

## Electronic Supplementary Information

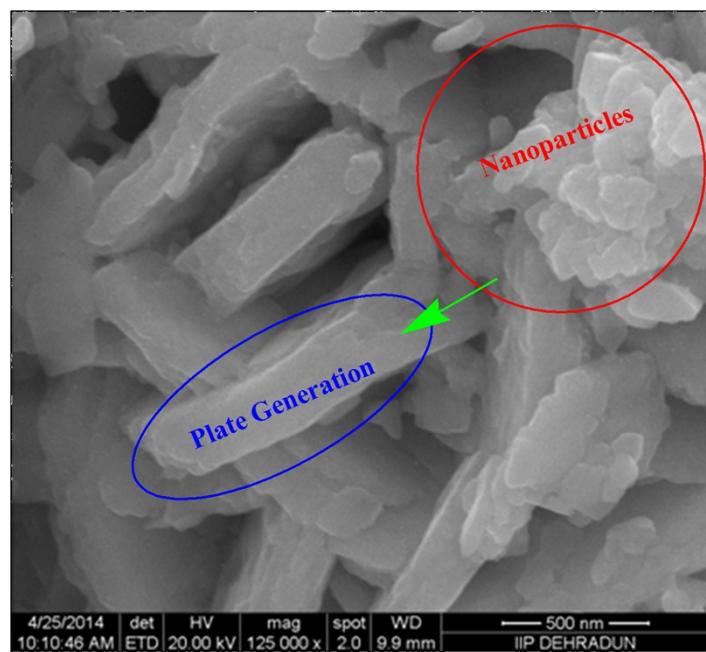
### **Chloride Promoted Room Temperature Preparation of Silver Nanoparticles on Two Dimensional Tungsten Oxide Nanoarchitechture for the Catalytic of Oxidation of Tertiary N-compounds to N-oxides**

Shilpi Ghosh, Shankha S. Acharyya, Malika Kumar and Rajaram Bal\*

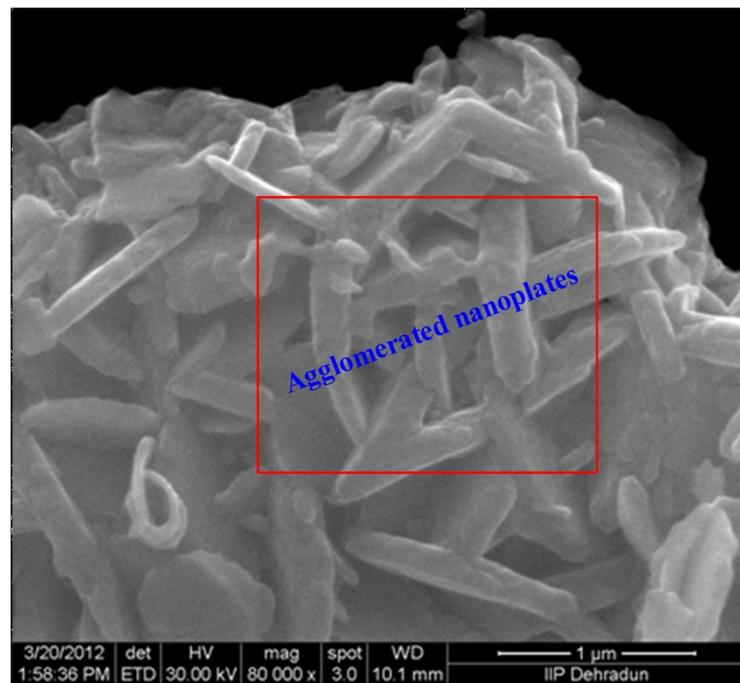
*Catalytic Conversion & Processes Division, CSIR-Indian Institute of Petroleum, Dehradun  
248005, India*

Corresponding author. Tel.: +91 135 2525917; Fax: +91 135 2660202

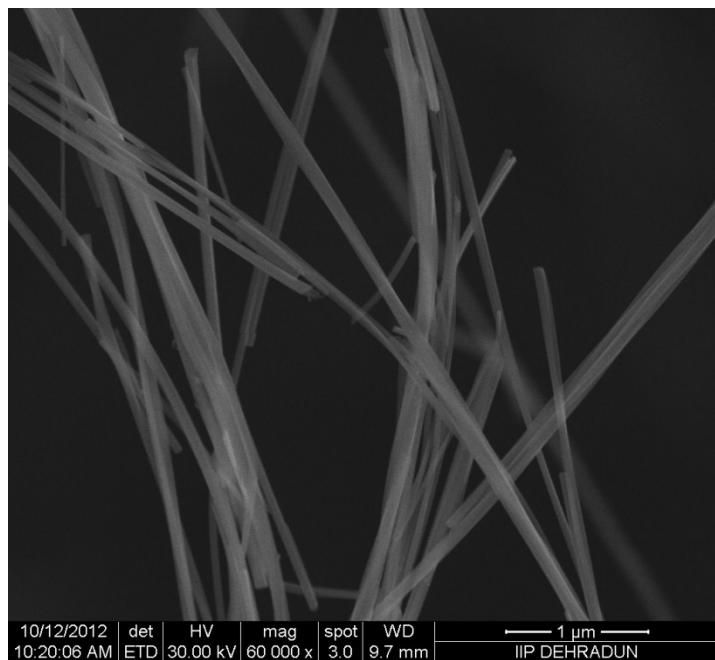
E-mail addresses: [raja@iip.res.in](mailto:raja@iip.res.in)



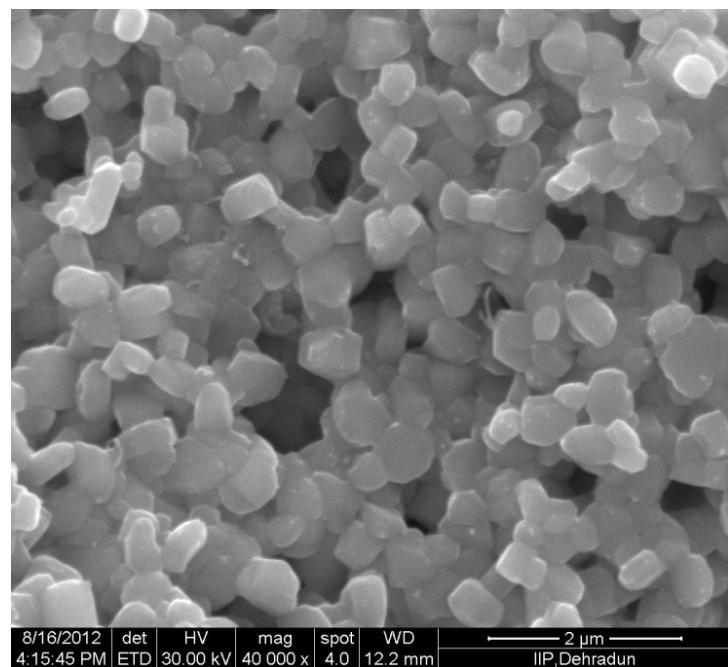
**Fig. S1.** SEM images of Ag/WO<sub>3</sub> catalyst after 1 h stirring time.



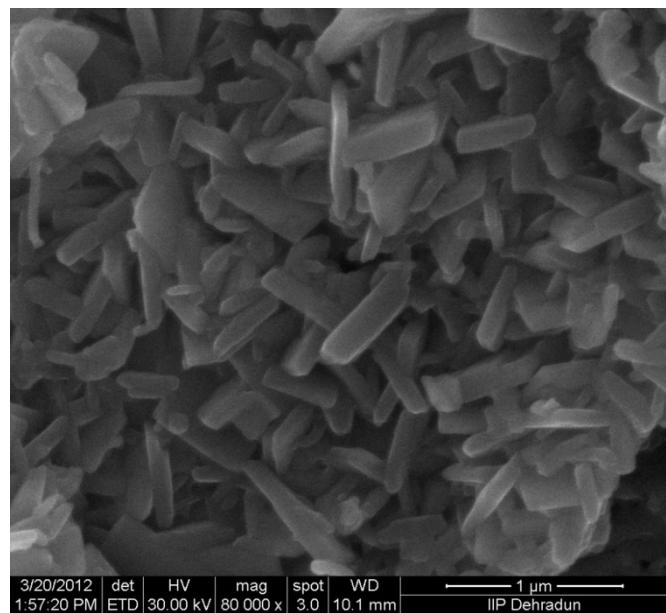
**Fig. S2.** SEM images of Ag/WO<sub>3</sub> catalyst after 10 hr stirring.



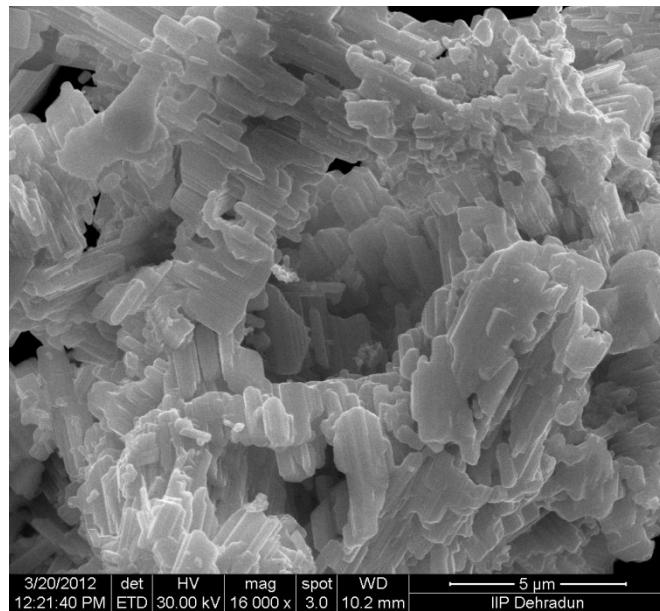
**Fig. S3.** SEM images of Ag/WO<sub>3</sub> catalyst in a basic solution.



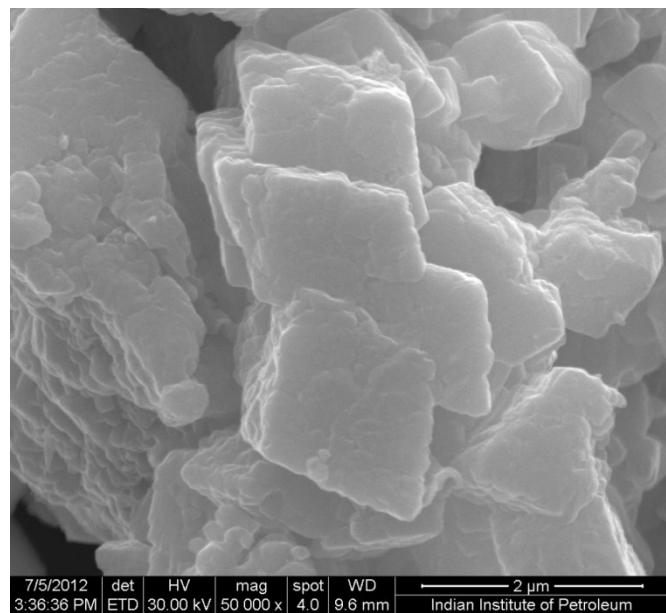
**Fig. S4.** SEM images of Ag/WO<sub>3</sub> catalyst without addition of chloride.



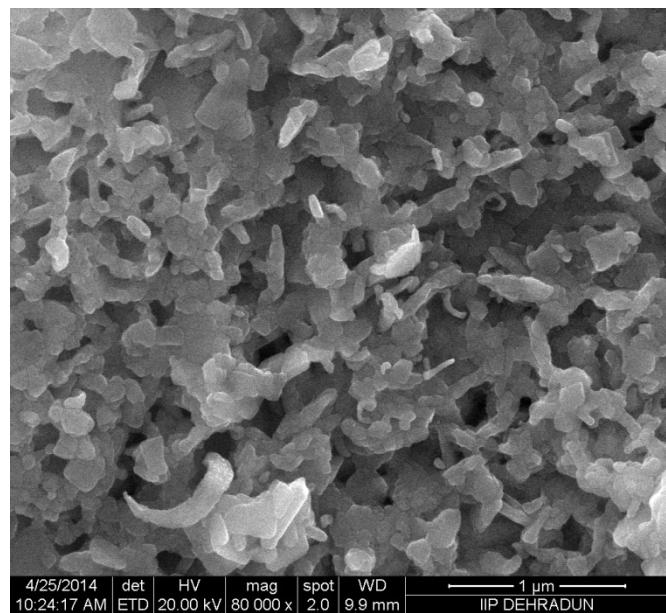
**Fig. S5.** SEM images of Ag/WO<sub>3</sub> catalyst in HF medium.



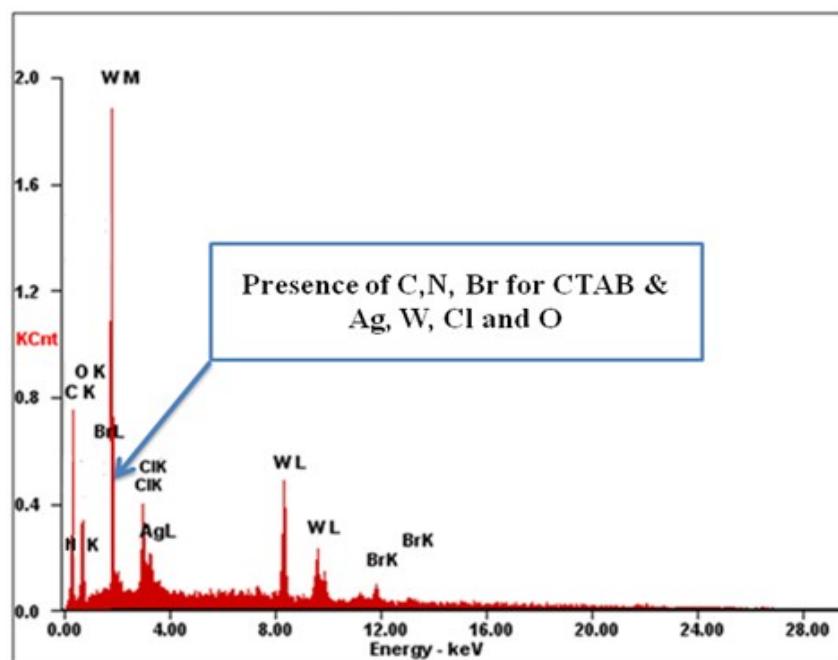
**Fig. S6.** SEM images of Ag/WO<sub>3</sub> catalyst without addition of CTAB.



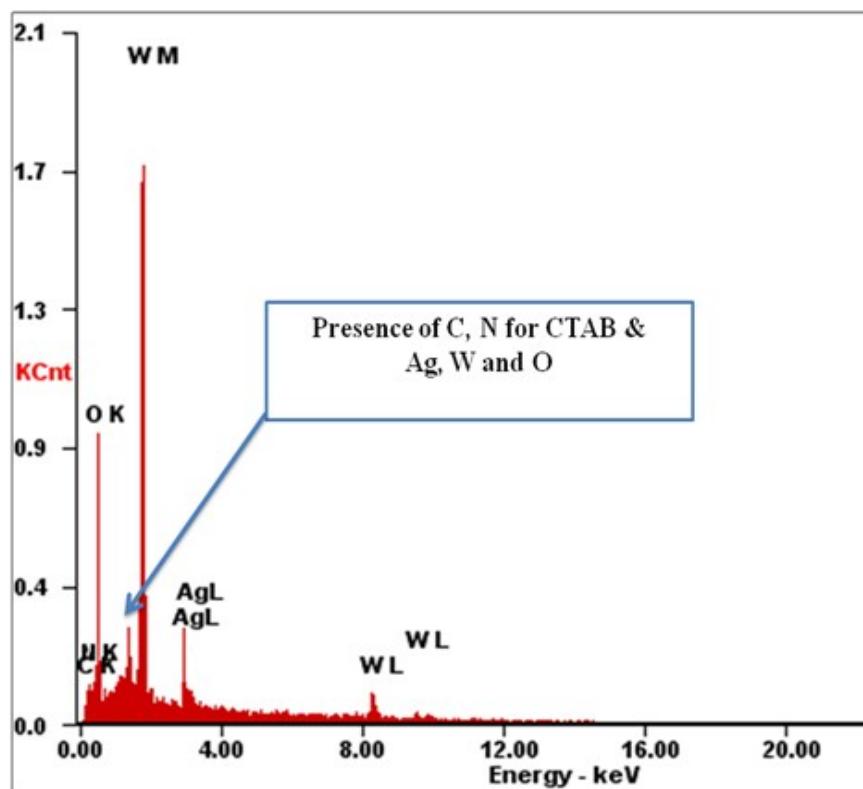
**Fig. S7.** SEM images of Ag/WO<sub>3</sub> catalyst when Ag: CTAB=1:5.



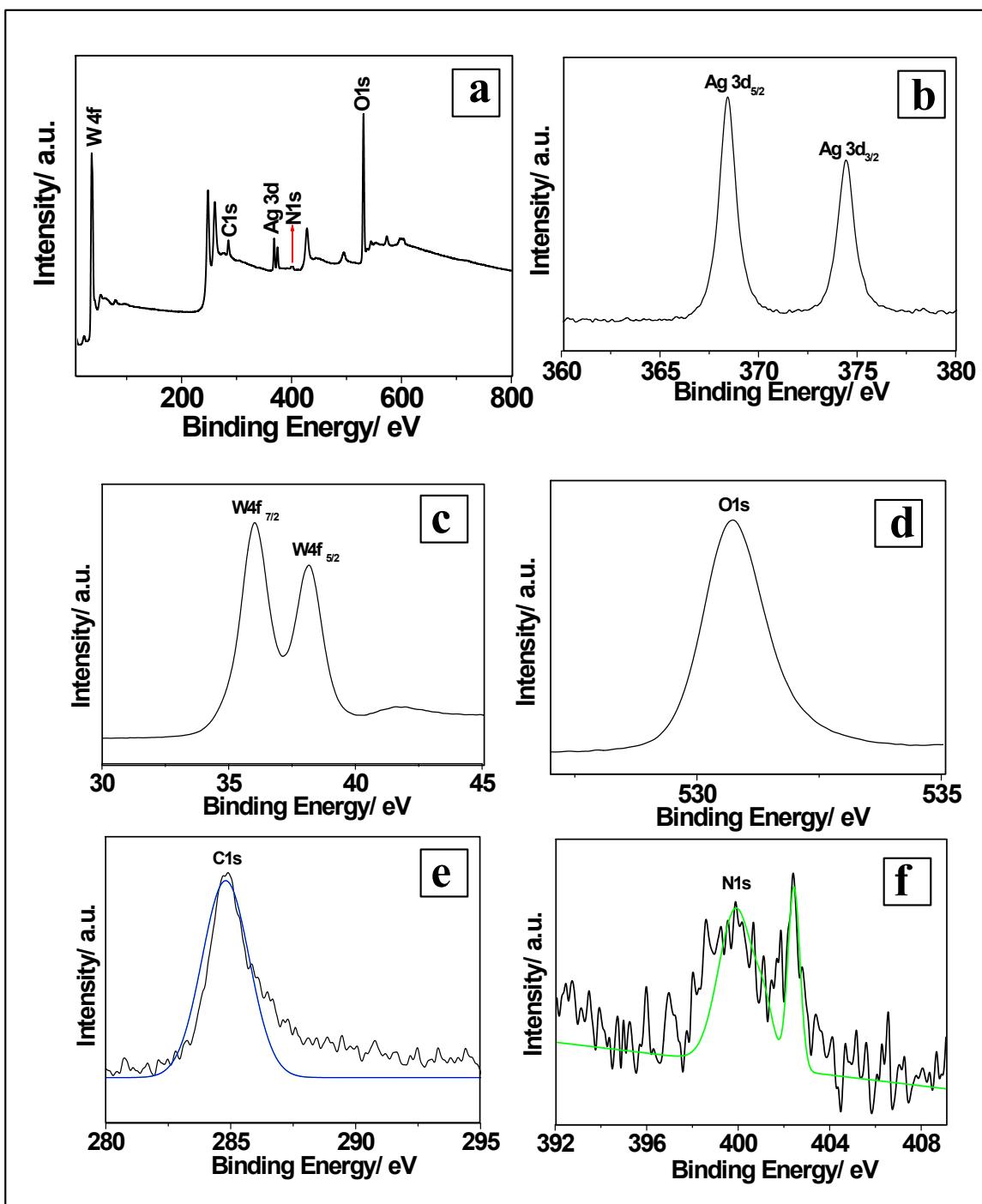
**Fig. S8.** SEM images of Ag/WO<sub>3</sub> catalyst when Ag: CTAB=1:0.5.



