



A new polymorph of phenylselenium trichloride

Hannah R. Bloomfield and Jamie S. Ritch

Acta Cryst. (2019). **C75**, 1471–1474



IUCr Journals

CRYSTALLOGRAPHY JOURNALS ONLINE

Copyright © International Union of Crystallography

Author(s) of this article may load this reprint on their own web site or institutional repository provided that this cover page is retained. Republication of this article or its storage in electronic databases other than as specified above is not permitted without prior permission in writing from the IUCr.

For further information see <http://journals.iucr.org/services/authorrights.html>

A new polymorph of phenylselenium trichloride

Hannah R. Bloomfield and Jamie S. Ritch*

Department of Chemistry, The University of Winnipeg, 515 Portage Avenue, Winnipeg, MB, R3B 2E9, Canada.

*Correspondence e-mail: j.ritch@uwinnipeg.ca

Received 31 August 2019

Accepted 20 September 2019

Edited by A. R. Kennedy, University of Strathclyde, Scotland

Keywords: selenium compound; polymorph; phenylselenium trichloride; crystal structure; chalcogen–halogen interaction.

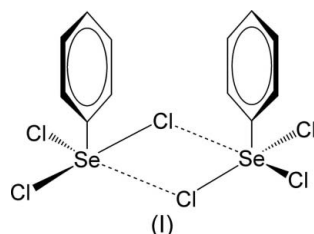
CCDC reference: 1954913

Supporting information: this article has supporting information at journals.iucr.org/c

A second polymorph of phenylselenium trichloride, PhSeCl_3 or $\text{C}_6\text{H}_5\text{Cl}_3\text{Se}$, is disclosed, which is comprised of asymmetric chlorine-bridged noncovalent dimer units rather than polymeric chains. These dimers are each weakly bound to an adjacent dimer through noncovalent $\text{Se}\cdots\text{Cl}$ bonding interactions. Phenyl rings within each dimer are oriented in a *syn* fashion. Density functional theory (DFT) calculations reveal that the putative *anti* isomer is within 5 kJ mol^{-1} of the experimentally observed form. This structure represents the first additional polymorph discovered for an organoselenium trihalide compound.

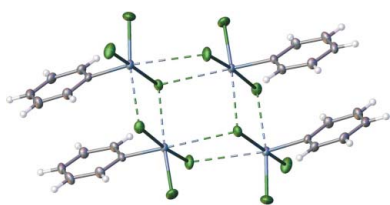
1. Introduction

Organoselenium compounds are of practical interest for synthetic transformations such as allylic oxidations and epoxidations. Due to the mild conditions often required, they are capable of stereo-, regio- and chemoselective reactivity (Tiecco, 2000).



Phenylselenium trichloride is a pale-yellow hygroscopic solid (Engman, 1987). The high polarizability of selenium, paired with its high oxidizability and weaker carbon–selenium bond (compared to the sulfur analogue), makes the phenylselenium group a more versatile auxiliary group compared to the sulfur analogue (Wessjohann & Sinks, 1998). It has found use in the large-scale synthesis of α -phenylselenyl aldehydes (Houllemare *et al.*, 1997) and has been reacted with alkenes to form β -chloroalkyl selenide dichloride adducts for the synthesis of polyfunctional molecules in a stereospecific manner (Garratt & Schmid, 1974). It also been used as a successful alternative to PhSeBr_3 in the synthesis of selenosulfonates, showing improved yields (Back *et al.*, 1987).

Organoselenium halides also exhibit unique structural motifs in the solid state, due to the possibility of various combinations of chalcogen- and halogen-bonding interactions, which are subtly influenced by substituent effects and crystallization conditions. While quite a few structures have been reported for selenium(II) halogen compounds, crystallographic data for selenium(IV) compounds remain rare: 213 entries in the Cambridge Structural Database (Groom *et al.*, 2016; Version 5.40, with updates 1 and 2) are found for the $\text{C}-\text{Se}-\text{X}$ fragment, but only ten for the $\text{C}-\text{SeX}_3$ fragment ($\text{X} = \text{F}, \text{Cl}, \text{Br}$ and I). This dearth of structural information for



higher-valent organoselenium halides provides an opportunity for new structural discoveries within this class of compounds.

