Supporting Information

Bis(chalcogenones) as pincer ligands: Isolation and Heck activity of the selone-ligated unsymmetrical C,C,Se-Pd pincer complex

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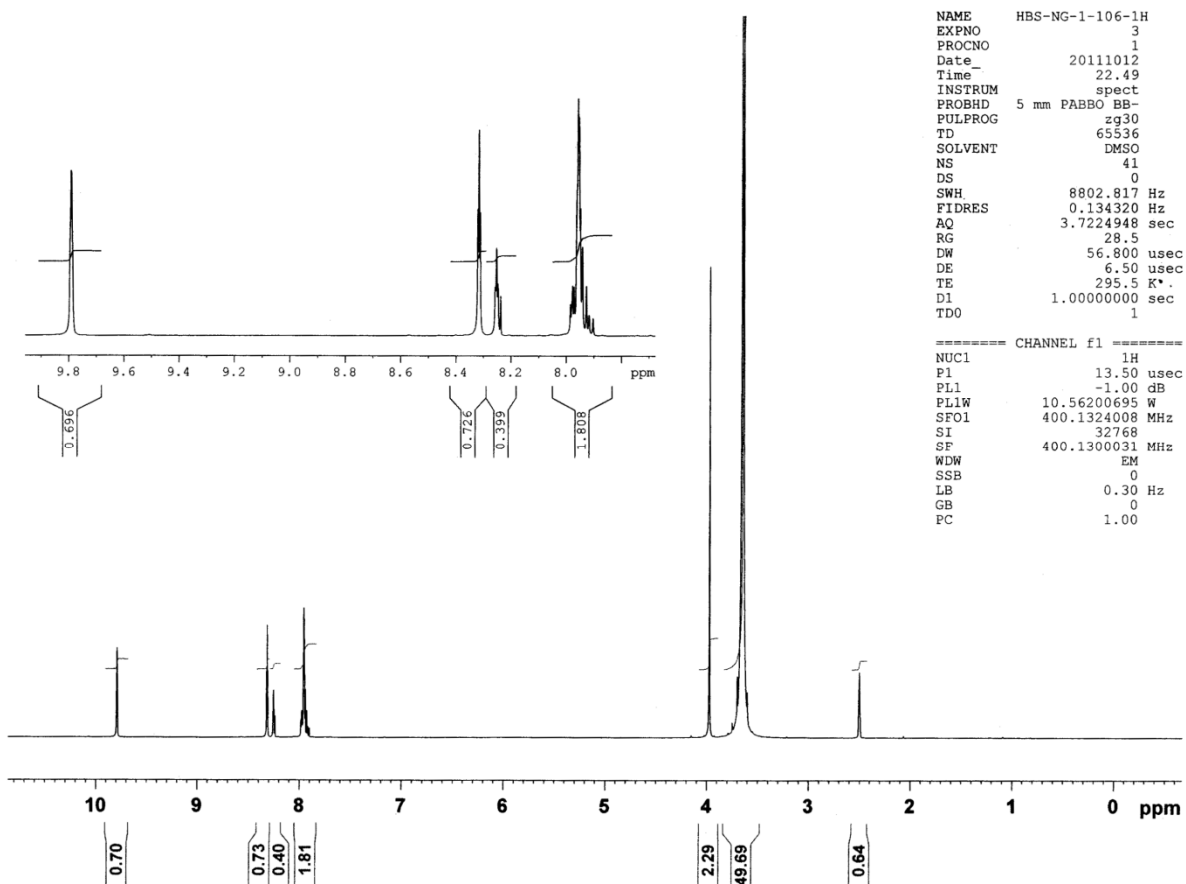[c]*Department of Chemistry, Howard University, Washington, D. C. 20059, United States*

Figure 1. ^{1}H NMR spectrum of **1**.

HBS-NG-1-106-13C

NAME   HBS-NG-1-106-13C
EXPNO   12
PROCNO   1
Date_   20111013
Time   0.09
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgpg30
TD   65536
SOLVENT   DMSO
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   295.7 K
D1   2.00000000 sec
D11   0.03000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   13C
P1   8.75 usec
PL1   -2.00 dB
PL1W   56.53121948 W
SFO1   100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   -1.00 dB
PL12   14.50 dB
PL13   14.50 dB
PL2W   10.56200695 W
PL12W   0.29767781 W
PL13W   0.29767781 W
SFO2   400.1316005 MHz
SI   32768
SF   100.6127974 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40
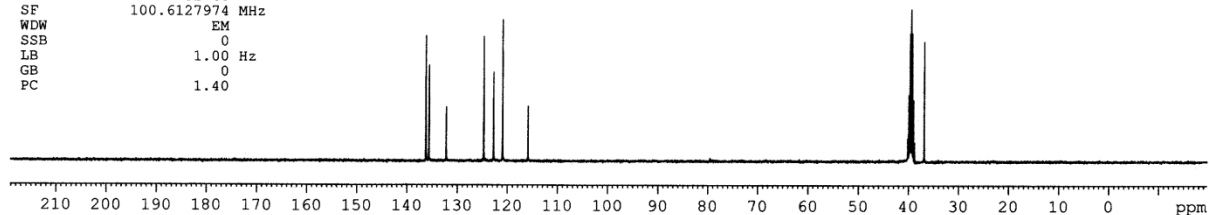
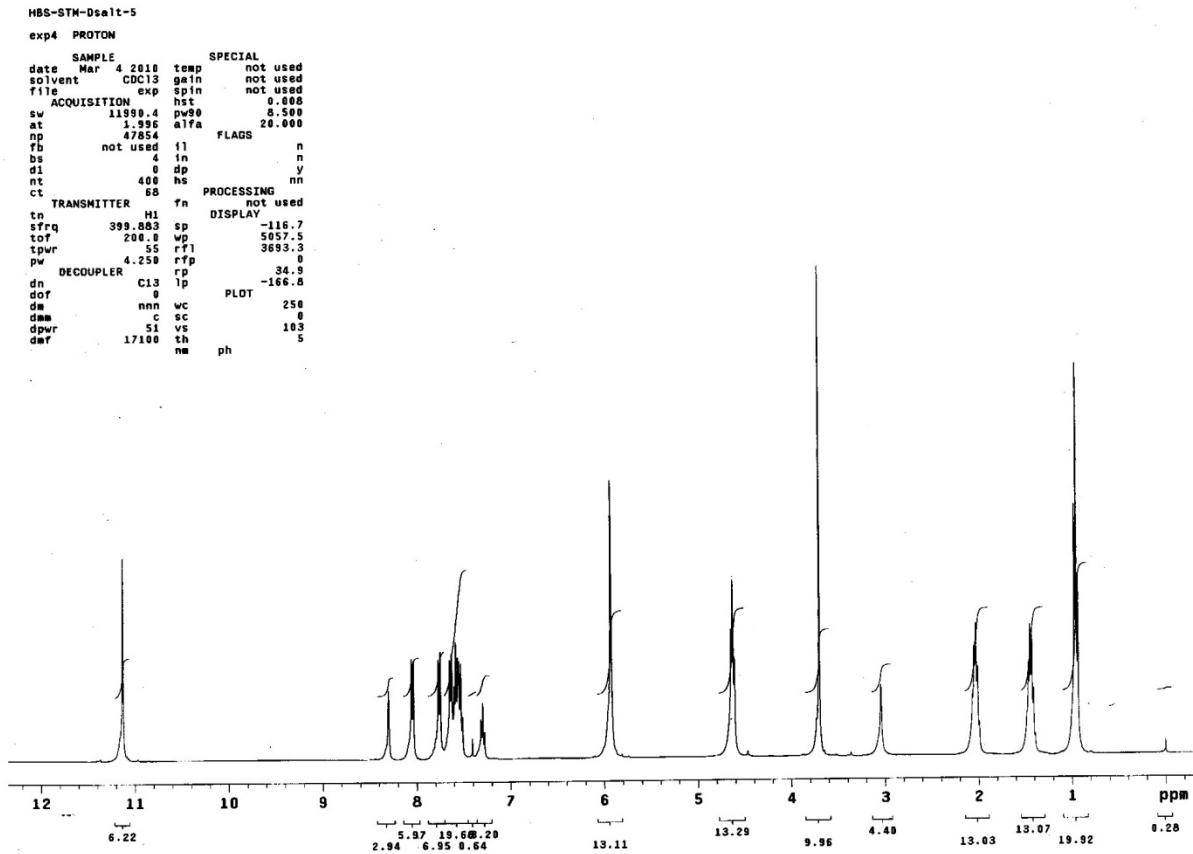Figure 2. ^{13}C NMR spectrum of **1**.

HBS-STM-Dsalt-5

exp4 PROTON

         SAMPLE   SPECIAL
date   Mar 4 2010   temp   not used
solvent   CDC13   gain   not used
file   exp   spin   not used
     ACQUISITION   hst   0.008
sw   11990.4   pw90   8.500
at   1.996   alfa   20.000
np   47854   FLAGS
fb   not used   il   n
bs   4   in   n
d1   0   dp   y
nt   400   hs   nn
ct   68   PROCESSING
    TRANSMITTER   fn   not used
tn   H1   DISPLAY
sfrq   399.883   sp   -116.7
tof   200.0   wp   5057.5
tpwr   55   rfl   3693.3
pw   4.250   rfp   0
    DECOUPLER   rp   34.9
dn   C13   lp   -166.8
dof   0   PLOT
dm   nnn   wc   250
dmm   c   sc   0
dpwr   51   vs   103
dmf   17100   th   5
                    nm   ph

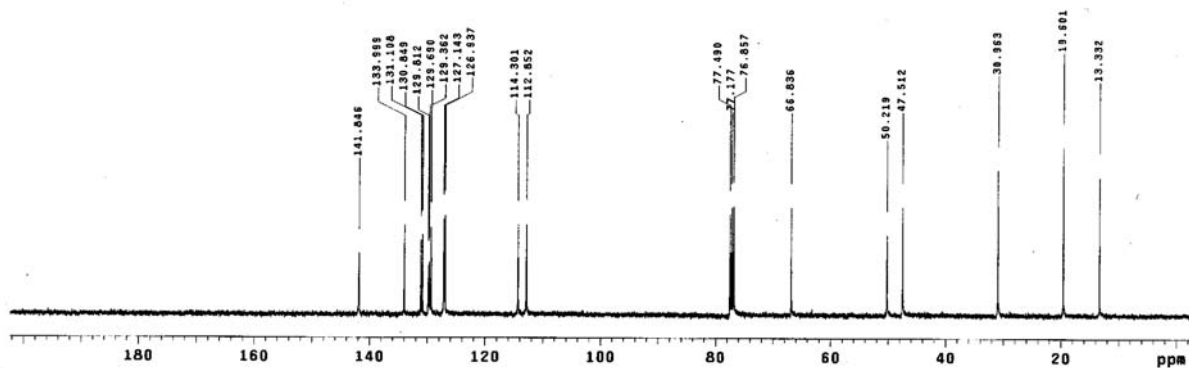Figure 3. ^{1}H NMR spectrum of **2**.

Figure 4. ^{13}C NMR spectrum of **2**.

Eager 300 Report

Page: 1   Sample: STM147 (STM147)

```
Method Name    : SP310310
Method File    : D:\CHNS2008\SP310310.mth
Chromatogram   : STM147
Operator ID    : SP                Company  Name : C.E. Instruments
Analysed       : 03/31/2010  12:09  Printed       : 3/31/2010  15:46
Sample ID      : STM147 (# 7)       Instrument N. : Instrument #1
Analysis Type  : UnkNown (Area)     Sample weight : .738

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.
```

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	8.2429	43	76688	RS	14.851930	.126064E+07
Carbon	57.3574	66	1138965	RS	1.000000	.268567E+07
Hydrogen	6.0422	169	357826	RS	3.183013	.691717E+07
Totals	71.6425		1573479			

Figure 5. Elemental analysis of **2**.

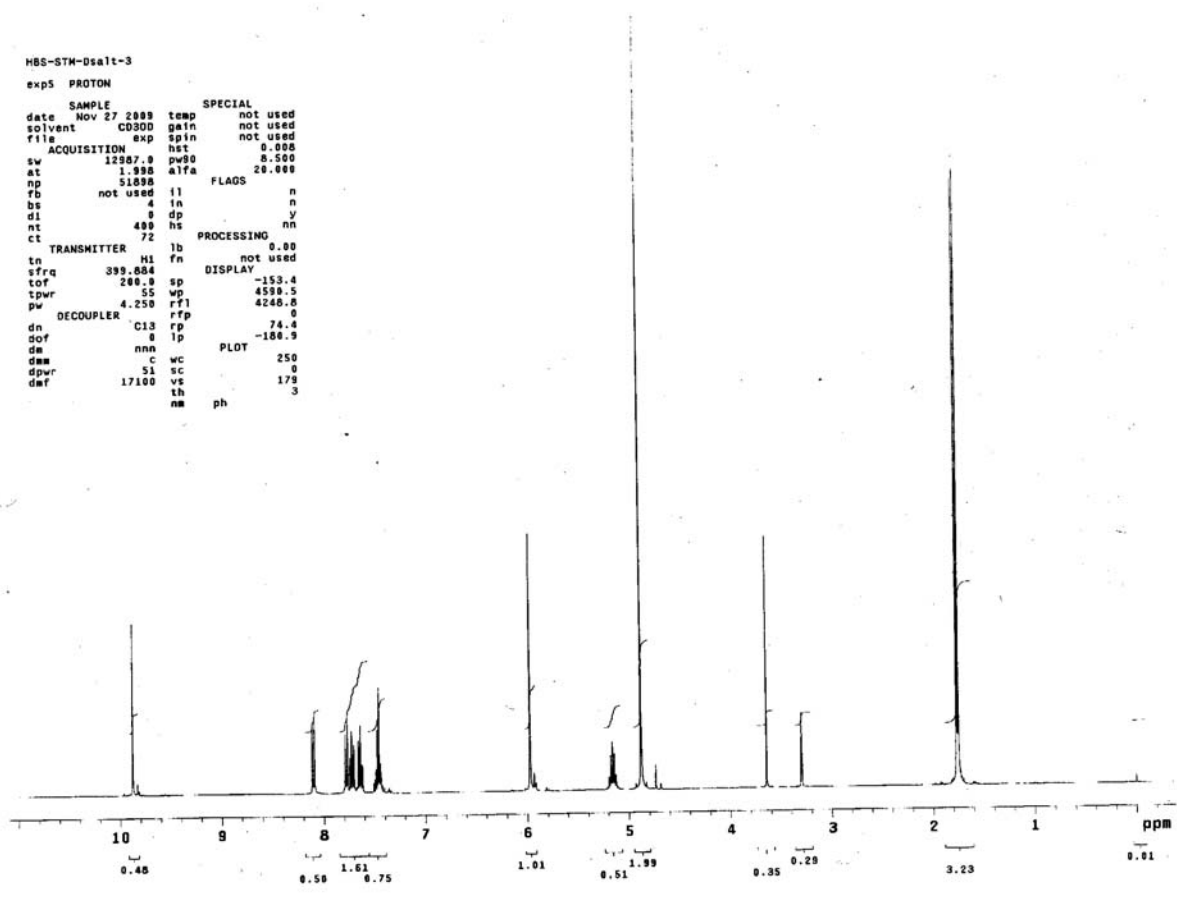Figure 6. ^{1}H NMR spectrum of **3**.

HBS-STM-Dsalt-3

exp4 CARBON

SAMPLE   SPECIAL
date   Nov 28 2009   temp   not used
solvent   CD3OD   gain   not used
file /export/home/~   spin   not used
hbs1/2009/NOV/HBS-~   hst   0.008
STM-Dsalt-3-13C.fi~   pw90   14.000
                 d   alfa   20.080
   ACQUISITION   FLAGS
sw   25125.6   ll   n
at   1.199   in   n
np   60270   dp   y
fb   13800   hs   nn
bs   4   PROCESSING
d1   1.000   lb   1.00
nt   40000   fn   not used
ct   1236   DISPLAY
   TRANSMITTER   -192.9
tn   C13   wp   18587.9
sfrq   100.561   rfl   6152.8
tof   1554.3   rfp   4937.0
tpwr   56   rp   -154.3
pw   7.000   lp   -321.0
   DECOUPLER   PLOT
dn   H1   wc   250
dof   -713.0   sc   0
dm   yyy   vs   80
dmm   w   th   4
dpwr   41   nm   ph
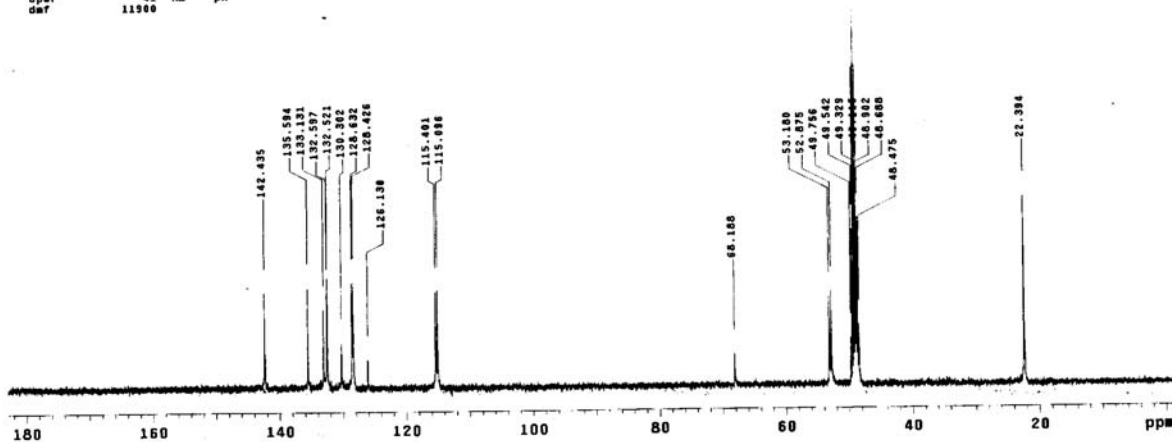dmf   11900

Figure 7. ^{13}C NMR spectrum of **3**.

Eager 300 Report

Page: 1   Sample: STM61 (STM61)

Method Name   : SP111209
Method File   : D:\CHNS2008\SP111209.mth
Chromatogram   : STM61
Operator ID   : SP   Company Name : C.E. Instruments
Analysed   : 12/11/2009 14:04   Printed   : 12/11/2009 16:55
Sample ID   : STM61 (# 23)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : .788

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
1	0.0000	18	10273	RS		0.0000
Nitrogen	8.9889	42	84533	RS	12.283290	.119342E+07
Carbon	49.6985	66	1038338	RS	1.000000	.265136E+07
Hydrogen	4.2008	172	220379	RS	4.711599	.616890E+07
Totals	62.8882		1353522			

