Electronic Supplementary Information

For

Efficient iodine catalyzed three components domino reaction for the synthesis of 1-((phenylthio)(phenyl)methyl)pyrrolidin-2-one derivatives possessing anticancer activity

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GENERAL EXPERIMENT METHOD:

Chemicals were purchased from Aldrich and they were used without further purification. TLC -Thin layer chromatography (Merck, Silica gel 60 F254) was performed on pre-coated silica gel on alumina plates. IR spectra were recorded in the range 4000-400cm-1 on Thermo Nicolet Avatar 330 FTIR spectrometer in KBr pellets. 1H NMR and 13C NMR spectra were recorded using a Bruker AMX 400 FT. The X-ray crystallographic diffractions were determined using a Smart – CCD (Bruker, 2004).

PREPARATION OF SIMULATED GASTRIC JUICE

Simulated gastric juice was prepared using glucose (3.5 g/liter), NaCl (2.05 g/liter), KH_2PO_4 (0.60 g/liter), $CaCl_2$ (0.11 g/liter), and KCl (0.37 g/liter), adjusted to pH 2.0 using 1 M HCl, and autoclaved at 121°C for 15 min. Porcine bile (0.05 g/liter), lysozyme (0.1 g/liter), and pepsin (13.3 mg/liter) were added as stock solutions prior to analysis.

GENERAL PROCEDURE FOR REMOVAL OF FATTY ACIDS FROM SERUM ALBUMIN:

Albumin (7.0 g) was dissolved in 70 ml of Millipore water at 23". Darco (3.5 g) was mixed into the solution, and the pH was lowered to 3.0 by the addition of 0.2 N HCl. The solution was then placed in an ice bath and mixed magnetically for 1 hour. Charcoal was then removed by centrifugation at 20,200 for 20 min in a REMI cooling centrifuge at 2°C. The clarified solution was then brought to pH 7.0 by the addition of 0.2 N NaOH.

PURIFICATION OF BSA:

Bovine serum albumin which was free from fatty acid was used. BSA solutions (~40%) was prepared in 0.1 M KH_2PO_4 buffer, pH – 4.7. To it o.1 % Norit suspension prepared in the

same buffer was slowly introduced and kept it for stirring at low speed in a magnetic stirrer for an hour at room temperature. The solution was filtered through Whatman filter paper ≠ 1 and 0.2 Amicon filter under vacuum. After filtering successively, the BSA solution was adjusted to pH 7.4 with 5.0 M KOH. The BSA solution was lyophilized and kept for further use in cold.

EXPERIMENTAL PROCEDURE:

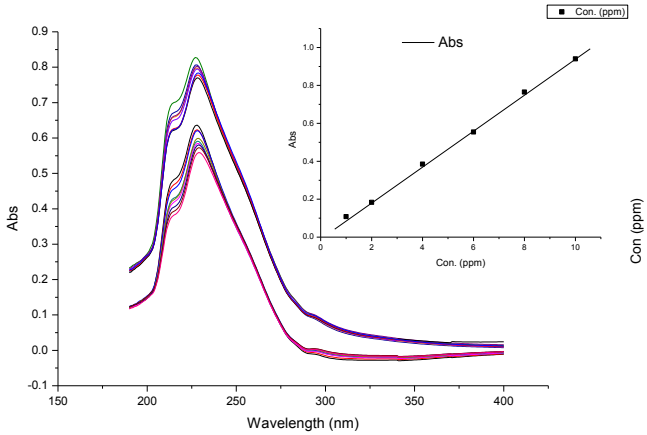
A dry 100ml Erlenmeyer flask was charged with γ-lactam (1.0 mmol); Aromatic aldehydes (1.0 mmol); Substituted thiophenol (1.0 mmol); Iodine (0.3 mmol) and DCM (15 ml). The reaction mixture was stirred at room temperature for 1-2 hrs. The reaction was monitored by TLC and after the completion of reaction iodine was liberated by treating it with aqueous sodium thiosulphate solution, extracted into DCM (2 X 20 ml) and the crude reaction mixture was purified by column chromatography on silica gel using ethylacetate/hexane as the eluents.

CELL LINE MAINTENANCE:

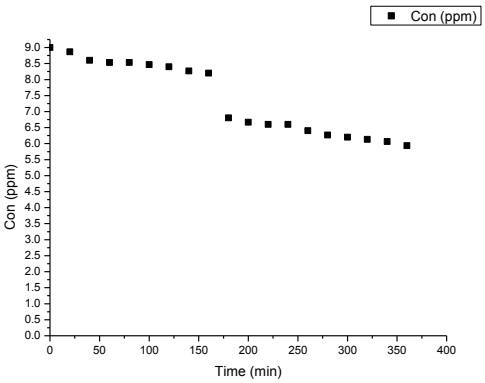
A panel of five cancer cells representing multiple cancers of clinical relevance were selected namely; ACHN (human renal cell, ATCC, CRL-1611), Panc-1 (human pancreatic, ATCC, CRL-1469) cultured on MEM with 2mM Lglutamine and 10% FBS, H460 (human non small cell lung carcinoma, ATCC, HTB- 177), Calu-1 (human lung carcinoma, ATCC, HTB-54) and MCF7 (human breast adenocarcinoma, ATCC HTB-22) cultured on RPMI, 2mM Lglutamine and 10% FBS, HCT-116 (human colon cancer, ATCC, CCL- 247) cultured on McCoy's 5a medium and 10% FBS and MCF10A (normal breast epithelium cells) cultured on MEM with 2mM L-glutamine and 10% FBS.

Stability of the compound using UV-Visible Spectra:

In SGF



Stability of 4b in SGF using UV spectra at specified time interval.



Concentration loss of 4b in SGF using UV spectra at specified time interval.

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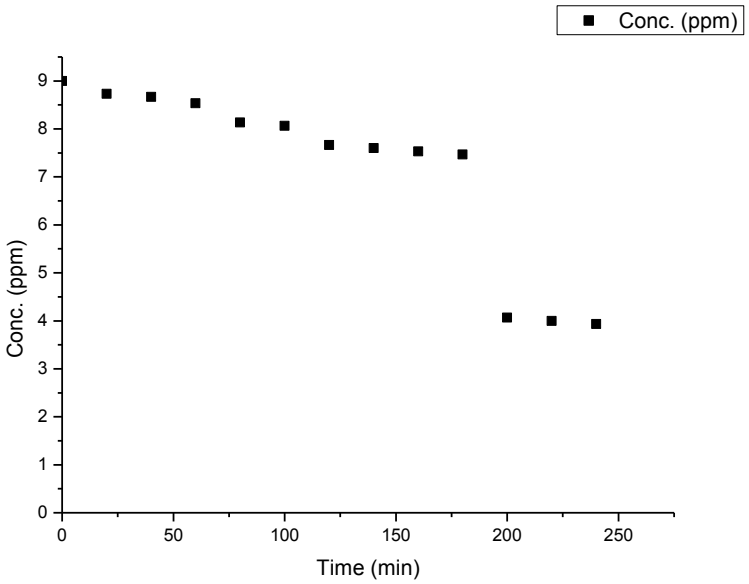

Concentration loss of 4b in BSA using UV-Visible spectra at specified time interval.

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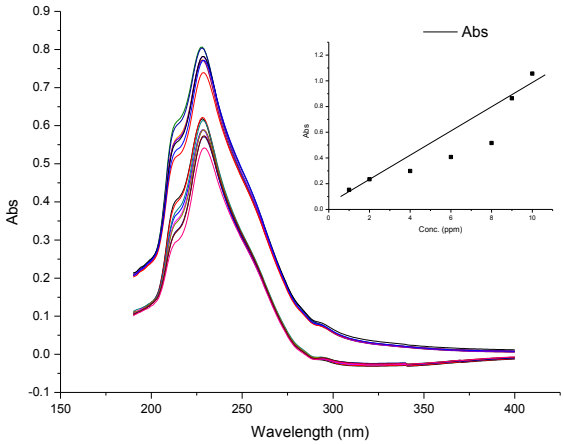

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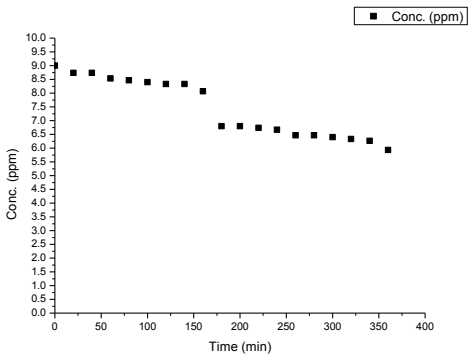

Concentration loss of 4f in SGF using UV spectra at specified time interval.

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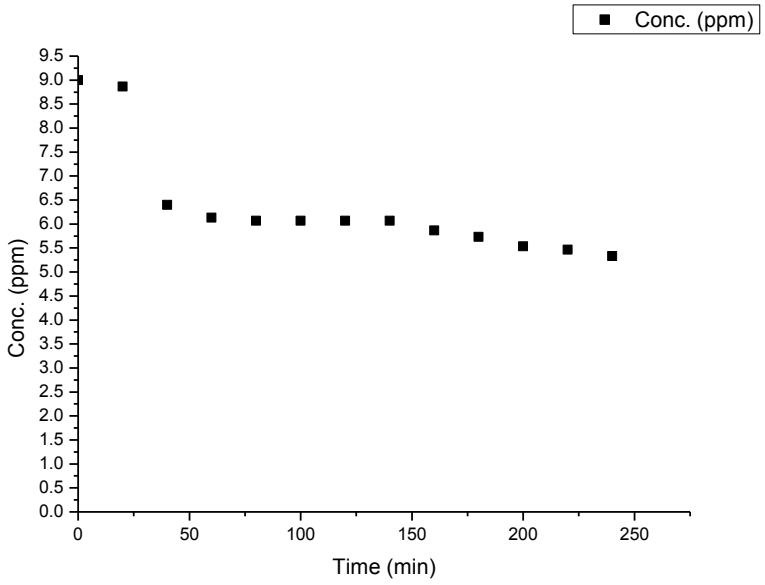

Concentration loss of 4f in BSA using UV- Visible spectra at specified time interval.

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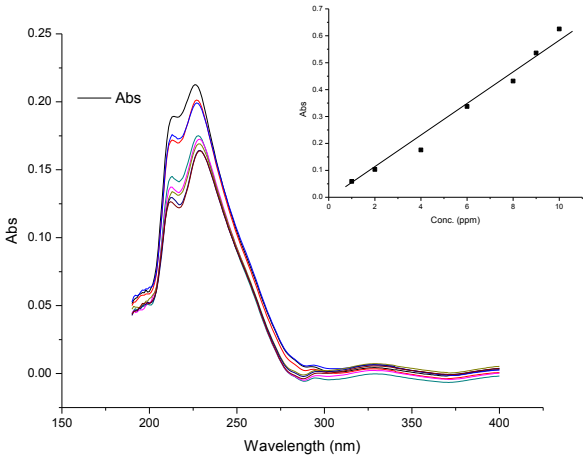

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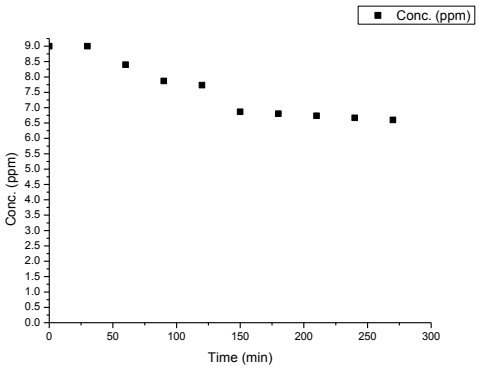

Concentration loss of 4g in SGF using UV spectra at specified time interval.

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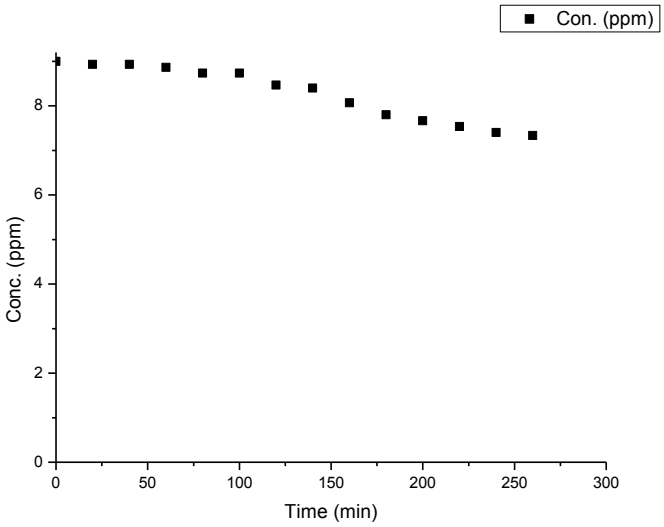

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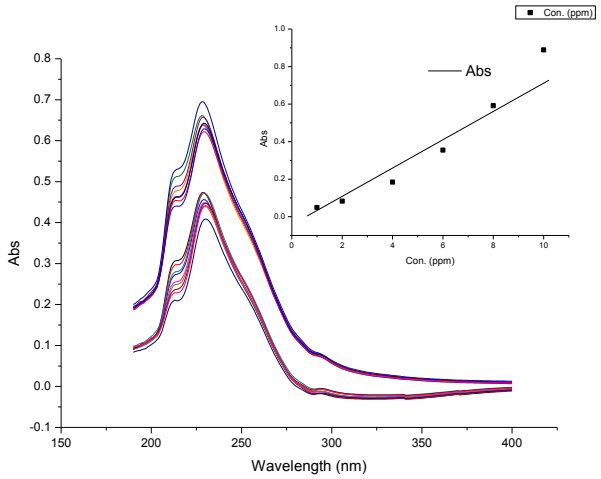

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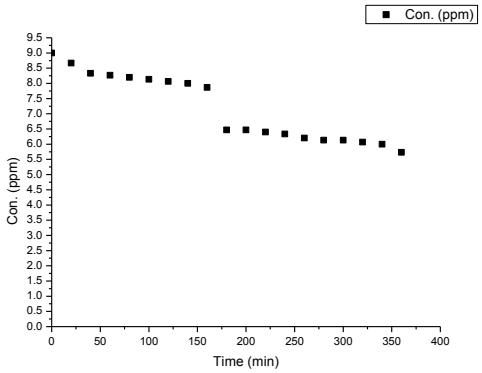

Concentration loss of 4i in SGF using UV spectra at specified time interval.

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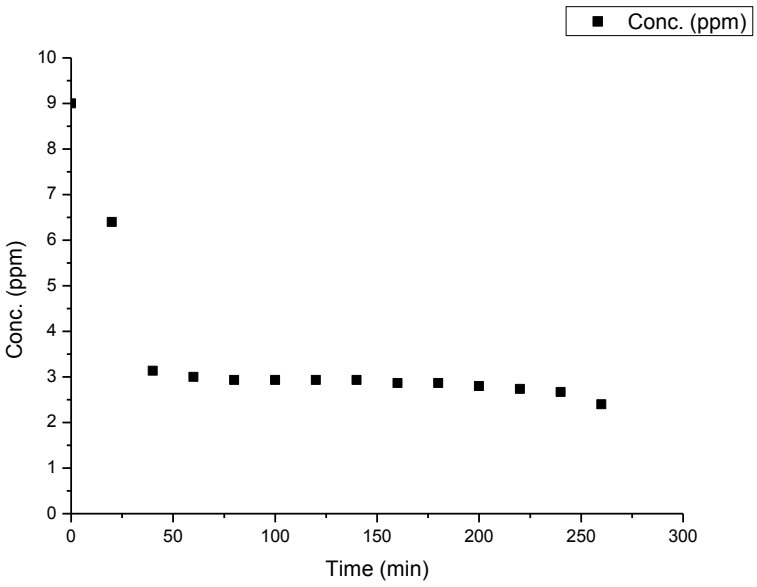

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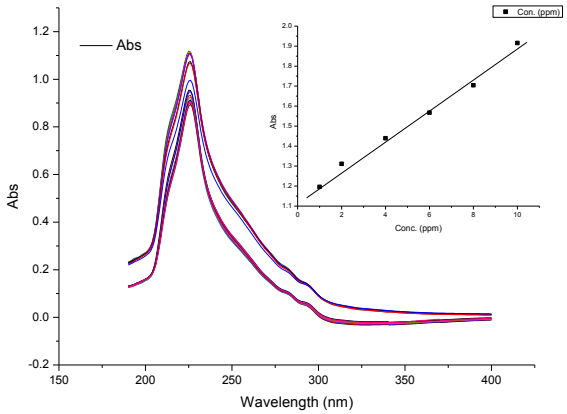

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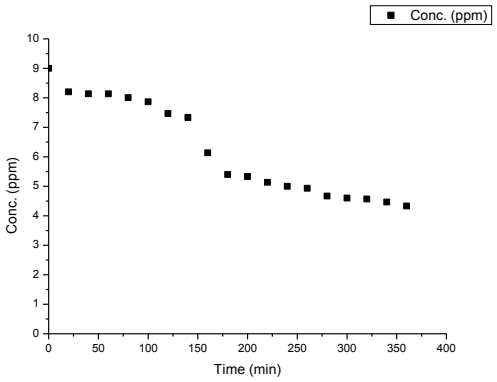

Concentration loss of 4j in SGF using UV spectra at specified time interval.

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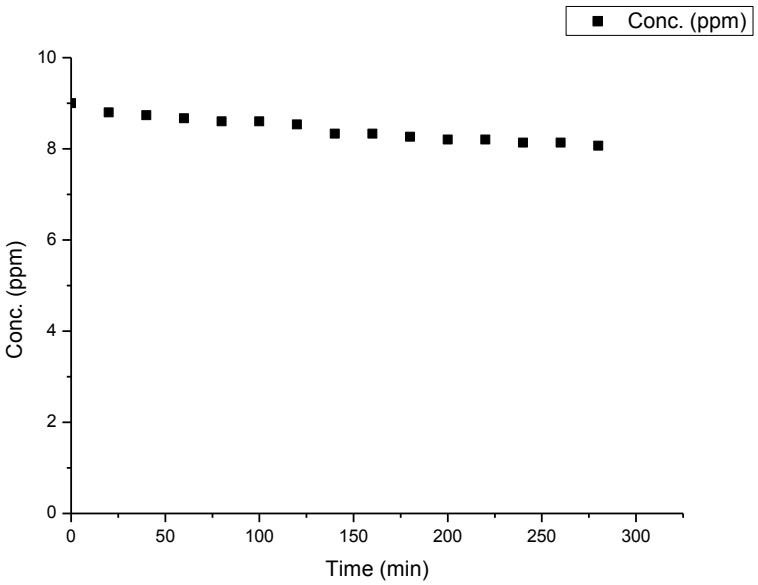

Concentration loss of the 4j in BSA using UV-Visible spectra at specified time interval.

