# $P(i\text{-}PrNCH_2CH_2)_3N$ as a Lewis-Base Catalyst for the Synthesis of β-Hydroxynitriles Using TMSAN

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**Supporting Information**

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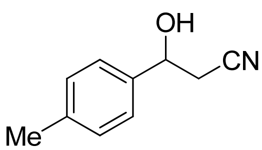
## General Information

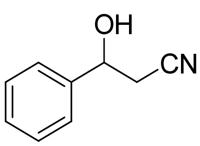
All reactions were carried out under inert atmosphere using oven dried glassware and a magnetic stirrer. THF was distilled and dried over sodium. Trimethylsilylacetonitrile (TMSAN), proazaphosphatrane **1a** and all aldehydes were purchased from commercial sources and were used without further purification. Products were purified via column chromatography using hexane/ethyl acetate. $^{1}$H and $^{13}$C nmr spectra were obtained on a VXR-300 and a VXR-400 NMR spectrometer, respectively. All NMR spectra were taken in $CDCl_3$. Thin layer chromatography was used to monitor reaction progress.

**β-Hydroxy-4-methyl- benzenepropanenitrile (Table 1, entry 6)[1]:**



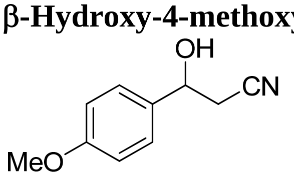

The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 91% isolated yield.

**β-Hydroxybenzenepropanenitrile (Table 2, entry 1)[2]:**



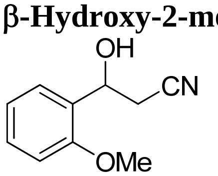

The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 89% isolated yield.

**β-Hydroxy-4-methoxy- benzenepropanenitrile (Table 2, entry 2)[2]:**



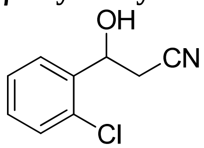

The general procedure was followed for the synthesis and purification; product was afforded as a colorless oil in 83% isolated yield.

**β-Hydroxy-2-methoxy- benzenepropanenitrile (Table 2, entry 3)[3]:**



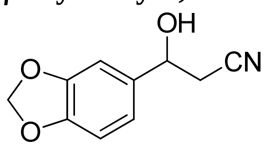

The general procedure was followed for the synthesis and purification; product was afforded as a white solid in 77% isolated yield.

**β-Hydroxy-2-chlorobenzenepropanenitrile (Table 2, entry 4)[3]:**



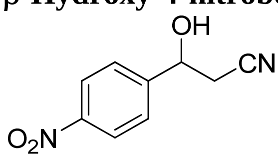
The general procedure was followed for the synthesis and purification; product was afforded as a colorless oil in 94% isolated yield.

**β-Hydroxy-1,3-benzodioxole-5-propanenitrile (Table 2, entry 5):**



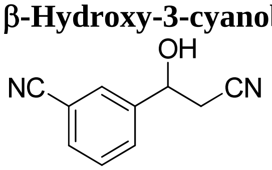
The general procedure was followed for the synthesis and purification; product was afforded as a white solid in 82% isolated yield. $^1$H NMR ($CDCl_3$, 400 MHz): $\delta$ 6.86−6.76 (m, 3H), 5.95 (s, 2H), 4.93−4.89 (m, 1H), 2.81 (d, 1H, $J$ = 4.0 Hz), 2.70−2.69 (m, 2H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 148.1, 135.1, 119.3, 117.6, 108.5, 106.1, 101.4, 69.9, 28.1 ppm; HRMS $m/z$ Calcd. for $C_{10}H_9NO_3$: 191.05824. Found: 191.05876.

**β-Hydroxy-4-nitrobenzenepropanenitrile (Table 2, entry 6)[1]:**



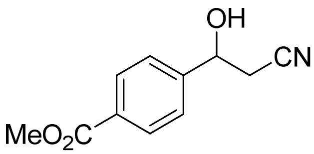
The general procedure was followed for the synthesis and purification; product was afforded as a yellow solid in 94% isolated yield.

**β-Hydroxy-3-cyanobenzenepropanenitrile (Table 2, entry 7):**



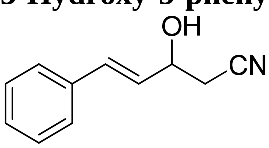
The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 89% isolated yield. $^1$H NMR ($CDCl_3$, 400 MHz): $\delta$ 7.70 (s, 1H), 7.65−7.59 (m, 2H), 7.50 (t, 1H, $J$ = 8.0 Hz), 5.08 (q, 1H, $J$ = 4.8 Hz), 3.62 (d, 1H, $J$ = 4.4 Hz), 2.82−2.71 (m, 2H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 142.9, 132.4, 130.5, 129.9, 129.5, 118.7, 117.2, 112.7, 68.8, 28.3 ppm; HRMS $m/z$ Calcd. for $C_{10}H_8N_2O$: 172.06366. Found: 172.06395.

**Methyl 4-(2-cyano-1-hydroxyethyl)benzoate (Table 2, entry 8):**



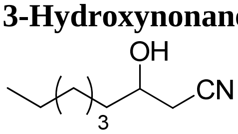
The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 93% isolated yield. $^{1}$H NMR ($CDCl_3$, 300 MHz): $\delta$ 8.99 (d, 2H, $J$ = 8.0 Hz), 7.45 (d, 2H, J = 8.0 Hz), 5.07 (q, 1H, $J$ = 4.0 Hz), 3.89 (s, 3H), 3.27 (d, 1H, $J$ = 4.0 Hz), 2.76−2.74 (m, 2H) ppm; $^{13}$C NMR ($CDCl_3$, 75 MHz): $\delta$ 166.9, 146.2, 130.5, 130.3, 125.8, 117.3, 69.7, 52.6, 28.1 ppm; HRMS $m/z$ Calcd. for $C_{11}H_{11}NO_3$: 205.07389. Found: 205.07416.

**3-Hydroxy-5-phenyl-, 4-pentenenitrile (Table 2, entry 9)[4]:**



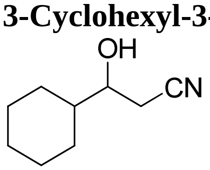
The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 94% isolated yield. $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 135.8, 133.1, 128.9, 128.7, 128.3, 127.0, 117.5, 68.8, 26.6 ppm.

**3-Hydroxynonanenitrile (Table 2, entry 10)[5]:**



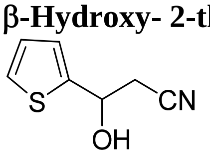
The general procedure was followed for the synthesis and purification; product was afforded as a colorless oil in 85% isolated yield.

**3-Cyclohexyl-3-hydroxypropionitrile (Table 2, entry 11)[5]:**



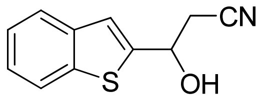
The general procedure was followed for the synthesis and purification; product was afforded as a colorless oil in 86% isolated yield.

**β-Hydroxy- 2-thiophenepropanenitrile, (Table 3, entry 1)[6]:**



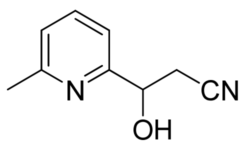
The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 88% isolated yield.

**β-Hydroxy-benzo[*b*]thiophene-2-propanenitrile (Table 3, entry 2)**[6]::



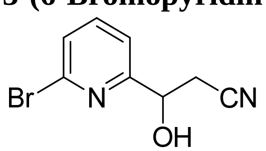
The general procedure was followed for the synthesis and purification; product was afforded as a yellow solid in 93% isolated yield.

**3-Hydroxy-3-(6-methylpyridin-2-yl)propanenitrile (Table 3, entry 3)**:



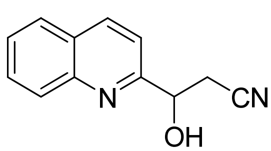
The general procedure was followed for the synthesis and purification; product was afforded as a white solid in 92% isolated yield. $^1$H NMR ($CDCl_3$, 400 MHz): $\delta$ 7.61 (t, 1H, $J$ = 8.0 Hz), 7.18 (d, 1H, $J$ = 8.0 Hz), 7.10 (d, 1H, $J$ = 8.0 Hz), 5.09 (s, 1H), 4.97 (t, 1H, $J$ = 4.0 Hz), 2.87−2.75 (m, 2H), 2.51 (s, 3H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 157.9, 157.4, 137.8, 123.3, 117.6, 117.5, 68.6, 27.4, 24.4 ppm; HRMS *m*/*z* Calcd. for $C_9H_{10}N_2O$: 162.07931. Found: 162.07961.

**3-(6-Bromopyridin-2-yl)-3-hydroxypropanenitrile (Table 3, entry 4)**:



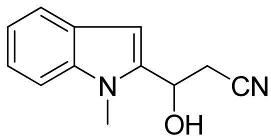
The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 87% isolated yield. $^1$H NMR ($CDCl_3$, 400 MHz): $\delta$ 7.62 (t, 1H, $J$ = 8.0 Hz), 7.46 (d, 2H, $J$ = 4.0 Hz), 5.03 (m, 1H), 4.02 (d, 1H, $J$ = 4.0 Hz), 2.97−2.82 (m, 2H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 160.4, 141.7, 139.8, 128.1, 119.7, 117.3, 69.1, 27.0 ppm; HRMS *m*/*z* Calcd. for $C_8H_7BrN_2O$: 225.97417. Found: 225.97456.

**β-Hydroxy-2-Quinolinepropanenitrile (Table 3, entry 5)**



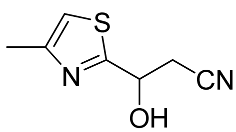
The general procedure was followed for the synthesis and purification; product was afforded as a red oil in 93% isolated yield. $^1$H NMR ($CDCl_3$, 300 MHz): $\delta$ 8.25 (d, 1H, $J$ = 9.0 Hz), 8.10 (d, 1H, $J$ = 9.0 Hz), 7.87 (d, 1H, $J$ = 9.0 Hz), 7.77 (t, 1H, $J$ = 9.0 Hz), 7.59 (t, 1H, $J$ = 9.0 Hz), 7.46 (d, 1H, $J$ = 9.0 Hz), 5.25 (bs, 1H), 5.19 (bs, 1H), 2.96−2.93 (m, 2H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 157.8, 146.7, 138.1, 130.6, 129.1, 128.1, 127.9, 127.4, 118.1, 117.2, 68.9, 27.3 ppm; HRMS *m*/*z* Calcd. for $C_{12}H_{10}N_2O$: 198.07931. Found: 198.07973.

**3-Hydroxy-3-(1-methyl-1*H*-indol-2-yl)propanenitrile (Table 3, entry 6)**



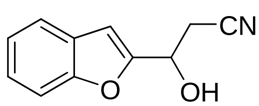

The general procedure was followed for the synthesis and purification; product was afforded as a yellow solid in 81% isolated yield. $^{1}$H NMR ($CD_3CN$, 400 MHz): $\delta$ 7.59 (d, 1H, $J$ = 8.0 Hz), 7.41 (d, 1H, $J$ = 8.0 Hz), 7.24 (t, 1H, $J$ = 8.0 Hz), 7.09 (t, 1H, $J$ = 8.0 Hz), 6.54 (s, 1H), 5.23 (q, 1H, $J$ = 8.0 Hz), 3.97 (d, 1H, $J$ = 8.0 Hz), 3.80 (s, 3H), 3.08−3.06 (m, 2H) ppm; $^{13}$C NMR ($CD_3CN$, 100.6 MHz): $\delta$ 139.9, 138.1, 127.3, 122.1, 120.8, 119.7, 118.2, 117.5, 109.6, 99.2, 62.6, 29.9, 25.3 ppm; HRMS *m*/*z* Calcd. for $C_{12}H_{12}N_2O$: 200.09496. Found: 200.09532.

**3-Hydroxy-3-(4-methylthiazol-2-yl)propanenitrile (Table 3, entry 7)**:



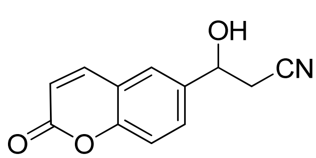

The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 95% isolated yield. $^{1}$H NMR ($CDCl_3$, 400 MHz): $\delta$6.90 (s, 1H), 5.28 (s, 1H), 5.11 (s, 1H), 3.06−2.87 (m, 2H), 2.40 (s, 3H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 171.1, 153.1, 117.2, 114.8, 67.5, 27.2, 17.1 ppm; HRMS *m*/*z* Calcd. for $C_7H_8N_2OS$: 168.03573. Found: 168.03606.

**3-(Benzofuran-2-yl)-3-hydroxypropanenitrile (Table 3, entry 8)**:



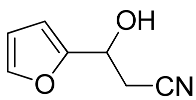

The general procedure was followed for the synthesis and purification; product was afforded as a yellow oil in 64% isolated yield. $^{1}$H NMR ($CDCl_3$, 400 MHz): $\delta$ 7.58−7.55 (m, 1H), 7.48−7.45 (m, 1H), 7.34−7.28 (m, 1H), 7.27−7.22 (m, 1H), 6.77 (s, 1H) 5.17 (q, 1H, $J$ = 8.0 Hz), 3.05−2.91 (m, 3H) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 155.4, 155.0, 127.8, 125.1, 123.4, 121.7, 117.1, 111.6, 104.4, 64.4, 25.2 ppm; HRMS *m*/*z* Calcd. for $C_{11}H_9NO_2$: 187.06333. Found: 187.06371.

**3-Hydroxy-3-(2-oxo-2*H*-chromen-6-yl)propanenitrile (Table 3, entry 9)**:



The general procedure was followed for the synthesis and purification; product was afforded as a yellow solid in 70% isolated yield.$^1$H NMR ($CDCl_3$, 300 MHz): $\delta$ 7.71−7.68 (m, 1H), 7.57−7.54 (m, 2H), 7.30−7.25 (m, 1H), 6.40 (d, 1H, $J$ = 12.4 Hz), 5.13 (t, 1H, $J$ = 6.0 Hz), 3.34 (s, 1H), 2.80 (d, 2H, $J$ = 4.0 Hz) ppm; $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 160.7, 154.1, 143.3, 137.6, 129.2, 125.2, 119.1, 117.7, 117.6, 116.9, 115.5, 69.4, 28.4 ppm; HRMS *m*/*z* Calcd. for $C_{12}H_9NO_3$: 215.05824. Found: 215.05862.

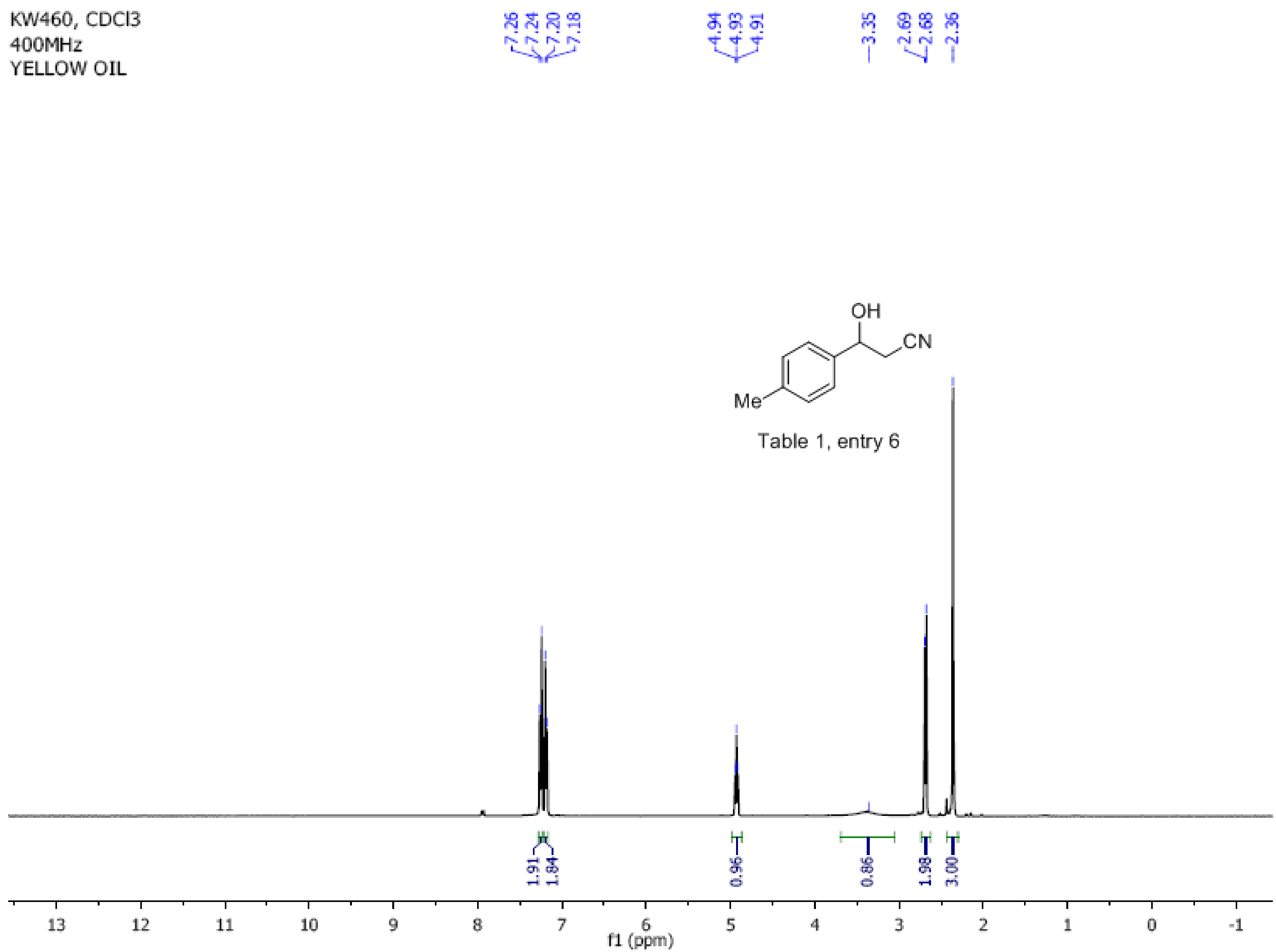
**β-Hydroxy-2-furanpropanenitrile (Table 3, entry 10)**[7]:

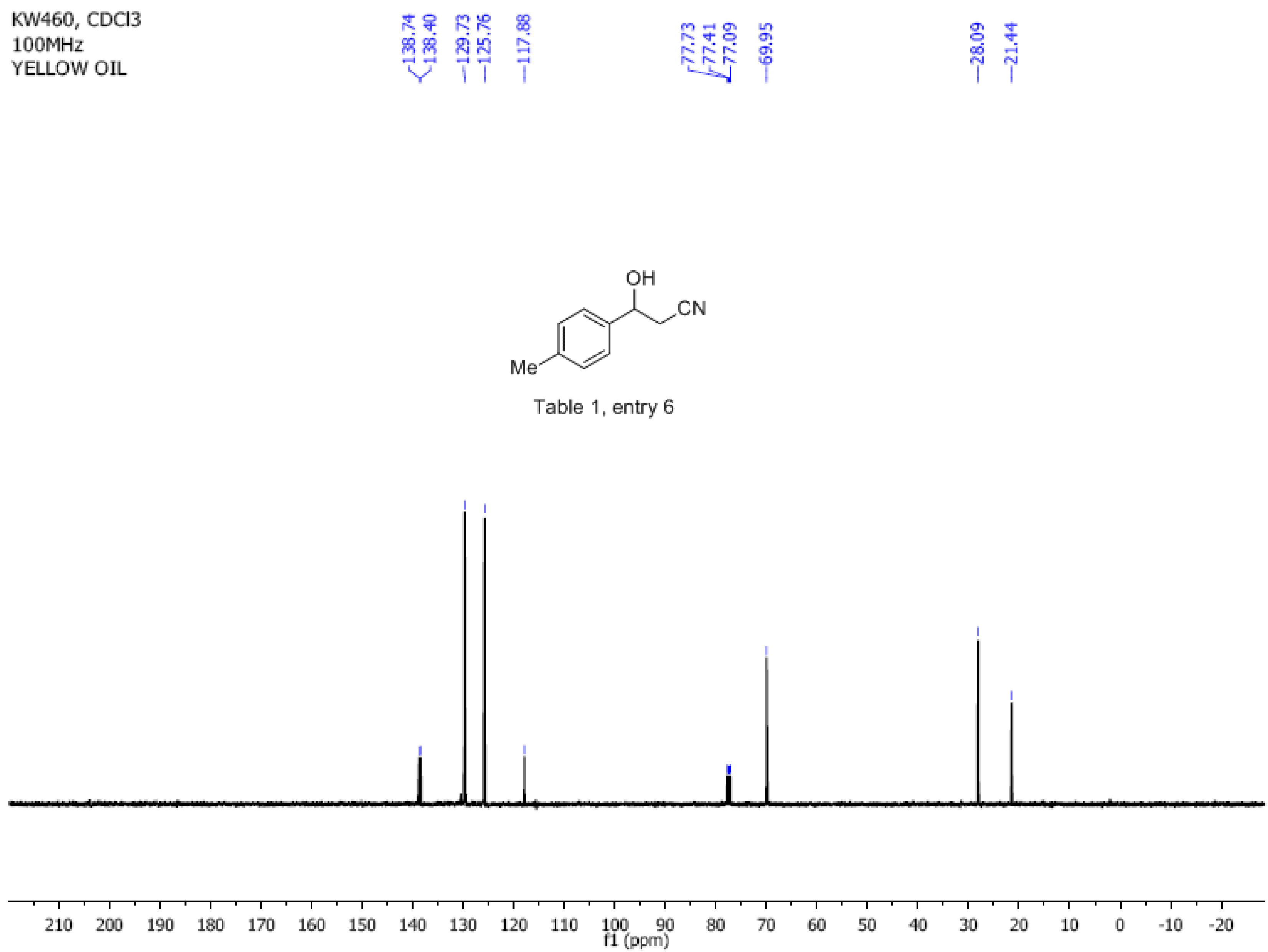



The general procedure was followed for the synthesis and purification; product was afforded as a colorless oil in 81% isolated yield. $^{13}$C NMR ($CDCl_3$, 100.6 MHz): $\delta$ 153.1, 143.1, 117.3, 110.8, 107.7, 63.9, 25.1 ppm.

**Reference**


1. Ankati, H.; Zhu, D.; Yang, Y.; Biehl, E. R.; Hua, L. *J. Org. Chem.* **2009**, *74*, 1658−1662.
2. Feroci, M.; Orsini, M.; Sotgiu, G.; Inesi, A. *Electrochim. Acta* **2008**, *53*, 2346−2354.
3. Kamila, S.; Zhu, D.; Biehl, E. R.; Hua, L. *Org. Lett.* **2006**, *8*, 4429−4431.
4. Itoh, T.; Takagi, Y.; Nishiyama, S. *J. Org. Chem.* **1991**, *56*, 1521−1524.
5. Elenkov, M. M.; Hauer, B.; Janssen, D. B. *Adv. Synth. Catal.* **2006**, *348*, 579−585.
6. Turcu, M. C.; Perkiő, P.; Kanerva, L. T. ARKIVOC **2009**, *3*, 251−263.
7. Kashin, A. N.; Tul'chinskii, M. L.; Beletskaya, I. P. *J. Organomet. Chem.* **1985**, *292*, 205−215.


KW460, CDCl3
400MHz
YELLOW OIL
7.26
7.24
7.20
7.18
4.94
4.93
4.91
3.35
2.69
2.68
2.36
OH
CN
Me
Table 1, entry 6
1.91
1.84
0.96
0.86
1.98
3.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


KW460, CDCl3
100MHz
YELLOW OIL
138.74
138.40
129.73
125.76
117.88
77.73
77.41
77.09
69.95
28.09
21.44
OH
CN
Me
Table 1, entry 6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

