

## Supporting Information

### **Metal triflate-Catalyzed Cyclization of Arylvinylcarbinols: Formal Synthesis of (±)-Dichroanone and (±)-Taiwaniaquinone H**

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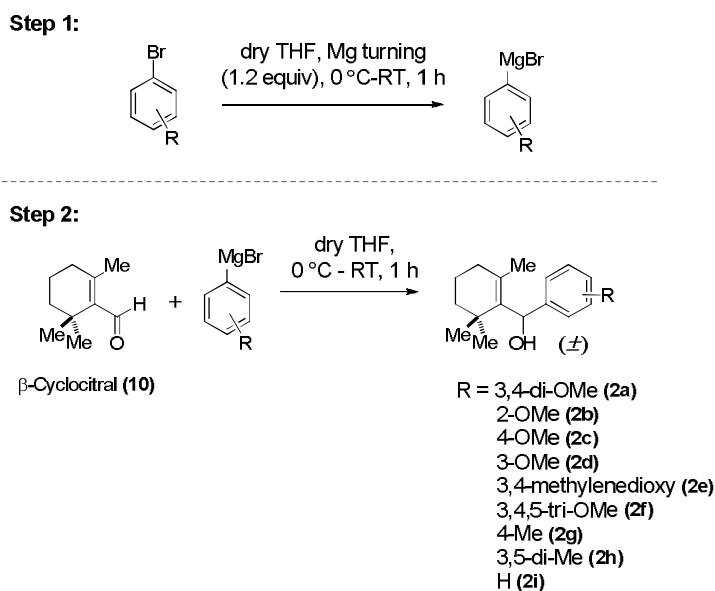
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## Materials and Methods

Unless otherwise stated, reactions were performed in oven-dried glassware fitted with rubber septa under a nitrogen atmosphere and were stirred with Teflon-coated magnetic stirring bars. Liquid reagents and solvents were transferred via syringe using standard Schlenk techniques. Tetrahydrofuran (THF), diethyl ether (Et<sub>2</sub>O) was distilled over sodium/benzophenone ketyl. Dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) and toluene were distilled over calcium hydride. All other solvents such as DMF, Chloroform, Dioxane, DMSO, DCE, Acetonitrile, CCl<sub>4</sub>, Methanol and reagents such as β-Cyclocitral, 2-Isopropyl phenol, *ortho*-Vanillin, Phenyl magnesium bromide, Methyl magnesium bromide, 2-Bromo anisole, 3-Bromo anisole, 4-Bromo anisole, 4-Bromoveratrole, 4-Bromo-1,2-(methylenedioxy)benzene, 4-Bromotoluene, 5-Bromo-1,2,3-trimethoxybenzene, 1-Bromo-3,5-dimethylbenzene, 2-Bromobenzaldehyde, 6-Bromoveratraldehyde, Piperonal, Bromine, Ceric ammonium nitrate, Dimethyl sulphate, Potassium carbonate, PCC, Ammonium tribromide, *n*-Butyllithium, NaSEt, different types of Metal triflate etc. were used as received, unless otherwise noted.

Thin layer chromatography was performed using Merck Silicagel 60 F-254 precoated plates (0.25 mm) and visualized by UV irradiation, anisaldehyde, yellow dip stain and other stains. Silicagel from Merck (particle size 230-400 mesh) was used for flash chromatography. Melting points were recorded on a digital melting point apparatus from Jyoti Scientific (AN ISO 9001:2000) and are uncorrected. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Bruker 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 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 hydrogens). 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) from PerkinElmer spectrometer and are reported in frequency of absorption (cm<sup>-1</sup>). Only selected IR absorbencies are reported. High resolution mass spectra were obtained from the Central Instrumentation Facility (CIF) at the Indian Institute of Science Education and Research (IISER) Bhopal.

**Scheme 1:** General procedure for synthesis of arylvinylcarbinols (**2a-i**) of  $\beta$ -cyclocitral.



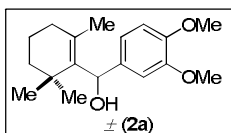
**Step 1:**

A flame-dried round-bottom flask was charged with Mg-turning (1.44 equiv.) & a pinch of  $I_2$  under nitrogen atmosphere in dry THF (3 mL for per mmol) and cooled to  $0^\circ\text{C}$  on an ice-bath. Then substituted bromobenzene (1.2 equiv.) was added dropwise (red to colourless to brown colour) over a period of 15 minutes at  $0^\circ\text{C}$ . The reaction mixture was stirred at room temperature until the Mg turning was almost consumed. The freshly prepared Grignard was directly used for next step.

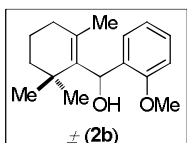
**Step 2:**

A flame-dried round-bottom flask was charged with  $\beta$ -cyclocitral [1.0 equiv. (generally in 500 mg scale)] under nitrogen atmosphere in dry THF (5 mL for per mmol) and cooled to  $0^\circ\text{C}$  on an ice-bath. The Grignard solution (1.2 equiv. as prepared earlier) was added drop wise to the reaction mixture by a syringe and allowed to warm to room temperature. The stirring was continued till TLC showed complete consumption of starting materials. The reaction mixture was quenched by saturated  $\text{NH}_4\text{Cl}$  solution and then diluted with 20 mL of EtOAc. The whole reaction mixture was taken in a separatory funnel and extracted with 20 mL of water. The organic filtrate was separated and the aqueous part was again washed with 10 mL EtOAc, the combined organic filtrate dried over anhydrous  $\text{Na}_2\text{SO}_4$

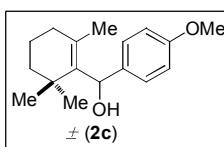
and concentrated in a rotary evaporator under vacuum. The crude products were purified by flash chromatography (10:1 hexanes/EtOAc) to afford **2a-i**.



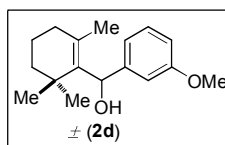
(±)-(3,4-dimethoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol **±(2a)**: 59% yield as colorless solid,  $R_f = 0.5$  (20% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.07 (d,  $J = 1.4$  Hz, 1H), 6.90 (dq,  $J = 1.08, 8.36$  Hz, 1H), 6.81 (d,  $J = 8.32$  Hz, 1H), 5.39 (s, 1H), 3.89 (s, 3H), 3.88 (s, 3H), 2.00 (t,  $J = 6.12$  Hz, 2H), 1.89 (s, 1H), 1.69-1.63 (m, 2H), 1.56-1.53 (m, 2H), 1.46 (s, 3H), 1.20 (s, 3H), 1.05 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 148.7, 147.4, 140.4, 137.3, 133.8, 118.1, 110.7, 109.7, 70.6, 55.9, 55.8, 39.7, 34.8, 33.7, 28.6, 21.6, 21.6, 19.3; **IR** (film)  $\nu_{\text{max}}$  3448, 2917, 2834, 2361, 1598, 1530, 1513, 1463, 1410, 1382, 1360, 1253, 1156, 1137, 1030, 940, 895, 858, 805, 763, 639, 581  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  313.1774  $[(M + \text{Na})]^+$ ; calculated for  $[\text{C}_{18}\text{H}_{26}\text{O}_3 + \text{Na}]^+$ : 313.1774; **MP** 53–55 °C.



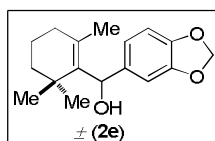
(±)-(2-methoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol **±(2b)**: 86% yield as colorless solid,  $R_f = 0.5$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.25 (t,  $J = 9.88$  Hz, 2H), 6.92 (t,  $J = 8.72$  Hz, 2H), 5.67 (s, 1H), 3.95 (s, 3H), 2.13 (brs, 2H), 1.70 (brs, 3H), 1.54 (q,  $J = 10.8$  Hz, 2H), 1.23 (s, 3H), 0.83 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 157.8, 135.4, 134.6, 130.8, 129.0, 128.5, 120.3, 110.3, 68.4, 55.3, 39.7, 34.7, 33.7, 28.6, 27.9, 22.7, 19.4; **IR** (film)  $\nu_{\text{max}}$  3565, 2928, 1600, 1586, 1486, 1463, 1403, 1279, 1231, 1184, 1120, 1100, 1029, 975, 940, 863, 754, 735, 623, 574  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  283.1652  $[(M + \text{Na})]^+$ ; calculated for  $[\text{C}_{17}\text{H}_{24}\text{O}_2 + \text{Na}]^+$ : 283.1669; **MP** 69–71 °C.



(±)-(4-methoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2c): 82% yield as colorless gel,  $R_f = 0.5$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.35 (dd,  $J = 6.76, 0.88$  Hz, 2H), 6.88 (dd,  $J = 8.84, 2.96$  Hz, 2H), 5.41 (s, 1H), 3.82 (s, 3H), 2.01 (t,  $J = 6.0$  Hz, 2H), 1.95 (s, 1H), 1.70-1.64 (m, 2H), 1.57-1.53 (m, 2H), 1.45 (s, 3H), 1.21 (s, 3H), 1.05 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 158.1, 140.3, 136.8, 133.6, 127.3, 113.4, 70.5, 55.2, 39.8, 34.8, 33.7, 28.7, 28.6, 21.7, 19.3; **IR** (film)  $\nu_{\text{max}}$  3479, 2928, 1890, 1610, 1583, 1506, 1455, 1362, 1246, 1172, 1105, 1038, 974, 968, 857, 821, 761, 734, 691, 589  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  283.1699  $[(M + \text{Na})^+]$ ; calculated for  $[\text{C}_{17}\text{H}_{24}\text{O}_2 + \text{Na}]^+$ : 283.1669.

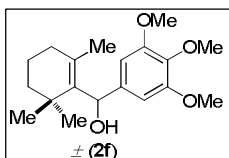


(±)-(3-methoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2d): 97% yield as colorless gel,  $R_f = 0.45$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.21 (t,  $J = 7.92$  Hz, 1H), 7.00 (s, 1H), 6.97 (d,  $J = 7.76$  Hz, 1H), 6.74 (dd,  $J = 8.12, 2.44$  Hz, 1H), 3.79 (s, 3H), 2.15 (s, 3H), 1.96 (t,  $J = 6$  Hz, 2H), 1.81 (d,  $J = 4.88$  Hz, 1H), 1.65-1.59 (m, 2H), 1.59 (brs, 1H), 1.52 (q,  $J = 5.28$  Hz, 2H), 1.38 (s, 3H), 1.17 (s, 3H), 1.06 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 159.5, 146.8, 140.5, 134.0, 129.0, 118.4, 111.8, 111.3, 70.6, 55.2, 39.7, 34.9, 33.6, 30.9, 28.9, 28.6, 21.6, 19.3; **IR** (film)  $\nu_{\text{max}}$  3391, 2930, 2840, 2360, 1920, 1600, 1494, 1286, 1198, 1149, 1078, 1041, 996, 944, 923, 838, 766, 784, 686, 579  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  283.1683  $[(M + \text{Na})^+]$ ; calculated for  $[\text{C}_{17}\text{H}_{24}\text{O}_2 + \text{Na}]^+$ : 283.1669.

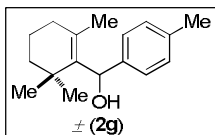


(±)-Benzo[*d*][1,3]dioxol-5-yl(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2e): 99% yield as colorless gel,  $R_f = 0.45$  (20% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.95 (s, 1H), 6.91 (dt,  $J = 8.08, 1.16$  Hz, 1H), 6.78 (d,  $J = 8.08$  Hz, 1H), 5.96 (s, 2H), 5.33 (d,  $J = 3.96$  Hz, 1H), 2.01 (t,  $J = 6.4$  Hz, 2H), 1.83 (d,  $J = 4.8$  Hz, 1H), 1.69-1.63 (m, 2H), 1.54 (q,  $J = 4.36$  Hz, 2H), 1.46 (s, 3H), 1.19 (s, 3H), 1.06 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 147.5, 145.9, 140.4, 138.9, 133.9, 119.1, 107.8, 107.0, 100.8, 70.6, 39.7, 34.8, 33.7, 28.8, 28.6, 21.6, 19.3; **IR** (film)  $\nu_{\text{max}}$  3465, 2927, 2361, 1609, 1487, 1435, 1363, 1237, 1124, 1084,

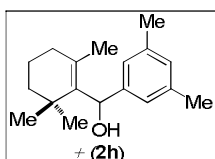
1041, 992, 939, 868, 809, 768, 731, 710, 555  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  297.1456  $[(M + Na)]^+$ ; calculated for  $[C_{17}H_{22}O_3 + Na]^+$ : 297.1461.



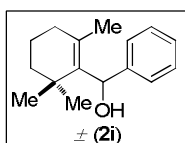
**(±)-(3,4,5-trimethoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2f)**: 90% yield as colorless gel,  $R_f = 0.45$  (35% EtOAc in hexane);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.69 (d,  $J = 0.92$  Hz, 2H), 5.36 (s, 1H), 3.86 (s, 3H), 3.85 (s, 6H), 2.01 (t,  $J = 6.08$  Hz, 2H), 1.85 (brs, 1H), 1.70-1.63 (m, 2H), 1.57-1.54 (m, 2H), 1.46 (s, 3H), 1.20 (s, 3H), 1.09 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 152.9, 140.6, 140.4, 136.4, 134.2, 103.2, 70.8, 60.9, 56.1, 39.8, 34.9, 33.6, 28.8, 28.7, 21.6, 19.3; **IR** (film)  $\nu_{\text{max}}$  3501, 2931, 2361, 1590, 1505, 1455, 1416, 1362, 1325, 1233, 1184, 1127, 1047, 1011, 950, 920, 834, 777, 728  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  343.1794  $[(M + Na)]^+$ ; calculated for  $[C_{19}H_{28}O_4 + Na]^+$ : 343.1880.



**(±)-p-tolyl(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2g)**: 98% yield as colorless gel,  $R_f = 0.6$  (10% EtOAc in hexane);  **$^1\text{H NMR}$**  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.34 (d,  $J = 7.76$  Hz, 2H), 7.16 (d,  $J = 7.96$  Hz, 2H), 5.43 (d,  $J = 4.2$  Hz, 1H), 2.37 (s, 3H), 2.02 (t,  $J = 6.12$  Hz, 2H), 1.91 (d,  $J = 4.88$  Hz, 1H), 1.72-1.64 (m, 2H), 1.57 (q,  $J = 4.72$  Hz, 2H), 1.45 (s, 3H), 1.23 (s, 3H), 1.08 (s, 3H);  **$^{13}\text{C NMR}$**  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 141.8, 140.4, 135.7, 133.7, 128.7, 126.0, 70.7, 39.8, 34.8, 33.7, 28.8, 28.6, 21.7, 21.0, 19.4; **IR** (film)  $\nu_{\text{max}}$  3460, 2923, 2731, 1907, 1799, 1651, 1511, 1455, 1363, 1247, 1171, 1109, 1038, 1009, 974, 898, 857, 808, 757, 728, 691, 578  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  267.1757  $[(M + Na)]^+$ ; calculated for  $[C_{17}H_{24}O + Na]^+$ : 268.1719.

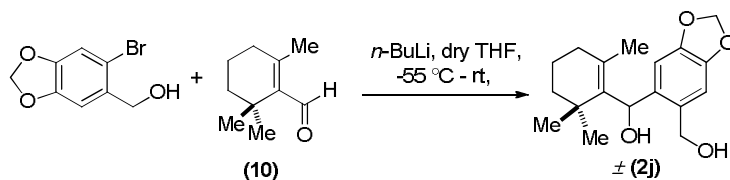


(±)-(3,5-dimethylphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2h): 97% yield as colorless solid,  $R_f = 0.7$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.05 (d,  $J = 0.52$  Hz, 2H), 6.88 (d,  $J = 0.64$  Hz, 1H), 5.38 (s, 1H), 2.34 (s, 6H), 2.04-2.01 (m, 2H), 1.83 (s, 1H), 1.71-1.65 (m, 2H), 1.59-1.55 (m, 2H), 1.44 (s, 3H), 1.21 (s, 3H), 1.09 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 144.7, 140.4, 137.4, 133.7, 127.8, 123.7, 70.8, 39.7, 34.8, 33.6, 28.8, 28.7, 21.7, 21.5, 19.4; **IR** (film)  $\nu_{\text{max}}$  3461, 2927, 2874, 2360, 1602, 1456, 1363, 1238, 1149, 1038, 976, 850, 735  $\text{cm}^{-1}$ ; **LRMS** (ESI)  $m/z$  281.1859  $[(\text{M} + \text{Na})]^+$ ; calculated for  $[\text{C}_{18}\text{H}_{26}\text{O} + \text{Na}]^+$ : 281.1876; **MP** 78–80 °C.



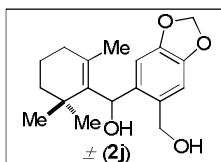
(±)-phenyl(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2i): 99% yield as colorless gel,  $R_f = 0.5$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.47-7.44 (m, 2H), 7.34 (t,  $J = 1.8$  Hz, 2H), 7.23 (td,  $J = 7.6, 0.84$  Hz, 1H), 5.45 (d,  $J = 3.44$  Hz, 1H), 2.01 (t,  $J = 6.04$  Hz, 2H), 1.87 (d,  $J = 4.52$  Hz, 1H), 1.71-1.64 (m, 2H), 1.59-1.55 (m, 2H), 1.40 (s, 3H), 1.22 (s, 3H), 1.10 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 144.9, 140.5, 133.9, 128.0, 126.2, 125.9, 70.7, 39.7, 34.9, 33.7, 28.9, 28.6, 21.6, 19.4; **IR** (film)  $\nu_{\text{max}}$  3460, 3060, 3028, 2928, 2866, 2360, 1651, 1601, 1493, 1448, 1363, 1248, 1171, 1116, 1032, 1009, 919, 895, 845, 781, 753, 704, 624  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  253.1590  $[(\text{M} + \text{H})]^+$ ; calculated for  $[\text{C}_{16}\text{H}_{22} + \text{Na}]^+$ : 253.1563.

**Scheme 2:** General procedure for synthesis of arylvinylcarbinols of  $\beta$ -cyclocitral (2j).



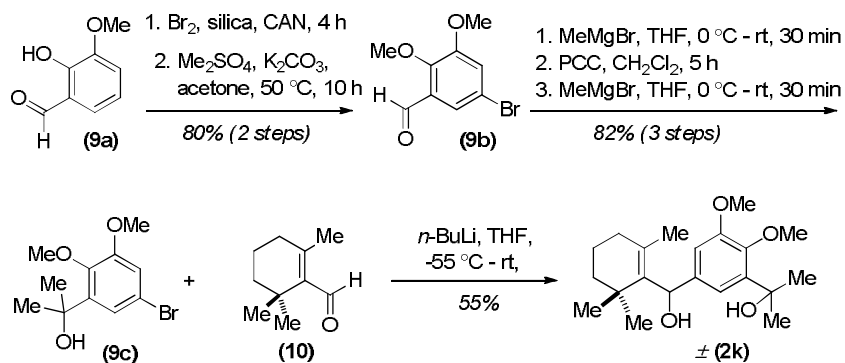
A flame-dried round-bottom flask was charged with 2-bromo substituted benzyl alcohol [1.0 equiv. (generally in 500 mg scale)] under nitrogen atmosphere in dry THF (5 mL for per mmol) and cooled to -55 °C on julabo. Then *n*-BuLi (2.05 equiv.) was added dropwise to the reaction mixture. The reaction mixture was stirred at -55 °C for 15 min. Then  $\beta$ -cyclocitral solution (1.0 equiv.) (5 mL dry THF) was added dropwise to the reaction mixture

over a period of 10 min at  $-55\text{ }^{\circ}\text{C}$ . And then allowed to warm at room temperature and stirred for another 1h. The stirring was continued till TLC showed complete consumption of starting materials. The reaction mixture was quenched by saturated  $\text{NH}_4\text{Cl}$  solution and then diluted with 20 mL of EtOAc. The whole reaction mixture was taken in a separatory funnel and extracted with 20 mL of water. The organic filtrate was separated and the aqueous part was again washed with 10 mL EtOAc, the combined organic filtrate dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated in a rotary evaporator under vacuum. The crude products were purified by flash chromatography (1:1 hexanes/EtOAc) to afford (**2j**).

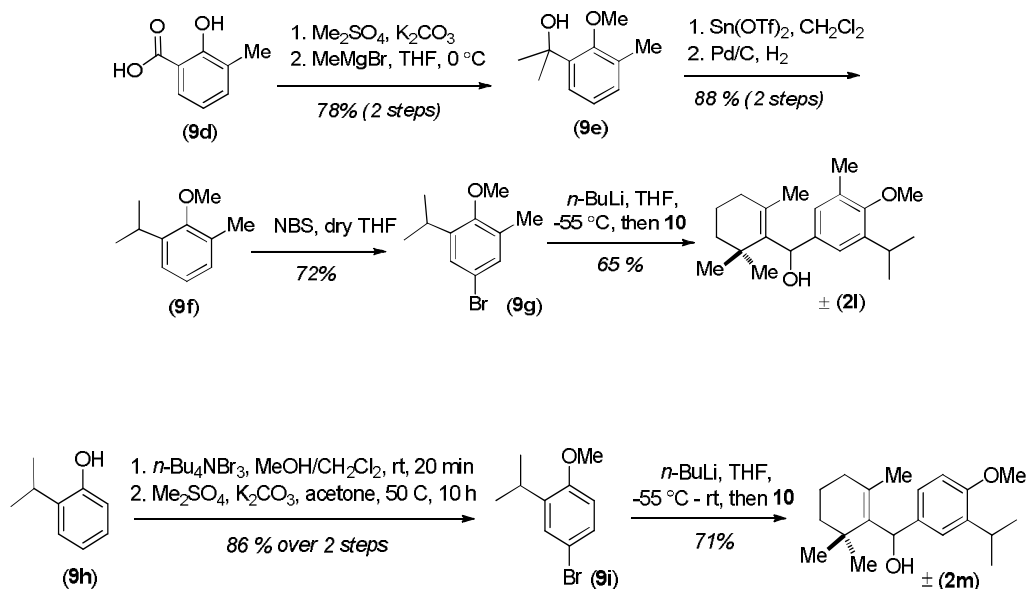


( $\pm$ )-(6-(hydroxymethyl)benzo[d][1,3]dioxol-5-yl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ( $\pm$ (**2j**): 71% yield as colorless solid,  $R_f = 0.5$  (50% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.91 (s, 1H), 6.86 (s, 1H), 5.97 (dd,  $J = 4.13, 1.46$  Hz, 2H), 5.66 (s, 1H), 5.32 (s, 1H), 4.89 (d,  $J = 11.94$  Hz, 1H), 4.4 (d,  $J = 11.96$  Hz, 1H), 2.19-2.05 (m, 2H), 1.76 (s, 3H), 1.7-1.67 (m, 2H), 1.54-1.48 (m, 2H), 1.21 (s, 3H), 0.75 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 146.8, 138.2, 134.7, 134.6, 134.2, 111.3, 109.4, 101.1, 70.4, 64.1, 39.5, 34.7, 33.7, 28.6, 28.1, 22.7, 19.1; **IR** (film)  $\nu_{\text{max}}$  3361, 2927, 1651, 1503, 1480, 1362, 1335, 1267, 1230, 1158, 1118, 1040, 971, 937, 854, 809, 737, 701  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  327.1577 [(M + Na) $^+$ ]; calculated for  $[\text{C}_{18}\text{H}_{24}\text{O}_4 + \text{Na}]^+$ : 327.1567; **MP** 116–118  $^{\circ}\text{C}$ .

**Scheme 3:** General procedure for synthesis of arylvinylcarbinols of  $\beta$ -cyclocitral (**2k-m**)

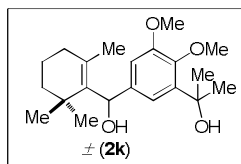






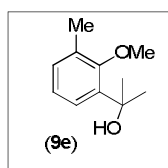
## References and Notes:

- Li, W.; Jing, H.; Bu, X.; Chang, T.; Jin, L.; Liang, Y. *Catal. Commun.* **2007**, *8*, 80.
- Peat, A. J.; Buchwald S. L. *J. Am. Chem. Soc.* **1996**, *118*, 1028.

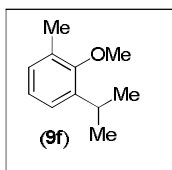


### $\pm$ -2-(5-(hydroxy(2,6,6-trimethylcyclohex-1-en-1-yl)methyl)-2,3-

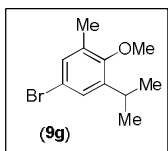
**dimethoxyphenyl)propan-2-ol**  $\pm$ (**2k**): 55% yield as colorless solid,  $R_f = 0.5$  (50% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.02 (s, 1H), 6.93 (s, 1H), 5.35 (s, 1H), 4.49 (s, 1H), 3.99 (s, 3H), 3.87 (s, 3H), 2.00 (t,  $J = 6.08$  Hz, 2H), 1.93 (brs, 1H), 1.69-1.63 (m, 2H), 1.59 (s, 3H), 1.58 (s, 3H) 1.56-1.53 (m, 2H), 1.43 (s, 3H), 1.19 (s, 3H), 1.05 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 152.3, 145.1, 140.3, 140.1, 140.0, 134.0, 115.7, 109.7, 73.0, 70.7, 61.0, 55.8, 39.8, 34.8, 33.7, 30.9, 28.8, 28.7, 21.7, 19.3; **IR** (film)  $\nu_{\text{max}}$  3445, 2930, 1584, 1456, 1303, 1128, 1066, 1008  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  371.2183  $[(\text{M} + \text{Na})]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{32}\text{O}_4 + \text{Na}]^+$ : 371.2193; **MP** 76–79  $^\circ\text{C}$ .



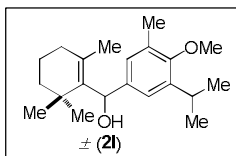
**2(2-methoxy-3-methylphenyl)propan-2-ol (9e):** 78% yield as colorless liquid gel,  $R_f = 0.4$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.17 (dd,  $J = 7.72, 1.04$  Hz, 1H), 7.07 (d,  $J = 7.36$  Hz, 1H), 6.96 (t,  $J = 7.6$  Hz, 1H), 4.17 (brs, 1H), 3.85 (s, 3H), 2.31 (s, 3H), 1.60 (s, 6H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 156.6, 140.4, 131.1, 130.8, 124.1, 123.8, 72.9, 61.1, 31.3, 16.9; **IR** (film)  $\nu_{\text{max}}$  3445, 2973, 2360, 1469, 1414, 1367, 1258, 1167, 1090, 1009, 955, 903, 833, 784, 761  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  203.1052  $[(\text{M} + \text{Na})]^+$ ; calculated for  $[\text{C}_{11}\text{H}_{16}\text{O}_2 + \text{Na}]^+$ : 203.1043.



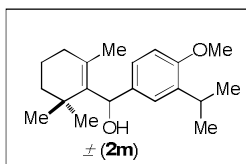
**(1-isopropyl-2-methoxy-3-methylbenzene) (9f):** 88% yield as colorless liquid gel,  $R_f = 0.4$  (in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.12 (d,  $J = 5.28$  Hz, 1H), 7.02 (s, 1H), 7.0 (d,  $J = 1.36$  Hz, 1H), 3.74 (s, 3H), 3.35 (septet,  $J = 6.92$  Hz, 1H), 2.31 (s, 3H), 1.23 (d,  $J = 6.92$  Hz, 6H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 155.8, 141.6, 130.8, 128.6, 124.2, 124.1, 60.8, 29.7, 26.4, 23.9; **IR** (film)  $\nu_{\text{max}}$  2968, 2928, 1458, 1418, 1364, 1260, 1220, 1167, 1090, 1012, 955, 904, 790, 760  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  163.1157  $[(\text{M} - \text{H})]^+$ ; calculated for  $[\text{C}_{11}\text{H}_{16}\text{O} - \text{H}]^+$ : 163.1117.



**(5-bromo-1-isopropyl-2-methoxy-3-methylbenzene) (9g):** 72% yield as colorless liquid gel,  $R_f = 0.4$  (in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.17 (d,  $J = 2.28$  Hz, 1H), 7.13 (d,  $J = 2.04$  Hz, 1H), 3.69 (s, 3H), 3.27 (pentate,  $J = 6.92$  Hz, 1H), 2.25 (s, 3H), 1.19 (d,  $J = 6.92$  Hz, 6H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 155.0, 143.9, 133.2, 131.3, 127.4, 117.0, 60.9, 26.6, 23.7; **IR** (film)  $\nu_{\text{max}}$  2964, 2828, 1577, 1470, 1423, 1384, 1363, 1332, 1259, 1236, 1205, 1169, 1110, 1065, 1012, 865, 824, 774, 704, 568  $\text{cm}^{-1}$ .

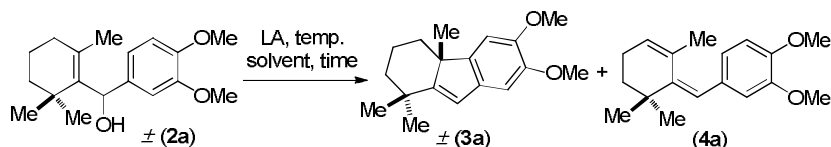


**(±)-(3-isopropyl-4-methoxy-5-methylphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2l):** 65% yield as colorless liquid gel,  $R_f = 0.4$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz, DMSO- $\text{D}_6$ )  $\delta$ : 7.0 (s, 1H), 6.94 (s, 1H), 5.13 (d,  $J = 4.8$  Hz, 1H), 5.03 (d,  $J = 4.92$  Hz, 1H), 3.56 (s, 3H), 3.15 (m, 1H), 2.13 (s, 3H), 1.86 (t,  $J = 6.0$  Hz, 2H), 1.52 (t,  $J = 2.92$  Hz, 2H), 1.37 (d,  $J = 5.76$  Hz, 2H), 1.28 (s, 3H), 1.06 (d,  $J = 6.8$  Hz, 6H), 1.0 (s, 3H), 0.92 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz, DMSO- $\text{D}_6$ )  $\delta$ : 153.7, 141.7, 140.0, 139.9, 131.4, 129.4, 126.6, 122.2, 69.1, 60.8, 40.3, 34.8, 33.5, 29.2, 29.1, 26.2, 24.4, 24.3, 21.9, 19.5, 16.8; **IR** (film)  $\nu_{\text{max}}$  3459, 2959, 2930, 1471, 1362, 1214, 1166, 1126, 1082, 1016, 871, 726  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  339.2328  $[(\text{M} + \text{Na})]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{32}\text{O}_2 + \text{Na}]^+$ : 339.2295.



**(±)-(3-isopropyl-4-methoxyphenyl)(2,6,6-trimethylcyclohex-1-en-1-yl)methanol ±(2m):** 71% yield as colorless liquid gel,  $R_f = 0.5$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz, DMSO- $\text{D}_6$ )  $\delta$ : 7.22 (d,  $J = 1.48$  Hz, 1H), 7.10 (d,  $J = 8.44$  Hz, 1H), 6.81 (d,  $J = 8.48$  Hz, 1H), 5.25 (d,  $J = 4.76$  Hz, 1H), 5.07 (d,  $J = 4.8$  Hz, 1H), 3.74 (s, 3H), 3.35 (s, 1H), 3.23 (m, 1H), 1.93 (t,  $J = 6.0$  Hz, 2H), 1.59 (d,  $J = 4.4$  Hz, 2H), 1.45 (t,  $J = 2.68$  Hz, 2H), 1.37 (s, 3H), 1.14 (s, 3H), 1.12 (s, 3H), 1.08 (s, 3H), 0.98 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz, DMSO- $\text{D}_6$ )  $\delta$ : 154.8, 140.2, 138.1, 135.1, 131.3, 124.6, 124.2, 110.2, 69.2, 55.7, 34.8, 33.6, 29.1, 29.0, 26.5, 23.2, 23.2, 21.9, 19.5; **IR** (film)  $\nu_{\text{max}}$  3450, 2958, 2929, 2867, 1500, 1463, 1362, 1244, 1166, 1089, 1036, 993, 811  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  325.2161  $[(\text{M} + \text{Na})]^+$ ; calculated for  $[\text{C}_{20}\text{H}_{30}\text{O}_2 + \text{Na}]^+$ : 325.2138.

**Table 1:** Optimization of cyclization of arylvinylcarbinols.



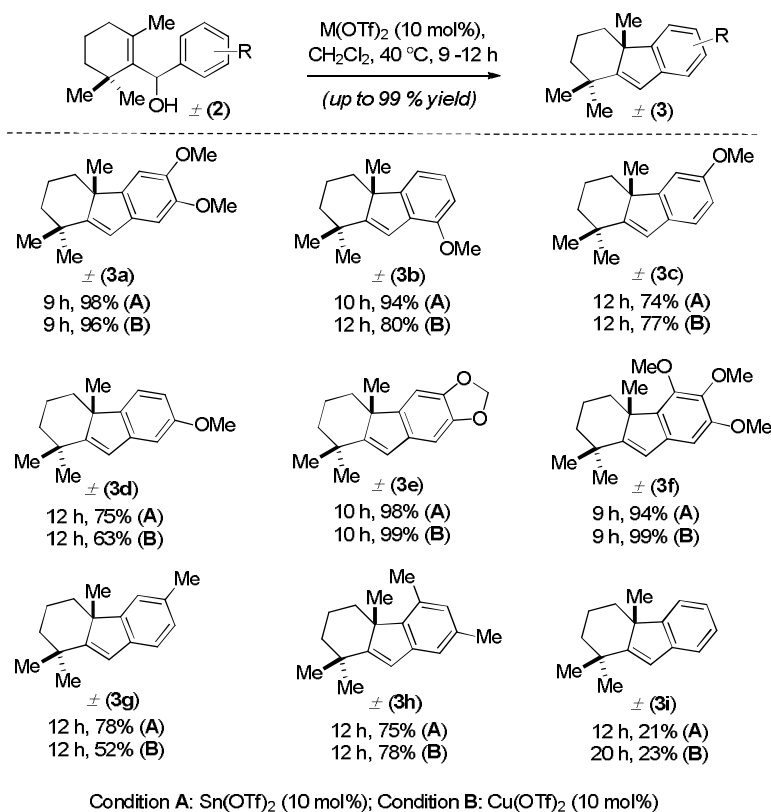
entry	catalyst (mol%)	solvent	temp	time	% of $\pm$ (3a)	% of (4a)
1.	Cu(OTf) <sub>2</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	24 h	78%	12%
2.	Sn(OTf) <sub>2</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	24 h	82%	11%
3.	Zn(OTf) <sub>2</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	12 h	60%	25%
4.	Bi(OTf) <sub>3</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	12 h	70%	16%
5.	In(OTf) <sub>3</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	12 h	56%	23%
6.	Sm(OTf) <sub>3</sub> (10 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	rt	12 h	45%	37%
7.	<b>Sn(OTf)<sub>2</sub> (10 mol%)</b>	<b>CH<sub>2</sub>Cl<sub>2</sub></b>	<b>40 °C</b>	<b>09 h</b>	<b>98%</b>	<b>00%</b>
8.	<b>Cu(OTf)<sub>2</sub> (10 mol%)</b>	<b>CH<sub>2</sub>Cl<sub>2</sub></b>	<b>40 °C</b>	<b>09 h</b>	<b>96%</b>	<b>00%</b>
9.	Cu(OTf) <sub>2</sub> (10 mol%)	PhMe	90 °C	12 h	88%	00%
10.	Cu(OTf) <sub>2</sub> (10 mol%)	THF	70 °C	18 h	60%	15%
11.	Cu(OTf) <sub>2</sub> (10 mol%)	Et <sub>2</sub> O	35 °C	12 h	72%	13%
12.	Sn(OTf) <sub>2</sub> (10 mol%)	dioxane	75 °C	12 h	80%	06%
13.	Sn(OTf) <sub>2</sub> (10 mol%)	DMF	90 °C	12 h	42%	40%
14.	Sn(OTf) <sub>2</sub> (10 mol%)	THF	70 °C	12 h	79%	15%
15.	Sn(OTf) <sub>2</sub> (10 mol%)	PhMe	90 °C	12 h	93%	00%
16.	Sn(OTf) <sub>2</sub> (10 mol%)	DMSO	90 °C	12 h	95%	00%
17.	Sn(OTf) <sub>2</sub> (10 mol%)	DCE	70 °C	12 h	93%	00%
18.	Cu(OTf) <sub>2</sub> (5 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	40 °C	18 h	90%	00%
19.	Sn(OTf) <sub>2</sub> (5 mol%)	CH <sub>2</sub> Cl <sub>2</sub>	40 °C	18 h	89%	00%

Reactions were carried out on a 1.0 mmol of  $\pm$ (2a) in 5 mL of solvent isolated yields reported after column chromatography.

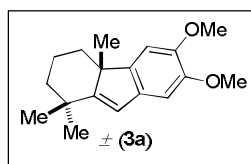
#### General procedure for metal triflate-catalyzed cyclization of arylvinylcarbinols:

In an oven-dried round-bottom flask, arylcarbinols of  $\beta$ -cyclocitral (1.0 mmol; 1.0 equiv.) and Sn(OTf)<sub>2</sub> (0.1 mmol; 10 mol%) [**Condition A**] or Cu(OTf)<sub>2</sub> (0.1 mmol; 10 mol%) [**Condition B**] were taken in dichloromethane (5 mL). The round-bottom flask was stirred at 40 °C for indicated time (9-12 h). Upon completion of the reactions, (TLC showed complete consumption of starting material) the reaction mixture was quenched by saturated NaHCO<sub>3</sub>

solution and diluted with 5 mL of dichloromethane. The whole reaction mixture was taken in a separatory funnel and extracted with 5 mL of water. The organic filtrate was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated in a rotary evaporator under vacuum. The crude products were purified by flash chromatography (10:1 hexanes/EtOAc) to afford Friedel-Crafts alkylation products.

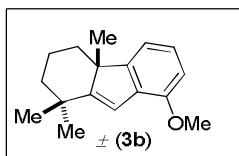


**Figure 1:** Substrates scope of metal triflate-catalyzed cyclization.

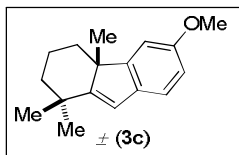


(±)-6,7-dimethoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene (**3a**): 98% yield as colorless gel, *R<sub>f</sub>* = 0.6 (10% EtOAc in hexane); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ: 6.89 (s, 1H), 6.85 (s, 1H), 6.31 (s, 1H), 3.92 (s, 3H), 3.91 (s, 3H), 2.13 (qd, *J* = 12.68, 2.92 Hz, 1H), 1.97 (qt, *J* = 14.36, 3.84 Hz, 1H), 1.70-1.60 (m, 2H), 1.37 (s, 3H), 1.31 (s, 3H), 1.26 (s, 3H), 1.12 (td, *J* = 12.96, 3.88 Hz, 1H), 1.00 (td, *J* = 13.2, 3.8 Hz, 1H); <sup>13</sup>C NMR (100 MHz,

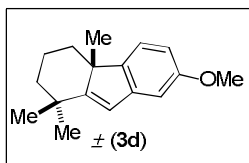
CDCl<sub>3</sub>)  $\delta$ : 163.0, 148.0, 147.6, 146.6, 134.6, 120.2, 105.7, 104.4, 56.4, 56.1, 51.0, 42.7, 38.4, 35.5, 31.3, 25.3, 23.6, 19.9; **IR** (film)  $\nu_{\max}$  3059, 2925, 1609, 1573, 1487, 1469, 1409, 1382, 1317, 1233, 1213, 1151, 1079, 1032, 992, 864, 841, 787, 743, 670 cm<sup>-1</sup>; **HRMS** (ESI)  $m/z$  273.1849 [(M + H)<sup>+</sup>]; calculated for [C<sub>18</sub>H<sub>24</sub>O<sub>2</sub> + H]<sup>+</sup>: 273.1849.



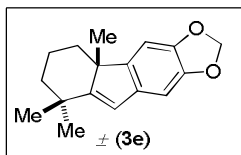
(±)-**8-methoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene**  $\pm$ (**3b**): 94% yield as colorless solid,  $R_f$  = 0.5 (5% EtOAc in hexane); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$ : 7.15 (t,  $J$  = 7.56 Hz, 1H), 6.92 (d,  $J$  = 7.4 Hz, 1H), 6.76 (d,  $J$  = 8.08 Hz, 1H), 6.56 (s, 1H), 3.91 (s, 3H), 2.17 (dq,  $J$  = 12.72, 4.72 Hz, 1H), 1.99 (qt,  $J$  = 13.92, 3.84 Hz, 1H), 1.71-1.57 (m, 2H), 1.39 (s, 3H), 1.34 (s, 3H), 1.27 (s, 3H), 1.17-0.98 (m, 2H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$ : 162.7, 157.3, 152.7, 130.2, 125.3, 116.5, 114.0, 108.4, 55.4, 51.5, 42.8, 38.0, 35.6, 31.3, 25.3, 23.4, 19.8; **IR** (film)  $\nu_{\max}$  2925, 1593, 1574, 1479, 1361, 1272, 1247, 1180, 1108, 1076, 1032, 971, 863, 784, 740, 672 cm<sup>-1</sup>; **HRMS** (ESI)  $m/z$  243.1751 [(M + H)<sup>+</sup>]; calculated for [C<sub>17</sub>H<sub>22</sub>O + H]<sup>+</sup>: 243.1743; **MP** 47–49 °C.



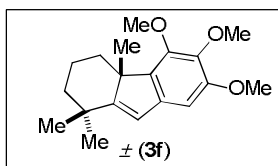
(±)-**6-methoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene**  $\pm$ (**3c**): 77% yield as colorless gel,  $R_f$  = 0.5 (5% EtOAc in hexane); **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>)  $\delta$ : 7.16 (d,  $J$  = 8.12 Hz, 1H), 6.83 (d,  $J$  = 2.32 Hz, 1H), 6.74 (dd,  $J$  = 8.12, 2.36 Hz, 1H), 6.29 (s, 1H), 3.81 (s, 3H), 2.10 (dd,  $J$  = 12.72, 1.52 Hz, 1H), 1.95 (qt, 14.4,  $J$  = 3.8 Hz, 1H), 1.66-1.59 (m, 2H), 1.36 (s, 3H), 1.28 (s, 3H), 1.23 (s, 3H), 1.15-0.97 (m, 2H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>)  $\delta$ : 162.1, 157.4, 157.0, 135.2, 120.6, 120.1, 111.1, 108.2, 55.5, 51.0, 42.7, 38.1, 35.4, 31.3, 25.5, 23.7, 19.8; **IR** (film)  $\nu_{\max}$  3061, 2923, 1733, 1597, 1574, 1507, 1470, 1382, 1370, 1282, 1246, 1217, 1202, 1174, 1083, 1035, 970, 943, 918, 860, 802, 751, 670, 623 cm<sup>-1</sup>; **HRMS** (ESI)  $m/z$  243.1739 [(M + H)<sup>+</sup>]; calculated for [C<sub>17</sub>H<sub>22</sub>O + H]<sup>+</sup>: 243.1743.



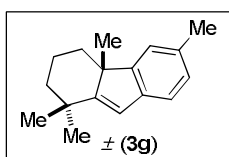
(±)-7-methoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene  $\pm$ (3d): 75% yield as colorless gel,  $R_f = 0.5$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.11 (d,  $J = 8.12$  Hz, 1H), 6.84 (d,  $J = 2.32$  Hz, 1H), 6.67 (dd,  $J = 8.12, 2.4$  Hz, 1H), 6.32 (s, 1H), 3.80 (s, 3H), 2.12 (dd,  $J = 12.8, 1.64$  Hz, 1H), 1.93 (qt,  $J = 14.52, 3.56$  Hz, 1H), 1.66-1.56 (m, 2H), 1.35 (s, 3H), 1.29 (s, 3H), 1.23 (s, 3H), 1.34-0.93 (m, 2H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 165.5, 158.7, 147.7, 143.5, 121.3, 120.6, 109.5, 106.2, 55.5, 50.3, 42.6, 38.3, 35.6, 31.3, 25.2, 23.6, 19.8; **IR** (film)  $\nu_{\text{max}}$  3063, 2923, 1613, 1470, 1370, 1344, 1281, 1235, 1187, 1149, 1109, 1041, 1022, 969, 939, 870, 805, 761, 673, 621, 583  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  243.1732  $[(M + H)]^+$ ; calculated for  $[\text{C}_{17}\text{H}_{22}\text{O} + \text{H}]^+$ : 243.1743.



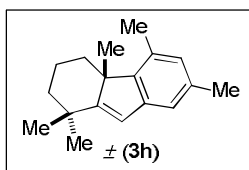
(±)-4b,8,8-trimethyl-5,6,7,8-tetrahydro-4bH-fluoreno[2,3-d][1,3]dioxole  $\pm$ (3e): 99% yield as colorless gel,  $R_f = 0.6$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.79 (s, 1H), 6.27 (s, 1H), 5.94 (s, 2H), 2.11 (d,  $J = 12.72$  Hz, 1H), 1.95 (q,  $J = 11.92$ , 1H), 1.65 (t,  $J = 11.6$  Hz, 2H), 1.35 (s, 3H), 1.31 (s, 3H), 1.25 (s, 3H), 1.12 (t,  $J = 13.44$  Hz, 1H), 0.99 (t,  $J = 13.16$  Hz, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 163.0, 149.2, 146.1, 145.0, 135.6, 120.3, 102.9, 101.6, 100.7, 50.7, 42.7, 38.3, 35.5, 31.2, 25.3, 23.5, 19.9; **IR** (film)  $\nu_{\text{max}}$  3063, 2924, 2767, 1622, 1499, 1471, 1316, 1284, 1240, 1187, 1168, 1145, 1108, 1040, 1011, 968, 943, 865, 836, 798, 670  $\text{cm}^{-1}$ ; **LRMS** (ESI)  $m/z$  255.2271  $[(M - H)]^+$ ; calculated for  $[\text{C}_{17}\text{H}_{20}\text{O}_2 - \text{H}]^+$ : 255.1380.



(±)-**5,6,7-trimethoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene** ±(**3f**): 99% yield as colorless solid,  $R_f = 0.5$  (10% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.66 (s, 1H), 6.24 (s, 1H), 3.97 (s, 3H), 3.88 (s, 3H), 3.87 (s, 3H), 2.48 (dq,  $J = 12.76, 2.96$  Hz, 1H), 1.96 (qt,  $J = 13.68, 3.72$  Hz, 1H), 1.67-1.59 (m, 1H), 1.47 (s, 3H), 1.29 (s, 3H), 1.26 (s, 3H), 1.17-1.08 (m, 2H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 165.2, 153.0, 149.5, 139.5, 138.3, 138.0, 120.2, 100.2, 60.89, 60.86, 56.2, 52.2, 42.6, 36.9, 35.5, 31.5, 25.4, 21.3, 19.6; **IR** (film)  $\nu_{\text{max}}$  2933, 2361, 1610, 1574, 1469, 1410, 1382, 1353, 1286, 1242, 1195, 1157, 1138, 1102, 1042, 1021, 994, 933, 907, 861, 790, 673  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  303.1943  $[(M + H)]^+$ ; calculated for  $[\text{C}_{19}\text{H}_{26}\text{O}_3 + \text{H}]^+$ : 303.1955; **MP** 89–91 °C.



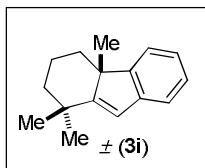
(±)-**1,1,4a,6-tetramethyl-2,3,4,4a-tetrahydro-1H-fluorene** ±(**3g**): 78% yield as colorless gel,  $R_f = 0.6$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.19 (d,  $J = 7.48$  Hz, 1H), 7.09 (s, 1H), 7.03 (d,  $J = 7.36$  Hz, 1H), 6.37 (s, 1H), 2.41 (s, 3H), 2.16 (d,  $J = 12.64$  Hz, 1H), 2.0 (q,  $J = 13.76$  Hz, 1H), 1.67 (t,  $J = 11.28$  Hz, 2H), 1.40 (s, 3H), 1.33 (s, 3H), 1.28 (s, 3H), 1.14 (t,  $J = 13.04$  Hz, 1H), 1.03 (t,  $J = 13.64$  Hz, 1H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 163.1, 155.5, 139.5, 126.9, 121.9, 120.5, 120.1, 50.7, 42.7, 38.0, 35.5, 31.3, 29.7, 25.3, 23.5, 21.6, 19.8; **IR** (film)  $\nu_{\text{max}}$  2997, 2924, 2865, 1599, 1467, 1370, 1285, 1176, 1138, 1032, 969, 861, 808, 780, 670  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  227.1799  $[(M)]^+$ ; calculated for  $[\text{C}_{17}\text{H}_{23}]^+$ : 227.1794.



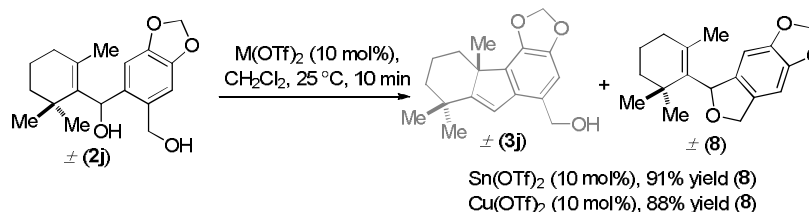
(±)-**1,1,4a,5,7-pentamethyl-2,3,4,4a-tetrahydro-1H-fluorene** ±(**3h**): 78% yield as colorless solid,  $R_f = 0.7$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.97 (s, 1H), 6.74 (s, 1H), 6.33 (s, 1H), 2.51 (d,  $J = 12.72$  Hz, 1H), 2.44 (s, 3H), 2.36 (s, 3H), 2.0 (q,  $J = 14.68$  Hz, 1H), 1.66 (d,  $J = 12.08$  Hz, 2H), 1.46 (s, 3H), 1.33 (s, 3H), 1.30 (s, 3H), 1.17 (t,  $J = 12.84$  Hz, 2H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 164.4, 149.1, 142.9, 135.9, 131.7, 127.6, 121.0, 119.0,



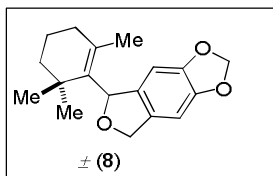
52.0, 42.2, 35.5, 35.4, 31.4, 31.6, 25.5, 21.2, 20.5, 19.5, 18.6; **IR** (film)  $\nu_{\max}$  2926, 2865, 1616, 1469, 1381, 1284, 1155, 1087, 1026, 967, 882, 858, 838, 761, 678  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  241.1955  $[(M + H)^+]$ ; calculated for  $[\text{C}_{18}\text{H}_{24} + \text{H}]^+$ : 241.1951; **MP** 59-60 °C.



**(±)-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene ±(3i)**: 23% yield as colorless gel,  $R_f$  = 0.5 (in hexane);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.26 (d,  $J$  = 7.32 Hz, 1H), 7.23 (d,  $J$  = 7.4 Hz, 1H), 7.18 (dt,  $J$  = 7.36, 1.28 Hz, 1H), 7.11 (dt,  $J$  = 7.36, 1.12 Hz, 1H), 6.36 (s, 1H), 2.15 (dd,  $J$  = 12.76, 1.8 Hz, 1H), 1.95 (qt,  $J$  = 13.68, 3.92 Hz, 1H), 1.66-1.59 (m, 2H), 1.36 (s, 3H), 1.29 (s, 3H), 1.24 (s, 3H), 1.14-0.94 (m, 2H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 164.1, 155.2, 142.2, 126.3, 124.0, 120.9, 120.7, 120.4, 50.9, 42.6, 38.0, 35.5, 31.3, 25.2, 23.4, 19.8; **IR** (film)  $\nu_{\max}$  3062, 2922, 1608, 1468, 1382, 1370, 1290, 1188, 1091, 969, 873, 849, 746, 671  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  213.1655  $[(M)^+]$ ; calculated for  $[\text{C}_{16}\text{H}_{22}\text{O}]^+$ : 213.1638.

