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N-*p*-Tolyl-1,3-selenazolo[5,4-*b*]pyridin-2-amine

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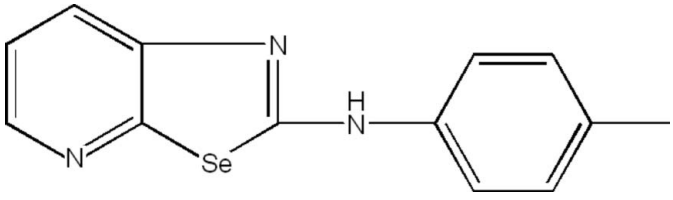
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.020 Å; R factor = 0.094; wR factor = 0.314; data-to-parameter ratio = 12.0.

In the title compound, $C_{13}H_{11}N_3Se$, the dihedral angle between the mean plane of the fused selenoazolopyridine ring system and the *p*-toluidine ring is 14.260 (5)°. In the crystal, molecules are linked by N—H···N hydrogen bonds, forming zigzag chains extending along the *b*-axis direction.

Related literature

For the bioactivity of organoselenium, see: Garud *et al.* (2007); Ling *et al.* (2010); Plamen *et al.* (2010). For crystallographic studies on selenazolo derivatives, see: Plamen *et al.* (2004).



Experimental

Crystal data

$C_{13}H_{11}N_3Se$
M_r = 288.21
Orthorhombic, *Pbca*
a = 13.138 (2) Å
b = 10.0323 (19) Å
c = 17.838 (3) Å
V = 2351.2 (7) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 4.15 mm^{-1}
T = 153 K
0.30 × 0.30 × 0.20 mm

Data collection

Agilent Xcalibur Sapphire3 Gemini ultra diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.369, T_{max} = 0.491
5248 measured reflections
1863 independent reflections
1423 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.094
$wR(F^2)$ = 0.314
S = 1.28
1863 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.47 e Å^{-3}
$\Delta\rho_{min}$ = −2.49 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N10—H10···N1^{i}	0.88	2.11	2.983 (16)	175

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2275).

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supporting information

Acta Cryst. (2013). E**69**, o1497 [doi:10.1107/S1600536813023659]

N-*p*-Tolyl-1,3-selenazolo[5,4-*b*]pyridin-2-amine

Zhaojun Wu, Yiqun Li, Hua Zhou and Meiyun Zhou

S1. Comment

Since the discovery of the importance of Se as a microelement in bacteria and animals, and the function of the seleno-enzyme glutathione peroxidase (GPx) as an antioxidant, the interest in organoselenium compounds has increased significantly (Garud *et al.*, 2007; Ling *et al.*, 2010; Plamen *et al.*, 2004, 2010). The design and synthesis of organoselenium compounds, especially Se-containing heterocycles, are our current interest. The title molecule, $C_{13}H_{11}N_3Se$, (Fig. 1) is built up from two fused rings, *viz*. the selenazolo and pyridine rings, linked to a *p*-toluidine group. The dihedral angle between these two ring systems is 14.260 (5)°. In the crystal, the molecules are linked by intermolecular N—H···N hydrogen bonds (Table 1), giving one-dimensional zigzag chains extending along *b*.

S2. Experimental

To a stirred solution of *N*-*p*-tolylformamide (10 mmol) in toluene (100 ml) in an ice bath, Et_3N (4.0 g, 40 mmol) and Se black powder were added. Phosgene (8 g of a 20% solution in toluene) was then added slowly over 30 min. giving an exothermic reaction. After complete addition, the suspension was heated under reflux for 10 h (TLC control). The mixture was filtered and washed with several portions of toluene, and the filtrate was then concentrated, affording the raw isoselenocyanatobenzene. This was added to a stirred solution of 2-chloropyridin-3-amine (1.28 g, 10 mmol) in 2-propanol at room temperature, and the mixture was heated to reflux for 6 h. After filtration, the precipitate was collected as a yellow solid. The impure product was dissolved in CCl_2H_2 at room temperature. Yellow crystals suitable for X-ray analysis (80.2% yield) grew over a period of one week when the solution was exposed to the air.

