

Supporting Information

A Versatile Palladium Catalyst System for Suzuki-Miyaura Coupling of Alkenyl Tosylates and Mesylates

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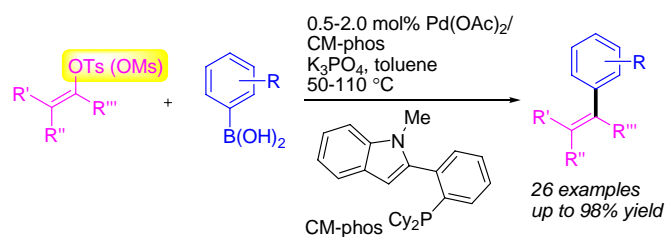


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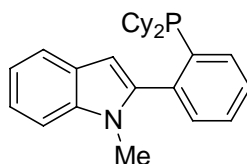
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1. General considerations

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All Suzuki reactions were performed in Rotaflor® (England) resealable screw cap Schlenk flask (approx. 20 mL volume) in the presence of Teflon coated magnetic stirrer bar (4 mm × 10 mm). *N,N*-Dimethylformamide (DMF) was distilled under calcium hydride under reduced pressure. Dioxane and isopropyl alcohol were distilled from sodium under nitrogen. *tert*-butanol was refluxing with sodium and the distillate was stored under CaH₂. It was distilled from calcium hydride under nitrogen prior to use.¹ New bottle of *n*-butyllithium was used (*Note*: since the concentration of *n*-BuLi from old bottle may vary, we recommend to perform a titration prior to use). K₂CO₃ and K₃PO₄ were purchased from Fluka. Thin layer chromatography was performed on Merck precoated silica gel 60 F₂₅₄ plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. ¹H NMR spectra were recorded on a Bruker (400 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in CDCl₃ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ¹³C NMR spectra were referenced to CDCl₃ (δ 77.0 ppm, the middle peak). Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Bruker APEX 47e FT-ICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP

6890 GC-FID system. Compounds described in the literatures were characterized by comparison of their ^1H , and/or ^{13}C NMR spectra to the previously reported data.

2. Preparation of indolyl phosphine ligand



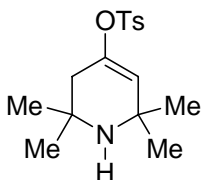
CM-phos

N-Methyl-2-(2'- Dicyclohexylphosphinophenyl)indole (CM-phos) was prepared according to the literature method.²

3. Preparation of alkenyl tosylates and mesylates substrates

4-(*p*-Toluenesulfonyloxy) coumarin, 1-methyl-4-(*p*-toluenesulfonyloxy)-2-quinolone, 6-Methyl-4-(*p*-toluenesulfonyloxy)-2-pyranone, 6-methyl-4-(*p*-toluenesulfonyloxy) coumarin, 6-methyl-4-(*p*-methanesulfonyloxy) coumarin, 4-(*p*-methanesulfonyloxy) coumarin, 7-methoxy-4-(*p*-toluenesulfonyloxy) coumarin, 1-methyl-4-(*p*-methanesulfonyloxy)-2-quinolone, 6-chloro-4-(*p*-toluenesulfonyloxy) coumarin were prepared from their corresponding precursors with TsCl or MsCl in the presence of triethylamine in CH_2Cl_2 according to the literature method without modifications.³

1,2-Dihydro-3-naphthyl tosylate, 2-Ethoxycarbonyl- 1-cyclohexenyl tosylate, 2,2-diphenyl-1-methylethenyl tosylate, 2-phenyl- 1-cyclohexenyl tosylate, 1,2-dihydro-3-methyl-4-naphthyl tosylate, and 2,2-diphenyl-1-methylethenyl mesylate were prepared from their corresponding species according to the literature method without modifications.⁴



1,2,3,6- Tetrahydro- 2,2,6,6-tetramethylpyridin-4-yl-4- tosylate

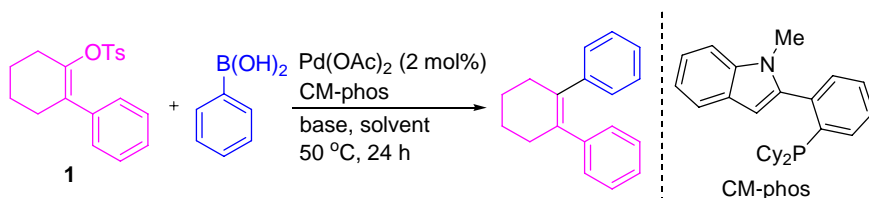
A round bottom flask was charged with 2,2,6,6-Tetramethyl-4-piperidone (40 mmol) and purged with nitrogen. NMP (80 ml) was added and was cooled to -15°C . Solid NaOtBu (44 mmol) was added and the solution was stirred at room temperature for 2h. The solution was cooled to -40°C and p-toluenesulfonic anhydride (44 mmol) was added in a single portion. The reaction mixture was stirred at -40°C to r.t. for overnight. The tan solution was combined MTBE (300ml) and washed with aq NaHCO_3 (400ml) and water. The organic phase was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product (65%). as tan oil that slowly solidified. M.p.: $43.4\text{-}47.4^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3) δ 1.07 (s, 6H), 1.09 (s, 6H), 1.98 (s, 2H), 2.43 (s, 3H), 5.18 (s, 1H), 7.32 (d, $J=7.2\text{Hz}$, 2H), 7.78 (d, $J=6.8\text{Hz}$, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.5, 29.9, 30.9, 40.2, 50.9, 51.4, 124.0, 128.3, 129.5, 133.0, 144.5, 144.9; HRMS: calcd. for $\text{C}_{16}\text{H}_{24}\text{N}_2\text{O}_3\text{SH}^+$: 310.1480, found 310.1477.

4. General procedures for reaction conditions screening and coupling reactions

General procedures for screening: $\text{Pd}(\text{OAc})_2$ (4.4 mg, 0.020 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for several times.

Precomplexation was applied by adding freshly distilled dichloromethane (1 mL) and Et₃N (100 μL) into the tube. The solution was stirred and warmed using a hair drier for about 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under a high vacuum. Alkenyl tosylate (1.0 mmol), phenylboronic acid (2.0 mmol) and base (3.0 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three cycles. The solvent (3.0 mL) was then added. The tube was stirred at room temperature for ~5 minutes and then placed into a preheated oil bath (50 °C) for 24 hours. After completion of reaction, the reaction tube was allowed to cool to room temperature. Ethyl acetate (~10 mL), dodecane (114 μL, internal standard) and water (~3 ml) were added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve.

Table S1. Screening of Reaction Conditions.^a



entry	base	solvent	yield % ^b
1	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	95 (91) ^c
2	K ₃ PO ₄	<i>t</i> -BuOH	59
3	K ₂ CO ₃	<i>t</i> -BuOH	49
4	Cs ₂ CO ₃	<i>t</i> -BuOH	22
5	K ₃ PO ₄ •H ₂ O	toluene	90
6	K ₃ PO ₄ •H ₂ O	DMF	61
7	K ₃ PO ₄ •H ₂ O	dioxane	58
8	K ₃ PO ₄ •H ₂ O	<i>i</i> -PrOH	64
9 ^d	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	0
10 ^e	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	76
11 ^f	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	87
12 ^g	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	22
13 ^h	K ₃ PO ₄ •H ₂ O	<i>t</i> -BuOH	28

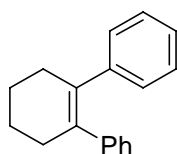
14ⁱ K₃PO₄•H₂O *t*-BuOH 19

^aReaction conditions: Pd(OAc)₂ (2.0 mol %), CM-phos (8.0 mol %), **1** (1.0 mmol), ArB(OH)₂ (2.0 mmol), base (3.0 mmol), solvent (3.0 mL) were stirred at 50 °C for 24 h under nitrogen. ^bCalibrated GC yields were reported using dodecane as the internal standard. ^cIsolated yield in parenthesis. ^dReaction was conducted at room temperature for 24 h. ^eSPhos ligand is used. ^fXPhos ligand is used. ^gAd₂PBu ligand is used. ^h*t*Bu₃P ligand is used. ⁱPh₃P ligand is used.

General procedure for Suzuki reaction of alkenyl mesylates or tosylates: Pd(OAc)₂ and CM-phos (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for several times. Precomplexation was applied by adding freshly distilled dichloromethane (1 mL) and Et₃N (100 μL) into the tube. The solution was stirred and warmed using a hair drier for about 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under a high vacuum. Alkenyl tosylates/mesylate (1.0 mmol), aryl boronic acid (2.0 mmol) and K₃PO₄•H₂O (3.0 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for several times. The solvent *tert*-butanol (3.0 mL) was then added. The tube was stirred at room temperature for several minutes and then placed into a preheated oil bath (50-110 °C) for the time period as indicated in Table. After completion of reaction as judged by GC analysis, the reaction tube was allowed to cool to room temperature and quenched with water and diluted with EtOAc. The organic layer was separated and the aqueous layer was washed with EtOAc. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

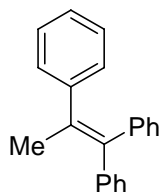
5. Characterization data for coupling products

1,2-Diphenylcyclohex-1-ene (Table 2, Entry 1)⁵



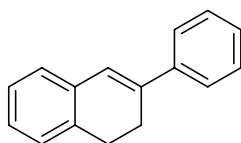
Hexane, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 2.04-2.05 (s, 4H), 2.68 (s, 4H), 7.19-7.24 (m, 6H), 7.27-7.29 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 23.21, 31.88, 125.6, 127.5, 128.9, 134.9, 143.8; MS (EI): m/z (relative intensity) 234 (M^+ , 100), 191 (20), 178 (5).

Prop-1-ene-1,1,2-triyltribenzene (Table 2, Entry 2 and Table 4, Entry 1)⁶



Hexane, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 2.37 (s, 3H), 7.14-7.23 (m, 5H), 7.31-7.39 (m, 5H), 7.45-7.54 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 23.27, 125.8, 126.1, 126.5, 127.3, 127.8, 128.0, 129.2, 30.0, 130.8, 135.6, 139.3, 143.0, 143.5, 143.9; MS (EI): m/z (relative intensity) 270 (M^+ , 100), 255(50), 239(15).

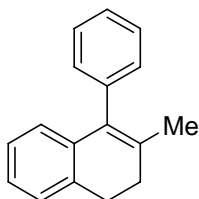
1,2-Dihydro-3-phenylnaphthalene (Table 2, Entry 3)⁷



Hexane, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 2.93-2.97 (m, 2H), 3.14-3.18 (m, 2H), 7.09 (s, 1H), 7.34-7.42 (m, 4H), 7.48-7.52 (m, 1H), 7.57-7.60 (m, 2H), 7.74-7.75 (m,

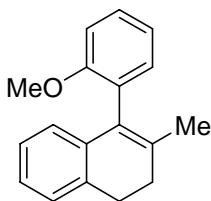
2H) 7.76-7.77 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 26.23, 28.07, 125.2, 125.0, 126.5, 126.9, 127.1, 127.2, 128.4, 134.6, 134.6, 138.5, 141.0; MS (EI): m/z (relative intensity) 206 (M^+ , 100), 191 (20), 178 (5).

3-Methyl-4-phenyl-1,2-dihydronaphthalene (Table 2, Entry 5)⁸



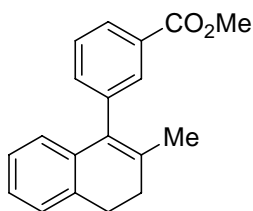
Hexane, $R_f=0.5$; ^1H NMR (400 MHz, CDCl_3) δ 1.96 (s, 3H), 2.58-2.62 (m, 2H), 3.07-3.11(t, $J=7.6\text{Hz}$, 2H), 6.82-6.83 (d, $J=4\text{Hz}$, 1H), 7.21-7.29 (m, 2H), 7.33-7.35 (m, 3H), 7.38-7.50 (m, 1H), 7.51-7.60 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.34, 28.24, 30.21, 125.2, 125.7, 126.1, 126.5, 127.0, 128.2, 130.2, 133.6, 133.9, 134.7, 136.9, 140.0; MS (EI): m/z (relative intensity) 220 (M^+ , 100), 205 (90), 191 (20).

4-(2-Methoxyphenyl)-3-methyl-1,2-dihydronaphthalene (Table 2, Entry 6)



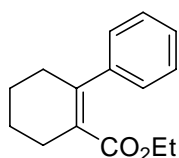
EA/Hexane 1:20, $R_f=0.5$; ^1H NMR (400 MHz, CDCl_3) δ 1.87 (s, 3H), 2.49-2.65 (m, 2H), 3.04-3.08 (t, $J=8\text{Hz}$, 2H), 3.04-3.08 (m, 2H), 3.84 (s, 3H) 6.71- 6.73 (d, $J=7.2\text{Hz}$, 1H), 7.10-7.29 (m, 6H), 7.45-7.50 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.31, 28.21, 30.03, 55.51, 111.1, 120.6, 124.4, 125.5, 126.0, 127.0, 128.2, 128.5, 130.0, 131.8, 134.5, 134.6, 136.5, 157.5; MS (EI): m/z (relative intensity) 250 (M^+ , 100), 235 (40), 219 (20).; HRMS: calcd. for $\text{C}_{18}\text{H}_{19}\text{OH}^+$: 251.1441, found 251.1436.

Methyl 3-(2-methyl-3,4-dihydronaphthalen-1-yl)benzoate (Table 2, Entry 7)



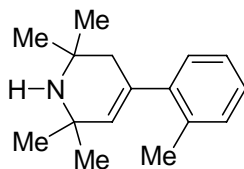
EA/Hexane 1:10, $R_f=0.5$; ^1H NMR (400 MHz, CDCl_3) δ 1.80 (s, 3H), 2.45-2.49 (t, $J=8.4\text{Hz}$ 2H), 2.84-2.97 (s, 2H), 3.97 (s, 3H), 6.58-6.60 (d, $J=7.6\text{Hz}$ 1H) 7.06-7.08 (m, 1H), 7.11-7.15 (m, 1H), 7.19-7.21 (m, 1H), 7.42-7.45 (m, 1H) 7.52-7.56 (m, 1H), 7.95-7.96 (t, $J=1.6\text{Hz}$, 1H) 8.01-8.10 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.27, 28.10, 30.15, 51.94, 125.0, 125.9, 126.1, 127.0, 128.4, 130.3, 131.3, 132.7, 134.7, 134.7, 134.9, 136.4, 140.2, 167.0; MS (EI): m/z (relative intensity) 278 (M^+ ,100), 263 (20), 249 (30); HRMS: calcd. for $\text{C}_{19}\text{H}_{19}\text{O}_2\text{H}^+$: 279.1390, found 279.1385.

Ethyl 2-phenylcyclohex-1-enecarboxylate (Table 2, Entry 8)⁹



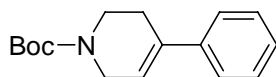
EA/Hexane 1:20, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 0.82-0.85 (t, $J=6\text{Hz}$, 3H), 1.72-1.77 (m, 4H), 2.37-2.45 (m, 4H), 3.85-3.89 (m, 2H), 7.13-7.15 (m, 2H), 7.23-7.30 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.31, 21.76, 22.33, 26.40, 32.42, 59.71, 126.6, 126.7, 127.7, 127.9, 143.4, 145.3, 169.7; MS (EI): m/z (relative intensity) 230 (M^+ ,68), 201 (10), 184 (100).

1,2,3,6-Tetrahydro-2,2,6,6-tetramethyl-4-*o*-tolylpyridine (Table 2, Entry 9)



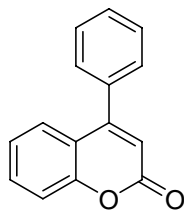
EA/Hexane 1:2, $R_f=0.3$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 1.27 (s, 6H), 1.3 (s, 6H), 2.12 (s, 2H), 2.34 (s, 3H), 5.55 (s, 1H), 7.08-7.10 (m, 1H), 7.16-7.18 (m, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 30.1, 31.4, 42.2, 49.5, 51.1, 125.5, 126.5, 128.1, 130.0, 133.7, 134.7, 143.2; MS (EI): m/z (relative intensity) 229 (M^+ ,3), 214 (100), 197 (5); HRMS: calcd. for $\text{C}_{16}\text{H}_{23}\text{NH}^+$: 230.1914, found 230.1909.

***Tert*-butyl 5,6-dihydro-4-phenylpyridine-1(2*H*)-carboxylate (Table 2, Entry 10)¹⁰**



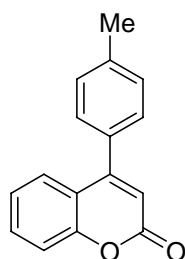
EA/Hexane 1:4, $R_f=0.5$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 1.53 (s, 9H), 2.54 (s, 2H), 3.66 (s, 2H), 4.10 (s, 2H), 6.04 (s, 1H), 7.26-7.40 (m, 5H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 28.3, 79.4, 124.6, 127.0, 128.1, 140.4, 154.6; MS (EI): m/z (relative intensity) 202 (M^+ ,100), 186 (10), 159 (60);

4-Phenylcoumarin (Table 3, Entry 1 and Table 4, Entry 3)³



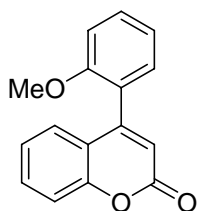
EA/Hexane 1:4, $R_f=0.45$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.34 (s, 1H), 7.19-7.23 (m, 1H), 7.35-7.49 (m, 8H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 114.9, 117.0, 118.7, 124.0, 126.8, 128.2, 128.7, 129.5, 131.7, 134.9, 153.9, 155.4, 160.4; MS (EI): m/z (relative intensity) 222 (M^+ , 100), 194 (80), 165 (70).

4-(4-Methylphenyl) coumarin (Table 3, Entry 2)¹¹



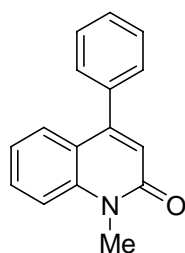
EA/Hexane 1:4, $R_f=0.5$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 6.25 (s, 1H), 7.13-7.16 (m, 1H), 7.27-7.30 (m, 5H), 7.44-7.47 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 21.02, 114.4, 115.0, 116.9, 118.6, 123.8, 126.7, 128.1, 129.2, 129.5, 131.5, 131.9, 139.6, 153.8, 155.3, 160.4; MS (EI): m/z (relative intensity) 236 (M^+ ,100), 221 (70), 208 (80).

4-(2-Methoxyphenyl) coumarin (Table 3, Entry 3)¹²



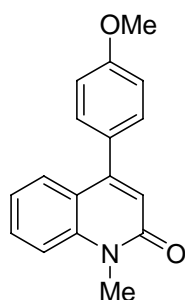
EA/Hexane 1:4, $R_f=0.4$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 3.72 (s, 3H), 6.337 (s, 1H), 7.03-7.06 (m, 2H), 7.14-7.19 (m, 3H), 7.33 (m, 1H), 7.44-7.46 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 55.21, 11.0, 115.9, 116.5, 119.1, 120.6, 123.7, 123.8, 127.0, 129.7, 130.8, 131.3, 153.4, 153.5, 156.1, 160.7; MS (EI): m/z (relative intensity) 252 (M^+ ,100), 237(20), 221(80).

1-Methyl-4-phenyl-2-quinolone (Table 3, Entry 4)³



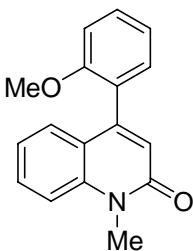
EA/Hexane 1:1, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 3.71 (s, 3H), 6.64 (s, 1H), 7.09-7.12 (t, $J = 7.2\text{Hz}$, 1H), 7.34-7.37(m, 3H), 7.41-7.50 (m, 3H), 7.52-7.54 (t, $J=8.4\text{Hz}$, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 29.09, 114.1, 120.0, 120.8, 121.6, 127.3, 128.2, 128.3, 128.5, 130.4, 136.7, 139.9, 150.5, 161.5; MS (EI): m/z (relative intensity) 235 (M^+ ,100), 207 (14).

4-(4-Methoxyphenyl)-1-methyl-2-quinolone (Table 3, Entry 5)³



EA/Hexane 1:1, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 3.62 (s, 3H), 3.75 (s, 3H), 6.54 (s, 1H), 6.89-6.91 (m, 2H), 7.02-7.06(m, 1H), 7.22-7.27 (m, 3H), 7.42-7.49 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 28.86, 54.85, 113.5, 114.0, 120.0, 120.4, 121.3, 127.1, 128.7, 129.7,130.1, 139.8, 150.1, 159.5, 161.4; MS (EI): m/z (relative intensity) 265 (M^+ ,70), 222 (16), 207 (100).

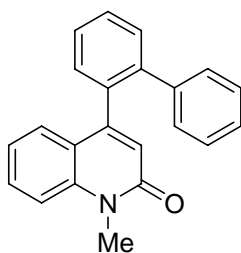
4-(2-Methoxyphenyl)-1-methyl-2-quinolone (Table 3, Entry 6 and Table 4, Entry 4)¹³



EA/Hexane 1:1, $R_f=0.3$; ^1H NMR (400 MHz, CDCl_3) δ 3.67(s, 3H), 3.71 (s, 3H), 6.66 (s, 1H), 6.98-7.09 (m, 3H), 7.17-7.19 (dd, $J=1.6,7.6\text{Hz}$ 1H) 7.24-7.26 (dd, $J=1.2, 8\text{Hz}$,

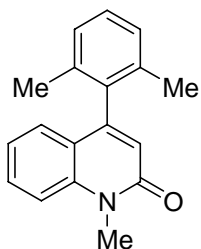
1H), 7.33-7.35 (d, $J=8\text{Hz}$, 1H), 7.38-7.42 (m, 1H), 7.47-7.51 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 29.00, 55.12, 110.7, 113.9, 120.4, 121.6, 125.6, 127.5, 129.9, 130.0, 130.1, 139.5, 148.3, 156.2, 161.7; MS (EI): m/z (relative intensity) 265 (M^+ , 100), 248 (16), 236(20).

4-(2-Biphenyl)-1-methyl-2-quinolone (Table 3, Entry 7)



EA/Hexane 1:1, $R_f=0.3$; ^1H NMR (400 MHz, CDCl_3) δ 3.67(s, 3H), 3.71 (s, 3H), 6.66 (s, 1H), 6.98-7.09 (m, 3H), 7.17-7.19 (dd, $J=1.6, 7.6\text{Hz}$ 1H) 7.24-7.26 (dd, $J=1.2, 8\text{Hz}$, 1H), 7.33-7.35 (d, $J=8\text{Hz}$, 1H), 7.38-7.42 (m, 1H), 7.47-7.51 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 29.00, 55.12, 110.7, 113.9, 120.4, 121.6, 125.6, 127.5, 129.9, 130.0, 130.1, 139.5, 148.3, 156.2, 161.7; MS (EI): m/z (relative intensity) 310 (M^+ , 100), 294 (50), 267(20). HRMS: calcd. for $\text{C}_{22}\text{H}_{18}\text{NOH}^+$: 312.1388, found 312.1376.

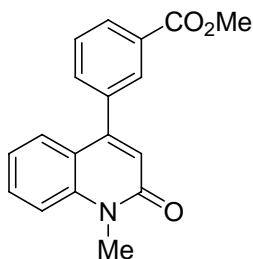
4-(2,6-Dimethylphenyl)-1-methyl-2-quinolone (Table 3, Entry 8)



EA/Hexane 1:1, $R_f=0.3$; ^1H NMR (400 MHz, CDCl_3) δ 3.67(s, 3H), 3.71 (s, 3H), 6.66 (s, 1H), 6.98-7.09 (m, 3H), 7.17-7.19 (dd, $J=1.6, 7.6\text{Hz}$ 1H) 7.24-7.26 (dd, $J=1.2, 8\text{Hz}$, 1H), 7.33-7.35 (d, $J=8\text{Hz}$, 1H), 7.38-7.42 (m, 1H), 7.47-7.51 (m, 1H); ^{13}C

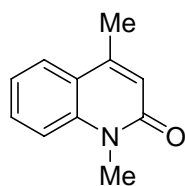
NMR (100 MHz, CDCl₃) δ29.00, 55.12, 110.7, 113.9, 120.4, 121.6, 125.6, 127.5, 129.9, 130.0, 130.1, 139.5, 148.3, 156.2, 161.7; MS (EI): *m/z* (relative intensity) 263 (M⁺, 100), 248 (60). HRMS: calcd. for C₁₈H₁₈NOH⁺: 264.1388, found 264.1375.

Methyl 3-(1-methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzoate (Table 3, Entry 9)



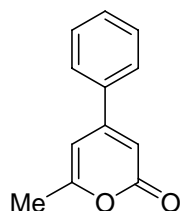
EA/Hexane 1:1, *R_f*=0.3; ¹H NMR (400 MHz, CDCl₃) δ3.74 (s, 3H), 3.89 (s, 3H), 6.64 (s, 1H), 7.11-7.15 (m, 1H), 7.40-7.43 (m, 2H) 7.53- 7.56 (m, 3H), 8.07-8.12 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ29.28, 52.08, 114.4, 119.9, 121.2, 121.9, 127.1, 128.6, 129.6, 129.7, 130.4, 130.7, 133.0, 137.1, 140.0, 149.6, 161.5, 166.2; MS (EI): *m/z* (relative intensity) 293 (M⁺, 100), 265 (30); HRMS: calcd. for C₁₈H₁₆NO₃H⁺: 294.1117, found 294.1130.

1, 4 Dimethyl-2-quinolone (Table 3, Entry 10)



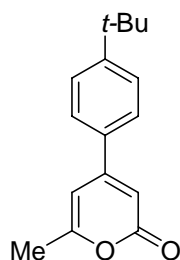
EA/Hexane 1:1, *R_f*=0.3; ¹H NMR (400 MHz, CDCl₃) δ3.74 (s, 3H), 3.89 (s, 3H), 6.64 (s, 1H), 7.11-7.15 (m, 1H), 7.40-7.43 (m, 2H) 7.53- 7.56 (m, 3H), 8.07-8.12 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ29.28, 52.08, 114.4, 119.9, 121.2, 121.9, 127.1, 128.6, 129.6, 129.7, 130.4, 130.7, 133.0, 137.1, 140.0, 149.6, 161.5, 166.2; MS (EI): *m/z* (relative intensity) 173 (M⁺, 100), 144 (70), 130 (40). HRMS: calcd. for C₁₁H₁₂NOH⁺: 174.0919, found 174.0910.

6-Methyl-4-phenyl-2-pyranone (Table 2, Entry 11)³



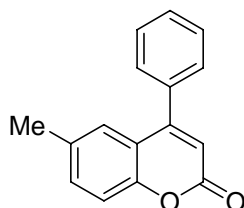
EA/Hexane 1:4, $R_f=0.3$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 2.23 (s, 3H), 6.25 (d, $J=0.4\text{Hz}$ 2H), 7.38-7.41 (m, 3H), 7.48-7.50 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 19.75, 103.1, 107.6, 126.3, 128.8, 130.3, 135.3, 155.1, 161.8, 170.0; MS (EI): m/z (relative intensity) 186 (M^+ ,58), 158 (100), 129 (20).

4-(4-*tert*-Butylphenyl)-6-methyl-2H-pyran-2-one (Table 3, Entry 12)



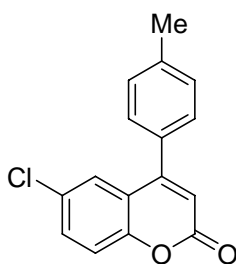
EA/Hexane 1:4, $R_f=0.4$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 1.35 (s, 9H), 2.32 (s, 3H) 6.31 (m, 2H), 6.35-6.36 (m, 2H), 7.48-7.53 (m, 4H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 20.40, 31.37, 35.09, 103.6, 107.7, 126.4, 126.7, 133.0, 154.5, 155.5, 162.2; MS (EI): m/z (relative intensity) 242 (M^+ ,78), 227 (19), 214 (20); HRMS: calcd. for $\text{C}_{16}\text{H}_{19}\text{O}_2\text{H}^+$: 243.1379, found 243.1385.

6-Methyl-4-phenyl-2H-chromen-2-one (Table 3 Entry 13 and Table 4, Entry 2)³



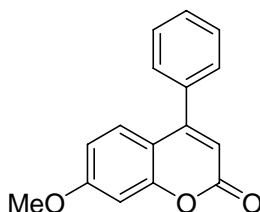
EA/Hexane 1:4, $R_f=0.4$; ^1H NMR (400 MHz, CDCl_3) δ 2.29 (s, 3H), 6.27 (s, 1H), 7.20 (s, 2H), 7.23 (s, 2H), 7.29-7.31 (m, 1H), 7.39-7.41 (m, 2H), 7.47-7.51 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 20.58, 114.7, 116.6, 118.3, 126.4, 128.1, 128.6, 129.3, 132.6, 133.6, 135.0, 151.2, 155.2, 160.4; MS (EI): m/z (relative intensity) 236 (M^+ ,100), 221 (10), 208 (80).

