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3-Methyl-4,5,6,7,8,9-hexahydrocycloocta[*d*]-[1,2,3]selenadiazol]-3-ium iodide triiodide (3/2/1)

Dieter Schollmeyer and Heiner Detert*

University Mainz, Duesbergweg 10-14, 55099 Mainz, Germany. *Correspondence e-mail: detert@uni-mainz.de

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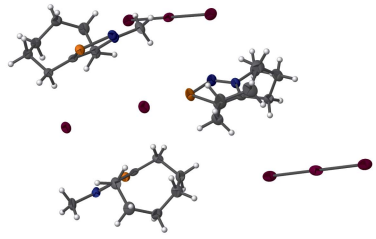
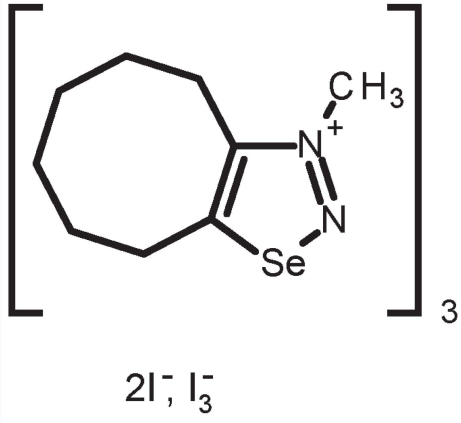
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $3C_9H_{15}N_2Se^+\cdot I_3^-\cdot 2I^-$, was prepared by methylation of the selenadiazole with methyl iodide. The asymmetric unit is composed of three independent selenadiazolium ions arranged in layers and connected *via* $I_3^-\cdot(I^-)_2$ anion layers. The distances between the iodine and selenium atoms are significantly shorter than the nitrogen-to-iodine distances.

3D view



Chemical scheme



Structure description

Selenadiazoles are synthons for the preparation of strained cycloalkynes (Bissinger *et al.*, 1988; Detert & Meier, 1997). Cycloocteno-1,2,3-selenadiazole is also known (Meier & Voigt, 1972). The structure of a chloro-1,2,3-selenadiazole, *viz* 3-chloro-5-methyl-2-*p*-tolyl-3*H*-3-selenaindazole, has been reported (Jones & De Ramírez Arellano, 1995). Benzo-annulated selenadiazolium salts have been described by Jaffari *et al.* (1970), and the only hitherto reported 1,2,3-selenadiazolium salt has been described by Butler & Fox (2001).

The asymmetric unit of the title compound, Fig. 1, is composed of three independent but nearly identical selenadiazolium ions (*A*, *B*, and *C*), two iodide and one triiodide anions. The selenadiolium ring is essentially planar, with a maximum deviation from the mean plane of 0.018 (8) Å for atom N3*A*, and the methyl group lying 0.039 (10) Å above the mean plane. The adjacent hexamethylene ether allows a staggered conformation.

In the crystal, the cations are arranged in ribbons connected *via* the iodide and triiodide anions. A view normal to plane (101) gives a *ABCA* sequence of cations (Fig. 2), but viewed along the *a* axis the sequence is *AABCCBAA* (Fig. 3). The shortest distances between anions and cations are Se1*A*—I6 = 3.349 (1) Å, Se1*B*—I3 = 3.319 (1) Å, and Se1*C*—I6 = 3.314 (1) Å. The distances to the nitrogen atom are more than *ca* 3.8 Å, like the distances of the triiodide to the selenadiazole atoms.

