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4-(5-Chlorothiophen-2-yl)-1,2,3-selenadiazole

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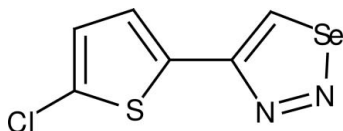
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.039; wR factor = 0.121; data-to-parameter ratio = 19.8.

In the title compound, $\text{C}_6\text{H}_3\text{ClN}_2\text{SSe}$, the selenadiazole and chlorothiophene rings are almost coplanar [dihedral angle = 5.24 (15°)]. In the crystal, $\text{C}-\text{H}\cdots\text{N}$ interactions link the molecules into chains extending along the b -axis direction. $\text{C}-\text{H}\cdots\pi$ interactions also occur.

Related literature

For the biological activity of selenadiazole derivatives, see: El-Bahaie *et al.* (1990); El-Kashef *et al.* (1986); Padmavathi *et al.* (2002); Plano *et al.* (2010); Stadtman (1991); Velusamy *et al.* (2005). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_6\text{H}_3\text{ClN}_2\text{SSe}$
 $M_r = 249.57$
 Monoclinic, $P2_1/c$
 $a = 6.0412$ (3) Å
 $b = 19.5870$ (11) Å

$c = 7.2010$ (4) Å
 $\beta = 110.257$ (3)°
 $V = 799.38$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 5.22$ mm⁻¹
 $T = 293$ K

$0.22 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART APEX CCD
 detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2008)
 $T_{\min} = 0.330$, $T_{\max} = 0.391$

7064 measured reflections
 1978 independent reflections
 1558 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.121$
 $S = 1.01$
 1978 reflections

100 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.44$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.52$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C1}-\text{H1}\cdots\text{N1}^i$ | 0.93 | 2.62 | 3.545 (5) | 171 |

Symmetry code: (i) $x + 1, y, z$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank the TBI Consultancy, University of Madras, India, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6854).

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supplementary materials

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4-(5-Chlorothiophen-2-yl)-1,2,3-selenadiazole

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Comment

Selenium containing compounds, like 1,2,3-selenadiazoles are of increasing interest because of their chemical properties and biological applications such as anti-bacterial (El-Kashef *et al.*, 1986), anti-microbial (El-Bahaie *et al.*, 1990), anti-cancer (Plano *et al.*, 2010) and insecticidal (Padmavathi *et al.*, 2002) activities. It has been found that the introduction of 1,2,3-selenadiazole ring system increase the biological activity. Selenium atom has a much larger size and less electronegativity than the sulfur atom, selenium containing compounds influence the electro-optical properties of the small molecules (Velusamy *et al.*, 2005).

Glutathione peroxidases (GPx) are the antioxidant selenoenzymes protecting various organisms from oxidative stress by catalyzing the reduction of hydroperoxides at the expense of glutathione (GSH) (Stadtman, 1991). In view of the growing importance of selenium containing compounds, the crystal structure of the title compound has been carried out.

The *ORTEP* plot of the molecule is shown in Fig. 1. The bond lengths [Se1—N1] 1.870 (3) Å and [Se1—C1] 1.825 (4) Å are comparable with the values reported in the literature (Allen *et al.*, 1987). The selenadiazole ring is planar [with the maximum deviation for atom C2 of 0.004 (3)°]. Both the selenadiazole ring and chlorothiophene ring are lie in a common plane, the corresponding torsion angle is [C4—C3—C2—N2] 175.5 (3)°.

The packing of the molecules is shown in Fig. 2. The crystal packing is stabilized by C—H···N intermolecular interactions, linking the molecules to chains extending along the *b* axis.

