

## **A one-pot preparation of cyanamide from dithiocarbamate using molecular iodine**

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Dithiocarbamate salt of amines can be prepared following any one of the literature reports (Wang, R.; Dolman, S. J. *J. Org. Chem.* **2007**, *72*, 3969, Li, G.; Tajima, H.; Ohtani, T. *J. Org. Chem.* **1997**, *62*, 4539) or the following modified literature procedure.

#### General procedure for the preparation of dithiocarbamate salt (1):

To a mixture of carbon disulfide (1.9g, 25 mmol) and triethylamine (3.03g, 30 mmol) under an ice cooled condition, was added aniline (0.93g, 10 mmol) drop wise with constant stirring. After complete addition of aniline, the reaction was stirred at room temperature for 2 hrs during which precipitation of phenyl dithiocarbamic acid salt was observed. Progress of the reaction was monitored by TLC. After completion of the reaction the product was filtered, washed with hexane : ethylacetate (5 : 95) (4 x 5 mL) to get rid of unreacted aniline and excess triethylamine. The product (Yield : 2.48g, 92 %) was dried in vacuum dessicator and used as such for the next reaction.

**Note :** This reaction is sluggish (18-24 hrs) particularly with bulky substrates and substrate containing electron withdrawing groups. In these cases the reactions were allowed to continue at room temperature until precipitation of dithiocarbamate salt was observed (18-24 hrs).

#### Spectral data:

**Phenyl cyanamide (1a).** Gummy; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 7.02-7.07 (m, 3H), 7.28-7.33 (m, 2H), 7.64 (brs, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 112.2, 115.5, 123.6, 129.8, 137.4; IR (KBr) 3175 (m), 2919 (m), 2227 (s), 1600 (s), 1501 (s), 1249 (m), 891 (w), 748 (s); elemental analysis for C<sub>7</sub>H<sub>6</sub>N<sub>2</sub> (118.13): calcd. C 71.17, H 5.12, N 23.71; found C 71.27, H 5.09, N 23.67.

**2-Methoxy-phenyl cyanamide (2a).** Oily Liquid; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 3.87 (s, 3H, OCH<sub>3</sub>), 6.88 (m, 1H), 6.95-7.05 (m, 2H), 7.18 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 55.9, 110.8, 111.0, 114.9, 121.5, 123.8, 126.8, 146.9; IR (KBr) 3219 (m), 2939 (m), 2839 (m), 2224 (s), 1603 (s), 1509 (s), 1454 (s), 1259 (s), 1026 (m), 746 (m); elemental analysis for C<sub>8</sub>H<sub>8</sub>N<sub>2</sub>O (148.16): calcd. C 64.85, H 5.44, N 18.90; found C 64.79, H 5.42, N 18.78.

**2-Chloro-phenyl cyanamide (4a).** White Solid; Mp: 101-103 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 6.56 (brs, 1H), 7.05 (m, 1H), 7.31 (m, 2H), 7.35 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 110.0, 116.2, 120.4, 124.5, 128.6, 129.9, 134.3; IR (KBr) 3163 (s), 2921 (w), 2243 (s), 1598 (s), 1500 (s), 1426 (m), 1295 (m), 1049 (w), 746 (s); elemental analysis for C<sub>7</sub>H<sub>5</sub>ClN<sub>2</sub> (152.58): calcd. C 55.10, H 3.30, N 18.36; found C 55.11, H 3.32, N 18.29.

**3-Nitro-phenyl cyanamide (6a).** Yellow Solid; Mp: 133-135 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO, 400 MHz,) δ (ppm) 7.38 (d, *J* = 8.4 Hz, 1H), 7.52 (t, *J* = 8.4 Hz, 1H), 7.85 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub> + DMSO, 100 MHz,) δ (ppm) 109.6, 110.7, 116.8, 120.8, 130.1, 139.9, 148.4; IR (KBr) 3147 (m), 2919 (m), 2241 (s), 1621 (w), 1531 (s), 1354 (s), 1260 (m), 1071 (w), 937 (w), 871 (m), 733 (s); elemental analysis for C<sub>7</sub>H<sub>5</sub>N<sub>3</sub>O<sub>2</sub> (163.14): calcd. C 51.54, H 3.09, N 25.76; found C 51.58, H 3.12, N 25.7

**4-Acetyl-phenylcyanamide (8a).** White Solid; Mp: 153-157 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO, 400 MHz,) δ (ppm) 2.56 (s, 3H, CH<sub>3</sub>), 7.08 (d, *J* = 8.8 Hz, 2H), 7.91 (d, *J* = 8.8 Hz, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub> + DMSO, 100 MHz,) δ (ppm) 25.9, 110.9, 114.5, 129.8, 131.2, 142.9, 196.2; IR (KBr) 3188 (s), 2966 (m), 2228 (s), 1666 (s), 1599 (s), 1585 (s), 1411 (m), 1362 (m), 1278 (s), 1176 (m), 962 (w); elemental analysis for C<sub>9</sub>H<sub>8</sub>N<sub>2</sub>O (160.18): calcd. C 67.49, H 5.03, N 17.48; found C 67.53, H 5.08, N 17.44.

**4-Chloro-phenyl cyanamide (9a).** White Solid; Mp: 95 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 6.91 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 111.4, 116.9, 128.9, 129.9, 136.2; IR (KBr) 3166 (m), 2954 (w), 2234 (s), 1600 (m), 1494 (s), 1399 (w), 1251 (m), 1091 (m), 1011 (w), 820 (m); elemental analysis for C<sub>7</sub>H<sub>5</sub>ClN<sub>2</sub> (152.58): calcd. C 55.10, H 3.30, N 18.36; found C 55.09, H 3.33, N 18.32.

**4-Hydroxy-phenyl cyanamide (10a).** White solid; Mp: 259-261 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO, 400 MHz,) δ (ppm) 5.67 (brs, 1H), 6.77 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 8.98 (brs, 1H, OH); <sup>13</sup>C NMR (CDCl<sub>3</sub> + DMSO, 100 MHz,) δ (ppm) 112.8, 115.6, 115.8, 129.5, 152.2; IR (KBr) 3213 (s), 2992 (w), 2230 (s), 1613 (w), 1519 (s), 1444 (m),

1258 (m), 1224(s), 815 (m); elemental analysis for C<sub>7</sub>H<sub>6</sub>N<sub>2</sub>O (134.14): calcd. C 62.68, H 4.51, N 20.88; found C 62.72, H 4.55, N 20.83.

**2-Iodo-4-methyl-phenyl cyanamide (13a).** White solid; Mp: 144 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 2.29 (s, 3H), 6.17 (brs, 1H), 7.17 (m, 2H), 7.56 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 20.4, 84.2, 110.7, 115.4, 130.9, 135.4, 139.6; IR (KBr) 3229 (s), 2919 (w), 2217 (s), 1603 (w), 1502 (s), 1420 (m), 1383 (m), 1283 (w), 1032 (w), 866 (w), 805 (m); elemental analysis for C<sub>8</sub>H<sub>7</sub>IN<sub>2</sub> (258.06): calcd. C 37.23, H 2.73, N 10.86; found C 37.28, H 2.68, N 10.80.

**2,4-Dimethyl-phenyl cyanamide (14a).** White solid; Mp: 115-119 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 2.18 (s, 3H, CH<sub>3</sub>), 2.26 (s, 3H, CH<sub>3</sub>), 6.74 (brs, 1H, NH), 6.93 (s, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 8.0 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 17.3, 20.7, 112.8, 115.7, 124.7, 127.9, 131.8, 133.2, 133.3; IR (KBr) 3186 (s), 2915 (w), 2233 (s), 1599 (w), 1512 (s), 1433 (m), 1271 (w), 1031 (w), 812 (m); elemental analysis for C<sub>9</sub>H<sub>10</sub>N<sub>2</sub> (146.19): calcd. C 73.94, H 6.89, N 19.16; found C 73.87, H 6.86, N 19.14.

**Cyclohexyl-cyanamide (16a).** Gummy; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 1.31 (m, 5H), 1.61 (m, 1H), 1.78 (m, 2H), 1.95 (m, 2H), 3.09 (m, 1H), 3.91 (brs, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 24.3, 25.1, 32.6, 54.3, 115.9; IR (KBr) 3196 (m), 2933 (s), 2857 (m), 2217 (s), 1453 (m), 1367 (w), 1167 (w), 892 (w); elemental analysis for C<sub>7</sub>H<sub>12</sub>N<sub>2</sub> (124.19): calcd. C 67.70, H 9.74, N 22.56; found C 67.67, H 9.70, N 22.50.

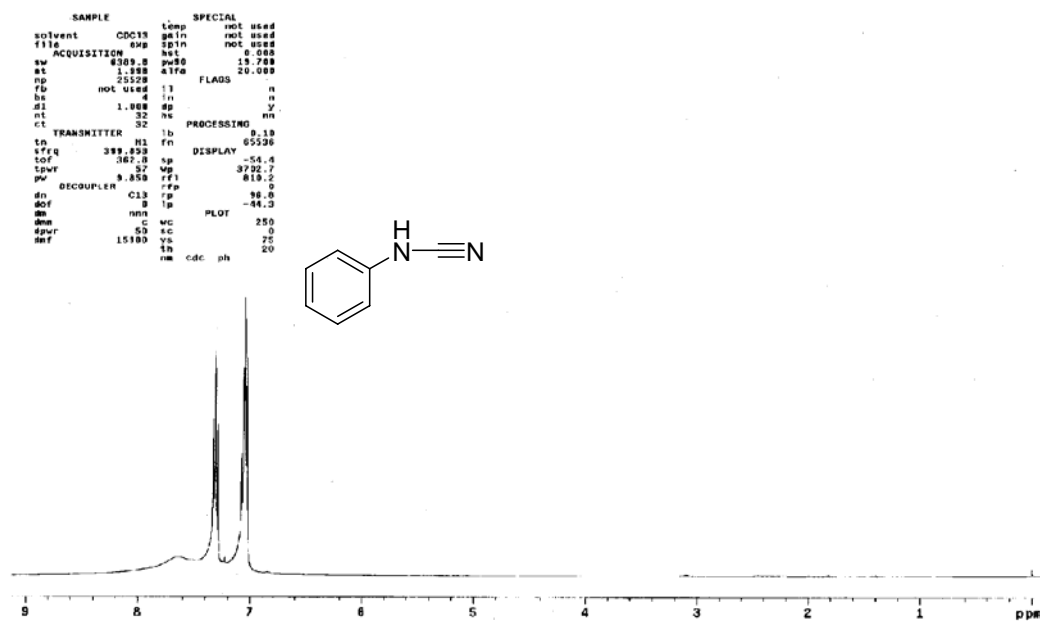
***n*-Butyl-cyanamide (17a).** Gummy; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 0.94 (t, *J* = 7.6 Hz, 3H, CH<sub>3</sub>), 1.40 (m, 2H, CH<sub>2</sub>), 1.58 (m, 2H, CH<sub>2</sub>), 3.06 (m, 2H, CH<sub>2</sub>), 4.61 (brs, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 13.6, 19.5, 31.7, 45.7, 117.2; IR (KBr) 3207 (s), 2961 (s), 2875 (m), 2221 (s), 1614 (m), 1463 (m), 1373 (w), 1171 (w), 1015 (w); elemental analysis for C<sub>5</sub>H<sub>10</sub>N<sub>2</sub> (98.15): calcd. C 61.19, H 10.27, N 28.54; found C 61.22, H 10.23, N 28.48.

**Benzyl cyanamide (18a).** Gummy; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 4.11 (d, *J* = 5.2 Hz, 2H, CH<sub>2</sub>), 4.66 (brs, 1H), 7.27-7.37 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 49.9, 116.7, 127.9, 128.4, 128.9, 136.4; IR (KBr) 3207 (m), 2925 (w), 2220 (s), 1455 (m),

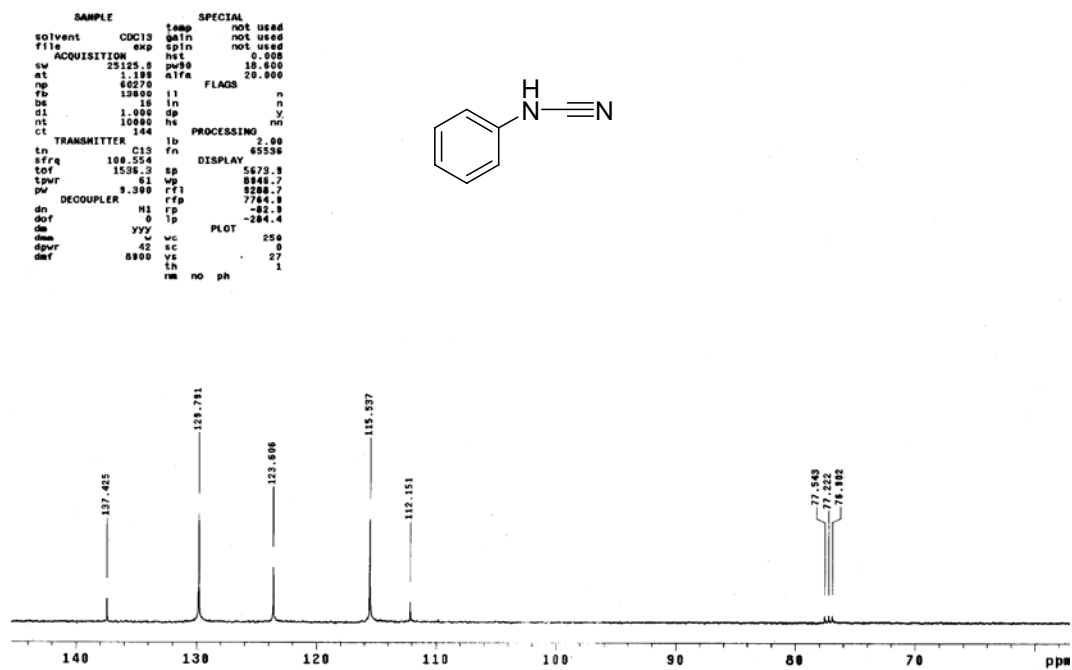
1359 (w), 1210 (w), 1155 (w), 1014 (w), 895 (w); elemental analysis for C<sub>8</sub>H<sub>8</sub>N<sub>2</sub> (132.17): calcd. C 72.70, H 6.10, N 21.19; found C 72.66, H 6.13, N 21.11.

**Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a).** White Solid; Mp: 82-84 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ (ppm) 4.05 (d, *J* = 5.2 Hz, 2H, CH<sub>2</sub>), 4.57 (brs, 1H), 5.94 (s, 2H, OCH<sub>2</sub>), 6.77 (m, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ (ppm) 49.9, 101.4, 108.46, 108.54, 116.5, 121.7, 130.1, 147.8, 148.2; IR (KBr) 3233 (m), 2952 (w), 2897 (w), 2220 (s), 1850 (w), 1609 (w), 1500 (m), 1445 (m), 1370 (m), 1323 (m), 1252 (s), 1038 (m), 925 (m), 862 (w), 809 (m); elemental analysis for C<sub>9</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub> (176.18): calcd. C 61.36, H 4.58, N 15.90; found C 61.41, H 4.61, N 15.85.