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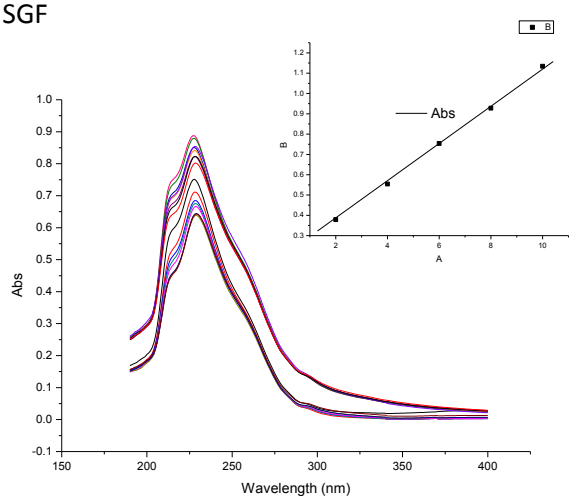

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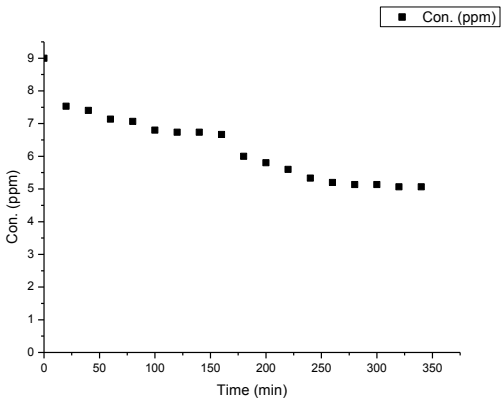
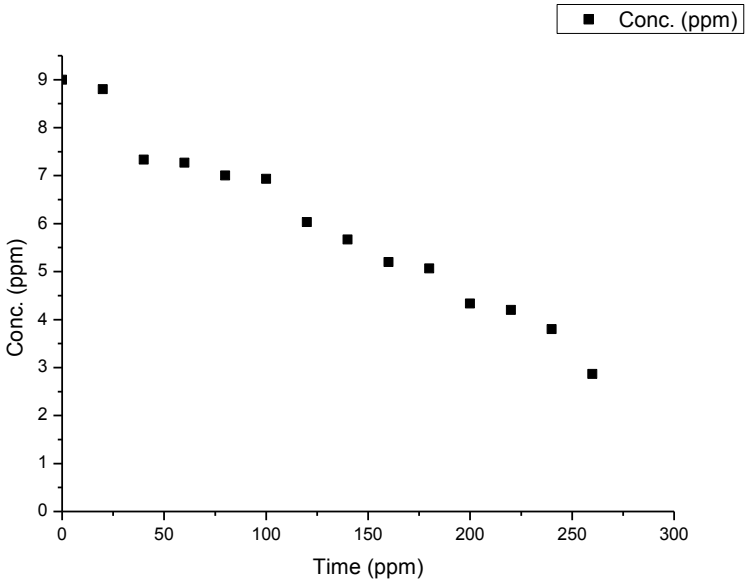


Concentration loss of 4m in SGF using UV spectra at specified time interval.

In BSA



Concentration loss of 4m in BSA using UV-Visible spectra at specified time interval.

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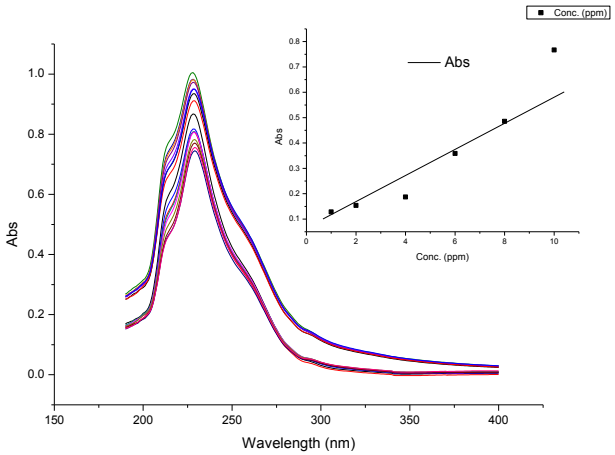

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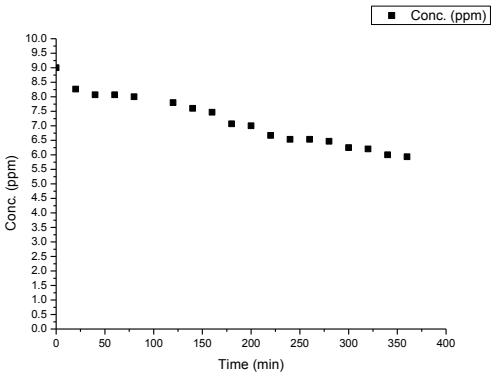
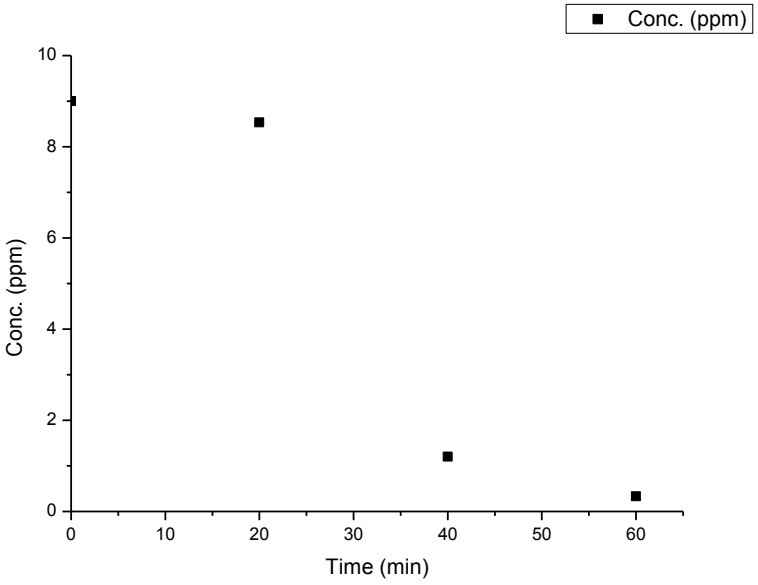


Concentration loss of 4n in SGF using UV spectra at specified time interval.

In BSA



Concentration loss of 4n in BSA using UV-Visible spectra at specified time interval.

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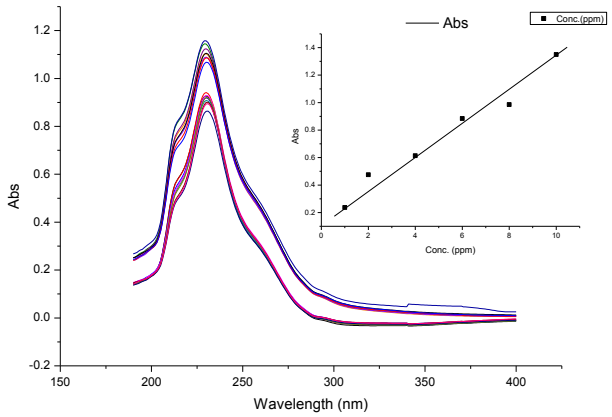
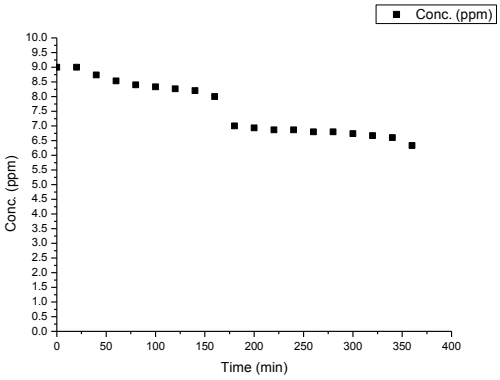
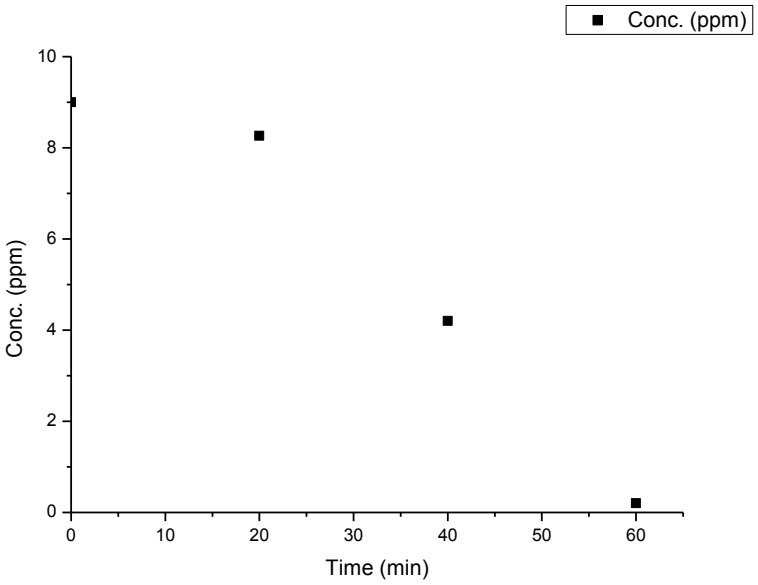


Stability of 4o in SGF using UV spectra at specified time interval.



Concentration loss of 4o in SGF using UV spectra at specified time interval.

In BSA



Concentration loss of 4o in BSA using UV-Visible spectra at specified time interval.

Table 5. Preliminary screening of pyrrolidones (4) for anticancer activity at 10 μM concentration.

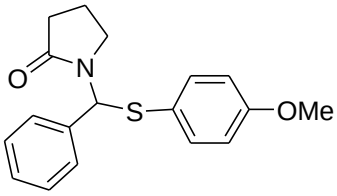
Entry	code	Screening Concentration (in 10 µM)							HTS Remarks *
		ACHN (Renal Cancer)	Panc1 (Pancreatic cancer)	Calu1 (lung cancer)	H460 (Non small cell lung cancer)	HCT116 (Colon cancer)	MCF 7 (Breast cancer)	MCF10A (Normal breast epitheliu m cells)	
1	4a	33	32	30	31	35	21	10	NA
2	4b	94	97	92	99	95	87	59	A
3	4c	46	45	42	41	52	39	45	NA
4	4d	41	40	41	42	45	34	42	NA
5	4e	0	10	11	17	12	7	0	NA
6	4f	98	97	95	93	99	79	52	A
7	4g	83	80	79	85	82	84	49	A
8	4i	95	96	99	92	94	81	39	A
9	4o	99	98	99	97	92	86	58	A
10	4p	4	9	18	16	21	17	0	NA
11	4n	99	97	95	96	93	77	37	A
12	4m	99	96	99	98	97	82	52	A
13	4k	0	10	12	16	10	14	0	NA
14	4l	24	31	32	30	29	32	20	NA
15	4j	97	89	86	85	84	75	49	A
16	4h	47	37	38	38	42	23	40	NA
Positive Control 1--->Flavopiridol		88	81	89	80	87	77	21	A
Positive Control 2 -->Gemcitabine		79	88	82	80	75	81	12	A

* A - Active and NA - Not Active.

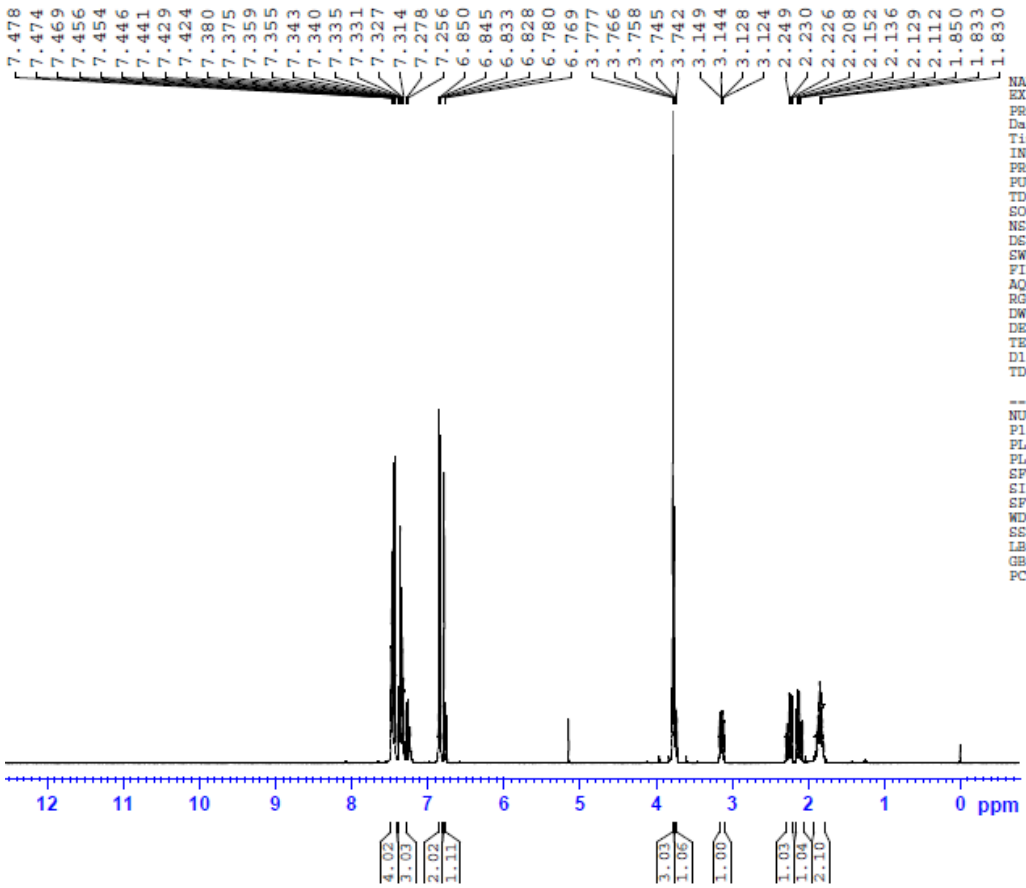
Table 6. Preliminary screening of pyrrolidones (4) for anticancer activity at lower concentration.

S.No	code	Screening conc. (µM)	ACHN	Panc1	Calu1	H460	HCT116	MCF7 (Breast cancer)	MCF10A	HTS Remarks
1	4a	0.3	0	0	2	8	0	2	0	Not Active
		1	5	14	8	15	11	7	0	
		3	18	24	16	23	24	20	6	
2	4b	0.3	20	19	13	3	22	23	2	Active
		1	42	47	36	34	38	49	25	
		3	67	68	54	52	66	70	40	
3	4c	0.3	2	0	0	5	2	2	0	Not Active
		1	17	14	8	16	4	16	12	
		3	24	33	28	32	39	35	21	
4	4d	0.3	0	2	9	5	0	4	0	Not Active
		1	15	16	17	21	16	17	10	
		3	24	38	29	34	27	27	22	
5	4e	0.3	0	0	0	0	0	3	0	Not Active
		1	0	0	0	0	0	3	0	
		3	0	5	8	1	2	7	0	
6	4f	0.3	36	28	35	27	24	30	16	Active
		1	52	62	55	53	50	54	24	
		3	76	79	83	75	76	65	45	
7	4g	0.3	21	16	11	21	15	18	0	Active
		1	50	54	49	47	54	56	26	
		3	72	79	65	68	76	81	34	
8	4i	0.3	32	24	19	16	13	26	10	Active
		1	55	51	64	56	59	53	22	
		3	76	72	86	77	72	74	27	

9	4p	0.3	28	22	24	17	16	3	16	Active
		1	70	66	56	53	53	3	24	
		3	84	83	87	68	87	85	43	
10	4o	0.3	0	0	0	0	0	2	0	Not Active
		1	0	0	0	0	0	2	0	
		3	0	0	6	4	16	2	0	
11	4n	0.3	12	16	28	11	7	18	4	Active
		1	48	44	47	32	29	47	18	
		3	64	75	63	59	52	45	26	
12	4m	0.3	16	8	4	6	7	2	0	Active
		1	37	24	32	29	31	3	16	
		3	56	67	64	57	68	69	34	
13	4k	0.3	0	0	0	0	0	3	0	Not Active
		1	0	0	0	0	0	3	0	
		3	2	4	1	7	6	5	0	
14	4l	0.3	5	2	0	0	8	3	0	Not Active
		1	7	8	4	2	12	3	0	
		3	17	26	16	24	18	28	13	
15	4j	0.3	6	19	13	9	13	21	4	Active
		1	18	33	26	20	24	35	16	
		3	50	53	49	44	56	35	32	
16	4h	0.3	5	0	0	5	2	3	0	Not Active
		1	17	16	11	15	16	3	12	
		3	26	24	18	23	27	5	35	
Positive Control 1--->Flavopiridol		1000nM	88	81	89	80	87	73	21	Active
Positive Control 2 -->Gemcitabine		1000nM	79	88	82	80	75	83	12	Active



1-((4-methoxyphenylthio)(phenyl)methyl)pyrrolidin-2-one (4a): White crystalline Solid; mp 99-100 ^{o}C; R_f 0.45 (60% ethyl acetate : hexanes); FTIR (KBr) *v*: 2946, 2885, 2831, 1689, 1585, 1488, 1453, 1403, 1349, 1248, 1171, 1016, 829 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.83-1.88 (m, CH_2, 2 H), 2.07-2.15(m, CH, 1 H), 2.20-2.27(m, CH, 1 H), 3.10-3.16(m, CH, 1 H), 3.72-3.76(m, CH, 1 H), 3.77(s, OCH_3, 3 H), 6.78 (s, CH, 1 H), 6.82-6.85 (d, J = 8.8 Hz, 2 H), 7.31-7.47 (m, ArH, 7 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 30.9, 41.9, 55.2, 62.0, 114.6, 122.3, 128.8, 129.0, 134.3, 135.0, 135.6, 160.1, 174.6 ppm; HRMS: calculated for $C_{18}H_{19}NO_2S$ ($[M]^+$) 313.1136, Found 313.1136.

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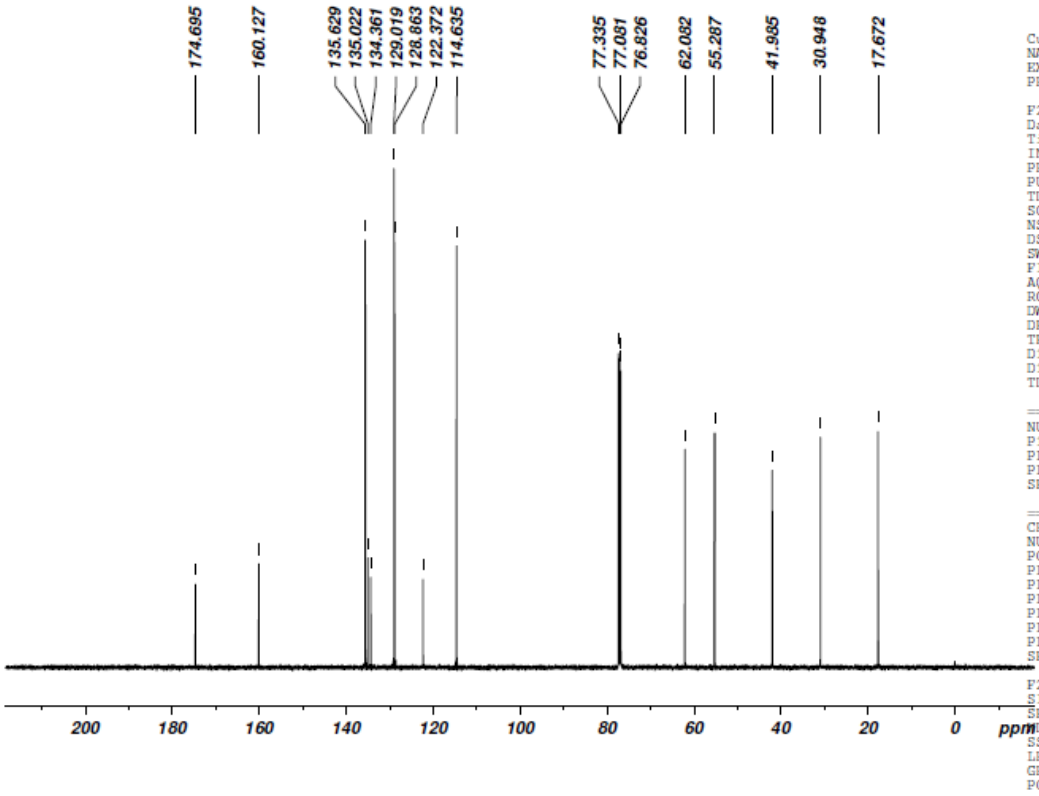