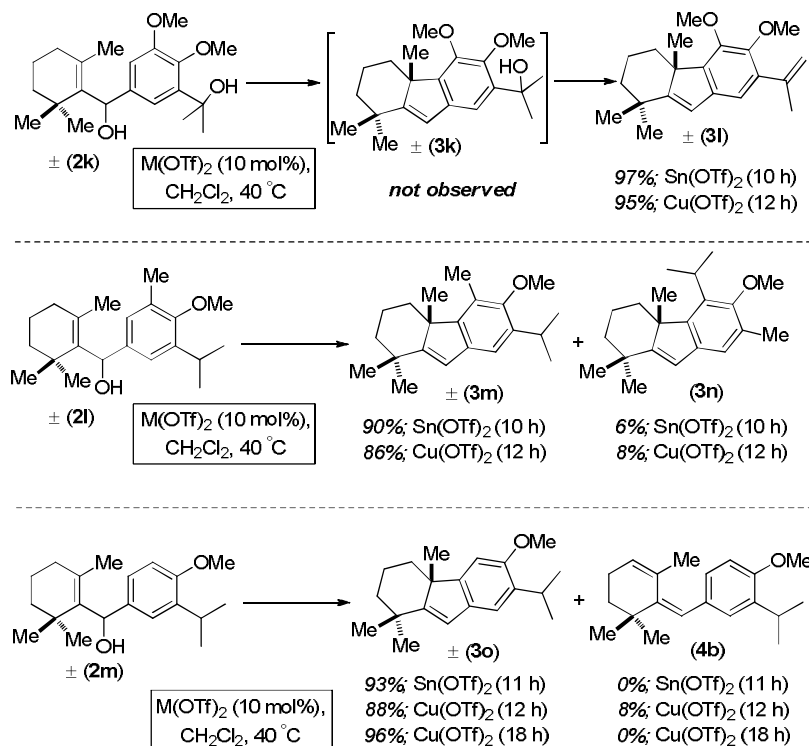


**Scheme 4:** Substrates scope using arylvinylcarbinols **(2j)**.

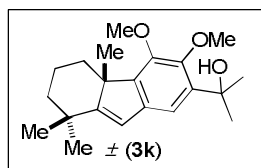


**(±)-5-(2,6,6-trimethylcyclohex-1-en-1-yl)-5,7-dihydro-[1,3]dioxolo[4,5-f]isobenzofuran ±(8)**: 91% yield as colorless solid,  $R_f$  = 0.45 (5% EtOAc in hexane);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.63 (s, 1H), 5.95 (dd,  $J$  = 9.76, 1.36 Hz, 2H), 5.68 (s, 1H), 5.02 (ABq,  $J$  = 11.76, 2.76 Hz, 2H), 2.64-1.88 (m, 2H), 1.61-1.58 (m, 2H), 1.52-1.49 (m, 2H), 1.32 (s, 3H), 1.13 (s, 6H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 147.5, 147.1, 137.1, 137.0, 131.9, 102.1, 101.4, 101.3,

77.2, 72.0, 34.6, 34.2, 29.7, 29.0, 28.3, 19.8, 19.4; **IR** (film)  $\nu_{\max}$  2928, 1503, 1476, 1362, 1335, 1269, 1244, 1157, 1128, 1040, 941, 835, 809, 767, 697  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  287.1651  $[(M + H)^+]$ ; calculated for  $[\text{C}_{18}\text{H}_{22}\text{O}_3 + \text{H}]^+$ : 287.1642; **MP** 101–103 °C.

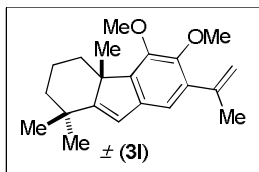


**Scheme 5:** Substrates scope using arylvinylcarbinols (**2k-m**).

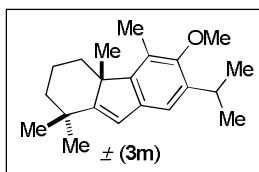


( $\pm$ )-2-(5,6-dimethoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluoren-7-yl)propan-2-ol  
 $\pm$ (**3k**): 90% yield as colorless solid,  $R_f = 0.4$  (10% EtOAc in hexane); **<sup>1</sup>H NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.84 (s, 1H), 6.26 (s, 1H), 3.90 (s, 3H), 3.86 (s, 3H), 3.16 (dd,  $J = 12.96, 0.88$  Hz, 1H), 1.91 (qt,  $J = 12.68, 2.8$  Hz, 1H), 1.78 (s, 3H), 1.74 (s, 3H), 1.64-1.61 (m, 2H), 1.59-1.53 (m, 1H), 1.56 (s, 3H), 1.31 (s, 3H), 1.28 (s, 3H), 1.12 (m, 2H); **<sup>13</sup>C NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 165.2, 151.8, 143.8, 143.6, 139.7, 139.2, 119.9, 103.8, 74.7, 61.0, 56.1, 55.6, 42.2, 37.4, 35.6, 33.2, 32.4, 32.0, 26.2, 22.3, 20.2; **IR** (film)  $\nu_{\max}$  3464, 2994, 2924, 2863, 1621, 1596, 1444, 1418, 1358, 1325, 1287, 1244, 1177, 1153, 1084, 1036, 1015, 962, 865, 834, 817, 738,

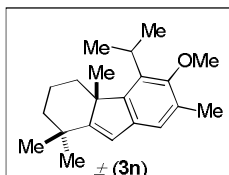
677  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  331.2274  $[(M + H)]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{30}\text{O}_3 + \text{H}]^+$ : 331.2268;  
**MP** 126–128 °C.



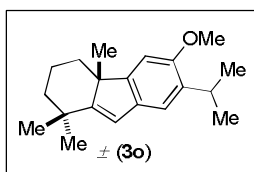
**(±)-5,6-dimethoxy-1,1,4a-trimethyl-7-(prop-1-en-2-yl)-2,3,4,4a-tetrahydro-1H-fluorene**  
**±(3l)**: 97% yield as colorless solid,  $R_f = 0.3$  (2% EtOAc in hexane);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.81 (s, 1H), 6.28 (s, 1H), 5.32 (d,  $J = 14.56$  Hz, 1H), 4.90 (s, 1H), 3.86 (s, 3H), 3.76 (d,  $J = 8.64$  Hz, 3H), 2.47 (d,  $J = 12.52$ , 1H), 2.05 (d,  $J = 28.52$  Hz, 3H), 1.88 (qt,  $J = 13.56$ , 3.28 Hz, 1H), 1.6 (m, 2H), 1.27 (s, 3H), 1.23 (s, 3H), 1.13–1.07 (m, 2H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 164.7, 151.3, 143.4, 143.1, 141.6, 138.8, 134.9, 121.5, 116.7, 103.6, 61.4, 55.8, 55.3, 42.5, 39.5, 35.6, 31.8, 25.4, 25.0, 21.0, 19.8; **IR** (film)  $\nu_{\text{max}}$  2929, 2361, 1608, 1590, 1455, 1417, 1349, 1283, 1267, 1242, 1207, 1155, 1094, 1047, 1022, 900, 864, 792, 671, 617  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  313.2150  $[(M + H)]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{28}\text{O}_2 + \text{H}]^+$ : 313.2162; **MP** 70–73 °C.



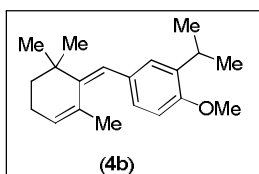
**(±)-7-isopropyl-6-methoxy-1,1,4a,5-tetramethyl-2,3,4,4a-tetrahydro-1H-fluorene** **±(3m)**:  
90% yield as colorless gel,  $R_f = 0.4$  (in hexane);  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.0 (s, 1H), 6.27 (s, 1H), 3.69 (s, 3H), 3.33 (m, 1H), 2.48 (d,  $J = 12.56$  Hz, 1H), 2.37 (s, 3H), 1.84 (qt,  $J = 13.92$ , 3.32 Hz, 1H), 1.63–1.60 (m, 2H), 1.42 (s, 3H), 1.27 (s, 3H), 1.24 (s, 6H), 1.22 (s, 3H), 1.20–1.08 (m, 2H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 163.6, 153.4, 150.7, 139.3, 138.4, 125.7, 120.8, 115.5, 61.1, 52.7, 42.1, 35.5, 35.3, 31.6, 26.6, 25.6, 24.2., 20.7, 19.5, 12.0; **IR** (film)  $\nu_{\text{max}}$  2960, 2928, 2867, 1464, 1410, 1380, 1365, 1307, 1287, 1241, 1209, 1131, 1022, 882, 785, 675  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  321.2207  $[(M + \text{Na})]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{30}\text{O} + \text{Na}]^+$ : 321.2189; **MP** 67–69 °C.



(±)-5-isopropyl-6-methoxy-1,1,4a,7-tetramethyl-2,3,4,4a-tetrahydro-1H-fluorene  $\pm$ (3n): 8% yield as colorless gel,  $R_f = 0.5$  (in hexane);  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.92 (s, 1H), 6.22 (s, 1H), 3.76 (s, 3H), 3.41 (m, 1H), 2.45 (d,  $J = 12.3$  Hz, 1H), 2.30 (s, 3H), 1.93 (qt,  $J = 13.8, 3.4$  Hz, 1H), 1.62-1.59 (m, 2H), 1.41 (s, 3H), 1.39 (d,  $J = 7.85$  Hz, 3H), 1.33 (d,  $J = 7.08$  Hz, 3H), 1.26 (s, 6H), 1.24 (s, 3H), 1.21-1.07 (m, 2H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$ : 163.6, 156.3, 150.1, 138.2, 136.4, 129.4, 121.1, 120.5, 70.0, 53.0, 42.0, 36.5, 35.3, 31.7, 28.8, 25.7, 22.4, 21.7, 21.6, 19.7, 17.1; **IR** (film)  $\nu_{\text{max}}$  2926, 1449, 1357, 1232, 1020, 857, 672  $\text{cm}^{-1}$ . **HRMS** (ESI)  $m/z$  299.2340  $[(M + H)]^+$ ; calculated for  $[\text{C}_{21}\text{H}_{30}\text{O} + \text{H}]^+$ : 299.2369.



(±)-7-isopropyl-6-methoxy-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluorene  $\pm$ (3o): 96% yield as colorless solid,  $R_f = 0.4$  (in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3 + \text{DMSO-}D_6$ )(4:1)  $\delta$ : 7.00 (s, 1H), 6.67 (s, 1H), 6.18 (s, 1H), 3.72 (s, 3H), 3.19 (septet,  $J = 6.96$  Hz, 1H), 1.99 (d,  $J = 12.68$  Hz, 1H), 1.84 (q,  $J = 14.08$  Hz, 1H), 1.51 (t,  $J = 12.44$  Hz, 2H), 1.24 (s, 3H), 1.16 (s, 3H), 1.11 (s, 3H), 1.10 (d,  $J = 1.48$  Hz, 3H), 1.08 (d,  $J = 1.16$  Hz, 3H), 1.02-0.84 (m, 2H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3 + \text{DMSO-}D_6$ )  $\delta$ : 161.8, 154.4, 153.6, 134.7, 134.5, 120.2, 50.9, 42.6, 38.1, 35.3, 31.2, 55.8, 50.9, 42.6, 38.1, 35.3, 31.2, 26.6, 25.4, 23.7, 23.0, 22.9, 19.7; **IR** (film)  $\nu_{\text{max}}$  2959, 1620, 1593, 1572, 1483, 1463, 1417, 1381, 1369, 1309, 1289, 1221, 1199, 1082, 1065, 1032, 969, 941, 888, 857, 785, 732, 669  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  285.2217  $[(M + H)]^+$ ; calculated for  $[\text{C}_{20}\text{H}_{28}\text{O} + \text{H}]^+$ : 285.2213; **MP** 67–69 °C.

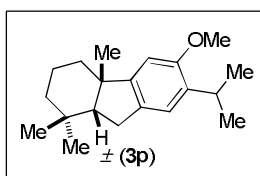


**(Z)-2-isopropyl-1-methoxy-4-((2,6,6-trimethylcyclohex-2-en-1-ylidene)methyl)benzene**

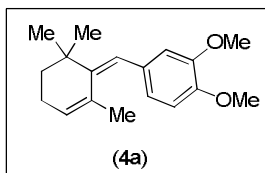
**(4b)**: 8% yield as colorless gel,  $R_f = 0.5$  (in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.08 (d,  $J = 2.0$  Hz, 1H), 7.00 (dd,  $J = 1.68, 8.32$  Hz, 1H), 6.77 (d,  $J = 8.32$  Hz, 1H), 6.41 (s, 1H), 5.57-5.56 (m, 1H), 3.85 (s, 3H), 3.34 (septet,  $J = 6.92$  Hz, 1H), 2.25-2.23 (m, 2H), 1.61 (t,  $J = 6.44$  Hz, 2H), 1.52 (d,  $J = 1.32$  Hz, 3H), 1.23 (d,  $J = 6.92$  Hz, 6H), 1.18 (s, 6H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 155.2, 146.3, 135.9, 132.5, 132.3, 128.4, 127.3, 127.2, 121.3, 109.6, 55.4, 37.3, 35.8, 27.2, 26.6, 24.0, 23.0, 22.7; **IR** (film)  $\nu_{\text{max}}$  2960, 2926, 2870, 1660, 1600, 1496, 1463, 1454, 1382, 1362, 1287, 1249, 1171, 1089, 1034, 813, 737  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  285.2209  $[(\text{M} + \text{H})^+]$ ; calculated for  $[\text{C}_{20}\text{H}_{28}\text{O} + \text{H}]^+$ : 285.2213.



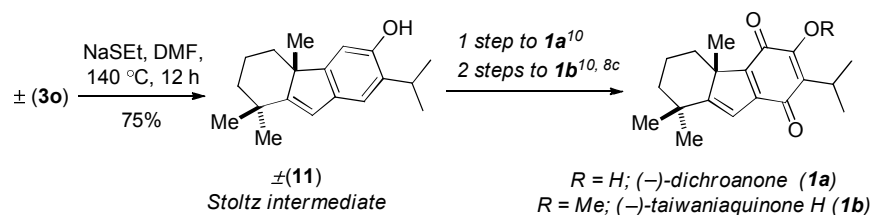
**Scheme 6:** Hydrogenation of carbocyclic compound  $\pm(3\text{o})$



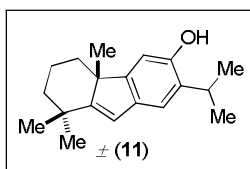
**( $\pm$ )-7-isopropyl-6-methoxy-1,1,4a-trimethyl-2,3,4,4a,9,9a-hexahydro-1H-fluorene  $\pm(3\text{p})$ :** 99% yield as colorless solid,  $R_f = 0.6$  (in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.65 (s, 1H), 6.51 (s, 1H), 3.73 (s, 3H), 3.19 (septet,  $J = 6.88$  Hz, 1H), 2.61 (d,  $J = 9.56$  Hz, 2H), 1.78 (t,  $J = 9.56$  Hz, 1H), 1.60-1.48 (m, 1H), 1.39-1.27 (m, 3H), 1.35 (s, 3H), 1.20-1.16 (m, 2H), 1.11 (t,  $J = 6.84$  Hz, 6H), 1.03 (s, 3H), 0.86 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 154.7, 152.6, 134.7, 132.6, 121.9, 104.2, 57.9, 55.7, 45.4, 36.4, 35.1, 33.3, 32.2, 31.2, 29.5, 26.7, 25.6, 23.1, 22.9, 19.0; **IR** (film)  $\nu_{\text{max}}$  2956, 2930, 2863, 1490, 1463, 1414, 1293, 1221, 1197, 1178, 1056, 1045, 881, 839  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  304.2662  $[(\text{M} + \text{NH}_4)^+]$ ; calculated for  $[\text{C}_{20}\text{H}_{30}\text{O} + \text{NH}_4]^+$ : 304.2635; **MP** 57–59  $^\circ\text{C}$ .



**(Z)-1,2-dimethoxy-4-((2,6,6-trimethylcyclohex-2-en-1-ylidene)methyl)benzene (4a):** Colorless gel,  $R_f = 0.6$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 6.76-6.71 (m, 3H), 6.34 (s, 1H), 5.52 (s, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 2.19-2.17 (m, 2H), 1.55 (t,  $J = 6.44$  Hz, 2H), 1.47 (d,  $J = 1.33$  Hz, 3H), 1.12 (s, 6H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 148.1, 147.4, 133.0, 132.1, 128.7, 121.7, 120.9, 112.4, 110.5, 55.83, 55.81, 37.2, 35.8, 27.1, 23.9, 22.9; **IR** (film)  $\nu_{\text{max}}$  2957, 2925, 1658, 1595, 1513, 1464, 1453, 1418, 1268, 1255, 1239, 1156, 1137, 1029, 809, 764  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$  273.1858  $[(M + H)]^+$ ; calculated for  $[\text{C}_{18}\text{H}_{24}\text{O}_2 + \text{H}]^+$ : 273.1849.



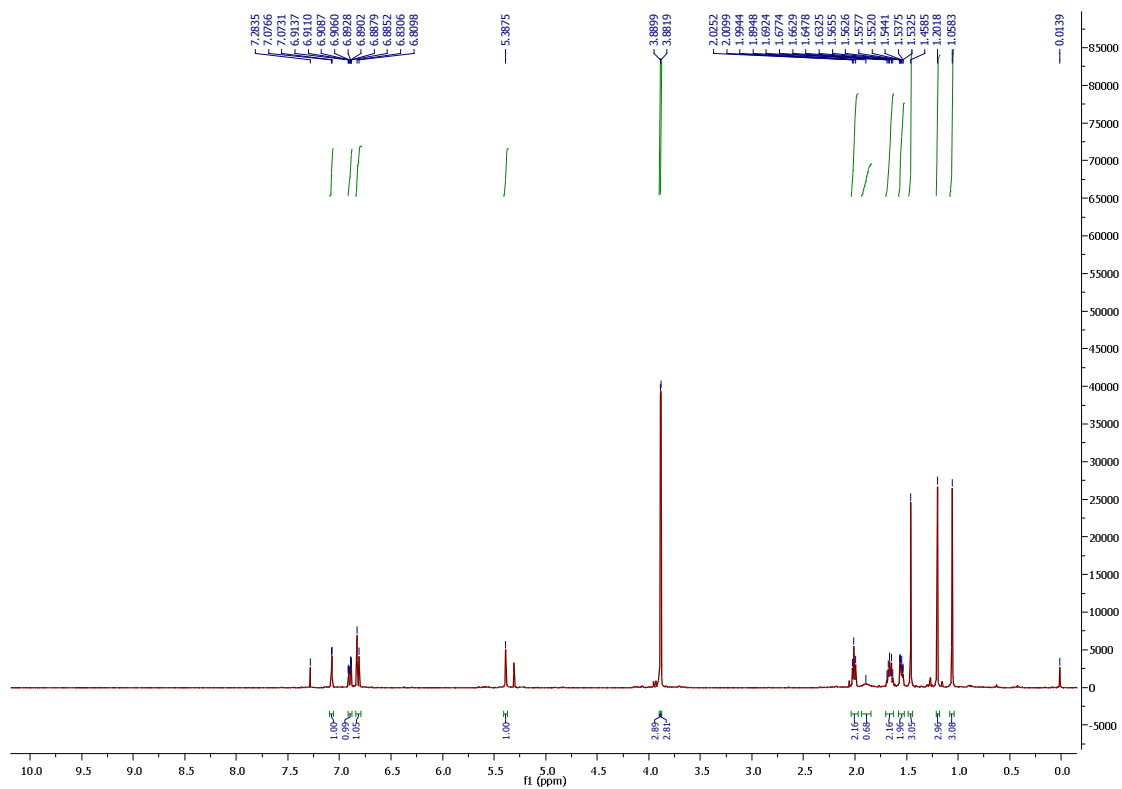
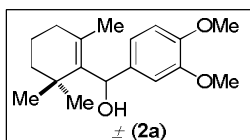
**Scheme 7:** Formal total synthesis of (±)-dichroanone (**1a**) and (±)-taiwaniaquinone H (**1b**)



**(±)-7-isopropyl-1,1,4a-trimethyl-2,3,4,4a-tetrahydro-1H-fluoren-6-ol  $\pm(11)$ :** 75% yield as colorless solid,  $R_f = 0.4$  (5% EtOAc in hexane);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$ : 7.12 (s, 1H), 6.67 (s, 1H), 6.30 (s, 1H), 4.31 (brs, 1H), 3.20 (septet,  $J = 6.84$  Hz, 1H), 1H), 2.06 (dd,  $J = 12.7, 1.36$  Hz, 1H), 1.92 (qt,  $J = 13.56, 3.52$  Hz, 1H), 1.65-1.58 (m, 2H), 1.34 (s, 3H), 1.29 (s, 3H), 1.27 (dd,  $J = 6.84, 4.08$  Hz, 6H), 1.23 (s, 3H), 1.14-0.97 (m, 2H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$ : 161.9, 154.1, 150.0, 135.3, 132.0, 120.3, 117.8, 109.2, 50.8, 42.7, 38.1, 35.4, 31.3, 27.1, 25.5, 23.7, 22.9, 22.88, 19.9; **IR** (film)  $\nu_{\text{max}}$  2959, 2925, 2871, 1621, 1593, 1484, 1464, 1417, 1360, 1310, 1289, 1221, 1200, 1065, 1033, 888, 840, 786, 670  $\text{cm}^{-1}$ ; **HRMS** (ESI)  $m/z$

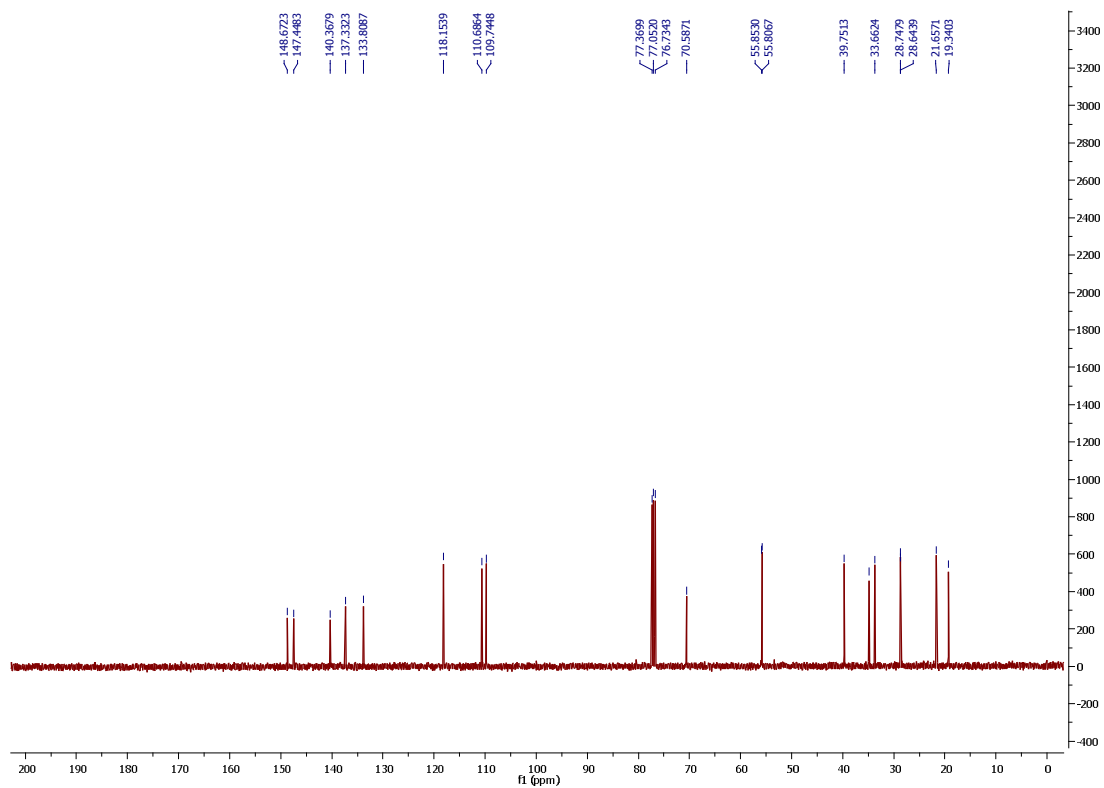
271.2046 [(M + H)<sup>+</sup>]; calculated for [C<sub>19</sub>H<sub>26</sub>O + H]<sup>+</sup>: 271.2056; **MP** 104–106 °C, [lit. (McFadden, R. M.; Stoltz, B. M. *J. Am. Chem. Soc.* **2006**, 128, 7738)]; **MP** 105–106 °C.

## <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, and Mass Spectra

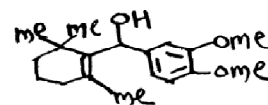


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound **±(2a)**





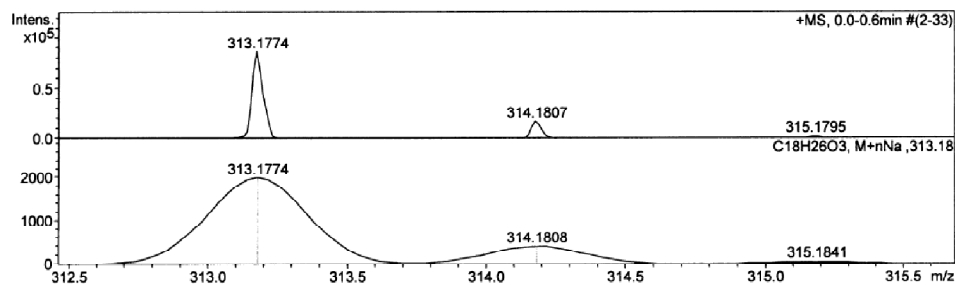
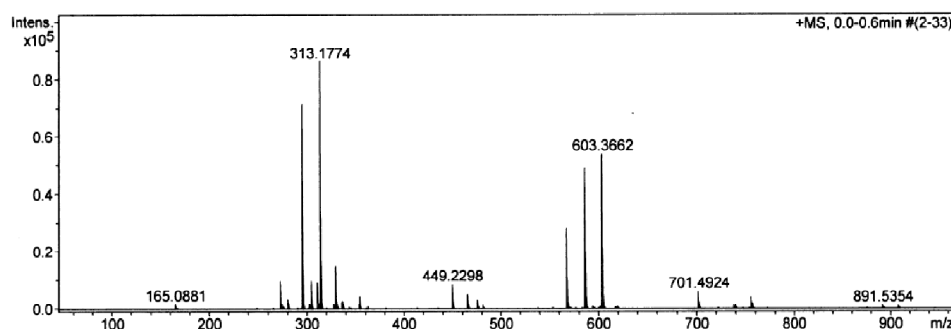
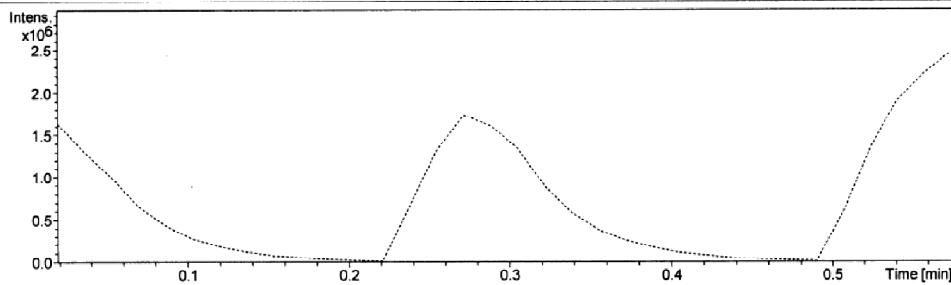
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound **2a**



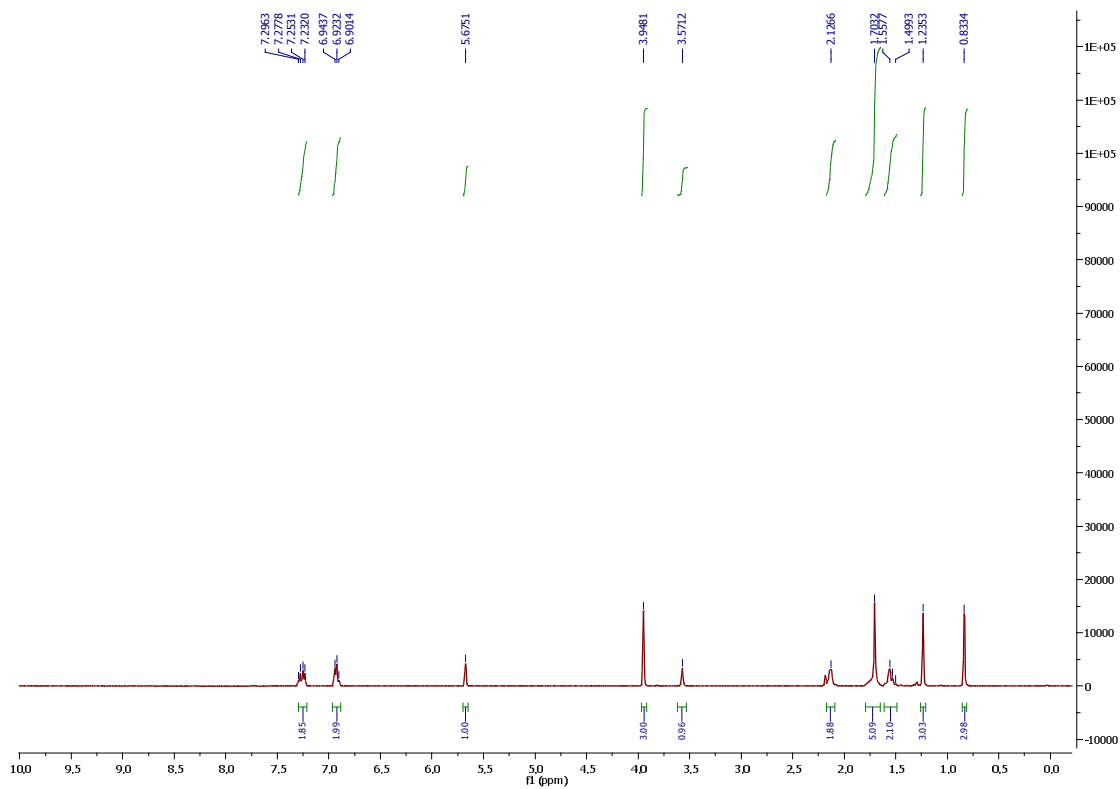
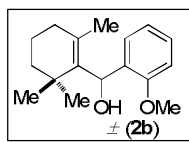
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Operator IISERB  
Instrument micrOTOF-Q II 10330

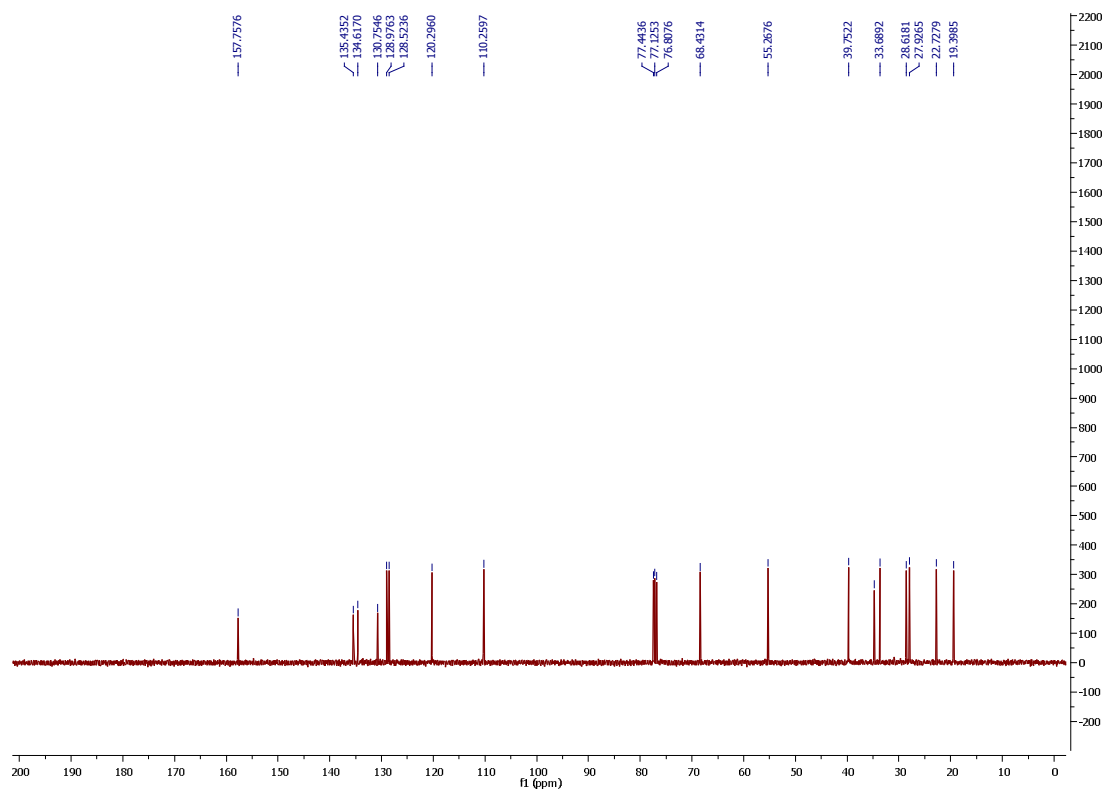
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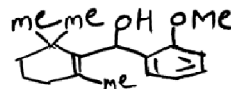
Scanned copy of mass spectrum (HRMS) of  $\pm(2a)$



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound ±(2b)



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{b})$

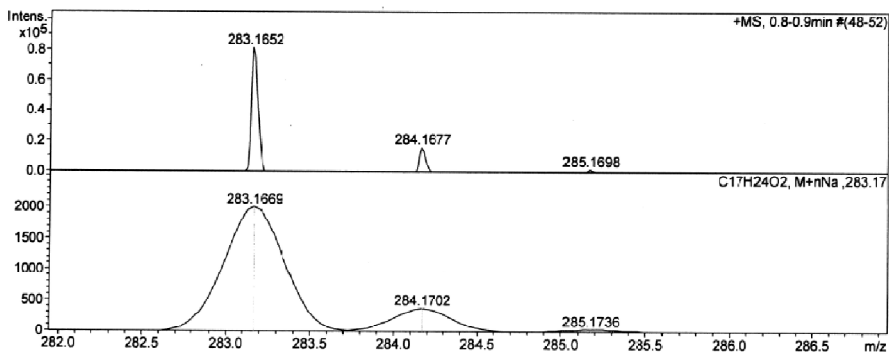
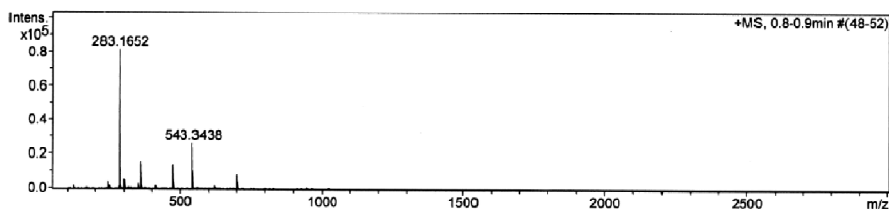
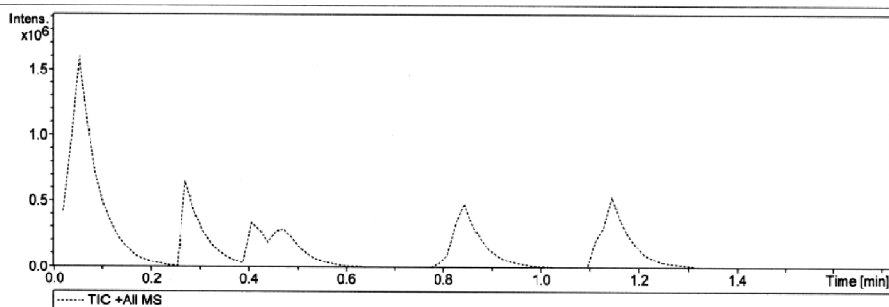


### Display Report

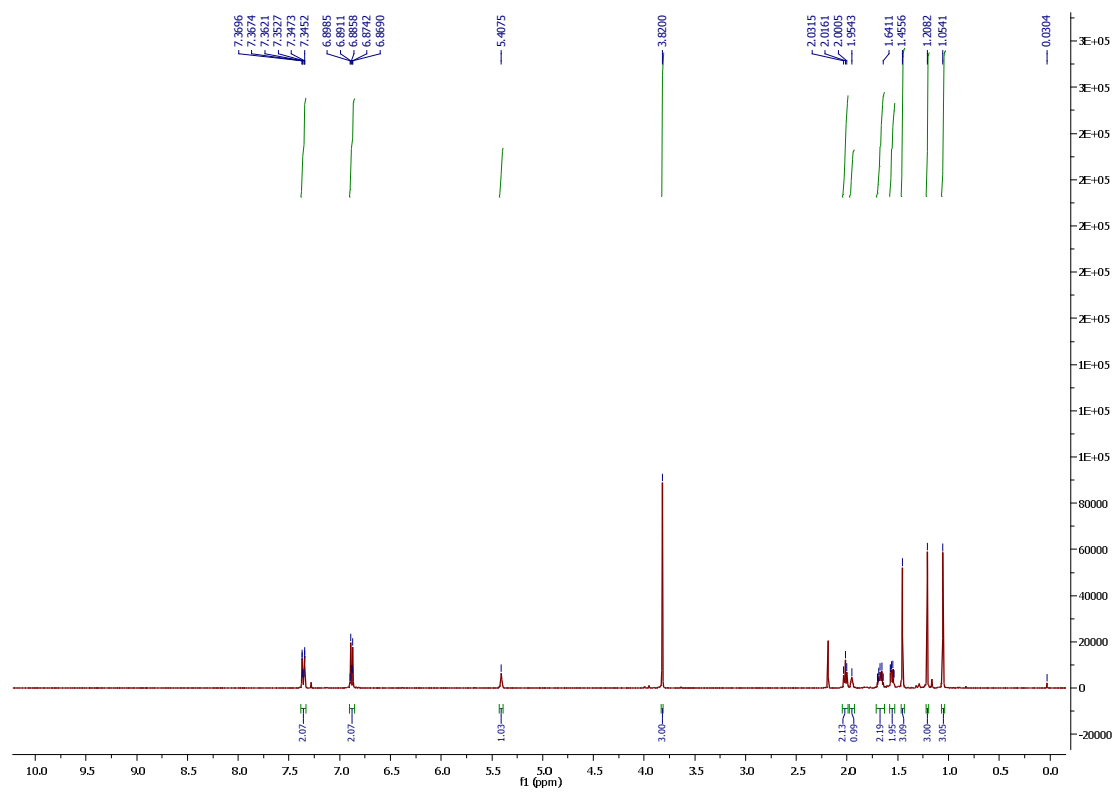
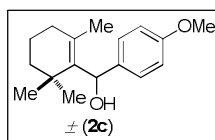
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Sample Name AB-BK-2-226 Instrument micrOTOF-Q II 10330  
Comment

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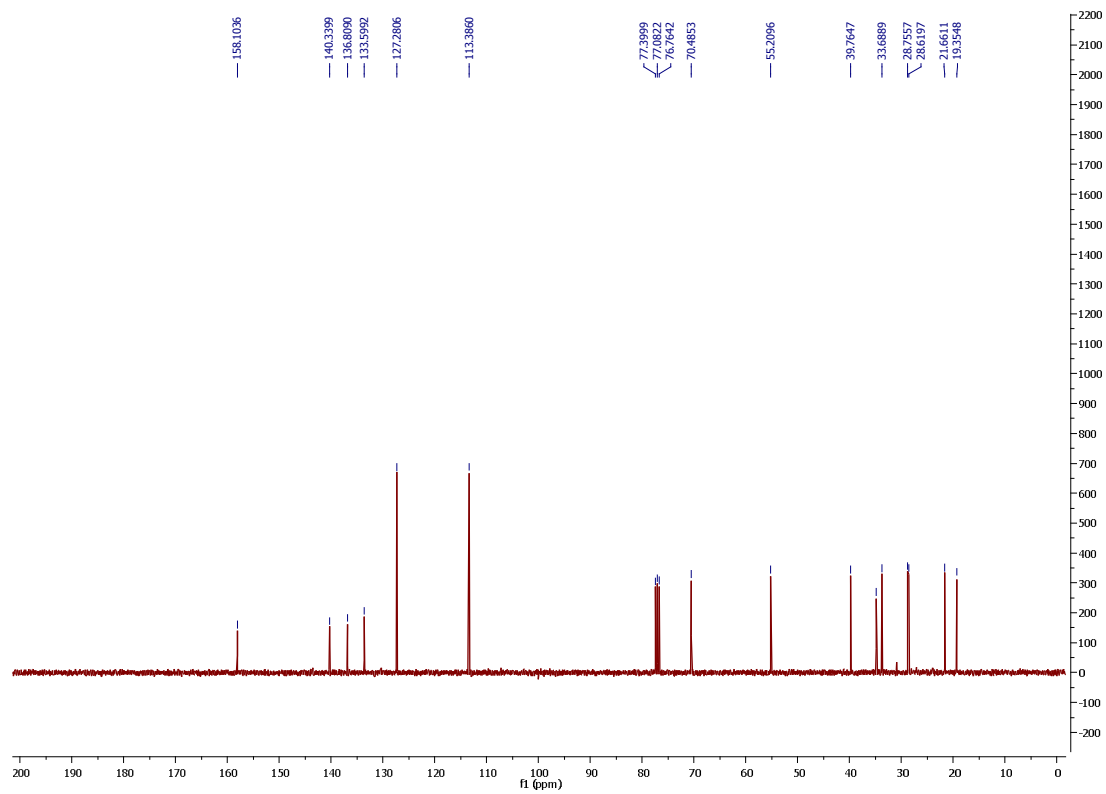
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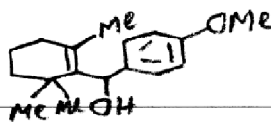
Scanned copy of mass spectrum (HRMS) of  $\pm(2b)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2c)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{c})$



### Display Report

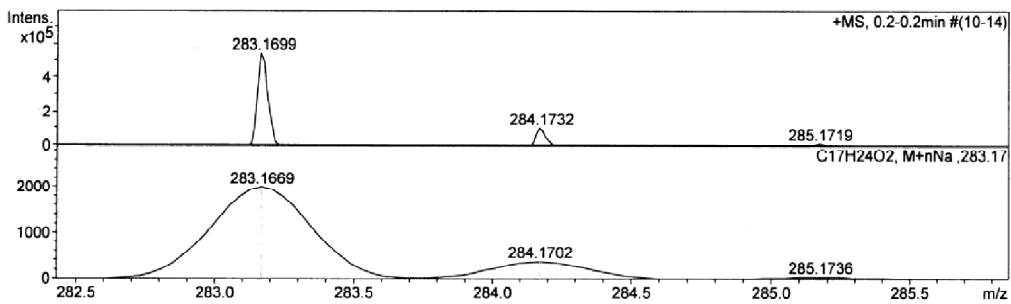
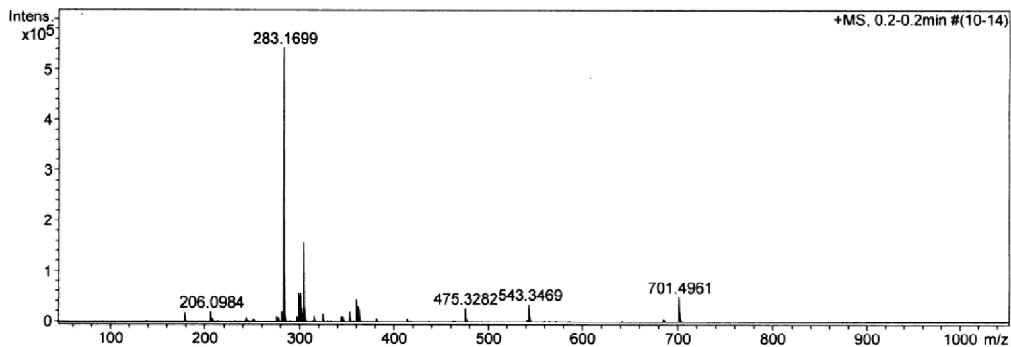
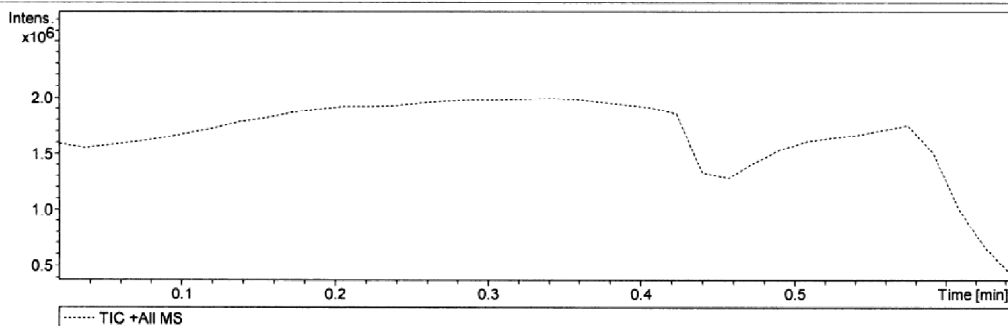
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Operator IISERB  
Instrument micrOTOF-Q II 10330

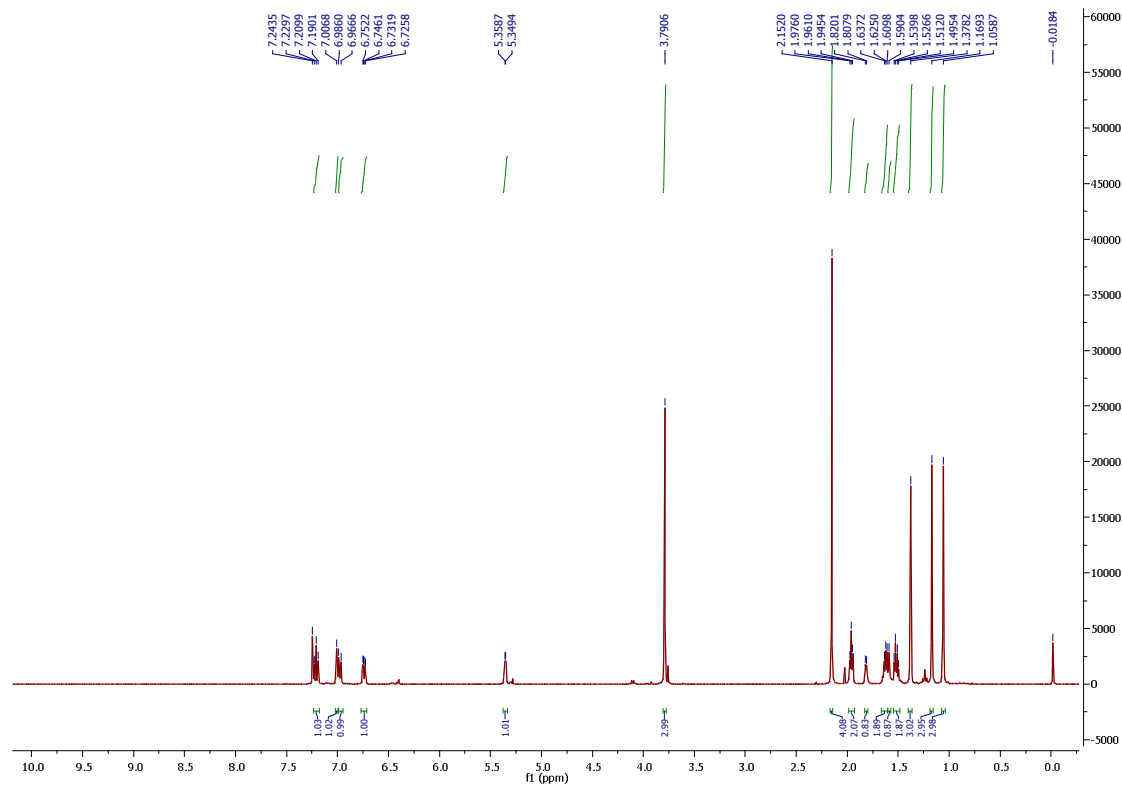
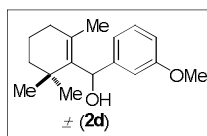
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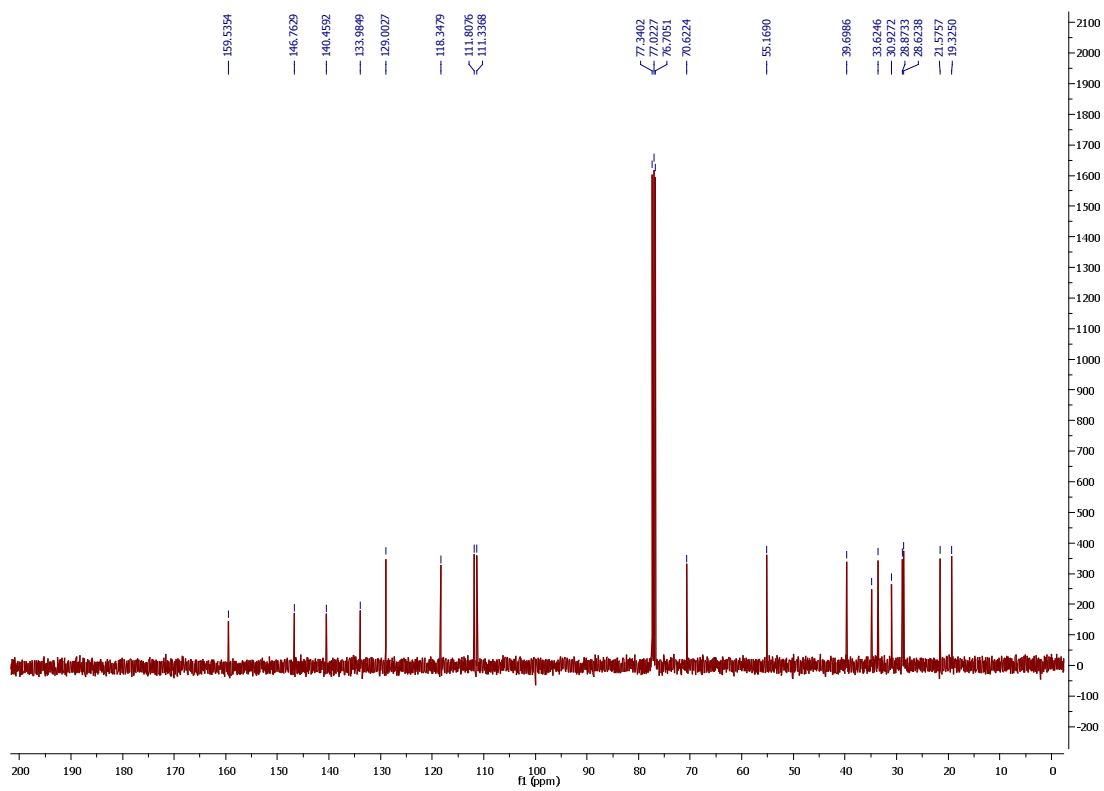