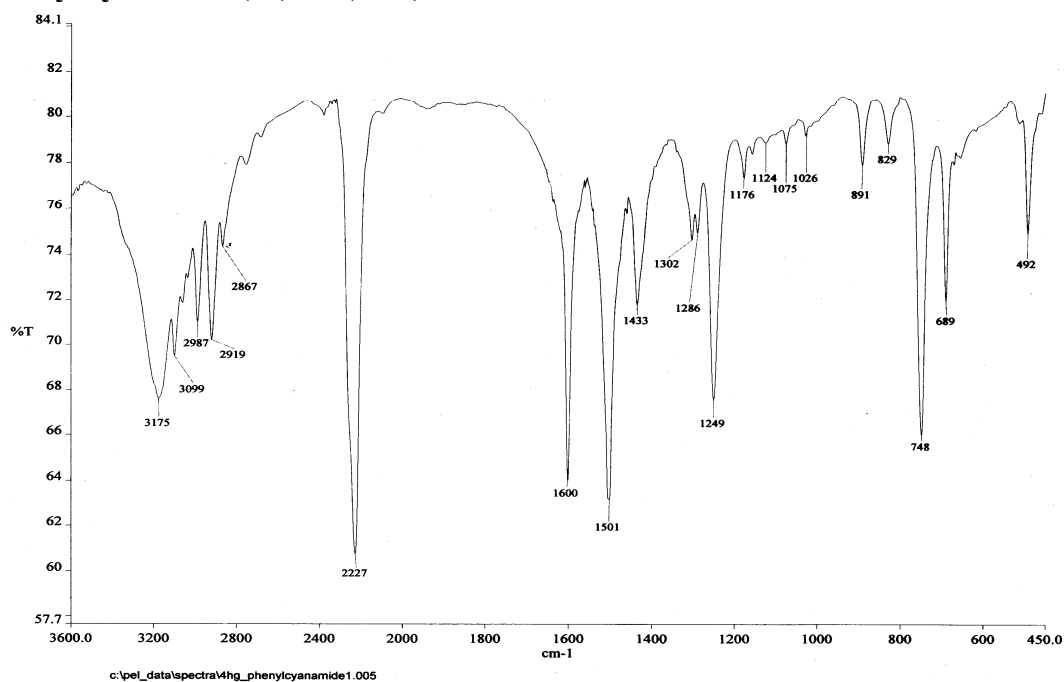
Phenyl cyanamide (1a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):



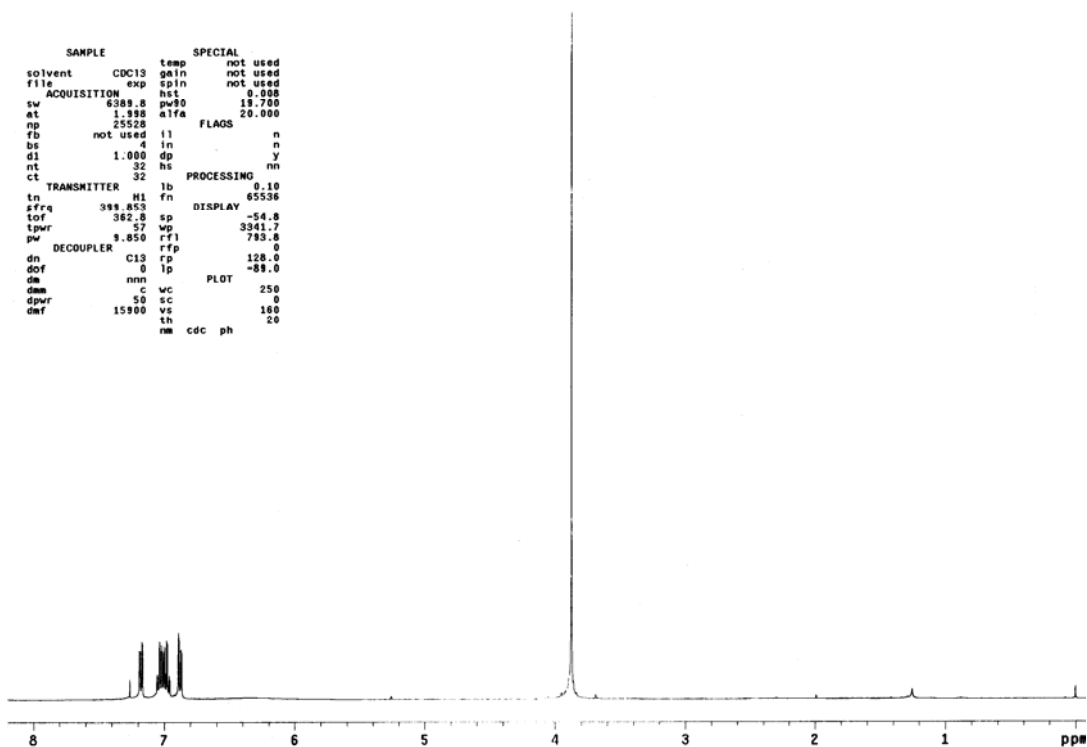
Phenyl cyanamide (1a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



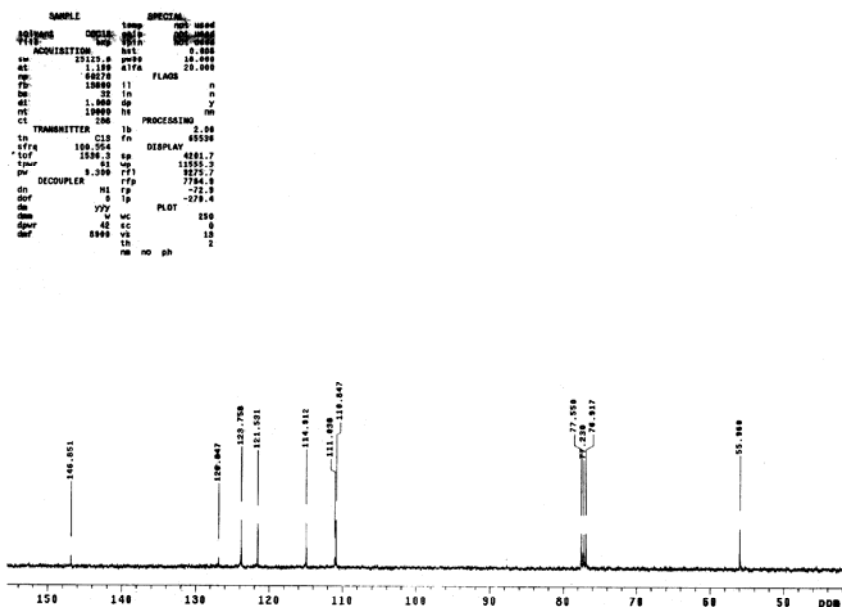
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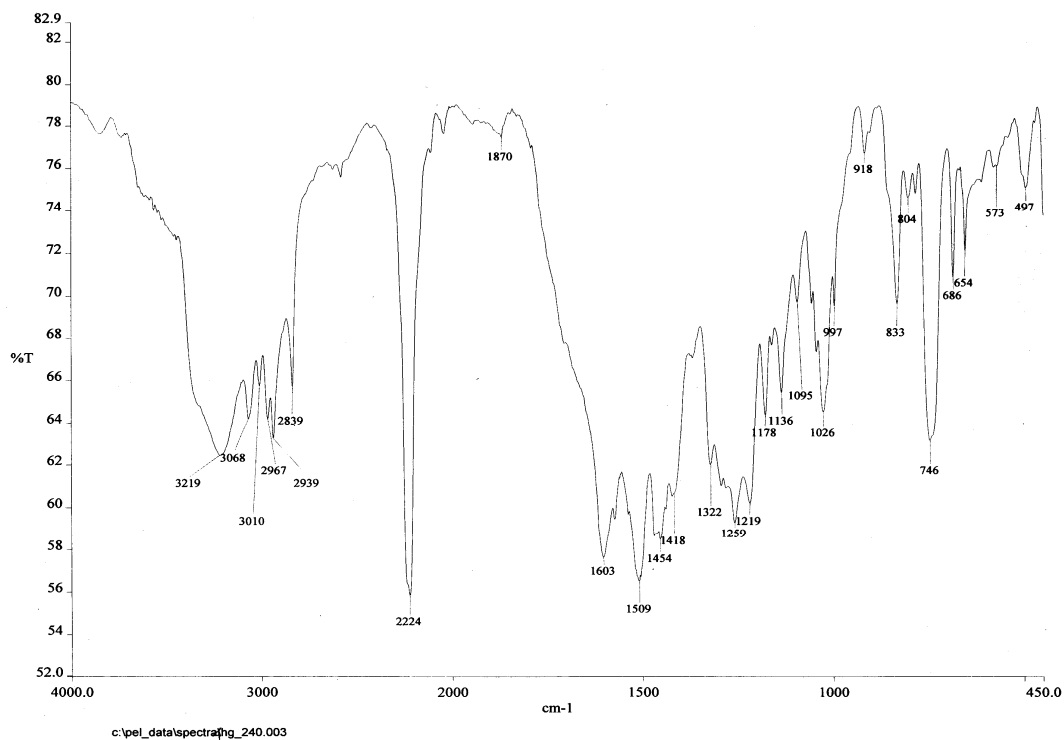
2-Methoxy-phenyl cyanamide (2a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):



2-Methoxy-phenyl cyanamide (2a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):

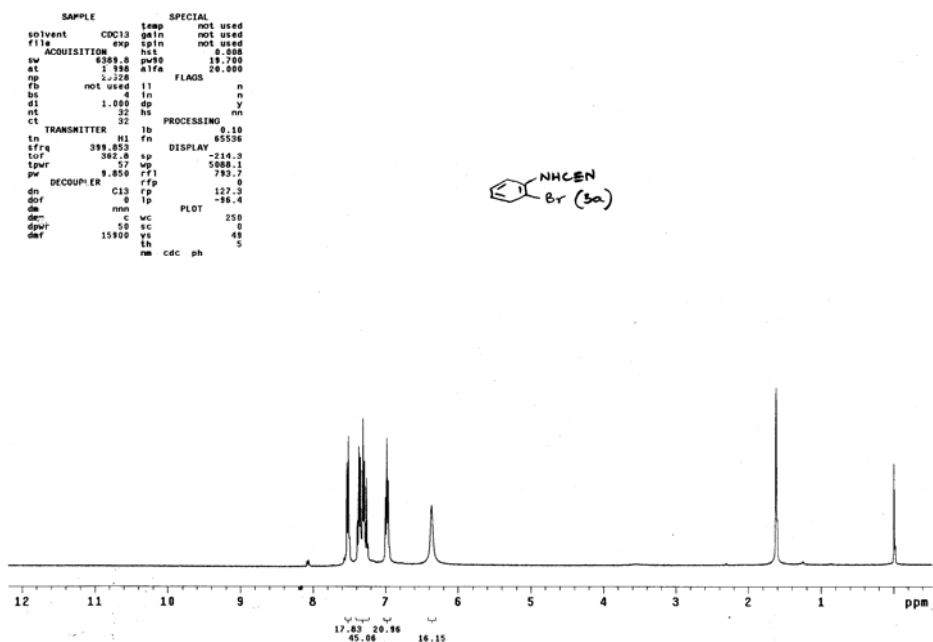


2-Methoxy-phenyl cyanamide (2a): IR(KBr):

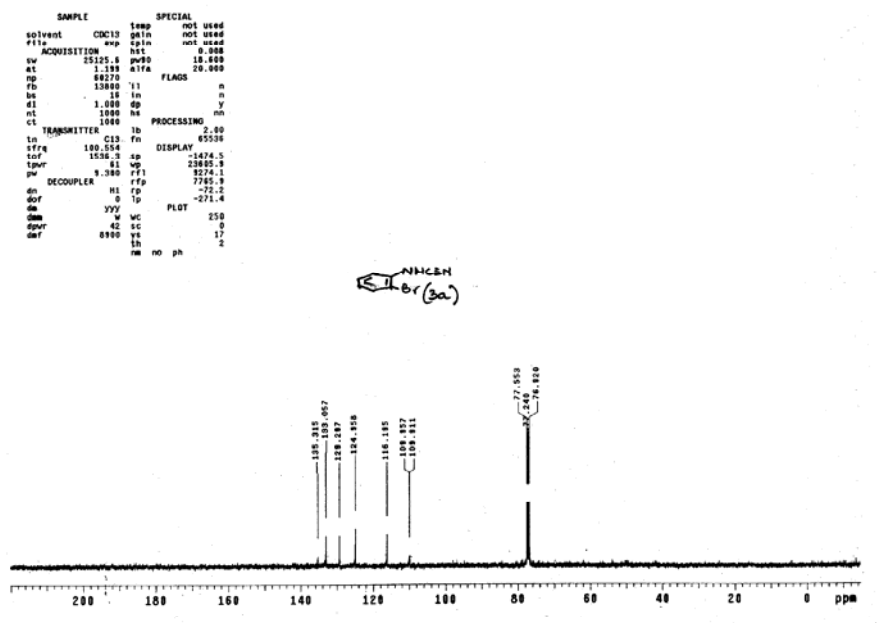




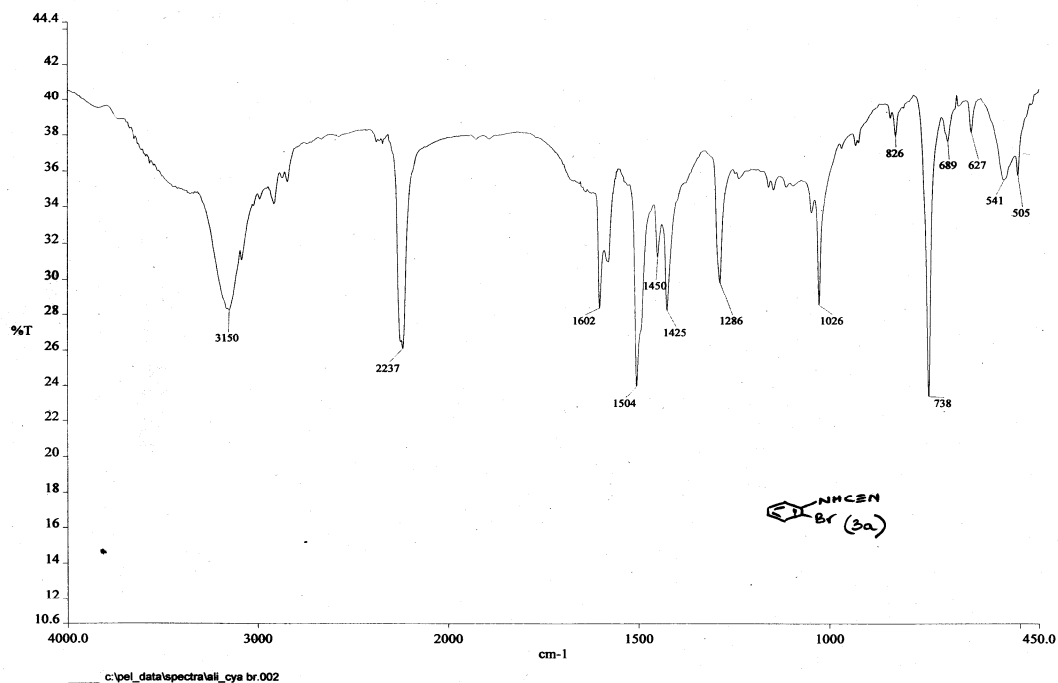
**2-Bromo phenyl cyanamide (3a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):**



**2-Bromo phenyl cyanamide (3a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):**

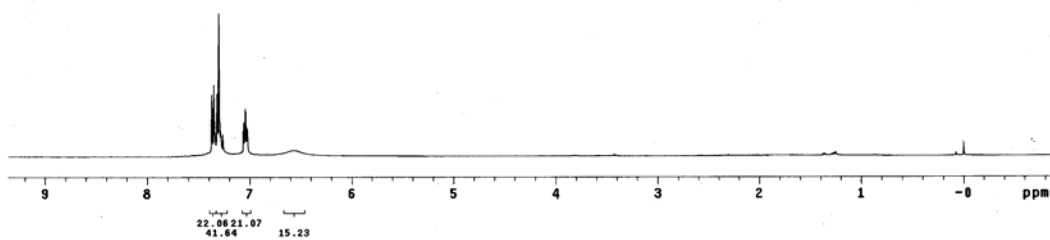
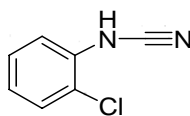


2-Bromo phenyl cyanamide (3a): IR (KBr):



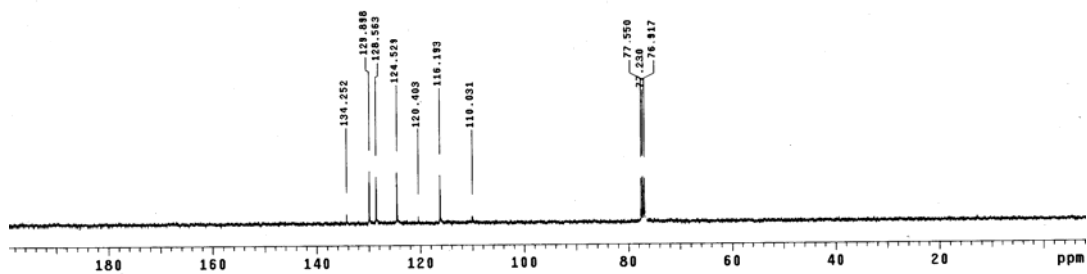
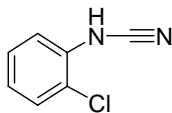
2-Chloro-phenyl cyanamide (4a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):

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file            exp    spin   not used
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sv             6308.8   pvs0   15.700
at            1.980   a1fa   25.000
np            25528   FLAGS
fb            not used 11      n
bs            not used 4      in    n
d1            1.000   dp     y
nt            32     hs     no
ct
TRANSMITTER    32     lb     0.10
tn            H1     fn     65538
sfrq          399.853  sp     DISPLAY -353.5
tcf           382.8   sp
tpwr          57     wp     4986.8
pw           9.850   rF1    783.7
DECOUPLER     C13   rp     115.8
dn            0      1p     -32.4
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dms           C     WC     250
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dpc          15900  vs     20
dpc          nm    cdc   ph
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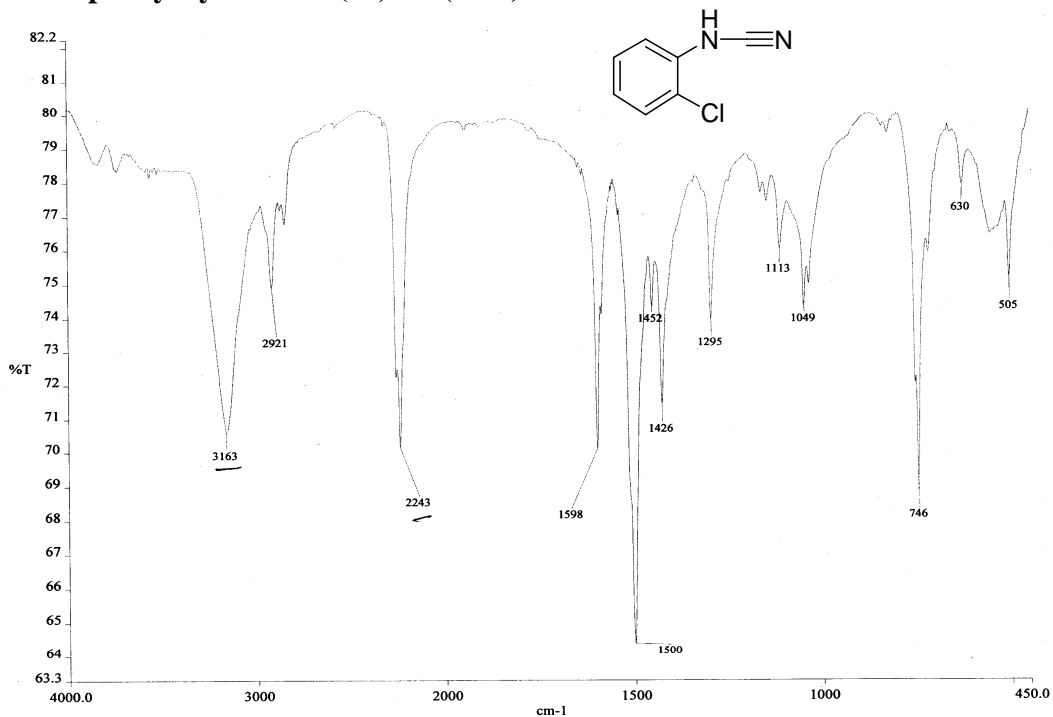


