

Supplemental material for:

**Catalytic Decarboxylative Allylation of Enol Carbonates:  
Synthesis of Enantioenriched 3-Allyl-3'-Aryl 2-Oxindoles and  
Core Structure of Azonazine**

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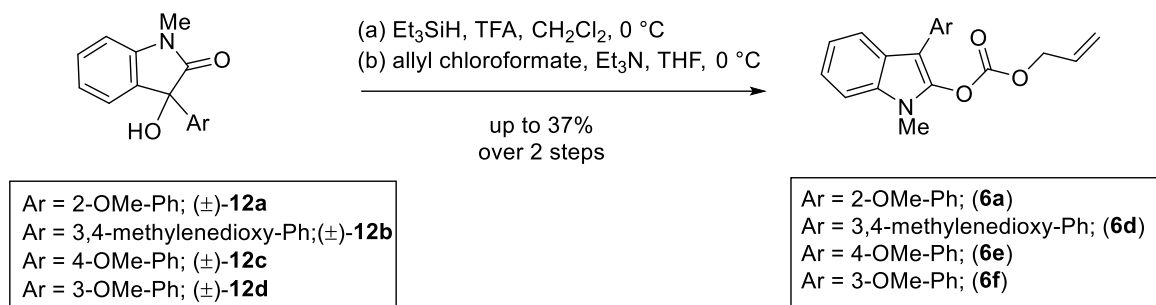
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## Materials and Methods

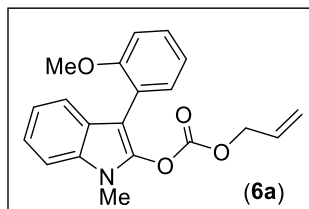
Unless otherwise stated, reactions were performed in oven-dried glassware fitted with rubber septa under an inert atmosphere and were stirred with Teflon-coated magnetic stirring bars. Liquid reagents and solvents were transferred *via* syringe using standard Schlenk techniques. Tetrahydrofuran (THF) and diethyl ether (Et<sub>2</sub>O) were distilled over sodium/benzophenone ketyl. Dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>), toluene, and benzene were distilled over calcium hydride. All other solvents and reagents were used as received unless otherwise noted. Reaction temperatures above 23 °C refer to oil bath temperature. Thin layer chromatography was performed using silica gel 60 F-254 precoated plates (0.25 mm) and visualized by UV irradiation, anisaldehyde stain and other stains. Silicagel of particle size 100-200 mesh was used for flash chromatography. Melting points were recorded on a digital melting point apparatus and are uncorrected. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded 400, 500 MHz spectrometers with <sup>13</sup>C operating frequencies of 100, 125 MHz respectively. Chemical shifts (δ) are reported in ppm relative to the residual solvent (CDCl<sub>3</sub>) signal (δ = 7.26 for <sup>1</sup>H NMR and δ = 77.0 for <sup>13</sup>C NMR). Data for <sup>1</sup>H NMR spectra are reported as follows: chemical shift (multiplicity, coupling constants, and number of hydrogen). Abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad). IR spectra were recorded on a FT-IR system (Spectrum BX) and are reported in frequency of absorption (cm<sup>-1</sup>). Only selected IR absorbencies are reported. High-Resolution Mass Spectrometry (HRMS) and Low-Resolution Mass Spectrometry (LRMS) data were recorded on MicrOTOF-Q-II mass spectrometer using methanol as solvent. Optical rotations were measured on an automatic polarimeter. Enantiomeric excess was determined by chiral HPLC analysis performed on HPLC system with Daicel Chiralpak AD-H, Daicel Chiralpak OD-3, Daicel Chiralpak OZ-3 and Daicel Chiralpak IB column.

General procedure for the synthesis of compounds (**6a** and **6d-f**):

In an oven-dried round-bottom flask was charged with compound ( $\pm$ )-**12** (7.0 mmol, 1.0 equiv.) in dichloromethane (30 mL) under an argon atmosphere at room temperature. To this solution, trifluoroacetic acid (TFA) (42.0 mmol; 6.0 equiv.) was added dropwise at  $0\text{ }^\circ\text{C}$ . After 5 minutes of stirring triethyl silane (35.0 mmol, 5.0 equiv.) was added dropwise over 2 minutes and stirring was continued for 12 h. After completion of the reaction (judged by TLC analysis under UV and  $\text{I}_2$  stain) 5% (w/v) aqueous solution of sodium citrate (5 mL) was added dropwise to the reaction mixture followed by addition of 20 mL of dichloromethane. Then whole mixture was taken in a separatory funnel and the organic layer was separated. The organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was directly charged for the next step.

The crude 3-aryl 2-oxindole (7.0 mmol, 1.0 equiv.) was taken in THF (30 mL) under nitrogen atmosphere at  $0\text{ }^\circ\text{C}$ . Then  $\text{Et}_3\text{N}$  (2.9 mL, 21.0 mmol, 3.0 equiv.) was added to the solution. After 5 minutes of stirring at  $0\text{ }^\circ\text{C}$ , allyl chloroformate (0.9 mL, 8.4 mmol, 1.2 equiv.) was added drop-wise over a period of 2 minutes and stirring was continued. Upon completion of the reaction (judged by TLC under UV light and  $\text{I}_2$  stain), the reaction mixture was diluted with EtOAc (30 mL) and quenched with  $\text{H}_2\text{O}$ . Then the organic layer was separated, dried with anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was purified by flash chromatography with *n*-Hexane-EtOAc (7:3) to afford product (**6a** and **6d-f**) as yellow gel.

[For synthesis of 3-hydroxy 2-oxindoles ( $\pm$ )-**12a-d** see the reference 18.]



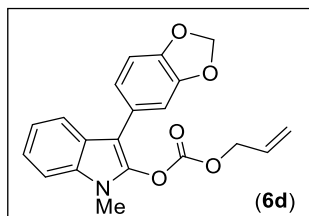
**Allyl (3-(2-methoxyphenyl)-1-methyl-1H-indol-2-yl) carbonate (6a):** The compound **6a** was obtained as orange color gel (7.0 mmol scale of reaction; 0.80 g; 34% over 2 steps).  $R_f = 0.40$  (30% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (d,  $J = 7.9$  Hz, 1H), 7.53 (dd,  $J = 7.5, 1.9$  Hz, 1H), 7.37 - 7.27 (m, 3H), 7.19 (td,  $J = 7.4, 6.8, 1.4$  Hz, 1H), 7.09 (td,  $J = 7.4, 1.3$  Hz, 1H), 7.04 (d,  $J = 8.3$  Hz, 1H), 5.95 (ddt,  $J = 16.5, 10.6, 5.8$  Hz, 1H), 5.41 (dt,  $J = 17.3, 1.5$  Hz, 1H), 5.34 (dd,  $J = 10.5, 1.5$  Hz, 1H), 4.76 (dd,  $J = 5.7, 1.4$  Hz, 2H), 3.80 (s, 3H), 3.70 (s, 3H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.0, 152.3, 139.2, 132.8, 131.4, 130.8, 128.1, 125.4, 121.8, 121.6, 120.8, 120.2, 120.2, 119.8, 111.3, 109.2, 99.1, 69.8, 55.5, 28.7.

**IR** (film)  $\nu_{\text{max}}$  3736, 3368, 2916, 2540, 1779, 1490, 1421, 1300, 1214, 1104, 921, 710, 620  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{19}\text{NO}_4 + \text{Na}]^+$  360.1206; Found 360.1191.



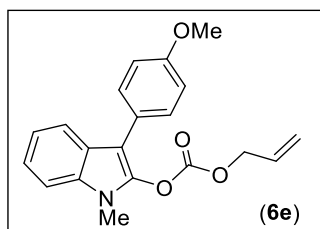
**Allyl (3-(benzo[*d*][1,3]dioxol-5-yl)-1-methyl-1H-indol-2-yl) carbonate (6d):** The compound **6d** was obtained as orange color gel (7.0 mmol scale of reaction, 0.8 g, 33% over 2 steps).  $R_f = 0.41$  (30% EtOAc in hexane).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.79 (d, *J* = 7.9 Hz, 1H), 7.34 - 7.27 (m, 2H), 7.24 - 7.18 (m, 1H), 7.11 - 7.06 (m, 2H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.00 (s, 2H), 5.94 (ddd, *J* = 16.5, 10.9, 5.4 Hz, 1H), 5.41 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.34 (d, *J* = 10.4 Hz, 1H), 4.74 (d, *J* = 5.7 Hz, 2H), 3.66 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 152.2, 147.9, 146.1, 138.6, 132.5, 130.6, 126.7, 124.8, 122.1, 121.8, 120.5, 120.0, 119.6, 109.3, 109.0, 108.8, 102.9, 101.0, 70.0, 28.4.

**IR** (film)  $\nu_{\max}$  3666, 3328, 2906, 2510, 1769, 1500, 1431, 1320, 1213, 1154, 951, 718, 650 cm<sup>-1</sup>.

**HRMS** (ESI-TOF) *m/z*: [M + Na]<sup>+</sup> Calcd for [C<sub>20</sub>H<sub>17</sub>NO<sub>5</sub> + Na]<sup>+</sup> 374.0999; Found 374.1005.



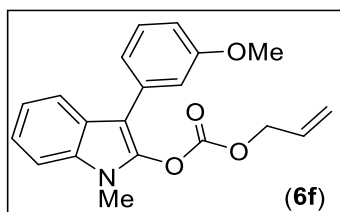
**Allyl (3-(4-methoxyphenyl)-1-methyl-1H-indol-2-yl) carbonate (6e)**: The compound **6e** was obtained as orange color gel (7.0 mmol scale of reaction, 0.70 g, 37% over 2 steps). *R<sub>f</sub>* = 0.43 (30% EtOAc in hexane).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.92 (d, *J* = 8.1 Hz, 1H), 7.68 - 7.66 (m, 2H), 7.39 - 7.38 (m, 2H), 7.30 (ddd, *J* = 8.1, 4.8, 3.4 Hz, 1H), 7.13 - 7.11 (m, 2H), 5.99 (ddt, *J* = 17.3, 10.5, 5.8 Hz, 1H), 5.46 (dd, *J* = 17.2, 1.5 Hz, 1H), 5.39 (dq, *J* = 10.5, 1.3 Hz, 1H), 4.79 (dt, *J* = 5.8, 1.4 Hz, 2H), 3.91 (s, 3H), 3.71 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 158.3, 152.4, 138.7, 132.7, 130.7, 129.6, 125.5, 125.0, 122.2, 120.6, 119.9, 119.7, 114.4, 109.4, 102.9, 70.0, 55.3, 28.4.

**IR** (film)  $\nu_{\max}$  3621, 3338, 2996, 2520, 1774, 1520, 1421, 1350, 1210, 1104, 901, 710, 610  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{20}\text{NO}_4]^+$  338.1387; Found 338.1373.



**Allyl (3-(3-methoxyphenyl)-1-methyl-1H-indol-2-yl) carbonate (6f)**: The compound **6f** was obtained as an orange color gel. (7.0 mmol scale of reaction; 0.70 g; 32% over 2 steps).  $R_f = 0.50$  (20% EtOAc in hexane).

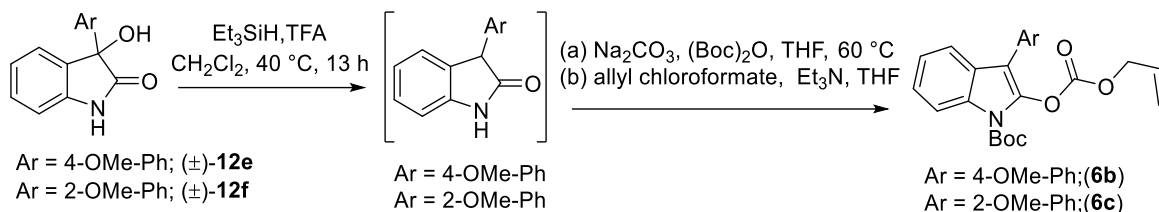
**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.84 (d,  $J = 7.9$  Hz, 1H), 7.38 - 7.24 (m, 3H), 7.22 - 7.17 (m, 3H), 6.85 (dd,  $J = 8.3, 2.5$  Hz, 1H), 5.90 (ddt,  $J = 16.5, 10.4, 5.8$  Hz, 1H), 5.38 (dd,  $J = 17.2, 1.5$  Hz, 1H), 5.31 (dd,  $J = 10.5, 1.3$  Hz, 1H), 4.71 (dt,  $J = 5.8, 1.4$  Hz, 2H), 3.85 (s, 3H), 3.67 (s, 3H).

**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  159.9, 152.1, 138.9, 134.3, 132.6, 130.5, 129.7, 124.6, 122.2, 120.8, 120.6, 119.9, 119.7, 113.6, 112.1, 109.3, 102.9, 70.0, 55.2, 28.4.

**IR** (film)  $\nu_{\max}$  3574, 3040, 2959, 2372, 1768, 1579, 1484, 1377, 1238, 1076, 904, 788, 521  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{20}\text{NO}_4]^+$  338.1387; Found 338.1375.

**General procedure for the synthesis of compounds (6b and 6c):**



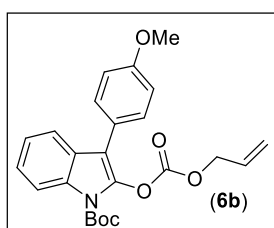
In an oven-dried round-bottom flask was charged with (±)-**12e-f** (7.8 mmol; 1.0 equiv.) in dichloromethane (40 mL) under argon atmosphere at room temperature. To this solution trifluoroacetic acid (TFA) (3.6 mL; 47 mmol; 6.0 equiv) was added dropwise over a period of 2 minutes at 0 °C. Then triethyl silane (6.2 mL; 39.0 mmol; 5.0 equiv.) was added dropwise over a period of 2 minutes and stirring was continued for 13 h at 40 °C. After completion of the reaction (judged by TLC analysis under UV and I<sub>2</sub> stain) 5% (w/v) aqueous solution of sodium citrate (5 mL) was added drop wise to neutralize the mixture. Then the reaction mixture was taken in a separatory funnel and the organic layer was separated. The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced. The crude product was directly charged for the next step.

The crude 3-aryl 2-oxindole (7.8 mmol, 1.0 equiv.) was taken in tetrahydrofuran (40 mL) and Na<sub>2</sub>CO<sub>3</sub> (6.6 g, 62.4 mmol; 8.0 equiv.) was added. Then di-*tert*-butyl pyrocarbonate (Boc-anhydride) (1.8 mL, 8.6 mmol, 1.1 equiv.) solution in THF (3 mL) was slowly added to the reaction mixture at room temperature. The reaction mixture was then placed in a preheated oil bath maintaining 60 °C and the stirring was continued for 24 h. Upon completion of the reaction (judged by TLC under UV light and I<sub>2</sub> stain) reaction mixture was diluted with EtOAc (50 mL) and saturated NH<sub>4</sub>Cl solution (30 mL). The whole reaction mixture was taken in a separatory funnel to separate the organic layer. The aqueous layer was further extracted with EtOAc (30 mL). The combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced. The crude product was directly charged for the next step.

The crude *N*-Boc protected 2-oxindole (7.8 mmol, 1.0 equiv.) was taken in dry THF (30 mL) under nitrogen atmosphere at 0 °C. Then triethyl amine (2.4 mL, 23.4 mmol, 3.0 equiv.) was added to the solution. After 5 minutes of stirring, allyl chloroformate (1.0

mL, 9.4 mmol, 1.2 equiv.) was added drop-wise over a period of 2 minutes at 0 °C and stirring was continued for 13 h. Upon completion of the reaction (judged by TLC under UV light and I<sub>2</sub> stain), the reaction mixture was diluted with EtOAc (50 mL) and quenched with H<sub>2</sub>O. The whole reaction mixture was taken in a separatory funnel to separate the organic layer. Then the organic layer was separated, dried with Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude product was purified by flash chromatography with *n*-Hexane-EtOAc (7:3) to afford **6b-c** as yellow gel.

[For synthesis of 3-hydroxy 2-oxindoles ( $\pm$ )-**12e-f** see the reference 18.]



**tert-Butyl 2-(((allyloxy)carbonyl)oxy)-3-(4-methoxyphenyl)-1H-indole-1-carboxylate (6b):** The compound **6b** was obtained as an orange color gel. (7.8 mmol scale of reaction; 1.0 g of product; 30.7%, over three steps).  $R_f = 0.45$  (30% EtOAc in hexane).

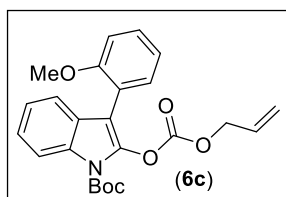
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.18 (d,  $J = 8.3$  Hz, 1H), 7.69 – 7.61 (m, 1H), 7.52 (d,  $J = 8.8$  Hz, 2H), 7.38 (ddd,  $J = 8.4, 7.2, 1.4$  Hz, 1H), 7.33 – 7.29 (m, 1H), 7.04 (d,  $J = 8.7$  Hz, 2H), 5.97 (ddt,  $J = 16.5, 10.5, 5.8$  Hz, 1H), 5.41 (dd,  $J = 17.2, 1.5$  Hz, 1H), 5.33 (dd,  $J = 10.4, 1.4$  Hz, 1H), 4.76 (dt,  $J = 5.8, 1.5$  Hz, 2H), 3.89 (s, 3H), 1.69 (s, 9H).

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>)  $\delta$  159.0, 152.3, 148.9, 136.9, 132.2, 130.7, 130.2, 126.5, 124.6, 123.3, 123.1, 119.5, 115.5, 114.2, 109.8, 84.7, 69.8, 55.3, 28.

**IR** (film)  $\nu_{\max}$  3710, 3338, 2946, 2418, 1764, 1517, 1430, 1317, 1210, 1024, 957, 826, 782, 622 cm<sup>-1</sup>.

**HRMS** (ESI-TOF)  $m/z$ : [M + Na]<sup>+</sup> Calcd for [C<sub>24</sub>H<sub>25</sub>NO<sub>6</sub> + Na]<sup>+</sup> 446.1574; Found 446.1572.





**tert-Butyl 2-(((allyloxy)carbonyl)oxy)-3-(2-methoxyphenyl)-1H-indole-1-carboxylate (6c):** The compound **6c** was obtained as orange color gel (7.8 mmol scale of reaction; 1.0 g of product; 30% over three steps).  $R_f = 0.30$  (10% EtOAc in hexane).

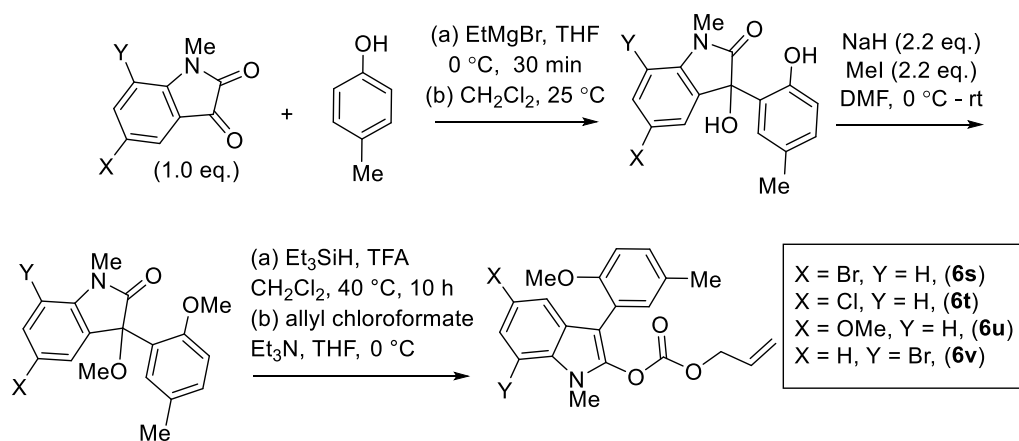
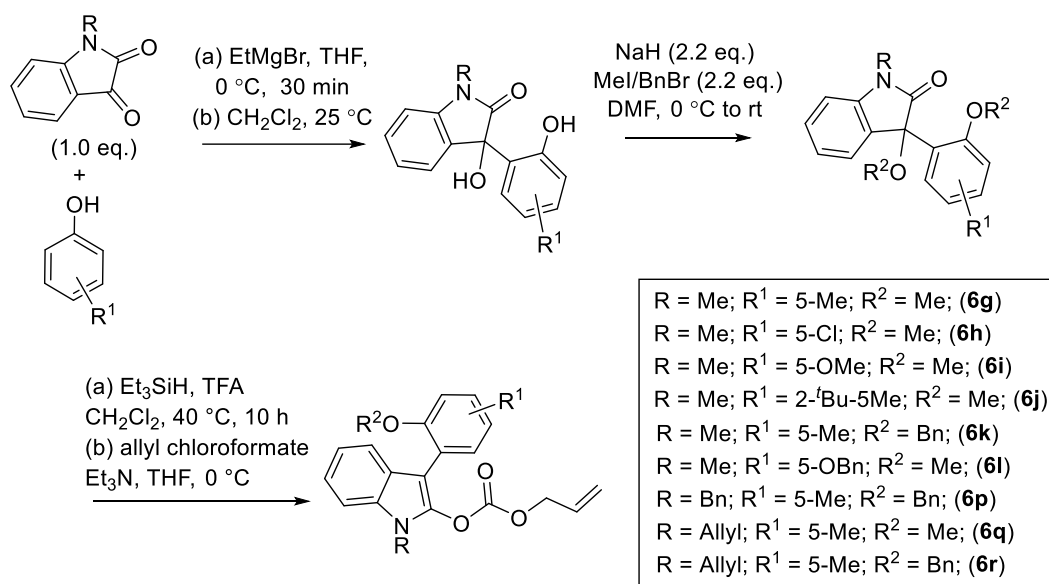
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17 (dd,  $J = 8.4, 0.9$  Hz, 1H), 7.44 – 7.39 (m, 3H), 7.35 (ddd,  $J = 8.5, 7.3, 1.4$  Hz, 1H), 7.28 – 7.24 (m, 1H), 7.09 – 7.04 (m, 2H), 5.98 (ddt,  $J = 17.2, 10.5, 5.8$  Hz, 1H), 5.41 (dd,  $J = 17.2, 1.4$  Hz, 1H), 5.32 (dq,  $J = 10.5, 1.2$  Hz, 1H), 4.77 (dt,  $J = 5.8, 1.4$  Hz, 2H), 3.79 (s, 3H), 1.69 (s, 9H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.5, 152.2, 148.9, 137.6, 132.3, 131.6, 130.8, 129.3, 126.9, 124.3, 123.0, 120.7, 120.2, 119.5, 119.4, 115.5, 111.4, 106.9, 84.5, 69.7, 55.5, 28.2.

**IR** (film)  $\nu_{\text{max}}$  3711, 3328, 2956, 2420, 1776, 1520, 1420, 1310, 1200, 1014, 917, 790, 782, 610  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{24}\text{H}_{26}\text{NO}_6]^+$  424.1755; Found 424.1756.

**General synthetic procedure for synthesis of compound (6g-l and 6p-v):**



A tetrahydrofuran solution of ethyl magnesium bromide (3 M in THF), (1.2 equiv.) was added dropwise to phenolic derivative (1.0 equiv.) in anhydrous THF (10 mL) at 0 °C. The resultant white suspension was concentrated by rotary evaporation to dryness and anhydrous methylene chloride (15 mL) was added and placed this reaction mixture at 0 °C. Solid *N*-alkyl isatin (12.4 mmol; 1.0 equiv.) was added pinch wise to this reaction mixture over 2 minutes. Then the reaction was stirred at room temperature for 12-16 h (TLC showed complete consumption of starting materials under UV light and I<sub>2</sub> stain). Then the reaction mixture was quenched with 1 N hydrochloric acid (5 mL) at room temperature. Then whole reaction mixture was taken into a separatory funnel and the organic layer was separated and dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>. The crude products were

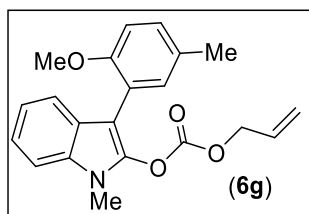
evaporated to dryness under reduced pressure and utilized for next step without purification.

The crude 3-aryl 3'-hydroxy 2-oxindole (12.4 mmol; 1.0 equiv.) was taken in DMF (20 mL) under argon atmosphere. To this reaction solution, NaH (60% in mineral oil) (2.2 equiv.) was added pinch wise at 0 °C for 2 minutes. After 5 minutes of stirring at the same temperature, MeI/BnBr (2.2 equiv.) was added drop wise to the reaction mixture. After 2 h of stirring (TLC showed complete consumption of the starting material), the reaction mixture was quenched by careful addition of water (5 mL), diluted with EtOAc (50 mL). Then the organic layer was separated, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude products were utilized for next step without purification.

The crude hydroxy protected 3-aryl 2-oxindole (12.4 mmol, 1.0 equiv.) was taken in dichloromethane (30 mL) under argon atmosphere. To this reaction solution, trifluoroacetic acid (TFA, 6.0 equiv.) was added dropwise at room temperature. Then triethyl silane (5.0 equiv.) was added dropwise and stirring was continued for 5-8 h. After completion of the reaction (judged by TLC analysis under UV and I<sub>2</sub> stain). 5% (w/v) aqueous solution of sodium citrate (10 mL) was added dropwise to neutralize the mixture followed by addition of 30 mL of dichloromethane. The whole mixture was taken in a separatory funnel and the organic layer was separated. It was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure to get crude products which were utilized for next step without purification.

The crude 3-aryl 2-oxindole (12.4 mmol, 1.0 equiv.) was taken in THF (30 mL) under argon atmosphere at 0 °C. Triethyl amine (5.0 equiv.) was added to the solution and stirred for 10 minutes. After that allyl chloroformate (1.2 equiv.) was added drop-wise over a period of 5 minutes at 0 °C. After completion of reaction (judged by TLC analysis under UV and I<sub>2</sub> stain), the reaction mixture was diluted with EtOAc (50 mL) and quenched with H<sub>2</sub>O. Then the whole reaction mixture was taken in a separatory funnel to separate the organic layer. The organic layer was dried over anhydrous sodium sulphate

and concentrated under reduced pressure. The crude mixture was purified by column chromatography using EtOAc and hexane mixture as eluent to afford the desired product (**6g-l** and **6p-v**).



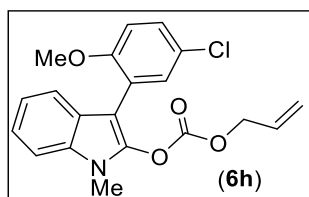
**Allyl (3-(2-methoxy-5-methylphenyl)-1-methyl-1H-indol-2-yl) carbonate (6g):** The compound **6g** was obtained as orange color gel (12.4 mmol, 1.4 g, 32% over four steps).  $R_f = 0.52$  (30% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (d,  $J = 8.5$  Hz, 1H), 7.33 - 7.25 (m, 3H), 7.19 - 7.12 (m, 2H), 6.92 (d,  $J = 8.3$  Hz, 1H), 5.95 (ddt,  $J = 16.4, 10.7, 5.8$  Hz, 1H), 5.41 (dd,  $J = 17.3, 1.7$  Hz, 1H), 5.33 (dd,  $J = 10.6, 1.4$  Hz, 1H), 4.75 (d,  $J = 5.5$  Hz, 2H), 3.76 (s, 3H), 3.69 (s, 3H), 2.37 (s, 3H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0, 152.3, 139.2, 132.8, 131.9, 130.8, 129.9, 128.4, 125.4, 121.8, 121.3, 120.2, 120.2, 119.7, 111.4, 109.1, 99.1, 69.7, 55.7, 28.7, 20.6.

**IR** (film)  $\nu_{\text{max}}$  3726, 3428, 2906, 2440, 1770, 1429, 1401, 1360, 1204, 1124, 901, 750, 610  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{21}\text{H}_{21}\text{NO}_4 + \text{Na}]^+$  374.1363; Found 374.1356.



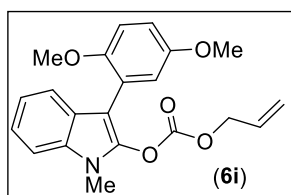
**Allyl (3-(5-chloro-2-methoxyphenyl)-1-methyl-1*H*-indol-2-yl) carbonate (6h):** The compound **6h** was obtained as an orange colored gel. (12.4 mmol, 1.9 g, 41% over four steps).  $R_f = 0.40$  (20% EtOAc in hexane).

**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61 (dt,  $J = 8.0, 1.0$  Hz, 1H), 7.48 (d,  $J = 2.7$  Hz, 1H), 7.36 - 7.28 (m, 3H), 7.21 - 7.18 (m, 1H), 6.95 (d,  $J = 8.8$  Hz, 1H), 5.98 (ddt,  $J = 17.2, 10.4, 5.9$  Hz, 1H), 5.44 (dt,  $J = 17.2, 1.4$  Hz, 1H), 5.37 (dq,  $J = 10.4, 1.2$  Hz, 1H), 4.78 (dt,  $J = 5.9, 1.3$  Hz, 2H), 3.79 (s, 3H), 3.71 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.6, 152.1, 139.4, 132.7, 130.8, 130.6, 127.6, 125.6, 125.0, 123.4, 122.0, 120.5, 120.0, 119.9, 112.5, 109.3, 97.9, 69.9, 55.9, 28.7.

**IR** (film)  $\nu_{\text{max}}$  3771, 3408, 2936, 2408, 1771, 1597, 1468, 1327, 1223, 1027, 947, 806, 740, 602  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{18}\text{ClNO}_4 + \text{Na}]^+$  394.0817; Found 394.0825.



**Allyl (3-(2,5-dimethoxyphenyl)-1-methyl-1*H*-indol-2-yl) carbonate (6i):** The compound **6i** was obtained as an orange color gel. (12.4 mmol scale of reaction, 1.8 g, 39% over four steps).  $R_f = 0.50$  (40% EtOAc in hexane).

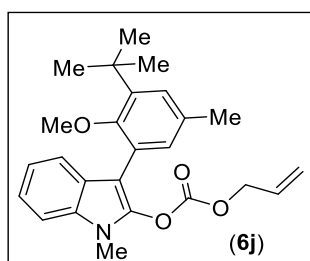
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (d,  $J = 8.0$  Hz, 1H), 7.32 - 7.24 (m, 2H), 7.1 - 7.13 (m, 1H), 7.07 (d,  $J = 3.1$  Hz, 1H), 6.94 (d,  $J = 8.9$  Hz, 1H), 6.85 (dd,  $J = 8.9, 3.1$  Hz, 1H),

5.94 (ddt,  $J = 16.5, 10.4, 5.8$  Hz, 1H), 5.40 (dq,  $J = 17.3, 1.5$  Hz, 1H), 5.31 (dd,  $J = 10.5, 1.3$  Hz, 1H), 4.74 (dt,  $J = 6.0, 1.4$  Hz, 2H), 3.79 (s, 3H), 3.71 (s, 3H), 3.67 (s, 3H).

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.7, 152.2, 151.4, 139.2, 132.8, 130.8, 125.2, 122.6, 121.8, 120.3, 120.1, 119.7, 116.7, 113.0, 112.7, 109.2, 99.0, 69.8, 56.3, 55.8, 28.7.

IR (film)  $\nu_{\text{max}}$  3430, 2835, 2116, 1774, 1846, 1735, 1621, 1477, 1373, 1329, 1225, 1136, 1082, 1047, 944, 842, 780, 741, 642  $\text{cm}^{-1}$ .

HRMS (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{21}\text{H}_{21}\text{NO}_5 + \text{Na}]^+$  390.1312; Found 390.1324.



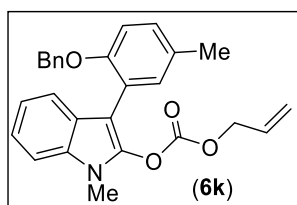
**Allyl (3-(3-(*tert*-butyl)-2-methoxy-5-methylphenyl)-1-methyl-1*H*-indol-2-yl) carbonate (6j):** The compound **6j** was obtained as an orange colored gel. (12.4 mmol scale of reaction; 1.3 g; 26% over four steps).  $R_f = 0.33$  (30% EtOAc in hexane).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 (d,  $J = 7.9$  Hz, 1H), 7.40 – 7.26 (m, 2H), 7.21 (ddd,  $J = 8.3, 7.1, 1.4$  Hz, 1H), 7.16 (dd,  $J = 17.3, 2.3$  Hz, 2H), 5.97 (ddt,  $J = 16.6, 10.4, 5.9$  Hz, 1H), 5.41 (dt,  $J = 17.1, 1.5$  Hz, 1H), 5.37 – 5.23 (m, 1H), 4.74 (d,  $J = 5.9$  Hz, 2H), 3.72 (s, 3H), 3.30 (s, 3H), 2.37 (s, 3H), 1.47 (s, 9H).

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  156.2, 152.2, 142.6, 138.9, 132.9, 132.0, 130.8, 130.4, 126.5, 125.7, 124.8, 121.9, 120.3, 120.2, 119.7, 109.1, 100.7, 69.8, 60.6, 34.9, 30.8, 28.7, 21.2.

**IR** (film)  $\nu_{\max}$  3499, 2908, 2135, 1752, 1620, 1408, 1400, 1301, 1211, 1007, 841, 702, 509  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{25}\text{H}_{29}\text{NO}_4 + \text{Na}]^+$  430.1989; Found 430.1989.



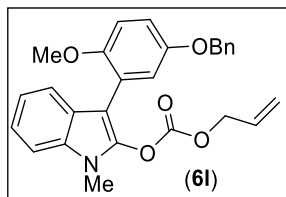
**Allyl (3-(2-(benzyloxy)-5-methylphenyl)-1-methyl-1H-indol-2-yl) carbonate (6k)**: The compound **6k** was obtained as an orange color gel. (12.4 mmol scale of reaction, 3.0 g, 25% over four steps).  $R_f = 0.48$  (30% EtOAc in hexane).

**$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (d,  $J = 1.1$  Hz, 1H), 7.34 – 7.26 (m, 5H), 7.25 – 7.18 (m, 3H), 7.03 (dd,  $J = 8.4, 2.4$  Hz, 1H), 6.88 (d,  $J = 8.4$  Hz, 1H), 5.85 (ddt,  $J = 17.3, 10.4, 5.8$  Hz, 1H), 5.32 (dd,  $J = 17.2, 1.4$  Hz, 1H), 5.26 (dd,  $J = 10.4, 1.3$  Hz, 1H), 5.01 (s, 2H), 4.57 (dt,  $J = 5.8, 1.4$  Hz, 2H), 3.67 (s, 3H), 2.33 (s, 3H).

**$^{13}\text{C}$  NMR** (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.4, 152.2, 139.2, 137.7, 132.7, 132.1, 130.7, 130.5, 128.4, 128.2, 127.3, 126.9, 125.5, 122.2, 121.7, 120.4, 120.1, 119.6, 114.5, 109.0, 99.5, 71.2, 69.6, 28.6, 20.6.

**IR** (film)  $\nu_{\max}$  3394, 2909, 2118, 1754, 1622, 1408, 1301, 1210, 1083, 911, 702, 509  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{27}\text{H}_{25}\text{NO}_4 + \text{Na}]^+$  450.1676; Found 450.1696.



**Allyl (3-(5-(benzyloxy)-2-methoxyphenyl)-1-methyl-1H-indol-2-yl) carbonate (6l):**

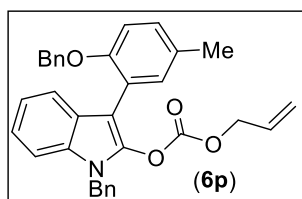
The compound **6l** was obtained as an orange color gel. (12.4 mmol scale of reaction, 1.4 g, 25% over four steps).  $R_f = 0.40$  (40% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.48 - 7.30 (m, 6H), 7.24 (ddd,  $J = 8.1, 6.1, 1.2$  Hz, 1H), 7.13 - 7.08 (m, 2H), 6.92 (d,  $J = 1.7$  Hz, 2H), 5.93 (ddt,  $J = 17.2, 10.4, 5.8$  Hz, 1H), 5.39 (dd,  $J = 17.2, 1.4$  Hz, 1H), 5.31 - 5.28 (m, 1H), 5.05 (s, 2H), 4.73 (dt,  $J = 5.9, 1.4$  Hz, 2H), 3.70 (s, 3H), 3.66 (s, 3H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  152.8, 152.2, 151.5, 139.2, 137.4, 132.8, 130.7, 128.6, 127.8, 127.5, 125.1, 122.5, 121.8, 120.3, 120.0, 119.7, 117.7, 114.3, 112.7, 109.1, 98.9, 70.6, 69.8, 56.3, 28.7.

**IR** (film)  $\nu_{\text{max}}$  3432, 2110, 1744, 1647, 1498, 1464, 1366, 1225, 1080, 1041, 1029, 946, 740, 698  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{27}\text{H}_{26}\text{NO}_5]^+$  444.1805; Found 444.1819.



**Allyl (1-benzyl-3-(2-(benzyloxy)-5-methylphenyl)-1H-indol-2-yl) carbonate (6p):** The compound **6p** was obtained as an orange color gel. (12.4 mmol scale of reaction, 1.8 g, 30% over four steps).  $R_f = 0.40$  (40% EtOAc in hexane).

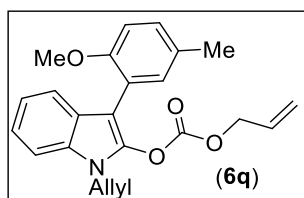


**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.68 (d, *J* = 7.7 Hz, 1H), 7.38 (d, *J* = 2.1 Hz, 1H), 7.30 – 7.14 (m, 13H), 7.09 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 5.77 (ddt, *J* = 16.5, 11.0, 5.7 Hz, 1H), 5.32 – 5.21 (m, 4H), 5.04 (s, 2H), 4.50 (d, *J* = 5.7 Hz, 2H), 2.37 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 154.4, 151.6, 139.0, 137.6, 136.9, 132.2, 132.1, 130.6, 130.5, 128.7, 128.5, 128.2, 127.4, 127.3, 127.0, 126.7, 125.9, 122.1, 121.8, 120.3, 120.2, 119.4, 114.2, 109.7, 99.9, 71.1, 69.6, 46.1, 20.6.

**IR** (film)  $\nu_{\max}$  3746, 3408, 2900, 2440, 1760, 1420, 1400, 1361, 1214, 1144, 981, 754, 620 cm<sup>-1</sup>.

**HRMS** (ESI-TOF) *m/z*: [M + Na]<sup>+</sup> Calcd for [C<sub>33</sub>H<sub>29</sub>NO<sub>4</sub> + Na]<sup>+</sup> 526.1989; Found 526.1987.



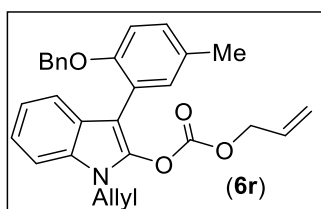
**Allyl (1-allyl-3-(2-methoxy-5-methylphenyl)-1H-indol-2-yl) carbonate (6q)**: The compound (**6q**) was obtained as an orange color gel. (12.4 mmol scale of reaction, 1.3 g, 27% over four steps). *R<sub>f</sub>* = 0.43 (40% EtOAc in hexane).

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.70 - 7.66 (m, 1H), 7.39 - 7.66 (m, 2H), 7.32 - 7.27 (m, 1H), 7.24 - 7.16 (m, 2H), 6.98 - 6.94 (m, 1H), 6.08 - 6.01 (m, 1H), 5.99 - 5.91 (m, 1H), 5.44 - 5.39 (m, 1H), 5.37 - 5.33 (m, 1H), 5.29 - 5.23 (m, 2H), 4.77 - 4.73 (m, 4H), 3.81 - 3.78 (m, 3H), 2.42 - 2.40 (m, 3H).

**$^{13}\text{C}$  NMR** (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.0, 152.1, 138.8, 132.9, 132.3, 131.9, 130.8, 129.9, 128.5, 125.7, 121.8, 121.3, 120.3, 120.3, 119.6, 117.3, 111.4, 109.7, 99.6, 69.7, 55.7, 45.2, 20.6.

**IR** (film)  $\nu_{\text{max}}$  3434, 2949, 2128, 1774, 1624, 1498, 1464, 1361, 1212, 1087, 941, 742, 559  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{23}\text{H}_{24}\text{NO}_4]^+$  378.1700; Found 378.1709.



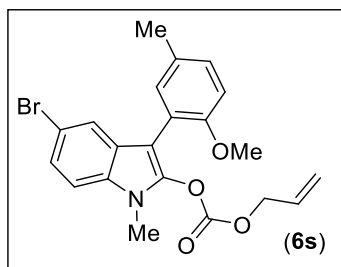
**Allyl (1-allyl-3-(2-(benzyloxy)-5-methylphenyl)-1H-indol-2-yl) carbonate (6r)**: The compound **6r** was obtained as an orange colored gel. (12.4 mmol scale of reaction, 1.5 g, 27% over four steps).  $R_f = 0.42$  (30% EtOAc in hexane).

**$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta = 7.63$  (d,  $J = 8.0$ , 1H), 7.32 – 7.26 (m, 4H), 7.22 - 7.21 m, 4H), 7.14 (t,  $J = 7.5$ , 1H), 7.07 – 7.02 (m, 1H), 6.91 (d,  $J = 8.3$ , 1H), 5.97 (ddd,  $J = 16.0$ , 10.5, 5.2, 1H), 5.82 (ddt,  $J = 16.5$ , 10.9, 5.7, 1H), 5.31 – 5.112 (m, 4H), 5.01 (s, 2H), 4.70 (d,  $J = 5.2$ , 2H), 4.56 (d,  $J = 5.8$ , 2H), 2.34 (s, 3H).

**$^{13}\text{C}$  NMR** (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.4, 151.9, 138.9, 137.7, 132.8, 132.1, 130.7, 130.5, 128.5, 128.2 (2C), 127.3, 126.9, 125.8, 122.2, 121.7, 120.4, 120.2, 119.5, 117.2, 114.4, 109.5, 99.8, 71.2, 69.6, 45.0, 20.6.

**IR** (film)  $\nu_{\text{max}}$  3630, 3049, 2928, 1754, 1644, 1508, 1464, 1361, 1262, 1187, 941, 792, 659  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[M + Na]^+$  Calcd for  $[C_{29}H_{27}NO_4 + Na]^+$  476.1832; Found 476.1809.



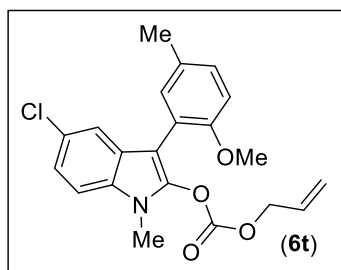
**Allyl (5-bromo-3-(2-methoxy-5-methylphenyl)-1-methyl-1H-indol-2-yl) carbonate (6s)**: The compound **6s** was obtained as yellow liquid (0.58 mmol scale of reaction, 0.221 g, 89% over four steps).  $R_f = 0.65$  (40% EtOAc in hexane).

**$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta = 7.77 - 7.58$  (m, 1H), 7.38 – 7.29 (m, 2H), 7.25 – 7.11 (m, 2H), 6.93 (dd,  $J = 8.4, 1.6$ , 1H), 6.08 – 5.82 (m, 1H), 5.52 – 5.21 (m, 2H), 4.76 (ddt,  $J = 5.7, 4.2, 1.4$ , 2H), 3.77 (s, 3H), 3.69 (d,  $J = 10.7$ , 3H), 2.38 (d,  $J = 2.6$ , 3H).

**$^{13}C$  NMR** (125 MHz,  $CDCl_3$ )  $\delta$  155.0, 152.0, 134.0, 132.0, 130.6, 129.9, 129.5, 128.8, 128.4, 127.0, 121.0, 120.4, 119.8, 119.5, 111.3, 103.7, 99.9, 69.9, 55.7, 53.4, 20.5.

**IR** (film) 2618, 2092, 1782, 1595, 1469, 1326, 1196, 1101, 569  $cm^{-1}$ .

**HRMS** (ESI)  $m/z$   $[M + H]^+$  Calcd for  $[C_{21}H_{20}BrNO_4 + H]^+$  430.0648; Found 430.0659.



**Allyl (5-chloro-3-(2-methoxy-5-methylphenyl)-1-methyl-1*H*-indol-2-yl) carbonate**

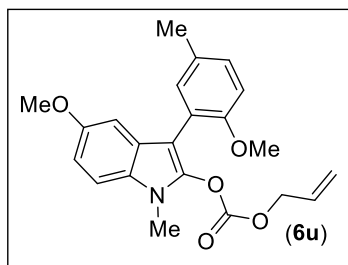
**(6t):** The compound **6t** was obtained as colorless gel (0.58 mmol scale of reaction, 0.116 g, 63% over four steps).  $R_f = 0.55$  (40% EtOAc in hexane).

**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 – 7.53 (m, 1H), 7.35 (dd,  $J = 8.7, 1.9$ , 1H), 7.25 – 7.17 (m, 2H), 7.14 (dd,  $J = 8.3, 2.3$ , 1H), 6.92 (d,  $J = 8.3$ , 1H), 5.95 (ddtd,  $J = 17.2, 10.4, 5.9, 0.9$ , 1H), 5.48 – 5.10 (m, 2H), 4.75 (dt,  $J = 5.9, 1.3$ , 2H), 3.77 (s, 3H), 3.68 (s, 3H), 2.37 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.8, 152.0, 139.9, 131.7, 130.6, 128.7, 129.8, 125.9, 126.3, 124.6, 122.1, 120.5, 119.8, 119.7, 111.3, 110.2, 99.2, 69.9, 55.7, 28.8, 20.6.

**IR** (film) 2526, 1991, 1765, 1428, 1169, 1210, 1053, 952, 542  $\text{cm}^{-1}$ .

**HRMS** (ESI)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{21}\text{H}_{20}\text{ClNO}_4 + \text{H}]^+$  386.1154; Found 386.1144.

**Allyl (5-methoxy-3-(2-methoxy-5-methylphenyl)-1-methyl-1*H*-indol-2-yl) carbonate**

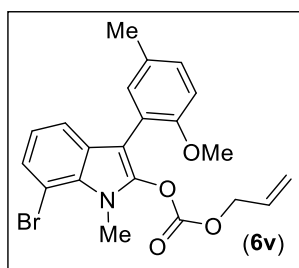
**(6u):** The compound **6u** was obtained as yellowish gel (0.30 mmol scale of reaction, 0.069 g, 61% over four steps).  $R_f = 0.55$  (30% EtOAc in hexane).

**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.31 (d,  $J = 12.9$ , 1H), 7.22 (d,  $J = 8.9$ , 1H), 7.13 (dd,  $J = 8.4, 2.3$ , 1H), 7.09 (d,  $J = 2.5$ , 1H), 6.93 (dd,  $J = 8.5, 1.8$ , 2H), 5.95 (ddt,  $J = 17.4, 10.6, 5.8$ , 1H), 5.47 – 5.25 (m, 2H), 4.75 (dt,  $J = 5.8, 1.4$ , 2H), 3.83 (s, 3H), 3.77 (s, 3H), 3.66 (s, 3H), 2.36 (s, 3H).

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>)  $\delta$  = 154.9, 154.6, 152.2, 139.5, 131.7, 130.7, 129.9, 128.3, 127.9, 125.6, 121.4, 119.7, 111.7, 111.3, 109.9, 102.7, 99.1, 69.7, 68.0, 56.1, 55.6, 25.6, 20.6.

**IR** (film) 2245, 1569, 1499, 1454, 1219, 1056, 1068, 584 cm<sup>-1</sup>.

**HRMS** (ESI)  $m/z$  [M + H]<sup>+</sup> Calcd for [C<sub>22</sub>H<sub>23</sub>NO<sub>5</sub> + H]<sup>+</sup> 382.1649; Found 382.1647.



**Allyl (7-bromo-3-(2-methoxy-5-methylphenyl)-1-methyl-1H-indol-2-yl) carbonate (6v):** The compound **6v** was obtained as yellow liquid (0.30 mmol scale of reaction; 0.094 g; 76% over four steps).  $R_f$  = 0.65 (30% EtOAc in hexane).

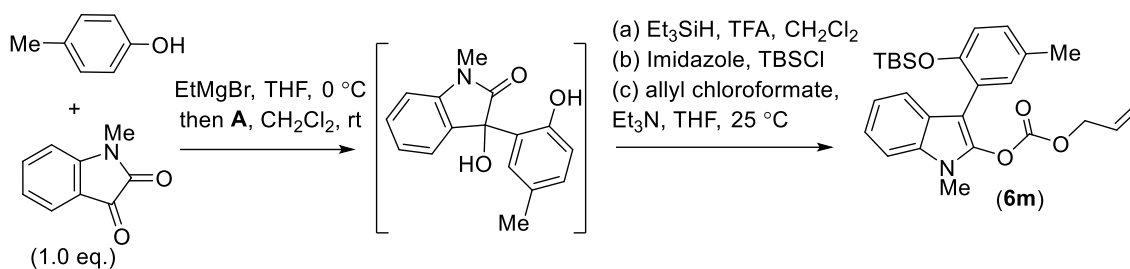
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.48 (dd,  $J$  = 7.9, 1.1, 1H), 7.38 (dd,  $J$  = 7.7, 1.1, 1H), 7.21 (d,  $J$  = 2.1, 1H), 7.14 (ddd,  $J$  = 8.3, 2.4, 0.8, 1H), 7.00 – 6.89 (m, 2H), 5.94 (ddt,  $J$  = 17.2, 10.5, 5.8, 1H), 5.50 – 5.23 (m, 2H), 4.74 (dt,  $J$  = 5.8, 1.4, 2H), 4.04 (s, 3H), 3.75 (s, 3H), 2.35 (s, 3H).

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>)  $\delta$  155.0, 152.0, 134.0, 132.0, 130.6, 129.9, 129.5, 128.8, 128.4, 127.0, 121.0, 120.4, 119.8, 119.5, 111.3, 103.7, 99.9, 69.9, 55.7, 53.4, 20.5.

**IR** (film) 2622, 2063, 1690, 1411, 1215, 1100, 1023, 514 cm<sup>-1</sup>.

**HRMS** (ESI)  $m/z$  [M + H]<sup>+</sup> Calcd for [C<sub>21</sub>H<sub>20</sub>BrNO<sub>4</sub> + Na]<sup>+</sup> 452.0468; Found 452.0525.

**Synthetic procedure for synthesis of compound (6m):**

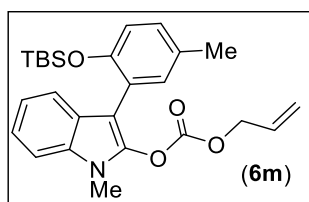


In an oven dried round bottom flask *p*-Cresol (2.0 g; 12.4 mmol; 1.0 equiv.) was taken in anhydrous THF (20 mL) and the reaction vessel was cooled at 0 °C. To this solution, ethyl magnesium bromide (3M in THF) (5.0 mL; 14.9 mmol; 1.2 equiv.) was added dropwise over a period of 2 minutes. The resultant white suspension was concentrated by rotary evaporation to dryness and immediately dissolved in anhydrous methylene chloride (30 mL). To this solution was added solid *N*-methyl isatin (2.0 g; 12.4 mmol; 1.0 equiv.) pinch wise over a period of 2 minutes. The reaction was allowed to warm to room temperature and stirring was continued for 10 h. After completion of the reaction (TLC showed complete consumption of starting materials), 1N hydrochloric acid (5 mL) was added to the reaction mixture and whole mixture was taken in a separatory funnel. Then the organic layer was separated, dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and evaporated to dryness under reduced pressure. The crude products were utilized for next step without purification.

Crude 3-hydroxy 2-oxindole (12.4 mmol; 1.0 equiv.) was taken in dichloromethane (50 mL) and the reaction mixture was cooled to 0 °C. Trifluoroacetic acid (TFA) (12.6 mL; 74.4 mmol; 6.0 equiv.) was added to the reaction mixture drop-wise over a period of 2 minutes at same temperature. Then triethyl silane (9.9 mL; 62.05 mmol; 5.0 equiv.) was added to the mixture at 0 °C for 2 minutes and stirring was continued for 3 h. After completion of the reaction (judged by TLC analysis under UV and I<sub>2</sub> stain), 5% (w/v) aqueous solution of sodium citrate (10 mL) was added drop wise to neutralize the mixture. Then whole reaction mixture was taken in separatory funnel and the organic layer was separated. It was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude products were utilized for next step without purification.

The crude 3-aryl 2-oxindole (12.4 mmol; 1.0 equiv.) was taken in dichloromethane (50 mL) under argon atmosphere at rt. Then imidazole (5.1 gm; 74.4 mmol; 6.0 equiv.) was added to the reaction mixture and stirred for 10 minutes. Then *tert*-butyl dimethyl chlorosilane (TBSCl) (3.7 gm; 24.8 mmol; 2.0 equiv.) was added to the reaction mixture and stirring was continued for 10 h. After completion of the reaction, (judged by TLC analysis under UV and I<sub>2</sub> stain), water (20 mL) was added and whole reaction mixture was taken in a separatory funnel. The organic layer was separated and dried over anhydrous sodium sulphate and concentrated under reduced pressure. The crude products were utilized for next step without purification.

The crude TBS protected product (12.4 mmol; 1.0 equiv. as prepared earlier) was taken in THF (60 mL) under argon atmosphere at 0 °C. Then triethyl amine (8.7 mL; 62.0 mmol; 5.0 equiv.) was added to this solution. After 10 minutes of stirring allyl chloroformate (1.6 mL; 14.9 mmol; 1.2 equiv.) was added drop-wise over a period of 5 minutes at 0 °C. After completion of reaction (judged by TLC analysis under UV and I<sub>2</sub> stain), it was diluted with EtOAc (50 mL) and quenched with H<sub>2</sub>O (30 mL). Then the whole reaction mixture was taken in a separatory funnel to separate the organic layer. The organic layer was dried over anhydrous sodium sulphate and concentrated under reduced pressure. The crude mixture was purified by column chromatography using EtOAc and hexane mixture as eluent to afford the desired product (**6m**).



**Allyl (3-(2-((*tert*-butyldimethylsilyloxy)-5-methylphenyl)-1-methyl-1*H*-indol-2-yl) carbonate (6m):** The compound **6m** was obtained as an orange color gel. (12.4 mmol scale of reaction; 2.1 g of product; 38% over four steps);  $R_f = 0.39$  (10% EtOAc in hexane).

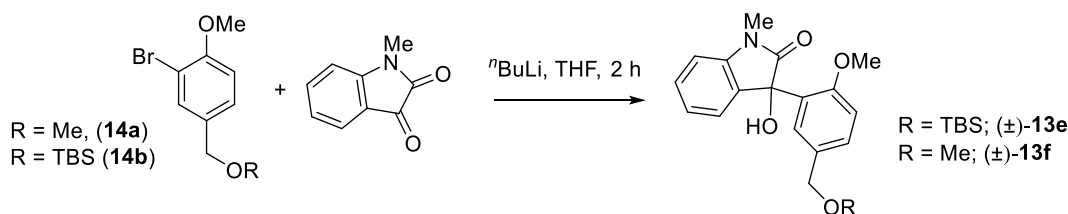
**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 (d,  $J = 7.9$  Hz, 1H), 7.30 (d,  $J = 7.7$  Hz, 1H), 7.27 - 7.23 (m, 1H), 7.21 (d,  $J = 2.2$  Hz, 1H), 7.16 - 7.10 (m, 1H), 7.03 (dd,  $J = 8.3, 2.2$  Hz, 1H), 6.85 (d,  $J = 8.3$  Hz, 1H), 5.96 - 5.86 (m, 1H), 5.37 (d,  $J = 17.2$  Hz, 1H), 5.30 (d,  $J = 10.2$  Hz, 1H), 4.70 (d,  $J = 5.7$  Hz, 2H), 3.68 (s, 3H), 2.34 (s, 3H), 0.72 (s, 9H), -0.18 (s, 6H).

**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  152.0, 151.5, 138.9, 132.4, 131.9, 130.7, 130.4, 128.5, 125.8, 124.1, 121.5, 121.0, 120.2, 119.9, 119.5, 108.6, 100.5, 69.6, 28.4, 25.5, 20.7, 17.9, -4.9.

**IR** (film)  $\nu_{\text{max}}$  3400, 1702, 1600, 1409, 1205, 1070, 814, 617  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{26}\text{H}_{33}\text{NO}_4\text{Si} + \text{Na}]^+$  474.2071; Found 474.2092.

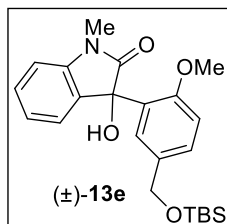
**General procedure for the synthesis of compound (13e-f):**



Compound **14a** (15.0 mmol, 1.0 equiv.) was taken in THF (60 mL) and the reaction mixture was cooled to 0 °C. To this solution,  $n\text{BuLi}$  (1.2 equiv.) was added drop wise at 0 °C over a period of 15 min and stirred for another 10 minutes. Then, THF solution (10 mL) of *N*-methyl isatin (1.0 equiv.) was added at 0 °C. The reaction mixture was allowed to warm to room temperature and the stirring was continued for 6 h. After completion of the reaction (monitoring by TLC under UV light and  $\text{I}_2$  stain), it was quenched by slow addition of water (10 mL) and diluted with ethyl acetate (75 mL). The whole reaction mixture was taken in a separatory funnel and washed with brine (60 mL). Then the organic layer was separated, dried with anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under



reduced pressure. The crude product was purified by flash chromatography with *n*-Hexane-EtOAc (7:3) to afford ( $\pm$ )-**13e-f** as yellow gel.



**3-(5-((*tert*-Butyldimethylsilyl)oxy)methyl)-2-methoxyphenyl)-3-hydroxy-1-**

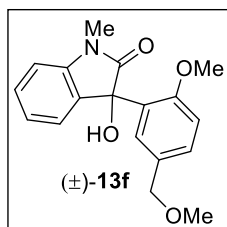
**methylindolin-2-one [( $\pm$ )-**13e**]:** The compound ( $\pm$ )-**13e** was obtained as orange solid (15.0 mmol scale of reaction; 1.98 g of product; 32% over four steps).  $R_f = 0.10$  (30% EtOAc in hexane).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.55 (d,  $J = 2.2$  Hz, 1H), 7.30 (td,  $J = 7.8, 1.3$  Hz, 1H), 7.23 (d,  $J = 2.1$  Hz, 1H), 7.13 (dd,  $J = 7.4, 1.3$  Hz, 1H), 7.03 – 6.94 (m, 1H), 6.85 (d,  $J = 7.8$  Hz, 1H), 6.80 (d,  $J = 8.4$  Hz, 1H), 4.69 (s, 2H), 3.62 (s, 3H), 3.25 (s, 3H), 0.91 (s, 9H), 0.08 (s, 3H), -0.01 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  177.4, 155.5, 144.3, 134.1, 130.6, 129.7, 128.5, 127.3, 124.9, 124.5, 122.9, 111.9, 108.0, 64.7, 56.3, 26.3, 25.9, 18.4, 0.0, -5.2.

**IR** (film)  $\nu_{\max}$  3430, 2925, 2866, 2010, 1716, 1610, 1404, 1269, 1238, 1024, 1001, 937, 857, 710, 619, 501cm<sup>-1</sup>.

**HRMS** (ESI-TOF)  $m/z$ : [M + H]<sup>+</sup> Calcd for [C<sub>23</sub>H<sub>32</sub>NO<sub>4</sub>Si]<sup>+</sup> 414.2095; Found 414.2079.



**3-Hydroxy-3-(2-methoxy-5-(methoxymethyl)phenyl)-1-methylindolin-2-one [(±)-13f]**: The compound (±)-**13f** was obtained as orange solid (15.0 mmol scale of reaction; 1.4 g of product; 30% over four steps).  $R_f = 0.50$  (30% EtOAc in hexane).

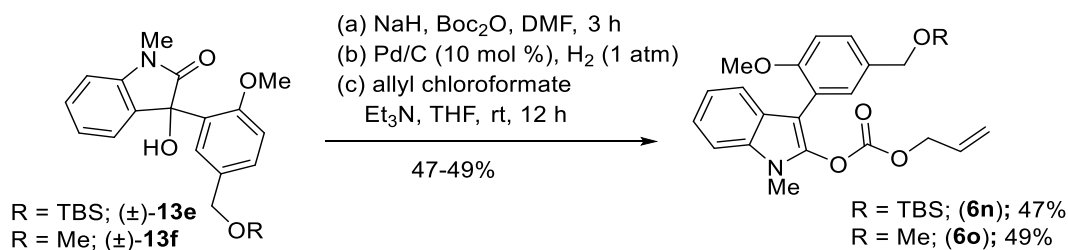
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 2.2$  Hz, 1H), 7.30 – 7.22 (m, 2H), 7.09 (d,  $J = 7.3$  Hz, 1H), 6.96 (t,  $J = 7.5$  Hz, 1H), 6.84 (d,  $J = 7.8$  Hz, 1H), 6.77 (d,  $J = 8.3$  Hz, 1H), 4.41 (s, 2H), 3.56 (s, 3H), 3.36 (s, 3H), 3.25 (s, 3H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  177.5, 155.7, 144.3, 130.7, 130.6, 129.6, 129.1, 129.0, 126.7, 124.3, 122.9, 111.8, 108.0, 76.3, 74.4, 57.9, 56.2, 26.3.

**IR** (film)  $\nu_{\text{max}}$  3444, 2910, 2846, 2016, 1714, 1614, 1404, 1263, 1228, 1014, 1001, 947, 837, 726, 669,  $521\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{18}\text{H}_{20}\text{NO}_4]^+$  314.1387; Found 314.1401.

### General procedure for the synthesis of compound (6n-o):

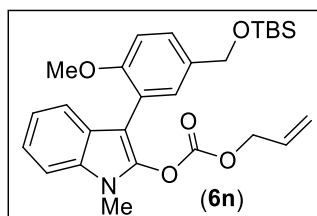


An oven-dried round-bottom flask was charged with (±)-**13e-f** (1.8 mmol; 1.0 equiv) in DMF (40 mL) under nitrogen atmosphere at room temperature. To this solution, NaH (60% dispersion in mineral oil) (1.5 equiv.) was added pinch wise. After 5 minutes of stirring, di-*tert*-butyl dicarbonate (1.5 equiv.) was added drop-wise over a period of 2 minutes. Then the reaction mixture was placed to a preheated oil bath maintaining  $60^\circ\text{C}$  and stirring continued for 3 h. Upon completion of the reaction (judged by TLC under

UV light and I<sub>2</sub> stain), the reaction mixture was quenched with H<sub>2</sub>O (50 mL) and diluted with EtOAc (60 mL). Then the organic layer was separated, dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude product was directly charged for the next step.

The crude Boc protected compound (1.8 mmol; 1.0 equiv.) was taken in MeOH (10 mL) at room temperature. The solution was degassed by using argon balloon over a period of 10 minutes. Pd on activated charcoal [40 mg, 10% (w/w)] was added to the degassed solution under nitrogen atmosphere. The reaction vessel was purged with hydrogen gas for 10 minutes and stirring was continued for additional 3 h. Upon completion of the reaction (judged by TLC under UV light and I<sub>2</sub> stain), the reaction mixture was filtered through celite and concentrated under rotatory evaporator. The crude product was utilized for next step without purification.

The crude 3-aryl 2-oxindole (1.8 mmol scale; 1.0 equiv. as prepared earlier) was taken in THF (40 mL) under nitrogen atmosphere at 0 °C. Then Et<sub>3</sub>N (0.75 mL; 5.4 mmol; 3.0 equiv.) was added to the solution. After 5 minutes of stirring, allyl chloroformate (0.25 mL; 2.15 mmol; 1.2 equiv.) was added drop-wise over a period of 2 minutes at 0 °C and stirring was continued. Upon completion of the reaction (judged by TLC under UV light and I<sub>2</sub> stain), it was quenched with H<sub>2</sub>O and diluted with EtOAc (40 mL). Then the organic layer was separated, dried with anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The crude product was purified by flash chromatography with *n*-Hexane-EtOAc (6:4) to afford (**6n-o**) as yellow gel.



**Allyl (3-(5-(((*tert*-butyldimethylsilyl)oxy)methyl)-2-methoxyphenyl)-1-methyl-1*H*-indol-2-yl) carbonate (6n)**: The compound **6n** was obtained as orange color gel (1.8

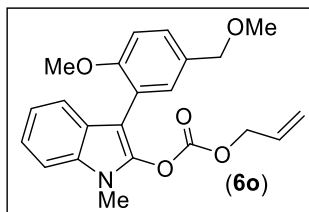
mmol scale of reaction; 0.4 g of product; 47% over four steps).  $R_f = 0.50$  (30% EtOAc in hexane).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (d,  $J = 7.9$  Hz, 1H), 7.46 (d,  $J = 2.1$  Hz, 1H), 7.34 - 7.26 (m, 4H), 7.18 - 7.14 (m, 1H), 7.00 (d,  $J = 8.4$  Hz, 1H), 5.96 (ddt,  $J = 16.5, 11.1, 5.7$  Hz, 1H), 5.42 (dt,  $J = 17.2, 1.5$  Hz, 1H), 5.34 (d,  $J = 10.4$  Hz, 1H), 4.76 (d,  $J = 4.4$  Hz, 4H), 3.79 (s, 3H), 3.70 (s, 3H), 0.98 (d,  $J = 1.5$  Hz, 9H), 0.14 (d,  $J = 1.4$  Hz, 6H).

$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  156.0, 152.2, 139.1, 133.6, 132.8, 130.8, 129.5, 126.0, 125.3, 121.7, 121.3, 120.1(2C), 119.7, 111.1, 109.0, 99.0, 69.7, 64.8, 55.7, 28.7, 26.0, - 5.1.

**IR** (film)  $\nu_{\text{max}}$  3434, 2930, 2856, 2116, 1774, 1624, 1464, 1363, 1328, 1226, 1084, 1031, 947, 837, 776, 741, 669,  $561\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{27}\text{H}_{35}\text{NO}_5\text{Si} + \text{Na}]^+$  504.2177; Found 504.2171.



**Allyl (3-(2-methoxy-5-(methoxymethyl)phenyl)-1-methyl-1H-indol-2-yl) carbonate (6o)**: The compound **6o** was obtained as orange color gel (1.8 mmol scale of reaction; 0.34g of product; 49% over four steps).  $R_f = 0.48$  (30% EtOAc in hexane).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 (dt,  $J = 8.0, 1.0$  Hz, 1H), 7.48 (d,  $J = 2.2$  Hz, 1H), 7.36 - 7.28 (m, 3H), 7.18 (ddd,  $J = 8.1, 7.0, 1.2$  Hz, 1H), 7.02 (d,  $J = 8.4$  Hz, 1H), 5.97 (ddt,  $J = 17.3, 10.6, 5.8$  Hz, 1H), 5.43 (dt,  $J = 17.2, 1.4$  Hz, 1H), 5.35 (dt,  $J = 10.5, 1.2$

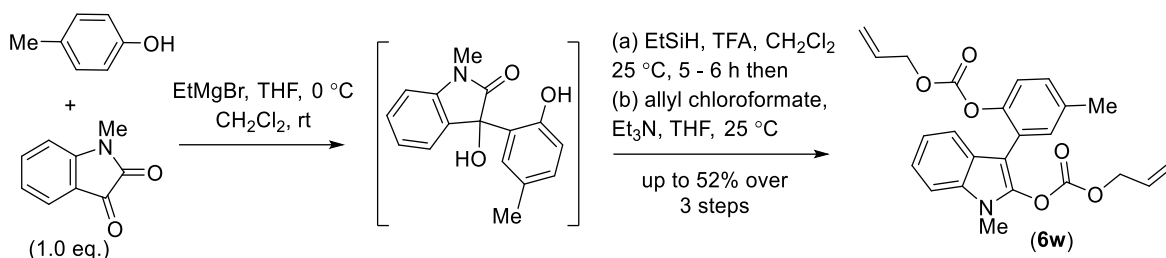
Hz, 1H), 4.77 (dt,  $J = 5.8, 1.4$  Hz, 2H), 4.48 (s, 2H), 3.81 (s, 3H), 3.71 (s, 3H), 3.42 (s, 3H).

$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  156.6, 152.2, 139.2, 132.8, 131.2, 130.7, 130.3, 127.8, 125.3, 121.8, 121.4, 120.2, 120.1, 119.8, 111.3, 109.1, 98.9, 74.4, 69.8, 57.7, 55.7, 28.7.

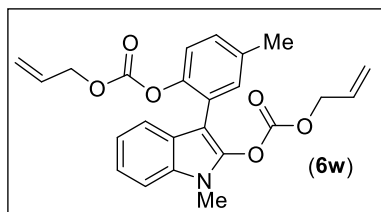
IR (film)  $\nu_{\text{max}}$  3474, 3050, 2929, 2272, 1777, 1589, 1464, 1367, 1228, 1086, 984, 748, 561  $\text{cm}^{-1}$ .

HRMS (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{22}\text{H}_{23}\text{NO}_5 + \text{Na}]^+$  404.1468; Found 404.1457.

### Synthesis of compound **6w**:



Synthetic procedure for the compound **6w** (5.0 mmol; 52% yield over 3 steps) is similar as synthesis of compound **6e-j**.



**Allyl (3-(2-(((allyloxy)carbonyloxy)-5-methylphenyl)-1-methyl-1H-indol-2-yl) carbonate (6w)**: The compound **6w** was obtained as orange color gel (5.0 mmol scale of reaction; 0.5g of product; 48% over three steps).  $R_f = 0.38$  (10% EtOAc in hexane).

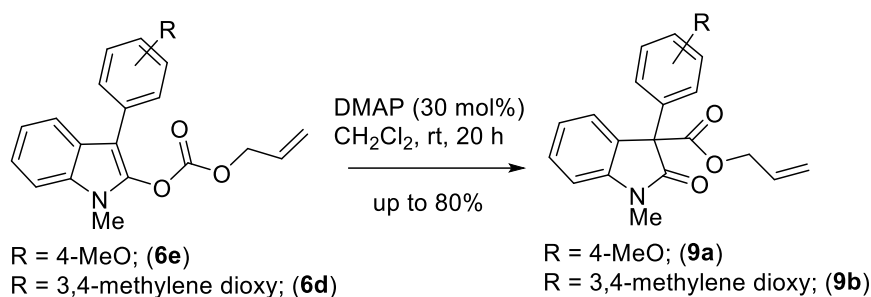
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.57 (d,  $J$  = 7.9, 1H), 7.34 (s, 1H), 7.31 – 7.20 (m, 2H), 7.15 – 7.12 (m, 3H), 5.92 (ddt,  $J$  = 16.5, 10.4, 5.8, 1H), 5.78 (ddt,  $J$  = 16.5, 11.0, 5.7, 1H), 5.36 (dd,  $J$  = 17.2, 1.6, 1H), 5.31 – 5.25 (m, 1H), 5.19 (dd,  $J$  = 17.2, 1.6, 1H), 5.14 (dd,  $J$  = 10.4, 1.4, 1H), 4.69 (dt,  $J$  = 5.8, 1.4, 2H), 4.54 (dt,  $J$  = 5.7, 1.4, 2H), 3.63 (s, 3H), 2.37 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  153.5, 151.9, 146.7, 139.2, 135.8, 132.7, 132.1, 131.4, 130.7, 128.7, 125.2, 124.8, 122.3, 122.0, 120.5, 119.8, 119.7, 118.6, 109.1, 98.2, 69.9, 68.8, 28.6, 20.9.

**IR** (film)  $\nu_{\max}$  3432, 2927, 2311, 2210, 1711, 1635, 1459, 1388, 1207, 1093, 1000, 888, 701, 509 cm<sup>-1</sup>.

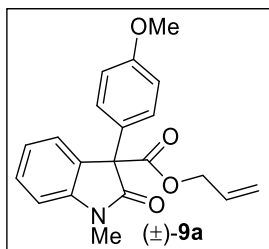
**HRMS** (ESI-TOF)  $m/z$ : [M + H]<sup>+</sup> Calcd for [C<sub>24</sub>H<sub>24</sub>NO<sub>6</sub>]<sup>+</sup> 422.1598; Found 422.1621.

**General procedure for the synthesis of compounds ( $\pm$ )-**9a-b**:**



An oven dried round bottom flask was charged with compound **6d-e** (1.5 mmol; 1.0 equiv.) in dichloromethane (15 mL) under nitrogen atmosphere at room temperature. *N,N*-dimethyl amino pyridine (DMAP, 0.3 equiv.) was added to the solution and stirring was continued for 20 h at same temperature. Upon completion of the reaction (judged by TLC analysis), the reaction mixture was evaporated and the crude product was purified

by column chromatography by using EtOAc and hexane as an eluent to afford the desired product ( $\pm$ )-**9a-b**.



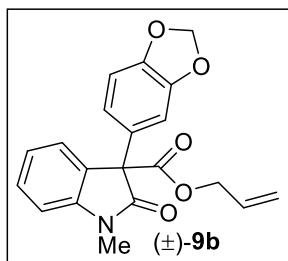
**Allyl 3-(4-methoxyphenyl)-1-methyl-2-oxindoline-3-carboxylate** [( $\pm$ )-**9a**]: The compound ( $\pm$ )-**9a** was obtained as orange color gel (1.5 mmol scale of reaction; 0.4g of product; 80%).  $R_f$  = 0.46 (40% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44 (dd,  $J$  = 7.5, 1.2 Hz, 1H), 7.40 (td,  $J$  = 7.7, 1.3 Hz, 1H), 7.26 (dd,  $J$  = 8.8, 2.0 Hz, 2H), 7.15 (td,  $J$  = 7.6, 1.1 Hz, 1H), 6.91 (d,  $J$  = 7.8 Hz, 1H), 6.88 – 6.81 (m, 2H), 5.79 (ddt,  $J$  = 17.2, 10.7, 5.4 Hz, 1H), 5.17 (dt,  $J$  = 12.5, 1.5 Hz, 1H), 5.15 – 5.11 (m, 1H), 4.76 – 4.49 (m, 2H), 3.76 (s, 3H), 3.21 (s, 3H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  173.1, 169.0, 159.5, 144.4, 131.3, 129.6, 129.1, 127.7, 127.1, 125.9, 122.8, 118.4, 113.9, 108.7, 66.4, 63.3, 55.3, 26.7.

**IR** (film)  $\nu_{\text{max}}$  3932, 2827, 2411, 2440, 1722, 1645, 1559, 1488, 1117, 1003, 990, 808, 700, 505  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{20}\text{NO}_4]^+$  338.1387; Found 338.1374.



**Allyl 3-(benzo[*d*][1,3]dioxol-5-yl)-1-methyl-2-oxindoline-3-carboxylate [(±)-**9b**]:** The compound (±)-**9b** was obtained as orange color gel (1.5 mmol scale of reaction; 0.5g of product; 48% over three steps).  $R_f = 0.45$  (40% EtOAc in hexane).

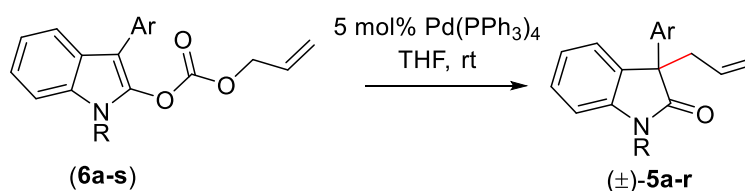
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49 – 7.40 (m, 1H), 7.39 (td,  $J = 7.7, 1.2$  Hz, 1H), 7.22 – 7.08 (m, 1H), 6.91 (dd,  $J = 4.9, 3.0$  Hz, 2H), 6.81 – 6.65 (m, 2H), 5.91 (d,  $J = 2.2$  Hz, 2H), 5.79 (ddt,  $J = 17.3, 10.7, 5.5$  Hz, 1H), 5.21 – 5.16 (m, 1H), 5.15 – 5.11 (m, 1H), 4.76 – 4.50 (m, 2H), 3.21 (s, 3H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  172.8, 168.9, 147.8, 147.7, 144.3, 131.2, 129.7, 129.3, 126.9, 125.9, 122.9, 121.4, 118.5, 108.8, 108.7, 108.0, 101.3, 66.4, 63.5, 26.7.

**IR** (film)  $\nu_{\text{max}}$  3632, 2827, 2351, 2300, 1721, 1644, 1409, 1288, 1117, 1003, 900, 790, 601, 529  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{17}\text{NO}_5 + \text{Na}]^+$  374.0999; Found 374.0997.

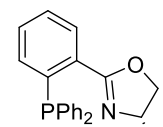
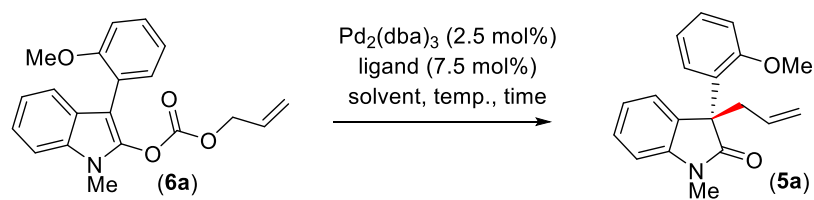
**General Synthetic procedure for compound (±)-**5a-r**:**



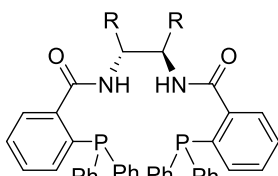
An oven-dried round-bottom flask was charged with carbonate **6a-s** (0.07 mmol; 1.0 equiv) in dry degassed THF (3 mL) at room temperature. Then 5 mol% of  $\text{Pd(PPh}_3)_4$  was added at same temperature and stirring was continued for 15 h. Upon completion of reaction (monitored by TLC), the reaction mixture was concentrated and purified by column chromatography using EtOAc and hexane mixture as eluent to afford the desired product (±)-**5a-r**.