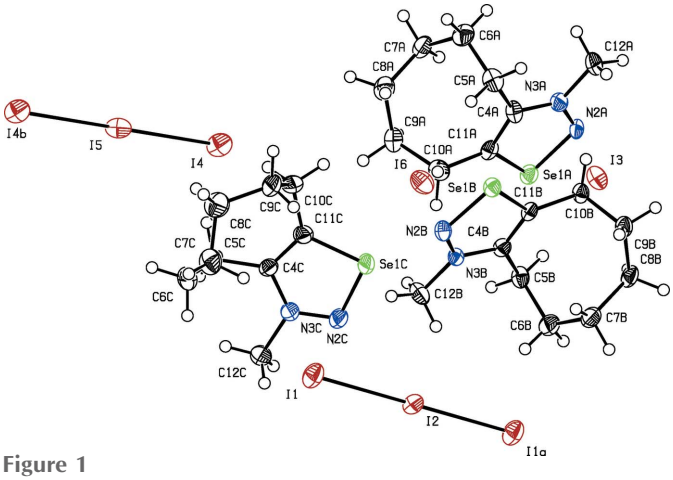


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The title compound was prepared by adding methyl iodide (3 ml) to a solution of cycloocteno-1,2,3-selenadiazole (0.65 g, 3 mmol) in nitromethane (10 ml). The mixture was kept in the dark at ambient temperature for 24 d. Evaporation of the solvent and chromatography on silica gel using chloroform/propanol-2 as eluent yielded 0.32 g of the pure title compound (34%). ^{1}H NMR: ($CDCl_3$, 400 MHz): δ = 4.48 (*s*, 3 H, CH_3), 3.68 (‘*t* 2 H, 9-H), 3.21 (‘*t* 2 H, 4-H), 1.83 (*m*, 4 H), 1.37 (*m*, 4 H), NOE: irradiation into δ = 4.48 gives positive NOEs at δ = 3.18, δ = 1.8; ^{13}C NMR ($CDCl_3$): 173.7 (C—Se), 154.4 (C—N), 49.1 CH_3, 31.2 C-9, 31.1 C-8), 27.9 C-5, 26.68, 25.64 C-6, 24.78 C-7; ^{77}Se NMR ($CDCl_3$, Me_2Se): 1299.63; MS (FD) 231.1 (100%, Se-pattern, cation), 587 (9%, Se_2 pattern, $M_2 - I^-$). Recrystallization from chloroform gave brownish-yellow crystals (m.p. 444 K).

Table 1
Experimental details.

Crystal data	
Chemical formula	$3C_9H_{15}N_2Se^+\cdot I_3^-\cdot 2I^-$
M_r	1325.07
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, *b*, *c* (Å)	7.2438 (4), 13.9980 (8), 20.1035 (11)
α, β, γ (°)	81.913 (2), 86.409 (2), 78.801 (2)
V (Å^3)	1978.46 (19)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	6.72
Crystal size (mm)	0.74 × 0.34 × 0.05
Data collection	
Diffractometer	Bruker SMART APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 1997)
T_{min}, T_{max}	0.095, 0.810
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	64010, 9472, 8038
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.659
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.149, 1.16
No. of reflections	9472
No. of parameters	376
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	3.41, −2.67

Computer programs: *SMART* and *SAINT* (Bruker (1997), *SIR2004* (Altomare *et al.*, 1999), *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

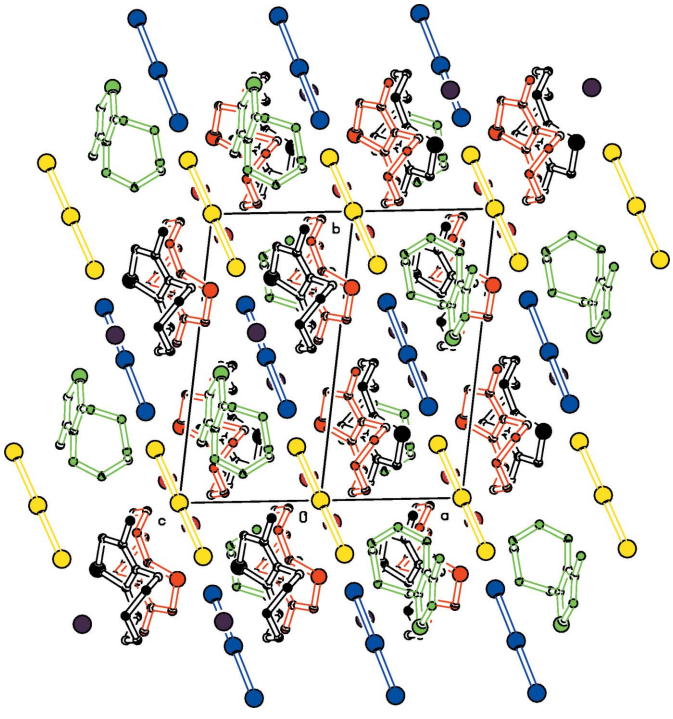


Figure 2
A view normal to plane (101) of the crystal packing of the title compound. H atoms have been omitted for clarity and the residues are drawn with different colours.

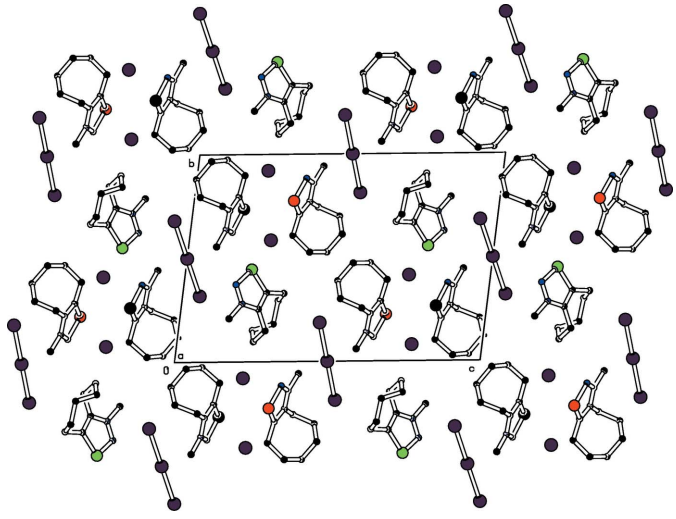


Figure 3
A view along the *a* axis of the crystal packing of the title compound. H atoms have been omitted for clarity and the residues are drawn with different colours.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