Scanned copy of mass spectrum (HRMS) of  $\pm(2c)$

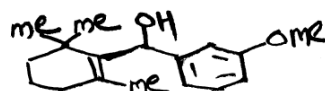




<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound **±(2d)**



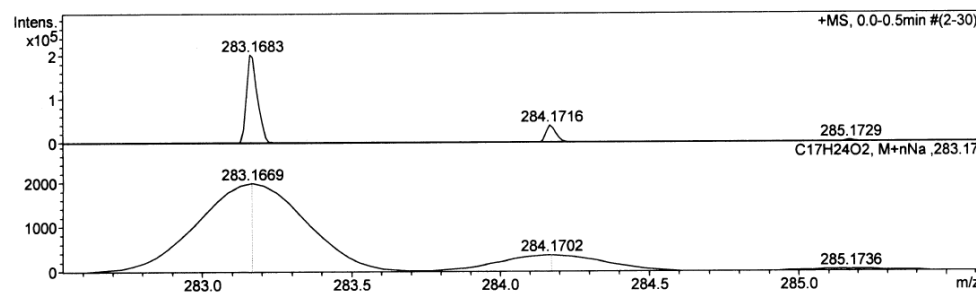
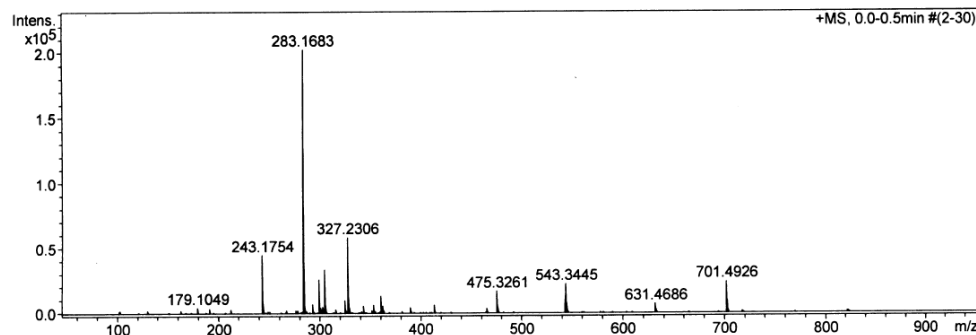
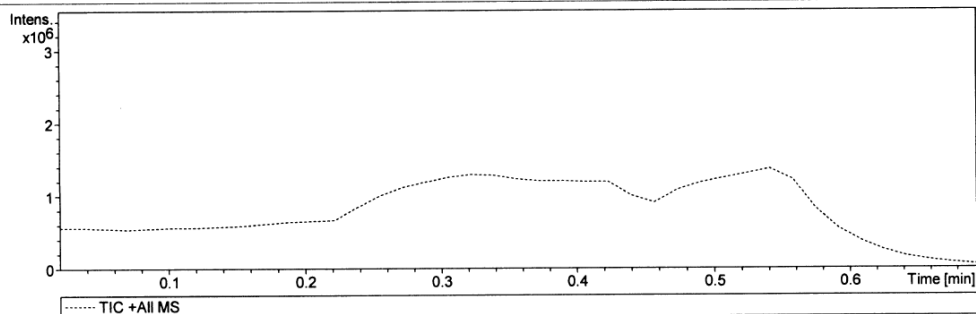
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{d})$



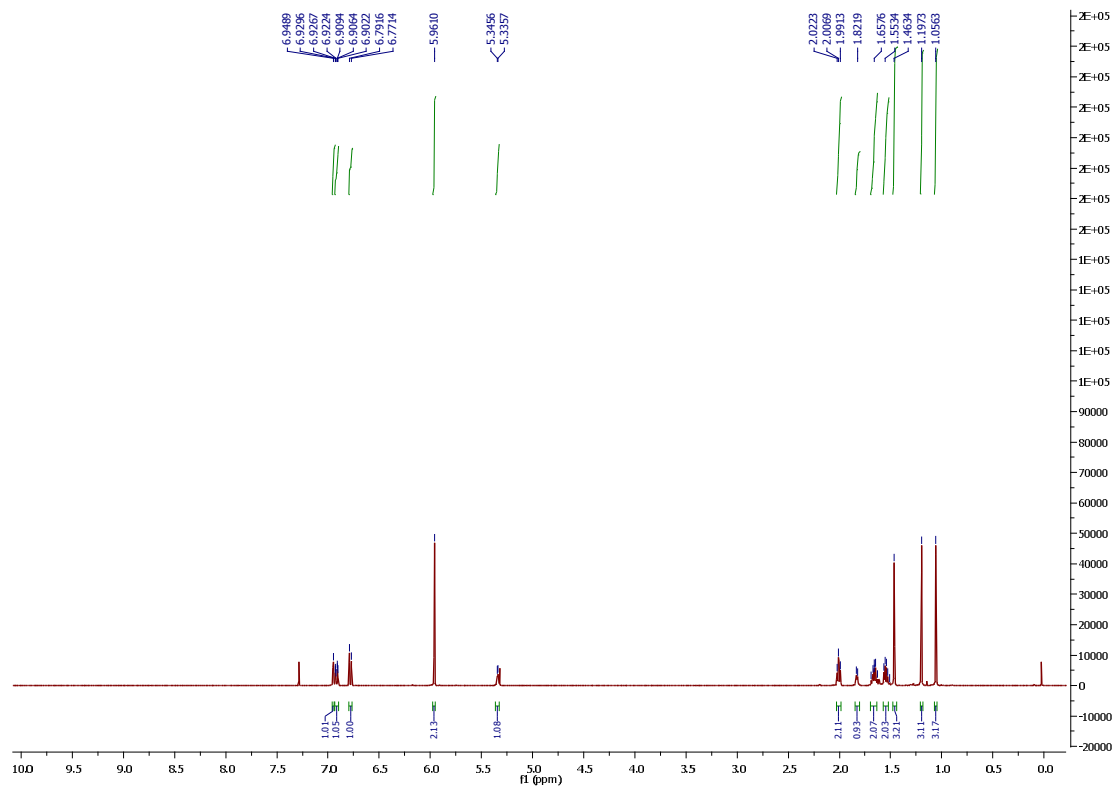
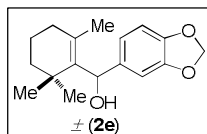
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Method	tune_low.m	Instrument	microTOF-Q II 10330
Sample Name	BK-3-167		
Comment			

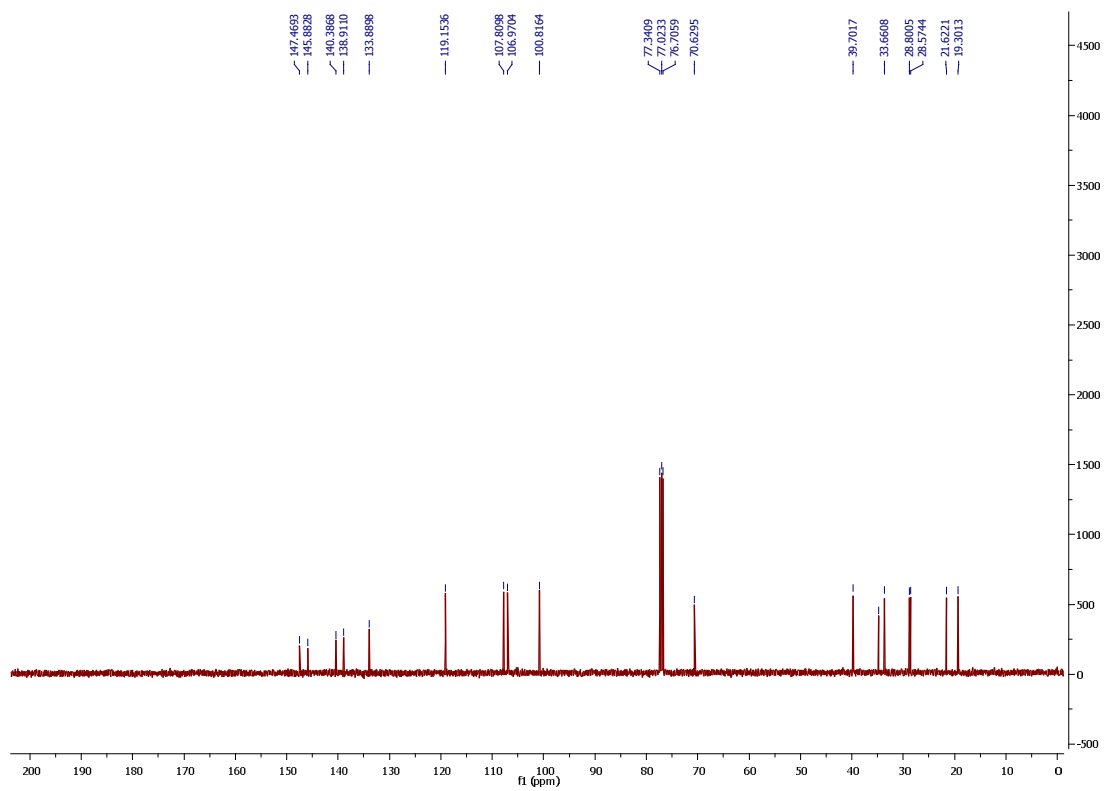
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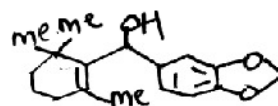
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$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2e)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{e})$



## Display Report

### Analysis Info

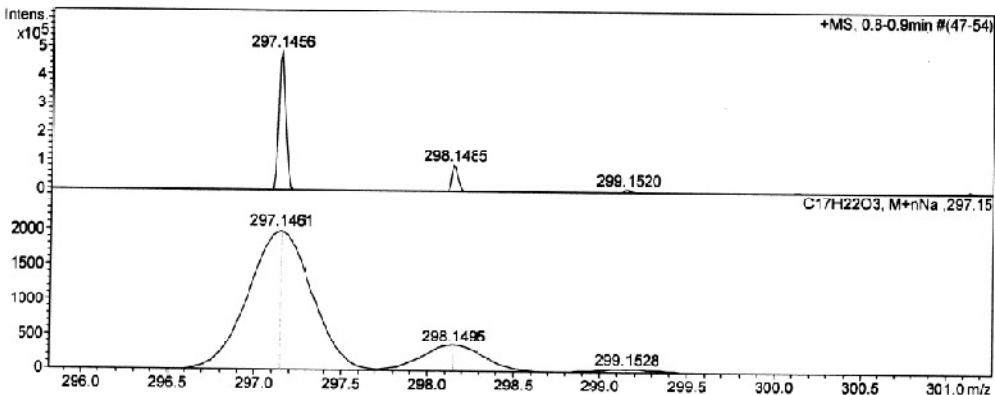
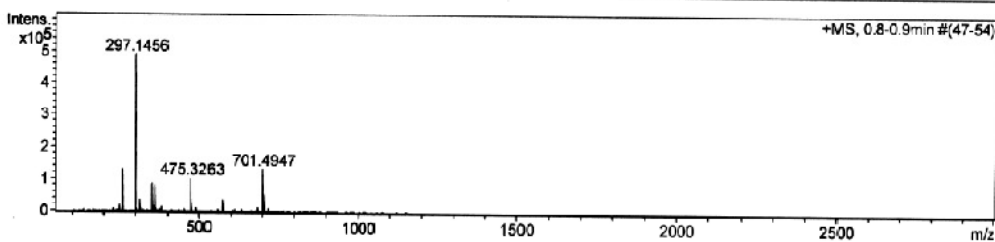
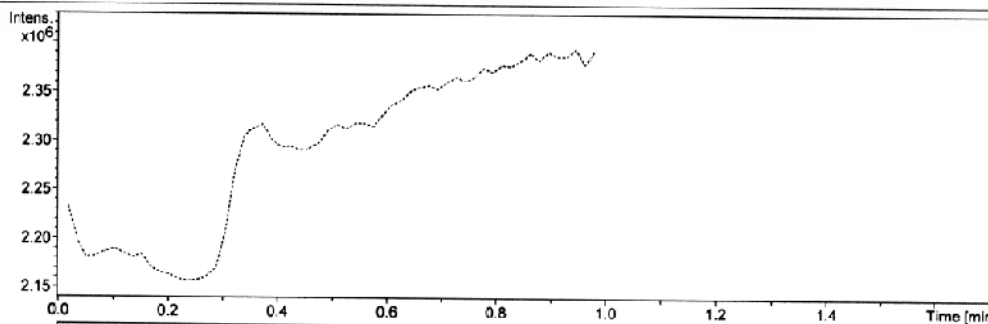
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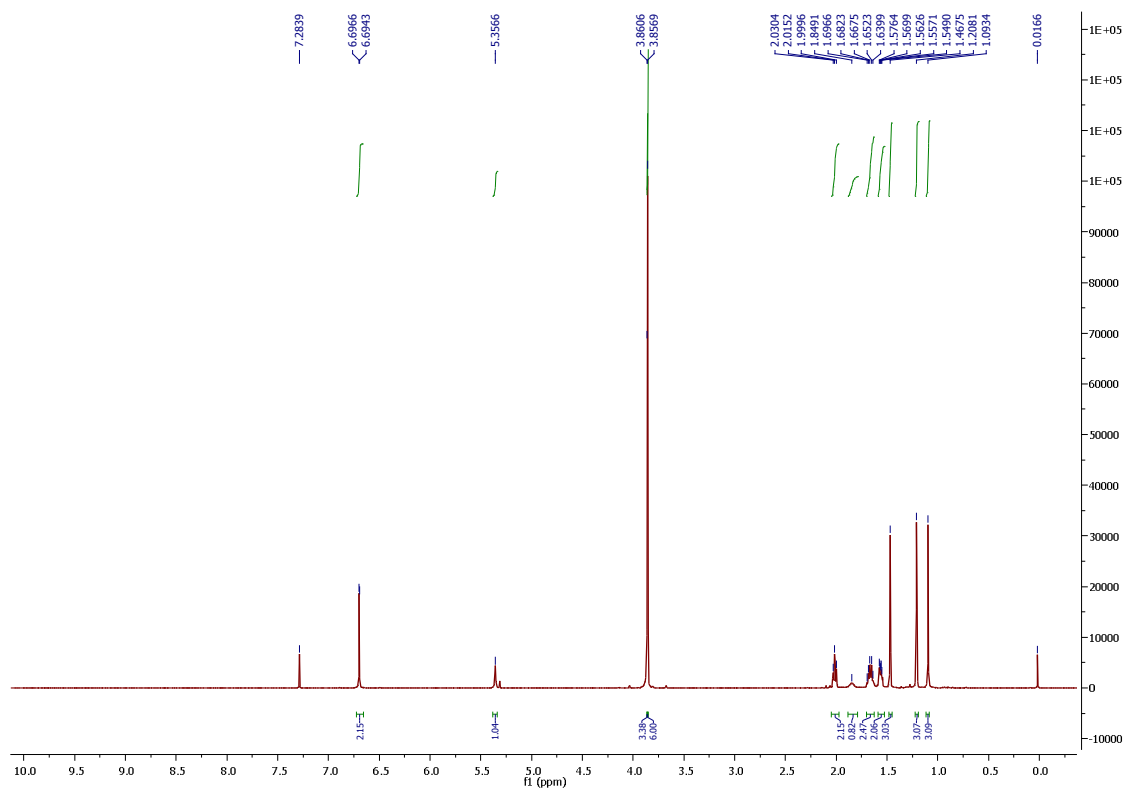
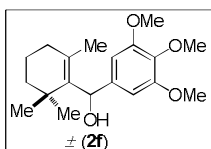
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

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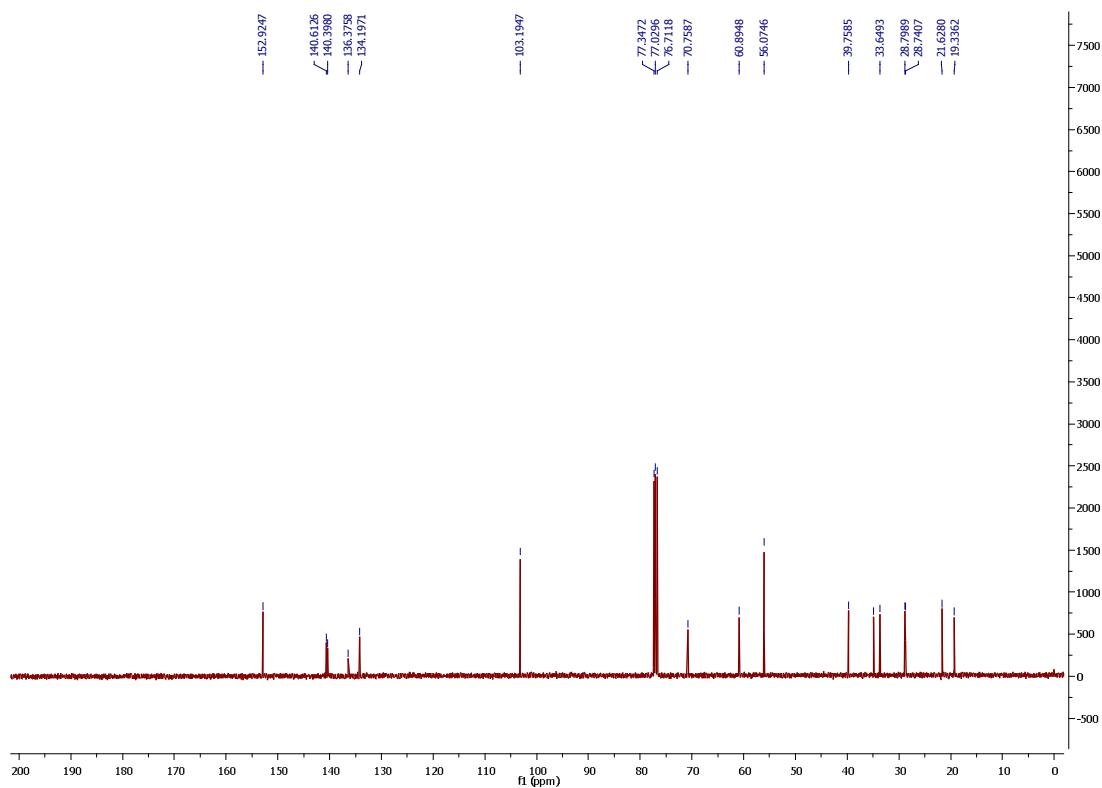
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Scanned copy of mass spectrum (HRMS) of  $\pm(2e)$

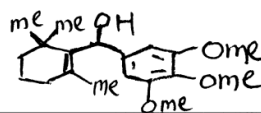


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2f)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{f})$





### Display Report

#### Analysis Info

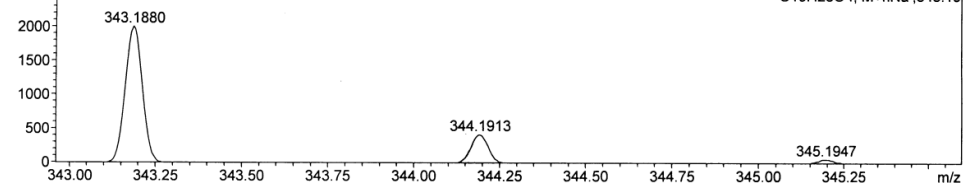
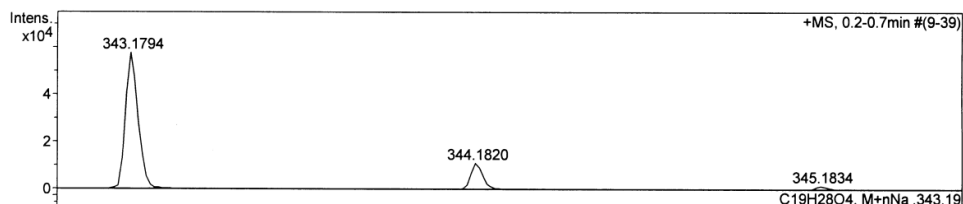
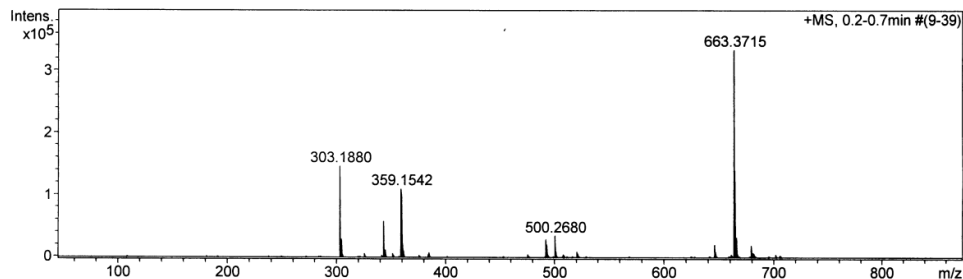
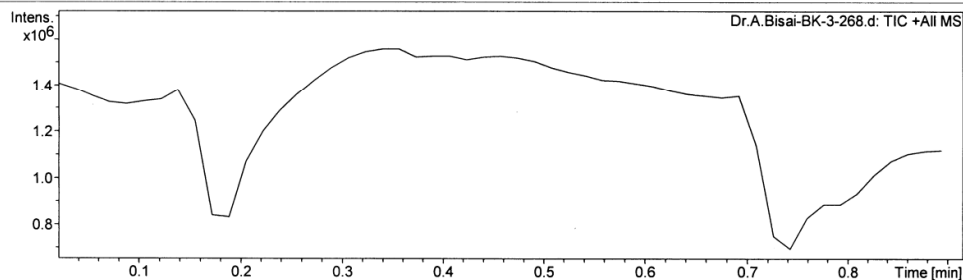
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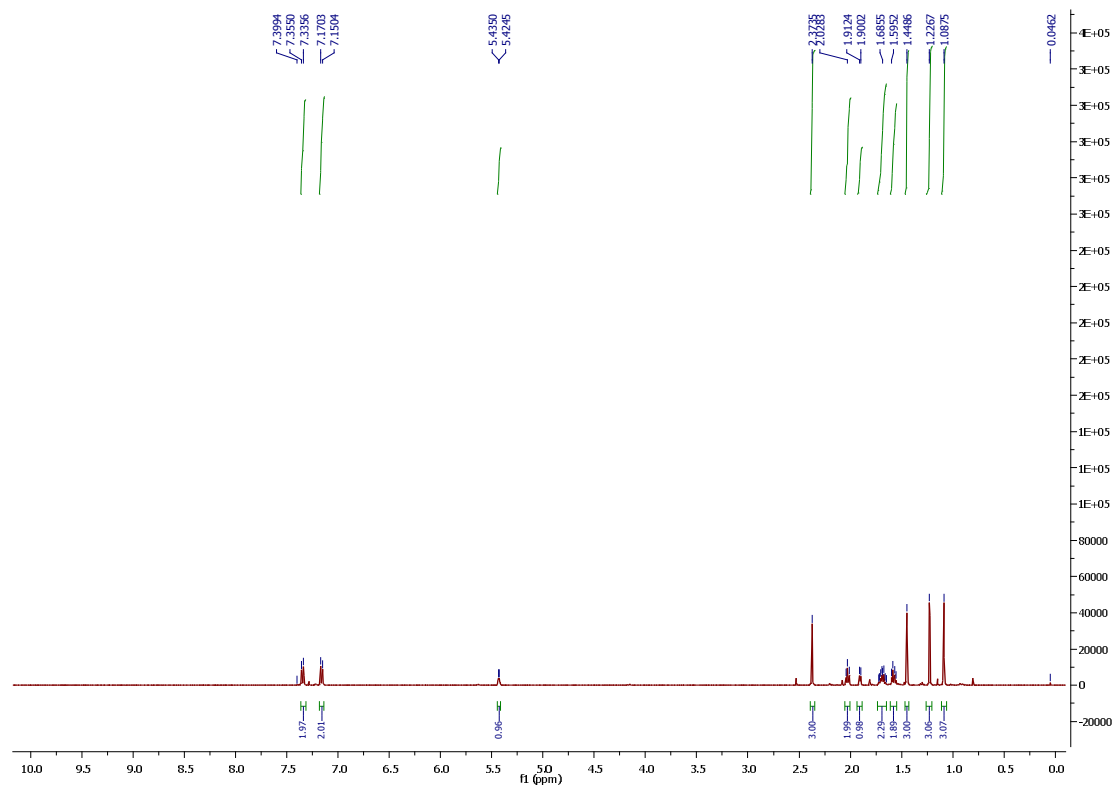
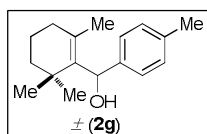
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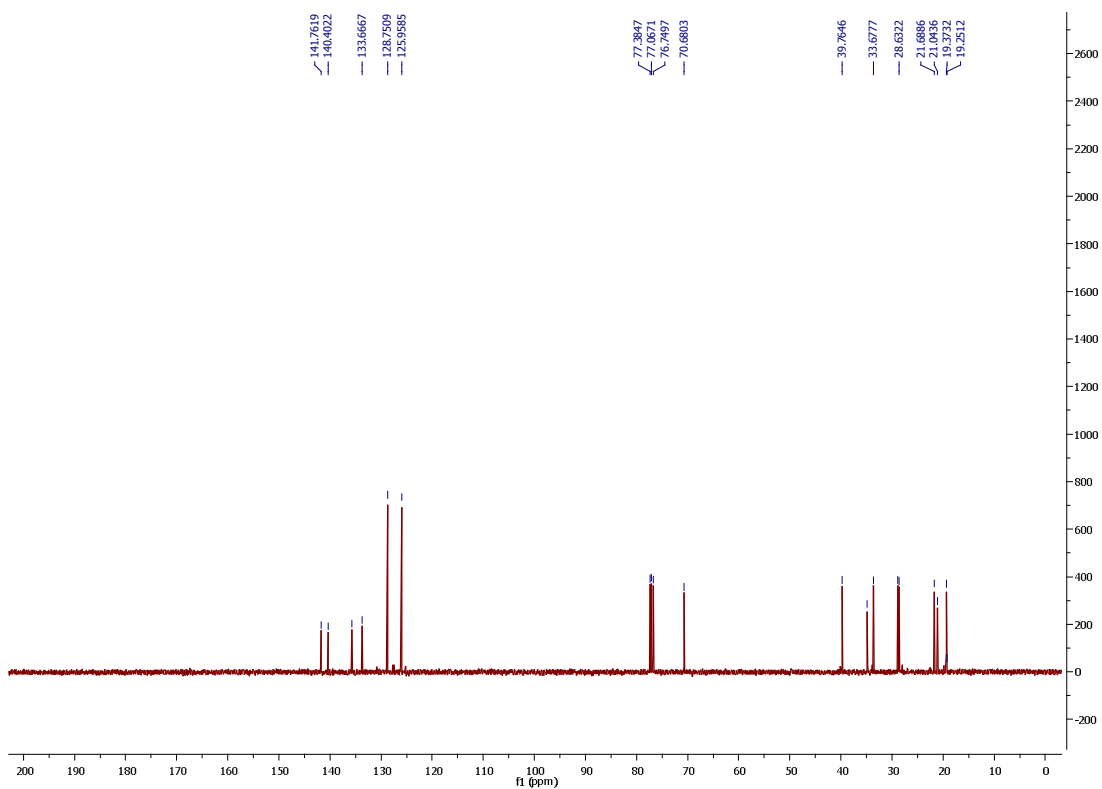
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



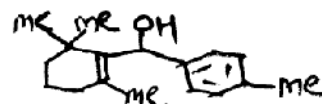
Scanned copy of mass spectrum (HRMS) of ±(2f)



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2g)$



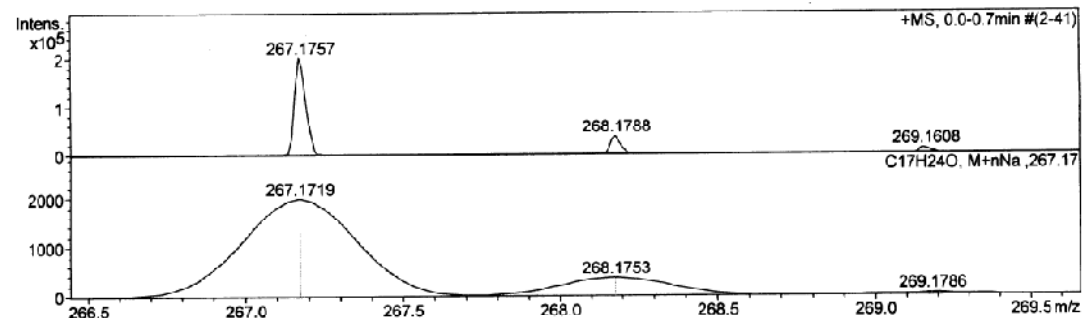
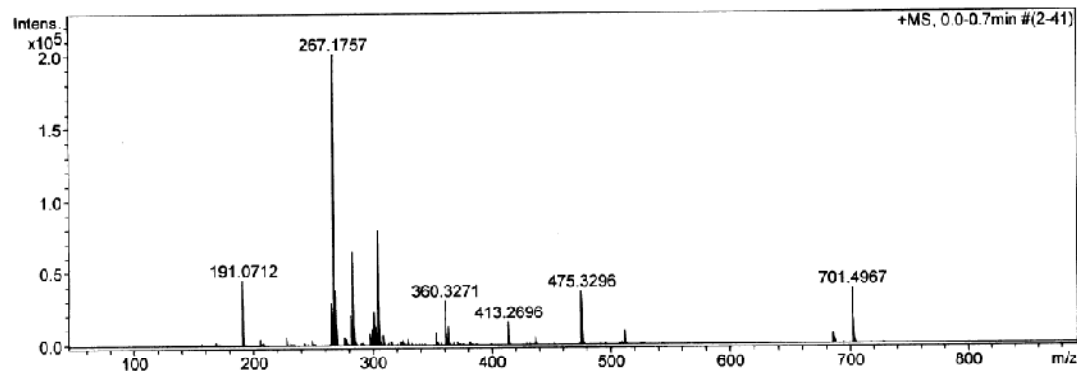
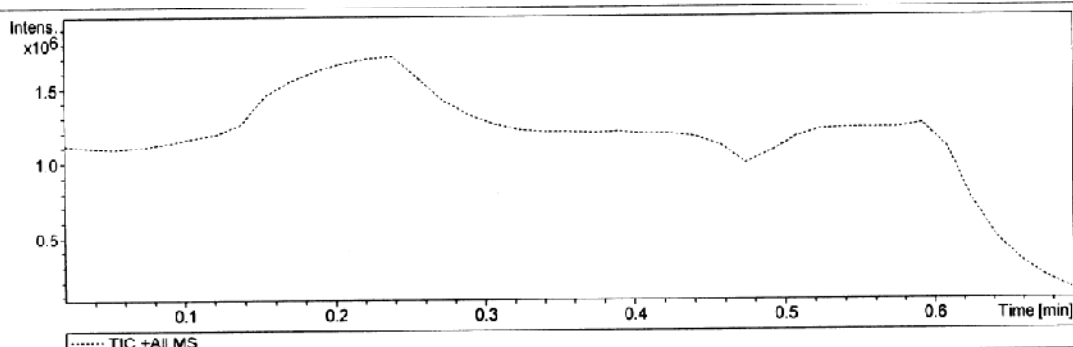
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\text{g})$

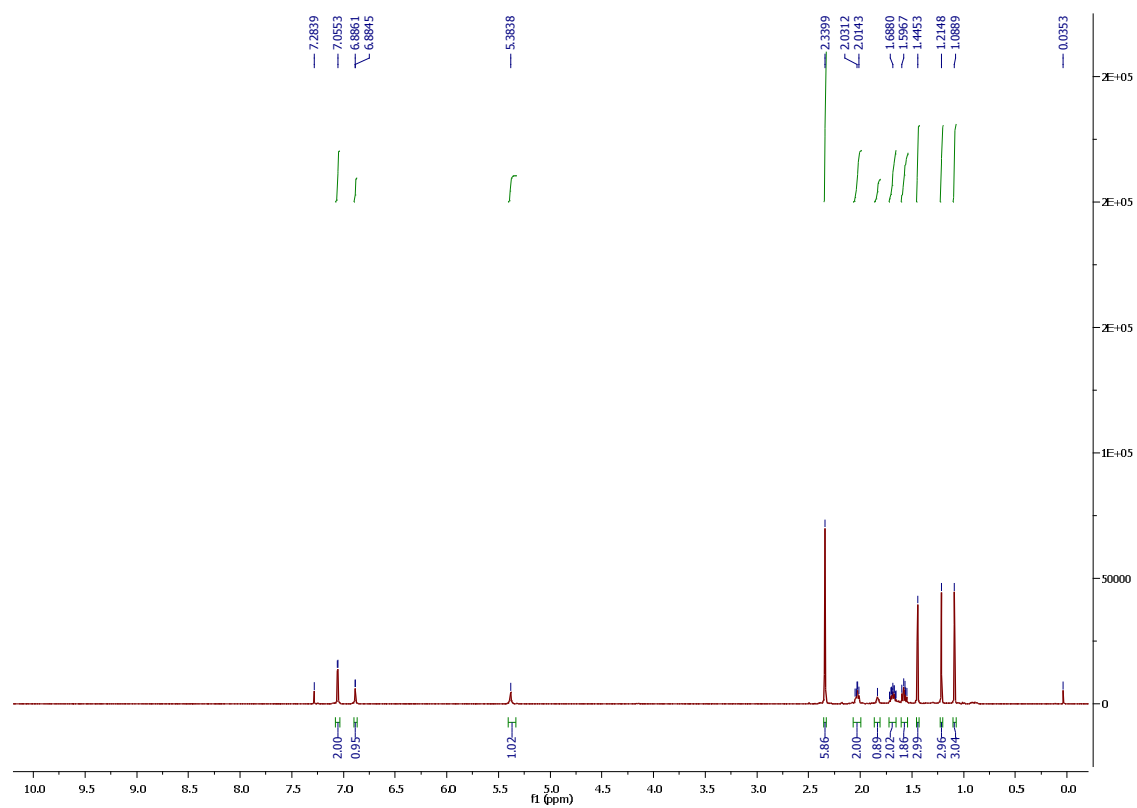
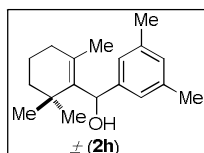


### Display Report

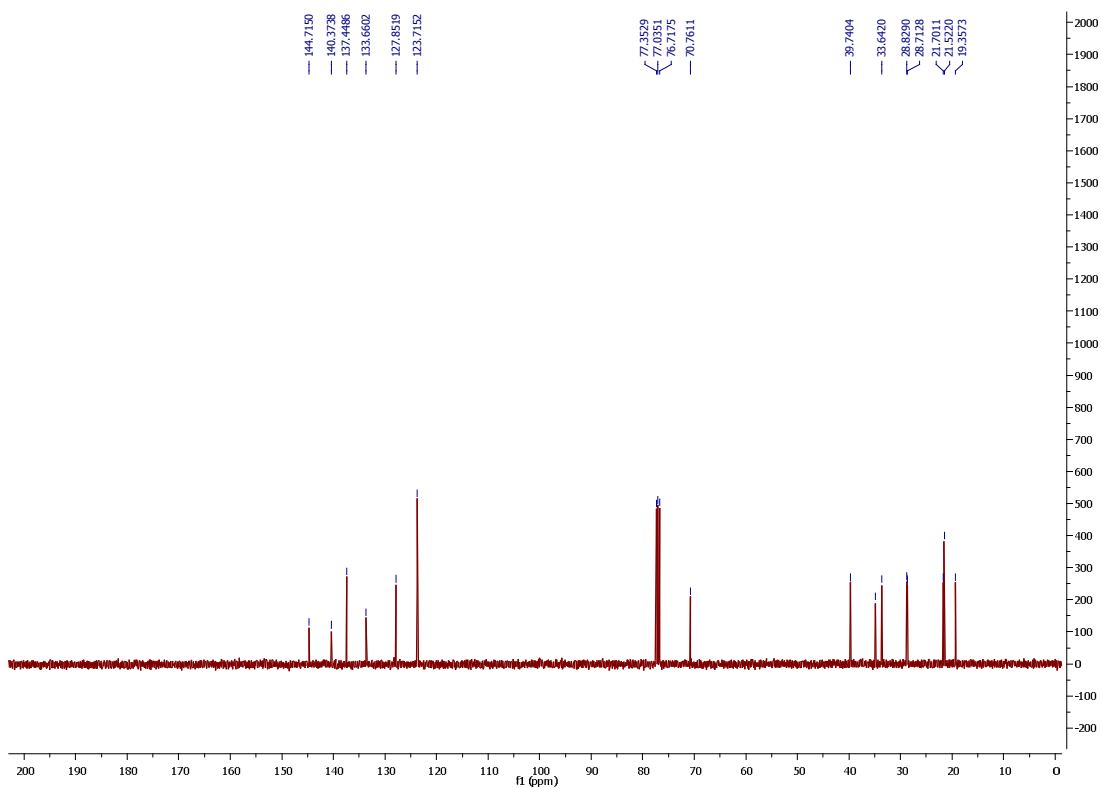
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Analysis Name	D:\Data\user data\2013\JAN\18 JAN\Dr.A.Bisai-BK-2-233.d	Operator	IISERB
Method	tune_low.m	Instrument	micrOTOF-Q II 10330
Sample Name	BK-2-233		
Comment			

<b>Acquisition Parameter</b>					
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	130.0 Vpp	Set Divert Valve	Waste

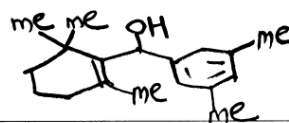




$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2h)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{h})$



### Display Report

#### Analysis Info

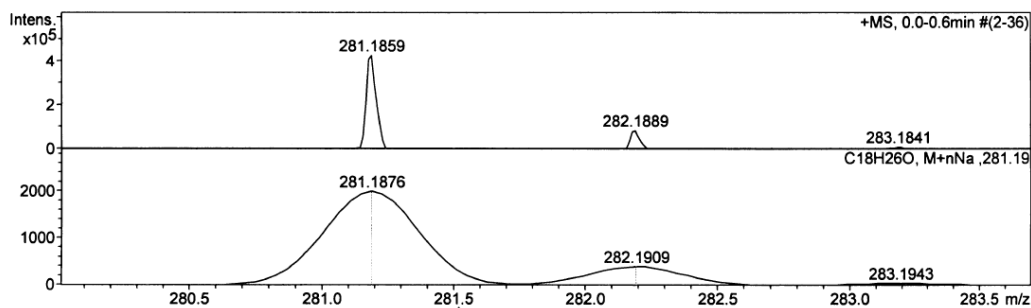
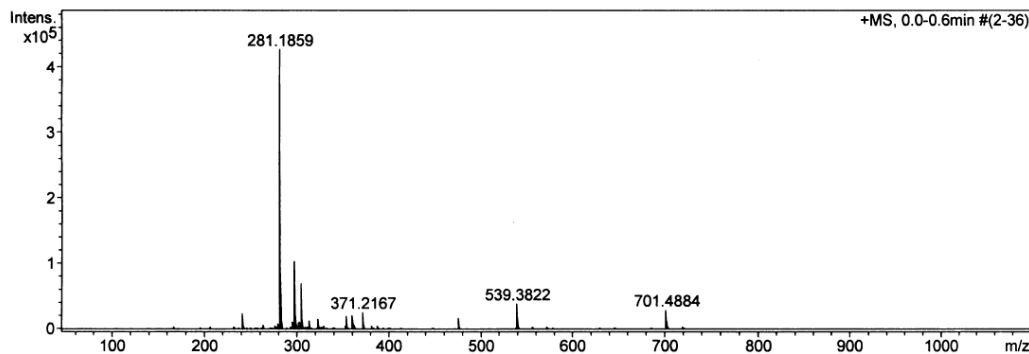
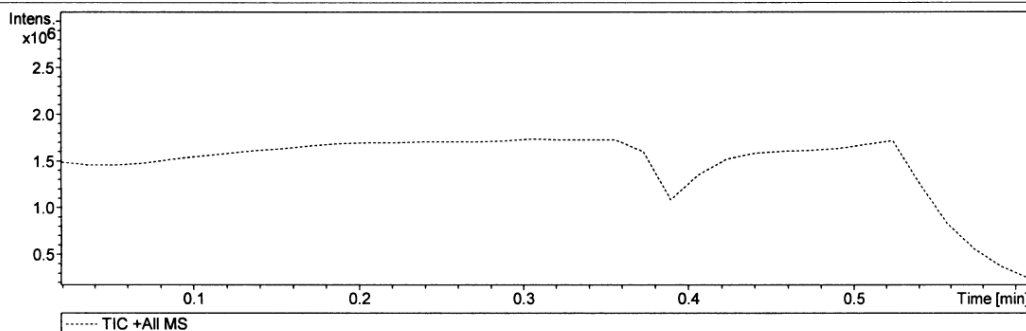
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Sample Name BK-2-249  
Comment

Acquisition Date 1/18/2013 12:24:39 PM

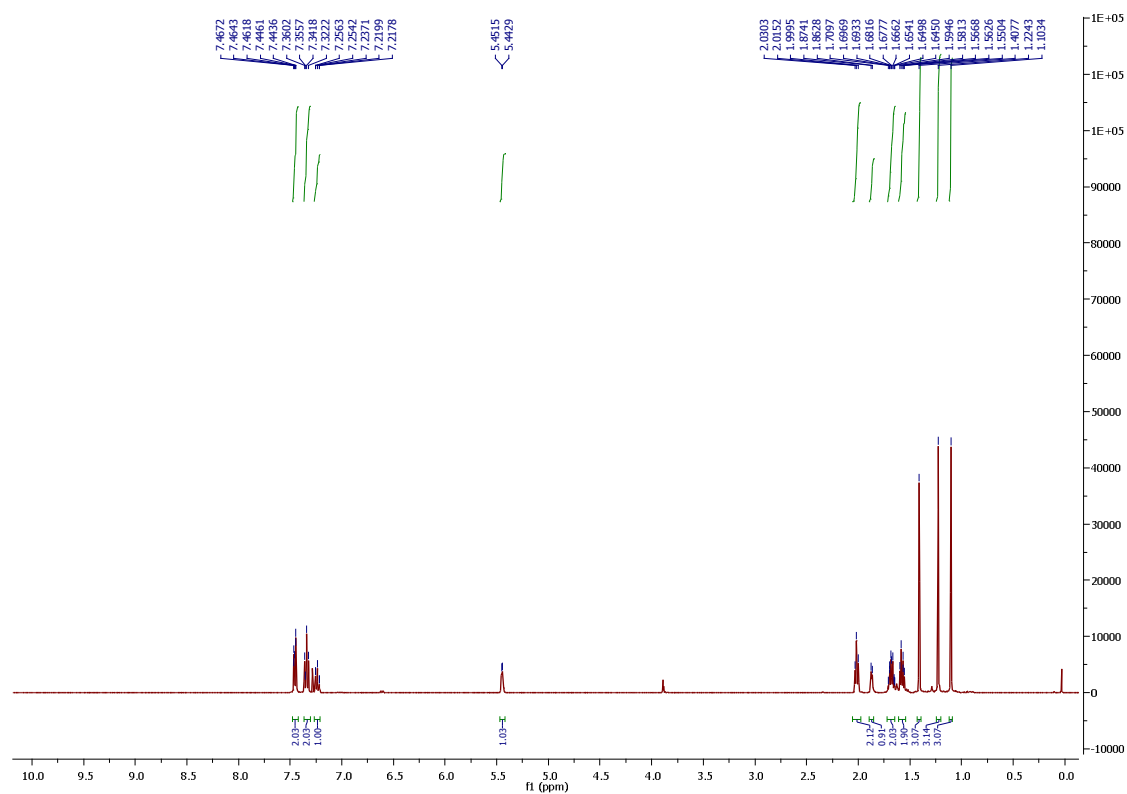
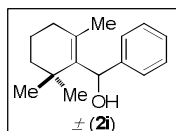
Operator IISERB  
Instrument micrOTOF-Q II 10330

#### 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	130.0 Vpp	Set Divert Valve	Waste

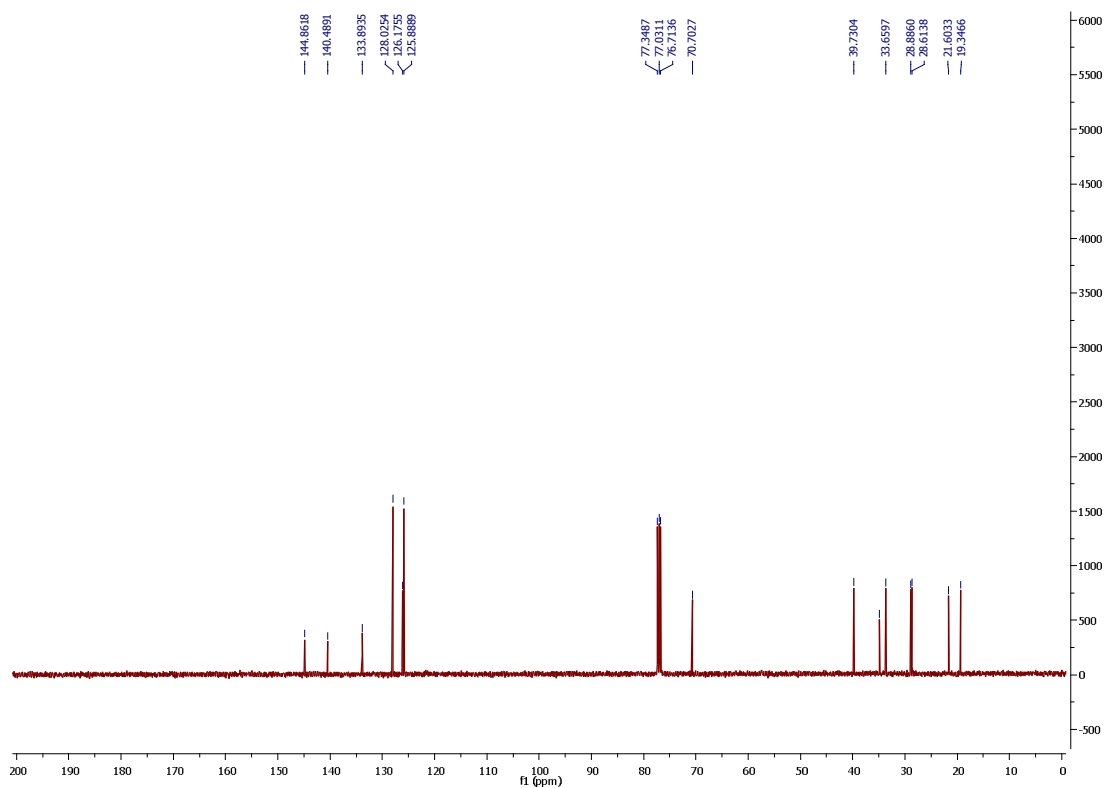


Scanned copy of mass spectrum (HRMS) of  $\pm(2h)$

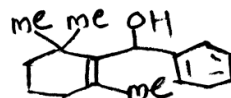


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2i)$





$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2i)$



## Display Report

### Analysis Info

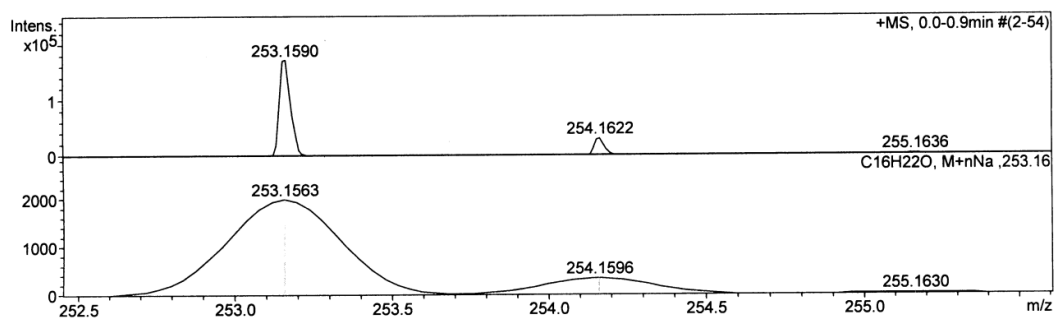
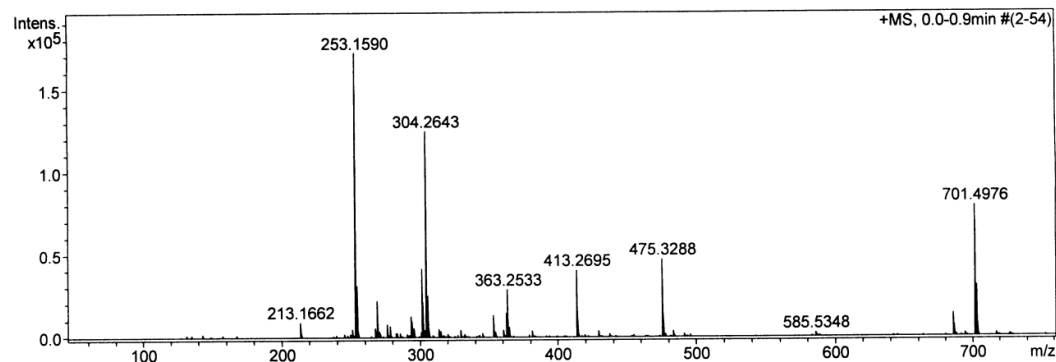
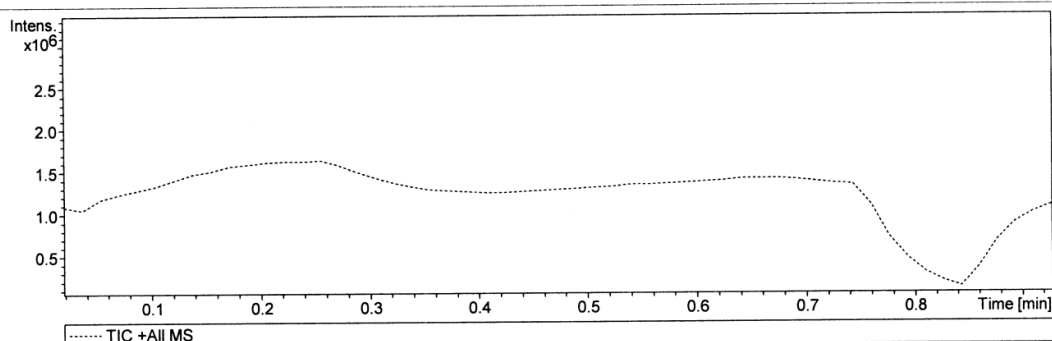
Analysis Name D:\Data\user data\2013\JAN\15 JAN\Dr.A.Bisai-BK-2-219-1.d  
Method tune\_low.m  
Sample Name BK-2-219-1  
Comment

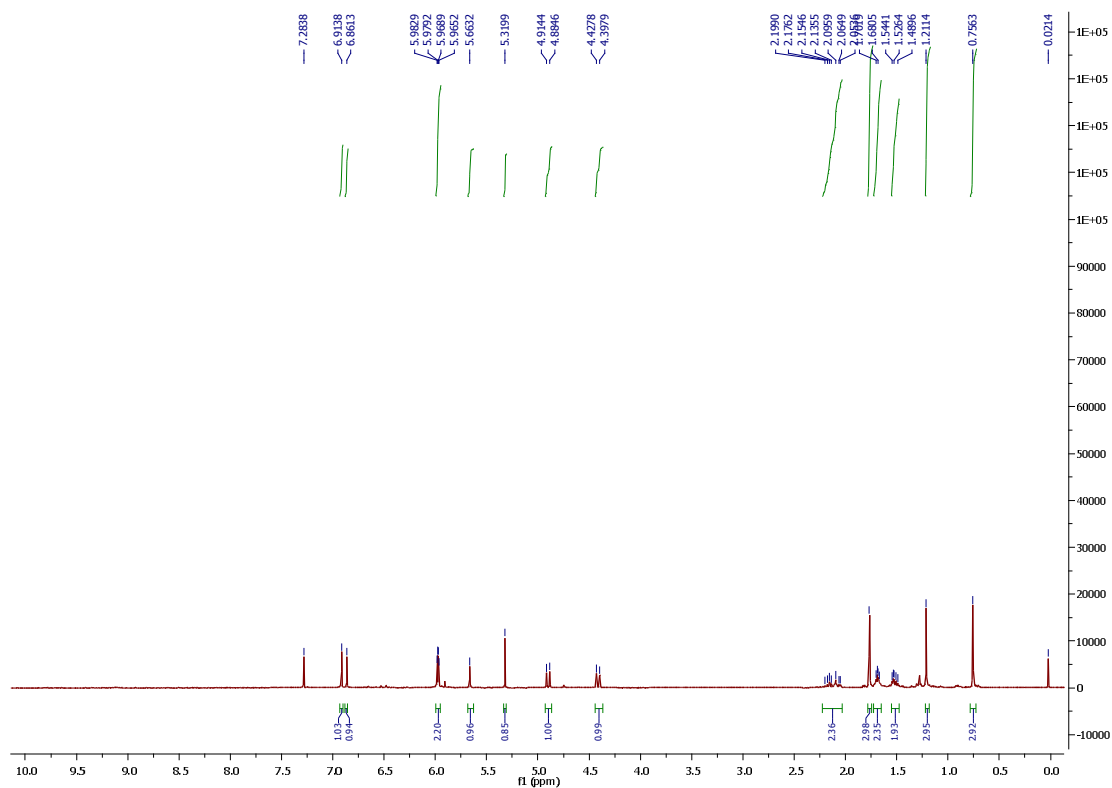
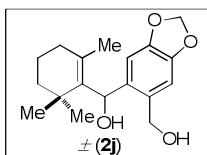
Acquisition Date 1/16/2013 11:17:31 AM