The sole reported polymorph of PhSeCl₃ (Barnes *et al.*, 2005) exhibits a repeating polymeric motif where each PhSeCl₃ molecule bridges two others *via* a Cl atom. In this article, we present a second polymorph of phenylselenium trichloride featuring a dimeric motif grown under different crystallization conditions, and examine its structural features.

2. Experimental

2.1. Synthesis and crystallization

Reactions were conducted under an inert atmosphere of argon using standard Schlenk techniques. Diethyl ether and hexanes were dried using a solvent purification system, and stored in PTFE-stoppered flasks over activated 4 Å molecular sieves. CDCl₃ was dried by stirring over calcium hydride, then distilled, degassed using four freeze–pump–thaw cycles and stored in a PTFE-stoppered flask over activated 4 Å molecular sieves. Benzeneseleninic acid was prepared according to the reported procedure of Syper & Młochowski (1984) and converted to benzeneseleninic anhydride by heating to 180 °C under dynamic vacuum for 3 h. The title compound, (I), was prepared in an attempt to synthesize benzeneseleninyl chloride [*i.e.* PhSe(O)Cl] *via* a reported procedure starting with benzeneseleninic anhydride (Rosenfeld, 1976).

For the synthesis of phenylselenium trichloride, a 100 ml Schlenk flask was charged with phenylseleninic anhydride (1.010 g, 2.80 mmol) and diethyl ether (40 ml). The resulting suspension was cooled to –78 °C and thionyl chloride (3.7 ml, 50.7 mmol) was added dropwise over a period of 2 min. After stirring for an additional 10 min, the mixture was allowed to warm slowly to room temperature and stirred for 2 h, followed by gentle heating to 35 °C for 80 min. The volatiles were then removed under vacuum and the crude product was washed with hexanes (3 × 20 ml). After drying under vacuum, a pale-cream-coloured solid was obtained (yield: 1.405 g, 5.35 mmol, 95%). ¹H NMR (CDCl₃): δ 8.27–8.19 (*m*, 2H), 7.61–7.53 (*m*, 3H). Crystals suitable for X-ray diffraction were obtained from a solution of the compound in CDCl₃, layered with hexanes and stored at –20 °C.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Carbon-bound H atoms were placed in calculated positions (C–H = 0.95 Å) and refined according to a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. No disorder or solvent molecules were noted in the difference density map.

2.3. Computational studies

Closed-shell density functional theory (DFT) calculations were performed using the GAMESS software package (Schmidt *et al.*, 1993). The dispersion-corrected functional ωB97X-D (Chai & Head-Gordon, 2008) was used, with the triple-ζ quality basis set def2-TZVP (Weigend & Ahlrichs,

Table 1
Experimental details.

Crystal data	
Chemical formula	C ₆ H ₅ Cl ₃ Se
M_r	262.41
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	150
a, b, c (Å)	8.6245 (3), 9.5342 (4), 11.7229 (5)
α, β, γ (°)	78.332 (2), 77.789 (2), 66.341 (2)
V (Å ³)	855.50 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm ^{–1})	5.24
Crystal size (mm)	0.53 × 0.52 × 0.35
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Numerical (SADABS; Bruker, 2016)
$T_{\text{min}}, T_{\text{max}}$	0.456, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15455, 4426, 3470
R_{int}	0.037
$(\sin \theta/\lambda)_{\text{max}}$ (Å ^{–1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.029, 0.051, 1.05
No. of reflections	4426
No. of parameters	182
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ^{–3})	0.46, –0.57

Computer programs: APEX3 (Bruker, 2016), SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b) and OLEX2 (Dolomanov *et al.*, 2009).

2005) applied to all atoms. No symmetry restrictions were applied during geometry optimizations. The nature of all stationary points, and the Gibbs energy corrections, were obtained by analysis of the vibrational frequencies.

