Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

In situ Generated Polysiloxanes Stabilizing μ_3-Oxo Bridged Sb_3Triangles

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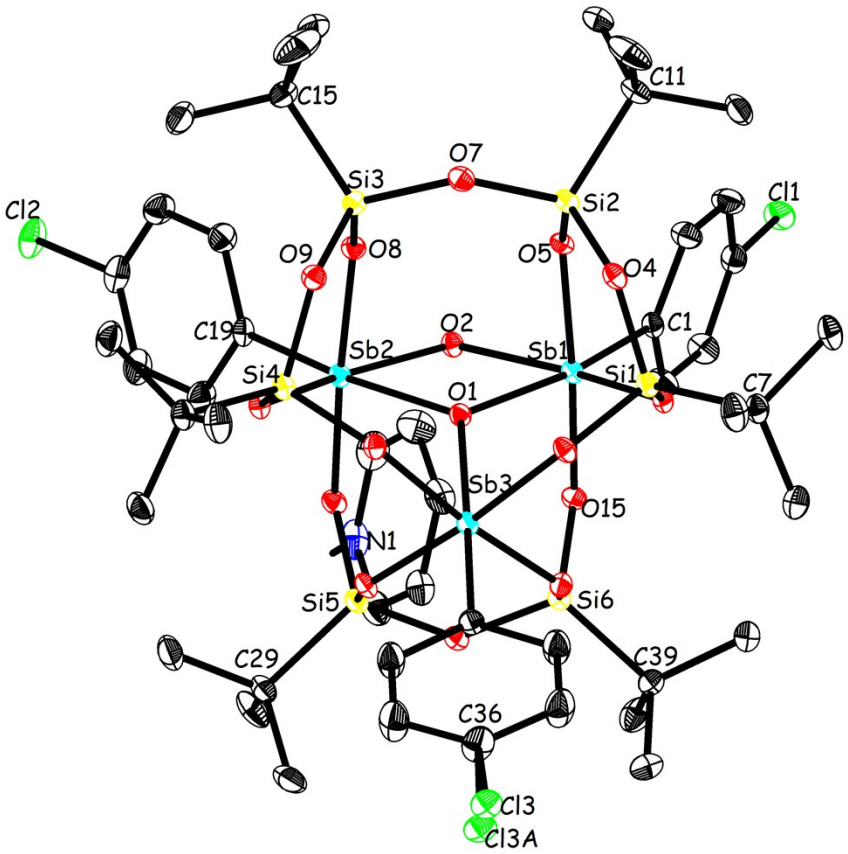

Figure. S1 ORTEP view of molecular structure **1** with thermal ellipsoids shown at 30% probability and hydrogen atoms are omitted for clarity.

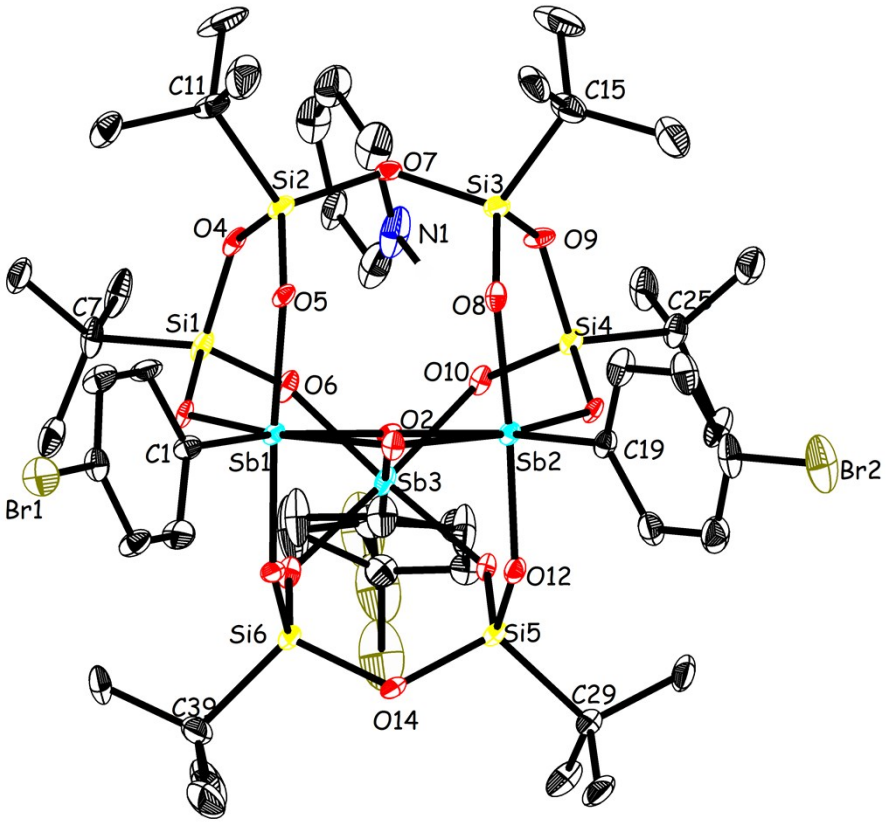

Figure. S2 ORTEP view of molecular structure **2** with thermal ellipsoids shown at 30% probability and hydrogen atoms are omitted for clarity.

