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Bis(4-nitrophenyl) selenide

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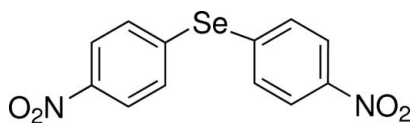
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Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.030; wR factor = 0.074; data-to-parameter ratio = 17.9.

In the title compound, $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_4\text{Se}$, the Se atom is situated on a twofold rotational axis, so the asymmetric unit contains one half-molecule. In the molecule, the C–Se–C angle is $99.48(13)^\circ$, the two benzene rings are inclined to each other at an angle of $63.8(1)^\circ$ and the nitro group is twisted by $15.9(1)^\circ$ from the attached benzene ring. In the crystal, molecules are held together through weak C–H...O interactions, forming a three-dimensional network.

Related literature

For applications of organoselenium compounds, see: Mugesh *et al.* (2001); Nogueira *et al.* (2004); Wirth (1999). For details of the synthesis, see: Taniguchi (2005). The crystal structures of the related compounds bis(*p*-tolyl) selenide and bis(4-acetylphenyl) selenide were reported by Blackmore & Abrahams (1955) and Bouraoui *et al.* (2011), respectively.



Experimental

Crystal data

$\text{C}_{12}\text{H}_8\text{N}_2\text{O}_4\text{Se}$

$M_r = 323.16$

Monoclinic, $C2/c$
 $a = 7.207(4)$ Å
 $b = 14.176(7)$ Å
 $c = 11.686(5)$ Å
 $\beta = 101.870(7)^\circ$
 $V = 1168.3(9)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 3.23$ mm⁻¹
 $T = 153$ K
 $0.47 \times 0.34 \times 0.34$ mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
 $T_{\min} = 0.314$, $T_{\max} = 0.402$

4848 measured reflections
1557 independent reflections
1298 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.074$
 $S = 1.00$
1557 reflections

87 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.70$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.50$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2}\cdots\text{O2}^{\text{i}}$	0.95	2.51	3.426 (3)	162
$\text{C6}-\text{H6}\cdots\text{O2}^{\text{ii}}$	0.95	2.50	3.427 (3)	164

Symmetry codes: (i) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $x + \frac{1}{2}, -y - \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5389).

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supporting information

Acta Cryst. (2013). E69, o636 [https://doi.org/10.1107/S1600536813007526]

Bis(4-nitrophenyl) selenide**Zong-Le Zuo****S1. Comment**

During the past decade, organoselenium compounds have emerged their potential as drug candidates (Nogueira *et al.*, 2004; Mugesh *et al.*, 2001). Organoselenium compounds also exert catalytic role in organic synthesis (Wirth, 1999). Herewith we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), each Se atom is situated on a twofold rotational axis, so asymmetric part contains a half of the molecule. The C—Se—C angle is 99.48 (13)°, that is similar to 99.58 (13)° observed in bis(4-acetylphenyl) selenide (Bouraoui *et al.*, 2011), but different from 106.2 (1)° found in bis(*p*-tolyl) selenide (Blackmore & Abrahams, 1955). Two benzene rings in (I) are inclined to each other at 63.8 (1)° and each nitro group is twisted at 15.9 (1)° from the attached benzene ring. The crystal packing is stabilized by the weak C—H···O hydrogen bonds (Table 1).

S2. Experimental

The title compound has been synthesized following the procedure proposed by Taniguchi (2005). 1-Iodo-4-nitrobenzene (1.0 mmol), selenium (1.2 mmol), cuprous oxide (0.1 mmol), aluminium (2 mmol), magnesium chloride (0.5 mmol), acetylacetone (0.3 mmol), TBAF (0.2 mmol) and DMF/water (3:1, 1.0 ml) were put into a Teflon septum screw-capped tube and then sealed in the air. The reaction mixture was stirred at 120 °C for 36 h, then cooled to room temperature. Subsequently, the resulting mixture was diluted with ethyl acetate and water, and the combined organic extracts were dried with sodium sulfate anhydrous. After the solvent being removed under reduced pressure, the residue was purified by silica-gel column chromatography to afford the corresponding product. Yellow single crystals suitable for X-ray diffraction were obtained by recrystallization from acetone.

