SUPPORTING INFORMATION FOR

Coinage metal complexes of selenoureas derived from N-heterocyclic carbenes

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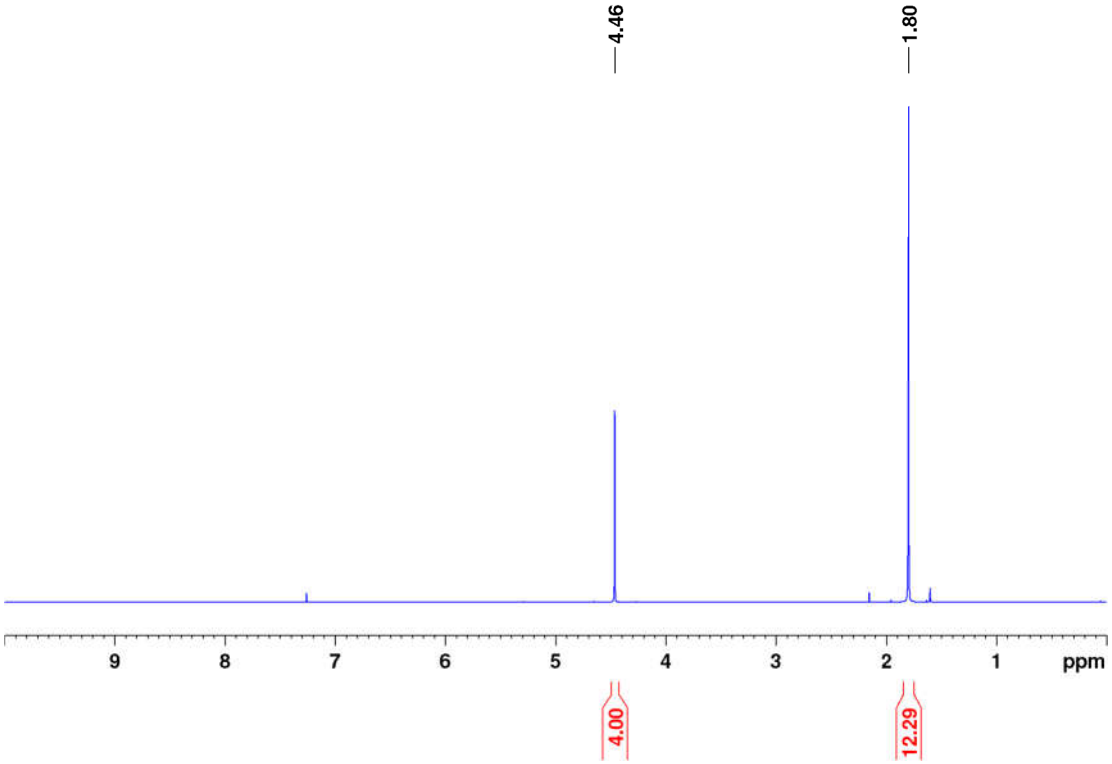
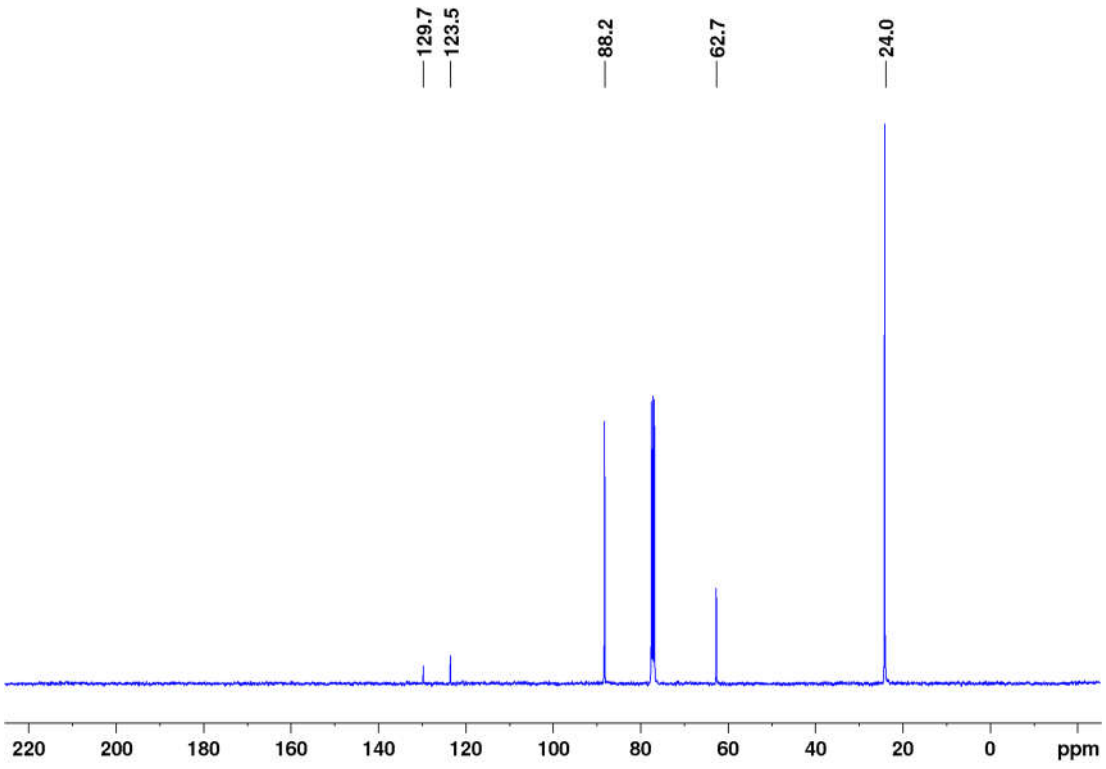
CONTENTS

[Se(IBioxMe$_4$)] (**11**)

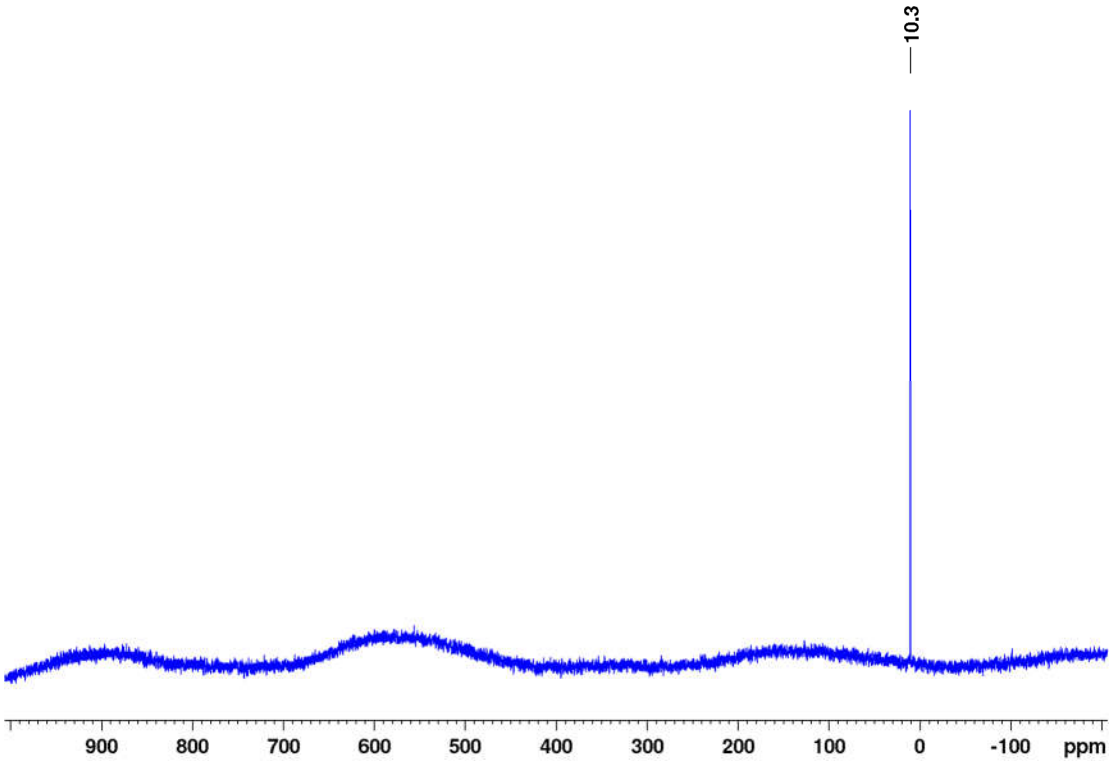
^{1}H NMR



^{13}C{^{1}H} NMR

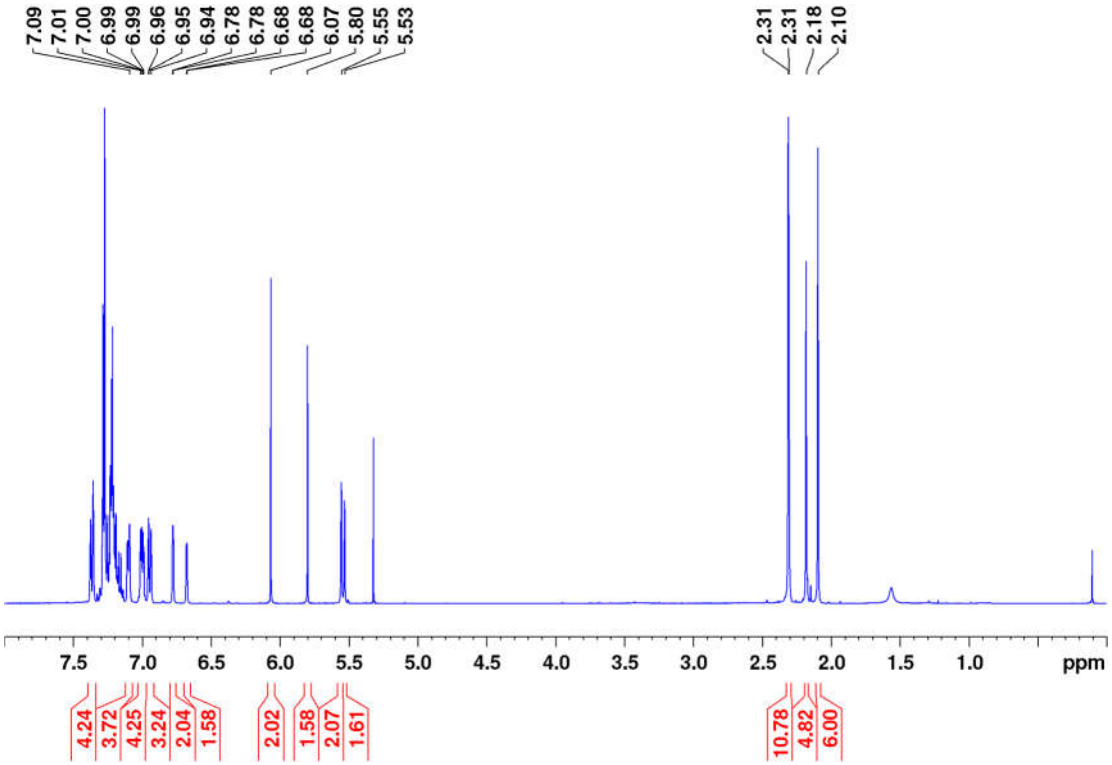


^{77}Se NMR

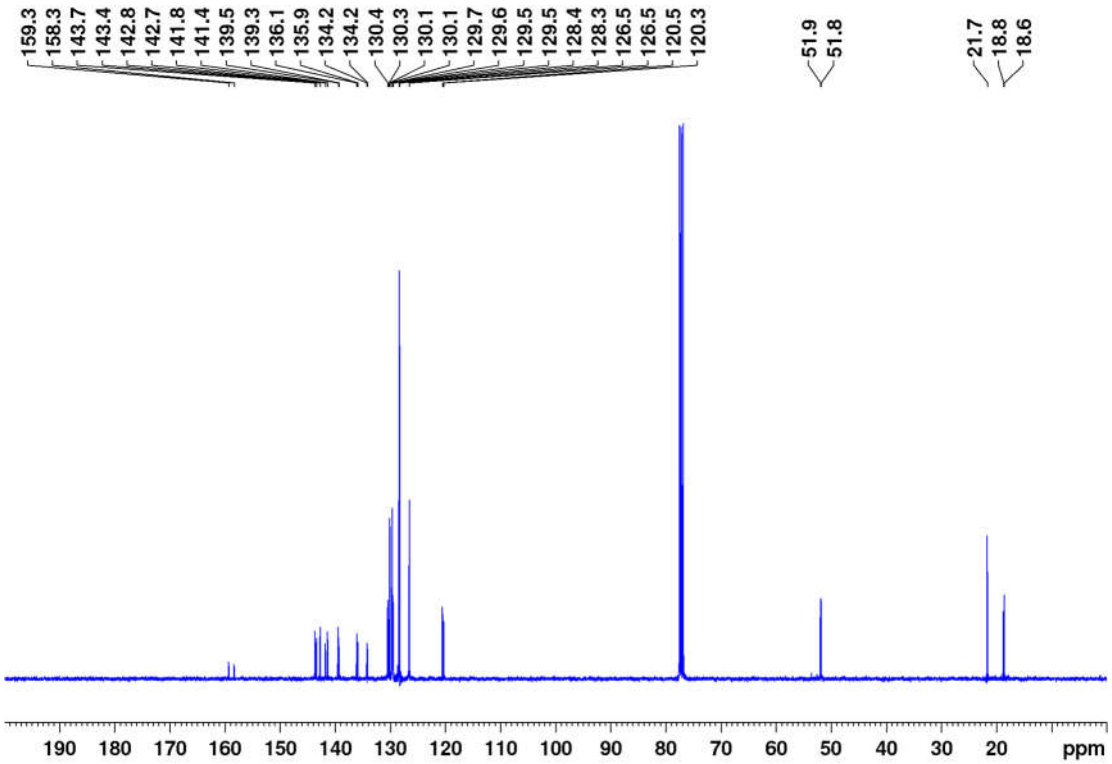


[Se(IPaul)] (**12**)

^{1}H NMR



$^{13}C\{^{1}H\}$ NMR



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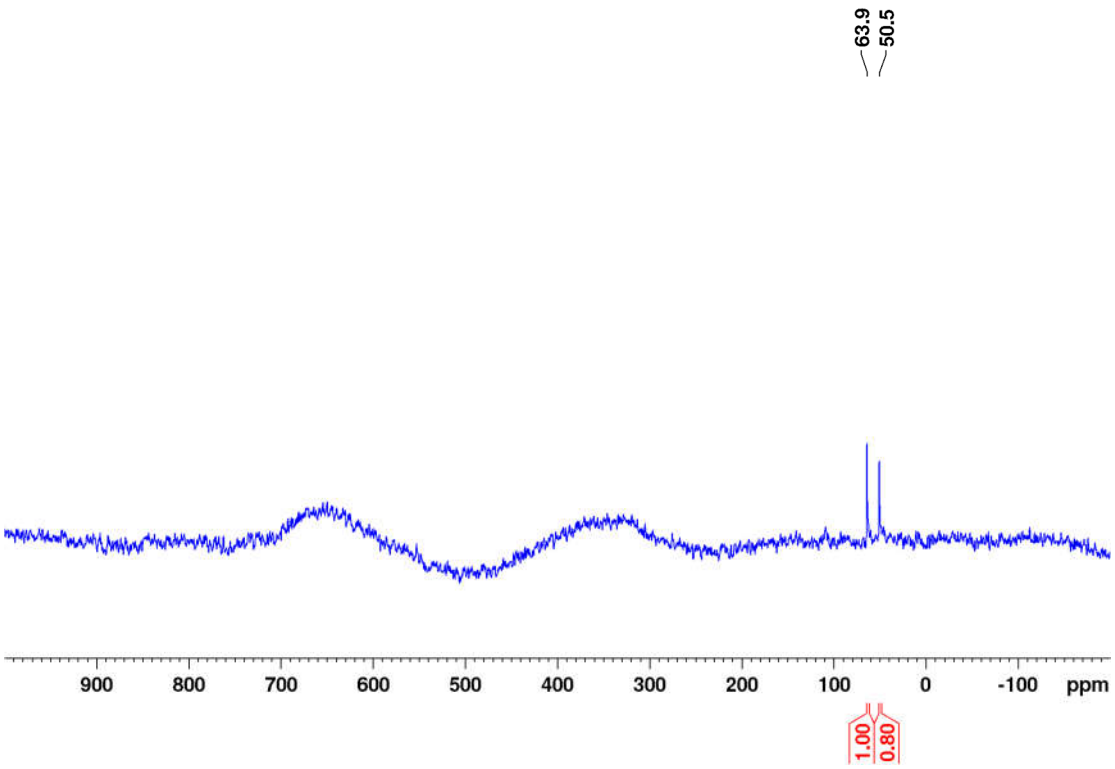
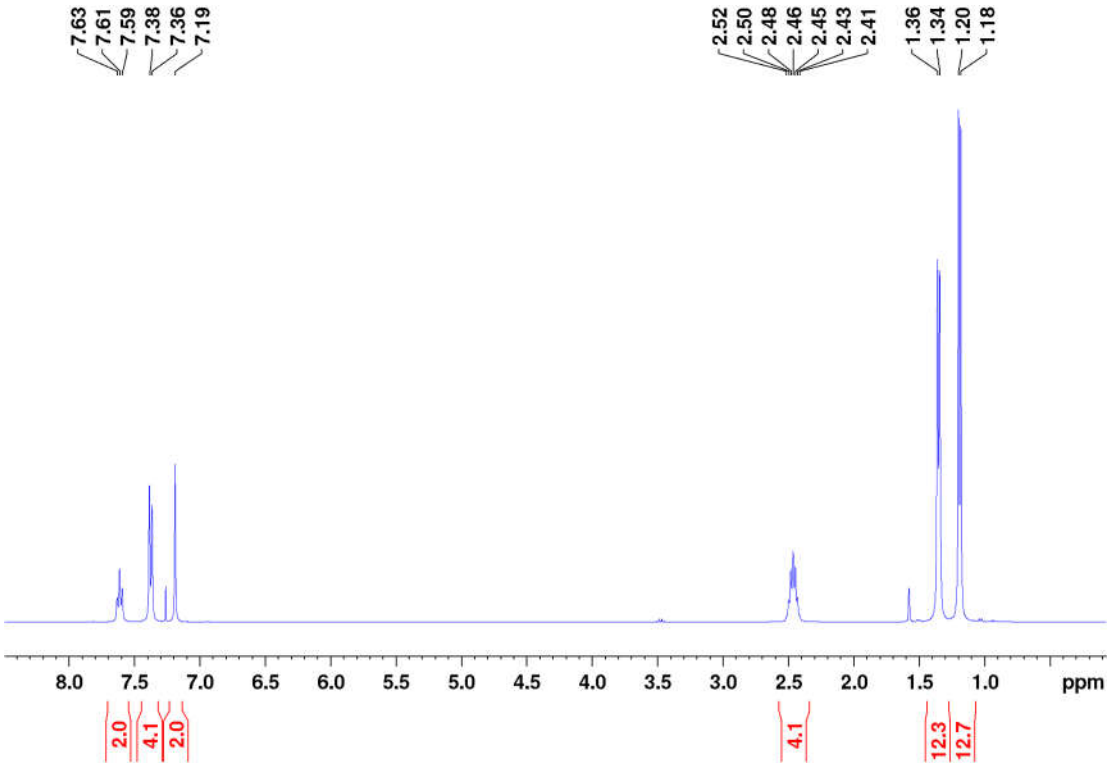
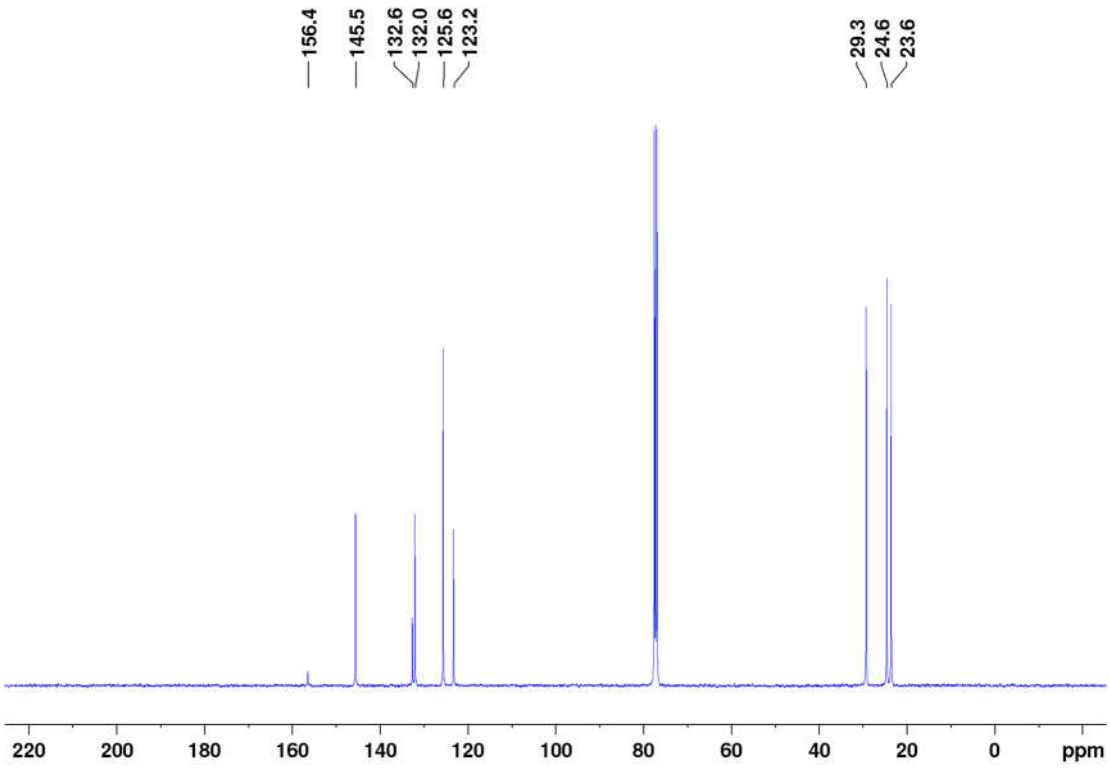