2-Chloro-phenyl cyanamide (4a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):

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ap             1.189   a17a   20.000
np             80270
fb            13800   f1      n
bs             16     f2      n
d1             1.000  dp      y
nt            2000   hs      n
ct             884
TRANSMITTER    C13    fb      2.00
tn            100.554 f2      65536
sfrq          1536.3  sp      -879.7
tof           81     wp      20964.3
tpwr          9.300  rf1     8272.6
pw            DECOUPLER H1     rfp     7764.9
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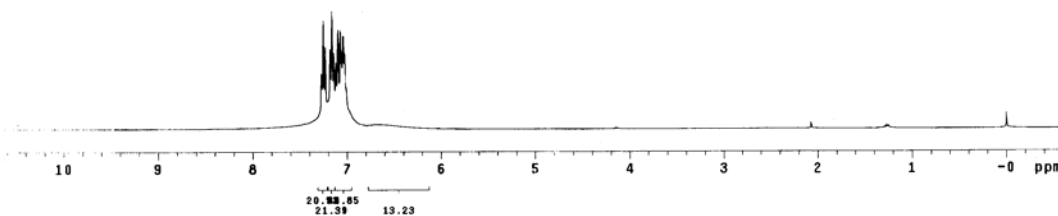
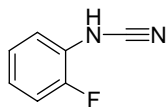
2-Chloro-phenyl cyanamide (4a): IR(KBr):



c:\pe\_data\spectra\rameshry\_1\_284.sp - O-Cl cyanamide

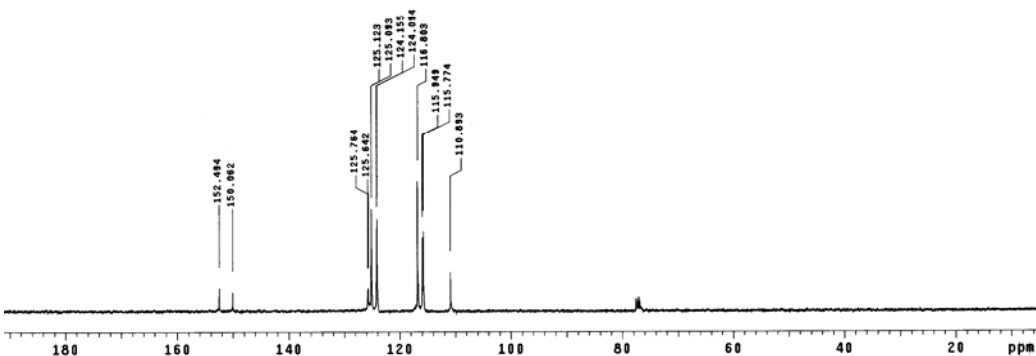
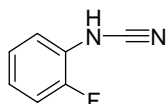
2-Fluoro-phenyl cyanamide (5a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):

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ACQUISITION    exp    spin   not used
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at              1.998  atfa  20.000
np              25528
fb              not used  i1     n
bs              4       in     n
d1              1.000  dp    y
nt              32     hs    nn
ct
TRANSMITTER    32     lb     0.10
tr              H1     fn     65536
sfrq           399.853  sp     -254.7
tof            57     wp     4580.7
tpwr           8.850  r1     782.1
pw
DECOUPLER     C13    rp     0
dn             0       rfp    0
dof            0       rp     137.0
dm             nnn    lp     -85.1
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dpt            15900  vs     28
dqt            nm     th     20
nm             cdc  ph
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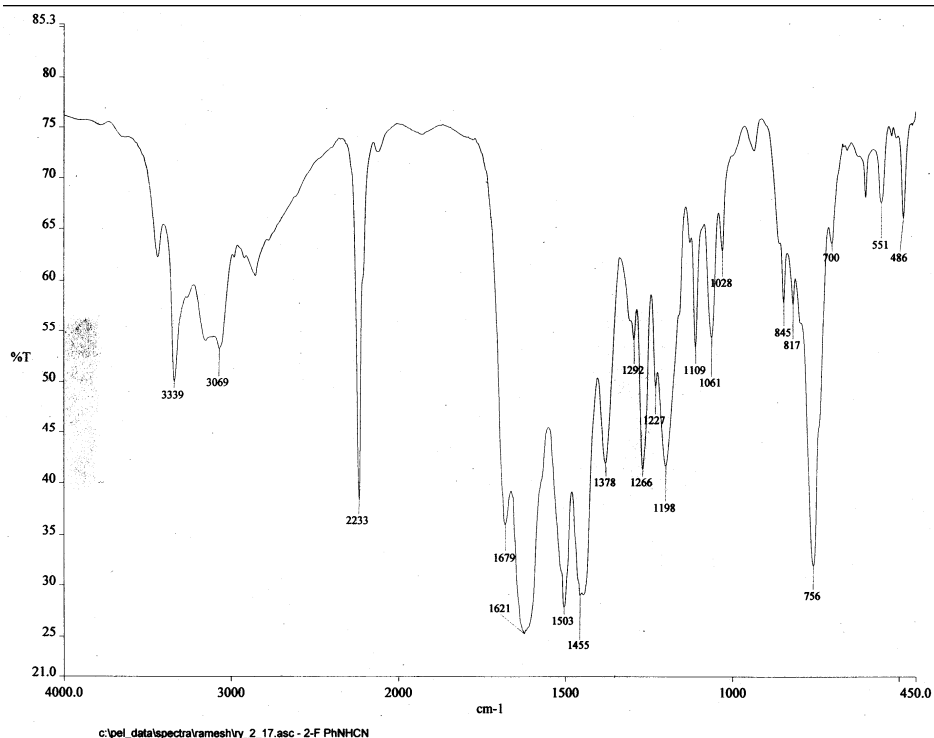


2-Fluoro-phenyl cyanamide (5a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):

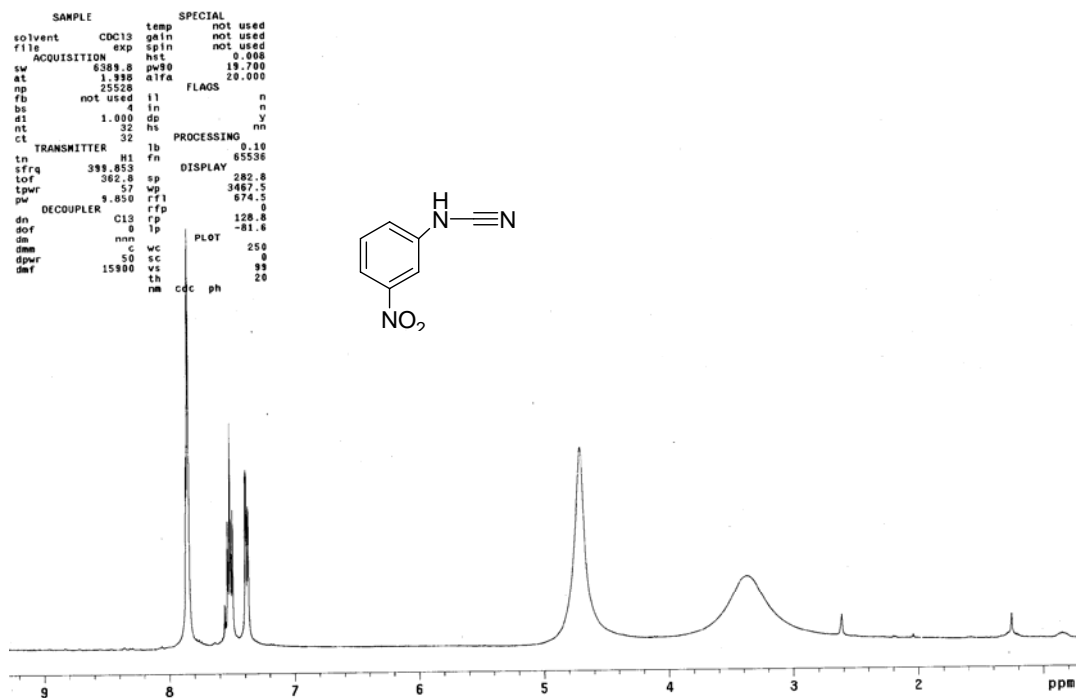
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file            /export/home/- spin   not used
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at              1.138  i1     n
np              60270  in     n
fb              13000  op    y
bs              16     hs    nn
d1              1.000  dp    nn
nt              2000  lb     2.00
ct
TRANSMITTER    224    fn     65536
tr              C13    sp     508.9
sfrq           100.554  wp     18868.0
tof            1536.3  r1     3286.4
tpwr           61     rfp    7764.9
pw             9.300  rp     -86.8
DECOUPLER     H1     lp     -271.4
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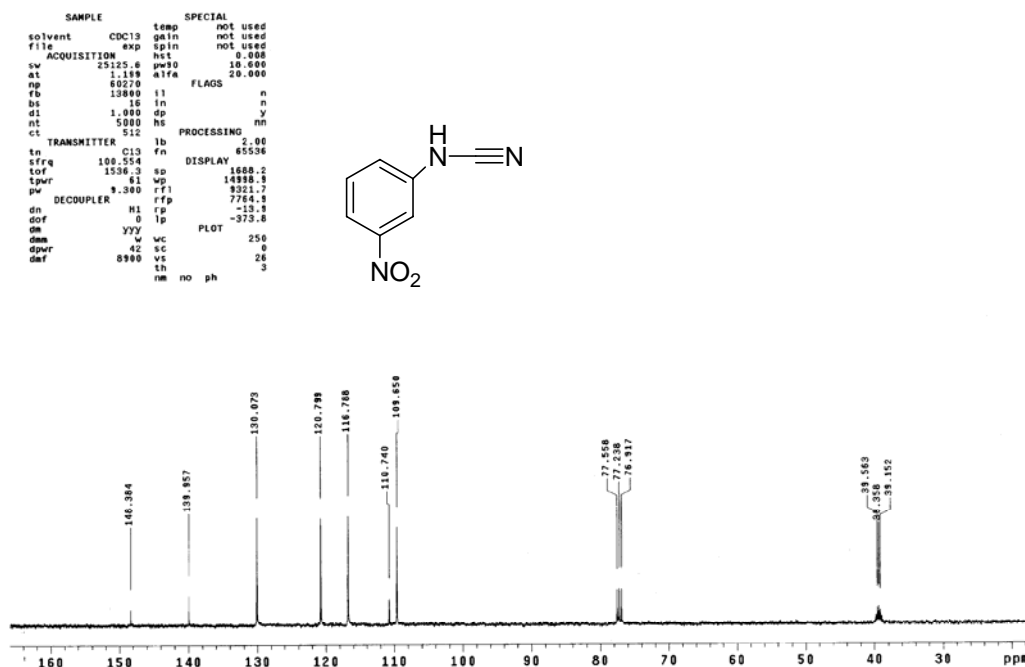
**2-Fluoro-phenyl cyanamide (5a): IR (KBr):**



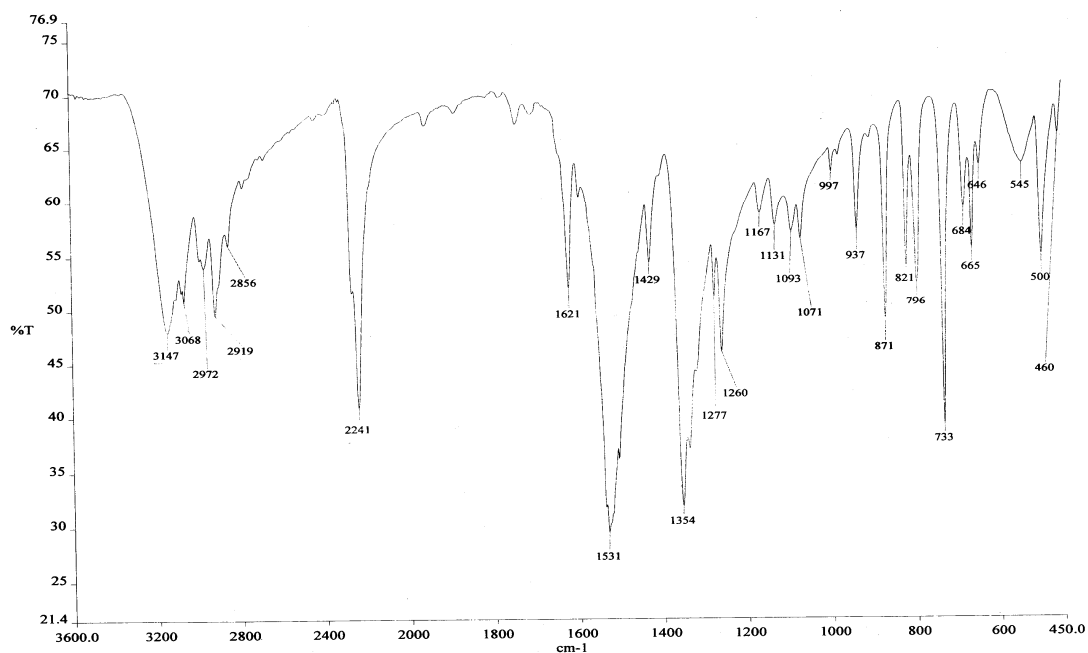
**3- Nitrophenylcyanamide (6a): <sup>1</sup>HNMR (CDCl<sub>3</sub> + DMSO, 400MHz):**



3-Nitro-phenyl cyanamide (6a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3 + \text{DMSO}$ , 100 MHz):



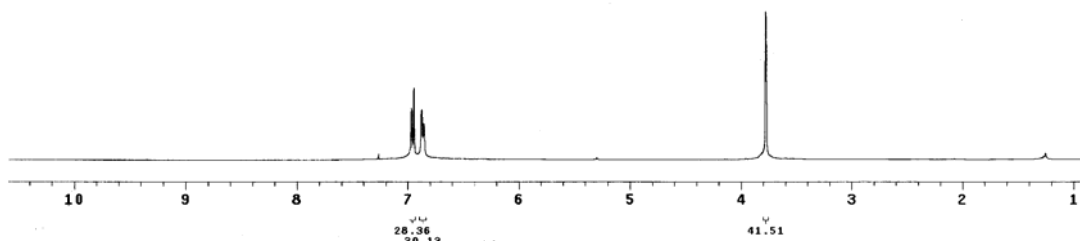
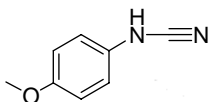
3-Nitro-phenyl cyanamide (6a): IR (KBr):



c:\pel\_data\spectra\4hg\_nitro\_cyanamide.003

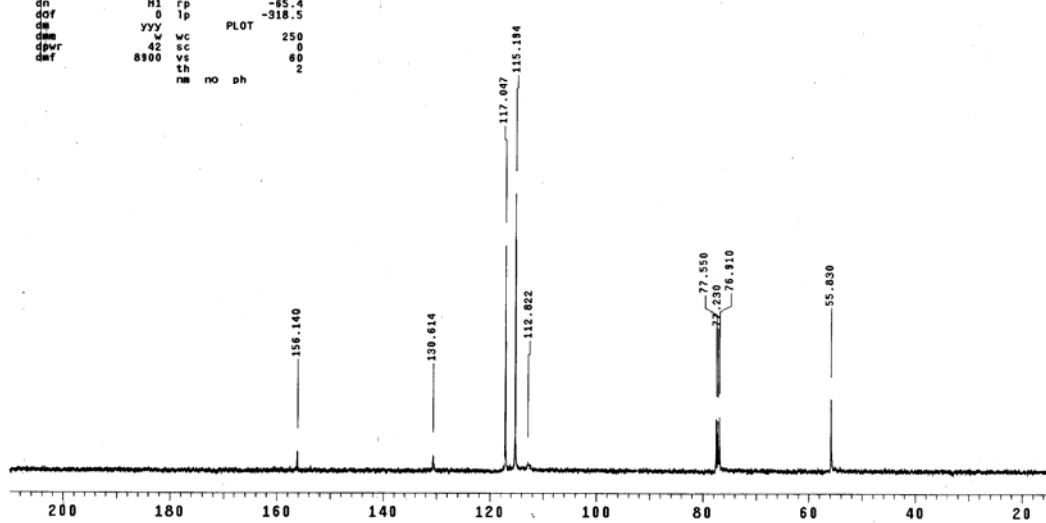
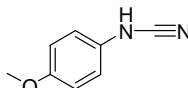
4-Methoxy-phenyl cyanamide (7a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):

```
SAMPLE          SPECIAL
solvent         CDC13   temp   not used
file            /export/home/... gain   not used
patel/RV_2_05.fid hst    not used
ACQUISITION    pw90    0.008
sw             6389.8  a1fa   20.000
at            1.998
np            25528  11
fb            not used 1n
bs            4      dp
di            1.000  hs
nt            32
ct            32
TRANSMITTER    H1      fn
tn            389.853 sp
sfrq          362.8  wp
tof           57     rfl
tpwr          9.850  rfp
pw            127.4
DECOUPLER     C13    1p
dn            0      PLOT
dof           0
dm            nnn   wc
dpcr          50    vs
dwr           15900 th
dfr            20
```