References

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full crystallographic data

IUCrData (2016). **1**, x161950 [https://doi.org/10.1107/S2414314616019507]

3-Methyl-4,5,6,7,8,9-hexahydrocycloocta[*d*][1,2,3]selenadiazol]-3-ium iodide triiodide (3/2/1)

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Crystal data

$3C_9H_{15}N_2Se^+·I_3^-·2I^-$
M_r = 1325.07
Triclinic, $P\overline{1}$
a = 7.2438 (4) Å
b = 13.9980 (8) Å
c = 20.1035 (11) Å
α = 81.913 (2)°
β = 86.409 (2)°
γ = 78.801 (2)°
V = 1978.46 (19) Å^3
Z = 2
$F(000)$ = 1232
D_x = 2.224 Mg m^{-3}
Melting point: 444 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9806 reflections
θ = 2.4–27.9°
μ = 6.72 mm^{-1}
T = 173 K
Plate, brown
0.74 × 0.34 × 0.05 mm

Data collection

Bruker SMART APEX2 diffractometer
Radiation source: sealed Tube
Graphite monochromator
CCD scan
Absorption correction: multi-scan (SADABS; Bruker, 1997)
T_{min} = 0.095, T_{max} = 0.810
64010 measured reflections
9472 independent reflections
8038 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.9°, θ_{min} = 1.0°
h = −9→9
k = −18→18
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.149
S = 1.16
9472 reflections
376 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 36.6012P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 3.41 e Å^{-3}
$\Delta\rho_{min}$ = −2.67 e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1A	−0.19773 (12)	0.77479 (6)	0.32688 (4)	0.02677 (18)
N2A	−0.1532 (11)	0.8825 (5)	0.3593 (4)	0.0280 (15)
N3A	0.0154 (10)	0.8599 (5)	0.3836 (4)	0.0260 (15)
C4A	0.1281 (12)	0.7720 (6)	0.3791 (4)	0.0261 (17)
C5A	0.3182 (12)	0.7445 (7)	0.4088 (5)	0.0302 (19)
H5A	0.374675	0.804114	0.405033	0.036*
H5B	0.399634	0.697554	0.381973	0.036*
C6A	0.3181 (13)	0.6989 (7)	0.4819 (5)	0.035 (2)
H6A	0.261513	0.750555	0.509917	0.042*
H6B	0.450164	0.675027	0.495058	0.042*
C7A	0.2097 (14)	0.6121 (7)	0.4978 (5)	0.034 (2)
H7A	0.226613	0.584570	0.545598	0.040*
H7B	0.073816	0.638640	0.492167	0.040*
C8A	0.2688 (14)	0.5273 (7)	0.4544 (5)	0.037 (2)
H8A	0.279913	0.464019	0.484315	0.044*
H8B	0.394947	0.531190	0.433428	0.044*
C9A	0.1315 (14)	0.5272 (7)	0.3982 (5)	0.035 (2)
H9A	0.162767	0.462572	0.381488	0.042*
H9B	0.002360	0.533559	0.418548	0.042*
C10A	0.1303 (12)	0.6081 (7)	0.3372 (5)	0.0300 (18)
H10A	0.261990	0.608223	0.320790	0.036*
H10B	0.062713	0.590546	0.300727	0.036*
C11A	0.0417 (11)	0.7103 (6)	0.3501 (4)	0.0230 (16)
C12A	0.0779 (15)	0.9412 (7)	0.4105 (5)	0.035 (2)
H12A	0.175533	0.964714	0.380199	0.053*
H12B	0.128929	0.917515	0.455104	0.053*
H12C	−0.029551	0.995140	0.414068	0.053*
Se1B	1.21598 (11)	0.73576 (6)	0.16638 (4)	0.02384 (17)
N2B	1.1750 (9)	0.6258 (5)	0.1369 (4)	0.0240 (14)
N3B	1.0036 (9)	0.6393 (5)	0.1162 (3)	0.0199 (13)
C4B	0.8875 (11)	0.7294 (6)	0.1200 (4)	0.0238 (16)
C5B	0.6933 (11)	0.7517 (6)	0.0947 (5)	0.0265 (17)
H5C	0.613381	0.799480	0.121370	0.032*
H5D	0.640456	0.690831	0.101318	0.032*
C6B	0.6870 (14)	0.7947 (7)	0.0190 (5)	0.036 (2)
H6C	0.746374	0.741910	−0.007743	0.043*
H6D	0.553481	0.814355	0.006687	0.043*
C7B	0.7817 (14)	0.8813 (7)	−0.0010 (5)	0.0327 (19)
H7C	0.753979	0.906879	−0.048488	0.039*

