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5-Chloro-4-chloromethyl-2-phenyl-1,3-selenazole

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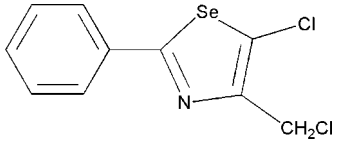
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 19.8.

The title compound, $C_{10}H_7Cl_2NSe$, crystallizes with two crystallographically independent molecules in the asymmetric unit. All atoms in each molecule are nearly coplanar, except for H_2Cl of the chloromethyl group. The molecular structure presents two weak intramolecular C—H···Se and C—H···Cl hydrogen bonds.

Related literature

For related literature, see: Koketsu & Ishihara, 2003; Geisler *et al.*, 2004.



Experimental

Crystal data

$C_{10}H_7Cl_2NSe$
M_r = 291.03
Triclinic, $P\overline{1}$
a = 8.1640 (5) Å
b = 10.4050 (6) Å
c = 13.7225 (7) Å
α = 76.264 (3)°
β = 74.357 (3)°
γ = 86.475 (3)°
V = 1090.39 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.89 mm^{-1}
T = 296 (2) K
0.57 × 0.30 × 0.22 mm

Data collection

Bruker APEX2 area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.256, T_{max} = 0.423
17299 measured reflections
5021 independent reflections
3550 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
S = 1.02
5021 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···Cl2	0.97	2.78	3.213 (5)	108
C6—H6A···Se1	0.93	2.79	3.219 (4)	109
C11—H11B···Cl3	0.97	2.80	3.222 (4)	107
C16—H16A···Se2	0.93	2.76	3.200 (3)	110

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2004); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2124).

References

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supplementary materials

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5-Chloro-4-chloromethyl-2-phenyl-1,3-selenazole

X. Shi and G.-L. Zhao

Comment

Selenium-containing molecules are of considerable biochemical and pharmacological relevance. A prominent example is the antitumor and antiviral active *C*-glycosyl selenazole selenazofurin (Koketsu & Ishihara, 2003; Geisler *et al.*, 2004). In order to prepare compounds with higher anticancer activity, we planed to synthesize some selenazole derivatives containing amino acids. By reacting 2-phenyl-1,3-selenazole methanol with glycine in sulfuryl dichloride, we unexpectedly obtained the title compound, (I), and obtained single crystals suitable for X-ray diffraction.

The asymmetric unit of (I) contains two independent molecules, quoted 1 and 2 for Se1 and Se2 containing molecules, respectively (Fig. 1). The dihedral angle between phenyl and selenazole rings are 7.25 (9) and 7.47 (9)° for molecules 1 and 2, respectively. The Se—C distances, 1.852 (4), 1.883 (3) Å as well as the bond angle C—Se—C, 83.79 (14) ° in molecule 1, are slightly different than corresponding bond lengths [1.851 (3) and 1.878 (3) Å] and angle [83.25 (13)°] in molecule 2. The N═C and N—C distances in molecule 1 are 1.282 (4) and 1.386 (4) Å; the corresponding distances in molecule 2 are 1.291 (3) and 1.372 (4) Å.

In the asymmetric unit, the dihedral angle between two neighboring phenyl rings is 65.79 (11)°, and angle between two neighboring selenazole rings is 51.12 (9)°. Furthermore, there are weak C—H···Se and C—H···Cl intramolecular hydrogen bonds, involving C6 and Se1, C16 and Se2, C1 and Cl2, C11 and Cl3, forming five-membered rings. *Via* hydrogen bond interactions, all the skeletal atoms in each independent molecule, except for chloromethyl Cl atoms, are approximately coplanar, with maximum deviations of −0.112 Å for C20 and 0.102 Å for C7.

Experimental

Glycine (5 mmol) was refluxed in sulfuryl dichloride (10 ml) for 6 h., and then 2-phenyl-1,3-selenazole methanol was added to the solution. The mixture was refluxed for 4 h., sulfuryl dichloride was evaporated and the residue was washed with water. The residue was dissolved in ether, giving a yellow solution, which afforded yellow single crystals after 7 days.

