Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[(5,6,7-Trimethyl[1,2,4]triazolo[1,5-*a*]-pyrimidin-2-ylsulfanyl)methyl]benzoic acid

Yan-Zhen He

Key Laboratory of Pesticides and Chemical Biology of the Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: hyzccnu@yahoo.com.cn

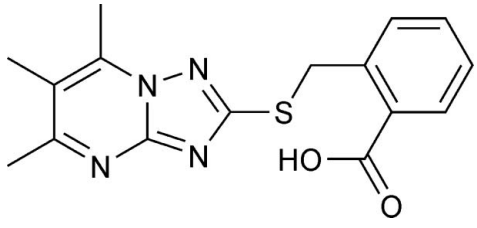
Received 13 April 2007; accepted 19 April 2007

Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.154; data-to-parameter ratio = 16.6.

In the crystal structure of the title compound, $C_{16}H_{16}N_4O_2S$, there are two independent molecules with different conformations in the asymmetric unit; these are linked into chains by O—H···N hydrogen bonds. Further stability may be provided by offset π–π stacking interactions involving inversion-related pyrimidine rings [centroid–centroid distance = 3.84 (1) Å] and benzene rings [centroid–centroid distance = 3.45 (1) Å].

Related literature

For related structures, see: Chen, Li *et al.* (2005); Chen, Wu *et al.* (2005); Teng *et al.* (2005). For related literature, see: Allen *et al.* (1987); Janiak (2000); Kleschich *et al.* (1990).



Experimental

Crystal data

$C_{16}H_{16}N_4O_2S$
M_r = 328.39
Triclinic, $P\overline{1}$
a = 7.9704 (5) Å
b = 13.3058 (8) Å
c = 15.9084 (9) Å
α = 110.257 (1)°
β = 96.446 (1)°
γ = 92.620 (1)°
V = 1566.28 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 299 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.936, T_{max} = 0.978
17990 measured reflections
7079 independent reflections
5206 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.154
S = 1.04
7079 reflections
427 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···N1^{i}	0.82 (3)	1.90 (3)	2.721 (2)	175 (3)
O1—H1···N8^{ii}	0.99 (3)	1.79 (3)	2.762 (2)	168 (3)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

The author acknowledges the National Basic Research Program of China (grant No. 2004CCA00100) and the National Natural Science Foundation of China (grant No. 20102001).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2373).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2001). *SMART* (Version 5.628), *SAINT* (Version 6.45) and *SHELXTL* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, Q., Li, Y.-X., Hu, X.-W. & Yang, G.-F. (2005). *Acta Cryst.* E**61**, o3842–o3843.
Chen, Q., Wu, Q.-Y., Hu, X.-W. & Yang, G.-F. (2005). *Acta Cryst.* E**61**, o2079–o2080.
Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
Kleschich, W. A., Costales, M. J. & Dunbar, J. E. (1990). *Pestic. Sci.* pp. 341–355.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Teng, D.-Y., Ji, F.-Q. & Huang, W. (2005). *Acta Cryst.* E**61**, o1992–o1993.

supplementary materials

Acta Cryst. (2007). E**63**, o2675 [**doi:10.1107/S1600536807019538**]

2-[(5,6,7-Trimethyl[1,2,4]triazolo[1,5-*a*]pyrimidin-2-ylsulfanyl)methyl]benzoic acid

Y.-Z. He

Comment

Triazolopyrimidine derivatives have broad biological properties: in particular triazolopyrimidine sulfonamide is a highly effective herbicide with acetohydroxyacid synthase (AHAS) as target (Kleschich *et al.*, 1990). We herein report the crystal structure of one such triazolopyrimidine derivative, the title compound, (I).

The crystal structure of the the title compound contains two independent molecules (A and B) in the asymmetric unit (Fig. 1) with bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In molecule A, the triazolopyrimidine fused rings (N1—N4/C9—C13) are close to planarity with a maximum deviation of 0.028 (2)Å for C13. The dihedral angle between triazolopyrimidine and benzene rings is 70.56 (3)°.

In molecule B, the triazolopyrimidine fused rings (N5—N8/C25—C29) are almost planar with a maximum deviation of 0.031 (2)Å for C25. The dihedral angle between triazolopyrimidine and benzene rings is 54.83 (3)°.

In the crystal of (I), intermolecular O—H···N hydrogen bonds (Table 2) lead to chains of molecules (Fig. 2). Further stability is provided by offset π-π stacking interactions (Janiak, 2000). The adjacent pyrimidine rings (N6/N8/C26—C29) have a centroid-centroid distance of 3.84 (1) %A [symmetry code: - x, 1 - y, 1 - z], while the adjacent benzene rings (C1—C6) have a centroid-centroid distance of 3.45 (1) %A [symmetry

code: 1 - x, 2 - y, - z].