3. Results and discussion

In an attempt to prepare benzeneseleninyl chloride [*i.e.* PhSe(O)Cl], we conducted the reaction of benzeneseleninic anhydride with excess thionyl chloride as described previously (Rosenfeld, 1976). In our hands, this procedure afforded an excellent yield of phenylselenium trichloride. It seems possible that in the previously described work, adventitious moisture partially hydrolyzed the PhSeCl₃ product to PhSe(O)Cl, which was the isolated species. This procedure therefore represents a new route to PhSeCl₃, which is more commonly prepared by the chlorination of Ph₂Se₂ with SO₂Cl₂ (Engman, 1987). Other methods are known, including the reaction of benzeneseleninic acid with thionyl chloride (Stuhr-Hansen *et al.*, 1996).

The structure of the title compound, (I), was confirmed by X-ray studies of single crystals obtained from CDCl₃/hexanes, where a unique unit cell in the space group $P\bar{1}$ was found. The only previously characterized polymorph of PhSeCl₃, also with space group $P\bar{1}$, was obtained by the slow cooling of a diethyl ether solution of the compound. The presently determined structure has a slightly larger unit-cell volume (by *ca* 3%) compared to the known structure.

The unit cell contains two crystallographically independent molecules arranged in an asymmetric dimeric fashion (Fig. 1) *via* two Se···Cl bridging interactions [Se···Cl = 2.7975 (7) and

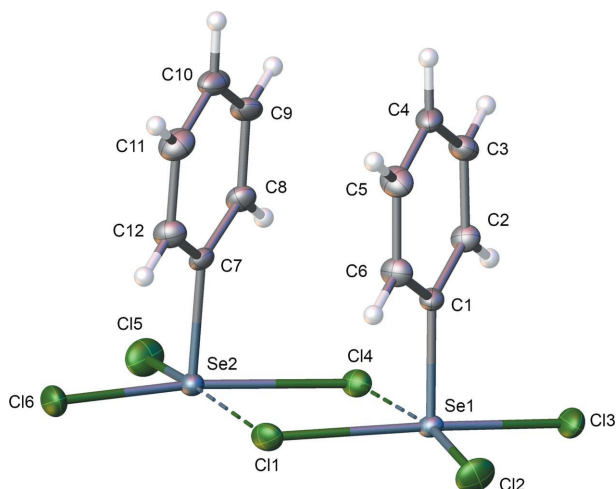


Figure 1
Displacement ellipsoid plot (non-H atoms are drawn at the 50% probability level) of the dimeric unit of PhSeCl_3 .

2.8070 (7) Å]. These distances are well within the sum of the van der Waals radii of 3.65 Å (Bondi, 1964). Within each monomer, the Se–Cl bond lengths for the bridging Cl atoms are 2.5648 (7)–2.5900 (6) Å, while for the terminal Cl atoms they are in the range 2.1769 (7)–2.2643 (7) Å, which are in accordance with the sum of the covalent radii of 2.22 Å (Cordero *et al.*, 2008). The atoms comprising the Se_2Cl_6 unit are nearly coplanar (r.m.s. deviation of 0.060 Å), and the *cis* Cl–Se–Cl angles are in the range 84.06 (2)–94.38 (3)°. The two phenyl rings of the dimer adopt a *syn* configuration and are close to being parallel, with a mean-plane-to-mean-plane dihedral angle of 5.5 (1)°. The centroid-to-centroid distance is 3.6987 (2) Å. Given the face-to-face nature of the stacked aromatic rings, an attractive π – π stacking interaction is unlikely as this orientation is normally electrostatically repulsive in nature (Martinez & Iverson, 2012). Taking the formula as $\text{C}_6\text{H}_5\text{SeCl}_3$, the unit cell features $Z = 4$ (Fig. 2).