**Optimization of catalytic enantioselective decarboxylative allylations:**

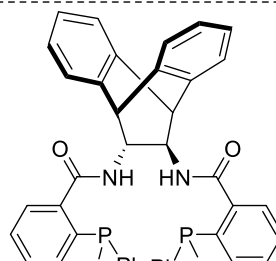




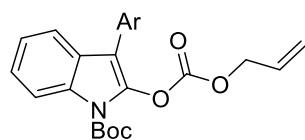
R = *i*Pr, (S)-**L1**  
 R = *i*Bu, (S)-**L2**  
 R = *s*Bu, (S)-**L3**  
 R = *t*Bu, (S)-**L4**



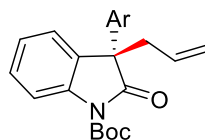
R = Ph, (R,R)-**L5**  
 R =  $-(\text{CH}_2)_4-$ , (R,R)-**L6**



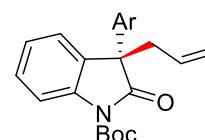
(R,R)-**L7**



Ar = *p*-OMePh; **(6b)**  
 Ar = *o*-OMePh; **(6c)**



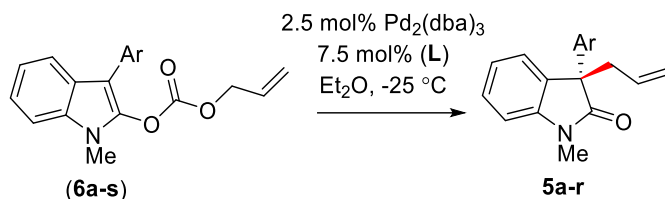
Ar = *p*-OMePh; (S)-**5b**  
 -25 °C, 21 h  
 87%, 35% ee



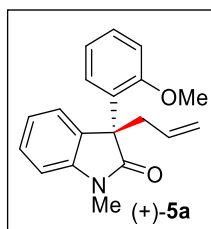
Ar = *o*-OMePh; (R)-**5c**  
 -25 °C, 19 h  
 91%, 42% ee

entry <sup>a</sup> .	Lig-and	solvent	temp.	time	% yield <sup>b</sup>	% ee <sup>c</sup>
1	<b>L1</b>	Et <sub>2</sub> O	25 °C	13 h	92%	-07
2	<b>L2</b>	Et <sub>2</sub> O	25 °C	12 h	89%	-08
3	<b>L3</b>	Et <sub>2</sub> O	25 °C	14 h	90%	-09
4	<b>L4</b>	Et <sub>2</sub> O	25 °C	14 h	88%	-15
5	<b>L5</b>	Et <sub>2</sub> O	25 °C	13 h	90%	35
6	<b>L6</b>	Et <sub>2</sub> O	25 °C	12 h	88%	39
7	<b>L7</b>	Et <sub>2</sub> O	25 °C	12 h	90%	79%
8	<b>L7</b>	CH <sub>2</sub> Cl <sub>2</sub>	25 °C	11 h	78%	42%
9	<b>L7</b>	(CH <sub>2</sub> Cl) <sub>2</sub>	25 °C	13 h	72%	40%
10	<b>L7</b>	CHCl <sub>3</sub>	25 °C	13 h	75%	46%
11	<b>L7</b>	Xylene	25 °C	14 h	85%	60%
12	<b>L7</b>	MTBE	25 °C	12 h	91%	78%
13	<b>L7</b>	THF	25 °C	10 h	89%	79%
14	<b>L7</b>	Et <sub>2</sub> O	25 °C	12 h	90%	81%
15	<b>L7<sup>d</sup></b>	Et <sub>2</sub> O	25 °C	18 h	88%	79%
16	<b>L7</b>	Et <sub>2</sub> O	0 °C to rt	12 h	91%	82%
17	<b>L7</b>	Et <sub>2</sub> O	0 °C	13 h	92%	84%
18	<b>L7</b>	Et <sub>2</sub> O	-10 °C	16 h	91%	85%
19	<b>L7</b>	Et <sub>2</sub> O	-25 °C	18 h	91%	90%
20	<b>L7</b>	Et <sub>2</sub> O	-40 °C	30 h	90%	85%
21	<b>L7</b>	PhMe	-10 °C	18 h	87%	82%
22	<b>L7</b>	PhMe	-25 °C	20 h	84%	87%
23	<b>L7</b>	PhMe	-40 °C	36 h	60%	82%

<sup>a</sup>reactions were carried out using 0.06 mmol of **6a-c** in 3.0 mL solvent under argon condition. <sup>b</sup>isolated yields. <sup>c</sup>ee's were determined by using chiral HPLC. <sup>d</sup>reaction was carried out using 1.25 mol% of Pd<sub>2</sub>(dba)<sub>3</sub>.

**General procedure for catalytic enantioselective decarboxylative allylations:**

In an oven-dried round-bottom flask, Et<sub>2</sub>O (1.5 mL) was degassed by using nitrogen balloon at room temperature over a period of 10 minutes. To this solution, 0.025 mol% of Pd<sub>2</sub>(dba)<sub>3</sub> and 0.075 mol% of ligand were added and stirring was continued for 20 minutes to make the metal-complex mixture. After that reaction mixture was cooled to the -25 °C. In another vessel, carbonate (**6**) (0.06 mmol; 1.0 equiv) was dissolved in dry degassed (1.5 mL) of Et<sub>2</sub>O. This solution was added drop-wise to metal-complex solution at -25 °C and stirring was continued for specified time. After complete consumption of starting material (monitored by TLC), the reaction mixture was concentrated and purified by column chromatography to afford the desired enantioenriched compound (*R/S*)-**5a-v**.



**(*R*)-3-Allyl-3-(2-methoxyphenyl)-1-methylindolin-2-one [(+)-**5a**]:** The compound (+)-**5a** was obtained as orange color gel (0.06 mmol scale of reaction; 16 mg of product; 91%).  $R_f = 0.35$  (30% EtOAc in hexane).

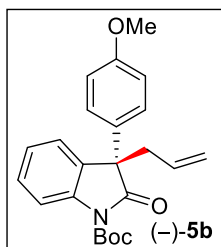
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.64 (d,  $J = 7.9$  Hz, 1H), 7.53 (dd,  $J = 7.5, 1.9$  Hz, 1H), 7.37 - 7.27 (m, 3H), 7.19 (td,  $J = 7.4, 6.8, 1.4$  Hz, 1H), 7.09 (td,  $J = 7.4, 1.3$  Hz, 1H), 7.04 (d,  $J = 8.3$  Hz, 1H), 5.95 (ddt,  $J = 16.5, 10.6, 5.8$  Hz, 1H), 5.41 (dt,  $J = 17.3, 1.5$  Hz, 1H), 5.34 (dd,  $J = 10.5, 1.5$  Hz, 1H), 4.76 (dd,  $J = 5.7, 1.4$  Hz, 2H), 3.80 (s, 3H), 3.70 (s, 3H).

**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  157.0, 152.3, 139.2, 132.8, 131.4, 130.8, 128.1, 125.4, 121.8, 121.6, 120.8, 120.2, 120.2, 119.8, 111.3, 109.2, 99.1, 69.8, 55.5, 28.7.

**IR** (film)  $\nu_{\text{max}}$  3042, 2946, 2845, 1713, 1620, 1449, 1312, 1290, 1230, 1220, 792, 583  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{19}\text{H}_{19}\text{NO}_2 + \text{Na}]^+$  316.1308; Found 316.1287.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IB column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_{\text{R}}$  major = 12.45 min.  $t_{\text{R}}$  minor = 17.38 min,  $[\alpha]_{\text{D}}^{25.0} = +60.5$  ( $c = 0.26$ ,  $\text{CH}_2\text{Cl}_2$  for 90% ee).



***tert*-Butyl (*S*)-3-allyl-3-(4-methoxyphenyl)-2-oxoindoline-1-carboxylate [(-)-5a]:** The compound (-)-5b was obtained as orange color gel (0.06 mmol scale of reaction; 20 mg of product; 87%).  $R_f = 0.40$  (10% EtOAc in hexane).

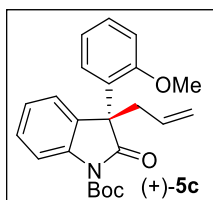
**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.31 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.24 - 7.22 (m, 1H), 7.09 (td,  $J = 7.5, 1.0$  Hz, 1H), 6.90 (d,  $J = 1.9$  Hz, 1H), 6.87 (d,  $J = 7.8$  Hz, 1H), 6.79 (dd,  $J = 8.2, 1.9$  Hz, 1H), 6.70 (d,  $J = 8.2$  Hz, 1H), 5.89 (q,  $J = 1.5$  Hz, 2H), 5.37 (ddt,  $J = 17.1, 10.0, 7.1$  Hz, 1H), 5.00 (dq,  $J = 17.0, 1.5$  Hz, 1H), 4.92 - 4.89 (m, 1H), 3.18 (s, 3H), 3.00 - 2.90 (m, 2H).

**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  178.0, 147.9, 146.8, 143.7, 133.3, 132.4, 131.7, 128.3, 125.1, 122.5, 120.4, 119.2, 108.2, 108.0, 107.9, 101.1, 56.0, 42.0.

**IR** (film)  $\nu_{\max}$  3442, 2937, 2301, 2211, 1721, 1623, 1449, 1358, 1267, 1033, 1040, 816, 701, 589  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$   $[M + H]^+$  Calcd for  $[\text{C}_{23}\text{H}_{26}\text{NO}_4]^+$  380.1856; Found 380.1844.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IC-3 column; solvent: hexane/2-propanol = 80/20; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  major = 4.99 min.  $t_R$  minor = 6.49 min,  $[\alpha]_D^{23.1} = -7.1$  ( $c = 0.31$ ,  $\text{CH}_2\text{Cl}_2$  for 35% ee).



***tert*-Butyl (*R*)-3-allyl-3-(2-methoxyphenyl)-2-oxoindoline-1-carboxylate [(+)-5c]**: The compound (+)-5c was obtained as orange color gel (0.06 mmol scale of reaction; 21 mg of product; 91%).  $R_f = 0.45$  (10% EtOAc in hexane).

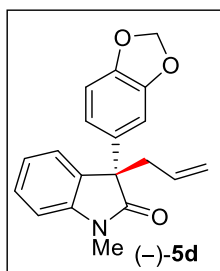
**$^1\text{H}$  NMR** (400 MHz  $\text{CDCl}_3$ )  $\delta$  7.81 (d,  $J = 8.1$  Hz, 1H), 7.55 (dd,  $J = 7.7, 1.6$  Hz, 1H), 7.23 (qd,  $J = 8.1, 1.5$  Hz, 2H), 7.02 (tdd,  $J = 7.5, 4.4, 1.1$  Hz, 2H), 6.83 (dd,  $J = 7.5, 1.4$  Hz, 1H), 6.75 (dd,  $J = 8.1, 1.2$  Hz, 1H), 5.40 (ddt,  $J = 17.2, 10.1, 7.1$  Hz, 1H), 5.04 - 4.99 (m, 1H), 4.95 (dd,  $J = 10.2, 1.9$  Hz, 1H), 3.43 (s, 3H), 3.04 - 2.97 (m, 2H), 1.65 (s, 9H).

**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  177.5, 156.8, 149.7, 140.2, 132.1, 131.0, 129.9, 128.9, 127.6, 127.1, 124.1, 122.6, 120.9, 119.9, 114.3, 112.3, 83.6, 55.7, 54.1, 41.5, 28.2.

**IR** (film)  $\nu_{\max}$  3332, 2857, 2321, 2191, 1720, 1643, 1429, 1308, 1247, 1103, 1000, 810, 721, 689  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[M + H]^+$  Calcd for  $[C_{23}H_{26}NO_4]^+$  380.1856; Found 380.1858.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 97/3; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 11.55 min,  $t_R$  major = 14.89 min.  $[\alpha]_D^{23.1} = +31.9$  ( $c = 0.12$ ,  $CH_2Cl_2$  for 42% ee).



**(S)-3-Allyl-3-(benzo[*d*][1,3]dioxol-5-yl)-1-methylindolin-2-one** **[(-)-5d]**: The compound **(-)-5d** was obtained as orange color gel (0.06 mmol scale of reaction; 16 mg of product; 89%).  $R_f = 0.30$  (30% EtOAc in hexane).

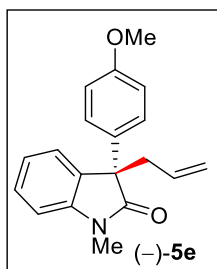
**$^1H$  NMR** (500 MHz,  $CDCl_3$ )  $\delta$  7.35 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.27 (dd,  $J = 7.5, 1.2$  Hz, 1H), 7.14 (td,  $J = 7.5, 1.0$  Hz, 1H), 6.94 (d,  $J = 2.0$  Hz, 1H), 6.93 - 6.89 (m, 1H), 6.83 (dd,  $J = 8.2, 1.9$  Hz, 1H), 6.74 (d,  $J = 8.2$  Hz, 1H), 5.94 - 5.92 (m, 2H), 5.41 (dddd,  $J = 16.9, 10.2, 7.6, 6.7$  Hz, 1H), 5.05 (dq,  $J = 17.1, 1.4$  Hz, 1H), 4.94 (ddt,  $J = 10.0, 1.8, 0.9$  Hz, 1H), 3.22 (s, 3H), 3.22 - 2.94 (m, 2H).

**$^{13}C$  NMR** (125 MHz,  $CDCl_3$ )  $\delta$  178.0, 147.9, 146.8, 143.7, 133.3, 132.4, 131.7, 128.3, 125.1, 122.5, 120.4, 119.2, 108.2, 108.0, 107.9, 101.1, 56.0, 42.1, 26.3.

**IR** (film)  $\nu_{max}$  3042, 2936, 2815, 1717, 1610, 1489, 1372, 1287, 1220, 1010, 742, 513  $cm^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[M + Na]^+$  Calcd for  $[C_{19}H_{17}NO_3 + Na]^+$  330.1101; Found 330.1087.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AS-3 column; solvent: hexane/2-propanol = 60/40; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 5.03 min,  $t_R$  major = 5.87 min.  $[\alpha]_D^{25.0} = -83.5$  ( $c = 0.26$ ,  $\text{CH}_2\text{Cl}_2$  for 90% ee).



**(*S*)-3-Allyl-3-(4-methoxyphenyl)-1-methylindolin-2-one [(-)-**5e**]:** The compound (-)-**5e** was obtained as orange color gel (0.06 mmol scale of reaction; 15 mg of product; 87%;  $R_f = 0.30$  (30% EtOAc in hexane)).

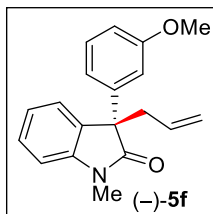
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 -7.24 (m, 4H), 7.10 (td,  $J = 7.5, 1.1$  Hz, 1H), 6.88 (d,  $J = 7.8$  Hz, 1H), 6.85 - 6.81 (m, 2H), 5.40 (ddt,  $J = 17.1, 10.1, 7.1$  Hz, 1H), 5.02 (dq,  $J = 17.0, 1.5$  Hz, 1H), 4.91 (dd,  $J = 10.2, 1.9$  Hz, 1H), 3.75 (s, 3H), 3.18 (s, 3H), 2.99 (d,  $J = 7.0$  Hz, 2H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  178.25, 158.82, 143.84, 132.56, 131.84, 131.55, 128.2 (two different chemical shift carbons), 125.15, 122.44, 119.08, 113.92, 108.22, 55.75, 55.25, 42.14, 26.33.

**IR** (film)  $\nu_{\text{max}}$  3042, 2936, 2815, 1717, 1610, 1489, 1372, 1287, 1220, 1010, 742, 513  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{19}\text{H}_{19}\text{NO}_2 + \text{Na}]^+$  316.1308; Found 316.1304.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AS-3 column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  major = 5.80 min, minor  $t_R$  = 7.93 min.  $[\alpha]_D^{24.5} = -75.9$  ( $c = 0.18$ ,  $\text{CH}_2\text{Cl}_2$  for 90% ee).



**(S)-3-Allyl-3-(3-methoxyphenyl)-1-methylindolin-2-one [(-)-5f]:** The compound (-)-5f was obtained as orange color gel (0.06 mmol scale of reaction; 18 mg of product; 96%).  $R_f = 0.40$  (50% EtOAc in hexane).

**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (td,  $J = 7.7, 1.2$  Hz, 1H), 7.30 - 7.23 (m, 1H), 7.25 (t,  $J = 8.0$  Hz, 1H), 7.13 (td,  $J = 7.6, 1.1$  Hz, 1H), 7.01 - 6.98 (m, 2H), 6.91 (dt,  $J = 7.8, 0.8$  Hz, 1H), 6.82 (ddd,  $J = 8.2, 2.6, 0.9$  Hz, 1H), 5.46 - 5.38 (m, 1H), 5.06 (dq,  $J = 17.1, 1.5$  Hz, 1H), 4.95 (ddt,  $J = 10.1, 1.9, 1.0$  Hz, 1H), 3.79 (s, 3H), 3.23 (s, 3H), 3.05 - 3.03 (m, 2H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  177.8, 159.6, 143.8, 141.1, 132.4, 131.6, 129.4, 128.2, 125.2, 122.3, 119.5, 119.1, 113.5, 112.2, 108.2, 56.3, 55.2, 41.9, 26.4.

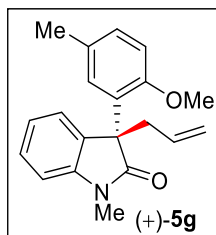
**IR** (film)  $\nu_{\text{max}}$  3022, 2926, 2901, 1720, 1610, 1450, 1311, 1217, 1140, 1020, 722, 563  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{19}\text{H}_{20}\text{NO}_2]^+$  294.1489; Found 294.1492.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min;



detection: at 254 nm):  $t_R$  minor = 4.57 min,  $t_R$  major = 5.33 min.  $[\alpha]_D^{24.5} = -71.5$  ( $c = 0.16$ ,  $\text{CH}_2\text{Cl}_2$  for 84% ee).



**(R)-3-Allyl-3-(2-methoxy-5-methylphenyl)-1-methylindolin-2-one** [(+)-5g]: The compound (+)-5g was obtained as orange color gel (0.06 mmol scale of reaction; 18 mg of product; 96%).  $R_f = 0.47$  (30% EtOAc in hexane).

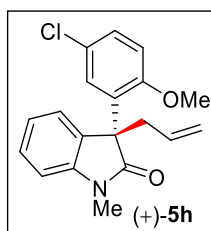
**$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 (dt,  $J = 8.0, 1.0$  Hz, 1H), 7.48 (d,  $J = 2.2$  Hz, 1H), 7.36 - 7.28 (m, 3H), 7.18 (ddd,  $J = 8.1, 7.0, 1.2$  Hz, 1H), 7.02 (d,  $J = 8.4$  Hz, 1H), 5.97 (ddt,  $J = 17.3, 10.6, 5.8$  Hz, 1H), 5.43 (dt,  $J = 17.2, 1.4$  Hz, 1H), 5.35 (dt,  $J = 10.5, 1.2$  Hz, 1H), 4.77 (dt,  $J = 5.8, 1.4$  Hz, 2H), 4.48 (s, 2H), 3.81 (s, 3H), 3.71 (s, 3H), 3.42 (s, 3H).

**$^{13}\text{C}$  NMR** (125 MHz,  $\text{CDCl}_3$ )  $\delta$  156.6, 152.2, 139.2, 132.8, 131.2, 130.7, 130.3, 127.8, 125.3, 121.8, 121.4, 120.2, 120.1, 119.8, 111.3, 109.1, 98.9, 74.4, 69.8, 57.7, 55.7, 28.7.

**IR** (film)  $\nu_{\text{max}}$  3052, 2926, 2855, 1719, 1611, 1499, 1347, 1257, 1140, 1020, 752, 543  $\text{cm}^{-1}$ .

**HRMS** ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{22}\text{NO}_2]^+$  308.1645; Found 308.1651.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 4.20 min,  $t_R$  major = 8.19 min.  $[\alpha]_D^{24.0} = +85.5$  ( $c = 0.19$ ,  $\text{CH}_2\text{Cl}_2$  for 91% ee).



**(R)-3-Allyl-3-(5-chloro-2-methoxyphenyl)-1-methylindolin-2-one [(+)-5h]:** The compound (+)-**5h** was obtained as orange color gel (0.06 mmol scale of reaction; 18 mg of product; 94%).  $R_f = 0.23$  (20% EtOAc in hexane).

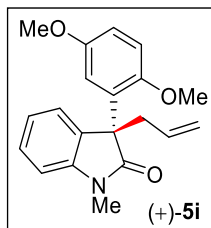
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (d,  $J = 2.6$  Hz, 1H), 7.24 - 7.18 (m, 2H), 6.95 (td,  $J = 7.5, 1.0$  Hz, 1H), 6.86 (dd,  $J = 7.4, 1.3$  Hz, 1H), 6.81 (d,  $J = 7.7$  Hz, 1H), 6.66 (d,  $J = 8.7$  Hz, 1H), 5.30 (ddt,  $J = 17.1, 10.1, 7.1$  Hz, 1H), 5.02 - 4.97 (m, 1H), 4.89 (dd,  $J = 10.2, 2.0$  Hz, 1H), 3.38 (s, 3H), 3.24 (s, 3H), 2.95 - 2.93 (m, 2H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  178.5, 155.8, 144.4, 132.5, 131.4, 131.1, 128.3, 127.9, 127.7, 125.9, 122.5, 122.3, 119.4, 113.4, 107.2, 56.2, 53.6, 40.5, 26.2.

**IR** (film)  $\nu_{\text{max}}$  3420, 2898, 2827, 2114, 1721, 1642, 1404, 1367, 1320, 1222, 1107, 1092, 1009, 956, 788, 690, 640  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{19}\text{H}_{19}\text{ClNO}_2]^+$  328.1099; Found 328.1108.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 4.49 min,  $t_R$  major = 9.19 min.  $[\alpha]_{\text{D}}^{23.7} = +98.5$  ( $c = 0.13$ ,  $\text{CH}_2\text{Cl}_2$  for 92% ee).



**(R)-3-Allyl-3-(2,5-dimethoxyphenyl)-1-methylindolin-2-one [(+)-5i]:** The compound (+)-**5i** was obtained as orange color gel (0.06 mmol scale of reaction; 18 mg of product; 92%).  $R_f = 0.20$  (30% EtOAc in hexane).

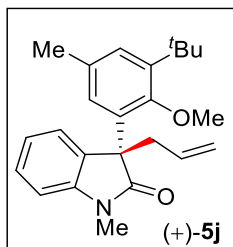
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21 (td,  $J = 7.6, 1.5$  Hz, 1H), 7.16 (d,  $J = 2.9$  Hz, 1H), 6.94 - 6.86 (m, 2H), 6.80 (d,  $J = 7.7$  Hz, 1H), 6.75 (dd,  $J = 8.8, 2.9$  Hz, 1H), 6.68 (d,  $J = 8.8$  Hz, 1H), 5.35 (ddt,  $J = 17.2, 10.1, 7.1$  Hz, 1H), 4.99 (dq,  $J = 17.0, 1.5$  Hz, 1H), 4.90 - 4.87 (m, 1H), 3.80 (s, 3H), 3.32 (s, 3H), 3.25 (s, 3H), 2.98 - 2.92 (m, 2H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  179.1, 153.9, 151.5, 144.4, 133.1, 131.6, 131.2, 127.5, 122.6, 122.1, 119.1, 114.9, 113.7, 112.2, 107.1, 56.8, 55.8, 53.7, 40.5, 26.1.

**IR** (film)  $\nu_{\text{max}}$  3440, 2998, 2797, 2214, 1720, 1621, 1432, 1325, 1320, 1210, 1137, 1052, 1019, 920, 798, 670, 510  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{22}\text{NO}_3]^+$  324.1594; Found 324.1592.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 5.58 min,  $t_R$  major = 14.91 min.  $[\alpha]_D^{24.0} = +73.5$  ( $c = 0.22$ ,  $\text{CH}_2\text{Cl}_2$  for 90% ee).



**(R)-3-Allyl-3-(3-(*tert*-butyl)-2-methoxy-5-methylphenyl)-1-methylindolin-2-one [(+)-5j]**: The compound (+)-5j was obtained as orange color solid (0.06 mmol scale of reaction; 20 mg of product; 92%). mp = 110-112 °C.

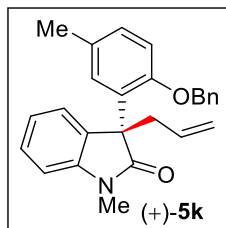
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.32 (d, *J*=2.2 Hz, 1H), 7.26 (td, *J*=7.6, 1.4 Hz, 1H), 7.18 - 7.17 (m, 1H), 7.04 (dd, *J*=7.3, 1.4 Hz, 1H), 6.99 (td, *J*=7.4, 1.0 Hz, 1H), 6.85 (d, *J*=7.7 Hz, 1H), 5.38 - 5.30 (m, 1H), 4.97 (dq, *J*=17.0, 1.4 Hz, 1H), 4.90 (ddt, *J*=10.2, 2.0, 0.9 Hz, 1H), 3.31 (s, 3H), 3.02 - 2.98 (m, 1H), 2.94 - 2.89 (m, 1H), 2.80 (s, 3H), 2.39 (s, 3H), 1.34 (s, 9H).

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>) δ 178.8, 155.4, 144.3, 142.9, 135.0, 133.7, 132.4, 131.8, 129.2, 127.7, 126.9, 123.2, 122.1, 119.0, 107.3, 62.6, 53.9, 42.7, 35.4, 32.0, 26.1, 21.4.

**IR** (film)  $\nu_{\max}$  3337, 2920, 2106, 1717, 1638, 1405, 1480, 1308, 1224, 1164, 1094, 1001, 931, 818, 748, 707, 603 cm<sup>-1</sup>.

**HRMS** (ESI-TOF) *m/z*: [M + H]<sup>+</sup> Calcd for [C<sub>24</sub>H<sub>30</sub>NO<sub>2</sub>]<sup>+</sup> 364.2271; Found 364.2263.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak ID-3 column; solvent: hexane/2-propanol = 80/20; flow rate: 1.0 mL/min; detection: at 254 nm): *t<sub>R</sub>* major = 10.52 min. *t<sub>R</sub>* minor = 11.55 min, [α]<sub>D</sub><sup>26.6</sup> = +26.0 (*c* = 0.05, CH<sub>2</sub>Cl<sub>2</sub> for 64% ee).



**(R)-3-Allyl-3-(2-(benzyloxy)-5-methylphenyl)-1-methylindolin-2-one [(+)-5k]:** The compound (+)-5k was obtained as orange color gel (0.06 mmol scale of reaction; 21 mg of product; 90%).  $R_f = 0.45$  (20% EtOAc in hexane).

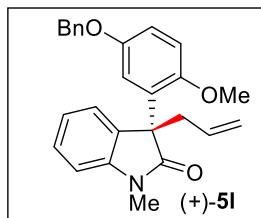
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.44 (d,  $J = 2.3$  Hz, 1H), 7.31 - 7.26 (m, 3H), 7.21 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.07 - 7.05 (m, 1H), 6.99 (td,  $J = 7.4, 1.0$  Hz, 1H), 6.91 - 6.88 (m, 3H), 6.74 (d,  $J = 8.2$  Hz, 1H), 6.48 (d,  $J = 7.7$  Hz, 1H), 5.31 (ddt,  $J = 17.2, 10.1, 7.1$  Hz, 1H), 4.98 (dq,  $J = 17.0, 1.4$  Hz, 1H), 4.88 - 4.85 (m, 1H), 4.69 (d,  $J = 10.6$  Hz, 1H), 4.56 (d,  $J = 10.6$  Hz, 1H), 3.03 - 2.96 (m, 2H), 2.60 (s, 3H), 2.40 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  178.9, 154.0, 144.3, 136.2, 133.2, 131.8, 129.7, 128.8, 128.6, 128.6, 128.5, 128.2, 127.8, 127.0, 122.3, 121.8, 118.9, 111.7, 107.6, 70.2, 53.6, 40.8, 25.2, 20.9.

**IR** (film)  $\nu_{\text{max}}$  3430, 2928, 2857, 2104, 1718, 1612, 1494, 1467, 1376, 1347, 1252, 1137, 1091, 1019, 916, 748, 696, 649  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{26}\text{H}_{26}\text{NO}_2]^+$  384.1958; Found 384.1984.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 60/40; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_{\text{R}}$  minor = 8.62 min,  $t_{\text{R}}$  major = 11.87 min.  $[\alpha]_{\text{D}}^{22.0} = +43.5$  ( $c = 0.13$ ,  $\text{CH}_2\text{Cl}_2$  for 85% ee).



**(R)-3-Allyl-3-(5-(benzyloxy)-2-methoxyphenyl)-1-methylindolin-2-one [(+)-51]:** The compound (+)-**51** was obtained as orange color gel (0.06 mmol scale of reaction; 22 mg of product; 92%).  $R_f = 0.30$  (30% EtOAc in hexane).

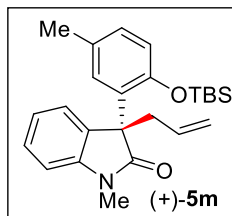
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 - 7.31 (m, 5H), 7.24 - 7.19 (m, 2H), 6.92 (td,  $J = 7.5$ , 1.0 Hz, 1H), 6.88 - 6.79 (m, 3H), 6.68 (d,  $J = 8.8$  Hz, 1H), 5.33 (ddt,  $J = 17.2$ , 10.1, 7.1 Hz, 1H), 5.04 (s, 2H), 5.01 - 4.96 (m, 1H), 4.88 (dd,  $J = 10.2$ , 1.9 Hz, 1H), 3.33 (s, 3H), 3.25 (s, 3H), 2.99 - 2.90 (m, 2H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  179.1, 153.1, 151.67, 144.4, 137.2, 133.1, 131.6, 131.1, 128.6, 128.0, 127.6, 127.5, 122.6, 122.1, 119.1, 116.1, 113.6, 113.4, 107.1, 70.8, 56.7, 53.7, 40.5, 26.2.

**IR** (film)  $\nu_{\text{max}}$  3434, 2100, 1713, 1640, 1494, 1470, 1375, 1348, 1283, 1225, 1070, 1021, 967, 916, 736, 697, 515  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{26}\text{H}_{25}\text{NO}_3 + \text{Na}]^+$  422.1727; Found 422.1724.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 60/40; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 5.47 min,  $t_R$  major = 14.07 min.  $[\alpha]_D^{21.8} = +85.7$  ( $c = 0.21$ ,  $\text{CH}_2\text{Cl}_2$  for 93% ee).



**(R)-3-Allyl-3-(2-((*tert*-butyldimethylsilyloxy)-5-methylphenyl)-1-methylindolin-2-one [(+)-5m]:** The compound (+)-5m was obtained as orange color gel (0.06 mmol scale of reaction; 24 mg of product; 96%).

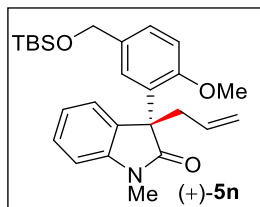
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  = 7.34 (d,  $J$  = 2.3 Hz, 1H), 7.17 (td,  $J$  = 7.5, 1.7 Hz, 1H), 6.93 - 6.86 (m, 3H), 6.75 (d,  $J$  = 7.7 Hz, 1H), 6.62 (d,  $J$  = 8.2 Hz, 1H), 5.24 (ddt,  $J$  = 17.2, 10.1, 7.1 Hz, 1H), 4.95 - 4.90 (m, 1H), 4.85 (dd,  $J$  = 10.1, 2.0 Hz, 1H), 3.19 (s, 3H), 2.92 (d,  $J$  = 7.1 Hz, 2H), 2.32 (s, 3H), 0.67 (s, 9H), -0.02 (s, 3H), -0.07 (s, 2H).

**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  = 178.4, 151.4, 144.1, 133.5, 131.9, 129.2 (2C), 128.9, 128.5, 127.2, 122.5, 122.1, 118.8, 117.6, 107.3, 54.2, 42.1, 26.3, 20.9, 19.1, -3.6, -3.6.

**IR** (film)  $\nu_{\text{max}}$  3437, 2960, 2116, 1717, 1628, 1495, 1422, 1318, 1204, 1104, 1084, 1001, 996, 802, 772, 617, 603  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{25}\text{H}_{33}\text{NO}_2\text{Si} + \text{Na}]^+$  430.2173; Found 430.2160.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IC-3 column; solvent: hexane/2-propanol = 75/25; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_{\text{R}}$  minor = 6.81 min,  $t_{\text{R}}$  major = 7.86 min.  $[\alpha]_{\text{D}}^{23.7} = +13.0$  ( $c = 0.35$ ,  $\text{CH}_2\text{Cl}_2$  for 82% ee).



**(*R*)-3-Allyl-3-(5-(((*tert*-butyldimethylsilyl)oxy)methyl)-2-methoxyphenyl)-1-**

**methylinindolin-2-one [(+)-5n]:** The compound (+)-5n was obtained as orange color gel (0.06 mmol scale of reaction; 24 mg of product; 92%).  $R_f = 0.40$  (10% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (d,  $J = 2.2$  Hz, 1H), 7.25 - 7.16 (m, 2H), 6.91 (t,  $J = 7.4$  Hz, 1H), 6.85 (dd,  $J = 7.4, 1.3$  Hz, 1H), 6.80 (d,  $J = 7.7$  Hz, 1H), 6.71 (d,  $J = 8.3$  Hz, 1H), 5.34 (ddt,  $J = 17.1, 10.0, 7.0$  Hz, 1H), 4.98 (dd,  $J = 17.0, 1.9$  Hz, 1H), 4.88 (dd,  $J = 10.1, 2.0$  Hz, 1H), 4.73 (d,  $J = 1.7$  Hz, 2H), 3.38 (s, 3H), 3.25 (s, 3H), 2.99 (d,  $J = 7.1$  Hz, 2H), 0.95 (s, 9H), 0.11 (d,  $J = 2.2$  Hz, 6H).

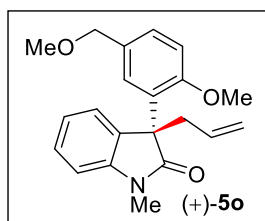
**$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  179.3, 156.2, 144.4, 133.7, 133.3, 131.7, 129.4, 127.4, 126.4, 125.7, 122.6, 122.1, 119.0, 112.2, 107.0, 64.9, 56.1, 53.8, 40.5, 26.1, 26.0, 18.4, -5.1.

**IR** (film)  $\nu_{\text{max}}$  3152, 2916, 2825, 1730, 1601, 1429, 1387, 1277, 1110, 1090, 782, 563  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{26}\text{H}_{35}\text{NO}_3\text{Si} + \text{Na}]^+$  460.2278; Found 460.2279.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IC-3 column; solvent: hexane/2-propanol = 85/15; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  major = 5.78 min.  $t_R$  minor = 7.36 min,  $[\alpha]_D^{25.0} = +57.5$  ( $c = 0.39$ ,  $\text{CH}_2\text{Cl}_2$  for 93% ee).





**(*R*)-3-Allyl-3-(2-methoxy-5-(methoxymethyl)phenyl)-1-methylindolin-2-one [(+)-5o]:**

The compound (+)-5o was obtained as orange color gel (0.06 mmol scale of reaction; 19 mg of product; 93%).  $R_f = 0.51$  (30% EtOAc in hexane).

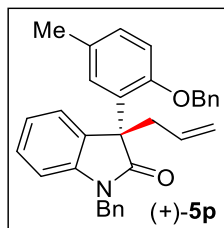
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 (d,  $J = 2.1$  Hz, 1H), 7.29 - 7.22 (m, 2H), 6.95 (td,  $J = 7.4, 1.0$  Hz, 1H), 6.90 (dd,  $J = 7.4, 1.3$  Hz, 1H), 6.84 (dd,  $J = 7.8, 0.8$  Hz, 1H), 6.76 (d,  $J = 8.3$  Hz, 1H), 5.36 (ddt,  $J = 17.1, 10.1, 7.1$ , 1H), 5.02 (dq,  $J = 17.0, 1.4$  Hz, 1H), 4.93 - 4.90 (m, 1H), 4.51 - 4.45 (m, 2H), 3.43 (d,  $J = 5.2$  Hz, 6H), 3.29 (s, 3H), 3.05 - 3.04 (m, 2H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  179.2, 156.7, 144.4, 133.1, 131.6, 130.4, 129.7, 128.3, 127.4, 127.4, 122.6, 122.1, 119.1, 112.1, 107.0, 74.6, 58.0, 56.0, 53.7, 40.6, 26.1.

**IR** (film)  $\nu_{\text{max}}$  3432, 2927, 2401, 2111, 1711, 1643, 1469, 1348, 1257, 1093, 1020, 916, 751, 602  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{21}\text{H}_{24}\text{NO}_3]^+$  338.1751; Found 338.1777.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 4.17 min,  $t_R$  major = 6.60 min.  $[\alpha]_D^{22.1} = +101.5$  ( $c = 0.31$ ,  $\text{CH}_2\text{Cl}_2$  for 94% ee).



**(R)-3-Allyl-1-benzyl-3-(2-(benzyloxy)-5-methylphenyl)indolin-2-one [(+)-5p]:** The compound (+)-**5p** was obtained as orange color gel (0.06 mmol scale of reaction; 27 mg of product; 93%).  $R_f = 0.30$  (10% EtOAc in hexane).

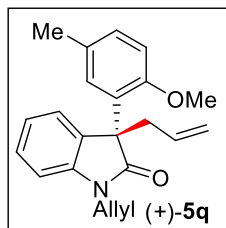
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (d,  $J = 2.2$  Hz, 1H), 7.39 - 7.31 (m, 3H), 7.25 - 7.20 (m, 4H), 7.13 - 7.05 (m, 2H), 6.97 - 6.91 (m, 4H), 6.73 (d,  $J = 8.3$  Hz, 1H), 6.41 (d,  $J = 7.8$  Hz, 1H), 5.39 (ddt,  $J = 17.1, 10.2, 7.0$  Hz, 1H), 5.09 - 5.06 (m, 1H), 4.93 - 4.90 (m, 2H), 4.65 (d,  $J = 11.0$  Hz, 1H), 4.57 (d,  $J = 11.0$  Hz, 1H), 3.37 (d,  $J = 15.8$  Hz, 1H), 3.12 - 3.04 (m, 2H), 2.41 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  179.0, 152.0, 143.8, 136.7, 136.4, 133.3, 132.1, 129.7, 128.8, 128.8, 128.6, 128.5, 128.4, 128.3, 127.9, 127.6, 127.1, 126.9, 122.5, 121.9, 119.3, 111.8, 108.8, 70.0, 53.5, 43.4, 41.0, 20.9.

**IR** (film)  $\nu_{\text{max}}$  3430, 2109, 1715, 1641, 1488, 1348, 1247, 1174, 1015, 917, 805, 746, 697  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{32}\text{H}_{30}\text{NO}_2]^+$  460.2271; Found 460.2288.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 60/40; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 8.62 min,  $t_R$  major = 11.87 min.  $[\alpha]_D^{22.1} = +111.9$  ( $c = 0.34$ ,  $\text{CH}_2\text{Cl}_2$  for 85% ee).



**(R)-1,3-Diallyl-3-(2-methoxy-5-methylphenyl)indolin-2-one [(+)-5q]:** The compound (+)-5q was obtained as orange color gel (0.06 mmol scale of reaction; 18 mg of product; 90%).  $R_f = 0.40$  (20% EtOAc in hexane).

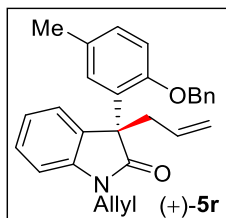
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 (d,  $J = 2.1$  Hz, 1H), 7.21 (td,  $J = 7.5, 1.7$  Hz, 1H), 7.07 (dd,  $J = 8.2, 2.1$  Hz, 1H), 6.98 – 6.91 (m, 2H), 6.86 (d,  $J = 7.7$  Hz, 1H), 6.70 (d,  $J = 8.2$  Hz, 1H), 6.01 – 5.83 (m, 1H), 5.44 – 5.36 (m, 2H), 5.29 (dq,  $J = 10.1, 1.5$  Hz, 1H), 5.07 – 5.02 (m, 1H), 4.96 – 4.92 (m, 1H), 4.50 (ddt,  $J = 16.0, 5.5, 1.7$  Hz, 1H), 4.36 (ddt,  $J = 16.1, 5.7, 1.7$  Hz, 1H), 3.39 (s, 3H), 3.04 (d,  $J = 7.2$  Hz, 2H), 2.39 (s, 9H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  178.8, 155.1, 143.6, 133.4, 132.5, 131.8, 130.0, 129.3, 128.9, 128.3, 127.2, 122.6, 122.0, 119.2, 117.6, 112.5, 108.0, 55.9, 53.6, 42.7, 40.8, 20.9.

**IR** (film)  $\nu_{\text{max}}$  3310, 2219, 1719, 1602, 1448, 1322, 1208, 1107, 1021, 940, 850, 716, 607  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{22}\text{H}_{24}\text{NO}_2]^+$  334.1802; Found 334.1810.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 4.45 min,  $t_R$  major = 9.93 min.  $[\alpha]_D^{24.3} = +69.0$  ( $c = 0.39$ ,  $\text{CH}_2\text{Cl}_2$  for 90% ee).



**(R)-1,3-Diallyl-3-(2-(benzyloxy)-5-methylphenyl)indolin-2-one** [(+)-5r]: The compound (+)-5r was obtained as orange color gel (0.06 mmol scale of reaction; 22 mg of product; 87%).  $R_f = 0.50$  (20% EtOAc in hexane).

**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta = 7.44$  (d,  $J = 2.1$  Hz, 1H), 7.30 - 7.29 (m, 1H), 7.28 - 7.25 (m, 2H), 7.18 (td,  $J = 7.6, 1.4$  Hz, 1H), 7.04 (ddd,  $J = 8.1, 2.2, 0.8$  Hz, 1H), 6.97 (td,  $J = 7.5, 1.0$  Hz, 1H), 6.91 (ddd,  $J = 7.4, 1.4, 0.6$  Hz, 1H), 6.87 - 6.85 (m, 2H), 6.71 (d,  $J = 8.2$  Hz, 1H), 6.55 (dt,  $J = 7.8, 0.8$  Hz, 1H), 5.60 (dddd,  $J = 17.3, 10.3, 6.3, 4.8$  Hz, 1H), 5.40 - 5.32 (m, 1H), 5.16 (dq,  $J = 17.2, 1.6$  Hz, 1H), 5.08 (dq,  $J = 10.3, 1.5$  Hz, 1H), 5.00 - 4.99 (m, 1H), 4.90 (ddd,  $J = 10.0, 1.9, 0.9$  Hz, 1H), 4.70 (d,  $J = 11.1$  Hz, 1H), 4.58 (d,  $J = 11.1$  Hz, 1H), 4.29 (ddt,  $J = 16.2, 4.8, 1.9$  Hz, 1H), 3.13 (ddt,  $J = 16.3, 6.3, 1.5$  Hz, 1H), 3.04 - 3.01 (m, 2H), 2.39 (s, 3H).

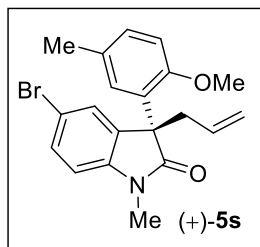
**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta = 178.5, 153.9, 143.7, 136.3, 133.3, 132.5, 131.9, 129.7, 128.8, 128.6, 128.6, 128.3, 128.3, 127.8, 126.9, 122.4, 121.8, 119.2, 117.1, 111.8, 108.6, 70.0, 53.5, 42.0, 41.0, 20.9$ .

**IR** (film)  $\nu_{\text{max}}$  3420, 2119, 1721, 1622, 1418, 1352, 1278, 1137, 1091, 949, 800, 706, 617  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{28}\text{H}_{27}\text{NO}_2 + \text{Na}]^+$  432.1934; Found 432.1943.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min;

detection: at 254 nm):  $t_R$  minor = 7.78 min,  $t_R$  major = 22.76 min.  $[\alpha]_D^{23.3} = +66.0$  ( $c = 0.05$ ,  $\text{CH}_2\text{Cl}_2$  for 84% ee).



**(R)-3-Allyl-5-bromo-3-(2-methoxy-5-methylphenyl)-1-methylindolin-2-one [(+)-5s]:**

The compound (+)-**5s** was obtained as colorless gel (0.05 mmol scale of reaction; 15 mg of product; 81% yield).  $R_f = 0.40$  (30% EtOAc in hexane).

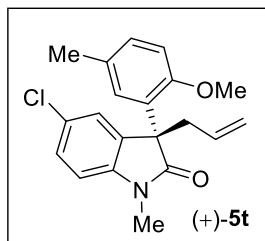
$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta = 7.38$  (d,  $J = 2.1$ , 1H), 7.24 (td,  $J = 7.6$ , 1.4, 1H), 7.08 – 7.04 (m, 1H), 6.99 – 6.89 (m, 1H), 6.84 (d,  $J = 7.8$ , 1H), 6.69 (d,  $J = 8.2$ , 1H), 5.36 (ddt,  $J = 17.2$ , 10.1, 7.1, 1H), 5.08 – 4.96 (m, 1H), 4.91 (ddt,  $J = 10.2$ , 2.0, 1.0, 1H), 3.39 (s, 3H), 3.29 (s, 3H), 3.02 (dt,  $J = 7.2$ , 1.2, 2H), 2.38 (s, 3H).

$^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta = 179.4$ , 155.1, 144.4, 133.4, 131.7, 130.0, 129.4, 128.9, 128.3, 127.4, 122.6, 122.1, 119.0, 112.5, 107.0, 56.2, 53.8, 40.5, 26.2, 20.9.

**IR** (film) 2803, 2729, 1690, 1611, 1236, 1102, 999, 768, 519  $\text{cm}^{-1}$ .

**HRMS** (ESI)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{20}\text{BrNO}_2 + \text{H}]^+$  386.0750; Found 386.0741.

Enantiomeric excess of pure compound was determined via HPLC analysis using a Chiralpak IB column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 273 nm:  $t_R$  minor = 9.03 min,  $t_R$  major = 6.30 min.  $[\alpha]_D^{22.6} = +60.0$  ( $c = 0.1$ ,  $\text{CHCl}_3$  for 92% ee).



**(R)-3-Allyl-5-chloro-3-(2-methoxy-5-methylphenyl)-1-methylindolin-2-one [(+)-5t]:**

The compound (+)-5t was obtained as colorless gel (0.052 mmol scale of reaction; 16 mg of product; 91%).  $R_f = 0.45$  (30% EtOAc in hexane).

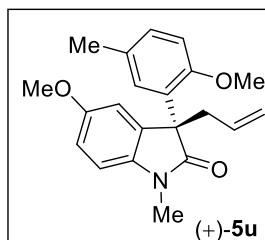
**$^1\text{H NMR}$**  (500 MHz,  $\text{CDCl}_3$ )  $\delta = 7.38 - 7.33$  (m, 1H), 7.21 (dd,  $J = 8.2, 2.1$ , 1H), 7.08 (ddd,  $J = 8.3, 2.2, 0.9$ , 1H), 6.95 (dd,  $J = 64.0, 2.0$ , 1H), 6.78 – 6.67 (m, 2H), 5.35 (ddt,  $J = 15.6, 10.1, 7.3$ , 1H), 5.04 (ddd,  $J = 17.1, 2.0, 1.1$ , 1H), 4.94 (ddt,  $J = 10.1, 1.9, 0.9$ , 1H), 3.42 (s, 3H), 3.27 (d,  $J = 3.0$ , 3H), 3.09 – 2.93 (m, 2H), 2.38 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta = 178.9, 154.9, 143.0, 135.1, 131.2, 130.2, 129.2, 128.5, 128.2, 127.4, 127.3, 123.1, 119.5, 112.2, 107.9, 56.0, 53.9, 40.4, 26.3, 20.9$ .

**IR** (film) 2799, 2614, 1601, 1526, 1165, 1098, 973, 842, 416  $\text{cm}^{-1}$ .

**HRMS** (ESI)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{20}\text{H}_{20}\text{ClNO}_2 + \text{H}]^+$  342.1255; Found 342.1252.

Enantiomeric excess of pure compound was determined via HPLC analysis using a Chiralpak IB column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 8.63 min,  $t_R$  major = 6.38 min.  $[\alpha]_D^{22.0} = +174.1$  ( $c = 0.1$ ,  $\text{CHCl}_3$  for 92% ee).



**(R)-3-Allyl-5-methoxy-3-(2-methoxy-5-methylphenyl)-1-methylindolin-2-one [(+)-5u]:** The compound (+)-**5u** was obtained as yellowish gel (0.04 mmol scale of reaction; 10 mg of product; 76% yield).  $R_f = 0.25$  (30% EtOAc in hexane).

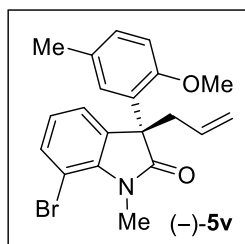
**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 – 7.31 (m, 1H), 7.09 – 6.98 (m, 1H), 6.80 – 6.71 (m, 2H), 6.68 (d,  $J = 8.2$ , 1H), 6.54 (d,  $J = 2.3$ , 1H), 5.36 (ddt,  $J = 17.1, 10.1, 7.1$ , 1H), 5.03 (dd,  $J = 16.7, 2.1$ , 1H), 4.96 – 4.87 (m, 1H), 3.73 (s, 3H), 3.42 (s, 3H), 3.26 (s, 3H), 3.00 (d,  $J = 7.1$ , 2H), 2.37 (s, 3H).

**$^{13}\text{C NMR}$**  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  179.0, 155.7, 155.1, 138.1, 134.8, 131.8, 130.0, 129.3, 128.9, 128.3, 119.0, 112.5, 111.4, 110.3, 107.2, 56.2, 55.7, 54.2, 40.6, 26.3, 20.9.

**IR** (film) 2402, 2326, 1546, 1469, 1191, 1126, 984, 823, 559  $\text{cm}^{-1}$ .

**HRMS** (ESI)  $m/z$   $[\text{M} + \text{H}]^+$  Calcd for  $[\text{C}_{21}\text{H}_{23}\text{NO}_3 + \text{H}]^+$  338.1751; Found 338.1734.

Enantiomeric excess of pure compound was determined via HPLC analysis using a Chiralpak IB column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 10.62 min,  $t_R$  major = 7.59 min.  $[\alpha]_D^{22.1} = +162.4$  ( $c = 0.1$ ,  $\text{CHCl}_3$  for 84% ee).



**(R)-3-Allyl-7-bromo-3-(2-methoxy-5-methylphenyl)-1-methylindolin-2-one [(-)-5v]:** The compound (-)-**5v** was obtained as yellow liquid (0.023 mmol scale of reaction; 7 mg of product; 83% yield).  $R_f = 0.20$  (30% EtOAc in hexane).

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.32 (m, 2H), 7.07 (dd, *J* = 8.2, 2.1, 1H), 6.84 – 6.75 (m, 2H), 6.69 (d, *J* = 8.2, 1H), 5.59 – 5.28 (m, 1H), 5.13 – 4.86 (m, 2H), 3.66 (s, 3H), 3.45 (s, 3H), 3.08 – 2.90 (m, 2H), 2.38 (s, 3H).

**<sup>13</sup>C NMR** (125 MHz, CDCl<sub>3</sub>) δ 179.5, 155.1, 144.4, 131.7, 130.0, 129.4, 128.9, 128.3, 127.4, 122.6, 122.1, 119.0, 112.5, 107.0, 100.0, 56.2, 53.8, 53.4, 40.5, 26.2.

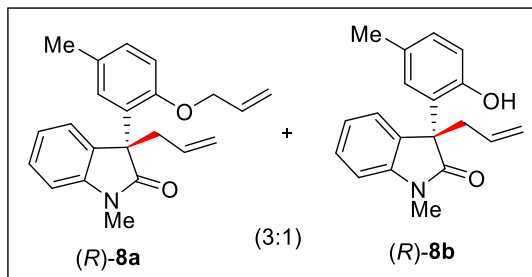
**IR** (film) 2699, 2601, 1524, 1496, 1191, 1063, 911, 519 cm<sup>-1</sup>.

**HRMS** (ESI) *m/z* [M + H]<sup>+</sup> Calcd for [C<sub>20</sub>H<sub>20</sub>BrNO<sub>2</sub> + H]<sup>+</sup> 386.0750; Found 386.0739.

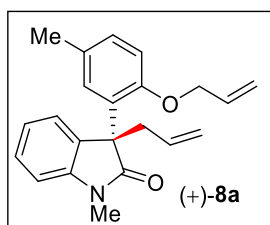
Enantiomeric excess of pure compound was determined via HPLC analysis using a Chiralpak IB column; solvent: hexane/2-propanol = 90/10; flow rate: 1.0 mL/min; detection: at 273 nm): *t*R minor = 5.17 min, *t*R major = 4.40 min. [α]<sub>D</sub><sup>22.4</sup> = -119.6 (*c* = 0.1, CHCl<sub>3</sub> for 90% ee).

**Procedure for catalytic enantioselective decarboxylative allylation of **6w**:** In an oven-dried round-bottom flask, Et<sub>2</sub>O (1.5 mL) was degassed by using nitrogen balloon at room temperature over a period of 10 minutes. 0.025 mol% of Pd<sub>2</sub>(dba)<sub>3</sub> and 0.075 mol% of ligand were added to it and stirring was continued for 20 minutes to make the complex mixture. After that reaction mixture was cooled to the -25 °C. In another vessel (1:1) mixture of carbonate (**6w**) (0.06 mmol; 1.0 equiv) were dissolved in dry degassed (1.5 mL) of Et<sub>2</sub>O, then the resulting solution was added drop-wise to the complex solution and stirring was continued for specified time at -25 °C. After complete consumption of starting material (monitored by TLC) the reaction mixture was concentrated and purified by column chromatography to afford the desired enantioenriched mixture of compounds **8a** and **8b**. Further this mixture was reacted with TBSCl (4.0 equiv.) in presence of imidazole (8.0 equiv.) to afford compounds **8a** and **5m**.





Compounds (*R*)-**8a** and (*R*)-**8b** are inseparable mixture in column chromatography, we have separated these two compounds in HPLC and we have observed 3:1 ratio of the peaks in the HPLC chromatogram; Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak ID-3 column; solvent: hexane/2-propanol = 60/40; flow rate: 1.0 mL/min; detection: at 254 nm). For minor compound  $t_{R1}$  minor = 5.38 min,  $t_{R}$  major = 5.72 min. (94% ee). For major compound  $t_{R1}$  minor = 6.90 min,  $t_{R}$  major = 11.04 min. (85% ee). These two compounds were separated by after TBSCl reaction and provided individual data for the same.



(*R*)-3-Allyl-3-(2-(allyloxy)-5-methylphenyl)-1-methylindolin- 2-one [(+)-**8a**]: The compound (+)-**8a** was obtained as orange color gel (0.06 mmol scale of reaction; 14 mg of product; 71%).  $R_f$  = 0.35 (20% EtOAc in hexane).

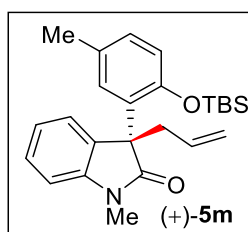
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.40 (d,  $J$  = 2.0 Hz, 1H), 7.23 (t,  $J$  = 7.6 Hz, 1H), 7.04 (dt,  $J$  = 8.3, 1.4 Hz, 1H), 6.95 (t,  $J$  = 7.4 Hz, 1H), 6.89 (d,  $J$  = 7.2 Hz, 1H), 6.80 (d,  $J$  = 7.7 Hz, 1H), 6.65 (d,  $J$  = 8.2 Hz, 1H), 5.51 - 5.40 (m, 1H), 5.38 - 5.30 (m, 1H), 5.09 - 4.93 (m, 3H), 5.06 - 4.98 (m, 1H), 4.20 (dd,  $J$  = 12.0, 6.1 Hz, 1H), 4.01 (dd,  $J$  = 12.1, 5.5 Hz, 1H), 3.22 (s, 3H), 3.01 (d,  $J$  = 7.2 Hz, 2H), 2.38 (s, 3H).

**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  179.1, 153.9, 144.6, 133.5, 133.2, 131.8, 129.9, 129.2, 128.8, 128.5, 127.3, 122.5, 122.0, 119.0, 117.6, 112.8, 107.1, 69.5, 53.7, 40.7, 26.1, 20.9.

**IR** (film)  $\nu_{\max}$  3430, 2219, 1718, 1662, 1430, 1362, 1208, 1147, 1021, 920, 820, 713, 610  $\text{cm}^{-1}$ .

**HRMS** (ESI-TOF)  $m/z$ :  $[\text{M} + \text{Na}]^+$  Calcd for  $[\text{C}_{22}\text{H}_{23}\text{NO}_2 + \text{Na}]^+$  356.1621; Found 356.1650.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak ID-3 column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_{\text{R}}$  minor = 6.00 min,  $t_{\text{R}}$  major = 9.68 min.  $[\alpha]_{\text{D}}^{22.0} = +33.0$  ( $c = 0.13$ , MeOH for 85% ee).



**(*R*)-3-Allyl-3-(2-((*tert*-butyldimethylsilyloxy)-5-methylphenyl)-1-methylindolin-2-one [(+)-5m]:** The compound (+)-5m was obtained as orange color gel (0.06 mmol scale of reaction; 5 mg of product; 22% yield.

**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 (d,  $J = 2.3$  Hz, 1H), 7.17 (td,  $J = 7.5, 1.7$  Hz, 1H), 6.93 - 6.86 (m, 3H), 6.75 (d,  $J = 7.7$  Hz, 1H), 6.62 (d,  $J = 8.2$  Hz, 1H), 5.24 (ddt,  $J = 17.2, 10.1, 7.1$  Hz, 1H), 4.95 - 4.90 (m, 1H), 4.85 (dd,  $J = 10.1, 2.0$  Hz, 1H), 3.19 (s, 3H), 2.92 (d,  $J = 7.1$  Hz, 2H), 2.32 (s, 3H), 0.67 (s, 9H), -0.02 (s, 3H), -0.07 (s, 2H).

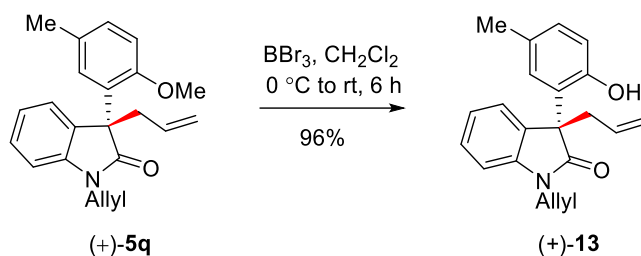
**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$  178.4, 151.4, 144.1, 133.5, 131.9, 129.2 (2C), 128.9, 128.5, 127.2, 122.5, 122.1, 118.8, 117.6, 107.3, 54.2, 42.1, 26.3, 20.9, 19.1, -3.6, -3.6.

**IR** (film)  $\nu_{\max}$  3437, 2960, 2116, 1717, 1628, 1495, 1422, 1318, 1204, 1104, 1084, 1001, 996, 802, 772, 617, 603  $\text{cm}^{-1}$ .

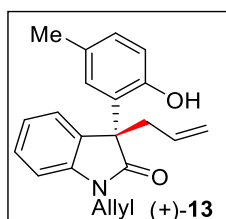
**HRMS** (ESI-TOF)  $m/z$ :  $[M + Na]^+$  Calcd for  $[C_{25}H_{33}NO_2Si + Na]^+$  430.2173; Found 430.2160.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IC-3 column; solvent: hexane/2-propanol = 75/25; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  minor = 6.67 min,  $t_R$  major = 7.36 min.  $[\alpha]_D^{24.0} = +18.0$  ( $c = 0.28$ ,  $CH_2Cl_2$  for 94% ee).

**Synthetic procedure for compound (+)-13:**



In an oven dried round-bottom flask, compound (+)-**5q** (100 mg; 0.28 mmol; 1.0 equiv) was taken in dry dichloromethane under nitrogen atmosphere. After cooling the reaction mixture at 0 °C, boran tribromide (53  $\mu\text{L}$ ); 0.56 mmol; 2.0 equiv.) was added stirring was continued to room temperature for 6 h. After completion of the reaction (judged by TLC analysis under UV and  $I_2$  stain), diluted with EtOAc (20 mL) and quenched with  $H_2O$ . Then the organic layer was separated, dried with  $Na_2SO_4$ . The crude mixture was purified by column chromatography using EtOAc and hexane mixture as eluent to afford the desired product (+)-**13**.



**(R)-1,3-diallyl-3-(2-hydroxy-5-methylphenyl)indolin-2-one [(+)-13]**: The compound (+)-**13** was obtained as orange color gel (0.28 mmol scale of reaction; 93 mg of product; 96%).  $R_f = 0.50$  (20% EtOAc in hexane).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$  10.31 (s, 1H), 7.41 (d,  $J = 7.4$  Hz, 1H), 7.35 (td,  $J = 7.7, 1.3$  Hz, 1H), 7.24 (dd,  $J = 8.7, 6.4$  Hz, 1H), 6.99 (dd,  $J = 8.1, 2.1$  Hz, 1H), 6.94 - 6.91 (m, 2H), 6.80 (d,  $J = 2.1$  Hz, 1H), 5.78 (ddt,  $J = 17.4, 10.3, 5.2$  Hz, 1H), 5.29 - 5.16 (m, 3H), 5.04 - 4.99 (m, 1H), 4.91 (dd,  $J = 10.0, 1.9$  Hz, 1H), 4.55 - 4.33 (m, 1H), 4.24 (ddt,  $J = 16.3, 5.3, 1.7$  Hz, 1H), 3.47 (dd,  $J = 13.7, 8.3$  Hz, 1H), 2.93 (dd,  $J = 13.7, 6.3$  Hz, 1H), 2.14 (s, 3H).

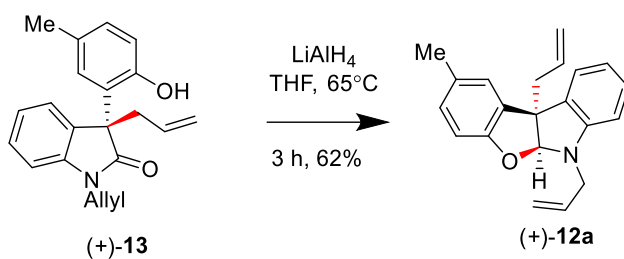
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$  180.8, 154.6, 142.3, 132.0, 130.7, 130.0, 129.9, 129.1, 128.7, 128.5, 126.6, 123.5, 123.0, 120.1, 119.7, 117.9, 109.9, 57.8, 42.7, 39.4, 20.6.

**IR** (film)  $\nu_{\max}$  3427, 2860, 2216, 1725, 1630, 1425, 1420, 1398, 1264, 1114, 1034, 1001, 946, 856, 788, 714, 714, 653 cm<sup>-1</sup>.

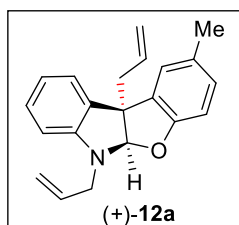
**HRMS** (ESI-TOF)  $m/z$ : [M + Na]<sup>+</sup> Calcd for [C<sub>21</sub>H<sub>21</sub>NO<sub>2</sub> + Na]<sup>+</sup> 342.1465; Found 338.1481.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak IC-3 column; solvent: hexane/2-propanol = 70/30; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  major = 4.37 min.  $t_R$  minor = 6.72 min,  $[\alpha]_D^{22.4} = +427.0$  ( $c = 0.047$ , MeOH for 90% ee).

**Synthetic procedure for compound (+)-12a:**



In an oven-dried round-bottom flask was charged with (+)-**13** (50 mg; 0.20 mmol; 1.0 equiv) was taken in tetrahydrofuran under nitrogen atmosphere. To this solution  $\text{LiAlH}_4$  (16 mg; 0.40 mmol; 2.0 equiv.) was added at RT, stirring was continued at 65 °C for 3 h. After completion of the reaction (Judged by TLC analysis under UV and  $\text{I}_2$  stain), quenched with EtOAc (15 mL) and diluted with water. Then the organic layer was separated, dried with  $\text{Na}_2\text{SO}_4$ . The crude mixture was purified by column chromatography using EtOAc and hexane mixture as eluent to afford the desired product (+)-**12a**.



**(R)-6,10b-Diallyl-2-methyl-5a,10b-dihydro-6H-benzofuro[2,3-b]indole [(+)-12a]**: The compound (+)-**12a** was obtained as orange color gel (0.20 mmol scale of reaction; 38 mg of product; 62%).  $R_f = 0.72$  (5% EtOAc in hexane).

**$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.24 - 7.21 (m, 1H), 7.10 (d,  $J = 1.7$  Hz, 1H), 7.05 (t,  $J = 7.7$  Hz, 1H), 6.87 (dd,  $J = 8.2, 1.8$  Hz, 1H), 6.71 (t,  $J = 7.3$  Hz, 1H), 6.66 (d,  $J = 8.1$  Hz, 1H), 6.42 (d,  $J = 7.8$  Hz, 1H), 6.01 (s, 1H), 5.90 (ddt,  $J = 16.0, 10.5, 5.3$  Hz, 1H), 5.53 (ddt,  $J = 17.2, 10.1, 7.1$  Hz, 1H), 5.28 (dd,  $J = 17.1, 1.9$  Hz, 1H), 5.19 (dd,  $J = 10.4, 1.7$  Hz, 1H), 5.16 - 5.02 (m, 2H), 4.03 - 4.01 (m, 2H), 2.80 (d,  $J = 7.3$  Hz, 2H), 2.27 (s, 3H).

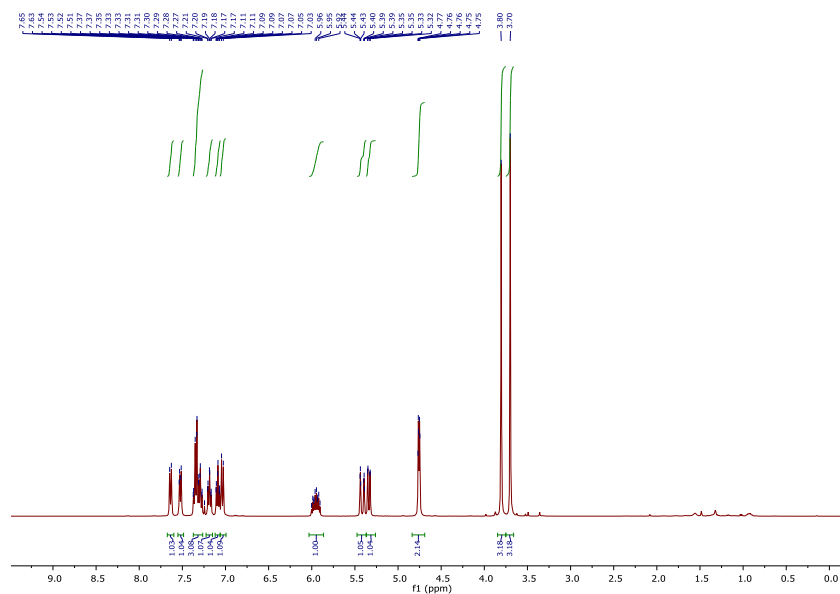
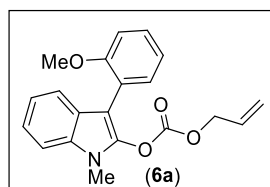
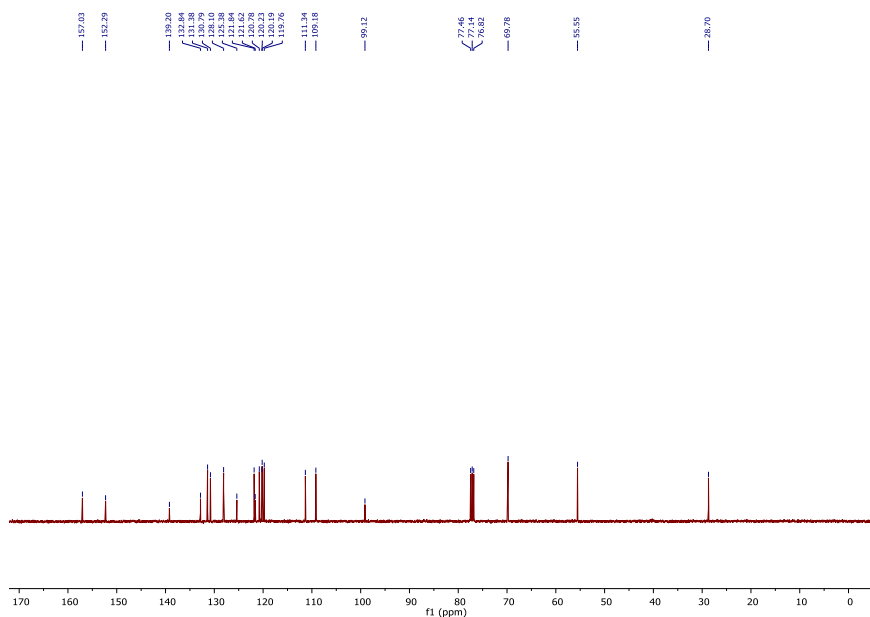
**<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>) δ 156.6, 148.4, 133.4, 133.3, 132.1, 131.5, 130.2, 128.7, 128.1, 123.5, 122.3, 118.6, 118.2, 117.1, 109.4, 106.4, 105.3, 58.4, 47.8, 41.7, 20.9.

**IR** (film)  $\nu_{\max}$  3337, 2960, 2816, 2101, 1680, 1465, 1410, 1378, 1204, 1110, 1074, 1021, 906, 799, 708, 650 cm<sup>-1</sup>.

**HRMS** (ESI-TOF)  $m/z$ : [M + H]<sup>+</sup> Calcd for [C<sub>21</sub>H<sub>22</sub>NO]<sup>+</sup> 304.1696; Found 304.1717.

Enantiomeric excess of pure compound was determined *via* HPLC analysis using a Chiralpak AD-H column; solvent: hexane/2-propanol = 95/05; flow rate: 1.0 mL/min; detection: at 254 nm):  $t_R$  major = 4.37 min.  $t_R$  minor = 5.34 min,  $[\alpha]_D^{24.2} = +63.6$  ( $c = 0.13$ , MeOH for 90% ee).

