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Supporting Information

Crystallographic data

New diphenyl diselenides *o*-substituted by O(S, Se)-caranyl skeleton – synthesis and application in asymmetric reactions

by

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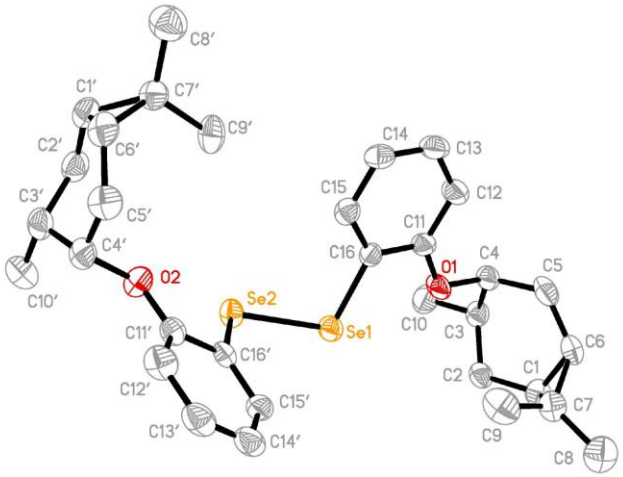
Content:

1. Crystallographic data for bis(2-((1*S*,3*R*,4*S*,6*R*)-3,7,7-Trimethyl-bicyclo[4.1.0]heptan-4-yloxy)phenyl)diselenide (19).

Crystals of diselenide **19** were grown from the methanol solution. The X-ray intensities were collected with an Oxford Sapphire CCD diffractometer using MoKα radiation λ =0.71073 Å, at 292(2) K, by ω-2θ method. Structure was solved by direct methods and refined with the full-matrix least-squares method on F^2 with the use of SHELX97 and SHELX2014 program packages [1]. Analytical absorption corrections were applied (RED171 package of programs [2] Oxford Diffraction, 2000), the maximum and minimum transmission of 0.2026 and 0.4609. Hydrogen atoms were located from the electron density maps and their positions were constrained in the refinement. The absolute structure was determined with the Flack method [3], the Flack x being -0.045(17). The structural data have been deposited with Cambridge Crystallographic Data Centre, the CCDC number 1453055.

X-ray structure of compounds **19**:



2. Table 1. Crystal data and structure refinement for **19**.

Identification code	**19**
Empirical formula	C32 H42 O2 Se2
Formula weight	616.57
Temperature; K	293(2)
Wavelength; Å	0.71073
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions; Å, °	a = 10.0882(8)
	b = 12.8630(10)
	c = 11.8710(10)
	= 102.335(6)
Volume; $Å^3$	1504.9(2)
Z	2
Density (calculated); Mg/m^3	1.361
Absorption coefficient; mm^{-1}	2.482
F(000)	636
Crystal size; mm	0.400 x 0.390 x 0.160
Theta range for data collection; °	2.883 to 31.168.
Index ranges	-14<=h<=14, -17<=k<=18, -14<=l<=16
Reflections collected	14937
Independent reflections	8535 [R(int) = 0.0740]
Completeness to theta = 25.242°	99.8 %
Absorption correction	Analytical
Max. and min. transmission	0.4609 and 0.2026
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8535 / 1 / 325
Goodness-of-fit on F^2	0.879
Final R indices [I>2sigma(I)]	R1 = 0.0587, wR2 = 0.1367
R indices (all data)	R1 = 0.0950, wR2 = 0.1535
Absolute structure parameter	-0.045(17)
Extinction coefficient	n/a
Largest diff. peak and hole; $e.Å^{-3}$	0.966 and -0.414

3. Table 2. Selected bond lengths [Å] and angles [°] for **19**.

C(1)-C(7)	1.498(13)
C(1)-C(2)	1.505(12)
C(1)-C(6)	1.515(12)
C(2)-C(3)	1.509(12)
C(3)-C(4)	1.511(12)
C(3)-C(10)	1.518(13)
C(4)-O(1)	1.453(9)
C(4)-C(5)	1.533(13)
C(5)-C(6)	1.493(12)
C(6)-C(7)	1.488(13)
C(7)-C(8)	1.519(15)
C(7)-C(9)	1.534(16)
O(1)-C(11)	1.362(9)
C(11)-C(12)	1.388(11)
C(11)-C(16)	1.389(10)
C(12)-C(13)	1.379(12)
C(13)-C(14)	1.384(12)
C(14)-C(15)	1.386(11)
C(15)-C(16)	1.376(10)
C(16)-Se(1)	1.936(7)
Se(1)-Se(2)	2.3198(10)
Se(2)-C(16')	1.930(7)
C(16')-C(15')	1.357(11)
C(16')-C(11')	1.417(11)
C(15')-C(14')	1.405(13)
C(14')-C(13')	1.355(14)
C(13')-C(12')	1.365(12)
C(12')-C(11')	1.394(12)
C(11')-O(2)	1.358(9)
O(2)-C(4')	1.432(9)
C(1')-C(2')	1.485(13)
C(1')-C(6')	1.522(13)
C(1')-C(7')	1.528(13)
C(2')-C(3')	1.536(12)