[CuCl(1)]

^{1}H NMR



^{13}C{^{1}H} NMR



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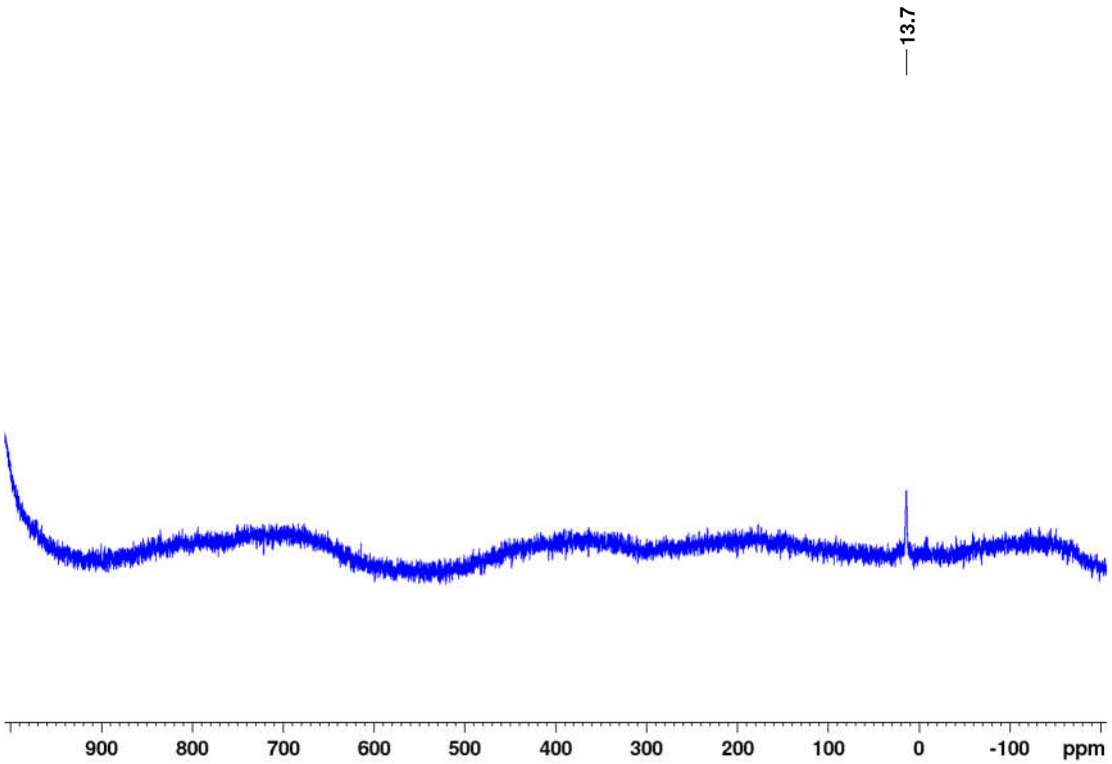

[CuCl(2))]

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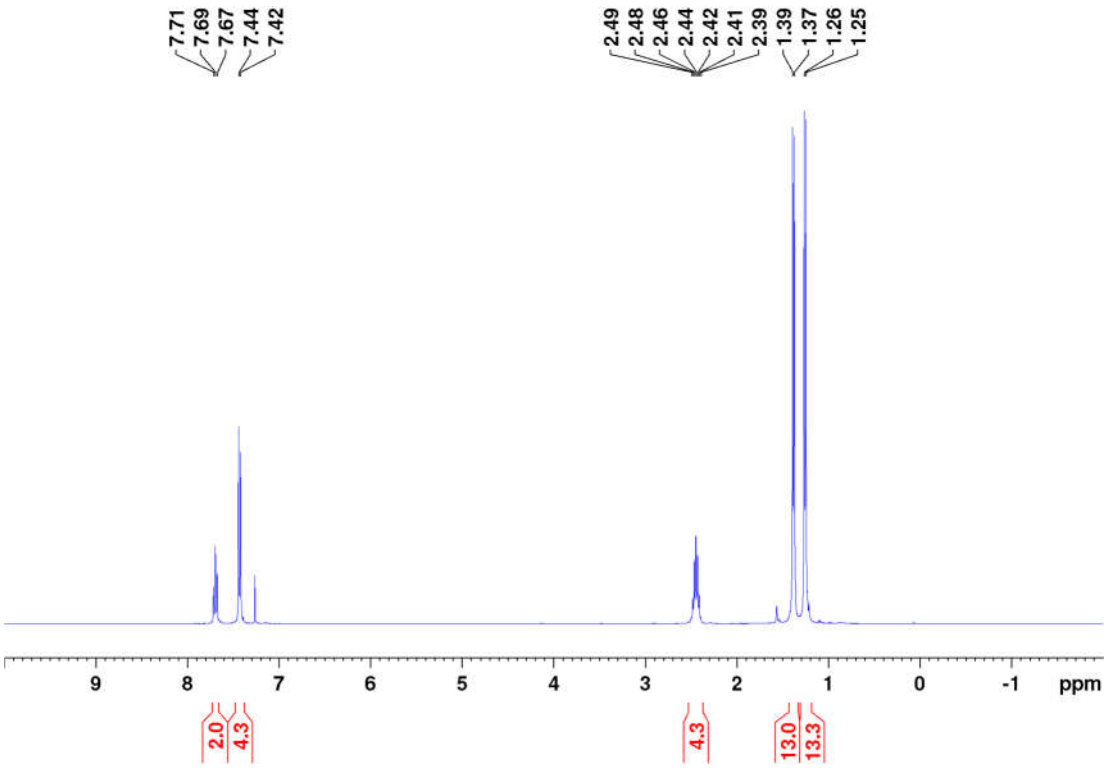

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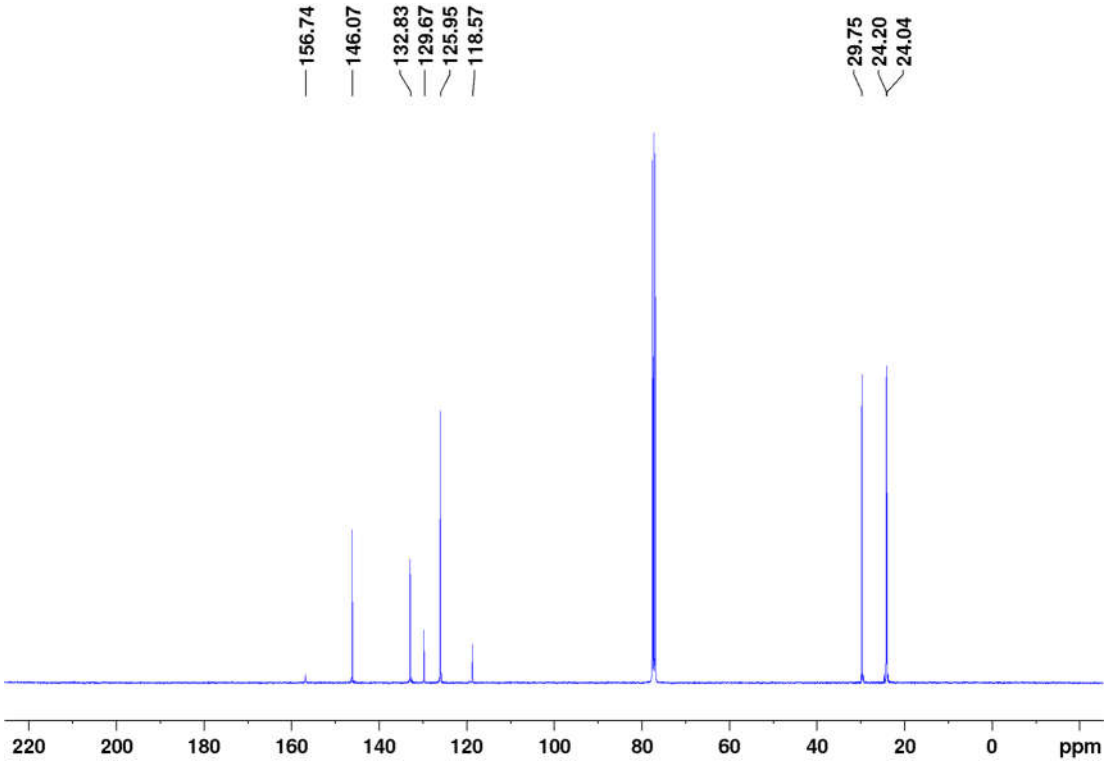

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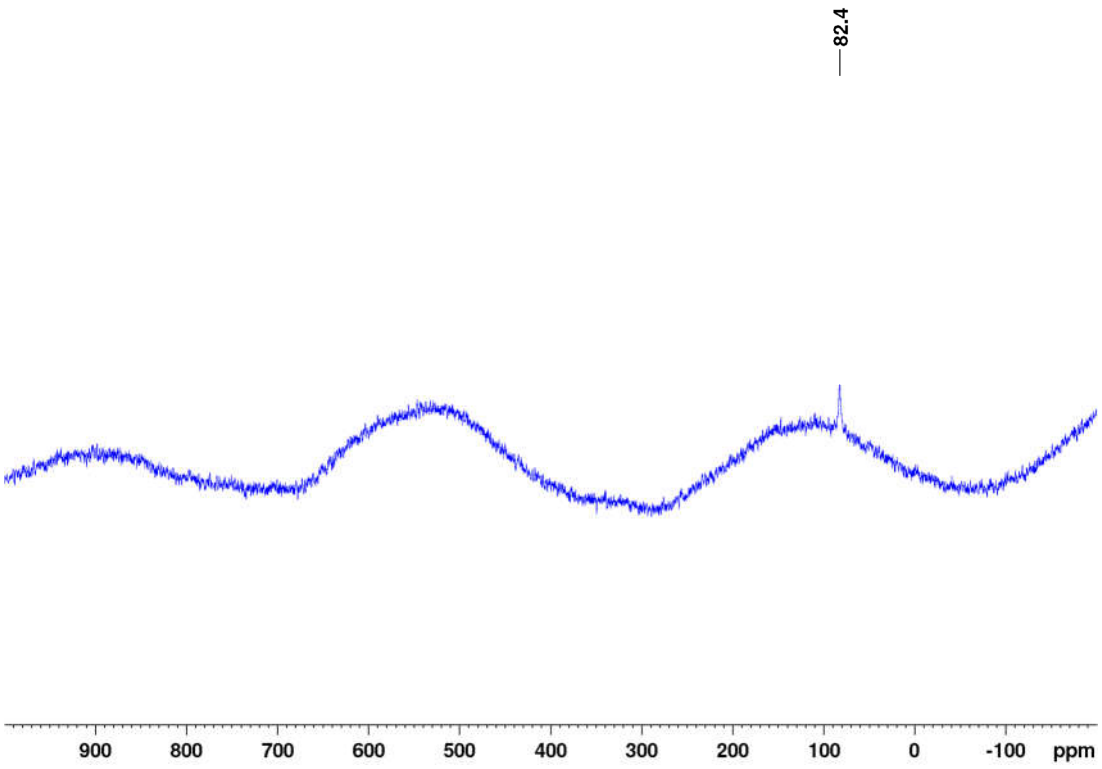

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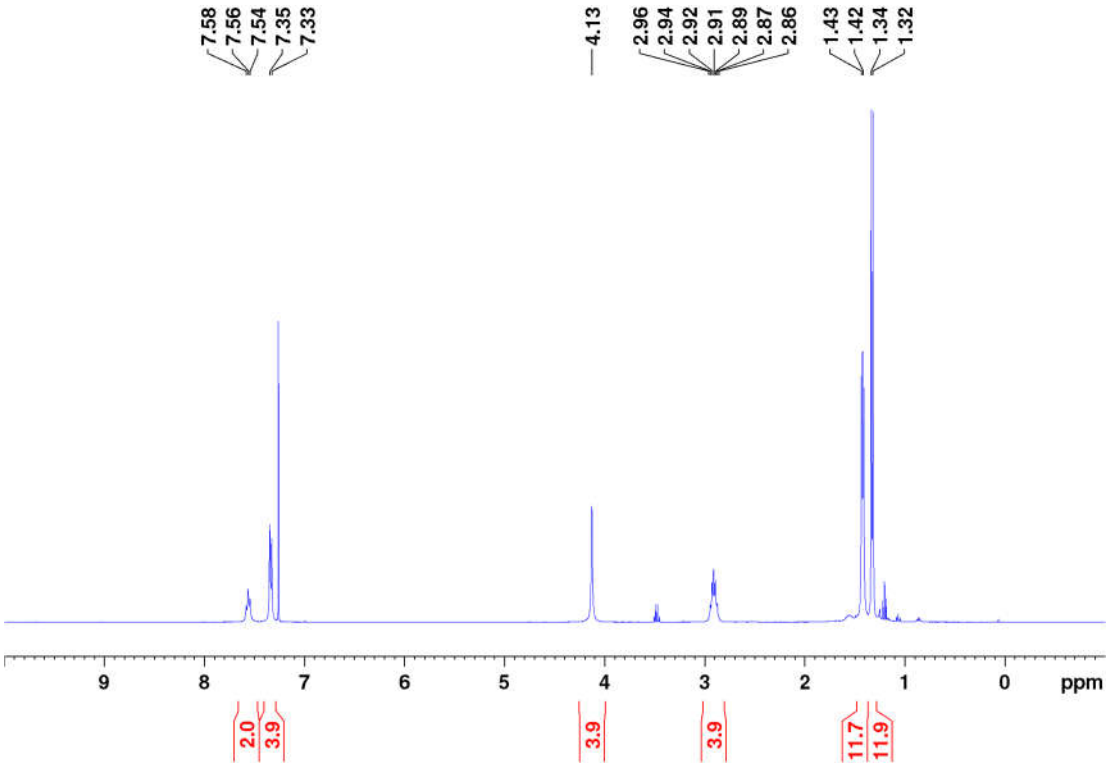
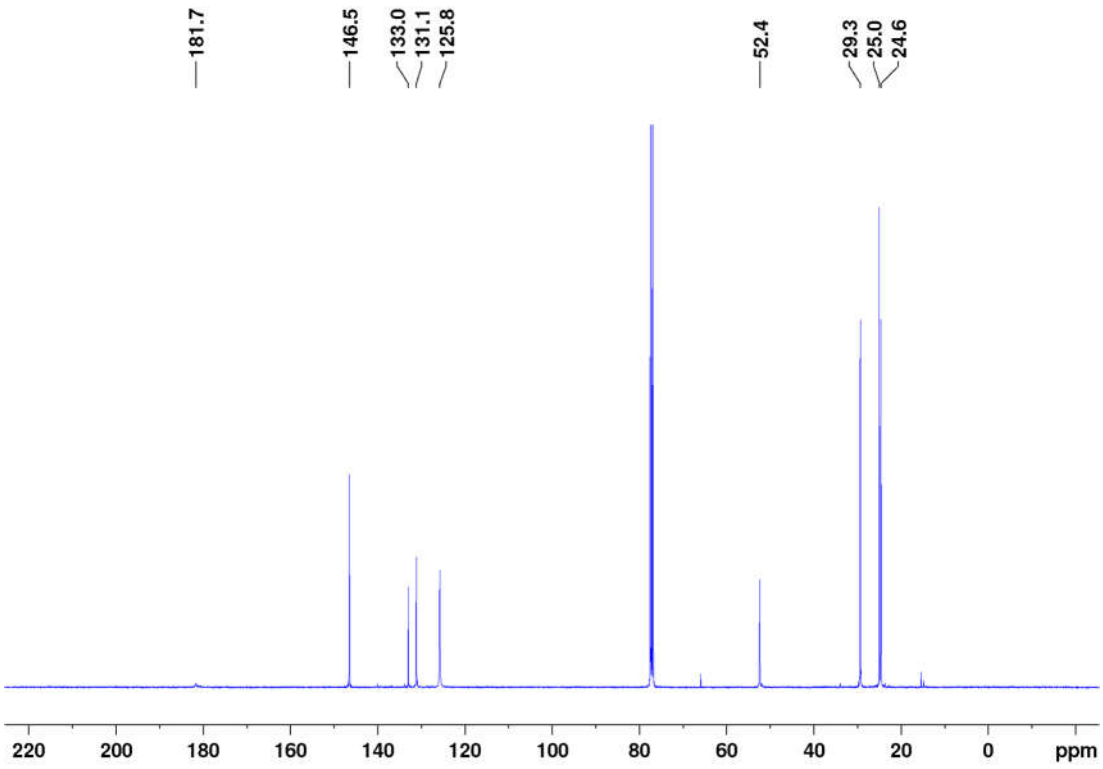
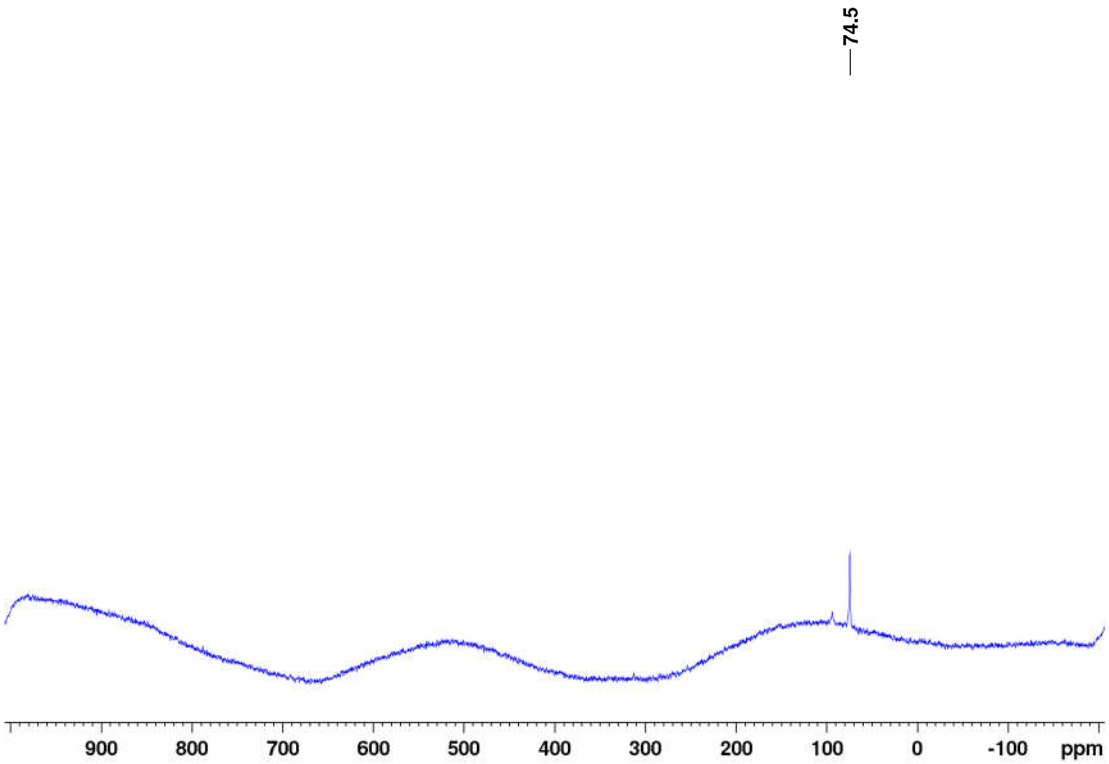