Experimental

Sodium hydroxide (0.08 g, 2 mmol) was dissolved in ethanol (5 ml), and then 5-amino-1*H*-1,2,4-triazole-3-thiol (0.23 g, 2 mmol) was added. Then, methyl 2-(chloromethyl)benzoate (0.37 g, 2 mmol) was added dropwise until the solid dissolved completely. After stirring for 2 h at room temperature, the precipitate was filtered off, washed with water and dried to give methyl 2-((5-amino-1*H*-1,2,4-triazol-3-ylthio)methyl)benzoate (0.38 g, yield 72%). This ester (1.06 g, 4 mmol) in 4 ml glacial acetic acid with 3-methylpentane-2,4-dione (0.46 g, 4 mmol) was refluxed for 6 h. The solution was poured into 50 ml ice and water and stirred overnight. The solid which separated from water was dried to obtain methyl 2-((5,6,7-trimethyl-[1,2,4]triazolo[1,5-a]pyrimidin-2-ylthio)methyl) benzoate (1.24 g, yield 91%). Then it (0.6 g, 1.75 mmol) was added into the solution of NaOH (0.14 g, 3.5 mmol) in ethanol (5 ml) and water (5 ml) and the mixture was refluxed for 30 minutes. The solution was quenched with 20 ml water and then treated with diluted hydrochloric acid until pH = 1 was reached. The precipitate was collected by filtration and recrystallized from ethanol to obtain golden yellow crystals of the title compound (0.40 g, yield 67%). Crystals of (I) suitable for X-ray analysis were grown from acetone at 277 K.

Refinement

O-bound H atoms were located in a difference Fourier map and freely refined with fixed isotropic displacement parameters. All other H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

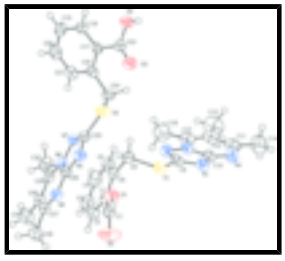


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

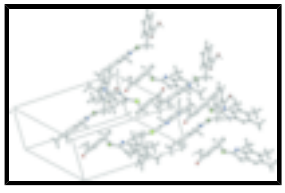


Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

2-[(5,6,7-Trimethyl[1,2,4]triazolo[1,5-a]pyrimidin-2-ylsulfanyl)methyl]benzoic acid

Crystal data

$C_{16}H_{16}N_4O_2S$	$Z = 4$
$M_r = 328.39$	$F_{000} = 688$
Triclinic, $P\overline{1}$	$D_x = 1.393$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.9704$ (5) Å	Cell parameters from 2157 reflections
$b = 13.3058$ (8) Å	$\theta = 2.2$–24.5º
$c = 15.9084$ (9) Å	$\mu = 0.22$ mm^{-1}
$\alpha = 110.257$ (1)º	$T = 299$ (2) K
$\beta = 96.446$ (1)º	Block, golden yellow
$\gamma = 92.620$ (1)º	0.30 × 0.20 × 0.10 mm
$V = 1566.28$ (16) Å^3	

Data collection

Bruker SMART 4K CCD diffractometer	7079 independent reflections
Radiation source: fine-focus sealed tube	5206 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 299$(2) K	$\theta_{max} = 27.5$º

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.936, T_{max} = 0.978

17990 measured reflections

θ_{min} = 1.6º

h = −10→10

k = −17→17

l = −20→20

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.058

$wR(F^2)$ = 0.154

S = 1.04

7079 reflections

427 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.1225P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.36 e Å^{-3}

$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Extinction correction: none

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0916 (3)	0.66968 (18)	0.02579 (15)	0.0404 (5)
C2	0.1370 (3)	0.5831 (2)	−0.04390 (17)	0.0525 (6)
H2	0.0930	0.5724	−0.1033	0.063*
C3	0.2454 (3)	0.5129 (2)	−0.0268 (2)	0.0621 (7)
H3	0.2743	0.4554	−0.0744	0.075*
C4	0.3110 (3)	0.5280 (2)	0.0610 (2)	0.0585 (7)
H4	0.3861	0.4816	0.0729	0.070*
C5	0.2649 (3)	0.6122 (2)	0.13099 (18)	0.0498 (6)
H5	0.3078	0.6210	0.1902	0.060*
C6	0.1559 (3)	0.68464 (17)	0.11557 (15)	0.0378 (5)