6-Chloro-4-*p*-tolyl-2H-chromen-2-one (Table 3, Entry 14)¹⁴



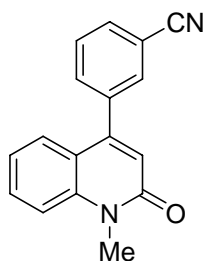
EA/Hexane 1:4, $R_f=0.5$; ^1H NMR (400 MHz, CDCl_3) δ 2.47 (s, 3H), 6.39 (s, 1H), 7.34-7.750 (m, 7H), ^{13}C NMR (100 MHz, CDCl_3) δ 21.2, 115.7, 118.6, 126.3, 128.1, 129.6, 131.5, 131.6, 140.1, 152.5, 154.5; MS (EI): m/z (relative intensity) 270 (M^+ ,100), 255 (70), 242 (90);

4-(4-*tert*-Butylphenyl)-6-methyl-2H-pyran-2-one (Table 3, Entry 14)¹²



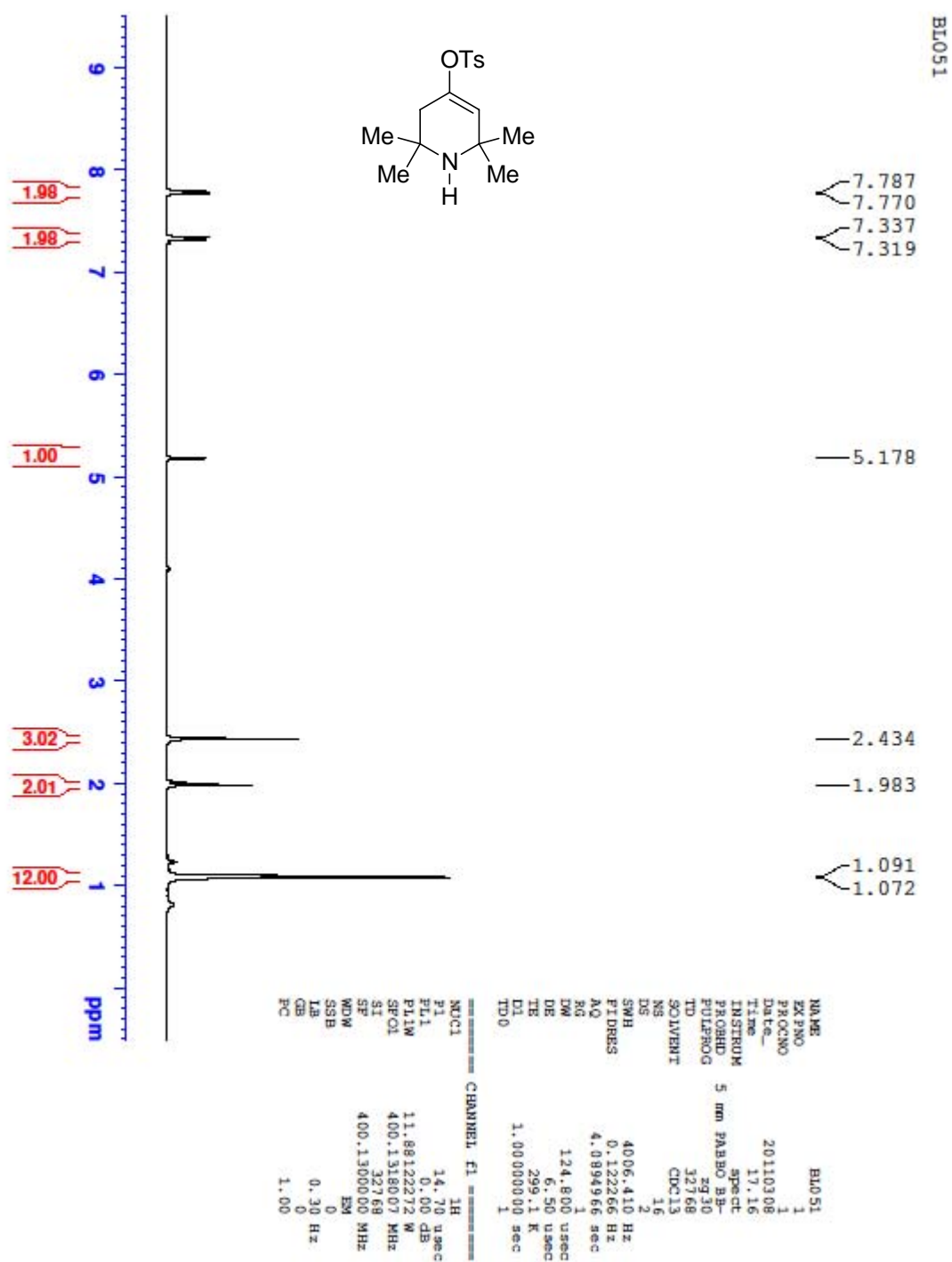
EA/Hexane 1:4, $R_f=0.5$; ^1H NMR (400 MHz, CDCl_3) δ 3.87 (s, 3H), 6.20 (s, 1H), 6.77-6.87 (m, 2H), 7.37-7.52 (m,6H), ^{13}C NMR (100 MHz, CDCl_3) δ 55.6, 100.9, 111.6, 112.1, 112.3, 127.8, 128.2, 128.6, 129.4, 135.3, 155.6, 155.8, 161.0, 162.6; MS (EI): m/z (relative intensity) 252 (M^+ ,100), 224 (95), 209 (80)

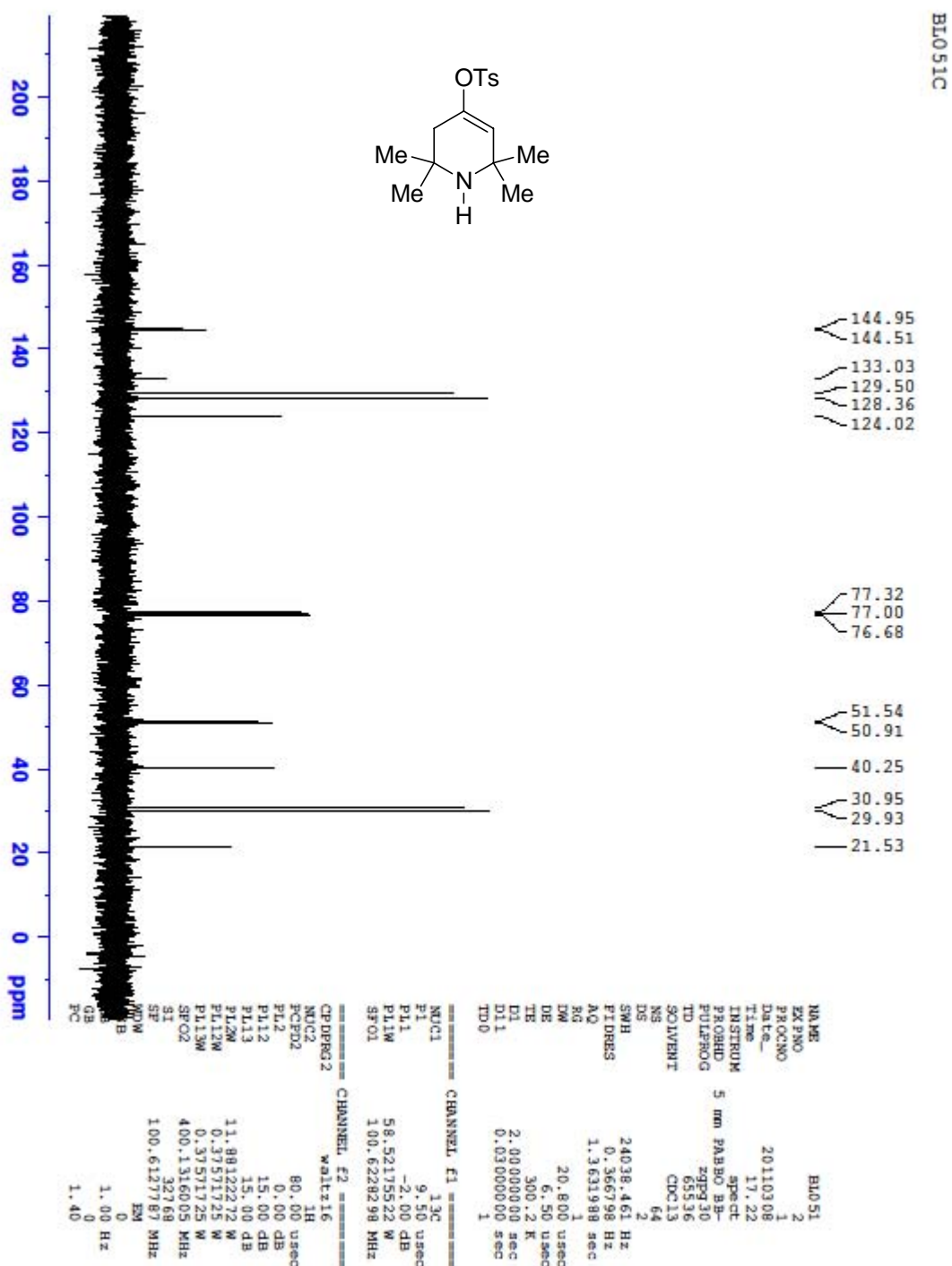
3-(1-Methyl-2-oxo-1,2-dihydroquinolin-4-yl)benzonitrile (Table 4, Entry 5)



EA/Hexane 1:1, $R_f=0.3$; ^1H NMR (400 MHz, CDCl_3) δ 3.76 (s, 1H), 6.63 (s, 1H), 7.19-7.21 (t, $J=1.2\text{Hz}$, 1H), 7.35-7.38 (m, 1H), 7.44-7.46 (d, $J=8\text{Hz}$, 1H), 7.59-7.65 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 29.49, 112.9, 114.7, 119.1, 119.6, 121.6, 122.2, 126.9, 130.0, 131.1, 132.2, 132.2, 133.1, 138.2, 140.2, 148.4, 161.37; MS (EI): m/z (relative intensity) 260 (M^+ ,100), 232 (30), 190 (20). HRMS: calcd. for $\text{C}_{17}\text{H}_{13}\text{N}_2\text{O}^+$: 261.1028, found 261.1028.

6. ¹H, ¹³C, NMR, and HRMS spectra





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Odd and Even Electron Ions

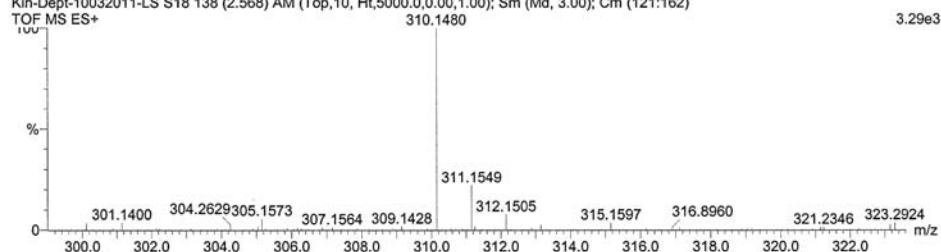
672 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-16 H: 0-24 N: 0-2 O: 0-8 Na: 0-1 S: 0-10

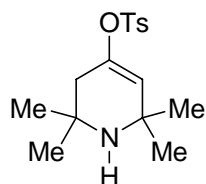
Kin-Dept-10032011-LS S18 138 (2.568) AM (Top,10, Ht,5000.0,0.00,1.00); Sm (Md, 3.00); Cm (121:162)

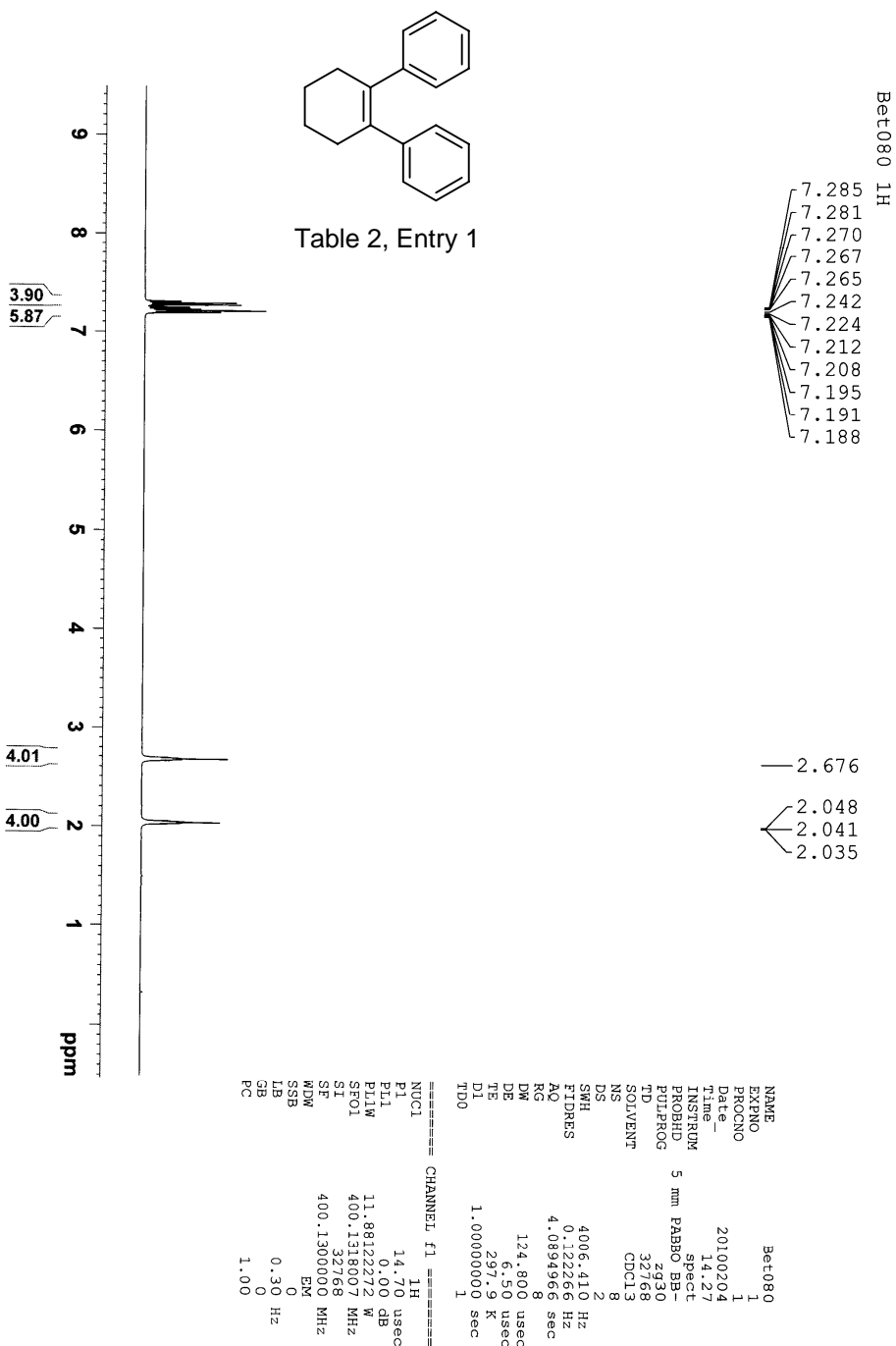
TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
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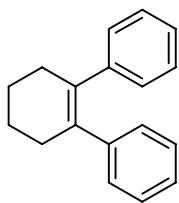
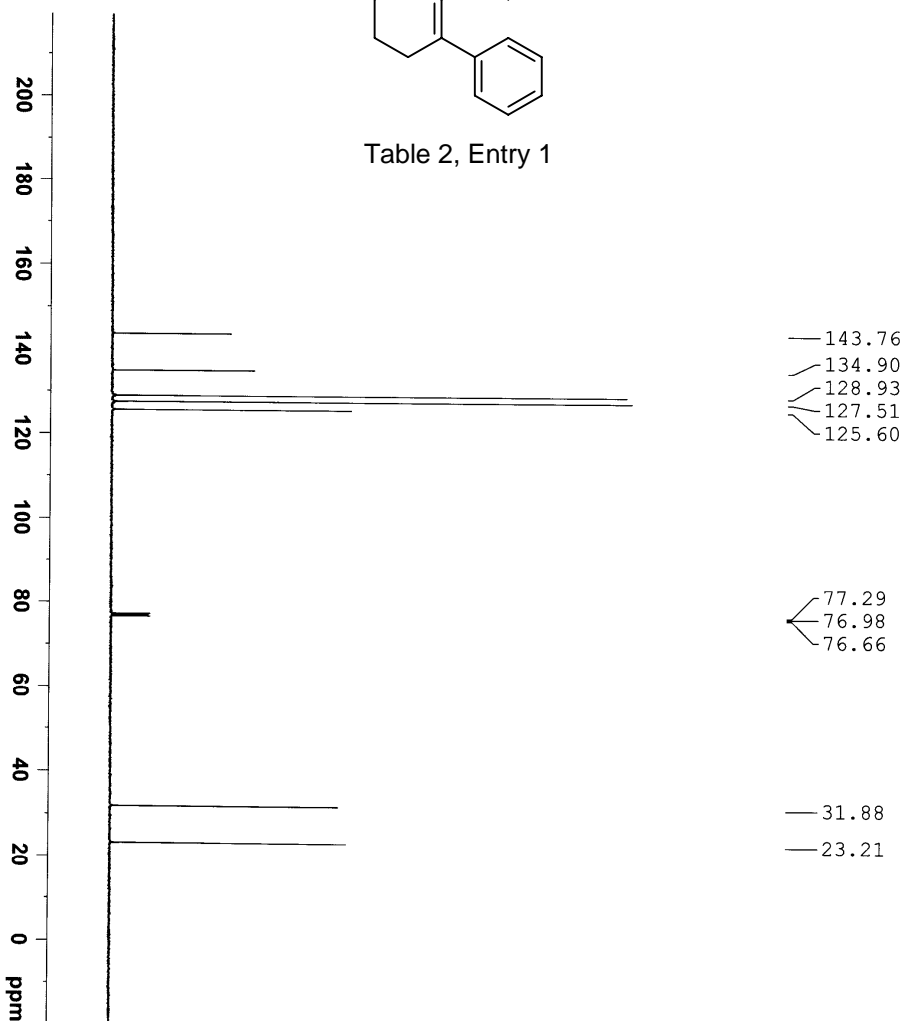


Table 2, Entry 1

Bet080 13C



143.76
 134.90
 128.93
 127.51
 125.60

77.29
 76.98
 76.66

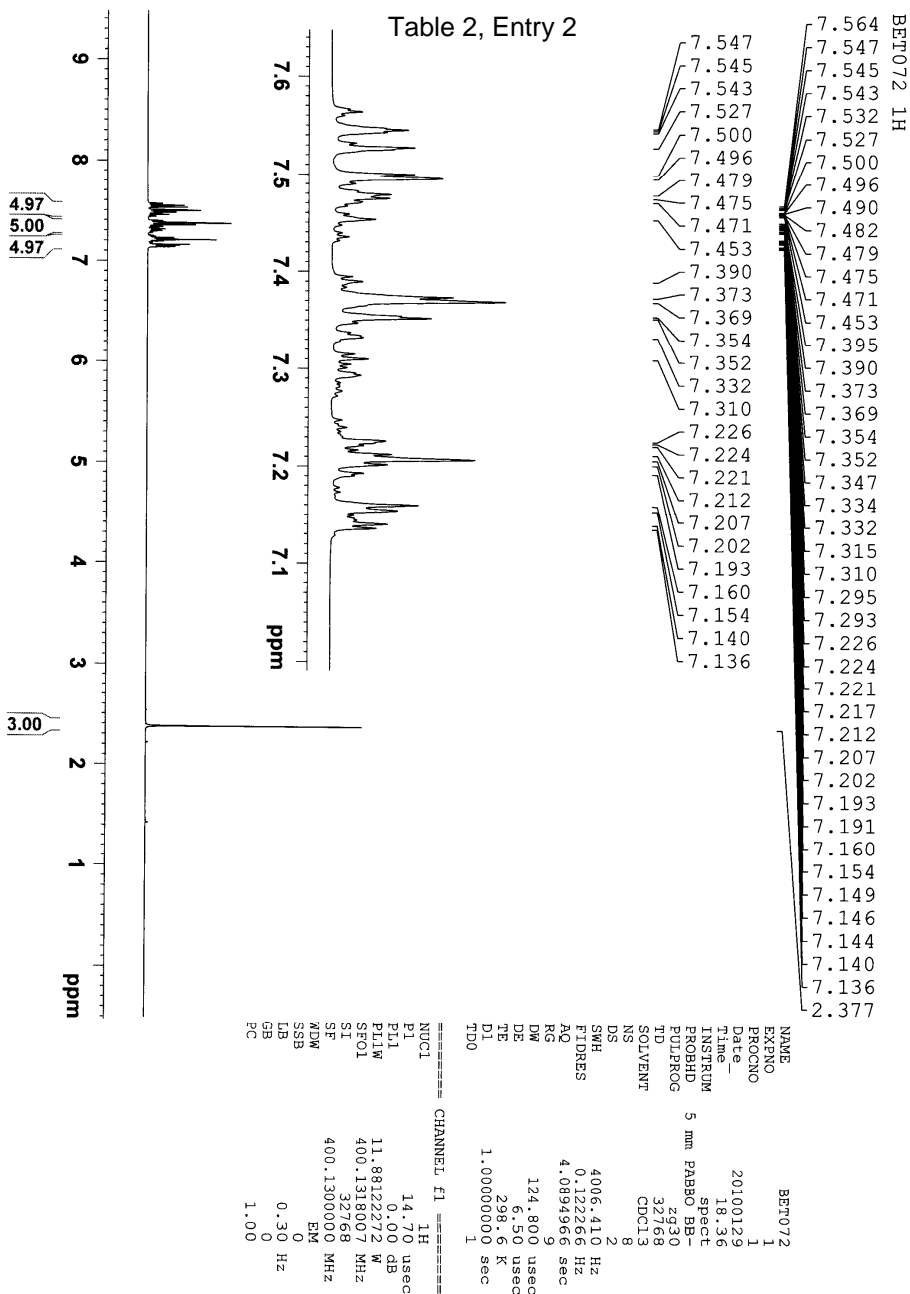
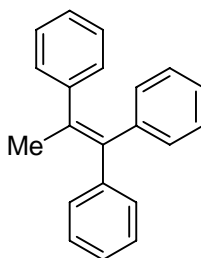
31.88
 23.21

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EXPNO 2
PROCNO 1
Date_ 20100204
Time_ 14.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 161
DE 20.800 usec
TE 298.4 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -2.00 dB
PL1W 58.5217522 W
SFO1 100.628298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.00 dB
PL13 15.00 dB
PL1W 11.8812272 W
PL12W 0.37571725 W
PL13W 0.37571725 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127986 MHz
WDW EM
SSB 0
GB 1.00 Hz
PC 1.40
    
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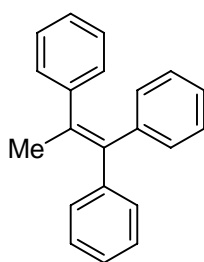
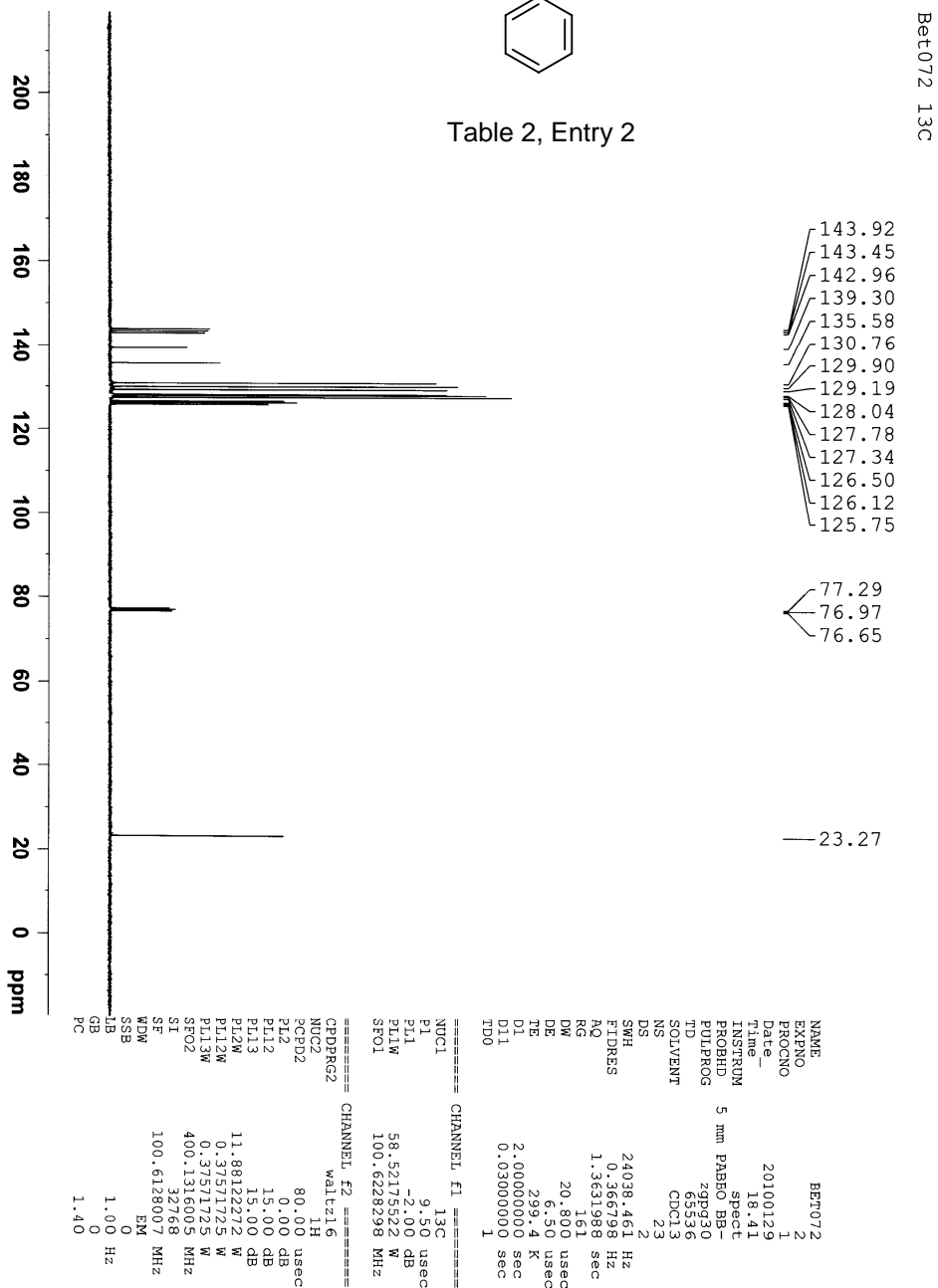


Table 2, Entry 2



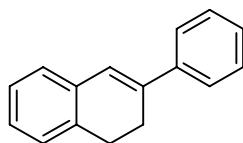
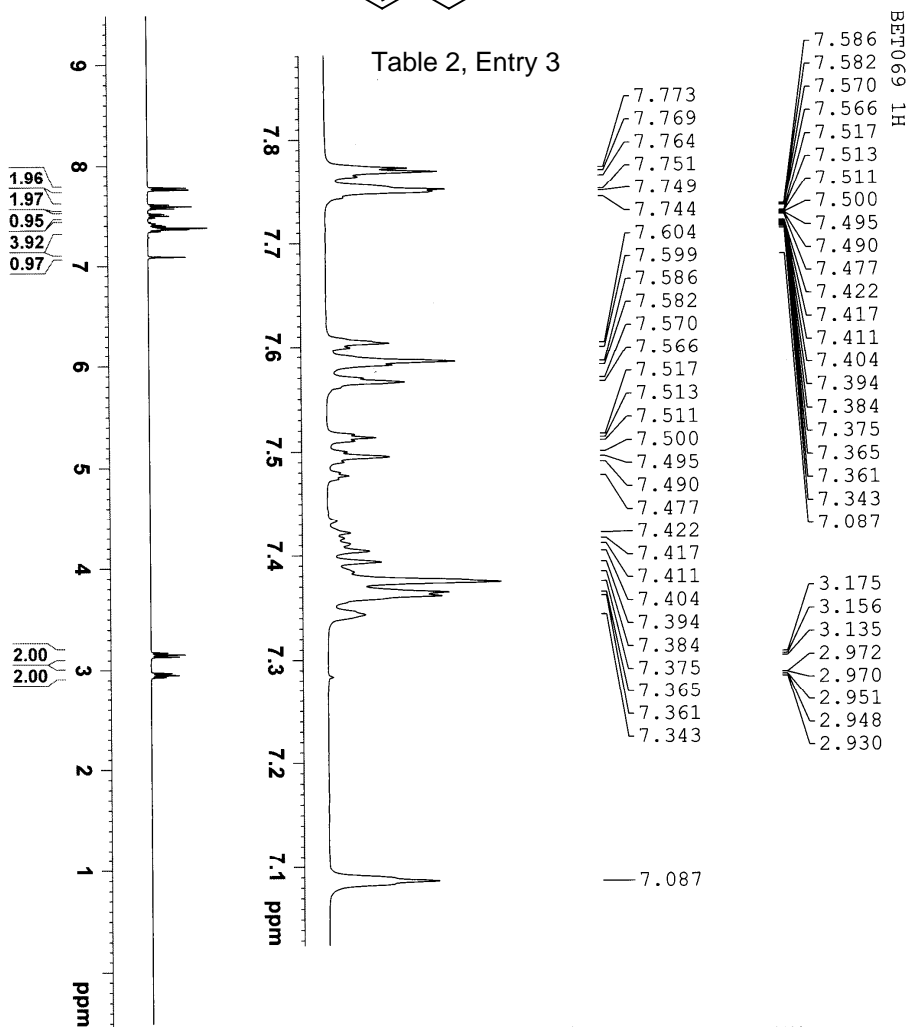


Table 2, Entry 3



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EXPNO         1
PROCNO        1
PROCNO        20100122
F2 -         18.40
NAME_         18.40
INSTRUM       spect
PROBHD        5 mm PABBO-
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            8
DS            2
SMH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            9
DE            124.800 usec
TE            298.3 K
D1            1.00000000 sec
TD0           1

===== CHANNEL F1 =====
NUC1          1H
P1            14.70 usec
PL1           0.00 dB
PL1W         11.88122272 W
SFO1         400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
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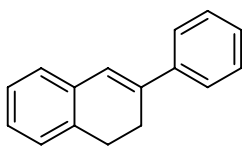
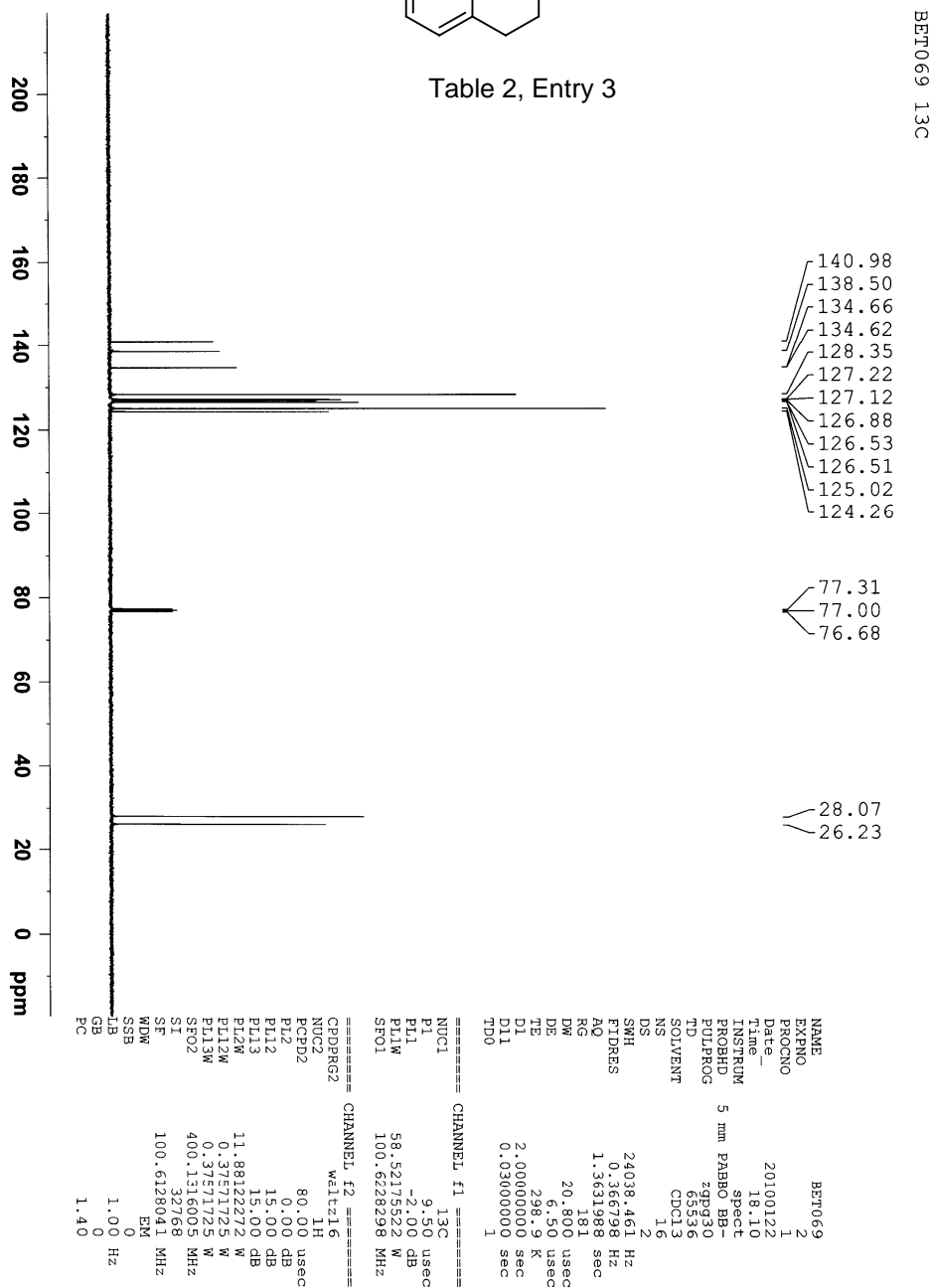


