

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; ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 7.02-7.07 (m, 3H), 7.28-7.33 (m, 2H), 7.64 (brs, 1H); ^{13}C NMR (CDCl_3 , 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 $\text{C}_7\text{H}_6\text{N}_2$ (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; ^1H NMR (CDCl_3 , 400 MHz) δ (ppm) 3.87 (s, 3H, OCH_3), 6.88 (m, 1H), 6.95-7.05 (m, 2H), 7.18 (m, 1H); ^{13}C NMR (CDCl_3 , 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 $\text{C}_8\text{H}_8\text{N}_2\text{O}$ (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; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 6.56 (brs, 1H), 7.05 (m, 1H), 7.31 (m, 2H), 7.35 (m, 1H); ¹³C NMR (CDCl₃, 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 C₇H₅ClN₂ (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; ¹H NMR (CDCl₃ + 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); ¹³C NMR (CDCl₃ + 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 C₇H₅N₃O₂ (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; ¹H NMR (CDCl₃ + DMSO, 400 MHz,) δ (ppm) 2.56 (s, 3H, CH₃), 7.08 (d, *J* = 8.8 Hz, 2H), 7.91 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (CDCl₃ + 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 C₉H₈N₂O (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; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 6.91 (d, *J* = 8.0 Hz, 2H,), 7.28 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (CDCl₃, 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 C₇H₅ClN₂ (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; ¹H NMR (CDCl₃ + 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); ¹³C NMR (CDCl₃ + 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 C₇H₆N₂O (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; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 2.29 (s, 3H), 6.17 (brs, 1H), 7.17 (m, 2H), 7.56 (s, 1H); ¹³C NMR (CDCl₃, 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 C₈H₇IN₂ (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; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 2.18 (s, 3H, CH₃), 2.26 (s, 3H, CH₃), 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); ¹³C NMR (CDCl₃, 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 C₉H₁₀N₂ (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; ¹H NMR (CDCl₃, 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); ¹³C NMR (CDCl₃, 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 C₇H₁₂N₂ (124.19): calcd. C 67.70, H 9.74, N 22.56; found C 67.67, H 9.70, N 22.50.

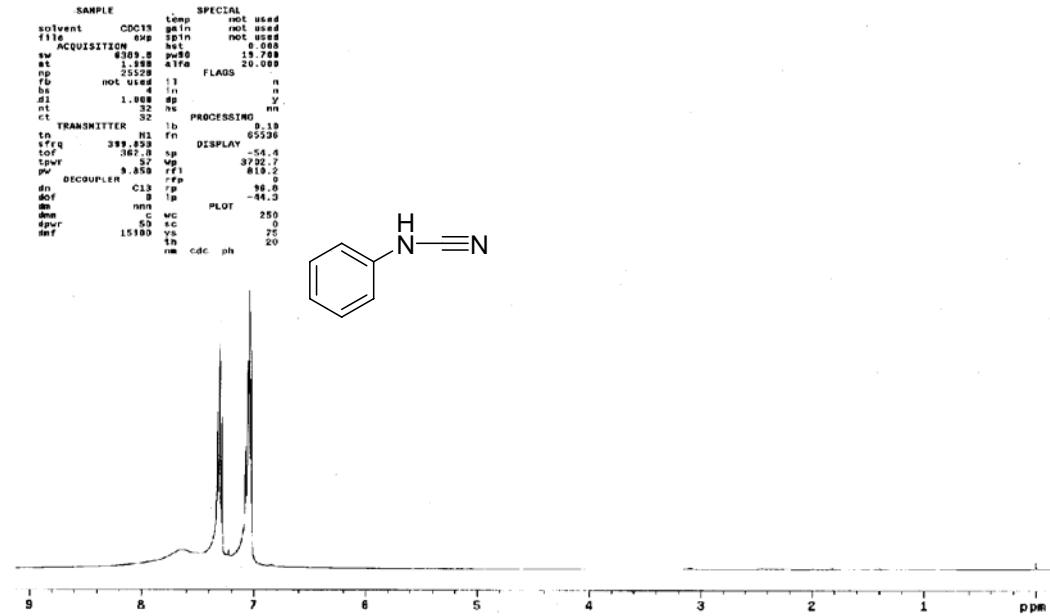
n-Butyl-cyanamide (17a). Gummy; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 0.94 (t, *J* = 7.6 Hz, 3H, CH₃), 1.40 (m, 2H, CH₂), 1.58 (m, 2H, CH₂), 3.06 (m, 2H, CH₂), 4.61 (brs, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ (ppm) 13.6, 19.5, 31.7, 45.7, 117.2; IR (KBr) 3207 (s), 2961 (s), 2875 (m), 2221 (s), 1614 (m), 1463 (m), 1373 (w), 1171 (w), 1015 (w); elemental analysis for C₅H₁₀N₂ (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; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 4.11 (d, *J* = 5.2 Hz, 2H, CH₂), 4.66 (brs, 1H), 7.27-7.37 (m, 5H); ¹³C NMR (CDCl₃, 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),

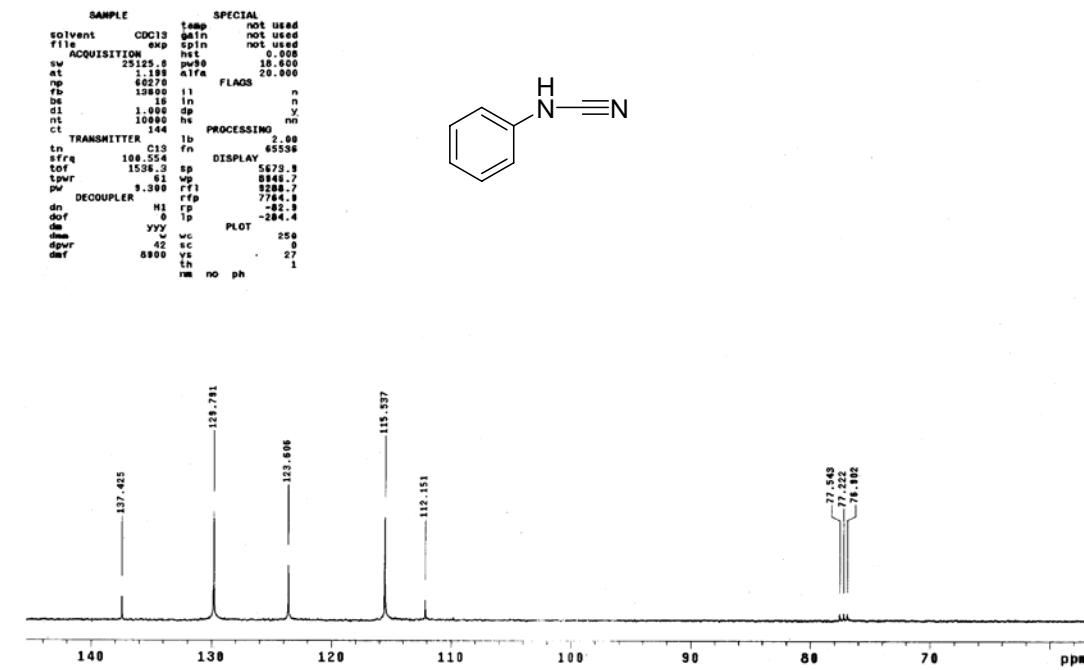
1359 (w), 1210 (w), 1155 (w), 1014 (w), 895 (w); elemental analysis for C₈H₈N₂ (132.17): calcd. C 72.70, H 6.10, N 21.19; found C 72.66, H 6.13, N 21.11.

Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a). White Solid; Mp: 82-84 °C; ¹H NMR (CDCl₃, 400 MHz) δ (ppm) 4.05 (d, *J* = 5.2 Hz, 2H, CH₂), 4.57 (brs, 1H), 5.94 (s, 2H, OCH₂), 6.77 (m, 3H); ¹³C NMR (CDCl₃, 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 C₉H₈N₂O₂ (176.18): calcd. C 61.36, H 4.58, N 15.90; found C 61.41, H 4.61, N 15.85.

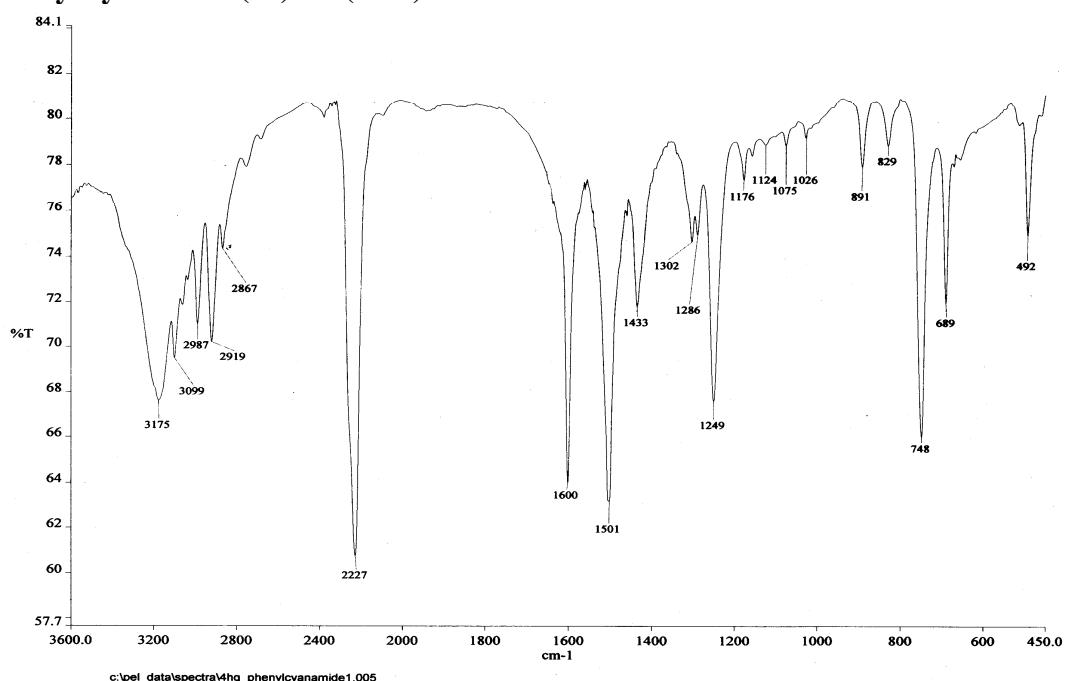
Phenyl cyanamide (1a): ^1H NMR (CDCl_3 , 400 MHz):



Phenyl cyanamide (1a): ^{13}C NMR (CDCl_3 , 100 MHz):

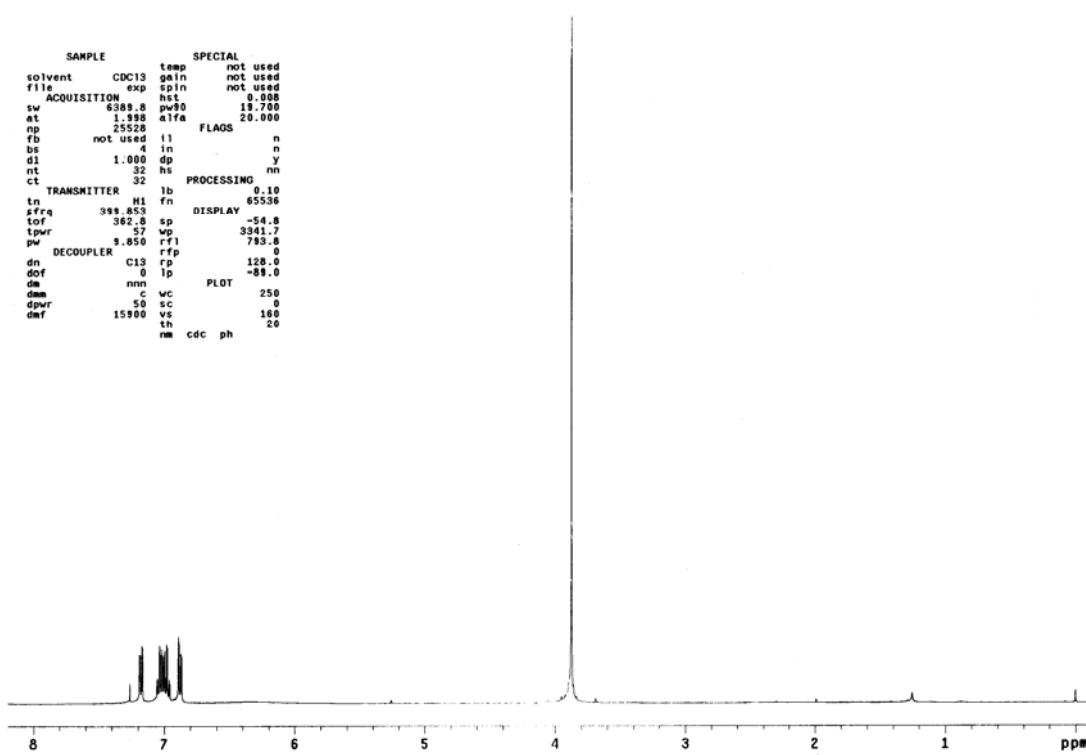


Phenyl cyanamide (1a): IR(KBr):



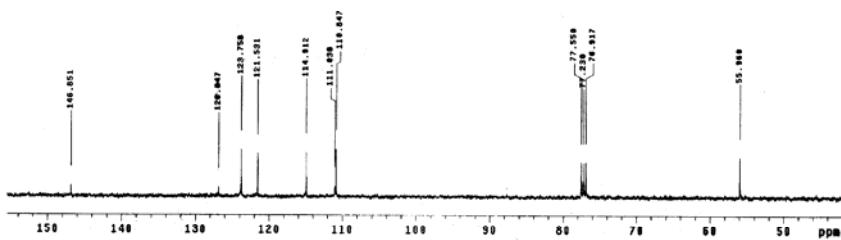
2-Methoxy-phenyl cyanamide (2a): ^1H NMR (CDCl_3 , 400 MHz):

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at             1.338  a1fa  20.000
np             25528  FLAGS
fb             not used  i1      n
bs             not used  in      n
dl             1.000   ap      y
nt             32     hs      nn
ct             32     lb      0.10
TRANSMITTER    HI   fn      65536
tn             65536
sfrq          399.853  DISPLAY
tof            362.8  sp      -54.8
tppw          362.8  rfp     362.7
pw             1.850   rf1     783.8
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dn             C13  rp      128.0
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nm   cdc ph
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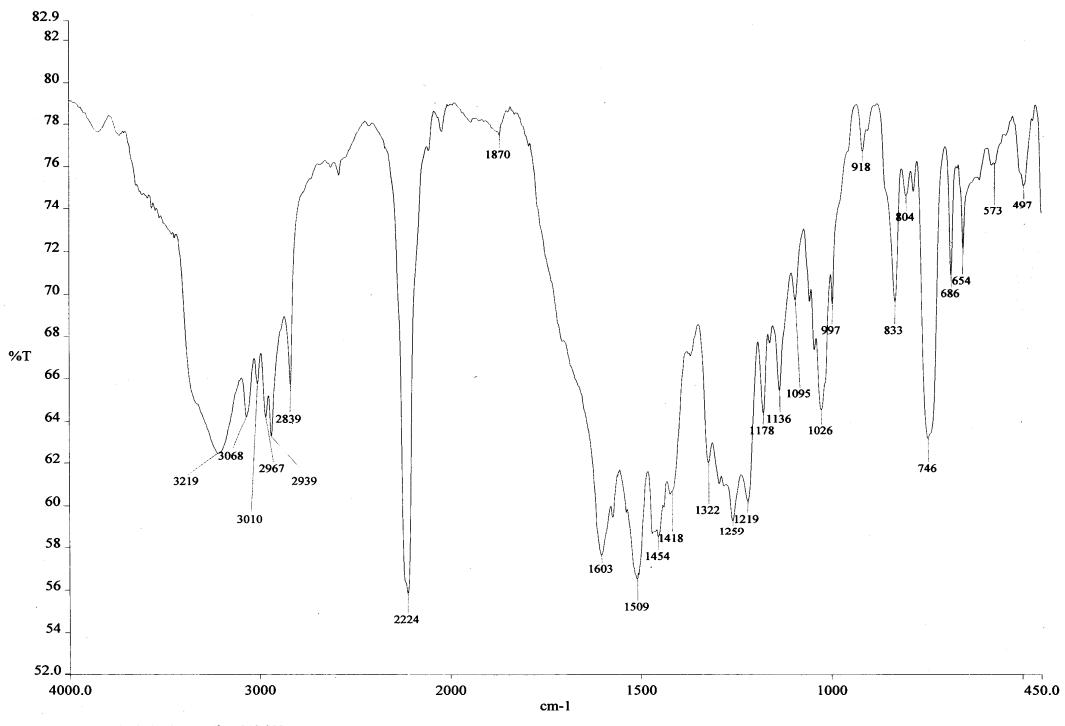


