Pyramidalization of a carbonyl C atom in (2S)-N-([selenoacetyl]proline methyl ester

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Key indicators: single-crystal X-ray study; T = 105 K; mean σ(C–C) = 0.019 Å; r factor = 0.051; wR factor = 0.135; data-to-parameter ratio = 13.4.

The title compound, C₈H₁₄NO₂Se, crystallizes as a non-merohedral twin with an approximate 9:1 component ratio with two symmetry-independent molecules in the asymmetric unit. Our density-functional theory (DFT) computations indicate that the carboxy C atom is expected to be slightly pyramidal due to an n → π* interaction, wherein the lone pair (n) of the Se atom overlap with the antibonding orbital (π*) of the carbonyl group. Such pyramidalization is observed in one molecule of the title compound but not the other.

Related literature

For background to hybrid density functional theory (DFT) and natural bond orbital (NBO) analysis, see: Glendingen et al. (2001); Weinhold (1998); Weinhold & Landis (2005). For literature related to the synthesis, see: Bhattacharyya & Woolfins (2001) and for NBO studies of the title compound, see: Choudhary & Raines (2011a); DeRider et al. (2002); Choudhary et al. (2009, 2010a,b); Jakobsche et al. (2010); Bartlett et al. (2010); Choudhary & Raines (2011b). For geometrical checks with ConQuest and Mercury, see: Bruno et al. (2002). For Gaussian 03 software, see: Frisch (2004). For puckering parameters, see: Cremer & Pople (1975).

Data collection

Bruker SMART APEX2 area detector diffractometer

Absorption correction: multi-scan

Bruker, SADABS

Tmin = 0.268, Tmax = 0.349

Refinement

R[F² > 2σ(F²)] = 0.051

wR(F²) = 0.135

S = 1.12

3012 reflections

224 parameters

3 restraints

H-atom parameters constrained

Data collection: APEX2 (Bruker, 2012); cell refinement: SAINT-Plus (Bruker, 2007); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: OLEX2 (Dolomanov et al., 2009) and NBOView (Wendt & Weinhold, 2001); software used to prepare material for publication: OLEX2, GX and FCF_filter (Guzei, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2221).

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Comment

We have previously reported on extensive studies of geometrical and conformational attributes of several amide bond isosteres (Choudhary & Raines, 2011a). In contrast, selenoamides have not received much attention. Herein we report the crystal structure of the title compound N-selenoacetyl–(2S)-proline methyl ester (Scheme 1, (I)) and the results of a hybrid density functional theory (DFT) and Natural Bond Orbital (NBO) analysis (Glendening et al., 2001, Weinhold, 1998, Weinhold & Landis, 2005) of its geometrical features.

Compound (I), Figure 1, crystallizes as a non-merohedral twin with the minor component contribution of 10 (2)%%. The two components are related by 179° degree rotation about the [1T0] vector. The asymmetric unit in the relatively rare space group P1 contains two symmetry-independent molecules with the same handedness. The absolute structures of both components have been unequivocally established by anomalous dispersion effects: the Flack x parameters for the two components have been refined independently to 0.04 (2) and 0.02 (2). The two molecules have essentially identical geometries and their non-H atoms can be superimposed with a RMS of 0.042 Å. All geometrical parameters in the molecules are typical within experimental error (Bruno et al., 2002). The conformations of the five-membered rings in (I) are characterized by the puckering coordinates (Cremer & Pople, 1975) q2 and φ2 which measured 0.376 (14) Å and 89.8 (19)° for the Se1 molecule and 0.370 (15) Å and 85 (2)° for the Se1a molecule. Whereas the extent of puckering of the rings is the same, the ring in the Se1 molecule is in twisted conformation 3T4 whereas the ring in the other molecule is in the 3T4 conformation with a small 3E envelope character. The envelope character is probably statistically significant.

The key feature of (I) is pyramidalization of atom C7 described with parameters ∆ and Θ defined in Figure 2. These parameters are 0.016 (12) Å and 0.06 (5)° for the Se1 molecule and 0.040 (13) Å and 1.5 (5)° for the Se1a molecule. In the Se1 molecule the pyramidalization is not observed whereas in the second molecule the slight pyramidalization is statistically significant. For comparison, in the sulfur analog of (I) the relevant pyramidalization parameters ∆ and Θ are 0.0293 (13) Å and 1.10 (5)°, also small and statistically significant.