Figure 8. Elemental analysis of **3**.
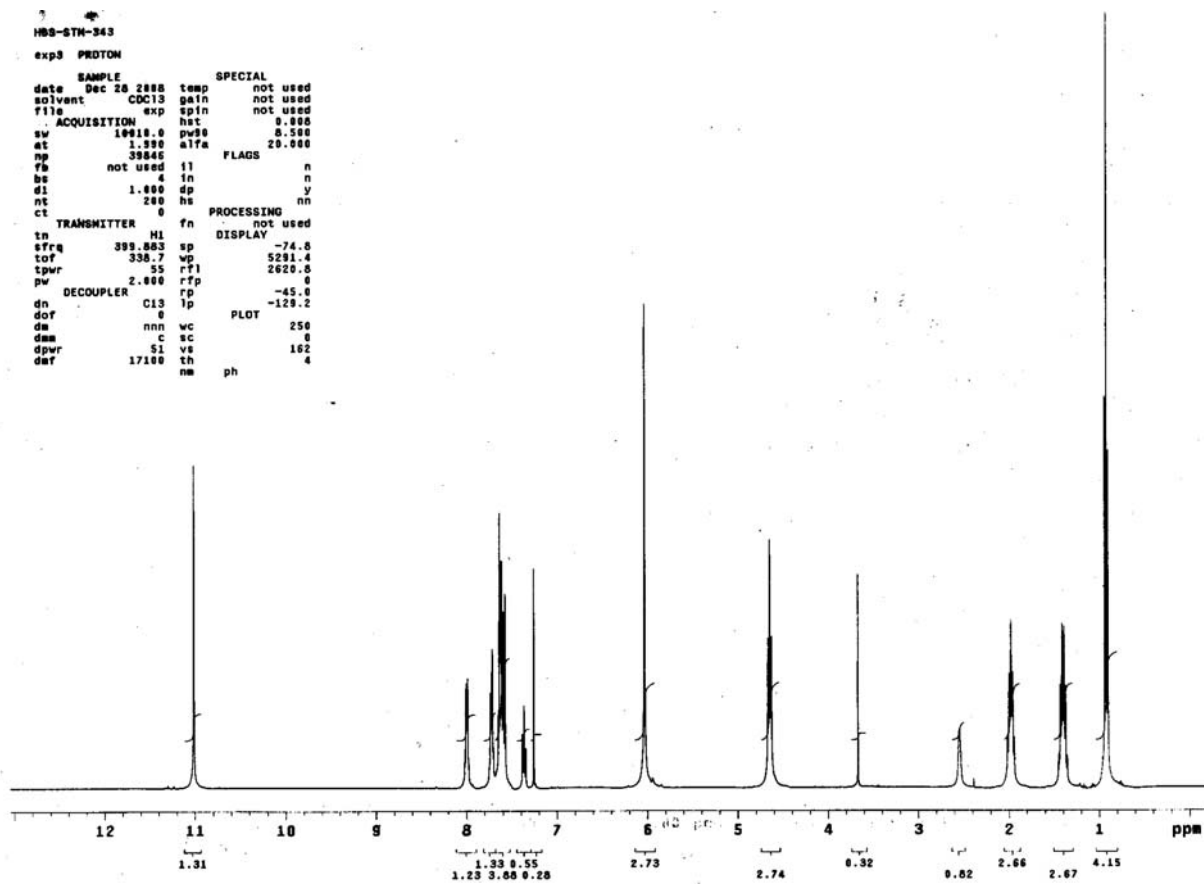
Figure 9. ^{1}H NMR spectrum of **4**.
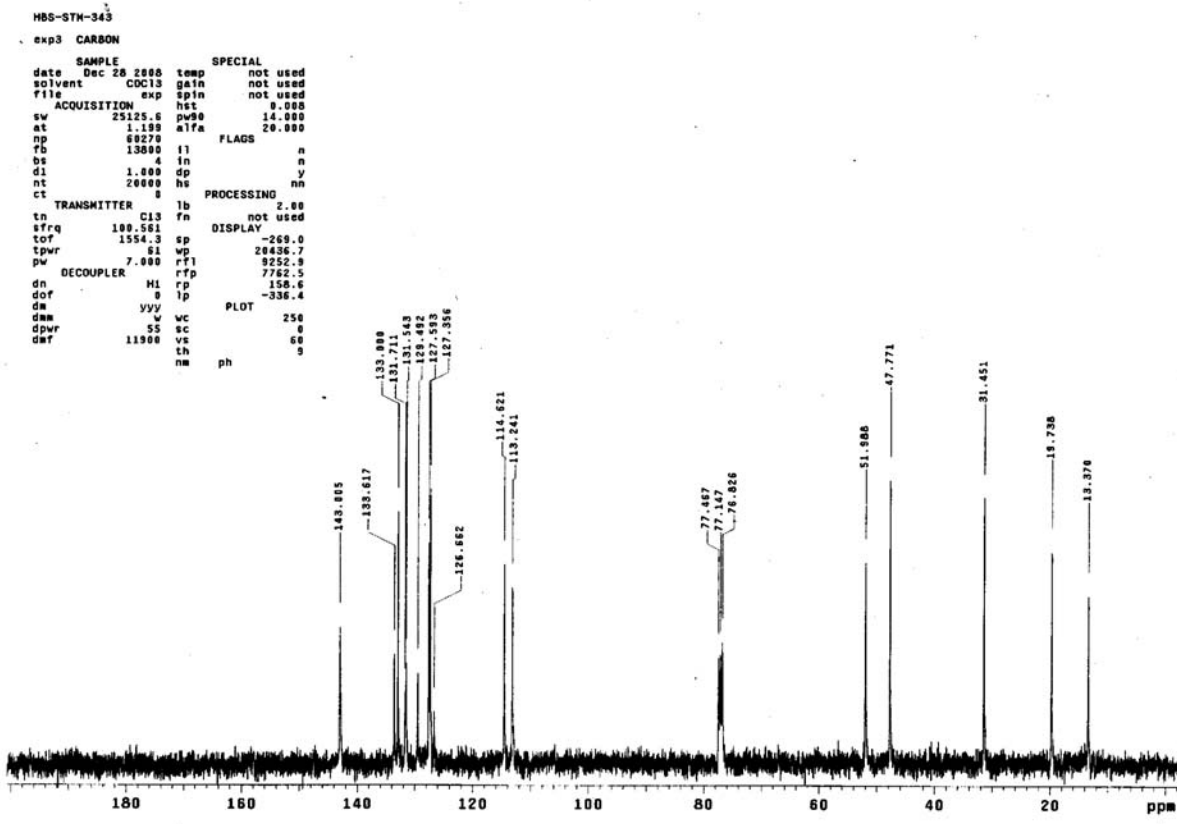
Figure 10. ^{13}C NMR spectrum of **4**.

Eager 300 Report

Page: 1   Sample: STM343 (STM343)

```
Method Name    : sp020109
Method File    : D:\CHNS2008\sp020109.mth
Chromatogram   : STM343
Operator ID    : SP              Company  Name : C.E. Instruments
Analysed       : 01/02/2009  14:27   Printed     : 1/5/2009  09:55
Sample ID      : STM343 (# 20)   Instrument N. : Instrument #1
Analysis Type  : UnkNown (Area)   Sample weight : 1.653

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.
```

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
1	0.0000	18	4015	RS		0.0000
Nitrogen	8.1561	43	141748	RS	15.919730	.105138E+07
Carbon	50.7601	65	2256582	RS	1.000000	.268556E+07
Hydrogen	4.4929	172	532476	RS	4.237904	.691806E+07
Totals	63.4092		2934821			

Figure 11. Elemental analysis of **4**.

exp4 PROTON

SAMPLE		SPECIAL	
date	Dec 21 2009	temp	not used
solvent	CD3OD	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	12987.0	pw90	8.500
at	1.998	alfa	20.000
np	51898	FLAGS	
fb	not used	il	n
bs	4	in	n
d1	0	dp	y
nt	400	hs	nn
ct	92	PROCESSING	
TRANSMITTER		lb	0.00
tn	H1	fn	not used
sfrq	399.884	DISPLAY	
tof	280.0	sp	-101.9
tpwr	55	wp	4573.4
pw	4.250	rfl	4248.8
DECOUPLER		rfp	0
dn	C13	rp	81.0
dof	0	lp	-178.5
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	51	sc	0
dmf	17100	vs	312
		th	4
		nm	ph

Figure 12. ^{1}H NMR spectrum of **5**.

HBS-STM-79

exp4 CARBON

            SAMPLE   SPECIAL
date   Dec 20 2009   temp   not used
solvent   CD3OD   gain   not used
file   exp   spin   not used
      ACQUISITION   hst   0.008
sw   25125.6   pw90   9.700
at   1.199   alfa   20.000
np   60270   FLAGS
fb   13800   il   n
bs   4   in   n
d1   1.000   dp   y
nt   64000   hs   nn
ct   804   PROCESSING
      TRANSMITTER   lb   2.00
tn   C13   fn   not used
sfrq   100.561   DISPLAY
tof   1554.3   sp   -171.4
tpwr   60   wp   20469.6
pw   4.850   rfl   6296.9
      DECOUPLER   rfp   4937.0
dn   H1   rp   168.3
dof   -713.0   lp   -273.1
dm   yyy   PLOT
dmm   w   wc   250
dpwr   39   sc   0
dmf   11900   vs   84
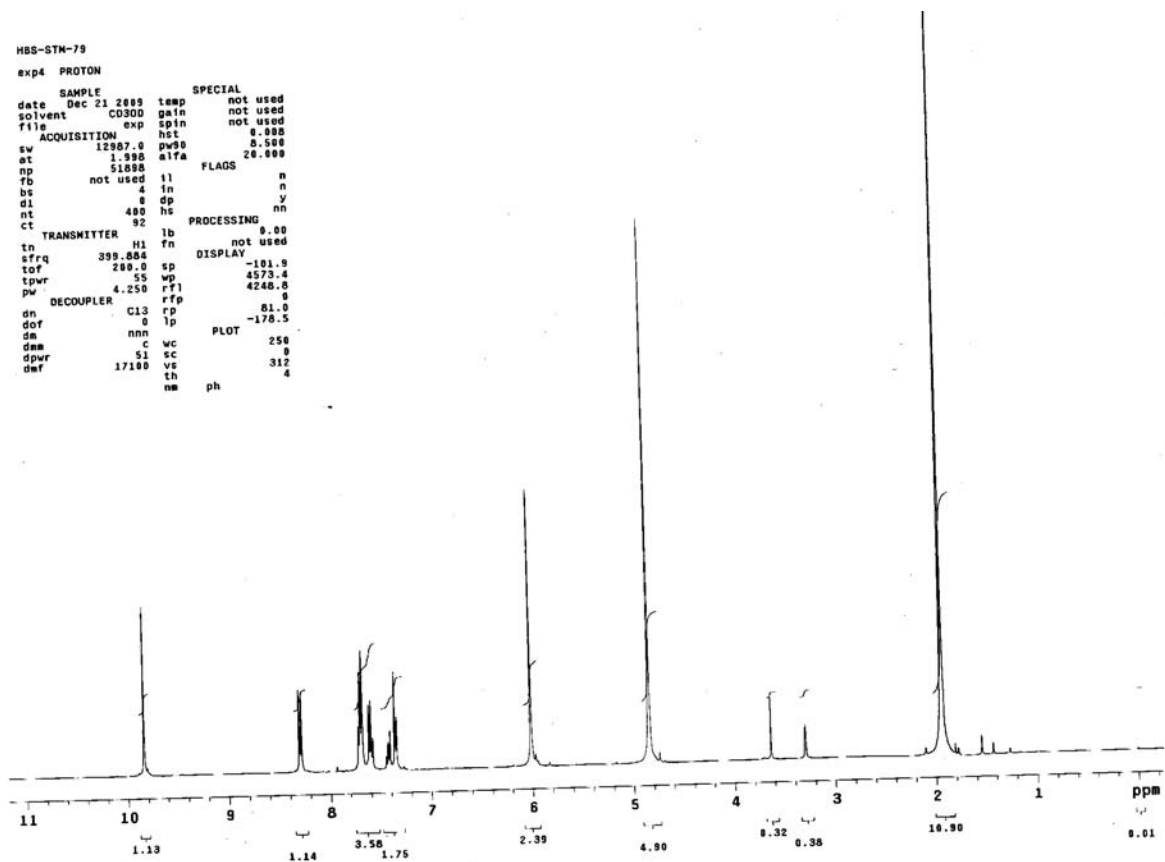   th   7
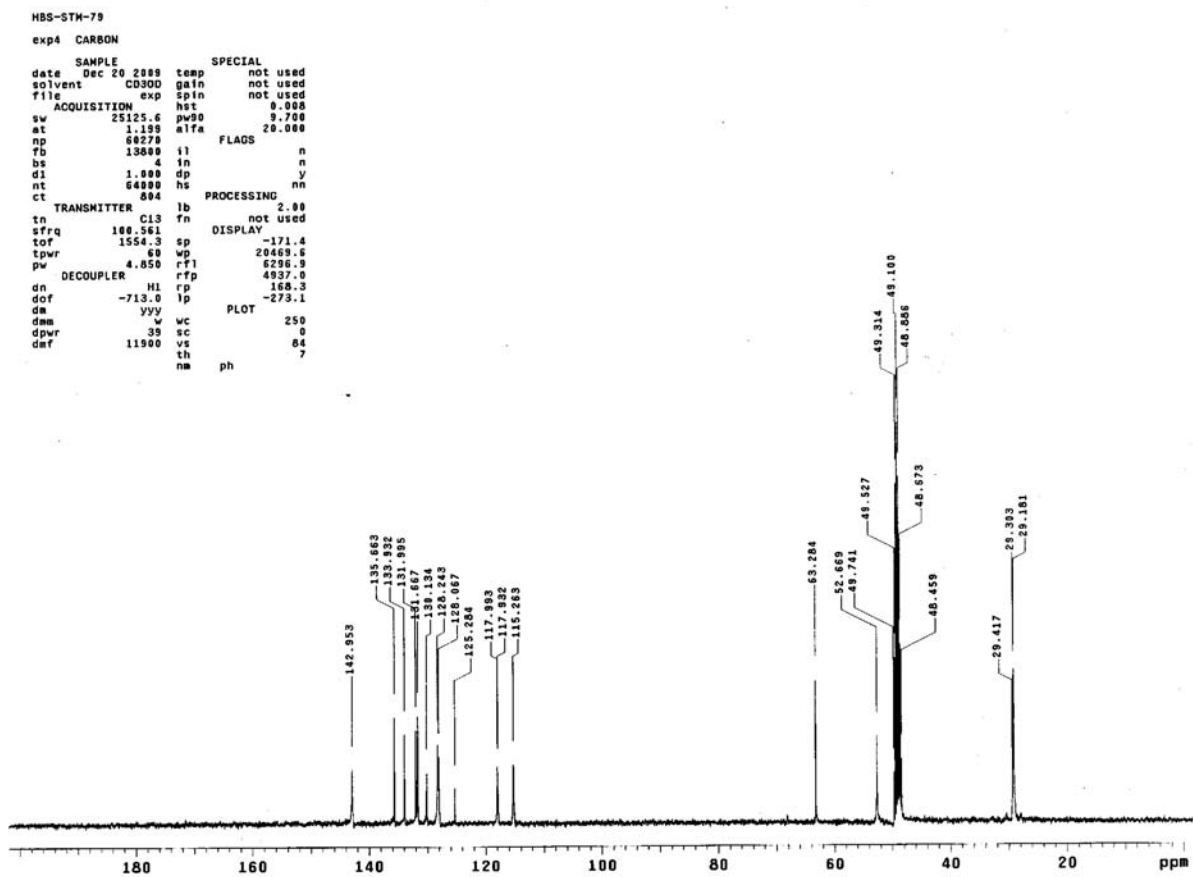   nm   ph

Figure 13. ^{13}C NMR spectrum of **5**.

Eager 300 Report

Page: 1   Sample: STM79 (STM79)

```
Method Name    : SP200110
Method File    : D:\CHNS2008\SP200110.mth
Chromatogram   : STM79
Operator ID    : SP              Company  Name : C.E. Instruments
Analysed       : 01/20/2010  15:09    Printed      : 1/29/2010  16:41
Sample ID      : STM79 (# 32)    Instrument N. : Instrument #1
Analysis Type  : UnkNown (Area)  Sample weight : .546
```

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	8.5459	44	56745	RS	12.988510	.121612E+07
Carbon	50.7847	67	737033	RS	1.000000	.265804E+07
Hydrogen	4.4613	175	165066	RS	4.465081	.612939E+07
Totals	63.7919		958844			

Figure 14. Elemental analysis of **5**.

HBS-STM-Dsalt-2

exp3 PROTON

         SAMPLE   SPECIAL
date   Nov 25 2009   temp   not used
solvent   CDCl3   gain   not used
file   exp   spin   not used
    ACQUISITION   hst   0.008
sw   10010.0   pw90   8.500
at   1.995   alfa   20.000
np   39942   FLAGS
fb   not used   il   n
bs   4   in   n
d1   0   dp   y
nt   400   hs   nn
ct   112   PROCESSING
    TRANSMITTER   lb   0.00
tn   H1   fn   not used
sfrq   399.883   DISPLAY
tof   200.0   sp   -47.5
tpwr   55   wp   4866.8
pw   4.250   rfl   5651.0
    DECOUPLER   rfp   999.7
dn   C13   rp   38.6
dof   0   lp   -105.1
dm   nnn   PLOT
dmm   c   wc   250
dpwr   51   sc   0
dmf   17100   vs   2169
                      th   13
                      nm   ph

Figure 15. ^{1}H NMR spectrum of **6**.

         SAMPLE   SPECIAL
date   Nov 29 2009   temp   not used
solvent   DMSO   gain   not used
file /export/home/~   spin   not used
aks2/2009/NOv/28th~   hst   0.008
/HBS-STM-Dsalt-2-1~   pw90   14.000
         3C.fid   alfa   20.000
      ACQUISITION   FLAGS
sw   25125.6   il   n
at   1.199   in   n
np   60270   dp   y
fb   13800   hs   nn
bs   4   PROCESSING
d1   1.000   lb   1.00
nt   48000   fn   not used
ct   11856   DISPLAY
     TRANSMITTER   sp   -209.6
tn   C13   wp   18620.9
sfrq   100.562   rfl   1563.0
tof   1554.3   rfp   0
tpwr   56   rp   -172.8
pw   7.000   lp   -329.8
      DECOUPLER   PLOT
dn   H1   wc   250
dof   -713.0   sc   0
dm   yyy   vs   55
dmm   w   th   3
dpwr   41   ai   ph
dmf   11900

Figure 16. ^{13}C NMR spectrum of **6**.

Eager 300 Report

Page: 1   Sample: STM55 (STM55)

Method Name   : SP111209
Method File   : D:\CHNS2008\SP111209.mth
Chromatogram : STM55
Operator ID   : SP   Company Name : C.E. Instruments
Analysed   : 12/11/2009 14:14   Printed   : 12/11/2009 16:55
Sample ID   : STM55 (# 24)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : .679

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
1	0.0000	18	10223	RS		0.0000
Nitrogen	12.5421	42	101632	RS	9.169681	.119342E+07
Carbon	51.7662	66	931933	RS	1.000000	.265136E+07
Hydrogen	2.9200	175	138485	RS	6.729487	.616890E+07
Totals	67.2283		1182273			

Figure 17. Elemental analysis of **6**.
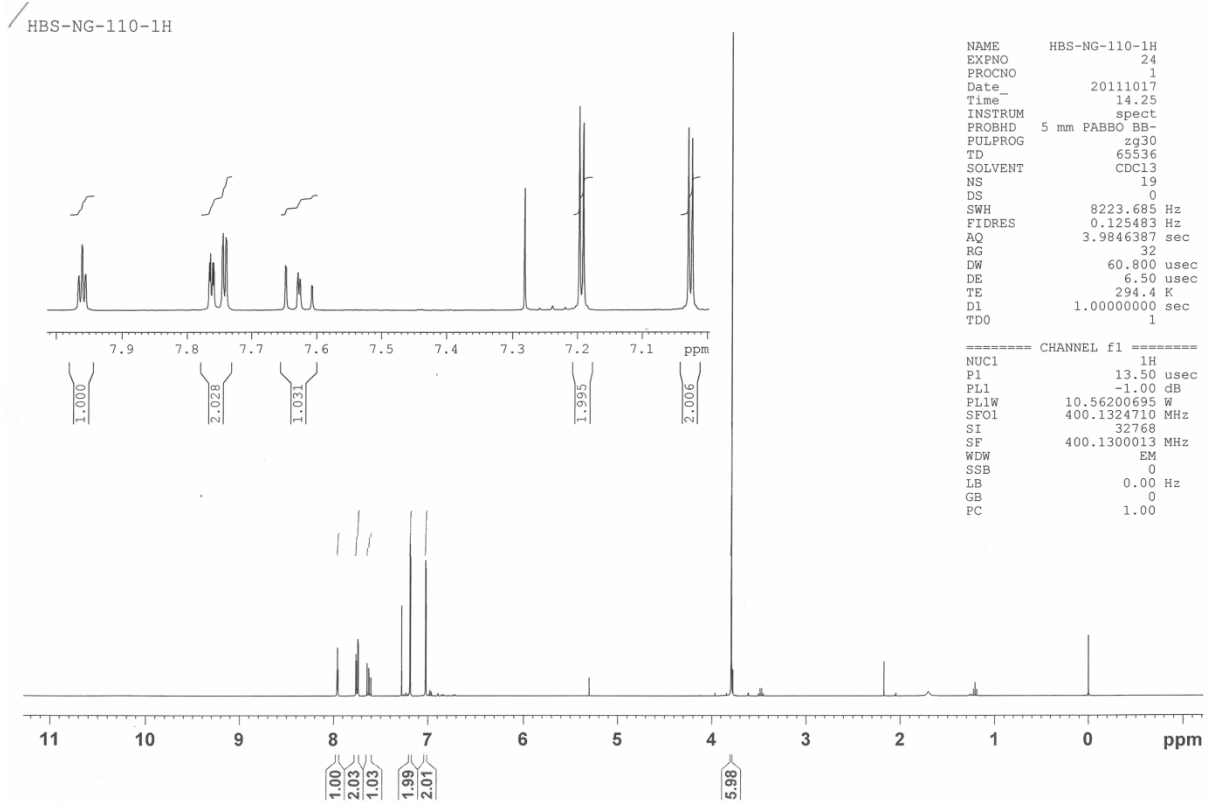
Figure 18. ^{1}H NMR spectrum of **7** in CDCl$_3$.

HBS-NG-1-110-13C

NAME	HBS-NG-1-110-13C
EXPNO	6
PROCNO	1
Date_	20111023
Time	14.27
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	44
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	295.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	8.75 usec
PL1	-2.00 dB
PL1W	56.53121948 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.50 dB
PL13	14.50 dB
PL2W	10.56200695 W
PL12W	0.29767781 W
PL13W	0.29767781 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127655 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

Figure 19. ^{13}C NMR spectrum of **7** in CDCl$_3$.

HBS-NG-1-110-77Se

32.233

32.233
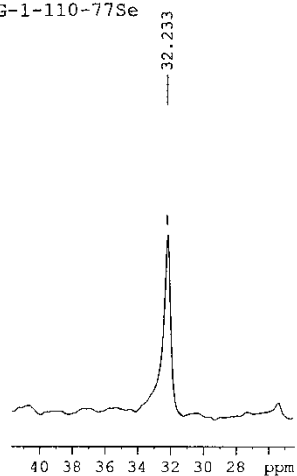
NAME   HBS-NG-1-110-77Se
EXPNC   7
PROCNO   1
Date_   20111023
Time   14.36
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zg
TD   65536
SOLVENT   CDCl3
NS   111
DS   4
SWH   300000.000 Hz
FIDRES   4.577637 Hz
AQ   0.1092767 sec
RG   228
DW   1.667 usec
DE   6.50 usec
TE   295.0 K
D1   1.00000000 sec
TDO   1

======== CHANNEL f1 ========
NUC1   77Se
P1   10.00 usec
PL1   0.00 dB
SFO1   76.3490004 MHz
SI   65536
SF   76.3110246 MHz
WDW   EM
SSB   0
LB   30.00 Hz
GB   0
PC   0.10

40 38 36 34 32 30 28   ppm

2000   1500   1000   500   0   -500   -1000   ppm
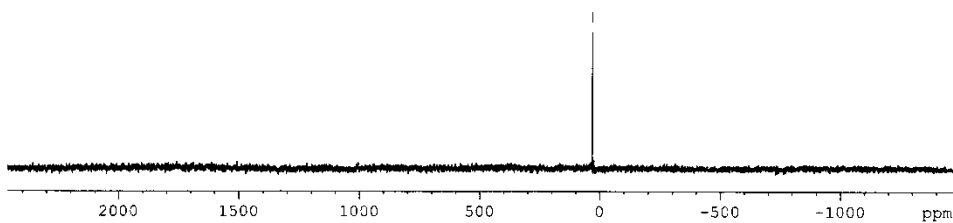
Figure 20. ^{77}Se NMR spectrum of **7** in CDCl$_3$.