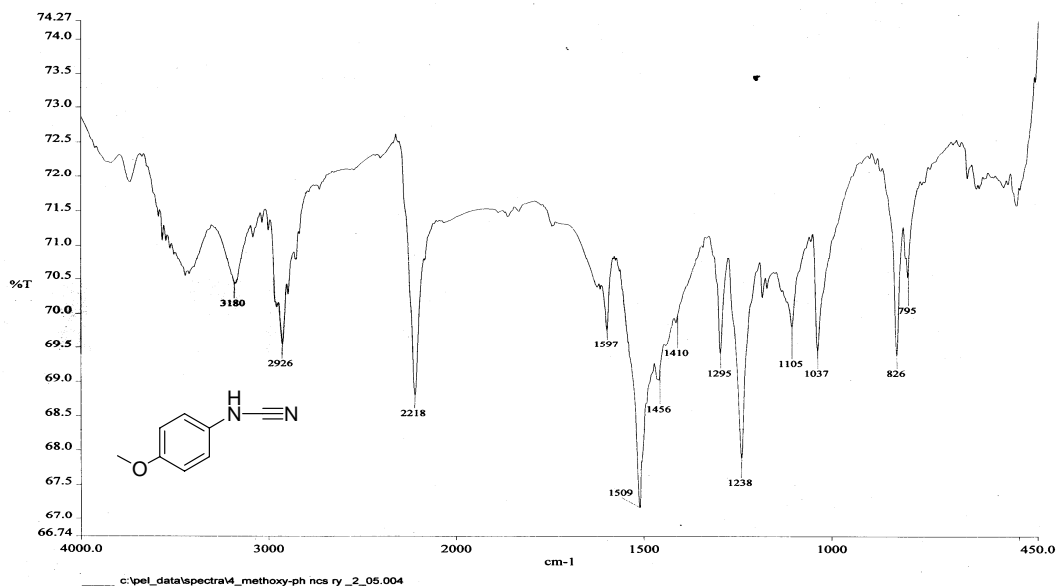


4-Methoxy-phenyl cyanamide (7a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):

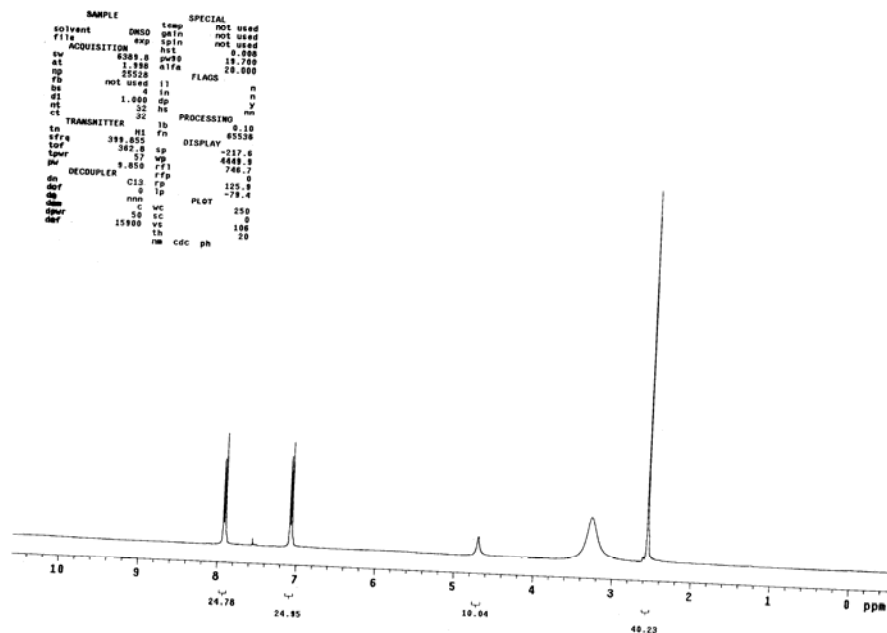
```
SAMPLE          SPECIAL
solvent         CDC13   temp   not used
file            /export/home/... gain   not used
patel/RV_2_05.fid hst    not used
ACQUISITION    pw90    0.008
sw             25125.6 pw90   18.000
at            1.199  a1fa   20.000
np            60220
fb            13800  11
bs            16     1n
di            1.000  dp
nt            2000  hs
ct            1056
TRANSMITTER    C13    fn
tn            100.554 sp
sfrq          1538.3 wp
tof           61     rfl
tpwr          9.300  rfp
pw            9274.1
DECOUPLER     H1      1p
dn            0      PLOT
dof           0
dm            yyw   wc
dpcr          42    vs
dwr           8900  th
dfr            2
```



**4-Methoxy-phenyl cyanamide (7a): IR (KBr):**

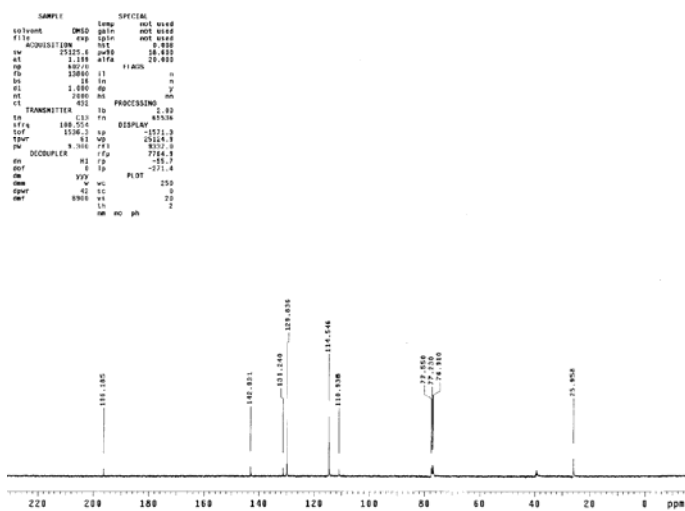


**4-Acetyl-phenylcyanamide (8a): <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO, 400 MHz):**

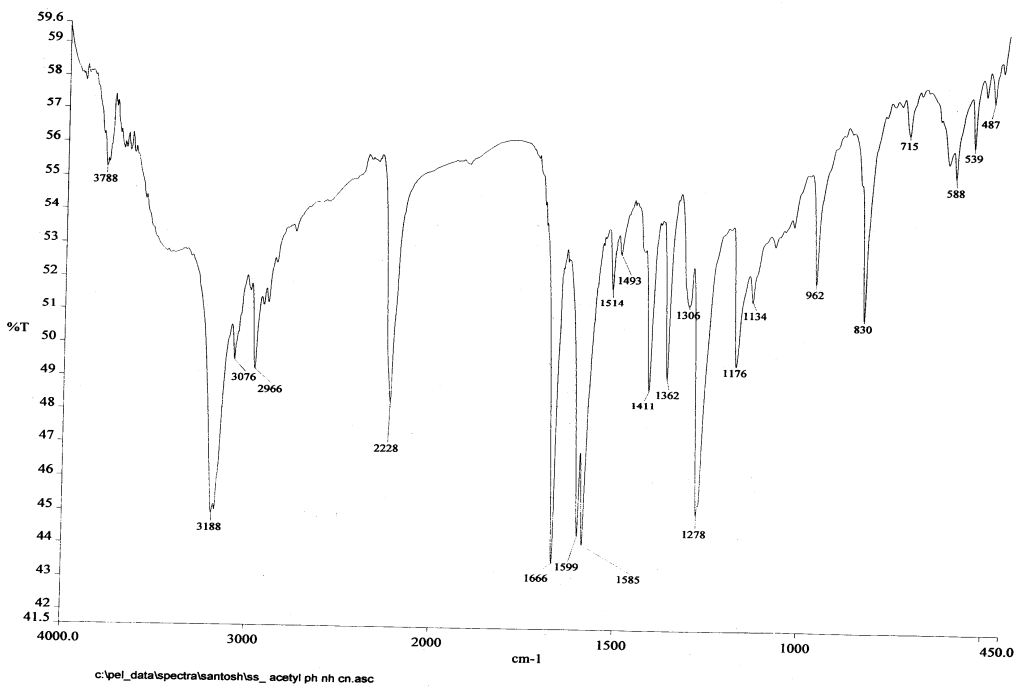




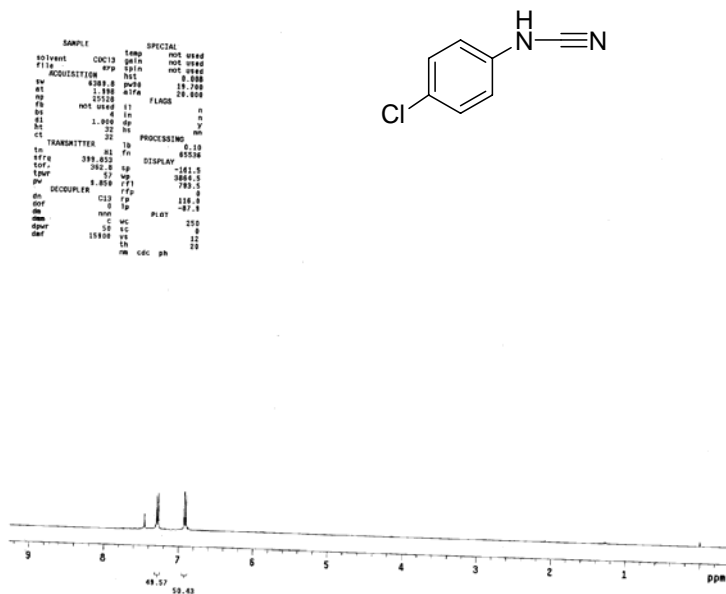
**4-Acetyl-phenylcyanamide (8a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$  + DMSO, 100 MHz):**



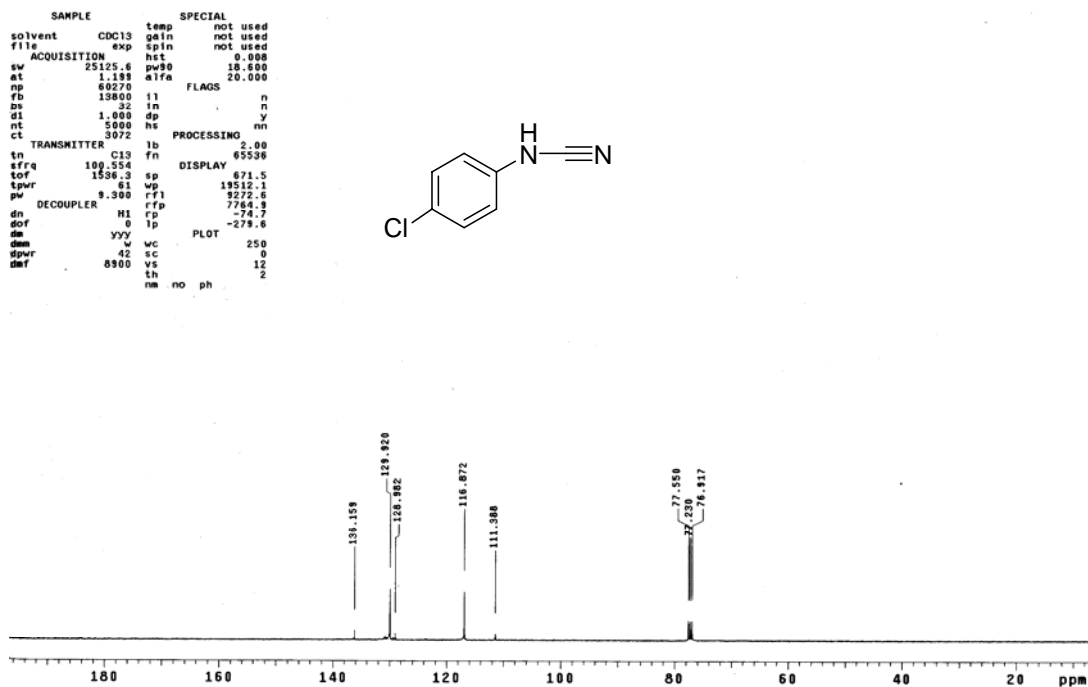
**4-Acetyl-phenylcyanamide (8a): IR (KBr):**



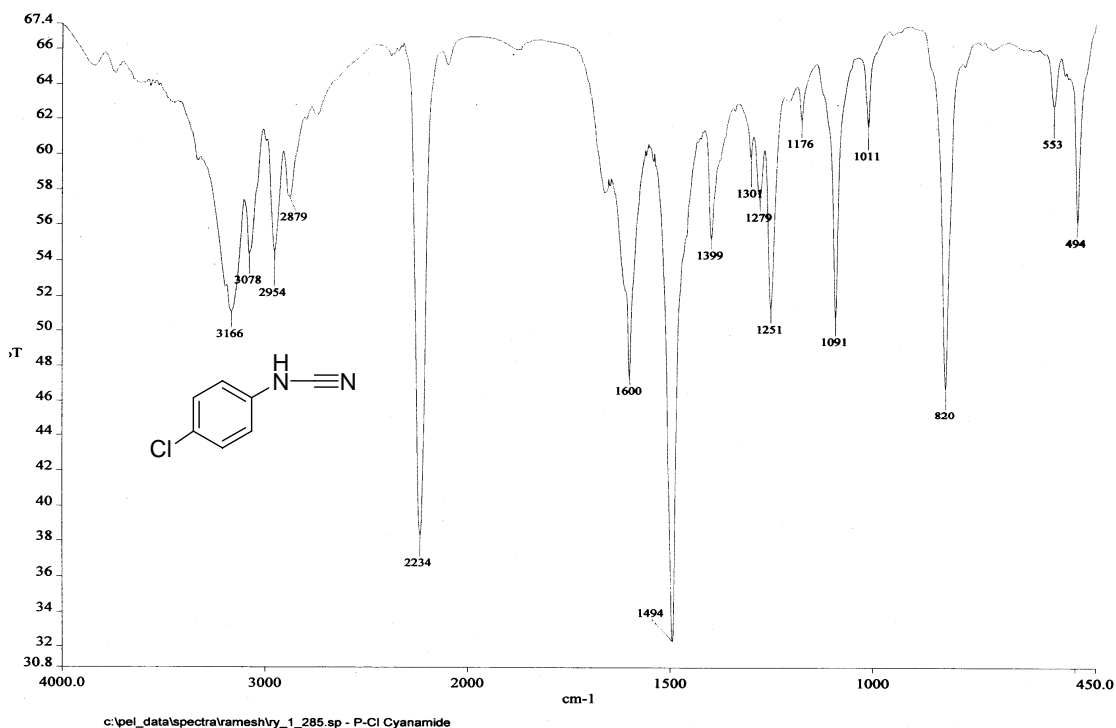
4-Chloro-phenyl cyanamide (9a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):



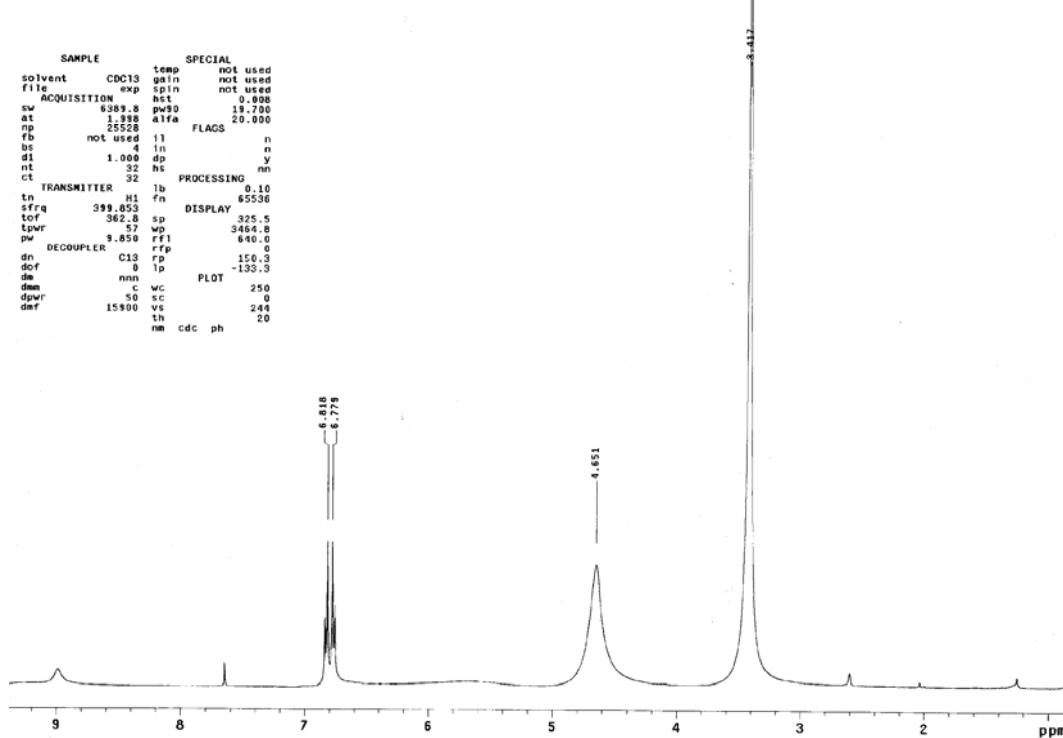
4-Chloro-phenyl cyanamide (9a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



