**Supporting information**

# Asymmetric reduction of electron-rich ketones with tethered Ru(II)/TsDPEN catalysts using formic acid/triethylamine or aqueous sodium formate.

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| | |
|---|---|
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**Tables of results.**

**Full Table 1, with time course studies:**

| reaction no. | Catalyst | S/C | Solvent | T | t/h | % Conv | % ee |
|---|---|---|---|---|---|---|---|
| **Water reactions** | | | | | | | |
| TH236 | SS-3C teth **2** | 100 | $H_2O$ | 40 $^oC$ | 24 | >99 | 97 (*S*) |
| SRC 1177a | RR-4-Methoxy **5** | 100 | $H_2O$ | 40 $^oC$ | 1h<br>2h<br>4h<br>5h | 32<br>53<br>84<br>97 | 98(*R*)<br>98(*R*)<br>98(*R*)<br>98(*R*) |
| SRC1244 | RR-3C teth **2** | 100 | $H_2O$ | 60 $^oC$ | 1h | 99 | 88(*R*) |
| SRC 1177b | RR-4-Methoxy **5** | 100 | $H_2O$ | 60 $^oC$ | 1h<br>2h | 77<br>>99 | 98(*R*)<br>97(*R*) |
| SRC 1193 | RR-3,5-dimethoxy **6** | 100 | $H_2O$ | 60 $^oC$ | 2h | >99 | 77(*R*) |
| SRC 1195 | RR-3,5-dimethoxy **6** | 500 | $H_2O$ | 60 $^oC$ | 1h<br>3h<br>5h | 35<br>82<br>99 | 86(*R*)<br>82(*R*)<br>81(*R*) |
| **$H_2O$/MeOH** | | | | | | | |
| TH232 | SS-3C teth **2** | 100 | $H_2O$ MeOH (1:1) | 60 $^oC$ | 1 | >99 | 96 (*S*) |
| TH233 | RR-4-OMe **5** | 100 | $H_20$ meOH (1:1) | 60 $^oC$ | 0.5 | 96 | 98 (*R*) |
| SRC 1200b | RR-4-Methoxy **5** | 500 | $H_2O$:MeOH (1:1) | 60 $^oC$ | 1h<br>3h<br>4h<br>5h<br>6h | 35<br>74<br>88<br>98<br>99 | 97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*) |
| SRC 1203b | RR-4-Methoxy **5** | 500 | $H_2O$:MeOH (1:1) | 60 $^oC$ | 1h<br>2h<br>4h | 41<br>73<br>99 | 97(*R*)<br>97(*R*)<br>97(*R*) |
| SRC 1203c | RR-3,5-dimethoxy **6** | 500 | $H_2O$:MeOH (1:1) | 60 $^oC$ | 1h<br>2h<br>3h | 55<br>92<br>99 | 85(*R*)<br>85(*R*)<br>85(*R*) |
| SRC 1201b | RR-3,5-dimethoxy **6** | 500 | $H_2O$:MeOH (1:0.5) | 60 $^oC$ | 1h<br>2h | 67<br>99 | 86(*R*)<br>86(*R*) |
| **Other conditions** | | | | | | | |
| SRC 1230a | RR-4-Methoxy **5** | 100 | $HCOONH_4$ $H_2O$ | 60 $^oC$ | 1h<br>3.5h<br>6.5h<br>22.5 h | 11<br>20<br>25<br>56 | 97(*R*)<br>98(*R*)<br>98(*R*)<br>98(*R*) |
| SRC 1230b | RR-4-Methoxy **5** | 100 | SDS $H_2O$ | 60 $^oC$ | 1h<br>3.5h<br>6.5h<br>22.5 | 31<br>48<br>54<br>57 | 97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*) |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | | | h | | |
| **Recycling** | | | | | | | |
| SRC 1234(1) | RR-4-Methoxy **5 *** | 100 | $H_2O$ | 60 $^oC$ | 1h | 99 | 98(*R*) |
| SRC 1234(2) | | | HCOOH | 60 $^oC$ | 1h<br>2h<br>3.5h | 40<br>60<br>99 | 98(*R*)<br>97(*R*)<br>97(*R*) |
| SRC 1234(3) | | | HCOOH | 60 $^oC$ | 1h<br>2.5h<br>15h<br>rt<br>5h<br>9.5 h | 26<br>52<br>57<br>67<br>76 | 97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*) |

* n-Hexane (3x 2 mL) was used to extract product after each cycle from reaction mixture. 1 mol of HCOOH was added to regenerate HCOONa.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| SRC 1235(1) | RR-4-Methoxy **5 *** | 100 | $H_2O$ | 60 $^oC$ | 1h | 99 | 97(*R*) |
| SRC 1235(2) | | | HCOOH | 60 $^oC$ | 1h | 99 | 98(*R*) |
| SRC 1235(3) | | | HCOOH | 60 $^oC$ | 1h<br>1.5h | 85<br>99 | 97(*R*)<br>98(*R*) |
| SRC 1235(4) | | | HCOOH | 60 $^oC$ | 1h<br>2h<br>15hrt<br>4h<br>6h<br>8h | 34<br>64<br>68<br>83<br>90<br>93 | 96(*R*)<br>96(*R*)<br>96(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*) |

* n-Pentane (3x 2 mL) was used to extract product after each cycle from reaction mixture. 1 mol of HCOOH was added to regenerate HCOONa.

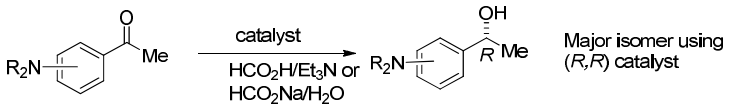
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| SRC 1236(1) | RR-4-Methoxy **5 *** | 100 | $H_2O$ | 60 $^oC$ | 1h | 99 | 98(*R*) |
| SRC 1236(2) | | | HCOOH | 60 $^oC$ | 1h<br>1.5h | 88<br>99 | 96(*R*)<br>96(*R*) |
| SRC 1236(3) | | | HCOOH | 60 $^oC$ | 1h<br>4h<br>17hrt<br>5h<br>7h<br>9h | 20<br>80<br>88<br>91<br>94<br>96 | 96(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*)<br>97(*R*) |

* Pet. ether (3x 2 mL) was used to extract product after each cycle from reaction mixture. 1 mol of HCOOH was added to regenerate HCOONa.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| SRC 1237(1) | RR-4-Methoxy **5 *** | 100 | $H_2O$ | 60 $^oC$ | 1h | 99 | 97(*R*) |
| SRC 1237(2) | | | HCOOH | 60 $^oC$ | 1h | 99 | 97(*R*) |
| SRC 1237(3) | | | HCOOH | 60 $^oC$ | 1h<br>1.5h | 93<br>99 | 97(*R*)<br>97(*R*) |
| 15h rt<br>SRC 1237(4) | | | HCOOH | 60 $^oC$ | 1h<br>2.5h<br>4.5h<br>6.5 h | 23<br>53<br>85<br>97 | 95(*R*)<br>95(*R*)<br>96(*R*)<br>96(*R*) |

* n-Pentane (3x 2 mL) was used to extract product after each cycle from the reaction mixture. 1 mol of HCOOH was added to regenerate HCOONa.

**Full Table 2:**

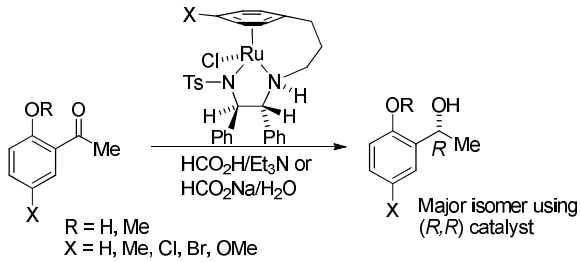



| Entry reaction no | Substrate | Catal-yst | S/C | Solvent | Temp/oC or rt | t/h | % Conv. (yield) | % ee |
|---|---|---|---|---|---|---|---|---|
| BM2 | $pNH_2$ | SS-3C teth **2** | 100 | $H_2O$ | 60 | 3.5 | 97 | 94 (*S*) |
| BM72 | $pNH_2$ | SS 3C-teth **2** | 100 | $H_2O$/MeOH | 40 | 7.5 | 80 (69) | 92 (*S*) |
| SRC 1256 | $pNH_2$ | SS-3C-teth **2** | 100 | $H_2O$/MeOH | 60 | 4.5h | 99 (79) | 94 (*S*) |
| SRC 1261 | $pNH_2$ | RR-4-OMe **5** | 100 | $H_2O$/MeOH | 60 | 4.5h | 99 (75) | 94 (*R*) |
| SRC 1267 | $pNH_2$ | [Ru(cymene RR-TsDPEN)Cl | 100 | $H_2O$/MeOH | 60 | 15.5 | 78 (ND) | 91 (*R*) |
| SRC 1253 | $pNH_2$ | RR-4-OMe **5** | 100 | FA:TEA | 60 | 24 | NR | Polymerises |
| BM14 | $pNH_2$ | RR-3C teth **2** | 100 | iPrOH | Rt | 22/144 | 17/50 (ND) | ND |
| BM17 | $pNMe_2$ | SS 3C teth **2** | 100 | $H_2O$ | 28 | 3 | 76 (41) | 88 (*S*) |
| BM32 | $pNMe_2$ | SS 3C teth **2** | 100 | $H_2O$/MeOH | 40 | 5 | 99 (76) | 92 (*S*) |
| SRC 1258 | $pNMe_2$ | RR-4-OMe **5** | 100 | $H_2O$: MeOH | 60 | 4 | 97 (78) | 91 (*R*) |
| SRC 1260 | $pNMe_2$ | 3C-teth (S,S) **2** | 250 | FA:TEA | 45 | 6 | 93 (68) | 91 (*S*) |
| BM44 | $pNMe_2$ | 3C teth RR | 100 | FA:TEA | 40 | 45 | 95 (60) | 93 (*R*) |
| BM7 | $pNMe_2$ | RR 3C teth **2** | 100 | iPrOH | 28 | 26 | 35 (17) | ND |
| BM47 | Pyrrolidine | RR 3C teth **2** | 100 | FA/TEA | 40 | 72 | 100 (81) | 89 (*R*) |
| BM45 | Pyrrolidine | RR 3C teth **2** | 100 | $H_2O$/MeOH | 40 | 69 | 98 (78) | 89 (*R*) |
| BM70 | Pyrrolidine | SS 3C teth **2** | 100 | $H_2O$/MeOH | 40 | 70 | 98 (72) | 89 (*R*) |
| BM41 | Piperidine | RR 3C teth **2** | 100 | FA/TEA | 40 | 69 | 99 (68) | 94 (*R*) |
| BM34 | Piperidine | SS 3C teth **2** | 100 | $H_2O$/MeOH | 40 | 24 | 99 (84) | 95 (*S*) |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| BM39 | Morpholine | RR 3C teth **2** | 100 | FA/TEA | 40 | 23 | 99 (74) | 94 (*R)* |
| BM43 | Morpholine | SS 3C teth **2** | 100 | $H_2O$/MeOH | 40 | 45 | 100 (88) | 91 (*S*) |

ND = not determined. 1:1 $H_2O$/MeOH throughout.

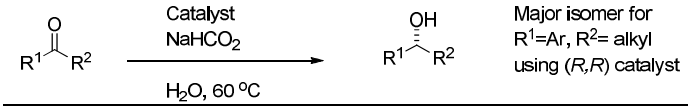
**Full Table 3**, including results under aqueous conditions where studied**:**

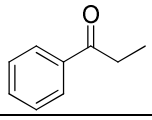
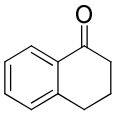
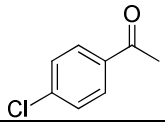
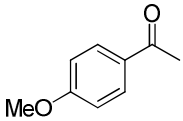
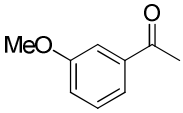
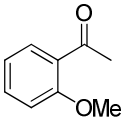
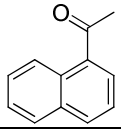
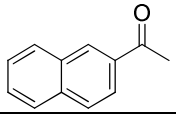
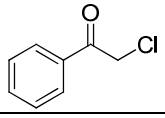


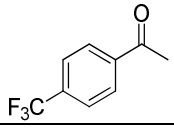
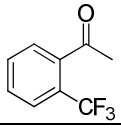
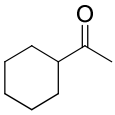

| **Entry/exno** | **Aceto-phenone** | **Catalyst** | **S/C** | **Solvent** | **T/ °C** | **t/h** | **% Conv (y. = yield where isolated** | **% ee** |
|---|---|---|---|---|---|---|---|---|
| TH163 | 2-OH, 5-methyl | SS-3C teth **2** | 200 | FA/TEA | 40 | 5.5 | 99/ 72% y. | 94 (*S*) |
| TH156 | 2-OH, 5-methyl | RR-p-OMe **5** | 200 | FA/TEA | 40 | 23 | 100/ 100 y | 95 (*R*) |
| TH162 | 2-OH, 5-Cl | SS-3C teth **2** | 200 | FA/TEA | 40 | 4.75 | 99/ 72% y | 90 (*S*) |
| TH155 | 2-OH, 5-Cl | RR-p-OMe **5** | 200 | FA/TEA | 40 | 23 | 97/ 94% y | 93 (*R*) |
| TH161 | 2-OH, 5-Br | SS-3C teth **2** | 200 | FA/TEA | 40 | 4 | >99/ 65% | 91 (*S*) |
| TH158 | 2-OH, 5-Br | RR-p-OMe **5** | 200 | FA/TEA | 40 | 5.5 | 98/ 42% y | 94 (*R*) |
| TH148 | 2-OH, 5-OMe | SS-3C teth **2** | 200 | FA/TEA | 40 | 5.5 | 92%/ 24% y | 93 (*S*) |
| TH150 | 2-OH, 5-OMe | RR-p-OMe **5** | 200 | FA/TEA | 40 | 6.5 | 91/ 84% y | 93 (*R*) |
| TH182 | 2-OMe | SS-3C teth **2** | 200 | FA/TEA | 40 | 3.75 | >95/ 64% y | 68 (*S*) |
| TH238<br>TH262 | 2-OMe | SS 3C teth **2** | 200<br>100 | $H_2O$<br>$H_2O$ | 60<br>60 | 4<br>5 | 89<br>98 | 55(*S*)<br>58(*S*) |
| TH183 | 2-OMe | RR-p-OMe **5** | 200 | FA/TEA | 40 | 3.75 | >98/ 70% y | 96 (*R*) |
| SRC1246<br>TH243 | 2-OMe | RR-p-OMe **5** | 100<br>200 | $H_2O$<br>$H_2O$ | 60<br>60 | 2<br>3.5 | 99<br>92 | 95(*R*)<br>96(*R*) |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| TH187 | 2-OMe, 5-OMe | SS-3C teth **2** | 200 | FA/TEA | 40 | 3.75 | >99/ 80% y | 69 (*S*) |
| TH188 | 2-OMe, 5-OMe | RR-p-OMe **5** | 200 | FA/TEA | 40 | 3.75 | >99/ 87% y | 90 (*R*) |

**Data for products in Figure 2,** including time course of reductions where studied:




| Entry/ reaction | Substrate | Catalyst | S/C | Solvent | t/h | % Conv | % ee |
|---|---|---|---|---|---|---|---|
| SRC 1240<br>MW237A |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>1.5 | 88<br>99 | 98 (*R*)<br>98 (*R*) |
| SRC 1241<br>MW237B |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>3<br>5<br>7 | 36<br>74<br>93<br>98 | Nd<br>nd<br>nd<br>97 (*R*) |
| SRC 1243 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>1.5<br>2 | 72<br>98<br>99 | 95 (*R*)<br>95 (*R*)<br>95 (*R*) |
| SRC 1242 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>2<br>2.5<br>3 | 53<br>97<br>98<br>99 | 98 (*R*)<br>97 (*R*)<br>97 (*R*)<br>97 (*R*) |
| SRC 1245 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>2 | 96<br>96 | 98 (*R*)<br>97 (*R*) |
| SRC 1246 |  | RR-4-Methoxy **5** (example in Table 3) | 100 | $H_2O$ | 1<br>2 | 65<br>99 | 96 (*R*)<br>95 (*R*) |
| SRC 1247<br>MW237C |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>3 | 36<br>99 | 99 (*R*)<br>99 (*R*) |
| SRC 1248<br>MW237D |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>3 | 35<br>99 | 97 (*R*)<br>96 (*R*) |
| SRC 1249 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1 | 99 | 96 (*S*) |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| SRC 1250 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>1.5 | 85<br>99 | 94 (*R*)<br>94 (*R*) |
| SRC 1251 |  | RR-4-Methoxy **5** | 100 | $H_2O$ | 1<br>2<br>2.5 | 81<br>99<br>99 | 71 (*R*)<br>69 (*R*)<br>69 (*R*) |
| SRC 1194<br>MW237F |  | RR-3,5-dimethoxy **6** | 100 | $H_2O$ | 2 | 99 | 85 (*S*) |
| SRC 1196 | | RR-3,5-dimethoxy **6** | 500 | $H_2O$ | 1<br>3<br>5 | 22<br>73<br>99 | Nd<br>nd<br>85 (*S*) |

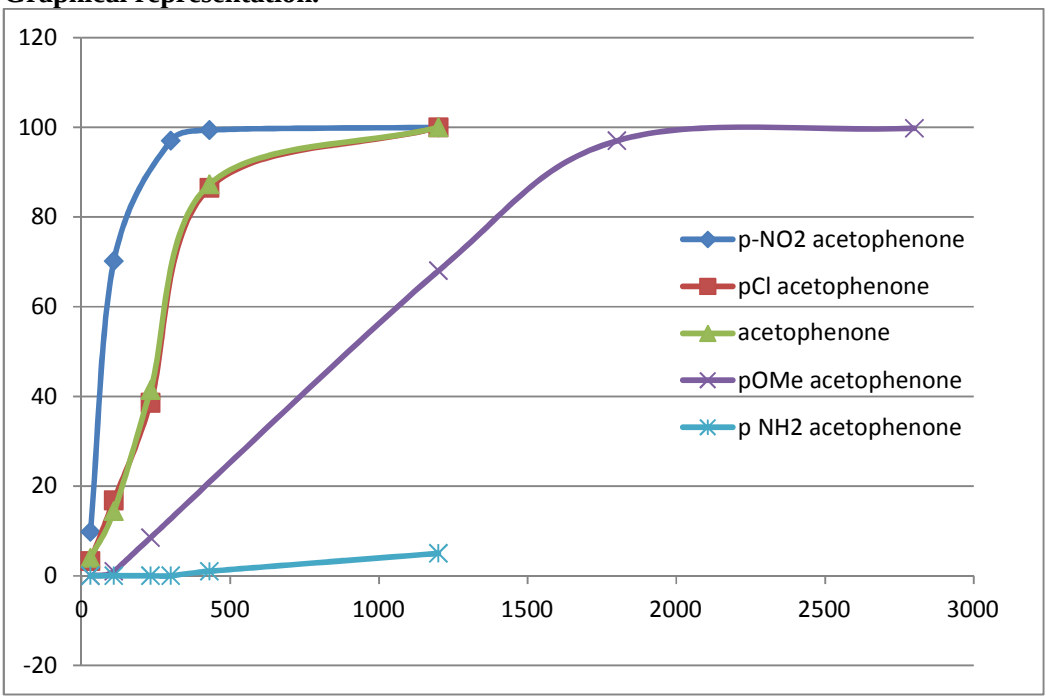
ND = not determined.

**Time course of reductions of ketones to illustrate effect of electron-donating and electron-withdrawing groups on rates** (Formic acid/triethylamine 5:2, 0.2 mol% cat. (*S*,*S*)-**2** [S]=0.5 M, rt). The electron-rich substrates are reduced at a lower rate than the electron-poor substrates:

| | $pNO_2$ acetophenone | p-Cl acetophenone | Acetophenone |
|---|---|---|---|
| Time | conversion | conversion | conversion |
| 30 | 9.8 | 3.3 | 4 |
| 109 | 70.2 | 16.8 | 14.4 |
| 300 | 97 | 38.6 | 41.4 |
| 430 | 99.4 | 86.5 | 87.3 |
| 1200 | 100 | 100 | 100 |

| | pOMe acetophenone | | pNH acetophenone |
|---|---|---|---|
| Time | conversion | Time | conversion |
| 30 | 0 | 30 | 0 |
| 109 | 1 | 109 | 0 |
| 232 | 8.5 | 232 | 0 |
| 1200 | 68 | 300 | 0 |
| 1800 | 97 | 430 | 1 |
| 2800 | 99.8 | 1200 | 5 |

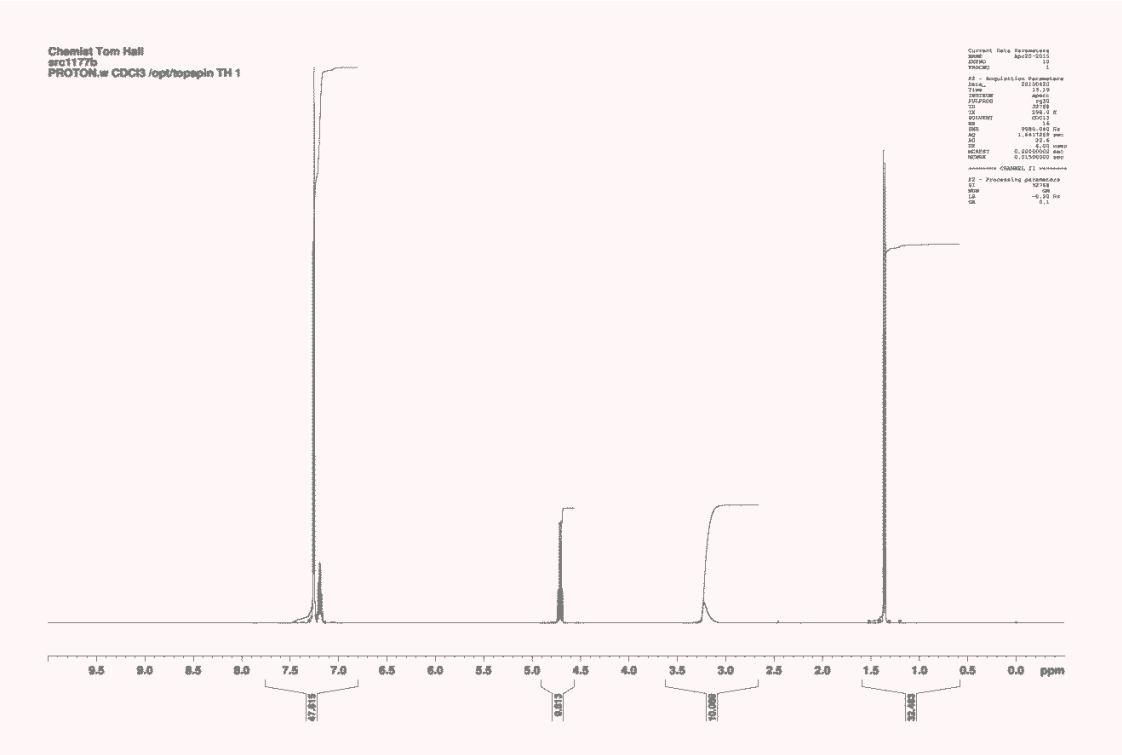
**Graphical representation.**



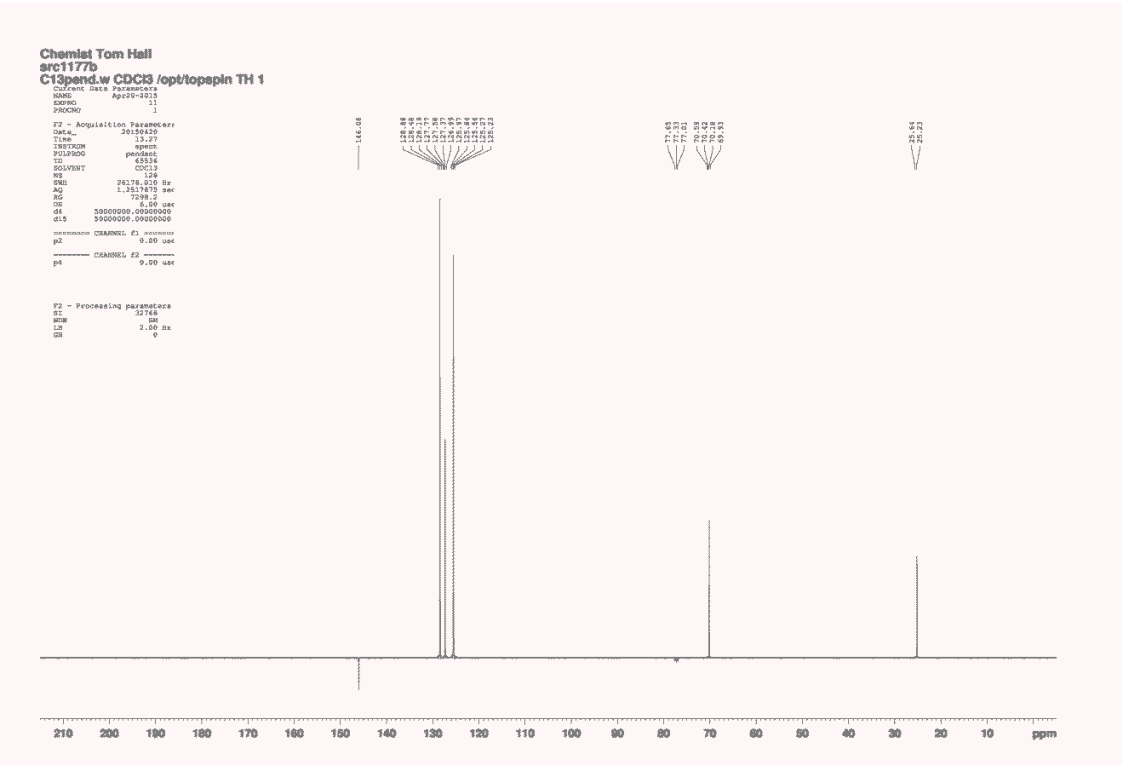
**$^{1}$H/$^{13}$C NMR Spectroscopic data.**

**1-Phenylethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

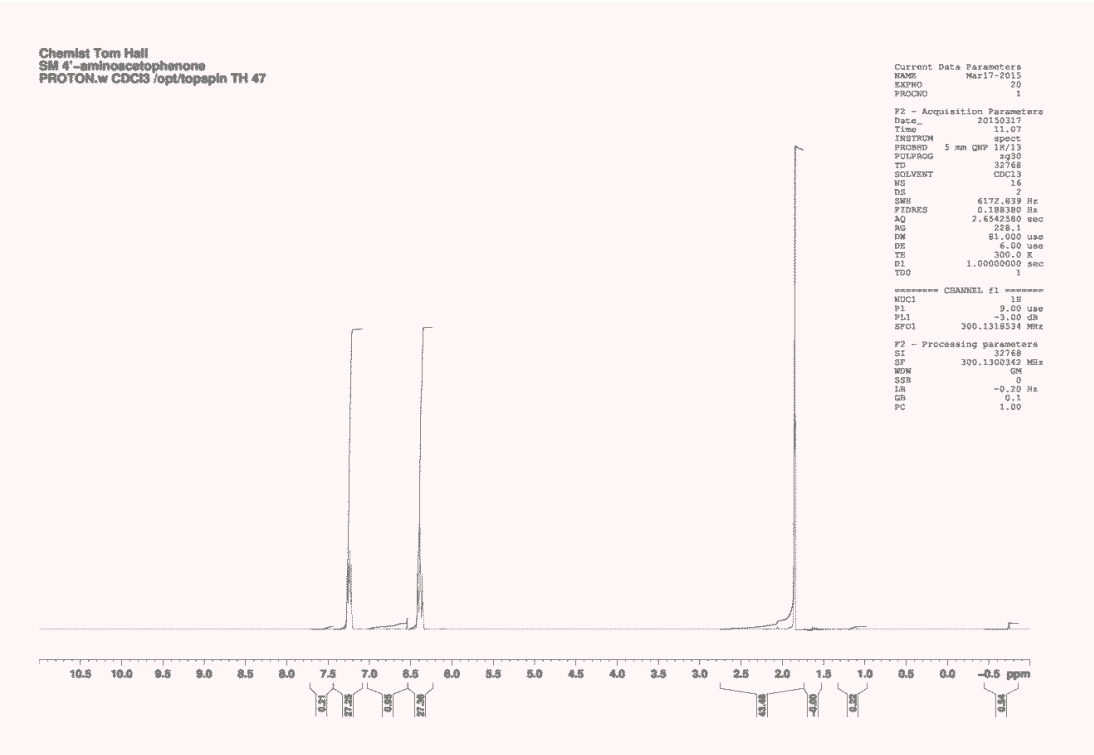



$^{13}$C, 100 MHz, $CDCl_3$

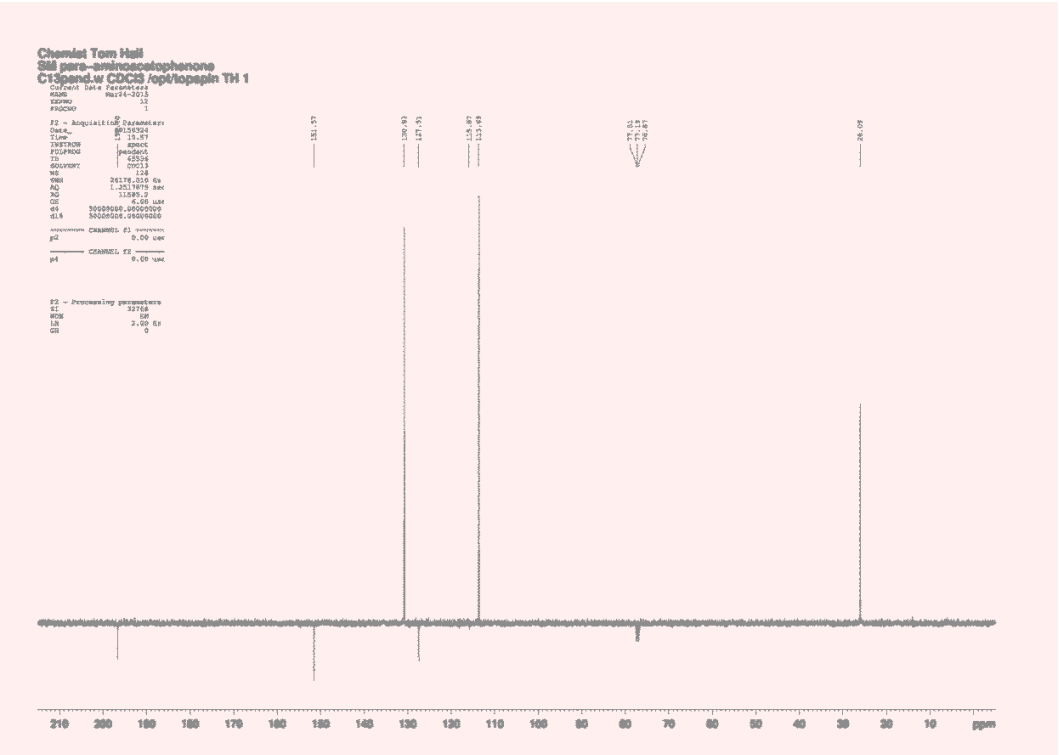


Commercial p-amino acetophenone to conform peak positions:

$^{1}$H, 300 MHz, $CDCl_3$

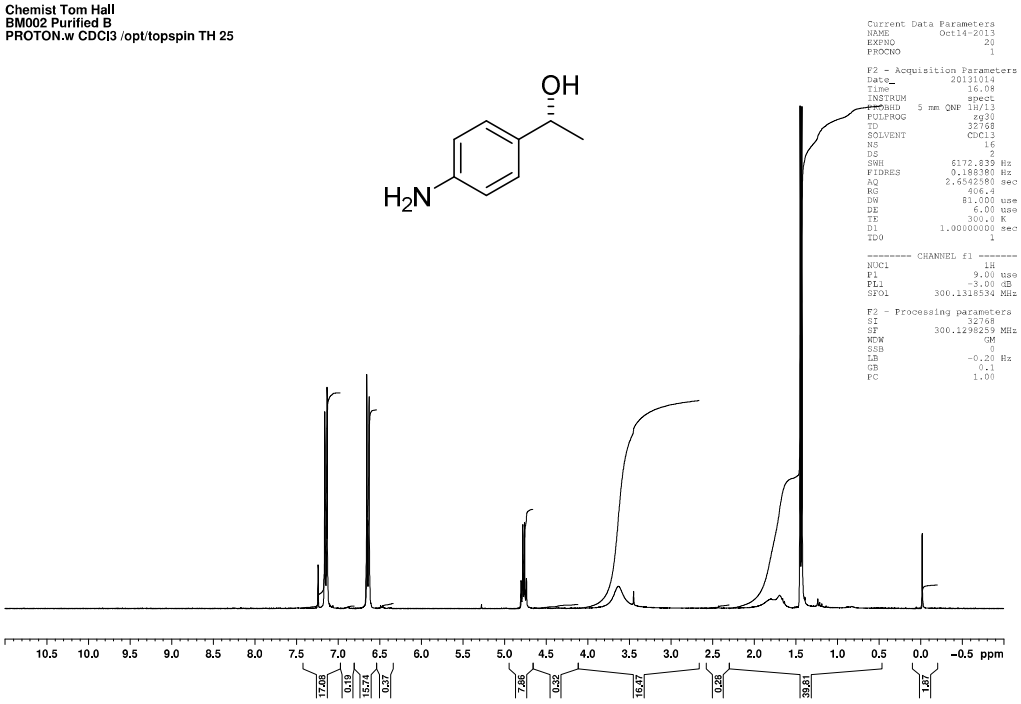



$^{13}$C, 75 MHz, $CDCl_3$

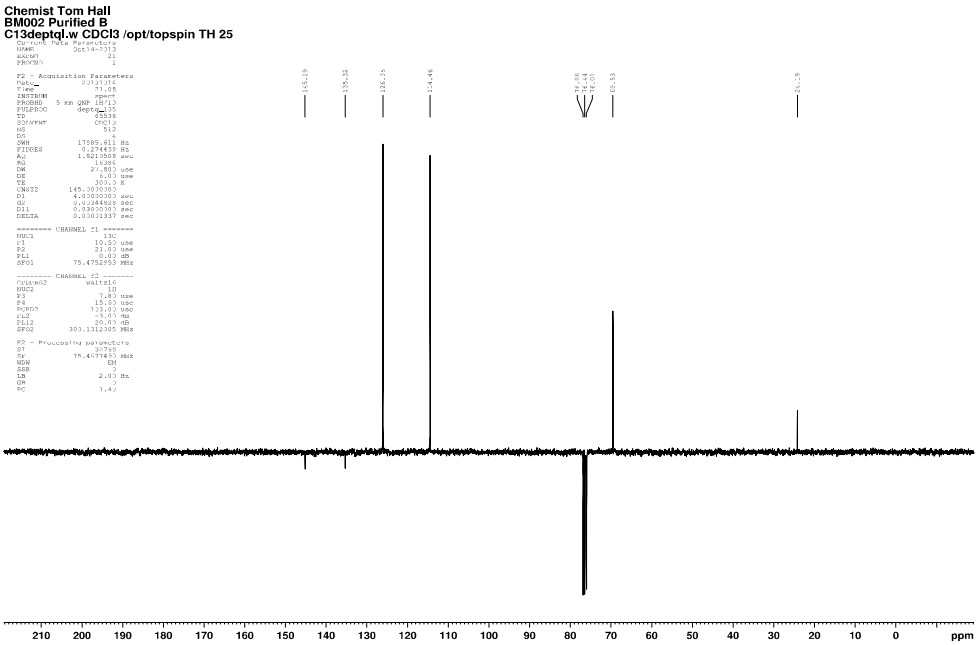


## 1-(4-Aminophenyl)ethanol

$^{1}$H, 300 MHz, $CDCl_3$

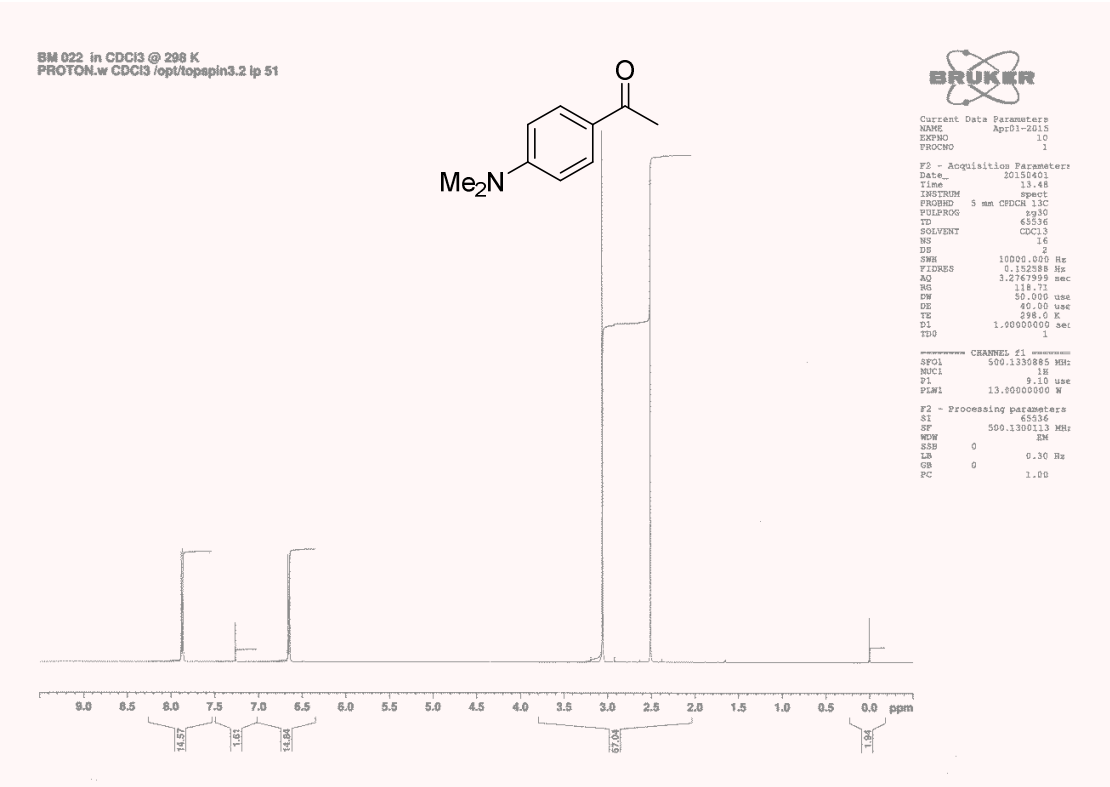



$^{13}$C, 75 MHz, $CDCl_3$

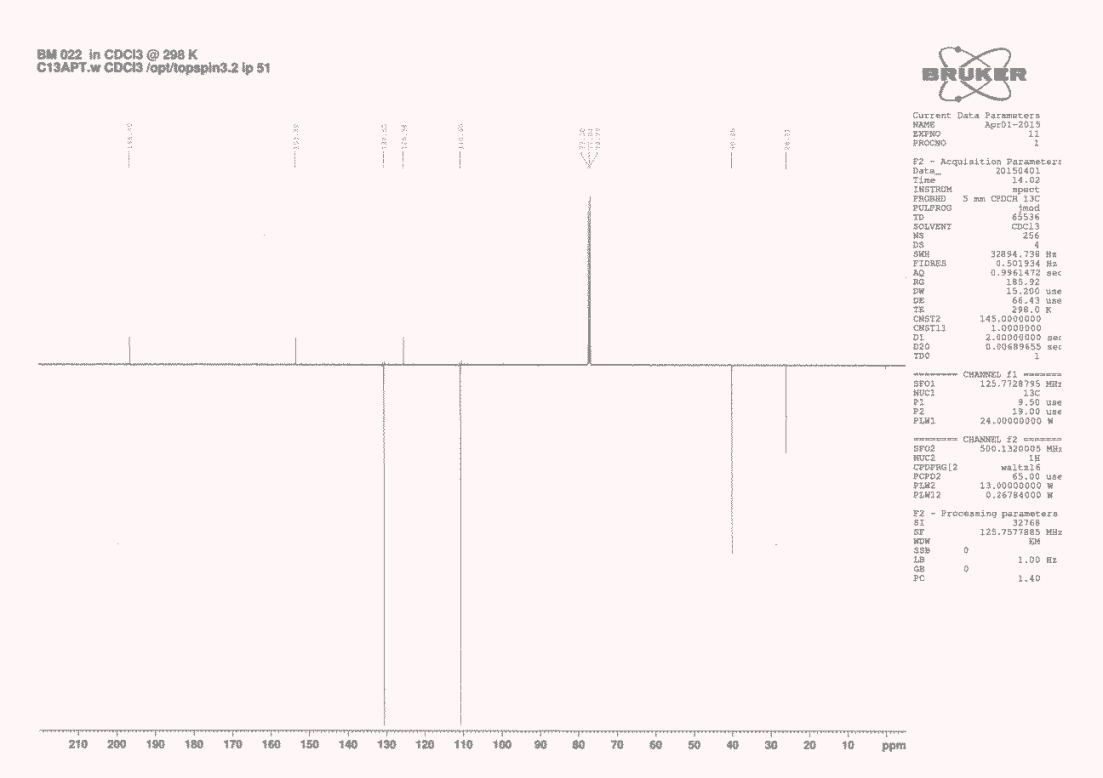


**1-[4-(Dimethylamino)phenyl]ethanone.**

$^{1}H$, 500 MHz, $CDCl_3$

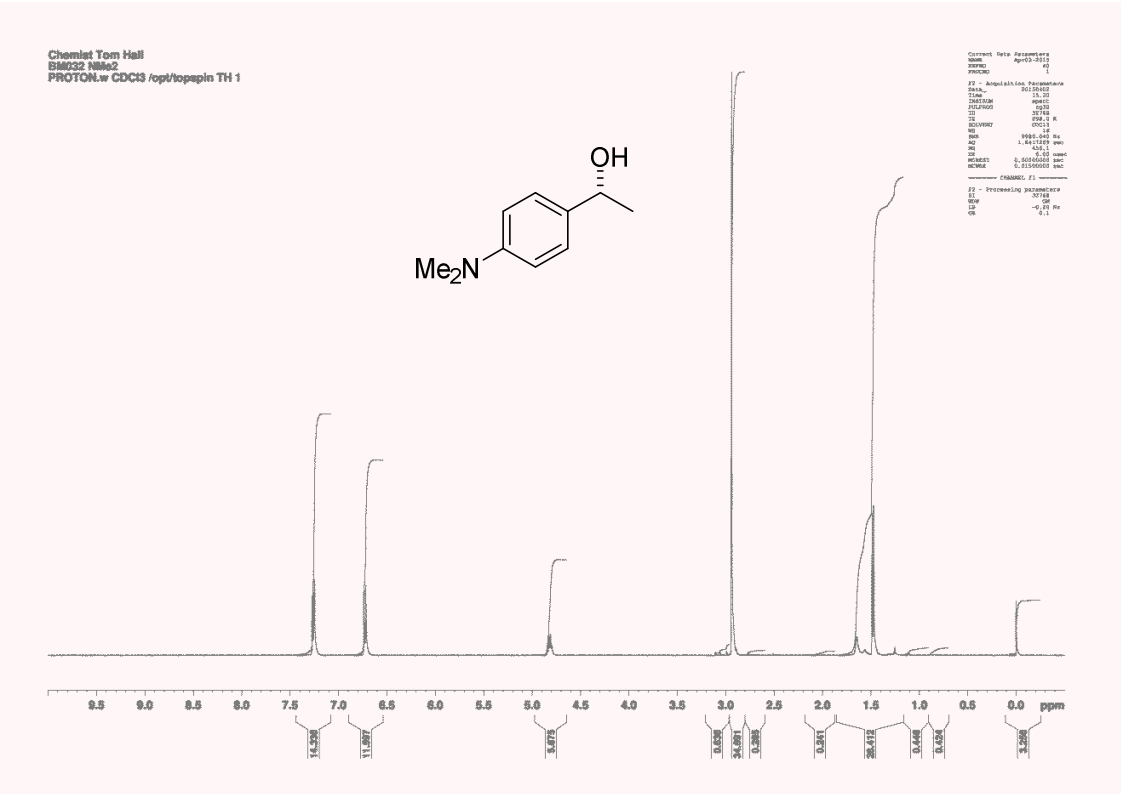



$^{13}C$, 125 MHz, $CDCl_3$

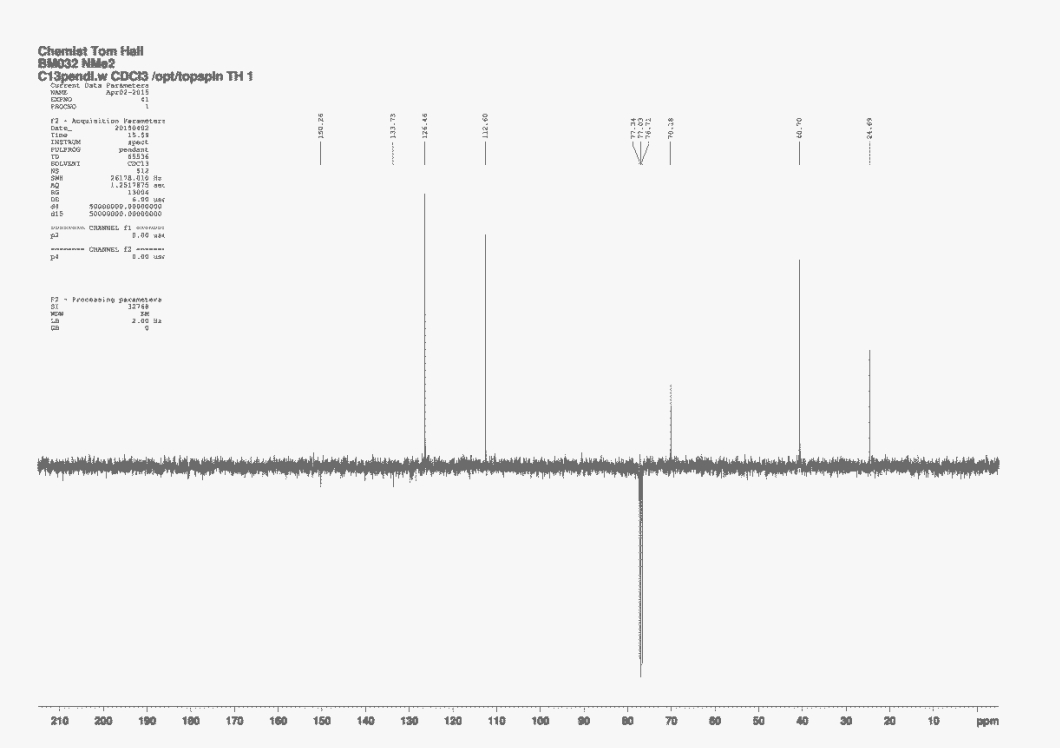


**1-[(4'-Dimethylamino)phenyl]ethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

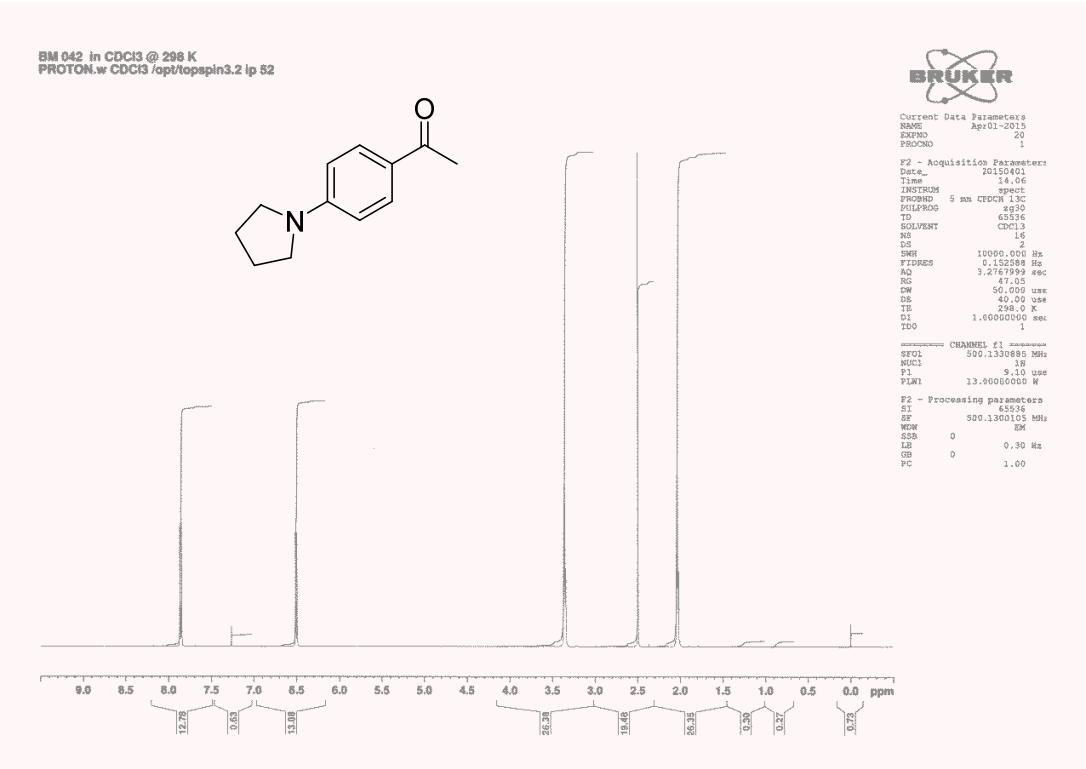



$^{13}$C, 100 MHz, $CDCl_3$.

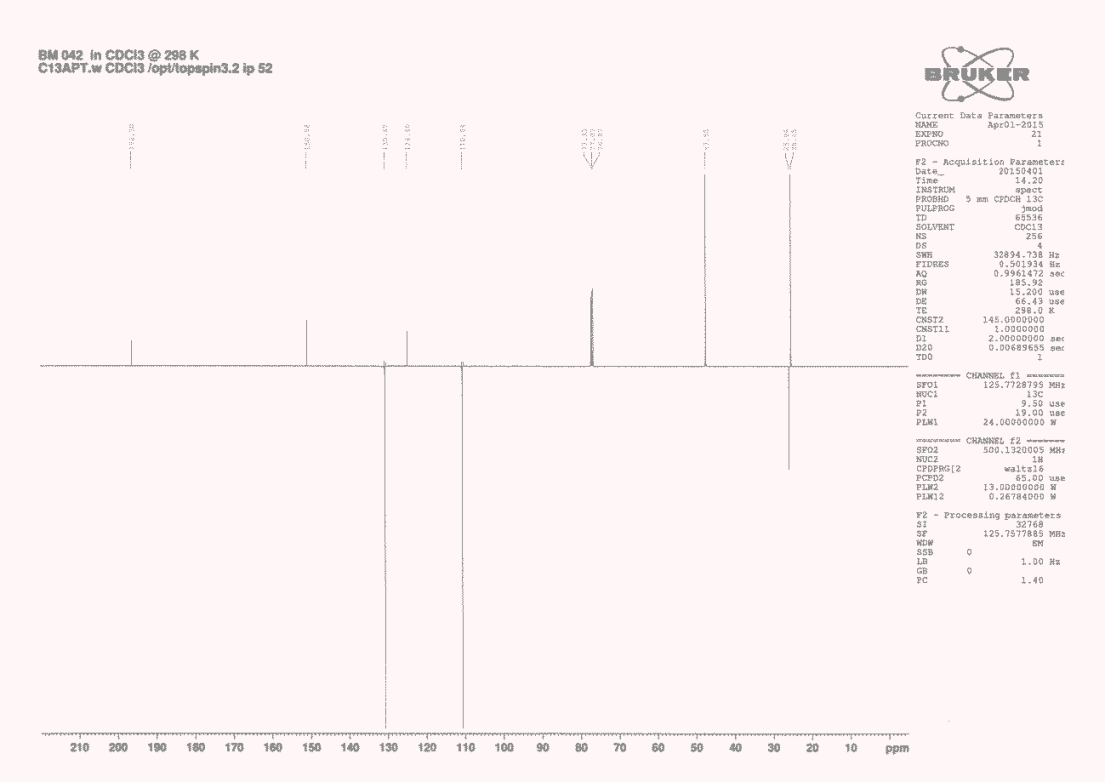


**1-[4-(Pyrrolidin-1-yl)phenyl]ethanone.**

$^{1}H$, 500 MHz, $CDCl_3$

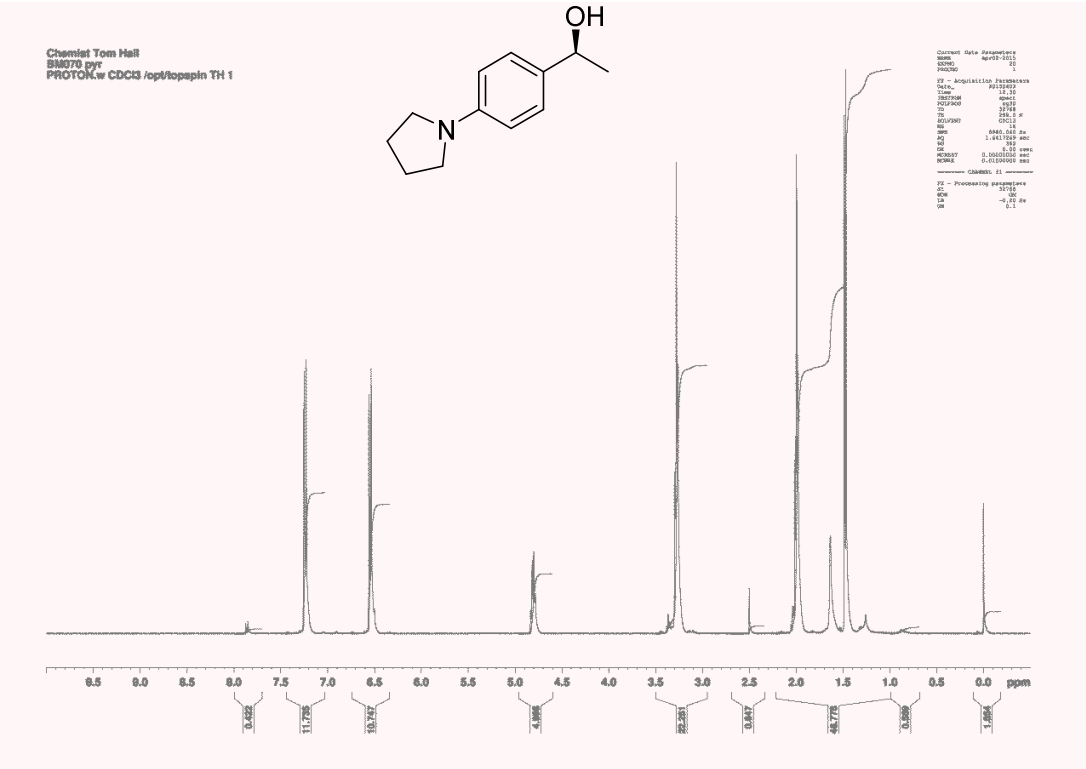



$^{13}C$, 125 MHz, $CDCl_3$.