Refinement

All H atom were positioned geometrically and refined using a riding model. Constrained C—H bond lengths: 0.93 (aromatic CH) or 0.97 Å (methylene CH_2). isotropic displacement parameters for H atoms: $U_{iso}(H) = 1.2U_{eq}$(carrier C).

Figures

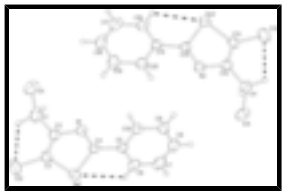


Fig. 1. A view of the molecule of (I) showing the atom-labeling scheme, with displacement ellipsoids drawn at the 30% probability level. dashed lines represent intramolecular hydrogen bonds.

5-Chloro-4-chloromethyl-2-phenyl-1,3-selenazole

Crystal data

$C_{10}H_7Cl_2NSe$	$Z = 4$
$M_r = 291.03$	$F_{000} = 568$
Triclinic, $P\overline{1}$	$D_x = 1.773$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.1640$ (5) Å	Cell parameters from 5903 reflections
$b = 10.4050$ (6) Å	$\theta = 1.6$–27.7º
$c = 13.7225$ (7) Å	$\mu = 3.89$ mm^{-1}
$\alpha = 76.264$ (3)º	$T = 296$ (2) K
$\beta = 74.357$ (3)º	Block, yellow
$\gamma = 86.475$ (3)º	0.57 × 0.30 × 0.22 mm
$V = 1090.39$ (11) Å^3	

Data collection

Bruker APEX2 area-detector diffractometer	5021 independent reflections
Radiation source: fine-focus sealed tube	3550 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
$T = 296$(2) K	$\theta_{max} = 27.7$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.256$, $T_{max} = 0.423$	$k = -13 \rightarrow 13$
17299 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 0.4631P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
5021 reflections	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
253 parameters	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	1.37993 (5)	1.00206 (4)	−0.11871 (3)	0.06946 (13)
Se2	0.72129 (4)	0.96216 (3)	0.65950 (2)	0.06129 (12)
Cl1	1.2736 (2)	1.44955 (11)	−0.02164 (11)	0.1177 (5)
Cl2	1.43418 (18)	1.27694 (13)	−0.27490 (8)	0.1086 (4)
Cl3	0.56928 (17)	0.69066 (11)	0.79275 (8)	0.1012 (4)
Cl4	0.70828 (12)	0.54767 (9)	0.52221 (9)	0.0791 (3)
N1	1.1682 (3)	1.1290 (2)	0.01349 (19)	0.0552 (6)
N2	0.6747 (3)	0.8781 (2)	0.49813 (19)	0.0500 (6)
C1	1.1660 (6)	1.3615 (3)	−0.0819 (3)	0.0866 (12)
H1A	1.0453	1.3592	−0.0477	0.104*
H1B	1.1811	1.4075	−0.1540	0.104*
C2	1.2296 (4)	1.2242 (3)	−0.0772 (3)	0.0601 (8)
C3	1.3401 (4)	1.1821 (3)	−0.1550 (3)	0.0650 (8)
C4	1.2275 (3)	1.0130 (3)	0.0093 (2)	0.0488 (6)
C5	1.1829 (4)	0.8961 (3)	0.0952 (2)	0.0498 (7)
C6	1.2630 (5)	0.7759 (3)	0.0919 (3)	0.0771 (10)
H6A	1.3466	0.7661	0.0329	0.093*
C7	1.2180 (6)	0.6693 (4)	0.1774 (4)	0.0937 (13)
H7A	1.2740	0.5889	0.1760	0.112*
C8	1.0924 (6)	0.6819 (4)	0.2632 (3)	0.0832 (11)
H8A	1.0617	0.6098	0.3194	0.100*
C9	1.0126 (5)	0.7990 (4)	0.2667 (3)	0.0712 (9)
H9A	0.9269	0.8070	0.3254	0.085*
C10	1.0572 (4)	0.9069 (3)	0.1840 (2)	0.0577 (7)
H10A	1.0027	0.9874	0.1878	0.069*
C11	0.5462 (4)	0.6569 (3)	0.5694 (3)	0.0686 (9)
H11A	0.4769	0.6834	0.5207	0.082*
H11B	0.4733	0.6107	0.6353	0.082*
C12	0.6184 (3)	0.7770 (3)	0.5839 (3)	0.0543 (7)
C13	0.6302 (4)	0.7979 (3)	0.6745 (3)	0.0599 (8)
C14	0.7312 (3)	0.9791 (3)	0.5186 (2)	0.0455 (6)
C15	0.7991 (3)	1.0989 (3)	0.4403 (2)	0.0464 (6)
C16	0.8770 (4)	1.1978 (3)	0.4642 (3)	0.0589 (8)
H16A	0.8828	1.1904	0.5320	0.071*
C17	0.9459 (4)	1.3072 (3)	0.3878 (3)	0.0716 (10)
H17A	0.9982	1.3731	0.4043	0.086*
C18	0.9374 (5)	1.3189 (3)	0.2881 (3)	0.0776 (11)
H18A	0.9855	1.3922	0.2367	0.093*
C19	0.8585 (5)	1.2234 (4)	0.2632 (3)	0.0740 (10)