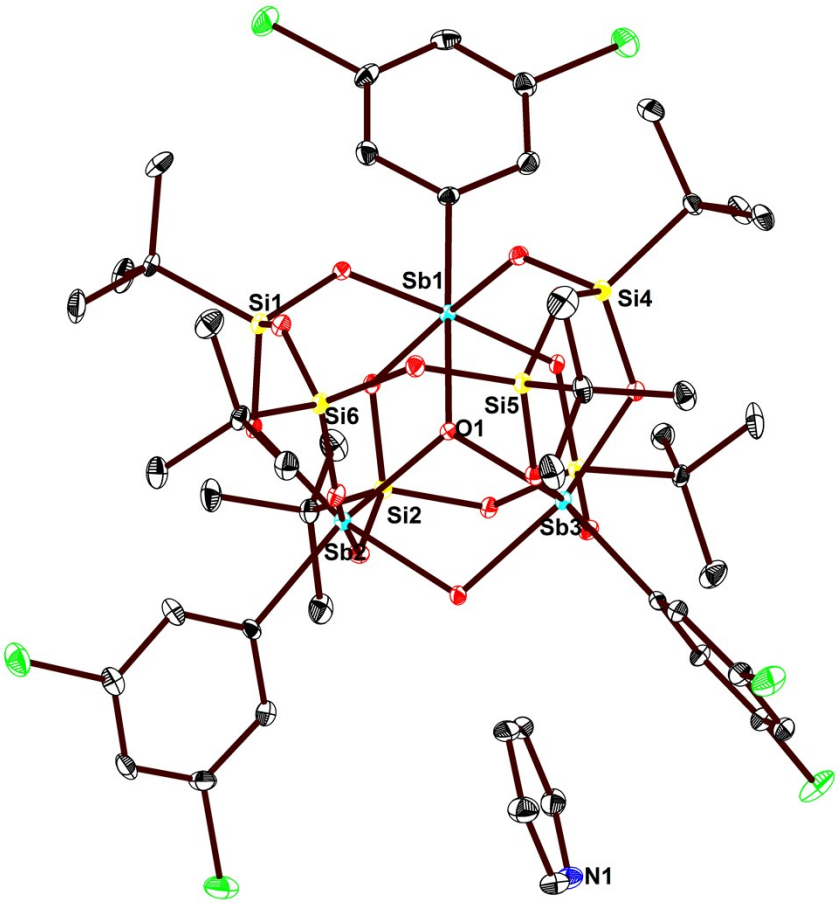

Figure. S3 ORTEP view of molecular structure **3** with thermal ellipsoids shown at 30% probability and hydrogen atoms are omitted for clarity.

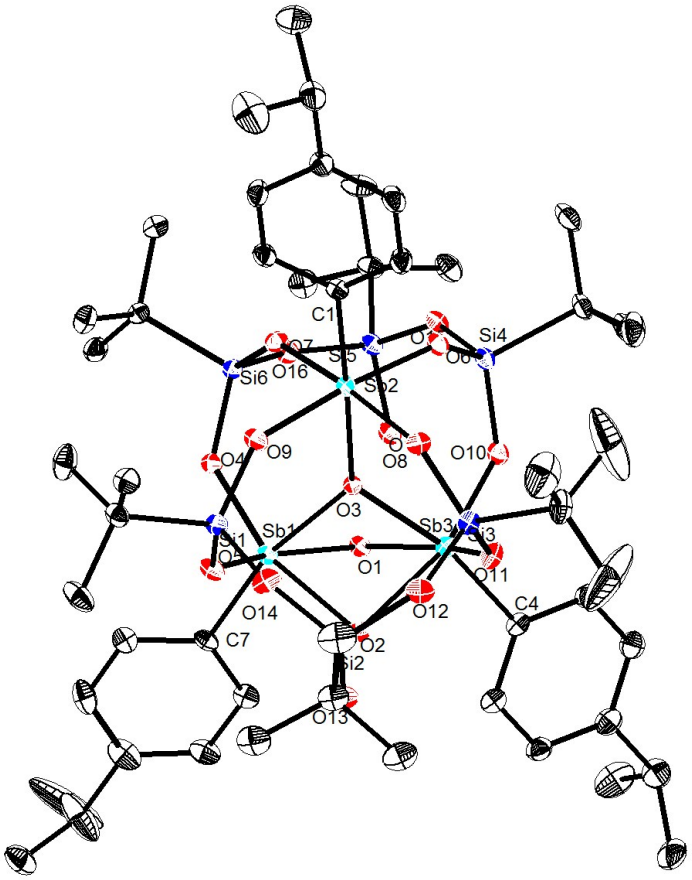

Figure. S4 ORTEP view of molecular structure **4** with thermal ellipsoids shown at 30% probability and hydrogen atoms are omitted for clarity.