Supplementary Material (ESI) for Green Chemistry
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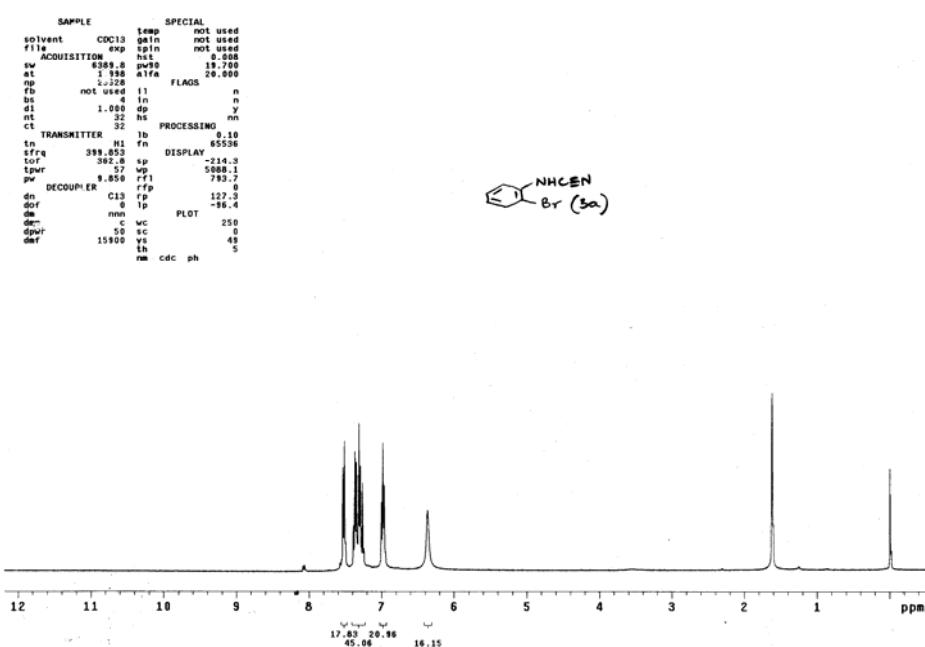
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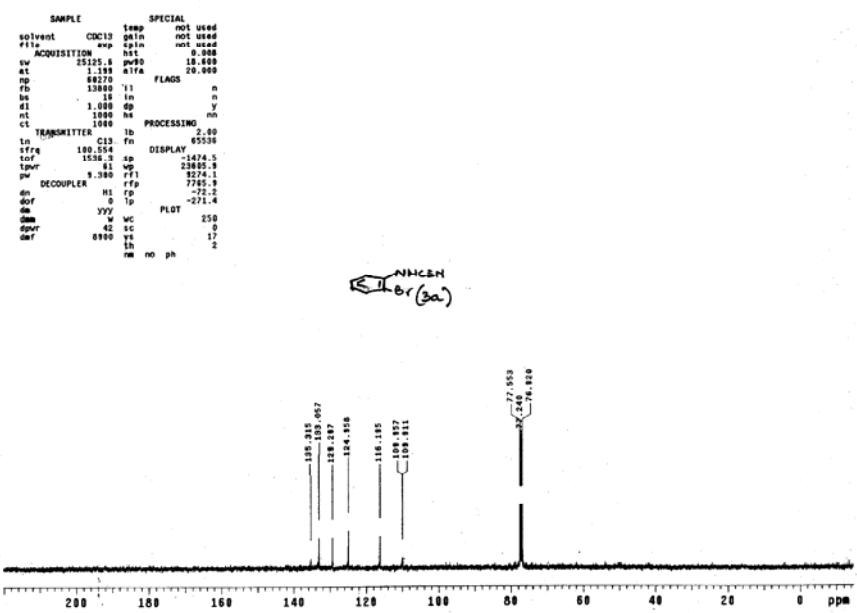
2-Methoxy-phenyl cyanamide (2a): IR(KBr):



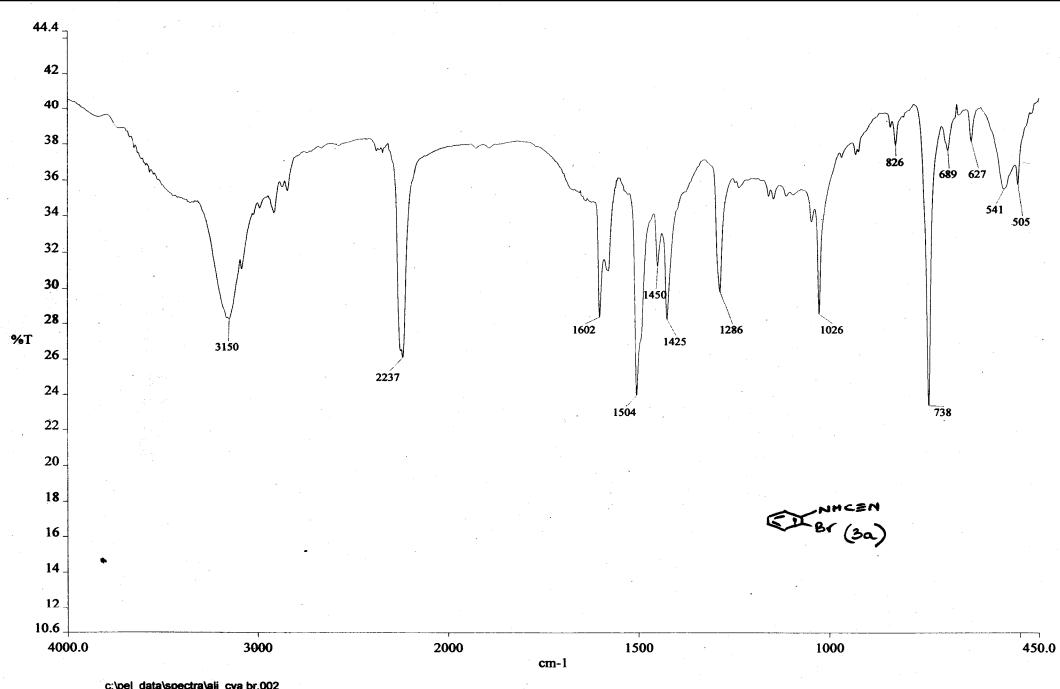
2-Bromo phenyl cyanamide (3a): ^1H NMR (CDCl_3 , 400 MHz):



2-Bromo phenyl cyanamide (3a): ^{13}C NMR (CDCl_3 , 100 MHz):

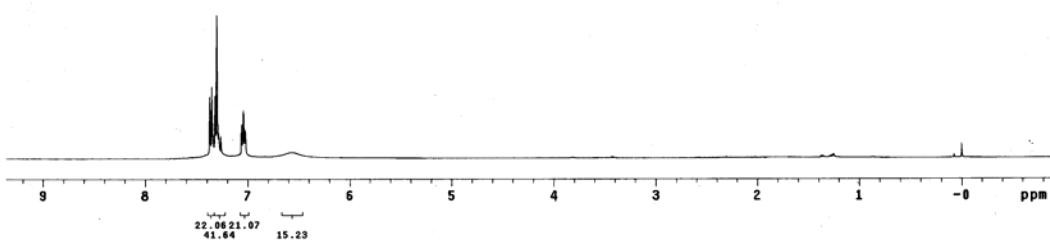
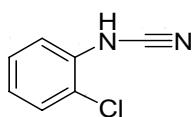


2-Bromo phenyl cyanamide (3a): IR (KBr):



2-Chloro-phenyl cyanamide (4a): ^1H NMR (CDCl_3 , 400 MHz):

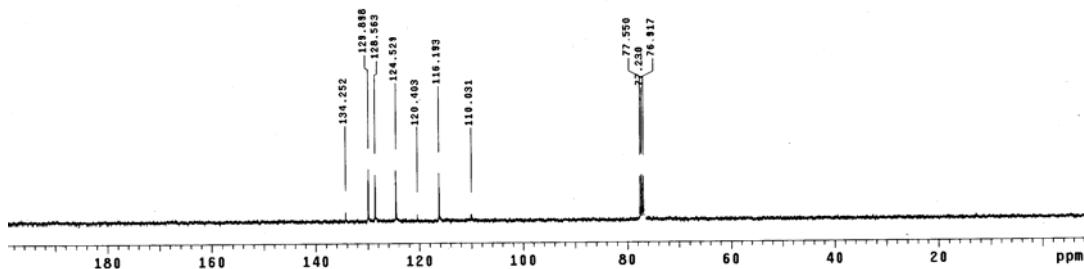
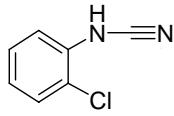
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file           exp      gain    not used
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at             6511.0      a1fa   20.000
rt              1.998      a1fa   20.000
np            25528      flags
rb            not used    11      n
bs             4          11      n
d1            1.000      dp      y
nt             32         hs      nn
ct             32         PROCESSING
ct           TRANSMITTER  1b      0.10
tn            H1      fm      65536
trfq        398.05      DISPLAY
trf          362.8      sp      -353.5
tpur         57         vp      4096.8
pw           8.850      r1      793.7
DECOUPLER    C13      rfp
dn            C13      rp      115.8
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nm           cdc      ph      20
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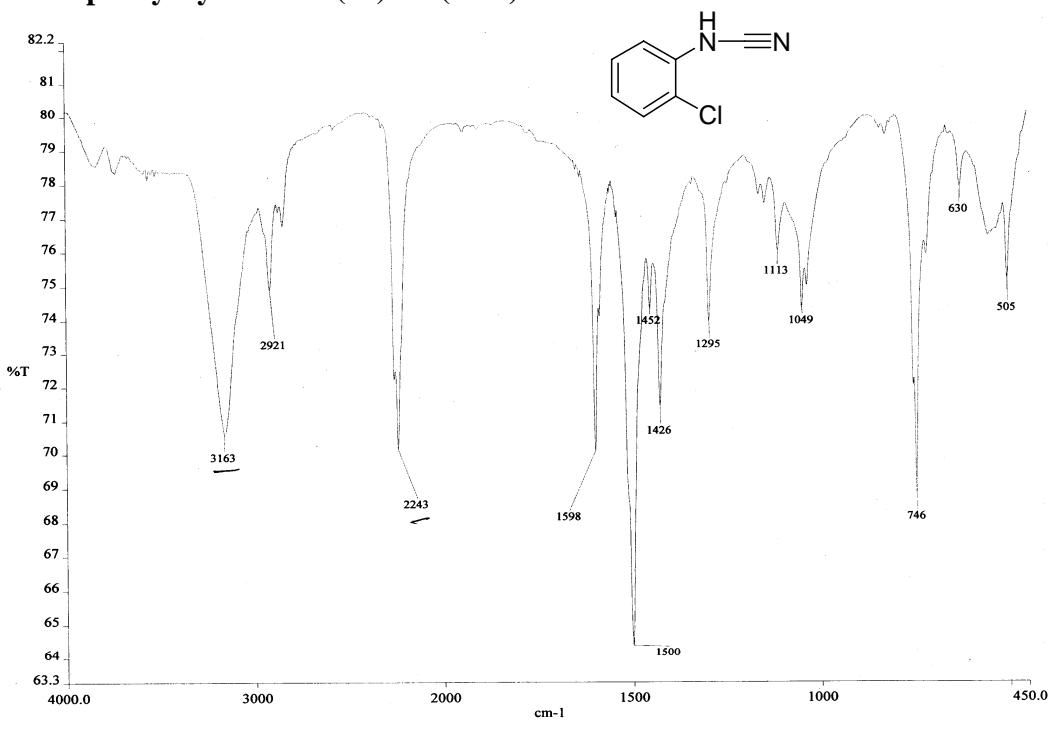
2-Chloro-phenyl cyanamide (4a): ^{13}C NMR (CDCl_3 , 100 MHz):

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ACQUISITION spin not used
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et       1.199 aifra 20.000
np       60270 flags
fb       13800 n
ns       100 n
ds       1.000 dp y
nt       2800 hs nn
ct       884
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tn      100.554 fm 65536
sfrq   100.554 sp -879.7
tdf    1538.3 61 vp 20964.3
tpwr   9.300 rrf1 9272.6
DECOUPLER H1 rfp 7764.9
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nm no ph
    
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2-Chloro-phenyl cyanamide (4a): IR(KBr):



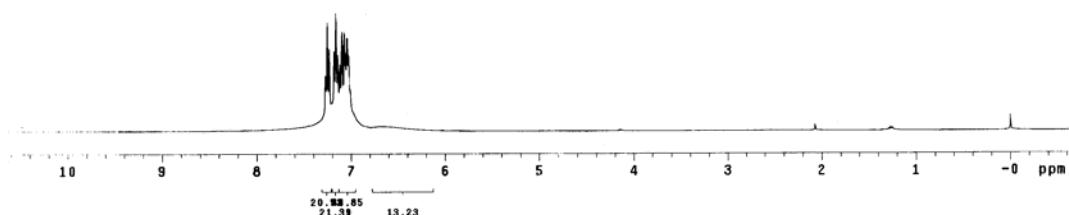
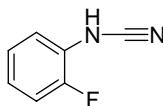
c:\pel_data\spectra\ramesh\ry_1_284.sp - O-Cl cyanamide

