Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Bis(furan-2-ylcarbonyl) diselenide

**David B. Cordes, Guoxiong Hua, Alexandra M. Z. Slawin* and J. Derek Woollins**

School of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST, Scotland
Correspondence e-mail: amzs@st-and.ac.uk

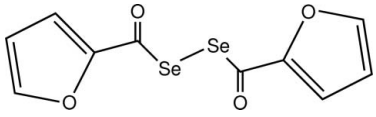
Received 24 May 2011; accepted 30 May 2011

Key indicators: single-crystal X-ray study; $T$ = 125 K; mean $\sigma$(C–C) = 0.013 Å; $R$ factor = 0.050; $wR$ factor = 0.131; data-to-parameter ratio = 12.3.

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

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

### Data collection

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

### Refinement

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

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

Aucott, S. M., Milton, H. L., Robertson, S. D., Slawin, A. M. Z. & Woollins, J. D. (2004). *Heteroat. Chem.* **15**, 530–542.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fuller, A. L., Scott-Hayward, L. A. S., Li, Y., Bühl, M., Slawin, A. M. Z. & Woollins, J. D. (2010). *J. Am. Chem. Soc.* **132**, 5799–5802.
Rigaku (1998). *REQAB*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2009). *CrystalClear*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Rigaku (2010). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supplementary materials

*Acta Cryst.* (2011). E**67**, o1586 [ **doi:10.1107/S160053681102085X** ]

# Bis(furan-2-ylcarbonyl) diselenide

**D. B. Cordes, G. Hua, A. M. Z. Slawin and J. D. Woollins**

## 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 diphenyldiselenide (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).

## 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 ($CD_2Cl_2$, 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 ($CD_2Cl_2$, δ), 179.1 (C=O), 154.1, 148.1, 125.0, 117.1 p.p.m.. $^{77}$Se NMR ($CD_2Cl_2$, δ), 624.5 p.p.m.. MS ($CI^+$, m/*z*), 351 $[M+H]^+$.

## 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).

## Figures

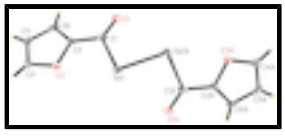


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level (symmetry code: (A) -x, -y+1, z).

## 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, λ = 0.71075 Å |
| Hall symbol: P 2 2ab | Cell parameters from 1723 reflections |
| $a$ = 9.615 (8) Å | θ = 2.1–26.4° |

| | |
|---|---|
| $b$ = 14.132 (14) Å | μ = 6.81 mm$^{-1}$ |
| $c$ = 3.991 (4) Å | $T$ = 125 K |
| $V$ = 542.4 (9) Å$^3$ | Prism, colourless |
| $Z$ = 2 | 0.18 × 0.12 × 0.03 mm |

*Data collection*

| | |
|---|---|
| Rigaku Saturn70 diffractometer | 873 reflections with $F^2 > 2\sigma(F^2)$ |
| Detector resolution: 14.629 pixels mm$^{-1}$ | $R_{int}$ = 0.057 |
| ω scans | $\theta_{max}$ = 25.0° |
| Absorption correction: multi-scan (REQAB; Rigaku, 1998) | $h$ = −9→11 |
| $T_{min}$ = 0.384, $T_{max}$ = 0.815 | $k$ = −12→16 |
| 1716 measured reflections | $l$ = −4→4 |
| 895 independent reflections | |

*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 | $(\Delta/\sigma)_{max} < 0.001$ |
| 895 reflections | $\Delta\rho_{max}$ = 1.63 e Å$^{-3}$ |
| 73 parameters | $\Delta\rho_{min}$ = −2.07 e Å$^{-3}$ |
| 0 restraints | Absolute structure: Flack (1983), 322 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: 0.03 (5) |
| Secondary atom site location: difference Fourier map | |

*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 codes: (i) −*x*, −*y*+1, *z*.

**Fig. 1**