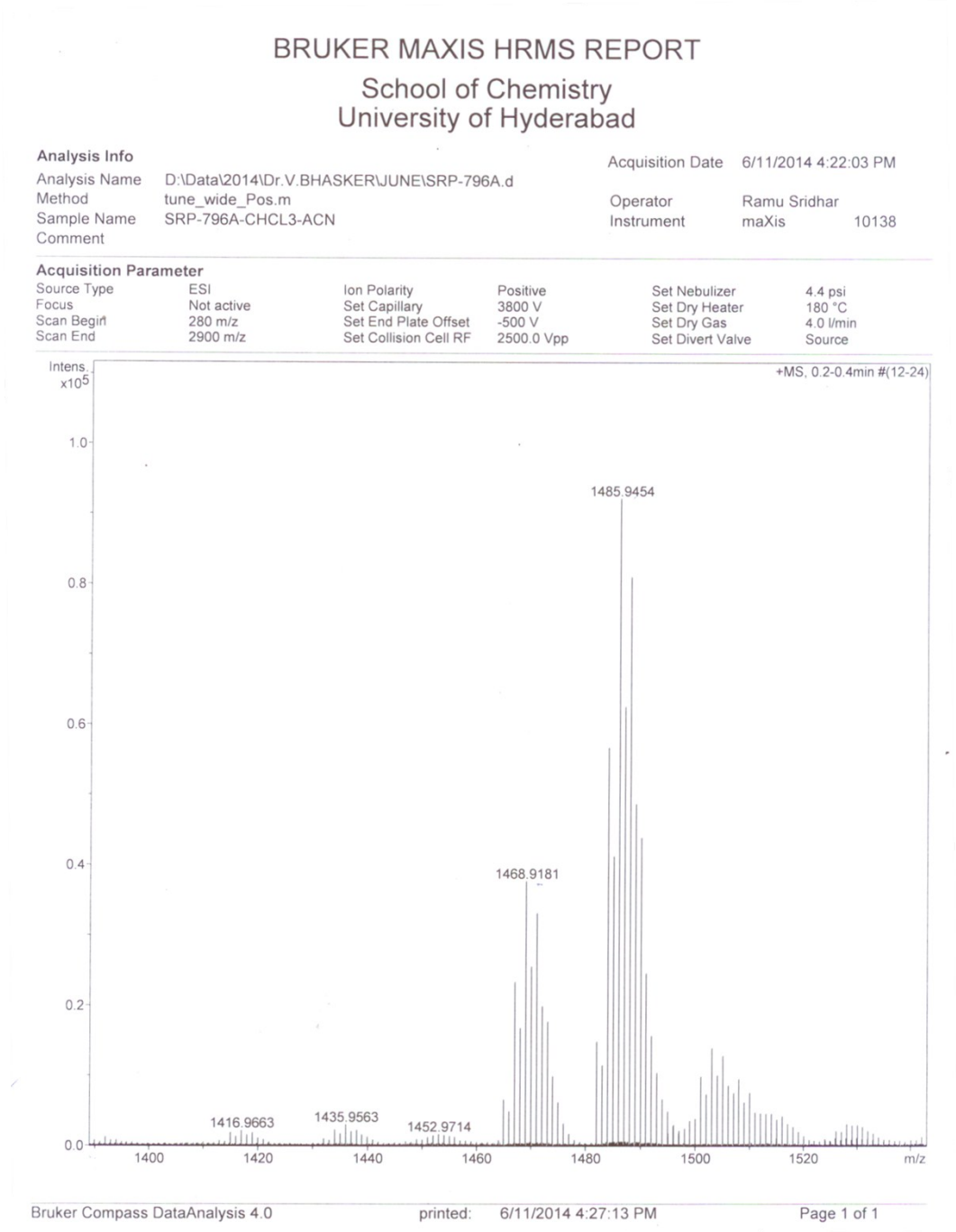

Figure. S5 ESI-MS of compound **1** in positive ion mode.

HRMS (ESI) calcd for ($C_{42}H_{67}Cl_3O_{16}Sb_3Si_6$) [*M*+2H]$^+$ 1468.9356; found 1468.9181