## Spectral graphs and HPLC data

 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **6a** $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **6a**

## Display Report

## Analysis Info

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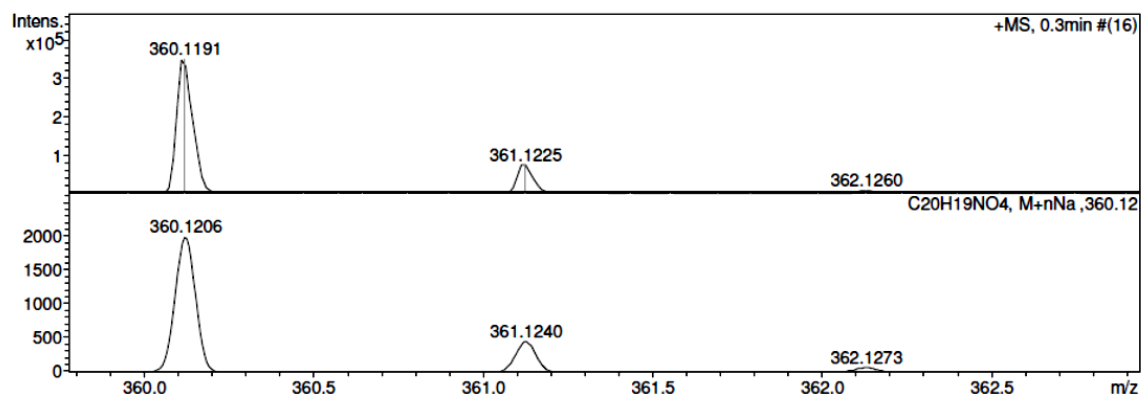
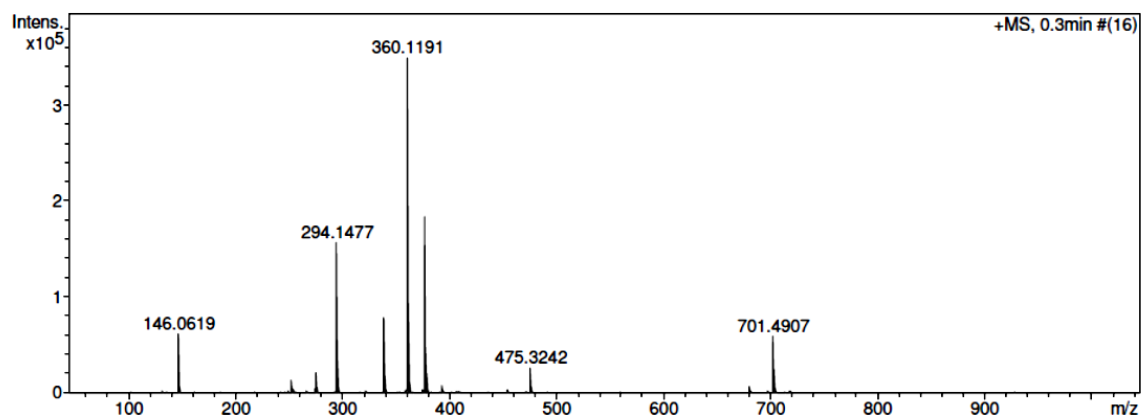
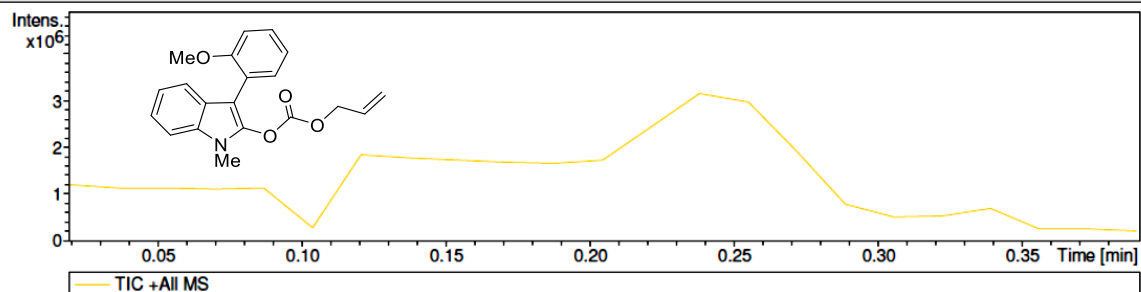
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Operator RUCHI

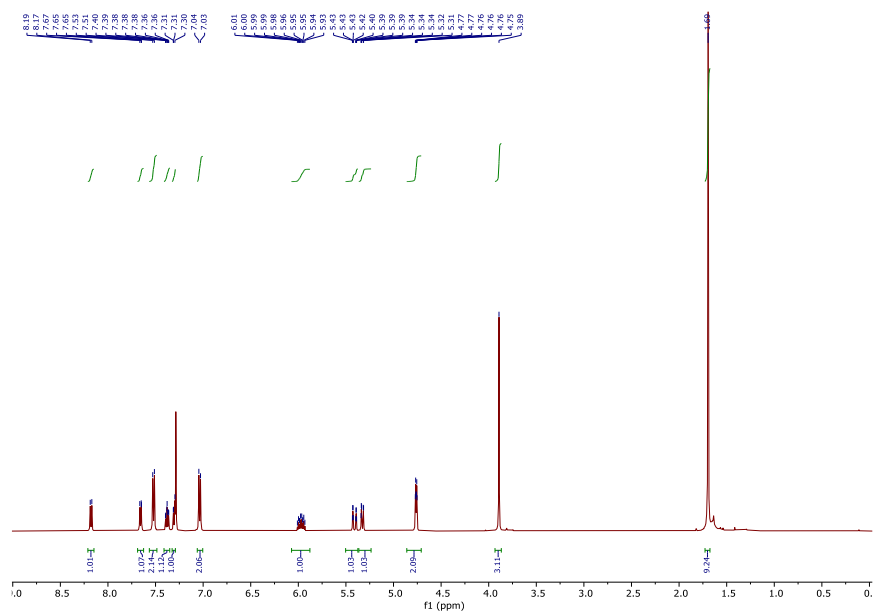
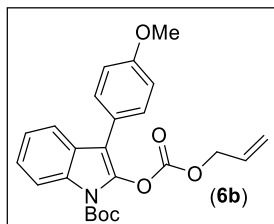
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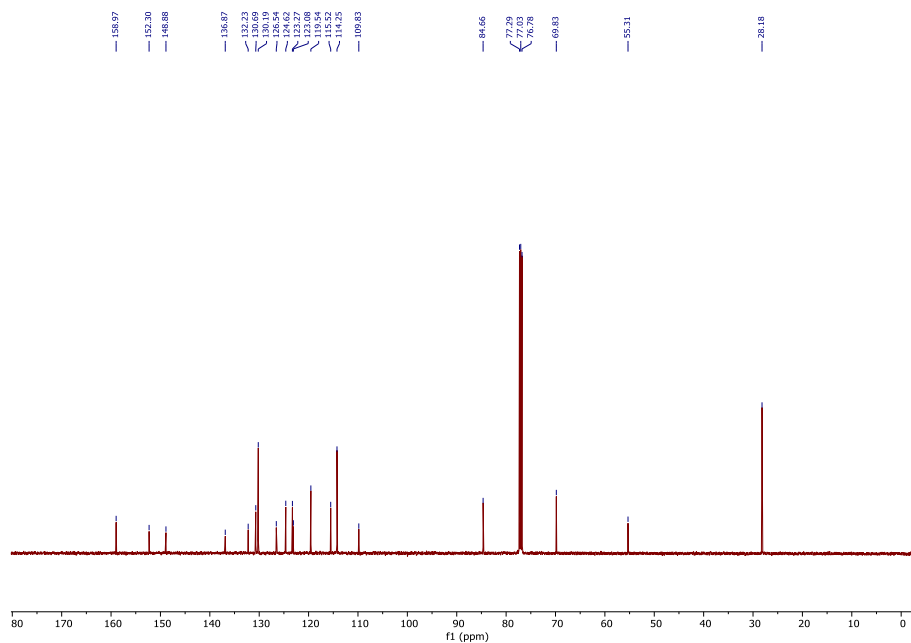
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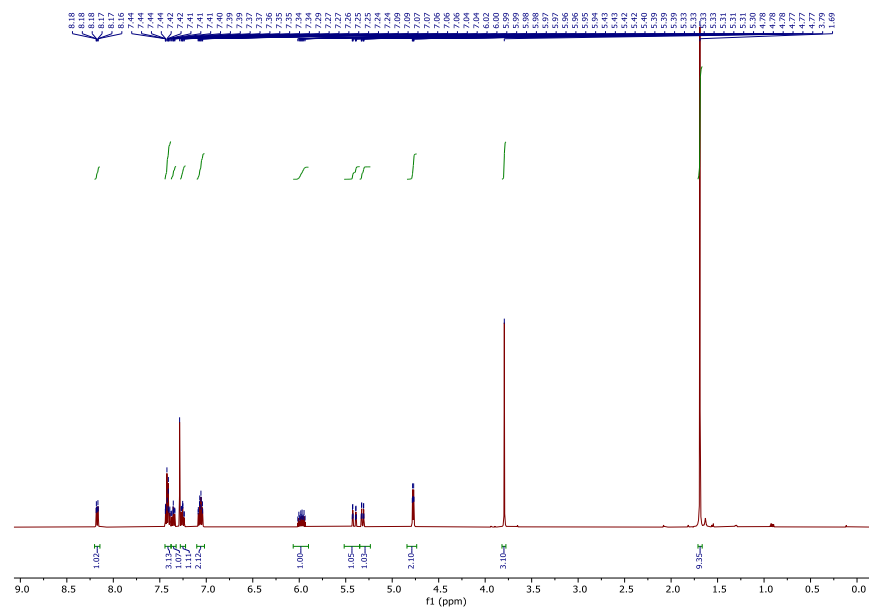
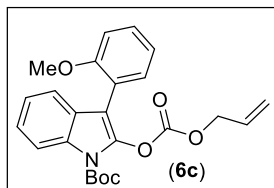
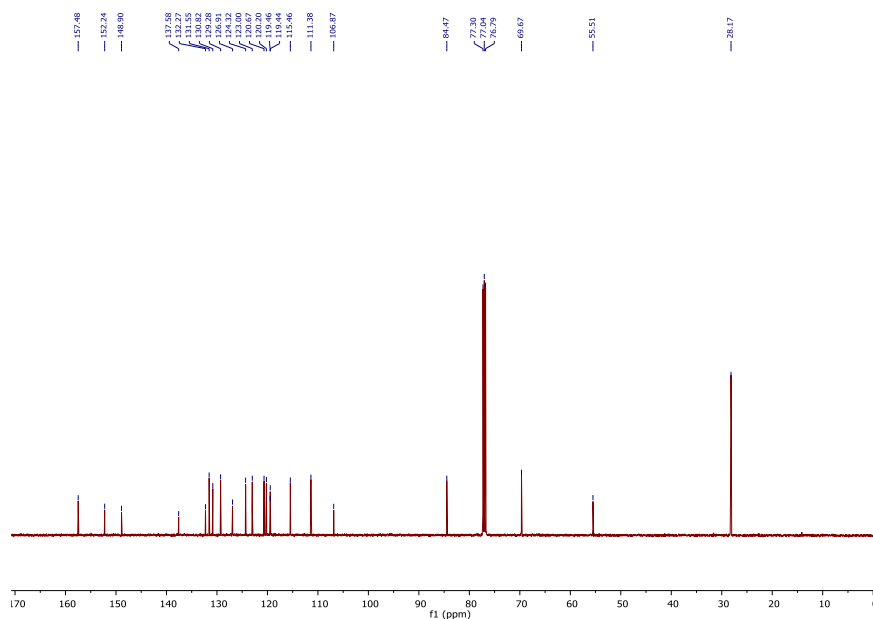


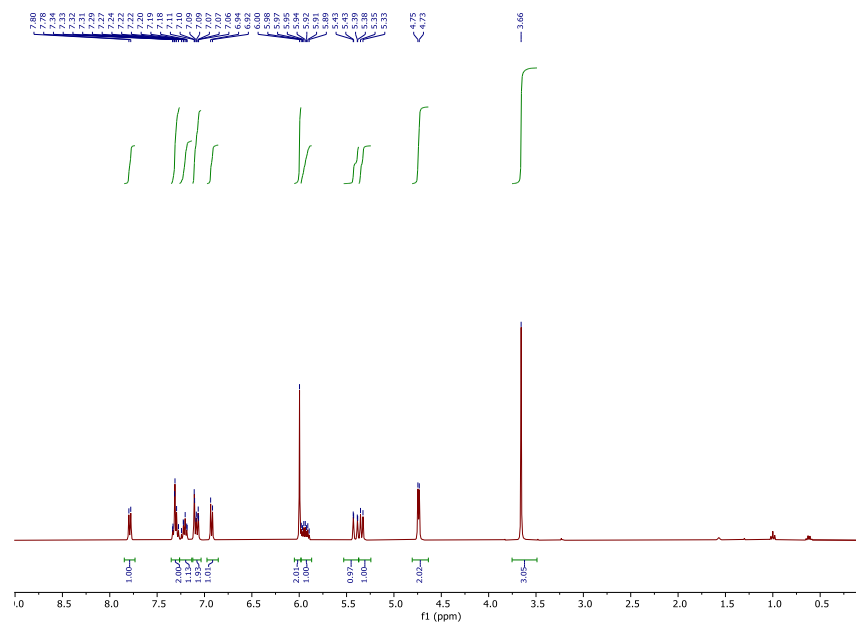
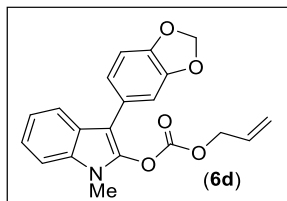


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6b**

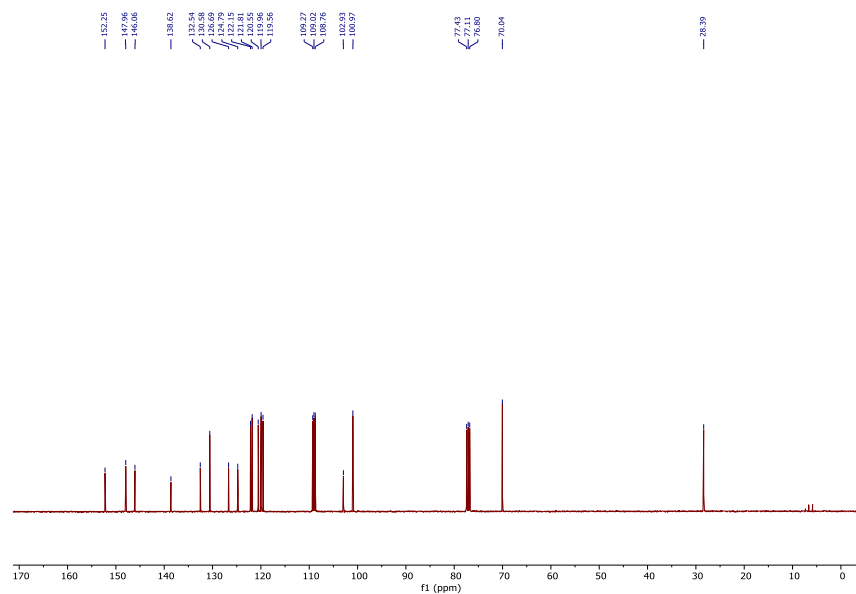


$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound **6b**

 $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6c** $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound **6c**



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound **6d**



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound **6d**

## Display Report

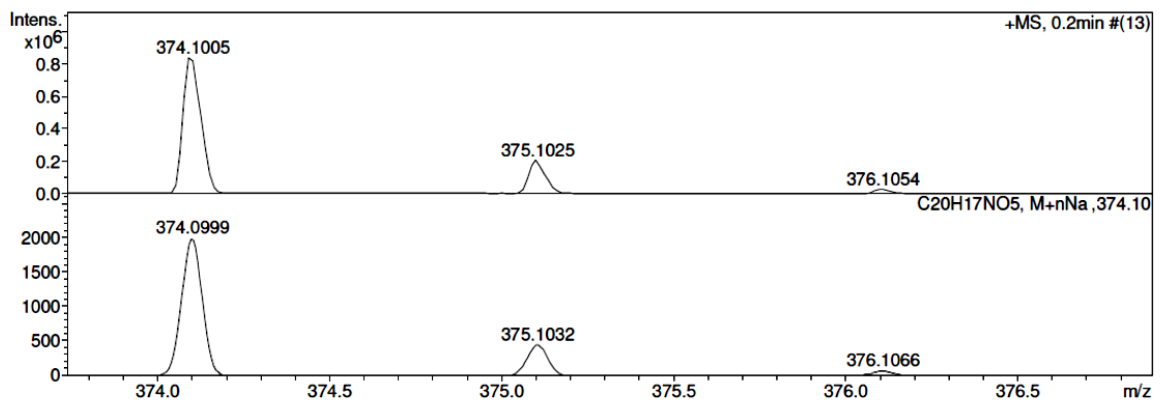
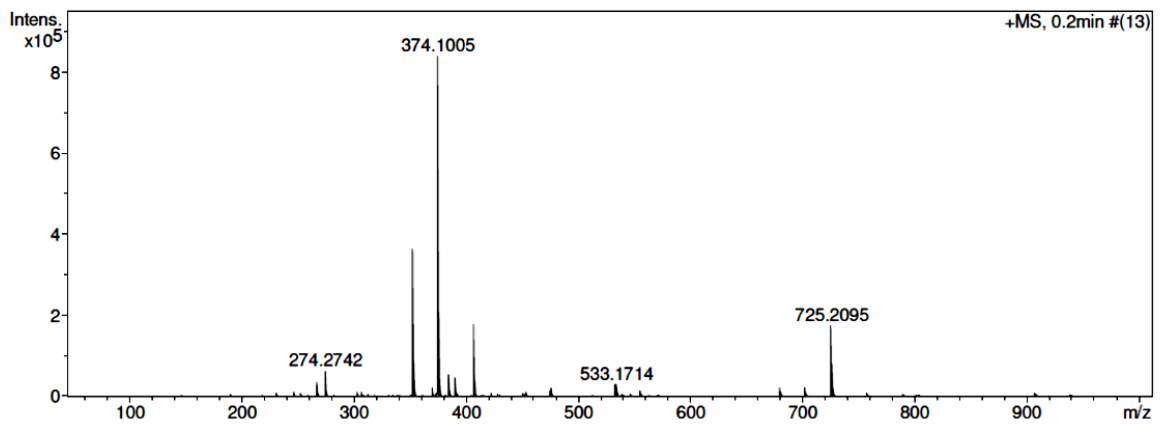
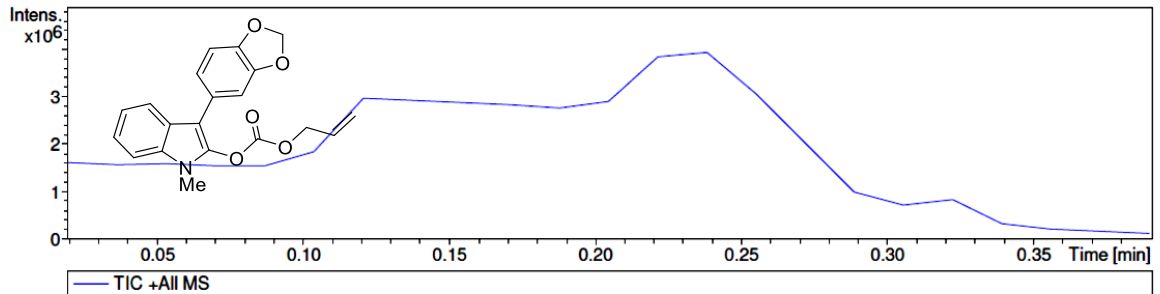
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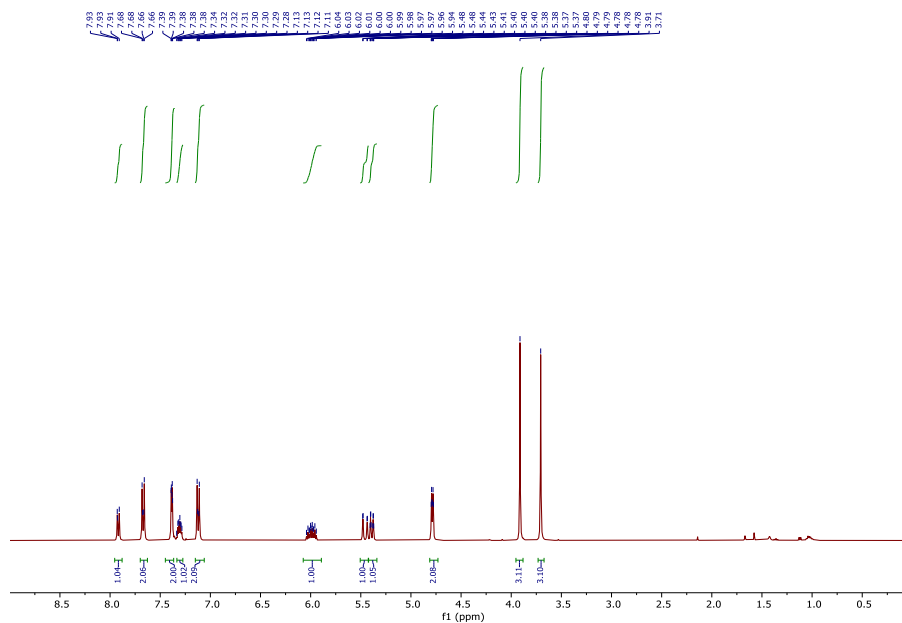
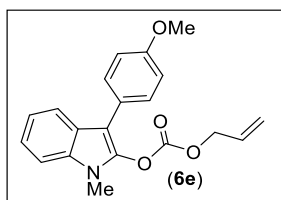
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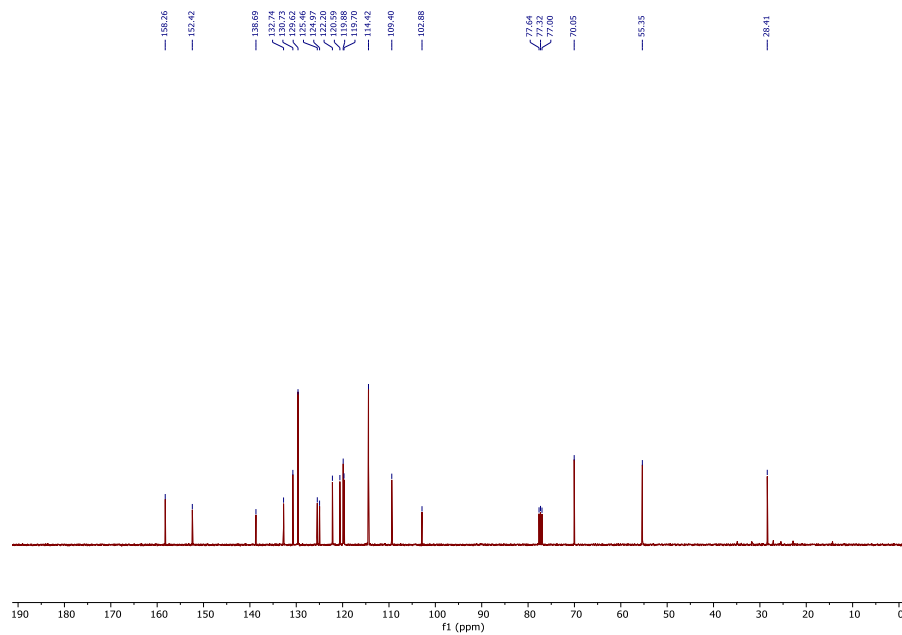
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$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **6e**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **6e**

## Display Report

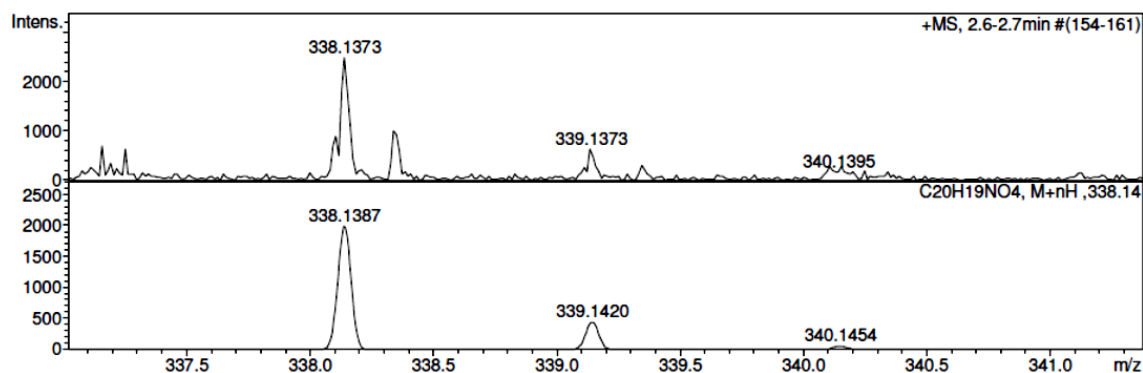
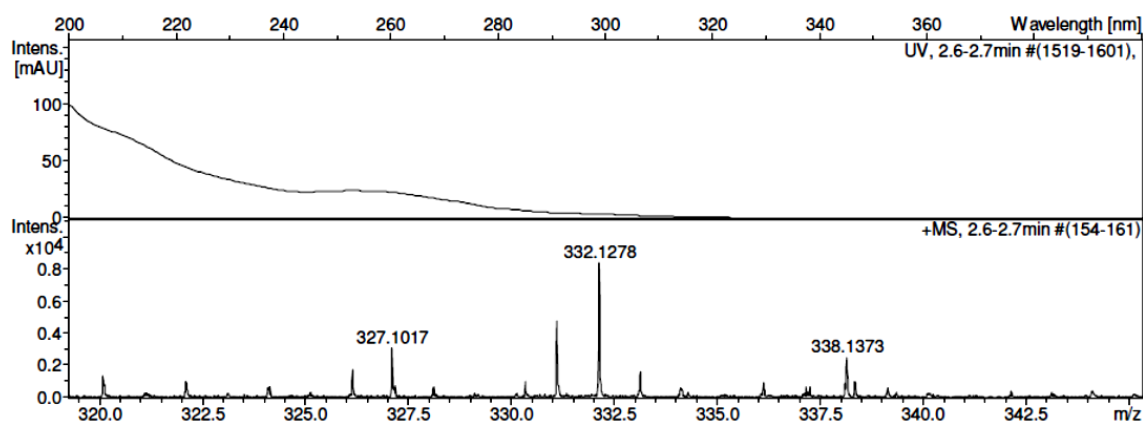
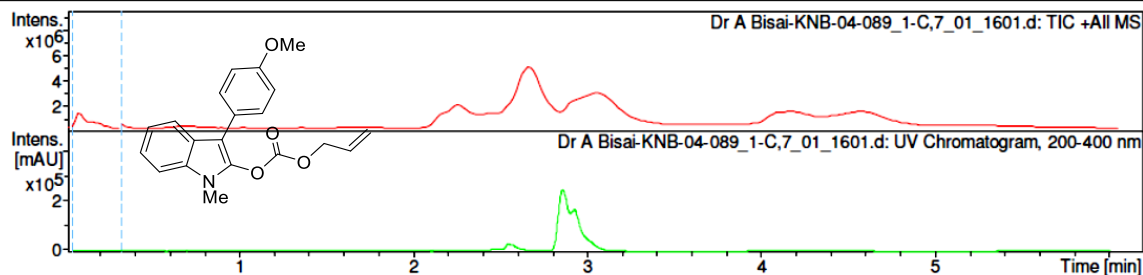
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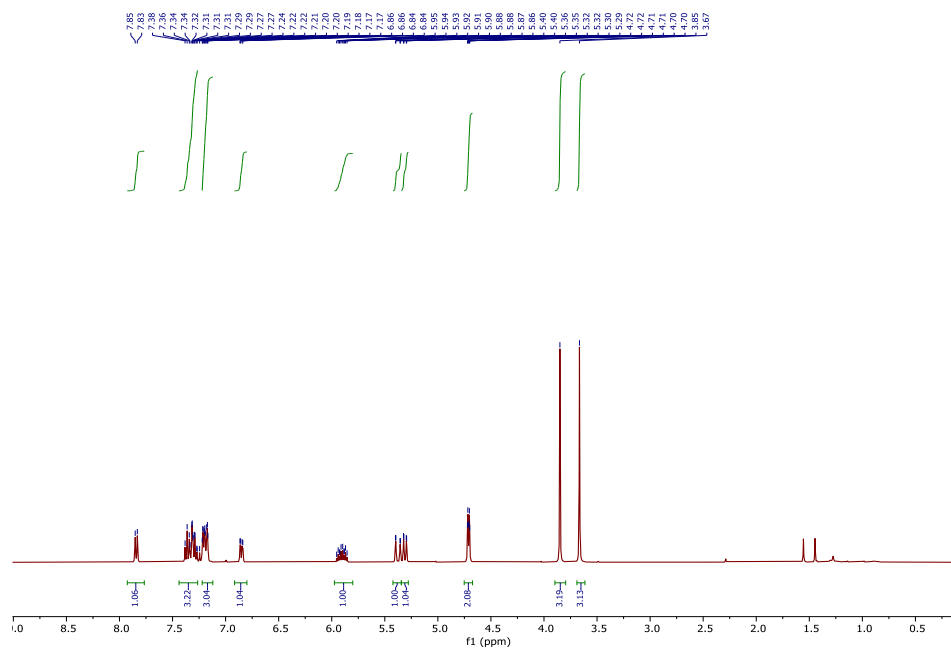
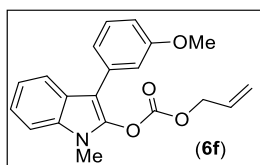
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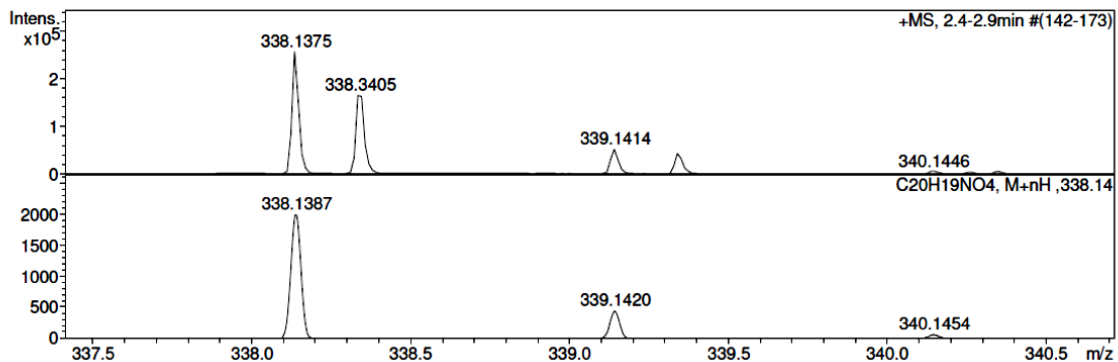
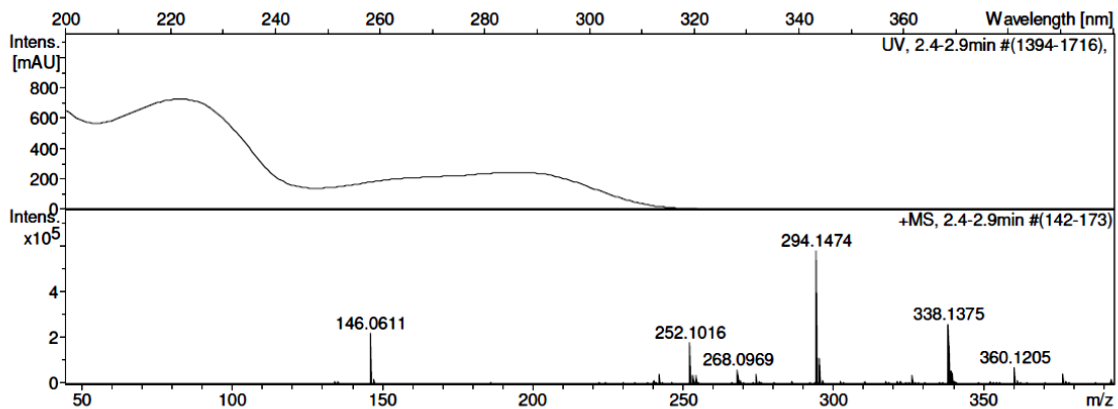
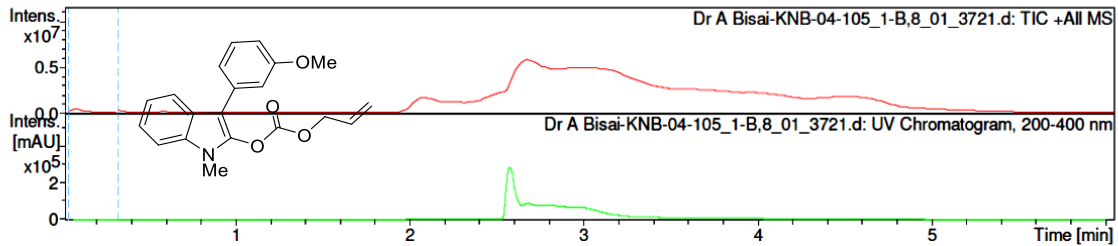


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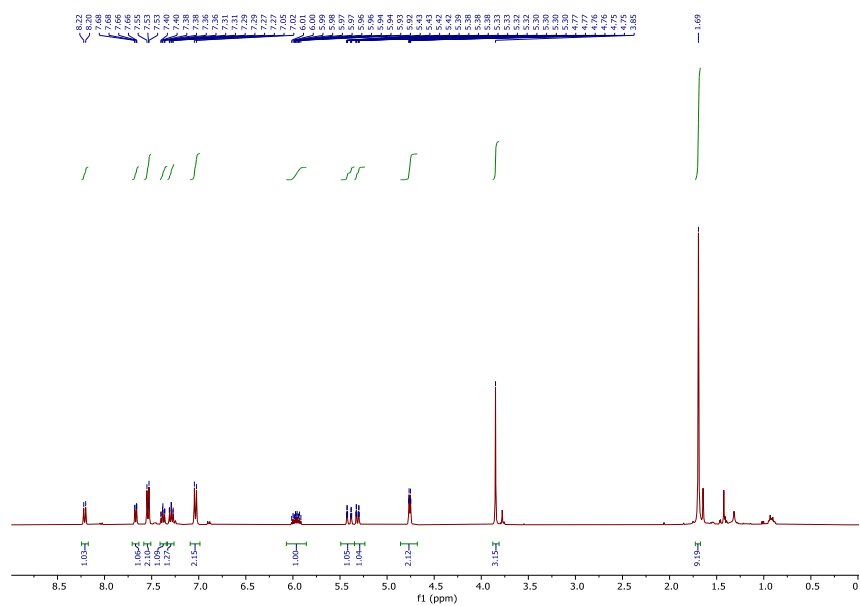
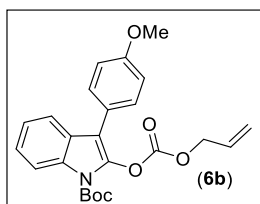
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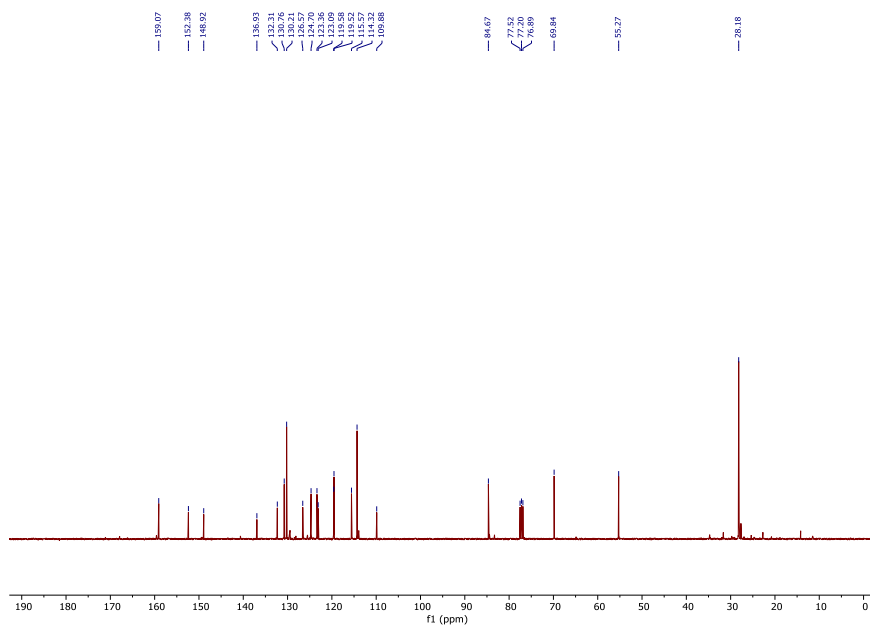
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$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound **6b**



$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ) of compound **6b**

## Display Report

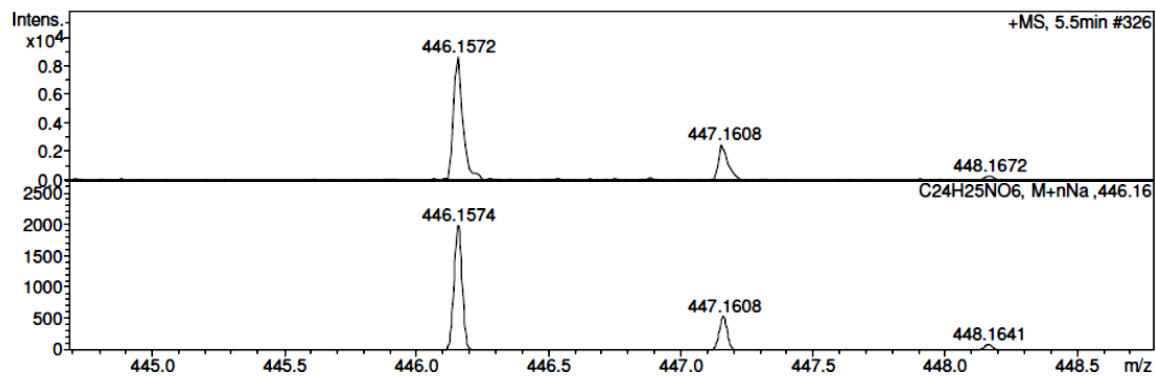
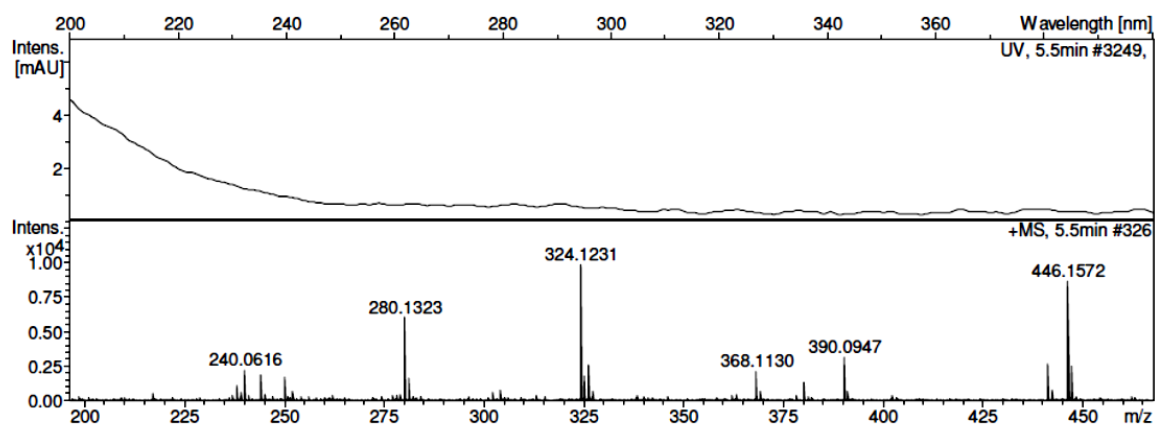
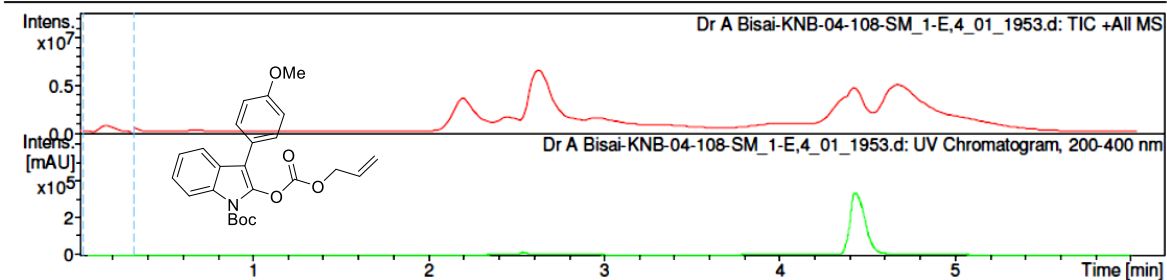
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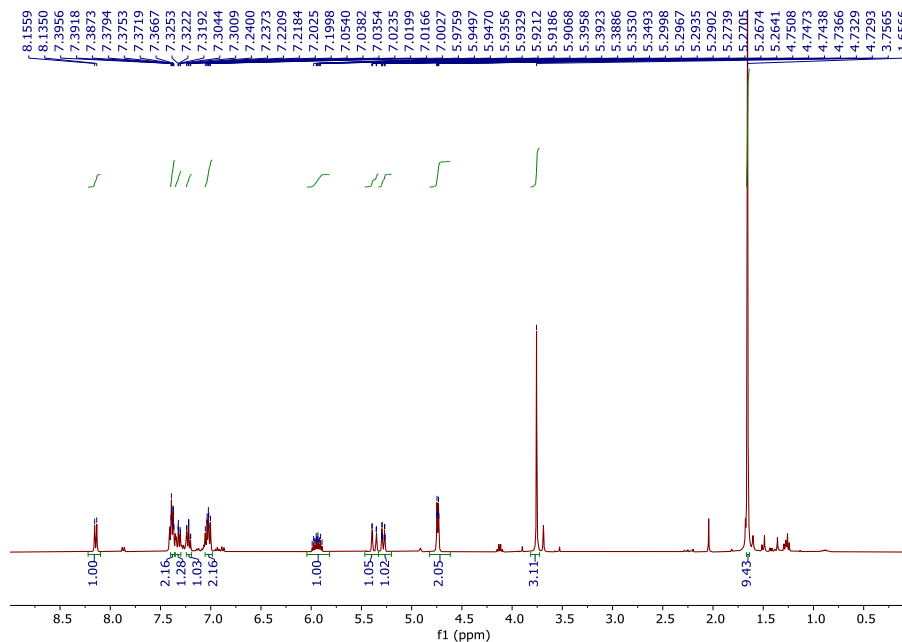
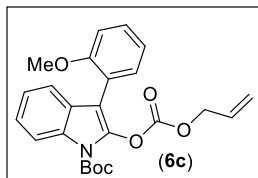
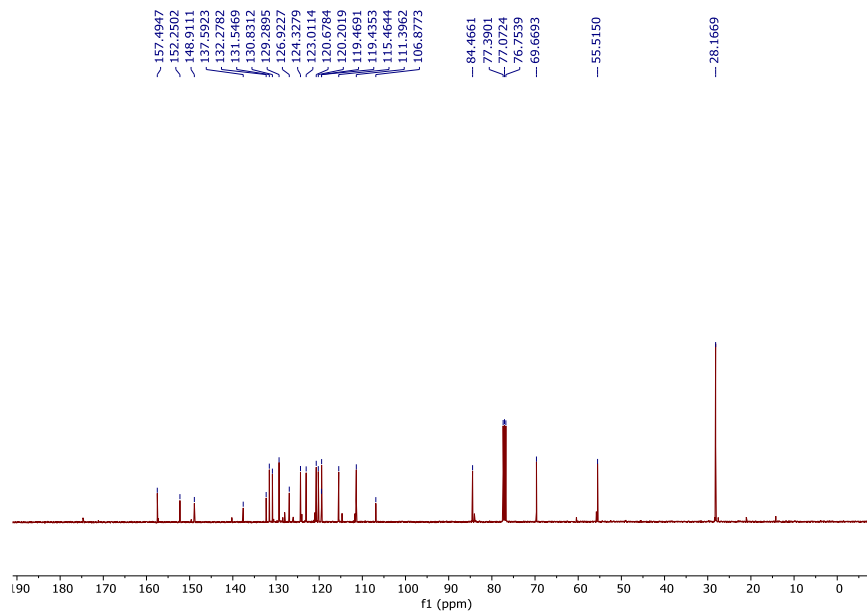
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Operator RUCHI  
Instrument micrOTOF-Q II 10330

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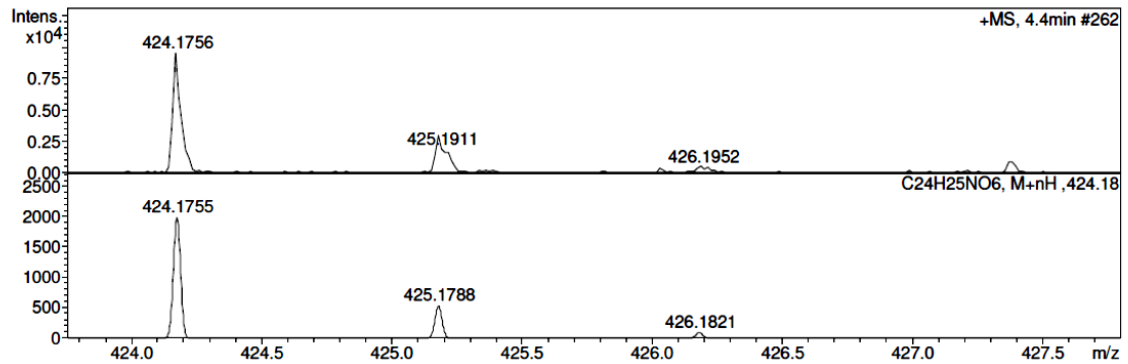
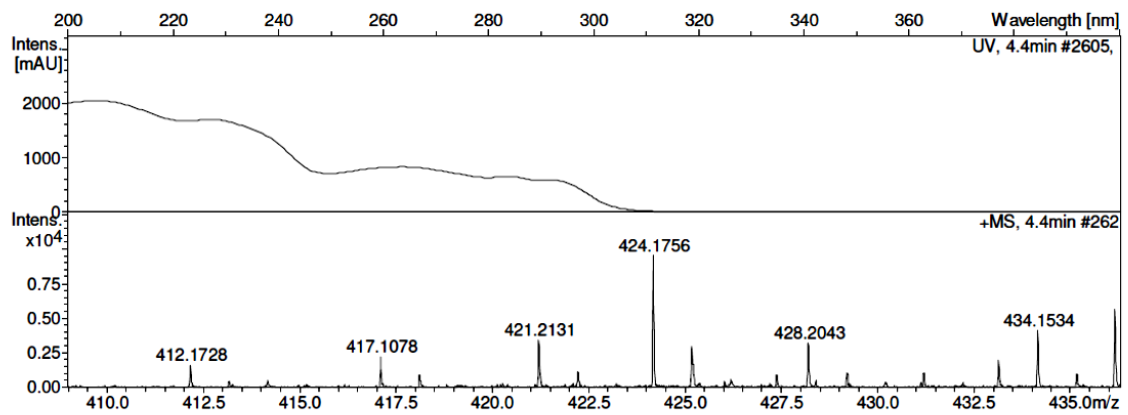
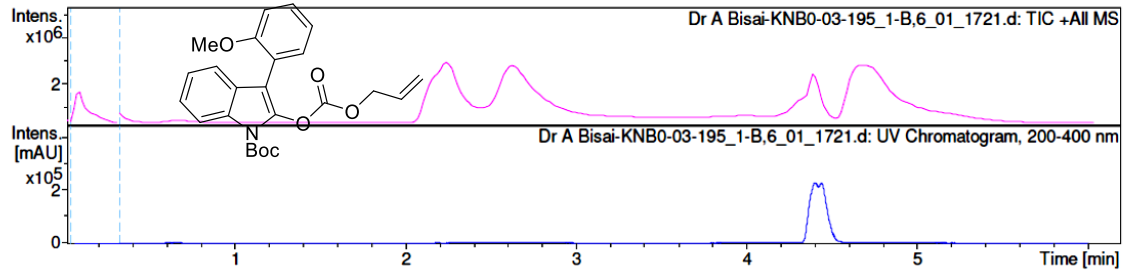
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound **6c**<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound **6c**

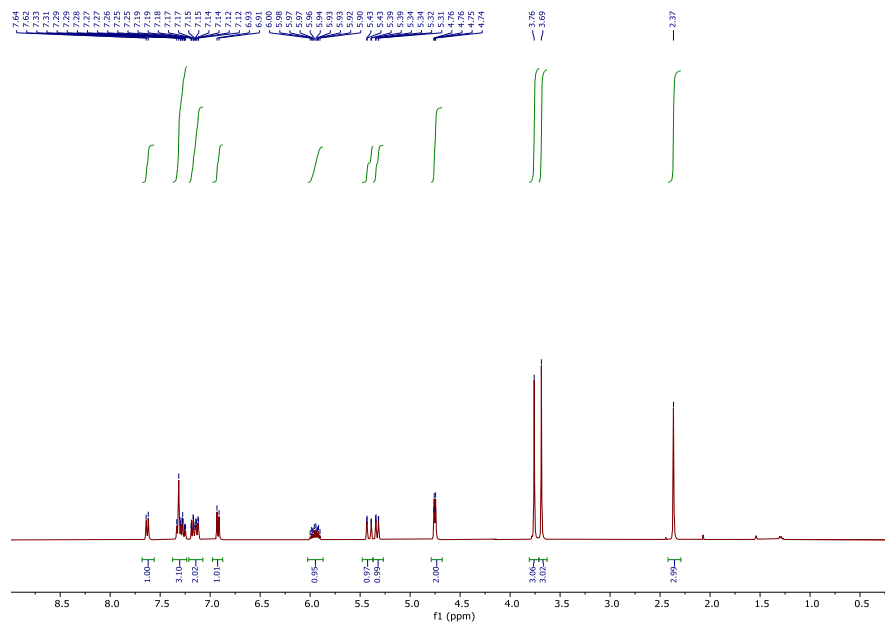
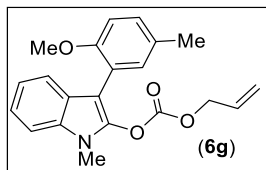
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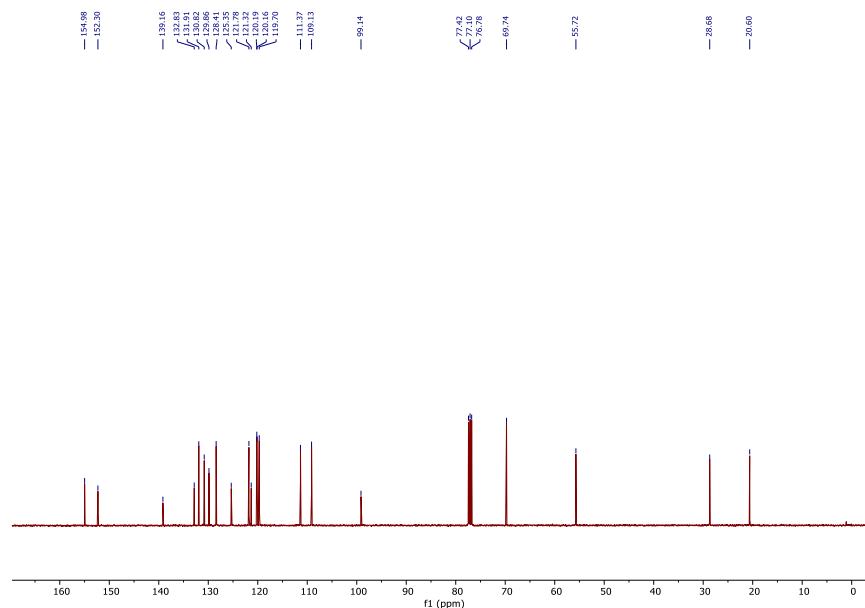
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Scanned copy of mass spectrum of **6c**



$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) of compound **6g**



$^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ) of compound **6g**

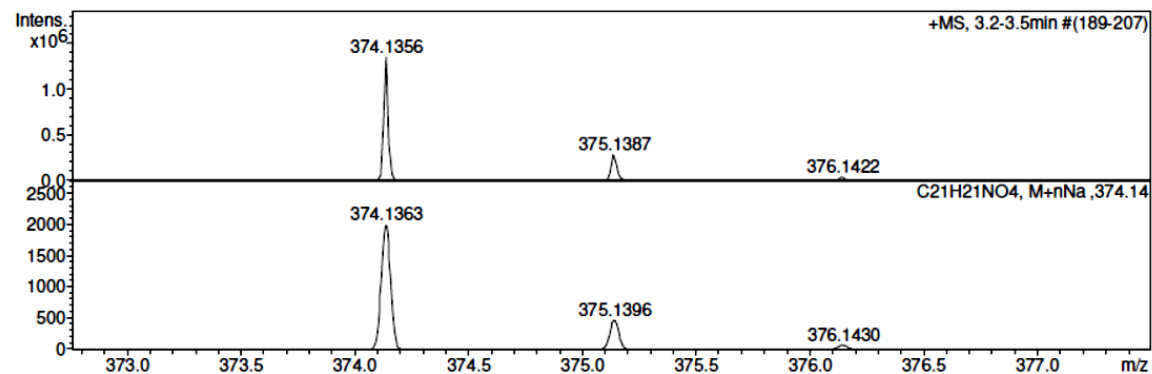
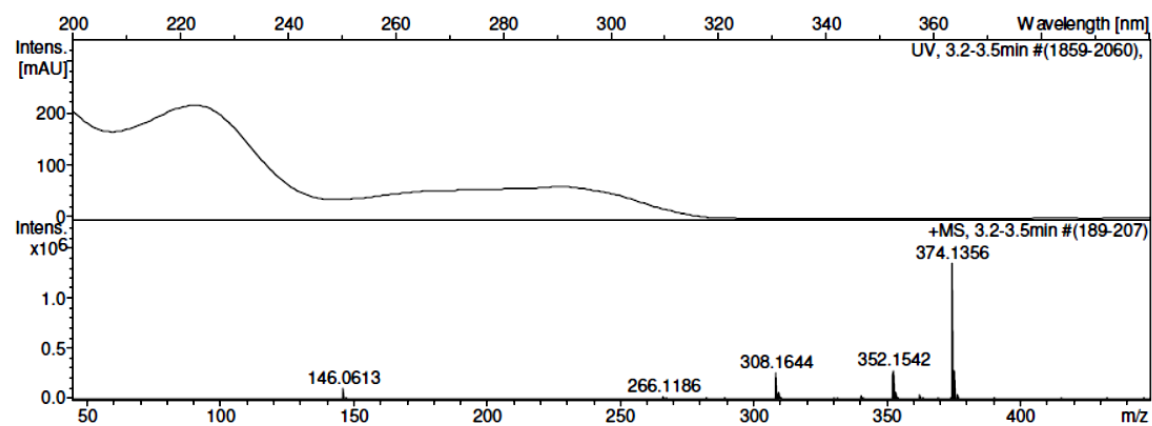
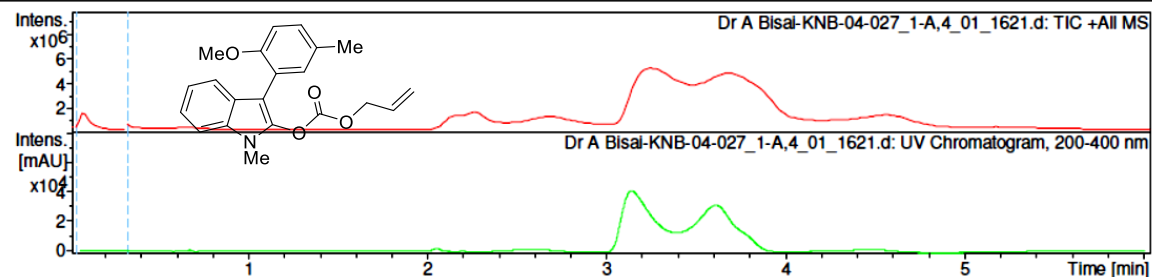
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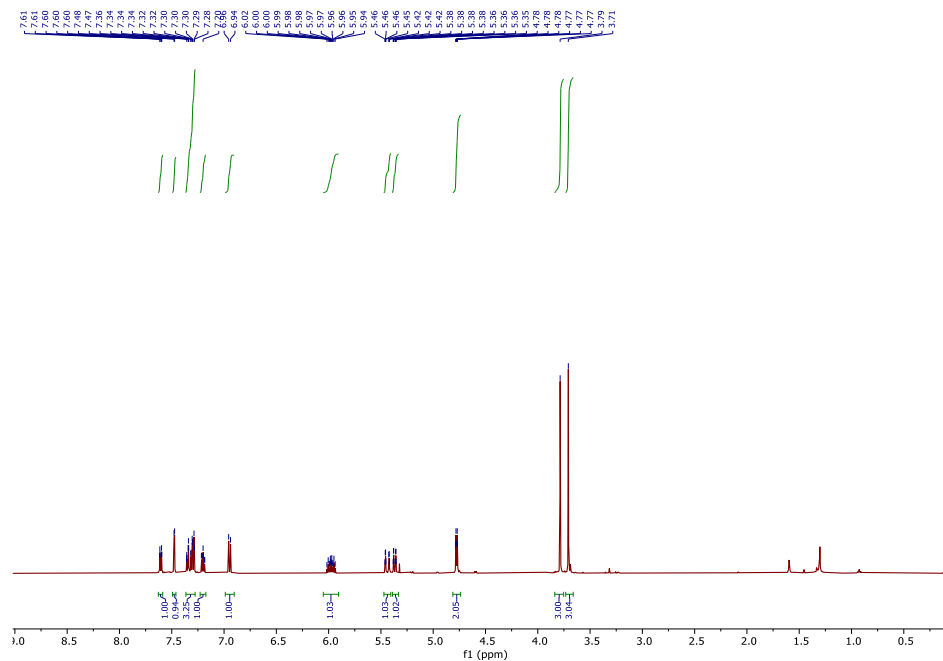
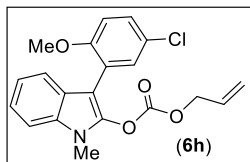
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Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-KNB-04-027  
Comment

Acquisition Date 5/8/2018 11:36:01 AM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

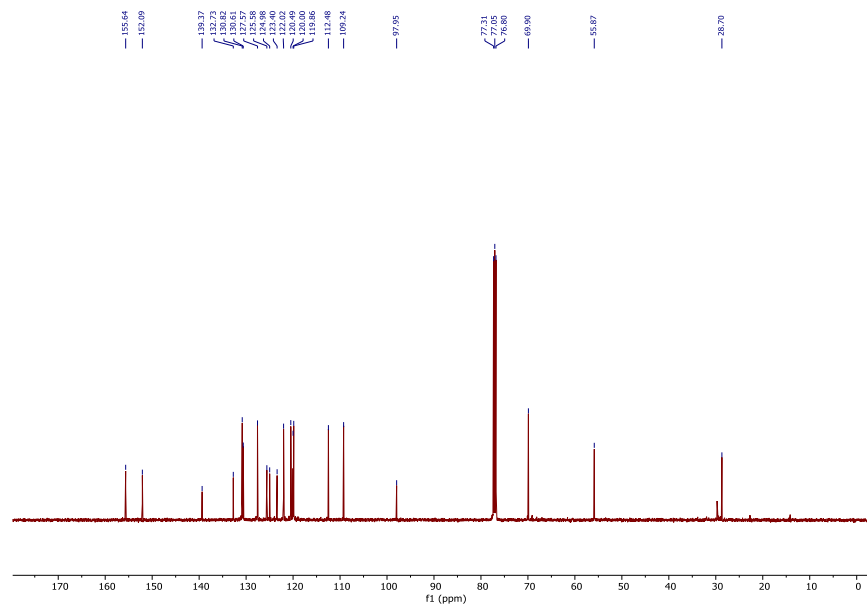
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **6g**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6h**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound **6h**

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\May-2018\05-05-2018\Dr.A.Bisai-AB-KNB-03-049\_1-A,6\_01\_1576.d  
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Sample Name Dr.A.Bisai-AB-KNB-03-049  
Comment

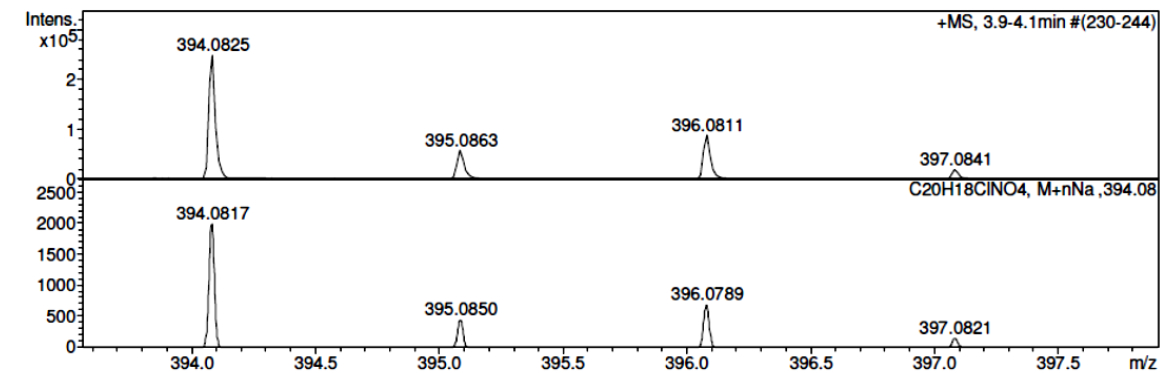
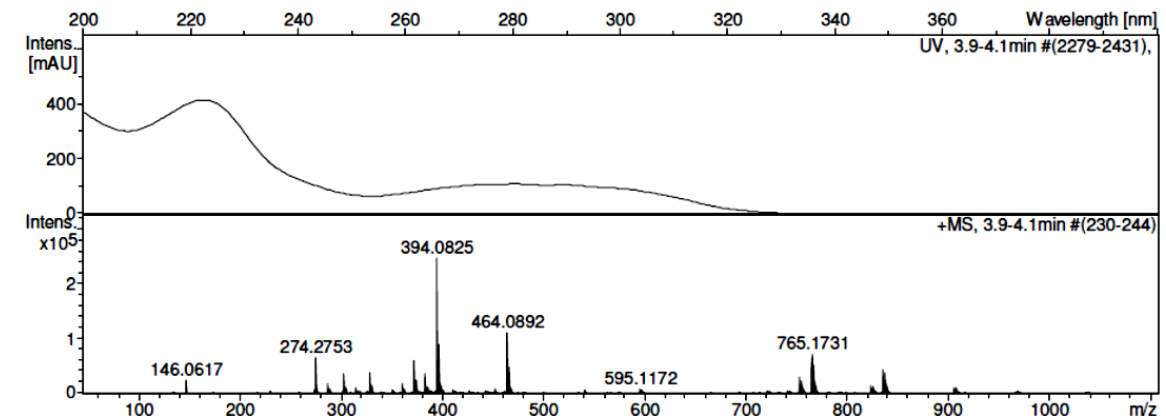
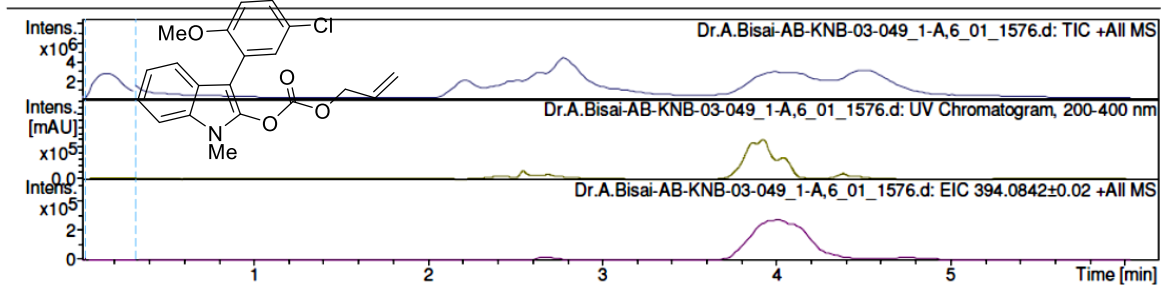
Acquisition Date 5/5/2018 10:55:09 AM

Operator RUCHI

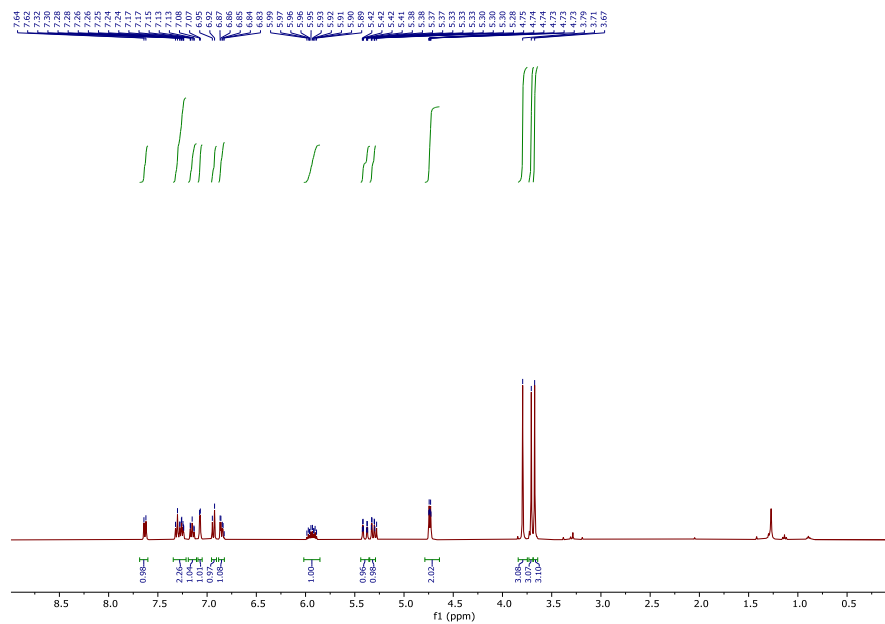
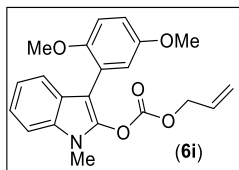
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

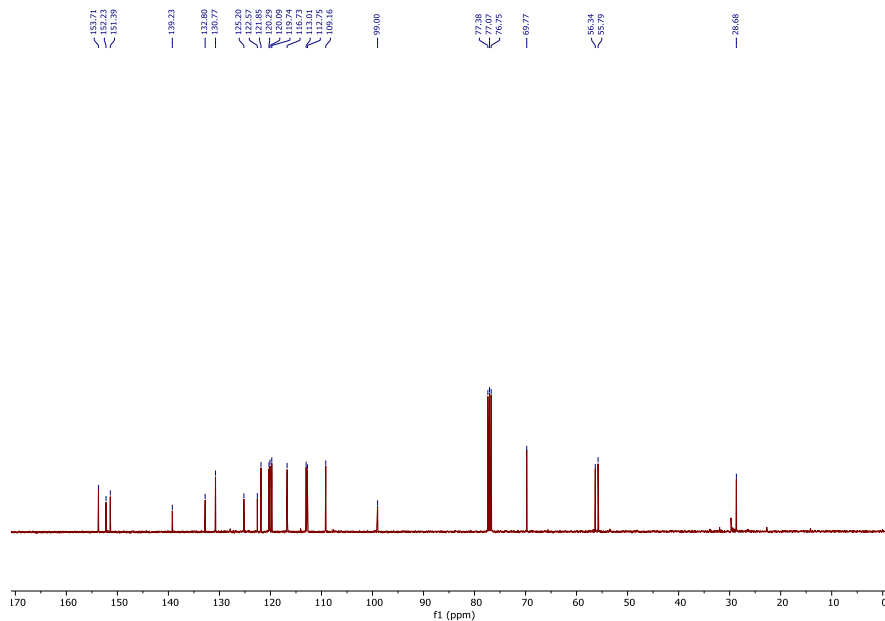
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6i**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound **6i**

## Display Report

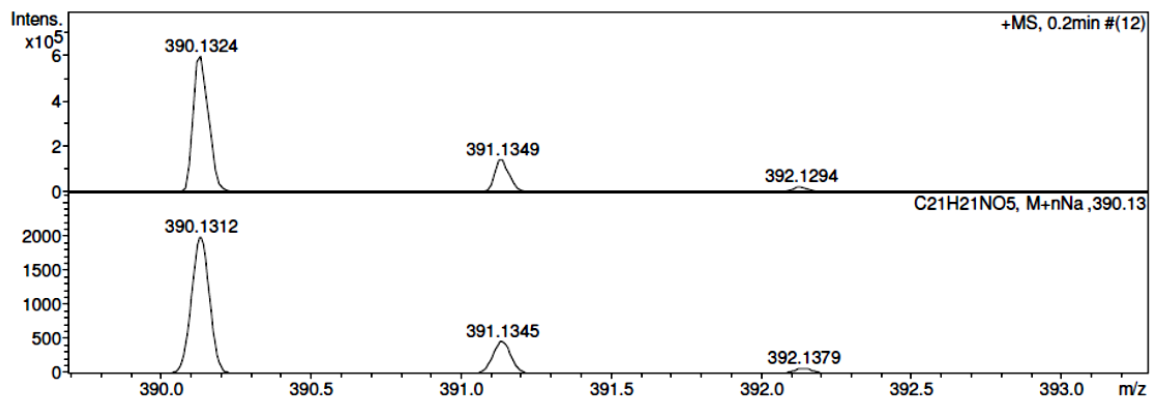
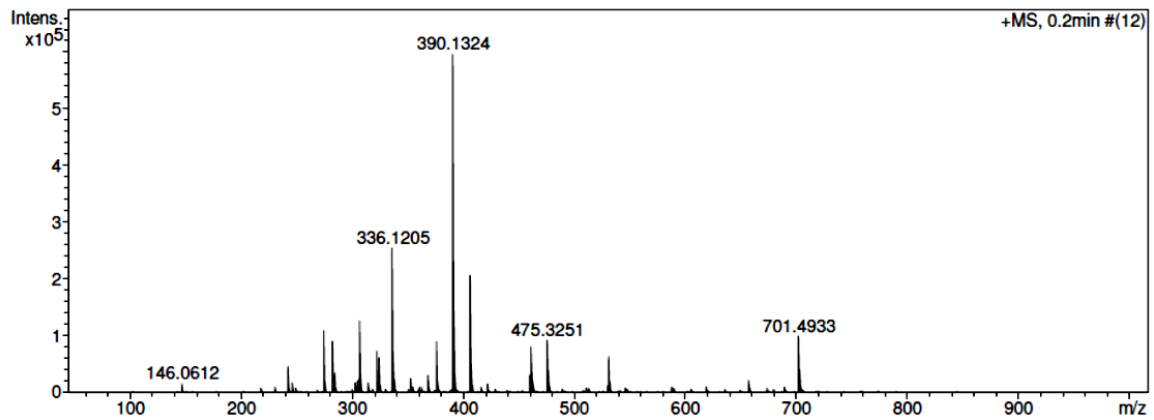
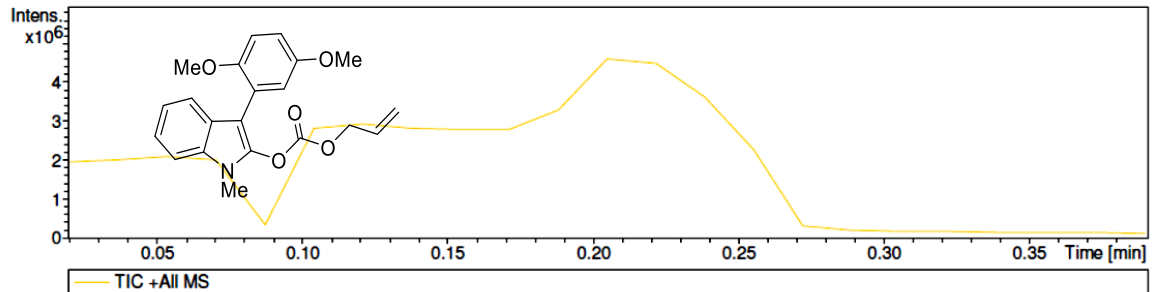
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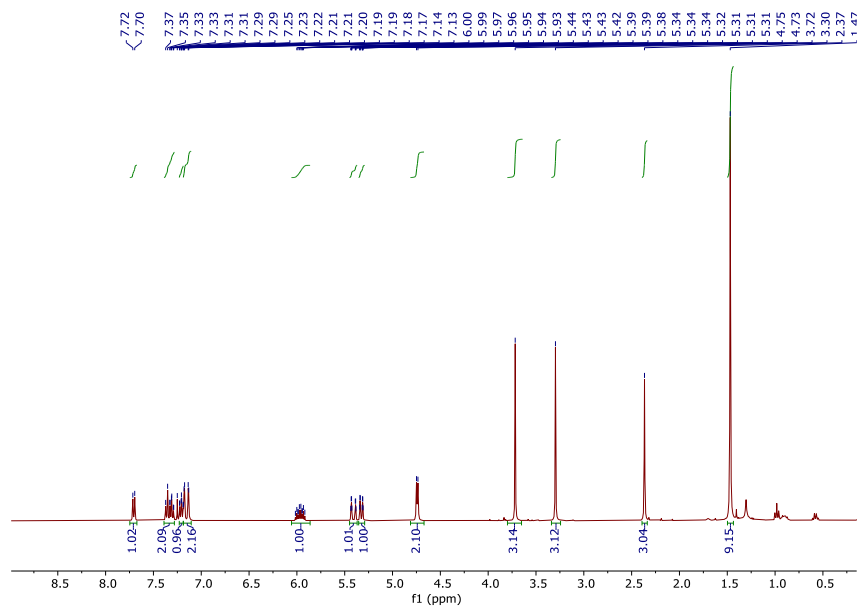
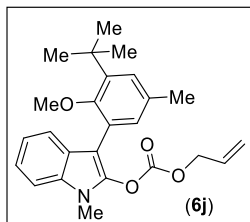
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Sample Name AB-KNB-05-46  
Comment

Acquisition Date 4/6/2018 3:40:34 PM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

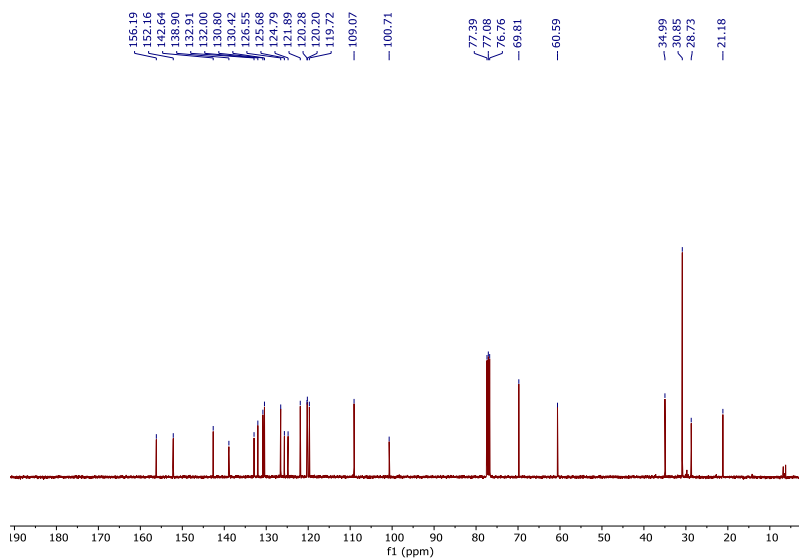
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste





<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) of compound **6j**



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of compound **6j**

## Display Report

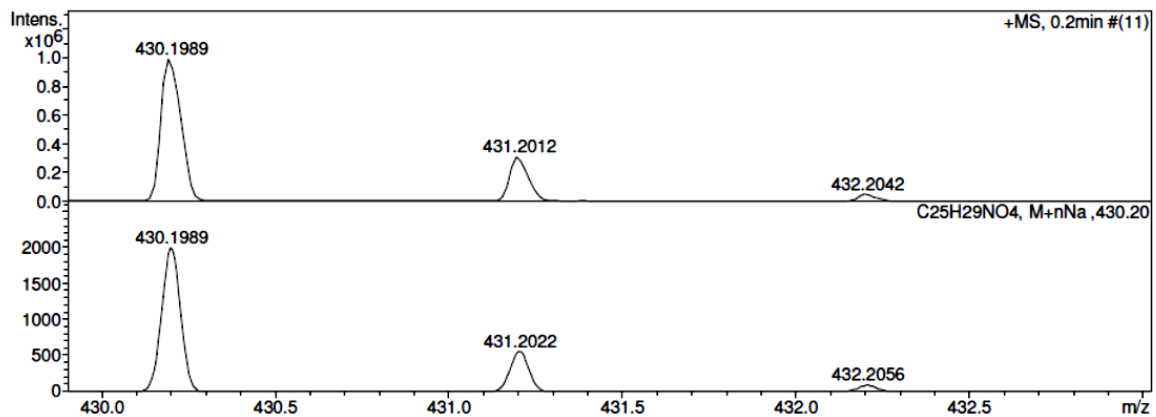
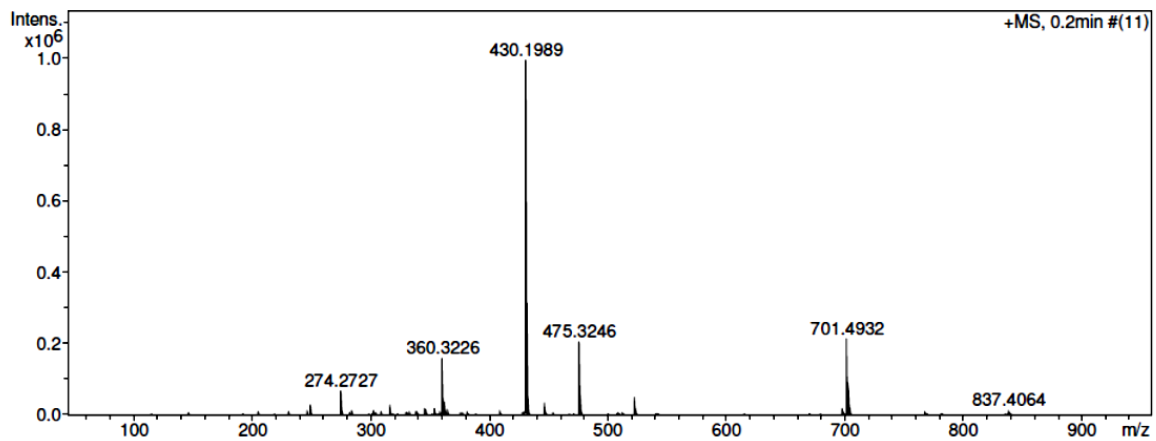
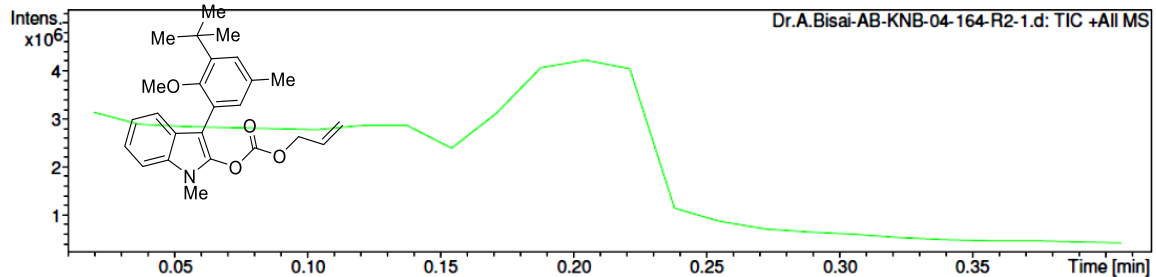
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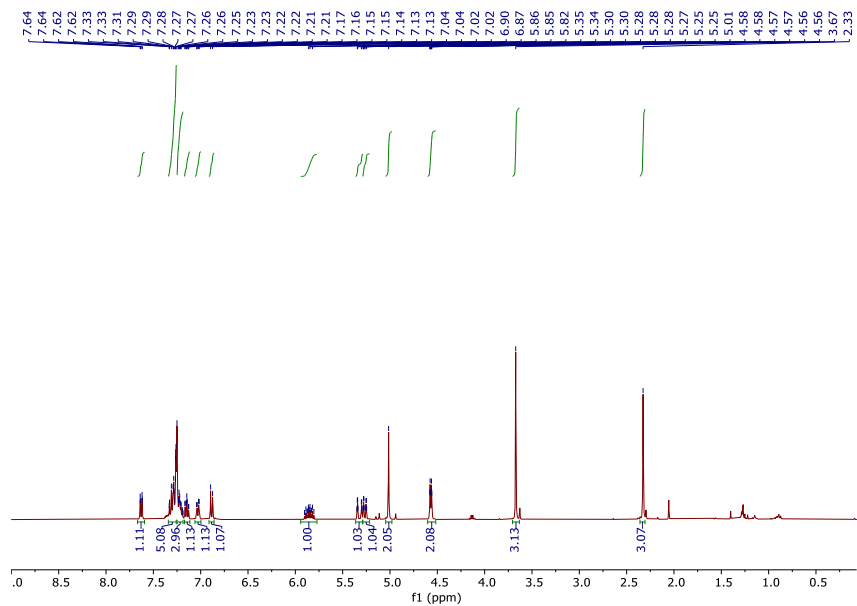
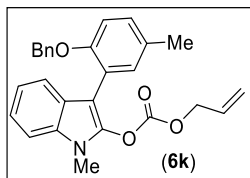
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 Comment

Acquisition Date 6/1/2018 4:23:26 PM  
 Operator RUCHI  
 Instrument micrOTOF-Q II 10330

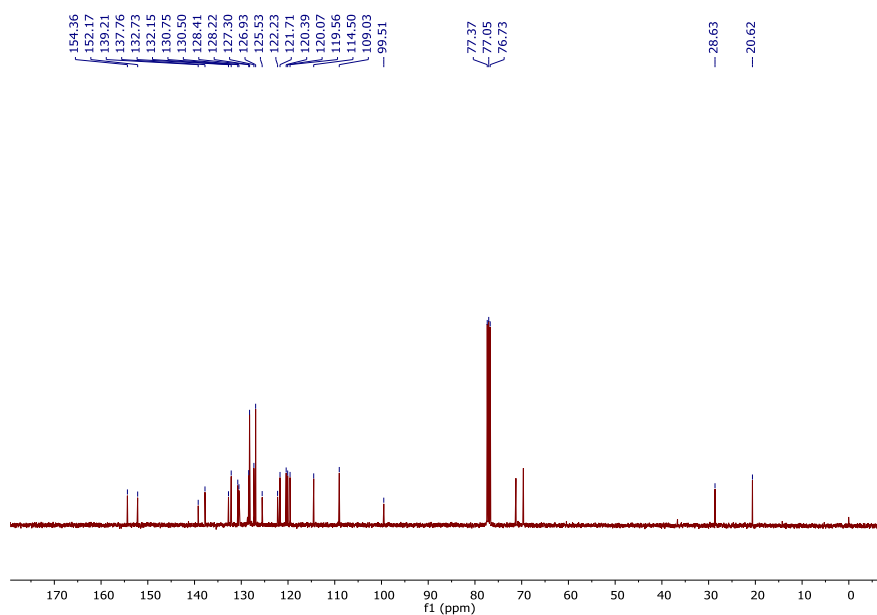
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste





<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) of compound **6k**



<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of compound **6k**

## Display Report

## Analysis Info

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Sample Name Dr.A.Bisai-AB-KNB-03-077  
Comment

Acquisition Date 5/5/2018 10:47:58 AM

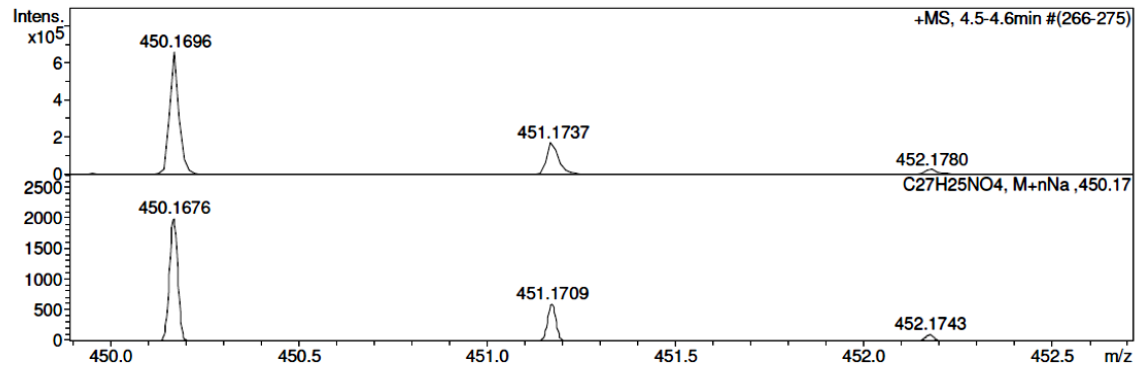
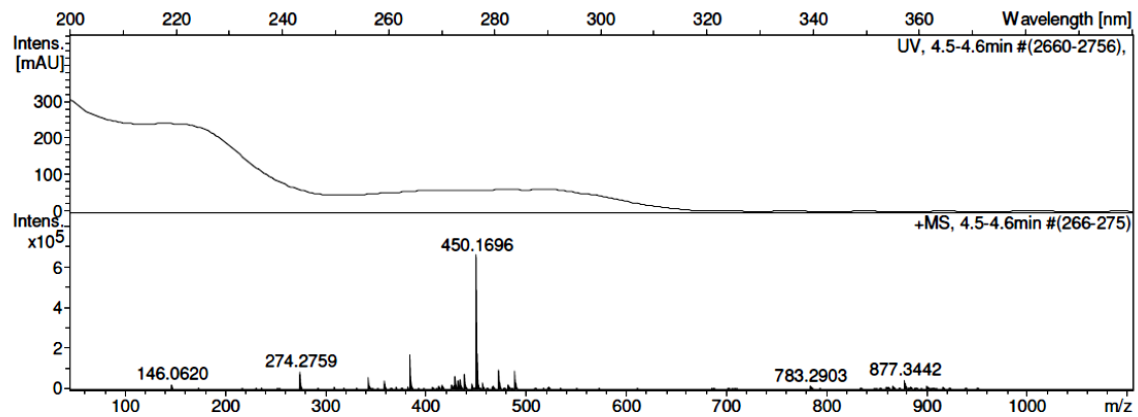
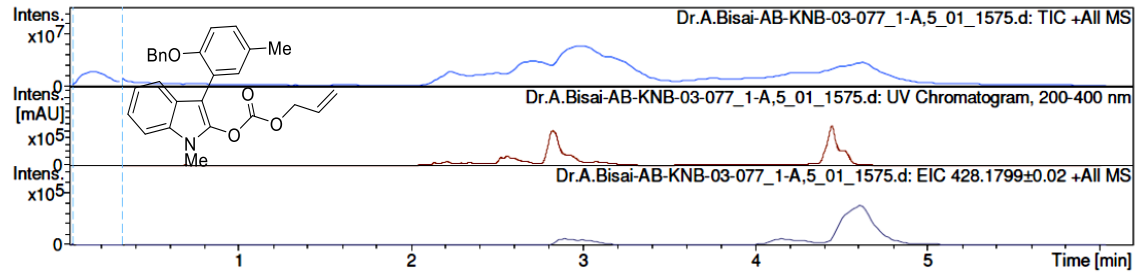
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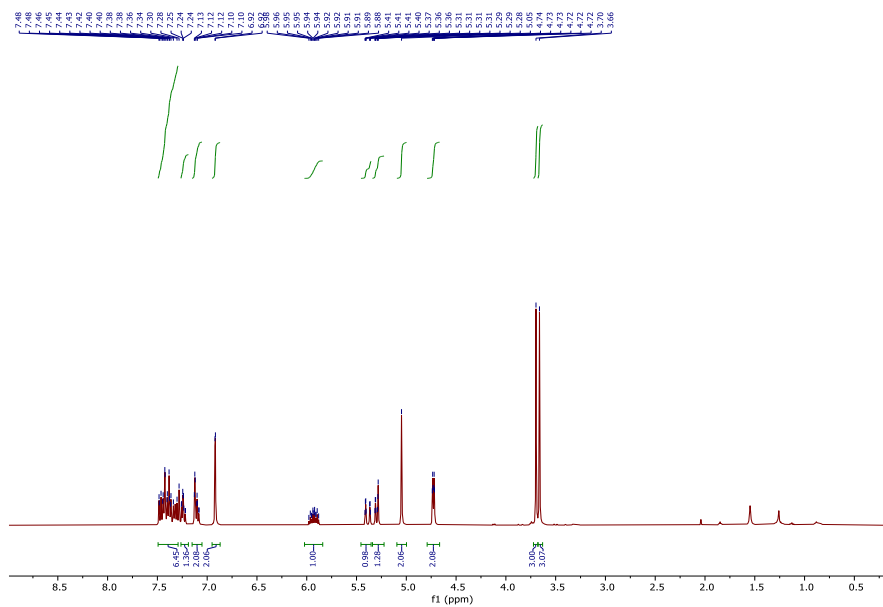
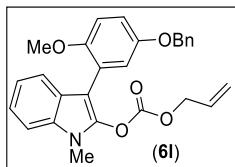
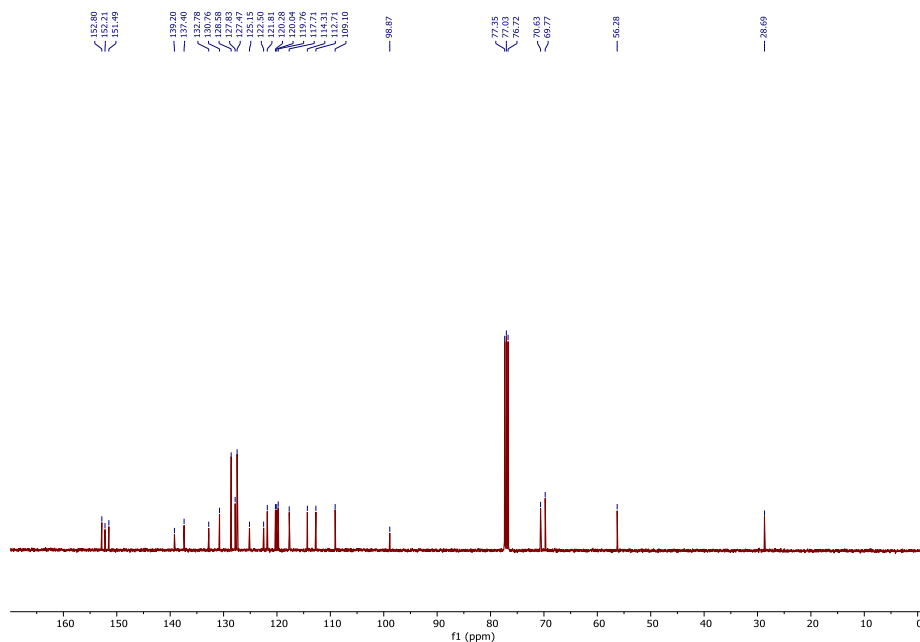
Operator RUCHI

Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **61** $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **61**

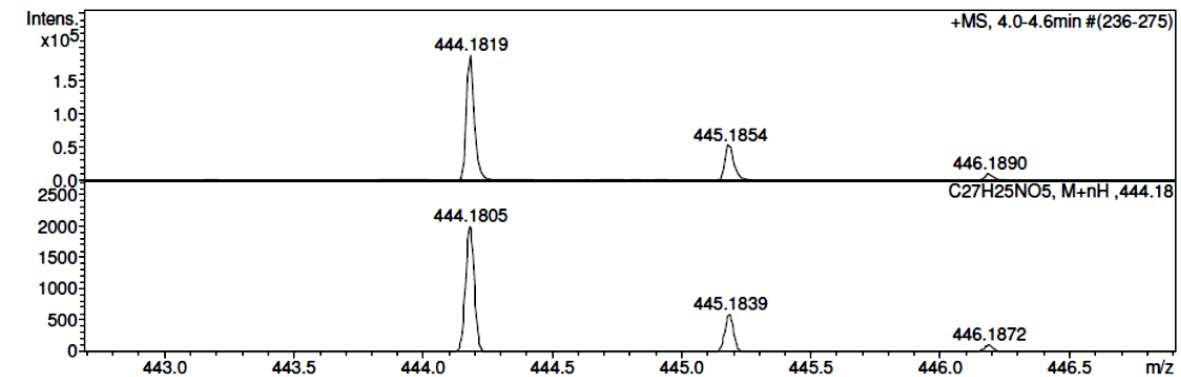
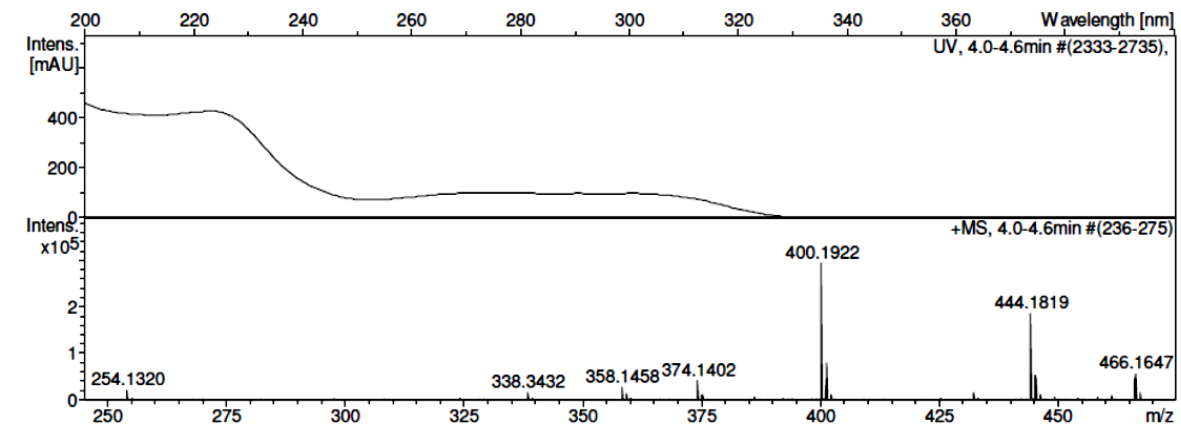
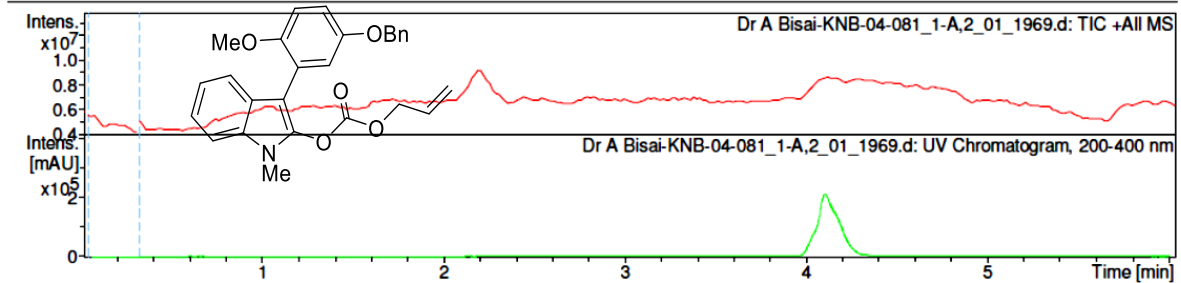
## Display Report

## Analysis Info

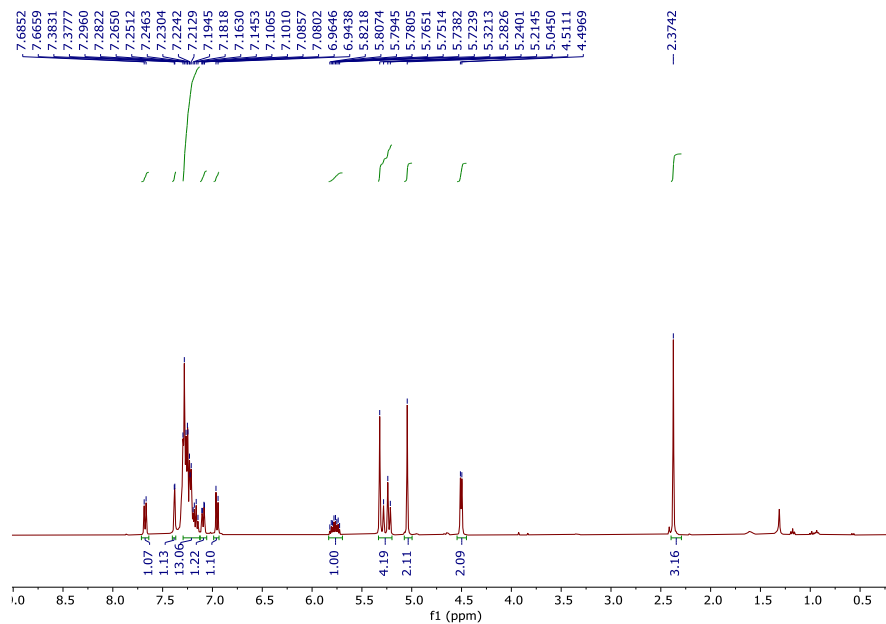
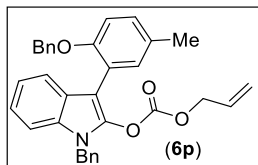
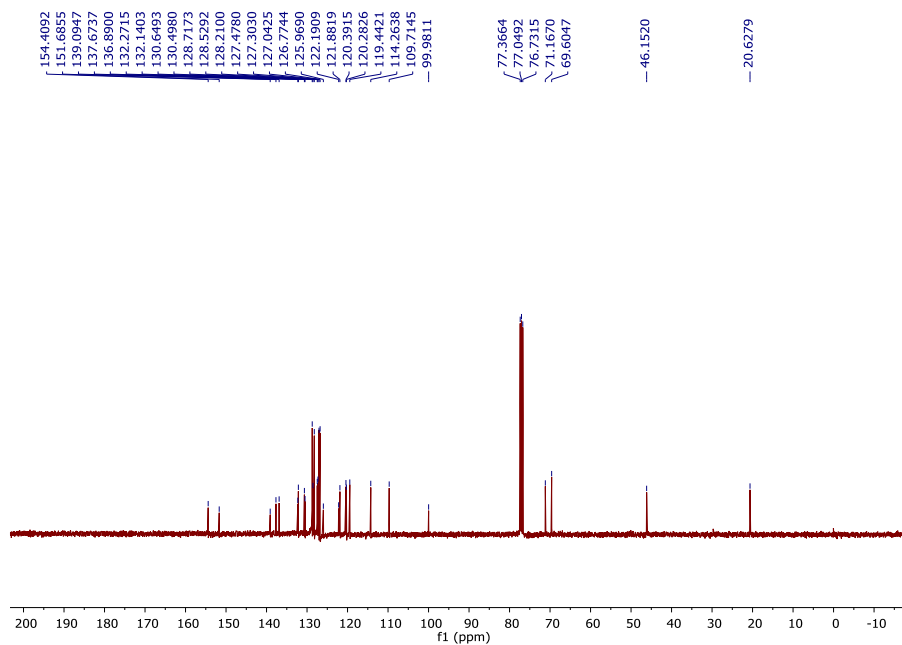
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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr A Bisai-KNB-04-081	Instrument	micrOTOF-Q II 10330
Comment			

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **6p** $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **6p**

## Display Report

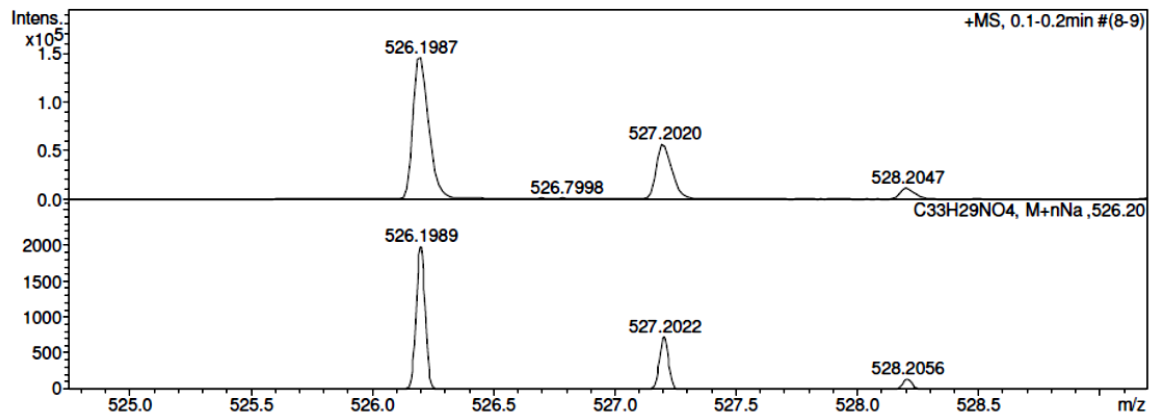
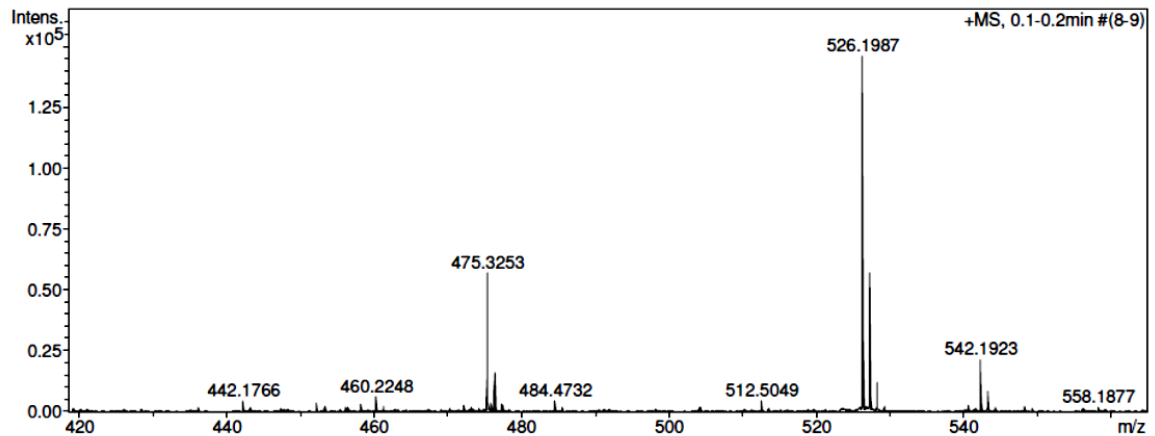
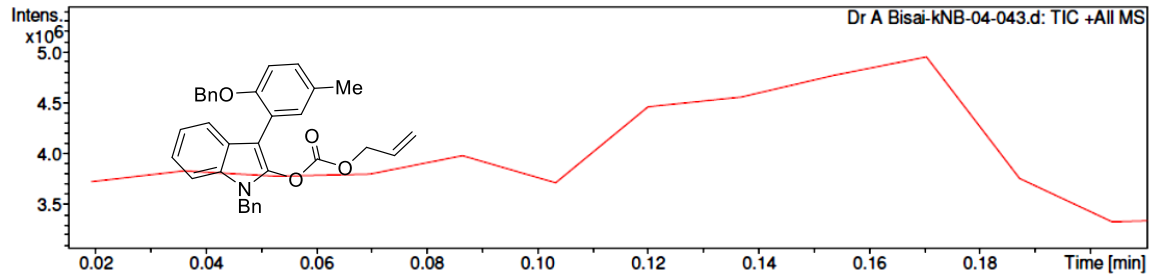
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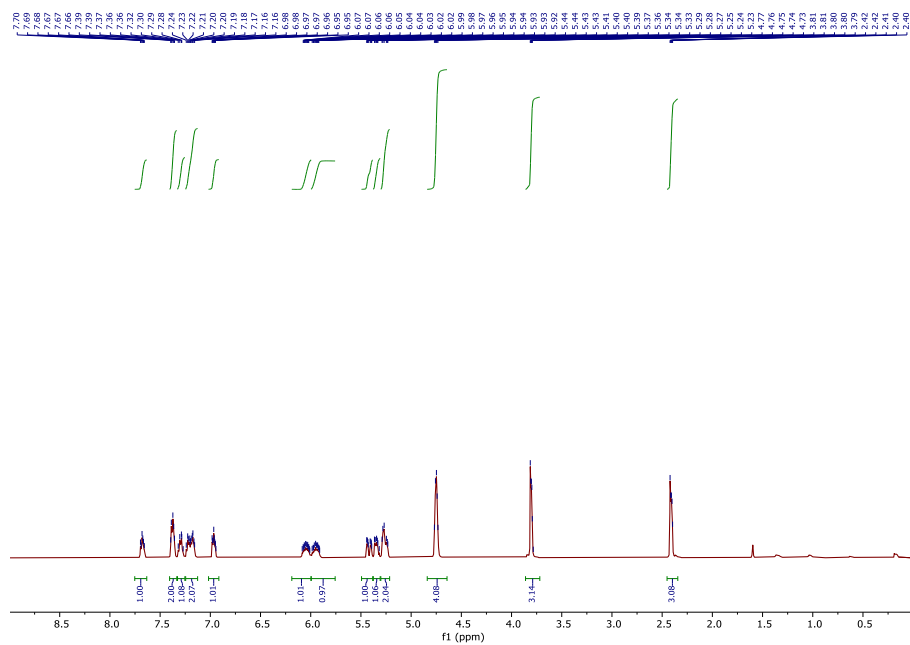
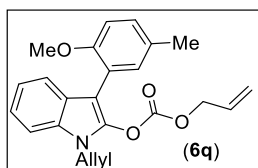
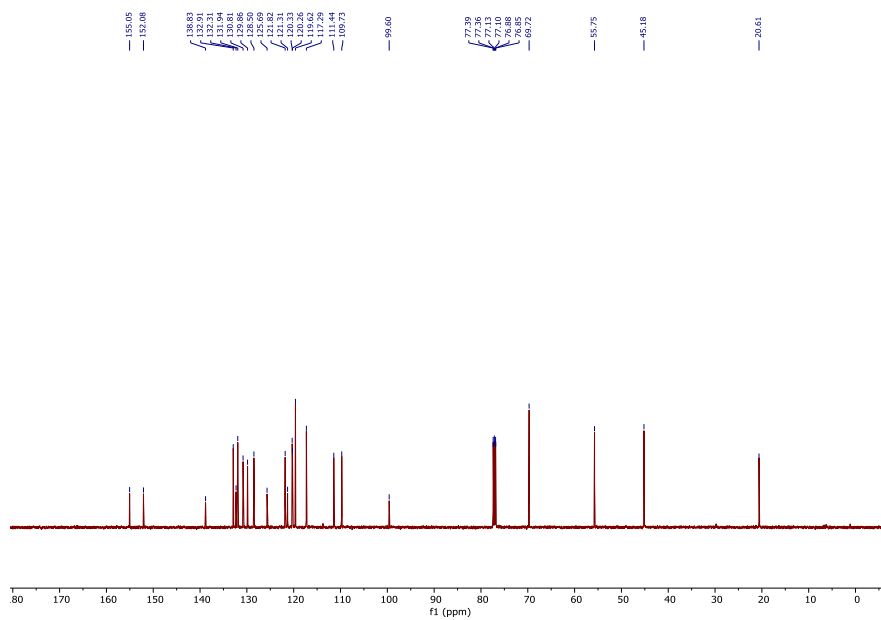
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Sample Name kNB-04-043  
Comment

Acquisition Date 5/24/2018 10:58:17 AM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) of compound 6q<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) of compound 6q

## Display Report

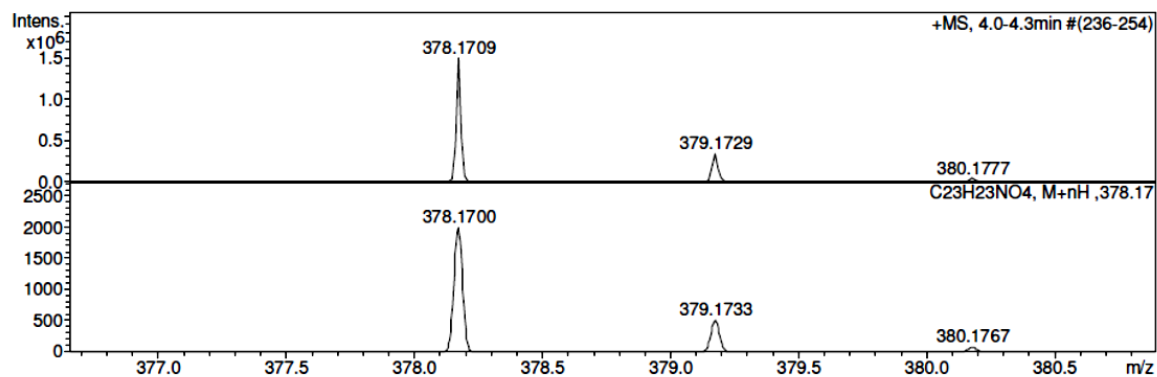
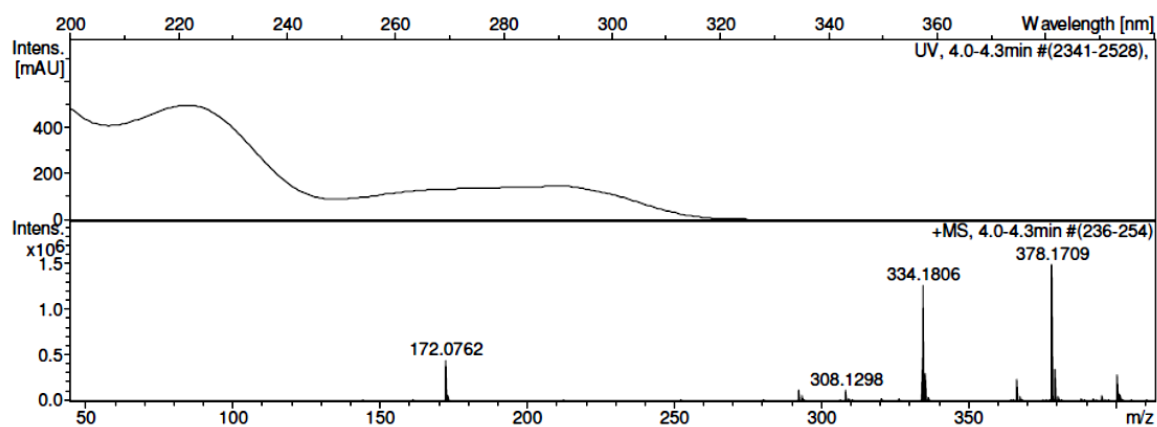
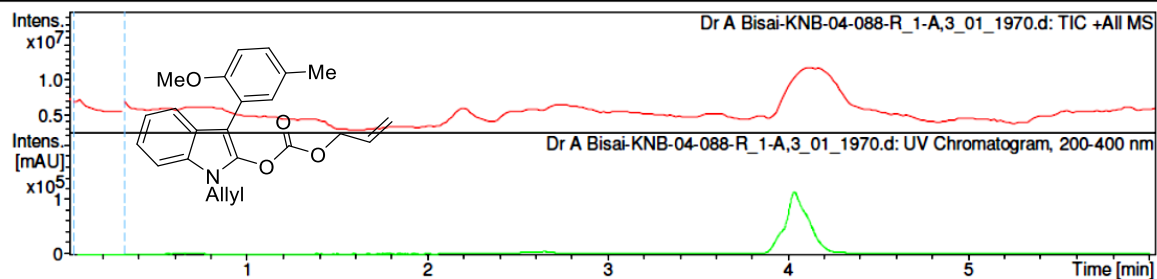
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Sample Name Dr A Bisai-KNB-04-088-R  
Comment

Acquisition Date 5/23/2018 10:58:09 AM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





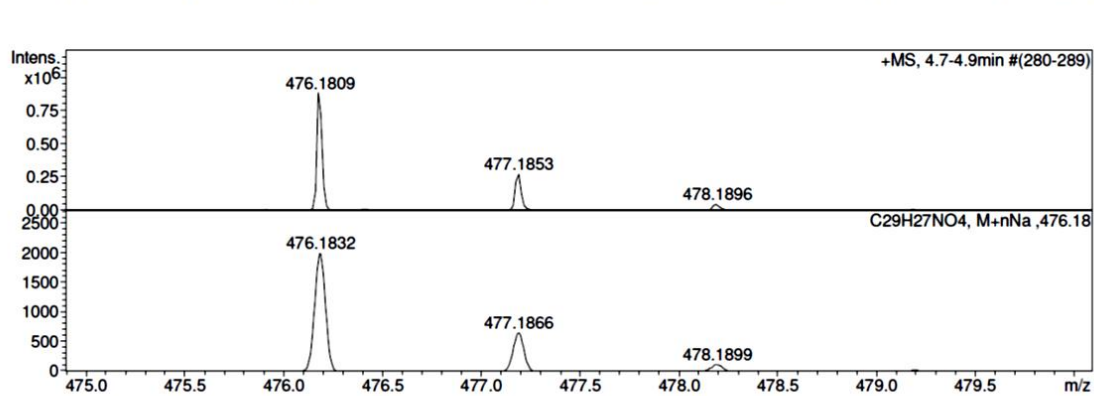
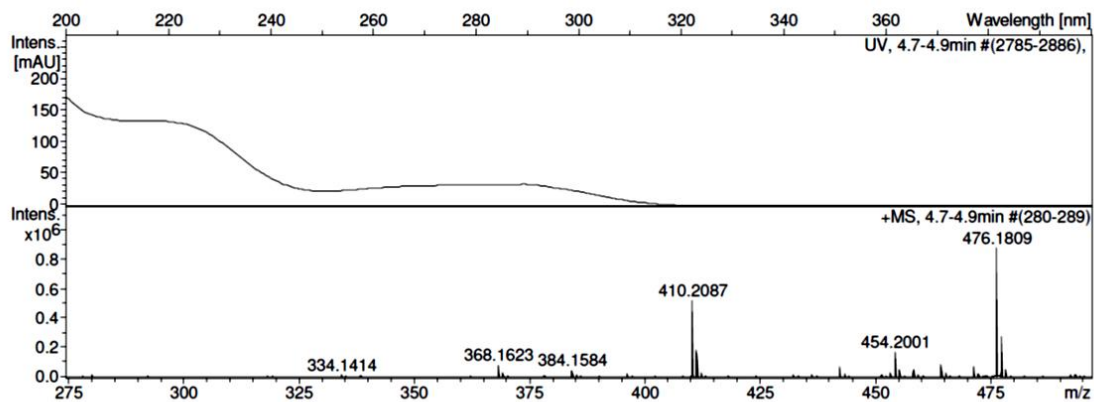
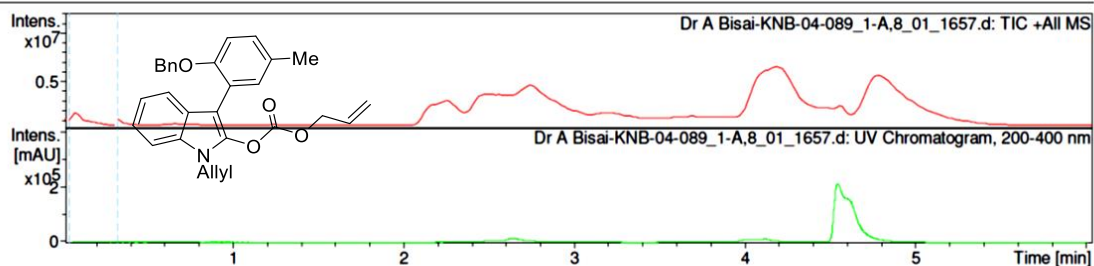
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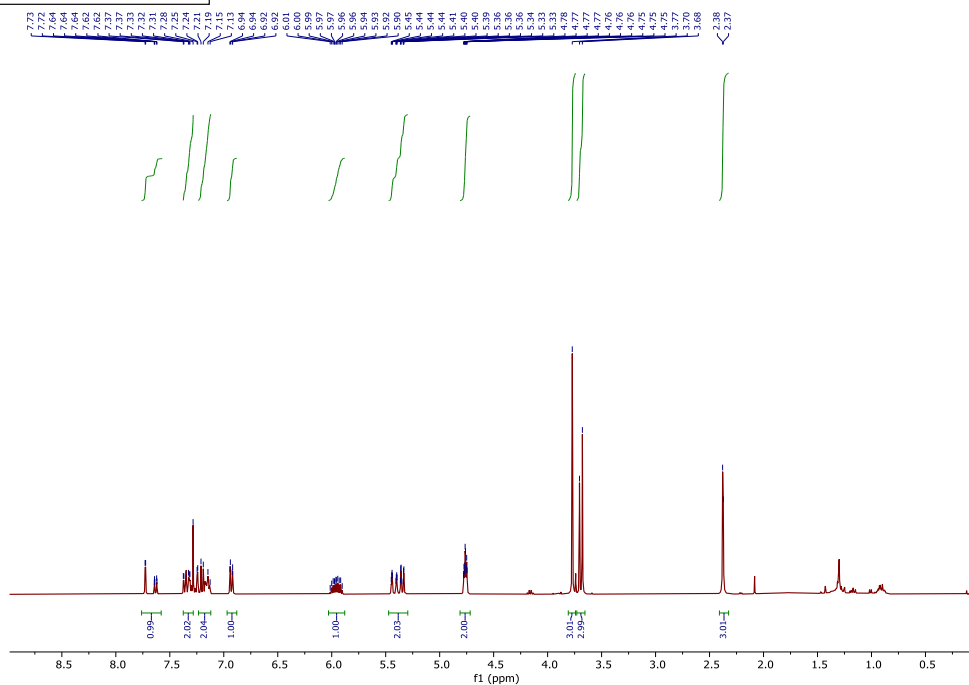
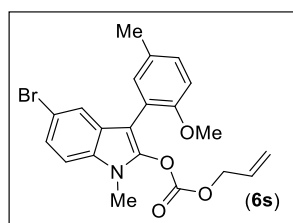
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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr A Bisai-KNB-04-089	Instrument	micrOTOF-Q II 10330
Comment			

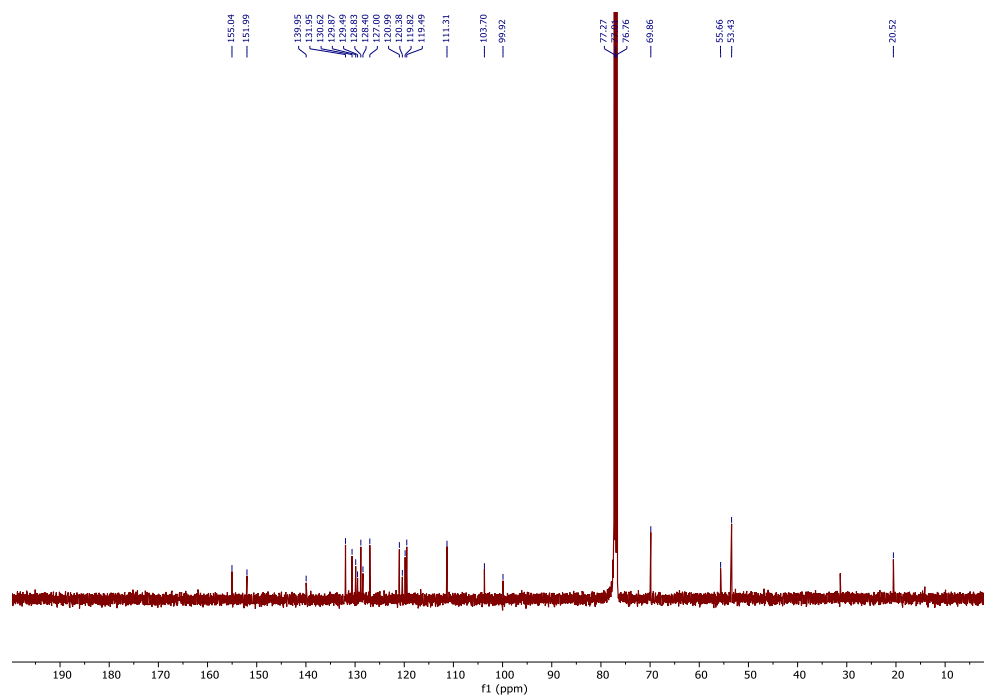
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Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **6r**



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6s**



$^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **6s**

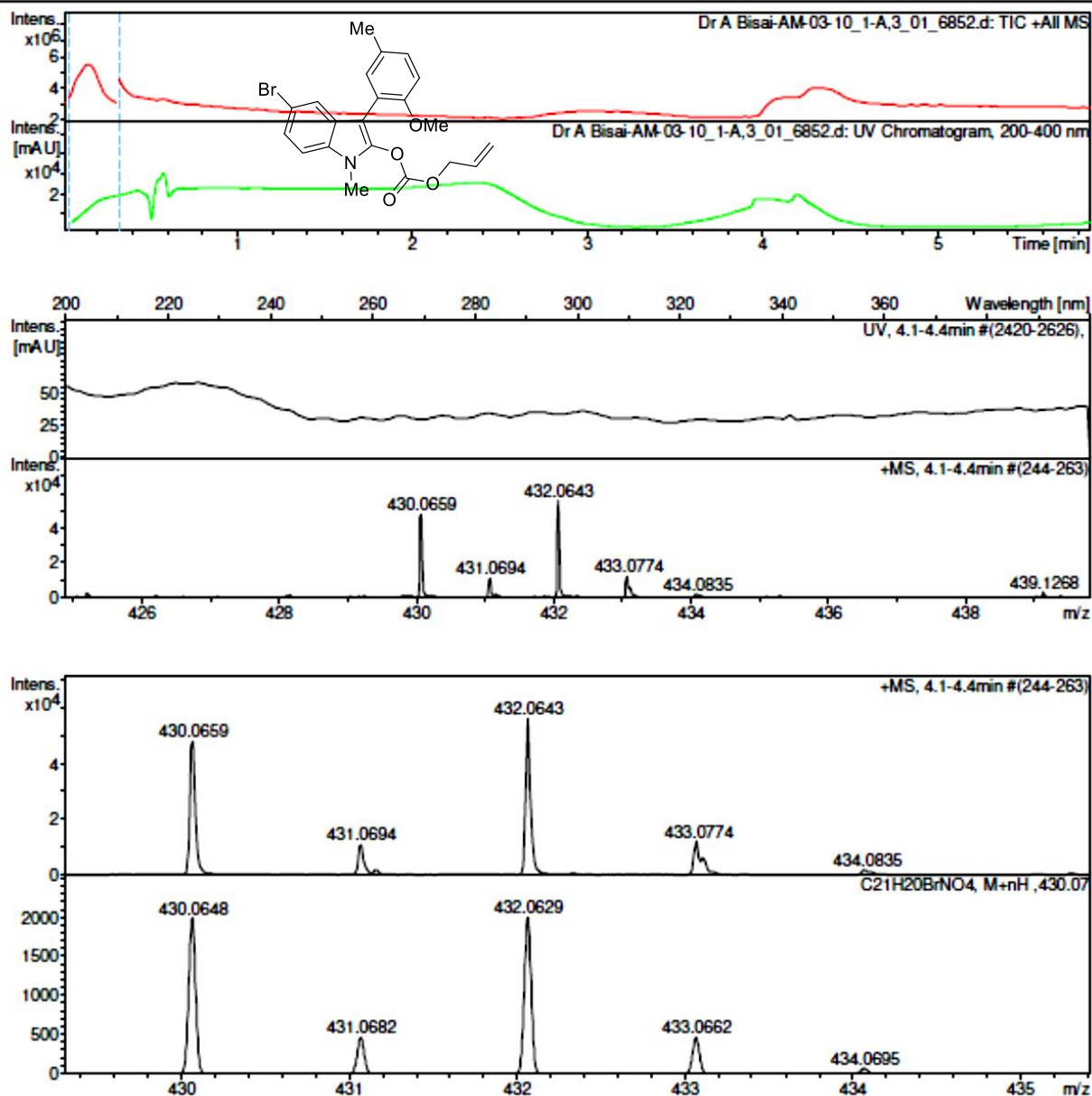
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**Analysis Info**

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Sample Name	Dr A Bisai-AM-03-10	Instrument	micrOTOF-Q II 10330
Comment			

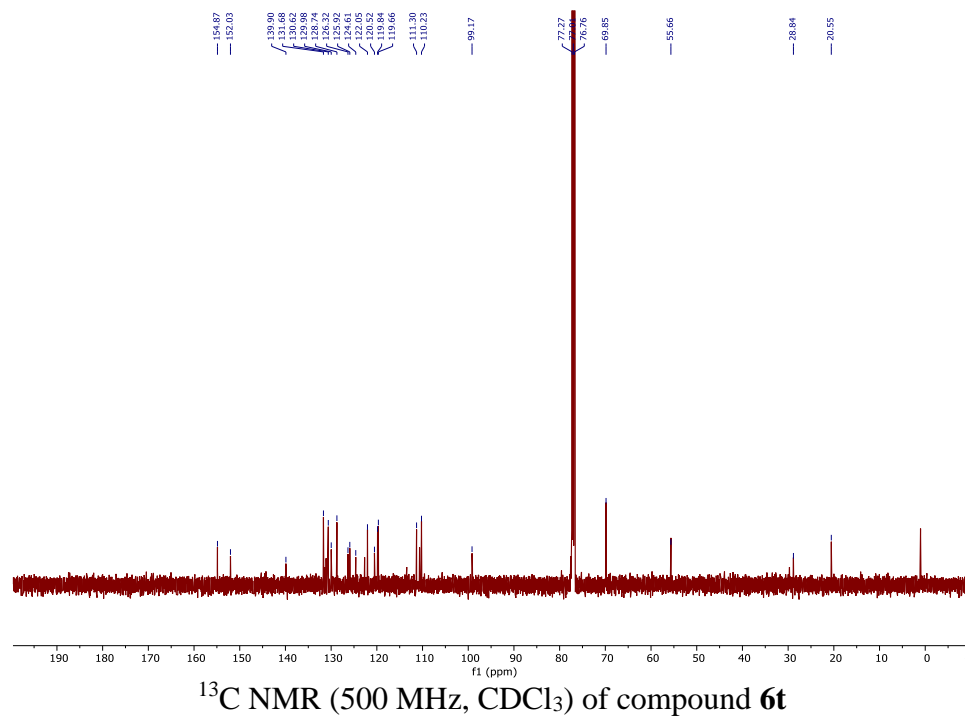
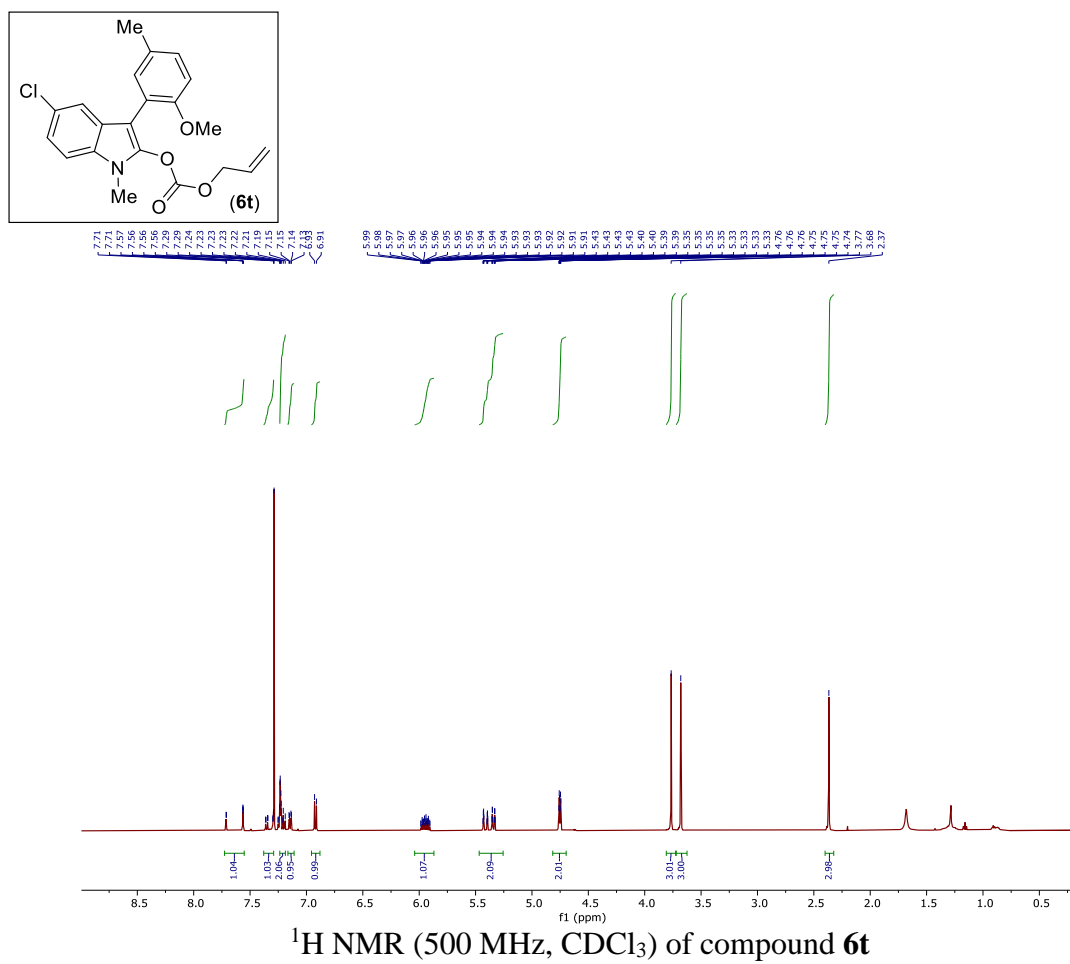
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of 6s



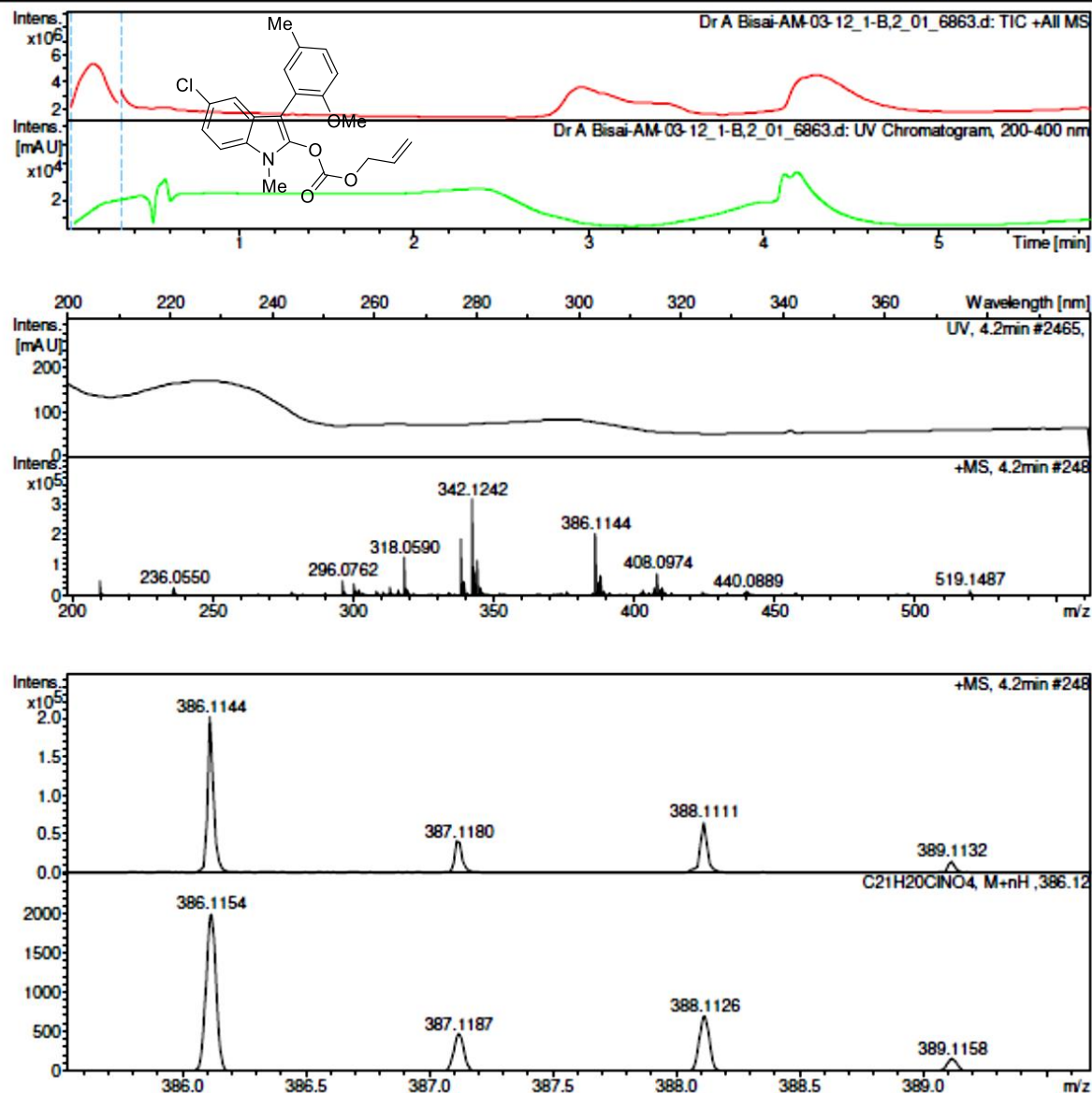


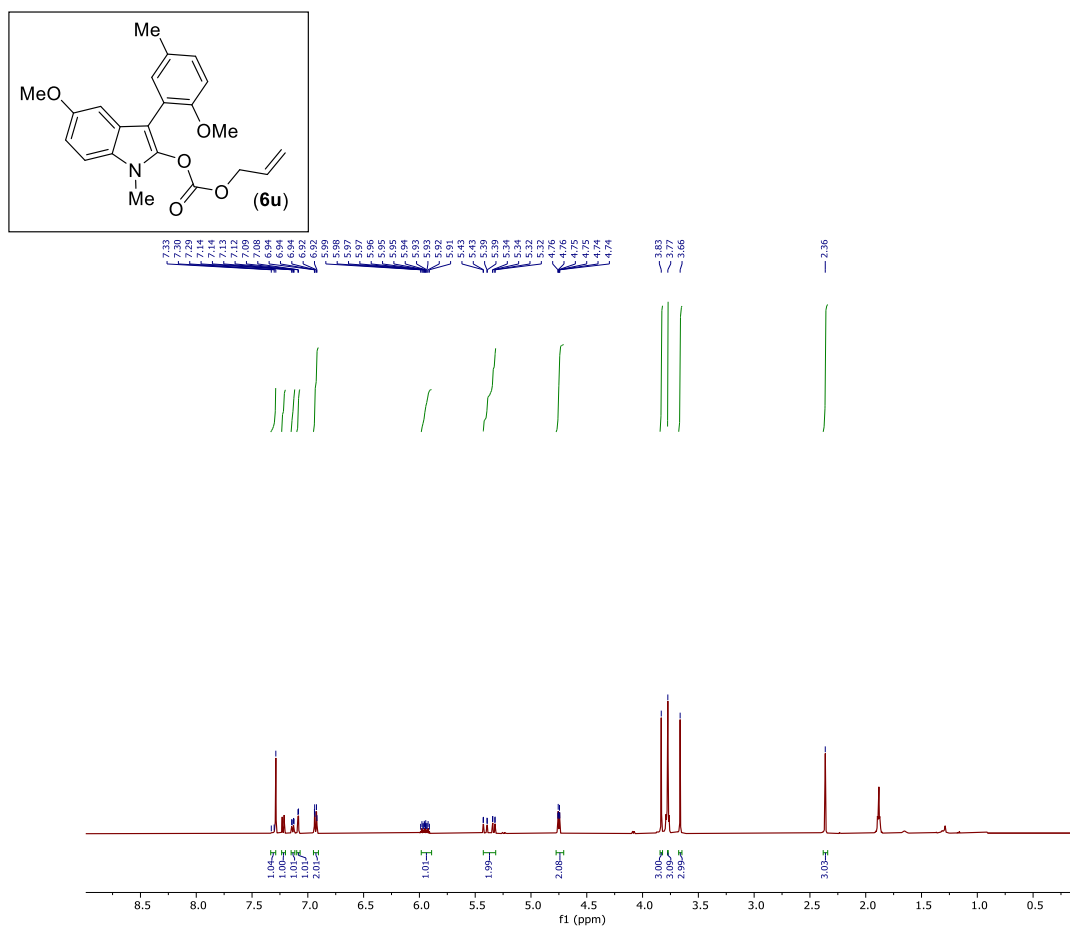
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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr A Bisai-AM-03-12	Instrument	micrOTOF-Q II 10330
Comment			

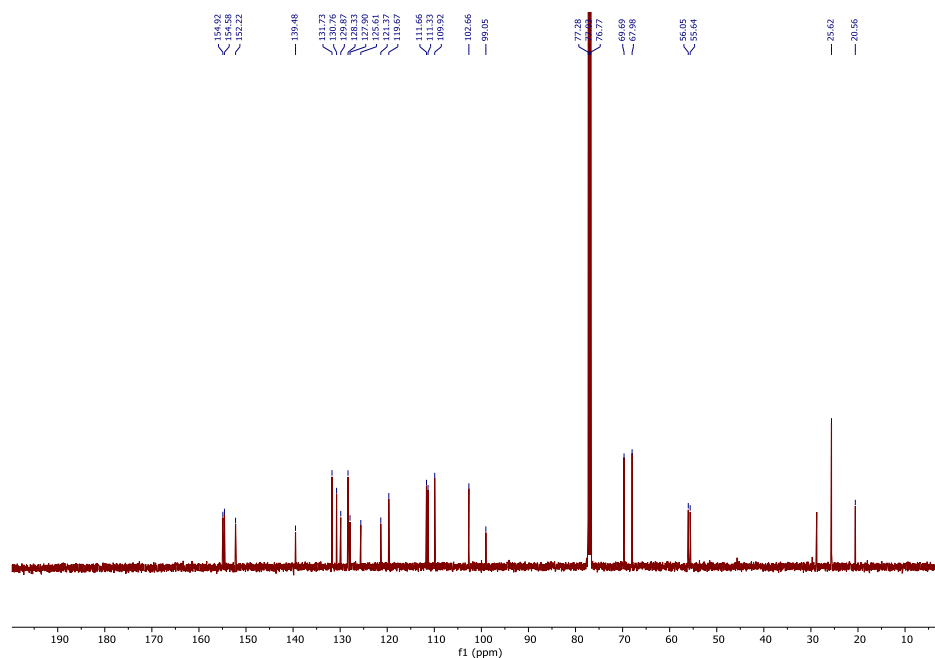
### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **6t**



**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) of compound **6u****



**<sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) of compound **6u****

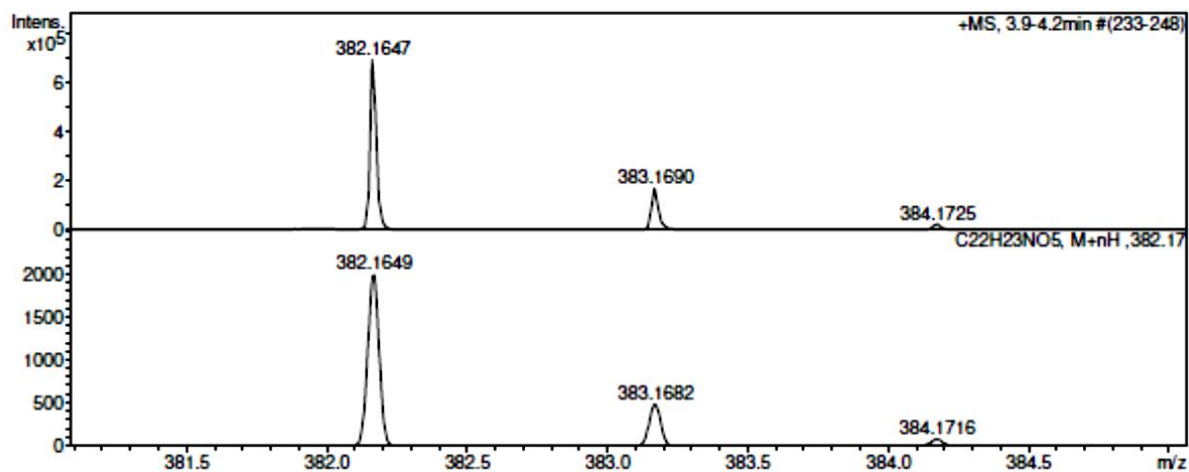
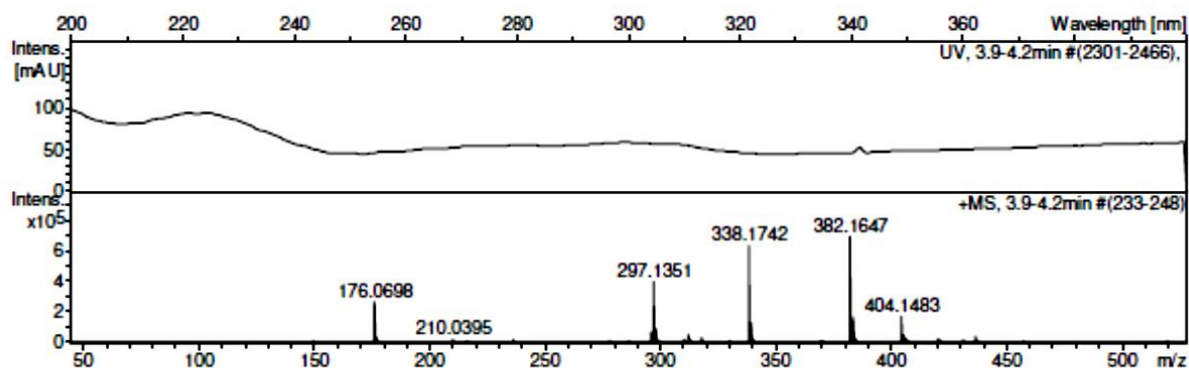
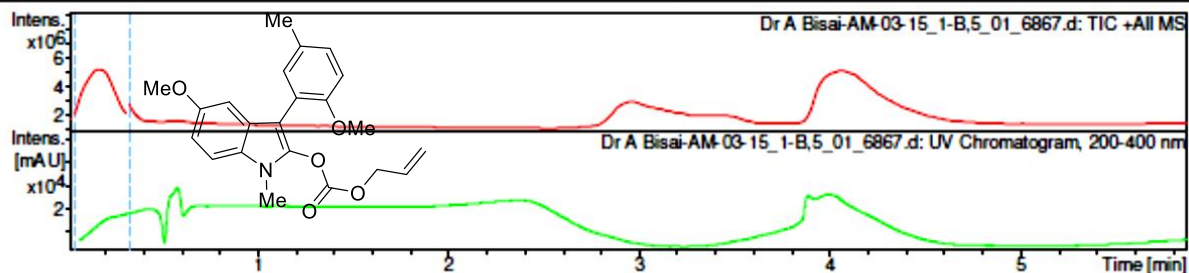
## Display Report

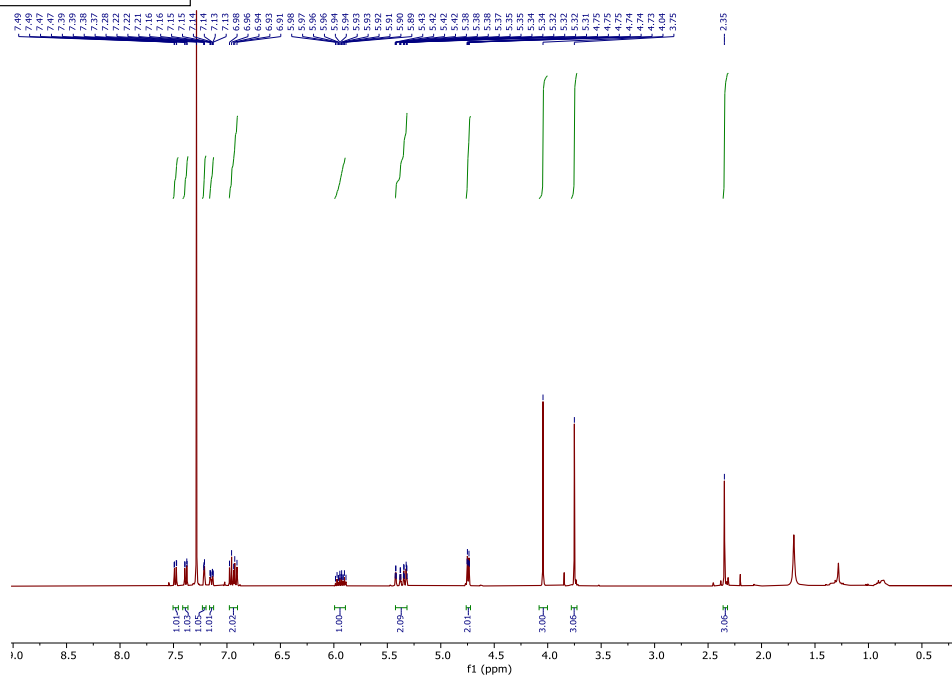
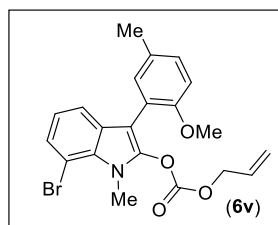
**Analysis Info**

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Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr A Bisai-AM-03-15	Instrument	micrOTOF-Q II 10330
Comment			

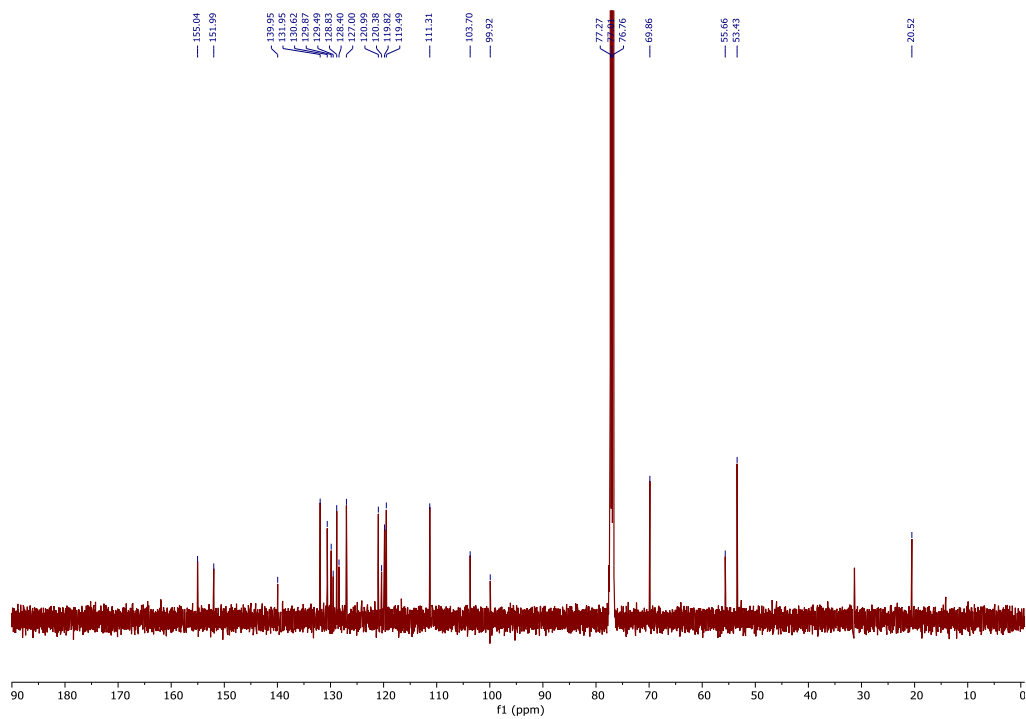
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **6u**



<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) of compound **6v**



<sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) of compound **6v**

## Display Report

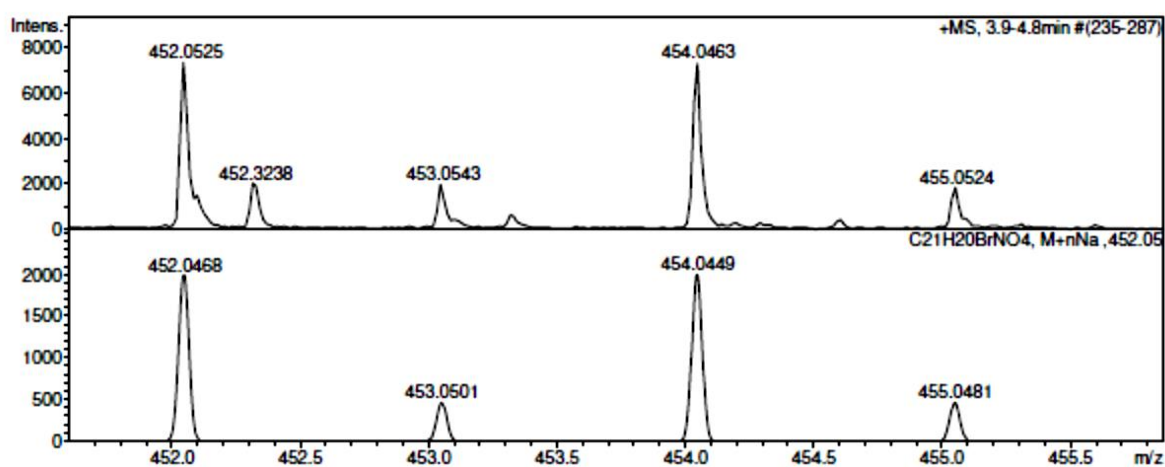
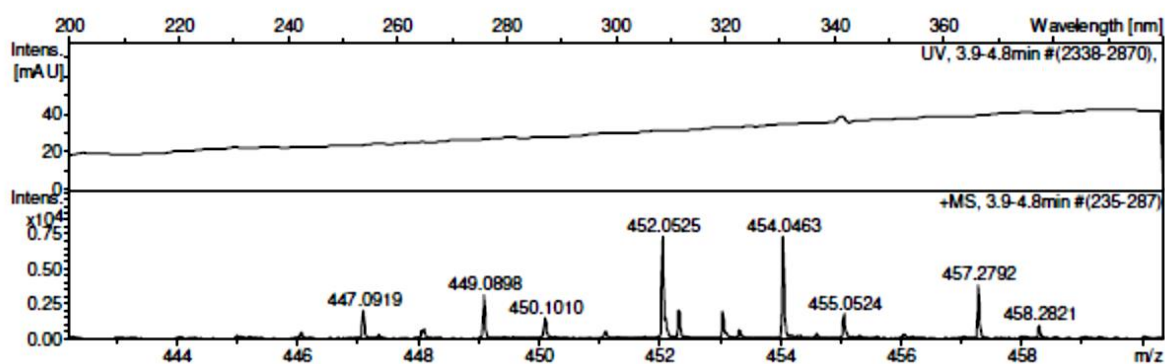
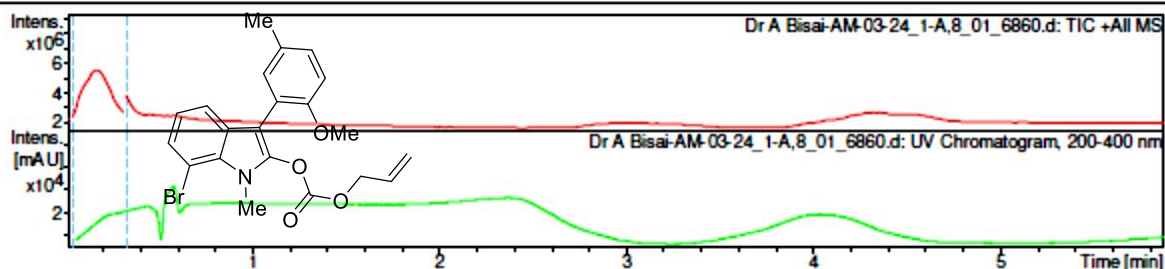
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Sample Name Dr A Bisai-AM-03-24  
Comment

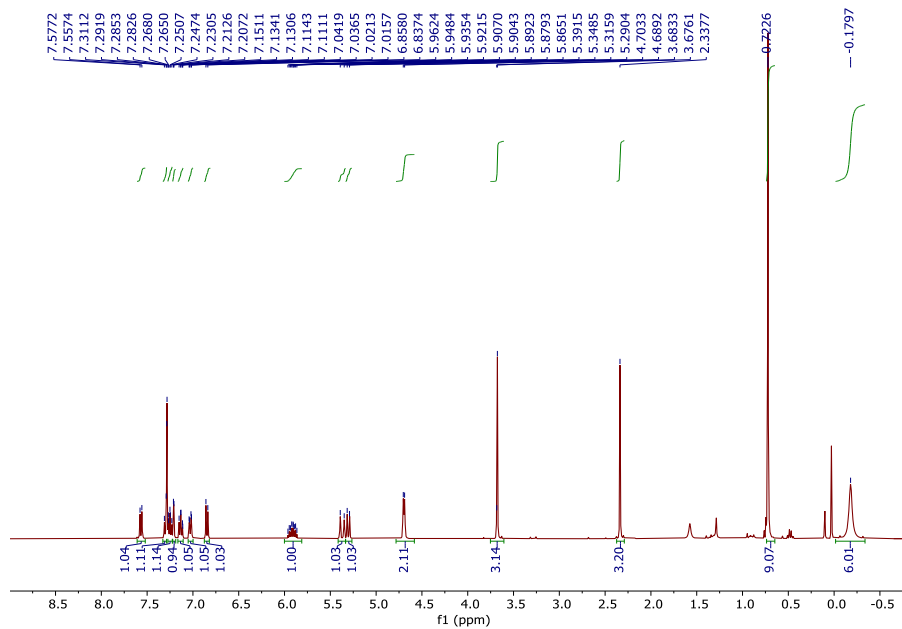
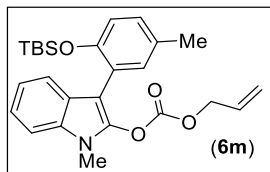
Acquisition Date 6/10/2019 11:16:43 AM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

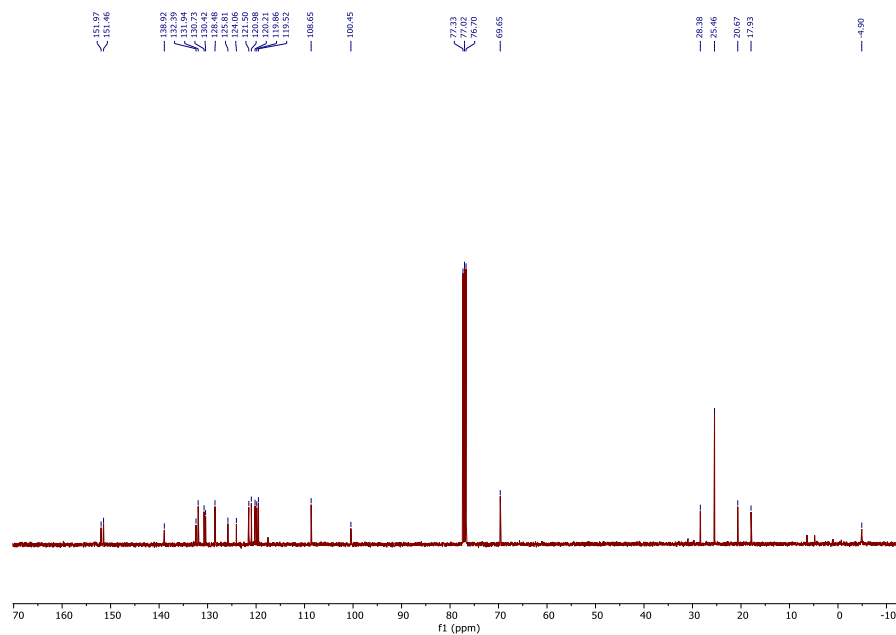
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of 6v



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound **6m**



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound **6m**

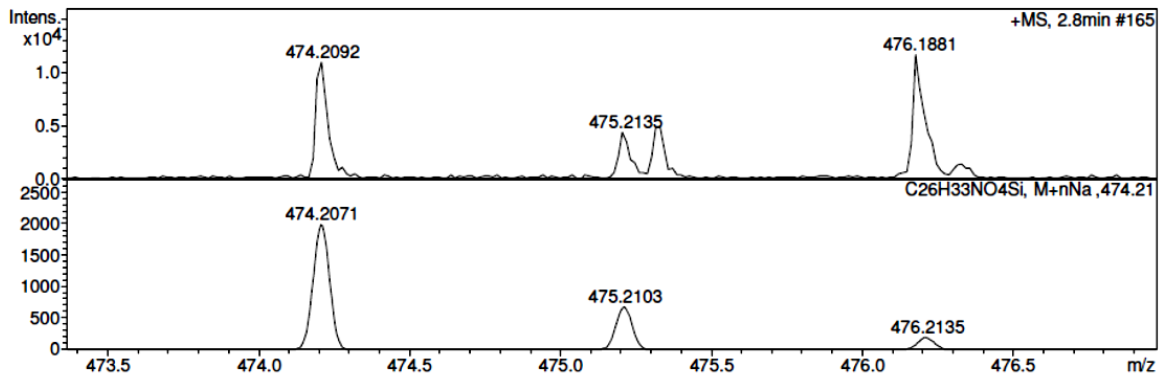
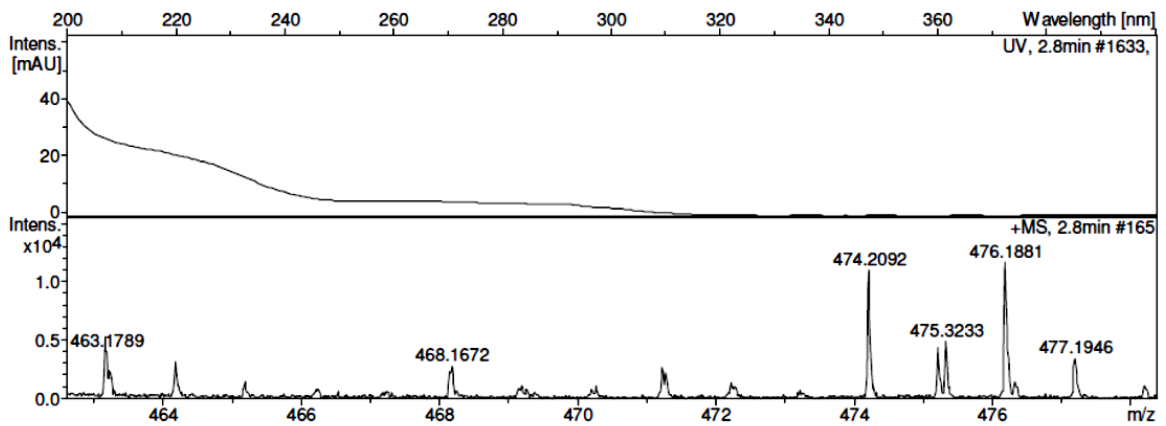
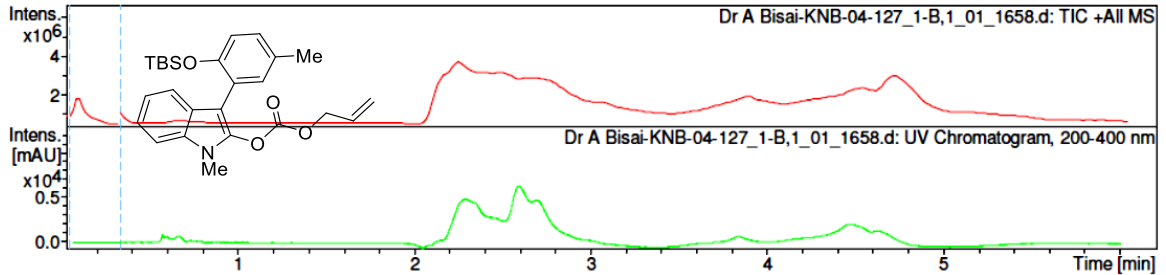
## Display Report

