Monoorganoantimony (V) Phosphonates and PhosphoSelininates

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Contents:

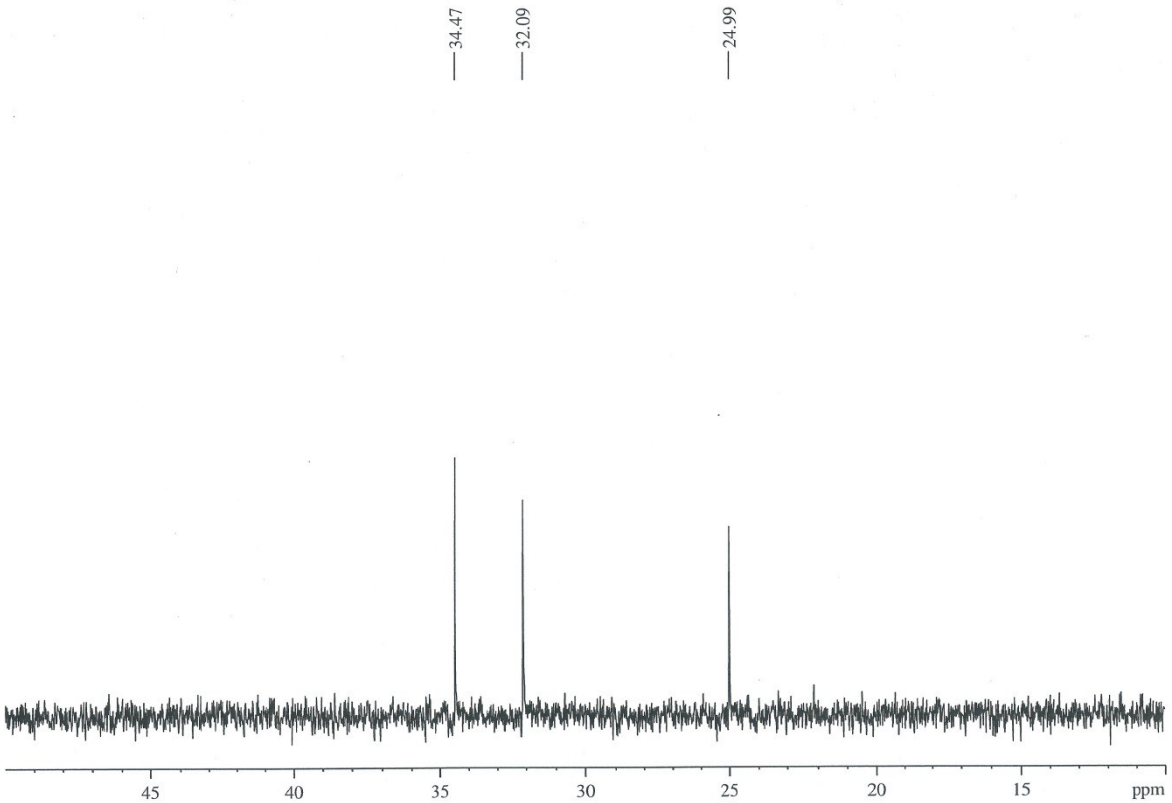

Figure S1. Solution ^{31}P NMR spectrum of **1** in $CDCl_3$ solvent.