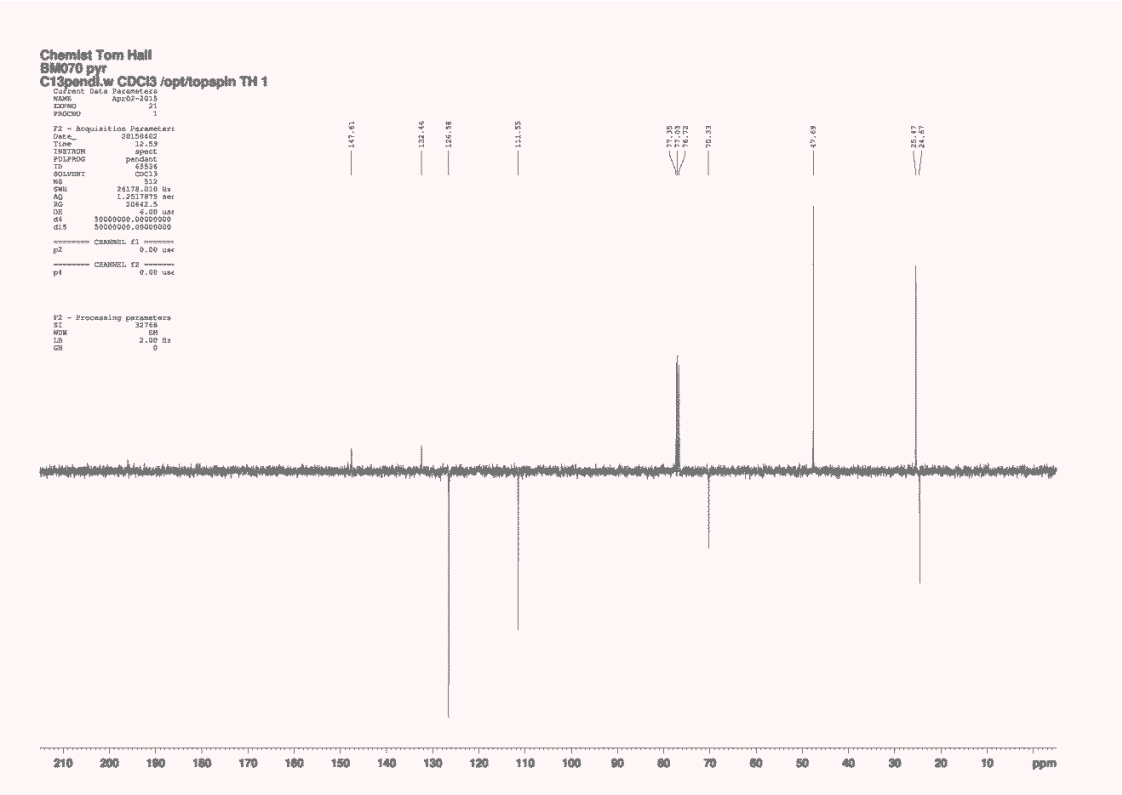


**1-[4-(Pyrrolidin-1-yl)phenyl]ethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

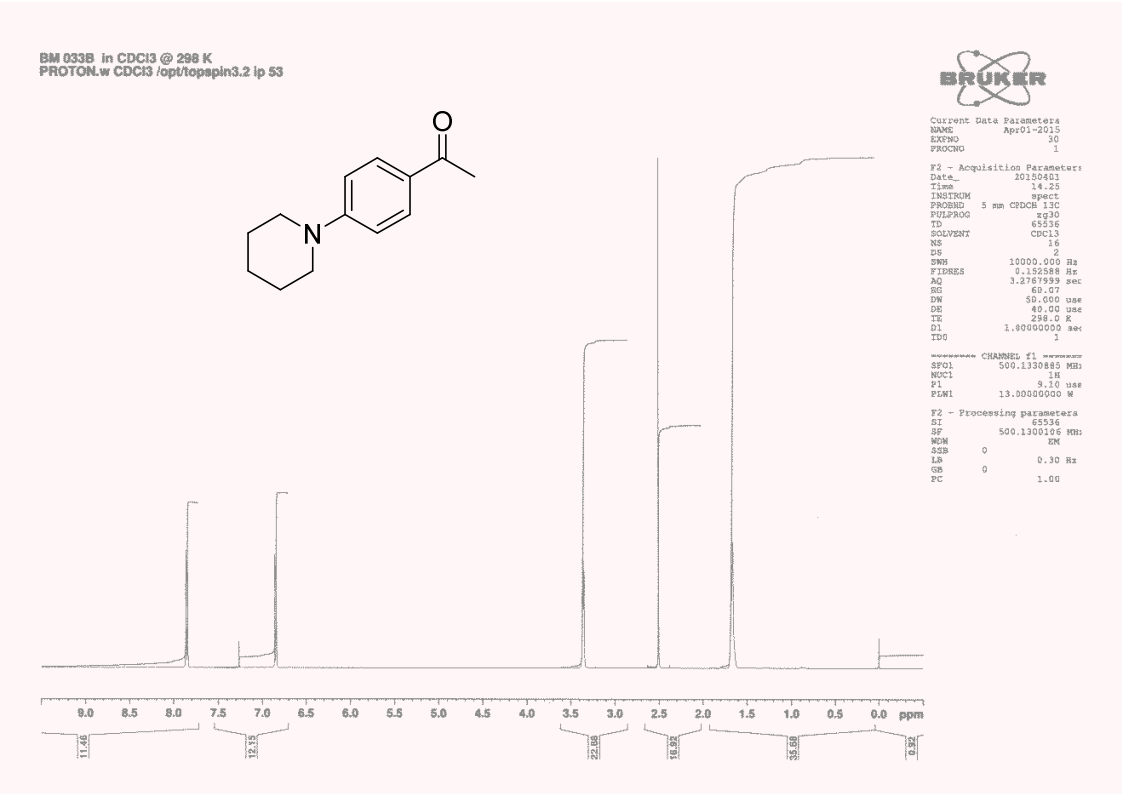



$^{13}$C, 100 MHz, $CDCl_3$

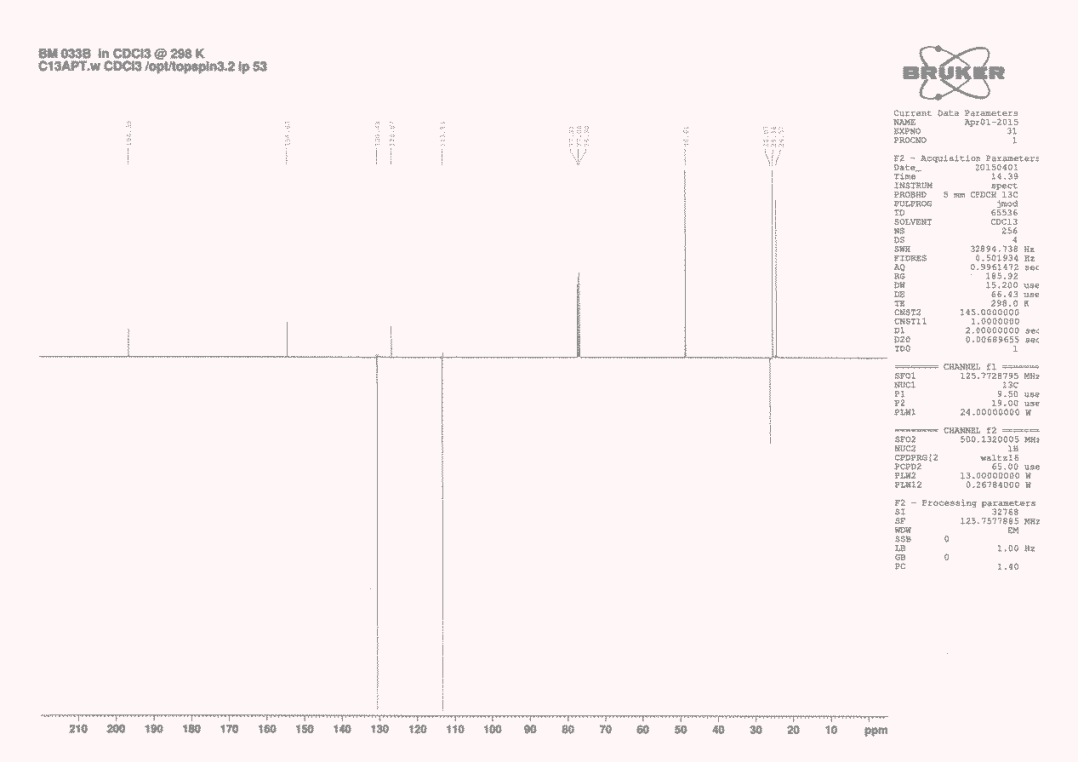


**1-[4-(Piperidin-1-yl)phenyl]ethanone.**

$^{1}$H, 500 MHz, $CDCl_3$

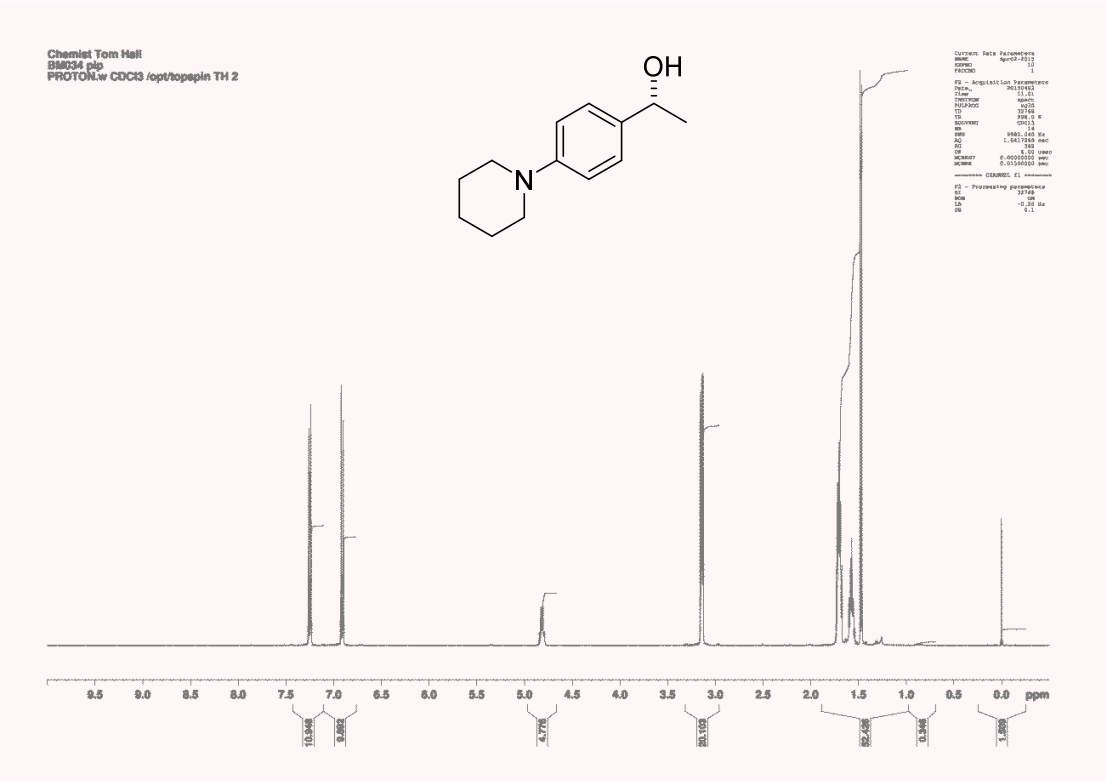



$^{13}$C, 125 MHz, $CDCl_3$

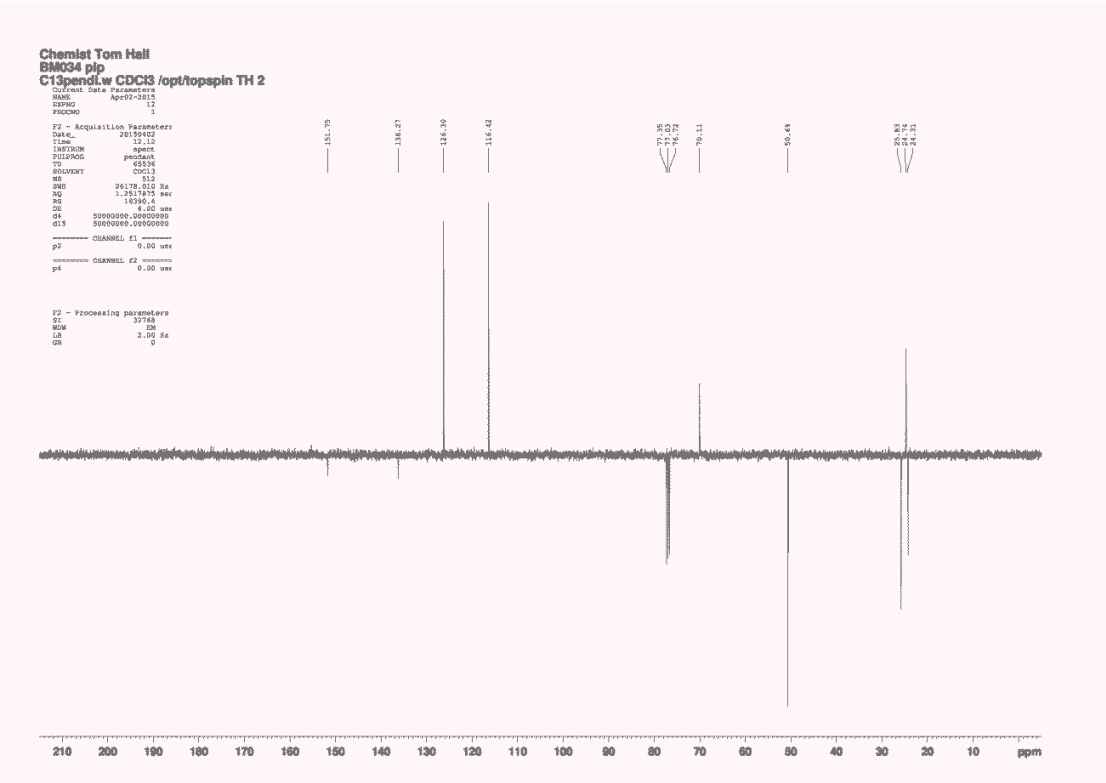


**1-[4-(Piperidin-1-yl)phenyl]ethanol.**

$^{1}H$, 400 MHz, $CDCl_3$

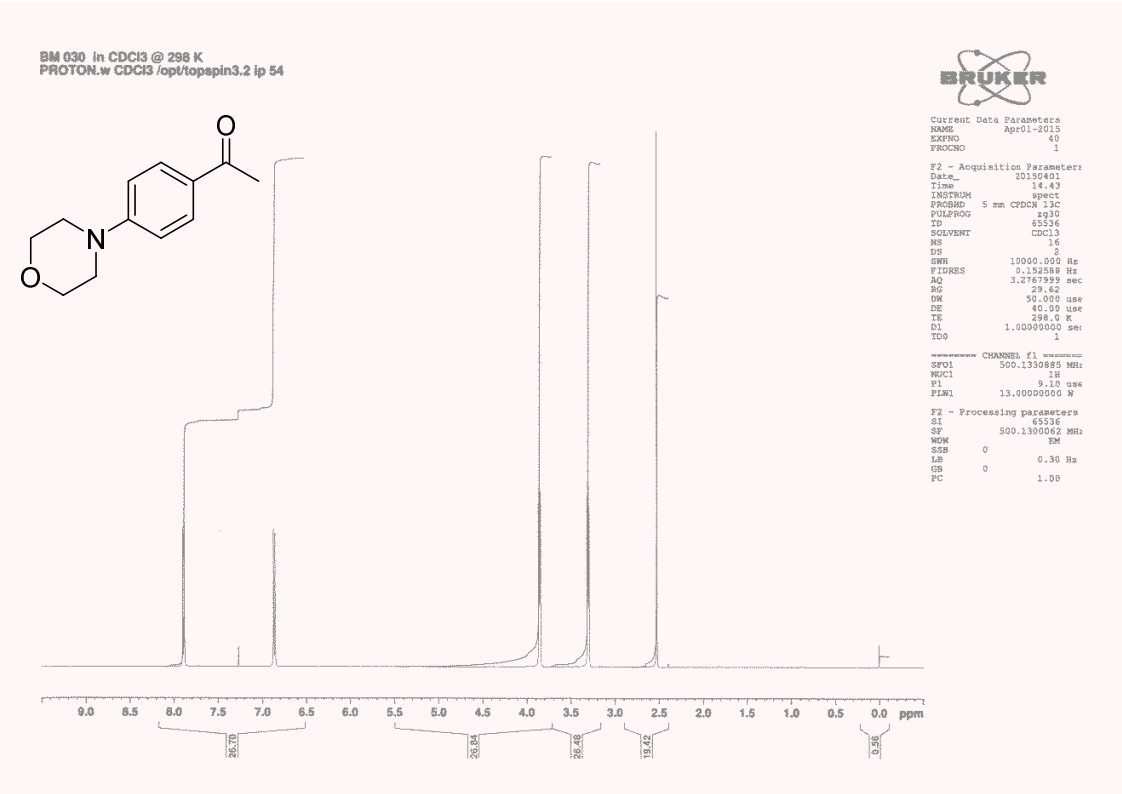



$^{13}C$, 100 MHz, $CDCl_3$

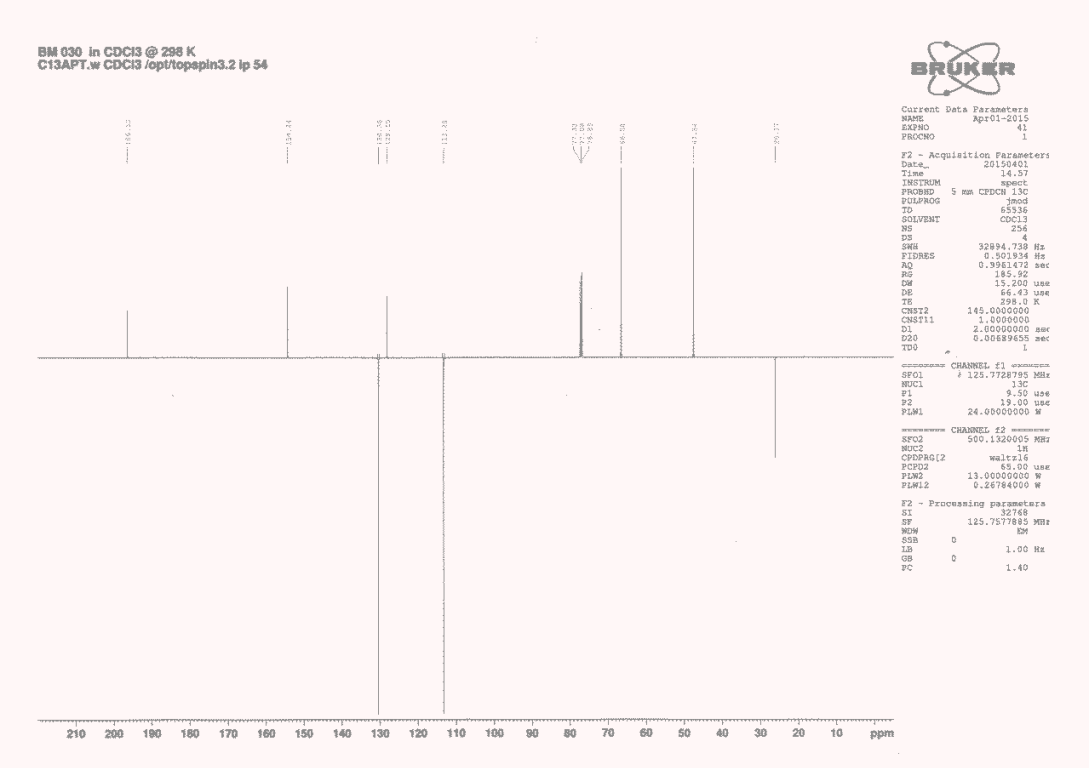


**1-[4-(Morpholin-4-yl)phenyl]ethanone.**

$^{1}$H, 500 MHz, $CDCl_3$

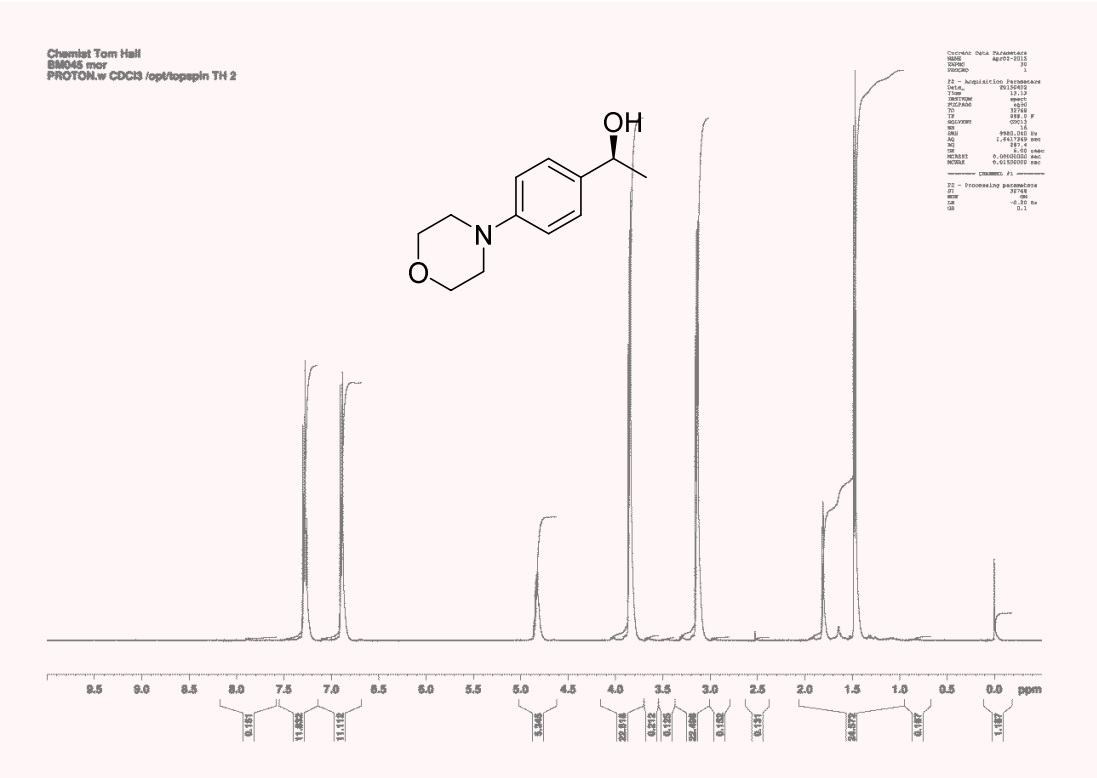



$^{13}$C, 125 MHz, $CDCl_3$

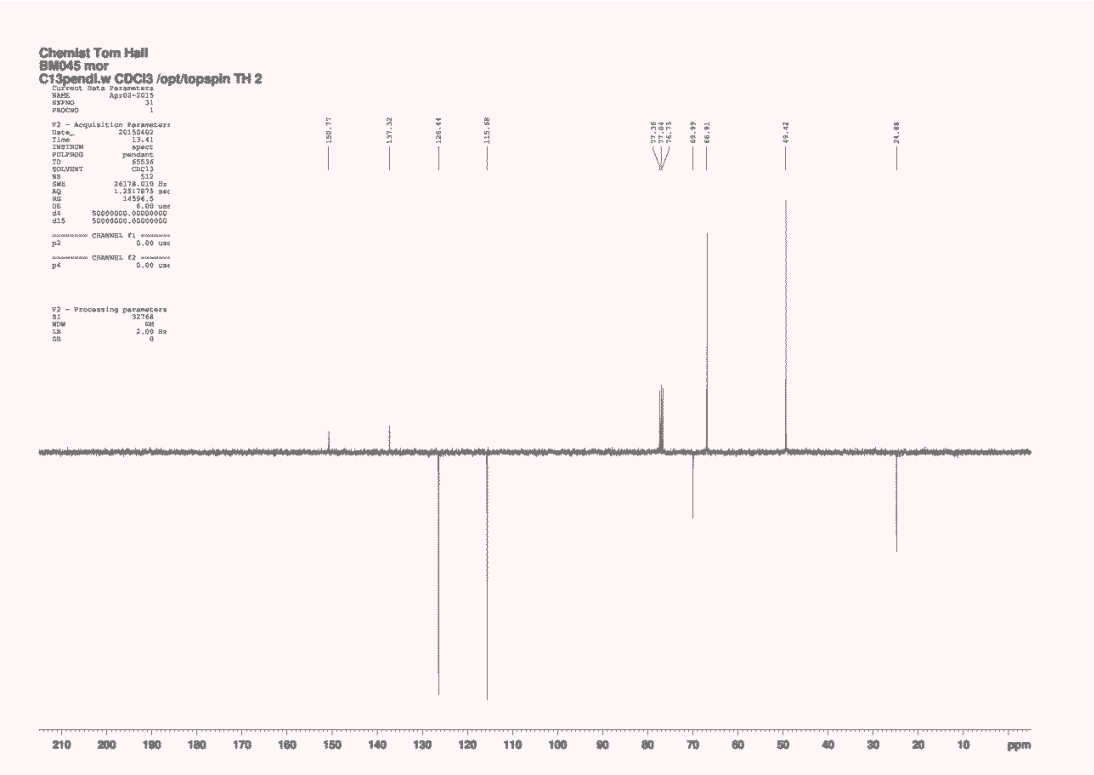


**1-[4-(Morpholin-4-yl)phenyl]ethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

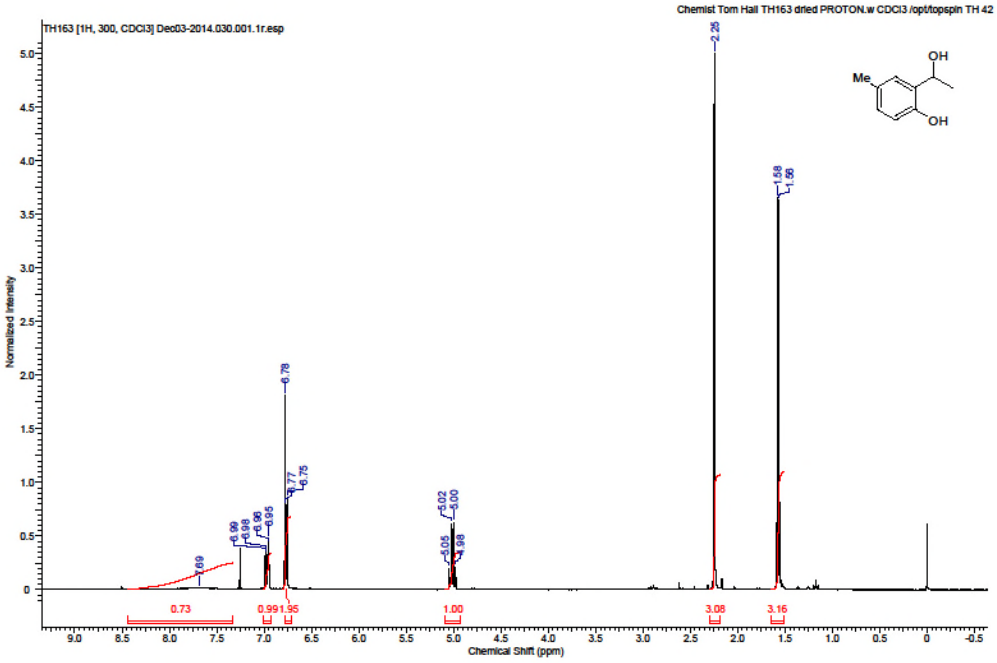



$^{13}$C, 100 MHz, $CDCl_3$

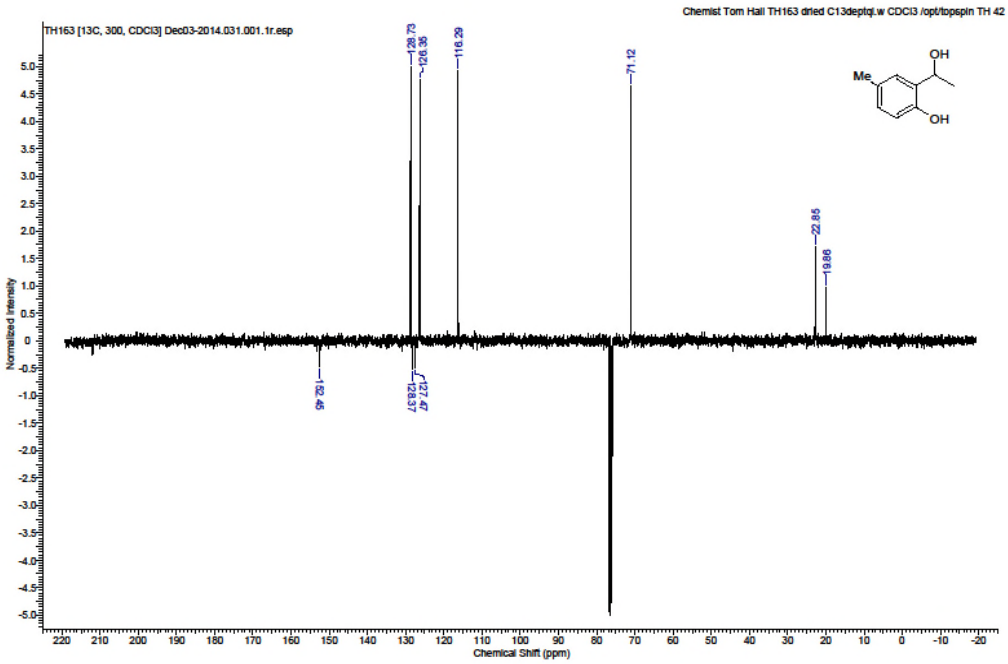


**2-(1-Hydroxyethyl)-4-methylphenol.**

$^{1}H$, 300 MHz, $CDCl_3$

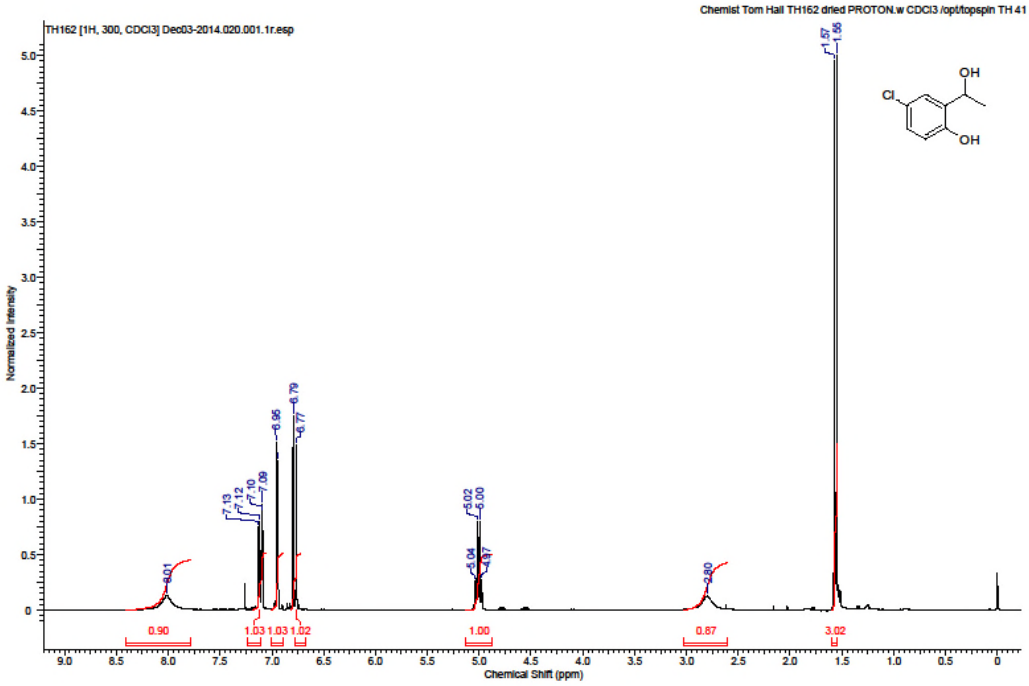



$^{13}C$, 75 MHz, $CDCl_3$

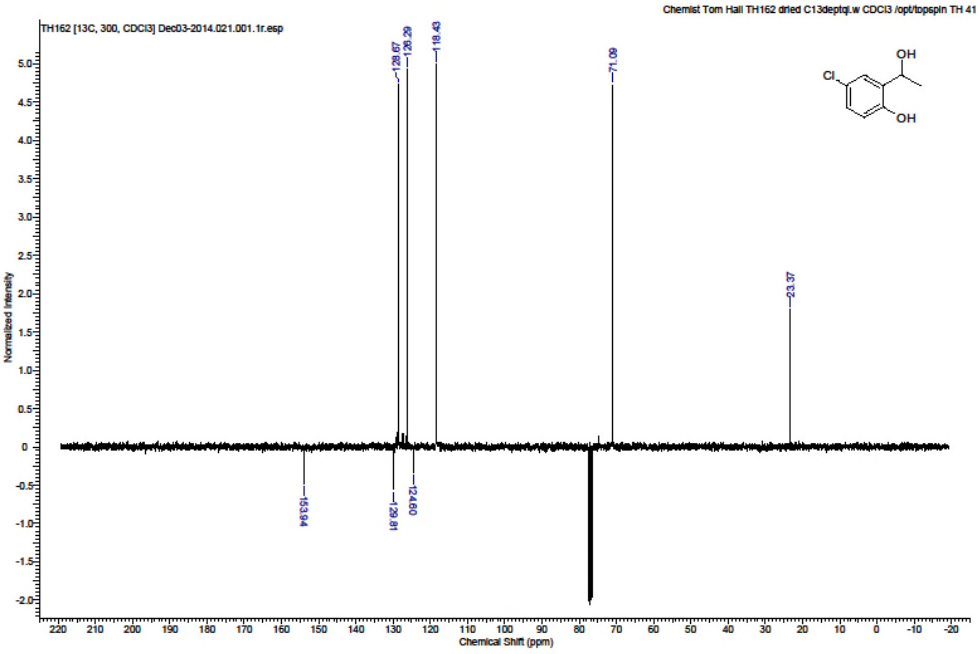


**4-Chloro-2-(1-hydroxyethyl)phenol**

$^{1}$H, 300 MHz, $CDCl_3$

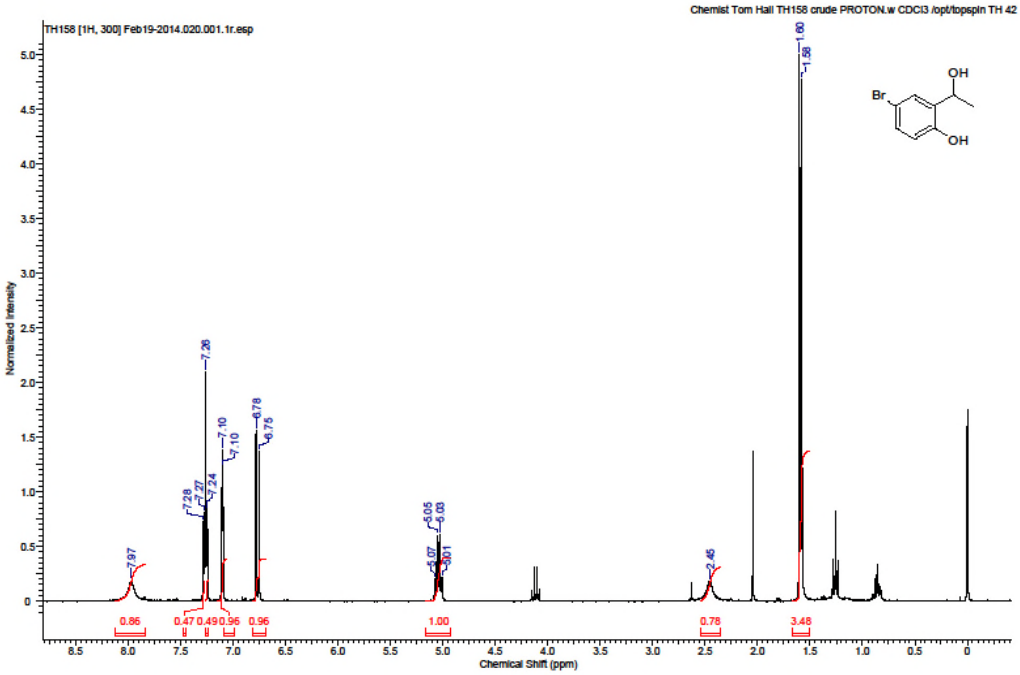



$^{13}$C, 75 MHz, $CDCl_3$

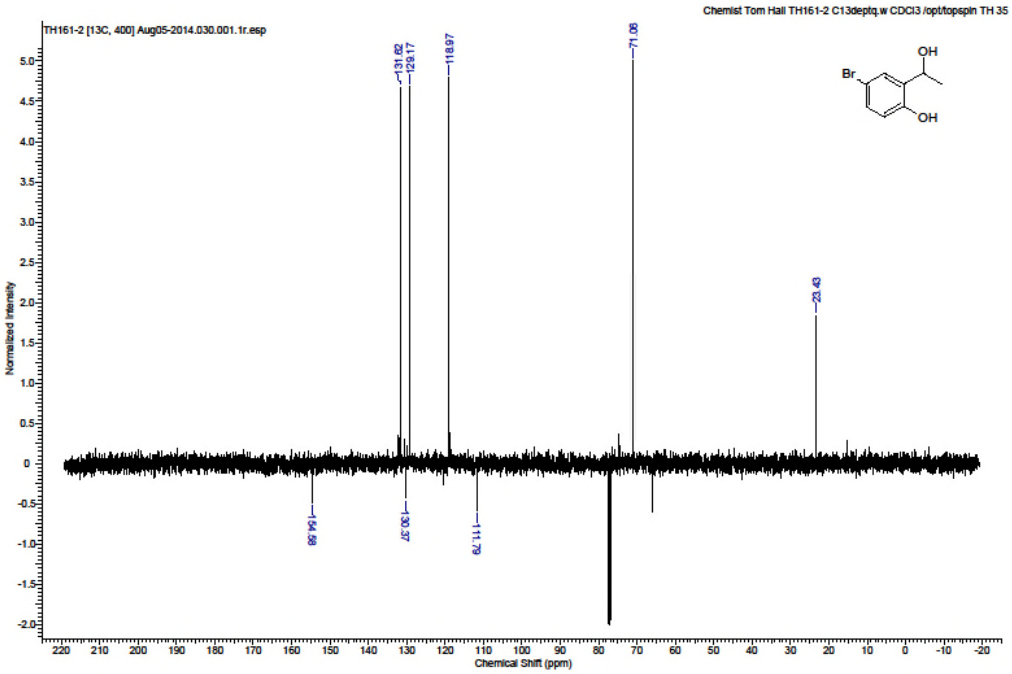


**4-Bromo-2-(1-hydroxyethyl)phenol**

$^{1}H$, 300 MHz, $CDCl_3$

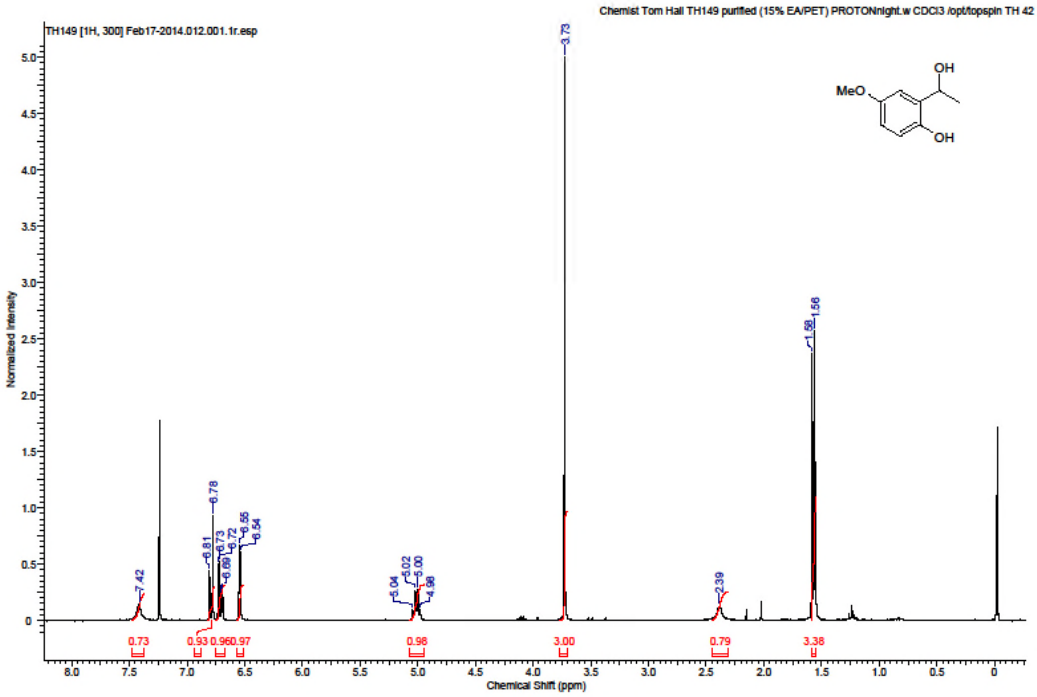



$^{13}C$, 100 MHz, $CDCl_3$

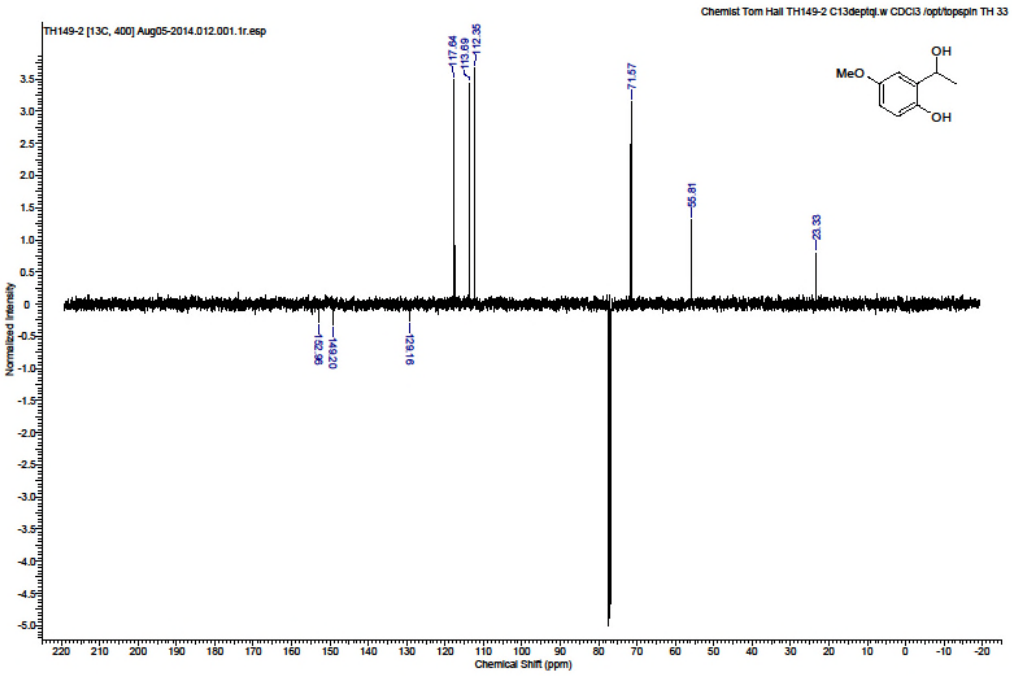