S21

Eager 300 Report

Page: 1   Sample: NG-1-110 (NG-1-110)

```
Method Name    : SD111111
Method File    : D:\CHNS2011\SD111111.mth
Chromatogram   : NG-1-110
Operator ID    : SD                    Company  Name : C.E. Instruments
Analysed       : 11/11/2011  14:12     Printed       : 11/11/2011  17:13
Sample ID      : NG-1-110 (# 16)       Instrument N. : Instrument #1
Analysis Type  : UnkNown (Area)        Sample weight : .704

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.
```

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	14.6766	44	80465	RS	9.268458	.146976E+07
Carbon	42.4441	68	745787	RS	1.000000	.249589E+07
Hydrogen	3.5628	190	84980	RS	8.776024	.496282E+07
Totals	60.6835		911232			

Figure 21. Elemental analysis of **7**.

Single Mass Analysis
Tolerance = 10.0 PPM   /   DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0   Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
28 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Micromass : Q-Tof micro (YA-105)
C14H14N4Se2   Dept. Of Chemistry I.I.T.(B)   21-Sep-201111:18:18
HBS-NG-1-82 21 (0.207) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (Mn, 2x4.00); Sb (5,40.00); Cm (1:45)   TOF MS ES+
                                                                                                                  1.26e3
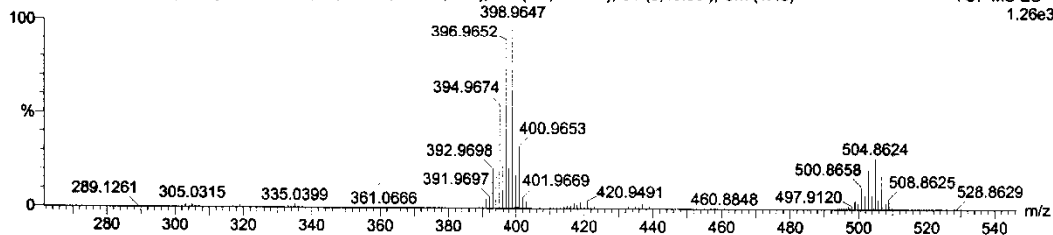
Minimum:				-1.5			
Maximum:		200.0	10.0	50.0			

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula	
398.9647	398.9627	1.9	4.9	11.5	1	C14 H15 N4 Se2	

Figure 22. Mass spectrum of **7**.

         SAMPLE   SPECIAL
date   Mar Z 2010   temp   not used
solvent   CDCL3   gain   not used
file   exp   spin   not used
     ACQUISITION   hst   8.008
sw   11896.4   pw90   8.500
at   1.996   alfa   20.000
np   47854   FLAGS
fb   not used   il   n
bs   4   in   n
d1   0   dp   y
nt   400   hs   nn
ct   80   PROCESSING
     TRANSMITTER   fn   not used
tn   H1   DISPLAY
sfrq   389.863   sp   -123.7
tof   200.0   wp   4222.5
tpwr   55   rfl   3747.5
pw   4.250   rfp   0
     DECOUPLER   rp   36.1
dn   C13   lp   -168.9
dof   8   PLOT
dm   nnn   wc   250
dmm   c   sc   0
dpwr   51   vs   74
dmf   17180   th   5
                       nm   ph
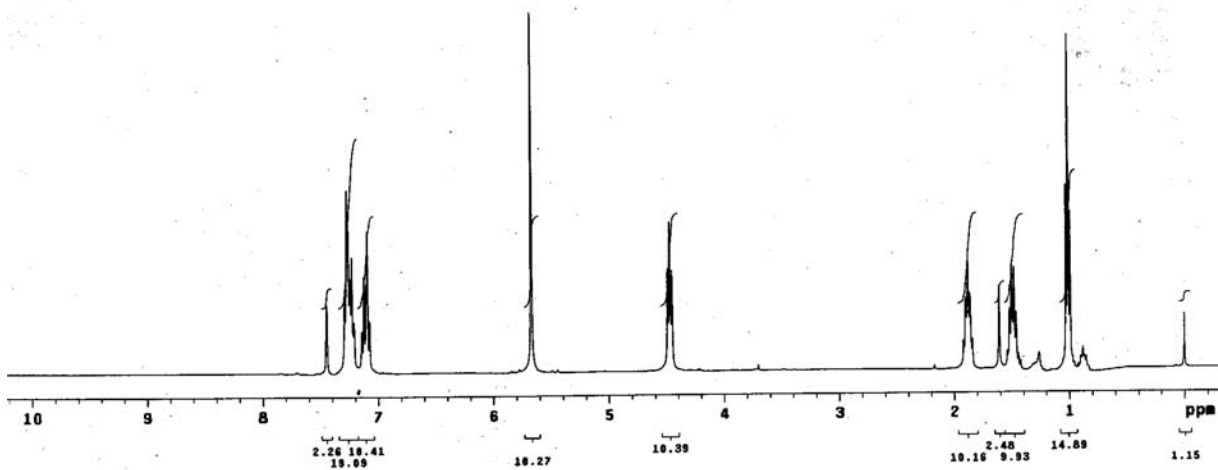
Figure 23. ^{1}H NMR spectrum of **8**.

Figure 24. ^{13}C NMR spectrum of **8**.

HBS-STN-2-148

expl s2pul

```
        SAMPLE              DEC. & VT
date    Mar 30 2010   dn              H1
solvent     CDC13     dof              0
file          exp     dm             nnn
    ACQUISITION       dmm              c
sfrq      57.219      dmf            200
tn          Se77      PROCESSING
at         0.640      lb          10.00
np        128000      fn        not used
sw       100000.0
fb         55000      werr
bs             8      wexp
pw           3.0      wbs
pw           3.0      wnt
tpwr          58      DISPLAY
d1             0      sp        -35753.1
tof      -1200.0      wp         59998.5
nt         32000      vs             22
ct           528      sc              0
alock          n      wc            250
gain           6      hzmm       378.80
    FLAGS             is       488281.25
il             n      rfl        35754.6
in             n      rfp              0
dp             y      th               8
                      ins         65.734
                      nm     ph
```
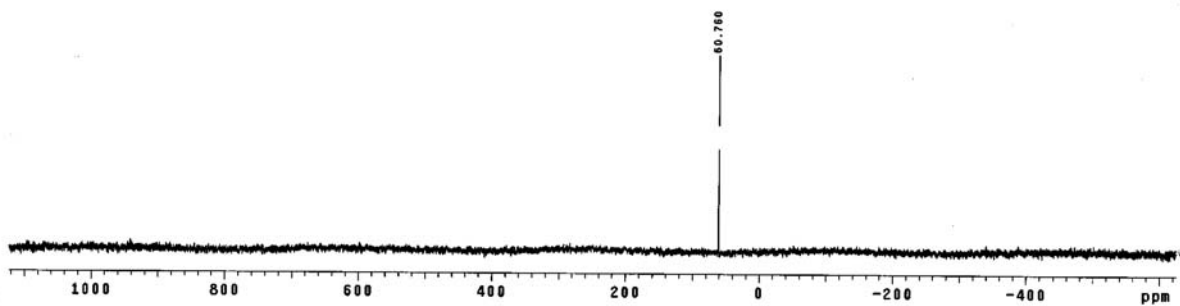

Figure 25. ^{77}Se NMR spectrum of **8**.

Eager 300 Report

Page: 1   Sample: STM149 (STM149)

Method Name   : SP240310
Method File   : D:\CHNS2008\SP240310.mth
Chromatogram   : STM149
Operator ID   : SP   Company Name : C.E. Instruments
Analysed   : 03/24/2010 12:28   Printed   : 3/24/2010 16:27
Sample ID   : STM149 (# 12)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : .658

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	9.9591	44	80425	RS	13.217620	.122729E+07
Carbon	59.8100	67	1063027	RS	1.000000	.270113E+07
Hydrogen	5.4894	169	309545	RS	3.434160	.713951E+07
Totals	75.2584		1452997			

Figure 26. Elemental analysis of **8**.

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM   /   DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0   Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
12 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   12-May-2011 10:35:24

C30H34N4Se2
HBS-STM-2-149 1 (0.010) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (SG, 3x6.00); Cm (1:33)   TOF MS ES+
                                                                                                        5.83e3
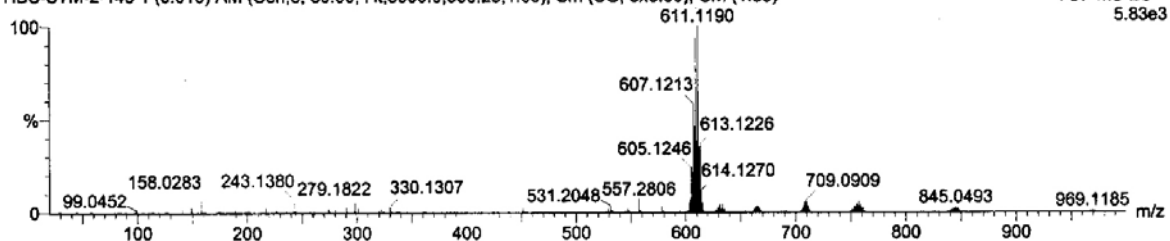
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
				-1.5		
	200.0	5.0	50.0			
611.1190	611.1192	-0.2	-0.3	17.5	1	C30 H35 N4 Se2

Minimum: -1.5
Maximum: 200.0   5.0   50.0

Figure 27. Mass spectrum of **8**.

Figure 28. ^{1}H NMR spectrum of **9**.

HBS-STM-2-329-13C

146.21
135.62
134.20
133.80
129.37
127.29
127.20
123.85
123.78
111.24
110.38

77.51
77.20
76.88

53.22
50.18

30.59

20.19
13.90
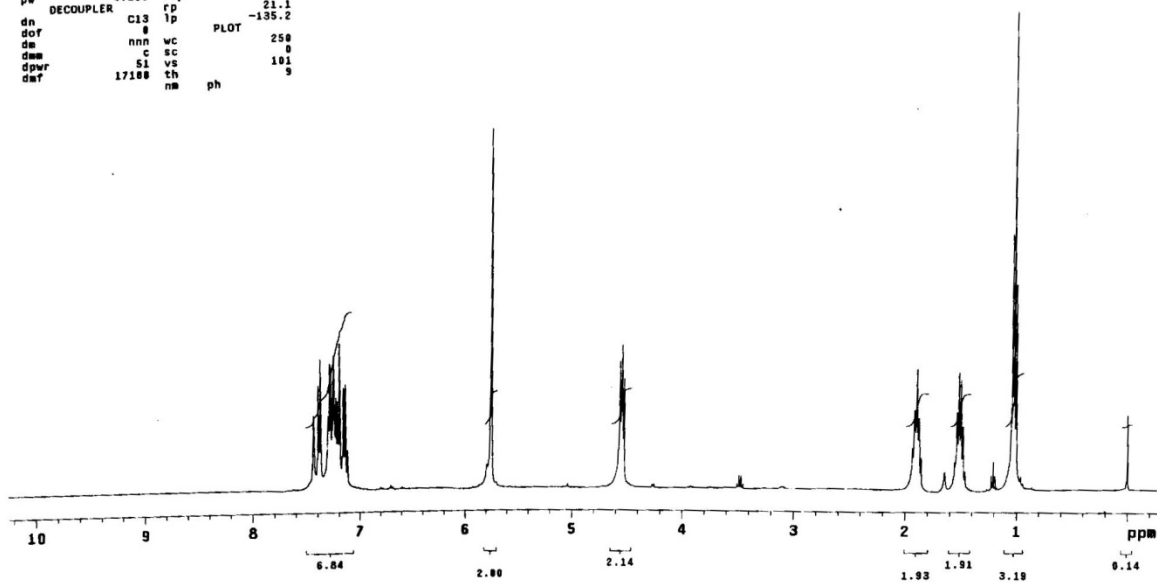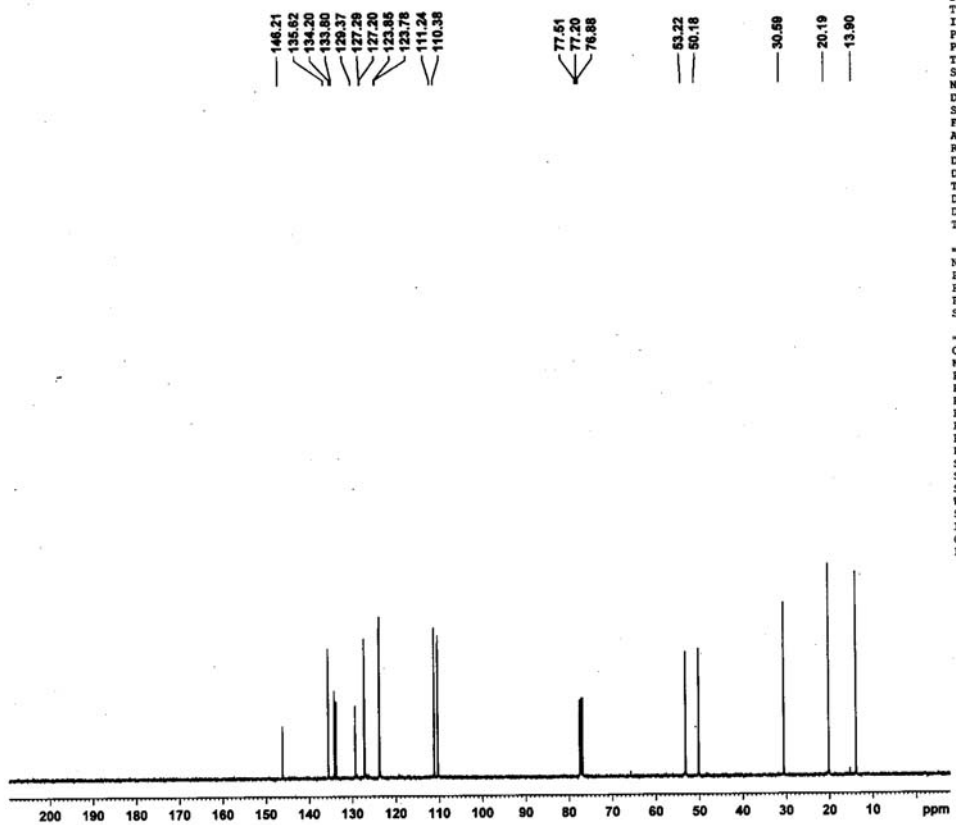
NAME   HBS-STM-2-329-13C
EXPNO   29
PROCNO   1
Date_   20101220
Time   14.59
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgpg30
TD   65536
SOLVENT   CDCl3
NS   185
DS   4
SWH   28409.092 Hz
FIDRES   0.433488 Hz
AQ   1.1534836 sec
RG   2050
DW   17.600 usec
DE   6.50 usec
TE   294.2 K
D1   1.00000000 sec
D11   0.03000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   13C
P1   8.75 usec
PL1   -2.00 dB
PL1W   56.53121948 W
SFO1   100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   -1.00 dB
PL12   14.50 dB
PL13   14.50 dB
PL2W   10.56200695 W
PL12W   0.29767781 W
PL13W   0.29767781 W
SFO2   400.1316005 MHz
SI   32768
SF   100.6127737 MHz
WDW   EM
SSB   0
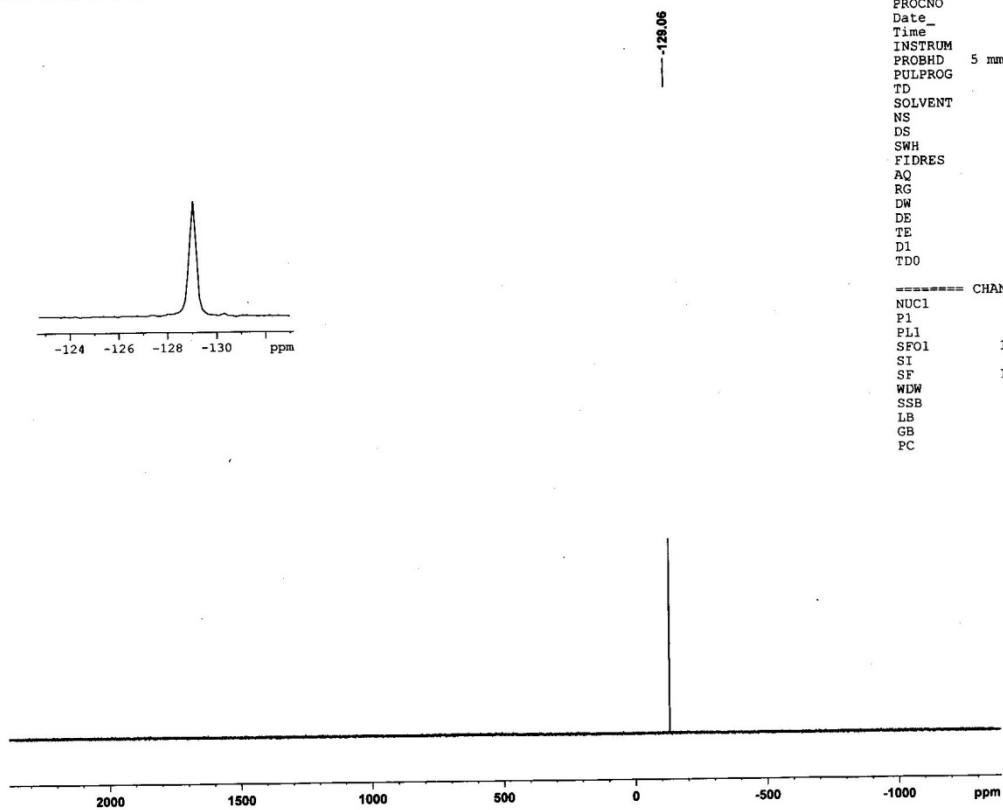LB   1.00 Hz
GB   0.
PC   1.40

Figure 29. ^{13}C NMR spectrum of **9**.

HBS-STM-2-329-Te125

−128.06

NAME   HBS-STM-2-329-Te125
EXPNO   28
PROCNO   1
Date_   20101220
Time   14.50
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zg
TD   65536
SOLVENT   CDCl3
NS   180
DS   4
SWH   476190.469 Hz
FIDRES   7.266090 Hz
AQ   0.0688628 sec
RG   1820
DW   1.050 usec
DE   6.50 usec
TE   294.2 K
D1   1.00000000 sec
TDO   1

======== CHANNEL f1 ========
NUC1   125Te
P1   7.50 usec
PL1   0.00 dB
SFO1   126.3032110 MHz
SI   32768
SF   126.2402248 MHz
WDW   EM
SSB   0
LB   10.00 Hz
GB   0
PC   1.40

Figure 30. ^{125}Te NMR spectrum of **9**.

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM   /   DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0   Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
25 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
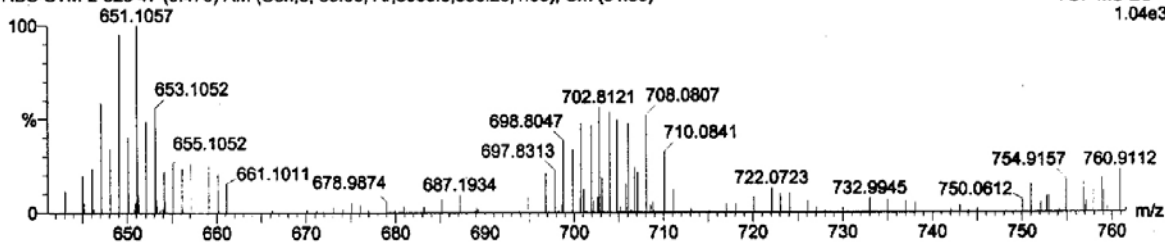
Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   12-May-201111:30:09

C30H34N4Te2
HBS-STM-2-329 47 (0.470) AM (Cen,5, 80.00, Ar,5000.0,556.28,1.00); Cm (34:50)   TOF MS ES+
                                                                                                             1.04e3

Minimum:			-1.5			
Maximum:	200.0	5.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
711.0993	711.0986	0.7	0.9	15.5	1	C30 H35 N4 Te2

Figure 31. Mass spectrum of **9**.
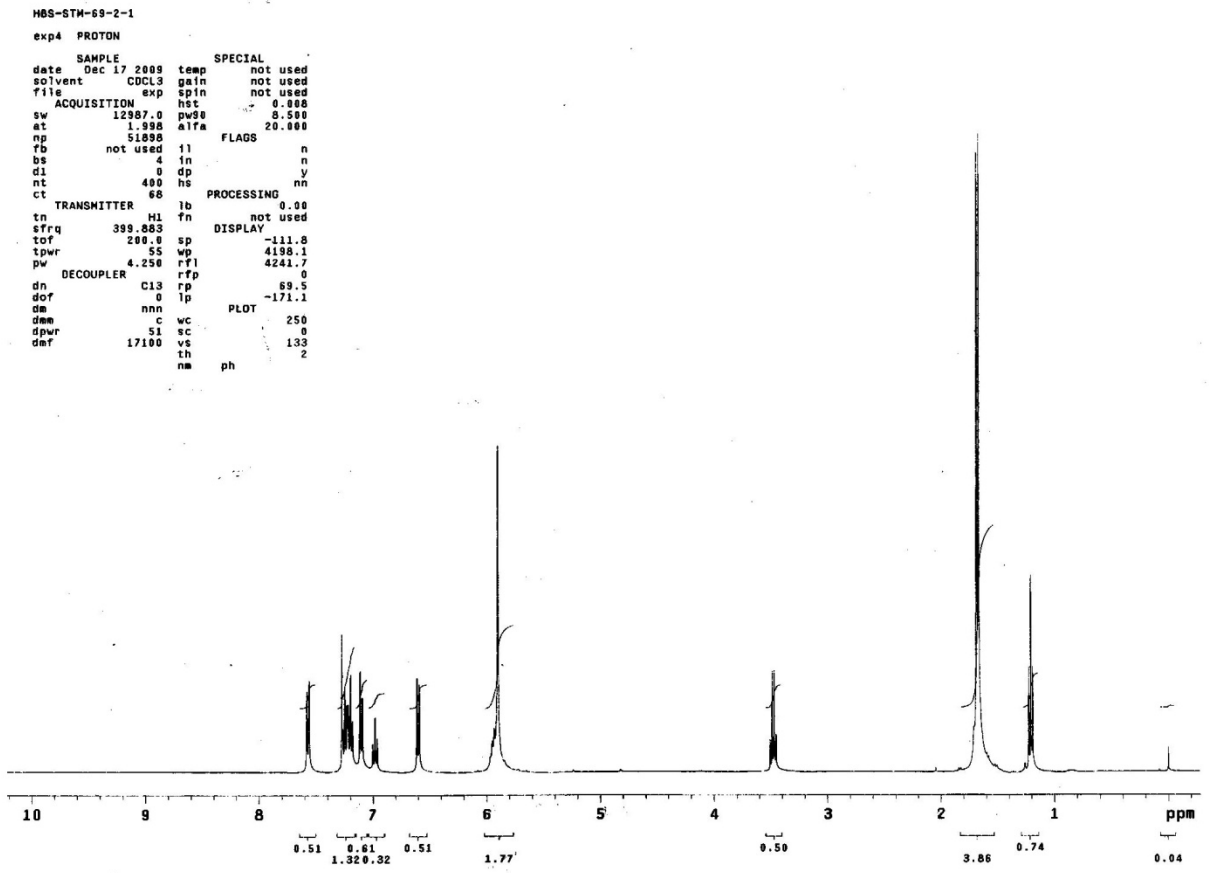
Figure 32. ^{1}H NMR spectrum of **10**.