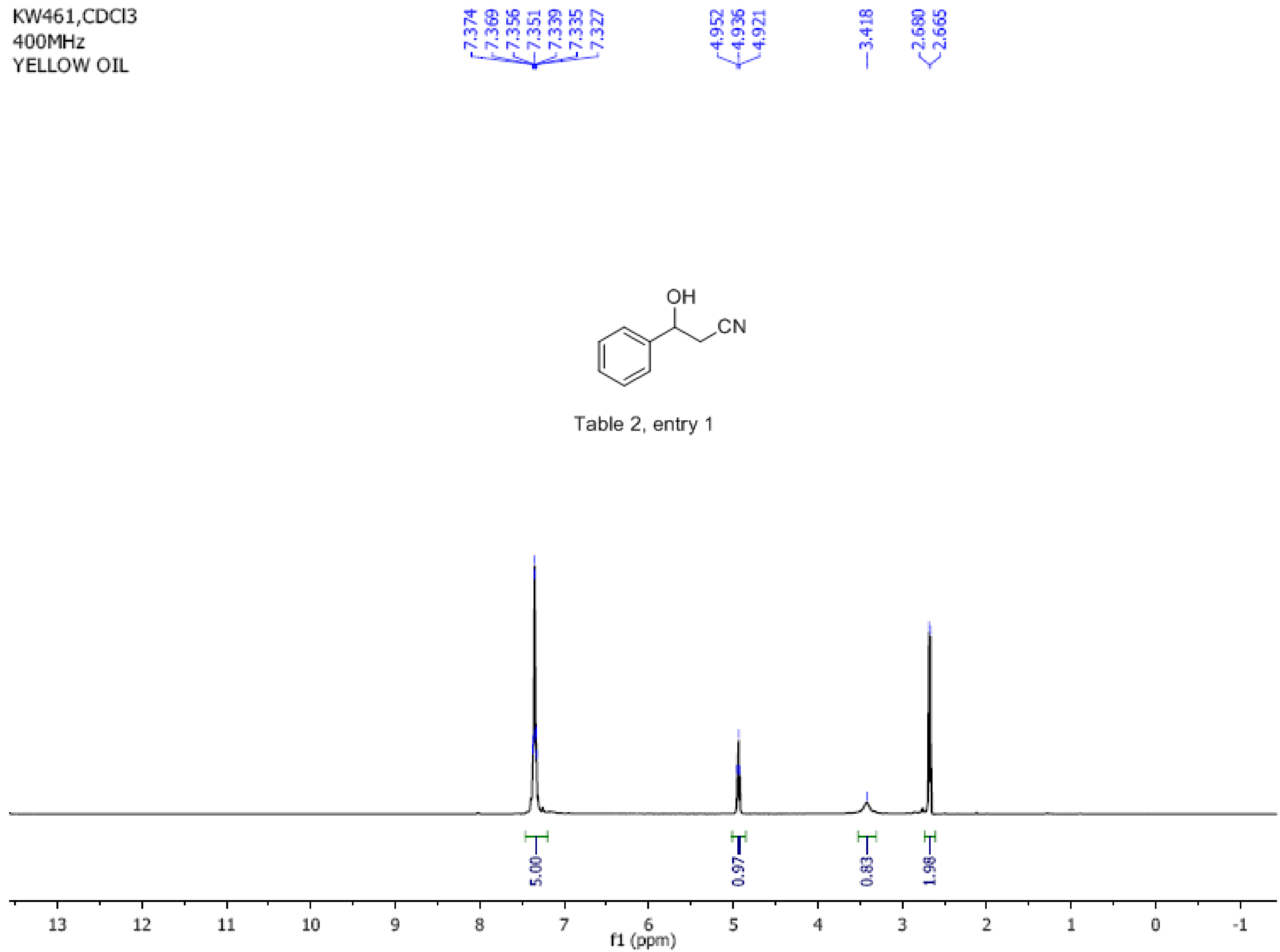
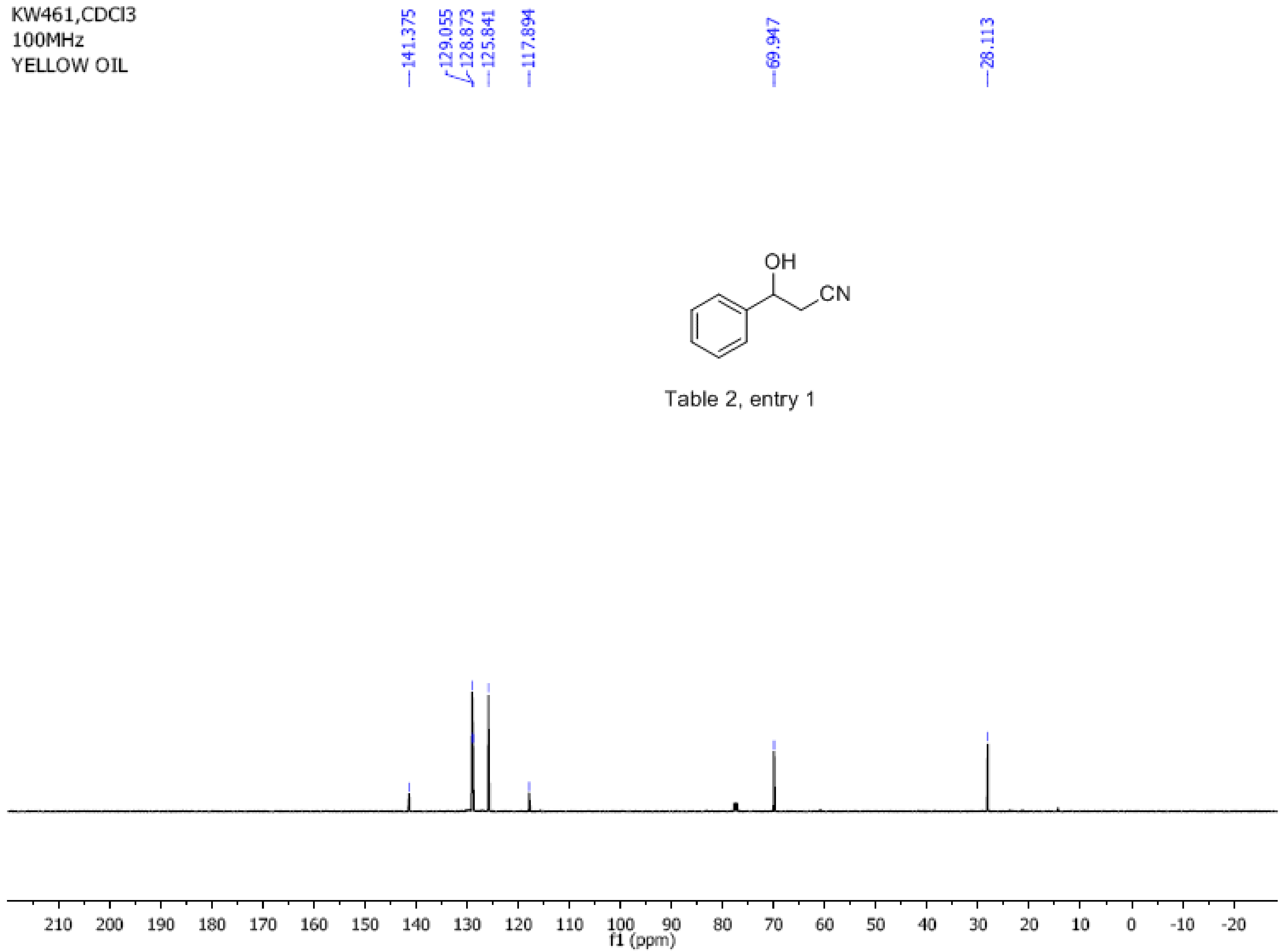
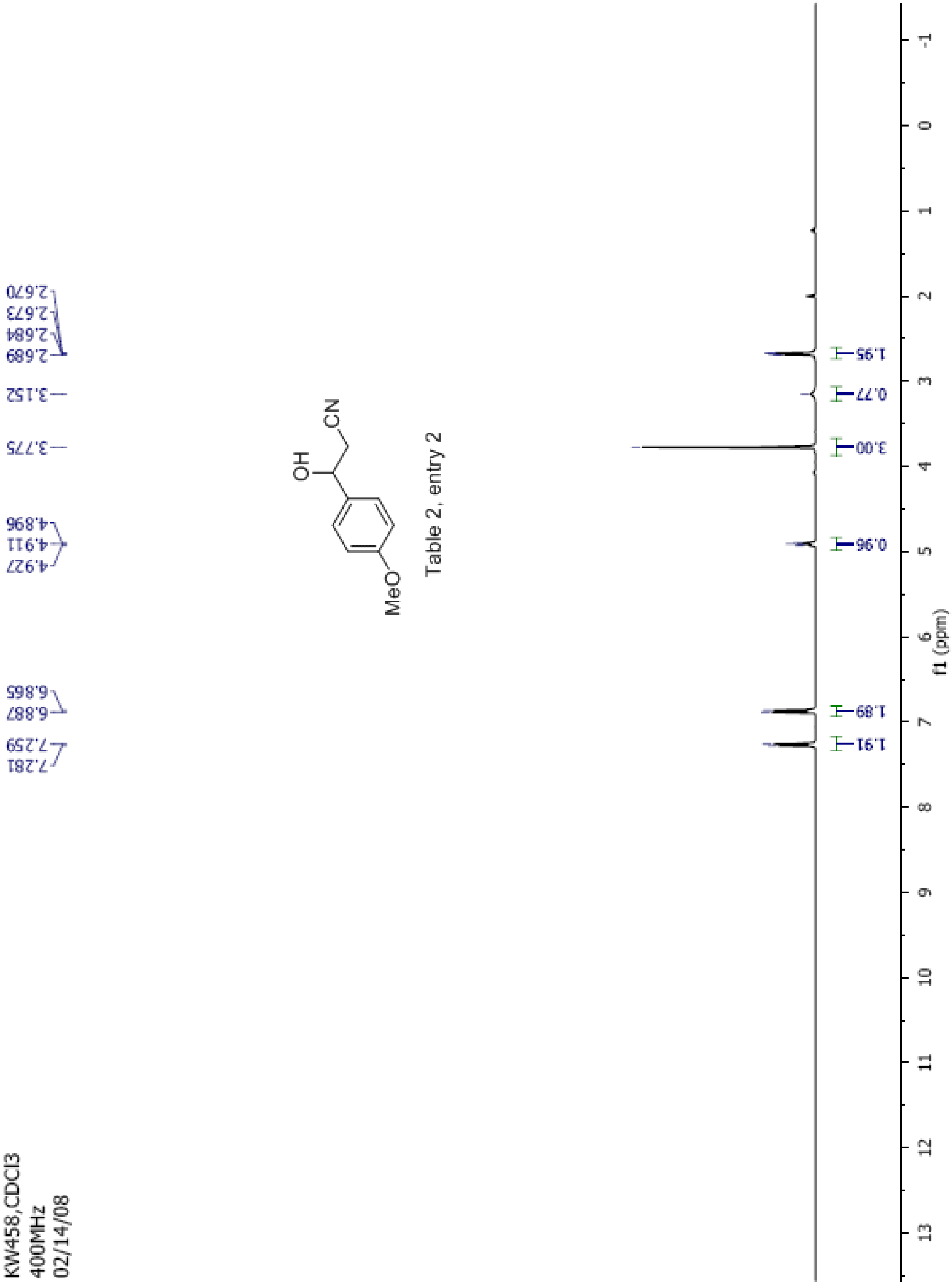
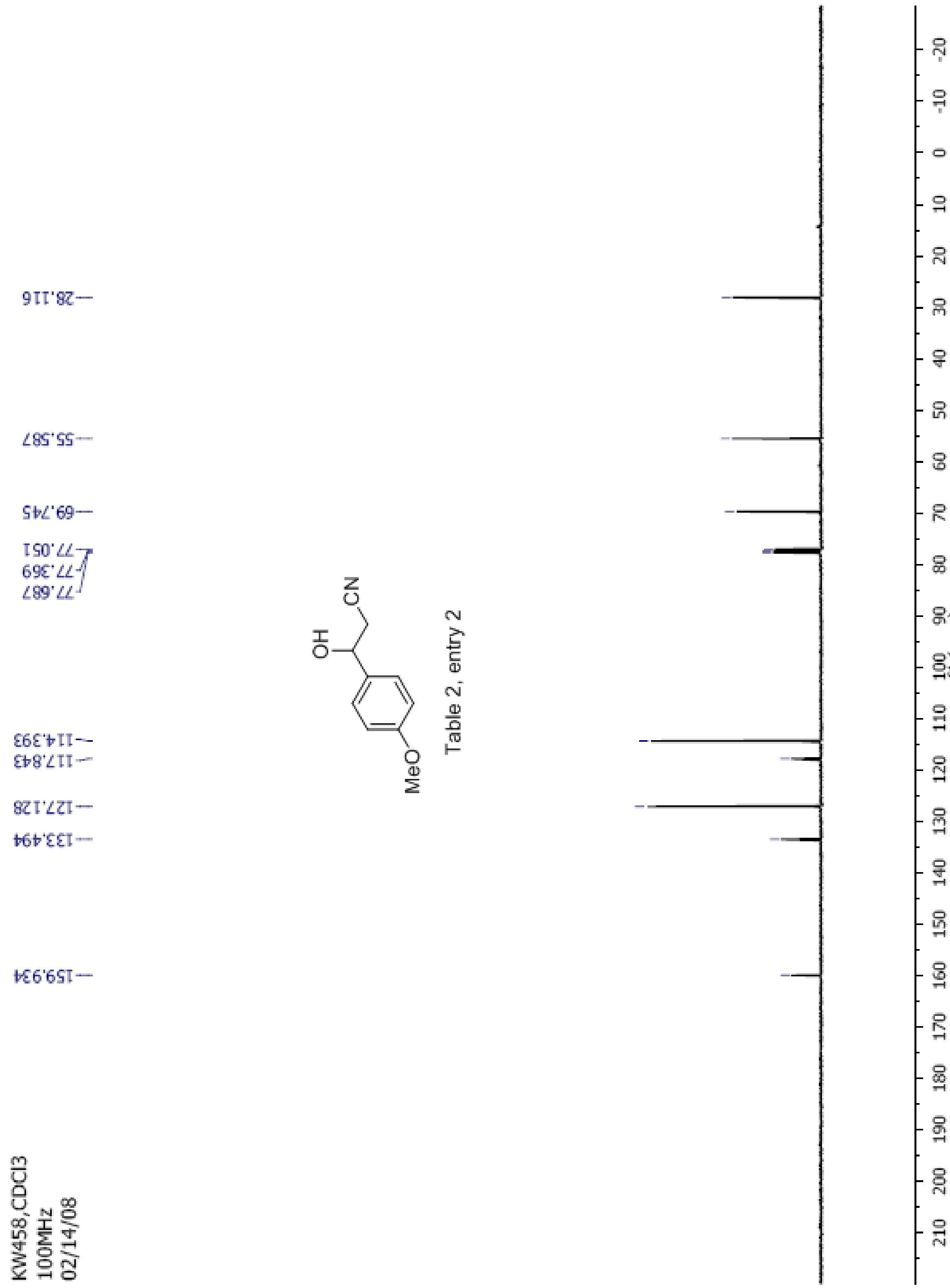


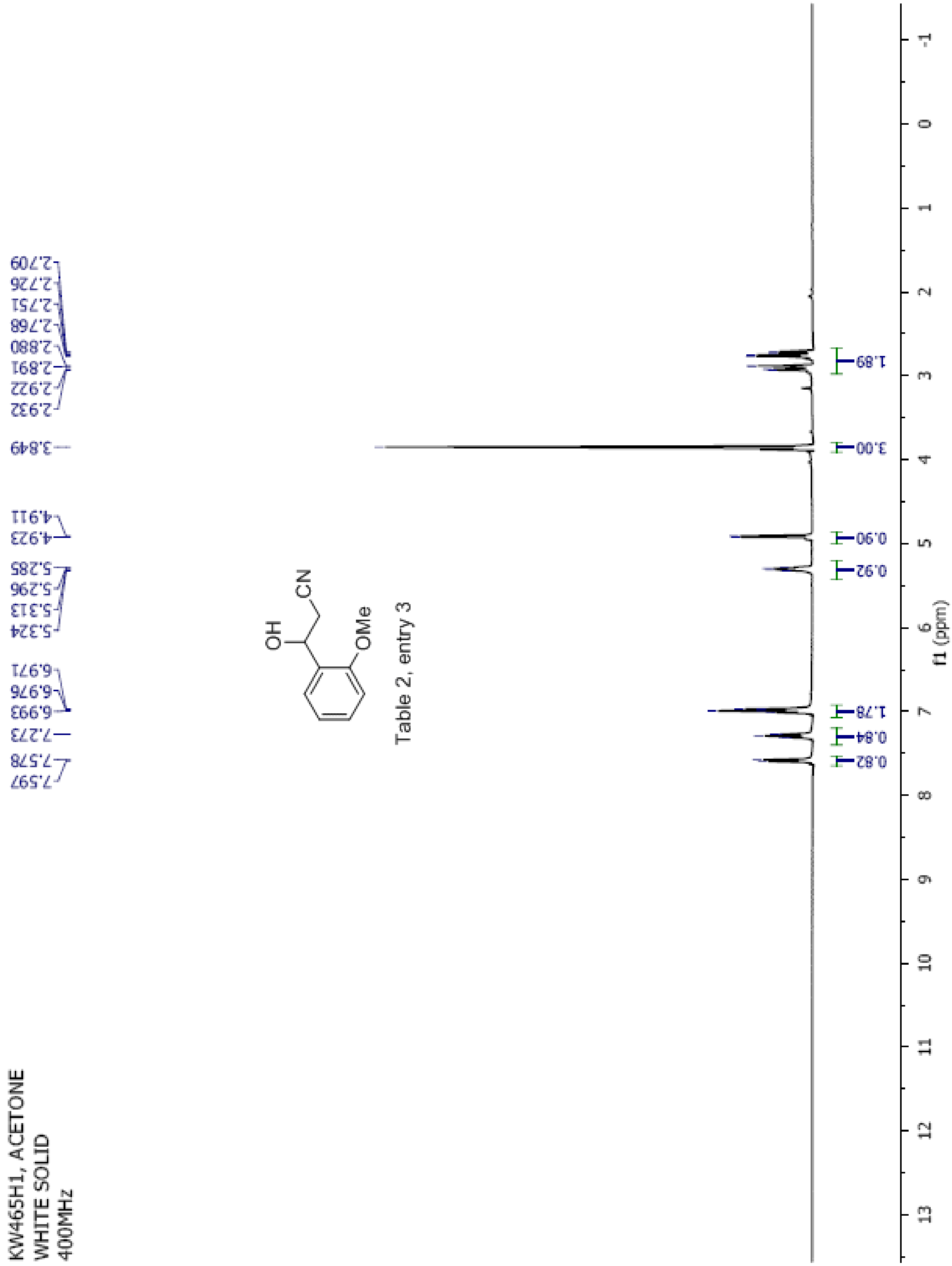

Table 2, entry 1

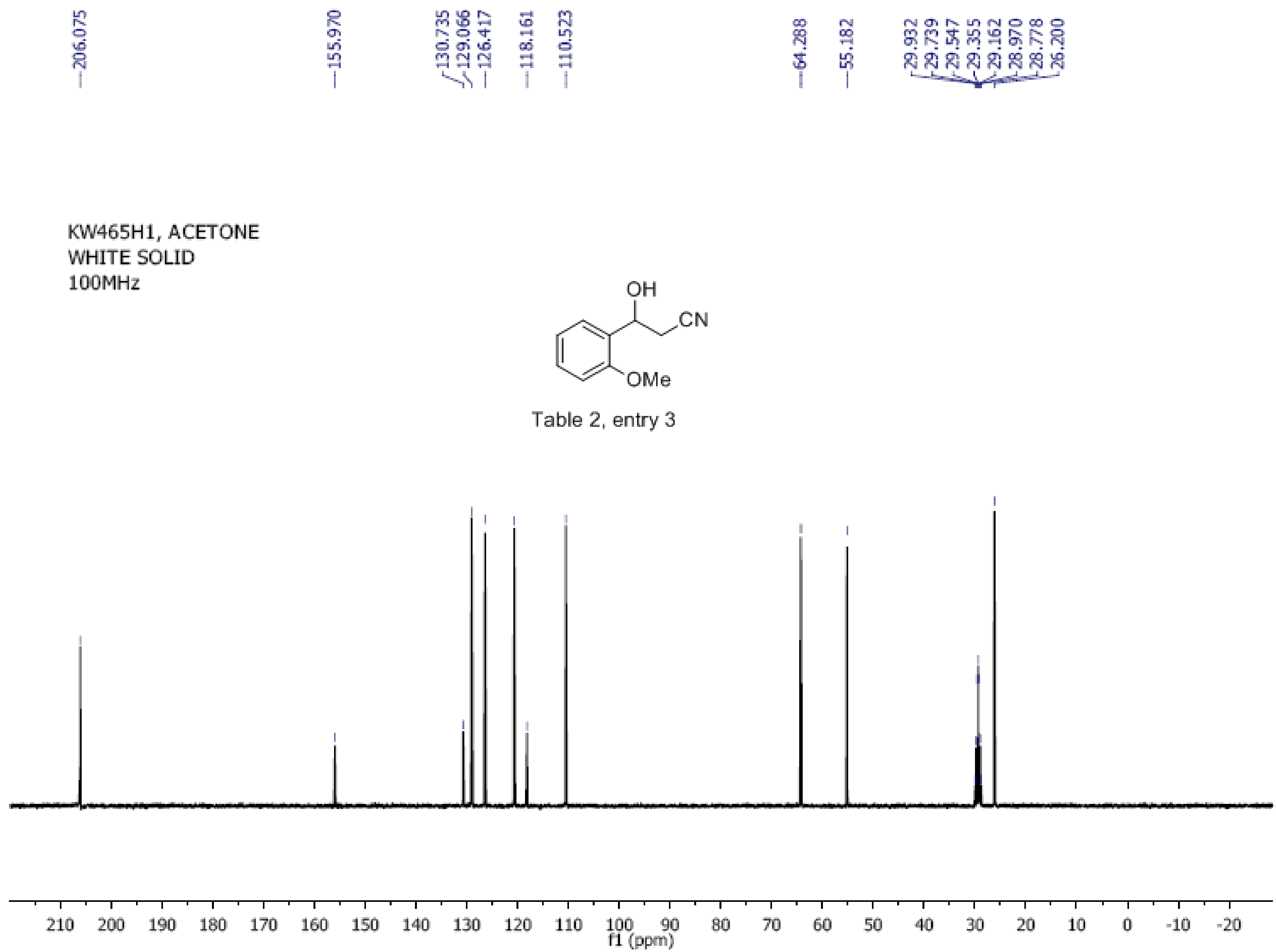

KW461,CDCl3
100MHz
YELLOW OIL
141.375
129.055
128.873
125.841
117.894
69.947
28.113
OH
CN
Table 2, entry 1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

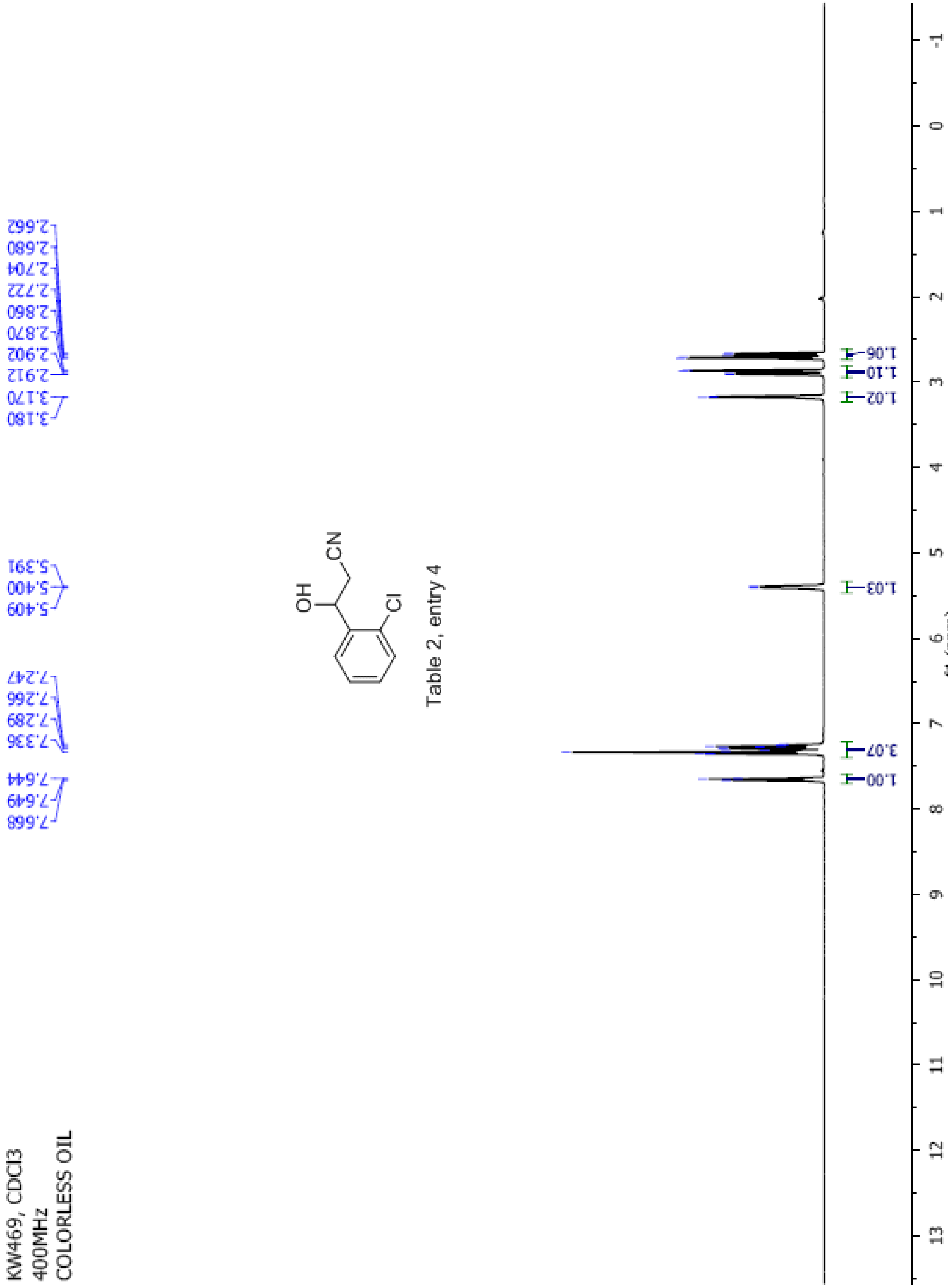


KW458,CDCl3
400MHz
02/14/08
7.281
7.259
6.887
6.865
4.927
4.911
4.896
3.775
3.152
2.689
2.684
2.673
2.670
OH
CN
MeO
Table 2, entry 2
1.91
1.89
0.96
3.00
0.77
1.95
f1 (ppm)


KW458,CDCl3
100MHz
02/14/08
159.934
133.494
127.128
117.843
114.393
77.687
77.369
77.051
69.745
55.587
28.116
MeO
OH
CN
Table 2, entry 2
f1 (ppm)


KW465H1, ACETONE
WHITE SOLID
400MHz
7.597
7.578
7.273
6.993
6.976
6.971
5.324
5.313
5.296
5.285
4.923
4.911
3.849
2.932
2.922
2.891
2.880
2.768
2.751
2.726
2.709
OH
CN
OMe
Table 2, entry 3
0.82
0.84
1.78
0.92
0.90
3.00
1.89
f1 (ppm)


206.075
155.970
130.735
129.066
126.417
118.161
110.523
64.288
55.182
29.932
29.739
29.547
29.355
29.162
28.970
28.778
26.200
KW465H1, ACETONE
WHITE SOLID
100MHz
OH
CN
OMe
Table 2, entry 3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)


KW469, CDCl3
400MHz
COLORLESS OIL
7.668
7.649
7.644
7.336
7.289
7.266
7.247
5.409
5.400
5.391
3.180
3.170
2.912
2.902
2.870
2.860
2.722
2.704
2.680
2.662
OH
CN
Cl
Table 2, entry 4
1.00
3.07
1.03
1.02
1.10
1.06
f1 (ppm)

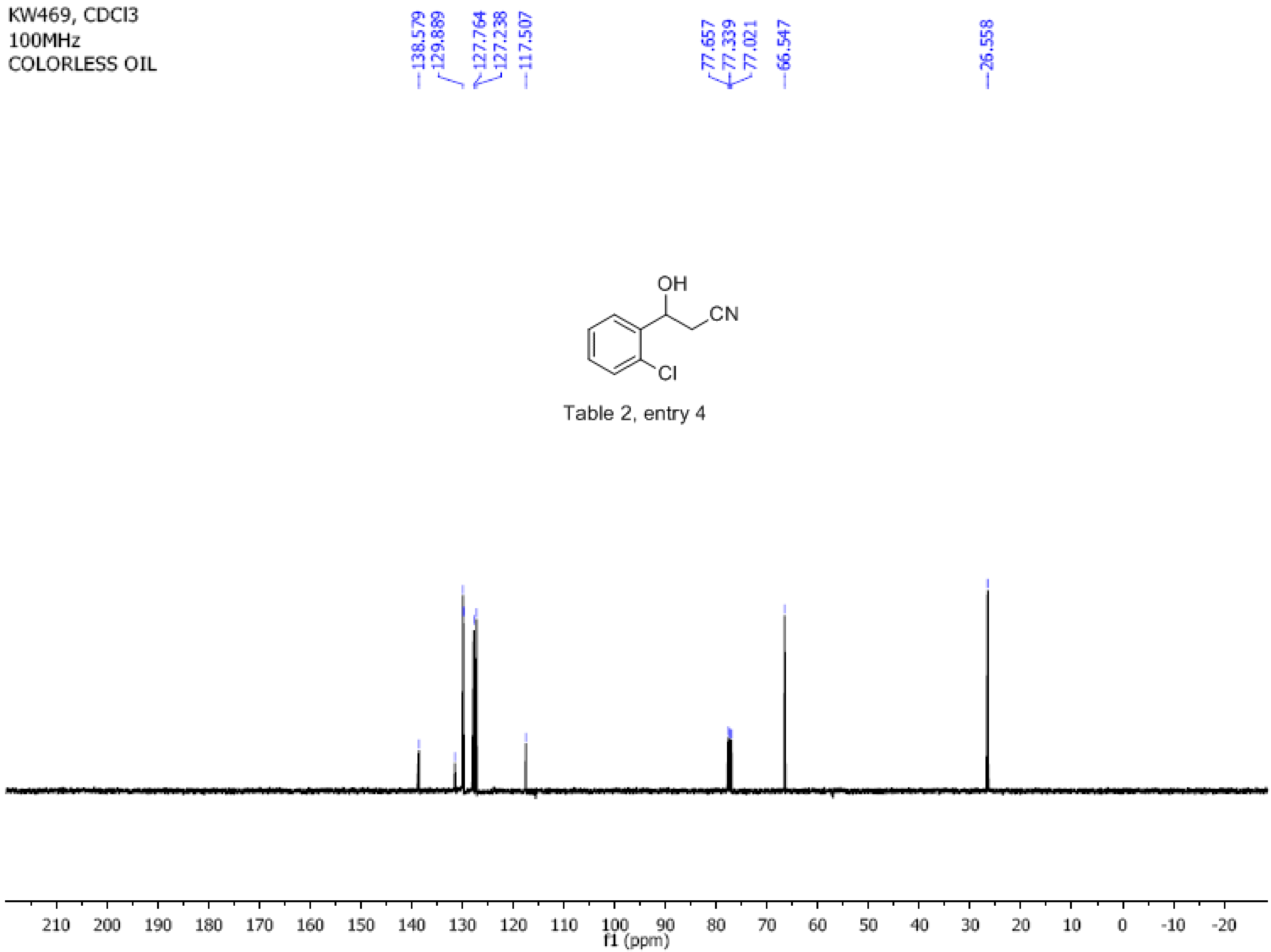
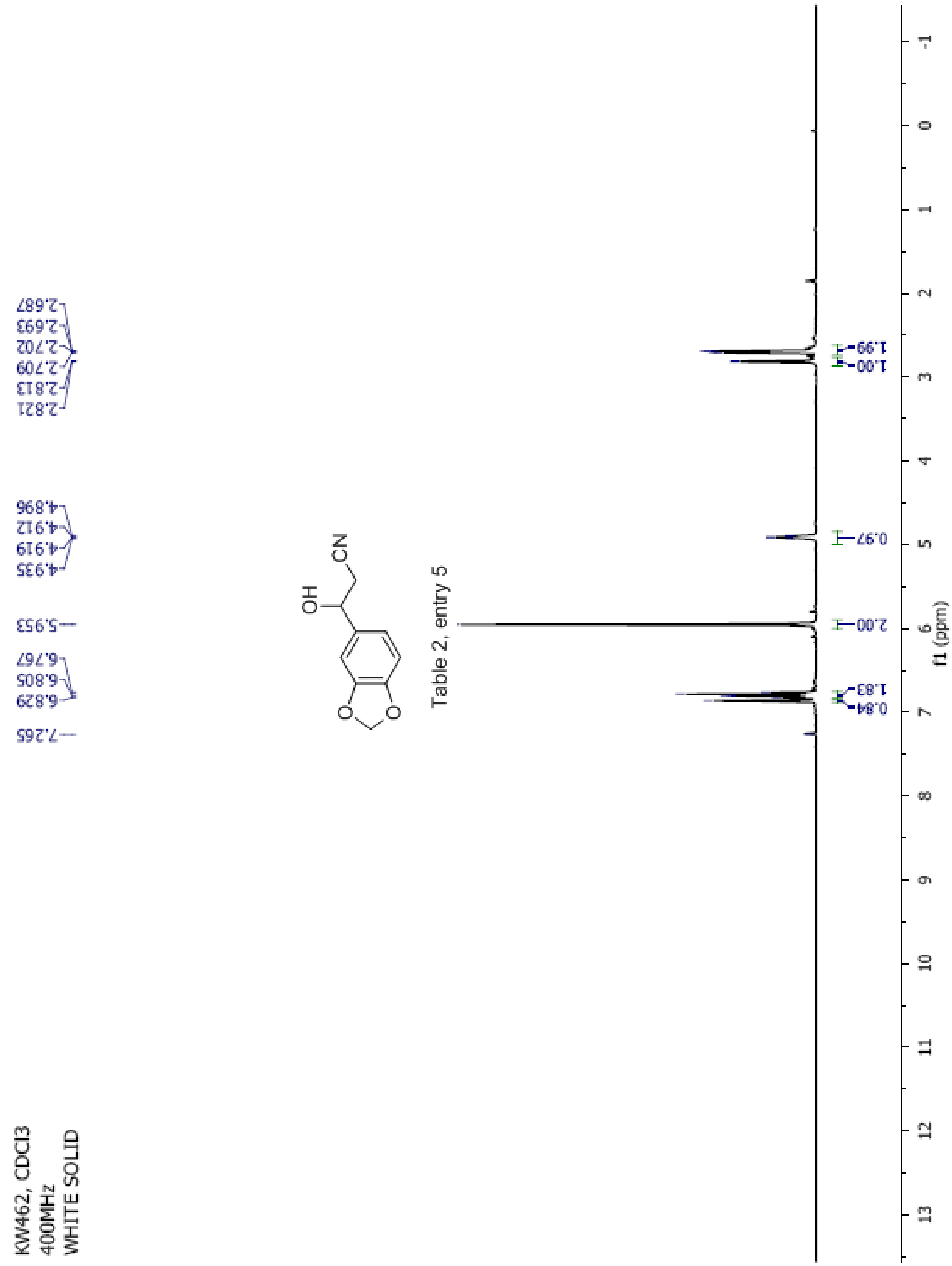