We conducted DFT and NBO analyses of four low energy conformations of (I) (DeRider et al., 2002, Choudhary et al., 2009, Choudhary et al., 2010b, Choudhary et al., 2010a, Jakobsche et al., 2010) at the B3LYP/6-311+G(2 d,p) level of theory using Gaussian 03 (Frisch et al., 2004) and comment here on the most stable conformer. We have previously reported an interaction in proteins, termed the n→π* interaction, wherein the lone pairs (n) of an oxygen (O, ) of a carbonyl group overlap with the antibonding orbital (π*) of C=O of an adjacent carbonyl group. The similar overlap in (I) between the lone pairs (n) of the selenium and the antibonding orbital (π*) of the carbonyl group is shown in Figure 3. (Bartlett et al., 2010, Choudhary & Raines, 2011b). This interaction resembles the Bürgi–Dunitz trajectory for nucleophilic additions to the carbonyl group and induces pyramidalization of the acceptor carbonyl group (Choudhary et al., 2009). The second-order perturbation theory as implemented in NBO 5.0 suggests n→π* interaction mediated
stabilization of the trans conformation by 0.84 kcal/mol. The findings of our crystallographic studies partially support our theoretical findings: molecule Se1A shows pyramidalization whereas molecule Se1 does not. We attribute these results to the twinned nature of the crystals that lead to relatively high e.s.d.'s on geometrical parameters, but it was not possible to isolate a better crystal.

Experimental

Compound (I) was synthesized following from its oxygen congener by using (PhPSe)₂ (Woolins’ reagent) following a procedure reported previously (Bhattacharyya & Woollins, 2001). A small amount of (I) was dissolved in hexanes with a minimal amount of ethyl acetate. Slow evaporation of the solution afforded X-ray quality crystals of (I) after ~4 days.

Refinement

All H-atoms were placed in idealized locations and refined as riding with appropriate thermal displacement coefficients $U_{iso}(H) = 1.2$ or $1.5$ times $U_{eq}$(bearing atom).

Computing details

Data collection: APEX2 (Bruker, 2012); cell refinement: SAINT-Plus (Bruker, 2007); data reduction: SAINT-Plus (Bruker, 2007); program(s) used to solve structure: SHELTXL (Sheldrick, 2008); program(s) used to refine structure: SHELTXL (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov et al., 2009) and NBOView (Wendt & Weinhold, 2001); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009), GX and FCF_filter (Guzei, 2012).

Figure 1

Molecular structure of (I). The thermal ellipsoids are shown at 50% probability level.
**Figure 2**
Pyramidalization parameters $\Delta$ and $\Theta$ of an $n\rightarrow\pi^*$ interaction in (I).

**Figure 3**
An NBO depiction of the $n\rightarrow\pi^*$ orbital overlap in (I) generated with NBOView (Wendt & Weinhold, 2001)

**Methyl (2S)-1-ethaneselenoylpyrrolidine-2-carboxylate**

*Crystal data*

- $C_8H_{13}NO_2Se$
- $M_r = 234.15$
- Triclinic, $P1$
- $a = 7.050 (3)$ Å
- $b = 7.442 (3)$ Å
- $c = 10.334 (4)$ Å
- $\alpha = 85.166 (6)^\circ$
- $\beta = 86.220 (6)^\circ$
Cell parameters from 746 reflections

$\gamma = 64.682 (4)^\circ$
$V = 488.1 (3) \text{ Å}^3$
$Z = 2$
$F(000) = 236$
$D_x = 1.593 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ Å}$

$\theta = 3.0$–$29.0^\circ$
$\mu = 3.81 \text{ mm}^{-1}$
$T = 105 \text{ K}$

Block, colourless
$0.47 \times 0.37 \times 0.35 \text{ mm}$

Data collection

Bruker SMART APEX2 area detector
diffractometer
Radiation source: microfocus sealed X-ray tube,
Incoatec I\text{$\mu$s}
Mirror optics monochromator
Detector resolution: 7.9 pixels mm$^{-1}$

$\theta_{\text{max}} = 25.1^\circ$, $\theta_{\text{min}} = 2.0^\circ$

$0.5^\circ \omega$ and $0.5^\circ \phi$ scans

Absorption correction: multi-scan
(TWINABS; Bruker, 2007)

$T_{\text{min}} = 0.268$, $T_{\text{max}} = 0.349$
3012 measured reflections
3012 independent reflections
2938 reflections with $I > 2\sigma(I)$