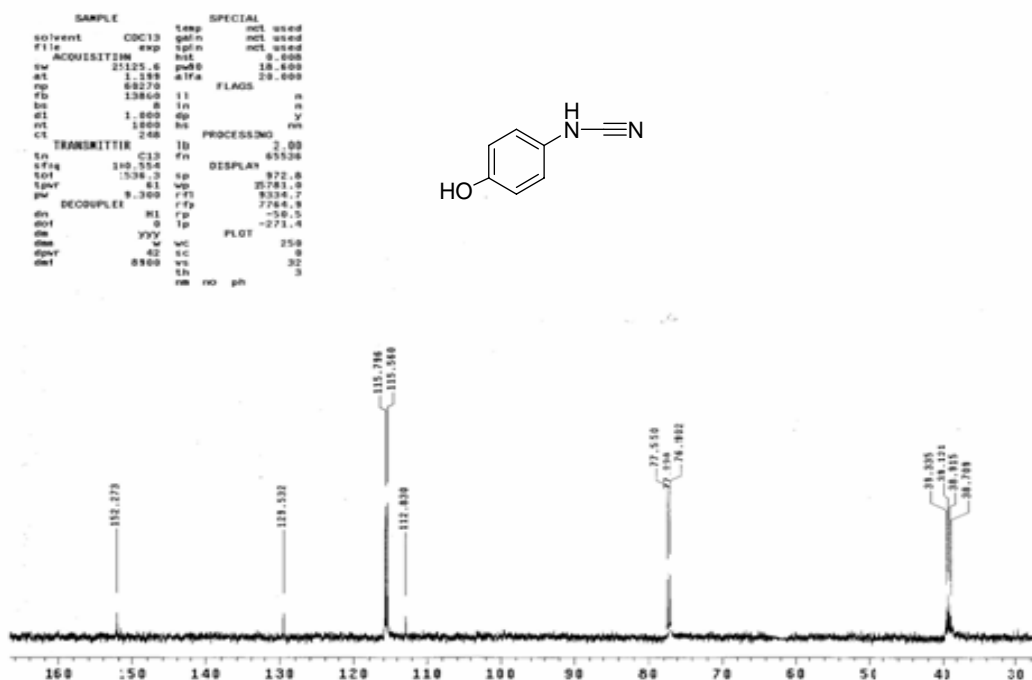
4-Chloro-phenyl cyanamide (9a): IR (KBr):



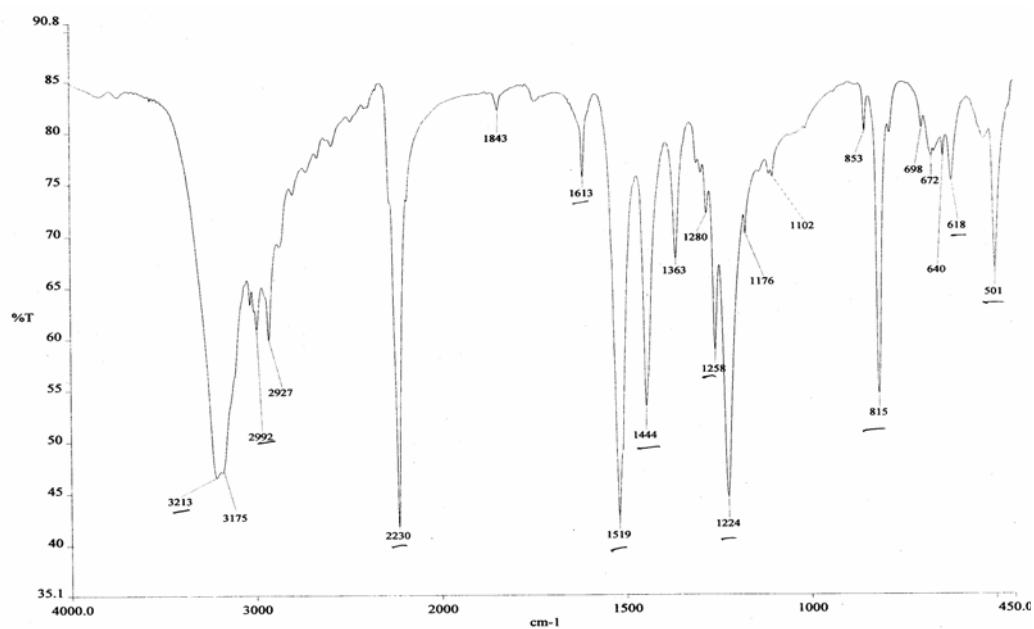
4-Hydroxy-phenyl cyanamide (10a): <sup>1</sup>H NMR (CDCl<sub>3</sub> + DMSO, 400 MHz):



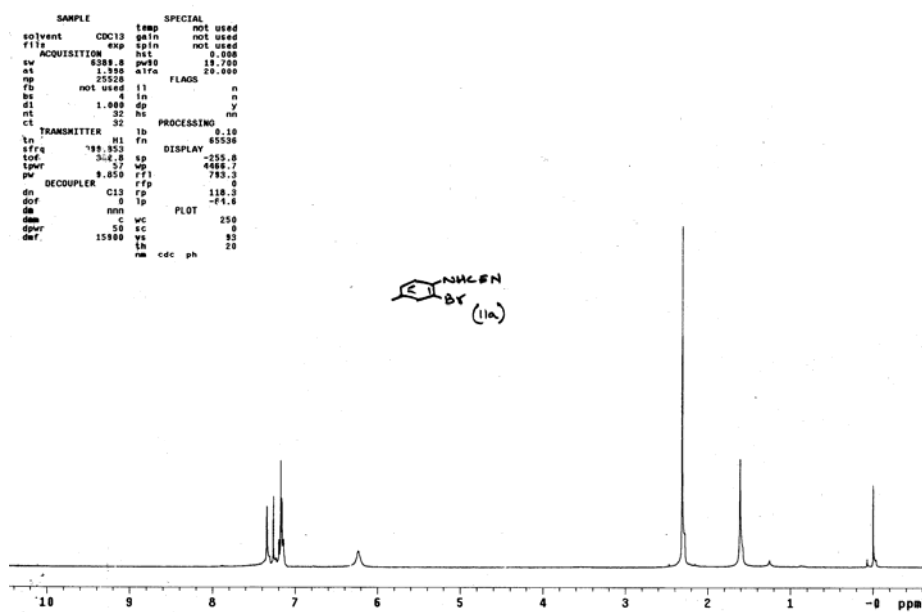
**4-Hydroxy-phenyl cyanamide (10a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3 + \text{DMSO}$ , 100 MHz):**



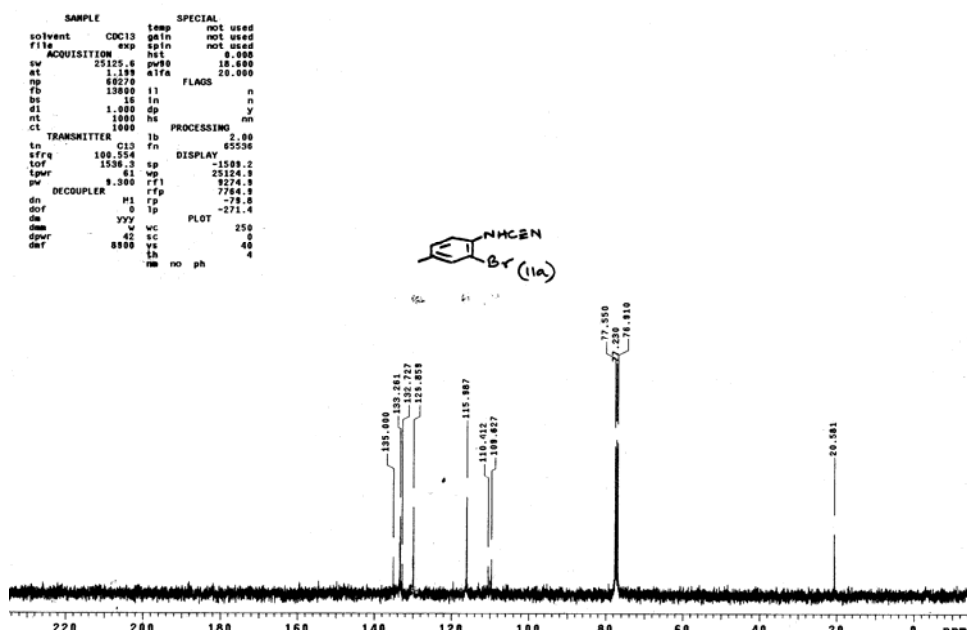
**4-Hydroxy-phenyl cyanamide (10a): IR (KBr):**



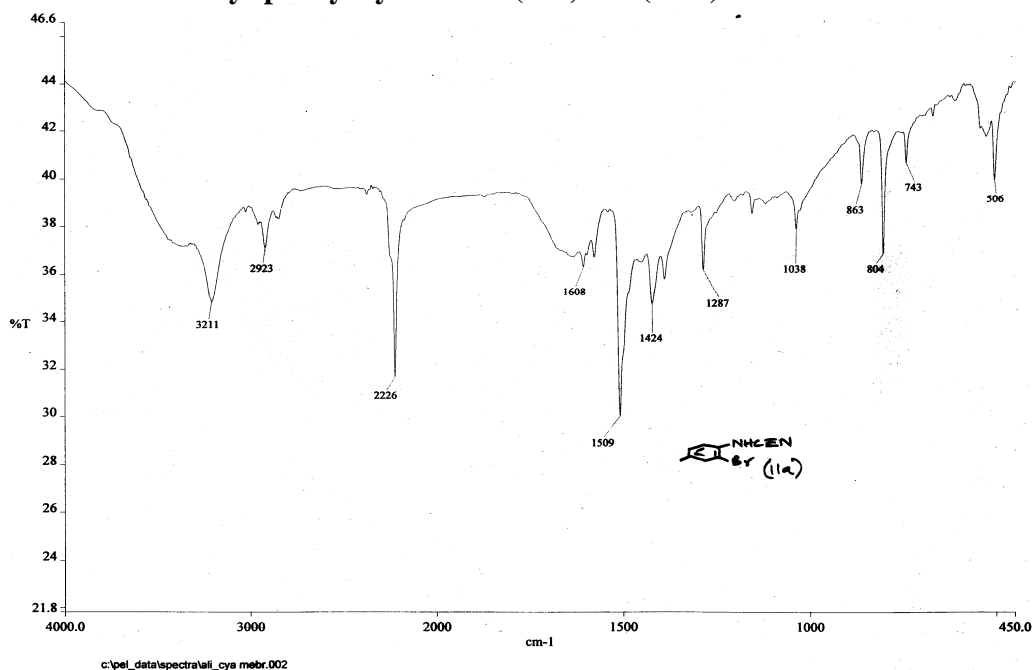
2-Bromo-4-Methyl-phenyl cyanamide (11a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):



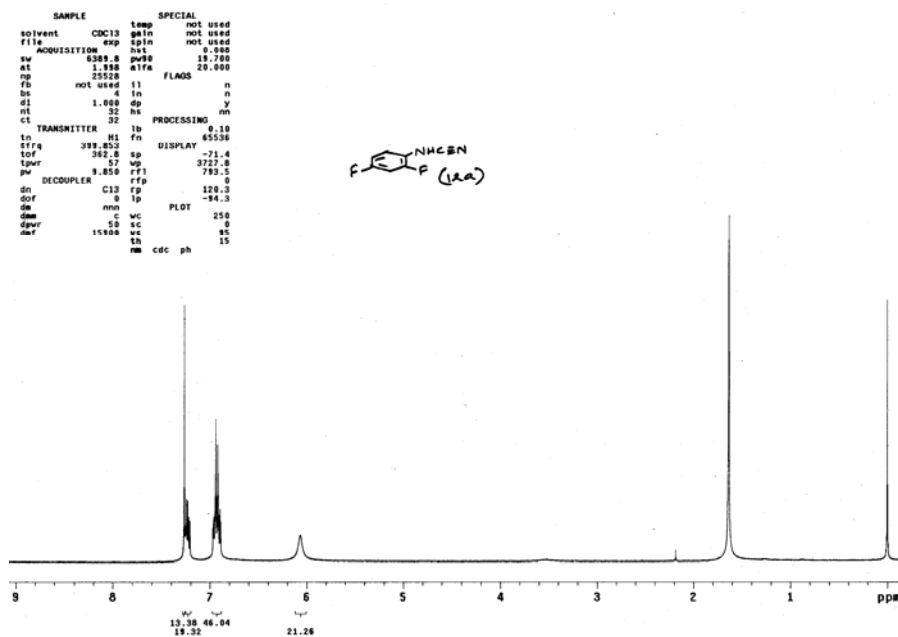
2-Bromo-4-Methyl-phenyl cyanamide (11a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



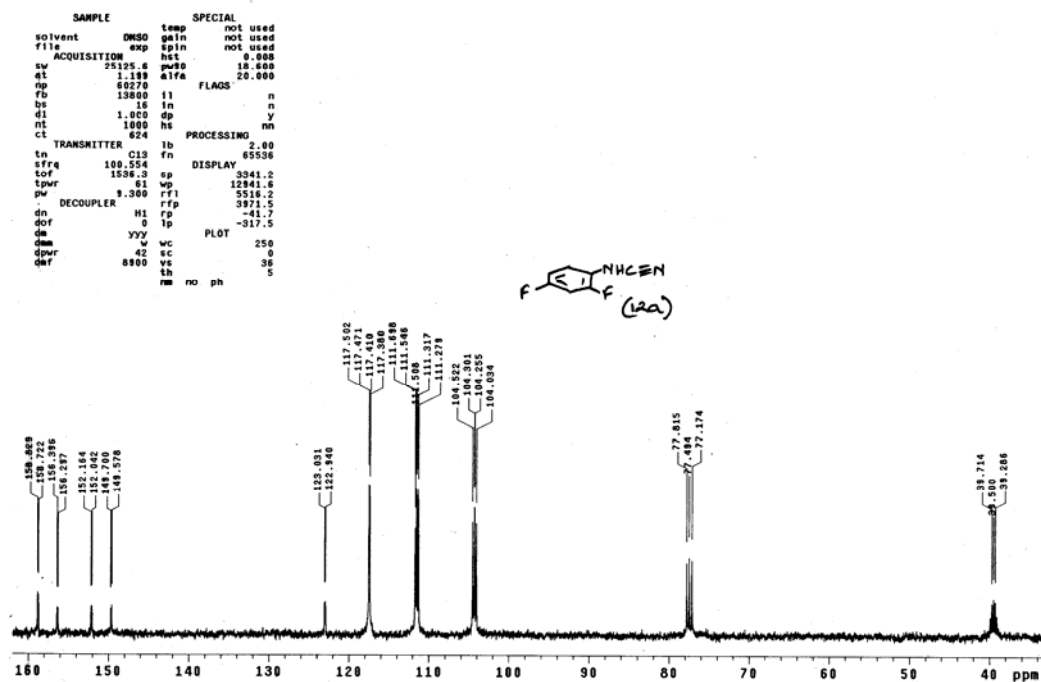
**2-Bromo-4-Methyl-phenyl cyanamide (11a): IR (KBr):**



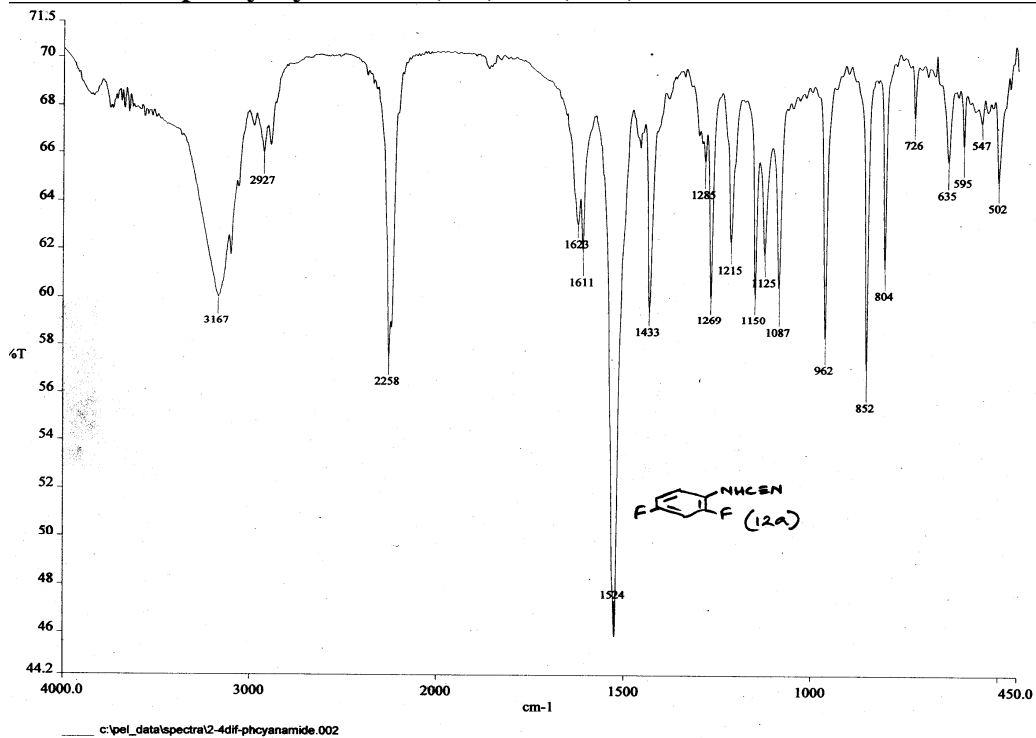
**2,4-Difluoro phenyl cyanamide (12a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):**