Operator IISERB  
Instrument micrOTOF-Q II 10330

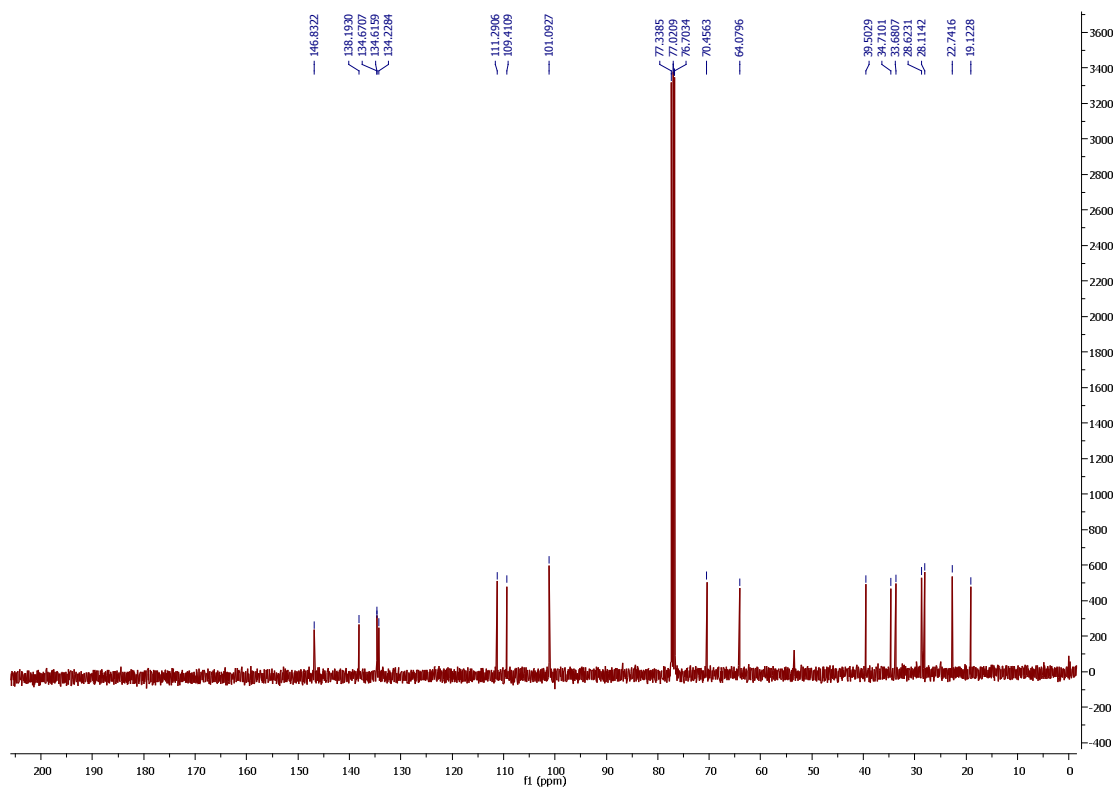
### Acquisition Parameter

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	130.0 Vpp	Set Divert Valve	Waste

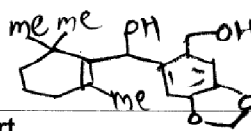




<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound  $\pm(2j)$



<sup>13</sup>C NMR (400 MHz, CDCl<sub>3</sub>) compound **±(2j)**



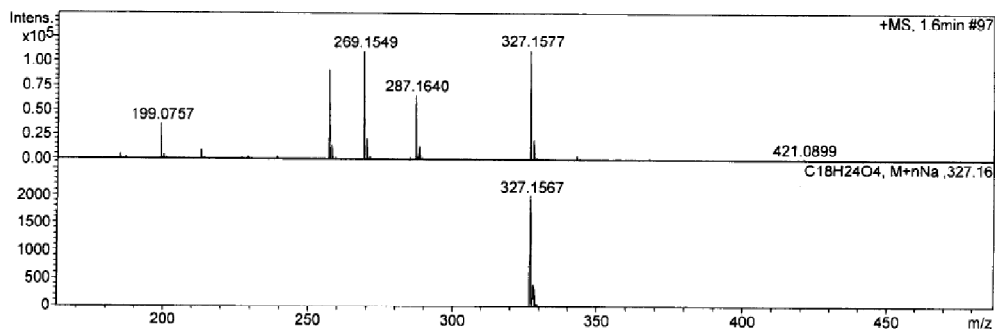
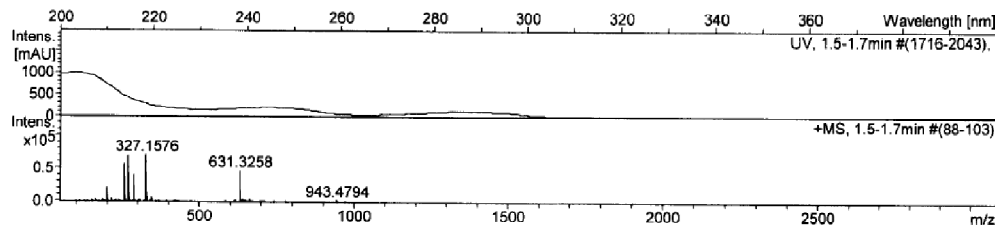
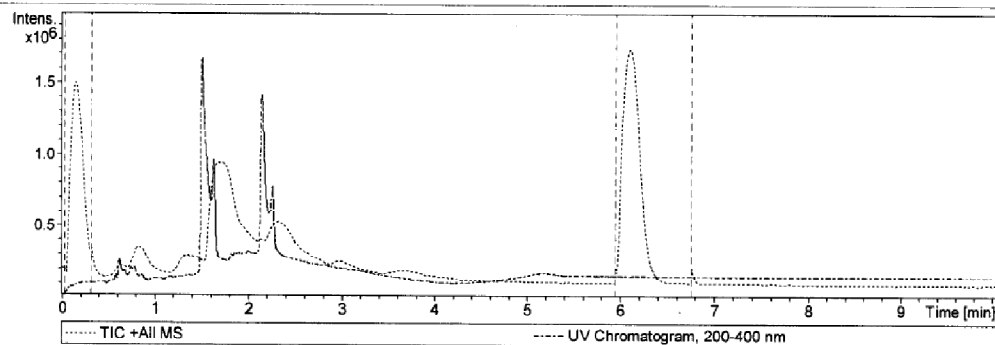
## Display Report

### Analysis Info

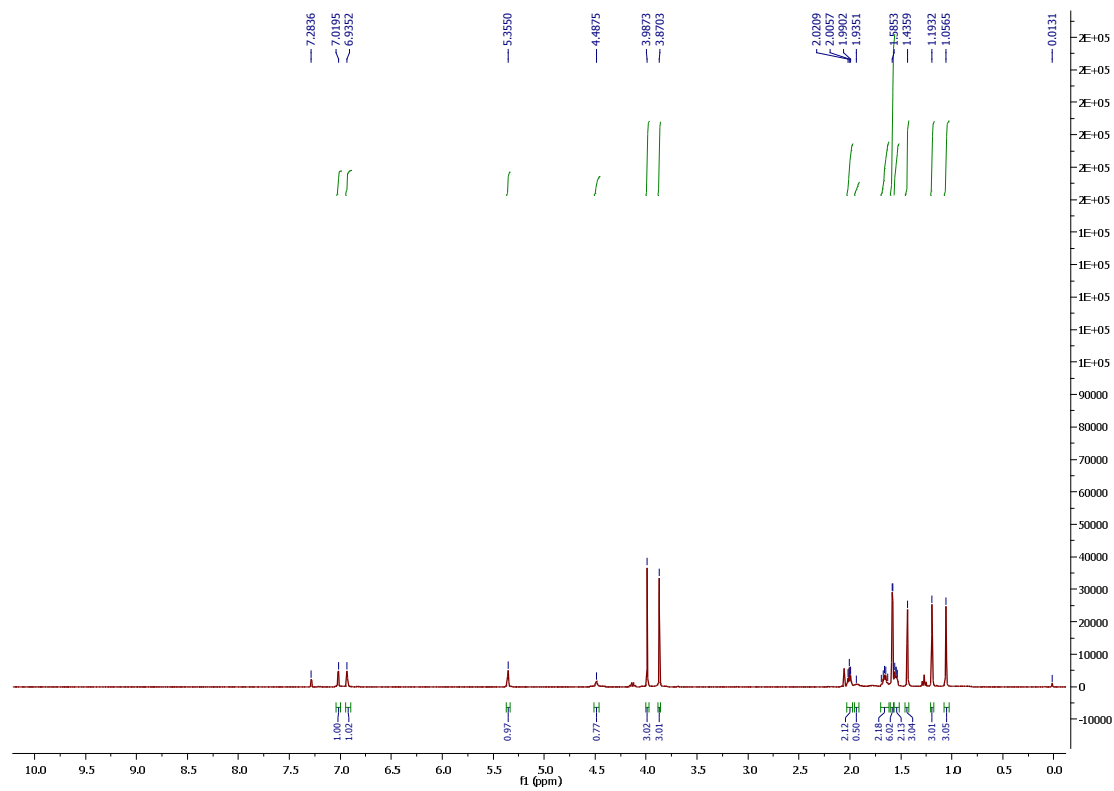
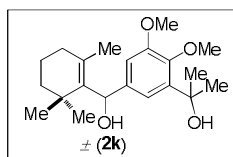
Analysis Name	D:\Data\user data\may2012\19 may\Dr. A. Bisai- BK-2-266_1-A,3_01_2159.d	Acquisition Date	5/9/2012 3:33:27 PM
Method	HRLCMS-20 Sept.m	Operator	Meena Sharma
Sample Name	Dr. A. Bisai- BK-2-266	Instrument	micrOTOF-Q II 10330
Comment			

### Acquisition Parameter

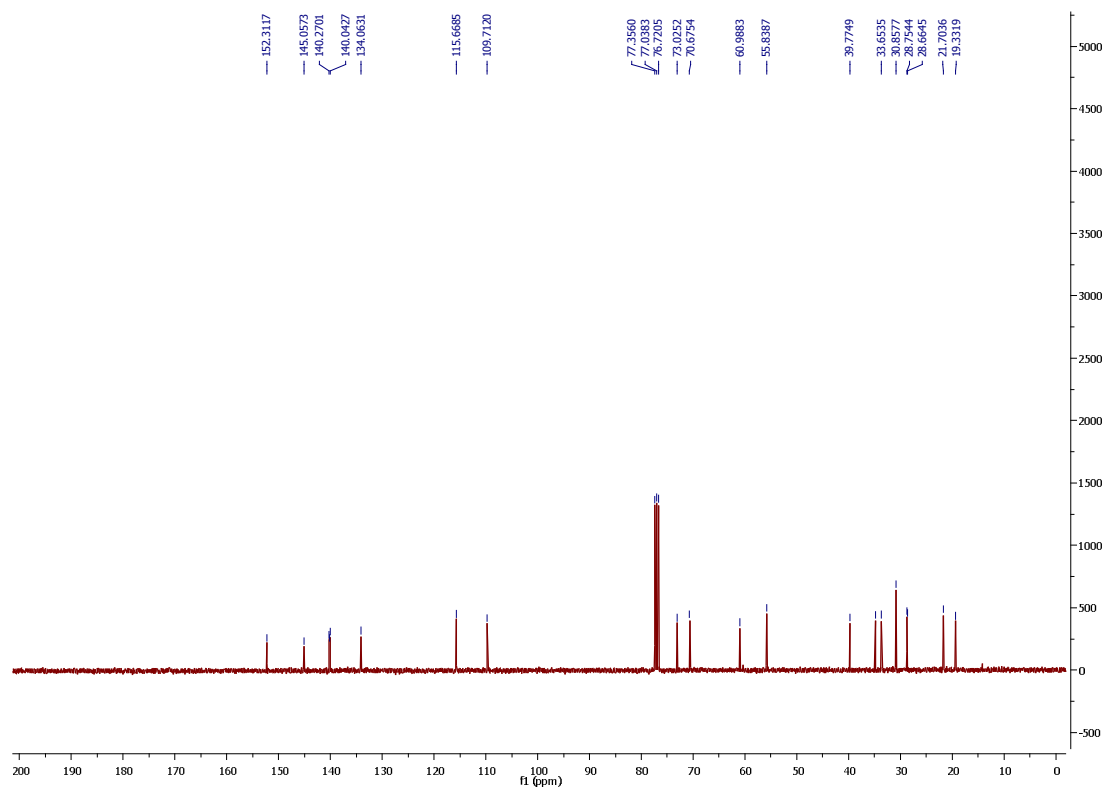
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



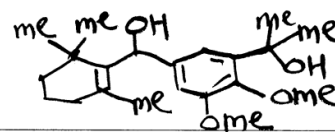
Scanned copy of mass spectrum (HRMS) of  $\pm(2j)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2k)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(2\mathbf{k})$



## Display Report

### Analysis Info

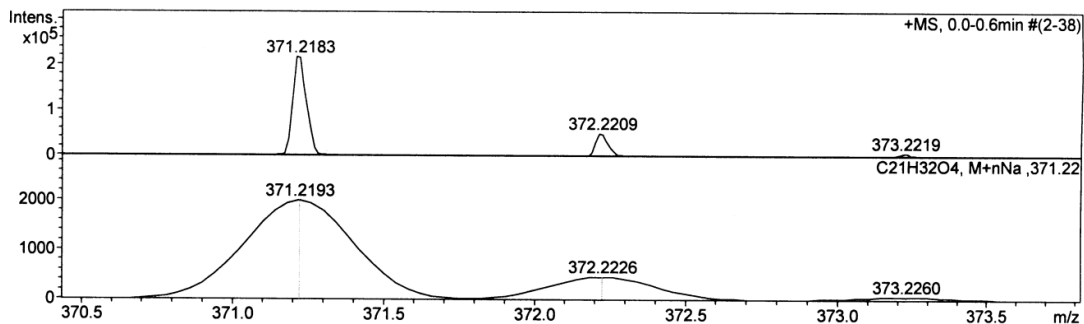
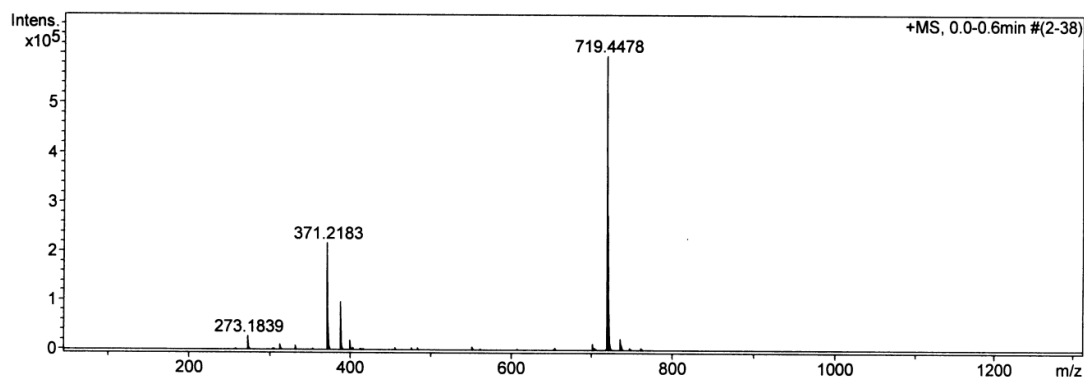
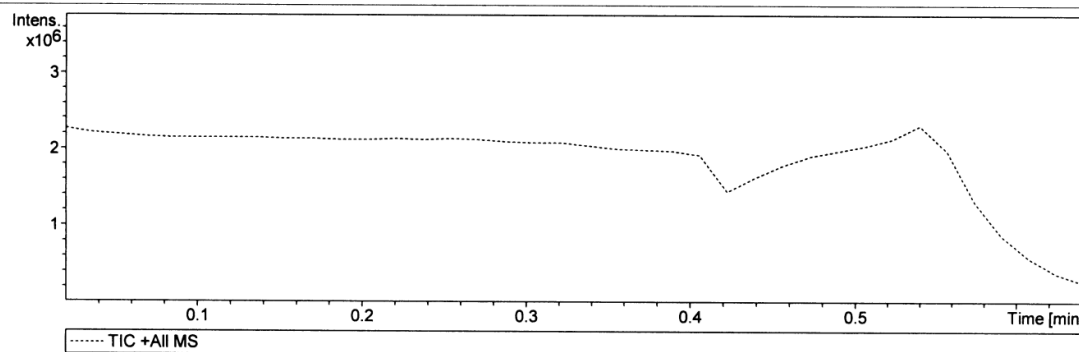
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Method tune\_low.m  
Sample Name BK-2-334  
Comment

Acquisition Date 1/18/2013 12:16:53 PM

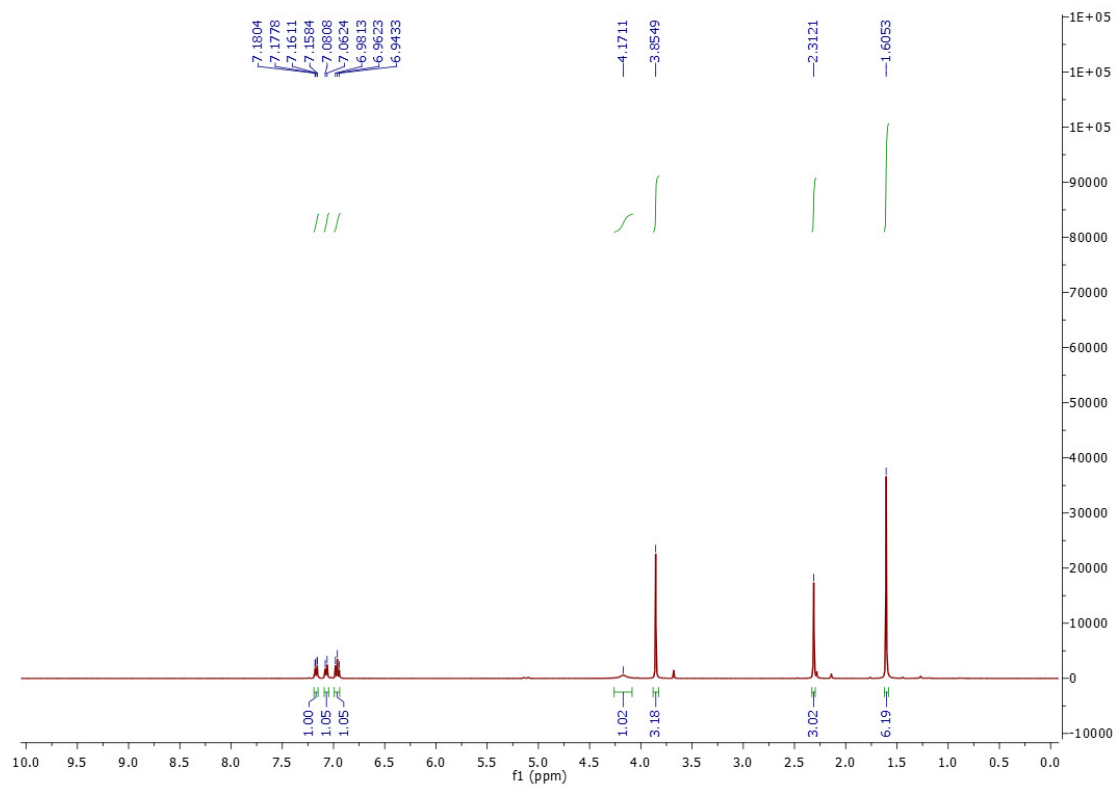
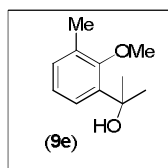
Operator IISERB  
Instrument micrOTOF-Q II 10330

### Acquisition Parameter

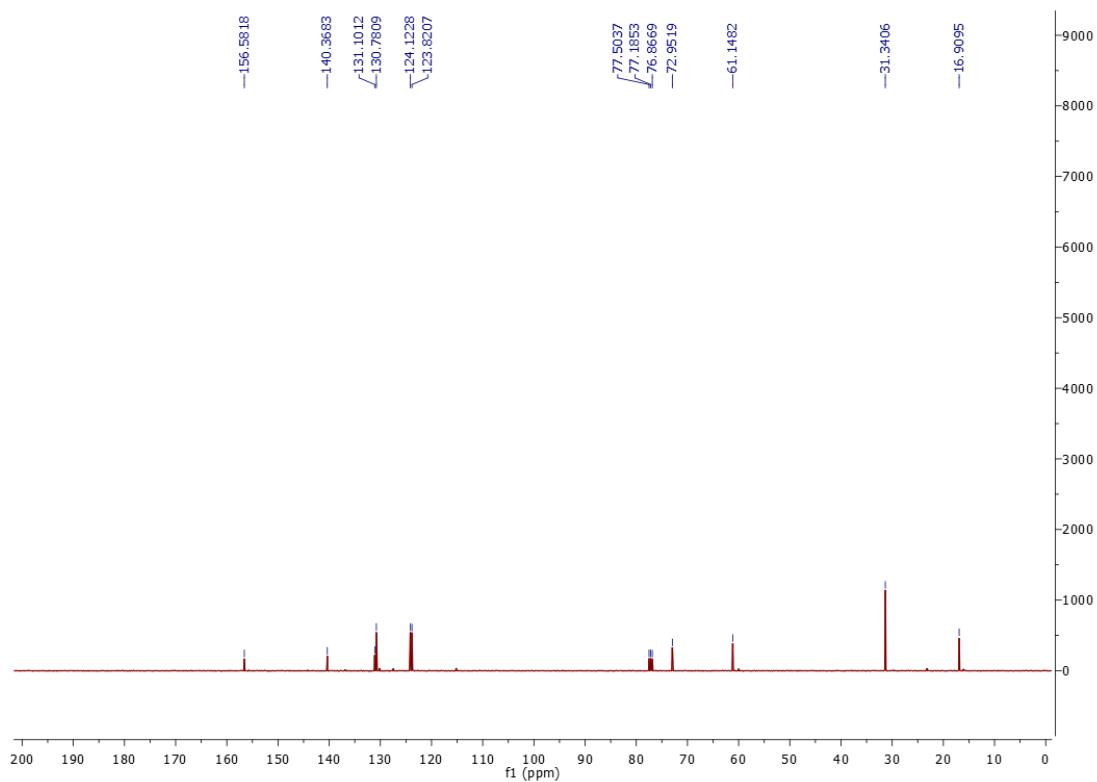
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Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



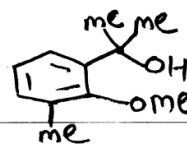




$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound **(9e)**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound **9e**



### Display Report

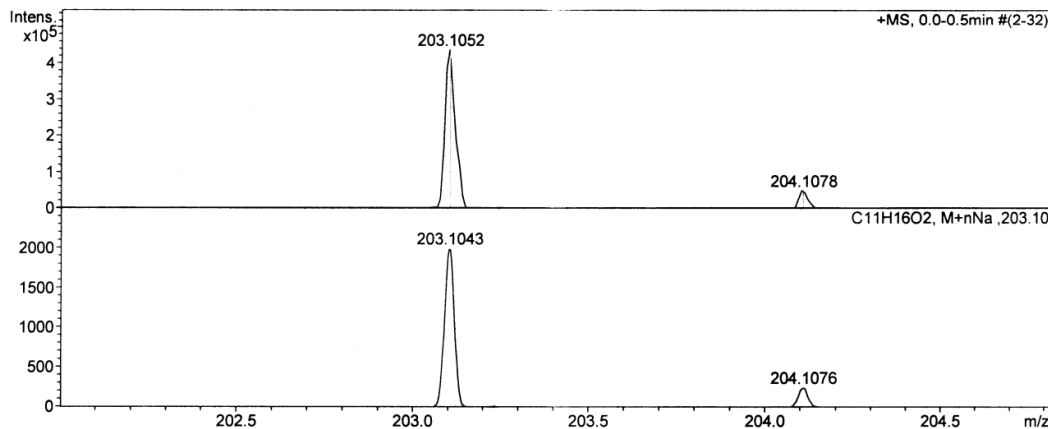
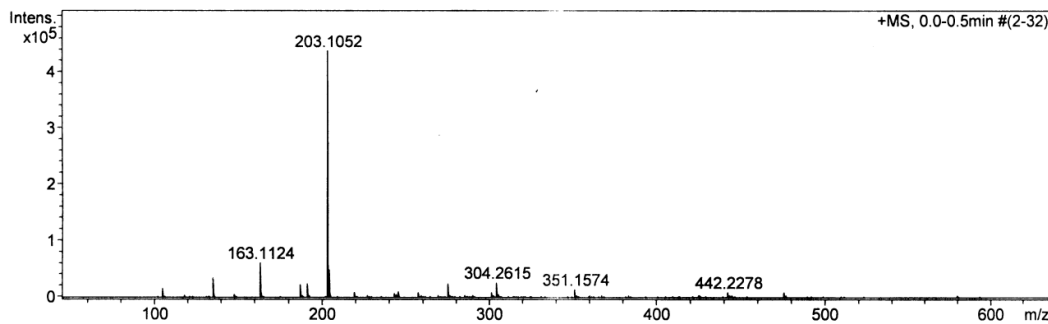
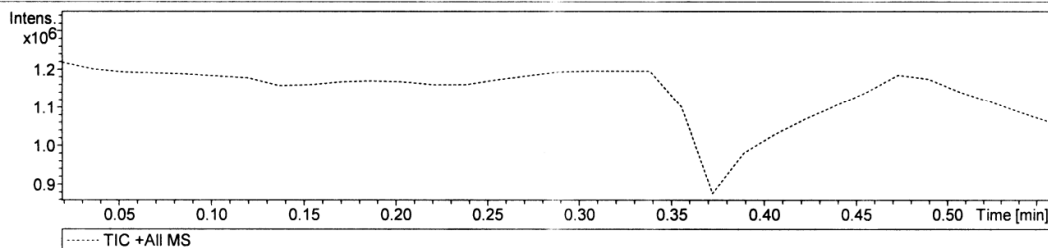
#### Analysis Info

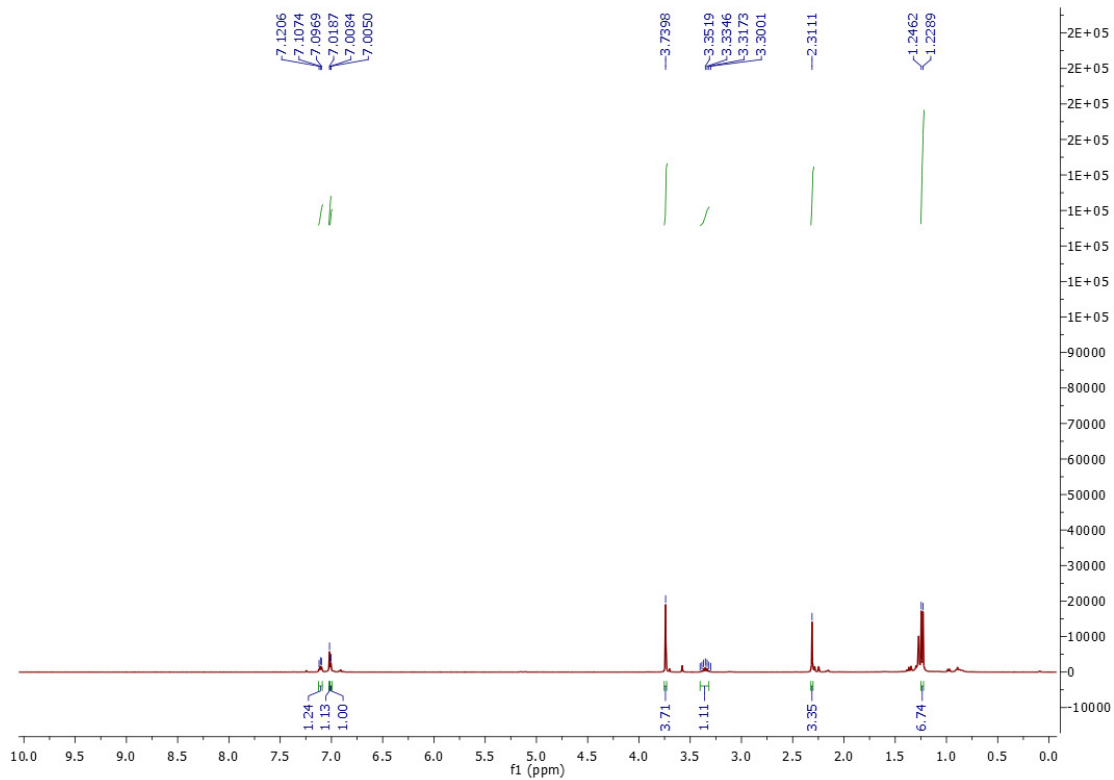
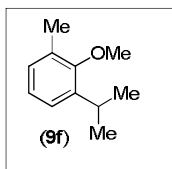
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Method tune\_low.m  
Sample Name BK-3-202  
Comment

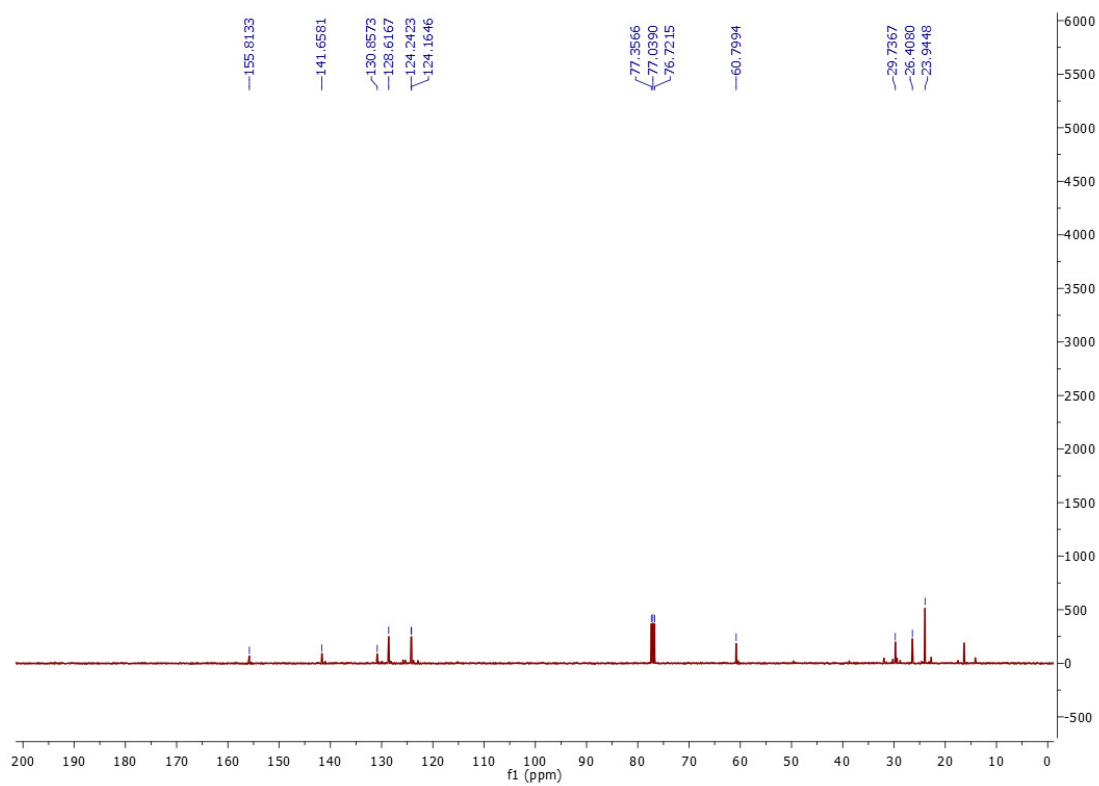
Acquisition Date 2/15/2013 10:39:00 AM  
Operator Amit  
Instrument micrOTOF-Q II 10330

#### Acquisition Parameter

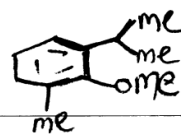
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound **9f**



## Display Report

### Analysis Info

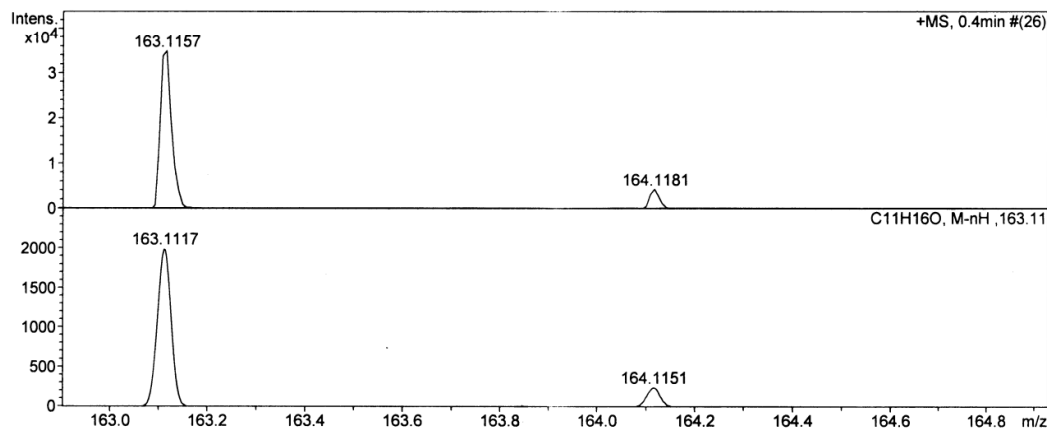
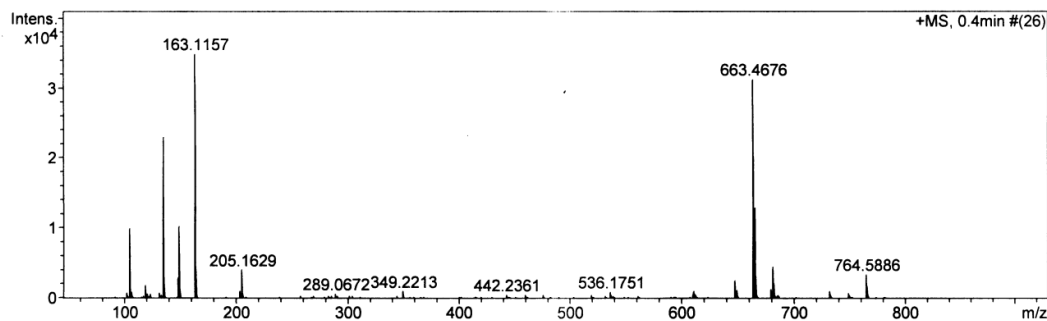
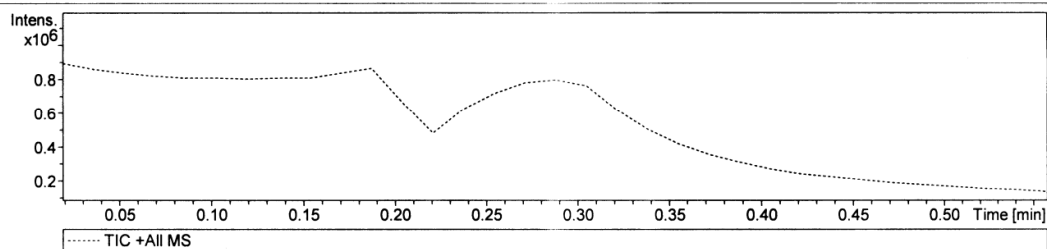
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Method tune\_low.m  
Sample Name BK-3-203  
Comment

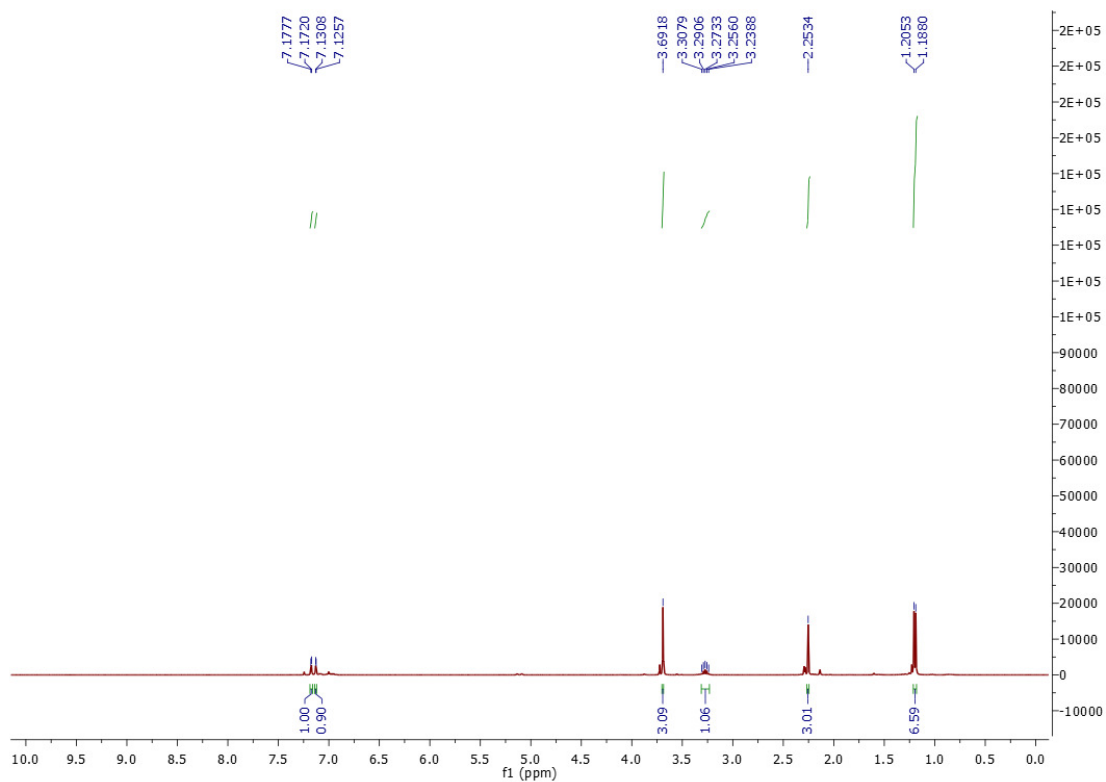
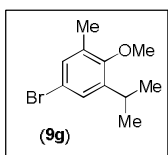
Acquisition Date 2/15/2013 10:53:30 AM

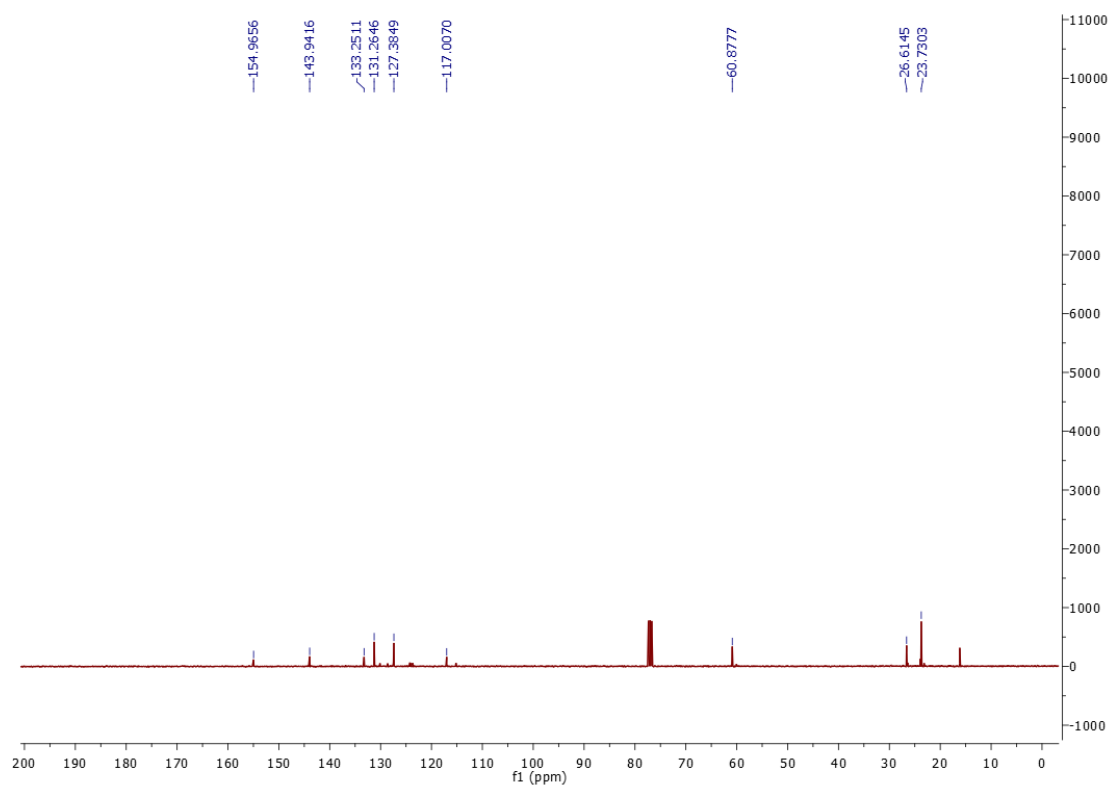
Operator Amit  
Instrument micrOTOF-Q II 10330

### 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	130.0 Vpp	Set Divert Valve	Waste

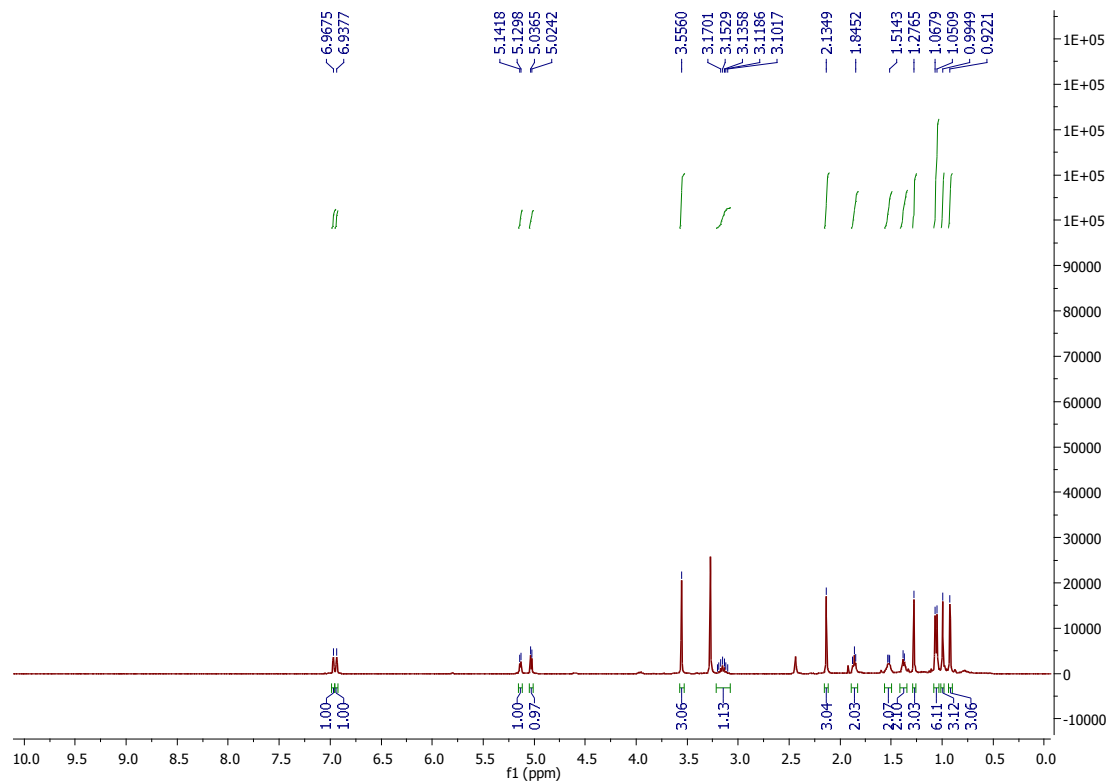
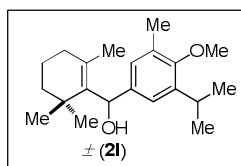




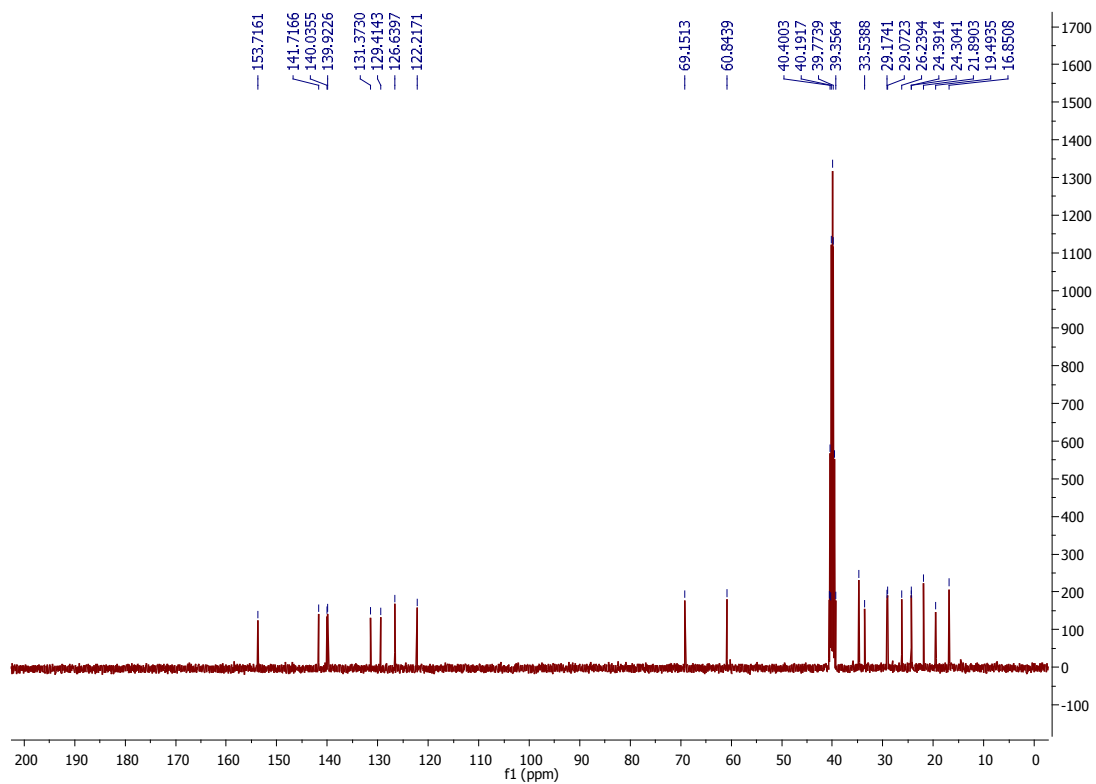


$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound **9g**

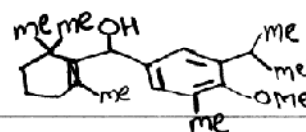




<sup>1</sup>H NMR (400 MHz, DMSO-D<sub>6</sub>) compound **21**



<sup>13</sup>C NMR (100 MHz, DMSO-D<sub>6</sub>) compound **21**

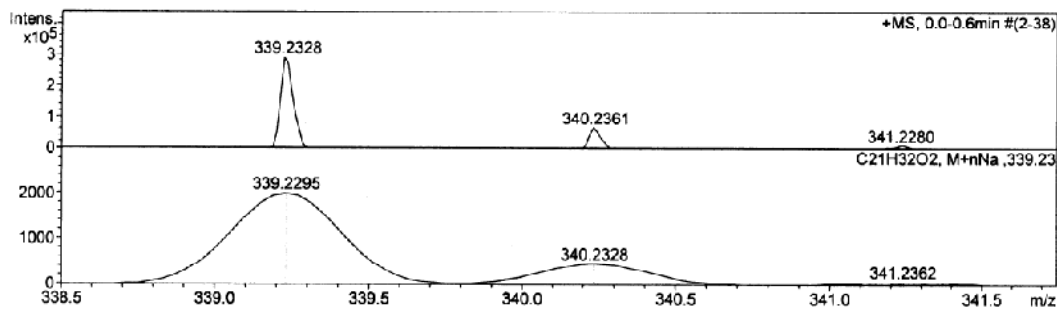
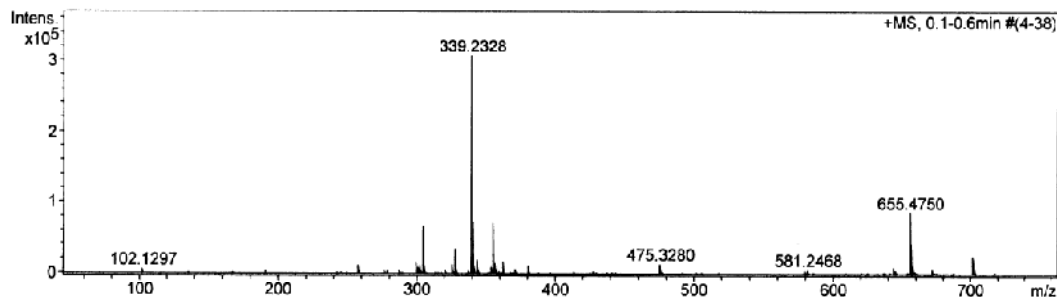
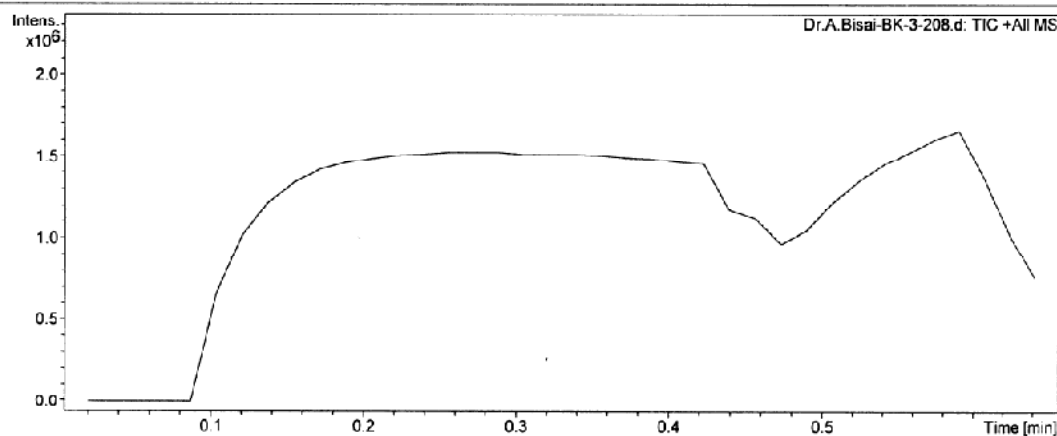