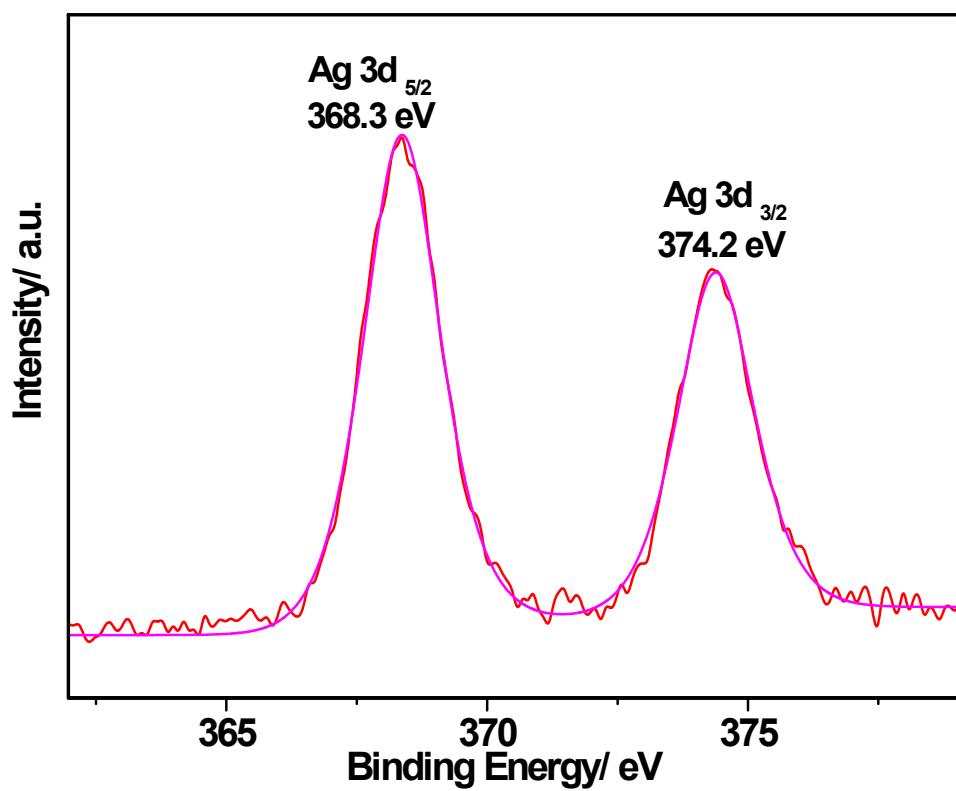
**Figure S9.** SEM-EDAX of Ag-W catalyst taken in the mid of catalyst preparation.



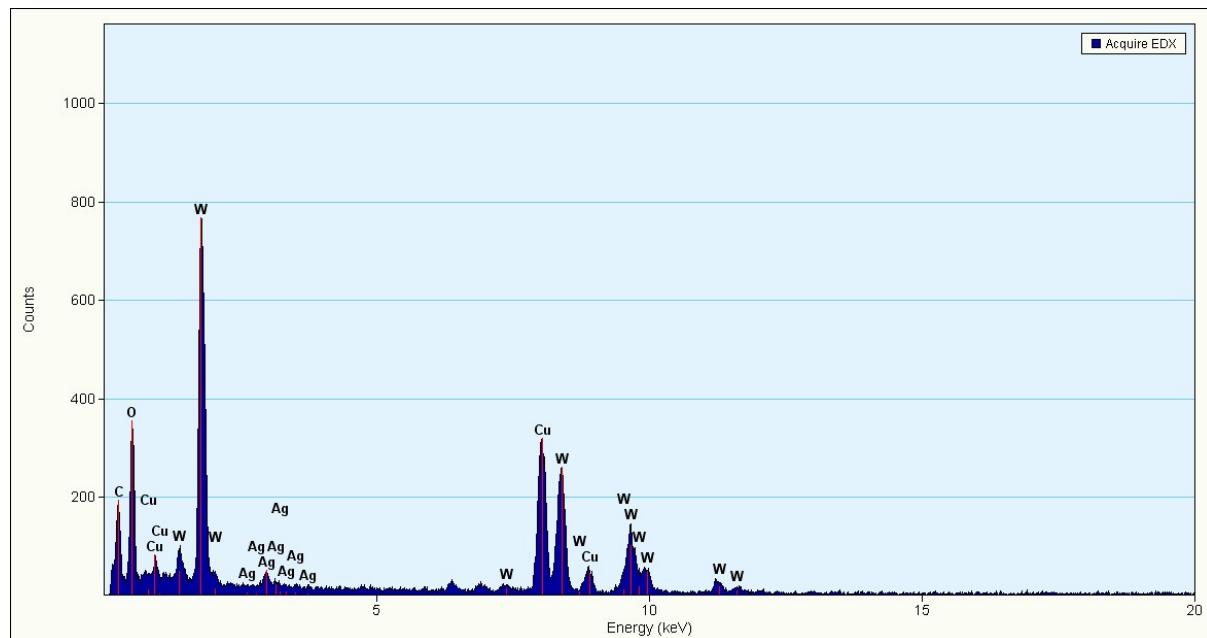
**Figure S10.** SEM-EDAX of Ag-W catalyst taken just before calcination.



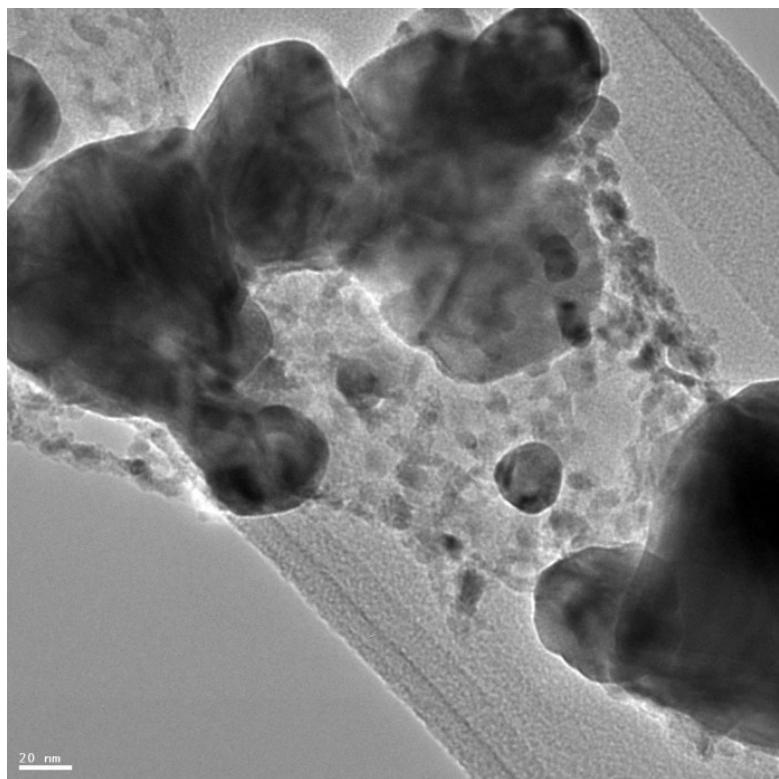
**Figure S11.** a) XPS survey spectrum of Ag/WO<sub>3</sub> before calcination catalyst, b) Ag 3d, c) W 4f , d) O <sub>1s</sub> , e) c1s and f) N1s.



**Figure S12.** a) XPS of Ag/WO<sub>3</sub> spent catalyst (Ag 3d).



**Fig. S13.** TEM EDX image of Ag/WO<sub>3</sub> catalyst.



**Figure S14.** TEM image of Ag-W catalyst prepared by conventional impregnation method.

## **NMR data of N-oxides:**

**Pyridine N-oxide** (Table 1 entry 1):  $^1\text{H-NMR}$  (500 MHz, DMSO-d<sub>6</sub>):  $\delta$  7.35-7.44 (m, 3H); 8.22-8.23 (m, 2H).

**3-Picoline N-oxide** (Table 1 entry 2):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  2.33 (s, 3H), 7.12-7.37 (d,  $J$ = 7.5 Hz, 2H); 8.06-8.09 (d, 2H).

**3-Chloro Pyridine N-oxide** (Table 1 entry 3):  $^1\text{H-NMR}$  (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.15-7.23 (d,  $J$ = 7.5 Hz, 2H); 8.05-8.07 (d,  $J$ = 8.0 Hz, 1H); 8.195 (s, 1H).

**Isoquinoline N-oxide** (Table 1 entry 4):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  7.591 (m, 2H); 7.63 (m, 1H); 7.69 (m, 1H); 7.79 (d,  $J$ = 7.5 Hz, 1H); 8.139 (m, 1H); 8.77 (s, 1H).

**Quinoxaline N-oxide** (Table 1 entry 5):  $^1\text{H-NMR}$  (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.24 (m, 1H); 7.85 (m, 2H); 8.03 (d,  $J$ = 8.0 Hz, 1H); 8.15 (s, 1H); 8.36(s, 1H).

**Pyrazine N-oxide** (Table 1 entry 6):  $^1\text{H-NMR}$  (400MHz, CDCl<sub>3</sub>):  $\delta$  8.09-8.1 (d,  $J$ =2.5 Hz, 2H);  $\delta$ 8.45-8.46 (d,  $J$ = 3.1 Hz, 2H).

**Phenazine N-oxide** (Table 1 entry 7):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  7.87 (m, 5H); 7.91 (m, 2H); 8.58 (m, 1H).

**Triphenyl amine N-oxide** (Table 1 entry 8):  $^1\text{H-NMR}$  (400 MHz, CDCl<sub>3</sub>):  $\delta$ 7.06 (m, 3H); 7.14 (m, 6H); 7.34 (m, 6H).

**N, N- dimethyl aniline N-oxide** (Table 1 entry 9):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  3.65 (s, 6 H); 7.31 (m, 1H); 7.48 (m, 2H); 7.92 (m, 2H).

**N,N- dimethyl cyclohexyl amine N-oxide** (Table 1 entry 10):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  1.176 (m, 5H); 1.32 (m,3H); 1.90 (m, 2H); 3.11(m, 7H).

**Quinclidine N-oxide** (Table 1 entry 11):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  1.90 (s, 6H); 2.04 (m, 3H); 2.08 (m, 1H); 3.40 (m, 3H).

**N,N- dimethyl butyl amine N-oxide** (Table 1 entry 12):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  0.92( t,  $J$ =2.5 Hz, 3H); 1.40 (m, 2H); 1.82 (m, 2H); 3.30 (s, 6H); 3.31 (m, 2H).

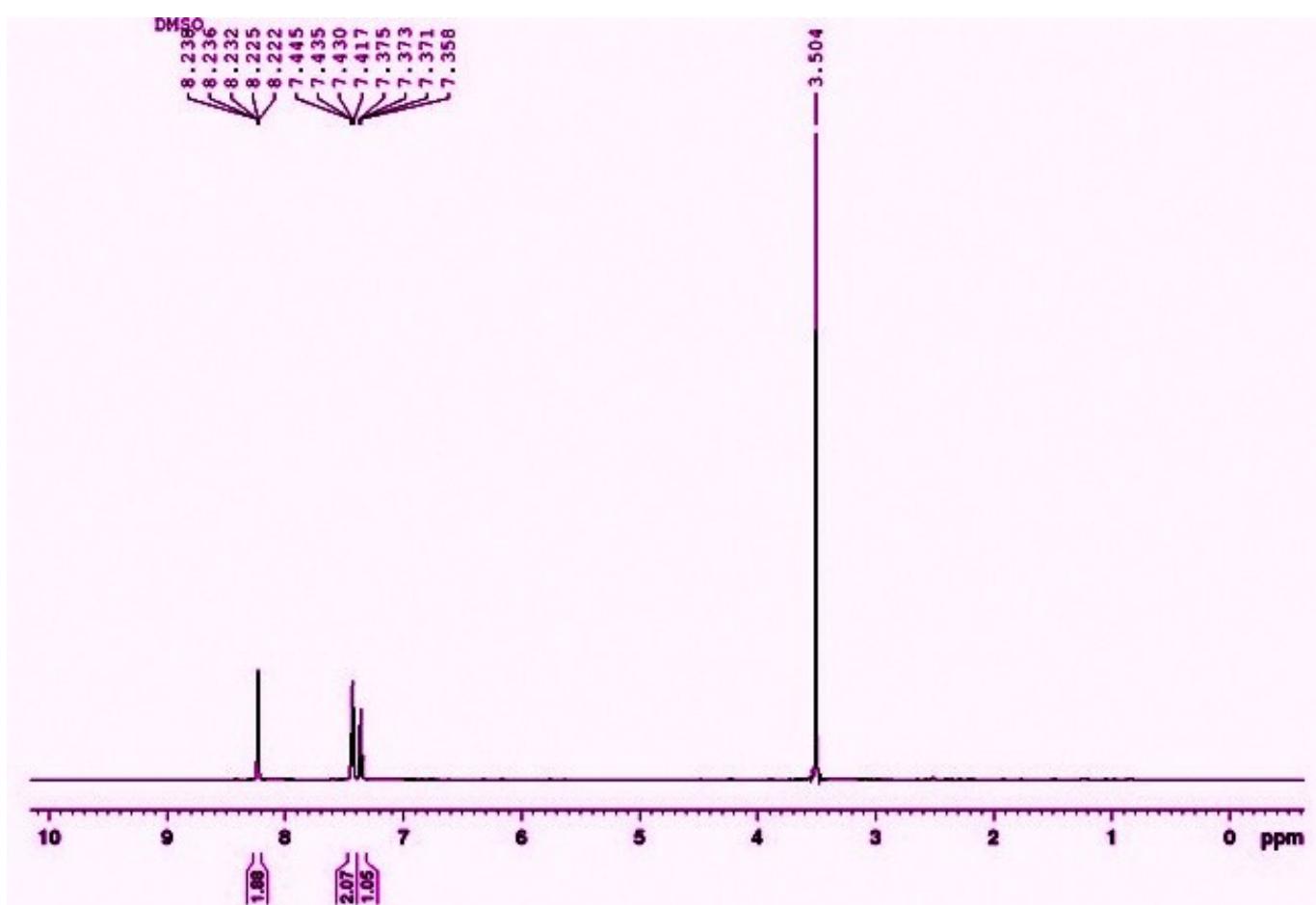
**N,N- dimethyl o-toluidine N-oxide** (Table 1 entry 13):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  1.91 (s, 3H); 3.79 (s, 6H); 7.23 (m, 3H); 7.79(m, 1H).

**N,N- dimethyl m-toluidine N-oxide** (Table 1 entry 14):  $^1\text{H-NMR}$  (500 MHz, CDCl<sub>3</sub>):  $\delta$  2.39 (s, 3H); 5.95 (s, 6H); 6.57 (d,  $J$ = 6.0 Hz, 1H); 7.33 (m, 1H); 7.63 (m, 1H); 8.43(s, 1H).

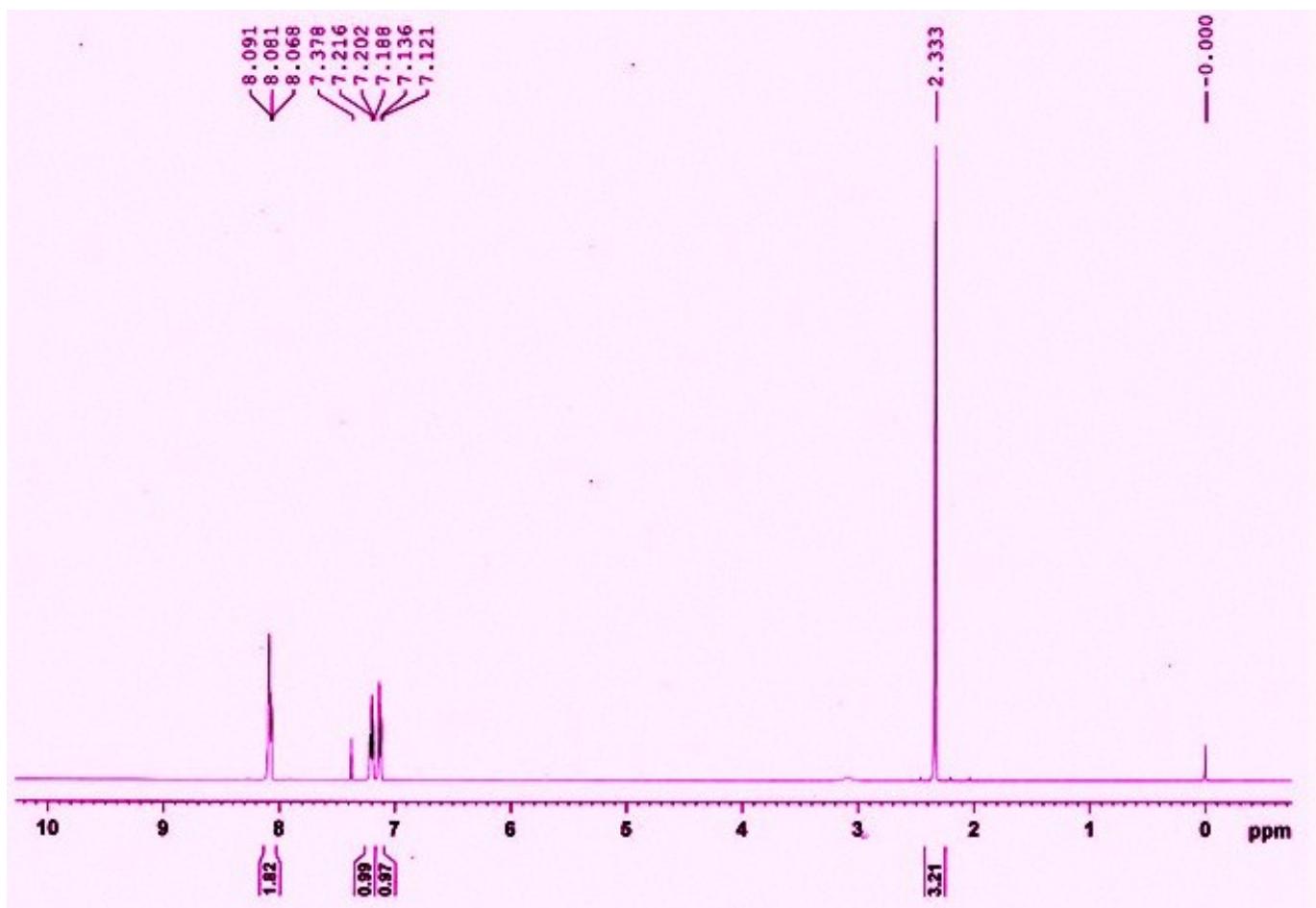
**N,N- dimethyl p-toluidine N-oxide** (Table 1 entry 15):  $^1\text{H-NMR}$  (400 MHz, CDCl<sub>3</sub>):  $\delta$  2.14 (s, 3H), 3.44 (s, 6H), 7.02-7.04 (d,  $J$ = 10.5 Hz, 2H), 7.59–7. 61 (d,  $J$ = 10.5 Hz, 2H).

