

2,2'-(Diselane-1,2-diyl)bis(*N,N*-dimethylnicotinamide)

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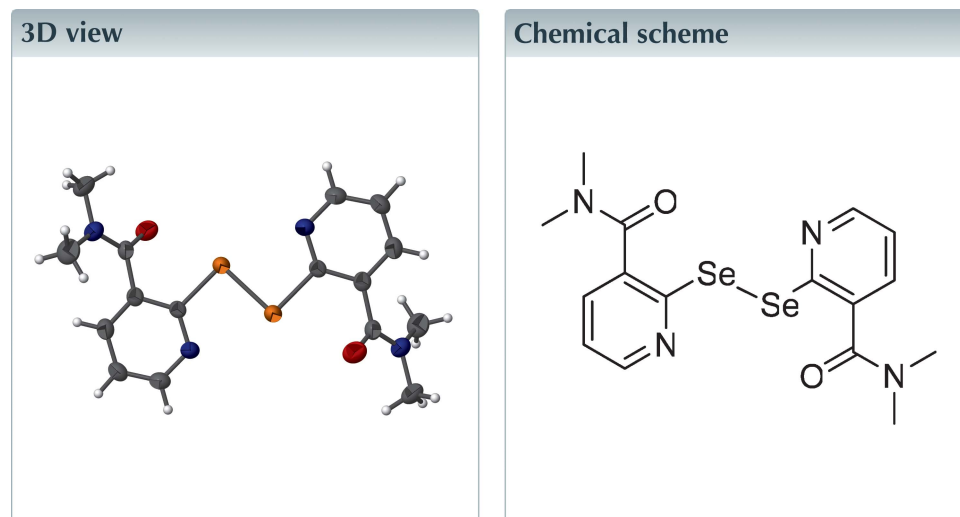
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Structural data: full structural data are available from iucrdata.iucr.org

The title compound, C₁₆H₁₈N₄O₂Se₂, is centrosymmetric. The dihedral angle between the pyridine ring and the amide side chain is 56.20 (16)°. In the crystal, a weak C—H···O interaction links the molecules into [010] chains.



Structure description

Many investigations have demonstrated that organoselenium compounds are less toxic than those of inorganic selenium compounds (Jalbout *et al.* 2008). However, to gain further insight into the role of organoselenium compounds, detailed studies are still needed. As part of our research in this area, we report herein the synthesis and crystal structure of the title compound (Fig. 1).

The complete molecule is generated by a crystallographic centre of symmetry at the mid-point of the Se—Se bond. This implies, of course, that the C—Se—Se—C torsion angle is 180°, which minimizes repulsion of the Se lone pairs, and the dihedral angle between the pyridine rings is 0°. The pyridine ring is substituted at the 2-position [C1—Se = 1.923 (3) Å] and the 3-position [C2—C6 = 1.490 (4) Å]. The X—C—Se—Se torsion angles (X = C, N) are 14.01 (2) and -164.79 (2)°, respectively, indicating that the Se—Se bond lies close to the plane of each pyridine ring. In the crystal, weak C—H···O bonds (Table 1) link the molecules into [010] chains.

Synthesis and crystallization

The title compound was prepared follows a modified literature procedure (Feng *et al.*, 2010). To a vigorously stirred solution of selenium powder (1.00 g, 12.6 mmol) and absolute ethanol (30 ml), sodium borohydride (0.35 g, 9.3 mmol) was added at 0°C. The mixture was warmed to room temperature and stirred for 2 h. 2-Chloro-*N,N*-dimethyl-

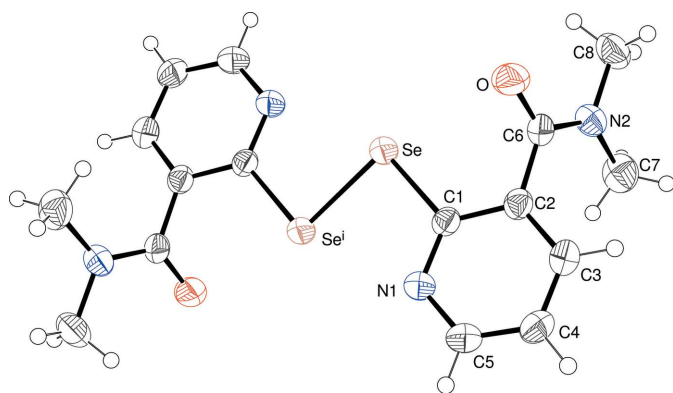


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x + 1, -y + 1, -z + 2.$]

nicotinamide (1.55 g, 8.4 mmol) was added and stirred for 7 d. O_2 was passed through the solution slowly for 2 h after the reaction mixture was acidified by glacial acetic acid. The solvents were removed *in vacuo* and the residue was extracted with dichloromethane (CH_2Cl_2) and filtered. The filtrate was evaporated *in vacuo*. The precipitate was separated by filtration and recrystallized from $C_6H_{12}-CHCl_3$ (1:2) mixed solvent to give the product as colorless block-shaped crystals, yield: 1.0 g, 52%.