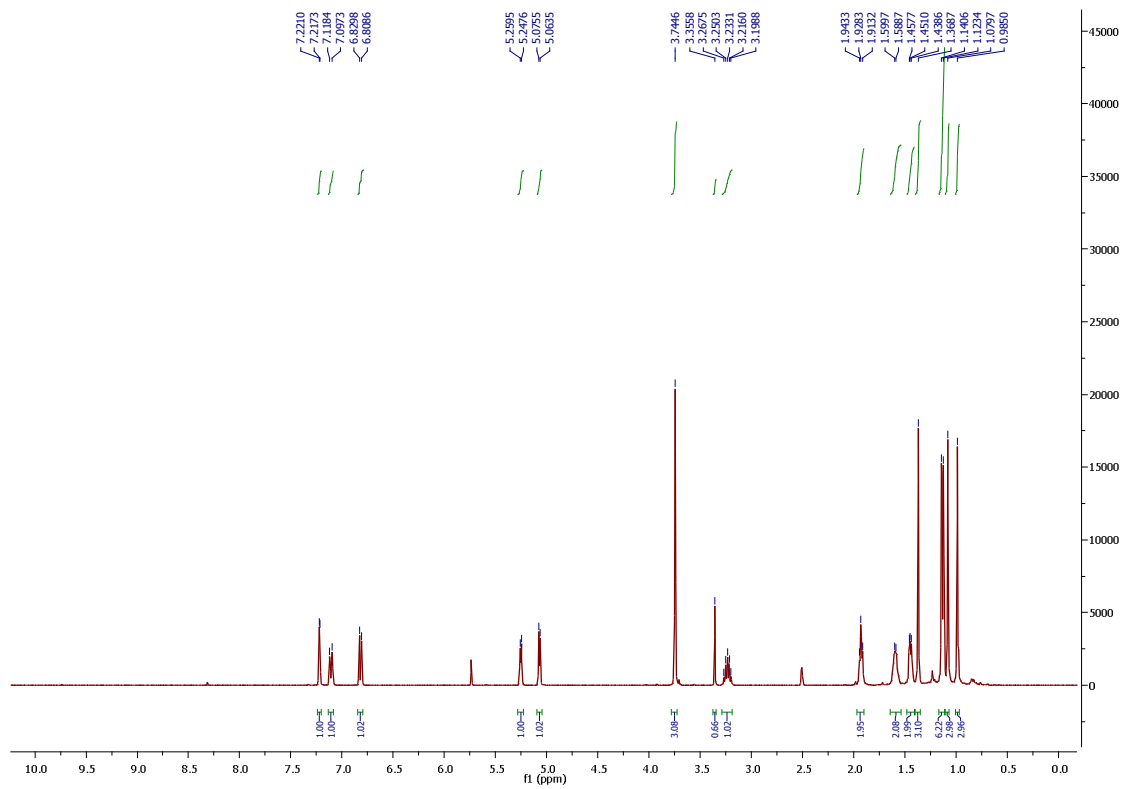
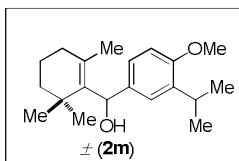
## Display Report

**Analysis Info**  
Analysis Name: D:\Data\user data\2013\FEB\05 FEB\Dr.A.Bisai-BK-3-208.d  
Method: tune\_low.m  
Sample Name: BK-3-208  
Comment:  
Acquisition Date: 2/6/2013 9:33:16 AM  
Operator: Amit  
Instrument: microTOF-Q II 10330

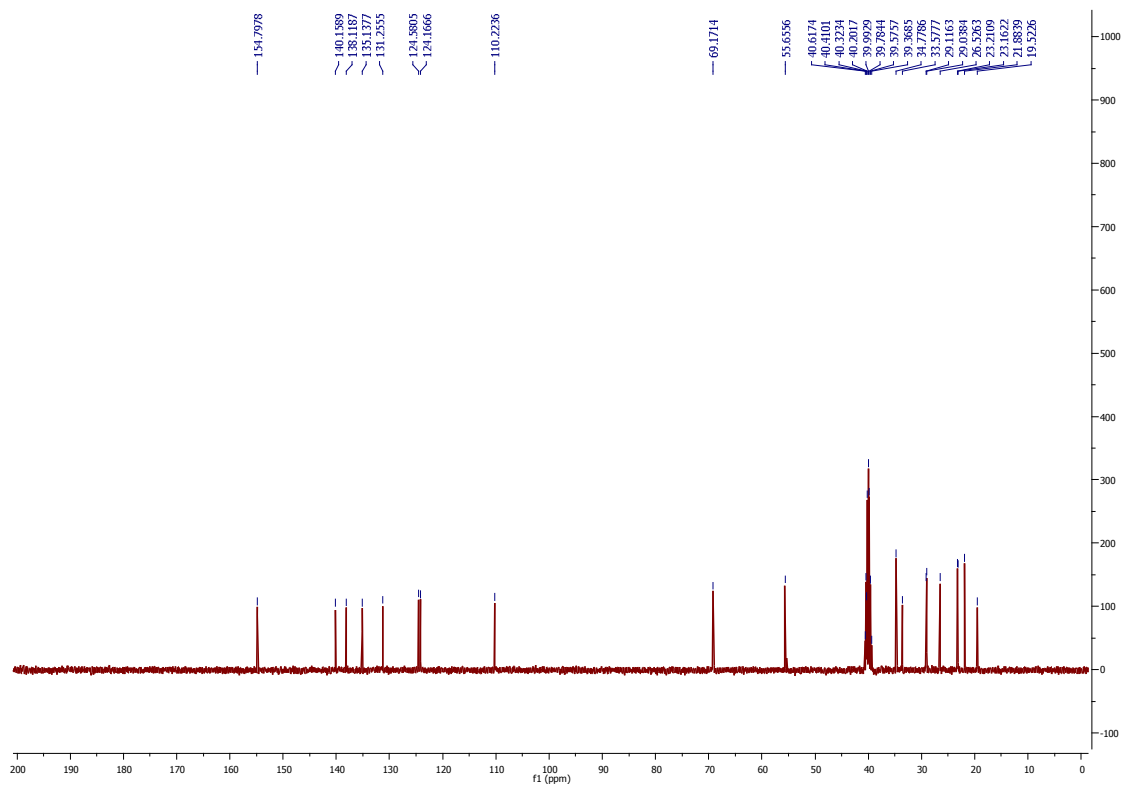
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Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

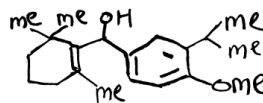




$^1\text{H}$  NMR (400 MHz, DMSO- $\text{D}_6$ ) compound  $\pm(2m)$



$^{13}\text{C}$  NMR (100 MHz, DMSO- $\text{D}_6$ ) compound  $\pm(2\text{m})$



## Display Report

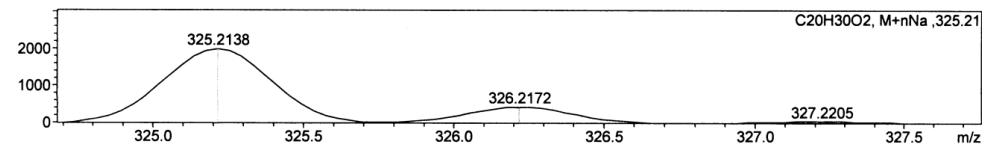
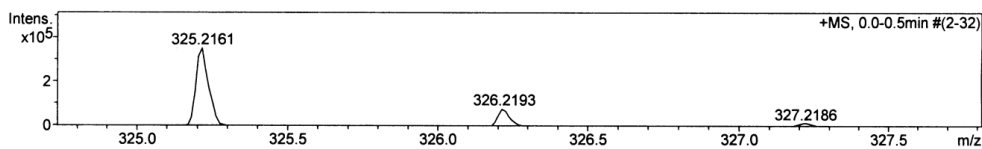
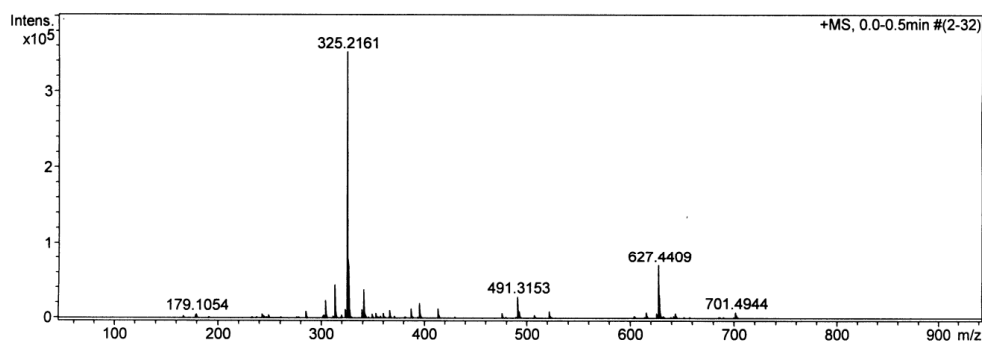
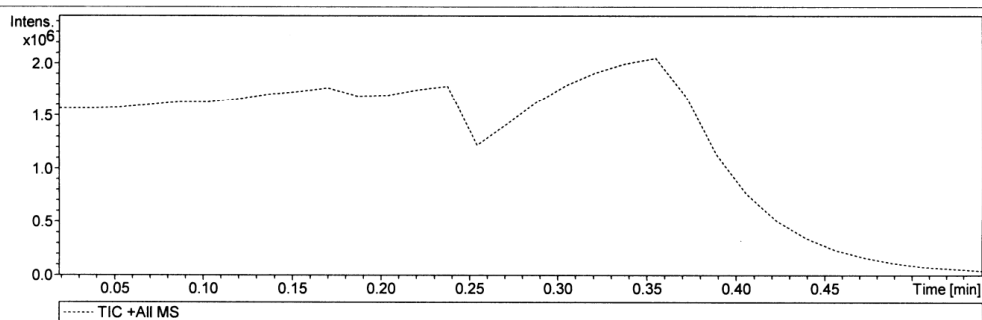
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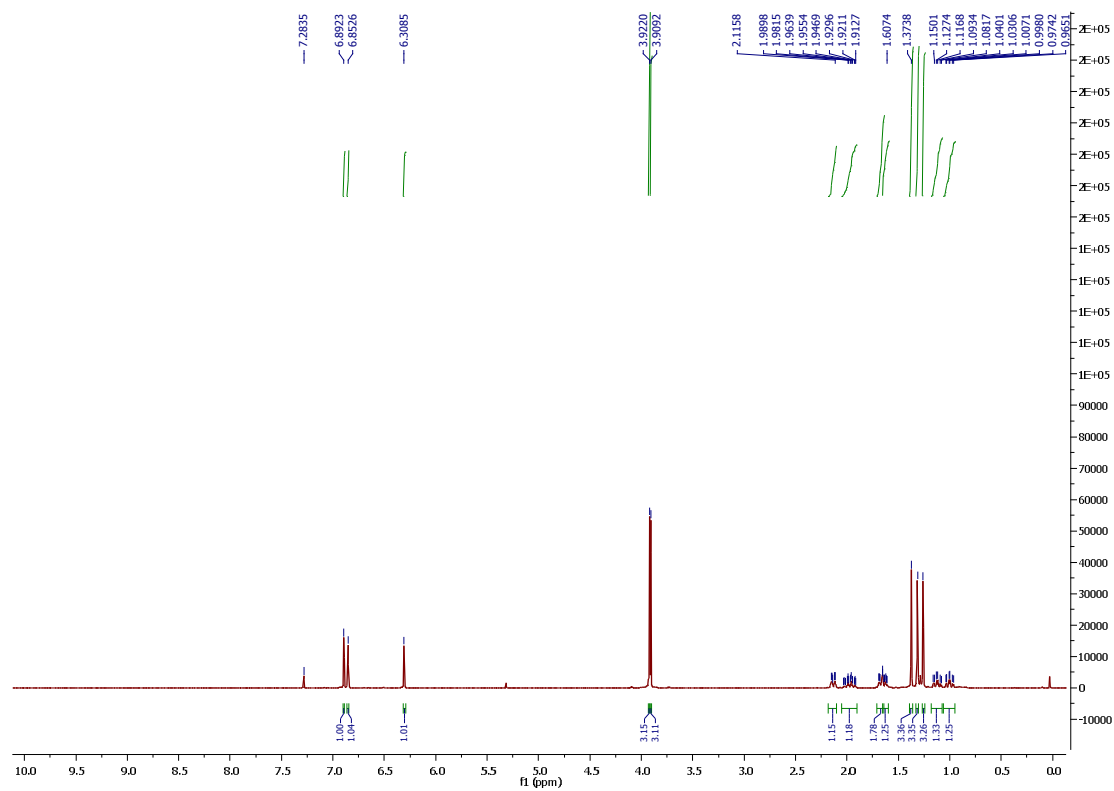
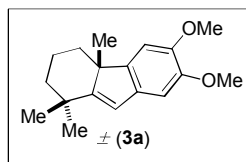
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Method tune\_low.m  
Sample Name BK-3-16  
Comment

Acquisition Date 1/17/2013 5:09:26 PM  
Operator IISERB  
Instrument micrOTOF-Q II 10330

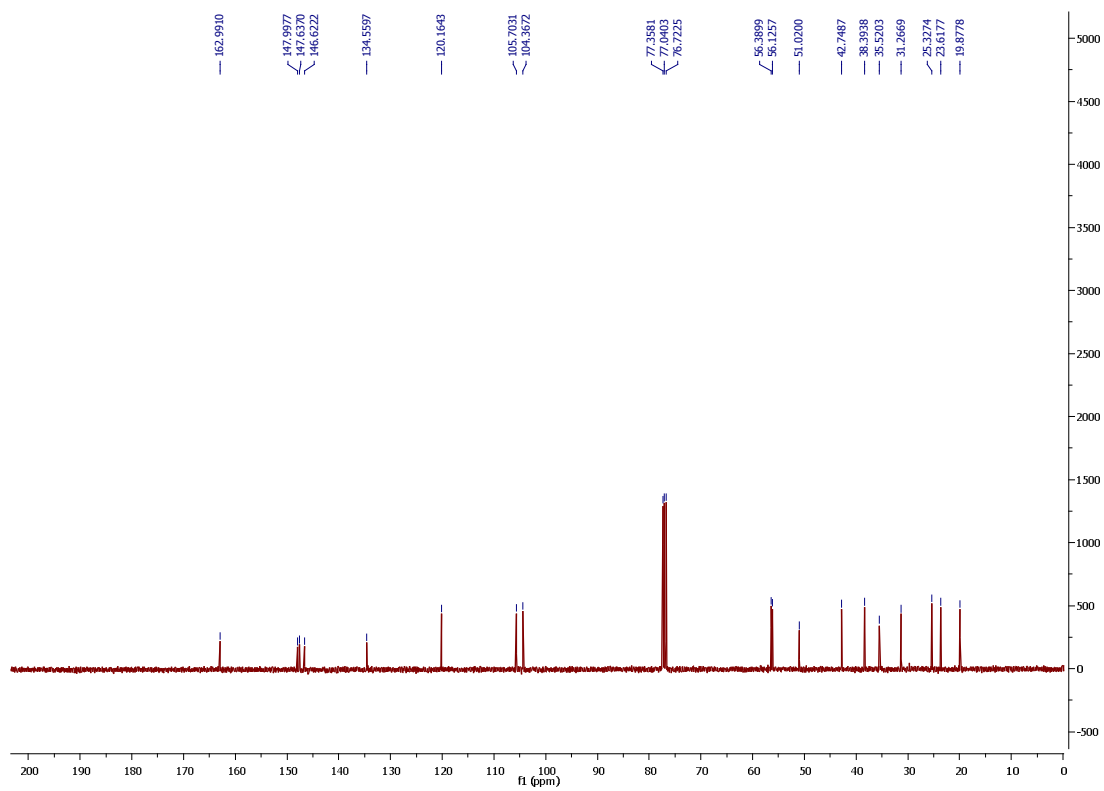
### Acquisition Parameter

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	130.0 Vpp	Set Divert Valve	Waste





$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3a)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(\mathbf{3a})$

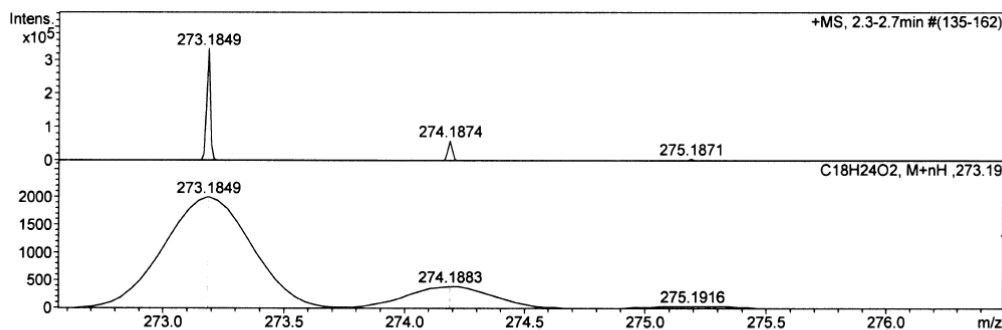
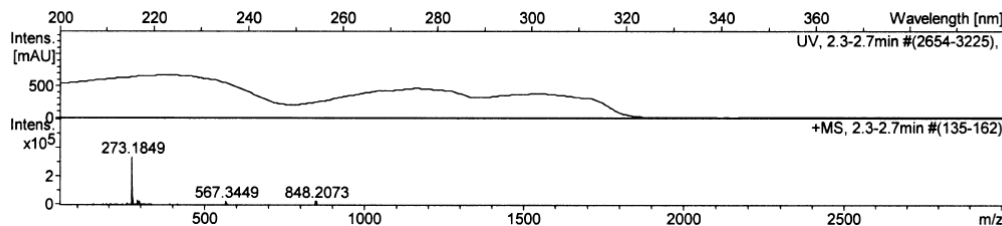
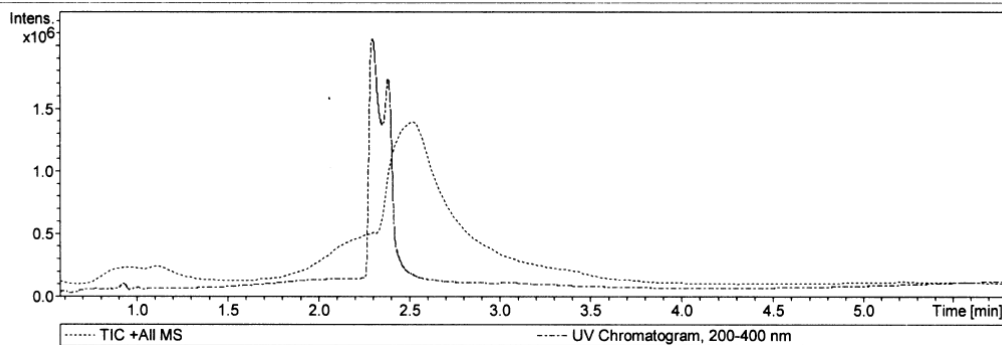




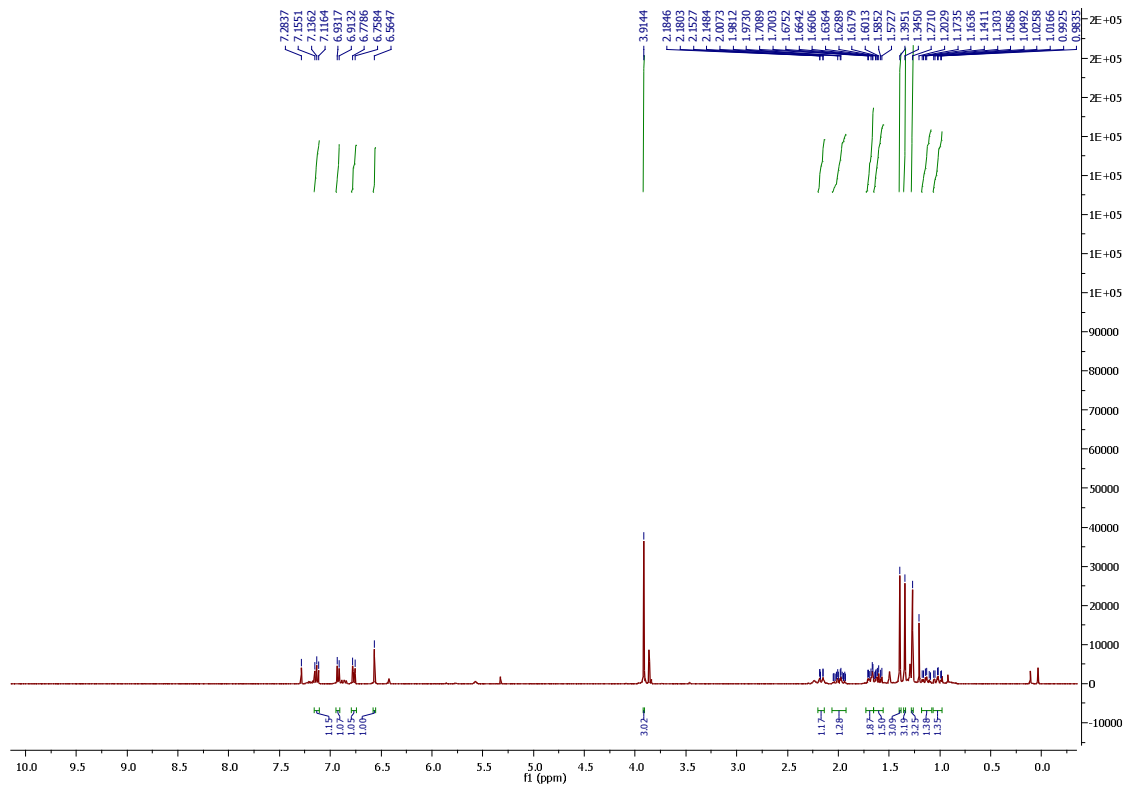
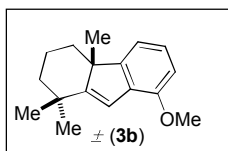
### Display Report

**Analysis Info**  
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Method HRLCMS-20 Sept.m  
Sample Name Dr.A . Bisai- BK-2-257  
Comment  
Acquisition Date 5/12/2012 3:58:33 PM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

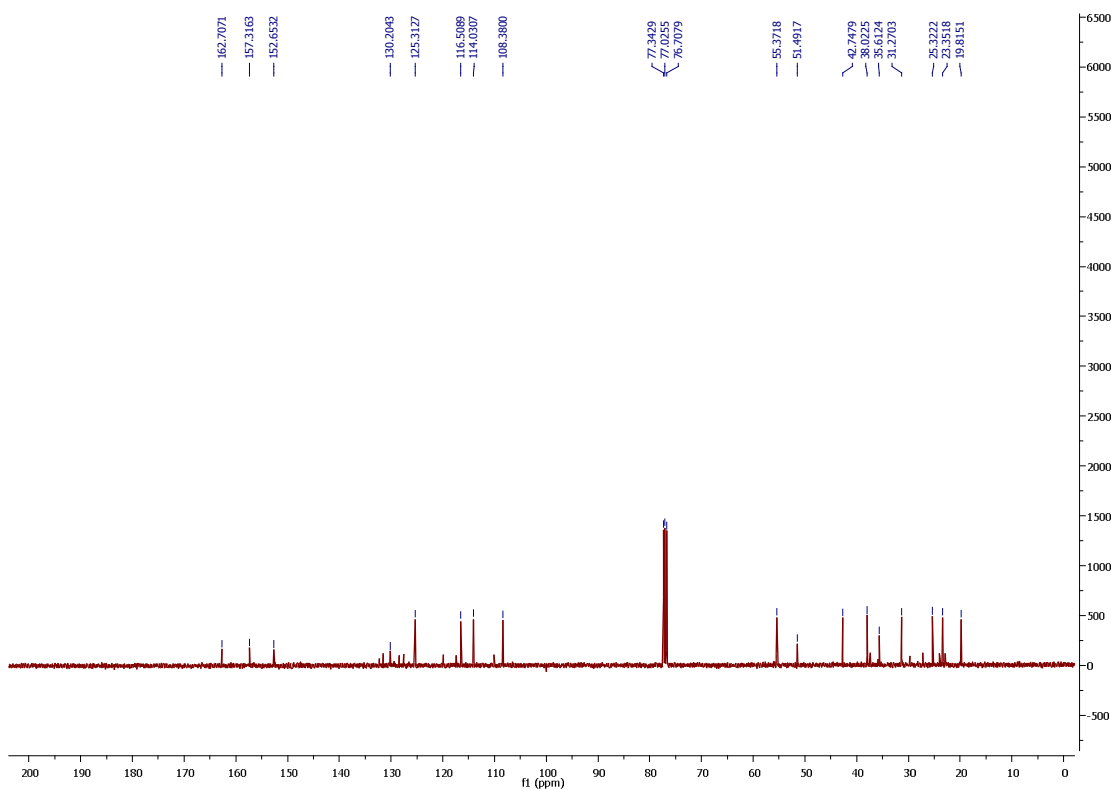
**Acquisition Parameter**  
Source Type ESI Ion Polarity Positive Set Nebulizer 1.2 Bar  
Focus Active Set Capillary 4500 V Set Dry Heater 200 °C  
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Scan End 3000 m/z Set Collision Cell RF 130.0 Vpp Set Divert Valve Waste



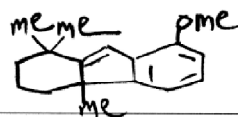
Scanned copy of mass spectrum (HRMS) of  $\pm(3a)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3b)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(\mathbf{3b})$

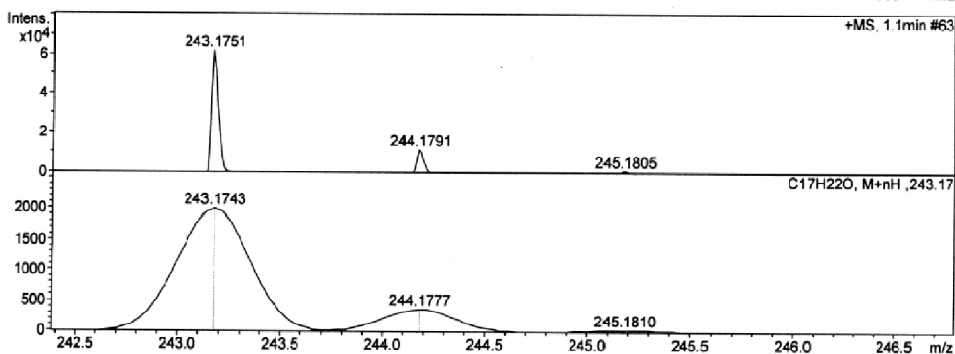
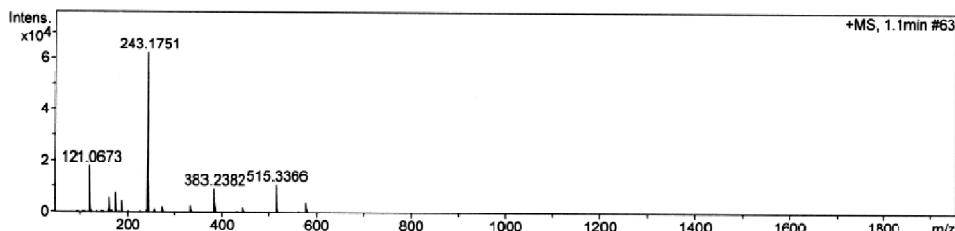
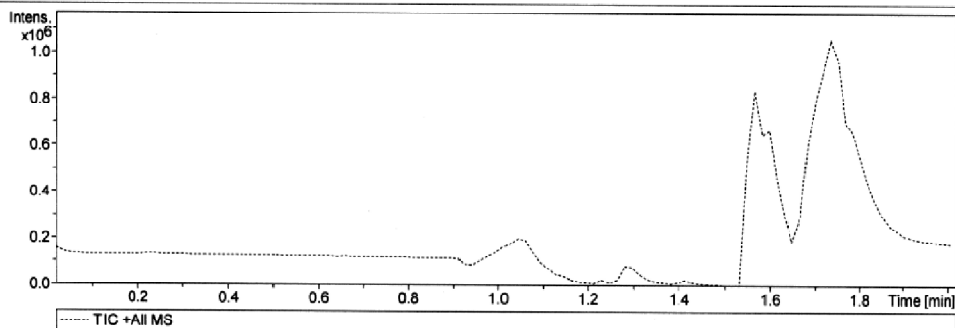


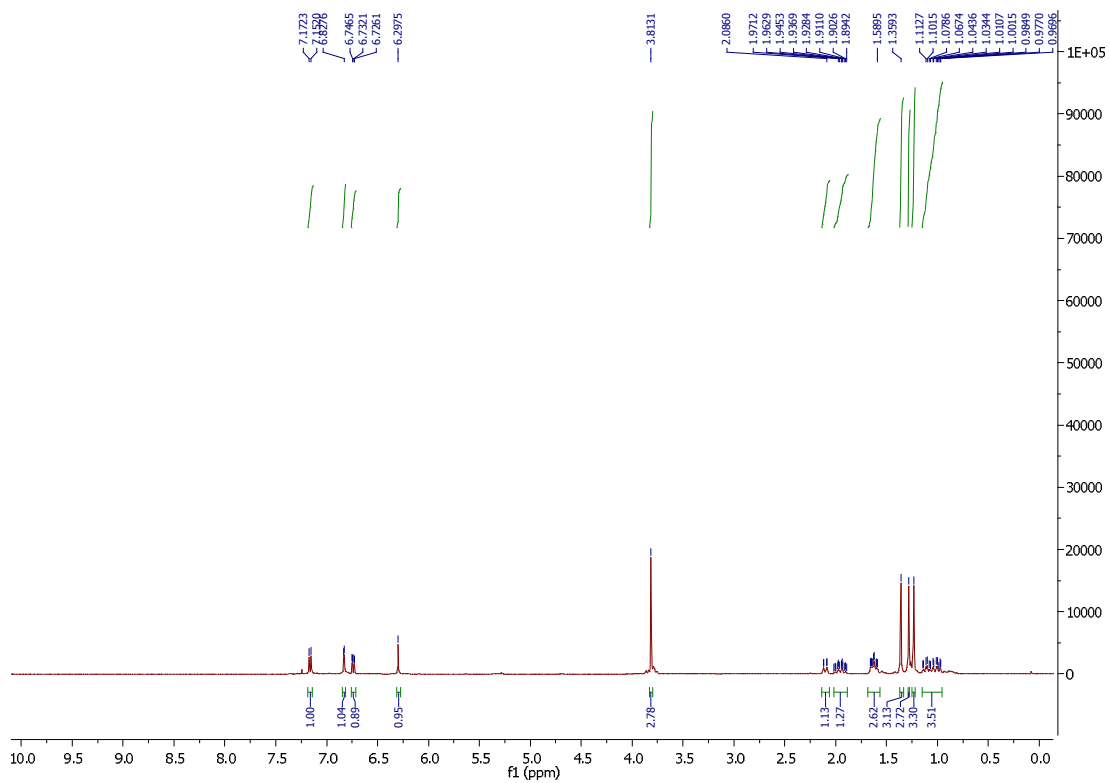
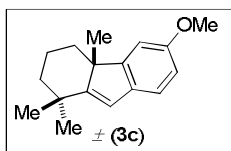
## Display Report

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Method tune\_low.m Operator Meena Sharma  
Sample Name AB-Bk-2-259 Instrument micrOTOF-Q II 10330  
Comment

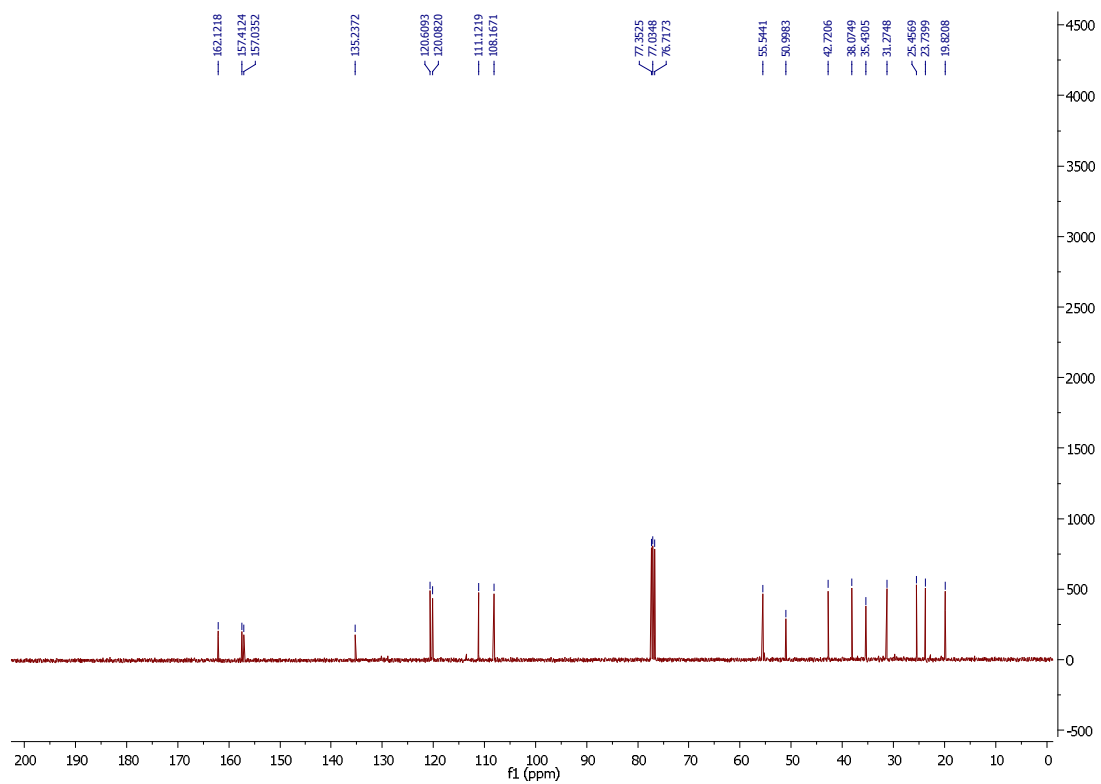
**Acquisition Parameter**

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	130.0 Vpp	Set Divert Valve	Waste





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



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\text{c})$



## Display Report

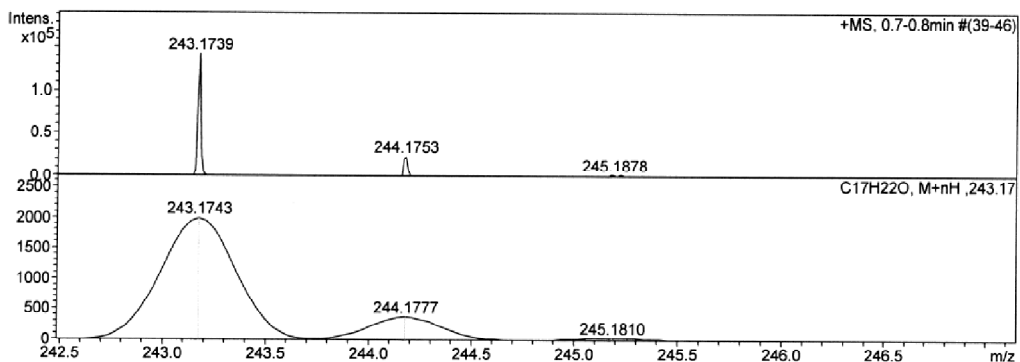
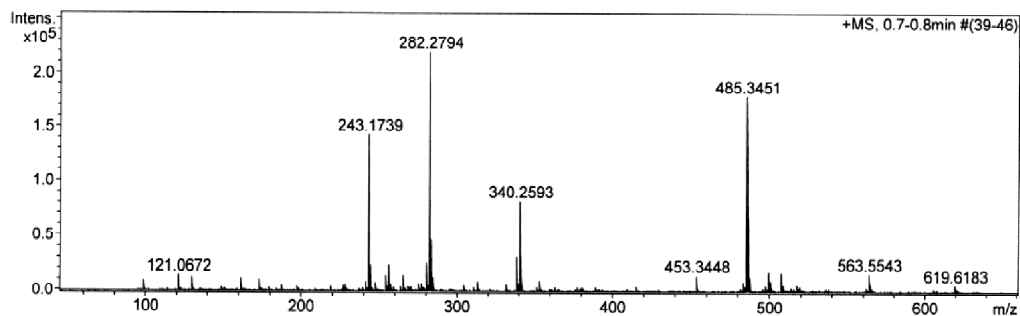
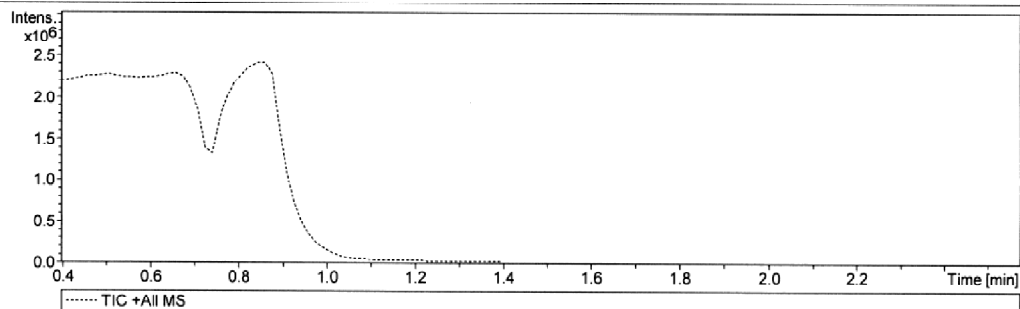
### Analysis Info

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Method tune\_low.m  
Sample Name BK-3-123  
Comment

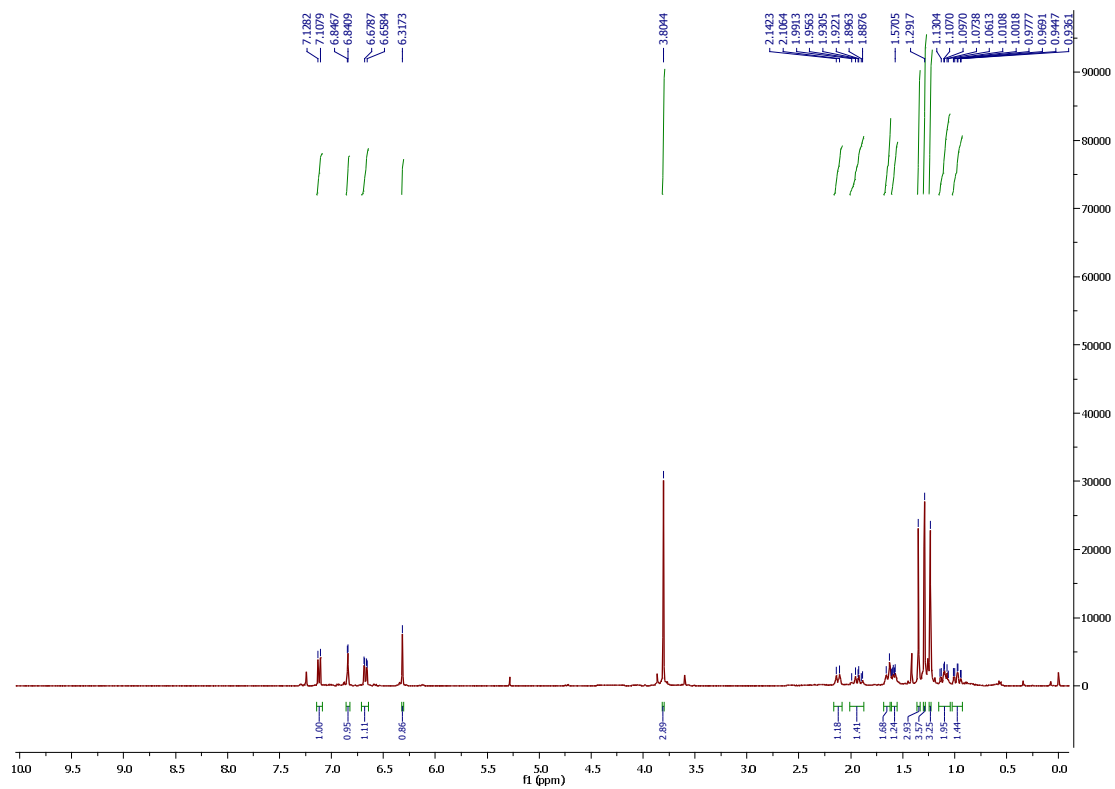
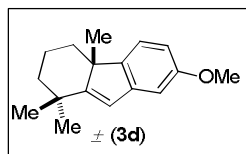
Acquisition Date 11/9/2012 3:45:49 PM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	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	130.0 Vpp	Set Divert Valve	Waste

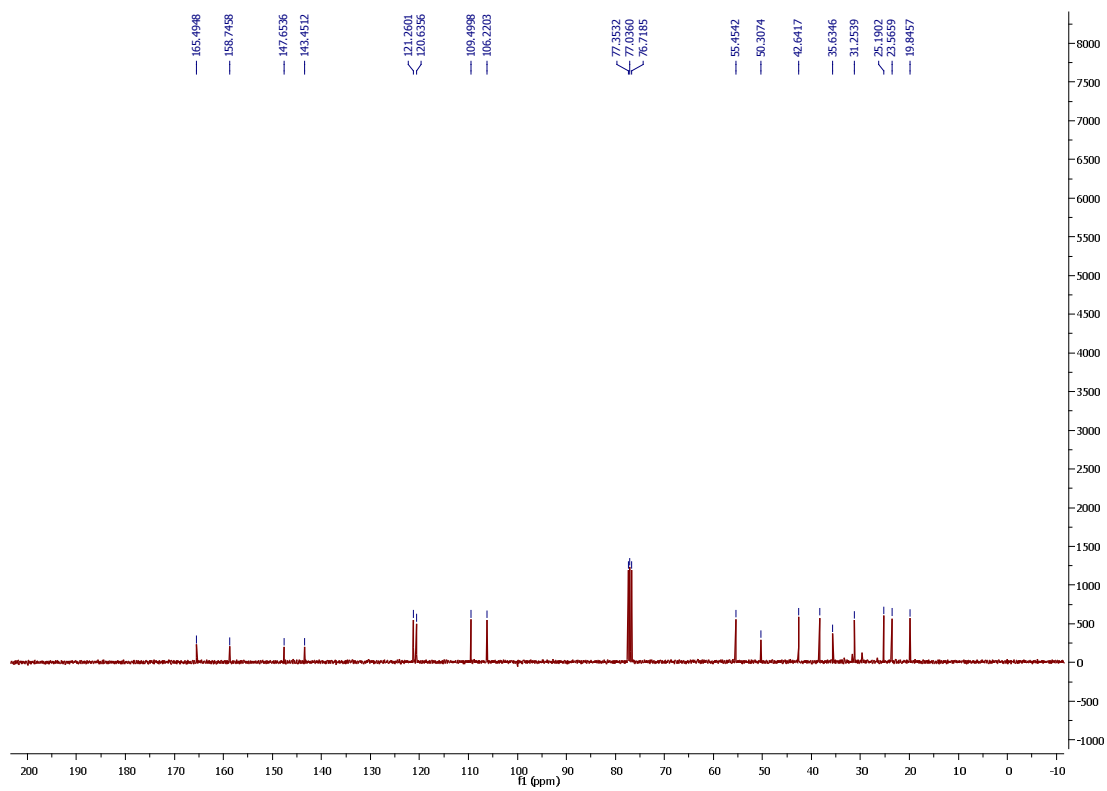


Scanned copy of mass spectrum (HRMS) of  $\pm(3c)$

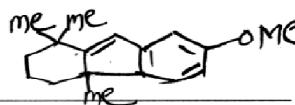


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\text{d})$





$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(\mathbf{3d})$



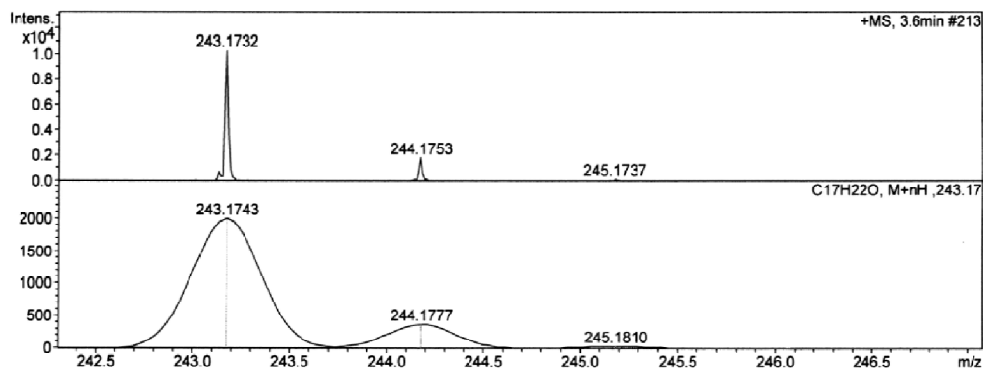
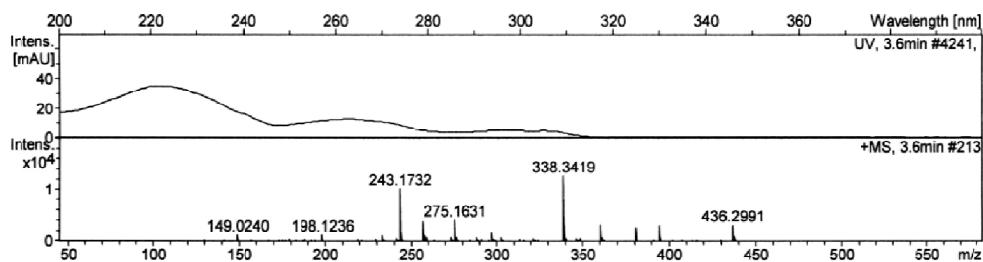
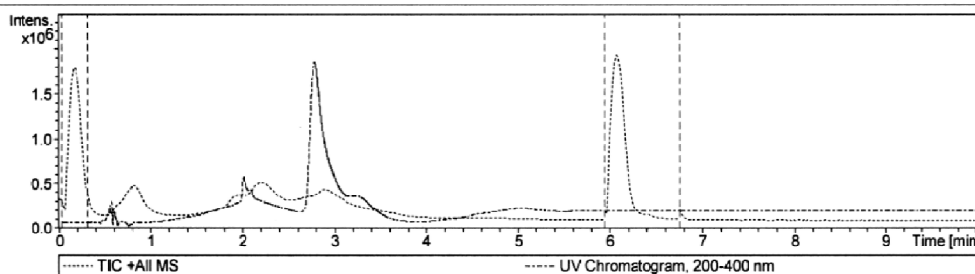
## Display Report

### Analysis Info

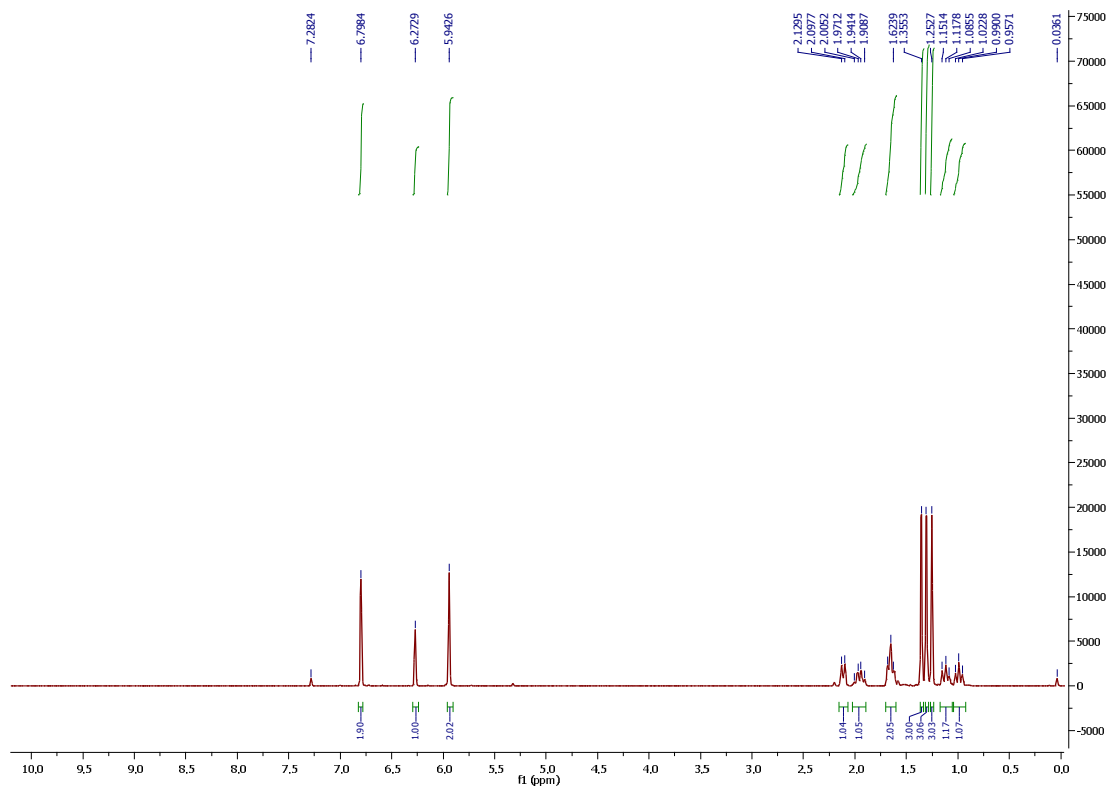
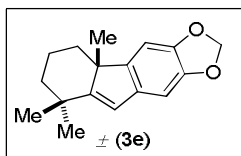
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Method	HRLCMS-20 Sept.m	Operator	Meena Sharma
Sample Name	Dr. A. Bisai- BK-2-171(BF3)	Instrument	micrOTOF-Q II 10330
Comment			

### Acquisition Parameter

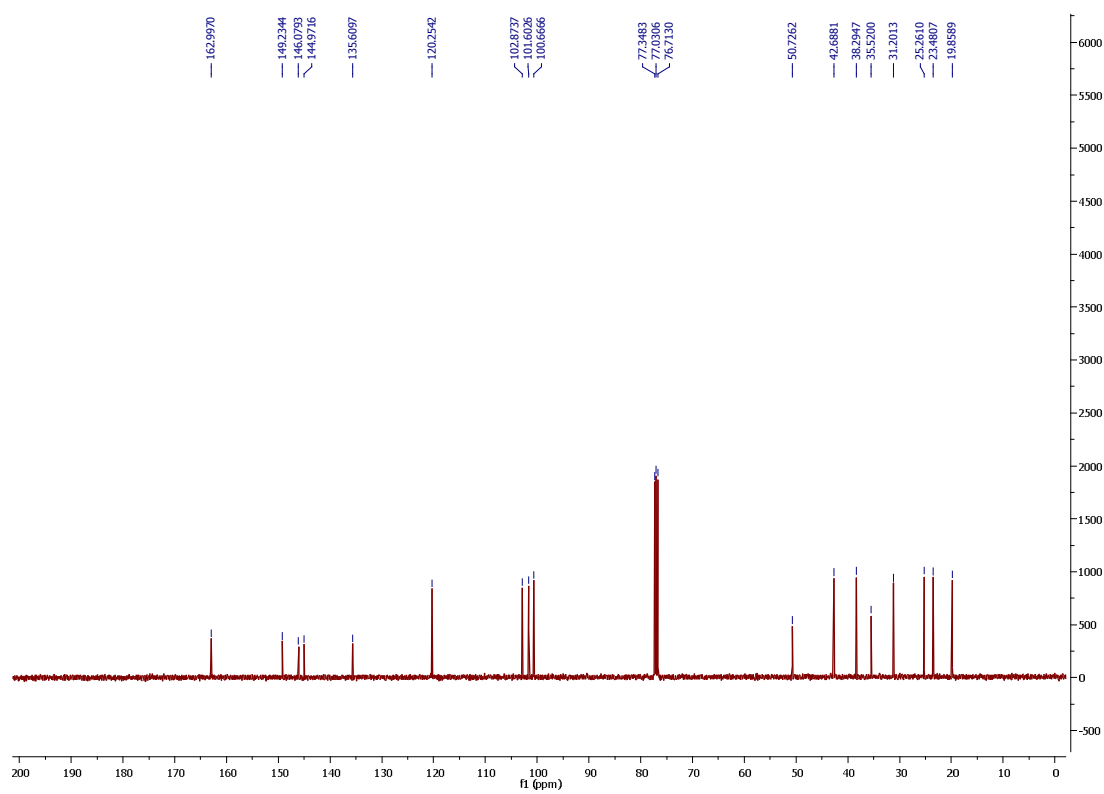
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