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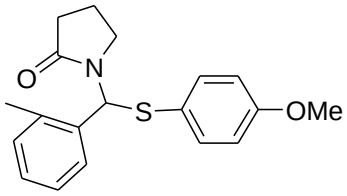
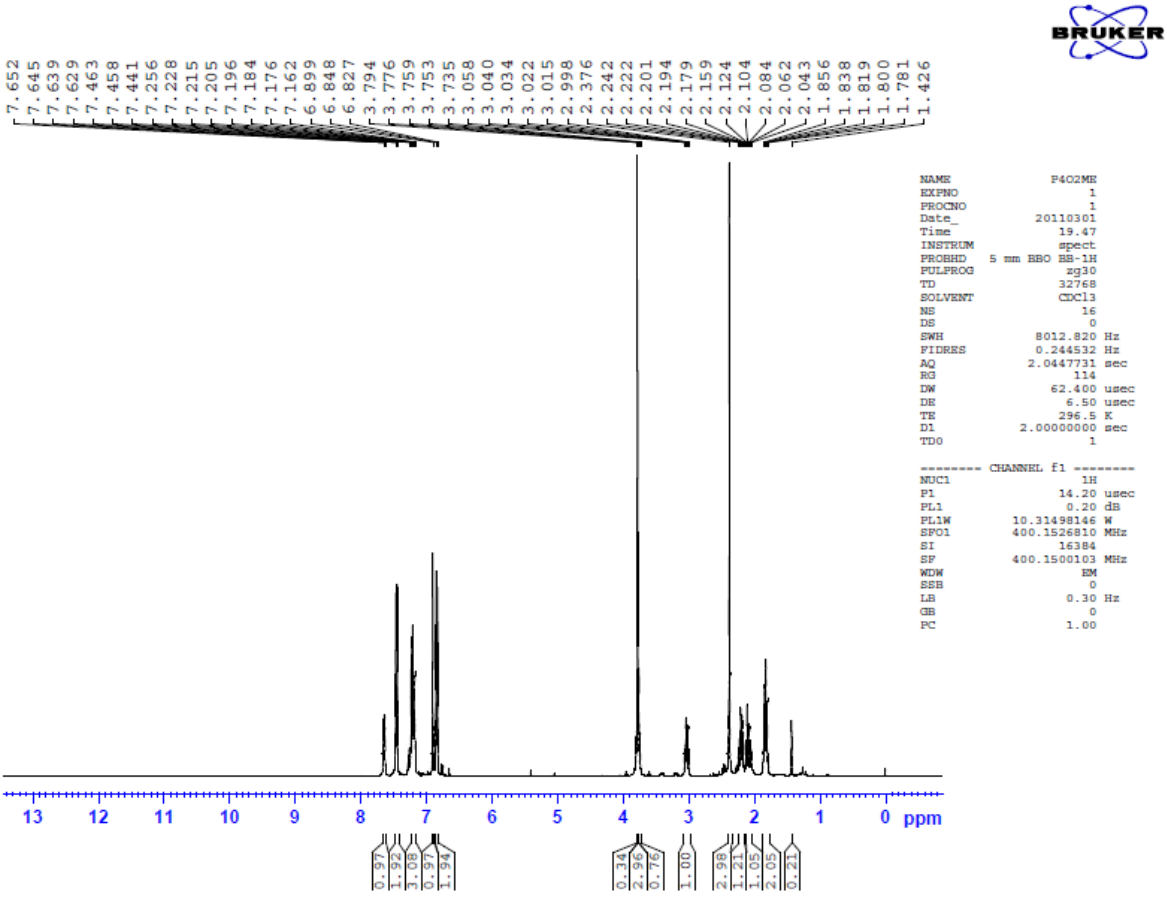
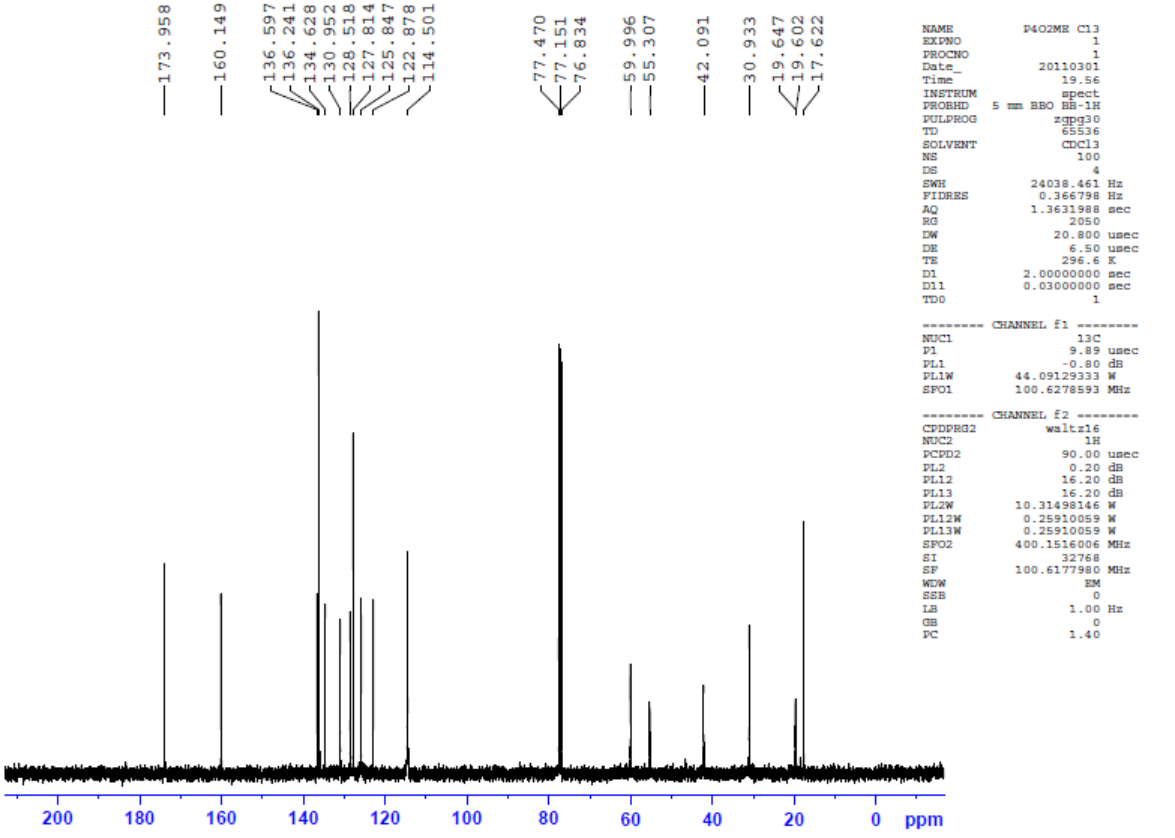
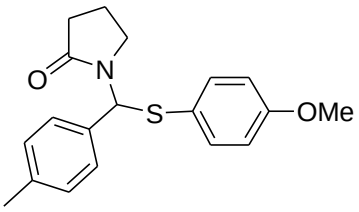


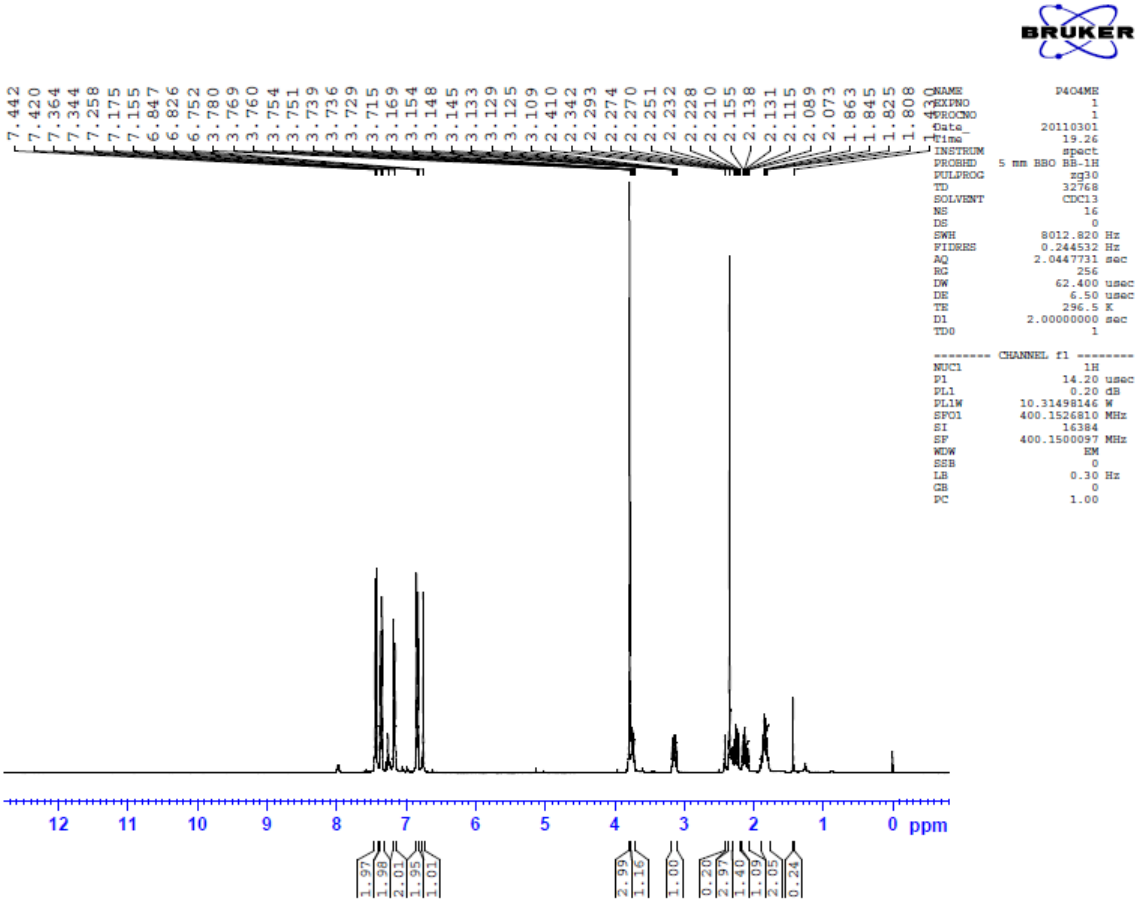
1-((4-methoxyphenylthio)(o-tolyl)methyl)pyrrolidin-2-one (4b): White crystalline Solid; mp 89-90 ^{o}C; R_f 0.46 (60% ethyl acetate : hexanes); FTIR (KBr) *v*: 2937, 2881, 1689, 1587, 1486, 1405, 1347, 1251, 1171, 1019, 829 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.78-1.85 (m, CH_2, 2 H), 2.04-2.12 (m, CH, 1 H), 2.15-2.24 (m, CH, 1 H), 2.37 (s, CH_3, 3 H), 2.99-3.05 (m, CH, 1 H), 3.73-3.79 (m, CH, 1 H), 3.77 (s, OCH_3, 3 H), 6.82-6.84 (d, *J*= 8.4 Hz, 2 H), 6.89 (s, CH, 1 H), 7.16-7.65 (m, ArH, 6 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 19.6, 30.9, 42.0, 55.3, 59.9, 114.5, 122.8, 125.8, 127.8, 128.5, 130.9, 134.6, 136.2, 136.5, 160.1, 173.9 ppm; HRMS: calculated for $C_{19}H_{21}NO_2S$ ([M]$^{+}$) 327.1293, Found 327.1293.







1-((4-methoxyphenylthio)(p-tolyl)methyl)pyrrolidin-2-one (4c): White crystalline Solid; mp 108-109 ^{o}C; R_f 0.47 (60% ethyl acetate : hexanes); FTIR (KBr) : 2945, 2898, 1676, 1585, 1494, 1458, 1407, 1106, 1027, 818 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.80-1.88 (m, CH_2, 2 H), 2.07-2.15 (m, CH, 1 H), 2.21-2.31 (m, CH, 1 H), 2.34 (s, CH_3, 1 H), 3.10-3.16 (m, CH, 1 H), 3.71-3.76 (m, CH, 1 H), 3.78 (s, OCH_3, 3 H), 6.75 (s, CH, 1 H), 6.82-6.84 (d, J = 8.4 Hz, 2 H), 7.15-7.44 (m, ArH, 6 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 21.1, 31.0, 42.0, 55.3, 62.4, 114.5, 122.9, 129.3, 127.8, 133.4, 135.5, 138.3, 159.9, 174.6 ppm; HRMS: calculated for $C_{19}H_{21}NO_2S$ ([M]$^{+}$) 327.1293, Found 327.1293.

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PROCNO               1
Date_         20110301
Time             19.26
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PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG                 256
DW              62.400 usec
DE               6.50 usec
TE              296.5 K
D1            2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               14.20 usec
PL1               0.20 dB
PL1W        10.31498146 W
SFO1        400.1526810 MHz
SI                16384
SF          400.1500097 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
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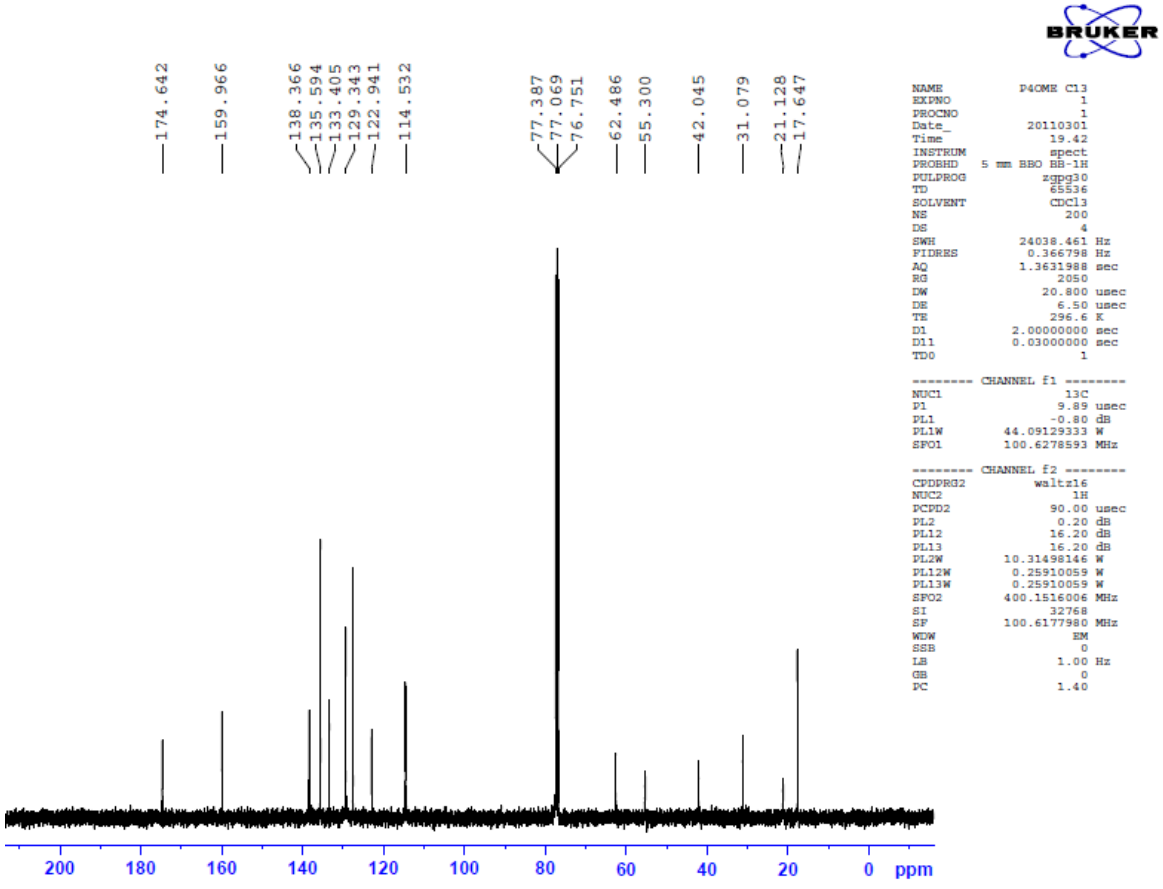


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EXPNO                1
PROCNO               1
Date_         20110301
Time             19.42
INSTRUM          spect
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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  200
DS                    4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW              20.800 usec
DE               6.50 usec
TE              296.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1               9.89 usec
PL1              -0.80 dB
PL1W        44.09129333 W
SFO1        100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2               0.20 dB
PL12             16.20 dB
PL13             16.20 dB
PL2W        10.31498146 W
PL12W        0.25910059 W
PL13W        0.25910059 W
SFO2        400.1516006 MHz
SI                32768
SF          100.6177980 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
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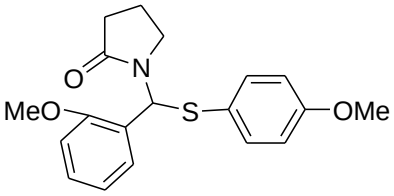
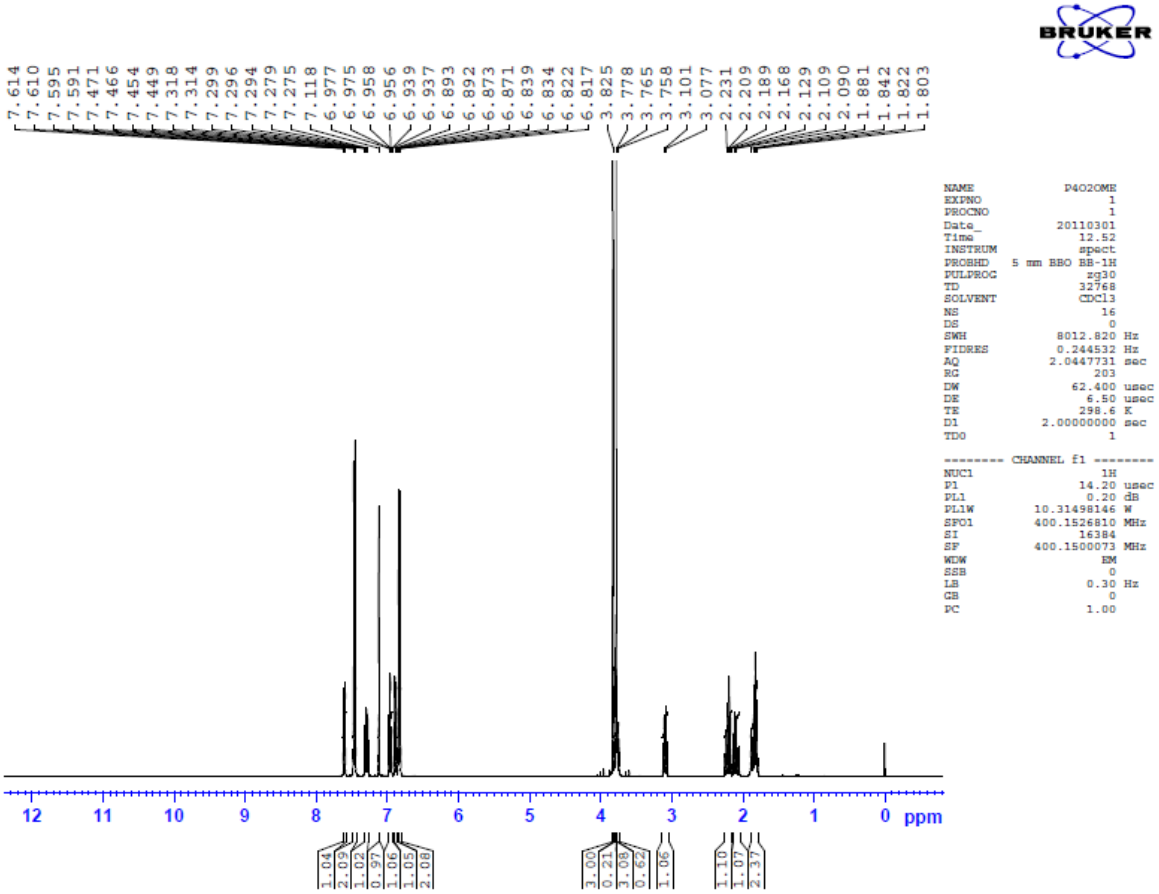
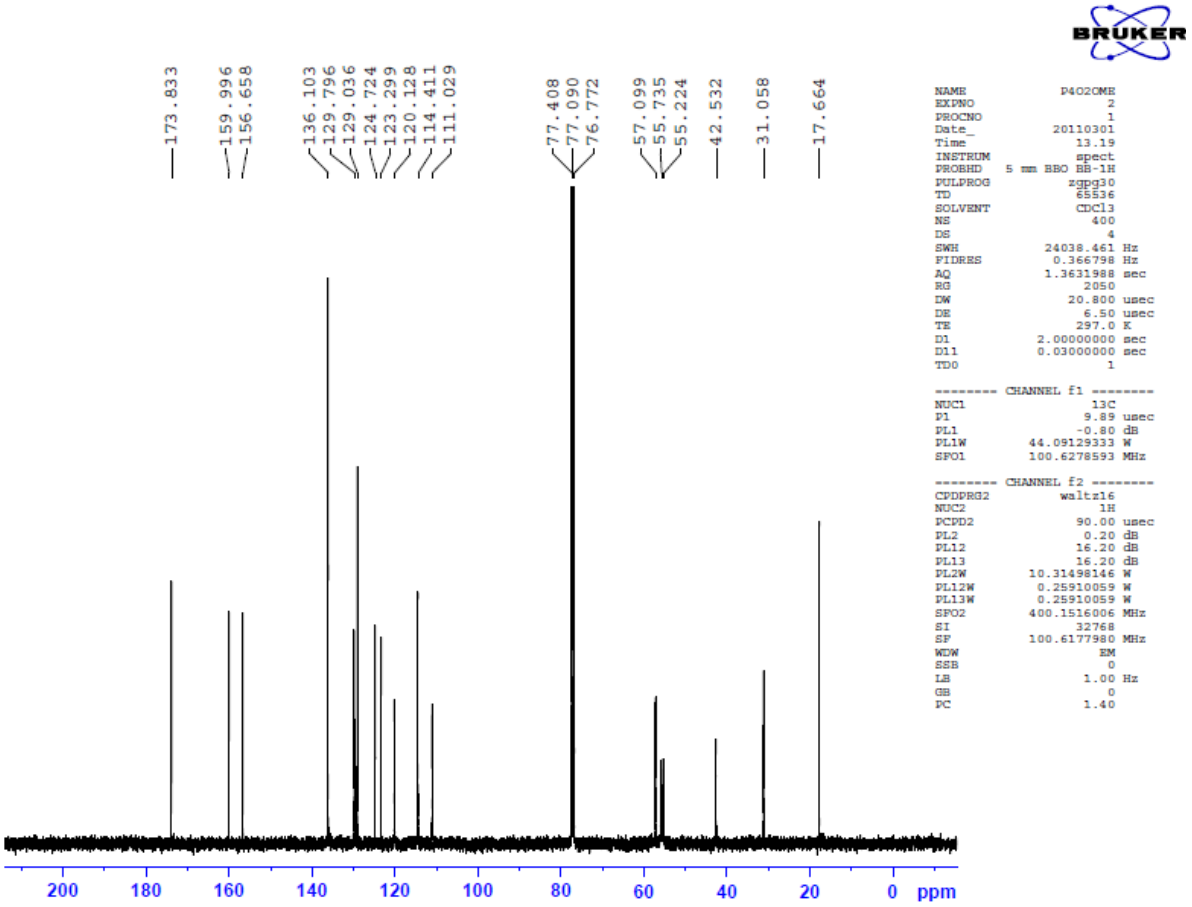
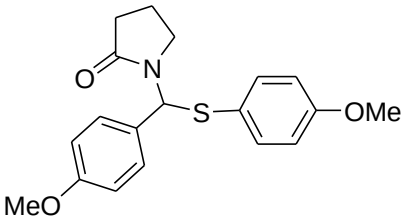


