

Chiral Phosphine-Silver(I) Complex Catalyzed Enantioselective Interrupted Feist-Bénary Reaction with Ynones: the Aldol-Cycloisomerization Cascade

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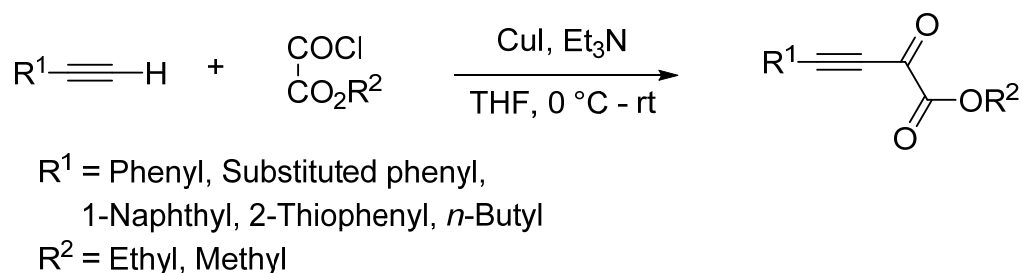
Supplementary Data

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General Experimental Information

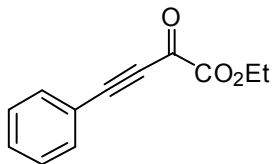
All reactions were carried out in oven-dried glassware under nitrogen atmosphere. Solvents and molecular sieves were dried following suitable protocols. Commercially available 1,3-diketones were used without further purification. All the ynones used in this study were prepared by coupling the corresponding acetylene derivatives with chlorooxoacetates, using literature methods (Scheme S1).^[1] Progress of the reactions were monitored by TLC. Analytical TLC was performed on commercially available aluminum-backed silica gel plates and the spots were visualized under UV light ($\lambda = 254$ nm). The interrupted Feist-Bénary reaction products were purified by flash column chromatography using hexane-ethyl acetate combination as the eluent on silica gel (100-200 mesh). ^1H (400 MHz), ^{13}C (100 MHz) and HMBC NMR (500 MHz) spectra were recorded at 25 °C using CDCl_3 as solvent. ^1H NMR chemical shifts are expressed in parts per million (δ) calibrated with reference to CHCl_3 ($\delta = 7.26$); ^{13}C NMR chemical shifts are expressed in parts per million (δ) relative to the central CDCl_3 resonance ($\delta = 77.0$). The enantioselectivities were determined by HPLC analyses using Chiralpak AD-H and IC columns, and are presented in terms of *ee* values.



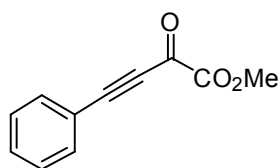
Scheme S1. Preparation of ynone substrates.^[1]

Spectral Characterization Data of Ynones (1a–k)

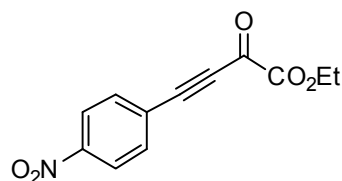
Ethyl 2-oxo-4-phenylbut-3-ynoate (1a): Yield = 60%; ^1H NMR (500 MHz, CDCl_3): δ 7.64–7.69 (m, 2H), 7.49–7.55 (m, 1H), 7.40–7.45 (m, 2H), 4.44 (q, J = 10.0 Hz, 2H), 1.45 (t, J = 5 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 169.6, 159.2, 133.8 (2C), 131.8, 128.8 (2C), 119.1, 98.0, 87.1, 63.3, 14.0.



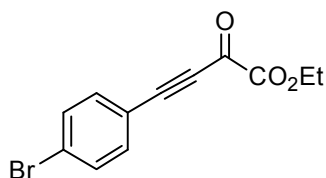
Methyl 2-oxo-4-phenylbut-3-ynoate (1b): Yield = 68%; ^1H NMR (400 MHz, CDCl_3): δ 7.64–7.69 (m, 2H), 7.49–7.56 (m, 1H), 7.39–7.46 (m, 2H), 3.96 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.1, 159.7, 133.8 (2C), 131.9, 128.8 (2C), 119.0, 98.2, 87.1, 53.7.



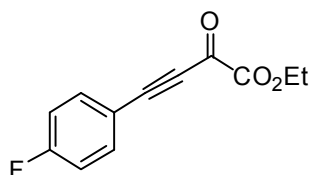
Ethyl 4-(4-nitrophenyl)-2-oxobut-3-ynoate (1c): Yield = 71%; ^1H NMR (400 MHz, CDCl_3): δ 8.28 (d, J = 8.8 Hz, 2H), 7.82 (d, J = 8.8 Hz, 2H), 4.42 (q, J = 7.2 Hz, 2H), 1.42 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.2, 158.7, 149.0, 134.3 (2C), 125.5, 123.8 (2C), 93.2, 89.2, 63.6, 13.9.



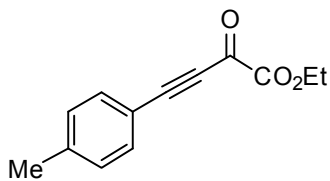
Ethyl 4-(4-bromophenyl)-2-oxobut-3-ynoate (1d): Yield = 65%; ^1H NMR (400 MHz, CDCl_3): δ 7.58 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 8.4 Hz, 2H), 4.41 (q, J = 7.2 Hz, 2H), 1.42 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.4, 159.1, 135.0 (2C), 132.3 (2C), 127.0, 118.0, 96.4, 87.8, 63.4, 14.0.



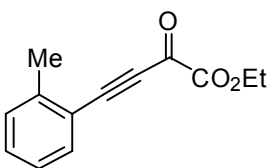
Ethyl 4-(4-fluorophenyl)-2-oxobut-3-ynoate (1e): Yield = 42%; ^1H NMR (400 MHz, CDCl_3): δ 7.64–7.71 (m, 2H), 7.08–7.16 (m, 2H), 4.40 (q, J = 7.2 Hz, 2H), 1.41 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.4, 165.9, 163.4, 159.2, 136.3 (2C), 116.4 (2C), 96.8, 87.1, 63.3, 13.9.



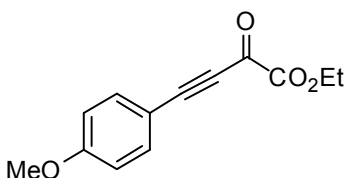
Ethyl 2-oxo-4-(*p*-tolyl)but-3-ynoate (1f): Yield = 56%; ^1H NMR (400 MHz, CDCl_3): δ 7.56 (d, $J = 8.0$ Hz, 2H), 7.22 (d, $J = 8.0$ Hz, 2H), 4.40 (q, $J = 7.2$ Hz, 2H), 2.40 (s, 3H), 1.41 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.5, 159.3, 142.9, 133.9 (2C), 129.6 (2C), 115.9, 98.9, 87.3, 63.2, 21.8, 14.0.



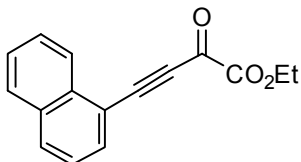
Ethyl 2-oxo-4-(*o*-tolyl)but-3-ynoate (1g): Yield = 48%; ^1H NMR (400 MHz, CDCl_3): δ 7.61 (d, $J = 7.6$ Hz, 1H), 7.41 (t, $J = 7.6$ Hz, 1H), 7.18–7.33 (m, 2H), 4.40 (q, $J = 7.2$ Hz, 2H), 2.56 (s, 3H), 1.42 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.4, 159.2, 143.7, 134.3, 131.9, 130.0, 126.0, 118.9, 97.4, 91.0, 63.2, 20.5, 14.0.



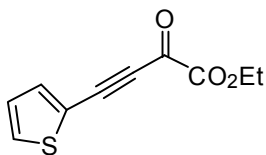
Ethyl 4-(4-methoxyphenyl)-2-oxobut-3-ynoate (1h): Yield = 65%; ^1H NMR (400 MHz, CDCl_3): δ 7.61 (d, $J = 8.8$ Hz, 2H), 6.91 (d, $J = 8.8$ Hz, 2H), 4.40 (q, $J = 4.0$ Hz, 2H), 3.86 (s, 3H), 1.41 (t, $J = 4.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.4, 162.6, 159.5, 136.1 (2C), 114.6 (2C), 110.8, 99.8, 87.8, 63.1, 55.5, 14.0.



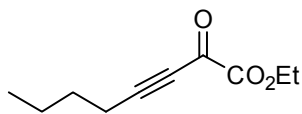
Ethyl 4-(naphthalen-1-yl)-2-oxobut-3-ynoate (1i): Yield = 48%; ^1H NMR (400 MHz, CDCl_3): δ 8.48 (d, $J = 8.0$ Hz, 1H), 8.04 (d, $J = 8.0$ Hz, 1H), 7.96 (d, $J = 8.0$ Hz, 1H), 7.91 (d, $J = 8.0$ Hz, 1H), 7.68 (t, $J = 8.0$ Hz, 1H), 7.60 (t, $J = 8.0$ Hz, 1H), 7.52 (t, $J = 8.0$ Hz, 1H), 4.48 (q, $J = 8.0$ Hz, 2H), 1.49 (t, $J = 8.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.3, 159.3, 134.6, 134.2, 133.1, 132.9, 128.6, 128.1, 127.2, 125.8, 125.2, 116.7, 96.8, 92.1, 63.4, 14.1.



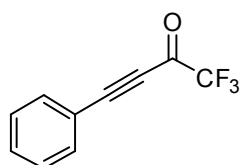
Ethyl 2-oxo-4-(thiophen-2-yl)but-3-ynoate (1j): Yield = 42%; ^1H NMR (400 MHz, CDCl_3): δ 7.61–7.63 (t, $J = 4.0$ Hz, 2H), 7.11–7.13 (dd, $J = 4.0$ Hz, 1H), 4.40 (q, $J = 8.0$ Hz, 2H), 1.41 (t, $J = 8.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 168.9, 159.1, 138.6, 133.8, 128.1, 118.9, 92.6, 92.2, 63.3, 13.9.



Ethyl 2-oxooct-3-ynoate (1k): Yield = 10%; ^1H NMR (400 MHz, CDCl_3): δ 4.35 (q, J = 7.2 Hz, 2H), 2.48 (t, J = 7.2 Hz, 2H), 1.67–1.55 (m, 2H), 1.52–1.40 (m, 2H), 1.37 (t, J = 7.2 Hz, 3H), 0.93 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.7, 159.3, 102.6, 79.7, 63.1, 29.3, 21.9, 19.1, 13.9, 13.4.



1,1,1-Trifluoro-4-phenylbut-3-yn-2-one (1l):² Yield = 51%; ^1H NMR (400 MHz, CDCl_3): δ 7.68 (dd, J = 8.4, 1.2 Hz, 2H), 7.61–7.54 (m, 1H), 7.50–7.41 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 167.2 (q, J = 41.9, 1C), 133.9 (2C), 132.5, 128.9 (2C), 118.1, 114.9 (q, J = 286.7 Hz, 1C), 100.5 (d, J = 0.6 Hz, 1C), 83.4.

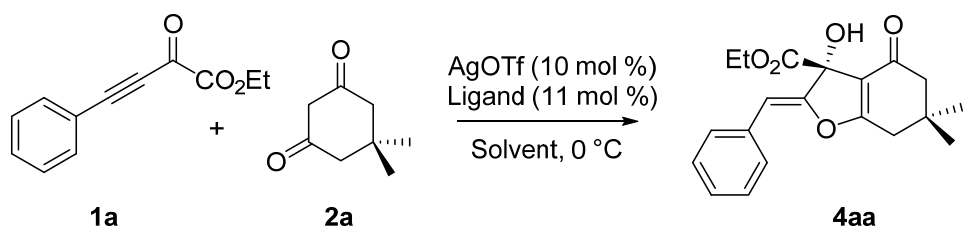


General Procedure for IFB Reaction

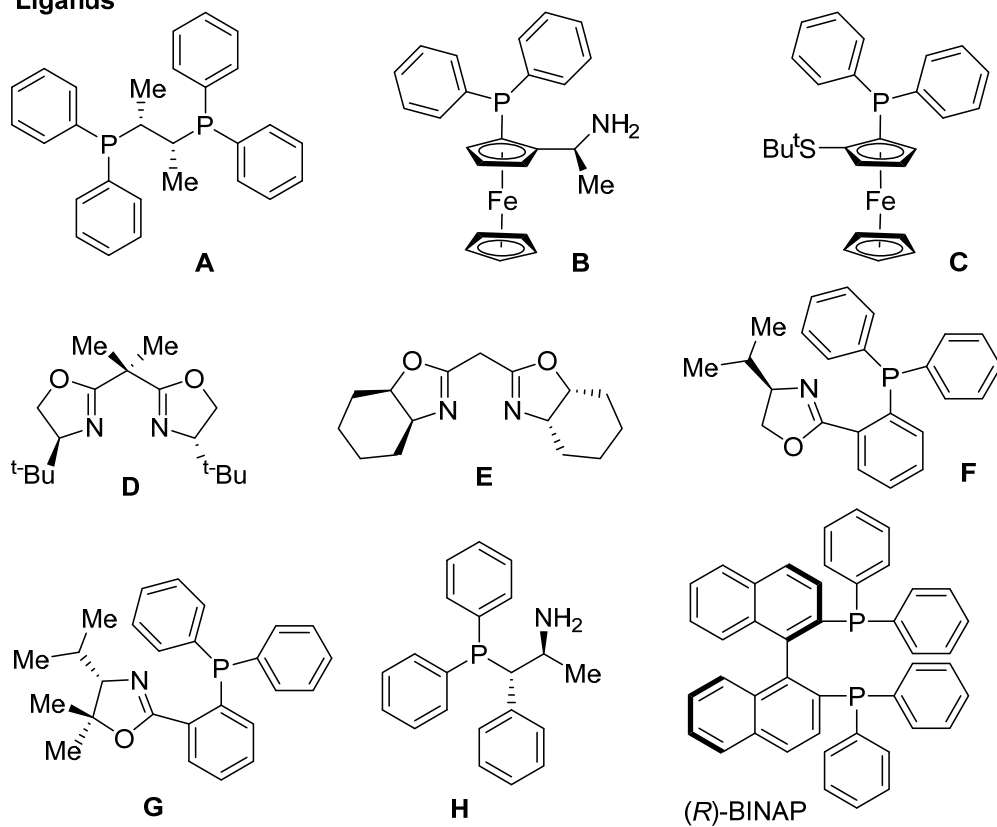
The procedure for IFB reaction between ethyl 2-oxo-4-phenylbut-3-ynoate (**1a**), and dimedone (**2a**) is described as representative.

In a round bottomed flask, was taken silver trifluoromethanesulfonate (5.1 mg, 0.02 mmol, 10 mol %) and (*R*)-BINAP (6.2 mg, 0.01 mmol, 5 mol %) in dichloromethane (1.0 mL), and the mixture was stirred at room temperature (25 °C) for 30 minutes under nitrogen atmosphere. Afterwards, this mixture was added slowly to a stirring suspension of ethyl 2-oxo-4-phenylbut-3-ynoate (**1a**, 40.4 mg, 0.20 mmol, 1.0 equiv.), dimedone (**2a**, 56.1 mg, 0.40 mmol, 2.0 equiv.), and MS 4 Å (50 mg) in dichloromethane (1.0 mL) at -60 °C. Reaction was continued at the aforesaid temperature, and progress was monitored by TLC. Upon completion, the reaction mixture was allowed to attain room temperature, and charged over to a column packed with silica gel. The dihydrofuran product **4aa** was isolated by flash column chromatography using ethyl acetate and hexane as eluent, and finally characterized by spectral analysis (48.0 mg, 0.14 mmol, 70% yield).

Table S1: Additional screening of IFB reaction with ligands, solvents and additives.^a



Ligands



entry	ligand	solvent	time (h)	yield (%) ^b	ee (%) ^c
1	A	CH ₂ Cl ₂	3.0	91	0
2	B	CH ₂ Cl ₂	1.0	92	14
3	C	CH ₂ Cl ₂	4.0	89	-3
4	D	CH ₂ Cl ₂	1.0	93	-2

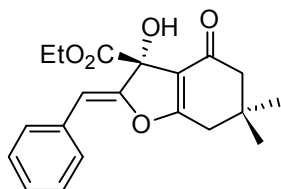
Table contd...

5	E	CH ₂ Cl ₂	1.0	91	2
6	F	CH ₂ Cl ₂	1.0	78	9
7	G	CH ₂ Cl ₂	4.0	80	-4
8	H	CH ₂ Cl ₂	1.0	89	4
9	(<i>R</i>)-BINAP	CH ₂ Cl ₂ /MeOH (10:01)	2.0	61	60
10	(<i>R</i>)-BINAP	Cl(CH ₂) ₂ Cl	8.0	71	68
11	(<i>R</i>)-BINAP	CHCl ₃	2.0	82	54
12	(<i>R</i>)-BINAP	THF	1.0	72	32
13	(<i>R</i>)-BINAP	CH ₃ CN	12.0	79	2
14	(<i>R</i>)-BINAP	DMF	2.0	90	15
15	(<i>R</i>)-BINAP	AcOEt	3.0	92	47
16	(<i>R</i>)-BINAP	PhCH ₃	12.0	90	46
17 ^d	(<i>R</i>)-BINAP	CH ₂ Cl ₂	4.0	89	60
18 ^e	(<i>R</i>)-BINAP	CH ₂ Cl ₂	0.5	90	0

^aUnless otherwise mentioned all the reactions were carried out with 0.20 mmol ynone **1a**, 0.40 mmol dimedone (**2a**), 10 mol % silver triflate, and 11 mol % chiral ligand at 0 °C. ^bIsolated yield of dihydrofuran **4aa**. ^cEnantiomeric excess of the product **4aa** was determined by chiral HPLC, using AD-H column. Negative *ee* values represent the (*S*)-enantiomer to be major. ^dReaction was conducted with 10 mol % benzoic acid additive. ^eReaction was conducted with 10 mol % triethylamine additive.

Characterization Data for Dihydrofuran and Related Compounds

Ethyl (*R,Z*)-2-benzylidene-3-hydroxy-6,6-dimethyl-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-

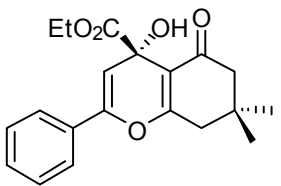


3-carboxylate (4aa): Yield = 70%; White solid, mp = 144–145 °C; ¹H

NMR (400 MHz, CDCl₃): δ 7.63–7.54 (m, 2H), 7.39–7.31 (m, 2H), 7.30–7.22 (m, 1H), 5.90 (s, 1H), 4.37 (s, 1H), 4.37–4.27 (m, 1H), 4.23–4.12 (m, 1H), 2.69 (d, *J* = 18.0 Hz, 1H), 2.54 (dd, *J* = 18.0, 1.2 Hz, 1H), 2.40 (d, *J* = 16.0 Hz, 1H), 2.24 (dd, *J* = 16.0, 1.2 Hz, 1H), 1.22 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H), 1.14 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ

192.7, 176.0, 171.3, 154.8, 132.9, 129.2 (2C), 128.5 (2C), 127.8, 116.0, 107.5, 78.7, 63.2, 51.1, 37.4, 35.0, 29.5, 27.4, 13.9; FTIR ν_{max} (NaCl film, cm⁻¹): 3015, 2918, 1733, 1695, 1651, 1540, 1395, 1025; HRMS (ESI) calcd. for C₂₀H₂₁O₄ [M - OH]⁺: 325.1440, found 325.1467; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (80/20 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 17.8 min (major) and 27.4 min (minor), 90% ee. After first recrystallization, ee of mother liquor >99%; [α]_D²⁵ = +71.3 (*c* = 1.0, CH₂Cl₂).

Ethyl (*S*)-4-hydroxy-7,7-dimethyl-5-oxo-2-phenyl-5,6,7,8-tetrahydro-4*H*-chromene-4-

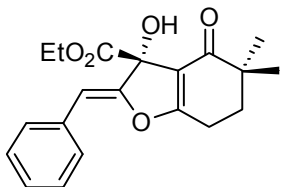


carboxylate (5aa): Yield = 26%; Colorless gummy liquid; ¹H NMR (400

MHz, CDCl₃): δ 7.66–7.58 (m, 2H), 7.45–7.35 (m, 3H), 5.67 (s, 1H), 4.44 (s, 1H), 4.34–4.22 (m, 1H), 4.22–4.09 (m, 1H), 2.67 (d, *J* = 17.6 Hz, 1H), 2.55 (d, *J* = 17.6 Hz, 1H), 2.43–2.26 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.17 (s, 3H), 1.16 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 198.4, 173.6, 165.4, 150.3, 131.9, 129.7, 128.6 (2C), 125.1 (2C), 112.5, 101.2,

66.9, 62.5, 50.8, 41.2, 32.6, 29.0, 27.3, 14.1; FTIR ν_{max} (NaCl film, cm⁻¹): 3152, 3077, 2980, 1740, 1732, 1681, 1383, 1285, 1230, 1056; HRMS (ESI) calcd. for C₂₀H₂₃O₅ [M + H]⁺: 343.1545, found 343.1528.

Ethyl (*R,Z*)-2-benzylidene-3-hydroxy-7,7-dimethyl-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-



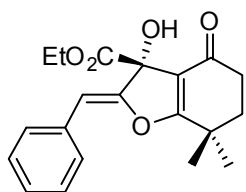
3-carboxylate (4ab): Yield = 51%; Colorless gummy liquid; ¹H NMR

(400 MHz, CDCl₃): δ 7.59 (d, *J* = 7.2 Hz, 2H), 7.35 (*pseudo*-t, *J* = 7.2 Hz, 2H), 7.29–7.23 (m, 1H), 5.89 (s, 1H), 4.37 (s, 1H), 4.36–4.27 (m, 1H), 4.20–4.10 (m, 1H), 2.83–2.68 (m, 2H), 2.12–1.99 (m, 1H), 1.94 (dt, *J* = 13.6, 5.6 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H), 1.18 (s, 3H), 1.14 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 198.2, 175.0, 171.2, 154.9, 133.0, 129.1

(2C), 128.5 (2C), 127.8, 115.7, 107.4, 79.0, 63.0, 41.3, 35.1, 24.0, 23.8, 21.2, 13.9; FTIR ν_{max} (NaCl film, cm⁻¹): 3609, 2918, 2851, 1731, 1693, 1556, 1536, 1391; HRMS (ESI) calcd. for C₂₀H₂₁O₄ [M - OH]⁺: 325.1440, found 325.1477; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length

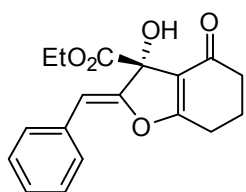
UV, Retention time = 10.7 min (major) and 25.1 min (minor), 92% ee; $[\alpha]_D^{25} = +67.1$ ($c = 1.0$, CH_2Cl_2).

Ethyl (R,Z)-2-benzylidene-3-hydroxy-5,5-dimethyl-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-



3-carboxylate (4ab'): Yield = 26%; Colorless gummy liquid; ^1H NMR (400 MHz, CDCl_3): δ 7.58 (d, $J = 7.2$ Hz, 2H), 7.37 (*pseudo*-t, $J = 7.6$ Hz, 2H), 7.30–7.23 (m, 1H), 5.89 (s, 1H), 4.35 (s, 1H), 4.35–4.24 (m, 1H), 4.24–4.13 (m, 1H), 2.57–2.39 (m, 2H), 2.09–1.99 (m, 1H), 1.99–1.89 (m, 1H), 1.44 (s, 3H), 1.40 (s, 3H), 1.21 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.0, 182.3, 171.2, 154.5, 133.2, 129.1 (2C), 128.6 (2C), 127.8, 115.1, 107.4, 79.2, 63.1, 36.6, 34.4, 32.9, 24.82, 24.76, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2963, 2914, 2848, 1730, 1695, 1537, 1395, 989; HRMS (ESI) calcd. for $\text{C}_{20}\text{H}_{22}\text{NaO}_5$ $[\text{M} + \text{Na}]^+$: 365.1365, found 365.1387; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 8.6 min (major) and 13.3 min (minor), 56% ee; $[\alpha]_D^{25} = +37.7$ ($c = 1.0$, CH_2Cl_2).

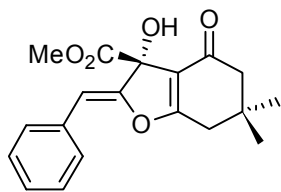
Ethyl (R,Z)-2-benzylidene-3-hydroxy-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate



(4ac): Yield = 82%; White solid, mp = 131 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, $J = 7.6$ Hz, 2H), 7.39–7.31 (m, 2H), 7.29–7.22 (m, 1H), 5.89 (s, 1H), 4.42 (s, 1H), 4.33–4.15 (m, 2H), 2.82–2.63 (m, 2H), 2.52–2.31 (m, 2H), 2.24–2.10 (m, 2H), 1.21 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): 193.1, 176.9, 171.2, 154.6, 132.9, 129.1, 128.5, 127.8, 117.3, 107.5, 78.7, 63.2, 36.5, 23.5, 21.3, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2846, 1729, 1695, 1650, 1559, 1538, 1392; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{17}\text{O}_4$ $[\text{M} - \text{OH}]^+$: 297.1127, found 297.1154; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 12.5 min (major) and 20.2 min (minor), 95% ee. After first recrystallization, ee of crystal >99%; $[\alpha]_D^{25} = +70.3$ ($c = 1.0$, CH_2Cl_2).

Methyl

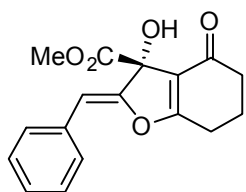
(R,Z)-2-benzylidene-3-hydroxy-6,6-dimethyl-4-oxo-2,3,4,5,6,7-



hexahydrobenzofuran-3-carboxylate (4ba): Yield = 70%; White solid, mp = 146–147 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, $J = 7.6$ Hz, 2H), 7.36 (*pseudo*-t, $J = 7.2$ Hz, 2H), 7.30–7.23 (m, 1H), 5.91 (s, 1H), 4.29 (s, 1H), 3.79 (s, 3H), 2.67 (d, $J = 17.6$ Hz, 1H), 2.55 (dd, $J = 17.6$, 4.0 Hz, 1H), 2.38 (d, $J = 16.0$ Hz, 1H), 2.27 (dd, $J = 16.0$, 4.0 Hz, 1H), 1.21 (s, 3H), 1.15 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 192.8, 176.1, 171.8, 154.6, 132.8, 129.2 (2C), 128.5 (2C), 127.9, 115.9, 107.7, 78.7, 54.0, 51.0, 37.4, 34.8, 29.2, 27.8; FTIR ν_{max} (NaCl film, cm^{-1}): 3021, 2848, 1733, 1652, 1555, 1535, 1399, 1257, 1222; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{O}_4$ $[\text{M} - \text{OH}]^+$: 311.1283, found 311.1321; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min

flow rate), 254 nm wave length UV, Retention time = 11.6 min (major) and 19.5 min (minor), 87% *ee*. After first recrystallization, *ee* of mother liquor >99%; $[\alpha]_D^{25} = +38.2$ ($c = 1.0$, CH_2Cl_2).

Methyl

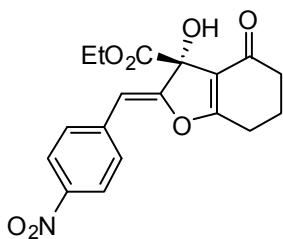


(*R,Z*)-2-benzylidene-3-hydroxy-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-

carboxylate (**4bc**):

Yield = 73%; White solid, mp = 168–170 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.59 (d, $J = 7.6$ Hz, 2H), 7.39–7.31 (m, 2H), 7.29–7.22 (m, 1H), 5.90 (s, 1H), 4.34 (s, 1H), 3.79 (s, 3H), 2.82–2.66 (m, 2H), 2.53–2.34 (m, 2H), 2.24–2.12 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.2, 177.0, 171.7, 154.4, 132.8, 129.2 (2C), 128.5 (2C), 127.9, 117.2, 107.7, 78.7, 54.0, 36.5, 23.5, 21.3; FTIR ν_{max} (NaCl film, cm^{-1}): 3018, 2918, 1733, 1695, 1651, 1540, 1397, 994; HRMS (ESI) calcd. for $\text{C}_{17}\text{H}_{15}\text{O}_4$ $[\text{M} - \text{OH}]^+$: 283.0970, found 283.0975; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 11.2 min (major) and 18.9 min (minor), 91% *ee*. After first recrystallization, *ee* of crystal >99%; $[\alpha]_D^{25} = +48.1$ ($c = 1.0$, CH_2Cl_2).

Ethyl

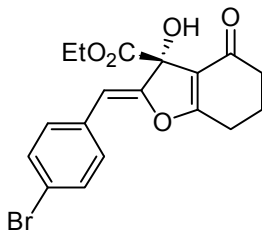


(*R,Z*)-3-hydroxy-2-(4-nitrobenzylidene)-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-

carboxylate (**4cc**):

Yield = 95%; White solid, mp = 114 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.20 (d, $J = 8.8$ Hz, 2H), 7.73 (d, $J = 8.8$ Hz, 2H), 5.95 (s, 1H), 4.47 (s, 1H), 4.37–4.18 (m, 2H), 2.87–2.68 (m, 2H), 2.55–2.35 (m, 2H), 2.28–2.12 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 192.9, 176.6, 170.6, 157.7, 146.6, 139.5, 129.6 (2C), 123.8 (2C), 117.6, 105.2, 79.0, 63.5, 36.5, 23.4, 21.3, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2918, 1733, 1697, 1537, 1397, 998; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{16}\text{NO}_6$ $[\text{M} - \text{OH}]^+$: 342.0978, found 342.0997; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 13.2 min (major) and 21.9 min (minor), 71% *ee*. After first recrystallization, *ee* of mother liquor = 99%; $[\alpha]_D^{25} = +89.6$ ($c = 1.0$, CH_2Cl_2).

Ethyl



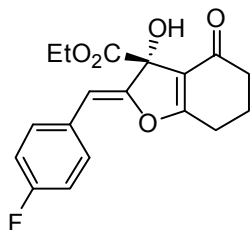
(*R,Z*)-2-(4-bromobenzylidene)-3-hydroxy-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-

carboxylate (**4dc**):

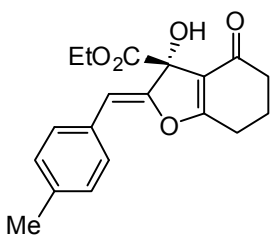
Yield = 88%; White solid, mp = 106 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.56–7.33 (m, 4H), 5.82 (s, 1H), 4.36 (s, 1H), 4.36–4.15 (m, 2H), 2.85–2.65 (m, 2H), 2.54–2.32 (m, 2H), 2.28–2.08 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.0, 176.7, 171.0, 155.2, 131.9, 131.7 (2C), 130.6 (2C), 121.8, 117.4, 106.4, 78.8, 63.3, 36.6, 23.5, 21.3, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2934, 2918, 1733, 1695, 1537, 1395; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{16}\text{BrO}_4$ $[\text{M} - \text{OH}]^+$: 375.0232, found 375.0237; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention

time = 10.5 min (major) and 20.9 min (minor), 79% ee. After first recrystallization, ee of crystal >99%; $[\alpha]_D^{25} = +71.6$ ($c = 1.0$, CH_2Cl_2).

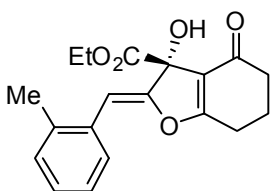
Ethyl (R,Z)-2-(4-fluorobenzylidene)-3-hydroxy-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4ec): Yield = 82%; White solid, mp = 110 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.56 (dd, $J = 8.8, 5.6$ Hz, 2H), 7.03 (pseudo-t, $J = 8.8$ Hz, 2H), 5.85 (s, 1H), 4.40 (s, 1H), 4.35–4.16 (m, 2H), 2.82–2.66 (m, 2H), 2.52–2.33 (m, 2H), 2.26–2.08 (m, 2H), 1.21 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.0, 176.7, 171.1, 163.3, 160.8, 154.2 (d, $J = 2.6$ Hz, 1C), 130.9 (d, $J = 8.1$ Hz, 1C), 129.1 (d, $J = 3.4$ Hz, 1C), 117.4, 115.6, 115.4, 106.4, 78.7, 63.2, 36.5, 23.5, 21.3, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2918, 1730, 1697, 1540, 1393, 992; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{16}\text{FO}_4$ $[\text{M} - \text{OH}]^+$: 315.1033, found 315.1069; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 10.9 min (major) and 19.9 min (minor), 85% ee; $[\alpha]_D^{25} = +57.7$ ($c = 1.0$, CH_2Cl_2).



Ethyl (R,Z)-3-hydroxy-2-(4-methylbenzylidene)-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4fc): Yield = 66%; White solid, mp = 128–129 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.48 (d, $J = 8.0$ Hz, 2H), 7.16 (d, $J = 8.0$ Hz, 2H), 5.86 (s, 1H), 4.34 (s, 1H), 4.34–4.17 (m, 2H), 2.82–2.66 (m, 2H), 2.52–2.35 (m, 2H), 2.35 (s, 3H), 2.26–2.10 (m, 2H), 1.21 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.1, 176.9, 171.2, 154.0, 137.8, 130.1, 129.2 (2C), 129.1 (2C), 117.3, 107.5, 78.6, 63.1, 36.6, 23.5, 21.32, 21.27, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2918, 2848, 1730, 1665, 1651, 1564, 1537, 1397, 1246; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{O}_4$ $[\text{M} - \text{OH}]^+$: 311.1283, found 311.1314; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 10.1 min (major) and 23.0 min (minor), 93% ee. After first recrystallization, ee of mother liquor >99%; $[\alpha]_D^{25} = +76.3$ ($c = 1.0$, CH_2Cl_2).

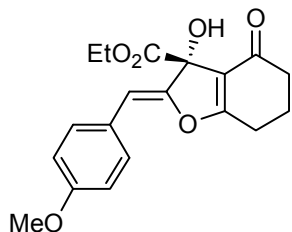


Ethyl (R,Z)-3-hydroxy-2-(2-methylbenzylidene)-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4gc): Yield = 40%; White solid, mp = 116° C; ^1H NMR (400 MHz, CDCl_3): δ 7.76 (d, $J = 7.2$ Hz, 1H), 7.25–7.13 (m, 3H), 6.06 (s, 1H), 4.41 (s, 1H), 4.28 (q, $J = 7.2$ Hz, 2H), 2.79–2.61 (m, 2H), 2.53–2.34 (m, 2H), 2.31 (s, 3H), 2.25–2.08 (m, 2H), 1.24 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.1, 177.1, 171.3, 155.0, 136.3, 131.4, 130.2, 129.3, 127.9, 125.9, 117.4, 105.0, 78.5, 63.1, 36.6, 23.5, 21.3, 20.0, 14.0; FTIR ν_{max} (NaCl film, cm^{-1}): 2971, 2844, 1733, 1652, 1395, 1242, 994; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{19}\text{O}_4$ $[\text{M} - \text{OH}]^+$: 311.1283, found 311.1295; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm

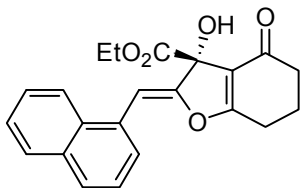


wave length UV, Retention time = 12.3 min (major) and 24.2 min (minor), 98% *ee*. After first recrystallization, *ee* of crystal >99%; $[\alpha]_D^{25} = +93.2$ ($c = 1.0$, CH_2Cl_2).

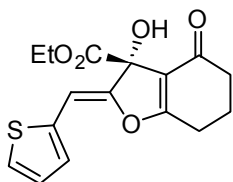
Ethyl (R,Z)-3-hydroxy-2-(4-methoxybenzylidene)-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4hc): Yield = 42%; White solid, mp = 144–145 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.54 (dd, $J = 6.8, 2.0$ Hz, 2H), 6.89 (dd, $J = 6.8, 2.0$ Hz, 2H), 5.84 (s, 1H), 4.33 (s, 1H), 4.35–4.16 (m, 2H), 3.82 (s, 3H), 2.83–2.65 (m, 2H), 2.54–2.32 (m, 2H), 2.27–2.10 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.1, 176.8, 171.3, 159.2, 153.1, 130.6 (2C), 125.7, 117.3, 114.0 (2C), 107.3, 78.6, 63.1, 55.3, 36.6, 23.6, 21.4, 14.0; FTIR ν_{max} (NaCl film, cm^{-1}): 2918, 1733, 1697, 1557, 1540, 1395; HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{20}\text{NaO}_6$ $[\text{M} + \text{Na}]^+$: 367.1158, found 367.1163; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 15.5 min (major) and 36.5 min (minor), 96% *ee*. After first recrystallization, *ee* of crystal >99%; $[\alpha]_D^{25} = +39.2$ ($c = 0.5$, CH_2Cl_2).