C7	−0.0240 (3)	0.7404 (2)	−0.00253 (16)	0.0468 (6)
C8	0.1109 (3)	0.77229 (18)	0.19690 (15)	0.0422 (5)
H8A	0.1385	0.8410	0.1908	0.051*
H8B	0.1818	0.7709	0.2501	0.051*
C9	−0.1156 (3)	0.66328 (17)	0.26135 (13)	0.0378 (5)
C10	−0.2154 (3)	0.55875 (18)	0.32078 (14)	0.0423 (5)
C11	−0.2305 (4)	0.4334 (2)	0.38613 (17)	0.0561 (7)
C12	−0.0607 (4)	0.4109 (2)	0.37478 (17)	0.0553 (7)
C13	0.0312 (3)	0.46493 (19)	0.33293 (16)	0.0504 (6)
C14	−0.3379 (5)	0.3743 (3)	0.4296 (2)	0.0879 (10)
H14A	−0.4440	0.4054	0.4374	0.132*
H14B	−0.2796	0.3801	0.4875	0.132*
H14C	−0.3584	0.2999	0.3916	0.132*
C15	0.0175 (5)	0.3289 (3)	0.4084 (2)	0.0859 (10)
H15A	0.1310	0.3218	0.3937	0.129*
H15B	−0.0486	0.2608	0.3801	0.129*
H15C	0.0200	0.3517	0.4728	0.129*
C16	0.2060 (4)	0.4496 (3)	0.3089 (2)	0.0736 (9)
H16A	0.2021	0.4268	0.2444	0.110*
H16B	0.2525	0.3959	0.3299	0.110*
H16C	0.2759	0.5163	0.3369	0.110*
C17	0.6049 (2)	1.14480 (16)	0.50037 (13)	0.0326 (4)
C18	0.7736 (3)	1.17560 (18)	0.53904 (15)	0.0389 (5)
H18	0.7989	1.2305	0.5951	0.047*
C19	0.9034 (3)	1.1257 (2)	0.49523 (17)	0.0497 (6)
H19	1.0156	1.1475	0.5212	0.060*
C20	0.8663 (3)	1.0437 (2)	0.41324 (18)	0.0547 (7)
H20	0.9534	1.0095	0.3836	0.066*
C21	0.7000 (3)	1.01192 (19)	0.37471 (16)	0.0465 (6)
H21	0.6766	0.9555	0.3194	0.056*
C22	0.5665 (3)	1.06159 (17)	0.41607 (13)	0.0349 (5)
C23	0.4685 (3)	1.20209 (17)	0.54894 (14)	0.0374 (5)
C24	0.3889 (3)	1.02269 (18)	0.36945 (15)	0.0415 (5)
H24A	0.3184	1.0156	0.4133	0.050*
H24B	0.3894	0.9520	0.3238	0.050*
C25	0.3128 (3)	1.04492 (17)	0.20231 (14)	0.0373 (5)
C26	0.2631 (3)	1.01239 (17)	0.06348 (14)	0.0361 (5)
C27	0.2523 (3)	0.9258 (2)	−0.08829 (15)	0.0443 (5)
C28	0.3533 (3)	0.8463 (2)	−0.07553 (16)	0.0464 (6)
C29	0.4127 (3)	0.85393 (18)	0.01195 (16)	0.0404 (5)
C30	0.1838 (4)	0.9204 (3)	−0.18193 (17)	0.0663 (8)
H30A	0.0950	0.8629	−0.2076	0.099*
H30B	0.2732	0.9075	−0.2191	0.099*
H30C	0.1394	0.9873	−0.1789	0.099*
C31	0.3967 (4)	0.7532 (2)	−0.15416 (19)	0.0719 (8)
H31A	0.4912	0.7211	−0.1340	0.108*
H31B	0.4259	0.7788	−0.2006	0.108*
H31C	0.3007	0.7006	−0.1778	0.108*
C32	0.5236 (3)	0.7820 (2)	0.04002 (18)	0.0527 (6)