The known polymorph of PhSeCl_3 (Barnes *et al.*, 2005) exhibits a chain polymer structure through noncovalent interactions [Se...Cl = 2.616 (5)–2.726 (5) Å], giving each selenium centre a square-pyramidal geometry. The monomers feature similar metrical parameters to those in the present polymorph. Phenyl substituents from adjacent chains are interwoven in a slip-stacked fashion, which could possibly lead to an attractive quadrupole–quadrupole interaction between the rings (Martinez & Iverson, 2012). Adjacent monomers feature *ca* 70° angles between the SeCl_4 basal planes. By contrast, in the present structure, these are nearly coplanar and no polymer chains are observed. Instead, longer Se...Cl contacts in the range 3.3760 (6)–3.4099 (7) Å join two adjacent dimeric units related by a centre of symmetry, giving each selenium centre a pseudo-octahedral geometry and forming a double *seco*-cubane motif (Fig. 3). These Se_4 clusters are weakly bound to adjacent clusters *via* type I chlorine...chlorine contacts of *ca* 3.3 Å, forming chains.

Two *para*-halogenated analogues of PhSeCl_3 have been crystallographically characterized (Barnes *et al.*, 2012). The fluorinated molecule crystallizes in the $C2/c$ space group and

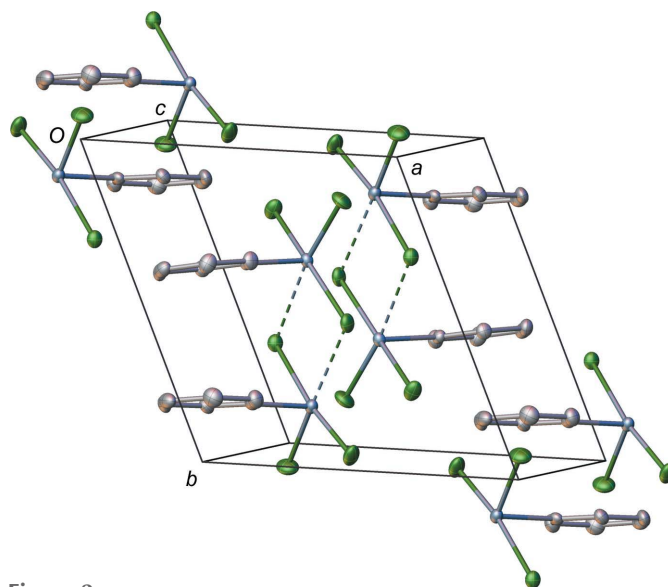


Figure 2
The packing diagram for PhSeCl_3 .

features a structural motif quite similar to the new PhSeCl_3 polymorph, with two *syn* dimers linked together to give pseudo-octahedral selenium centres with similar bond lengths and angles. The chlorinated molecule also crystallizes in the $C2/c$ space group, but is a cocrystallized mixture of *syn*- and *anti*-configuration dimers, and only one of the Se atoms within the *syn*-conformer features an additional Se...Cl contact to another dimer. This previous study illustrates the effect aromatic ring substituents can have on the aggregation between ArSeX_3 fragments. The present discovery of an additional polymorph of PhSeCl_3 further illustrates that changing aggregation is possible by simply varying the crystallization conditions, *e.g.* solvent choice.

To gain further insight into the energetics of the PhSeCl_3 system, DFT calculations were performed on the $\text{PhSeCl}_2(\mu\text{-Cl})_2\text{SeCl}_2\text{Ph}$ dimers. Several dispersion-corrected functionals (B97-D3, M06-2X, PBE0-D3 and ω B97X-D) were tested to examine how closely they reproduced the experimental geometry of the dimer (ignoring the long-range Se...Cl contacts). The M06-2X functional had convergence issues and