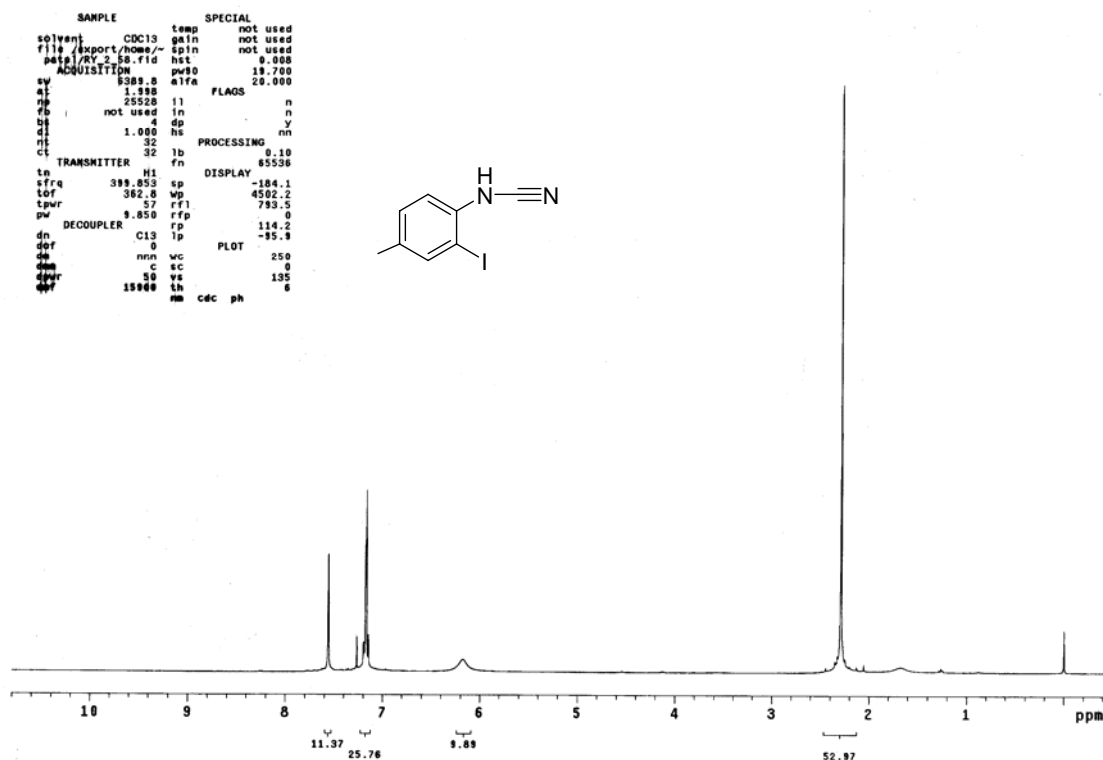
2-4-Difluoro phenyl cyanamide (12a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



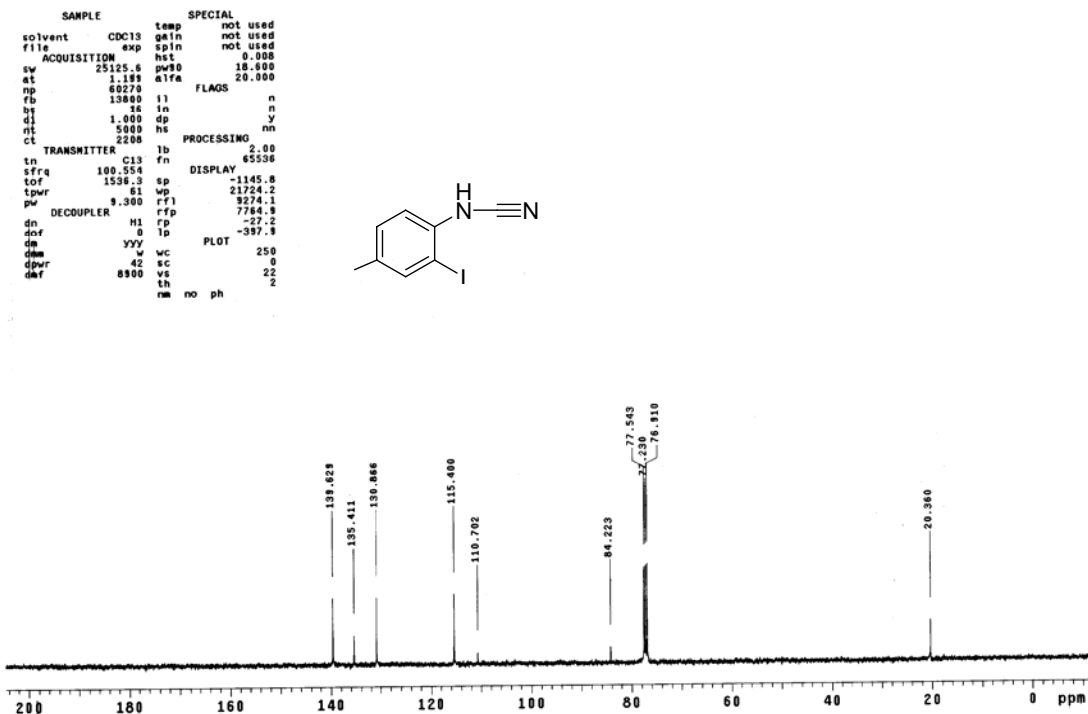
2-4-Difluoro phenyl cyanamide (12a): IR (KBr):



2-Iodo-4-methyl-phenyl cyanamide (13a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):

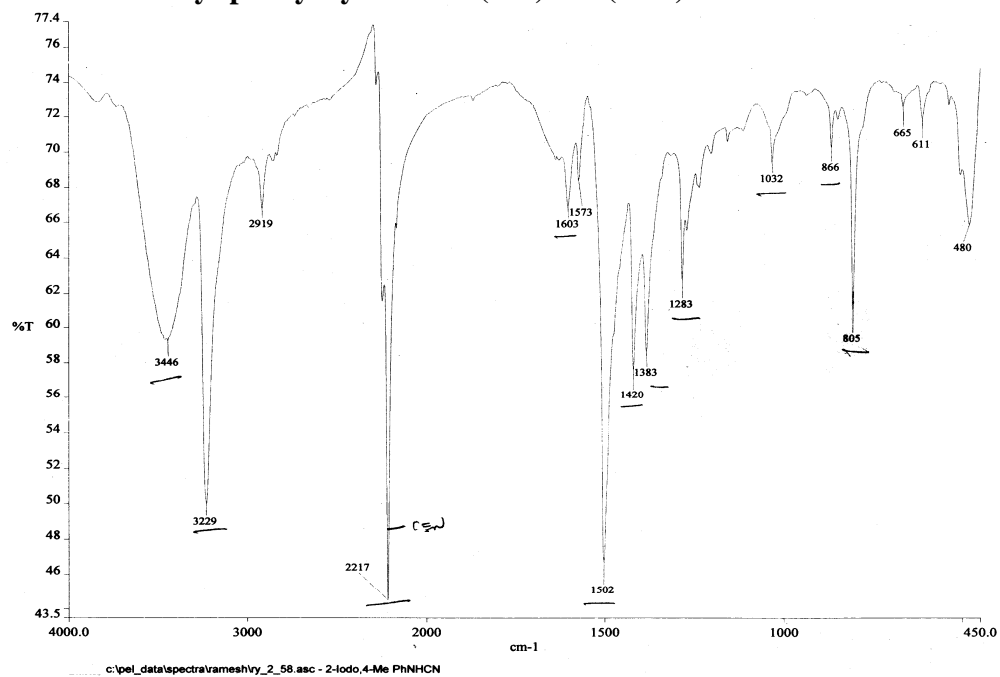


2-Iodo-4-methyl-phenyl cyanamide (13a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):

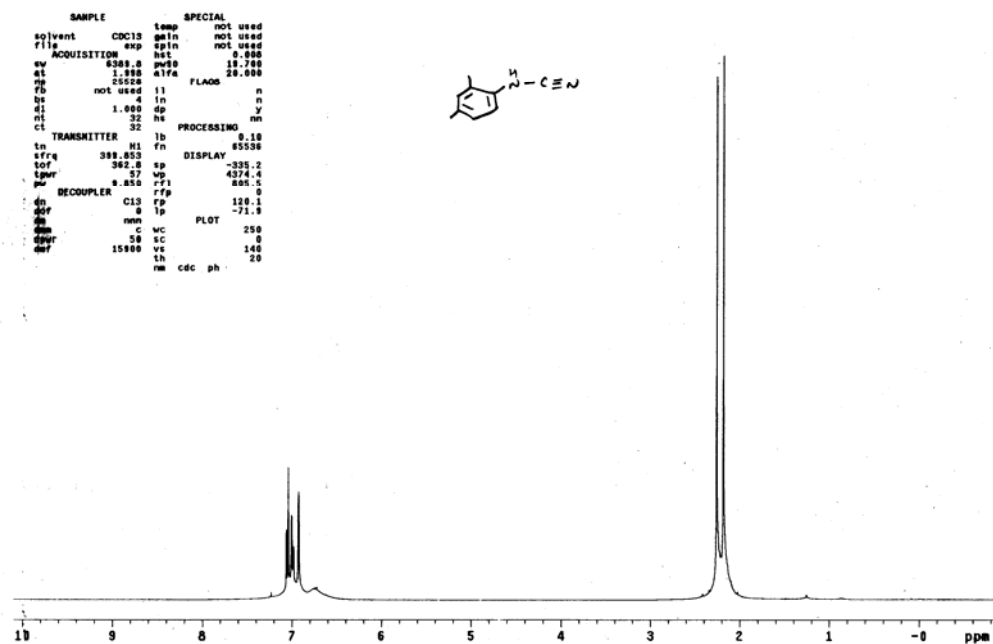




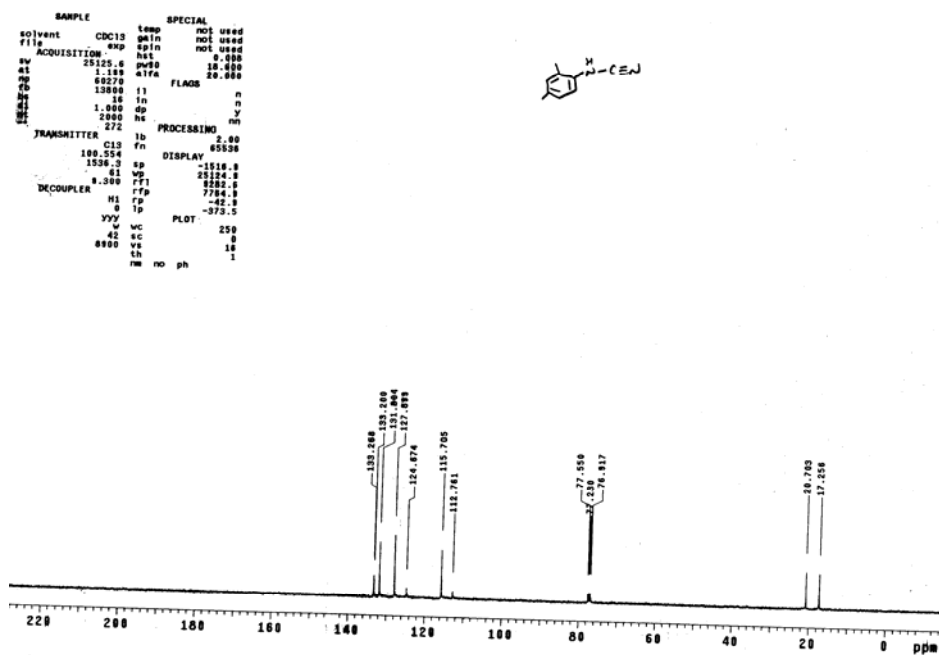
**2-Iodo-4-methyl-phenyl cyanamide (13a): IR (KBr):**



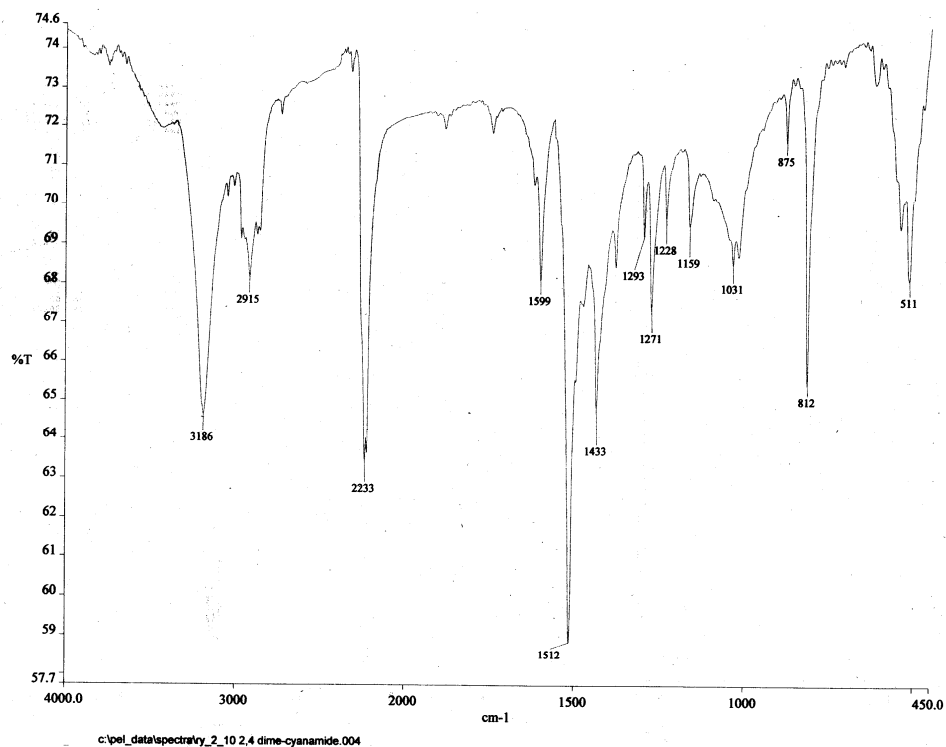
**2,4-Dimethyl-phenyl cyanamide (14a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):**



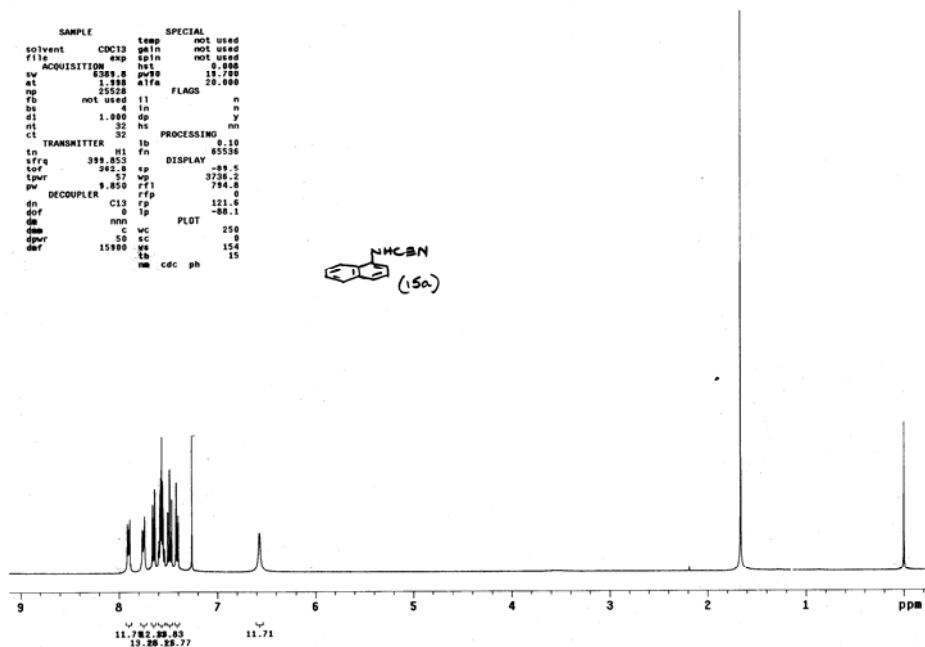
2,4-Dimethyl-phenyl cyanamide (14a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



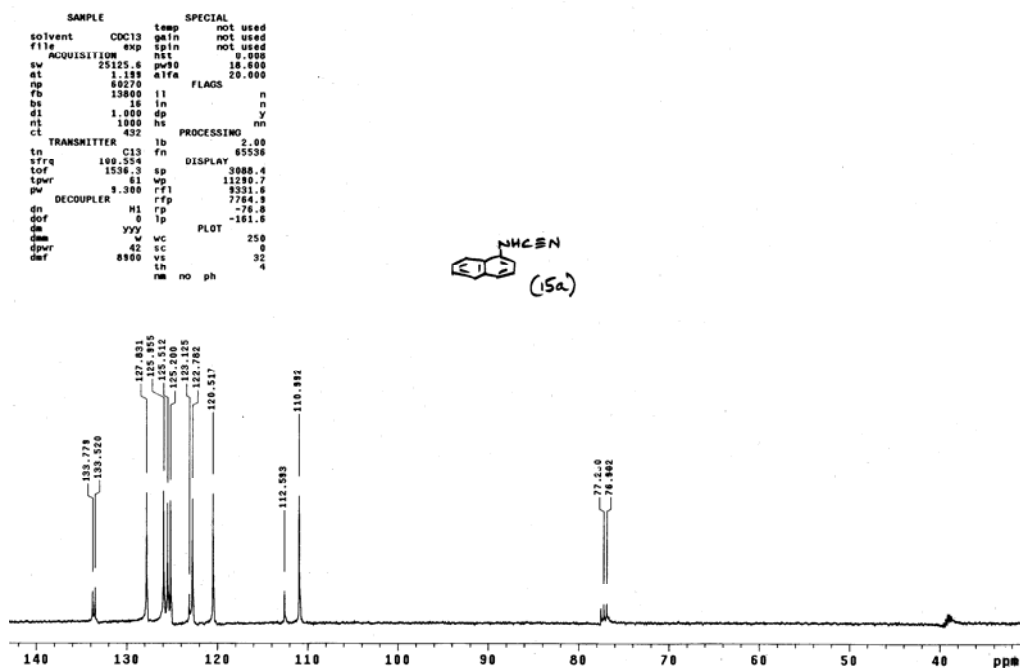
2,4-Dimethyl-phenyl cyanamide (14a): IR (KBr):