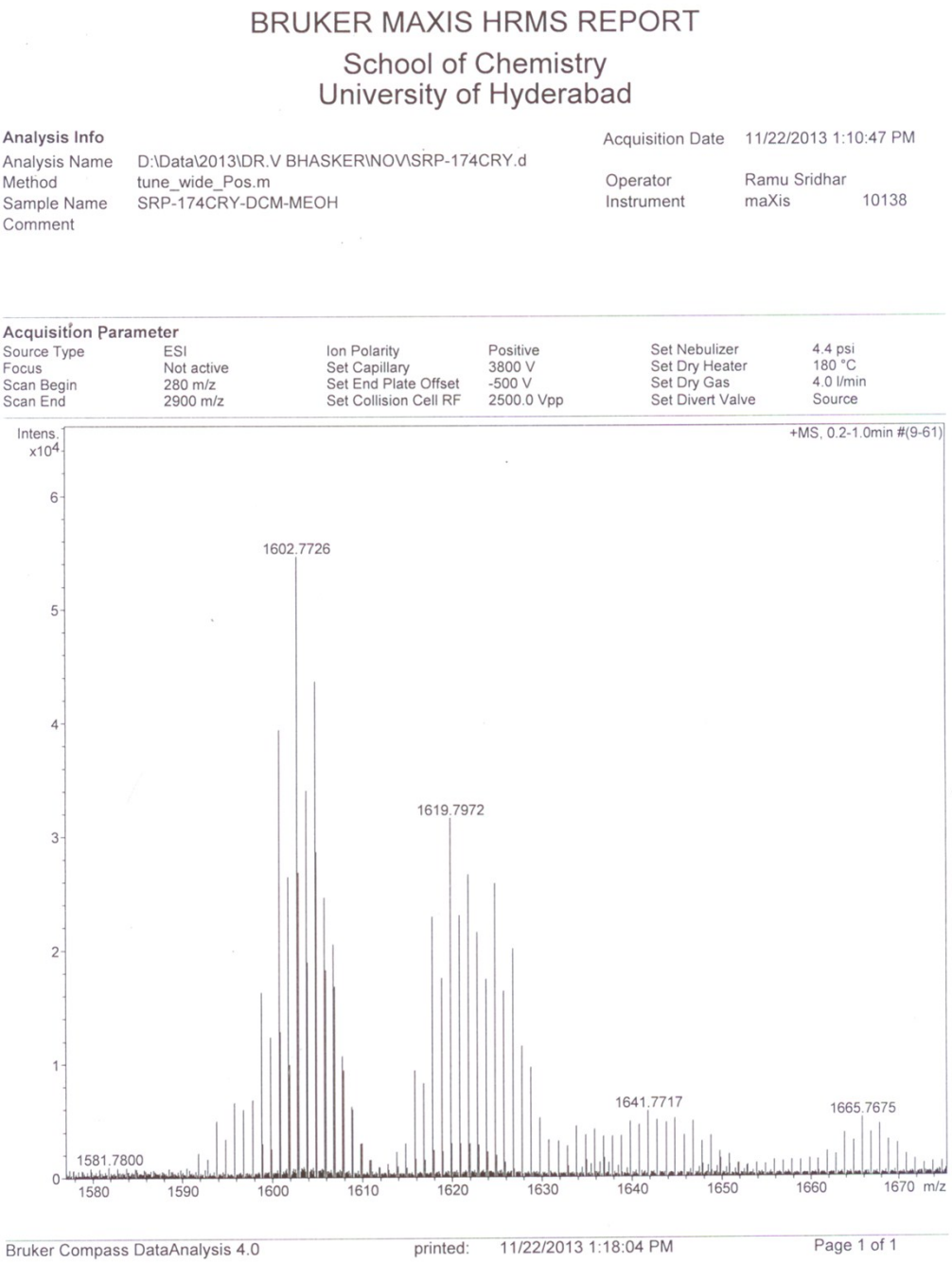

Figure. S6 ESI-MS of compound **2** in positive ion mode.

HRMS (ESI) calcd for ($C_{42}H_{67}Br_3O_{16}Sb_3Si_6$) [*M*+2H]$^+$ 1602.7829; found 1602.7726