Figure 33. ^{13}C NMR spectrum of **10**.

HBS-STM-69-2
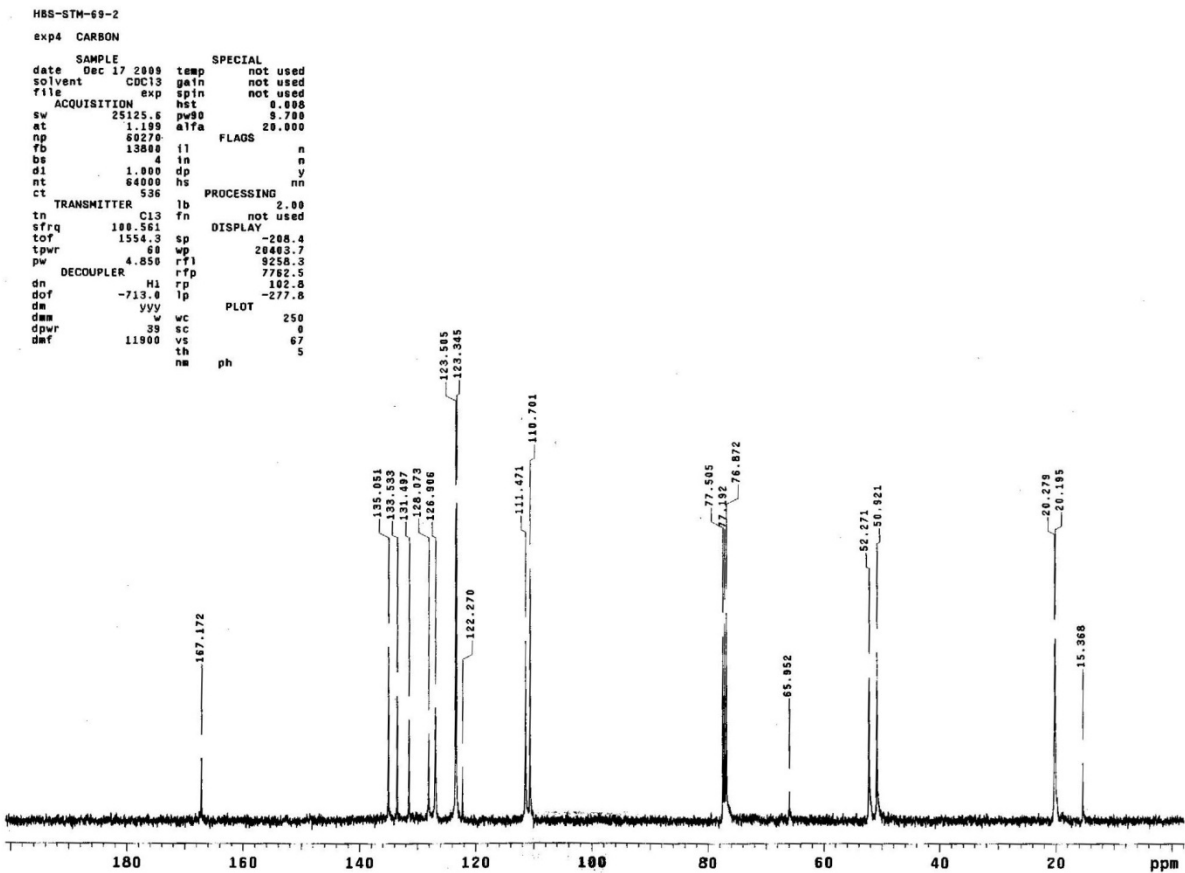
exp9 s2pul

         SAMPLE   DEC. & VT
date   Jan 25 2010   dn   H1
solvent   CDC13   dof   0
file   exp   dm   nnn
     ACQUISITION   dmm   c
sfrq   57.220   dmf   200
tn   Se77   PROCESSING
at   0.640   lb   20.00
np   128000   fn   not used
sw   100000.0
fb   55000   werr
bs   8   wexp
pw   3.0   wbs
pw   3.0   wnt
tpwr   58   DISPLAY
d1   0   sp   -34553.1
tof   0.0   wp   99998.5
nt   32000   vs   20
ct   2168   sc   0
alock   n   wc   250
gain   6   hzmm   48.91
      FLAGS   is   6826.51
il   n   rfl   34554.6
ln   n   rfp   0
dp   y   th   11
                       ins   65.734
                       nm   ph
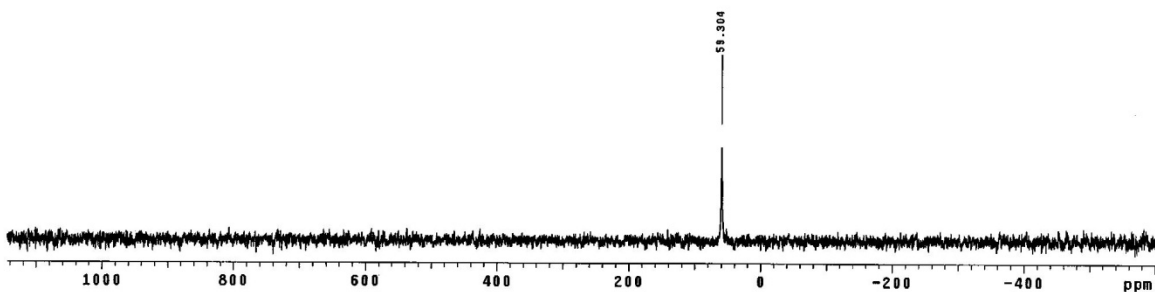
Figure 34. ^{77}Se NMR spectrum of **10**.

Eager 300 Report

Page: 1   Sample: STM69-2 (STM69-2)

```
Method Name    : SP200110
Method File    : D:\CHNS2008\SP200110.mth
Chromatogram   : STM69-2
Operator ID    : SP                    Company  Name : C.E. Instruments
Analysed       : 01/20/2010  15:01     Printed      : 1/29/2010  16:41
Sample ID      : STM69-2 (# 31)        Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)         Sample weight : .844

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.
```

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	8.0573	43	82701	RS	13.745680	.121612E+07
Carbon	50.6726	67	1136782	RS	1.000000	.265804E+07
Hydrogen	4.5040	172	248762	RS	4.569756	.612939E+07
Totals	63.2339		1468245			

Figure 35. Elemental analysis of **10**.

Single Mass Analysis (displaying only valid results)
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
65 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
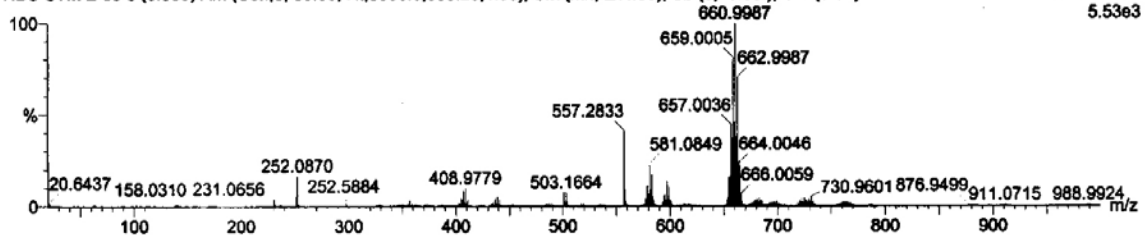
Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   21-Apr-201111:02:48

C28H29N4Se2Br
HBS-STM-2-69 3 (0.030) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (Mn, 2x4.00); Sb (5,40.00); Cm (3:50)   TOF MS ES+
5.53e3

Minimum:				-1.5		
Maximum:		200.0	10.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
660.9987	660.9984	0.2	0.4	17.5	1	C28 H30 N4 Se2 Br

Figure 36. Mass spectrum of **10**.
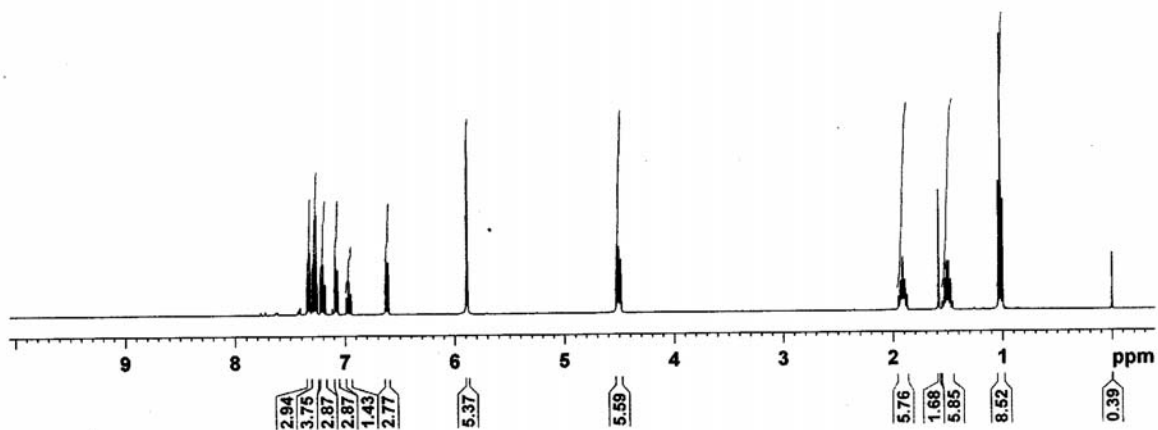
Figure 37. ^{1}H spectrum of **11**.

HBS-STM-3-82-3a-13C

167.55

135.12
133.22
132.91
129.08
128.51
128.06
127.01
123.80
123.75
122.38
110.46
109.87

77.52
77.20
76.88

50.53
47.02

30.30

20.31

13.98
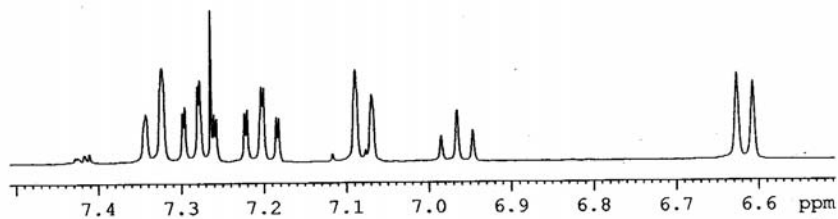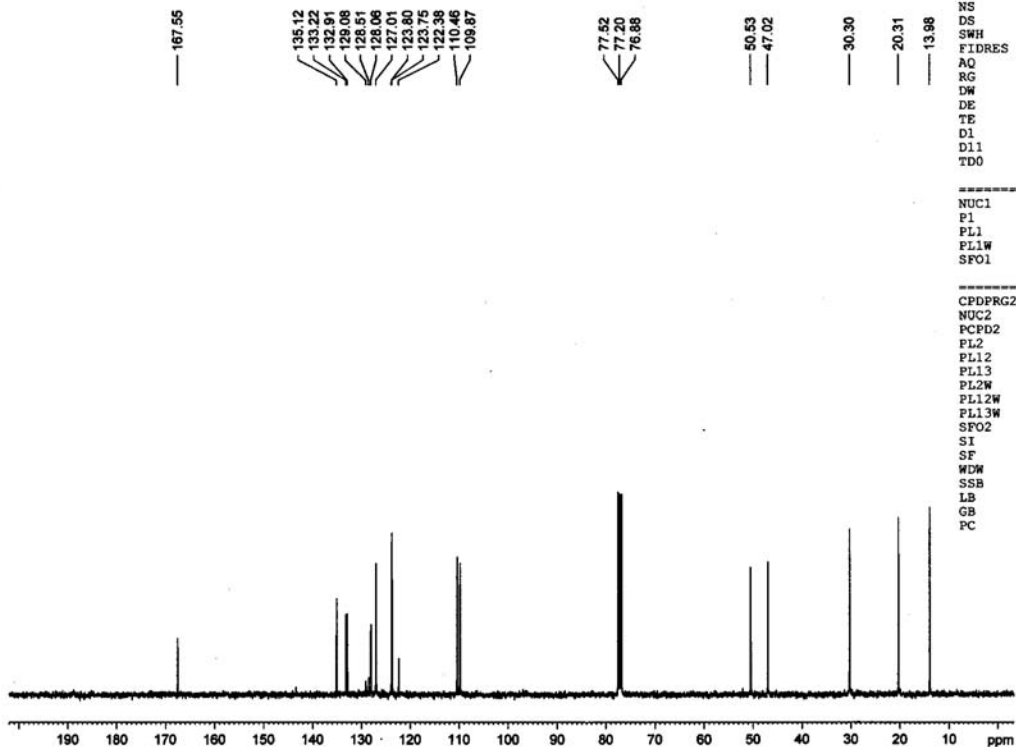
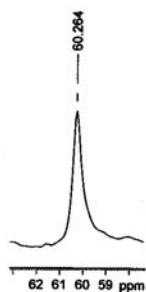NAME   HBS-STM-3-82-3a-13C
EXPNO   8
PROCNO   1
Date_   20110623
Time   9.22
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgpg30
TD   65536
SOLVENT   CDCl3
NS   148
DS   2
SWH   27173.912 Hz
FIDRES   0.414641 Hz
AQ   1.2059124 sec
RG   2050
DW   18.400 usec
DE   6.50 usec
TE   296.4 K
D1   1.00000000 sec
D11   0.03000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   13C
P1   8.75 usec
PL1   -2.00 dB
PL1W   56.53121948 W
SFO1   100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   -1.00 dB
PL12   14.50 dB
PL13   14.50 dB
PL2W   10.56200695 W
PL12W   0.29767781 W
PL13W   0.29767781 W
SFO2   400.1316005 MHz
SI   32768
SF   100.6127595 MHz
WDW   EM
SSB   0
LB   2.00 Hz
GB   0
PC   1.40

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Figure 38. ^{13}C spectrum of **11**.

HBS-STM-3-82-3-77Se

60.264

62 61 60 59 ppm

60.264

NAME   HBS-STM-3-82-3-77Se
EXPNO   7
PROCNO   1
Date_   20110623
Time   8.49
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zg
TD   428544
SOLVENT   CDCl3
NS   100
DS   4
SWH   300000.000 Hz
FIDRES   0.700045 Hz
AQ   0.7142900 sec
RG   228
DW   1.667 usec
DE   6.50 usec
TE   300.0 K
D1   1.00000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   77Se
P1   10.00 usec
PL1   0.00 dB
SFO1   76.3642626 MHz
SI   65536
SF   76.3110246 MHz
WDW   EM
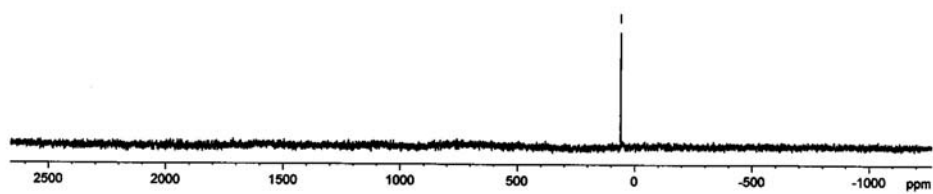SSB   0
LB   30.00 Hz
GB   0
PC   0.10

2500   2000   1500   1000   500   0   -500   -1000   ppm

Figure 39. ^{77}Se spectrum of **11**.

Eager 300 Report

Page: 1   Sample: STM356-1 (STM356-1)

Method Name   : sp270109
Method File   : D:\CHNS2008\sp270109.mth
Chromatogram   : STM356-1
Operator ID   : AGK   Company Name : C.E. Instruments
Analysed   : 01/27/2009 12:28   Printed   : 1/27/2009 15:16
Sample ID   : STM356-1 (# 13)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : 1.188

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	8.9443	43	114340	RS	14.511700	.107605E+07
Carbon	52.5677	66	1659268	RS	1.000000	.265265E+07
Hydrogen	4.5887	169	472792	RS	3.509508	.703485E+07
Totals	66.1007		2246400			

Figure 40. Elemental analysis of **11**.

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM   /   DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0   Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
54 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
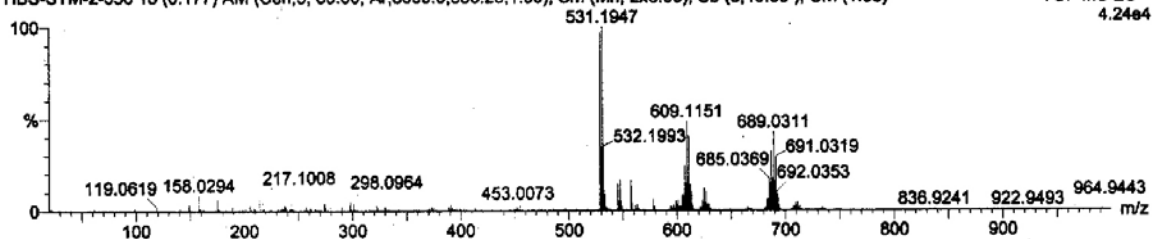
Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   12-May-201111:12:15

C30H33N4Se2Br
HBS-STM-2-356 18 (0.177) AM (Cen,5, 80.00, Ar,5000.0,556.28,1.00); Sm (Mn, 2x6.00); Sb (5,40.00); Cm (1:33)   TOF MS ES+
                                                                                              4.24e4

Minimum:				-1.5					
Maximum:		200.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula			
689.0311	689.0297	1.3	1.9	17.5	1	C30	H34	N4	Br Se2

Figure 41. Mass spectrum of **11**.
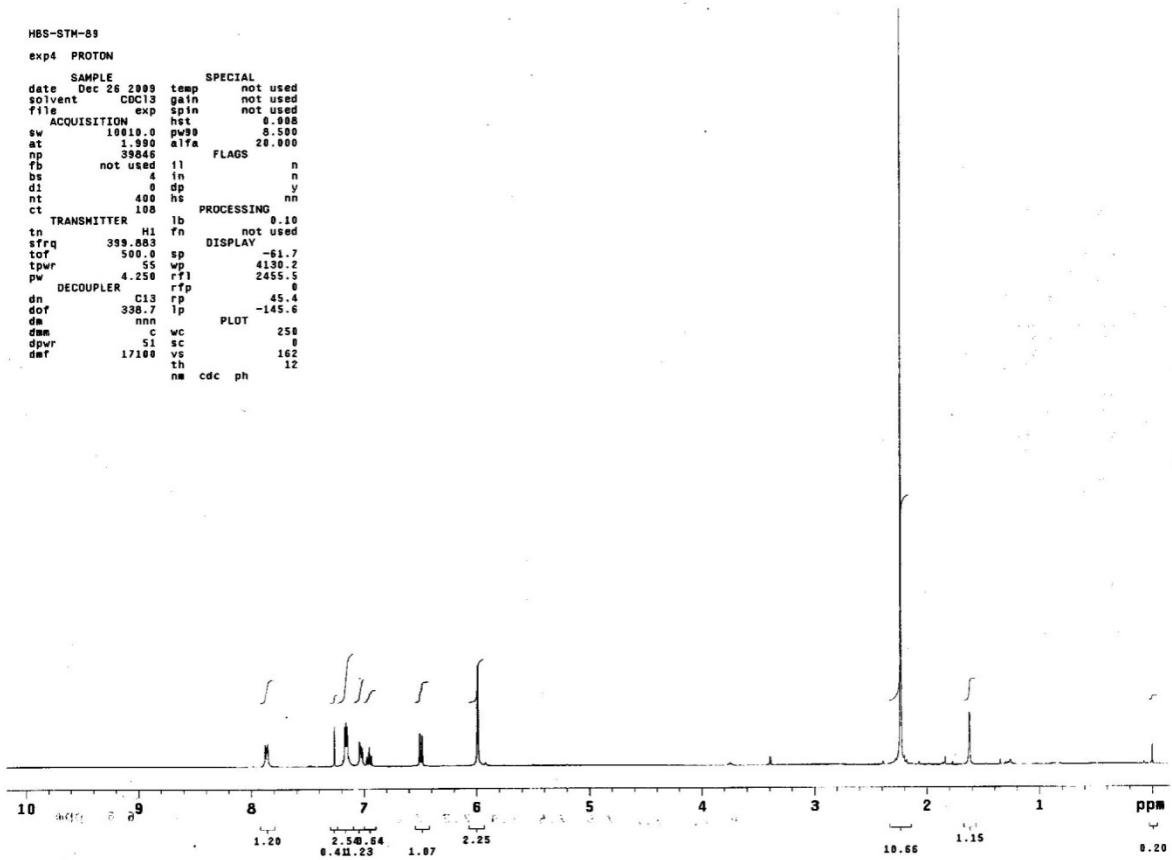
Figure 42. ^{1}H NMR spectrum of **12**.