1H NMR (300 MHz, $DCCl_3$) δ (p.p.m.): 3.07 (s, 12H, Me), 7.10 (q, 2H, ArH), 7.44 (dd, 2H, ArH), 8.41 (dd, 2H, ArH). ^{13}C NMR (75 MHz, $DCCl_3$) δ (p.p.m.): 40.77 (Me), 41.69 (Me), 123.71, 135.47, 136.92, 153.18, 154.68, 171.27(C=O). ^{77}Se NMR (57 MHz, $DCCl_3$) δ (p.p.m.): 486.10. Analysis calculated for $C_{16}H_{18}N_4O_2Se_2$: C: 42.12, H: 3.98, N: 12.28; found: C:41.74, H: 3.957, N: 12.03.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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Table 1
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots O^i$	0.93	2.52	3.431 (4)	165

Symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{18}N_4O_2Se_2$
M_r	456.26
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (\AA)	7.167 (2), 8.726 (3), 13.947 (4)
β ($^\circ$)	96.375 (4)
V (\AA^3)	866.8 (4)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.28
Crystal size (mm)	0.50 \times 0.20 \times 0.20
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (SADABS; Bruker, 2004)
T_{\min}, T_{\max}	0.223, 0.481
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3376, 1524, 1300
R_{int}	0.035
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.030, 0.077, 1.06
No. of reflections	1524
No. of parameters	111
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ($e \text{\AA}^{-3}$)	0.64, -0.43

Computer programs: APEX2 and SAINT (Bruker, 2004), SHELXS97 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015) and ORTEP-3 for Windows (Farrugia, 2012).

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full crystallographic data

IUCrData (2017). **2**, x170882 [<https://doi.org/10.1107/S2414314617008823>]

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2,2'-(Diselane-1,2-diyl)bis(*N,N*-dimethylnicotinamide)*Crystal data*

$C_{16}H_{18}N_4O_2Se_2$	$F(000) = 452$
$M_r = 456.26$	$D_x = 1.748 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 7.167 (2) \text{ \AA}$	Cell parameters from 2532 reflections
$b = 8.726 (3) \text{ \AA}$	$\theta = 2.8\text{--}27.5^\circ$
$c = 13.947 (4) \text{ \AA}$	$\mu = 4.28 \text{ mm}^{-1}$
$\beta = 96.375 (4)^\circ$	$T = 293 \text{ K}$
$V = 866.8 (4) \text{ \AA}^3$	Block, colorless
$Z = 2$	$0.50 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker APEXII CCD diffractometer	1524 independent reflections
phi and ω scans	1300 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$R_{\text{int}} = 0.035$
$T_{\text{min}} = 0.223$, $T_{\text{max}} = 0.481$	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.8^\circ$
3376 measured reflections	$h = -8 \rightarrow 6$
	$k = -8 \rightarrow 10$
	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.0053P]$
$wR(F^2) = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1524 reflections	$\Delta\rho_{\text{max}} = 0.64 \text{ e \AA}^{-3}$
111 parameters	$\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$
0 restraints	

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.

Refinement. All H atoms were positioned geometrically ($C-H = 0.93\text{--}0.96 \text{ \AA}$), and refined as riding with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}$ of the adjacent carbon atom ($1.5U_{\text{eq}}$ for methyl H atoms).

