Assembling Anionic Sb(V)/(III) containing polyoxostibonates stabilized by triphenyltellurium cations

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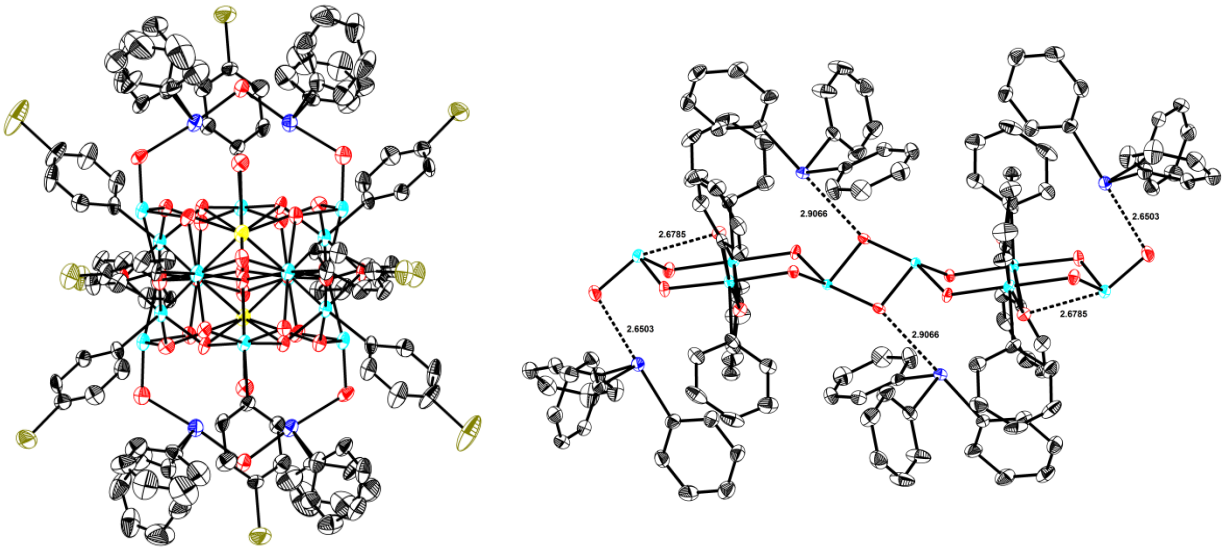


(a) (b)

Figure S1: ORTEP representations of **1**(a) and **2**(b)**.**Thermal ellipsoids are shownat 50% probability. Triphenyl telluriumcations, dichloromethane solvates in **1** and hydrozen atoms in **1**and **2** were omitted for clarity.