Table 2, Entry 3



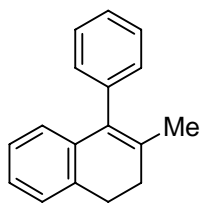
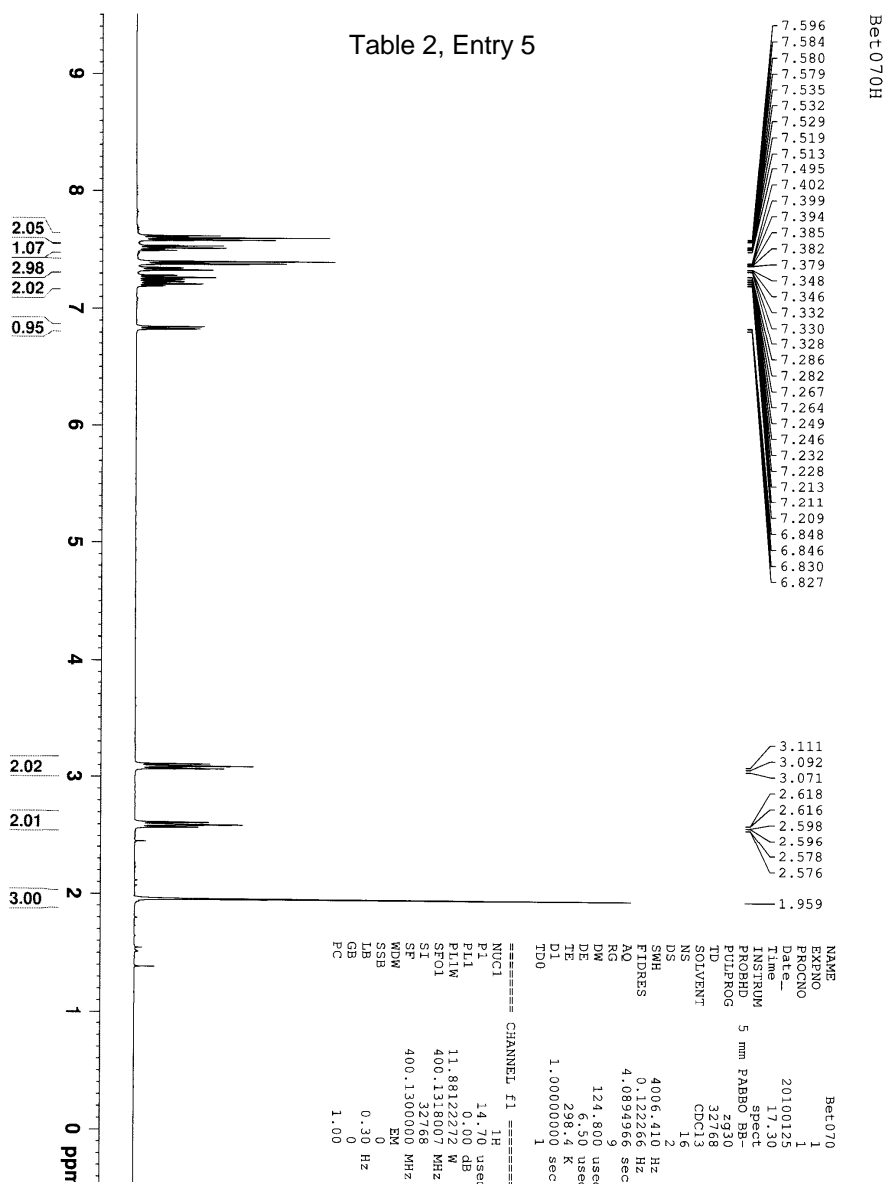


Table 2, Entry 5



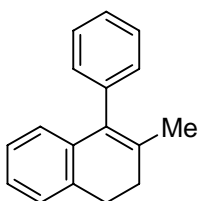
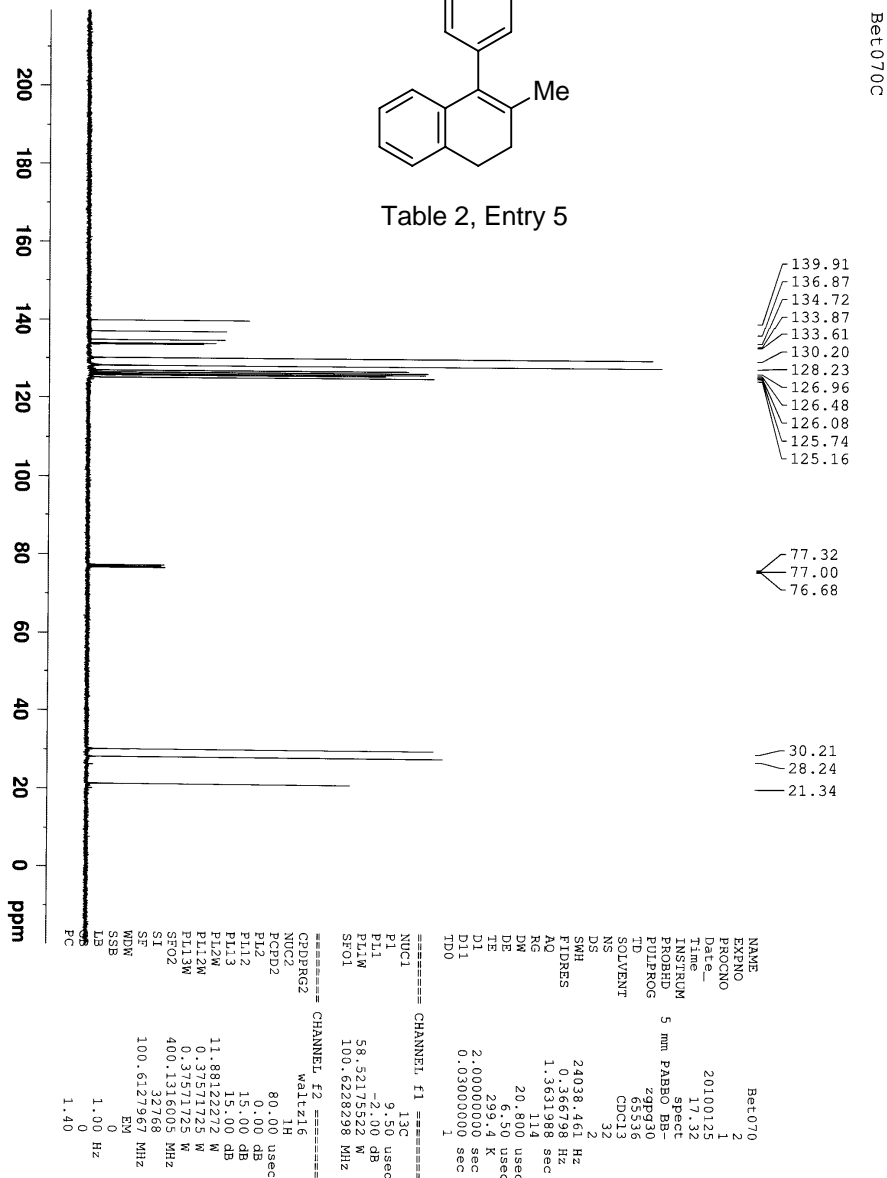


Table 2, Entry 5



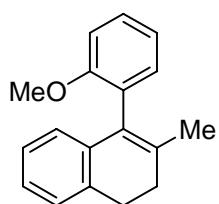
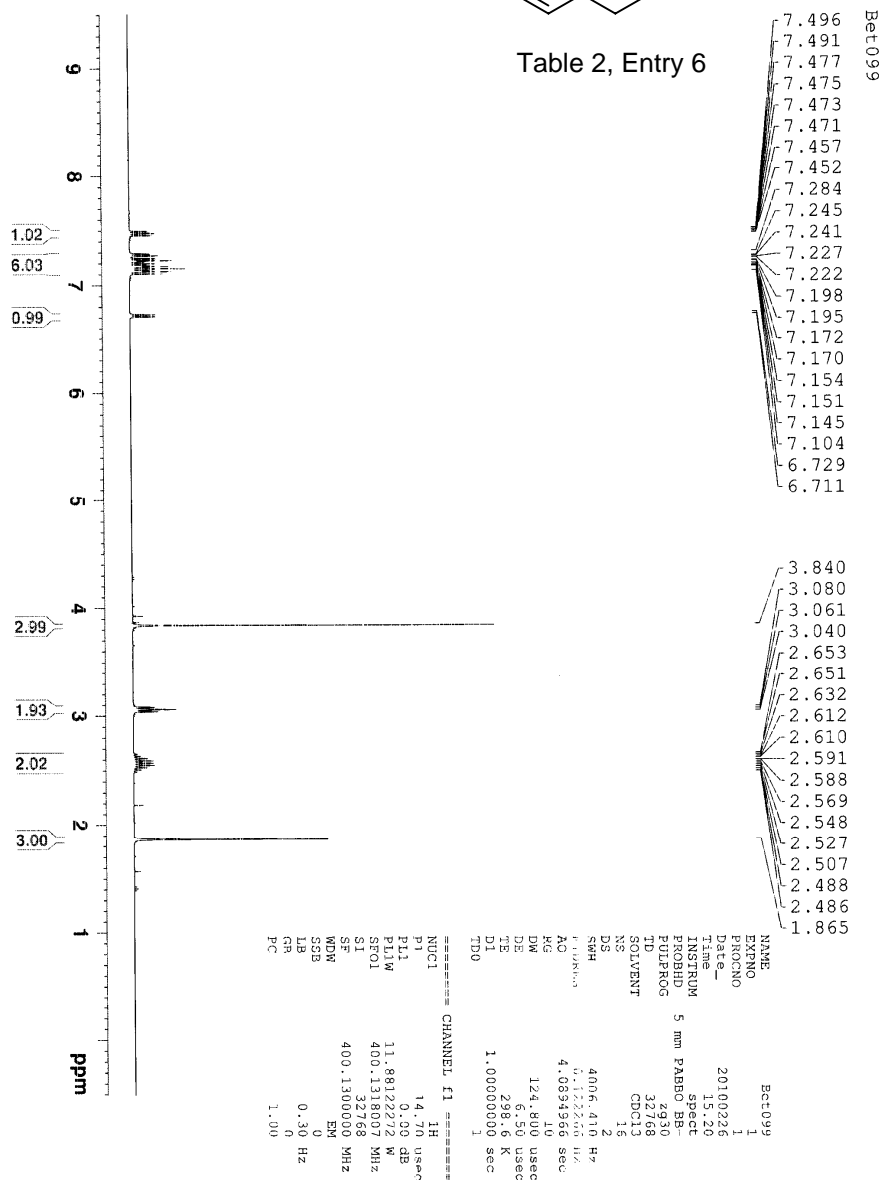


Table 2, Entry 6



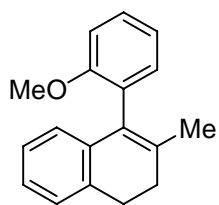
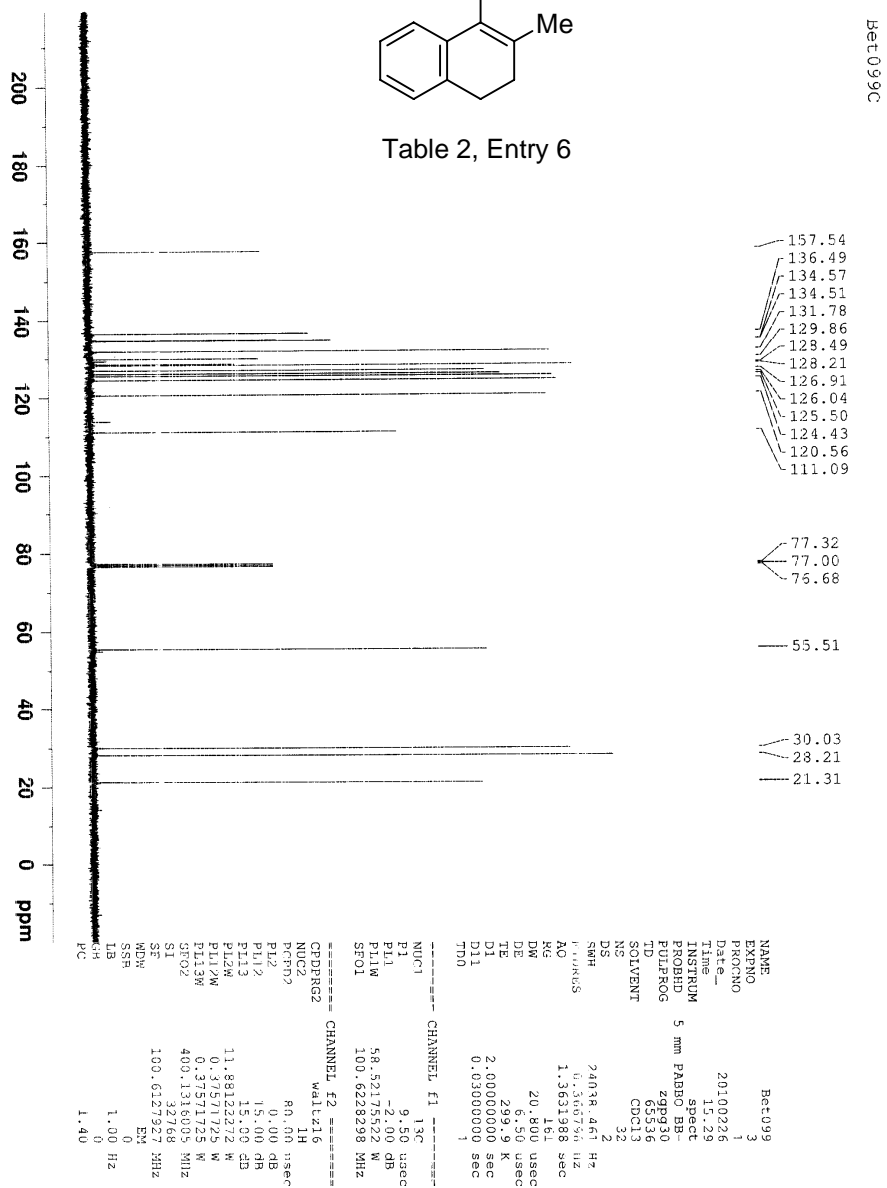


Table 2, Entry 6



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

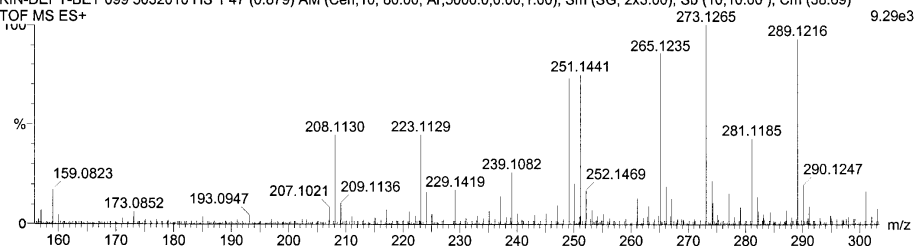
123 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-28 H: 0-45 N: 0-5 O: 0-2 Na: 0-1

KIN-DEPT-BET 099 5032010 HS 1 47 (0.879) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (38:69)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
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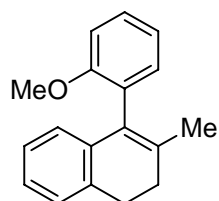
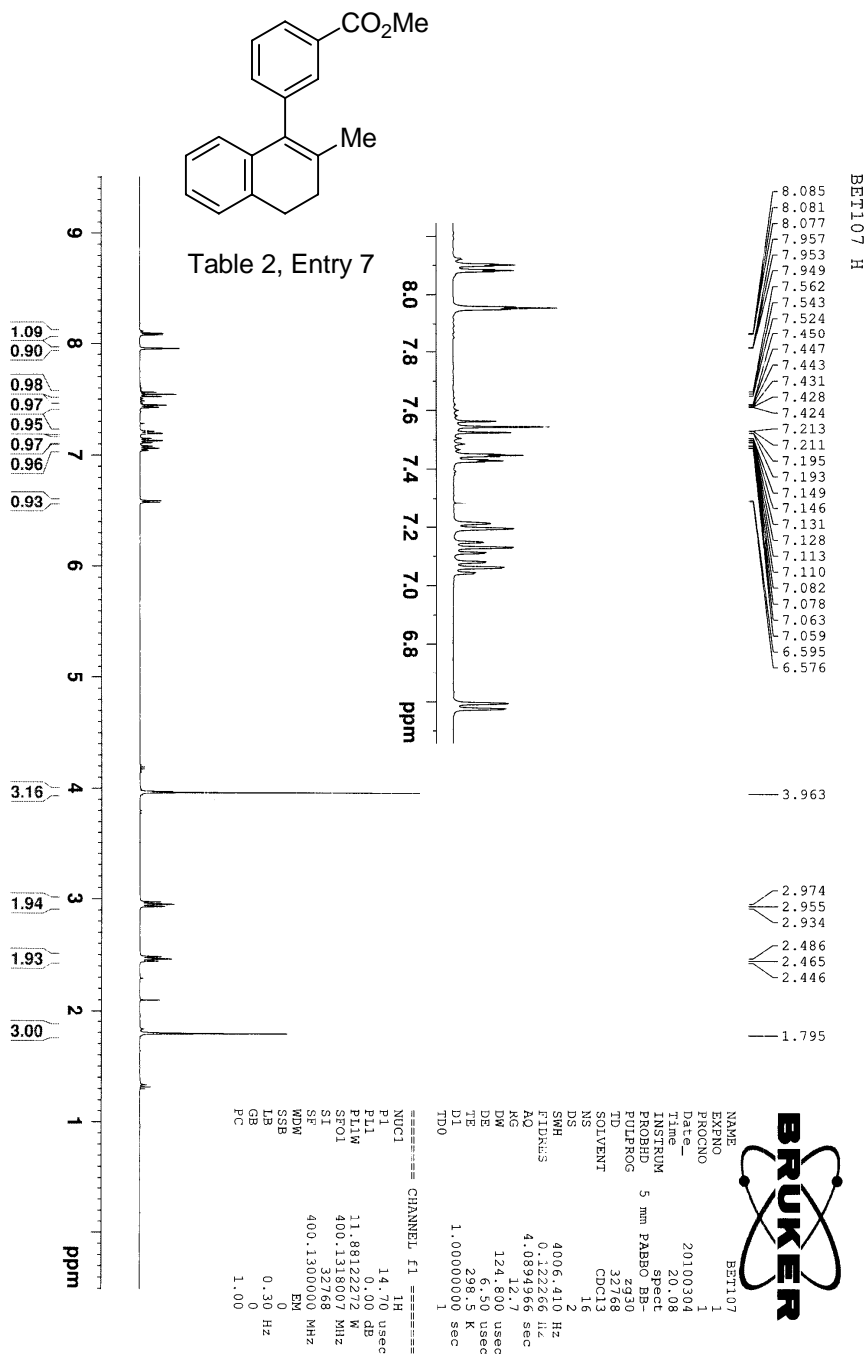


Table 2, Entry 6



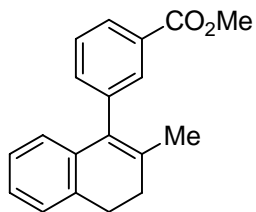
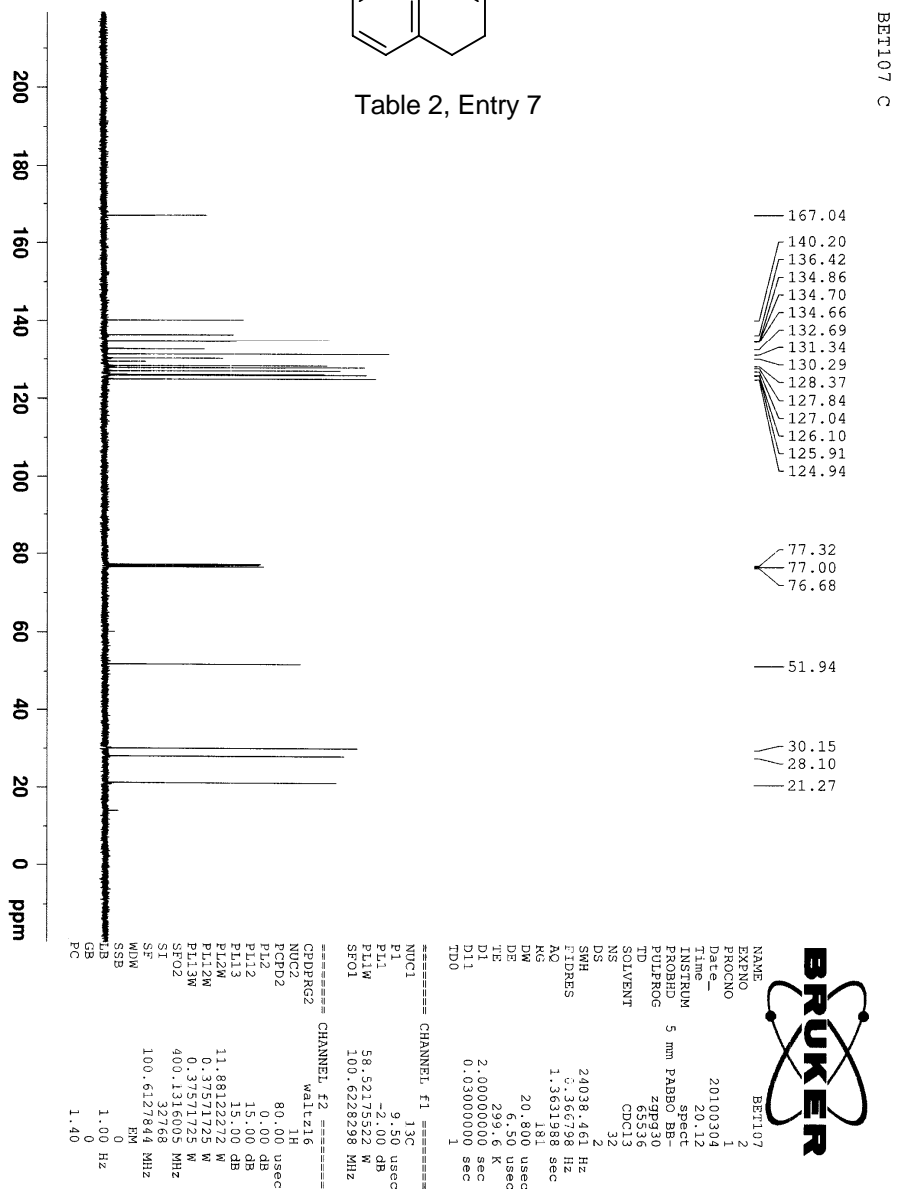


Table 2, Entry 7



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

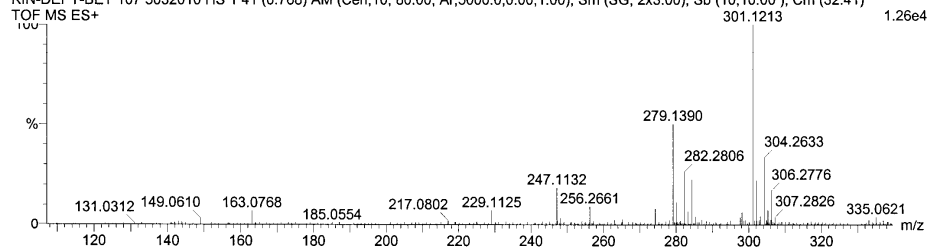
134 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-28 H: 0-45 N: 0-5 O: 0-2 P: 0-1

KIN-DEPT-BET 107 5032010 HS 1 41 (0.768) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (32:41)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
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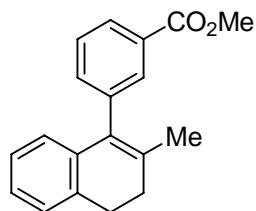


Table 2, Entry 7

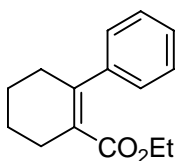
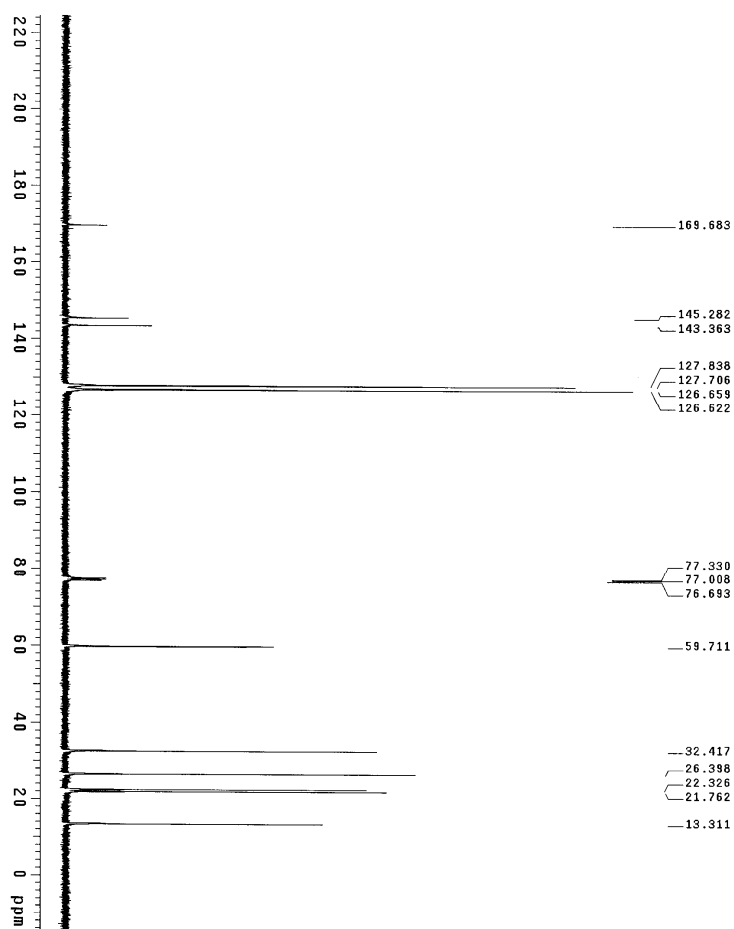


Table 2, Entry 8

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exp1 Carbon

SAMPLE 19.2008 4amp SPECIAL
date NOV 19.2008 4amp not used
solvent CDCl3 8amp not used
file /home/kiv/vmm~ not used
rsys /rvv/Bet039C.f~ hst 0.008
ACQUISITION 1d 6178 10.000
sw 24118.2 6178 FLAGS
at 1.300 11 n
np 62.50 1h n
bs 130.64 8s n
d1 1.000 1d PROCESSING 0.50
ne 1008 1d DISPLAY not used
ct TRANSMITTER 120 fm
tn C13 SP -1549.6
sfreq 100.518 MP1 2117.4
sf 100.518 MP1 2117.4
tqwr 1092.56 FFP 7239.9
pw 4.550 TP 149.6
DECOUPLER H1 TP -145.6
dof 0 WC PLOT 180
dm 0
dmm 4 YYY SC 208
dmm 4 YYY SC 208
dnt 11000 at cdc ph 4
    
```



B752

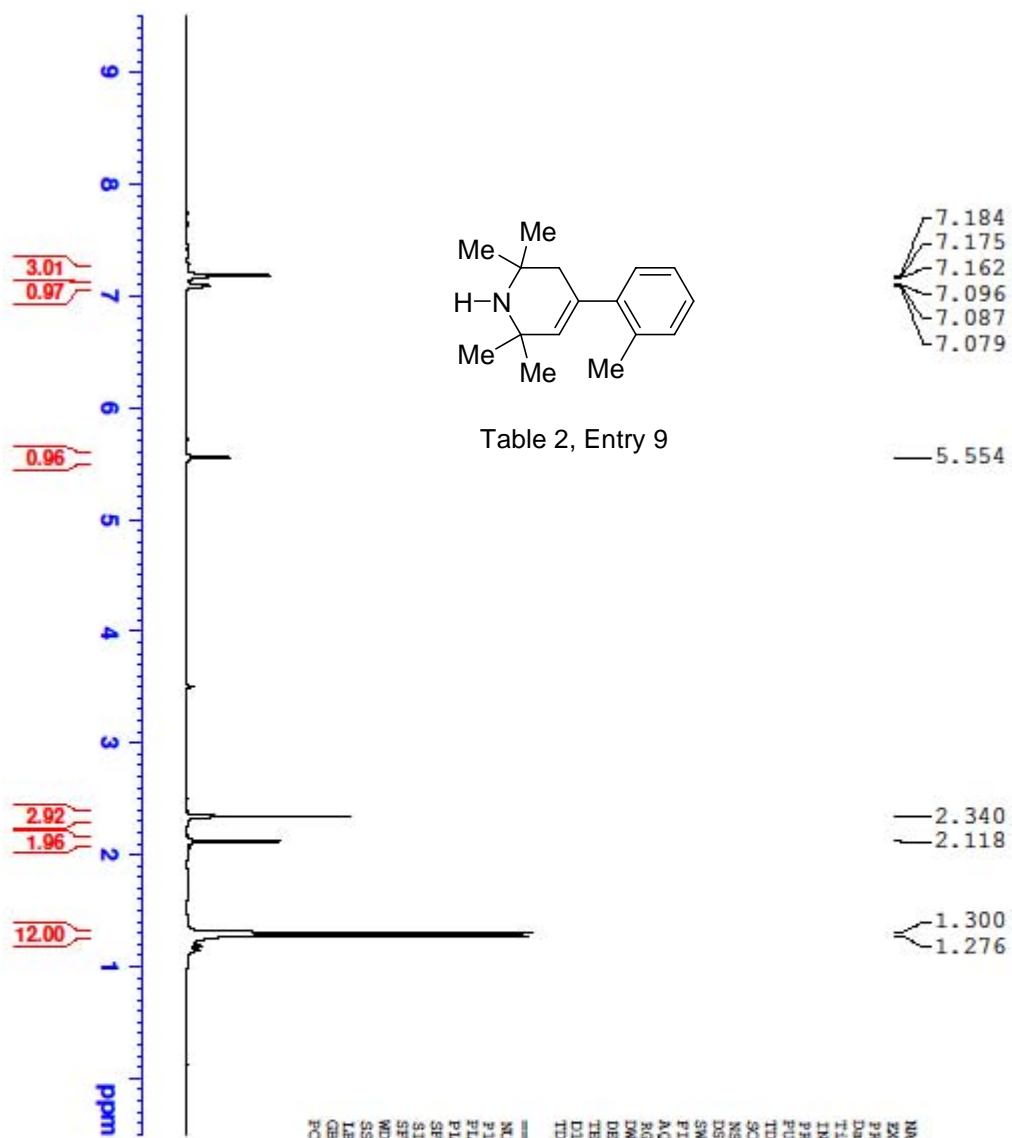


Table 2, Entry 9

```

NAME          B752
EXPNO         3
PROCNO        1
Date_         20110312
Time         17.08
INSTRUM       5 mm PABBO BB-
PROBHD        zg30
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG            1
RG            1
DE            124.800 usec
TR            6.50 usec
TE            299.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.79 usec
PL1           0.00 dB
PL1W          11.88122272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

B752C

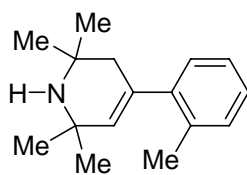
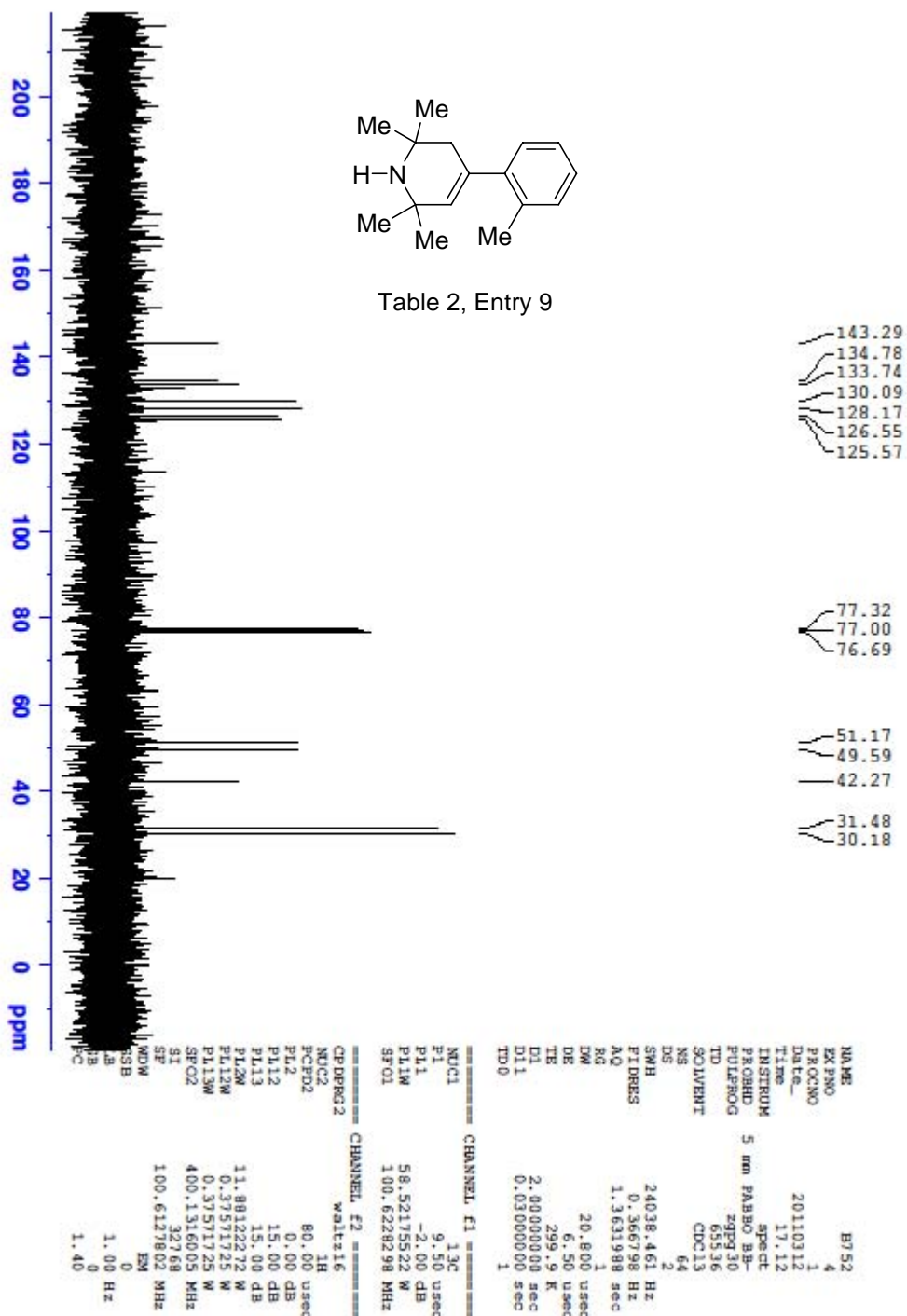