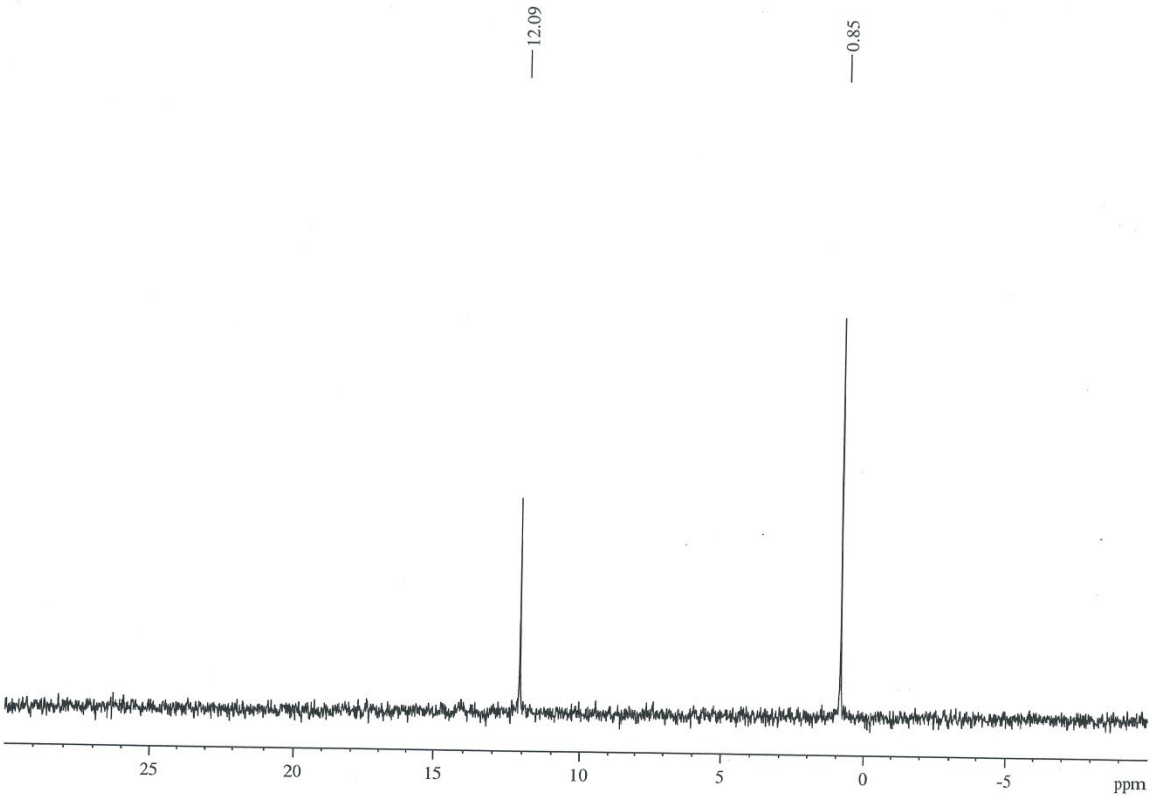

Figure S2. Solution ^{31}P NMR spectrum of **2** in $CDCl_3$ solvent

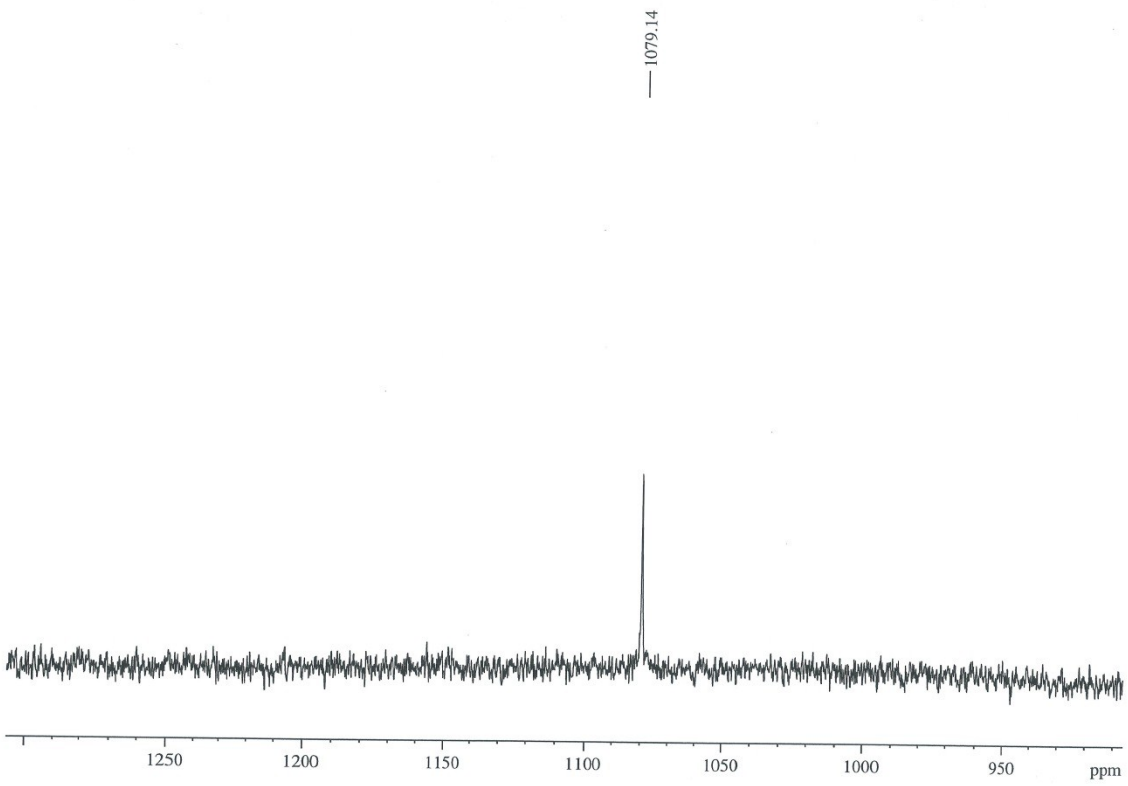

Figure S3. Solution ^{77}Se NMR spectrum of **3** in $CDCl_3$ solvent.

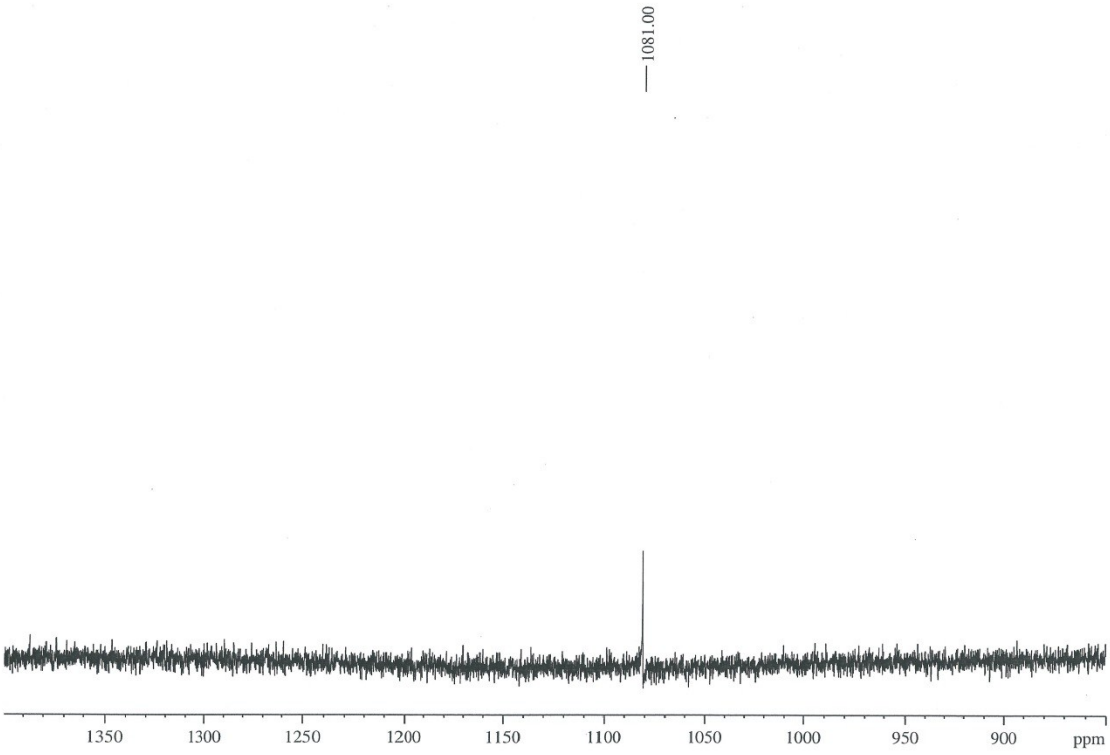

Figure S4. Solution ^{77}Se NMR spectrum of **4** in $CDCl_3$ solvent.

