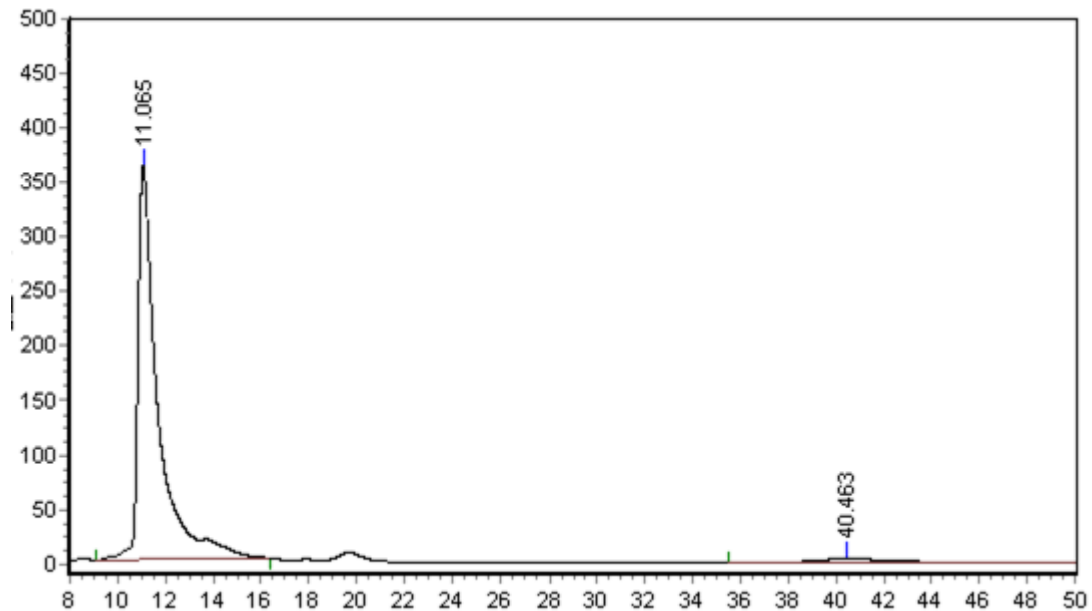
HPLC of 4j

mV



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	11.005	185894.375	10298760.000	44.7711
2	37.177	47316.285	12704383.000	55.2289

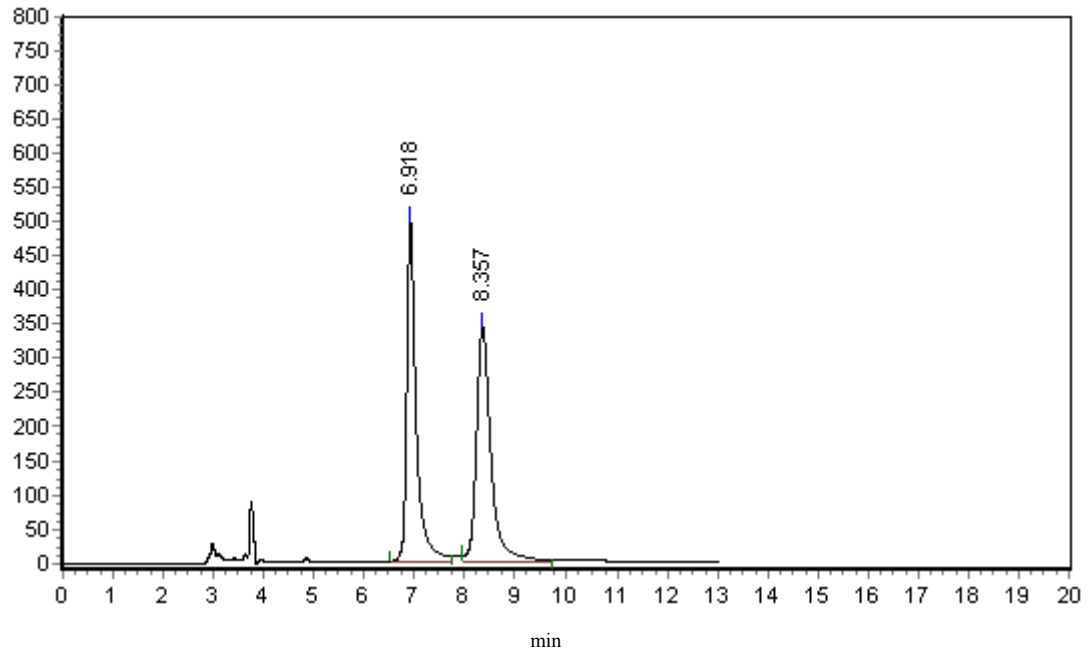
mV



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	11.065	360846.094	21111378.000	94.5442
2	40.463	4082.953	1218253.875	5.4558

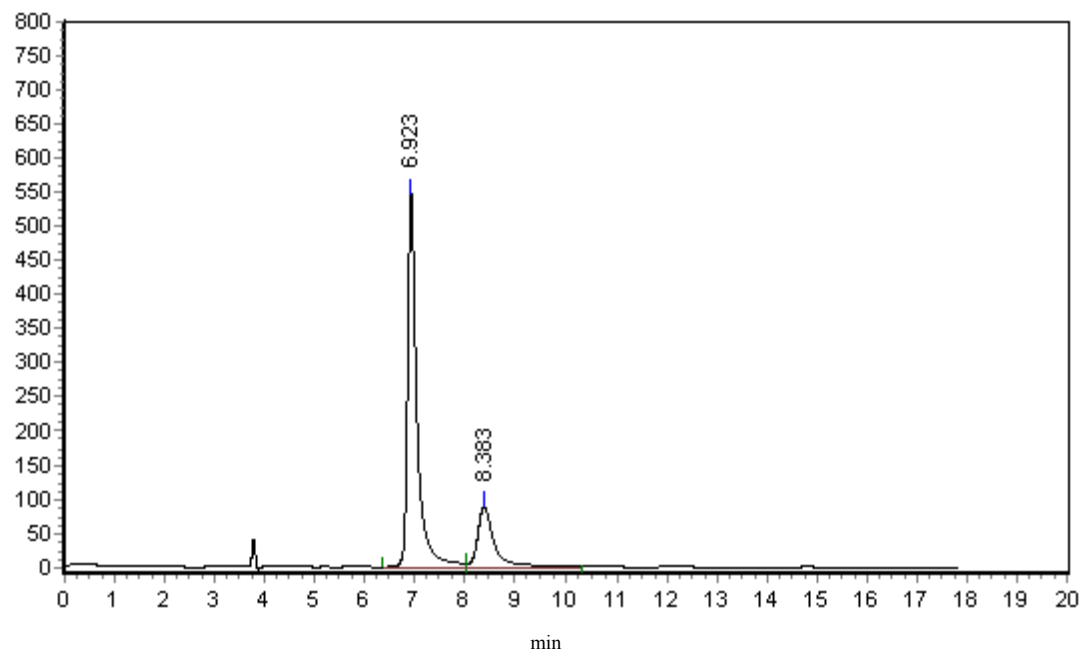
HPLC of 4k

mV



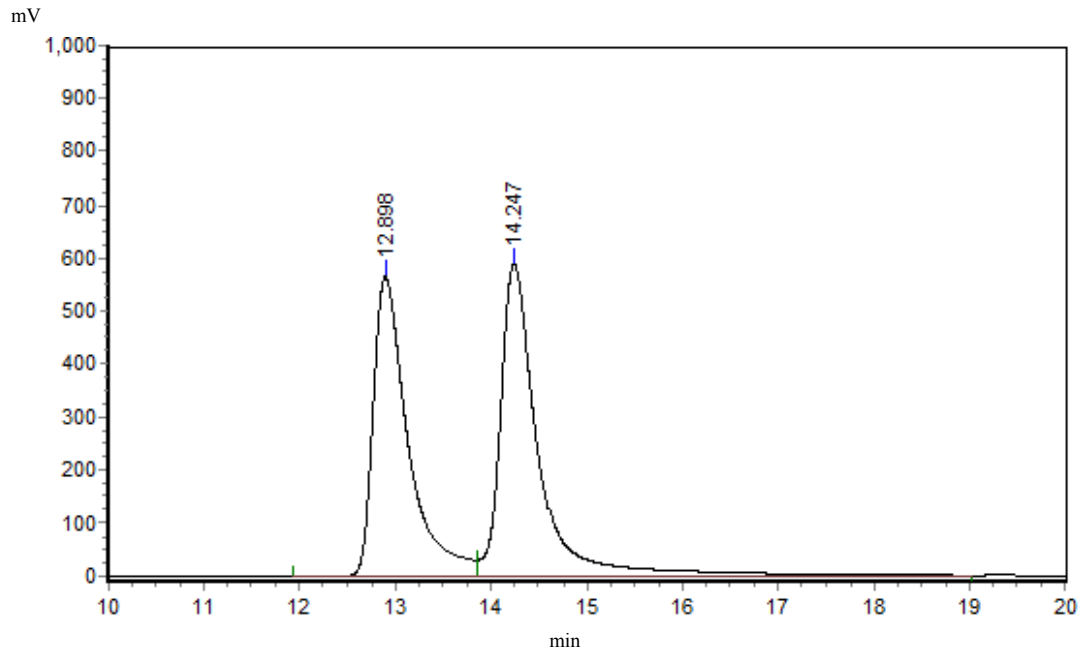
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	6.918	496906.563	6561655.000	48.5883
2	8.357	343034.656	6942956.500	51.4117

mV

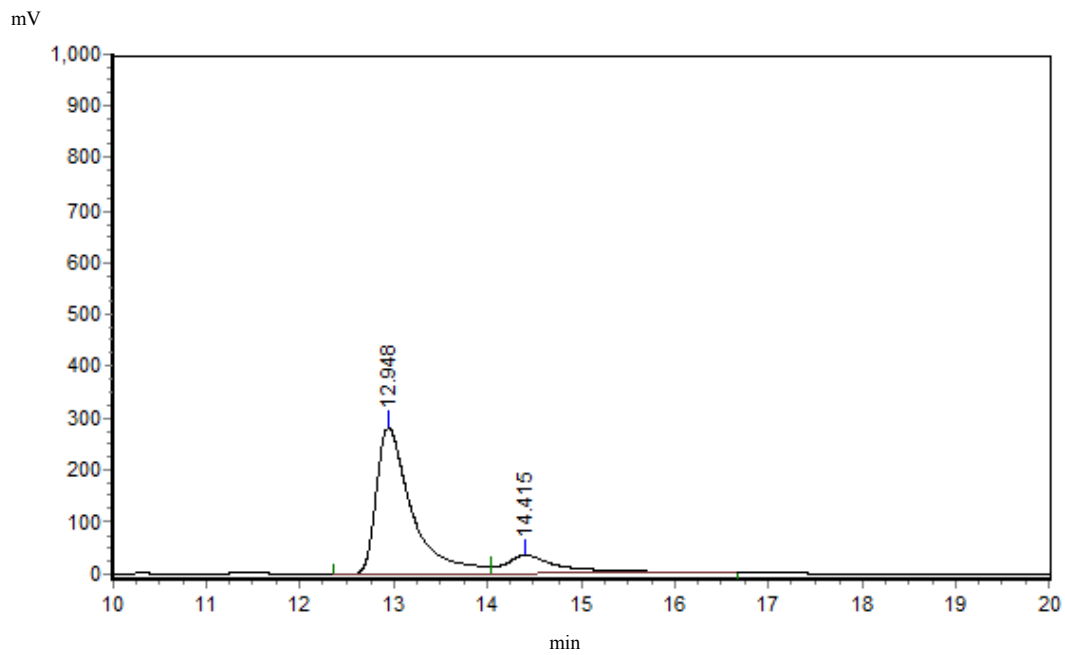


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	6.923	547429.875	7151476.500	77.5960
2	8.383	88634.570	2064822.375	22.4040

