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3-Hydroxy-*N'*-(*E*)-2-thienylmethylidene]-2-naphthohydrazide

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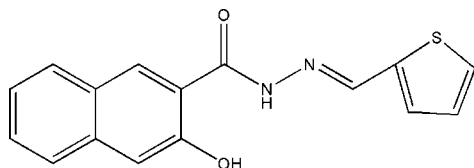
Received 10 December 2010; accepted 24 December 2010

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.055; wR factor = 0.078; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound, $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$, contains three independent molecules. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds in the three molecules lead to very similar conformations: the thiophene ring and naphthalene ring system in the three molecules form dihedral angles of 10.3 (2), 9.1 (2) and 9.3 (3)°. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains propagating in [031].

Related literature

For related structures, see: Huang (2009); Liang *et al.* (2008); Shafiq *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$ $M_r = 296.34$ Orthorhombic, $Pna2_1$ $a = 19.8191$ (16) Å $b = 6.2487$ (5) Å $c = 33.947$ (3) Å $V = 4204.1$ (6) Å³ $Z = 12$ Mo $K\alpha$ radiation $\mu = 0.24$ mm⁻¹ $T = 298$ K $0.41 \times 0.17 \times 0.06$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) $T_{\min} = 0.909$, $T_{\max} = 0.986$

20887 measured reflections

7258 independent reflections

3449 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.077$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.055$ $wR(F^2) = 0.078$ $S = 0.97$

7258 reflections

569 parameters

11 restraints

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.28$ e Å⁻³ $\Delta\rho_{\min} = -0.24$ e Å⁻³

Absolute structure: Flack (1983),

3473 Friedel pairs

Flack parameter: 0.01 (7)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O6}-\text{H51}\cdots\text{O2}$	0.82	2.03	2.771 (5)	149
$\text{N6}-\text{H54}\cdots\text{O6}$	0.86	1.96	2.641 (5)	135
$\text{N2}-\text{H2A}\cdots\text{O4}$	0.86	1.96	2.660 (5)	138
$\text{N4}-\text{H53}\cdots\text{O5}$	0.86	1.90	2.614 (5)	139
$\text{O5}-\text{H50}\cdots\text{O1}$	0.82	1.87	2.682 (5)	169
$\text{O4}-\text{H4}\cdots\text{O3}^i$	0.82	2.40	2.688 (5)	102

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5019).

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supplementary materials

Acta Cryst. (2011). E67, o277 [doi:10.1107/S160053681005405X]

3-Hydroxy-*N'*-[(*E*)-2-thienylmethylidene]-2-naphthohydrazide

Q. Wu, H. Yin and D. Wang

Comment

In continuation of our research of organotin derivatives, the title compound (I) - a novel Schiff base - have been synthesized. Herewith we present its crystal structure.

In (I), there are three independent molecules in the asymmetric unit (Fig. 1). All the bond lengths and angles are normal and correspond to those observed in the related compounds (Huang (2009); Liang *et al.* (2008); Shafiq *et al.* (2009)). Intramolecular N—H \cdots O hydrogen bonds (Table 1) lead to very close conformations - the thiophene ring and naphthalene bicycle in three molecules form dihedral angles of 9.67 (27) $^\circ$, 9.16 (29) $^\circ$ and 9.41 (26) $^\circ$, respectively.

In the crystal structure, intermolecular O—H \cdots O hydrogen bonds (Table 1) link the molecules into chains propagated in direction [031].

Experimental

The methanol solution of thiophene(0.1 mol) was added dropwise to a solution of 3-hydroxyl-2-naphthohydrazide(0.1 mol) in methanol. Then the mixture was stirred at room temperature for 6 h, during which time a yellow precipitate can be observed. The mixture was filtrated and the obtained solid was recrystallized from methanol. Yield 80%. *M.p.* 245 $^\circ$ C. Anal. Calcd (%) for C₁₆H₁₂N₂O₂S (Mr = 296.34): C, 64.84; H, 4.08; N, 9.45. Found (%): C, 64.82; H, 4.03; N, 9.49.

Refinement

All H atoms were placed in geometrically idealized positions (C—H=0.93 Å, O—H=0.82 Å, N—H=0.86 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{O}, \text{N})$.

Figures

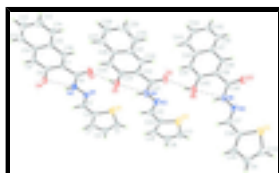


Fig. 1. Three independent molecules of (I) showing the atomic numbering and 50% probability displacement ellipsoids. The hydrogen bonds are shown as dashed lines.

