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Crystal structure of 2-(3-nitrophenyl)-1,3-thiazolo[4,5-*b*]pyridine

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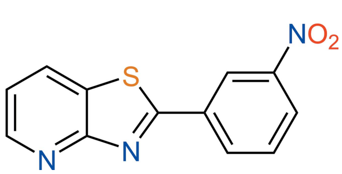
In the title compound, $C_{12}H_7N_3O_2S$, the dihedral angle between the planes of the thiazolopyridine ring system (r.m.s. deviation = 0.005 Å) and the benzene ring is 3.94 (6)°. The nitro group is rotated by 7.6 (2)° from its attached ring. In the crystal, extensive aromatic π–π stacking [shortest centroid–centroid separation = 3.5295 (9) Å] links the molecules into (001) sheets.

Keywords: crystal structure; nitrophenyl; thiazolopyridine derivatives; thiazolo[4,5-*b*]pyridine.

CCDC reference: 1430578

1. Related literature

For a related structure and background references, see: El-Hiti *et al.* (2015). For further synthetic details, see: Smith *et al.* (1995); El-Hiti (2003).



2. Experimental

2.1. Crystal data

$C_{12}H_7N_3O_2S$
M_r = 257.27
Monoclinic, $P2_1/c$
a = 9.5596 (2) Å
b = 9.8733 (2) Å
c = 11.5606 (3) Å
β = 98.122 (2)°
V = 1080.20 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.66 mm^{-1}
T = 296 K
0.36 × 0.24 × 0.03 mm

2.2. Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
Absorption correction: Gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.883, T_{max} = 0.986
4063 measured reflections
2104 independent reflections
1930 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.06
2104 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7519).

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supporting information

Acta Cryst. (2015). E**71**, o877 [doi:10.1107/S2056989015019118]

Crystal structure of 2-(3-nitrophenyl)-1,3-thiazolo[4,5-*b*]pyridine

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Mansour D. Ajarim and Benson M. Kariuki

S1. Introduction

As part of our ongoing studies of thiazolopyridines (El-Hiti *et al.*, 2015), the title compound was prepared by two different processes (El-Hiti, 2003; Smith *et al.*, 1995) and its structure was determined.

S2. Experimental

S2.1. Synthesis and crystallization

2-(3-Nitrophenyl)-1,3-thiazolo[4,5-*b*]pyridine was obtained in 90% yield from acid hydrolysis (HCl, 5 M) of 3-(diisopropylaminothiocarbonylthio)-2-(3-nitrophenylcarbonylamino)pyridine under reflux for 5 h (Smith *et al.*, 1995) or in 58% yield from reaction of 3-(diisopropylaminothiocarbonylthio)-2-aminopyridine with 3-nitrobenzoic acid in the presence of phosphorus oxychloride under reflux for 4 h (El-Hiti, 2003). Crystallization of the crude product from chloroform gave the title compound as colourless crystals. The structure of the title compound was elucidated by various spectroscopic and analytical data, which were consistent with those reported (Smith *et al.*, 1995).

S2.2. Refinement

H atoms were positioned geometrically and refined using a riding model with U_{iso}(H) constrained to be 1.2 times U_{eq} for the atom it is bonded to.

S3. Results and discussion

The asymmetric unit comprises one molecule of $C_{12}H_7N_3O_2S$ (Fig. 1). The phenylthiazolopyridine ring system is flat with a maximum deviation of 0.072 (1)Å from the least squares plane. The nitro group is twisted from this plane by only 7.6 (2)°. In the crystal, extensive π - π overlap occurs between pairs of inversion related molecules with a phenyl to thiazolopyridine centroid distance of 3.47 (2)Å (Fig. 2).