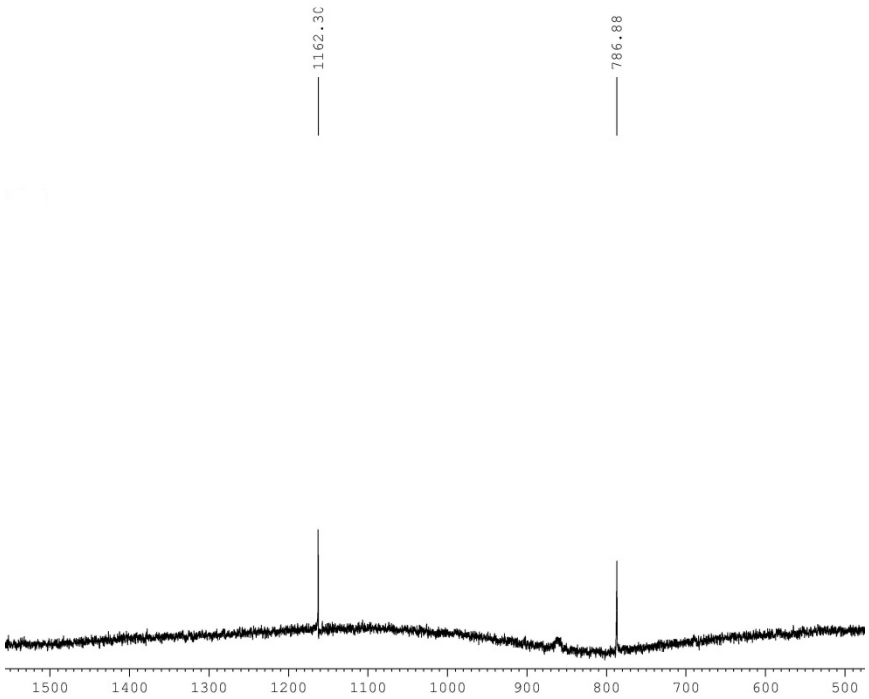


Figure s2: Solution ^{125}Te NMR spectrum of **1** in CDCl3.

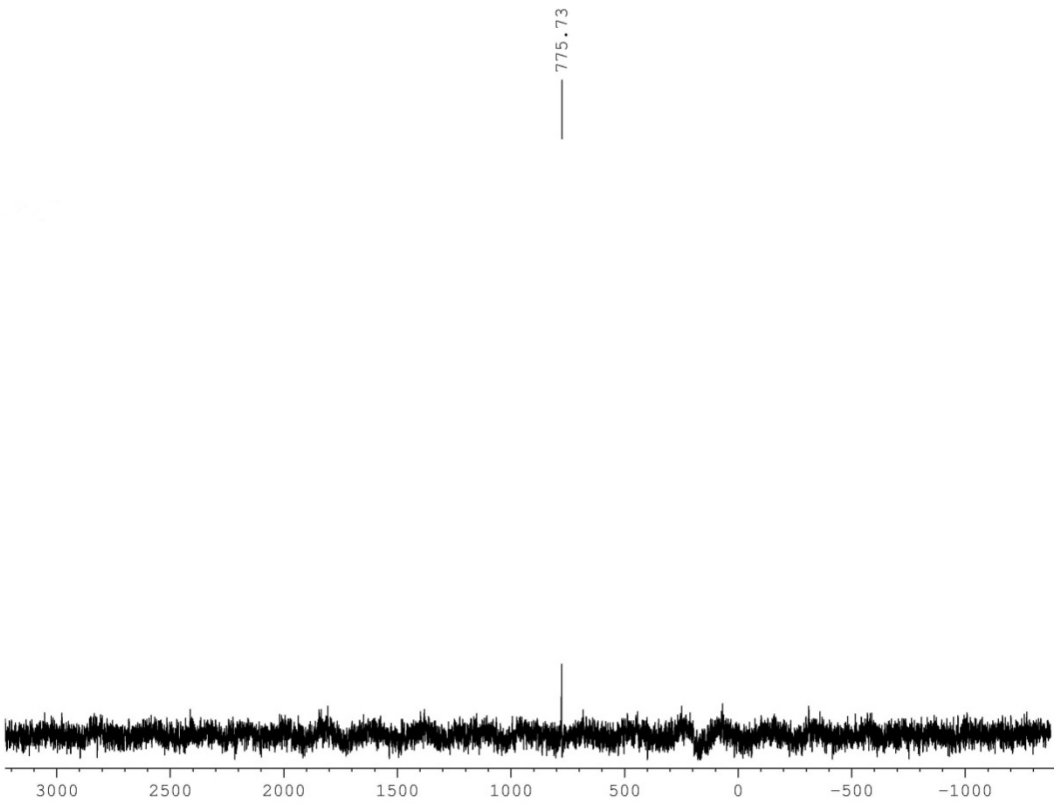


Figure s3: Solution ^{125}Te NMR spectrum of **2** in CD_3OD-CD_2Cl_2 mixture.