H7D	0.919476	0.857741	0.001150	0.039*
C8B	0.7278 (13)	0.9675 (7)	0.0408 (5)	0.0303 (19)
H8C	0.708259	1.029589	0.009518	0.036*
H8D	0.606017	0.962390	0.064946	0.036*
C9B	0.8711 (12)	0.9740 (6)	0.0924 (4)	0.0273 (17)
H9C	0.997699	0.966334	0.069923	0.033*
H9D	0.841455	1.040638	0.106164	0.033*
C10B	0.8797 (11)	0.8990 (6)	0.1557 (4)	0.0234 (16)
H10C	0.949369	0.920714	0.189849	0.028*
H10D	0.749787	0.898269	0.174011	0.028*
C11B	0.9722 (11)	0.7956 (6)	0.1450 (4)	0.0207 (15)
C12B	0.9456 (14)	0.5560 (7)	0.0915 (5)	0.033 (2)
H12D	1.054861	0.502759	0.088906	0.050*
H12E	0.894350	0.577125	0.046678	0.050*
H12F	0.848982	0.532511	0.122332	0.050*
Se1C	0.40931 (14)	0.44524 (7)	0.21655 (5)	0.0337 (2)
N2C	0.2961 (11)	0.3728 (6)	0.1706 (4)	0.0312 (16)
N3C	0.3370 (10)	0.2803 (5)	0.1976 (4)	0.0271 (15)
C4C	0.4462 (12)	0.2538 (6)	0.2531 (4)	0.0258 (17)
C5C	0.4998 (13)	0.1494 (6)	0.2839 (4)	0.0297 (18)
H5E	0.395430	0.114562	0.279766	0.036*
H5F	0.520468	0.147235	0.332371	0.036*
C6C	0.6789 (14)	0.0972 (7)	0.2500 (5)	0.039 (2)
H6E	0.647724	0.086074	0.204782	0.046*
H6F	0.719466	0.032028	0.276371	0.046*
C7C	0.8449 (14)	0.1516 (8)	0.2423 (6)	0.041 (2)
H7E	0.813469	0.211196	0.209141	0.050*
H7F	0.956168	0.108806	0.224006	0.050*
C8C	0.8968 (16)	0.1819 (9)	0.3080 (6)	0.048 (3)
H8E	1.034891	0.162552	0.312322	0.058*
H8F	0.836955	0.144455	0.345946	0.058*
C9C	0.8386 (16)	0.2934 (7)	0.3147 (6)	0.043 (3)
H9E	0.911668	0.308138	0.350708	0.051*
H9F	0.874475	0.331420	0.272119	0.051*
C10C	0.6295 (14)	0.3290 (7)	0.3303 (5)	0.036 (2)
H10E	0.588918	0.284786	0.368991	0.043*
H10F	0.612445	0.395417	0.344016	0.043*
C11C	0.5052 (13)	0.3329 (7)	0.2735 (4)	0.0295 (18)
C12C	0.2703 (15)	0.2090 (7)	0.1611 (5)	0.037 (2)
H12G	0.133439	0.227198	0.157111	0.056*
H12H	0.302298	0.142946	0.186038	0.056*
H12I	0.331139	0.209767	0.116168	0.056*
I1	0.74529 (9)	0.31836 (5)	0.05629 (3)	0.03944 (16)
I2	0.500000	0.500000	0.000000	0.02823 (17)
I4	0.24632 (9)	0.18708 (5)	0.45955 (3)	0.04046 (17)
I5	0.500000	0.000000	0.500000	0.0348 (2)
I3	0.33131 (8)	0.92120 (4)	0.23051 (3)	0.03323 (15)
I6	0.67814 (9)	0.58573 (5)	0.26699 (4)	0.03840 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1A	0.0252 (4)	0.0255 (4)	0.0271 (4)	0.0002 (3)	−0.0019 (3)	−0.0016 (3)
N2A	0.042 (4)	0.015 (3)	0.025 (3)	−0.002 (3)	−0.001 (3)	0.002 (3)
N3A	0.030 (4)	0.023 (3)	0.024 (3)	−0.006 (3)	0.001 (3)	−0.002 (3)
C4A	0.025 (4)	0.020 (4)	0.033 (4)	−0.004 (3)	0.007 (3)	−0.003 (3)
C5A	0.018 (4)	0.032 (5)	0.042 (5)	−0.007 (3)	0.002 (3)	−0.007 (4)