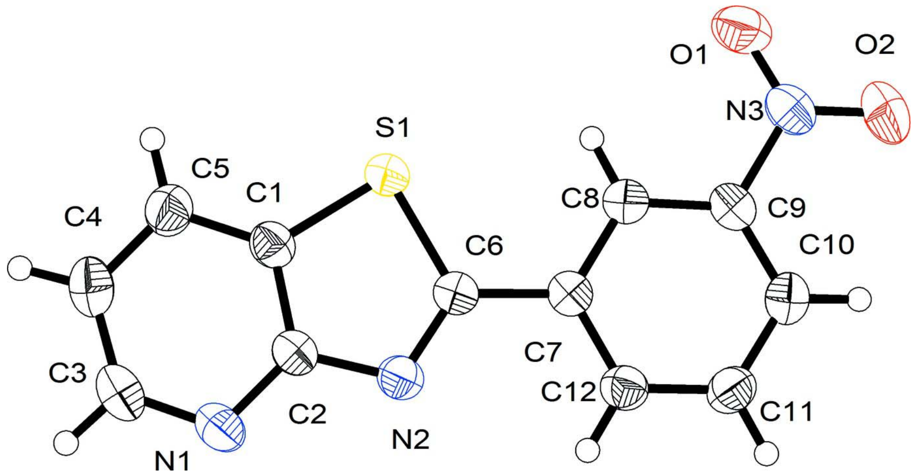

Figure 1

The asymmetric unit of $C_{12}H_7N_3O_2S$ with 50% probability displacement ellipsoids for nonhydrogen atoms.

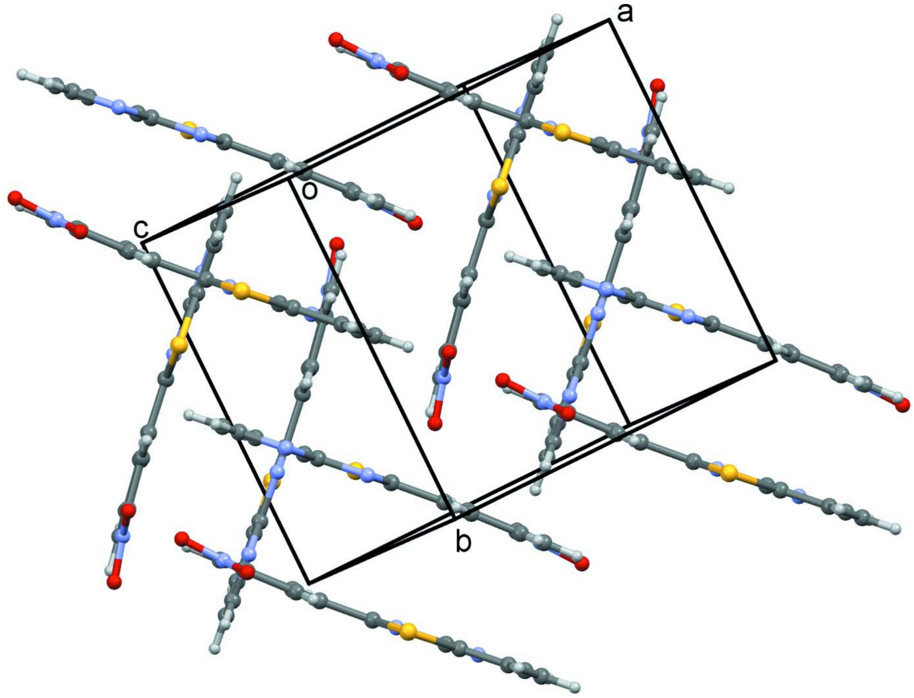

Figure 2

A segment of the crystal structure with H atoms omitted for clarity.