H19A	0.8515	1.2328	0.1954	0.089*
C20	0.7894 (4)	1.1132 (3)	0.3388 (2)	0.0581 (8)
H20A	0.7362	1.0484	0.3217	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0758 (2)	0.0742 (2)	0.0559 (2)	−0.00109 (17)	−0.00420 (16)	−0.02466 (17)
Se2	0.0703 (2)	0.0623 (2)	0.0538 (2)	0.00479 (15)	−0.01144 (15)	−0.02474 (15)
Cl1	0.1783 (13)	0.0675 (7)	0.1169 (10)	−0.0122 (7)	−0.0441 (9)	−0.0301 (6)
Cl2	0.1377 (10)	0.1113 (9)	0.0589 (6)	−0.0308 (8)	−0.0132 (6)	0.0084 (6)
Cl3	0.1291 (9)	0.0759 (6)	0.0670 (6)	0.0129 (6)	0.0058 (6)	0.0052 (5)
Cl4	0.0805 (6)	0.0569 (5)	0.1084 (8)	0.0072 (4)	−0.0265 (5)	−0.0352 (5)
N1	0.0638 (15)	0.0485 (14)	0.0533 (15)	0.0038 (12)	−0.0148 (12)	−0.0136 (12)
N2	0.0474 (12)	0.0482 (14)	0.0561 (15)	0.0012 (10)	−0.0112 (11)	−0.0185 (12)
C1	0.117 (3)	0.055 (2)	0.088 (3)	0.005 (2)	−0.040 (2)	−0.0034 (19)
C2	0.073 (2)	0.0517 (18)	0.059 (2)	−0.0047 (15)	−0.0263 (16)	−0.0078 (15)
C3	0.076 (2)	0.070 (2)	0.0488 (18)	−0.0167 (17)	−0.0182 (16)	−0.0062 (16)
C4	0.0501 (15)	0.0526 (17)	0.0481 (16)	−0.0009 (13)	−0.0135 (12)	−0.0193 (13)
C5	0.0538 (16)	0.0471 (16)	0.0545 (17)	−0.0025 (12)	−0.0197 (13)	−0.0160 (13)
C6	0.089 (2)	0.052 (2)	0.085 (3)	0.0110 (18)	−0.013 (2)	−0.0213 (19)
C7	0.122 (4)	0.045 (2)	0.116 (4)	0.012 (2)	−0.042 (3)	−0.014 (2)
C8	0.113 (3)	0.058 (2)	0.078 (3)	−0.017 (2)	−0.035 (2)	0.000 (2)
C9	0.083 (2)	0.073 (2)	0.056 (2)	−0.0168 (19)	−0.0186 (17)	−0.0083 (17)
C10	0.0669 (18)	0.0546 (18)	0.0531 (18)	−0.0008 (14)	−0.0164 (15)	−0.0142 (15)
C11	0.0552 (17)	0.0511 (18)	0.096 (3)	−0.0055 (14)	−0.0069 (17)	−0.0230 (18)
C12	0.0448 (15)	0.0468 (17)	0.067 (2)	0.0047 (12)	−0.0049 (13)	−0.0179 (15)
C13	0.0588 (18)	0.0527 (18)	0.0571 (19)	0.0058 (14)	−0.0010 (14)	−0.0089 (15)
C14	0.0391 (13)	0.0483 (16)	0.0517 (16)	0.0072 (11)	−0.0091 (11)	−0.0217 (13)
C15	0.0411 (13)	0.0422 (15)	0.0564 (17)	0.0070 (11)	−0.0096 (12)	−0.0177 (13)
C16	0.0584 (17)	0.0489 (17)	0.073 (2)	0.0038 (14)	−0.0169 (15)	−0.0215 (16)
C17	0.065 (2)	0.0465 (19)	0.103 (3)	−0.0019 (15)	−0.020 (2)	−0.0188 (19)
C18	0.071 (2)	0.0480 (19)	0.097 (3)	0.0032 (16)	−0.009 (2)	−0.0001 (19)
C19	0.084 (2)	0.064 (2)	0.065 (2)	0.0100 (18)	−0.0134 (18)	−0.0054 (18)
C20	0.0585 (17)	0.0553 (18)	0.061 (2)	0.0048 (14)	−0.0126 (15)	−0.0198 (15)