HPLC of 4I

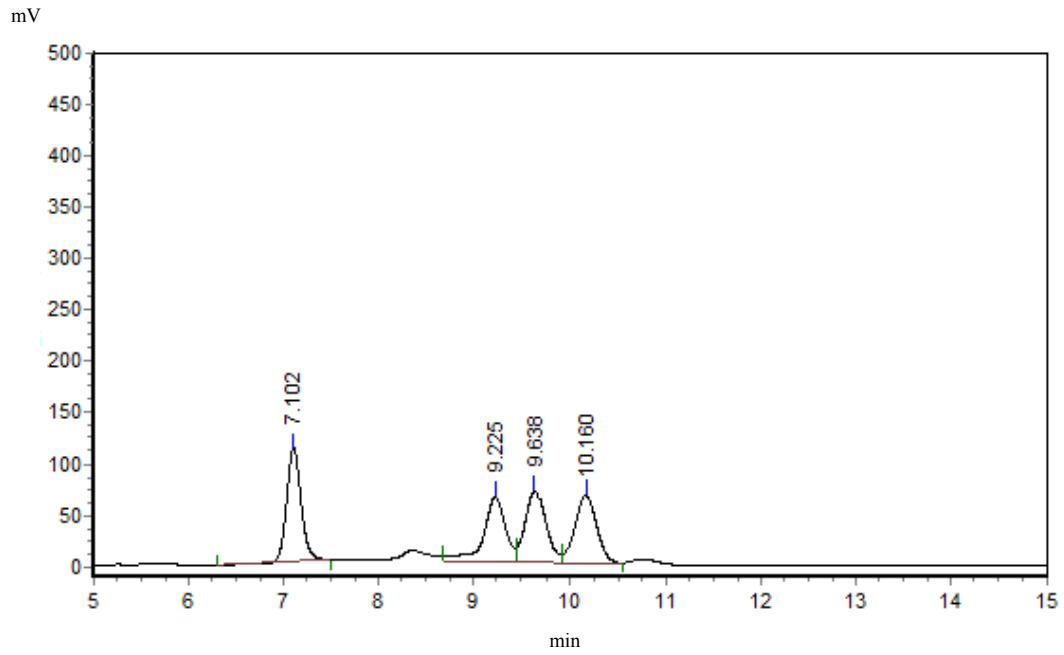


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	12.898	564359.813	14282760.000	46.6219
2	14.247	587730.000	16352512.000	53.3781

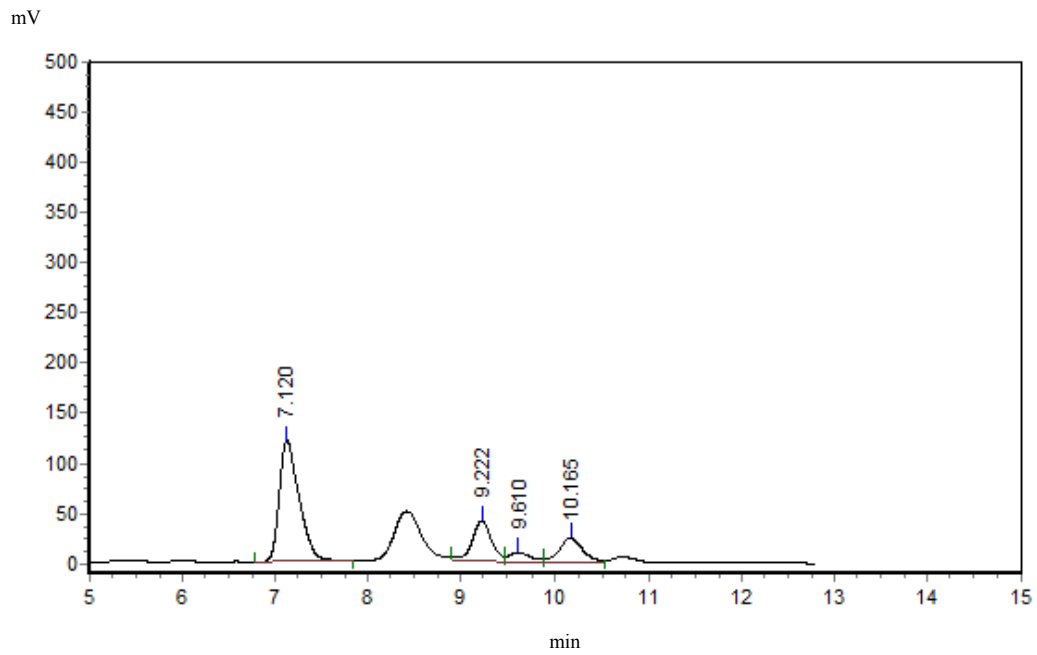


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	12.948	281216.250	7416119.500	82.7825
2	14.415	36398.543	1542440.625	17.2175

HPLC of 4m

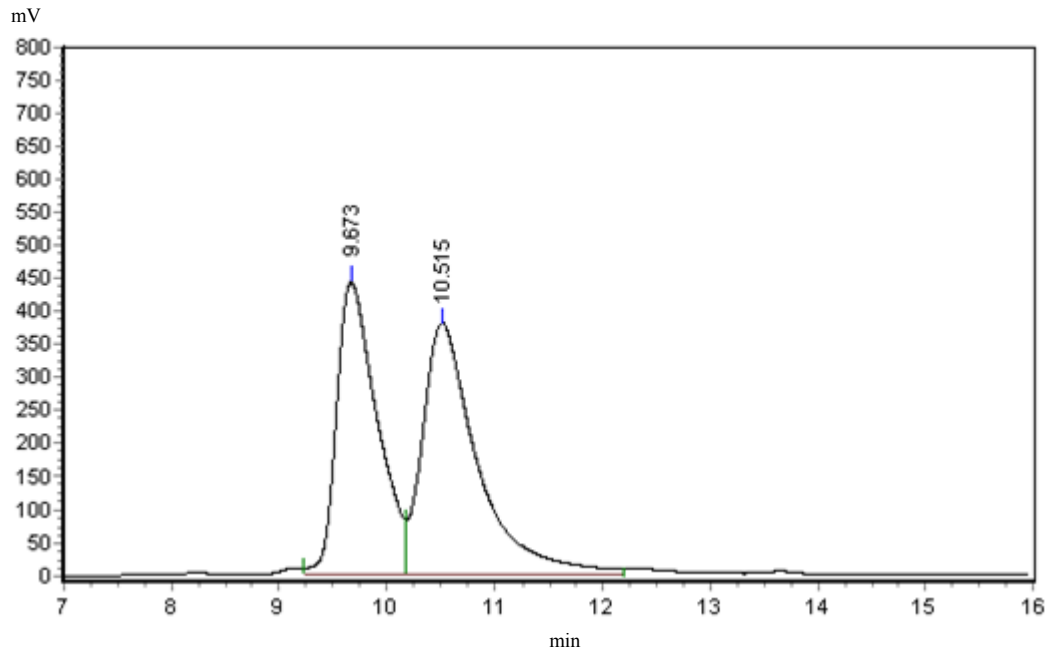


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	7.102	109165.617	1129207.875	26.8996
2	9.225	63122.781	1024552.750	24.4065
3	9.638	68817.586	996239.500	23.7321
4	10.160	66661.102	1047861.125	24.9618

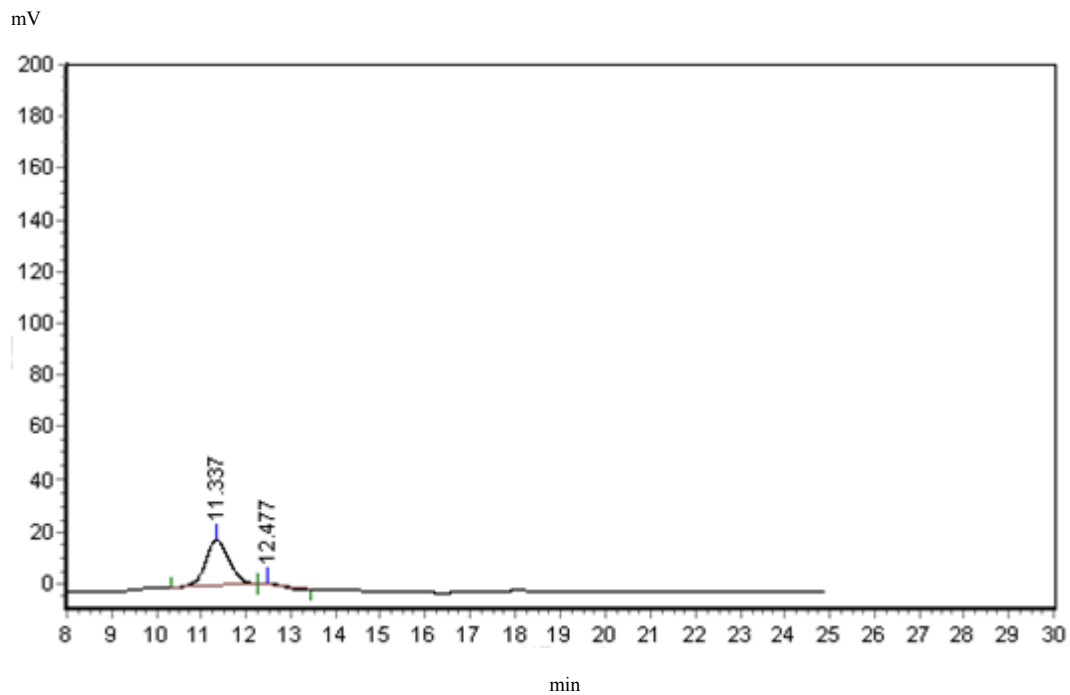


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	7.120	121000.328	1803288.500	61.4996
2	9.222	40532.441	584209.438	19.9239
3	9.610	8910.404	139049.641	4.7422
4	10.165	23656.100	405649.563	13.8343