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se	0.52041 (4)	0.56001 (3)	0.92626 (2)	0.04189 (16)
N1	0.1797 (4)	0.6437 (3)	0.99097 (18)	0.0386 (6)
N2	0.4942 (4)	0.9794 (3)	0.79999 (18)	0.0403 (6)
O	0.4595 (4)	0.7382 (3)	0.74625 (17)	0.0541 (7)
C1	0.2981 (4)	0.6827 (3)	0.9288 (2)	0.0313 (6)
C2	0.2725 (4)	0.8092 (3)	0.8665 (2)	0.0341 (7)
C3	0.1072 (5)	0.8902 (4)	0.8669 (2)	0.0423 (8)
H3	0.0823	0.9734	0.8257	0.051*
C4	-0.0211 (5)	0.8469 (4)	0.9289 (2)	0.0450 (8)
H4	-0.1344	0.8987	0.9289	0.054*
C5	0.0220 (4)	0.7258 (4)	0.9903 (2)	0.0433 (8)
H5	-0.0627	0.6996	1.0335	0.052*
C6	0.4161 (4)	0.8408 (3)	0.7996 (2)	0.0349 (7)
C7	0.4669 (5)	1.1022 (4)	0.8681 (3)	0.0546 (9)
H7A	0.3853	1.1787	0.8368	0.082*
H7B	0.5860	1.1477	0.8902	0.082*
H7C	0.4116	1.0610	0.9222	0.082*
C8	0.6280 (5)	1.0111 (4)	0.7320 (3)	0.0535 (9)
H8A	0.7067	0.9231	0.7268	0.080*
H8B	0.7042	1.0973	0.7541	0.080*
H8C	0.5619	1.0339	0.6699	0.080*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se	0.0395 (2)	0.0410 (2)	0.0480 (2)	0.01035 (14)	0.01743 (16)	0.01223 (14)
N1	0.0347 (14)	0.0393 (15)	0.0434 (14)	0.0022 (12)	0.0121 (12)	0.0045 (11)
N2	0.0425 (16)	0.0382 (15)	0.0405 (14)	-0.0051 (12)	0.0072 (12)	0.0041 (11)
O	0.0702 (17)	0.0403 (13)	0.0569 (14)	-0.0031 (12)	0.0293 (13)	-0.0065 (11)
C1	0.0288 (15)	0.0274 (15)	0.0377 (15)	0.0003 (12)	0.0044 (13)	-0.0021 (12)
C2	0.0380 (17)	0.0283 (16)	0.0358 (15)	0.0008 (13)	0.0039 (13)	-0.0018 (12)
C3	0.046 (2)	0.0345 (17)	0.0469 (19)	0.0062 (15)	0.0049 (15)	0.0037 (14)
C4	0.0374 (19)	0.0432 (19)	0.055 (2)	0.0098 (15)	0.0097 (16)	0.0010 (15)
C5	0.0362 (18)	0.0479 (19)	0.0479 (18)	0.0017 (15)	0.0139 (15)	0.0012 (15)
C6	0.0378 (17)	0.0301 (17)	0.0365 (16)	0.0040 (13)	0.0028 (13)	0.0045 (13)
C7	0.064 (3)	0.0413 (19)	0.058 (2)	-0.0088 (18)	0.0045 (18)	-0.0050 (16)
C8	0.052 (2)	0.055 (2)	0.055 (2)	-0.0063 (18)	0.0144 (18)	0.0171 (17)

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

Se—C1	1.923 (3)	C3—C4	1.383 (4)
Se—Se ⁱ	2.3551 (8)	C3—H3	0.9300
N1—C1	1.324 (4)	C4—C5	1.373 (4)
N1—C5	1.338 (4)	C4—H4	0.9300
N2—C6	1.332 (4)	C5—H5	0.9300

N2—C8	1.449 (4)	C7—H7A	0.9600
N2—C7	1.460 (4)	C7—H7B	0.9600
O—C6	1.226 (3)	C7—H7C	0.9600
C1—C2	1.403 (4)	C8—H8A	0.9600
C2—C3	1.381 (4)	C8—H8B	0.9600
C2—C6	1.490 (4)	C8—H8C	0.9600
C1—Se—Se ⁱ	92.77 (8)	N1—C5—H5	118.4
C1—N1—C5	117.4 (3)	C4—C5—H5	118.4
C6—N2—C8	118.6 (3)	O—C6—N2	122.0 (3)
C6—N2—C7	125.6 (3)	O—C6—C2	118.9 (3)
C8—N2—C7	115.6 (3)	N2—C6—C2	119.0 (3)
N1—C1—C2	124.0 (3)	N2—C7—H7A	109.5
N1—C1—Se	117.5 (2)	N2—C7—H7B	109.5
C2—C1—Se	118.6 (2)	H7A—C7—H7B	109.5
C3—C2—C1	117.0 (3)	N2—C7—H7C	109.5
C3—C2—C6	124.1 (3)	H7A—C7—H7C	109.5
C1—C2—C6	118.8 (3)	H7B—C7—H7C	109.5
C2—C3—C4	119.5 (3)	N2—C8—H8A	109.5
C2—C3—H3	120.2	N2—C8—H8B	109.5
C4—C3—H3	120.2	H8A—C8—H8B	109.5
C5—C4—C3	118.8 (3)	N2—C8—H8C	109.5
C5—C4—H4	120.6	H8A—C8—H8C	109.5
C3—C4—H4	120.6	H8B—C8—H8C	109.5
N1—C5—C4	123.2 (3)		
C5—N1—C1—C2	3.6 (4)	C3—C4—C5—N1	-2.4 (5)
C5—N1—C1—Se	-177.7 (2)	C8—N2—C6—O	2.9 (5)
N1—C1—C2—C3	-4.4 (4)	C7—N2—C6—O	-172.7 (3)
Se—C1—C2—C3	176.9 (2)	C8—N2—C6—C2	-177.5 (3)
N1—C1—C2—C6	-179.9 (3)	C7—N2—C6—C2	6.9 (5)
Se—C1—C2—C6	1.4 (4)	C3—C2—C6—O	-121.7 (3)
C1—C2—C3—C4	1.6 (4)	C1—C2—C6—O	53.5 (4)
C6—C2—C3—C4	176.9 (3)	C3—C2—C6—N2	58.7 (4)
C2—C3—C4—C5	1.5 (5)	C1—C2—C6—N2	-126.1 (3)
C1—N1—C5—C4	-0.1 (5)		

Symmetry code: (i) $-x+1, -y+1, -z+2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots O ⁱⁱ	0.93	2.52	3.431 (4)	165

Symmetry code: (ii) $-x+1/2, y+1/2, -z+3/2$.