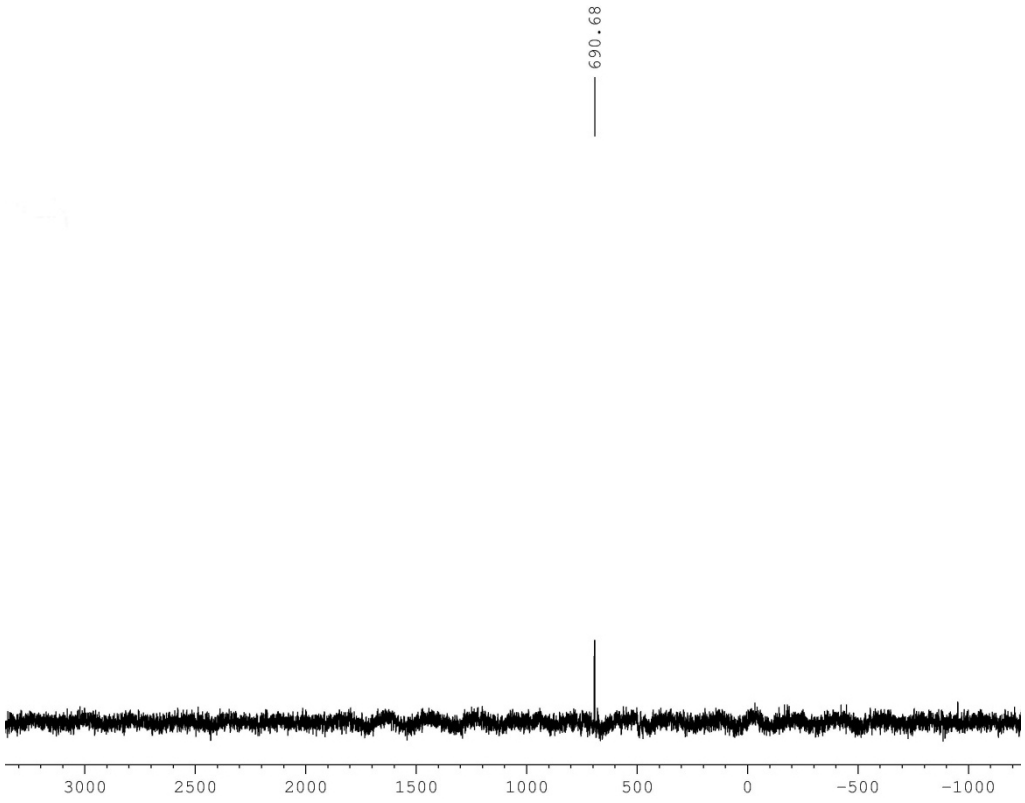
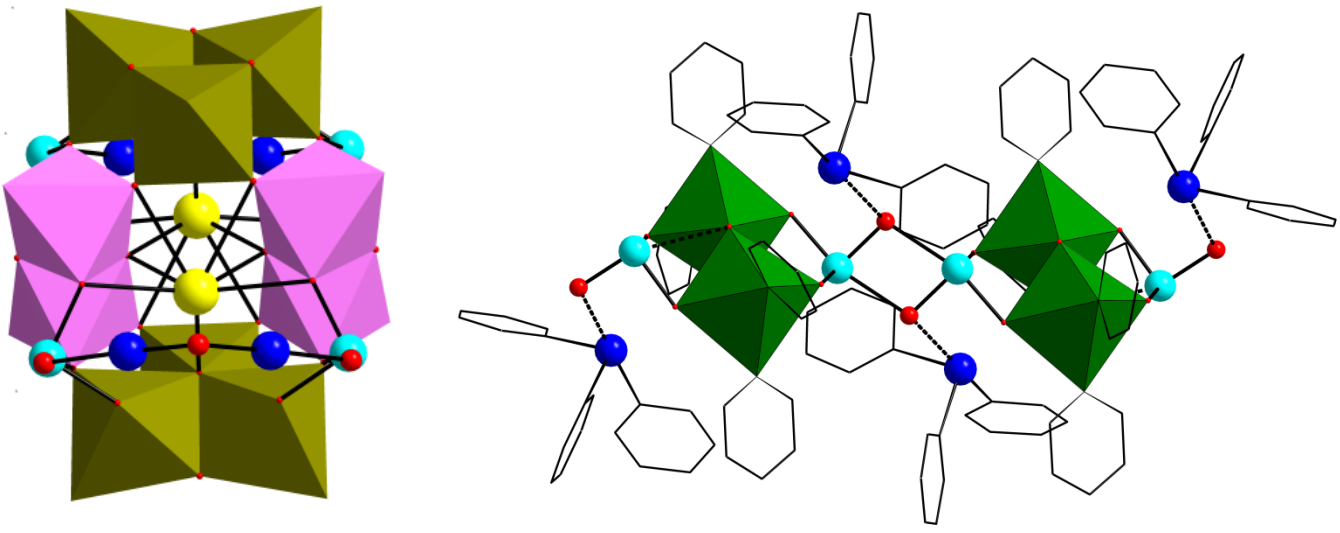