**NMR spectra of N-oxides:**

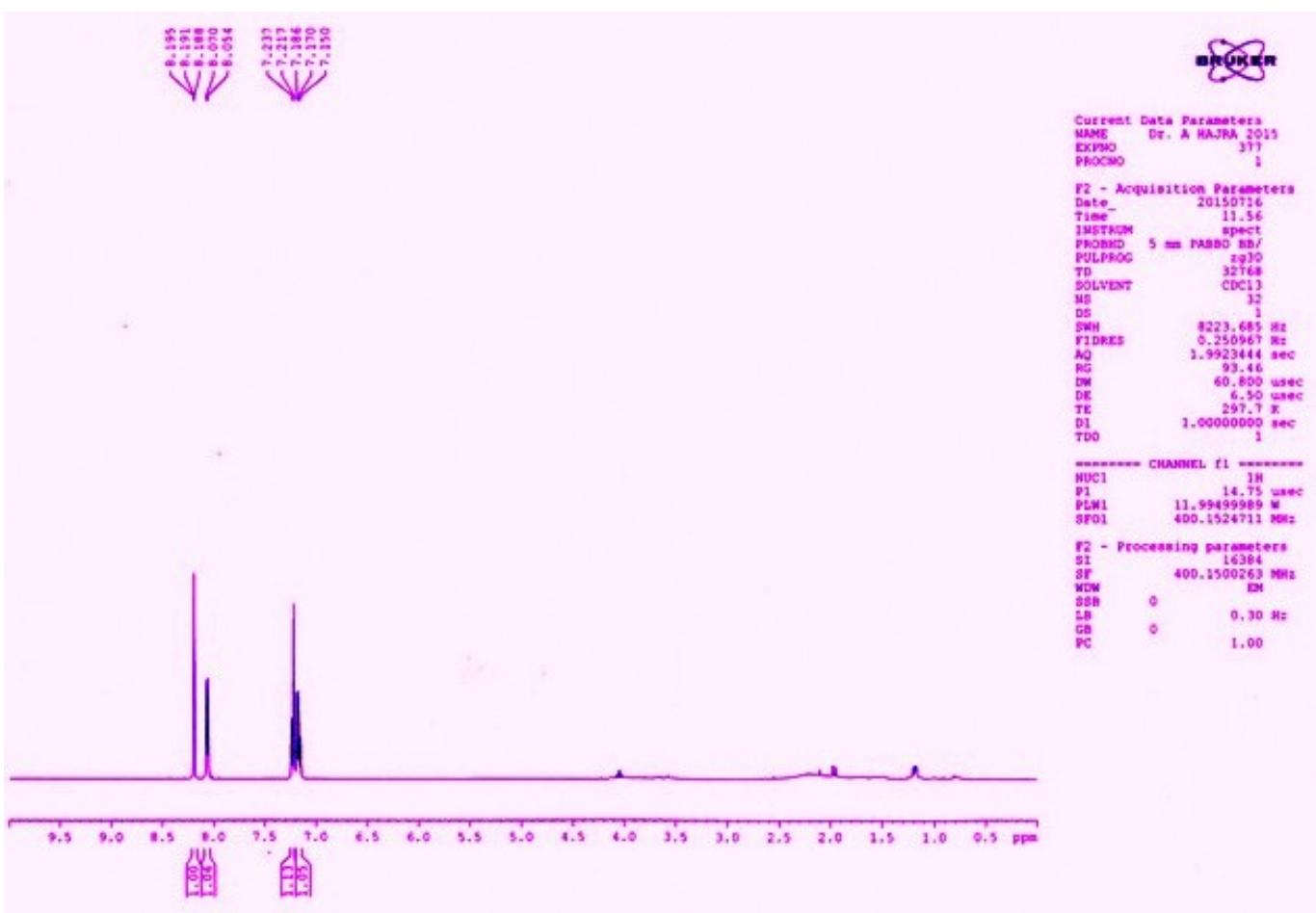
Pyridine N-oxide (Table 1 entry 1)



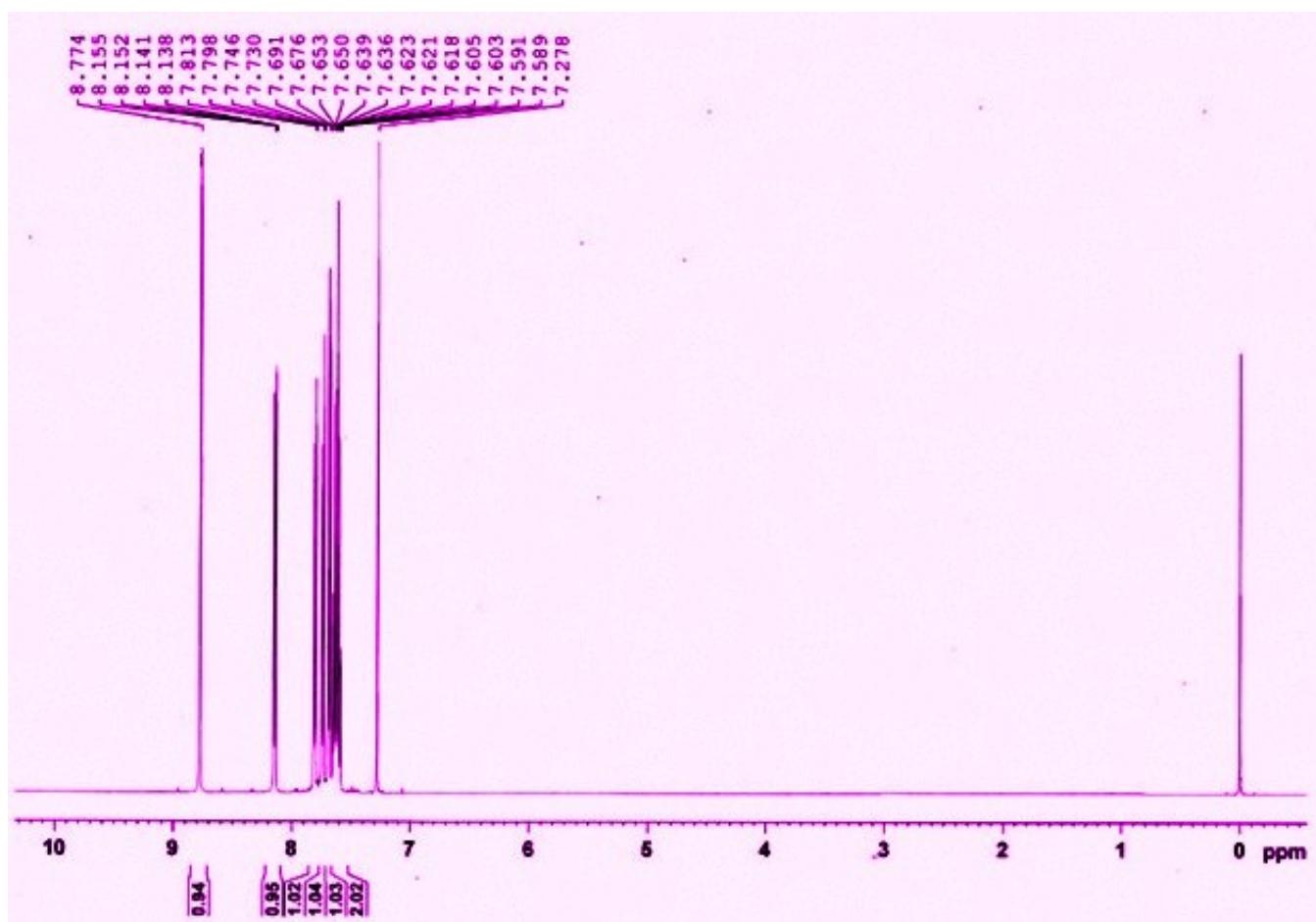
**3-Picoline N-oxide (Table 1 entry 2):**



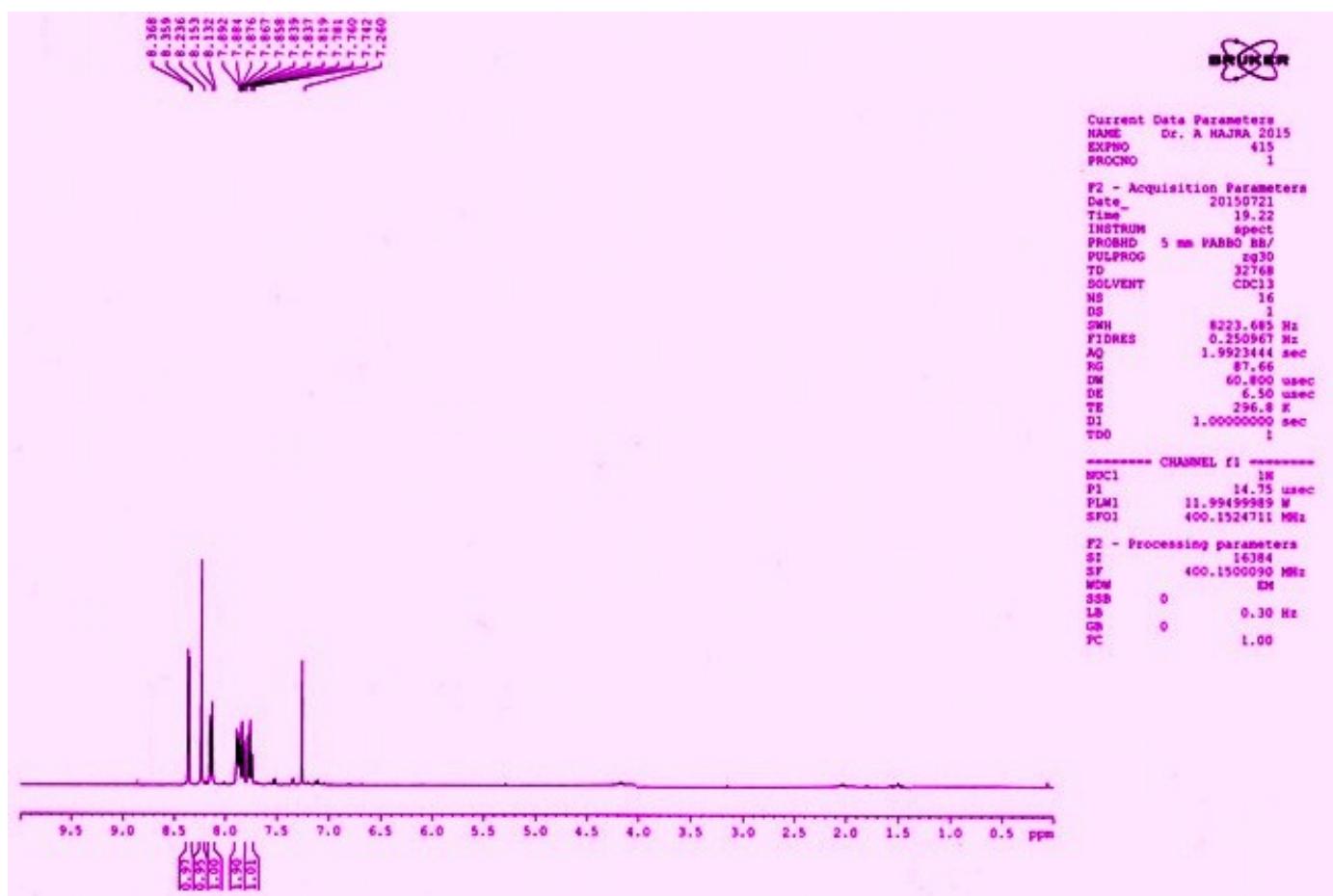
**3-Chloro Pyridine N-oxide (Table 1 entry 3)**



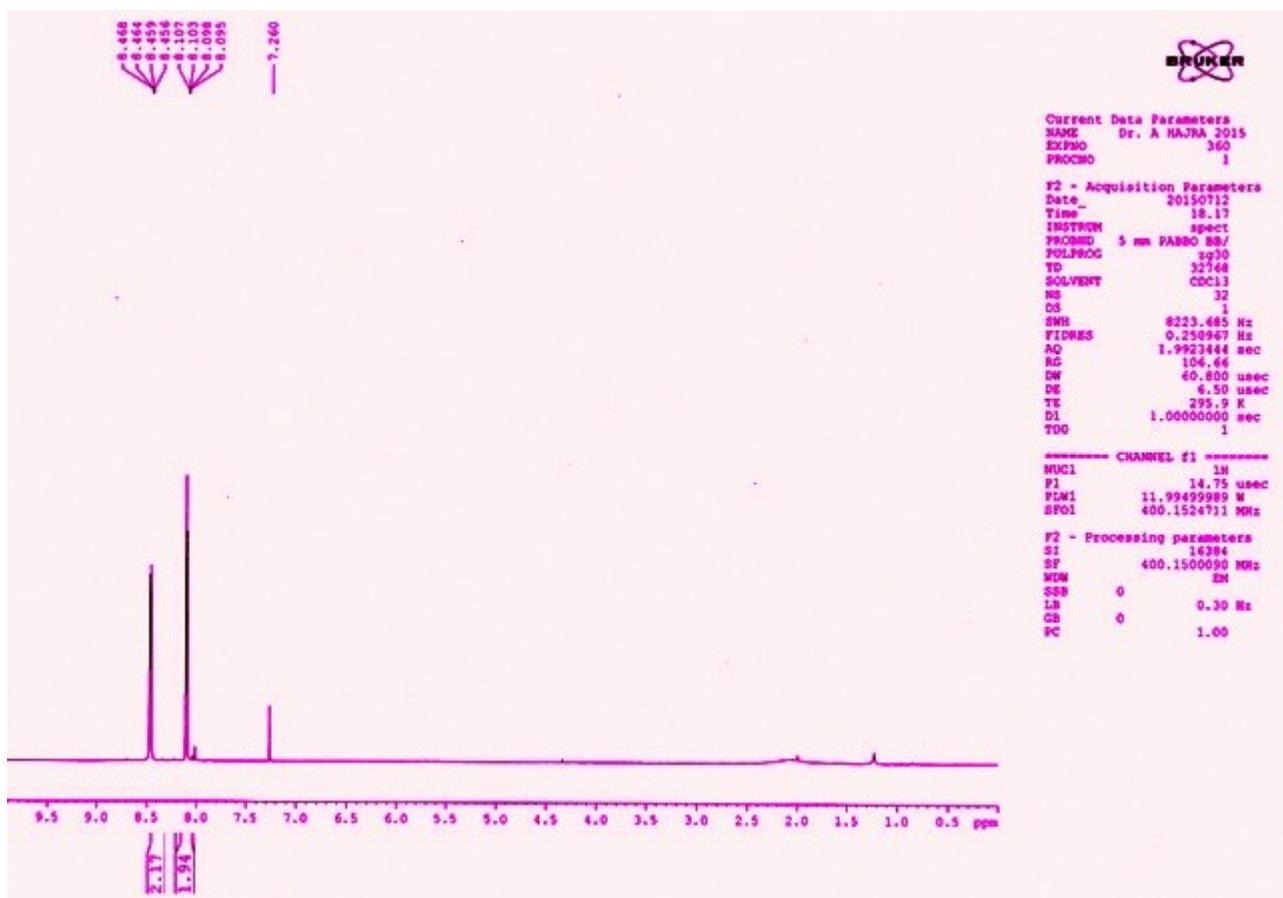
**Isoquinoline N-oxide (Table 1 entry 4):**



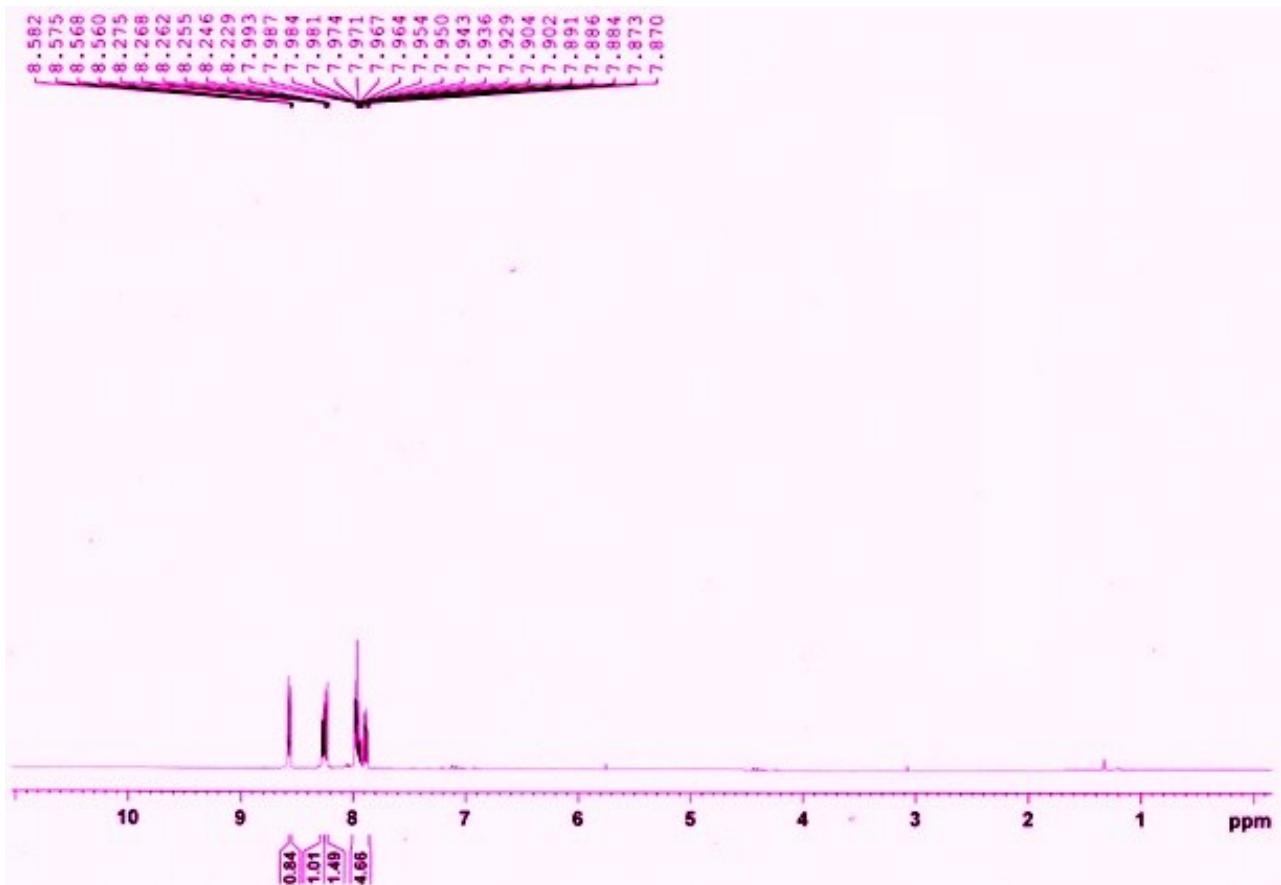
**Quinoxaline N-oxide (Table 1 entry 5)**