**2-(1-Hydroxyethyl)-4-methoxyphenol**

$^{1}$H, 300 MHz, $CDCl_3$

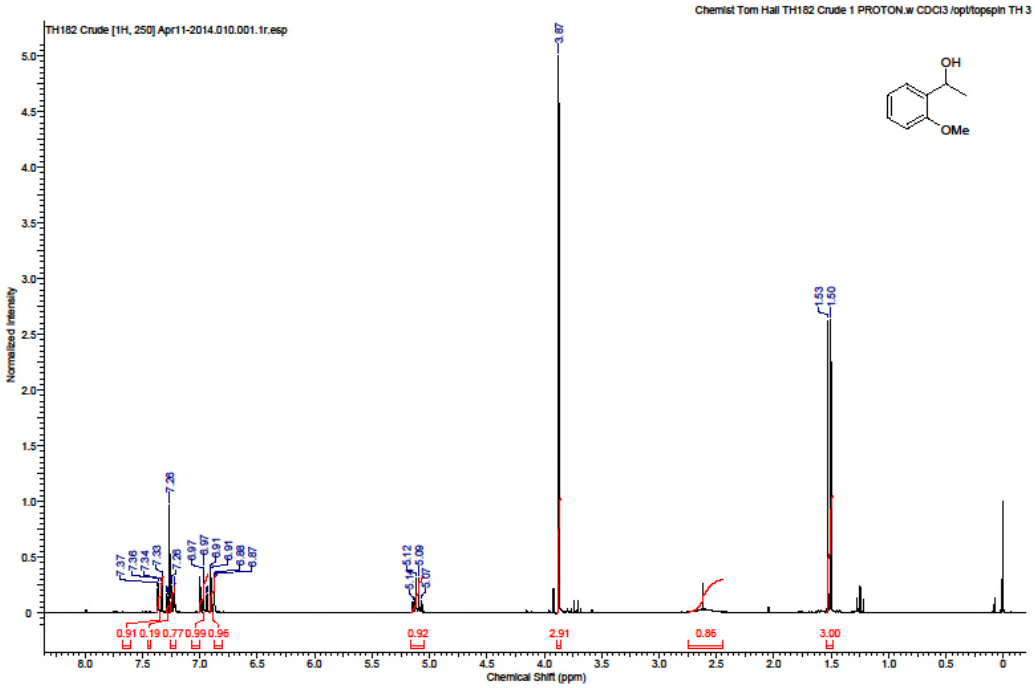



$^{13}$C, 100 MHz, $CDCl_3$

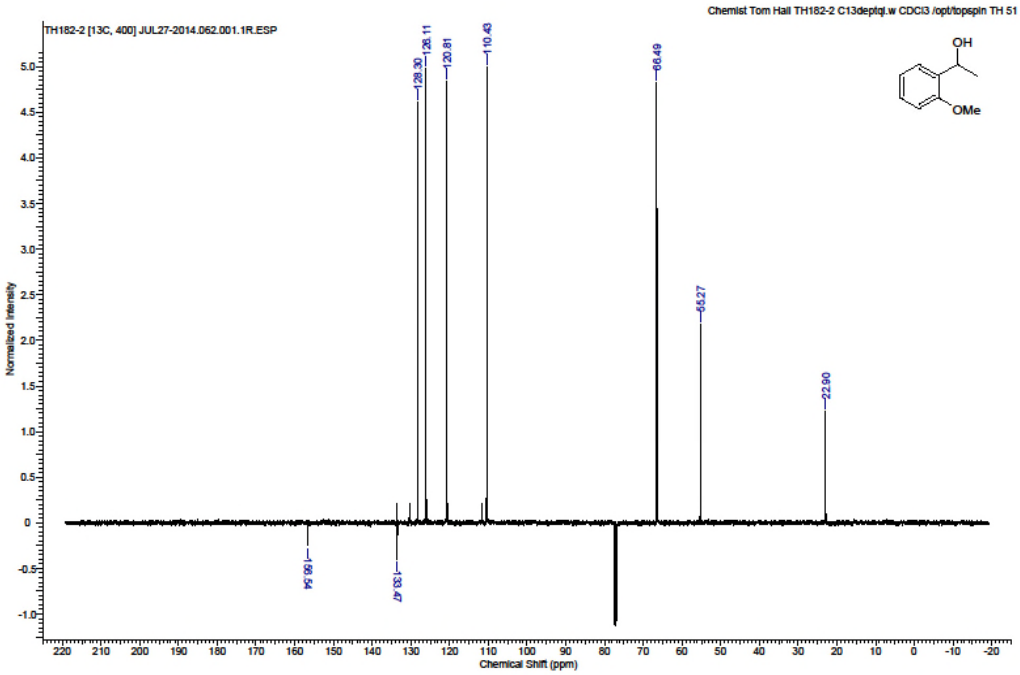


**1-(2-Methoxyphenyl)ethanol**

$^{1}H$, 250 MHz, $CDCl_3$

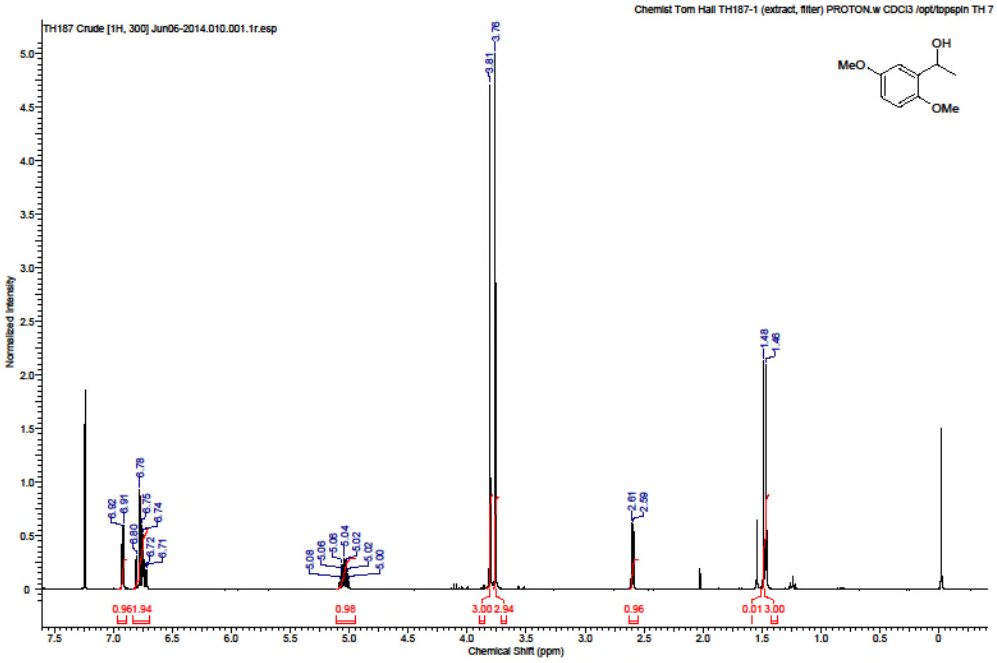



$^{13}C$, 100 MHz, $CDCl_3$



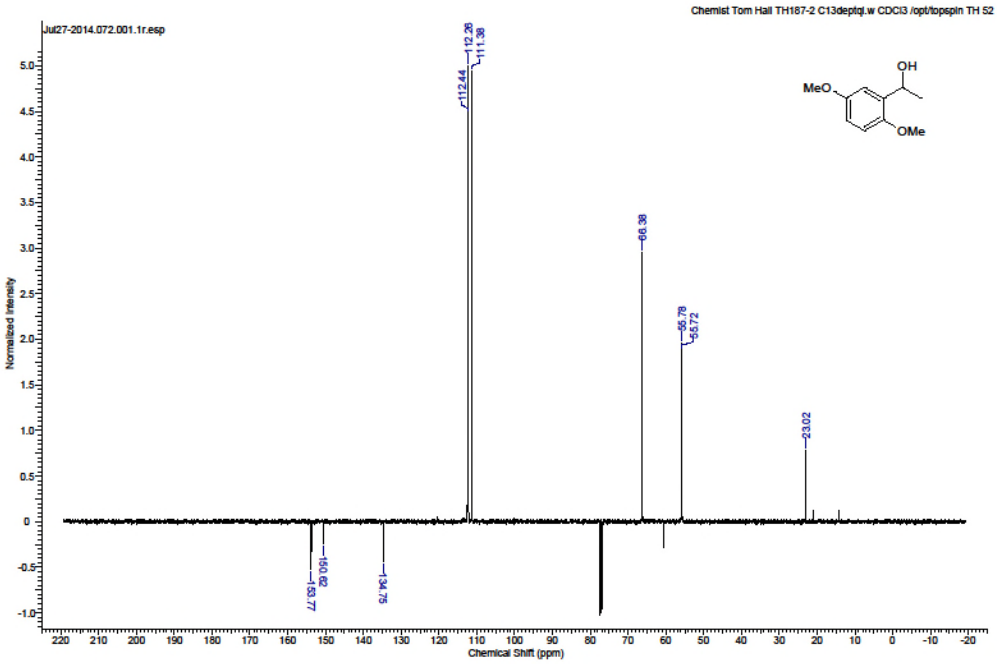
**1-(2,5-Dimethoxyphenyl)ethan-1-ol**

$^{1}$H, 300 MHz, $CDCl_3$



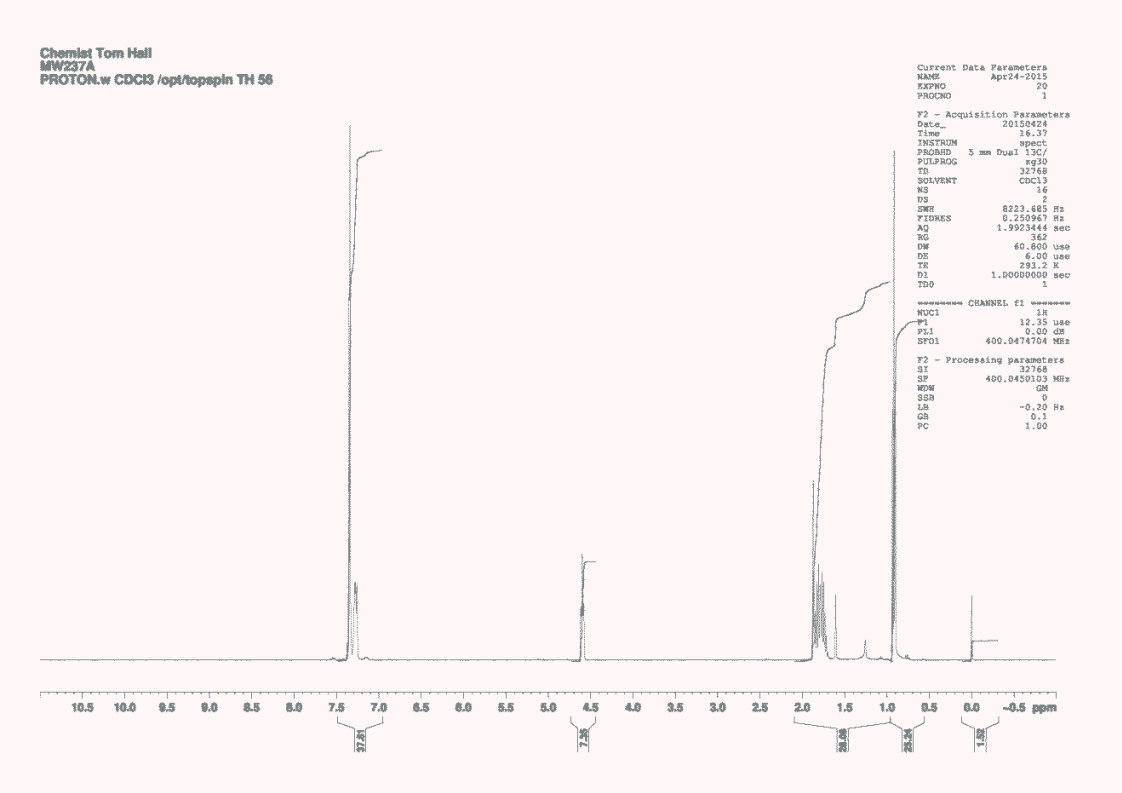

$^{13}$C,

$^{13}$C, 100 MHz, $CDCl_3$

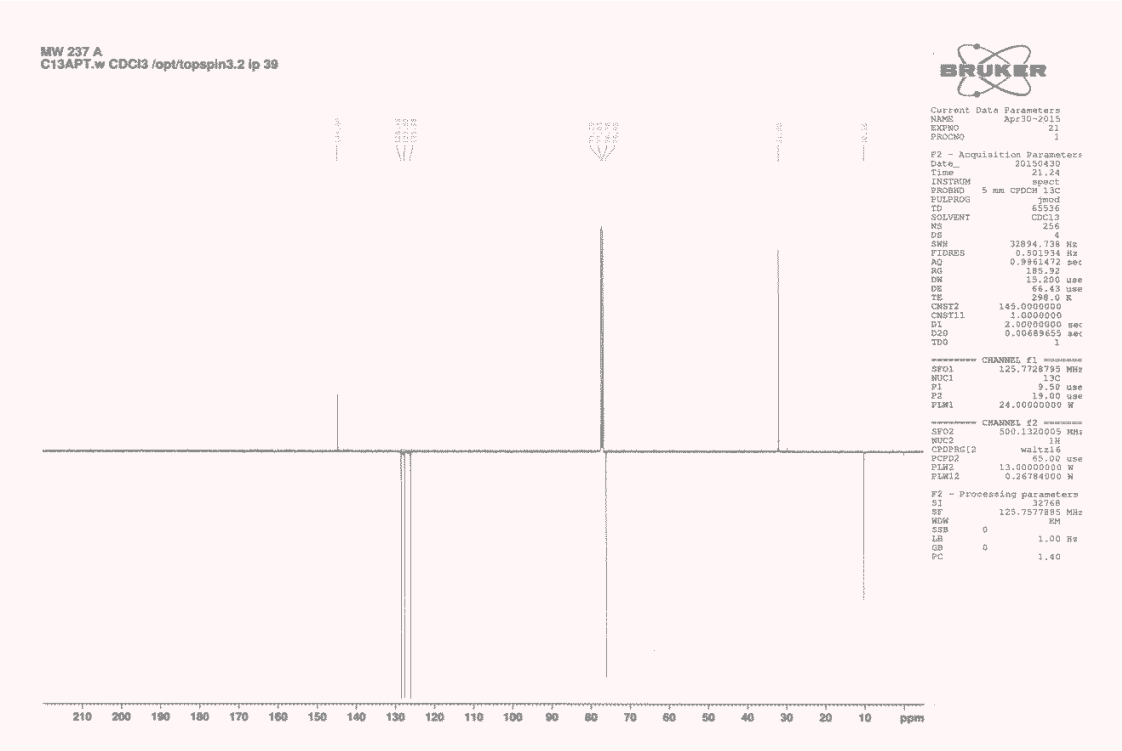


**(*R*)-1-Phenyl-1-propanol.**

$^{1}$H, 400 MHz, $CDCl_3$

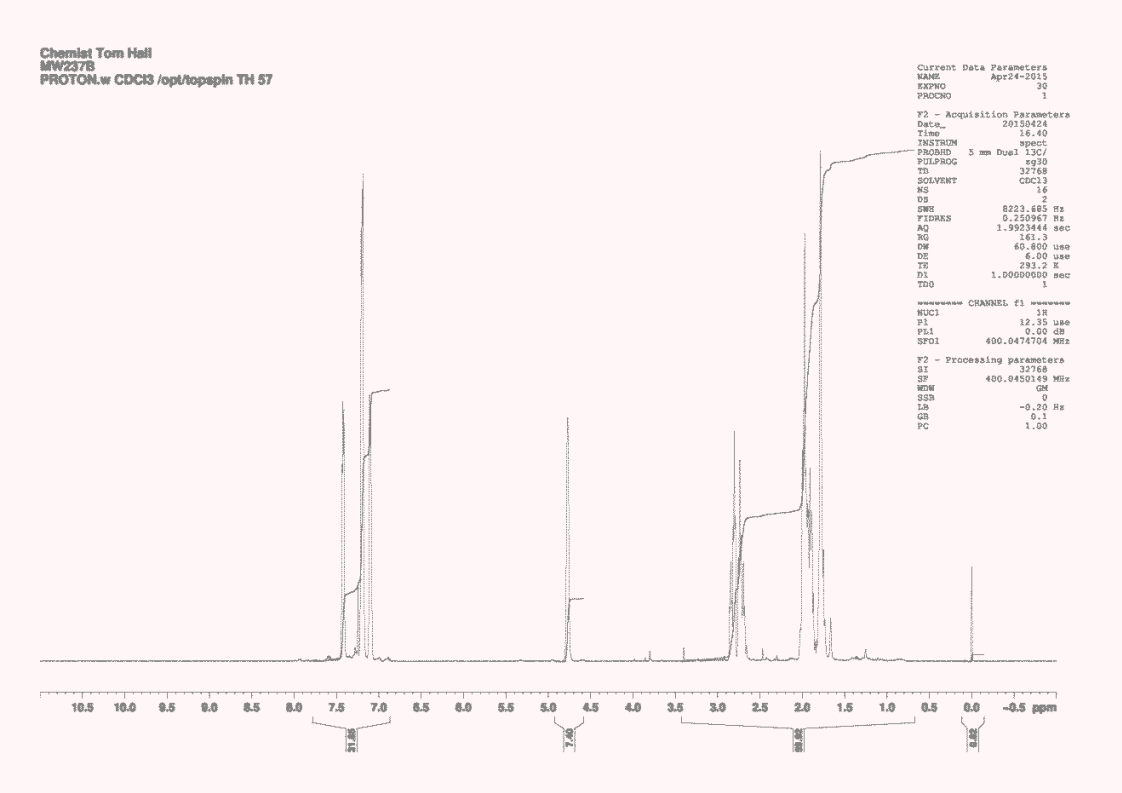



$^{13}$C, 125 MHz, $CDCl_3$

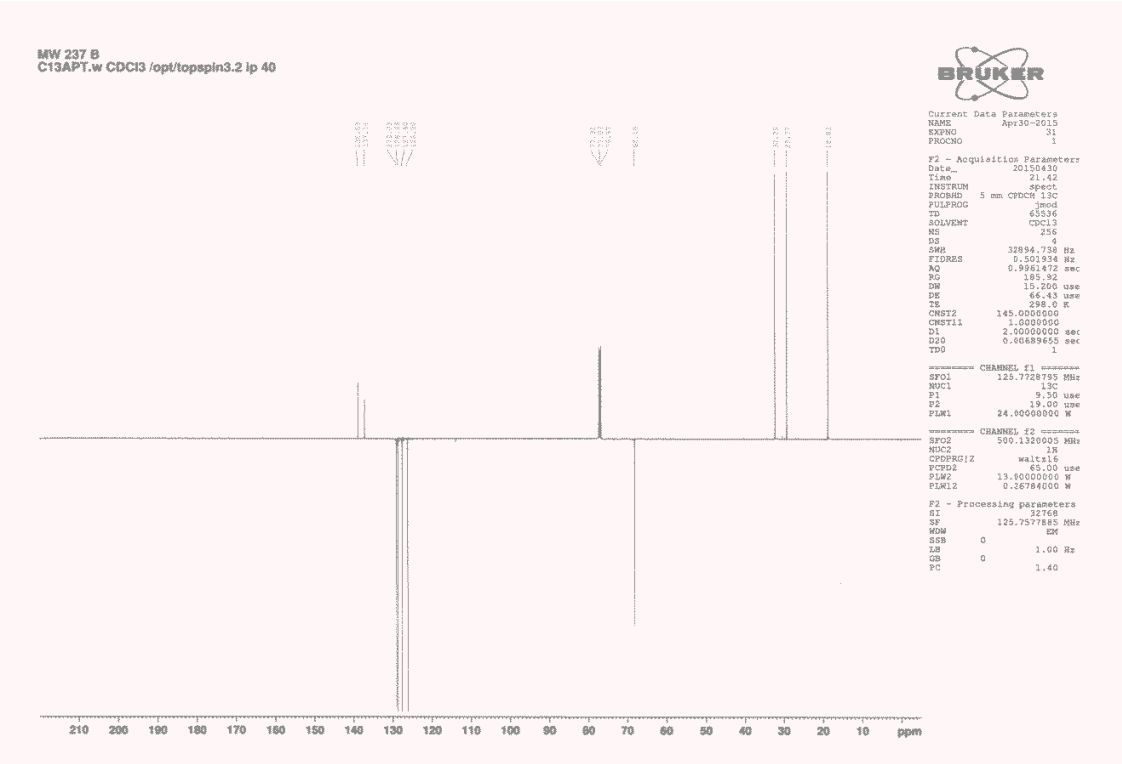


**(*R*)-1-Tetralol.**

$^{1}$H, 400 MHz, $CDCl_3$

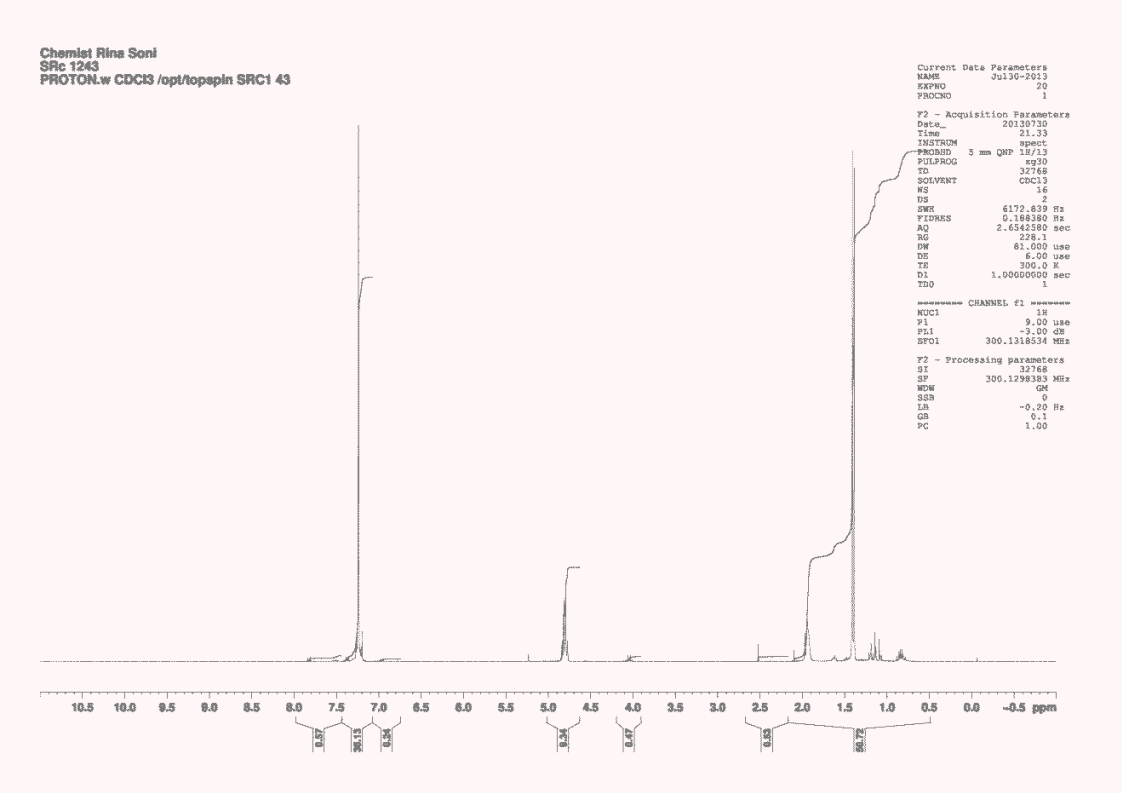



$^{13}$C, 125 MHz, $CDCl_3$

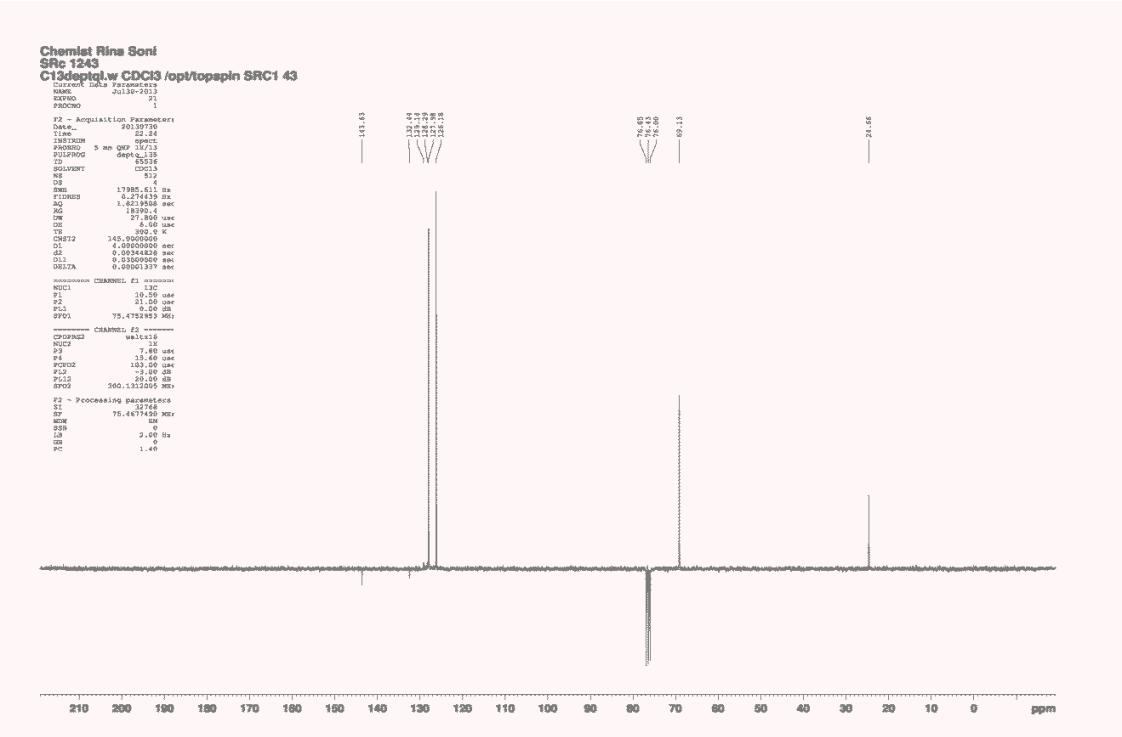


**(*R*)-1-(4-Chlorophenyl)ethanol.**

$^{1}$H, 300 MHz, $CDCl_3$

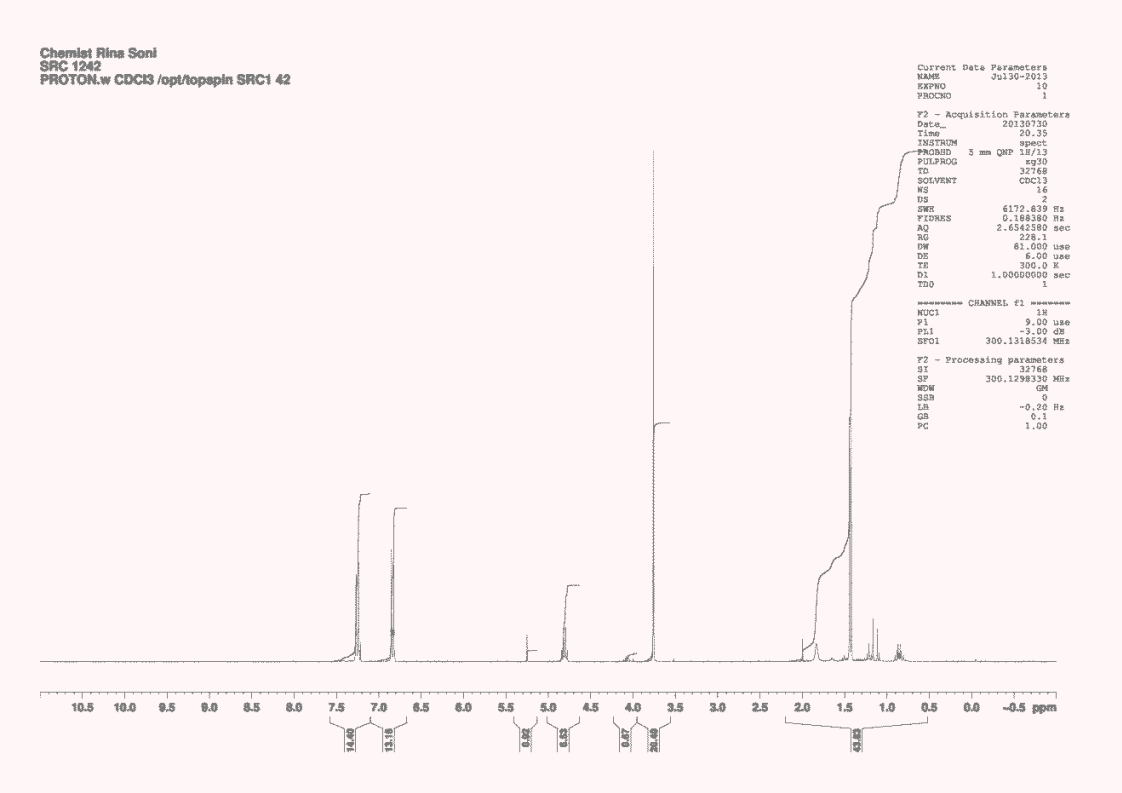



$^{13}$C, 75 MHz, $CDCl_3$

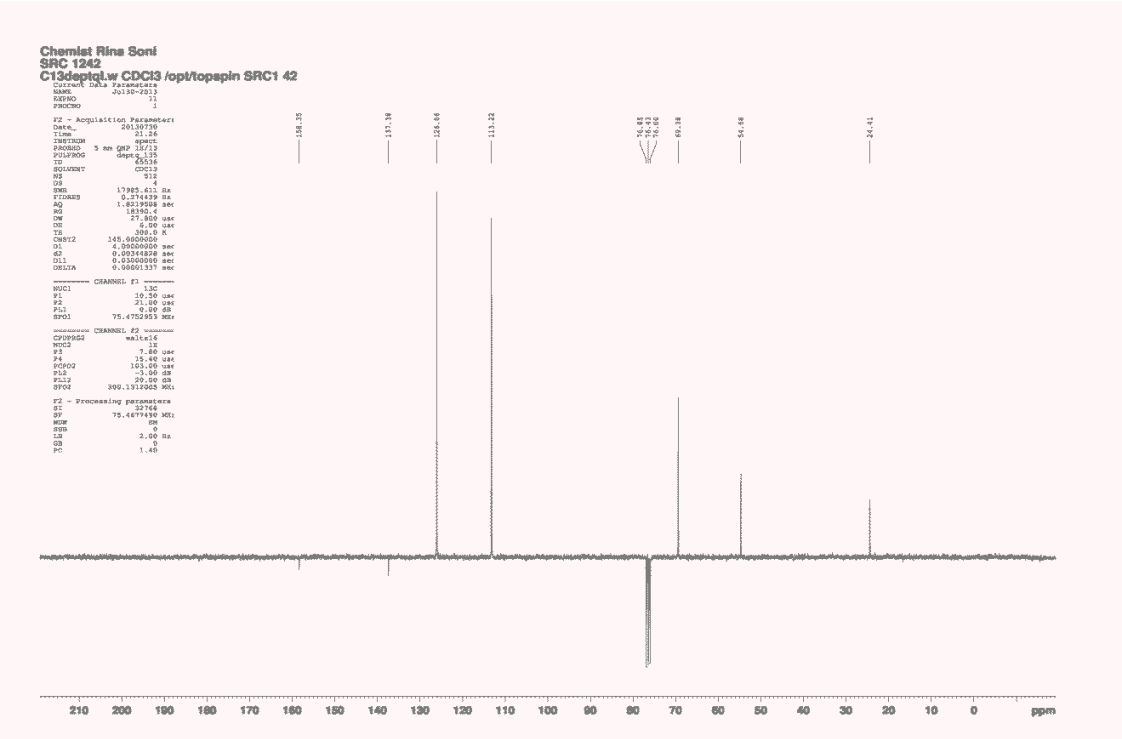


**(*R*)-1-(4-Methoxyphenyl)ethanol.**

$^{1}$H, 300 MHz, $CDCl_3$

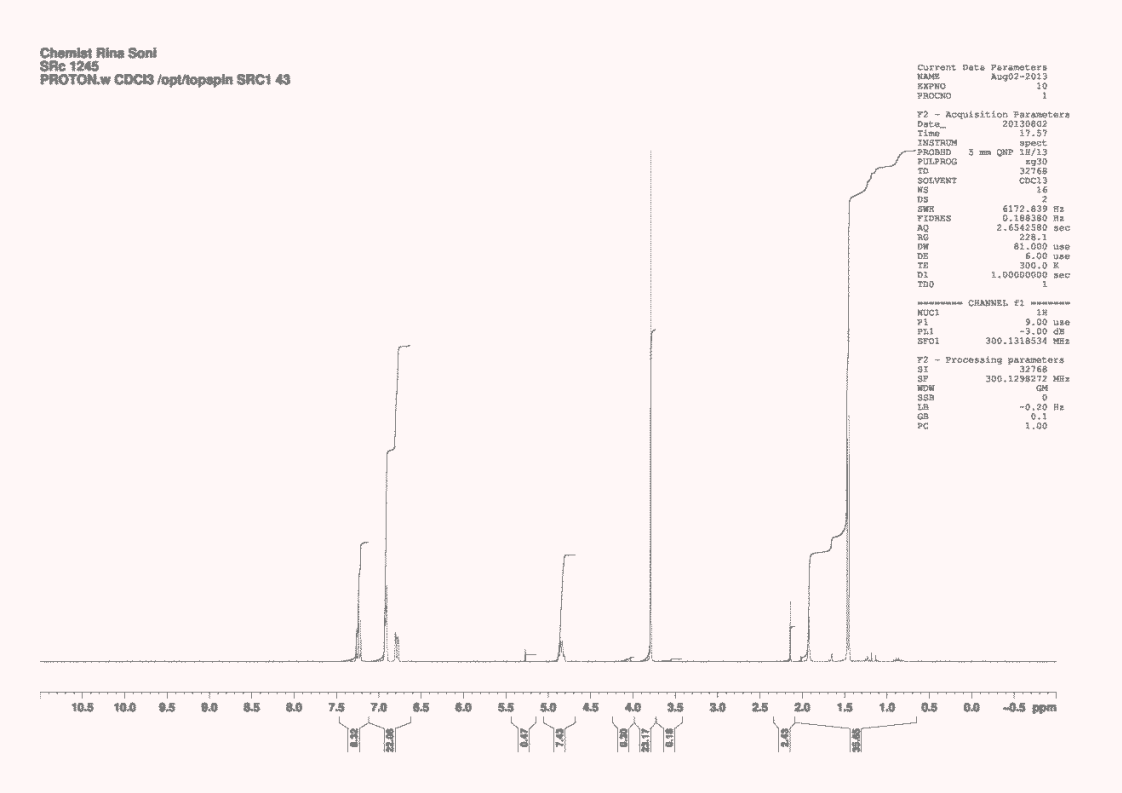



$^{13}$C, 75 MHz, $CDCl_3$

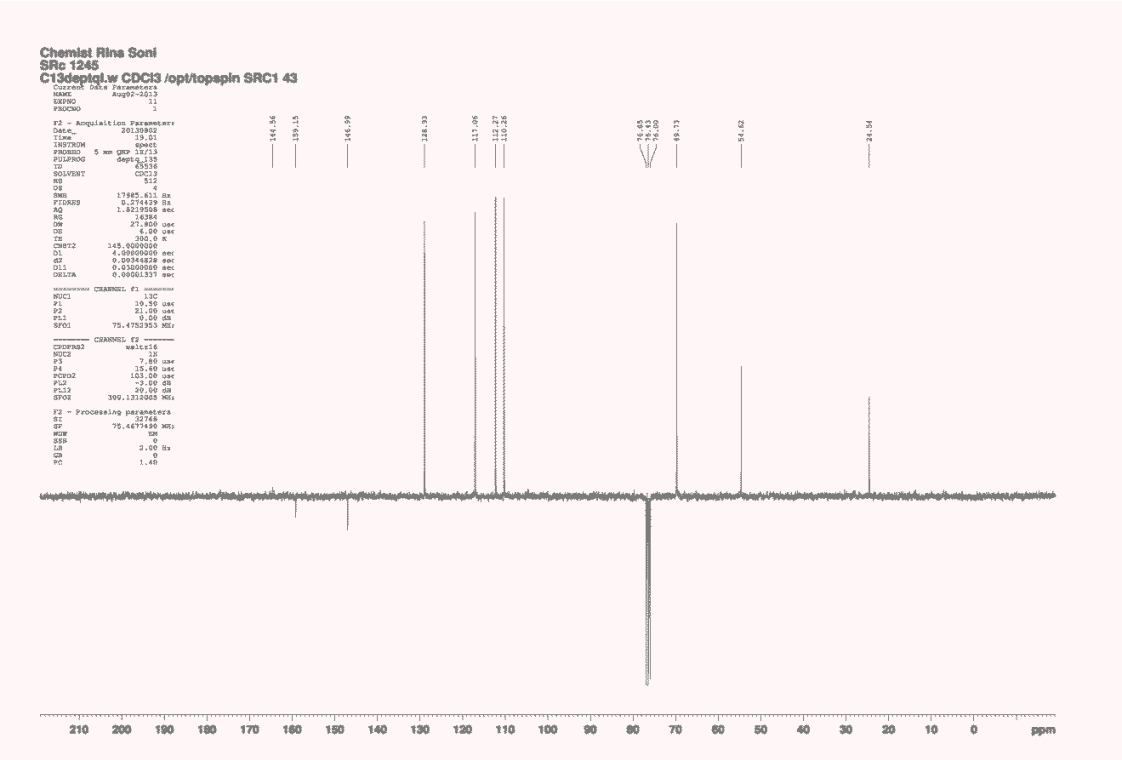


**(*R*)-1-(3-Methoxyphenyl)ethanol.**

$^{1}$H, 300 MHz, $CDCl_3$

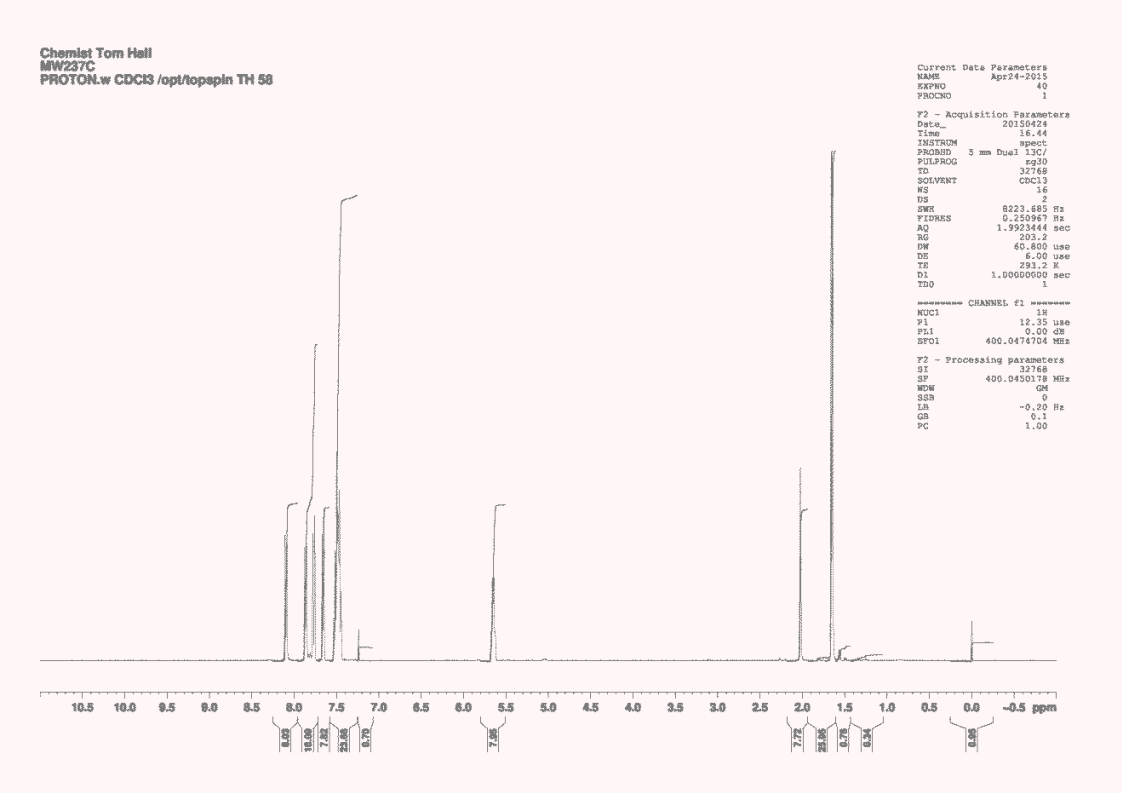



$^{13}$C, 75 MHz, $CDCl_3$

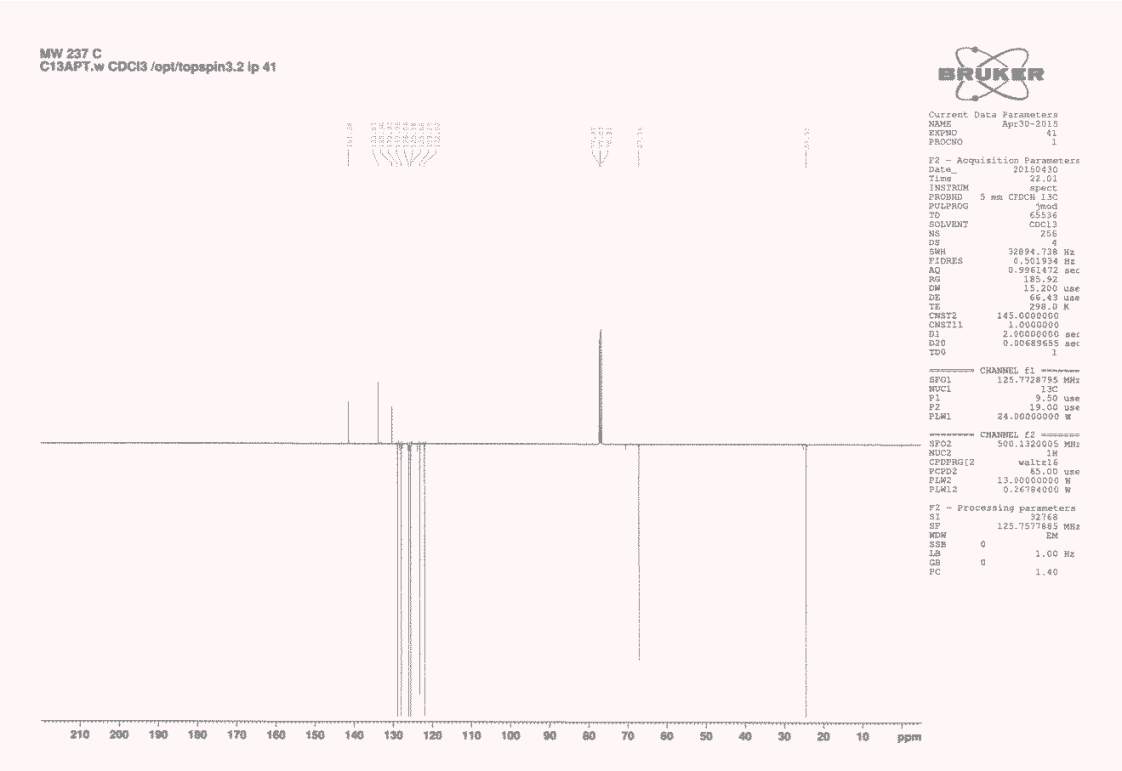


**(*R*)-1-(1-Naphthyl)ethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

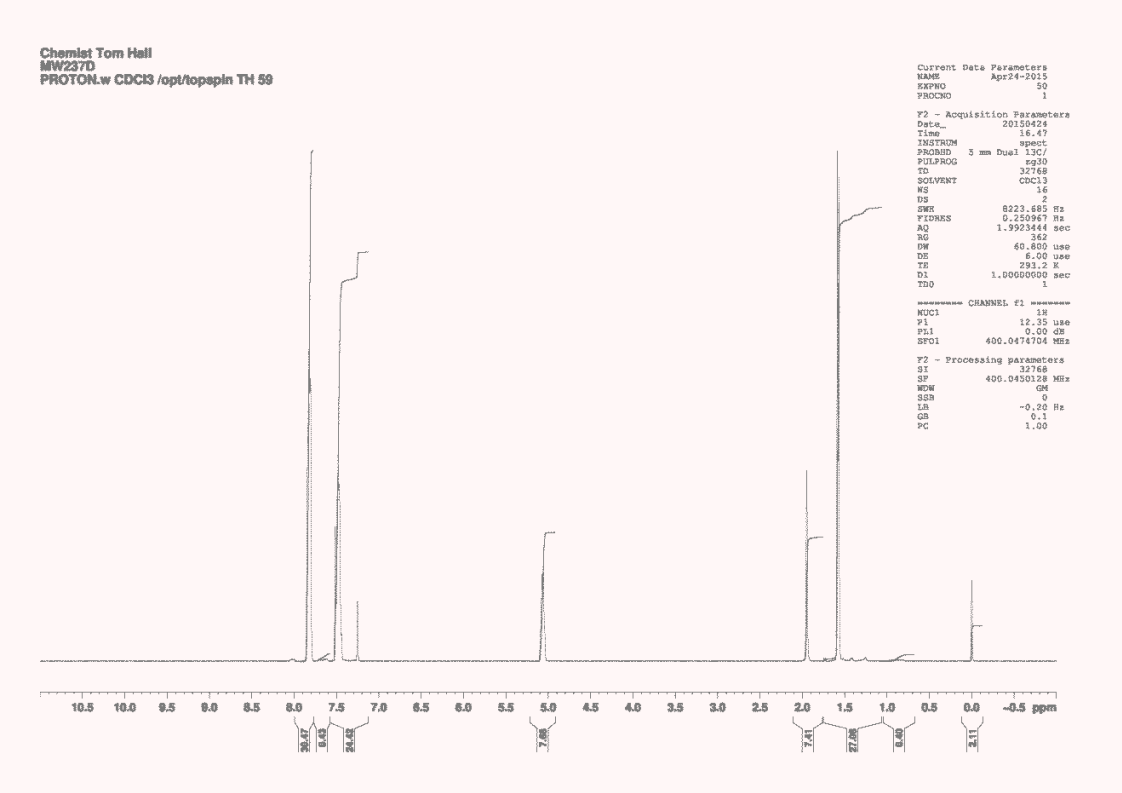