H32A	0.5969	0.8230	0.0946	0.079*
H32B	0.5908	0.7488	−0.0069	0.079*
H32C	0.4553	0.7274	0.0506	0.079*
N1	−0.2602 (2)	0.63664 (15)	0.28941 (12)	0.0436 (5)
N2	−0.0514 (2)	0.54084 (14)	0.30882 (12)	0.0411 (4)
N3	0.0152 (2)	0.60808 (15)	0.26981 (12)	0.0417 (4)
N4	−0.3095 (3)	0.50543 (17)	0.35896 (14)	0.0550 (5)
N5	0.2299 (2)	1.08180 (14)	0.14094 (12)	0.0407 (4)
N6	0.3622 (2)	0.93763 (14)	0.07882 (11)	0.0353 (4)
N7	0.3971 (2)	0.95893 (14)	0.17080 (12)	0.0380 (4)
N8	0.2086 (2)	1.00780 (15)	−0.02147 (12)	0.0421 (4)
O1	−0.0147 (2)	0.83985 (14)	0.05479 (11)	0.0506 (4)
H1	−0.084 (4)	0.889 (2)	0.0340 (19)	0.076*
O2	−0.1170 (3)	0.70967 (18)	−0.07322 (14)	0.0903 (7)
O3	0.3282 (2)	1.20440 (16)	0.51303 (11)	0.0607 (5)
O4	0.5167 (2)	1.25087 (15)	0.63675 (11)	0.0541 (5)
H4A	0.444 (4)	1.287 (2)	0.661 (2)	0.081*
S1	−0.10910 (8)	0.76491 (5)	0.21583 (4)	0.04530 (17)
S2	0.29647 (8)	1.11214 (5)	0.31609 (4)	0.04801 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0380 (11)	0.0426 (12)	0.0398 (12)	−0.0046 (9)	0.0061 (9)	0.0142 (10)
C2	0.0513 (14)	0.0559 (15)	0.0410 (13)	−0.0019 (12)	0.0086 (11)	0.0058 (11)
C3	0.0586 (16)	0.0462 (15)	0.0687 (19)	0.0025 (12)	0.0208 (14)	0.0008 (13)
C4	0.0485 (14)	0.0522 (15)	0.080 (2)	0.0142 (12)	0.0184 (14)	0.0263 (14)
C5	0.0419 (13)	0.0565 (15)	0.0550 (15)	0.0050 (11)	0.0063 (11)	0.0248 (12)
C6	0.0336 (11)	0.0398 (12)	0.0402 (12)	−0.0020 (9)	0.0062 (9)	0.0150 (10)
C7	0.0468 (13)	0.0542 (15)	0.0403 (13)	−0.0032 (11)	0.0007 (10)	0.0206 (11)
C8	0.0469 (13)	0.0416 (12)	0.0363 (12)	−0.0042 (10)	−0.0002 (9)	0.0145 (10)
C9	0.0453 (12)	0.0382 (11)	0.0278 (10)	0.0086 (10)	0.0076 (9)	0.0079 (9)
C10	0.0473 (13)	0.0428 (13)	0.0349 (12)	0.0071 (10)	0.0073 (10)	0.0105 (10)
C11	0.0758 (18)	0.0456 (14)	0.0461 (14)	−0.0007 (13)	0.0053 (13)	0.0171 (12)
C12	0.0789 (19)	0.0411 (13)	0.0440 (14)	0.0094 (13)	0.0019 (13)	0.0142 (11)
C13	0.0621 (16)	0.0446 (13)	0.0428 (13)	0.0136 (12)	0.0016 (11)	0.0139 (11)
C14	0.108 (3)	0.082 (2)	0.091 (2)	−0.0063 (19)	0.019 (2)	0.053 (2)
C15	0.118 (3)	0.065 (2)	0.086 (2)	0.0262 (19)	0.008 (2)	0.0412 (18)
C16	0.0677 (19)	0.084 (2)	0.080 (2)	0.0341 (16)	0.0126 (16)	0.0382 (18)
C17	0.0325 (10)	0.0382 (11)	0.0320 (10)	0.0051 (8)	0.0059 (8)	0.0179 (9)
C18	0.0348 (11)	0.0474 (13)	0.0371 (12)	0.0000 (9)	0.0019 (9)	0.0197 (10)
C19	0.0316 (11)	0.0684 (17)	0.0593 (16)	0.0095 (11)	0.0064 (11)	0.0345 (14)
C20	0.0483 (14)	0.0714 (18)	0.0568 (16)	0.0264 (13)	0.0229 (12)	0.0309 (14)
C21	0.0581 (15)	0.0474 (13)	0.0377 (12)	0.0158 (11)	0.0124 (11)	0.0164 (10)
C22	0.0411 (11)	0.0382 (11)	0.0307 (11)	0.0050 (9)	0.0052 (9)	0.0185 (9)
C23	0.0337 (11)	0.0444 (12)	0.0334 (11)	0.0036 (9)	0.0037 (9)	0.0132 (9)
C24	0.0466 (13)	0.0418 (12)	0.0356 (12)	−0.0024 (10)	−0.0024 (9)	0.0161 (10)
C25	0.0341 (11)	0.0393 (12)	0.0372 (11)	−0.0017 (9)	−0.0036 (9)	0.0152 (9)