2-Fluoro-phenyl cyanamide (5a): ^1H NMR (CDCl_3 , 400 MHz):

```

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at             1.000    alfa   20.000
np            25528    FLAGS
fb            not used  11      n
bs             32      1n      n
dt             1.000    32      y
nt             32      ns      nn
ct              32      PROCESSING
TRANSMITTER   1b      H1      tn      0.10
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$sp            362.8   sp      254.7
$ppr          57      wp      4550.7
$pw            8.850   rf1    792.1
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$dmf           15000   vs      28
$rt             th      th      20
$nm            cdc    ph

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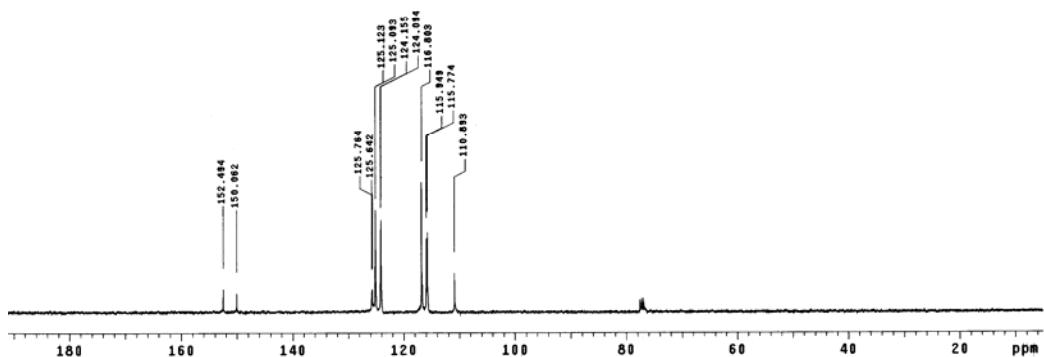
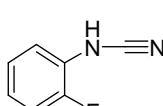


2-Fluoro-phenyl cyanamide (5a): ^{13}C NMR (CDCl_3 , 100 MHz):

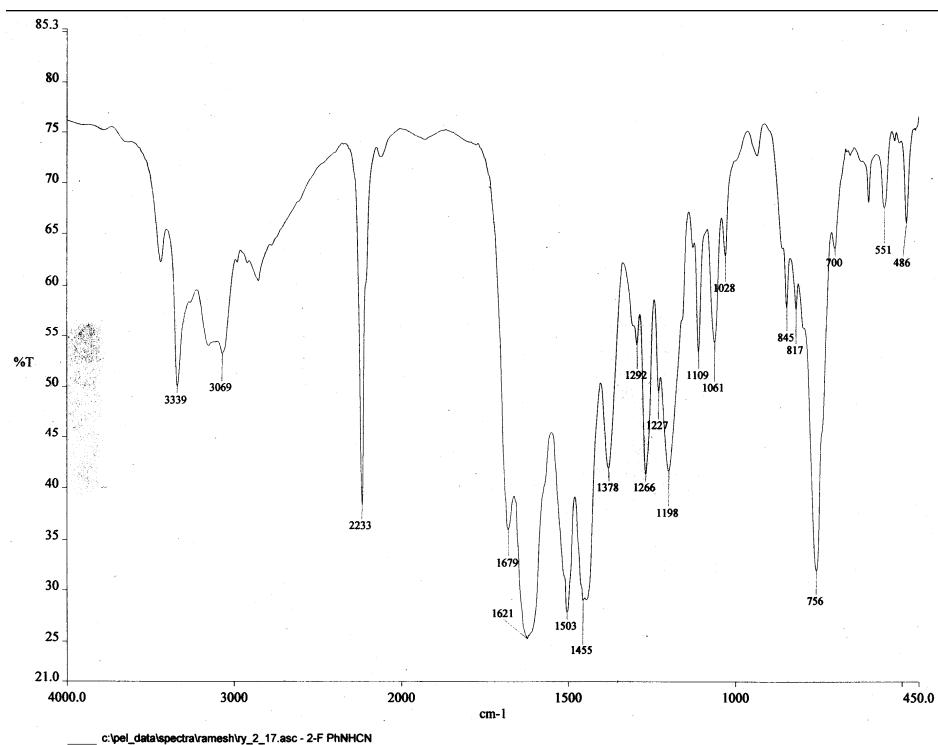
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file           exp     gain     not used
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np             63870   1n      250
$ppr          15000   0p      Y
$pw            16      hs      nn
$dt             1.000   1b      2.00
$ct             224    fn      65536
$TRANSMITTER  C13    sp      500.9
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$tof           1536.3  rf1    9286.4
$ppr          61      rfp    7764.9
$pw            9.300   1p      10.0
$DECOUPLER    C13    1p    -271.4
$dn            C13    rfp    137.0
$de             VVY    wc      250
$dm             VV    vs      31
$dpw           42      th      3
$rt             8800   $nm no ph

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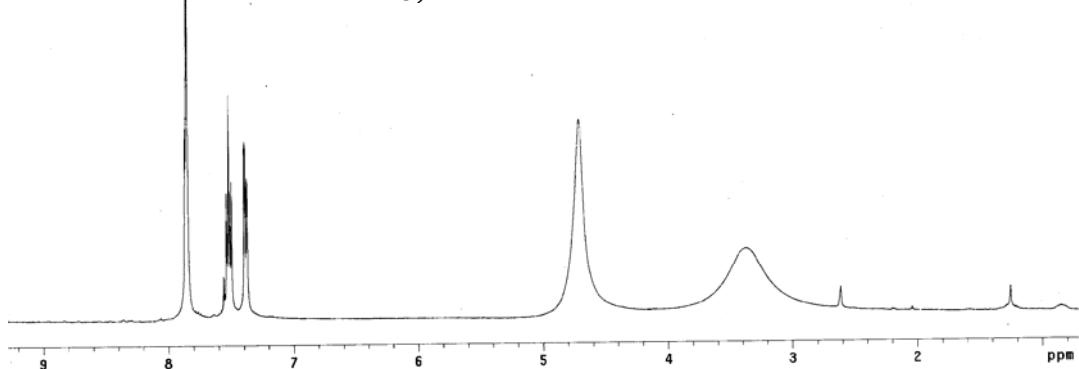
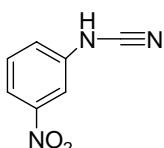


2-Fluoro-phenyl cyanamide (5a): IR (KBr):

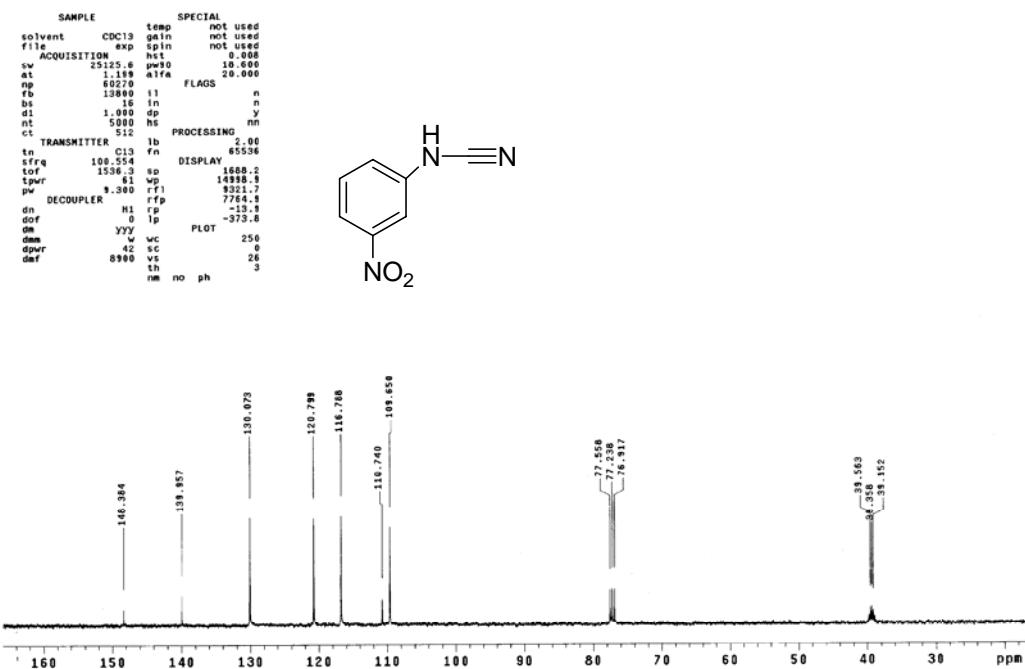


3- Nitrophenylcyanamide (6a): ^1H NMR ($\text{CDCl}_3 + \text{DMSO}$, 400MHz):

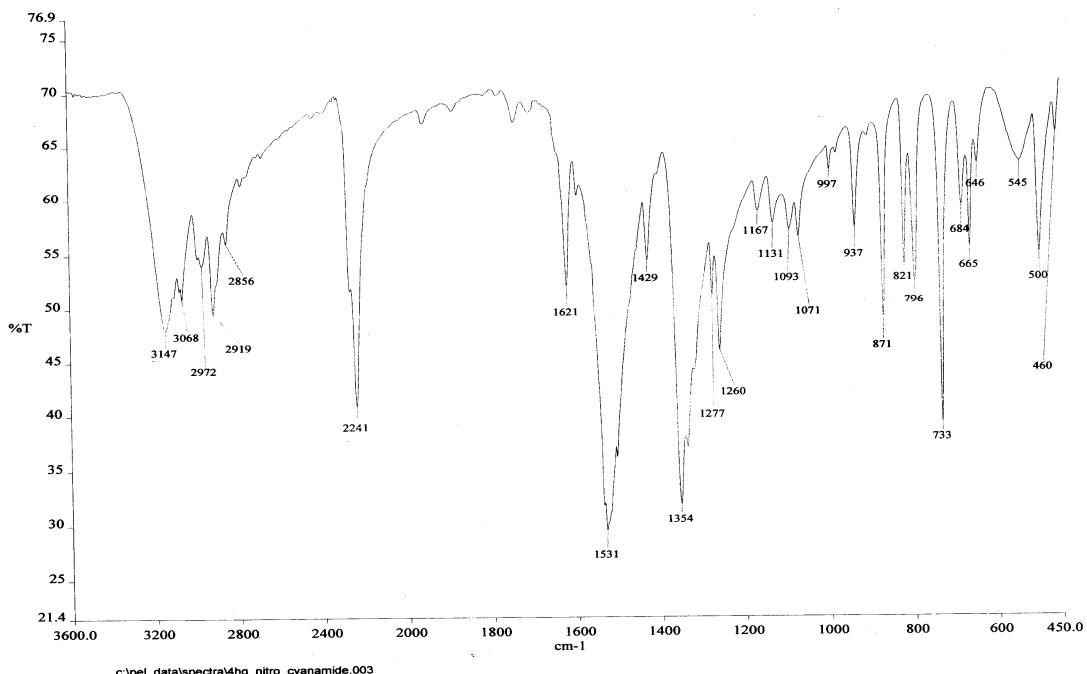
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file		spin	not used
ACQUISITION	hst	hst	0.006
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at	25528	alpha	20.000
np	25528	FLAOS	
fb	not used	il	n
bs	4	n	n
dt	1.000	dp	y
nt	32	hs	nn
ct	32	PROCESSING	
tn	TRANSMITTER	lb	0.10
sfrq	H1	rn	65536
tot	399.85	DISPLAY	
tpwr	362.8	sp	282.8
pw	5.57	wp	3467.5
pw	9.850	rfl	674.5
DECOUPLER		rfp	0
dn	C13	p	128.8
dof	0	1p	-81.6
dm	nnn	PLOT	
dm	wc		250
dppr	50	sc	0
dppr	15900	vs	99
dppr	nm	th	20
	cdc	ph	



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



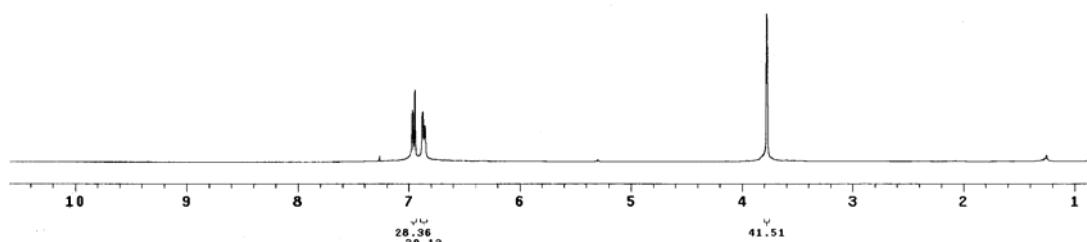
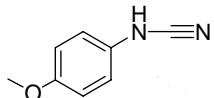
3-Nitro-phenyl cyanamide (6a): IR (KBr):



4-Methoxy-phenyl cyanamide (7a): ^1H NMR (CDCl_3 , 400 MHz):

```

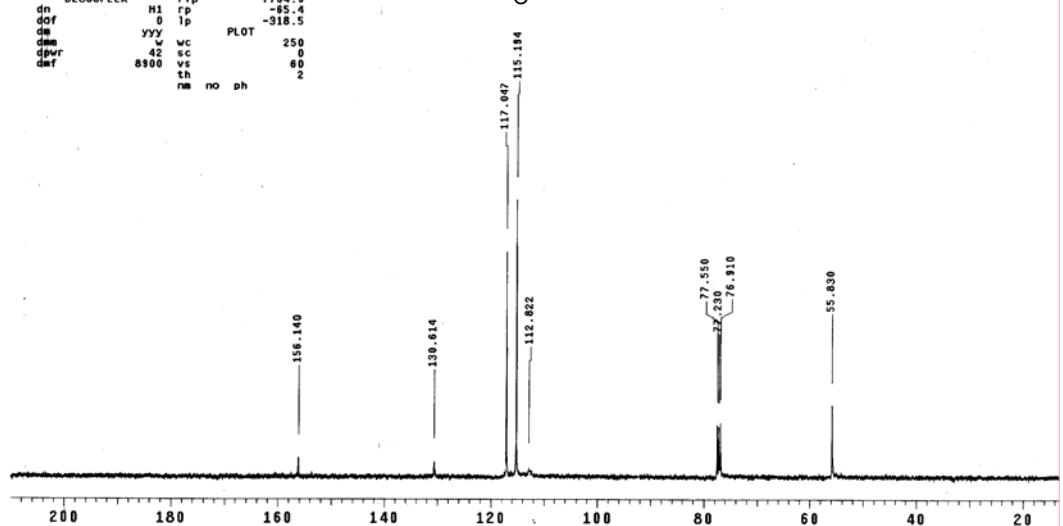
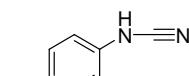
SAMPLE          SPECIAL
solvent      CDCl3   temp   not used
file        exp     gain   not used
ACQUISITION   hst    spin   0.008
sw           6385.1  pw80   18.700
at            1.199  alfa   20.000
np           25528   t1    FLAGs
fb           not used
di           1.000   dt    y
nt            32     ht    nn
ct            32     lb    0.10
tn      TRANSMITTER   H1    DISPLAY
sfrq      399.853  sp    -172.4
tof       362.0    wp    4498.6
tpwr      57       rf1   793.8
pw         9.850   rfp   0
DECOUPLER   C13   fp    127.4
dn           0       ip    -37.0
dof          0       PLOT  250
dm           c       wc    250
dpw        50       sc    35
dpwcr     15900   th    20
dmf        nm      cdc   ph
    
```



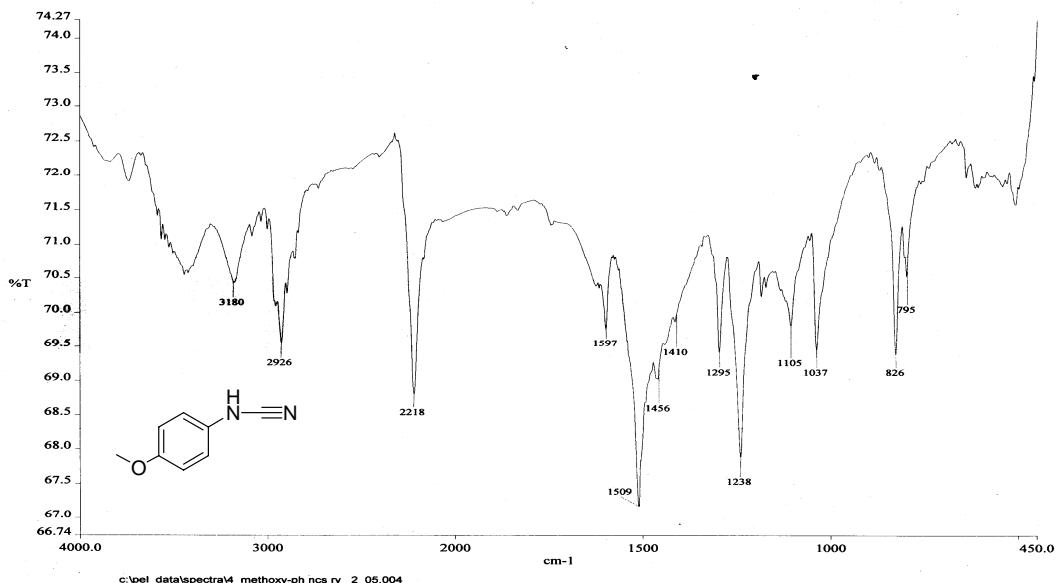
4-Methoxy-phenyl cyanamide (7a): ^{13}C NMR (CDCl_3 , 100 MHz):

```

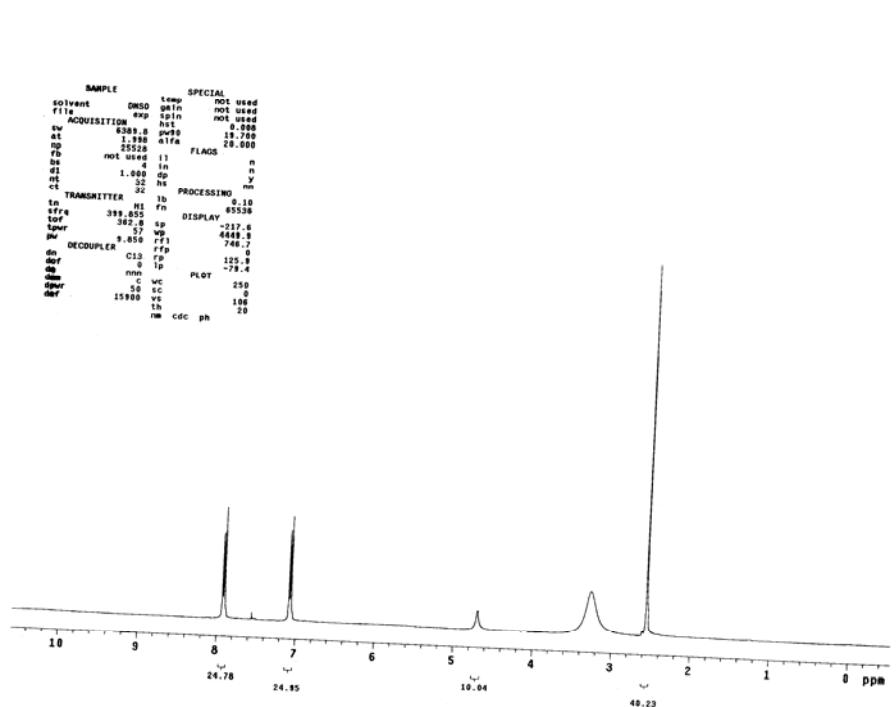
SAMPLE          SPECIAL
solvent      CDCl3   temp   not used
file        exp     gain   not used
ACQUISITION   hst    spin   0.008
sw           2525.6  pw80   18.600
at            1.199  alfa   20.000
np           60270   t1    FLAGs
fb           13800   t1    n
dt           1.000   dt    n
di           2.000   ht    y
nt            1056   ht    nn
ct            1056   lb    2.00
tn      TRANSMITTER   C13   DISPLAY
sfrq      100.554  sp    -650.4
tof       1536.3   wp    21757.2
tpwr      61       rf1   7764.8
pw         9.300   rfp   1
DECOUPLER   C13   rfp   7764.8
dn           1       rp    -65.4
dof          0       ip    -318.5
dm           v       wc    250
dpwcr     8300    vs    60
dmf        nm      no    2
    