C6A	0.026 (4)	0.037 (5)	0.041 (5)	−0.004 (4)	−0.015 (4)	−0.001 (4)
C7A	0.036 (5)	0.031 (5)	0.029 (4)	0.000 (4)	−0.002 (4)	0.005 (4)
C8A	0.030 (5)	0.034 (5)	0.042 (5)	0.002 (4)	−0.006 (4)	−0.002 (4)
C9A	0.035 (5)	0.025 (4)	0.042 (5)	−0.004 (4)	0.001 (4)	−0.001 (4)
C10A	0.023 (4)	0.033 (5)	0.033 (5)	0.001 (3)	0.002 (3)	−0.010 (4)
C11A	0.021 (4)	0.025 (4)	0.020 (4)	−0.004 (3)	0.006 (3)	0.003 (3)
C12A	0.045 (6)	0.020 (4)	0.042 (5)	−0.010 (4)	−0.004 (4)	−0.004 (4)
Se1B	0.0184 (4)	0.0231 (4)	0.0285 (4)	0.0008 (3)	−0.0026 (3)	−0.0037 (3)
N2B	0.018 (3)	0.021 (3)	0.030 (4)	0.002 (3)	0.001 (3)	−0.004 (3)
N3B	0.020 (3)	0.018 (3)	0.024 (3)	−0.008 (3)	−0.002 (2)	−0.001 (2)
C4B	0.021 (4)	0.025 (4)	0.023 (4)	−0.001 (3)	0.000 (3)	−0.002 (3)
C5B	0.019 (4)	0.023 (4)	0.037 (5)	−0.002 (3)	−0.003 (3)	−0.003 (3)
C6B	0.035 (5)	0.034 (5)	0.036 (5)	−0.003 (4)	−0.012 (4)	−0.001 (4)
C7B	0.036 (5)	0.032 (5)	0.027 (4)	−0.002 (4)	−0.003 (4)	0.001 (4)
C8B	0.028 (4)	0.026 (4)	0.030 (4)	0.007 (3)	−0.003 (3)	0.003 (3)
C9B	0.025 (4)	0.023 (4)	0.031 (4)	0.002 (3)	0.001 (3)	−0.003 (3)
C10B	0.021 (4)	0.021 (4)	0.026 (4)	0.002 (3)	0.004 (3)	−0.003 (3)
C11B	0.017 (4)	0.022 (4)	0.019 (3)	0.003 (3)	0.004 (3)	0.001 (3)
C12B	0.031 (5)	0.026 (4)	0.044 (5)	−0.003 (4)	−0.003 (4)	−0.014 (4)
Se1C	0.0388 (5)	0.0218 (4)	0.0387 (5)	0.0037 (4)	−0.0162 (4)	−0.0038 (4)
N2C	0.034 (4)	0.027 (4)	0.032 (4)	−0.004 (3)	−0.012 (3)	0.001 (3)
N3C	0.028 (4)	0.025 (4)	0.028 (4)	−0.003 (3)	−0.006 (3)	−0.004 (3)
C4C	0.025 (4)	0.028 (4)	0.021 (4)	0.001 (3)	−0.002 (3)	−0.003 (3)
C5C	0.038 (5)	0.027 (4)	0.022 (4)	−0.001 (4)	−0.005 (3)	0.001 (3)
C6C	0.039 (5)	0.029 (5)	0.044 (6)	0.007 (4)	−0.005 (4)	−0.010 (4)
C7C	0.031 (5)	0.038 (5)	0.050 (6)	0.006 (4)	0.000 (4)	−0.008 (5)
C8C	0.036 (6)	0.049 (7)	0.054 (7)	0.010 (5)	−0.015 (5)	−0.010 (5)
C9C	0.052 (6)	0.030 (5)	0.043 (6)	0.003 (4)	−0.023 (5)	0.000 (4)
C10C	0.042 (5)	0.035 (5)	0.031 (5)	−0.002 (4)	−0.015 (4)	−0.007 (4)
C11C	0.030 (4)	0.030 (4)	0.027 (4)	0.001 (4)	−0.004 (3)	−0.007 (3)
C12C	0.046 (6)	0.032 (5)	0.037 (5)	−0.013 (4)	−0.012 (4)	−0.005 (4)
I1	0.0347 (3)	0.0381 (3)	0.0390 (3)	0.0032 (3)	−0.0030 (3)	0.0041 (3)
I2	0.0236 (4)	0.0337 (4)	0.0250 (4)	−0.0022 (3)	0.0000 (3)	−0.0005 (3)
I4	0.0324 (3)	0.0504 (4)	0.0351 (3)	−0.0014 (3)	0.0037 (2)	−0.0050 (3)
I5	0.0231 (4)	0.0508 (5)	0.0319 (4)	−0.0064 (4)	0.0006 (3)	−0.0120 (4)
I3	0.0282 (3)	0.0291 (3)	0.0442 (3)	−0.0070 (2)	0.0031 (2)	−0.0107 (2)
I6	0.0360 (3)	0.0374 (3)	0.0461 (4)	−0.0137 (3)	0.0014 (3)	−0.0117 (3)