Table 2, entry 4


KW462, CDCl3
400MHz
WHITE SOLID
7.265
6.829
6.805
6.767
5.953
4.935
4.919
4.912
4.896
2.821
2.813
2.709
2.702
2.693
2.687
Table 2, entry 5
OH
CN
O
O
0.84
1.83
2.00
0.97
1.00
1.99
f1 (ppm)

KW462,CDCl3
100MHz
WHITE SOLID

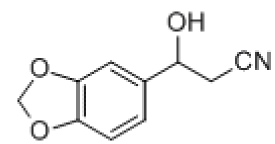
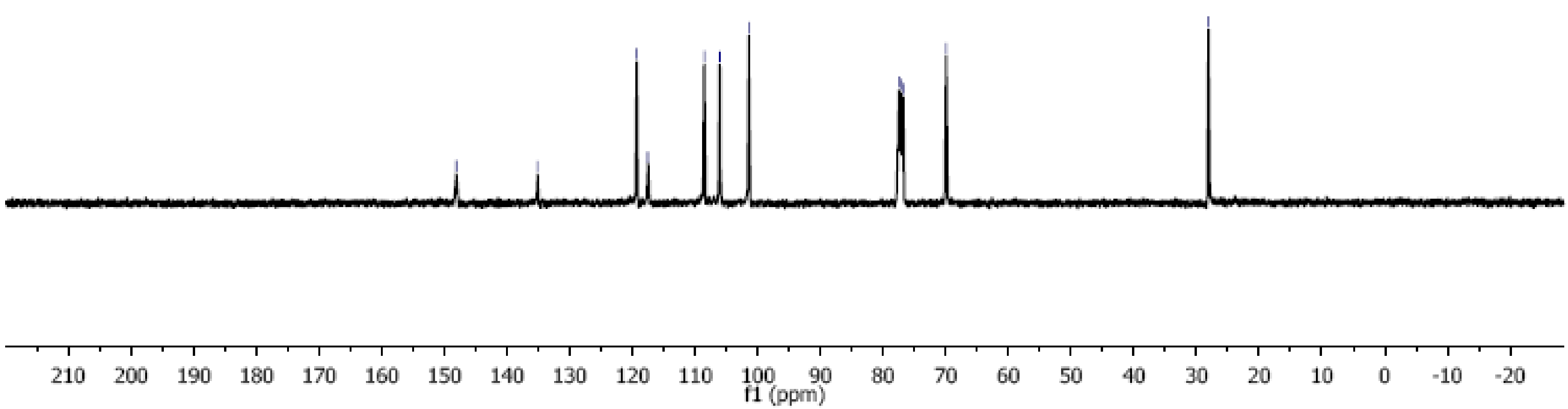



Table 2, entry 5



Manual Peak Matching Report
For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 191.05824 | 191.05876 | 180.98882 | 2.7 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

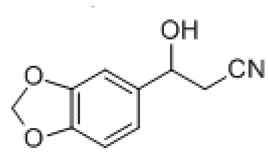



Table 2, entry 5

SPEC: fin084362.dat (06-APR-09 14:49:32)
Samp: KW462
Comm: DP/EI
Oper: kh
Base: 147.93
Peak: 1000.0 mmu
Scan 42 @ 0.71 min (EI +Q1MS LMR UP LR)

Study: ms services
Masses: 35.01 > 650.00
Intensity: 5766834

Scans: 1 > 44

Client: Kuldeep
#Peaks: 633
RIC: 28025415

5.8E+06

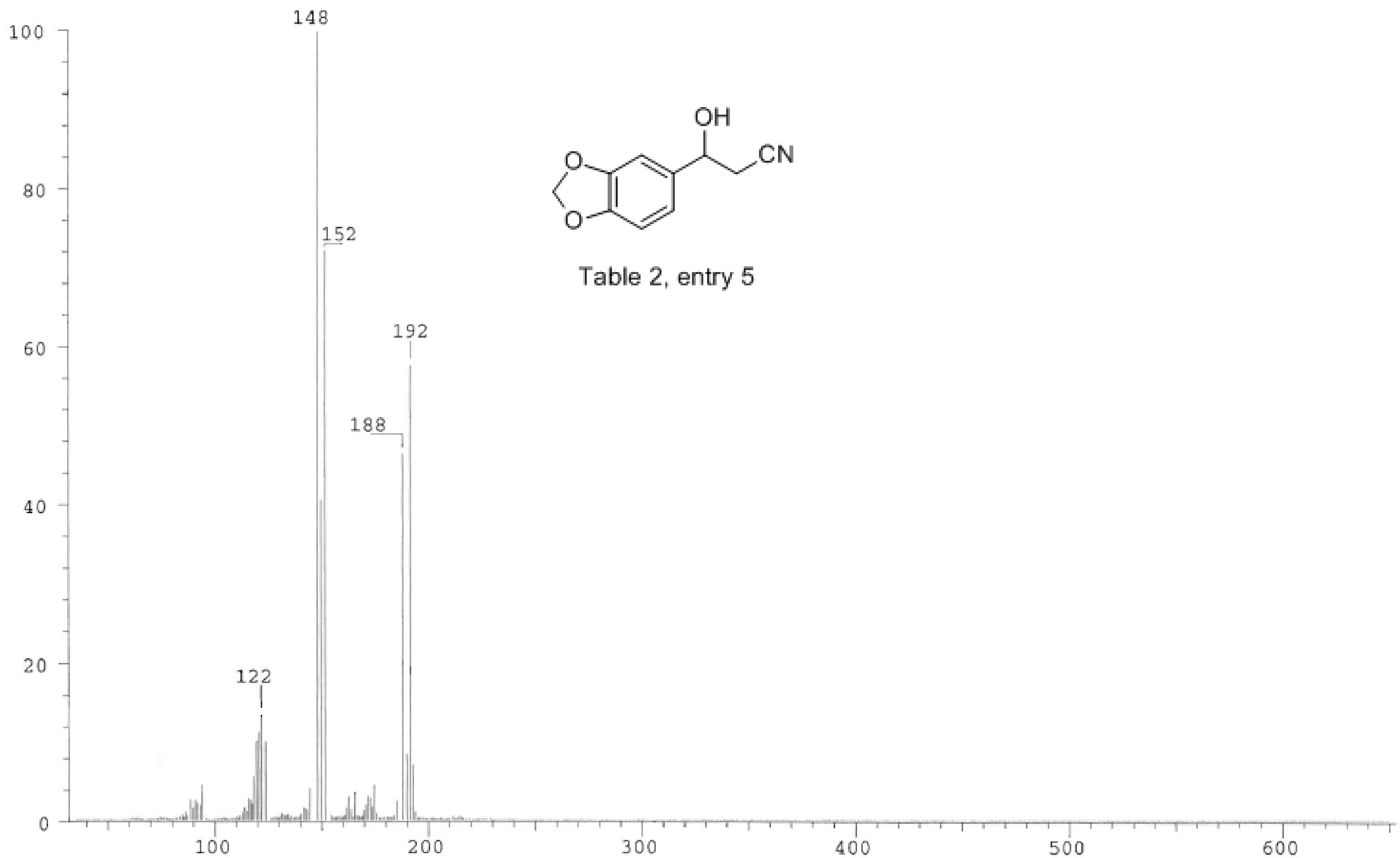



Table 2, entry 5

Date: Mon Apr 6 14:51:42 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

KW463, ACETONE
YELLOW SOLID
400MHz

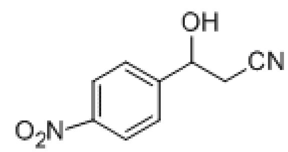
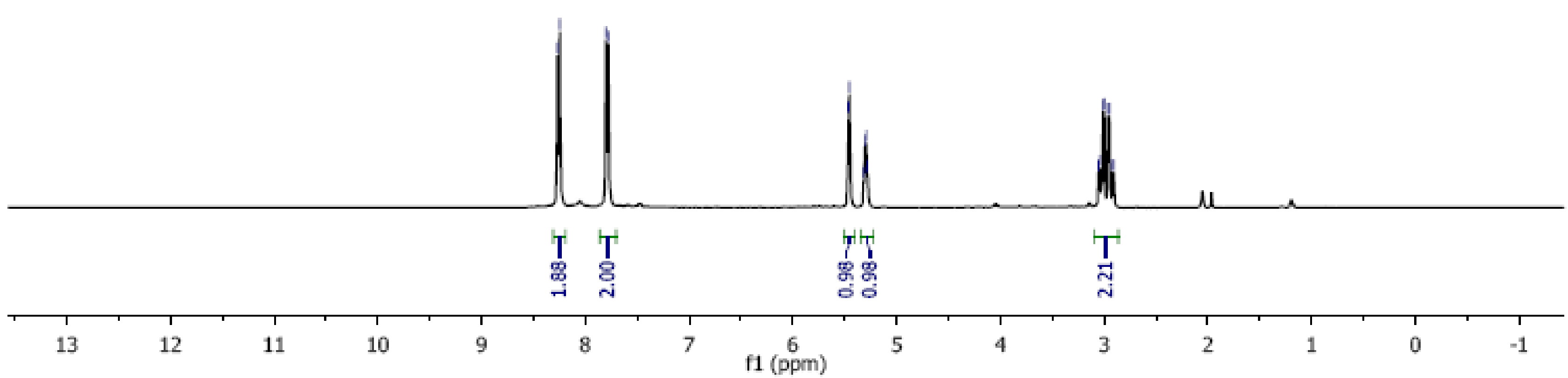
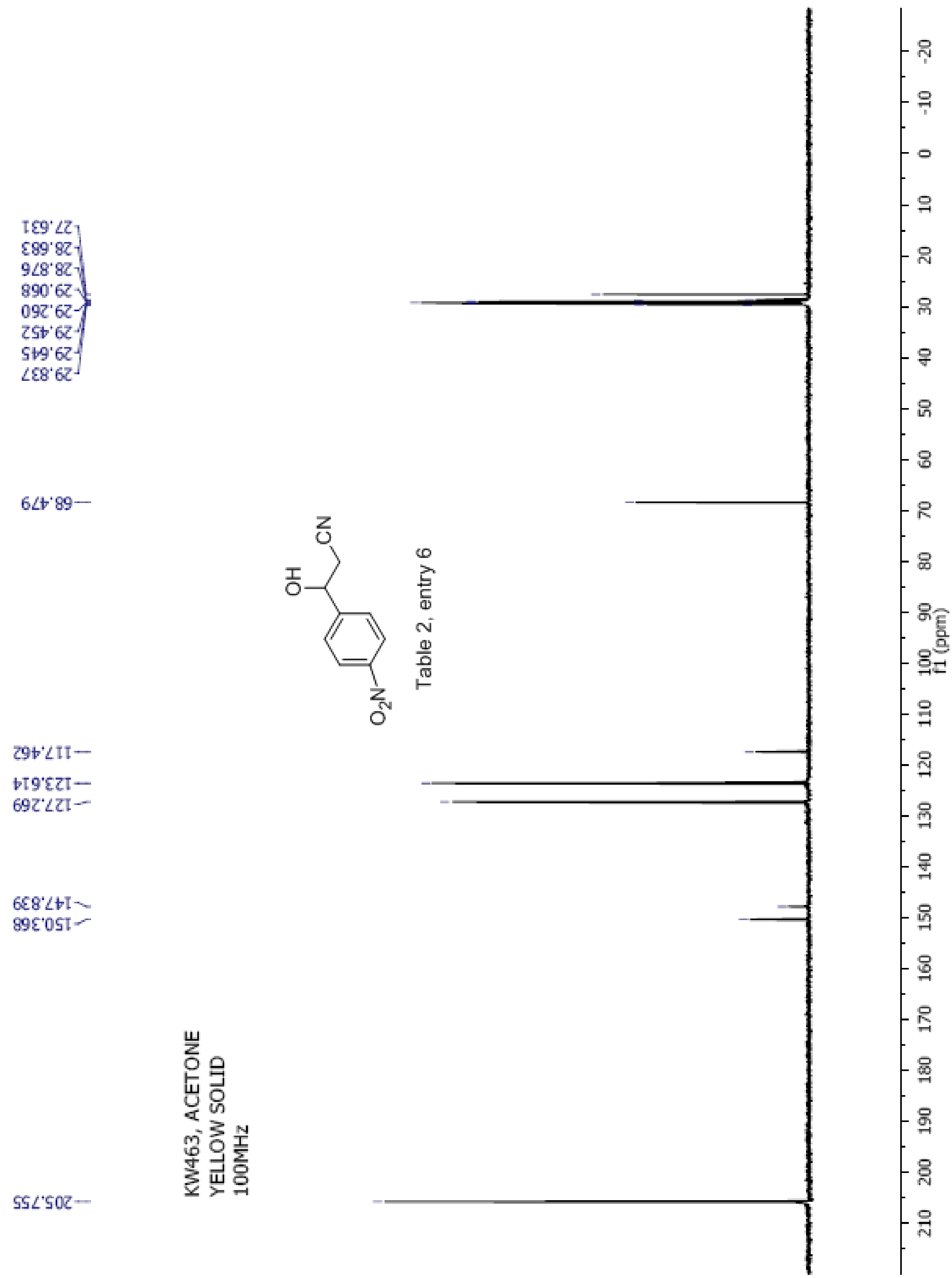


Table 2, entry 6





27.631
28.683
28.876
29.068
29.260
29.452
29.645
29.837
68.479
117.462
123.614
127.269
147.839
150.368
205.755
KW463, ACETONE
YELLOW SOLID
100MHz
OH
CN
$O_2N$
Table 2, entry 6
f1 (ppm)

KW489H, CDCl3
400MHz
YELLOW OIL

7.706, 7.655, 7.635, 7.612, 7.595, 7.592, 7.520, 7.501, 7.482, 7.259
5.099, 5.086, 5.074, 5.060
3.631, 3.620
2.825, 2.812, 2.783, 2.772, 2.756, 2.731, 2.714

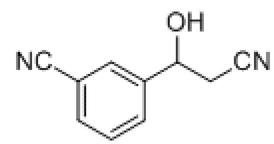
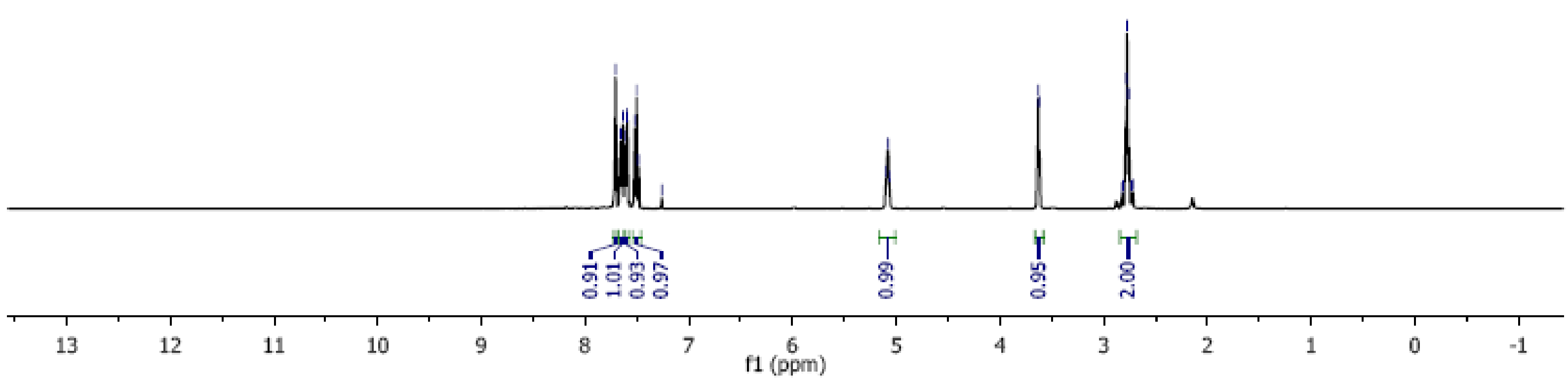



Table 2, entry 7



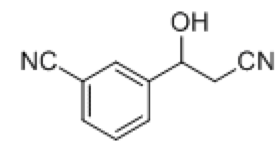
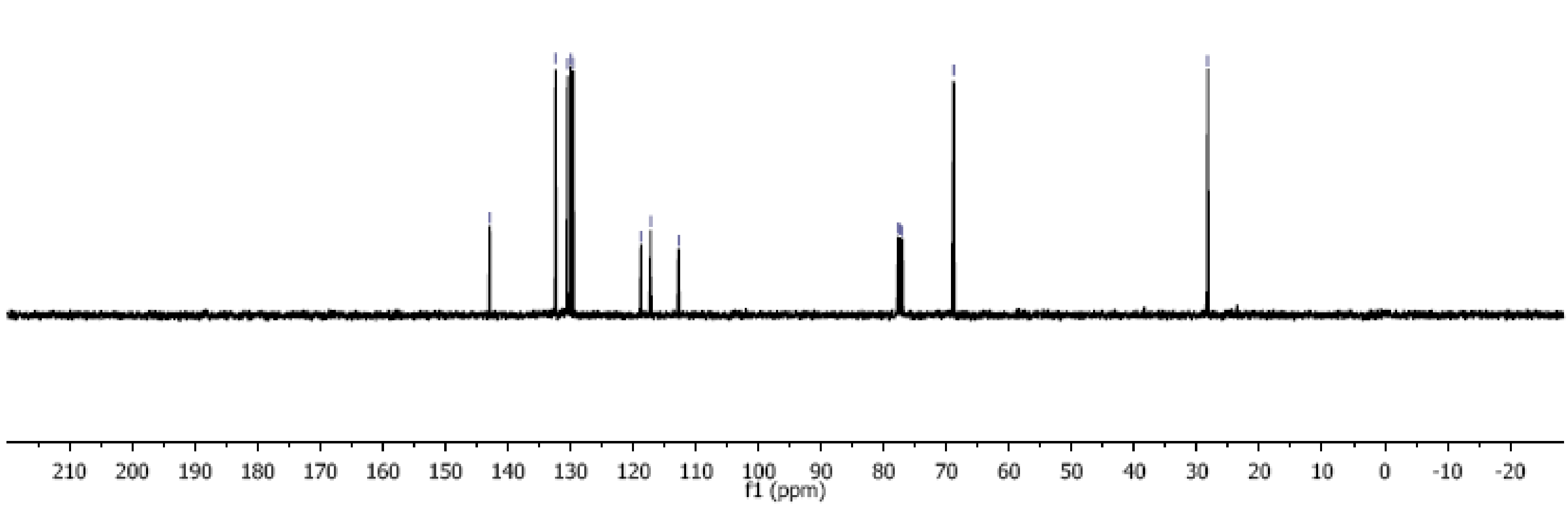


Table 2, entry 7



# Manual Peak Matching Report
# For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 172.06366 | 172.06395 | 168.98882 | 1.7 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

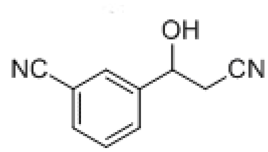



Table 2, entry 7

```
SPEC:  fin083791.dat (22-JUL-08 11:05:49)                                    Scans:   1 > 74
Samp:  KW489
Comm:  SP 70 eV EI
Oper:  kh                          Study:  MS services                        Client:  Kuldup
Base:  131.91                      Masses:  35.01 > 650.00                    #Peaks:  651
Peak:  1000.0 mmu                  Intensity:   16777215                      RIC:  137516812
Scan 73 @ 1.65 min (EI +Q1MS LMR UP LR)                                                1.7E+07
```

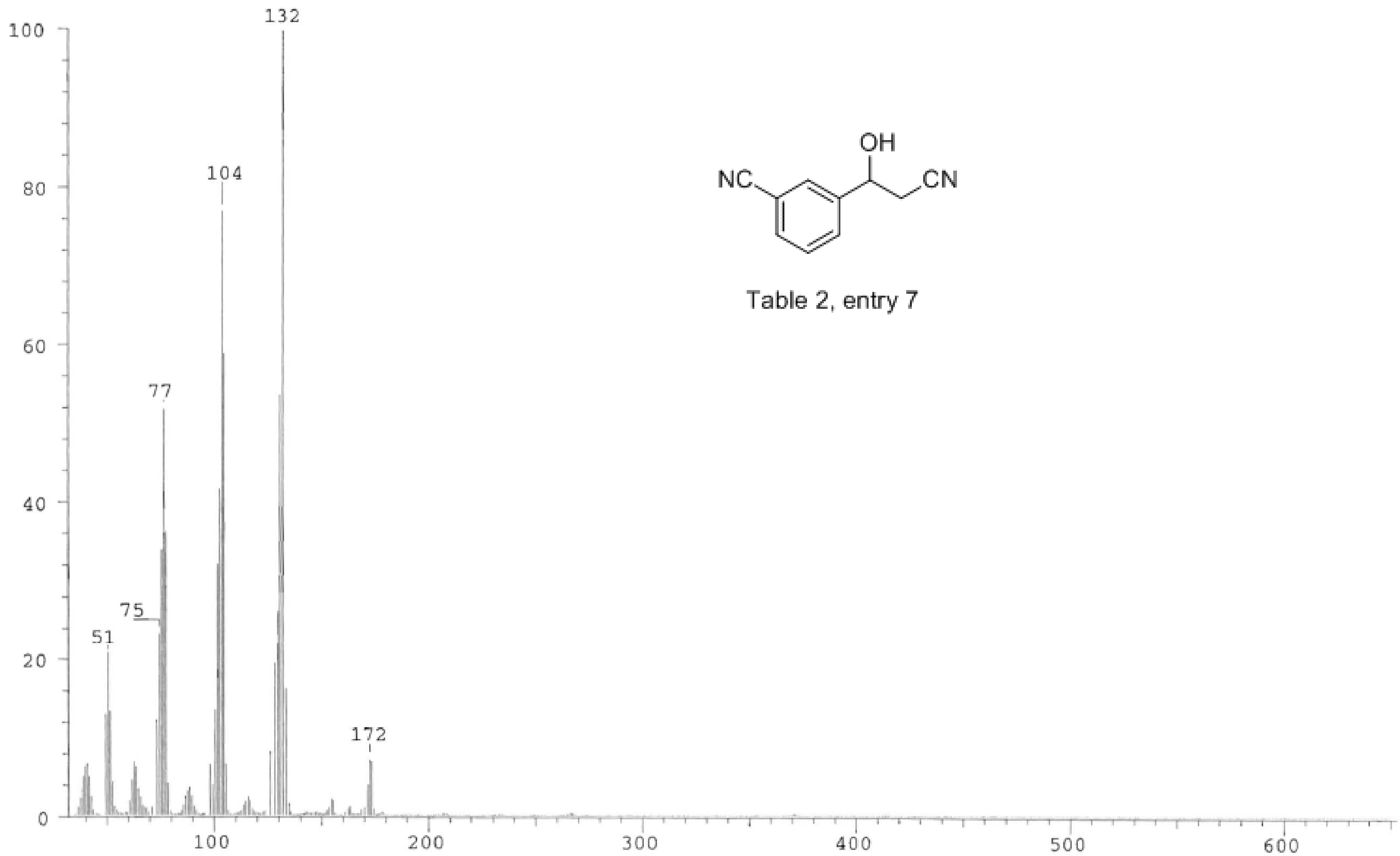
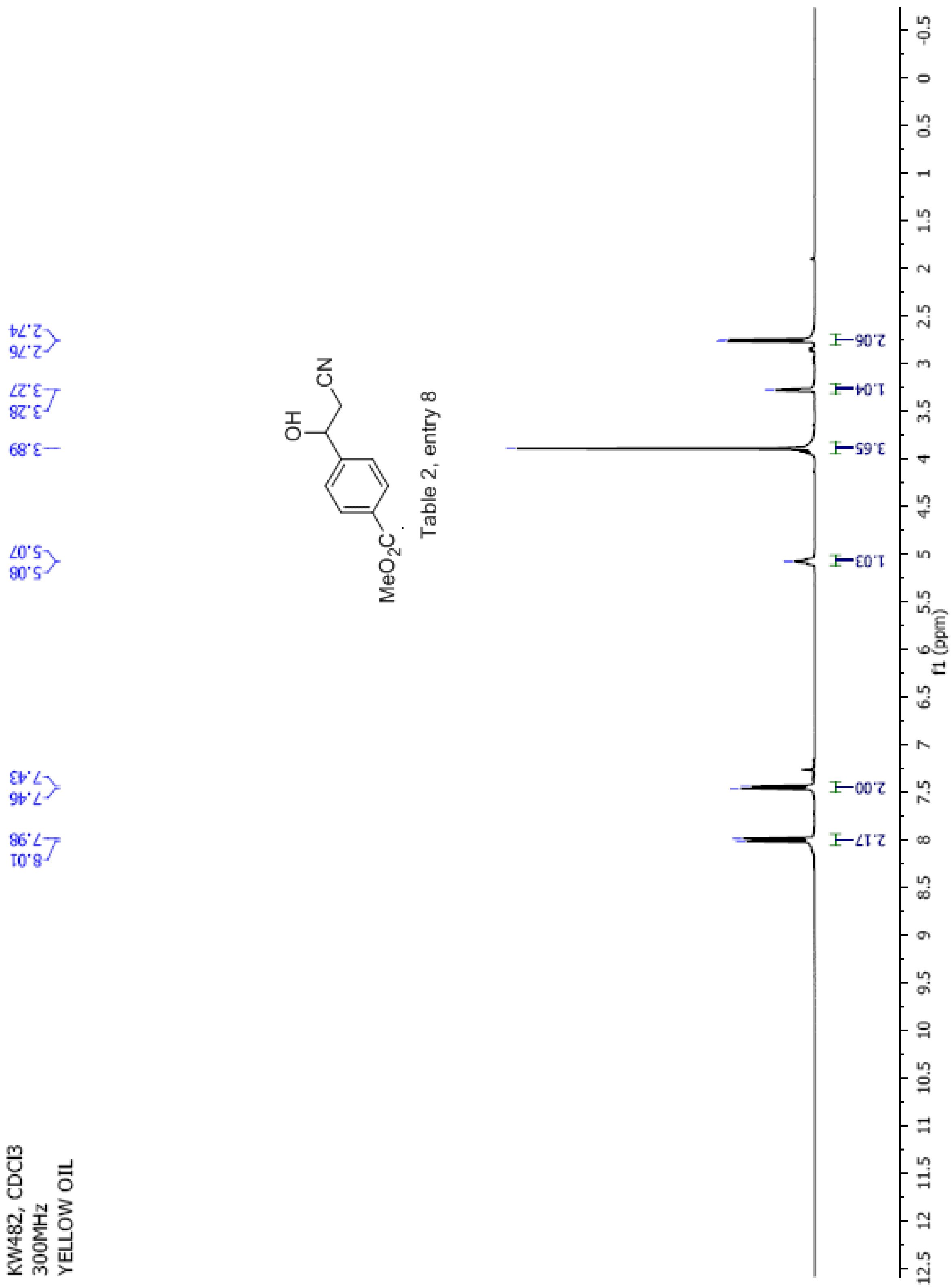
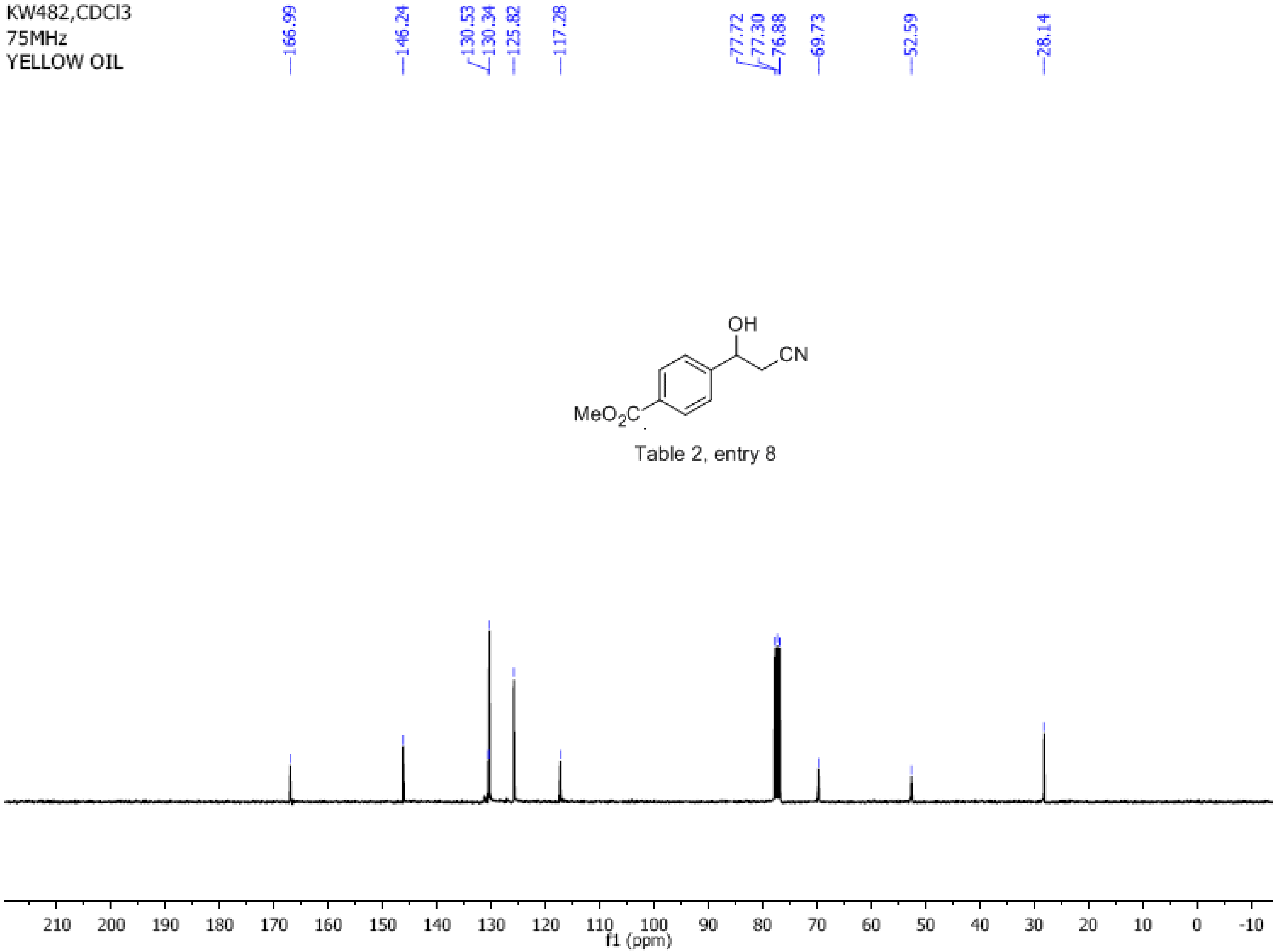