```



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

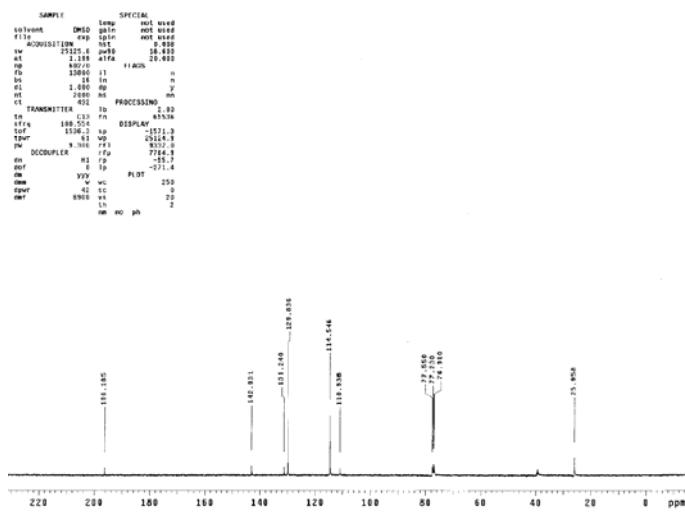


4-Acetyl-phenylcyanamide (8a): ^1H NMR ($\text{CDCl}_3 + \text{DMSO}$, 400 MHz):

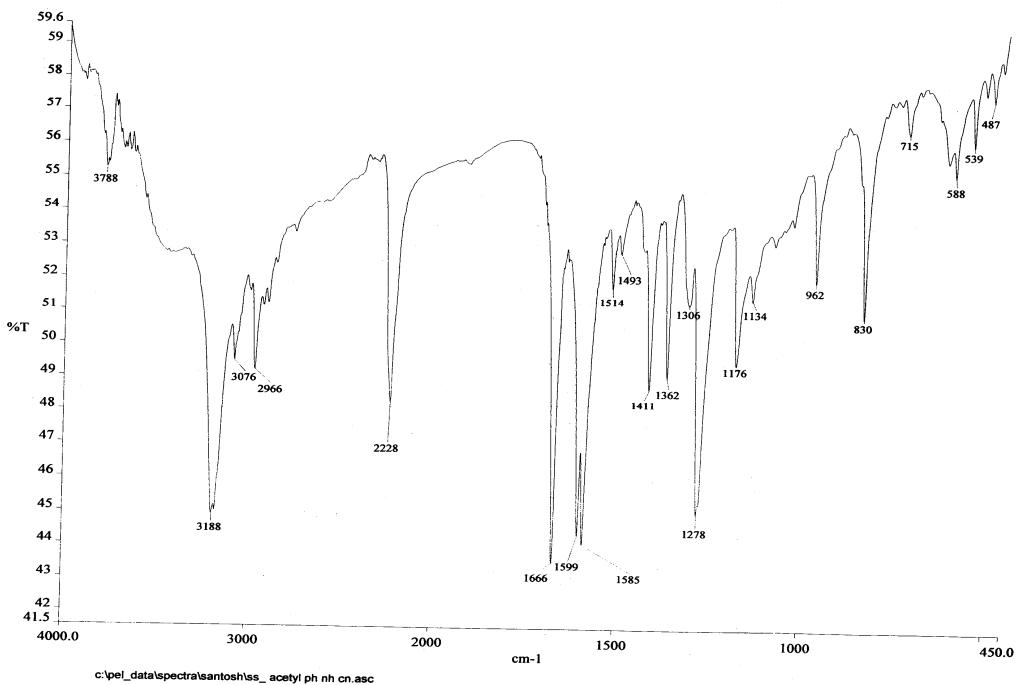


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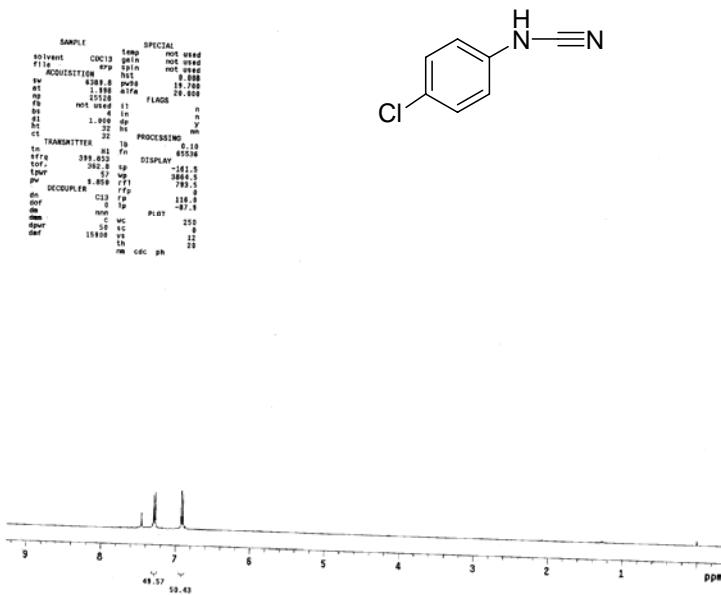
4-Acetyl-phenylcyanamide (8a): ^{13}C NMR ($\text{CDCl}_3 + \text{DMSO}$, 100 MHz):



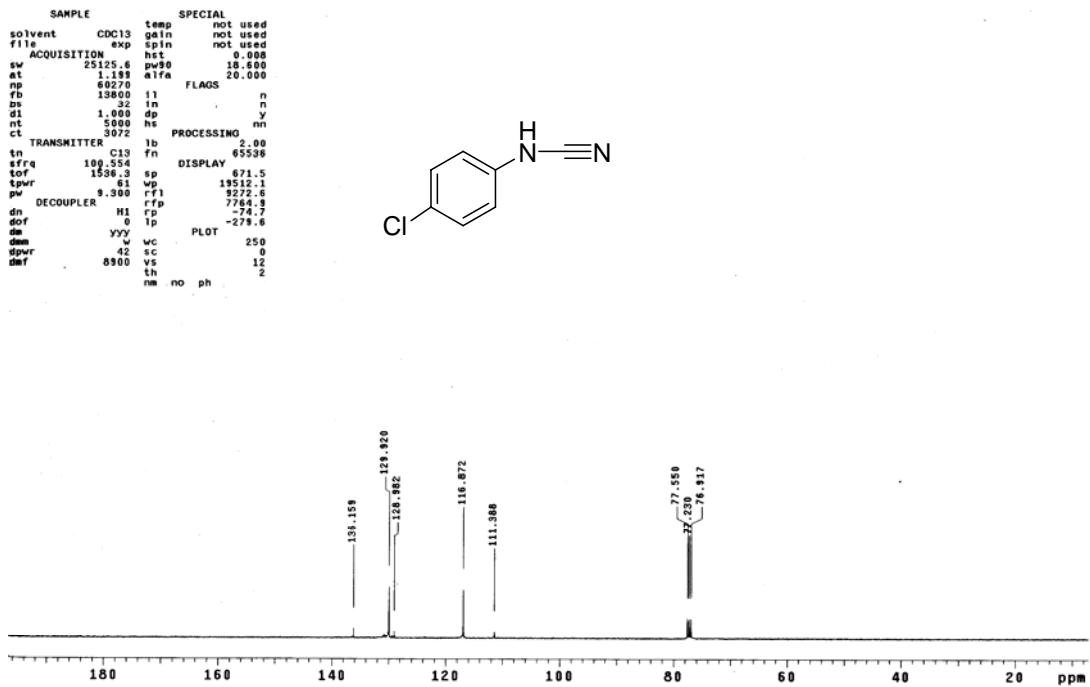
4-Acetyl-phenylcyanamide (8a): IR (KBr):



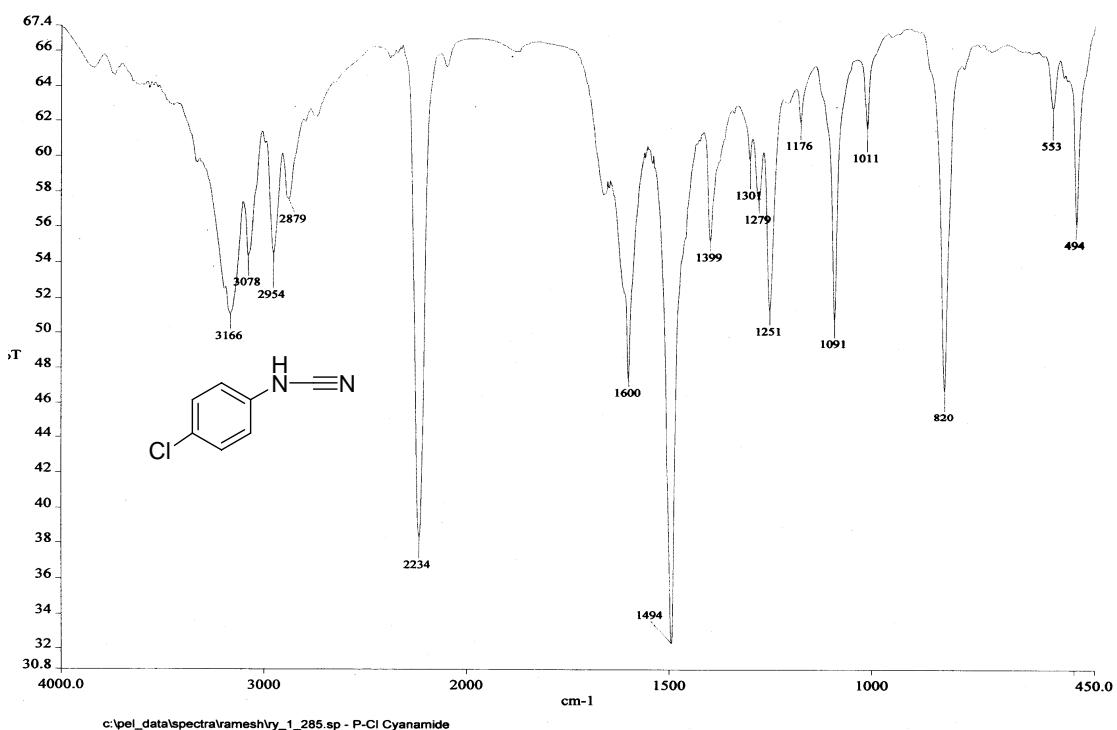
4-Chloro-phenyl cyanamide (9a): ^1H NMR (CDCl_3 , 400 MHz):



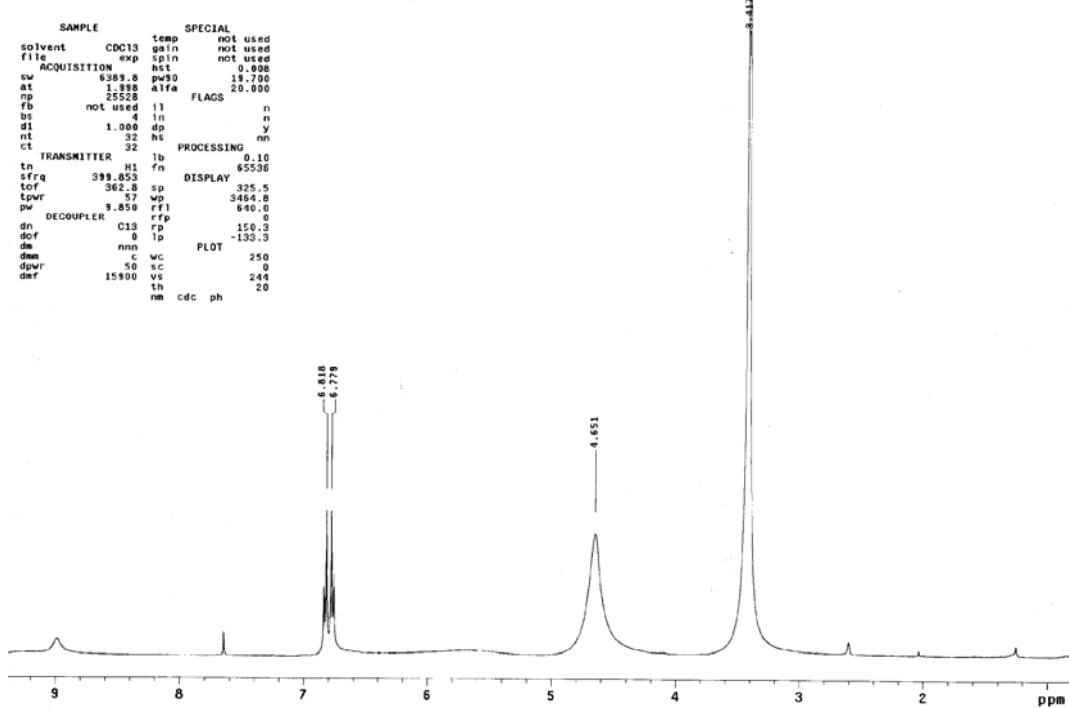
4-Chloro-phenyl cyanamide (9a): ^{13}C NMR (CDCl_3 , 100 MHz):



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



4-Hydroxy-phenyl cyanamide (10a): ¹H NMR (CDCl₃ + DMSO, 400 MHz):

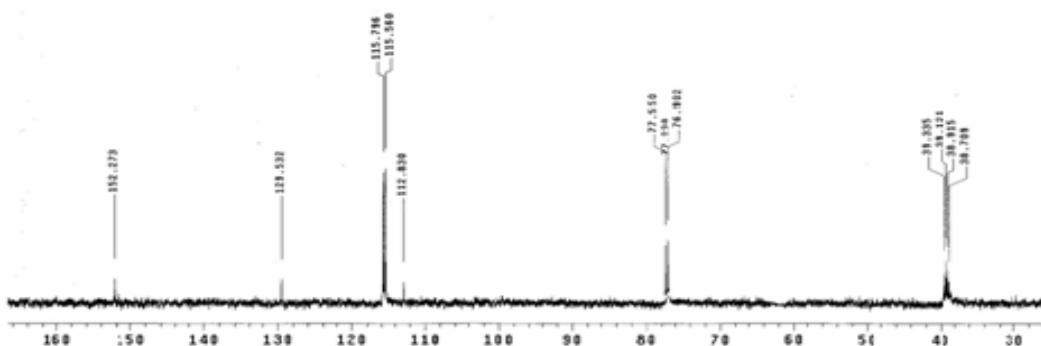
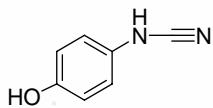


4-Hydroxy-phenyl cyanamide (10a): ^{13}C NMR ($\text{CDCl}_3 + \text{DMSO}$, 100 MHz):

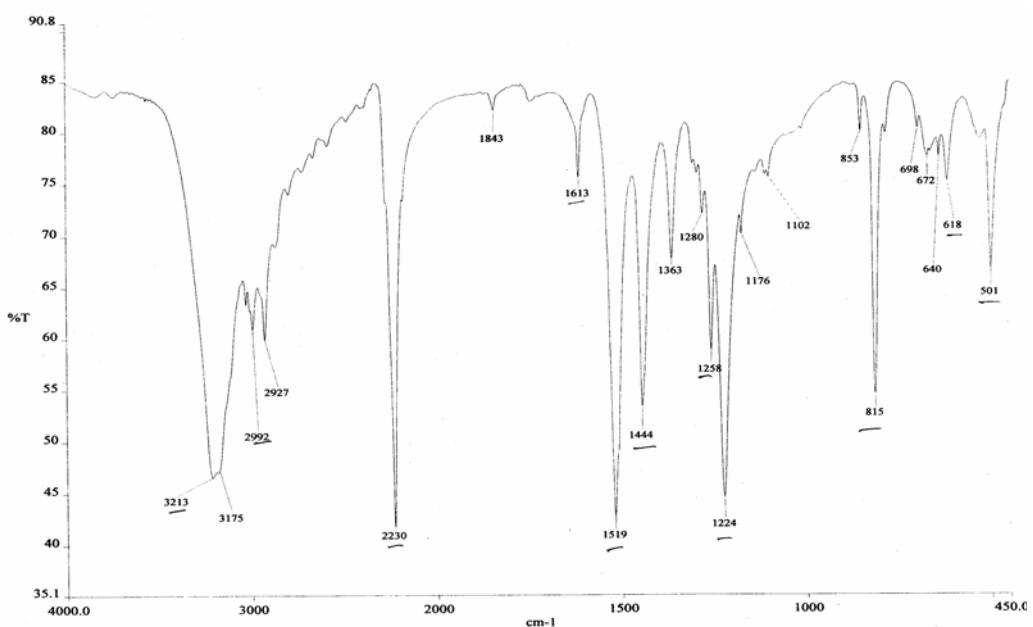
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SAMPLE          SPECIAL
solvent        CDCl3   texp   not used
F1             gain   not used
F11            exp   not used
F12            int    0.000
SW             25125.6  pw0.8  18.600
ALC            65270   a1fa   25.000
TD             13860   11      n
TM             1.000   1n    n