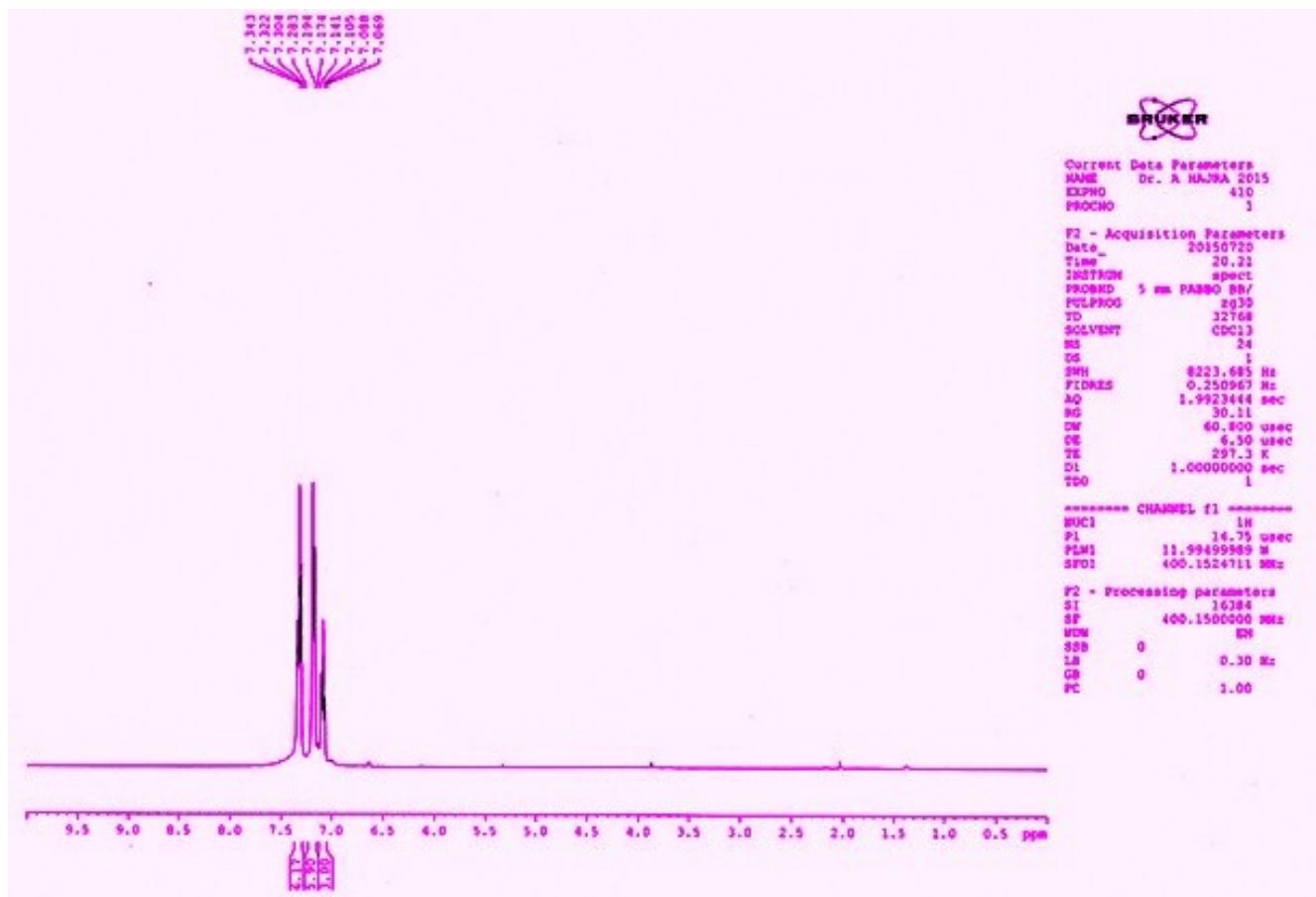
**Pyrazine N-oxide (Table 1 entry 6)**



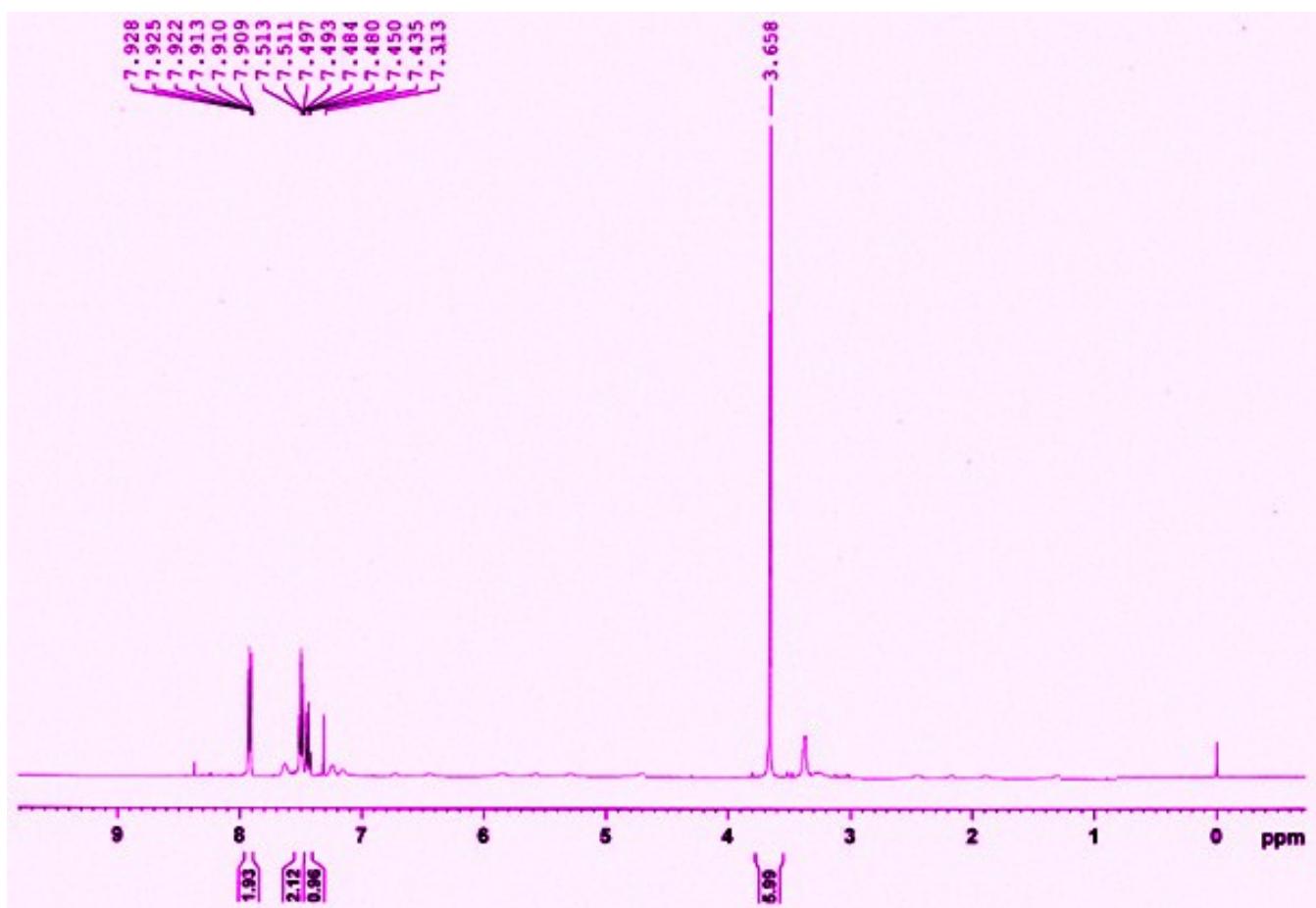
**Phenazine N-oxide** (Table 1 entry 7):



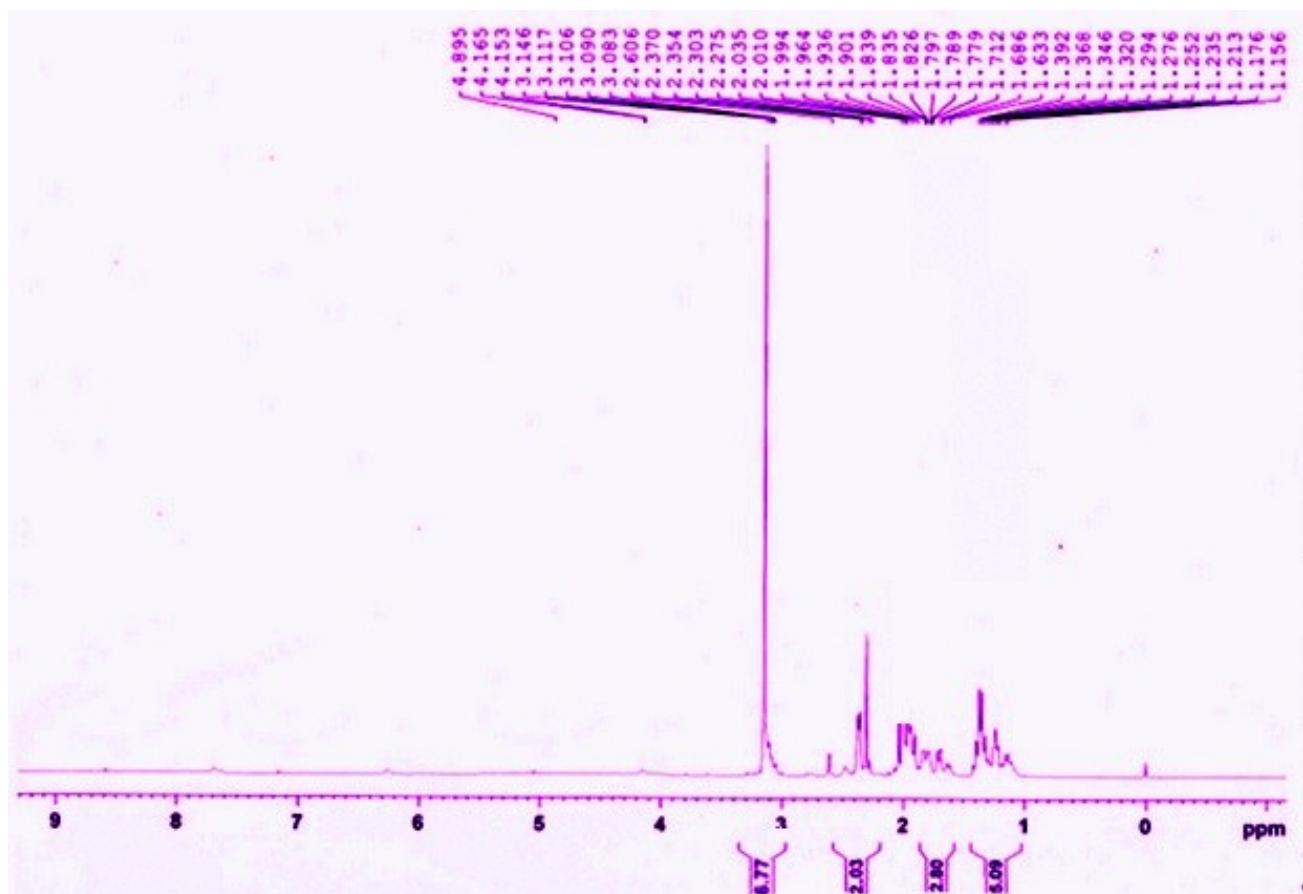
**Triphenyl amine N-oxide (Table 1 entry 8):**



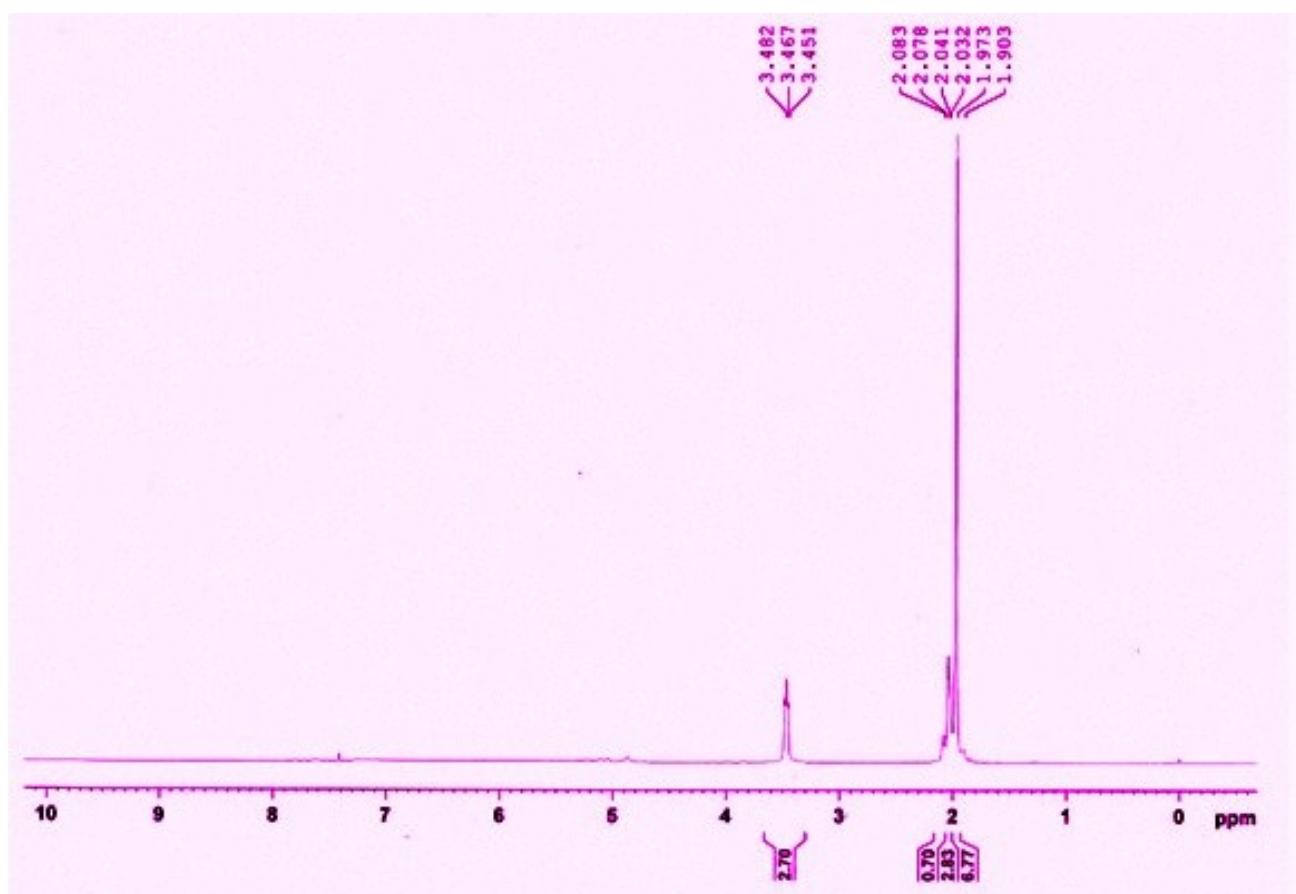
**N,N-dimethyl aniline N-oxide (Table 1 entry 9)**



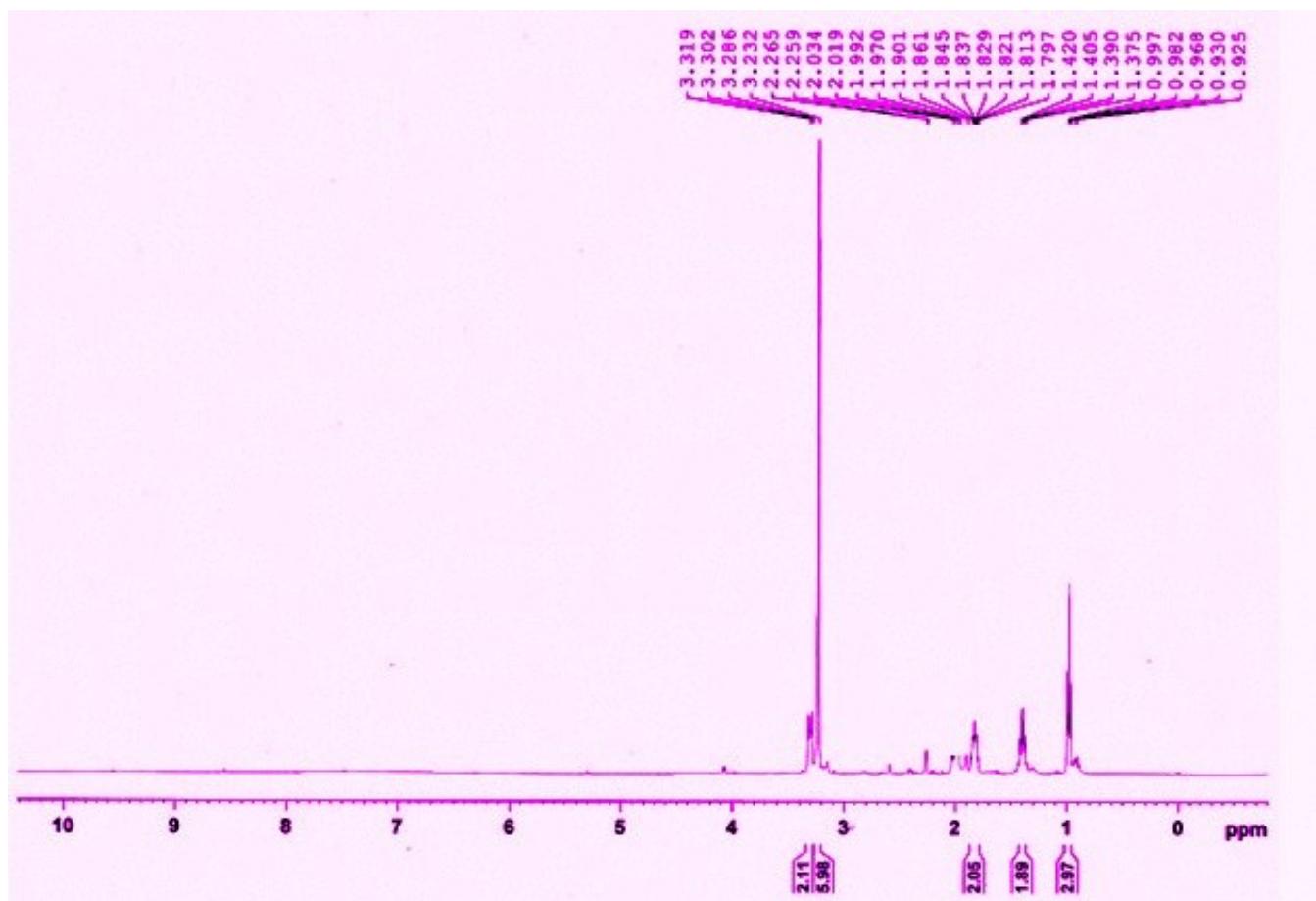
**N,N- dimethyl cyclohexyl amine N-oxide** (Table 1 entry 10):



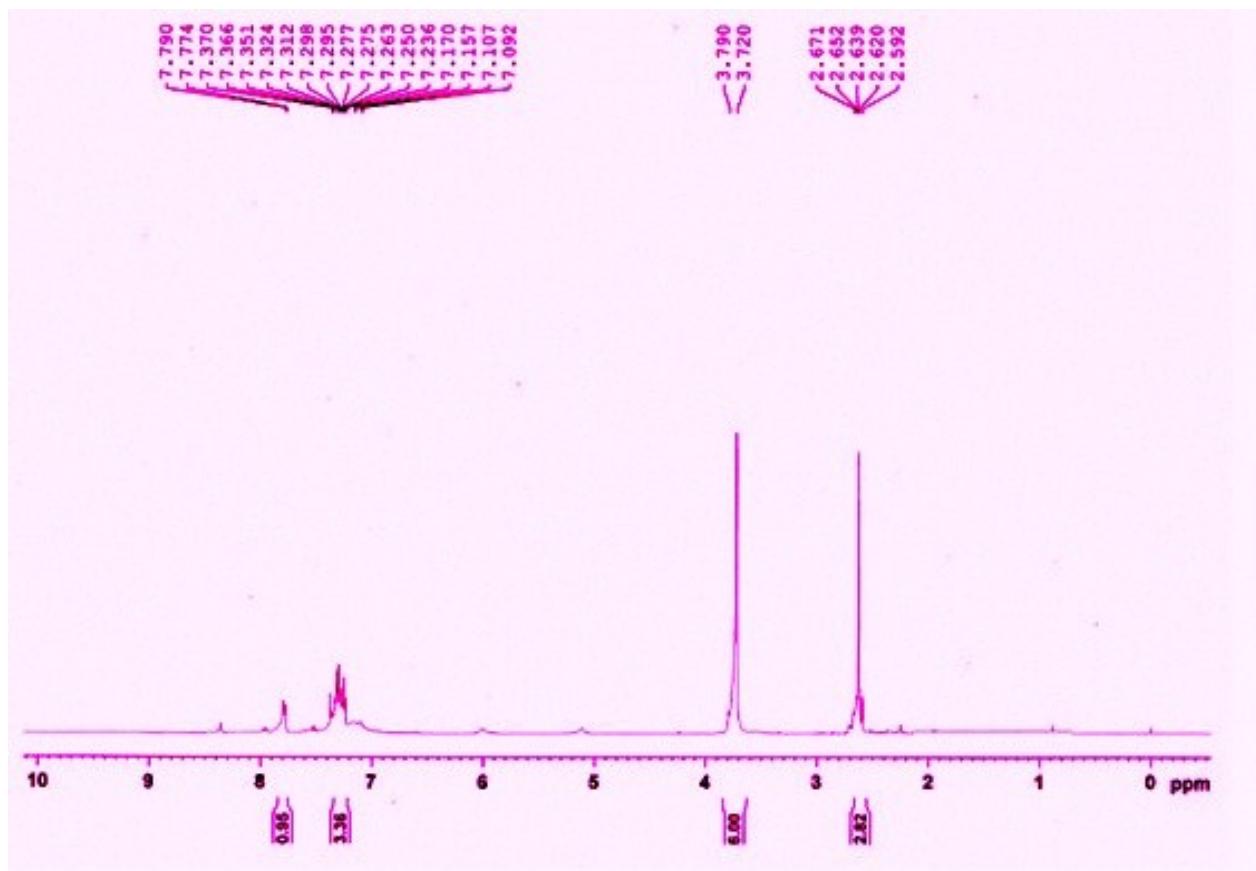
**Quinclidine N-oxide** (Table 1 entry 11):