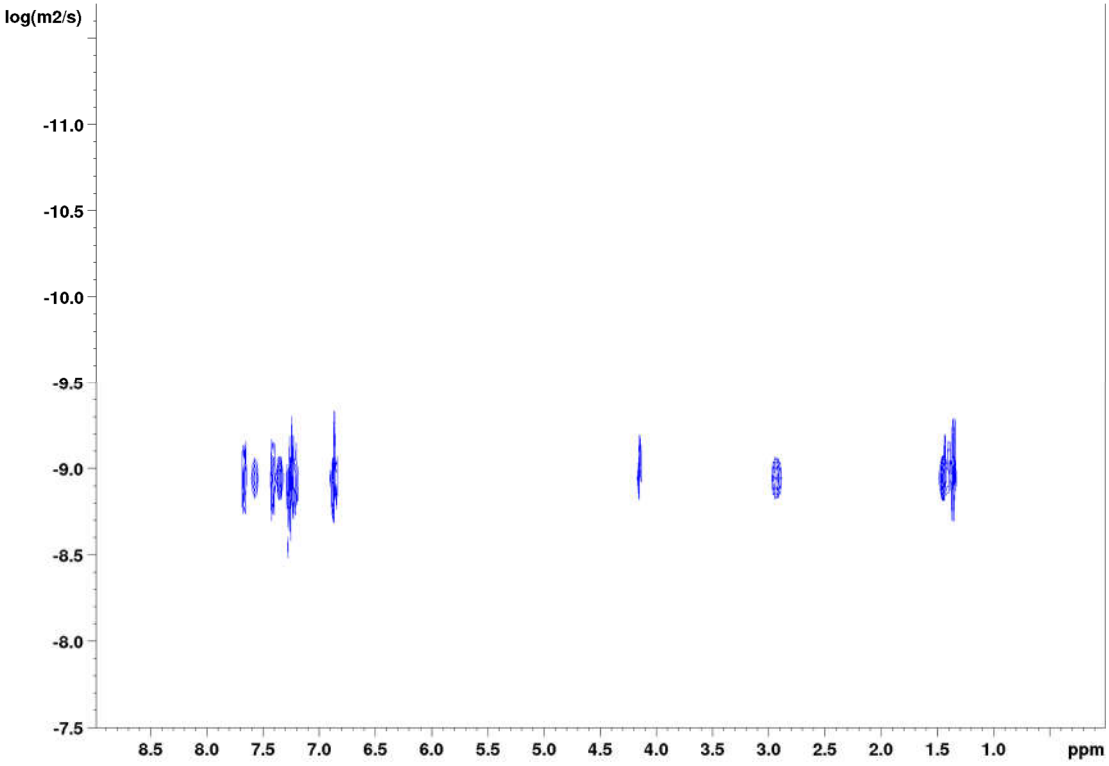
^{13}C{^{1}H} NMR



^{77}Se NMR



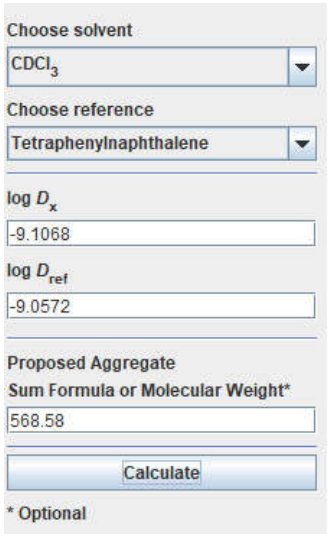
2D DOSY NMR



logD (tetraphenylnaphthalene) = -9.0572

logD (analyte) = -9.1068

Mass of [CuCl(**3**)] = 568.58 g mol^{-1}



Solvent = $CDCl_3$

Reference = Tetraphenylnaphthalene

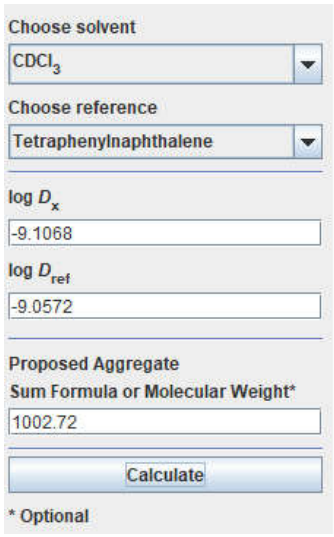
$\log D_{x,norm}$ = -9.1662 g/mol

MW_{calc} = 569 g/mol

ECC	MW_{det}	MW_{dif}	Within expected error interval: Empirical	Within expected error interval: Theoretical
CS	798 g/mol	-29 %		
Merge	649 g/mol	-12 %	✓	✓
DSE	570 g/mol	0 %	✓	✓
ED	506 g/mol	12 %		✓

Date: Sun Apr 15 06:27:55 BST 2018

Mass of [Cu(**3**)$_2$]$^+$ = 834.39 g mol^{-1}



Solvent = $CDCl_3$

Reference = Tetraphenylnaphthalene

$\log D_{x,norm}$ = -9.1662 g/mol

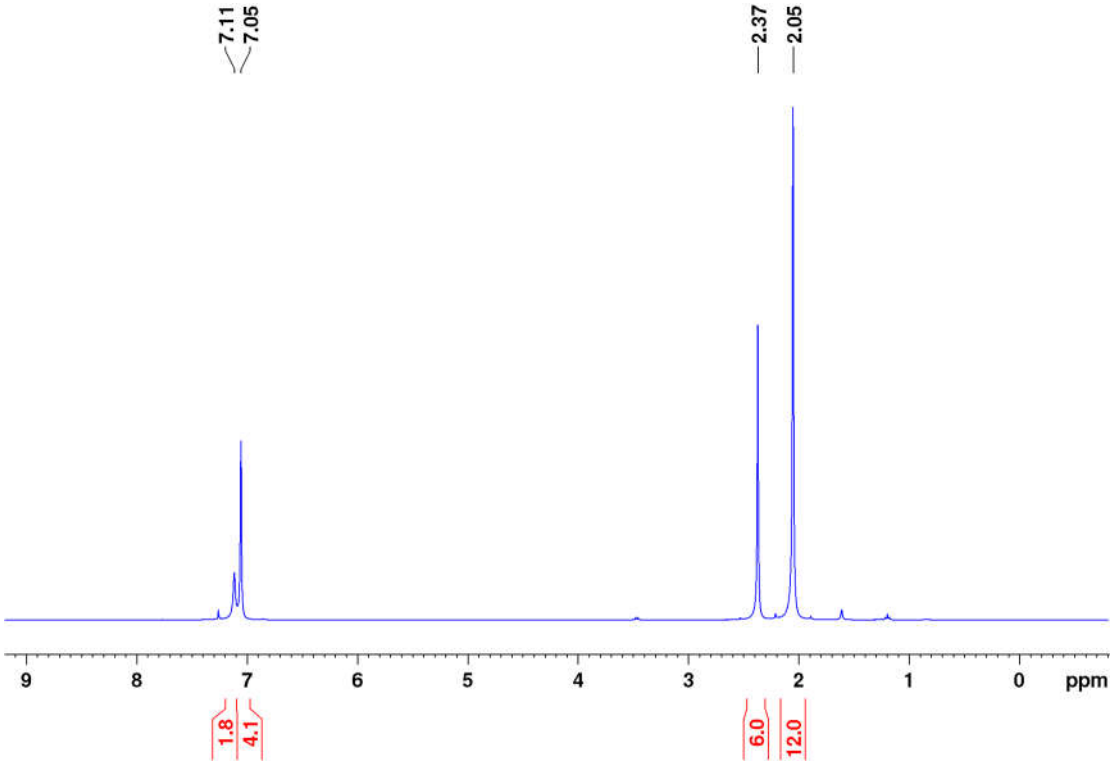
MW_{calc} = 1003 g/mol

ECC	MW_{det}	MW_{dif}	Within expected error interval: Empirical	Within expected error interval: Theoretical
CS	798 g/mol	26 %		
Merge	649 g/mol	55 %		
DSE	570 g/mol	76 %		
ED	506 g/mol	98 %		

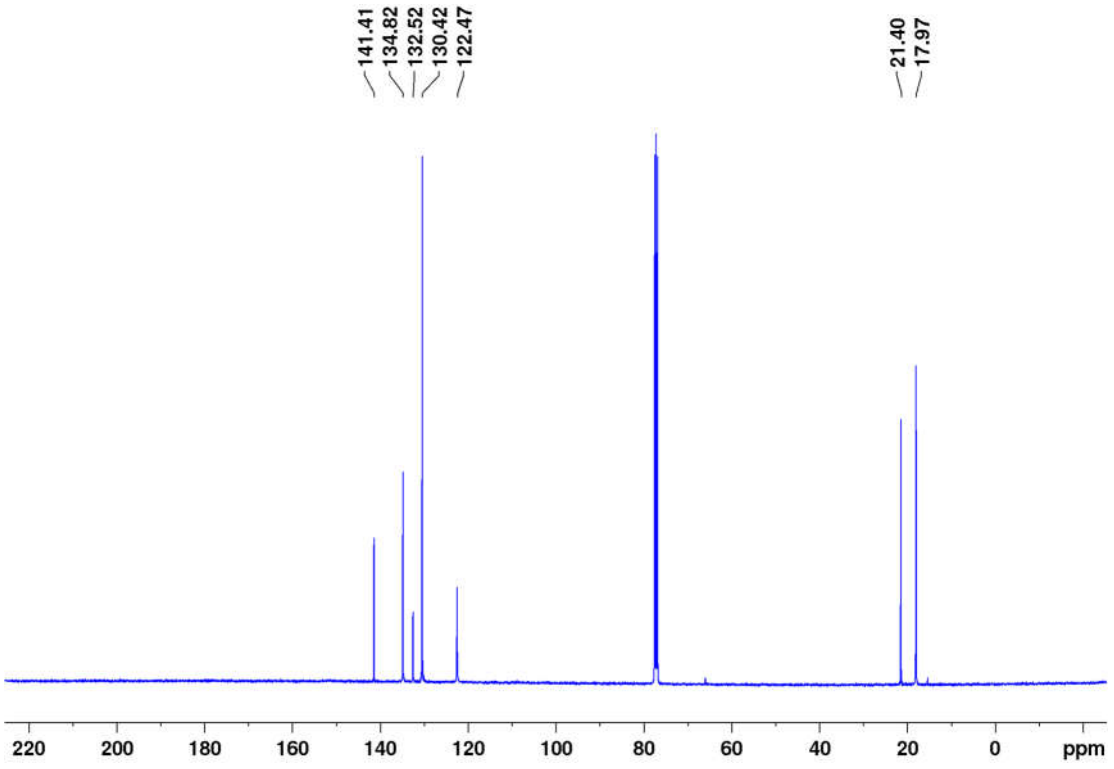
Date: Sun Apr 15 06:28:42 BST 2018

[CuCl(4)]

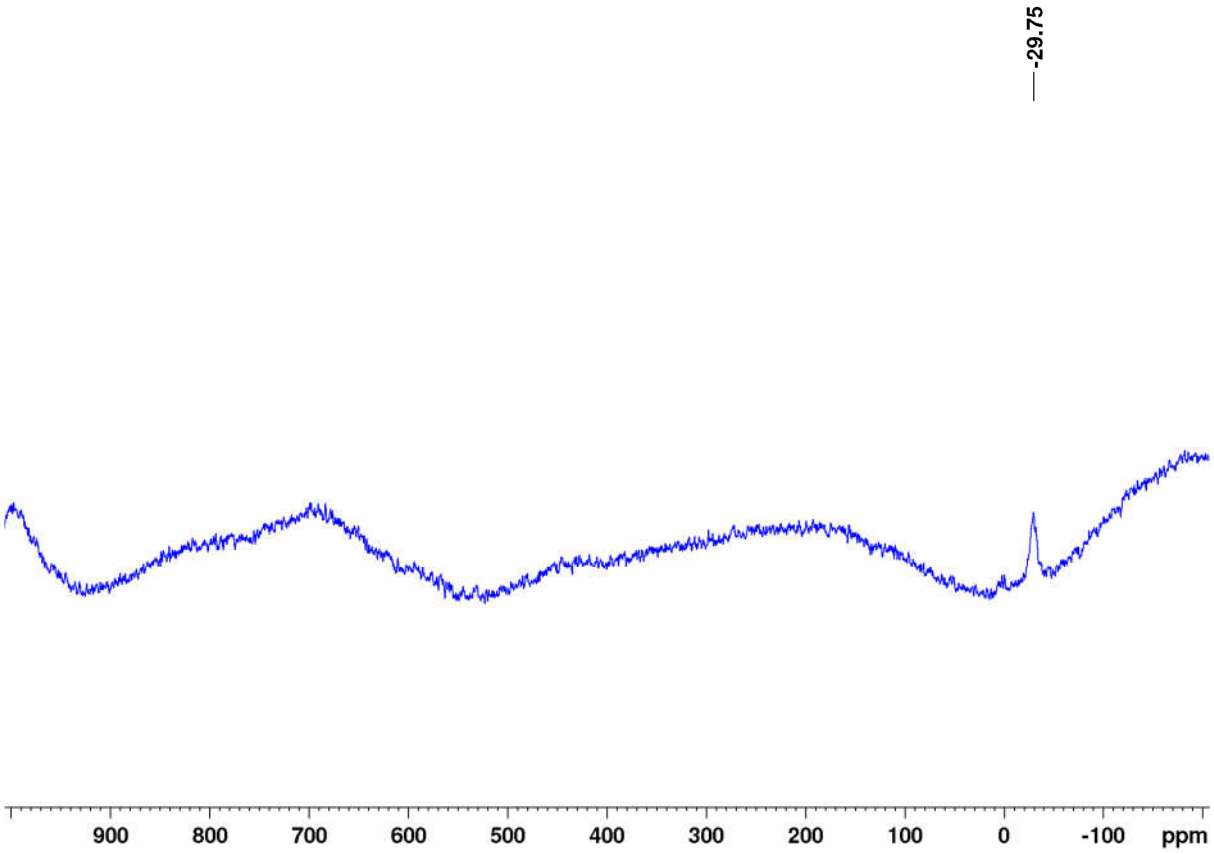
^{1}H NMR



^{13}C{^{1}H} NMR

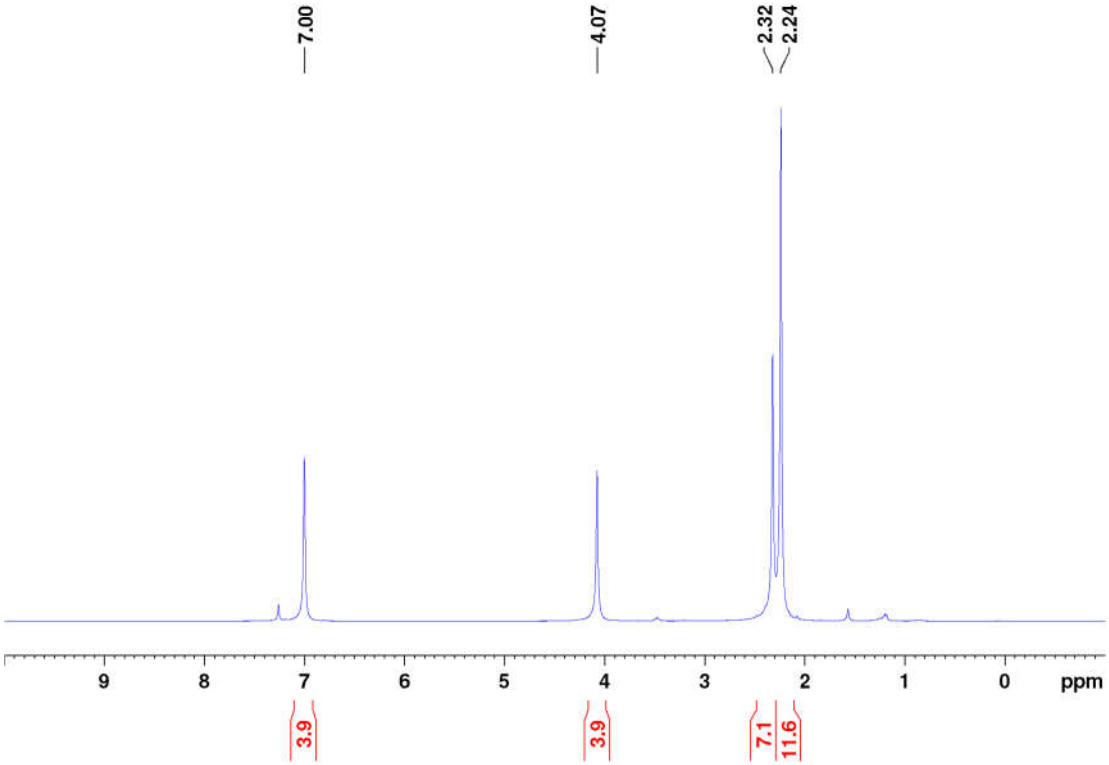


^{77}Se NMR

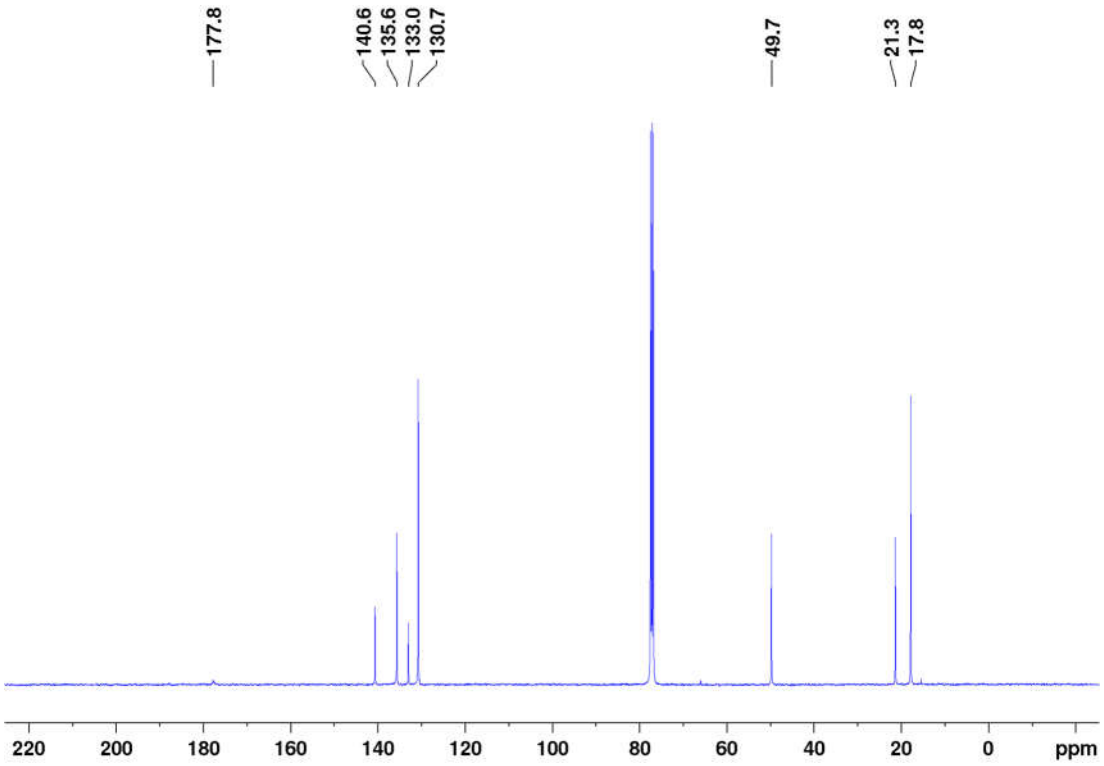

-29.75
900
800
700
600
500
400
300
200
100
0
-100
ppm

[CuCl(5)]