S3. Refinement

H atoms were placed in calculated positions, with C—H = 0.95 Å, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

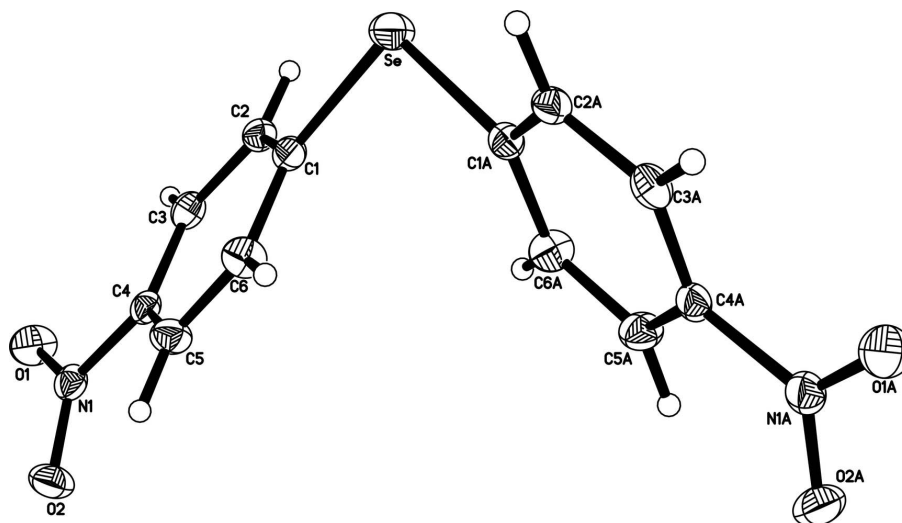


Figure 1

The molecular structure of (I) showing 50% probability displacement ellipsoids and the atomic numbering [symmetry code: (A) $-x, y, 3/2-z$].

Bis(4-nitrophenyl) selenide

Crystal data

$C_{12}H_8N_2O_4Se$

$M_r = 323.16$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 7.207\ (4)\ \text{\AA}$

$b = 14.176\ (7)\ \text{\AA}$

$c = 11.686\ (5)\ \text{\AA}$

$\beta = 101.870\ (7)^\circ$

$V = 1168.3\ (9)\ \text{\AA}^3$

$Z = 4$

$F(000) = 640$

$D_x = 1.837\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1957 reflections

$\theta = 2.9\text{--}29.1^\circ$

$\mu = 3.23\ \text{mm}^{-1}$

$T = 153\ \text{K}$

Block, yellow

$0.47 \times 0.34 \times 0.34\ \text{mm}$

Data collection

Rigaku AFC10/Saturn724+
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $28.5714\ \text{pixels mm}^{-1}$

phi and ω scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2008)

$T_{\min} = 0.314$, $T_{\max} = 0.402$

4848 measured reflections

1557 independent reflections

1298 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 29.1^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -9 \rightarrow 9$