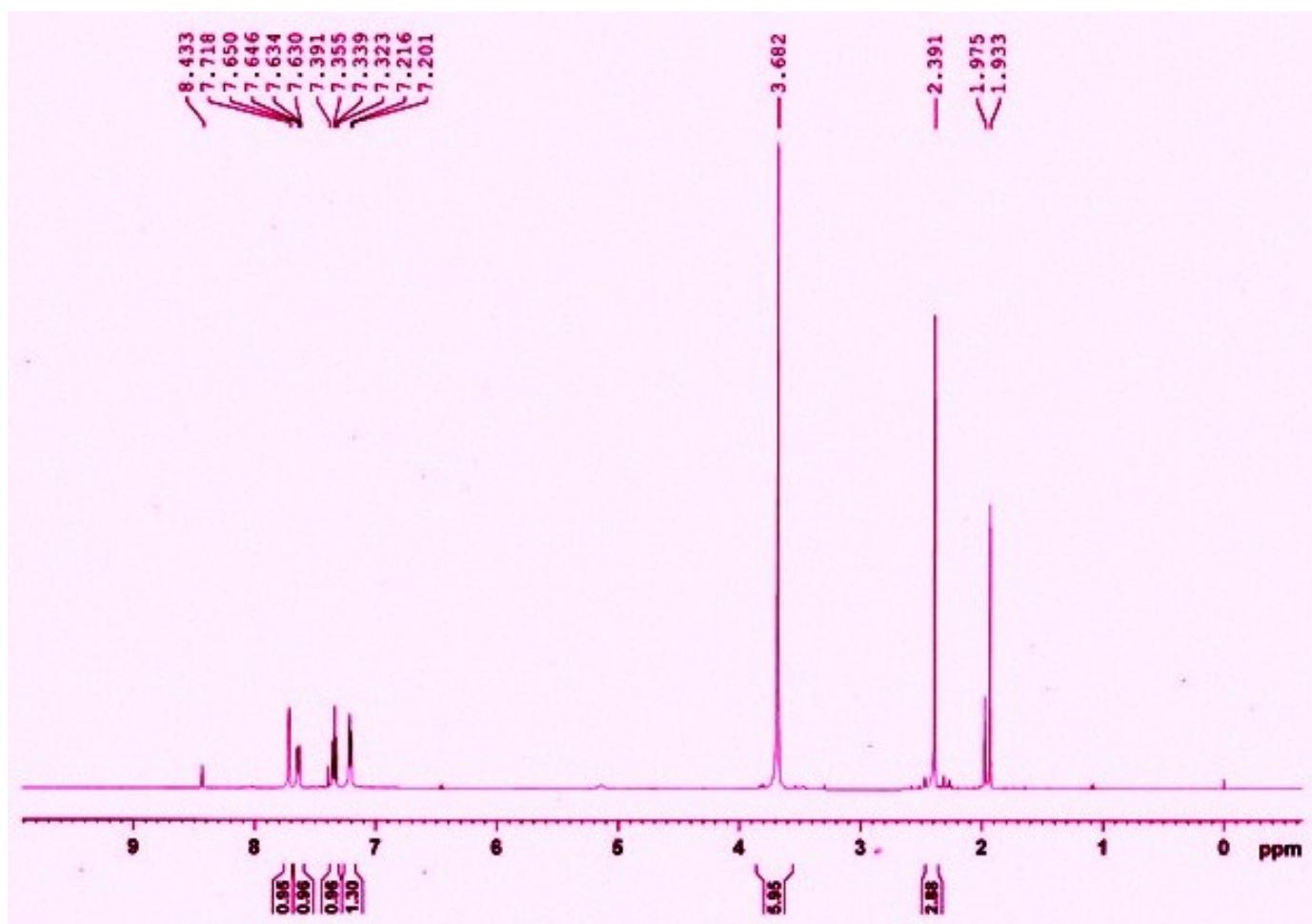
**N,N- dimethyl butyl amine N-oxide (Table 1 entry 12):**



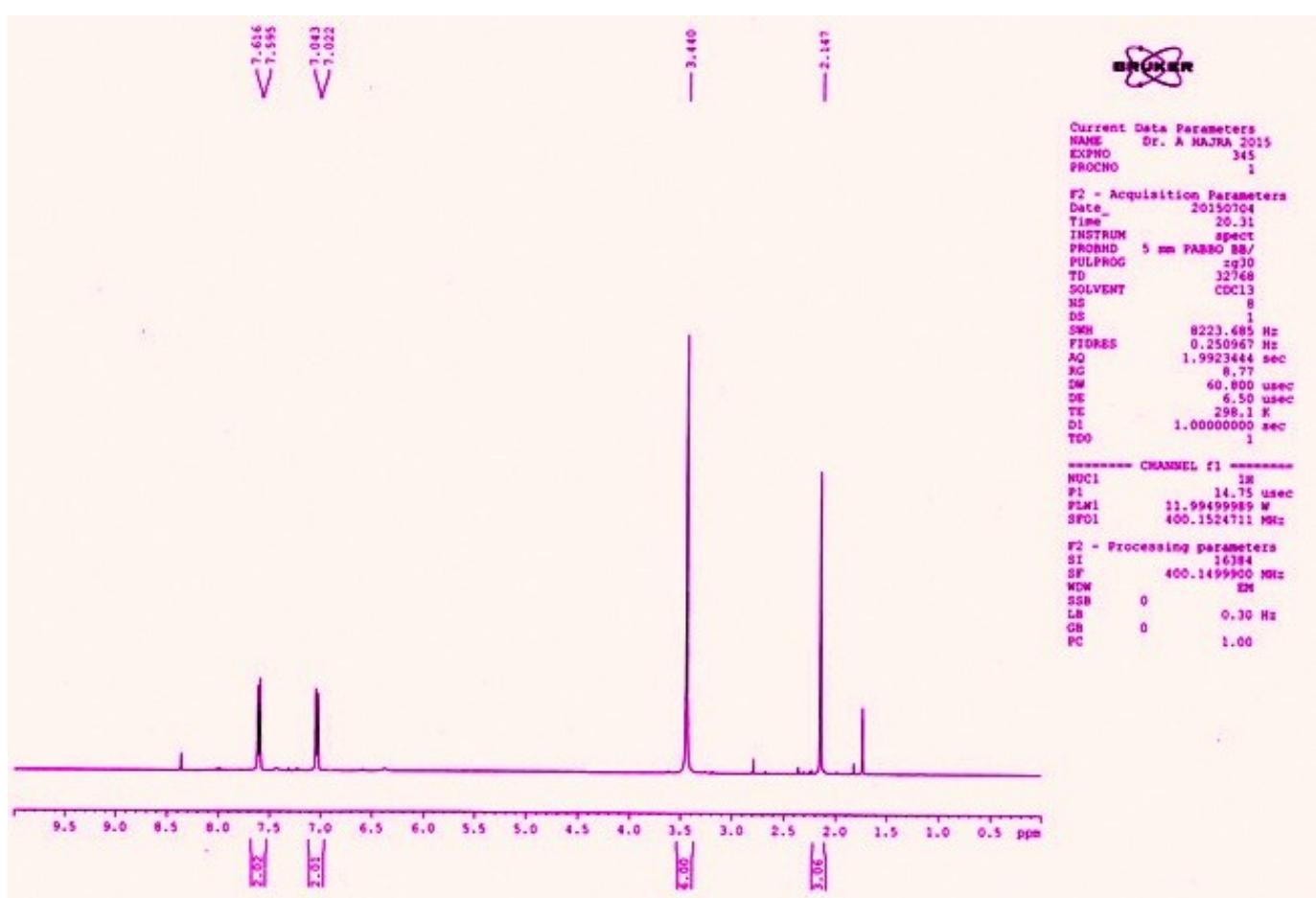
**N,N- dimethyl o-toluidine N-oxide (Table 1 entry 13)**



**N,N- dimethyl m-toluidine N-oxide** (Table 1 entry 14):

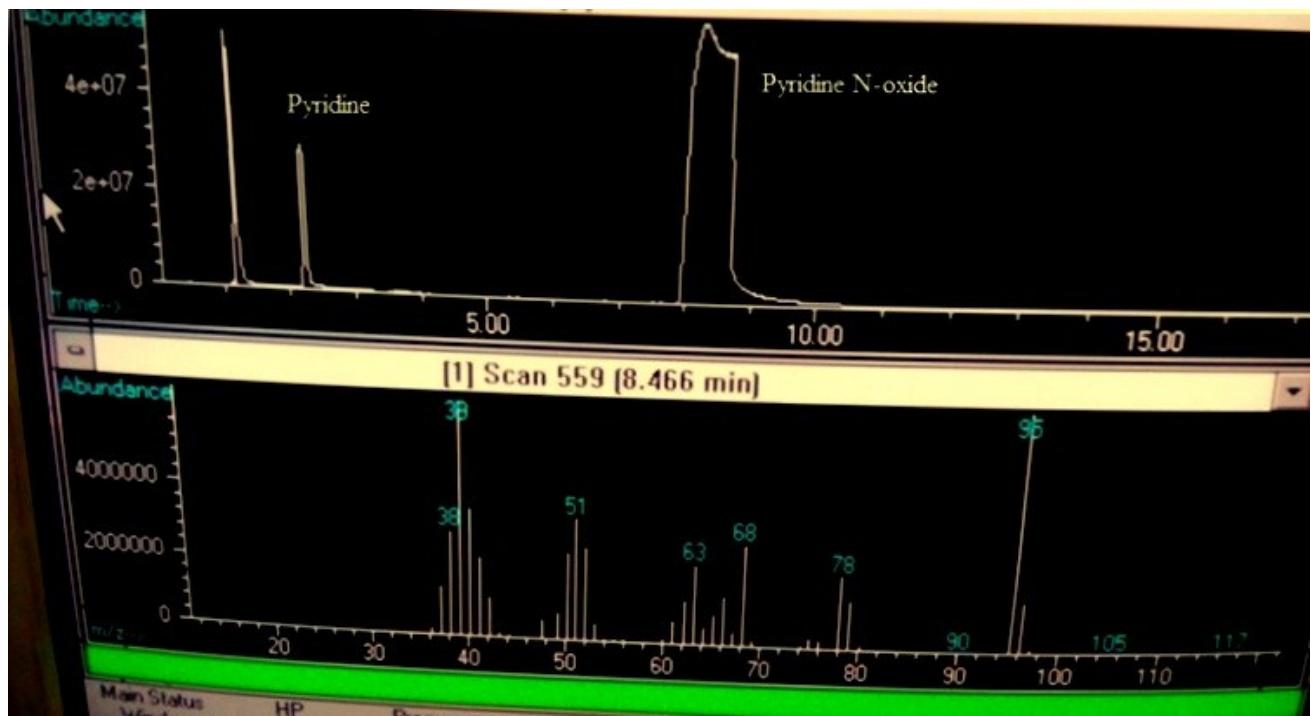


**N,N- dimethyl p-toluidine N-oxide (Table 1 entry 15):**

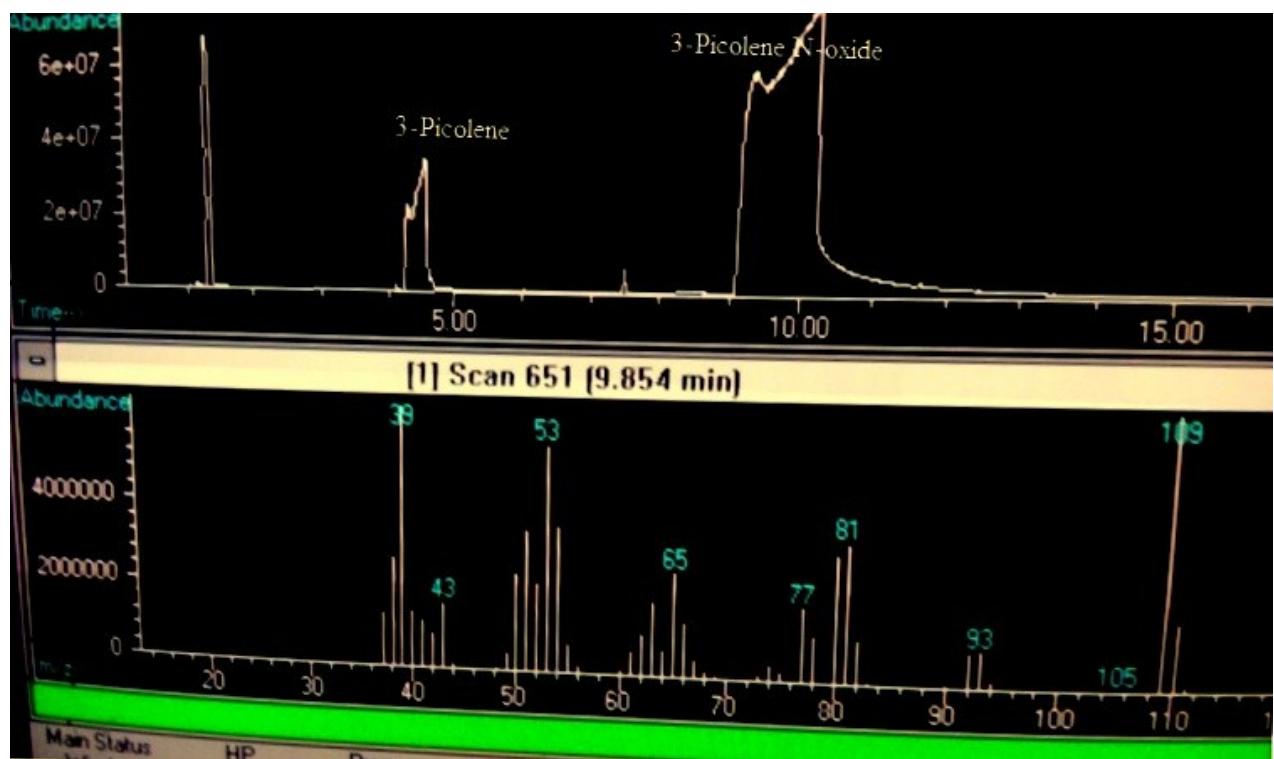


GC-MS :

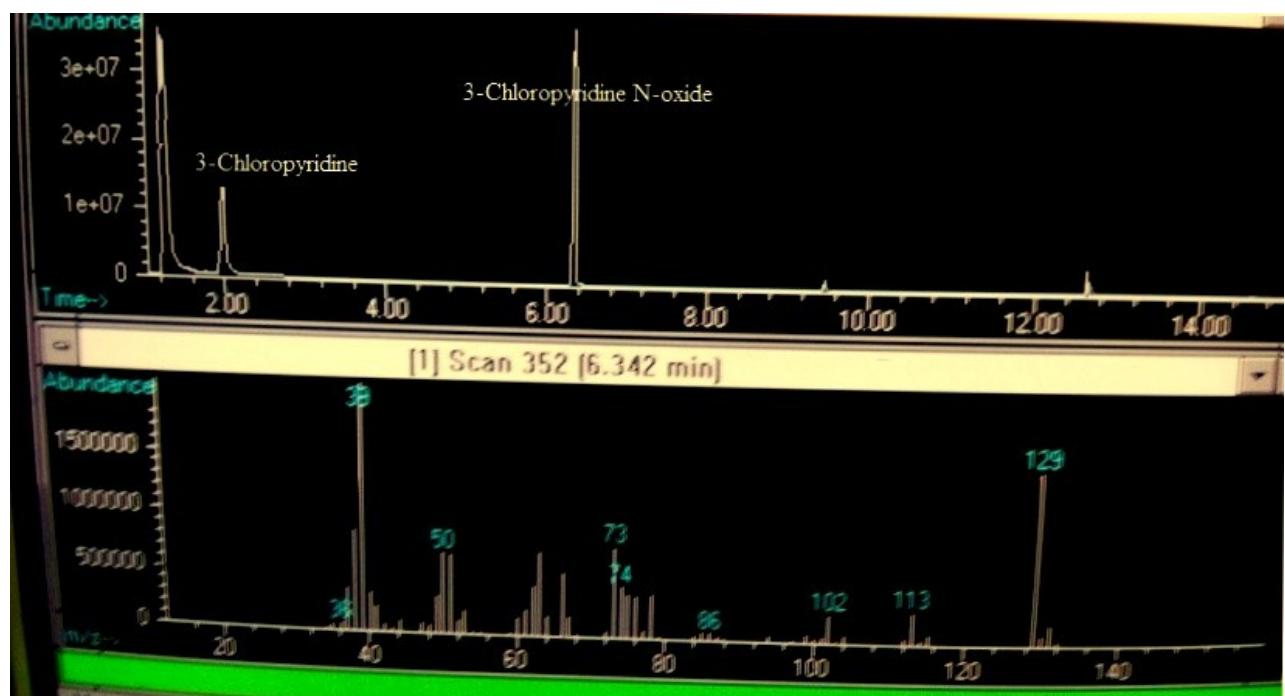
Pyridine N-oxide (Table 1 entry 1)



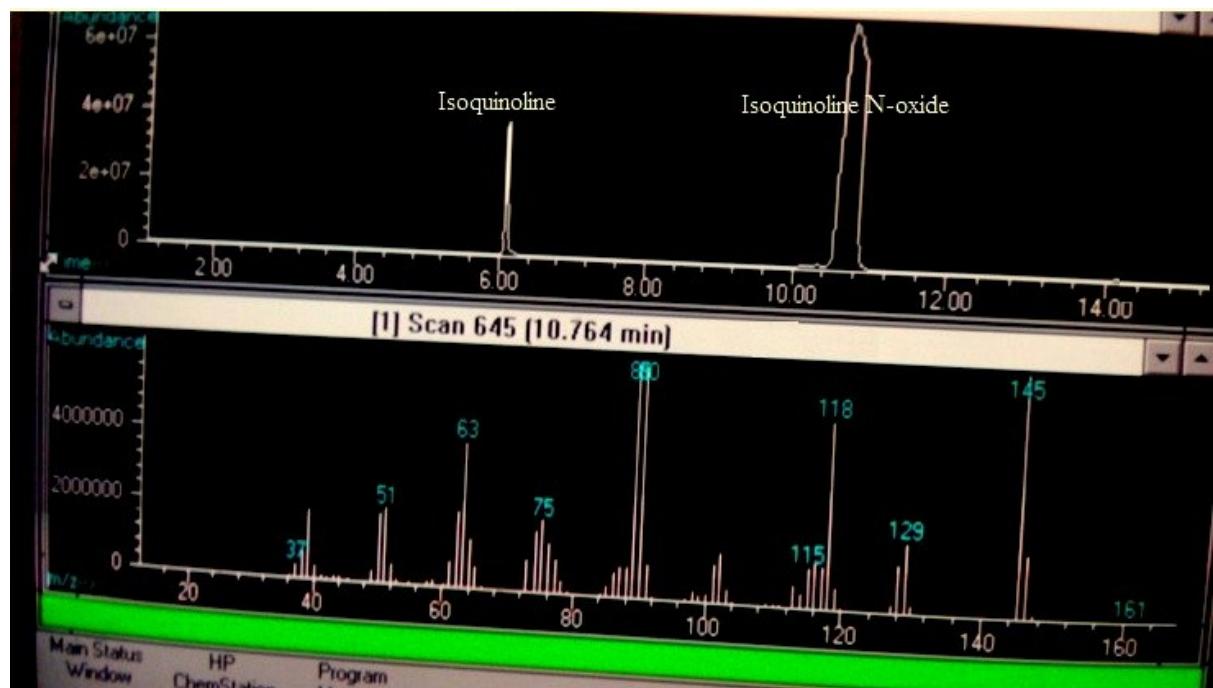
**3-Picoline N-oxide (Table 1 entry 2):**