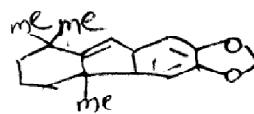
Scanned copy of mass spectrum (HRMS) of  $\pm(3d)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3e)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\mathbf{e})$

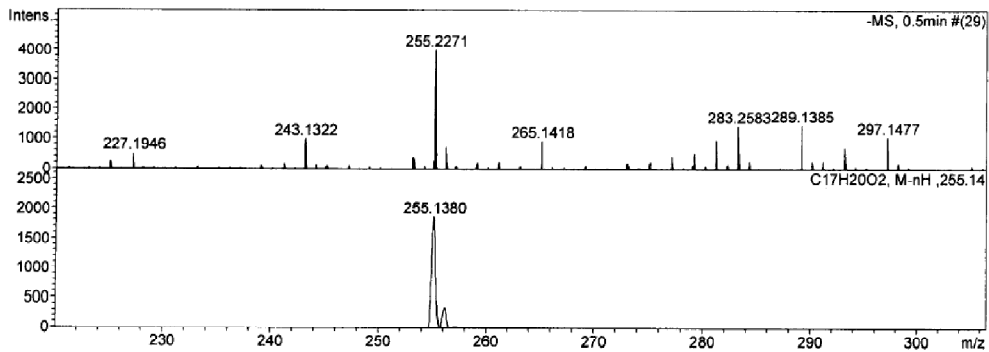
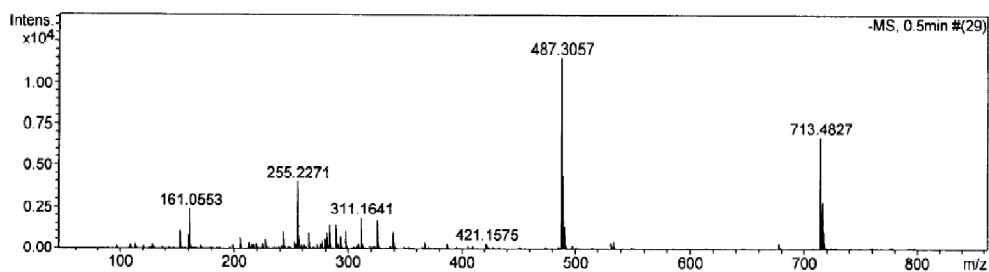
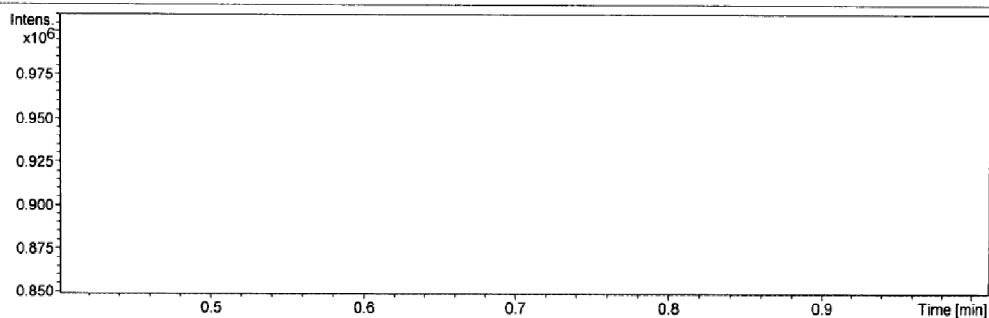


### Display Report

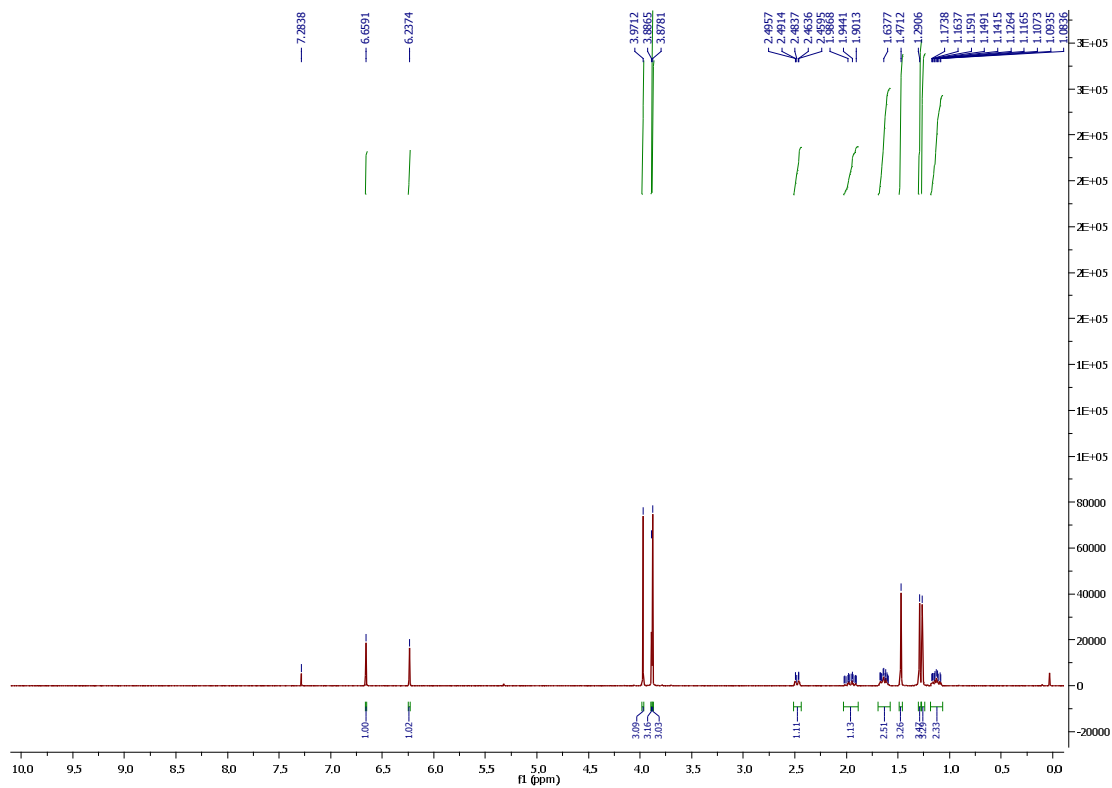
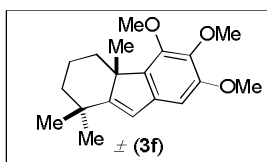
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Method: tune\_low.m  
Sample Name: BK-2-258(P-6)  
Comment:  
Acquisition Date: 9/29/2012 10:44:53 AM  
Operator: Meena Sharma  
Instrument: micrOTOF-Q II 10330

**Acquisition Parameter**

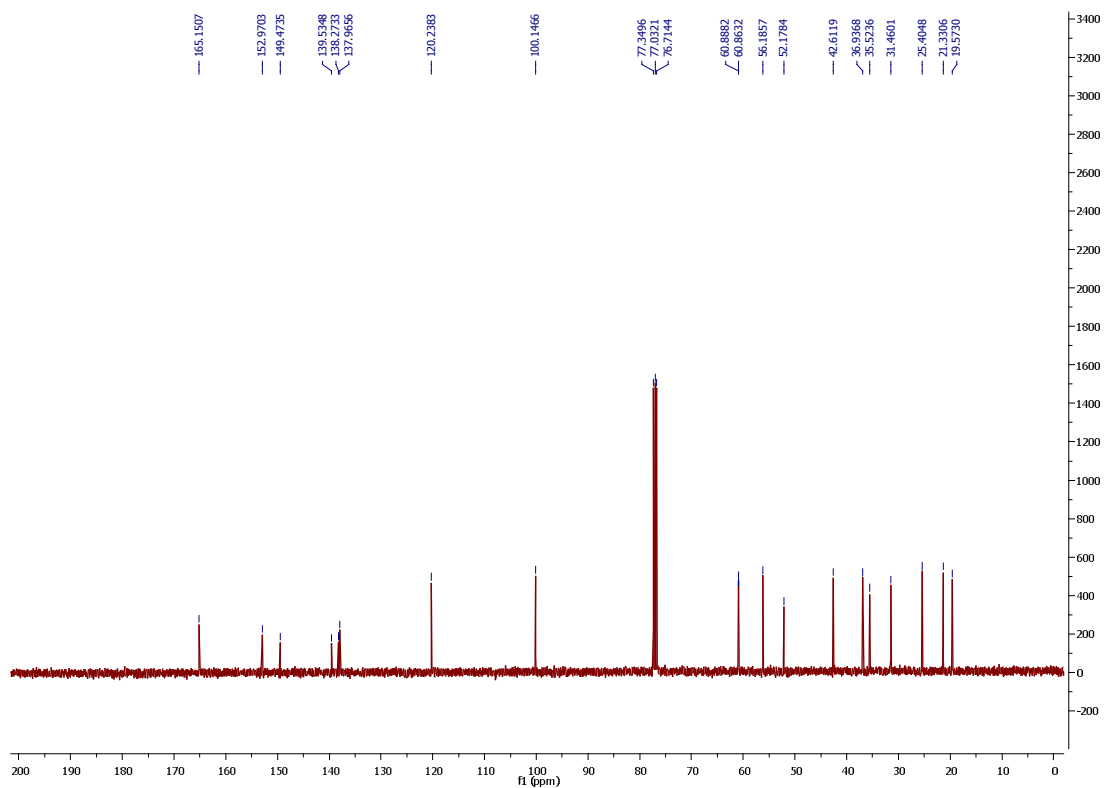
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum (LRMS) of  $\pm(3e)$



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



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3f)$



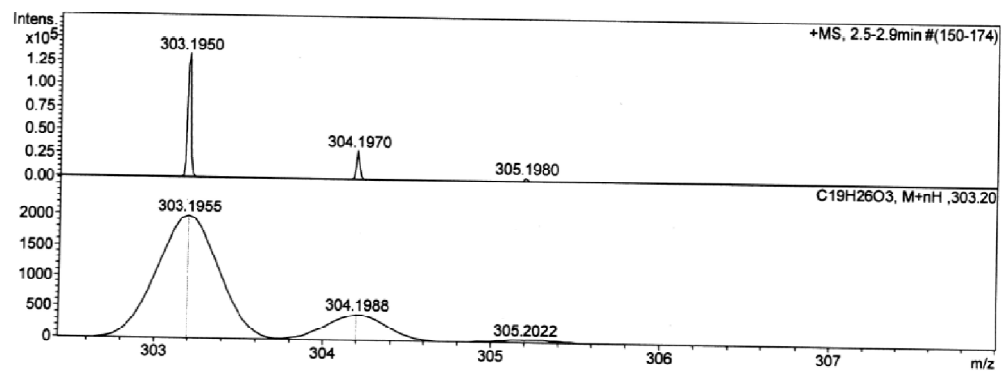
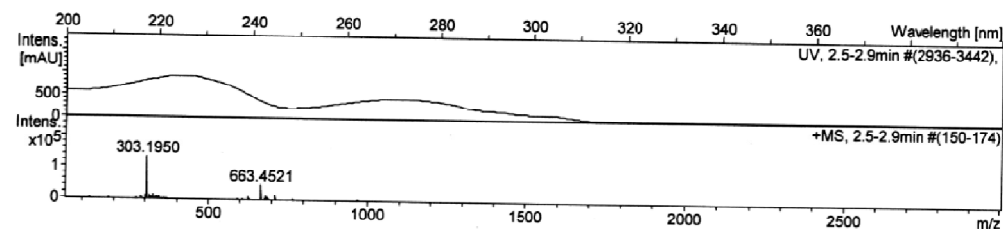
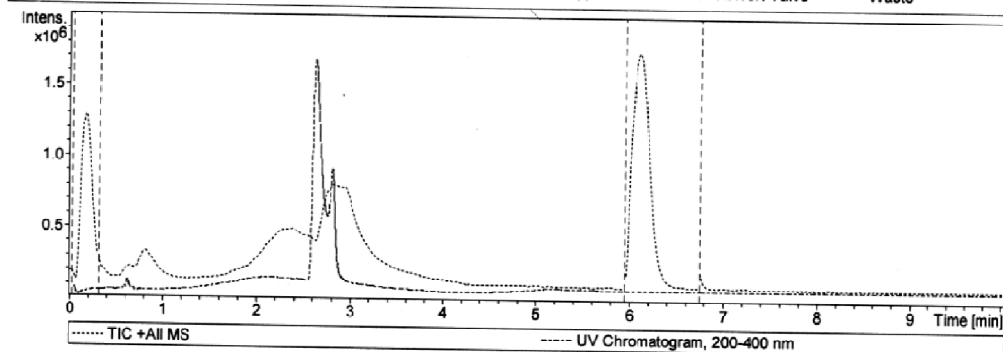
## Display Report

### Analysis Info

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Method	HRLCMS-20 Sept.m	Operator	Meena Sharma
Sample Name	Dr. A. Bisai- Bk-271	Instrument	micrOTOF-Q II 10330
Comment			

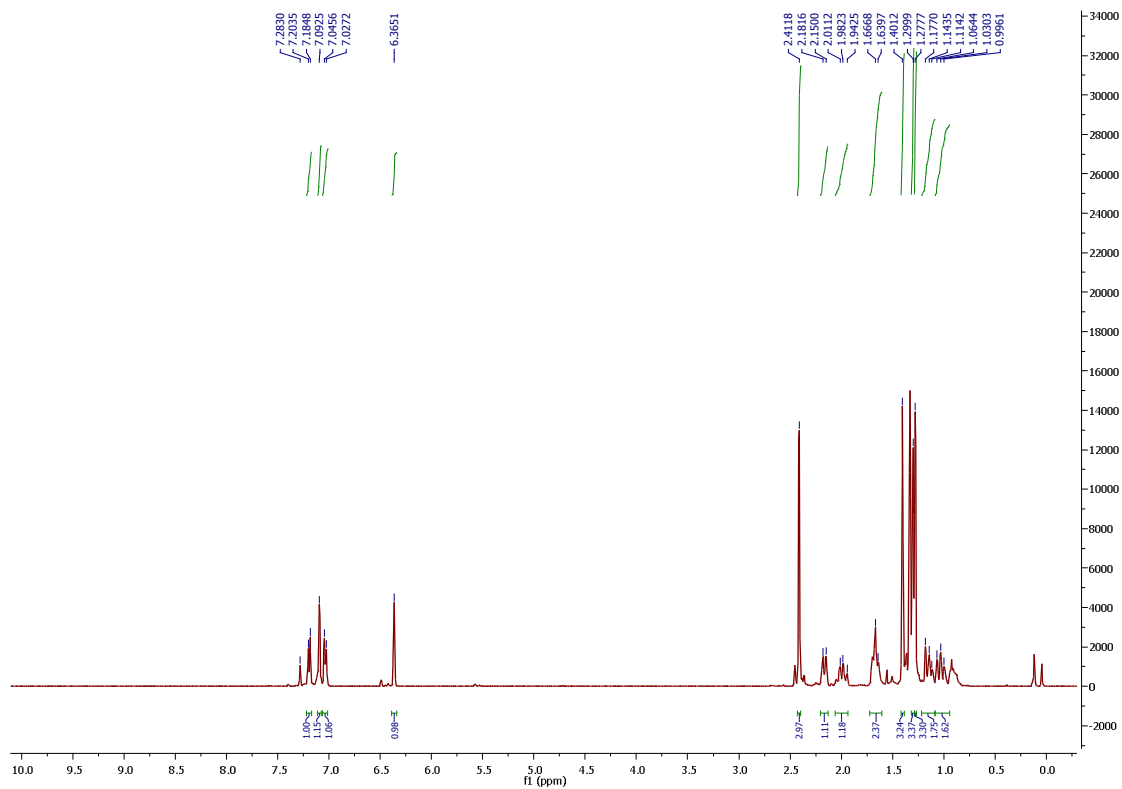
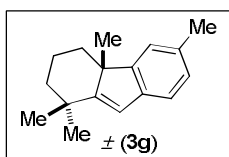
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Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

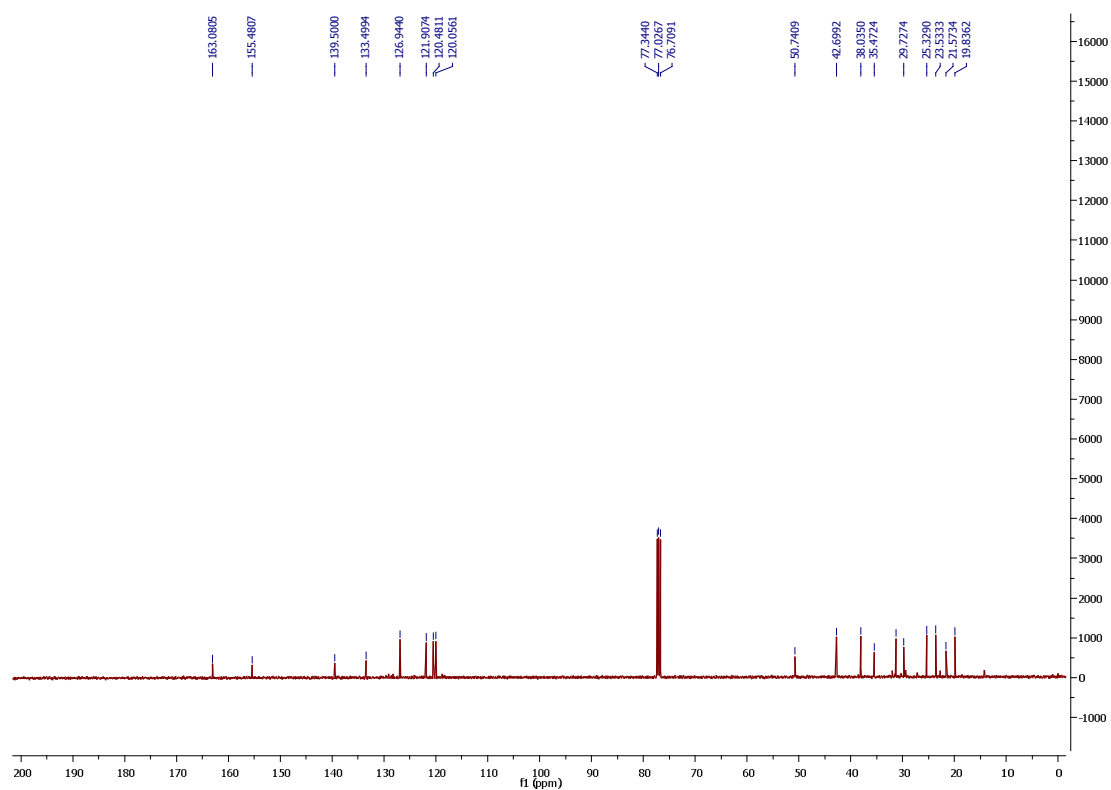


Scanned copy of mass spectrum (HRMS) of  $\pm(3f)$

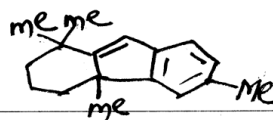




$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3g)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\mathbf{g})$

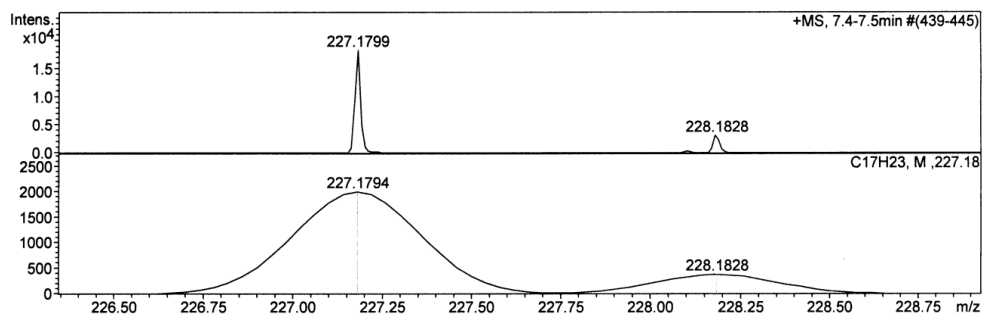
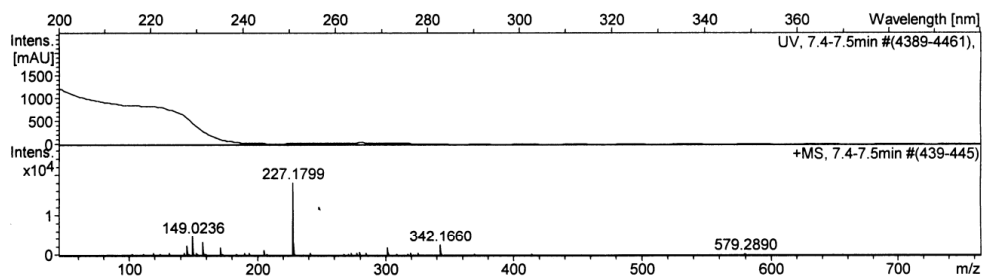
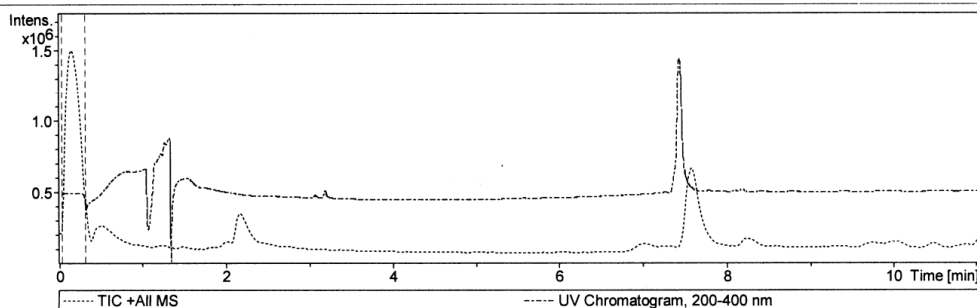


## Display Report

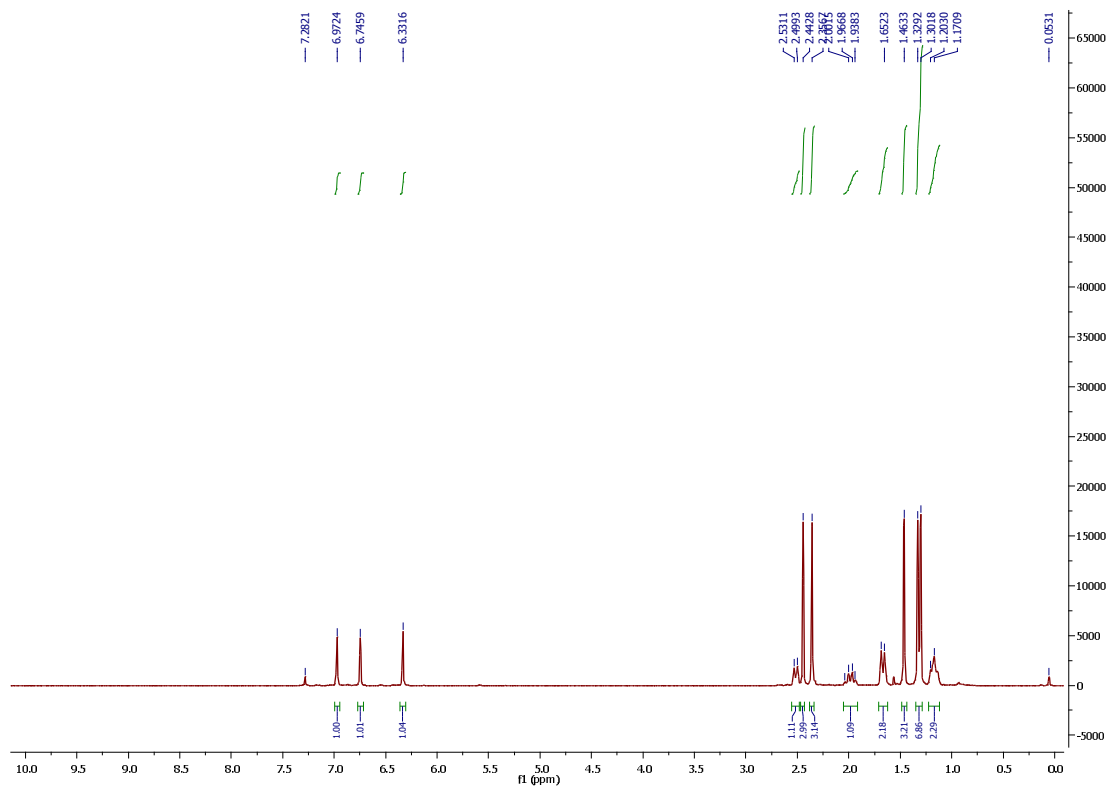
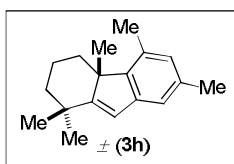
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Analysis Name D:\Data\user data\JULY 2012\20 July\Dr.A.Bisai-BK-2-233\_1-A,5\_01\_2834.d Acquisition Date 7/23/2012 12:00:15 PM  
Method HRLCMS-20 Sept.m Operator Meena Sharma  
Sample Name Dr.A.Bisai-BK-2-233 Instrument microTOF-Q II 10330  
Comment

**Acquisition Parameter**

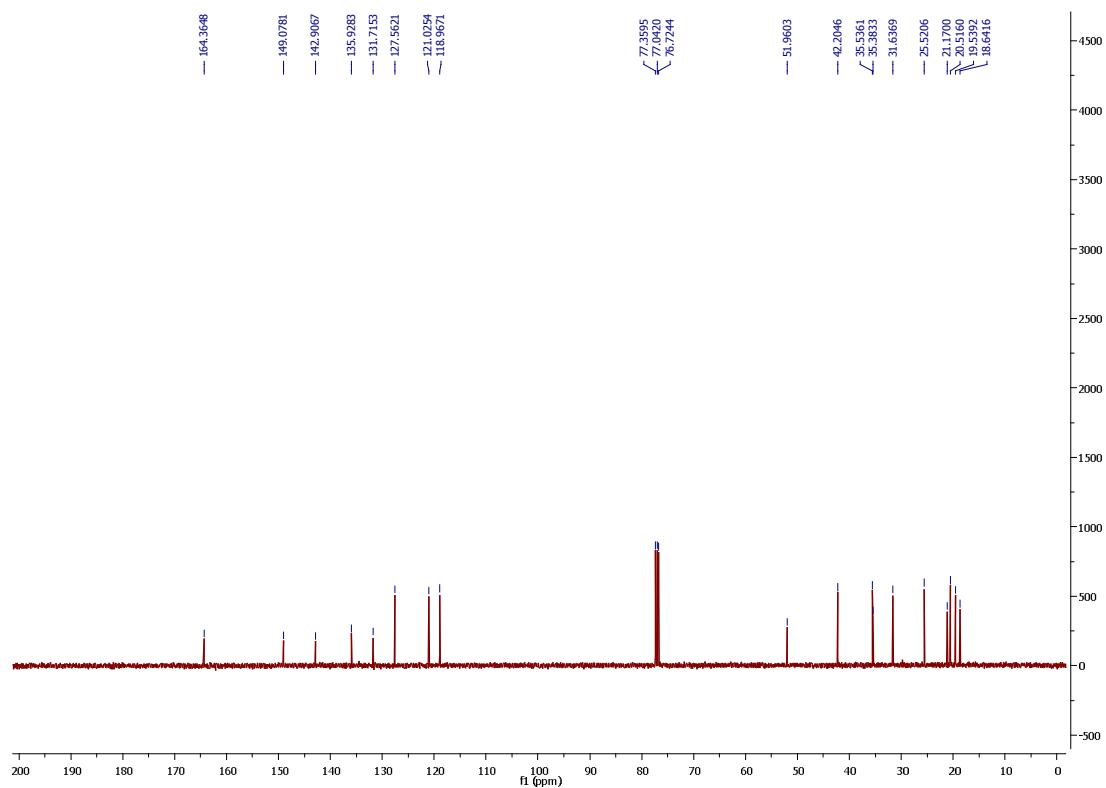
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



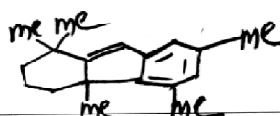
Scanned copy of mass spectrum (HRMS) of  $\pm(3g)$



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound **±(3h)**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(\mathbf{3h})$

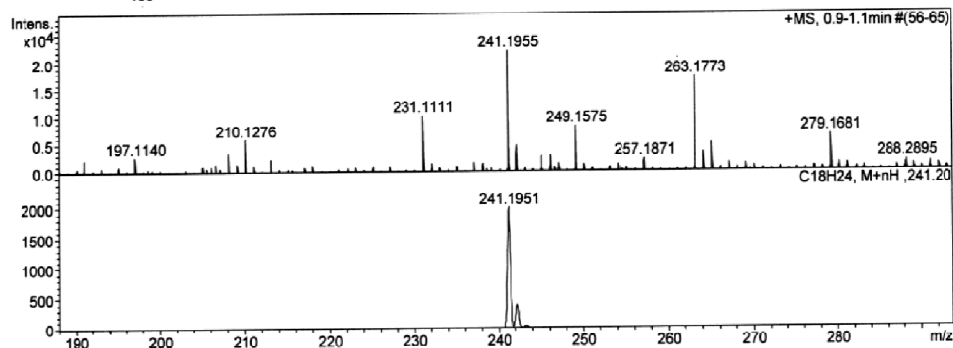
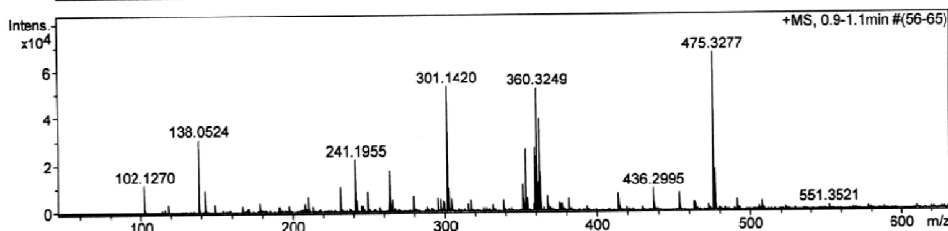
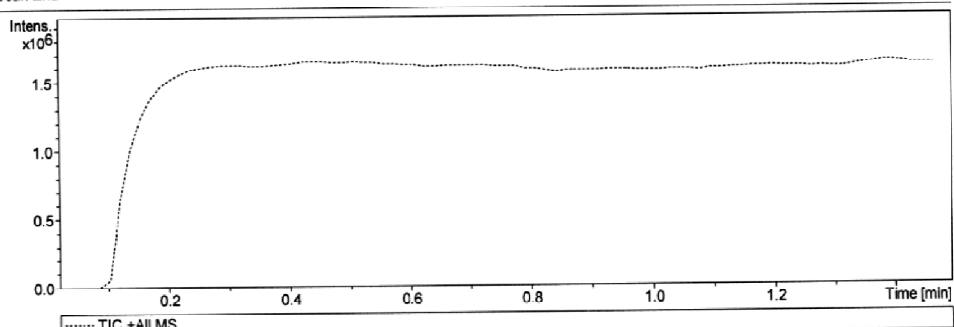


## Display Report

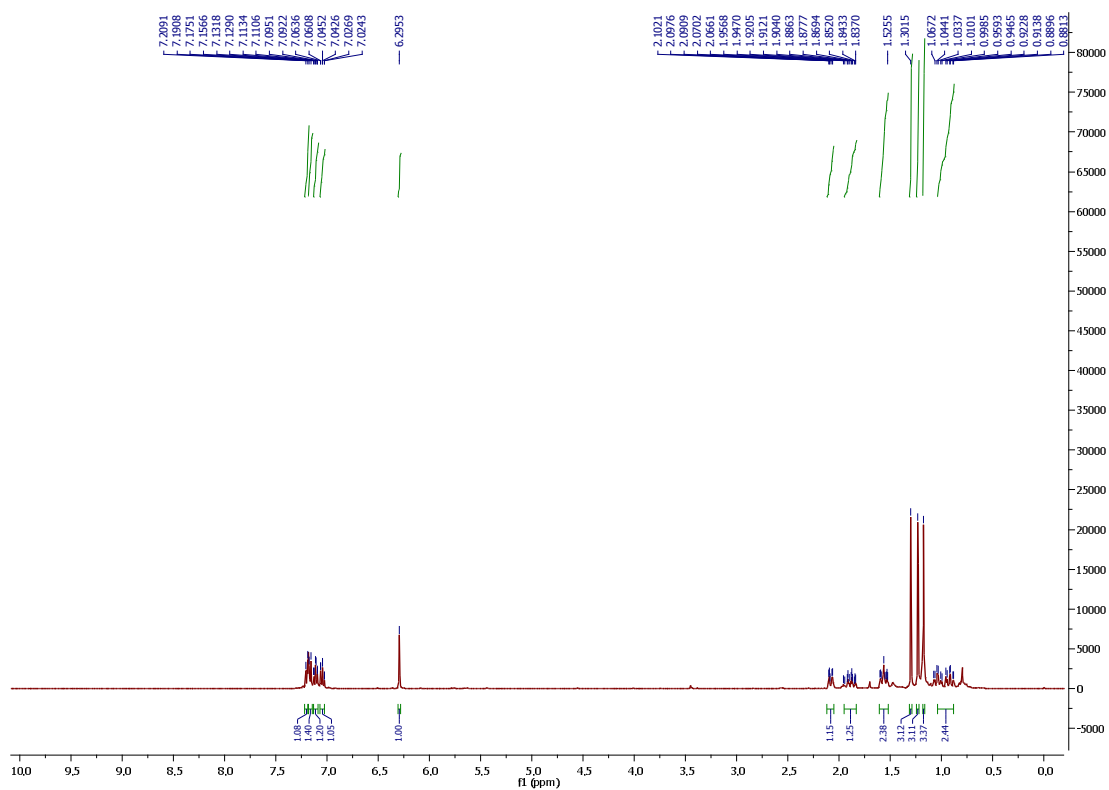
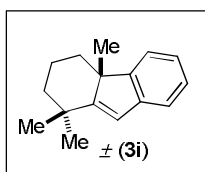
**Analysis Info**  
Analysis Name D:\Data\user data\may2012\7 may\Dr. A. Bisai- BK-2-262-1.d  
Method tune\_low.m  
Sample Name AB-BK-2-262  
Comment  
Acquisition Date 5/7/2012 4:16:53 PM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

**Acquisition Parameter**

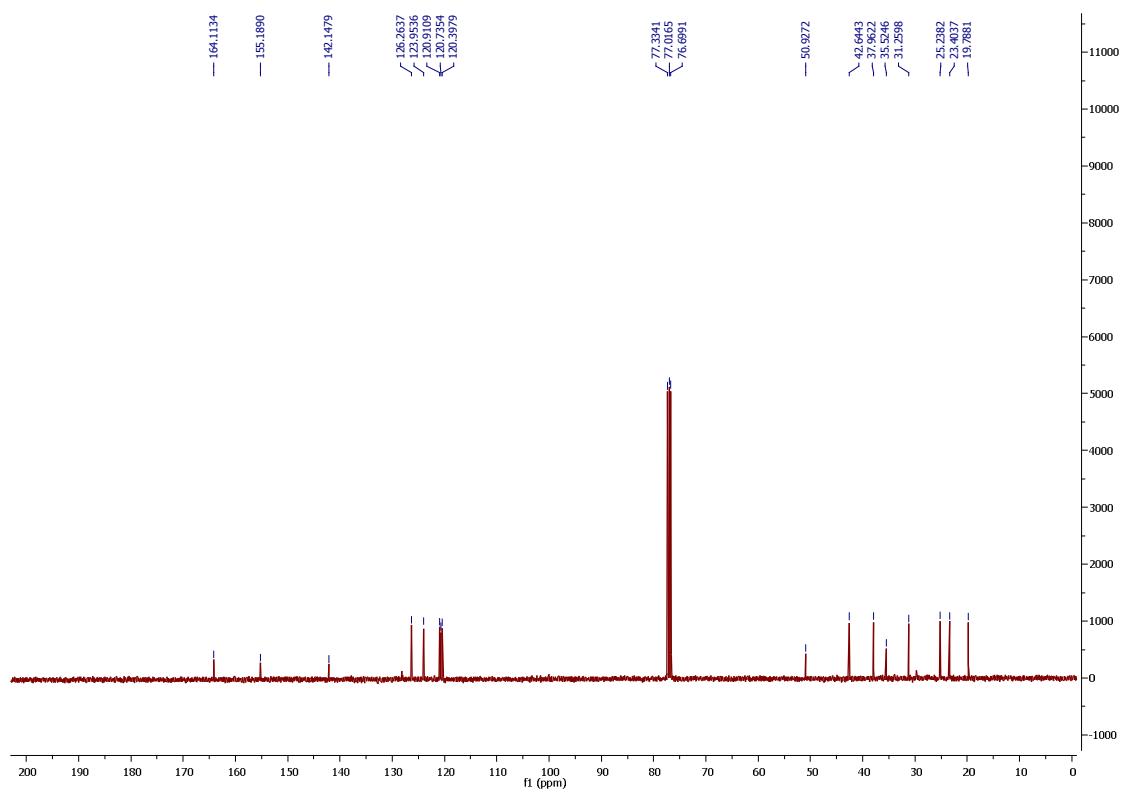
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum (HRMS) of  $\pm(3h)$

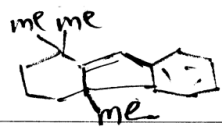


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3i)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(\mathbf{3i})$





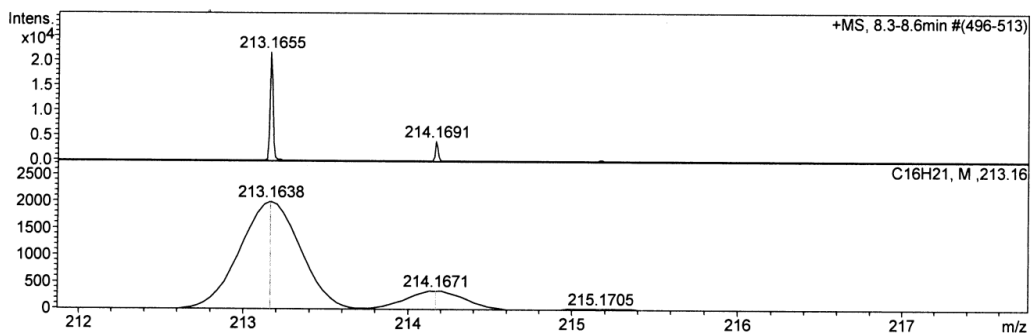
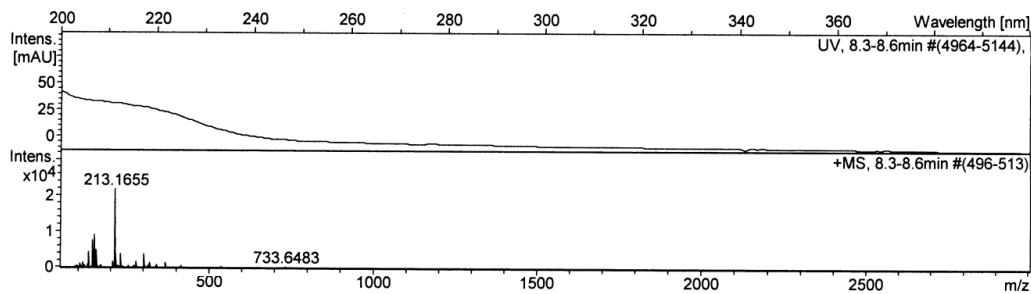
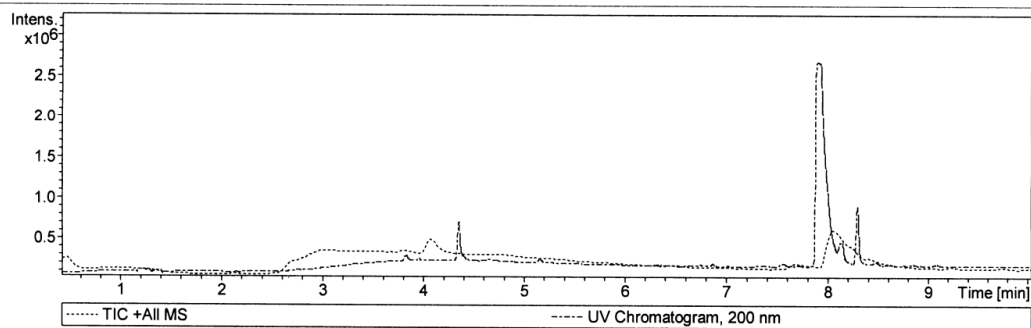
## Display Report

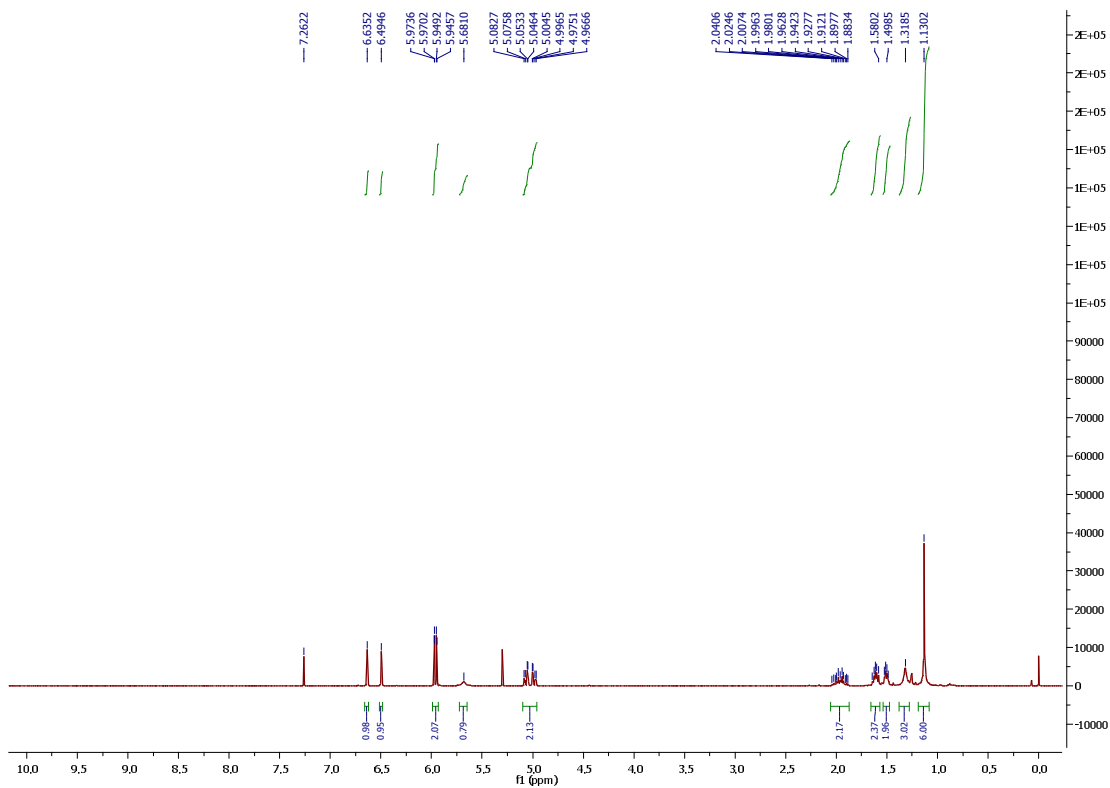
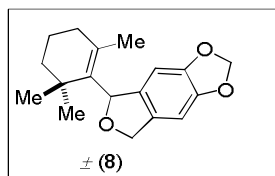
### Analysis Info

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Method HRLCMS-20 Sept.m  
Sample Name Dr. A. Bisai-BK-2-219  
Comment  
Acquisition Date 7/21/2012 5:22:01 PM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

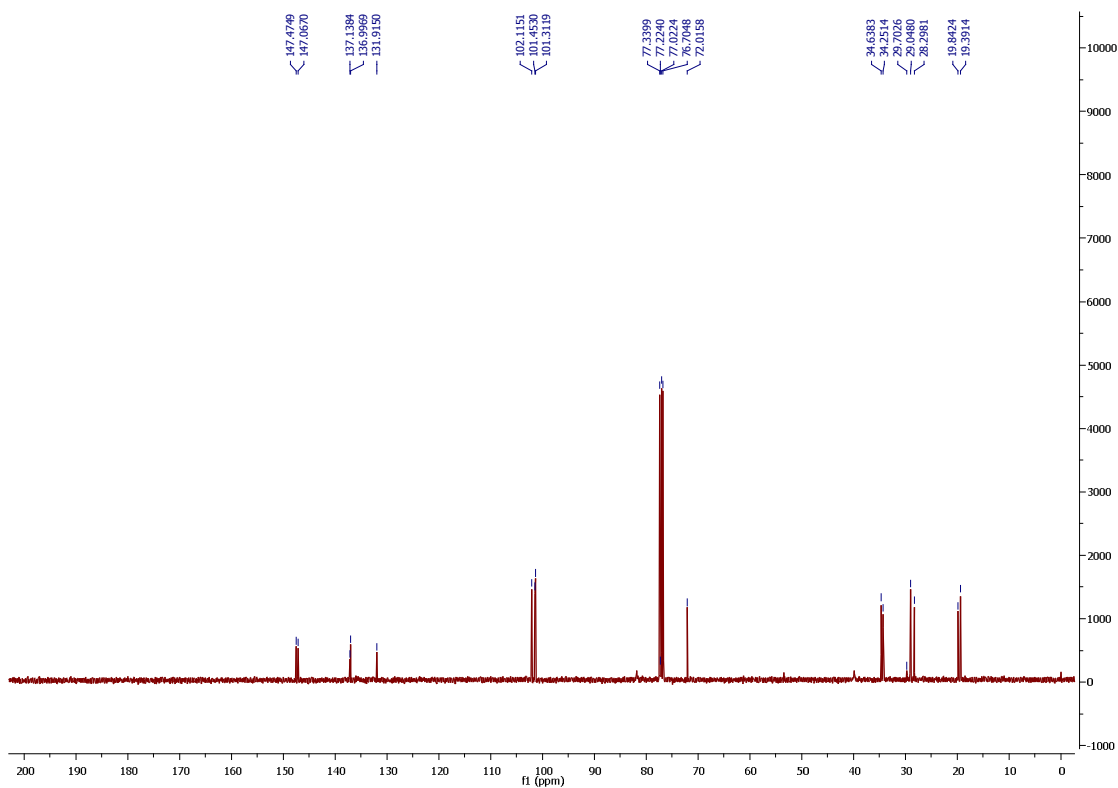
### Acquisition Parameter

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

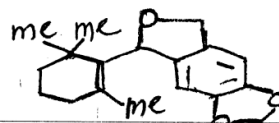




<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound ±(8)



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



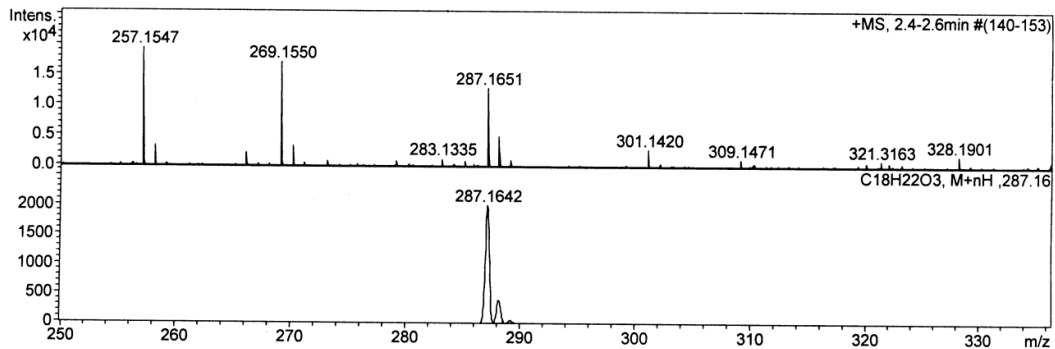
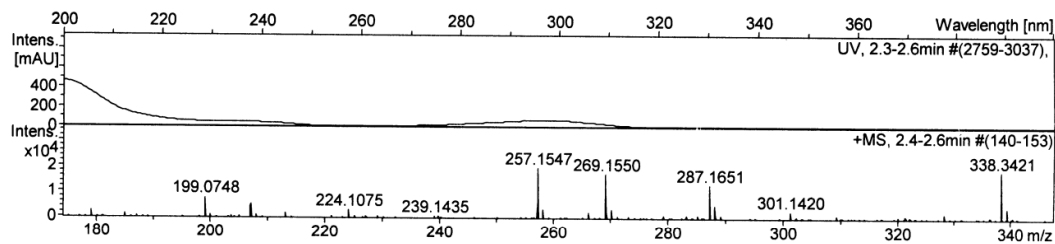
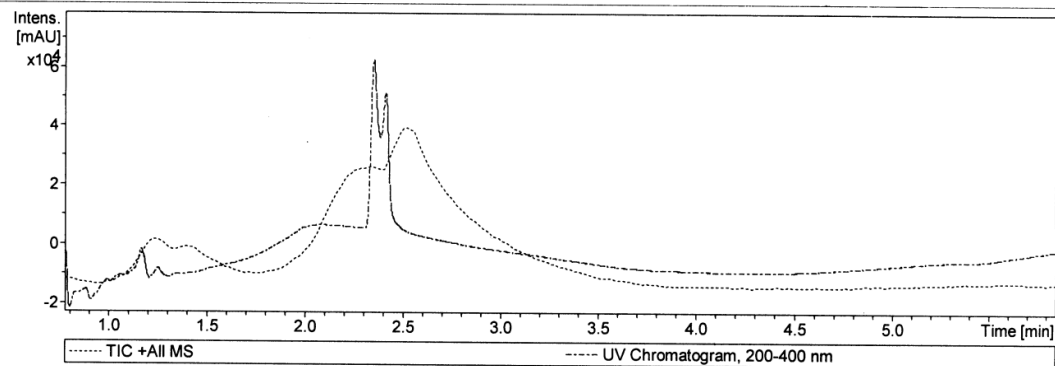
## Display Report