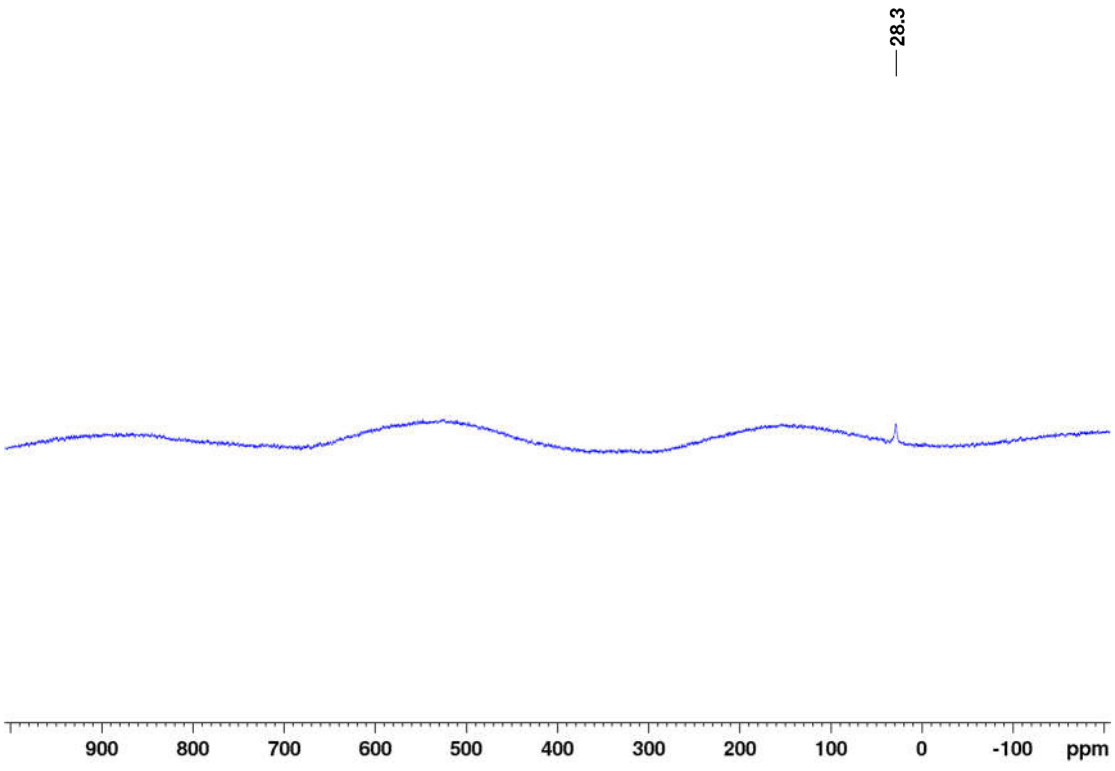
^{1}H NMR


7.00
4.07
2.32
2.24
9
8
7
6
5
4
3
2
1
0
ppm
3.9
3.9
7.1
11.6

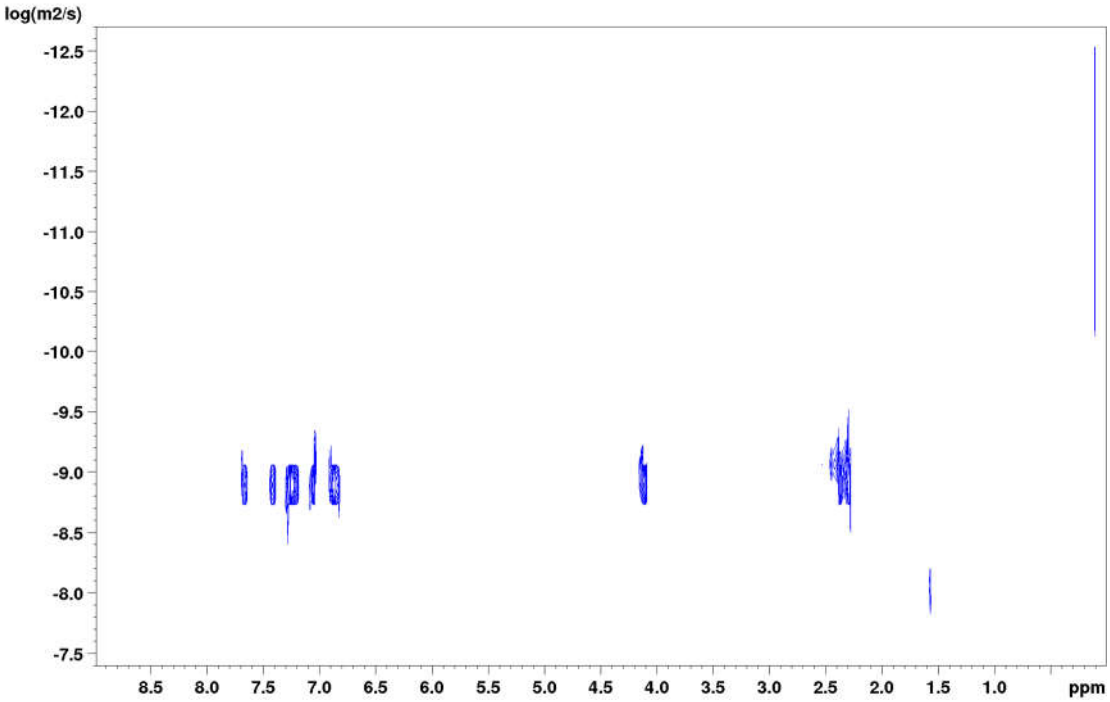
$^{13}C\{^{1}H\}$ NMR



^{77}Se NMR



2D DOSY NMR



logD (tetraphenylnaphthalene) = -9.0781

logD (analyte) = -9.1128

Mass of [CuCl(**5**)] = 484.42 g mol^{-1}

Choose solvent

$CDCl_3$

Choose reference

Tetraphenylnaphthalene

log D_x

-9.1128

log D_{ref}

-9.0781

Proposed Aggregate

Sum Formula or Molecular Weight*

484.42

Calculate

* Optional

Solvent = $CDCl_3$

Reference = Tetraphenylnaphthalene

log$D_{x,norm}$ = -9.1513 g/mol

MW_{calc} = 484 g/mol

			Within expected error interval	
ECC	MW_{det}	MW_{dif}	Empirical	Theoretical
CS	740 g/mol	-35 %		
Merge	609 g/mol	-20 %	✓	
DSE	536 g/mol	-10 %	✓	✓
ED	483 g/mol	0 %	✓	✓

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Mass of $[Cu(\mathbf{5})_2]^+$ = 834.39 g mol^{-1}

Choose solvent

$CDCl_3$

Choose reference

Tetraphenylnaphthalene

log D_x

-9.1128

log D_{ref}

-9.0781

Proposed Aggregate

Sum Formula or Molecular Weight*

834.39

Calculate

* Optional

Solvent = $CDCl_3$

Reference = Tetraphenylnaphthalene

log$D_{x,norm}$ = -9.1513 g/mol

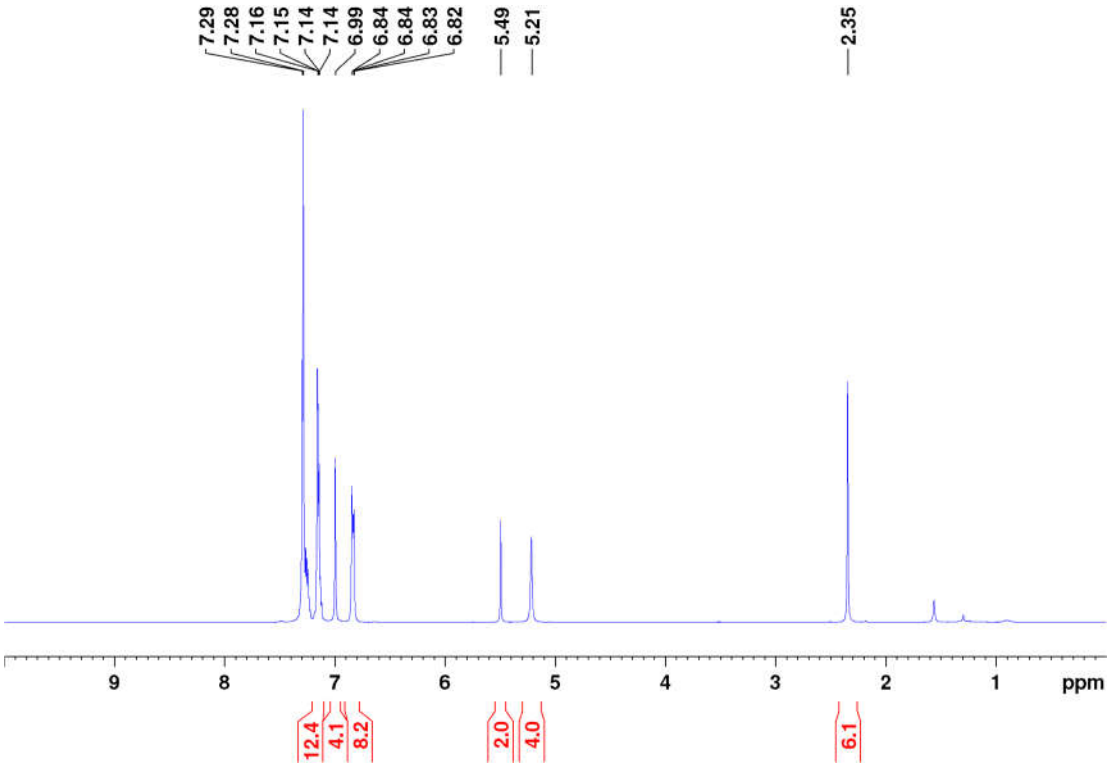
MW_{calc} = 834 g/mol

			Within expected error interval	
ECC	MW_{det}	MW_{dif}	Empirical	Theoretical
CS	740 g/mol	13 %	✓	✓
Merge	609 g/mol	37 %		
DSE	536 g/mol	56 %		
ED	483 g/mol	73 %		

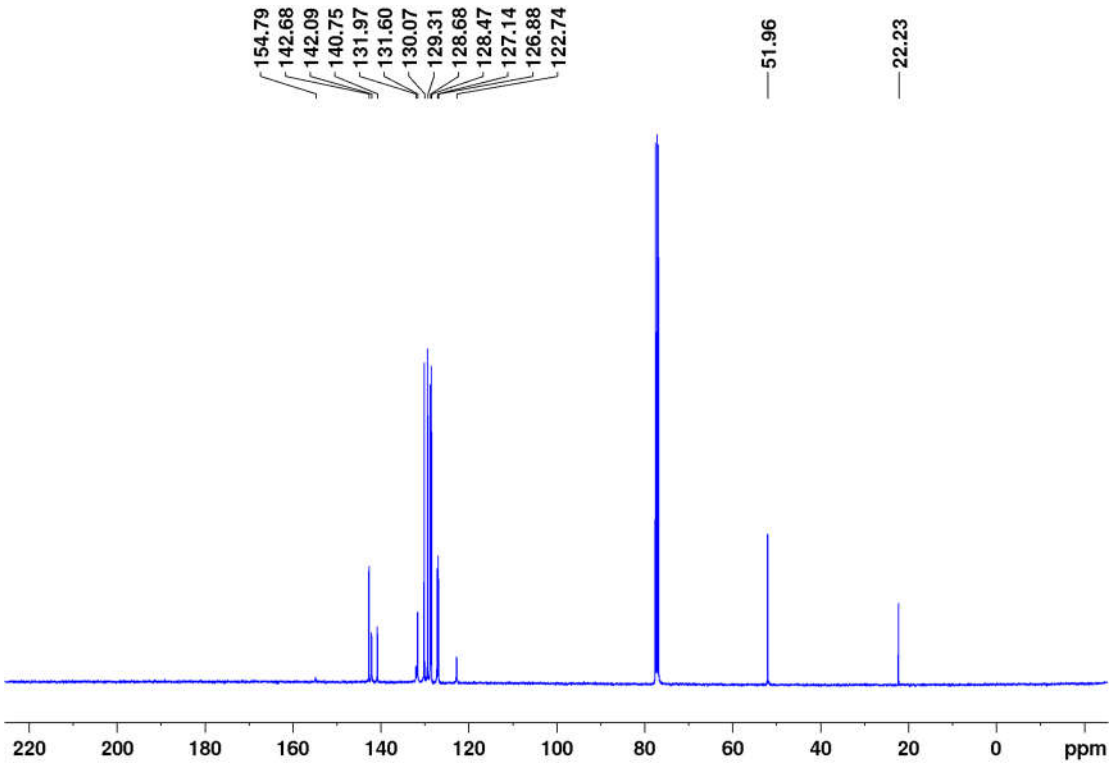
Date: Sun Apr 15 06:19:39 BST 2018

[CuCl(6)]

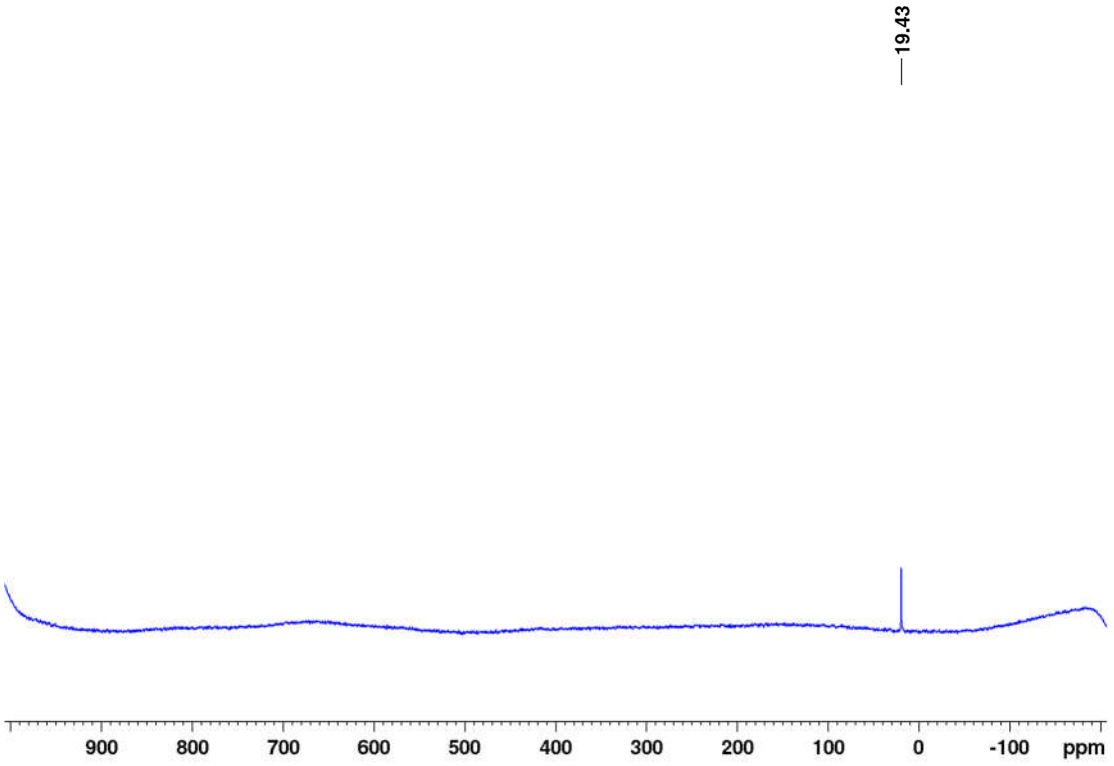
^{1}H NMR



$^{13}C\{^{1}H\}$ NMR

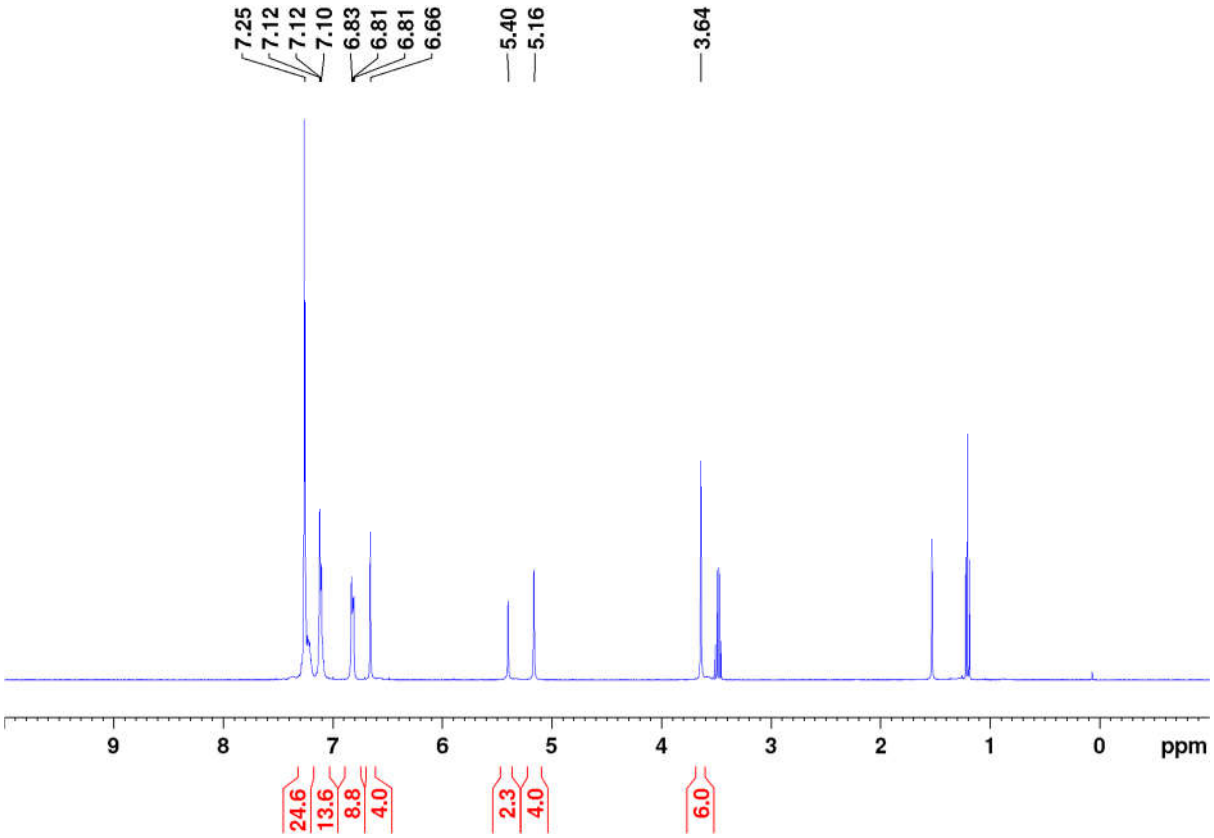


^{77}Se NMR

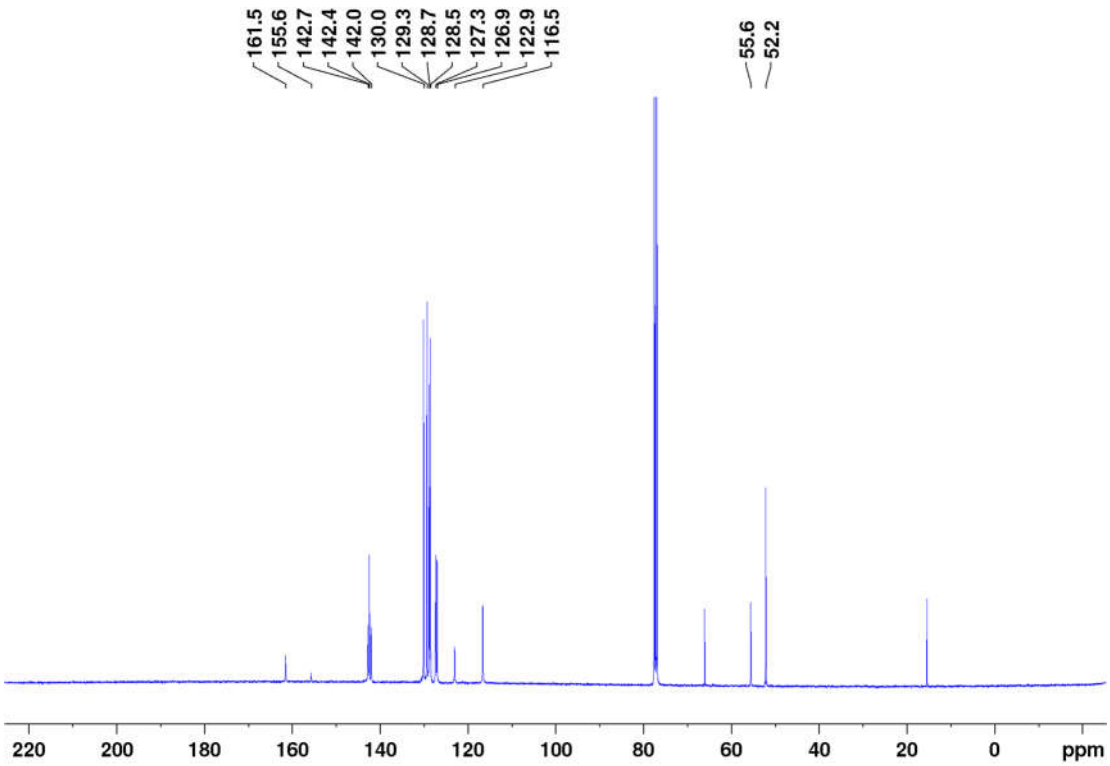


[CuCl(7)]