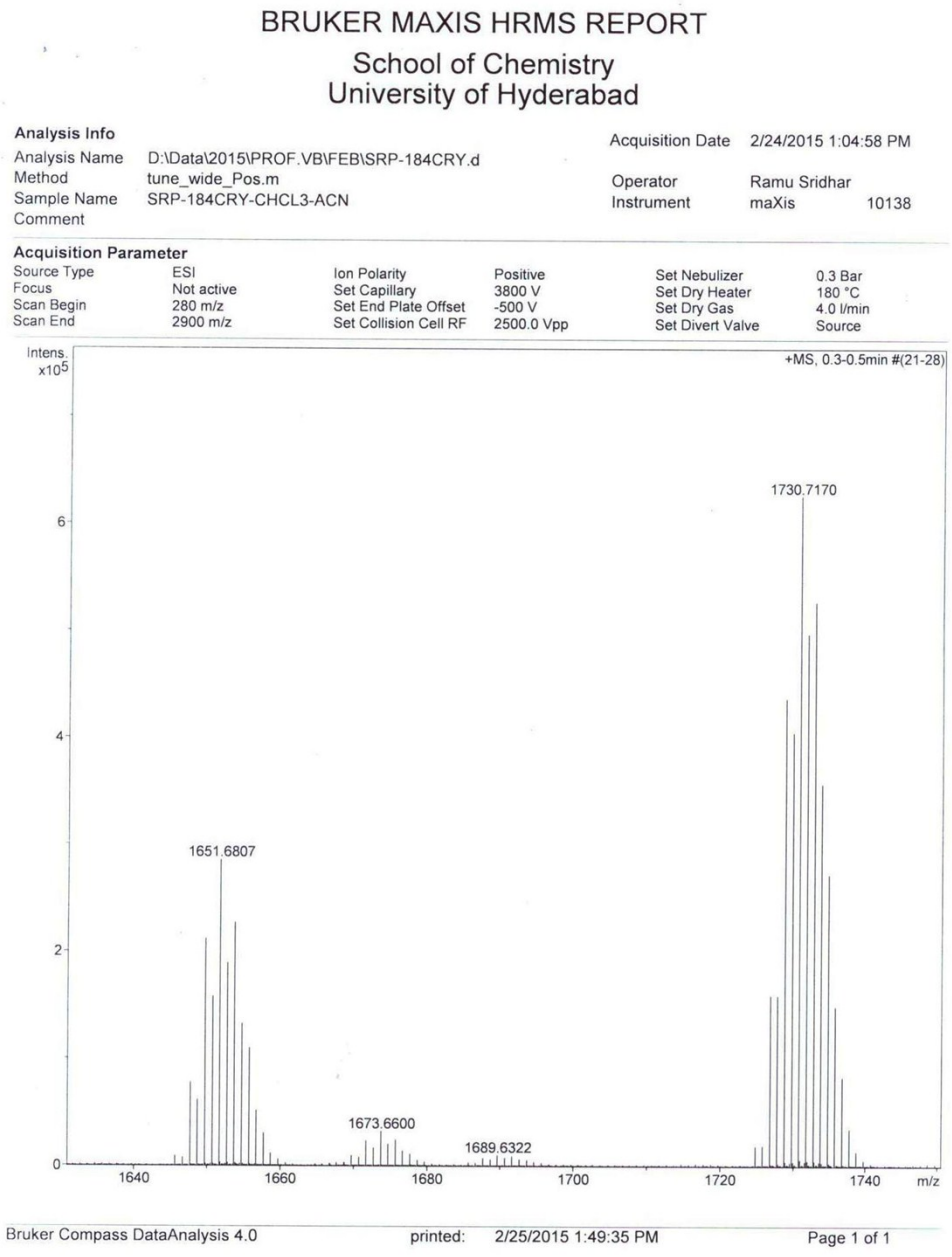

Figure S7. ESI-MS of compound **3** in positive ion mode.

HRMS (ESI) calcd for ($C_{42}H_{63}Cl_6O_{16}Sb_3Si_6$) [*M*+2K+3H]$^+$ 1651.0119; found 1651.6807

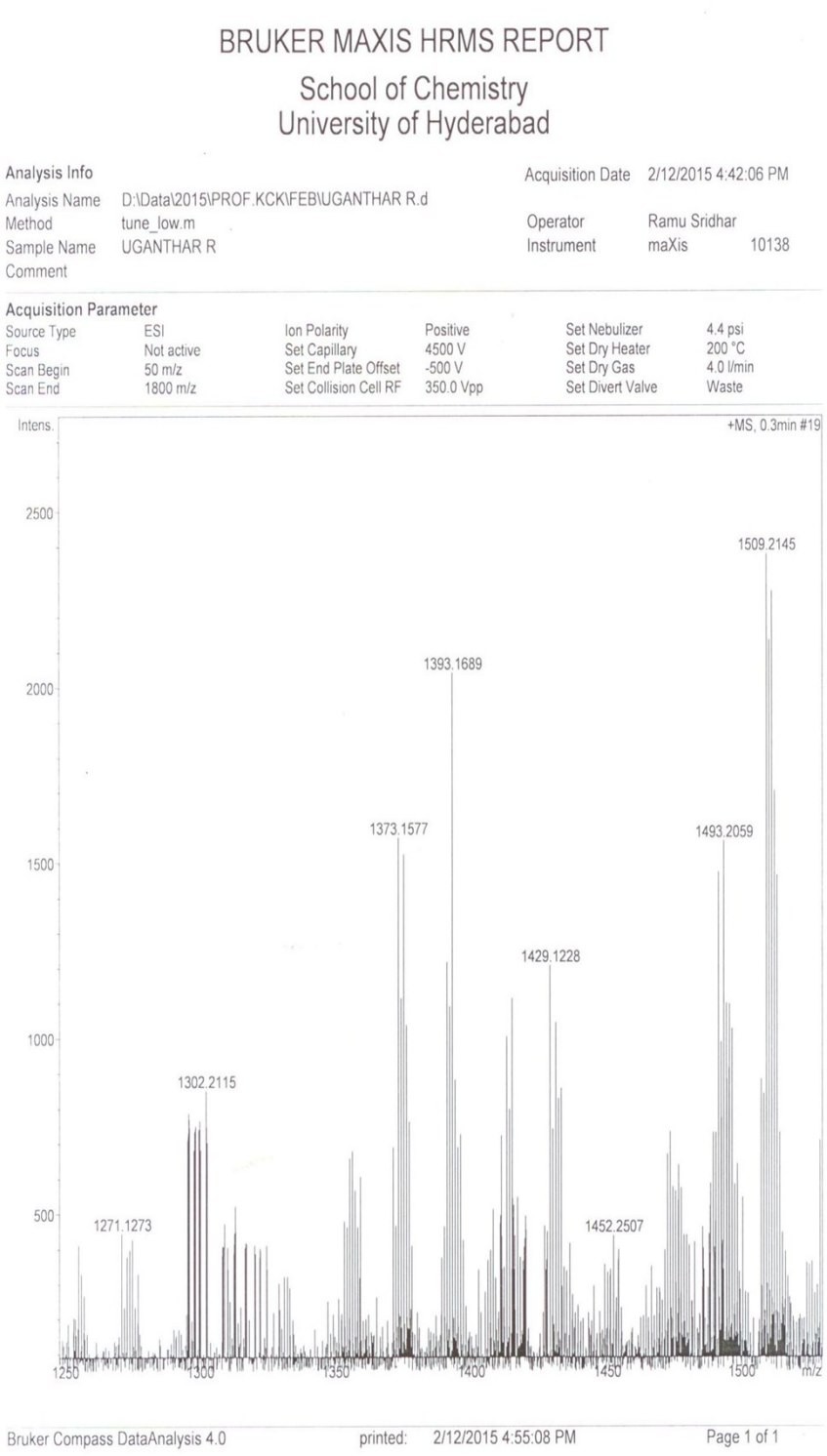

Figure S8. ESI-MS of compound **4** in positive ion mode.