Ethyl (R,Z)-3-hydroxy-2-(naphthalen-1-ylmethylene)-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4ic): Yield = 75%; White solid, mp = 107–108 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 7.6$ Hz, 1H), 7.90 (d, $J = 7.2$ Hz, 1H), 7.85 (dd, 7.2, 2.0 Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.58–7.42 (m, 3H), 6.62 (s, 1H), 4.53 (s, 1H), 4.40–4.25 (m, 2H), 2.77–2.61 (m, 2H), 2.55–2.35 (m, 2H), 2.25–2.08 (m, 2H), 1.27 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.1, 177.2, 171.3, 156.0, 133.5, 131.2, 128.9, 128.6, 128.4, 127.5, 126.4, 125.9, 125.3, 123.6, 117.5, 104.1, 78.6, 63.2, 36.6, 23.5, 21.3, 14.0; FTIR ν_{max} (NaCl film, cm^{-1}): 3090, 3027, 2855, 1608, 1508, 1463, 1030, 757, 727, 698; HRMS (ESI) calcd. for $\text{C}_{22}\text{H}_{20}\text{NaO}_5$ $[\text{M} + \text{Na}]^+$: 387.1208, found 387.1226; HPLC condition: CHIRALPAK-IC (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (70/30 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 26.5 min (minor) and 29.3 min (major), 96% *ee*. After first recrystallization, *ee* of mother liquor = 97%; $[\alpha]_D^{25} = +79.2$ ($c = 1.0$, CH_2Cl_2).

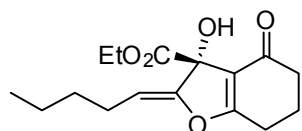


Ethyl (R,Z)-3-hydroxy-4-oxo-2-(thiophen-2-ylmethylene)-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (4jc): Yield = 85%; White solid, mp = 130–132 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.34 (d, $J = 5.2$ Hz, 1H), 7.15 (d, $J = 3.2$ Hz, 1H), 7.02 (dd, $J = 5.2, 3.6$ Hz, 1H), 6.19 (s, 1H), 4.35 (s, 1H), 4.35–4.25 (m, 1H), 4.25–4.16 (m, 1H), 2.84–2.67 (m, 2H), 2.53–2.32 (m, 2H), 2.26–2.10 (m, 2H), 1.22 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 193.1, 176.6, 171.0, 152.8, 135.5, 128.2, 127.2, 127.0, 117.6, 101.7, 78.2, 63.2, 36.6, 23.5, 21.3, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 3086, 3069, 3024, 2917, 1741, 1640, 1601, 1496, 1452,



1185, 1053, 992, 757; HRMS (ESI) calcd. for $C_{16}H_{15}O_4S$ $[M - OH]^+$: 303.0691, found 303.0687; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 10.1 min (major) and 16.9 min (minor), 80% *ee*. After first recrystallization, *ee* of crystal >99%; $[\alpha]_D^{25} = +68.7$ ($c = 1.0$, CH_2Cl_2).

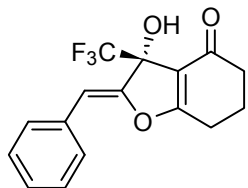
Ethyl (R,Z)-3-hydroxy-4-oxo-2-pentylidene-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate



(4kc): Yield = 64%; Colorless gummy liquid; 1H NMR (400 MHz, $CDCl_3$): δ 5.03 (t, $J = 7.6$ Hz, 1H), 4.29–4.17 (m, 3H), 2.71–2.55 (m, 2H), 2.48–2.29 (m, 2H), 2.28–2.17 (m, 2H), 2.17–2.05 (m, 2H), 1.45–1.25 (m, 4H), 1.22 (t, $J = 7.2$ Hz, 3H), 0.89 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.1, 176.9, 171.5, 155.3, 117.4, 109.1,

77.1, 62.9, 36.5, 31.0, 24.8, 23.5, 22.1, 21.3, 13.9, 13.8; FTIR ν_{max} (NaCl film, cm^{-1}): 3088, 3033, 2998, 2935, 2861, 1602, 1496, 996, 752, 692, 667; HRMS (ESI) calcd. for $C_{16}H_{21}O_4$ $[M - OH]^+$: 277.1440, found 277.1462; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (80/20 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 8.6 min (major) and 13.1 min (minor), 95% *ee*, $[\alpha]_D^{25} = +37.2$ ($c = 0.5$, CH_2Cl_2).

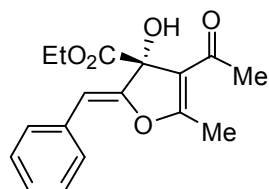
(R,Z)-2-benzylidene-3-hydroxy-3-(trifluoromethyl)-3,5,6,7-tetrahydrobenzofuran-4(2H)-



one (4lc): Yield = 58%; White solid, mp = 145–146 °C; 1H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, $J = 7.6$ Hz, 2H), 7.43–7.34 (m, 2H), 7.34–7.27 (m, 1H), 6.26 (s, 1H), 4.48 (s, 1H), 2.84–2.62 (m, 2H), 2.60–2.47 (m, 1H), 2.46–2.33 (m, 1H), 2.26–2.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.0, 178.5, 150.1, 132.4, 129.5 (2C), 128.5 (2C), 128.3, 123.7 (q, $J =$

283.9, 1C), 112.5, 111.0 (d, $J = 1.8$, 1C), 79.2 (q, $J = 33.3$, 1C), 36.6, 23.7, 20.9; FTIR ν_{max} (NaCl film, cm^{-1}): 3077, 1514, 1369, 1303, 1207, 1131, 1096, 969, 701; HRMS (ESI) calcd. for $C_{16}H_{12}F_3O_2$ $[M - OH]^+$: 293.0789, found 293.0795; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (90/10 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 9.8 min (major) and 11.2 min (minor), 44% *ee*, $[\alpha]_D^{25} = +50.7$ ($c = 1.0$, CH_2Cl_2).

Ethyl (R,Z)-4-acetyl-2-benzylidene-3-hydroxy-5-methyl-2,3-dihydrofuran-3-carboxylate

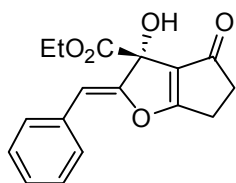


(4ad): Yield = 92%; White solid, mp = 128–130 °C; 1H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, $J = 7.6$ Hz, 2H), 7.38–7.30 (m, 2H), 7.28–7.21 (m, 1H), 5.82 (s, 1H), 4.40 (s, 1H), 4.34–4.18 (m, 2H), 2.52 (s, 3H), 2.29 (s, 3H), 1.23 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 192.8, 171.8, 167.9, 153.3, 133.2, 129.0 (2C), 128.4 (2C), 127.6, 118.2, 106.0, 81.8, 63.1, 29.0, 15.2, 13.9; FTIR ν_{max} (NaCl film, cm^{-1}): 2914, 2844,

1715, 1697, 1557, 1540, 1395, 1121; HRMS (ESI) calcd. for $C_{17}H_{17}O_4$ $[M - OH]^+$: 285.1127, found 285.1151; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH

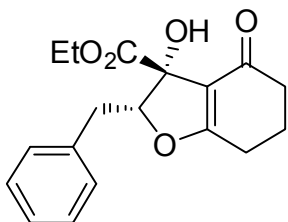
eluent (80/20 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 9.9 min and 11.6 min, 0% *ee*.

Ethyl (R,Z)-2-benzylidene-3-hydroxy-4-oxo-3,4,5,6-tetrahydro-2H-cyclopenta[b]furan-3-carboxylate (4ae):



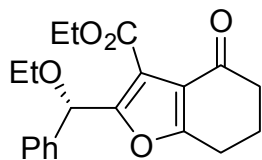
Yield = 90%; White solid, mp = 117–118 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.70–7.62 (m, 2H), 7.46–7.37 (m, 3H), 5.68 (s, 1H), 4.33 (s, 1H), 4.36–4.18 (m, 2H), 2.90 (dd, *J* = 5.6, 4.4 Hz, 2H), 2.64–2.51 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 201.7, 179.1, 172.6, 151.8, 131.8, 129.9, 128.6 (2C), 125.4 (2C), 116.3, 102.1, 65.9, 63.2, 33.5, 25.7, 14.0; FTIR $\nu_{\max}$ (NaCl film, cm⁻¹): 2918, 1733, 1697, 1559, 1395, 1044; HRMS (ESI) calcd. for C₁₇H₁₅O₄ [M - OH]⁺: 283.0970, found 283.0974; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (60/40 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 8.0 min and 10.7 min, 0% *ee*.

Ethyl (2R,3R)-2-benzyl-3-hydroxy-4-oxo-2,3,4,5,6,7-hexahydrobenzofuran-3-carboxylate (6ac):



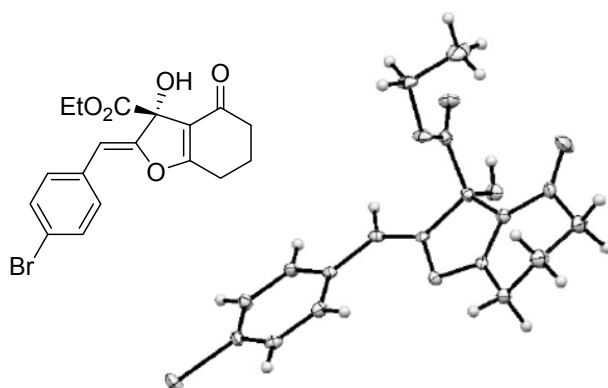
Yield = 73%; Gummy liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.38–7.29 (m, 2H), 7.29–7.20 (m, 3H), 4.88 (dd, *J* = 10.4, 4.0 Hz, 1H), 4.28 (q, *J* = 7.2 Hz, 2H), 4.05 (s, 1H), 3.04 (dd, *J* = 14.4, 10.4 Hz, 1H), 2.87 (dd, *J* = 14.4, 4.0 Hz, 1H), 2.52 (t, *J* = 6.4 Hz, 2H), 2.44–2.26 (m, 2H), 2.18–2.02 (m, 2H), 1.29 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 194.2, 179.9, 171.5, 136.5, 129.0 (2C), 128.6 (2C), 127.0, 116.9, 94.9, 82.4, 62.8, 36.8, 36.4, 24.2, 21.4, 14.1; FTIR $\nu_{\max}$ (NaCl film, cm⁻¹): 3085, 3033, 2928, 1732, 1644, 1402, 1232, 1187, 1094, 982, 759, 576, 504; HRMS (ESI) calcd. for C₁₈H₂₁O₅ [M + H]⁺: 317.1389, found 317.1395; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (90/10 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 22.7 min (minor) and 26.4 min (major), >99% *ee*, [α]_D²⁵ = -53.5 (*c* = 1.0, CH₂Cl₂).

Ethyl (S)-2-(ethoxy(phenyl)methyl)-4-oxo-4,5,6,7-tetrahydrobenzofuran-3-carboxylate (7ac):



Yield = 95%; Gummy liquid; ¹H NMR (400 MHz, CDCl₃): δ 7.55–7.42 (m, 2H), 7.40–7.32 (m, 2H), 7.32–7.26 (m, 1H), 5.95 (s, 1H), 4.48–4.32 (m, 2H), 3.64–3.48 (m, 2H), 2.95–2.70 (m, 2H), 2.58–2.38 (m, 2H), 2.21–2.05 (m, 2H), 1.41 (t, *J* = 7.2 Hz, 3H), 1.26 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 192.0, 167.1, 163.1, 157.3, 138.4, 128.5 (2C), 128.0, 126.7 (2C), 119.1, 113.9, 74.3, 65.1, 61.3, 38.5, 23.5, 22.0, 15.1, 14.1; FTIR $\nu_{\max}$ (NaCl film, cm⁻¹): 2919, 1730, 1691, 1577, 1432, 1299, 1226, 1058, 1005; HRMS (ESI) calcd. for C₂₀H₂₃O₅ [M + H]⁺: 343.1545, found 343.1538; HPLC condition: CHIRALPAK AD-H (250.0 mm x 4.6 mm), Hexane/*i*-PrOH eluent (97/03 ratio, 1.0 mL/min flow rate), 254 nm wave length UV, Retention time = 19.0 min (minor) and 21.0 min (major), 02% *ee*, [α]_D²⁵ = -1.6 (*c* = 1.0, CH₂Cl₂).

Crystal Data of Compound (+)-4dc



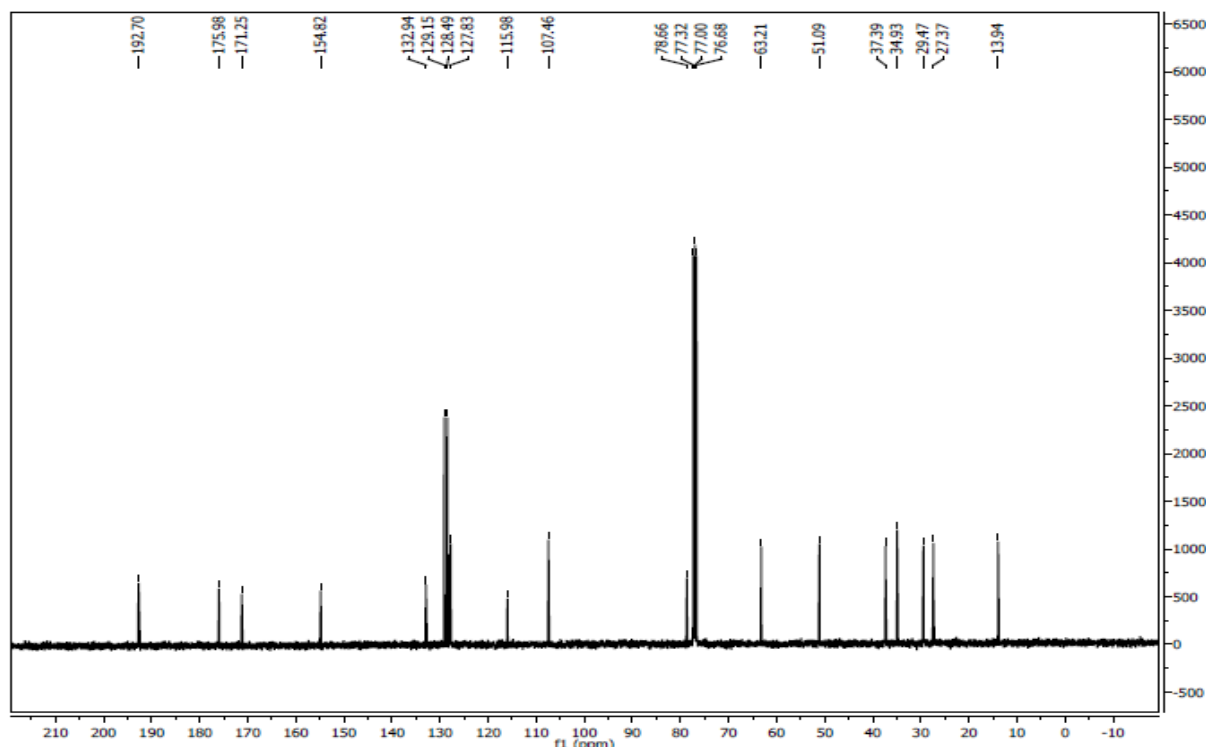
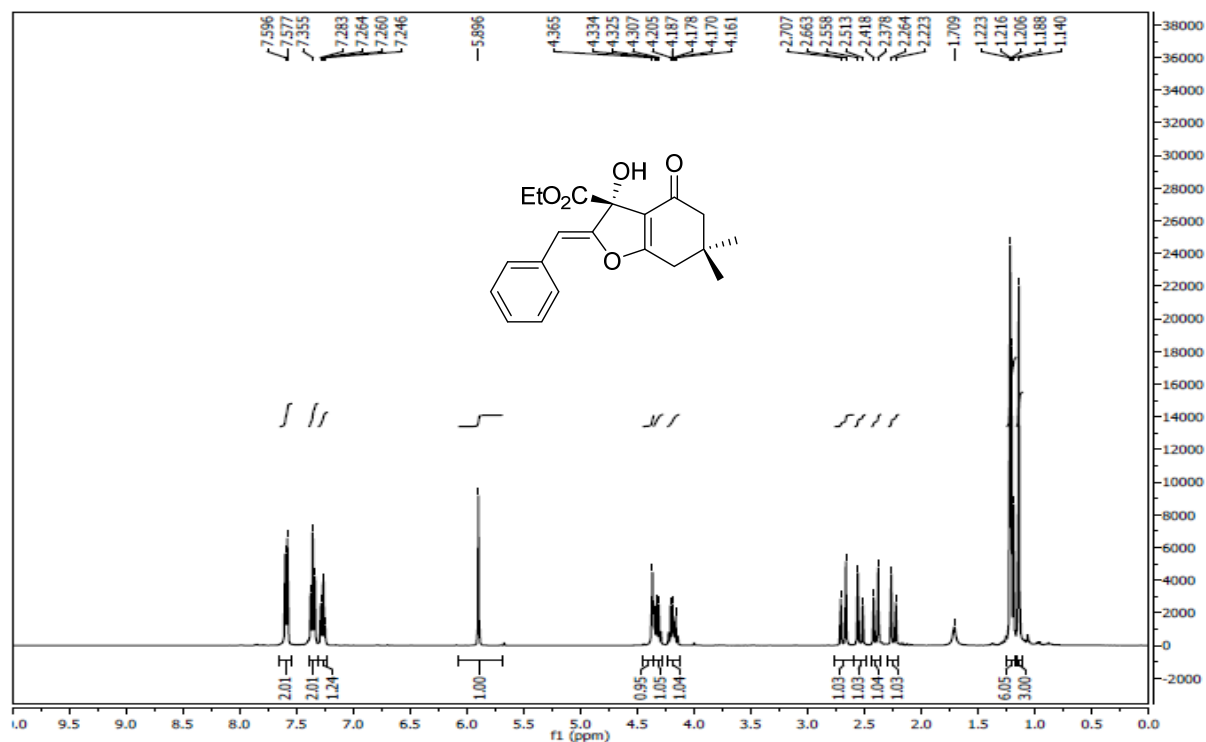
Datablock: 4dc

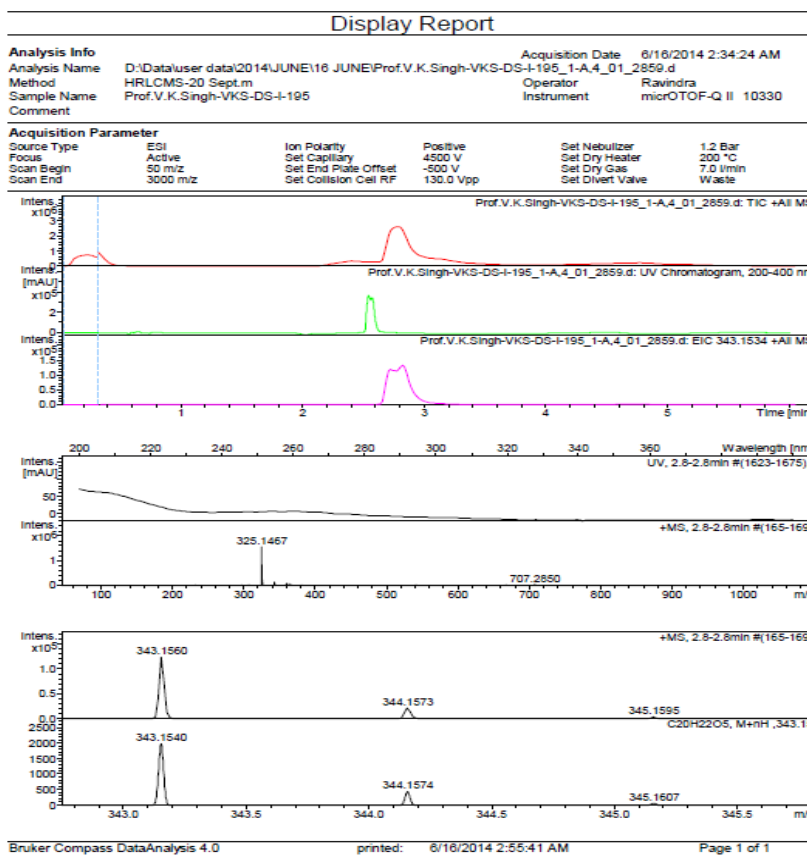
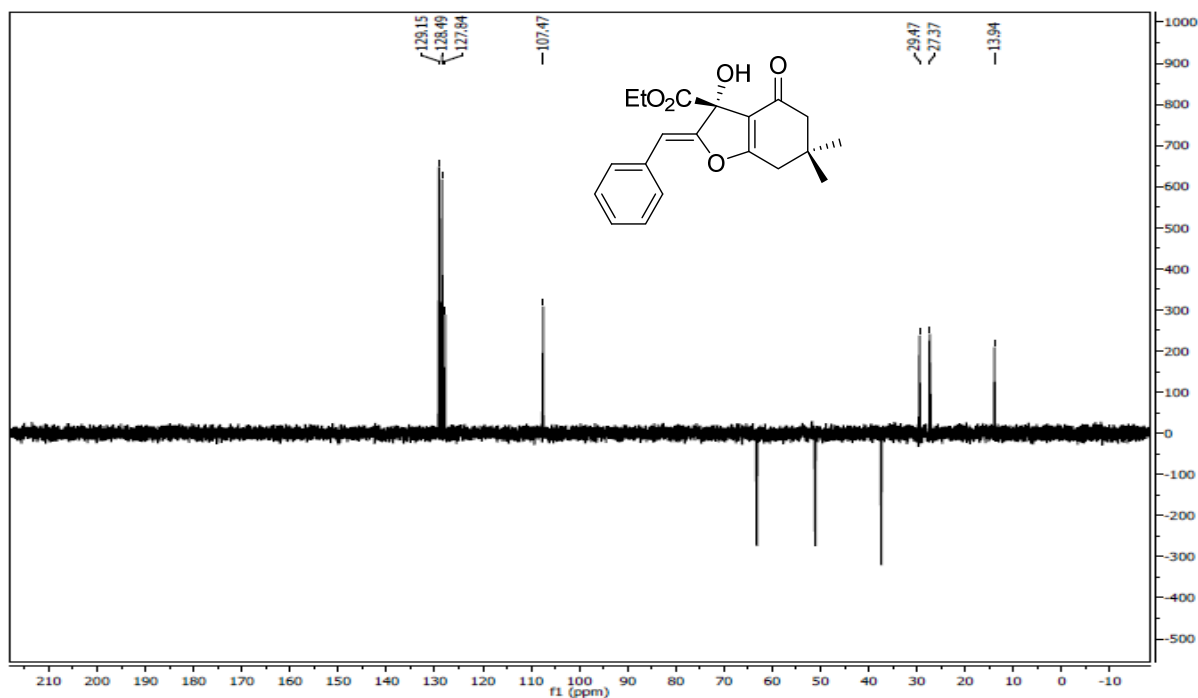
Bond precision:	C-C = 0.0087 Å	Wavelength=0.71073
Cell:	a=5.6762(6) b=10.5443(11) c=14.1611(14)	
	alpha=90 beta=100.294(4) gamma=90	
Temperature:	100 K	
	Calculated	Reported
Volume	833.92(15)	833.92(15)
Space group	P 21	P 21
Hall group	P 2yb	P 2yb
Moiety formula	C18 H17 Br O5	?
Sum formula	C18 H17 Br O5	C18 H17 Br O5
Mr	393.22	393.23
Dx, g cm ⁻³	1.566	1.566
Z	2	2
Mu (mm ⁻¹)	2.489	2.489
F000	400.0	400.0
F000'	399.63	
h, k, lmax	8, 15, 20	8, 15, 19
Nref	5273[2767]	14514
Tmin, Tmax	0.556, 0.780	0.556, 0.780
Tmin'	0.469	
Correction method=	# Reported T Limits: Tmin=0.556	
Tmax=0.780 AbsCorr =	MULTI-SCAN	
Data completeness=	5.25/2.75 Theta(max)= 30.891	
R(reflections)=	0.0518(11178) wR2(reflections)= 0.1256(14514)	
S =	0.904 Npar= 220	

Additional References

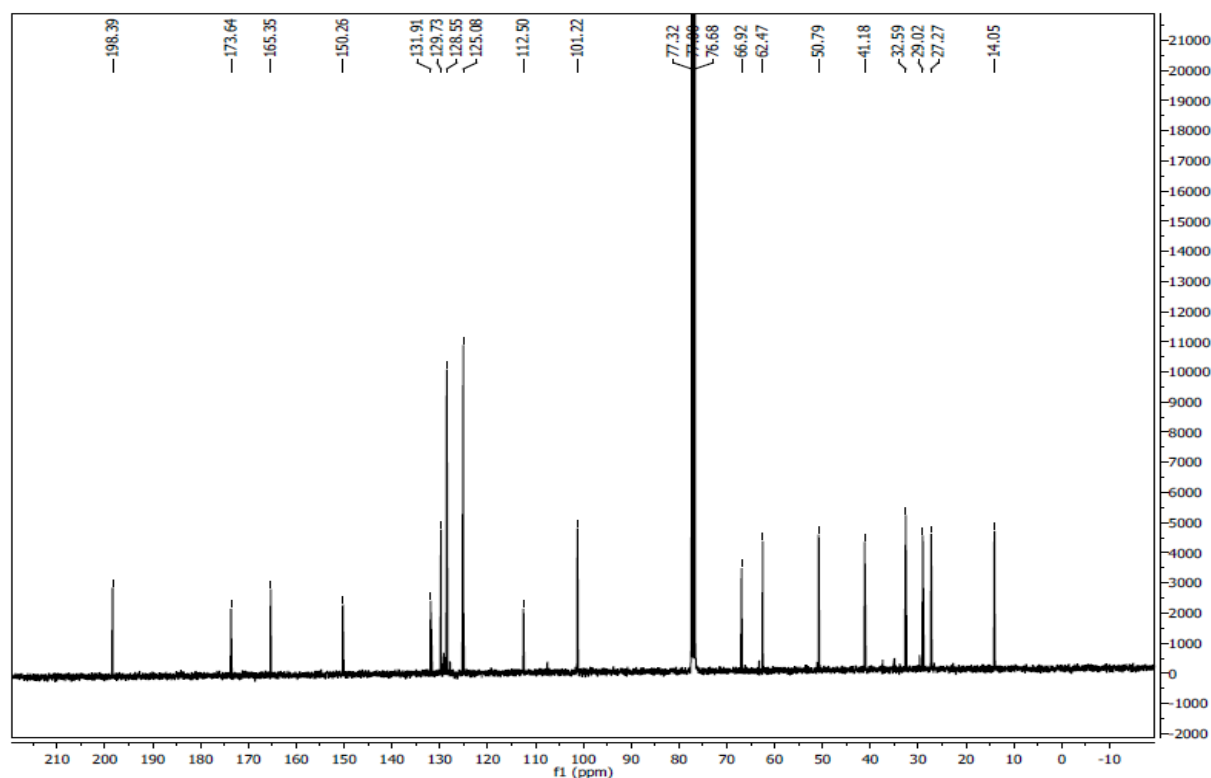
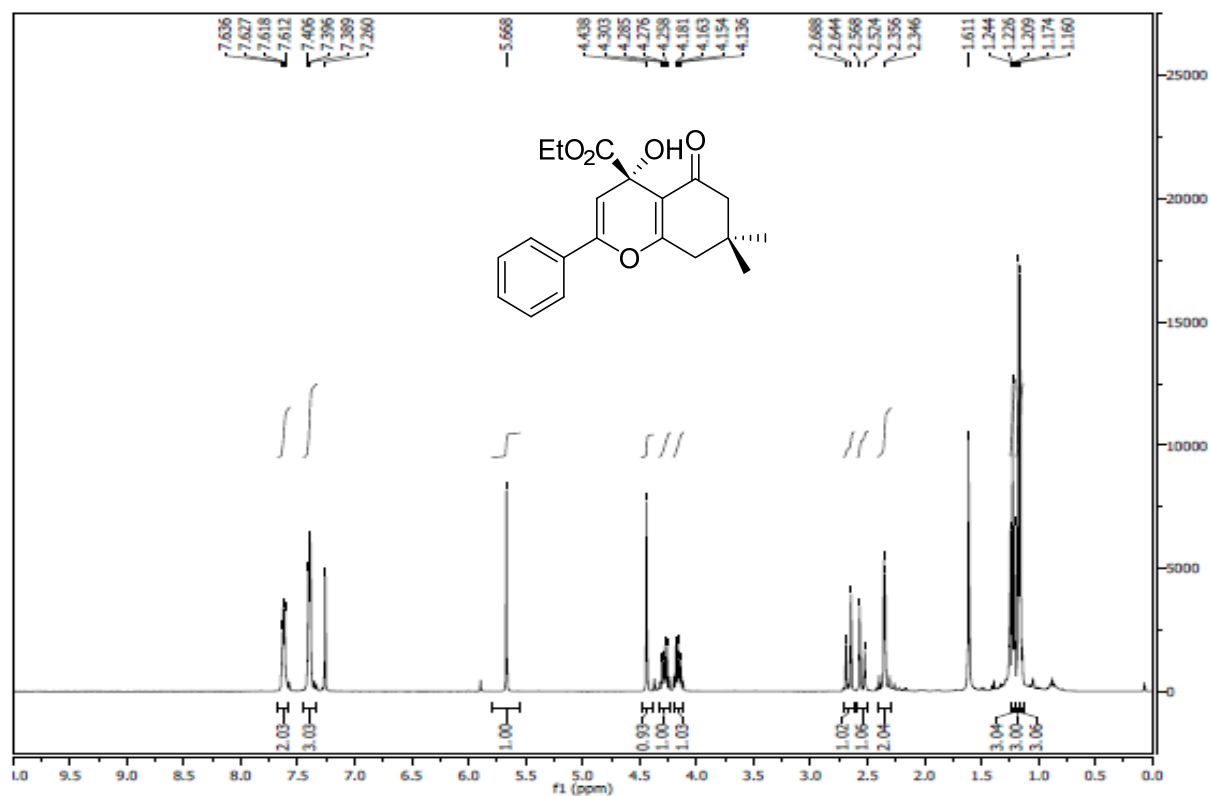
- (1) a) Guo, M.; Li, D.; Zhang, Z. *J. Org. Chem.* **2003**, *68*, 10172–10174. b) Mizota, I.; Matsuda, Y.; Kamimura, S.; Tanaka, H.; Shimizu, M. *Org. Lett.* **2013**, *15*, 4206.
- (2) Singh, R. P.; Cao, G.; Kirchmeier, R. L.; Shreeve, J. M. *J. Org. Chem.* **1999**, *64*, 2873.

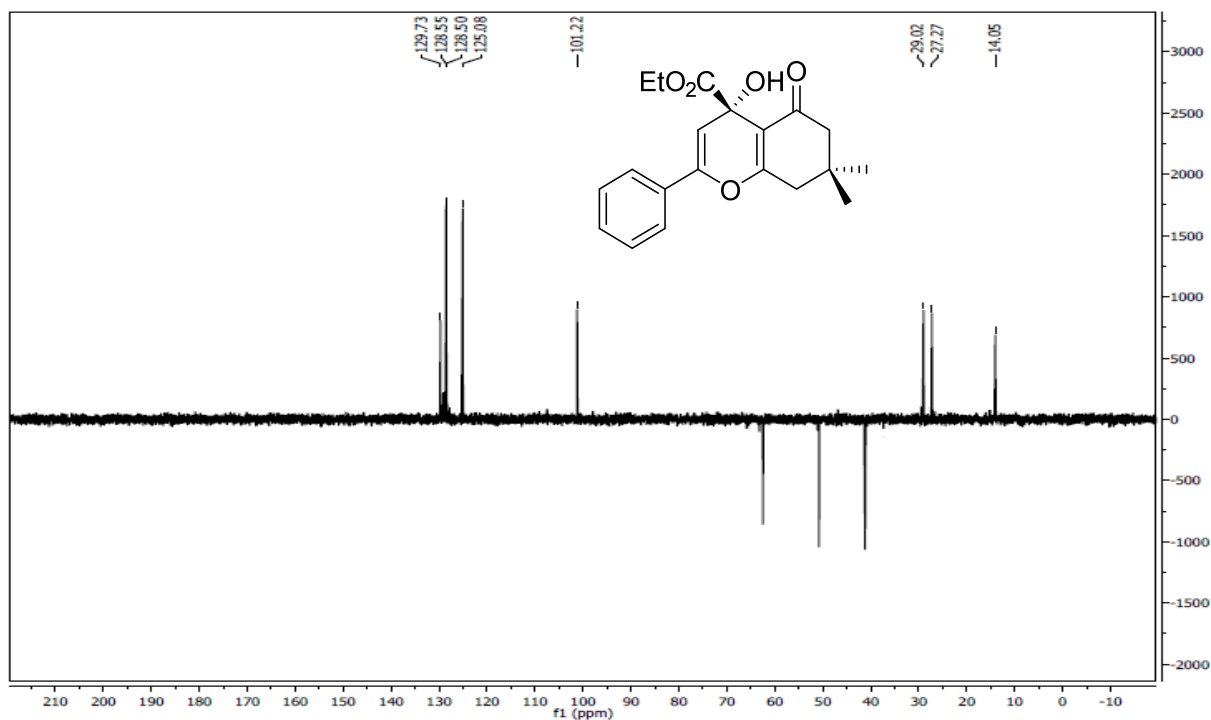
NMR Spectra of Dihydrofuran and Related Compounds



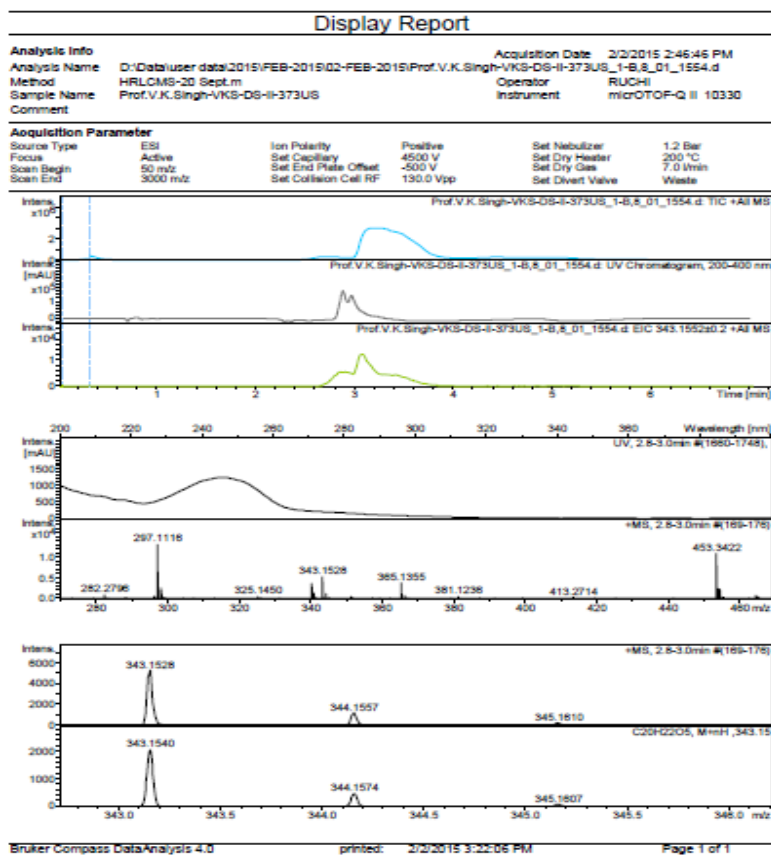


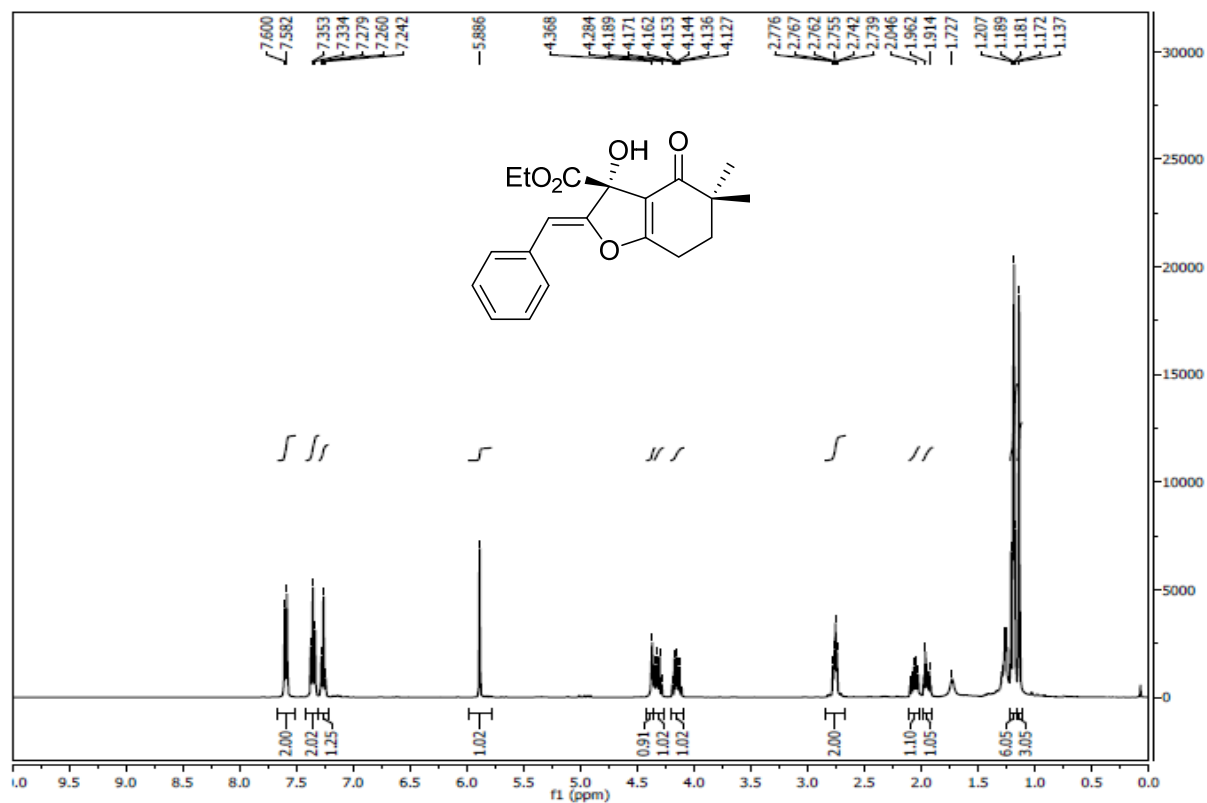
Mass Spectra of Compound 4aa (ESI)