C26	0.0332 (10)	0.0383 (11)	0.0394 (12)	−0.0022 (9)	−0.0003 (9)	0.0193 (10)
C27	0.0378 (12)	0.0587 (15)	0.0366 (12)	−0.0080 (11)	0.0030 (9)	0.0194 (11)
C28	0.0412 (12)	0.0529 (14)	0.0401 (13)	−0.0066 (11)	0.0091 (10)	0.0103 (11)
C29	0.0314 (11)	0.0416 (12)	0.0489 (13)	−0.0026 (9)	0.0090 (9)	0.0164 (10)
C30	0.0600 (16)	0.098 (2)	0.0409 (15)	0.0016 (15)	0.0020 (12)	0.0274 (15)
C31	0.079 (2)	0.0725 (19)	0.0550 (17)	0.0015 (16)	0.0204 (15)	0.0087 (14)
C32	0.0441 (13)	0.0485 (14)	0.0663 (17)	0.0083 (11)	0.0126 (12)	0.0194 (13)
N1	0.0460 (11)	0.0474 (11)	0.0413 (11)	0.0119 (9)	0.0140 (8)	0.0171 (9)
N2	0.0477 (11)	0.0398 (10)	0.0360 (10)	0.0096 (8)	0.0061 (8)	0.0128 (8)
N3	0.0432 (10)	0.0424 (10)	0.0417 (10)	0.0077 (8)	0.0069 (8)	0.0170 (8)
N4	0.0614 (13)	0.0547 (13)	0.0538 (13)	0.0023 (10)	0.0146 (10)	0.0237 (11)
N5	0.0425 (10)	0.0419 (10)	0.0389 (10)	0.0046 (8)	−0.0014 (8)	0.0178 (8)
N6	0.0319 (9)	0.0394 (10)	0.0348 (9)	−0.0007 (7)	0.0016 (7)	0.0149 (8)
N7	0.0374 (9)	0.0414 (10)	0.0342 (10)	0.0010 (8)	−0.0019 (7)	0.0145 (8)
N8	0.0410 (10)	0.0507 (11)	0.0380 (10)	−0.0021 (8)	0.0003 (8)	0.0225 (9)
O1	0.0548 (10)	0.0472 (10)	0.0511 (10)	0.0056 (8)	−0.0034 (8)	0.0222 (8)
O2	0.1126 (18)	0.0799 (15)	0.0601 (13)	0.0129 (13)	−0.0336 (12)	0.0152 (11)
O3	0.0384 (9)	0.0916 (14)	0.0435 (10)	0.0207 (9)	0.0016 (7)	0.0124 (9)
O4	0.0445 (9)	0.0730 (12)	0.0340 (9)	0.0208 (8)	0.0044 (7)	0.0037 (8)
S1	0.0544 (4)	0.0425 (3)	0.0446 (3)	0.0164 (3)	0.0148 (3)	0.0185 (3)
S2	0.0550 (4)	0.0494 (4)	0.0350 (3)	0.0131 (3)	−0.0044 (3)	0.0113 (3)

Geometric parameters (Å, °)

C1—C2	1.389 (3)	C17—C23	1.489 (3)
C1—C6	1.404 (3)	C18—C19	1.379 (3)
C1—C7	1.489 (3)	C18—H18	0.9300
C2—C3	1.375 (4)	C19—C20	1.370 (4)
C2—H2	0.9300	C19—H19	0.9300
C3—C4	1.377 (4)	C20—C21	1.378 (3)
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.377 (3)	C21—C22	1.386 (3)
C4—H4	0.9300	C21—H21	0.9300
C5—C6	1.391 (3)	C22—C24	1.501 (3)
C5—H5	0.9300	C23—O3	1.204 (2)
C6—C8	1.503 (3)	C23—O4	1.322 (3)
C7—O2	1.203 (3)	C24—S2	1.822 (2)
C7—O1	1.315 (3)	C24—H24A	0.9700
C8—S1	1.816 (2)	C24—H24B	0.9700
C8—H8A	0.9700	C25—N7	1.327 (3)
C8—H8B	0.9700	C25—N5	1.359 (3)
C9—N3	1.323 (3)	C25—S2	1.742 (2)
C9—N1	1.357 (3)	C26—N5	1.322 (3)
C9—S1	1.742 (2)	C26—N8	1.352 (3)
C10—N4	1.337 (3)	C26—N6	1.368 (3)
C10—N1	1.343 (3)	C27—N8	1.325 (3)
C10—N2	1.362 (3)	C27—C28	1.414 (3)
C11—N4	1.335 (3)	C27—C30	1.504 (3)
C11—C12	1.417 (4)	C28—C29	1.386 (3)