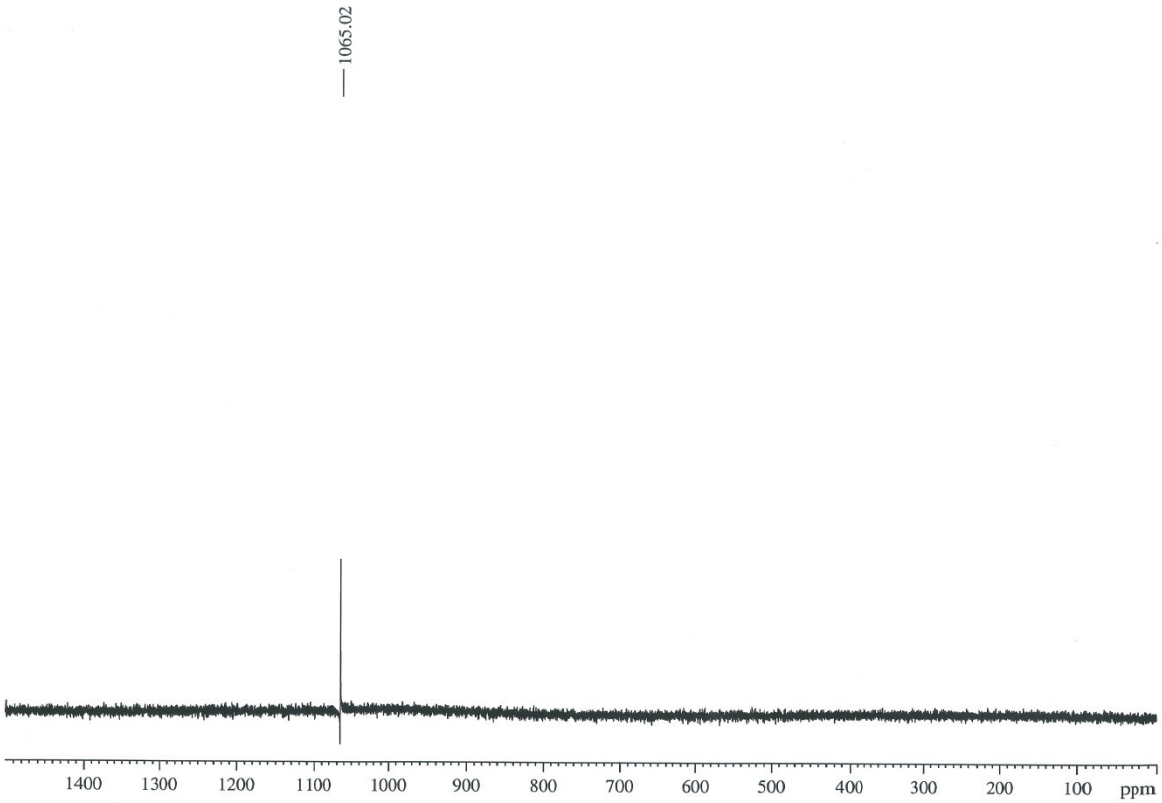

Figure S5. Solution ^{77}Se NMR spectrum of **5** in $CDCl_3$ solvent.