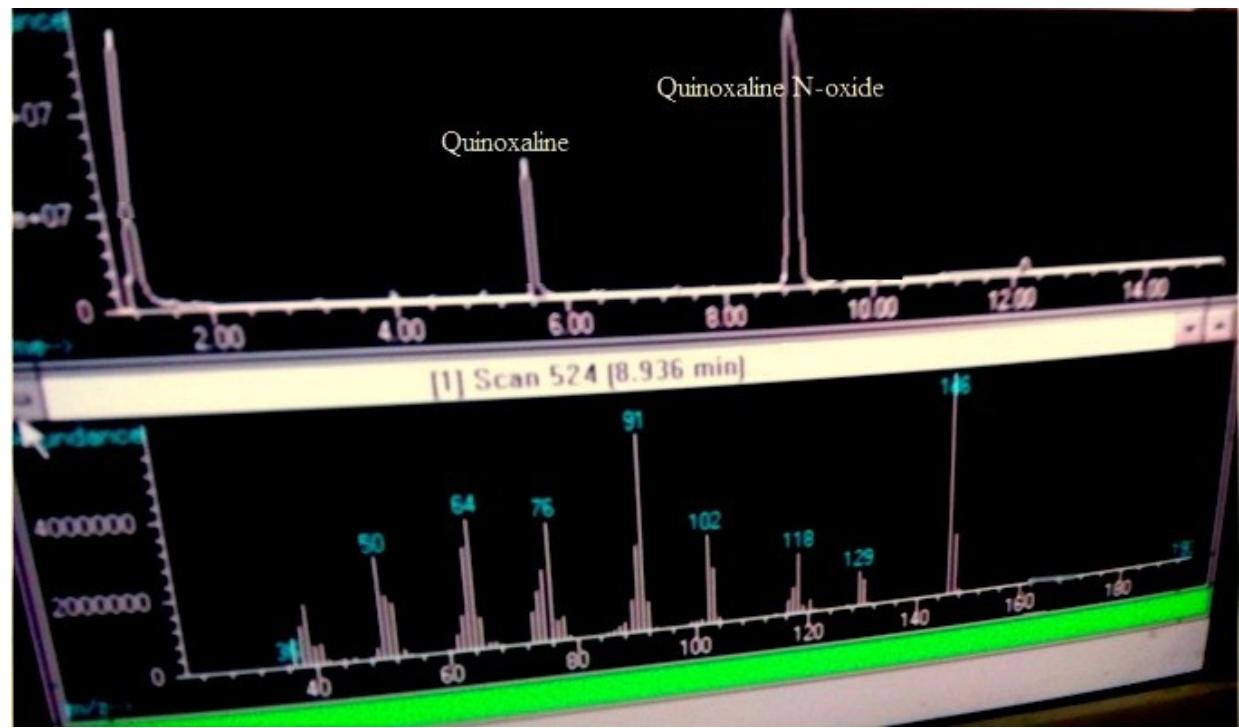
**3-Chloro Pyridine N-oxide (Table 1 entry 3)**



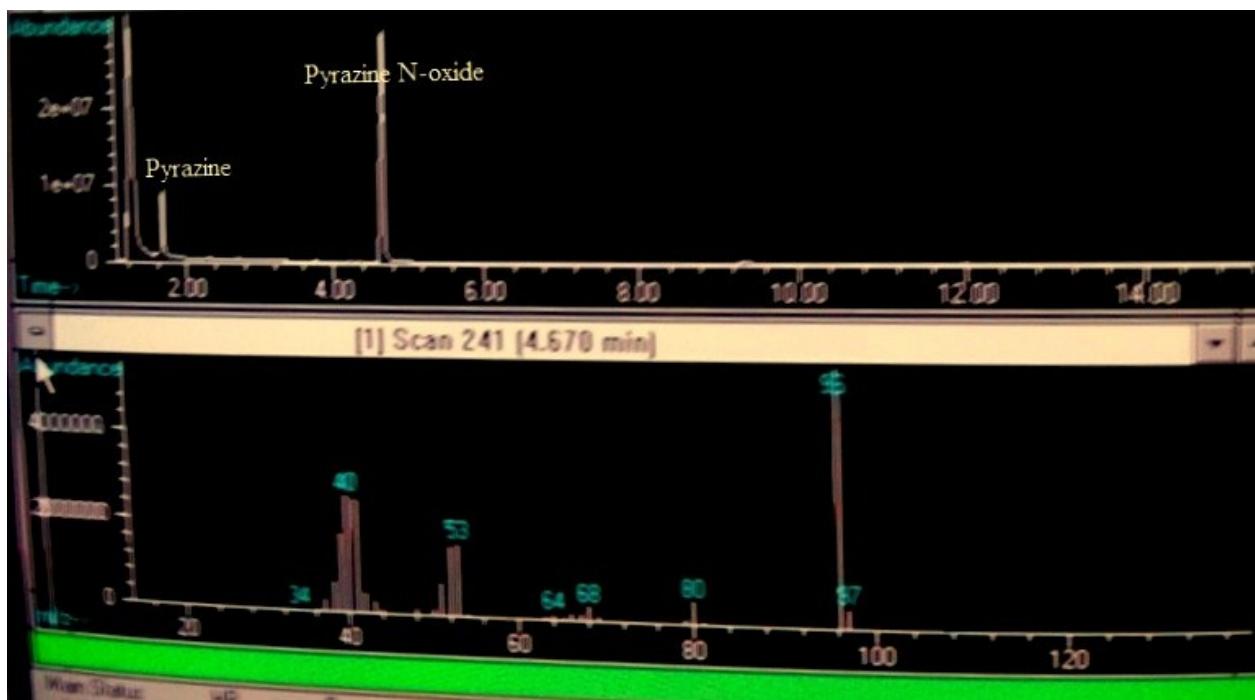
**Isoquinoline N-oxide (Table 1 entry 4)**



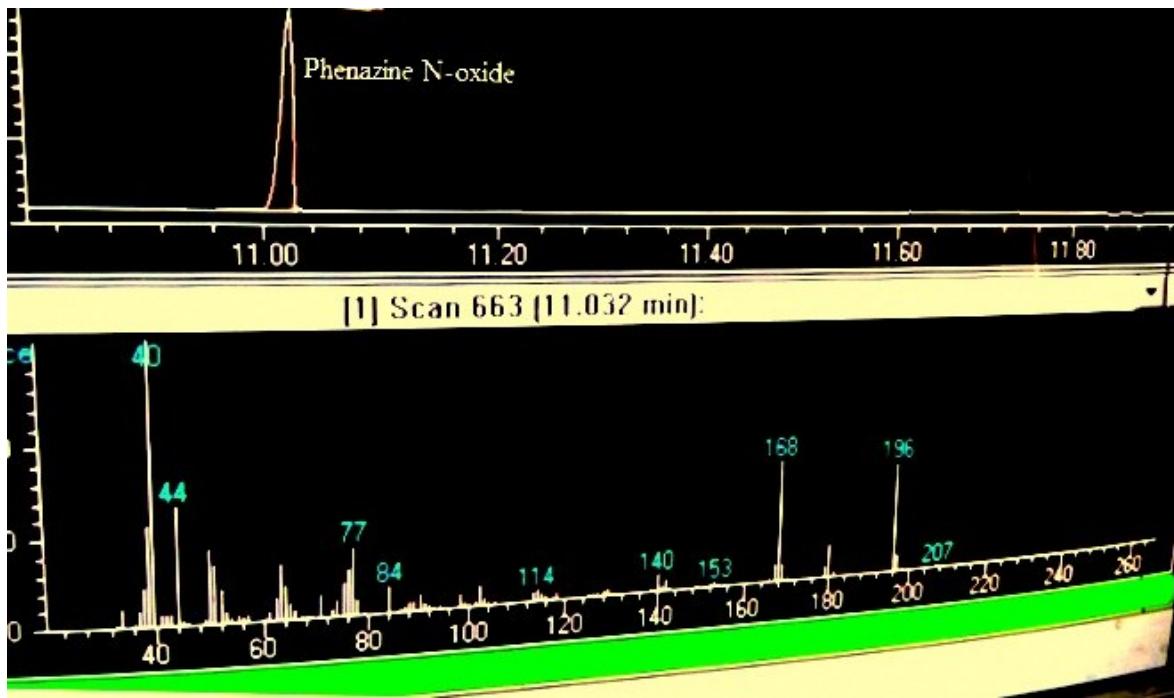
**Quinoxaline N-oxide (Table 1 entry 5)**



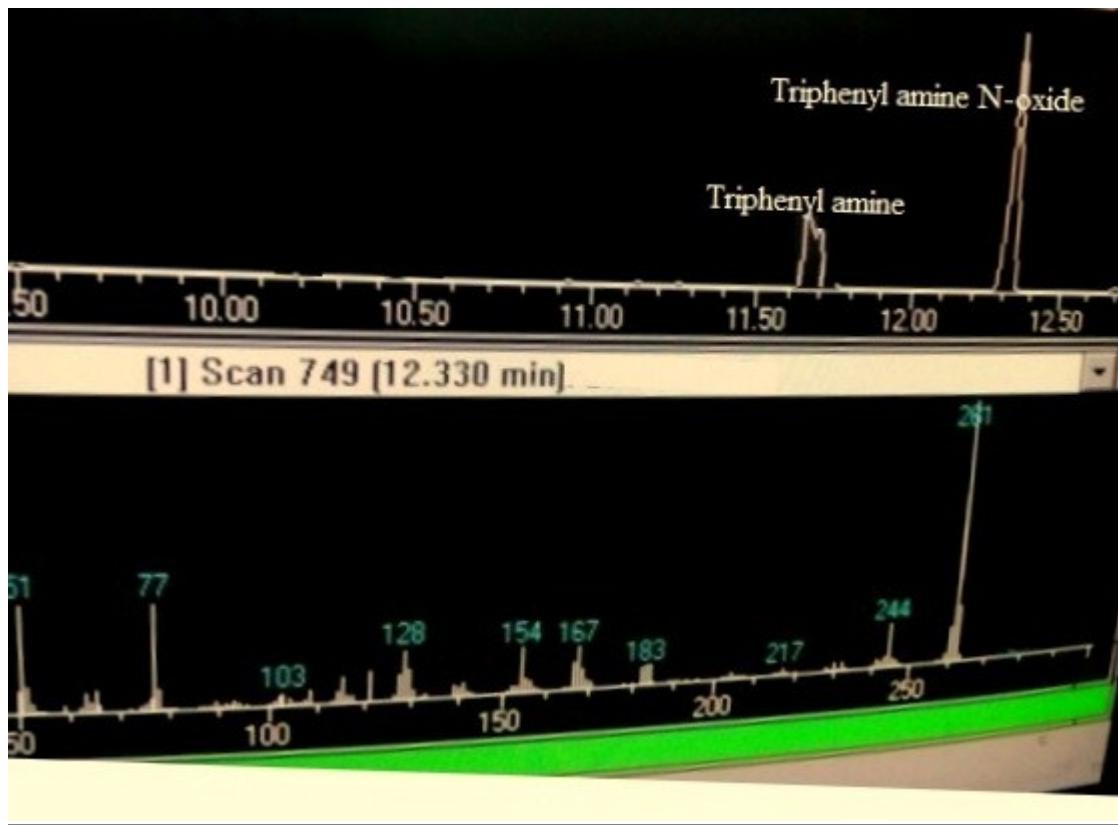
**Pyrazine N-oxide (Table 1 entry 6)**



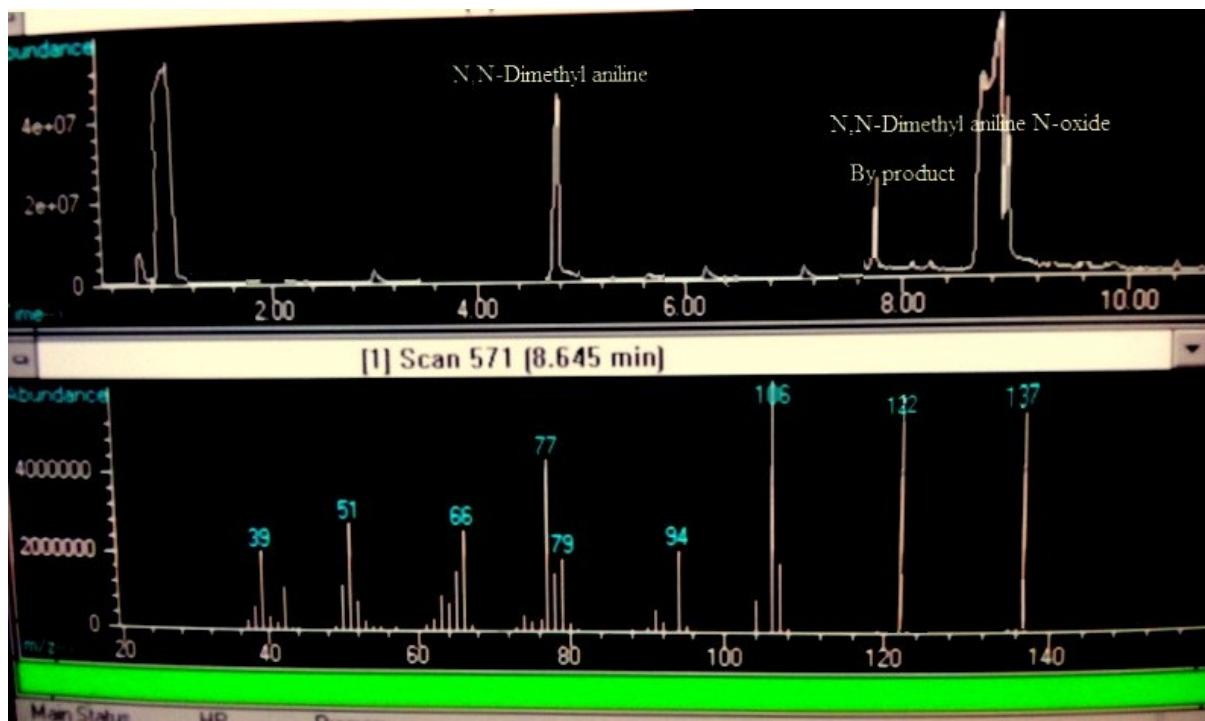
**Phenazine N-oxide (Table 1 entry 7):**



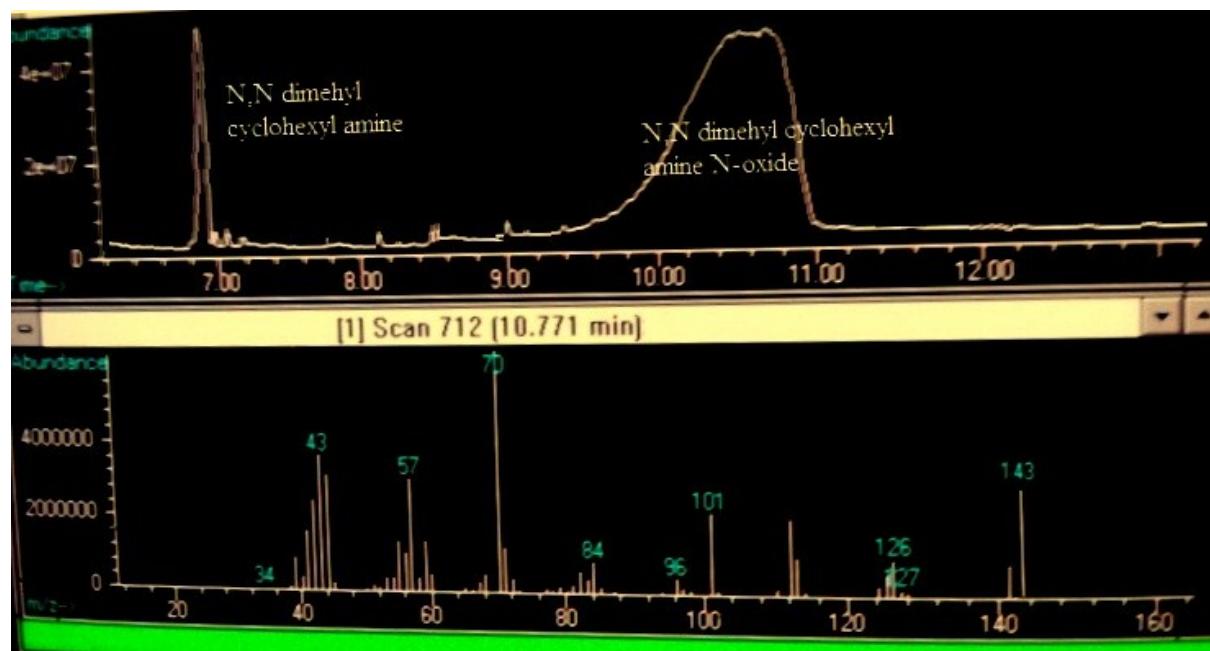
**Triphenyl amine N-oxide** (Table 1 entry 8):



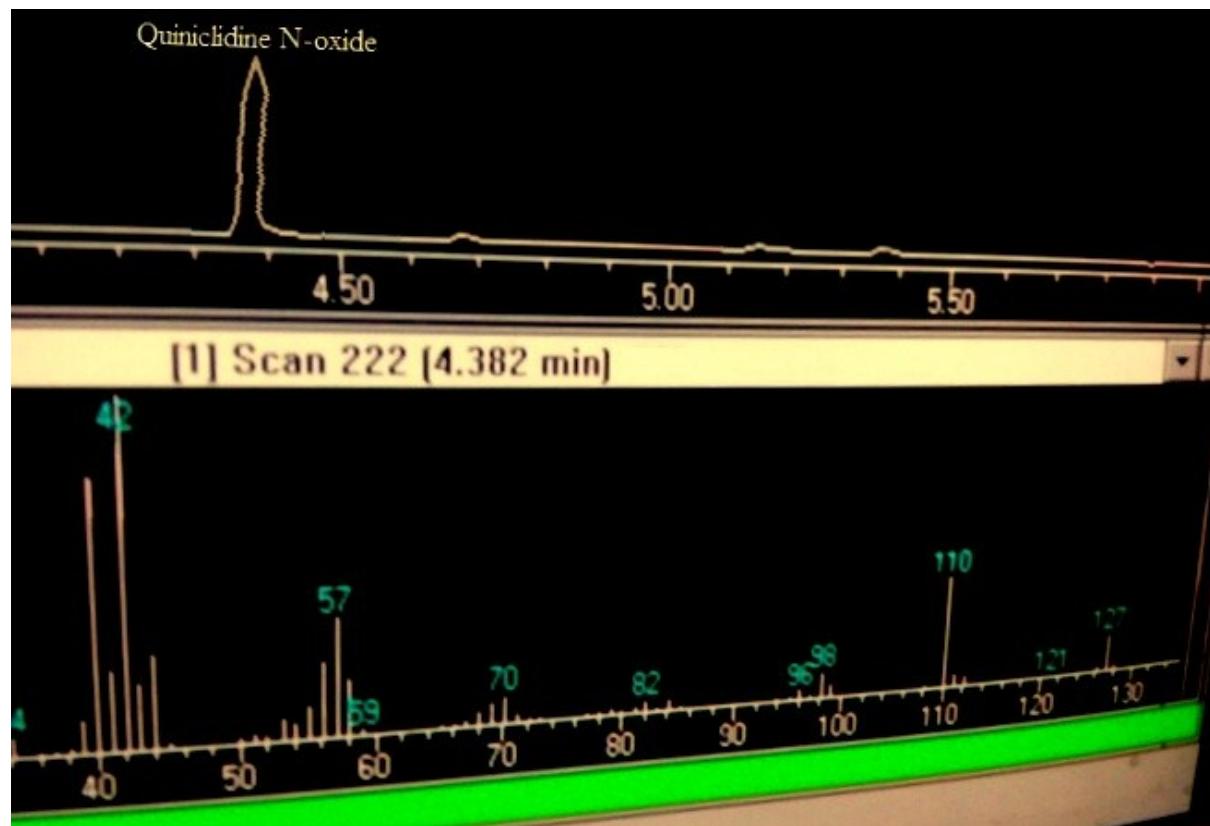
**N,N- dimethyl aniline N-oxide** (Table 1 entry 9)