Geometric parameters (Å, °)

Se1—C3	1.852 (4)	C7—H7A	0.9300
Se1—C4	1.883 (3)	C8—C9	1.351 (5)
Se2—C13	1.851 (3)	C8—H8A	0.9300
Se2—C14	1.878 (3)	C9—C10	1.379 (5)
Cl1—C1	1.773 (4)	C9—H9A	0.9300
Cl2—C3	1.706 (3)	C10—H10A	0.9300
Cl3—C13	1.703 (3)	C11—C12	1.490 (4)
Cl4—C11	1.779 (3)	C11—H11A	0.9700
N1—C4	1.282 (4)	C11—H11B	0.9700
N1—C2	1.386 (4)	C12—C13	1.341 (4)
N2—C14	1.291 (3)	C14—C15	1.464 (4)

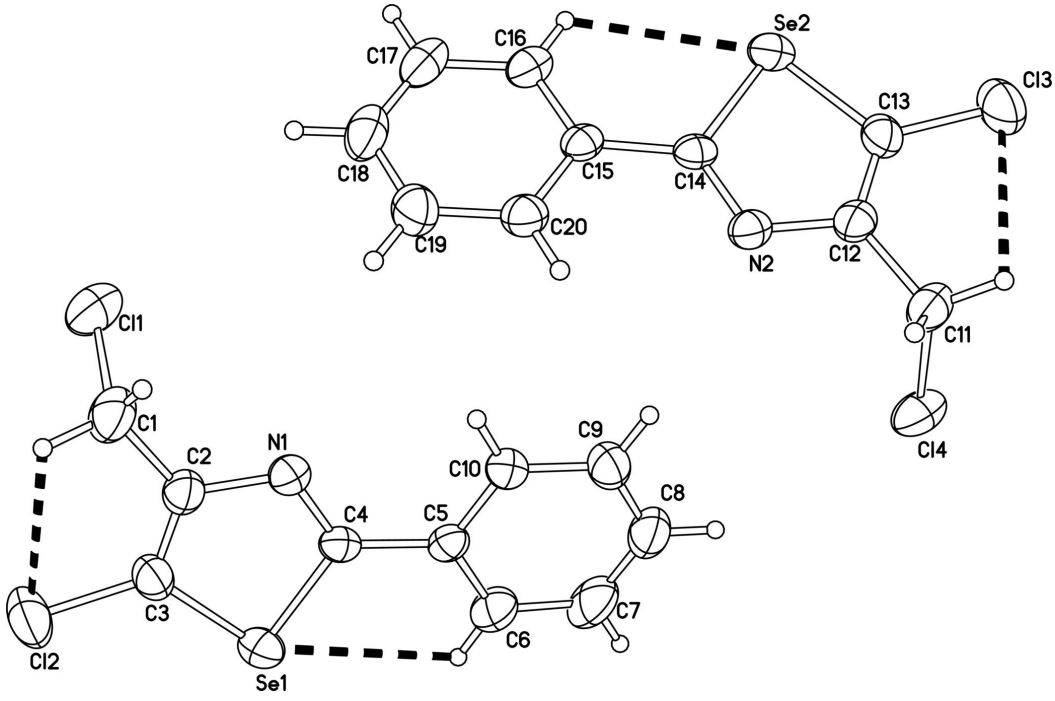
N2—C12	1.372 (4)	C15—C16	1.386 (4)
C1—C2	1.482 (5)	C15—C20	1.389 (4)
C1—H1A	0.9700	C16—C17	1.380 (5)
C1—H1B	0.9700	C16—H16A	0.9300
C2—C3	1.342 (5)	C17—C18	1.365 (6)
C4—C5	1.464 (4)	C17—H17A	0.9300
C5—C6	1.380 (4)	C18—C19	1.371 (5)
C5—C10	1.388 (4)	C18—H18A	0.9300
C6—C7	1.393 (6)	C19—C20	1.381 (5)
C6—H6A	0.9300	C19—H19A	0.9300
C7—C8	1.364 (6)	C20—H20A	0.9300
C3—Se1—C4	83.79 (14)	C9—C10—H10A	119.8
C13—Se2—C14	83.25 (13)	C5—C10—H10A	119.8
C4—N1—C2	113.8 (3)	C12—C11—Cl4	111.8 (2)
C14—N2—C12	113.7 (3)	C12—C11—H11A	109.3
C2—C1—Cl1	112.0 (3)	Cl4—C11—H11A	109.3
C2—C1—H1A	109.2	C12—C11—H11B	109.3
Cl1—C1—H1A	109.2	Cl4—C11—H11B	109.3
C2—C1—H1B	109.2	H11A—C11—H11B	107.9
Cl1—C1—H1B	109.2	C13—C12—N2	115.9 (3)