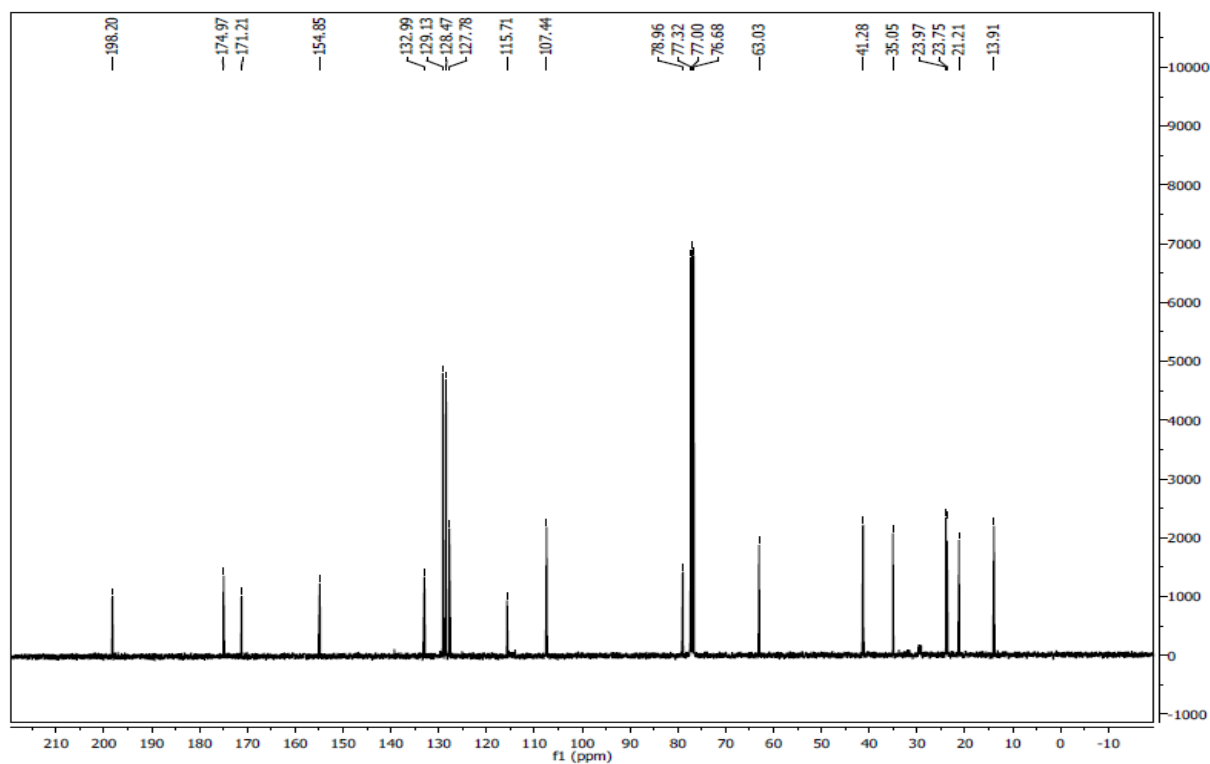


DEPT-135 of Compound 5aa (100 MHz, CDCl₃)

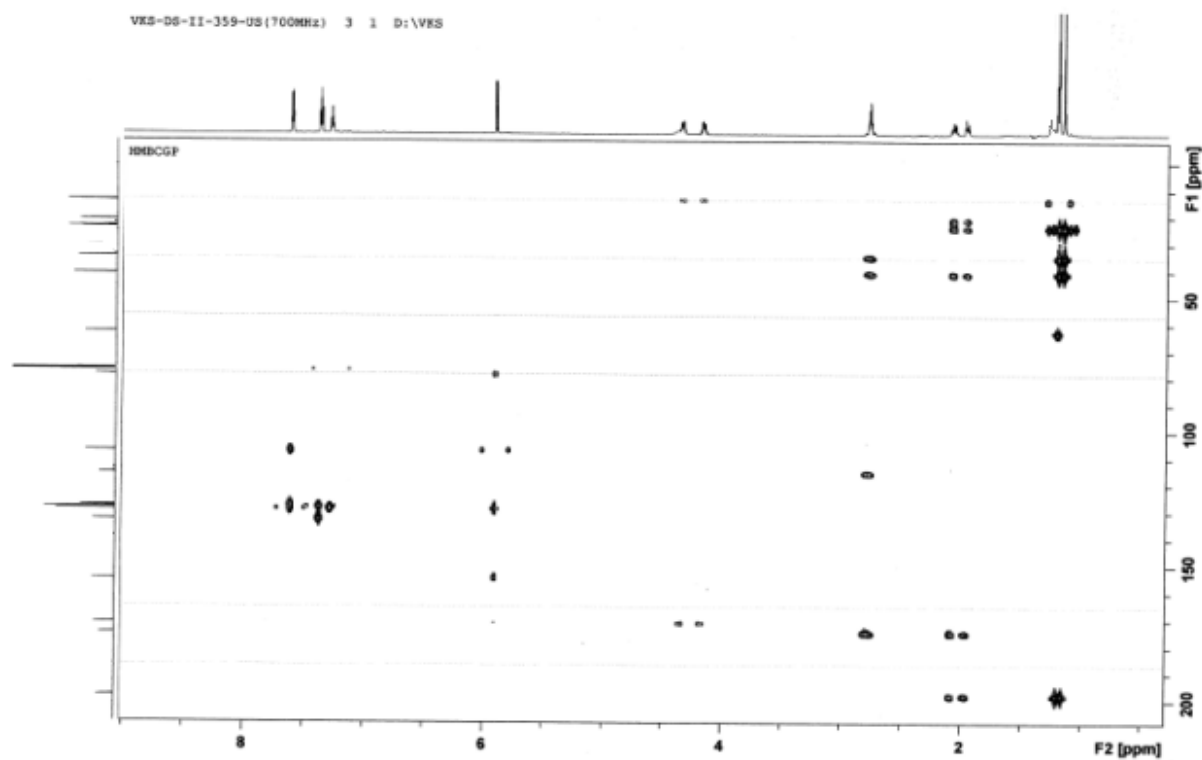
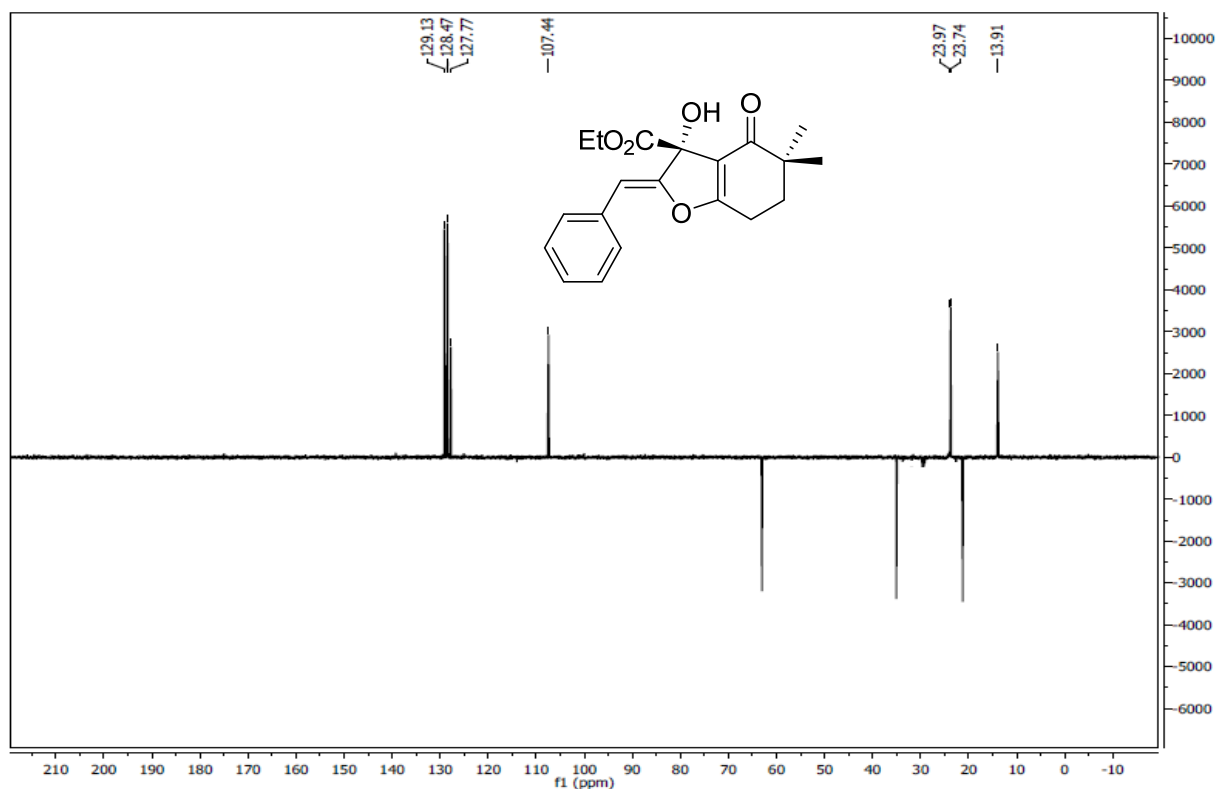


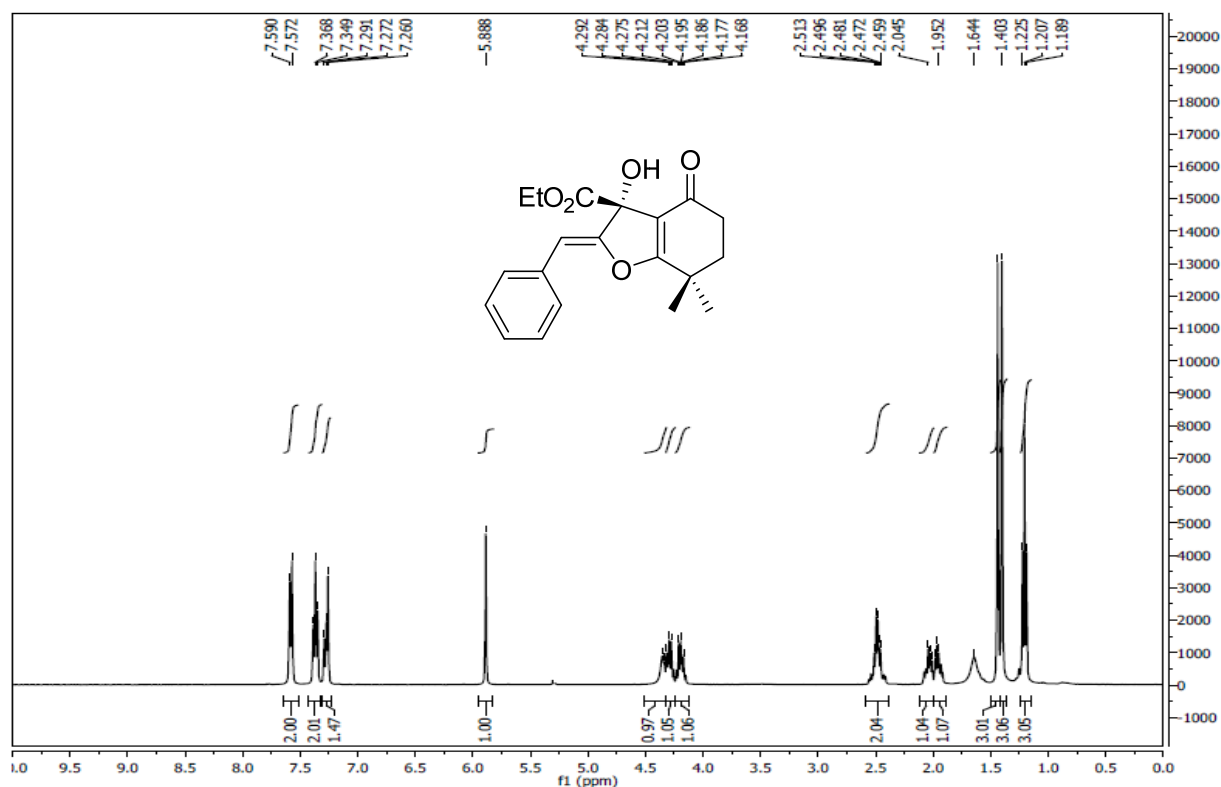


¹H NMR of Compound 4ab (400 MHz, CDCl₃)

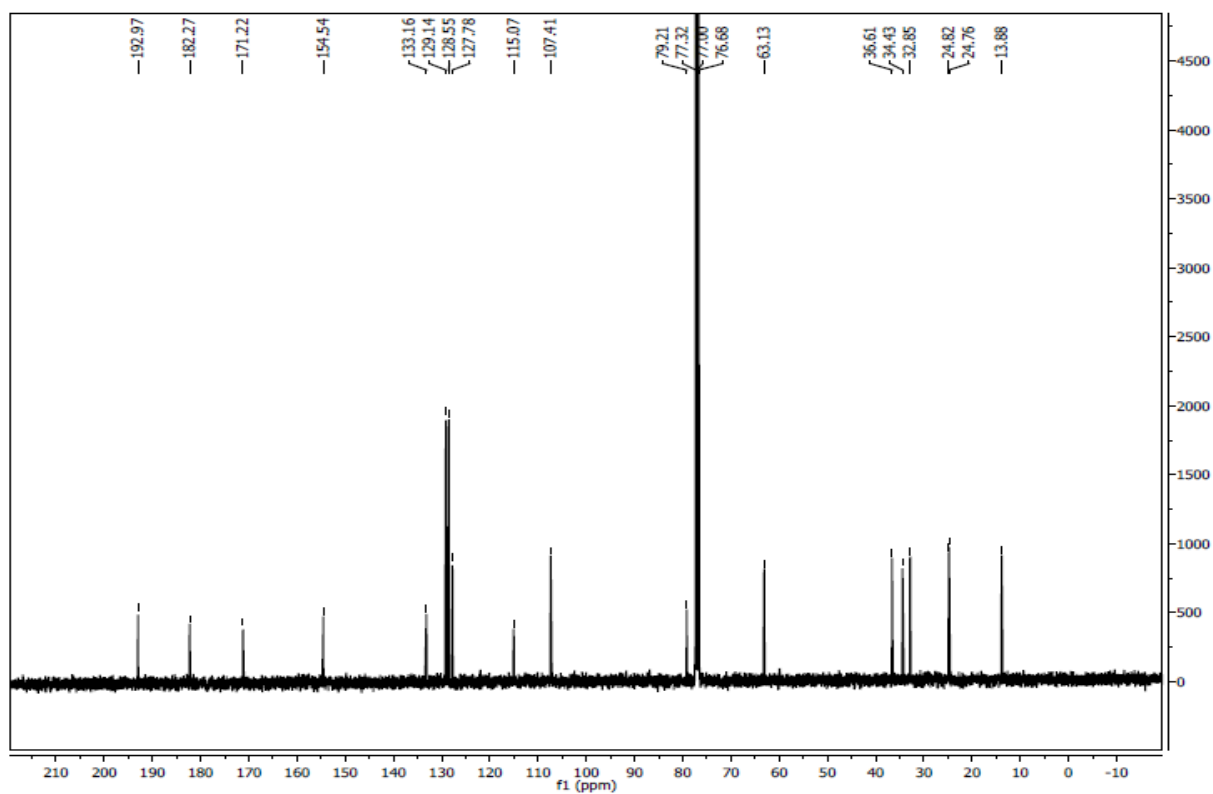


¹³C NMR of Compound 4ab (100 MHz, CDCl₃)

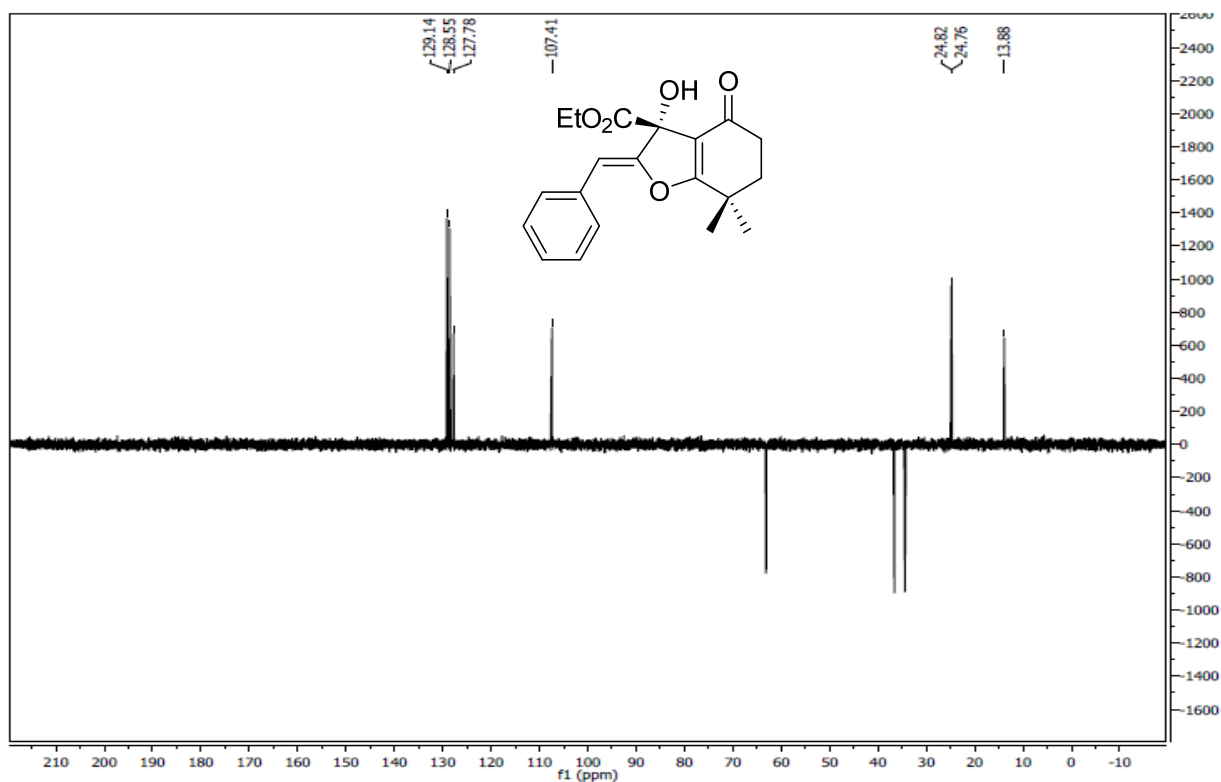




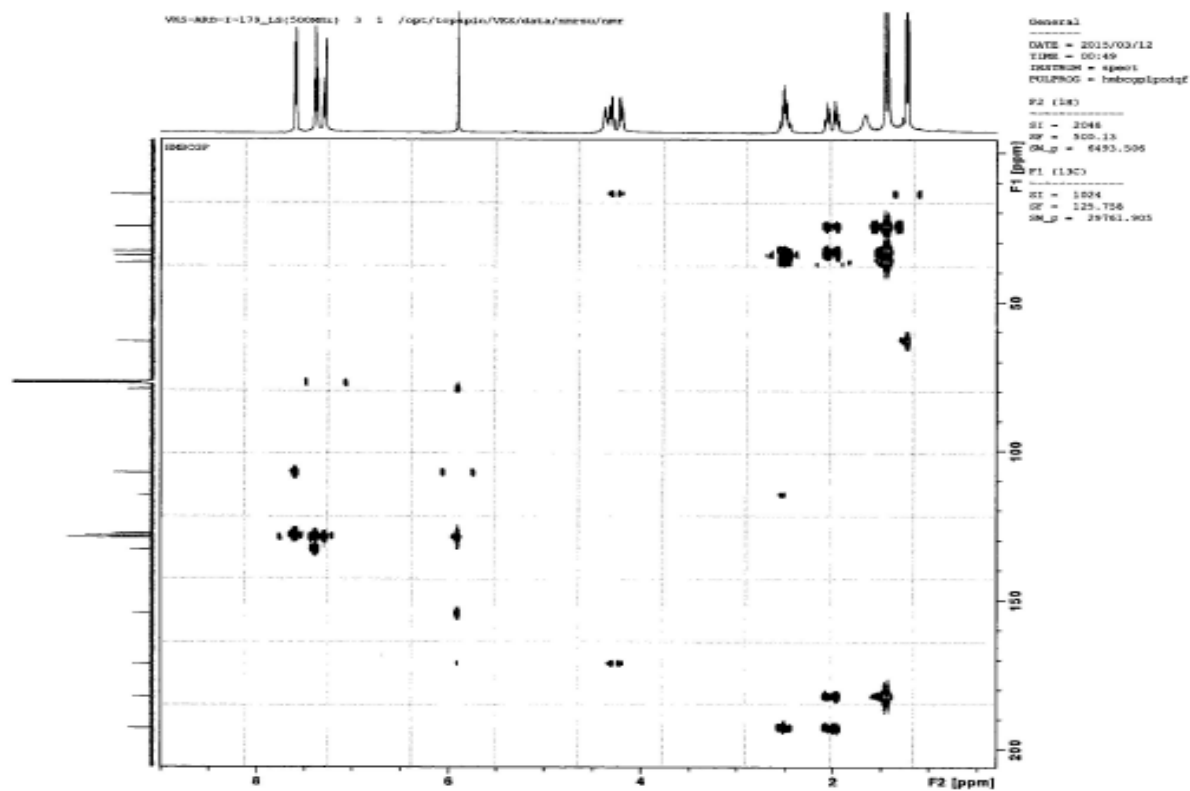
¹H NMR of Compound 4ab' (400 MHz, CDCl₃)



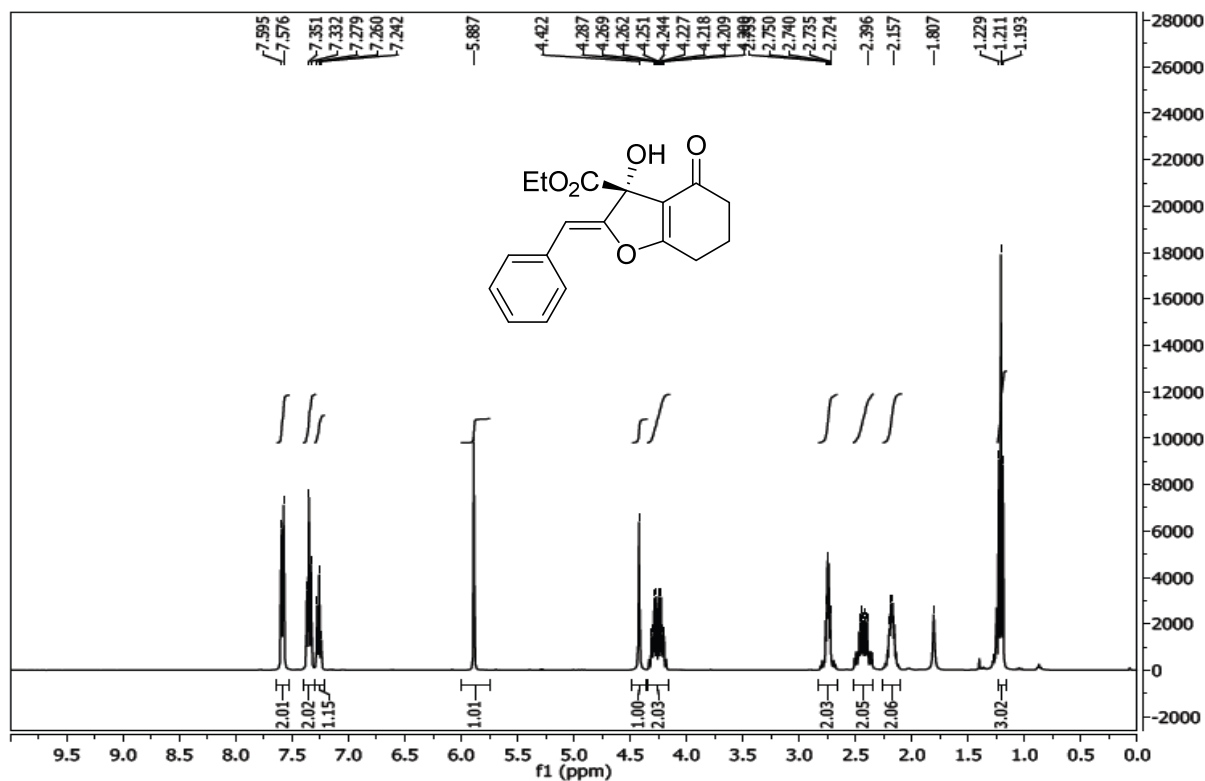
¹³C NMR of Compound 4ab' (100 MHz, CDCl₃)



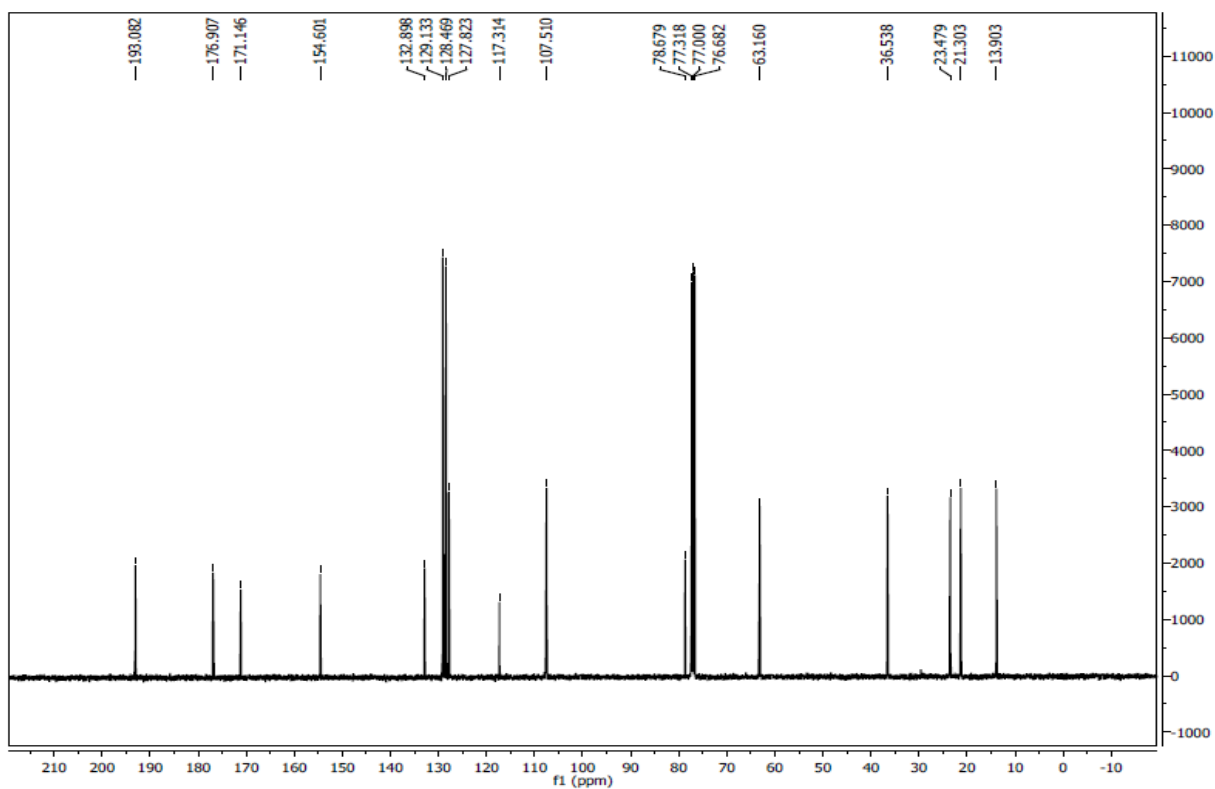
DEPT-135 of Compound 4ab' (100 MHz, CDCl₃)



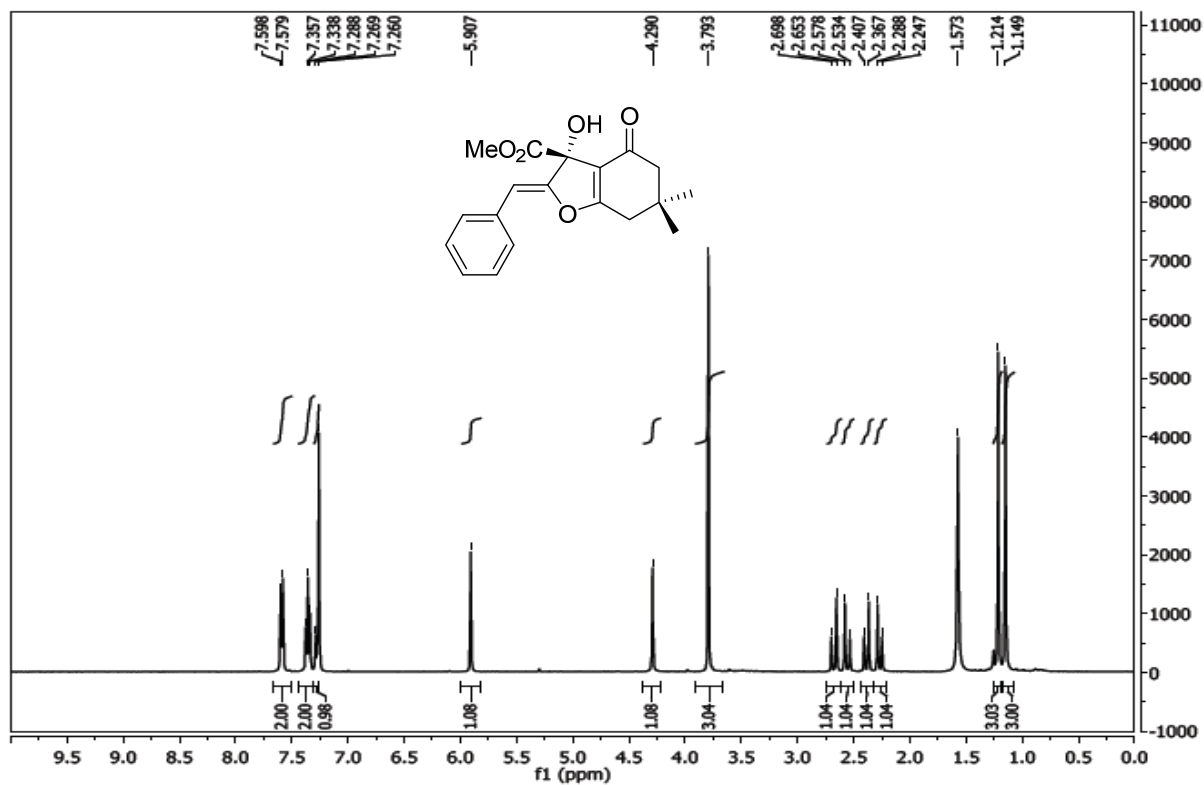
HMBC of Compound 4ab' (500 MHz, CDCl₃)



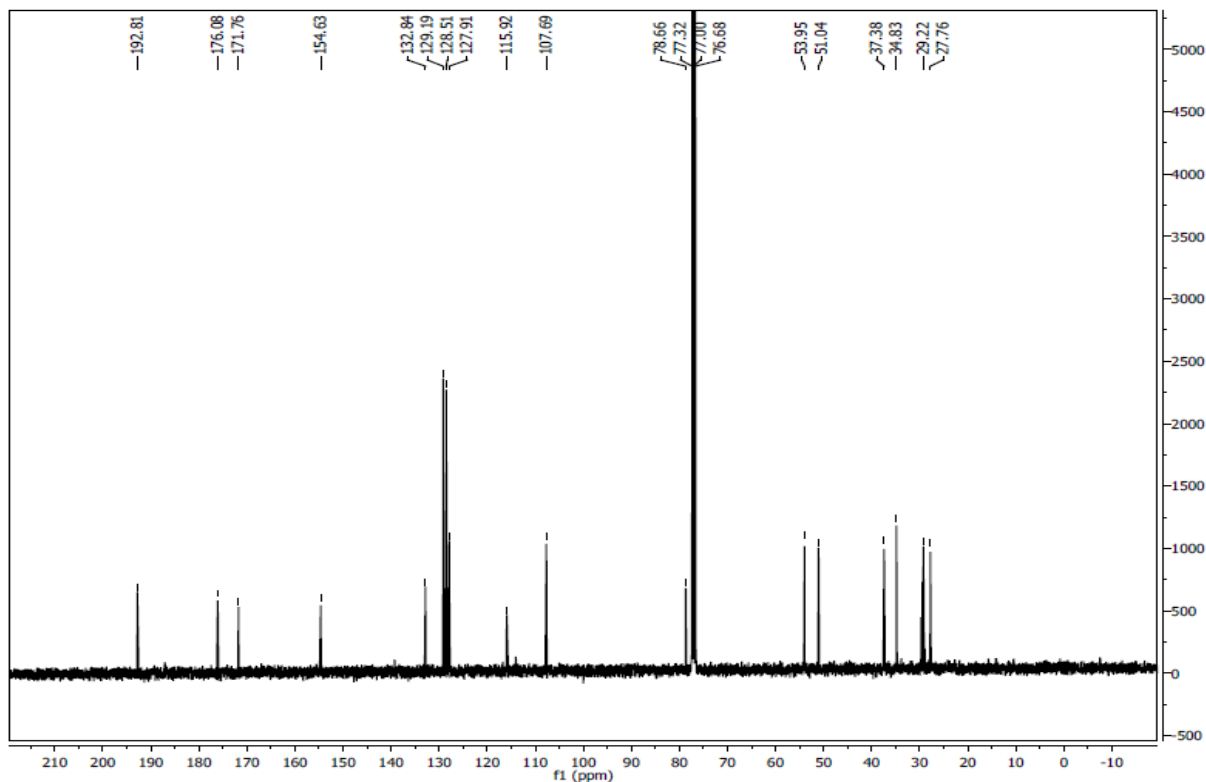
¹H NMR of Compound 4ac (400 MHz, CDCl₃)



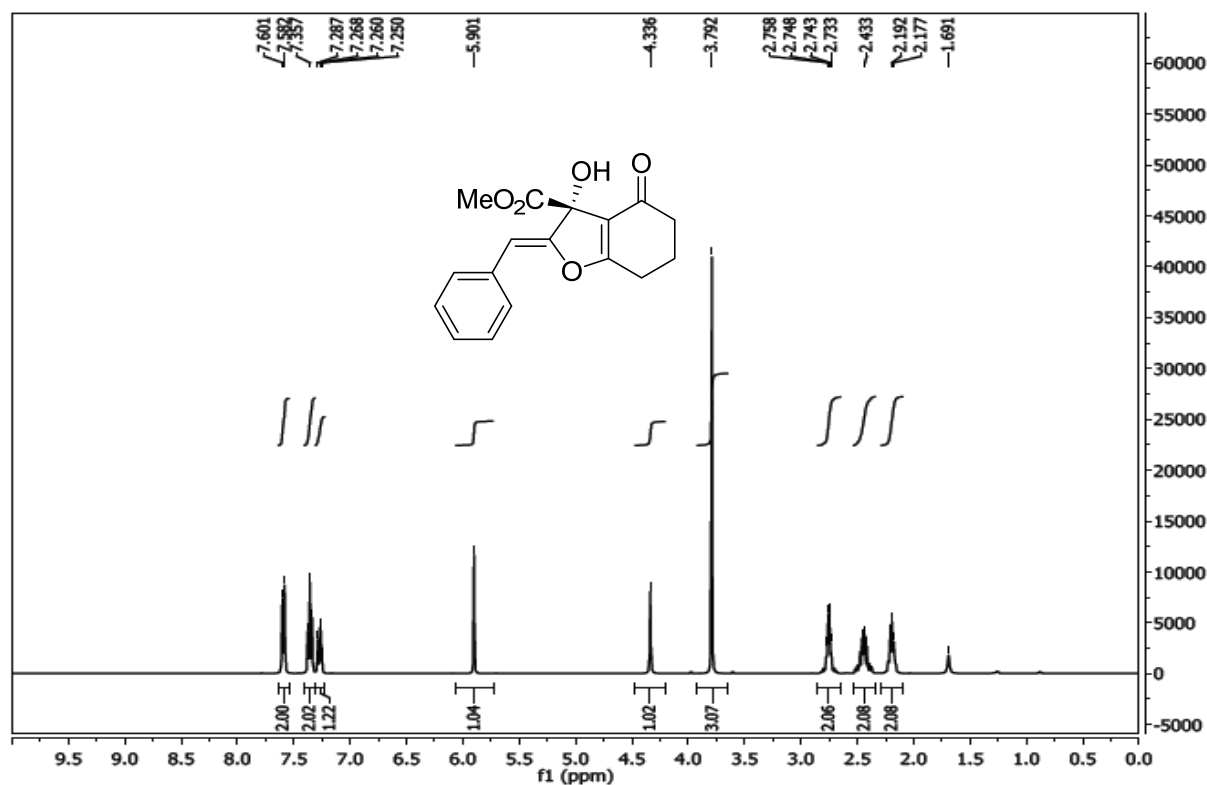
¹³C NMR of Compound 4ac (100 MHz, CDCl₃)