S3. Refinement

Hydrogen atoms were placed at calculated positions [N—H = 0.88 Å, C—H(aromatic) = 0.95 Å, C—H(methyl) = 0.98 Å] and treated as riding, with U_{iso}(H) = 1.2U_{eq}(N and aromatic C) and U_{iso}(H) = 1.5U_{eq}(methyl C). A check using *TwinRotMat* within *PLATON* (Spek, 2009) detected no twin law.

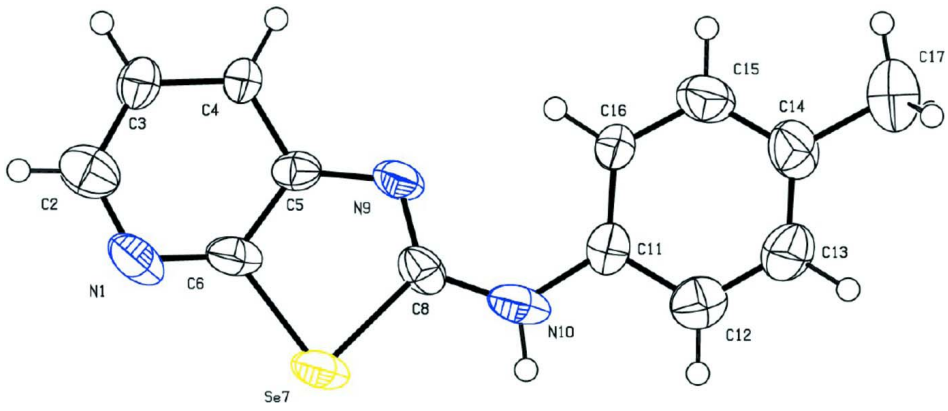

Figure 1

The molecular structure of the title compound showing the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

N-*p*-Tolyl-1,3-selenazolo[5,4-*b*]pyridin-2-amine

Crystal data

$C_{13}H_{11}N_3Se$
$M_r = 288.21$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.138$ (2) Å
$b = 10.0323$ (19) Å
$c = 17.838$ (3) Å
$V = 2351.2$ (7) Å^3
$Z = 8$
$F(000) = 1152$
$D_x = 1.628$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 7867 reflections
$\theta = 1.3$–$61.9°$
$\mu = 4.15$ mm^{-1}
$T = 153$ K
Prism, yellow
0.30 × 0.30 × 0.20 mm