HPLC of 4n

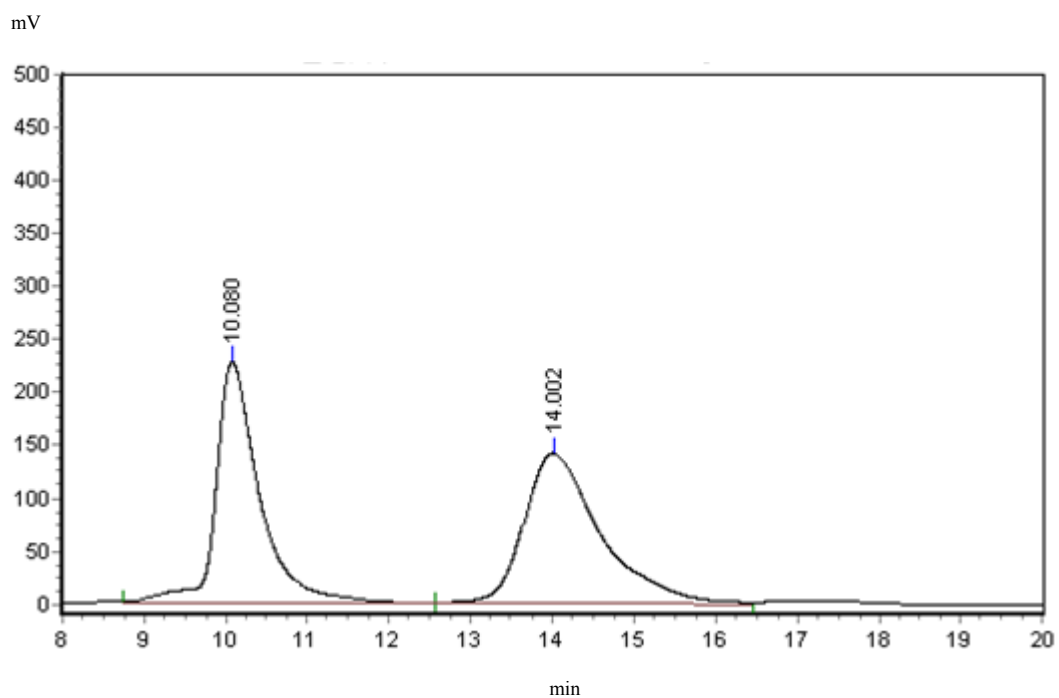


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	9.673	442705.375	11584841.000	46.0644
2	10.515	381609.469	13564391.000	53.9356

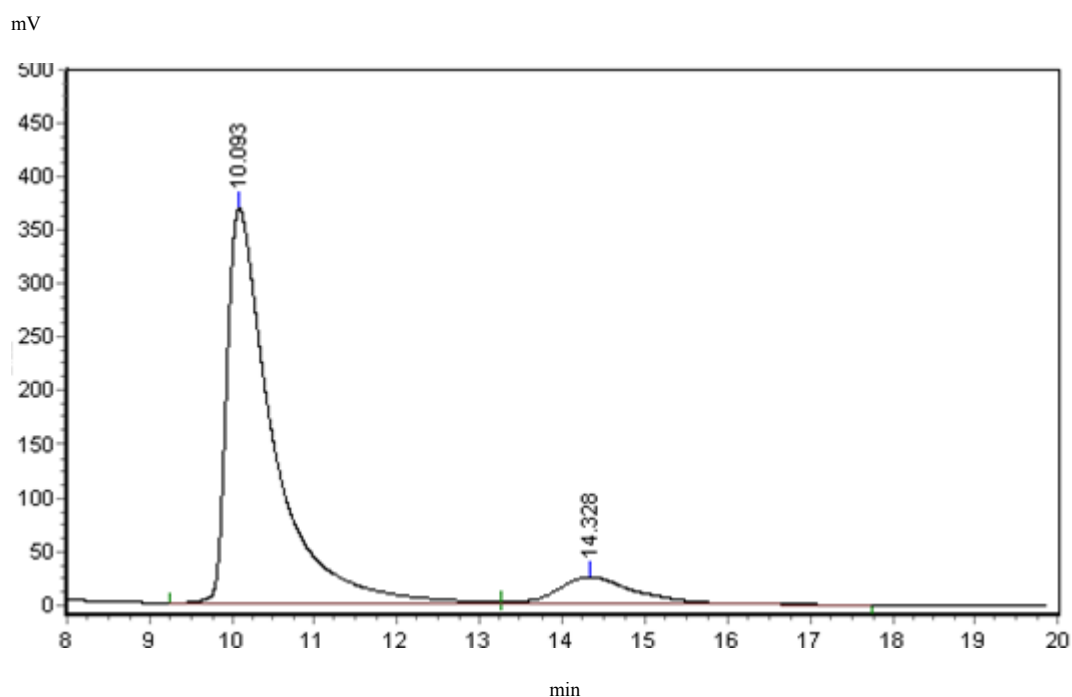


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	11.337	17853.051	651255.688	98.1479
2	12.477	563.472	12289.600	1.8521

HPLC of 4o

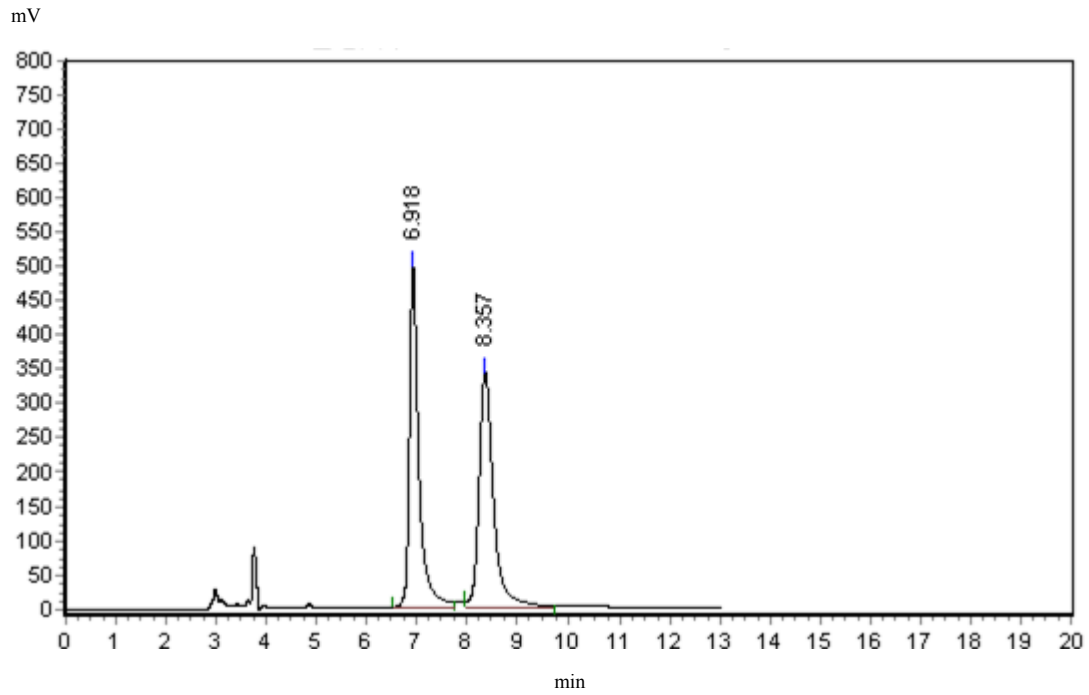


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	10.080	226868.469	8554892.000	48.0722
2	14.002	141295.547	9241038.000	51.9278

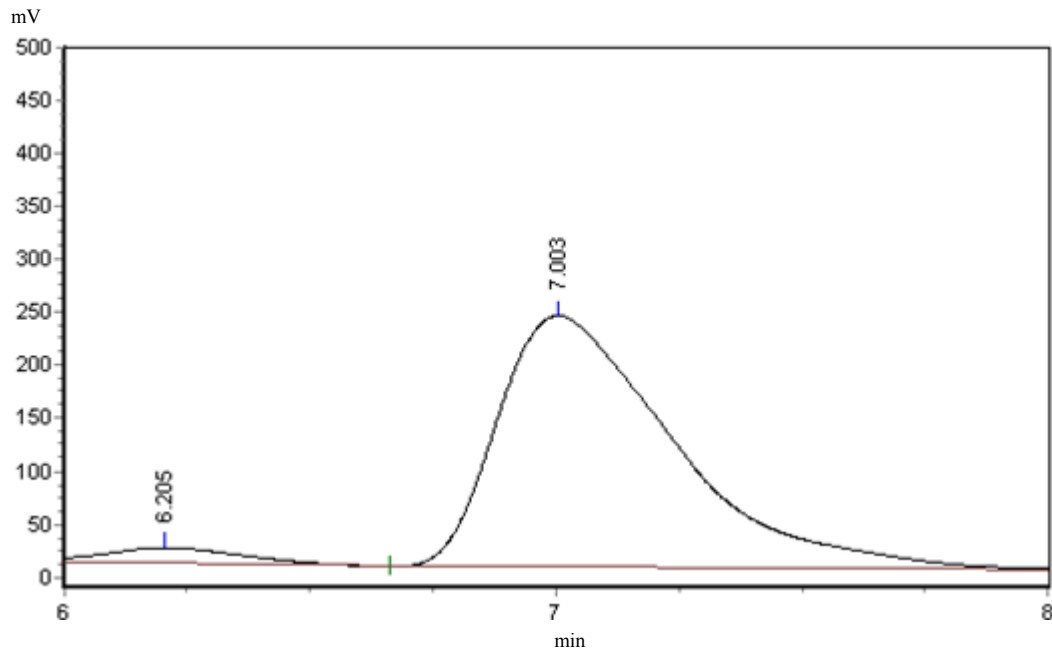


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	10.093	368045.313	14275149.000	89.2708
2	14.328	25302.035	1715685.750	10.7292

HPLC of 4p

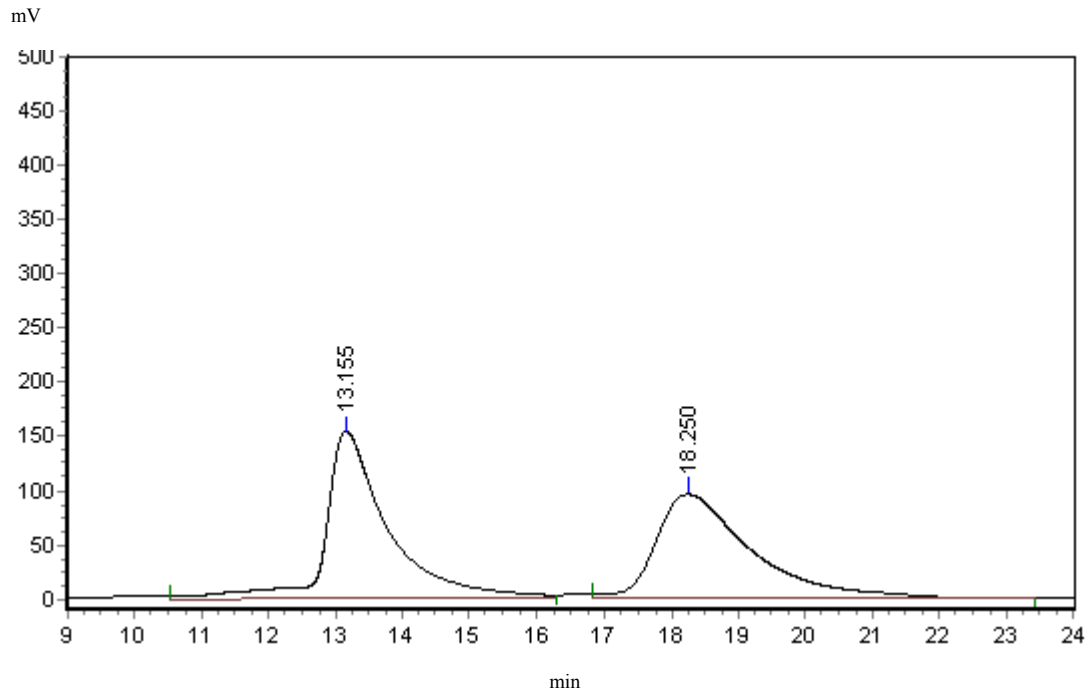


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	6.683	479092.938	10453592.000	48.1533
2	7.900	313905.781	11255392.000	51.8467