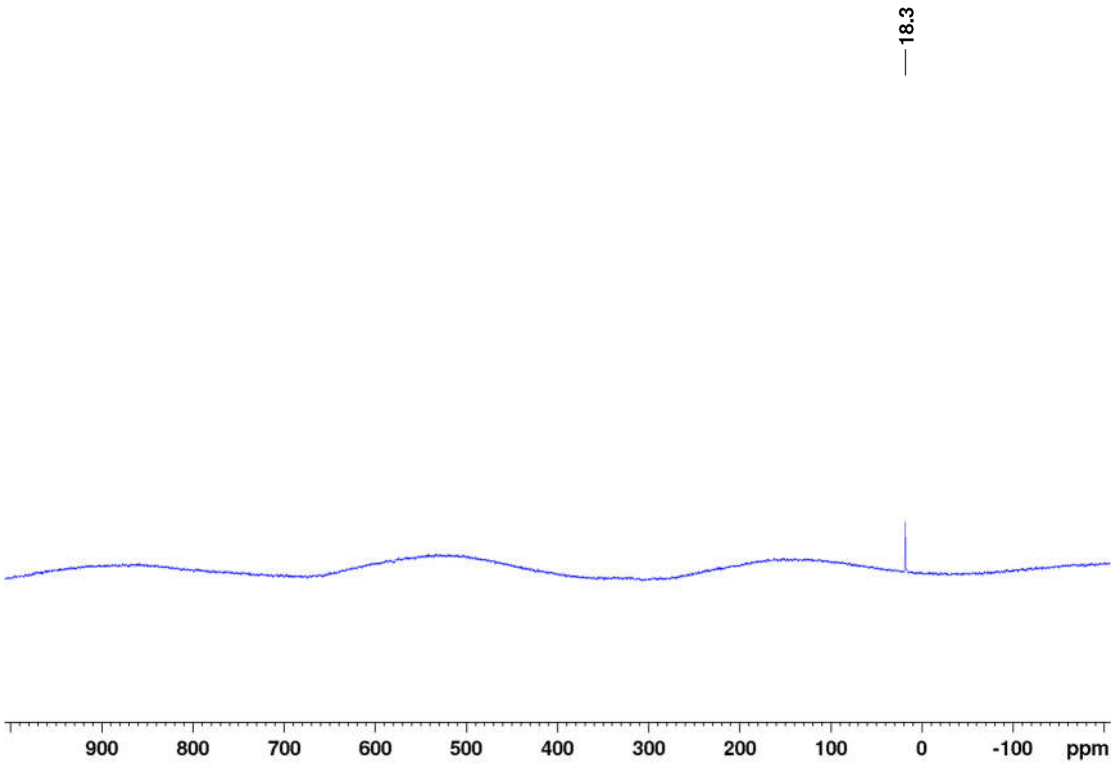
^{1}H NMR



^{13}C{^{1}H} NMR

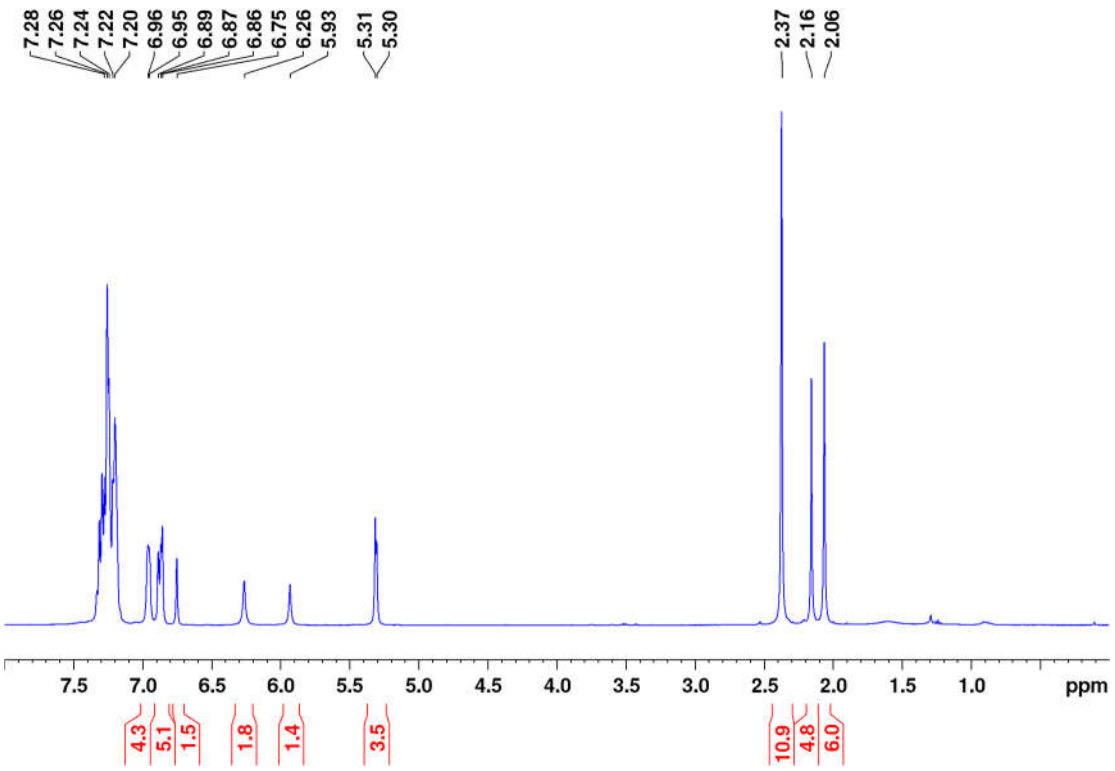


^{77}Se NMR

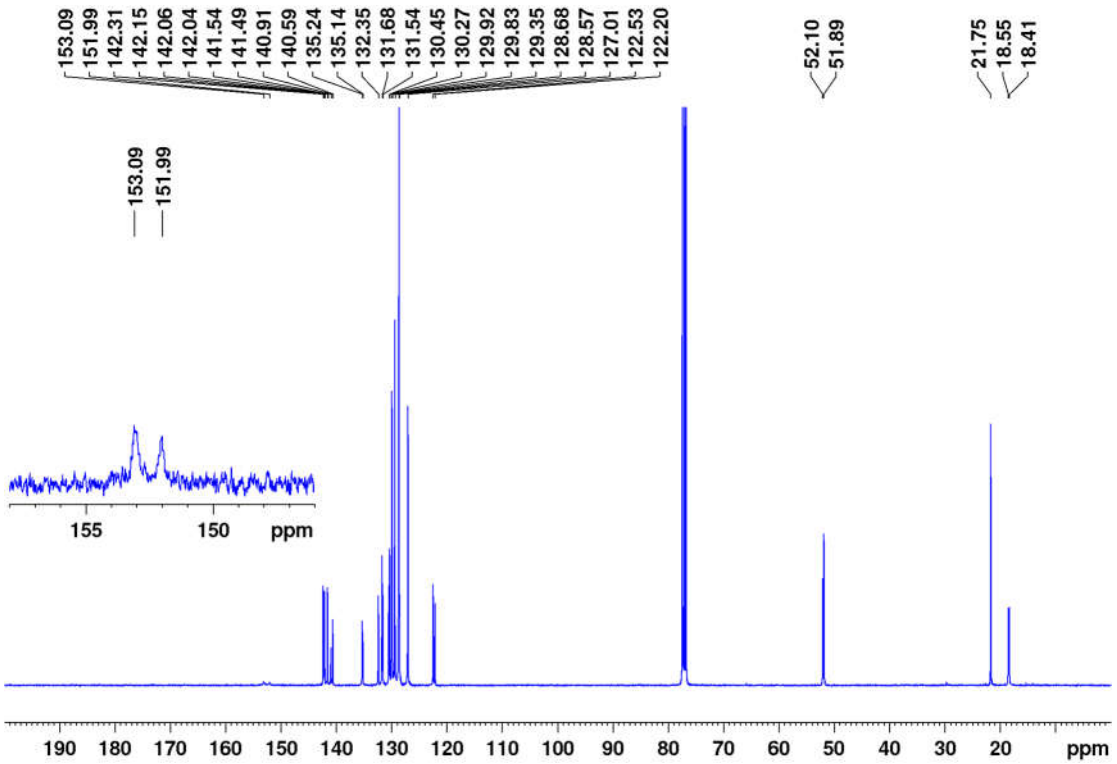


[CuCl(12)]

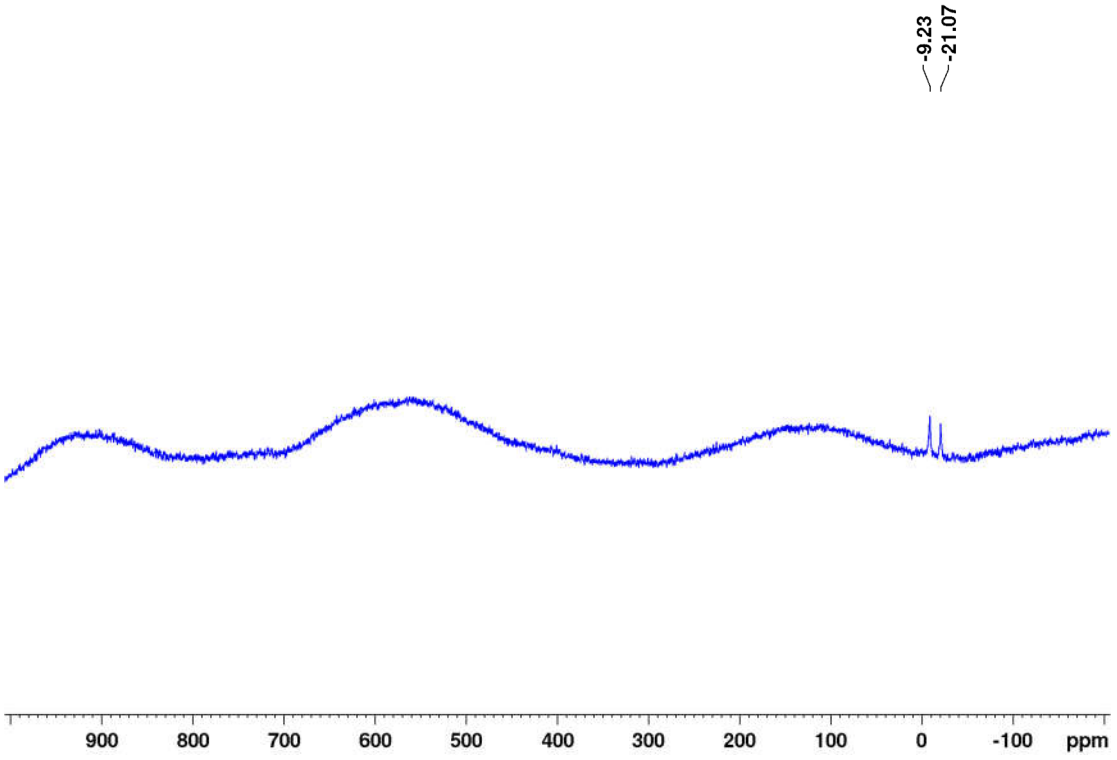
^{1}H NMR



$^{13}C\{^{1}H\}$ NMR

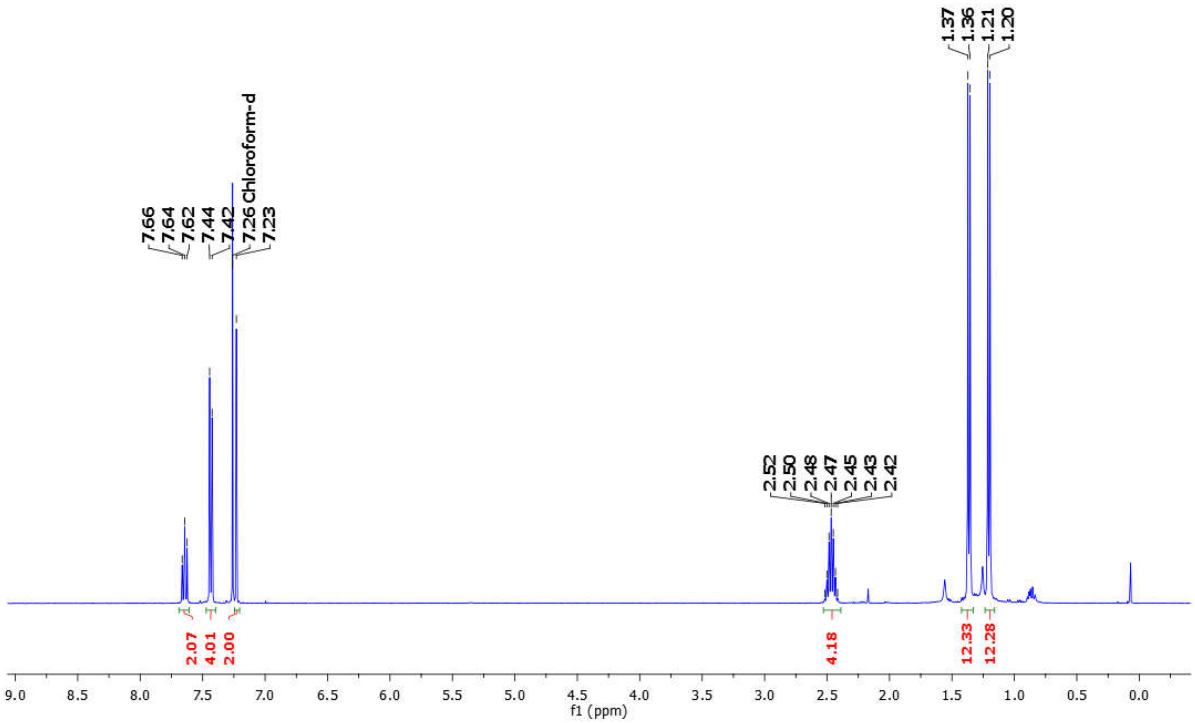


^{77}Se NMR

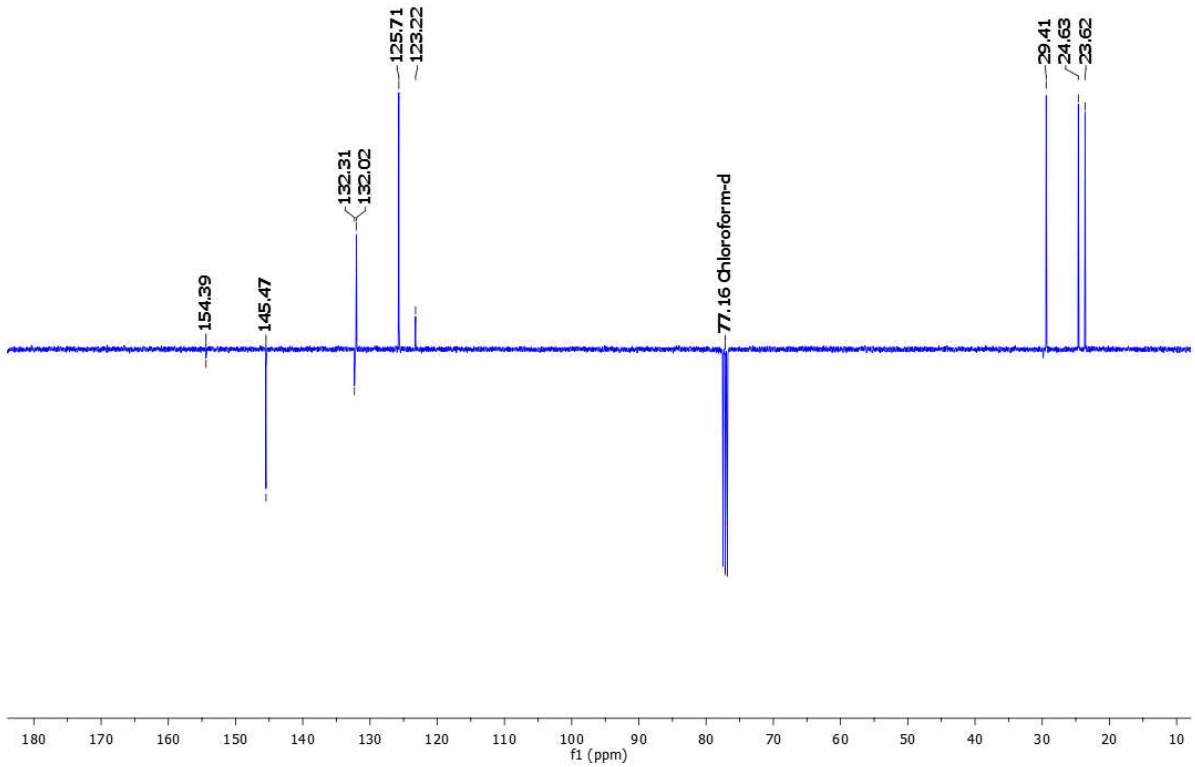


$[Ag(1)_2][NO_3]$

^{1}H NMR

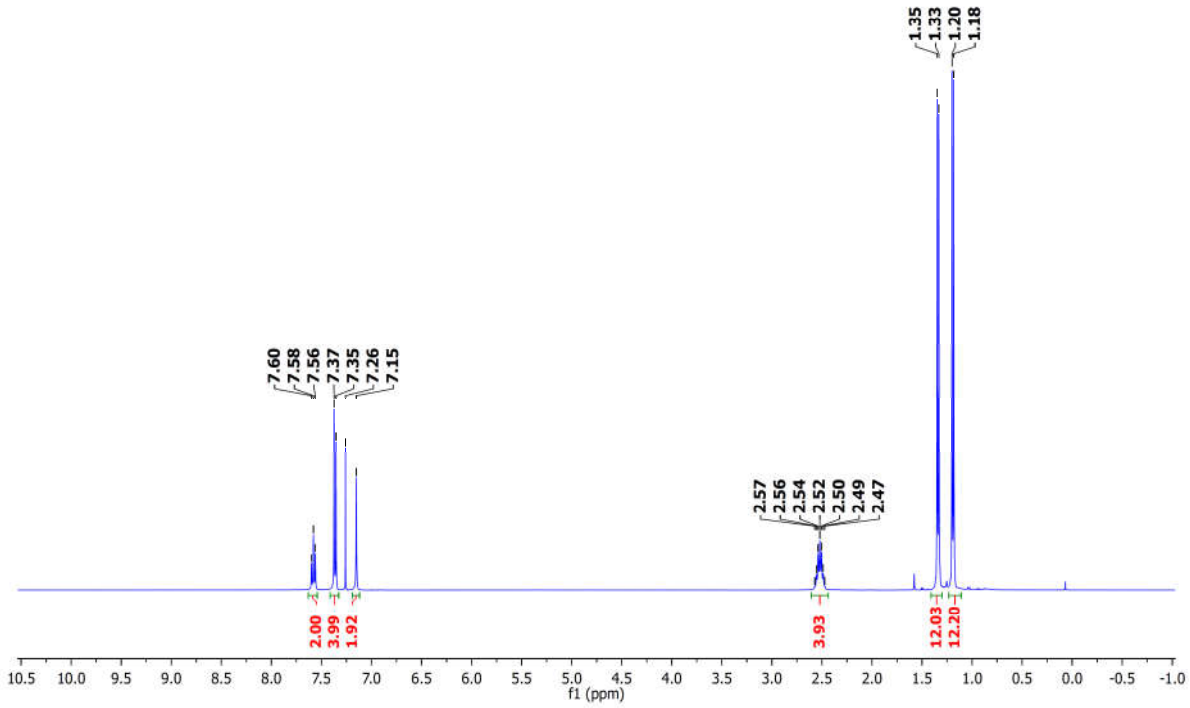


^{13}C{^{1}H}-APT NMR

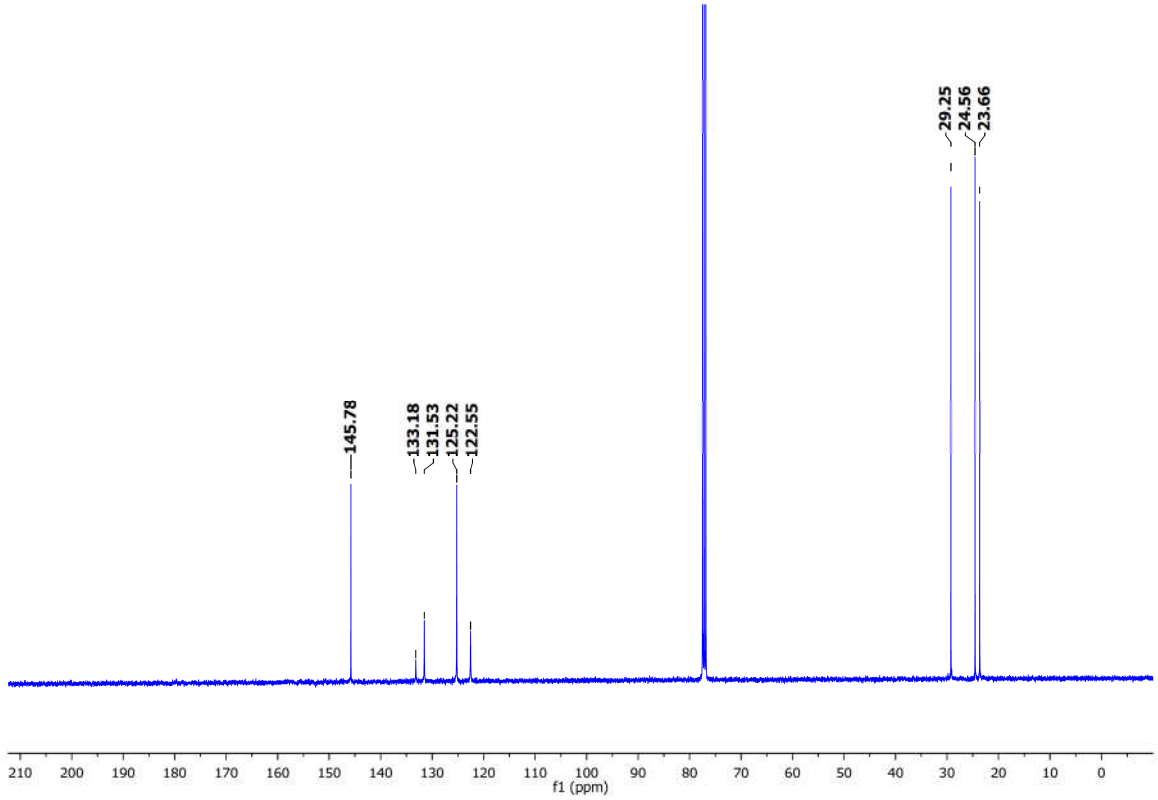


[AgCl(1)]