Table 2, Entry 9



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -100.0, max = 1000.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

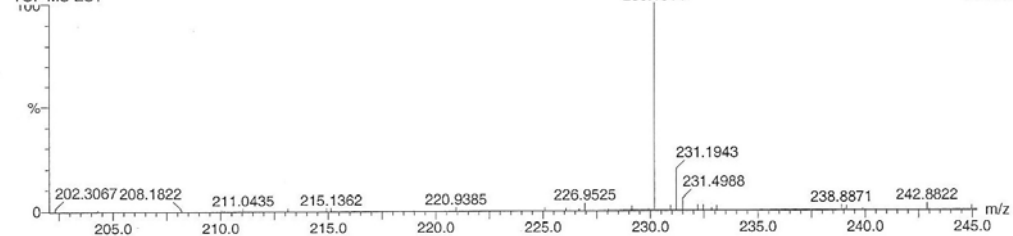
43 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-26 H: 0-35 N: 0-6 Na: 0-1

Kin-Dept-23032011 HS S22 61 (1.139) Cn (Cen,10, 80.00, Ar); Sm (SG, 2x3.00); Sb (10,10.00); Cm (58:67)

TOF MS ES+



Minimum:

Maximum: 5.0 5.0 -100.0 1000.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
230.1914	230.1909	0.5	2.2	5.5	3.4	C16 H24 N

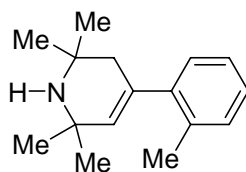
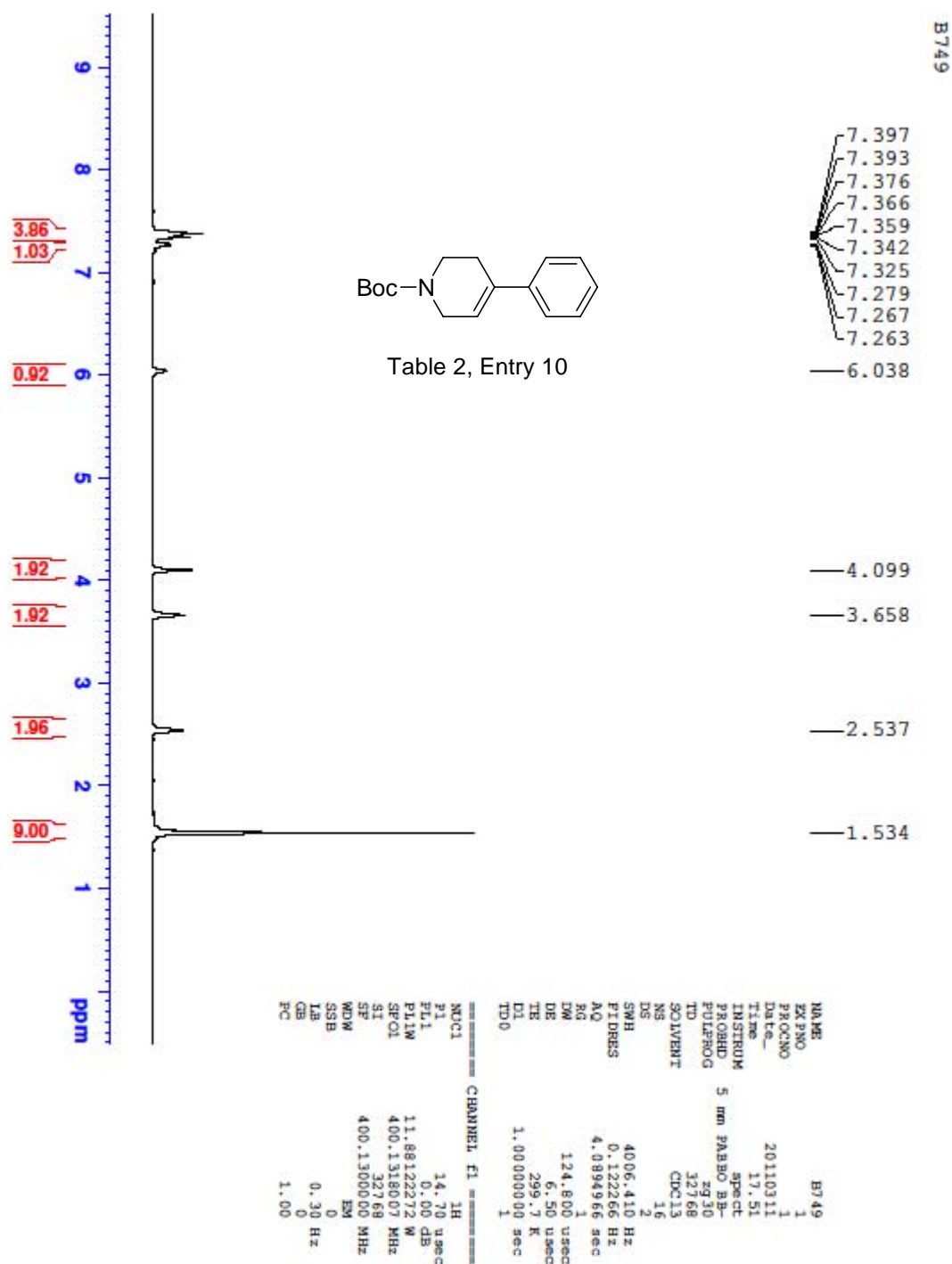


Table 2, Entry 9



B749C

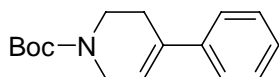
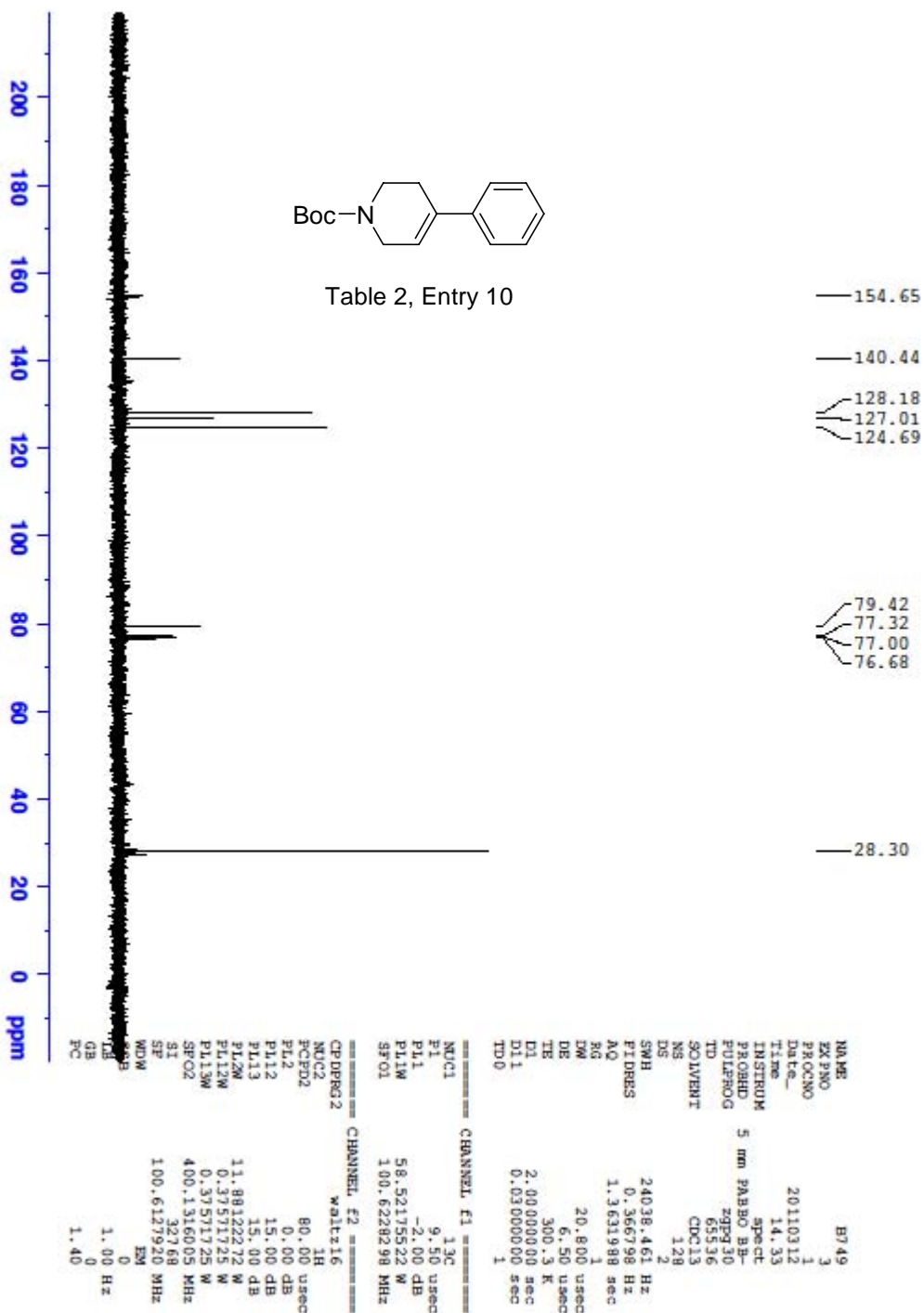


Table 2, Entry 10



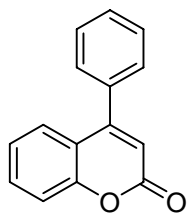
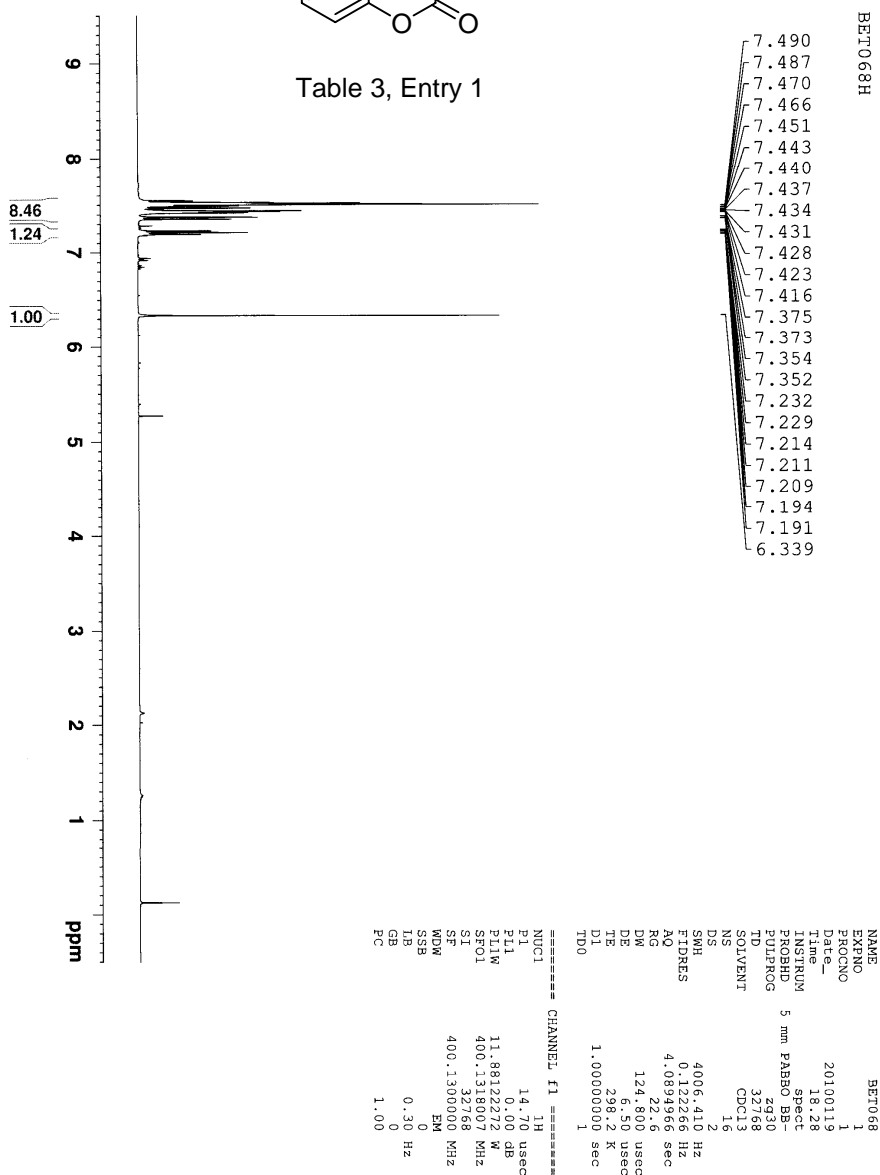


Table 3, Entry 1



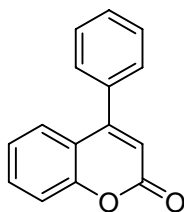
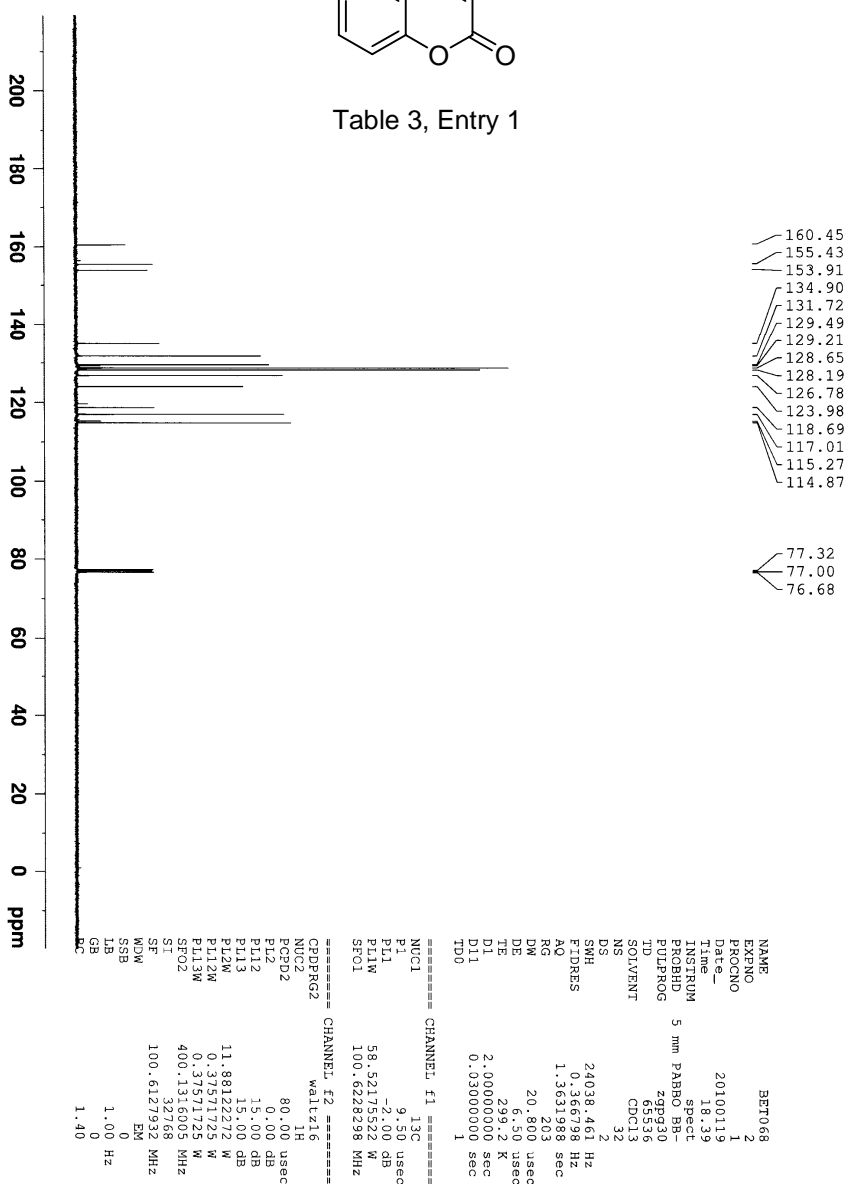


Table 3, Entry 1



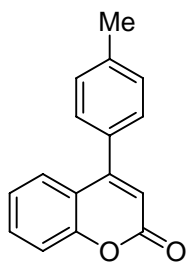
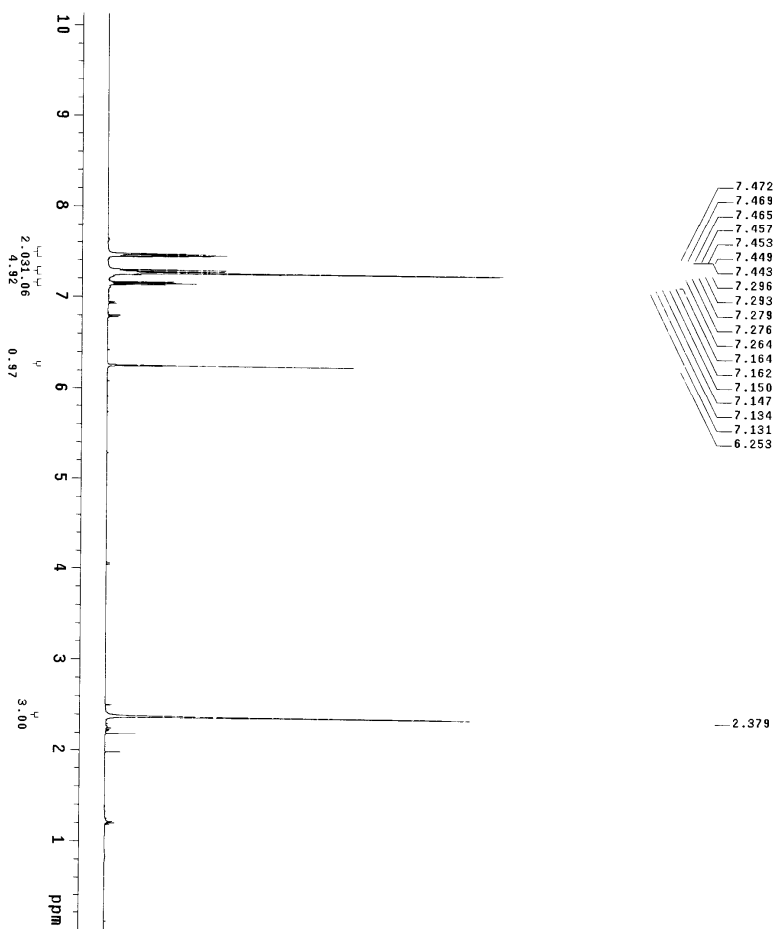


Table 3, Entry 2

```

BETG2
expi s2pu1
SAMPLE date Oct 27 2009
solvent CDCl3 temp
F1 ACQUISITION Exp gain
SW 7995.00 pw30 not used
AT 2.949 atfa 6.800
F1 4200 11
bc 32 1:4
ns 1.006 ns
ct 4 1b PROCESSING 0.20
tn TRANSMITTER 4 fn DISPLAY 65538
sfreq 489.742 sp -9.4
tof 489.6 wp 5071.9
zdwf 4.230 f: f1 939.8
nw DECOUPLER C13 1P 63.1
dn -2.5
dof 0 PLOT 180
dm 0
dwm 50 vs 24
dwr 13768 th at cdc ph 9
    
```



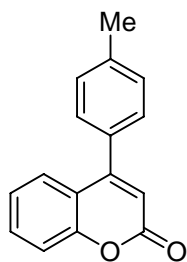
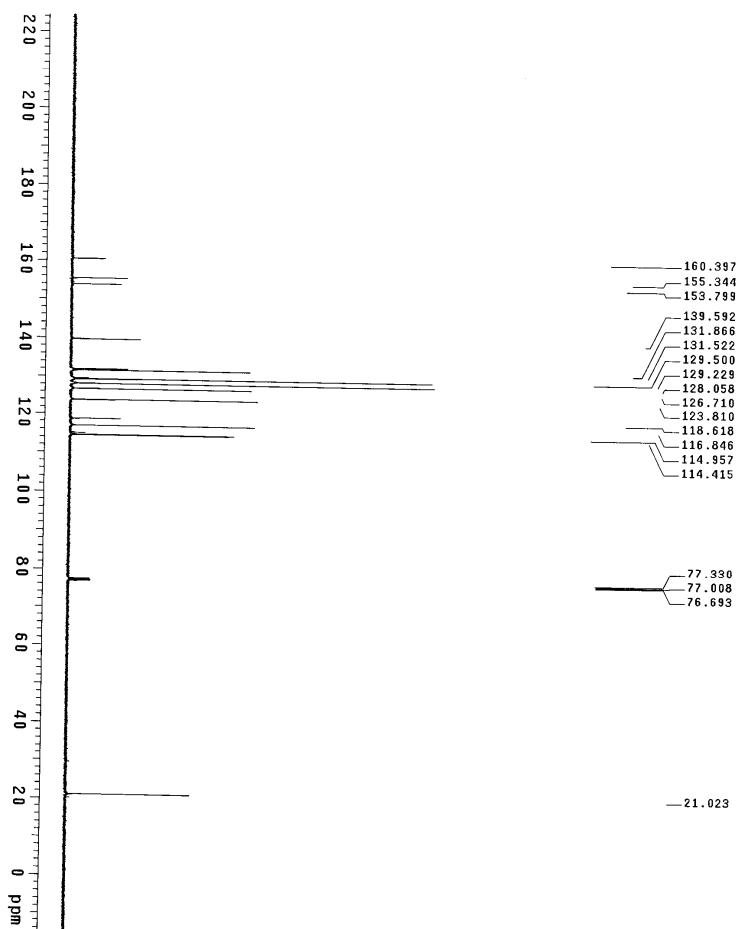


Table 3, Entry 2



~~4-(4-methylphenyl)coumarin~~ PK1012

```

exptl carbon
SAMPLe 57 2009
date 06/27/2009
solvent CDCl3
file 00013
sw ACQUISITION 2
at 1.300
np 62730
fd 13000
d1 1.000
nt 512
ct TRANSMITTER 64
tn 100.518
sffrq 10422.4
lort 10422.4
pwr 4.550
DECOUPLER HI
dn 42
dm VVY
dnam W
dpmr 42
dmf 11800
ai cdc ph

SPECIAL
temp gain not used
exp spin not used
nst 0.008
atfq 10.000
FLAGS 10.000
PROGRESSING 0.50
not used
DISPLAY 2
-1558.4
4131.4
7739.9
-166.1
-145.9
PLOT 180
49
0
2
    
```

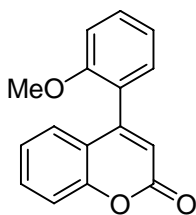
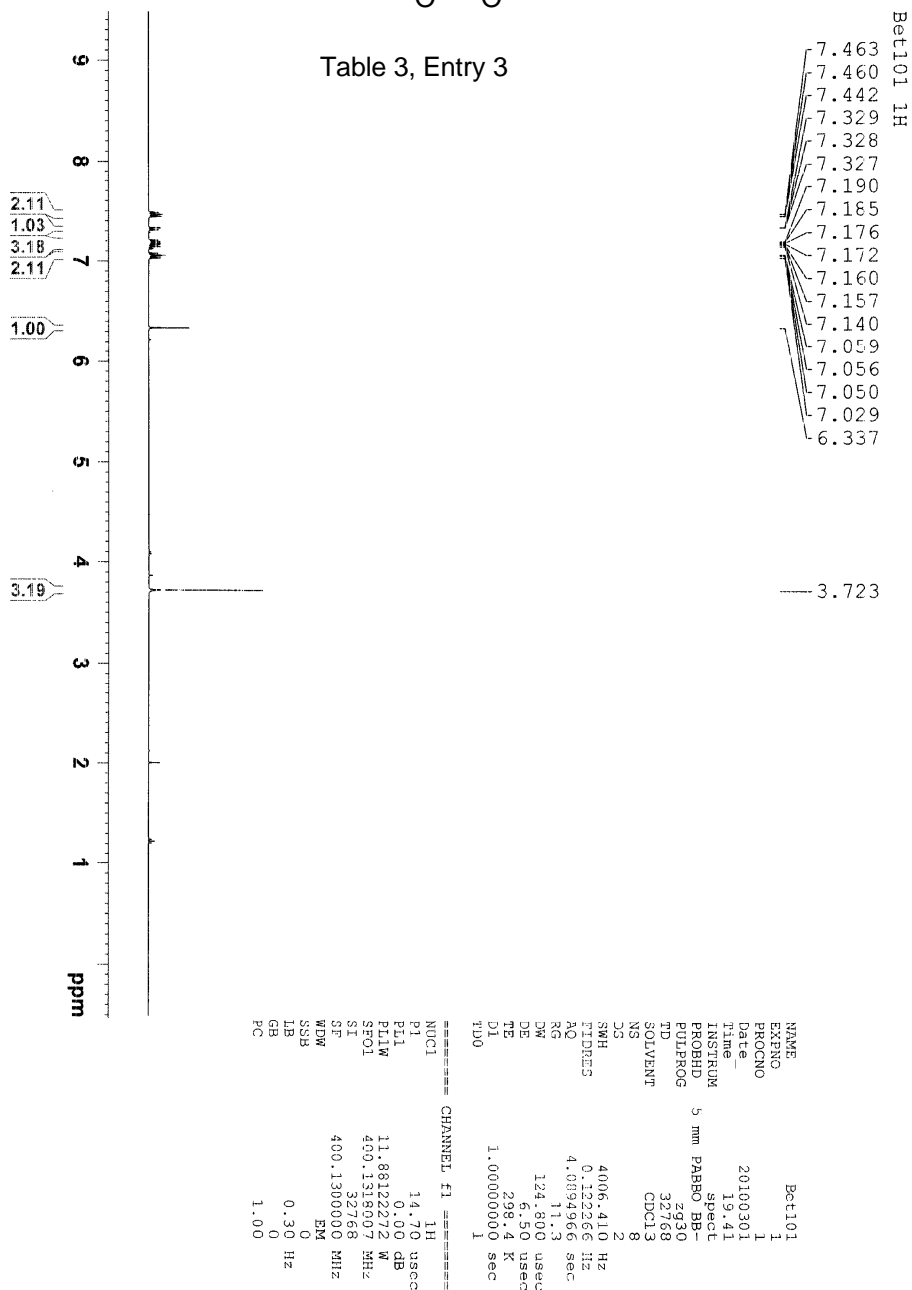


Table 3, Entry 3



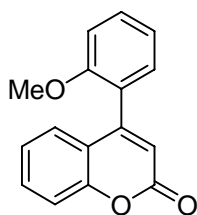
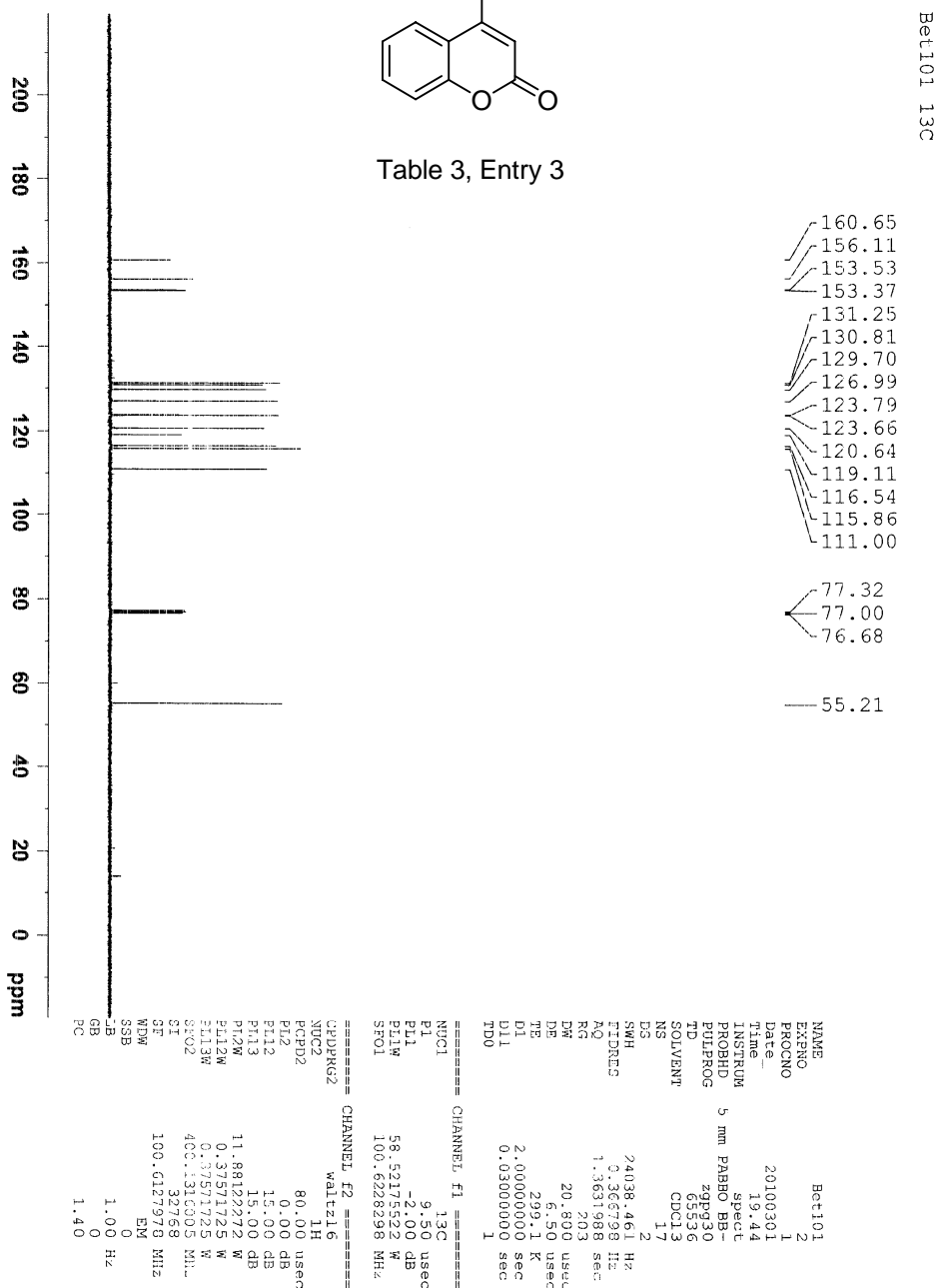