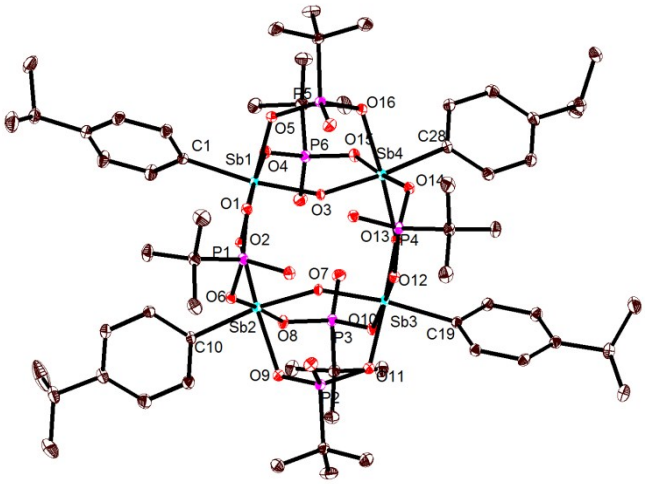

Figure S6. ORTEP view of **1** with thermal ellipsoids shown at 30% probability

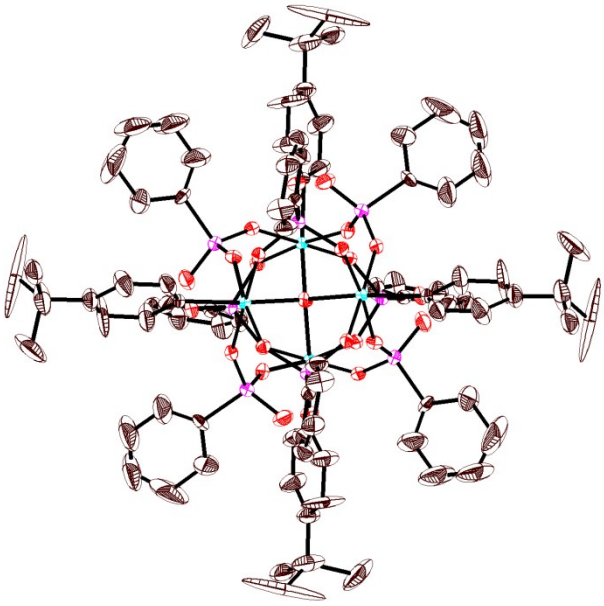

Figure S7. ORTEP view of **2** with thermal ellipsoids shown at 30% probability

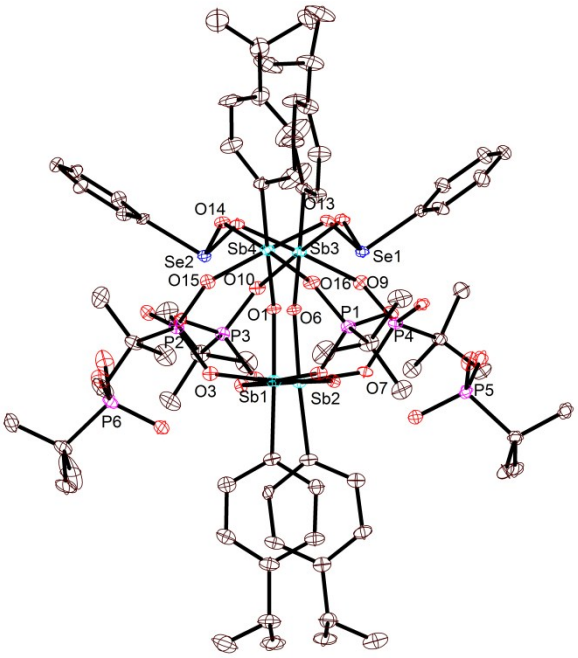

Figure S8. ORTEP view of **3** with thermal ellipsoids shown at 30% probability

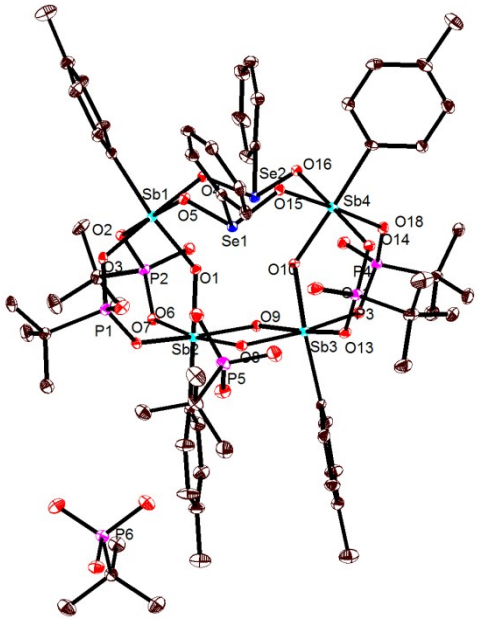

Figure S9. ORTEP view of **4** with thermal ellipsoids shown at 30% probability

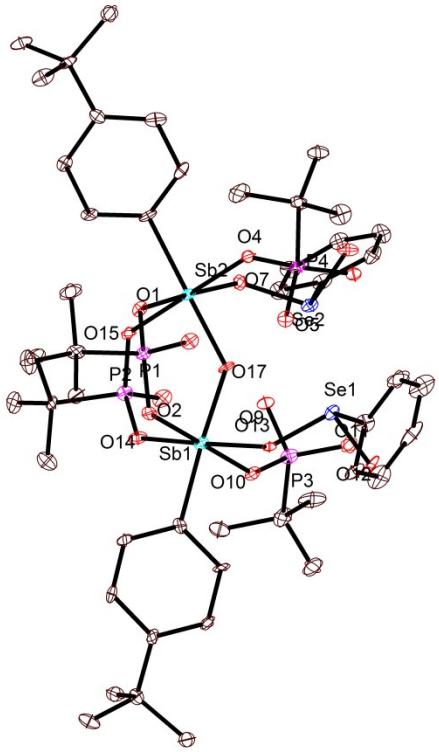

Figure S10. ORTEP view of **5** with thermal ellipsoids shown at 30% probability

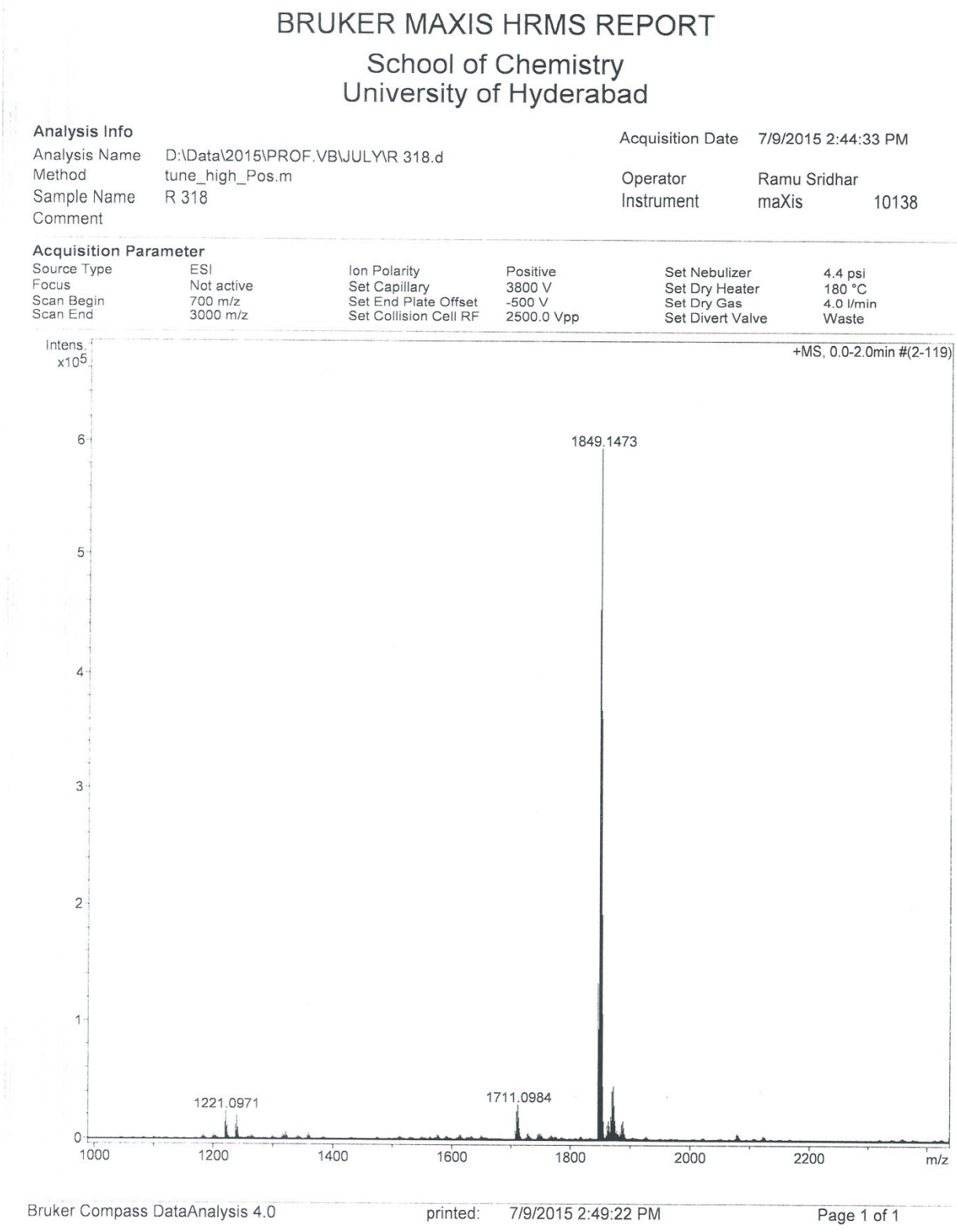

Figure S11. ESI-MS of compound **1** in $CHCl_3$-CH_3CN.

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Analysis Info

Analysis Name	D:\Data\2014\Dr.V.BHASKER\JUL\U 291A.d	Acquisition Date	7/8/2014 3:23:26 PM
Method	tune_high_Pos.m	Operator	Ramu Sridhar
Sample Name	U 291A-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

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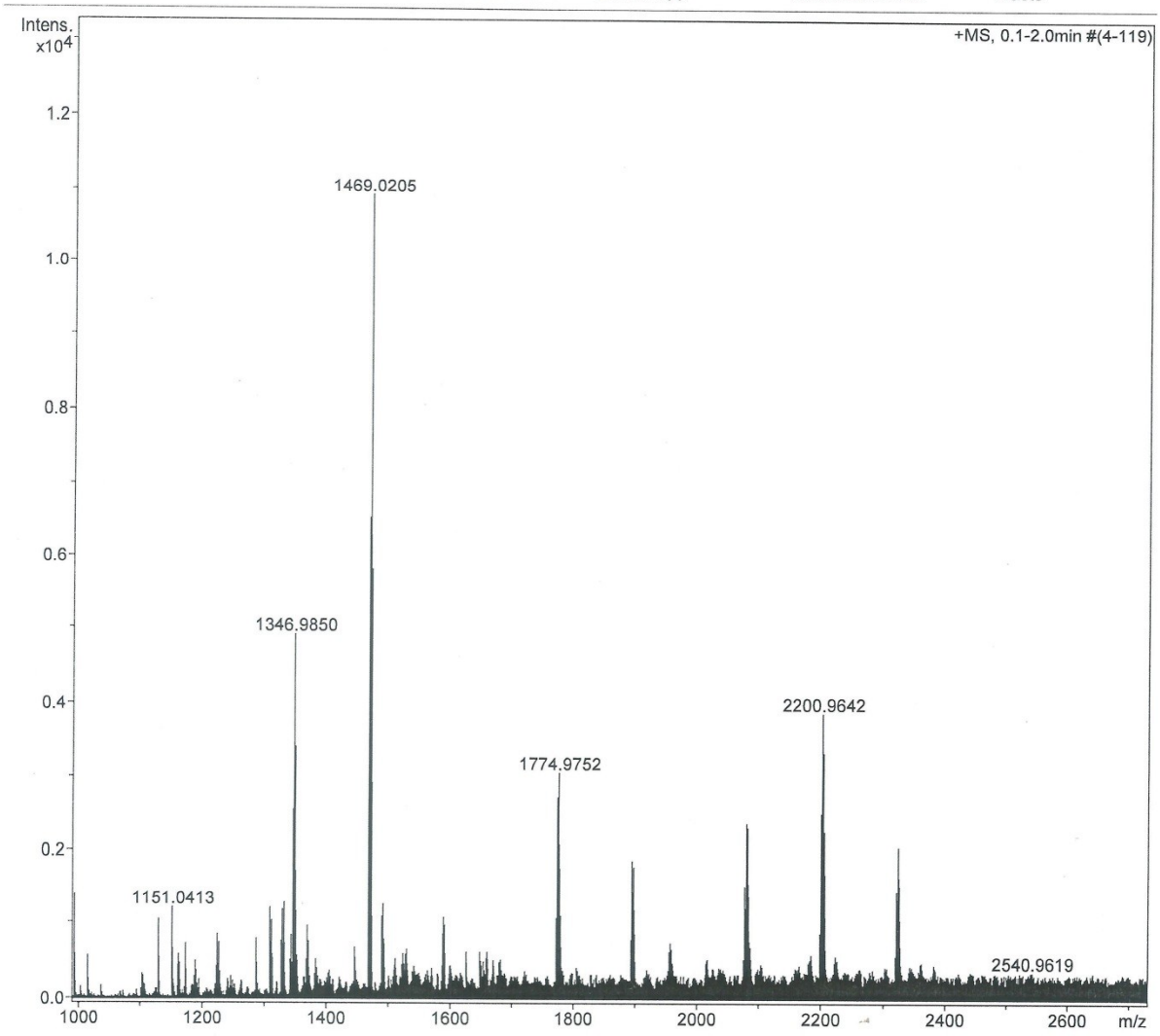

