Synthesis and Structural Characterisation of the Aggregates of Benzo-1,2-Chalcogenazole 2-Oxides

Peter C. Ho,[a] Jamal Rafique,[b] Jiwon Lee,[a] Lucia M. Lee,[a] Hilary A. Jenkins,[a] James F. Britten,[a] Antonio L. Braga,[b]* Ignacio Vargas-Baca[a]*

Electronic Supplementary Information

a) *Department of Chemistry and Chemical Biology, McMaster University, 1280 Main Street West, Hamilton, Ontario, Canada L8S 4M1.*
b) *Department of Chemistry, Federal University of Santa Catarina, Florianópolis, Santa Catarina, Brazil 88040-900.*

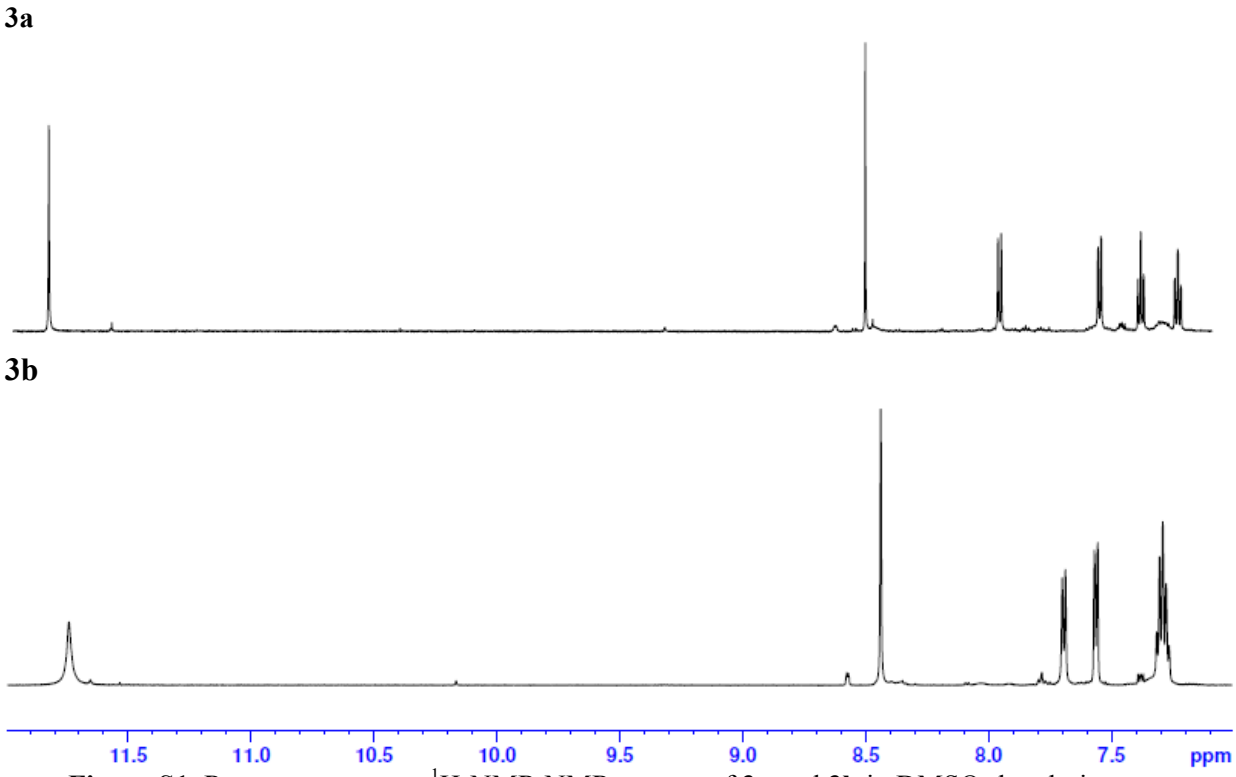


Figure S1. Room temperature ^{1}H-NMR NMR spectra of **3a** and **3b** in DMSO-d_6 solution.