$k = -19 \rightarrow 16$

$l = -15 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.074$

$S = 1.00$

1557 reflections

87 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
 $w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.160P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.70 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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	0.0000	0.05282 (2)	0.7500	0.02293 (12)
O1	-0.6302 (3)	-0.18522 (11)	0.34112 (15)	0.0281 (4)
O2	-0.5748 (2)	-0.29360 (11)	0.47417 (15)	0.0251 (4)
N1	-0.5486 (3)	-0.21464 (13)	0.43644 (16)	0.0182 (4)
C1	-0.1638 (3)	-0.03449 (14)	0.65264 (19)	0.0162 (4)
C2	-0.2197 (3)	-0.01493 (15)	0.53474 (19)	0.0166 (4)
H2	-0.1711	0.0392	0.5030	0.020*
C3	-0.3464 (3)	-0.07375 (15)	0.4622 (2)	0.0176 (5)
H3	-0.3873	-0.0601	0.3813	0.021*
C4	-0.4114 (3)	-0.15292 (14)	0.51142 (19)	0.0157 (4)
C5	-0.3547 (3)	-0.17464 (16)	0.6281 (2)	0.0195 (5)
H5	-0.3994	-0.2303	0.6588	0.023*
C6	-0.2320 (3)	-0.11472 (15)	0.70005 (19)	0.0198 (5)
H6	-0.1942	-0.1279	0.7812	0.024*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se	0.02479 (19)	0.01636 (18)	0.0236 (2)	0.000	-0.00441 (13)	0.000
O1	0.0308 (10)	0.0249 (9)	0.0228 (9)	0.0009 (7)	-0.0082 (7)	-0.0017 (7)
O2	0.0284 (10)	0.0164 (8)	0.0296 (10)	-0.0054 (7)	0.0037 (8)	-0.0012 (7)
N1	0.0158 (10)	0.0187 (10)	0.0197 (10)	0.0021 (7)	0.0024 (8)	-0.0048 (7)
C1	0.0145 (10)	0.0153 (11)	0.0180 (11)	0.0013 (8)	0.0016 (8)	-0.0020 (8)
C2	0.0163 (10)	0.0147 (10)	0.0191 (12)	0.0014 (8)	0.0039 (9)	0.0038 (8)
C3	0.0174 (11)	0.0205 (12)	0.0142 (11)	0.0038 (8)	0.0016 (8)	0.0019 (8)
C4	0.0130 (10)	0.0154 (11)	0.0182 (11)	0.0023 (8)	0.0016 (8)	-0.0045 (8)
C5	0.0223 (12)	0.0160 (11)	0.0199 (12)	-0.0010 (9)	0.0038 (9)	0.0031 (8)
C6	0.0245 (12)	0.0205 (12)	0.0134 (11)	-0.0014 (9)	0.0018 (9)	0.0026 (8)

Geometric parameters (Å, °)

Se—C1	1.915 (2)	C2—H2	0.9500
Se—C1 ⁱ	1.915 (2)	C3—C4	1.386 (3)
O1—N1	1.221 (2)	C3—H3	0.9500
O2—N1	1.232 (2)	C4—C5	1.375 (3)
N1—C4	1.468 (3)	C5—C6	1.380 (3)
C1—C2	1.382 (3)	C5—H5	0.9500
C1—C6	1.398 (3)	C6—H6	0.9500
C2—C3	1.389 (3)		
C1—Se—C1 ⁱ	99.48 (13)	C4—C3—H3	121.0
O1—N1—O2	123.79 (19)	C2—C3—H3	121.0
O1—N1—C4	118.68 (18)	C5—C4—C3	122.4 (2)
O2—N1—C4	117.53 (18)	C5—C4—N1	118.99 (19)
C2—C1—C6	120.3 (2)	C3—C4—N1	118.59 (19)
C2—C1—Se	118.71 (16)	C4—C5—C6	119.3 (2)
C6—C1—Se	120.96 (17)	C4—C5—H5	120.4
C1—C2—C3	120.6 (2)	C6—C5—H5	120.4
C1—C2—H2	119.7	C5—C6—C1	119.5 (2)
C3—C2—H2	119.7	C5—C6—H6	120.2
C4—C3—C2	117.9 (2)	C1—C6—H6	120.2
C1 ⁱ —Se—C1—C2	-139.8 (2)	O2—N1—C4—C5	-15.9 (3)
C1 ⁱ —Se—C1—C6	42.66 (16)	O1—N1—C4—C3	-14.9 (3)
C6—C1—C2—C3	1.0 (3)	O2—N1—C4—C3	165.32 (19)
Se—C1—C2—C3	-176.55 (16)	C3—C4—C5—C6	1.5 (3)
C1—C2—C3—C4	-1.2 (3)	N1—C4—C5—C6	-177.24 (19)
C2—C3—C4—C5	-0.1 (3)	C4—C5—C6—C1	-1.7 (3)
C2—C3—C4—N1	178.67 (18)	C2—C1—C6—C5	0.4 (3)
O1—N1—C4—C5	163.9 (2)	Se—C1—C6—C5	177.94 (17)

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

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots O2 ⁱⁱ	0.95	2.51	3.426 (3)	162
C6—H6 \cdots O2 ⁱⁱⁱ	0.95	2.50	3.427 (3)	164

Symmetry codes: (ii) $x+1/2, y+1/2, z$; (iii) $x+1/2, -y-1/2, z+1/2$.