Table 2, entry 7

Date: Tue Jul 22 11:07:55 2008    ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98


KW482, CDCl3
300MHz
YELLOW OIL
Table 2, entry 8
f1 (ppm)


KW482,CDCl3
75MHz
YELLOW OIL
166.99
146.24
130.53
130.34
125.82
117.28
77.72
77.30
76.88
69.73
52.59
28.14
OH
CN
MeO2C
Table 2, entry 8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

Manual Peak Matching Report
For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 205.07389 | 205.07416 | 180.98882 | 1.3 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

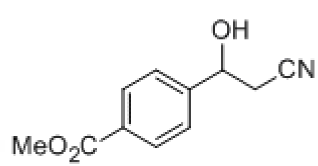


Table 2, entry 8

SPEC: fin084361.dat (06-APR-09 14:45:04)
Samp: KW482
Comm: DP/EI
Oper: kh
Base: 164.65
Peak: 1000.0 mmu

Study: ms services
Masses: 35.01 > 650.00
Intensity: 6828005

Scans: 1 > 84
Client: Kuldeep
#Peaks: 637
RIC: 18425668

Scan 60 @ 0.94 min (EI +Q1MS LMR UP LR)

6.8E+06

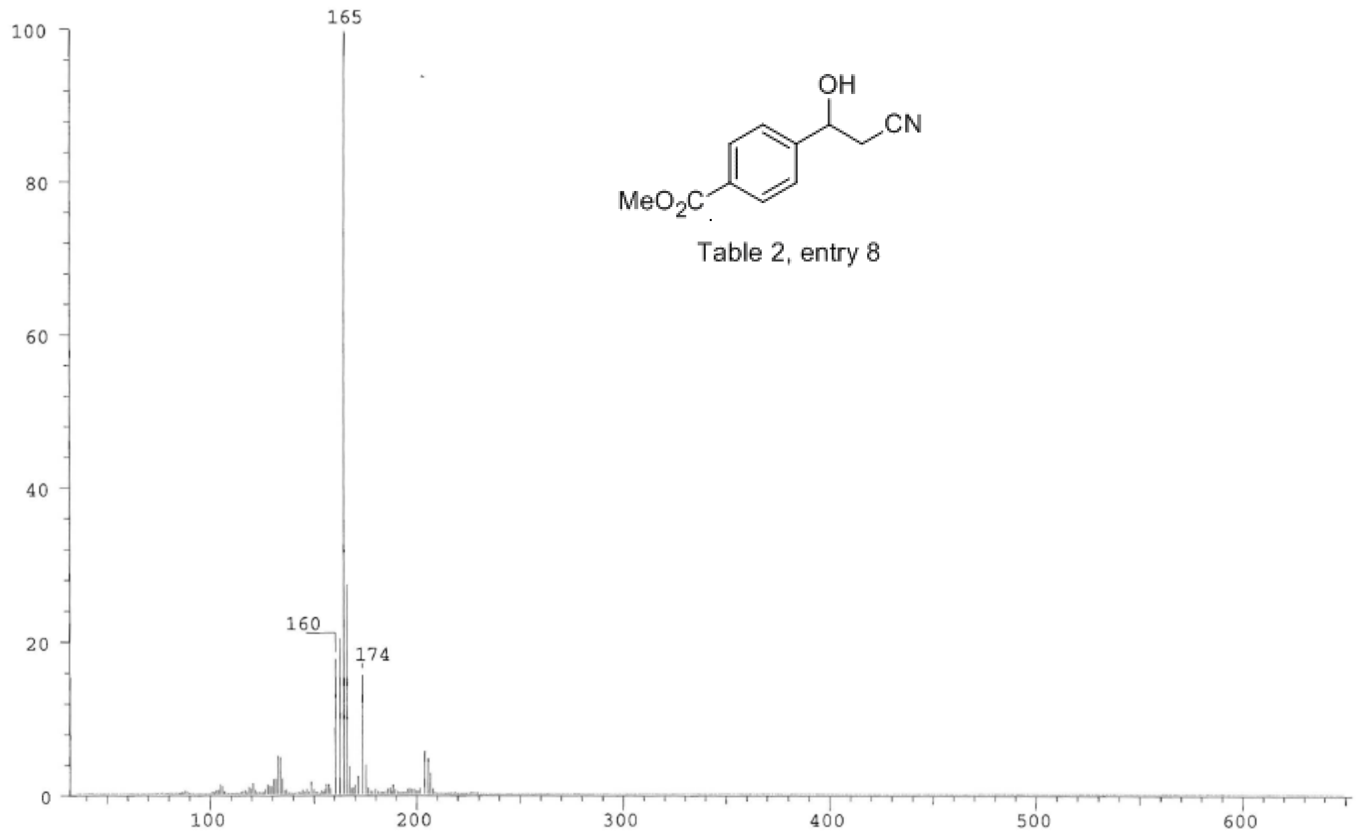



Table 2, entry 8

Date: Mon Apr 6 14:47:58 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

KW494, CDCl3
400MHz
YELLOW OIL

7.405 7.401 7.384 7.358 7.355 7.338 7.319 7.303 7.285 6.719 6.679 6.238 6.215 6.199 4.628 4.617 4.605 2.721 2.708 2.680 2.674 2.666 2.650 2.624 2.608

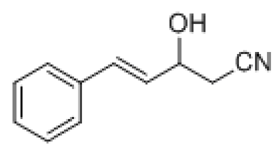
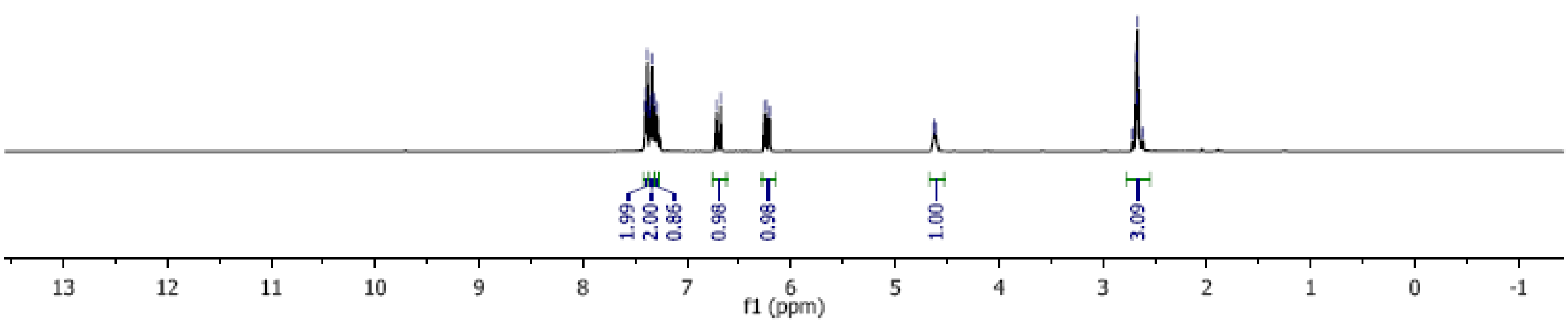



Table 2, entry 9



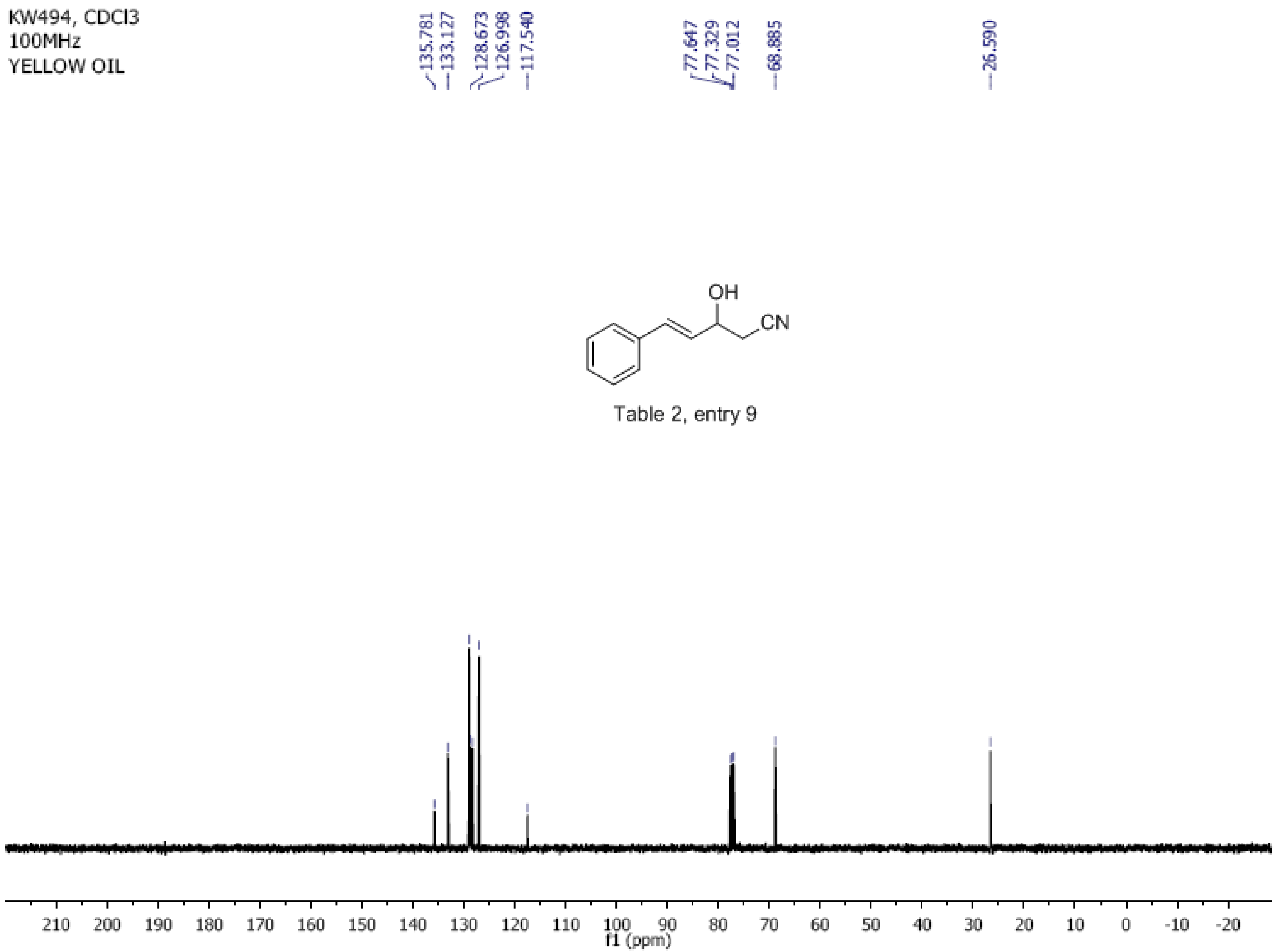



Table 2, entry 9

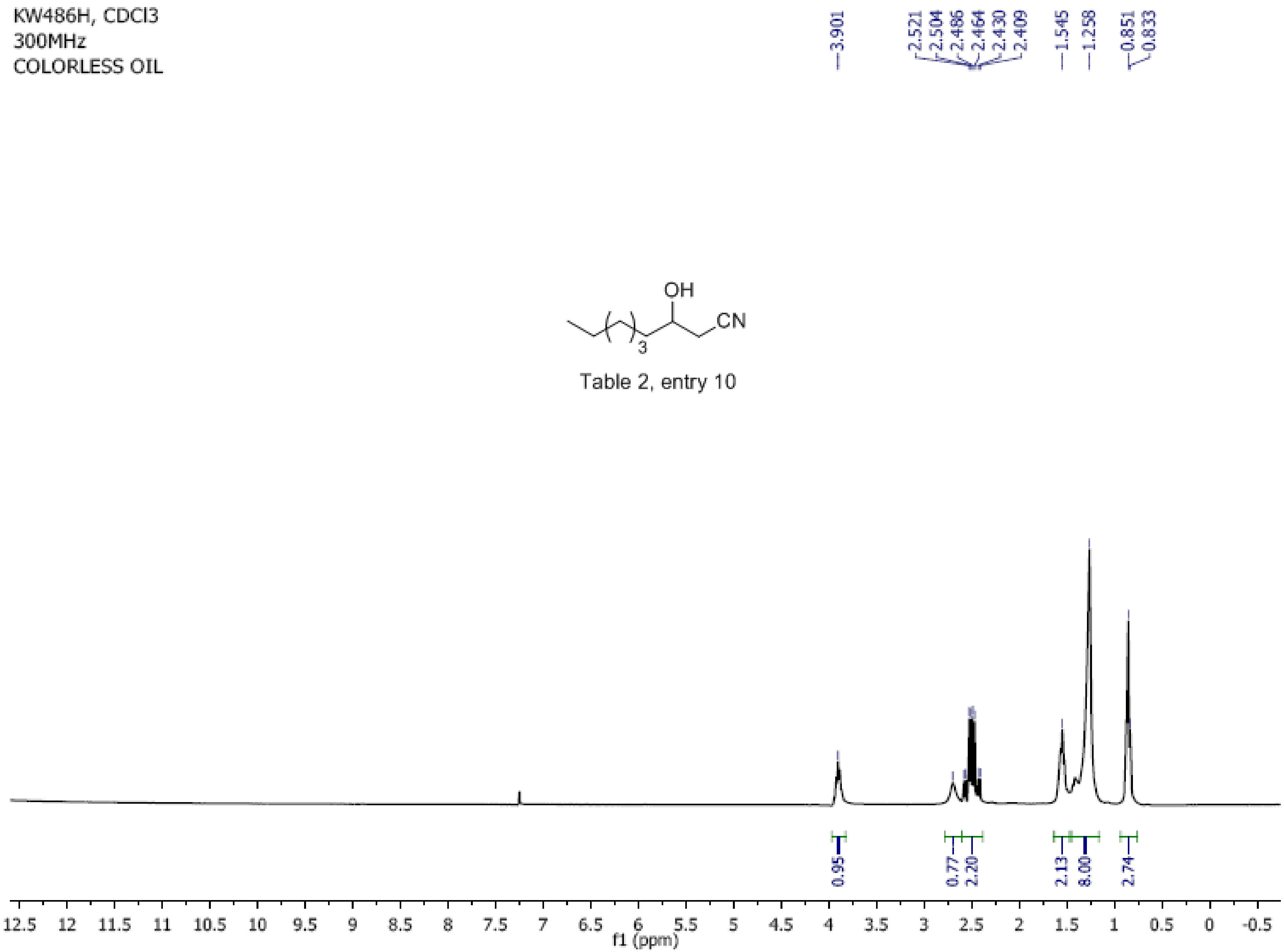
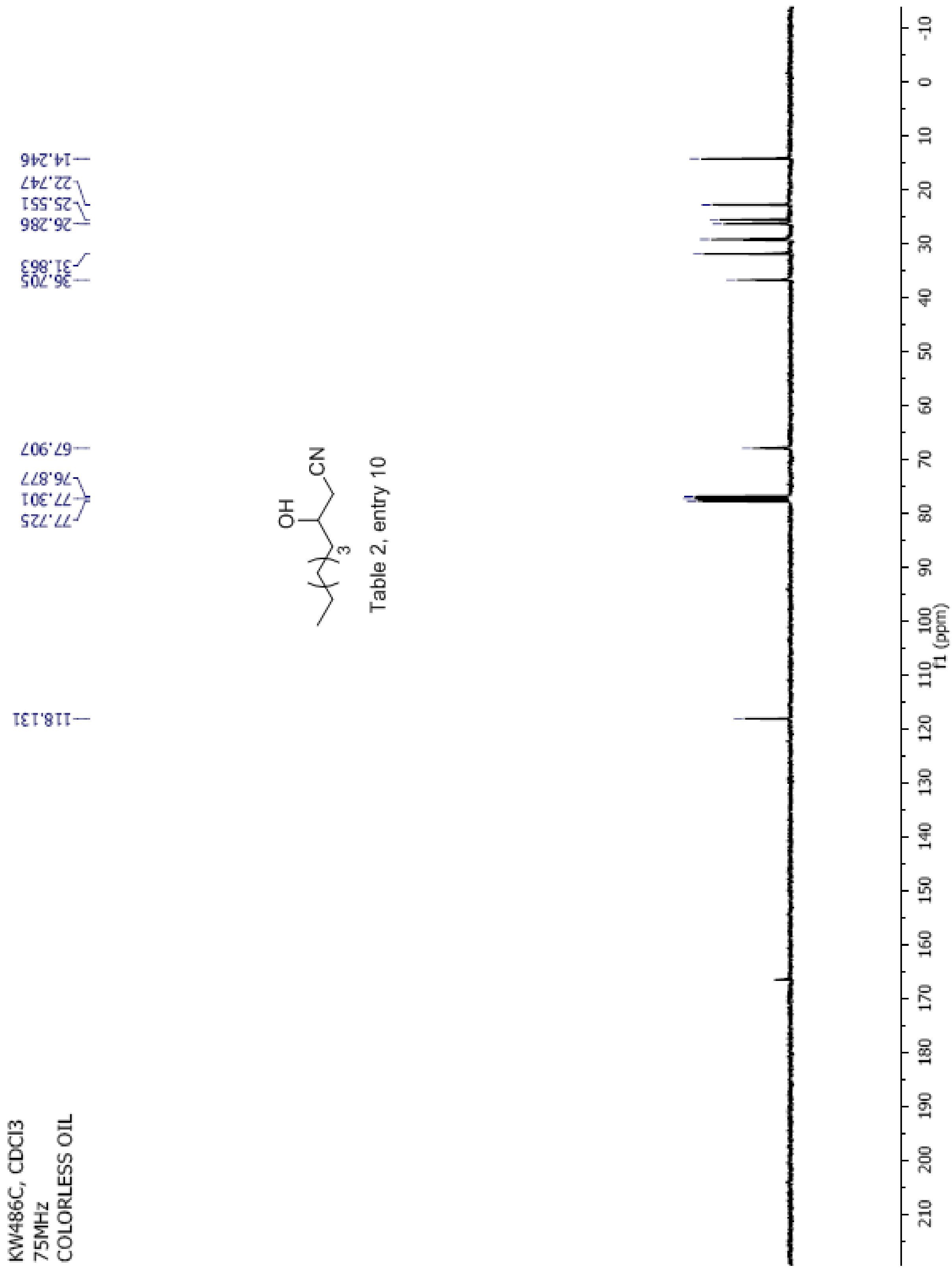
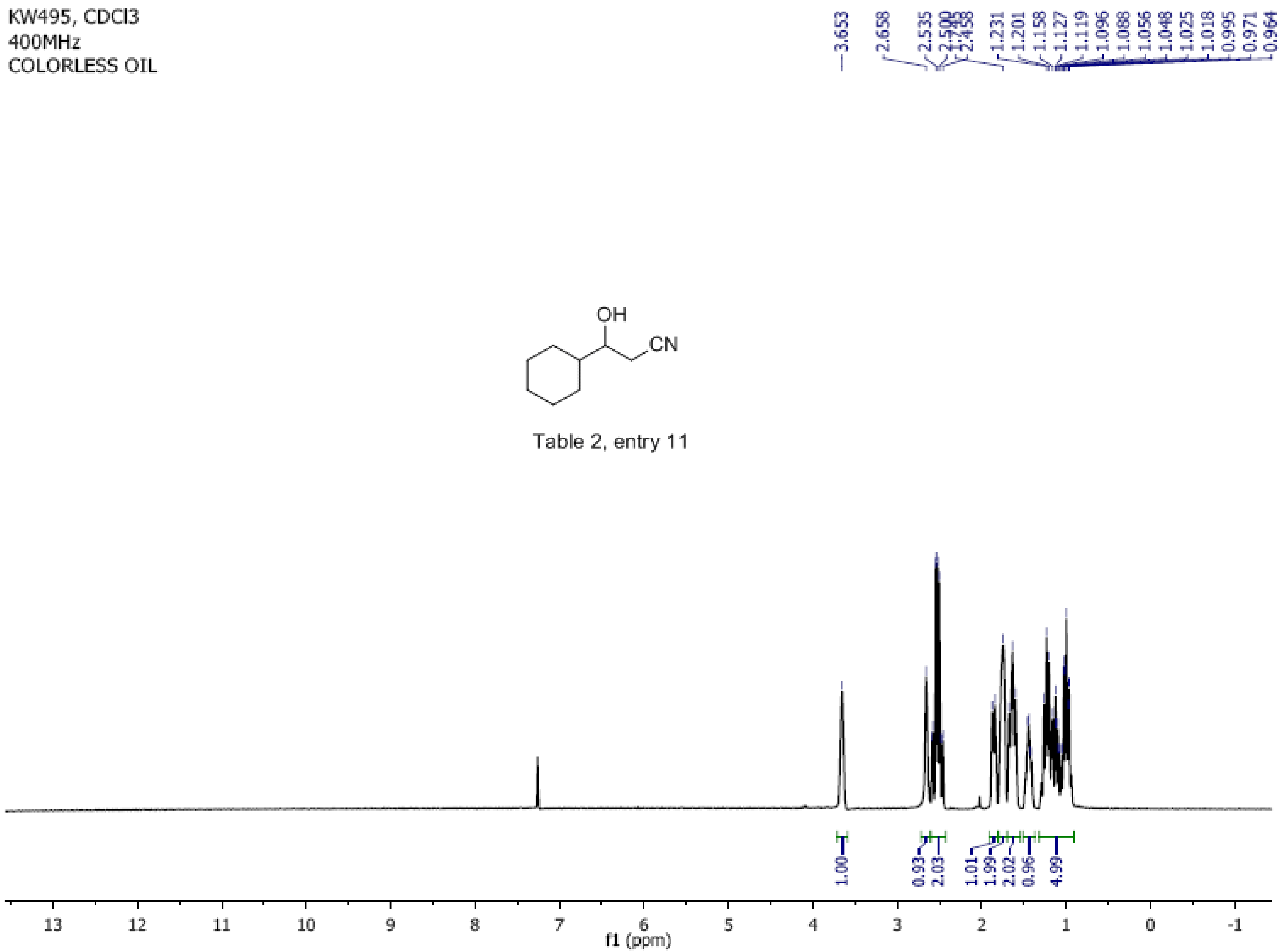



Table 2, entry 10


KW486C, CDCl3
75MHz
COLORLESS OIL
118.131
77.725
77.301
76.877
67.907
36.705
31.863
26.286
25.551
22.747
14.246
OH
CN
3
Table 2, entry 10
f1 (ppm)


OH
CN
Table 2, entry 11
KW495, CDCl3
400MHz
COLORLESS OIL
3.653
2.658
2.535
2.500
2.458
1.231
1.201
1.158
1.127
1.119
1.096
1.088
1.056
1.048
1.025
1.018
0.995
0.971
0.964
1.00
0.93
2.03
1.01
1.99
2.02
0.96
4.99
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

KW495, CDCl3
100MHz
COLORLESS OIL

118.612
77.654
77.336
77.018
72.080
43.032
29.118
28.099
26.345
26.094
25.927
23.818