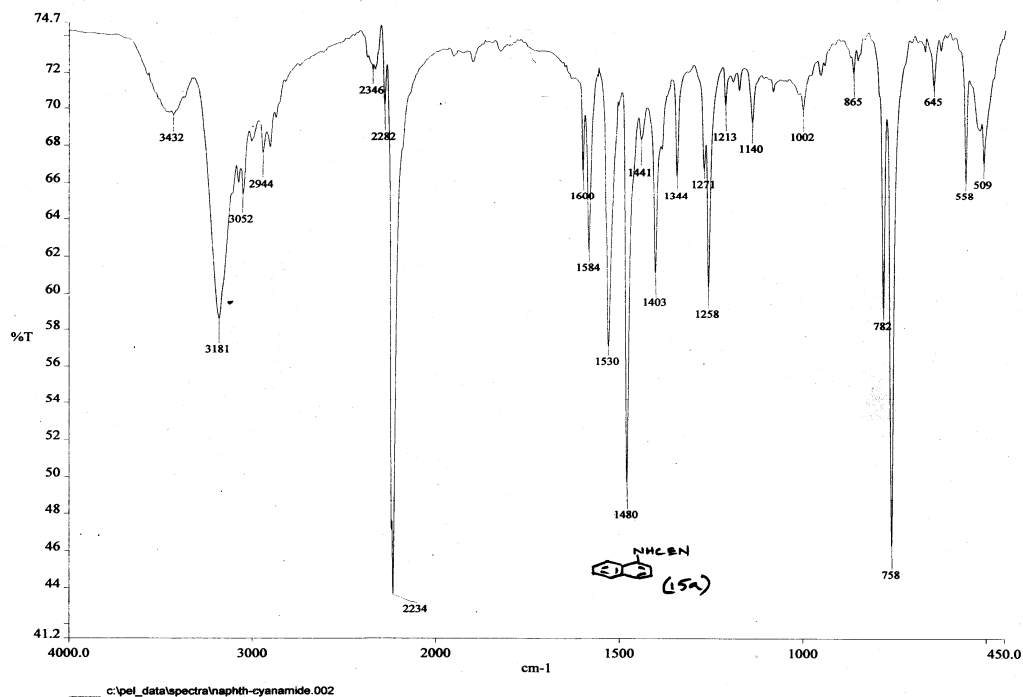
1-Naphthyl cyanamide (15a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):



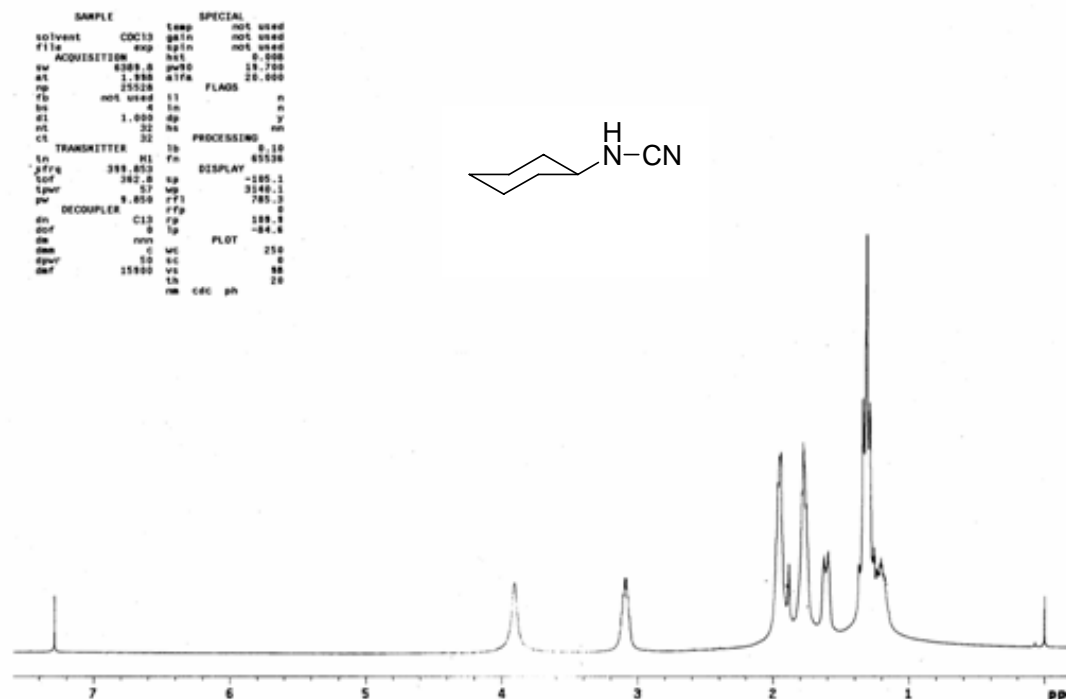
1-Naphthyl cyanamide (15a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



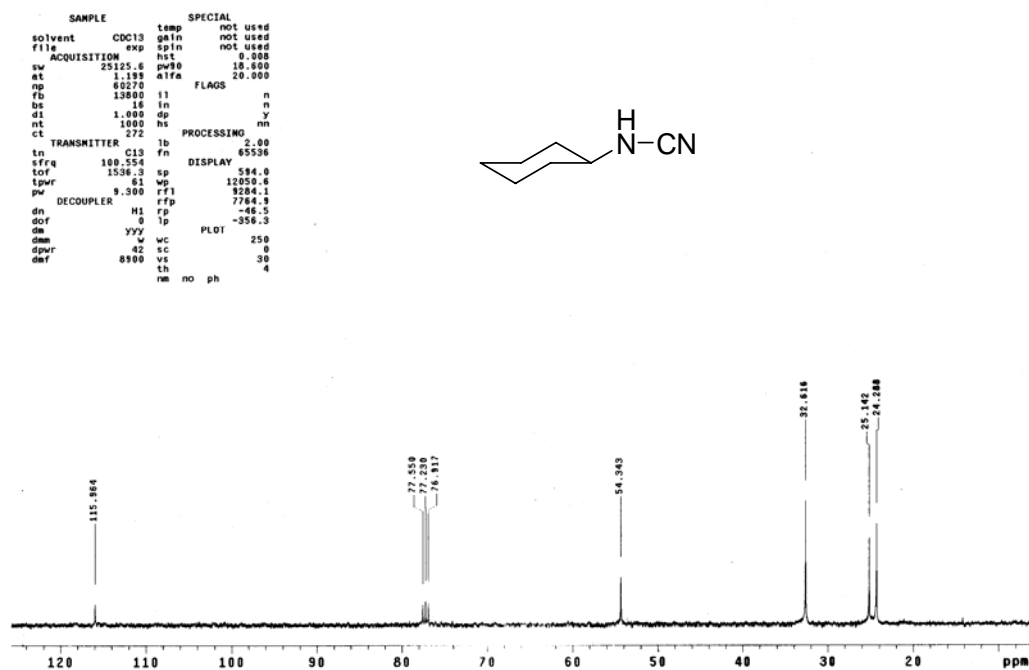
1-Naphthyl cyanamide (15a): IR (KBr):



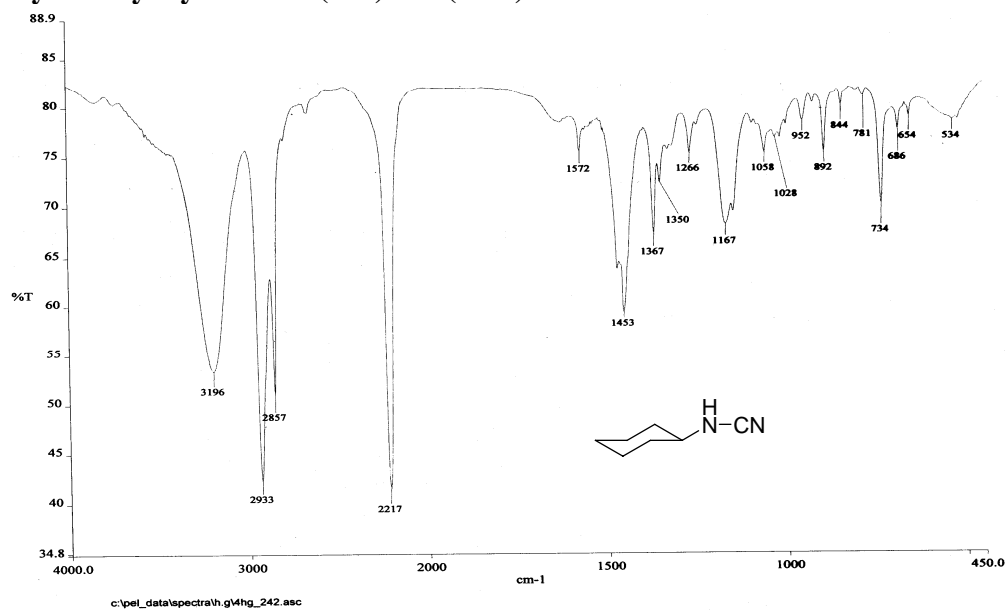
Cyclohexyl-cyanamide (16a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):



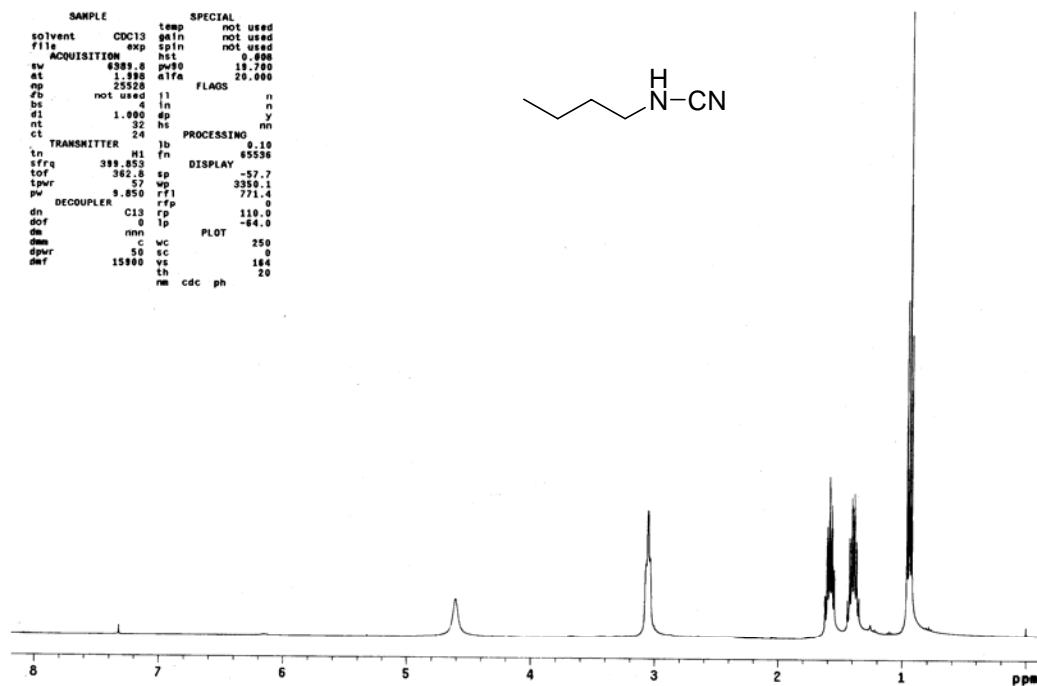
Cyclohexyl-cyanamide (16a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):



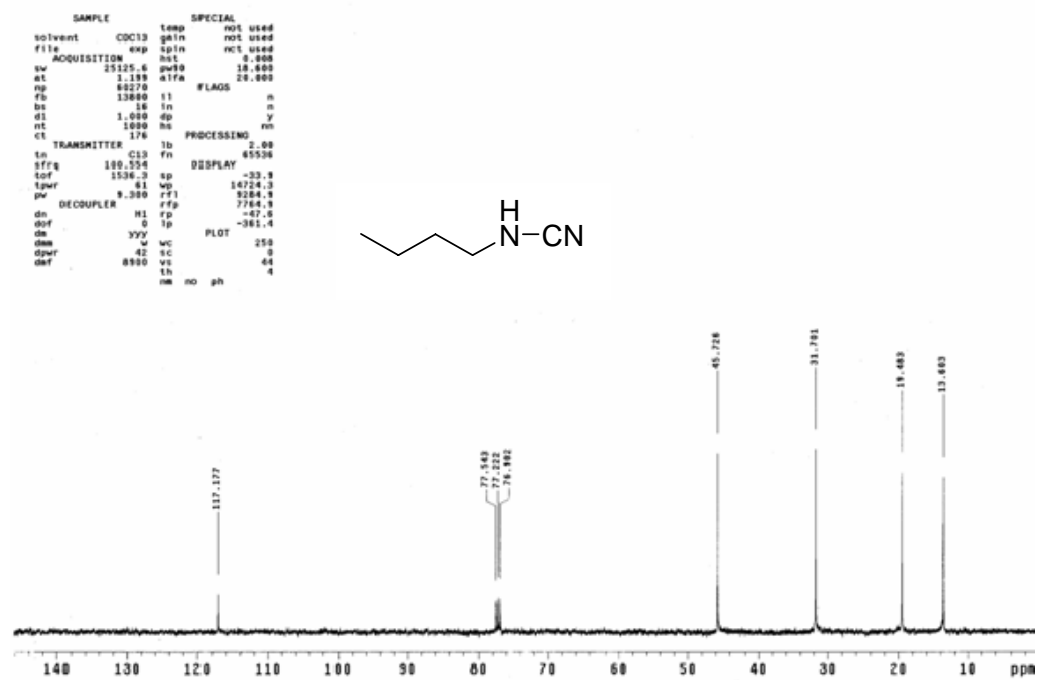
Cyclohexyl-cyanamide (16a): IR (KBr):



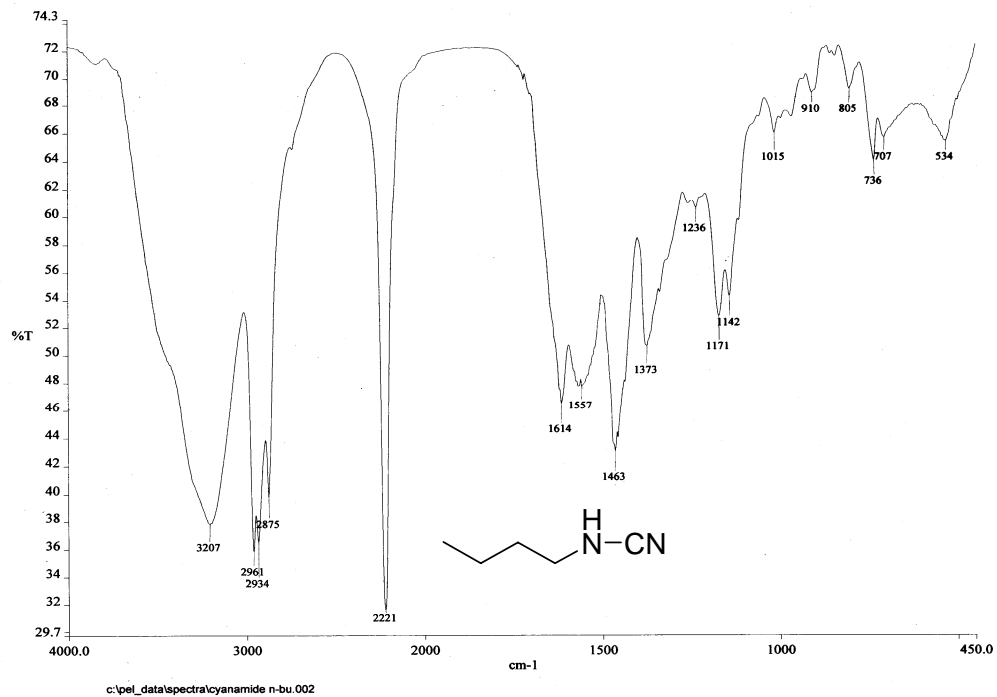
***n*-Butyl-cyanamide (17a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):**