Figure S12. ESI-MS of compound **2** in $CHCl_3$-CH_3CN.

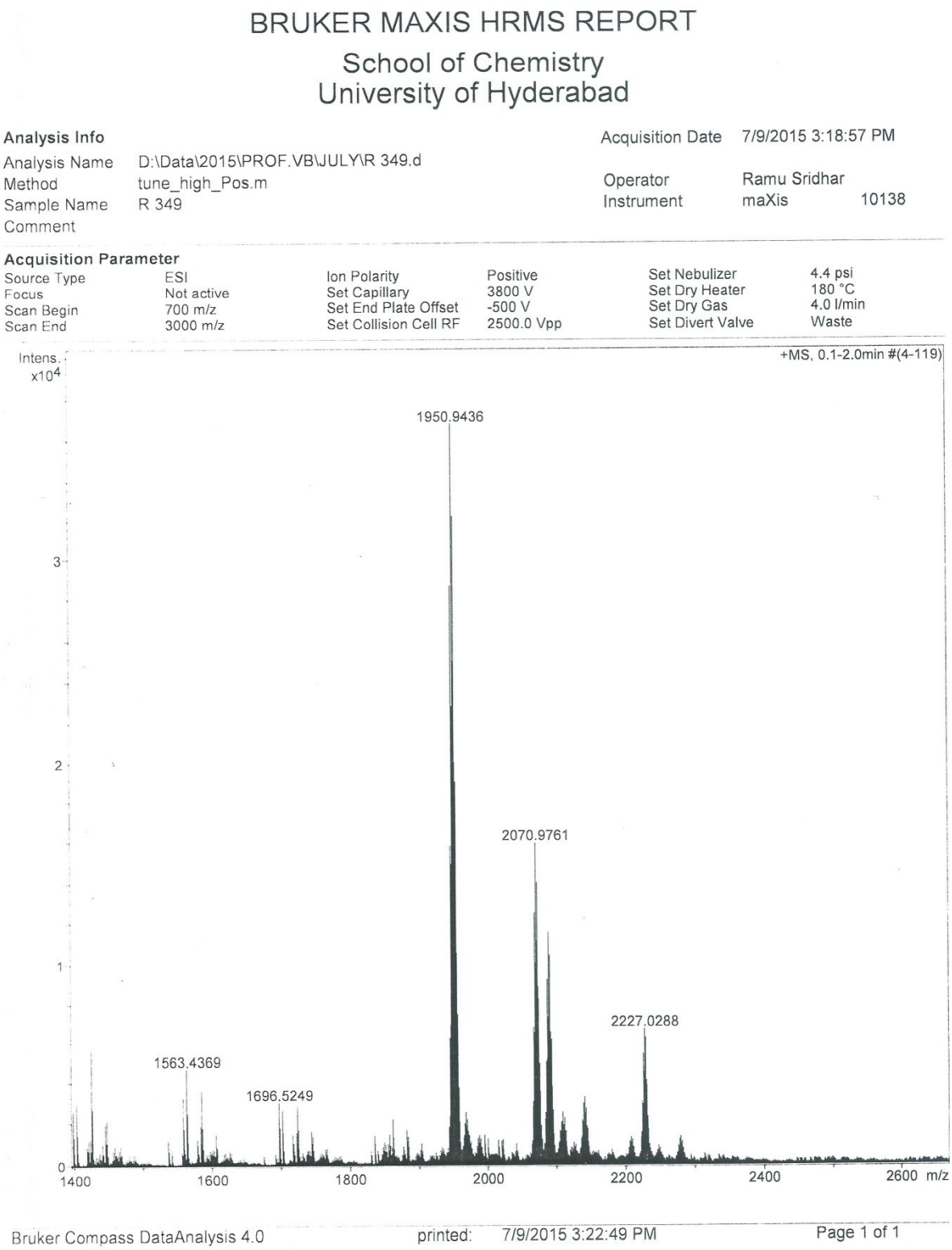

Figure S13. ESI-MS of compound **3** in $CHCl_3$-CH_3CN.