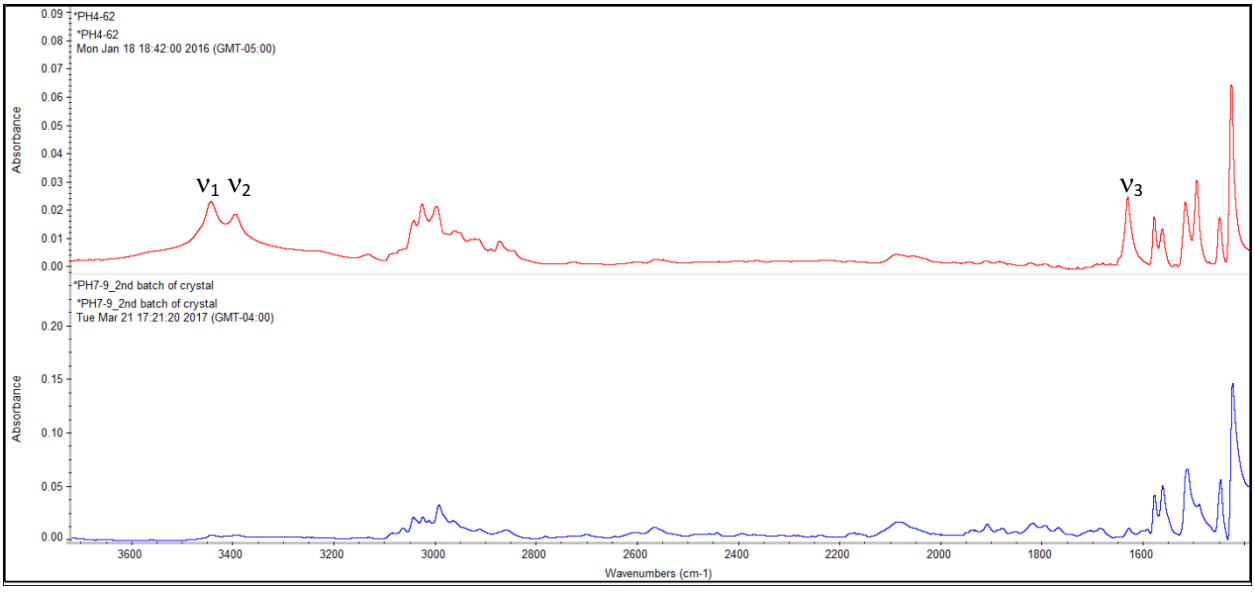
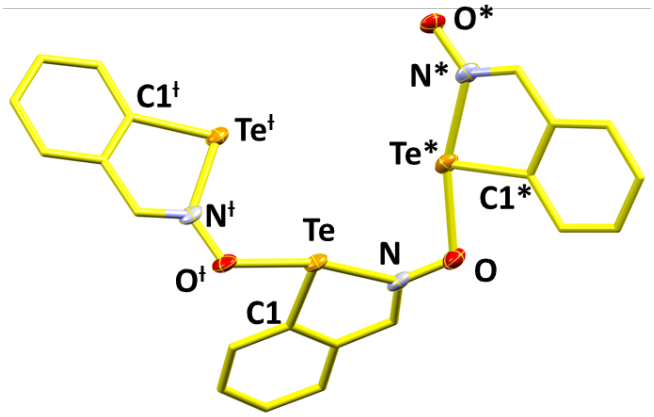


Figure S2. ATR- IR spectra of **2a**$_6$$(CH_2Cl_2)_{1.78}(H_2O)_{0.22}$ (top) and **2a** (bottom). In the former, the three normal vibrational modes of molecular water are visible: ν_1 asymmetric stretch, ν_2 symmetric stretch, and bending ν_3.

Table S1. Selected structural data for all compounds.



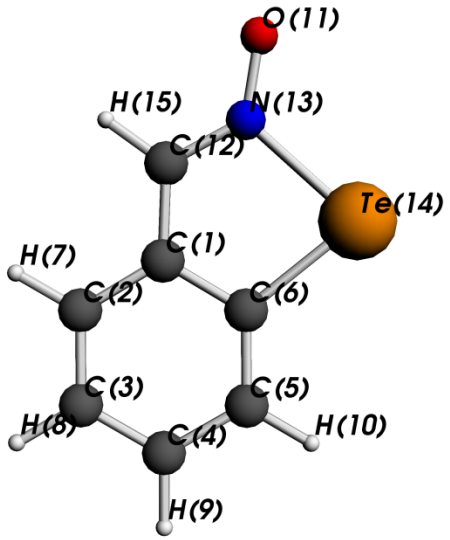
Labeling Scheme: * and † denote chemically equivalent atoms in neighboring molecules of **2** as shown.