$^{13}$C, 125 MHz, $CDCl_3$

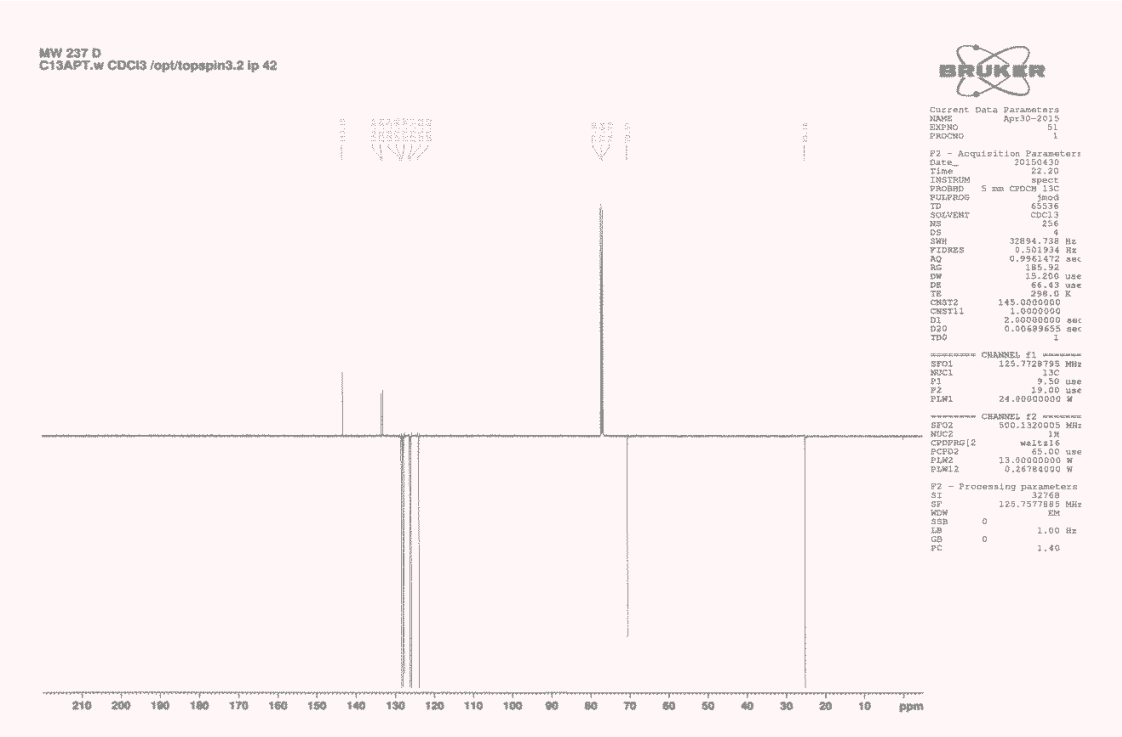


**(*R*)-1-(2-Naphthyl)ethanol.**

$^{1}$H, 400 MHz, $CDCl_3$

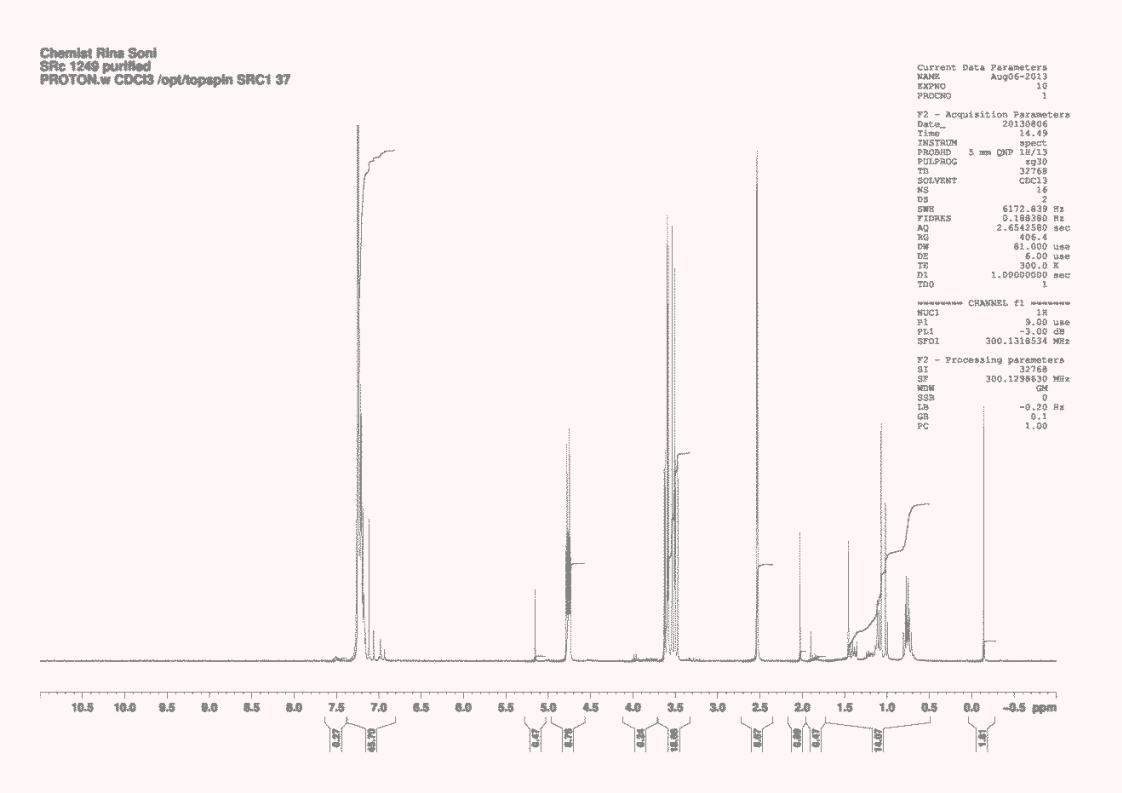



$^{13}$C, 125 MHz, $CDCl_3$

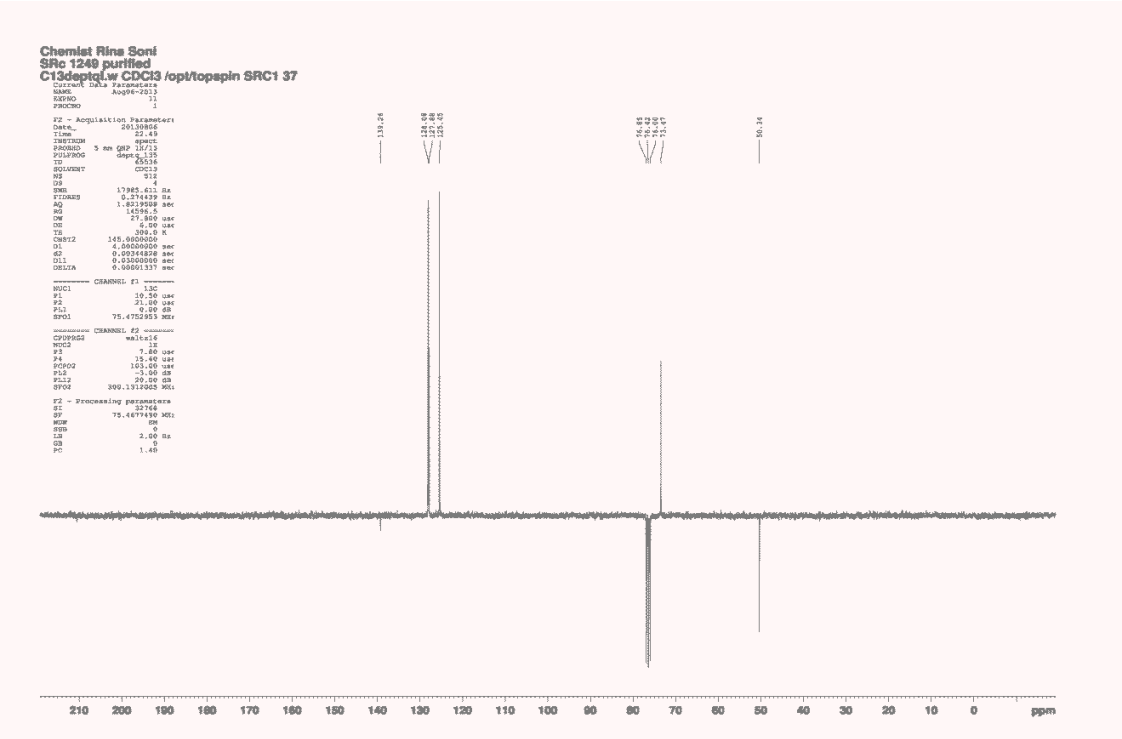


**(*S*)-1-Phenyl-2-chloroethanol.**

$^{1}$H, 300 MHz, $CDCl_3$

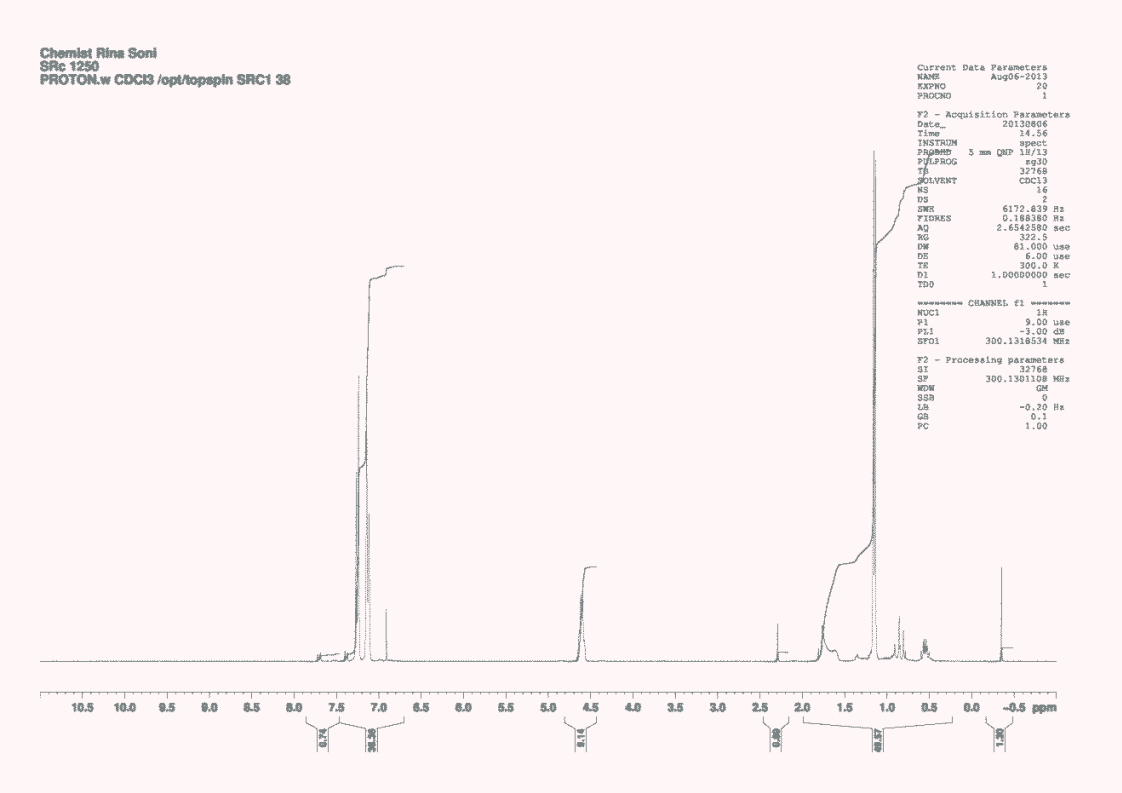



$^{13}$C, 75 MHz, $CDCl_3$

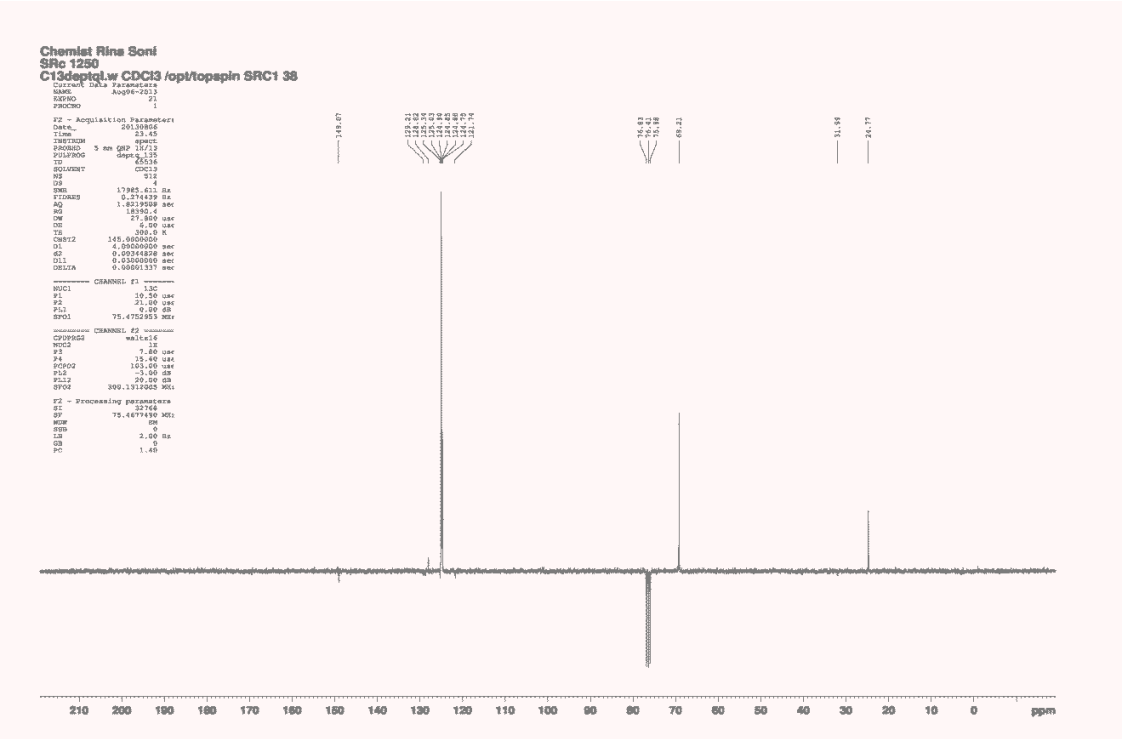


**(*R*)-1-(4-Trifluoromethylphenyl)ethanol.** *zero - 0.3.*

$^{1}$H, 300 MHz, $CDCl_3$

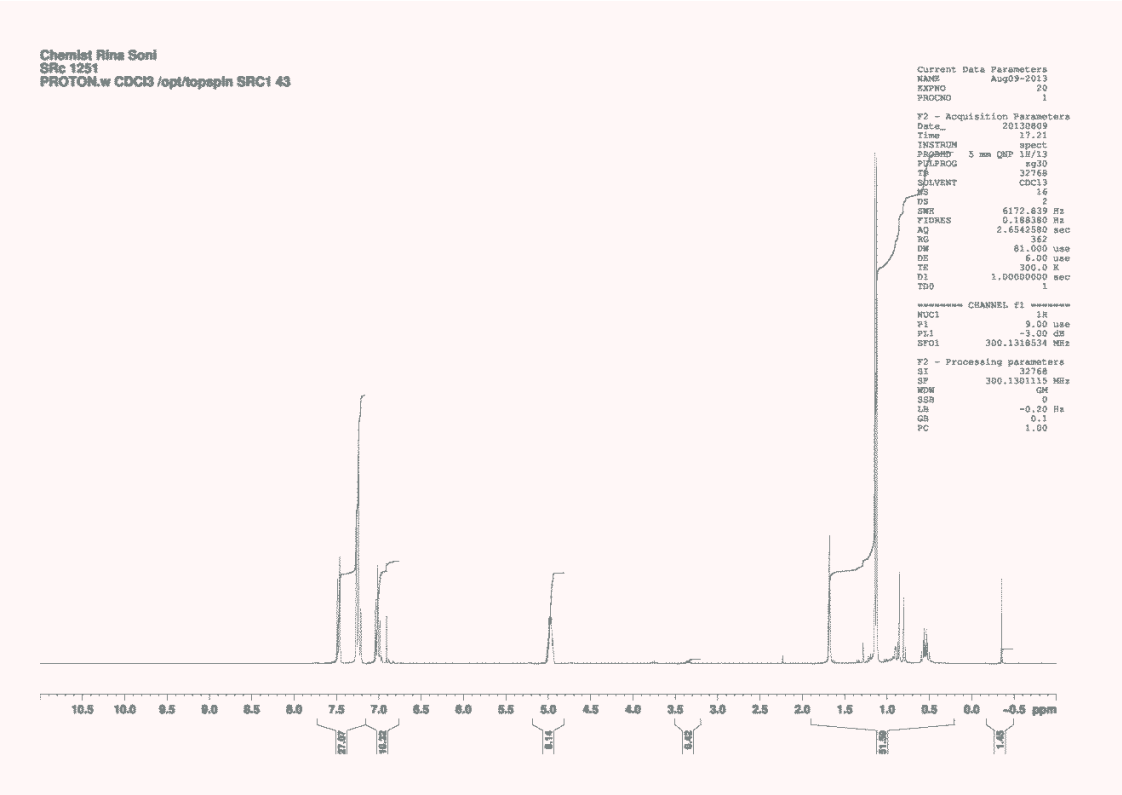



$^{13}$C, 75 MHz, $CDCl_3$

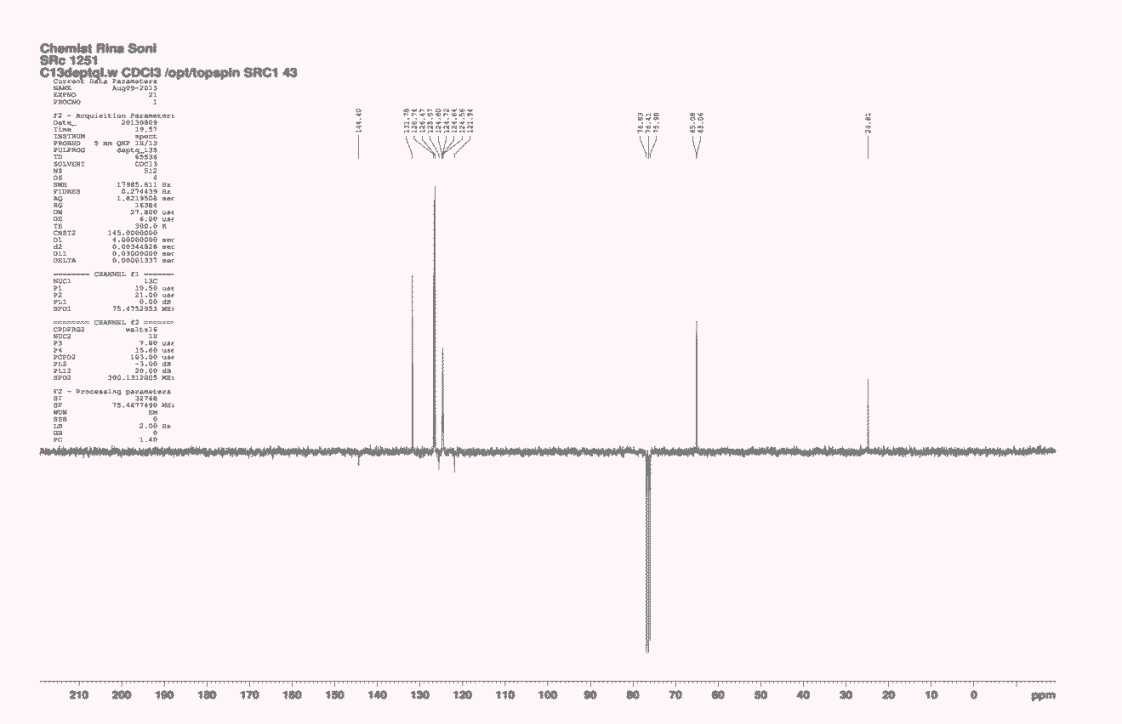


**(*R*)-1-(2-Trifluoromethylphenyl)ethanol**. *Zero - 0.3.*

$^{1}$H, 300 MHz, $CDCl_3$

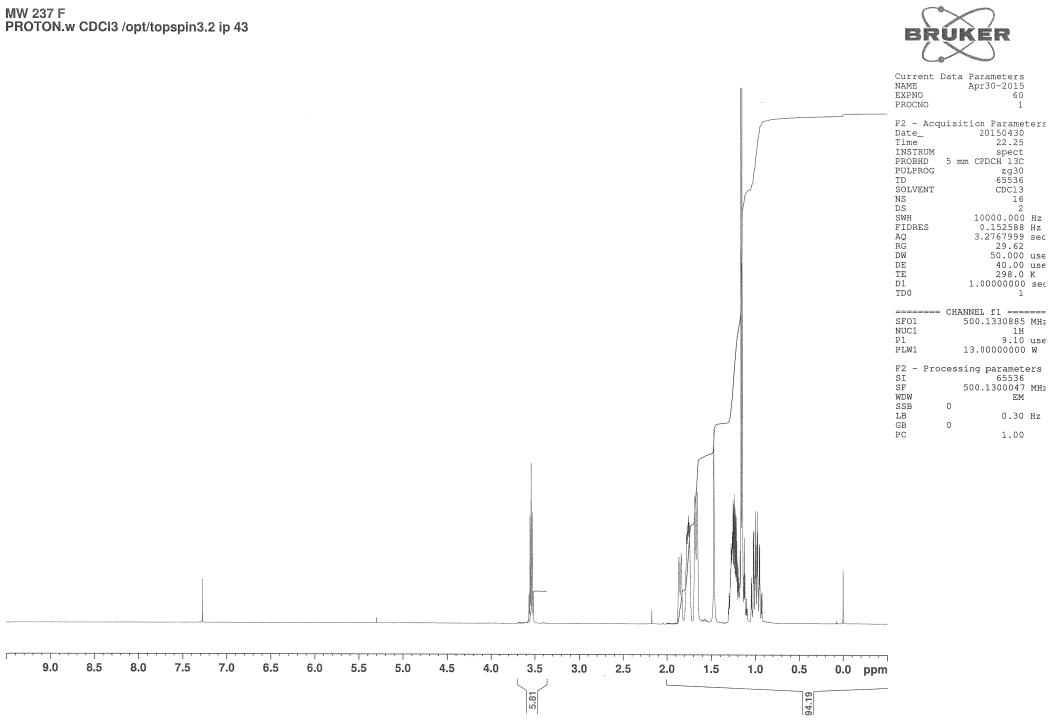



$^{13}$C, 75 MHz, $CDCl_3$

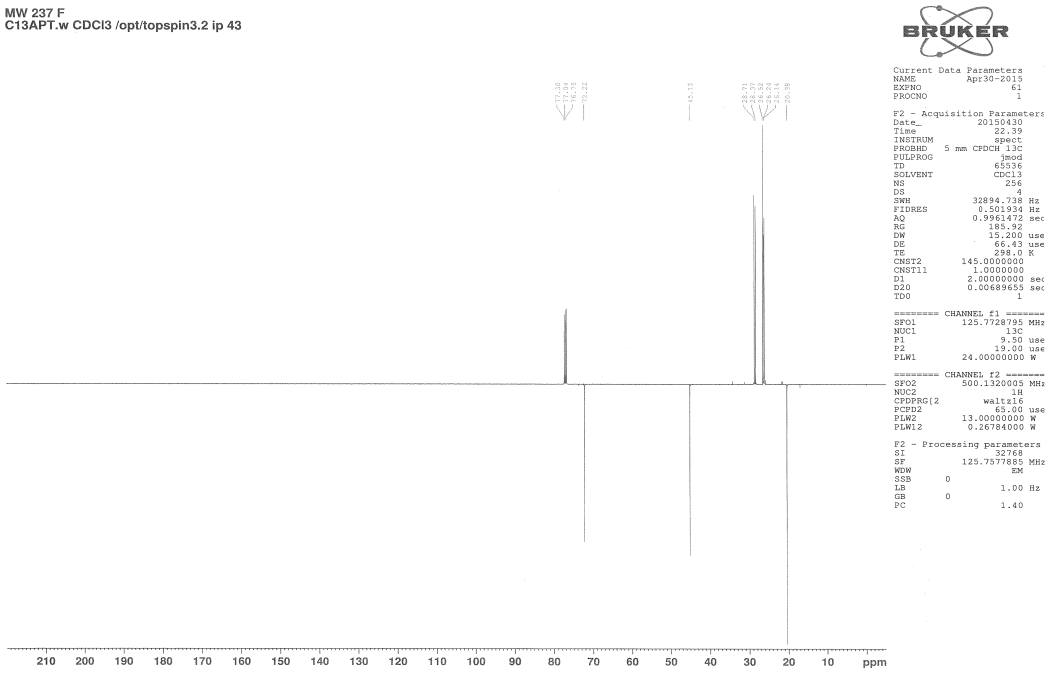


**(*S*)-1-Cyclohexylethanol.**

$^{1}$H, 500 MHz, $CDCl_3$

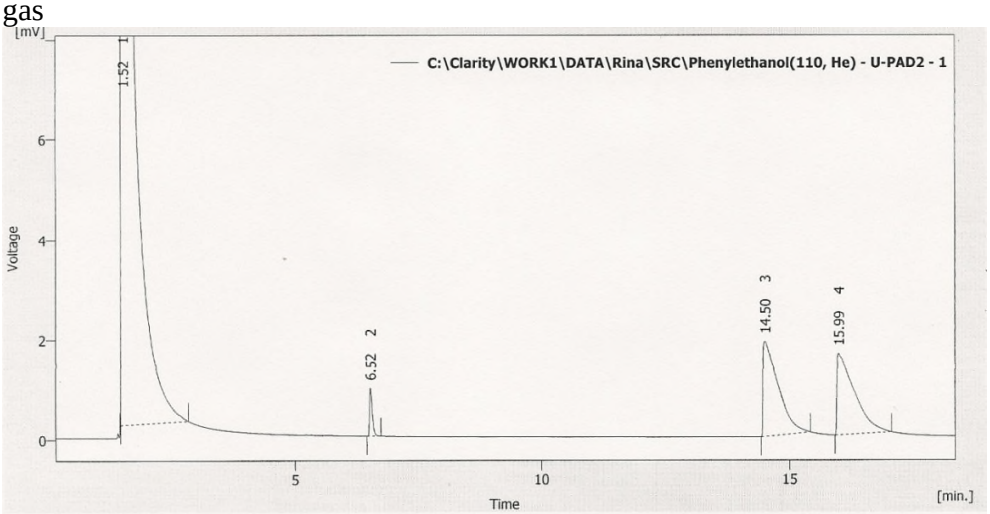



$^{13}$C, 125 MHz, $CDCl_3$



**Chiral GC/HPLC data**

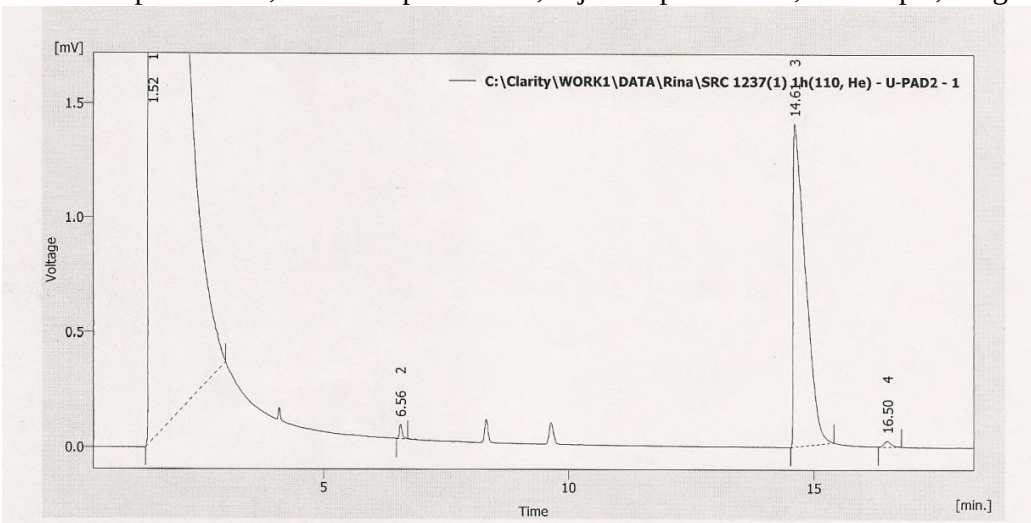
Racemic 1-Phenylethanol: GC analysis on CP-Chirasil-DEX CB 25m x 0.25mm x 0.25µm, Oven Temp = 110 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\Phenylethanol(110, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.516 | 5636.443 | 1093.867 | 98.5 | 99.6 | 0.08 | |
| 2 | 6.524 | 3.778 | 0.965 | 0.1 | 0.1 | 0.06 | |
| 3 | 14.500 | 40.444 | 1.901 | 0.7 | 0.2 | 0.34 | |
| 4 | 15.988 | 40.269 | 1.628 | 0.7 | 0.1 | 0.39 | |
| | Total | 5720.935 | 1098.363 | 100.0 | 100.0 | | |