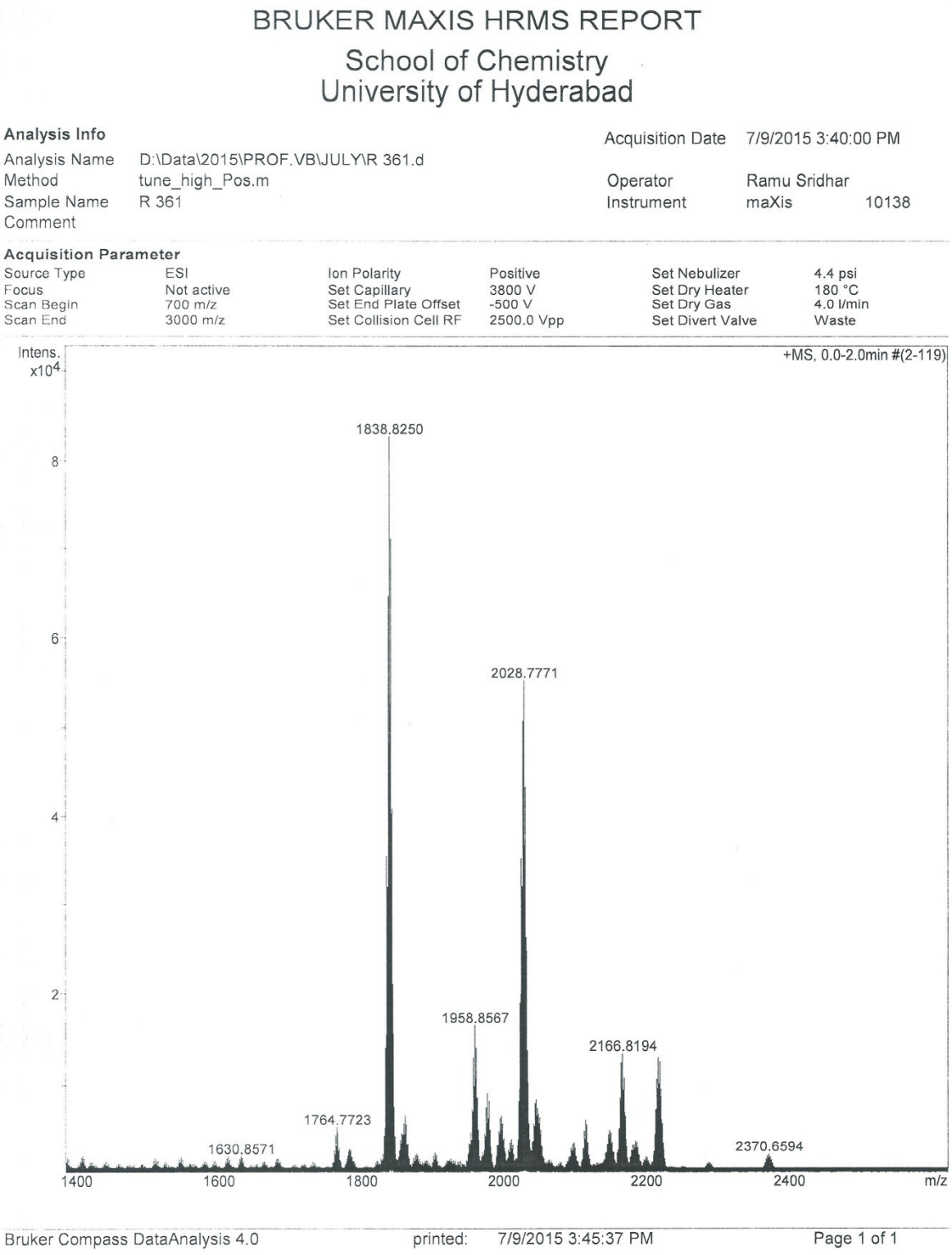

Figure S14. ESI-MS of compound **4** in $CHCl_3$-CH_3CN.

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Analysis Info

Analysis Name	D:\Data\2015\PROF.VB\JULY\R-351a.d	Acquisition Date	7/23/2015 2:49:18 PM
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Sample Name	R-351a	Instrument	maXis 10138
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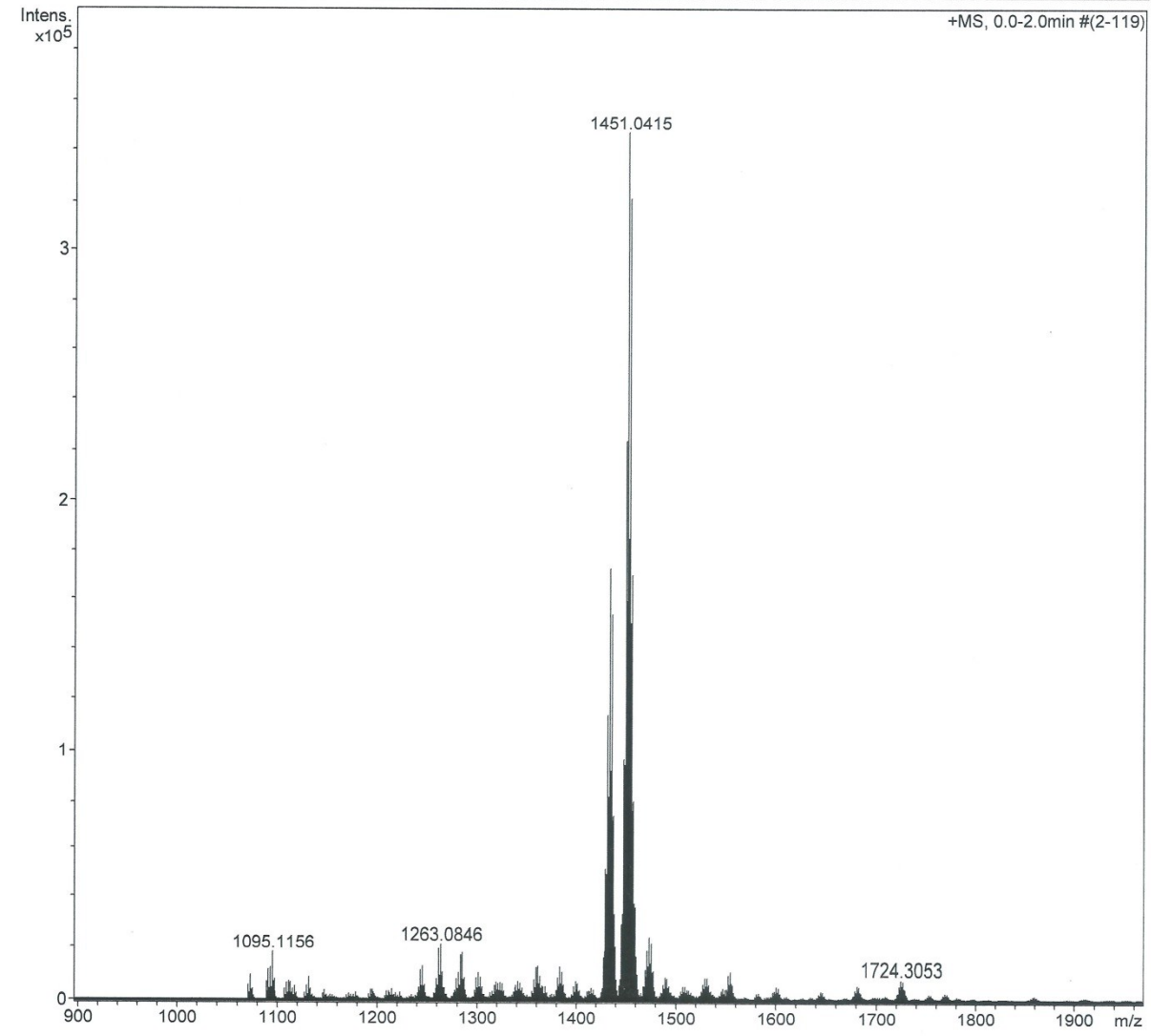
Scan End	3000 m/z	Set Collision Cell RF	2500.0 Vpp	Set Divert Valve	Waste



Bruker Compass DataAnalysis 4.0 printed: 7/23/2015 2:52:56 PM Page 1 of 1

Figure S15. ESI-MS of compound **5** in $CHCl_3$-CH_3CN.