Geometric parameters (Å, º)

Se1A—N2A	1.815 (7)	C7B—H7D	0.9900
Se1A—C11A	1.848 (8)	C8B—C9B	1.536 (13)
N2A—N3A	1.308 (11)	C8B—H8C	0.9900
N3A—C4A	1.350 (11)	C8B—H8D	0.9900
N3A—C12A	1.482 (11)	C9B—C10B	1.528 (12)
C4A—C11A	1.369 (12)	C9B—H9C	0.9900
C4A—C5A	1.495 (12)	C9B—H9D	0.9900
C5A—C6A	1.517 (13)	C10B—C11B	1.511 (10)
C5A—H5A	0.9900	C10B—H10C	0.9900
C5A—H5B	0.9900	C10B—H10D	0.9900
C6A—C7A	1.556 (14)	C12B—H12D	0.9800
C6A—H6A	0.9900	C12B—H12E	0.9800
C6A—H6B	0.9900	C12B—H12F	0.9800
C7A—C8A	1.550 (14)	Se1C—N2C	1.798 (8)
C7A—H7A	0.9900	Se1C—C11C	1.853 (9)
C7A—H7B	0.9900	N2C—N3C	1.315 (10)
C8A—C9A	1.551 (14)	N3C—C4C	1.375 (11)
C8A—H8A	0.9900	N3C—C12C	1.484 (11)
C8A—H8B	0.9900	C4C—C11C	1.382 (13)
C9A—C10A	1.547 (13)	C4C—C5C	1.491 (12)
C9A—H9A	0.9900	C5C—C6C	1.531 (13)
C9A—H9B	0.9900	C5C—H5E	0.9900
C10A—C11A	1.501 (12)	C5C—H5F	0.9900
C10A—H10A	0.9900	C6C—C7C	1.533 (15)
C10A—H10B	0.9900	C6C—H6E	0.9900
C12A—H12A	0.9800	C6C—H6F	0.9900
C12A—H12B	0.9800	C7C—C8C	1.533 (15)
C12A—H12C	0.9800	C7C—H7E	0.9900
Se1B—N2B	1.806 (7)	C7C—H7F	0.9900
Se1B—C11B	1.851 (8)	C8C—C9C	1.558 (15)
N2B—N3B	1.305 (9)	C8C—H8E	0.9900
N3B—C4B	1.382 (10)	C8C—H8F	0.9900
N3B—C12B	1.471 (10)	C9C—C10C	1.527 (15)
C4B—C11B	1.371 (12)	C9C—H9E	0.9900
C4B—C5B	1.486 (11)	C9C—H9F	0.9900
C5B—C6B	1.557 (13)	C10C—C11C	1.487 (12)
C5B—H5C	0.9900	C10C—H10E	0.9900
C5B—H5D	0.9900	C10C—H10F	0.9900
C6B—C7B	1.501 (14)	C12C—H12G	0.9800
C6B—H6C	0.9900	C12C—H12H	0.9800
C6B—H6D	0.9900	C12C—H12I	0.9800
C7B—C8B	1.541 (13)	I1—I2	2.9309 (6)
C7B—H7C	0.9900	I4—I5	2.9351 (7)
N2A—Se1A—C11A	89.1 (4)	C7B—C8B—H8C	108.3
N3A—N2A—Se1A	107.4 (5)	C9B—C8B—H8D	108.3