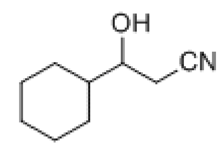
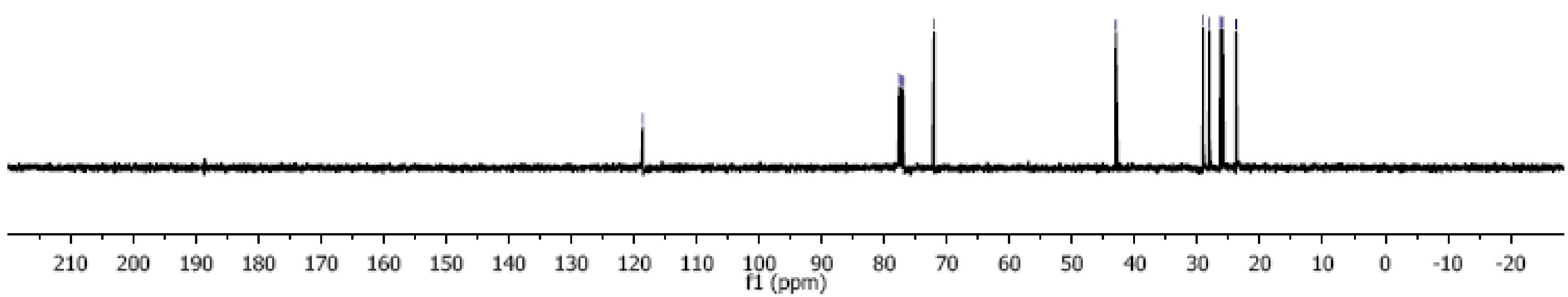
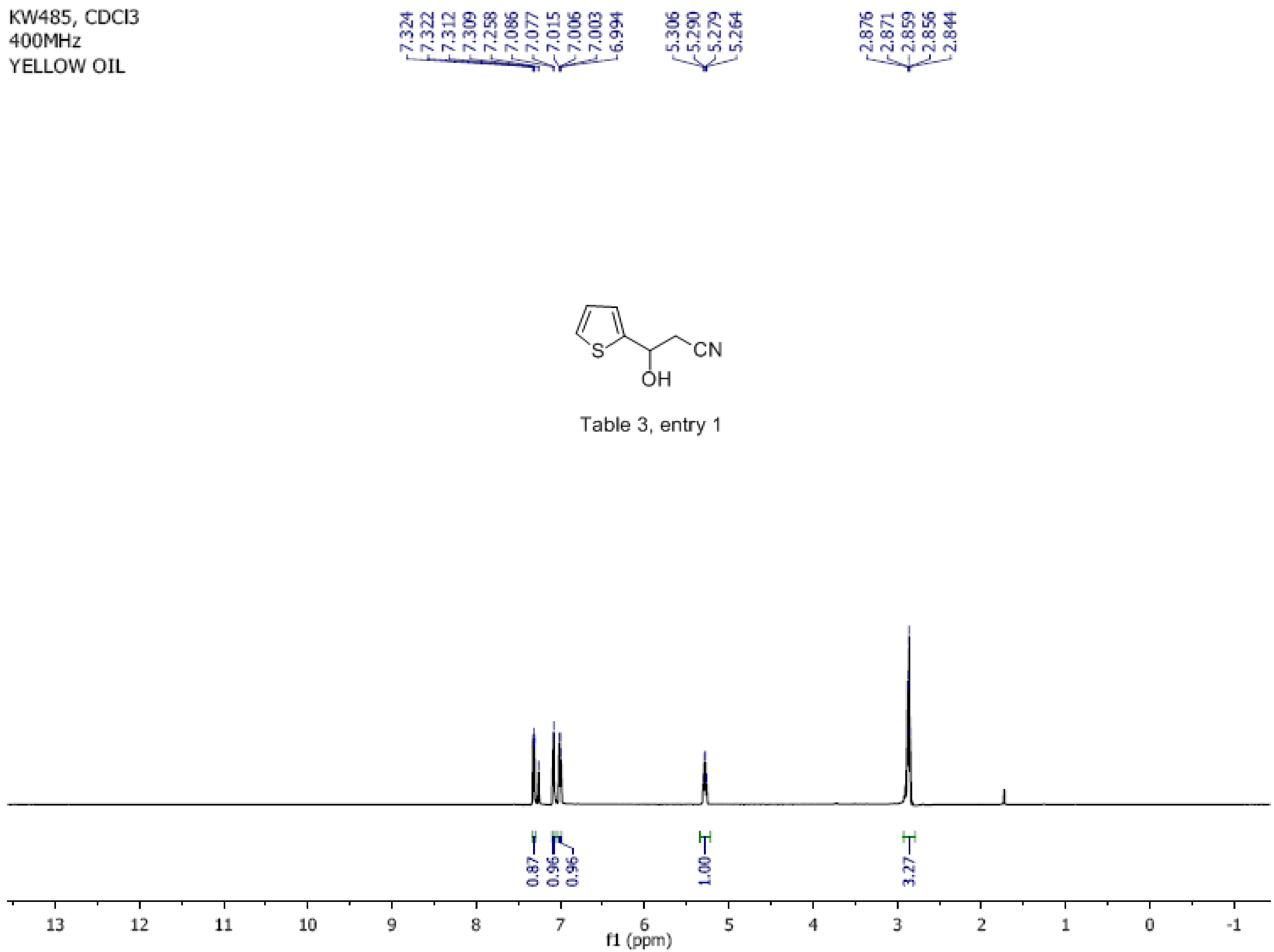



Table 2, entry 11




KW485, CDCl3
400MHz
YELLOW OIL
7.324
7.322
7.312
7.309
7.258
7.086
7.077
7.015
7.006
7.003
6.994
5.306
5.290
5.279
5.264
2.876
2.871
2.859
2.856
2.844
S
CN
OH
Table 3, entry 1
0.87
0.96
0.96
1.00
3.27
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

KW485, CDCl3
100 MHZ
YELLOW OIL

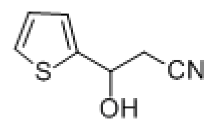
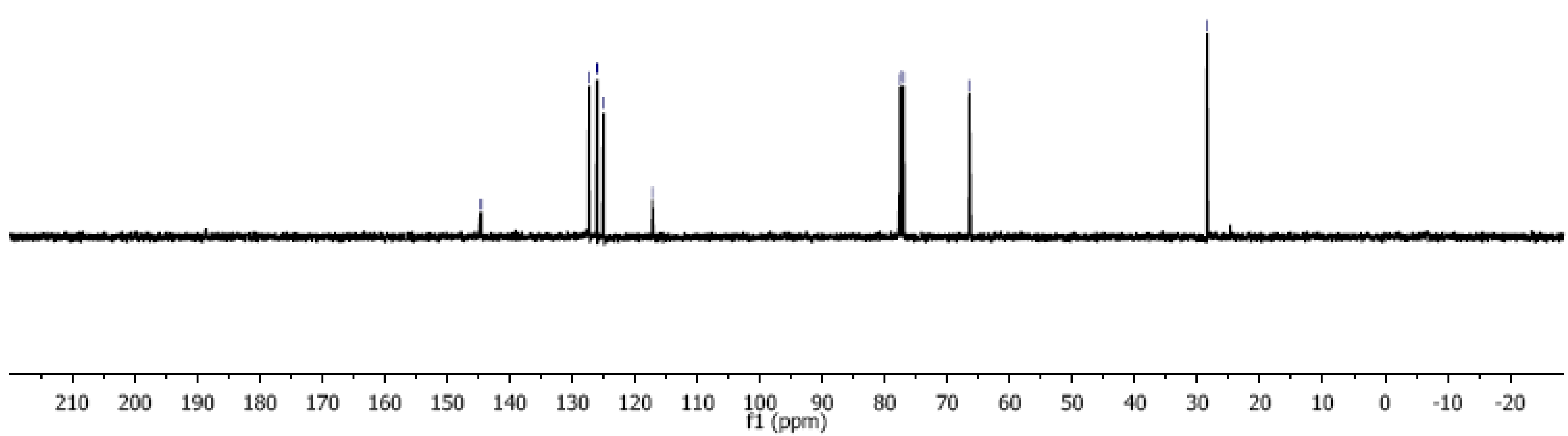
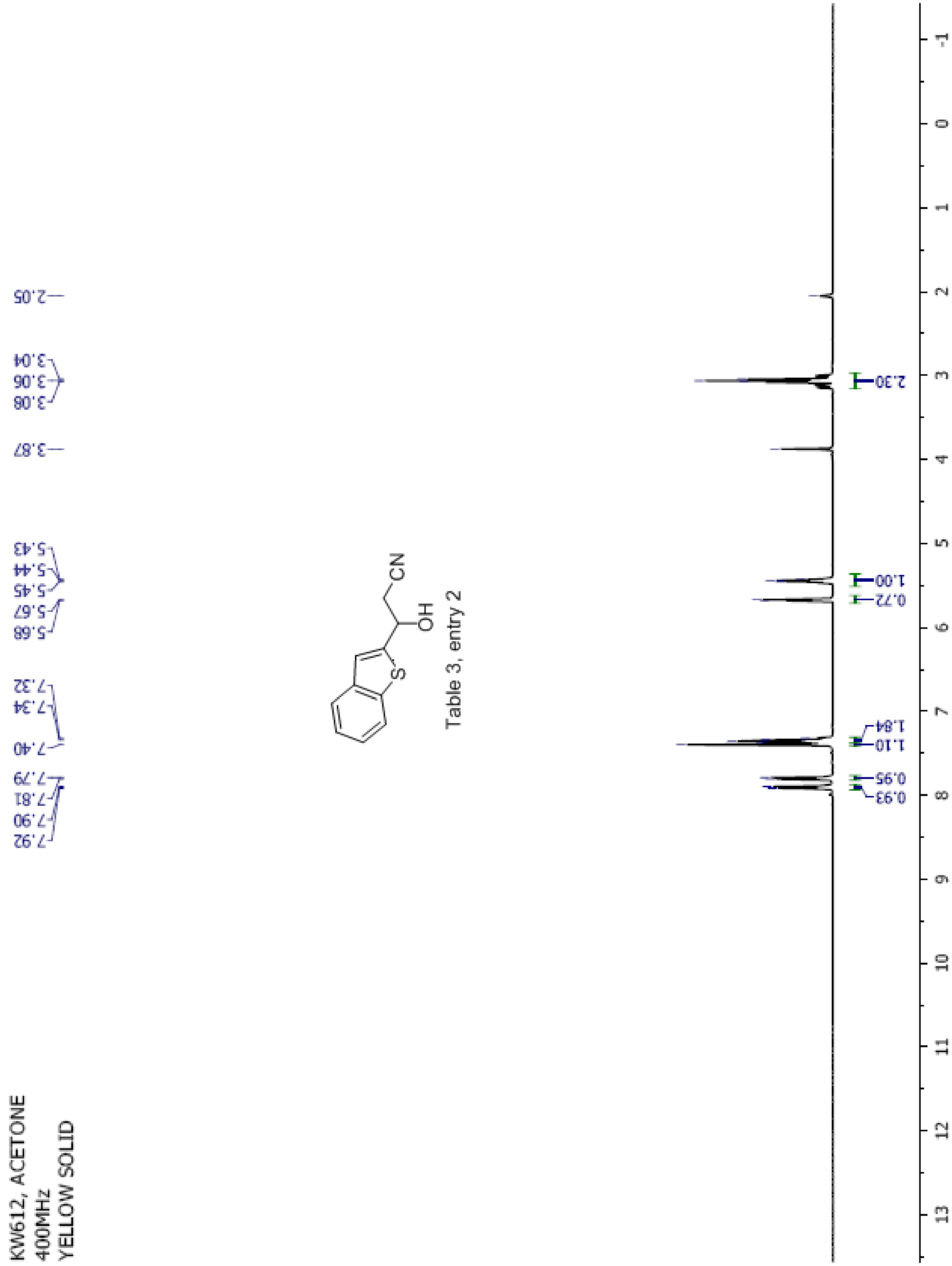
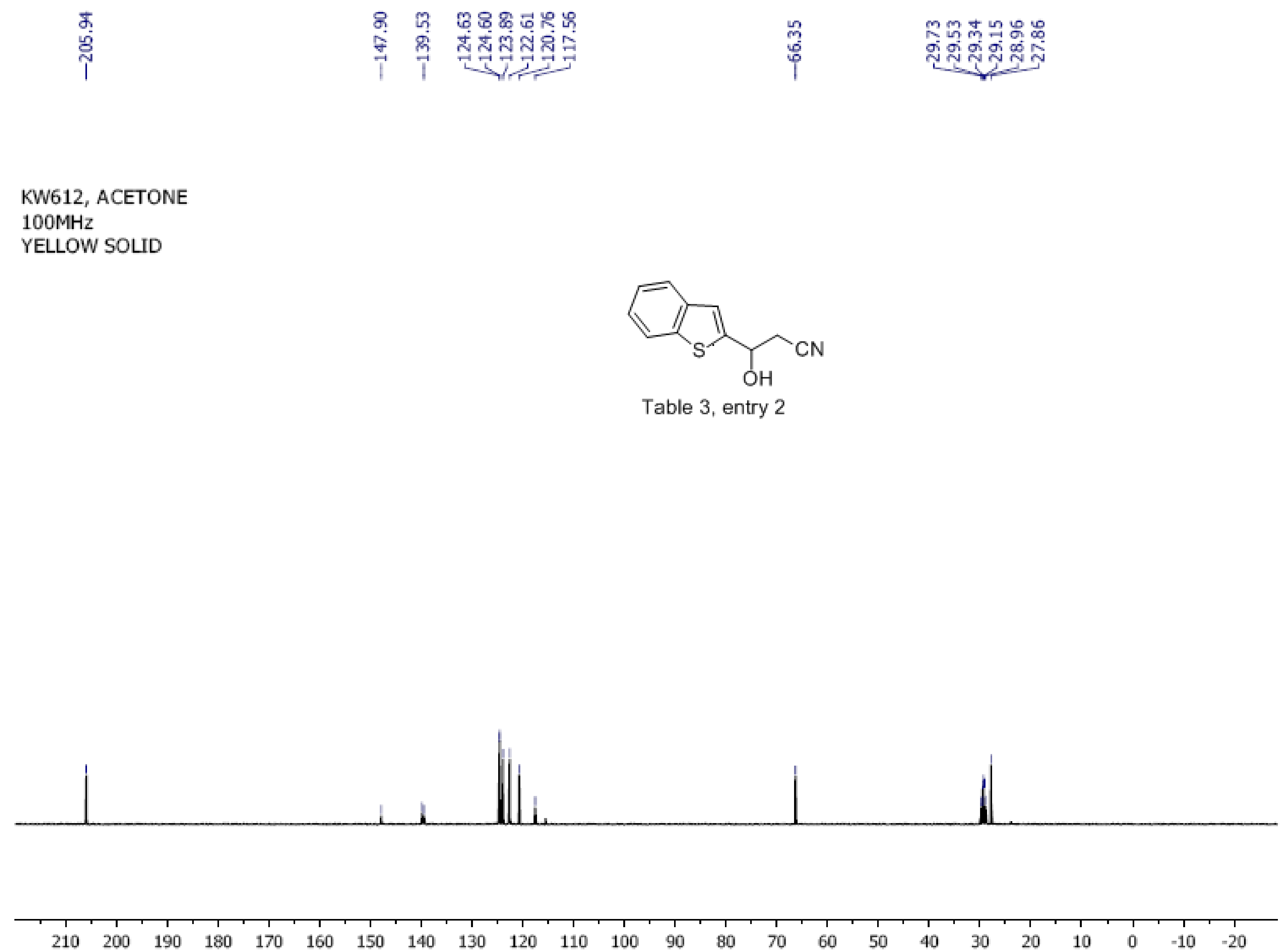


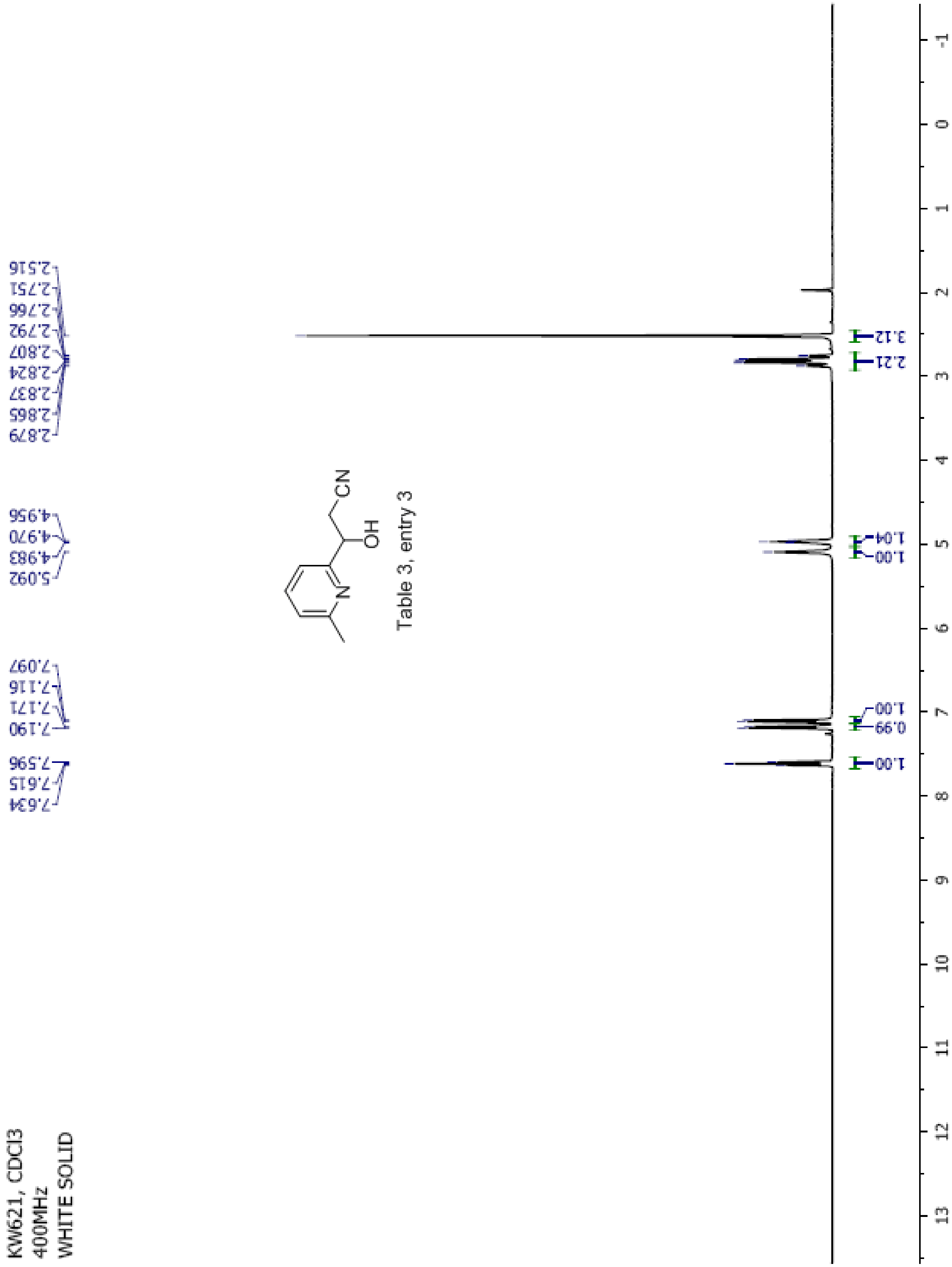

Table 3, entry 1

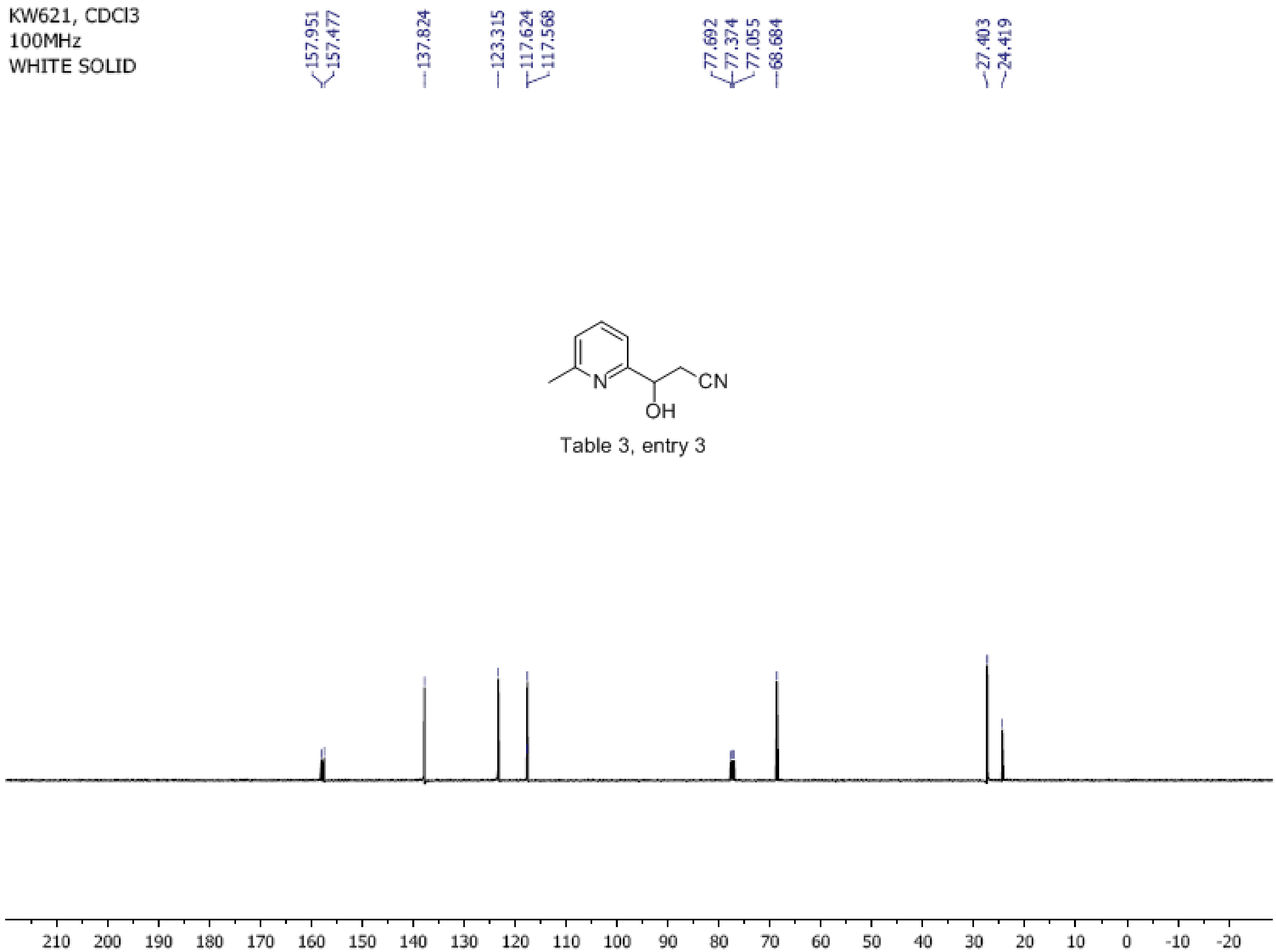



S39


KW612, ACETONE
400MHz
YELLOW SOLID
7.92
7.90
7.81
7.79
7.40
7.34
7.32
5.68
5.67
5.45
5.44
5.43
3.87
3.08
3.06
3.04
2.05
CN
OH
S
Table 3, entry 2
0.93
0.95
1.10
1.84
0.72
1.00
2.30
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1


205.94
147.90
139.53
124.63
124.60
123.89
122.61
120.76
117.56
66.35
29.73
29.53
29.34
29.15
28.96
27.86
KW612, ACETONE
100MHz
YELLOW SOLID
S
CN
OH
Table 3, entry 2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20


KW621, CDCl3
400MHz
WHITE SOLID
Table 3, entry 3
OH
CN
N
7.634
7.615
7.596
7.190
7.171
7.116
7.097
5.092
4.983
4.970
4.956
2.879
2.865
2.837
2.824
2.807
2.792
2.766
2.751
2.516
1.00
0.99
1.00
1.00
1.04
2.21
3.12
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1


KW621, CDCl3
100MHz
WHITE SOLID
157.951
157.477
137.824
123.315
117.624
117.568
77.692
77.374
77.055
68.684
27.403
24.419
CN
N
OH
Table 3, entry 3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

Manual Peak Matching Report
For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 162.07931 | 162.07961 | 130.99201 | 1.8 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

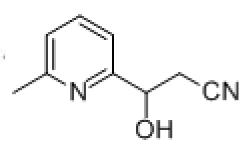


Table 3, entry 3

SPEC: fin063649.dat (19-MAY-08 10:47:44)
Samp: KW621
Comm: 70 eV EI
Oper: kh
Base: 64.82
Peak: 1000.0 mmu
Scan 1 @ 0.16 min (EI +Q1MS LMR UP LR)

Study: Service
Masses: 35.01 > 650.00
Intensity: 237074

Scans: 1 > 13
Client: Kuldup
#Peaks: 665
RIC: 2630508

2.4E+05

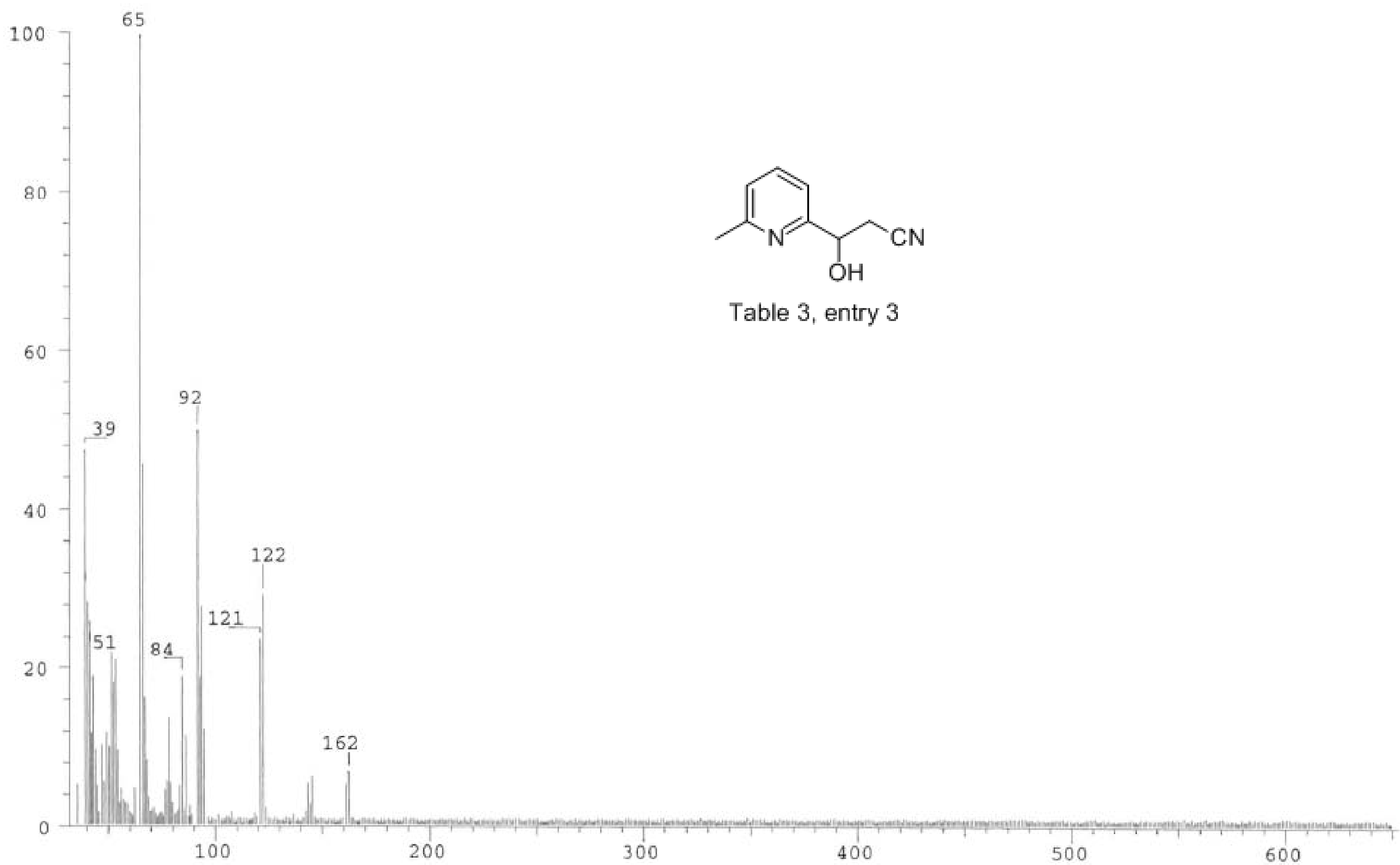



Table 3, entry 3

Date: Mon May 19 10:48:26 2008 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

KW632, CDCL3
400MHz
YELLOW OIL

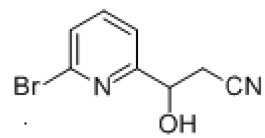
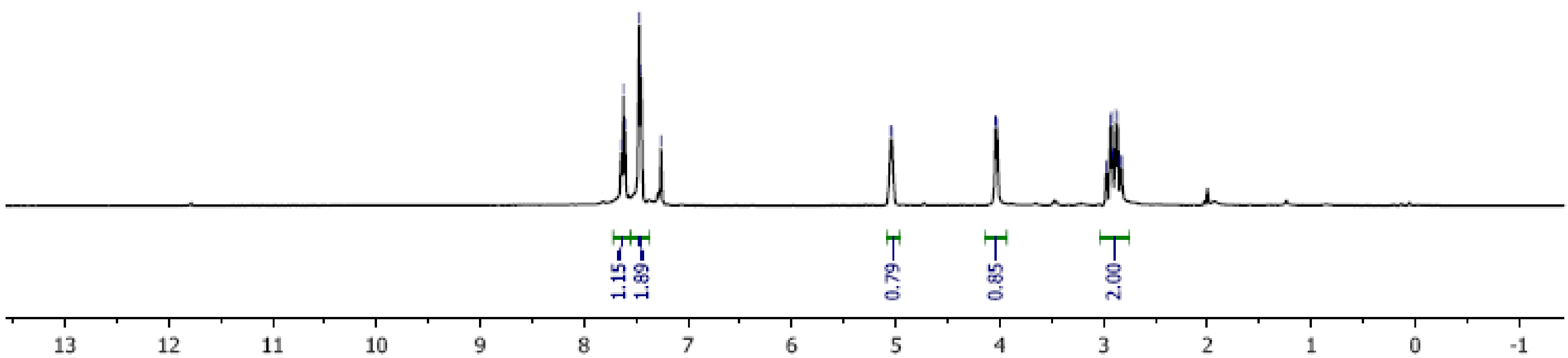



