organic compounds

Bis(furan-2-ylcarbonyl) diselenide

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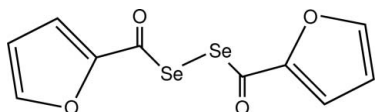Correspondence e-mail: amzs@st-and.ac.uk

The title molecule, $C_{10}H_6O_4Se_2$, lies on a twofold rotation axis. The Se—Se bond length of 2.305 (3) Å is similar to that in diphenyl diselenide [2.3066 (7) and 2.3073 (10) Å for the P and M isomers, respectively] and longer than that in 1,8-diselenonaphthalene [2.0879 (8) Å]. The molecule adopts a *gauche* conformation with respect to the C=O groups.

Related literature

For background information and the structure of diphenyl diselenide, see: Fuller *et al.* (2010). For the structure of 1,8-diselenonaphthalene, see: Aucott *et al.* (2004).

Experimental

Crystal data

$C_{10}H_6O_4Se_2$	V = 542.4 (9) Å^3
M_r = 348.07	Z = 2
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation
a = 9.615 (8) Å	μ = 6.81 mm^{-1}
b = 14.132 (14) Å	T = 125 K
c = 3.991 (4) Å	0.18 × 0.12 × 0.03 mm

Data collection

Rigaku Saturn70 diffractometer	1716 measured reflections
Absorption correction: multi-scan (*REQAB*; Rigaku, 1998)	895 independent reflections
	873 reflections with $F^2 > 2\sigma(F^2)$
T_{min} = 0.384, T_{max} = 0.815	R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050	$\Delta\rho_{max}$ = 1.63 e Å^{-3}
$wR(F^2)$ = 0.131	$\Delta\rho_{min}$ = −2.07 e Å^{-3}
S = 1.09	Absolute structure: Flack (1983), 322 Friedel pairs
895 reflections	Flack parameter: 0.03 (5)
73 parameters	
H-atom parameters constrained	

Data collection: *CrystalClear* (Rigaku, 2009); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5259).

References

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supporting information

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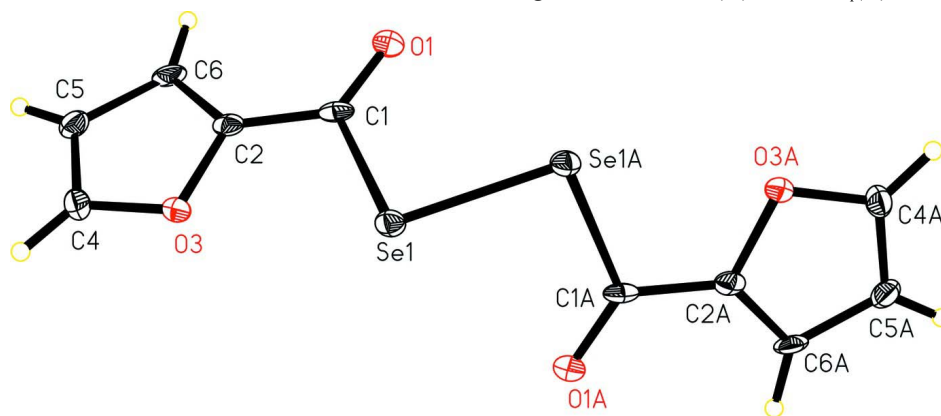
S1. Comment

We have recently reported (Fuller *et al.*, 2010) on the crystallization of Ph-Se-Se-Ph as one isomer. We were interested to see if this homocrystallization occurs for other diselenides. In the title compound, we observe a single isomer in the crystal rather than a mixture of *P* and *M* isomers. The Se—Se bond length of 2.305 (3)Å is similar to that in diphenyl-diselenide (2.3066 (7) and 2.3073 (10) Å for *P* and *M* isomers, respectively, Fuller *et al.*, 2010) and longer than that in 1,8-diselenonaphthalene (2.0879 (8)Å, Aucott *et al.*, 2004).