Experimental

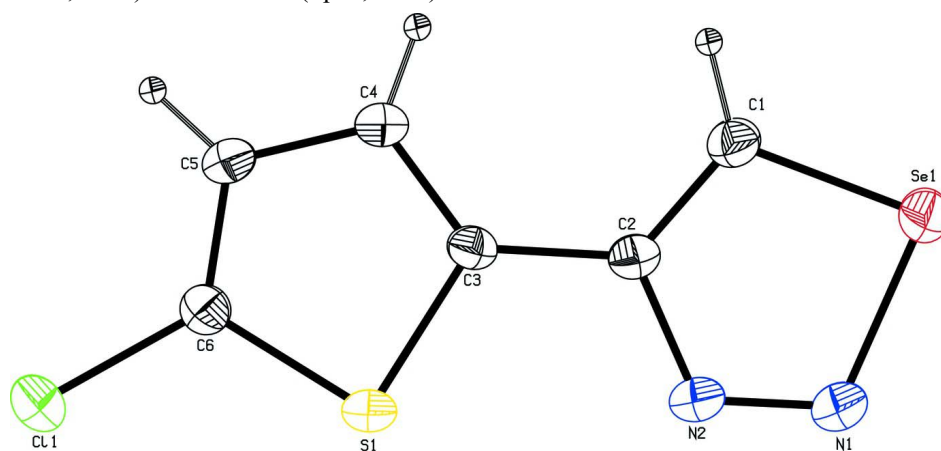
A mixture of 2-acetyl-5-chlorothiophene (1 mmol), semicarbazide hydrochloride (2 mmol) and sodium acetate (3 mmol) in ethanol (10 ml) was refluxed for 4 hrs. After completion of the reaction as monitored by TLC, the mixture was poured into ice cold water and the resulting semicarbazone was filtered off. Then, a mixture of semicarbazone (1 mmol) and SeO₂ (2 mmol) in tetrahydrofuran (10 ml) were refluxed on a water bath for 1 h. The selenium deposited on cooling was removed by filtration, and the filtrate was poured into crushed ice, extracted with dichloromethane, and purified by column chromatography using silica gel (60–120 mesh) with 97:3 petroleum ether: ethyl acetate as eluent to give 4-(5-chloro-2,5-dihydrothiophen-2-yl)-1,2,3-selenadiazole.

Refinement

H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

**Figure 1**

The molecular structure of the title compound, showing the atomic numbering and displacement ellipsoids drawn at 30% probability level.

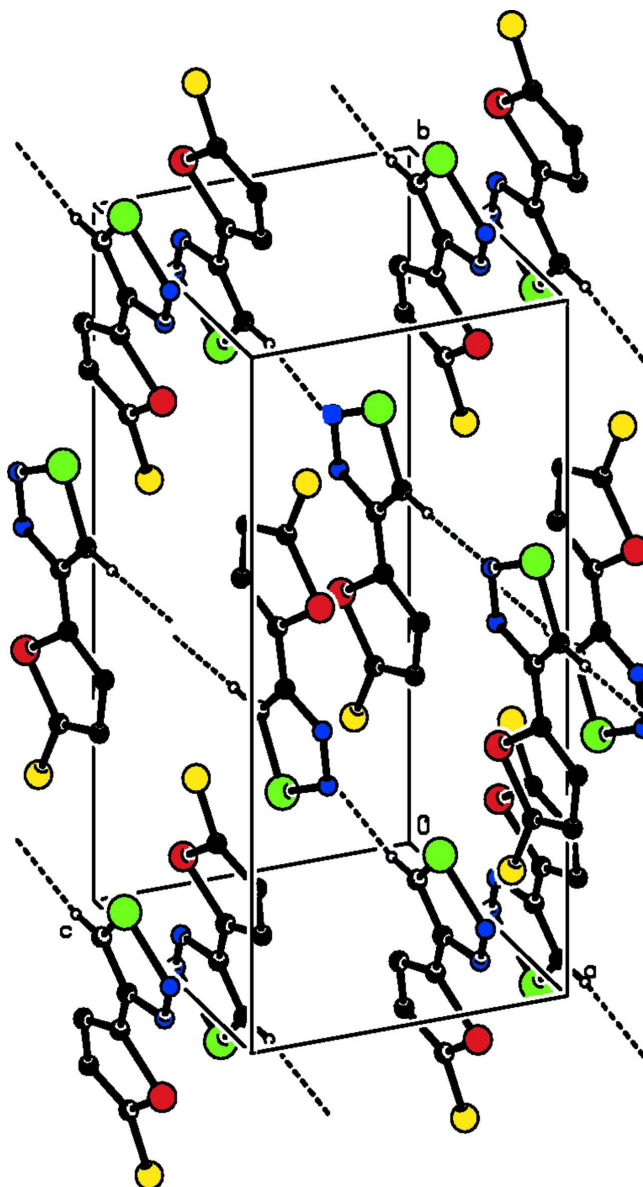


Figure 2

The crystal packing.