C(3')-C(10')	1.516(13)
C(3')-C(4')	1.519(13)
C(4')-C(5')	1.548(13)
C(5')-C(6')	1.484(13)
C(6')-C(7')	1.503(12)
C(7')-C(8')	1.486(14)
C(7')-C(9')	1.514(12)
C(7)-C(1)-C(2)	124.9(8)
C(7)-C(1)-C(6)	59.2(6)
C(2)-C(1)-C(6)	118.6(7)
C(1)-C(2)-C(3)	112.2(7)
C(2)-C(3)-C(4)	111.3(7)
C(2)-C(3)-C(10)	112.7(8)
C(4)-C(3)-C(10)	113.2(7)
O(1)-C(4)-C(3)	106.2(6)
O(1)-C(4)-C(5)	111.6(7)
C(3)-C(4)-C(5)	113.3(7)
C(6)-C(5)-C(4)	118.3(7)
C(7)-C(6)-C(5)	124.7(8)
C(7)-C(6)-C(1)	59.8(6)
C(5)-C(6)-C(1)	119.2(7)
C(6)-C(7)-C(1)	61.0(6)
C(6)-C(7)-C(8)	116.5(9)
C(1)-C(7)-C(8)	115.4(9)
C(6)-C(7)-C(9)	120.2(9)
C(1)-C(7)-C(9)	119.6(9)
C(8)-C(7)-C(9)	114.1(9)
C(11)-O(1)-C(4)	119.7(6)
O(1)-C(11)-C(12)	125.5(7)
O(1)-C(11)-C(16)	115.6(6)
C(12)-C(11)-C(16)	118.9(7)
C(13)-C(12)-C(11)	120.5(8)
C(12)-C(13)-C(14)	120.0(8)
C(13)-C(14)-C(15)	119.9(7)
C(16)-C(15)-C(14)	119.8(7)

C(15)-C(16)-C(11)	120.8(7)
C(15)-C(16)-Se(1)	123.8(5)
C(11)-C(16)-Se(1)	115.4(5)
C(16)-Se(1)-Se(2)	102.6(2)
C(16')-Se(2)-Se(1)	103.5(2)
C(15')-C(16')-C(11')	120.6(7)
C(15')-C(16')-Se(2)	124.3(6)
C(11')-C(16')-Se(2)	114.7(5)
C(16')-C(15')-C(14')	120.2(9)
C(13')-C(14')-C(15')	118.6(9)
C(14')-C(13')-C(12')	122.6(9)
C(13')-C(12')-C(11')	119.8(9)
O(2)-C(11')-C(12')	125.7(7)
O(2)-C(11')-C(16')	116.4(7)
C(12')-C(11')-C(16')	117.9(8)
C(11')-O(2)-C(4')	119.4(6)
C(2')-C(1')-C(6')	118.5(8)
C(2')-C(1')-C(7')	124.4(7)
C(6')-C(1')-C(7')	59.1(6)
C(1')-C(2')-C(3')	111.4(8)
C(10')-C(3')-C(4')	111.7(8)
C(10')-C(3')-C(2')	113.9(8)
C(4')-C(3')-C(2')	109.4(7)
O(2)-C(4')-C(3')	107.1(7)
O(2)-C(4')-C(5')	111.3(7)
C(3')-C(4')-C(5')	114.0(7)
C(6')-C(5')-C(4')	117.8(8)
C(5')-C(6')-C(7')	126.4(7)
C(5')-C(6')-C(1')	119.4(8)
C(7')-C(6')-C(1')	60.7(6)
C(8')-C(7')-C(6')	118.0(8)
C(8')-C(7')-C(9')	113.9(9)
C(6')-C(7')-C(9')	118.8(8)
C(8')-C(7')-C(1')	117.6(9)
C(6')-C(7')-C(1')	60.3(6)