**Analysis Info**

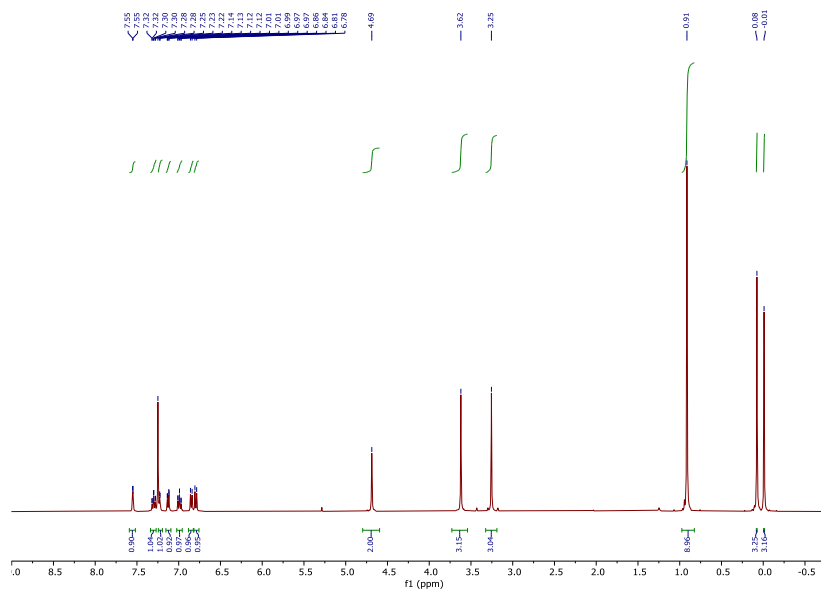
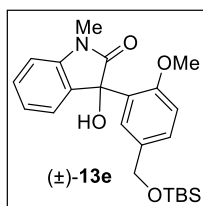
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Method	hrlcms-20 sept.m
Sample Name	Dr A Bisai-KNB-04-127
Comment	
Acquisition Date	5/9/2018 12:19:19 PM
Operator	RUCHI
Instrument	micrOTOF-Q II 10330

**Acquisition Parameter**

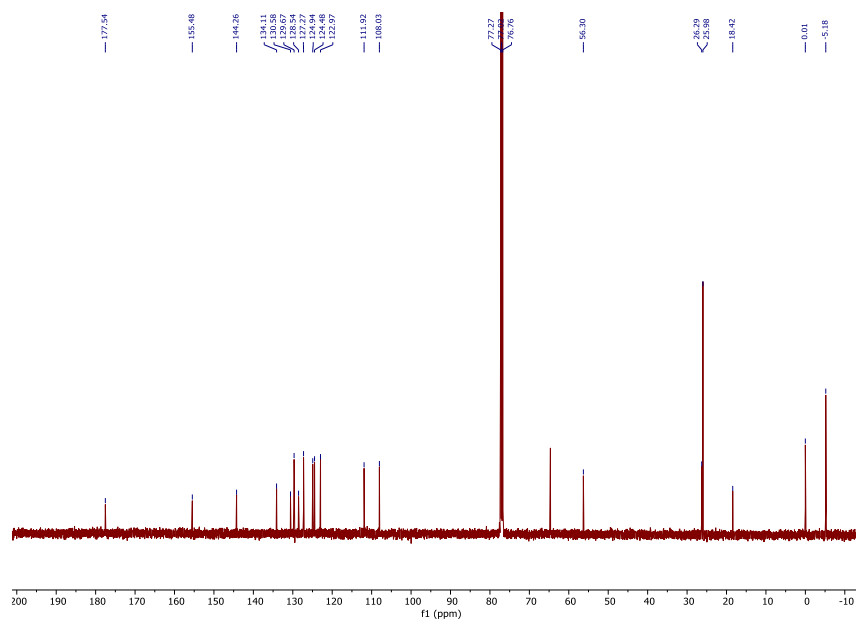
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound (±)-**13e**



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound (±)-**13e**

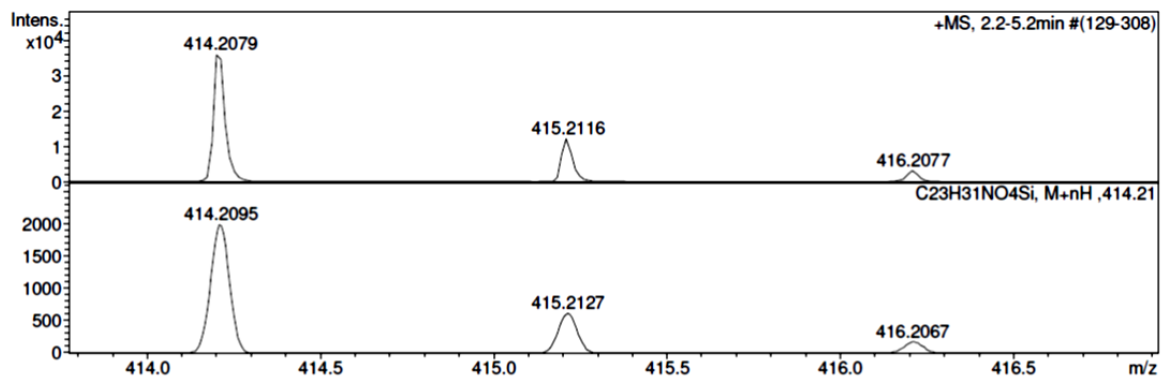
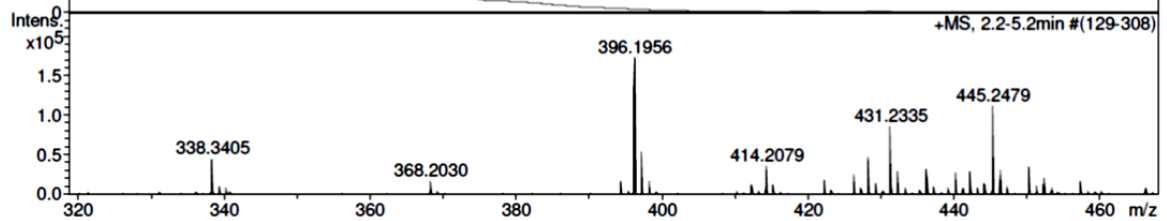
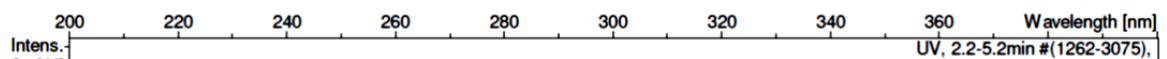
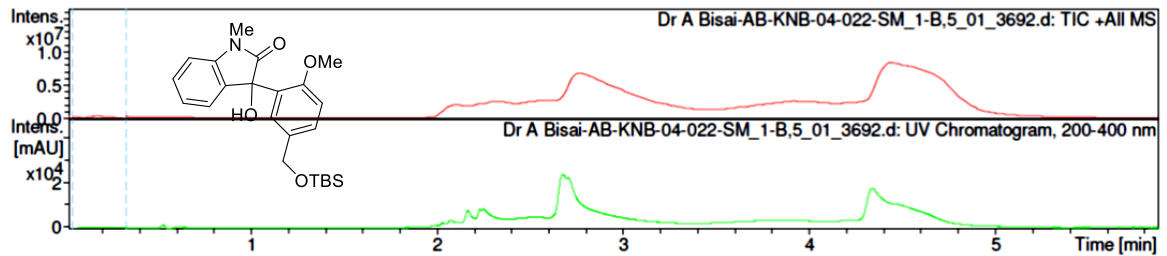
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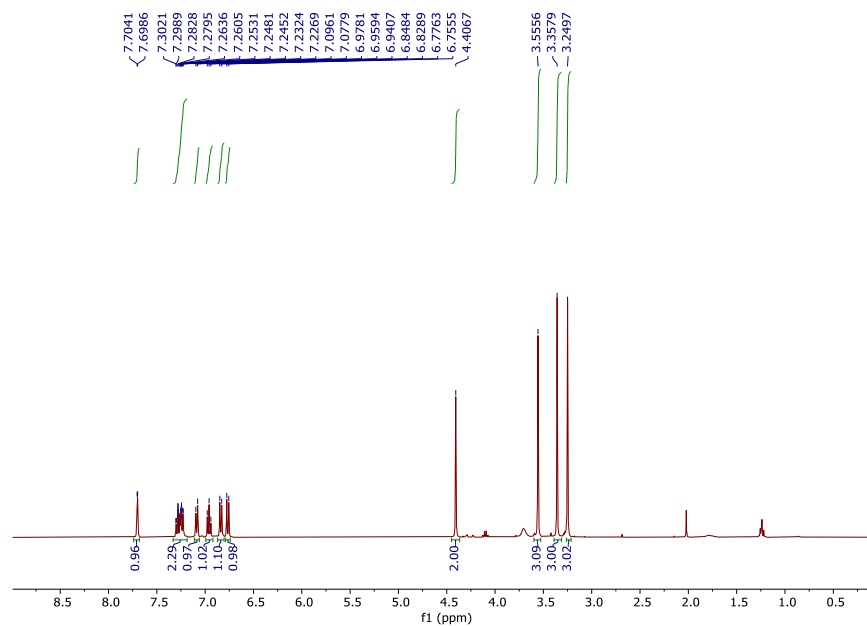
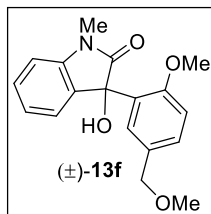
## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\05 oct18\Dr A  
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 Sample Name Dr A Bisai-AB-KNB-04-022-SM Instrument RUCHI  
 Comment micrOTOF-Q II 10330

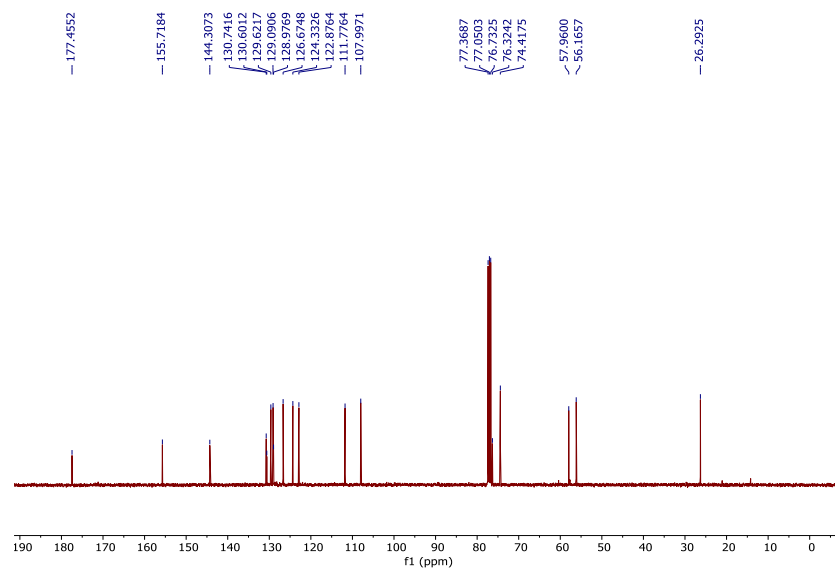
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound (±)-**13f**



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound (±)-**13f**

## Display Report

## Analysis Info

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 Method hrlcms-20 sept.m  
 Sample Name Dr A Bisai-AB-KNB-04-028  
 Comment

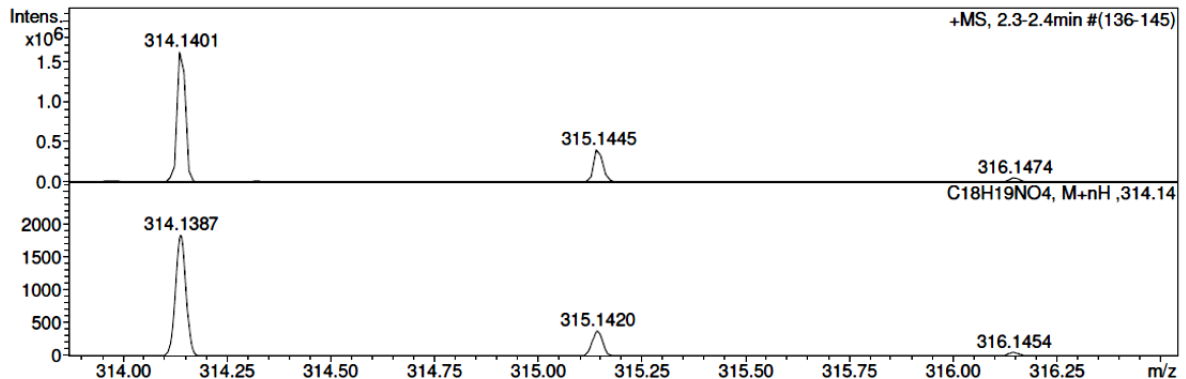
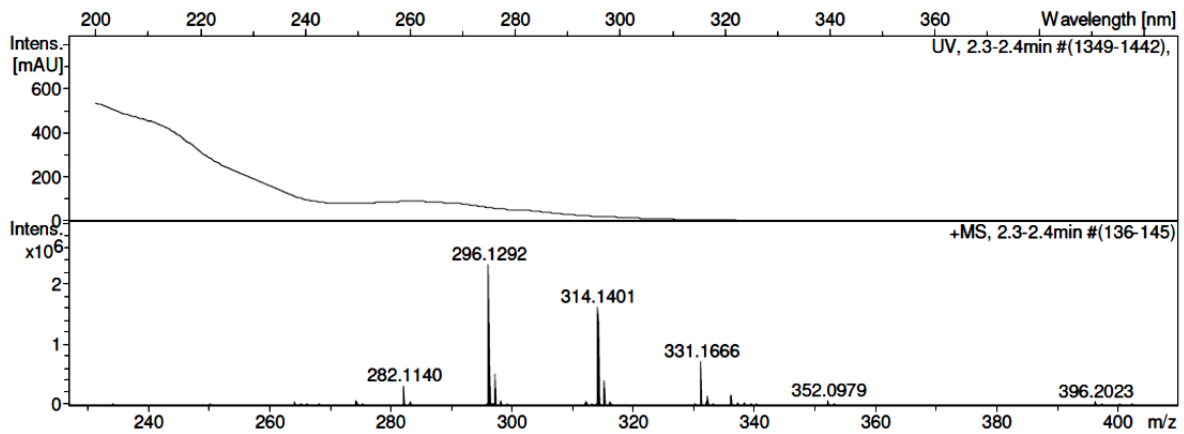
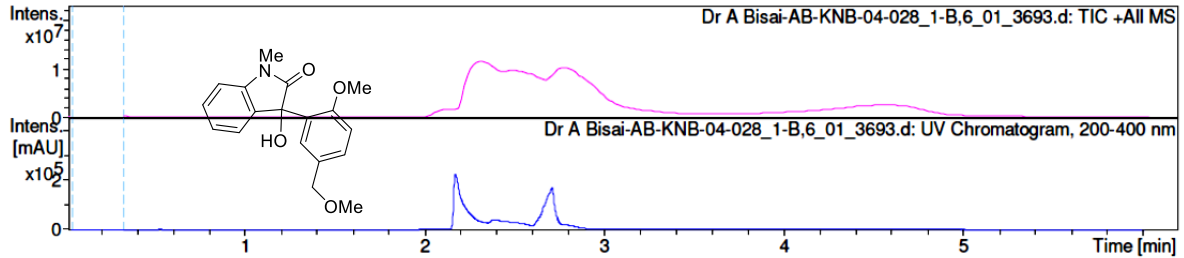
Acquisition Date 10/5/2018 1:08:19 PM

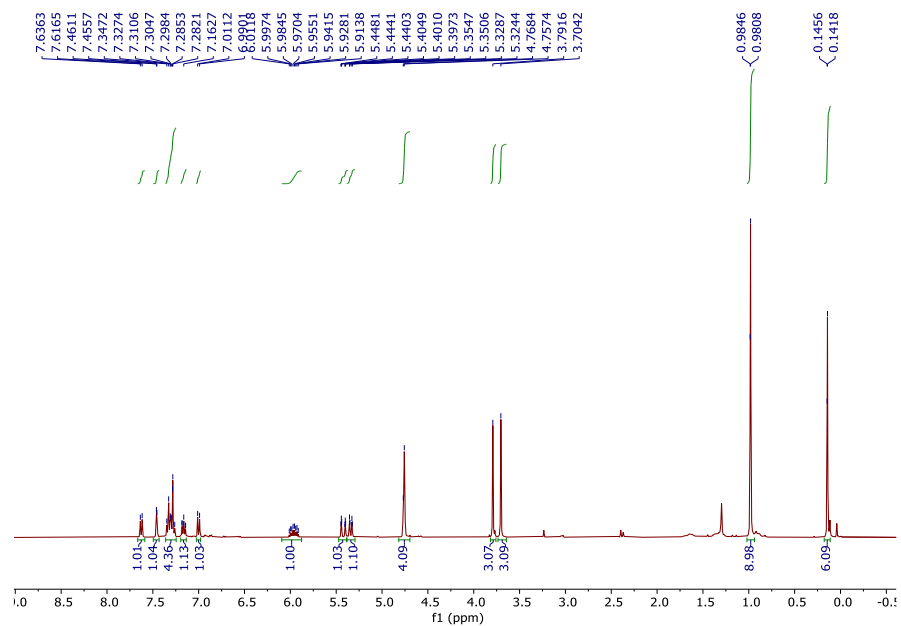
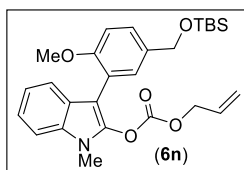
Operator RUCHI

Instrument micrOTOF-Q II 10330

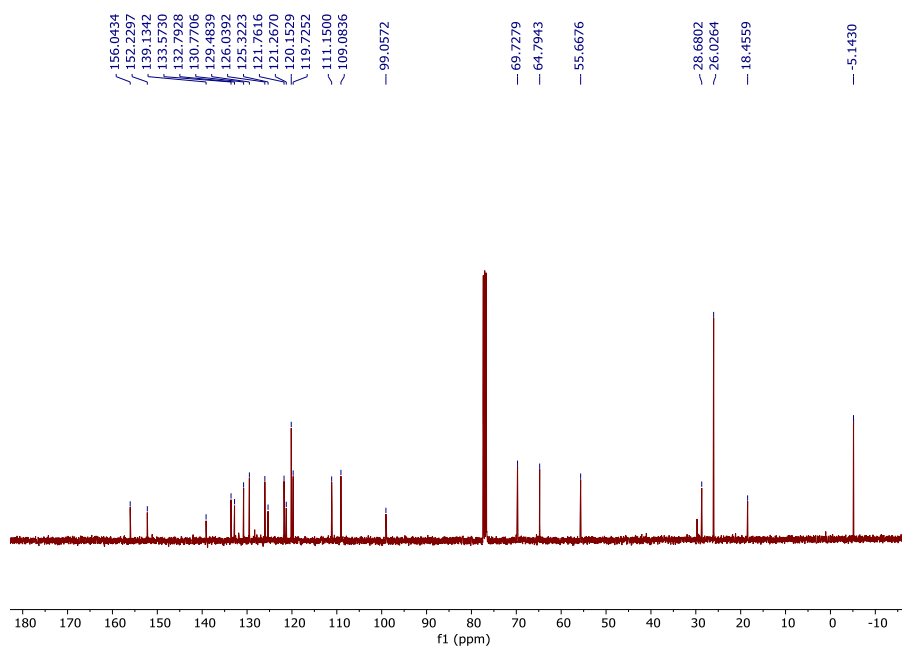
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **6n**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **6n**

## Display Report

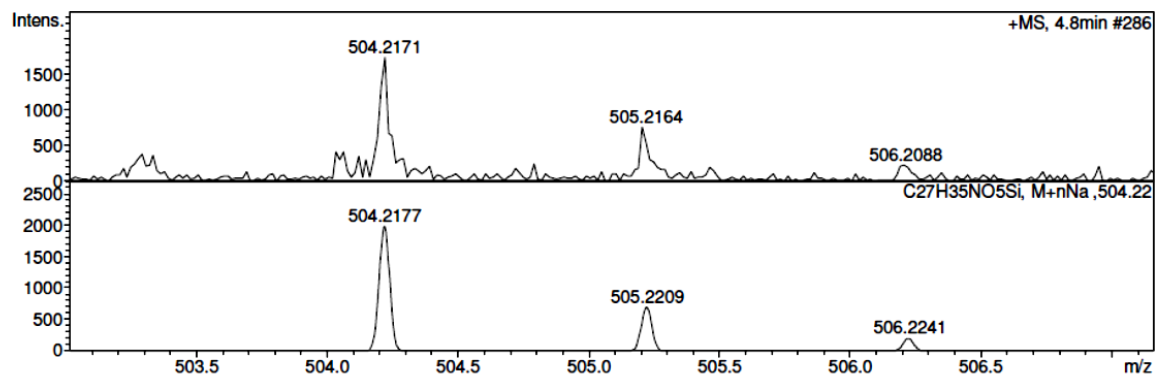
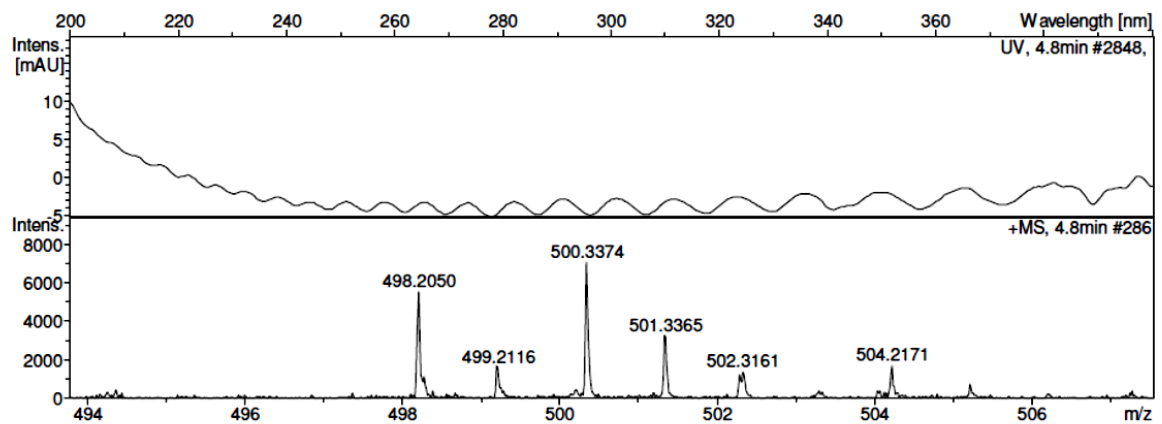
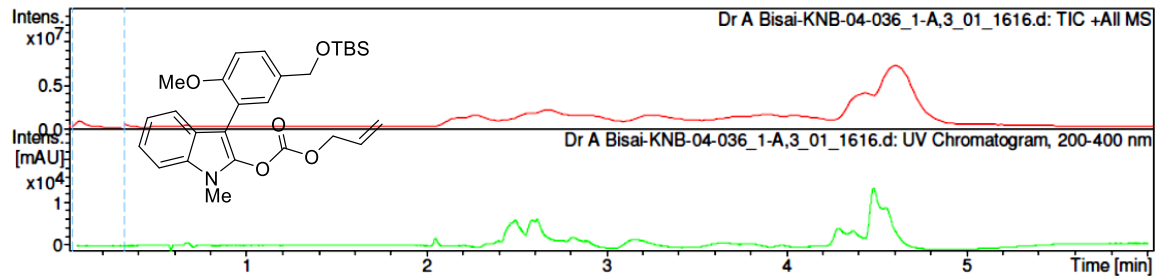
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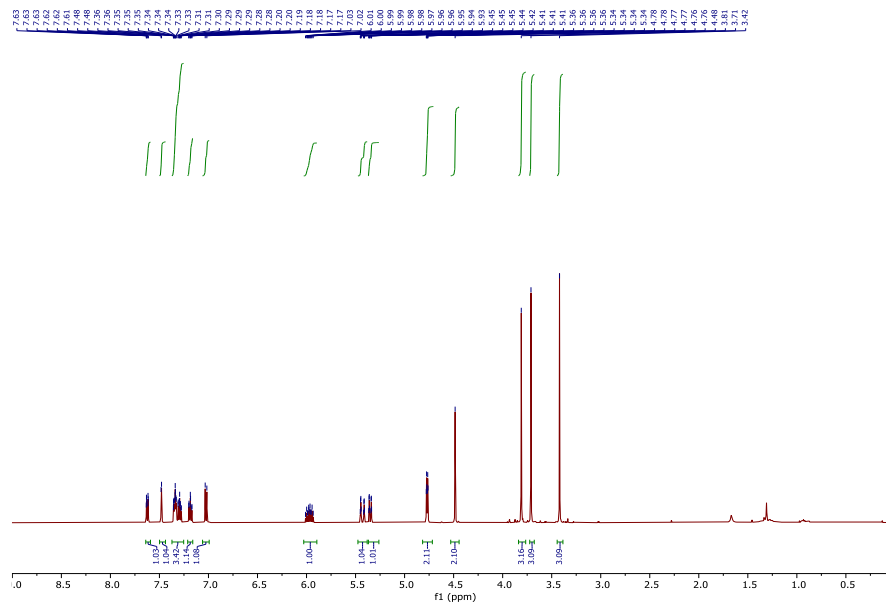
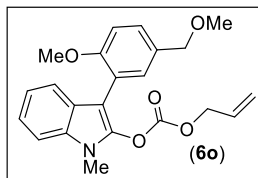
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 Sample Name Dr A Bisai-KNB-04-036  
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 Operator RUCHI  
 Instrument micrOTOF-Q II 10330

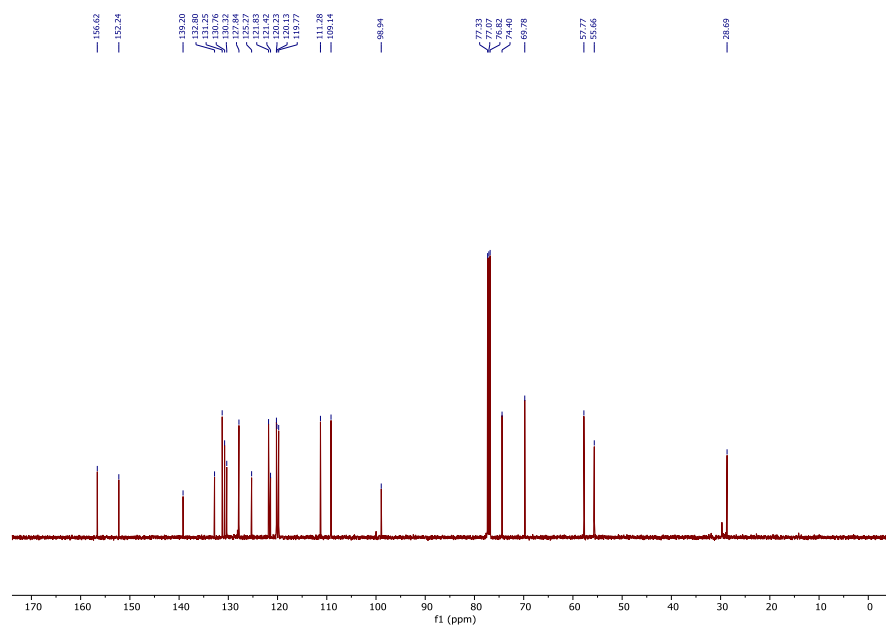
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste





$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound **60**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound **60**

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\May-2018\08-05-2018\Dr A Bisai-KNB-04-034-R\_1-A,2\_01\_1615.d  
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Sample Name Dr A Bisai-KNB-04-034-R  
Comment

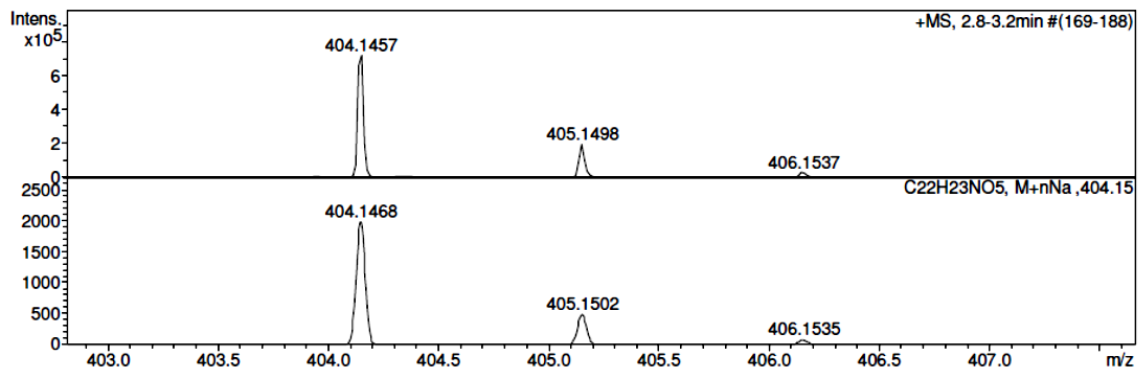
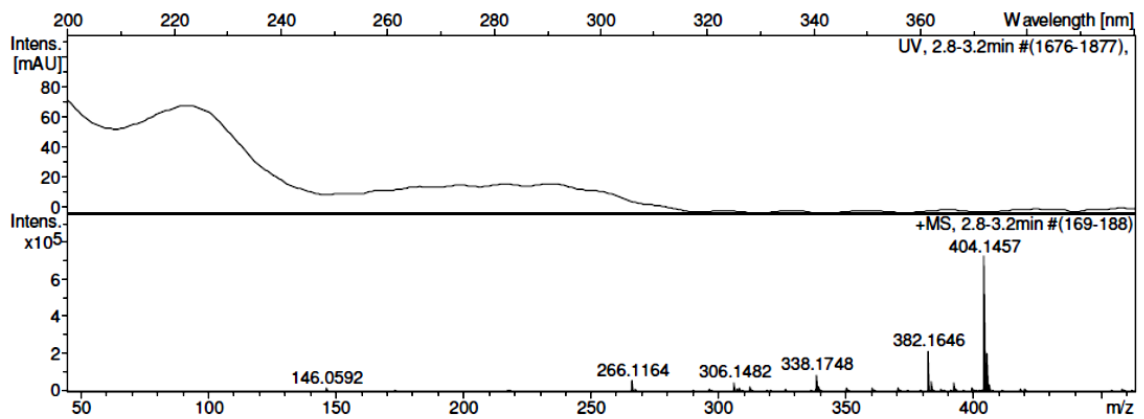
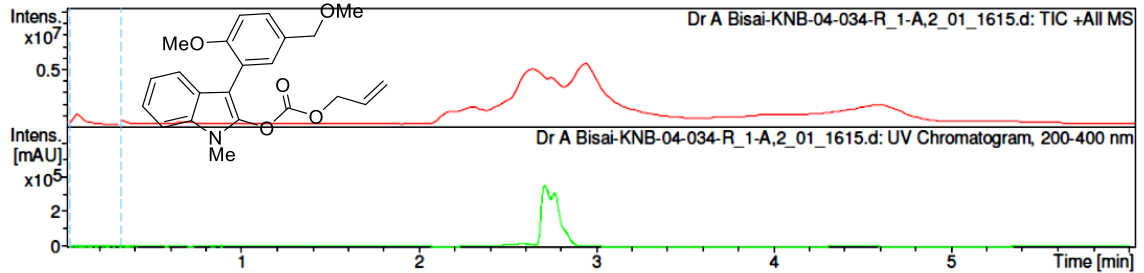
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Operator RUCHI

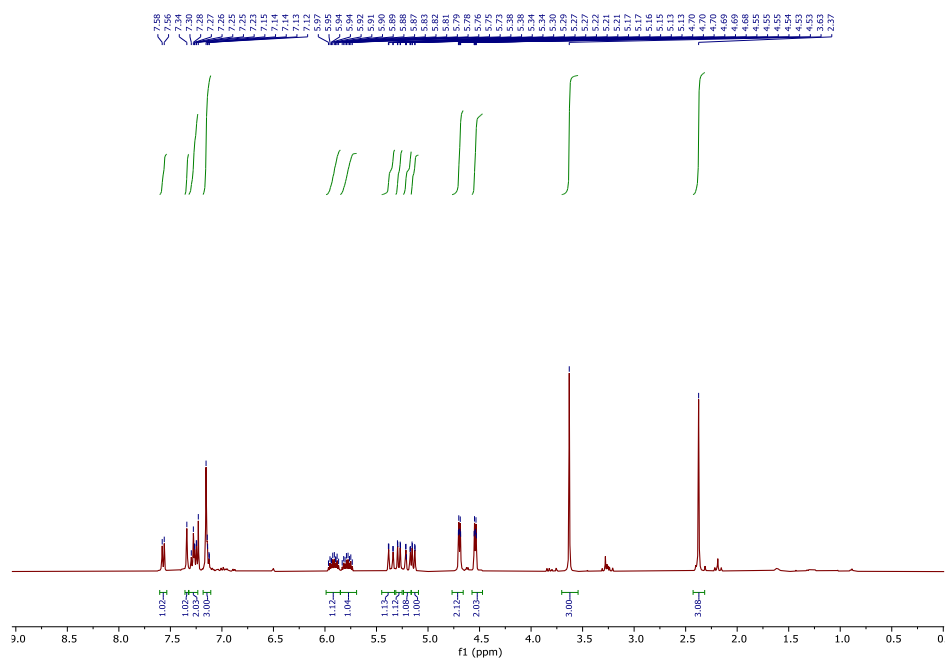
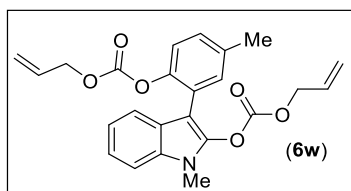
Instrument micrOTOF-Q II 10330

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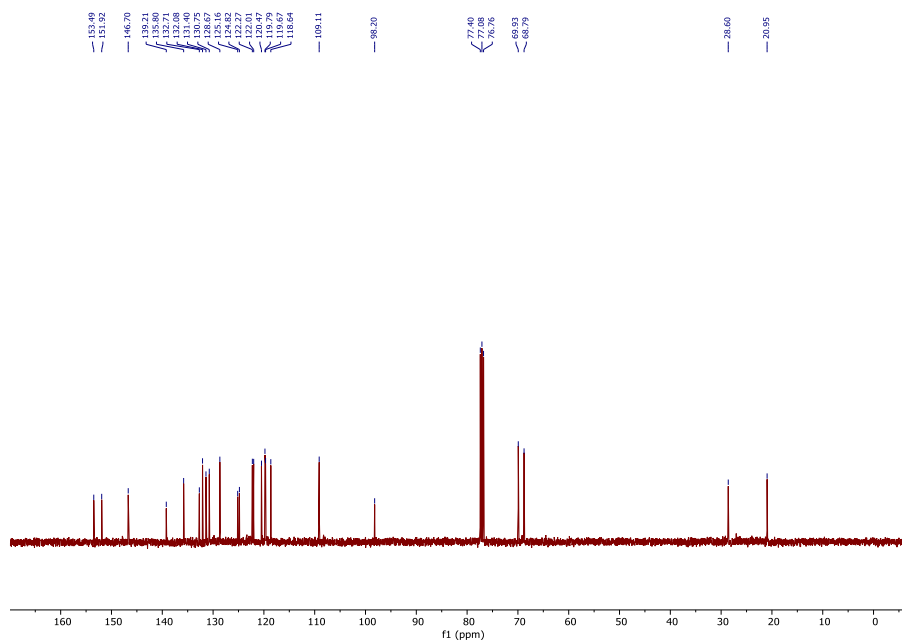
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **6w**



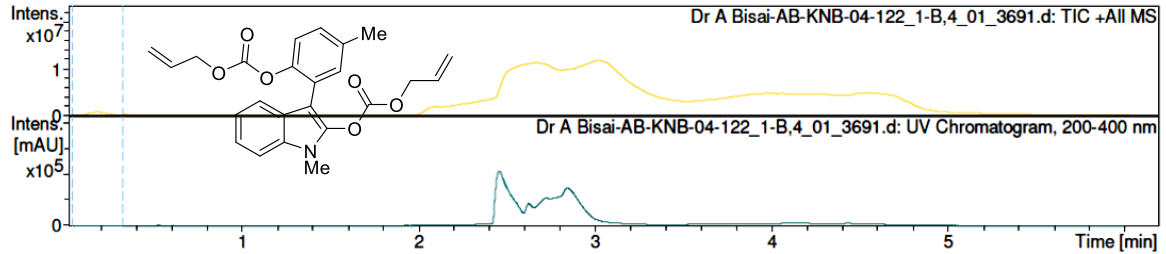
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **6w**

## Display Report

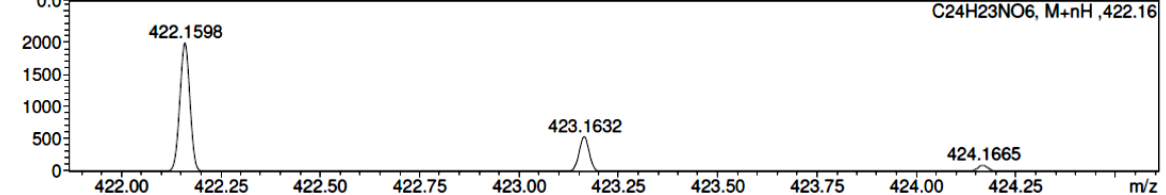
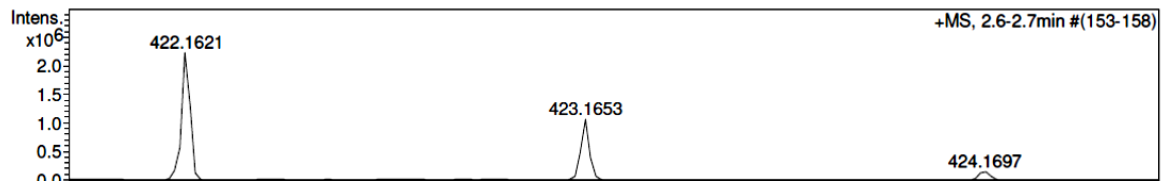
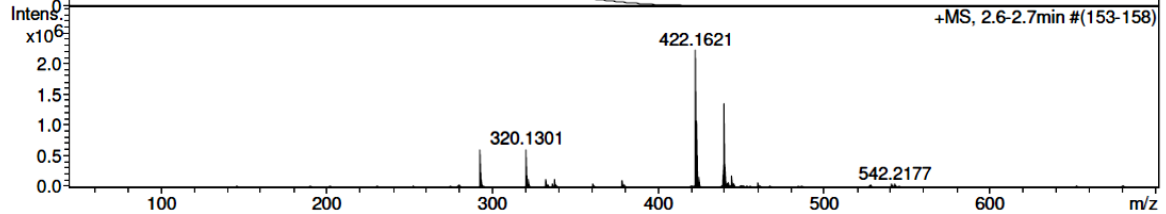
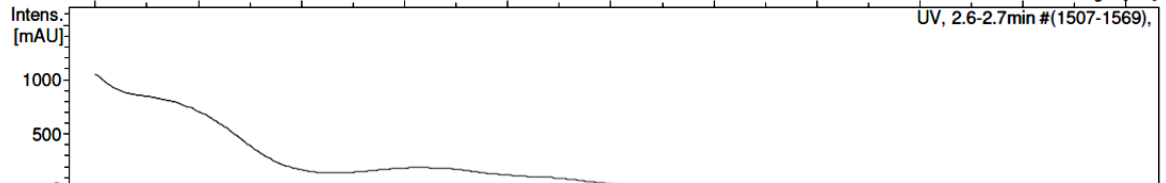
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Method	hrlcms-20 sept.m	Instrument	micrOTOF-Q II 10330
Sample Name	Dr A Bisai-AB-KNB-04-122		
Comment			

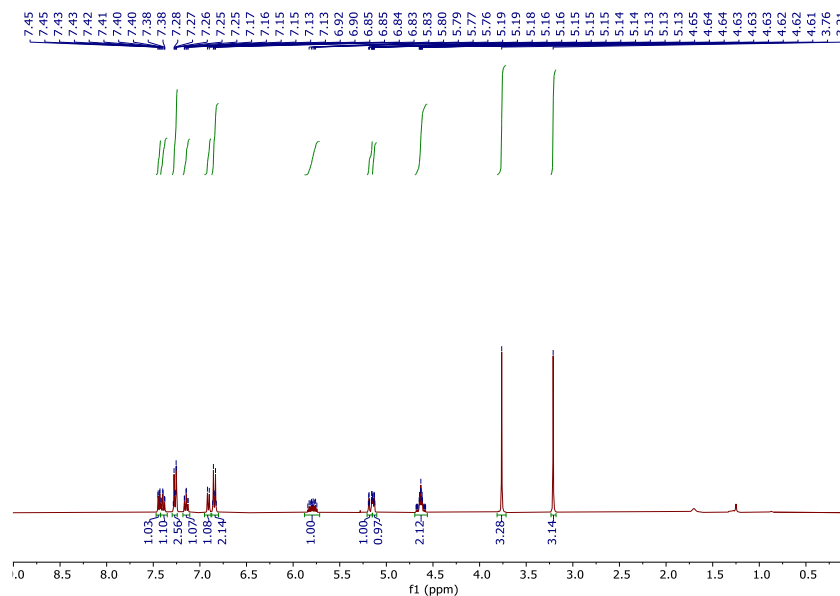
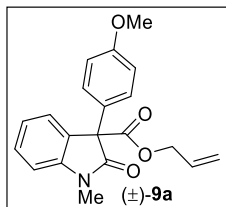
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

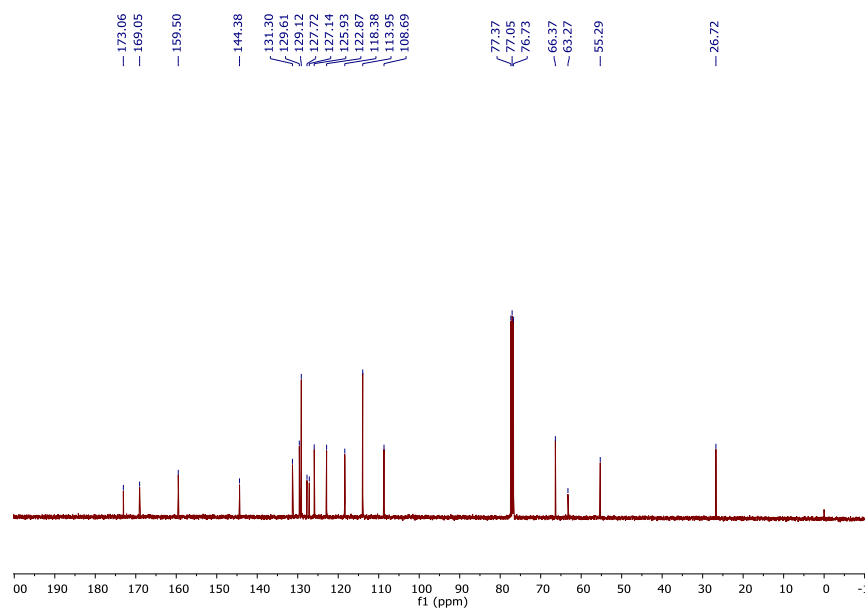


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$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (±)-9a



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (±)-9a

## Display Report

## Analysis Info

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 Sample Name Dr A Bisai-KNB-05-093-R  
 Comment

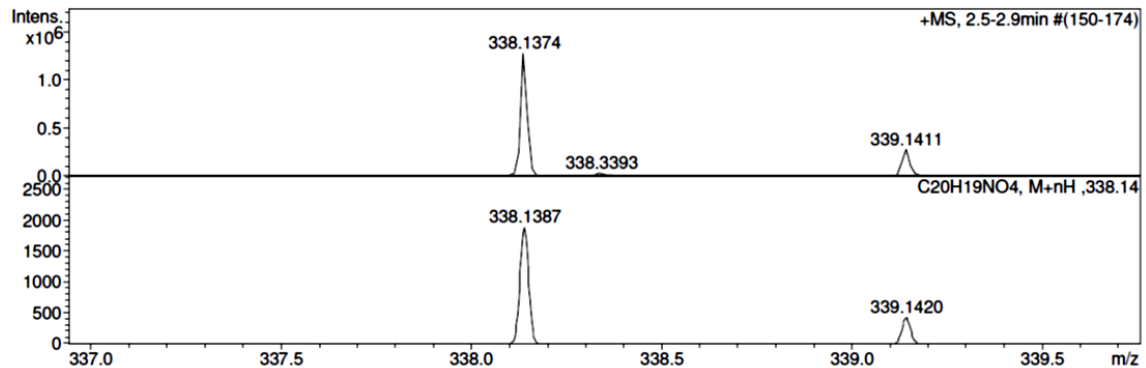
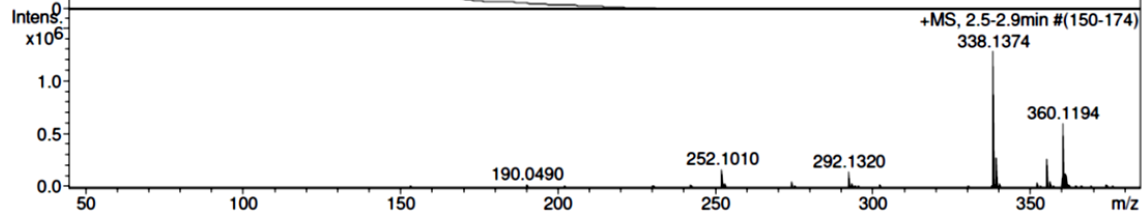
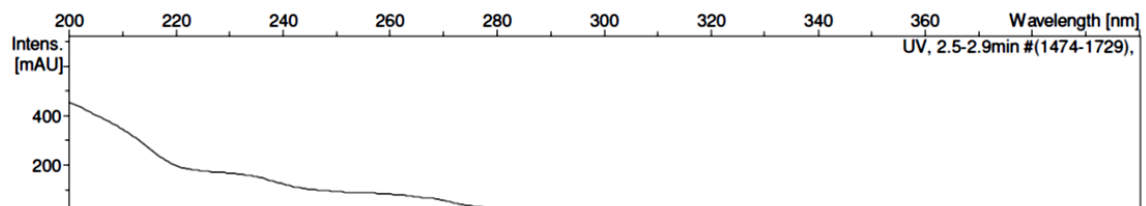
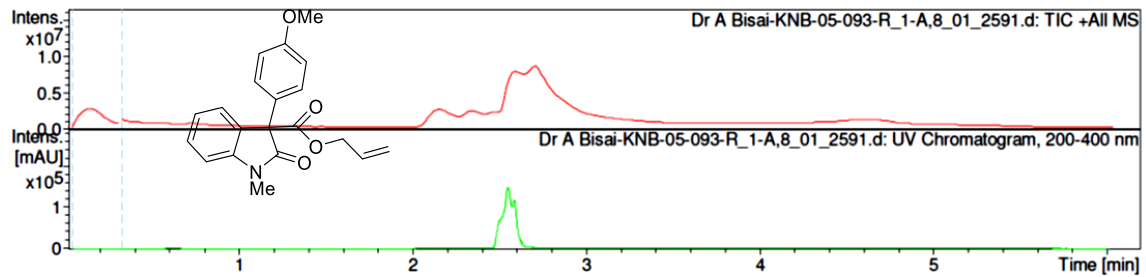
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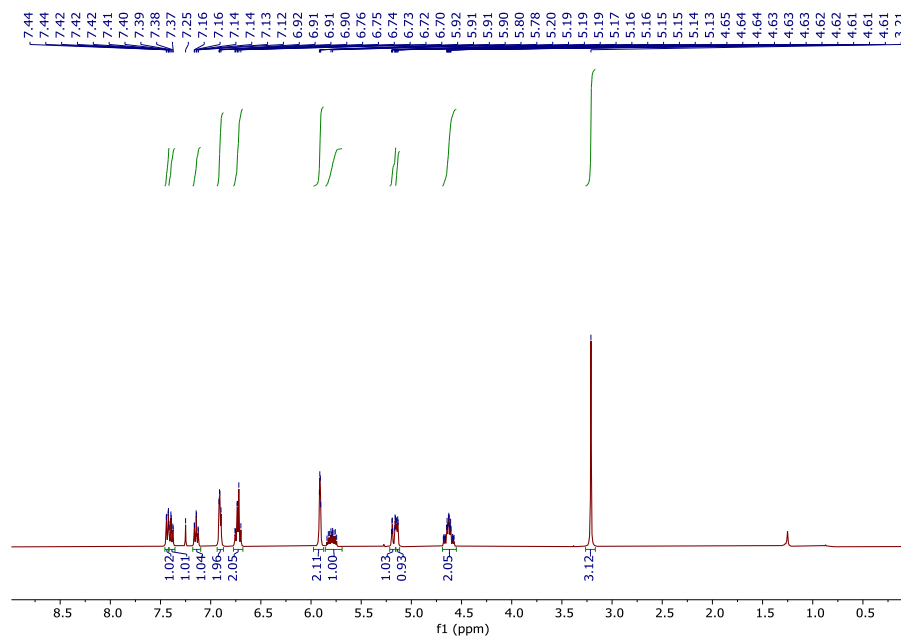
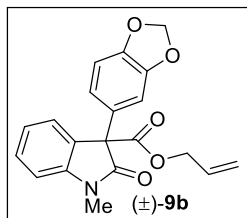
Operator RUCHI

Instrument micrOTOF-Q II 10330

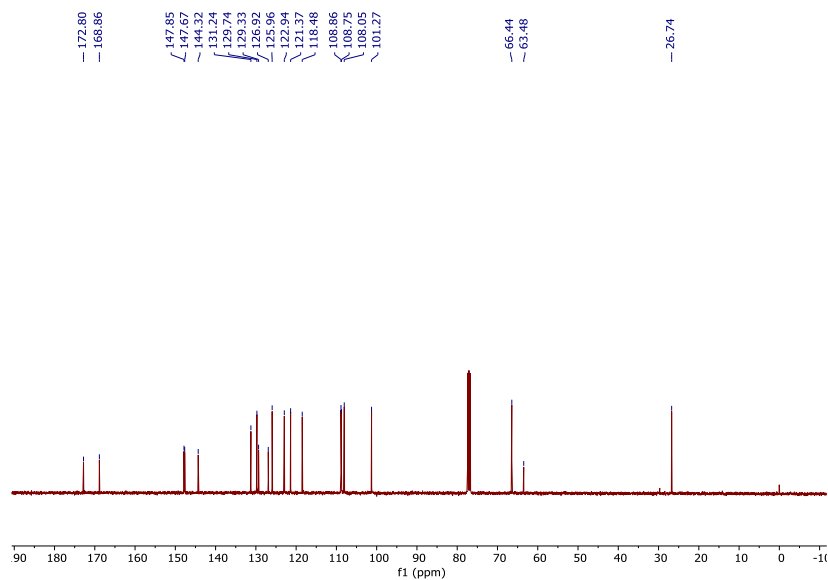
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **9a**



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (±)-**9b**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (±)-**9b**

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\JULY-2018\06-07-2018\Dr A Bisai-KNB-05-90-R\_1-A,7\_01\_2590.d  
 Method hrlcms-20 sept.m  
 Sample Name Dr A Bisai-KNB-05-90-R  
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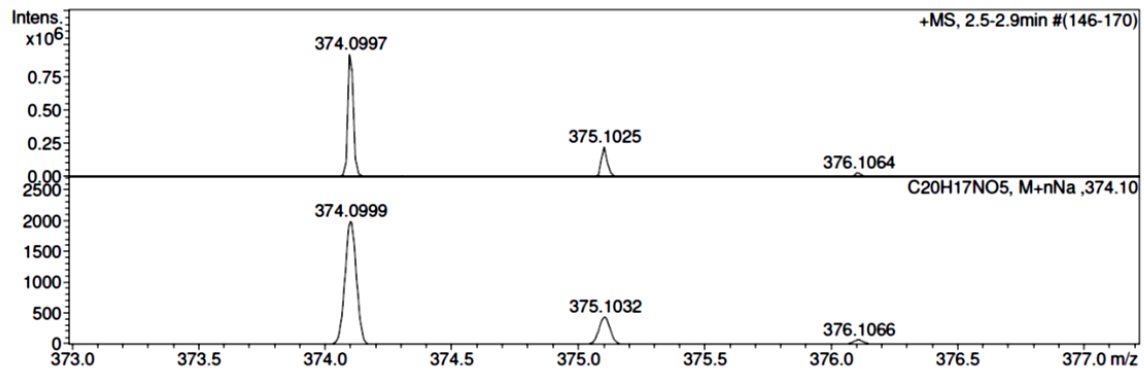
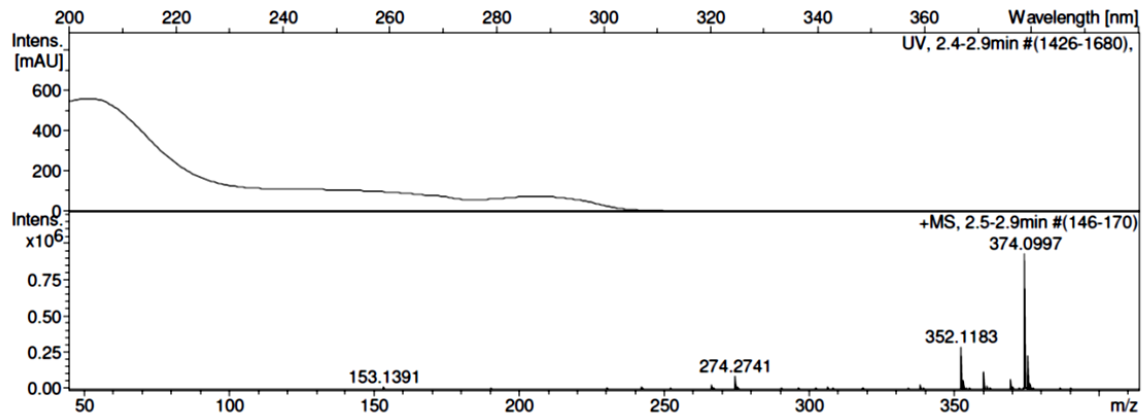
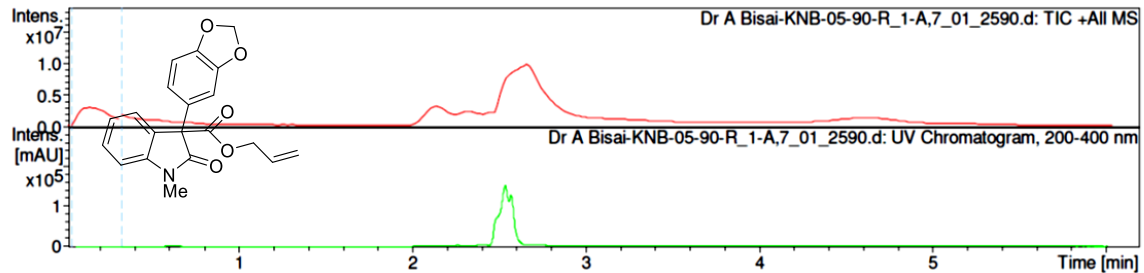
Acquisition Date 7/6/2018 12:19:07 PM

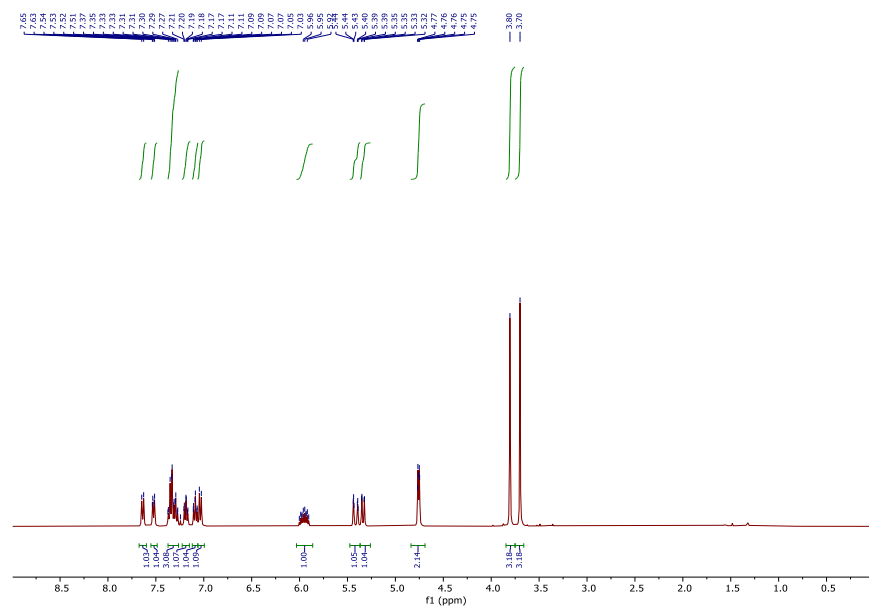
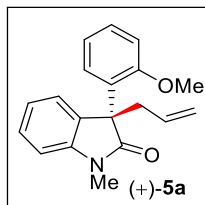
Operator RUCHI

Instrument micrOTOF-Q II 10330

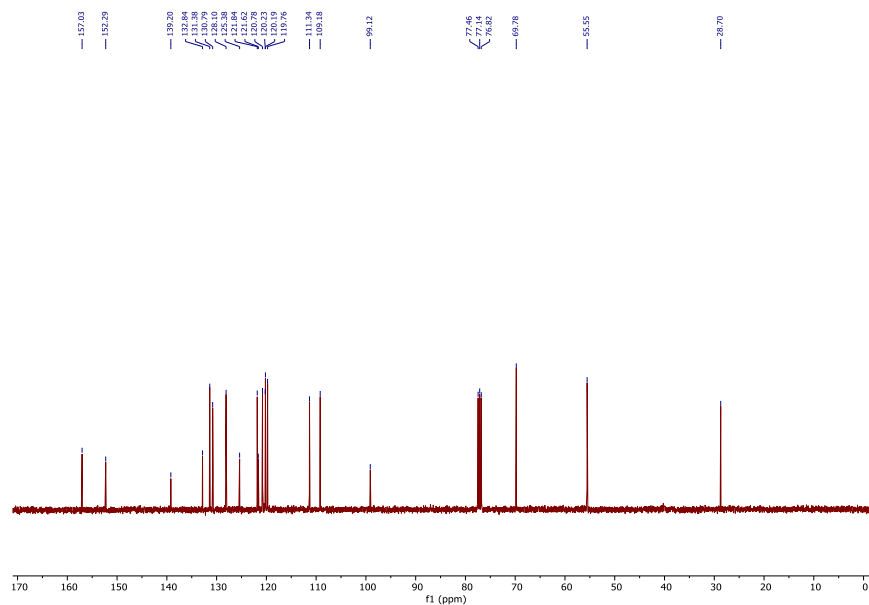
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **9b**



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5a**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5a**

## Display Report

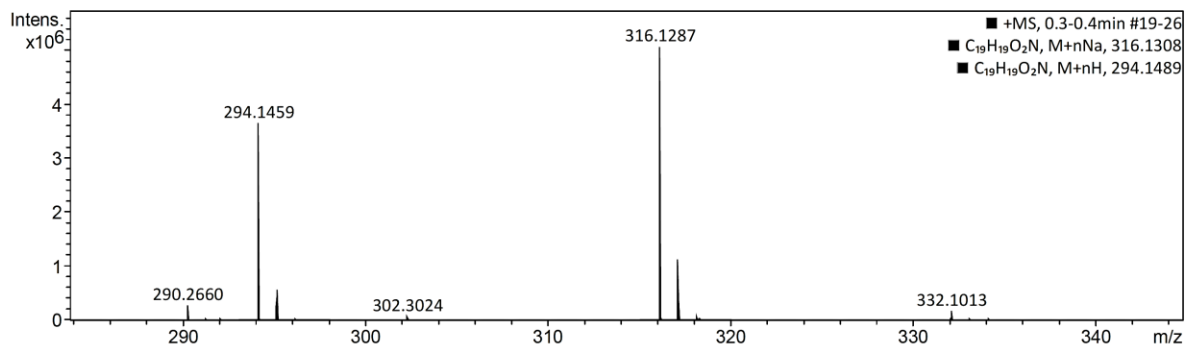
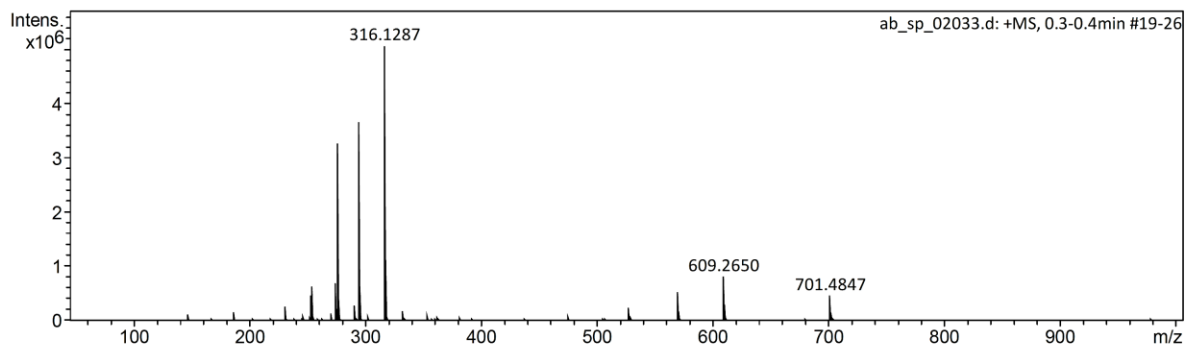
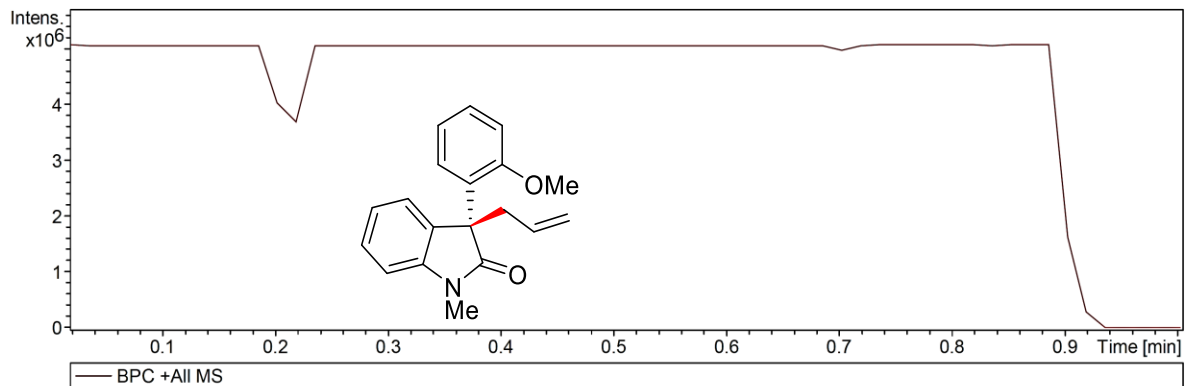
## Analysis Info

Analysis Name D:\Data\User data\2021\NOV\lab\_sp\_02033.d  
Method Tune\_pos\_Standard.m  
Sample Name ab\_sp\_02033  
Comment

Acquisition Date 11/22/2021 11:42:20 AM  
Operator IISER Kolkata  
Instrument maXis impact 8282001.00127

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Focus	Active	Set Capillary	3400 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C



ab\_sp\_02033.d

Bruker Compass DataAnalysis 4.1

printed: 11/22/2021 11:45:45 AM

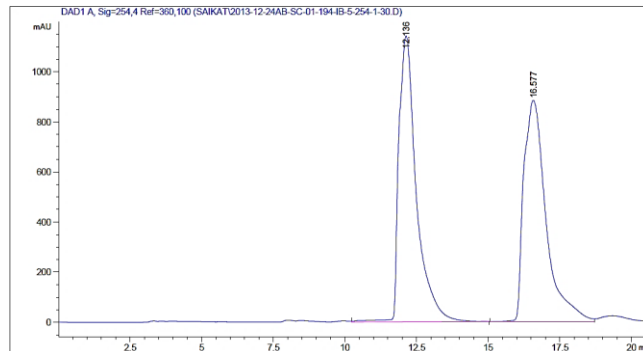
by: IISER Kolkata

Page 1 of 1

Scanned copy of mass spectrum of **5a**



Data File C:\CHEM32\1\DATA\SAIKAT\2013-12-24AB-SC-01-194-IB-5-254-1-30.D  
 Sample Name: AB-SC-01-194-IB-5-254-1-30



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

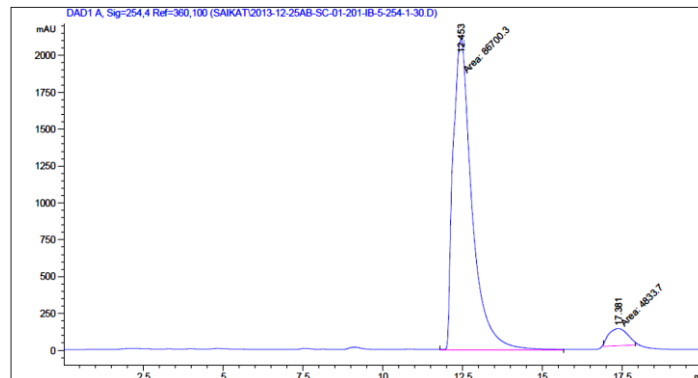
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.136	VB	0.5896	4.82314e4	1140.05469	50.2433
2	16.577	BV	0.7634	4.77643e4	882.53760	49.7567

Totals : 9.59957e4 2022.59229

\*\*\* End of Report \*\*\*

### HPLC data of (±)-5a

Data File C:\CHEM32\1\DATA\SAIKAT\2013-12-25AB-SC-01-201-IB-5-254-1-30.D  
 Sample Name: AB-SC-01-201-IB-5-254-1-30



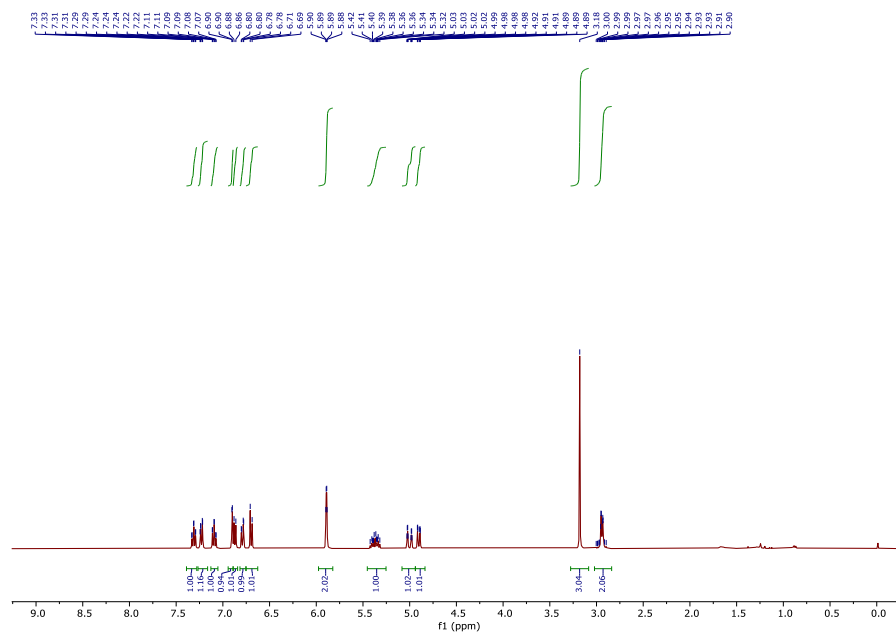
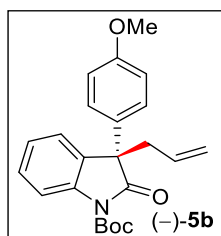
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.453	MM	0.6864	8.67003e4	2105.13916	94.7192
2	17.381	MM	0.6852	4833.70459	117.57524	5.2808

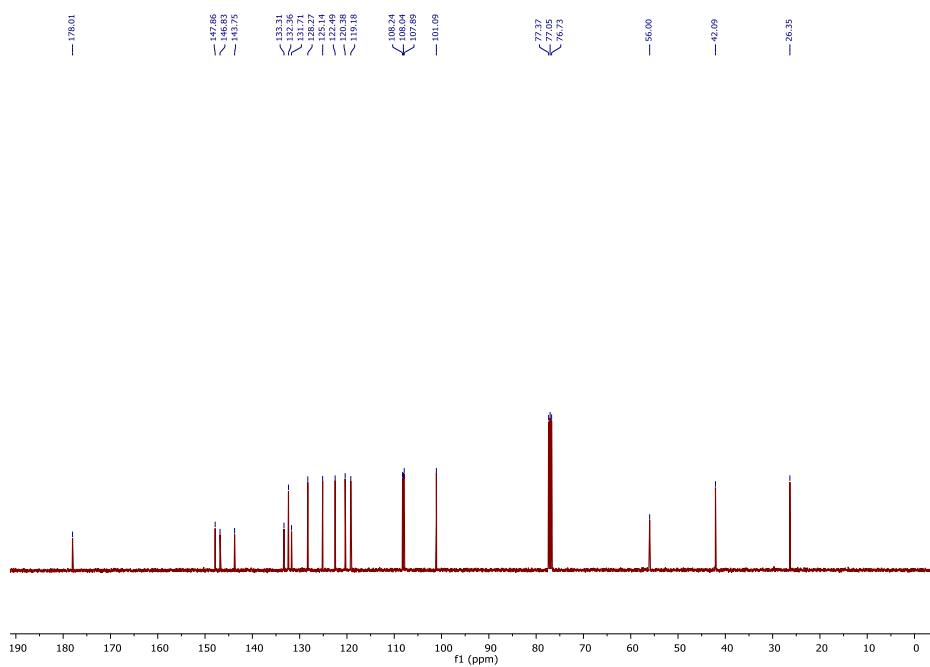
Totals : 9.15340e4 2222.71440

\*\*\* End of Report \*\*\*

### HPLC data of (+)-5a



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound **(-)-5b**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound **(-)-5b**

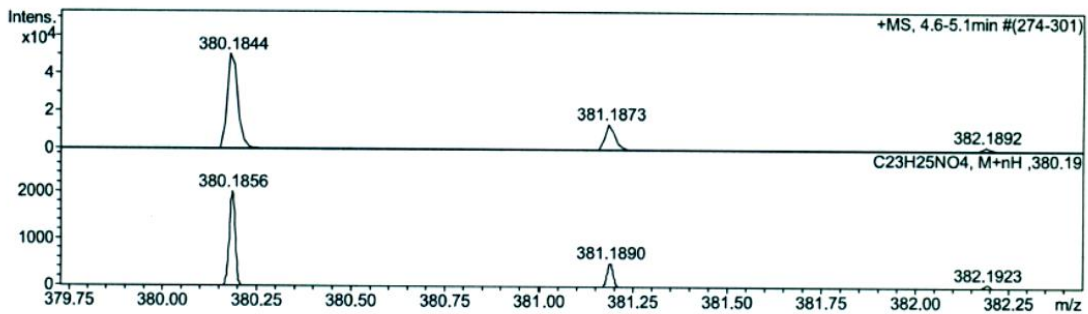
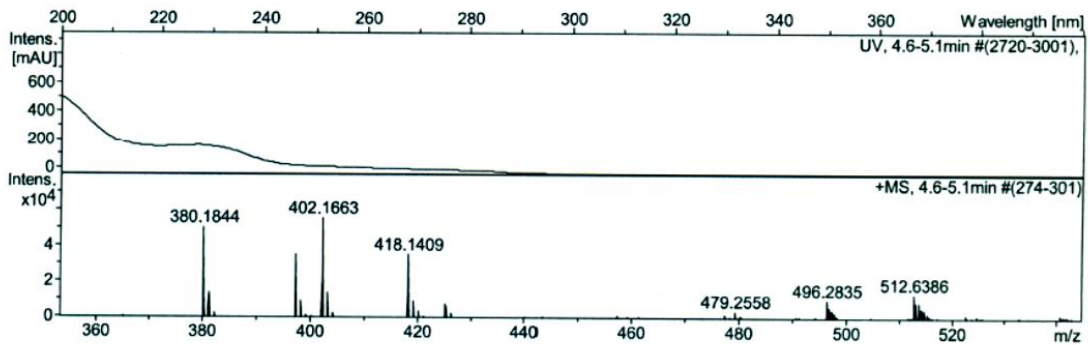
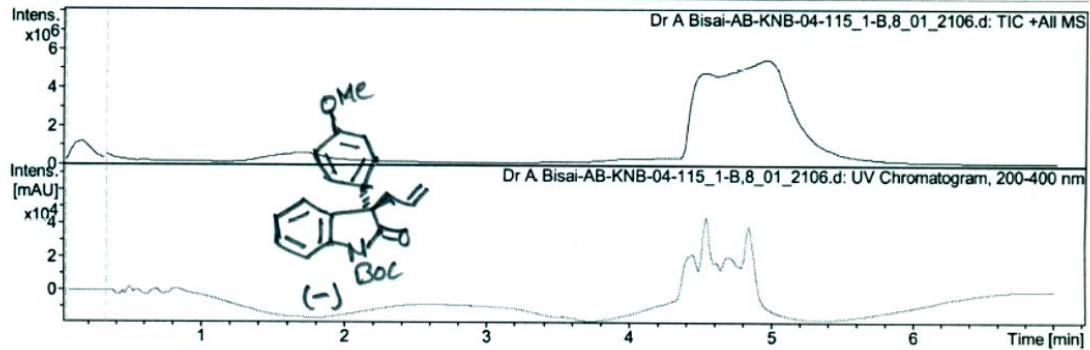
## Display Report

## Analysis Info

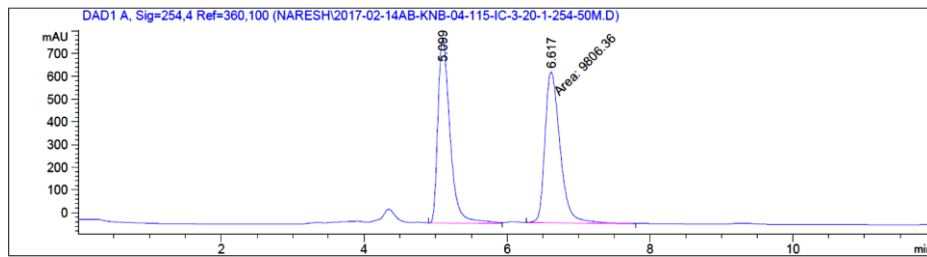
Analysis Name D:\Data\user data\2017\JUNE 2017\15 june\Dr A Bisai-AB-KNB-04-115\_1-B,8\_01\_2106.d Acquisition Date 6/15/2017 2:42:47 PM  
 Method hrlcms\_pos\_mid\_tunemix.m Operator RUCHI SHRIVASTAVA  
 Sample Name Dr A Bisai-AB-KNB-04-115 Instrument microTOF-Q II 10330  
 Comment

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5b**

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-115-IC-3-20-1-254-50M.D  
 Sample Name: AB-KNB-04-115-IC-3-20-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

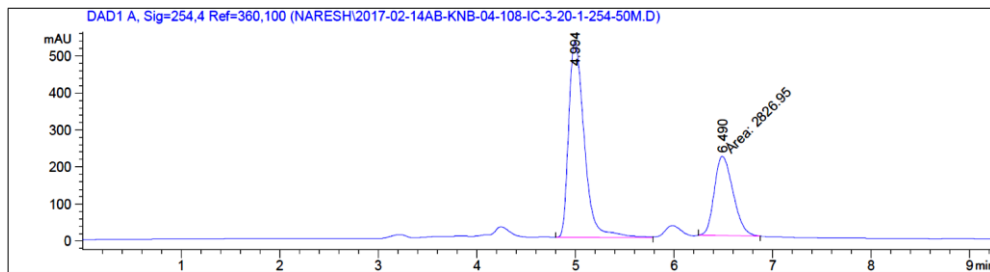
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.099	BV	0.1822	9584.14258	808.81201	49.4270
2	6.617	MM	0.2458	9806.36426	665.05725	50.5730

Totals : 1.93905e4 1473.86926

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

### HPLC data of (±)-5b

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-108-IC-3-20-1-254-50M.D  
 Sample Name: AB-KNB-04-108-IC-3-20-1-254-50M



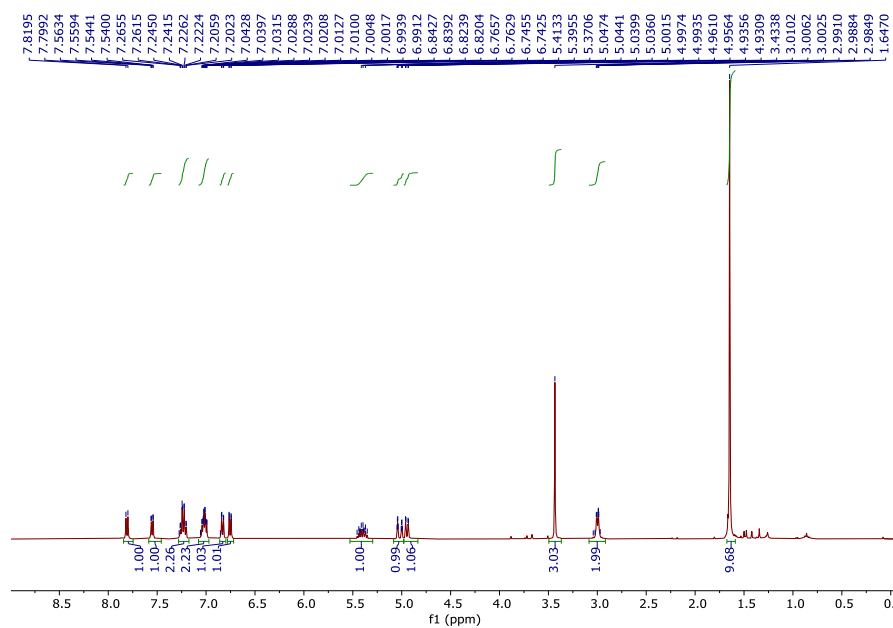
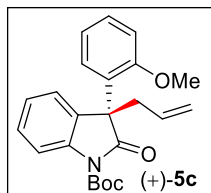
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.994	BV	0.1727	5828.86377	528.29065	67.3404
2	6.490	MM	0.2205	2826.95044	213.67680	32.6596