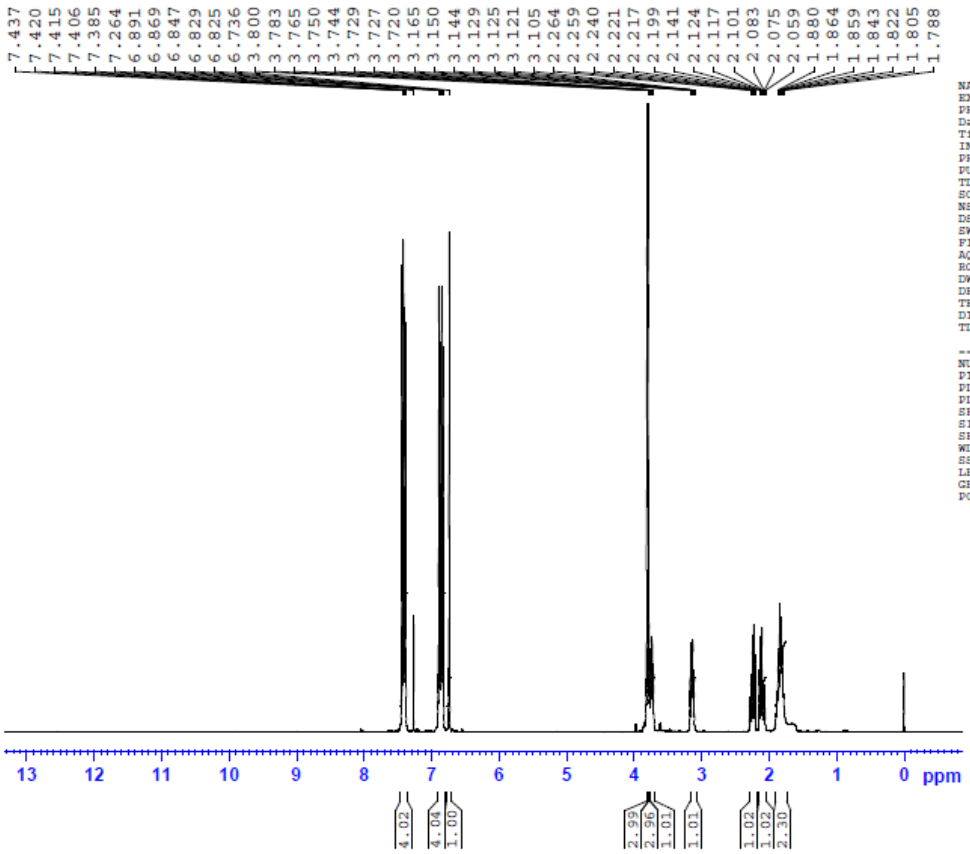
1-((4-methoxyphenylthio)(2-methoxyphenyl)methyl)pyrrolidin-2-one (4d): White crystalline Solid; mp 103-104 ^{o}C; R_f 0.42 (60% ethyl acetate : hexanes); FTIR (KBr) : 2963, 2891, 1694, 1590, 1487, 1401, 1101, 1024, 835 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.80-1.88 (m, CH_2, 2 H), 2.04-2.12 (m, CH, 1 H), 2.16-2.25 (m, CH, 1 H), 3.05-3.11 (m, CH, 1 H), 3.74-3.80 (m, CH, 1 H), 3.77 (s, OCH_3, 3 H), 3.82 (s, OCH_3, 3 H), 6.80-6.97 (m, ArH, 4 H), 7.11 (s, CH, 1 H), 7.26-7.61 (m, ArH, 4 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 31.0, 42.5, 55.2, 55.7, 57.0, 111.0, 114.4, 120.1, 123.2, 124.7, 129.0, 129.7, 136.1, 156.6, 159.9, 173.8 ppm; HRMS: calculated for $C_{19}H_{21}NO_3S$ ([M]$^{+}$) 343.1242, Found 343.1242.


7.614 7.610 7.595 7.591 7.471 7.466 7.454 7.449 7.318 7.314 7.299 7.296 7.294 7.279 7.275 7.118 6.977 6.975 6.958 6.956 6.939 6.937 6.893 6.892 6.873 6.871 6.839 6.834 6.822 6.817 3.825 3.778 3.765 3.758 3.101 3.077 2.231 2.209 2.189 2.168 2.129 2.109 2.090 1.881 1.842 1.822 1.803
NAME P402OME
EXPNO 1
PROCNO 1
Date_ 20110301
Time 12.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 203
DW 62.400 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 0.20 dB
PL1W 10.31498146 W
SFO1 400.1526810 MHz
SI 16384
SF 400.1500073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.04 2.09 1.02 0.97 1.06 1.05 2.08
3.00 0.21 3.08 0.62 1.06
1.10 1.07 2.37


173.833 159.996 156.658 136.103 129.796 129.036 124.724 123.299 120.128 114.411 111.029 77.408 77.090 76.772 57.099 55.735 55.224 42.532 31.058 17.664
NAME P402OME
EXPNO 2
PROCNO 1
Date_ 20110301
Time 13.19
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.89 usec
PL1 -0.80 dB
PL1W 44.09129333 W
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.20 dB
PL12 16.20 dB
PL13 16.20 dB
PL2W 10.31498146 W
PL12W 0.25910059 W
PL13W 0.25910059 W
SFO2 400.1516006 MHz
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



1-((4-methoxyphenylthio)(4-methoxyphenyl)methyl)pyrrolidin-2-one (4e): White crystalline Solid; mp 102-103 ^{o}C; R_f 0.43 (60% ethyl acetate : hexanes); FTIR (KBr) : 2954, 2891, 1691, 1593, 1503, 1404, 1176, 1024, 826 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.78-1.88 (m, CH_2, 2 H), 2.05-2.14 (m, CH, 1 H), 2.19-2.28 (m, CH, 1 H), 3.15-3.16 (m, CH, 1 H), 3.72-3.76 (m, CH, 1 H), 3.78 (s, OCH_3, 3 H), 3.80 (s, OCH_3, 3 H), 6.73 (s, CH, 1 H), 6.82-6.89 (m, ArH, 4 H), 7.38-7.43 (m, ArH, 4 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 31.0, 42.0, 55.2, 62.1, 113.9, 114.5, 122.9, 128.5, 128.9, 132.6, 135.5, 159.6, 159.9, 174.5 ppm; HRMS: calculated for $C_{19}H_{21}NO_3S$ ([M]$^{+}$) 343.1242, Found 343.1243.

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EXPNO                     1
PROCNO                    1
Date_              20110301
Time                  12.46
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES                0.244532 Hz
AQ                 2.0447731 sec
RG                      322
DW                   62.400 usec
DE                    6.50 usec
TE                   299.0 K
D1                 2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                   14.20 usec
PL1                   0.20 dB
PL1W               10.31498146 W
SFO1              400.1526810 MHz
SI                        16384
SF                400.1500070 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
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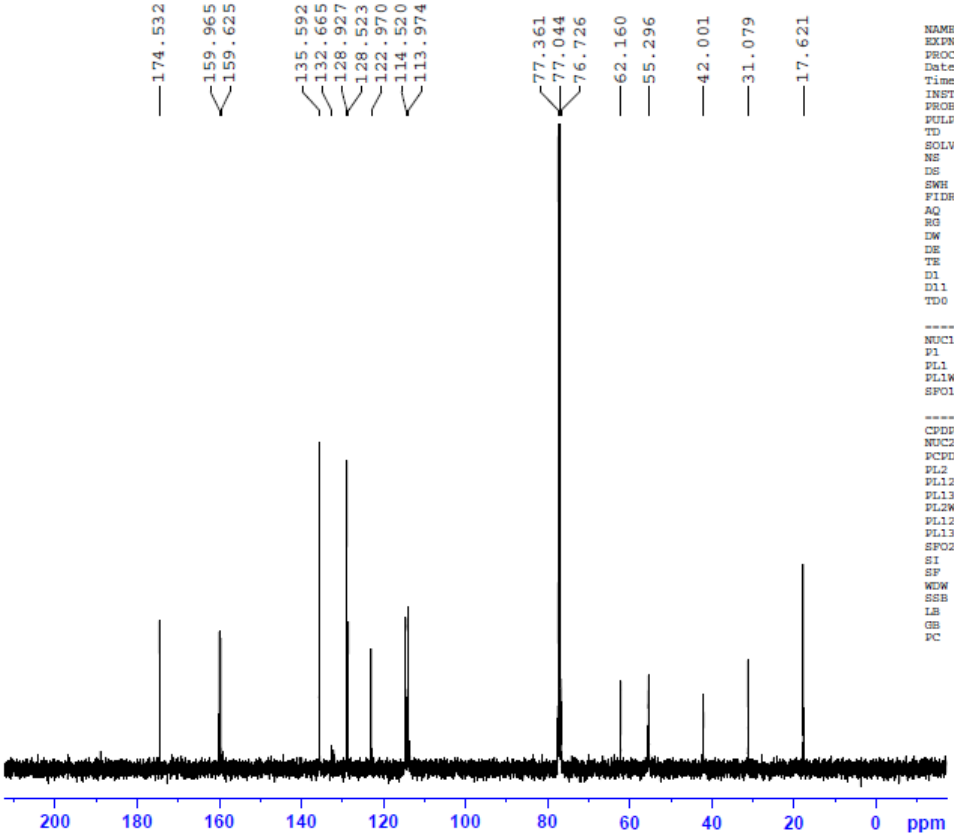


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NAME              24OHMS C13
EXPNO                     1
PROCNO                    1
Date_              20101207
Time                  13.59
INSTRUM               spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               24038.461 Hz
FIDRES                0.366798 Hz
AQ                 1.3631988 sec
RG                     2050
DW                   20.800 usec
DE                    6.50 usec
TE                   298.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                    9.89 usec
PL1                  -0.80 dB
PL1W              44.09129333 W
SFO1             100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                90.00 usec
PL2                   0.20 dB
PL12                 16.20 dB
PL13                 16.20 dB
PL2W              10.31498146 W
PL12W              0.25910059 W
PL13W              0.25910059 W
SFO2             400.1516006 MHz
SI                        32768
SF                100.6177980 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                    1.40
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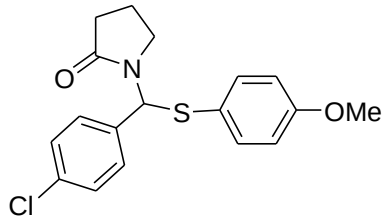
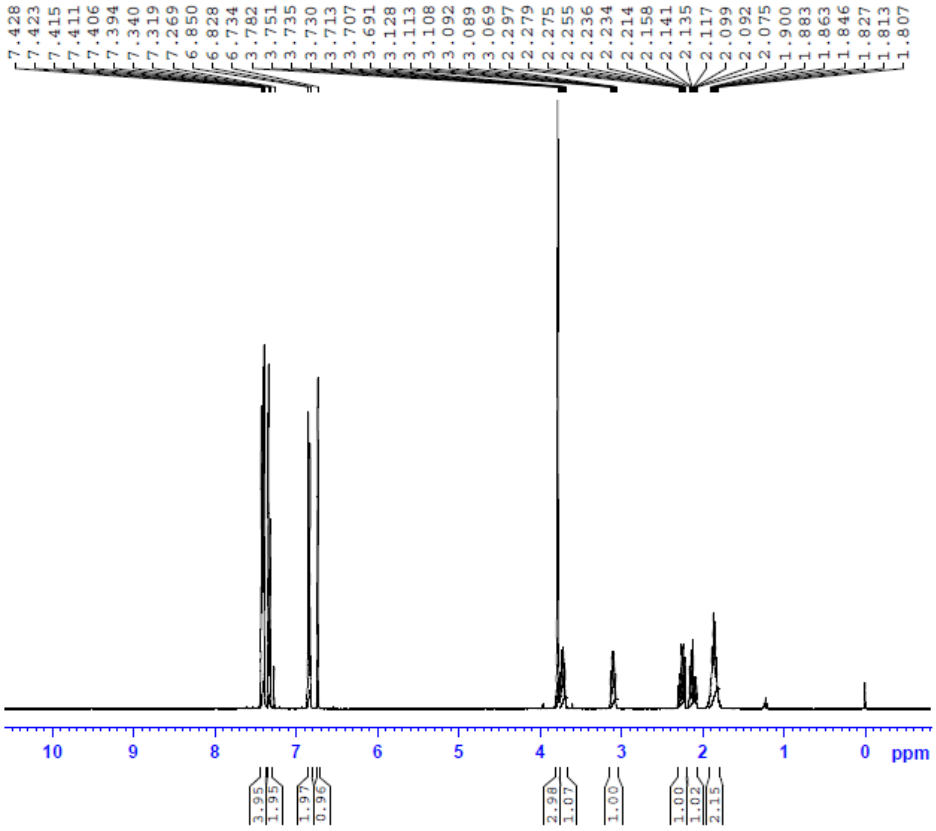


1-((4-methoxyphenylthio)(4-chlorophenyl)methyl)pyrrolidin-2-one (4f): White crystalline Solid; mp 111-112 ^{o}C; R_f 0.41 (60% ethyl acetate : hexanes); FTIR (KBr) : 2948, 2836, 1698, 1584, 1488, 1452, 1173, 1091, 1017, 823 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.80-1.93 (m, CH_2, 2 H), 2.07-2.11 (m, CH, 1 H), 2.13-2.29 (m, CH, 1 H), 3.06-3.12 (m, CH, 1 H), 3.69-3.75 (m, CH, 1 H), 3.78 (s, OCH_3, 3 H), 6.73 (s, CH, 1 H), 6.82-6.85 (d, J = 8.8 Hz, 2 H), 7.31-7.42 (m, ArH, 6 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 30.9, 41.9, 55.2, 62.0, 114.6, 122.3, 128.8, 129.0, 134.3, 135.0, 135.6, 160.1, 174.6 ppm; HRMS: calculated for $C_{18}H_{18}ClNO_2S$ ($[M]^+$) 347.0747, Found 347.0747.



BRUKER

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PROCNO	1
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INSTRUM	spect
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TD	32768
SOLVENT	CDCl3
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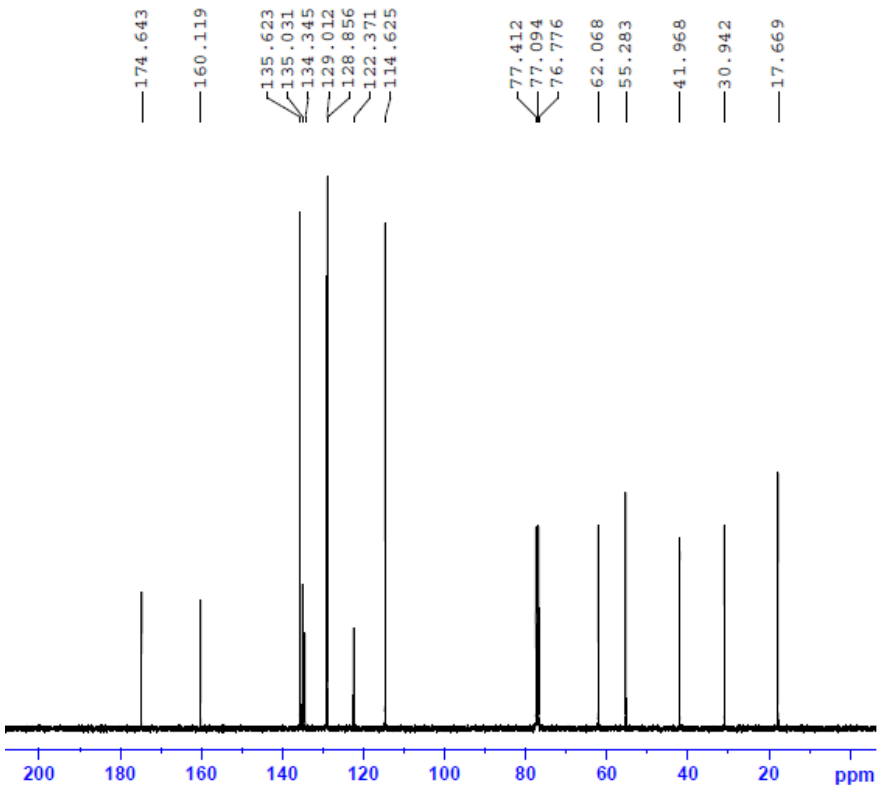
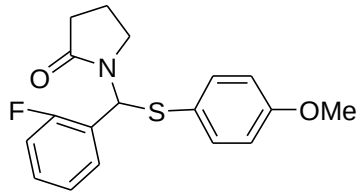
DS	0
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	2.0447731 sec
RG	181
DW	62.400 use
DE	6.50 use
TE	298.1 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.20 use
PL1	0.20 dB
PL1W	10.31498146 W
SFO1	400.1526810 MHz
SI	16384
SF	400.1500048 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