4-(5-Chlorothiophen-2-yl)-1,2,3-selenadiazole

Crystal data

$C_6H_3ClN_2SSe$

$M_r = 249.57$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 6.0412\ (3)\ \text{\AA}$

$b = 19.5870\ (11)\ \text{\AA}$

$c = 7.2010\ (4)\ \text{\AA}$

$\beta = 110.257\ (3)^\circ$

$V = 799.38\ (7)\ \text{\AA}^3$

$Z = 4$

$F(000) = 480$

$D_x = 2.074\ \text{Mg m}^{-3}$

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

Cell parameters from 1558 reflections

$\theta = 2.1\text{--}28.3^\circ$

$\mu = 5.22\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Black, white crystalline

$0.22 \times 0.20 \times 0.18\ \text{mm}$

Data collection

| | |
|--|--|
| Bruker SMART APEX CCD detector diffractometer | 7064 measured reflections 1978 independent reflections |
| Radiation source: fine-focus sealed tube | 1558 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{\text{int}} = 0.044$ |
| ω scans | $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.1^\circ$ |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2008) | $h = -8 \rightarrow 6$ $k = -25 \rightarrow 26$ $l = -9 \rightarrow 9$ |
| $T_{\text{min}} = 0.330$, $T_{\text{max}} = 0.391$ | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H-atom parameters constrained |
| $wR(F^2) = 0.121$ | $w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.3075P]$ |
| $S = 1.01$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1978 reflections | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| 100 parameters | $\Delta\rho_{\text{max}} = 0.44 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.52 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