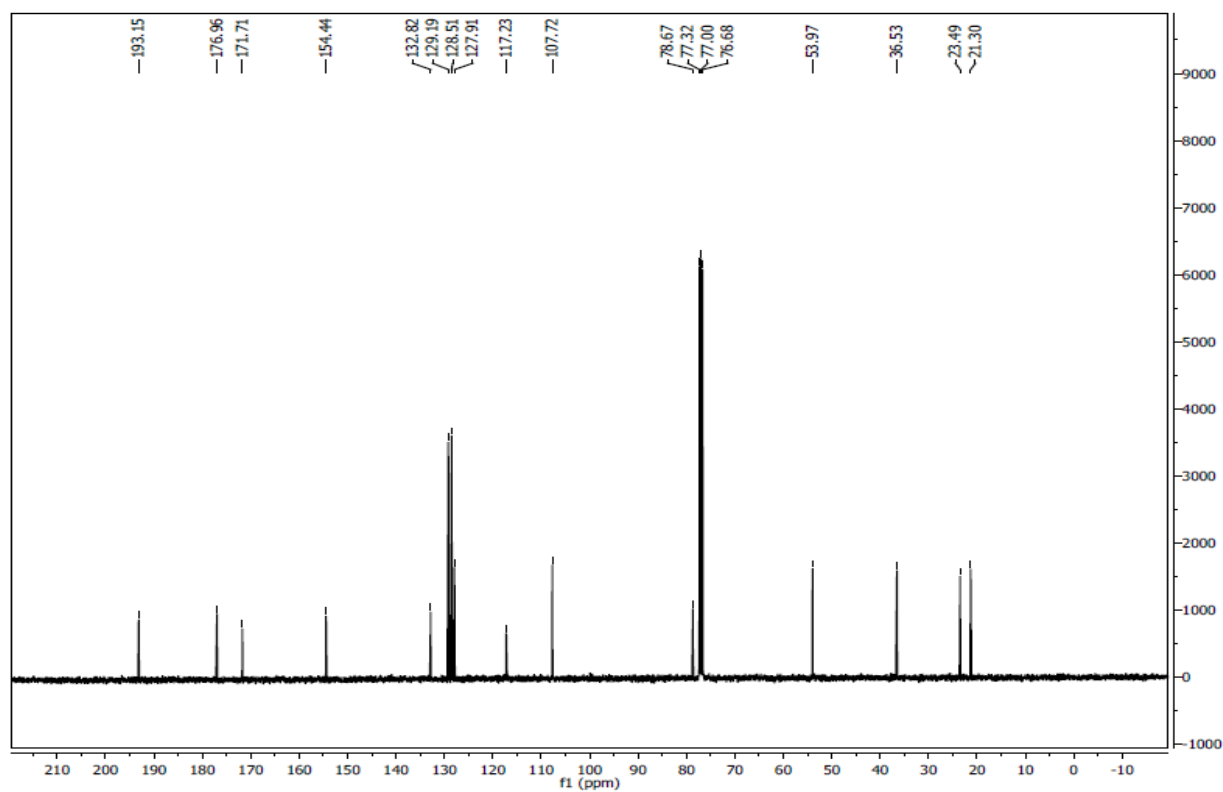
¹H NMR of Compound 4ba (400 MHz, CDCl₃)



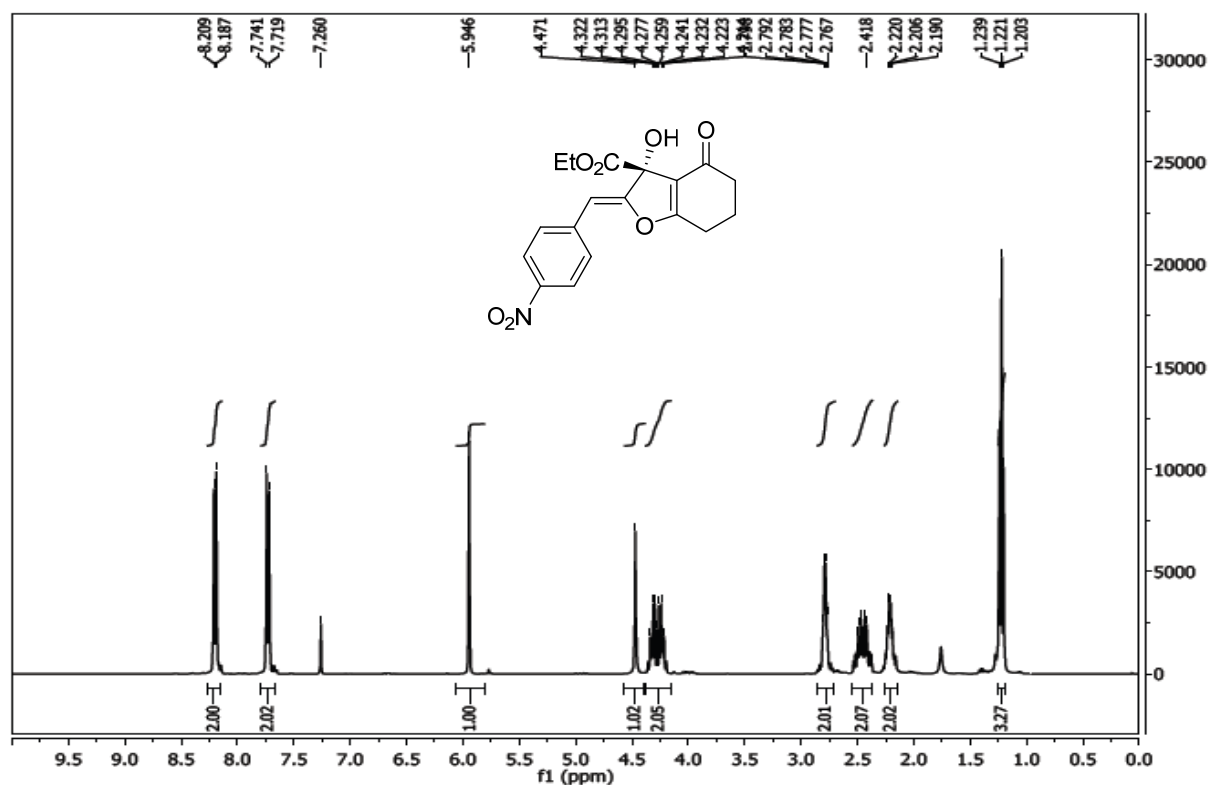
¹³C NMR of Compound 4ba (100 MHz, CDCl₃)



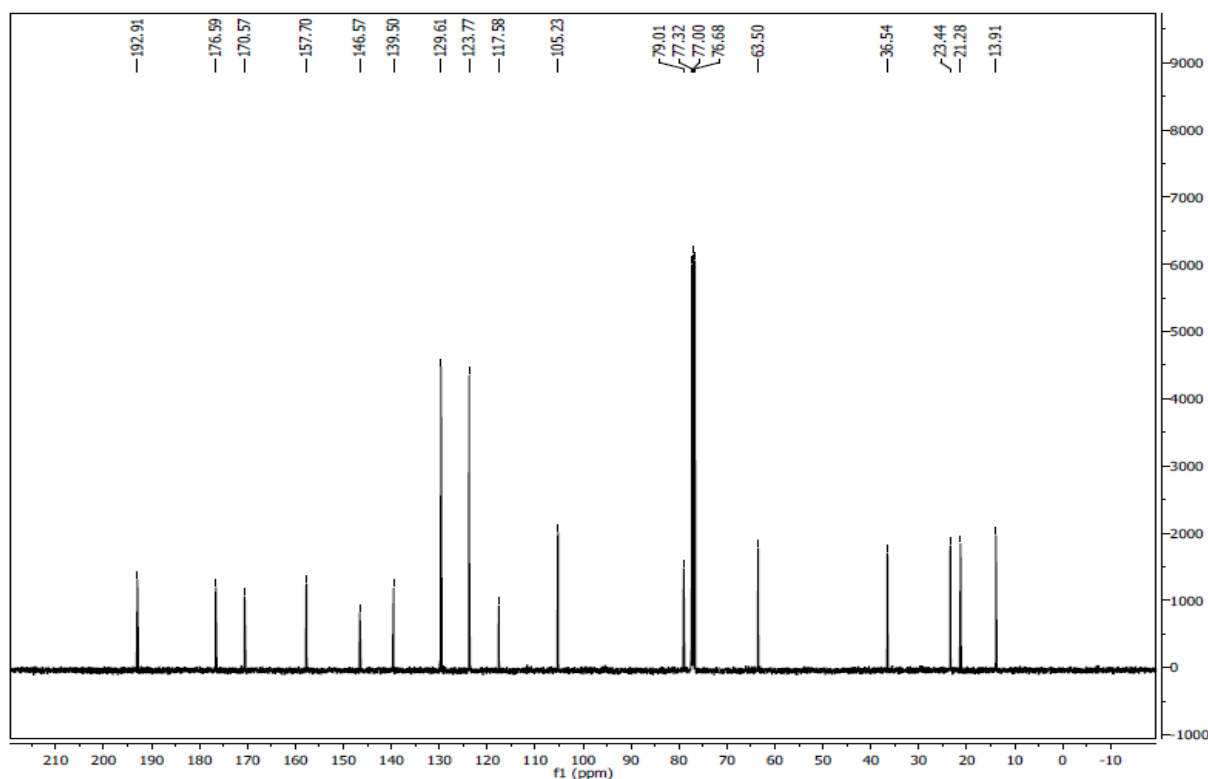
¹H NMR of Compound 4bc (400 MHz, CDCl₃)



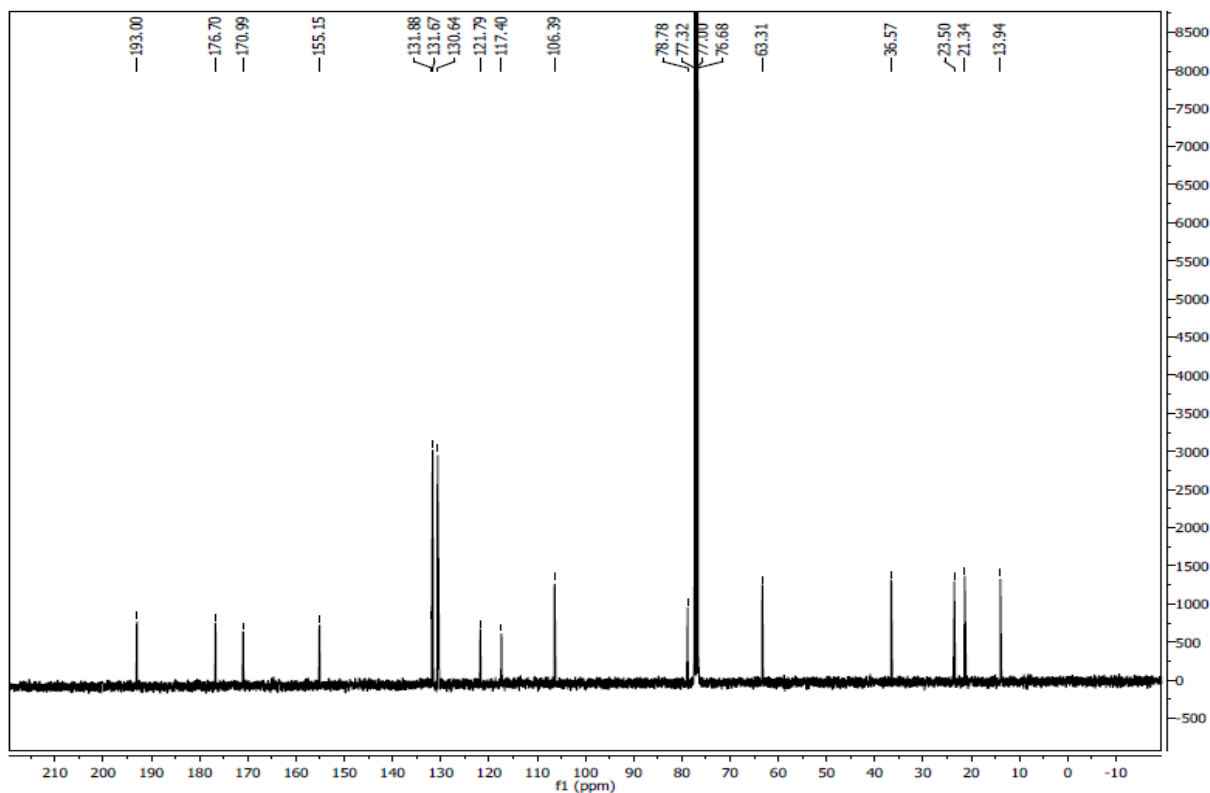
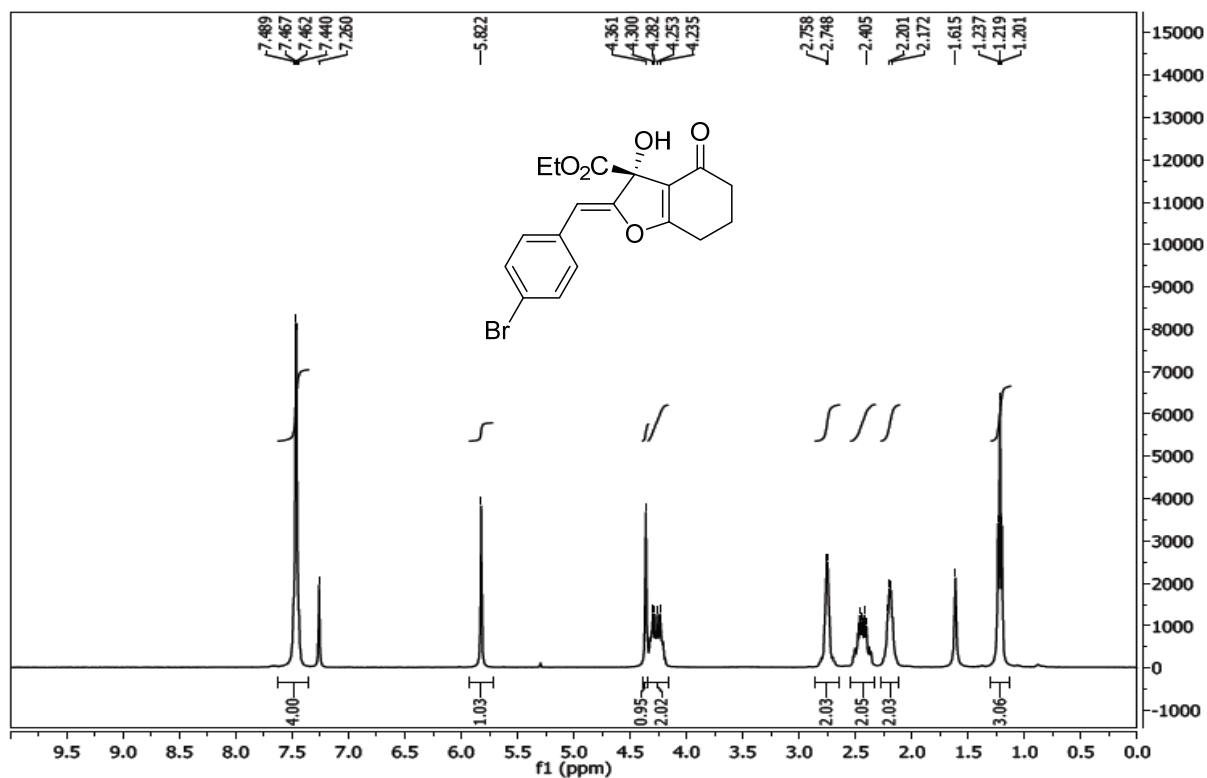
¹³C NMR of Compound 4bc (100 MHz, CDCl₃)

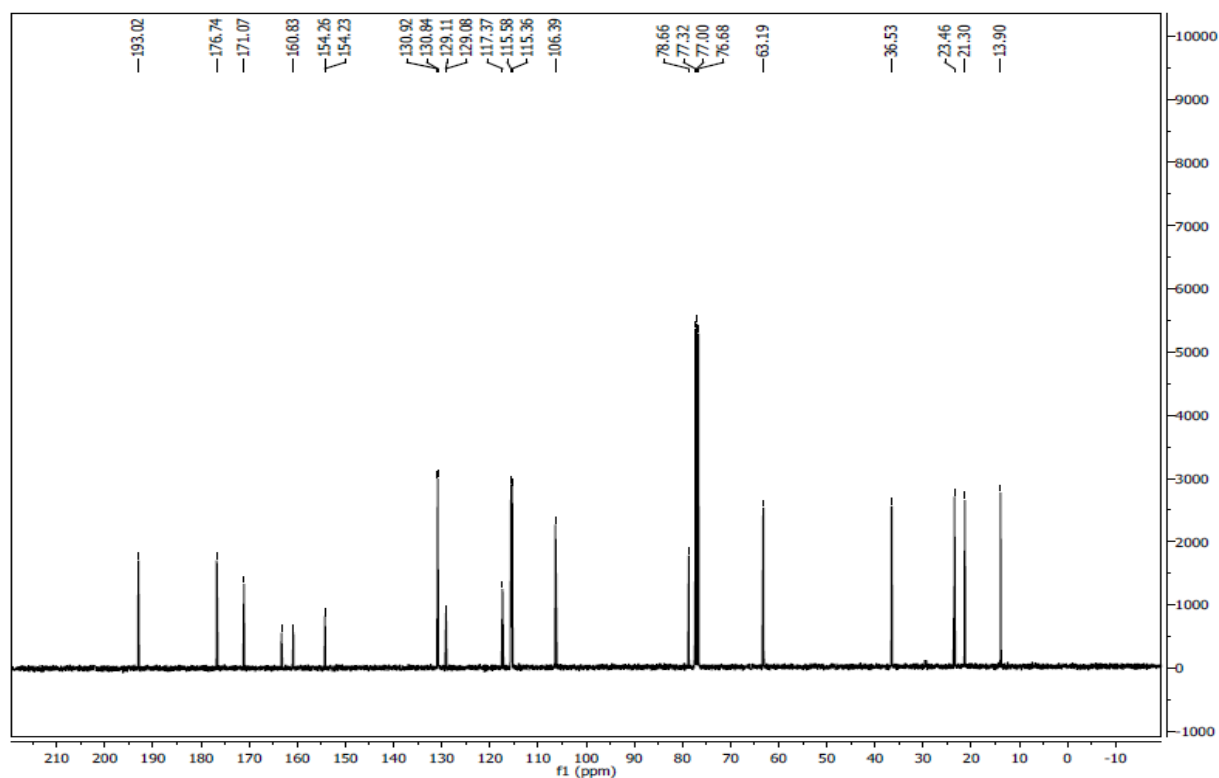
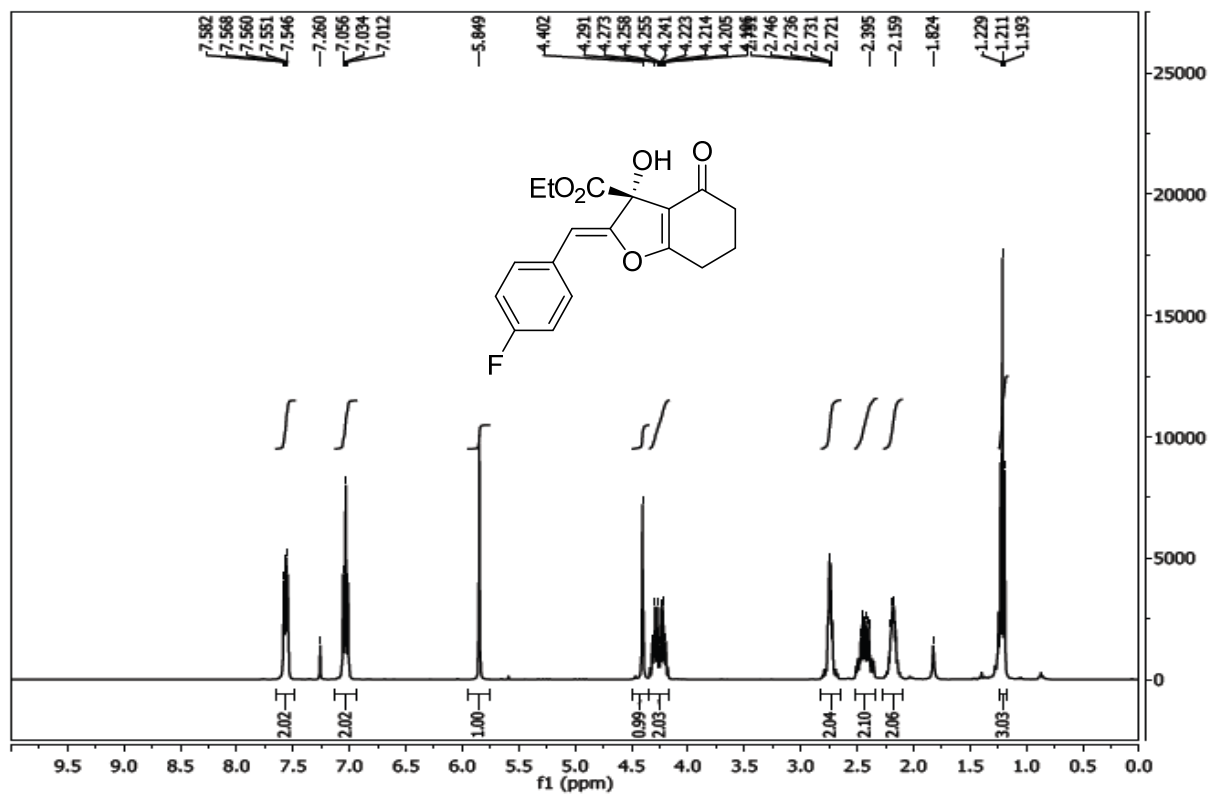


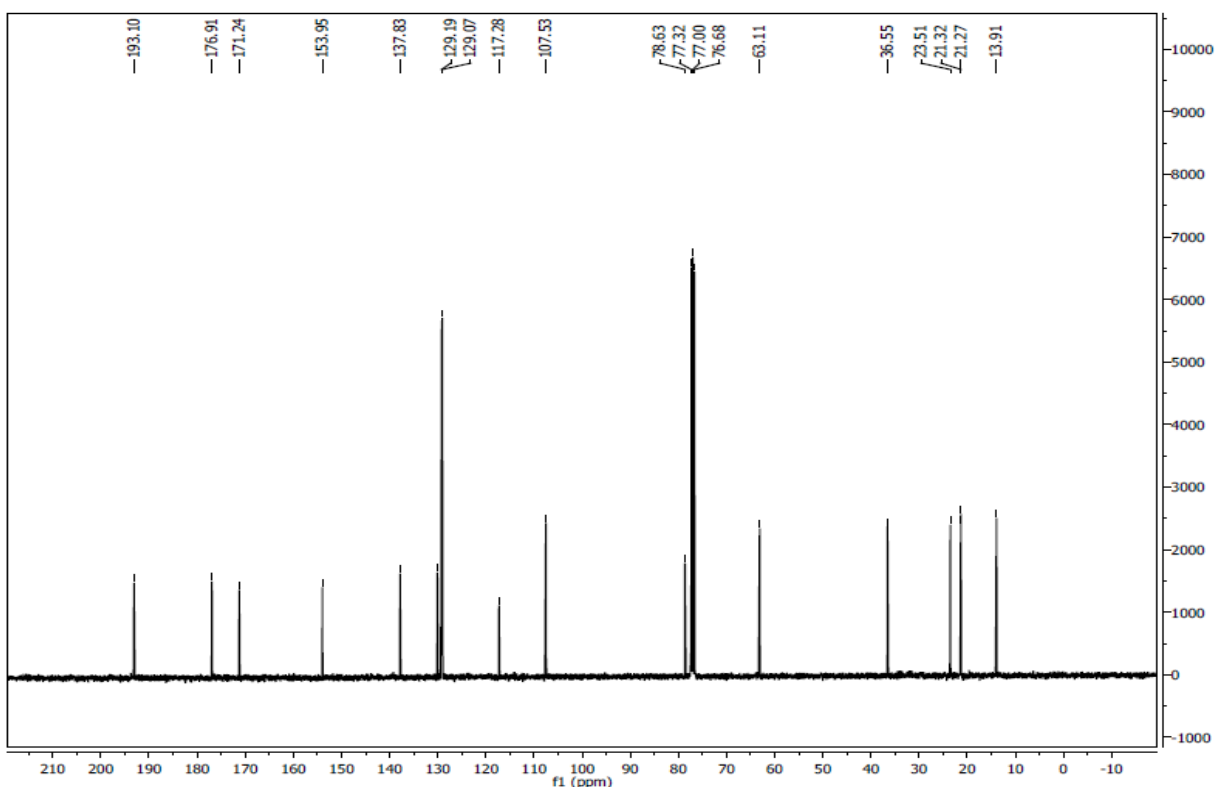
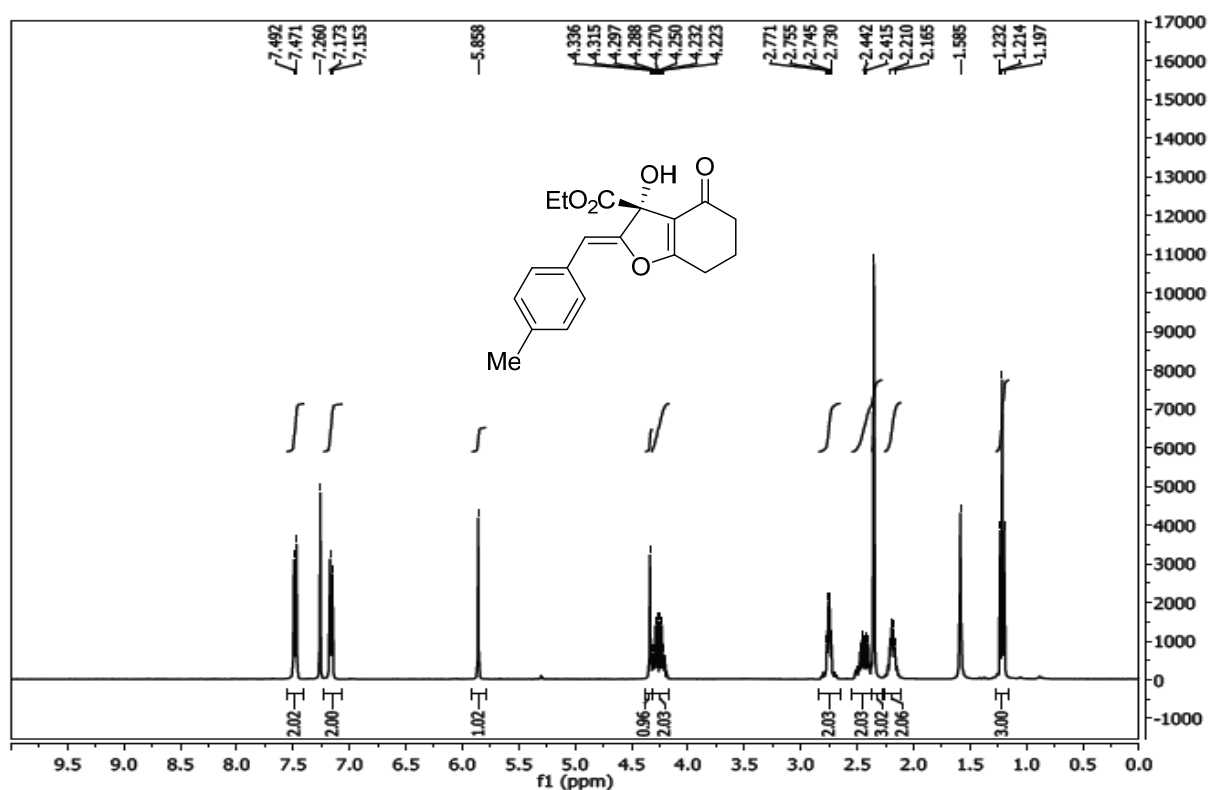
¹H NMR of Compound 4cc (400 MHz, CDCl₃)

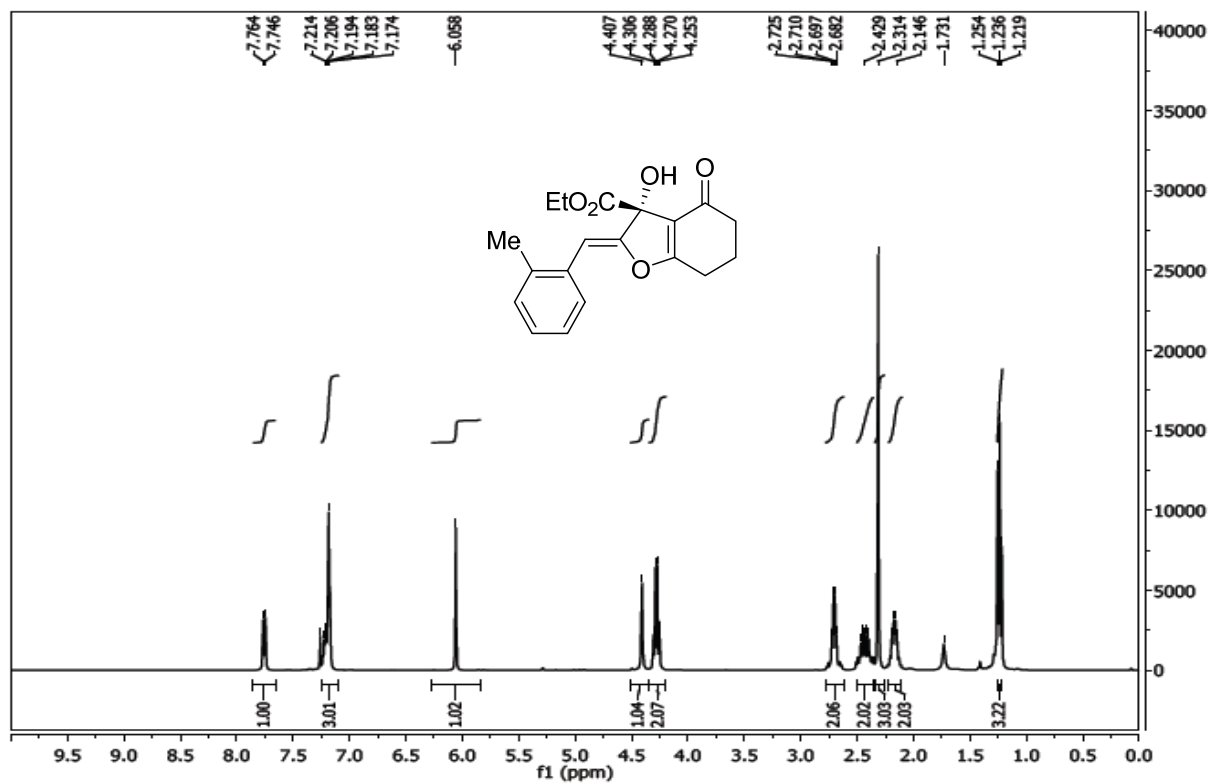


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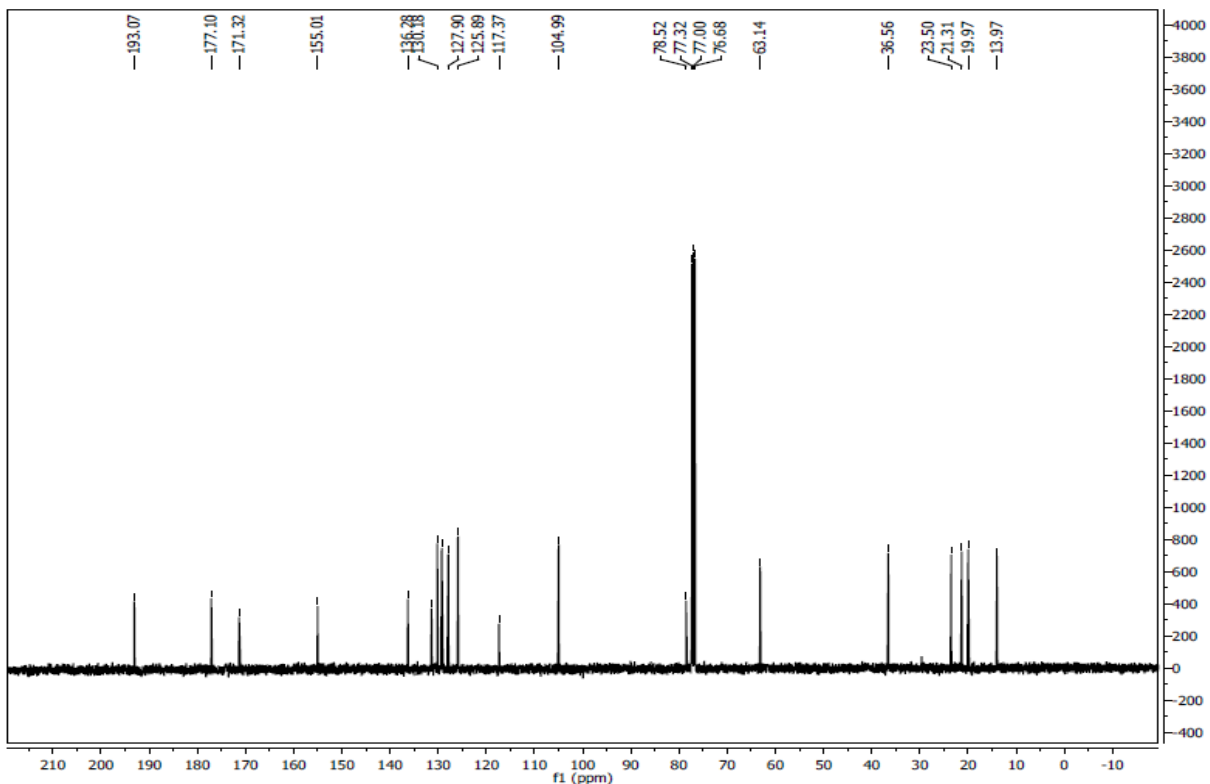




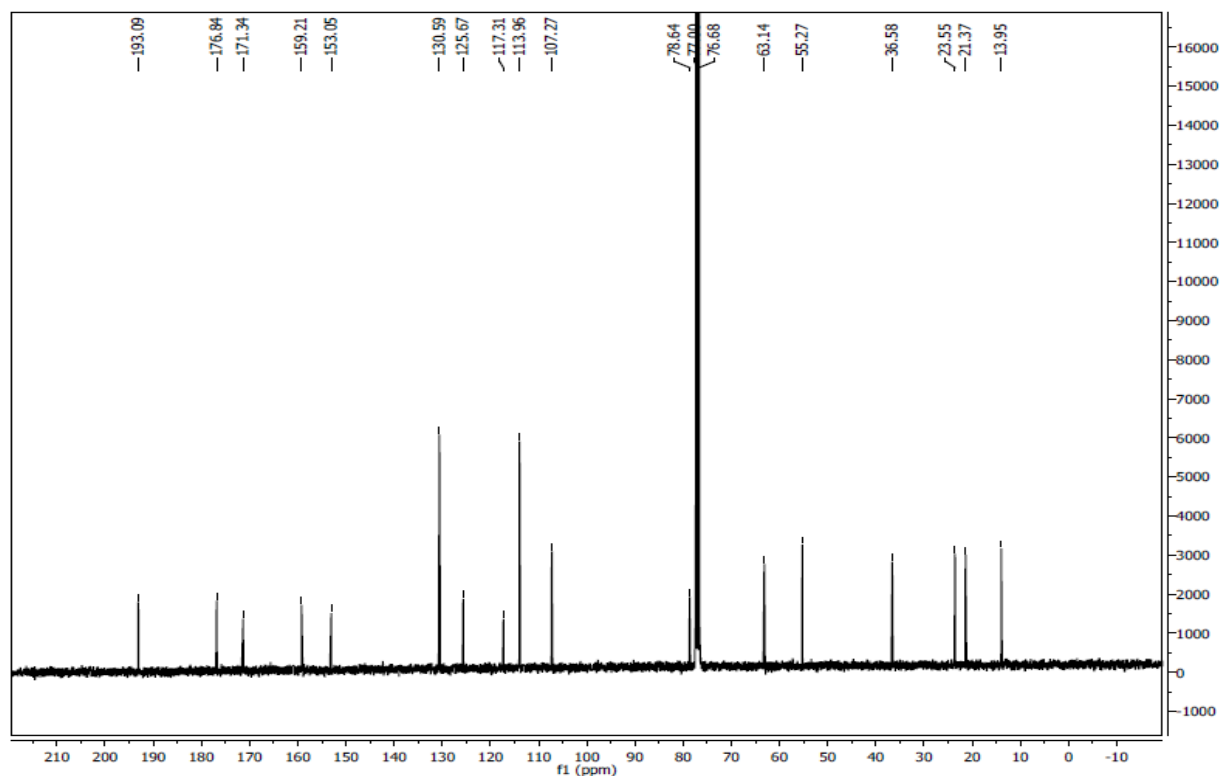
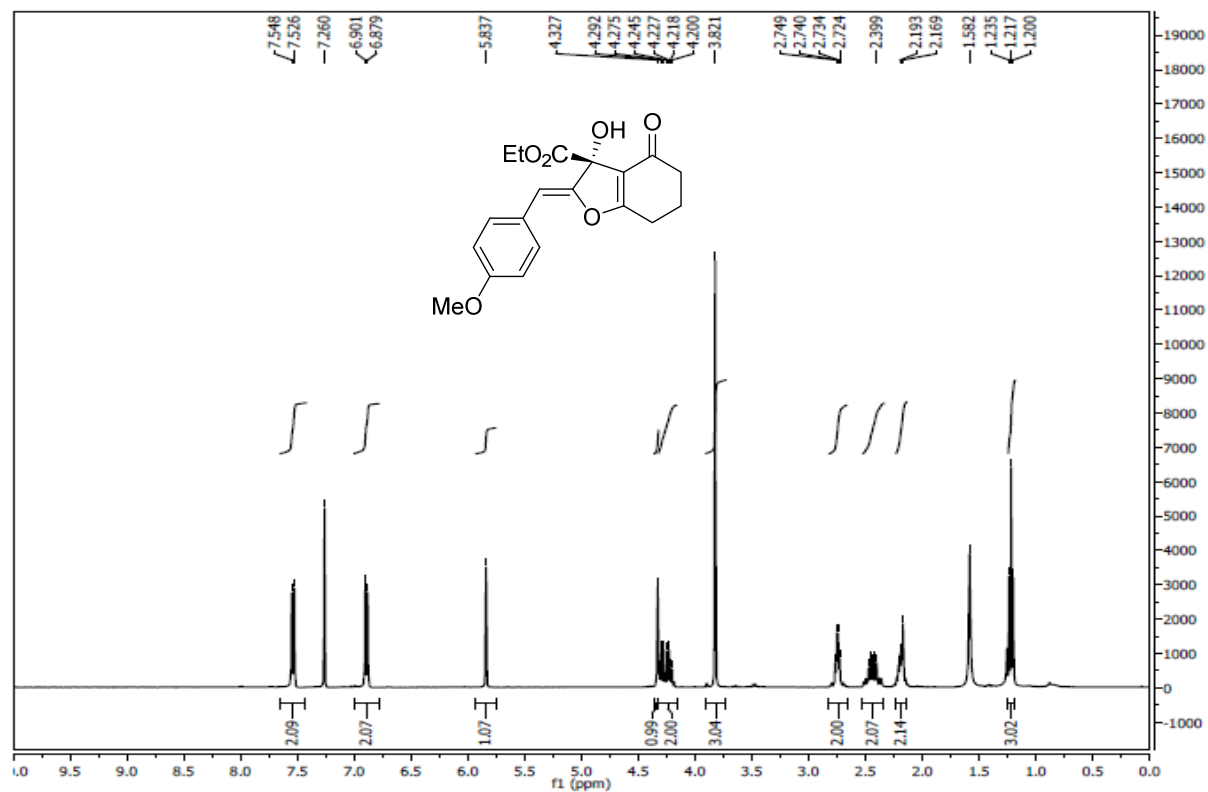