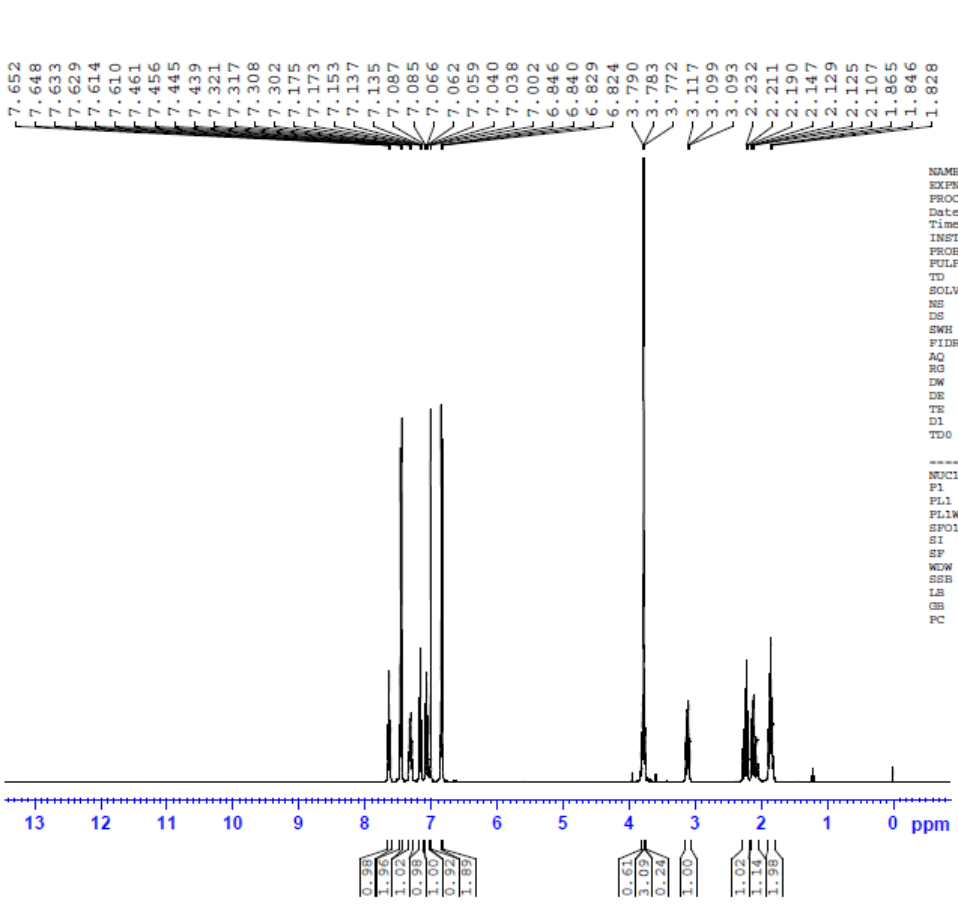


BRUKER

NAME	4CLOMS C13
EXPNO	1
PROCNO	1
Date_	20101207
Time	12.50
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.89 usec
PL1	-0.80 dB
PL1W	44.09129333 W
SFO1	100.6278593 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.20 dB
PL12	16.20 dB
PL13	16.20 dB
PL2W	10.31498146 W
PL12W	0.25910059 W
PL13W	0.25910059 W
SFO2	400.1516006 MHz
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



1-((4-methoxyphenylthio)(2-fluorophenyl)methyl)pyrrolidin-2-one (4g): White crystalline Solid; mp 113-114 ^{o}C; R_f 0.44 (60% ethyl acetate : hexanes); FTIR (KBr) : 2973, 2887, 1691, 1586, 1487, 1450, 1178, 1093, 1024, 834 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.82-1.88 (m, CH_2, 2 H), 2.06-2.14 (m, CH, 1 H), 2.19-2.27 (m, CH, 1 H), 3.07-3.13 (m, CH, 1 H), 3.74-3.80 (m, CH, 1 H), 3.77 (s, OCH_3, 3 H), 6.82-6.84 (d, *J*= 8.8 Hz, 2 H), 7.00 (s, CH, 1 H), 7.03-7.65 (m, ArH, 6 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 30.8, 42.3, 55.2, 56.5, 114.5, 115.8, 122.4, 123.7, 123.8, 124.0, 129.4, 130.4, 136.0, 158.5, 160.2, 161.0, 174.0 ppm; HRMS: calculated for $C_{18}H_{18}FNO_2S$ ([M]$^{+}$) 331.1042, Found 331.1042.