Detector resolution: 7.9 pixels mm$^{-1}$

$\Delta \rho_{\text{max}} = 1.46 \text{ e } \text{Å}^{-3}$
$\Delta \rho_{\text{min}} = -0.63 \text{ e } \text{Å}^{-3}$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R(F^2) = 0.051$
$wR(F^2) = 0.135$

$S = 1.12$
3012 reflections
224 parameters
3 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_c^2) + (0.0947P)^2 + 1.2373P]$
where $P = (F_c^2 + 2F_{o}^2)/3$

$\Delta /\sigma$ max < 0.001

$\Delta \rho_{\text{max}} = 1.46 \text{ e } \text{Å}^{-3}$
$\Delta \rho_{\text{min}} = -0.63 \text{ e } \text{Å}^{-3}$

Absolute structure: Classical Flack method
preferred over Parsons because s.u. lower.

Flack parameter: 0.01 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)

|         | $x$     | $y$       | $z$       | $U_{iso}$/$U_{eq}$ |
|---------|---------|-----------|-----------|---------------------|
| Se1     | 1.02636 | 0.03198 (10) | 0.80590 (9) | 0.0268 (3) |
| O1      | 0.6582 (13) | 0.5319 (12) | 0.9097 (8) | 0.0258 (17) |
| O2      | 0.6687 (15) | 0.5834 (13) | 0.6919 (8) | 0.0267 (19) |
| N1      | 0.6036 (14) | 0.1857 (14) | 0.8793 (9) | 0.0209 (18) |
| C1      | 0.807 (2) | $-0.108$ (2) | 1.0113 (14) | 0.024 (3) |
| H1A     | 0.7617 | $-0.0430$ | 1.0937 | 0.036* |
| H1B     | 0.9523 | $-0.2093$ | 1.0174 | 0.036* |
| H1C     | 0.7156 | $-0.1708$ | 0.9932 | 0.036* |
| C2      | 0.7948 (17) | 0.0453 (16) | 0.9034 (12) | 0.023 (2) |
| C3      | 0.4089 (18) | 0.2096 (19) | 0.9573 (12) | 0.024 (3) |
| H3A     | 0.4309 | 0.2039 | 1.0515 | 0.029* |
| H3B     | 0.3636 | 0.1052 | 0.9401 | 0.029* |
| C4      | 0.2498 (18) | 0.4141 (18) | 0.9102 (13) | 0.027 (3) |

*Estimated standard deviation in last digit

Geometric details

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refined as a 4-component twin.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{Å}^2$)
### Atomic displacement parameters (Å$^2$)

|     | $U^{11}$       | $U^{22}$       | $U^{33}$       | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|----------------|----------------|----------------|--------------|--------------|--------------|
| Se1 | 0.0198 (6)     | 0.0286 (7)     | 0.0363 (8)     | −0.0150 (6)  | 0.0045 (5)   | −0.0029 (5)  |
| O1  | 0.028 (4)      | 0.027 (4)      | 0.026 (5)      | −0.016 (4)   | 0.000 (3)    | −0.004 (3)   |
| O2  | 0.030 (5)      | 0.030 (5)      | 0.029 (5)      | −0.021 (4)   | −0.007 (4)   | 0.000 (3)    |
| N1  | 0.020 (5)      | 0.021 (5)      | 0.026 (5)      | −0.014 (4)   | 0.002 (3)    | −0.001 (3)   |
| C1  | 0.019 (6)      | 0.023 (7)      | 0.027 (7)      | −0.007 (6)   | −0.004 (5)   | −0.001 (5)   |
| C2  | 0.016 (5)      | 0.019 (5)      | 0.037 (7)      | −0.008 (5)   | 0.000 (4)    | −0.006 (4)   |
| C3  | 0.020 (6)      | 0.022 (6)      | 0.035 (6)      | −0.012 (5)   | 0.001 (5)    | −0.004 (5)   |
| C4  | 0.015 (5)      | 0.021 (6)      | 0.047 (7)      | −0.011 (5)   | 0.001 (5)    | −0.004 (5)   |
| C5  | 0.021 (6)      | 0.020 (6)      | 0.045 (7)      | −0.009 (5)   | −0.010 (5)   | 0.002 (5)    |