C11—C14	1.509 (4)	C28—C31	1.511 (4)
C12—C13	1.374 (4)	C29—N6	1.360 (3)
C12—C15	1.502 (4)	C29—C32	1.477 (3)
C13—N2	1.369 (3)	C30—H30A	0.9600
C13—C16	1.490 (4)	C30—H30B	0.9600
C14—H14A	0.9600	C30—H30C	0.9600
C14—H14B	0.9600	C31—H31A	0.9600
C14—H14C	0.9600	C31—H31B	0.9600
C15—H15A	0.9600	C31—H31C	0.9600
C15—H15B	0.9600	C32—H32A	0.9600
C15—H15C	0.9600	C32—H32B	0.9600
C16—H16A	0.9600	C32—H32C	0.9600
C16—H16B	0.9600	N2—N3	1.372 (3)
C16—H16C	0.9600	N6—N7	1.383 (2)
C17—C18	1.394 (3)	O1—H1	0.99 (3)
C17—C22	1.402 (3)	O4—H4A	0.82 (3)
C2—C1—C6	119.2 (2)	C18—C19—H19	120.2
C2—C1—C7	115.7 (2)	C19—C20—C21	120.0 (2)
C6—C1—C7	125.1 (2)	C19—C20—H20	120.0
C3—C2—C1	121.4 (2)	C21—C20—H20	120.0
C3—C2—H2	119.3	C20—C21—C22	121.9 (2)
C1—C2—H2	119.3	C20—C21—H21	119.1
C2—C3—C4	119.8 (2)	C22—C21—H21	119.1
C2—C3—H3	120.1	C21—C22—C17	118.0 (2)
C4—C3—H3	120.1	C21—C22—C24	118.6 (2)
C5—C4—C3	119.6 (2)	C17—C22—C24	123.40 (19)
C5—C4—H4	120.2	O3—C23—O4	122.8 (2)
C3—C4—H4	120.2	O3—C23—C17	124.2 (2)
C4—C5—C6	121.8 (2)	O4—C23—C17	113.04 (17)
C4—C5—H5	119.1	C22—C24—S2	113.11 (15)
C6—C5—H5	119.1	C22—C24—H24A	109.0
C5—C6—C1	118.2 (2)	S2—C24—H24A	109.0
C5—C6—C8	117.4 (2)	C22—C24—H24B	109.0
C1—C6—C8	124.3 (2)	S2—C24—H24B	109.0
O2—C7—O1	122.3 (2)	H24A—C24—H24B	107.8
O2—C7—C1	122.7 (2)	N7—C25—N5	117.43 (19)
O1—C7—C1	114.94 (19)	N7—C25—S2	124.95 (16)
C6—C8—S1	115.71 (15)	N5—C25—S2	117.61 (16)
C6—C8—H8A	108.4	N5—C26—N8	128.4 (2)
S1—C8—H8A	108.4	N5—C26—N6	110.32 (18)
C6—C8—H8B	108.4	N8—C26—N6	121.2 (2)
S1—C8—H8B	108.4	N8—C27—C28	124.1 (2)
H8A—C8—H8B	107.4	N8—C27—C30	115.2 (2)
N3—C9—N1	116.7 (2)	C28—C27—C30	120.6 (2)
N3—C9—S1	123.20 (17)	C29—C28—C27	118.7 (2)
N1—C9—S1	120.07 (16)	C29—C28—C31	119.3 (2)
N4—C10—N1	128.0 (2)	C27—C28—C31	122.0 (2)
N4—C10—N2	123.1 (2)	N6—C29—C28	115.6 (2)
N1—C10—N2	108.87 (19)	N6—C29—C32	117.1 (2)