Table 3, Entry 3



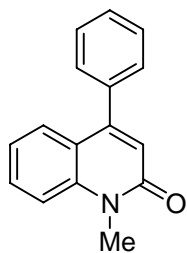
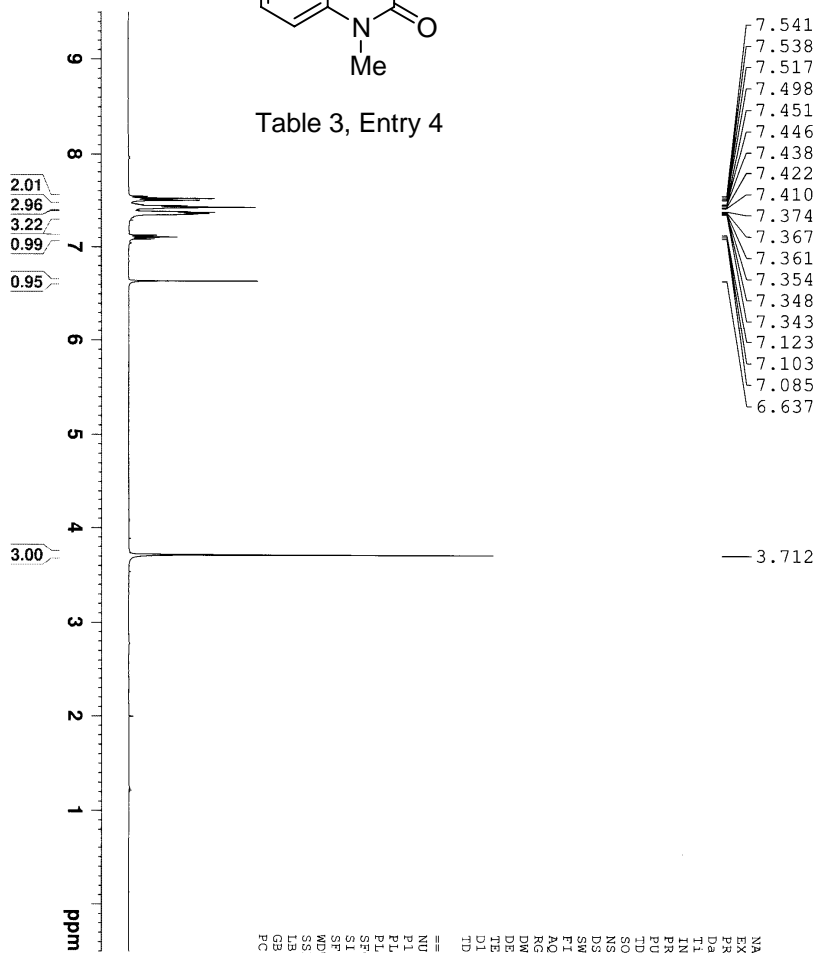


Table 3, Entry 4



Bec063

- 7.541
- 7.538
- 7.517
- 7.498
- 7.451
- 7.446
- 7.438
- 7.422
- 7.410
- 7.374
- 7.367
- 7.361
- 7.354
- 7.348
- 7.343
- 7.123
- 7.103
- 7.085
- 6.637
- 3.712

```

NAME          Bec063
EXPNO         1
PROCNO        1
Date_         20100116
Time          15.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4006.410 Hz
FWDRES        0.17226 Hz
AQ            4.0894966 sec
RG            16
DE            124.800 usec
TE            298.9 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            14.70 usec
PL1           0.00 dB
PL1W          11.8812272 W
SFO1          400.1318007 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

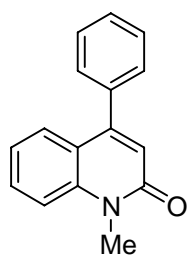
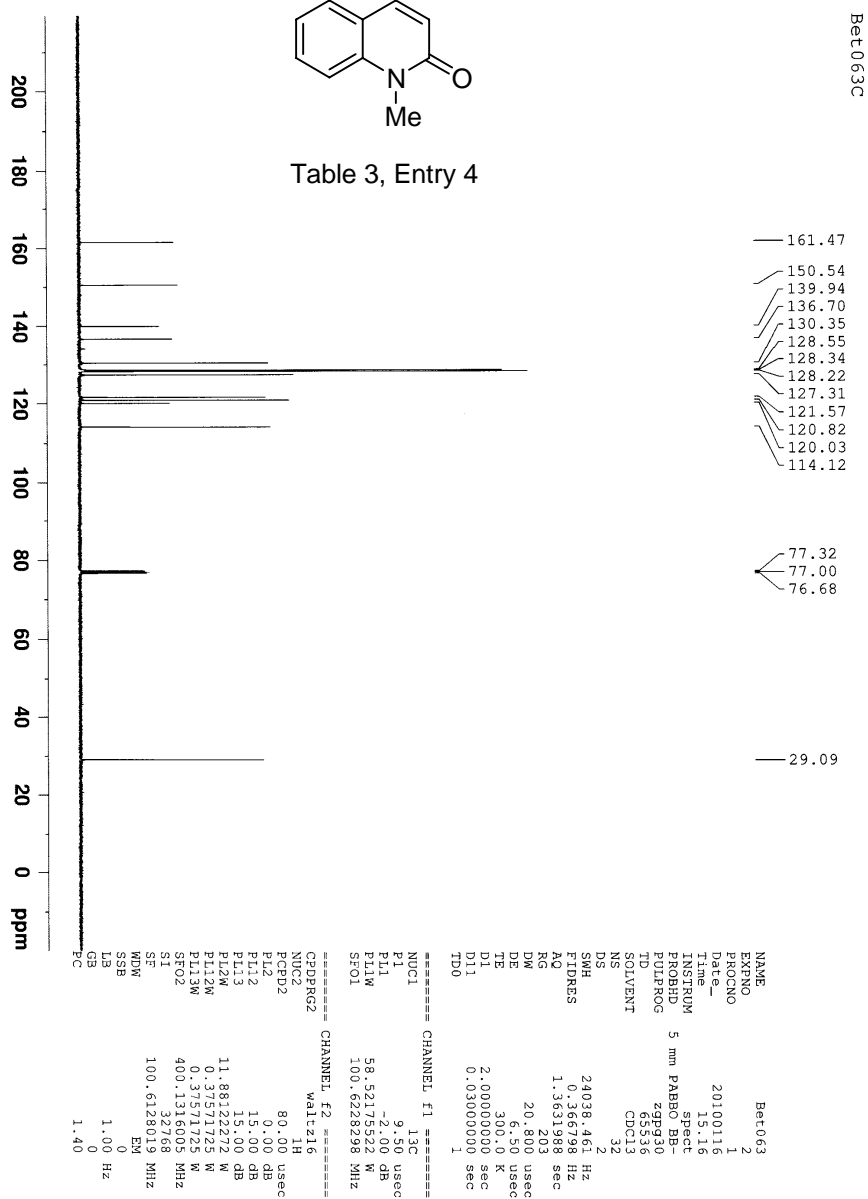



Table 3, Entry 4



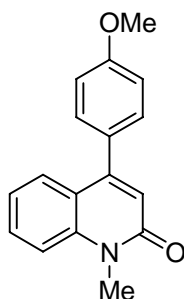
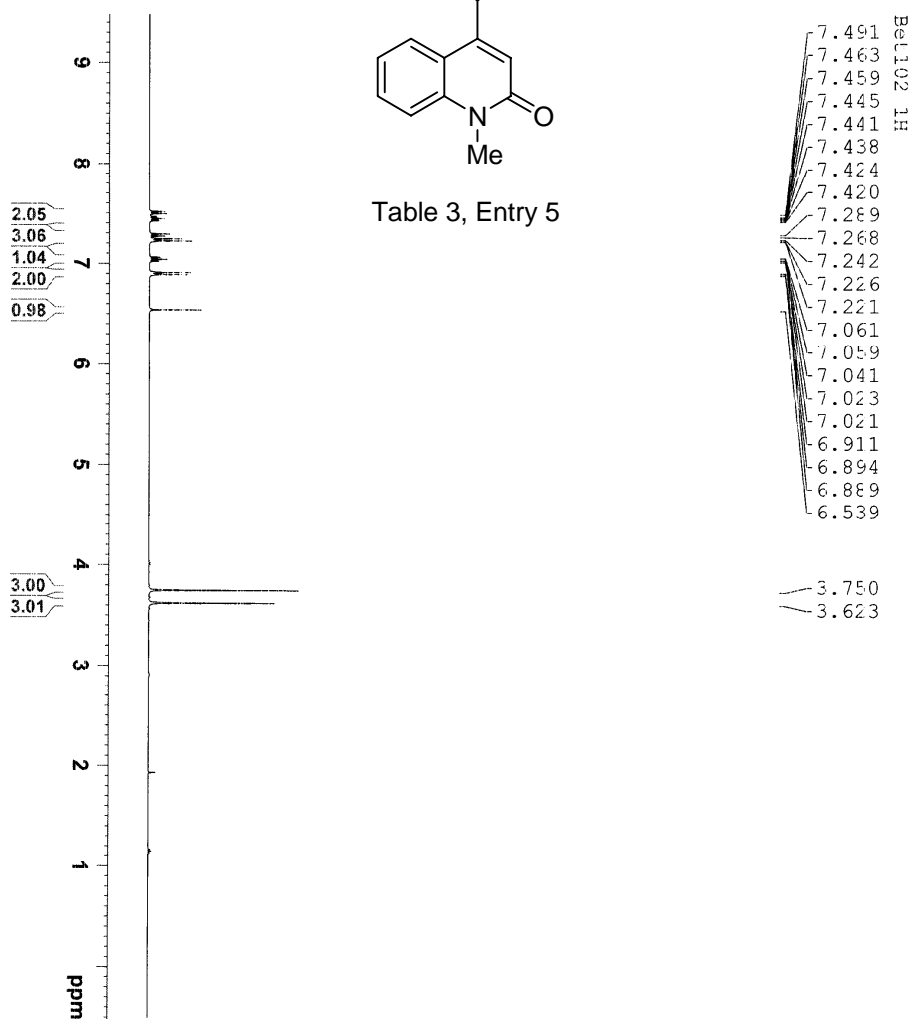


Table 3, Entry 5



Bel102 1H

7.491
 7.463
 7.459
 7.445
 7.441
 7.438
 7.424
 7.420
 7.289
 7.268
 7.242
 7.226
 7.221
 7.061
 7.059
 7.041
 7.023
 7.021
 6.911
 6.894
 6.869
 6.539

3.750
 3.623

```

NAME          Bel102
EXPNO         1
PROCNO        1
Date_         20100304
Time          15.44
INSTRUM       spect
PROBHD        5 mm PABBO B3-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            2
DS            8
SWH           4006.410 Hz
FIDRES       0.122266 Hz
AQ           4.0891966 sec
RG            9
DE           124.800 usec
TE           298.2 K
D1           1.00000000 sec
ID0          1

===== CHANNEL f1 =====
NUC1          1H
P1           14.76 usec
PL           0.00 dB
RG           11.8912202
SFO1         400.1352706 MHz
SF           400.1300000 MHz
WDW          EM
SSB          0
GB           0.30 Hz
PC           1.00
    
```

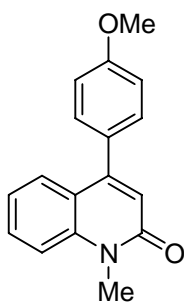
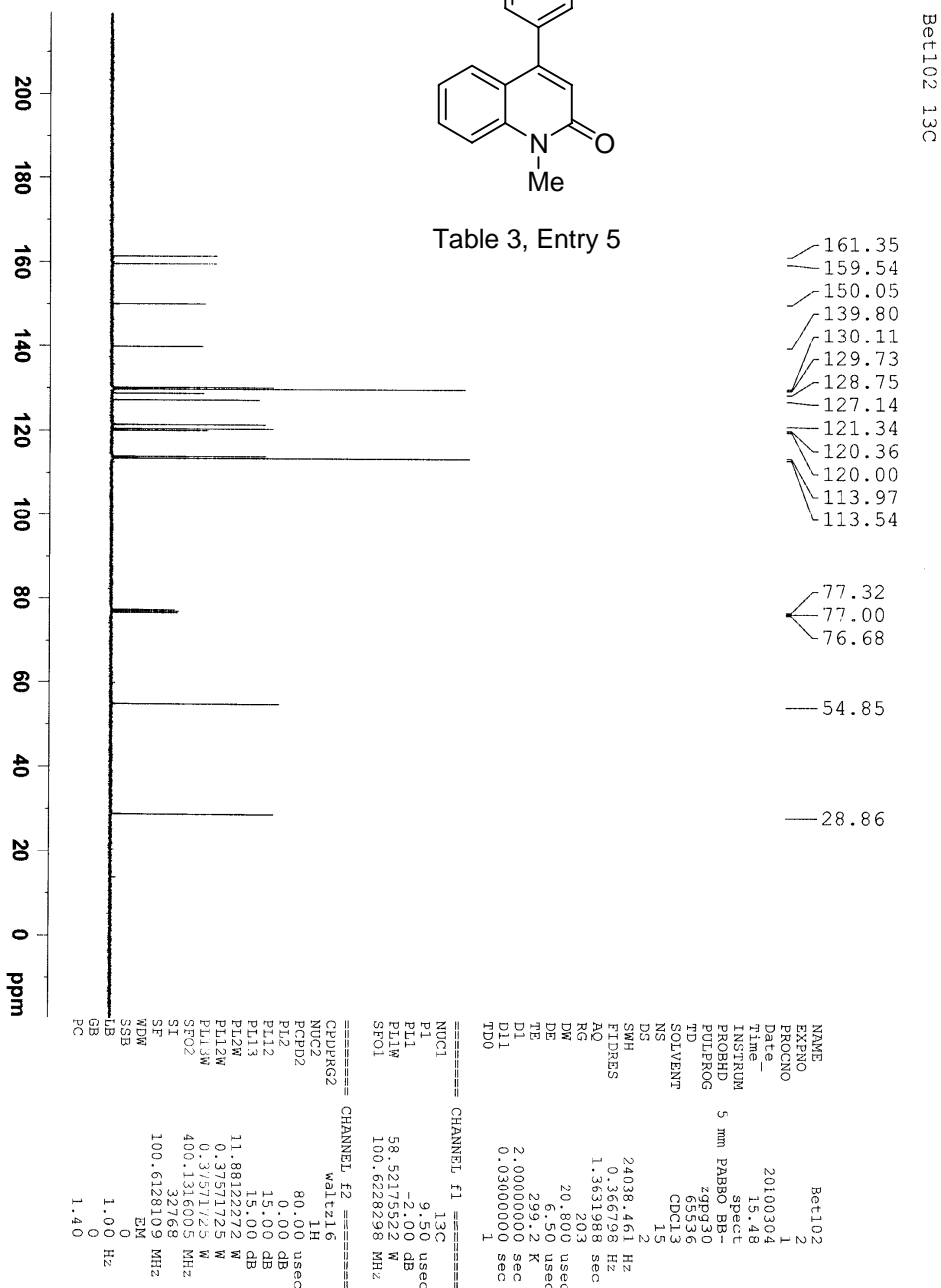


Table 3, Entry 5



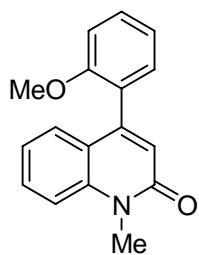
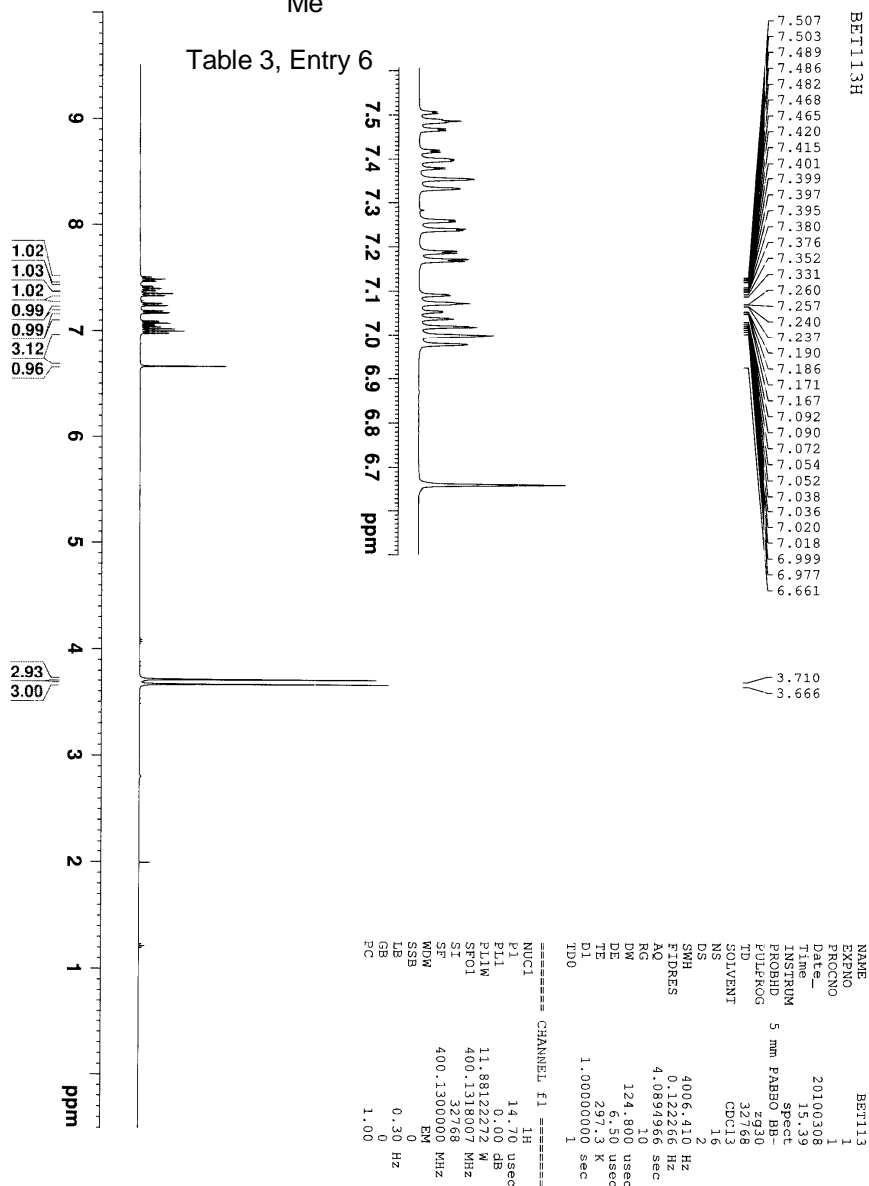


Table 3, Entry 6



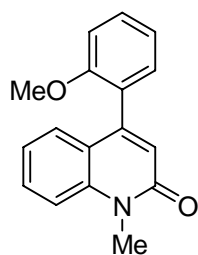
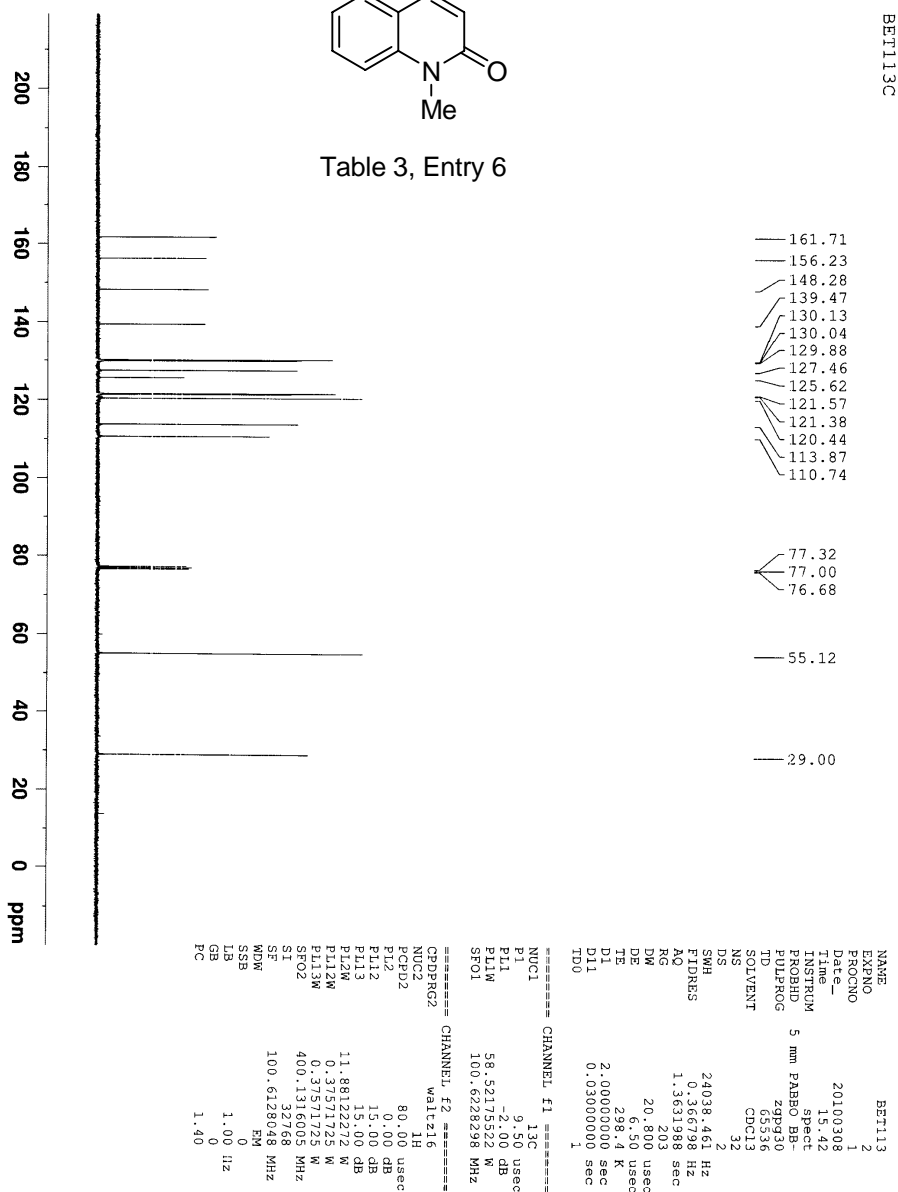


Table 3, Entry 6



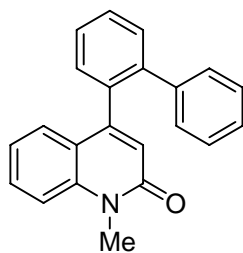
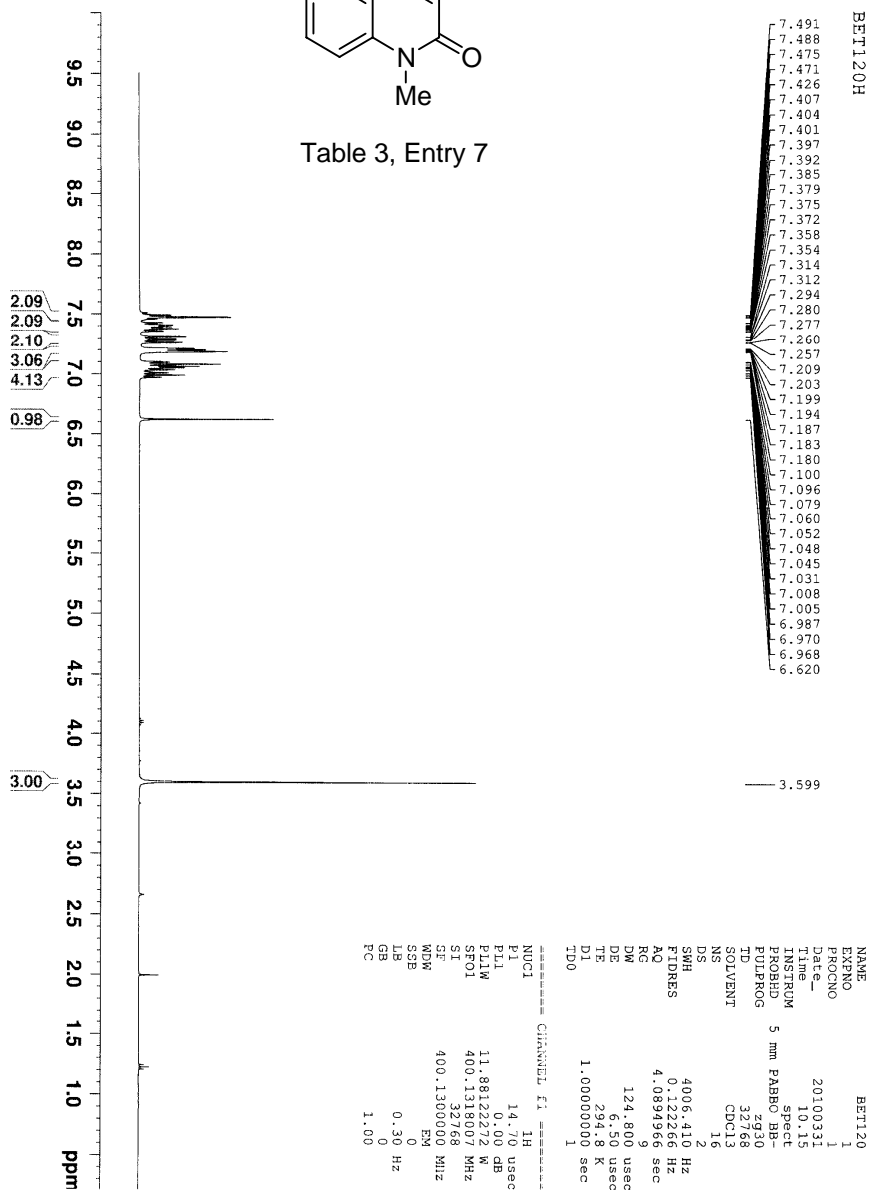


Table 3, Entry 7



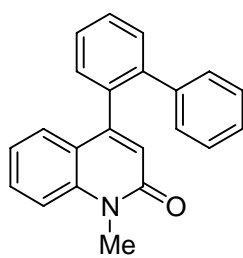
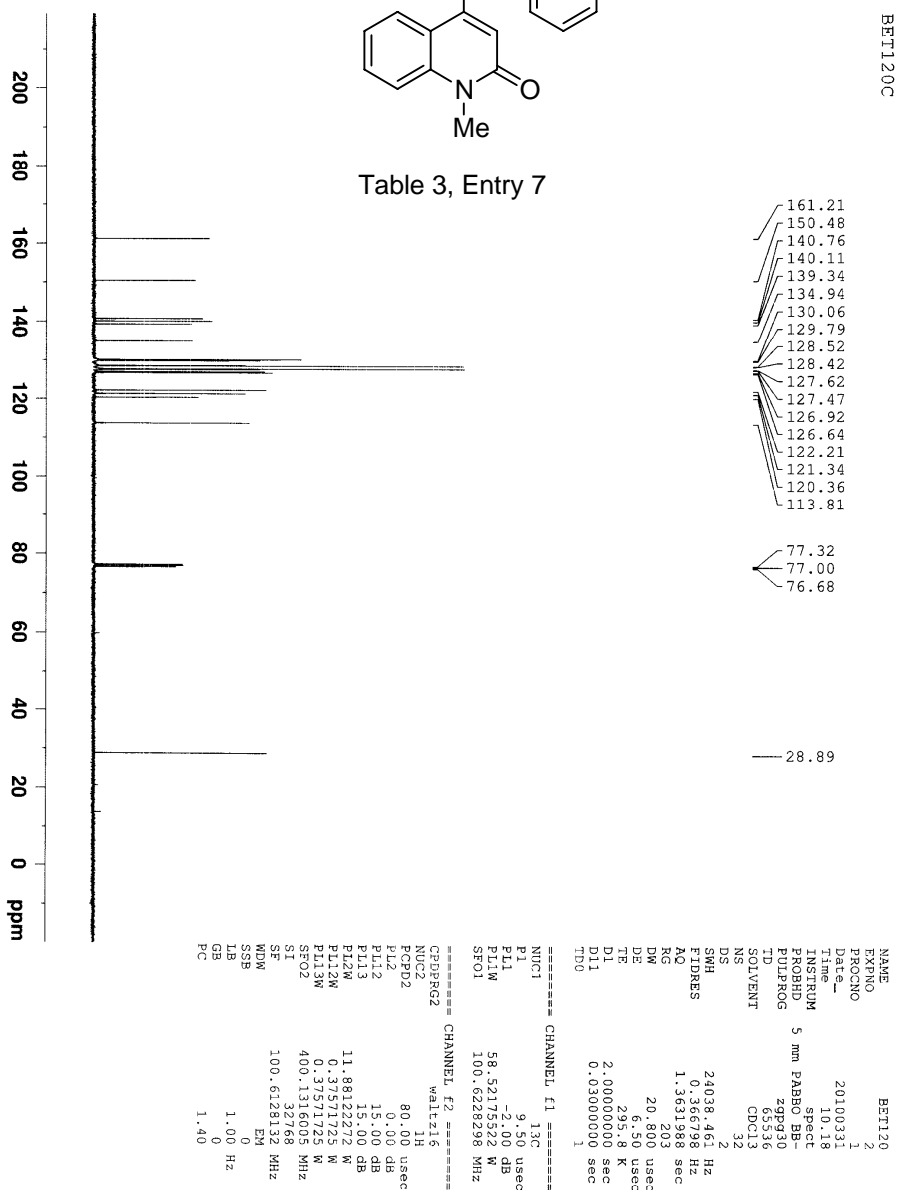


Table 3, Entry 7



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

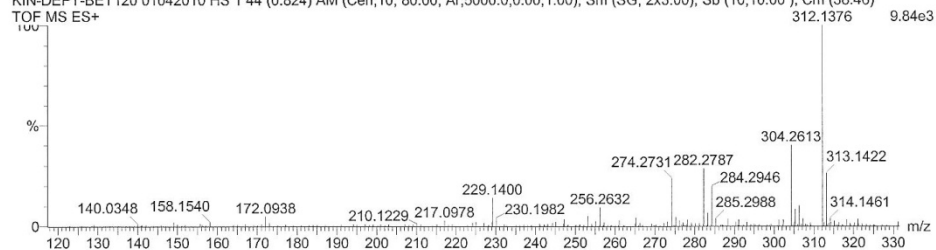
21 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-20 N: 0-3 O: 0-2

KIN-DEPT-BET120 01042010 HS 1 44 (0.824) AM (Cen,10, 80.00, Ar,5000.0.0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (38:46)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 20.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
312.1376	312.1388	-1.2	-3.8	14.5	7.3	C22 H18 N O

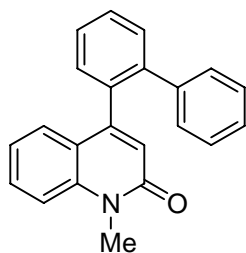


Table 3, Entry 7

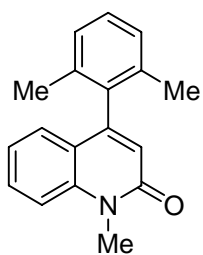
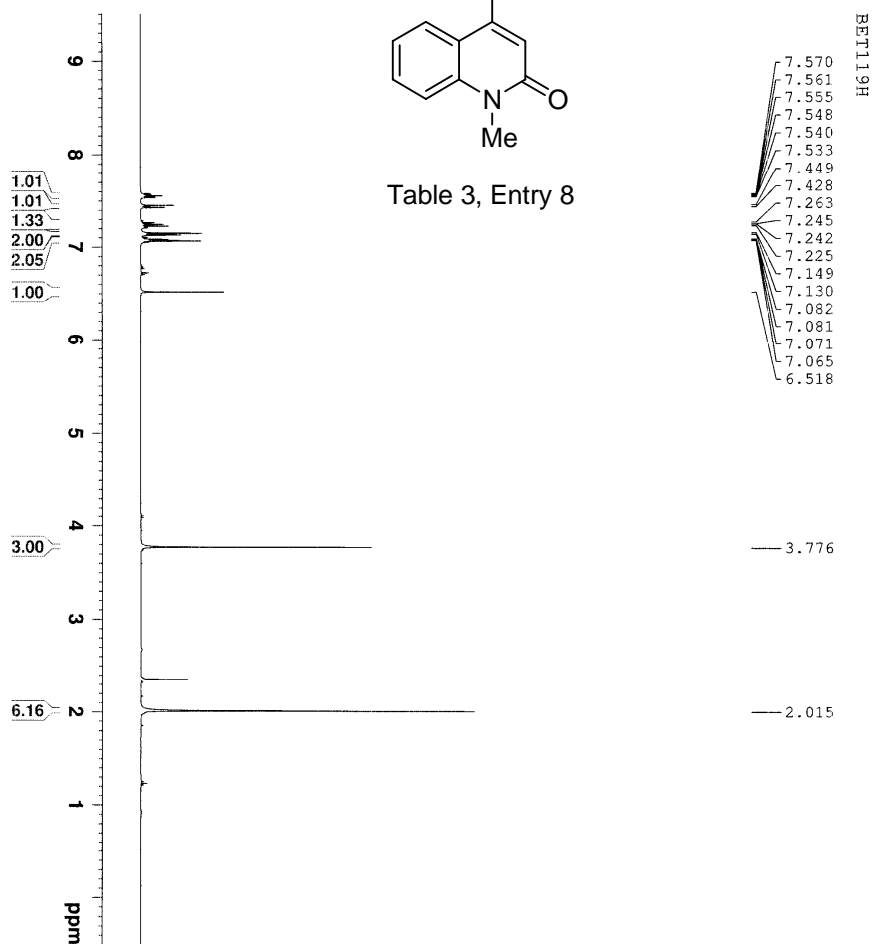


Table 3, Entry 8



BET119H

7.570
7.561
7.555
7.548
7.540
7.533
7.449
7.428
7.263
7.245
7.242
7.225
7.149
7.130
7.082
7.081
7.071
7.065
6.518

3.776

2.015

NAME	BET119
EXPNO	1
PROCNO	1
Date_	20100331
Time	10.08
INSTRUM	5 mm PABBO RH-
PROBHD	spec1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4006.411 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	9
DM	124.800 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	13H
P1	14.71 usec
PL1	0.00 dB
PL1W	11.88122272 W
SFO1	400.1318007 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

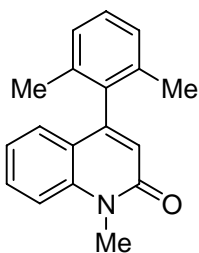
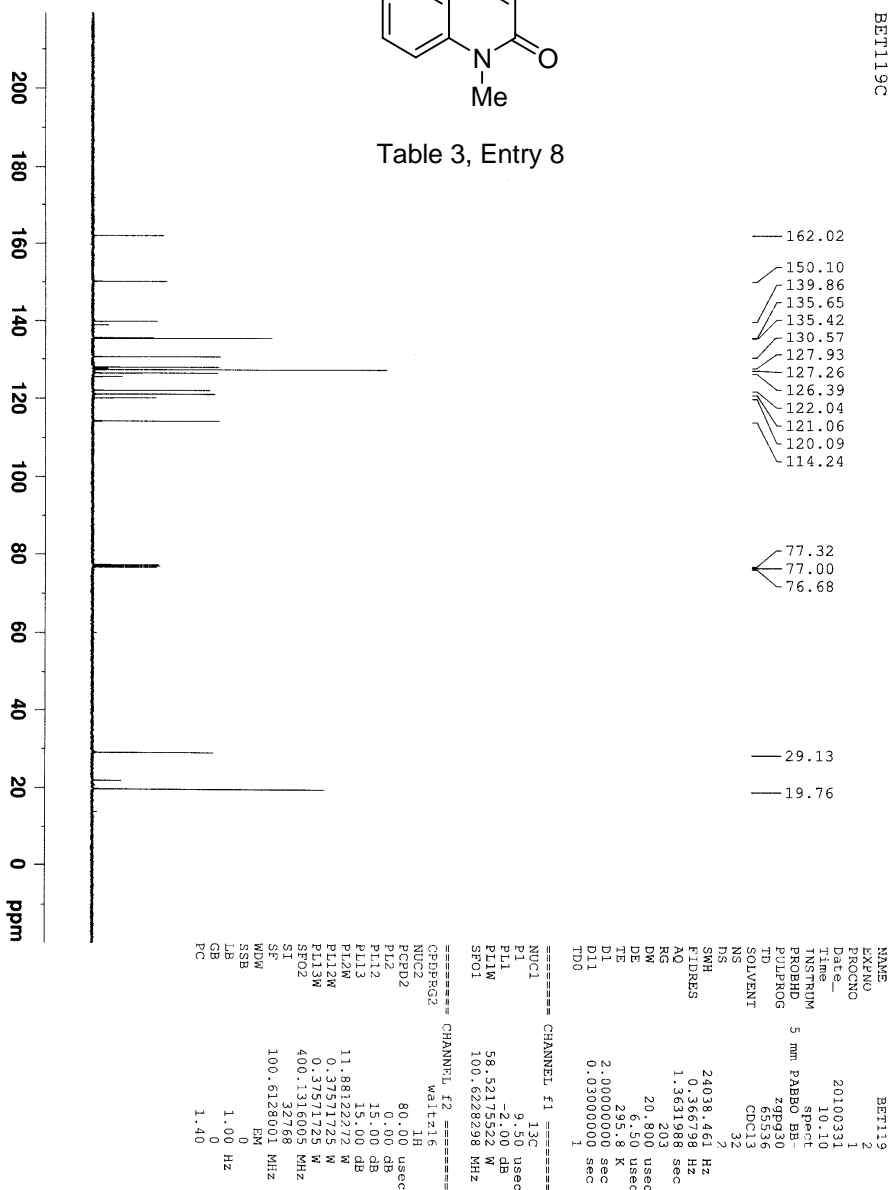


Table 3, Entry 8



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

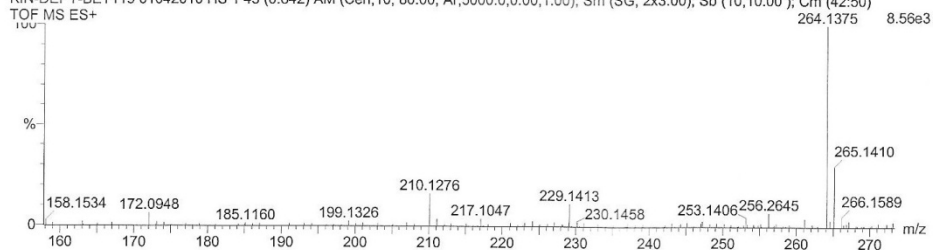
20 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-20 H: 0-20 N: 0-3 O: 0-2

KIN-DEPT-BET119 01042010 HS 1 45 (0.842) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (42:50)

TOF MS ES+



Minimum: -1.5
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
264.1375	264.1388	-1.3	-4.9	10.5	155.8	C18 H18 N O

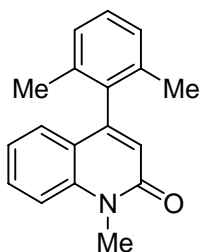


Table 3, Entry 8

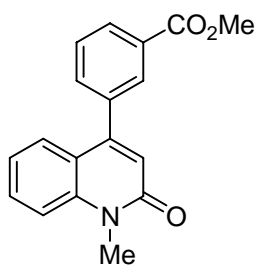
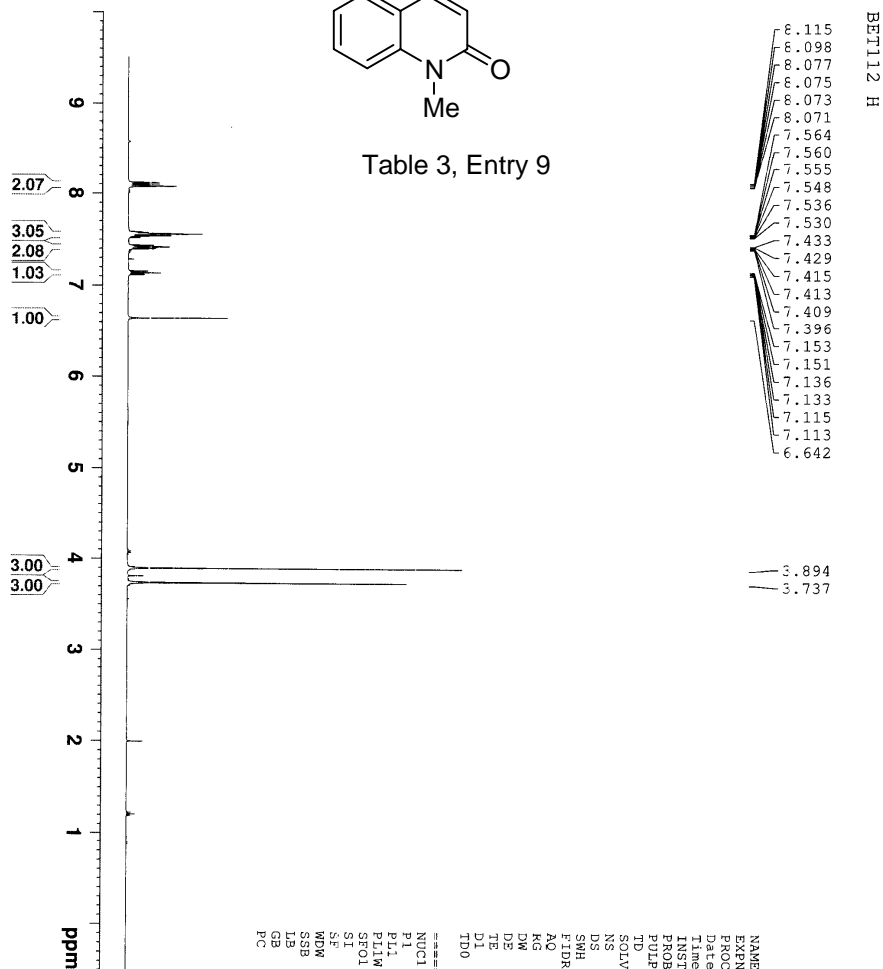


Table 3, Entry 9