Table 3, entry 4



KW632, CDCL3
100MHz
YELLOW OIL

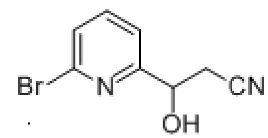
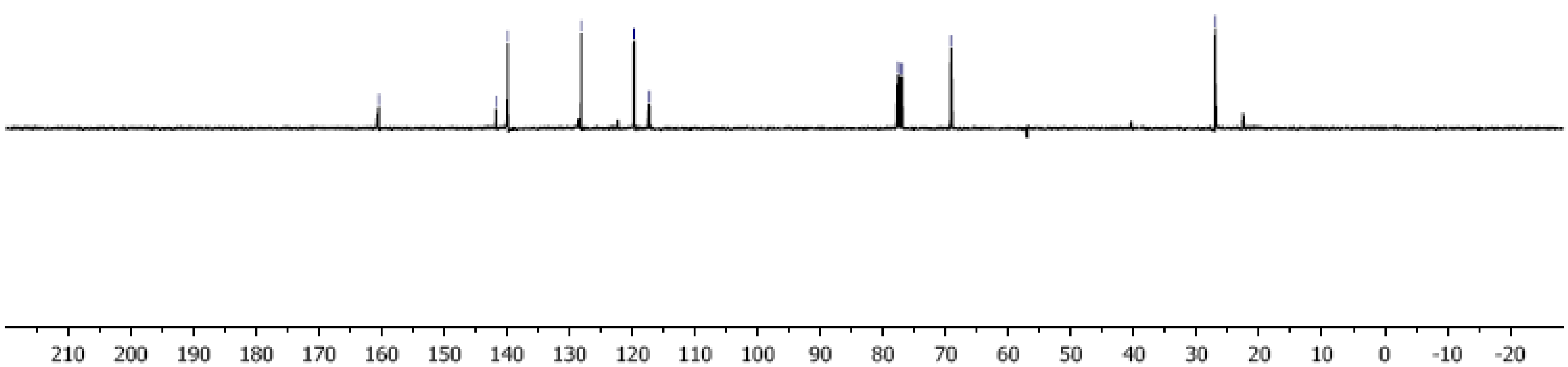




Table 3, entry 4



# Manual Peak Matching Report
# For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 225.97417 | 225.97456 | 218.98562 | 1.7 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

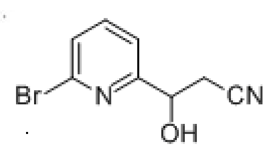



Table 3, entry 4

SPEC: fin083701.dat (11-JUN-08 10:57:21) Scans: 1 > 159
Samp: KW632
Comm: 70 eV EI
Oper: kh Study: MS services Client: Kuldup
Base: 186.27 Masses: 35.01 > 650.00 #Peaks: 663
Peak: 1000.0 mmu Intensity: 27336 RIC: 319370
Scan 13 @ 0.42 min (EI +Q1MS LMR UP LR) 2.7E+04

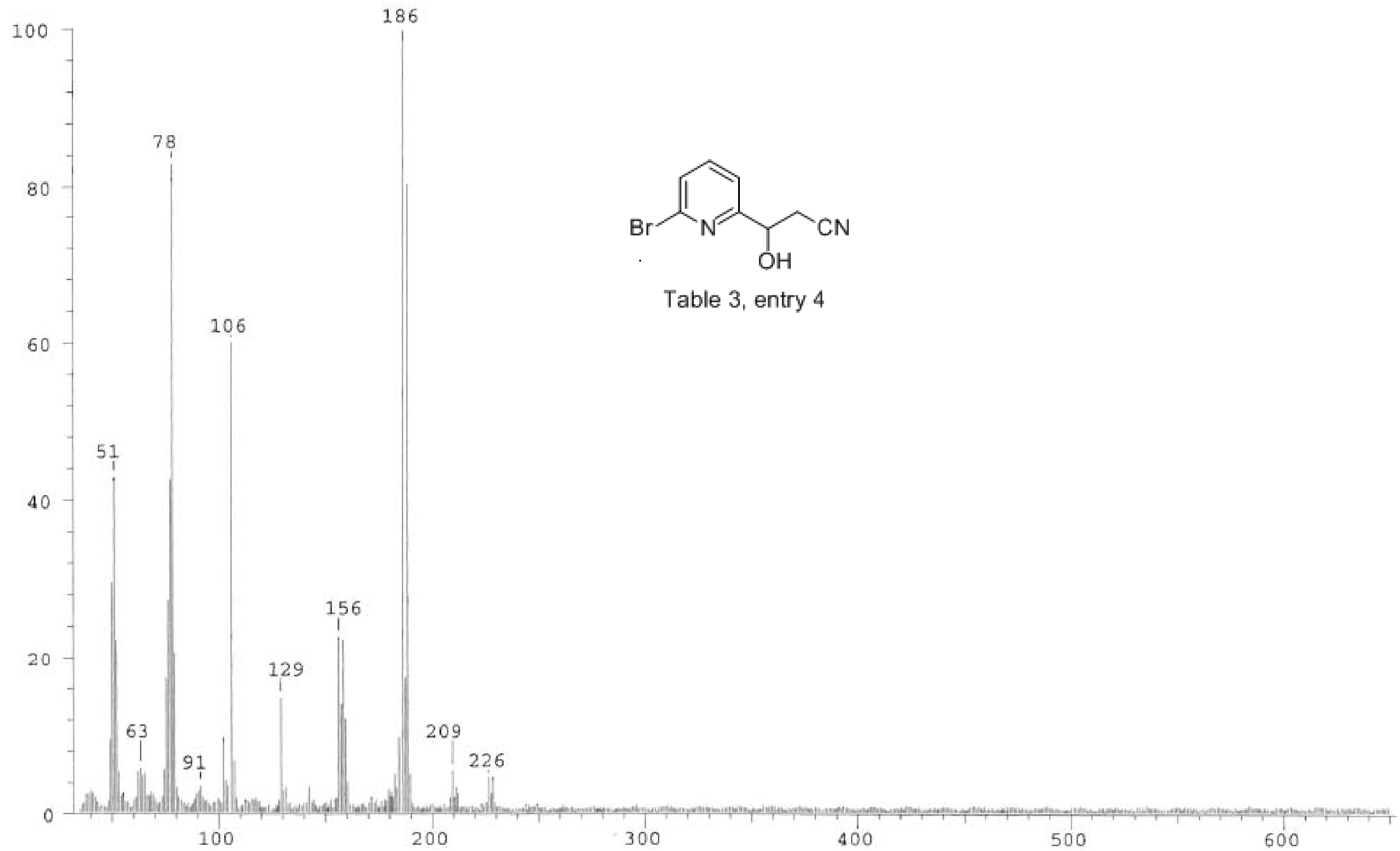



Table 3, entry 4

Date: Wed Jun 11 11:08:22 2008 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

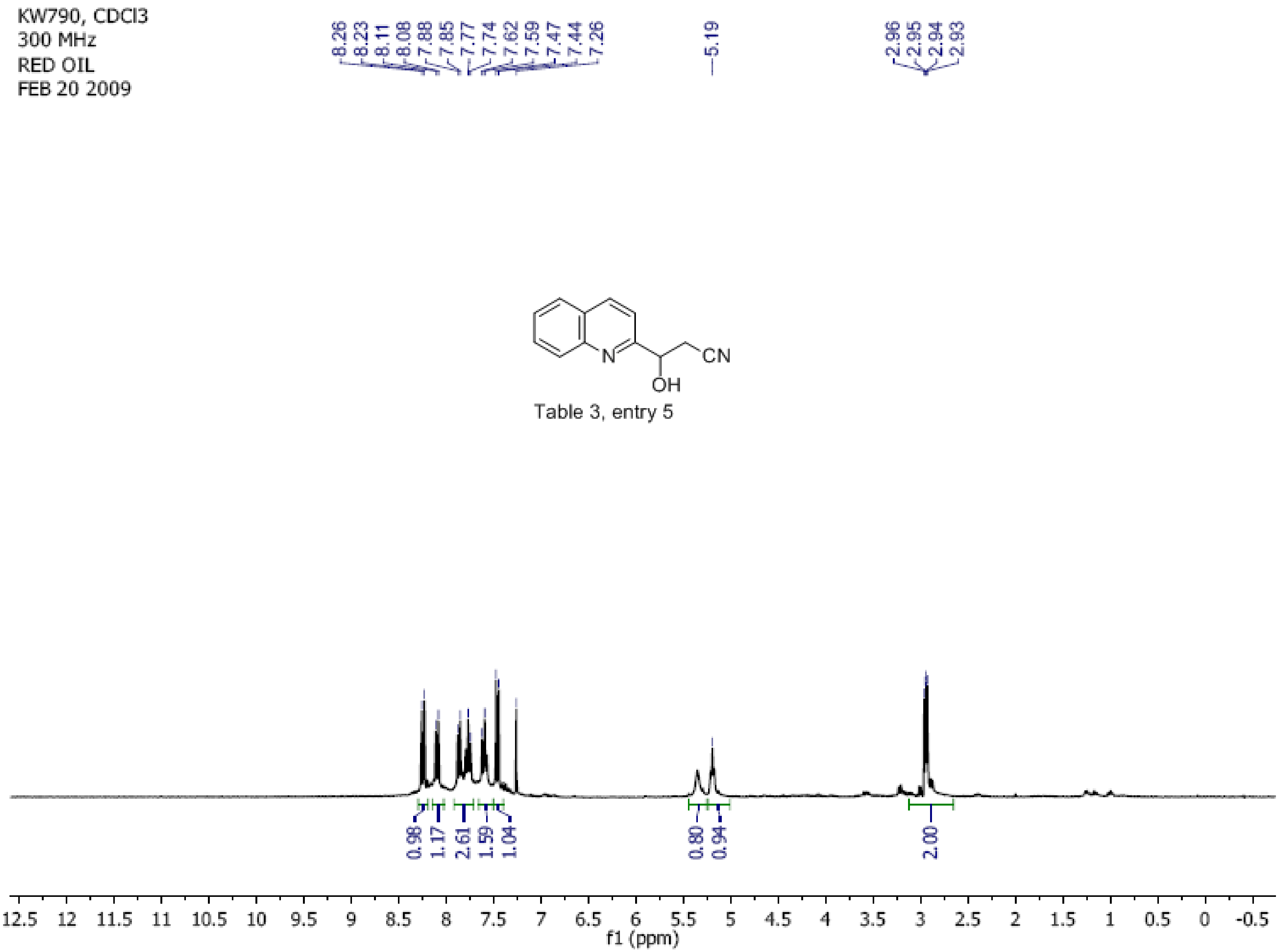
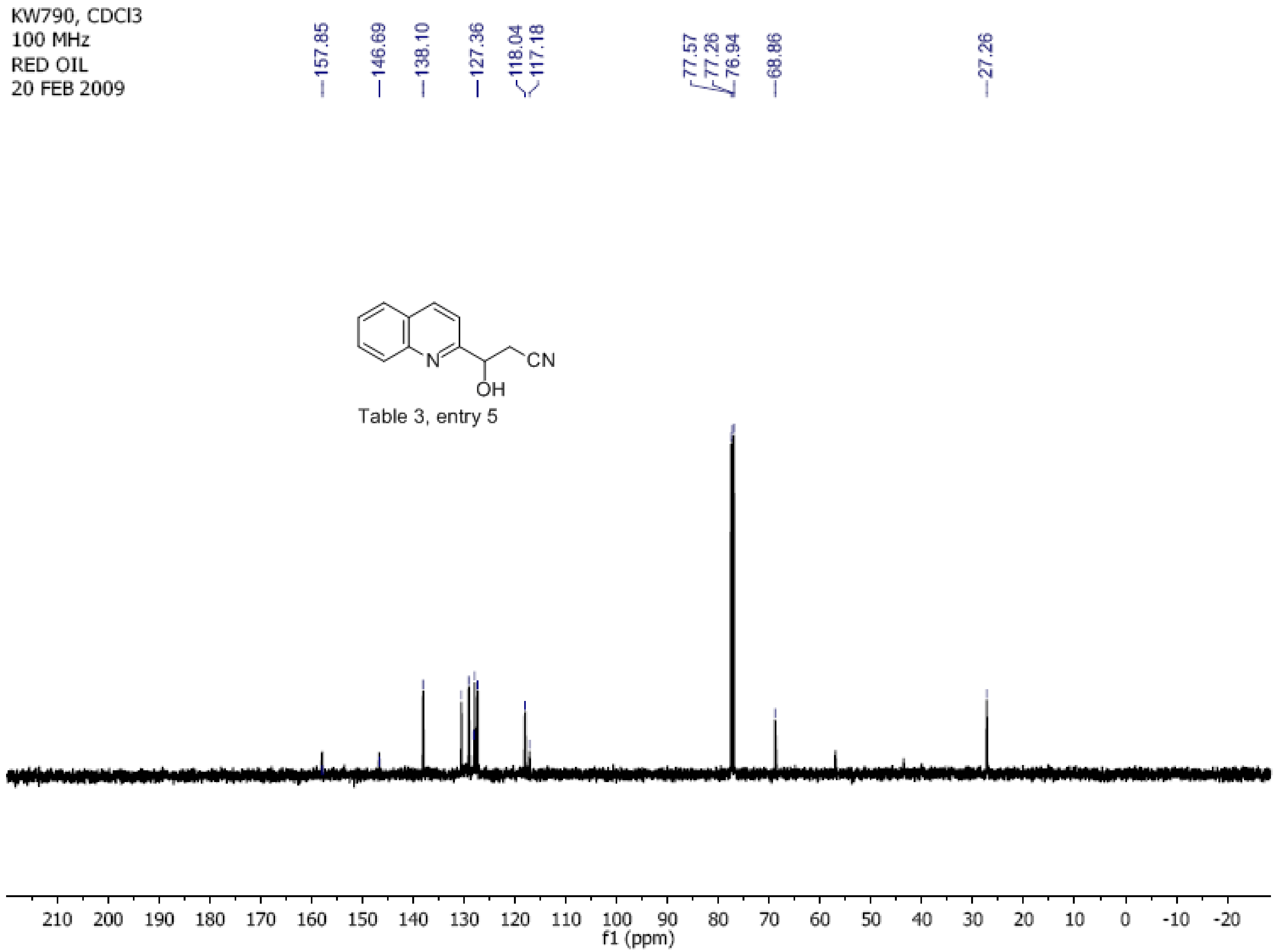



Table 3, entry 5


KW790, CDCl3
100 MHz
RED OIL
20 FEB 2009
157.85
146.69
138.10
127.36
118.04
117.18
77.57
77.26
76.94
68.86
27.26
CN
N
OH
Table 3, entry 5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

Manual Peak Matching Report
For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 198.07931 | 198.07973 | 180.98882 | 2.1 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

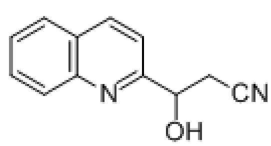


Table 3, entry 5

SPEC: fin084295.dat (06-MAR-09 11:04:28)
Samp: KW790
Comm: DP/EI
Oper: kh
Base: 196.33
Peak: 1000.0 mmu

Study: ms services
Masses: 35.01 > 650.00
Intensity: 137676

Scans: 1 > 32

Client: Kuldeep
#Peaks: 635
RIC: 1469464

Scan 29 @ 0.76 min (EI +Q1MS LMR UP LR)

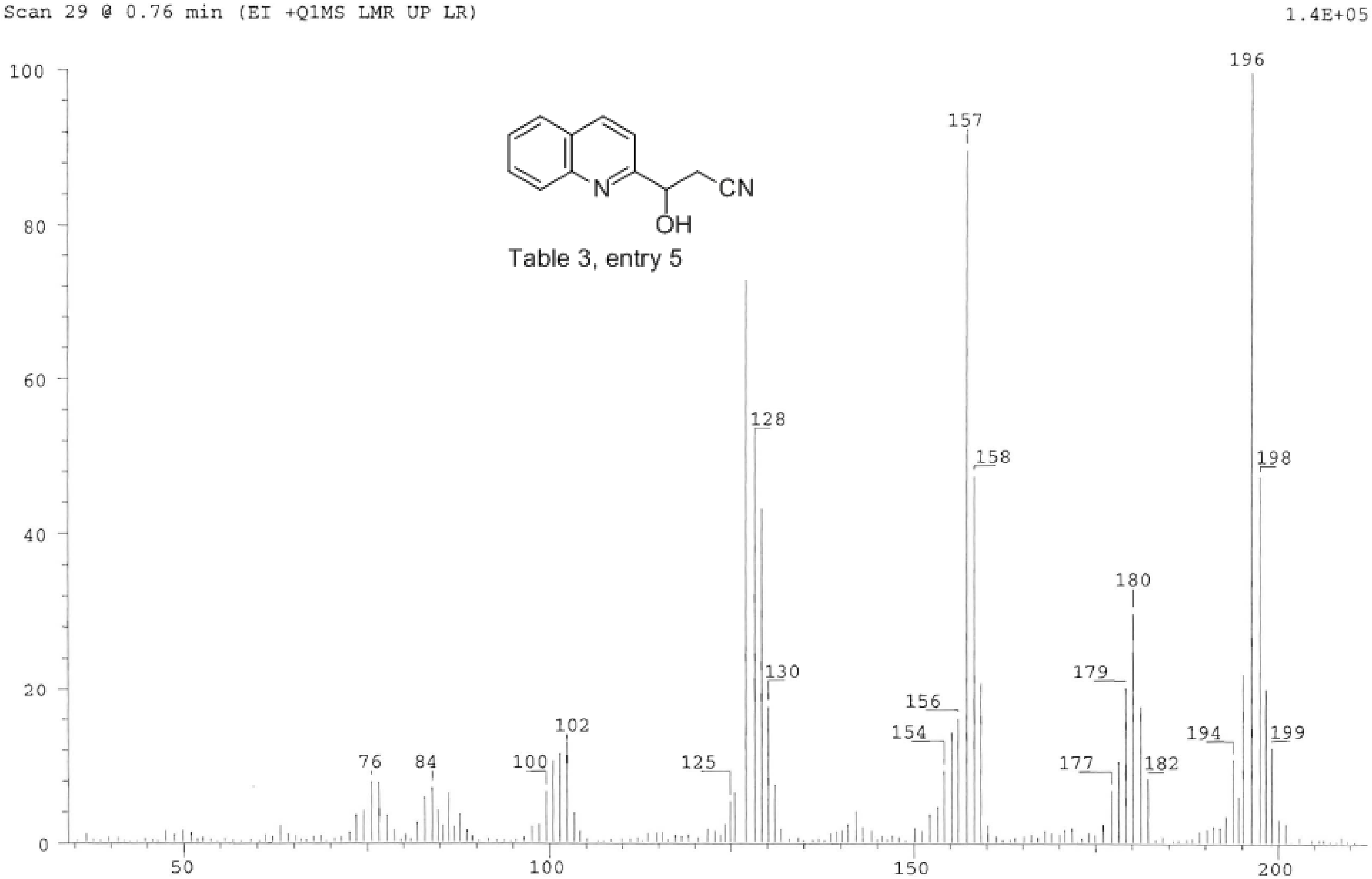
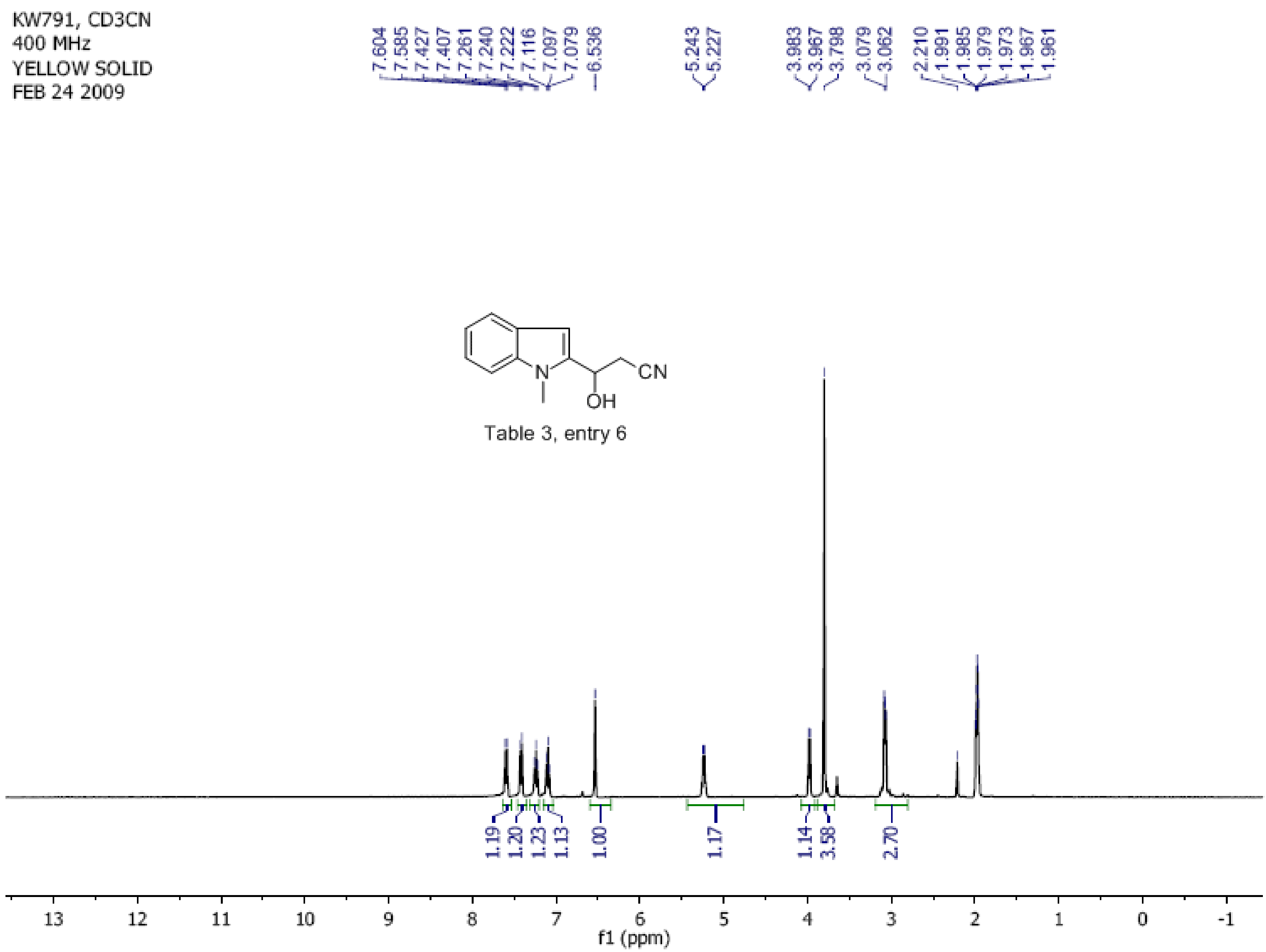
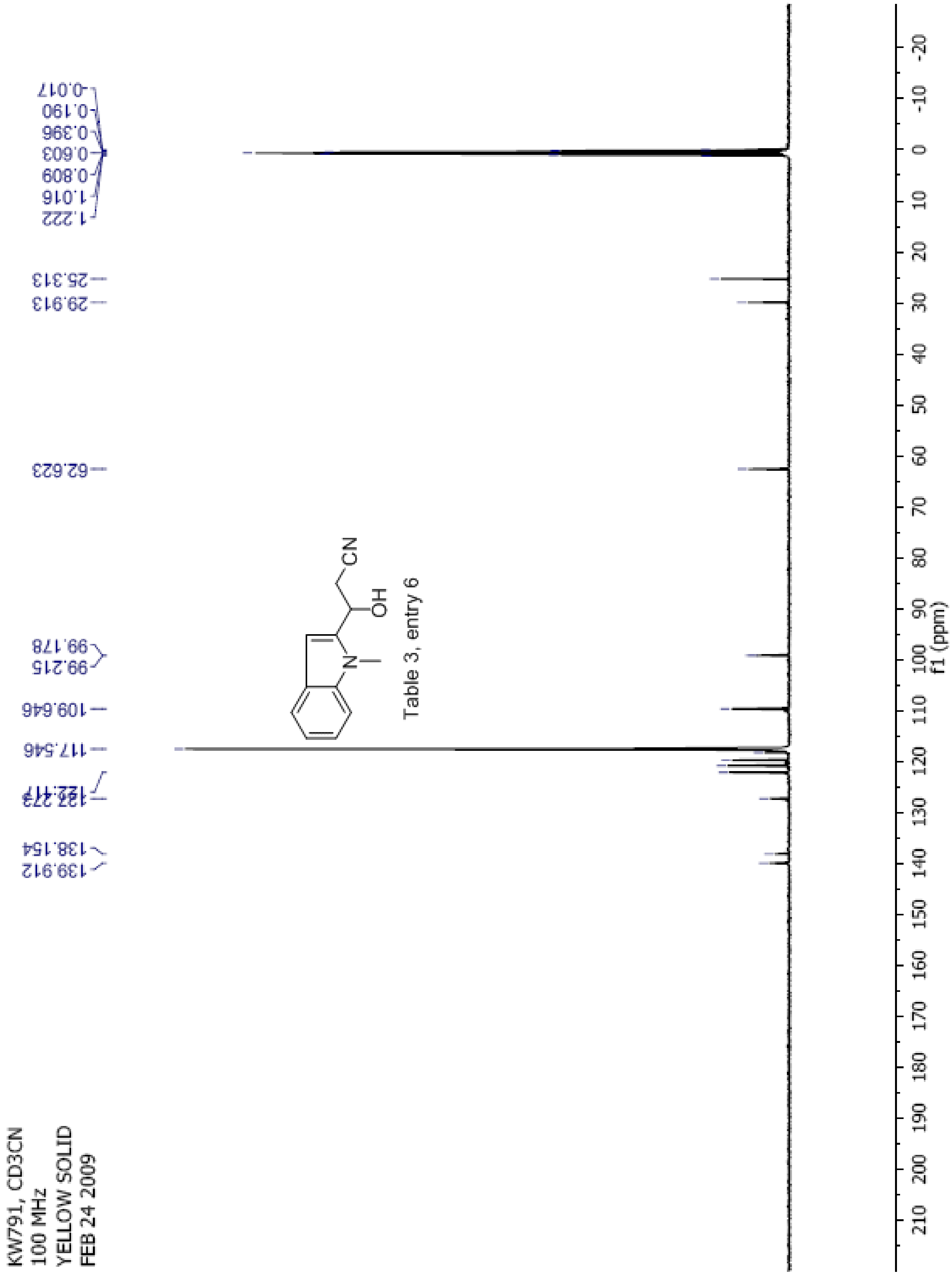



Table 3, entry 5

Date: Fri Mar 6 11:06:37 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98



KW791, CD3CN
400 MHz
YELLOW SOLID
FEB 24 2009
7.604
7.585
7.427
7.407
7.261
7.240
7.222
7.116
7.097
7.079
6.536
5.243
5.227
3.983
3.967
3.798
3.079
3.062
2.210
1.991
1.985
1.979
1.973
1.967
1.961
CN
N
OH
Table 3, entry 6
1.19
1.20
1.23
1.13
1.00
1.17
1.14
3.58
2.70
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


KW791, CD3CN
100 MHz
YELLOW SOLID
FEB 24 2009
-0.017
0.190
0.396
0.603
0.809
1.016
1.222
25.313
29.913
62.623
99.178
99.215
109.646
117.546
138.154
139.912
CN
OH
N
Table 3, entry 6
f1 (ppm)

# Manual Peak Matching Report
# For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 200.09496 | 200.09532 | 180,98882 | 1.8 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