N2A—N3A—C4A	122.1 (7)	C7B—C8B—H8D	108.3
N2A—N3A—C12A	114.7 (7)	H8C—C8B—H8D	107.4
C4A—N3A—C12A	123.0 (8)	C10B—C9B—C8B	116.0 (8)
N3A—C4A—C11A	112.3 (8)	C10B—C9B—H9C	108.3
N3A—C4A—C5A	122.3 (8)	C8B—C9B—H9C	108.3
C11A—C4A—C5A	125.1 (8)	C10B—C9B—H9D	108.3
C4A—C5A—C6A	114.6 (7)	C8B—C9B—H9D	108.3
C4A—C5A—H5A	108.6	H9C—C9B—H9D	107.4
C6A—C5A—H5A	108.6	C11B—C10B—C9B	114.5 (7)
C4A—C5A—H5B	108.6	C11B—C10B—H10C	108.6
C6A—C5A—H5B	108.6	C9B—C10B—H10C	108.6
H5A—C5A—H5B	107.6	C11B—C10B—H10D	108.6
C5A—C6A—C7A	114.9 (8)	C9B—C10B—H10D	108.6
C5A—C6A—H6A	108.6	H10C—C10B—H10D	107.6
C7A—C6A—H6A	108.6	C4B—C11B—C10B	125.7 (7)
C5A—C6A—H6B	108.6	C4B—C11B—Se1B	108.6 (6)
C7A—C6A—H6B	108.6	C10B—C11B—Se1B	125.7 (6)
H6A—C6A—H6B	107.5	N3B—C12B—H12D	109.5
C8A—C7A—C6A	115.9 (8)	N3B—C12B—H12E	109.5
C8A—C7A—H7A	108.3	H12D—C12B—H12E	109.5
C6A—C7A—H7A	108.3	N3B—C12B—H12F	109.5
C8A—C7A—H7B	108.3	H12D—C12B—H12F	109.5
C6A—C7A—H7B	108.3	H12E—C12B—H12F	109.5
H7A—C7A—H7B	107.4	N2C—Se1C—C11C	89.6 (4)
C7A—C8A—C9A	114.6 (8)	N3C—N2C—Se1C	108.9 (5)
C7A—C8A—H8A	108.6	N2C—N3C—C4C	120.4 (7)
C9A—C8A—H8A	108.6	N2C—N3C—C12C	115.6 (7)
C7A—C8A—H8B	108.6	C4C—N3C—C12C	123.9 (8)
C9A—C8A—H8B	108.6	N3C—C4C—C11C	112.5 (8)
H8A—C8A—H8B	107.6	N3C—C4C—C5C	121.9 (8)
C10A—C9A—C8A	116.5 (8)	C11C—C4C—C5C	125.5 (8)
C10A—C9A—H9A	108.2	C4C—C5C—C6C	111.5 (8)
C8A—C9A—H9A	108.2	C4C—C5C—H5E	109.3
C10A—C9A—H9B	108.2	C6C—C5C—H5E	109.3
C8A—C9A—H9B	108.2	C4C—C5C—H5F	109.3
H9A—C9A—H9B	107.3	C6C—C5C—H5F	109.3
C11A—C10A—C9A	115.7 (7)	H5E—C5C—H5F	108.0
C11A—C10A—H10A	108.4	C5C—C6C—C7C	115.5 (8)
C9A—C10A—H10A	108.4	C5C—C6C—H6E	108.4
C11A—C10A—H10B	108.4	C7C—C6C—H6E	108.4
C9A—C10A—H10B	108.4	C5C—C6C—H6F	108.4
H10A—C10A—H10B	107.4	C7C—C6C—H6F	108.4
C4A—C11A—C10A	125.3 (8)	H6E—C6C—H6F	107.5
C4A—C11A—Se1A	109.0 (6)	C6C—C7C—C8C	113.9 (10)
C10A—C11A—Se1A	125.7 (6)	C6C—C7C—H7E	108.8
N3A—C12A—H12A	109.5	C8C—C7C—H7E	108.8
N3A—C12A—H12B	109.5	C6C—C7C—H7F	108.8
H12A—C12A—H12B	109.5	C8C—C7C—H7F	108.8

N3A—C12A—H12C	109.5	H7E—C7C—H7F	107.7
H12A—C12A—H12C	109.5	C7C—C8C—C9C	115.5 (9)
H12B—C12A—H12C	109.5	C7C—C8C—H8E	108.4
N2B—Se1B—C11B	88.9 (3)	C9C—C8C—H8E	108.4
N3B—N2B—Se1B	110.2 (5)	C7C—C8C—H8F	108.4
N2B—N3B—C4B	118.5 (7)	C9C—C8C—H8F	108.4
N2B—N3B—C12B	117.3 (7)	H8E—C8C—H8F	107.5
C4B—N3B—C12B	124.2 (7)	C10C—C9C—C8C	115.3 (10)
C11B—C4B—N3B	113.8 (7)	C10C—C9C—H9E	108.5
C11B—C4B—C5B	124.4 (7)	C8C—C9C—H9E	108.5
N3B—C4B—C5B	121.6 (7)	C10C—C9C—H9F	108.5
C4B—C5B—C6B	112.5 (7)	C8C—C9C—H9F	108.5
C4B—C5B—H5C	109.1	H9E—C9C—H9F	107.5
C6B—C5B—H5C	109.1	C11C—C10C—C9C	114.5 (8)
C4B—C5B—H5D	109.1	C11C—C10C—H10E	108.6
C6B—C5B—H5D	109.1	C9C—C10C—H10E	108.6
H5C—C5B—H5D	107.8	C11C—C10C—H10F	108.6
C7B—C6B—C5B	115.9 (8)	C9C—C10C—H10F	108.6
C7B—C6B—H6C	108.3	H10E—C10C—H10F	107.6
C5B—C6B—H6C	108.3	C4C—C11C—C10C	126.0 (8)
C7B—C6B—H6D	108.3	C4C—C11C—Se1C	108.7 (6)
C5B—C6B—H6D	108.3	C10C—C11C—Se1C	125.2 (7)
H6C—C6B—H6D	107.4	N3C—C12C—H12G	109.5
C6B—C7B—C8B	116.9 (8)	N3C—C12C—H12H	109.5
C6B—C7B—H7C	108.1	H12G—C12C—H12H	109.5
C8B—C7B—H7C	108.1	N3C—C12C—H12I	109.5
C6B—C7B—H7D	108.1	H12G—C12C—H12I	109.5
C8B—C7B—H7D	108.1	H12H—C12C—H12I	109.5
H7C—C7B—H7D	107.3	$I1^{i}$—I2—I1	180.0
C9B—C8B—C7B	115.7 (7)	I4—I5—$I4^{ii}$	180.0
C9B—C8B—H8C	108.3		