DT             1.000   1n    n
RT              1.000   1n    n
CT              2.000   1n    n
TRANSMITTER    1B     2.00
Tm             C13   fm    65536
SFID           1H, 554   ap    372.6
TDZ            1.000   r1f1  35781.3
PDR            9.300   r1f2  9334.7
7784.9
DECOUPLER      XI     1p    -20.0
M1             0      1p    -273.4
DW             42     nc    258
DW1            8500   ws    32
DW1T           8500   ws    32
D1T            1.00   1n    3
RD              no    ph

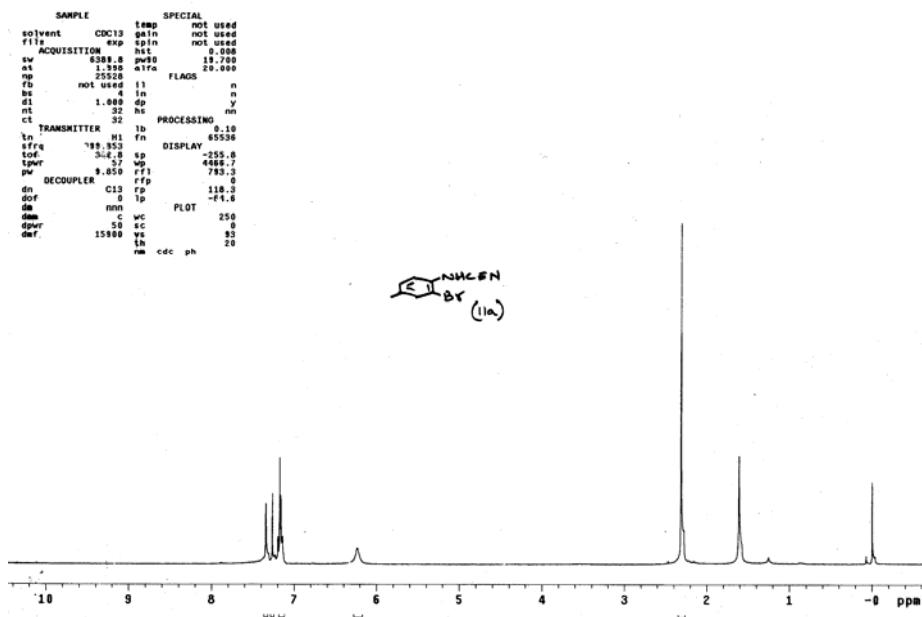
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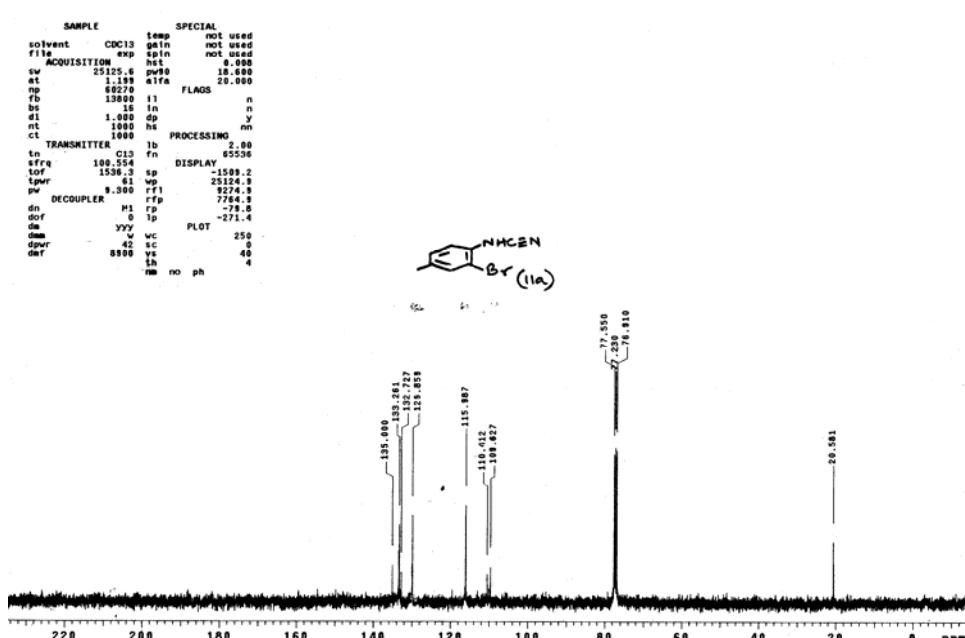
4-Hydroxy-phenyl cyanamide (10a): IR (KBr):



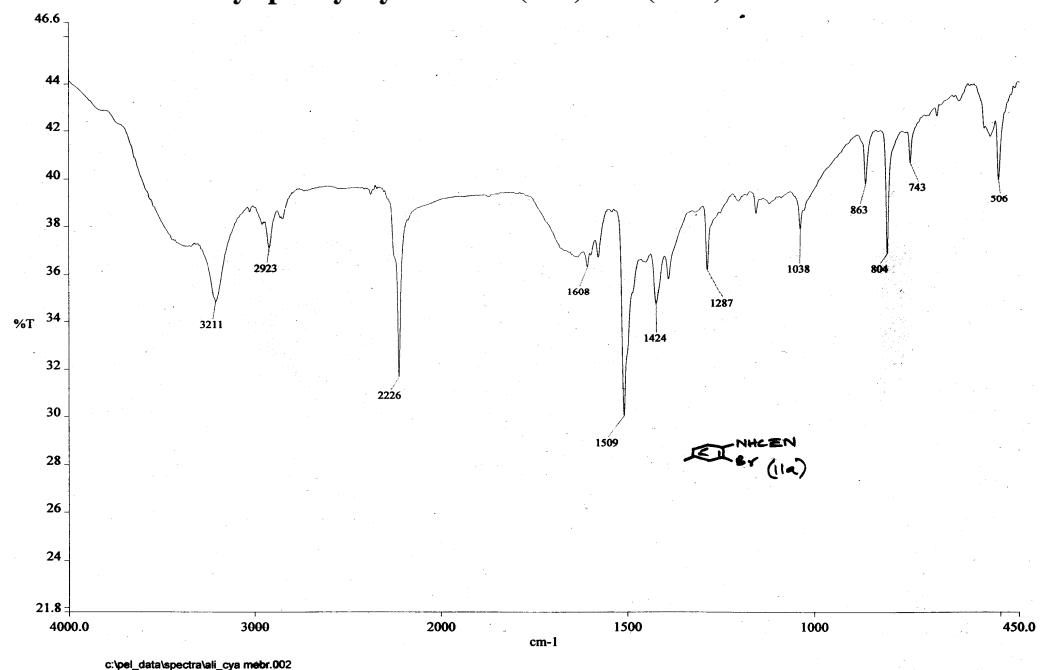
2-Bromo-4-Methyl-phenyl cyanamide (11a): ^1H NMR (CDCl_3 , 400 MHz):



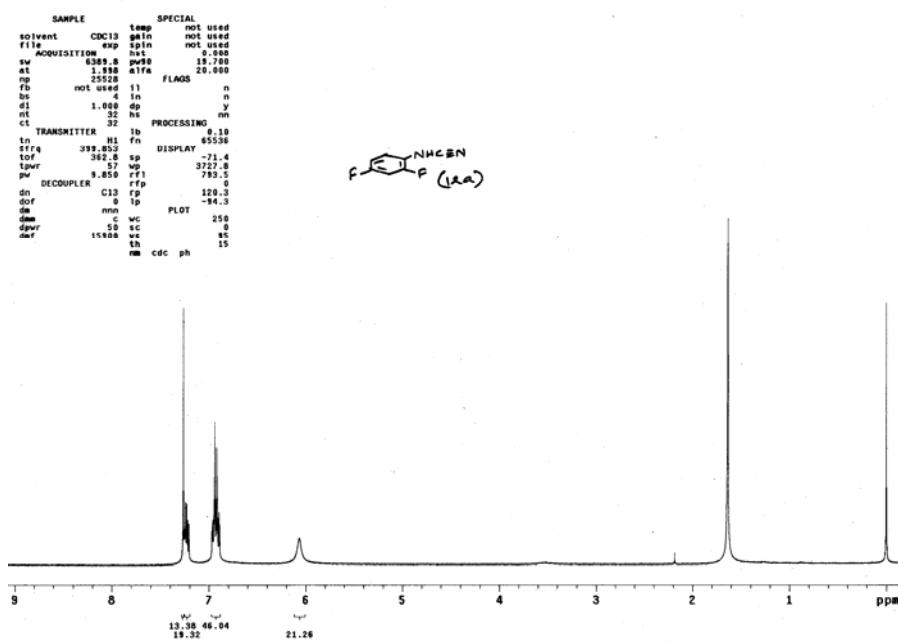
2-Bromo-4-Methyl-phenyl cyanamide (11a): ^{13}C NMR (CDCl_3 , 100 MHz):



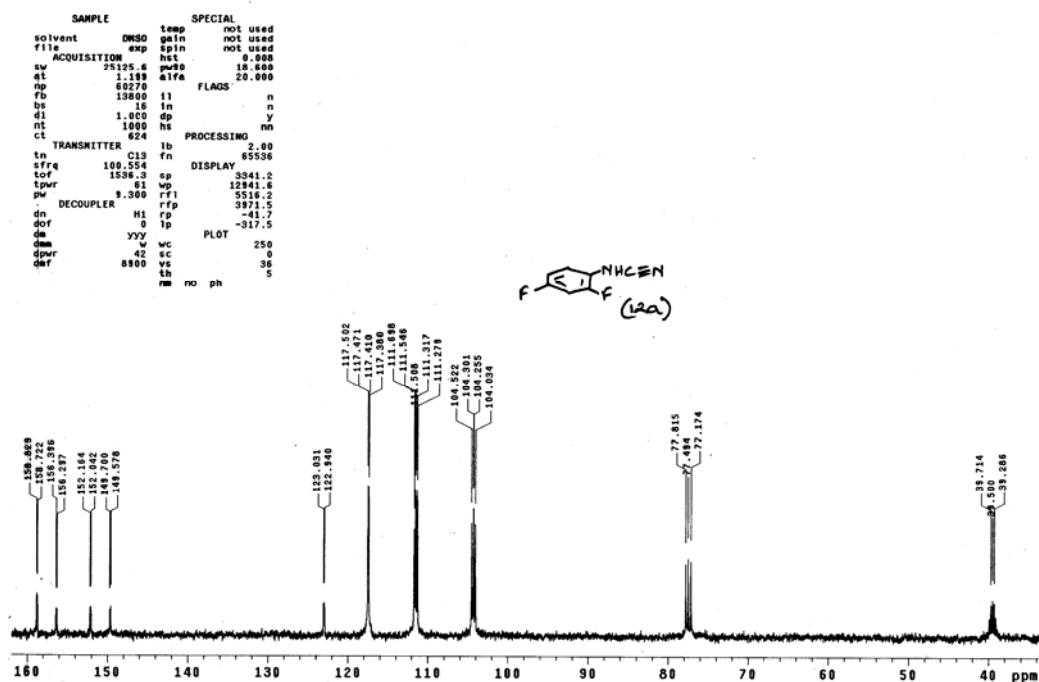
2-Bromo-4-Methyl-phenyl cyanamide (11a): IR (KBr):



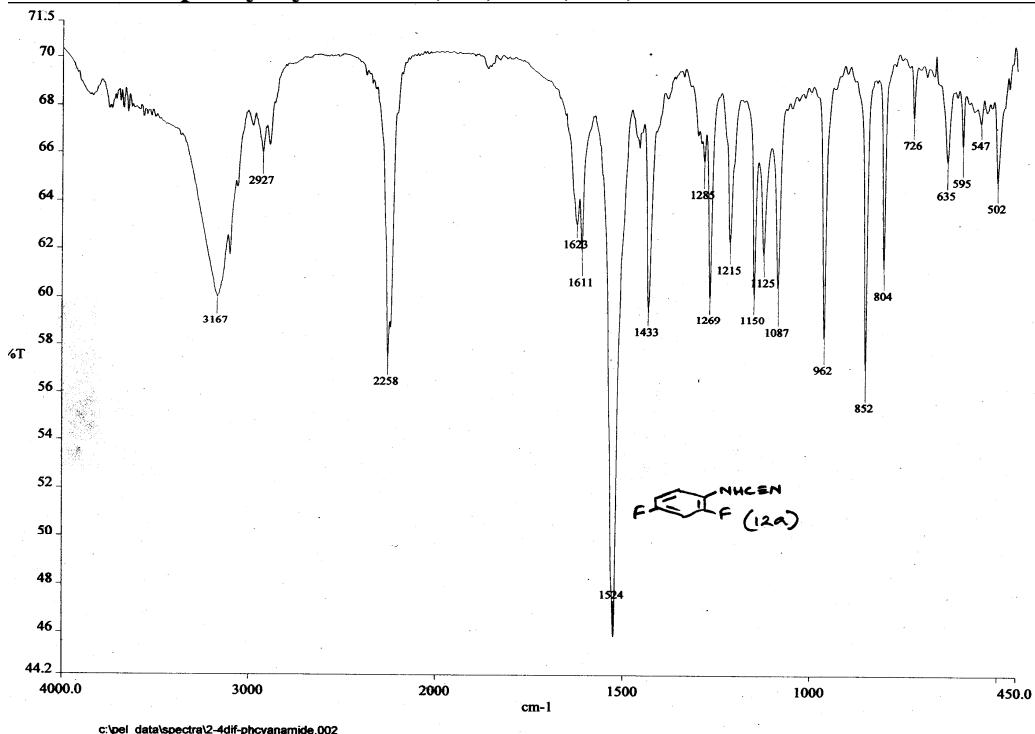
2-4-Difluoro phenyl cyanamide (12a): ¹H NMR (CDCl₃, 400 MHz):



2-4-Difluoro phenyl cyanamide (12a): ^{13}C NMR (CDCl_3 , 100 MHz):



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

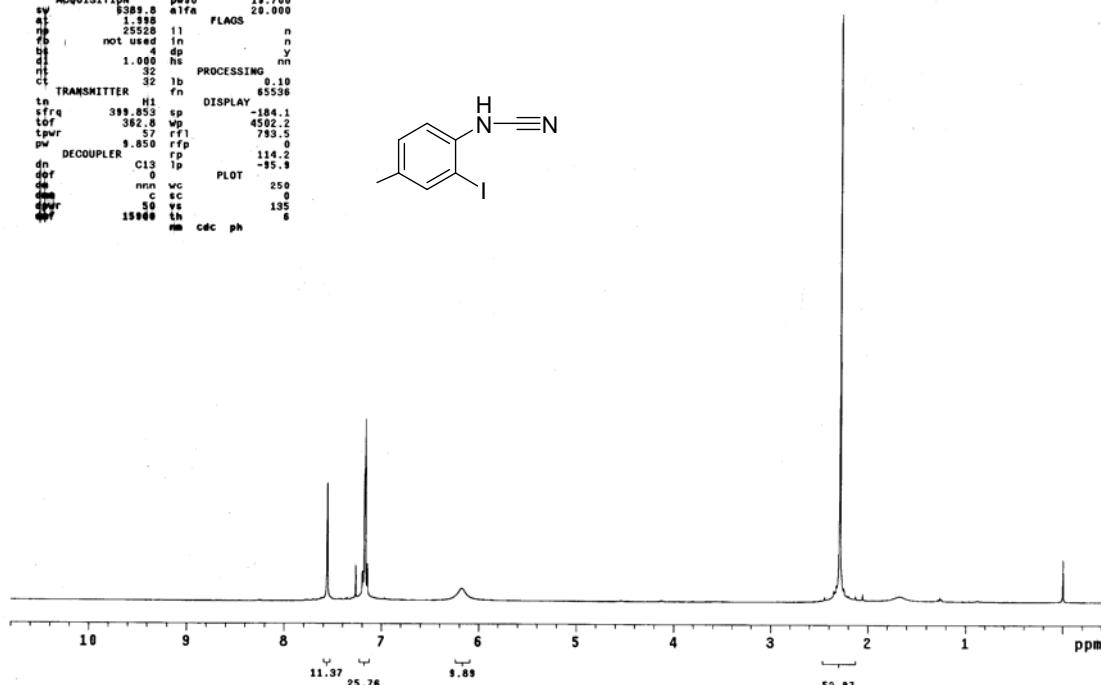
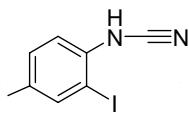


2-Iodo-4-methyl-phenyl cyanamide (13a): ^1H NMR (CDCl_3 , 400 MHz):