Figure 43. ^{13}C NMR spectrum of **12**.

HBS-STM-89

exp9 s2pul

            SAMPLE   DEC. & VT
date   Jan 25 2010   dn   H1
solvent   CDCl3   dof   0
file   exp   dm   nnn
      ACQUISITION   dmm   c
sfrq   57.220   dmf   200
tn   Se77   PROCESSING
at   0.840   lb   20.00
np   128000   fn   not used
sw   100000.0
fb   55000   werr
bs   8   wexp
pw   3.0   wbs
pw   3.0   wnt
tpwr   58   DISPLAY
d1   0   sp   -28966.8
tof   0.0   wp   86528.0
nt   32000   vs   26
ct   512   sc   0
alock   n   wc   250
gain   6   hzmm   346.11
      FLAGS   is   6826.51
il   n   rfl   34554.6
in   n   rfp   0
dp   y   th   11
                           ins   65.734
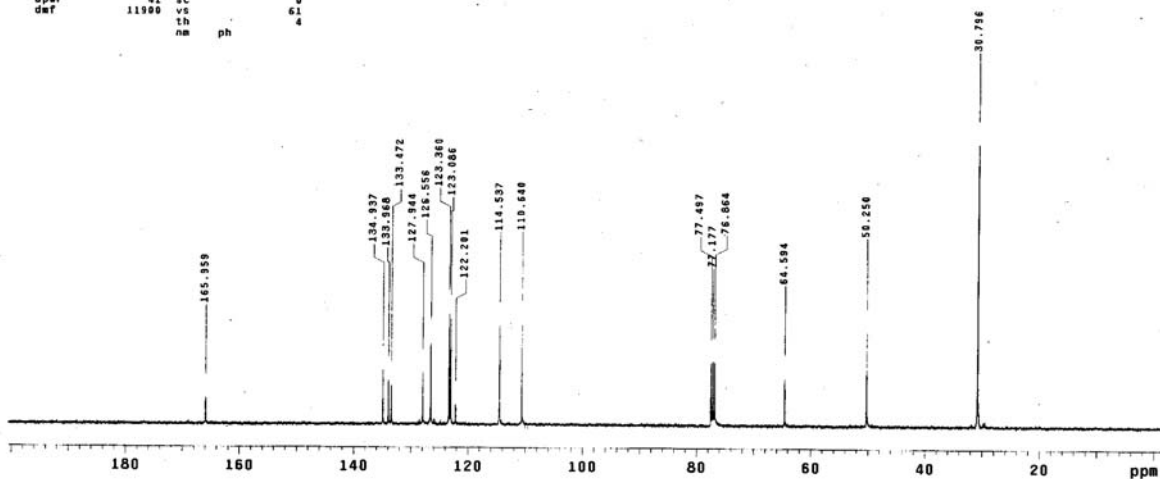   nm   ph
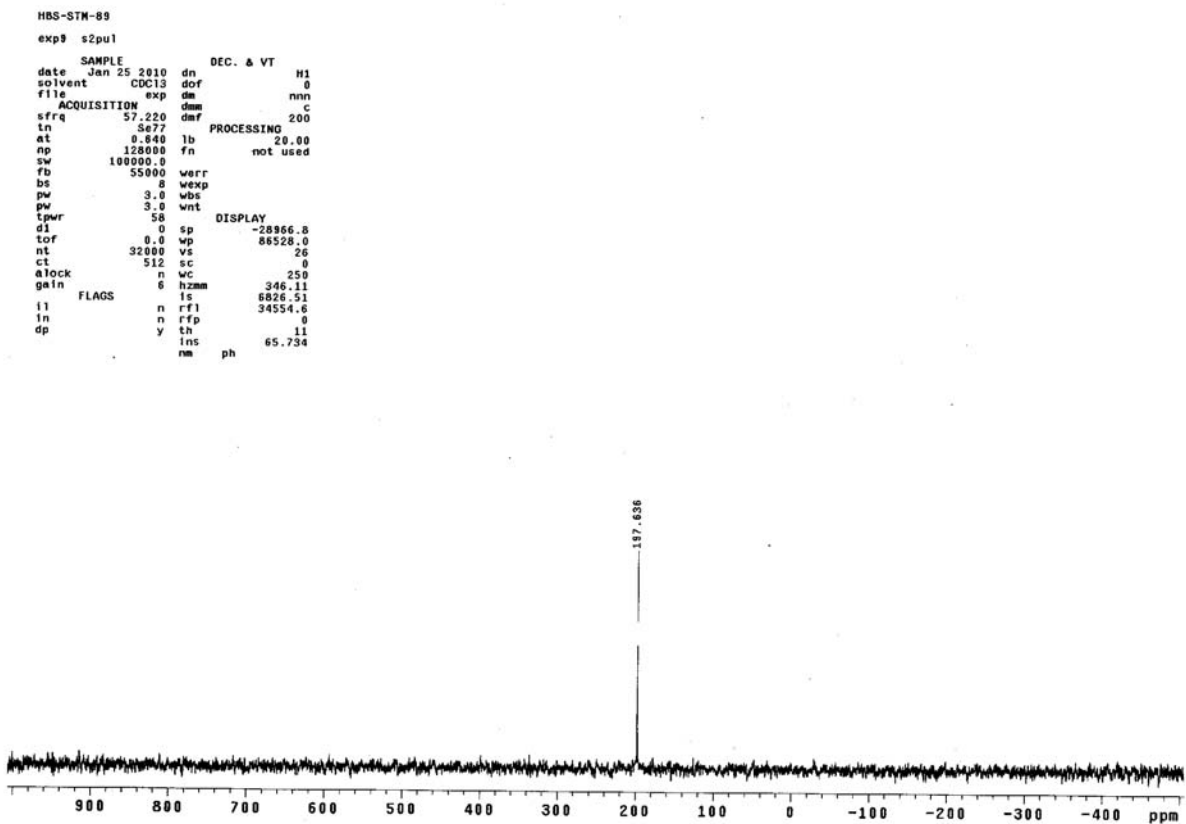
197.636

Figure 44. ^{77}Se NMR spectrum of **12**.

Eager 300 Report

Page: 1   Sample: STM89 (STM89)

Method Name   : SP200110
Method File   : D:\CHNS2008\SP200110.mth
Chromatogram : STM89
Operator ID   : SP   Company Name : C.E. Instruments
Analysed   : 01/20/2010 11:54   Printed   : 1/29/2010 16:41
Sample ID   : STM89 (# 10)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : .8

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	6.7360	43	65534	RS	16.943880	.121612E+0
Carbon	52.2190	67	1110400	RS	1.000000	.265804E+0
Hydrogen	4.7659	172	249458	RS	4.451250	.612939E+0
Totals	63.7208		1425392			

Figure 45. Elemental analysis of **12**.

Single Mass Analysis (displaying only valid results)
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
27 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
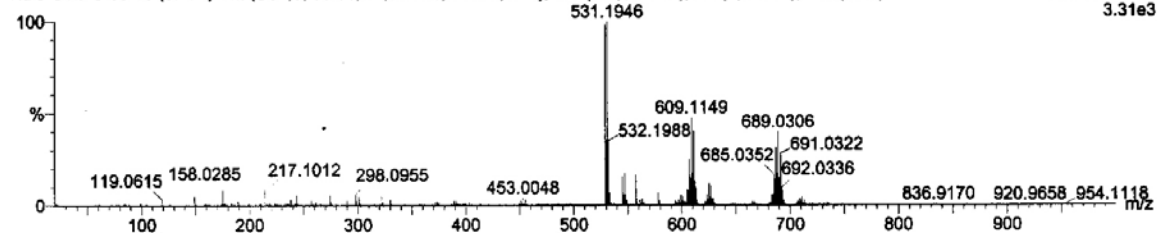
Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   12-May-201111:12:15

C30H33N4Se2Br
HBS-STM-2-89 18 (0.177) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (SG, 3x6.00); Sb (5,40.00); Cm (1:34)   TOF MS ES+
                                                                                                                       3.31e3

[mass spectrum: major peak at 531.1946; other labeled peaks: 119.0615, 158.0285, 217.1012, 298.0955, 453.0048, 532.1988, 609.1149, 685.0352, 689.0306, 691.0322, 692.0336, 836.9170, 920.9658, 954.1118]

Minimum:				-1.5
Maximum:		200.0	5.0	50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula				
689.0306	689.0297	0.9	1.3	17.5	1	C30	H34	N4	Se2	Br

Figure 46. Mass spectrum of **12**.
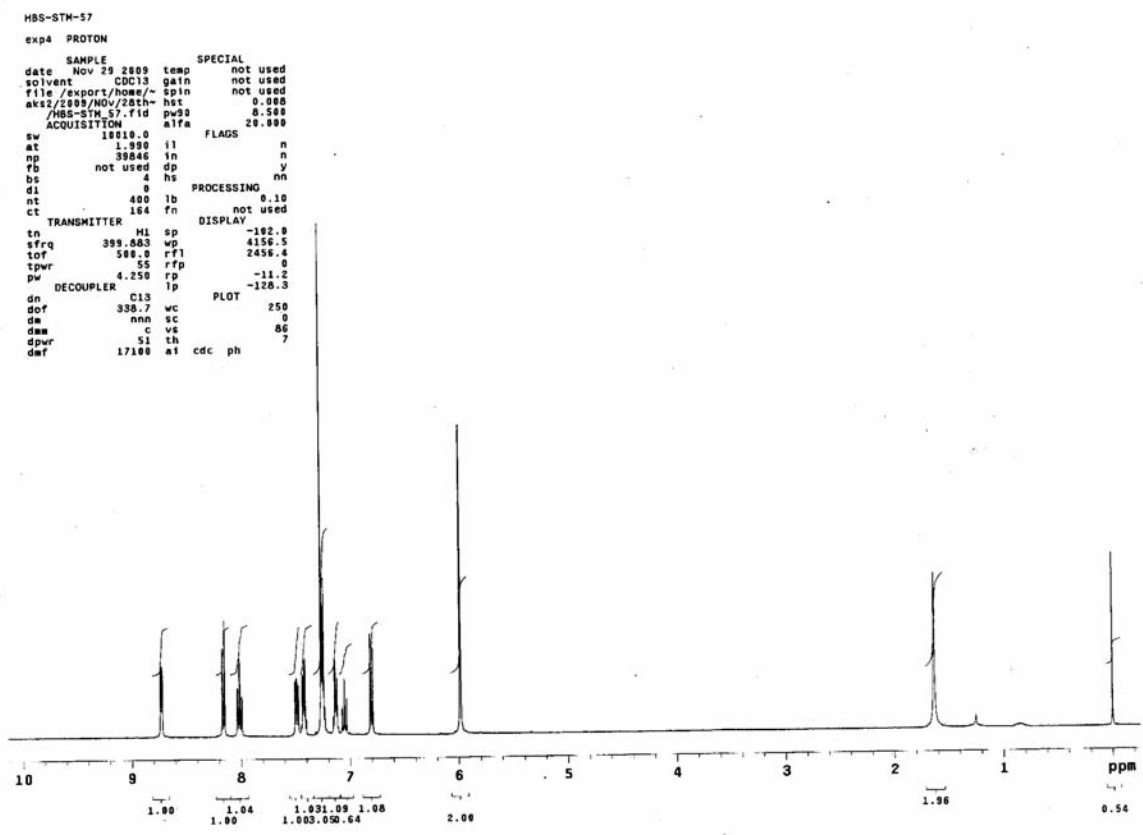
Figure 47. ^{1}H NMR spectrum of **13**.

HBS-STM-63

exp4   CARBON

        SAMPLE   SPECIAL
date   Dec 6 2009   temp   not used
solvent   CDC13   gain   not used
file /export/home/~   spin   not used
hbs1/2009/DEC/HBS-~   hst   0.008
      STM-63-13C.fid pw90   14.000
    ACQUISITION   alfa   20.000
sw   25125.6   FLAGS
at   1.199   il   n
np   60270   in   n
fb   13800   dp   y
bs   4   hs   nn
d1   1.000   PROCESSING
nt   40000   lb   1.00
ct   8204   fn   not used
   TRANSMITTER   DISPLAY
tn   C13   sp   -204.6
sfrq   100.561   wp   20370.7
tof   1554.3   rfl   9254.5
tpwr   56   rfp   7762.5
pw   7.000   rp   44.2
   DECOUPLER   lp   -299.8
dn   H1   PLOT
dof   -713.0   wc   250
dm   yyy   sc   0
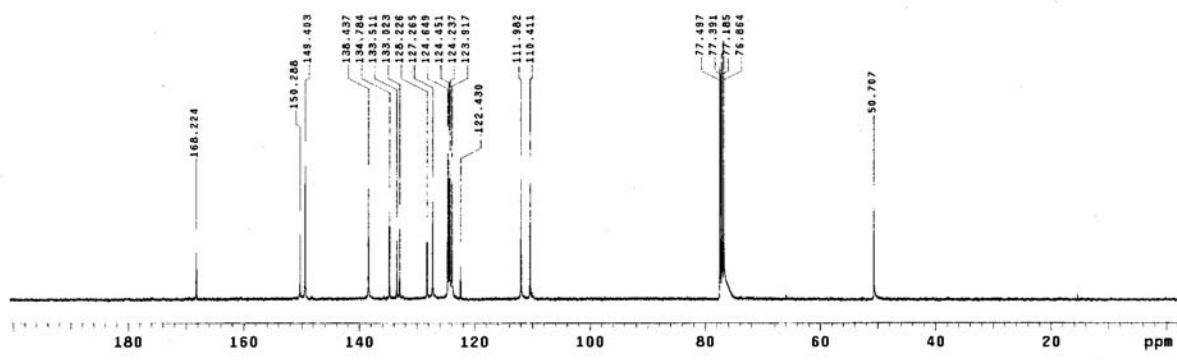dmm   w   vs   55
dpwr   41   th   7
dmf   11900   nm   ph

Figure 48. ^{13}C NMR spectrum of **13**.
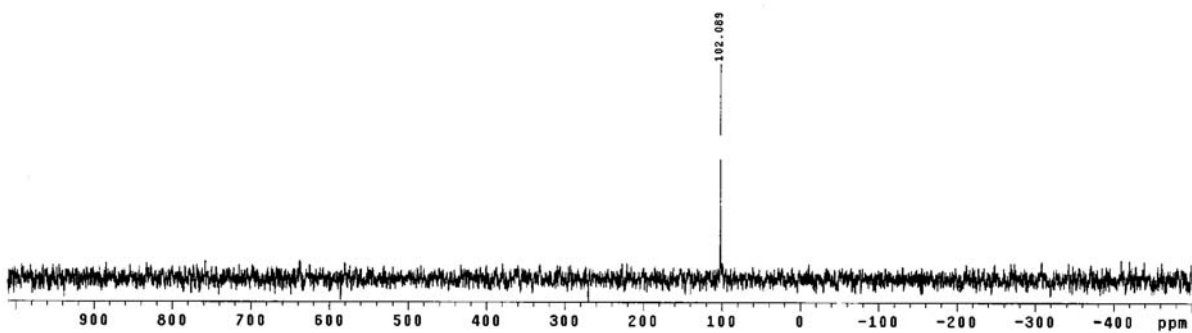
Figure 49. ^{77}Se NMR spectrum of **13**.

Eager 300 Report

Page: 1   Sample: STM89 (STM89)

Method Name   : SP200110
Method File   : D:\CHNS2008\SP200110.mth
Chromatogram : STM89
Operator ID   : SP   Company Name : C.E. Instruments
Analysed   : 01/20/2010 11:54   Printed   : 1/29/2010 16:41
Sample ID   : STM89 (# 10)   Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)   Sample weight : .8

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	6.7360	43	65534	RS	16.943880	.121612E+0
Carbon	52.2190	67	1110400	RS	1.000000	.265804E+0
Hydrogen	4.7659	172	249458	RS	4.451250	.612939E+0
Totals	63.7208		1425392			

Figure 50. Elemental analysis of **13**.

Single Mass Analysis (displaying only valid results)
Tolerance = 10.0 PPM   /   DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0   Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
82 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Micromass : Q-Tof micro (YA-105)   Dept. Of Chemistry I.I.T.(B)   21-Apr-201110:42:09

C32H23N6Se2Br
HBS-STM-2-57 5 (0.051) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Cm (5:28)

TOF MS ES+
723

651.0390 730.9544
728.9551 746.9496
557.2822
649.0433 762.9589
727.9573
286.0662 647.0394 654.0427 792.8774
365.9827 796.8760
367.4853 556.1188 829.8478 935.3365
39.9899 158.0294 214.0911

m/z
50 100 150 200 250 300 350 400 450 500 550 600 650 700 750 800 850 900 950
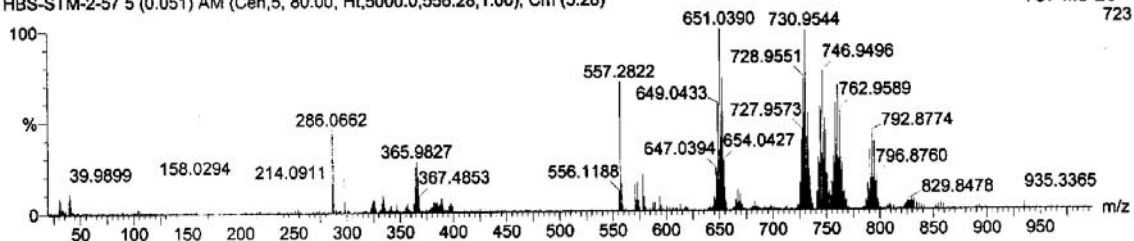
Minimum: -1.5
Maximum: 200.0   10.0   50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
730.9544	730.9576	-3.2	-4.4	25.5	1	C32 H24 N6 Se2 Br

Figure 51. Mass spectrum of **13**.

HBS-STM-177

exp4 PROTON

        SAMPLE   SPECIAL
date   Mar 18 2010   temp   not used
solvent   CDCl3   gain   not used
file   exp   spin   not used
   ACQUISITION   hst   0.008
sw   11990.4   pw90   8.500
at   1.996   alfa   20.000
np   47854   FLAGS
fb   not used   il   n
bs   4   in   n
d1   0   dp   y
nt   400   hs   nn
ct   0   PROCESSING
   TRANSMITTER   fn   not used
tn   H1   DISPLAY
sfrq   399.883   sp   -123.7
tof   200.0   wp   4191.0
tpwr   55   rfl   3747.5
pw   4.250   rfp   0
   DECOUPLER   rp   -0.4
dn   C13   lp   -148.3
dof   0   PLOT
dm   nnn   wc   250
dmm   c   sc   0
dpwr   51   vs   108
dmf   17100   th   8
                    nm   ph

Figure 52. ^{1}H NMR spectrum of complex **14**.