Special details

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|--------------|----------------------------------|
| C1 | 0.3771 (6) | 0.60797 (19) | 0.2132 (5) | 0.0397 (7) |
| H1 | 0.5288 | 0.6078 | 0.2081 | 0.048* |
| C2 | 0.2578 (6) | 0.55180 (18) | 0.2343 (4) | 0.0333 (7) |
| C3 | 0.3344 (6) | 0.48114 (17) | 0.2494 (5) | 0.0328 (7) |
| C4 | 0.5358 (6) | 0.45391 (19) | 0.2353 (5) | 0.0412 (8) |
| H4 | 0.6532 | 0.4799 | 0.2134 | 0.049* |
| C5 | 0.5478 (6) | 0.38256 (19) | 0.2573 (5) | 0.0413 (8) |
| H5 | 0.6726 | 0.3562 | 0.2502 | 0.050* |
| C6 | 0.3579 (6) | 0.35688 (18) | 0.2896 (5) | 0.0376 (7) |
| Cl1 | 0.29911 (17) | 0.27326 (5) | 0.32523 (15) | 0.0529 (3) |
| N1 | -0.0347 (5) | 0.62617 (17) | 0.2239 (5) | 0.0482 (8) |
| N2 | 0.0334 (5) | 0.56419 (16) | 0.2406 (4) | 0.0428 (7) |
| S1 | 0.15880 (15) | 0.41890 (5) | 0.29606 (14) | 0.0408 (2) |
| Se1 | 0.19762 (7) | 0.684213 (19) | 0.19625 (6) | 0.04757 (18) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0304 (16) | 0.0409 (19) | 0.0469 (18) | 0.0004 (14) | 0.0120 (14) | -0.0018 (15) |
| C2 | 0.0271 (15) | 0.0379 (18) | 0.0349 (15) | -0.0008 (12) | 0.0107 (12) | -0.0014 (13) |
| C3 | 0.0286 (15) | 0.0333 (16) | 0.0368 (15) | -0.0044 (13) | 0.0120 (13) | -0.0002 (13) |
| C4 | 0.0325 (17) | 0.0404 (19) | 0.056 (2) | -0.0015 (14) | 0.0216 (15) | 0.0038 (16) |
| C5 | 0.0336 (17) | 0.042 (2) | 0.055 (2) | 0.0011 (14) | 0.0232 (16) | -0.0024 (16) |
| C6 | 0.0353 (17) | 0.0351 (18) | 0.0427 (17) | -0.0030 (14) | 0.0138 (14) | -0.0027 (14) |
| C11 | 0.0501 (6) | 0.0375 (5) | 0.0727 (6) | -0.0066 (4) | 0.0234 (5) | 0.0031 (4) |
| N1 | 0.0335 (15) | 0.0461 (19) | 0.068 (2) | 0.0029 (13) | 0.0221 (15) | 0.0005 (15) |
| N2 | 0.0308 (14) | 0.0443 (18) | 0.0577 (18) | -0.0008 (13) | 0.0208 (13) | -0.0007 (14) |
| S1 | 0.0289 (4) | 0.0409 (5) | 0.0559 (5) | -0.0041 (3) | 0.0190 (4) | 0.0004 (4) |
| Se1 | 0.0375 (2) | 0.0371 (3) | 0.0659 (3) | 0.00047 (14) | 0.01514 (19) | 0.00089 (16) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------------|------------|--------------|------------|
| C1—C2 | 1.352 (5) | C4—H4 | 0.9300 |
| C1—Se1 | 1.825 (4) | C5—C6 | 1.345 (5) |
| C1—H1 | 0.9300 | C5—H5 | 0.9300 |
| C2—N2 | 1.394 (4) | C6—C11 | 1.714 (4) |
| C2—C3 | 1.451 (5) | C6—S1 | 1.721 (4) |
| C3—C4 | 1.364 (5) | N1—N2 | 1.274 (4) |
| C3—S1 | 1.724 (3) | N1—Se1 | 1.870 (3) |
| C4—C5 | 1.406 (5) | | |
| C2—C1—Se1 | 110.2 (3) | C5—C4—H4 | 123.4 |
| C2—C1—H1 | 124.9 | C6—C5—C4 | 112.2 (3) |
| Se1—C1—H1 | 124.9 | C6—C5—H5 | 123.9 |
| C1—C2—N2 | 115.1 (3) | C4—C5—H5 | 123.9 |
| C1—C2—C3 | 128.0 (3) | C5—C6—C11 | 128.1 (3) |
| N2—C2—C3 | 116.9 (3) | C5—C6—S1 | 112.7 (3) |
| C4—C3—C2 | 129.5 (3) | C11—C6—S1 | 119.2 (2) |
| C4—C3—S1 | 111.3 (3) | N2—N1—Se1 | 111.1 (2) |
| C2—C3—S1 | 119.2 (2) | N1—N2—C2 | 116.7 (3) |
| C3—C4—C5 | 113.1 (3) | C3—S1—C6 | 90.63 (17) |
| C3—C4—H4 | 123.4 | C1—Se1—N1 | 86.89 (15) |
| Se1—C1—C2—N2 | -0.7 (4) | C4—C5—C6—S1 | -0.4 (4) |
| Se1—C1—C2—C3 | 178.8 (3) | Se1—N1—N2—C2 | -0.3 (4) |
| C1—C2—C3—C4 | -4.0 (6) | C1—C2—N2—N1 | 0.7 (5) |
| N2—C2—C3—C4 | 175.4 (3) | C3—C2—N2—N1 | -178.8 (3) |
| C1—C2—C3—S1 | 174.7 (3) | C4—C3—S1—C6 | -1.4 (3) |
| N2—C2—C3—S1 | -5.8 (4) | C2—C3—S1—C6 | 179.7 (3) |
| C2—C3—C4—C5 | -179.7 (3) | C5—C6—S1—C3 | 1.0 (3) |
| S1—C3—C4—C5 | 1.4 (4) | C11—C6—S1—C3 | -179.5 (2) |
| C3—C4—C5—C6 | -0.7 (5) | C2—C1—Se1—N1 | 0.4 (3) |
| C4—C5—C6—C11 | -179.8 (3) | N2—N1—Se1—C1 | -0.1 (3) |

Hydrogen-bond geometry (Å, °)

| <i>D—H···A</i> | <i>D—H</i> | <i>H···A</i> | <i>D···A</i> | <i>D—H···A</i> |
|-------------------------|------------|--------------|--------------|----------------|
| C1—H1···N1 ⁱ | 0.93 | 2.62 | 3.545 (5) | 171 |

Symmetry code: (i) $x+1, y, z$.