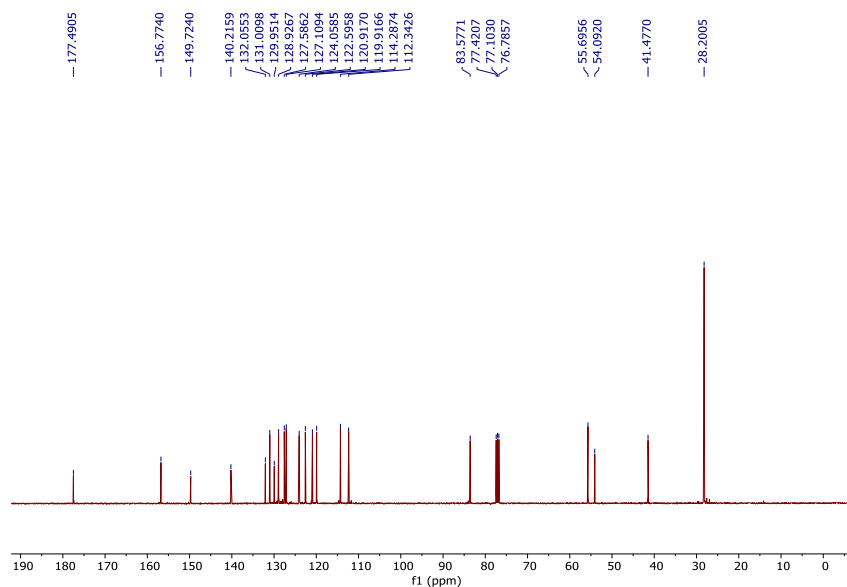
Totals : 8655.81421 741.96745

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 \*\*\* End of Report \*\*\*

### HPLC data of (-)-5b



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5c**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5c**

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\27 mar\Dr A Bisai-KNB-03-197\_1-A,8\_01\_1410.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-KNB-03-197  
Comment

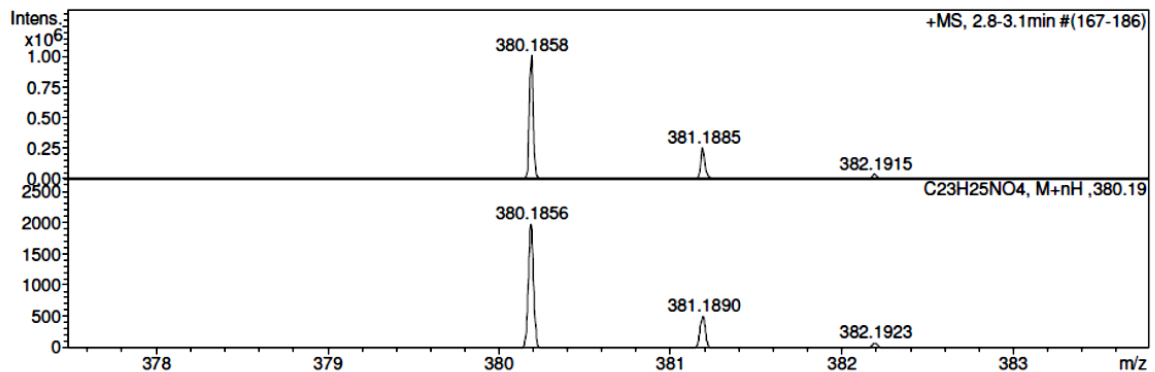
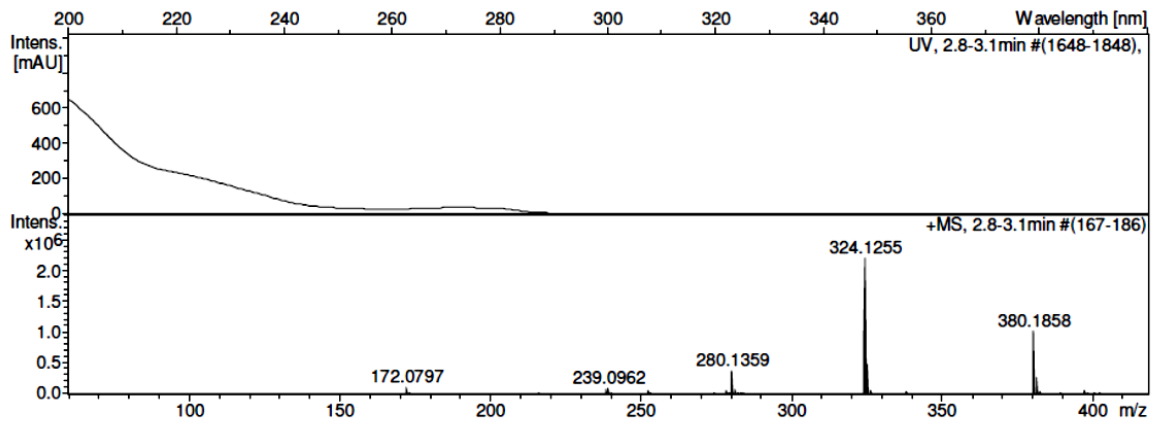
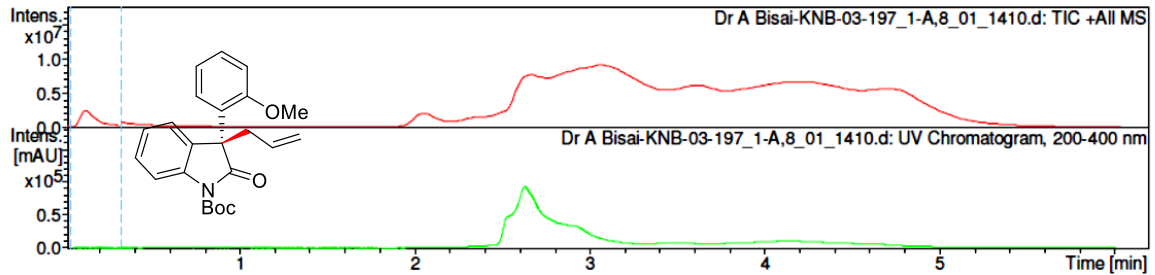
Acquisition Date 3/27/2018 11:49:33 AM

Operator RUCHI

Instrument micrOTOF-Q II 10330

## Acquisition Parameter

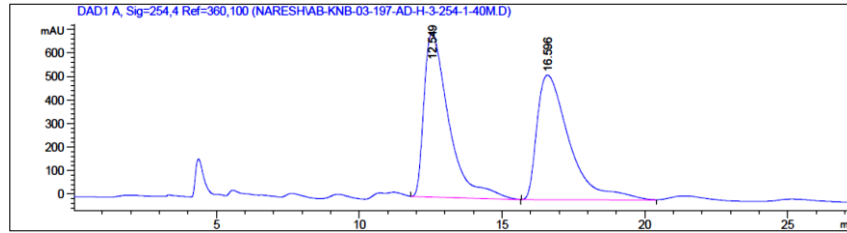
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of 5c

Data File C:\CHEM32\1\DATA\NARESH\AB-KNB-03-197-AD-H-3-254-1-40M.D

Sample Name: AB-KNB-03-197-AD-H-3-254-1-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.549	BB	0.9100	4.20021e4	698.02222	50.2558
2	16.596	BB	1.1960	4.15744e4	529.53717	49.7442

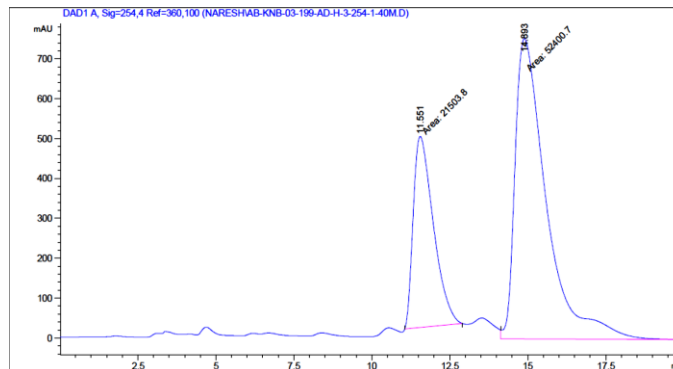
Totals : 8.35765e4 1227.55939

\*\*\* End of Report \*\*\*

HPLC data of (±)-5c

Data File C:\CHEM32\1\DATA\NARESH\AB-KNB-03-199-AD-H-3-254-1-40M.D

Sample Name: AB-KNB-03-199-AD-H-3-254-1-40M



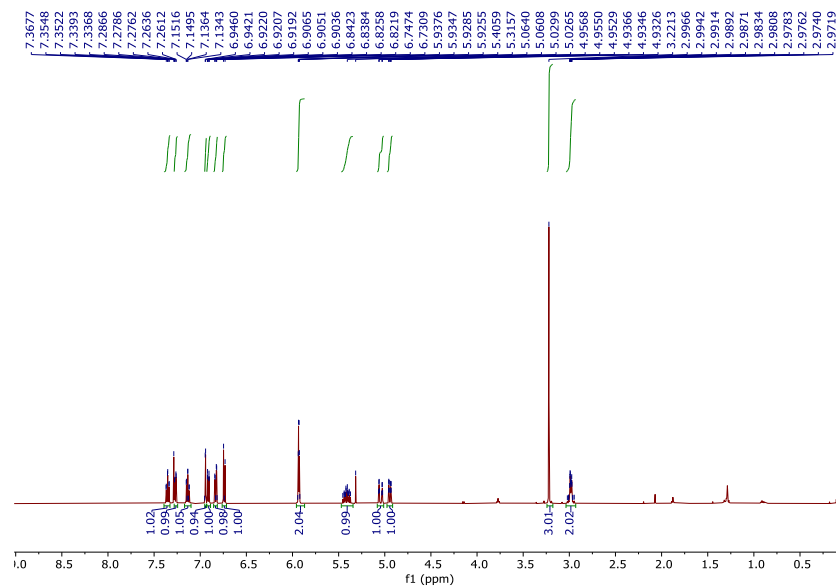
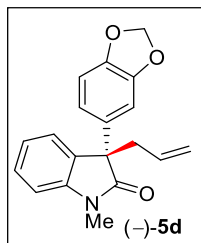
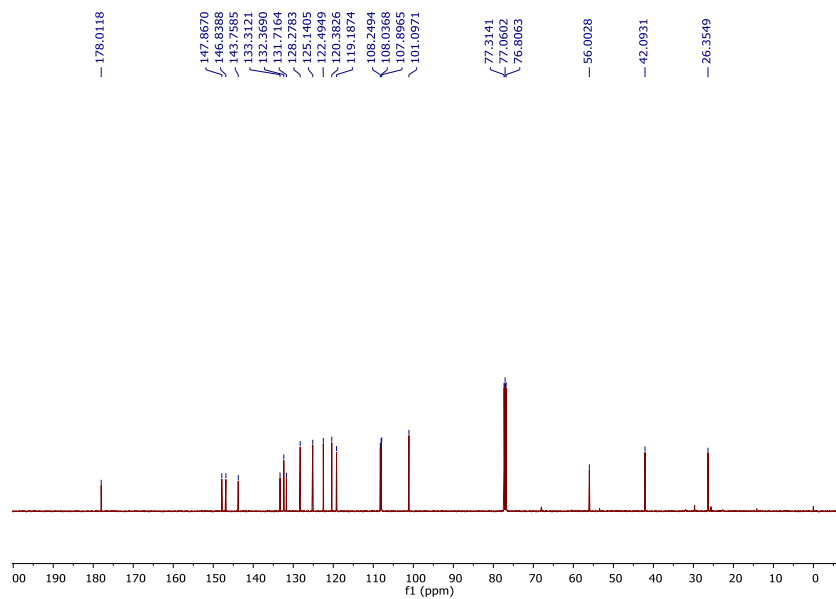
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.551	MM	0.7491	2.15038e4	478.44043	29.0967
2	14.893	MM	1.1608	5.24007e4	752.38489	70.9033

Totals : 7.39045e4 1230.82532

\*\*\* End of Report \*\*\*

HPLC data of (+)-5c

 $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (-)-5d $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (-)-5d



## Display Report

## Analysis Info

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Method Pos\_Mid\_tunemix.m  
Sample Name AB-KNB-01-104  
Comment

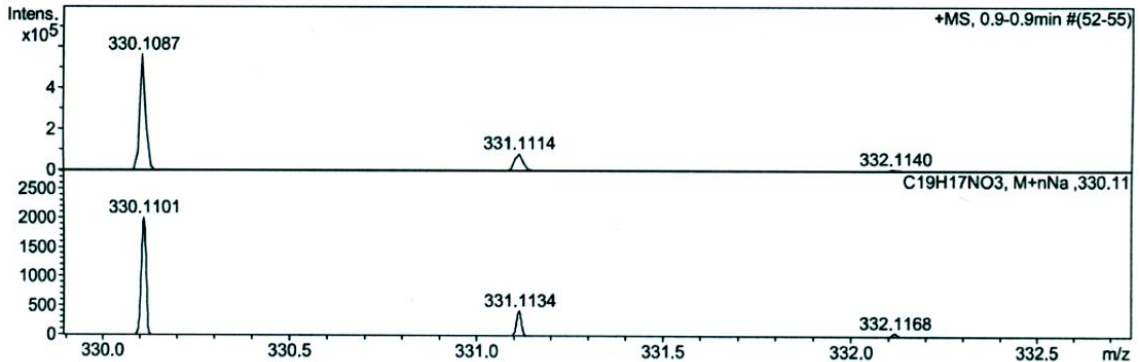
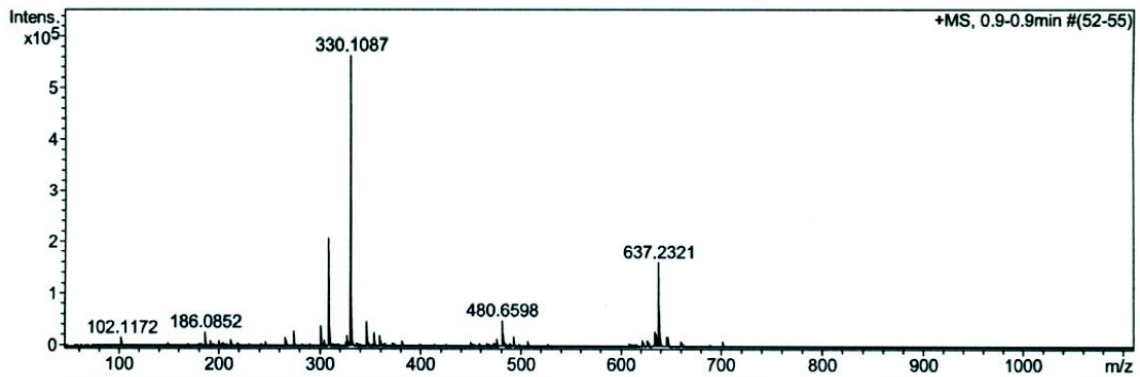
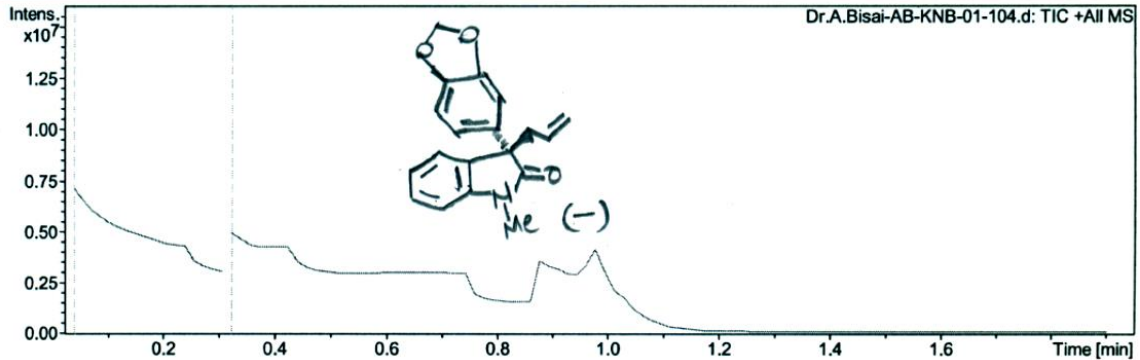
Acquisition Date 6/16/2017 4:25:41 PM

Operator RUCHI SHRIVASTAVA

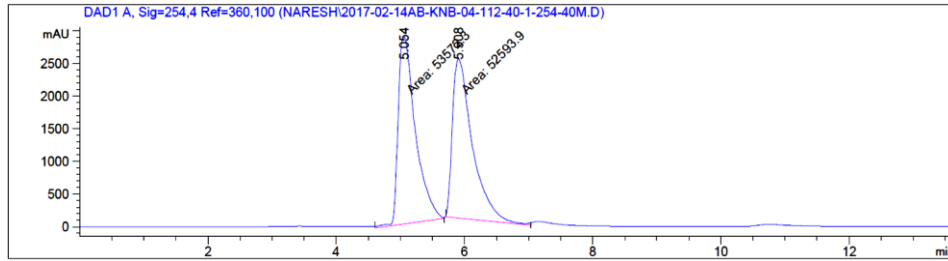
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-112-40-1-254-40M.D  
 Sample Name: AB-KNB-04-112-40-1-254-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

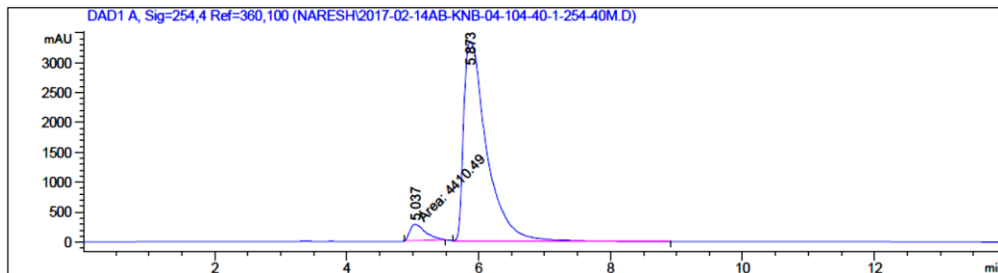
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.054	MM	0.3109	5.35723e4	2871.45532	50.4608
2	5.908	MM	0.3597	5.25939e4	2437.19238	49.5392

Totals : 1.06166e5 5308.64771

\*\*\* End of Report \*\*\*

### HPLC data of (±)-5d

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-104-40-1-254-40M.D  
 Sample Name: AB-KNB-04-104-40-1-254-40M



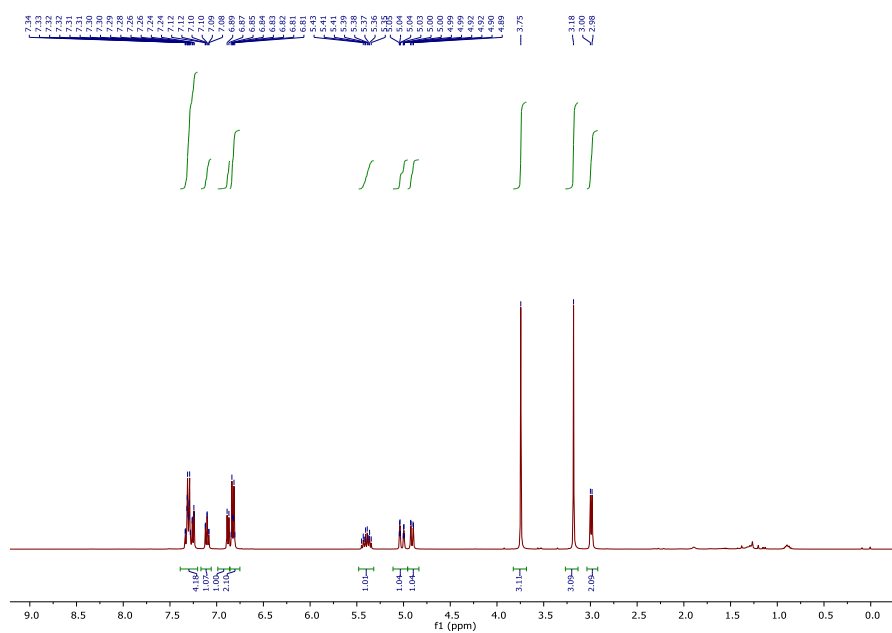
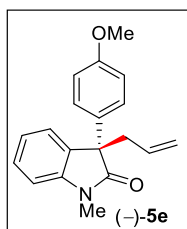
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.037	MM	0.2758	4410.48535	266.49200	5.1225
2	5.873	VB	0.3608	8.16890e4	3346.01221	94.8775

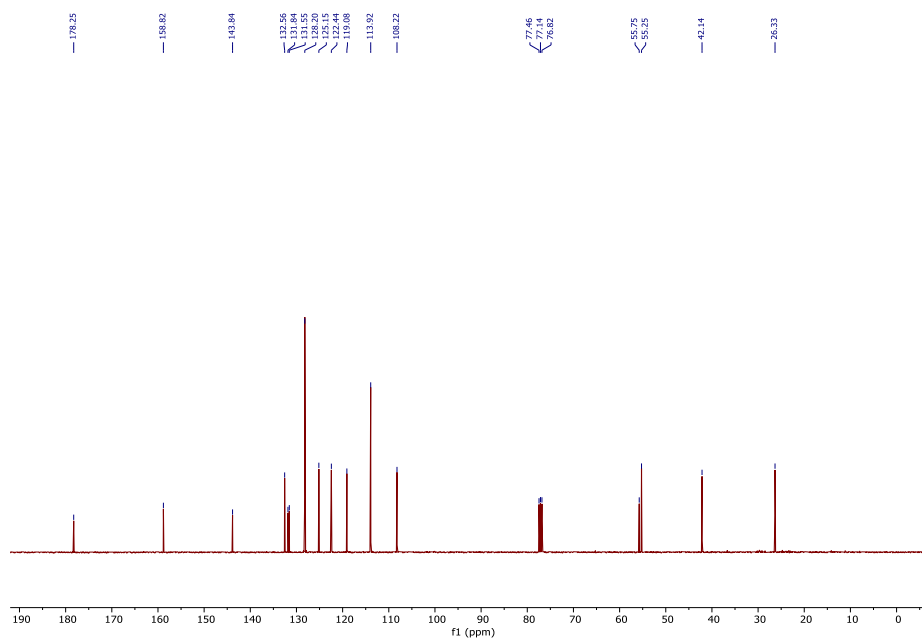
Totals : 8.60995e4 3612.50421

\*\*\* End of Report \*\*\*

### HPLC data of (-)-5d



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (-)-**5e**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (-)-**5e**

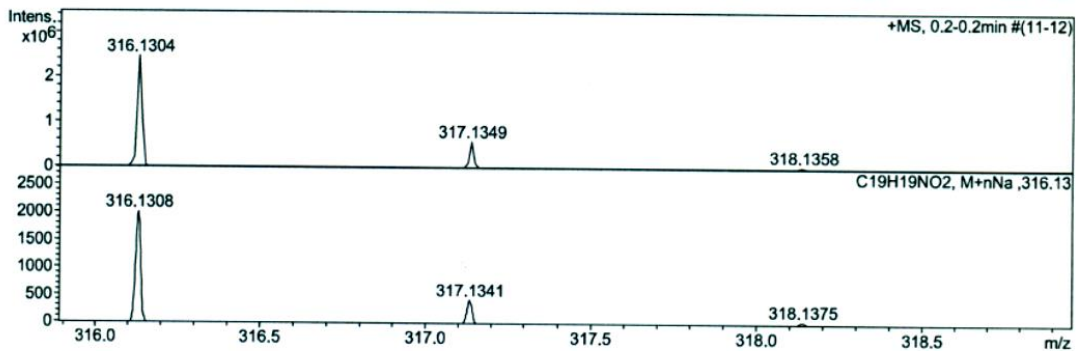
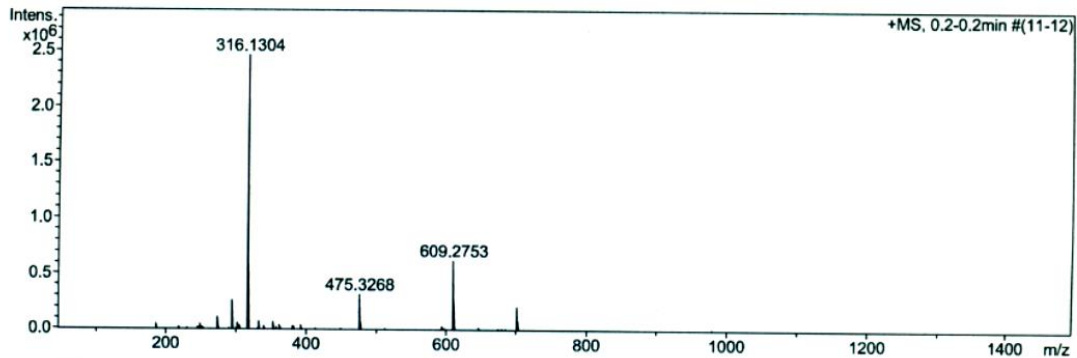
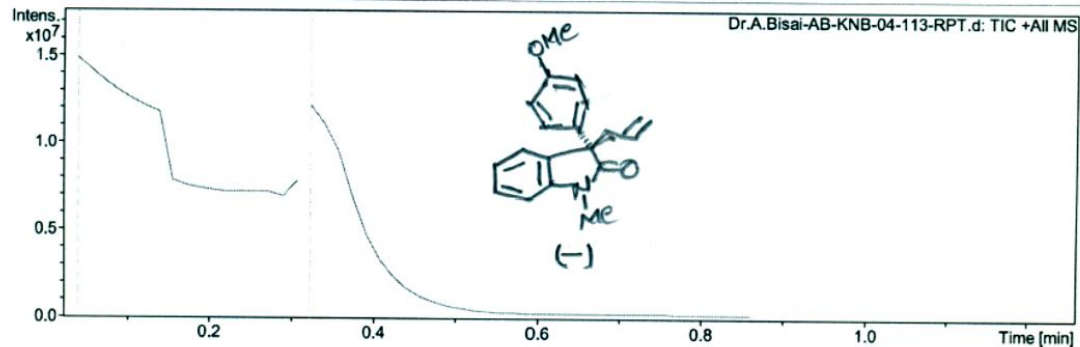
## Display Report

## Analysis Info

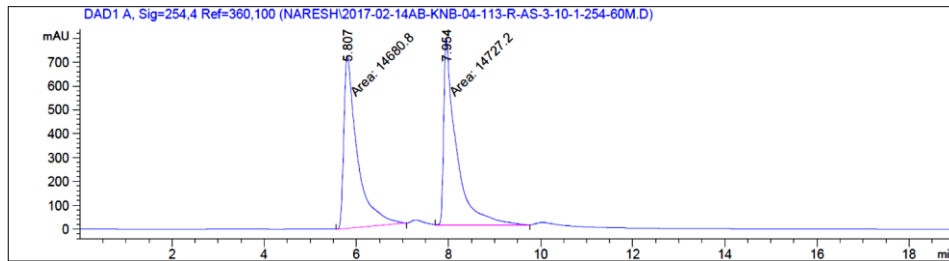
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Method	Pos_Mid_tunemix.m	Operator	RUCHI SHRIVASTAVA
Sample Name	AB-KNB-04-113-RPT	Instrument	micrOTOF-Q II 10330
Comment			

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-113-R-AS-3-10-1-254-60M.D  
 Sample Name: AB-KNB-04-113-R-AS-3-10-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

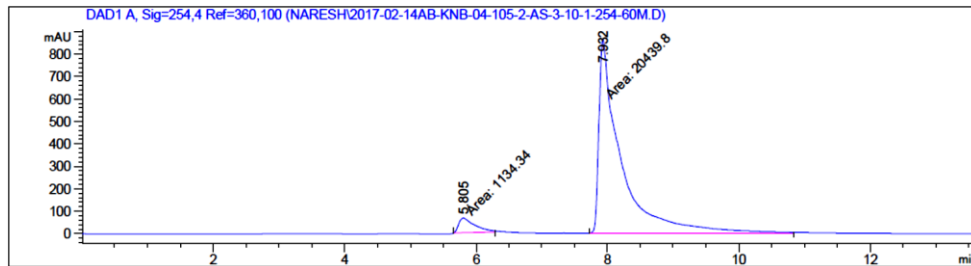
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.807	MM	0.3373	1.46808e4	725.41425	49.9211
2	7.954	MM	0.3135	1.47272e4	782.90521	50.0789

Totals : 2.94081e4 1508.31946

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 \*\*\* End of Report \*\*\*

### HPLC data of (±)-5e

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-105-2-AS-3-10-1-254-60M.D  
 Sample Name: AB-KNB-04-105-2-AS-3-10-1-254-60M



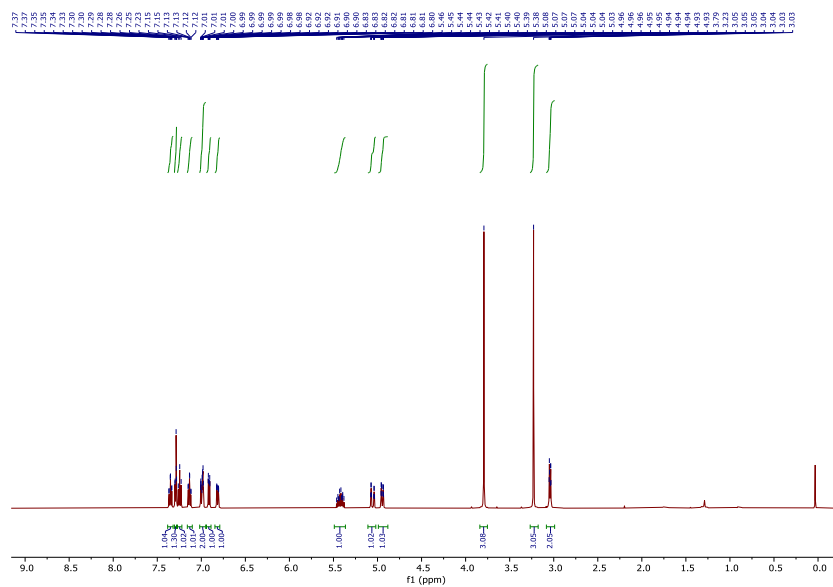
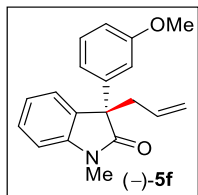
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.805	MM	0.2850	1134.33728	66.34151	5.2578
2	7.932	MM	0.3944	2.04398e4	863.64685	94.7422

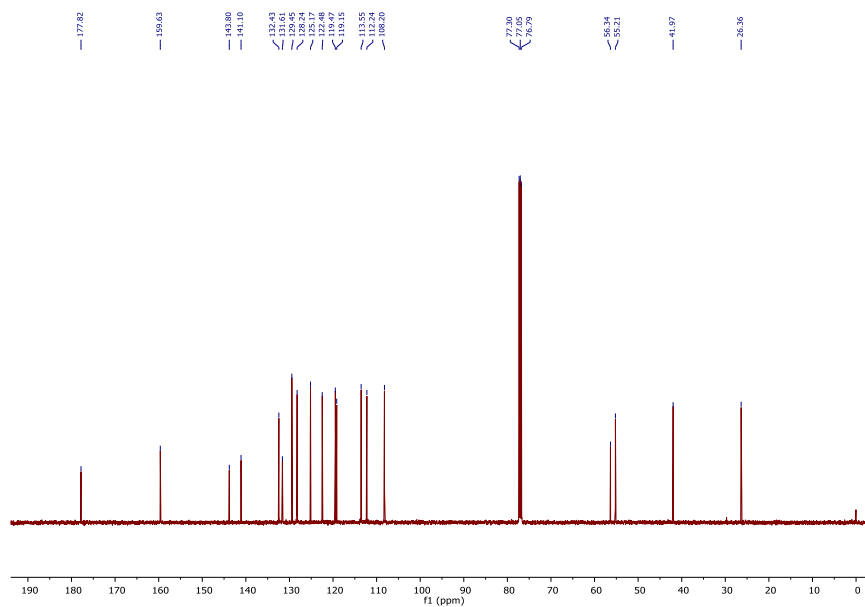
Totals : 2.15742e4 929.98837

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 \*\*\* End of Report \*\*\*

### HPLC data of (-)-5e



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (-)-**5f**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (-)-**5f**

## Display Report

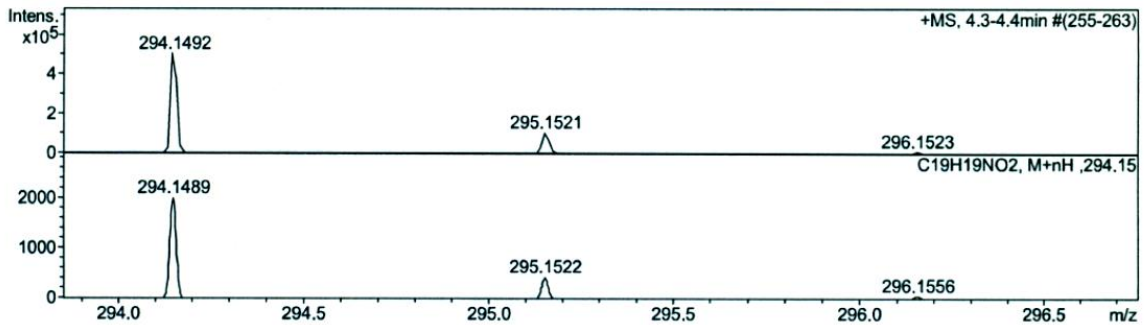
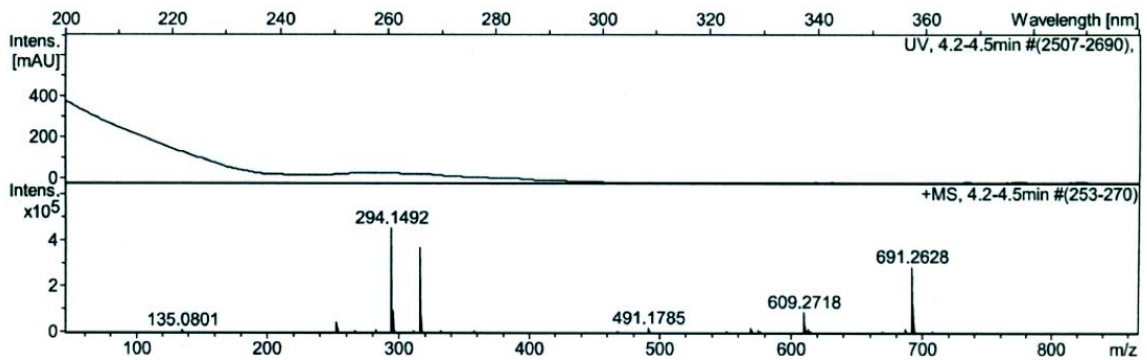
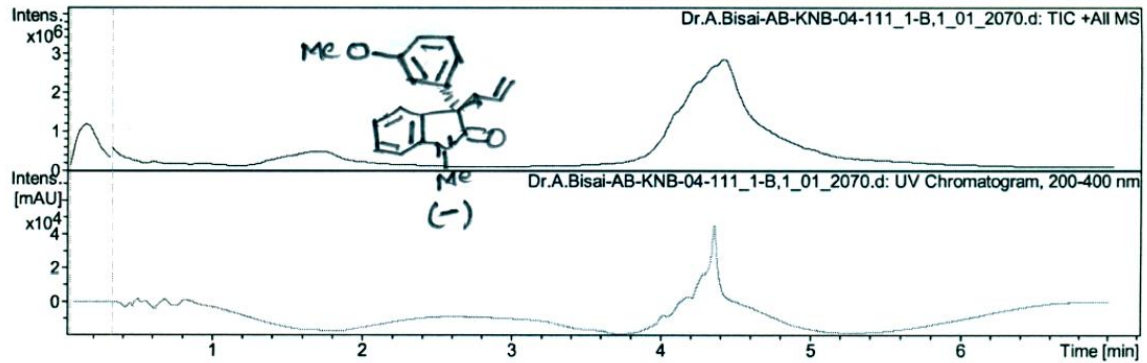
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\14 June\Dr.A.Bisai-AB-KNB-04-111\_1-B,1\_01\_2070.d  
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 Sample Name Dr.A.Bisai-AB-KNB-04-111  
 Comment

Acquisition Date 6/14/2017 12:39:38 PM  
 Operator RUCHI SHRIVASTAVA  
 Instrument micrOTOF-Q II 10330

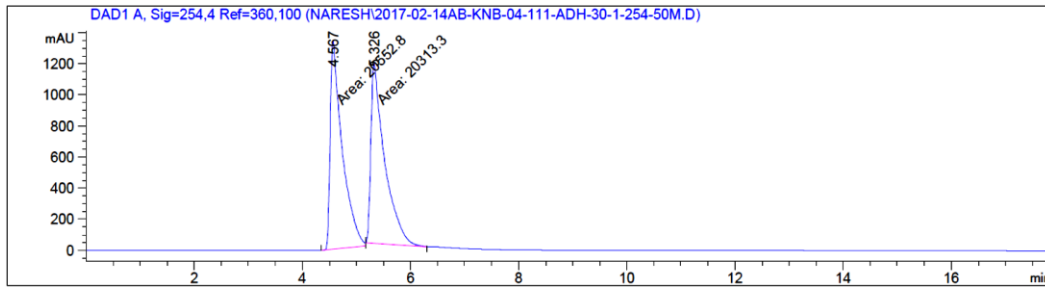
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-111-ADH-30-1-254-50M.D

Sample Name: AB-KNB-04-111-ADH-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.567	MM	0.2560	2.05528e4	1337.87659	50.2930
2	5.326	MM	0.2933	2.03133e4	1154.38843	49.7070

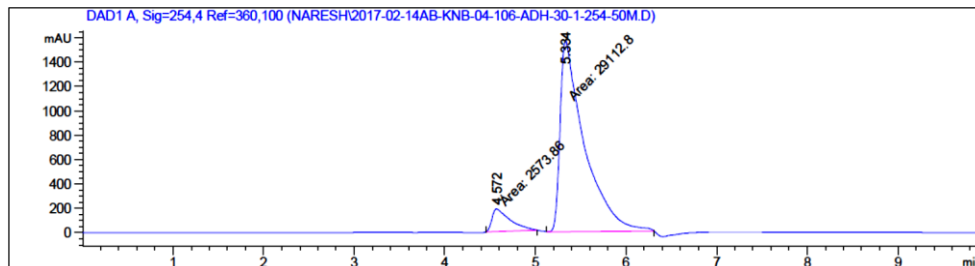
Totals : 4.08662e4 2492.26501

\*\*\* End of Report \*\*\*

## HPLC data of (±)-5f

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-106-ADH-30-1-254-50M.D

Sample Name: AB-KNB-04-106-ADH-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

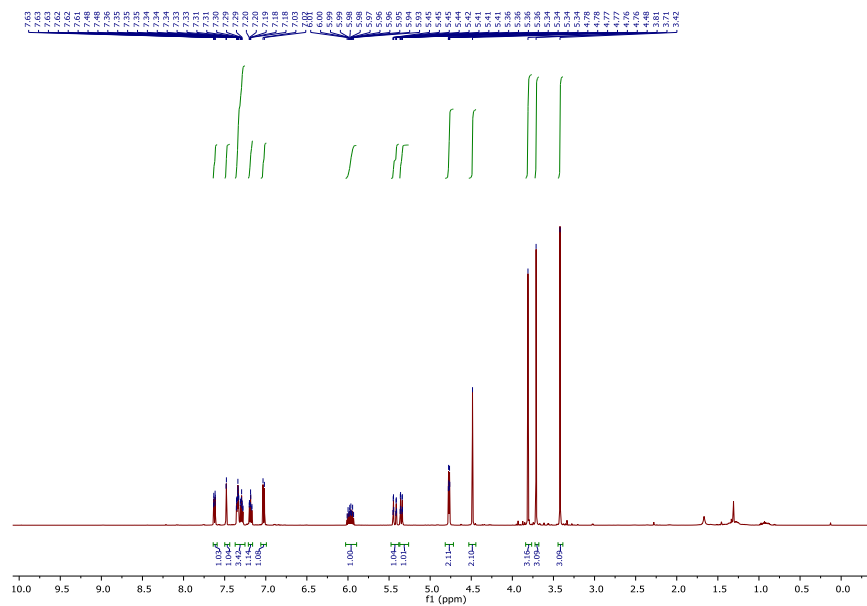
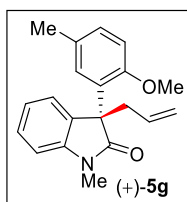
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.572	MM	0.2291	2573.85840	187.26247	8.1228
2	5.334	MM	0.3095	2.91128e4	1567.78357	91.8772

Totals : 3.16867e4 1755.04604

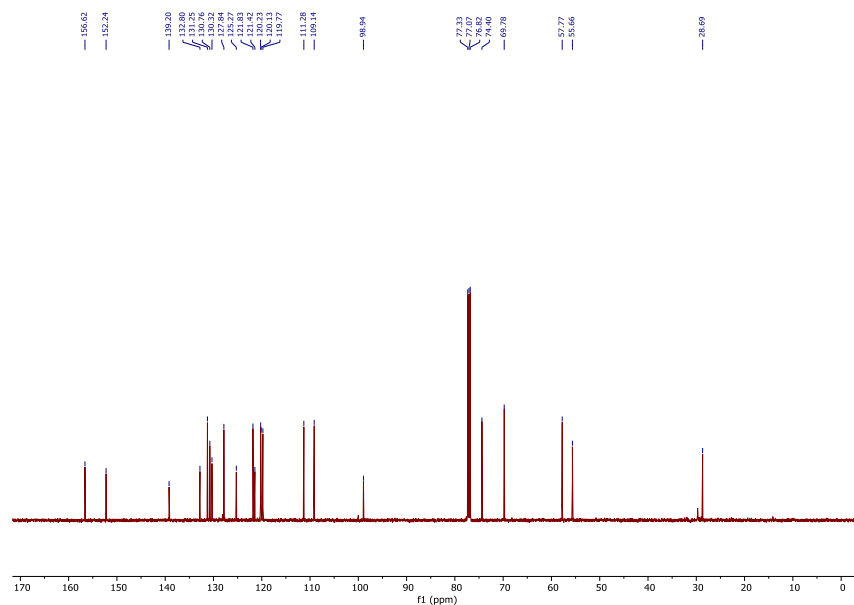
\*\*\* End of Report \*\*\*

## HPLC data of (-)-5f





$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5g**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5g**

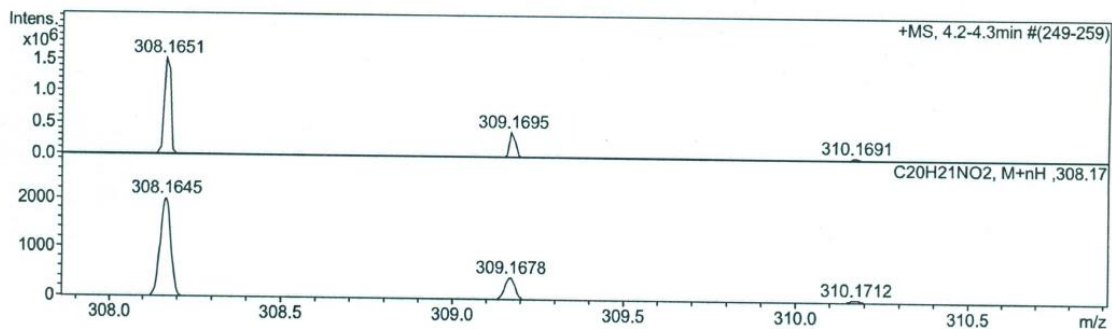
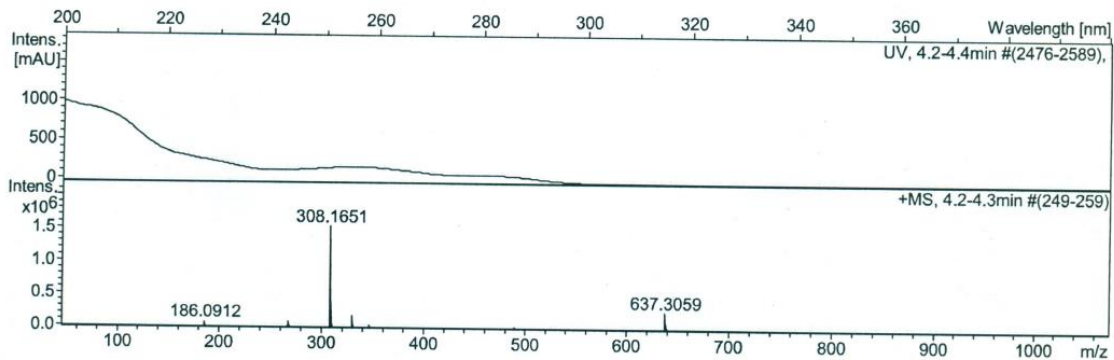
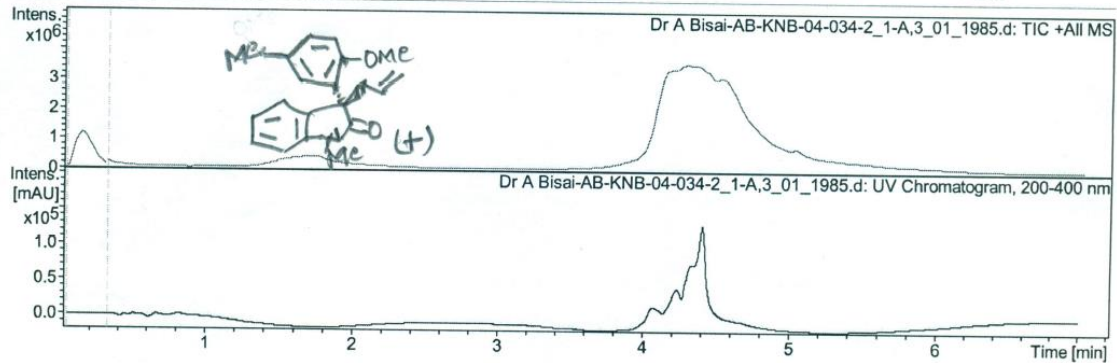
## Display Report

## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\09 JUNE\Dr A Bisai-AB-KNB-04-034-2\_1-A,3\_01\_1985.d Acquisition Date 6/9/2017 2:45:53 PM  
 Method hrlcms\_pos\_mid\_tunemix.m Operator RUCHI SHRIVASTAVA  
 Sample Name Dr A Bisai-AB-KNB-04-034-2 Instrument micrOTOF-Q II 10330  
 Comment

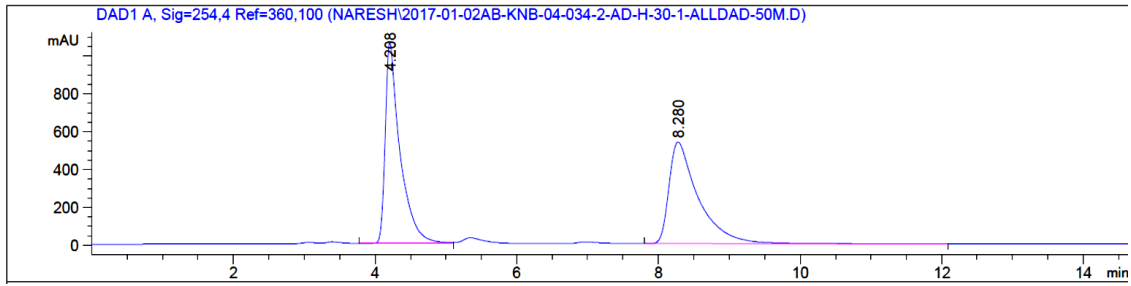
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (+)-5g

Data File C:\CHEM32\1\DATA\NARESH\2017-01-02AB-KNB-04-034-2-AD-H-30-1-ALLDAD-50M.D  
 Sample Name: AB-KNB-04-034-2-AD-H-30-1-ALLDAD-50M



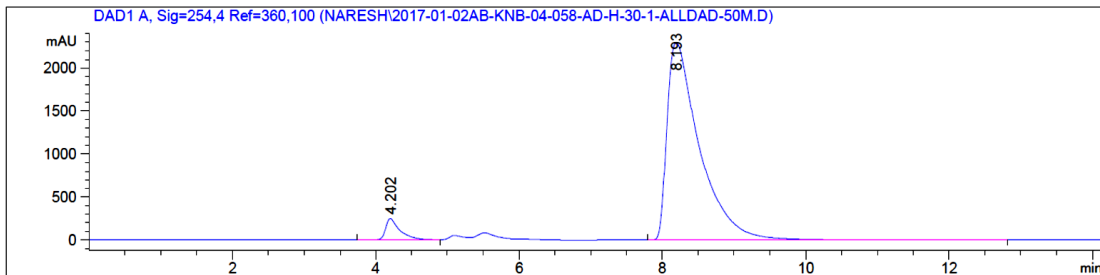
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.208	BV	0.2006	1.53703e4	1063.69080	49.9624
2	8.280	BB	0.4080	1.53935e4	537.75537	50.0376

Totals : 3.07638e4 1601.44617

HPLC data of (±)-5g

Data File C:\CHEM32\1\DATA\NARESH\2017-01-02AB-KNB-04-058-AD-H-30-1-ALLDAD-50M.D  
 Sample Name: AB-KNB-04-058-AD-H-30-1-ALLDAD-50M

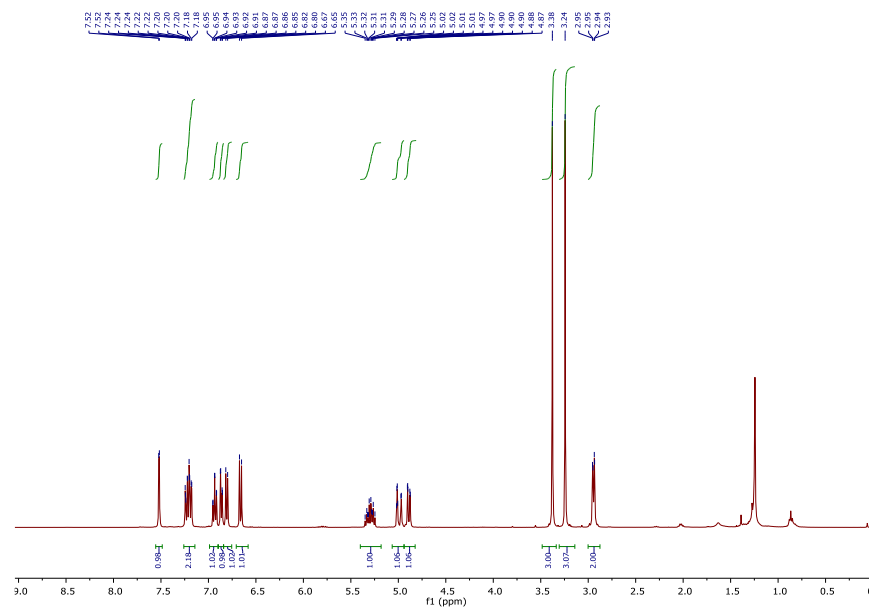
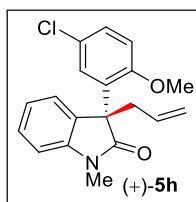


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

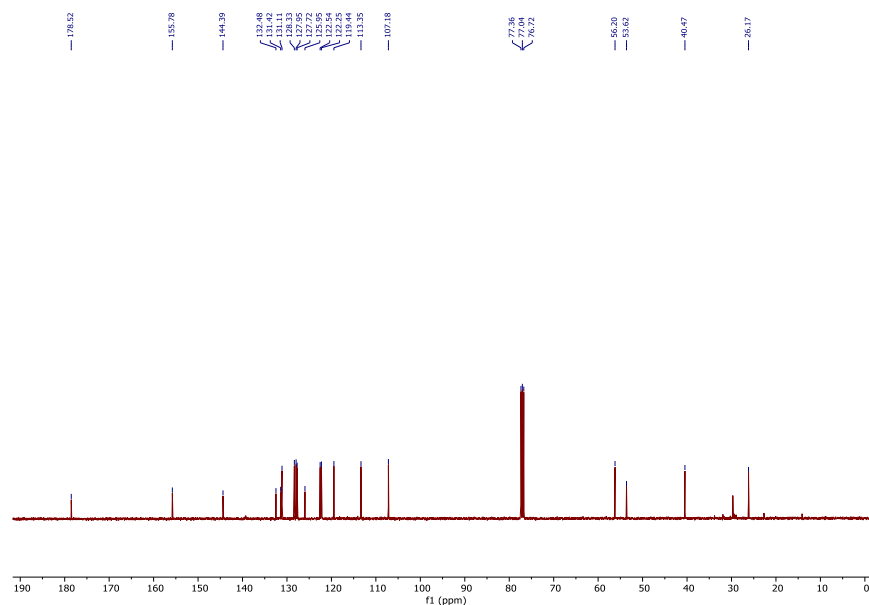
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.202	BB	0.1928	3386.71289	245.65765	4.4902
2	8.193	BB	0.4608	7.20378e4	2291.38867	95.5098

Totals : 7.54245e4 2537.04633

HPLC data of (+)-5g



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5h**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5h**

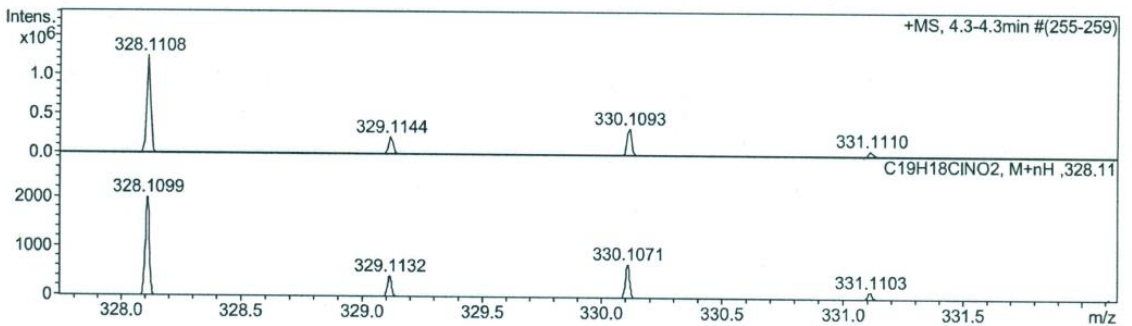
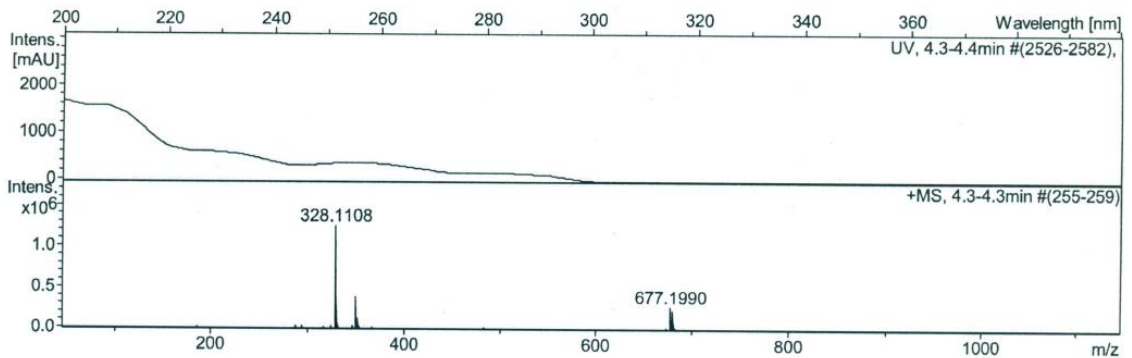
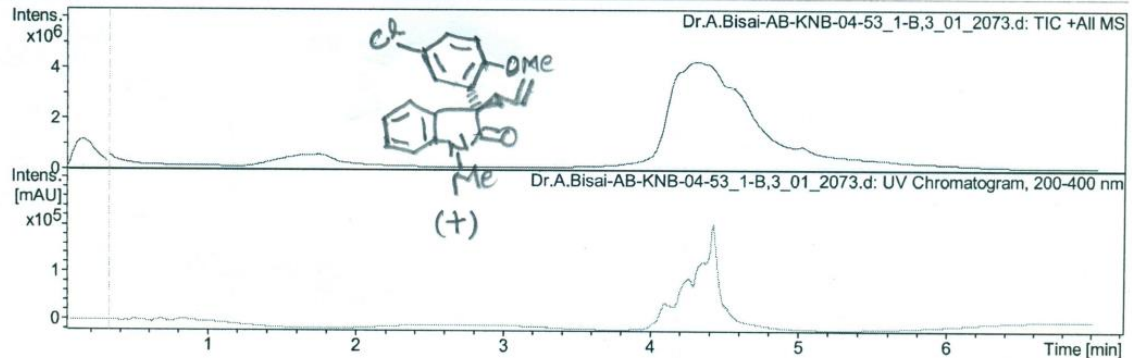
## Display Report

## Analysis Info

Analysis Name	D:\Data\user data\2017\JUNE 2017\14 June\Dr.A.Bisai-AB-KNB-04-53_1-B,3_01_2073.d	Acquisition Date	6/14/2017 1:04:07 PM
Method	hrlcms_pos_mid_tunemix.m	Operator	RUCHI SHRIVASTAVA
Sample Name	Dr.A.Bisai-AB-KNB-04-53	Instrument	micrOTOF-Q II 10330
Comment			

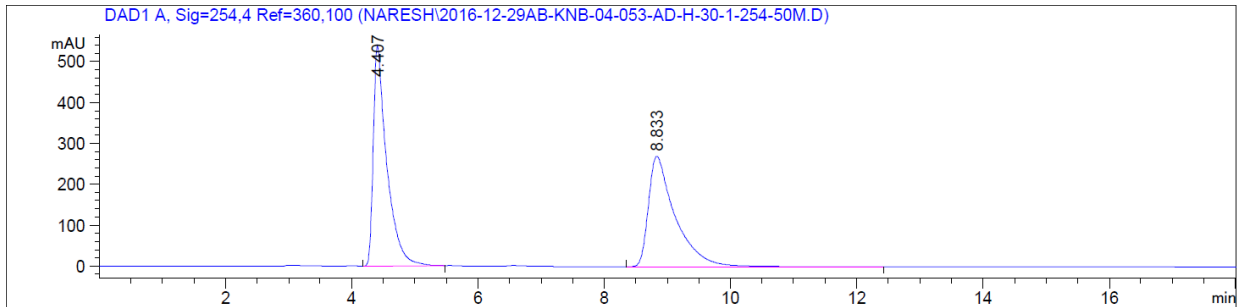
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5h**

Data File C:\CHEM32\1\DATA\NARESH\2016-12-29AB-KNB-04-053-AD-H-30-1-254-50M.D

Sample Name: AB-KNB-04-053-AD-H-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

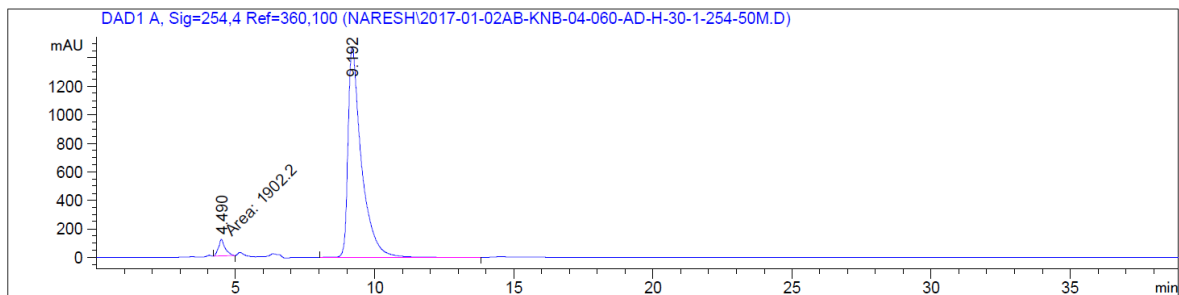
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.407	BB	0.2064	7975.66406	539.84253	50.1261
2	8.833	BB	0.4167	7935.55029	270.11868	49.8739

Totals : 1.59112e4 809.96121

## HPLC data of (±)-5h

Data File C:\CHEM32\1\DATA\NARESH\2017-01-02AB-KNB-04-060-AD-H-30-1-254-50M.D

Sample Name: AB-KNB-04-060-AD-H-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.490	MM	0.2711	1902.19641	116.96391	3.7157
2	9.192	BB	0.4823	4.92918e4	1473.56006	96.2843

Totals : 5.11940e4 1590.52397

## HPLC data of (+)-5h



## Display Report

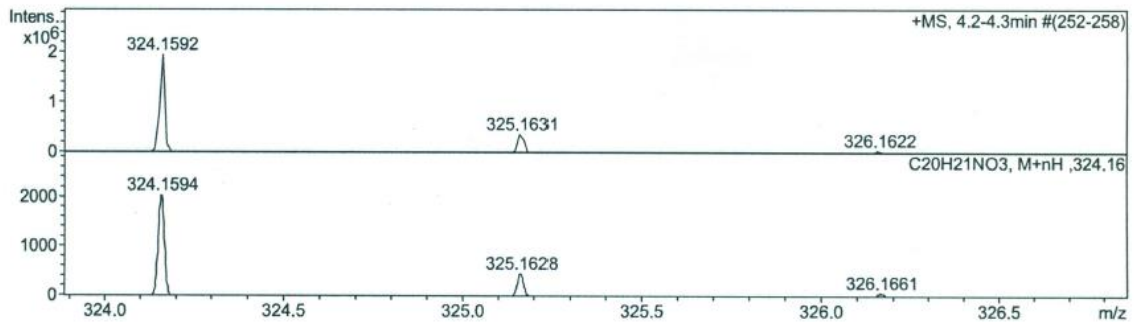
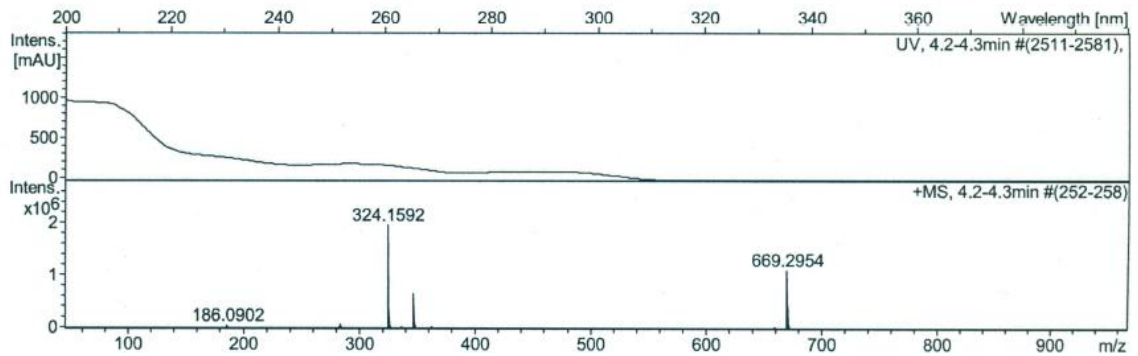
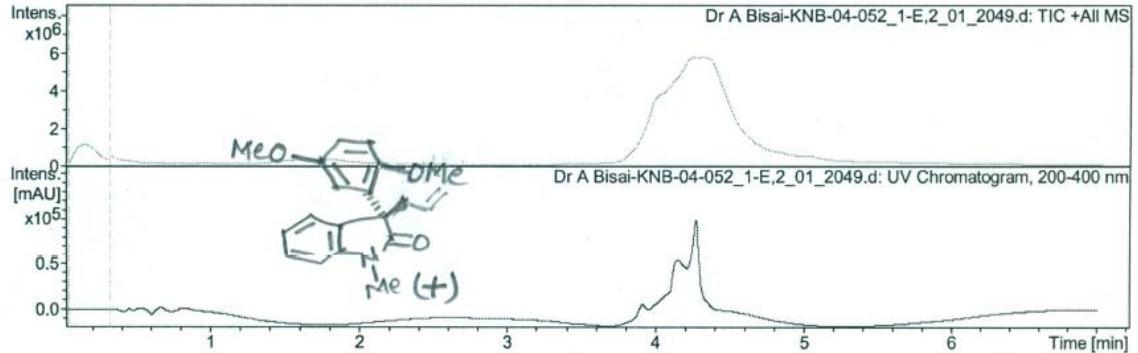
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\13 june\Dr A Bisai-KNB-04-052\_1-E,2\_01\_2049.d  
 Method hrlcms\_pos\_mid\_tunemix.m  
 Sample Name Dr A Bisai-KNB-04-052  
 Comment

Acquisition Date 6/13/2017 3:27:17 PM  
 Operator RUCHI SHRIVASTAVA  
 Instrument micrOTOF-Q II 10330

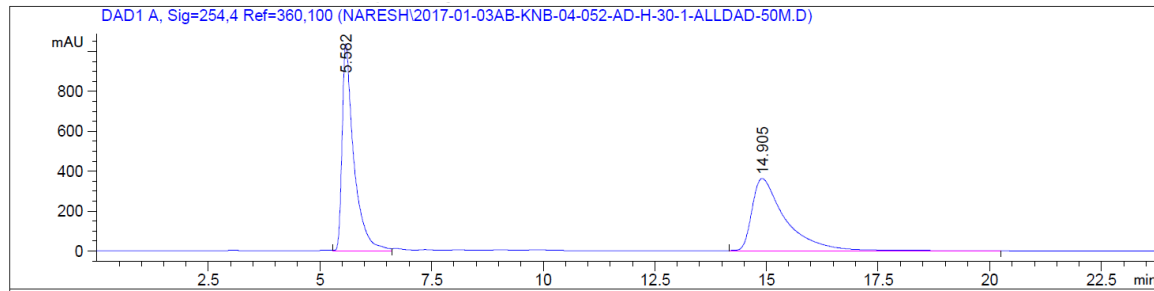
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste





Data File C:\CHEM32\1\DATA\NARESH\2017-01-03AB-KNB-04-052-AD-H-30-1-ALLDAD-50M.D  
 Sample Name: AB-KNB-04-052-AD-H-30-1-ALLDAD-50M



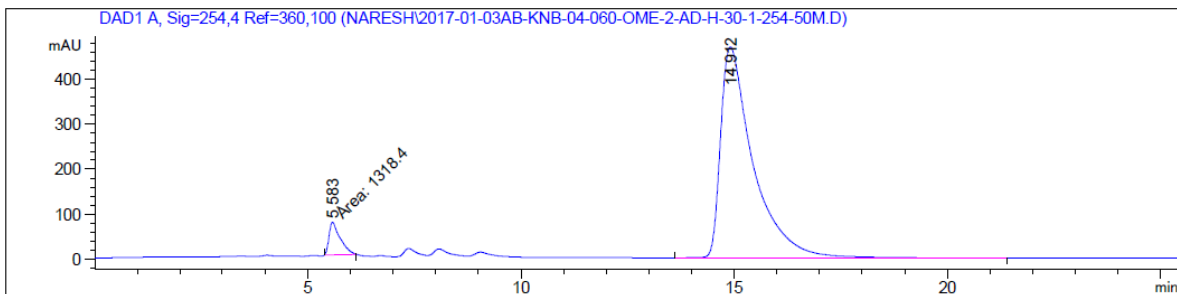
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.582	VV	0.2578	1.88038e4	1035.96680	50.3399
2	14.905	BB	0.7437	1.85499e4	360.88019	49.6601

Totals : 3.73538e4 1396.84698

HPLC data of (±)-5i

Data File C:\CHEM32\1\DATA\NARESH\2017-01-03AB-KNB-04-060-OME-2-AD-H-30-1-254-50M.D  
 Sample Name: AB-KNB-04-060-OME-2-AD-H-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.583	MM	0.3018	1318.40088	72.80338	5.0551
2	14.912	BB	0.7671	2.47621e4	468.10709	94.9449

Totals : 2.60805e4 540.91046

HPLC data of (+)-5i



## Display Report

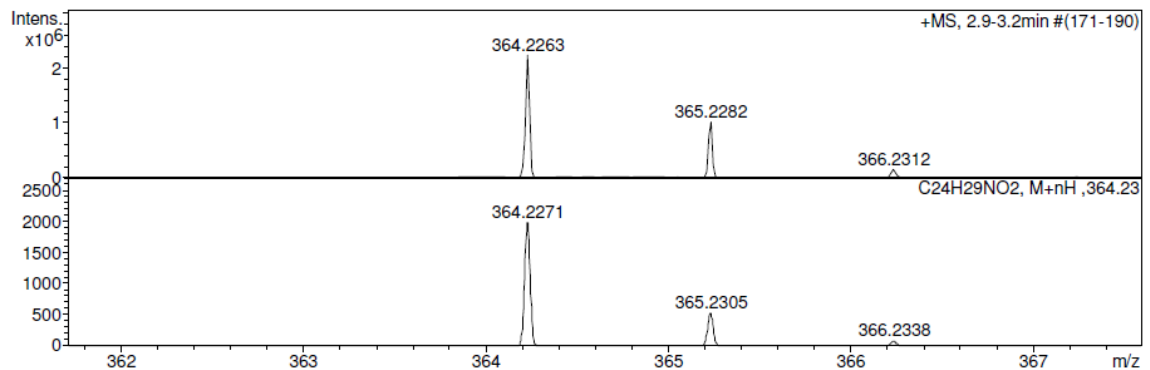
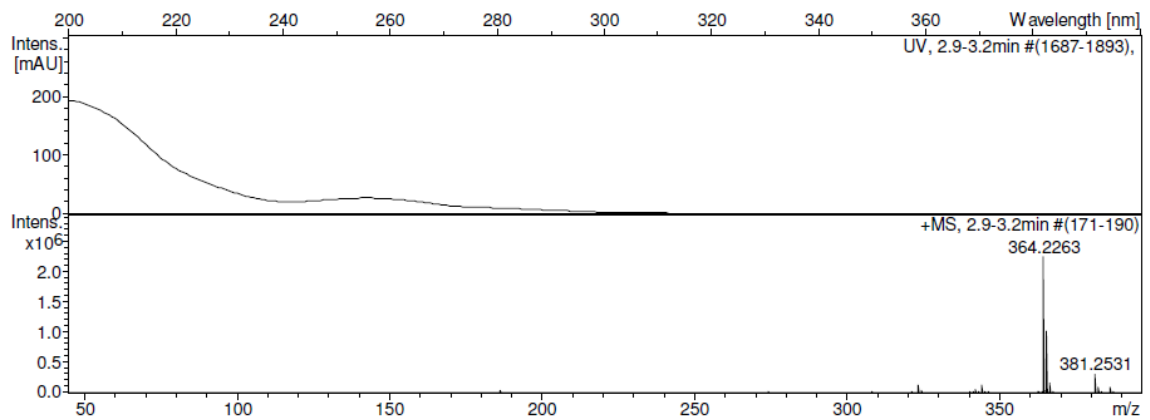
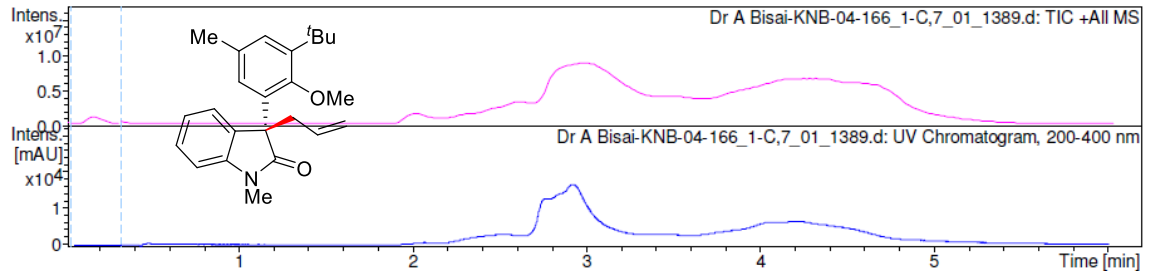
## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\26 mar\Dr A Bisai-KNB-04-166\_1-C,7\_01\_1389.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-KNB-04-166  
Comment

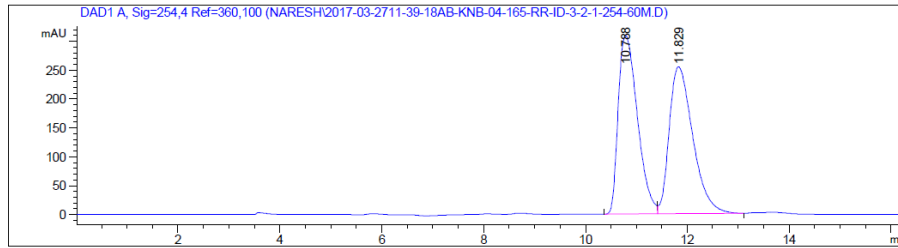
Acquisition Date 3/26/2018 1:14:36 PM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Data File C:\CHEM32\1\DATA\NARESH\2017-03-2711-39-18AB-KNB-04-165-RR-ID-3-2-1-254-60M.D  
 Sample Name: AB-KNB-04-165-RR-ID-3-2-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

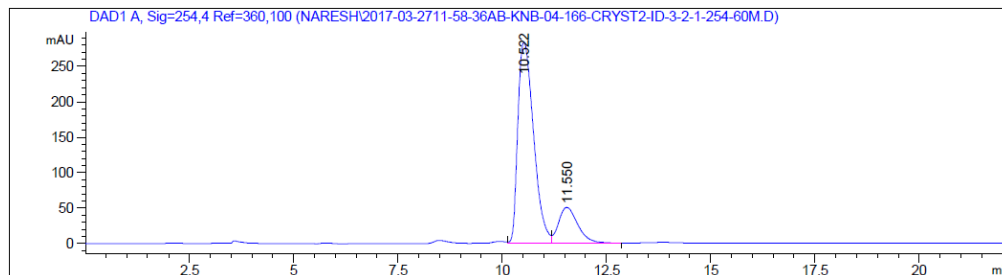
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.788	BV	0.4135	8094.69043	309.09021	49.6398
2	11.829	VB	0.5024	8212.16895	254.52612	50.3602

Totals : 1.63069e4 563.61633

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 \*\*\* End of Report \*\*\*

### HPLC data of (±)-5j

Data File C:\CHEM32\...TA\NARESH\2017-03-2711-58-36AB-KNB-04-166-CRYST2-ID-3-2-1-254-60M.D  
 Sample Name: AB-KNB-04-166-Cryst2-ID-3-2-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.522	WV	0.4133	7411.82178	283.27341	82.0706
2	11.550	VB	0.4944	1619.21118	50.45833	17.9294

Totals : 9031.03296 333.73174

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 \*\*\* End of Report \*\*\*

### HPLC data of (+)-5j



## Display Report

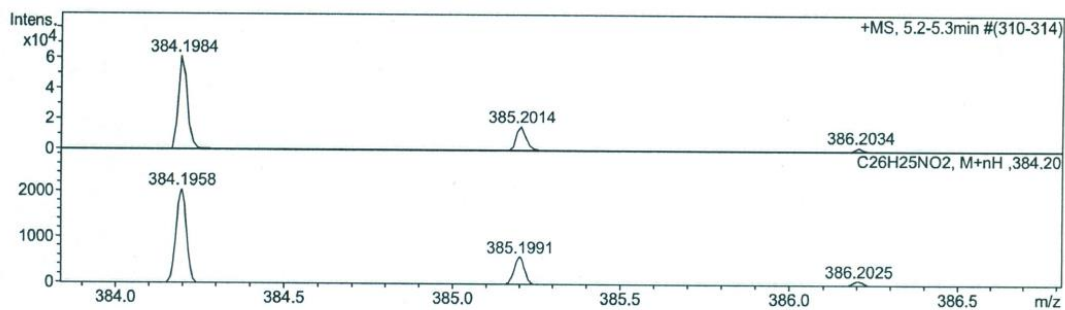
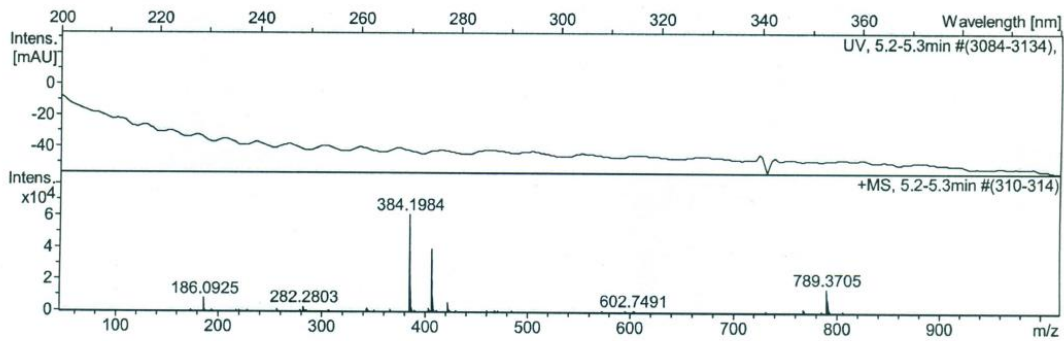
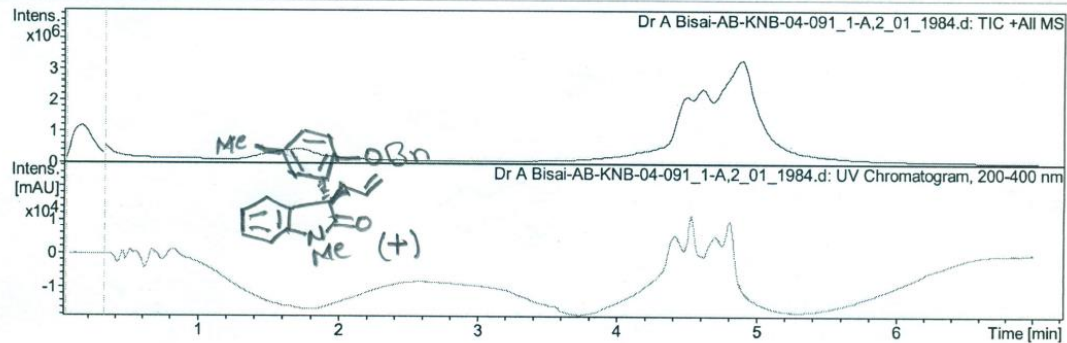
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\09 JUNE\Dr A Bisai-AB-KNB-04-091\_1-A,2\_01\_1984.d  
 Method hrlcms\_pos\_mid\_tunemix.m  
 Sample Name Dr A Bisai-AB-KNB-04-091  
 Comment