HRMS (ESI) calcd for ($C_{51}H_{89}O_{17}Sb_3Si_6$) [*M*+H]$^+$ 1509.1917; found 1509.2145.

Table S1: Crystal data and structure refinement details for compounds 1-4.

	1	**2**	**3**	**4**
formula	$C_{50}H_{81}Cl_3O_{17}NSb_3Si_6$	$C_{50}H_{81}Br_{2.99}O_{17}NSb_3Si_6$	$C_{51}H_{75}Cl_6N_3O_{16}Sb_3Si_6$	$C_{61}H_{106}N_5O_{17}Si_6Sb_3$
M	1608.29	1740.87	1732.63	1715.29
T(K)	100(2)	100(2)	100(2)	100(2)
λ (Å)	0.71073	0.71073	0.71073	0.71073
crystal system	Monoclinic	Monoclinic	Triclinic	Triclinic
space group	*P*2(1)/*n*	*P*2(1)/*c*	*P*-1	P -1
a(Å)	11.7571(10)	11.741 (10)	12.3465(14)	15.0752(12)
b(Å)	28.053(2)	28.21 (2)	13.1911(15)	16.0739(13)
c(Å)	21.7258 (19)	21.863 (18)	23.155(3)	16.9080(14)
α(deg)	90	90	99.920(2)	100.488(10)
β(deg)	104.9530 (10)	104.994 (14)	95.997(2)	91.038(10)
γ(deg)	90	90	105.084(2)	91.518(10)
V(Å^3)	6923.0 (10)	6993 (10)	3542.3(7)	4026.1(6)
Z	4	4	2	2
Dcalcd (Mg m^{-3})	1.543	1.653	1.624	1.415
μ (mm^{-1})	1.442	3.021	1.524	1.149
F(000)	3248	3463	1738	1760
θ range (deg)	1.212-25.088	1.205-25.026	1.634 to 26.441	1.225 to 25.038
index range	$-14 \le h \le 14$	$-13 \le h \le 13$	$-15 \le h \le 15$	$-17 \le h \le 17$
	$-33 \le k \le 33$	$-33 \le k \le 33$	$-16 \le k \le 16$	$-19 \le k \le 19$
	$-25 \le l \le 25$	$-25 \le l \le 25$	$-26 \le l \le 28$	$-20 \le l \le 20$
reflections collected	66491	66654	28581	38923
goodness-of-fit (GOF) on F^2	1.042	1.104	1.026	1.041
final R indices	$R_1 = 0.0311$	$R_1 = 0.0694$	$R_1 = 0.0413$	$R_1 = 0.0279$
$R_1[I > 2\sigma(I)]$	$wR_2 = 0.0776$	$wR_2 = 0.1310$	$wR_2 = 0.0943$	$wR_2 = 0.0704$
R indicies (all data)	$R_1 = 0.0347$	$R_1 = 0.0957$	$R_1 = 0.0505$	$R_1 = 0.0313$
	$wR_2 = 0.0796$	$wR_2 = 0.1414$	$wR_2 = 0.0991$	wR2 = 0.0725
large diff. peak and hole (e Å^{-3})	1.645 and -0.920	1.557 and -1.348	1.982 and -0.811	0.886 and -1.004

Table S2: Selected bond lengths (Å) and bond angles (°) parameters for compound 1