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PROCNO                  1
Date_            20110301
Time                16.48
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TD                 32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG                    114
DW               62.400 usec
DE                6.50 usec
TE                296.6 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.20 usec
PL1                 0.20 dB
PL1W          10.31498146 W
SFO1          400.1526810 MHz
SI                  16384
SF            400.1500014 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
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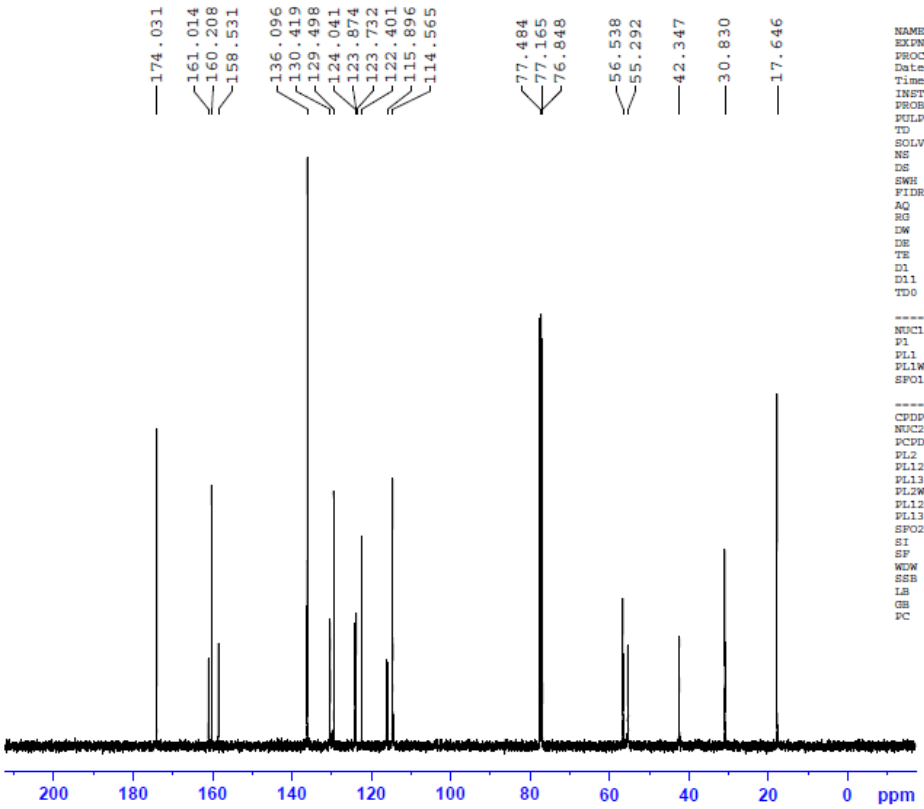


```
NAME             P402F C13
EXPNO                   1
PROCNO                  1
Date_            20110301
Time                17.03
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    200
DS                      4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   2050
DW               20.800 usec
DE                6.50 usec
TE                296.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.89 usec
PL1                -0.80 dB
PL1W         44.09129333 W
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                 0.20 dB
PL12               16.20 dB
PL13               16.20 dB
PL2W          10.31498146 W
PL12W          0.25910059 W
PL13W          0.25910059 W
SFO2          400.1516006 MHz
SI                  32768
SF            100.6177980 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
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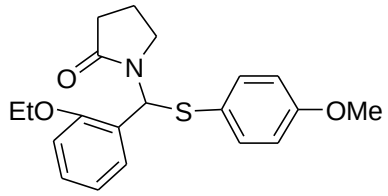
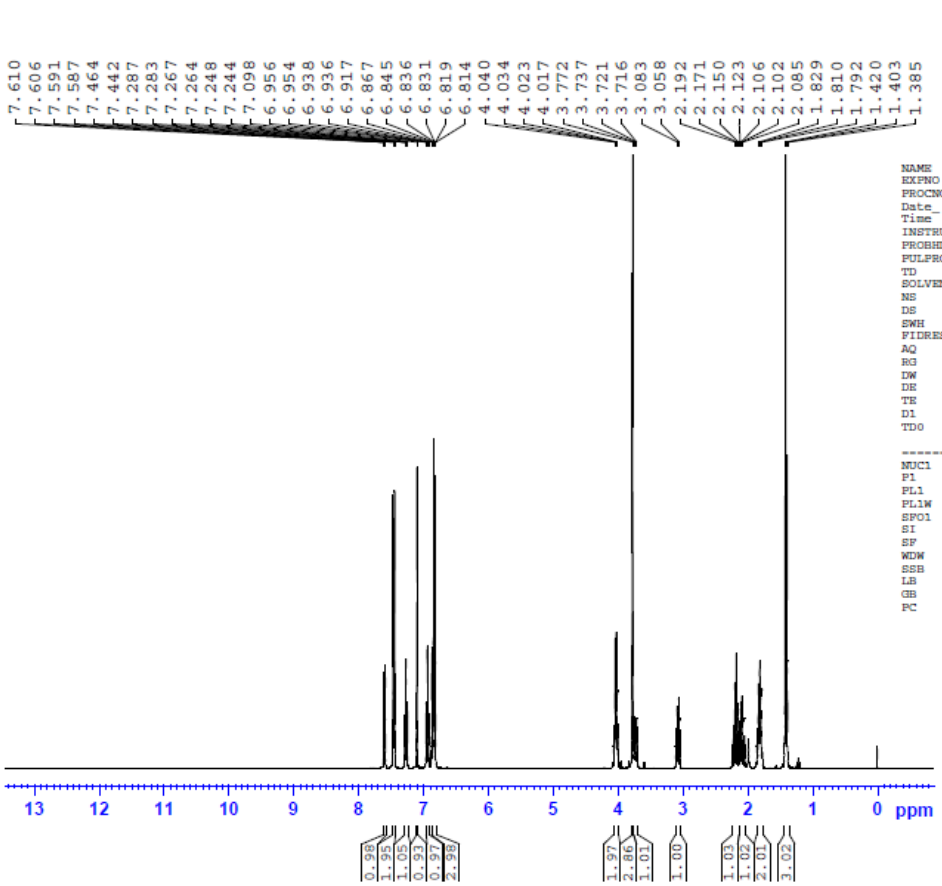


1-((4-methoxyphenylthio)(2-ethoxyphenyl)methyl)pyrrolidin-2-one (4h): White crystalline Solid; mp 115-116 ^{o}C; R_f 0.43 (60% ethyl acetate : hexanes); FTIR (KBr) : 2977, 2878, 1697, 1590, 1489, 1457, 1173, 1020, 828 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.38-1.42 (t, *J*=14 Hz, 3 H), 1.79-1.84 (m, CH_2, 2 H), 2.04-2.10 (m, CH, 1 H), 2.12-2.23 (m, CH, 1 H), 3.04-3.10 (m, CH, 1 H), 3.69-3.75 (m, CH, 1 H), 3.77 (s, OCH_3, 3 H), 4.00-4.04 (q, *J*= 8.8 Hz, 2 H), 6.81-6.95 (m, ArH, 4 H), 7.09 (s, CH, 1 H), 7.24-7.61 (m, ArH, 4 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 14.7, 17.2, 31.0, 42.6, 55.2, 57.5, 64.0, 112.0, 114.3, 120.0, 123.4, 125.1, 129.0, 129.6, 135.9, 156.1, 159.8, 173.5 ppm; HRMS: calculated for $C_{20}H_{23}NO_3S$ ([M]$^{+}$) 357.1399, Found 357.1398.

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EXPNO                   1
PROCNO                  1
Date_            20110301
Time                16.22
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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH           8012.820 Hz
FIDRES        0.244532 Hz
AQ            2.0447731 sec
RG                    144
DW               62.400 usec
DE                6.50 usec
TE                296.6 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.20 usec
PL1                 0.20 dB
PL1W         10.31498146 W
SFO1         400.1526810 MHz
SI                  16384
SF           400.1500073 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
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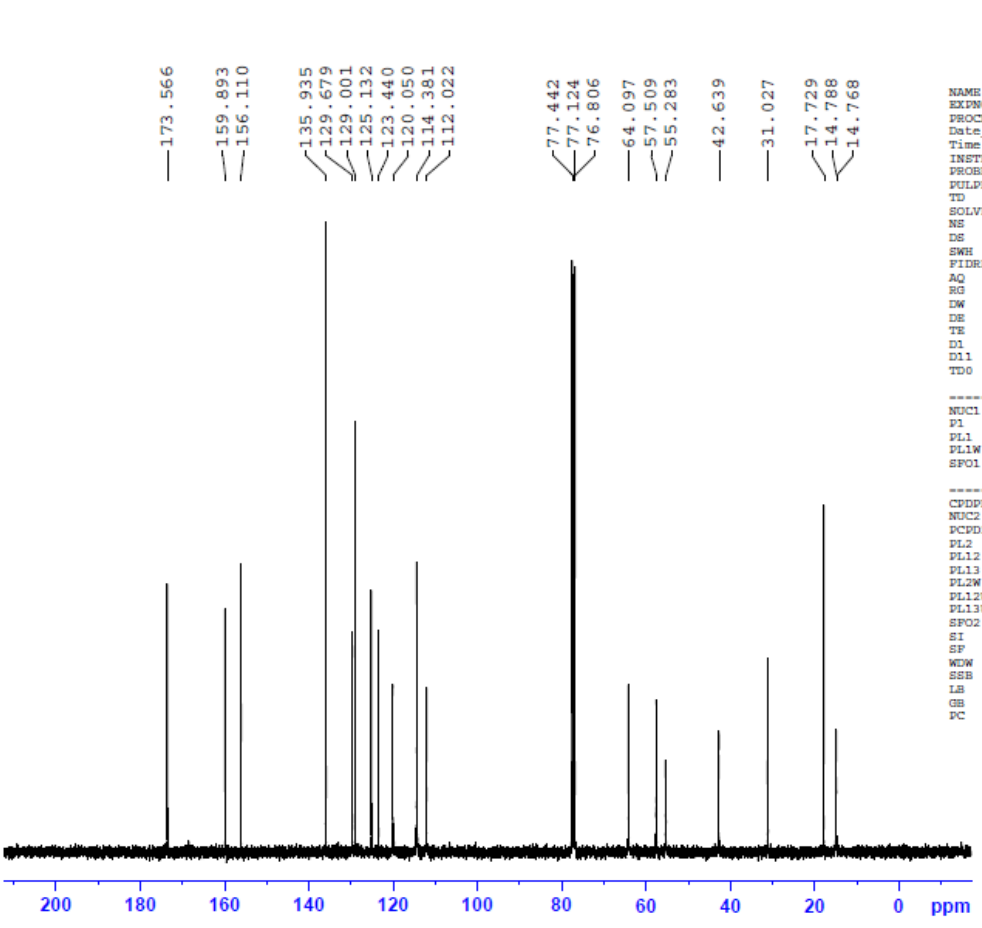


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NAME          P402OET C13
EXPNO                   1
PROCNO                  1
Date_            20110301
Time                16.43
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    300
DS                      4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   2050
DW               20.800 usec
DE                6.50 usec
TE                296.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.89 usec
PL1                -0.80 dB
PL1W         44.09129333 W
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             90.00 usec
PL2                 0.20 dB
PL12               16.20 dB
PL13               16.20 dB
PL2W         10.31498146 W
PL12W         0.25910059 W
PL13W         0.25910059 W
SFO2         400.1516006 MHz
SI                  32768
SF           100.6177980 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
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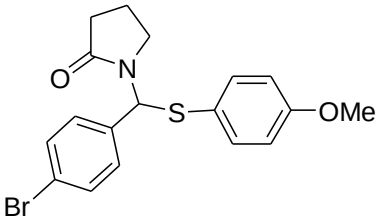
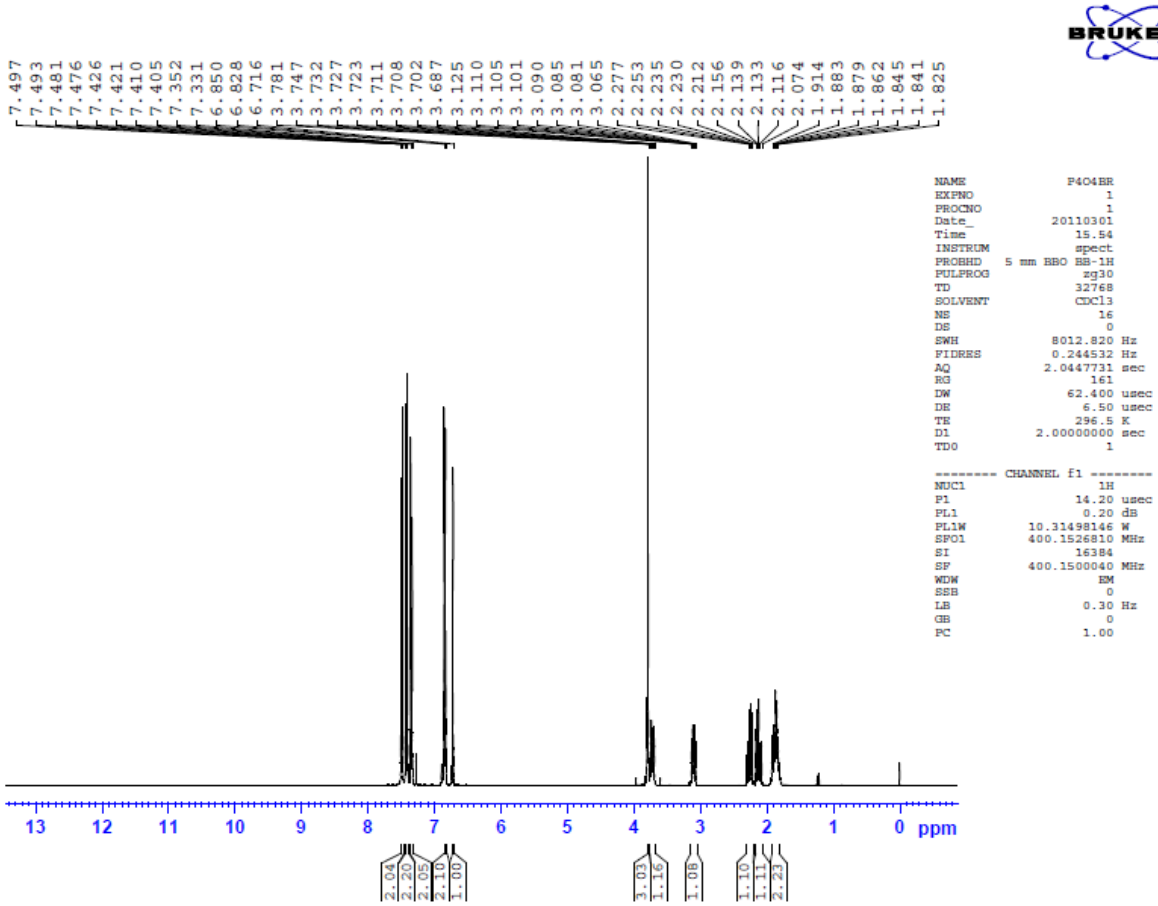
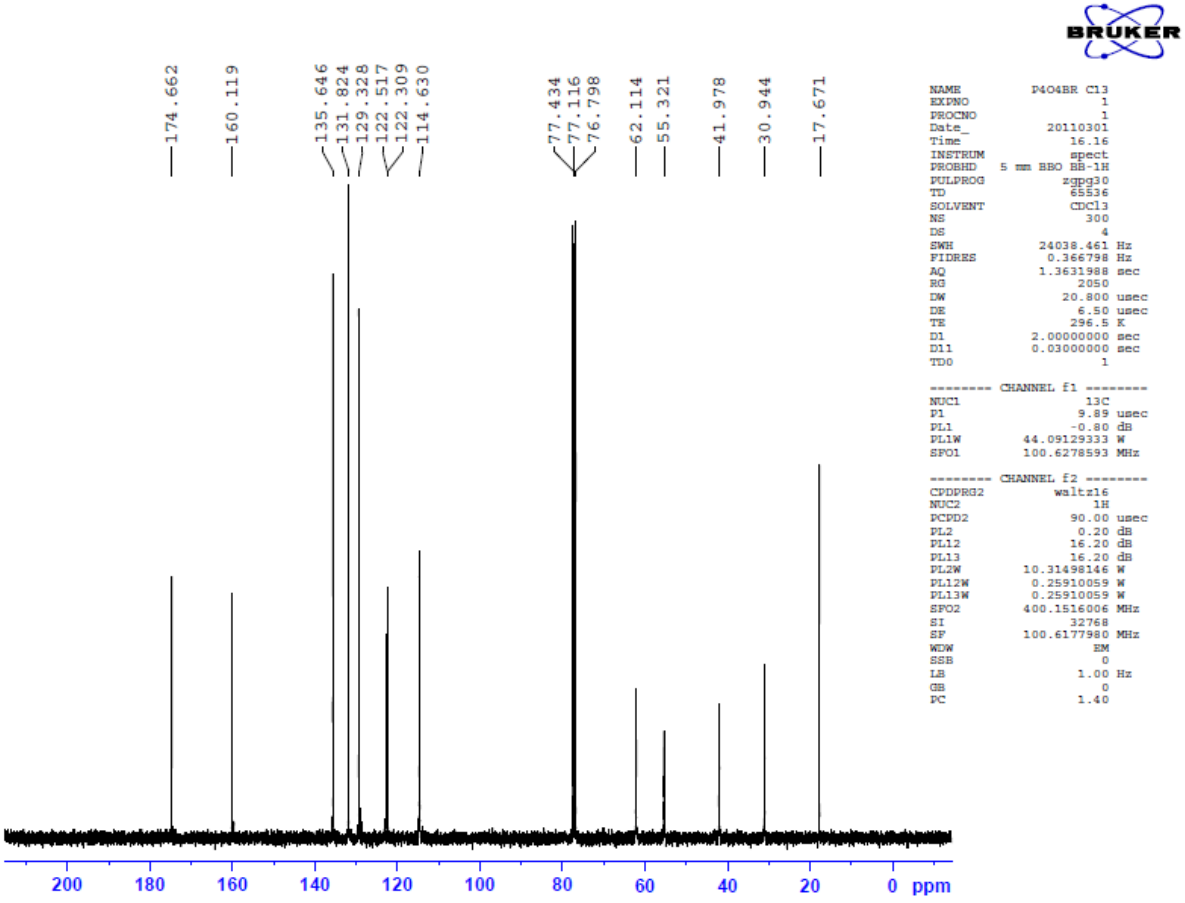
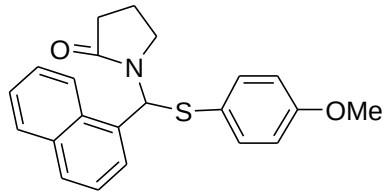


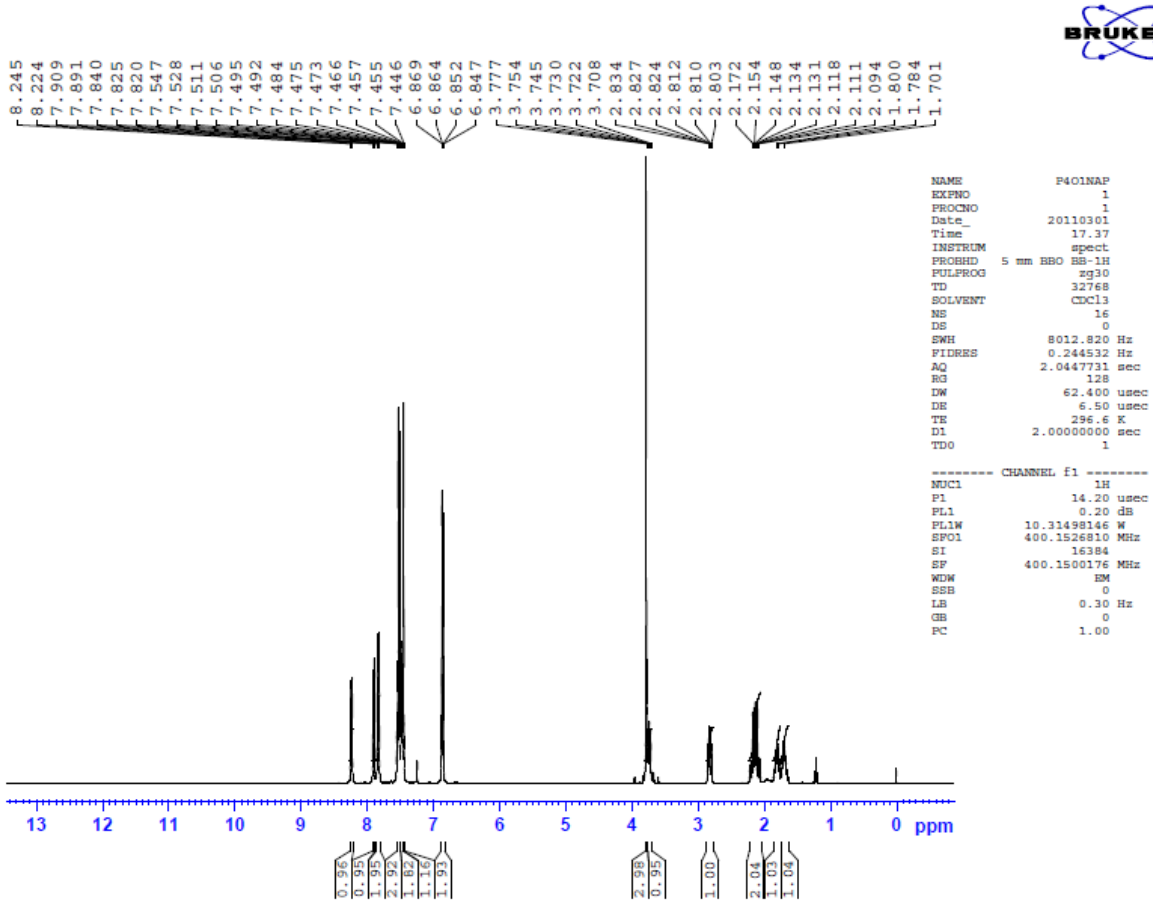
1-((4-methoxyphenylthio)(4-bromophenyl)methyl)pyrrolidin-2-one (4i): White crystalline Solid; mp 126-127 ^{o}C; R_f 0.46 (60% ethyl acetate : hexanes); FTIR (KBr) : 2950, 2888, 1698, 1583, 1485, 1173, 1009, 827 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.84-1.91 (m, CH_2, 2 H), 2.07-2.15 (m, CH, 1 H), 2.21-2.95 (m, CH, 1 H), 3.06-3.12 (m, CH, 1 H), 3.68-3.74 (m, CH, 1 H), 3.78 (s, OCH_3, 3 H), 6.71 (s, CH, 1 H), 6.82-6.85 (d, *J*= 8.8 Hz, 2 H), 7.34-7.49 (m, ArH, 6 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 30.9, 41,9, 55.3, 62.1, 114.6, 122.5, 129.3, 131.8, 135.6, 160.1, 174.6 ppm; HRMS: calculated for $C_{18}H_{18}BrNO_2S$ ([M] $^{+}$) 391.0242, Found 391.0242.







1-((4-methoxyphenylthio)(naphthalen-1-yl)methyl)pyrrolidin-2-one (4j): Light yellow crystalline Solid; mp 120-121 ^{o}C; R_f 0.48 (60% ethyl acetate : hexanes); FTIR (KBr) : 2964, 2830, 1686, 1588, 1491, 1182, 1026, 835 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.74-1.83 (m, CH_2, 2 H), 2.05-2.21 (m, CH, 2 H), 2.78-2.84 (m, CH, 1 H), 3.70-3.75 (m, CH, 1 H), 3.77 (s, OCH_3, 3 H), 6.84-6.86 (d, *J*= 8 Hz, 2 H), 7.44 (s, CH, 1 H), 7.45-8.24 (m, ArH, 9 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.4, 30.9, 41,8, 55.3, 59.7, 114.5, 122.5, 123.7, 124.8, 125.9, 126.1, 127.1, 128.6, 129.4, 131.0, 132.3, 133.8, 136.6, 160.3, 173.8 ppm; HRMS: calculated for $C_{22}H_{21}NO_2S$ ([M]$^{+}$) 363.1293, Found 363.1292.

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EXPNO                     1
PROCNO                    1
Date_              20110301
Time                  17.37
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PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8012.820 Hz
FIDRES            0.244532 Hz
AQ                2.0447731 sec
RG                      128
DW                   62.400 usec
DE                    6.50 usec
TE                   296.6 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    14.20 usec
PL1                    0.20 dB
PL1W             10.31498146 W
SFO1             400.1526810 MHz
SI                      16384
SF               400.1500176 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
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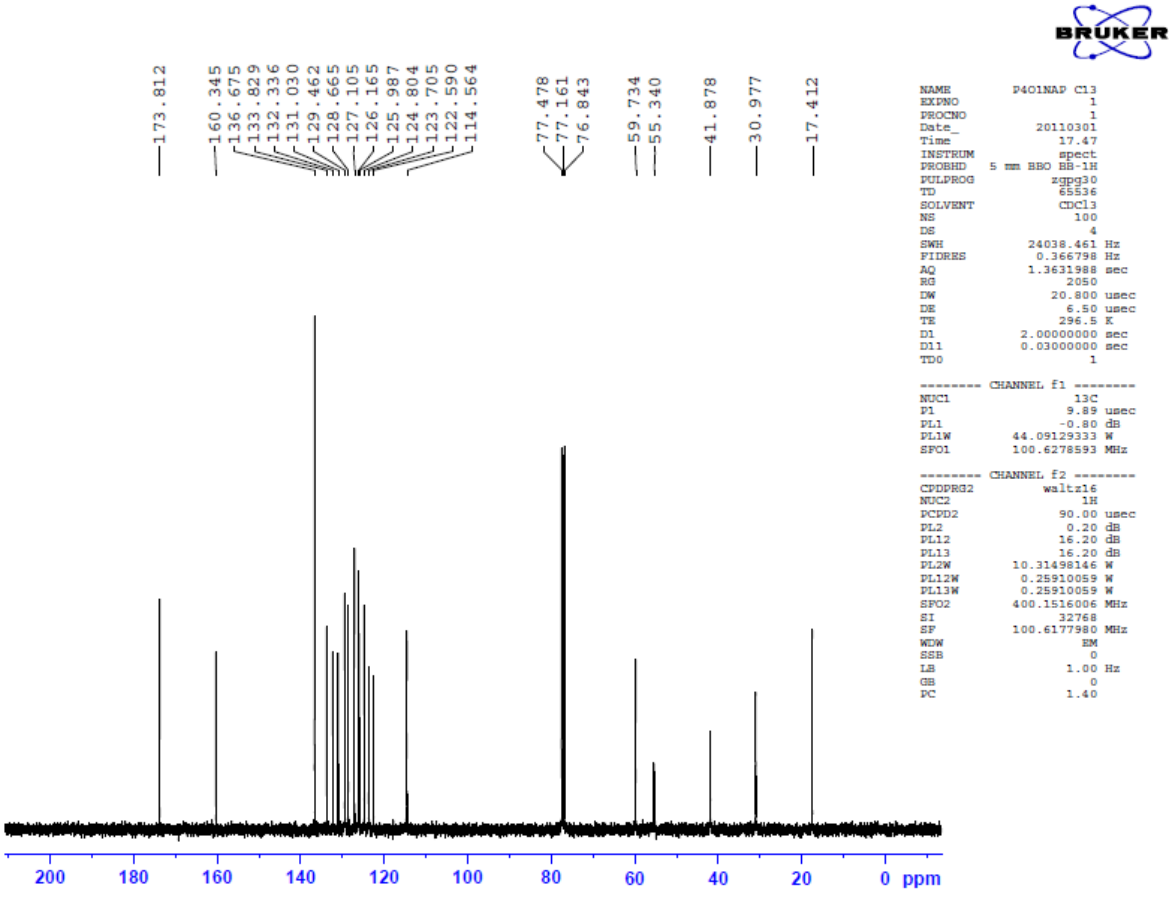


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NAME          P4O1NAP C13
EXPNO                  1
PROCNO                 1
Date_           20110301
Time               17.47
INSTRUM            spect
PROBHD  5 mm BBO BB-1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.89 usec
PL1                -0.80 dB
PL1W         44.0912933 W
SFO1         100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2                0.20 dB