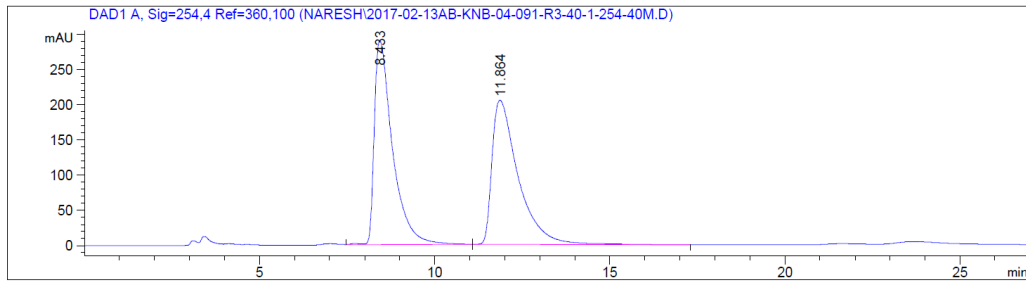
Acquisition Date 6/9/2017 2:37:44 PM  
 Operator RUCHI SHRIVASTAVA  
 Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5k**

Data File C:\CHEM32\1\DATA\NARESH\2017-02-13AB-KNB-04-091-R3-40-1-254-40M.D  
 Sample Name: AB-KNB-04-091-R3-40-1-254-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

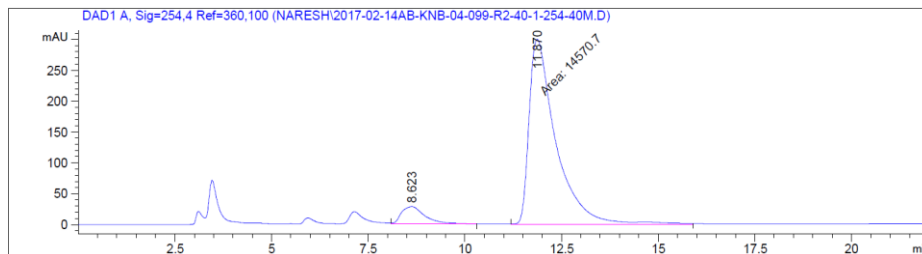
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.433	BB	0.5357	1.04627e4	289.43933	49.5718
2	11.864	BB	0.7729	1.06434e4	204.58707	50.4282

Totals : 2.11061e4 494.02640

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 \*\*\* End of Report \*\*\*

### HPLC data of (±)-5k

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-099-R2-40-1-254-40M.D  
 Sample Name: AB-KNB-04-099-R2-40-1-254-40M



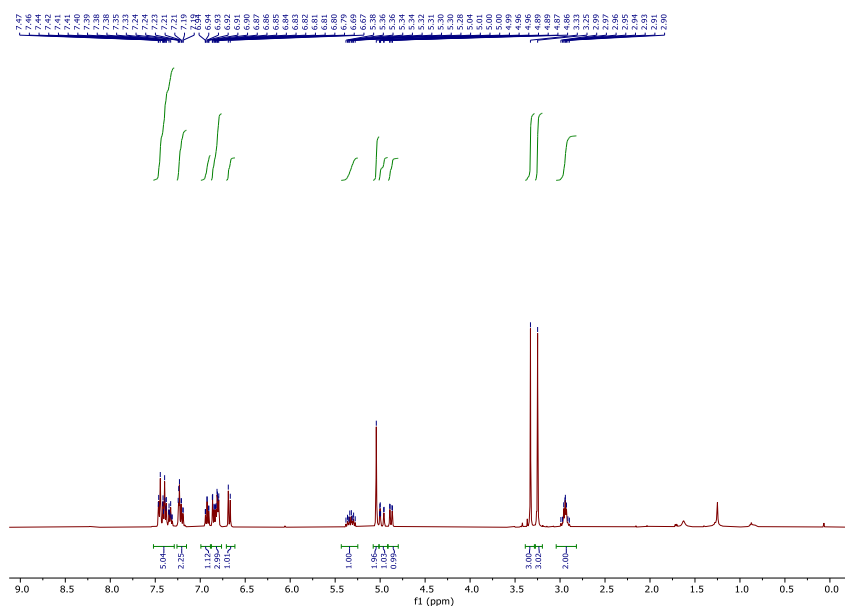
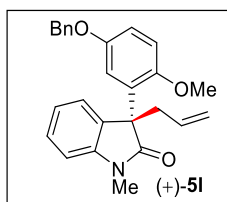
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.623	VB	0.6409	1165.56616	28.04263	7.4069
2	11.870	MM	0.8095	1.45707e4	299.97736	92.5931

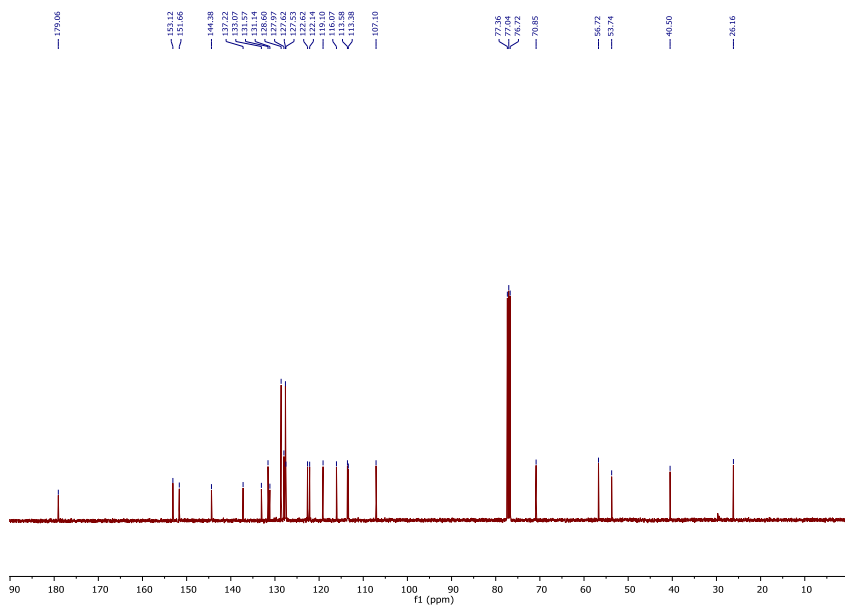
Totals : 1.57362e4 328.01999

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 \*\*\* End of Report \*\*\*

### HPLC data of (+)-5k



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5I**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5I**



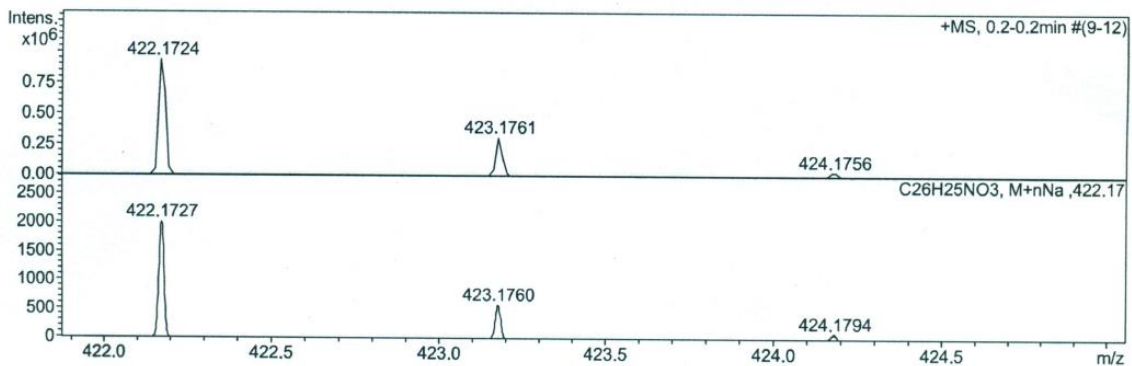
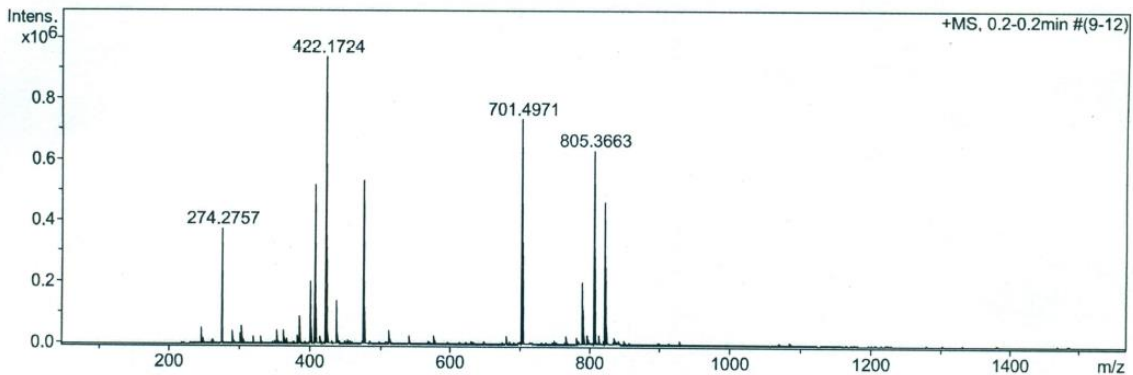
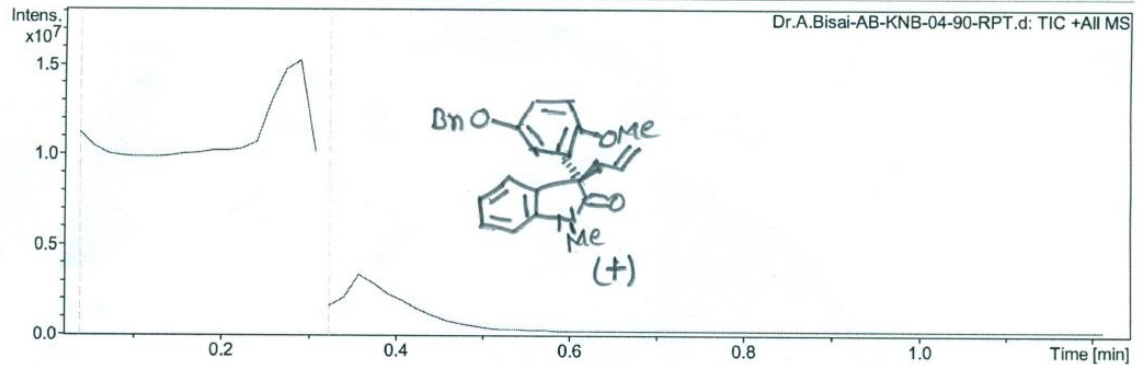
## Display Report

## Analysis Info

Analysis Name	D:\Data\user data\2017\JUNE 2017\15 june\Dr.A.Bisai-AB-KNB-04-90-RPT.d	Acquisition Date	6/15/2017 3:27:04 PM
Method	Pos_Mid_tunemix.m	Operator	RUCHI SHRIVASTAVA
Sample Name	AB-KNB-04-90-RPT	Instrument	micrOTOF-Q II 10330
Comment			

## Acquisition Parameter

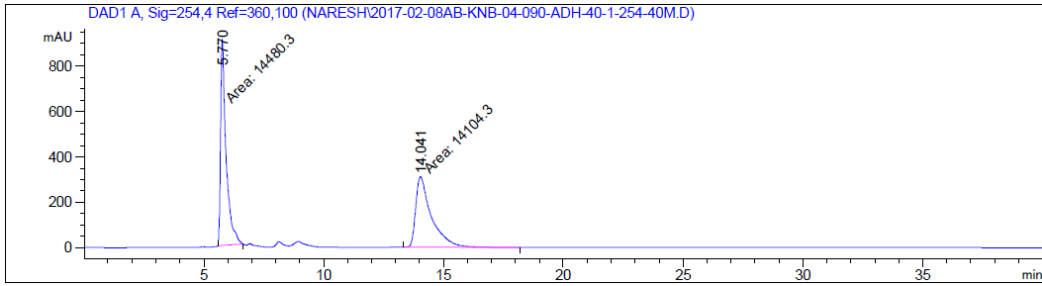
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (+)-51

Data File C:\CHEM32\1\DATA\NARESH\2017-02-08AB-KNB-04-090-ADH-40-1-254-40M.D

Sample Name: AB-KNB-04-090-ADH-40-1-254-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.770	MM	0.2658	1.44803e4	908.10840	50.6577
2	14.041	MM	0.7575	1.41043e4	310.32068	49.3423

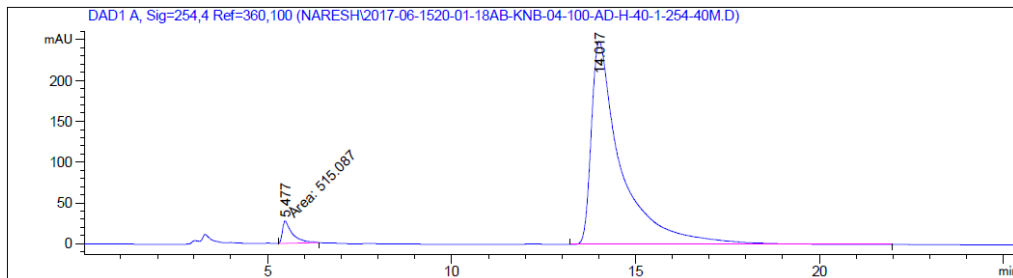
Totals : 2.85847e4 1218.42908

\*\*\* End of Report \*\*\*

## HPLC data of (±)-51

Data File C:\CHEM32\1\DATA\NARESH\2017-06-1520-01-18AB-KNB-04-100-AD-H-40-1-254-40M.D

Sample Name: AB-KNB-04-100-AD-H-40-1-254-40M



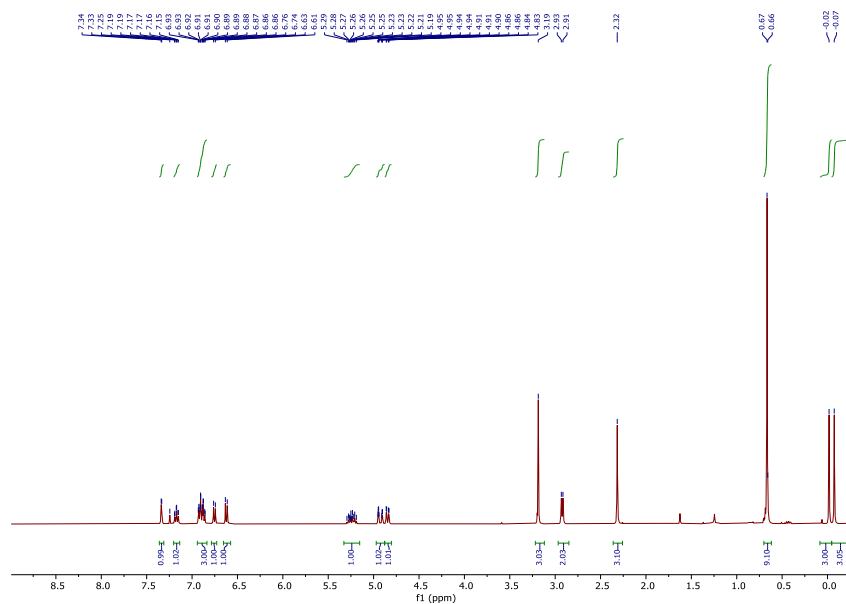
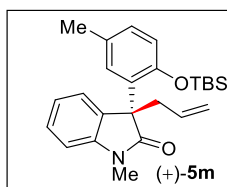
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.477	MM	0.3121	515.08655	27.50998	3.5338
2	14.017	BB	0.7855	1.40608e4	248.73813	96.4662

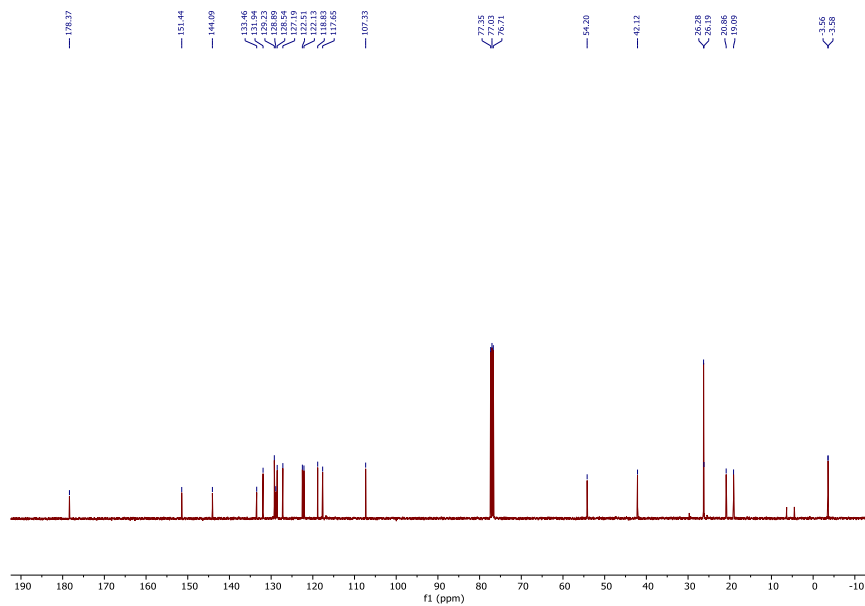
Totals : 1.45759e4 276.24810

\*\*\* End of Report \*\*\*

## HPLC data of (+)-51



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound (+)-5m



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound (+)-5m

## Display Report

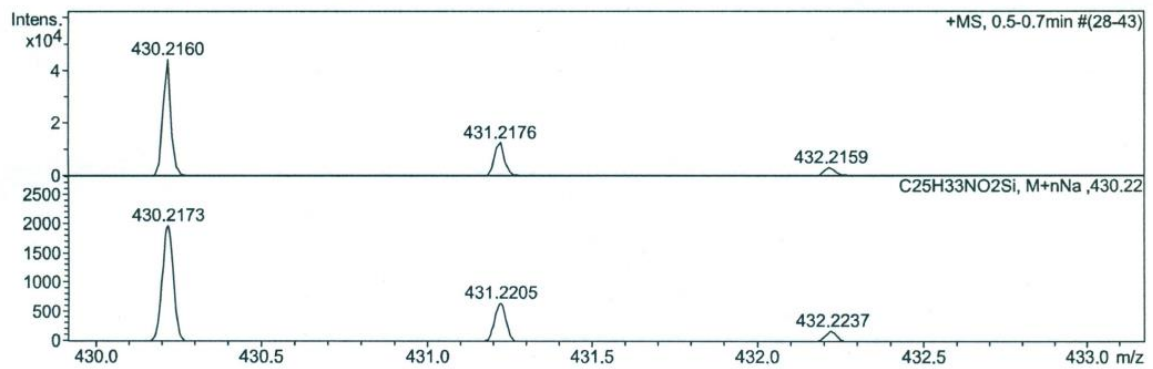
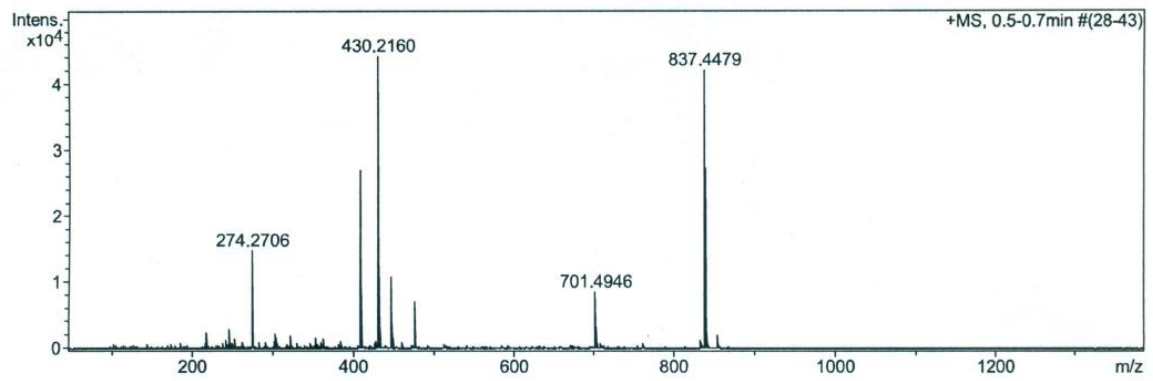
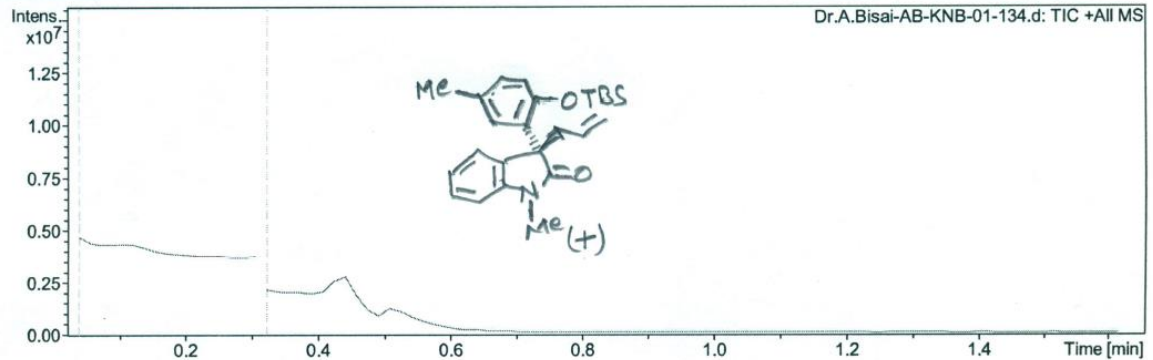
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\16 june\Dr.A.Bisai-AB-KNB-01-134.d  
Method Pos\_Mid\_tunemix.m  
Sample Name AB-KNB-01-134  
Comment

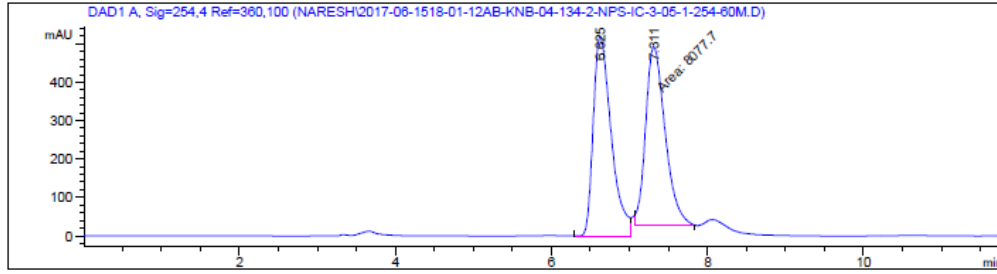
Acquisition Date 6/16/2017 4:33:36 PM  
Operator RUCHI SHRIVASTAVA  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5m**

Data File C:\CHEM32\...TA\NARESH\2017-06-1518-01-12AB-KNB-04-134-2-NPS-IC-3-05-1-254-60M.D  
 Sample Name: AB-KNB-04-134-2-NPS-IC-3-05-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

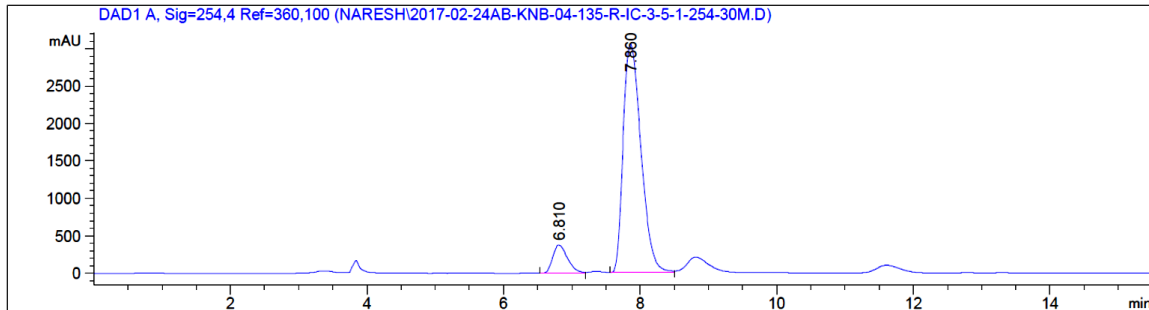
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.625	BV	0.2446	8311.57715	519.60382	50.7135
2	7.311	MM	0.2898	8077.69971	464.60922	49.2865

Totals : 1.63893e4 984.21304

\*\*\* End of Report \*\*\*

### HPLC data of (±)-5m

Data File C:\CHEM32\1\DATA\NARESH\2017-02-24AB-KNB-04-135-R-IC-3-5-1-254-30M.D  
 Sample Name: AB-KNB-04-135-R-IC-3-5-1-254-30M



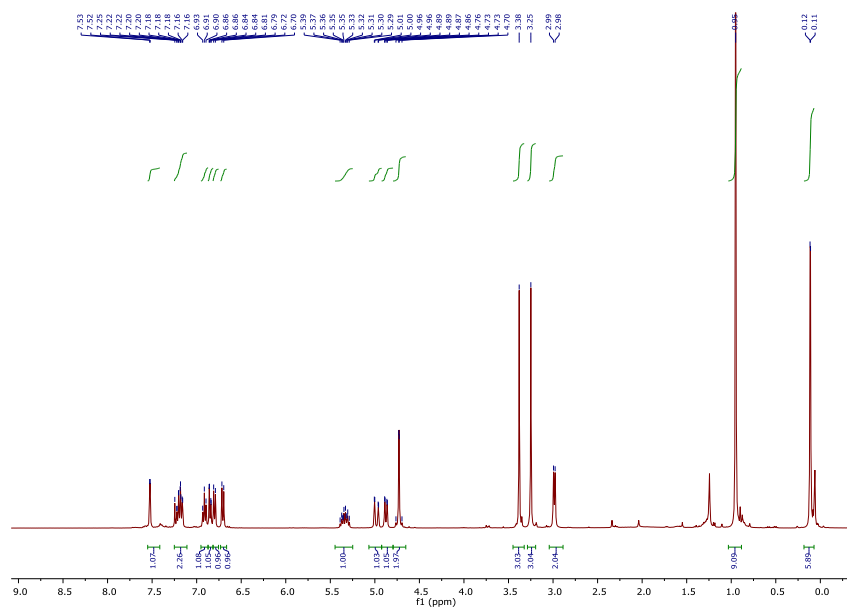
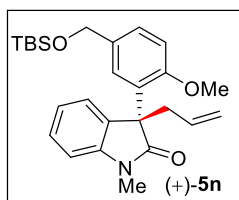
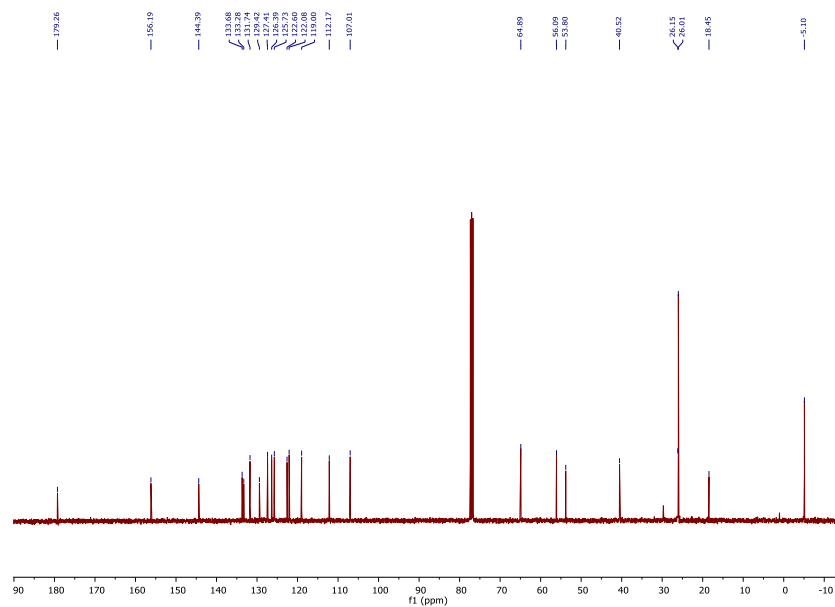
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.810	BV	0.2292	5558.22168	374.46814	9.1051
2	7.860	BV	0.2839	5.54872e4	3055.65625	90.8949

Totals : 6.10454e4 3430.12439

\*\*\* End of Report \*\*\*

### HPLC data of (+)-5m

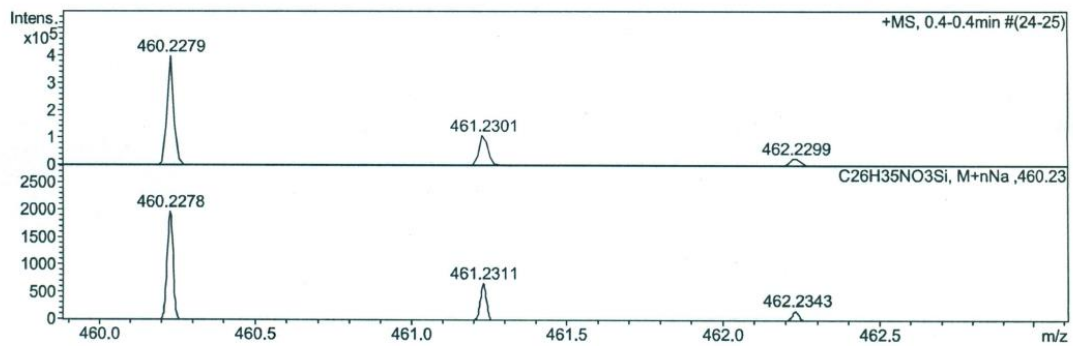
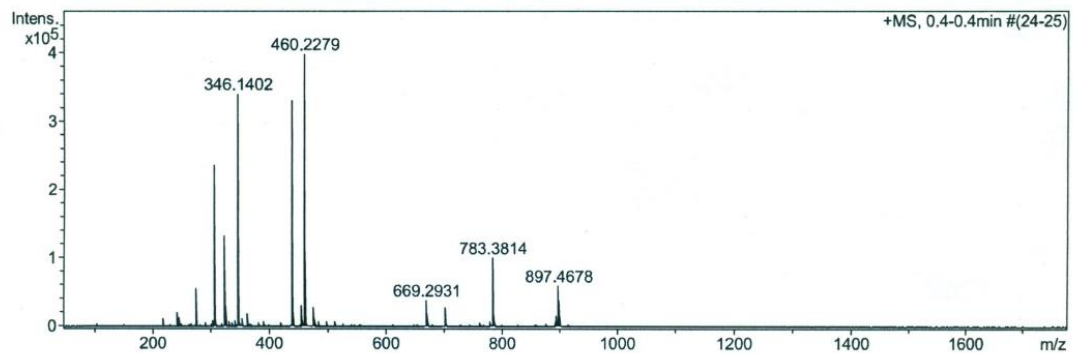
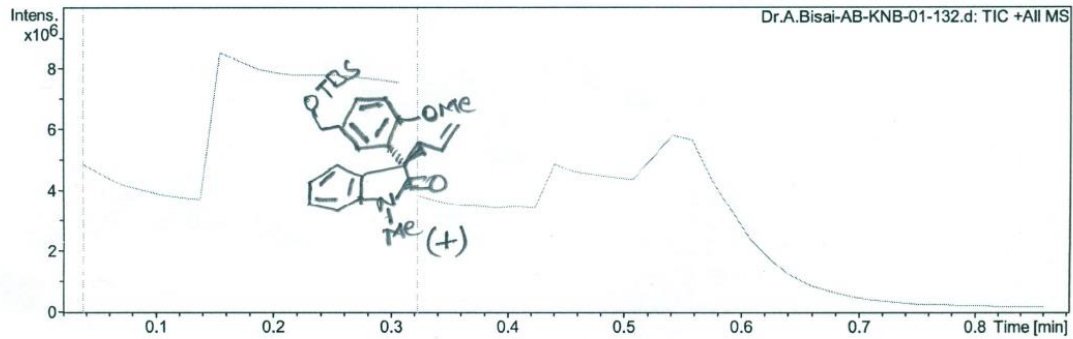
 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-5n $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-5n

## Display Report

<b>Analysis Info</b>		Acquisition Date	6/16/2017 4:29:31 PM
Analysis Name	D:\Data\user data\2017\JUNE 2017\16 june\Dr.A.Bisai-AB-KNB-01-132.d	Operator	RUCHI SHRIVASTAVA
Method	Pos_Mid_tunemix.m	Instrument	micrOTOF-Q II 10330
Sample Name	AB-KNB-01-132		
Comment			

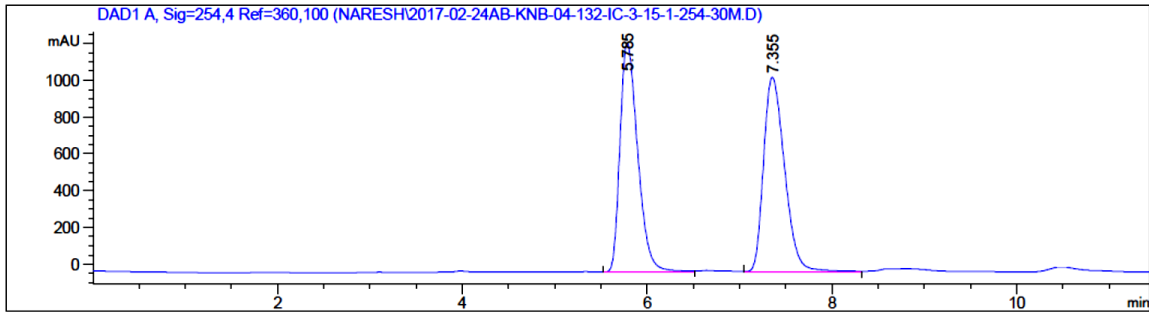
**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5n**

Data File C:\CHEM32\1\DATA\NARESH\2017-02-24AB-KNB-04-132-IC-3-15-1-254-30M.D

Sample Name: AB-KNB-04-132-IC-3-15-1-254-30M

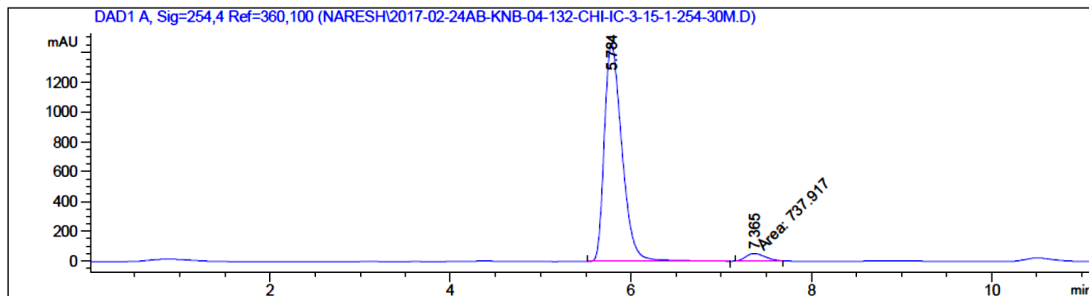


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.785	BV	0.2057	1.65776e4	1242.45691	49.9095
2	7.355	VB	0.2477	1.66377e4	1056.90479	50.0905

Totals : 3.32153e4 2299.36169

HPLC data of (±)-5n



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

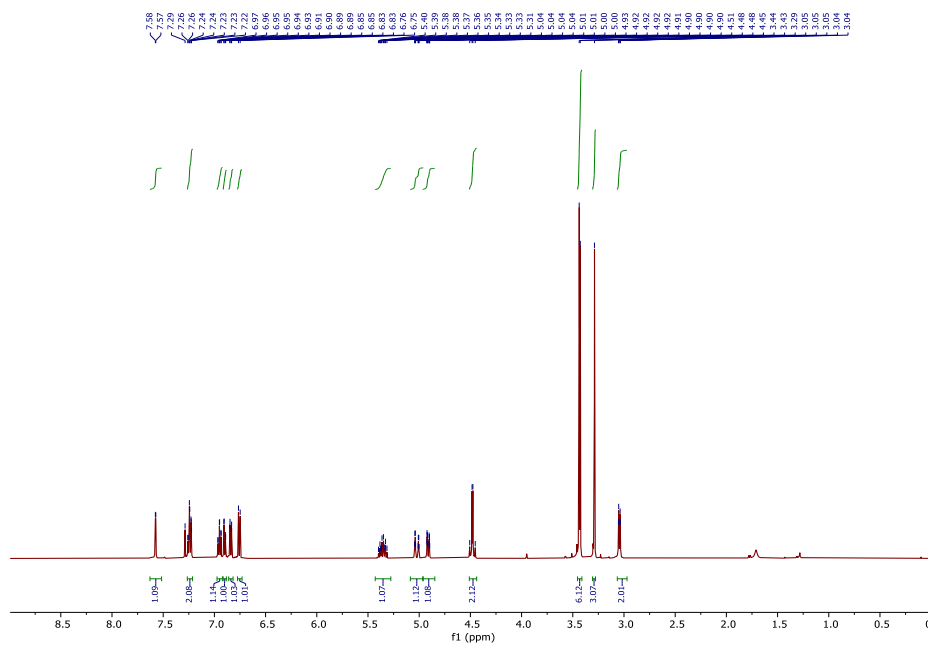
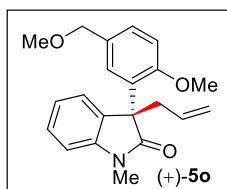
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.784	BV	0.2090	1.98198e4	1454.33374	96.4105
2	7.365	MM	0.2466	737.91748	49.86928	3.5895

Totals : 2.05577e4 1504.20302

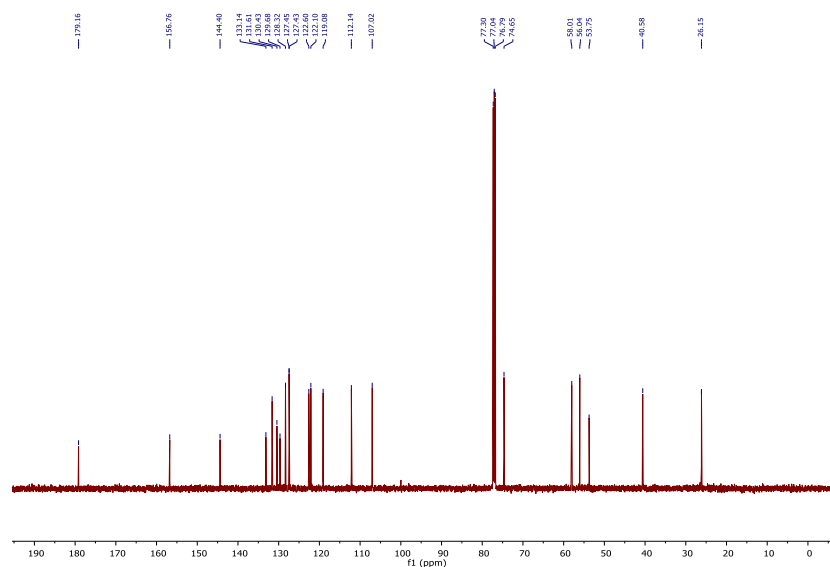
\*\*\* End of Report \*\*\*

HPLC data of (+)-5n





$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5o**



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5o**

## Display Report

## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\15 june\Dr A Bisai-AB-KNB-04-38R\_1-C,3\_01\_2107.d  
 Method hrlcms\_pos\_mid\_tunemix.m  
 Sample Name Dr A Bisai-AB-KNB-04-38R  
 Comment

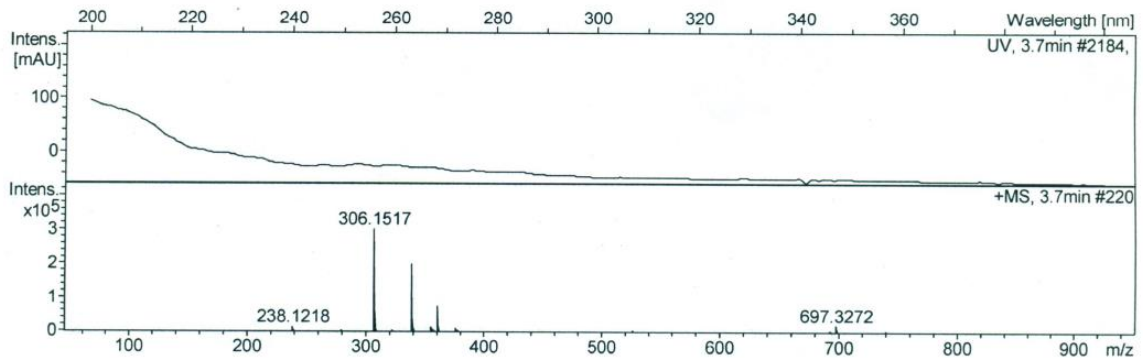
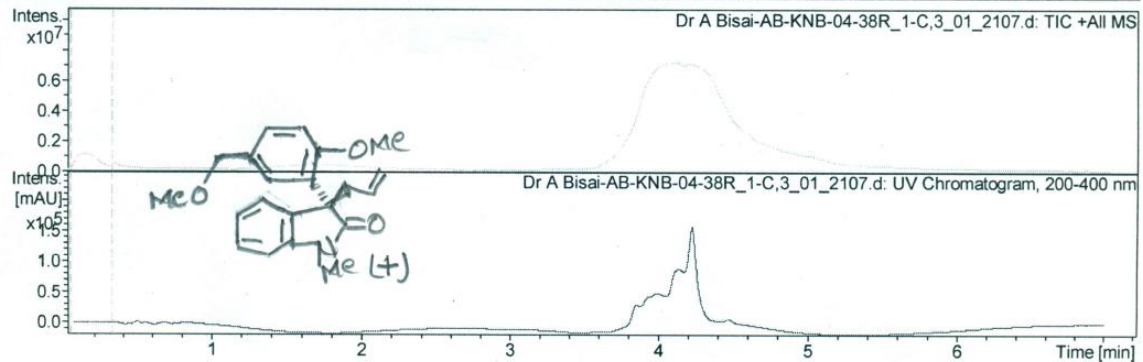
Acquisition Date 6/15/2017 2:50:56 PM

Operator RUCHI SHRIVASTAVA

Instrument micrOTOF-Q II 10330

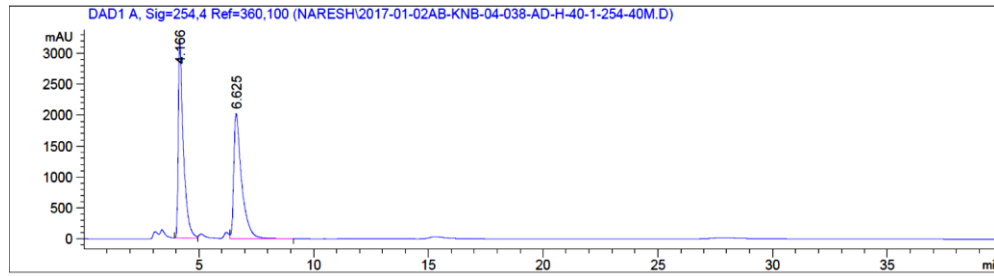
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **50**

Data File C:\CHEM32\1\DATA\NARESH\2017-01-02AB-KNB-04-038-AD-H-40-1-254-40M.D

Sample Name: AB-KNB-04-038-AD-H-40-1-254-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

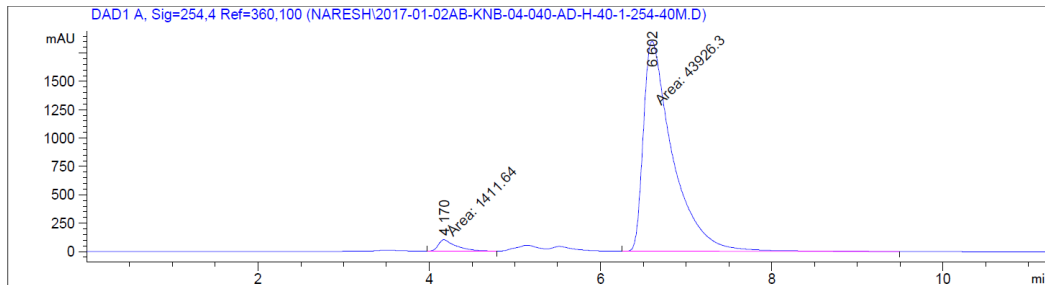
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.166	BV	0.2144	4.91079e4	3210.89624	50.5198
2	6.625	VB	0.3410	4.80972e4	2025.46716	49.4802

Totals : 9.72051e4 5236.36340

## HPLC data of (±)-5o

Data File C:\CHEM32\1\DATA\NARESH\2017-01-02AB-KNB-04-040-AD-H-40-1-254-40M.D

Sample Name: AB-KNB-04-040-AD-H-40-1-254-40M



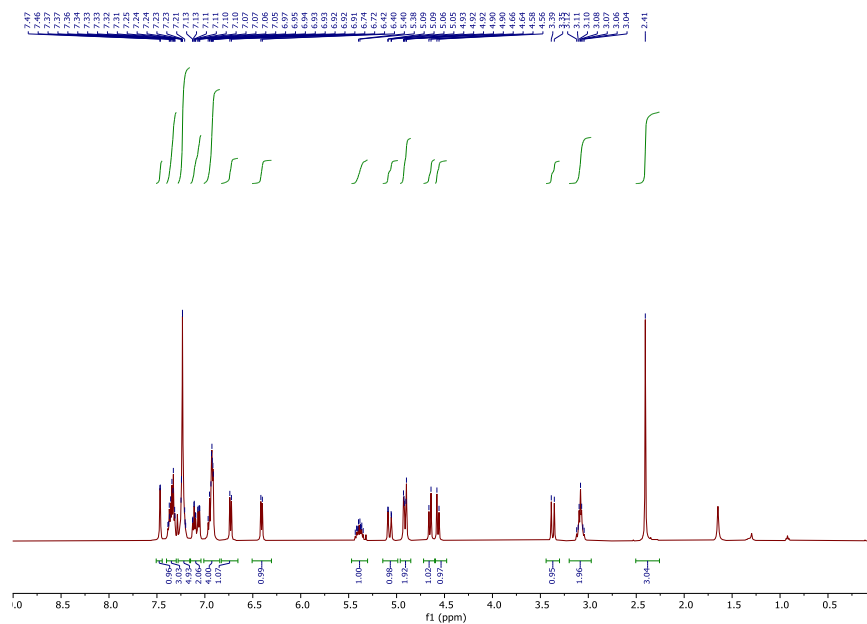
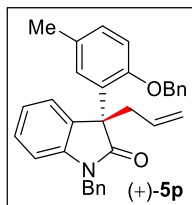
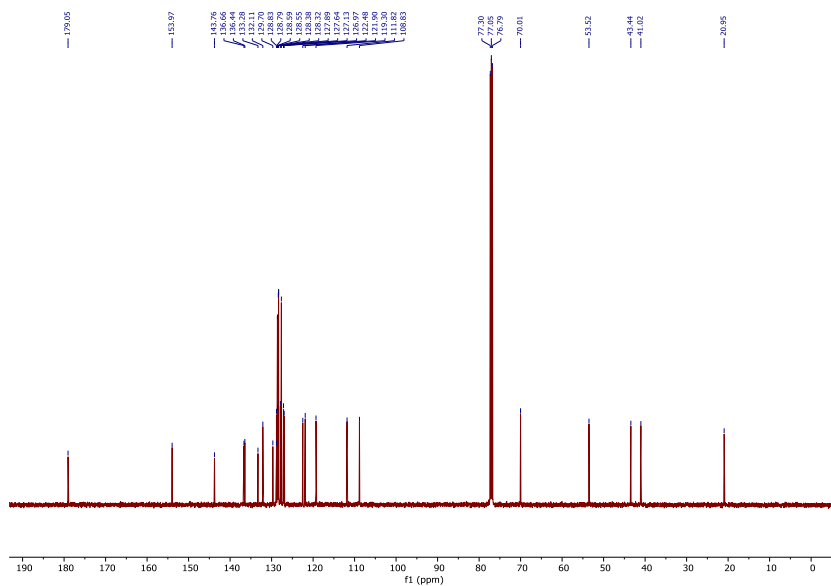
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.170	MM	0.2359	1411.64465	99.72189	3.1136
2	6.602	MM	0.3949	4.39263e4	1853.67590	96.8864

Totals : 4.53379e4 1953.39780

\*\*\* End of Report \*\*\*

## HPLC data of (+)-5o

 $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5p** $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (+)-**5p**

## Display Report

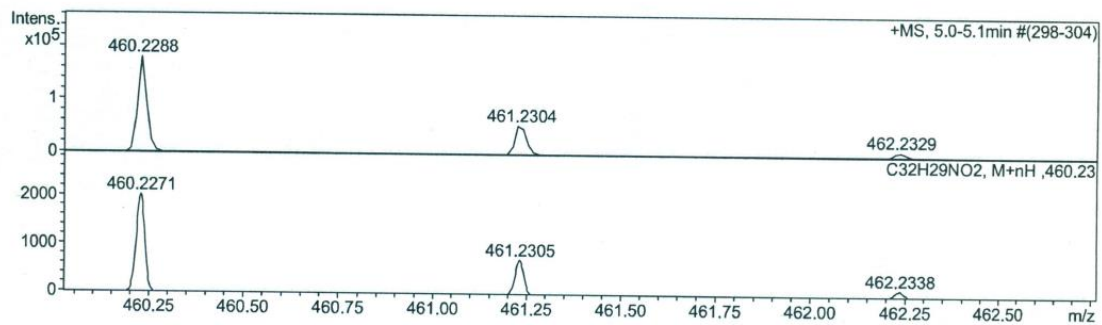
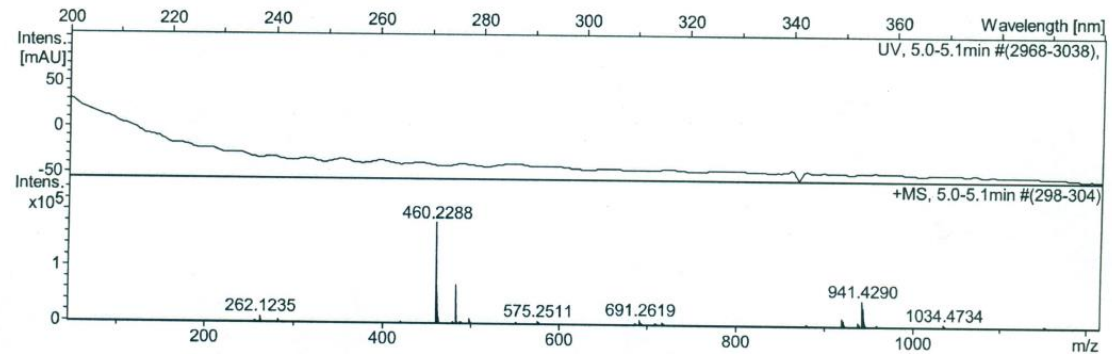
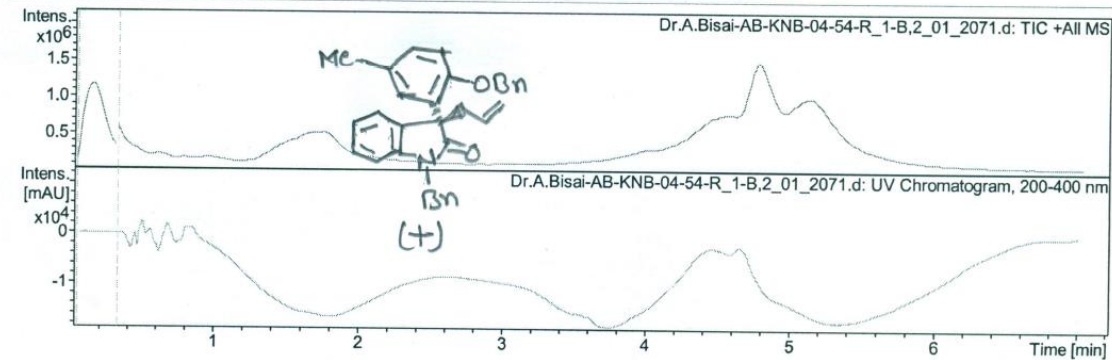
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\14 June\Dr.A.Bisai-AB-KNB-04-54-R\_1-B,2\_01\_2071.d  
 Method hrlcms\_pos\_mid\_tunemix.m  
 Sample Name Dr.A.Bisai-AB-KNB-04-54-R  
 Comment

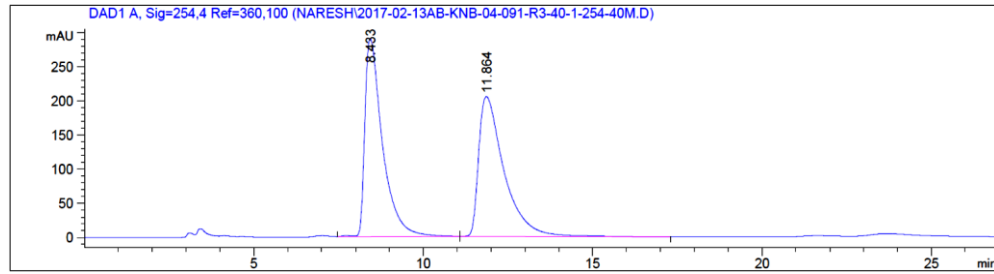
Acquisition Date 6/14/2017 12:47:47 PM  
 Operator RUCHI SHRIVASTAVA  
 Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Data File C:\CHEM32\1\DATA\NARESH\2017-02-13AB-KNB-04-091-R3-40-1-254-40M.D  
 Sample Name: AB-KNB-04-091-R3-40-1-254-40M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

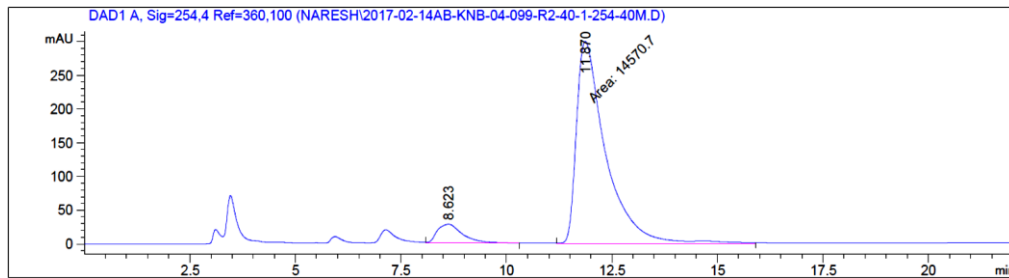
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.433	BB	0.5357	1.04627e4	289.43933	49.5718
2	11.864	BB	0.7729	1.06434e4	204.58707	50.4282

Totals : 2.11061e4 494.02640

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

### HPLC data of (±)-5p

Data File C:\CHEM32\1\DATA\NARESH\2017-02-14AB-KNB-04-099-R2-40-1-254-40M.D  
 Sample Name: AB-KNB-04-099-R2-40-1-254-40M

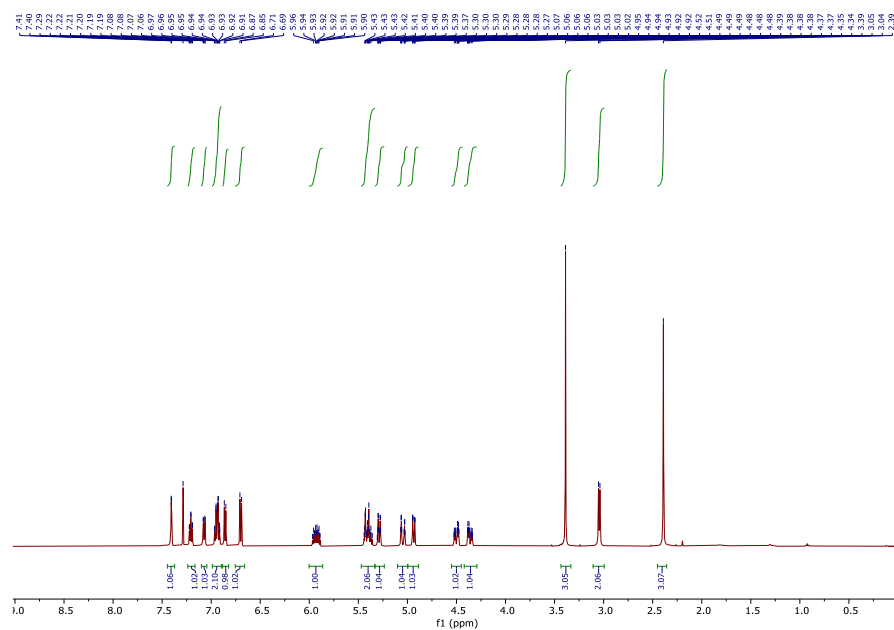
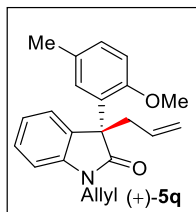


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

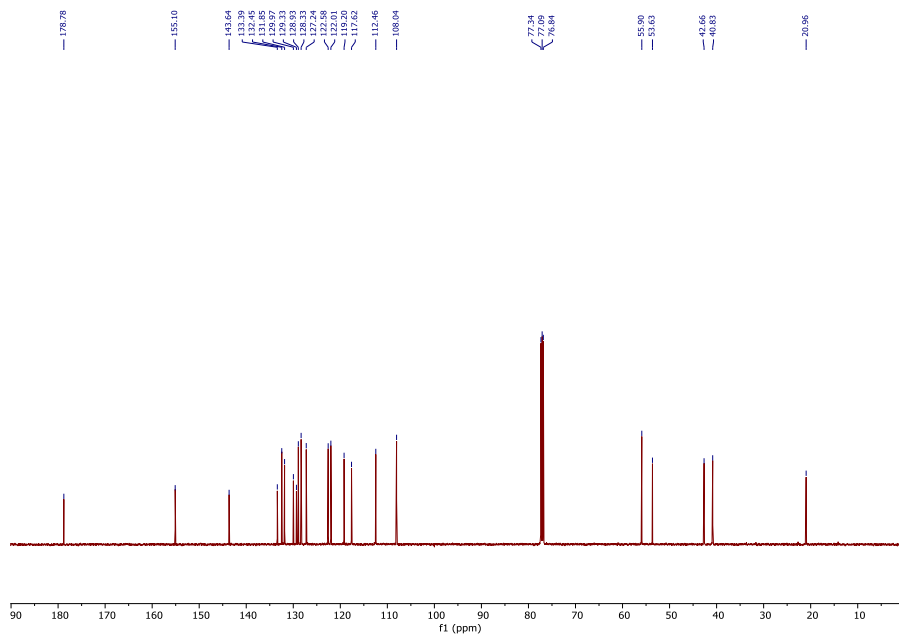
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.623	VB	0.6409	1165.56616	28.04263	7.4069
2	11.870	MM	0.8095	1.45707e4	299.97736	92.5931

Totals : 1.57362e4 328.01999

### HPLC data of (+)-5p



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-5q



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) of compound (+)-5q

## Display Report

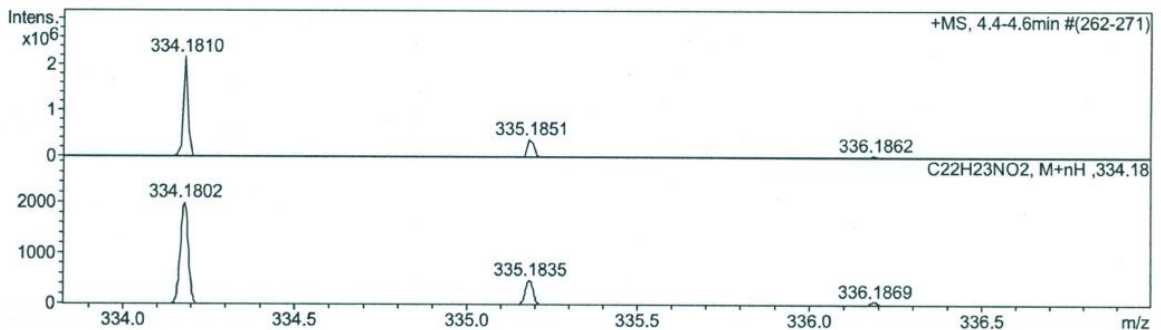
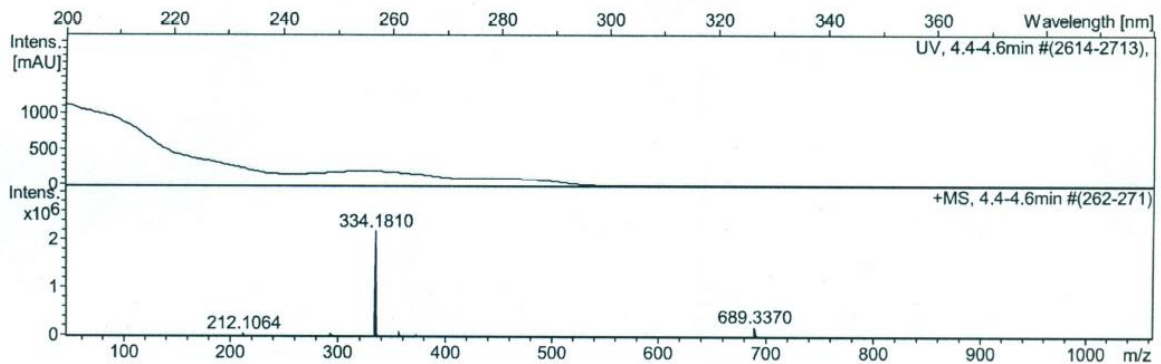
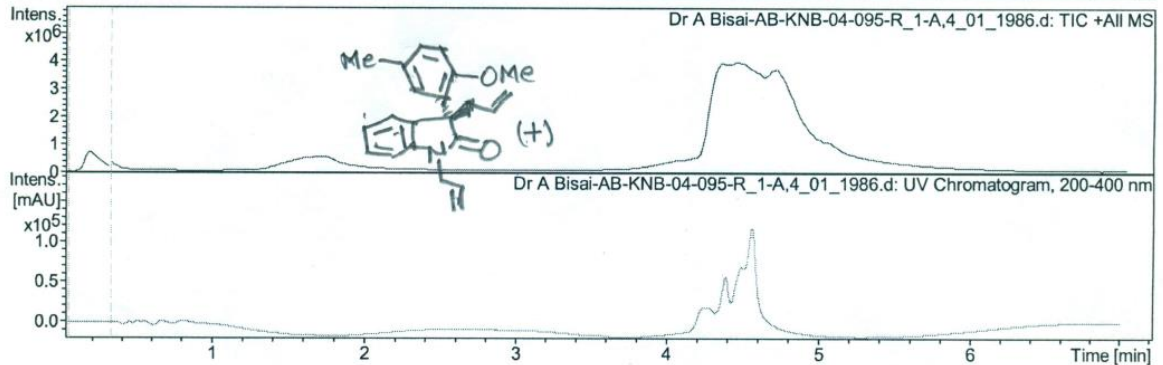
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\09 JUNE\Dr A Bisai-AB-KNB-04-095-R\_1-A,4\_01\_1986.d  
 Method hrlcms\_pos\_mid\_tunemix.m  
 Sample Name Dr A Bisai-AB-KNB-04-095-R  
 Comment

Acquisition Date 6/9/2017 2:54:02 PM  
 Operator RUCHI SHRIVASTAVA  
 Instrument micrOTOF-Q II 10330

## Acquisition Parameter

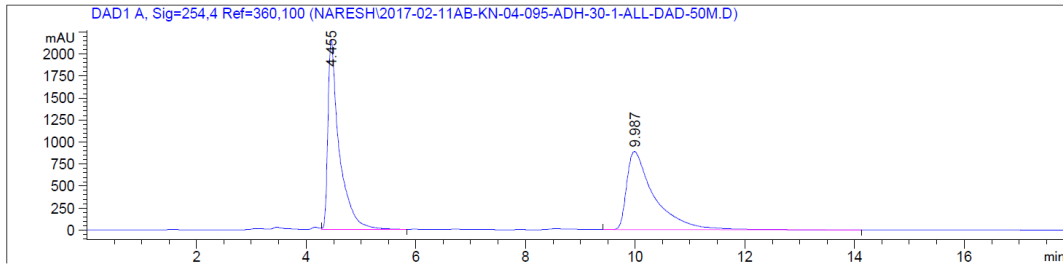
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste





Data File C:\CHEM32\1\DATA\NARESH\2017-02-11AB-KN-04-095-ADH-30-1-ALL-DAD-50M.D

Sample Name: AB-KN-04-095-ADH-30-1-ALL-DAD-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

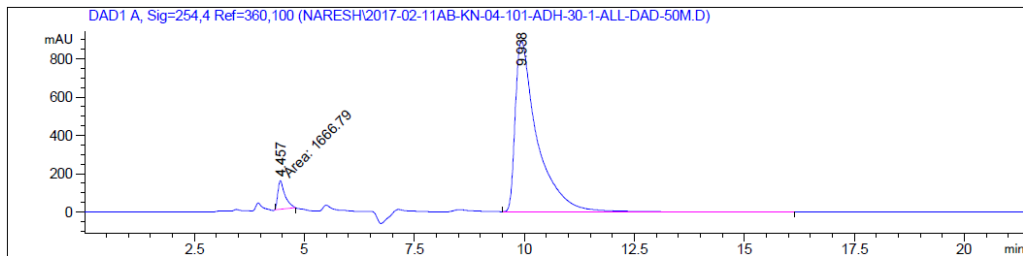
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.455	VB	0.1999	3.09807e4	2153.02539	49.9962
2	9.987	BBA	0.5004	3.09853e4	885.13470	50.0038

Totals : 6.19660e4 3038.16010

## HPLC data of (±)-5q

Data File C:\CHEM32\1\DATA\NARESH\2017-02-11AB-KN-04-101-ADH-30-1-ALL-DAD-50M.D

Sample Name: AB-KN-04-101-ADH-30-1-ALL-DAD-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.457	MM	0.1867	1666.78967	148.75523	5.1045
2	9.938	VB	0.4939	3.09868e4	895.06738	94.8955

Totals : 3.26536e4 1043.82262

## HPLC data of (+)-5q

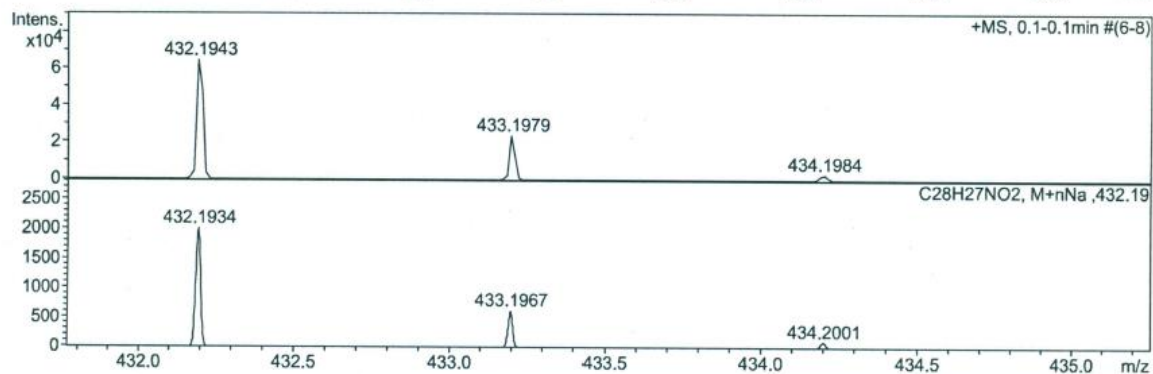
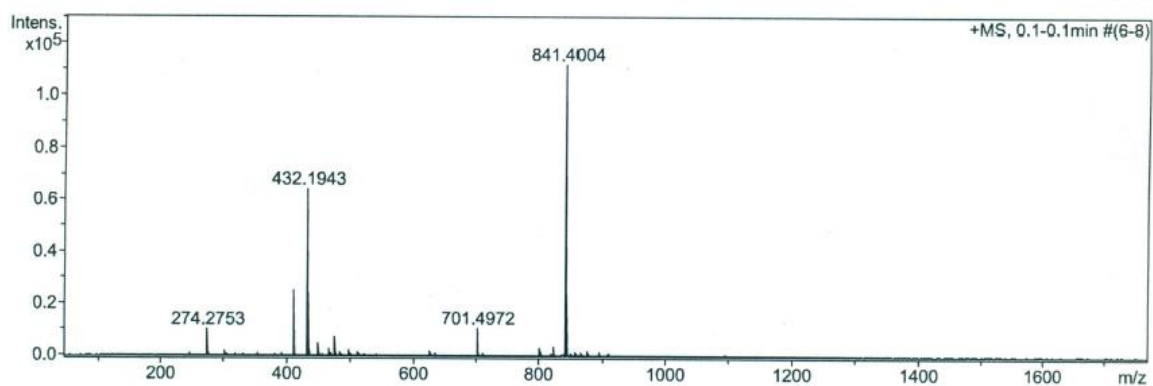
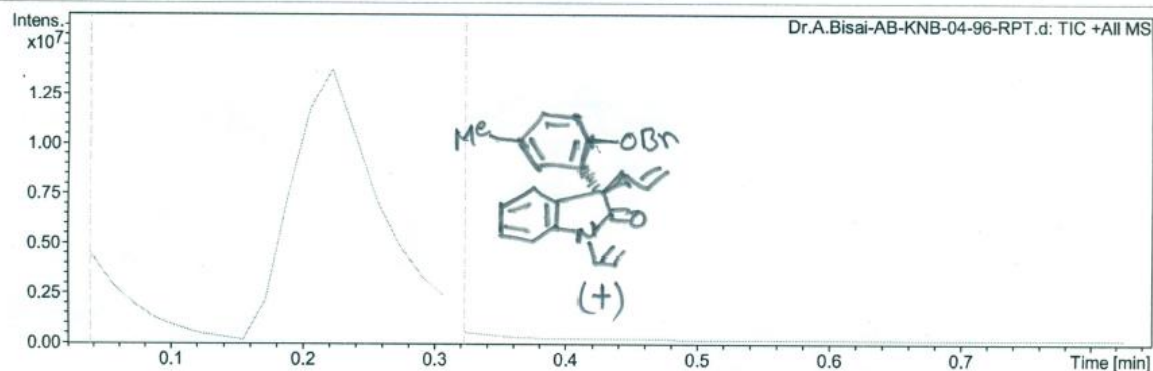


## Display Report

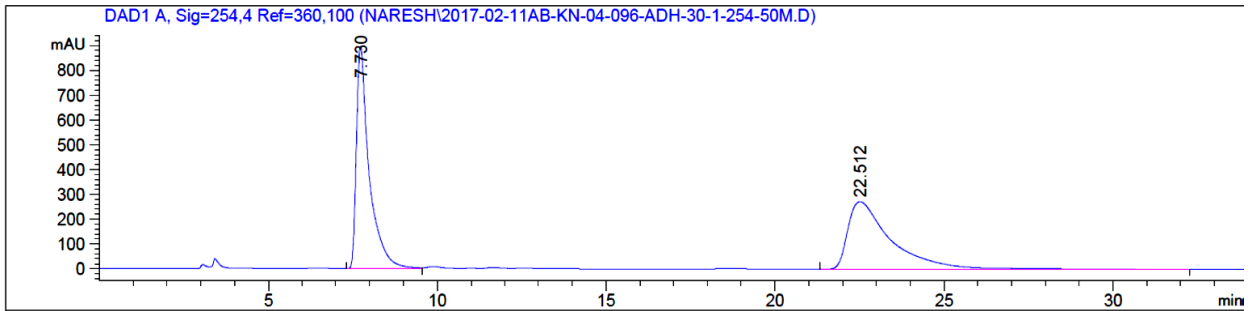
**Analysis Info**  
Analysis Name D:\Data\user data\2017\JUNE 2017\15 june\Dr.A.Bisai-AB-KNB-04-96-RPT.d  
Method Pos\_Mid\_tunemix.m  
Sample Name AB-KNB-04-96-RPT  
Comment  
Acquisition Date 6/15/2017 3:30:51 PM  
Operator RUCHI SHRIVASTAVA  
Instrument micrOTOF-Q II 10330

**Acquisition Parameter**

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **5r**

Data File C:\CHEM32\1\DATA\NARESH\2017-02-11AB-KN-04-096-ADH-30-1-254-50M.D  
 Sample Name: AB-KN-04-096-ADH-30-1-254-50M



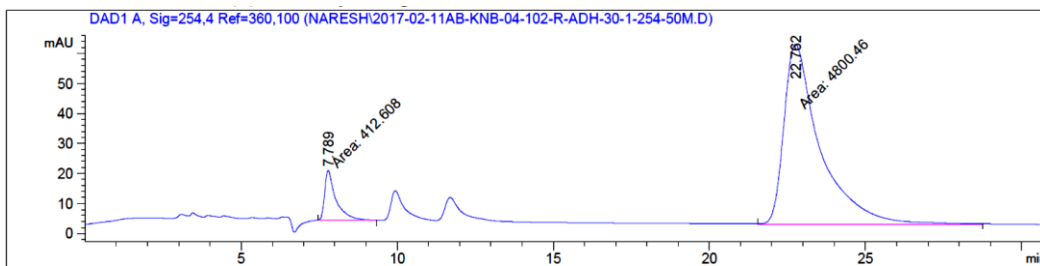
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.730	BV	0.3783	2.37294e4	898.04633	49.5529
2	22.512	BB	1.2775	2.41577e4	272.71533	50.4471

Totals : 4.78871e4 1170.76166

### HPLC data of (±)-5r

Data File C:\CHEM32\1\DATA\NARESH\2017-02-11AB-KNB-04-102-R-ADH-30-1-254-50M.D  
 Sample Name: AB-KNB-04-102-R-ADH-30-1-254-50M

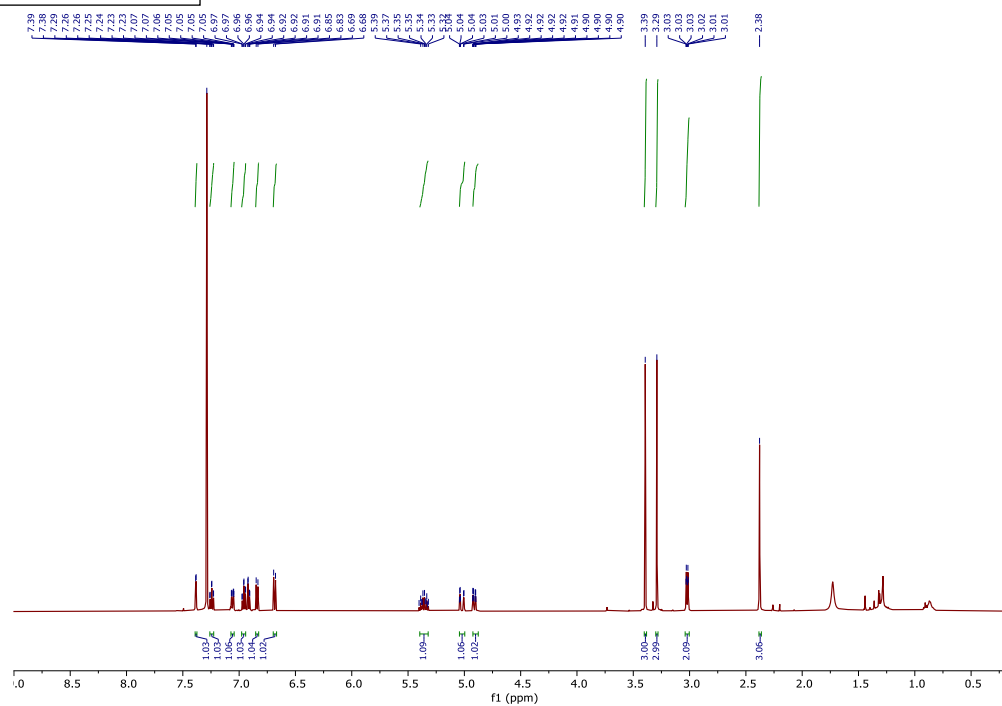
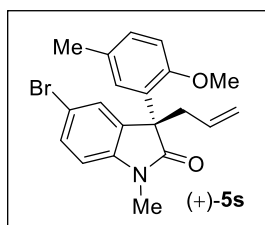