N4—C11—C12	124.1 (2)	C28—C29—C32	127.4 (2)
N4—C11—C14	114.6 (3)	C27—C30—H30A	109.5
C12—C11—C14	121.3 (3)	C27—C30—H30B	109.5
C13—C12—C11	119.3 (2)	H30A—C30—H30B	109.5
C13—C12—C15	120.6 (3)	C27—C30—H30C	109.5
C11—C12—C15	120.1 (3)	H30A—C30—H30C	109.5
N2—C13—C12	115.1 (2)	H30B—C30—H30C	109.5
N2—C13—C16	116.4 (2)	C28—C31—H31A	109.5
C12—C13—C16	128.4 (2)	C28—C31—H31B	109.5
C11—C14—H14A	109.5	H31A—C31—H31B	109.5
C11—C14—H14B	109.5	C28—C31—H31C	109.5
H14A—C14—H14B	109.5	H31A—C31—H31C	109.5
C11—C14—H14C	109.5	H31B—C31—H31C	109.5
H14A—C14—H14C	109.5	C29—C32—H32A	109.5
H14B—C14—H14C	109.5	C29—C32—H32B	109.5
C12—C15—H15A	109.5	H32A—C32—H32B	109.5
C12—C15—H15B	109.5	C29—C32—H32C	109.5
H15A—C15—H15B	109.5	H32A—C32—H32C	109.5
C12—C15—H15C	109.5	H32B—C32—H32C	109.5
H15A—C15—H15C	109.5	C10—N1—C9	102.63 (18)
H15B—C15—H15C	109.5	C10—N2—C13	123.1 (2)
C13—C16—H16A	109.5	C10—N2—N3	110.72 (17)
C13—C16—H16B	109.5	C13—N2—N3	126.16 (19)
H16A—C16—H16B	109.5	C9—N3—N2	101.02 (17)
C13—C16—H16C	109.5	C11—N4—C10	115.2 (2)
H16A—C16—H16C	109.5	C26—N5—C25	102.08 (18)
H16B—C16—H16C	109.5	C29—N6—C26	123.83 (18)
C18—C17—C22	119.63 (19)	C29—N6—N7	126.37 (18)
C18—C17—C23	119.25 (19)	C26—N6—N7	109.76 (17)
C22—C17—C23	121.12 (18)	C25—N7—N6	100.40 (16)
C19—C18—C17	120.9 (2)	C27—N8—C26	116.5 (2)
C19—C18—H18	119.6	C7—O1—H1	115.3 (16)
C17—C18—H18	119.6	C23—O4—H4A	112 (2)
C20—C19—C18	119.7 (2)	C9—S1—C8	101.72 (11)
C20—C19—H19	120.2	C25—S2—C24	102.45 (10)
C6—C1—C2—C3	−1.0 (3)	C27—C28—C29—C32	−177.9 (2)
C7—C1—C2—C3	178.9 (2)	C31—C28—C29—C32	2.6 (3)
C1—C2—C3—C4	0.0 (4)	N4—C10—N1—C9	178.1 (2)
C2—C3—C4—C5	1.2 (4)	N2—C10—N1—C9	−1.2 (2)
C3—C4—C5—C6	−1.4 (4)	N3—C9—N1—C10	1.6 (2)
C4—C5—C6—C1	0.4 (3)	S1—C9—N1—C10	−178.97 (15)
C4—C5—C6—C8	178.8 (2)	N4—C10—N2—C13	1.5 (3)
C2—C1—C6—C5	0.8 (3)	N1—C10—N2—C13	−179.1 (2)
C7—C1—C6—C5	−179.1 (2)	N4—C10—N2—N3	−178.8 (2)
C2—C1—C6—C8	−177.4 (2)	N1—C10—N2—N3	0.6 (2)
C7—C1—C6—C8	2.7 (3)	C12—C13—N2—C10	−3.2 (3)
C2—C1—C7—O2	24.1 (3)	C16—C13—N2—C10	175.5 (2)
C6—C1—C7—O2	−156.0 (3)	C12—C13—N2—N3	177.1 (2)
C2—C1—C7—O1	−154.6 (2)	C16—C13—N2—N3	−4.2 (3)

C6—C1—C7—O1	25.3 (3)	N1—C9—N3—N2	−1.2 (2)
C5—C6—C8—S1	−113.2 (2)	S1—C9—N3—N2	179.36 (15)
C1—C6—C8—S1	65.0 (2)	C10—N2—N3—C9	0.4 (2)
N4—C11—C12—C13	−0.4 (4)	C13—N2—N3—C9	−179.9 (2)
C14—C11—C12—C13	178.5 (3)	C12—C11—N4—C10	−1.4 (4)
N4—C11—C12—C15	179.5 (3)	C14—C11—N4—C10	179.6 (2)
C14—C11—C12—C15	−1.6 (4)	N1—C10—N4—C11	−178.4 (2)
C11—C12—C13—N2	2.6 (3)	N2—C10—N4—C11	0.9 (3)
C15—C12—C13—N2	−177.3 (2)	N8—C26—N5—C25	178.0 (2)
C11—C12—C13—C16	−175.9 (3)	N6—C26—N5—C25	−0.2 (2)
C15—C12—C13—C16	4.2 (4)	N7—C25—N5—C26	0.7 (2)
C22—C17—C18—C19	0.3 (3)	S2—C25—N5—C26	−177.89 (15)
C23—C17—C18—C19	−178.8 (2)	C28—C29—N6—C26	−1.6 (3)
C17—C18—C19—C20	−0.9 (3)	C32—C29—N6—C26	178.36 (19)
C18—C19—C20—C21	0.3 (4)	C28—C29—N6—N7	176.33 (18)
C19—C20—C21—C22	0.8 (4)	C32—C29—N6—N7	−3.7 (3)
C20—C21—C22—C17	−1.4 (3)	N5—C26—N6—C29	177.89 (18)
C20—C21—C22—C24	179.6 (2)	N8—C26—N6—C29	−0.4 (3)
C18—C17—C22—C21	0.8 (3)	N5—C26—N6—N7	−0.3 (2)
C23—C17—C22—C21	179.92 (19)	N8—C26—N6—N7	−178.66 (17)
C18—C17—C22—C24	179.73 (19)	N5—C25—N7—N6	−0.8 (2)
C23—C17—C22—C24	−1.1 (3)	S2—C25—N7—N6	177.61 (15)
C18—C17—C23—O3	158.2 (2)	C29—N6—N7—C25	−177.50 (19)
C22—C17—C23—O3	−21.0 (3)	C26—N6—N7—C25	0.7 (2)
C18—C17—C23—O4	−22.0 (3)	C28—C27—N8—C26	−1.4 (3)
C22—C17—C23—O4	158.90 (19)	C30—C27—N8—C26	177.56 (19)
C21—C22—C24—S2	−104.0 (2)	N5—C26—N8—C27	−176.1 (2)
C17—C22—C24—S2	77.1 (2)	N6—C26—N8—C27	1.9 (3)
N8—C27—C28—C29	−0.6 (3)	N3—C9—S1—C8	−3.9 (2)
C30—C27—C28—C29	−179.5 (2)	N1—C9—S1—C8	176.71 (16)
N8—C27—C28—C31	178.9 (2)	C6—C8—S1—C9	76.95 (18)
C30—C27—C28—C31	0.0 (3)	N7—C25—S2—C24	−8.8 (2)
C27—C28—C29—N6	2.0 (3)	N5—C25—S2—C24	169.62 (16)
C31—C28—C29—N6	−177.5 (2)	C22—C24—S2—C25	102.64 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···N1^{i}	0.82 (3)	1.90 (3)	2.721 (2)	175 (3)
O1—H1···N8^{ii}	0.99 (3)	1.79 (3)	2.762 (2)	168 (3)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*, −*y*+2, −*z*.

