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1,3-Bis[2-(dimethylaminomethyl)-phenyl]triselenide

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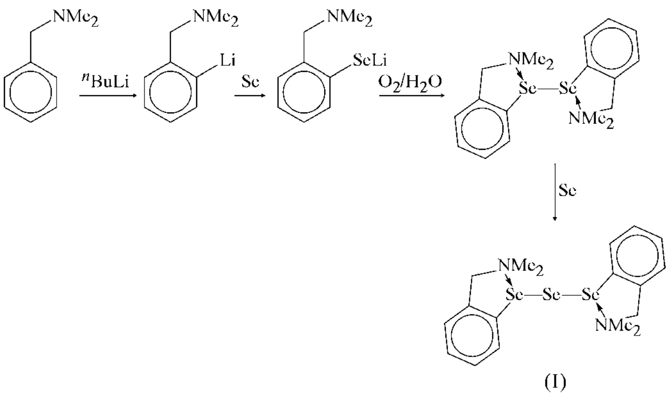
The title compound, $C_{18}H_{24}N_2Se_3$, consists of discrete molecules; owing to the presence of strong intramolecular N···Se interactions [N···Se = 2.671 (4) and 2.873 (4) Å], the chalcogen Se atoms of the angular Se_3 chain exhibit different coordination geometries, *i.e.* the terminal Se atoms are tricoordinated and exhibit a T-shaped environment of the $CNSe_2$ core [N···Se—Se = 173.73 (9) and 172.29 (9)°], while the central Se atom is dicoordinated to the other two Se atoms, with an Se—Se—Se angle of 108.32 (2)°.

Comment

Our research interests have focused on the synthesis, structural characterization and chemical reactivity of new *R*Se and *R*Te derivatives containing aryl groups with pendant arms, *e.g.* 2-$(Me_2NCH_2)C_6H_4$–, 2-$[O(CH_2CH_2)_2NCH_2]C_6H_4$– and 2-$[MeN(CH_2CH_2)_2NCH_2]C_6H_4$– groups (Drake *et al.*, 2001*a*,*b*; Deleanu *et al.*, 2002; Kulcsar *et al.*, 2005, 2007). Both organic groups attached to the Se atoms form intramolecular N···Se interactions, resulting in an increase in the coordination number at atoms Se1 and Se3 to three (Fig. 1). Some diorganotriselenides (R_2Se_3) of the type *R*—Se—Se—Se—*R* [*R* = ^{t}Bu$_2$CH (McKinnon *et al.*, 1978), 2,4-$Cl_2C_6H_3$ (Hansen *et al.*, 1989), $(Me_3Si)_3C$ (Jones & Jeske, 2004), 2-(2-phenyl-5,6-dihydro-4*H*-1,3-oxazinyl) (Kumar *et al.*, 2004), 4-(phenylamino)quinazolin-2-yl (Atanassov *et al.*, 2004), 2,6-(2,4,6-^{i}Pr$_3$C$_6$H$_2$)$_2$C$_6$H$_3$ (Krumm *et al.*, 2005), $(Me_2PhSi)_3C$ (Klapötke *et al.*, 2006) and $(Me_2PhSi)_2ClC$ (Klapötke *et al.*, 2007)] have already been reported. We report here the structure of the title compound, (I).



(I)