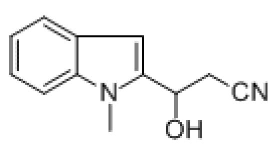



Table 3, entry 6

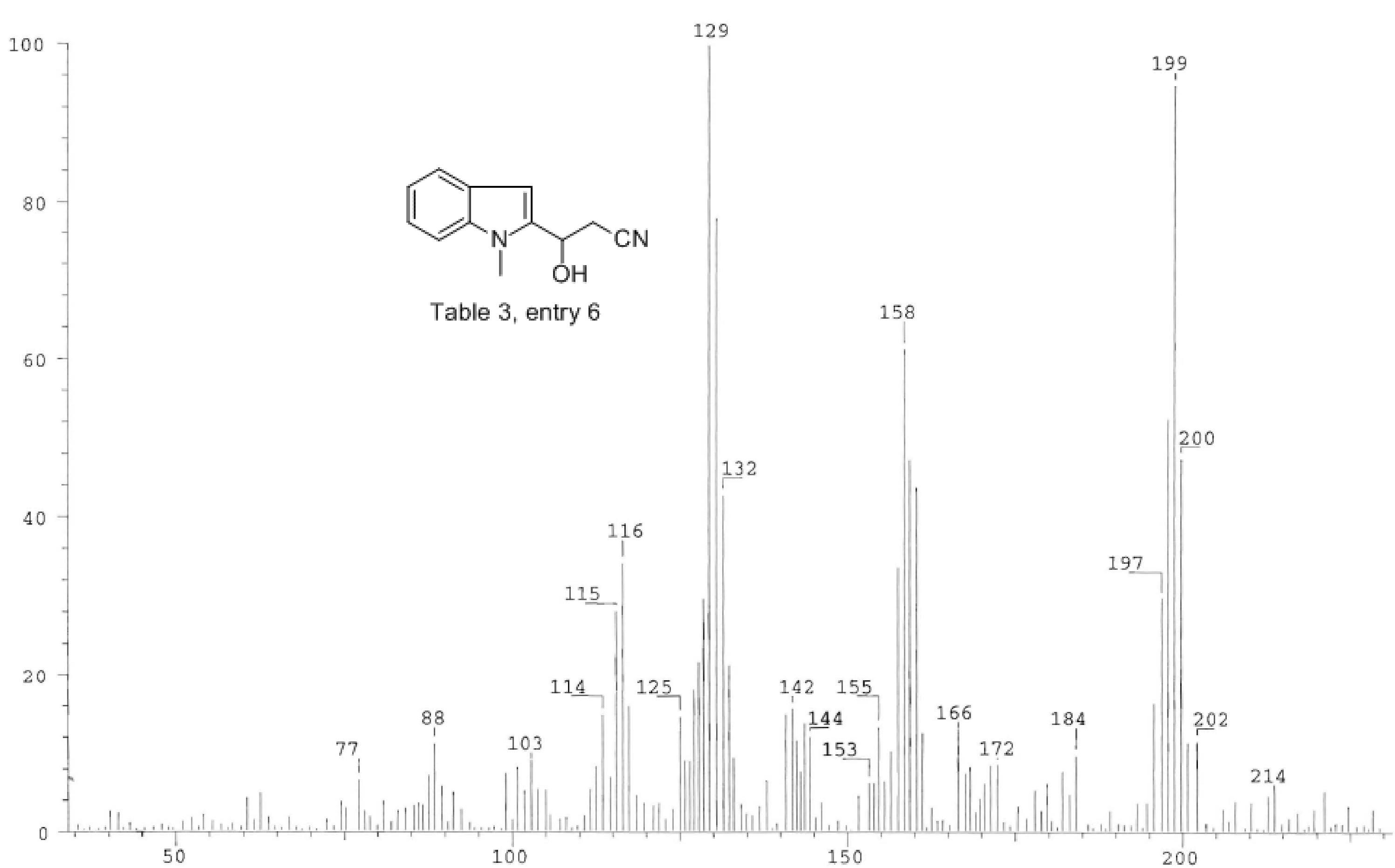
SPEC: fin084294.dat (06-MAR-09 10:59:25)
Samp: KW791
Comm: DP/EI
Oper: kh
Base: 129.35
Peak: 1000.0 mmu

Study: ms services
Masses: 35.01 > 650.00
Intensity: 10149

Scans: 1 > 67

Client: Kuldeep
#Peaks: 635
RIC: 210253

Scan 57 @ 1.33 min (EI +Q1MS LMR UP LR)




Date: Fri Mar 6 11:02:24 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

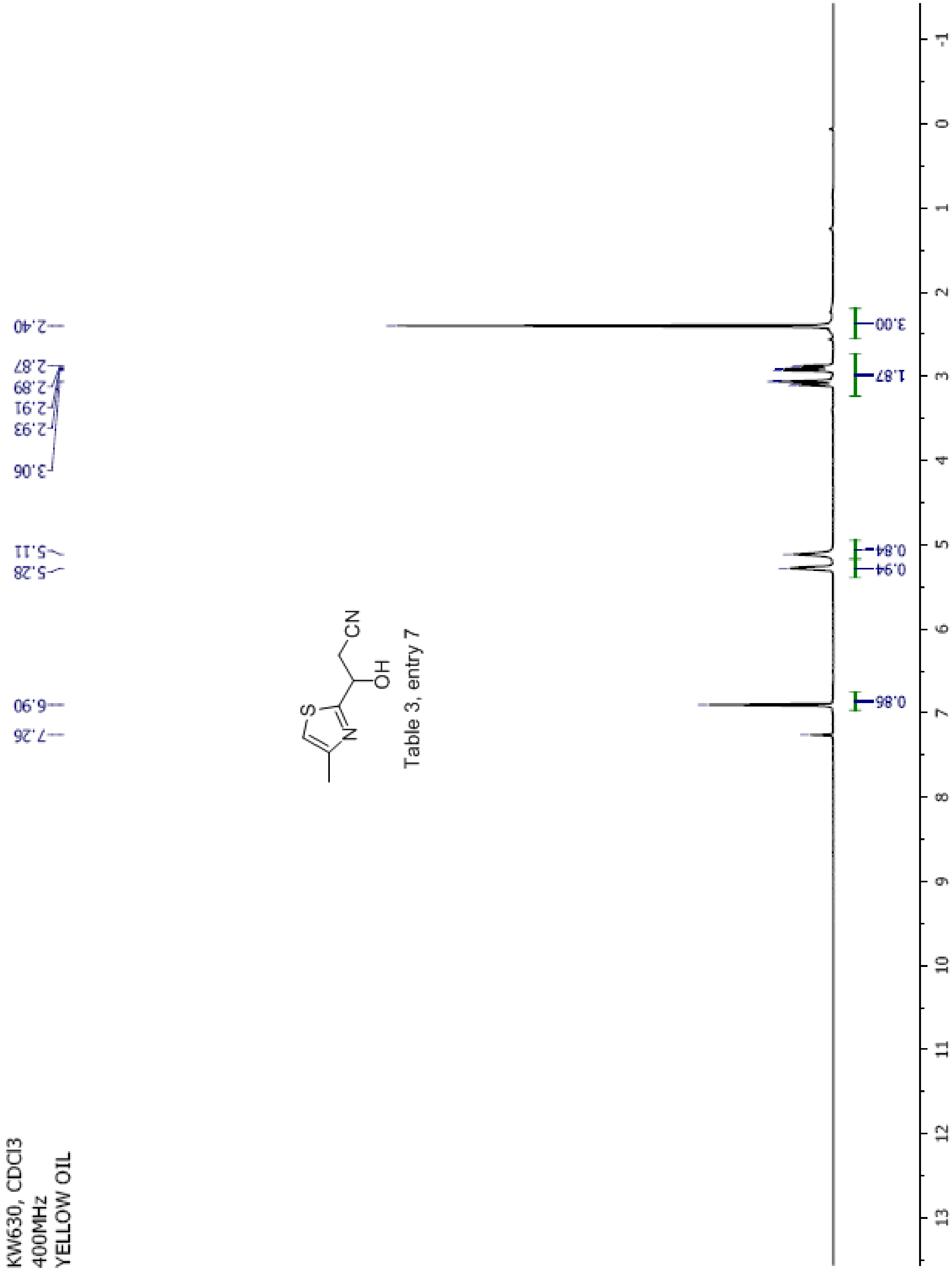
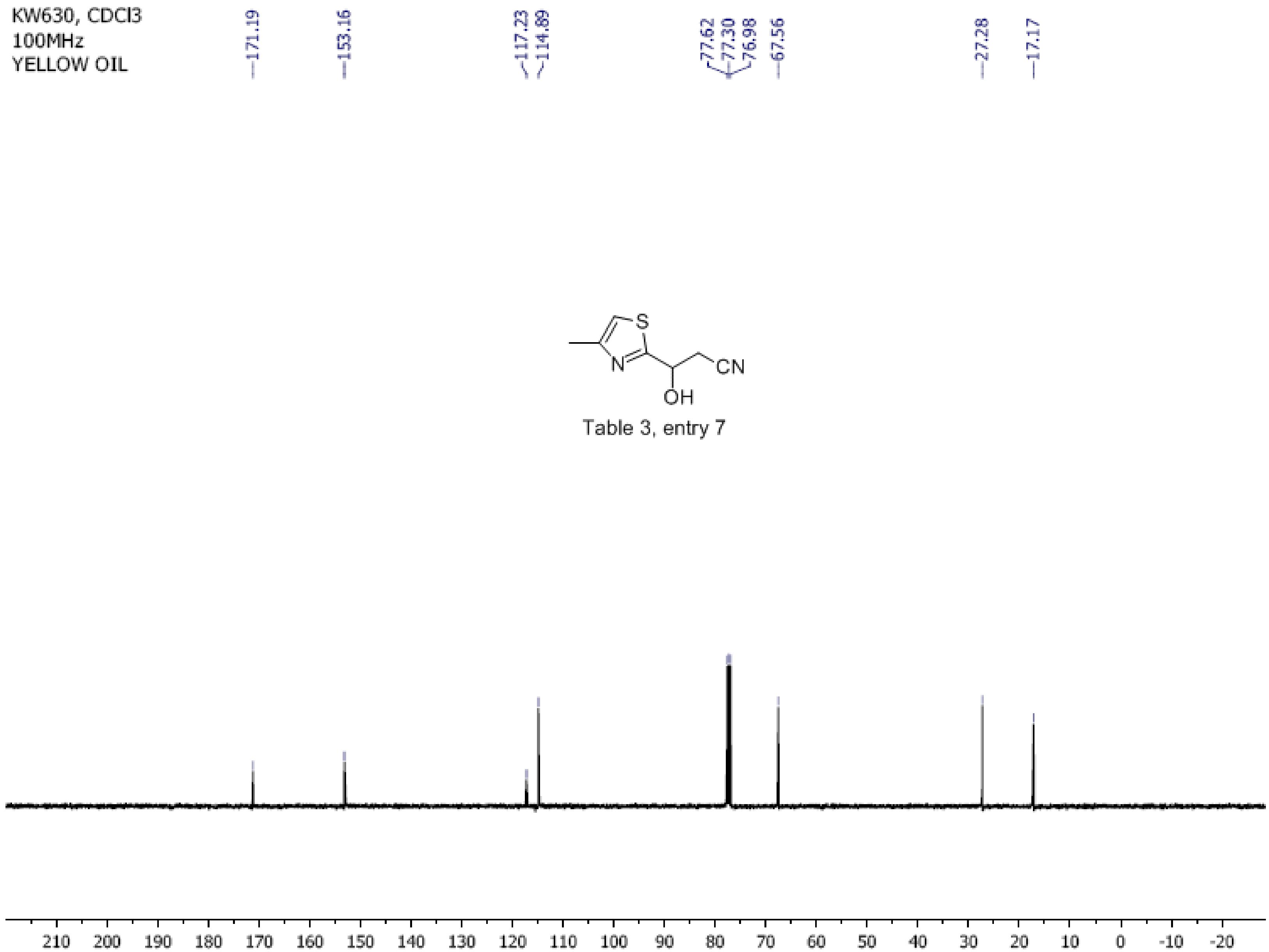


Table 3, entry 7


KW630, CDCl3
100MHz
YELLOW OIL
171.19
153.16
117.23
114.89
77.62
77.30
76.98
67.56
27.28
17.17
S
N
CN
OH
Table 3, entry 7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

Manual Peak Matching Report
For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 168.03573 | 168.03606 | 130.99201 | 2 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

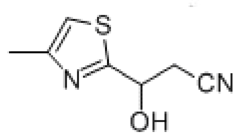


Table 3, entry 7

SPEC: fin083702 (11-JUN-08 11:12:31) Scans: 1 > 22
Samp: KW630
Comm: 70 eV EI
Oper: kh Study: MS services Client: Kuldup
Base: 128.15 Masses: 35.01 > 650.00 #Peaks: 662
Peak: 1000.0 mmu Intensity: 55743 RIC: 768012
REG #9 @ 0.41 min (EI +Q1MS LMR UP LR) (+13>19) 5.6E+04

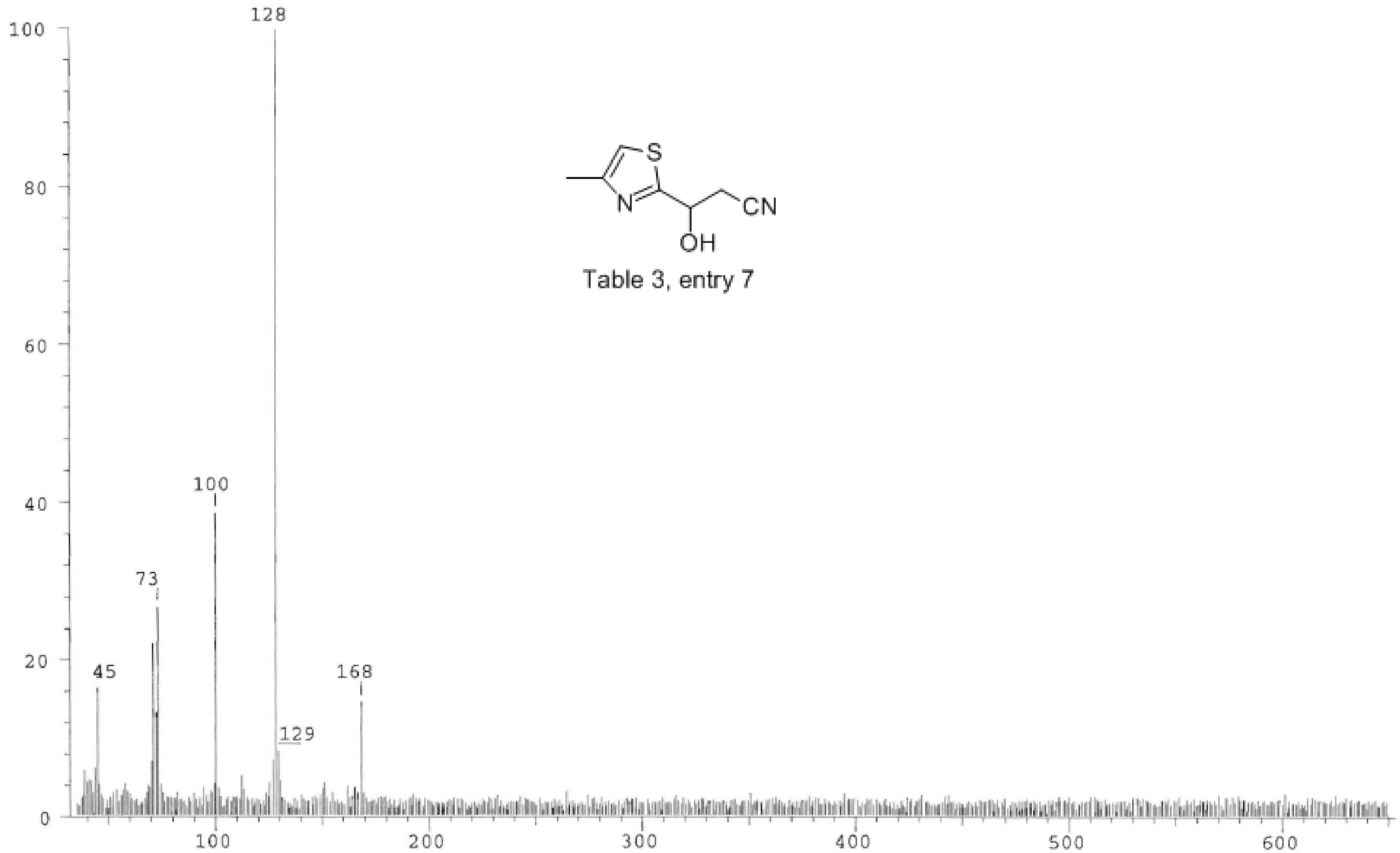


Table 3, entry 7

Date: Wed Jun 11 11:13:41 2008 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98


KW636, CDCl3
YELLOW OIL
300MHz

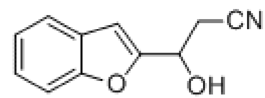
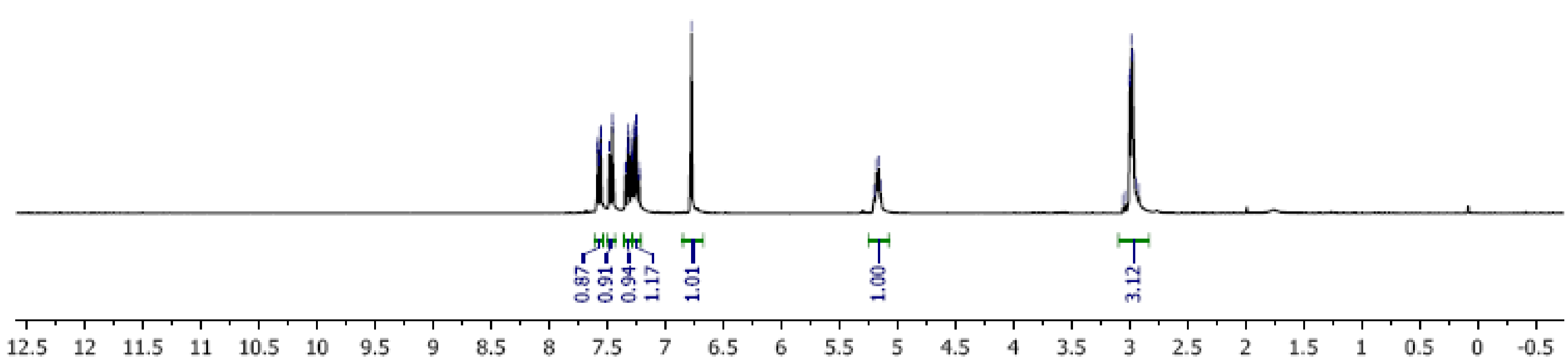



Table 3, entry 8



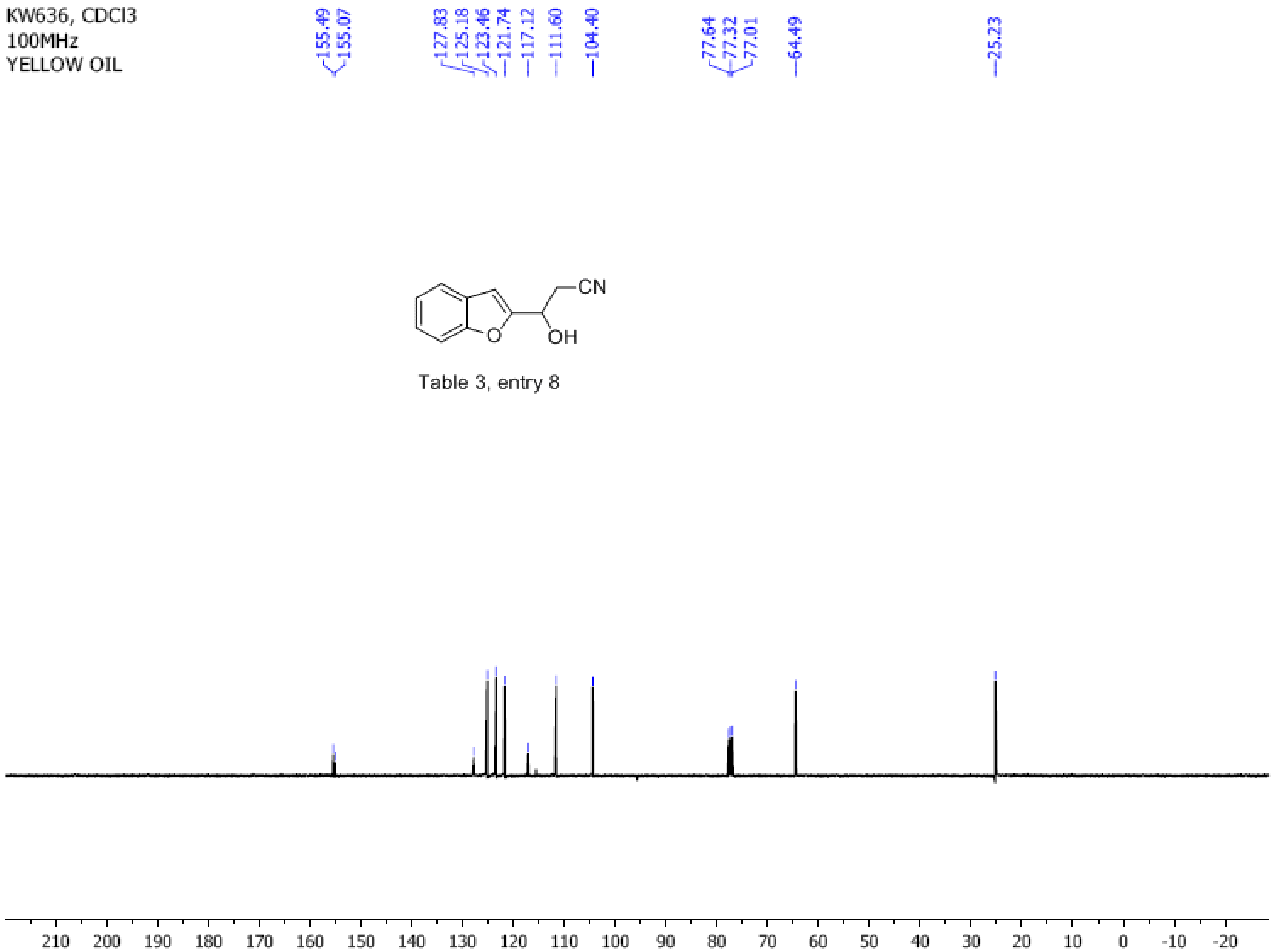



Table 3, entry 8

# Manual Peak Matching Report
# For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 187.06333 | 187.06371 | 180.98882 | 2 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass - theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

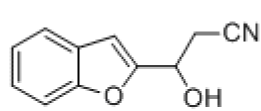



Table 3, entry 8

fio

SPEC: fin063689.dat (06-JUN-08 14:40:38)
Samp: KW636
Comm: 70 eV EI
Oper: kh
Base: 146.88
Peak: 1000.0 mmu

Study: Service
Masses: 35.01 > 650.00
Intensity: 22691

Scans: 1 > 35

Client: Kuldup
#Peaks: 623
RIC: 114141

Scan 30 @ 0.52 min (EI +Q1MS LMR UP LR)

2.3E+04

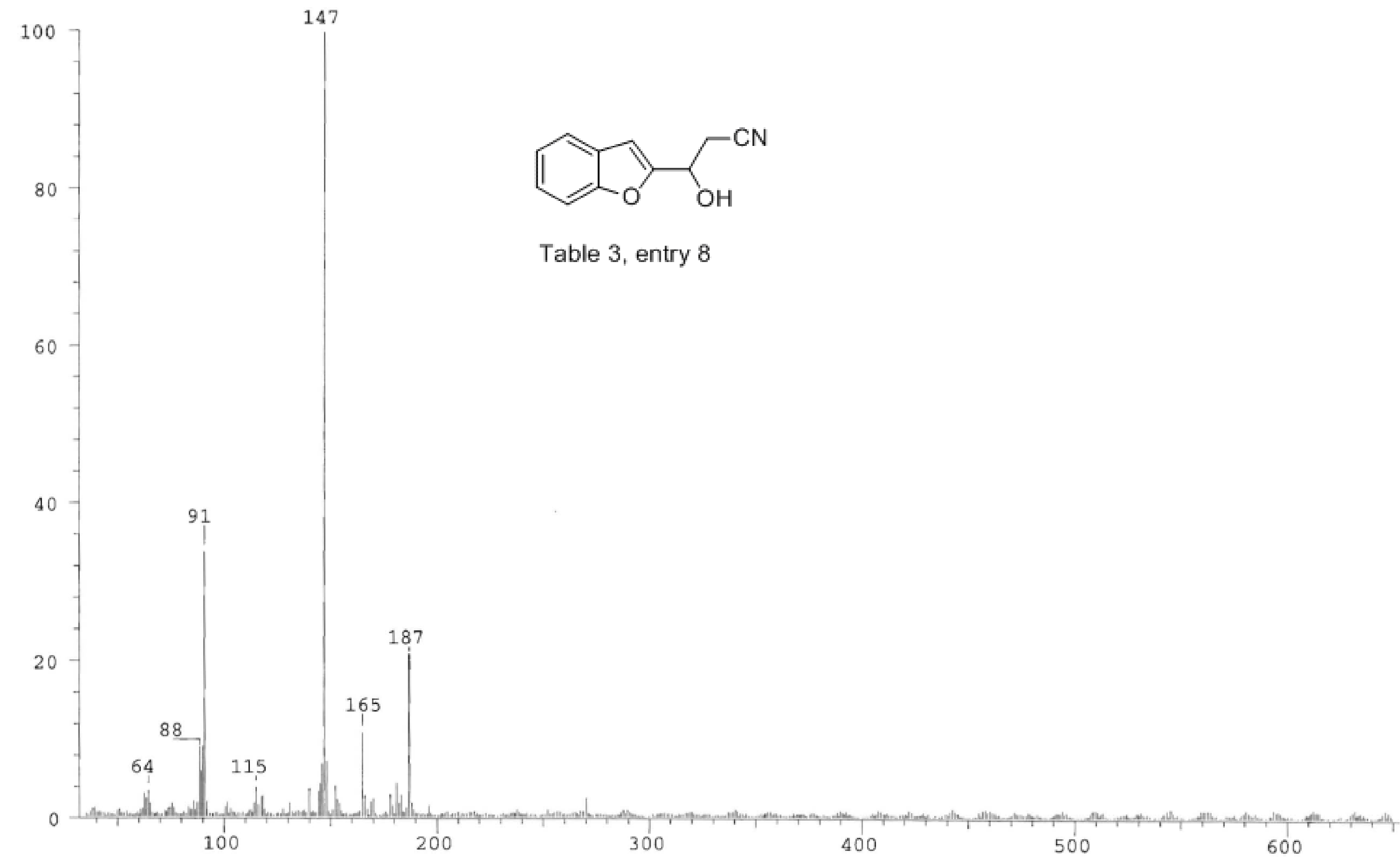



Table 3, entry 8

Date: Fri Jun 6 14:41:33 2008 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