Figure s4: Solution ^{125}Te NMR spectrum of the filtrate (in reaction 2, diphenyltelluride) in CDCl3.



(a) (b)

Figure s5: (a) Polyhedral view of **1**. Aryl groups attached to Sb/Teand triphenyl tellurium cations are omitted for clarity. (b) Polyhedral view of **2**.

Table 1: Crystallographic data for **1-2**

	1	**2**
Formulae	$C_{148}H_{126}Br_{10}Cl_8Na_2O_{36}Sb_{14}Te_6$	$C_{120}H_{104}O_{16}Sb_8Te_4$
Fw.g mol^{-1}	6079.27	3286.43
Crystsyst	Monoclinic	Monoclinic
Cryst size.mm	0.16 x 0.12 x 0.08	0.22 x 0.14 x 0.08
Space group	P2(1)/n	P2(1)/n
a, Å	19.1789(13)	16.709(2)
b, Å	22.2219(15)	16.711(2)
c, Å	20.1740(13)	19.386(2)
α, deg	90	90
β, deg	91.9930(10)	90.193(2)
γ, deg	90	90
v, $Å^3$	8592.8(10)	5413.0(11)
Z	2	2
Dcacld.Mgm^{-3}	2.350	2.016
T.K	100	100
μ, mm^{-1}	5.685	3.087
F(000)	5672	3136
θ range,deg	1.36 to 25.07	1.61 to 26.49
index ranges	-22<=h<=22 -26<=k<=26 -24<=l<=24	-20<=h<=20 -20<=k<=20 -24<=l<=24
no. of.reflncolled	82138	56992
completeness to θ max	99.8 %	99.7 %
no. of. indeprefln/R_{int}	15219	11162
GooF(F^2)	1.030	1.123
R_1 (F) (I>2σ(I))	0.0554	0.0442
wR_2 (F^2) (All data)	0.1503	0.1061
largest diff peak/hole,e $Å^{-3}$	2.897/ -1.861	2.802/ -1.824