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

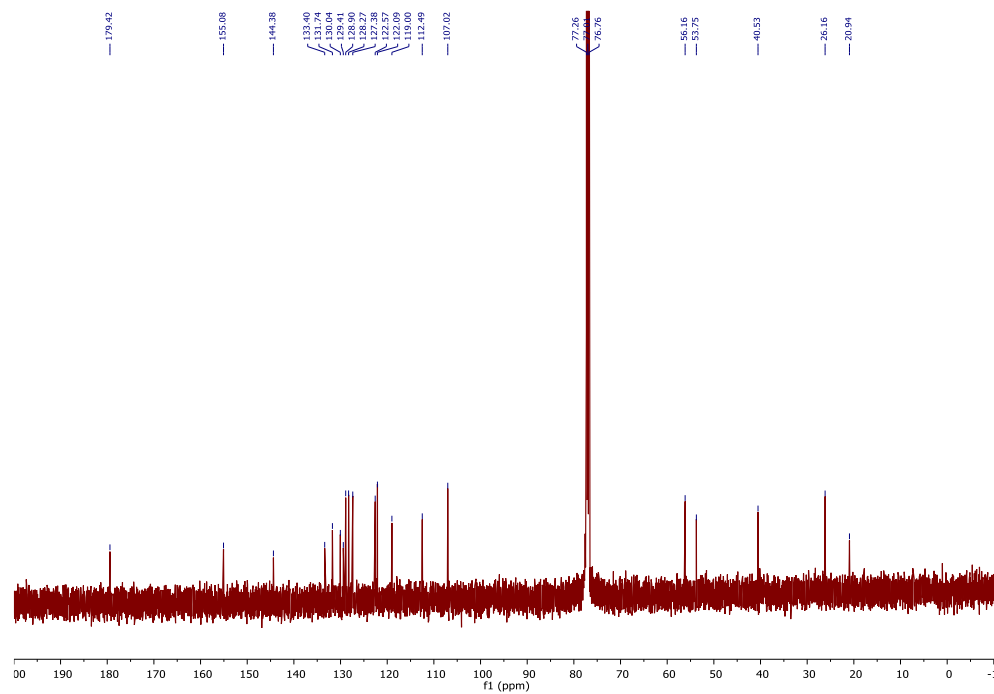
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.789	MM	0.4156	412.60794	16.54796	7.9149
2	22.762	MM	1.3375	4800.45703	59.81768	92.0851

Totals : 5213.06497 76.36564

### HPLC data of (+)-5r



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-5s



$^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (+)-5s

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2019\JUNE\10 june\Dr A Bisai-AM-03-11\_1-A\_4\_01\_6854.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-AM-03-11  
Comment

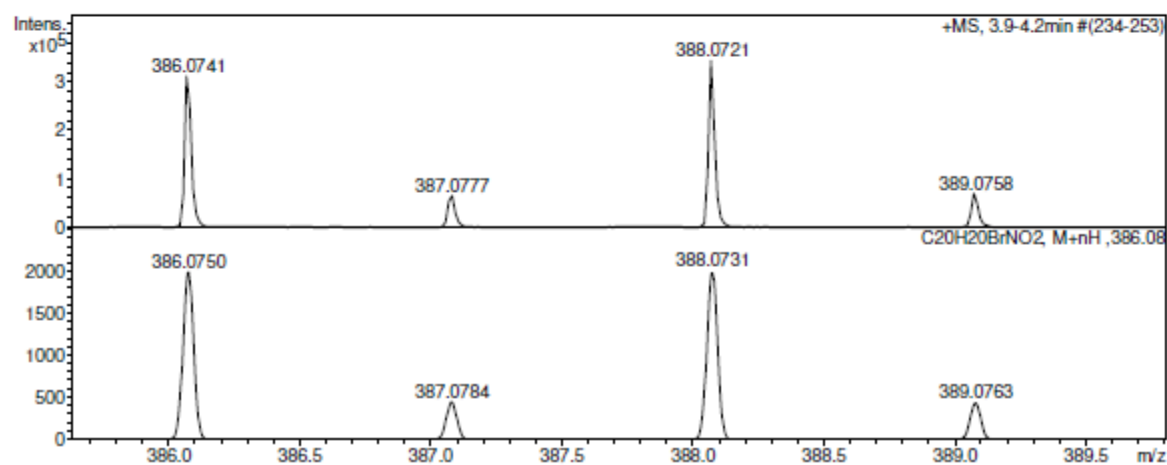
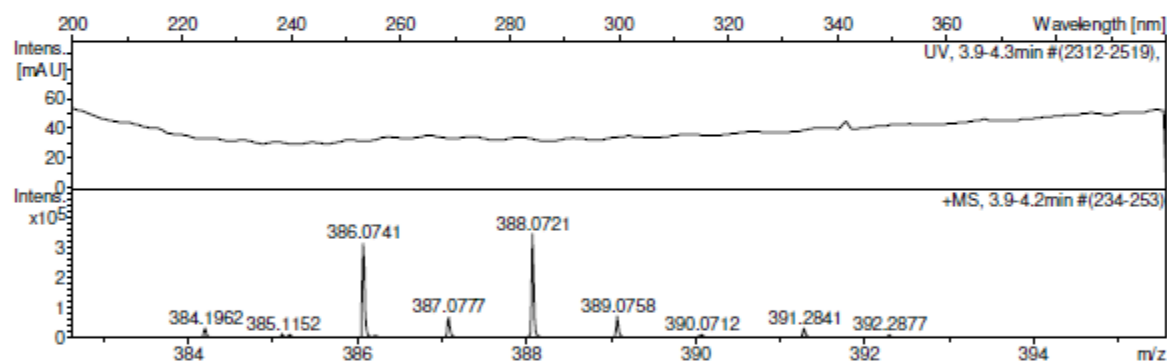
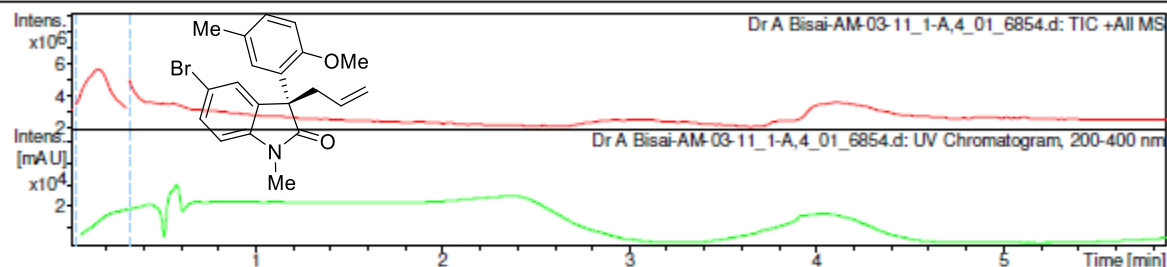
Acquisition Date 6/10/2019 10:33:28 AM

Operator RUCHI

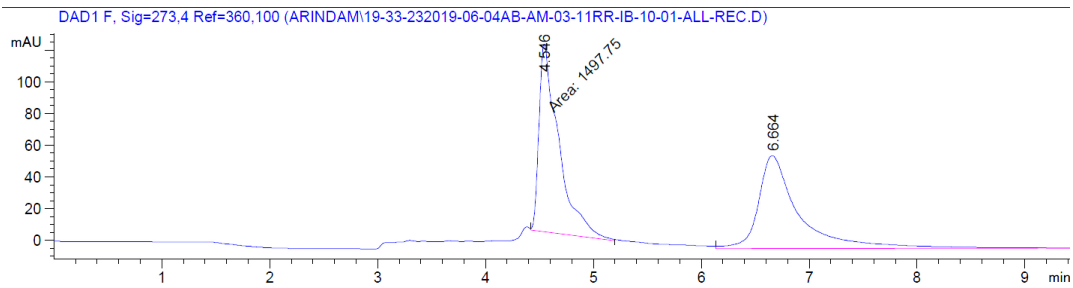
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of 5s

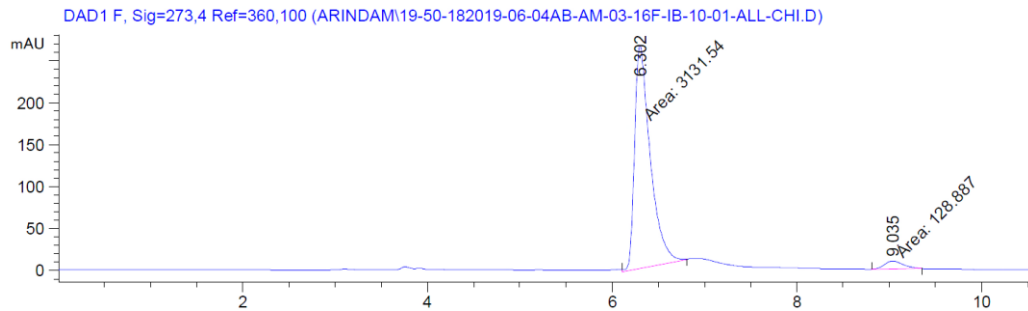


Signal 6: DAD1 F, Sig=273,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.546	MM	0.2113	1497.75293	118.15854	49.2627
2	6.664	VBA	0.3791	1542.58655	58.59653	50.7373

Totals : 3040.33948 176.75507

#### HPLC Data of (±)-5s

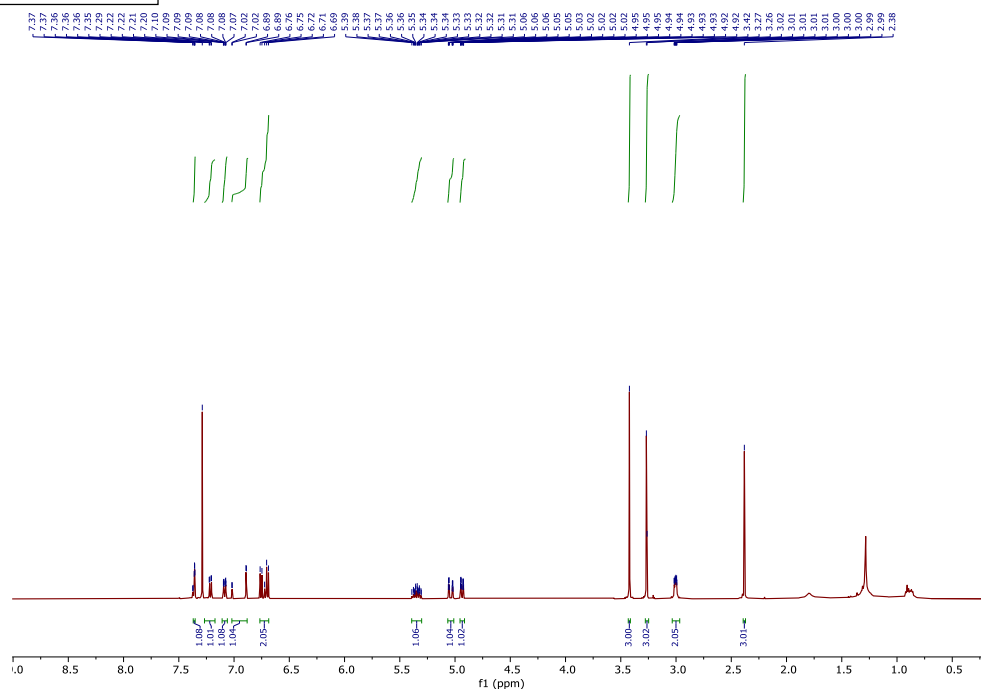
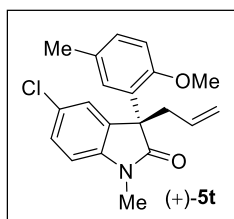


Signal 6: DAD1 F, Sig=273,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.302	MM	0.1945	3131.54224	268.29449	96.0469
2	9.035	MM	0.2282	128.88745	9.41365	3.9531

Totals : 3260.42969 277.70815

#### HPLC data of (+)-5s





## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2019\JUNE\10 june\Dr A Bisai-AM-03-14\_1-B,3\_01\_6864.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-AM-03-14  
Comment

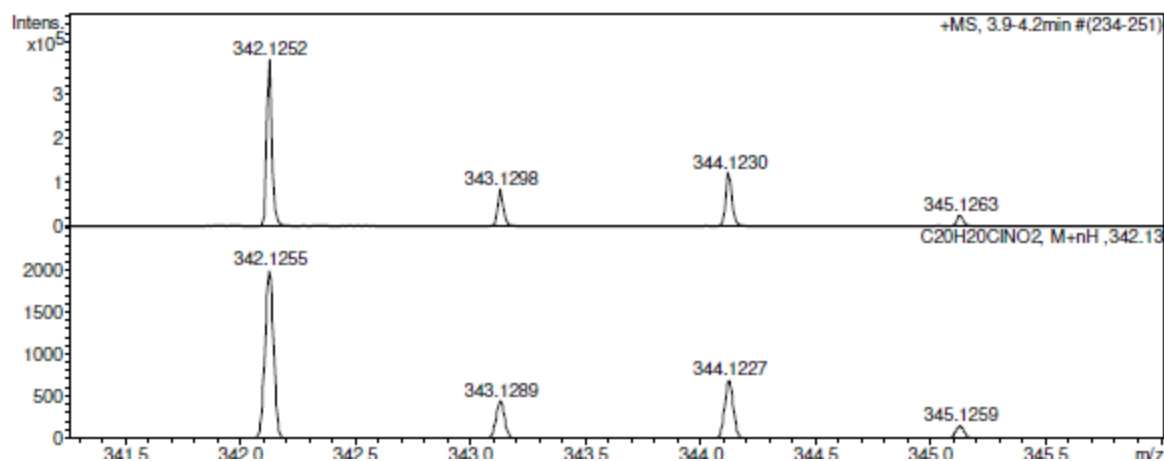
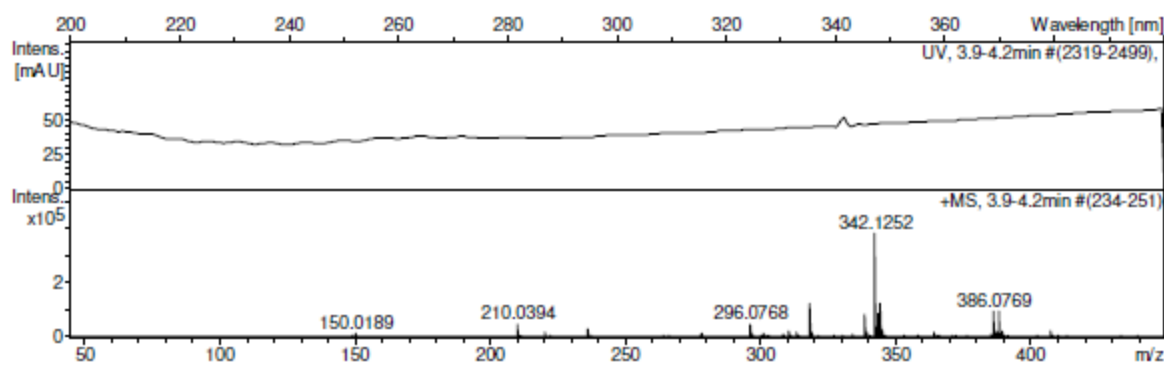
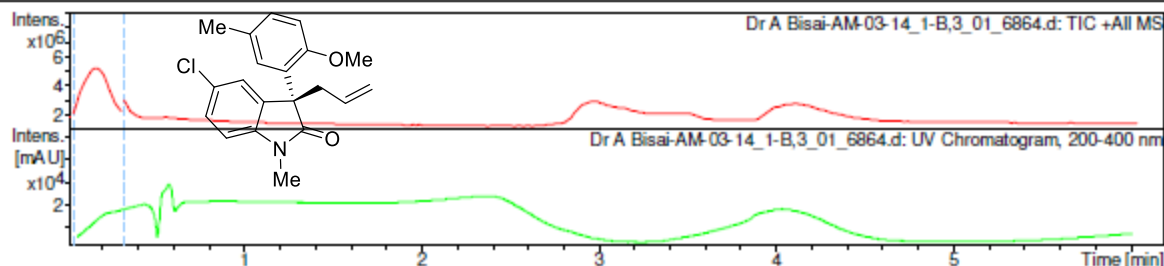
Acquisition Date 6/10/2019 11:45:28 AM

Operator RUCHI

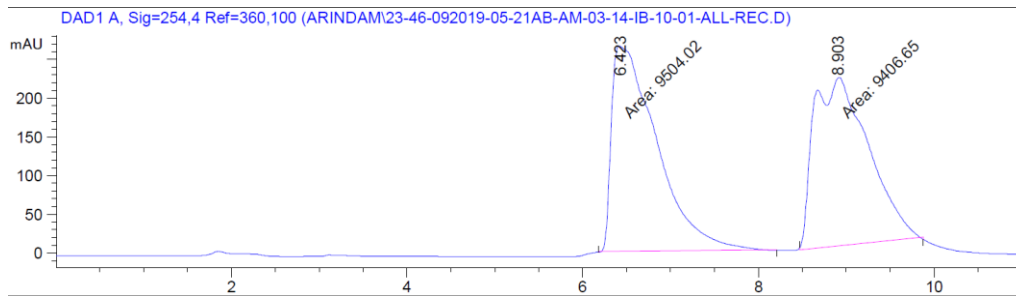
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of 5t

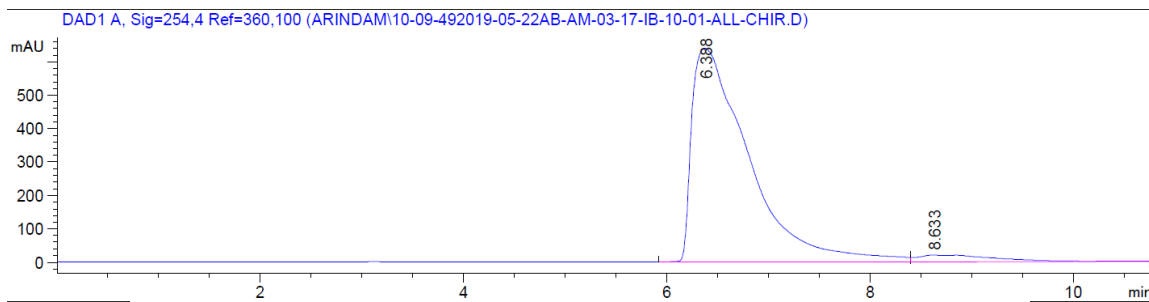


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.423	MM	0.6010	9504.02148	263.56543	50.2575
2	8.903	MM	0.7244	9406.64648	216.43089	49.7425

Totals : 1.89107e4 479.99632

#### HPLC Data of (±)-5t



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.388	BV	0.5343	2.49075e4	639.62952	95.8482
2	8.633	VBA	0.6649	1078.90356	20.40334	4.1518

Totals : 2.59864e4 660.03285

#### HPLC data of (+)-5t



## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2019\JUNE\10 june\Dr A Bisai-AM-03-18\_1-A,6\_01\_6857.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-AM-03-18  
Comment

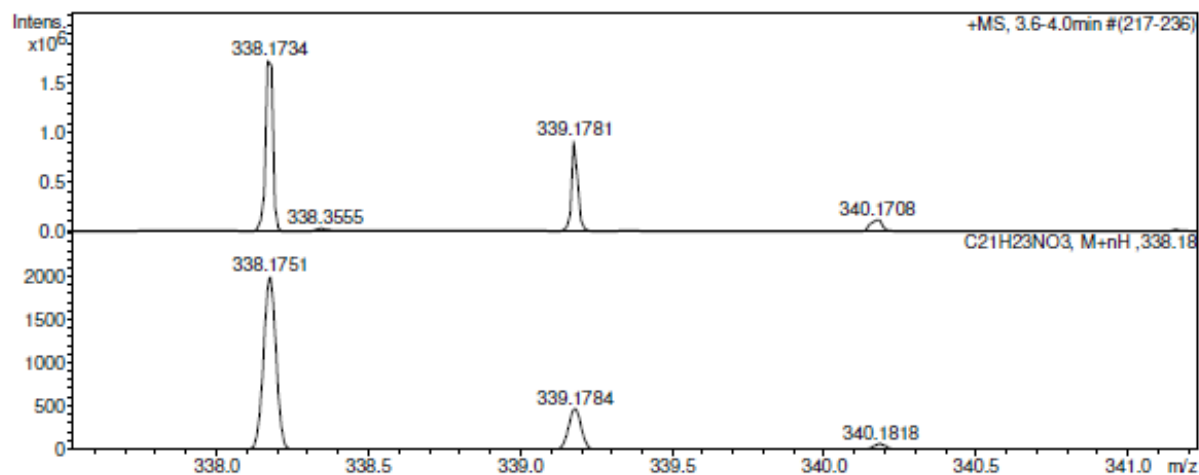
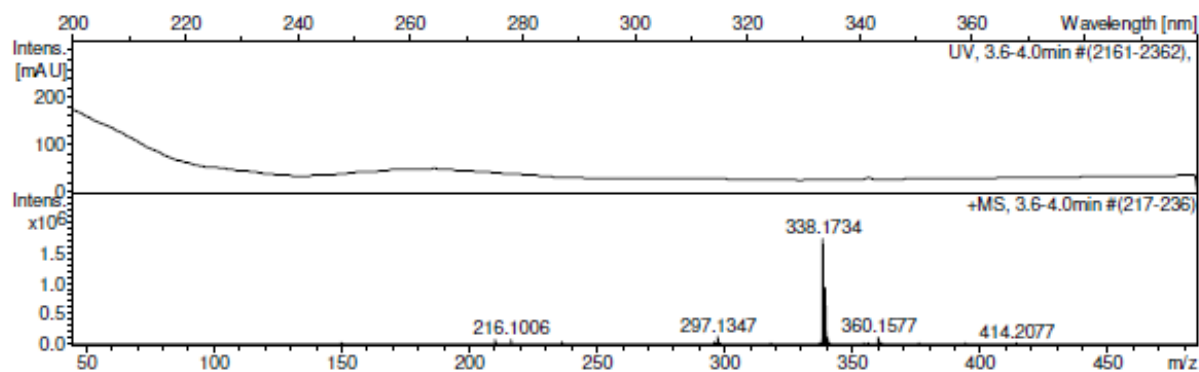
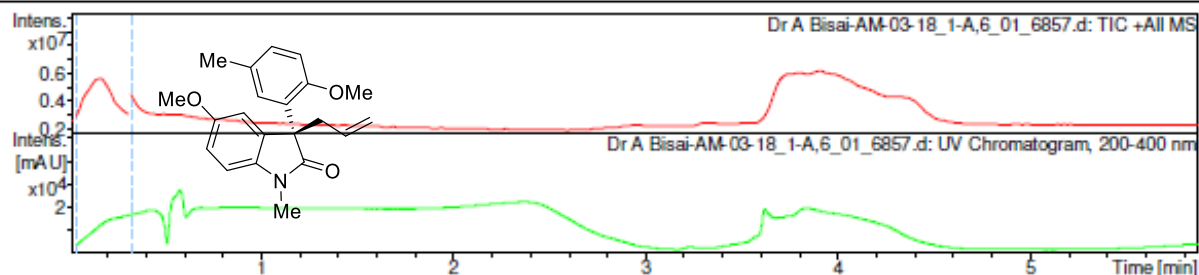
Acquisition Date 6/10/2019 10:55:03 AM

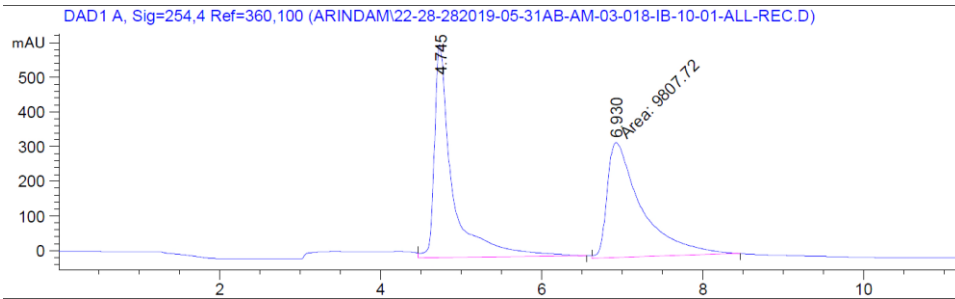
Operator RUCHI

Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned Mass data of compound **5u**

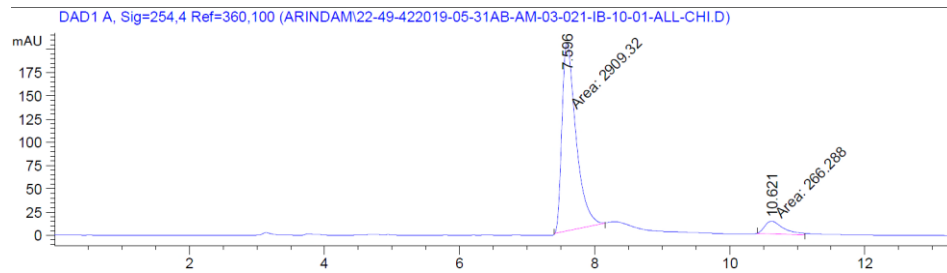


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.745	VB	0.2331	9571.66699	603.41498	49.3910
2	6.930	MM	0.4931	9807.71582	331.48758	50.6090

Totals : 1.93794e4 934.90256

#### HPLC data of (±)-5u

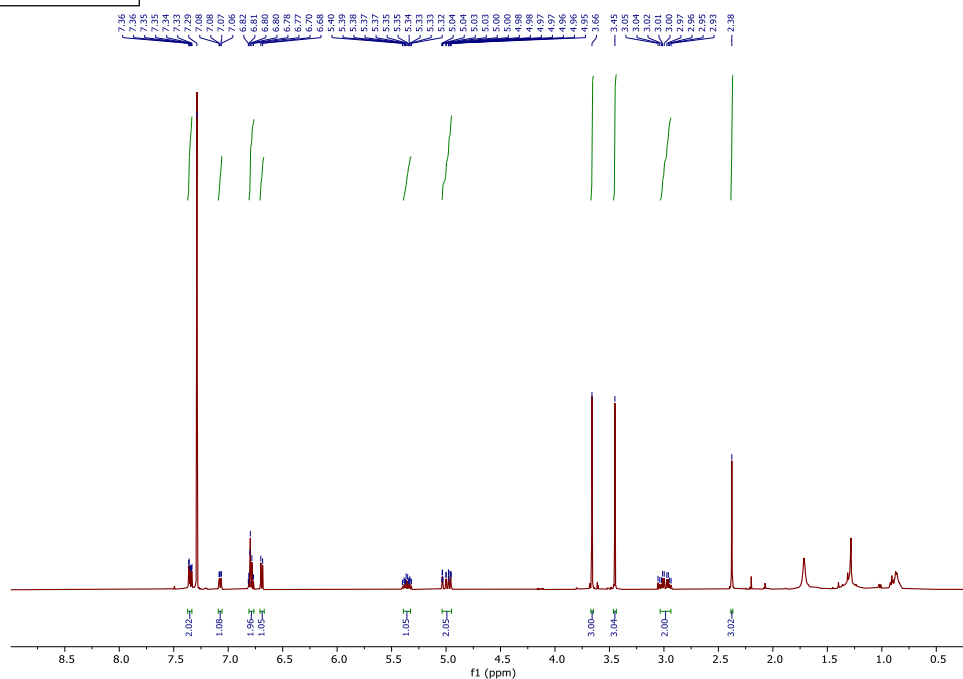
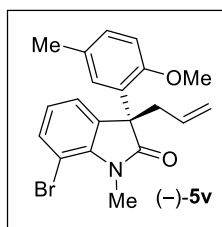


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

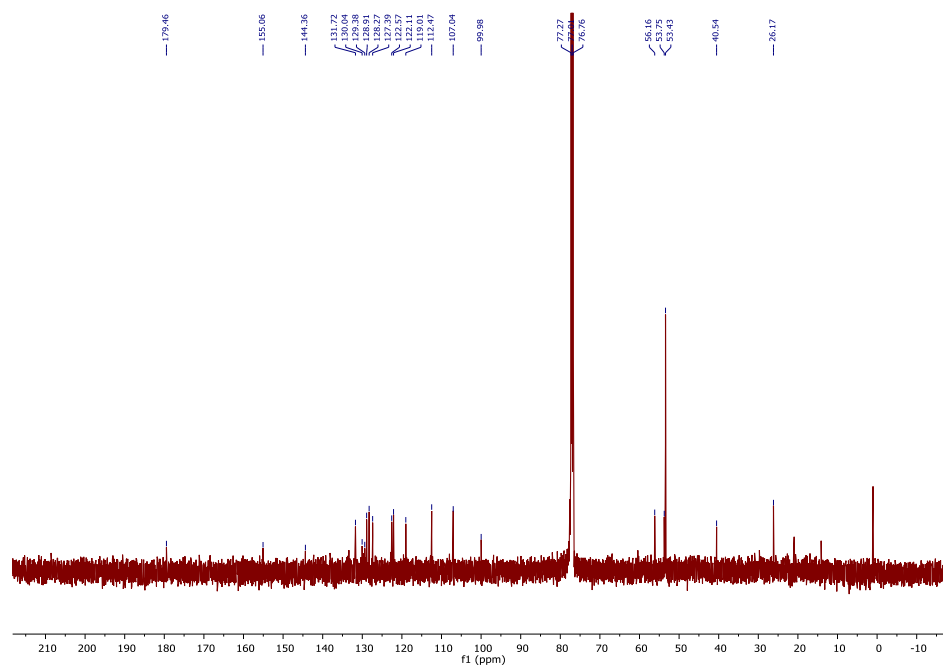
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.596	MM	0.2412	2909.32031	201.05243	91.6146
2	10.621	MM	0.3228	266.28760	13.74798	8.3854

Totals : 3175.60791 214.80041

#### HPLC Data of (+)-5u



$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (-)-5v



$^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ ) of compound (-)-5v

## Display Report

## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2019\JUNE\10 june\Dr A Bisai-AM-03-25\_1-B,1\_01\_6861.d  
 Method hrlcms-20 sept.m  
 Sample Name Dr A Bisai-AM-03-25  
 Comment

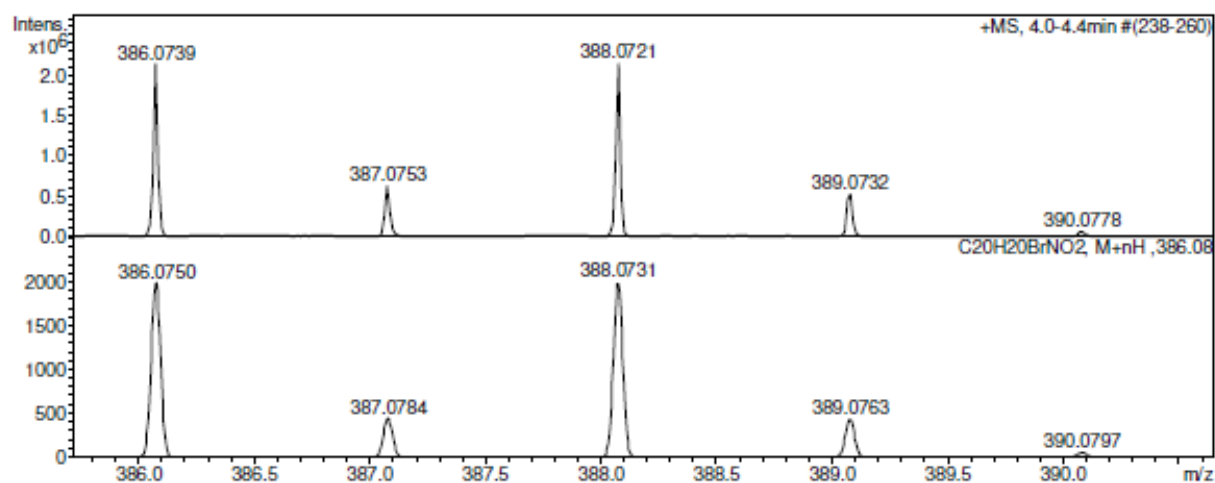
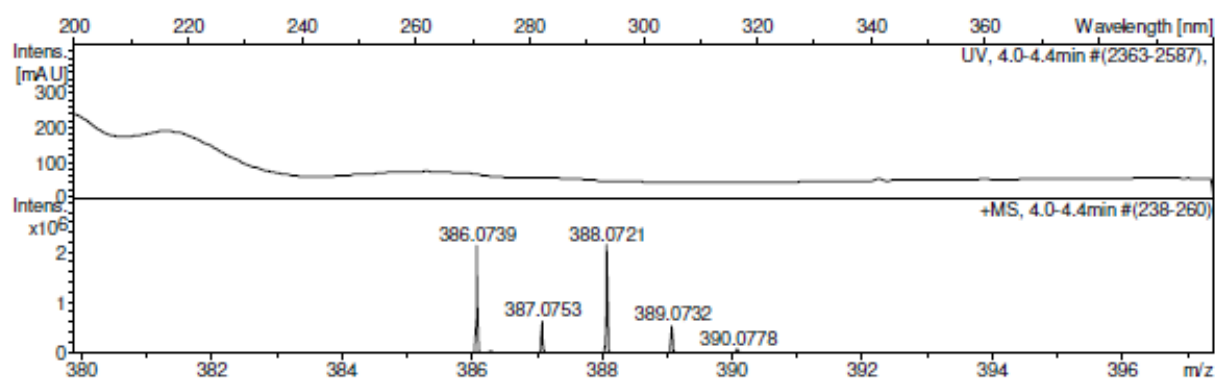
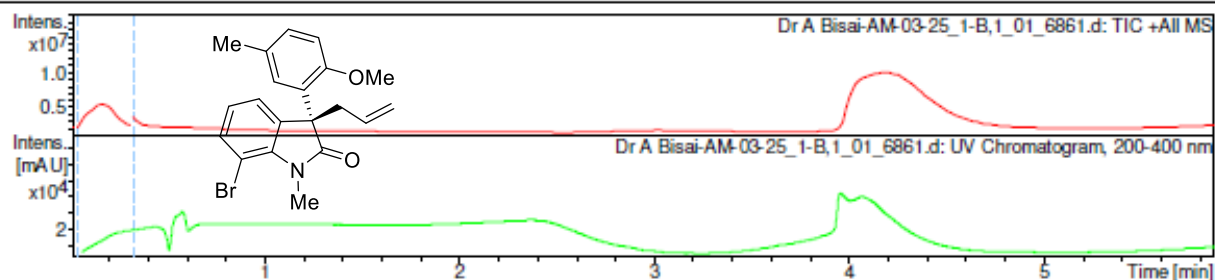
Acquisition Date 6/10/2019 11:23:53 AM

Operator RUCHI

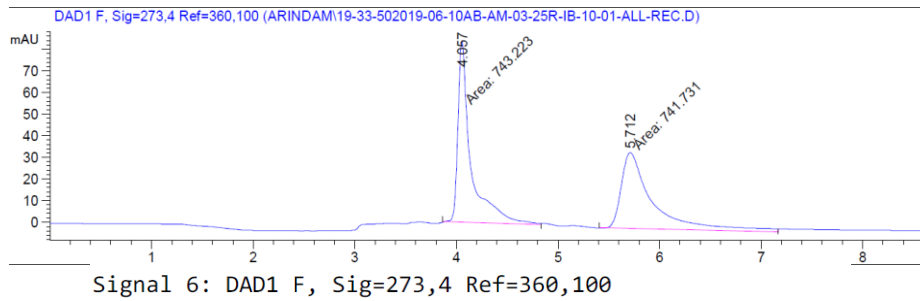
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



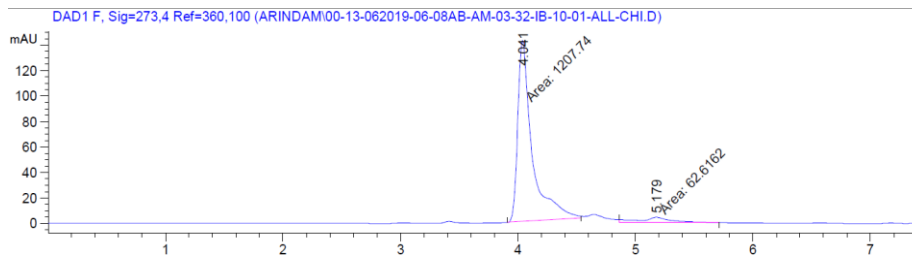
Scanned Mass data of compound 5v



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.057	MM	0.1471	743.22339	84.22432	50.0503
2	5.712	MM	0.3525	741.73071	35.06516	49.9497

Totals : 1484.95410 119.28948

### HPLC Data of (±)-5v



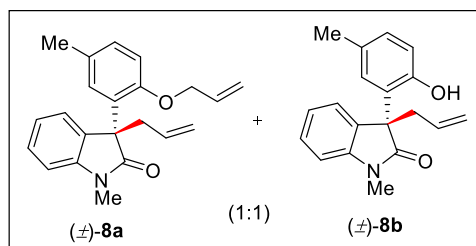
Signal 6: DAD1 F, Sig=273,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.041	MM	0.1409	1207.73901	142.87244	95.0710
2	5.179	MM	0.2744	62.61616	3.80319	4.9290

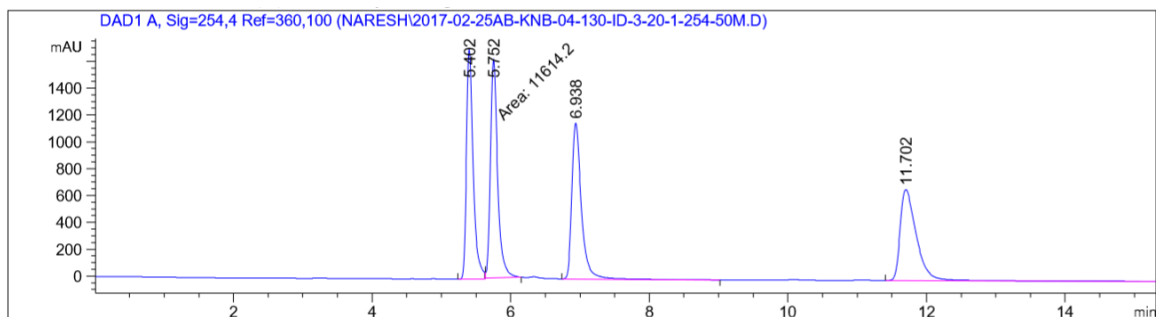
Totals : 1270.35518 146.67562

### HPLC data of (-)-5v





Data File C:\CHEM32\1\DATA\NARESH\2017-02-25AB-KNB-04-130-ID-3-20-1-254-50M.D  
 Sample Name: AB-KNB-04-130-ID-3-20-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

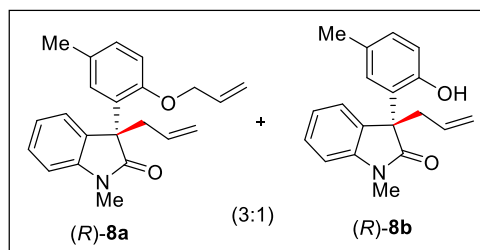
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.402	BV	0.1023	1.14922e4	1712.02576	25.3740
2	5.752	MM	0.1190	1.16142e4	1626.31165	25.6435
3	6.938	BB	0.1435	1.10967e4	1161.01721	24.5010
4	11.702	BBA	0.2466	1.10879e4	678.86127	24.4815

Totals : 4.52911e4 5178.21588

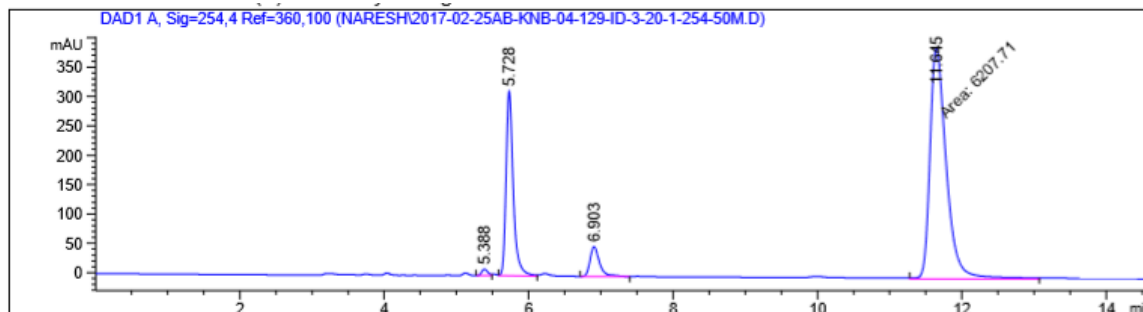
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 \*\*\* End of Report \*\*\*

HPLC data of (+)-**8a** and (+)-**8b**

(dr = 1:1)



Data File C:\CHEM32\1\DATA\NARESH\2017-02-25AB-KNB-04-129-ID-3-20-1-254-50M.D  
 Sample Name: AB-KNB-04-129-ID-3-20-1-254-50M



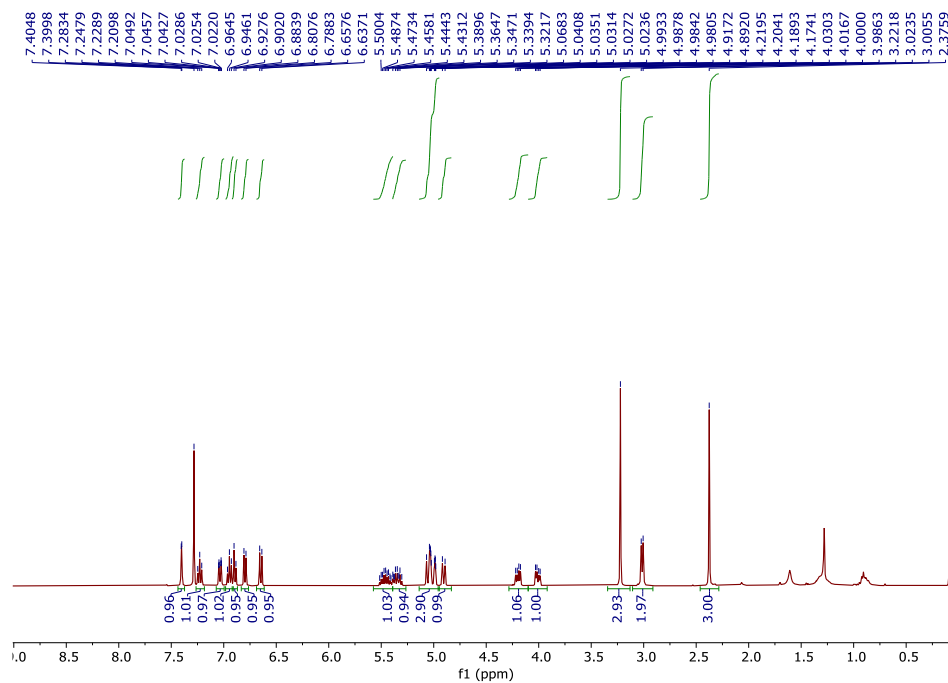
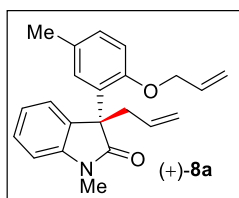
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.388	BV	0.0923	68.13051	11.31443	0.7650
2	5.728	VV	0.1046	2171.73901	314.52011	24.3856
3	6.903	BB	0.1356	458.25934	50.64569	5.1456
4	11.645	NM	0.2630	6207.70898	393.42474	69.7038

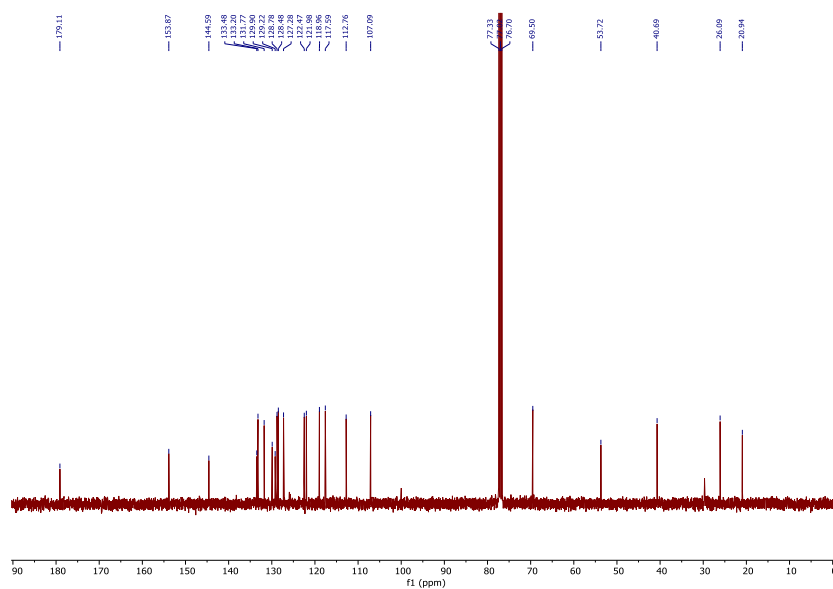
Totals : 8905.83784 769.90497

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

HPLC data of (+)-8a and (+)-8b  
 (dr = 3:1)



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-**8a**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-**8a**

## Display Report

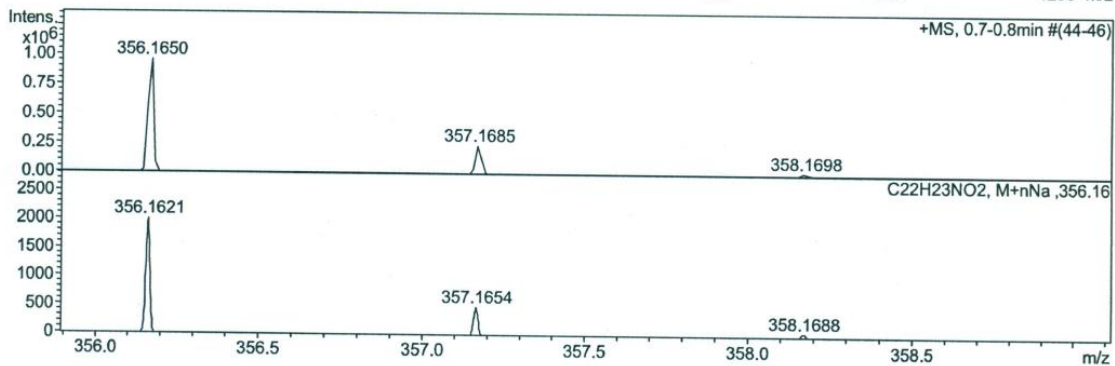
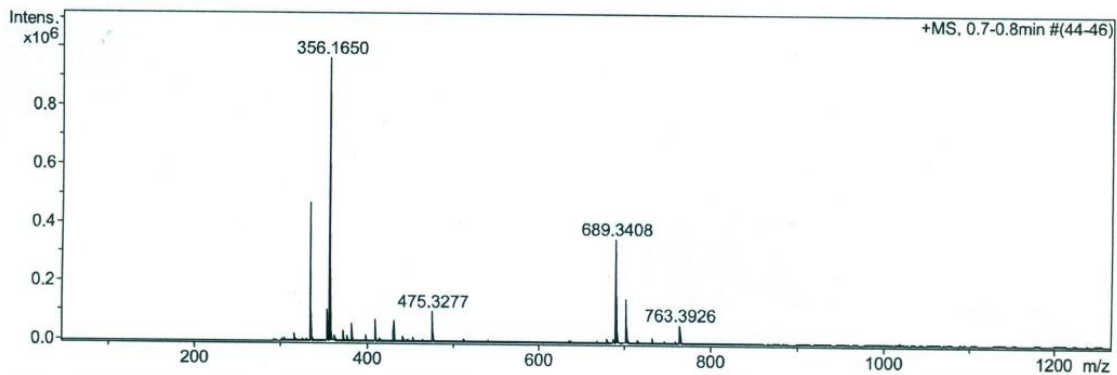
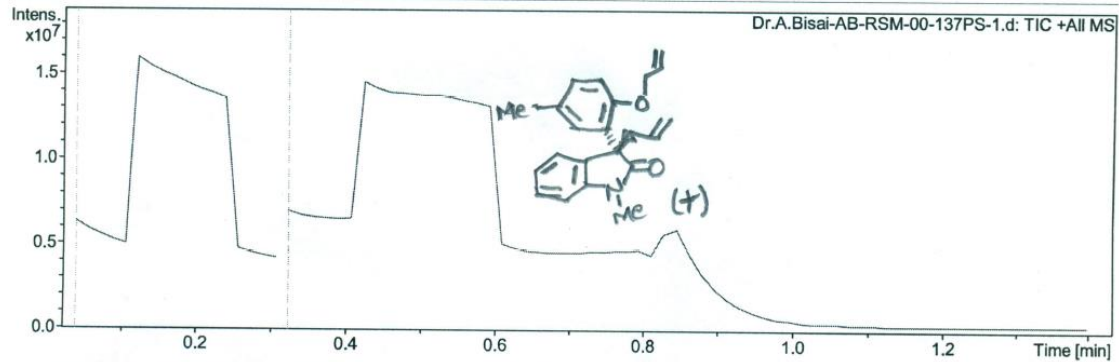
## Analysis Info

Analysis Name D:\Data\user data\2017\JUNE 2017\16 june\Dr.A.Bisai-AB-RSM-00-137PS-1.d  
Method Pos\_Mid\_tunemix.m  
Sample Name AB-RSM-00-137PS-1  
Comment

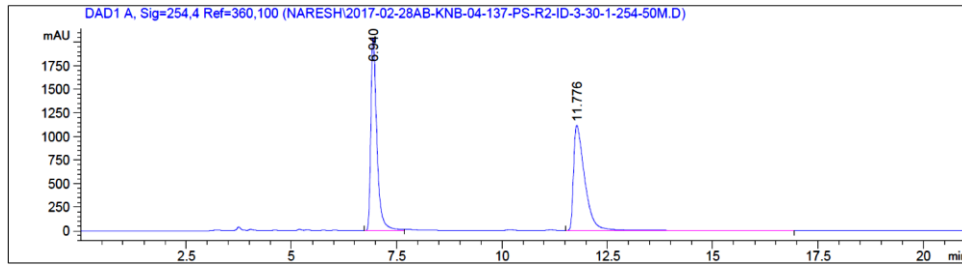
Acquisition Date 6/16/2017 4:17:41 PM  
Operator RUCHI SHRIVASTAVA  
Instrument micrOTOF-Q II 10330

## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of **8a**

Data File C:\CHEM32\1\DATA\NARESH\2017-02-28AB-KNB-04-137-PS-R2-ID-3-30-1-254-50M.D  
 Sample Name: AB-KNB-04-137-PS-R2-ID-3-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

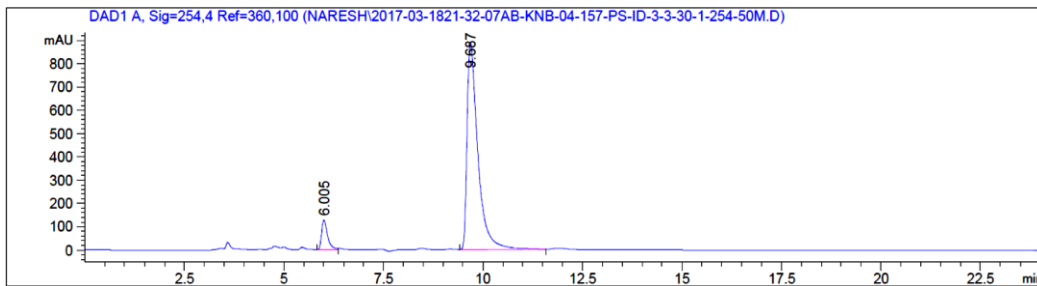
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.940	BV	0.1508	2.04694e4	2044.10193	49.5962
2	11.776	BB	0.2779	2.08027e4	1113.83081	50.4038

Totals : 4.12721e4 3157.93274

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

### HPLC data of (±)-8a

Data File C:\CHEM32\1\DATA\NARESH\2017-03-1821-32-07AB-KNB-04-157-PS-ID-3-3-30-1-254-50M.D  
 Sample Name: AB-KNB-04-157-PS-ID-3-3-30-1-254-50M



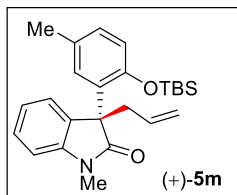
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.005	BV	0.1591	1346.84875	127.64511	7.4610
2	9.687	VB	0.2774	1.67050e4	888.16193	92.5390

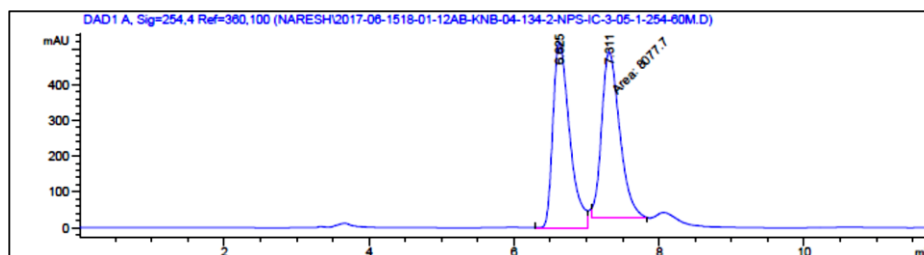
Totals : 1.80518e4 1015.80704

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 \*\*\* End of Report \*\*\*

### HPLC data of (+)-8a



Data File C:\CHEM32\...TA\NARESH\2017-06-1518-01-12AB-KNB-04-134-2-NPS-IC-3-05-1-254-60M.D  
 Sample Name: AB-KNB-04-134-2-NPS-IC-3-05-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

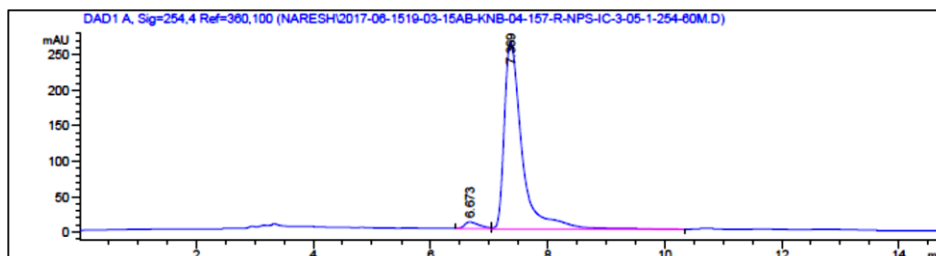
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.625	BV	0.2446	8311.57715	519.60382	50.7135
2	7.311	MM	0.2898	8077.69971	464.60922	49.2865

Totals : 1.63893e4 984.21304

\*\*\* End of Report \*\*\*

### HPLC data of (±)-5m

Data File C:\CHEM32\...TA\NARESH\2017-06-1519-03-15AB-KNB-04-157-R-NPS-IC-3-05-1-254-60M.D  
 Sample Name: AB-KNB-04-157-R-NPS-IC-3-05-1-254-60M



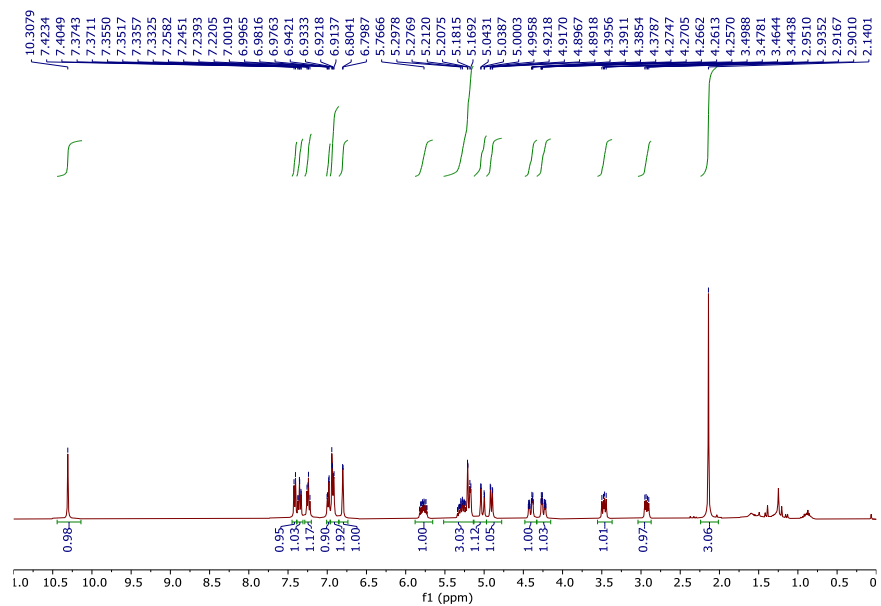
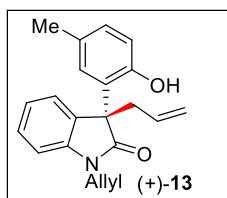
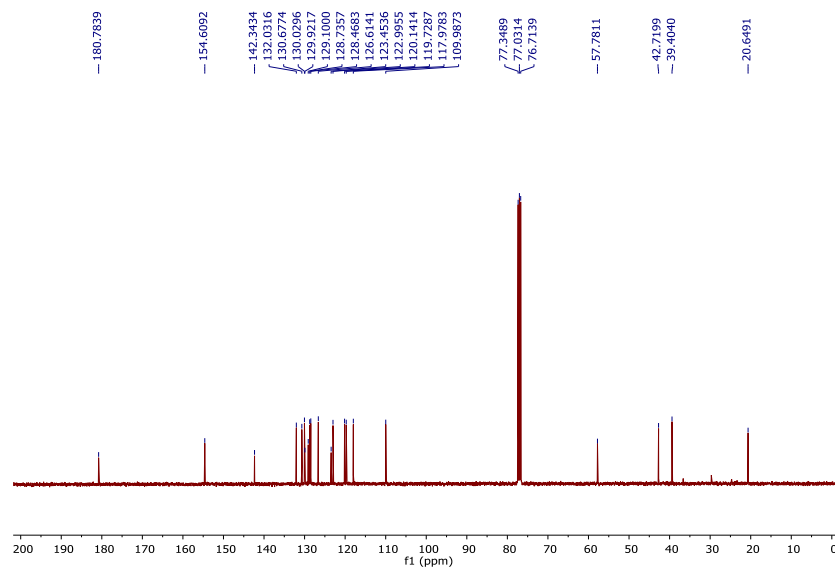
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.673	BV	0.2768	165.21983	9.23370	2.9355
2	7.369	VB	0.3140	5463.05273	263.37537	97.0645

Totals : 5628.27257 272.60907

\*\*\* End of Report \*\*\*

HPLC data of (R)-5m from TBS protection of decarboxylative O-protonation compound.

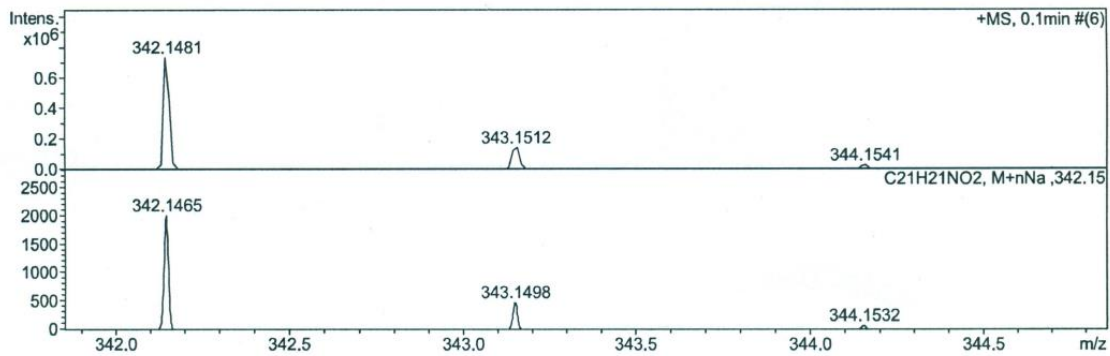
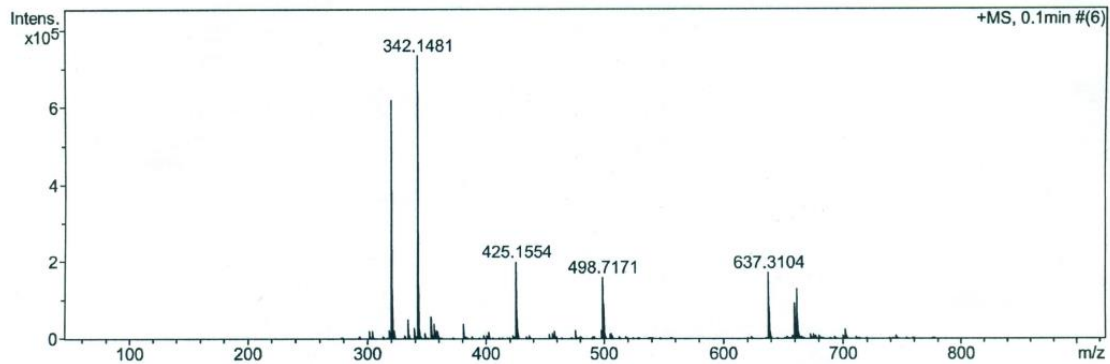
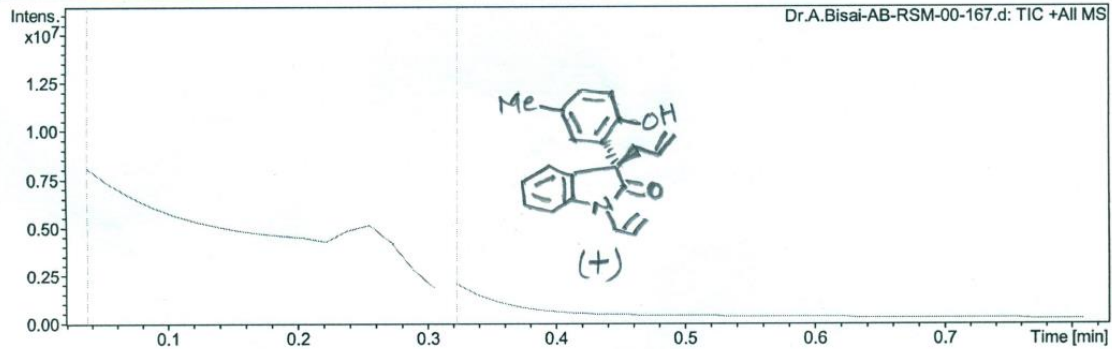
 $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) of compound (+)-13 $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) of compound (+)-13

## Display Report

<b>Analysis Info</b>		Acquisition Date	6/16/2017 4:21:25 PM
Analysis Name	D:\Data\user data\2017\JUNE 2017\16 june\Dr.A.Bisai-AB-RSM-00-167.d	Operator	RUCHI SHRIVASTAVA
Method	Pos_Mid_tunemix.m	Instrument	micrOTOF-Q II 10330
Sample Name	AB-RSM-00-167		
Comment			

**Acquisition Parameter**

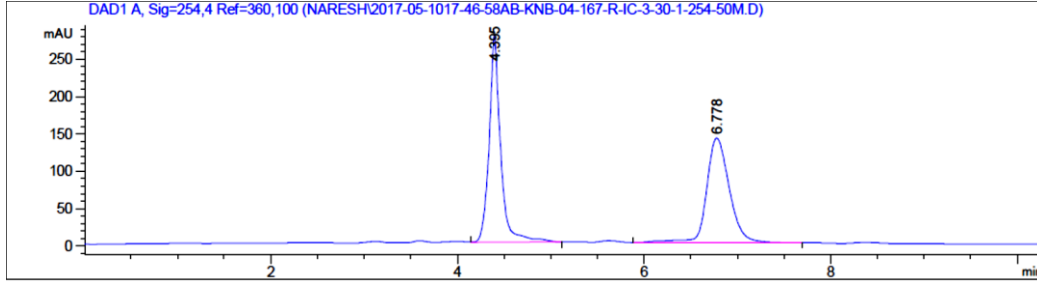
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	450.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (+)-13



Data File C:\CHEM32\1\DATA\NARESH\2017-05-1017-46-58AB-KNB-04-167-R-IC-3-30-1-254-50M.D  
 Sample Name: AB-KNB-04-167-R-IC-3-30-1-254-50M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

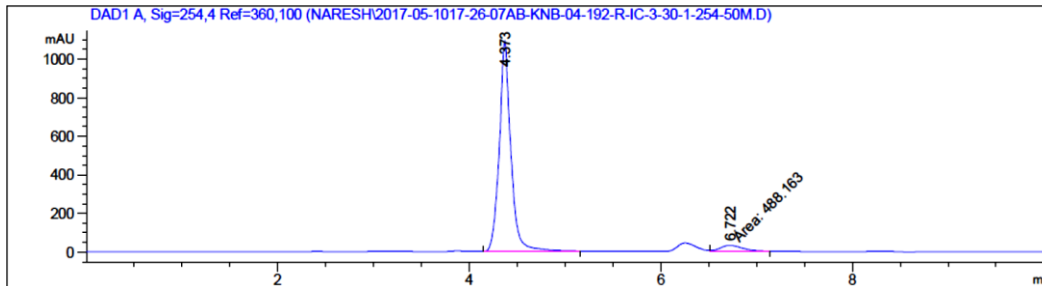
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.395	VB	0.1185	2379.25562	276.26971	50.1021
2	6.778	BB	0.2592	2369.55420	140.17566	49.8979

Totals : 4748.80981 416.44537

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 \*\*\* End of Report \*\*\*

### HPLC data of (±)-13

Data File C:\CHEM32\1\DATA\NARESH\2017-05-1017-26-07AB-KNB-04-192-R-IC-3-30-1-254-50M.D  
 Sample Name: AB-KNB-04-192-R-IC-3-30-1-254-50M



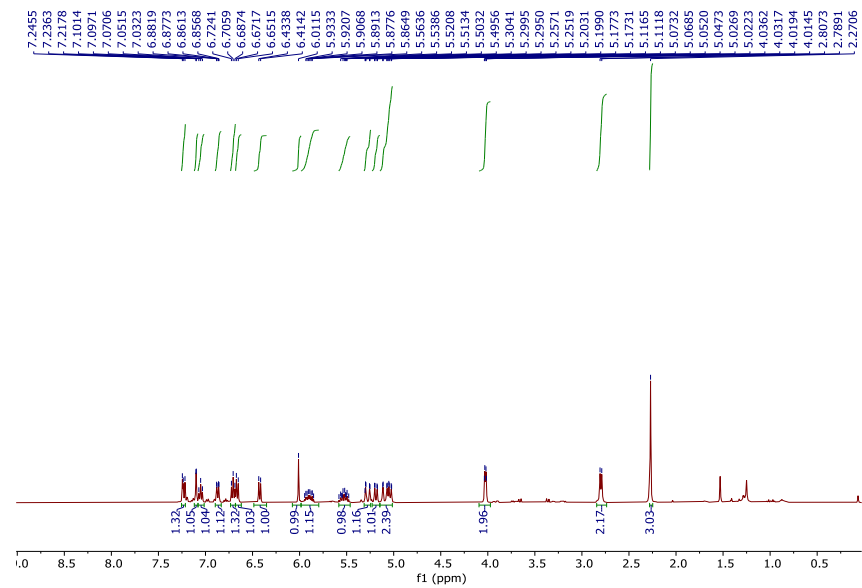
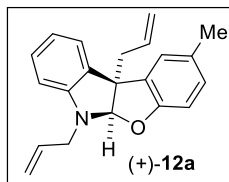
Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.373	BB	0.1158	9121.92578	1087.77991	94.9203
2	6.722	MM	0.2652	488.16257	30.67769	5.0797

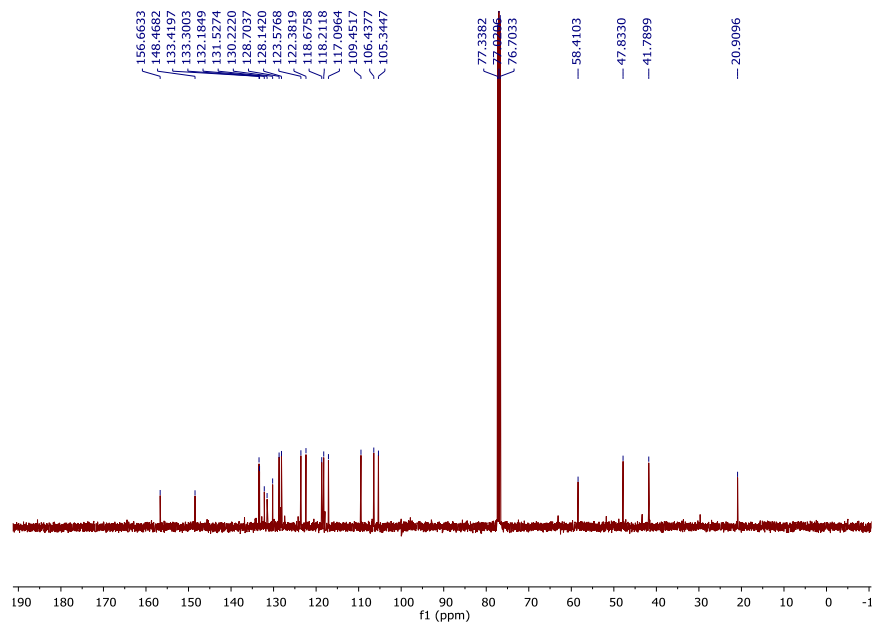
Totals : 9610.08835 1118.45760

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 \*\*\* End of Report \*\*\*

### HPLC data of (+)-13



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) of compound (+)-12a



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) of compound (+)-12a

## Display Report

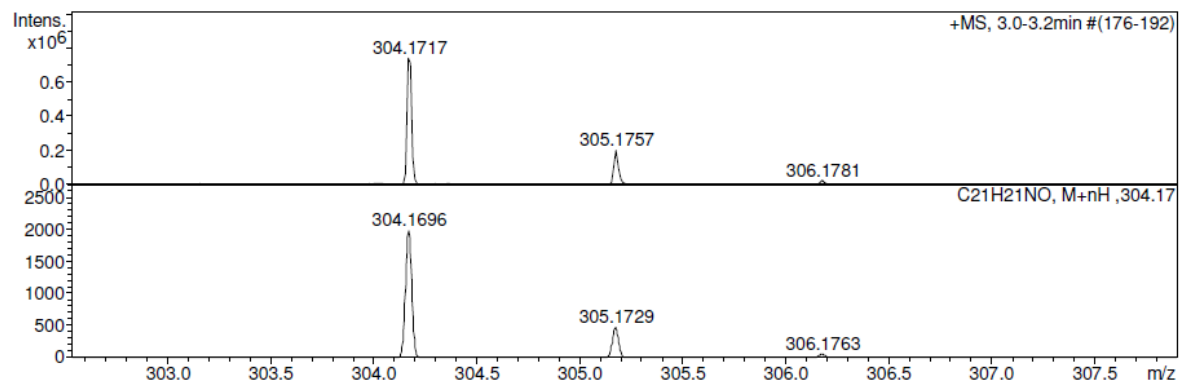
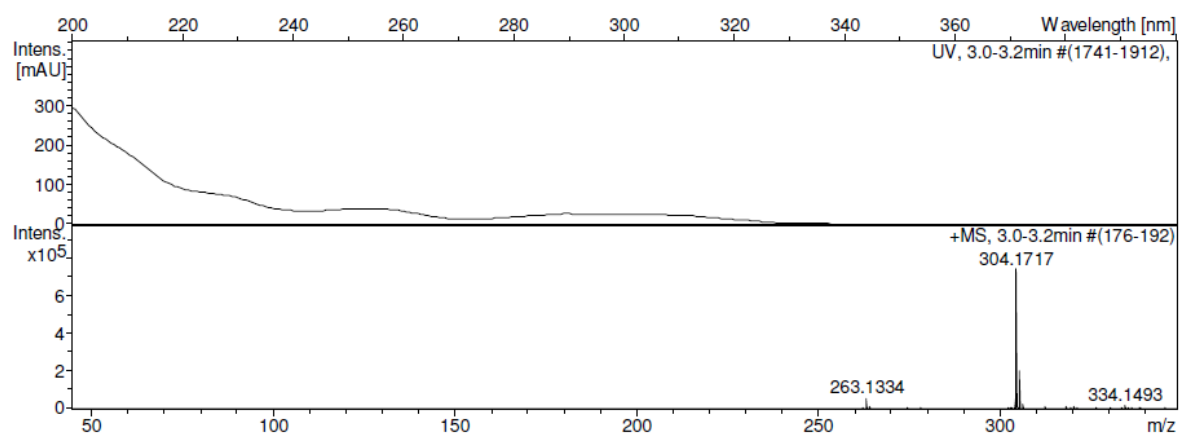
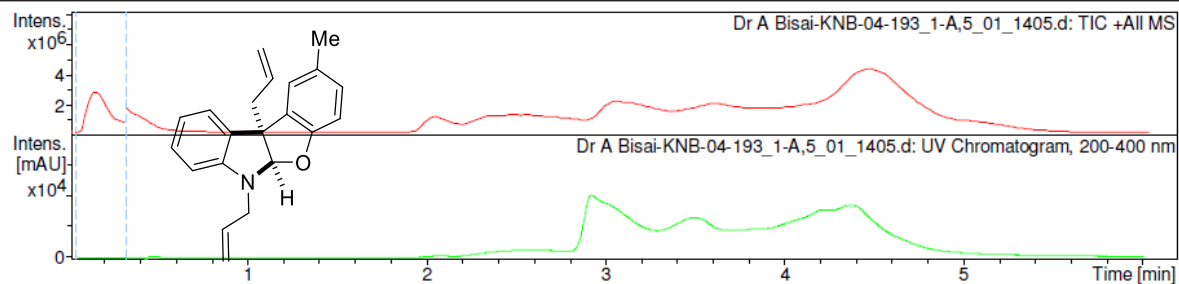
## Analysis Info

Analysis Name D:\Data\NEW USER DATA 2017\2018\27 mar\Dr A Bisai-KNB-04-193\_1-A,5\_01\_1405.d  
Method hrlcms-20 sept.m  
Sample Name Dr A Bisai-KNB-04-193  
Comment

Acquisition Date 3/27/2018 10:41:25 AM  
Operator RUCHI  
Instrument micrOTOF-Q II 10330

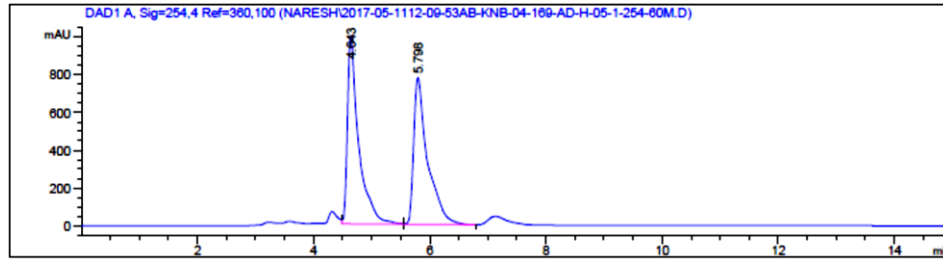
## Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (+)-12a

Data File C:\CHEM32\1\DATA\NARESH\2017-05-1112-09-53AB-KNB-04-169-AD-H-05-1-254-60M.D  
 Sample Name: AB-KNB-04-169-AD-H-05-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

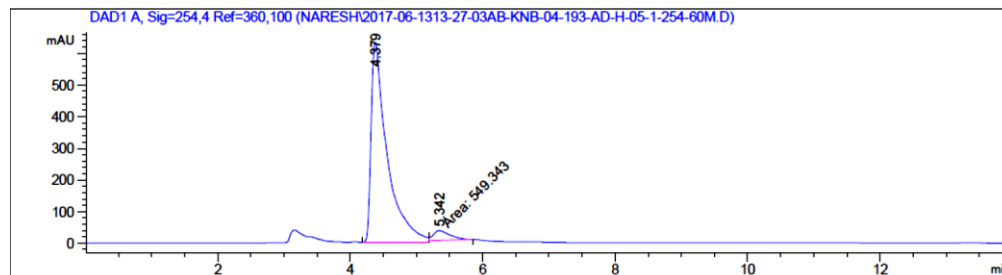
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.643	VV	0.1868	1.31793e4	993.25177	50.7396
2	5.798	VB	0.2321	1.27951e4	777.19336	49.2604

Totals : 2.59744e4 1770.44513

\*\*\* End of Report \*\*\*

### HPLC data of (±)-12a

Data File C:\CHEM32\1\DATA\NARESH\2017-06-1313-27-03AB-KNB-04-193-AD-H-05-1-254-60M.D  
 Sample Name: AB-KNB-04-193-AD-H-05-1-254-60M



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.379	BV	0.2320	1.06171e4	631.81897	95.0804
2	5.342	MM	0.2926	549.34290	31.28816	4.9196

Totals : 1.11664e4 663.10713

\*\*\* End of Report \*\*\*

### HPLC data of (+)-12a