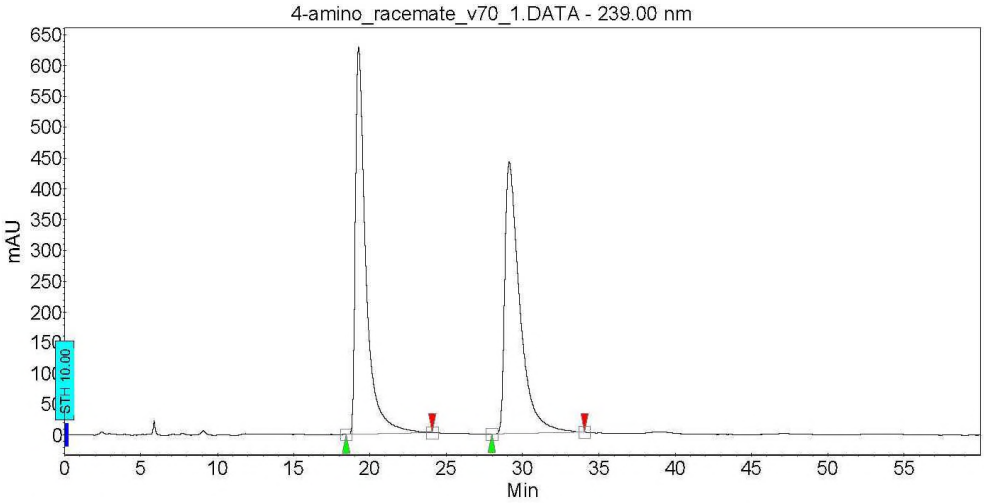
ATH of acetophenone: GC analysis on CP-Chirasil-DEX CB 25m x 0.25mm x 0.25µm, Oven Temp = 110 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1237(1) 1h(110, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.516 | 8694.583 | 1094.295 | 99.7 | 99.9 | 0.13 | |
| 2 | 6.556 | 0.252 | 0.062 | 0.0 | 0.0 | 0.06 | |
| 3 | 14.608 | 24.319 | 1.410 | 0.3 | 0.1 | 0.28 | |
| 4 | 16.504 | 0.261 | 0.025 | 0.0 | 0.0 | 0.16 | |
| | Total | 8719.414 | 1095.792 | 100.0 | 100.0 | | |

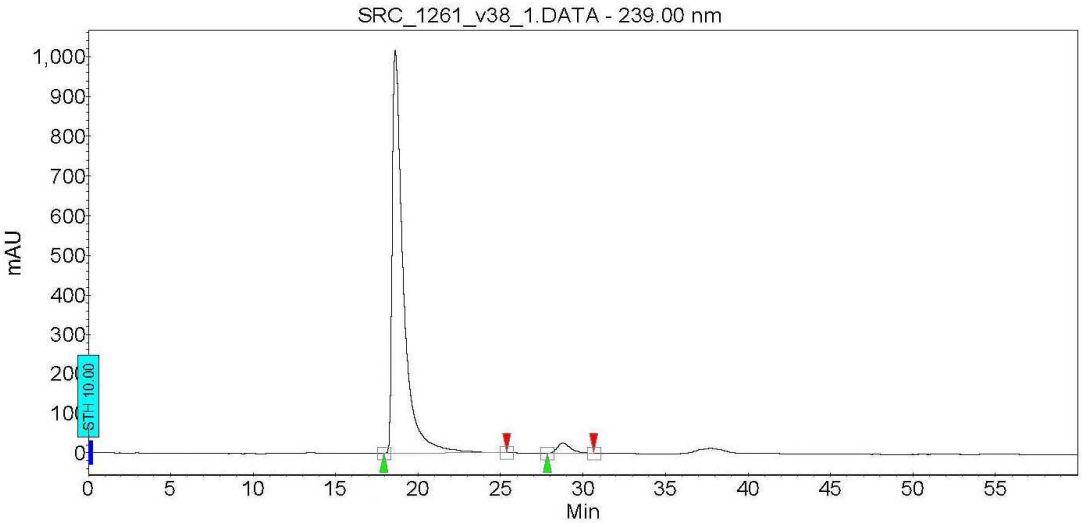
Racemic 1-(4'-aminophenyl)ethanol: HPLC analysis on ChiralPak OD-H Column: 0.46 cm x 25 cm, Hexane:IPA 80:20, 1.0 mL/min, 239 nM, 30$^{o}$C




**Peak results :**

| Index | Name | Time [Min] | Quantity [% Area] | Height [mAU] | Area [mAU.Min] | Area % [%] |
|---|---|---|---|---|---|---|
| 1 | UNKNOWN | 19.25 | 49.96 | 628.4 | 501.2 | 49.959 |
| 2 | UNKNOWN | 29.12 | 50.04 | 441.2 | 502.0 | 50.041 |
| | | | | | | |
| Total | | | 100.00 | 1069.6 | 1003.2 | 100.000 |

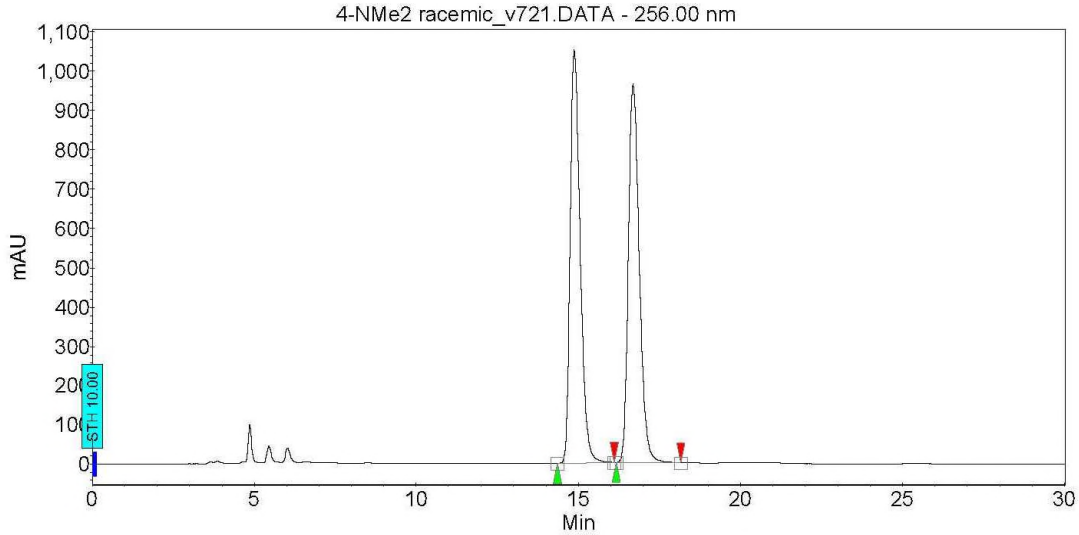
ATH of 4'-aminoacetophenone using RR-4-methoxy catalyst: HPLC analysis on ChiralPak IB Column: 0.46 cm x 25 cm, Hexane:IPA 80:20, 1.0 mL/min, 239 nM, 30$^{o}$C




**Peak results :**

| Index | Name | Time [Min] | Quantity [% Area] | Height [mAU] | Area [mAU.Min] | Area % [%] |
|---|---|---|---|---|---|---|
| 1 | UNKNOWN | 18.61 | 96.94 | 1017.2 | 803.7 | 96.937 |
| 2 | UNKNOWN | 28.76 | 3.06 | 26.6 | 25.4 | 3.063 |
| | | | | | | |
| Total | | | 100.00 | 1043.8 | 829.1 | 100.000 |

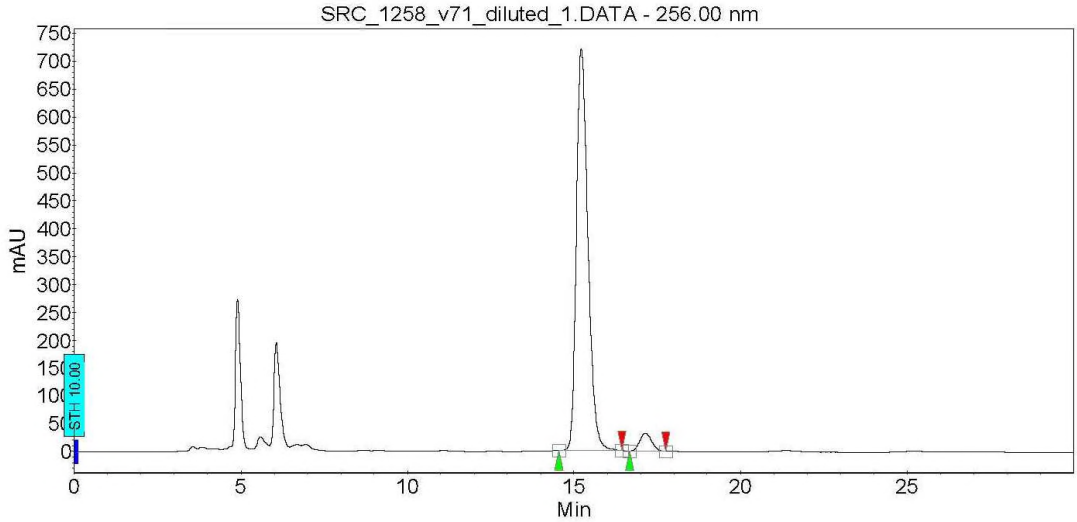
Racemic 1-[(4'-dimethylamino)phenyl])ethanol: HPLC analysis on ChiralPak OD-H Column: 0.46 cm x 25 cm, Hexane:IPA 95:5, 1.0 mL/min, 256 nM, 30$^{o}$C




**Peak results :**

| Index | Name | Time [Min] | Quantity [% Area] | Height [mAU] | Area [mAU.Min] | Area % [%] |
|---|---|---|---|---|---|---|
| 1 | UNKNOWN | 14.87 | 49.91 | 1052.6 | 376.5 | 49.914 |
| 2 | UNKNOWN | 16.68 | 50.09 | 965.0 | 377.8 | 50.086 |
| | | | | | | |
| Total | | | 100.00 | 2017.6 | 754.3 | 100.000 |

ATH of 4'-(dimethylamino)acetophenone: HPLC analysis on ChiralPak OD-H Column: 0.46 cm x 25 cm, Hexane:IPA 95:5, 1.0 mL/min, 256 nM, 30$^{o}$C

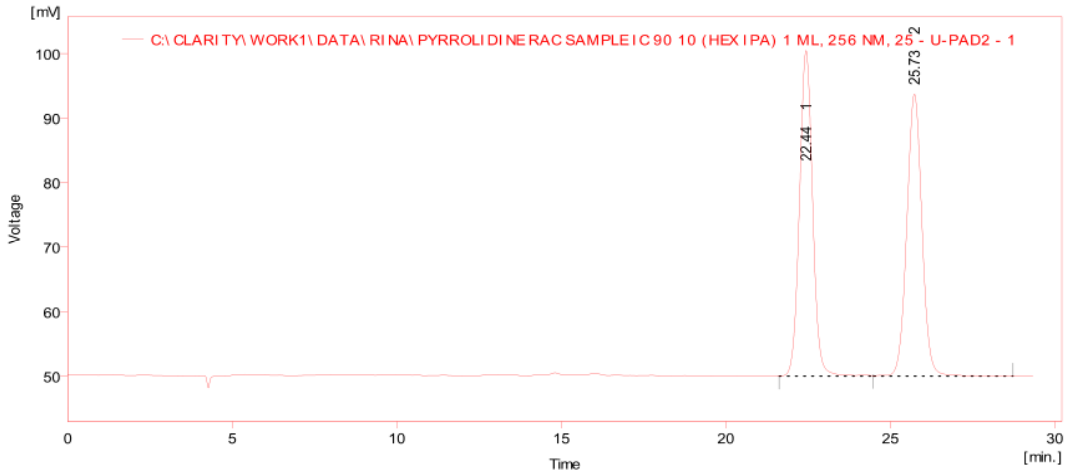



**Peak results :**

| Index | Name | Time [Min] | Quantity [% Area] | Height [mAU] | Area [mAU.Min] | Area % [%] |
|---|---|---|---|---|---|---|
| 1 | UNKNOWN | 15.21 | 95.55 | 720.0 | 287.5 | 95.554 |
| 2 | UNKNOWN | 17.15 | 4.45 | 32.3 | 13.4 | 4.446 |
| | | | | | | |
| Total | | | 100.00 | 752.4 | 300.9 | 100.000 |

**1-[4-(Pyrrolidin-1-yl)phenyl]ethanol** HPLC analysis (CHIRALPAK IC, 25cm x 4.6mm column, iPrOH:hexane 1:9, 1.0 mL/min, T = 25°C, λ = 256 nm).

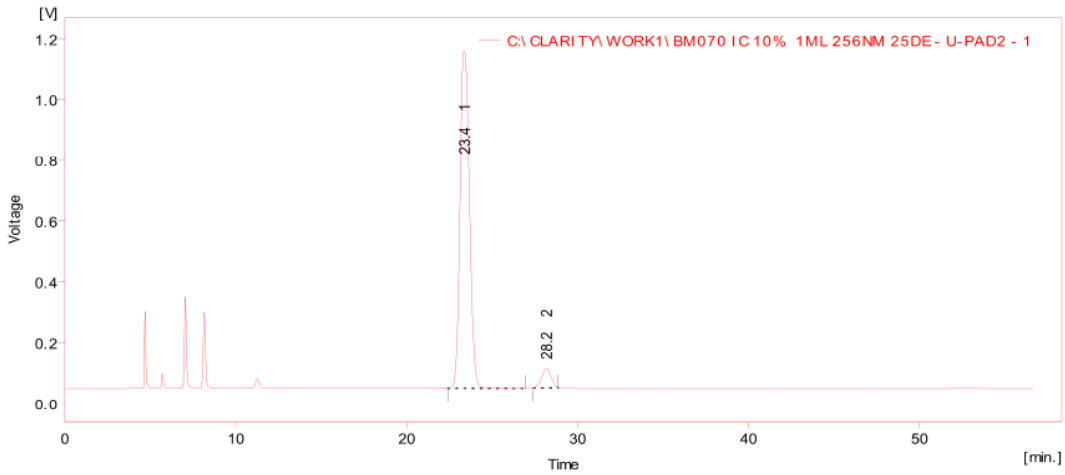
Racemic




*Result Table (Uncal - C:\CLARITY\WORK1\DATA\RINA\PYRROLIDINE RAC SAMPLE IC 90 10 (HEX IPA) 1 ML, 256 NM, 25 - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 22.440 | 1403.906 | 50.452 | 50.0 | 53.6 | 0.43 | |
| 2 | 25.732 | 1406.037 | 43.694 | 50.0 | 46.4 | 0.49 | |
| | Total | 2809.944 | 94.146 | 100.0 | 100.0 | | |

*S*- Enriched

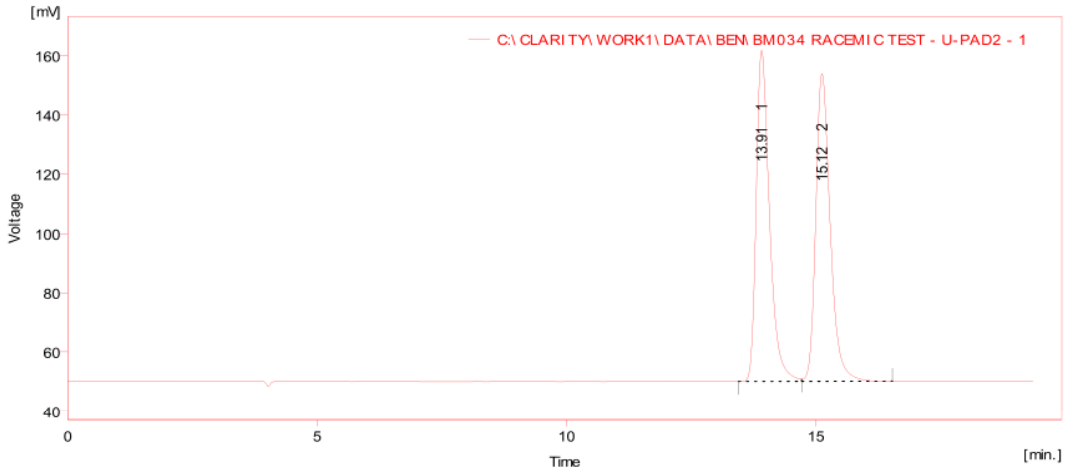



*Result Table (Uncal - C:\CLARITY\WORK1\BM070 IC 10% 1ML 256NM 25DE - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 23.392 | 42351.543 | 1107.623 | 94.6 | 94.6 | 0.60 | |
| 2 | 28.184 | 2427.821 | 63.279 | 5.4 | 5.4 | 0.61 | |
| | Total | 44779.364 | 1170.902 | 100.0 | 100.0 | | |

**1-[4-(Piperidin-1-yl)phenyl]ethanol** HPLC analysis (CHIRALPAK IC, 25cm x 4.6mm column, iPrOH:hexane 1:9, 1.0 mL/min, T = 25°C, λ = 256 nm).

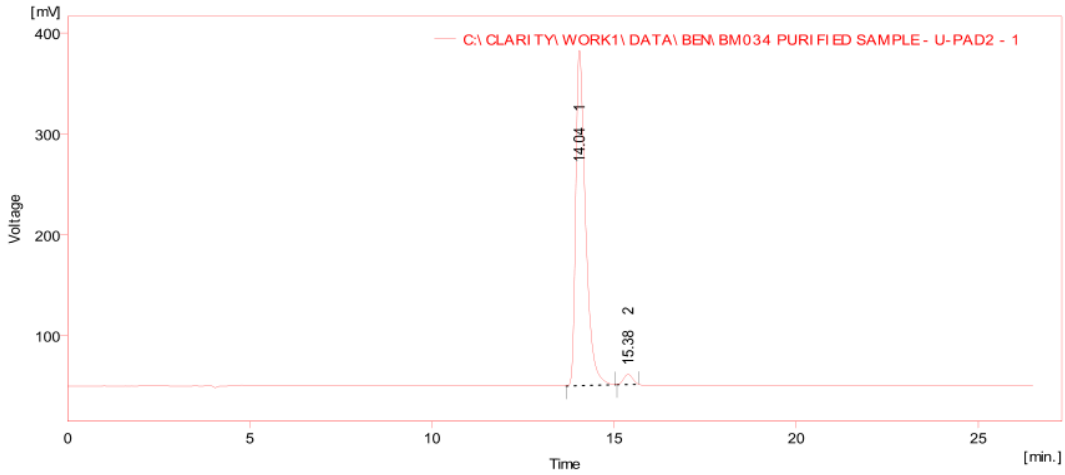
Racemic




*Result Table (Uncal - C:\CLARITY\WORK1\DATA\BEN\BM034 RACEMIC TEST - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 13.912 | 2068.974 | 111.661 | 49.8 | 51.8 | 0.28 | |
| 2 | 15.120 | 2084.738 | 103.881 | 50.2 | 48.2 | 0.30 | |
| | Total | 4153.712 | 215.542 | 100.0 | 100.0 | | |

*S*- Enriched

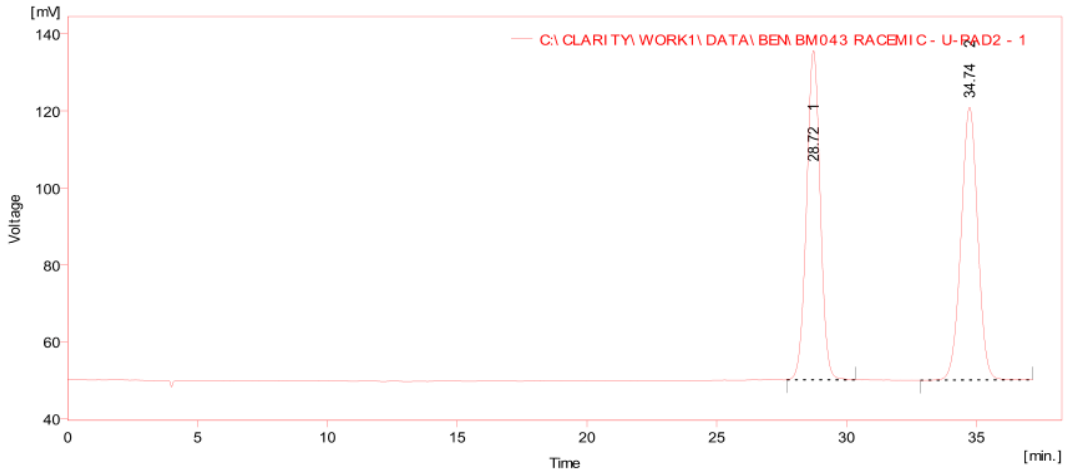



*Result Table (Uncal - C:\CLARITY\WORK1\DATA\BEN\BM034 PURIFIED SAMPLE - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 14.044 | 6225.286 | 332.182 | 97.3 | 97.0 | 0.28 | |
| 2 | 15.384 | 173.839 | 10.116 | 2.7 | 3.0 | 0.28 | |
| | Total | 6399.125 | 342.297 | 100.0 | 100.0 | | |

**1-[4-(Morpholin-4-yl)phenyl]ethanol** HPLC analysis (CHIRALPAK IC, 25cm x 4.6mm column, iPrOH:hexane 1:9, 1.0 mL/min, T = 25°C, λ = 256 nm, *S* isomer 29.2 min, *R* isomer 35.4 min).

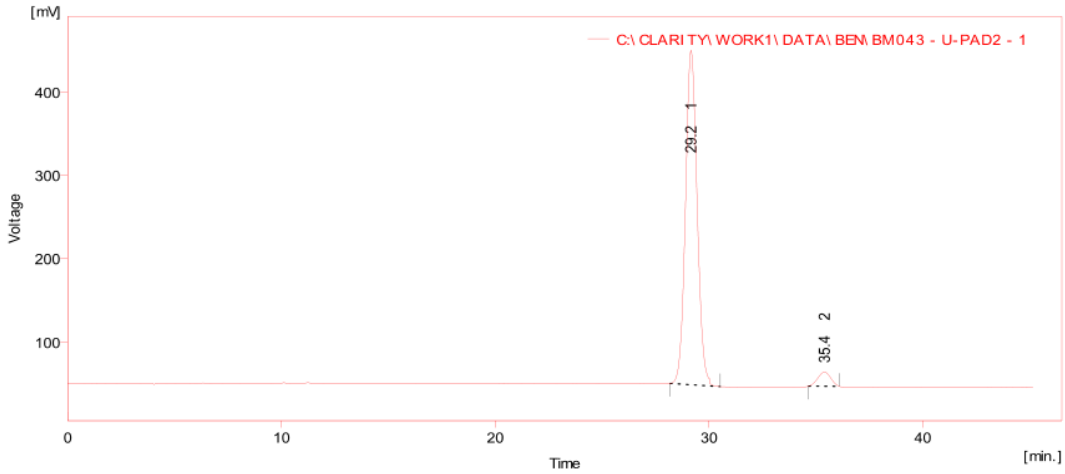
Racemic




*Result Table (Uncal - C:\CLARITY\WORK1\DATA\BEN\BM043 RACEMIC - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 28.724 | 3135.670 | 85.580 | 49.7 | 54.7 | 0.57 | |
| 2 | 34.740 | 3168.634 | 70.894 | 50.3 | 45.3 | 0.69 | |
| | Total | 6304.304 | 156.474 | 100.0 | 100.0 | | |

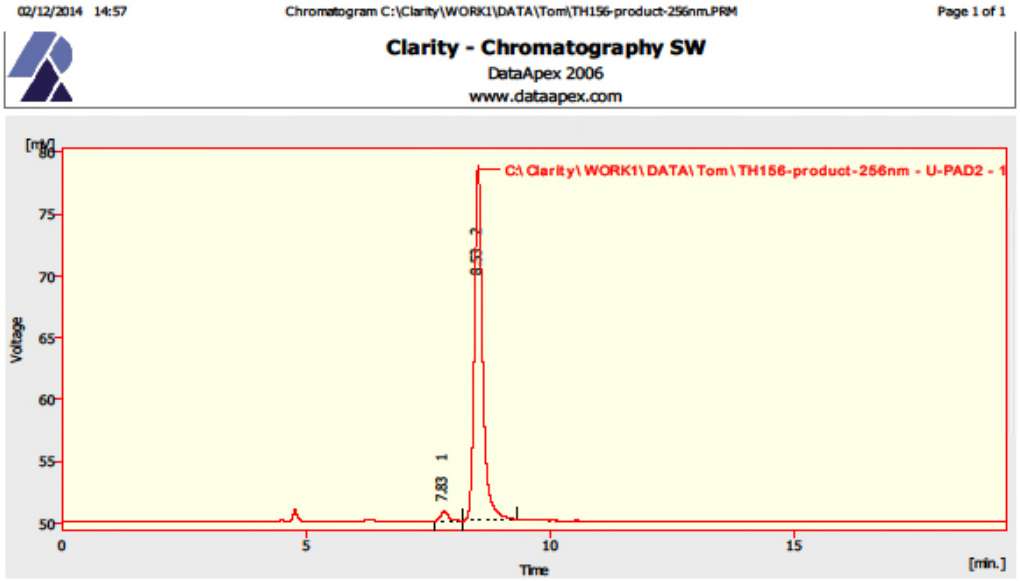
*S*- Enriched




*Result Table (Uncal - C:\CLARITY\WORK1\DATA\BEN\BM043 - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 29.156 | 15149.215 | 401.355 | 95.5 | 96.0 | 0.58 | |
| 2 | 35.400 | 712.070 | 16.902 | 4.5 | 4.0 | 0.69 | |
| | Total | 15861.285 | 418.257 | 100.0 | 100.0 | | |

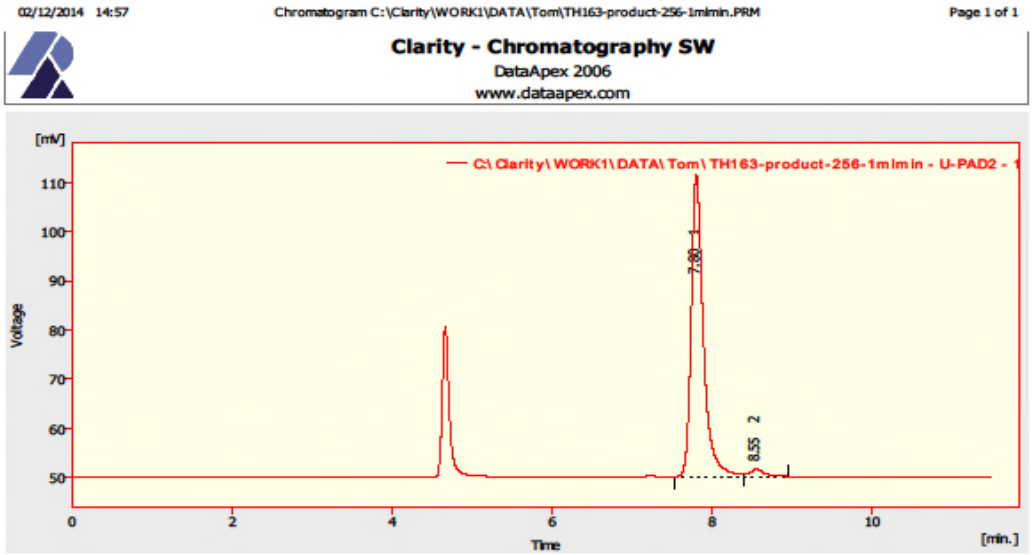
**(*R*)-2-(1-Hydroxyethyl)-4-methylphenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C .