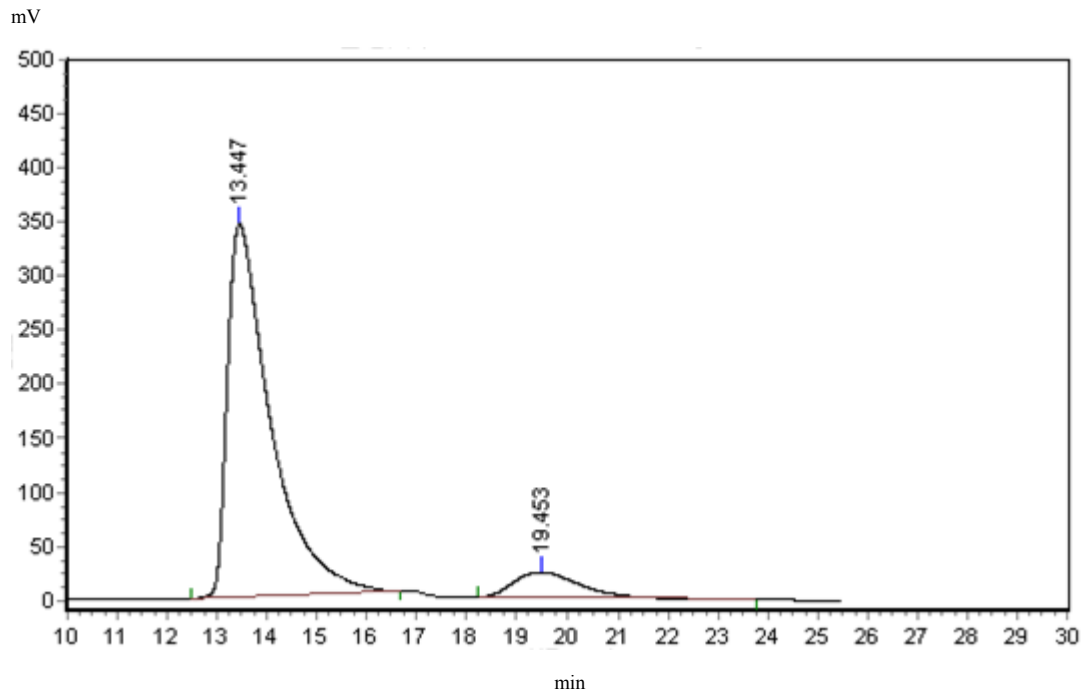


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	6.205	14882.733	302094.469	4.8603
2	7.003	237241.266	5913405.500	95.1397

HPLC of 4q

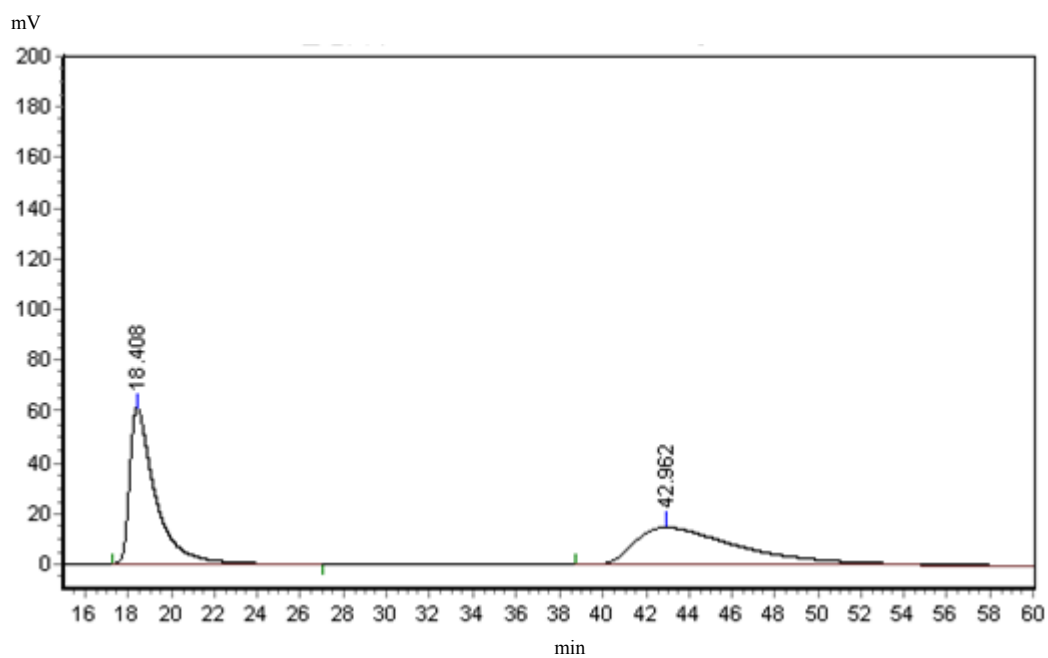


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	13.155	153791.766	10077794.000	51.2487
2	18.250	96628.953	9586699.000	48.7513

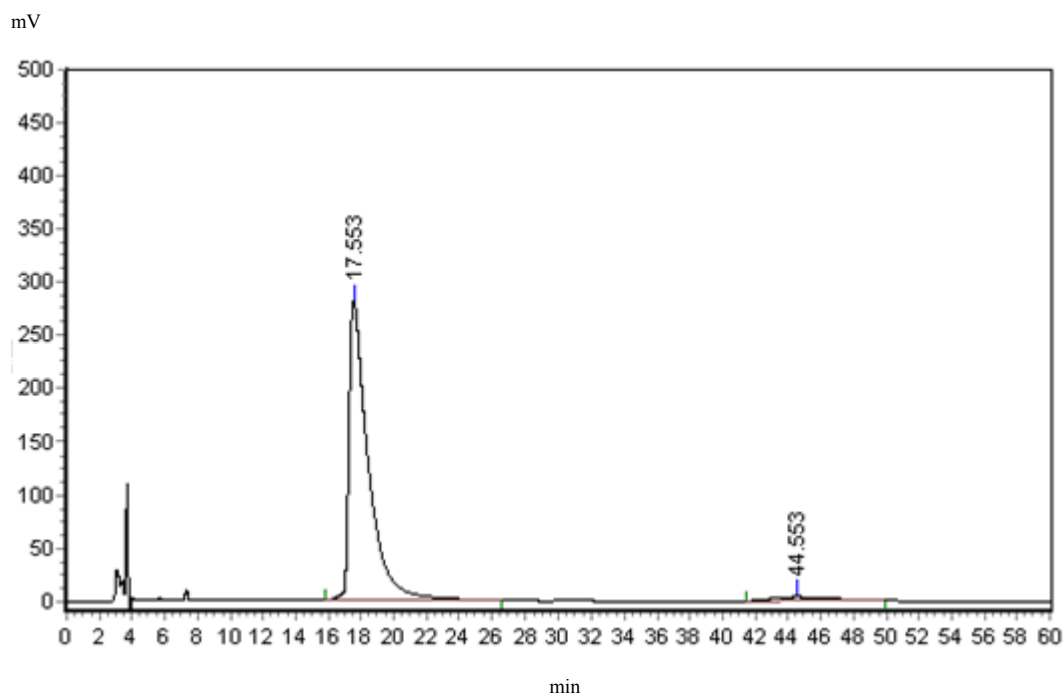


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	13.447	344779.594	20918870.000	90.5845
2	19.453	23620.676	2174350.250	9.4155

HPLC of 4r

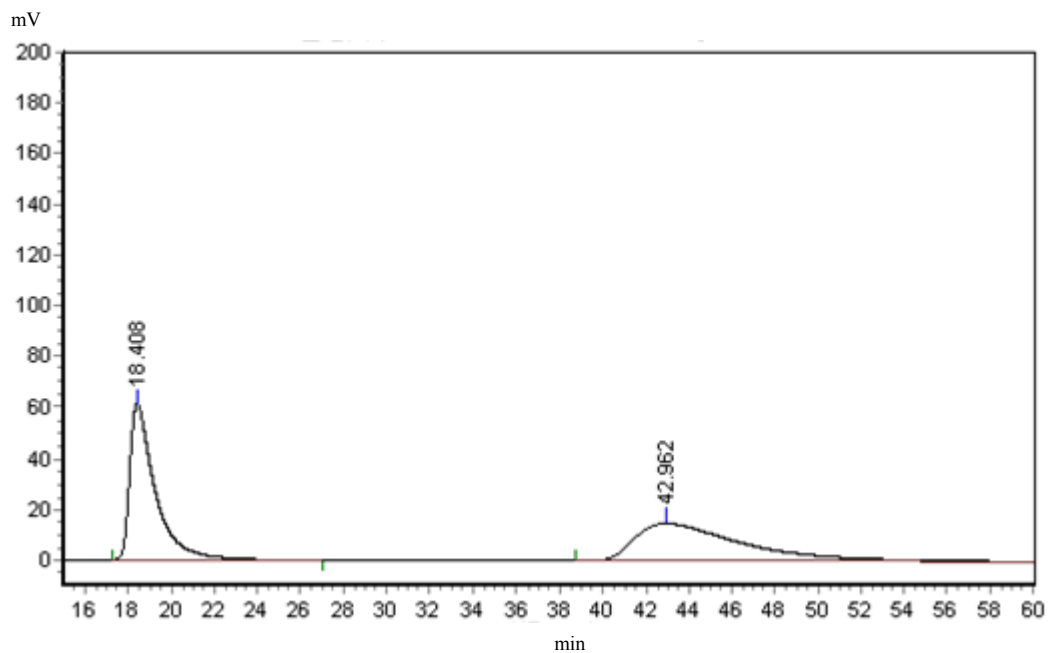


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	18.408	61593.785	5220820.000	50.2430
2	42.962	14787.083	5170321.500	49.7570