Data collection

Agilent Xcalibur Sapphire3 Gemini ultra diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0288 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.369$, $T_{max} = 0.491$
5248 measured reflections
1863 independent reflections
1423 reflections with $I > 2s\tilde{}I$)
$R_{int} = 0.064$
$\theta_{max} = 63.6°$, $\theta_{min} = 6.0°$
$h = -15 \rightarrow 13$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.094$
$wR(F^2) = 0.314$
$S = 1.28$
1863 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0796P)^2 + 53.5115P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.47$ e Å^{-3}
$\Delta\rho_{min} = -2.49$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.1440 (11)	0.5123 (17)	0.1134 (9)	0.058 (4)
H2	0.1245	0.5846	0.0821	0.069*
C3	0.2452 (11)	0.4817 (14)	0.1192 (8)	0.049 (3)
H3	0.2937	0.5321	0.0917	0.058*
C4	0.2779 (10)	0.3779 (13)	0.1647 (7)	0.042 (3)
H4	0.3483	0.3586	0.1694	0.050*
C6	0.1034 (9)	0.3460 (16)	0.1916 (7)	0.045 (3)
C5	0.2051 (9)	0.3016 (13)	0.2038 (7)	0.035 (3)
C8	0.1441 (10)	0.1537 (15)	0.2778 (7)	0.041 (3)
C11	0.2188 (10)	−0.0164 (13)	0.3649 (7)	0.038 (3)
C16	0.3195 (10)	0.0139 (14)	0.3591 (8)	0.045 (3)
H16	0.3399	0.0836	0.3263	0.054*
C15	0.3925 (11)	−0.0535 (18)	0.3993 (8)	0.059 (4)
H15	0.4618	−0.0279	0.3942	0.071*
C14	0.3684 (12)	−0.1555 (19)	0.4460 (8)	0.058 (4)
C13	0.2672 (13)	−0.189 (2)	0.4510 (8)	0.064 (5)
H13	0.2482	−0.2601	0.4834	0.077*
C12	0.1923 (12)	−0.1245 (16)	0.4116 (7)	0.052 (4)
H12	0.1234	−0.1523	0.4156	0.063*
C17	0.4499 (16)	−0.2322 (19)	0.4884 (9)	0.074 (5)
H17A	0.4471	−0.3265	0.4742	0.110*
H17B	0.4380	−0.2236	0.5425	0.110*
H17C	0.5170	−0.1960	0.4760	0.110*
N1	0.0717 (9)	0.4456 (15)	0.1496 (7)	0.058 (4)
N9	0.2255 (8)	0.1982 (14)	0.2518 (6)	0.049 (3)
N10	0.1388 (8)	0.0493 (14)	0.3276 (7)	0.054 (3)
H10	0.0772	0.0197	0.3375	0.065*
Se7	0.01740 (10)	0.22670 (19)	0.24564 (9)	0.0504 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.048 (8)	0.060 (10)	0.066 (10)	0.007 (8)	−0.001 (7)	−0.012 (8)
C3	0.053 (8)	0.028 (7)	0.065 (9)	0.000 (6)	0.001 (7)	0.010 (7)
C4	0.038 (7)	0.035 (8)	0.052 (8)	0.001 (6)	0.000 (6)	0.015 (6)
C6	0.028 (6)	0.066 (10)	0.041 (7)	−0.006 (7)	−0.001 (5)	−0.006 (7)

C5	0.031 (6)	0.032 (7)	0.043 (7)	−0.006 (5)	0.001 (5)	−0.007 (6)
C8	0.036 (7)	0.048 (8)	0.040 (7)	0.016 (6)	0.002 (6)	−0.005 (6)
C11	0.051 (8)	0.032 (7)	0.031 (6)	0.006 (6)	0.005 (5)	−0.003 (6)
C16	0.040 (7)	0.042 (8)	0.052 (8)	0.004 (6)	0.005 (6)	0.017 (7)
C15	0.039 (7)	0.082 (13)	0.056 (9)	0.003 (8)	0.004 (7)	0.004 (9)
C14	0.060 (9)	0.078 (12)	0.036 (7)	0.013 (8)	0.001 (6)	0.007 (8)
C13	0.067 (11)	0.084 (14)	0.041 (8)	−0.006 (9)	0.005 (7)	0.017 (9)
C12	0.055 (9)	0.059 (10)	0.043 (7)	−0.005 (8)	0.004 (7)	−0.006 (7)
C17	0.094 (14)	0.066 (12)	0.061 (10)	0.011 (10)	−0.010 (9)	0.007 (9)
N1	0.036 (6)	0.089 (11)	0.048 (7)	0.018 (7)	−0.007 (5)	0.000 (7)
N9	0.024 (6)	0.082 (10)	0.042 (6)	0.005 (5)	0.002 (4)	0.012 (6)
N10	0.025 (5)	0.082 (10)	0.054 (7)	−0.001 (6)	0.001 (5)	−0.009 (7)
Se7	0.0237 (9)	0.0734 (13)	0.0542 (10)	−0.0005 (7)	−0.0003 (6)	−0.0021 (8)

Geometric parameters (Å, º)