### Analysis Info

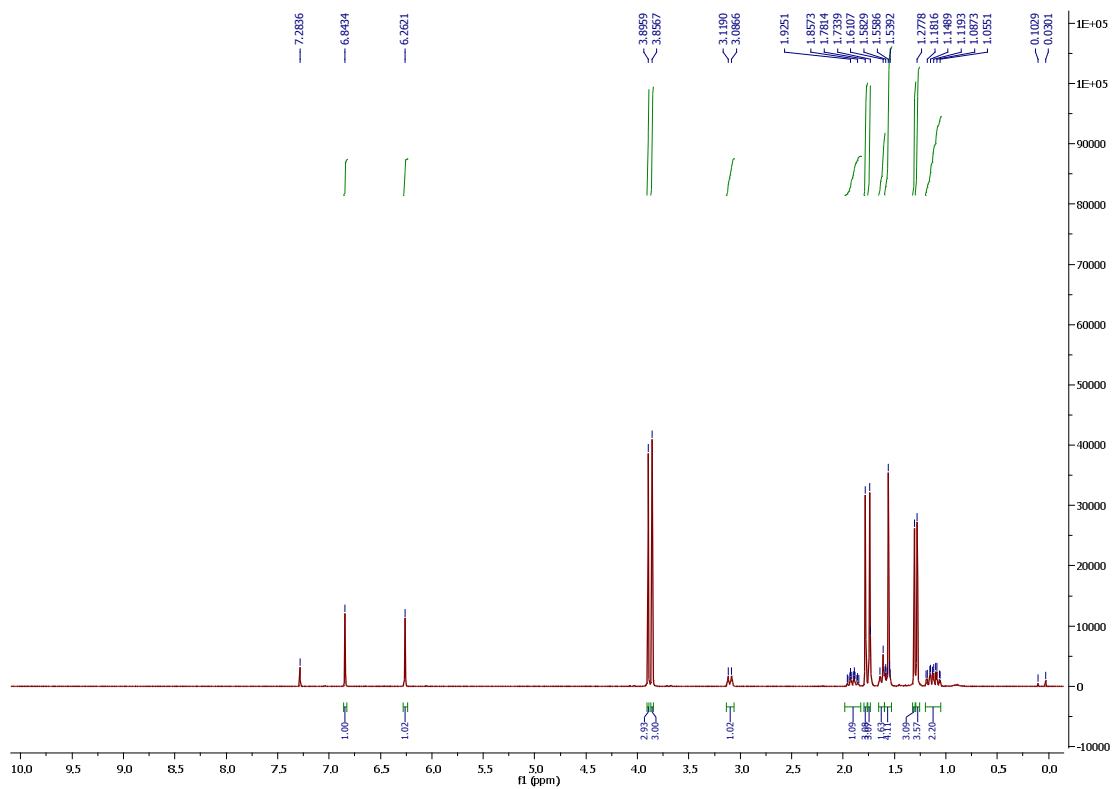
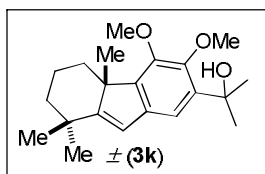
Analysis Name	D:\Data\user data\may2012\12 may\Dr.A . Bisai- BK-2-266(l)_1-A,4_01_2191.d	Acquisition Date	5/12/2012 3:47:23 PM
Method	HRLCMS-20 Sept.m	Operator	Meena Sharma
Sample Name	Dr.A . Bisai- BK-2-266(l)	Instrument	micrOTOF-Q II 10330
Comment			

### Acquisition Parameter

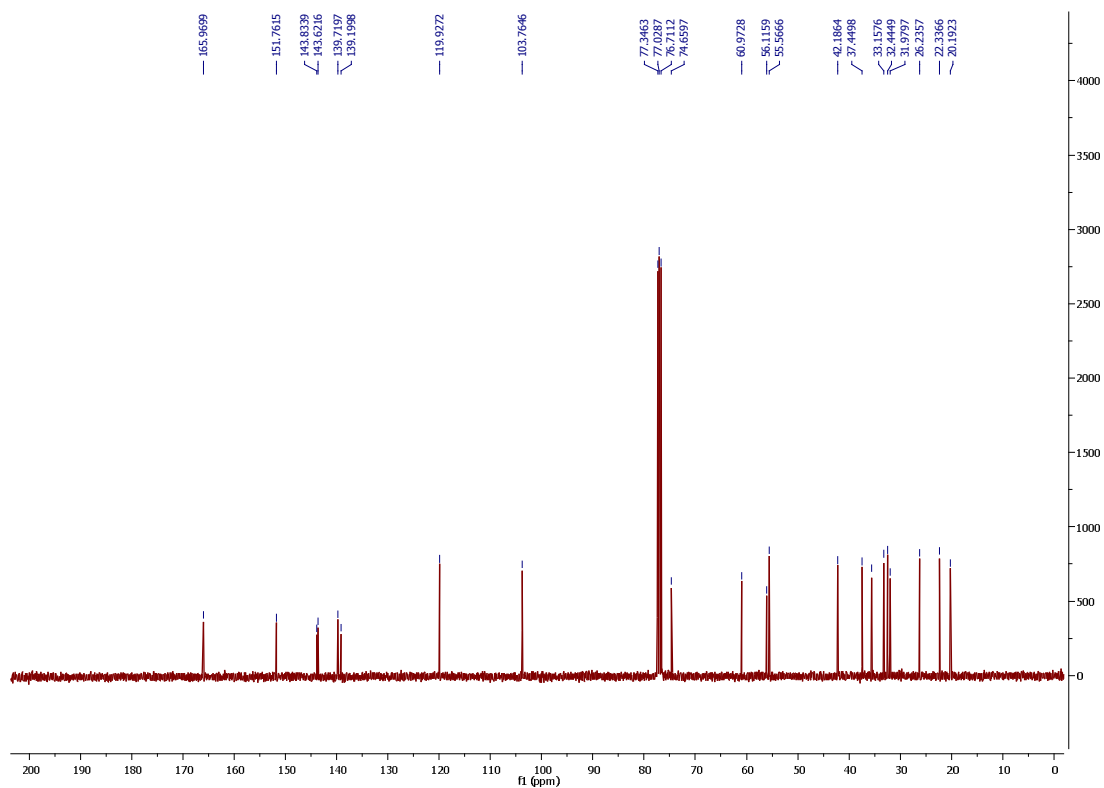
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



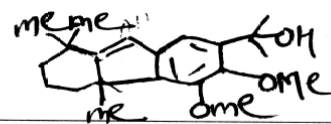
Scanned copy of mass spectrum (HRMS) of  $\pm(8)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3k)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\mathbf{k})$



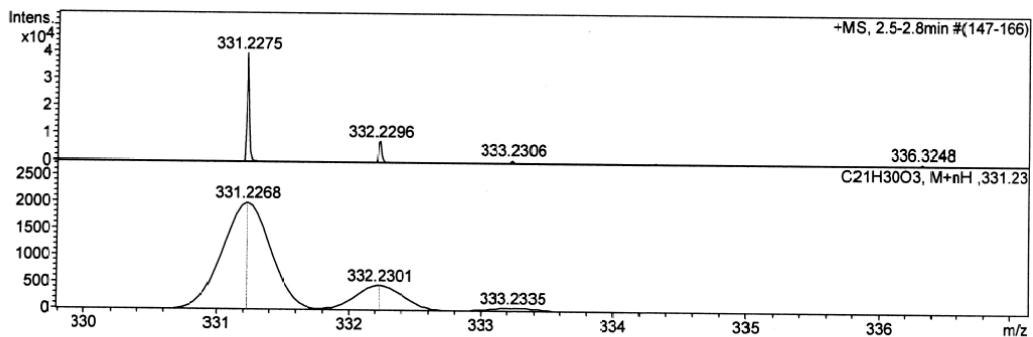
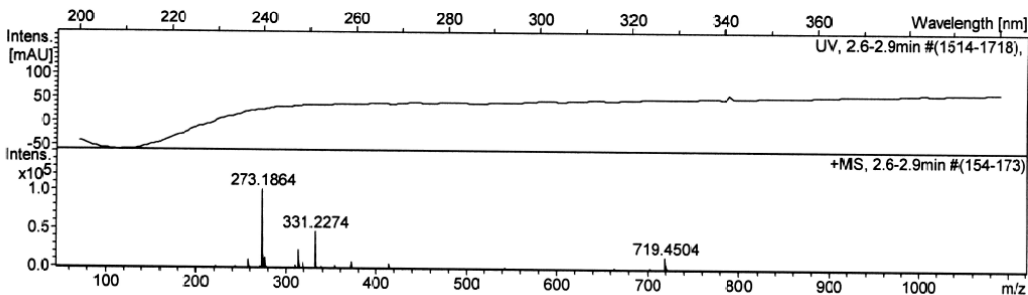
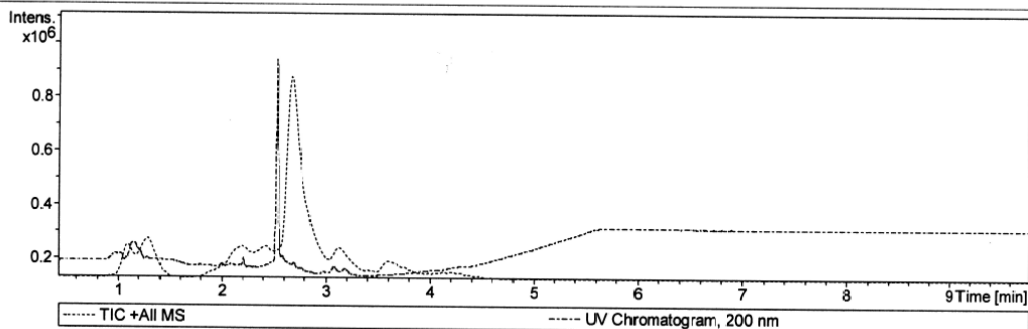
## Display Report

### Analysis Info

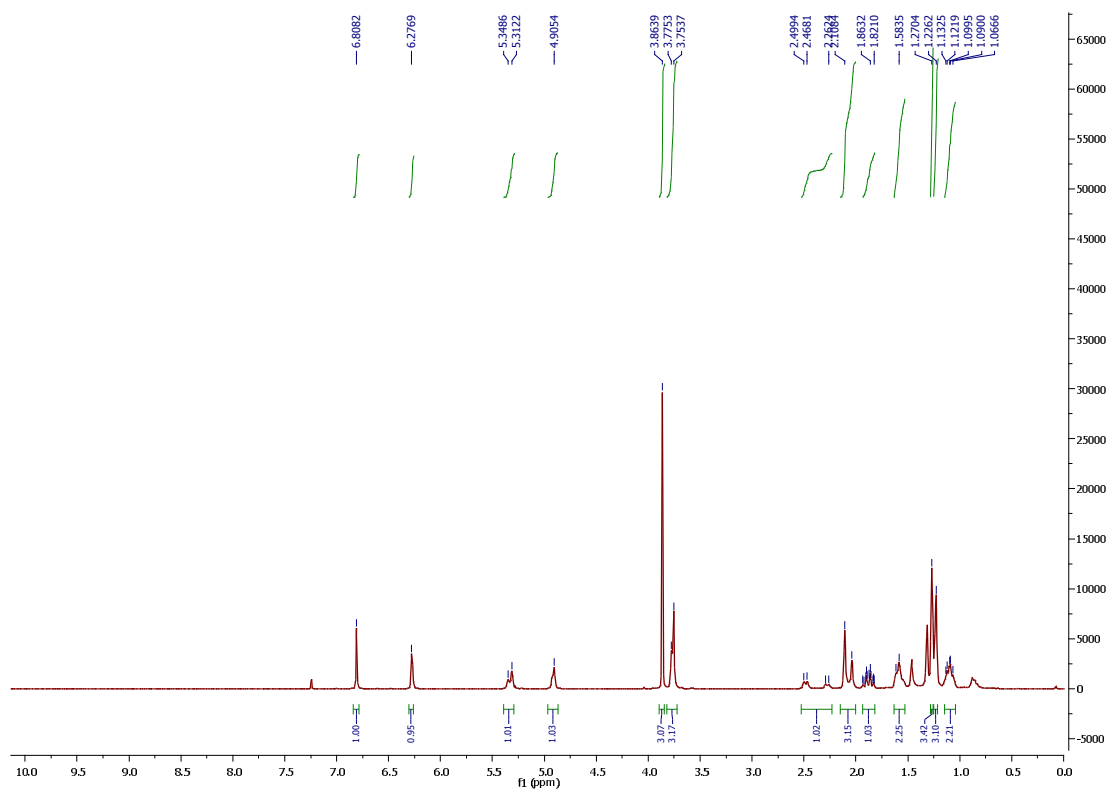
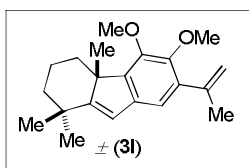
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Method HRLCMS-20 Sept.m  
Sample Name Dr. A. Bisai- BK-2-344(II) BF  
Comment  
Acquisition Date 7/19/2012 3:27:23 PM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

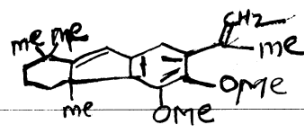


Scanned copy of mass spectrum (HRMS) of  $\pm(3k)$



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound **31**





## Display Report

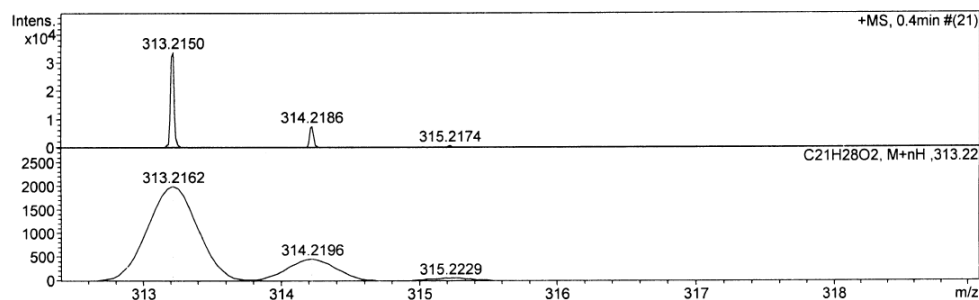
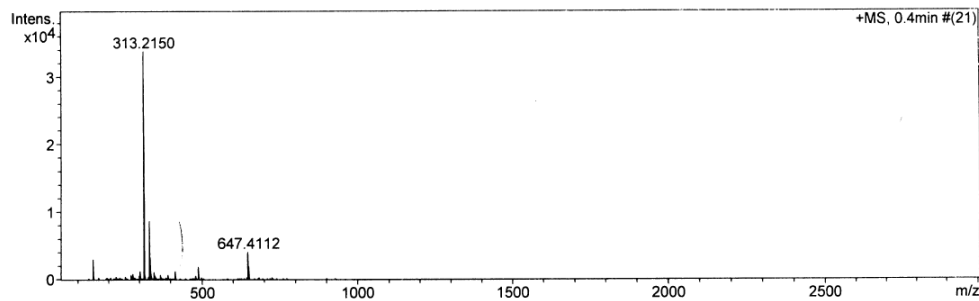
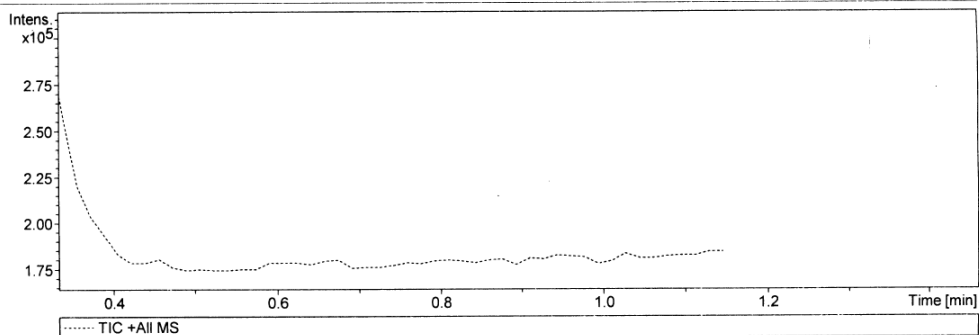
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Method HRLCMS-20 Sept.m  
Sample Name AB-BK-2-296  
Comment

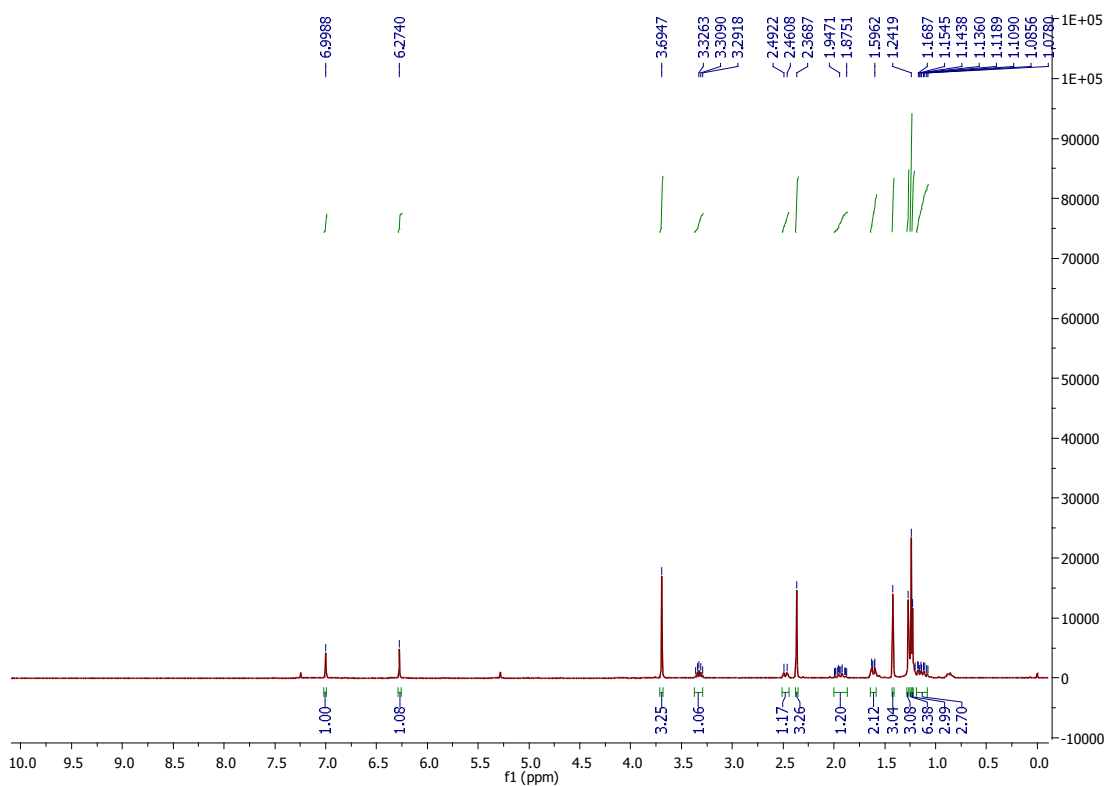
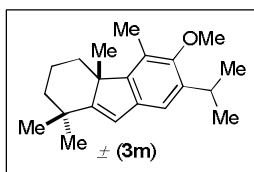
Acquisition Date 5/28/2012 10:28:40 AM  
Operator Meena Sharma  
Instrument micrOTOF-Q II 10330

### Acquisition Parameter

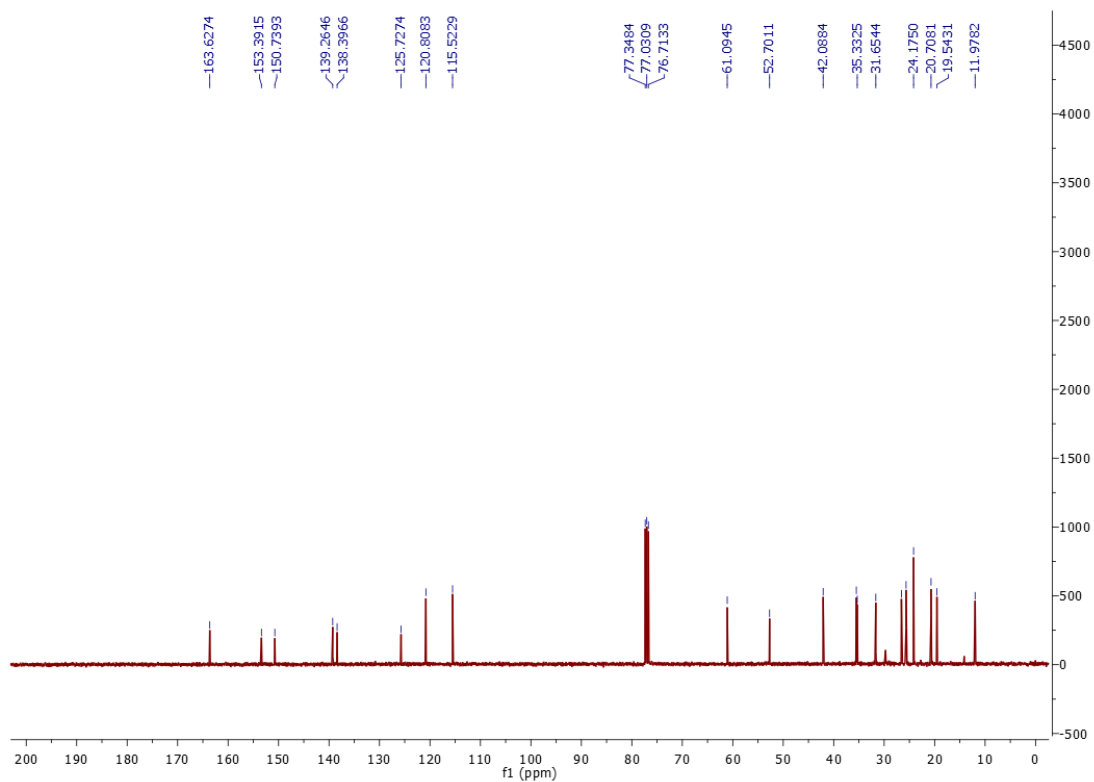
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



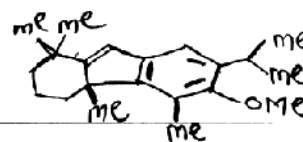
Scanned copy of mass spectrum (HRMS) of  $\pm(31)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3m)$



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\mathbf{m})$



## Display Report

### Analysis Info

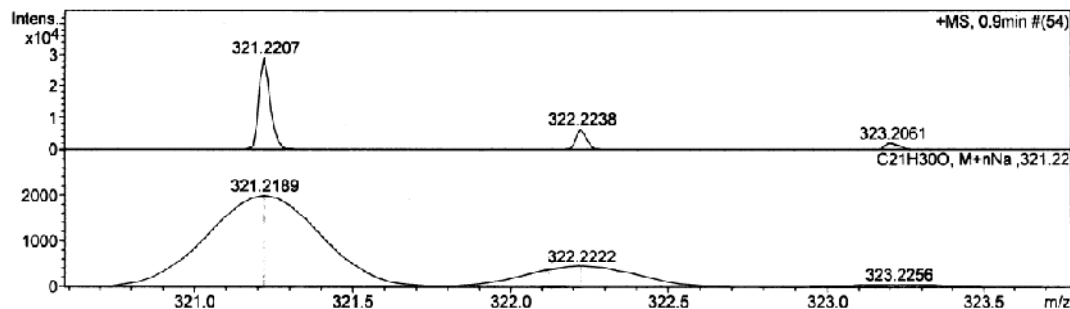
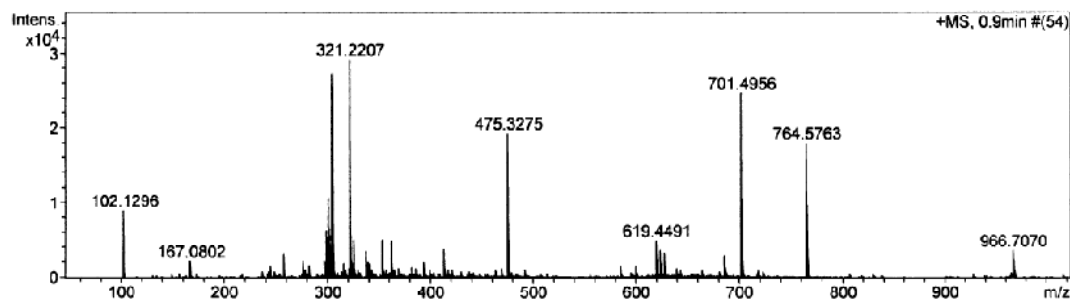
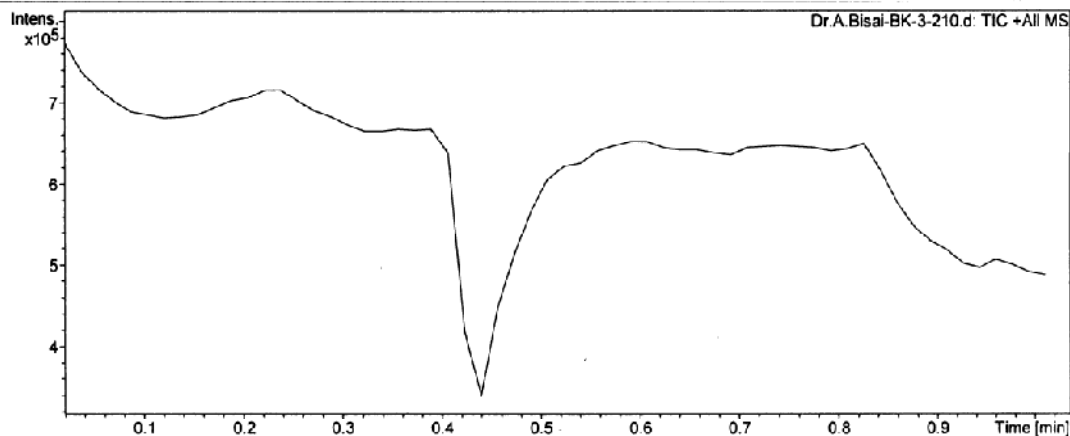
Analysis Name D:\Data\user data\2013\FEB\05 FEB\Dr.A.Bisai-BK-3-210.d  
Method tune\_low.m  
Sample Name BK-3-210  
Comment

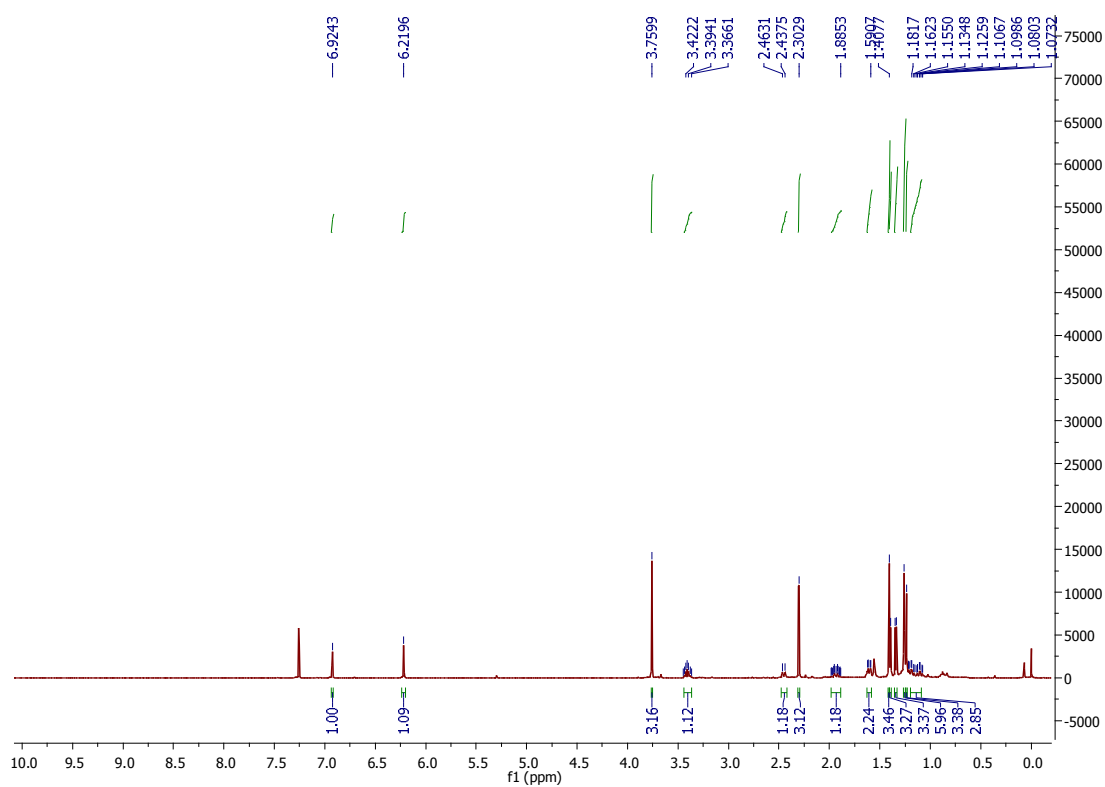
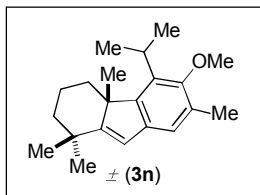
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Operator Amit  
Instrument micrOTOF-Q II 10330

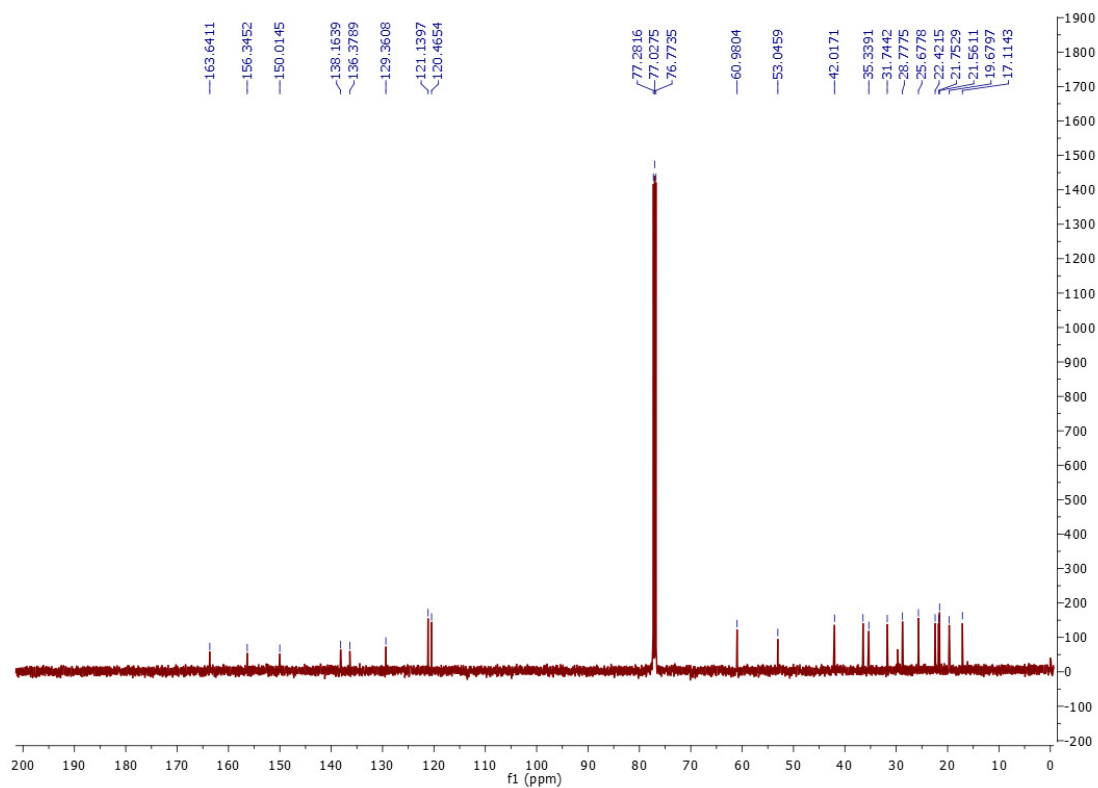
### Acquisition Parameter

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Focus	Not active	Set Capillary	4600 V	Set Dry Heater	180 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

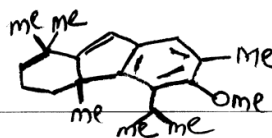




$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ) compound  $\pm$ (**3n**)



$^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\mathbf{n})$



## Display Report

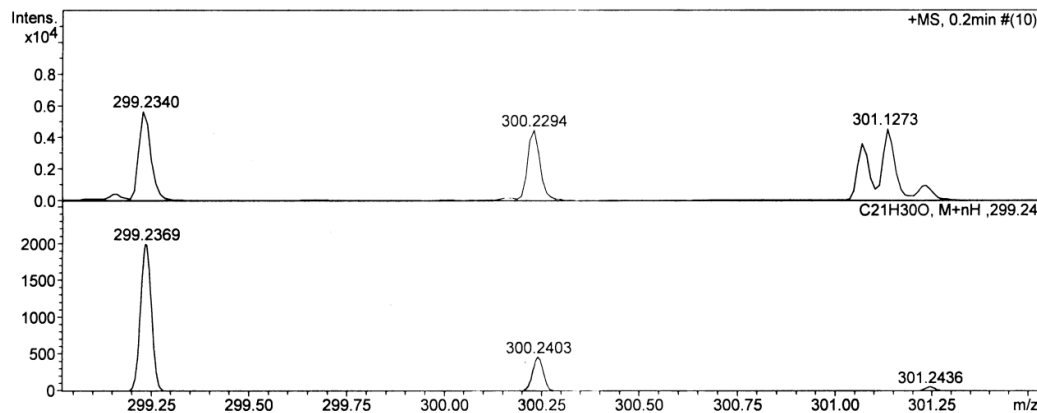
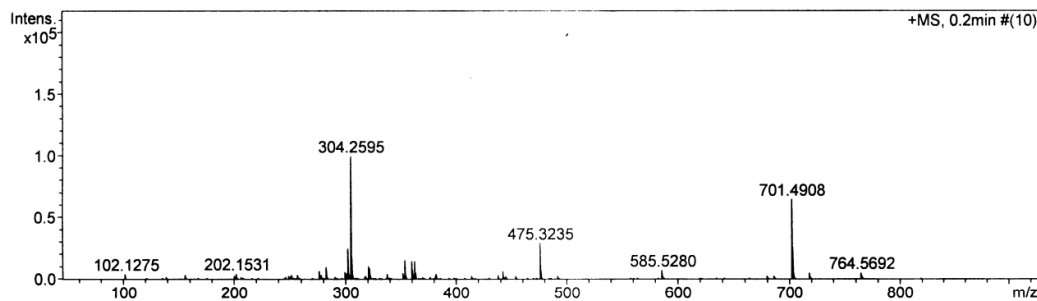
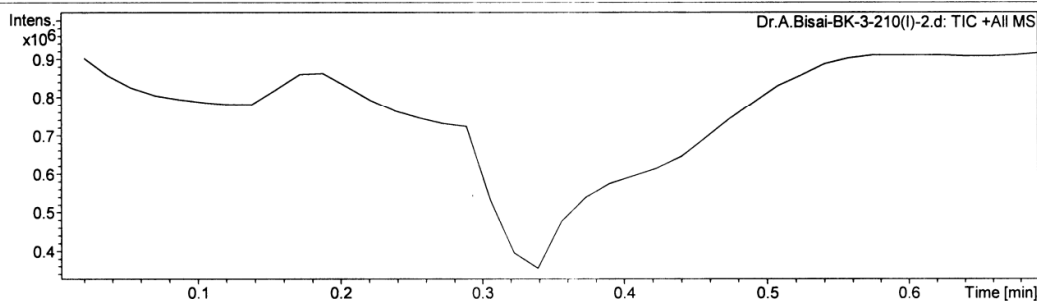
### Analysis Info

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Method tune\_low.m  
Sample Name BK-3-210(I)  
Comment

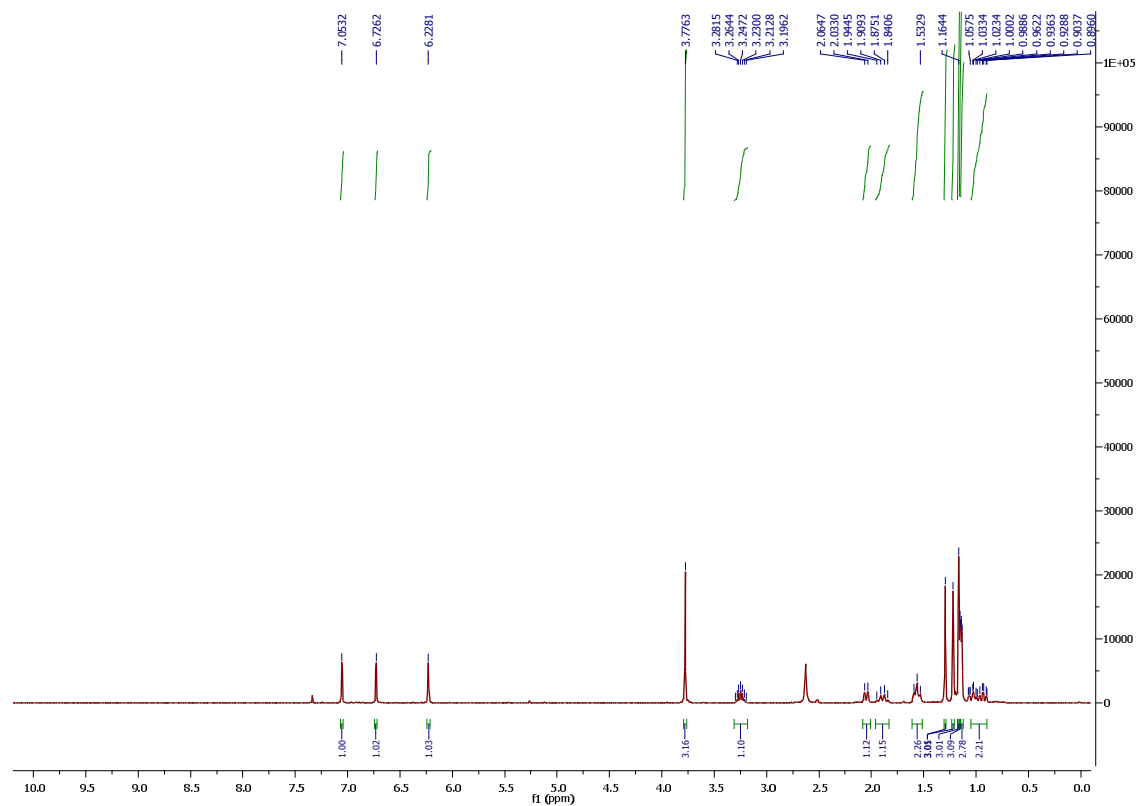
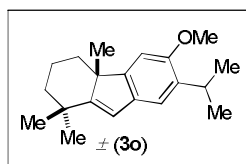
Acquisition Date 2/15/2013 4:03:47 PM  
Operator Amit  
Instrument micrOTOF-Q II 10330

### Acquisition Parameter

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

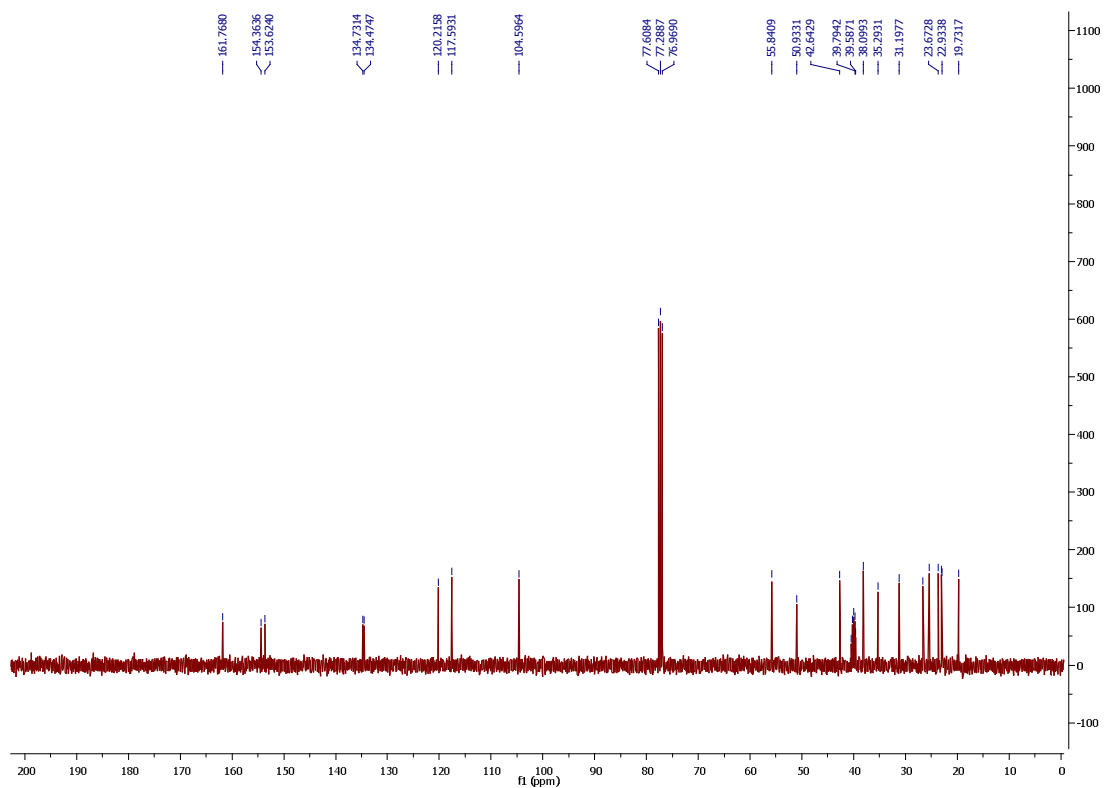


Scanned copy of mass spectrum (HRMS) of  $\pm(3n)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3 + \text{DMSO-D}_6$ ) compound  $\pm(3o)$





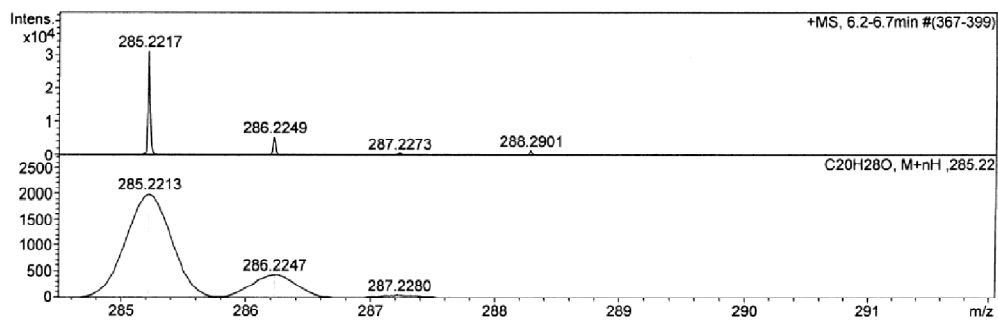
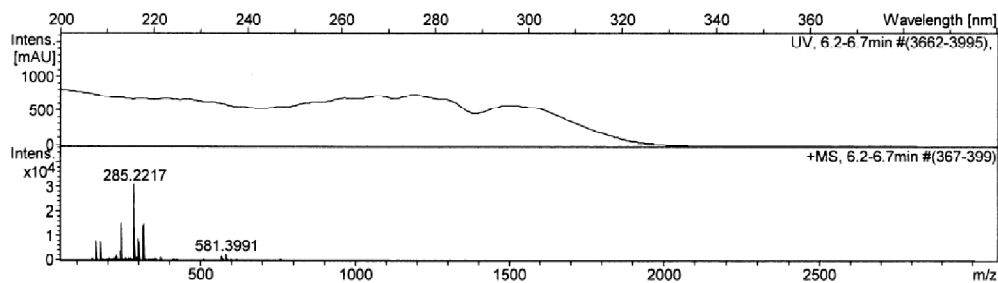
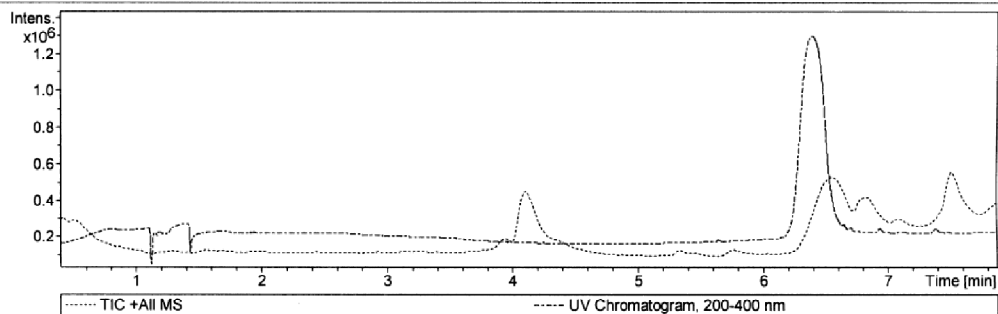
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3+\text{DMSO-D}_6$ ) compound  $\pm(30)$



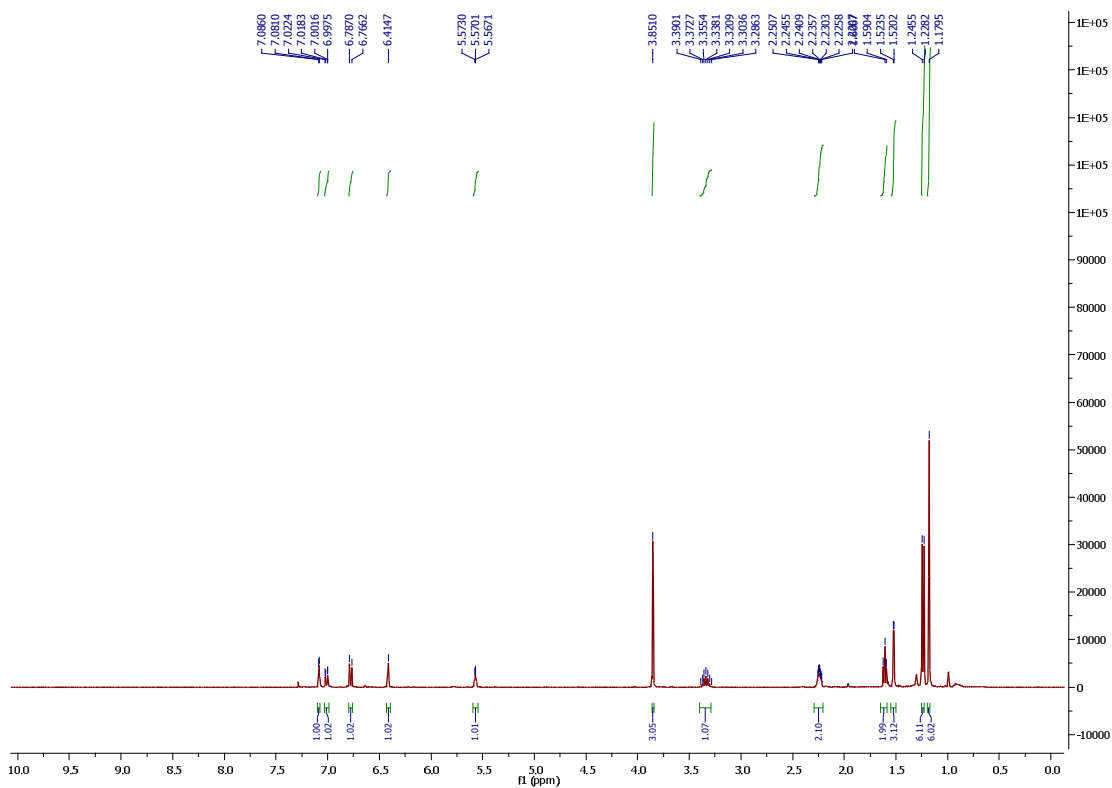
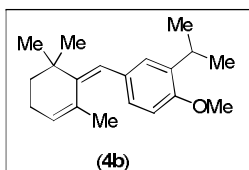
### Display Report

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Method: HRLCMS-20 Sept.m  
Sample Name: Dr. A. Bisai-BK-3-16 BF  
Comment:  
Acquisition Date: 7/24/2012 1:20:55 PM  
Operator: Meena Sharma  
Instrument: microTOF-Q II 10330

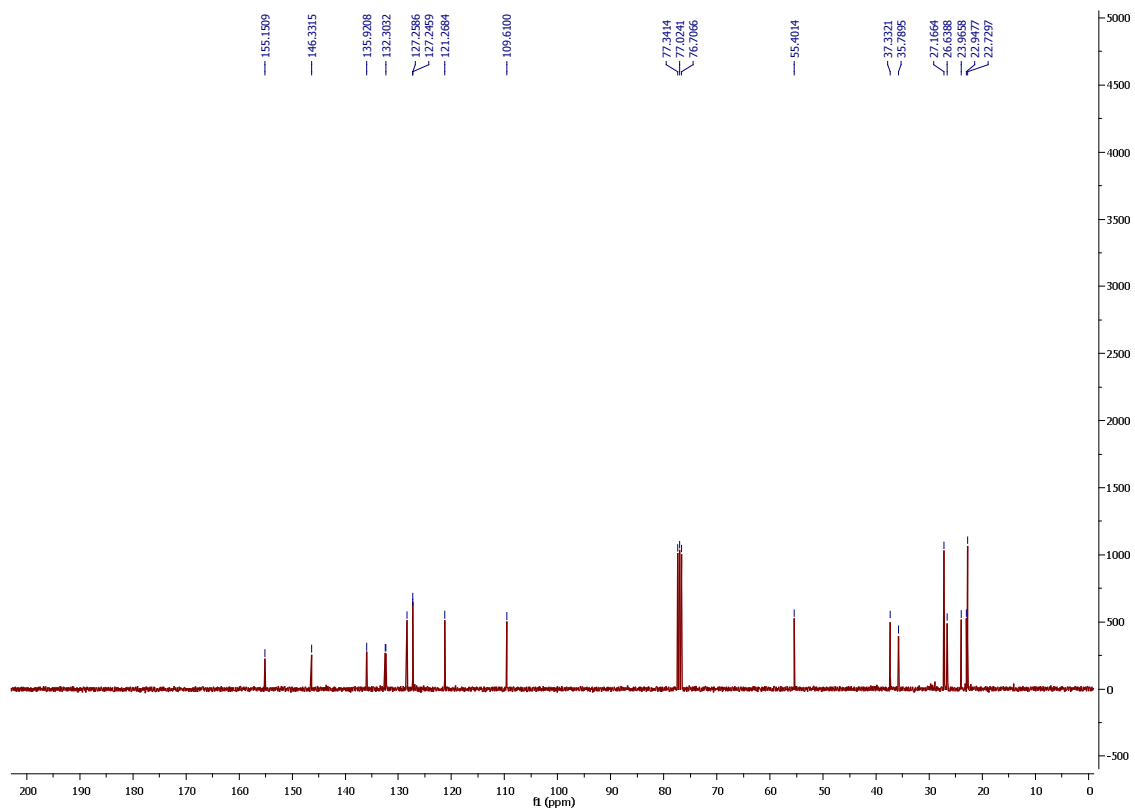
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Set Capillary: 4500 V  
Set End Plate Offset: -500 V  
Set Collision Cell RF: 130.0 Vpp  
Set Nebulizer: 1.2 Bar  
Set Dry Heater: 200 °C  
Set Dry Gas: 7.0 l/min  
Set Divert Valve: Waste



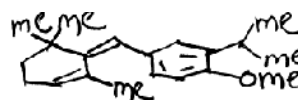
Scanned copy of mass spectrum (HRMS) of  $\pm(30)$



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound (4b)



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound (**4b**)