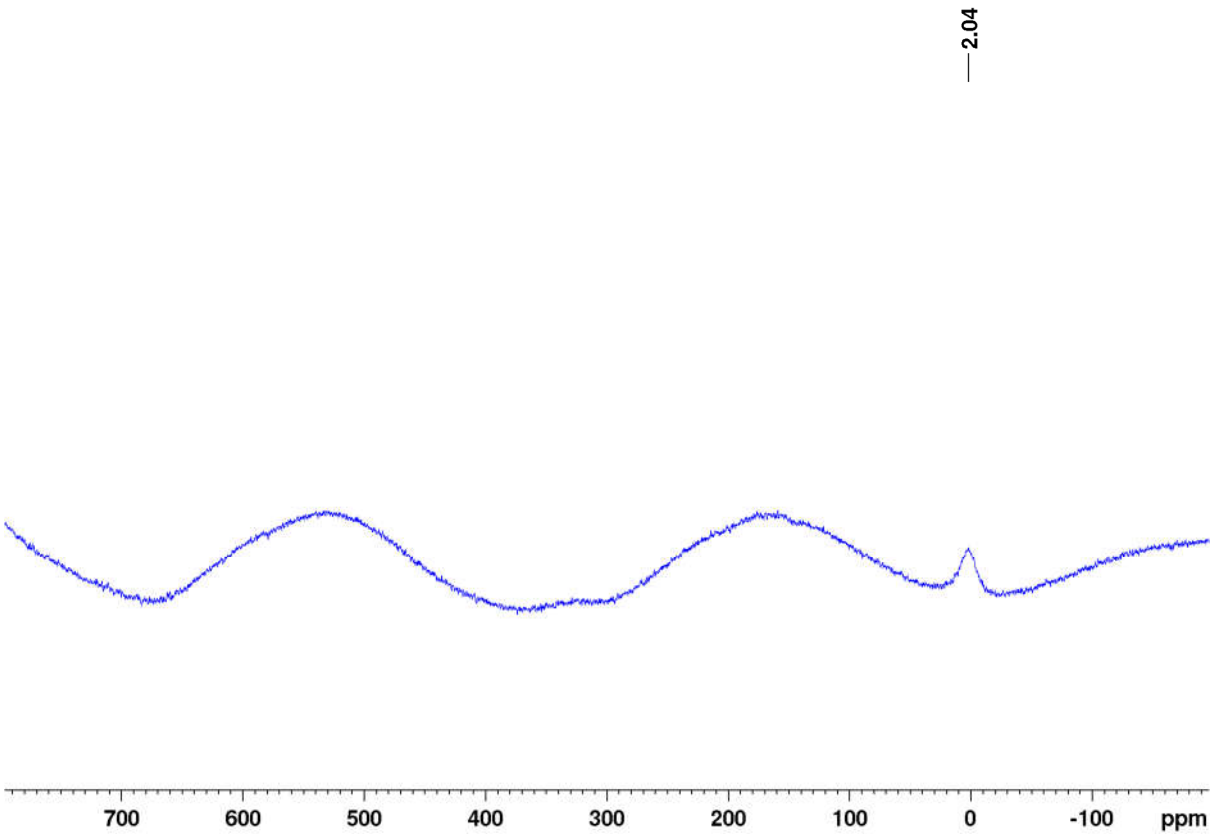
^{1}H NMR



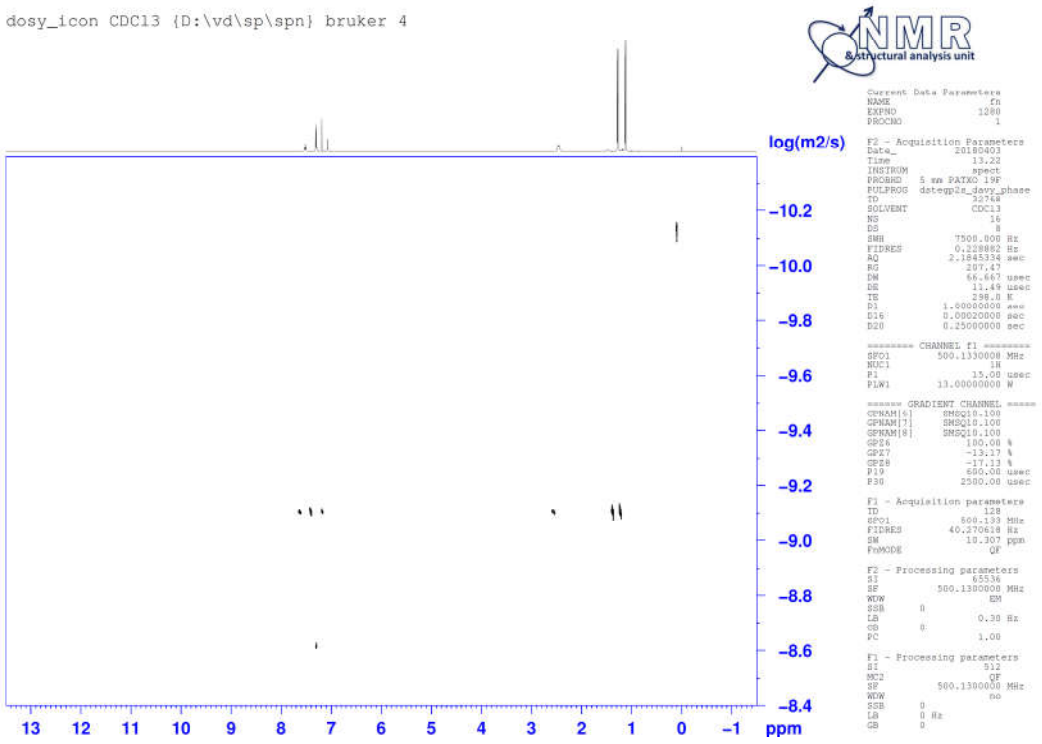
^{13}C{^{1}H} NMR



^{77}Se NMR

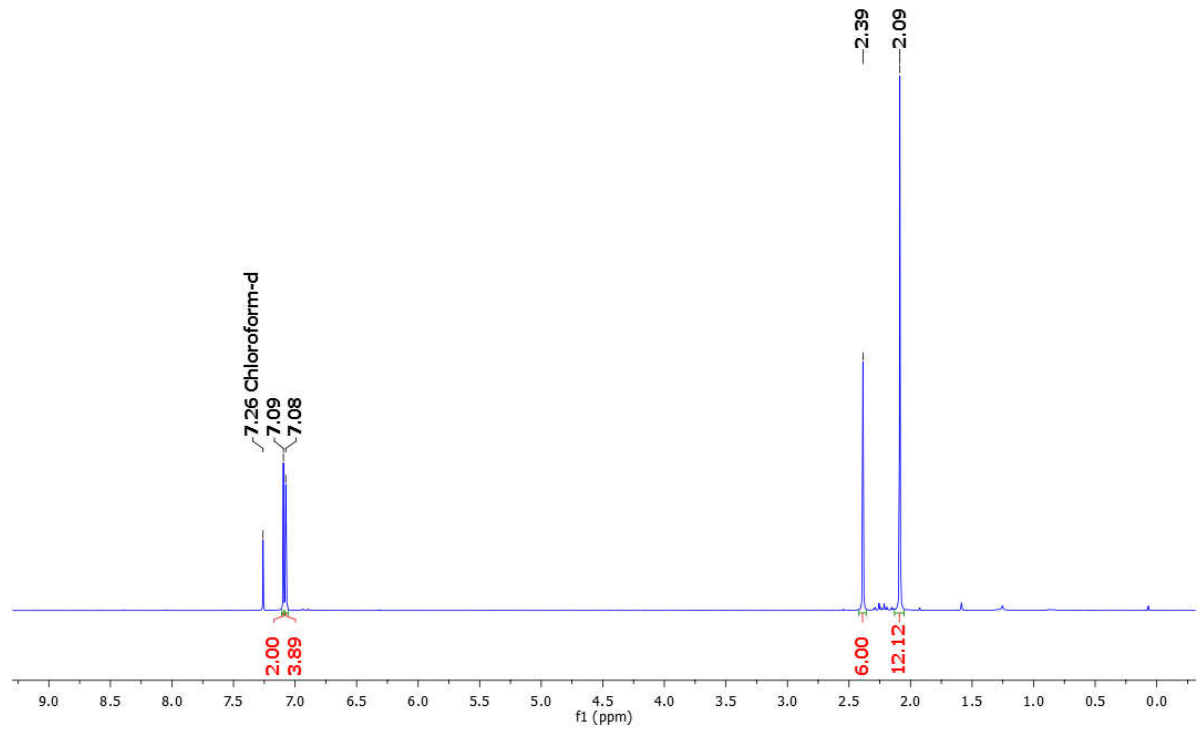


2D DOSY NMR

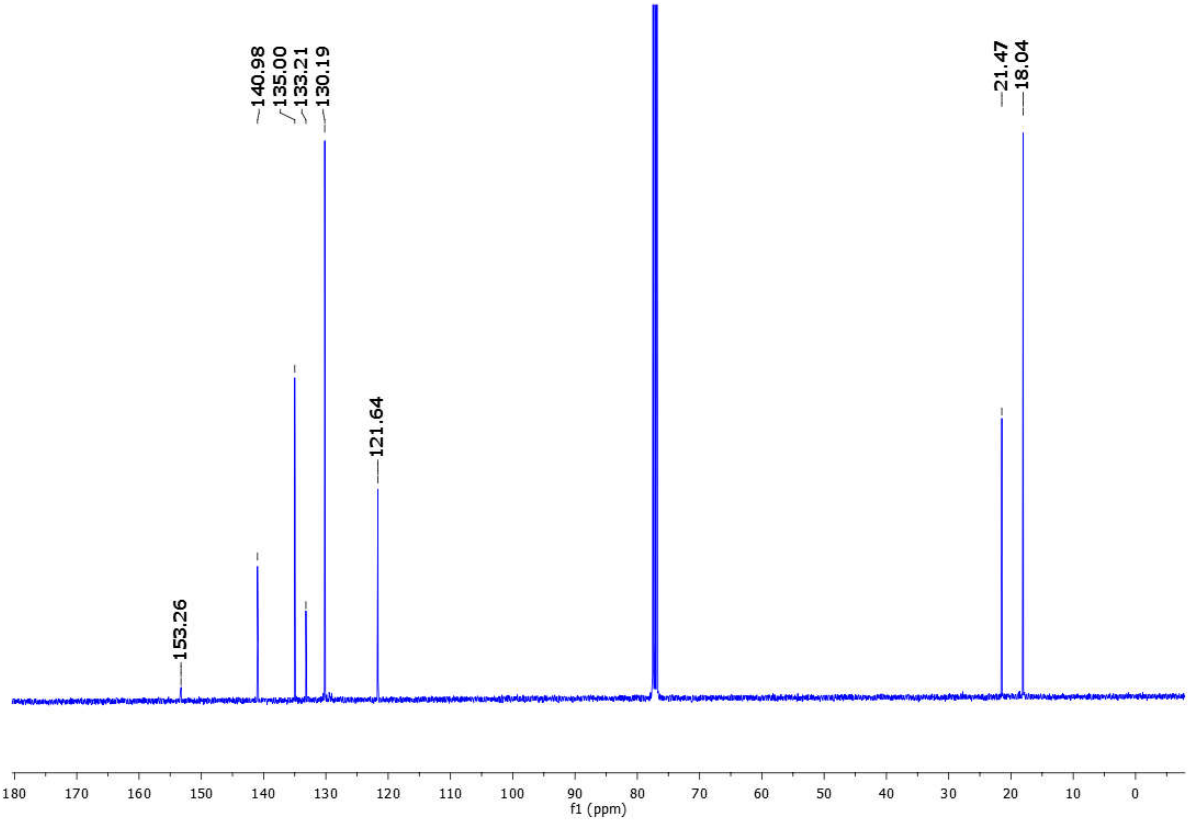


[AgCl(4)]

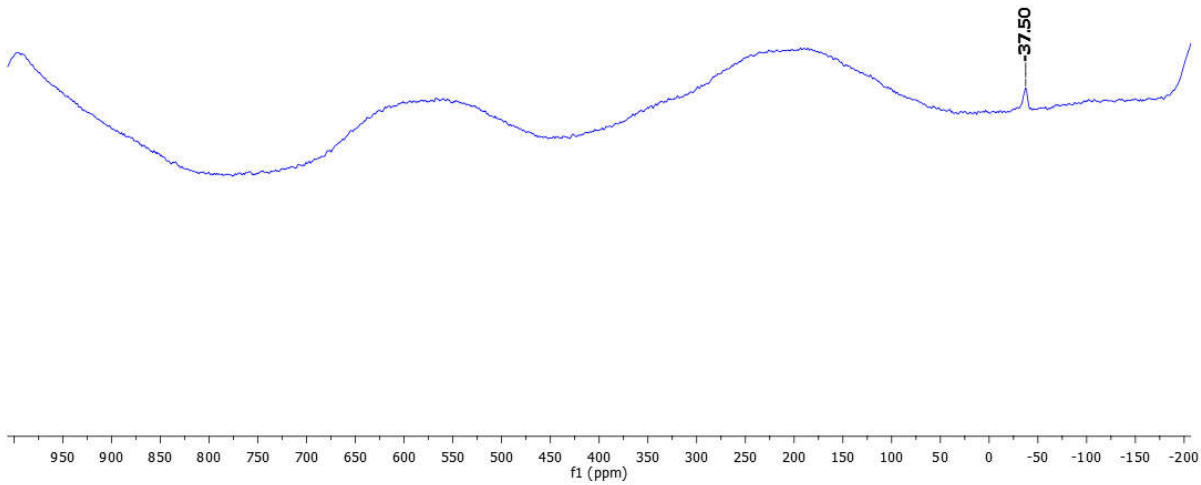
^{1}H NMR



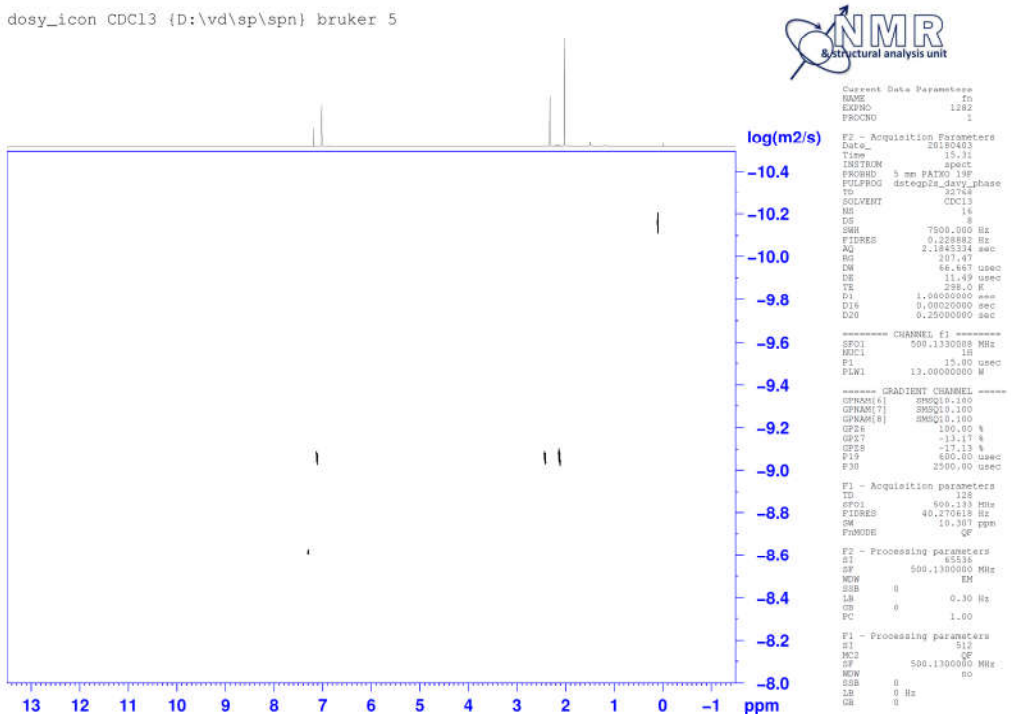
^{13}C{^{1}H} NMR



^{77}Se NMR

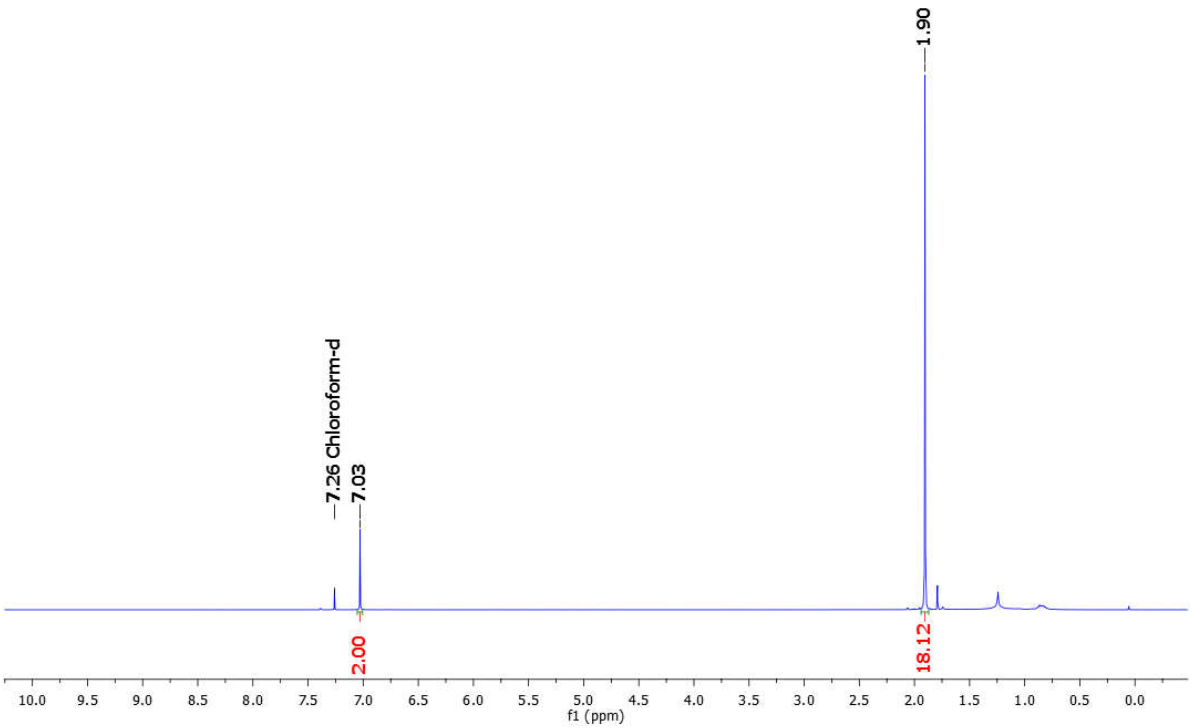


2D DOSY NMR

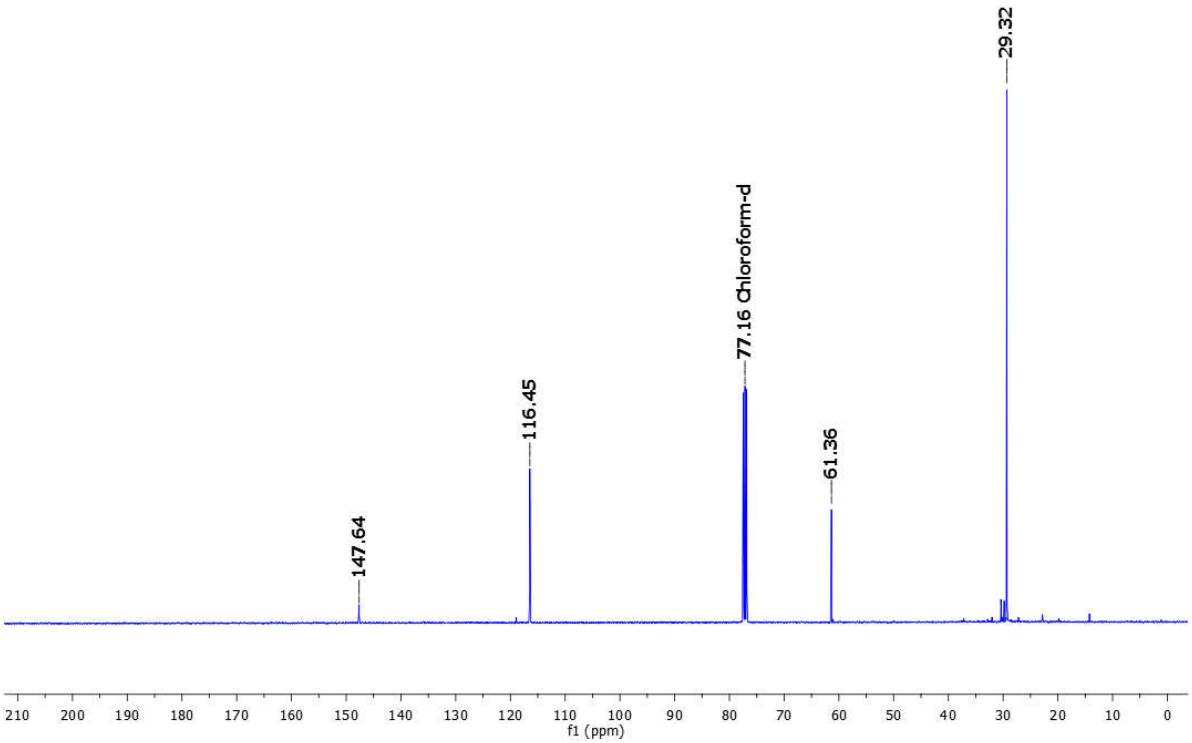


[AgCl(8)]