HBS-STM-2-177

exp1   s2pul

        SAMPLE   DEC. & VT
date   Mar 30 2010   dn   H1
solvent   CDC13   dof   0
file   exp   dm   nnn
    ACQUISITION   dmm   c
sfrq   57.219   dmf   200
tn   Se77   PROCESSING
at   0.640   lb   10.00
np   128000   fn   32768
sw   100000.0
fb   55000   werr
bs   8   wexp
pw   3.0   wbs
pw   3.0   wnt
tpwr   58   DISPLAY
d1   0   sp   14770.3
tof   -1200.0   wp   43231.2
nt   32000   vs   13
ct   3160   sc   0
alock   n   wc   250
gain   6   hzmm   172.92
    FLAGS   is   488281.25
il   n   rfl   35754.6
in   n   rfp   0
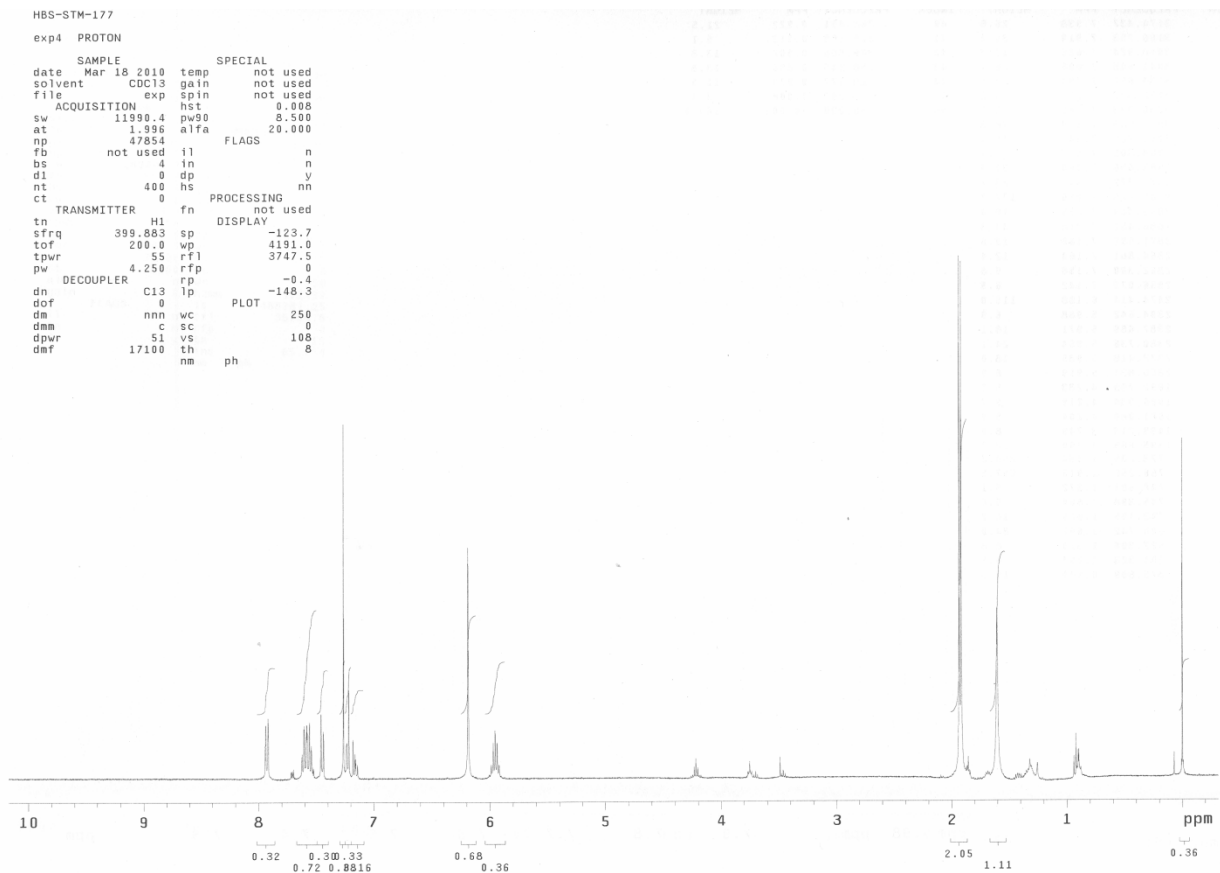dp   y   th   13
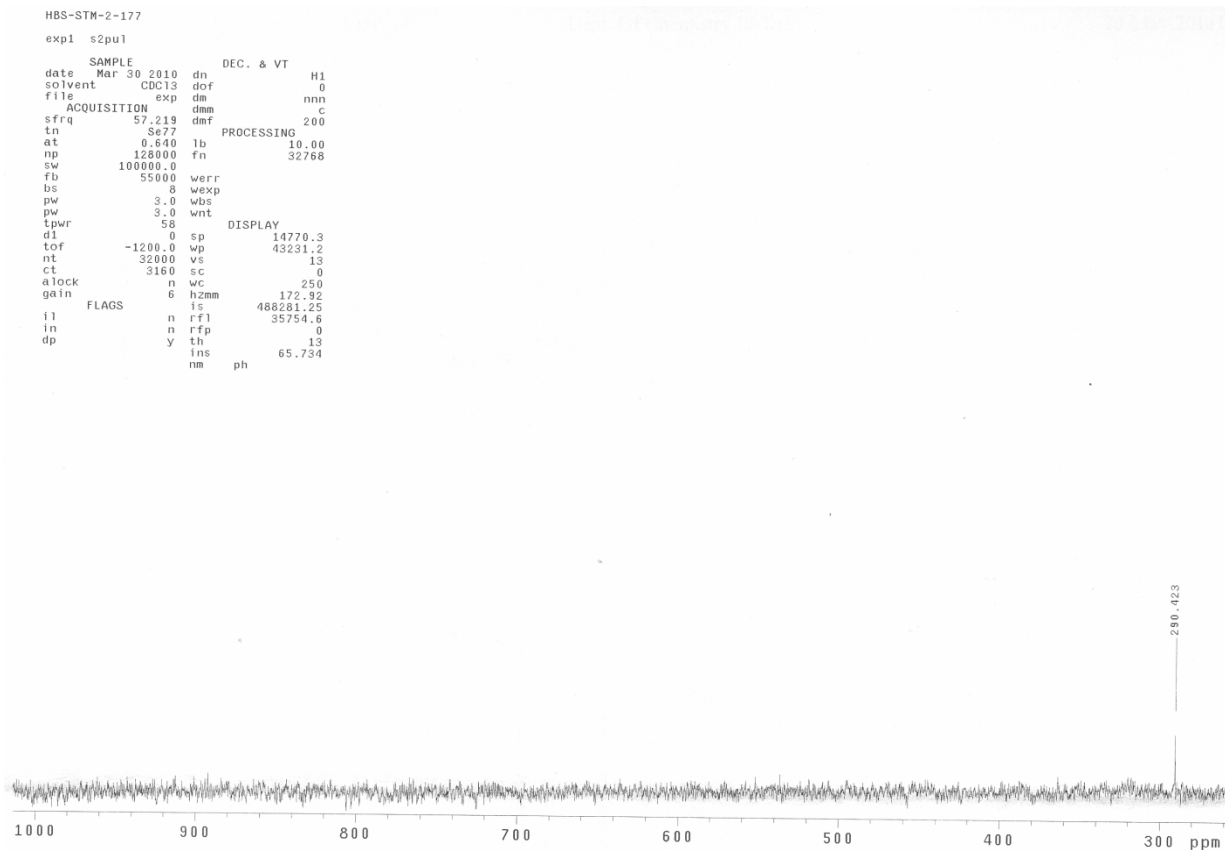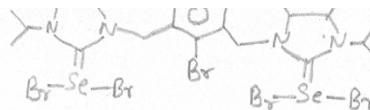   ins   65.734
                      nm   ph

Figure 53. ^{77}Se NMR spectrum of complex **14**.

Eager 300 Report

Page: 1   Sample: STM-2-177 (STM-2-177)

Method Name	: SP160410	
Method File	: D:\CHNS2008\SP160410.mth	
Chromatogram	: STM-2-177	
Operator ID	: SP	
Analysed	: 04/16/2010 11:40	
Sample ID	: STM-2-177 (# 7)	
Analysis Type	: UnkNown (Area)	

Company Name : C.E. Instruments
Printed   : 4/16/2010 14:54
Instrument N. : Instrument #1
Sample weight : .678

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

Element Name	%	Ret.Time		Area	BC	Area ratio	K factor
Nitrogen	5.72	6.0057	0.28 43	53640	RS	11.692450	.131732E+07
Carbon	34.35	34.1689	0.19 68	627183	RS	1.000000	.269542E+07
Hydrogen	2.99	2.8436	0.15 169	201035	RS	3.119770	.710139E+07
Totals		43.0182		881858			

Figure 54. Elemental analysis of **14**.

C28H29Br5N4Se2

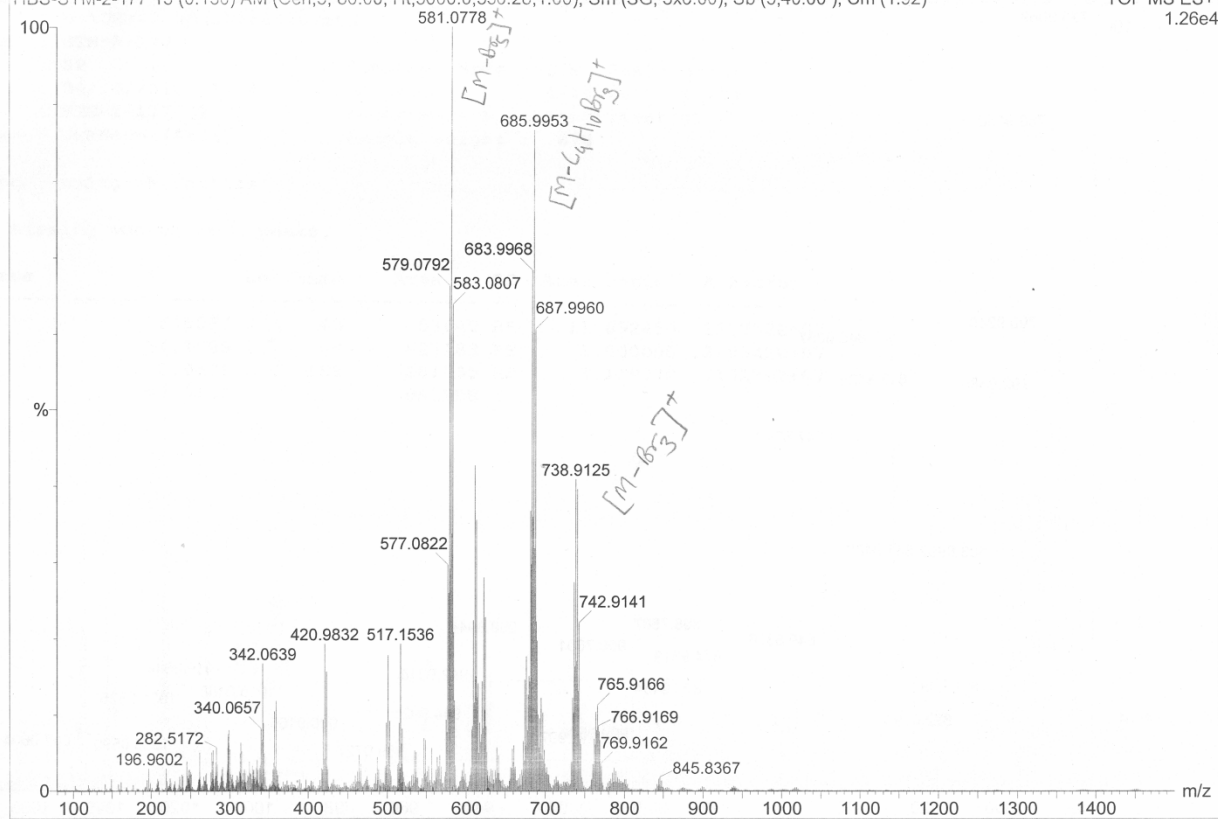HBS-STM-2-177 13 (0.130) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (SG, 3x6.00); Sb (5,40.00); Cm (1:92)　　　TOF MS ES+
　　　1.26e4
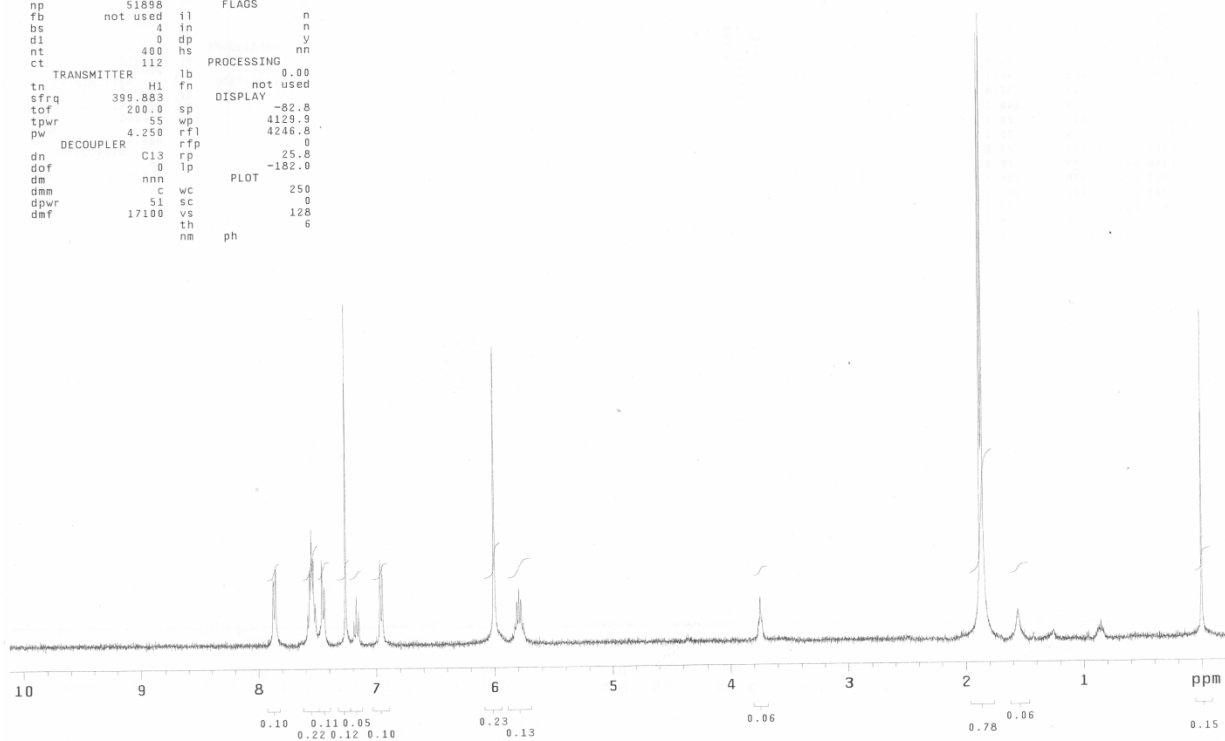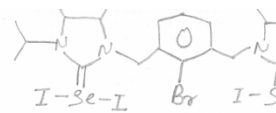

Figure 55. Mass spectrum of complex **14**.

Figure 56. ^{1}H NMR spectrum of complex **15**.
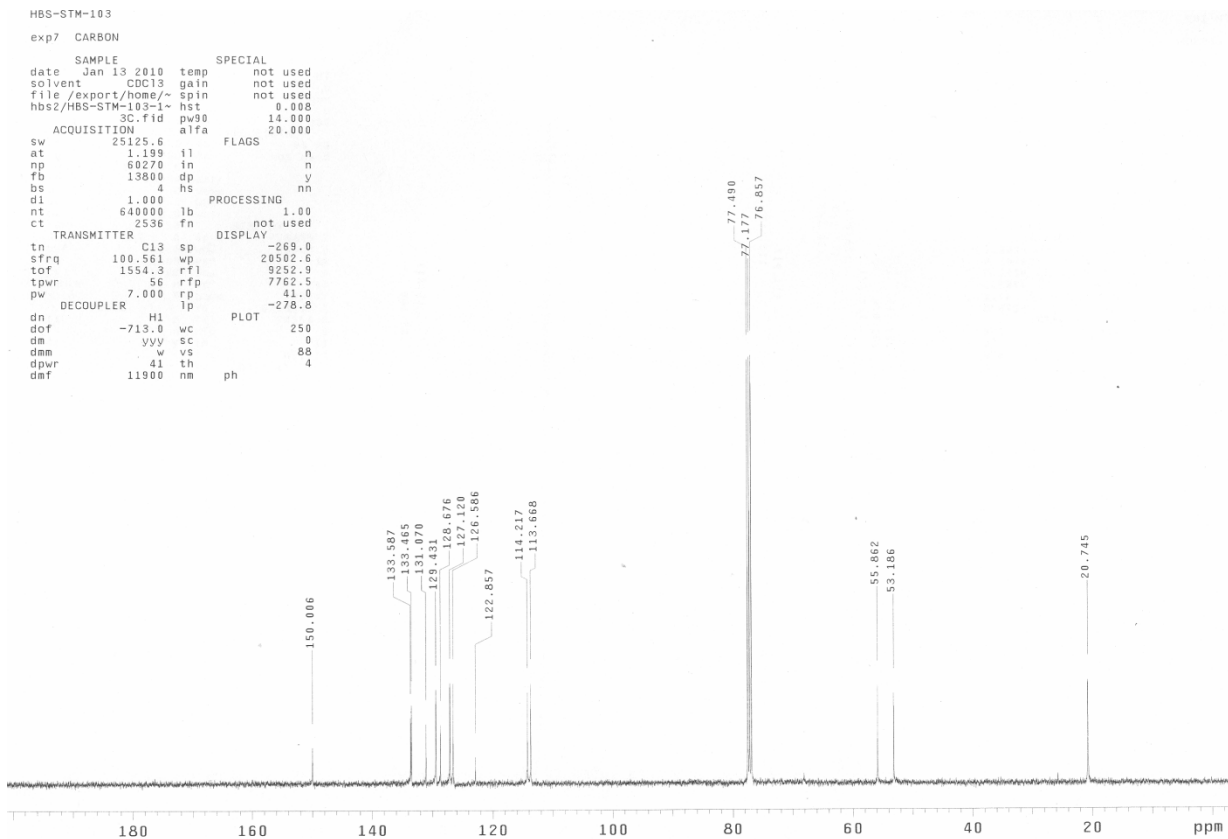
Figure 57. ^{13}C NMR spectrum of complex **15**.

HBS-STM-103

exp9 s2pul

        SAMPLE   DEC. & VT
date   Jan 25 2010   dn   H1
solvent   CDC13   dof   0
file   exp   dm   nnn
   ACQUISITION   dmm   c
sfrq   57.220   dmf   200
tn   Se77   PROCESSING
at   0.640   lb   60.00
np   128000   fn   not used
sw   100000.0
fb   55000   werr
bs   8   wexp
pw   3.0   wbs
pw   3.0   wnt
tpwr   58   DISPLAY
d1   0   sp   -29459.7
tof   0.0   wp   86367.8
nt   32000   vs   16
ct   5808   sc   0
alock   n   wc   250
gain   6   hzmm   345.47
       FLAGS   is   675824.52
il   n   rfl   34554.6
in   n   rfp   0
dp   y   th   11
                       ins   65.734
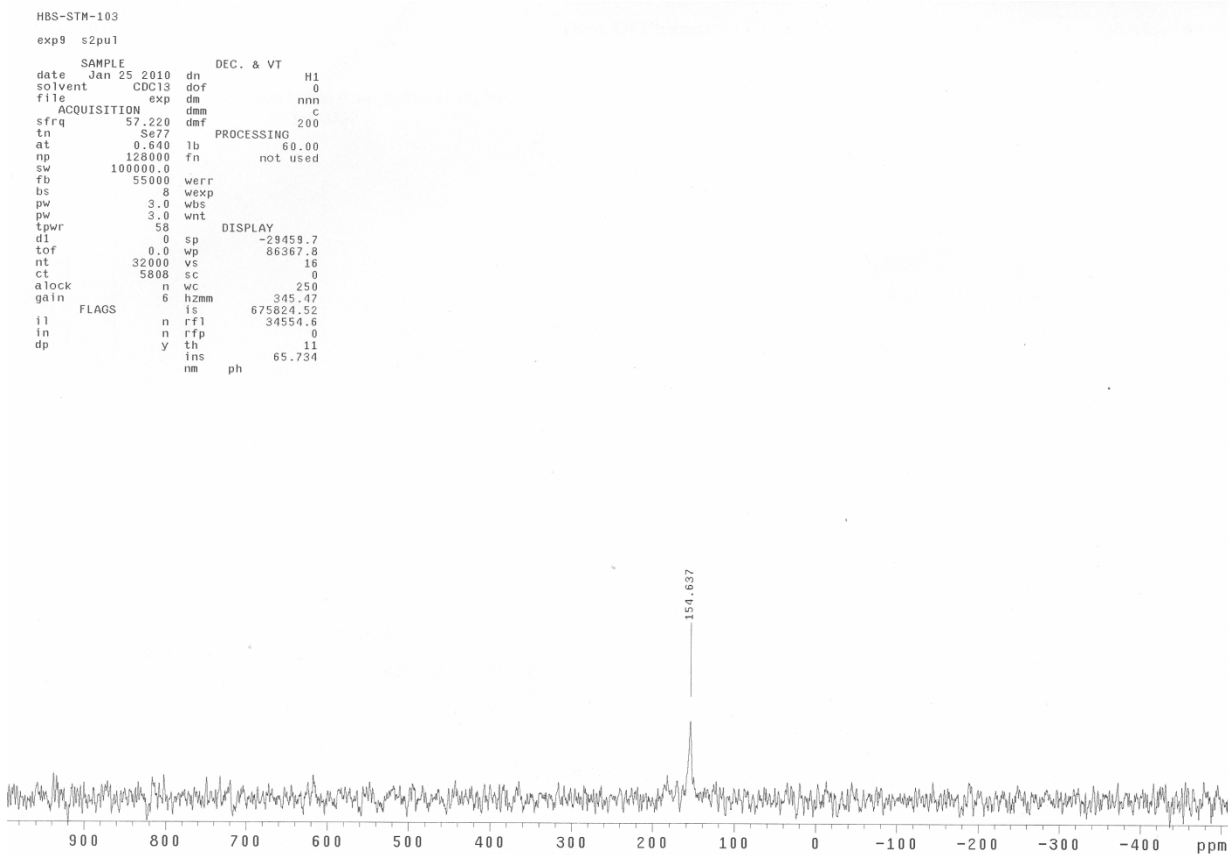   nm   ph

154.637

Figure 58. ^{77}Se NMR spectrum of complex **15**.