Table-1: Crystal data for compounds **1-4**

	1	2	3	4
Formula	$C_{62}H_{101}NO_{22}P_6Sb_4$	$C_{88}H_{92}O_{26}P_8Sb_4$	$C_{78}H_{123}N_3O_{26}P_6Sb_4Se_2$	$C_{70}H_{107}N_3O_{26}P_6Sb_4Se_2$
F.wt g/mol^{-1}	1885.25	2300.37	2349.53	2237.32
T, K	100(2)	100(2)	100(2)	100(2)
Crystal system	Triclinic	Tetragonal	Orthorhombic	Triclinic
Space group	P-1	I-4	Pbca	P-1
Crystal size mm^3	0.25 x 0.23 x 0.20	0.20 x 0.15 x 0.13	0.21 x 0.20 x 0.18	0.26 x 0.25 x 0.20
a, Å	13.7494(11)	13.1600(14)	24.238(2)	13.9433(12)
b, Å	17.0061(14)	13.1600(14)	27.209(2)	15.4586(13)
c, Å	19.0775(16)	26.614(4)	30.177(2)	21.2655(18)
α, deg	74.8200(10)	90	90	82.2130(10)
β, deg	83.9290(10)	90	90	83.783(2)
γ, deg	70.7270(10)	90	90	71.5500(10)
V, Å^3	4062.9(6)	4609.1(12)	19901(3)	4297.5(6)
Z	2	2	8	2
D_{calcd} Mg/m^3	1.541	1.658	1.568	1.634
μ, mm^{-1}	1.499	1.374	1.971	2.271
F(000)	1900	2304	9456	2104
Theta range, deg	1.307 to 26.384	1.530to 26.404	1.312 to 26.059	1.397 to 26.375
Index ranges	-17<=h<=17 -21<=k<=21 -23<=l<=23	-16<=h<=16 -16<=k<=16 -33<=l<=33	-29<=h<=29 -33<=k<=33 -37<=l<=37	-17<=h<=17 -19<=k<=19 -26<=l<=26
Total reflns	43677	24759	200512	46258
Ind. reflns / R(int)	16508/0.0218	4738/0.0307	19642/0.0883	17457/0.0431
Completeness to θ_{max}, %	99.6	100.0	100.0	99.7
GooF(F^2)	1.052	1.076	1.098	1.036
R1(F)[I>2σ(I)]	0.0244	0.0595	0.0569	0.0359
$wR_2(F^2)$ (all data)	0.0642	0.1604	0.1700	0.0825
Largest diff peak/hole, e.Å^{-3}	0.951/-0.507	2.936/-0.550	3.038/-0.963	0.880/-0.486

Crystal data for Compound **5**

Formula	$C_{50}H_{77}NO_{17}P_4Sb_2Se_2$
F.wt g/mol^{-1}	1489.43
T, K	100
Crystal system	Monoclinic
Space group	$P2_1/c$
Crystal size mm^3	0.12 x 0.10 x 0.09
a, Å	17.124(3)
b, Å	16.730(3)
c, Å	22.614(4)
α, deg	90
β, deg	107.967(4)
γ, deg	90
V, Å^3	6162.6(18)
Z	4
D_{calcd} Mg/m^3	1.605
μ, mm^{-1}	2.226
F(000)	3000
Theta range, deg	1.250 to 26.412
Index ranges	-21<=h<=21 -20<=k<=20 -28<=l<=28
Total reflns	64916
Ind. reflns / R(int)	12629
Completeness to θ_{max}, %	99.7
GooF(F^2)	1.107
R1(F)[I>2σ(I)]	0.0911
$wR_2(F^2)$ (all data)	0.2173
Largest diff peak/hole, e.Å^{-3}	2.924/-1.123

Table S2: Compound **1** selected bond lengths (Å) and bond angles (deg)

Sb(1)-O(3)	1.9196(14)	P(1)-O(6)	1.5371(16)
Sb(1)-O(2)	1.9389(15)	P(1)-O(23)	1.5400(16)
Sb(1)-O(4)	2.0134(15)	P(2)-O(11)	1.5302(16)
Sb(1)-O(1)	2.0615(15)	P(2)-O(9)	1.5345(16)
Sb(1)-O(5)	2.0884(15)	P(2)-O(20)	1.5610(17)
Sb(2)-O(7)	1.9218(14)	P(3)-O(19)	1.5021(17)
Sb(2)-O(2)	1.9350(15)	P(3)-O(8)	1.5480(16)
Sb(2)-O(8)	2.0123(15)	P(3)-O(10)	1.5545(16)
Sb(2)-O(6)	2.0508(15)	P(4)-O(22)	1.5350(16)
Sb(2)-O(9)	2.0984(15)	P(4)-O(12)	1.5365(16)
Sb(3)-O(7)	1.9228(14)	P(4)-O(14)	1.5395(16)
Sb(3)-O(13)	1.9389(15)	P(5)-O(5)	1.5325(16)
Sb(3)-O(10)	2.0080(15)	P(5)-O(16)	1.5406(16)
Sb(3)-O(12)	2.0597(15)	P(5)-O(17)	1.5604(17)
Sb(3)-O(11)	2.0898(15)	P(6)-O(18)	1.5108(17)
Sb(4)- O(3)	1.9148(14)	P(6)-O(4)	1.5447(16)
Sb(4)-O(14)	2.0343(15)	P(6)-O(15)	1.5483(16)
Sb(4)-O(13)	1.9417(14)		
Sb(4)-O(15)	2.0199(15)	Sb(3)-O(13)-Sb(4)	132.81(8)
Sb(4)-O(16)	2.0775(15)	Sb(2)-O(7)-Sb(3)	139.86(8)
P(1)-O(1)	1.5363(16)	Sb(4)-O(3)-Sb(1)	140.10(8)
		Sb(2)-O(2)-Sb(1)	133.32(8)

Table S3: Compound **2** selected bond lengths (Å) and bond angles (deg)

Sb(1)-O(1)	1.982(9)	P(2)-O(3)	1.525(8)
Sb(1)-O(4)	1.918(5)	P(3)-O(6)	1.504(10)
Sb(1)-O(8)#1	2.014(8)	P(3)-O(7)	1.549(8)
Sb(1)-O(3)#2	2.016(7)	P(3)-O(8)	1.558(8)
Sb(1)-O(7)	2.036(8)	Sb(1)#1-O(4)-Sb(1)	136.3(7)
O(3)-Sb(1)#3	2.017(7)	O(4)-Sb(1)-O(1)	82.7(4)
O(8)-Sb(1)#1	2.015(8)	O(4)-Sb(1)-O(8)#1	91.5(3)
P(2)-O(2)	1.506(10)	O(1)-Sb(1)-O(8)#1	173.4(3)
P(2)-O(1)	1.512(10)	O(4)-Sb(1)-O(3)#2	90.4(3)

O(1)-Sb(1)-O(3)#2	93.9(4)	O(8)#1-Sb(1)-O(7)	88.1(4)
O(8)#1-Sb(1)-O(3)#2	89.4(3)	O(3)#2-Sb(1)-O(7)	177.5(4)
O(4)-Sb(1)-O(7)	90.2(3)		
O(1)-Sb(1)-O(7)	88.7(4)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,z #2 y+1/2,-x+1/2,-z+1/2 #3 -y+1/2,x-1/2,-z+1/2