```

SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           report/home/-/      gain      not used
pfile          RY_2.fid   spin      not used
psfile         RY_2.fid   hst      0.006
ACQUISITION    p180      p180     19.700
sv            5388.5   alfa    20.000
et            25000    flags
at            25000
fb            25000    11      n
rb            not used   4      dp      y
dt            1.000   hs      nn
nt            5000    PROCESSING
ct            32    1b      0.10
CT TRANSMITTER H1      DISPLAY
sfreq        399.853   sp      -184.1
t0f          382.6    wp      4502.2
tpw          57       rf1     793.5
pw            9.850   rfp     0
DECOUPLER     C13    ip      -95.8
dn            C13    ip
dof           0      PLOT
dm            nnn   wc      250
dc            c     sc      0
dpr          50      vs      135
dppr         15000   th      6
nm            cdc   ph

```

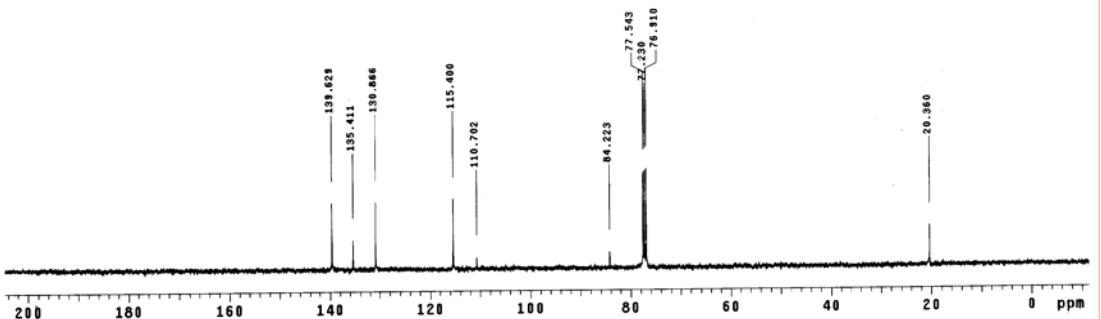
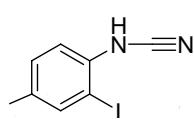


2-Iodo-4-methyl-phenyl cyanamide (13a): ^{13}C NMR (CDCl_3 , 100 MHz):

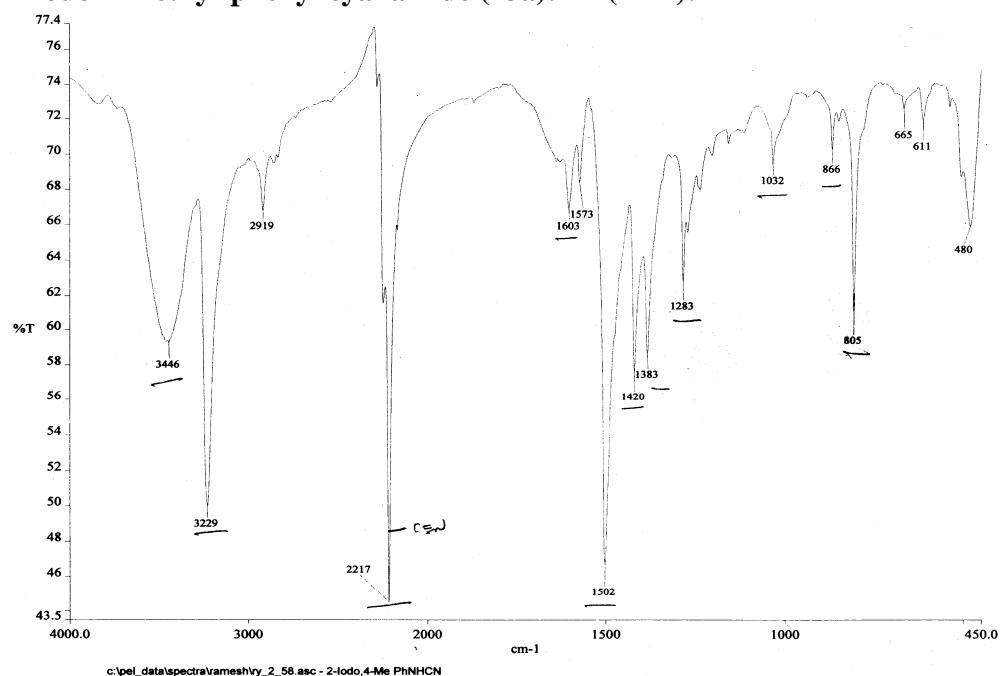
```

SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           report/home/-/      gain      not used
pfile          RY_2.fid   spin      not used
psfile         RY_2.fid   hst      0.006
ACQUISITION    p125.6   p180     19.700
sv            25125.6   alfa    20.000
et            60270    flags
fb            13800    11      n
dt            1.000   dp      n
nt            5000    PROCESSING
ct            2200    1b      2.00
CT TRANSMITTER C13    fn      65536
sfreq        100.554   sp      -1145.8
t0f          1536.3   wp      21724.2
tpw          3.300   rf1     9274.1
pw            9.300   rfp     7784.9
DECOUPLER     H1    rfp     -22.2
dn            VVY   ip      -397.9
dm            VVY   wc      250
dc            A2    sc      0
dpr          8900   vs      22
dppr         15000   th      2
nm            no   ph

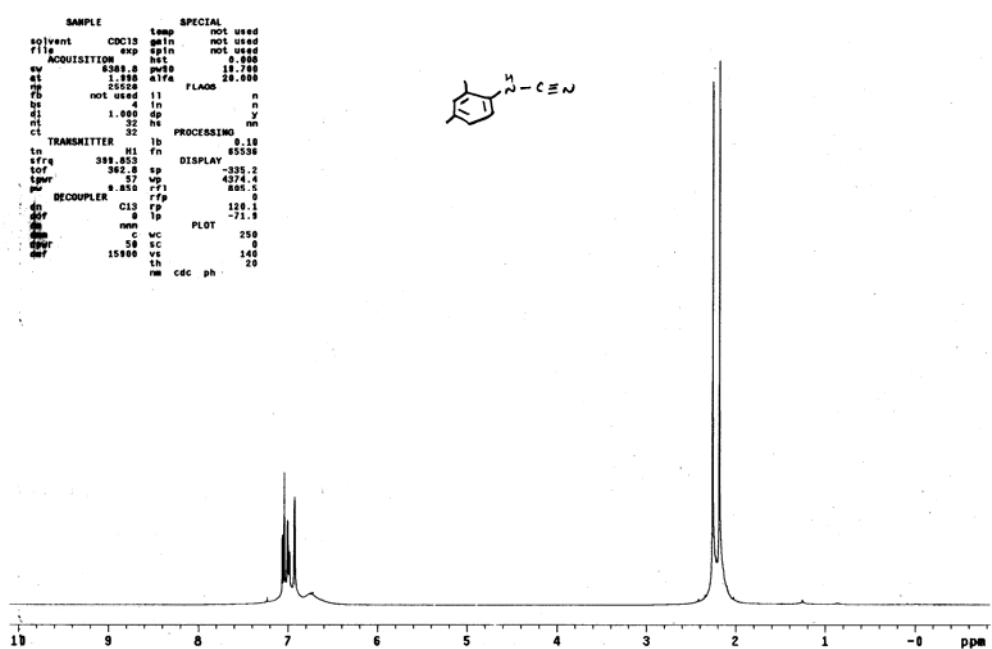
```



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



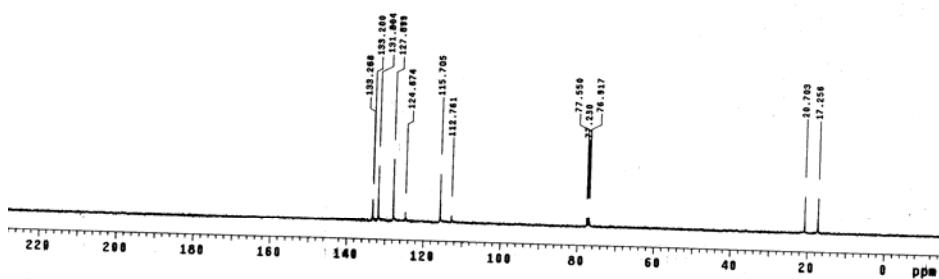
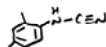
2,4-Dimethyl-phenyl cyanamide (14a):¹H NMR (CDCl₃, 400 MHz):



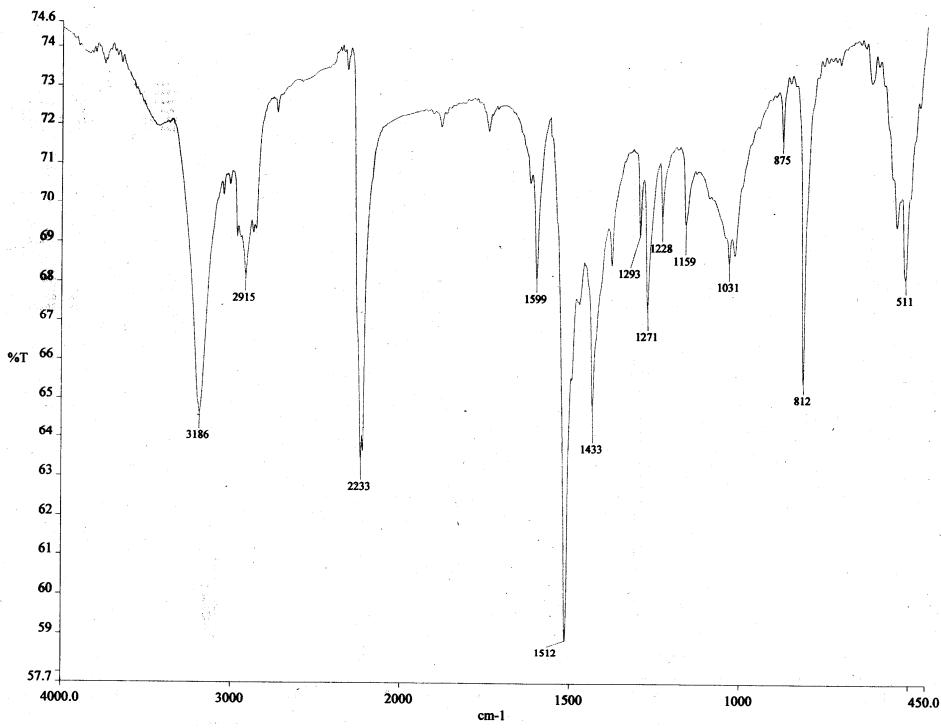
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2,4-Dimethyl-phenyl cyanamide (14a): ^{13}C NMR (CDCl_3 , 100 MHz):

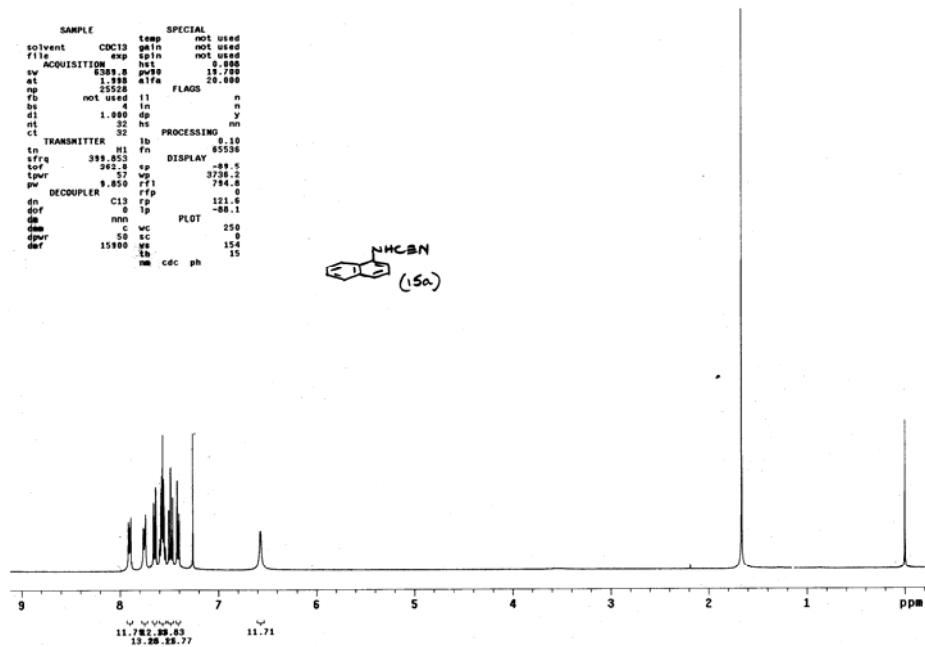
SAMPLE		SPECIAL	
Solvent	CDCl ₃	temp	not used
File		gain	not used
ACQUISITION:		ref	not used
41	2512.6	pw90	10.00
45	6070.0	psw90	20.00
46			
48	1380.0		
50	16	IN	n
51	1800	IN	n
52	272	IN	n
TRANSMITTER	C13	PROCESSING	2.00
100	TD		0.0000
105.554		DISPLAY	
105.554		-101.6	
105.554		-95.0	
4.900		-89.2	
4.900		-85.2	
DECOUPLER	H1	-77.9	
	RP	-73.9	
	PP	-69.9	
	VV	-37.5	
	WC	PLOT	
	45	250	
	8900	VE	
		10	
		1	
		NO	ph



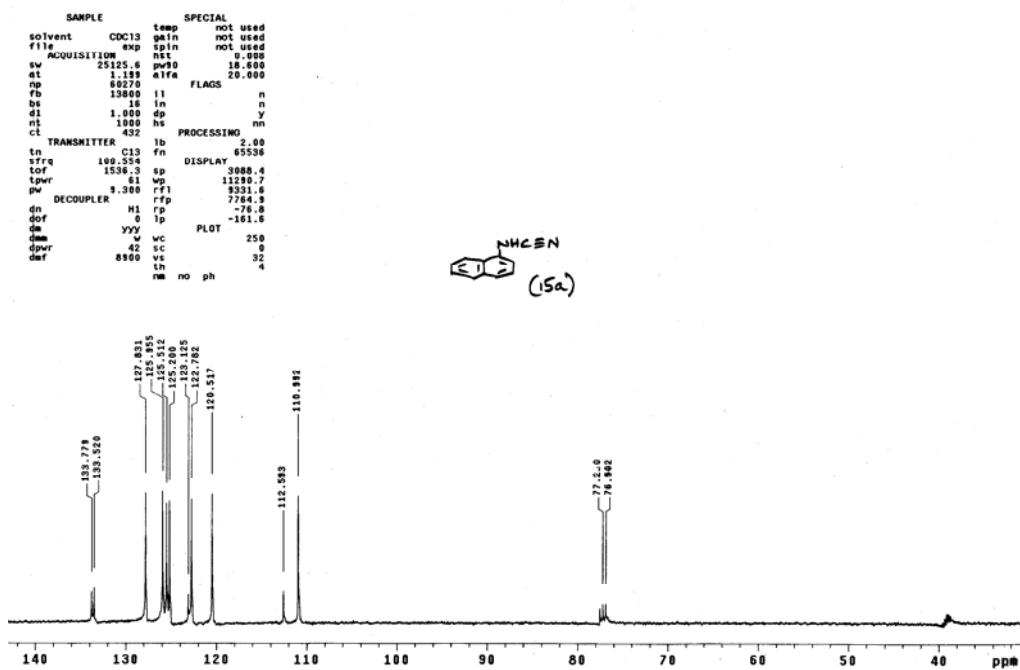
2,4-Dimethyl-phenyl cyanamide (14a): IR (KBr):



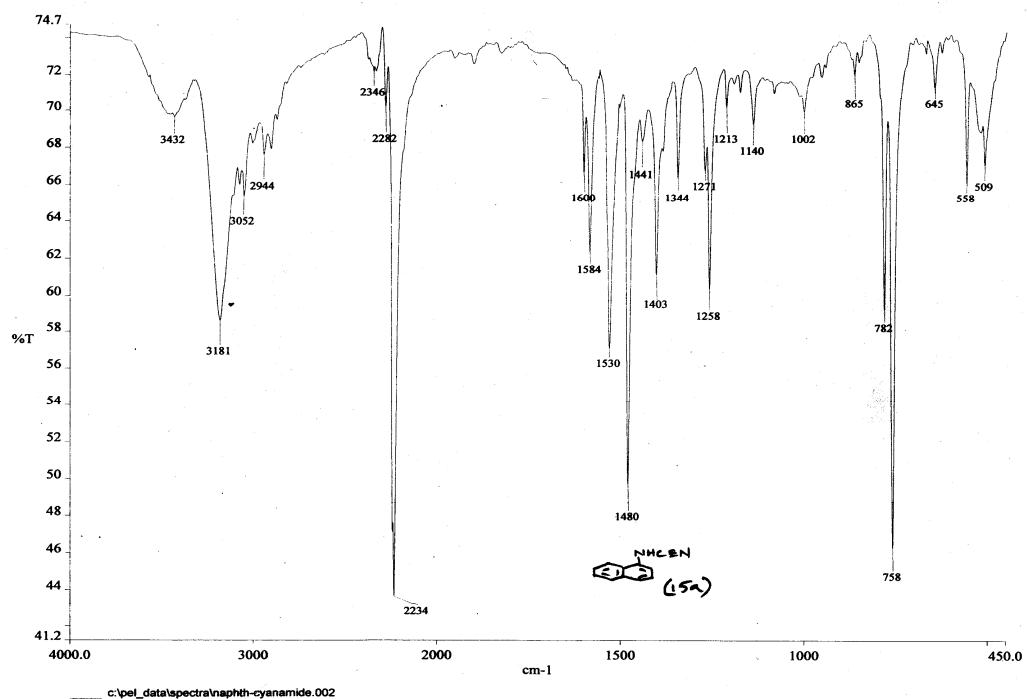
1-Naphthyl cyanamide (15a): ^1H NMR (CDCl_3 , 400 MHz):



1-Naphthyl cyanamide (15a): ^{13}C NMR (CDCl_3 , 100 MHz):

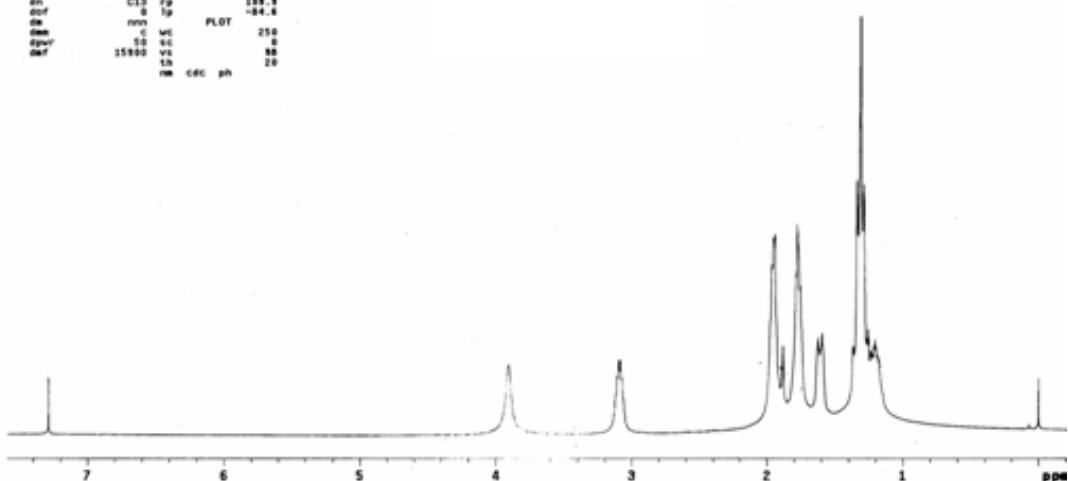
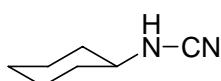


1-Naphthyl cyanamide (15a): IR (KBr):



Cyclohexyl-cyanamide (16a): ^1H NMR (CDCl_3 , 400 MHz):

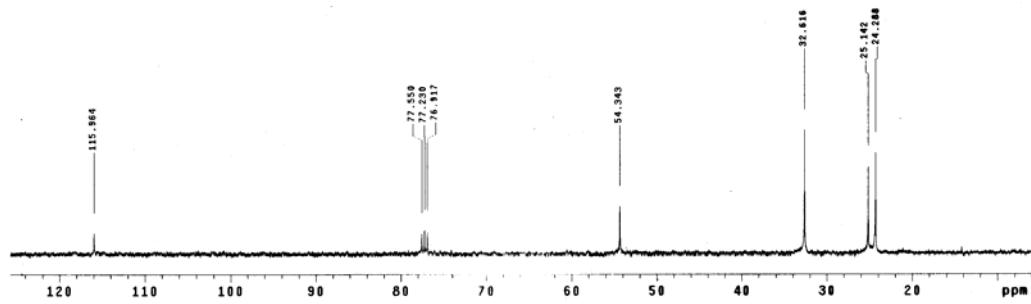
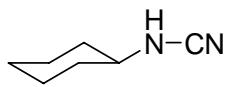
SAMPLE	SPECIAL
solvent	CDCl_3
file	exp
ACQUISITION	exp
sw	3381.8
et	1.998
ps	25528
fb	not used
bs	4
ds	1.000
rt	32
ct	32
TRANSMITTER	tb
tr	AI
ppr	399.853
spf	382.6
tpr	57
pw	8.858
DECOUPLER	C13
dn	0
ccf	0
dm	none
dpr	0
dif	15900
nm	640
cfc	ph



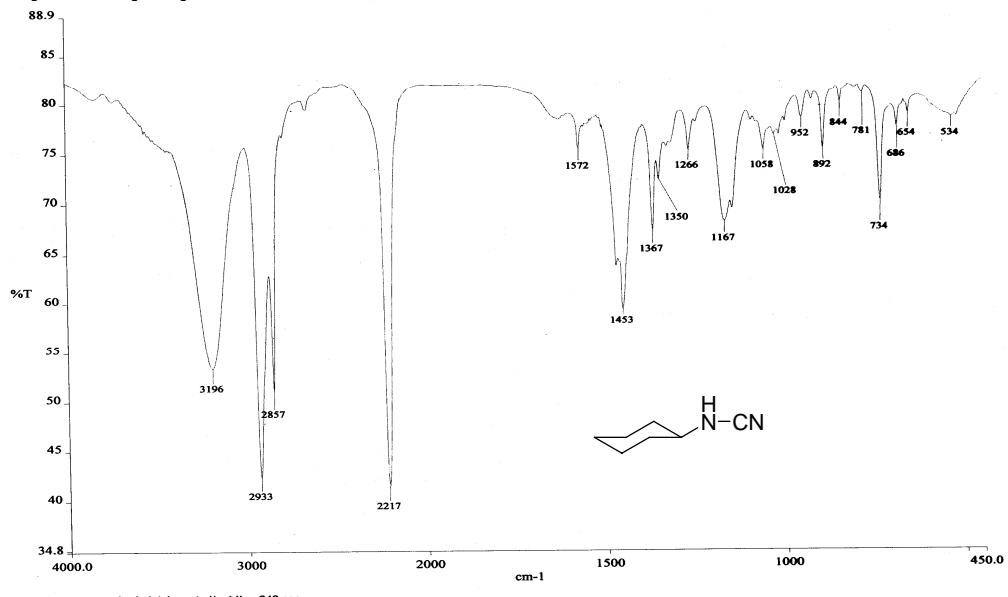
Cyclohexyl-cyanamide (16a): ^{13}C NMR (CDCl_3 , 100 MHz):