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH156-product-256nm - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.828 | 9.076 | 0.857 | 2.6 | 2.9 | 0.16 | |
| 2 | 8.528 | 335.101 | 28.711 | 97.4 | 97.1 | 0.17 | |
| | Total | 344.177 | 29.569 | 100.0 | 100.0 | | |

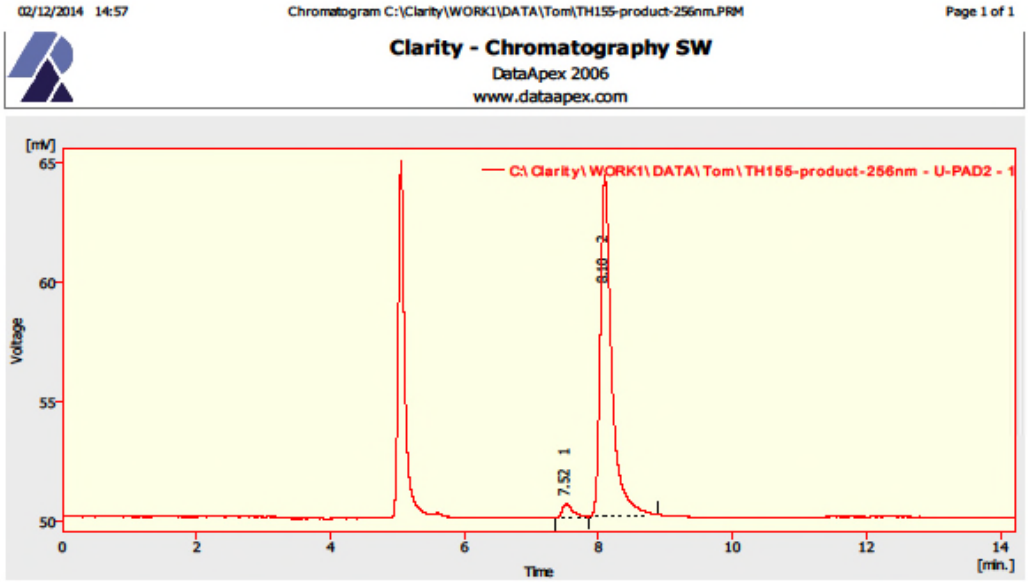
**(*S*)-2-(1-Hydroxyethyl)-4-methylphenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH163-product-256-1mlmin - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.800 | 648.784 | 61.550 | 97.2 | 97.7 | 0.15 | |
| 2 | 8.552 | 18.657 | 1.439 | 2.8 | 2.3 | 0.19 | |
| | Total | 667.441 | 62.989 | 100.0 | 100.0 | | |

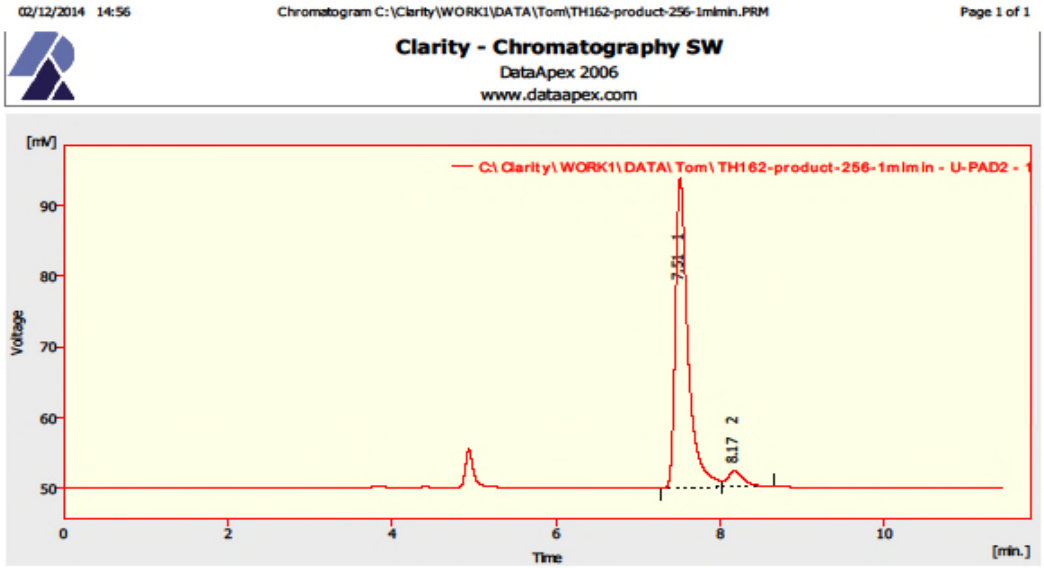
**(*R*)-4-Chloro-2-(1-hydroxyethyl)phenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH155-product-256nm - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.520 | 6.387 | 0.584 | 3.7 | 3.9 | 0.16 | |
| 2 | 8.096 | 168.341 | 14.281 | 96.3 | 96.1 | 0.16 | |
| | Total | 174.728 | 14.865 | 100.0 | 100.0 | | |

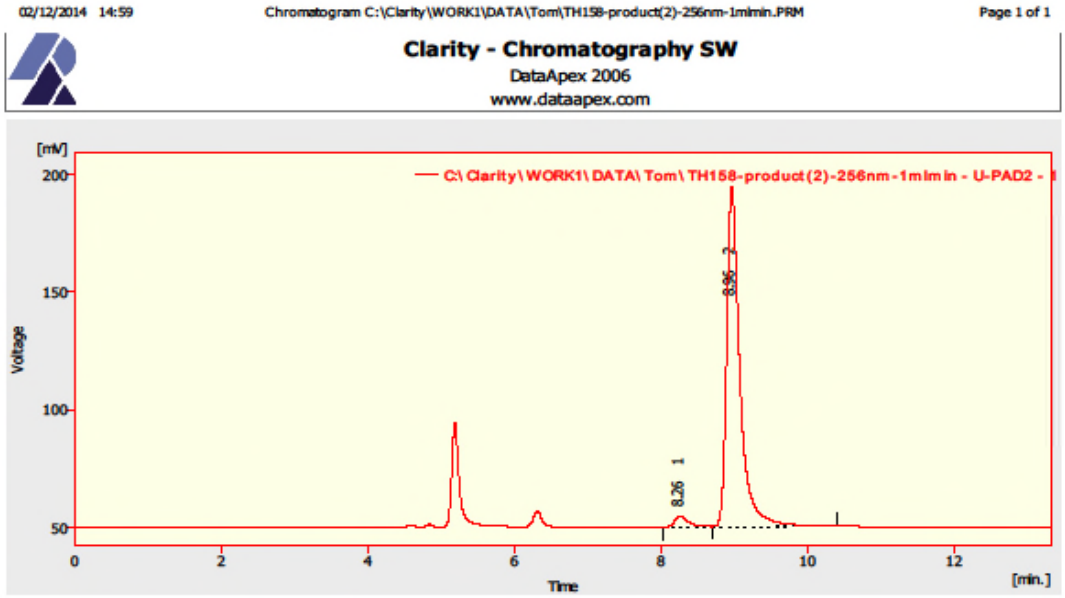
**(*S*)-4-Chloro-2-(1-hydroxyethyl)phenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH162-product-256-1mlmin - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.512 | 469.849 | 43.965 | 93.9 | 95.2 | 0.15 | |
| 2 | 8.172 | 30.458 | 2.236 | 6.1 | 4.8 | 0.20 | |
| | Total | 500.306 | 46.202 | 100.0 | 100.0 | | |

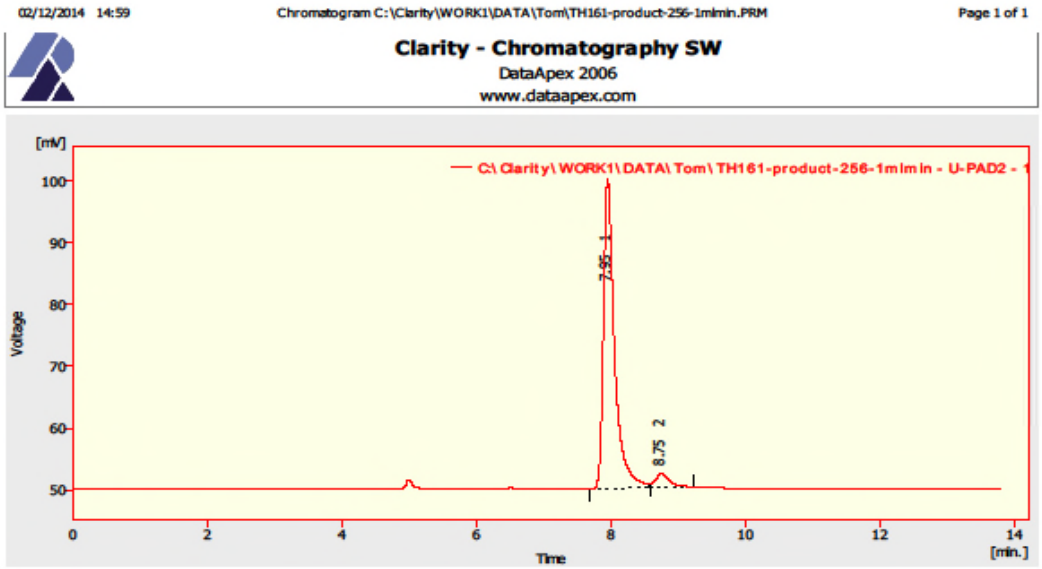
(***R***)**-4-Bromo-2-(1-hydroxyethyl)phenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH158-product(2)-256nm-1mlmin - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 8.256 | 59.026 | 4.632 | 3.2 | 3.1 | 0.18 | |
| 2 | 8.964 | 1809.477 | 143.962 | 96.8 | 96.9 | 0.18 | |
| | Total | 1868.503 | 148.594 | 100.0 | 100.0 | | |

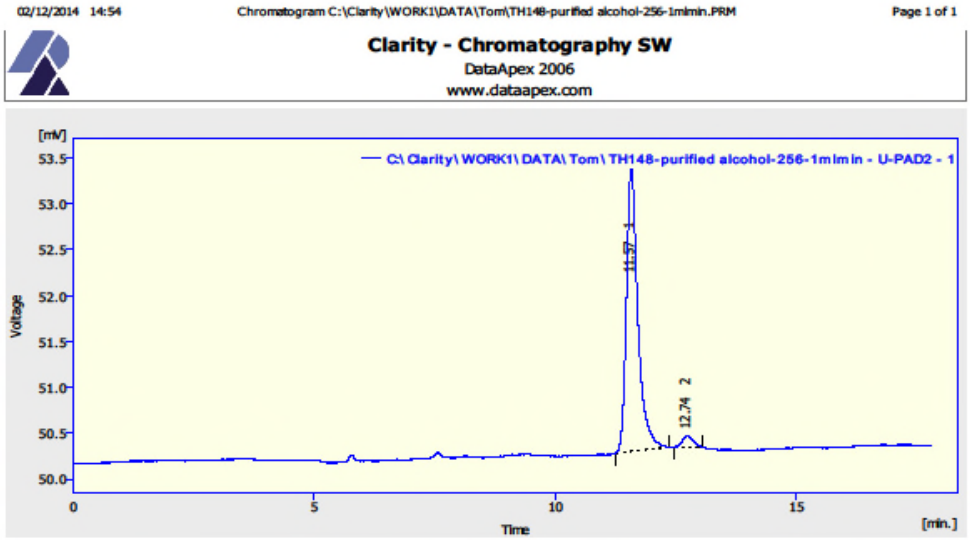
**(*S*)-4-Bromo-2-(1-hydroxyethyl)phenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH161-product-256-1mlmin - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.952 | 578.895 | 50.259 | 94.8 | 95.7 | 0.16 | |
| 2 | 8.752 | 32.064 | 2.267 | 5.2 | 4.3 | 0.21 | |
| | Total | 610.959 | 52.526 | 100.0 | 100.0 | | |

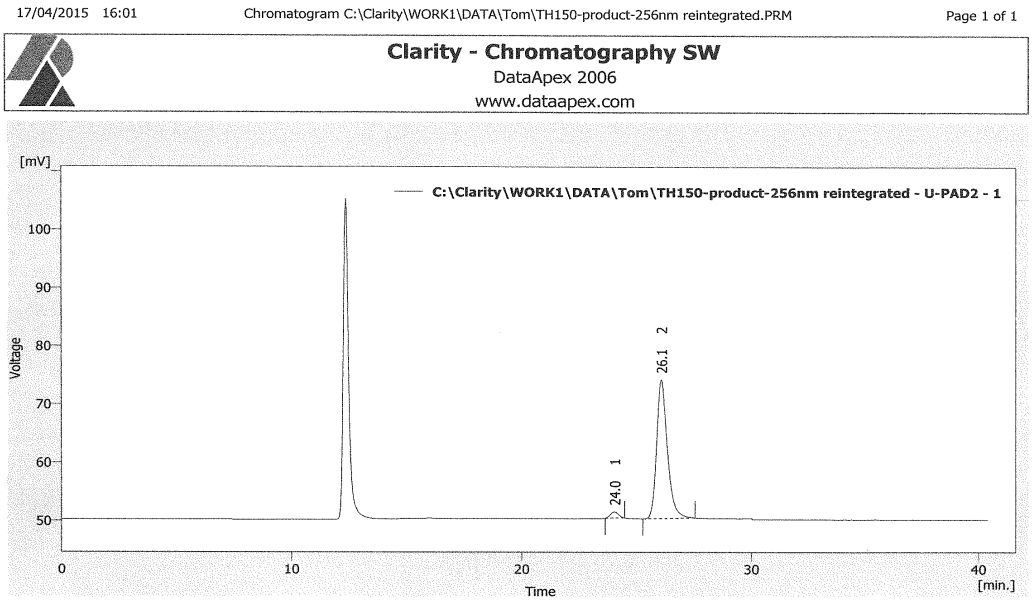
**(*S*)-2-(1-Hydroxyethyl)-4-methoxyphenol**. HPLC, IA Column, 1 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH148-purified alcohol-256-1mlmin - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 11.572 | 50.914 | 3.080 | 96.5 | 96.2 | 0.24 | |
| 2 | 12.740 | 1.837 | 0.120 | 3.5 | 3.8 | 0.25 | |
| | Total | 52.752 | 3.200 | 100.0 | 100.0 | | |

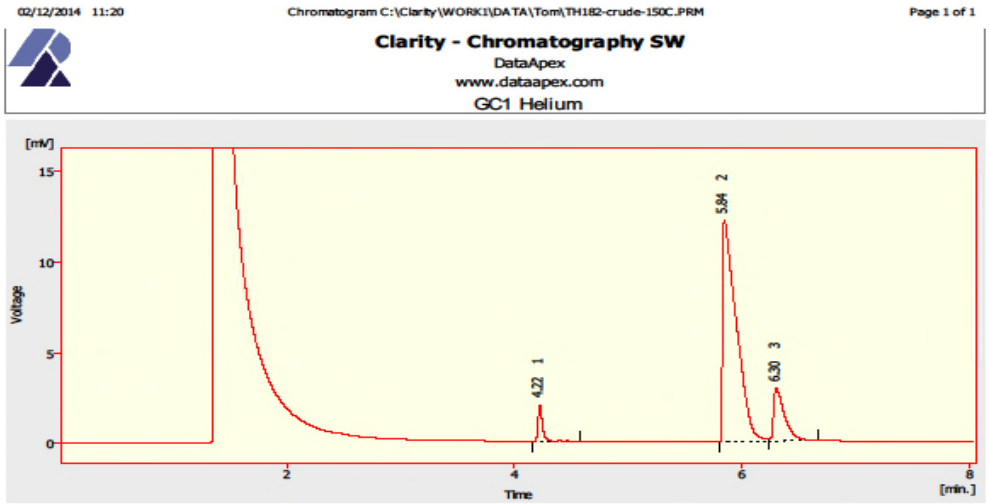
**(*R*)-2-(1-Hydroxyethyl)-4-methoxyphenol**. HPLC, IA Column, 0.5 mL/min, 10% IPA : 90% Hexane; 256 nm UV, 30 °C (note the longer retention times are due to a slower flow rate in this sample).




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH150-product-256nm reintegrated - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 24.032 | 26.112 | 1.059 | 3.3 | 4.3 | 0.40 | |
| 2 | 26.080 | 774.932 | 23.856 | 96.7 | 95.7 | 0.48 | |
| | Total | 801.044 | 24.915 | 100.0 | 100.0 | | |

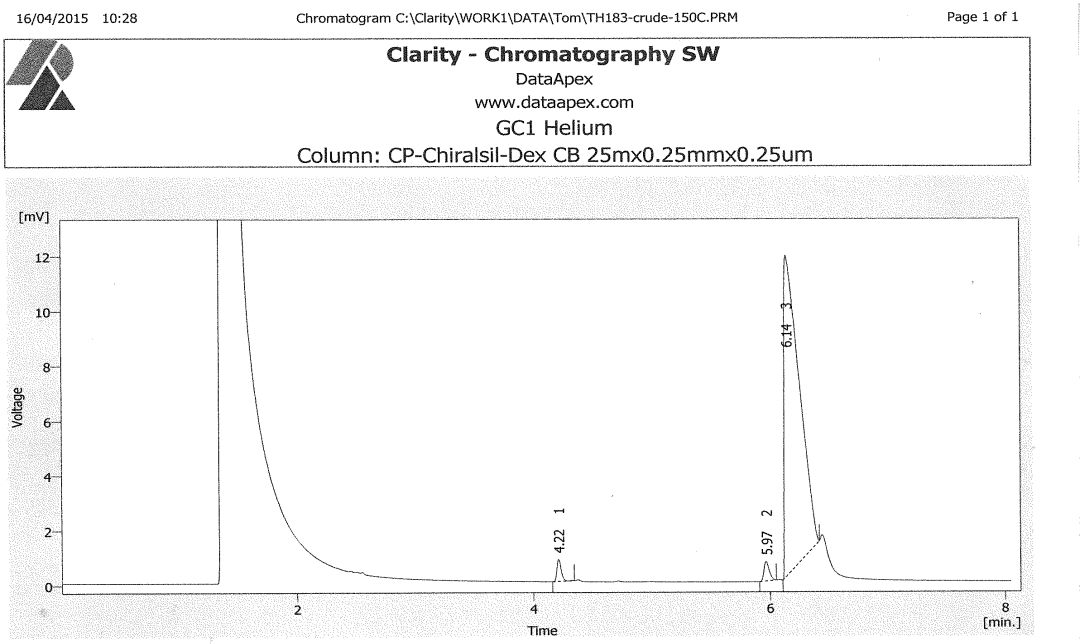
**(*S*)-1-(2-Methoxyphenyl)ethan-1-ol**. GC, CP-Chirasil-Dex-Cβ column, oven 150 ºC, inj.: split 220 ºC, det.: FID 250 ºC, 18 Psi He.68% ee (*S*) example.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH182-crude-150C - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 4.224 | 5.450 | 2.009 | 4.3 | 11.8 | 0.04 | |
| 2 | 5.944 | 102.695 | 12.187 | 80.4 | 71.4 | 0.14 | |
| 3 | 6.304 | 19.582 | 2.883 | 15.3 | 16.9 | 0.10 | |
| | Total | 127.727 | 17.079 | 100.0 | 100.0 | | |

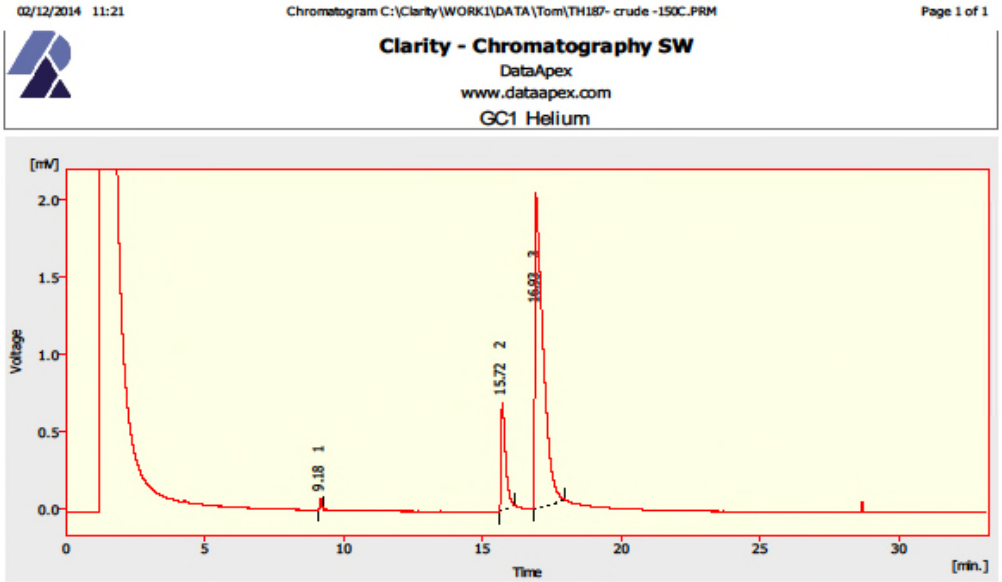
**(*R*)-1-(2-Methoxyphenyl)ethan-1-ol**. GC, CP-Chirasil-Dex-Cβ column, oven 150 ºC, inj.: split 220 ºC, det.: FID 250 ºC, 18 Psi He .Integration underestimates ee due to shoulder on peak. 96% ee (*R)* example.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH183-crude-150C - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 4.216 | 2.023 | 0.799 | 1.9 | 6.1 | 0.04 | |
| 2 | 5.968 | 2.185 | 0.708 | 2.0 | 5.4 | 0.05 | |
| 3 | 6.144 | 103.303 | 11.693 | 96.1 | 88.6 | 0.16 | |
| | Total | 107.510 | 13.200 | 100.0 | 100.0 | | |

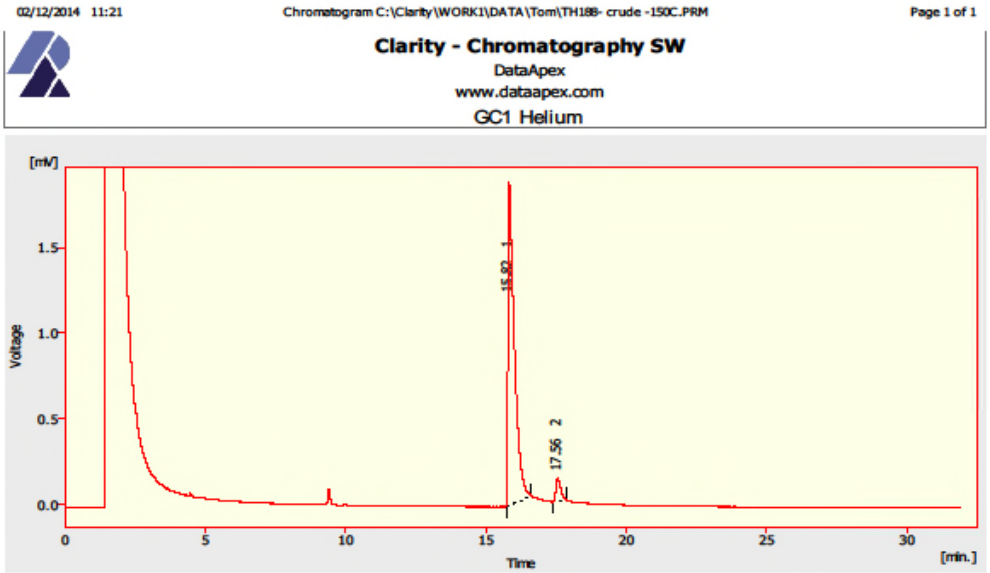
(***S***)**-1-(2,5-Dimethoxyphenyl)ethan-1-ol**. GC, CP-Chirasil-Dex-Cβ column, oven 150 ºC, inj.: split 220 ºC, det.: FID 250 ºC, 18 Psi He




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH187- crude -150C - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 9.176 | 0.273 | 0.064 | 0.5 | 2.3 | 0.07 | |
| 2 | 15.720 | 7.634 | 0.693 | 15.4 | 24.8 | 0.17 | |
| 3 | 16.932 | 41.689 | 2.034 | 84.1 | 72.9 | 0.32 | |
| | Total | 49.596 | 2.792 | 100.0 | 100.0 | | |

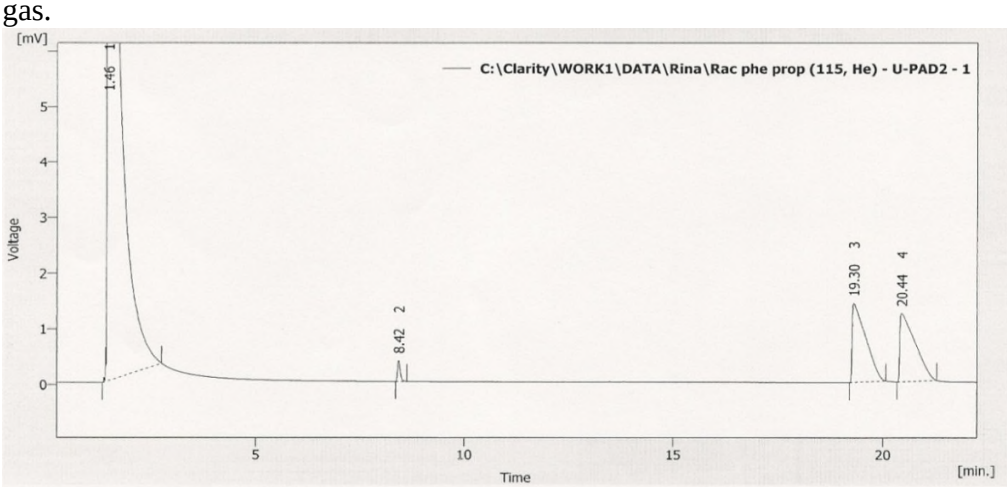
(***R***)**-1-(2,5-Dimethoxyphenyl)ethan-1-ol.** GC, CP-Chirasil-Dex-Cβ column, oven 150 ºC, inj.: split 220 ºC, det.: FID 250 ºC, 18 Psi He.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Tom\TH188- crude -150C - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 15.824 | 29.627 | 1.892 | 95.1 | 93.4 | 0.24 | |
| 2 | 17.556 | 1.540 | 0.134 | 4.9 | 6.6 | 0.19 | |
| | Total | 31.167 | 2.026 | 100.0 | 100.0 | | |

Racemic **phenyl propanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 115 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



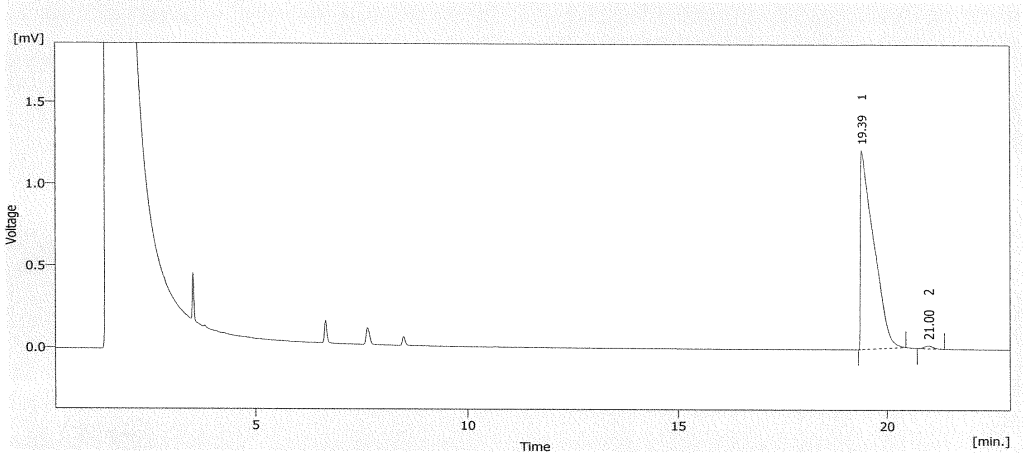

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\Rac phe prop (115, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.464 | 5298.929 | 1094.098 | 98.8 | 99.7 | 0.08 | |
| 2 | 8.424 | 1.723 | 0.373 | 0.0 | 0.0 | 0.08 | |
| 3 | 19.296 | 31.908 | 1.419 | 0.6 | 0.1 | 0.38 | |
| 4 | 20.444 | 31.605 | 1.229 | 0.6 | 0.1 | 0.43 | |
| | Total | 5364.165 | 1097.119 | 100.0 | 100.0 | | |

ATH of **propiophenone**: GC analysis CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 115 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:05 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1240 1.5h (115, He).PRM Page 1 of 1

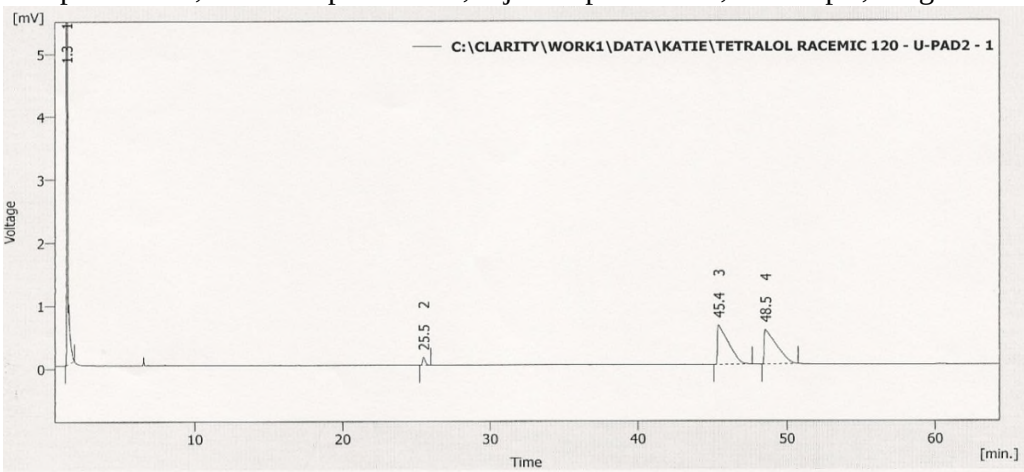
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1240 1.5h (115, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 19.392 | 28.196 | 1.217 | 99.1 | 98.5 | 0.38 | |
| 2 | 21.004 | 0.269 | 0.019 | 0.9 | 1.5 | 0.23 | |
| | Total | 28.464 | 1.235 | 100.0 | 100.0 | | |

Racemic **1-tetralol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 120 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



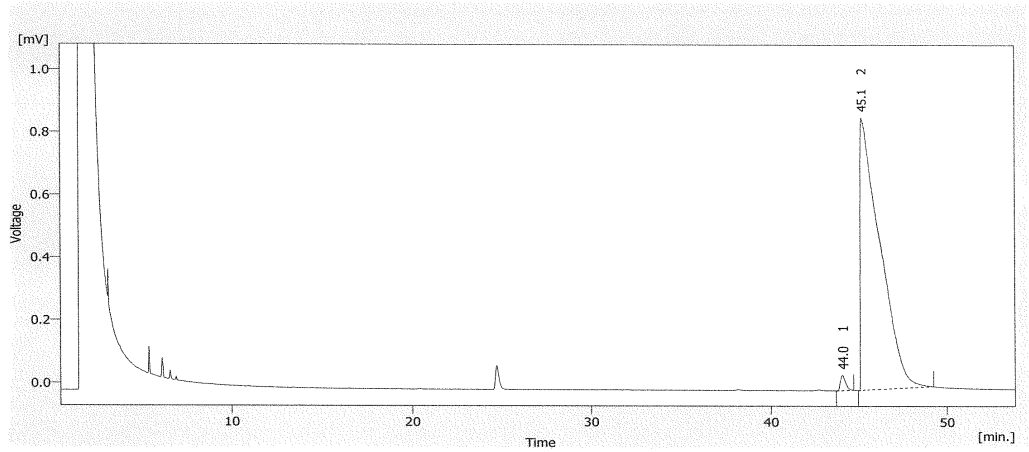

*Result Table (Uncal - C:\CLARITY\WORK1\DATA\KATIE\TETRALOL RACEMIC 120 - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.308 | 3032.760 | 986.249 | 97.9 | 99.9 | 0.06 | |
| 2 | 25.464 | 1.620 | 0.128 | 0.1 | 0.0 | 0.20 | |
| 3 | 45.412 | 32.022 | 0.624 | 1.0 | 0.1 | 0.83 | |
| 4 | 48.544 | 31.534 | 0.553 | 1.0 | 0.1 | 0.92 | |
| | Total | 3097.936 | 987.554 | 100.0 | 100.0 | | |

ATH of 1-**tetralone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 120 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:08 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1241 7h (120, He).PRM Page 1 of 1

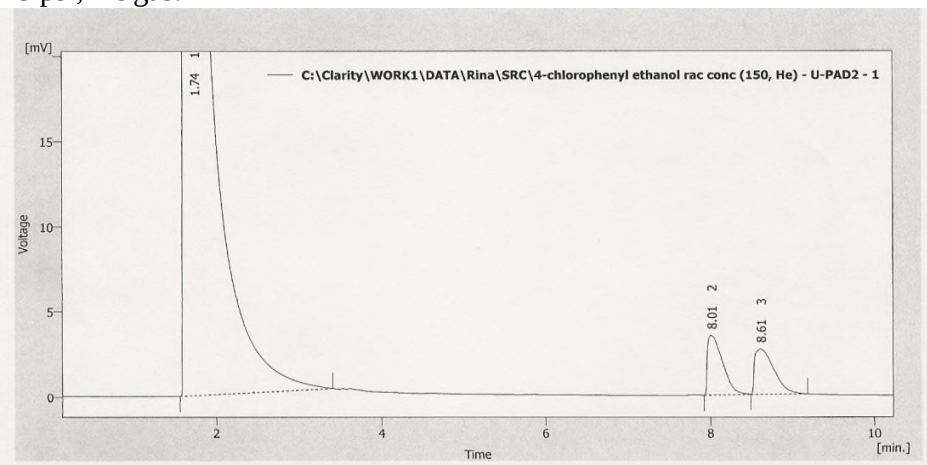
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1241 7h (120, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 43.988 | 1.013 | 0.049 | 1.4 | 5.3 | 0.32 | |
| 2 | 45.124 | 70.903 | 0.870 | 98.6 | 94.7 | 1.30 | |
| | Total | 71.915 | 0.919 | 100.0 | 100.0 | | |

Racemic **1-(4'-chlorophenyl)ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



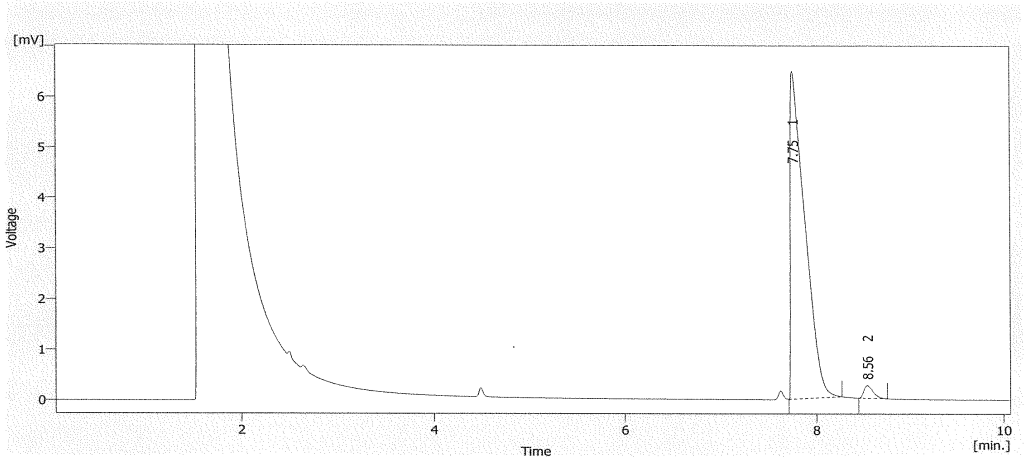

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\4-chlorophenyl ethanol rac conc (150, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.740 | 13402.188 | 1094.190 | 99.3 | 99.4 | 0.20 | |
| 2 | 8.012 | 44.675 | 3.497 | 0.3 | 0.3 | 0.21 | |
| 3 | 8.612 | 43.602 | 2.675 | 0.3 | 0.2 | 0.27 | |
| | Total | 13490.464 | 1100.362 | 100.0 | 100.0 | | |

ATH of **4'-chloroacetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:10 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1243 2.0h (150, He).PRM Page 1 of 1