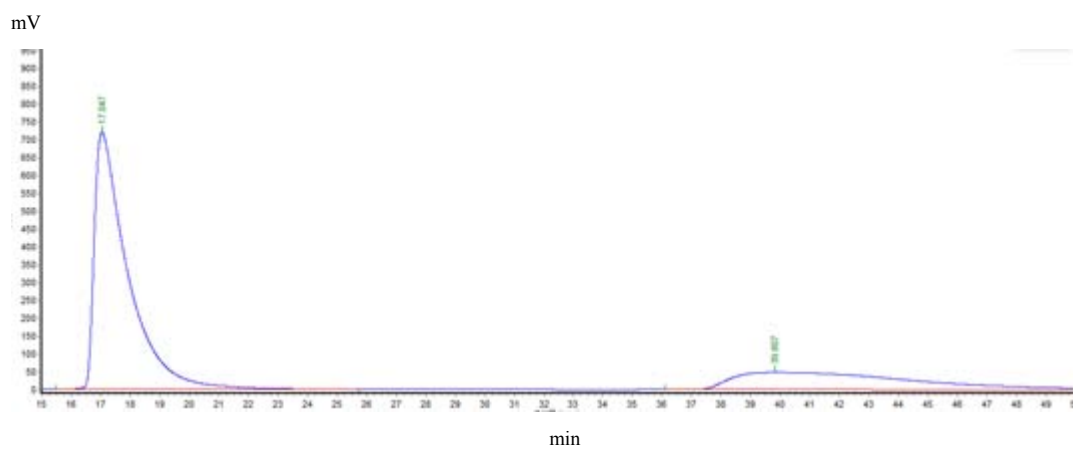


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	17.553	282925.813	23152622.000	95.9030
2	44.553	3829.811	989074.375	4.0970

HPLC of 4r



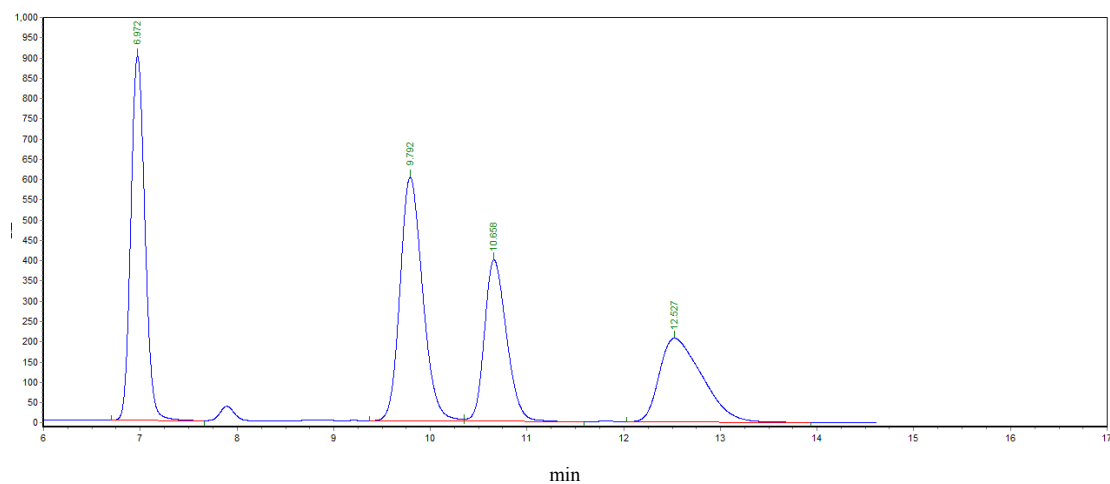
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	18.408	61593.785	5220820.000	50.2430
2	42.962	14787.083	5170321.500	49.7570



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	17.047	720118.250	56530640.000	73.5515
2	39.807	47790.965	20327964.000	26.4485

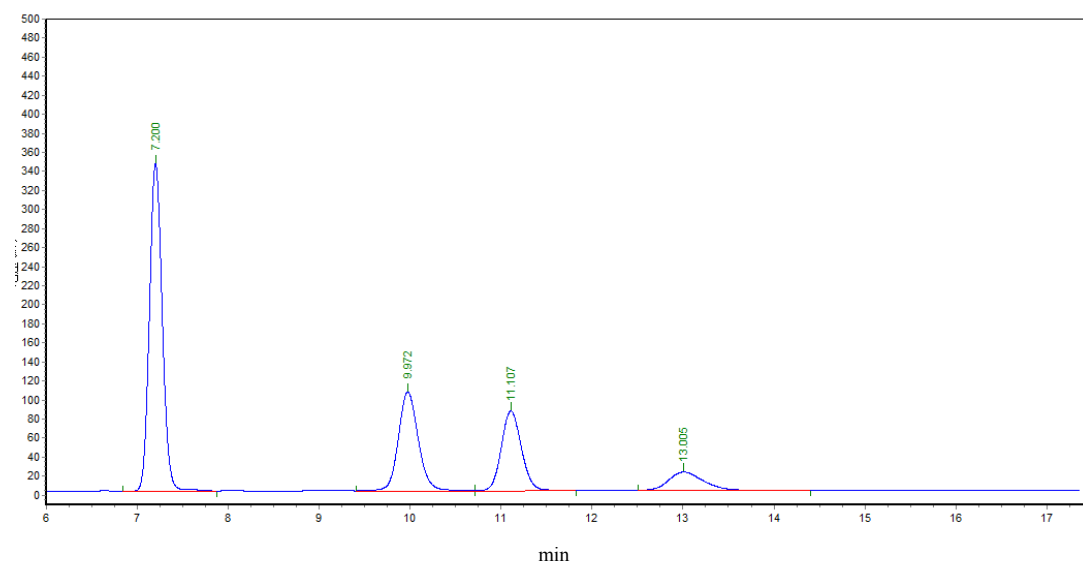
HPLC of 4s

mV



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	6.972	898813.188	9054237.000	28.7559
2	9.792	601734.750	9795805.000	31.1111
3	10.658	398656.031	6310513.500	20.0420
4	12.627	206747.531	6325955.500	20.0910

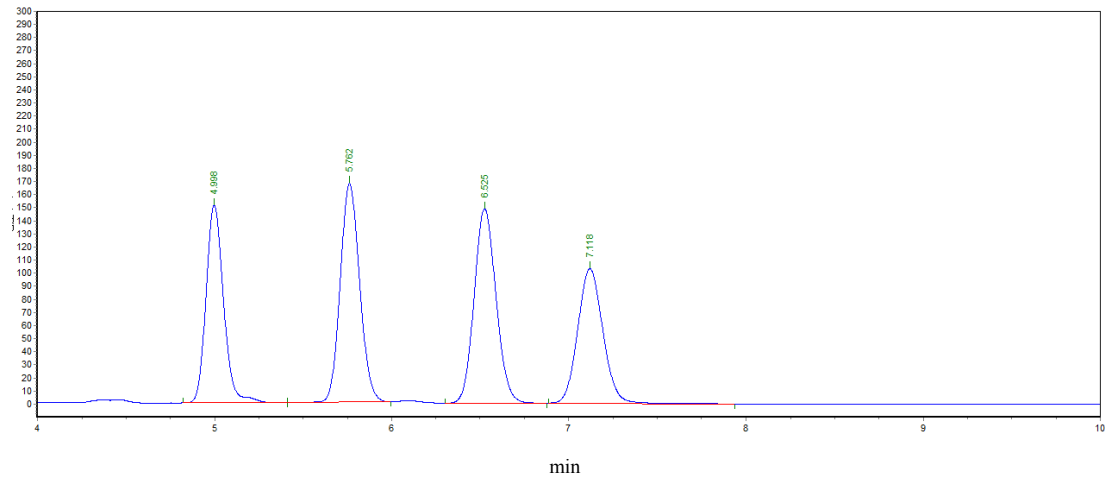
mV



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	7.200	344111.750	3367361.000	49.4937
2	9.972	103987.711	1591791.000	23.3963
3	11.107	83987.828	1301169.625	19.1247
4	13.005	19659.314	543293.188	7.9854

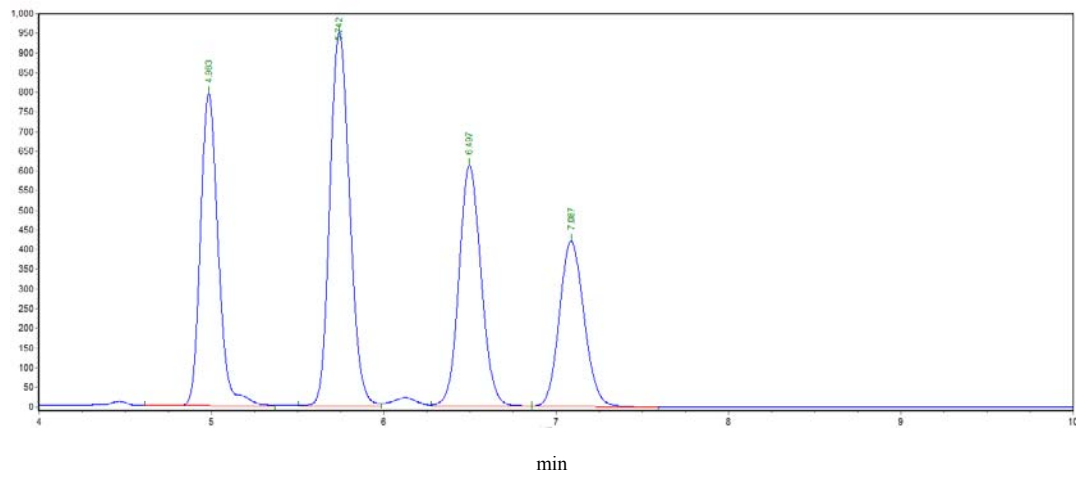
HPLC of 4t

mV



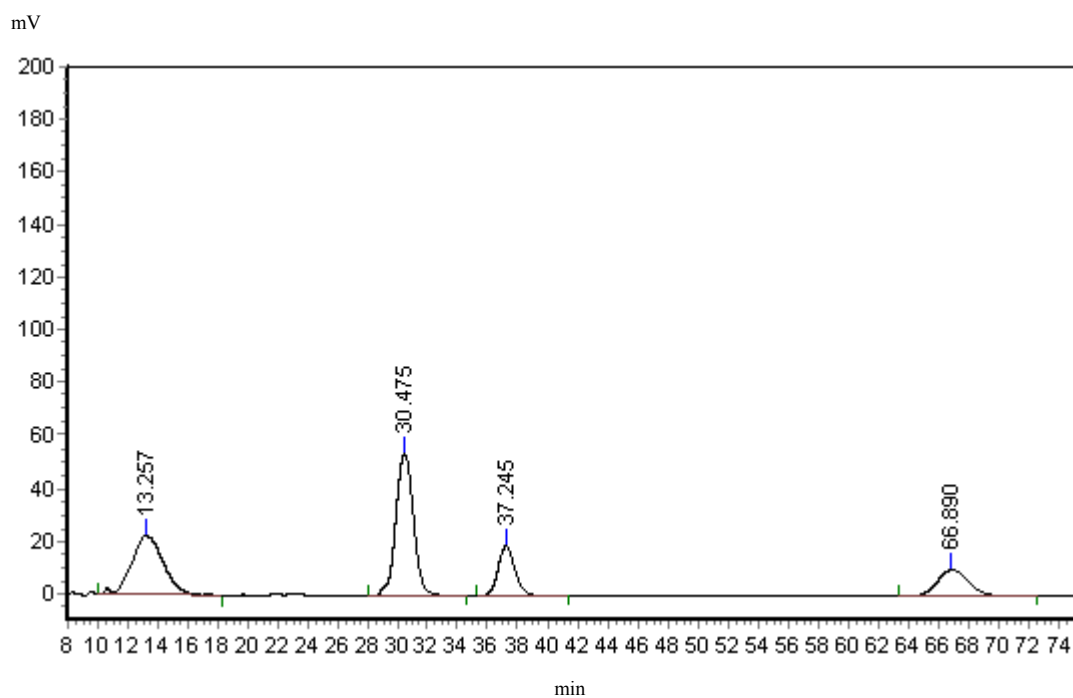
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	4.998	150850.672	1039758.813	22.5719
2	5.762	167162.016	1274317.625	27.6639
3	6.525	148394.813	1272198.500	27.6179
4	7.118	102594.703	1010974.750	21.9470