Table 2: Selected bond lengths(Å) and bond angles(º) in **1**

Sb1-O5 = 2.186(7)	Sb4-O14* = 2.091(7)	Te1-O15 = 2.175(8)	C7-Sb2-O5 = 168.3(3)
Sb1-O1 = 1.966(6)	Sb5-O1* = 1.962(7)	Te2-O16 = 2.258(8)	C7-Sb2-O10* = 99.8(3)
Sb1-O2 = 1.984(7)	Sb5-O11 = 2.080(7)	Te1-O17 = 2.053(8)	O2-Sb2-O13 = 167.8(3)
Sb1-O4 = 1.996(6)	Sb5-O10 = 2.004(7)	Te2-O17 = 2.008(8)	C13-Sb3-O5 = 170.7(3)
Sb1-O6 = 1.946(7)	Sb5-O7 = 1.977(6)	Te1-C31 = 2.128(12)	O4-Sb3-O12 = 169.8(3)
Sb2-O2 = 1.955(7)	Sb5-O9 = 1.993(7)	Te2-C43 = 2.131(13)	C19-Sb4-O7 = 171.6(3)
Sb2-O3 = 1.981(7)	Sb6-O8 = 2.504(7)	Te3-C55 = 2.112(11)	C25-Sb5-O7 = 169.0(3)
Sb2-O5 = 2.081(7)	Sb6-O12 = 1.906(7)	C4-Br1 = 1.895(11)	O1*-Sb5-O9 = 168.0(3)
Sb2-O10* = 2.006(7)	Sb6-O11 = 1.871(7)	Na1-O1 = 2.912(8)	O12-Sb6-O11 = 97.2(3)
Sb2-O13 = 2.039(7)	Sb6-O16 = 1.865(7)	Na1-O5 = 2.379(8)	O8-Sb6-O16 = 165.8(3)
Sb3-O3 = 1.978(7)	Sb7-O10* = 2.428(7)	Na1-O7 = 2.355(9)	O12-Sb6-O16 = 95.8(3)
Sb3-O5 = 2.088(6)	Sb7-O13 = 1.915(7)	Na1-O7* = 2.359(8)	O15-Sb7-O10* = 164.5(3)
Sb3-O4 = 1.969(7)	Sb7-O14 = 1.849(7)	Na1-O6* = 2.962(7)	Na1-O7-Na1* = 95.9(3)
Sb3-O12 = 2.044(7)	Sb7-O15 = 1.875(8)	Na1-O14 = 2.851(8)	Te1-O17-Te2 = 107.6(4)
Sb3-O8 = 1.984(7)	Sb1-C1 = 2.119(11)	Na1-O18 = 2.494(9)	Te2-O16-Sb6 = 117.8(3)
Sb4-O6 = 1.962(7)	Sb2-C7 = 2.109(11)	Na1-O11 = 2.779(8)	Te1-O15-Sb7 = 118.2(4)
Sb4-O8 = 2.017(7)	Sb3-C13 = 2.120(10)	C1-Sb1-O5 = 173.9(3)	Sb4*-O14-Sb7 = 131.8(4)
Sb4-O9 = 1.980(6)	Sb4-C19 = 2.102(10)	C1-Sb1-O1 = 94.0(3)	Sb7*-O10-Sb2* = 94.5(3)
Sb4-O7 = 1.996(7)	Sb5-C25 = 2.097(11)	O6-Sb1-O2 = 167.3(3)	Sb5-O11-Sb6 = 131.6(4)

Symmetry transformations used to generate equivalent atoms:*=-x+1,-y,-z+1

Table 3: Selected bond lengths(Å) and bond angles(º) in **2**

Sb1-O6 = 2.003(4)	Sb4-O5 = 1.871(4)	C1-Sb1-O8 = 164.6(2)	O5-Sb4-O6 = 88.28(17)
Sb1-O1 = 2.072(4)	Sb4-O6 = 1.943(4)	C7-Sb1-O1 = 93.0(2)	O6-Sb4-O5* = 164.37(15)
Sb1-O7 = 2.019(4)	Sb4-O4 = 1.906(4)	O7-Sb1-O8 = 78.89(16)	O4-Sb4-O6 = 97.49(16)
Sb1-O8 = 2.038(4)	Sb4-O5* = 2.361(4)	O1-Sb2-O2 = 95.1(2)	Sb1-O8-Sb3 = 99.81(17)
Sb2-O1 = 1.886(4)	Sb1-C1 = 2.143(6)	O1-Sb2-O3 = 102.79(18)	Sb1-O1-Sb2 = 116.9(2)
Sb2-O2 = 1.842(4)	Sb1-C7 = 2.150(6)	O2-Sb2-O3 = 94.83(19)	Sb3-O4-Sb4 = 120.26(19)
Sb2-O3 = 1.901(4)	Sb3-C13 = 2.149(6)	C13-Sb3-O8 = 167.18(19)	Sb4-O5-Sb4* = 130.69(17)
Sb3-O3 = 2.081(4)	Sb3-C19 = 2.141(6)	C19-Sb3-O7 = 169.3(2)	
Sb3-O4 = 2.059(4)	Te1-C25 = 2.134(6)	C13-Sb3-O4 = 94.89(19)	

Sb3-O7 = 1.998(4)	Te1-C43 = 2.124(6)	O3-Sb3-O7 = 91.83(17)	
Sb3-O8 = 2.025(4)	C1-Sb1-O7 = 167.9(2)	O4-Sb4-O5 = 96.66(17)	

Symmetry transformations used to generate equivalent atoms:*= -x,-y+1,-z+1