3-Hydroxy-*N'*-[(*E*)-2-thienylmethylidene]-2-naphthohydrazide

Crystal data

C₁₆H₁₂N₂O₂S

$M_r = 296.34$

$D_x = 1.405 \text{ Mg m}^{-3}$

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

supplementary materials

Orthorhombic, $Pna2_1$

$a = 19.8191$ (16) Å

$b = 6.2487$ (5) Å

$c = 33.947$ (3) Å

$V = 4204.1$ (6) Å³

$Z = 12$

$F(000) = 1848$

Cell parameters from 2181 reflections

$\theta = 2.4\text{--}26.4^\circ$

$\mu = 0.24$ mm⁻¹

$T = 298$ K

Block, yellow

$0.41 \times 0.17 \times 0.06$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

phi and ω scans

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

$T_{\min} = 0.909$, $T_{\max} = 0.986$

20887 measured reflections

7258 independent reflections

3449 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.077$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.1^\circ$

$h = -23 \rightarrow 23$

$k = -7 \rightarrow 6$

$l = -40 \rightarrow 38$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.078$

$S = 0.97$

7258 reflections

569 parameters

11 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

$w = 1/[\sigma^2(F_o^2) + (0.0055P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.28$ e Å⁻³

$\Delta\rho_{\min} = -0.24$ e Å⁻³

Absolute structure: Flack (1983), 3473 Friedel pairs

Flack parameter: 0.01 (7)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C17	0.1250 (3)	1.3649 (9)	0.3320 (2)	0.0772 (16)
H17	0.1041	1.4728	0.3465	0.093*
C18	0.1214 (3)	1.3474 (9)	0.2938 (2)	0.0852 (18)
H18	0.0986	1.4477	0.2785	0.102*
C19	0.1535 (2)	1.1707 (8)	0.27682 (17)	0.0632 (15)
H19	0.1533	1.1341	0.2503	0.076*
C20	0.1862 (3)	1.0582 (9)	0.30730 (18)	0.0638 (14)
C21	0.2286 (3)	0.8714 (9)	0.30409 (19)	0.0645 (17)
H21	0.2388	0.8180	0.2792	0.077*
C22	0.3225 (3)	0.4907 (9)	0.35439 (19)	0.0496 (14)

C23	0.3623 (3)	0.2970 (8)	0.34149 (16)	0.0469 (14)
C24	0.3623 (3)	0.2105 (8)	0.30317 (18)	0.0533 (15)
C25	0.3998 (3)	0.0317 (8)	0.29514 (19)	0.0615 (17)
H25	0.3989	-0.0254	0.2698	0.074*
C26	0.4393 (3)	-0.0666 (9)	0.32362 (19)	0.0524 (16)
C27	0.4776 (3)	-0.2522 (10)	0.3153 (2)	0.076 (2)
H27	0.4769	-0.3107	0.2901	0.091*
C28	0.5149 (3)	-0.3436 (11)	0.3434 (2)	0.084 (2)
H28	0.5397	-0.4657	0.3375	0.101*
C29	0.5173 (3)	-0.2589 (13)	0.3819 (2)	0.092 (2)
H29	0.5430	-0.3244	0.4014	0.111*
C30	0.4813 (3)	-0.0799 (11)	0.3897 (2)	0.078 (2)
H30	0.4836	-0.0205	0.4148	0.094*
C31	0.4410 (3)	0.0179 (10)	0.36158 (19)	0.0547 (16)
C32	0.4021 (3)	0.1999 (9)	0.36920 (17)	0.0573 (16)
H32	0.4032	0.2579	0.3944	0.069*
C33	0.4184 (3)	1.8592 (9)	0.5359 (2)	0.080 (2)
H33	0.4346	1.9553	0.5545	0.096*
C34	0.4305 (3)	1.8749 (10)	0.4964 (2)	0.0744 (19)
H34	0.4572	1.9798	0.4848	0.089*
C35	0.3980 (2)	1.7149 (9)	0.47596 (17)	0.0592 (16)
H35	0.3994	1.7044	0.4486	0.071*
C36	0.3635 (2)	1.5730 (8)	0.49900 (16)	0.0495 (14)
C37	0.3271 (3)	1.3905 (8)	0.48448 (16)	0.0524 (15)
H37	0.3261	1.3596	0.4577	0.063*
C38	0.2277 (3)	0.9641 (9)	0.51485 (17)	0.0455 (15)
C39	0.1955 (2)	0.7777 (8)	0.49573 (14)	0.0381 (13)
C40	0.2039 (2)	0.7167 (8)	0.45615 (16)	0.0461 (14)
C41	0.1721 (3)	0.5405 (9)	0.44212 (16)	0.0573 (16)
H41	0.1794	0.5006	0.4161	0.069*
C42	0.1280 (3)	0.4142 (8)	0.46550 (17)	0.0492 (15)
C43	0.0939 (3)	0.2328 (9)	0.45136 (18)	0.0686 (18)
H43	0.0990	0.1920	0.4252	0.082*
C44	0.0541 (3)	0.1183 (9)	0.47519 (19)	0.0651 (17)
H44	0.0310	0.0003	0.4654	0.078*
C45	0.0470 (2)	0.1750 (9)	0.51481 (19)	0.0633 (17)
H45	0.0196	0.0923	0.5311	0.076*
C46	0.0790 (3)	0.3478 (8)	0.53006 (16)	0.0531 (15)
H46	0.0735	0.3849	0.5564	0.064*
C47	0.1208 (2)	0.4700 (8)	0.50523 (16)	0.0447 (14)
C48	0.1555 (2)	0.6543 (8)	0.51967 (15)	0.0448 (14)
H48	0.1508	0.6918	0.5460	0.054*
N3	0.2524 (2)	0.7774 (8)	0.33437 (14)	0.0580 (14)
N4	0.2909 (2)	0.5966 (7)	0.32609 (14)	0.0588 (13)
H53	0.2943	0.5528	0.3022	0.071*
N5	0.2959 (2)	1.2714 (7)	0.50920 (13)	0.0537 (12)
N6	0.2642 (2)	1.0966 (7)	0.49271 (13)	0.0504 (12)
H54	0.2680	1.0729	0.4679	0.061*
O2	0.31792 (19)	0.5390 (6)	0.38926 (11)	0.0695 (12)