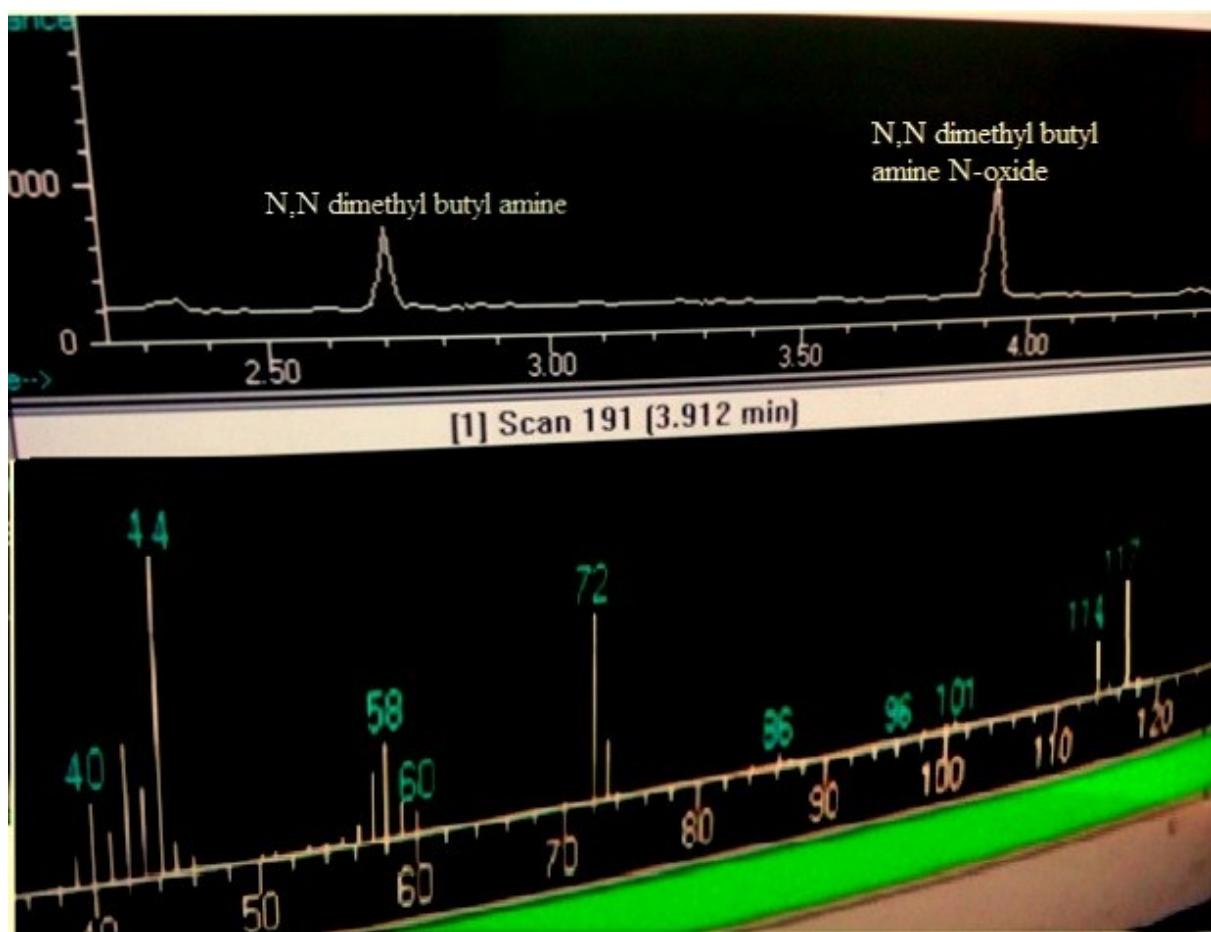
**N,N- dimethyl cyclohexyl amine N-oxide** (Table 1 entry 10):



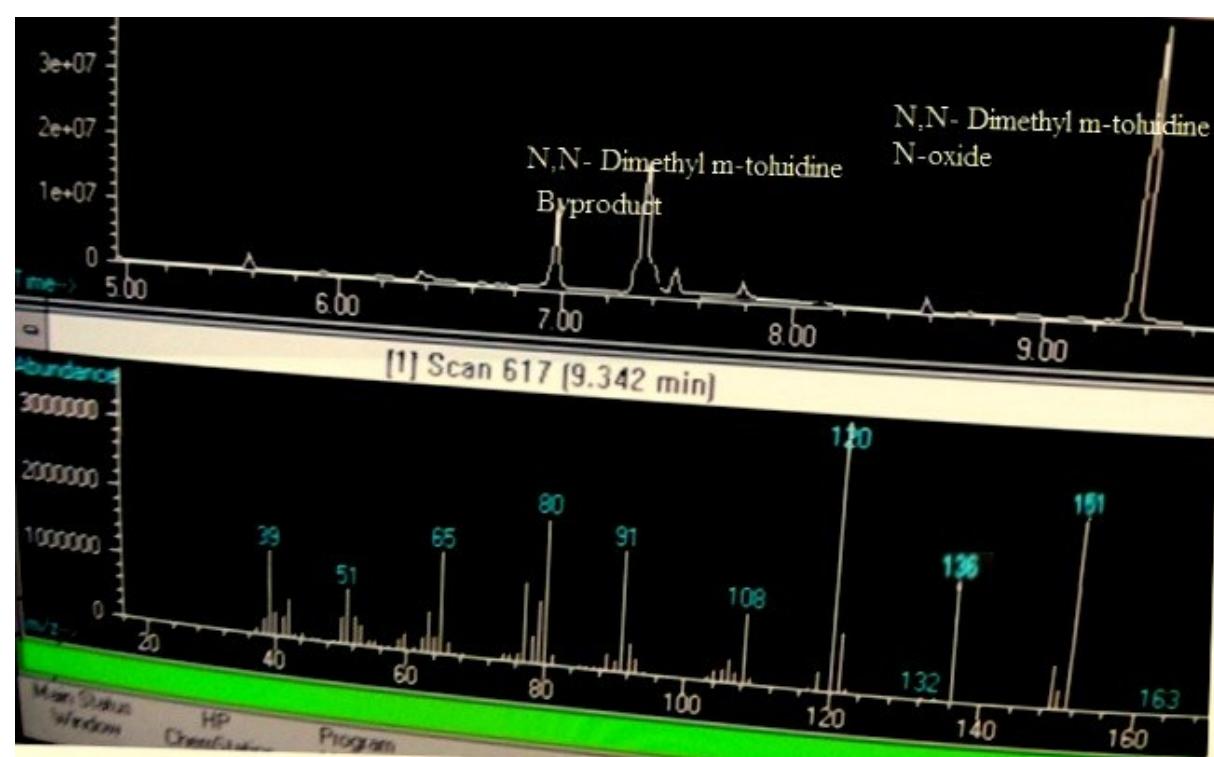
**Quiniclidine N-oxide** (Table 1 entry 11):



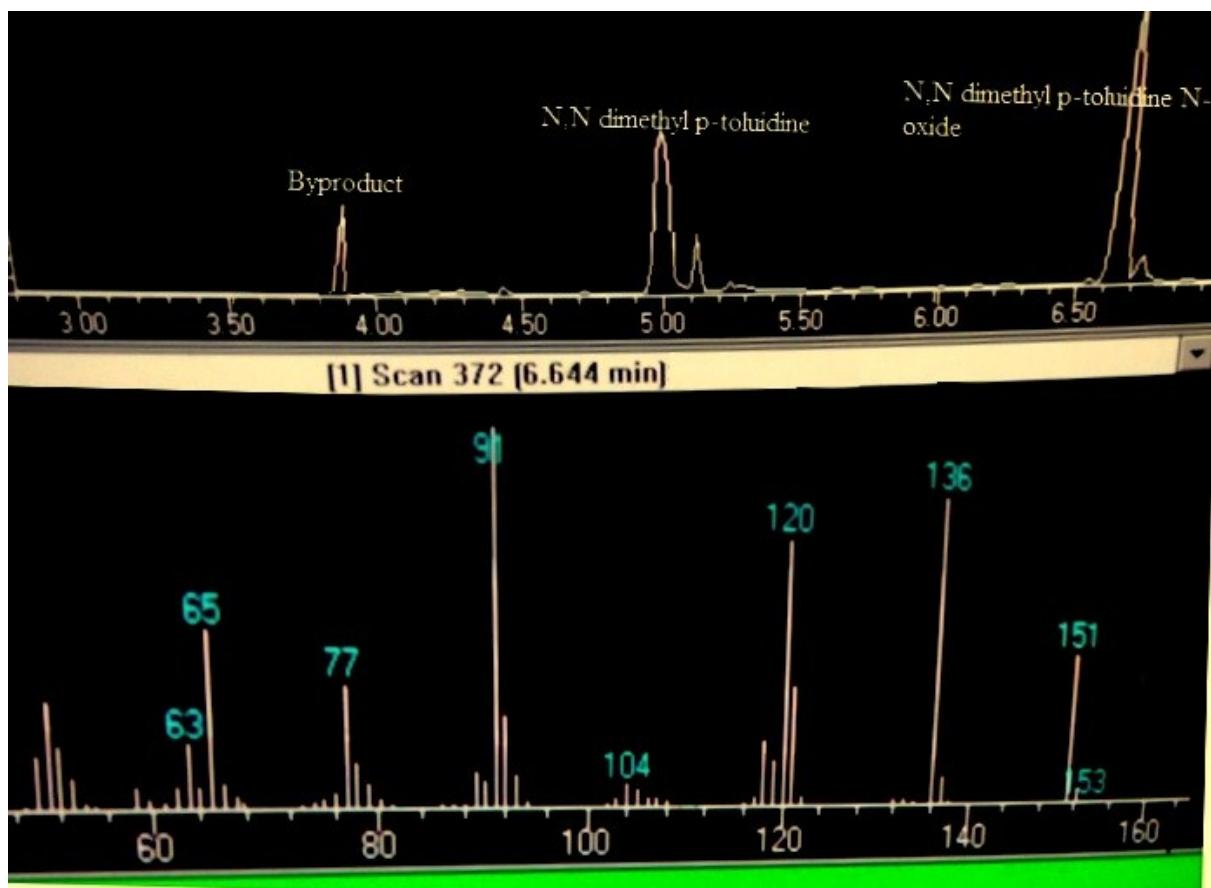
**N,N- dimethyl butyl amine N-oxide** (Table 1 entry 12):



**N,N- dimethyl m-toluidine N-oxide** (Table 1 entry 14):

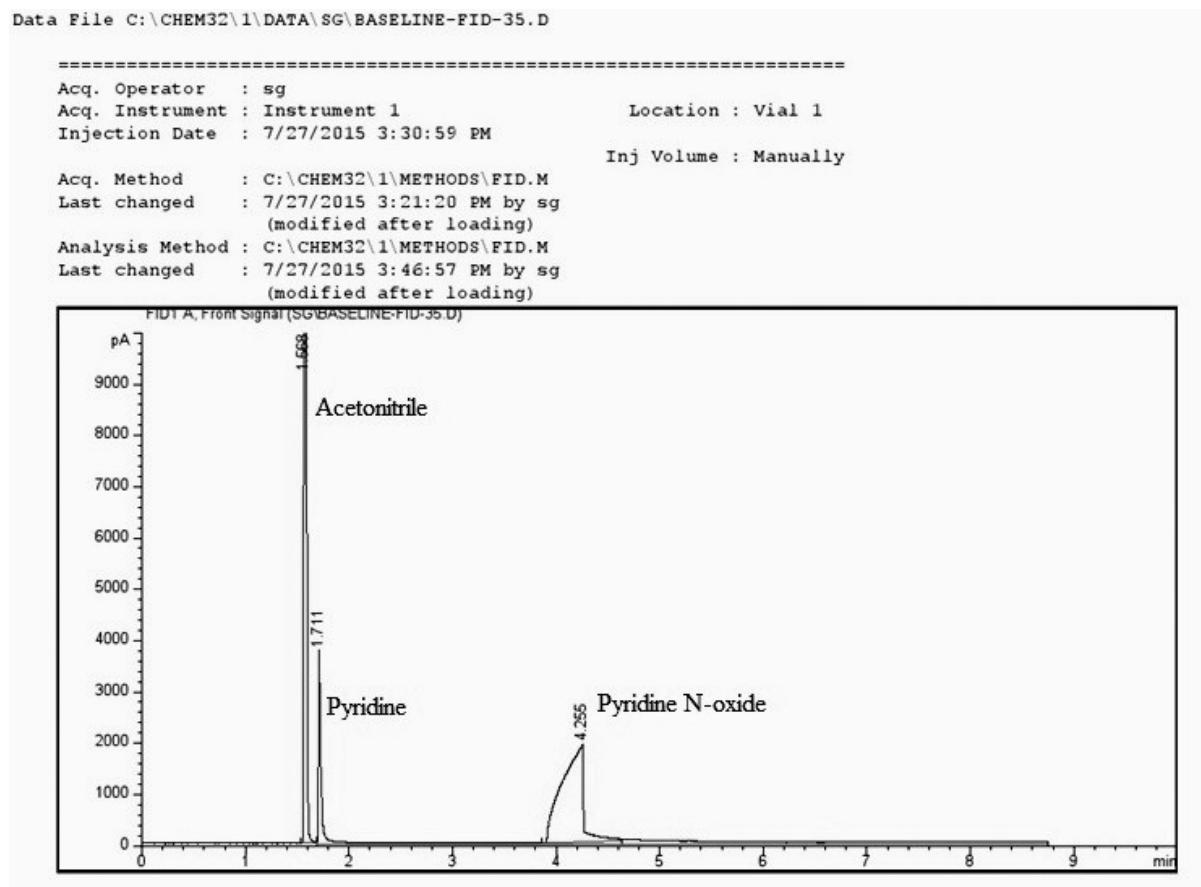


**N,N- dimethyl p-toluidine N-oxide** (Table 1 entry 15):



GC charts:

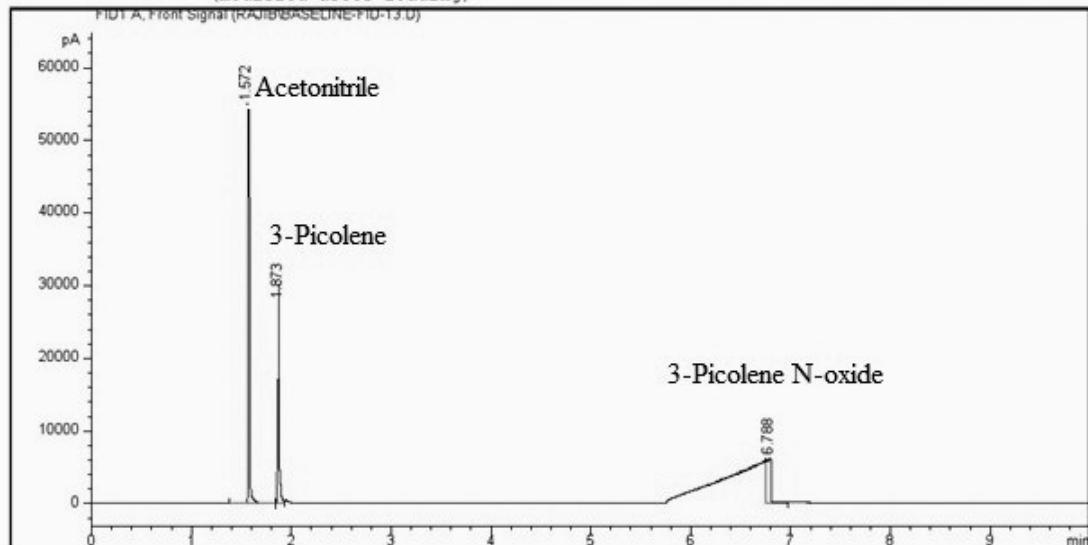
**Pyridine N-oxide (Table 1 entry 1):**



### 3-Picoline N-oxide (Table 1 entry 2)

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-13.D

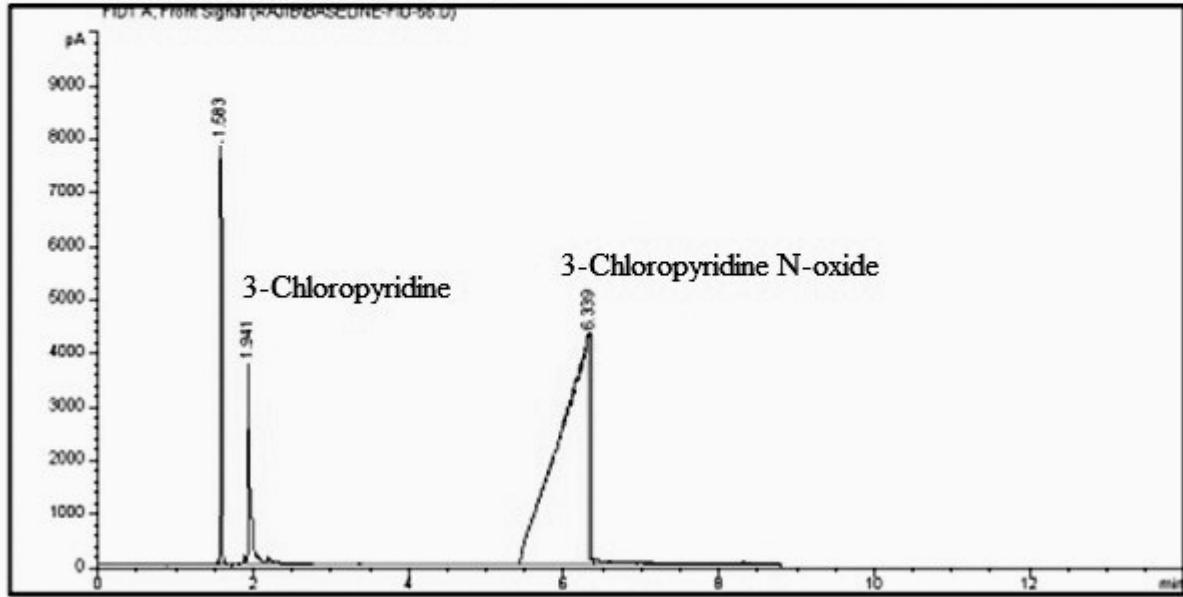
```
=====
Acq. Operator   : RAJIB
Acq. Instrument : Instrument 1
Injection Date   : 7/26/2015 5:25:59 PM
                           Location : Vial 1
                           Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 5:24:04 PM by RAJIB
                           (modified after loading)
Analysis Method  : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 5:38:30 PM by RAJIB
                           (modified after loading)
```



**3-Chloro Pyridine N-oxide (Table 1 entry 3):**

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-56.D

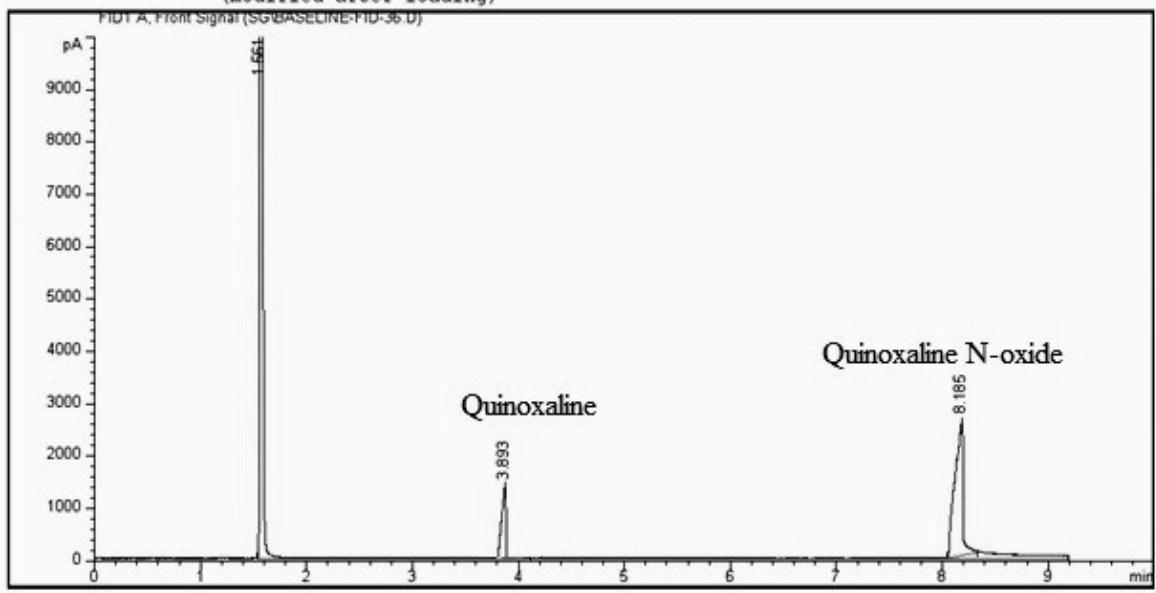
-----  
Acq. Operator : sg  
Acq. Instrument : Instrument 1 Location : Vial 1  
Injection Date : 7/30/2015 8:18:22 PM  
Inj Volume : Manually  
Acq. Method : C:\CHEM32\1\METHODS\FID.M  
Last changed : 7/30/2015 8:16:14 PM by sg  
(modified after loading)  
Analysis Method : C:\CHEM32\1\METHODS\FID.M  
Last changed : 7/30/2015 8:30:09 PM by sg  
(modified after loading)