Fig. 1

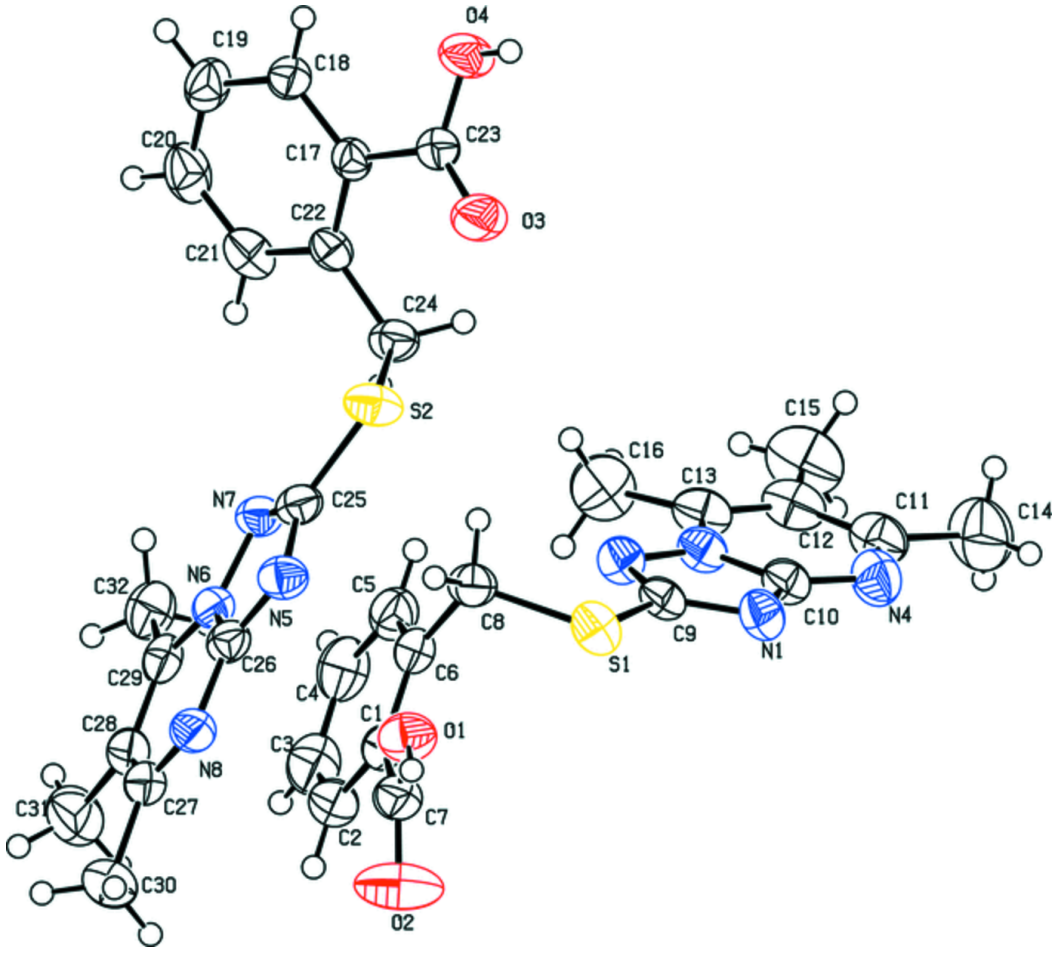

O4
C19
C18
C20
C17
C23
O3
C22
C21
C24
S2
C15
C16
C13
C11
C14
C12
N7
C25
N6
C32
N5
C5
C8
C10
N4
C9
N1
S1
C26
C29
C6
C4
C1
O1
C28
C3
N8
C27
C7
C31
C2
C30
O2

Fig. 2