mV

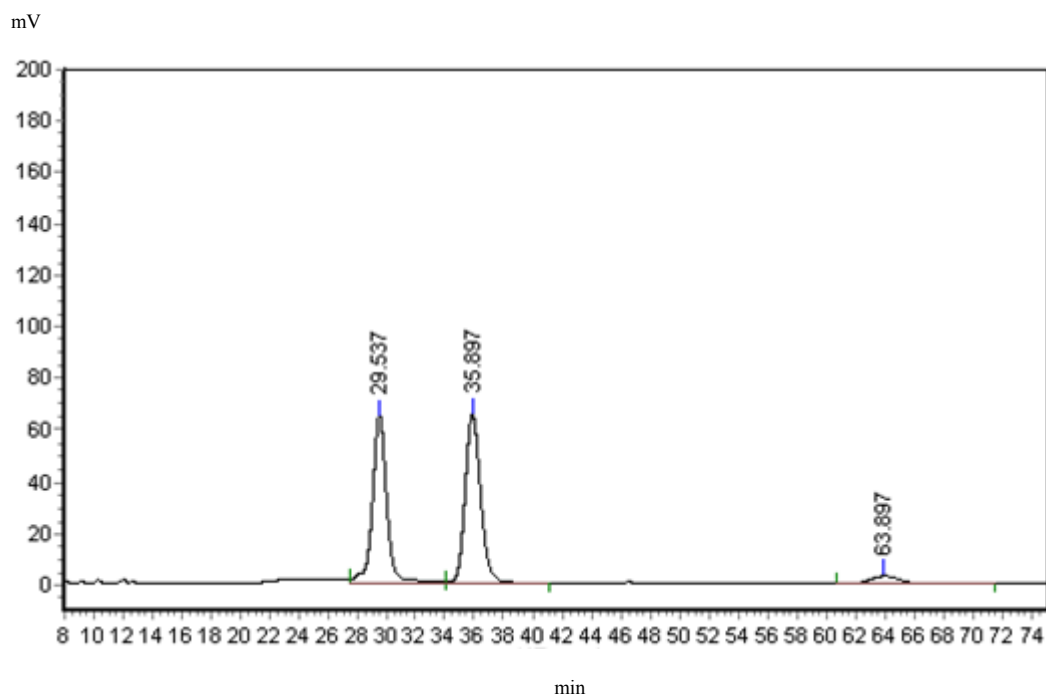


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	4.983	793119.813	5689085.500	24.8143
2	5.742	948559.563	7584803.500	33.0829
3	6.497	611976.313	5473074.500	23.8721
4	7.087	420453.281	4179677.500	18.2307

HPLC of 5a



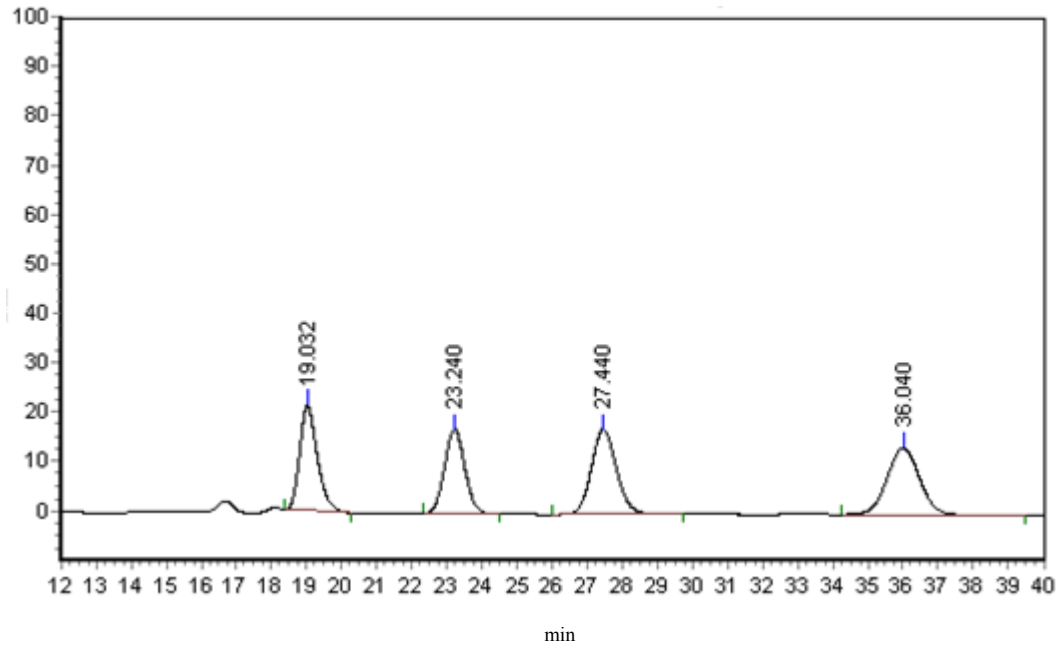
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	13.257	22426.738	3291263.250	30.7770
2	30.475	54164.758	4442871.000	41.5458
3	37.245	19012.500	1482697.750	13.8649
4	66.890	10181.925	1477084.125	13.8124



Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	29.537	65306.953	4570532.000	45.9597
2	35.897	65824.969	4949439.000	49.7698
3	63.897	2951.955	424689.719	4.2705

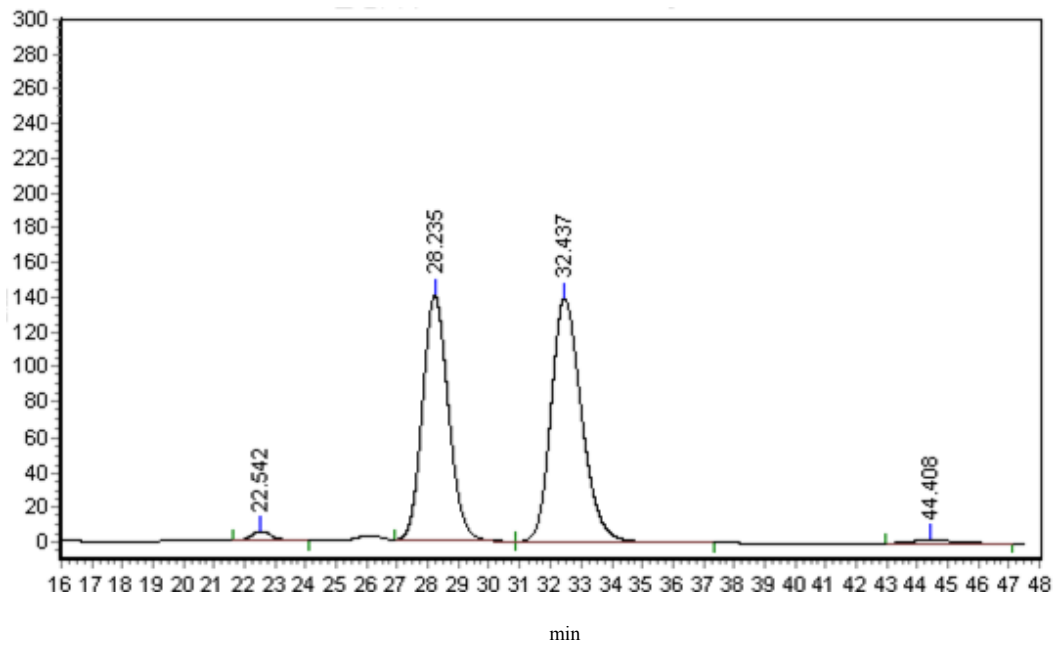
HPLC of 5b

mV



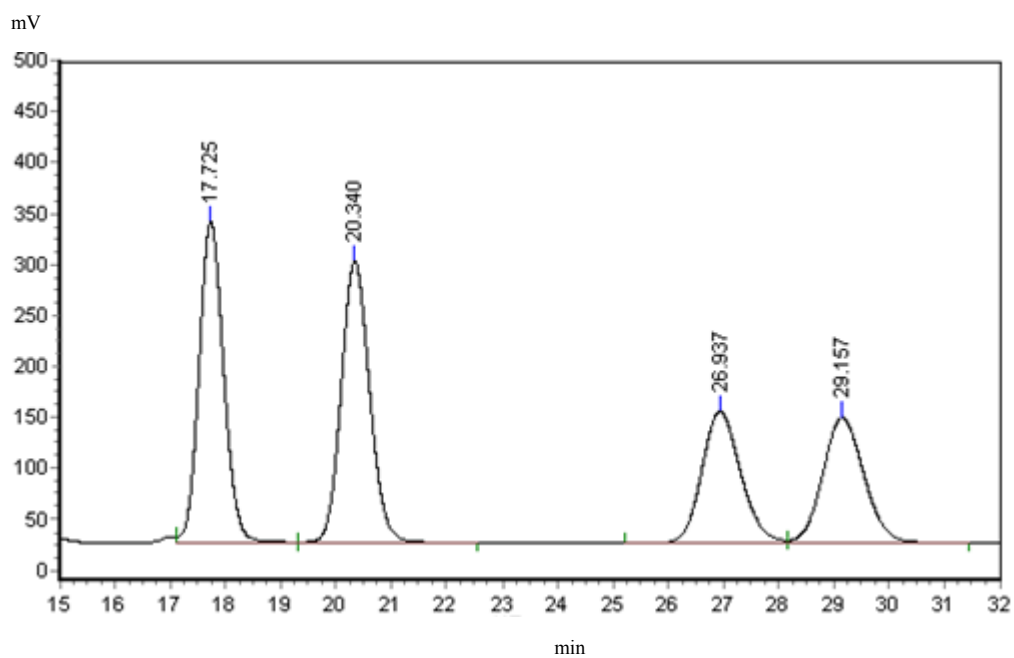
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	19.032	21143.773	738936.750	23.7678
2	23.240	16748.318	667075.375	21.4564
3	27.440	17012.141	843352.188	27.1263
4	36.040	13355.760	859622.813	27.6496