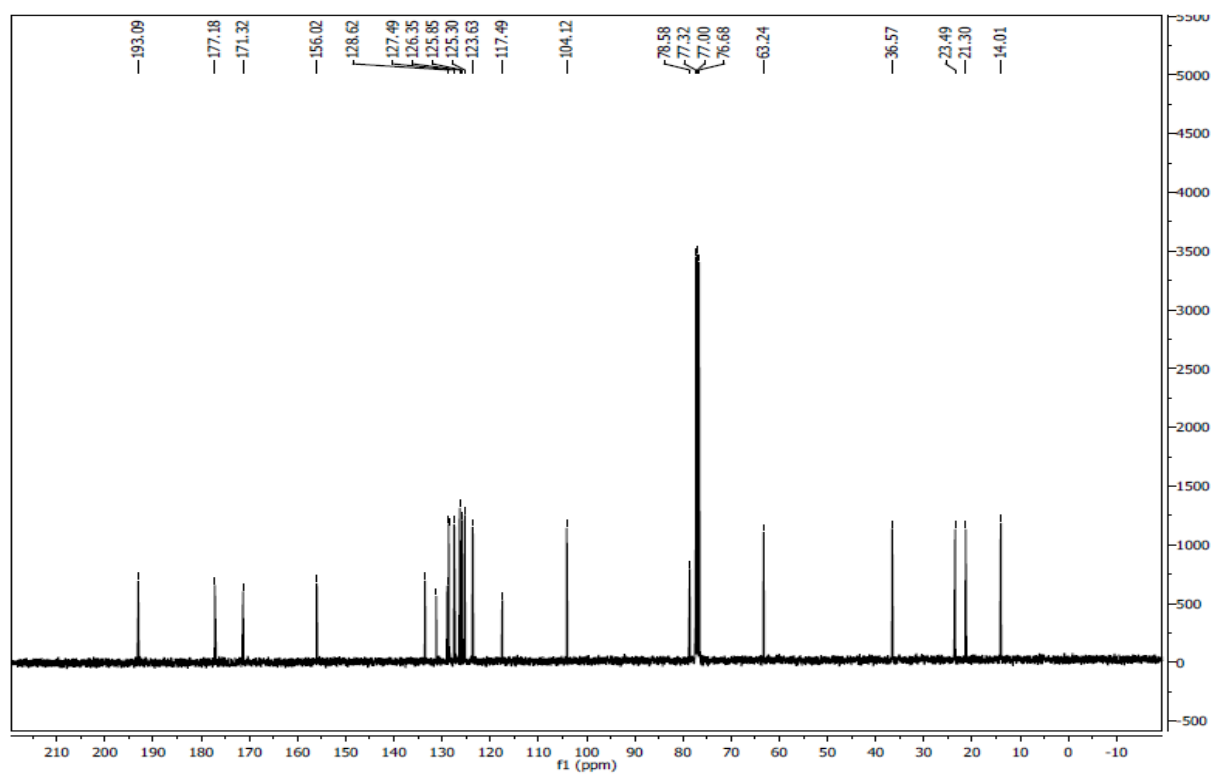
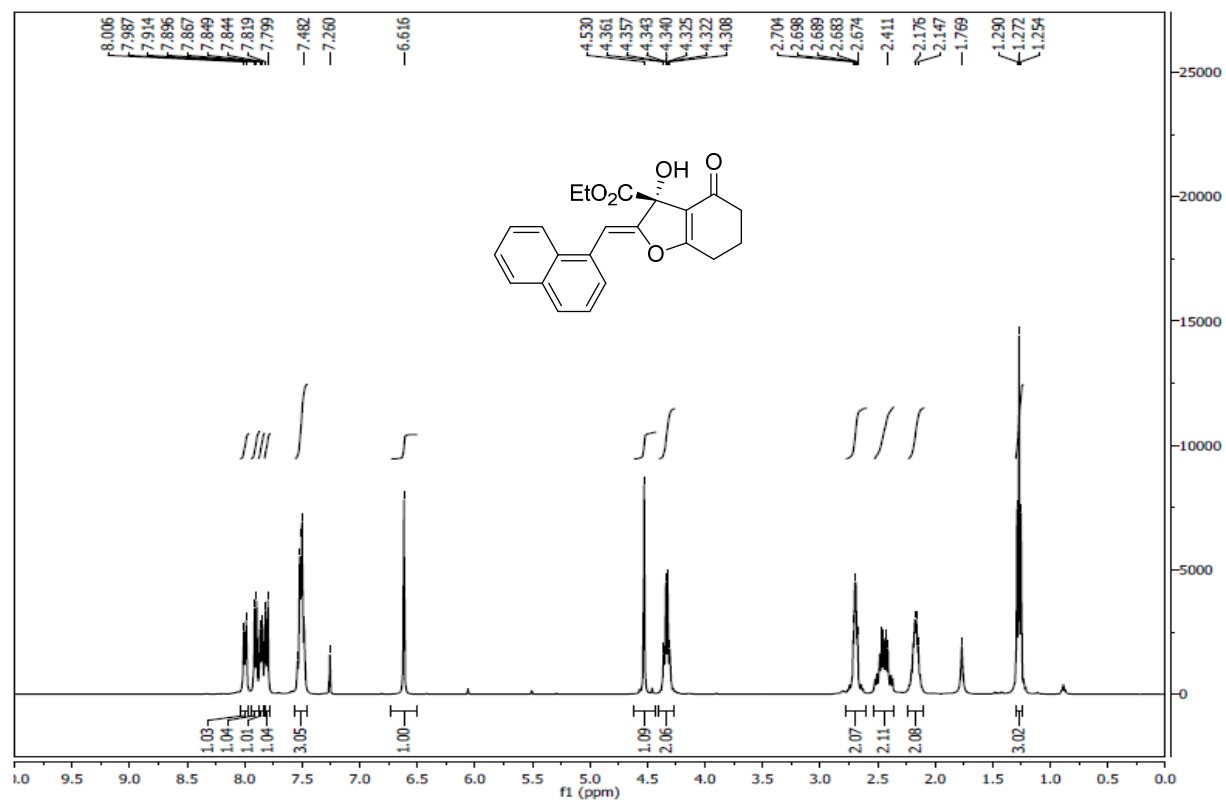


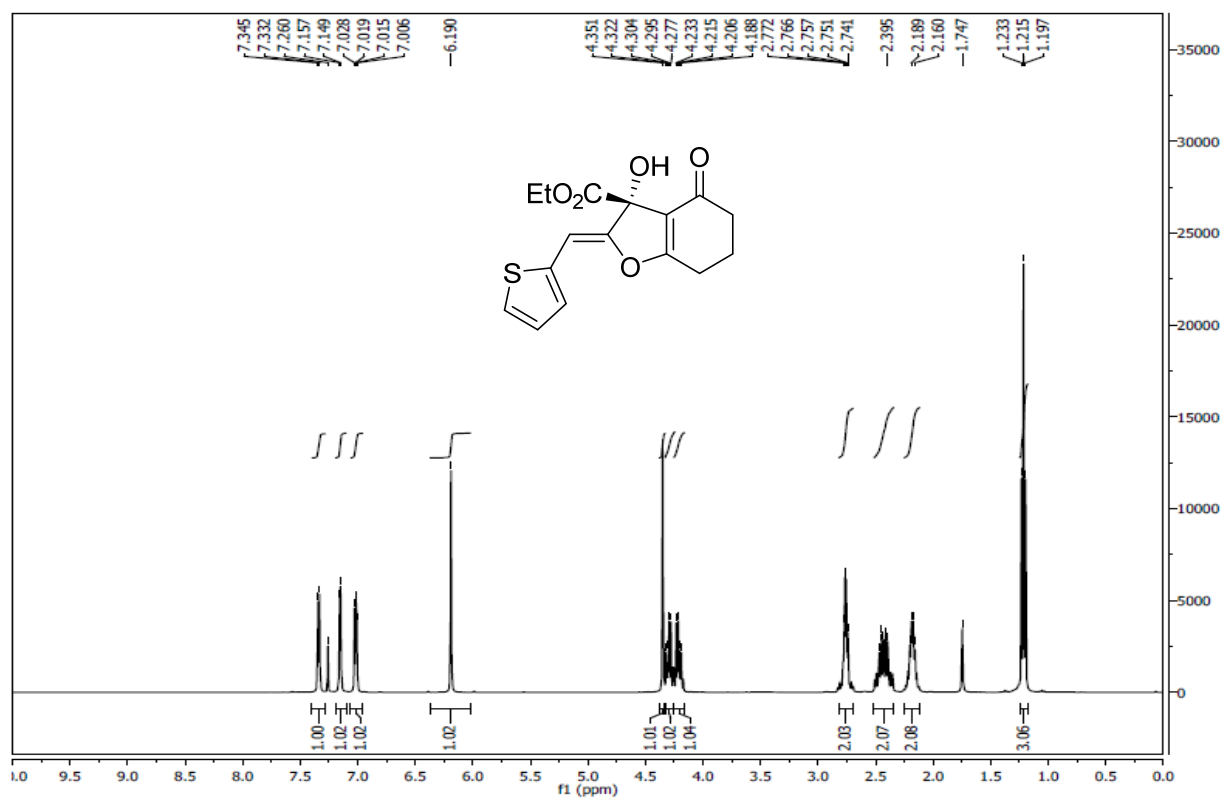
¹H NMR of Compound 4gc (400 MHz, CDCl₃)



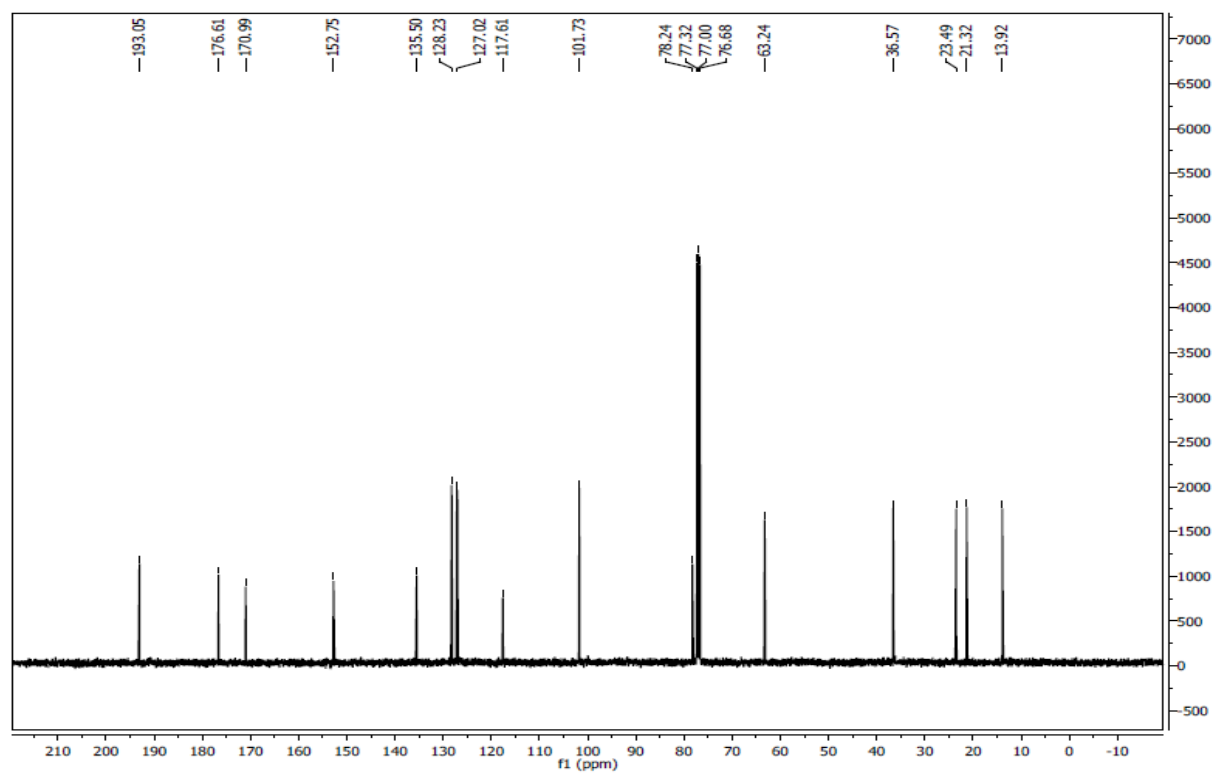
¹³C NMR of Compound 4gc (100 MHz, CDCl₃)



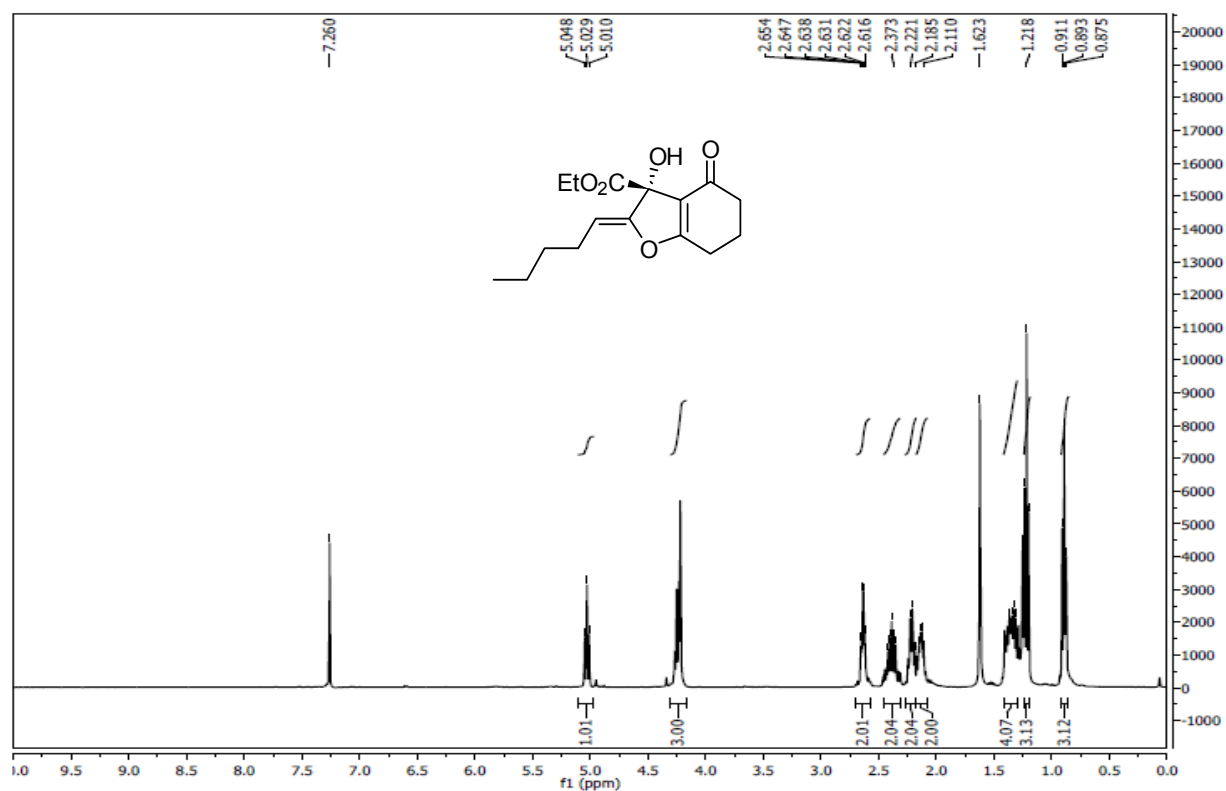




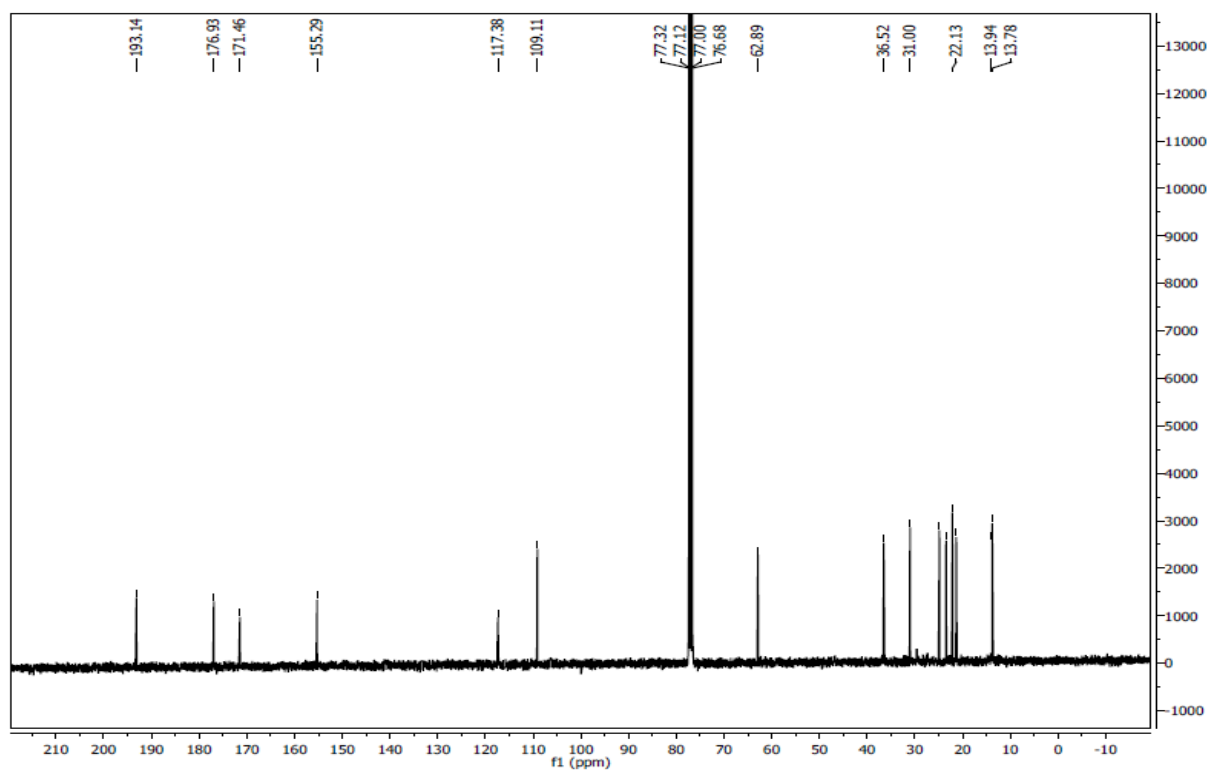
¹H NMR of Compound 4jc (400 MHz, CDCl₃)



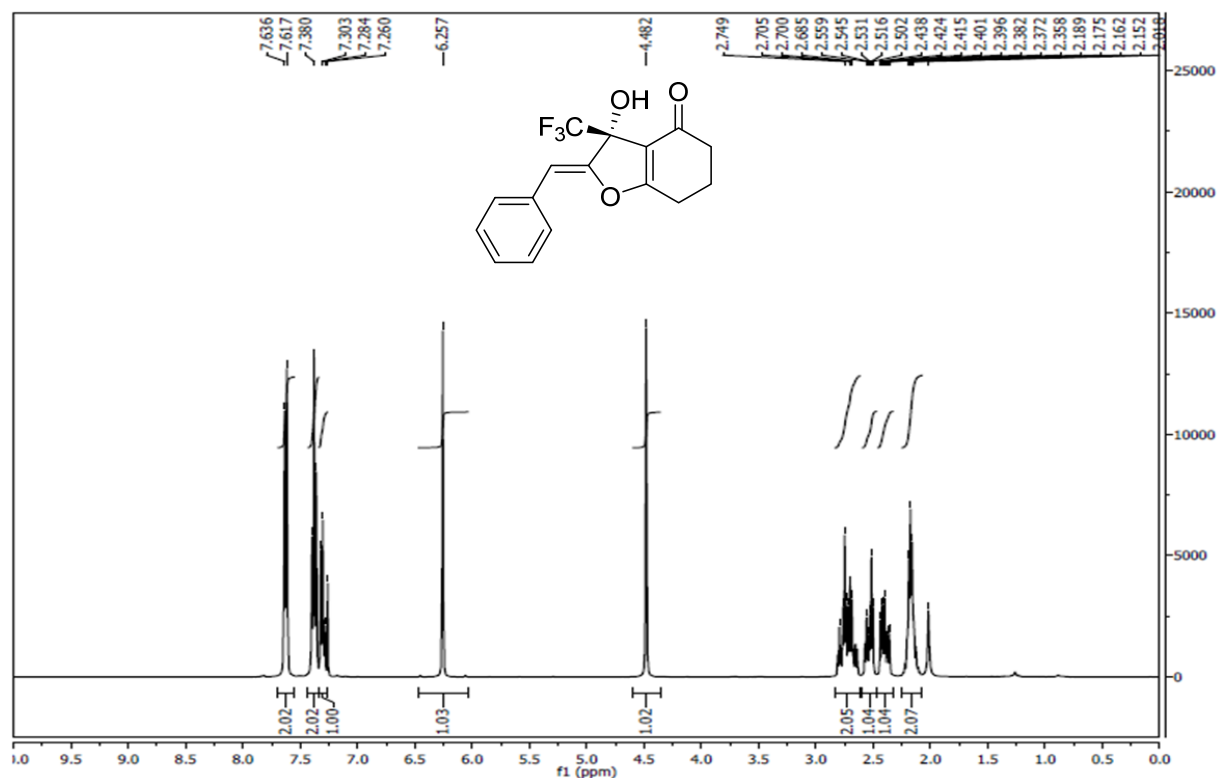
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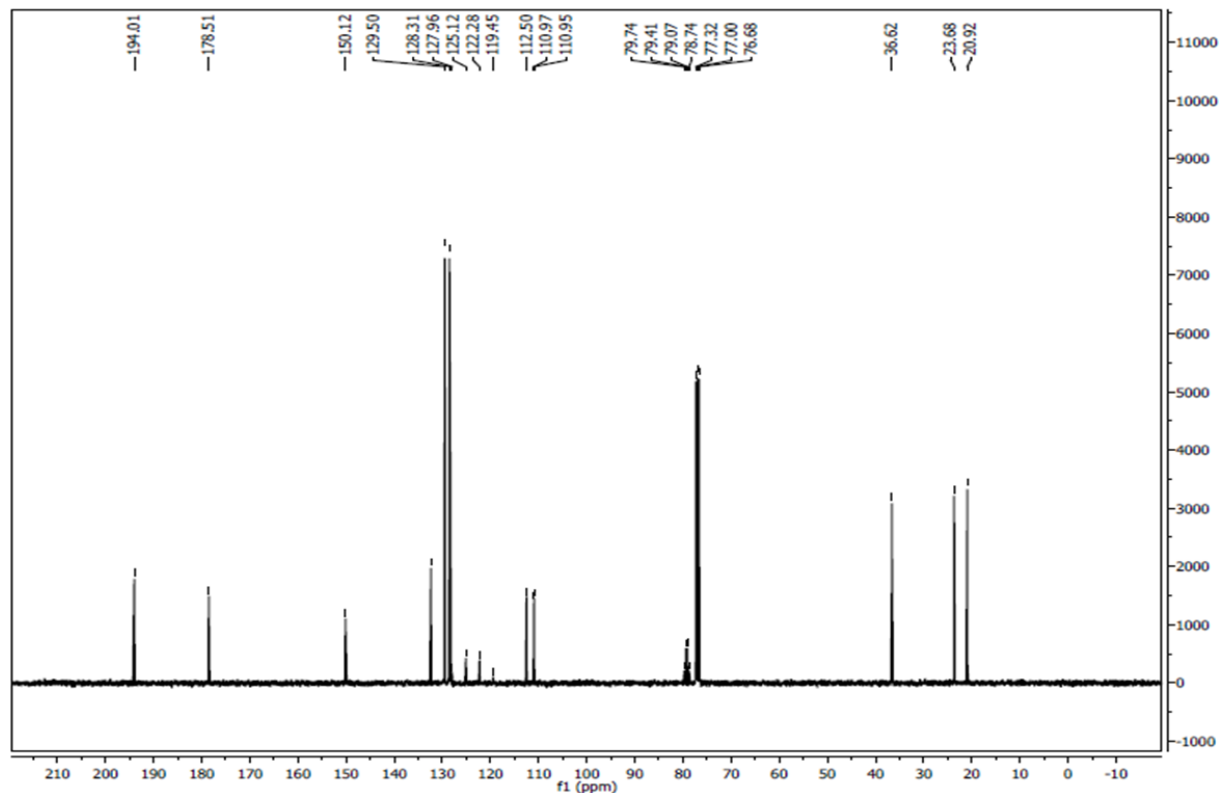
¹H NMR of Compound 4kc (400 MHz, CDCl₃)



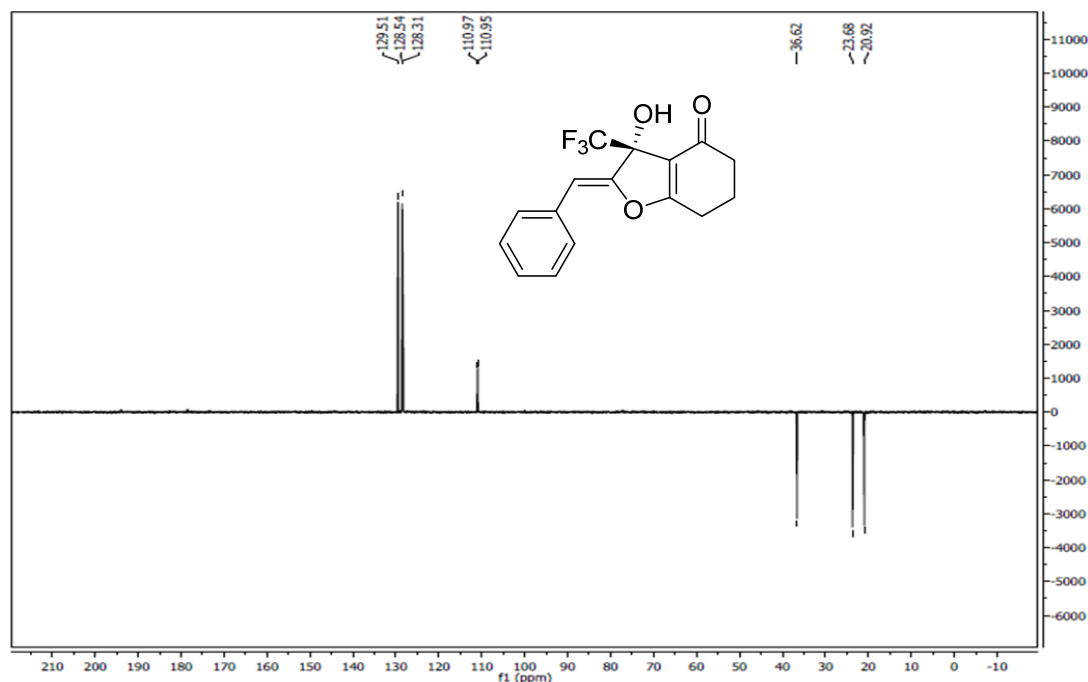
¹³C NMR of Compound 4kc (100 MHz, CDCl₃)



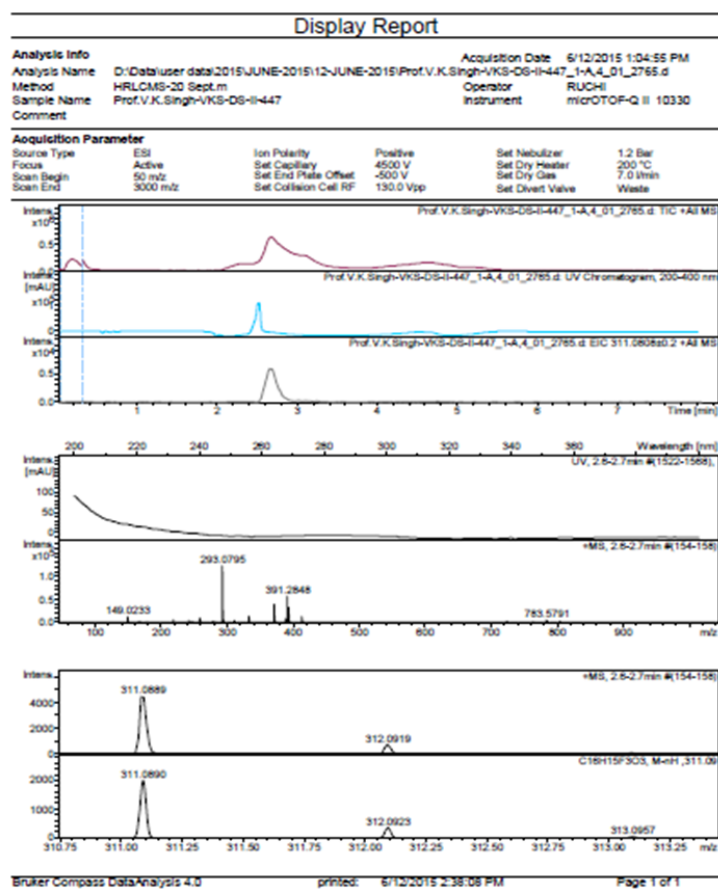
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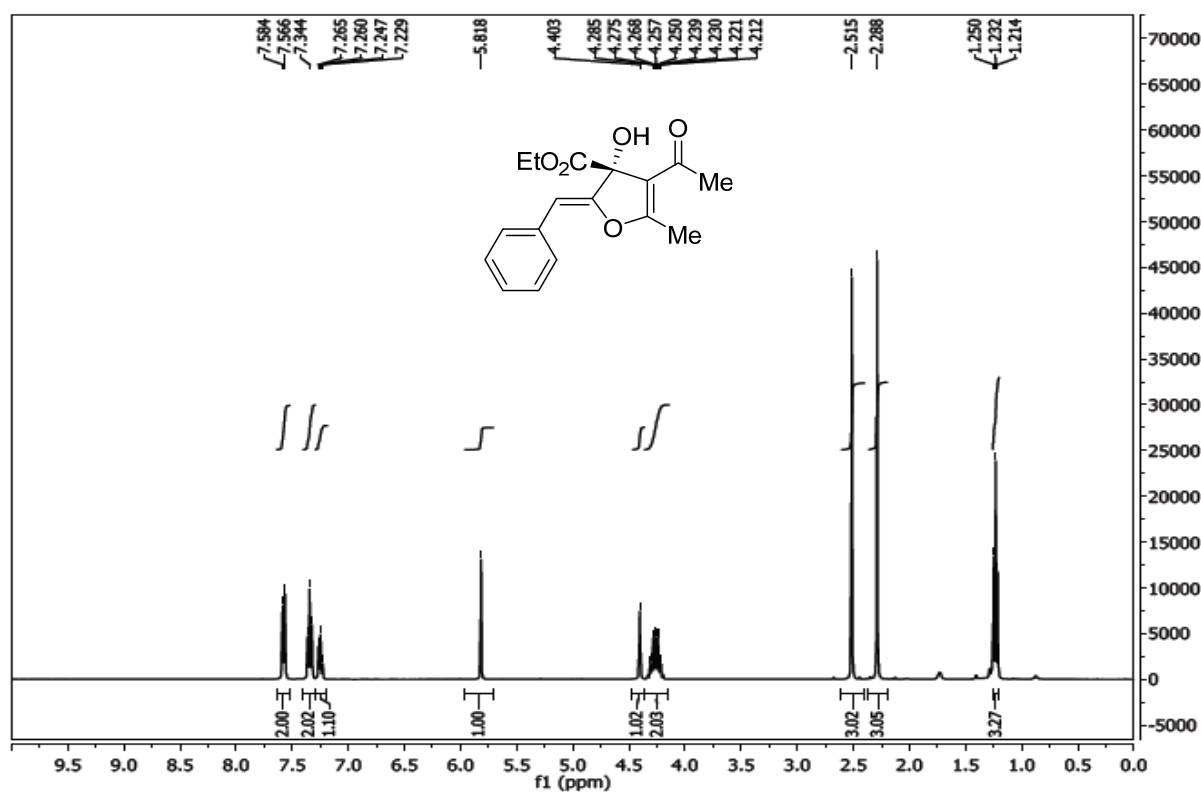
¹³C NMR of Compound 4lc (100 MHz, CDCl₃)



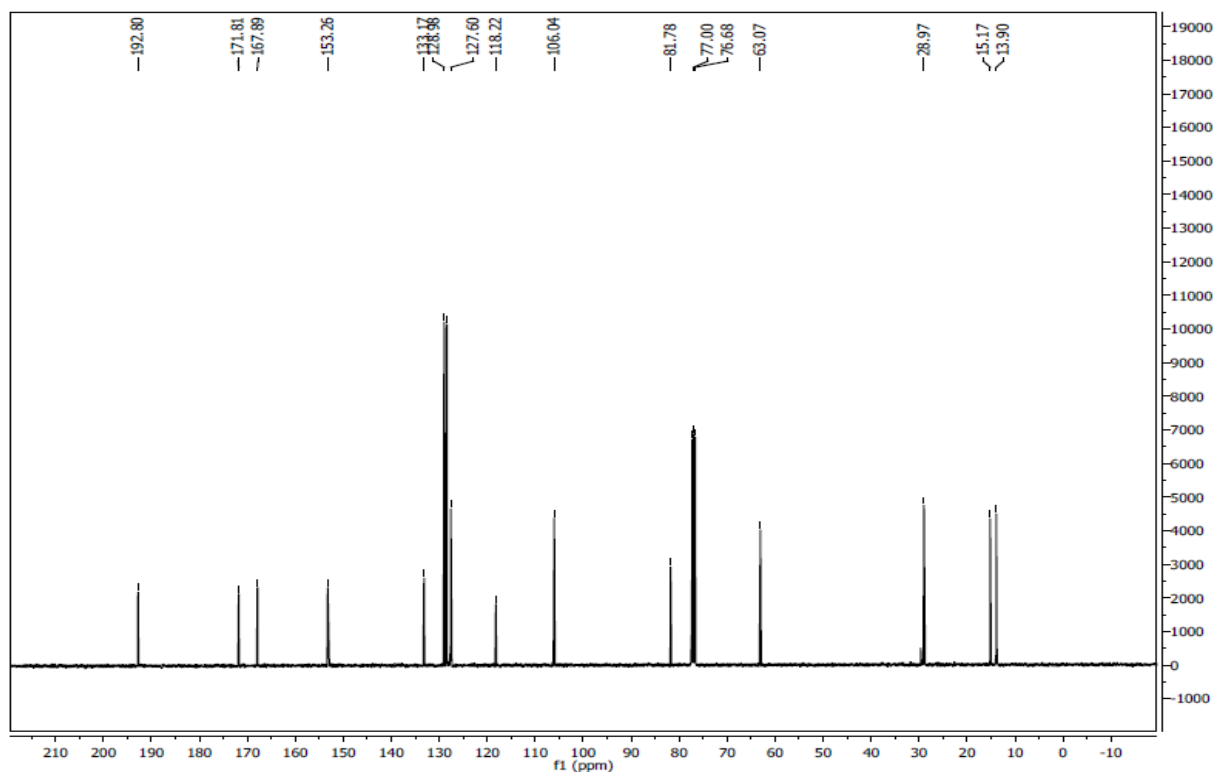
DEPT-135 of Compound 4lc (100 MHz, CDCl₃)