C11A—Se1A—N2A—N3A	−2.1 (6)	C6B—C7B—C8B—C9B	102.4 (10)
Se1A—N2A—N3A—C4A	3.5 (10)	C7B—C8B—C9B—C10B	−74.5 (10)
Se1A—N2A—N3A—C12A	178.2 (6)	C8B—C9B—C10B—C11B	73.3 (9)
N2A—N3A—C4A—C11A	−3.3 (12)	N3B—C4B—C11B—C10B	−179.1 (7)
C12A—N3A—C4A—C11A	−177.5 (8)	C5B—C4B—C11B—C10B	4.7 (13)
N2A—N3A—C4A—C5A	−177.9 (8)	N3B—C4B—C11B—Se1B	−1.3 (9)
C12A—N3A—C4A—C5A	7.9 (13)	C5B—C4B—C11B—Se1B	−177.4 (7)
N3A—C4A—C5A—C6A	87.2 (10)	C9B—C10B—C11B—C4B	−81.2 (10)
C11A—C4A—C5A—C6A	−86.7 (11)	C9B—C10B—C11B—Se1B	101.4 (8)
C4A—C5A—C6A—C7A	50.6 (11)	N2B—Se1B—C11B—C4B	1.0 (6)
C5A—C6A—C7A—C8A	53.8 (11)	N2B—Se1B—C11B—C10B	178.8 (7)
C6A—C7A—C8A—C9A	−103.0 (10)	C11C—Se1C—N2C—N3C	−1.1 (7)
C7A—C8A—C9A—C10A	72.2 (11)	Se1C—N2C—N3C—C4C	1.7 (10)
C8A—C9A—C10A—C11A	−72.0 (11)	Se1C—N2C—N3C—C12C	−173.9 (7)
N3A—C4A—C11A—C10A	179.2 (8)	N2C—N3C—C4C—C11C	−1.4 (12)
C5A—C4A—C11A—C10A	−6.4 (14)	C12C—N3C—C4C—C11C	173.8 (9)

N3A—C4A—C11A—Se1A	1.2 (9)	N2C—N3C—C4C—C5C	−178.3 (8)
C5A—C4A—C11A—Se1A	175.6 (7)	C12C—N3C—C4C—C5C	−3.1 (13)
C9A—C10A—C11A—C4A	81.1 (11)	N3C—C4C—C5C—C6C	86.5 (11)
C9A—C10A—C11A—Se1A	−101.2 (9)	C11C—C4C—C5C—C6C	−90.0 (11)
N2A—Se1A—C11A—C4A	0.5 (6)	C4C—C5C—C6C—C7C	49.6 (11)
N2A—Se1A—C11A—C10A	−177.5 (7)	C5C—C6C—C7C—C8C	53.0 (12)
C11B—Se1B—N2B—N3B	−0.4 (6)	C6C—C7C—C8C—C9C	−105.9 (12)
Se1B—N2B—N3B—C4B	−0.3 (9)	C7C—C8C—C9C—C10C	75.8 (13)
Se1B—N2B—N3B—C12B	−179.0 (6)	C8C—C9C—C10C—C11C	−71.6 (12)
N2B—N3B—C4B—C11B	1.1 (11)	N3C—C4C—C11C—C10C	−178.0 (9)
C12B—N3B—C4B—C11B	179.7 (8)	C5C—C4C—C11C—C10C	−1.2 (15)
N2B—N3B—C4B—C5B	177.3 (7)	N3C—C4C—C11C—Se1C	0.4 (9)
C12B—N3B—C4B—C5B	−4.0 (12)	C5C—C4C—C11C—Se1C	177.2 (7)
C11B—C4B—C5B—C6B	87.3 (10)	C9C—C10C—C11C—C4C	78.5 (13)
N3B—C4B—C5B—C6B	−88.5 (10)	C9C—C10C—C11C—Se1C	−99.6 (10)
C4B—C5B—C6B—C7B	−52.3 (11)	N2C—Se1C—C11C—C4C	0.4 (7)
C5B—C6B—C7B—C8B	−50.7 (11)	N2C—Se1C—C11C—C10C	178.7 (9)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y, -z+1$.