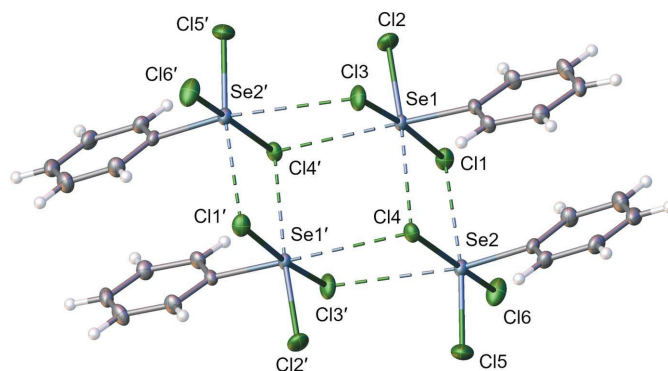


Figure 3
Secondary bonding interactions in the crystal structure of PhSeCl_3 . Atoms marked with a prime symbol are related to the unprimed atoms by $(-x + 1, -y + 1, -z + 1)$.

both functionals using Grimme's D3 dispersion correction yielded symmetrically-bridging Cl atoms. Only the ω B97X-D functional closely reproduced the asymmetric dimer structure. Bond lengths were closely replicated, though the two bridging interactions linking each PhSeCl₃ monomer were longer than experimentally observed by *ca* 7%, and the two phenyl rings were slightly slipped from a face-to-face orientation. It is possible that attempting to model a second dimer and the corresponding long-range contacts would result in a more accurate geometry reproduction, though it was deemed too computationally expensive at this level of theory.

When the hypothetical *anti* isomer was modelled it also exhibited an asymmetric dimer motif, with a Gibbs energy only *ca* 5 kJ mol⁻¹ higher than the *syn* isomer. While these computations are simplified from the solid-state structure, they illustrate the small energy differences between various conformers and highlight that other polymorphs, *i.e.* the *anti* isomer of PhSeCl₃, may be experimentally accessible under the right crystallization conditions.

Acknowledgements

Dr David Herbert (Department of Chemistry, University of Manitoba) is thanked for access to a single-crystal X-ray diffractometer. Funding for this research was provided by the Natural Sciences and Engineering Research Council of Canada.

References

Back, T. G., Collins, S. & Krishna, M. V. (1987). *Can. J. Chem.* **65**, 38–42.

- Barnes, N. A., Godfrey, S. M., Halton, R. T. A. & Pritchard, R. G. (2005). *Dalton Trans.* pp. 1759–1761.
- Barnes, N. A., Godfrey, S. M., Ollerenshaw, R. T. A., Khan, R. Z. & Pritchard, R. G. (2012). *Dalton Trans.* **41**, 14583–14593.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Bruker (2016). *APEX3, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chai, J.-D. & Head-Gordon, M. (2008). *Phys. Chem. Chem. Phys.* **10**, 6615–6620.
- Cordero, B., Gómez, V., Platero-Prats, A. E., Revés, M., Echeverría, J., Cremades, E., Barragán, F. & Alvarez, S. (2008). *Dalton Trans.* pp. 2832–2838.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Engman, L. (1987). *J. Org. Chem.* **52**, 4086–4094.
- Garratt, D. G. & Schmid, G. H. (1974). *Can. J. Chem.* **52**, 3599–3606.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Houllemare, D., Ponthieux, S., Outurquin, F. & Paulmier, C. (1997). *Synthesis*, **1997**, 101–106.
- Martinez, C. R. & Iverson, B. L. (2012). *Chem. Sci.* **3**, 2191–2201.
- Rosenfeld, M. N. (1976). PhD thesis, Imperial College London, UK.
- Schmidt, M. W., Baldrige, K. K., Boatz, J. A., Elbert, S. T., Gordon, M. S., Jensen, J. H., Koseki, S., Matsunaga, N., Nguyen, K. A., Su, S., Windus, T. L., Dupuis, M. & Montgomery, J. A. (1993). *J. Comput. Chem.* **14**, 1347–1363.
- Sheldrick, G. M. (2015a). *Acta Cryst.* **A71**, 3–8.
- Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
- Stuhr-Hansen, N., Henriksen, L. & Kodra, J. T. (1996). *Synth. Commun.* **26**, 3345–3350.
- Syper, L. & Młochowski, J. (1984). *Synthesis*, **1984**, 747–752.
- Tiecco, M. (2000). *Organoselenium Chemistry. Topics in Current Chemistry*, Vol. 208, edited by T. Wirth, pp. 7–54. Berlin, Heidelberg: Springer.
- Weigend, F. & Ahlrichs, R. (2005). *Phys. Chem. Chem. Phys.* **7**, 3297–3305.
- Wessjohann, L. & Sinks, U. (1998). *J. Prakt. Chem.* **340**, 189–203.