KW641, CDCl3
300MHz
YELLOW SOLID

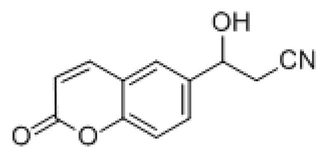
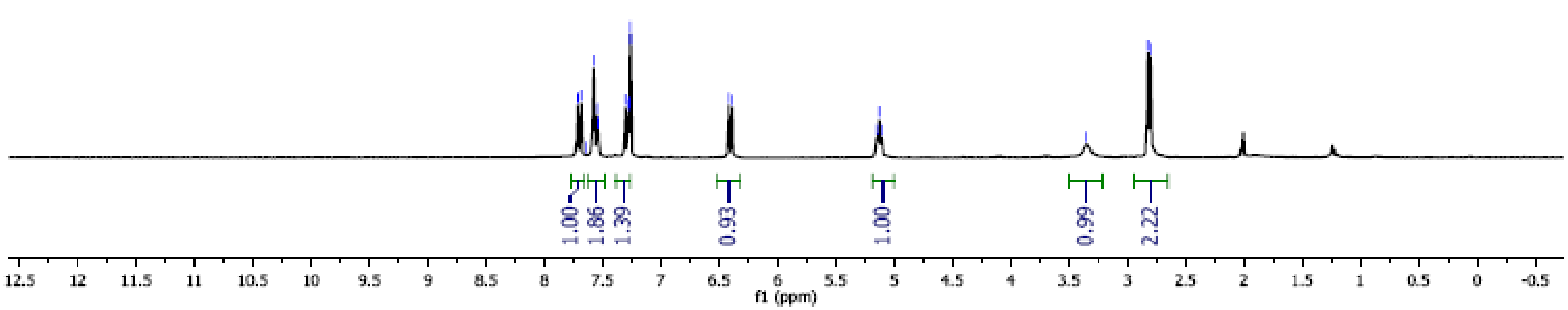
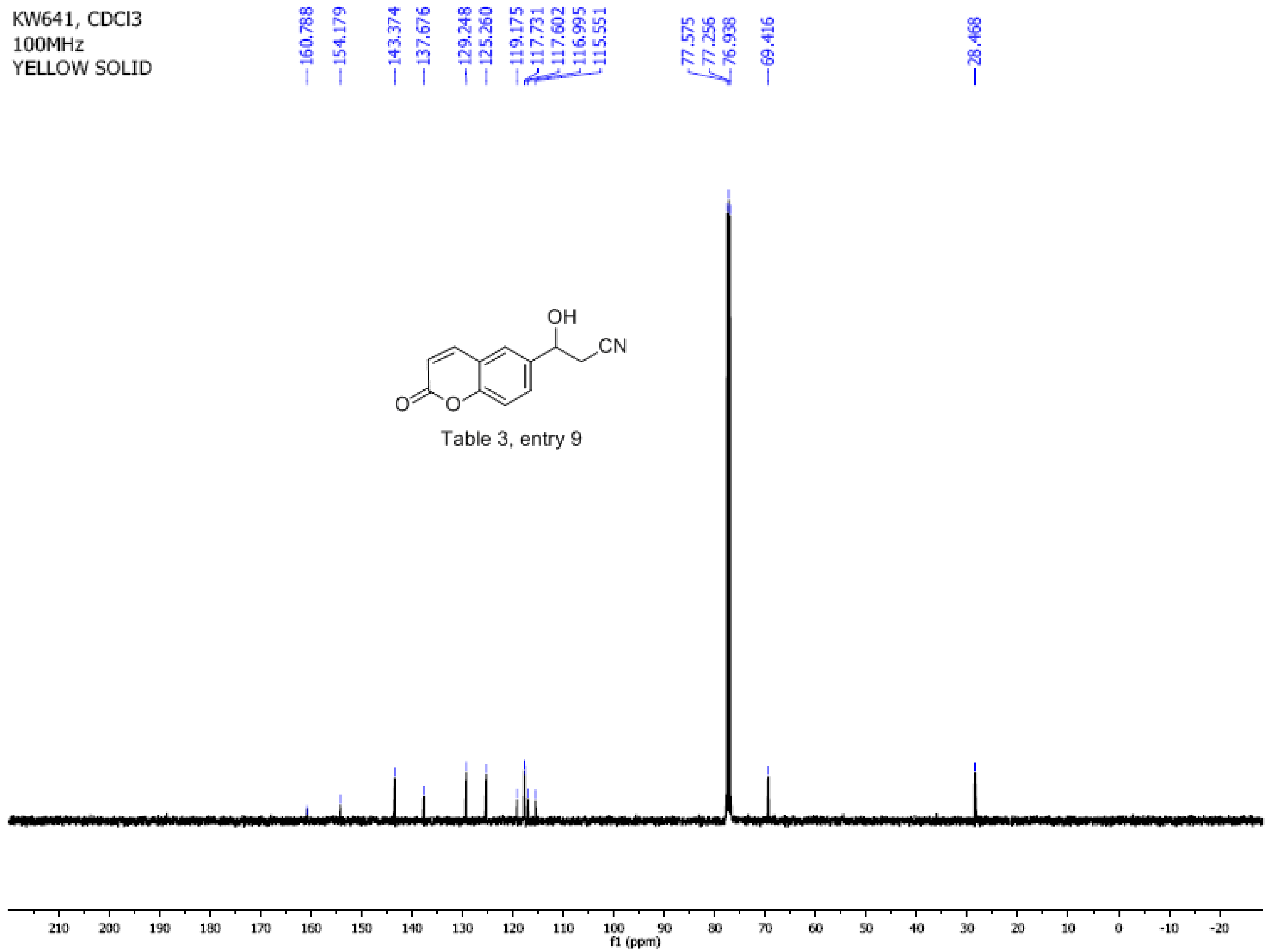


Table 3, entry 9




KW641, CDCl3
100MHz
YELLOW SOLID
160.788
154.179
143.374
137.676
129.248
125.260
119.175
117.731
117.602
116.995
115.551
77.575
77.256
76.938
69.416
28.468
OH
CN
O
O
Table 3, entry 9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

# Manual Peak Matching Report
# For Accurate Mass Determination

| Theoretical mass | Experimental mass | PFK matching mass | Deviation* |
|---|---|---|---|
| 215.05824 | 215.05862 | 180 98882 | 1.8 ppm |

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only $^{12}C$, $^{1}H$, $^{16}O$, $^{14}N$ etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

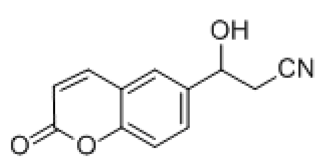



Table 3, entry 9

SPEC: fin083760.dat (02-JUL-08 12:53:17)
Samp: KW641
Comm: 70 eV EI
Oper: kh
Base: 43.96
Peak: 1000.0 mmu

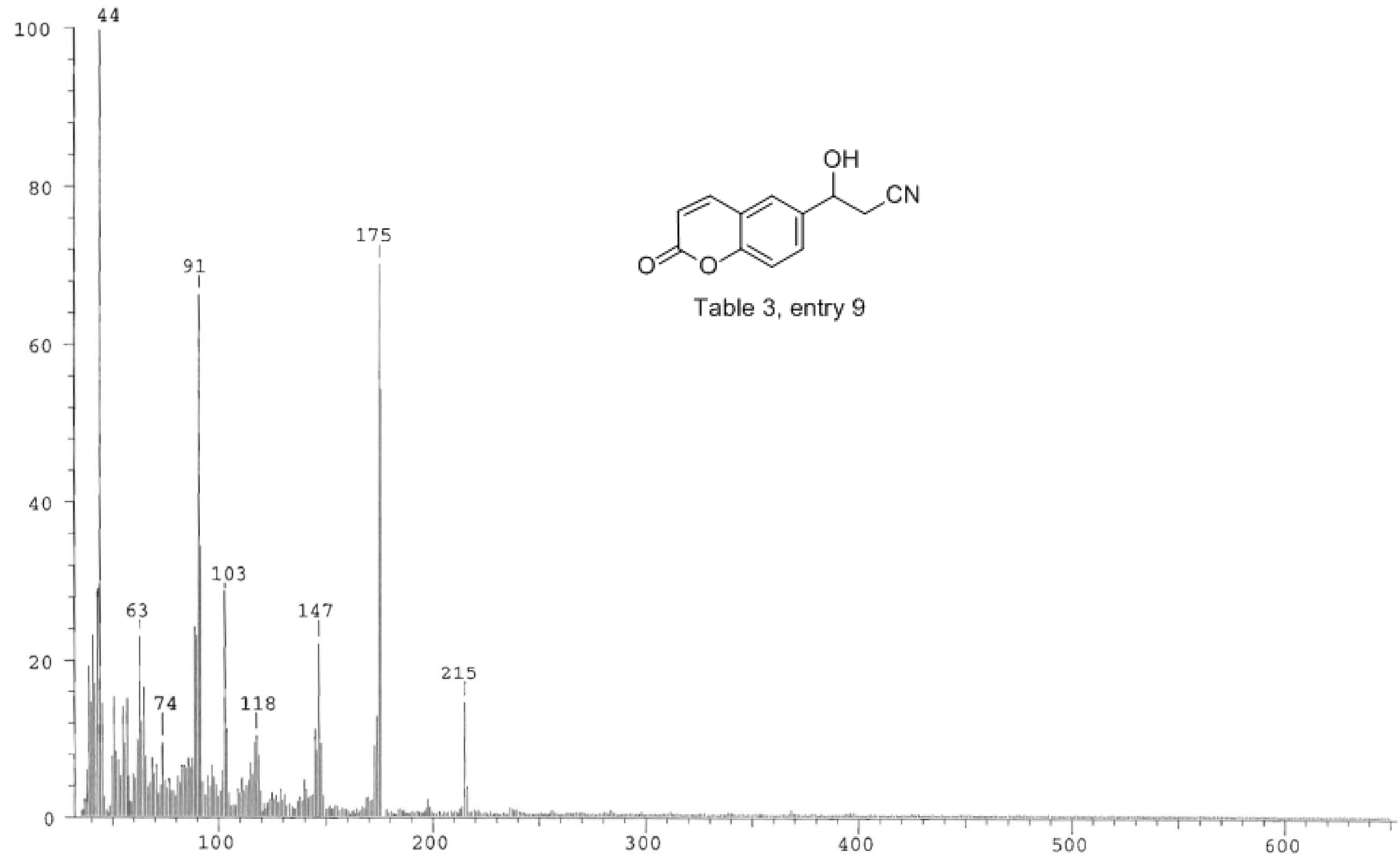
Study: MS services
Masses: 35.01 > 650.00
Intensity: 644784

Scans: 1 > 56

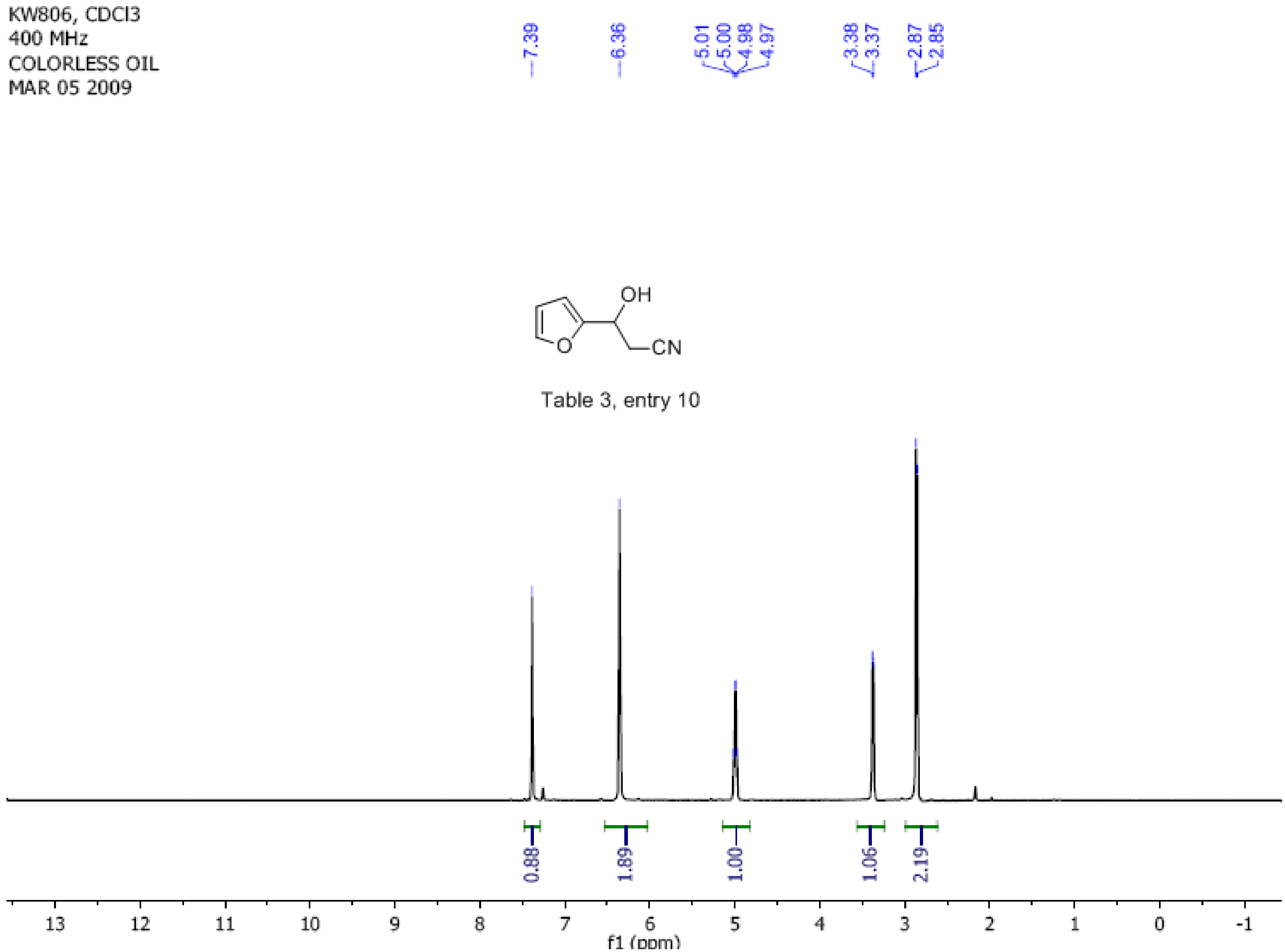
Client: Kuldup
#Peaks: 661
RIC: 8035415

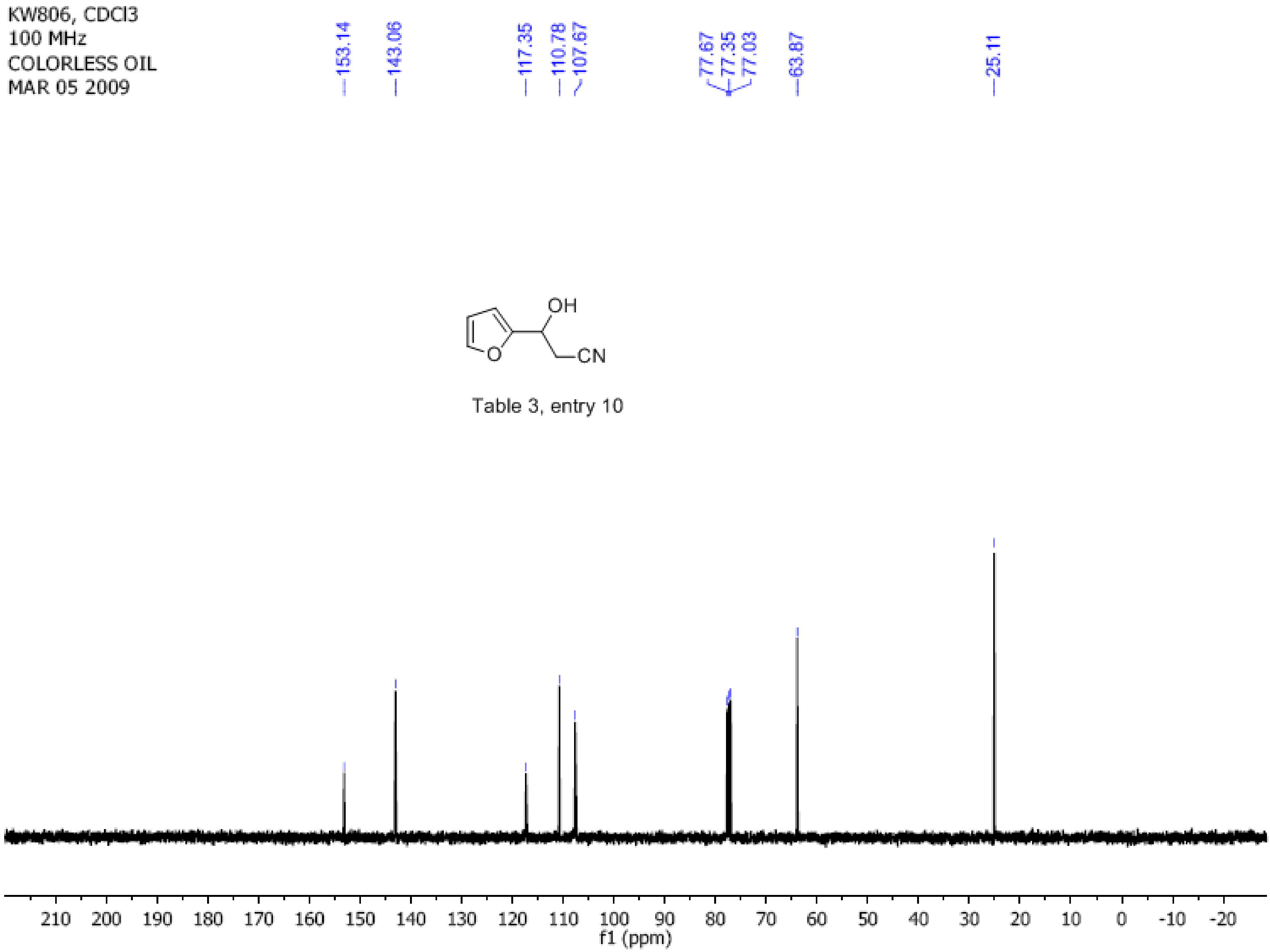
Scan 54 @ 1.27 min (EI +Q1MS LMR UP LR)

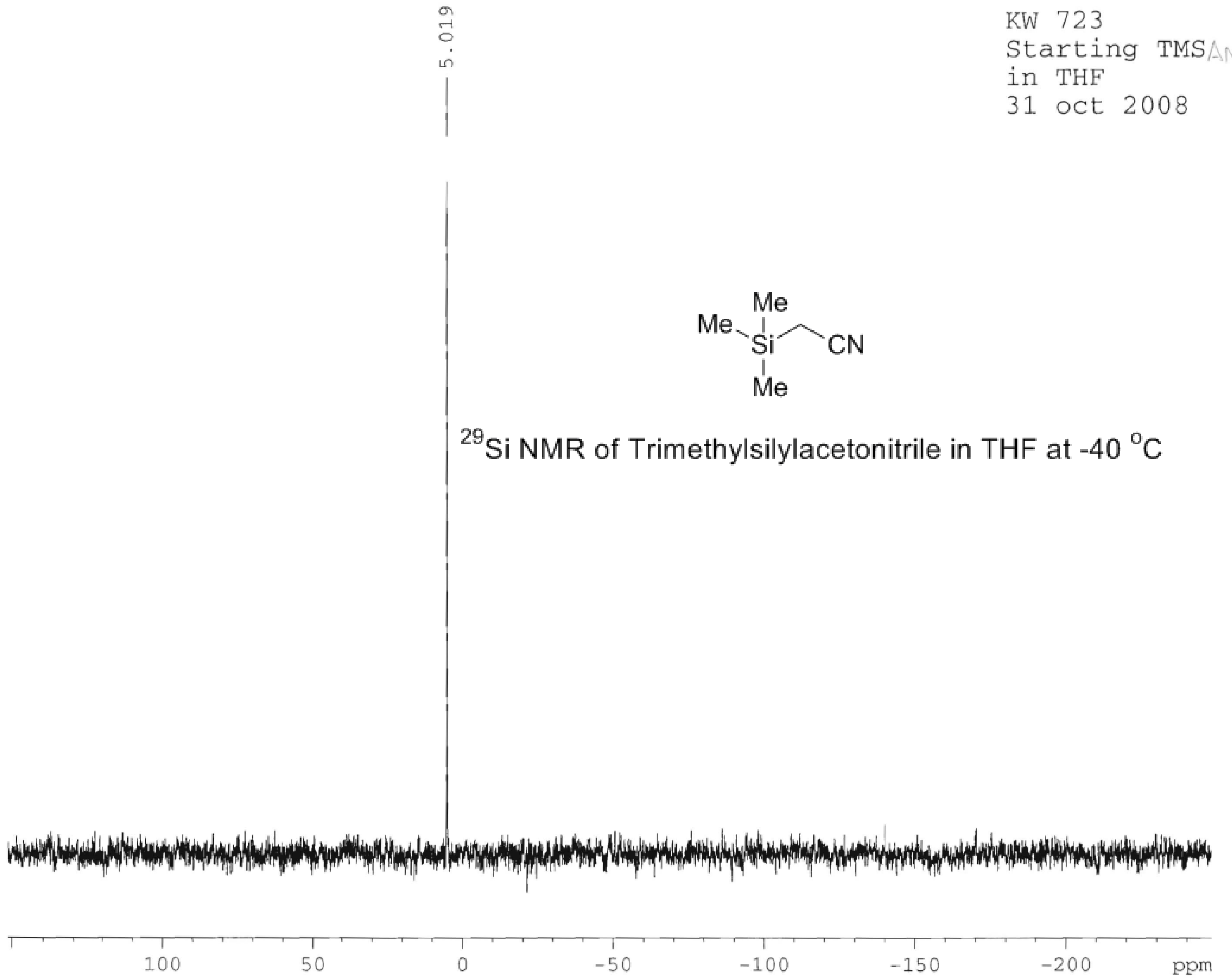
6.4E+05



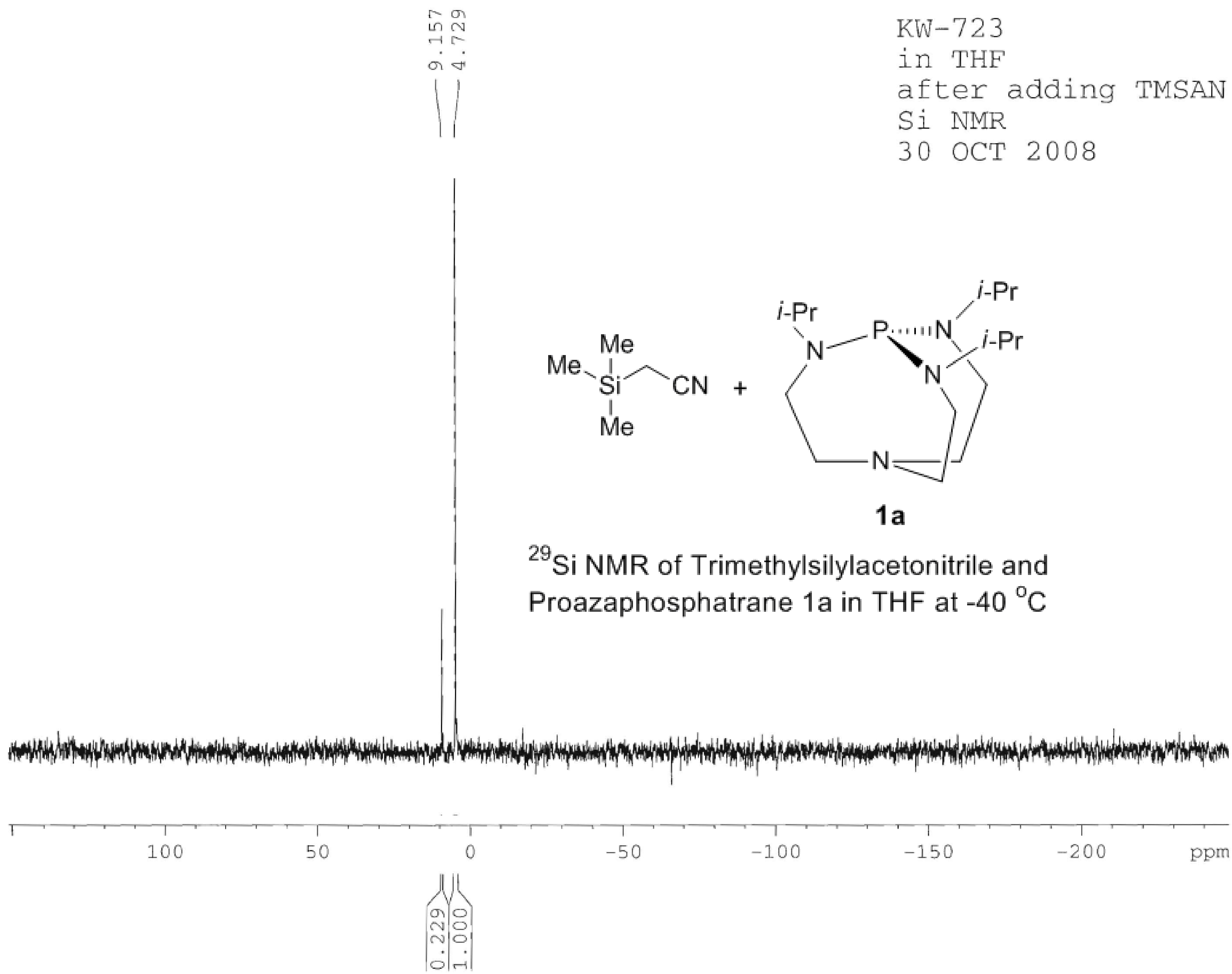

Date: Wed Jul 2 12:55:02 2008 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98


KW806, CDCl3
400 MHz
COLORLESS OIL
MAR 05 2009
7.39
6.36
5.01
5.00
4.98
4.97
3.38
3.37
2.87
2.85
OH
O
CN
Table 3, entry 10
0.88
1.89
1.00
1.06
2.19
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

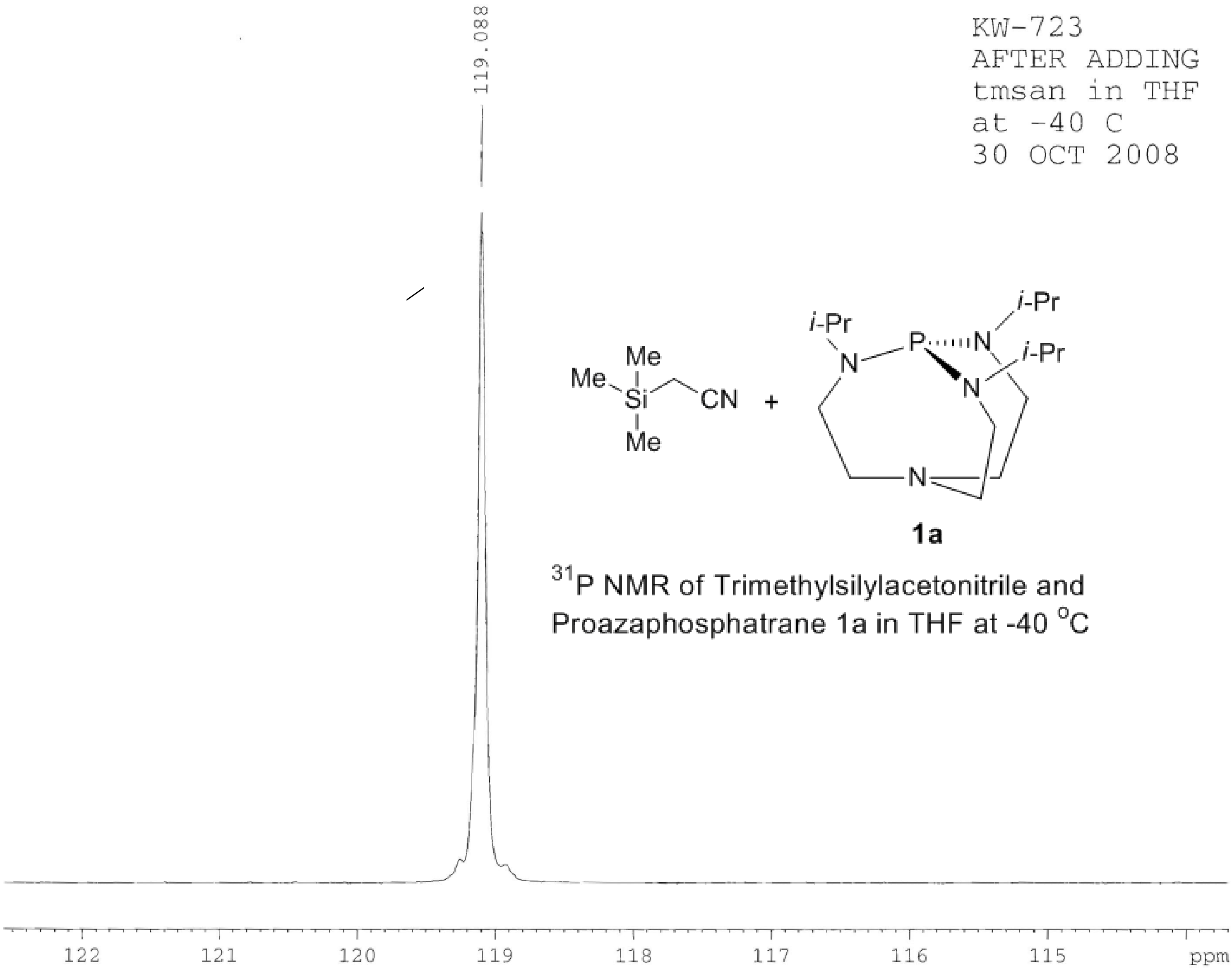

KW806, CDCl3
100 MHz
COLORLESS OIL
MAR 05 2009
153.14
143.06
117.35
110.78
107.67
77.67
77.35
77.03
63.87
25.11
OH
O
CN
Table 3, entry 10
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)




$^{29}Si$ NMR of Trimethylsilylacetonitrile in THF at -40 $^{o}C$




$^{29}$Si NMR of Trimethylsilylacetonitrile and Proazaphosphatrane 1a in THF at -40 $^{o}$C




$^{31}$P NMR of Trimethylsilylacetonitrile and Proazaphosphatrane 1a in THF at -40 $^{o}$C