mV

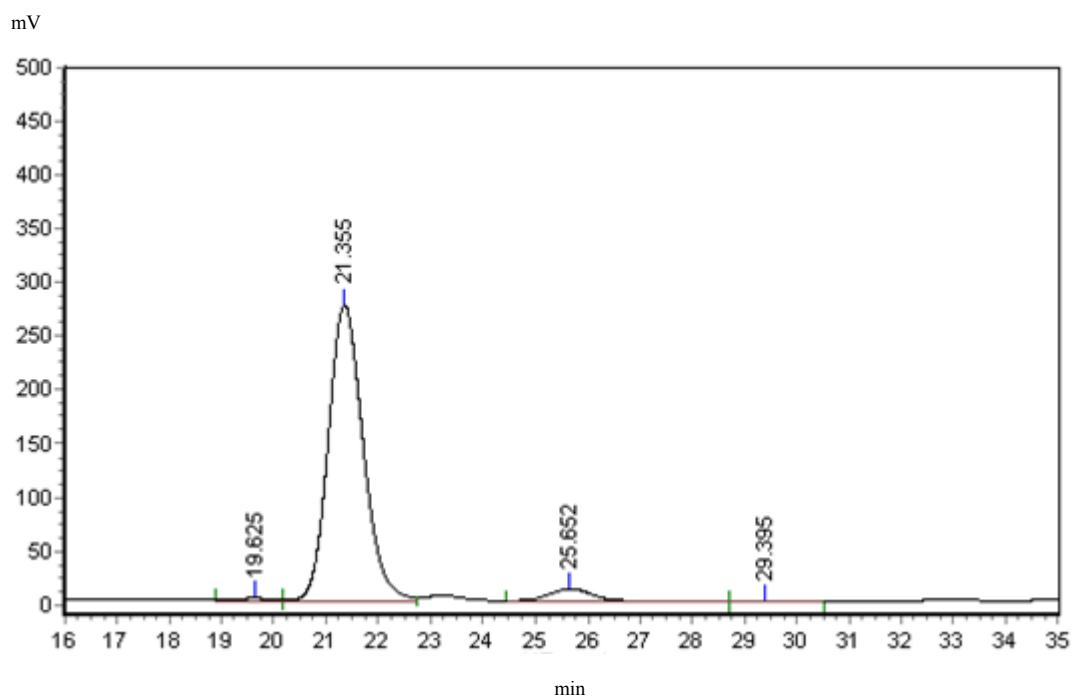


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	22.542	5459.855	263388.313	1.4285
2	28.235	140907.328	8323254.500	45.1427
3	32.437	139765.078	9652497.000	52.3521
4	44.408	1972.644	198518.500	1.0767

HPLC of 5c

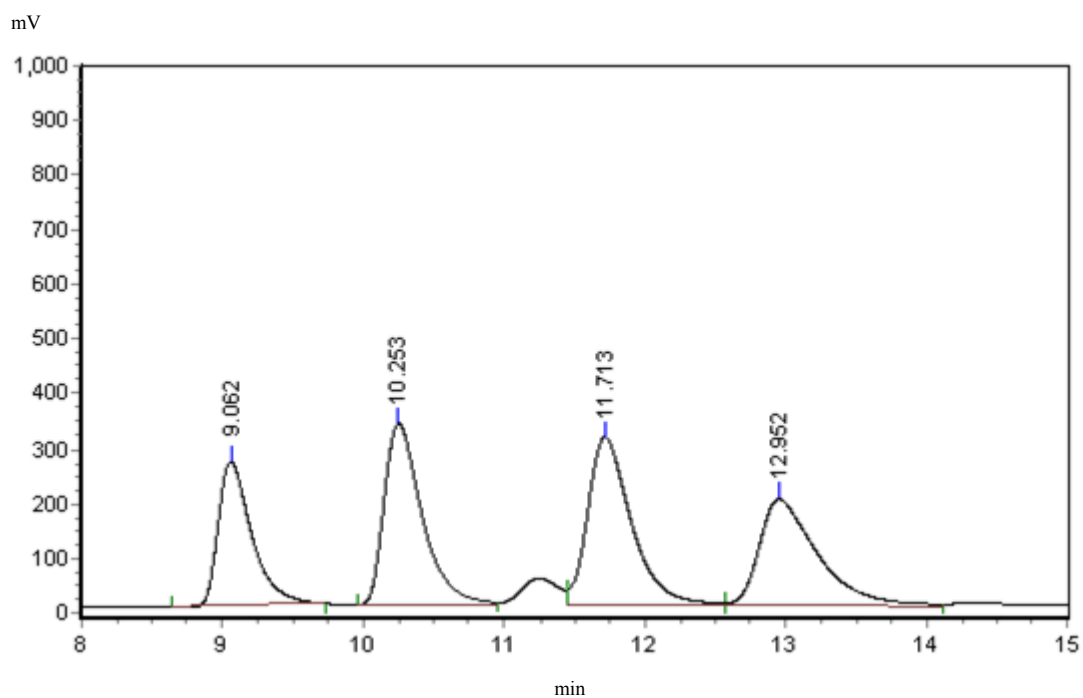


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	17.725	315418.906	9729642.000	30.2681
2	20.340	275811.125	9872428.000	30.7123
3	26.937	127705.289	6196280.500	19.2761
4	29.157	122038.148	6346519.000	19.7435

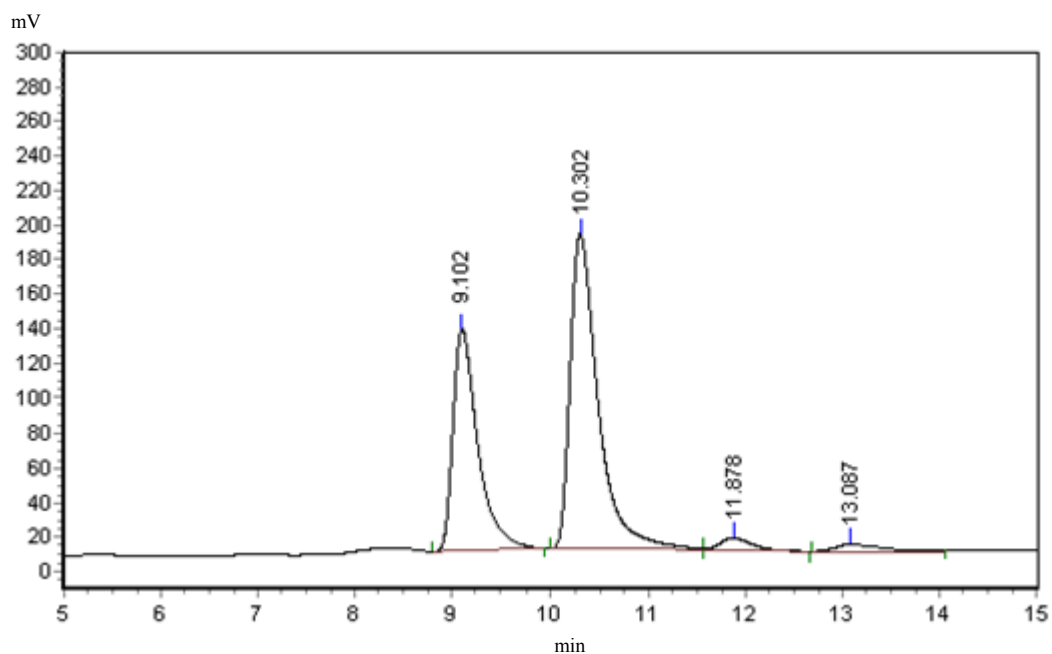


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	19.625	2955.859	99701.914	0.7379
2	21.355	275061.719	12745118.000	94.3279
3	25.652	11227.251	644778.375	4.7721
4	29.395	584.715	21909.023	0.1622

HPLC of 5d

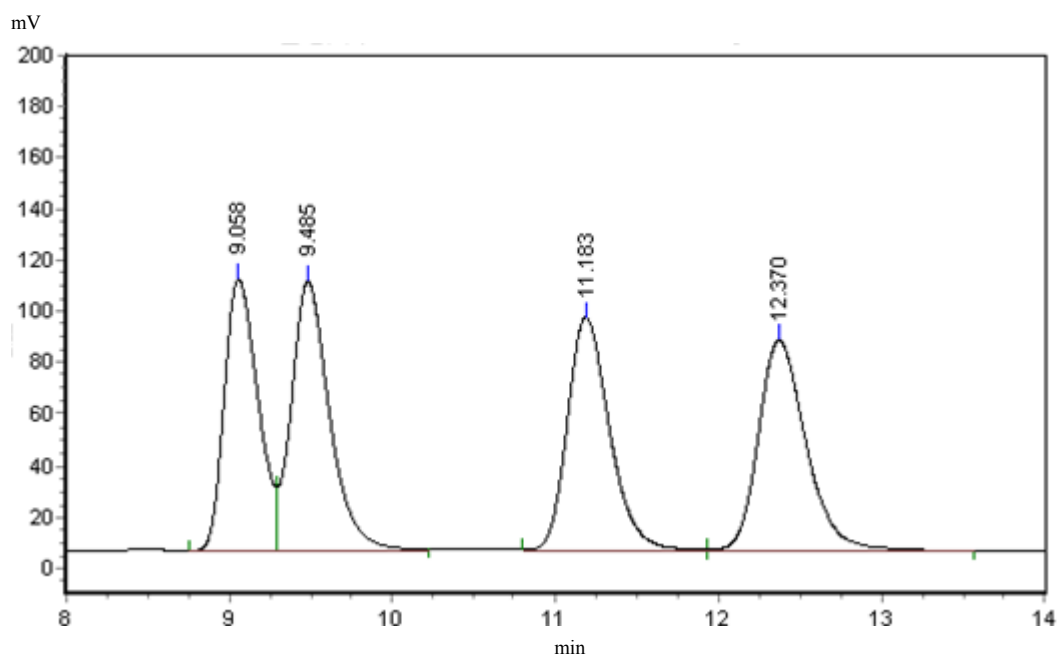


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	9.062	263193.344	4358545.500	19.0602
2	10.253	330281.500	6191629.000	27.0764
3	11.713	305050.313	6646415.500	29.0652
4	12.952	196104.734	5670702.000	24.7983