```

SAMPLE          SPECIAL
solvent        CDCl3  temp      not used
file           exp   gain      not used
              spin   not used
ACQUISITION   h1t   0.300
sw            21125.6  w0     10.000
et            1.195  a1f0   20.000
np            6000   flags
fb            13800  11      n
bs             16     dp      y
di            1.000  nn
nt            10000  ht
ct            272    PROCESSING
              1b      2.00
tr            C13   fn      65536
sfreq        100.554  DISPLAY
t0f          1536.3  tp      594.0
tpwr         1000000  t0     1284.6
px            9.300  r7t    3284.1
DECOUPLER    H1    rfp    7764.9
dn            H1    rp     -46.5
dof           0     p      -356.3
dm            vyy   PLOT
ds            wc      250
dper         42    cc      0
dref         8900  vs      30
nm            no    th      4
ph
    
```



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

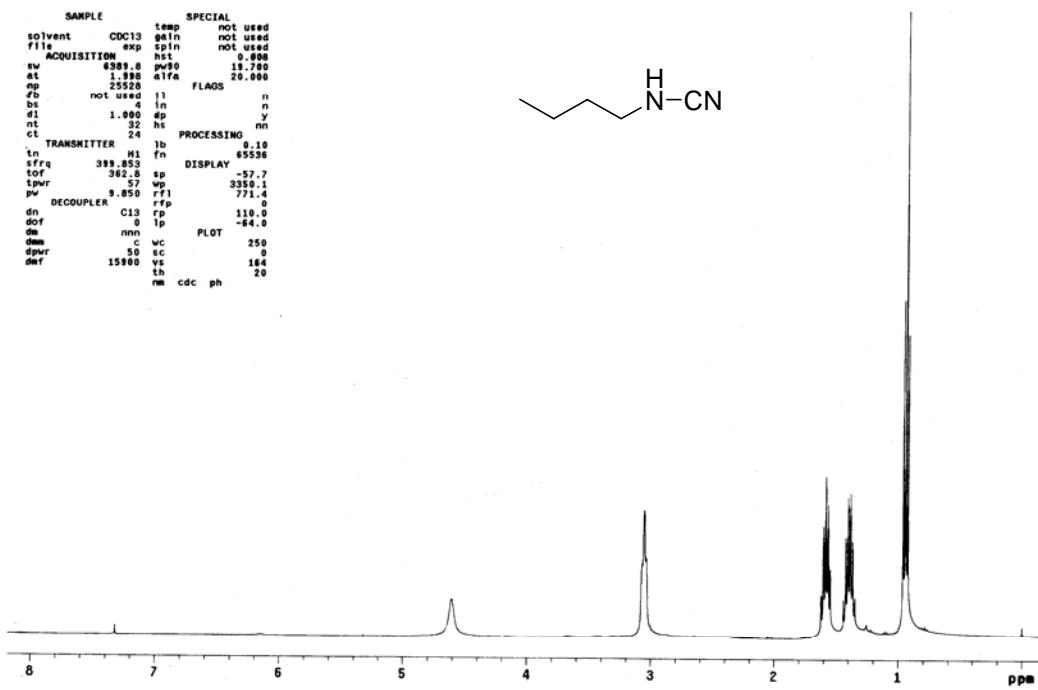
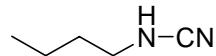


n-Butyl-cyanamide (17a): ^1H NMR (CDCl_3 , 400 MHz):

```

SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           exp     gain      not used
              spin    noci     8.000
ACQUISITION   time    pw0      1.000
              sw      5395.8  11.00
at             1.990  a1f0      20.000
swp            25528   flags
fb             not used  t1      n
bs             4       in      n
d1             1.000  dp      y
nt             1       hs      nn
ct             24      PROCESSING
TRANSMITTER   1b      rf      0.10
tn             H1      fn      65536
sfrq          399.853  DISPLAY
t0rf          362.8   sp      -57.7
tpwr          57      wp      3350.1
pwr           9.850   rfp     771.4
DECOUPLER     C13    rfp     0
dn             C13    rp      110.0
dof            0      lp      -84.0
dm             nnn    PLOT
dme            c      wc      250
dppr          50      vc      0
dpr           15900   vs      164
dfr           15900   th      20
nm            cdc   ph

```

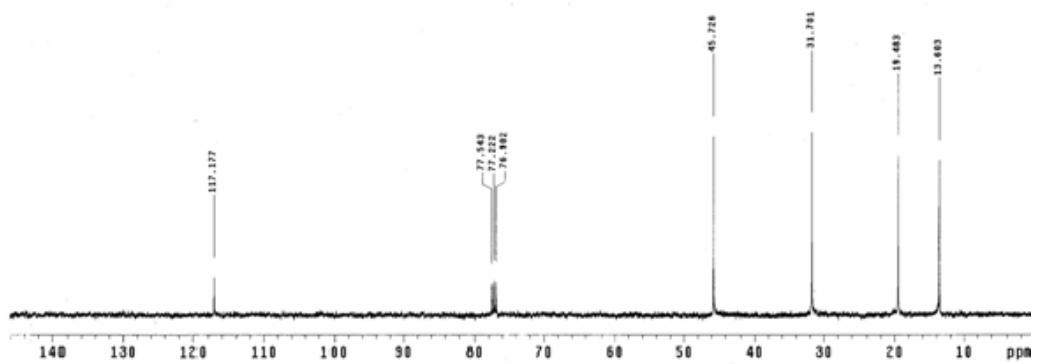


n-Butyl-cyanamide (17a): ^{13}C NMR (CDCl_3 , 100 MHz):

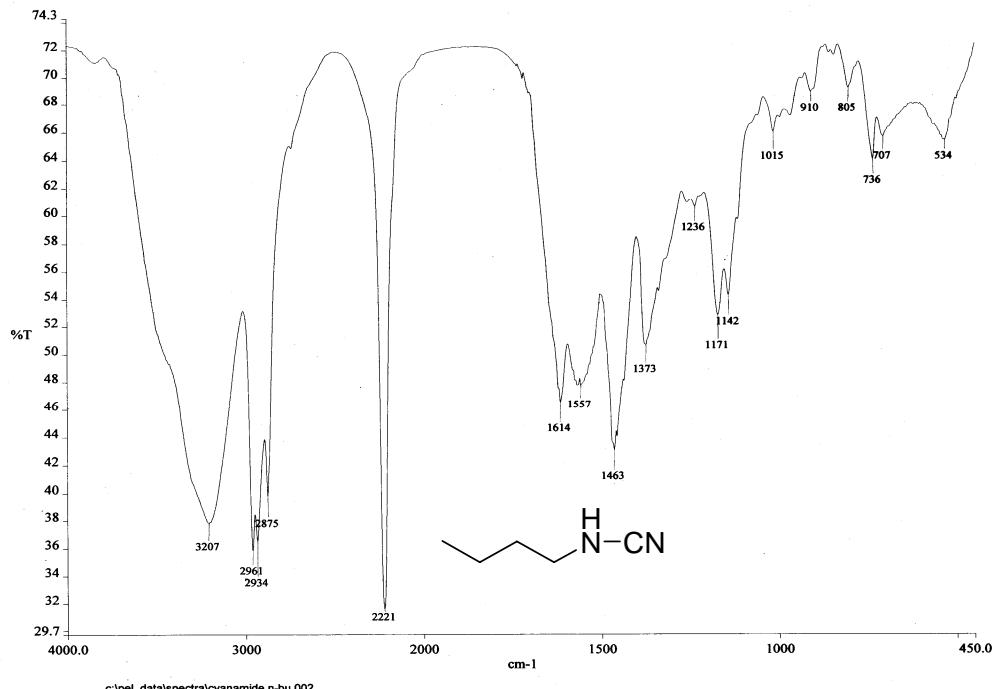
```

SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           exp     gain      not used
              spin    noci     8.000
ACQUISITION   time    pw0      18.000
              sw      25125.6  28.000
at             1.990  a1f0      28.000
swp            25270   flags
fb             13860   t1      n
bs             16      in      n
d1             1.000  dp      y
nt             1000   hs      nn
ct             176     PROCESSING
TRANSMITTER   1b      rf      2.00
tn             C13    fn      65536
sfrq          100.554  DISPLAY
t0rf          1536.4   sp      -33.3
tpwr          41      wp      14724.3
pwr           9.380   rfp     9264.9
DECOUPLER     H1      rfp     771.9
dn             H1      p      -47.5
dof            0      lp      -361.4
dm             vvv    PLOT
dme            c      wc      250
dppr          42      sc      0
dpr           8900   vs      64
dfr           8900   th      4
nm            no   ph

```



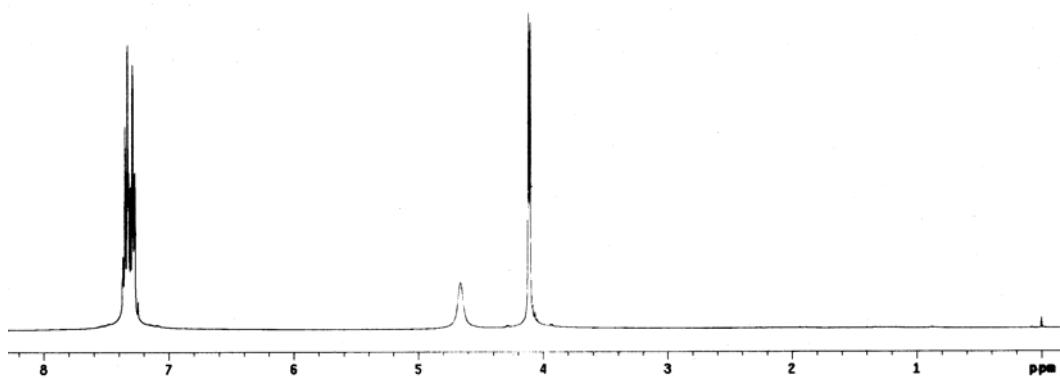
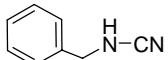
n-Butyl-cyanamide (17a): IR (KBr):



Benzyl cyanamide (18a): ¹H NMR (CDCl₃, 400 MHz):