2-(3-Nitrophenyl)-1,3-thiazolo[4,5-*b*]pyridine

Crystal data

$C_{12}H_7N_3O_2S$
$M_r = 257.27$
Monoclinic, $P2_1/c$
$a = 9.5596$ (2) Å
$b = 9.8733$ (2) Å
$c = 11.5606$ (3) Å
$\beta = 98.122$ (2)°
$V = 1080.20$ (4) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.582$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2610 reflections
$\theta = 5.9$–73.8°

μ = 2.66 mm^{-1}
T = 296 K

Plate, colourless
0.36 × 0.24 × 0.03 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Atlas detector
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.883, T_{max} = 0.986
4063 measured reflections

2104 independent reflections
1930 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 73.8°, θ_{min} = 5.9°
$h = -6 \rightarrow 11$
$k = -12 \rightarrow 10$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.06
2104 reflections
163 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.2004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) Numerical absorption correction based on gaussian integration over a multifaceted crystal model Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.03467 (14)	0.31902 (15)	0.40820 (12)	0.0359 (3)
C2	1.05966 (14)	0.29483 (14)	0.52938 (12)	0.0346 (3)
C3	1.23544 (16)	0.44974 (17)	0.55180 (15)	0.0466 (4)
H3	1.3056	0.4963	0.5999	0.056*
C4	1.21719 (17)	0.48032 (17)	0.43313 (15)	0.0476 (4)
H4	1.2737	0.5453	0.4044	0.057*
C5	1.11469 (16)	0.41357 (17)	0.35833 (14)	0.0446 (3)
H5	1.1001	0.4312	0.2785	0.054*
C6	0.88486 (14)	0.15122 (14)	0.48354 (11)	0.0327 (3)
C7	0.78177 (13)	0.04398 (14)	0.49734 (11)	0.0323 (3)
C8	0.69113 (14)	−0.00588 (14)	0.40184 (12)	0.0338 (3)
H8	0.6911	0.0309	0.3278	0.041*
C9	0.60130 (13)	−0.11128 (14)	0.41963 (12)	0.0342 (3)
C10	0.59533 (15)	−0.16807 (15)	0.52740 (13)	0.0392 (3)
H10	0.5333	−0.2386	0.5365	0.047*
C11	0.68472 (16)	−0.11691 (16)	0.62203 (13)	0.0419 (3)

H11	0.6827	−0.1532	0.6960	0.050*
C12	0.77673 (15)	−0.01253 (16)	0.60750 (12)	0.0381 (3)
H12	0.8362	0.0206	0.6719	0.046*
N1	1.16011 (14)	0.35893 (14)	0.60175 (11)	0.0442 (3)
N2	0.97356 (13)	0.19809 (13)	0.56961 (10)	0.0367 (3)
N3	0.50993 (13)	−0.16657 (14)	0.31766 (11)	0.0420 (3)
O1	0.51160 (13)	−0.11243 (14)	0.22310 (10)	0.0563 (3)
O2	0.43804 (16)	−0.26586 (16)	0.33152 (13)	0.0698 (4)
S1	0.89807 (4)	0.21799 (4)	0.34482 (3)	0.03972 (13)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0339 (7)	0.0341 (7)	0.0386 (7)	0.0002 (5)	0.0009 (5)	−0.0009 (6)
C2	0.0322 (7)	0.0348 (7)	0.0361 (7)	0.0004 (5)	0.0023 (5)	−0.0029 (5)
C3	0.0387 (7)	0.0429 (8)	0.0565 (9)	−0.0067 (6)	0.0004 (6)	−0.0100 (7)
C4	0.0423 (8)	0.0379 (8)	0.0627 (10)	−0.0072 (6)	0.0075 (7)	0.0012 (7)
C5	0.0445 (8)	0.0431 (8)	0.0458 (8)	−0.0041 (6)	0.0048 (6)	0.0068 (6)
C6	0.0318 (6)	0.0337 (7)	0.0322 (6)	0.0021 (5)	0.0037 (5)	−0.0016 (5)
C7	0.0293 (6)	0.0323 (6)	0.0355 (7)	0.0035 (5)	0.0049 (5)	−0.0017 (5)
C8	0.0316 (6)	0.0351 (7)	0.0346 (6)	0.0035 (5)	0.0047 (5)	−0.0004 (5)
C9	0.0279 (6)	0.0342 (7)	0.0398 (7)	0.0040 (5)	0.0024 (5)	−0.0050 (5)
C10	0.0353 (7)	0.0349 (7)	0.0479 (8)	−0.0002 (6)	0.0080 (6)	0.0028 (6)
C11	0.0436 (8)	0.0443 (8)	0.0380 (7)	0.0000 (6)	0.0060 (6)	0.0068 (6)
C12	0.0362 (7)	0.0417 (8)	0.0355 (7)	0.0001 (6)	0.0021 (5)	−0.0002 (6)
N1	0.0410 (6)	0.0476 (7)	0.0420 (7)	−0.0059 (5)	−0.0012 (5)	−0.0079 (6)
N2	0.0358 (6)	0.0401 (6)	0.0335 (6)	−0.0017 (5)	0.0029 (5)	−0.0017 (5)
N3	0.0352 (6)	0.0425 (7)	0.0467 (7)	0.0013 (5)	0.0006 (5)	−0.0082 (6)
O1	0.0582 (7)	0.0652 (8)	0.0421 (6)	−0.0030 (6)	−0.0052 (5)	−0.0054 (6)
O2	0.0684 (8)	0.0638 (9)	0.0724 (9)	−0.0315 (7)	−0.0067 (7)	−0.0050 (7)
S1	0.0419 (2)	0.0429 (2)	0.0323 (2)	−0.00887 (14)	−0.00199 (14)	0.00294 (13)