Aggregate	**2a**$_6$	**2a**$_4$ (chair)	**2a**$_4$ (boat)	**2b**
		Bond Distances (Å)		
Te(Se)1-C1	2.100(6) 2.102(7) 2.106(7) 2.107(8) 2.111(6) 2.114(8)	2.095(3) 2.101(2)	2.102(6) 2.103(6)	1.896(5)
Te(Se)1-N1	2.261(5) 2.262(5) 2.262(5) 2.263(5) 2.267(4) 2.268(4)	2.197(2) 2.234(2)	2.244(5) 2.250(4)	1.938(5)
Te(Se)1···O1†	2.178(5) 2.186(5) 2.189(4) 2.189(6) 2.190(4) 2.201(5)	2.235(2) 2.245(2)	2.201(4) 2.213(4)	2.412(5)
N1-O1	1.363(6) 1.363(9) 1.366(8) 1.367(9) 1.368(9) 1.376(6)	1.357(3) 1.354(3)	1.361(7) 1.368(5)	1.315(6)

N1-O1†	4.405(7)	4.379(2)	4.398(6)	4.340(7)
	4.407(7)	4.439(3)	4.411(6)	
	4.408(8)			
	4.410(7)			
	4.411(6)			
	4.423(8)			
C1-C2	1.40(1)	1.409(3)	1.403(9)	1.406(8)
	1.40(1)	1.409(4)	1.409(7)	
	1.40(1)			
	1.403(9)			
	1.405(8)			
	1.41(1)			
C2-C3	1.44(1)	1.441(3)	1.430(9)	1.446(8)
	1.44(1)	1.446(4)	1.443(8)	
	1.44(1)			
	1.440(7)			
	1.45(1)			
	1.451(7)			
C3-N1	1.286(9)	1.286(3)	1.286(7)	1.308(7)
	1.289(8)	1.294(3)	1.287(7)	
	1.29(1)			
	1.293(7)			
	1.298(9)			
	1.30(1)			
Te(Se)1···Te(Se)1†	3.6377(7)	3.6989(2)	3.6846(8)	4.0795(9)
	3.6524(7)	4.3637(4)	3.7134(8)	
	3.6719(5)			
	3.6737(7)			
	3.7285(5)			
	3.7327(7)			
Bond Angles (º)				
N1-Te(Se)1-O1†	163.6(2)	162.37(7)	161.3(2)	172.3(2)
	163.9(2)	164.45(7)	164.6(1)	
	164.1(2)			
	164.6(2)			
	164.6(2)			
	164.7(2)			
N1-Te(Se)1-C1	75.7(2)	76.40(8)	75.8(2)	84.3(2)
	75.6(2)	76.61(8)	76.0(2)	
	75.7(2)			
	75.7(2)			
	75.7(2)			
	75.3(2)			

O1-N1-Te(Se)1	126.9(4) 127.0(4) 127.2(4) 126.6(4) 126.6(4) 127.1(4) 127.1(4)	122.7(1) 126.3(1)	125.4(3) 127.2(3)	118.8(4)
Torsion Angles (°)				
Te(Se)1-N1-O1-Te(Se)1*	11.2(6) 12.6(6) 2.2(6) 6.6(6) 15.0(6) 5.5(6)	19.0(2) 89.4(1)	1.4(5) 42.6(4)	86.3(4)
N1-O1-Te(Se)1*-C1*	83.7(4) 78.5(4) 85.2(4) 84.2(4) 75.9(4) 83.3(4)	116.6(1) 122.6(1)	105.0(3) 159.2(3)	129.2(4)
C1-Te(Se)1-N1-O1	177.2(5) 179.3(5) 180.0(5) 179.9(5) 178.6(5) 179.5(5)	178.6(2) 179.0(2)	176.1(4) 178.5(4)	178.2(4)
Interplanar Angles (°)				
	80.09 82.04 83.07 83.12 86.52 88.77	70.84	50.87 74.51	72.10