```

SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           exp     spin     not used
ACQUISITION    exp     hst      0.000
sv            6381.0  acq2d   15.750
et            1.998  alfa    20.000
np            25528   FLAGS
rb            not used  11      n
bs             4       in      n
dt            1.000  dp      y
nt            32      hs      nn
ct            32      PROCESSING
TRANSMITTER   1b      0.10
tr            H1      f1      65536
zfrq         389.88   DISPLAY
tot            382.8  sp      -61.6
tpwr         57      wp      3408.8
pw            8.850  rf1     800.7
DECOUPLER    C13     rf2
dn            C13     rf2     115.5
dof           0       1p     -77.7
dm            mm      PLOT
dme           C      vc      250
dpwr        15900   vs      0
dmf          15900   th      75
        -nm cdc ph
    
```

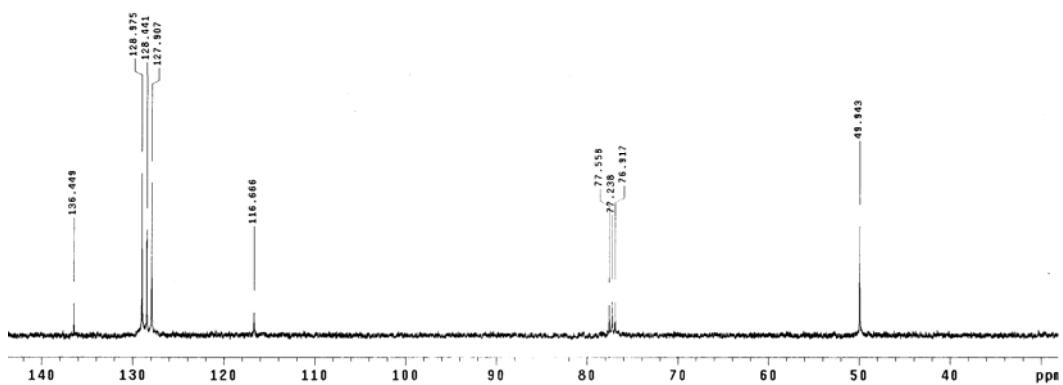
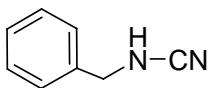


Benzyl cyanamide (18a): ^{13}C NMR (CDCl_3 , 100 MHz):

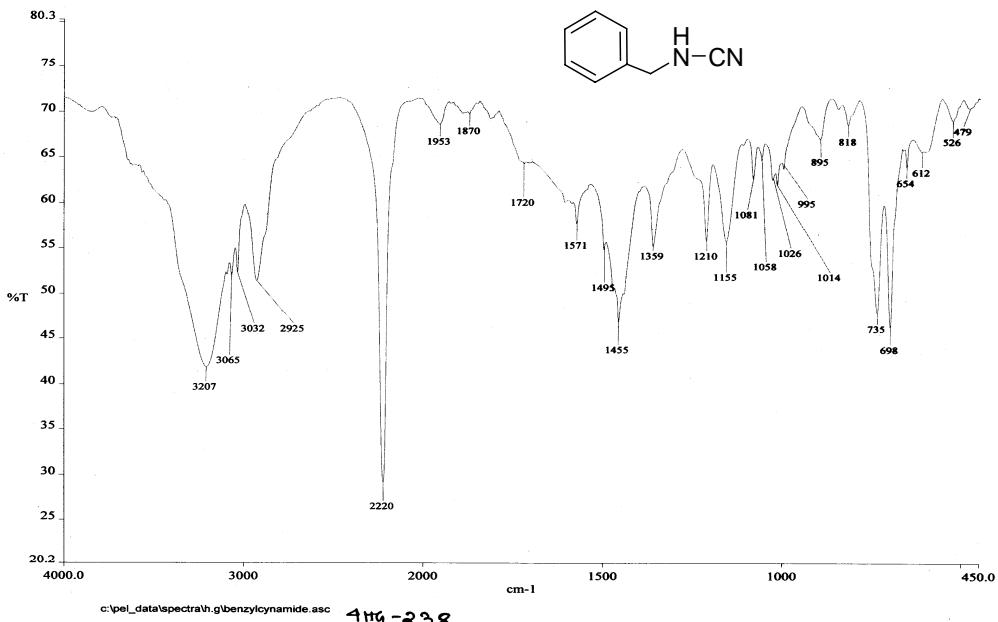
```

SAMPLE          SPECIAL
solvent        CDCl3   gain      not used
file           exp     spin      not used
              t1       t2       0.088
ACQUISITION   sw      pw90    18.660
at            1.199   alfa   20.000
sp            6024    flags
fb            19800   11      n
bs             16     in      n
d1            1.000   dp      y
rt            30.000   hs      nn
ct            192    PROCESSING
TRANSMITTER   1b      fm      2.00
in            100.513  fm      65536
stf            100.513  sp      DISPLAY
t0f            1536.3   sp      2722.6
tpwr           61     wp      11819.0
pw            1.380   r1f     500.1
DECOUPLER      HI     rfp     7784.9
dn            -54.0   rp      -54.0
dof             0      lp      -334.6
da            yyy     PLOT
dmm            v      wc      250
dpwr           42     sc      0
def            8900   vs      37
th            no     ph      3

```



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

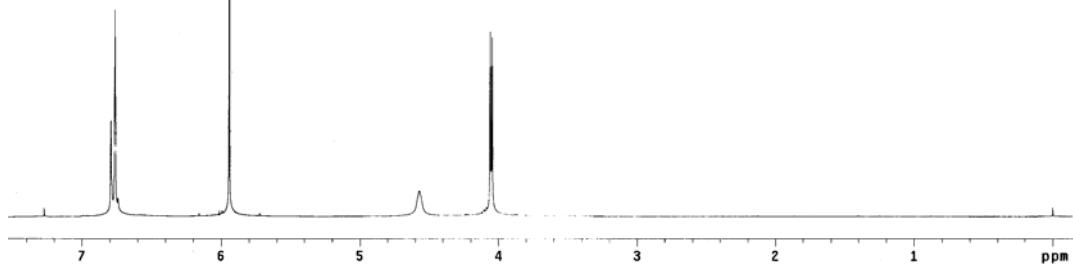
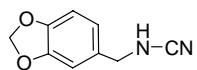


Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a): ^1H NMR (CDCl_3 , 400 MHz):

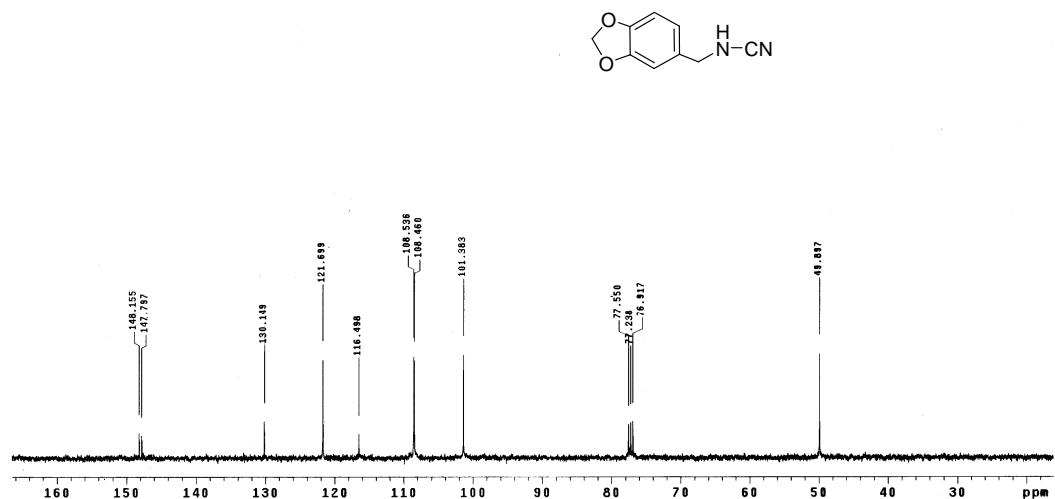
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SAMPLE          SPECIAL
solvent        CDCl3   temp      not used
file           exp      gain     not used
              spw1n   noise    0.008
ACQUISITION   preset  pw90    15.700
sw            6389.8  pw1f0   20.000
at             1.998   a1f0
np             2552    flags
fb            not used  l1      n
bs             4       in      n
d1            1.000   dp      y
nt             32      hs      nn
ct             32      PROCESSING
tr            TRANSMITTER 1b      0.10
tw            319.853  H1      65536
sfre          319.853  DISPLAY
t0f           362.8   sp      -58.7
tpwr          57      wp      3081.4
pw            9.850   r1      780.3
DECOUPLER     C13     rfp     0
dn            C13     rp      123.0
dof           0       ip      -37.4
de            nnn     plot
dme           c       wc      250
dpwr          50      sc      0
dmr           15900   ts      114
dtm           th      ph      20
nm            cdc

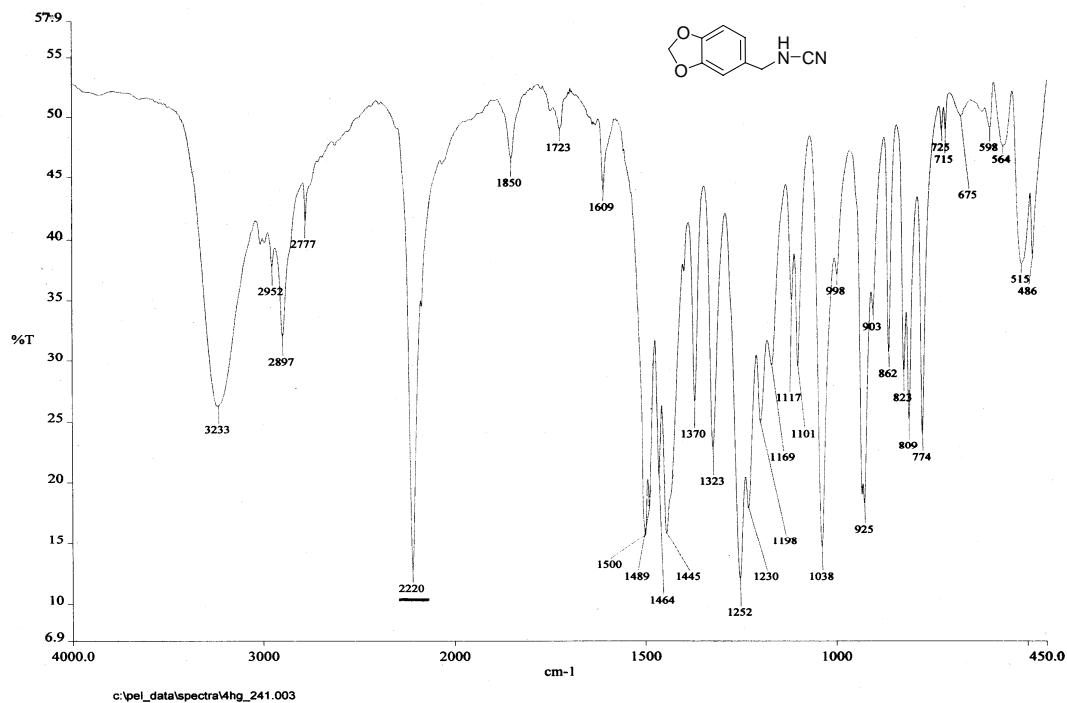
```



Benzo[1,3]dioxol-5-ylmethyl-cyanamide (19a): ^{13}C NMR (CDCl_3 , 100 MHz):



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

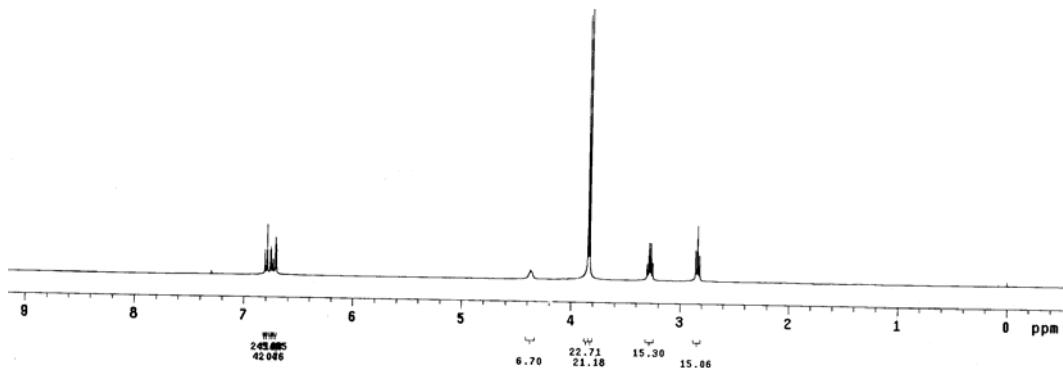
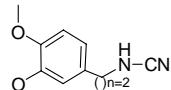


3,4-Dimethoxyphenylethylcyanamide (20a): ^1H NMR (CDCl_3 , 400 MHz):

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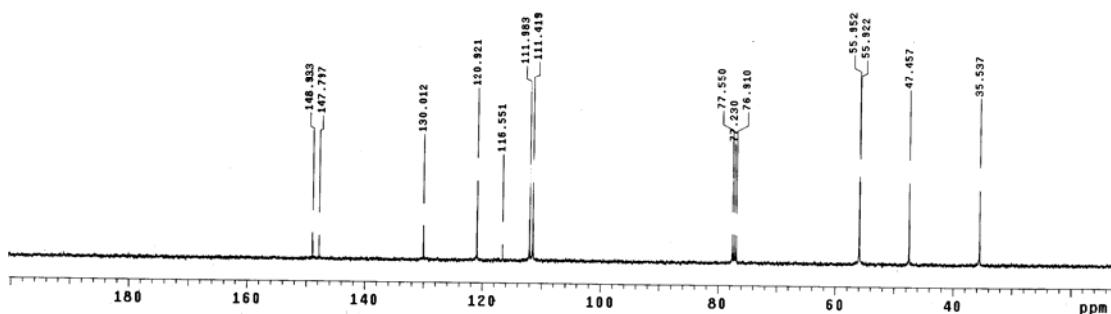
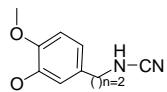
SAMPLE          SPECIAL
solvent      CDCl3   temp    not used
file          gain    not used
ACQUISITION   exp    spin   not used
sw            6388.0  hft   0.008
at            1.598   a17e  19.700
np            25528   FLAGs
fb            not used l1      n
bs            in      n
di            1.000   dp      y
nt            32     hs      nn
ct            32     PROCESSING
TRANSMITTER   1b      0.10
tn            H1     fn      65536
sfrq         388.85  DISPLAY
t0f           392.3  sp      211.0
tpwr          61     wp      3856.2
pw            9.850   rfp    780.6
DECOUPLER    C13    rfp    0
d1            0      1p     -86.8
dm            nnn    wc     250
dpw           50     sc     0
def           15800   vs     63
d1f          15.06   th     20
nm            cdc   ph
    
```



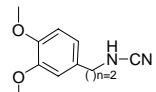
3,4-Dimethoxyphenylethylcyanamide (20a): ^1H NMR (CDCl_3 , 100 MHz):

```

SAMPLE          SPECIAL
solvent      CDCl3   temp    not used
file          gain    not used
ACQUISITION   exp    spin   not used
sw            25125.6  hft   0.008
at            1.198   a17e  18.800
np            60270   FLAGs
fb            138C6  l1      n
bs            1.000   dp      y
di            2000   hs      n
ct            272    PROCESSING
TRANSMITTER   1b      2.00
tn            C13    fn      65536
sfrq         109.554  DISPLAY
t0f           1536.3  sp      1284.8
tpwr          61     wp      1184.8
pw            9.000   rfp    5286.4
DECOUPLER    H1     rfp    7764.8
d1            0      1p     -43.0
dm            YYY    PLOT   -355.3
dpw           42     sc     250
def           8500   vs     0
d1f          15.06   th     20
nm            no    ph
    
```



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



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