H1A—C1—H1B	107.9	C13—C12—C11	125.9 (3)
C3—C2—N1	116.1 (3)	N2—C12—C11	118.1 (3)
C3—C2—C1	125.5 (3)	C12—C13—Cl3	126.1 (3)
N1—C2—C1	118.3 (3)	C12—C13—Se2	112.4 (2)
C2—C3—Cl2	126.1 (3)	Cl3—C13—Se2	121.5 (2)
C2—C3—Se1	111.7 (2)	N2—C14—C15	124.0 (3)
Cl2—C3—Se1	122.1 (2)	N2—C14—Se2	114.8 (2)
N1—C4—C5	124.1 (3)	C15—C14—Se2	121.2 (2)
N1—C4—Se1	114.5 (2)	C16—C15—C20	118.8 (3)
C5—C4—Se1	121.3 (2)	C16—C15—C14	121.7 (3)
C6—C5—C10	118.7 (3)	C20—C15—C14	119.5 (3)
C6—C5—C4	122.4 (3)	C17—C16—C15	120.4 (3)
C10—C5—C4	118.9 (3)	C17—C16—H16A	119.8
C5—C6—C7	119.7 (4)	C15—C16—H16A	119.8
C5—C6—H6A	120.2	C18—C17—C16	120.1 (3)
C7—C6—H6A	120.2	C18—C17—H17A	120.0
C8—C7—C6	120.5 (4)	C16—C17—H17A	120.0
C8—C7—H7A	119.7	C17—C18—C19	120.4 (3)
C6—C7—H7A	119.7	C17—C18—H18A	119.8
C9—C8—C7	120.1 (4)	C19—C18—H18A	119.8
C9—C8—H8A	119.9	C18—C19—C20	120.1 (4)
C7—C8—H8A	119.9	C18—C19—H19A	120.0
C8—C9—C10	120.5 (4)	C20—C19—H19A	120.0
C8—C9—H9A	119.7	C19—C20—C15	120.2 (3)
C10—C9—H9A	119.7	C19—C20—H20A	119.9
C9—C10—C5	120.5 (3)	C15—C20—H20A	119.9
C4—N1—C2—C3	0.2 (4)	C14—N2—C12—C13	0.0 (4)
C4—N1—C2—C1	178.5 (3)	C14—N2—C12—C11	178.4 (2)