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O3	0.21923 (17)	1.0000 (5)	0.55059 (11)	0.0628 (10)
O5	0.32458 (17)	0.3078 (5)	0.27397 (10)	0.0675 (11)
H50	0.3139	0.2188	0.2574	0.101*
O6	0.2452 (2)	0.8363 (6)	0.43242 (11)	0.0657 (11)
H51	0.2565	0.7649	0.4133	0.099*
S2	0.17219 (9)	1.1672 (3)	0.35224 (5)	0.0873 (5)
S3	0.36965 (8)	1.6406 (3)	0.54683 (5)	0.0731 (5)
C1	0.1105 (3)	0.8520 (9)	0.1823 (2)	0.078 (2)
H1	0.0887	0.9485	0.1988	0.094*
C2	0.1095 (3)	0.8610 (10)	0.1431 (2)	0.077 (2)
H2	0.0854	0.9638	0.1291	0.093*
C3	0.1482 (2)	0.7003 (8)	0.12479 (16)	0.0556 (16)
H3	0.1535	0.6864	0.0977	0.067*
C4	0.1772 (2)	0.5665 (8)	0.15220 (17)	0.0543 (15)
C5	0.2184 (2)	0.3795 (8)	0.14311 (17)	0.0520 (15)
H5	0.2278	0.3432	0.1171	0.062*
C6	0.3083 (3)	-0.0401 (8)	0.18524 (19)	0.0481 (15)
C7	0.3449 (2)	-0.2287 (8)	0.16877 (15)	0.0398 (13)
C8	0.3466 (2)	-0.2926 (8)	0.12870 (17)	0.0507 (16)
C9	0.3838 (3)	-0.4635 (8)	0.11708 (17)	0.0536 (15)
H9	0.3842	-0.5012	0.0906	0.064*
C10	0.4212 (3)	-0.5836 (8)	0.14371 (19)	0.0493 (15)
C11	0.4587 (3)	-0.7676 (10)	0.13163 (19)	0.0676 (18)
H11	0.4596	-0.8079	0.1053	0.081*
C12	0.4926 (3)	-0.8815 (9)	0.1585 (2)	0.0762 (19)
H12	0.5179	-0.9994	0.1506	0.091*
C13	0.4904 (3)	-0.8241 (10)	0.1989 (2)	0.0767 (19)
H13	0.5135	-0.9056	0.2174	0.092*
C14	0.4548 (3)	-0.6509 (9)	0.21041 (18)	0.0634 (18)
H14	0.4543	-0.6134	0.2369	0.076*
C15	0.4193 (2)	-0.5292 (8)	0.18397 (18)	0.0463 (15)
C16	0.3803 (2)	-0.3475 (8)	0.19406 (16)	0.0495 (14)
H16	0.3792	-0.3078	0.2205	0.059*
N2	0.27772 (19)	0.0878 (6)	0.15848 (14)	0.0506 (12)
H2A	0.2806	0.0598	0.1337	0.061*
O1	0.30712 (18)	-0.0019 (6)	0.22020 (12)	0.0651 (11)
S1	0.15723 (9)	0.6402 (2)	0.19813 (5)	0.0774 (5)
N1	0.2415 (2)	0.2660 (7)	0.17149 (14)	0.0509 (12)
O4	0.31007 (17)	-0.1791 (5)	0.10045 (10)	0.0603 (10)
H4	0.2714	-0.2252	0.0995	0.090*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C17	0.069 (4)	0.070 (4)	0.093 (3)	0.007 (3)	0.021 (4)	-0.023 (3)
C18	0.099 (5)	0.064 (4)	0.093 (3)	0.024 (3)	0.009 (4)	-0.010 (3)
C19	0.066 (4)	0.054 (4)	0.070 (2)	0.017 (3)	0.009 (3)	-0.004 (3)
C20	0.059 (4)	0.060 (4)	0.072 (3)	0.009 (2)	0.007 (3)	-0.009 (3)