S2. Experimental

N-(furan-2-carbonyl)furan-2-carboxamide (0.205 g, 1.0 mmol) and Woollins reagent (0.54 g, 1.0 mmol) in 20 ml of dry toluene was refluxed for 10 h. Upon cooling to room temperature and removing toluene in vacuum the residue was purified by silica gel column (eluented by 1: 1 hexane / dichloromethane) to give 0.213 g of **1** as a pale yellow solid in 61% yield. Crystals for X-ray data collection were obtained by diffusion of hexane into a dichloromethane solution of (I). ^{1}H NMR (CD$_2$Cl$_2$, δ), 8.01 (d, *J*(H,H) = 8.2 Hz, 2H), 7.54 (d, *J*(H,H) = 8.2 Hz, 2H), 6.90–6.84 (m, 2H) p.p.m.. ^{13}C NMR (CD$_2$Cl$_2$, δ), 179.1 (C=O), 154.1, 148.1, 125.0, 117.1 p.p.m.. ^{77}Se NMR (CD$_2$Cl$_2$, δ), 624.5 p.p.m.. MS (CI$^+$, m/z), 351 [*M*+H]$^+$.

S3. Refinement

The partial completeness of *ca* 95% data may affect the precision of the structure. All H atoms were included in calculated positions with C—H = 0.95Å and were refined as riding atoms with U_{iso}(H) = 1.2 U_{eq}(C).

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level (symmetry code: (A) -x, -y+1, z).

Bis(furan-2-ylcarbonyl) diselenide

Crystal data

$C_{10}H_6O_4Se_2$	$F(000) = 332.00$
$M_r = 348.07$	$D_x = 2.131$ Mg m^{-3}
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Hall symbol: P 2 2ab	Cell parameters from 1723 reflections
$a = 9.615$ (8) Å	$\theta = 2.1$–$26.4°$
$b = 14.132$ (14) Å	$\mu = 6.81$ mm^{-1}
$c = 3.991$ (4) Å	$T = 125$ K
$V = 542.4$ (9) Å^3	Prism, colourless
$Z = 2$	$0.18 \times 0.12 \times 0.03$ mm

Data collection

Rigaku Saturn70 diffractometer	895 independent reflections
Detector resolution: 14.629 pixels mm^{-1}	873 reflections with $F^2 > 2\sigma(F^2)$
ω scans	$R_{int} = 0.057$
Absorption correction: multi-scan (REQAB; Rigaku, 1998)	$\theta_{max} = 25.0°$
$T_{min} = 0.384$, $T_{max} = 0.815$	$h = -9\rightarrow11$
1716 measured reflections	$k = -12\rightarrow16$
	$l = -4\rightarrow4$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 2.1662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	
895 reflections	$(\Delta/\sigma)_{max} < 0.001$
73 parameters	$\Delta\rho_{max} = 1.63$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -2.07$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 322 Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.03 (5)

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0 \sigma(F^2)$ is used only for calculating R-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Se(1)	−0.03886 (8)	0.42284 (5)	−0.0428 (2)	0.0223 (4)
O(1)	0.2214 (7)	0.4163 (5)	0.2824 (18)	0.0307 (15)
O(3)	−0.0133 (6)	0.2267 (4)	0.1408 (17)	0.0245 (14)
C(1)	0.1221 (8)	0.3698 (6)	0.202 (3)	0.0220 (19)
C(2)	0.1076 (9)	0.2689 (6)	0.257 (3)	0.0221 (18)
C(4)	−0.0020 (10)	0.1321 (6)	0.230 (3)	0.029 (3)
C(5)	0.1174 (9)	0.1177 (6)	0.407 (3)	0.030 (2)
C(6)	0.1870 (9)	0.2064 (6)	0.420 (3)	0.029 (2)