**Quinoxaline N-oxide (Table 1 entry 5):**

Data File C:\CHEM32\1\DATA\SG\BASELINE-FID-36.D

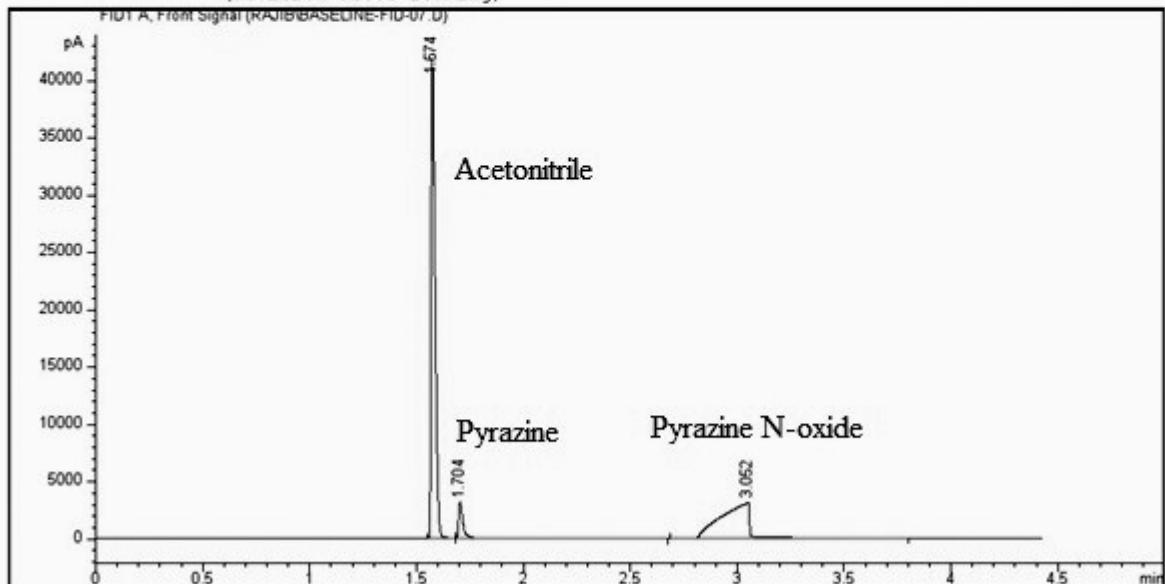
```
=====
Acq. Operator   : sg
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/27/2015 3:50:46 PM      Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/27/2015 3:46:57 PM by sg
                      (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/27/2015 4:04:38 PM by sg
                      (modified after loading)
```



### Pyrazine N-oxide (Table 1 entry 6):

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-07.D

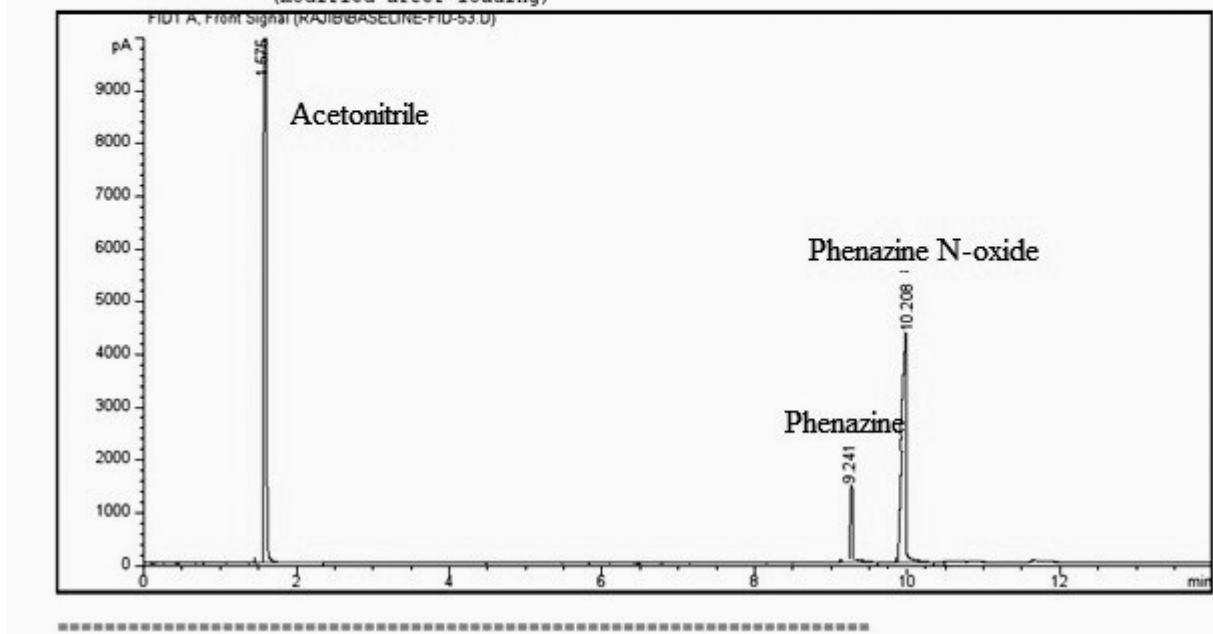
```
=====
Acq. Operator   : RAJIB
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/26/2015 4:26:33 PM
                                                Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 4:24:59 PM by RAJIB
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 4:31:02 PM by RAJIB
                  (modified after loading)
```



### Phenazine N-oxide (Table 1 entry 7)

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-53.D

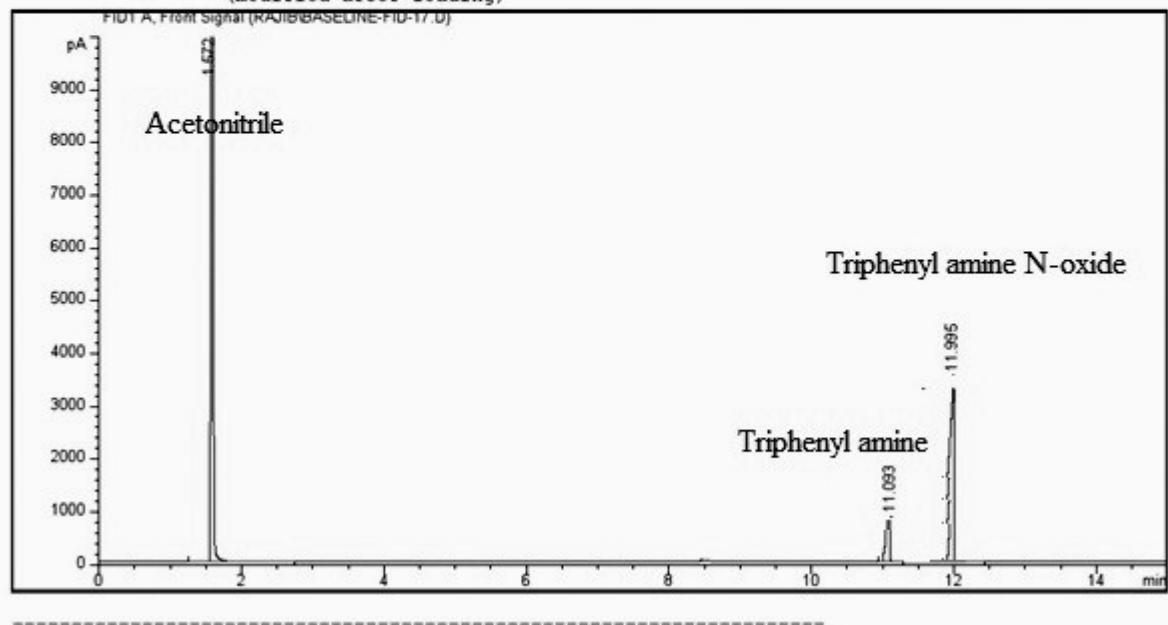
```
=====
Acq. Operator   : sg
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/30/2015 7:41:11 PM
                                                Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 7:39:59 PM by sg
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 7:58:38 PM by sg
                  (modified after loading)
```



Triphenyl amine N-oxide (Table 1 entry 8):

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-17.D

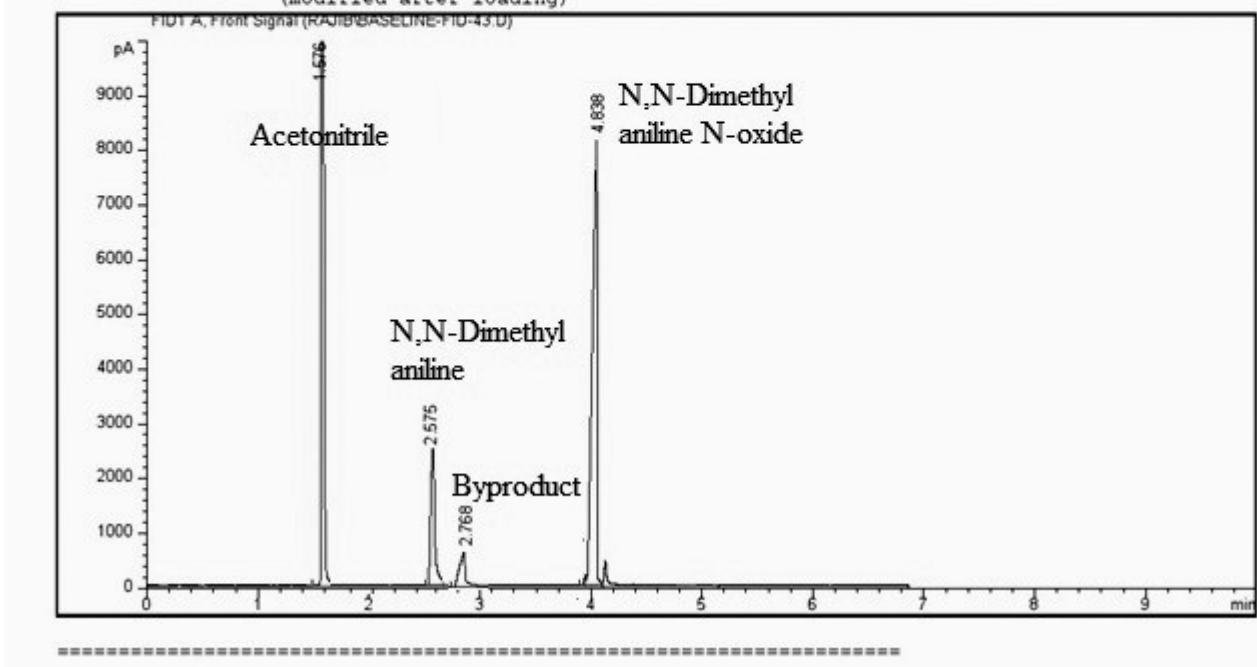
```
=====
Acq. Operator   : RAJIB
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/26/2015 6:39:28 PM
                                                Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 6:37:07 PM by RAJIB
                           (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/26/2015 6:56:11 PM by RAJIB
                           (modified after loading)
```



N, N- dimethyl aniline N-oxide (Table 1 entry 9)

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID=43.D

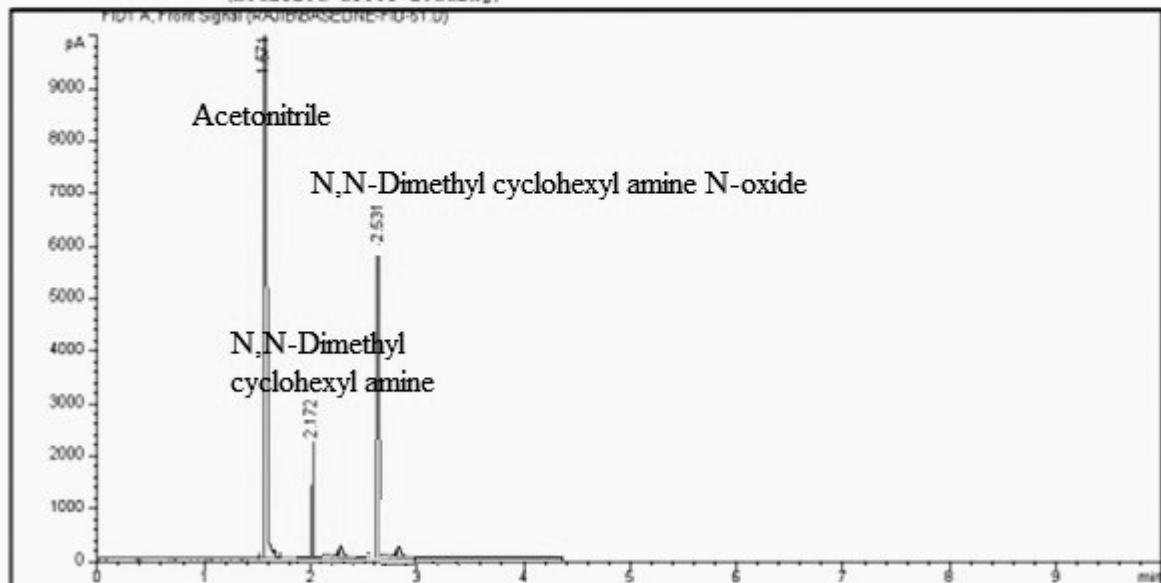
```
=====
Acq. Operator   : sg
Acq. Instrument : Instrument 1
Injection Date   : 7/30/2015 5:35:11 PM
                                         Location : Vial 1
                                         Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 5:13:07 PM by sg
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 5:44:04 PM by sg
                                         (modified after loading)
```



**N,N- dimethyl cyclohexyl amine N-oxide (Table 1 entry 10):**

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-51.D

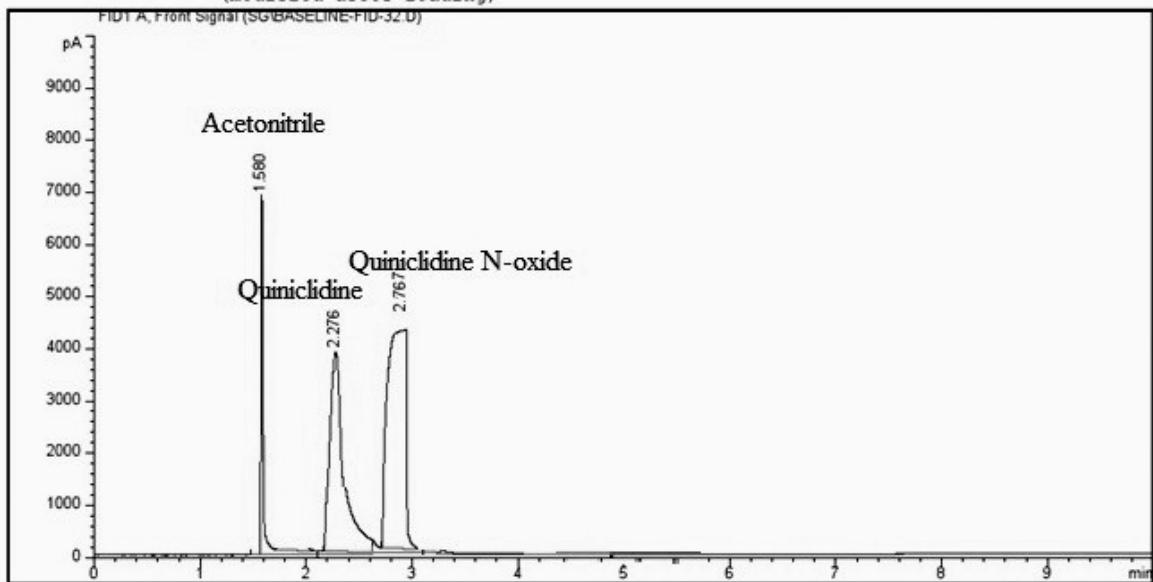
```
-----
Acq. Operator : sg                               Location : Vial 1
Acq. Instrument : Instrument 1
Injection Date : 7/30/2015 7:16:17 PM
Inj Volume : Manually
Acq. Method   : C:\CHEM32\1\METHODS\FID.M
Last changed  : 7/30/2015 7:14:32 PM by sg
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed  : 7/30/2015 7:23:21 PM by sg
(modified after loading)
```



### Quinclidine N-oxide (Table 1 entry 11):

Data File C:\CHEM32\1\DATA\SG\BASELINE-FID-32.D

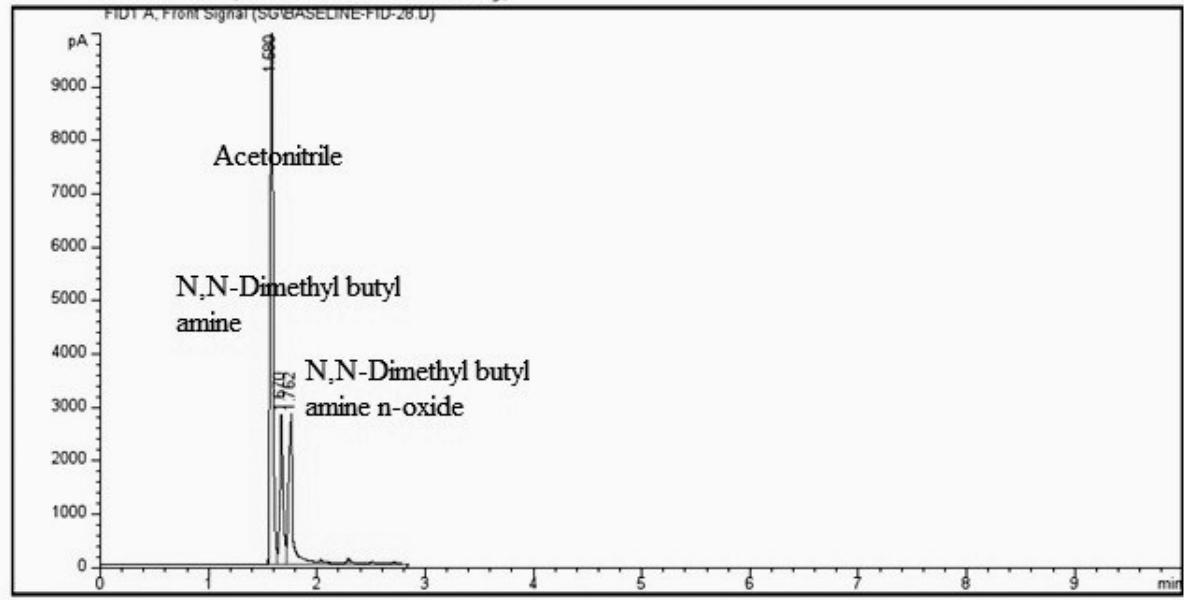
```
=====
Acq. Operator   : sg
Acq. Instrument : Instrument 1          Location : Vial 1
Injection Date  : 7/27/2015 2:49:23 PM      Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/27/2015 2:44:09 PM by sg
                      (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/27/2015 3:04:54 PM by sg
                      (modified after loading)
```



### N,N- dimethyl butyl amine N-oxide (Table 1 entry 12):

Data File C:\CHEM32\1\DATA\SG\BASELINE-FID-28.D

```
=====
Acq. Operator : sg
Acq. Instrument : Instrument 1
Injection Date : 7/26/2015 8:25:34 PM
Location : Vial 1
Inj Volume : Manually
Acq. Method : C:\CHEM32\1\METHODS\FID.M
Last changed : 7/26/2015 8:23:15 PM by sg
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed : 7/26/2015 8:29:14 PM by sg
(modified after loading)
```



N,N- dimethyl m-toluidine N-oxide (Table 1 entry 14):

Data File C:\CHEM32\1\DATA\RAJIB\BASELINE-FID-49.D

```
=====
Acq. Operator   : sg
Acq. Instrument : Instrument 1
Injection Date   : 7/30/2015 6:52:56 PM
                                         Location : Vial 1
                                         Inj Volume : Manually
Acq. Method     : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 6:49:59 PM by sg
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\FID.M
Last changed    : 7/30/2015 7:04:40 PM by sg
                  (modified after loading)
```