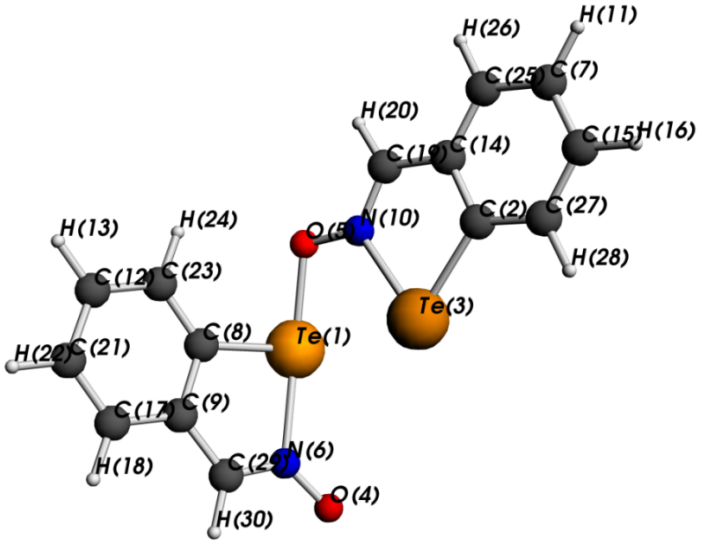
Table S2. DFT-Optimized Cartesian coordinates of **2a**, **2b** and their dimers.



2a

Total Bond Energy = -9314.82 kJ/mol, -3.55 hartree

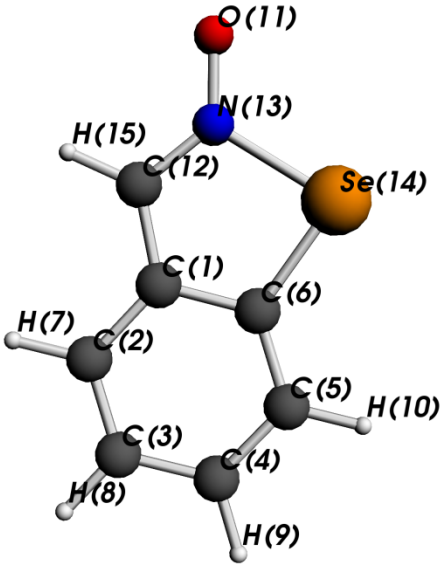
Atom	X	Y	Z (Angstrom)
1.C	-3.717693	-4.239105	0.000000
2.C	-4.927871	-4.963897	0.000000
3.C	-4.915857	-6.353082	0.000000
4.C	-3.699988	-7.052334	0.000000
5.C	-2.488146	-6.359998	0.000000
6.C	-2.497811	-4.964659	0.000000
7.H	-5.872448	-4.419102	0.000000
8.H	-5.857283	-6.901597	0.000000
9.H	-3.696025	-8.142001	0.000000
10.H	-1.546916	-6.909037	0.000000
11.O	-2.305413	-0.949017	0.000000
12.C	-3.668484	-2.812676	0.000000
13.N	-2.502871	-2.179840	0.000000
14.Te	-0.865454	-3.657946	0.000000
15.H	-4.552153	-2.177110	0.000000