supporting information

Acta Cryst. (2019). **C75**, 1471-1474 [https://doi.org/10.1107/S2053229619013019]

A new polymorph of phenylselenium trichloride

Hannah R. Bloomfield and Jamie S. Ritch

Computing details

Data collection: *APEX3* (Bruker, 2016; cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Phenylselenium trichloride

Crystal data

$C_6H_5Cl_3Se$

$M_r = 262.41$

Triclinic, $P\bar{1}$

$a = 8.6245$ (3) Å

$b = 9.5342$ (4) Å

$c = 11.7229$ (5) Å

$\alpha = 78.332$ (2)°

$\beta = 77.789$ (2)°

$\gamma = 66.341$ (2)°

$V = 855.50$ (6) Å³

$Z = 4$

$F(000) = 504$

$D_x = 2.037$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8248 reflections

$\theta = 2.6$ – 32.6 °

$\mu = 5.24$ mm⁻¹

$T = 150$ K

Prism, clear colourless

$0.53 \times 0.52 \times 0.35$ mm

Data collection

Bruker APEXII CCD

diffractometer

φ and ω scans

Absorption correction: numerical

(SADABS; Bruker, 2016)

$T_{\min} = 0.456$, $T_{\max} = 0.747$

15455 measured reflections

4426 independent reflections

3470 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 28.7$ °, $\theta_{\min} = 2.6$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.051$

$S = 1.05$

4426 reflections

182 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0095P)^2 + 0.5686P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.46$ e Å⁻³

$\Delta\rho_{\min} = -0.57$ e Å⁻³

Extinction correction: SHELXL2018

(Sheldrick, 2015b),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0114 (3)

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.46873 (3)	0.37174 (3)	0.37701 (2)	0.01526 (7)
Se2	0.33135 (3)	0.82730 (3)	0.29123 (2)	0.01719 (7)
Cl4	0.23956 (8)	0.64558 (7)	0.46326 (5)	0.02005 (13)
Cl1	0.57258 (8)	0.55087 (8)	0.21325 (5)	0.02304 (14)
Cl3	0.37555 (9)	0.22740 (8)	0.53285 (6)	0.02807 (16)
Cl2	0.67527 (8)	0.17097 (8)	0.30719 (6)	0.02984 (16)
Cl5	0.15514 (9)	1.02879 (8)	0.37443 (6)	0.03339 (17)
Cl6	0.44520 (9)	0.96373 (8)	0.14372 (6)	0.03359 (17)
C1	0.3008 (3)	0.3840 (3)	0.2832 (2)	0.0155 (5)
C7	0.1696 (3)	0.8222 (3)	0.1993 (2)	0.0170 (5)
C8	0.0049 (3)	0.8412 (3)	0.2539 (2)	0.0199 (5)
H8	-0.029076	0.855370	0.334641	0.024*
C2	0.1329 (3)	0.4140 (3)	0.3366 (2)	0.0185 (5)
H2	0.100984	0.424952	0.417965	0.022*
C6	0.3511 (3)	0.3669 (3)	0.1649 (2)	0.0213 (5)
H6	0.466331	0.347356	0.129613	0.026*
C9	-0.1095 (3)	0.8390 (3)	0.1870 (2)	0.0235 (6)
H9	-0.223387	0.852156	0.222208	0.028*
C4	0.0613 (3)	0.4096 (3)	0.1506 (2)	0.0218 (6)
H4	-0.021439	0.418381	0.104667	0.026*
C3	0.0130 (3)	0.4276 (3)	0.2690 (2)	0.0215 (6)
H3	-0.102844	0.449365	0.303758	0.026*
C12	0.2236 (3)	0.7984 (3)	0.0828 (2)	0.0229 (6)
H12	0.337873	0.784085	0.047616	0.027*
C10	-0.0587 (3)	0.8180 (3)	0.0700 (2)	0.0253 (6)
H10	-0.138341	0.818728	0.024752	0.030*
C5	0.2290 (3)	0.3791 (3)	0.0991 (2)	0.0248 (6)
H5	0.261162	0.366247	0.018056	0.030*
C11	0.1075 (4)	0.7958 (3)	0.0183 (2)	0.0281 (6)
H11	0.142480	0.778644	-0.061869	0.034*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.01433 (13)	0.01787 (13)	0.01336 (13)	-0.00551 (10)	-0.00342 (10)	-0.00121 (10)
Se2	0.01727 (13)	0.01849 (14)	0.01750 (14)	-0.00836 (11)	-0.00562 (10)	0.00074 (10)
Cl4	0.0202 (3)	0.0267 (3)	0.0139 (3)	-0.0100 (3)	-0.0013 (2)	-0.0028 (2)
Cl1	0.0160 (3)	0.0320 (4)	0.0201 (3)	-0.0105 (3)	0.0004 (2)	-0.0012 (3)
Cl3	0.0340 (4)	0.0354 (4)	0.0197 (3)	-0.0209 (3)	-0.0104 (3)	0.0092 (3)