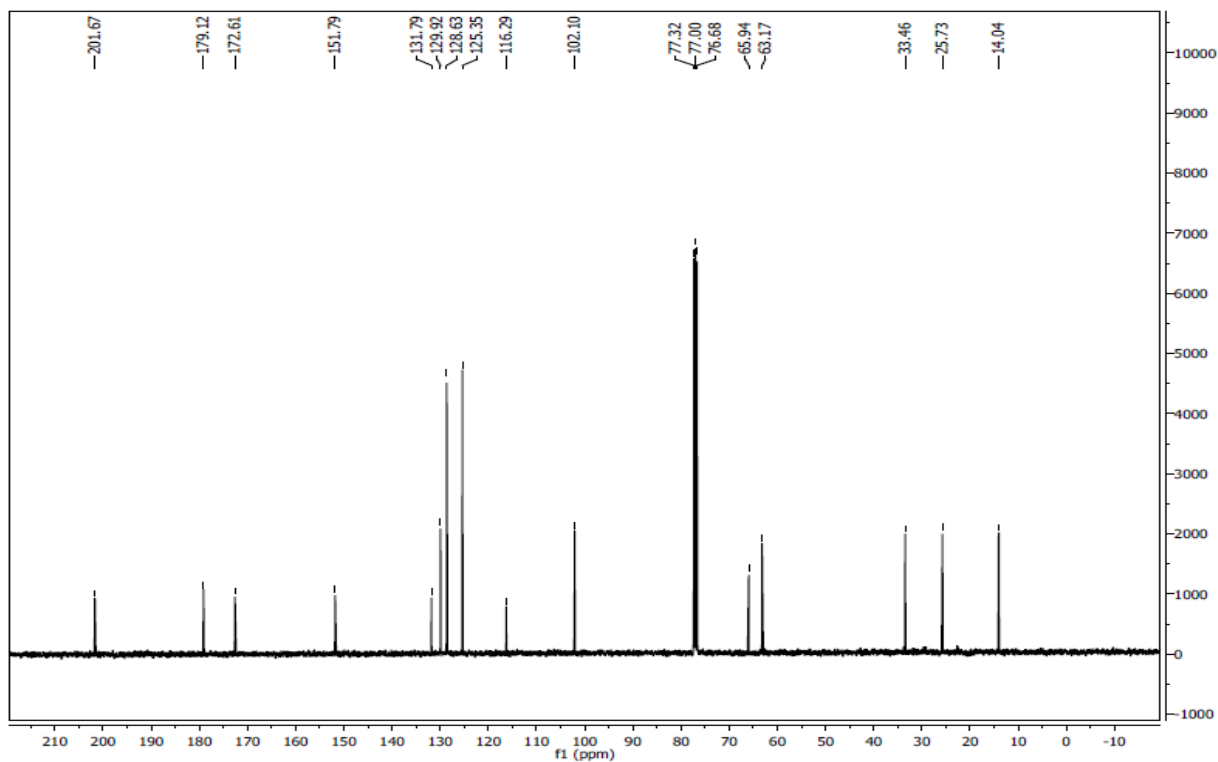
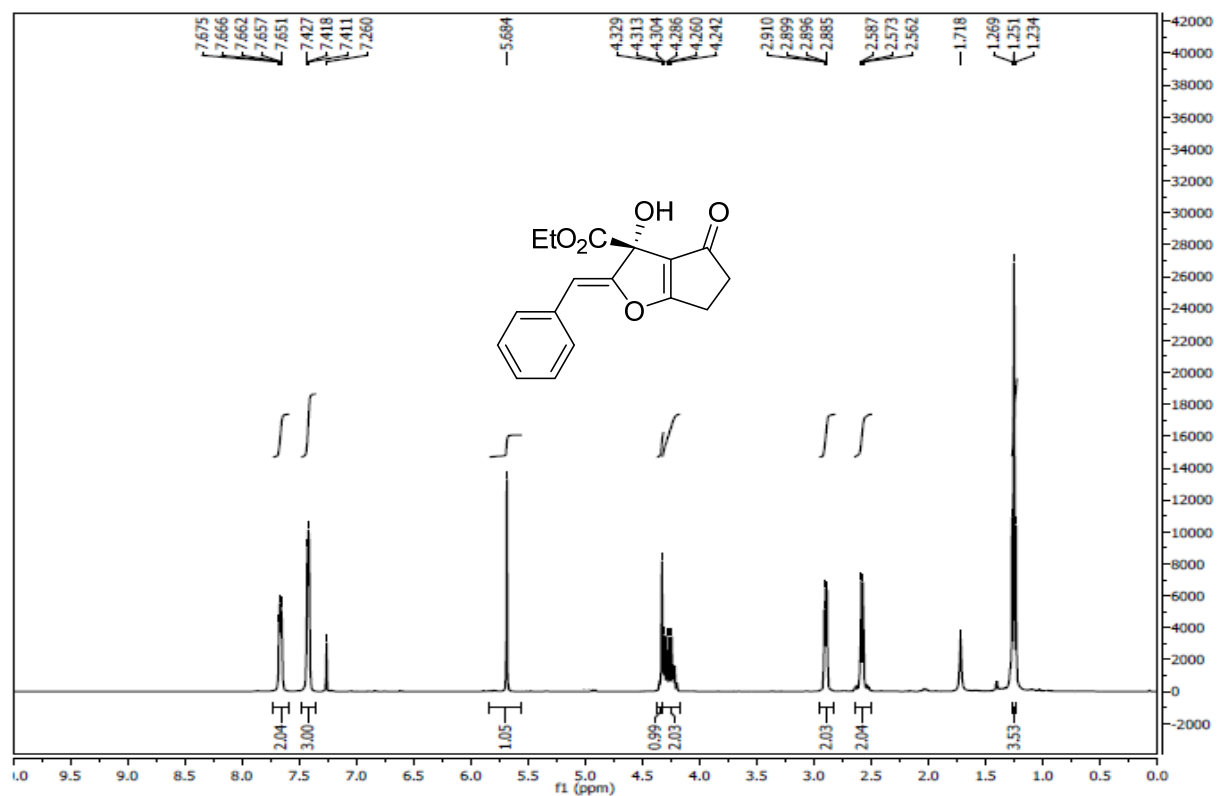
Mass Spectra of Compound 4lc (ESI)

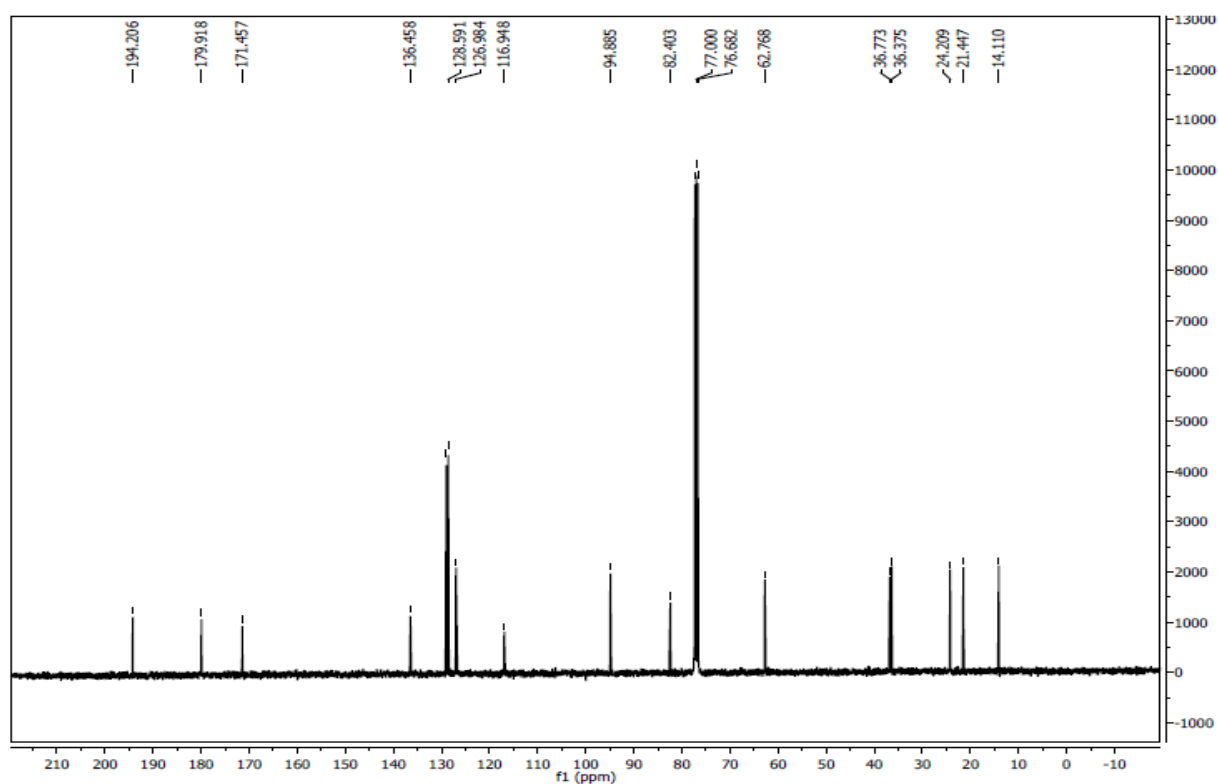
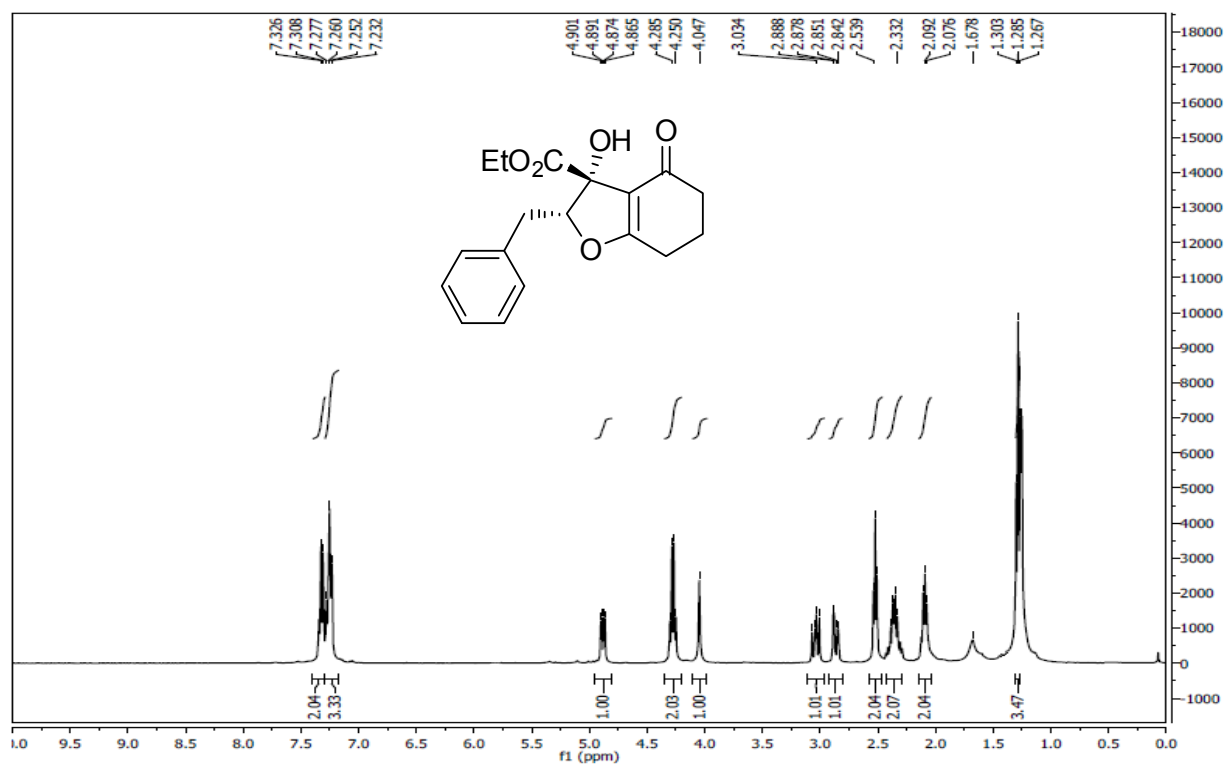


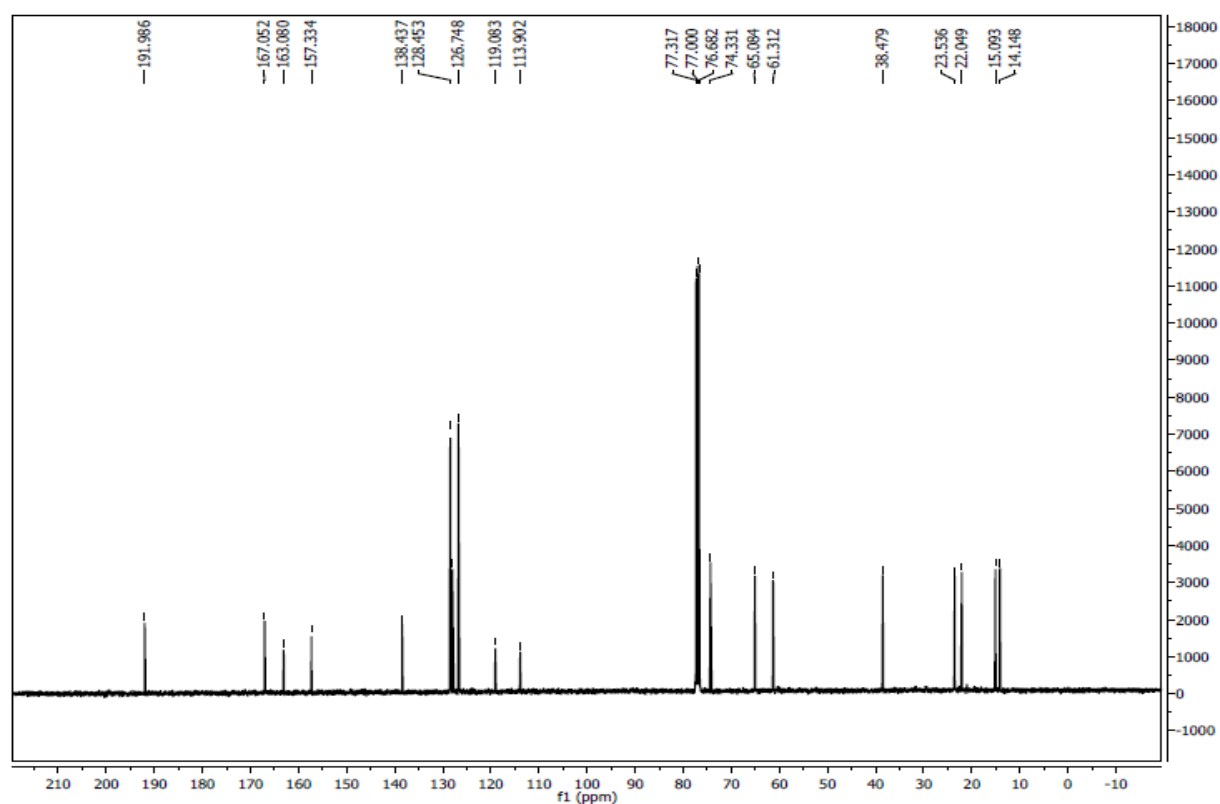
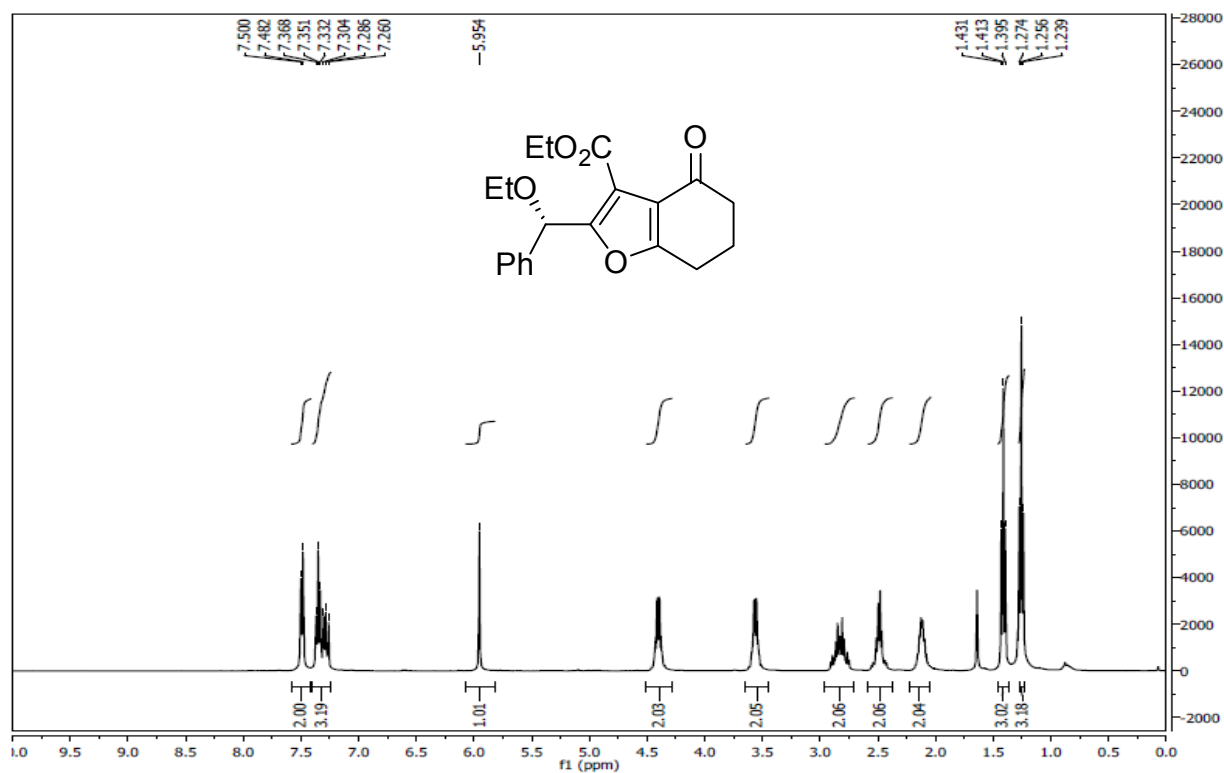
¹H NMR of Compound 4ad (400 MHz, CDCl₃)



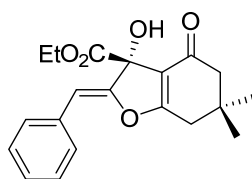
¹³C NMR of Compound 4ad (100 MHz, CDCl₃)







HPLC Chromatograms of Dihydrofuran Compounds



Compound-(±)-4aa

Data File C:\CHEM32\...ARSHI_DATA\DS-II-356-RAC-ADH-20PC-1ML-254NM-01-2014-10-2412-44-03.D
Sample Name: DS-II-356-RAC-ADH-20PC-1ML-254NM-01

Acq. Operator : SYSTEM

Location : Vial 1

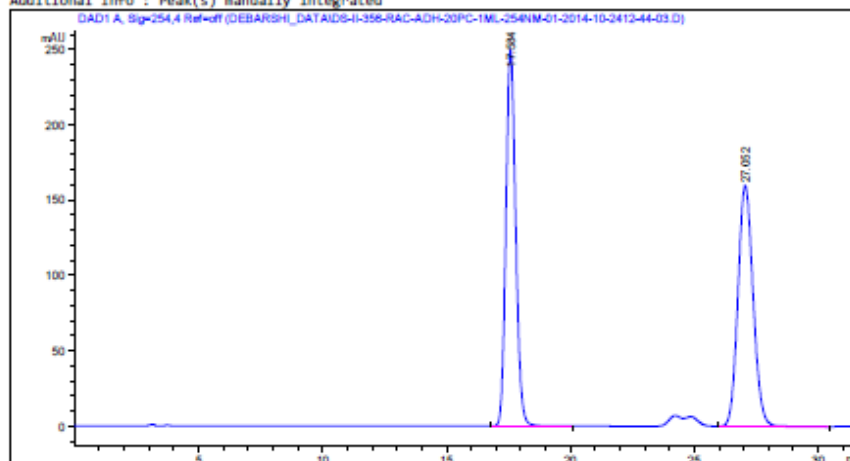
Injection Date : 24-10-2014 12:45:47

Acq. Method : 20_1_254_100.M

Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\30_1_254_100.M

Last changed : 12-07-2014 15:06:32 by SYSTEM

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

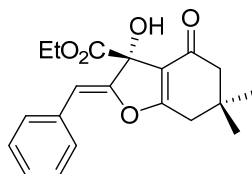
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.584	BB	0.4259	6853.52637	249.97211	50.1598
2	27.052	BB	0.6603	6809.85254	160.06598	49.8402

Totals : 1.36634e4 410.03809

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.

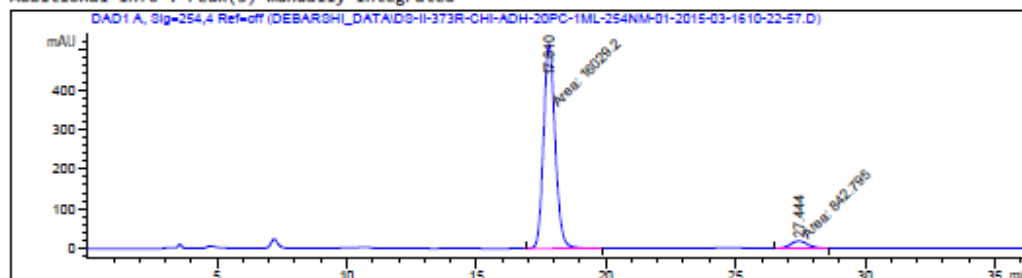


Compound-(+)-4aa

Data File C:\CHEM32\...RSHI_DATA\DS-II-373R-CHI-ADH-20PC-1ML-254NM-01-2015-03-1610-22-57.D
Sample Name: DS-II-373R-CHI-ADH-20PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 16-03-2015 10:24:41
Acq. Method    : 20_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M
Last changed   : 11-07-2014 22:47:28 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

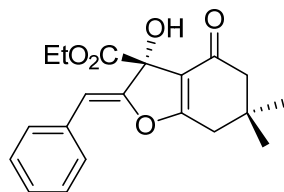
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.810	MM	0.5196	1.60292e4	514.13129	95.0048
2	27.444	MM	0.7588	842.79504	18.51275	4.9952

Totals : 1.68720e4 532.64404

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4aa

Data File C:\CHEM32\...I_DATA\DS-II-373R-ML-CHI-ADH-20PC-1ML-254NM-01-2015-03-1709-24-32.D
Sample Name: DS-II-373R-ML-CHI-ADH-20PC-1ML-254NM-01

=====

Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 17-03-2015 09:26:17

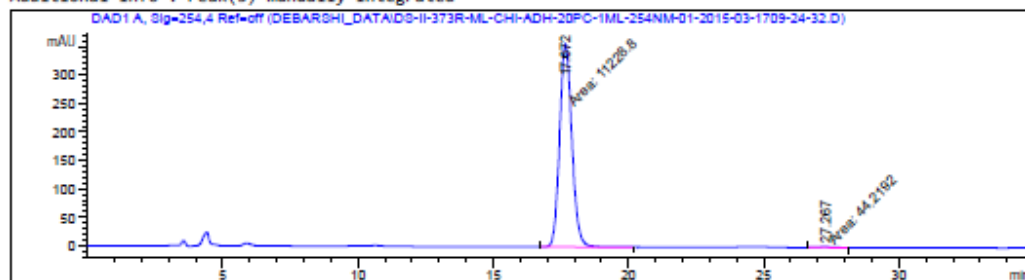
Acq. Method : 20_1_254_100.M

Analysis Method : C:\RAJSHEKHAR METHOD\10-1-254-200.M

Last changed : 11-07-2014 22:47:28 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====

Area Percent Report

=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

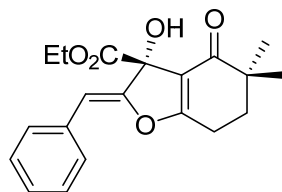
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.672	MM	0.5247	1.12288e4	356.68317	99.6077
2	27.267	MM	0.7431	44.21925	9.91746e-1	0.3923

Totals : 1.12730e4 357.67491

=====

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.

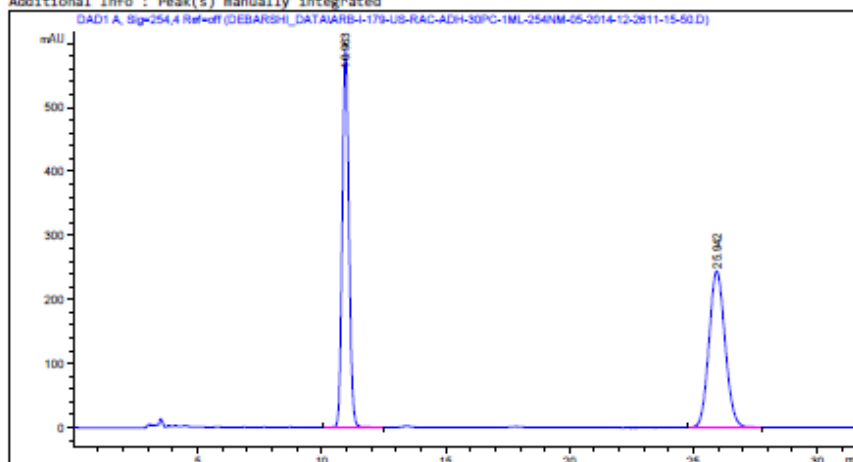


Compound-(±)-4ab

Data File C:\CHEM32\...HI_DATA\ARB-I-179-US-RAC-ADH-38PC-1ML-254NM-05-2014-12-2611-15-50.D
 Sample Name: ARB-I-179-US-RAC-ADH-38PC-1ML-254NM-05

```

=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 26-12-2014 11:17:34
Acq. Method    : 30_1_254_100.M
Analysis Method: C:\CHEM32\1\METHODS\DEBARSHI\30_1_254_100.M
Last changed   : 12-07-2014 15:06:32 by SYSTEM
Additional Info : Peak(s) manually integrated
  