$2a_2$

Total Bonding Energy = -18704.63 kJ/mol, -7.12 hartree

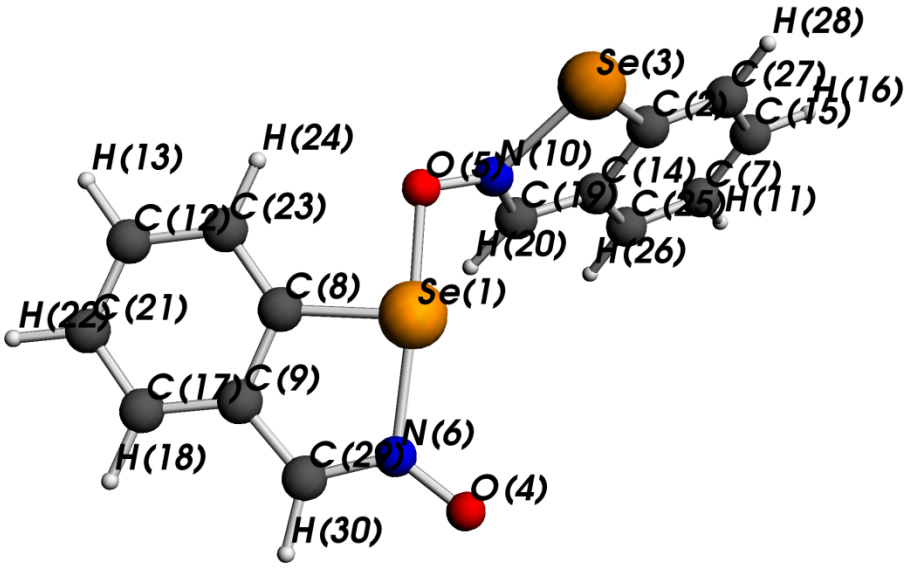
Atom	X	Y	Z (Angstrom)
1.Te	2.995963	3.400325	8.437837
2.C	4.383492	5.597551	4.132943
3.Te	3.836790	3.989314	5.415947
4.O	2.010968	0.521102	7.850344
5.O	3.780084	5.614603	8.066175
6.N	2.958047	1.162561	8.371368
7.C	5.063988	7.885571	2.670591
8.C	4.832221	2.956000	9.359365
9.C	5.028192	1.557234	9.477354
10.N	3.990722	5.730910	6.786840
11.H	5.327336	8.771144	2.092945
12.C	6.925835	3.393548	10.460881
13.H	7.662725	4.101032	10.840563
14.C	4.528008	6.840115	4.786482
15.C	4.919456	6.646873	2.026528
16.H	5.073292	6.574116	0.949658
17.C	6.203451	1.105532	10.108633
18.H	6.372540	0.032974	10.210598
19.C	4.311938	6.855932	6.203219
20.H	4.404223	7.748090	6.821513
21.C	7.137045	2.015685	10.595632
22.H	8.040200	1.650096	11.084123
23.C	5.766138	3.868789	9.840882
24.H	5.593652	4.938164	9.727421
25.C	4.870291	7.985962	4.042685
26.H	4.979194	8.945130	4.551127
27.C	4.578475	5.504856	2.752602
28.H	4.467369	4.549727	2.236910
29.C	4.038592	0.663571	8.946027
30.H	4.130851	-0.421839	8.980863



2b

Total Bond Energy = -9398.05 kJ/mol, -3.58 hartree

Atom	X	Y	Z (Angstrom)
1.C	-3.684885	-4.208221	0.000000
2.C	-4.872469	-4.964056	0.000000
3.C	-4.811105	-6.352797	0.000000
4.C	-3.572075	-7.012655	0.000000
5.C	-2.379970	-6.287624	0.000000
6.C	-2.442238	-4.893986	0.000000
7.H	-5.833990	-4.450360	0.000000
8.H	-5.732724	-6.933591	0.000000
9.H	-3.534956	-8.101720	0.000000
10.H	-1.420261	-6.803248	0.000000
11.O	-2.083335	-1.031538	0.000000
12.C	-3.604664	-2.786628	0.000000
13.N	-2.398083	-2.232742	0.000000
14.Se	-1.003445	-3.675095	0.000000
15.H	-4.449854	-2.103452	0.000000



$2b_2$

Total Bond Energy = -18827.30 kJ/mol, -7.17 hartree

Atom	X	Y	Z (Angstrom)
1.Se	3.357661	2.804381	8.465051
2.C	4.051302	6.086152	4.012839
3.Se	5.096985	4.518789	4.095027
4.O	0.754224	2.111902	9.404781
5.O	5.422081	3.798342	6.825674
6.N	1.942441	2.243790	9.762672
7.C	2.492576	8.369122	4.315591
8.C	4.553838	2.686695	9.925950
9.C	3.872677	2.281732	11.104669
10.N	4.902030	4.650726	6.062776
11.H	1.880286	9.263780	4.420914
12.C	6.632344	2.787605	11.124520
13.H	7.704749	2.981670	11.139236
14.C	3.676461	6.509950	5.314450
15.C	2.876661	7.935571	3.036331
16.H	2.561879	8.494962	2.155770
17.C	4.610077	2.137105	12.295242
18.H	4.098443	1.826795	13.206683
19.C	4.157222	5.699642	6.381902
20.H	3.968521	5.856838	7.441033
21.C	5.977281	2.388684	12.300075
22.H	6.543532	2.274397	13.224104
23.C	5.926045	2.939088	9.929560
24.H	6.430775	3.249766	9.016713
25.C	2.886063	7.667160	5.448076
26.H	2.588666	8.000887	6.441950
27.C	3.658555	6.792021	2.874950
28.H	3.951902	6.459976	1.879898
29.C	2.473311	2.056514	10.964599
30.H	1.798303	1.744579	11.757327