^{1}H NMR



^{13}C{^{1}H} NMR



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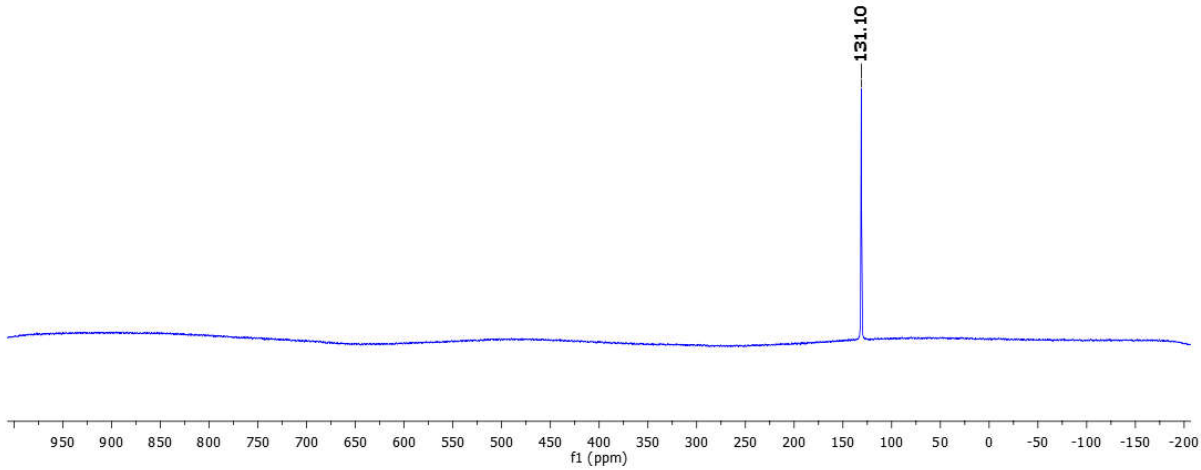

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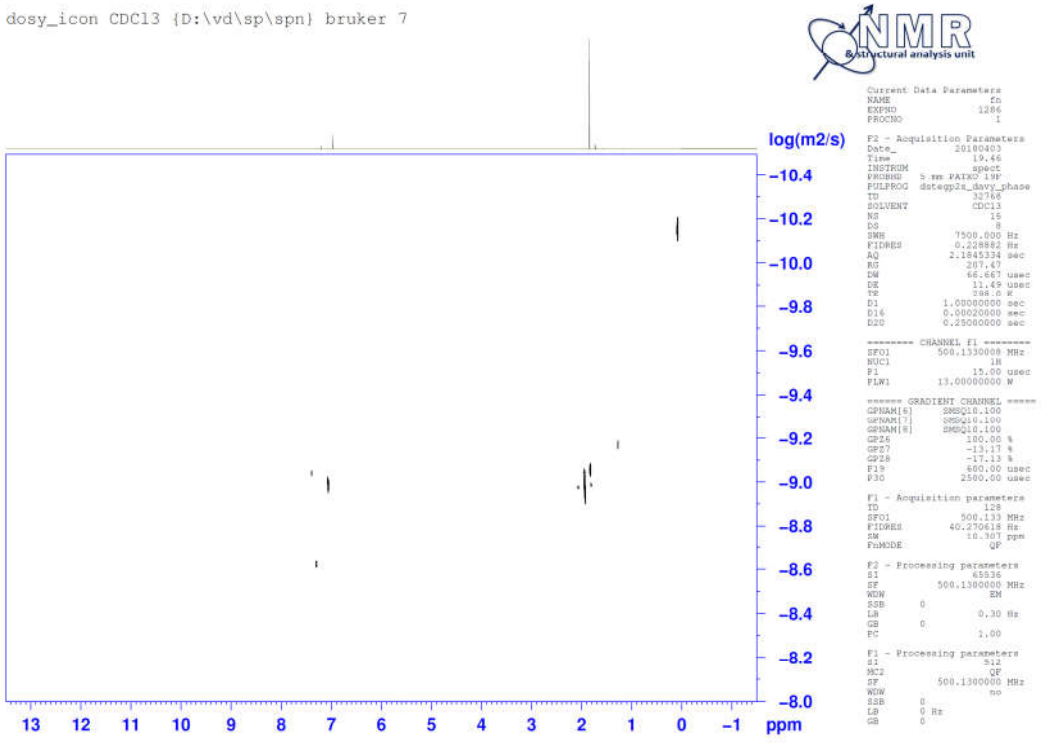
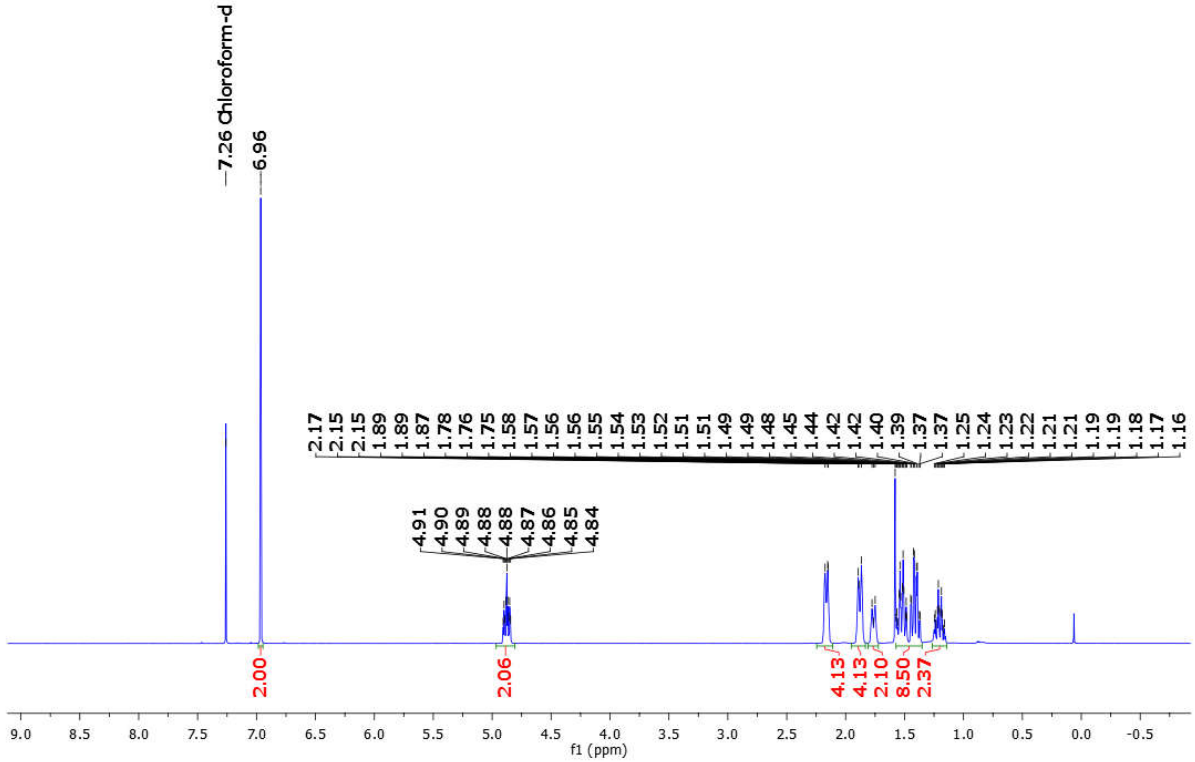
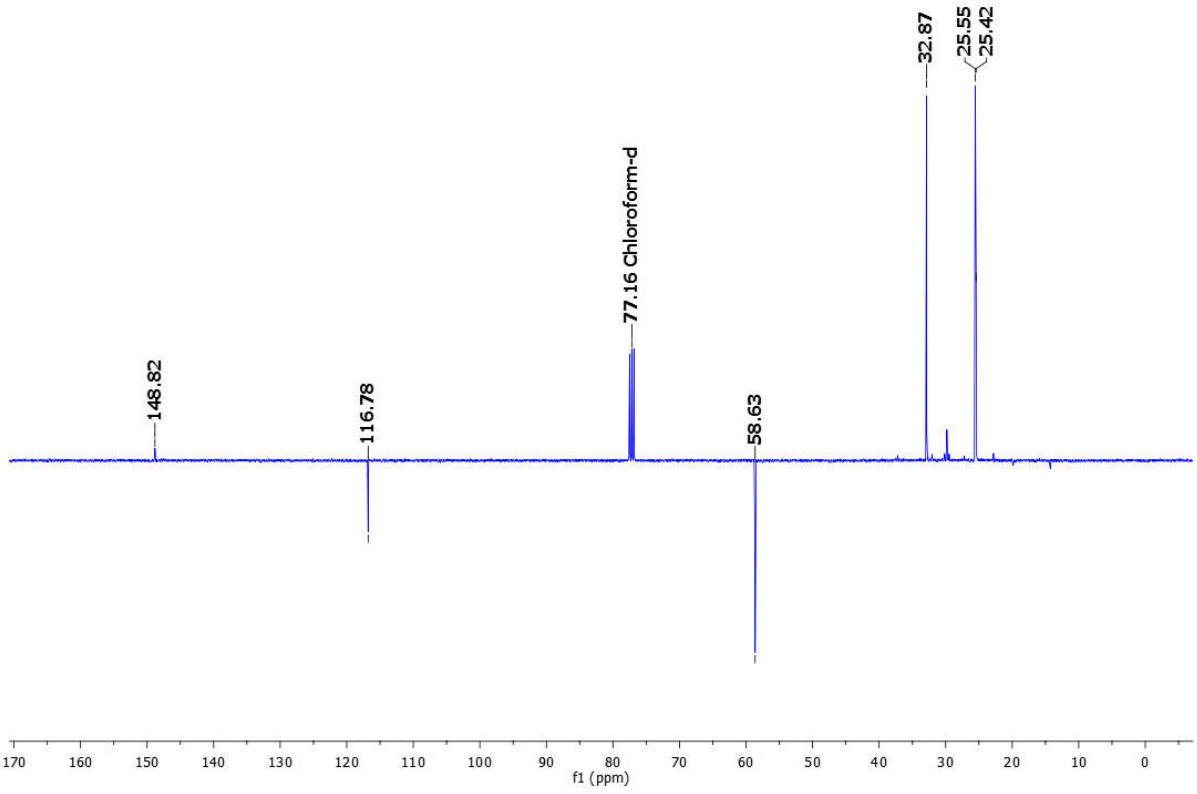


[AgCl(9)]

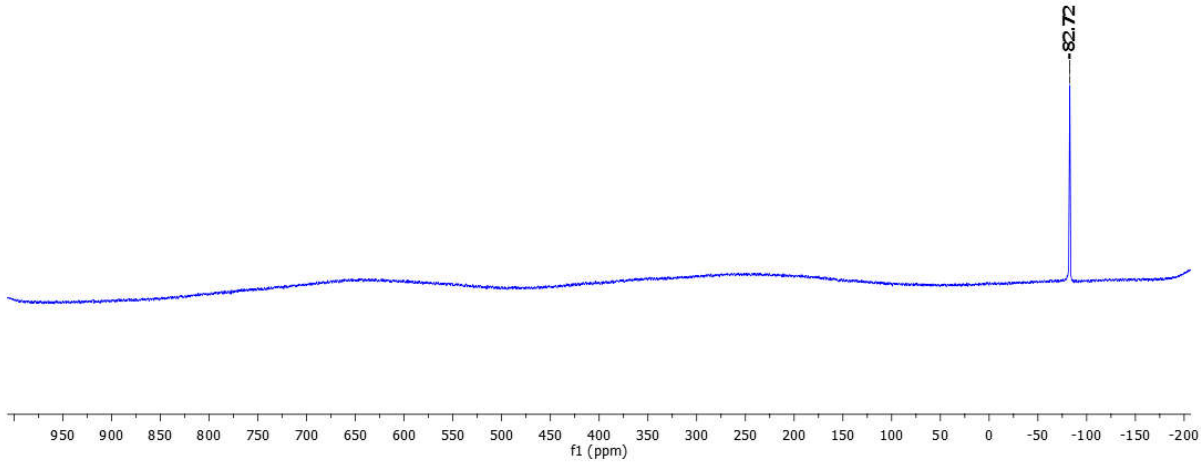
^{1}H NMR



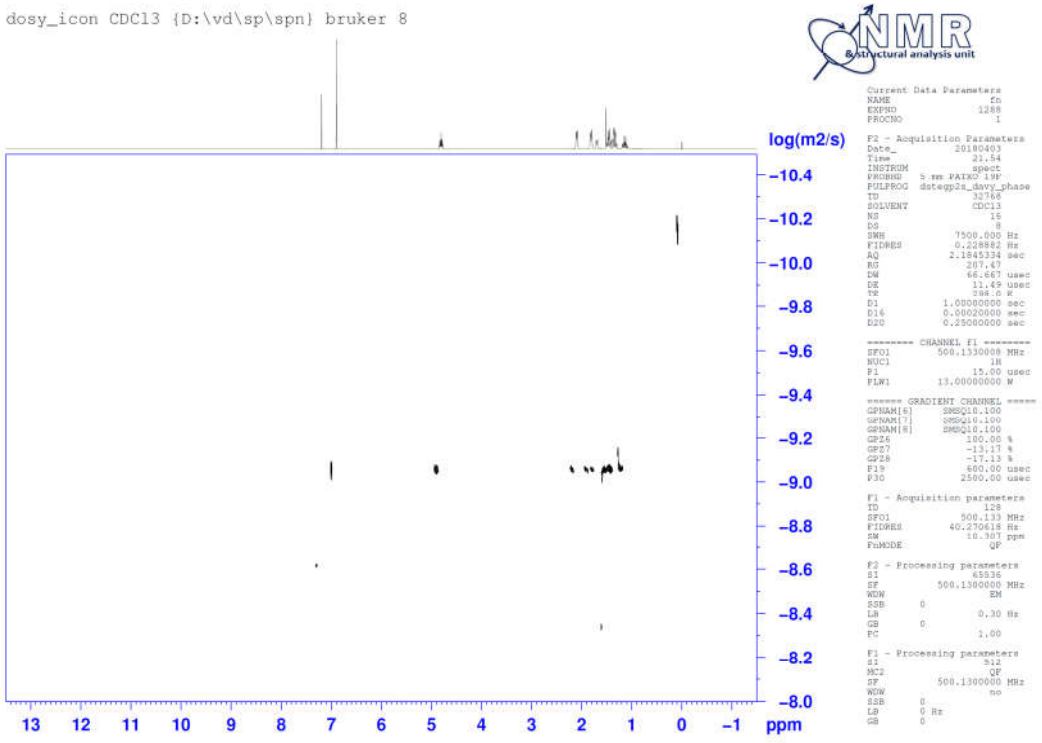
^{13}C{^{1}H}-APT NMR



^{77}Se NMR

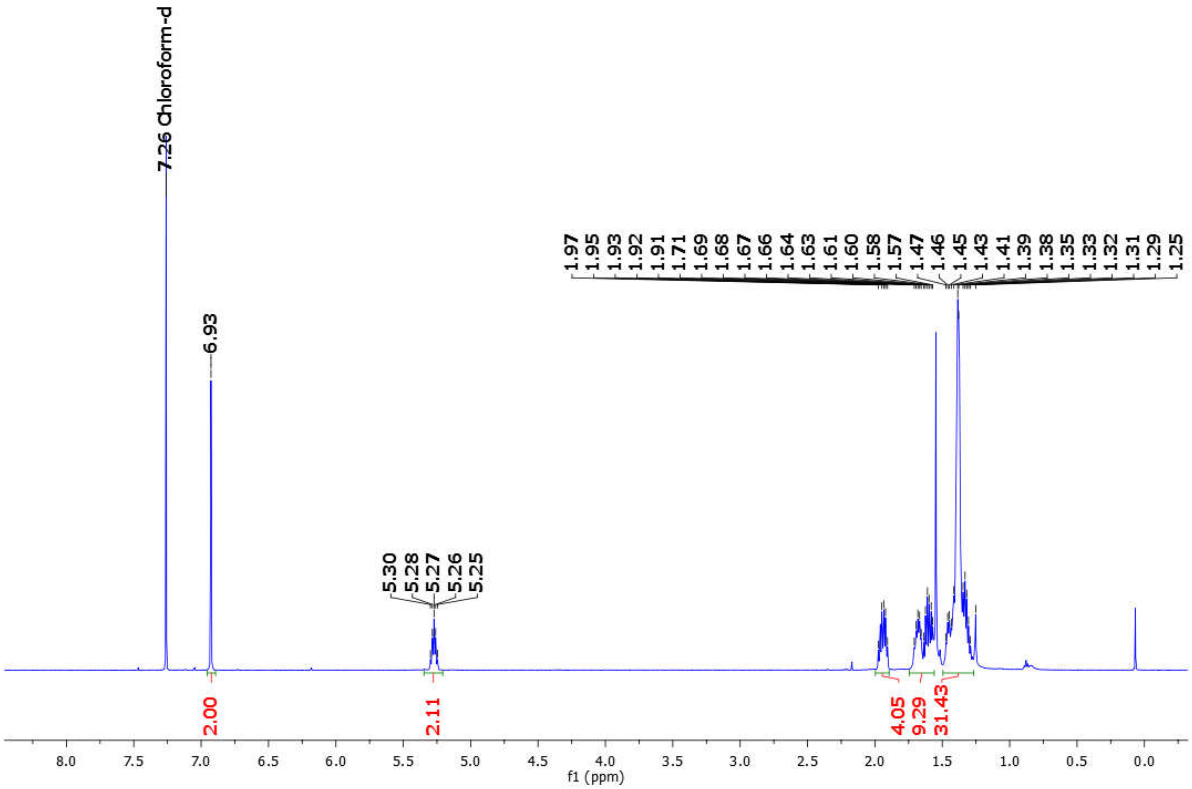


2D DOSY NMR

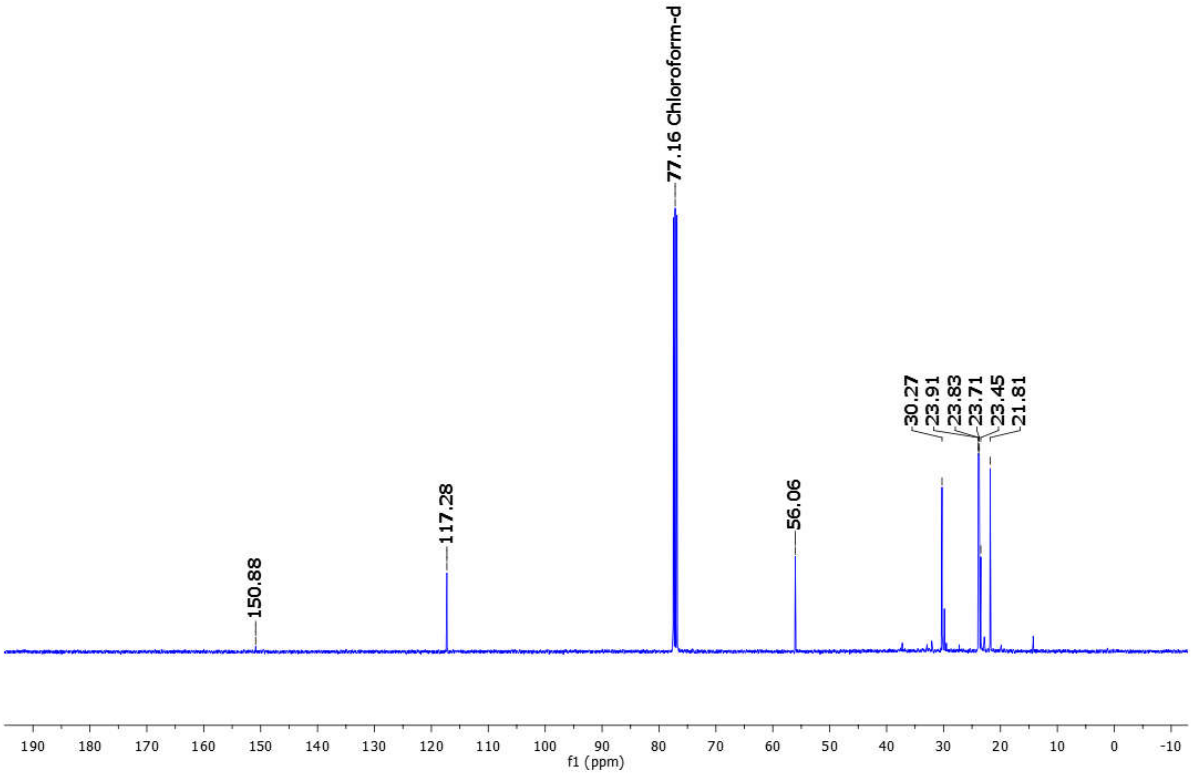


[AgCl(10)]