```



Area Percent Report

```

=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

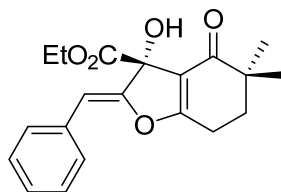
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.963	BB	0.2929	1.10358e4	588.23224	50.0996
2	25.942	BB	0.6997	1.09928e4	243.95198	49.9004

Totals : 2.20278e4 832.18422

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ab

Data File C:\CHEM32\...SHI_DATA\DS-II-378-US-CHI-ADH-30PC-1ML-254NM-012014-12-2915-11-18.D
Sample Name: DS-II-378-US-CHI-ADH-30PC-1ML-254NM-01

=====

Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 29-12-2014 15:13:04

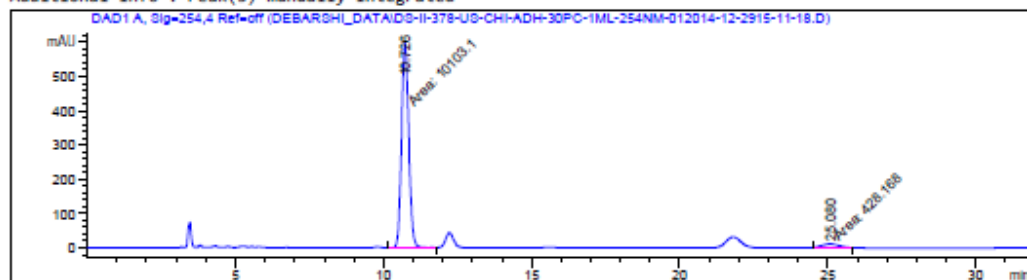
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M

Last changed : 11-07-2014 22:48:53 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====

Area Percent Report

=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

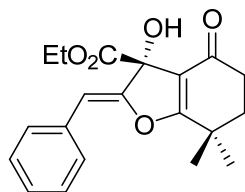
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.726	MM	0.2828	1.01031e4	595.45624	95.9343
2	25.080	MM	0.6504	428.16830	10.97117	4.0657

Totals : 1.05313e4 606.42741

=====

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4ab'

Data File C:\CHEM32\...ARSHI_DATA\ABR-I-179-RAC-ADH-30PC-1ML-254NM-01-2014-09-2514-31-52.D
Sample Name: ABR-I-179-RAC-ADH-30PC-1ML-254NM-01

=====
Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 25-09-2014 14:33:37

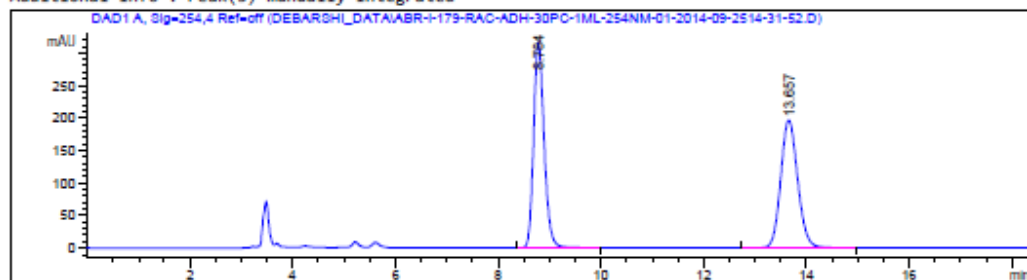
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M

Last changed : 11-07-2014 22:47:28 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====
Area Percent Report
=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

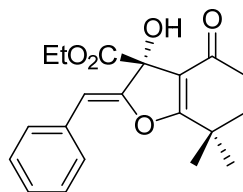
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.784	BB	0.2210	4486.61328	313.43860	50.0055
2	13.657	BB	0.3522	4485.62744	196.60342	49.9945

Totals : 8972.24072 510.04202

=====
*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ab'

Data File C:\CHEM32\...SHI_DATA\DS-II-378-LS-CHI-ADH-30PC-1ML-254NM-012014-12-2914-49-41.D
 Sample Name: DS-II-378-LS-CHI-ADH-30PC-1ML-254NM-01

=====
 Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 29-12-2014 14:51:27

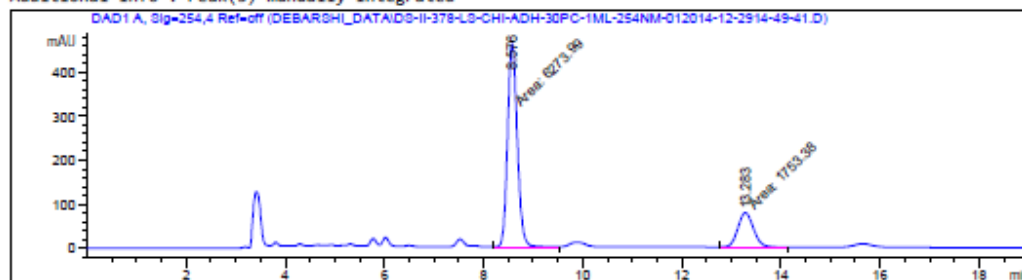
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M

Last changed : 11-07-2014 22:48:53 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====
 Area Percent Report
 =====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

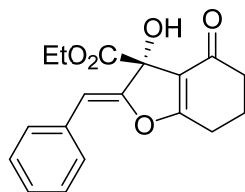
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.576	MM	0.2264	6273.99121	461.94580	78.1575
2	13.283	MM	0.3697	1753.38171	79.03529	21.8425

Totals : 8027.37292 540.98109

=====
 *** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.

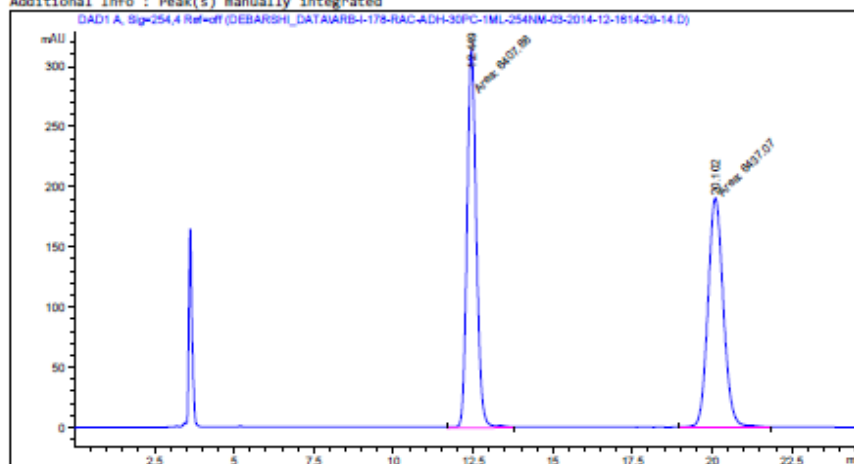


Compound-(±)-4ac

Data File C:\CHEM32\...ARSHI_DATA\ARB-I-178-RAC-ADH-38PC-1ML-254NM-03-2014-12-1614-29-14.D
Sample Name: ARB-I-178-RAC-ADH-38PC-1ML-254NM-03

Acq. Operator : SYSTEM
Location : Vial 1

Injection Date : 16-12-2014 14:38:59
Acq. Method : 30_1_254_100.M
Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\10_1_254_100.M
Last changed : 17-07-2014 19:32:48 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

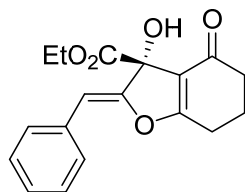
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.449	MM	0.3408	6407.88135	313.33447	49.8864
2	20.102	MM	0.5632	6437.06934	190.48563	50.1136

Totals : 1.28458e4 503.82010

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ac

Data File C:\CHEM32\...ARSHI_DATA\DS-II-361-CHI-ADH-30PC-1ML-254NM-01-2014-12-1109-31-41.D
Sample Name: DS-II-361-CHI-ADH-30PC-1ML-254NM-01

Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 11-12-2014 09:33:26

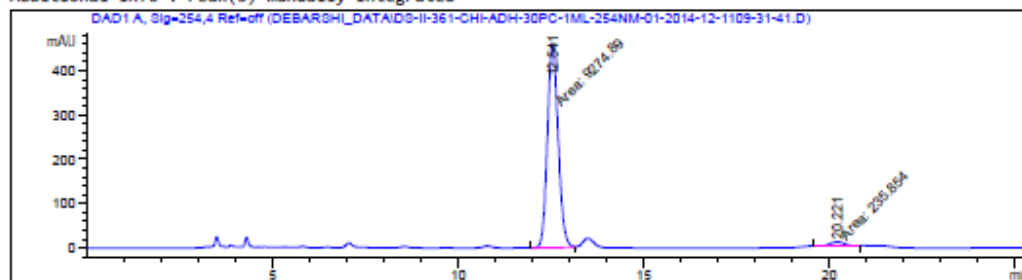
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJESHKAR METHOD\5-1-254-200.M

Last changed : 11-07-2014 22:48:53 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

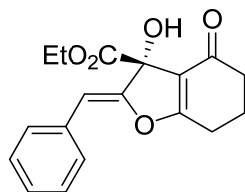
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.541	MM	0.3366	9274.88672	459.25107	97.5201
2	20.221	MM	0.4601	235.85362	8.54399	2.4799

Totals : 9510.74034 467.79506

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.

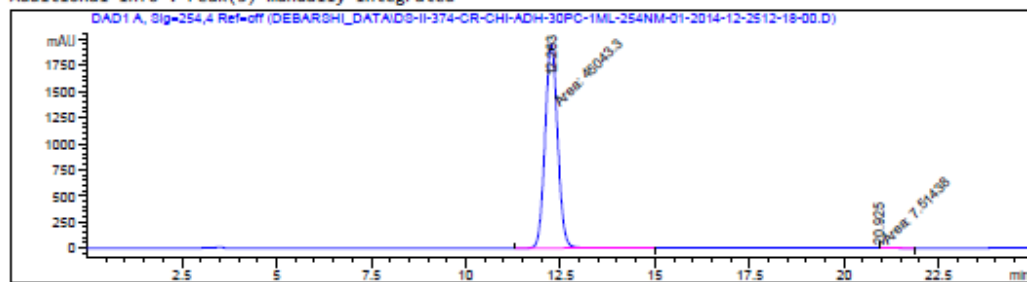


Compound-(+)-4ac

Data File C:\CHEM32\...HI_DATA\DS-II-374-CR-CHI-ADH-30PC-1ML-254NM-01-2014-12-2512-18-00.D
Sample Name: DS-II-374-CR-CHI-ADH-30PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 25-12-2014 12:19:45
Acq. Method     : 30_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\30-1-254-200.M
Last changed    : 11-07-2014 22:58:37 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

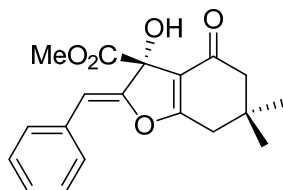
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.263	MM	0.3842	4.50433e4	1953.78674	99.9833
2	20.925	MM	0.4683	7.51438	1.88902e-1	0.0167

Totals : 4.50509e4 1953.97564

*** End of Report ***

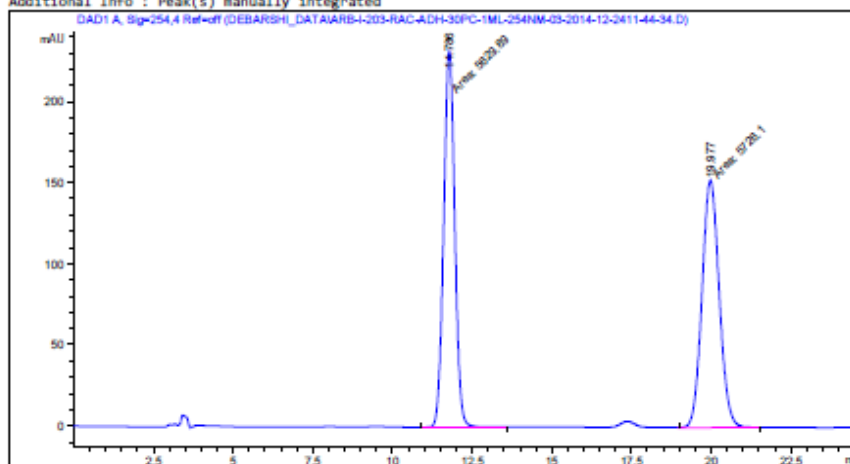
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4ba

Data File C:\CHEM32\...ARSHI_DATA\ARB-I-203-RAC-ADH-30PC-1ML-254NM-03-2014-12-2411-44-34.D
Sample Name: ARB-I-203-RAC-ADH-30PC-1ML-254NM-03

Acq. Operator : SYSTEM
Location : Vial 1
Injection Date : 24-12-2014 11:46:21
Acq. Method : 30_1_254_100.M
Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\40_1_254_100.M
Last changed : 22-07-2014 10:39:52 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

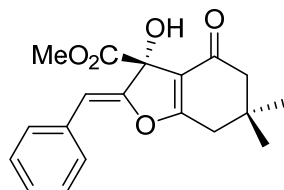
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.786	MM	0.4039	5629.89160	232.30302	49.5764
2	19.977	MM	0.6257	5726.09717	152.53506	58.4236

Totals : 1.13560e4 384.83809

*** End of Report ***

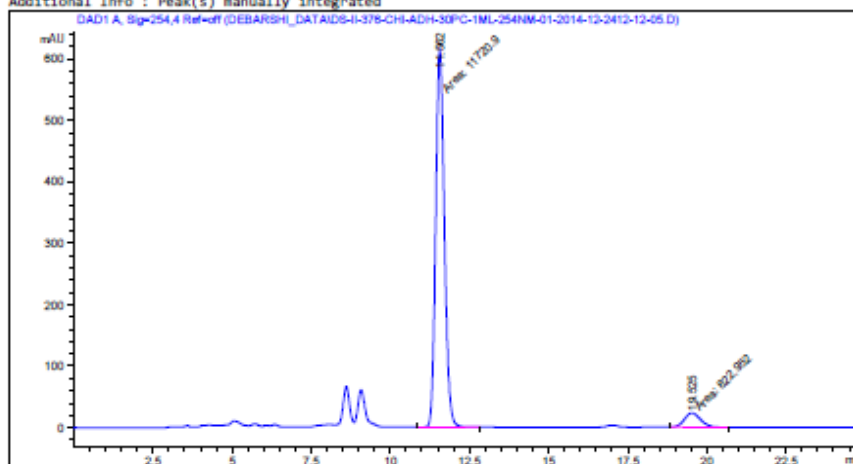
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ba

Data File C:\CHEM32\...\ARSHI_DATA\DS-II-376-CHI-ADH-38PC-1ML-254NM-01-2014-12-2412-12-05.D
Sample Name: DS-II-376-CHI-ADH-38PC-1ML-254NM-01

Acq. Operator : SYSTEM
Location : Vial 1
Injection Date : 24-12-2014 12:13:58
Acq. Method : 38_1_254_100.M
Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\48_1_254_100.M
Last changed : 22-07-2014 10:39:52 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

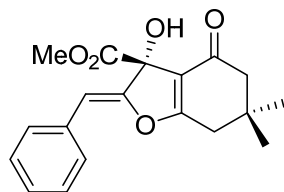
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.562	MM	0.3189	1.17209e4	612.56799	93.4394
2	19.525	MM	0.5946	822.95178	23.06701	6.5606

Totals : 1.25438e4 635.63500

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ba

Data File C:\CHEM32\...HI_DATA\DS-II-376-ML-CHI-ADH-30PC-1ML-254NM-02-2015-01-0811-18-32.D
Sample Name: DS-II-376-ML-CHI-ADH-30PC-1ML-254NM-02

=====
Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 08-01-2015 11:20:18

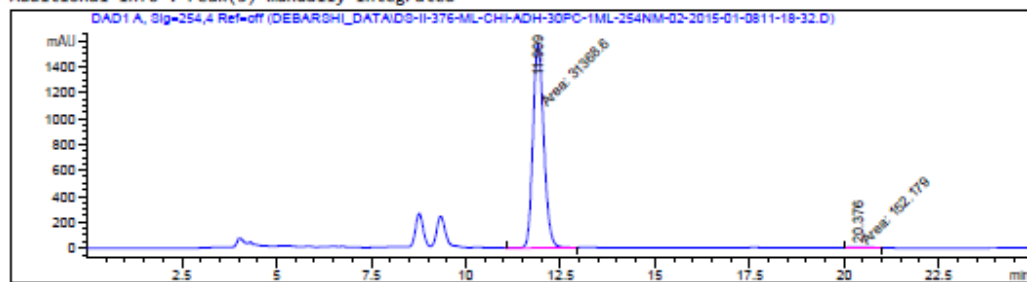
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJESHKAR METHOD\30-1-254-200.M

Last changed : 11-07-2014 22:58:37 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====
Area Percent Report
=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

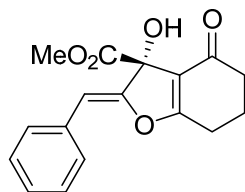
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.909	MM	0.3319	3.13686e4	1575.00049	99.5172
2	20.376	MM	0.5909	152.17894	4.29229	0.4828

Totals : 3.15208e4 1579.29278

=====
*** End of Report ***

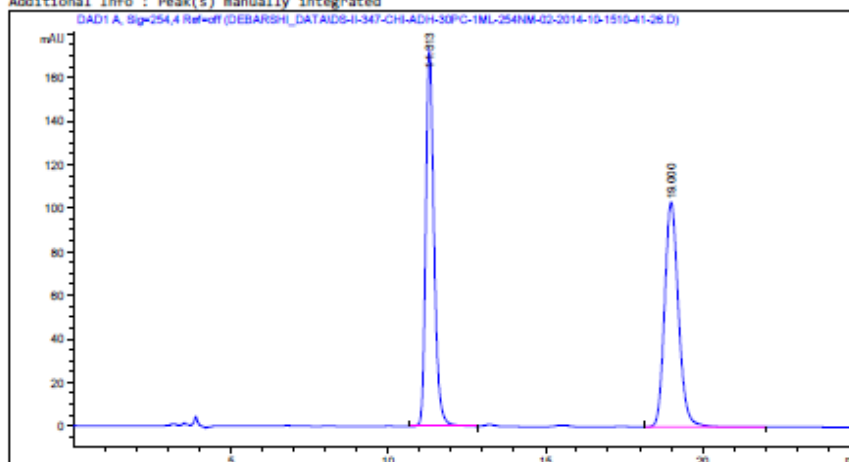
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4bc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-347-CHI-ADH-38PC-1ML-254NM-02-2014-10-1510-41-26.D
Sample Name: DS-II-347-CHI-ADH-38PC-1ML-254NM-02

Acq. Operator : SYSTEM
Location : Vial 1
Injection Date : 15-10-2014 10:43:10
Acq. Method : 30_1_254_100.M
Analysis Method : C:\NAGARAJU\METHODS\40-1-254-100 MIN.M
Last changed : 13-07-2014 16:44:57 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

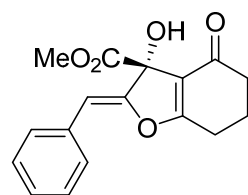
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.313	BB	0.2798	3161.79761	172.51352	50.0366
2	19.000	BB	0.4731	3157.17334	103.13749	49.9634

Totals : 6318.97095 275.65101

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4bc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-354-CHI-ADH-30PC-1ML-254NM-01-2014-10-2718-44-32.D
Sample Name: DS-II-354-CHI-ADH-30PC-1ML-254NM-01

Acq. Operator : SYSTEM

Location : Vial 1

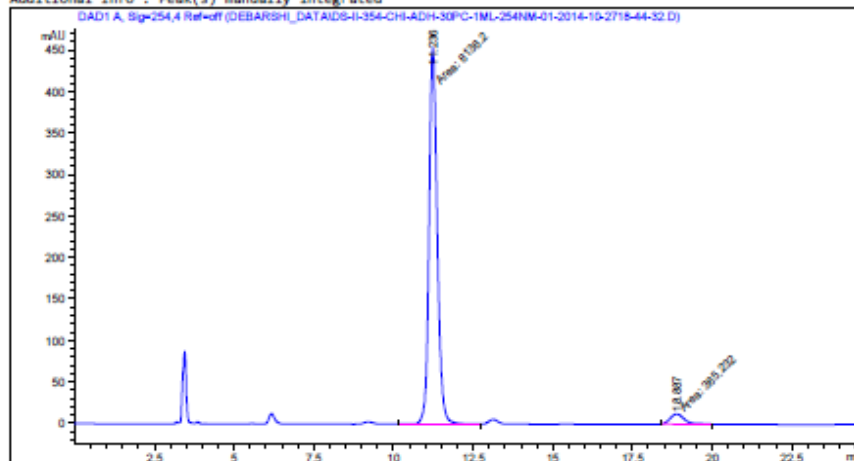
Injection Date : 27-10-2014 18:46:16

Acq. Method : 30_1_254_100.M

Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\10_1_254_100.M

Last changed : 17-07-2014 19:32:48 by SYSTEM

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

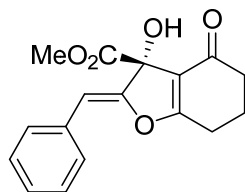
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.236	MM	0.2989	8136.20410	453.74030	95.4793
2	18.887	MM	0.5194	385.23215	12.36060	4.5207

Totals : 8521.43625 466.10090

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4bc

Data File C:\CHEM32\...HI_DATA\DS-II-375-CR-CHI-ADH-30PC-1ML-254NM-01-2014-12-1915-59-54.D
Sample Name: DS-II-375-CR-CHI-ADH-30PC-1ML-254NM-01

Acq. Operator : SYSTEM

Location : Vial 1

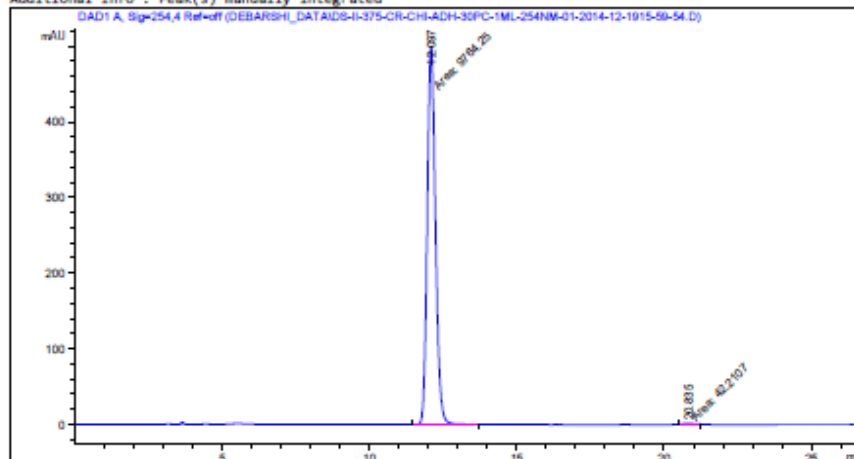
Injection Date : 19-12-2014 16:01:39

Acq. Method : 30_1_254_100.M

Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\20_1_254_100.M

Last changed : 21-07-2014 13:58:26 by SYSTEM

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

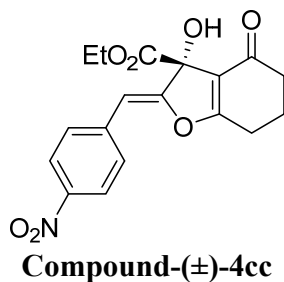
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.097	MM	0.3264	9764.25488	498.59076	99.5696
2	20.835	MM	0.4793	42.21871	1.46779	0.4304

Totals : 9806.46559 500.05855

*** End of Report ***

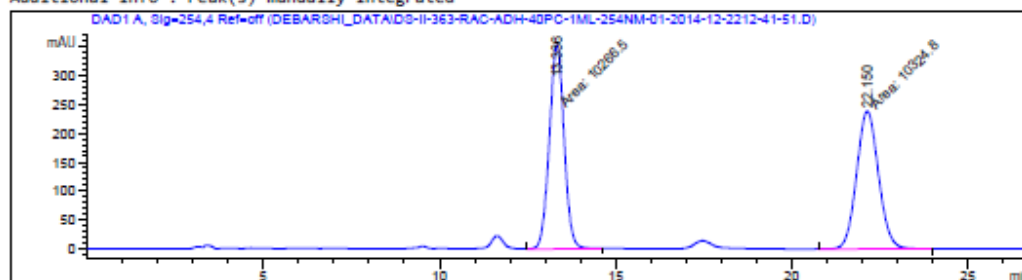
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\DS-II-363-RAC-ADH-40PC-1ML-254NM-01-2014-12-2212-41-51.D
Sample Name: DS-II-363-RAC-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 22-12-2014 12:43:37
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M
Last changed   : 11-07-2014 22:48:53 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



```
=====
                          Area Percent Report
=====

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

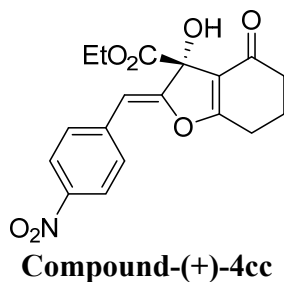
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.336	MM	0.4847	1.02665e4	353.02139	49.8584
2	22.150	MM	0.7218	1.03248e4	238.40982	50.1416

Totals : 2.05913e4 591.43121

```
=====
*** End of Report ***
```

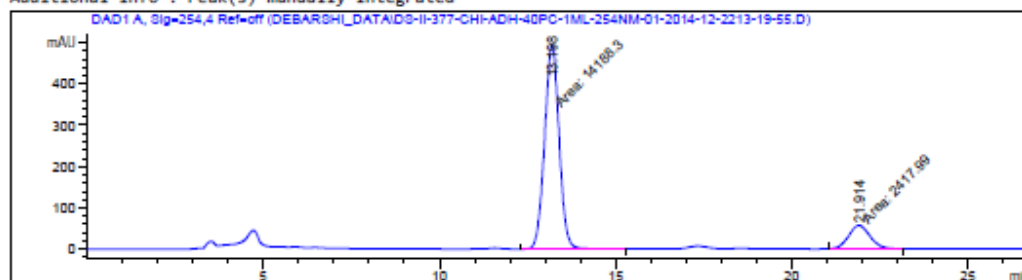
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\DS-II-377-CHI-ADH-40PC-1ML-254NM-01-2014-12-2213-19-55.D
 Sample Name: DS-II-377-CHI-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 22-12-2014 13:21:42
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M
Last changed   : 11-07-2014 22:48:53 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

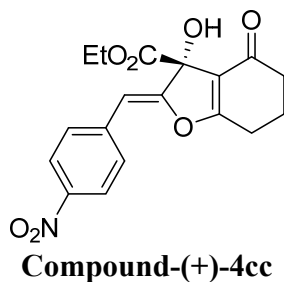
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.198	MM	0.4784	1.41883e4	494.33261	85.4393
2	21.914	MM	0.7118	2417.99023	56.61489	14.5607

Totals : 1.66063e4 550.94750

*** End of Report ***

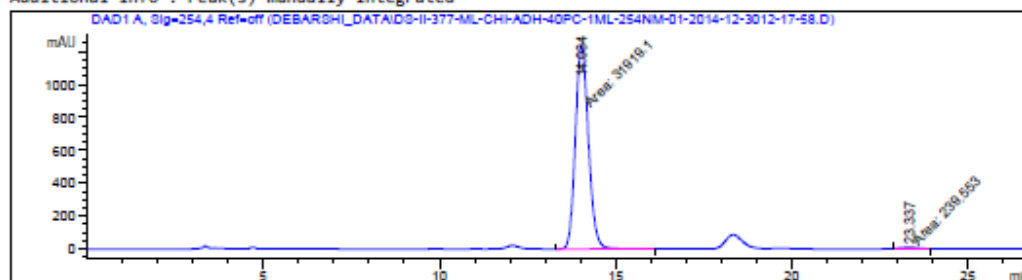
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...HI_DATA\DS-II-377-ML-CHI-ADH-40PC-1ML-254NM-01-2014-12-3012-17-58.D
 Sample Name: DS-II-377-ML-CHI-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 30-12-2014 12:19:44
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M
Last changed   : 11-07-2014 22:48:53 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



=====
 Area Percent Report
 =====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

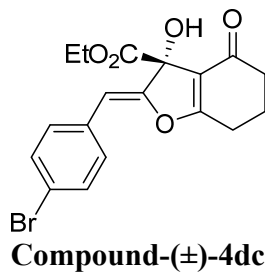
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.034	MM	0.4300	3.19191e4	1237.25110	99.2551
2	23.337	MM	0.5815	239.55321	6.86607	0.7449

Totals : 3.21587e4 1244.11716

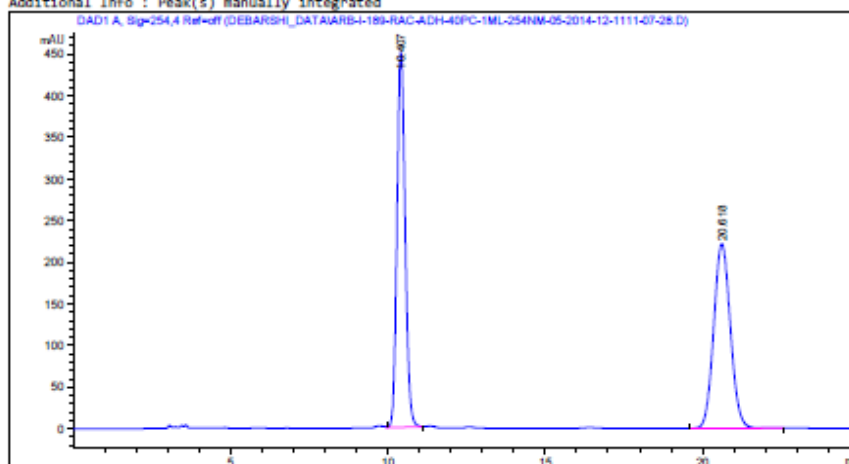
=====
 *** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\ARB-I-189-RAC-ADH-48PC-1ML-254NM-05-2014-12-1111-07-28.D
 Sample Name: ARB-I-189-RAC-ADH-48PC-1ML-254NM-05

 Acq. Operator : SYSTEM
 Location : Vial 1
 Injection Date : 11-12-2014 11:09:11
 Acq. Method : 48_1_254_100.M
 Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\18_1_254_100.M
 Last changed : 17-07-2014 19:32:48 by SYSTEM
 Additional Info : Peak(s) manually integrated



 Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

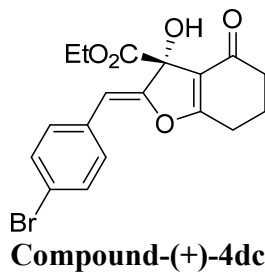
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.407	VB	0.2776	8103.91797	451.14810	49.8068
2	20.618	BB	0.5737	8166.78857	221.75168	50.1932

Totals : 1.62707e4 672.89978

 *** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.

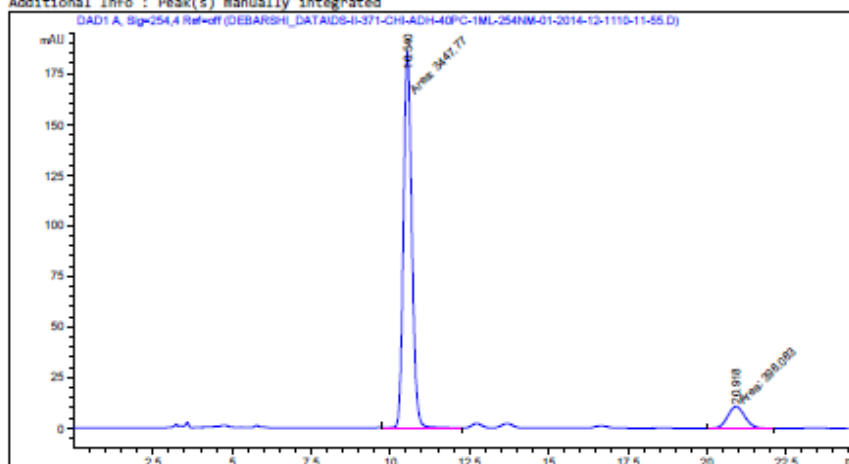


Data File C:\CHEM32\...ARSHI_DATA\DS-II-371-CHI-ADH-48PC-1ML-254NM-01-2014-12-1110-11-55.D
Sample Name: DS-II-371-CHI-ADH-48PC-1ML-254NM-01

```

=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 11-12-2014 10:13:42
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\10_1_254_100.M
Last changed    : 17-07-2014 19:32:48 by SYSTEM
Additional Info : Peak(s) manually integrated
=====

```



Area Percent Report

```

=====
Sorted By       : Signal
Multiplier      : 1.0000
Dilution        : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====

```

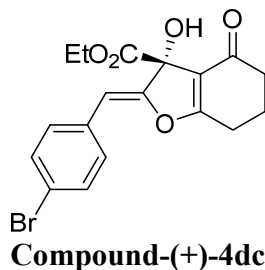
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.540	MM	0.3090	3447.76904	185.96625	89.6490
2	20.918	MM	0.6175	398.08258	10.74474	10.3510

Totals : 3845.85162 196.71099

*** End of Report ***

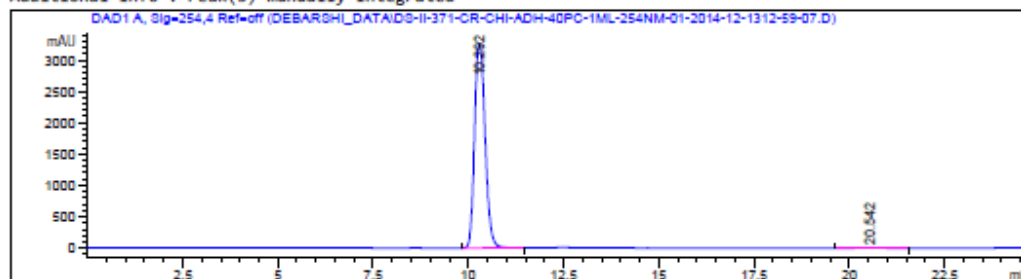
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...HI_DATA\DS-II-371-CR-CHI-ADH-40PC-1ML-254NM-01-2014-12-1312-59-07.D
 Sample Name: DS-II-371-CR-CHI-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 13-12-2014 13:00:51
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\30-1-254-200.M
Last changed   : 11-07-2014 22:58:37 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

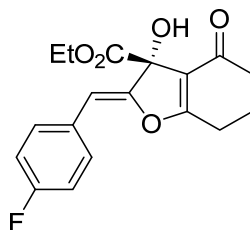
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.292	BB	0.3033	6.29054e4	3256.87036	99.7039
2	20.542	BB	0.5724	186.81763	5.04113	0.2961

Totals : 6.30922e4 3261.91149

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.

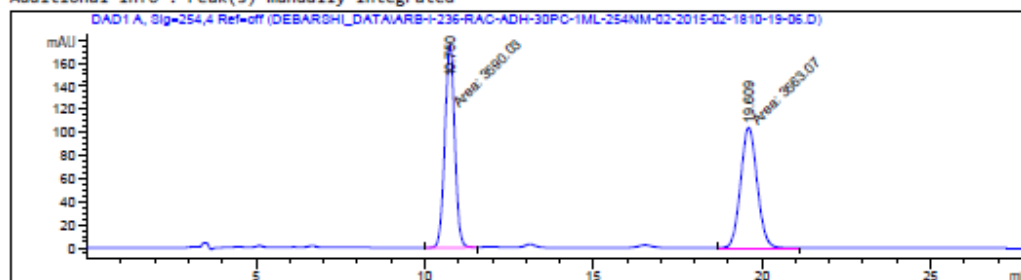


Compound-(±)-4ec

Data File C:\CHEM32\...ARSHI_DATA\ARB-I-236-RAC-ADH-30PC-1ML-254NM-02-2015-02-1810-19-06.D
Sample Name: ARB-I-236-RAC-ADH-30PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 18-02-2015 10:20:52
Acq. Method     : 30_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M
Last changed    : 11-07-2014 22:47:28 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

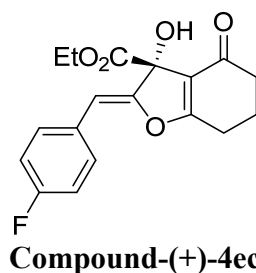
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.750	MM	0.3409	3590.02881	175.53738	50.1885
2	19.609	MM	0.5686	3563.06567	104.43810	49.8115

Totals : 7153.09448 279.97548

*** End of Report ***

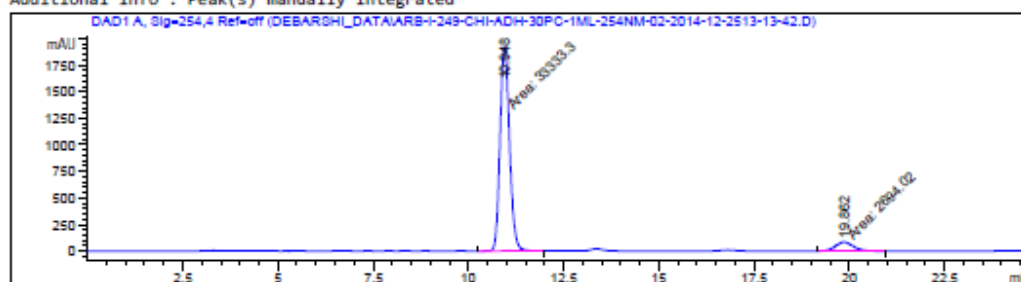
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\ARB-I-249-CHI-ADH-30PC-1ML-254NM-02-2014-12-2513-13-42.D
Sample Name: ARB-I-249-CHI-ADH-30PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 25-12-2014 13:15:26
Acq. Method    : 30_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\30-1-254-200.M
Last changed   : 11-07-2014 22:58:37 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

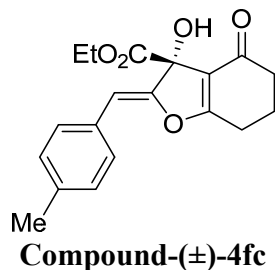
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.948	MM	0.2894	3.33333e4	1919.41785	92.5223
2	19.862	MM	0.5441	2694.01880	82.51991	7.4777

Totals : 3.60273e4 2001.93775

*** End of Report ***

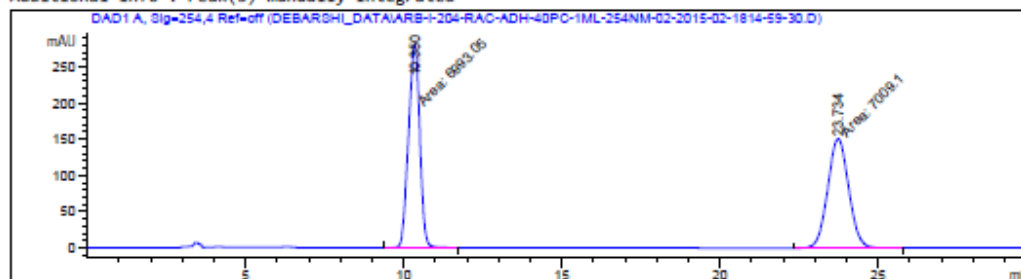
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\ARB-I-204-RAC-ADH-40PC-1ML-254NM-02-2015-02-1814-59-30.D
Sample Name: ARB-I-204-RAC-ADH-40PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 18-02-2015 15:01:15
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M
Last changed    : 11-07-2014 22:47:28 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

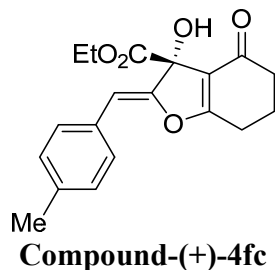
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.350	MM	0.4135	6993.04932	281.84897	49.9427
2	23.734	MM	0.7724	7009.10205	151.24463	50.0573

Totals : 1.40022e4 433.09360

*** End of Report ***

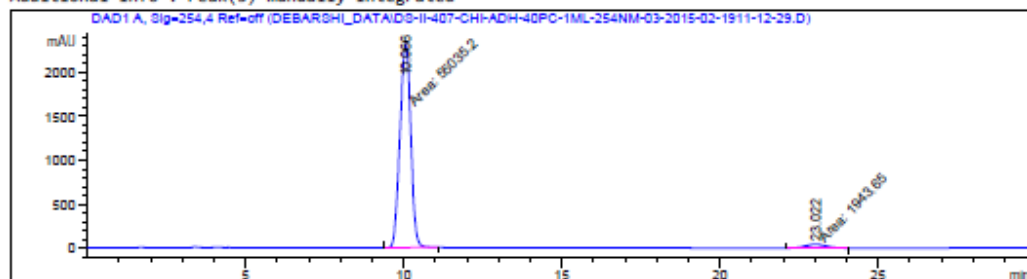
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\DS-II-407-CHI-ADH-40PC-1ML-254NM-03-2015-02-1911-12-29.D
Sample Name: DS-II-407-CHI-ADH-40PC-1ML-254NM-03

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 19-02-2015 11:14:13
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\10-1-254-200.M
Last changed    : 11-07-2014 22:47:28 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

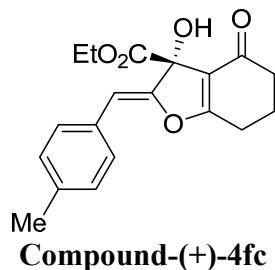
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.066	MM	0.3952	5.50352e4	2321.09277	96.5888
2	23.022	MM	0.7498	1943.64685	43.20223	3.4112

Totals : 5.69788e4 2364.29500

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...HI_DATA\DS-II-407-ML-CHI-ADH-40PC-1ML-254NM-01-2015-02-2311-17-06.D
Sample Name: DS-II-407-ML-CHI-ADH-40PC-1ML-254NM-01

=====

Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 23-02-2015 11:18:51

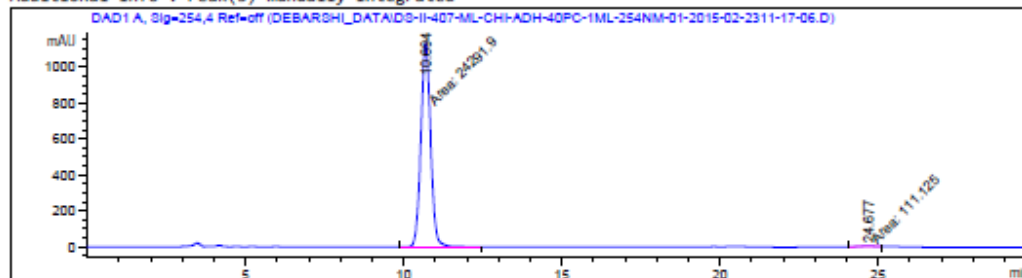
Acq. Method : 40_1_254_100.M

Analysis Method : C:\NAGARAJU\METHODS\30-1-254-100 MIN.M

Last changed : 23-02-2015 10:32:20 by SYSTEM

(modified after loading)

Additional Info : Peak(s) manually integrated



=====

Area Percent Report

=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

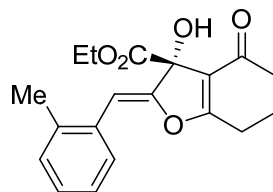
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.694	MM	0.3573	2.42919e4	1133.12048	99.5446
2	24.677	MM	0.5777	111.12463	3.20591	0.4554

Totals : 2.44030e4 1136.32639

=====

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4gc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-365-RAC-ADH-30PC-1ML-254NM-02-2015-02-1809-36-22.D
 Sample Name: DS-II-365-RAC-ADH-30PC-1ML-254NM-02

=====
 Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 18-02-2015 09:38:07

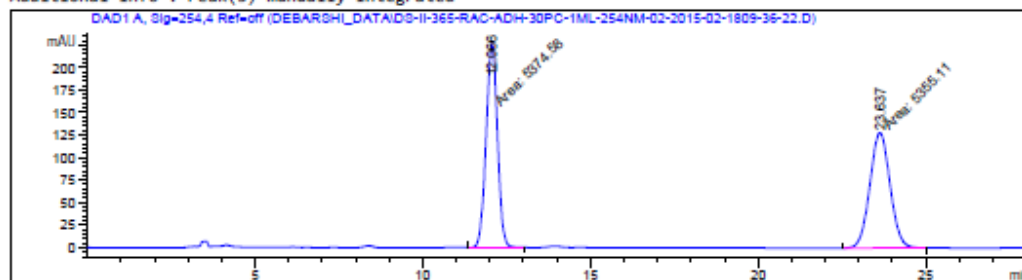
Acq. Method : 30_1_254_100.M

Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M

Last changed : 11-07-2014 22:47:28 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====
 Area Percent Report
 =====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

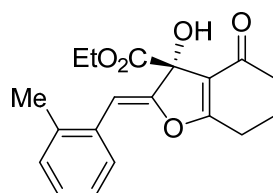
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.066	MM	0.3959	5374.58105	226.26297	50.0907
2	23.637	MM	0.6980	5355.10986	127.87329	49.9093

Totals : 1.07297e4 354.13626

=====
 *** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.