C2—N1	1.33 (2)	C11—N10	1.408 (17)
C2—C3	1.37 (2)	C11—C12	1.41 (2)
C2—H2	0.9500	C16—C15	1.38 (2)
C3—C4	1.388 (18)	C16—H16	0.9500
C3—H3	0.9500	C15—C14	1.36 (2)
C4—C5	1.410 (17)	C15—H15	0.9500
C4—H4	0.9500	C14—C13	1.37 (2)
C6—N1	1.316 (19)	C14—C17	1.52 (2)
C6—C5	1.425 (18)	C13—C12	1.37 (2)
C6—Se7	1.907 (14)	C13—H13	0.9500
C5—N9	1.371 (17)	C12—H12	0.9500
C8—N9	1.248 (17)	C17—H17A	0.9800
C8—N10	1.375 (18)	C17—H17B	0.9800
C8—Se7	1.907 (12)	C17—H17C	0.9800
C11—C16	1.362 (19)	N10—H10	0.8800
N1—C2—C3	123.0 (16)	C14—C15—C16	121.9 (14)
N1—C2—H2	118.5	C14—C15—H15	119.1
C3—C2—H2	118.5	C16—C15—H15	119.1
C2—C3—C4	120.8 (14)	C15—C14—C13	116.8 (15)
C2—C3—H3	119.6	C15—C14—C17	121.6 (15)
C4—C3—H3	119.6	C13—C14—C17	121.6 (16)
C3—C4—C5	119.2 (12)	C12—C13—C14	123.0 (16)
C3—C4—H4	120.4	C12—C13—H13	118.5
C5—C4—H4	120.4	C14—C13—H13	118.5
N1—C6—C5	128.4 (12)	C13—C12—C11	119.3 (14)
N1—C6—Se7	125.2 (10)	C13—C12—H12	120.4
C5—C6—Se7	106.4 (10)	C11—C12—H12	120.4
N9—C5—C4	126.0 (11)	C14—C17—H17A	109.5
N9—C5—C6	121.0 (11)	C14—C17—H17B	109.5
C4—C5—C6	113.0 (12)	H17A—C17—H17B	109.5
N9—C8—N10	123.7 (12)	C14—C17—H17C	109.5

N9—C8—Se7	119.9 (11)	H17A—C17—H17C	109.5
N10—C8—Se7	116.3 (9)	H17B—C17—H17C	109.5
C16—C11—N10	125.7 (12)	C6—N1—C2	115.7 (12)
C16—C11—C12	117.2 (13)	C8—N9—C5	109.6 (11)
N10—C11—C12	117.1 (12)	C8—N10—C11	128.7 (11)
C11—C16—C15	121.8 (13)	C8—N10—H10	115.7
C11—C16—H16	119.1	C11—N10—H10	115.7
C15—C16—H16	119.1	C8—Se7—C6	82.9 (6)
N1—C2—C3—C4	−1 (2)	N10—C11—C12—C13	−177.9 (13)
C2—C3—C4—C5	2 (2)	C5—C6—N1—C2	1 (2)
C3—C4—C5—N9	−178.7 (13)	Se7—C6—N1—C2	−176.5 (11)
C3—C4—C5—C6	−1.3 (18)	C3—C2—N1—C6	−1 (2)
N1—C6—C5—N9	177.7 (14)	N10—C8—N9—C5	179.9 (12)
Se7—C6—C5—N9	−4.7 (15)	Se7—C8—N9—C5	2.3 (17)
N1—C6—C5—C4	0 (2)	C4—C5—N9—C8	179.0 (13)
Se7—C6—C5—C4	177.8 (9)	C6—C5—N9—C8	1.8 (18)
N10—C11—C16—C15	178.2 (14)	N9—C8—N10—C11	8 (2)
C12—C11—C16—C15	−3 (2)	Se7—C8—N10—C11	−174.5 (11)
C11—C16—C15—C14	1 (2)	C16—C11—N10—C8	1 (2)
C16—C15—C14—C13	0 (2)	C12—C11—N10—C8	−177.8 (13)
C16—C15—C14—C17	178.1 (15)	N9—C8—Se7—C6	−4.1 (12)
C15—C14—C13—C12	0 (3)	N10—C8—Se7—C6	178.1 (11)
C17—C14—C13—C12	−177.8 (15)	N1—C6—Se7—C8	−178.1 (13)
C14—C13—C12—C11	−2 (3)	C5—C6—Se7—C8	4.2 (9)
C16—C11—C12—C13	3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H10···N1^{i}	0.88	2.11	2.983 (16)	175

Symmetry code: (i) −*x*, *y*−1/2, −*z*+1/2.