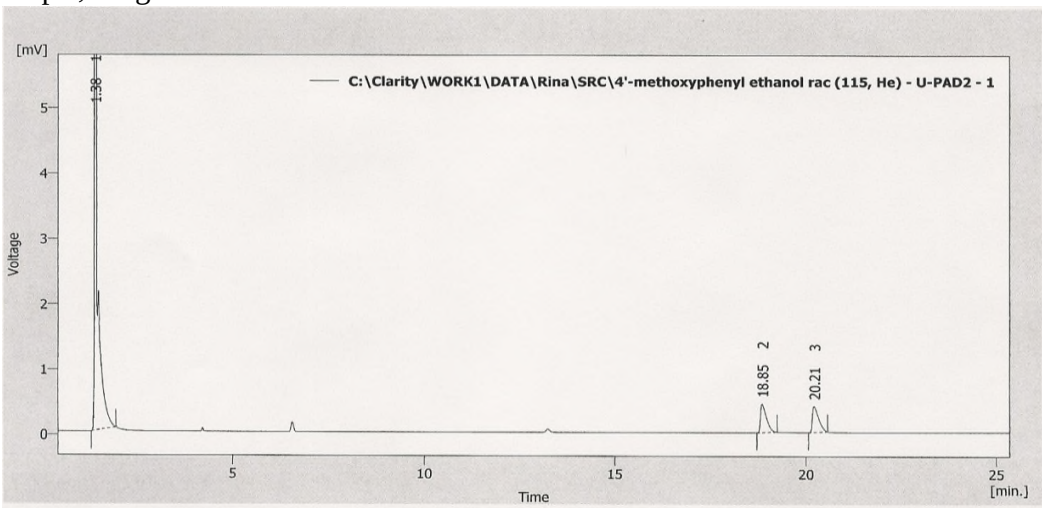
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1243 2.0h (150, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 7.752 | 68.191 | 6.494 | 97.6 | 96.3 | 0.17 | |
| 2 | 8.556 | 1.700 | 0.251 | 2.4 | 3.7 | 0.11 | |
| | Total | 69.891 | 6.745 | 100.0 | 100.0 | | |

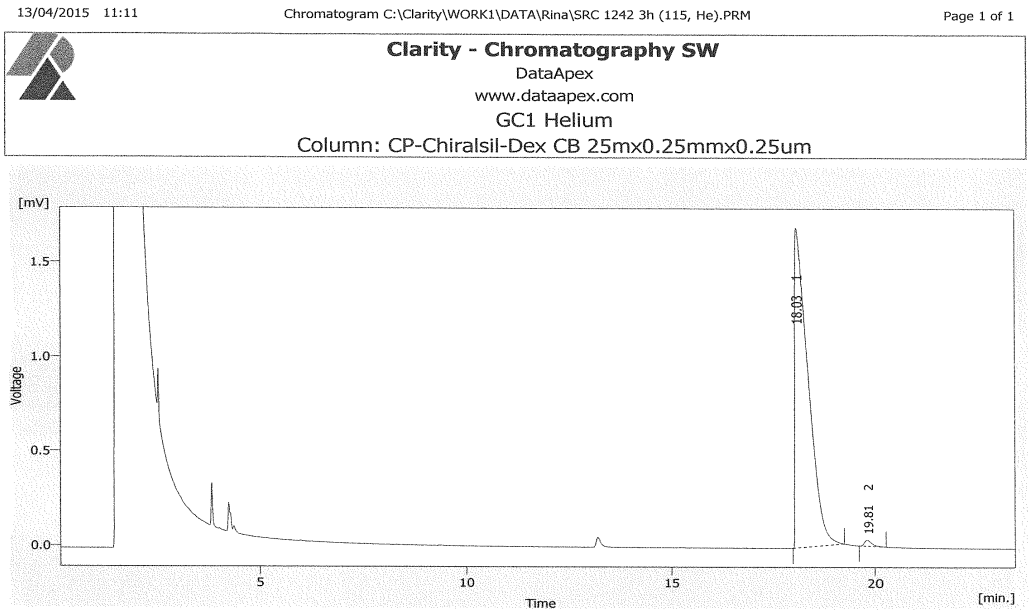
Racemic **1-(4'-methoxyphenyl)ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 115 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\4'-methoxyphenyl ethanol rac (115, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.380 | 2541.340 | 986.356 | 99.6 | 99.9 | 0.04 | |
| 2 | 18.848 | 4.725 | 0.440 | 0.2 | 0.0 | 0.17 | |
| 3 | 20.208 | 4.622 | 0.401 | 0.2 | 0.0 | 0.19 | |
| | Total | 2550.687 | 987.197 | 100.0 | 100.0 | | |

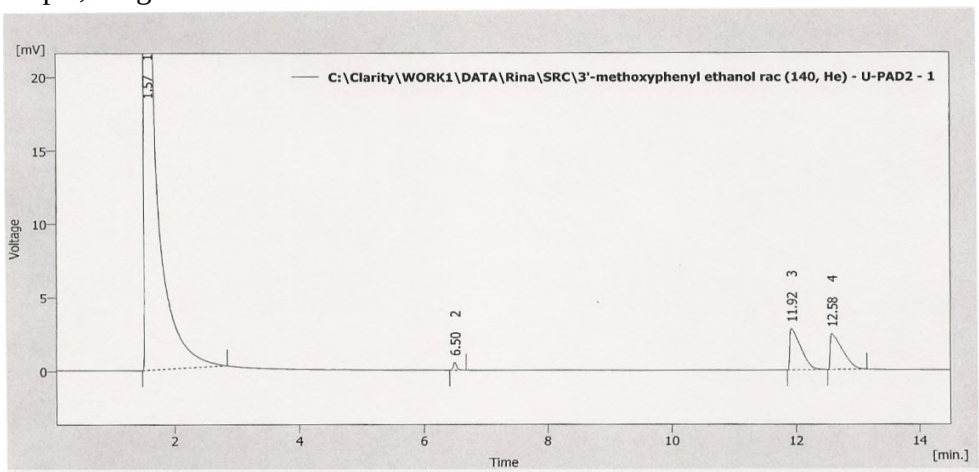
AT H of **4'-methoxyacetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 115 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1242 3h (115, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 18.032 | 38.367 | 1.697 | 98.8 | 98.1 | 0.36 | |
| 2 | 19.812 | 0.481 | 0.033 | 1.2 | 1.9 | 0.23 | |
| | Total | 38.848 | 1.730 | 100.0 | 100.0 | | |

Racemic **1-(3'-methoxyphenyl)ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 140 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



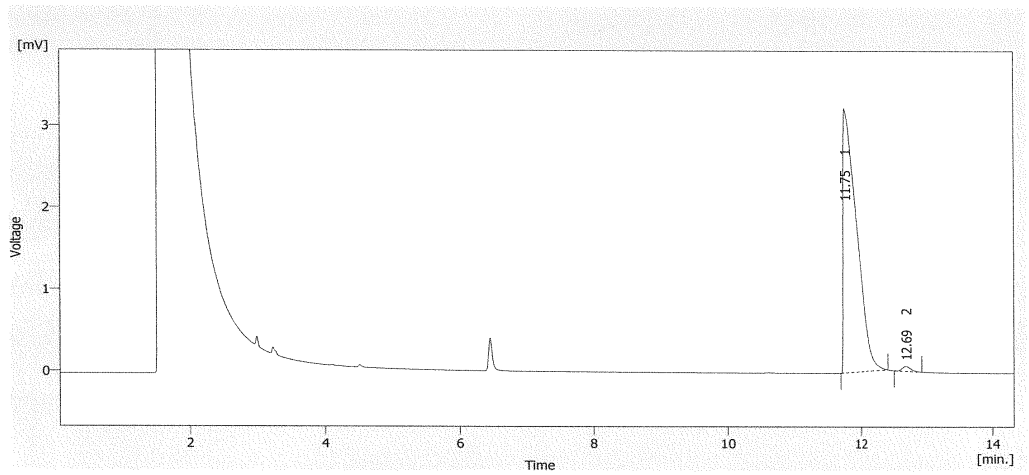

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\3'-methoxyphenyl ethanol rac (140, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.568 | 4891.936 | 1094.105 | 98.6 | 99.5 | 0.07 | |
| 2 | 6.500 | 1.948 | 0.540 | 0.0 | 0.0 | 0.06 | |
| 3 | 11.924 | 34.583 | 2.808 | 0.7 | 0.3 | 0.20 | |
| 4 | 12.580 | 33.715 | 2.442 | 0.7 | 0.2 | 0.23 | |
| | Total | 4962.182 | 1099.895 | 100.0 | 100.0 | | |

ATH of **3'-methoxyacetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 140 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:13 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1245 2h (140, He).PRM Page 1 of 1

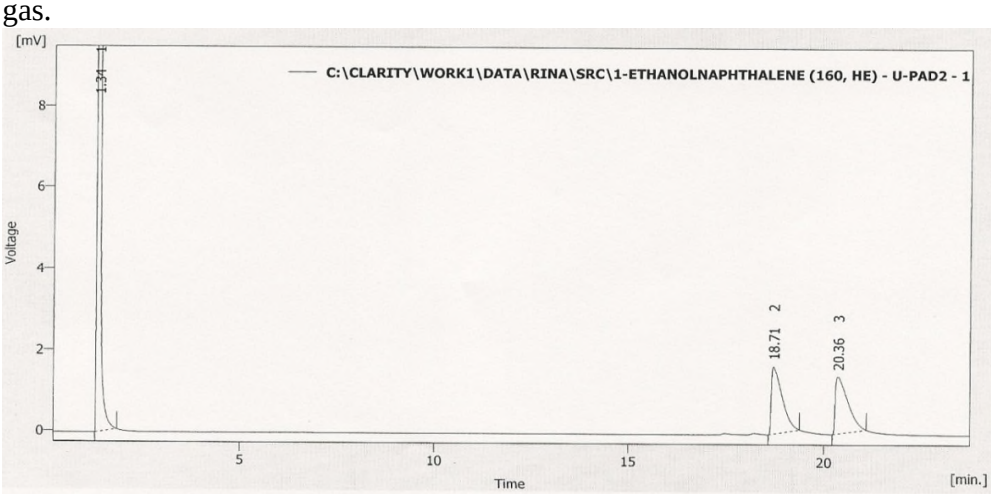
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1245 2h (140, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 11.748 | 47.564 | 3.236 | 98.8 | 98.2 | 0.24 | |
| 2 | 12.688 | 0.576 | 0.061 | 1.2 | 1.8 | 0.15 | |
| | Total | 48.140 | 3.296 | 100.0 | 100.0 | | |

Racemic **1-(1-naphthyl)ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 160 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



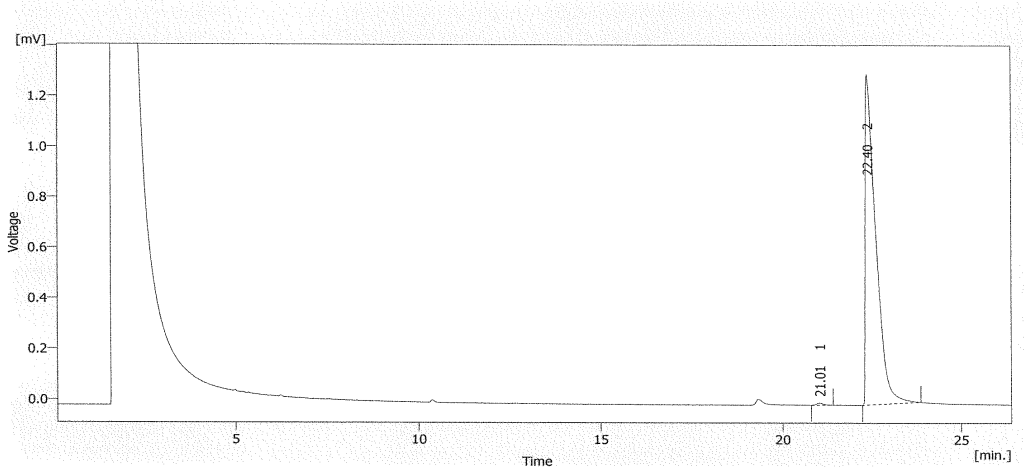

*Result Table (Uncal - C:\CLARITY\WORK1\DATA\RINA\SRC\1-ETHANOLNAPHTHALENE (160, HE) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.336 | 3544.441 | 986.533 | 98.3 | 99.7 | 0.06 | |
| 2 | 18.708 | 31.599 | 1.663 | 0.9 | 0.2 | 0.31 | |
| 3 | 20.356 | 31.364 | 1.399 | 0.9 | 0.1 | 0.37 | |
| | Total | 3607.404 | 989.595 | 100.0 | 100.0 | | |

ATH of **1-acetonaphthone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 160 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:15 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1247 3h (160, He).PRM Page 1 of 1

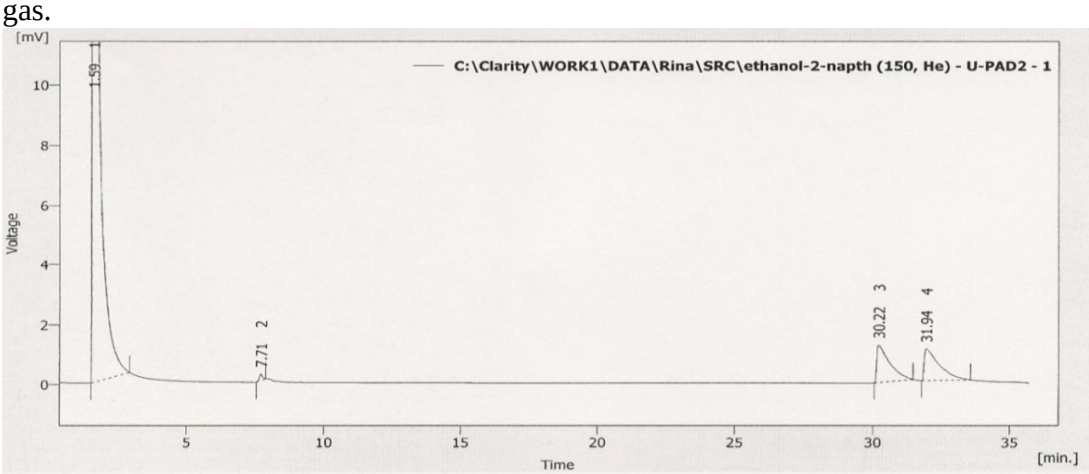
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1247 3h (160, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 21.012 | 0.119 | 0.009 | 0.5 | 0.7 | 0.19 | |
| 2 | 22.404 | 26.025 | 1.308 | 99.5 | 99.3 | 0.30 | |
| | Total | 26.143 | 1.317 | 100.0 | 100.0 | | |

Racemic **1-(2-naphthyl)ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



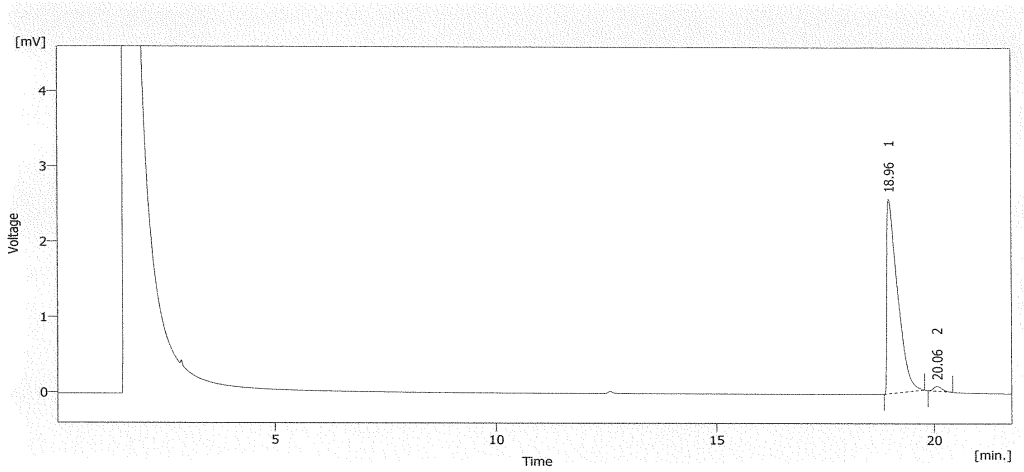

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\ethanol-2-napth (150, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.588 | 4871.620 | 1094.177 | 98.3 | 99.8 | 0.07 | |
| 2 | 7.708 | 1.694 | 0.222 | 0.0 | 0.0 | 0.12 | |
| 3 | 30.220 | 41.189 | 1.260 | 0.8 | 0.1 | 0.51 | |
| 4 | 31.944 | 40.797 | 1.064 | 0.8 | 0.1 | 0.56 | |
| | Total | 4955.299 | 1096.723 | 100.0 | 100.0 | | |

ATH of **2-acetonaphthone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:16 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1248 3h (160, He).PRM Page 1 of 1

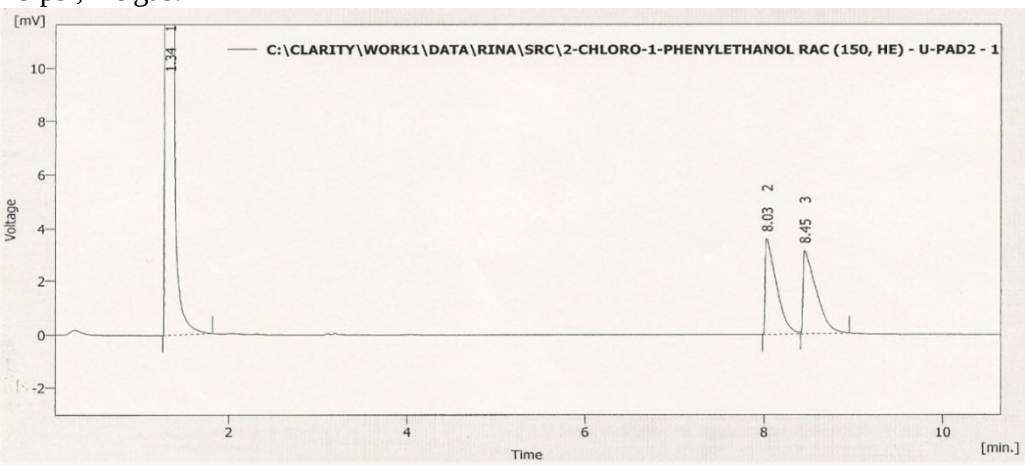
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1248 3h (160, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 18.960 | 44.726 | 2.588 | 98.1 | 97.6 | 0.27 | |
| 2 | 20.056 | 0.870 | 0.064 | 1.9 | 2.4 | 0.21 | |
| | Total | 45.595 | 2.653 | 100.0 | 100.0 | | |

Racemic **1-phenyl-2-chloroethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



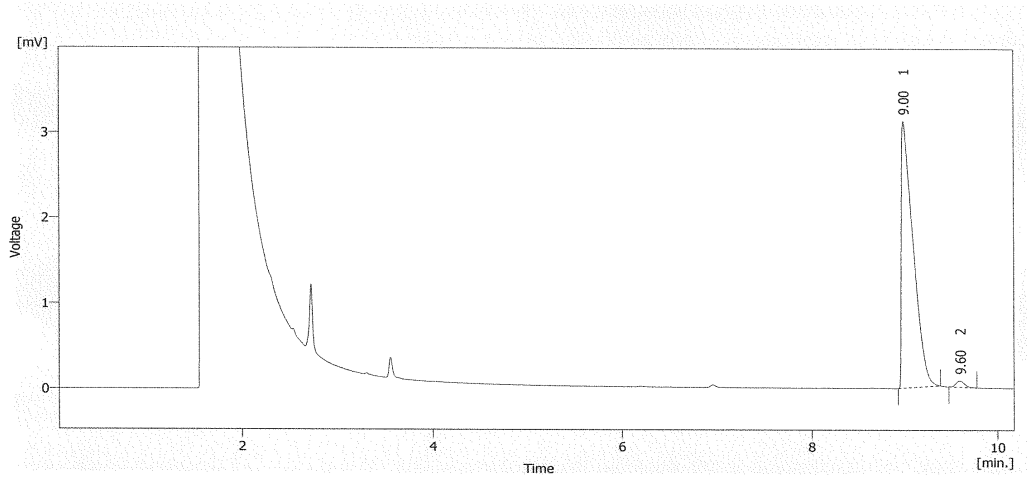

*Result Table (Uncal - C:\CLARITY\WORK1\DATA\RINA\SRC\2-CHLORO-1-PHENYLETHANOL RAC (150, HE) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.336 | 3875.140 | 986.493 | 98.4 | 99.3 | 0.06 | |
| 2 | 8.028 | 31.682 | 3.602 | 0.8 | 0.4 | 0.14 | |
| 3 | 8.448 | 31.688 | 3.126 | 0.8 | 0.3 | 0.16 | |
| | Total | 3938.511 | 993.221 | 100.0 | 100.0 | | |

ATH of **2-chloroacetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 150 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:17 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1249 1h (150, He).PRM Page 1 of 1

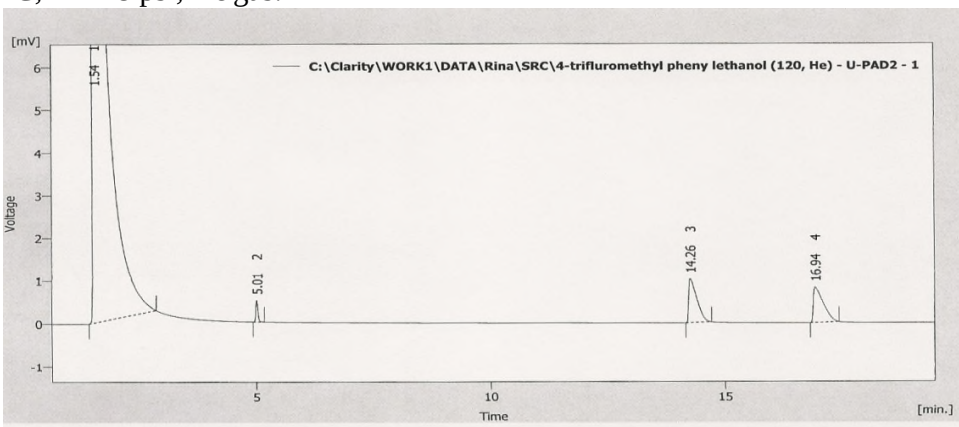
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1249 1h (150, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 8.996 | 27.613 | 3.132 | 98.3 | 97.6 | 0.14 | |
| 2 | 9.596 | 0.488 | 0.076 | 1.7 | 2.4 | 0.10 | |
| | Total | 28.101 | 3.208 | 100.0 | 100.0 | | |

Racemic **1-[4'-(trifluoromethyl)phenyl]ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 120 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



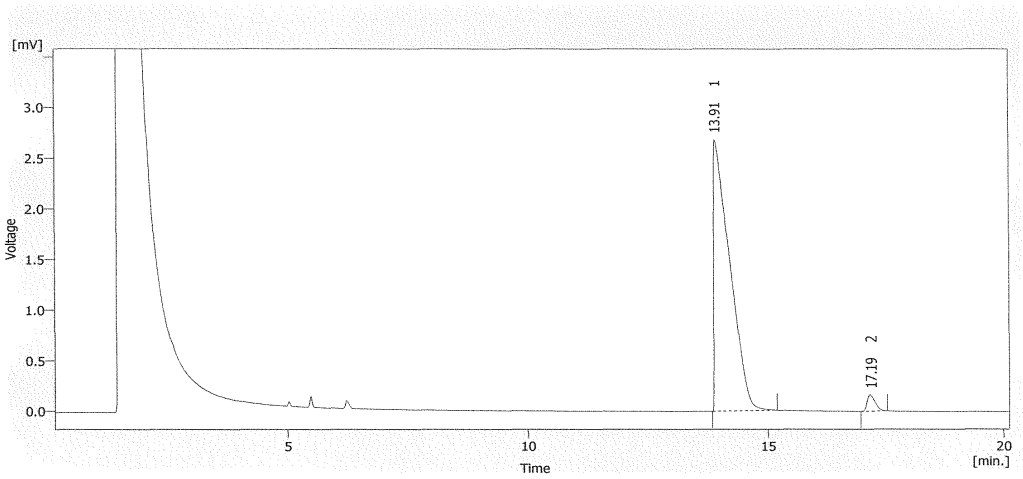

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\4-trifluromethyl pheny lethanol (120, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.544 | 5427.493 | 1094.171 | 99.5 | 99.8 | 0.08 | |
| 2 | 5.008 | 1.577 | 0.501 | 0.0 | 0.0 | 0.05 | |
| 3 | 14.264 | 12.355 | 1.028 | 0.2 | 0.1 | 0.19 | |
| 4 | 16.936 | 12.229 | 0.830 | 0.2 | 0.1 | 0.24 | |
| | Total | 5453.653 | 1096.532 | 100.0 | 100.0 | | |

ATH of **4'-(trifluoromethyl)acetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 120 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:19 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1250 1.5h (120, He).PRM Page 1 of 1

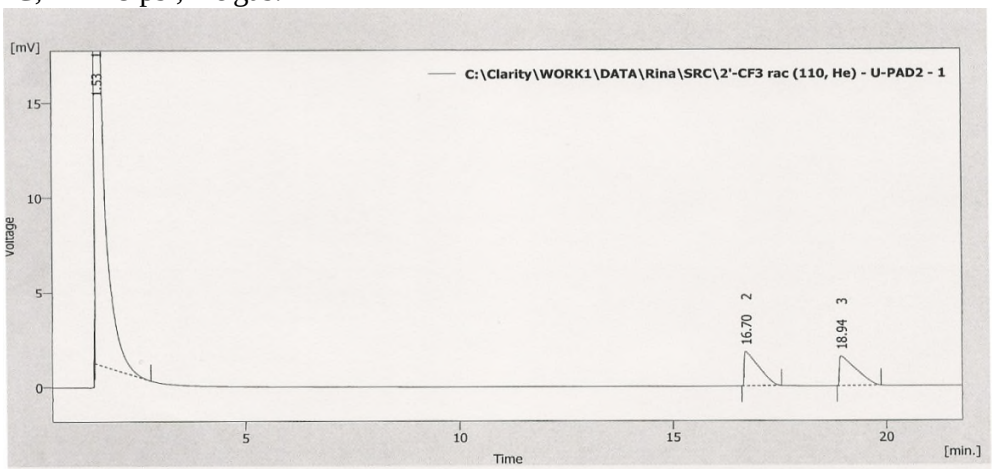
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1250 1.5h (120, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 13.912 | 61.981 | 2.689 | 97.2 | 94.2 | 0.38 | |
| 2 | 17.188 | 1.763 | 0.164 | 2.8 | 5.8 | 0.17 | |
| | Total | 63.744 | 2.853 | 100.0 | 100.0 | | |

Racemic **1-[2'-(trifluoromethyl)phenyl]ethanol**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 110 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



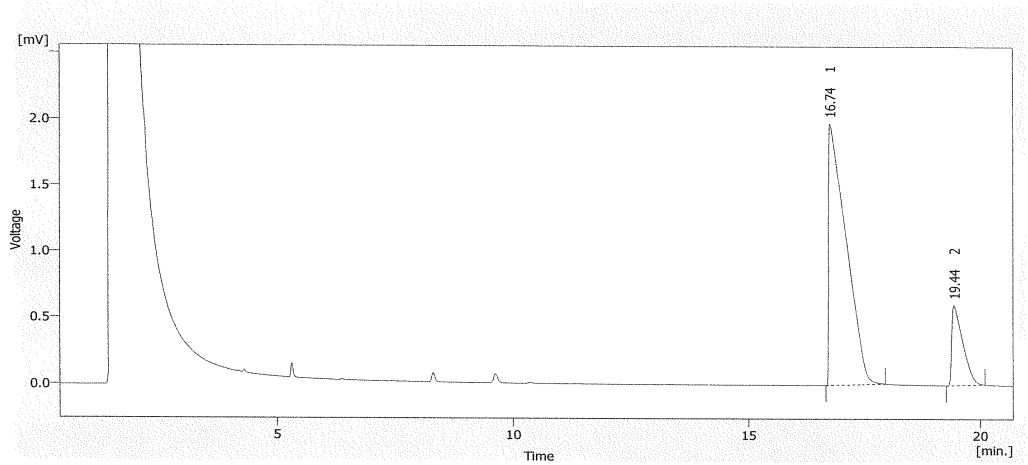

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\2'-CF3 rac (110, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.528 | 6120.494 | 1093.103 | 98.6 | 99.7 | 0.09 | |
| 2 | 16.704 | 42.618 | 1.821 | 0.7 | 0.2 | 0.38 | |
| 3 | 18.940 | 42.485 | 1.572 | 0.7 | 0.1 | 0.45 | |
| | Total | 6205.597 | 1096.495 | 100.0 | 100.0 | | |

ATH of **2'-(trifluoromethyl)acetophenone**: GC analysis on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 110 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:20 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1251 2.5h (110, He).PRM Page 1 of 1

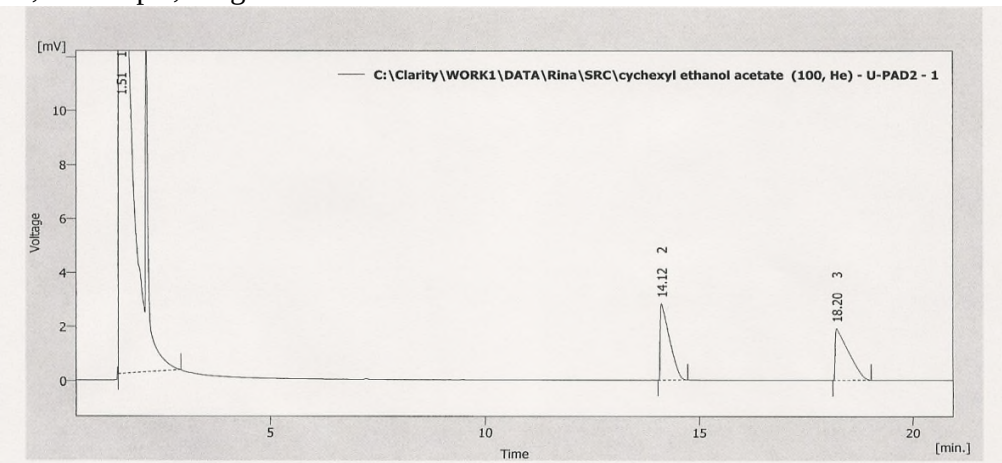
**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1251 2.5h (110, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 16.736 | 52.250 | 1.976 | 84.2 | 76.6 | 0.43 | |
| 2 | 19.444 | 9.827 | 0.603 | 15.8 | 23.4 | 0.26 | |
| | Total | 62.077 | 2.579 | 100.0 | 100.0 | | |

Racemic **1-cyclohexylethanol**: GC analysis of acetate derivative on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 100 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.



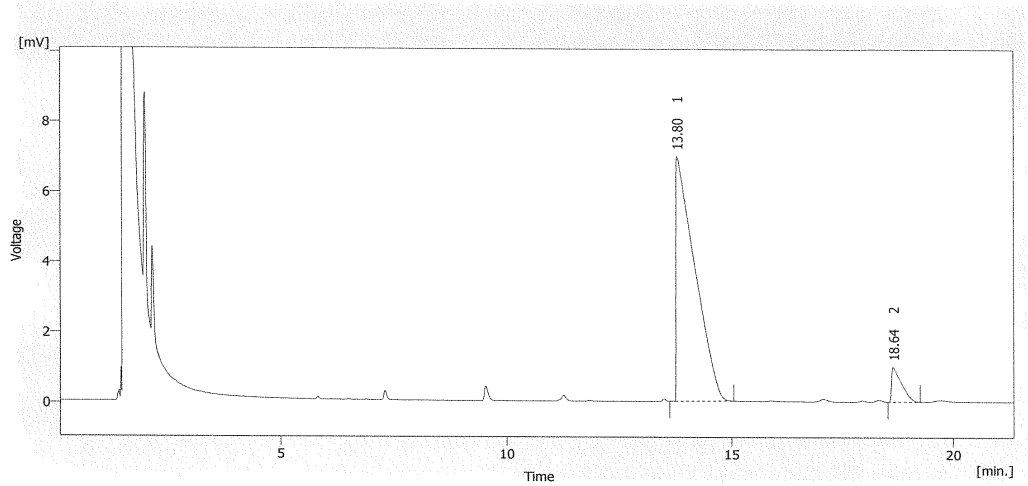

*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC\cychexyl ethanol acetate (100, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 1.508 | 6892.184 | 1094.028 | 98.8 | 99.6 | 0.10 | |
| 2 | 14.120 | 42.039 | 2.826 | 0.6 | 0.3 | 0.24 | |
| 3 | 18.204 | 42.112 | 1.920 | 0.6 | 0.2 | 0.36 | |
| | Total | 6976.335 | 1098.775 | 100.0 | 100.0 | | |

ATH of **cyclohexyl methyl ketone**: GC analysis of acetate derivative on CP-Chirasil-Dex-Cβ 25m x 0.25mm x 0.25µm, Oven Temp = 100 $^{o}$C, Det .Temp = 220 $^{o}$C, Inj. Temp = 220 $^{o}$C, P = 18 psi, He gas.

13/04/2015 11:23 Chromatogram C:\Clarity\WORK1\DATA\Rina\SRC 1101-1200\SRC 1196 final acetate (100, He).PRM Page 1 of 1

**Clarity - Chromatography SW**
DataApex
www.dataapex.com
GC1 Helium
Column: CP-Chiralsil-Dex CB 25mx0.25mmx0.25um




*Result Table (Uncal - C:\Clarity\WORK1\DATA\Rina\SRC 1101-1200\SRC 1196 final acetate (100, He) - U-PAD2 - 1)*

| | Reten. Time [min] | Area [mV.s] | Height [mV] | Area [%] | Height [%] | W05 [min] | Compound Name |
|---|---|---|---|---|---|---|---|
| 1 | 13.796 | 202.514 | 6.984 | 92.9 | 87.5 | 0.47 | |
| 2 | 18.644 | 15.540 | 0.997 | 7.1 | 12.5 | 0.25 | |
| | Total | 218.054 | 7.981 | 100.0 | 100.0 | | |