C28H29BrI4N4Se2

HBS-STM-2-103 95 (0.941) AM (Cen,5, 80.00, Ht,5000.0,556.28,1.00); Sm (Mn, 3x6.00); Sb (5,40.00); Cm (42:99) TOF MS ES+
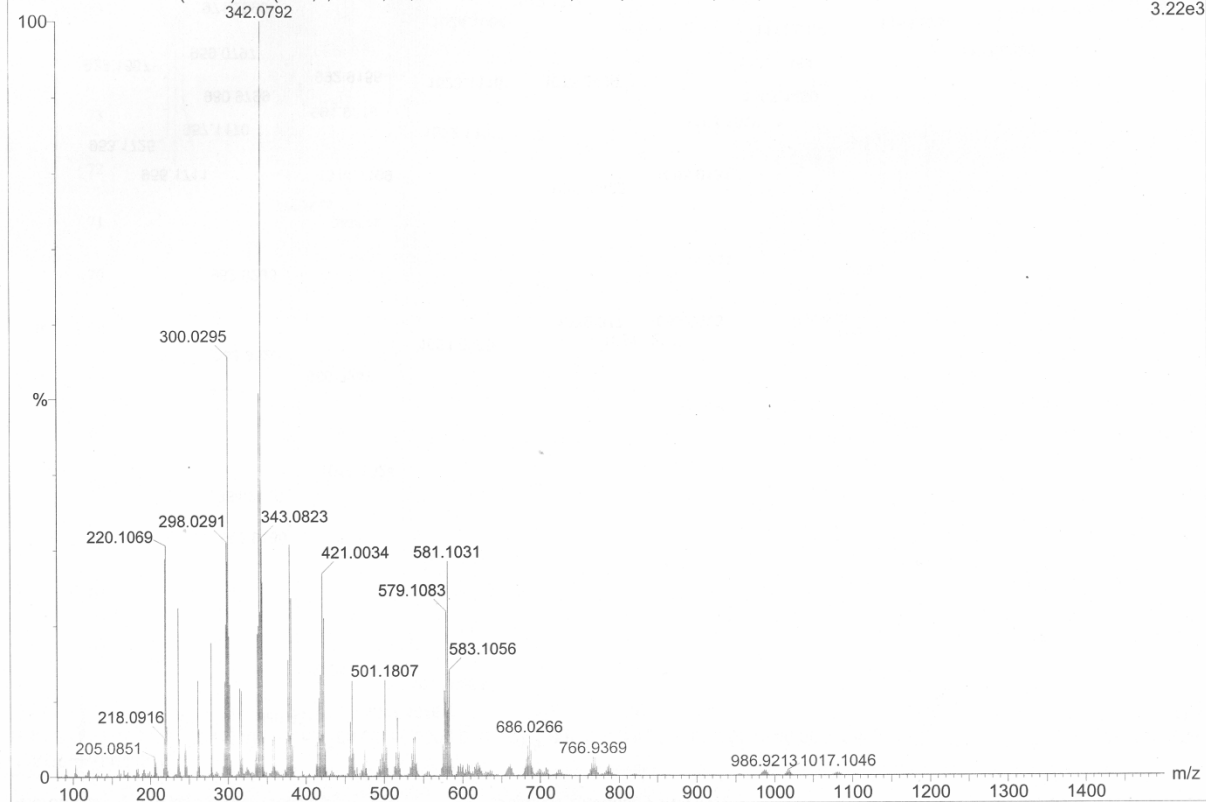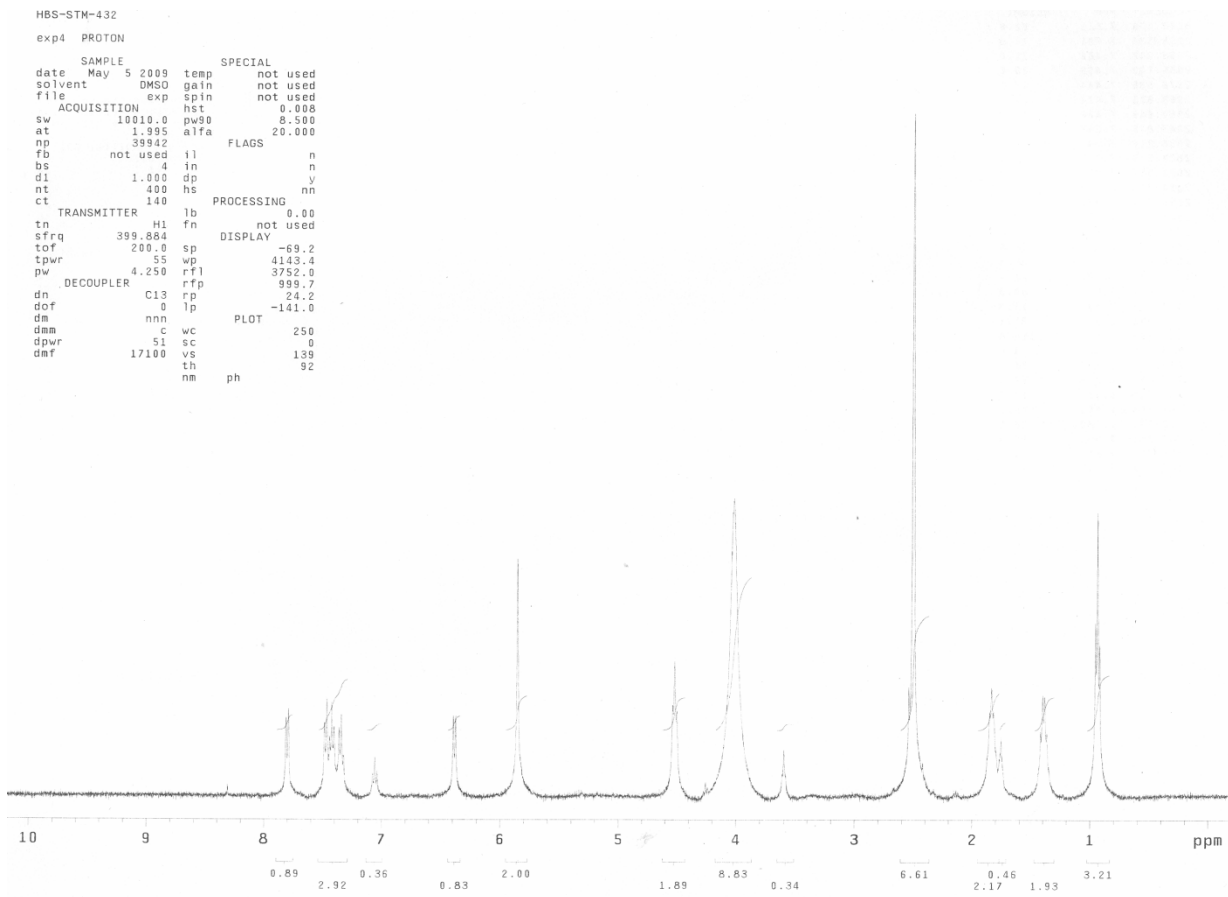3.22e3

Figure 59. Mass spectrum of complex **15**.

HBS-STM-432

exp4 PROTON

        SAMPLE   SPECIAL
date   May 5 2009   temp   not used
solvent   DMSO   gain   not used
file   exp   spin   not used
   ACQUISITION   hst   0.008
sw   10010.0   pw90   8.500
at   1.995   alfa   20.000
np   39942   FLAGS
fb   not used   il   n
bs   4   in   n
d1   1.000   dp   y
nt   400   hs   nn
ct   140   PROCESSING
   TRANSMITTER   lb   0.00
tn   H1   fn   not used
sfrq   399.884   DISPLAY
tof   200.0   sp   -69.2
tpwr   55   wp   4143.4
pw   4.250   rfl   3752.0
   DECOUPLER   rfp   999.7
dn   C13   rp   24.2
dof   0   lp   -141.0
dm   nnn   PLOT
dmm   c   wc   250
dpwr   51   sc   0
dmf   17100   vs   139
                        th   92
                        nm   ph

Figure 60. ^{1}H NMR spectrum of complex **16**.
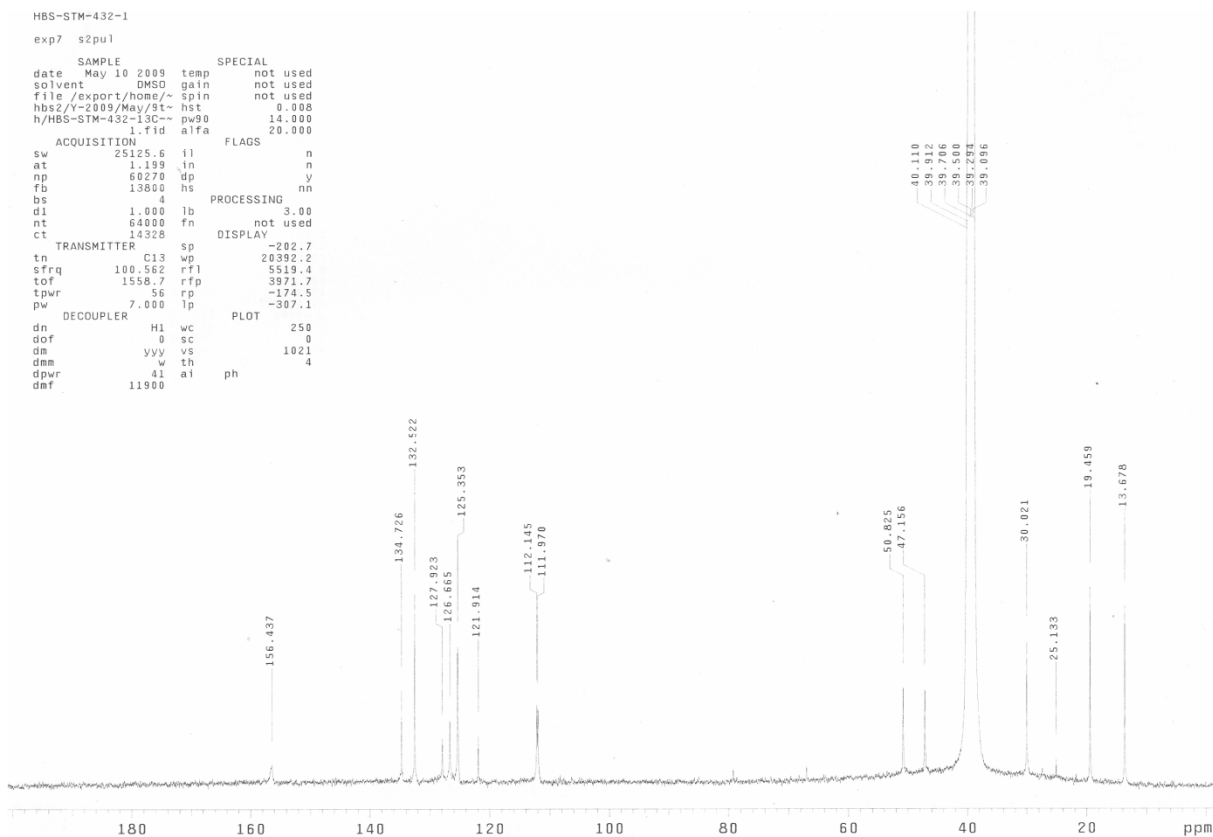
Figure 61. ^{13}C NMR spectrum of complex **16**.
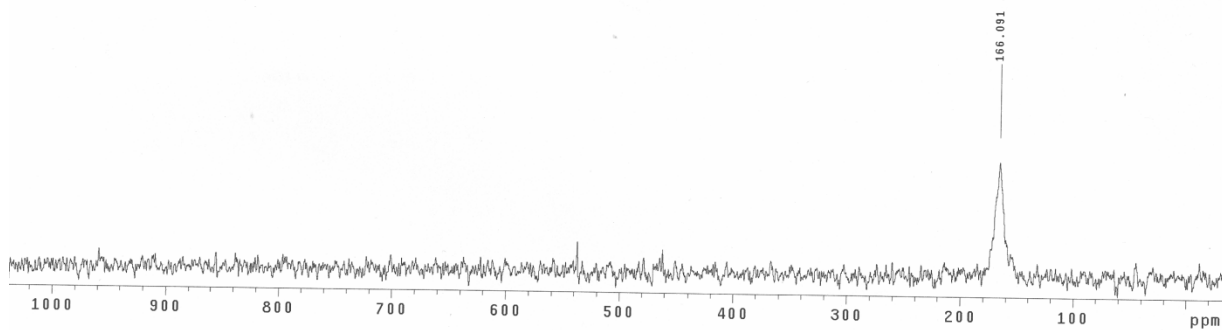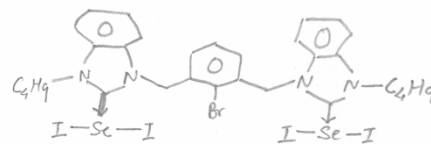
Figure 62. ^{77}Se NMR spectrum of complex **16**.

```
                      Eager 300 Report
Page: 1    Sample: STM432 (STM432)

Method Name    : SP060509
Method File    : D:\CHNS2008\SP060509.mth
Chromatogram   : STM432
Operator ID    : SP                    Company  Name : C.E. Instruments
Analysed       : 05/06/2009  14:45     Printed       : 5/6/2009  16:02
Sample ID      : STM432 (# 24)         Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)         Sample weight : 1.5


Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

   Element Name          %       Ret.Time     Area    BC  Area ratio   K factor
--------------- -------------- -------- ---------- -- ------------ -----------
Nitrogen        4.69    5.2153  0.52  43    100027 FU    12.102110 .127862E+07
        2               0.0000        59    119904 FU                  0.0000
Carbon          30.15  30.0746  0.08  66   1210533 FU     1.000000 .267797E+07
Hydrogen        2.78    2.4616  0.32 172    264102 RS     4.583580 .674873E+07
Totals                 37.7515            1694565
```
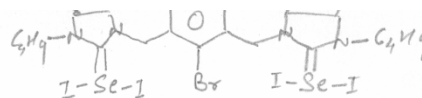
Figure 63. Elemental analysis of **16**.

HBS-NG-3-10-1h

NAME   HBS-NG-3-10-1h
EXPNO   15
PROCNO   1
Date_   20130305
Time   13.15
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zg30
TD   52630
SOLVENT   DMSO
NS   36
DS   0
SWH   8223.685 Hz
FIDRES   0.156255 Hz
AQ   3.1999540 sec
RG   32
DW   60.800 usec
DE   6.50 usec
TE   294.4 K
D1   1.00000000 sec
TD0   1

======== CHANNEL f1 ========
NUC1   1H
P1   13.50 usec
PL1   -1.00 dB
PL1W   10.56200695 W
SFO1   400.1324710 MHz
SI   32768
SF   400.1300025 MHz
WDW   EM
SSB   0
LB   0.10 Hz
GB   0
PC   1.00
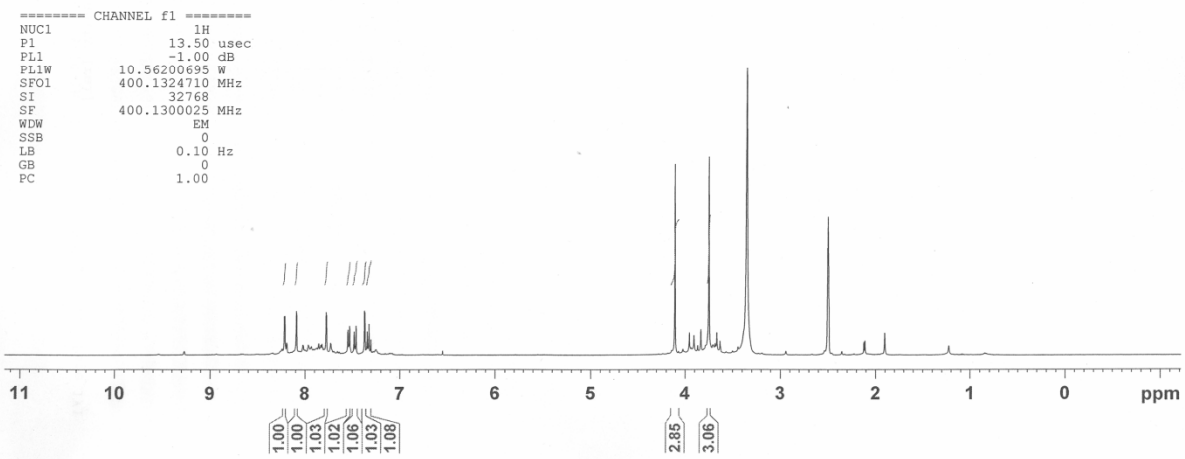
Figure 64: ^{1}H NMR spectrum of **18**.

HBS-NG-03-10-13C

NAME   HBS-NG-03-10-13C
EXPNO   11
PROCNO   1
Date_   20130206
Time   1.42
INSTRUM   spect
PROBHD   5 mm PABBO BB-
PULPROG   zgpg30
TD   65536
SOLVENT   CDCl3
NS   8642
DS   4
SWH   24038.461 Hz
FIDRES   0.366798 Hz
AQ   1.3631988 sec
RG   2050
DW   20.800 usec
DE   6.50 usec
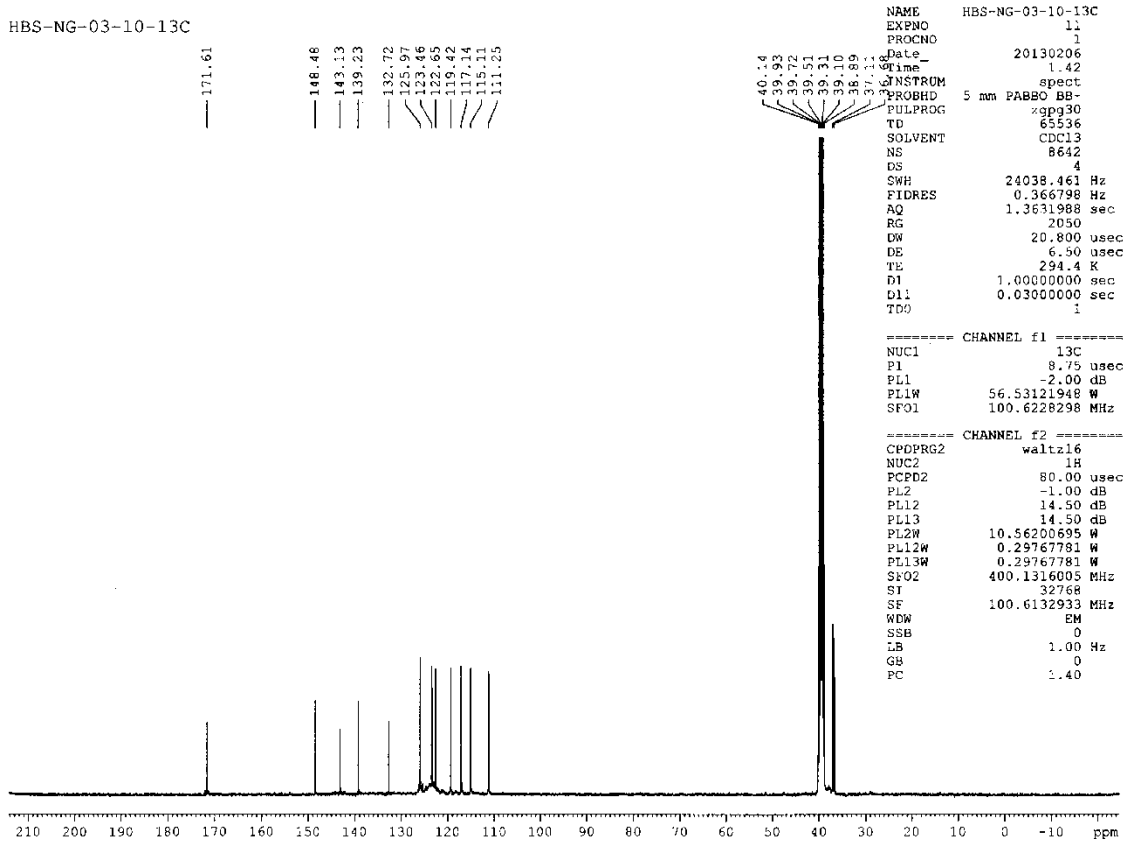TE   294.4 K
D1   1.00000000 sec
D11   0.03000000 sec
TD0   1

======= CHANNEL f1 =======
NUC1   13C
P1   8.75 usec
PL1   -2.00 dB
PL1W   56.53121948 W
SFO1   100.6228298 MHz

======= CHANNEL f2 =======
CPDPRG2   waltz16
NUC2   1H
PCPD2   80.00 usec
PL2   -1.00 dB
PL12   14.50 dB
PL13   14.50 dB
PL2W   10.56200695 W
PL12W   0.29767781 W
PL13W   0.29767781 W
SFO2   400.1316005 MHz
SI   32768
SF   100.6132933 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40

171.61

148.48
143.13
139.23
132.72
125.97
123.46
122.65
119.42
117.14
115.11
111.25

40.14
39.93
39.72
39.51
39.31
39.10
38.89
37.11

Figure 65: ^{13}C NMR spectrum of **18**.

HBS-NG-3-10-77Se

NAME	HBS-NG-3-10-77Se	
EXPNO	5	
PROCNO	1	
Date_	20130123	
Time	7.57	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg	
TD	65536	
SOLVENT	DMSO	
NS	147	
DS	4	
SWH	326086.969	Hz
FIDRES	4.975692	Hz
AQ	0.1005385	sec
RG	256	
DW	1.533	usec
DE	6.50	usec
TE	292.4	K
D1	1.00000000	sec
TD0	1	

======== CHANNEL f1 ========
NUC1	77Se	
P1	10.00	usec
PL1	0.00	dB
SFO1	76.3490004	MHz
SI	65536	
SF	76.3110246	MHz
WDW	EM	
SSB	0	
LB	30.00	Hz
GB	0	
PC	0.00	

97.858
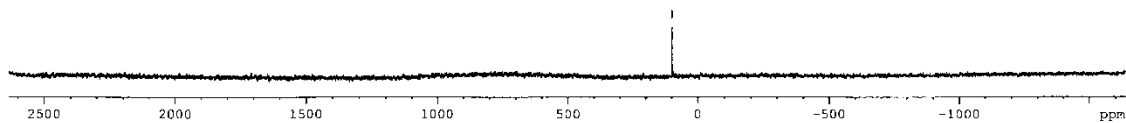
Figure 66 : ^{77}Se NMR spectrum of **18**.

Figure 67 : Mass spectrum of **18**.