4. Table 3. Torsion angles [°] for **19**.

C(7)-C(1)-C(2)-C(3)	101.4(11)
C(6)-C(1)-C(2)-C(3)	30.7(11)
C(1)-C(2)-C(3)-C(4)	-58.4(9)
C(1)-C(2)-C(3)-C(10)	173.1(8)
C(2)-C(3)-C(4)-O(1)	-68.1(8)
C(10)-C(3)-C(4)-O(1)	60.2(10)
C(2)-C(3)-C(4)-C(5)	54.8(9)
C(10)-C(3)-C(4)-C(5)	-177.0(8)
O(1)-C(4)-C(5)-C(6)	96.5(9)
C(3)-C(4)-C(5)-C(6)	-23.3(11)
C(4)-C(5)-C(6)-C(7)	-75.8(12)
C(4)-C(5)-C(6)-C(1)	-4.1(13)
C(2)-C(1)-C(6)-C(7)	115.7(9)
C(7)-C(1)-C(6)-C(5)	-115.4(9)
C(2)-C(1)-C(6)-C(5)	0.3(12)
C(5)-C(6)-C(7)-C(1)	106.5(10)
C(5)-C(6)-C(7)-C(8)	-147.8(10)
C(1)-C(6)-C(7)-C(8)	105.8(10)
C(5)-C(6)-C(7)-C(9)	-2.8(15)
C(1)-C(6)-C(7)-C(9)	-109.3(11)
C(2)-C(1)-C(7)-C(6)	-105.3(10)
C(2)-C(1)-C(7)-C(8)	147.1(9)
C(6)-C(1)-C(7)-C(8)	-107.6(10)
C(2)-C(1)-C(7)-C(9)	5.0(15)
C(6)-C(1)-C(7)-C(9)	110.3(10)
C(3)-C(4)-O(1)-C(11)	-157.1(7)
C(5)-C(4)-O(1)-C(11)	79.0(9)
C(4)-O(1)-C(11)-C(12)	-3.0(12)
C(4)-O(1)-C(11)-C(16)	176.6(7)
O(1)-C(11)-C(12)-C(13)	177.7(9)
C(16)-C(11)-C(12)-C(13)	-1.9(14)
C(11)-C(12)-C(13)-C(14)	1.4(15)
C(12)-C(13)-C(14)-C(15)	-0.8(14)
C(13)-C(14)-C(15)-C(16)	0.9(12)

C(14)-C(15)-C(16)-C(11)	-1.5(11)
C(14)-C(15)-C(16)-Se(1)	-178.7(6)
O(1)-C(11)-C(16)-C(15)	-177.7(7)
C(12)-C(11)-C(16)-C(15)	2.0(12)
O(1)-C(11)-C(16)-Se(1)	-0.2(9)
C(12)-C(11)-C(16)-Se(1)	179.4(7)
C(11')-C(16')-C(15')-C(14')	-4.2(11)
Se(2)-C(16')-C(15')-C(14')	-176.9(6)
C(16')-C(15')-C(14')-C(13')	3.0(13)
C(15')-C(14')-C(13')-C(12')	-2.8(14)
C(14')-C(13')-C(12')-C(11')	3.8(14)
C(13')-C(12')-C(11')-O(2)	175.0(8)
C(13')-C(12')-C(11')-C(16')	-4.8(12)
C(15')-C(16')-C(11')-O(2)	-174.8(6)
Se(2)-C(16')-C(11')-O(2)	-1.4(9)
C(15')-C(16')-C(11')-C(12')	5.0(11)
Se(2)-C(16')-C(11')-C(12')	178.4(6)
C(12')-C(11')-O(2)-C(4')	0.5(12)
C(16')-C(11')-O(2)-C(4')	-179.7(7)
C(6')-C(1')-C(2')-C(3')	36.1(10)
C(7')-C(1')-C(2')-C(3')	106.4(10)
C(1')-C(2')-C(3')-C(10')	172.6(8)
C(1')-C(2')-C(3')-C(4')	-61.6(9)
C(11')-O(2)-C(4')-C(3')	-156.9(7)
C(11')-O(2)-C(4')-C(5')	77.9(9)
C(10')-C(3')-C(4')-O(2)	57.5(9)
C(2')-C(3')-C(4')-O(2)	-69.6(9)
C(10')-C(3')-C(4')-C(5')	-179.0(8)
C(2')-C(3')-C(4')-C(5')	54.0(9)
O(2)-C(4')-C(5')-C(6')	100.2(9)
C(3')-C(4')-C(5')-C(6')	-21.0(11)
C(4')-C(5')-C(6')-C(7')	-79.1(12)
C(4')-C(5')-C(6')-C(1')	-5.5(12)
C(2')-C(1')-C(6')-C(5')	-2.6(12)
C(7')-C(1')-C(6')-C(5')	-117.7(9)

C(2')-C(1')-C(6')-C(7')	115.2(9)
C(5')-C(6')-C(7')-C(8')	-145.9(10)
C(1')-C(6')-C(7')-C(8')	107.5(10)
C(5')-C(6')-C(7')-C(9')	-1.2(14)
C(1')-C(6')-C(7')-C(9')	-107.7(9)
C(5')-C(6')-C(7')-C(1')	106.6(10)
C(2')-C(1')-C(7')-C(8')	146.6(9)
C(6')-C(1')-C(7')-C(8')	-108.2(9)
C(2')-C(1')-C(7')-C(6')	-105.3(10)
C(2')-C(1')-C(7')-C(9')	3.6(13)
C(6')-C(1')-C(7')-C(9')	108.9(9)

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