Cl2	0.0234 (3)	0.0235 (3)	0.0337 (4)	0.0028 (3)	-0.0049 (3)	-0.0083 (3)
Cl5	0.0405 (4)	0.0216 (3)	0.0387 (4)	-0.0062 (3)	-0.0105 (3)	-0.0118 (3)
Cl6	0.0313 (4)	0.0375 (4)	0.0365 (4)	-0.0235 (3)	-0.0116 (3)	0.0146 (3)
C1	0.0184 (12)	0.0160 (12)	0.0148 (12)	-0.0078 (10)	-0.0057 (10)	-0.0016 (10)
C7	0.0180 (12)	0.0139 (12)	0.0189 (13)	-0.0043 (10)	-0.0083 (10)	0.0002 (10)
C8	0.0166 (12)	0.0199 (13)	0.0199 (13)	-0.0030 (10)	-0.0034 (10)	-0.0028 (11)
C2	0.0199 (13)	0.0221 (13)	0.0132 (12)	-0.0075 (11)	-0.0009 (10)	-0.0041 (10)
C6	0.0192 (13)	0.0285 (14)	0.0160 (13)	-0.0094 (11)	0.0012 (10)	-0.0056 (11)
C9	0.0156 (13)	0.0227 (14)	0.0313 (15)	-0.0049 (11)	-0.0084 (11)	-0.0011 (12)
C4	0.0279 (15)	0.0236 (14)	0.0198 (13)	-0.0138 (12)	-0.0111 (11)	0.0012 (11)
C3	0.0182 (13)	0.0258 (14)	0.0223 (14)	-0.0105 (11)	-0.0036 (11)	-0.0018 (11)
C12	0.0192 (13)	0.0277 (14)	0.0203 (13)	-0.0070 (11)	-0.0022 (11)	-0.0046 (11)
C10	0.0280 (14)	0.0209 (14)	0.0310 (15)	-0.0082 (12)	-0.0154 (12)	-0.0026 (12)
C5	0.0301 (15)	0.0333 (16)	0.0150 (13)	-0.0154 (13)	-0.0019 (11)	-0.0062 (11)
C11	0.0339 (16)	0.0312 (16)	0.0195 (14)	-0.0100 (13)	-0.0074 (12)	-0.0052 (12)

Geometric parameters (Å, °)