In (I), the coordination geometry around the central Se atom is pseudo-tetrahedral [Se1—Se2—Se3 = 108.32 (3)°], with two positions occupied by the two lone pairs of electrons, while the other two chalcogen Se atoms have distorted T-shaped coordination geometries [N1···Se1—Se2 = 173.73 (9)° and Se2—Se3···N2 = 172.29 (9)°]. The two N···Se distances are considerably different (Table 1) and larger than the sum of the covalent radii [r_{cov}(Se,N) = 1.87 Å], but significantly shorter than the sum of the corresponding van der Waals radii [r_{vdW}(Se,N) = 3.54 Å] (Emsley, 1994). These distances are in the range of 2.61–2.97 Å observed for intramolecular N···Se interactions in R_2Se_2 (*e.g.* bis[(2-dimethylaminomethyl)phenyl]diselenide (Kaur *et al.*, 1996), [5-Me-2-(4-MeC$_6$H$_4$N=N)C$_6$H$_3$]$_2$Se$_2$ (Jones *et al.*, 1996), bis[8-(dimethylamino)-1-naphthyl]diselenide (Mugesh *et al.*, 2001, 2002), bis[2-(4,4-dimethyl-2-oxazolinyl)phenyl]diselenide, bis-[2-(4-ethyl-2-oxazolinyl)phenyl]diselenide (Mugesh *et al.*, 1998, 2001), bis{2-[(4*S*,5*S*)-4-hydroxymethyl-5-phenyloxazolin-2-yl]phenyl}diselenide (Miyake *et al.*, 2002), bis[3-(4,5-dihydro-4,4-dimethyl-1,3-oxazol-2-yl)-4-(3,5-dimethylphenyl)-2-naphthyl]diselenide (Kandasamy *et al.*, 2004), bis[2-(morpholin-1-ylmethyl)phenyl]diselenide and bis[2-(4-methylpiperazin-1-ylmethyl)phenyl]diselenide (Kulcsar *et al.*, 2007)). However, they are longer than those in the known R_2Se_3 [*R* = 2-(2-phenyl-5,6-dihydro-4*H*-1,3-oxazinyl)] derivative [N···Se = 2.562 (18) and 2.569 (14) Å; Kumar *et al.*, 2004]. There are two additional C—H···Se2 intramolecular contacts (Table 2), which are *ca* 0.3° shorter than the sum of the H/Se van der Waals radii (Fig. 2*a*).

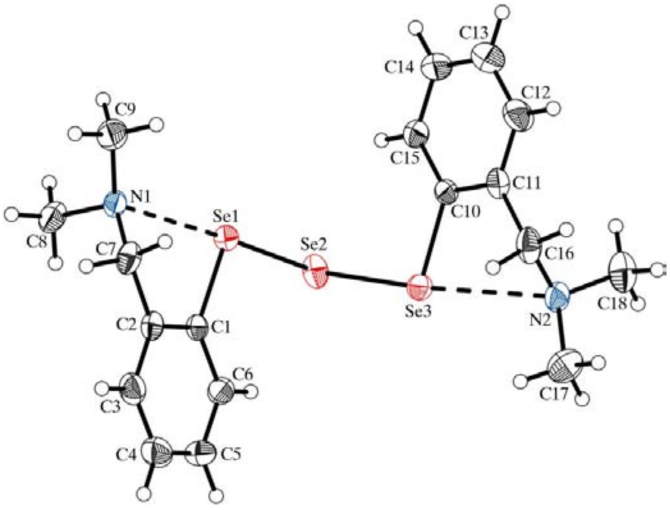


Figure 1
A view of (I), showing the atomic numbering scheme and displacement ellipsoids at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

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In spite of the asymmetry in the Se⋯N distances, the Se—Se bond lengths in (I) are only slightly different (Table 1) and fall in the range found for R_2Se_2 with aromatic groups with pendant arms, *i.e.* 2.32–2.38 Å, or the previously mentioned diorganotrichalcogenides R_2Se_3.

The two (C,N)SeSe cores are distorted as a result of the small bite of the organic ligand [C1—Se1⋯N1 = 76.58 (14)° and C10—Se3⋯N2 = 74.12 (15)°], the values diminished at the expense of the enlarged C—Se—Se angles [C1—Se1—Se2 = 102.65 (12)° and C10—Se3—Se2 = 102.73 (12)°]. The benzene rings are twisted with a C1—Se1—Se3—C10 torsion angle of −143.65 (18)°. The Se—Se—Se angle [108.32 (3)°] is in the range for reported triselenides [99.68 (6)–111.16 (3)°]. The conformation of the C—Se—Se—C skeleton in diorganodichalcogenides can be discussed in terms of '*cisoid*' (C—Se—Se—C < 90°) and '*transoid*' conformations (C—Se—Se—C > 90°). For the reported R_2Se_2, both conformations were found, *i.e.* the C—Se—Se—C torsion angle is 80.9° in [2-{O-$(CH_2CH_2)_2NCH_2$}C_6H_4]$_2$Se$_2$ (*cisoid*) and 114.0° in [2-{MeN-$(CH_2CH_2)_2NCH_2$}C_6H_4]$_2$Se$_2$ (*transoid*) (Kulcsar *et al.*, 2007). In (I), an overall *transoid* conformation can be assumed from C1—Se1—Se3—C10.