***n*-Butyl-cyanamide (17a): <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):**

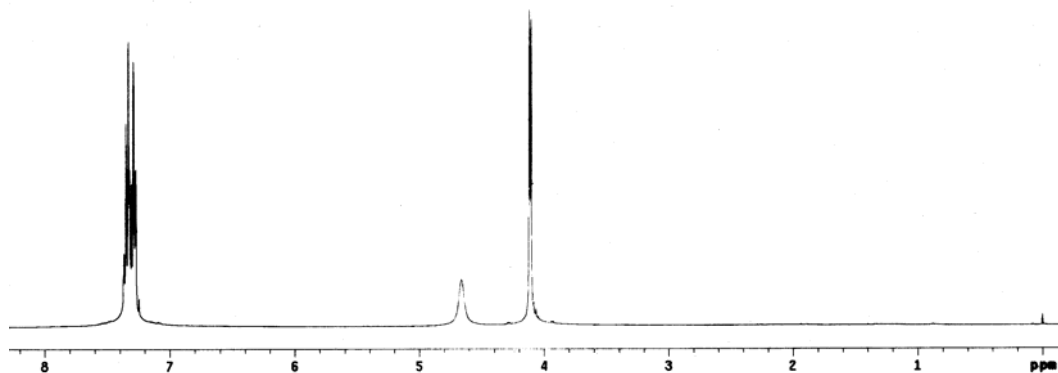
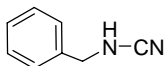


**n-Butyl-cyanamide (17a): IR (KBr):**

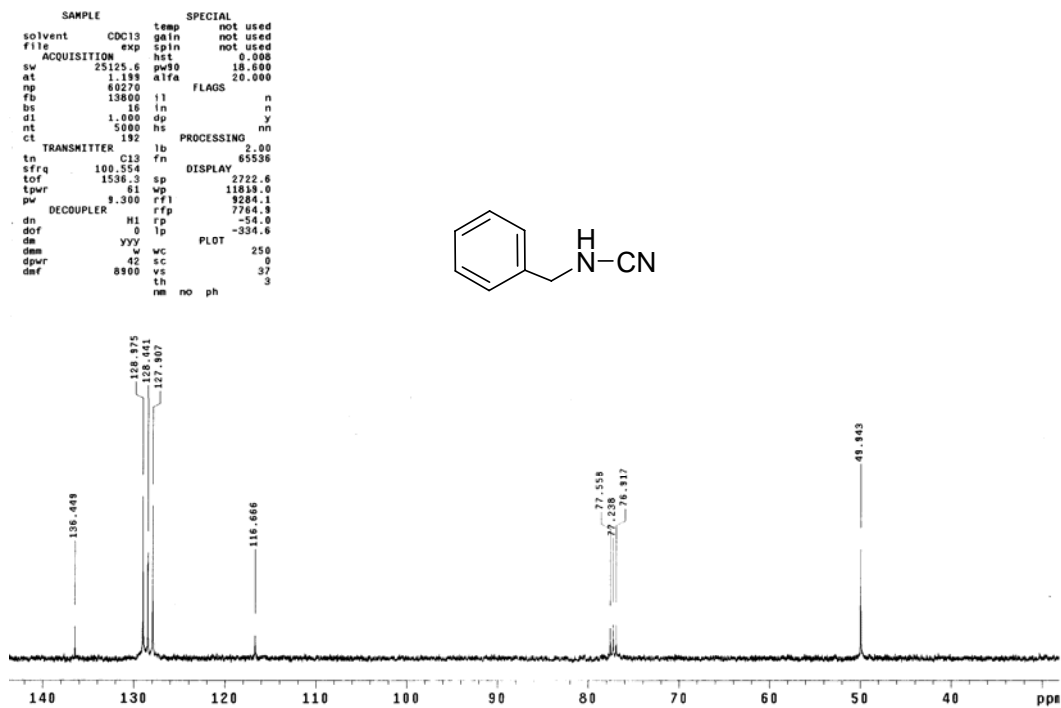


**Benzyl cyanamide (18a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):**

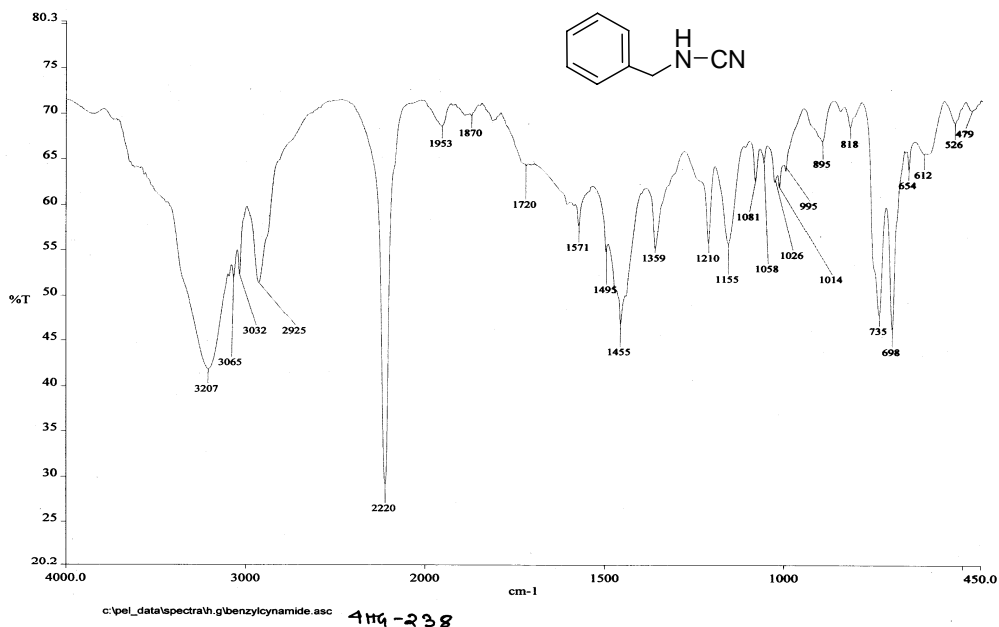
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SAMPLE          SPECIAL
solvent         CDC13   temp   not used
file            exp    spin  not used
ACQUISITION    hst     0.000
sv              6589.8  pw0    18.700
at              1.988  a1fa   20.000
np              25328
fb              not used 11   FLAGS  n
bs              4      in    n
d1              1.000  de    y
nt              32     hs    nn
ct              32
TRANSMITTER     1b    fn    0.10
tn              81     fn    65536
sfr             389.853  DISPLAY
tof             382.8  sp    -61.6
tpwr            57     wp    3408.8
pw              8.850  r1    890.7
DECOUPLER      C13   rp    0
dn              0      rp    115.5
dof             0      lb    -77.7
dm              c
dwm             c      wc    250
dpwr            50     sc    0
dwr             15900  vs    75
dwf             th     ch    20
nm             cdc  ph
```



**Benzyl cyanamide (18a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):**

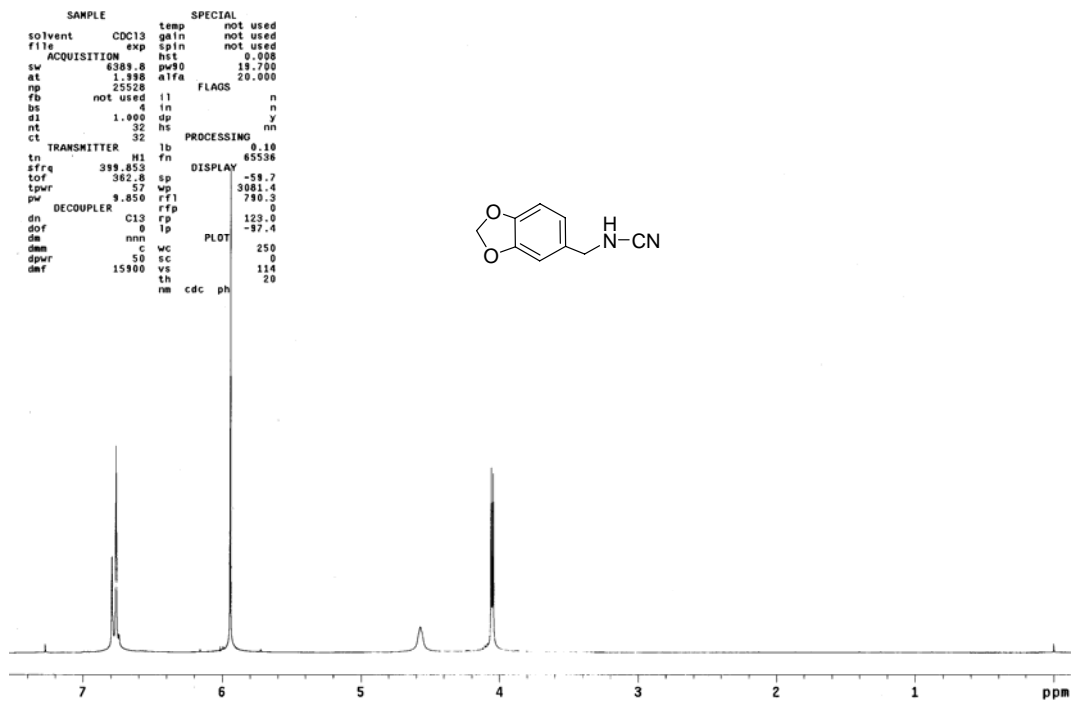


**Benzyl cyanamide (18a): IR (KBr):**

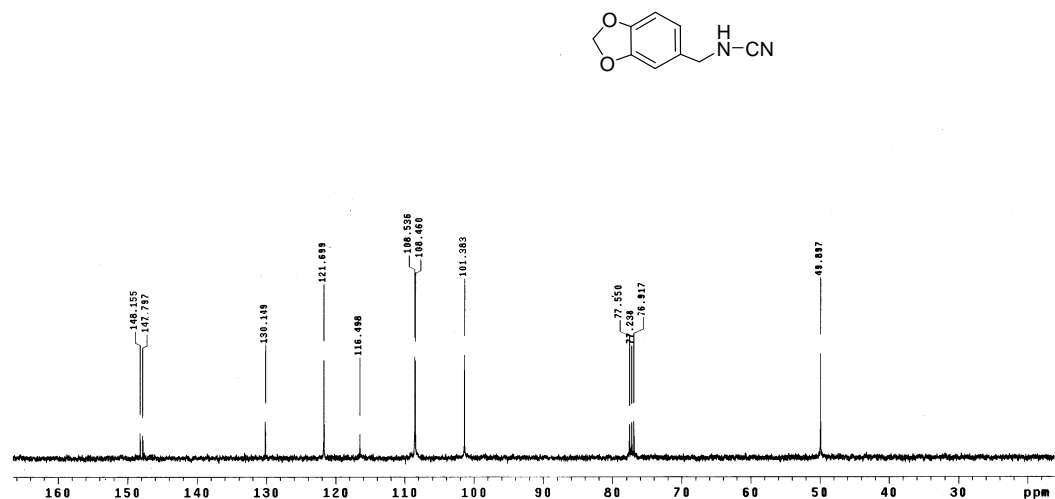




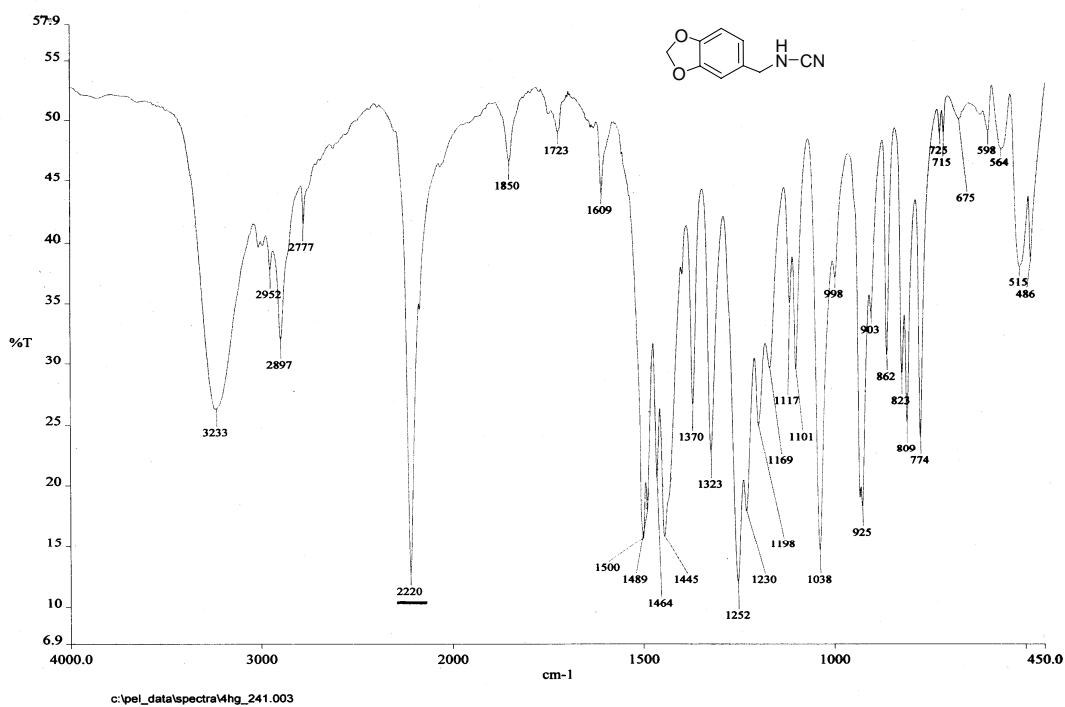
**Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):**



**Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a):  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):**

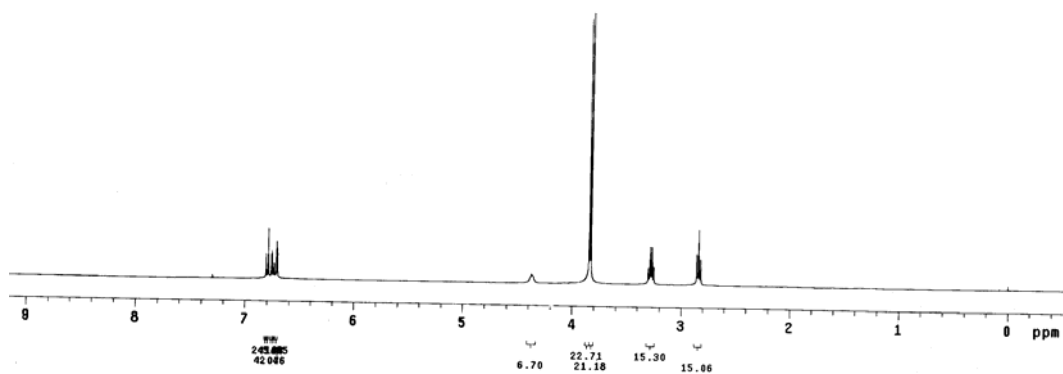
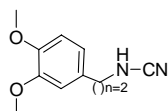


**Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a): IR (KBr):**



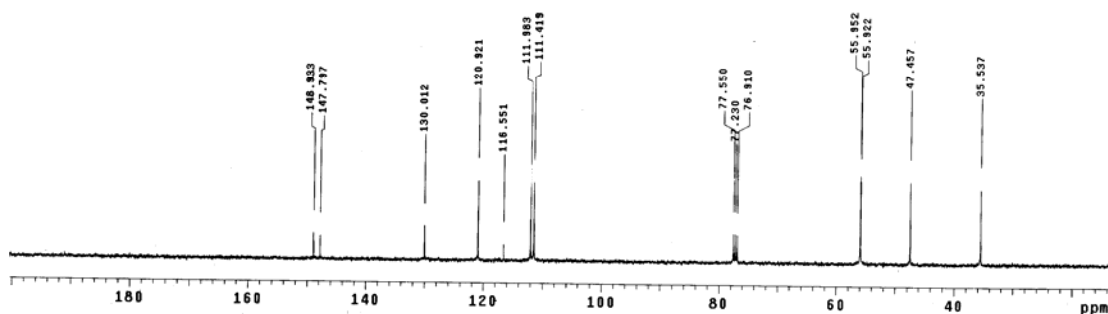
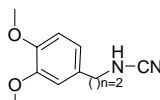
**3,4-Dimethoxyphenylethylcyanamide (20a): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):**

SAMPLE		SPECIAL	
solvent	CDC13	temp	not used
file		gtn	not used
ACQUISITION	exp	spn	not used
sw	6389.6	ht	0.008
at	1.998	pw99	19.700
np	25328	atfa	20.000
fb	not used	l1	n
bs	4	in	n
d1	1.000	dp	y
nt	32	hs	nn
ct			
TRANSMITTER		l1b	0.10
tn	H1	fn	65536
sfrq	398.853	sp	-211.0
tof	382.8	wp	3958.2
tpwr	57	rfl	780.6
pw	9.850	rfp	0
DECOUPLER		rp	127.8
dn	C13	lp	-86.8
dof	0	sc	0
dm	nnn	wc	250
dmm	c	vs	0
dpr	50	th	63
dof	15900	rm	20
			cdc ph

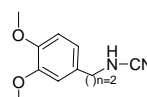


**3,4-Dimethoxyphenylethylcyanamide (20a): <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz):**

SAMPLE		SPECIAL	
solvent	CDC13	temp	not used
file		gtn	not used
ACQUISITION	exp	spn	not used
sw	25125.6	ht	0.000
at	1.188	pw99	18.600
np	60270	atfa	20.000
fb	13800	l1	n
bs	16	in	n
d1	1.000	dp	y
nt	2000	hs	nn
ct			
TRANSMITTER		l1b	2.00
tn	C13	fn	65538
sfrq	100.554	sp	1284.8
tof	1536.3	wp	18214.6
tpwr	61	rfl	9266.4
pw	9.300	rfp	7764.8
DECOUPLER		rp	-49.0
dn	H1	lp	-355.3
dof	0	sc	0
dm	yyy	wc	250
dmm	w	vs	0
dpr	42	th	20
dof	8900	rm	2
			no ph



**3,4-Dimethoxyphenylethylcyanamide (20a): IR (KBr):**



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