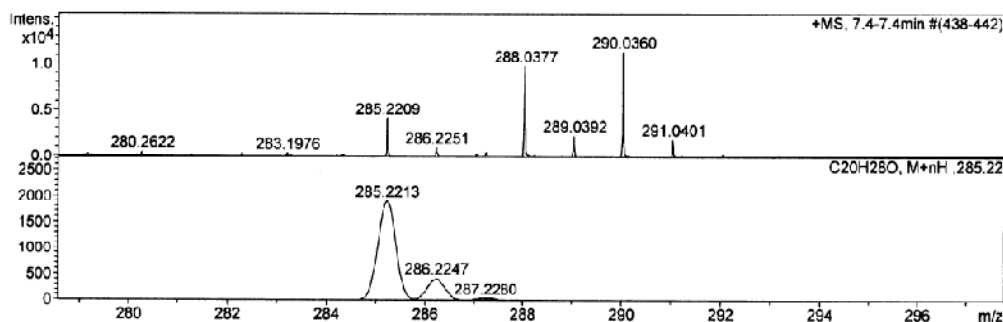
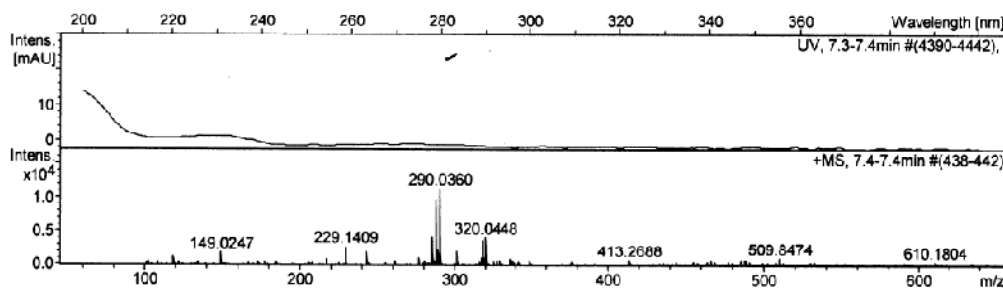
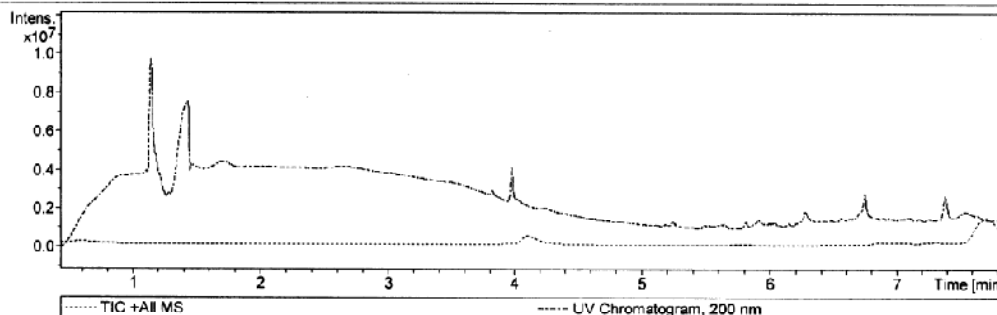
## Display Report

### Analysis Info

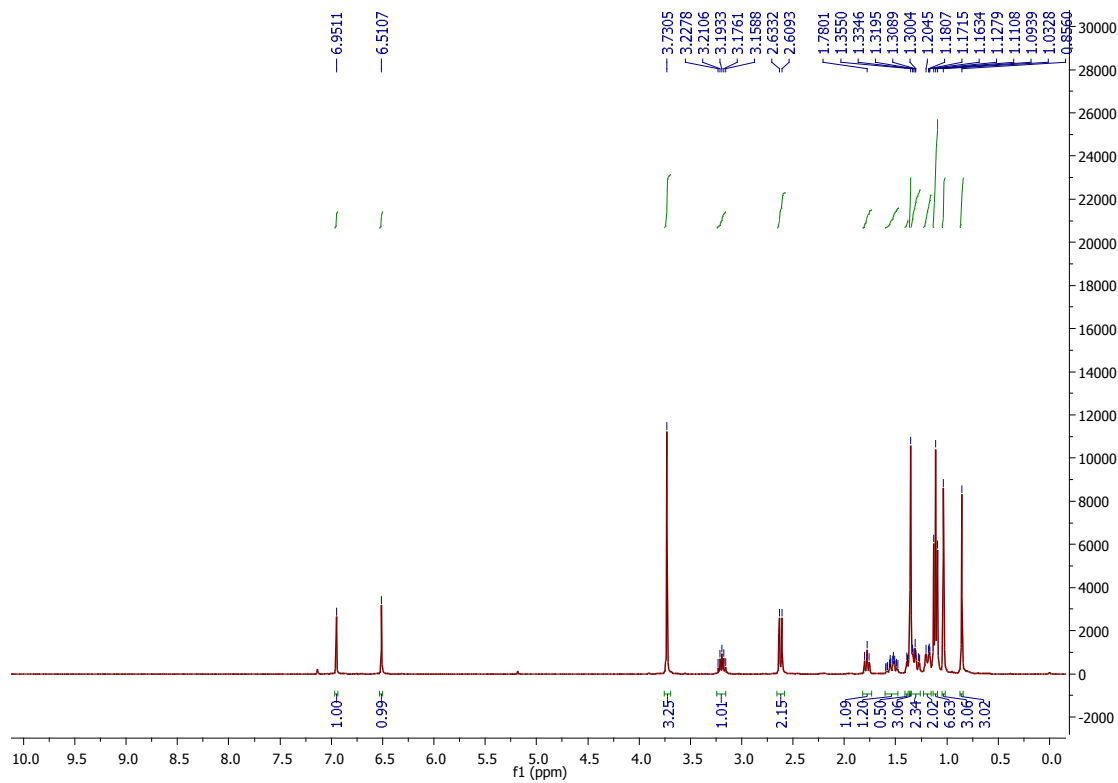
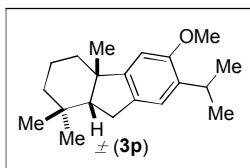
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Method	HRLCMS-20 Sept.m	Operator	Meena Sharma
Sample Name	Dr. A. Bisai-BK-3-17	Instrument	micrOTOF-Q II 10330
Comment			

### Acquisition Parameter

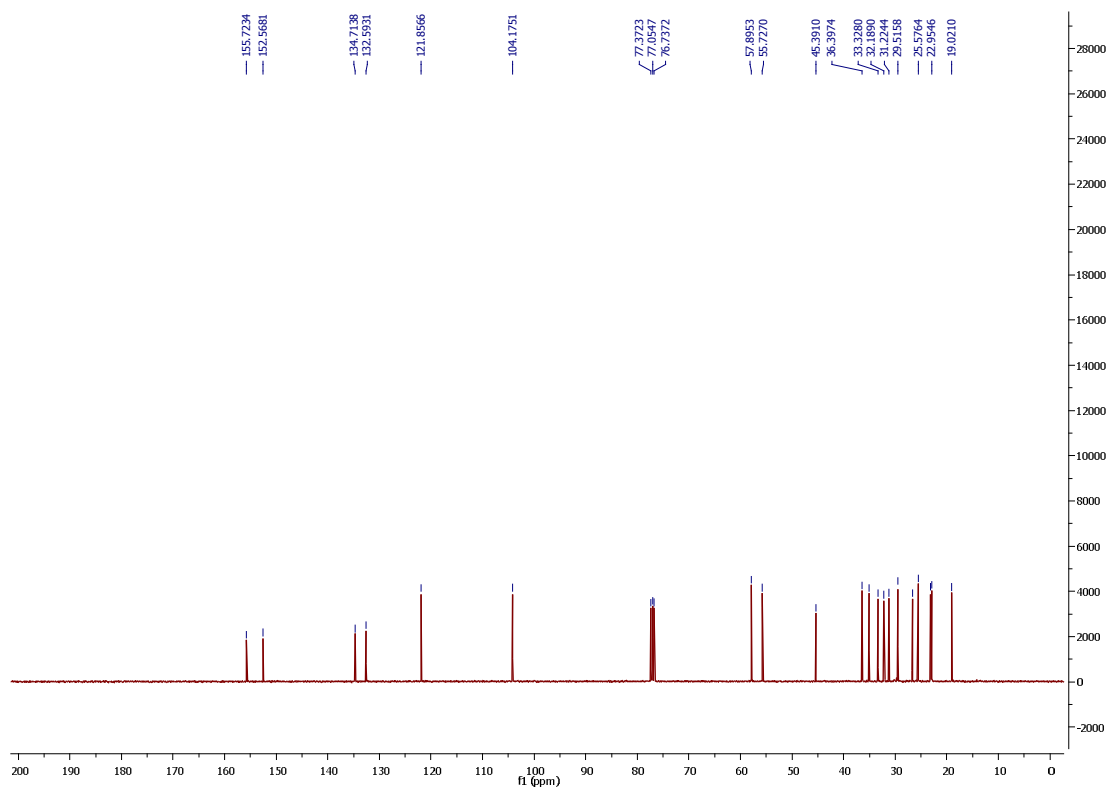
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



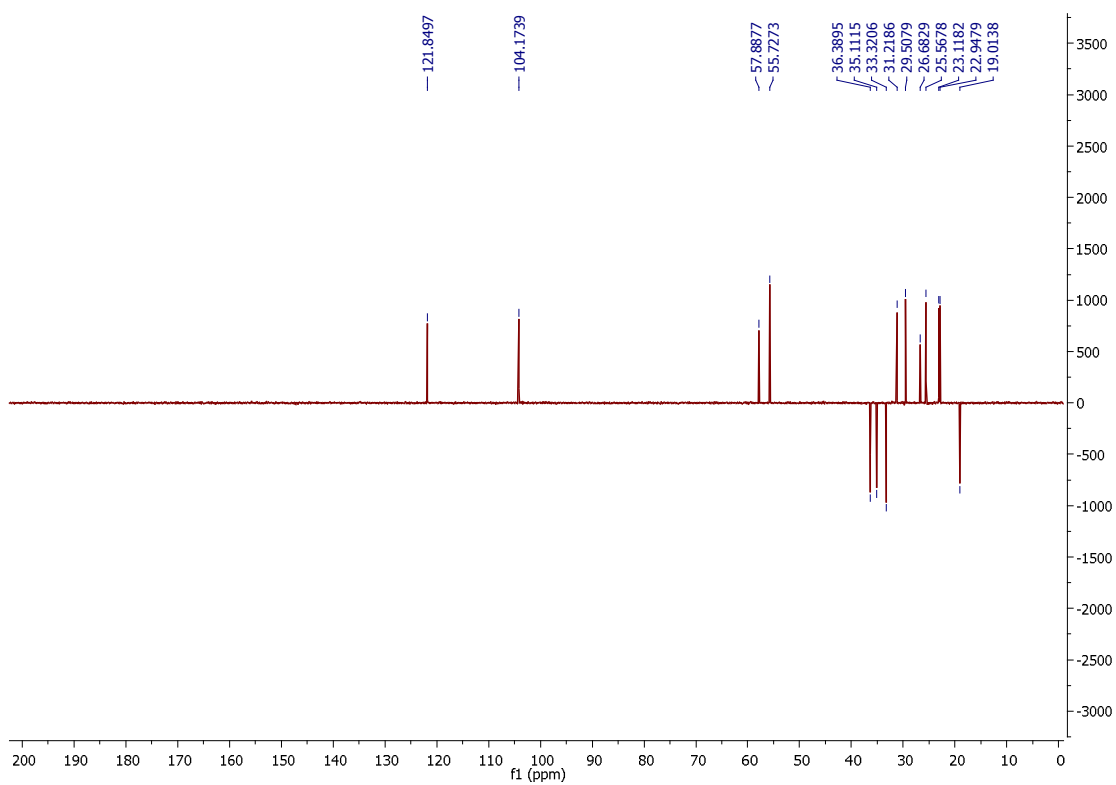
Scanned copy of mass spectrum (HRMS) of (4b)



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) compound **3p**

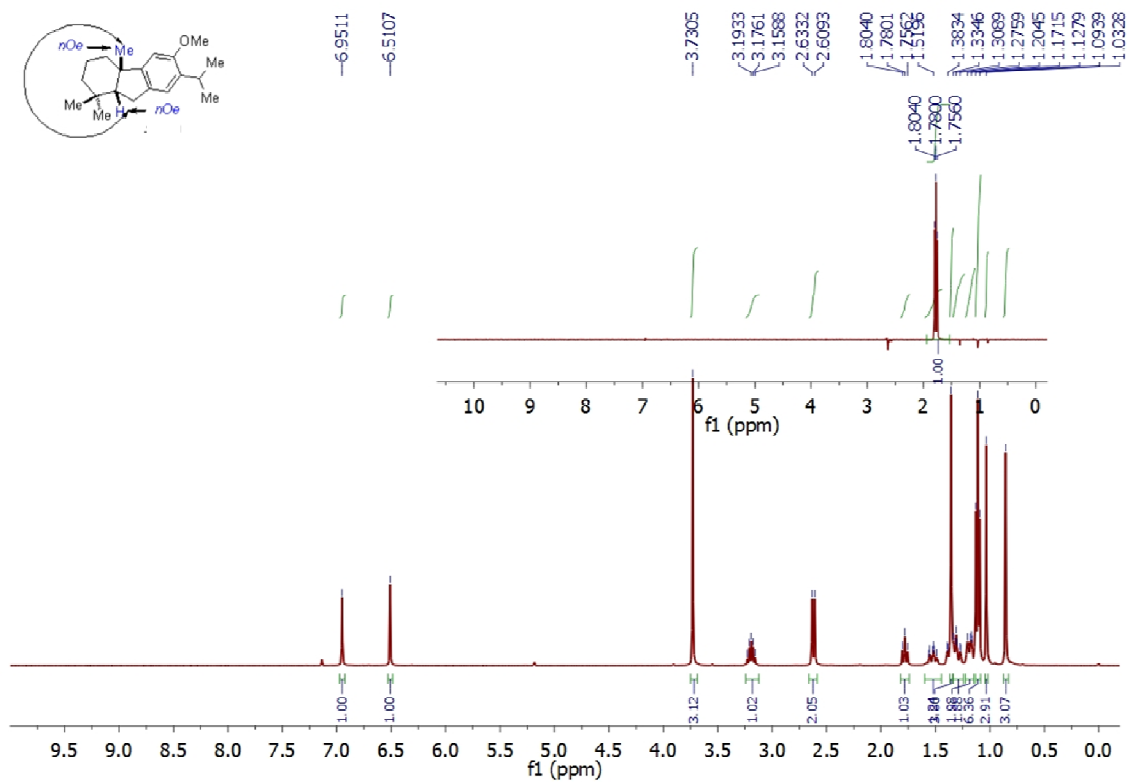


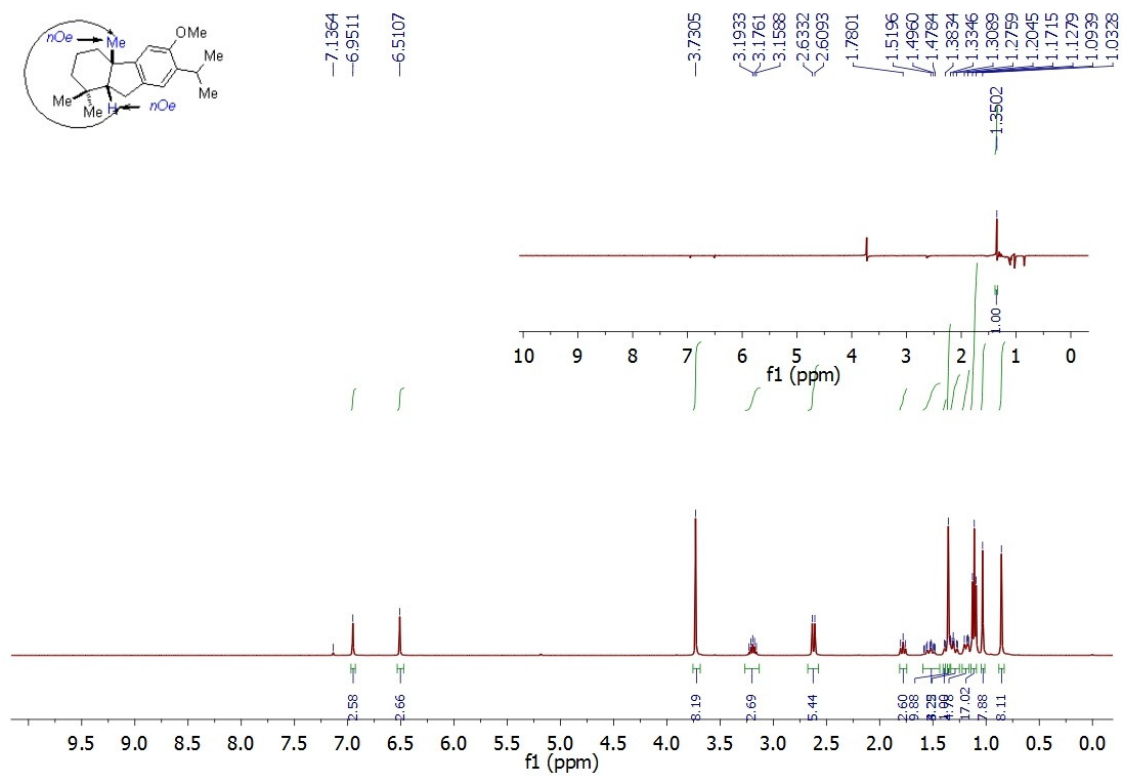
$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm(3\text{p})$



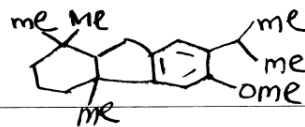
$^{13}\text{C}(\text{DEPT-135})$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound **±(3p)**







<sup>1</sup>H NMR (*nOe*) (400 MHz, CDCl<sub>3</sub>) compound **±(3p)**



### Display Report

#### Analysis Info

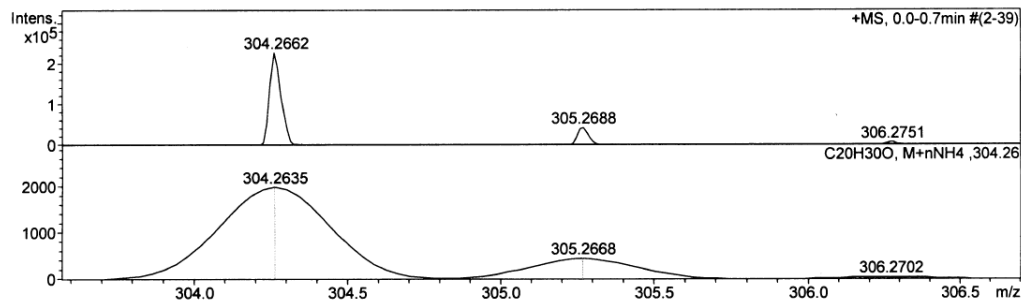
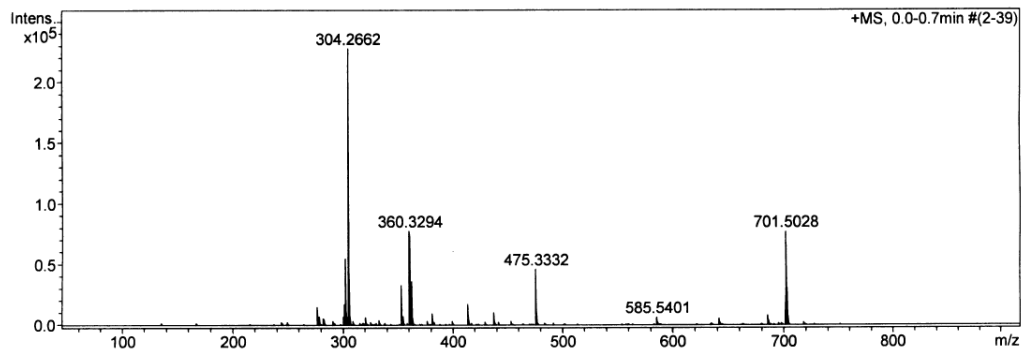
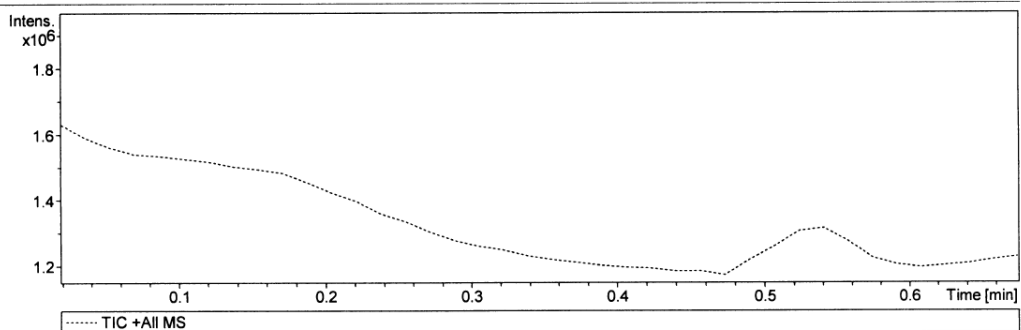
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Sample Name BK-3-194  
Comment

Acquisition Date 1/18/2013 11:38:03 AM

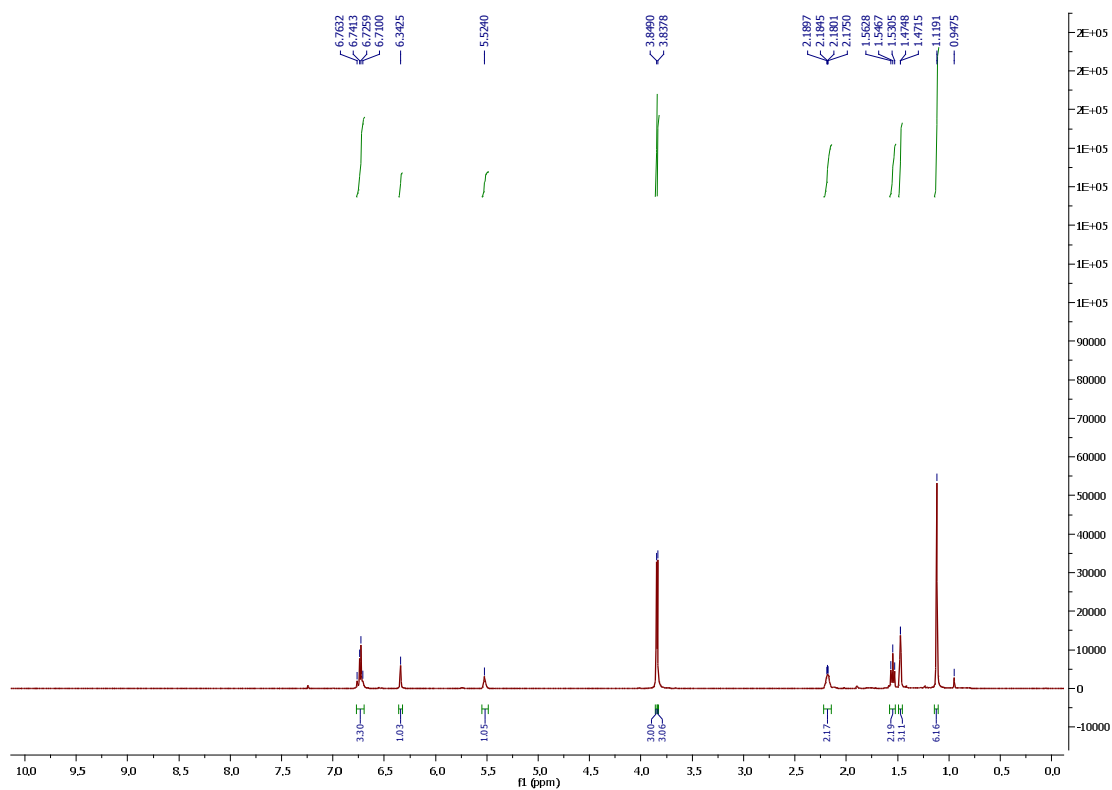
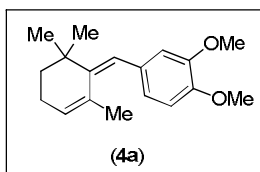
Operator IISERB  
Instrument micrOTOF-Q II 10330

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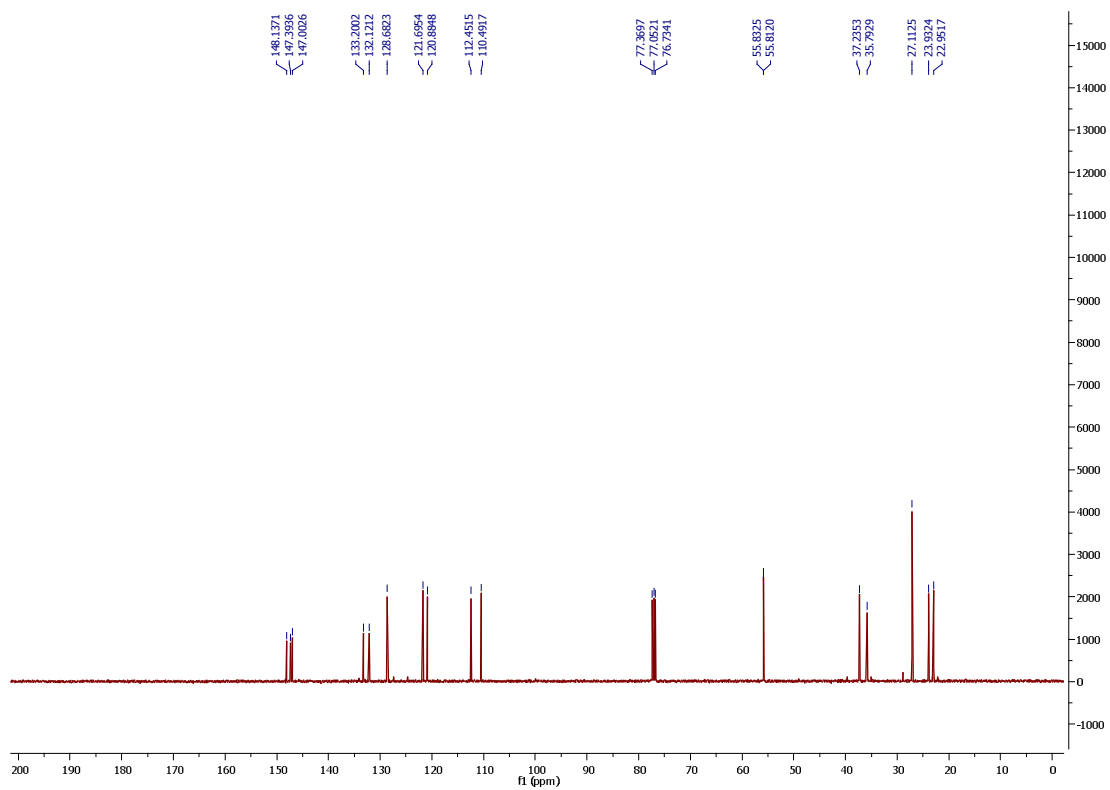
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum (HRMS) of  $\pm(3p)$



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound (4a)

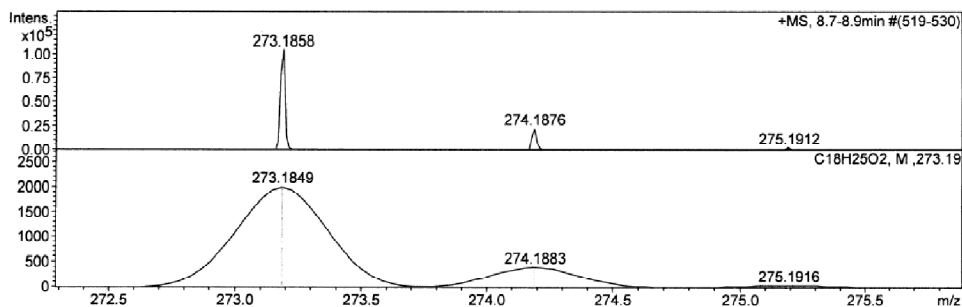
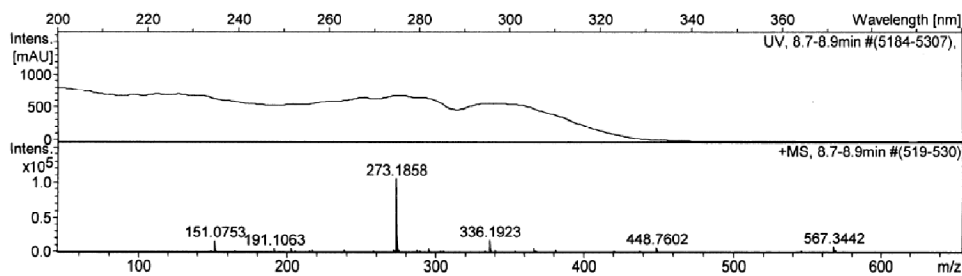
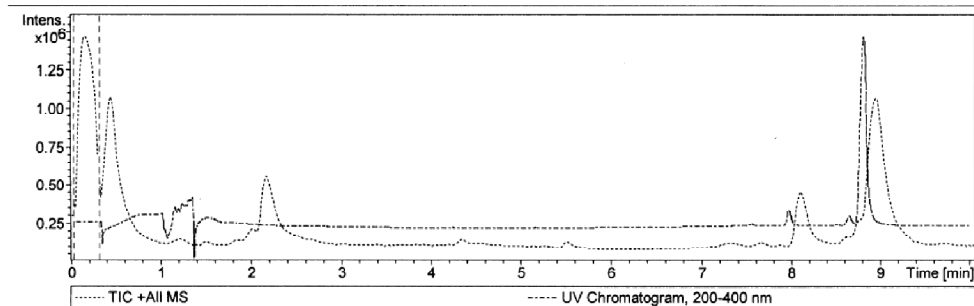




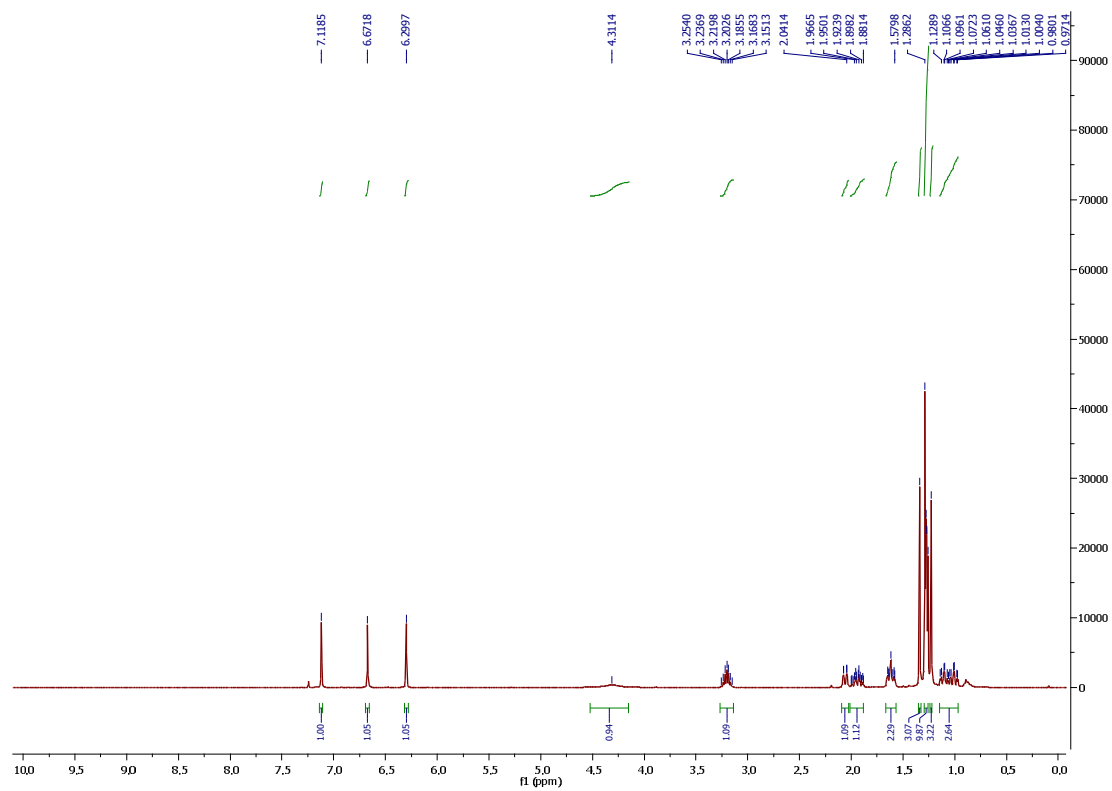
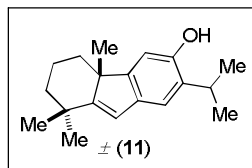
## Display Report

<b>Analysis Info</b>		Acquisition Date	7/23/2012 11:48:15 AM
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Method	HRLCMS-20 Sept.m	Instrument	microTOF-Q II 10330
Sample Name	Dr.A.Bisai-BK-2-215		
Comment			

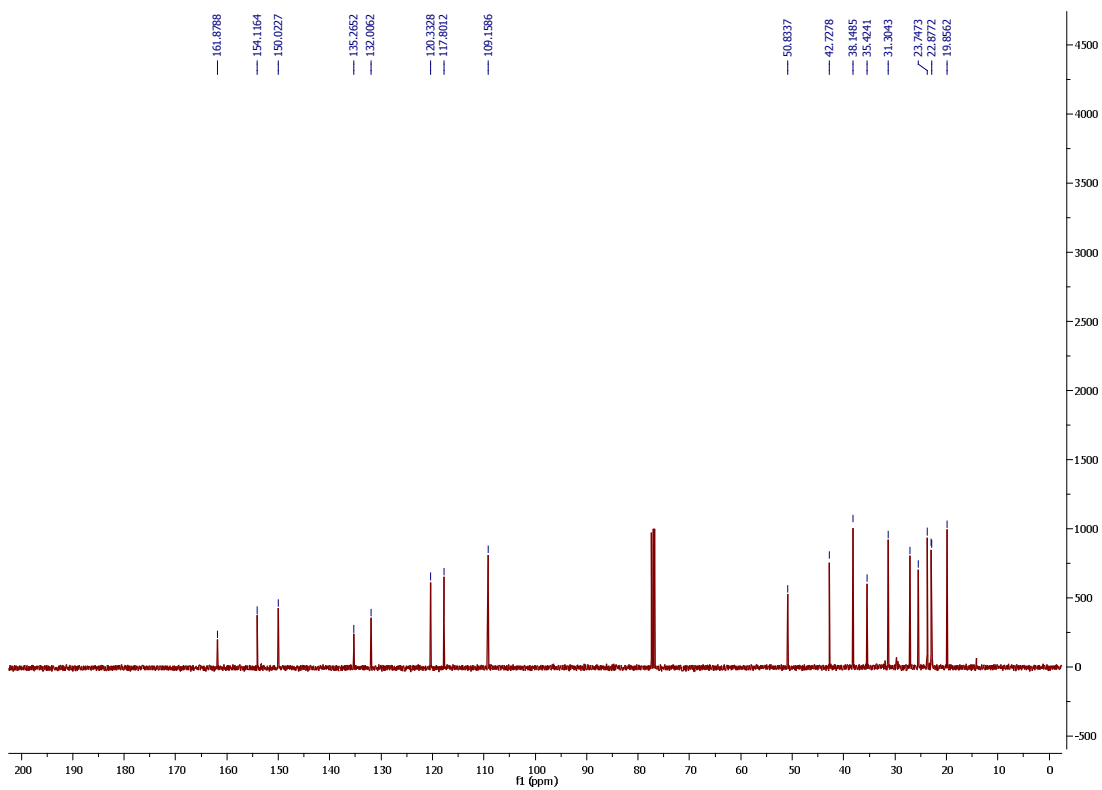
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum (HRMS) of (4a)

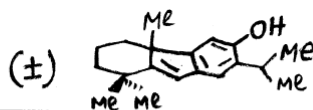


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) compound **11**



$^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ) compound  $\pm$ (11)





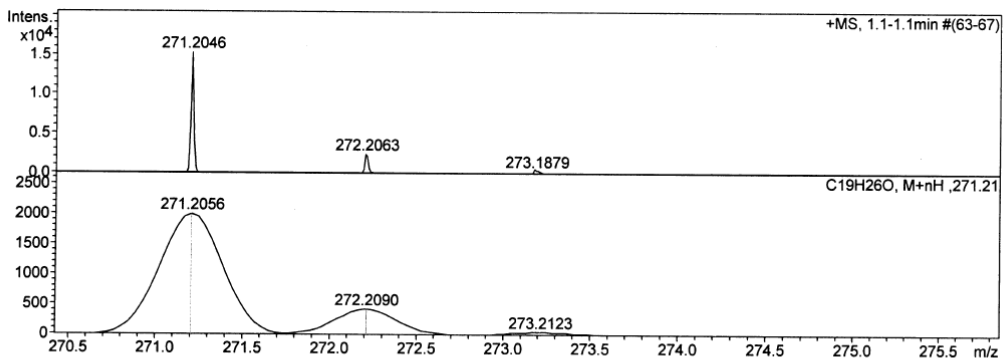
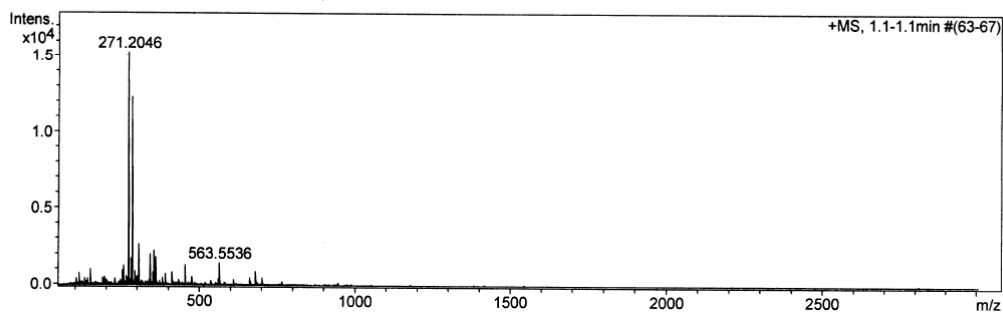
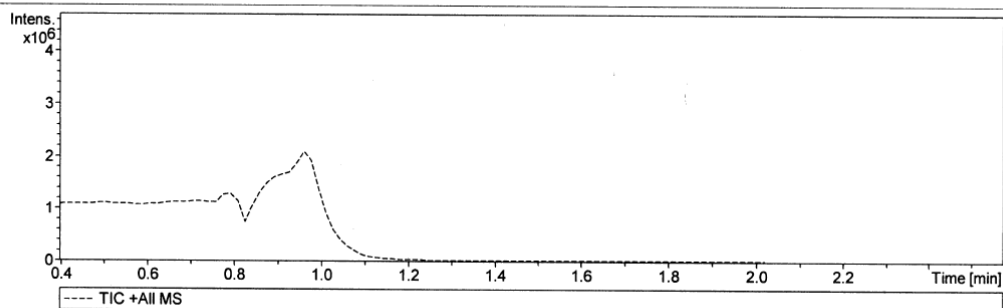
### Display Report

#### Analysis Info

Analysis Name	D:\Data\user data\NOV 2012\08 NOV\Dr. A. Bisai- BK-3-136 pos.d	Acquisition Date	11/9/2012 3:52:59 PM
Method	tune_low.m	Operator	Meena Sharma
Sample Name	BK-3-136	Instrument	micrOTOF-Q II 10330
Comment			

#### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	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	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum (HRMS) of ±(11)

## checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. [CIF dictionary](#)  
Please wait while processing .... [Interpreting this report](#)

## Datablock: Badrinath

Bond precision: C-C = 0.0035 Å Wavelength=0.71073  
Cell: a=6.8754 (3) b=10.7407 (5) c=11.7991 (6)  
alpha=91.303 (3) beta=92.041 (3) gamma=99.150 (3)  
Temperature: 298 K

	Calculated	Reported
Volume	859.97 (7)	859.97 (7)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C20 H28 O	?
Sum formula	C20 H28 O	C20 H28 O
Mr	284.42	284.42
Dx, g cm <sup>-3</sup>	1.098	1.098
Z	2	2
Mu (mm <sup>-1</sup> )	0.065	0.065
F000	312.0	312.0
F000'	312.12	
h, k, lmax	8, 12, 14	8, 12, 14
Nref	3158	3146
Tmin, Tmax	0.992, 0.994	0.987, 0.993
Tmin'	0.987	

Correction method= MULTI-SCAN  
Data completeness= 0.996 Theta (max)= 25.350  
R (reflections)= 0.0724 ( 2319) wR2 (reflections)= 0.2336 ( 3146)  
S = 1.041 Npar= 196

The following ALERTS were generated. Each ALERT has the format  
**test-name\_ALERT\_alert-type\_alert-level.**  
Click on the hyperlinks for more details of the test.

### Alert level B

[DIFMX01\\_ALERT\\_2\\_B](#) The maximum difference density is > 0.1\*ZMAX\*1.00  
\_refine\_diff\_density\_max given = 1.040  
Test value = 0.800  
[PLAT097\\_ALERT\\_2\\_B](#) Large Reported Max. (Positive) Residual Density 1.04 eA-3  
[PLAT412\\_ALERT\\_2\\_B](#) Short Intra XH3 .. XHn H18C .. H19B .. 1.78 Ang.

### Alert level C

[DIFMX02\\_ALERT\\_1\\_C](#) The maximum difference density is > 0.1\*ZMAX\*0.75  
The relevant atom site should be identified.  
[PLAT094\\_ALERT\\_2\\_C](#) Ratio of Maximum / Minimum Residual Density .... 3.10  
[PLAT230\\_ALERT\\_2\\_C](#) Hirshfeld Test Diff for C16 -- C17 .. 6.0 su  
[PLAT241\\_ALERT\\_2\\_C](#) Check High Ueq as Compared to Neighbors for C16  
[PLAT242\\_ALERT\\_2\\_C](#) Check Low Ueq as Compared to Neighbors for C2

### Alert level G

[PLAT072\\_ALERT\\_2\\_G](#) SHELXL First Parameter in WGHT Unusually Large. 0.13  
[PLAT154\\_ALERT\\_1\\_G](#) The su's on the Cell Angles are Equal ..... 0.00300 Deg.  
[PLAT194\\_ALERT\\_1\\_G](#) Missing \_cell\_measurement\_reflns\_used datum .... ?  
[PLAT793\\_ALERT\\_4\\_G](#) The Model has Chirality at C14 (Verify) .... S

- 0 ALERT level A = Most likely a serious problem - resolve or explain
  - 3 ALERT level B = A potentially serious problem, consider carefully
  - 5 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  - 4 ALERT level G = General information/check it is not something unexpected
- 3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data  
8 ALERT type 2 Indicator that the structure model may be wrong or deficient  
0 ALERT type 3 Indicator that the structure quality may be low  
1 ALERT type 4 Improvement, methodology, query or suggestion  
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories.  
Often the minor alerts point to easily fixed oversights, errors and omissions in your  
CIF or refinement strategy, so attention to these fine details can be worthwhile. In  
order to resolve some of the more serious problems it may be necessary to carry out

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checkCIF/PLATON (standard)

additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special\_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

#### Publication of your CIF in IUCr journals

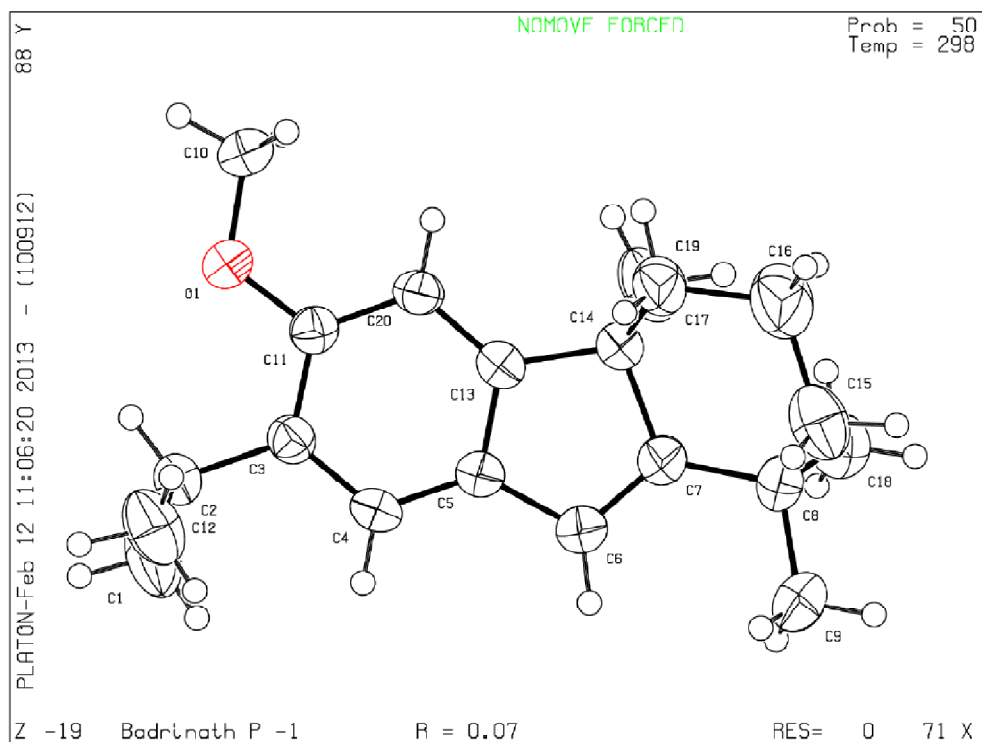
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

#### Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 05/11/2012; check.def file version of 05/11/2012

### Datablock Badrinath - ellipsoid plot



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Download CIF editor (pubCIF) from the IUCr  
Download CIF editor (enCIFer) from the CCDC  
Test a new CIF entry

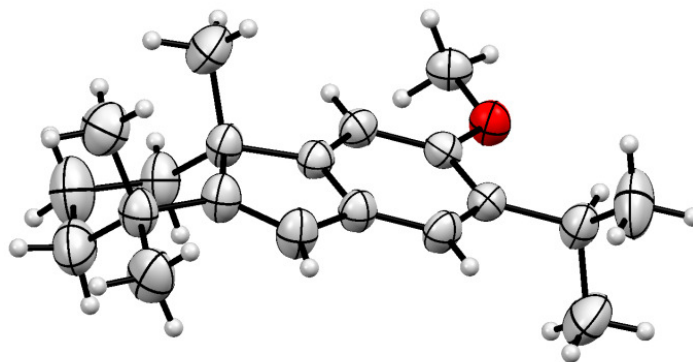


Figure: X-ray structure of carbotricycle  $\pm$ (**30**)

1/28/13

checkCIF/PLATON (standard)

## checkCIF/PLATON (standard)

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. Please wait while processing ....  
CIF dictionary Interpreting this report

## Datablock: shelxl

Bond precision: C-C = 0.0020 Å Wavelength=0.71073  
Cell: a=8.0057(15) b=19.331(4) c=9.6263(16)  
alpha=90 beta=95.807(11) gamma=90  
Temperature: 100 K

	Calculated	Reported
Volume	1482.1(5)	1482.1(5)
Space group	P 21/n	P 21/n
Hall group	-P 2yn	-P 2yn
Moiety formula	C10 H22 O3	?
Sum formula	C10 H22 O3	C10 H22 O3
Mr	286.36	286.36
Dx, g cm <sup>-3</sup>	1.293	1.293
Z	4	4
Mu (mm <sup>-1</sup> )	0.086	0.086
F000	616.0	616.0
F000'	616.30	
h, k, lmax	10, 24, 12	10, 24, 12
Nref	3150	3123
Tmin, Tmax	0.983, 0.983	0.983, 0.983
Tmin'	0.983	

Correction method= MULTI-SCAN  
Data completeness= 0.991 Theta(max)= 26.730  
R(reflections)= 0.0472( 2005) wR2(reflections)= 0.1254( 3123)  
s = 0.975 Npar= 193

The following ALERTS were generated. Each ALERT has the format  
**test-name\_ALERT\_alert-type\_alert-level**.  
Click on the hyperlinks for more details of the test.

### Alert level G

PLAT128\_ALERT\_4\_G Alternate Setting of Space-group P21/c ..... P21/n  
PLAT194\_ALERT\_1\_G Missing \_cell\_measurement\_refins\_used datum .... ?  
PLAT793\_ALERT\_4\_G The Model has Chirality at C5 (Verify) .... S

0 ALERT level A = Most likely a serious problem - resolve or explain  
0 ALERT level B = A potentially serious problem, consider carefully  
0 ALERT level C = Check. Ensure it is not caused by an omission or oversight  
3 ALERT level G = General information/check it is not something unexpected

1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data  
0 ALERT type 2 Indicator that the structure model may be wrong or deficient  
0 ALERT type 3 Indicator that the structure quality may be low  
2 ALERT type 4 Improvement, methodology, query or suggestion  
0 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special\_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

### Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

vm02b.iucr.org/cgi-bin/checkcif\_hkl.pl

1/2

1/29/13

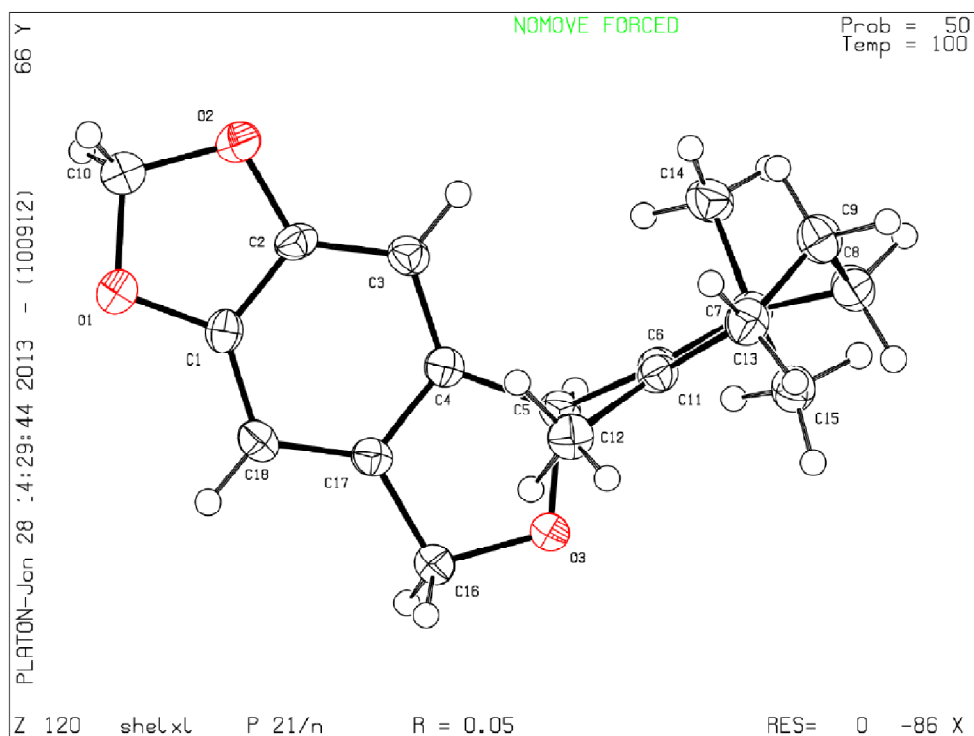
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**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 05/11/2012; check.def file version of 05/11/2012

**Datablock shelxl - ellipsoid plot**



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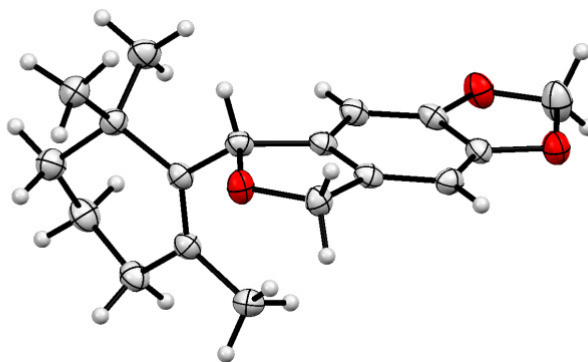


Figure: X-ray structure of  $\pm$ (**8**)