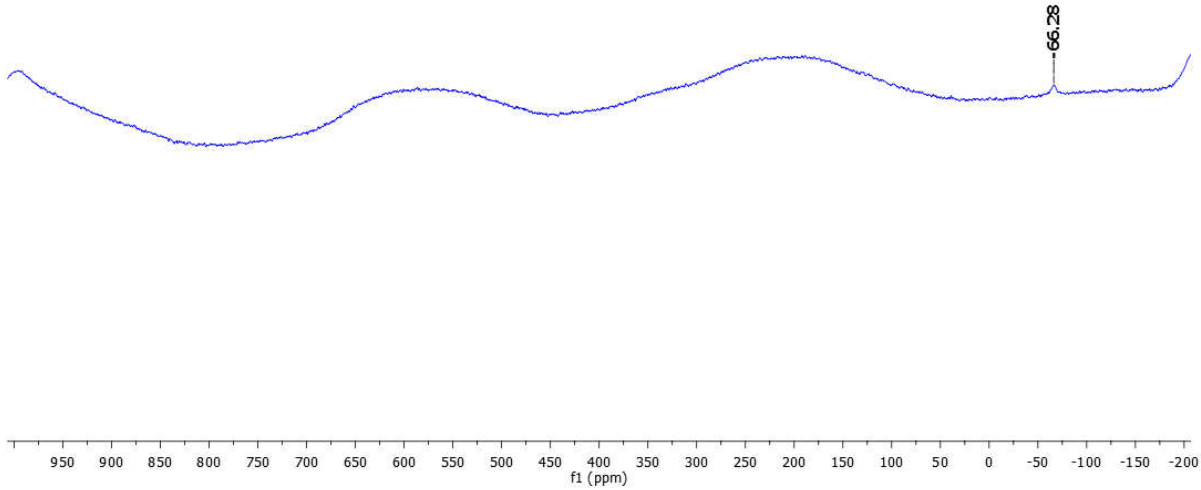
^{1}H NMR



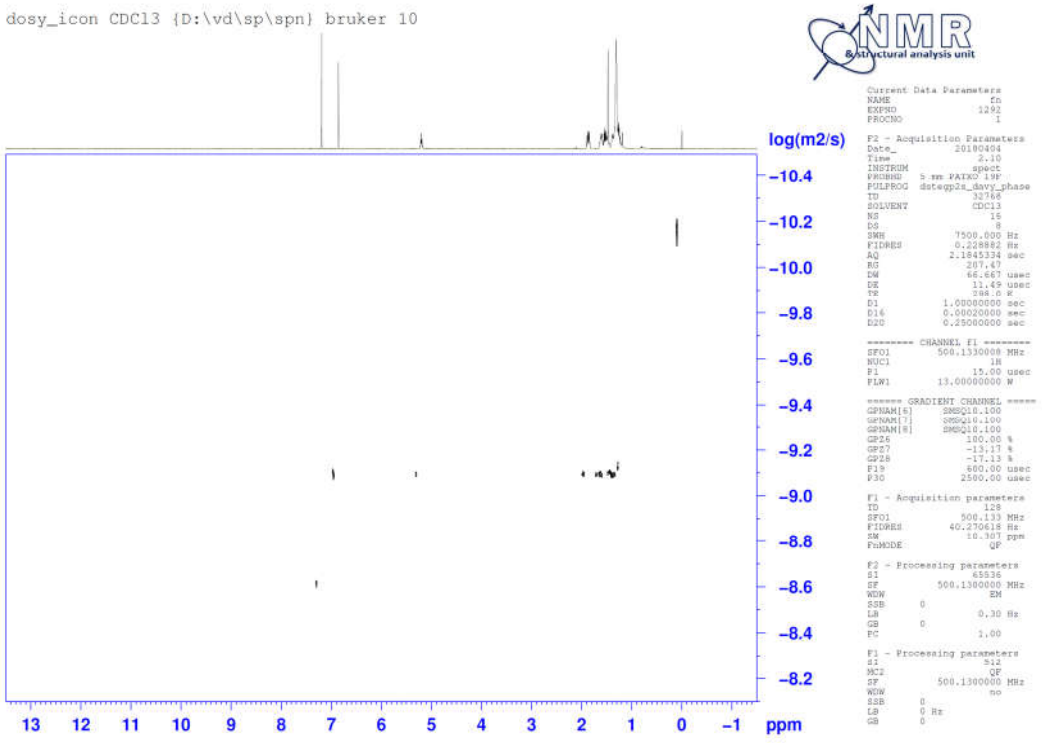
^{13}C{^{1}H} NMR



^{77}Se NMR

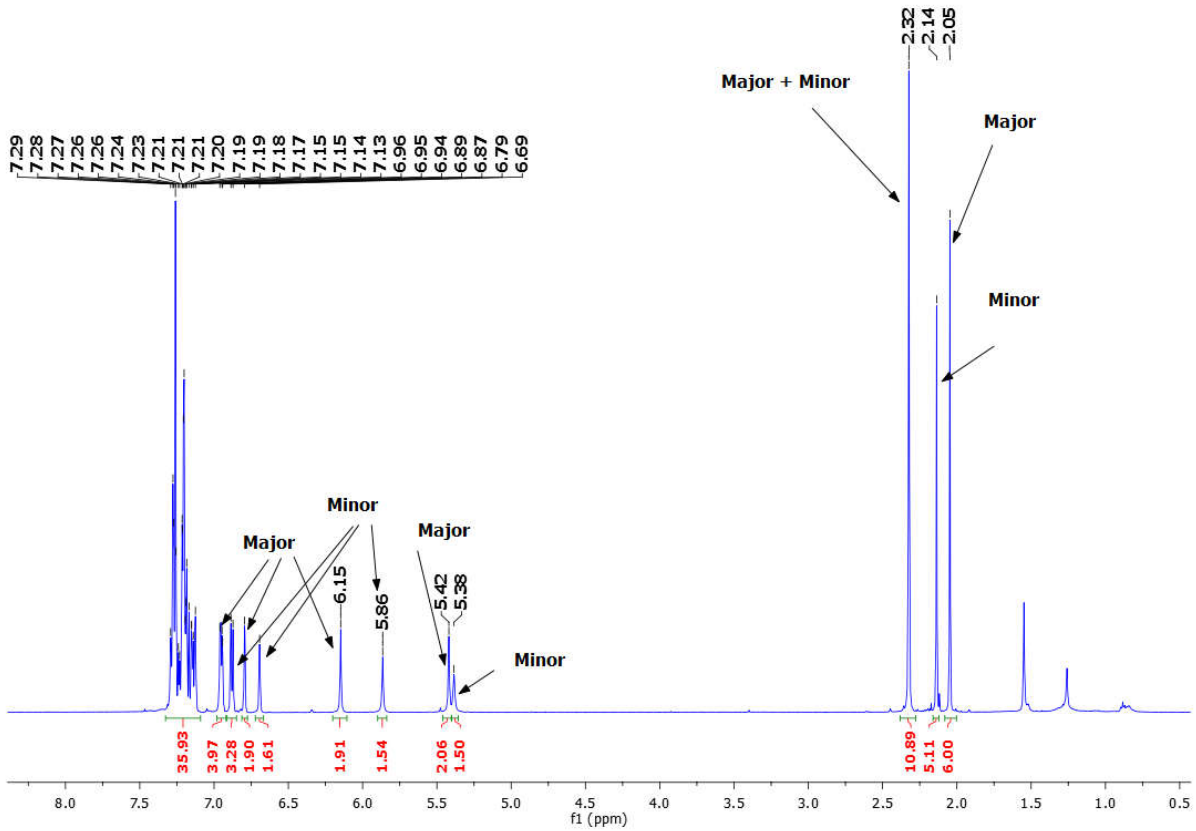


2D DOSY NMR

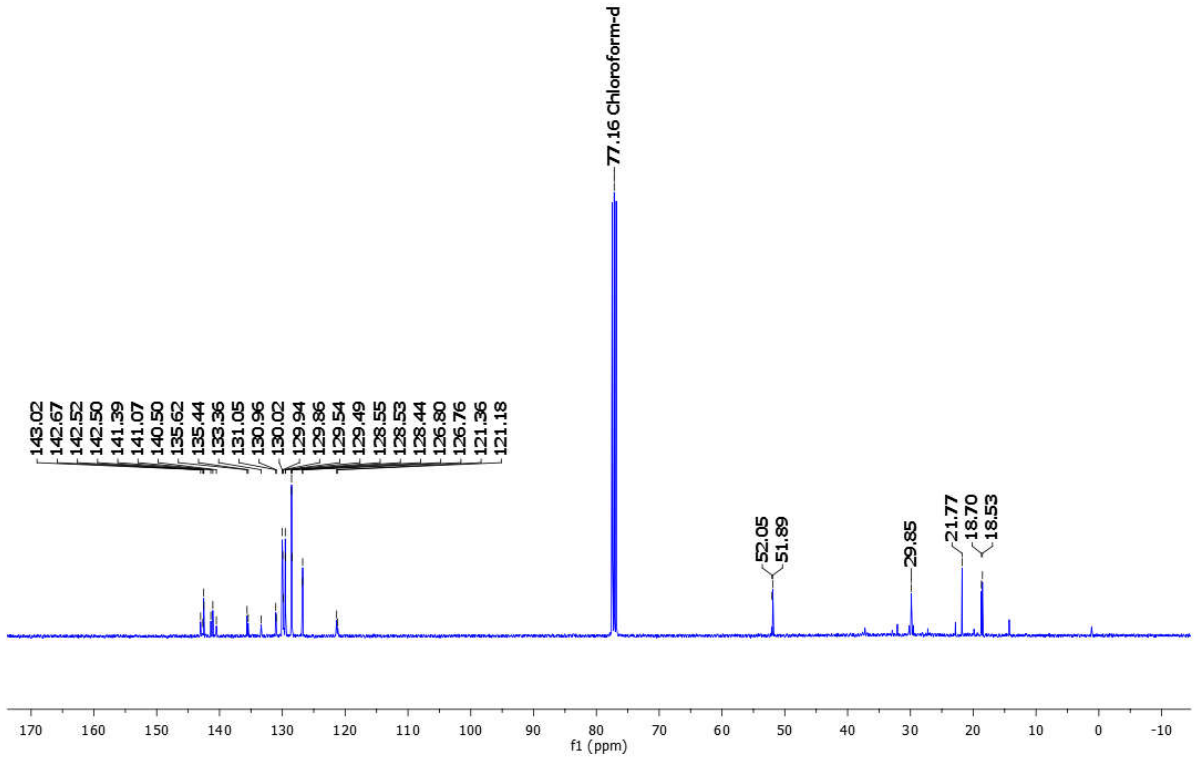


[AgCl(12)]

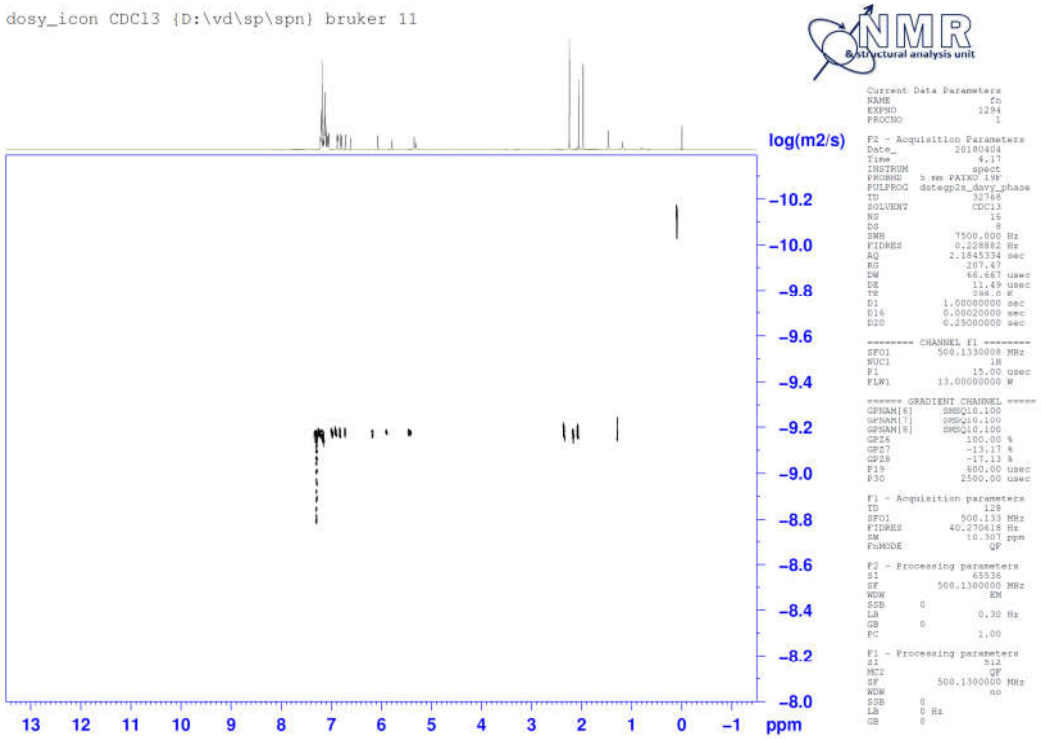
^{1}H NMR



^{13}C{^{1}H} NMR



2D DOSY NMR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NMR
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NUC1 1H
P1 15.00 usec
PLW1 13.00000000 W
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GPNAM[8] SMSQ10.100
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GPZ7 -13.17 %
GPZ8 -17.13 %
P19 600.00 usec
P30 2500.00 usec
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SSB 0
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PC 1.00
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WDW no
SSB 0
LB 0 Hz
GB 0

Experimental Crystallography

All crystallographic measurements were made with monochromatic radiation using an Oxford Diffraction Xcalibur sealed-tube diffractometer for the Cu complexes and a SuperNova microsource instrument for the Ag complexes. All structures were refined against F^2 to convergence using all unique reflections and Shelxl programs. Selected crystallographic and refinement parameters are given in Tables S1-S3 and full information has been deposited in cif format, CCDC 1837514-1837524.

Reference for Shelxl: G. M. Sheldrick, *Acta Cryst. A* 2008, **64**, 112-122.

Table S1. Selected crystallographic and refinement parameters for Cu complexes.

	[CuCl(1)]	**[CuCl(6)]**	**[Cu(4)$_2$][CuCl$_2$]**	**[Cu(5)$_2$][CuCl$_2$]**
Empirical formula	$C_{27}H_{36}ClCuN_2Se$	$C_{69}H_{56}ClCuN_2Se$	$C_{42}H_{48}Cl_2Cu_2N_4Se_2$	$C_{42}H_{52}Cl_2Cu_2N_4Se_2$
Molecular Weight	566.53	1091.10	964.74	968.77
Instrument	Xcalibur	Xcalibur	Xcalibur	Xcalibur
Temperature (K)	150(2)	173(2)	173(2)	173(2)
Wavelength (Å)	0.71073	0.71073	0.71073	0.71073
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$	$P2_1/n$	$P2_1/n$
a (Å)	10.6799(19)	12.8581(7)	8.3880(12)	14.3390(5)
b (Å)	18.245(3)	17.3981(9)	19.011(3)	14.7102(7)
c (Å)	14.256(2)	25.6343(15)	13.4119(19)	21.2622(8)
α (°)	90	90	90	90
β (°)	98.220(15)	103.042(5)	97.387(14)	103.538(4)
γ (°)	90	90	90	90
Cell volume (Å^3)	2749.3(8)	5586.6(5)	2121.0(5)	4360.2(3)
Z	4	4	2	4
2θ max(°)	54.00	57.99	59.12	52.00
Reflections collected	14109	32948	17998	27245
Reflections unique	6174	13415	5355	8559
Reflections obs.	4558	9045	4025	5088
R_{int}	0.0521	0.0529	0.0494	0.0676
Program	WinGX	WinGX	WinGX	WinGX
No. Parameters	297	669	243	481
GoF	1.022	1.040	1.074	1.019
R [$I>2\sigma(I)$]	0.0448	0.0497	0.0398	0.0556
Rw (all data)	0.1139	0.1157	0.0895	0.1106
Largest diff. peak and hole (e Å^{-3})	0.586/-0.588	0.421/-0.583	0.440/-0.504	1.071/-0.503

Table S2. Selected crystallographic and refinement parameters for Ag complexes of **1**/**4**/**8**.

	[Ag(1)$_2$]NO$_3$.CHCl$_3$	**[Ag(1)$_2$]Cl.4CHCl$_3$**	**[AgCl(4)]$_2$**	**[Ag(8)$_3$][Ag$_{0.5}$Cl$_{1.5}$].1/2DCM**
Empirical formula	$C_{55}H_{73}AgCl_3N_5O_3Se_2$	$C_{58}H_{76}AgCl_{13}N_4Se_2$	$C_{42}H_{48}Ag_2Cl_2N_4Se_2$	$C_{33.5}H_{61}Ag_{1.5}Cl_{2.5}N_6Se_3$
Molecular Weight	1224.33	1555.87	1053.40	1035.19
Instrument	SuperNova	SuperNova	SuperNova	SuperNova
Temperature (K)	100(2)	100(2)	100(2)	100(2)
Wavelength (Å)	1.54184	1.54184	0.71073	1.54184
Crystal system	monoclinic	monoclinic	triclinic	monoclinic
Space group	$P2_1/c$	$P2_1/n$	P-1	$P2_1/n$
a (Å)	16.1058(6)	16.4565(4)	9.6458(6)	15.2390(2)

b (Å)	23.5607(6)	20.0342(7)	11.2385(12)	10.43045(12)
c (Å)	19.0856(6)	24.4535(6)	11.6775(8)	27.0621(3)
α (°)	90	90	61.398(9)	90
β (°)	110.665(4)	106.086(2)	74.631(6)	90.6645(11)
γ (°)	90	90	66.612(8)	90
Cell volume (Å^3)	6776.3(4)	7746.5(4)	1015.67(17)	4301.22(9)
Z	4	4	1	4
2θ max(°)	151.64	151.12	59.36	151.18
Reflections collected	97436	119069	22703	80612
Reflections unique	13798	15710	5144	8840
Reflections obs.	9767	9907	3065	7465
R_{int}	0.1017	0.0989	0.1106	0.0701
Program	Olex2	Olex2	Olex2	WinGX
No. Parameters	638	719	241	454
GoF	1.077	1.046	1.066	1.037
R [*I*>2σ(*I*)]	0.0632	0.0913	0.0685	0.0388
Rw (all data)	0.1832	0.1786	0.0943	0.1080
Largest diff. peak and hole (e Å^{-3})	2.144/-1.468	1.048/-0.908	0.985/-0.742	0.889/-1.852

Table S3. Selected crystallographic and refinement parameters for Ag complexes of **9** and **10**.

	[Ag(9)$_3$]Cl.solvent	**[Ag$_2$(9)$_5$][NO$_3$]$_2$.2DCM**	**[AgCl(10)$_2$]**
Empirical formula	$C_{45.5}H_{73}AgCl_2N_6OSe_3$	$C_{77}H_{122}Ag_2Cl_4N_{12}O_6Se_5$	$C_{54}H_{96}AgClN_4Se_2$
Molecular Weight	1135.75	2064.21	1102.59
Instrument	SuperNova	SuperNova	SuperNova
Temperature (K)	100(2)	100(2)	100(2)
Wavelength (Å)	1.54184	1.54184	0.71073
Crystal system	monoclinic	monoclinic	triclinic
Space group	$P2_1/n$	$P2_1/n$	P-1
a (Å)	27.3848(4)	16.3612(2)	12.5173(3)
b (Å)	15.0419(2)	30.0343(4)	13.8453(2)
c (Å)	27.9825(4)	18.7111(3)	16.5647(3)
α (°)	90	90	92.768(2)
β (°)	106.277(2)	107.848(2)	99.075(2)
γ (°)	90	90	102.185(2)
Cell volume (Å^3)	11064.5(3)	8752.1(2)	2761.04(10)
Z	8	4	2
2θ max(°)	151.20	151.02	59.34
Reflections collected	103069	84706	61661
Reflections unique	22282	17820	14091
Reflections obs.	17427	13726	10517
R_{int}	0.0566	0.0696	0.0631
Program	Olex2	Olex2	Olex2
No. Parameters	1054	965	559
GoF	1.055	1.068	1.047
R [*I*>2σ(*I*)]	0.0634	0.0668	0.0495
Rw (all data)	0.1895	0.1960	0.1188
Largest diff. peak and hole (e Å^{-3})	2.081/-1.352	2.506/-1.335	2.362/-0.696