HBS-NG-2-45-1H

NAME	HBS-NG-2-45-1H
EXPNO	6
PROCNO	1
Date_	20120718
Time	23.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CD3CN
NS	52
DS	0
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	90.5
DW	60.800 usec
DE	6.50 usec
TE	292.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

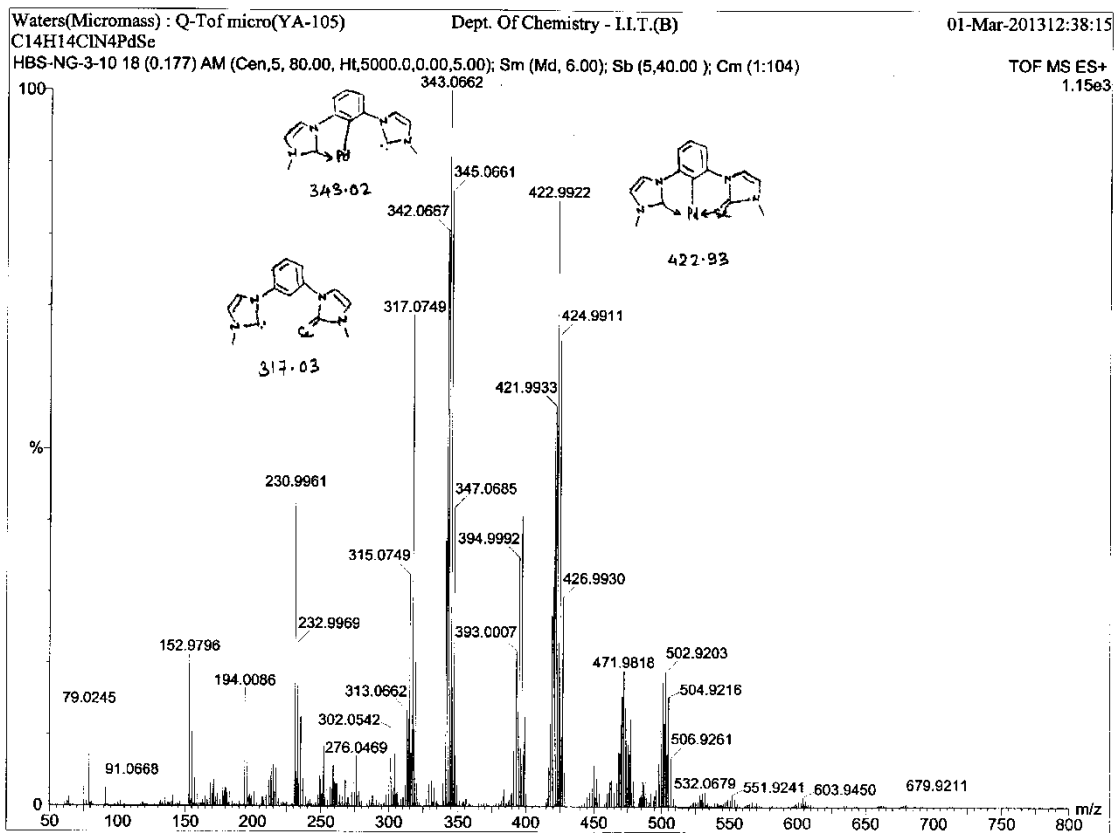| NUC1	1H
P1	13.50 usec
PL1	-1.00 dB
PL1W	10.56200695 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300111 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Figure 68. ^{1}H NMR spectrum of complex **19**.
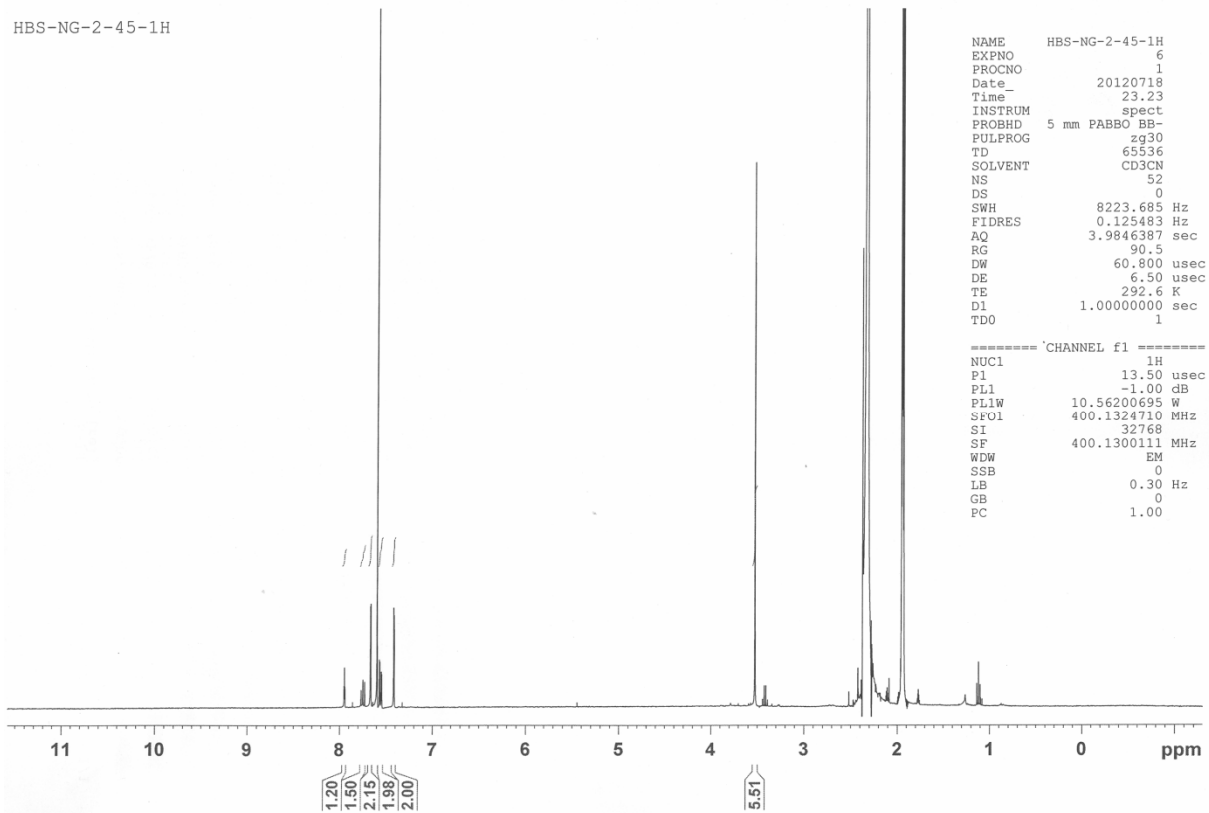
HBS-NG-2-45-19F

```
NAME         HBS-NG-2-45-19F
EXPNO                     12
PROCNO                     1
Date_               20120217
Time                    2.32
INSTRUM                spect
PROBHD      5 mm PABBO BB-
PULPROG              zgflqn
TD                    131072
SOLVENT               CD3CN
NS                        23
DS                         4
SWH           166666.672 Hz
FIDRES          1.271566 Hz
AQ             0.3932660 sec
RG                      2050
DW                 3.000 usec
DE                 6.50 usec
TE                   292.9 K
D1            1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                    19F
P1                 13.00 usec
PL1                -3.00 dB
PL1W        17.04036522 W
SFO1        376.4979895 MHz
SI                    65536
SF          376.4983660 MHz
WDW                      EM
SSB                        0
LB                   1.00 Hz
GB                         0
PC                     1.00
```
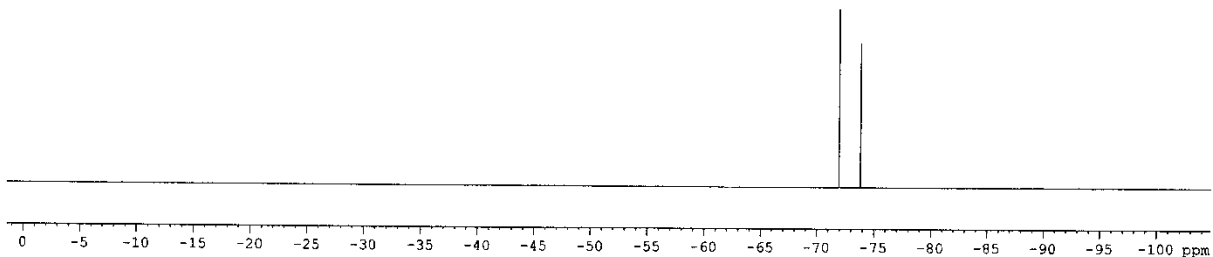
-71.93
-73.81

0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 ppm

Figure 69. ^{19}F NMR spectrum of complex **19**.

HBS-NG-2-45-31P

```
NAME          HBS-NG-2-45-31P
EXPNO               9
PROCNO              1
Date_        20120217
Time             2.28
INSTRUM         spect
PROBHD   5 mm PABBO BB-
PULPROG        zgpg30
TD              65536
SOLVENT         CD3CN
NS                 56
DS                  4
SWH        64102.563 Hz
FIDRES      0.978127 Hz
AQ         0.5112308 sec
RG               2050
DW             7.800 usec
DE              6.50 usec
TE             292.9 K
D1        1.00000000 sec
D11       0.03000000 sec
TDO                 1

======== CHANNEL f1 ========
NUC1              31P
P1              8.40 usec
PL1            -1.00 dB
PL1W      29.72541046 W
SFO1      161.9755930 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          80.00 usec
PL2            -1.00 dB
PL12           14.50 dB
PL13           14.50 dB
PL2W      10.56200695 W
PL12W      0.29767781 W
PL13W      0.29767781 W
SFO2      400.1316005 MHz
SI              32768
SF        161.9755930 MHz
WDW                EM
SSB                 0
LB              8.00 Hz
GB                  0
PC               1.40
```
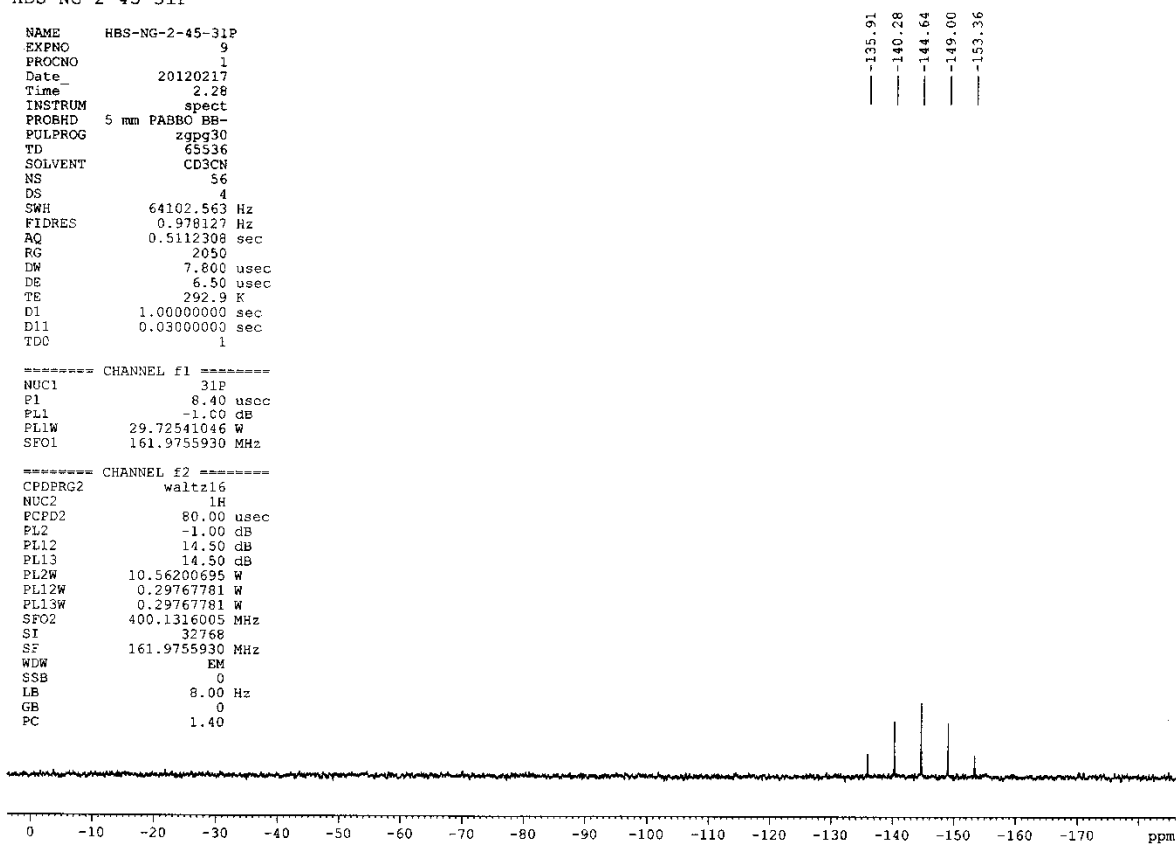
-135.91
-140.28
-144.64
-149.00
-153.36

Figure 70. ^{31}P NMR spectrum of complex **19**.

HBS-NG-2-45-77Se

42.815

NAME	HBS-NG-2-45-77Se
EXPNO	8
PROCNO	1
Date_	20120215
Time	20.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	65536
SOLVENT	CD3CN
NS	250
DS	4
SWH	326086.969 Hz
FIDRES	4.975692 Hz
AQ	0.1005385 sec
RG	645
DW	1.533 usec
DE	6.50 usec
TE	292.4 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========
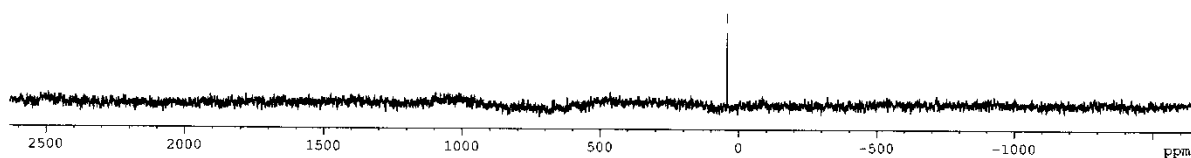| NUC1 | 77Se |
P1	10.00 usec
PL1	0.00 dB
SFO1	76.3490004 MHz
SI	65536
SF	76.3110246 MHz
WDW	EM
SSB	0
LB	60.00 Hz
GB	0
PC	0.00

Figure 71. ^{77}Se NMR spectrum of complex **19**.

Eager 300 Report

Page: 1   Sample: NG-2-45 (NG-2-45)

```
Method Name   : SD150612
Method File   : D:\CHNS2012\SD150612.mth
Chromatogram  : NG-2-45
Operator ID   : SHIKHA              Company  Name : C.E. Instruments
Analysed      : 06/15/2012  13:52   Printed       : 6/15/2012  15:07
Sample ID     : NG-2-45 (# 19)      Instrument N. : Instrument #1
Analysis Type : UnkNown (Area)      Sample weight : .843

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.
```

Element Name	%	Ret.Time	Area	BC	Area ratio	K factor
Nitrogen	8.7124	44	97229	RS	6.181736	.132383E+07
Carbon	28.4132	68	601044	RS	1.000000	.249735E+07
Hydrogen	2.1122	184	114035	RS	5.270698	.581082E+07
Totals	39.2377		812308			

C - 28.29
H - 2.37
N , 9.43

Figure 72. Elemental analysis of **19**.

Waters(Micromass) : Q-Tof micro(YA-105)   Dept. Of Chemistry - I.I.T.(B)   17-Aug-201211:09:55
C28H28F12N8P2PdSe4
HBS-NG-2-45 46 (0.461) AM (Top,5, Ht,5000.0,0.00,1.00); Sm (Md, 4.00); Sb (5,40.00); Cm (1:52)   TOF MS ES+
                                                                                                            3.05e3

100 — 239.2391
     343.1771
     345.1779
     347.1845 503.0938
     501.0959
     505.0956
     157.1445
     500.1044
     499.0901 556.5476   1045.2957
                                      1047.3162
                            897.2648
                      895.2509 1040.3131 1049.3041
                            894.2606   1051.2937
     149.0855   557.5432 892.2661   1053.3027
                                                  1259.1329 1323.4446
   0 —

   200   400   600   800   1000   1200   1400   1600   1800   m/z

Figure 73. Mass spectrum of complex **19**.
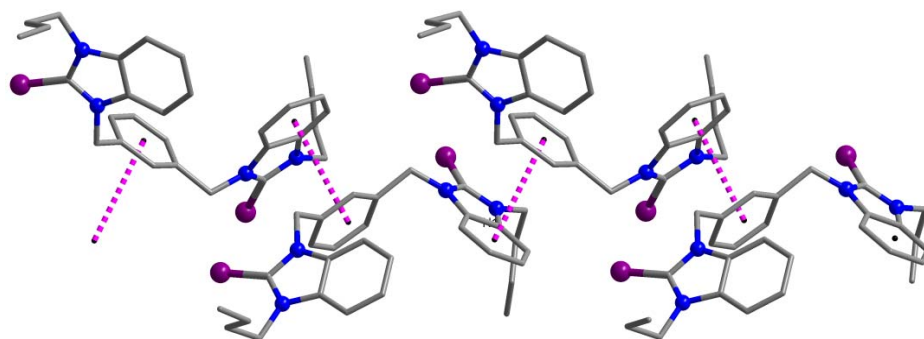
X-ray crystallography:

Figure 74. Packing diagram of compound **8**. Hydrogen atoms are omitted for clarity.
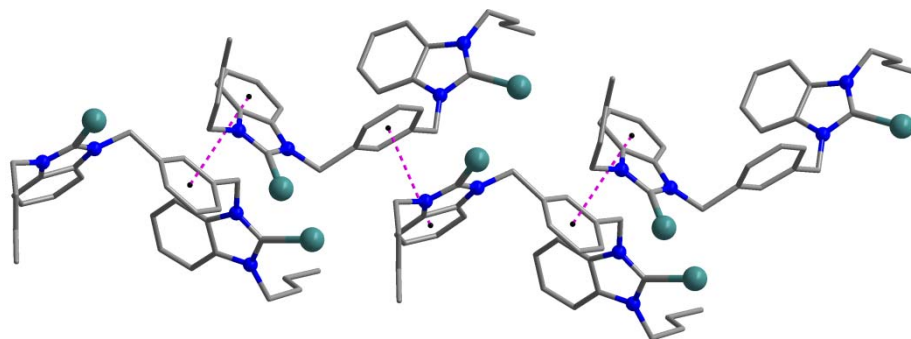

Figure 75. Packing diagram of compound **9.** Hydrogen atoms are omitted for clarity.
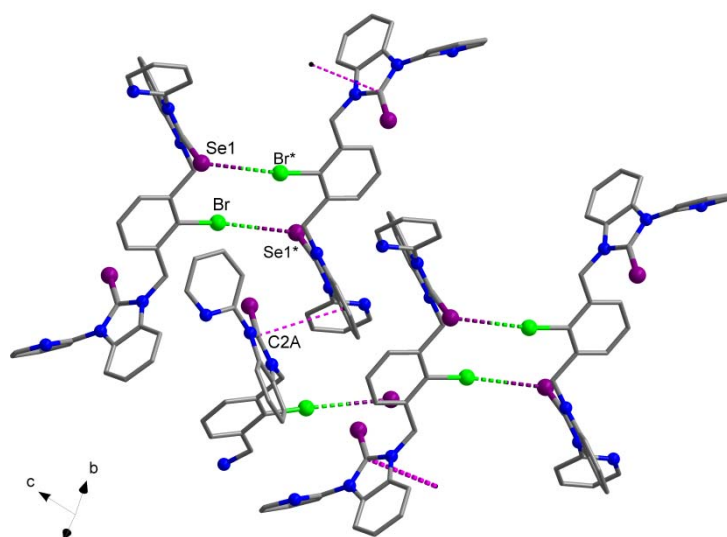

Figure 76. Packing diagram of compound **13.** Hydrogen atoms are omitted for clarity.

Table 1. Significant bond lengths [Å] and angles [°] for **8, 9** and **13**.

8

Se(1)-C(1)	1.833(4)	Se(2)-C(20)	1.842(5)
N(1)-C(1)	1.361(5)	N(2)-C(1)	1.358(5)
N(3)-C(20)	1.352(5)	C(20)-N(4A)	1.372(9)
N(1)-C(1)-Se(1)	126.6(3)	N(2)-C(1)-Se(1)	126.4(3)
N(3)-C(20)-Se(2)	125.5(3)	N(4B)-C(20)-Se(2)	131.0(4)

9

Te(1)-C(1)	2.087(10)	Te(2)-C(20)	2.073(9)
N(1)-C(1)	1.335(13)	N(2)-C(1)	1.342(12)
N(3)-C(20)	1.357(10)	N(4)-C(20)	1.346(13)
N(1)-C(1)-Te(1)	127.0(8)	N(2)-C(1)-Te(1)	124.6(7)
N(3)-C(20)-Te(2)	126.0(7)	N(4)-C(20)-Te(2)	127.5(6)

13

Se(1)-C(2B)	1.824(4)	Se(2)-C(2A)	1.836(4)
Br-C(1)	1.8865(16)		
N(1B)-C(2B)-Se(1)	126.8(3)	N(2B)-C(2B)-Se(1)	126.9(3)
N(1A)-C(2A)-Se(2)	126.1(3)	N(2A)-C(2A)-Se(2)	127.1(3)

Table 2. Significant bond lengths [Å] and angles [°] for **18**.

Pd-C(4)	2.014(4)	N(2)-C(4)	1.347(6)
Pd-C(10)	2.022(4)	N(2)-C(3)	1.382(6)
Pd-Se	2.3773(5)	N(2)-C(5)	1.414(6)
Pd-Cl	2.3967(10)	N(3)-C(13)	1.359(6)
Se-C(13)	1.862(4)	N(3)-C(9)	1.453(5)
N(1)-C(4)	1.341(6)	N(4)-C(13)	1.336(6)
N(1)-C(1)	1.465(6)	N(4)-C(14)	1.460(6)
C(4)-Pd-C(10)	82.01(18)	C(13)-Se-Pd	105.65(13)
C(4)-Pd-Se	176.36(13)	C(4)-N(2)-C(5)	118.9(4)
C(10)-Pd-Se	95.91(12)	C(13)-N(3)-C(9)	129.7(4)
C(4)-Pd-Cl	98.34(13)	C(9)-C(10)-Pd	134.0(3)
C(10)-Pd-Cl	178.57(12)	C(5)-C(10)-Pd	112.1(3)
Se-Pd-Cl	83.68(3)		

Table 3. Significant bond lengths [Å] and angles [°] for **19**.

Pd-Se(1)	2.4508(5)	N(1)-C(1)	1.336(7)
Pd-Se(1)#1	2.4508(5)	N(1)-C(2)	1.479(7)
Pd-Se(2)#1	2.4553(5)	N(2)-C(1)	1.360(7)
Pd-Se(2)	2.4553(5)	N(3)-C(13)	1.368(6)
Se(1)-C(1)	1.879(5)	N(4)-C(13)	1.341(6)
Se(2)-C(13)	1.877(5)	N(4)-C(14)	1.467(7)
Se(1)-Pd-Se(1)#1	180.000(1)	C(1)-Se(1)-Pd	101.33(15)
Se(1)-Pd-Se(2)#1	91.496(18)	C(13)-Se(2)-Pd	103.41(14)
Se(1)#1-Pd-Se(2)#1	88.504(18)	C(1)-N(1)-C(2)	124.9(5)
Se(1)-Pd-Se(2)	88.504(18)	C(13)-N(4)-C(14)	125.7(4)
Se(1)#1-Pd-Se(2)	91.496(18)	N(1)-C(1)-N(2)	106.9(4)
Se(2)#1-Pd-Se(2)	180.0	N(4)-C(13)-N(3)	106.6(4)