```

NAME          BET112
EXPNO         1
PROCNO        1
DATE_         20100301
TIME         14.47
INSTRUM       5 mm PABBO BB-
PROBHD        spect
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            16
DS            2
SWH           4005.410 Hz
FIDRES       0.124268 Hz
RG           4.089226
RG           124.800
DE           6.50 usec
TE           297.1 K
D1           1.00000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1          1H
P1           14.70 usec
PL1          0.00 dB
PL12         11.8812272 dB
SFO1         400.1318607 MHz
SI           32768
SF           400.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```

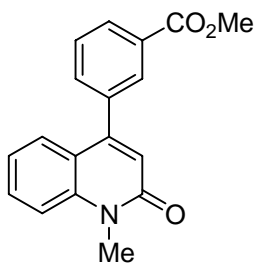
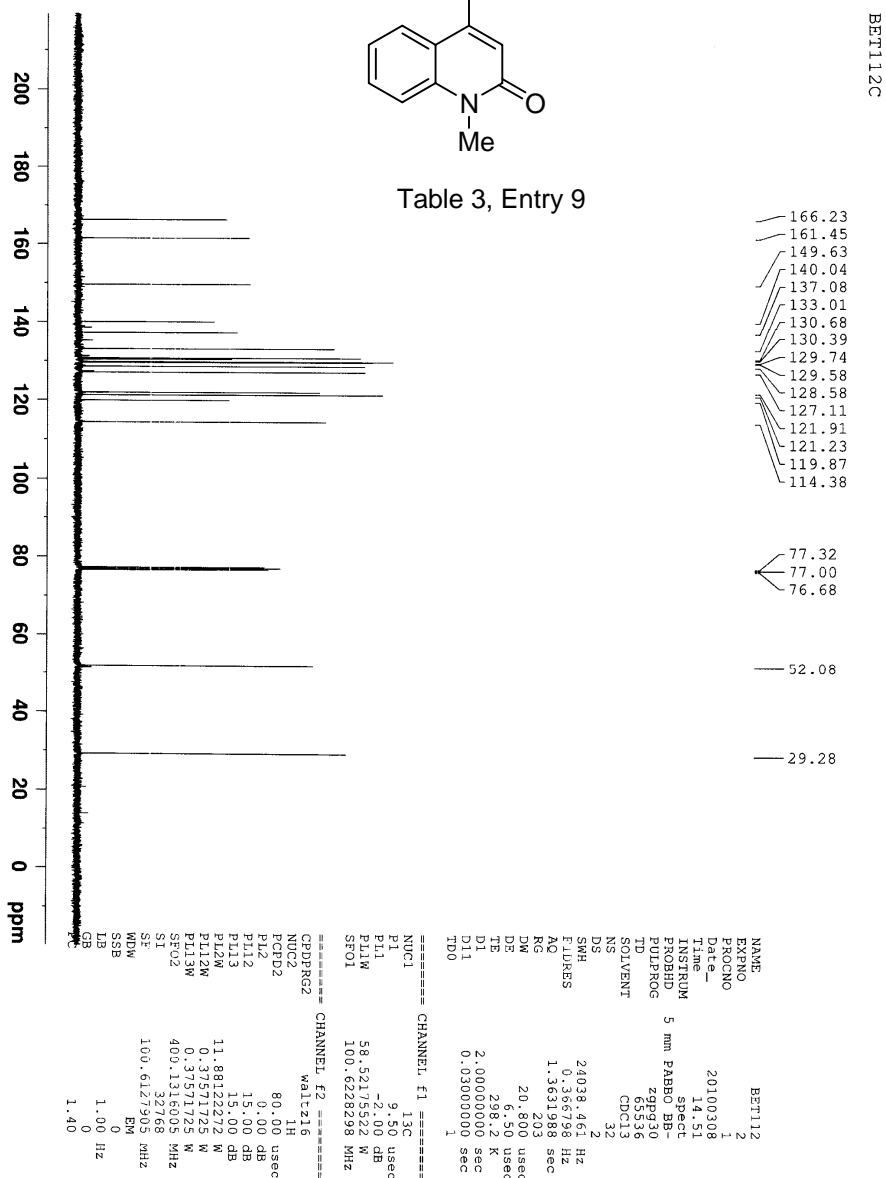


Table 3, Entry 9



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

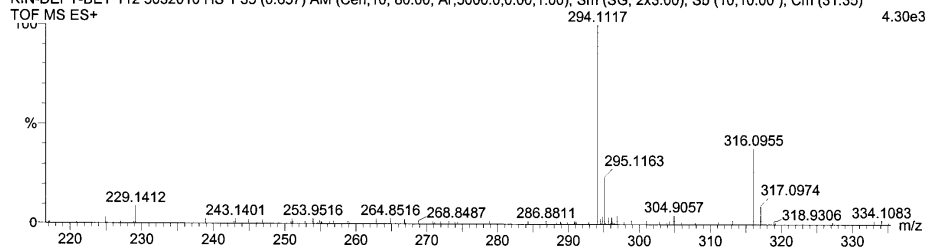
74 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-22 H: 0-25 N: 0-5 O: 0-5

KIN-DEPT-BET 112 5032010 HS 1.35 (0.657) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (31:35)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
294.1117	294.1130	-1.3	-4.4	11.5	15.1	C18 H16 N O3

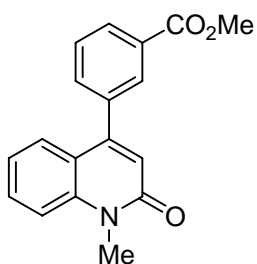


Table 3, Entry 9

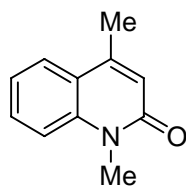
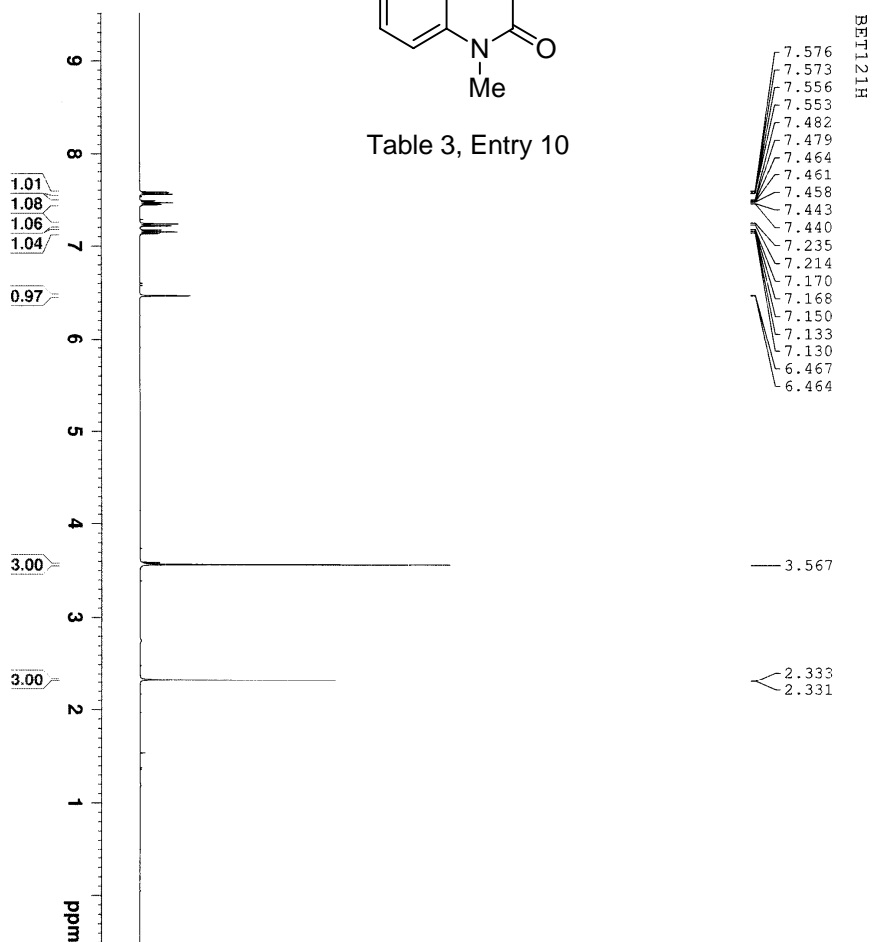


Table 3, Entry 10



BET121H

7.576
7.573
7.556
7.553
7.482
7.479
7.464
7.461
7.458
7.443
7.440
7.235
7.214
7.170
7.168
7.150
7.133
7.130
6.467
6.464

3.567

2.333
2.331

NAME	BET121
EXPNO	1
PROCNO	1
Date_	20100331
Time	10.23
INSTRUM	5 mm PABBO BB-
PROBHD	spect
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	12
DS	2
SWH	4006.410 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	18
DW	124.800 usec
DE	6.150 usec
TE	294.8 K
D1	1.00000000 sec
ID0	1

----- CHANNEL f1 -----	
NUC1	¹ H
P1	14.70 usec
PL1	0.00 dB
PL1M	11.88122272 W
SFO1	400.1318007 MHz
SI	32768
SE	400.1300000 MHz
MOR	EM
MSB	0
LSB	0.30 Hz
GB	0
PC	1.00

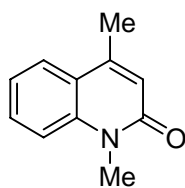
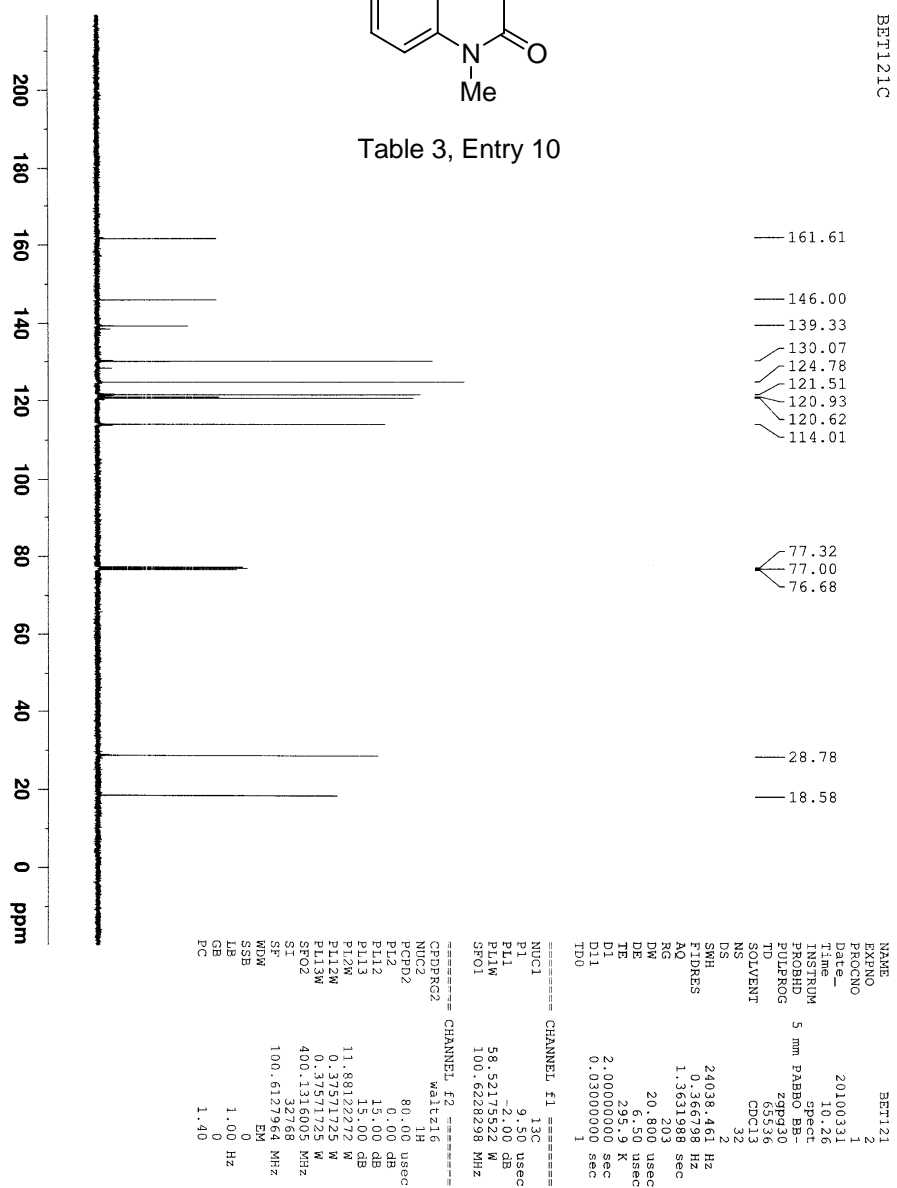


Table 3, Entry 10



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 6.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

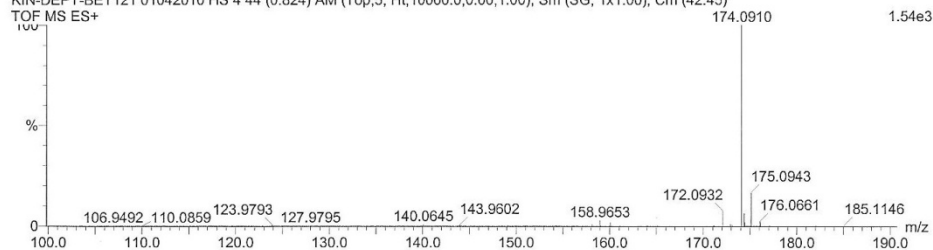
99 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-20 N: 0-3 O: 0-6 Na: 0-1

KIN-DEPT-BET121 01042010 HS 4 44 (0.824) AM (Top,5, Ht,10000.0,0.00,1.00); Sm (SG, 1x1.00); Cm (42:45)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 6.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
174.0910	174.0919	-0.9	-5.2	6.5	7.1	C11 H12 N O

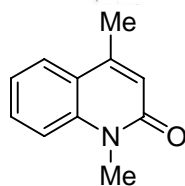


Table 3, Entry 10

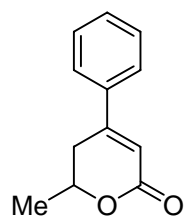
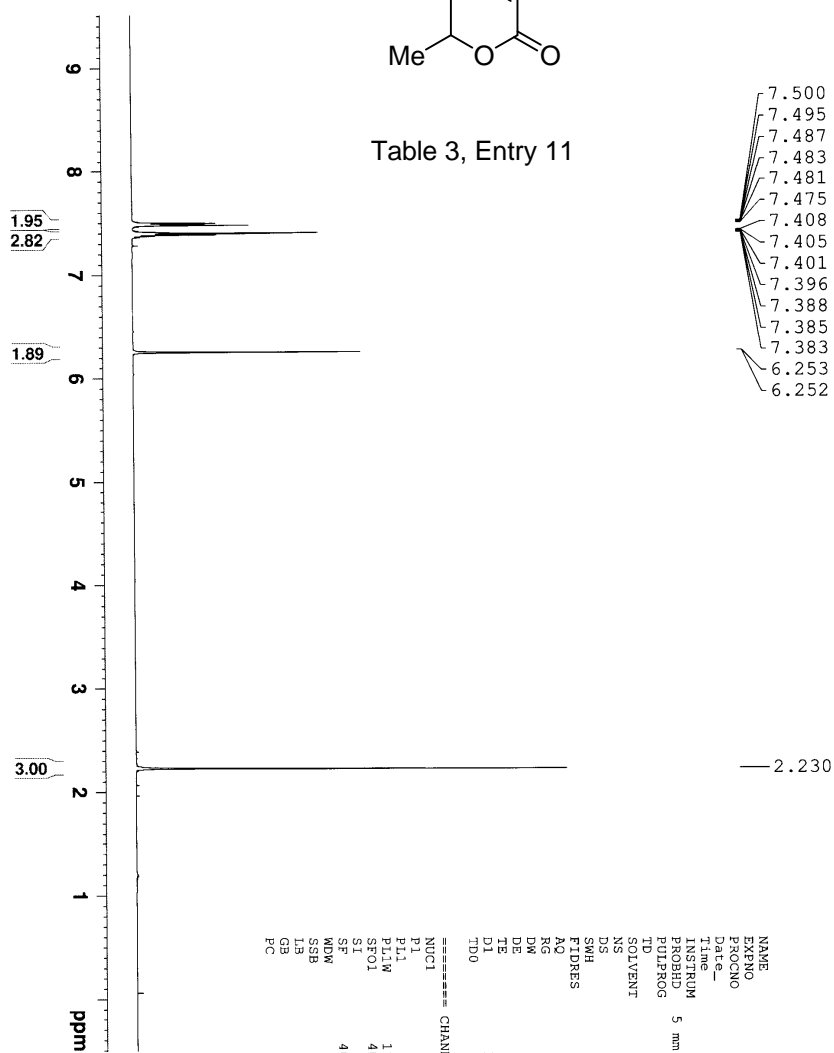


Table 3, Entry 11



```

NAME Bet065
EXPNO 1
PROCNO 1
Date_ 20100119
Time 18.45
INSTRUM spect
PROBHD BB-
PULPROG zgpg30
SOLVENT CDCl3
NS 16
DS 2
SWH 4006.410 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 18
DW 124.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
PL1W 11.88122272 W
SFO1 400.1318007 MHz
SI 32768
SF 400.1300000 MHz
MDW 0
SSB 0
GB 0.30 Hz
PC 1.00
    
```

Bet065

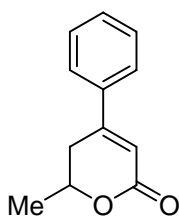
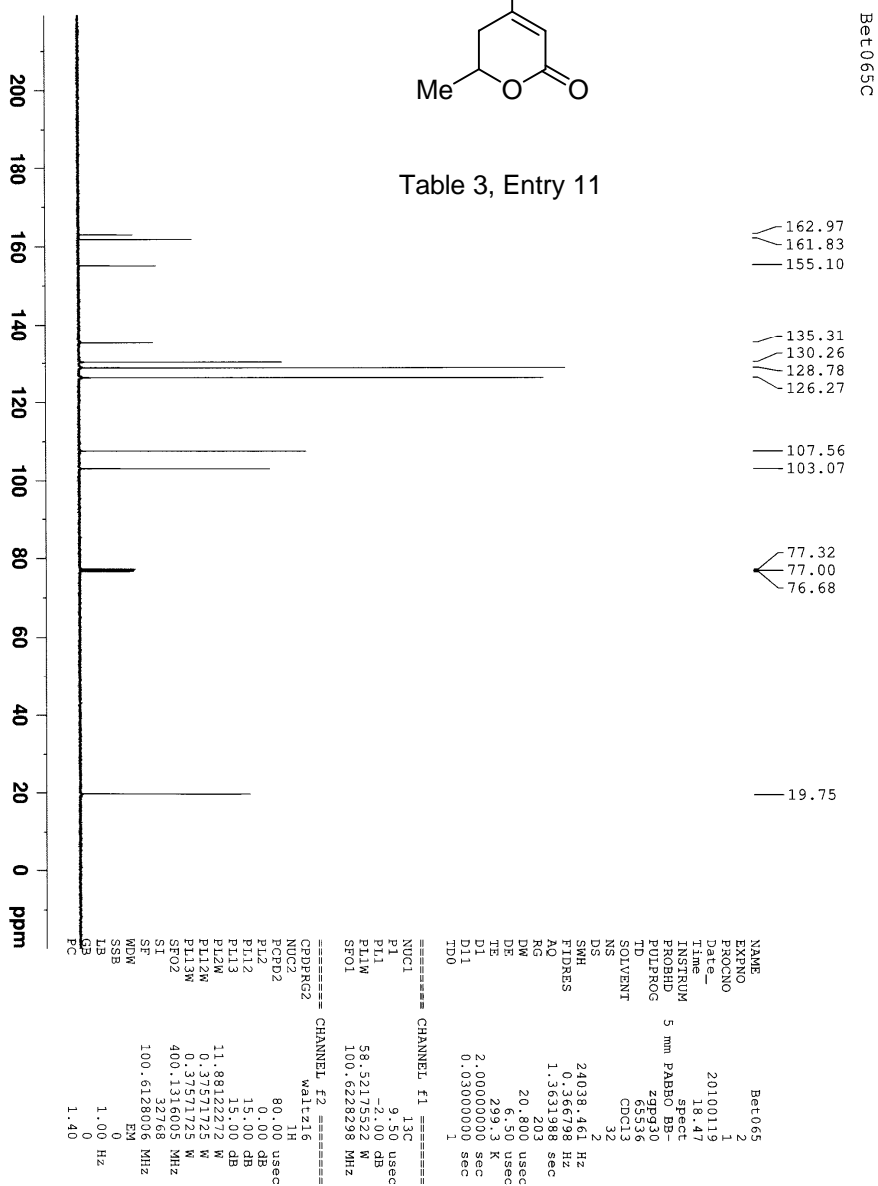


Table 3, Entry 11



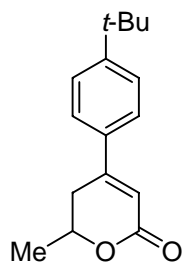
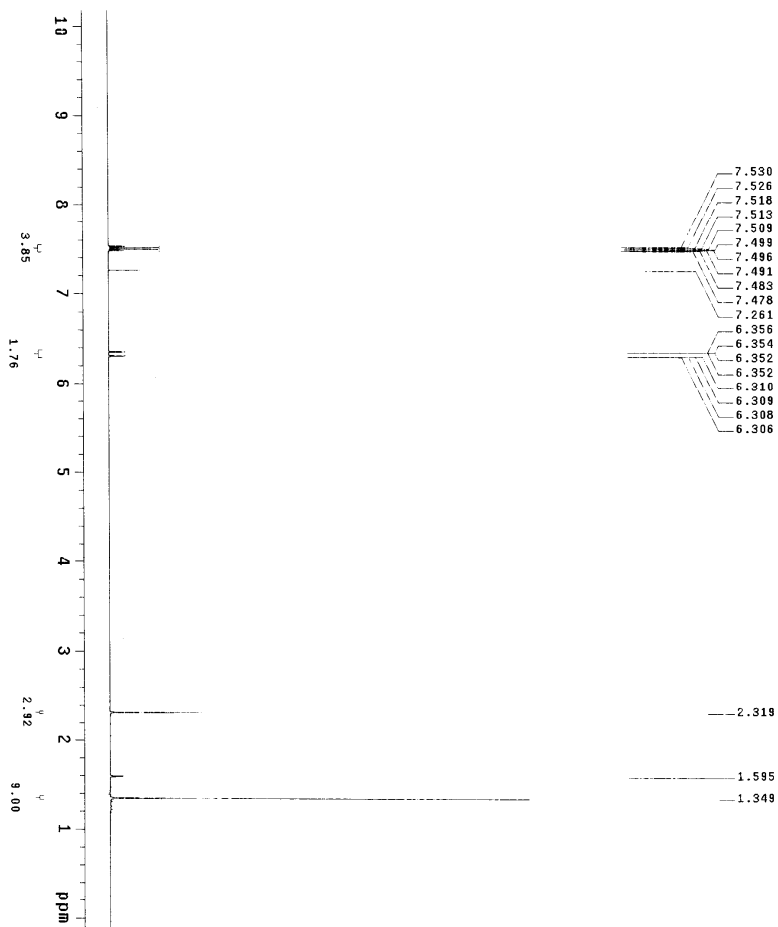