Se1—Cl4 ⁱ	3.3760 (6)	C8—C9	1.393 (3)
Se1—Cl4	2.7975 (7)	C2—H2	0.9500
Se1—Cl11	2.5648 (7)	C2—C3	1.382 (3)
Se1—Cl3	2.2643 (7)	C6—H6	0.9500
Se1—Cl2	2.1881 (7)	C6—C5	1.388 (3)
Se1—C1	1.951 (2)	C9—H9	0.9500
Se2—Cl4	2.5900 (6)	C9—C10	1.378 (4)
Se2—Cl11	2.8070 (7)	C4—H4	0.9500
Se2—Cl3 ⁱ	3.4099 (7)	C4—C3	1.388 (3)
Se2—Cl5	2.1769 (7)	C4—C5	1.380 (4)
Se2—Cl6	2.2458 (7)	C3—H3	0.9500
Se2—C7	1.955 (2)	C12—H12	0.9500
C1—C2	1.389 (3)	C12—C11	1.387 (3)
C1—C6	1.385 (3)	C10—H10	0.9500
C7—C8	1.384 (3)	C10—C11	1.381 (4)
C7—C12	1.380 (3)	C5—H5	0.9500
C8—H8	0.9500	C11—H11	0.9500
Cl4—Se1—Cl4 ⁱ	90.204 (17)	C6—C1—C2	121.9 (2)
Cl11—Se1—Cl4	84.71 (2)	C8—C7—Se2	118.66 (18)
Cl11—Se1—Cl4 ⁱ	89.433 (18)	C12—C7—Se2	119.06 (18)
Cl3—Se1—Cl4 ⁱ	87.29 (2)	C12—C7—C8	122.3 (2)
Cl3—Se1—Cl4	91.45 (2)	C7—C8—H8	121.0
Cl3—Se1—Cl11	174.95 (2)	C7—C8—C9	118.0 (2)
Cl2—Se1—Cl4 ⁱ	83.89 (2)	C9—C8—H8	121.0
Cl2—Se1—Cl4	172.03 (2)	C1—C2—H2	120.7
Cl2—Se1—Cl11	89.87 (3)	C3—C2—C1	118.7 (2)
Cl2—Se1—Cl3	93.60 (3)	C3—C2—H2	120.7
C1—Se1—Cl4	89.59 (7)	C1—C6—H6	120.8
C1—Se1—Cl4 ⁱ	179.21 (7)	C1—C6—C5	118.4 (2)

C1—Se1—C11	89.78 (7)	C5—C6—H6	120.8
C1—Se1—C13	93.48 (7)	C8—C9—H9	119.7
C1—Se1—C12	96.25 (7)	C10—C9—C8	120.5 (2)
C14—Se2—C11	84.06 (2)	C10—C9—H9	119.7
C14—Se2—C13 ⁱ	81.751 (18)	C3—C4—H4	119.8
C11—Se2—C13 ⁱ	83.668 (19)	C5—C4—H4	119.8
C15—Se2—C14	90.83 (3)	C5—C4—C3	120.3 (2)
C15—Se2—C11	172.41 (3)	C2—C3—C4	120.2 (2)
C15—Se2—C13 ⁱ	90.06 (2)	C2—C3—H3	119.9
C15—Se2—C16	94.38 (3)	C4—C3—H3	119.9
C16—Se2—C14	172.72 (3)	C7—C12—H12	120.7
C16—Se2—C11	90.24 (3)	C7—C12—C11	118.6 (2)
C16—Se2—C13 ⁱ	93.14 (2)	C11—C12—H12	120.7
C7—Se2—C14	90.64 (7)	C9—C10—H10	119.8
C7—Se2—C11	89.74 (7)	C9—C10—C11	120.3 (2)
C7—Se2—C13 ⁱ	170.39 (7)	C11—C10—H10	119.8
C7—Se2—C15	95.94 (7)	C6—C5—H5	119.8
C7—Se2—C16	93.87 (7)	C4—C5—C6	120.5 (2)
Se2—C14—Se1	95.36 (2)	C4—C5—H5	119.8
Se1—C11—Se2	95.70 (2)	C12—C11—H11	119.9
C2—C1—Se1	118.78 (17)	C10—C11—C12	120.3 (2)
C6—C1—Se1	119.29 (18)	C10—C11—H11	119.9

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