Geometric parameters (Å, º)

C1—C5	1.383 (2)	C7—C8	1.3935 (19)
C1—C2	1.408 (2)	C7—C12	1.3974 (19)
C1—S1	1.7224 (14)	C8—C9	1.383 (2)
C2—N1	1.3400 (19)	C8—H8	0.9300
C2—N2	1.3836 (19)	C9—C10	1.375 (2)
C3—N1	1.332 (2)	C9—N3	1.4696 (18)
C3—C4	1.391 (2)	C10—C11	1.385 (2)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.379 (2)	C11—C12	1.380 (2)
C4—H4	0.9300	C11—H11	0.9300
C5—H5	0.9300	C12—H12	0.9300
C6—N2	1.2980 (18)	N3—O1	1.2190 (18)
C6—C7	1.4705 (19)	N3—O2	1.221 (2)
C6—S1	1.7548 (14)		

C5—C1—C2	120.20 (13)	C9—C8—C7	118.53 (13)
C5—C1—S1	130.10 (12)	C9—C8—H8	120.7
C2—C1—S1	109.69 (11)	C7—C8—H8	120.7
N1—C2—N2	121.61 (13)	C10—C9—C8	123.22 (13)
N1—C2—C1	123.16 (14)	C10—C9—N3	118.65 (13)
N2—C2—C1	115.23 (12)	C8—C9—N3	118.12 (13)
N1—C3—C4	124.99 (14)	C9—C10—C11	117.85 (13)
N1—C3—H3	117.5	C9—C10—H10	121.1
C4—C3—H3	117.5	C11—C10—H10	121.1
C5—C4—C3	119.56 (15)	C12—C11—C10	120.58 (14)
C5—C4—H4	120.2	C12—C11—H11	119.7
C3—C4—H4	120.2	C10—C11—H11	119.7
C4—C5—C1	116.54 (14)	C11—C12—C7	120.90 (13)
C4—C5—H5	121.7	C11—C12—H12	119.5
C1—C5—H5	121.7	C7—C12—H12	119.5
N2—C6—C7	123.28 (12)	C3—N1—C2	115.53 (14)
N2—C6—S1	116.30 (11)	C6—N2—C2	110.10 (12)
C7—C6—S1	120.37 (10)	O1—N3—O2	123.28 (14)
C8—C7—C12	118.91 (13)	O1—N3—C9	118.36 (13)
C8—C7—C6	121.31 (12)	O2—N3—C9	118.35 (13)
C12—C7—C6	119.76 (12)	C1—S1—C6	88.68 (7)