H(4)	−0.0677	0.0843	0.1754	0.0354*
H(5)	0.1481	0.0598	0.5026	0.0358*
H(6)	0.2737	0.2190	0.5251	0.0346*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se(1)	0.0186 (5)	0.0255 (5)	0.0228 (5)	−0.0008 (4)	−0.0037 (4)	−0.0008 (4)
O(1)	0.019 (3)	0.029 (3)	0.044 (4)	−0.003 (3)	−0.004 (3)	−0.003 (4)
O(3)	0.017 (3)	0.023 (3)	0.033 (4)	−0.002 (3)	0.000 (3)	0.001 (3)
C(1)	0.011 (4)	0.039 (5)	0.016 (5)	0.002 (4)	−0.002 (4)	−0.006 (4)
C(2)	0.016 (4)	0.031 (4)	0.019 (5)	0.001 (4)	0.006 (4)	−0.008 (4)
C(4)	0.030 (5)	0.021 (4)	0.038 (6)	−0.004 (4)	0.004 (4)	−0.002 (4)
C(5)	0.024 (4)	0.025 (4)	0.041 (6)	0.007 (4)	0.003 (5)	0.006 (5)
C(6)	0.011 (4)	0.039 (5)	0.037 (6)	0.007 (4)	−0.007 (4)	−0.004 (5)

Geometric parameters (Å, °)

Se(1)—Se(1)^i	2.305 (3)	C(2)—C(6)	1.337 (13)
Se(1)—C(1)	1.977 (9)	C(4)—C(5)	1.363 (14)
O(1)—C(1)	1.203 (11)	C(5)—C(6)	1.422 (12)
O(3)—C(2)	1.386 (11)	C(4)—H(4)	0.950
O(3)—C(4)	1.388 (10)	C(5)—H(5)	0.950
C(1)—C(2)	1.449 (12)	C(6)—H(6)	0.950
Se(1)^i—Se(1)—C(1)	96.0 (3)	C(4)—C(5)—C(6)	106.5 (8)
C(2)—O(3)—C(4)	105.3 (7)	C(2)—C(6)—C(5)	107.2 (8)
Se(1)—C(1)—O(1)	123.1 (7)	O(3)—C(4)—H(4)	125.009
Se(1)—C(1)—C(2)	111.9 (6)	C(5)—C(4)—H(4)	125.002
O(1)—C(1)—C(2)	125.0 (8)	C(4)—C(5)—H(5)	126.736
O(3)—C(2)—C(1)	117.0 (8)	C(6)—C(5)—H(5)	126.737
O(3)—C(2)—C(6)	110.9 (8)	C(2)—C(6)—H(6)	126.404
C(1)—C(2)—C(6)	132.0 (9)	C(5)—C(6)—H(6)	126.395
O(3)—C(4)—C(5)	110.0 (8)		
Se(1)^i—Se(1)—C(1)—O(1)	−3.6 (7)	Se(1)—C(1)—C(2)—C(6)	−176.7 (7)
Se(1)^i—Se(1)—C(1)—C(2)	178.9 (5)	O(1)—C(1)—C(2)—O(3)	−178.8 (8)
C(1)—Se(1)—Se(1)^i—C(1)^i	−120.5 (3)	O(1)—C(1)—C(2)—C(6)	5.7 (16)
C(2)—O(3)—C(4)—C(5)	2.9 (10)	O(3)—C(2)—C(6)—C(5)	1.3 (11)
C(4)—O(3)—C(2)—C(1)	−178.9 (7)	C(1)—C(2)—C(6)—C(5)	176.9 (9)
C(4)—O(3)—C(2)—C(6)	−2.5 (9)	O(3)—C(4)—C(5)—C(6)	−2.1 (11)
Se(1)—C(1)—C(2)—O(3)	−1.3 (10)	C(4)—C(5)—C(6)—C(2)	0.5 (11)

Symmetry code: (i) −x, −y+1, z.