Table 3, Entry 12

```

BET037
expt s2pu1
SAMPLE
date Nov 13 2009 1:amp not used
solvent CHCl3 gain not used
f1 c acquisition exp cpin not used
sw 9996.0 Hz 0.000
ct 2.049 d1td 6.800
pp 32782 f1
hb 4002 f2
ls 2 f3
se 2 dp
d1 1.000 h5
nt 8 f6
nt TRANSMITTER 8 f6
f1 493.942 H1 SP DISPLAY 63.2
sftq 493.942 Hz 5151.2
lpwr 57 F11 399.6
pw 4.250 FFD 64.6
DECOUPLER C13 TP 8.4
NOF 0 nni vC 180
dm 5 c cc 9
dmn 5 c cc 9
dm 5 c cc 9
dmf 13786 f4
di cdc ph 1
    
```



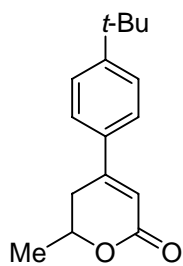
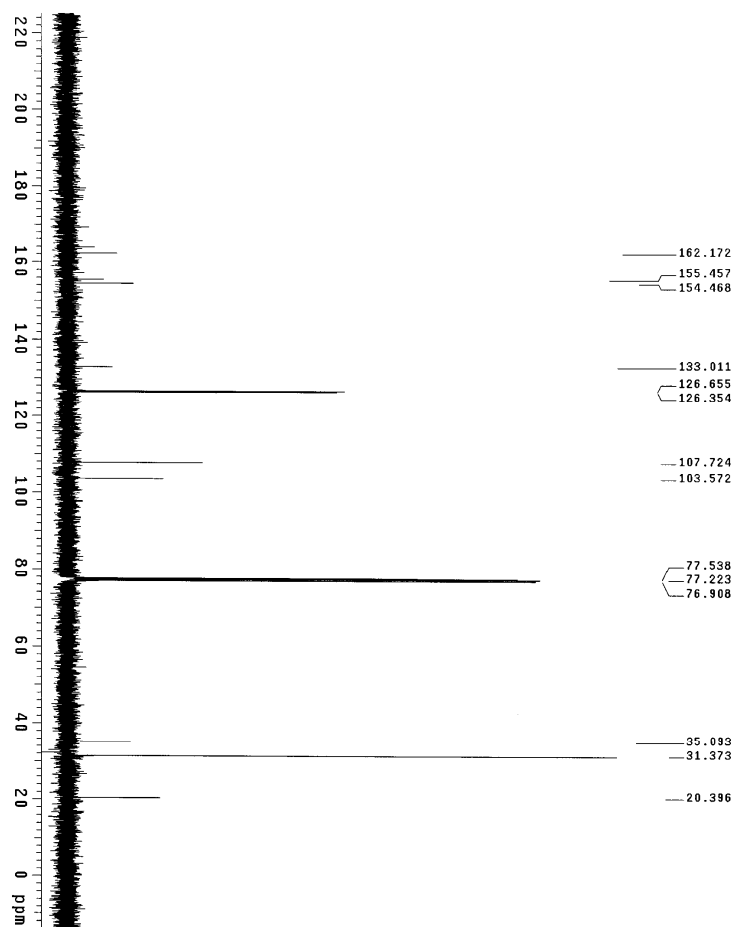


Table 3, Entry 12

```

BET1037
exp1 Carbon
SAMPLE
date nov 13 2008 temp SPECIAL
solvent CDCl3 exp not used
file G0013 exp not used
ACQUISITION exp not used
sv 21.18.2 hst 0.008
at 1.300 atfe 10.000
np 62730 flags
fd 13000 11
ds 1.000 dn
d1 1.000 ds
nt 1000 hs
ct TRANSMITTER 320 PROCESSING 0.50
tn TRN C13 fn not used
sfrq 100.518 sp DISPLAY
tof 1042.4 wf -1505.1
tpr 4.536 wf1 2150.4
pw DECOUPLER H1 TP 1505.0
dn 0 H1 TP -154.5
ddr 0 yyy PLOT
ddp 0 w wc 180
dmm 42 sc 1007
davr 11000 vs 0
dnt 4 at cdc ph
    
```



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

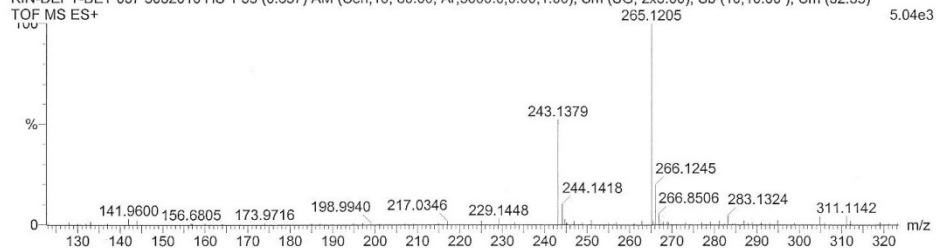
118 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-28 H: 0-45 N: 0-5 O: 0-2 Na: 0-1

KIN-DEPT-BET 037 5032010 HS 1 35 (0.657) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (32:35)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
243.1379	243.1385	-0.6	-2.5	7.5	1.5	C16 H19 O2

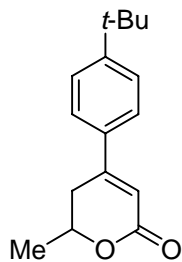


Table 3, Entry 12

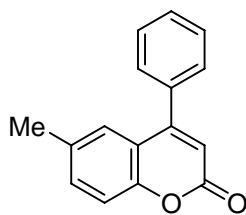
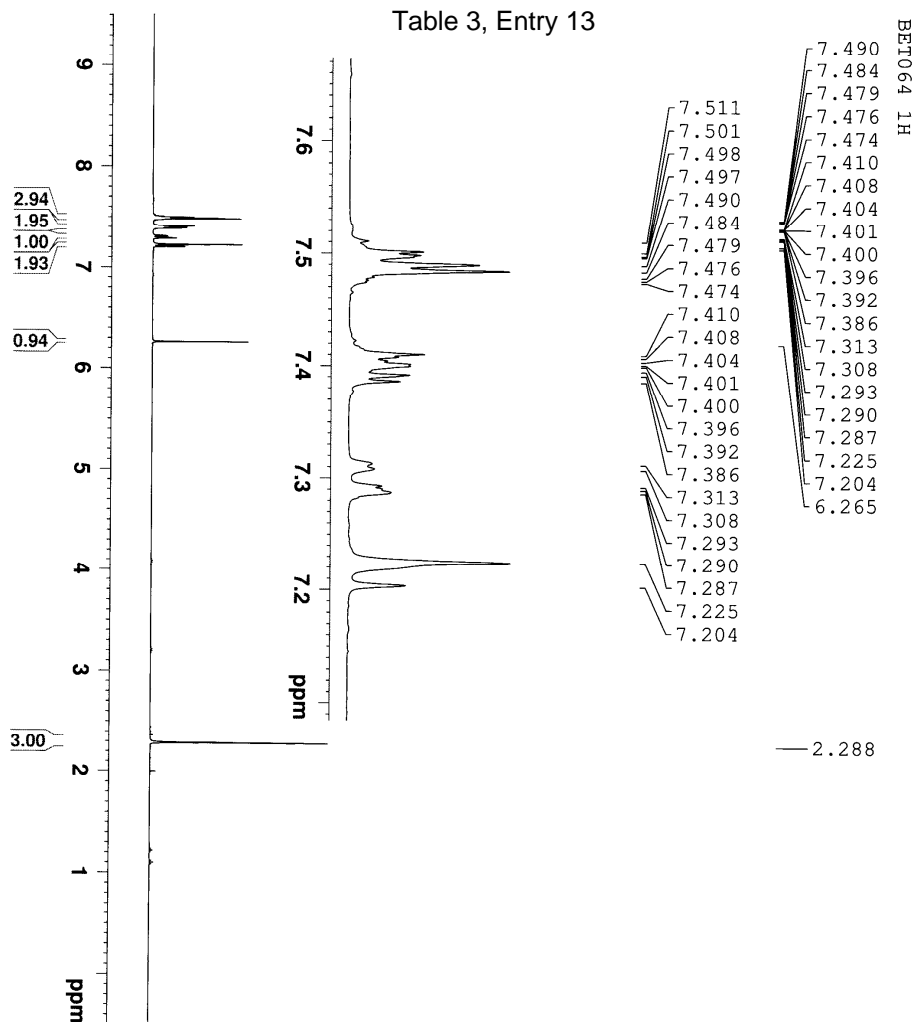


Table 3, Entry 13



```

===== CHANNEL F1 =====
NUC1  1H
P1    14.70 usec
PL1   0.00 dB
PL1W  11.88122272 W
SFO1  400.1318007 MHz
SI     32.768
SE     400.1300000 MHz
MDSW  EM
SSB   0
LB    0.30 Hz
GB    0
FC    1.00
=====
NAME      BET064
EXPNO    1
PROCNO   1
Date_    20100115
Time     17.35
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       32768
SOLVENT  CDCl3
NS       3
DS       2
SWH      4006.410 Hz
FIDRES   0.122265 Hz
AQ       4.0894966 sec
RG       10
DW       124.800 usec
DE       6.50 usec
TE       298.3 K
D1       1.00000000 sec
TD0      -
    
```

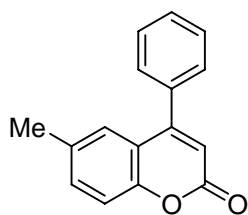
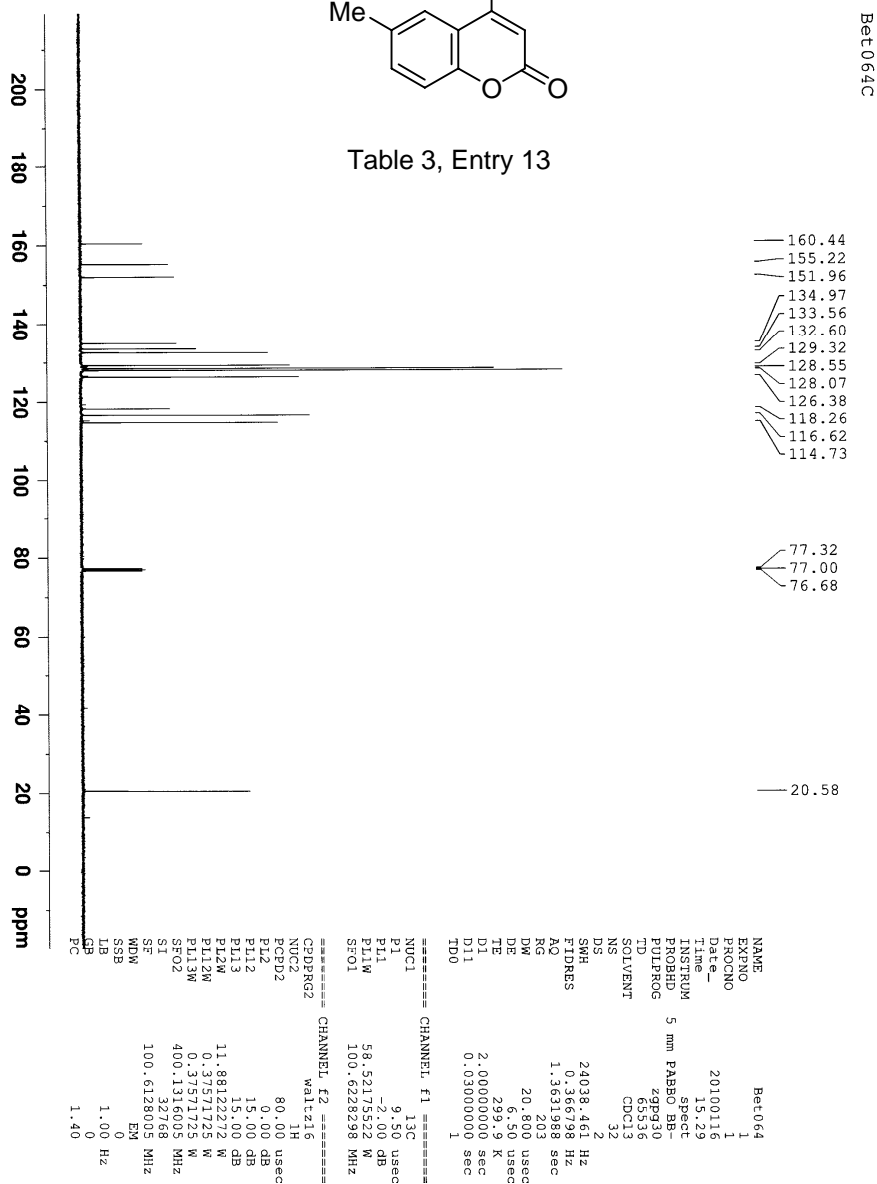
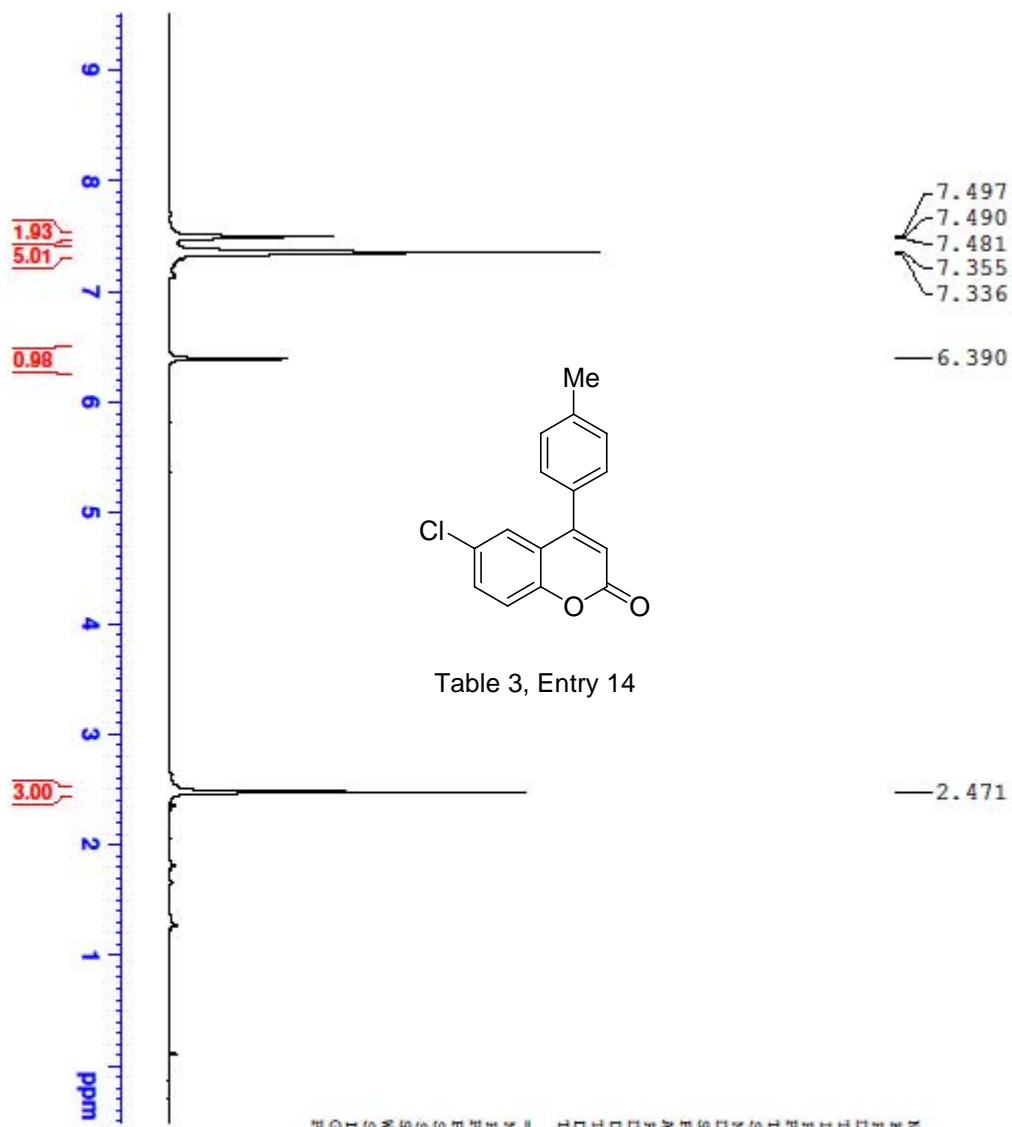


Table 3, Entry 13





B745

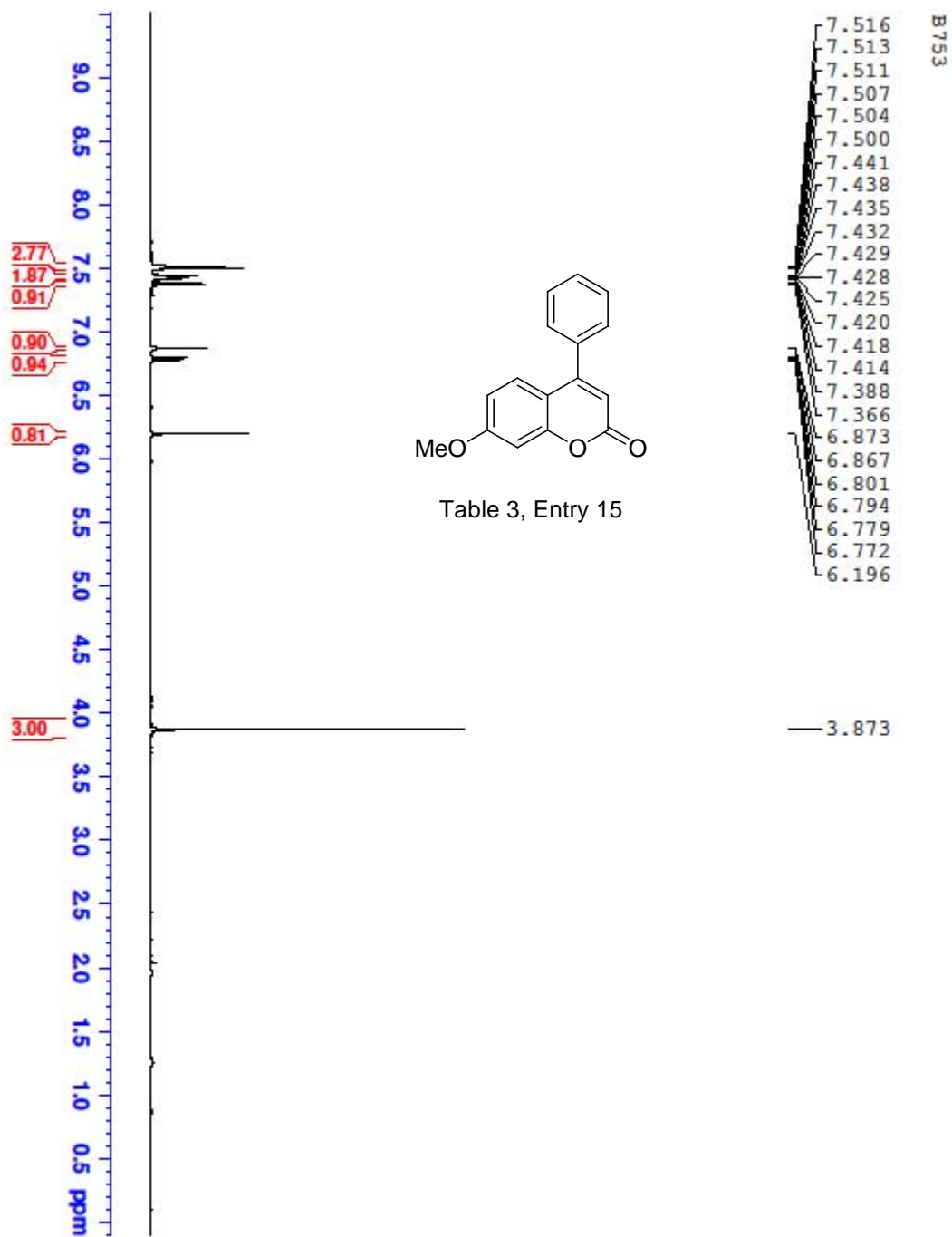
7.497
 7.490
 7.481
 7.355
 7.336
 — 6.390

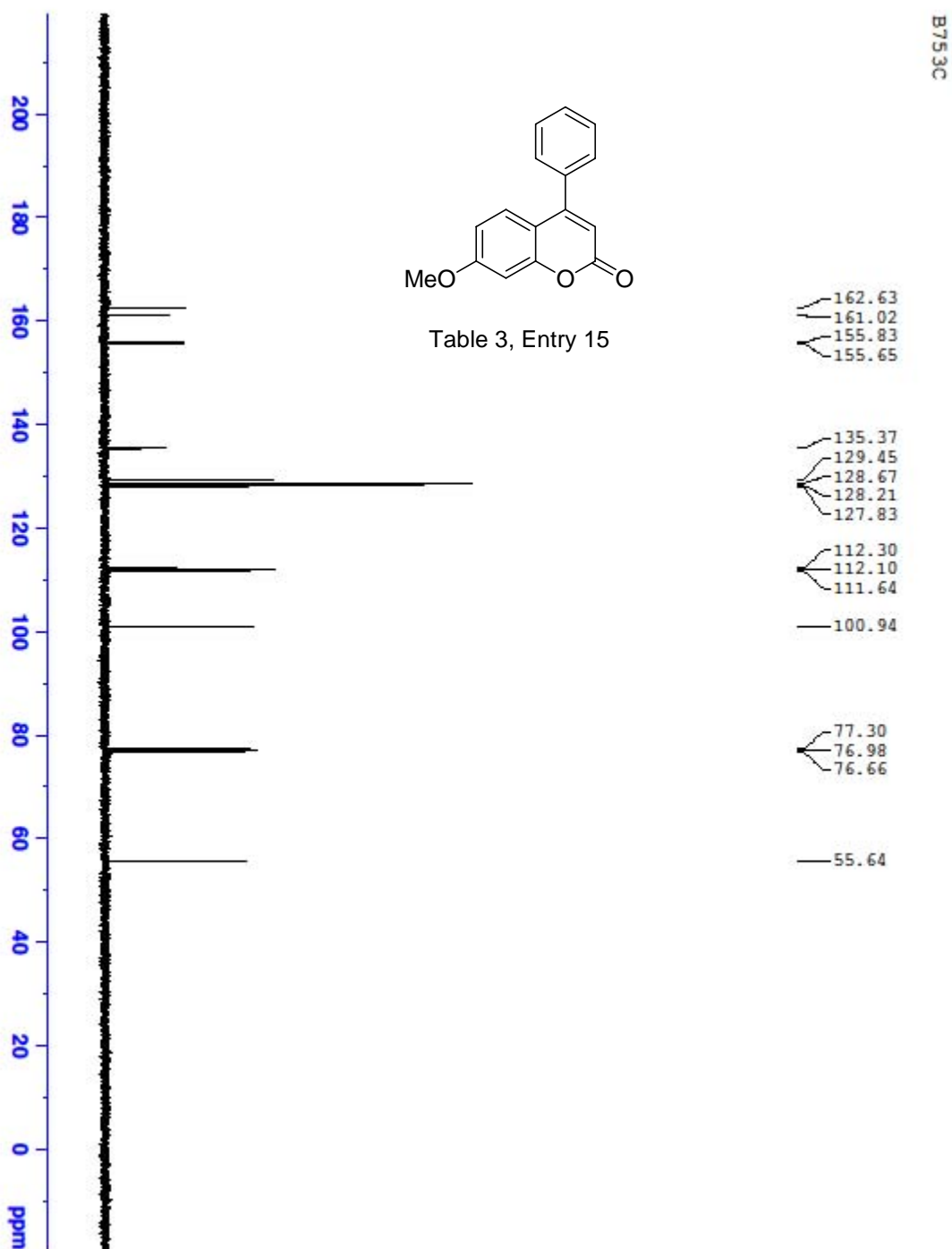
— 2.471

```

NAME          B745
EX PNO       1
PROCNO       1
Date_        20110309
Time         17.22
INSTRUM      5 mm PABBO BB-
PROBHD       zg30
PULPROG      zgpg30
TD           32768
SOLVENT      CDCl3
NS           16
DS           2
SWH          4006.410 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG           1
RG           1
DE           124.800 usec
TR           6.50 usec
TE           299.4 K
D1           1.00000000 sec
TD0          1

===== CHANNEL F1 =====
NUC1         1H
P1           14.79 usec
PL1         0.00 dB
PL1W        11.88122272 W
SFO1         400.1318007 MHz
SI           32768
SF           400.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```



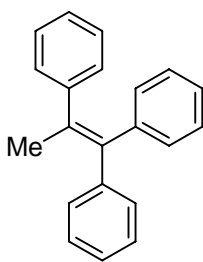
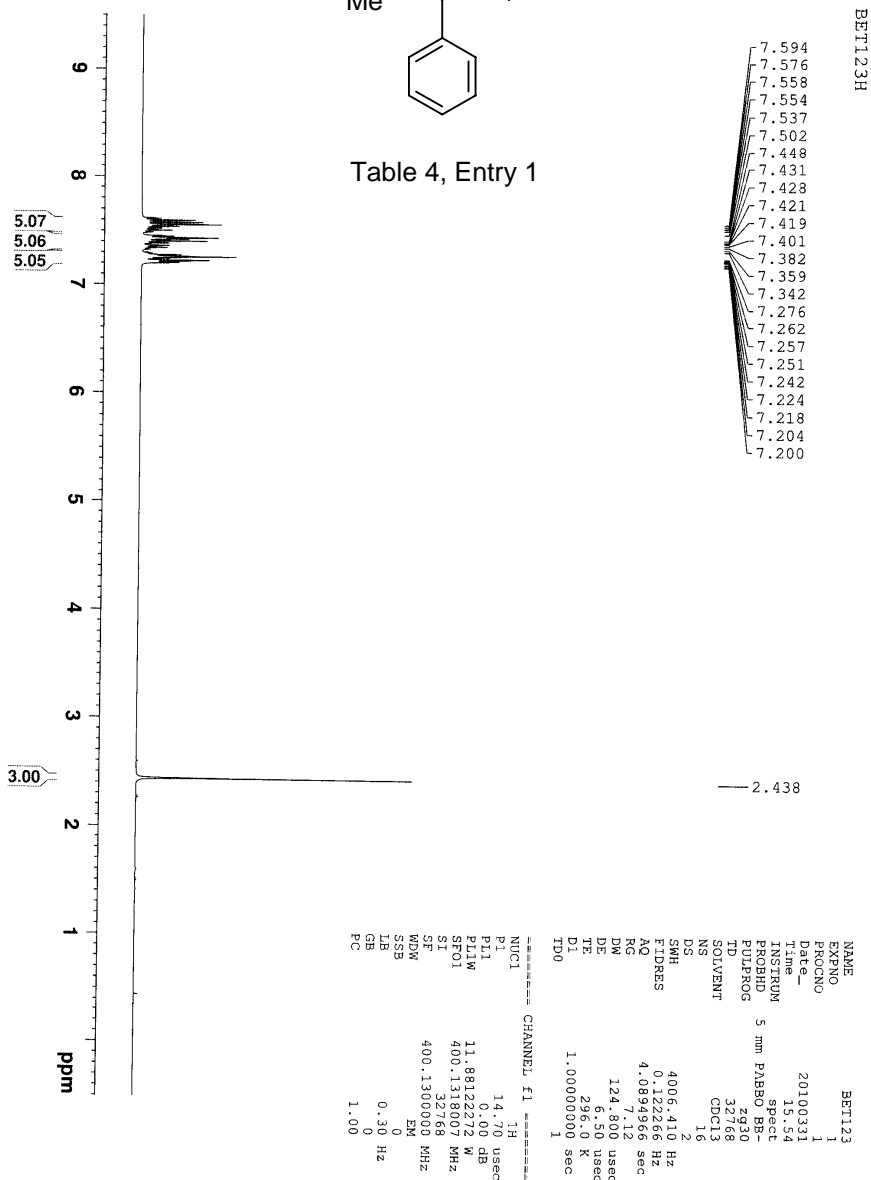


Table 4, Entry 1



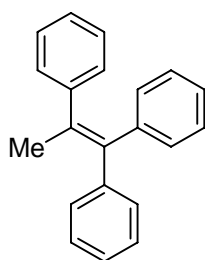
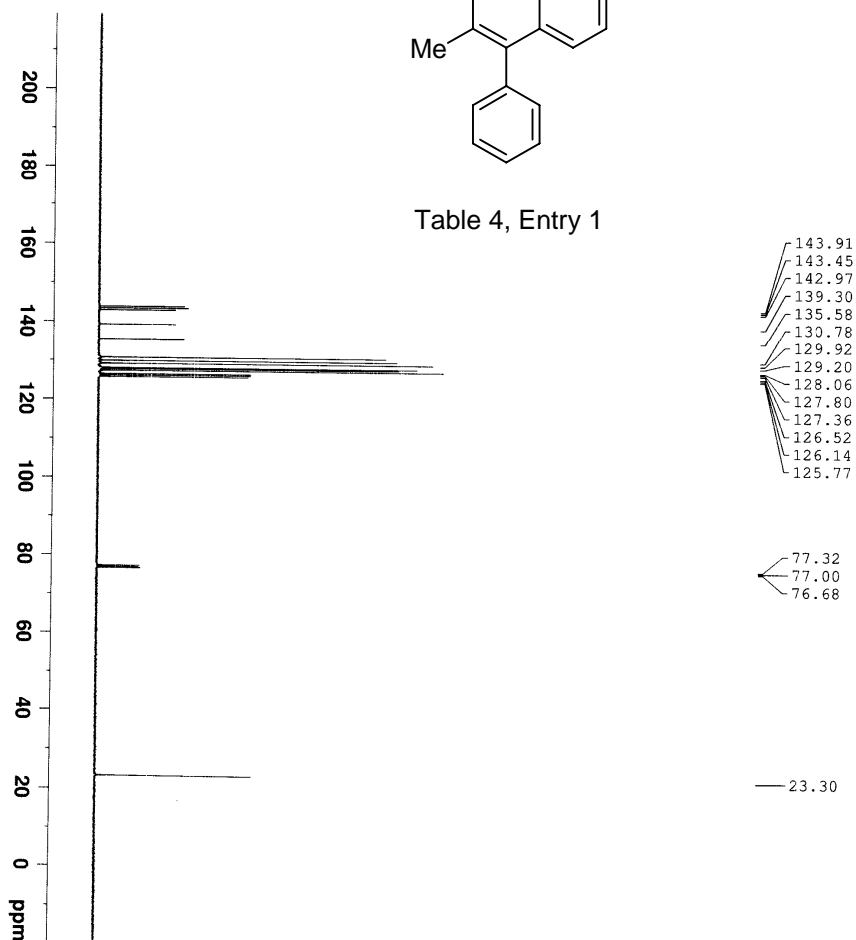


Table 4, Entry 1



BET123C

143.91
 143.45
 142.97
 139.30
 135.58
 130.78
 129.92
 129.20
 128.06
 127.80
 127.36
 126.52
 126.14
 125.77

77.32
 77.00
 76.68

23.30