PL12              16.20 dB
PL13              16.20 dB
PL2W         10.31498146 W
PL12W         0.25910059 W
PL13W         0.25910059 W
SFO2         400.1516006 MHz
SI                 32768
SF           100.6177980 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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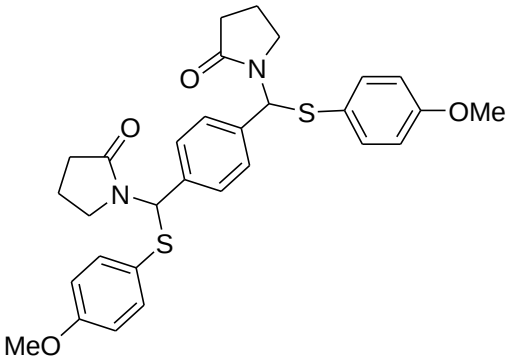
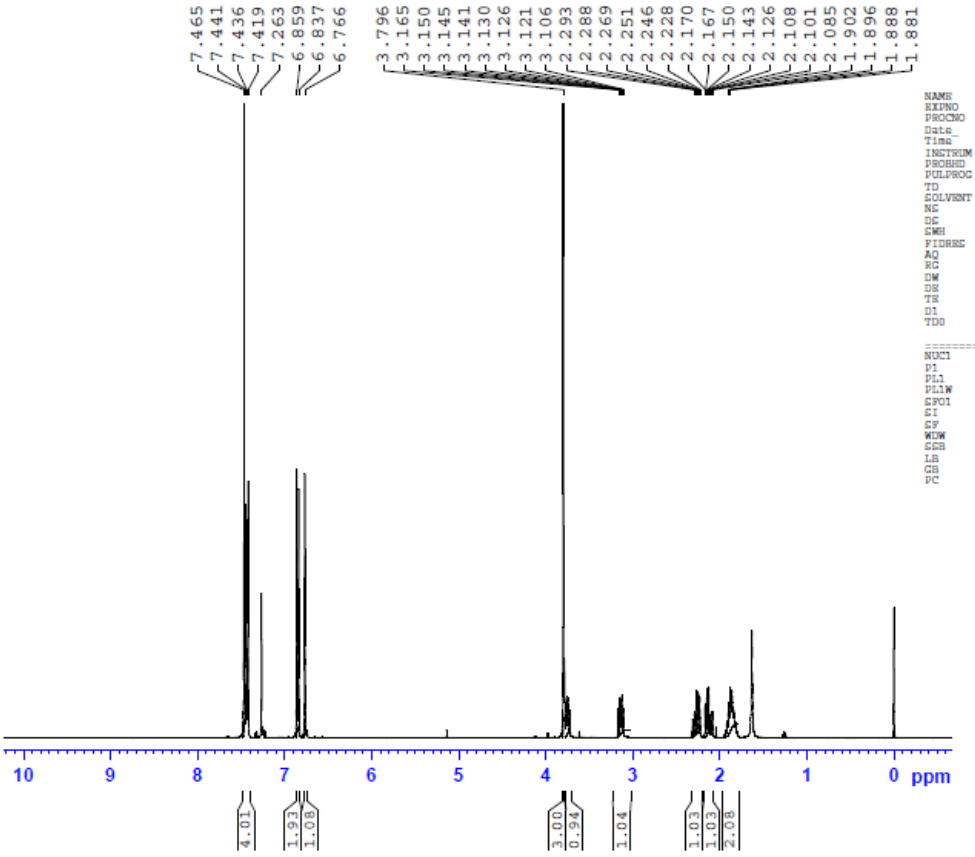


1,1'-(1,4-phenylenebis((4-methoxyphenylthio)methylene))dipyrrolidin-2-one (4k): White Solid; mp 167-168 ^{o}C; R_f 0.51 (60% ethyl acetate : hexanes); FTIR (KBr) : 2945, 2836, 1688, 1587, 1493, 1173, 1025, 846 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.84-1.90 (m, CH_2, 4 H), 2.08-2.29 (m, CH, 4 H), 3.10-3.14 (m, CH, 2 H), 3.70-3.78 (m, CH, 2 H), 3.79 (s, OCH_3, 6 H), 6.76 (s, CH, 2 H), 6.83-6.85 (d, *J*= 8Hz, 4 H), 7.26-7.46 (m, ArH, 8 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 30.9, 42,0, 55.2, 62.3, 114.3, 114.6, 122.5, 127.9, 135.5, 136.7, 160.0, 174.6 ppm; HRMS: calculated for $C_{30}H_{32}N_2O_4S_2$ ($[M]^+$) 548.1803, Found 548.1802.



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PROCNO	1
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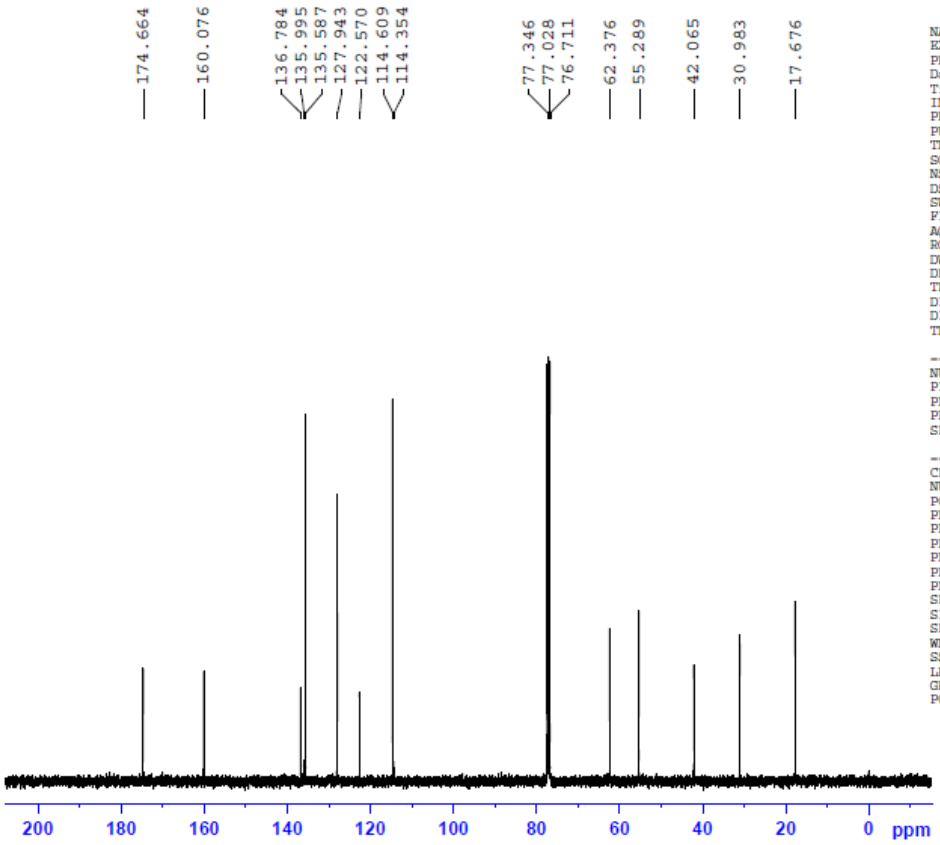
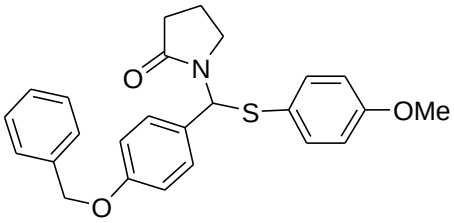
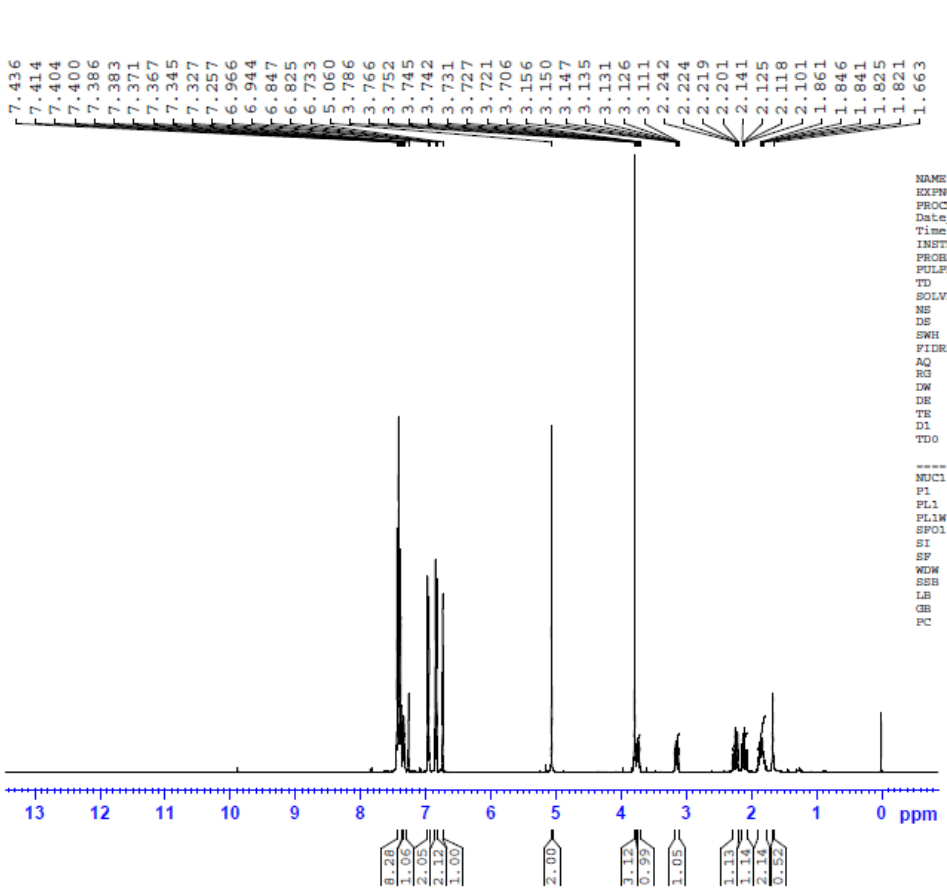
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	8012.820 Hz
FIDRES	0.244532 Hz
AQ	2.0447731 sec
RG	1030
DW	62.400 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.20 usec
PL1	0.20 dB
PL1W	10.31498146 W
SFO1	400.1526810 MHz
SI	16384
SF	400.1500077 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	TETOMS C13
EXPNO	1
PROCNO	1
Date_	20101207
Time	13.24
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	500
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	298.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.89 usec
PL1	-0.80 dB
PL1W	44.09129333 W
SFO1	100.6278593 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.20 dB
PL12	16.20 dB
PL13	16.20 dB
PL2W	10.31498146 W
PL12W	0.25910059 W
PL13W	0.25910059 W
SFO2	400.1516006 MHz
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



1-((4-methoxyphenylthio)(4-(benzyloxy)phenyl)methyl)pyrrolidin-2-one (4l): White Solid; mp 105-106 ^{o}C; R_f 0.42 (60% ethyl acetate : hexanes); FTIR (KBr) : 2942, 2836, 1691, 1592, 1498, 1171, 1009, 830 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.63-1.90 (m, CH_2, 2 H), 2.08-2.28 (m, CH_2, 2 H), 3.11-3.17 (m, CH, 2 H), 3.70-3.77 (m, CH, 2 H), 3.78 (s, OCH_3, 3 H), 6.73 (s, CH, 1 H), 6.82-6.84 (d, *J*= 8.8Hz, 2 H), 6.94-6.96 (d, *J*= 8.8Hz, 2 H), 7.32-7.43 (m, ArH, 8 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 31.0, 41.9, 55.2, 62.1, 70.0, 114.5, 114.8, 122.9, 127.4, 128.0, 128.6, 128.8, 128.9, 135.6, 136.7, 158.8, 159.9, 174.5 ppm; HRMS: calculated for $C_{25}H_{25}NO_3S$ ([M]$^{+}$) 419.1555, Found 419.1556.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6.847
6.825
6.733
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3.786
3.766
3.752
3.745
3.742
3.731
3.727
3.721
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3.150
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3.111
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2.219
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2.141
2.125
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2.101
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EXPNO 1
PROCNO 1
Date_ 20110301
Time 17.56
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 362
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.20 usec
PL1 0.20 dB
PL1W 10.31498146 W
SFO1 400.1526810 MHz
SI 16384
SF 400.1500103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
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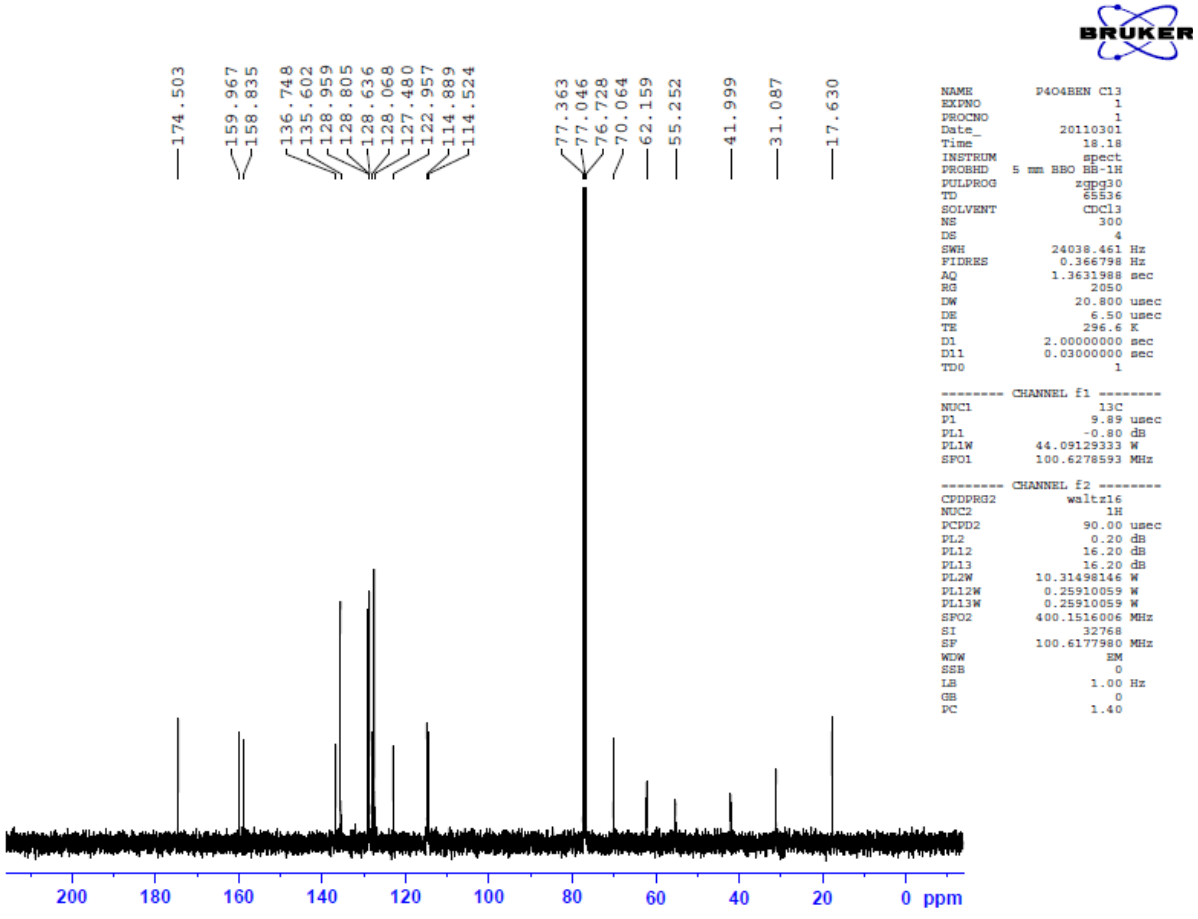
1.06
2.05
2.12
1.00
2.00
3.12
0.99
1.05
1.13
1.14
2.14
0.52


174.503
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136.748
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128.805
128.636
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127.480
122.957
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77.046
76.728
70.064
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55.252
41.999
31.087
17.630
NAME P4O4BEN C13
EXPNO 1
PROCNO 1
Date_ 20110301
Time 18.18
INSTRUM spect
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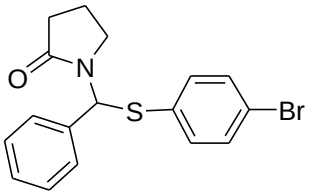
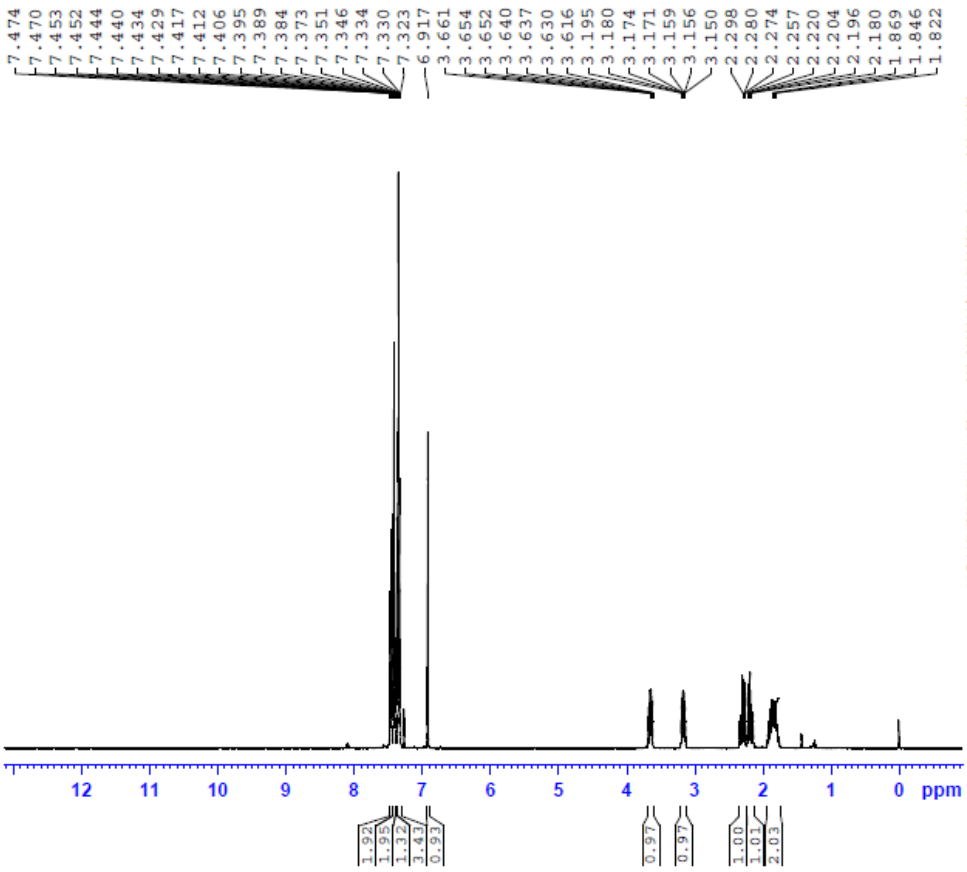
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.89 usec
PL1 -0.80 dB
PL1W 44.09129333 W
SFO1 100.6278593 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.20 dB
PL12 16.20 dB
PL13 16.20 dB
PL2W 10.31498146 W
PL12W 0.25910059 W
PL13W 0.25910059 W
SFO2 400.1516006 MHz
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



1-((4-bromophenylthio)(phenyl)methyl)pyrrolidin-2-one (4m): White Crystal; mp 100-101 ^{o}C; R_f 0.49 (60% ethyl acetate : hexanes); FTIR (KBr) : 2937, 2877, 1688, 1457, 1226, 1076, 821 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.82-1.86 (m, CH_2, 2 H), 2.18-2.22 (m, CH_2, 2 H), 2.25-2.29 (m, CH_2, 2 H), 3.15-3.19 (m, CH, 1 H), 3.61-3.66 (m, CH, 1 H), 6.91 (s, CH, 1 H), 7.32-7.47 (m, ArH, 9 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.6, 31.0, 42.2, 61.4, 122.0, 127.4, 128.7, 128.8, 131.9, 132.1, 133.6, 135.9, 174.8 ppm; HRMS: calculated for $C_{17}H_{16}BrNOS$ ($[M]^+$) 361.0136, Found 361.0137.

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PROCNO                   1
Date_             20110301
Time                 14.11
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PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               8012.820 Hz
FIDRES               0.244532 Hz
AQ                2.0447731 sec
RG                     228
DW                  62.400 usec
DE                   6.50 usec
TE                  296.6 K
D1                2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.20 usec
PL1                   0.20 dB
PL1W           10.31498146 W
SFO1          400.1526810 MHz
SI                   16384
SF            400.1500078 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
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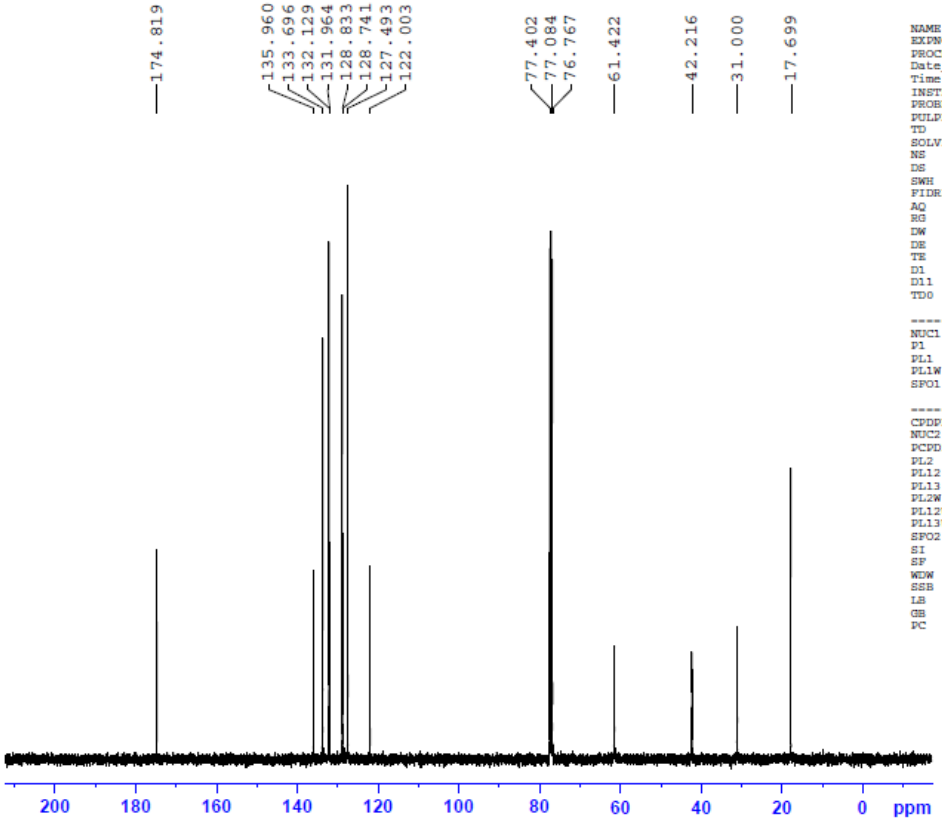


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NAME          P4BPAR C13
EXPNO                  1
PROCNO                 1
Date_           20110301
Time               14.38
INSTRUM            spect
PROBHD 5 mm BBO BB-1H
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 400
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                2050
DW               20.800 usec
DE                6.50 usec
TE               296.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.89 usec
PL1                -0.80 dB
PL1W        44.09129333 W
SFO1       100.6278593 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            90.00 usec
PL2                0.20 dB
PL12              16.20 dB
PL13              16.20 dB
PL2W        10.31498146 W
PL12W        0.25910059 W
PL13W        0.25910059 W
SFO2       400.1516006 MHz
SI                32768