Table S4: Compound **3** selected bond lengths (Å) and bond angles (deg)

Sb(1)-O(1)	1.937(5)	P(2)-O(20)	1.518(5)
Sb(1)-O(2)	2.014(5)	P(2)-O(15)	1.551(5)
Sb(1)-O(5)	2.017(5)	P(2)-O(3)	1.555(5)
Sb(1)-O(3)	2.017(5)	P(3)-O(19)	1.499(6)
Sb(1)-O(4)	2.022(5)	P(3)-O(10)	1.552(5)
Sb(2)-O(6)	1.939(4)	P(3)-O(8)	1.567(5)
Sb(2)-O(5)	2.005(5)	P(4)-O(18)	1.510(5)
Sb(2)-O(8)	2.010(5)	P(4)-O(7)	1.554(5)
Sb(2)-O(4)	2.025(5)	P(4)-O(9)	1.557(5)
Sb(2)-O(7)	2.040(5)	P(5)-O(21)	1.513(6)
Sb(3)-O(6)	1.911(5)	P(5)-O(23)	1.537(6)
Sb(3)-O(9)	2.017(5)	P(5)-O(22)	1.538(6)
Sb(3)-O(10)	2.024(5)	P(6)-O(24)	1.509(6)
Sb(3)-O(11)	2.027(5)	P(6)-O(25)	1.520(6)
Sb(3)-O(12)	2.033(5)	P(6)-O(26)	1.524(6)
Sb(4)-O(1)	1.915(5)	Sb(1)-O(4)-Sb(2)	102.3(2)
Sb(4)-O(15)	2.019(5)	Sb(2)-O(5)-Sb(1)	103.2(2)
Sb(4)-O(16)	2.019(5)	Sb(4)-O(1)-Sb(1)	139.7(3)
Sb(4)-O(13)	2.025(5)	Sb(3)-O(6)-Sb(2)	139.3(3)
Sb(4)-O(14)	2.032(5)	Se(2)-O(11)-Sb(3)	123.4(3)
Se(1)-O(13)	1.725(5)	Se(1)-O(12)-Sb(3)	122.7(3)
Se(1)-O(12)	1.735(5)	Se(2)-O(14)-Sb(4)	123.7(3)
Se(2)-O(14)	1.726(5)	Se(1)-O(13)-Sb(4)	124.3(3)
Se(2)-O(11)	1.740(5)	O(14)-Se(2)-O(11)	100.9(2)
P(1)-O(17)	1.499(6)	O(13)-Se(1)-O(12)	99.7(2)
P(1)-O(16)	1.543(5)	O(1)-Sb(4)-C(52)	177.9(3)
P(1)-O(2)	1.550(5)	O(1)-Sb(1)-C(25)	177.5(2

O(6)-Sb(2)-C(20)	174.7(2)	O(6)-Sb(3)-C(43)	179.3(3)

Table S5: Compound **4** selected bond lengths (Å) and bond angles (deg)

Sb(1)-O(1)	1.901(2)	P(3)-O(12)	1.496(3)
Sb(1)-O(2)	2.009(2)	P(3)-O(14)	1.546(3)
Sb(1)-O(3)	2.028(2)	P(3)-O(13)	1.551(3)
Sb(1)-O(4)	2.030(2)	P(4)-O(17)	1.503(3)
Sb(1)-O(5)	2.041(2)	P(4)-O(11)	1.555(3)
Sb(2)-O(1)	1.934(2)	P(4)-O(18)	1.557(3)
Sb(2)-O(8)	2.001(2)	P(5)-O(23)	1.531(3)
Sb(2)-O(7)	2.010(2)	P(5)-O(21)	1.531(3)
Sb(2)-O(9)	2.026(3)	P(5)-O(22)	1.543(3)
Sb(2)-O(6)	2.032(2)	P(6)-O(26)	1.504(3)
Sb(3)-O(10)	1.946(2)	P(6)-O(25)	1.539(3)
Sb(3)-O(8)	2.001(2)	P(6)-O(24)	1.548(3)
Sb(3)-O(11)	2.012(2)		
Sb(3)-O(13)	2.020(2)		
Sb(3)-O(9)	2.029(3)	Sb(1)-O(1)-Sb(2)	138.09(13)
Sb(4)-O(10)	1.921(2)	Se(2)-O(4)-Sb(1)	127.10(13)
Sb(4)-O(14)	2.012(2)	Sb(2)-O(9)-Sb(3)	101.33(11)
Sb(4)-O(18)	2.017(2)	Se(1)-O(15)-Sb(4)	125.32(13)
Sb(4)-O(16)	2.019(2)	Sb(2)-O(8)-Sb(3)	103.20(11)
Sb(4)-O(15)	2.044(2)	Se(2)-O(16)-Sb(4)	123.14(13)
Se(1)-O(5)	1.721(2)	Se(1)-O(5)-Sb(1)	128.43(13)
Se(1)-O(15)	1.724(2)	Sb(4)-O(10)-Sb(3)	138.21(13)
Se(2)-O(16)	1.730(2)	O(16)-Se(2)-O(4)	98.59(11)
Se(2)-O(4)	1.731(2)	O(5)-Se(1)-O(15)	101.76(12)
P(1)-O(20)	1.499(3)		
P(1)-O(7)	1.550(3)		
P(1)-O(3)	1.555(3)		
P(2)-O(19)	1.501(3)		
P(2)-O(6)	1.554(3)		
P(2)-O(2)	1.561(2)		

Table S6: Compound **5** selected bond lengths (Å) and bond angles (deg)

Sb(1)-O(17)	1.929(8)	P(1)-O(1)	1.551(8)
Sb(1)-O(14)	1.997(8)	P(2)-O(16)	1.488(9)
Sb(1)-O(10)	2.014(8)	P(2)-O(15)	1.556(8)
Sb(1)-O(2)	2.034(8)	P(2)-O(14)	1.558(8)
Sb(1)-O(13)	2.038(8)	P(3)-O(11)	1.496(10)
Sb(2)-O(17)	1.957(7)	P(3)-O(9)	1.540(9)
Sb(2)-O(1)	1.998(8)	P(3)-O(10)	1.550(9)
Sb(2)-O(4)	2.005(8)	P(4)-O(6)	1.503(9)
Sb(2)-O(15)	2.007(8)	P(4)-O(5)	1.532(8)
Sb(2)-O(7)	2.023(8)	P(4)-O(4)	1.555(9)
Se(1)-O(13)	1.707(8)	Sb(1)-O(17)-Sb(2)	134.7(4)
Se(1)-O(12)	1.732(8)	Se(1)-O(13)-Sb(1)	123.0(4)
Se(2)-O(8)	1.730(8)	Se(2)-O(7)-Sb(2)	124.0(4)
Se(2)-O(7)	1.735(8)	O(13)-Se(1)-O(12)	101.5(4)
P(1)-O(3)	1.503(9)	O(8)-Se(2)-O(7)	103.9(4)
P(1)-O(2)	1.550(9)		