```

NAME          BET123
EXPNO         2
PROCNO        1
Date_         20100331
Date_         16.01
INSTRUM       spect
PROBHD        5 mm PABP1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            32
DS            2
SWH           24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG           144
RG           144
DR           20.800 usec
TR           28.50 usec
TE           29.50 usec
D1           2.0000000 sec
D11          0.0300000 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1           9.50 usec
PL1          -2.00 dB
PL12         58.52175322 W
SFO1         100.6228298 MHz

===== CHANNEL f2 =====
CHDRFG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2          0.00 dB
PL12         15.00 dB
PL13         15.00 dB
PL23         11.88122272 W
PL24         0.33571725 W
PL14W        0.33571725 W
SFO2         400.1314025 MHz
SI           32768
SF           100.6128065 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
    
```

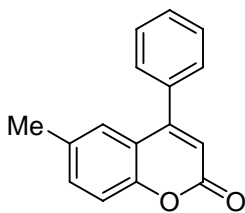
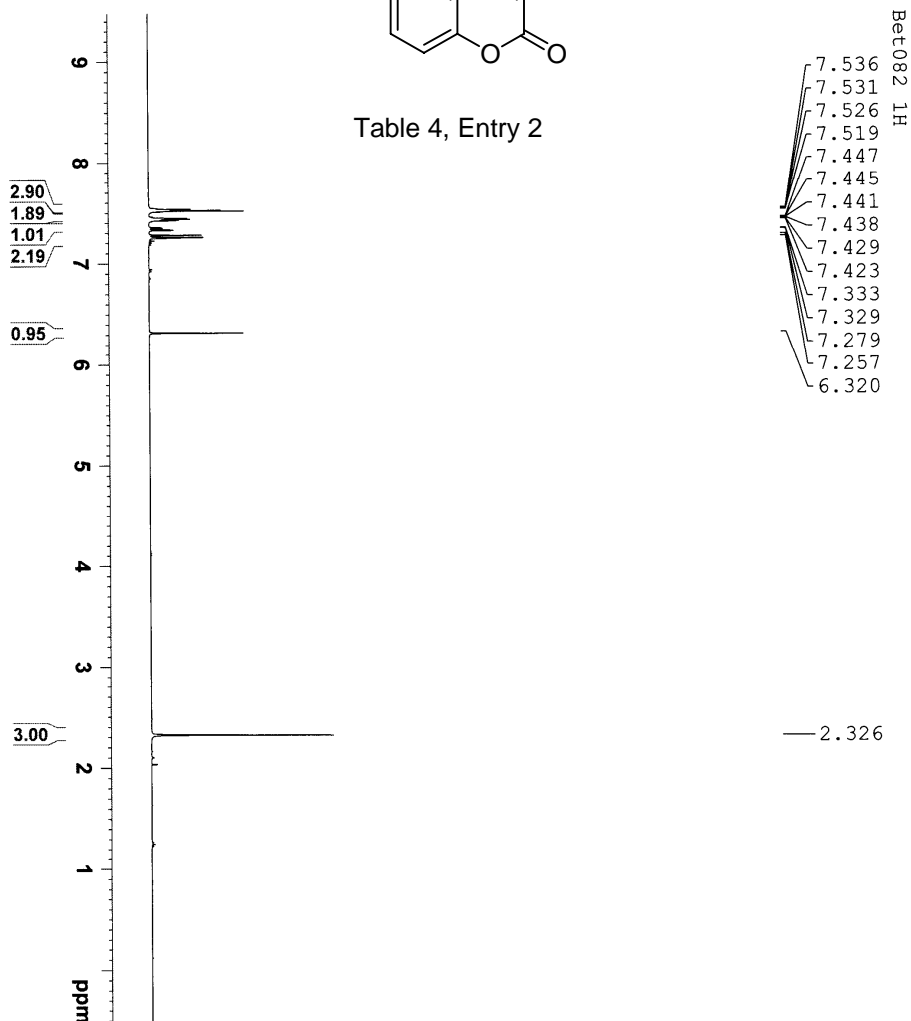


Table 4, Entry 2



```

NAME Bet082
EXPNO 1
PROCNO 1
Date_ 20100205
Time 19.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 2
DS 8
SWH 4006.410 Hz
FIDRES 0.122286 Hz
AQ 4.0894966 sec
RG 25.4
DM 124.690 usec
DE 6.90 usec
TE 297.6 K
D1 1.00000000 sec
ID0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
F1F2 11.8812272 MHz
SFO1 400.1318007 MHz
SI 32768
SF 400.1300000 MHz
HFR 0
SSB 0
GB 0
PC 1.00
    
```

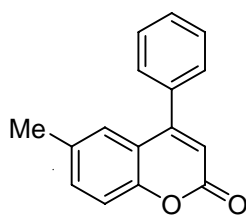
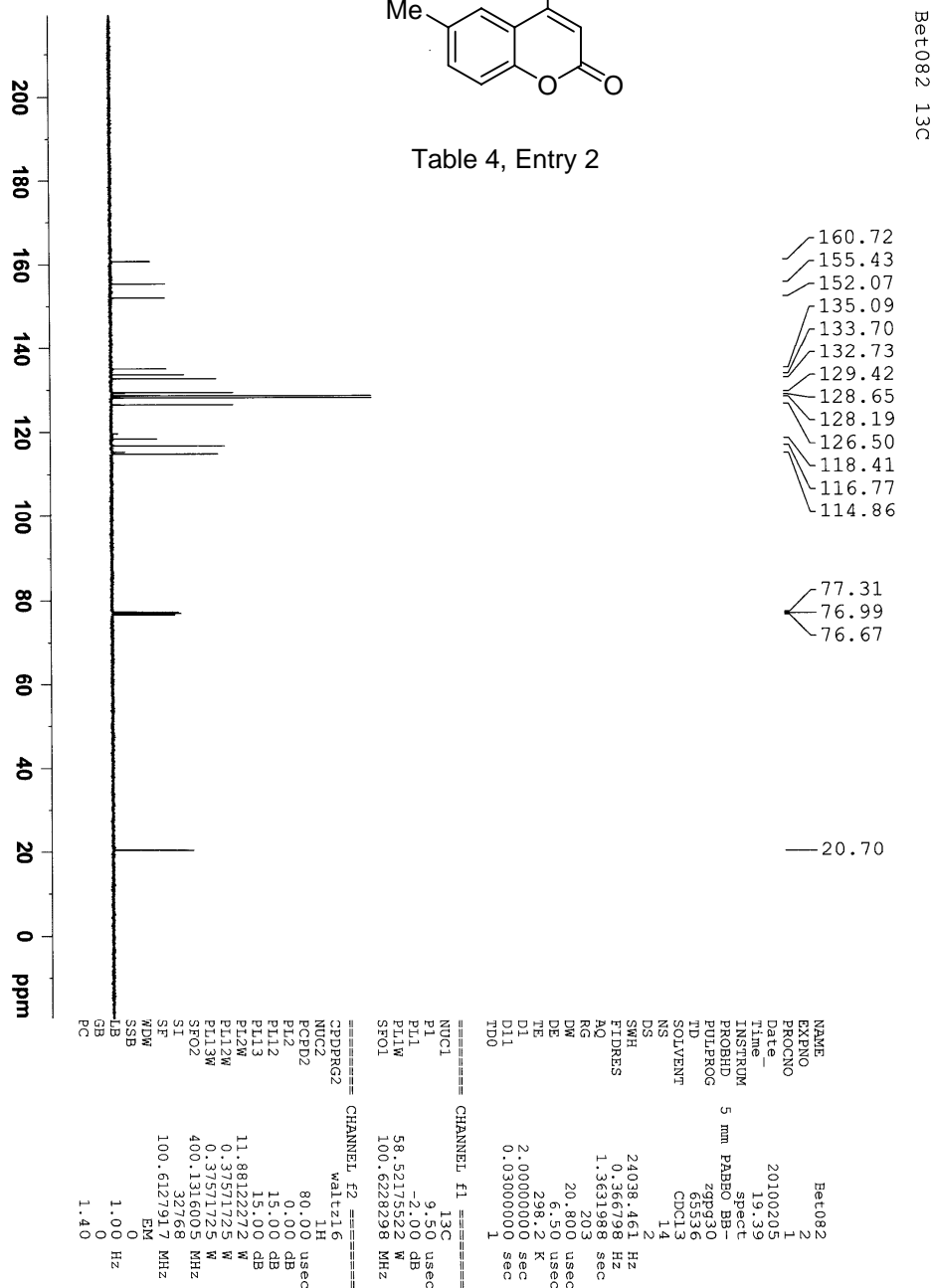


Table 4, Entry 2



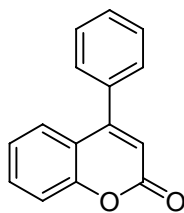
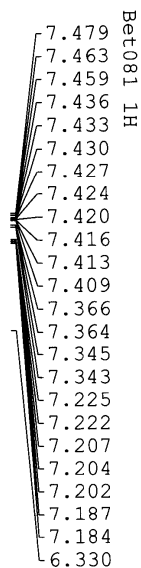
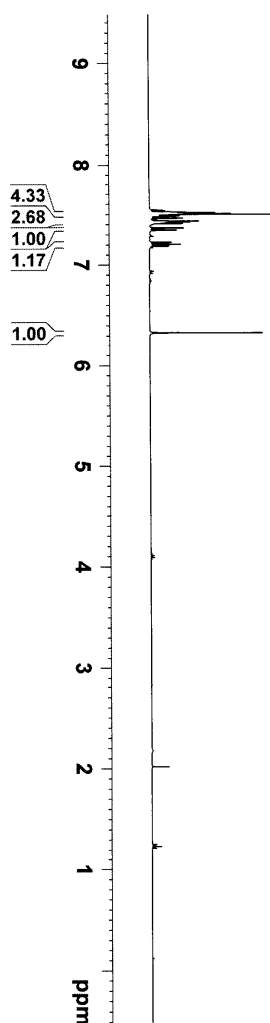


Table 4, Entry 3



```

NAME Bet081
EXPNO 1
PROCNO 1
Date_ 20100205
Time 19.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 2
DS 2
SWH 4006.410 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 22.6
DW 124.800 usec
DE 6.39 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 0.00 dB
FL1W 11.89122272 MHz
SEDL 400.132768 MHz
SF 400.1306000 MHz
RFW 0
SSB 0
GB 0.30 Hz
PC 1.00
    
```

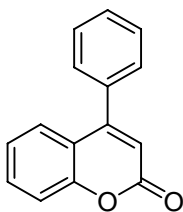
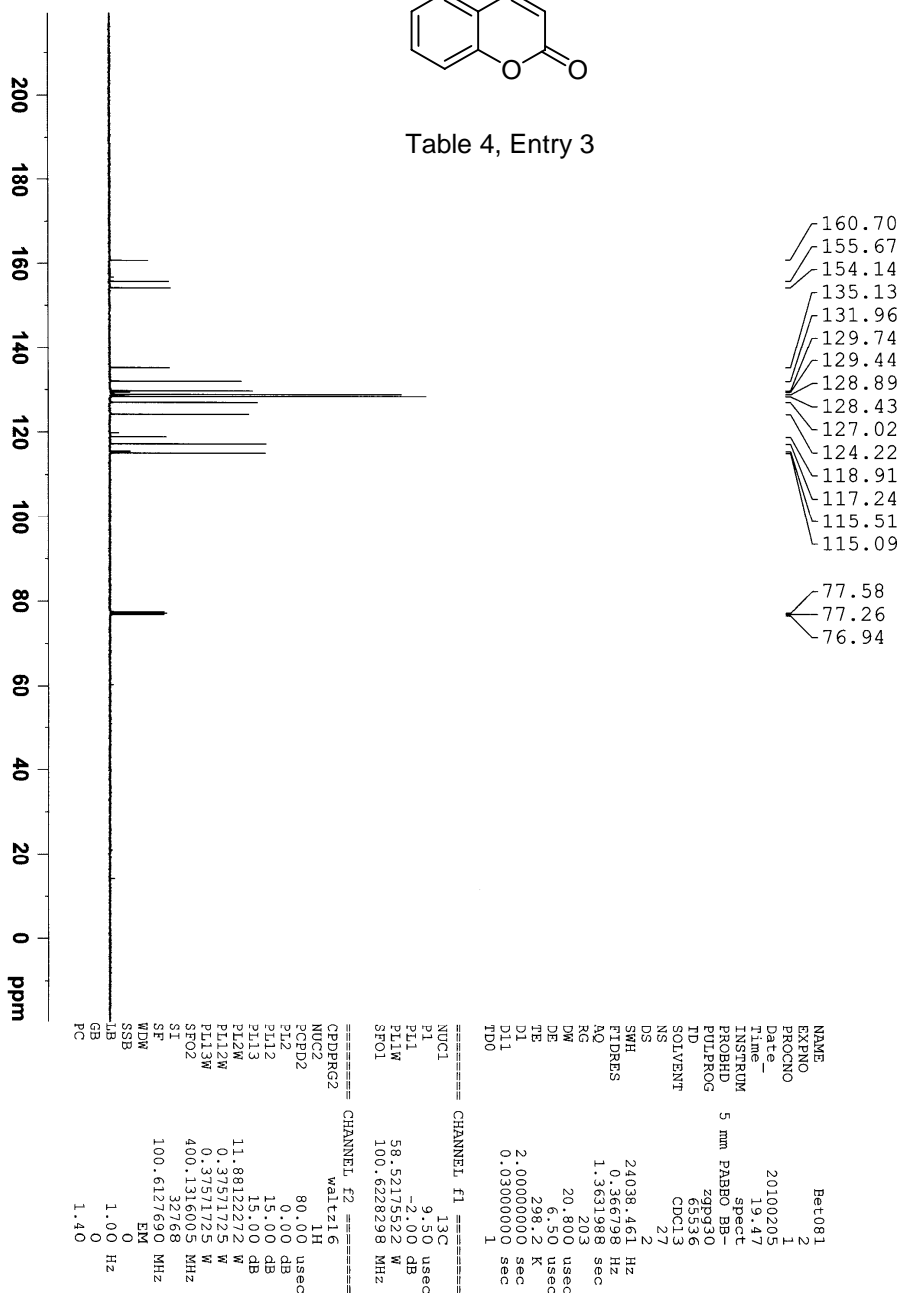


Table 4, Entry 3

Bet081 13C



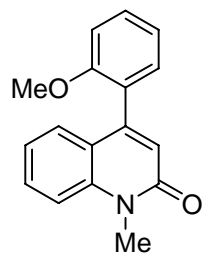
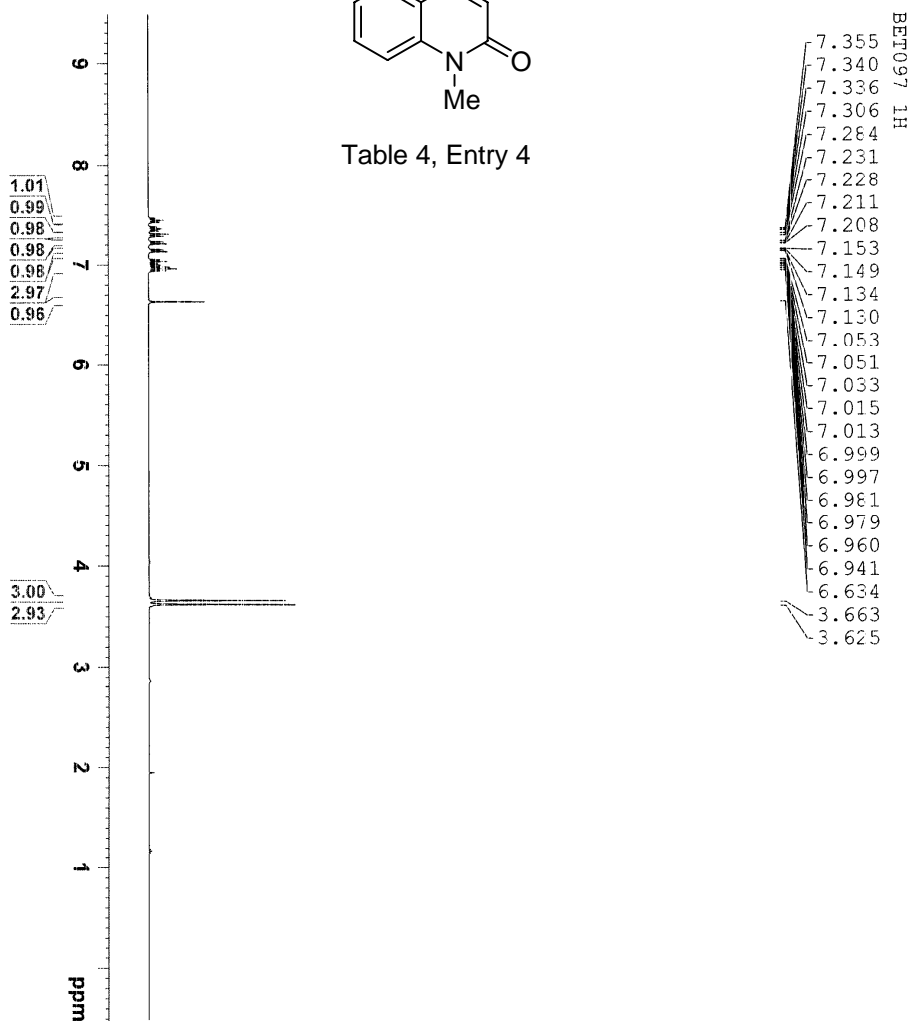


Table 4, Entry 4



BEP097 1H
 7.355
 7.340
 7.336
 7.306
 7.264
 7.231
 7.228
 7.211
 7.208
 7.153
 7.149
 7.134
 7.130
 7.053
 7.051
 7.033
 7.015
 7.013
 6.999
 6.997
 6.961
 6.979
 6.960
 6.941
 6.634
 3.663
 3.625

```

NAME          BEP097
EXPNO         1
PROCNO        1
Date_         20100301
Time         19.16
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            2
DS            2
SWH           4006.410 Hz
FIDRES       0.122268 Hz
AQ           4.0894966 sec
RG            9
DM           124.800 usec
DE           6.50 usec
TE           298.2 K
D1           1.00000000 sec
ID0          1

----- CHANNEL f1 -----
NUC1          1H
P1           14.70 usec
PL1          0.00 dB
F21W         11.88122572 MHz
SFO1         400.143756 MHz
SI           32
SF           400.130000 MHz
PDM          DM
SSB          0
GB           0
PC           1.00
    
```

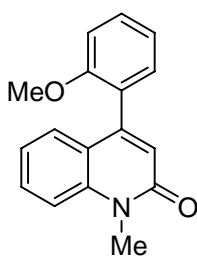
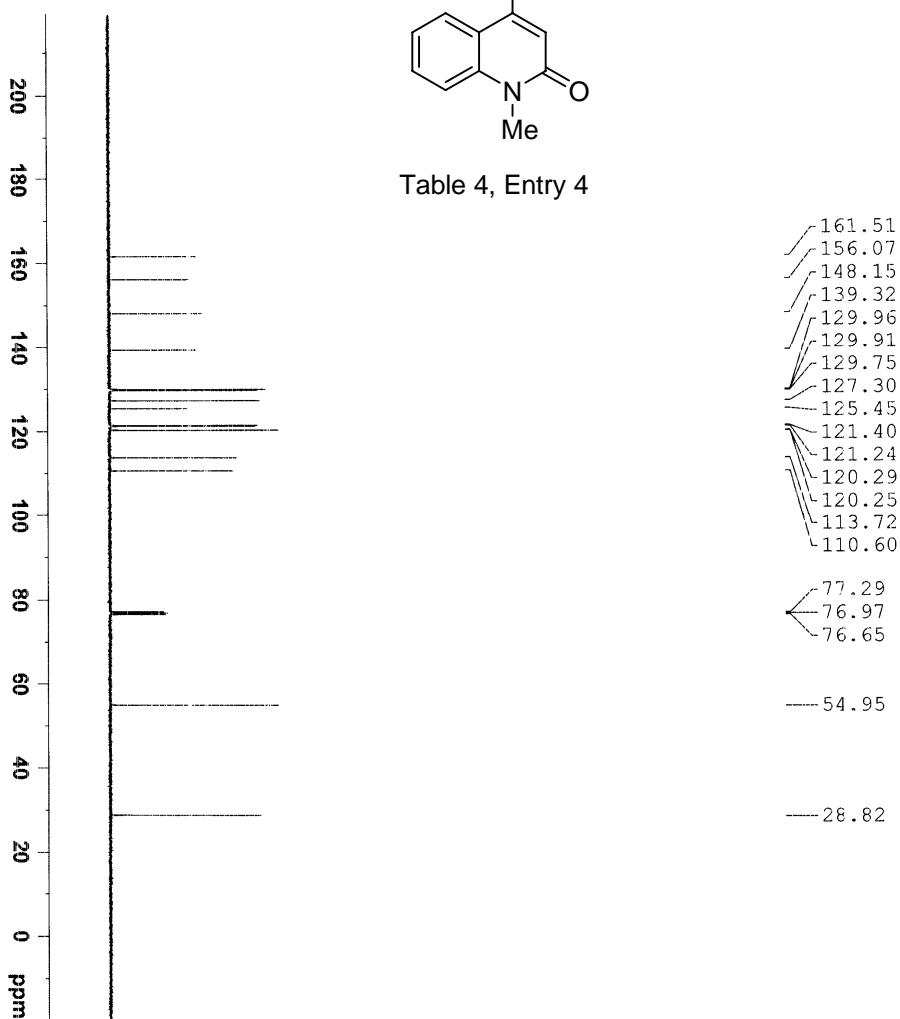


Table 4, Entry 4



BET097 13C

- 161.51
- 156.07
- 148.15
- 139.32
- 129.96
- 129.91
- 129.75
- 127.30
- 125.45
- 121.40
- 121.24
- 120.29
- 120.25
- 113.72
- 110.60
- 77.29
- 76.97
- 76.65
- 54.95
- 28.82

```

NAME          BET097
EXPNO         2
PROCNO        1
Date_         20100301
Time          19.25
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
ID            63536
SOLVENT       CDCl3
NS            16
DS            2
SWH           24038.461 Hz
FIDRES        0.256728 Hz
AQ            1.3631908 sec
RG            20.000
DM            20.000
DE            20.000 usec
TE            300.2 K
D1            2.00000000 sec
D11           0.030000000 sec
TD0           1

===== CHANNEL F1 =====
NUC1          13C
PI            9.50 usec
PL1           -2.00 dB
P1M1         58.52175522 W
SFO1         100.628298 MHz

===== CHANNEL F2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PI2           0.00 dB
P12           15.00 dB
P1M2         11.88122272 W
P1M3         0.37571725 W
P1M4         0.37571725 W
SFO2         100.1253085 MHz
SI           32768
SF           100.6120176 MHz
WDW          EM
SSB          0
GB           1.00 Hz
PC           1.40
    
```

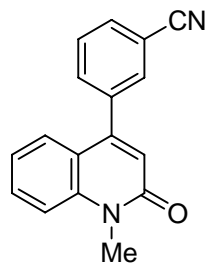
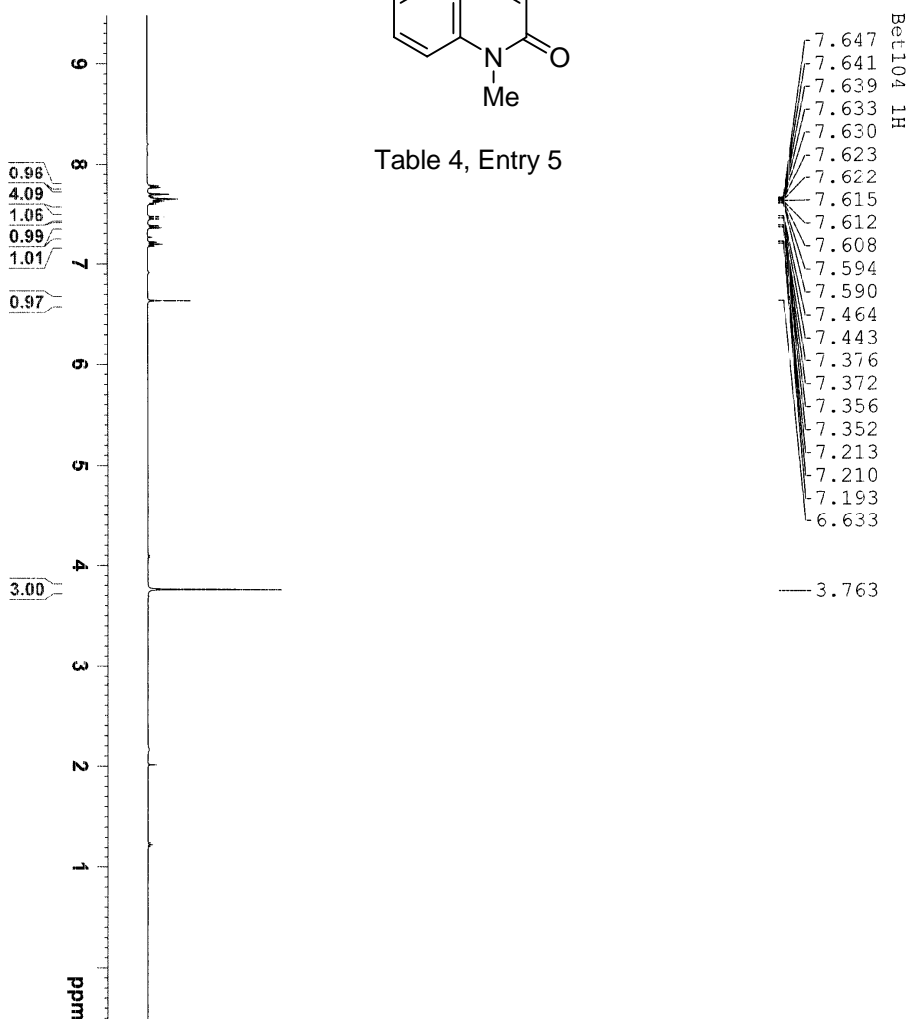


Table 4, Entry 5



Chemical shift list (ppm):

- 7.647
- 7.641
- 7.639
- 7.633
- 7.630
- 7.623
- 7.622
- 7.615
- 7.612
- 7.608
- 7.594
- 7.590
- 7.464
- 7.443
- 7.376
- 7.372
- 7.356
- 7.352
- 7.213
- 7.210
- 7.193
- 6.633
- 3.763

```

NAME          Bet104
EXPNO         1
PROCNO        1
Date_         20100301
Time          13.30
INSTRUM       spect
PROBHD        5 mm PABBO-BB-
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            2
DS            2
SWH           4006.410 Hz
FIDRES        0.122252 Hz
AQ           4.0894966 sec
RG            45.2
DE           124.800 usec
TE           298.4 K
D1           1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1           14.70 usec
PL1          0.00 dB
FL1W         11.8912222 MHz
SFO1         400.1318002 MHz
SI           32768
SF           400.1300027 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00
    
```

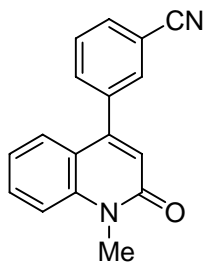
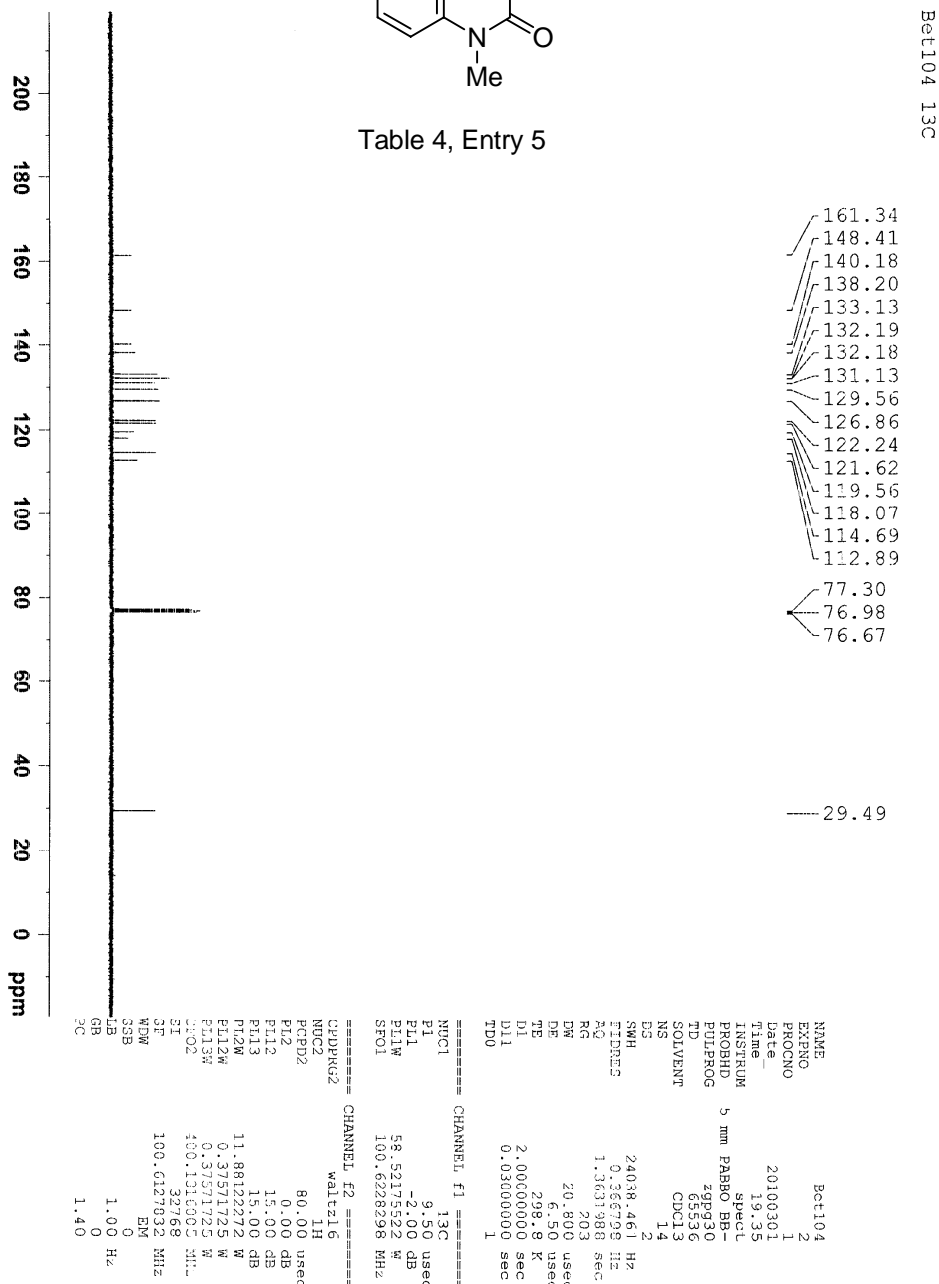


Table 4, Entry 5



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Selected filters: None

Monoisotopic Mass, Even Electron Ions

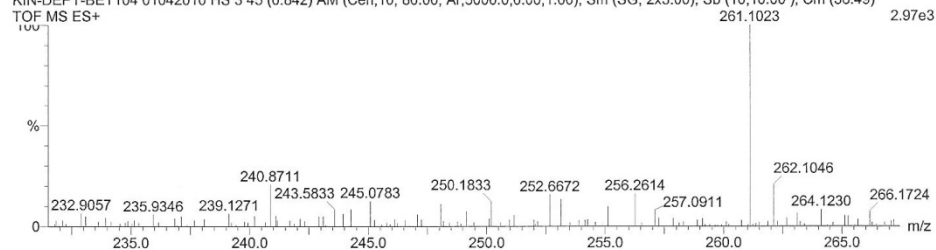
102 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 0-25 H: 0-20 N: 0-3 O: 0-6 Na: 0-1

KIN-DEPT-BET104 01042010 HS 3 45 (0.842) AM (Cen,10, 80.00, Ar,5000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (10,10.00); Cm (36:49)

TOF MS ES+



Minimum: -1.5
Maximum: 5.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
261.1023	261.1028	-0.5	-1.9	12.5	41.4	C17 H13 N2 O

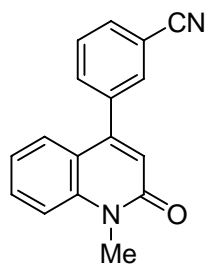


Table 4, Entry 5

7. References

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