SF         100.6177980 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
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PC                 1.40
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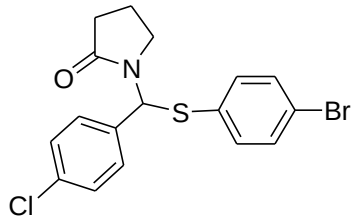
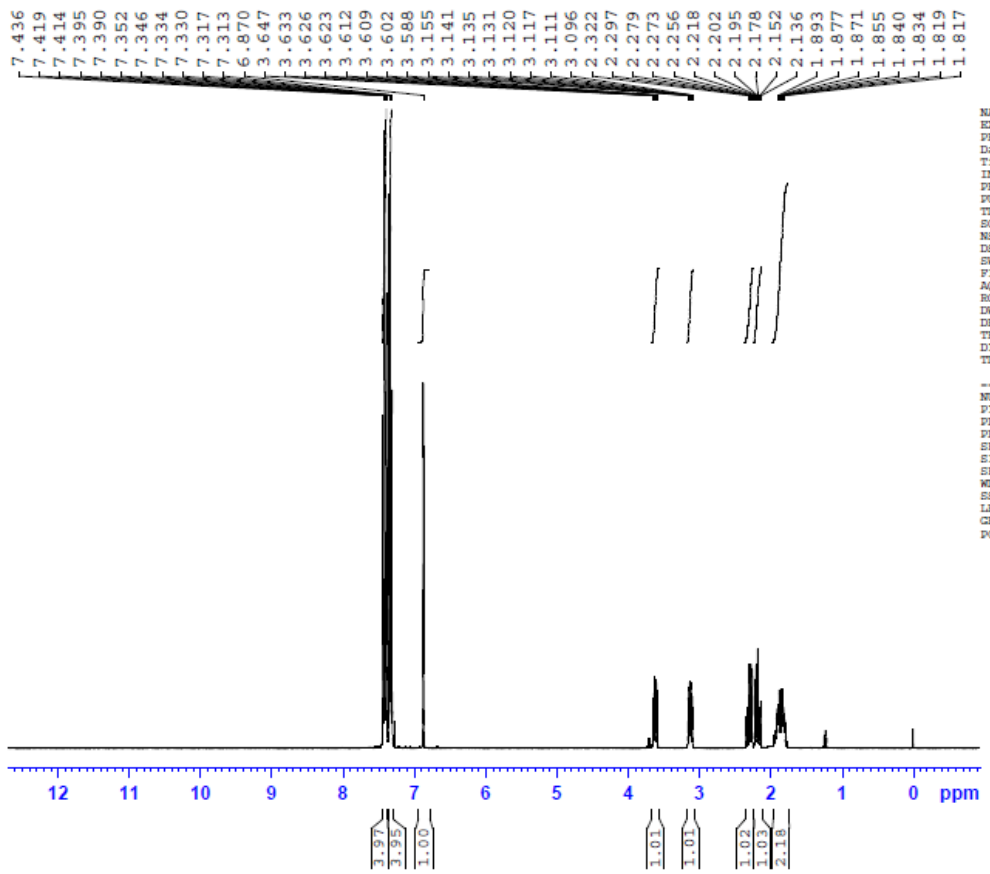


1-((4-bromophenylthio)(4-chlorophenyl)methyl)pyrrolidin-2-one 4n: White Solid; mp 119-120 ^{o}C; R_f 0.56 (60% ethyl acetate : hexanes); FTIR (KBr) : 2962, 2879, 1684, 1478, 1404, 1184, 1080, 817 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.78-1.93 (m, CH_2, 2 H), 2.13-2.33 (m, CH, 1 H), 3.09-3.14 (m, CH, 1 H), 3.58-3.64 (m, CH, 1 H), 6.87 (s, CH, 1 H), 7.31-7.43 (m, ArH, 8 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.7, 30.9, 42,1, 60.7, 122.2, 128.8, 129.0, 131.5, 132.2, 133.7, 134.5, 134.6, 174.8 ppm; HRMS: calculated for $C_{17}H_{15}BrClNOS$ ([M] $^{+}$) 394.9746, Found 394.9747



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TD	32768
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FIDRES	0.244532 Hz
AQ	2.0447731 sec
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DW	62.400 usec
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TE	296.5 K
D1	2.00000000 sec
TD0	1
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P1	14.20 usec
PL1	0.20 dB
PL1W	10.31498146 W
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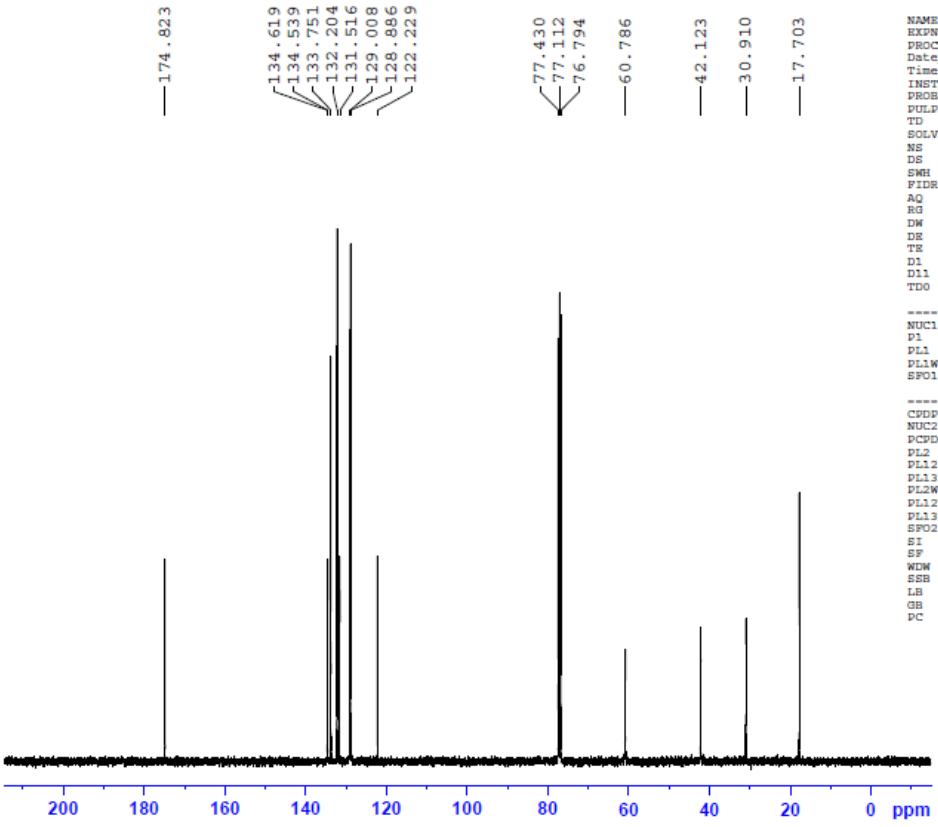
SI	16384
SF	400.1500030 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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EXPNO	1
PROCNO	1
Date_	20110301
Time	15.42
INSTRUM	spect
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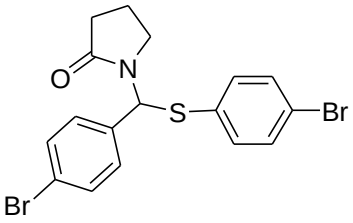
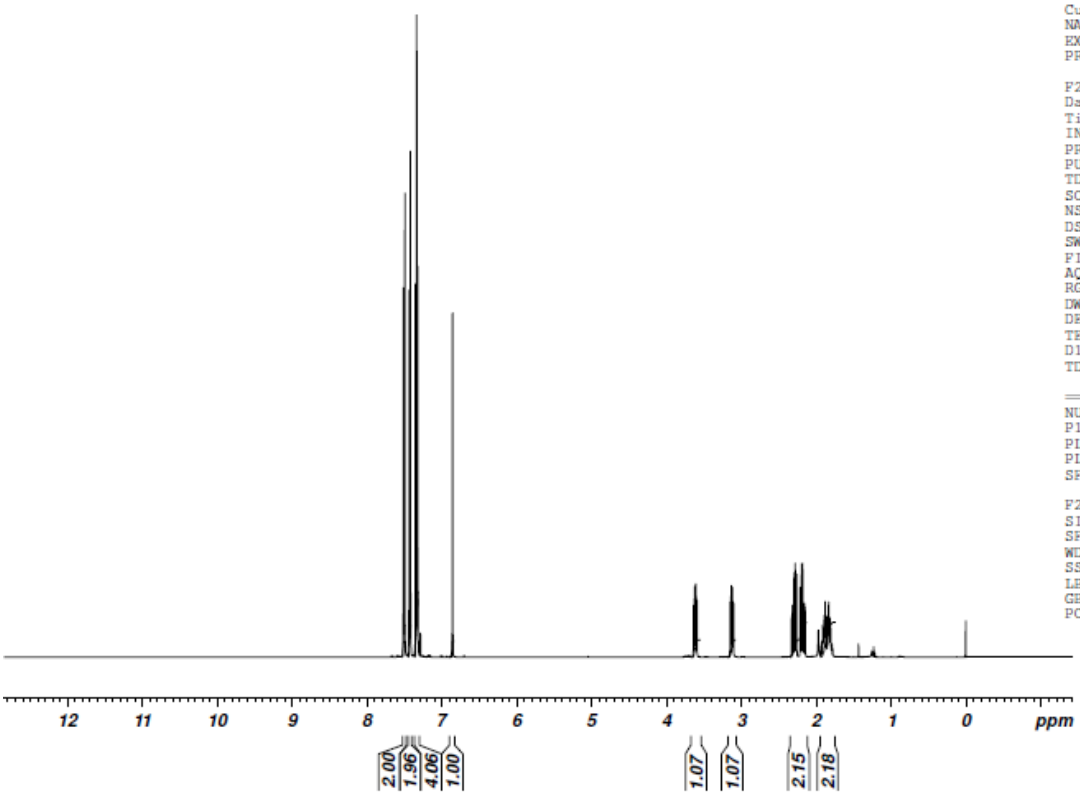
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.89 usec
PL1	-0.80 dB
PL1W	44.09129333 W
SFO1	100.6278593 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.20 dB
PL12	16.20 dB
PL13	16.20 dB
PL2W	10.31498146 W
PL12W	0.25910059 W
PL13W	0.25910059 W
SFO2	400.1516006 MHz
SI	32768
SF	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



1-((4-bromophenylthio)(2-bromophenyl)methyl)pyrrolidin-2-one (4o): White Solid; mp 121-122 ^{o}C; R_f 0.42 (60% ethyl acetate : hexanes); FTIR (KBr) : 2952, 2901, 1685, 1546, 1490, 1111, 1034, 859 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.84-1.91 (m, CH_2, 2 H), 2.14-2.31 (m, CH, 2 H), 3.10-3.15 (m, CH, 1 H), 3.59-3.63 (m, CH, 1 H), 6.85 (s, CH, 1 H), 7.31-7.51 (m, ArH, 8 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.71, 30.89, 42.12, 60.89, 122.23, 122.78, 129.18, 131.50, 131.97, 132.20, 133.74, 135.08, 174.79 ppm; HRMS: calculated for $C_{17}H_{15}Br_2NOS$ ([M]$^{+}$) 438.9241, Found 438.9240.

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PROCNO                         1

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PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           32
DS                            2
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AQ                    1.5860212 sec
RG                           57
DW                       48.400 usec
DE                        6.50 usec
TE                       298.0 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
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PL1                        0.00 dB
PL1W                23.53637505 W
SFO1                500.1330885 MHz

F2 - Processing parameters
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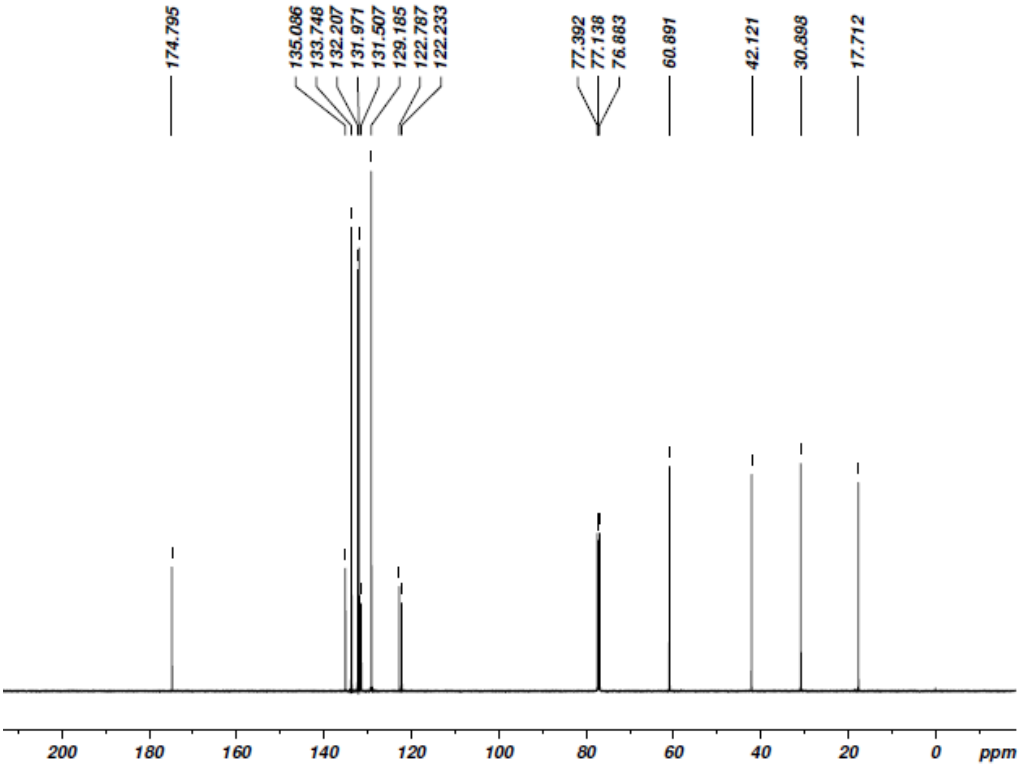


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PROCNO                         1

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PULPROG                  zgpg30
TD                        32768
SOLVENT                   CDCl3
NS                         1024
DS                            4
SWH                   29761.904 Hz
FIDRES                0.908261 Hz
AQ                    0.5505524 sec
RG                          203
DW                       16.800 usec
DE                        6.50 usec
TE                       299.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         7.80 usec
PL1                        0.00 dB
PL1W                70.83519745 W
SFO1                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        0.00 dB
PL12                      17.50 dB
PL13                      17.50 dB
PL2W                23.53637505 W
PL12W                0.41854253 W
PL13W                0.41854253 W
SFO2                500.1320005 MHz

F2 - Processing parameters
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SF                  125.7577890 MHz
WDW                          EM
SSB                           0
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PC                         1.40
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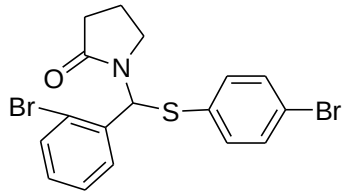
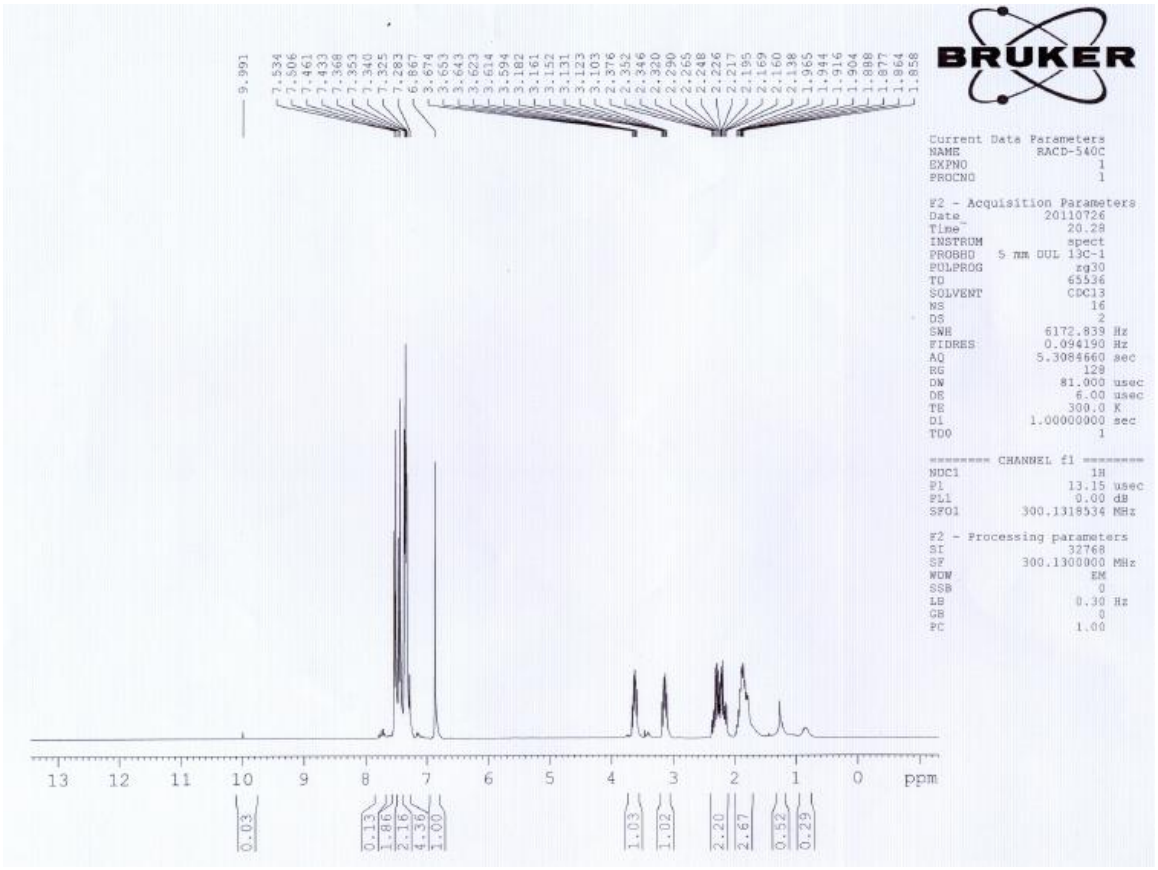
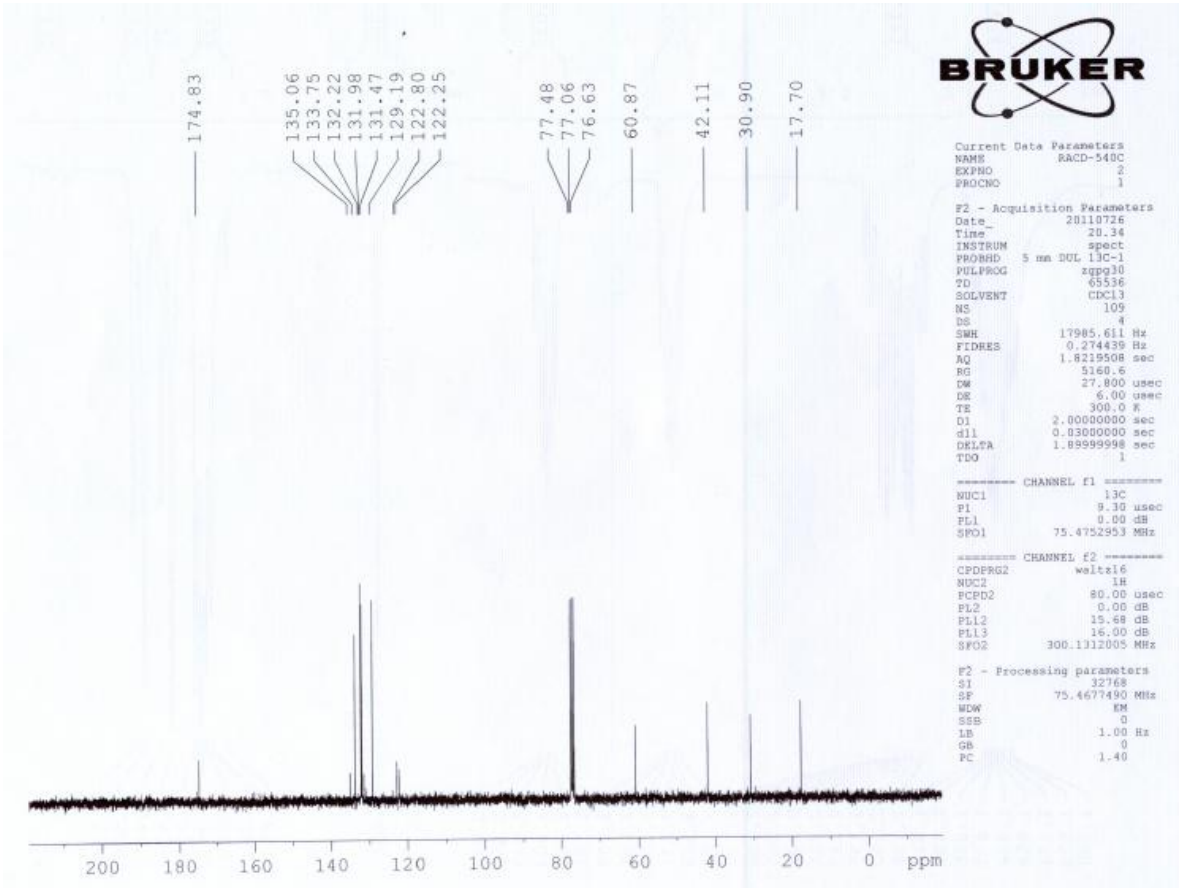


1-((4-bromophenylthio)(4-bromophenyl)methyl)pyrrolidin-2-one (4p): White Solid; mp 131-132 ^{o}C; R_f 0.41 (60% ethyl acetate : hexanes); FTIR (KBr) : 2966, 2879, 1685, 1552, 1475, 1184, 1086, 816 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): 1.85-1.96 (m, CH_2, 2 H), 2.13-2.37 (m, CH, 2 H), 3.10-3.18 (m, CH, 1 H), 3.59-3.67 (m, CH, 1 H), 6.86 (s, CH, 1 H), 7.28-7.53 (m, ArH, 8 H) ppm; ^{13}C NMR (100MHz, $CDCl_3$): 17.7, 30.9, 42.1, 60.8, 122.2, 122.8, 129.1, 131.4, 131.9, 132.2, 133.7, 135.0, 174.8 ppm; HRMS: calculated for $C_{17}H_{15}Br_2NOS$ ([M] $^{+}$) 438.9241, Found 438.9241.





LC-Ms spectra of N-acyliminium intermediate from 2-Flurobenzaldehyde

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    Last changed    : 3/22/2012 6:15:30 PM by sandeep
                      (modified after loading)
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    Last changed    : 3/22/2012 6:42:24 PM by narayan
    Method Info     : acidic


    Sample-related custom fields:

    Name                           |Value
    -------------------------------|--------------------------------------
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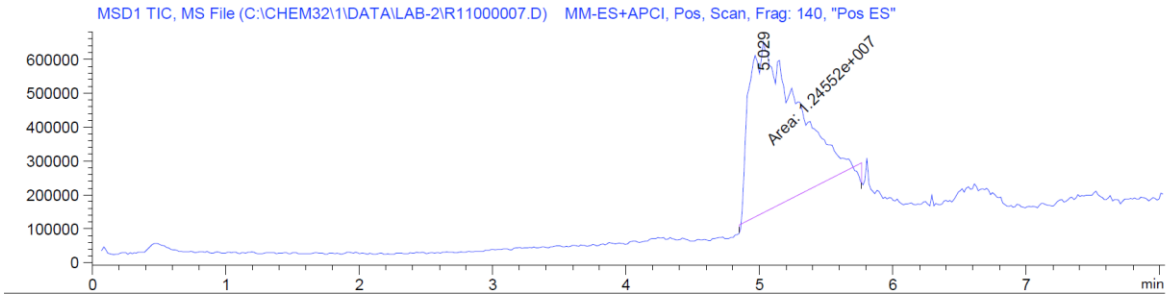


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    Name                           |Value
    -------------------------------|--------------------------------------
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    ======================================================================

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        Spectra averaged over upper half of peaks.
        Noise Cutoff: 500 counts.
        Reportable Ion Abundance: > 10%.


    Retention                    Mol. Weight
    Time (MS)      MS Area       or Ion

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                                  148.90 I
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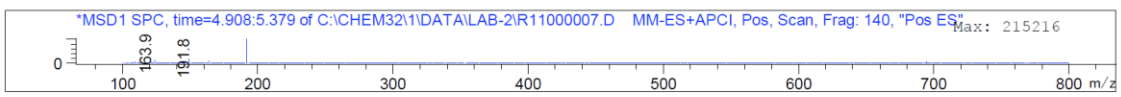


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*** End of Report ***

Instrument 1 3/22/2012 6:58:56 PM narayan                    Page   1 of   1
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