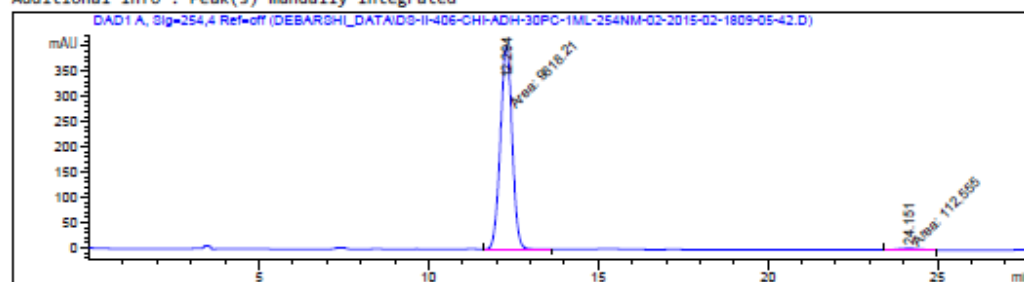


Compound-(+)-4gc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-406-CHI-ADH-30PC-1ML-254NM-02-2015-02-1809-05-42.D
Sample Name: DS-II-406-CHI-ADH-30PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 18-02-2015 09:07:26
Acq. Method     : 30_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\10-1-254-200.M
Last changed    : 11-07-2014 22:47:28 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

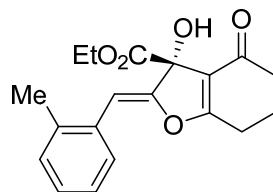
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.294	MM	0.4052	9818.20703	403.85474	98.8666
2	24.151	MM	0.6897	112.55532	2.71983	1.1334

Totals : 9930.76235 406.57456

*** End of Report ***

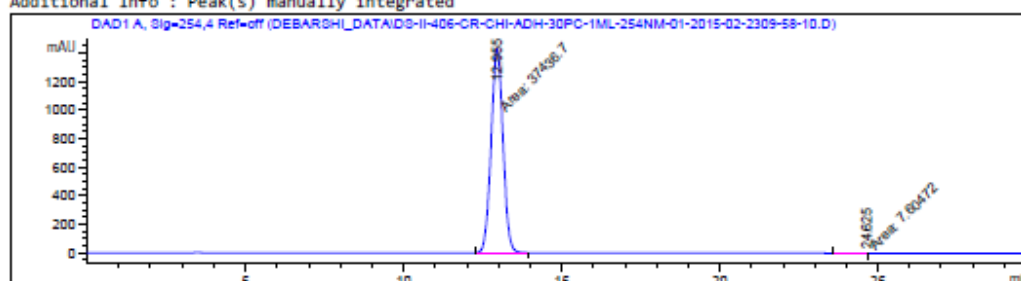
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4gc

Data File C:\CHEM32\...HI_DATA\DS-II-406-CR-CHI-ADH-30PC-1ML-254NM-01-2015-02-2309-58-10.D
Sample Name: DS-II-406-CR-CHI-ADH-30PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 23-02-2015 09:59:54
Acq. Method     : 30_1_254_100.M
Analysis Method : C:\NAGARAJU\METHODS\30-1-254-100 MIN.M
Last changed    : 23-02-2015 10:32:20 by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

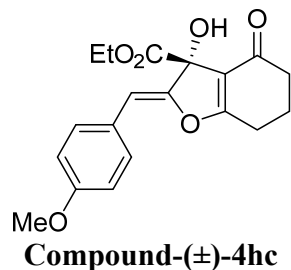
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.955	MM	0.4372	3.74367e4	1427.11145	99.9797
2	24.625	MM	1.0179	7.60472	1.24520e-1	0.0203

```
Totals :                      3.74443e4  1427.23597
```

```
=====
*** End of Report ***
```

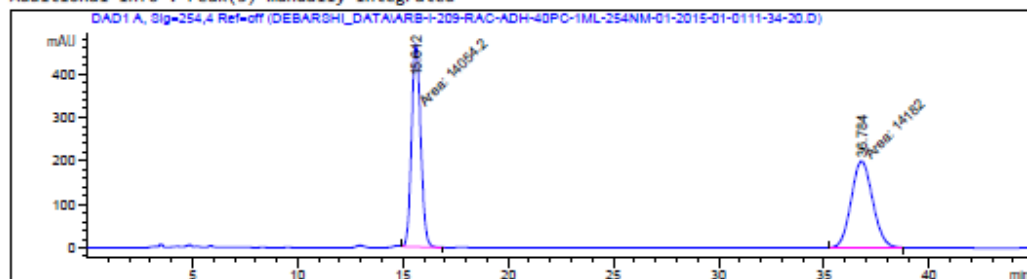
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\ARB-I-209-RAC-ADH-40PC-1ML-254NM-01-2015-01-0111-34-20.D
Sample Name: ARB-I-209-RAC-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 01-01-2015 11:36:03
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\30-1-254-200.M
Last changed   : 11-07-2014 22:58:37 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

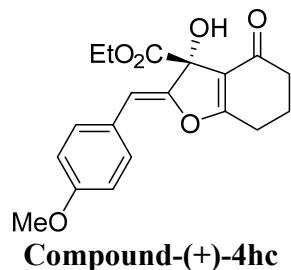
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.612	MM	0.5016	1.40542e4	467.01413	49.7736
2	36.784	MM	1.1848	1.41820e4	199.49828	50.2264

Totals : 2.82362e4 666.51241

*** End of Report ***

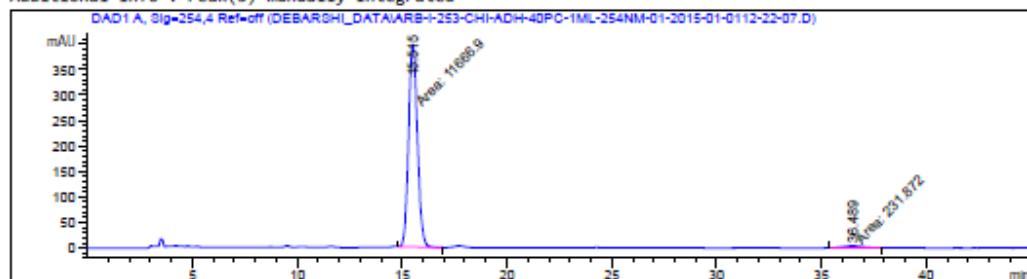
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\ARB-I-253-CHI-ADH-40PC-1ML-254NM-01-2015-01-0112-22-07.D
 Sample Name: ARB-I-253-CHI-ADH-40PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 01-01-2015 12:23:50
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\30-1-254-200.M
Last changed    : 11-07-2014 22:58:37 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



=====
 Area Percent Report
 =====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

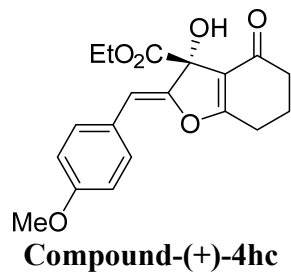
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.515	MM	0.4885	1.16669e4	398.03528	98.0513
2	36.489	MM	1.1065	231.87242	3.49269	1.9487

```
Totals :                      1.18988e4  401.52797
```

=====
 *** End of Report ***

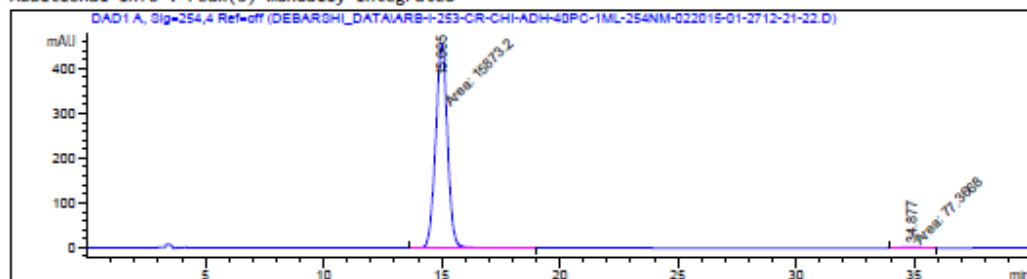
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...SHI_DATA\ARB-I-253-CR-CHI-ADH-40PC-1ML-254NM-022015-01-2712-21-22.D
 Sample Name: ARB-I-253-CR-CHI-ADH-40PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 27-01-2015 12:23:07
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\5-1-254-200.M
Last changed    : 11-07-2014 22:48:53 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

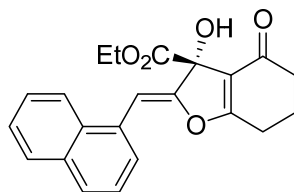
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.005	MM	0.5809	1.58732e4	455.40149	99.5150
2	34.877	MM	0.9413	77.36683	1.36979	0.4850

Totals : 1.59506e4 456.77128

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4ic

Data File C:\CHEM32\...\BARSHI_DATA\ARB-I-274-RAC-IC-30PC-1ML-254NM-02-2015-02-1212-54-20.D
 Sample Name: ARB-I-274-RAC-IC-30PC-1ML-254NM-02

=====

Acq. Operator : SYSTEM Location : Vial 1

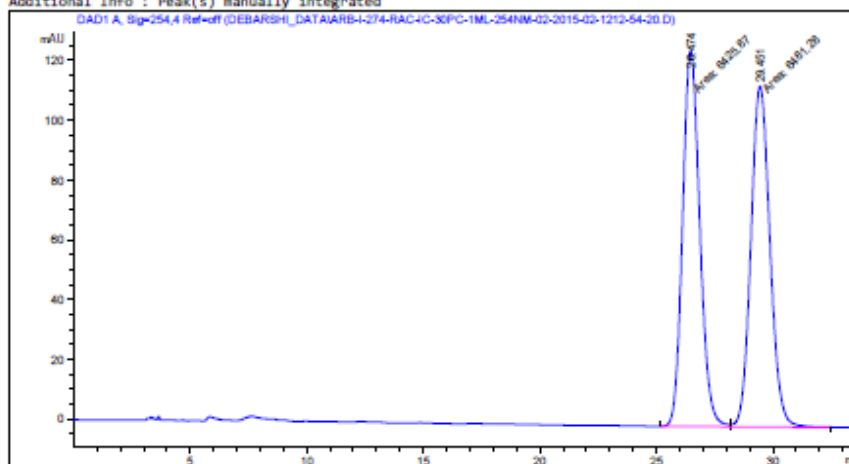
Injection Date : 12-02-2015 12:56:03

Acq. Method : 30_1_254_100.M

Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\20_1_254_100.M

Last changed : 21-07-2014 13:58:26 by SYSTEM

Additional Info : Peak(s) manually integrated



=====
 Area Percent Report
 =====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Use Multiplier & Dilution Factor with ISTDs

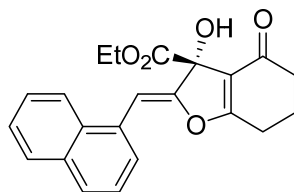
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.474	MF	0.8524	6425.87109	125.63918	49.7854
2	29.451	FM	0.9479	6481.27832	113.95558	50.2146

Totals : 1.29071e4 239.59467

=====
 *** End of Report ***

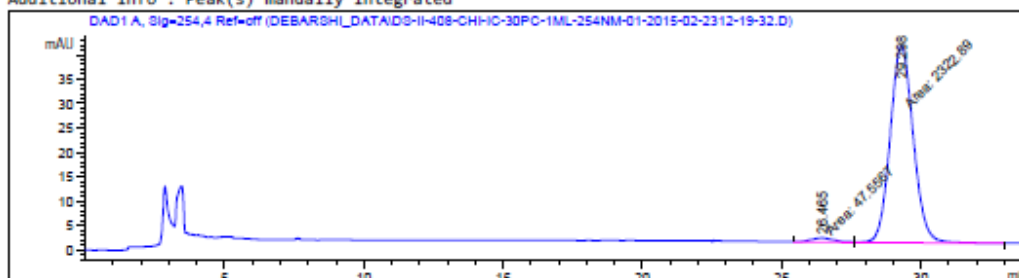
CHIRALPAK IC (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4ic

Data File C:\CHEM32\...\BARSHI_DATA\DS-II-408-CHI-IC-30PC-1ML-254NM-01-2015-02-2312-19-32.D
Sample Name: DS-II-408-CHI-IC-30PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 23-02-2015 12:21:15
Acq. Method     : 30_1_254_100.M
Analysis Method : C:\NAGARAJU\METHODS\30-1-254-100 MIN.M
Last changed    : 23-02-2015 10:32:20 by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



```
=====
Area Percent Report
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

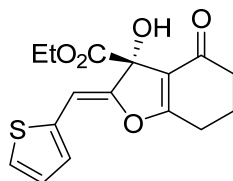
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.465	MF	0.9308	47.55674	8.51559e-1	2.0062
2	29.298	FM	0.9612	2322.89380	40.27945	97.9938

Totals : 2370.45054 41.13101

```
=====
*** End of Report ***
```

CHIRALPAK IC (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (70:30) @ 1.0 mL/min, 254 nm WL.

Compound-(+)-4ic

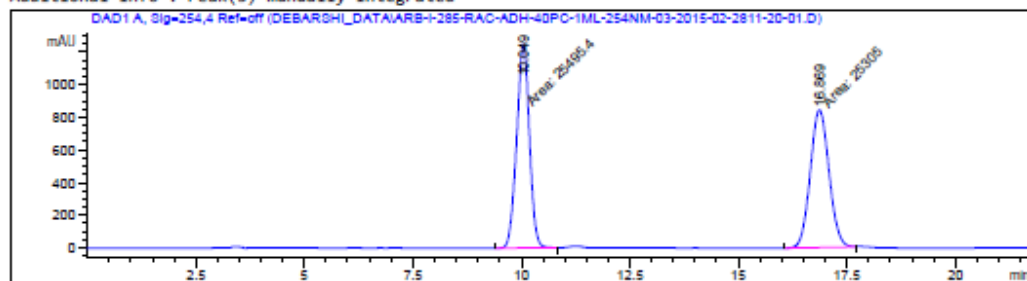


Compound-(±)-4jc

Data File C:\CHEM32\...ARSHI_DATA\ARB-I-285-RAC-ADH-40PC-1ML-254NM-03-2015-02-2811-20-01.D
 Sample Name: ARB-I-285-RAC-ADH-40PC-1ML-254NM-03

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 28-02-2015 11:21:45
Acq. Method    : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\15-1-254-200.M
Last changed   : 11-07-2014 23:02:57 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

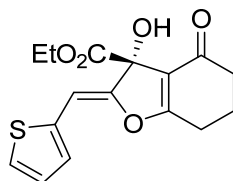
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.049	MM	0.3404	2.54954e4	1248.30737	50.1874
2	16.869	MM	0.5005	2.53050e4	842.64545	49.8126

```
Totals :                      5.08004e4  2090.95282
```

```
=====
*** End of Report ***
```

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.

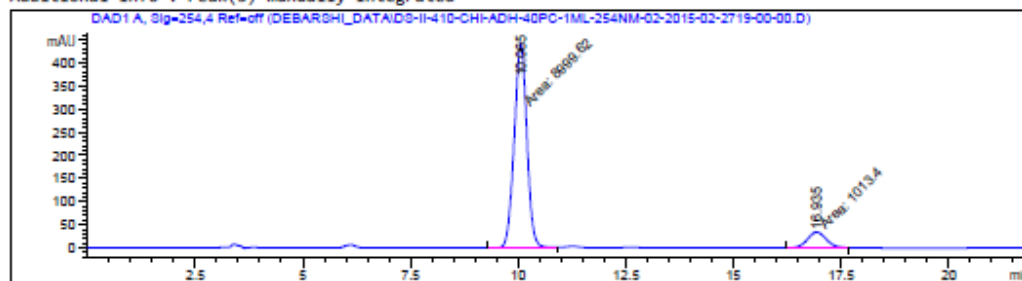


Compound-(+)-4jc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-410-CHI-ADH-40PC-1ML-254NM-02-2015-02-2719-00-00.D
Sample Name: DS-II-410-CHI-ADH-40PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 27-02-2015 19:01:44
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\15-1-254-200.M
Last changed    : 11-07-2014 23:02:57 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

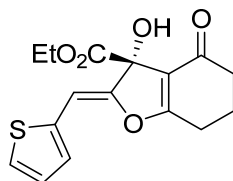
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.065	MM	0.3408	8999.62305	440.14484	89.8792
2	16.935	MM	0.5015	1013.40009	33.67909	10.1208

Totals : 1.00130e4 473.82393

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.

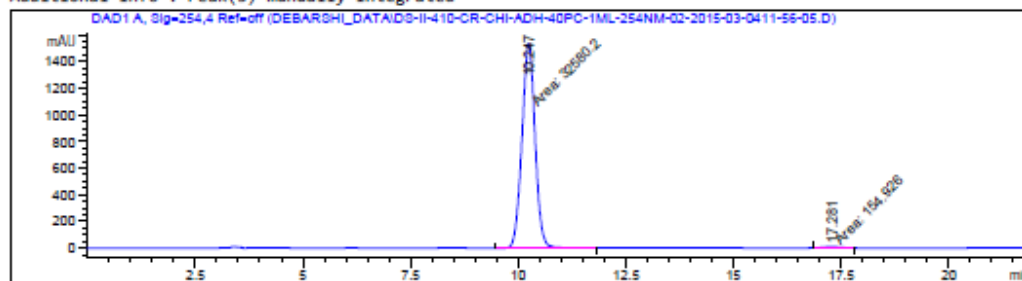


Compound-(+)-4jc

Data File C:\CHEM32\...HI_DATA\DS-II-410-CR-CHI-ADH-40PC-1ML-254NM-02-2015-03-0411-56-05.D
Sample Name: DS-II-410-CR-CHI-ADH-40PC-1ML-254NM-02

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 04-03-2015 11:57:49
Acq. Method     : 40_1_254_100.M
Analysis Method : C:\RAJESHEKHAR METHOD\5-1-254-200.M
Last changed    : 11-07-2014 22:48:53 by SYSTEM
Method Info     : ok
```

Additional Info : Peak(s) manually integrated



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

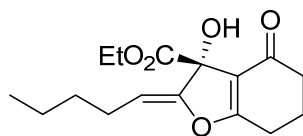
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.247	MM	0.3547	3.25802e4	1530.73499	99.5267
2	17.281	MM	0.4101	154.92575	6.29675	0.4733

```
Totals :                      3.27351e4  1537.03173
```

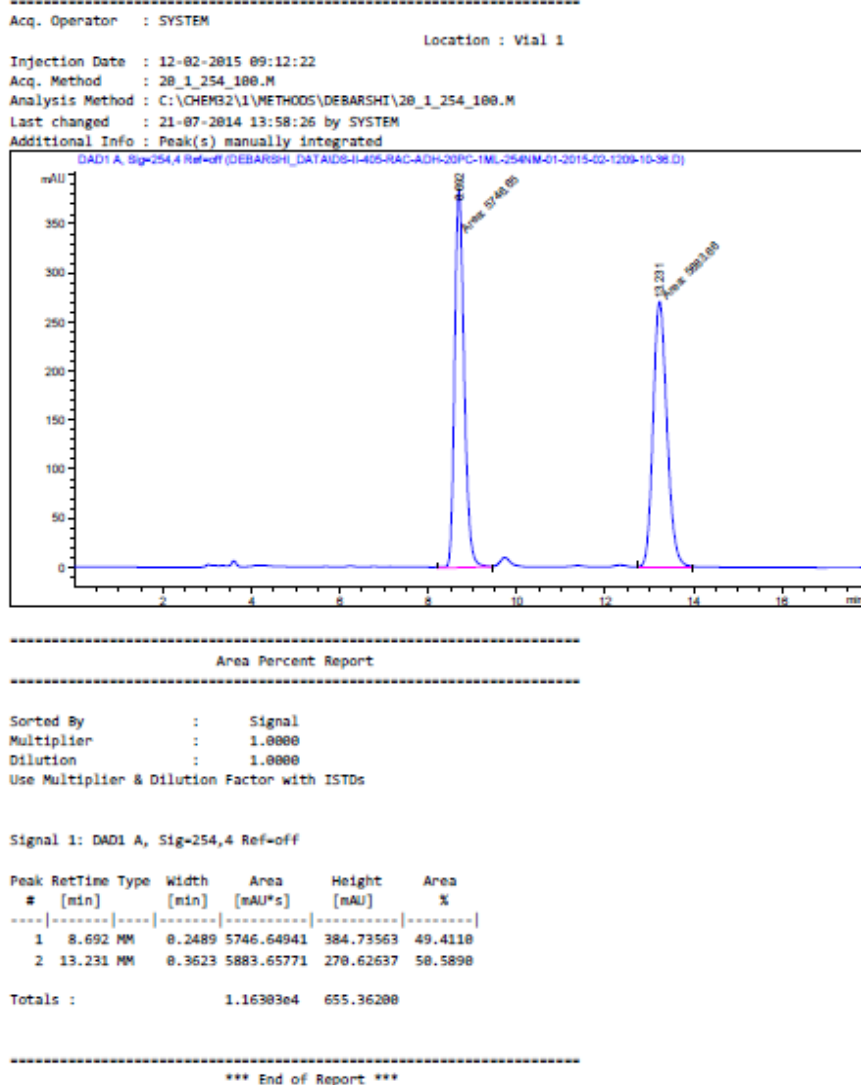
```
=====
*** End of Report ***
```

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.

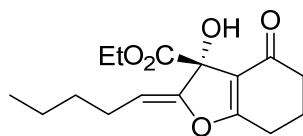


Compound-(±)-4kc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-485-RAC-ADH-28PC-1ML-254NM-01-2015-02-1209-10-36.D
Sample Name: DS-II-485-RAC-ADH-28PC-1ML-254NM-01

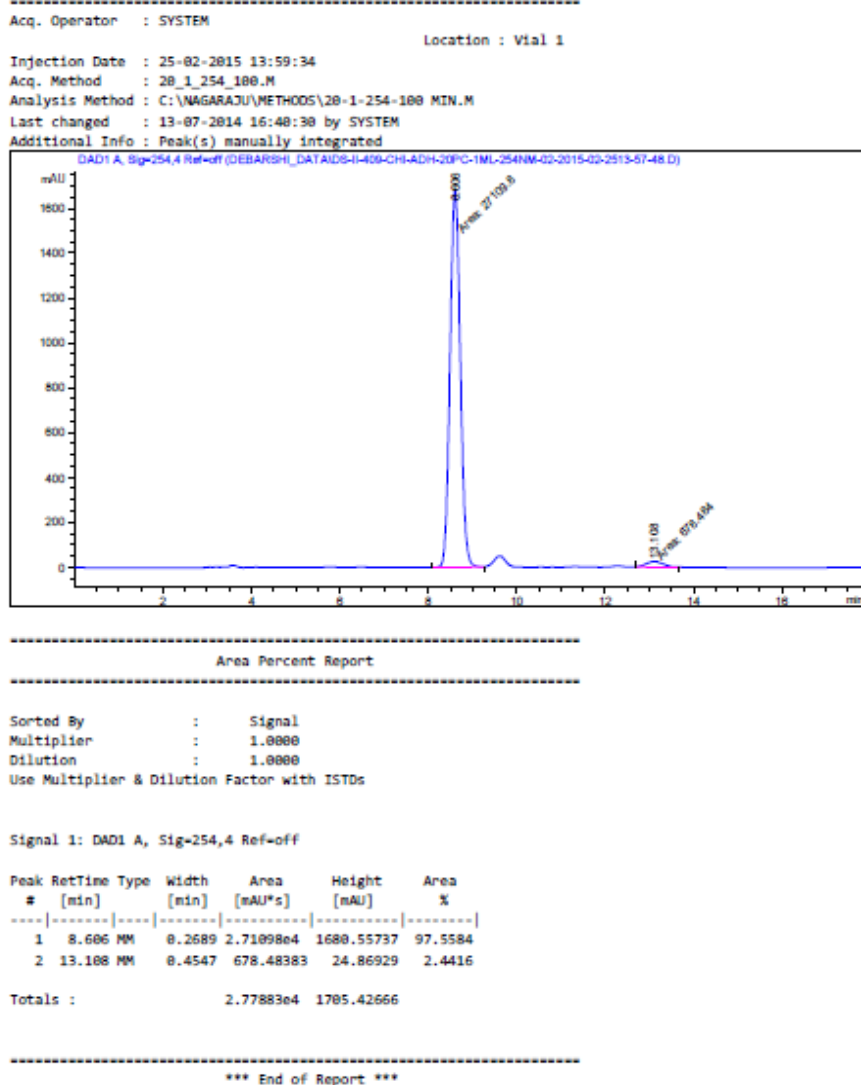


CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.

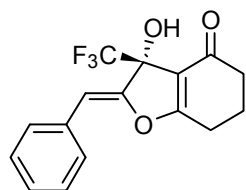


Compound-(+)-4kc

Data File C:\CHEM32\...ARSHI_DATA\DS-II-409-CHI-ADH-20PC-1ML-254NM-02-2015-02-2513-57-48.D
 Sample Name: DS-II-409-CHI-ADH-20PC-1ML-254NM-02



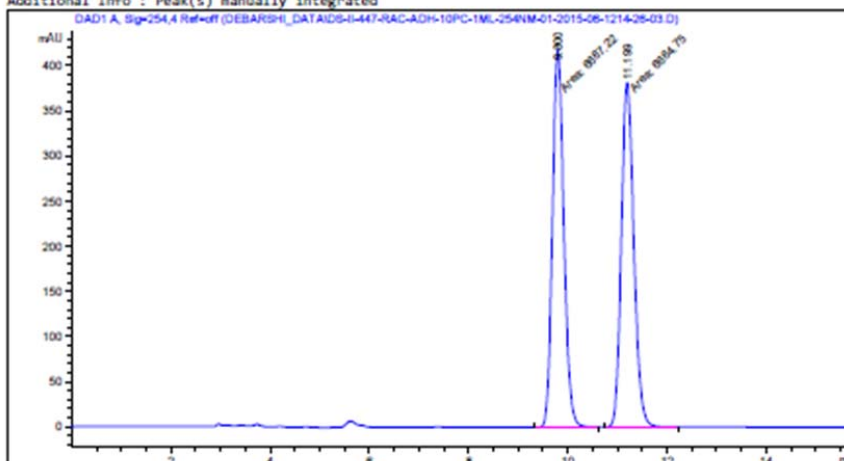
CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4lc

Data File C:\CHEM32\...\ARSHI_DATA\DS-II-447-RAC-ADH-10PC-1ML-254NM-01-2015-06-1214-26-03.D
Sample Name: DS-II-447-RAC-ADH-10PC-1ML-254NM-01

Acq. Operator : SYSTEM
Location : Vial 1
Injection Date : 12-06-2015 14:27:47
Acq. Method : 10_1_254_100.M
Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\20_1_254_100.M
Last changed : 21-07-2014 13:58:26 by SYSTEM
Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

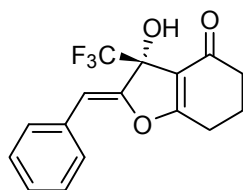
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.800	MM	0.2665	6687.22168	418.14304	50.0092
2	11.199	MM	0.2919	6684.75293	381.64825	49.9908

Totals : 1.33720e4 799.79129

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (90:10) @ 1.0 mL/min, 254 nm WL.



Compound-(+)-4lc

Data File C:\CHEM32\...RSHI_DATA\DS-II-446R-CHI-ADH-10PC-1ML-254NM-01-2015-06-1609-51-13.D
Sample Name: DS-II-446R-CHI-ADH-10PC-1ML-254NM-01

=====

Acq. Operator : SYSTEM

Location : Vial 1

Injection Date : 16-06-2015 09:52:57

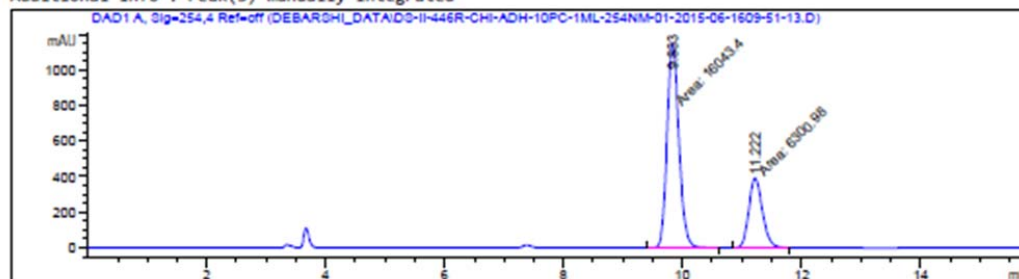
Acq. Method : 10_1_254_100.M

Analysis Method : C:\RAJESHEKHAR METHOD\10-1-254-200.M

Last changed : 11-07-2014 22:47:28 by SYSTEM

Method Info : ok

Additional Info : Peak(s) manually integrated



=====

Area Percent Report

=====

Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

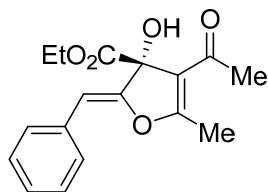
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.833	MM	0.2324	1.60434e4	1150.46301	71.8006
2	11.222	MM	0.2667	6300.98193	393.77457	28.1994

Totals : 2.23444e4 1544.23758

=====

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (90:10) @ 1.0 mL/min, 254 nm WL.

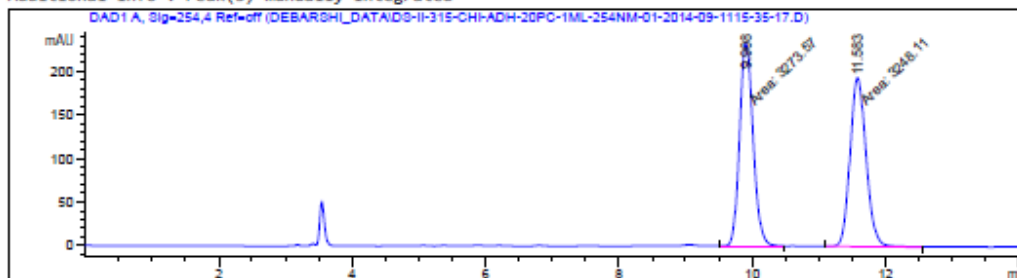


Compound-(±)-4ad

Data File C:\CHEM32\...ARSHI_DATA\DS-II-315-CHI-ADH-20PC-1ML-254NM-01-2014-09-1115-35-17.D
Sample Name: DS-II-315-CHI-ADH-20PC-1ML-254NM-01

```
=====
Acq. Operator   : SYSTEM                               Location : Vial 1
Injection Date  : 11-09-2014 15:37:01
Acq. Method    : 20_1_254_100.M
Analysis Method : C:\RAJSHEKHAR METHOD\5-1-254-200.M
Last changed   : 11-07-2014 22:48:53 by SYSTEM
Method Info    : ok
```

Additional Info : Peak(s) manually integrated



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

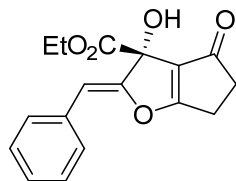
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.908	MM	0.2341	3273.56812	233.10045	50.1952
2	11.583	MM	0.2791	3248.11108	193.98209	49.8048

Totals : 6521.67920 427.08253

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (80:20) @ 1.0 mL/min, 254 nm WL.



Compound-(±)-4ae

Data File C:\CHEM32\...ARSHI_DATA\DS-II-319-RAC-ADH-48PC-1ML-254NM-01-2014-09-1615-18-08.D
Sample Name: DS-II-319-RAC-ADH-48PC-1ML-254NM-01

Acq. Operator : SYSTEM

Location : Vial 1

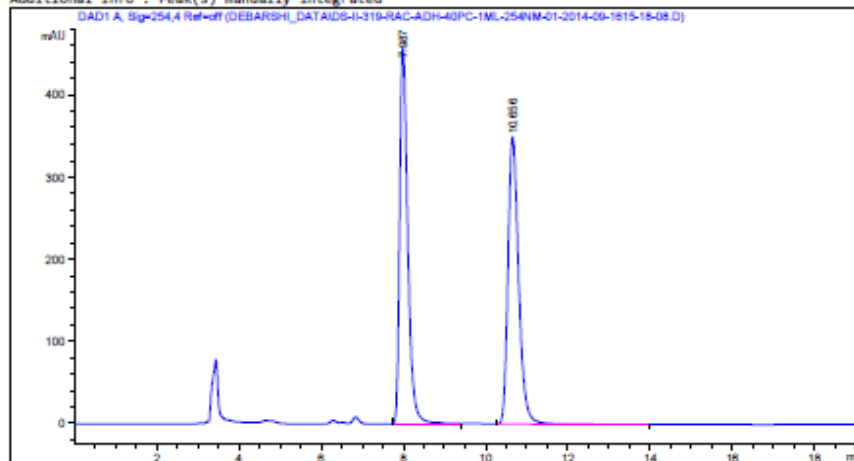
Injection Date : 16-09-2014 15:19:53

Acq. Method : 40_1_254_100.M

Analysis Method : C:\CHEM32\1\METHODS\DEBARSHI\30_1_254_100.M

Last changed : 12-07-2014 15:06:32 by SYSTEM

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

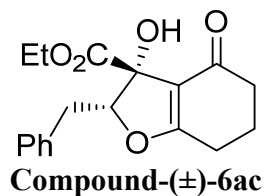
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.987	BB	0.2074	6256.22021	457.79544	49.7919
2	10.656	BB	0.2767	6308.51758	349.27304	50.2081

Totals : 1.25647e4 807.06848

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (60:40) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...\ARSHI_DATA\ARB-I-301-RAC-ADH-10PC-1ML-254NM-03-2015-03-3019-08-37.D
Sample Name: ARB-I-301-RAC-ADH-10PC-1ML-254NM-03

=====
Acq. Operator : SYSTEM

Location : Vial 1

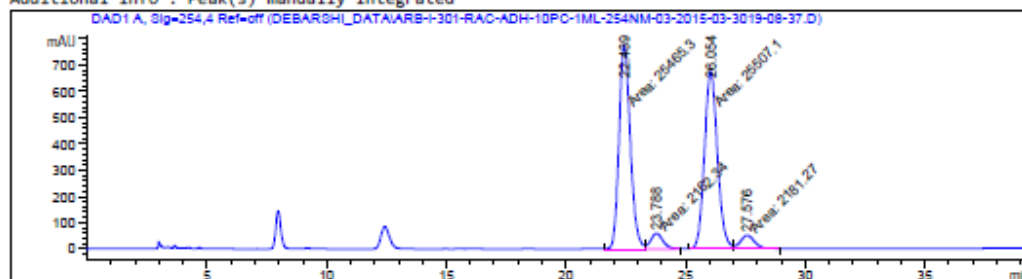
Injection Date : 30-03-2015 19:10:22

Acq. Method : 10_1_254_100.M

Analysis Method : C:\NAGARAJU\METHODS\10_1_254_INFINITY.M

Last changed : 21-12-2014 19:05:43 by SYSTEM

Additional Info : Peak(s) manually integrated



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Area Percent Report
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Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

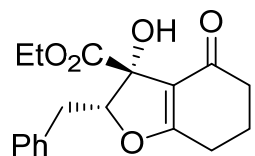
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.439	MF	0.5470	2.54653e4	775.94495	46.0361
2	23.788	FM	0.6008	2162.33765	59.98307	3.9091
3	26.054	MF	0.6304	2.55071e4	674.40613	46.1115
4	27.576	FM	0.7026	2181.27344	51.74578	3.9433

Totals : 5.53160e4 1562.07992

=====
*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (90:10) @ 1.0 mL/min, 254 nm WL.



Compound(-)-6ac

Data File C:\CHEM32\...ARSHI_DATA\ARB-I-310-CHI-ADH-10PC-1ML-254NM-03-2015-03-3018-25-17.D
Sample Name: ARB-I-310-CHI-ADH-10PC-1ML-254NM-03

=====

Acq. Operator : SYSTEM

Location : Vial 1

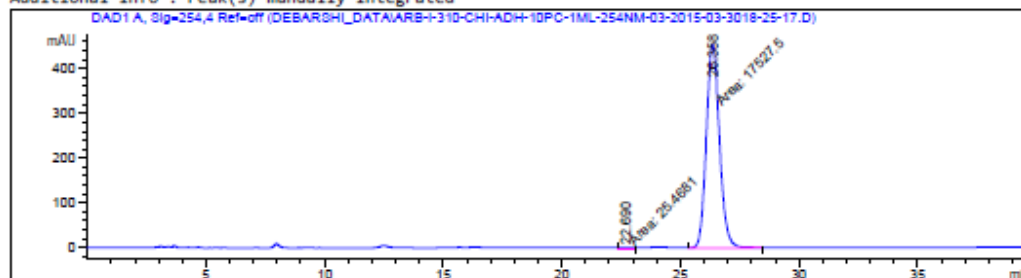
Injection Date : 30-03-2015 18:27:01

Acq. Method : 10_1_254_100.M

Analysis Method : C:\NAGARAJU\METHODS\10_1_254_INFINITY.M

Last changed : 21-12-2014 19:05:43 by SYSTEM

Additional Info : Peak(s) manually integrated



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Area Percent Report

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Sorted By : Signal

Multiplier : 1.0000

Dilution : 1.0000

Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

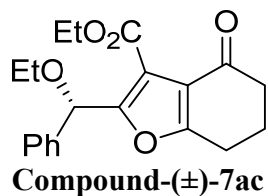
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.690	MM	0.5832	25.46806	7.27865e-1	0.1451
2	26.358	MM	0.6422	1.75275e4	454.91586	99.8549

Totals : 1.75530e4 455.64373

=====

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
Hexane/*i*-PrOH (90:10) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...\ARSHI_DATA\DS-II-417-RAC-ADH-03PC-1ML-254NM-02-2015-04-0610-39-49.D
 Sample Name: DS-II-417-RAC-ADH-03PC-1ML-254NM-02

Acq. Operator : SYSTEM

Location : Vial 1

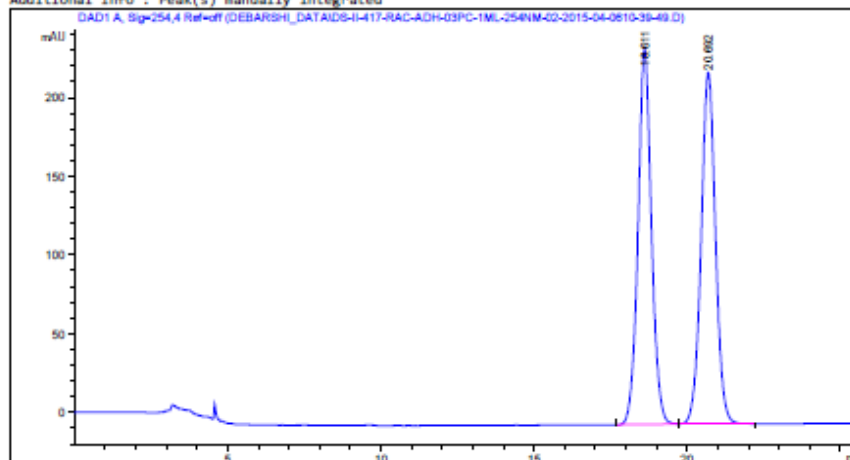
Injection Date : 06-04-2015 10:41:32

Acq. Method : 3_1_254_100.M

Analysis Method : C:\NAGARAJU\METHODS\20-1-254-100 MIN.M

Last changed : 13-07-2014 16:48:30 by SYSTEM

Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

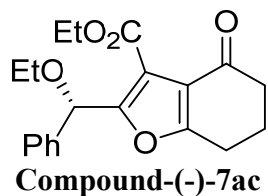
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.611	BB	0.4551	7260.64063	238.51656	49.9884
2	20.692	BB	0.4980	7264.01221	223.00827	50.0116

Totals : 1.45247e4 461.52483

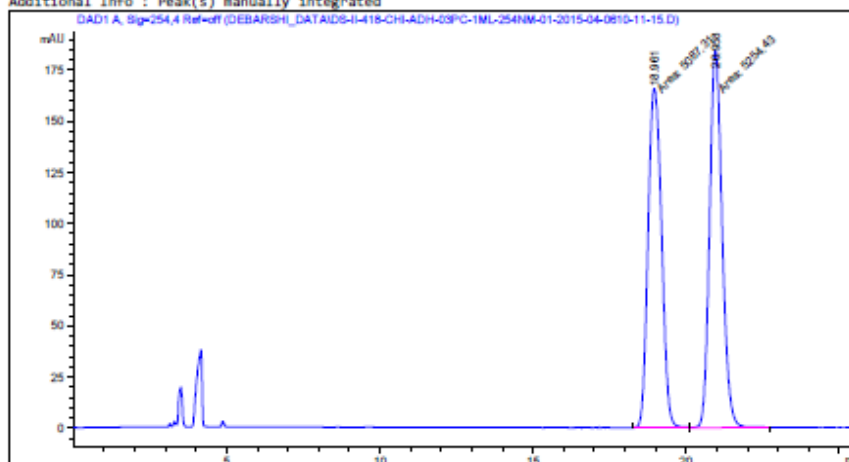
*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/*i*-PrOH (97:03) @ 1.0 mL/min, 254 nm WL.



Data File C:\CHEM32\...ARSHI_DATA\DS-II-418-CHI-ADH-03PC-1ML-254NM-01-2015-04-0610-11-15.D
 Sample Name: DS-II-418-CHI-ADH-03PC-1ML-254NM-01

Acq. Operator : SYSTEM
 Location : Vial 1
 Injection Date : 06-04-2015 10:13:00
 Acq. Method : 3_1_254_100.M
 Analysis Method : C:\NAGARAJU\METHODS\20-1-254-100 MIN.M
 Last changed : 13-07-2014 16:48:30 by SYSTEM
 Additional Info : Peak(s) manually integrated



Area Percent Report

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.961	MF	0.5116	5087.31006	165.73799	49.1920
2	20.958	FM	0.4752	5254.43359	184.28235	50.8080

Totals : 1.03417e4 350.02034

*** End of Report ***

CHIRALPAK AD-H (250.0 mm x 4.6 mm) column,
 Hexane/i-PrOH (97:03) @ 1.0 mL/min, 254 nm WL.