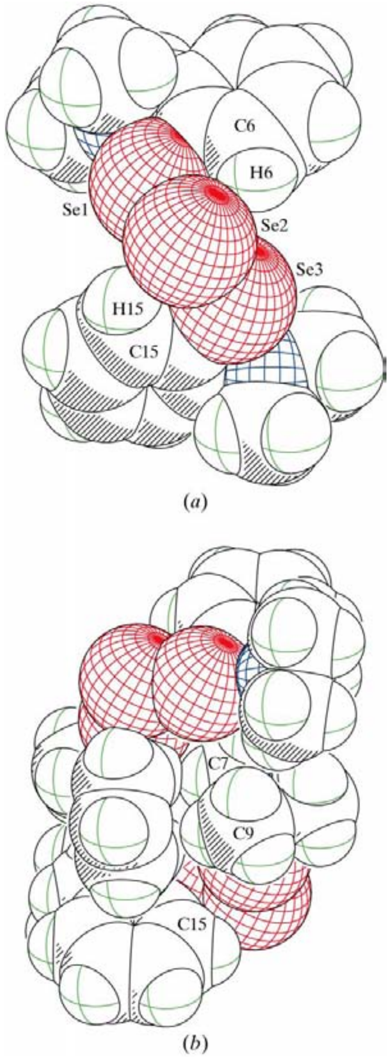

Figure 2
(*a*) A view of the two intramolecular C—H⋯Se2 contacts in (I). (*b*) A view of the intra- and intermolecular contacts between a pair of molecules in (I) (with atoms drawn as their van der Waals spheres).

The intramolecular N⋯Se interactions induce planar chirality in the molecule, since the SeC_3N rings are not planar but folded along the Se1⋯$C_{methylene}$ axis, with the N atom lying out of the mean plane defined by the residual SeC_3 system. Similar behavior was observed in related *R*Se systems (Kulcsar *et al.*, 2005, 2007). As a consequence, the compound crystallizes as a racemate, *i.e.* a mixture of $R_{N1}R_{N2}$ and $S_{N1}S_{N2}$ isomers [with the aromatic ring and the N atom as chiral plane and pivot atom, respectively (IUPAC, 1979)].

As with the related [2-$(Me_2NCH_2)C_6H_4$]$_2$Se$_2$ derivative, no short intermolecular interactions were found in (I). The structure of the analogous R_2Te_2 compound, [2-$(Me_2NCH_2)C_6H_4$]$_2$Te$_2$, contains discrete dimer associations of pairs of $R_{N1}S_{N2}$- and $S_{N1}R_{N2}$-[2-$(Me_2NCH_2)C_6H_4$]$_2$Te$_2$ isomers built through π—Te⋯Ph interactions established between atom Te2 of one molecule and the aromatic ring attached to atom Te1 of the other molecule [Te2⋯$Ph_{centroid}$ = 3.750 (1) Å; Drake *et al.*, 2001*a*].

Experimental

A few crystals of (I) were isolated as a by-product in the synthesis of [2-$(Me_2NCH_2)C_6H_4$]$_2$Se$_2$, as a result of insertion of some residual elemental Se into the Se—Se bond of the major product [2-$(Me_2NCH_2)C_6H_4$]$_2$Se$_2$ (see reaction scheme in *Comment*). Single crystals suitable for X-ray diffraction were obtained from a $CHCl_3$/*n*-hexane mixture.

Crystal data

$C_{18}H_{24}N_2Se_3$
M_r = 505.27
Monoclinic, $P2_1/c$
a = 13.5307 (12) Å
b = 9.8246 (9) Å
c = 16.0701 (14) Å
β = 111.013 (2)°
V = 1994.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.53 mm^{-1}
T = 297 (2) K
0.24 × 0.17 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SHELXTL*; Bruker, 2001)
T_{min} = 0.324, T_{max} = 0.515
18693 measured reflections
3509 independent reflections
2961 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.084
S = 1.14
3509 reflections
212 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected bond lengths (Å).

C1—Se1	1.946 (4)	Se2—Se3	2.3372 (7)
C10—Se3	1.935 (4)	Se1—N1	2.671 (4)
Se1—Se2	2.3545 (6)	Se3—N2	2.873 (4)

Table 2
Short intermolecular contact geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C6—H6⋯Se2	0.93	2.79	3.392 (4)	123
C15—H15⋯Se2	0.93	2.82	3.404 (5)	122

All H atoms were placed in calculated positions and refined using a riding model [C—H = 0.93–0.97 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl or 1.2$U_{\rm eq}$(C) for aryl H atoms]. The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3121). Services for accessing these data are described at the back of the journal.

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