* indicates 3σ deviation for U$_{ij}$.
supplementary materials

|    |     |
|----|-----|
| C6 | 0.021 (5) |
| C7 | 0.018 (5) |
| C8 | 0.039 (8) |
| Se1A | 0.0285 (7) |
| O1A | 0.019 (4) |
| O2A | 0.019 (4) |
| N1A | 0.018 (5) |
| C1A | 0.018 (6) |
| C2A | 0.015 (5) |
| C3A | 0.022 (6) |
| C4A | 0.026 (7) |
| C5A | 0.022 (6) |
| C6A | 0.017 (5) |
| C7A | 0.020 (6) |
| C8A | 0.027 (7) |
| C88 | 0.022 (5) |
| C89 | 0.029 (6) |
| C810 | 0.024 (5) |
| C811 | 0.015 (5) |
| C812 | 0.006 (4) |
| C813 | 0.002 (4) |

Geometric parameters (Å, °)

|    |     |
|----|-----|
| Se1—C2 | 1.831 (11) |
| O1—C7 | 1.194 (14) |
| O2—C7 | 1.337 (14) |
| O2—C8 | 1.450 (16) |
| N1—C2 | 1.329 (15) |
| N1—C3 | 1.495 (14) |
| N1—C6 | 1.465 (14) |
| C1—H1A | 0.9800 |
| C1—H1B | 0.9800 |
| C1—H1C | 0.9800 |
| C1—C2 | 1.504 (19) |
| C3—H3A | 0.9900 |
| C3—H3B | 0.9900 |
| C3—C4 | 1.516 (18) |
| C4—H4A | 0.9900 |
| C4—H4B | 0.9900 |
| C4—C5 | 1.514 (19) |
| C5—H5A | 0.9900 |
| C5—H5B | 0.9900 |
| C5—C6 | 1.555 (16) |
| C6—H6 | 1.0000 |
| C6—C7 | 1.529 (15) |
| C8—H8A | 0.9800 |
| C8—H8B | 0.9800 |
| C8—H8C | 0.9800 |
| C7—O2—C8 | 114.7 (10) |
| C2—N1—C3 | 124.9 (10) |
| C2—N1—C6 | 123.3 (9) |
| C6—N1—C3 | 111.8 (9) |
| H1A—C1—H1B | 109.5 |
| H1A—C1—H1C | 109.5 |
| H1A—C1—H1B | 109.5 |
| H1A—C1—H1C | 109.5 |

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| Bond          | Angle (°) | Bond          | Angle (°) |
|--------------|-----------|--------------|-----------|
| H1B—C1—H1C  | 109.5     | H1AB—C1A—H1AC| 109.5     |
| C2—C1—H1A   | 109.5     | C2A—C1A—H1AA | 109.5     |
| C2—C1—H1B   | 109.5     | C2A—C1A—H1AB | 109.5     |
| C2—C1—H1C   | 109.5     | C2A—C1A—H1AC | 109.5     |
| N1—C2—Se1    | 122.0 (9) | N1A—C2A—Se1A | 122.3 (9) |
| N1—C2—C1     | 115.8 (10)| N1A—C2A—C1A  | 117.2 (11)|
| C1—C2—Se1    | 122.2 (8) | C1A—C2A—Se1A | 120.4 (9) |
| N1—C3—H3A    | 111.1     | N1A—C3A—H3AA | 111.1     |
| N1—C3—H3B    | 111.1     | N1A—C3A—H3AB | 111.1     |
| N1—C3—C4     | 103.2 (10)| N1A—C3A—C4A  | 103.2 (9) |
| H3A—C3—H3B   | 109.1     | H3AA—C3A—H3AB| 109.1     |
| C4—C3—H3A    | 111.1     | C4A—C3A—H3AA | 111.1     |
| C4—C3—H3B    | 111.1     | C4A—C3A—H3AB | 111.1     |
| C3—C4—H4A    | 111.0     | C3A—C4A—H4AA | 111.0     |
| C3—C4—H4B    | 111.0     | C3A—C4A—H4AB | 111.0     |
| H4A—C4—H4B   | 109.0     | H4AA—C4A—H4AB| 109.0     |
| C5—C4—C3     | 104.0 (10)| C5A—C4A—C3A  | 103.9 (11)|
| C5—C4—H4A    | 111.0     | C5A—C4A—H4AA | 111.0     |
| C5—C4—H4B    | 111.0     | C5A—C4A—H4AB | 111.0     |
| C4—C5—H5A    | 111.1     | C4A—C5A—H5AA | 111.0     |
| C4—C5—H5B    | 111.1     | C4A—C5A—H5AB | 111.0     |
| C4—C5—C6     | 103.3 (10)| C4A—C5A—C6A  | 103.8 (9) |
| C5—C5—H5A    | 109.1     | H5AA—C5A—H5AB| 109.0     |
| C6—C5—H5A    | 111.1     | C6A—C5A—H5AA | 111.0     |
| C6—C5—H5B    | 111.1     | C6A—C5A—H5AB | 111.0     |
| N1—C6—C5     | 102.9 (9) | N1A—C6A—C5A  | 103.7 (9) |
| N1—C6—H6     | 111.1     | N1A—C6A—H6A  | 110.9     |
| N1—C6—C7     | 110.4 (8) | N1A—C6A—C7A  | 110.0 (9) |
| C5—C6—H6     | 111.1     | C5A—C6A—H6A  | 110.9     |
| C7—C6—C5     | 110.0 (9) | C5A—C6A—C7A  | 110.1 (9) |
| C7—C6—H6     | 111.1     | C7A—C6A—H6A  | 110.9     |
| O1—C7—O2     | 125.6 (10)| O1A—C7A—O2A  | 125.9 (11)|
| O1—C7—C6     | 125.5 (10)| O1A—C7A—C6A  | 126.3 (10)|
| O2—C7—C6     | 108.8 (9) | O2A—C7A—C6A  | 107.6 (9) |
| O2—C8—H8A    | 109.5     | O2A—C8A—H8AA | 109.5     |
| O2—C8—H8B    | 109.5     | O2A—C8A—H8AB | 109.5     |
| O2—C8—H8C    | 109.5     | O2A—C8A—H8AC | 109.5     |
| H8A—C8—H8B   | 109.5     | H8AA—C8A—H8AB| 109.5     |
| H8A—C8—H8C   | 109.5     | H8AA—C8A—H8AC| 109.5     |
| H8B—C8—H8C   | 109.5     | H8AB—C8A—H8AC| 109.5     |