Cl1—C1—C2—C3	−100.3 (4)	Cl4—C11—C12—C13	−100.8 (3)
Cl1—C1—C2—N1	81.6 (4)	Cl4—C11—C12—N2	81.0 (3)
N1—C2—C3—Cl2	179.5 (2)	N2—C12—C13—Cl3	179.6 (2)
C1—C2—C3—Cl2	1.3 (5)	C11—C12—C13—Cl3	1.4 (5)
N1—C2—C3—Se1	0.4 (4)	N2—C12—C13—Se2	0.2 (3)
C1—C2—C3—Se1	−177.8 (3)	C11—C12—C13—Se2	−178.1 (2)
C4—Se1—C3—C2	−0.6 (2)	C14—Se2—C13—C12	−0.2 (2)
C4—Se1—C3—Cl2	−179.7 (2)	C14—Se2—C13—Cl3	−179.7 (2)
C2—N1—C4—C5	179.6 (3)	C12—N2—C14—C15	179.6 (2)
C2—N1—C4—Se1	−0.6 (3)	C12—N2—C14—Se2	−0.2 (3)
C3—Se1—C4—N1	0.7 (2)	C13—Se2—C14—N2	0.2 (2)
C3—Se1—C4—C5	−179.5 (2)	C13—Se2—C14—C15	−179.6 (2)
N1—C4—C5—C6	−172.8 (3)	N2—C14—C15—C16	−172.1 (3)
Se1—C4—C5—C6	7.4 (4)	Se2—C14—C15—C16	7.7 (4)
N1—C4—C5—C10	6.2 (4)	N2—C14—C15—C20	6.4 (4)
Se1—C4—C5—C10	−173.6 (2)	Se2—C14—C15—C20	−173.8 (2)
C10—C5—C6—C7	−0.8 (5)	C20—C15—C16—C17	−1.1 (4)
C4—C5—C6—C7	178.2 (3)	C14—C15—C16—C17	177.4 (3)
C5—C6—C7—C8	1.7 (6)	C15—C16—C17—C18	0.2 (5)
C6—C7—C8—C9	−1.2 (7)	C16—C17—C18—C19	0.9 (5)
C7—C8—C9—C10	−0.1 (6)	C17—C18—C19—C20	−1.1 (6)
C8—C9—C10—C5	1.1 (5)	C18—C19—C20—C15	0.2 (5)
C6—C5—C10—C9	−0.6 (4)	C16—C15—C20—C19	0.9 (4)
C4—C5—C10—C9	−179.6 (3)	C14—C15—C20—C19	−177.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1B···Cl2	0.97	2.78	3.213 (5)	108
C6—H6A···Se1	0.93	2.79	3.219 (4)	109
C11—H11B···Cl3	0.97	2.80	3.222 (4)	107
C16—H16A···Se2	0.93	2.76	3.200 (3)	110

Fig. 1


Se2
C16
C17
C13
Cl3
C18
C15
C14
C20
C19
N2
C12
Cl1
C11
C1
C9
N1
C2
C10
C8
C4
C5
C3
C7
C6
Cl2
Se1
Cl4