Sb(1)-C(1)	2.121(3)	Si(4)-O(10)	1.637(2)
Sb(2)-C(19)	2.117(3)	Si(4)-O(9)	1.644(2)
Sb(3)-C(33)	2.112(3)	Si(5)-O(13)	1.626(2)
Sb(1)-O(3)	1.939(2)	Si(5)-O(12)	1.629(2)
Sb(1)-O(15)	1.960(2)	Si(5)-O(14)	1.645(2)
Sb(1)-O(5)	1.988(2)	Si(6)-O(16)	1.620(2)
Sb(1)-O(2)	2.087(2)	Si(6)-O(15)	1.628(2)
Sb(1)-O(1)	2.094(2)	Si(6)-O(14)	1.646(2)
Sb(2)-O(11)	1.946(2)	Sb(1)-O(1)-Sb(2)	105.31(9)
Sb(2)-O(12)	1.953(2)	Sb(1)-O(1)-Sb(3)	124.84(10)
Sb(2)-O(8)	1.978(2)	Sb(2)-O(1)-Sb(3)	125.58(10)
Sb(2)-O(2)	2.082(2)	Sb(2)-O(2)-Sb(1)	106.61(11)
Sb(2)-O(1)	2.110(2)	Si(4)-O(11)-Sb(2)	125.82(13)
Sb(3)-O(16)	1.951(2)	Si(1)-O(3)-Sb(1)	125.31(13)
Sb(3)-O(13)	1.953(2)	Si(6)-O(15)-Sb(1)	130.55(13)
Sb(3)-O(6)	1.953(2)	Si(4)-O(10)-Sb(3)	133.43(13)
Sb(3)-O(10)	1.983(2)	Si(5)-O(12)-Sb(2)	130.12(13)
Sb(3)-O(1)	2.176(2)	Si(6)-O(16)-Sb(3)	127.23(13)
Si(1)-O(6)	1.621(2)	Si(2)-O(4)-Si(1)	130.88(15)
Si(1)-O(3)	1.635(2)	Si(2)-O(5)-Sb(1)	132.56(13)
Si(1)-O(4)	1.639(2)	Si(1)-O(6)-Sb(3)	133.36(13)
Si(2)-O(5)	1.626(2)	Si(5)-O(14)-Si(6)	124.78(14)
Si(2)-O(4)	1.631(2)	Si(5)-O(13)-Sb(3)	127.28(13)
Si(2)-O(7)	1.637(2)	Si(3)-O(7)-Si(2)	138.01(15)
Si(3)-O(8)	1.618(2)	Si(3)-O(8)-Sb(2)	134.17(13)
Si(3)-O(7)	1.633(2)	Si(3)-O(9)-Si(4)	129.84(15)
Si(3)-O(9)	1.639(2)		
Si(4)-O(11)	1.626(2)		

Table S3: Selected bond lengths (Å) and bond angles (°) parameters for compound 2

Sb(1)-C(1)	2.123(8)	Si(4)-O(11)	1.612(6)
Sb(2)-C(19)	2.121(8)	Si(4)-O(9)	1.632(6)
Sb(3)-C(33B)	2.098(13)	Si(4)-O(10)	1.640(6)
Sb(3)-C(33A)	2.118(13)	Si(5)-O(12)	1.616(6)
Sb(1)-O(3)	1.926(5)	Si(5)-O(13)	1.625(6)
Sb(1)-O(15)	1.960(5)	Si(5)-O(14)	1.640(6)
Sb(1)-O(5)	1.979(5)	Si(6)-O(16)	1.612(6)
Sb(1)-O(1)	2.083(5)	Si(6)-O(15)	1.617(6)
Sb(1)-O(2)	2.0852(13)	Si(6)-O(14)	1.635(6)
Sb(2)-O(11)	1.943(5)	Sb(2)-O(2)-Sb(1)	106.17(5)
Sb(2)-O(12)	1.950(5)	Sb(1)-O(1)-Sb(2)	105.5(2)
Sb(2)-O(8)	1.959(5)	Sb(1)-O(1)-Sb(3)	124.7(2)
Sb(2)-O(2)	2.0844(13)	Sb(2)-O(1)-Sb(3)	125.7(2)
Sb(2)-O(1)	2.106(5)	Si(3)-O(8)-Sb(2)	134.2(3)
Sb(3)-O(13)	1.933(5)	Si(1)-O(3)-Sb(1)	125.3(3)
Sb(3)-O(6)	1.943(5)	Si(6)-O(15)-Sb(1)	130.5(3)
Sb(3)-O(16)	1.953(6)	Si(2)-O(5)-Sb(1)	132.2(3)
Sb(3)-O(10)	1.988(5)	Si(1)-O(6)-Sb(3)	133.9(3)
Sb(3)-O(1)	2.169(5)	Si(3)-O(9)-Si(4)	130.0(3)
Si(1)-O(6)	1.609(6)	Si(5)-O(12)-Sb(2)	130.0(3)
Si(1)-O(4)	1.634(6)	Si(5)-O(13)-Sb(3)	127.4(3)
Si(1)-O(3)	1.641(6)	Si(4)-O(10)-Sb(3)	133.0(3)
Si(2)-O(7)	1.628(6)	Si(4)-O(11)-Sb(2)	125.8(3)
Si(2)-O(4)	1.630(6)	Si(2)-O(4)-Si(1)	131.5(3)
Si(2)-O(5)	1.633(6)	Si(2)-O(7)-Si(3)	138.2(3)
Si(3)-O(8)	1.618(6)	Si(6)-O(16)-Sb(3)	126.9(3)
Si(3)-O(9)	1.631(6)	Si(6)-O(14)-Si(5)	124.6(4)
Si(3)-O(7)	1.635(6)		

Table S4: Selected bond lengths (Å) and bond angles (°) parameters for compound 3