N1—C3—C4—C5         31.5 (11)  N1A—C3A—C4A—C5A         32.7 (12)
N1—C6—C7—O1         −25.3 (15)  N1A—C6A—C7A—O1A         −30.5 (16)
N1—C6—C7—O2         157.1 (9)   N1A—C6A—C7A—O2A         155.3 (9)
C2—N1—C6—C5         167.0 (10)  C2A—N1A—C6A—C5A         169.8 (10)
C2—N1—C6—C7         169.2 (10)  C2A—N1A—C6A—C7A         166.9 (9)
C2—N1—C6—C7         −73.5 (12)  C2A—N1A—C6A—C7A         −75.3 (12)
C3—N1—C2—Se1        −177.9 (8)  C3A—N1A—C2A—Se1A        179.8 (8)
C3—N1—C2—C1         5.1 (16)    C3A—N1A—C2A—C1A         1.6 (15)
| Bond                     | Angle (deg) | Bond                     | Angle (deg) |
|-------------------------|-------------|-------------------------|-------------|
| C3—N1—C6—C5            | -11.6 (11)  | C3A—N1A—C6A—C5A        | -8.4 (11)   |
| C3—N1—C6—C7            | 105.7 (10)  | C3A—N1A—C6A—C7A        | 109.4 (10)  |
| C3—C4—C5—C6            | -39.0 (11)  | C3A—C4A—C5A—C6A        | -38.3 (12)  |
| C4—C5—C6—N1            | 30.9 (11)   | C4A—C5A—C6A—N1A        | 28.6 (11)   |
| C4—C5—C6—C7            | -86.8 (11)  | C4A—C5A—C6A—C7A        | -89.0 (11)  |
| C5—C6—C7—O1            | 87.6 (14)   | C5A—C6A—C7A—O1A        | 83.2 (15)   |
| C5—C6—C7—O2            | -90.1 (11)  | C5A—C6A—C7A—O2A        | -91.0 (11)  |
| C6—N1—C2—Se1           | 1.2 (14)    | C6A—N1A—C2A—Se1A       | 5.2 (14)    |
| C6—N1—C2—C1            | -175.8 (11) | C6A—N1A—C2A—C1A        | -173.0 (11) |
| C6—N1—C3—C4            | -12.1 (12)  | C6A—N1A—C3A—C4A        | -15.0 (12)  |
| C8—O2—C7—O1            | 0.0 (18)    | C8A—O2A—C7A—O1A        | 3.5 (18)    |
| C8—O2—C7—C6            | 177.7 (10)  | C8A—O2A—C7A—C6A        | 177.7 (11)  |