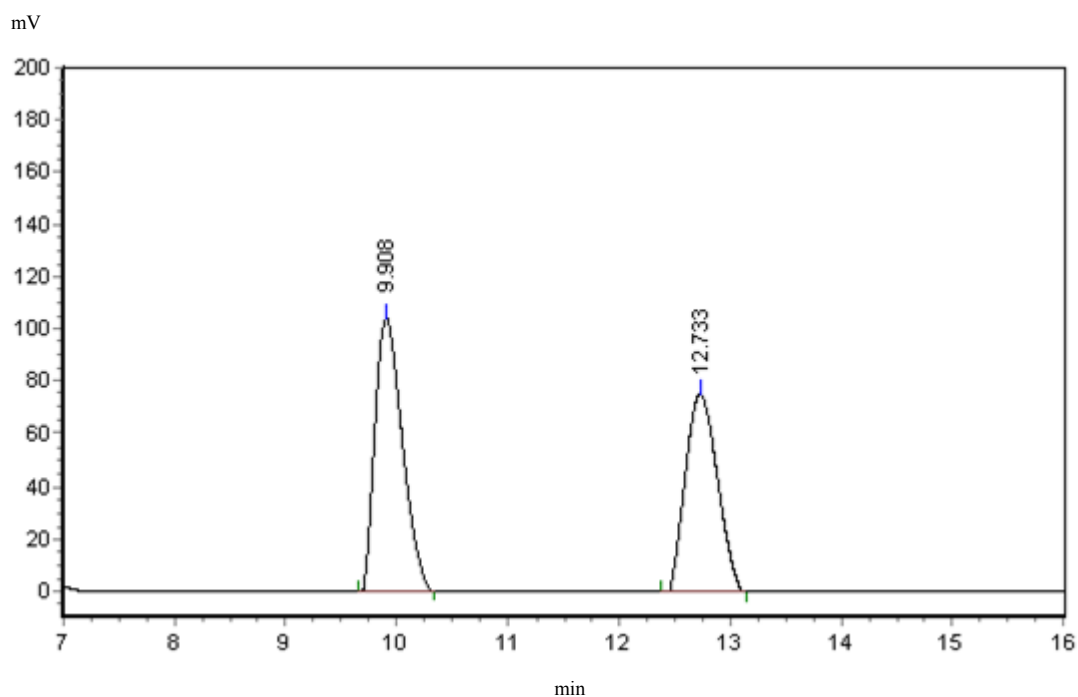


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	9.102	127964.516	2263274.250	36.1753
2	10.302	182153.156	3686440.500	58.9227
3	11.878	7326.750	176183.078	2.8160
4	13.087	3992.108	130501.336	2.0859

HPLC of 5e

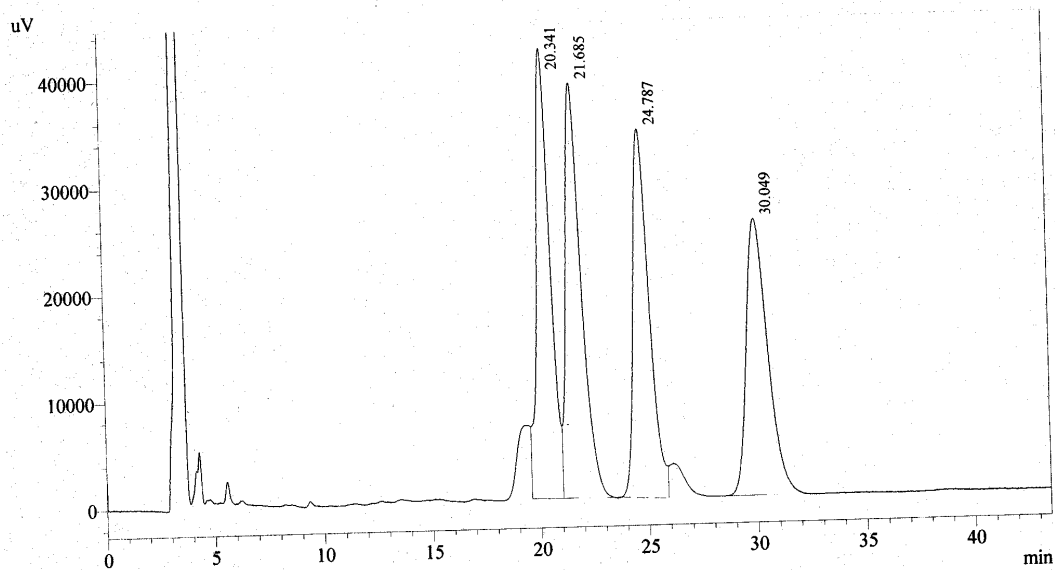


Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	9.058	105580.305	1526023.375	23.4054
2	9.485	104522.461	1703589.500	26.1288
3	11.183	90939.422	1637436.750	25.1142
4	12.370	81468.328	1652920.875	25.3517



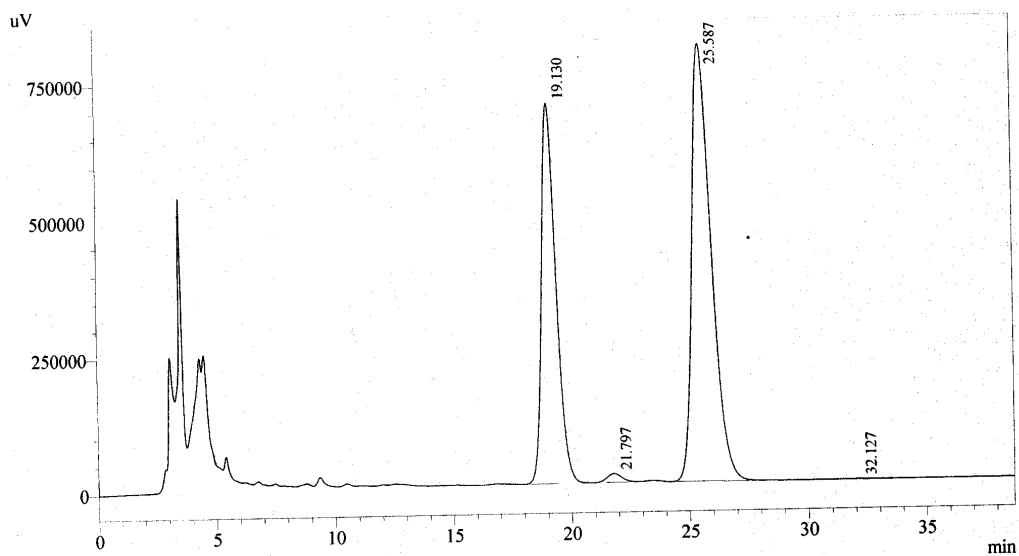
Peak	RT (min)	Height (mV* sec)	Area (mV)	Area (%)
1	9.908	104007.148	1770796.125	54.2347
2	12.733	74899.594	1494264.000	45.7653

HPLC of 5f



1 Det.A Ch1 / 215nm

Detector A Ch1 215nm					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.341	1868106	42128	25.393	29.783
2	21.685	1943418	38849	26.417	27.465
3	24.787	1782877	34581	24.234	24.448
4	30.049	1762432	25889	23.956	18.303
Total		7356832	141447	100.000	100.000

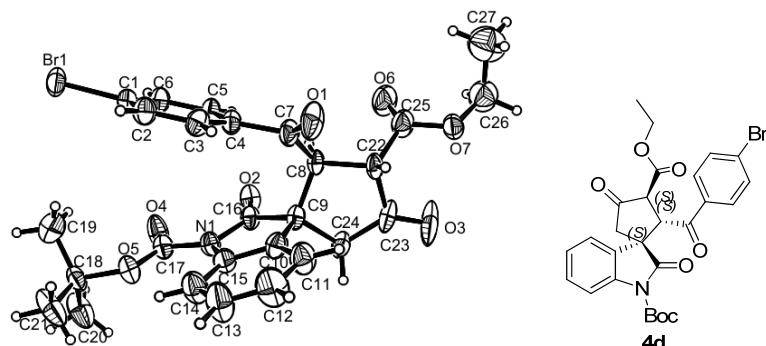


1 Det.A Ch1 / 254nm

Detector A Ch1 254nm					
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.130	29696795	697846	39.112	46.001
2	21.797	681413	15741	0.897	1.038
3	25.587	45541856	803258	59.980	52.950
4	32.127	8016	170	0.011	0.011
Total		75928080	1517015	100.000	100.000

9. Single-Crystal X-ray Crystallography of Product 4d and 5f

Single-Crystal X-ray Crystallography of Product **4d** (CDCC number: CCDC 1000582)



Bond precision: C-C = 0.0131 Å

Wavelength=0.71073

Cell: a=12.0921(5) b=12.0539(5) c=18.6826(8)
 alpha=90 beta=100.115(4) gamma=90

Temperature: 290 K

	Calculated	Reported
Volume	2680.8(2)	2680.8(2)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C27 H26 Br N O7	?
Sum formula	C27 H26 Br N O7	C54 H52 Br2 N2 O14
Mr	556.39	1112.79
Dx, g cm ⁻³	1.379	1.379
Z	4	2
Mu (mm ⁻¹)	1.578	1.578
F000	1144.0	1144.0
F000'	1143.41	
h,k,lmax	16,16,25	16,16,25
Nref	14466[7560]	12422
Tmin,Tmax	0.563,0.623	0.793,1.000
Tmin'	0.552	

Correction method= MULTI-SCAN

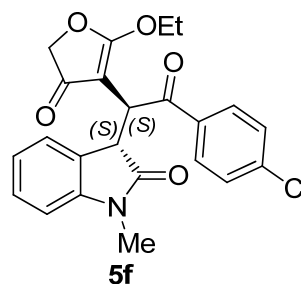
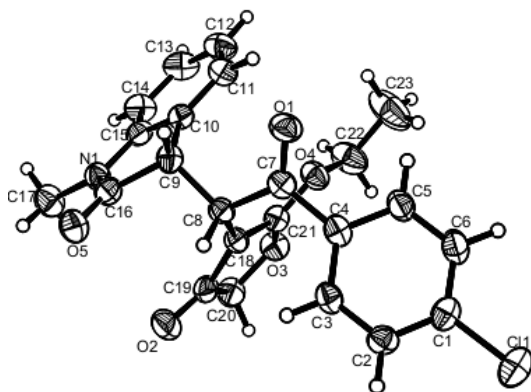
Data completeness= 1.64/0.86 Theta(max)= 29.131

R(reflections)= 0.0658(6446) wR2(reflections)= 0.1806(12422)

S = 1.002

Npar= Npar = 669

Single-Crystal X-ray Crystallography of Product **5f** (CDCC number: CCDC 999669)



Bond precision: C-C = 0.0086 Å

Wavelength=0.71073

Cell: a=10.7660(7) b=8.7896(5) c=13.4136(9)
alpha=90 beta=108.706(7) gamma=90

Temperature: 291 K

	Calculated	Reported
Volume	1202.26(14)	1202.26(14)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C ₂₃ H ₂₀ Cl N O ₅ , C ₂ H ₆ O	?
Sum formula	C ₂₅ H ₂₆ Cl N O ₆	C ₂₅ H ₂₆ Cl N O ₆
Mr	471.92	471.92
D _x , g cm ⁻³	1.304	1.304
Z	2	2
Mu (mm ⁻¹)	0.199	0.199
F ₀₀₀	496.0	496.0
F ₀₀₀ '	496.54	
h,k,l _{max}	13,10,16	13,10,16
N _{ref}	4899[2616]	4777
T _{min} ,T _{max}	0.931,0.942	0.779,1.000
T _{min} '	0.931	

Correction method= MULTI-SCAN

Data completeness= 1.83/0.98

Theta(max)= 26.369

R(reflections)= 0.0615(3268)

wR2(reflections)= 0.1532(4777)

S = 1.034

Npar= Npar = 300