Sb(1)-C(1)	2.117(4)	Si(4)-O(10)	1.629(3)
Sb(2)-C(19)	2.135(4)	Si(4)-O(11)	1.629(3)
Sb(3)-C(33)	2.122(4)	Si(4)-O(9)	1.641(3)
Sb(1)-O(3)	1.940(3)	Si(5)-O(13)	1.629(3)
Sb(1)-O(15)	1.961(3)	Si(5)-O(12)	1.633(3)
Sb(1)-O(5)	1.983(2)	Si(5)-O(14)	1.642(3)
Sb(1)-O(1)	2.073(3)	Si(6)-O(16)	1.626(3)
Sb(1)-O(2)	2.087(3)	Si(6)-O(15)	1.632(3)
Sb(2)-O(12)	1.939(3)	Si(6)-O(14)	1.648(3)
Sb(2)-O(11)	1.950(3)	Sb(1)-O(1)-Sb(2)	105.99(11)
Sb(2)-O(8)	1.958(2)	Sb(1)-O(1)-Sb(3)	124.24(12)
Sb(2)-O(1)	2.090(3)	Sb(2)-O(1)-Sb(3)	125.60(12)
Sb(2)-O(2)	2.103(3)	Sb(1)-O(2)-Sb(2)	105.02(12)
Sb(3)-O(16)	1.946(3)	Si(5)-O(12)-Sb(2)	129.47(16)
Sb(3)-O(6)	1.958(3)	Si(6)-O(15)-Sb(1)	129.00(16)
Sb(3)-O(13)	1.959(3)	Si(1)-O(6)-Sb(3)	132.20(16)
Sb(3)-O(10)	1.964(3)	Si(2)-O(5)-Sb(1)	132.71(16)
Sb(3)-O(1)	2.196(3)	Si(3)-O(8)-Sb(2)	133.82(15)
Si(1)-O(6)	1.624(3)	Si(1)-O(3)-Sb(1)	125.68(15)
Si(1)-O(3)	1.632(3)	Si(4)-O(10)-Sb(3)	132.65(16)
Si(1)-O(4)	1.640(3)	Si(5)-O(14)-Si(6)	124.77(17)
Si(2)-O(5)	1.621(3)	Si(5)-O(13)-Sb(3)	128.12(15)
Si(2)-O(4)	1.638(3)	Si(6)-O(16)-Sb(3)	128.20(15)
Si(2)-O(7)	1.641(3)	Si(4)-O(11)-Sb(2)	125.83(15)
Si(3)-O(8)	1.623(3)	Si(2)-O(4)-Si(1)	132.68(18)
Si(3)-O(7)	1.629(3)	Si(3)-O(7)-Si(2)	137.12(18)
Si(3)-O(9)	1.636(3)	Si(3)-O(9)-Si(4)	130.53(18)

Table S5: Selected bond lengths (Å) and bond angles (°) parameters for compound 4.

Sb(1)-C(1)	2.098(3)	Si(4)-O(10)	1.6012(19)
Sb(2)-C(10)	2.095(3)	Si(4)-O(11)	1.620(2)
Sb(3)-C(19)	2.095(3)	Si(4)-O(9)	1.6423(19)
Sb(1)-O(14)	1.9625(18)	Si(5)-O(11)	1.610(2)
Sb(1)-O(10)	1.9681(17)	Si(5)-O(13)	1.615(2)
Sb(1)-O(16)	1.9715(17)	Si(5)-O(12)	1.644(2)
Sb(1)-O(8)	1.9727(17)	Si(6)-O(14)	1.6077(19)
Sb(1)-O(5)	2.0959(16)	Si(6)-O(13)	1.628(2)
Sb(2)-O(15)	1.9147(17)	Si(6)-O(6)	1.6357(18)
Sb(2)-O(6)	1.9234(17)	Sb(2)-O(2)-Sb(3)	93.20(7)
Sb(2)-O(5)	2.0793(16)	Sb(2)-O(1)-Sb(3)	92.51(7)
Sb(2)-O(2)	2.1000(19)	Sb(3)-O(5)-Sb(2)	95.57 (7)
Sb(2)-O(1)	2.1014(18)	Sb(3)-O(5)-Sb(1)	131.79(8)
Sb(3)-O(9)	1.9176(18)	Sb(2)-O(5)-Sb(1)	132.64(8)
Sb(3)-O(7)	1.9236(18)	Si(6)-O(14)-Sb(1)	141.09(11)
Sb(3)-O(5)	2.0440(16)	Si(3)-O(16)-Sb(1)	138.97(11)
Sb(3)-O(2)	2.1033(18)	Si(1)-O(8)-Sb(1)	138.61(11)
Sb(3)-O(1)	2.1259(18)	Si(3)-O(15)-Sb(2)	134.18(10)
Si(1)-O(8)	1.6062(18)	Si(1)-O(7)-Sb(3)	135.21(11)
Si(1)-O(18)	1.6299(19)	Si(2)-O(17)-Si(3)	150.60(13)
Si(1)-O(7)	1.6400(19)	Si(6)-O(6)-Sb(2)	133.29(11)
Si(2)-O(17)	1.6053(19)	Si(4)-O(10)-Sb(1)	141.54(11)
Si(2)-O(18)	1.6189(19)	Si(2)-O(18)-Si(1)	142.80(12)
Si(2)-O(19)	1.6465(19)	Si(4)-O(9)-Sb(3)	135.35(11)
Si(3)-O(16)	1.6098(18)	Si(5)-O(13)-Si(6)	145.59(13)
Si(3)-O(17)	1.6297(19)	Si(5)-O(11)-Si(4)	145.05(14)
Si(3)-O(15)	1.6384(18)		