supplementary materials

C21	0.069 (4)	0.067 (5)	0.058 (5)	-0.002 (3)	0.001 (4)	-0.006 (4)
C22	0.047 (4)	0.059 (4)	0.043 (4)	-0.010 (3)	0.003 (3)	-0.001 (4)
C23	0.046 (4)	0.056 (4)	0.038 (4)	-0.005 (3)	0.007 (3)	-0.007 (3)
C24	0.050 (4)	0.051 (4)	0.059 (4)	0.001 (3)	0.004 (3)	-0.002 (3)
C25	0.065 (4)	0.056 (4)	0.063 (4)	0.013 (3)	-0.007 (3)	-0.018 (3)
C26	0.044 (4)	0.047 (4)	0.067 (5)	0.001 (3)	0.005 (3)	0.005 (3)
C27	0.070 (5)	0.061 (4)	0.097 (6)	0.002 (3)	-0.012 (4)	0.007 (4)
C28	0.065 (5)	0.069 (5)	0.118 (7)	0.008 (4)	0.013 (5)	0.022 (5)
C29	0.073 (5)	0.107 (7)	0.097 (7)	0.001 (5)	-0.002 (5)	0.058 (6)
C30	0.066 (5)	0.090 (6)	0.077 (5)	-0.004 (4)	0.011 (4)	0.023 (4)
C31	0.047 (4)	0.064 (4)	0.053 (5)	-0.004 (3)	0.000 (3)	0.010 (4)
C32	0.056 (4)	0.066 (4)	0.051 (4)	-0.014 (3)	-0.003 (3)	0.002 (3)
C33	0.056 (4)	0.067 (4)	0.118 (7)	-0.016 (3)	-0.017 (4)	-0.015 (5)
C34	0.070 (5)	0.071 (5)	0.082 (5)	-0.011 (4)	-0.009 (4)	0.011 (4)
C35	0.059 (4)	0.063 (4)	0.057 (4)	-0.004 (3)	0.000 (3)	-0.002 (4)
C36	0.044 (3)	0.048 (4)	0.056 (4)	0.007 (3)	-0.002 (3)	0.006 (3)
C37	0.059 (4)	0.054 (4)	0.045 (4)	-0.001 (3)	0.005 (3)	-0.006 (3)
C38	0.043 (4)	0.053 (4)	0.041 (4)	0.009 (3)	-0.002 (3)	-0.006 (3)
C39	0.043 (3)	0.044 (3)	0.028 (3)	0.004 (2)	0.002 (3)	-0.010 (3)
C40	0.043 (4)	0.043 (4)	0.052 (4)	0.005 (3)	0.007 (3)	0.004 (3)
C41	0.061 (4)	0.067 (4)	0.044 (4)	-0.009 (3)	0.006 (3)	-0.021 (3)
C42	0.044 (4)	0.049 (4)	0.055 (4)	0.004 (3)	-0.006 (3)	-0.006 (3)
C43	0.065 (5)	0.075 (5)	0.065 (5)	-0.025 (3)	0.003 (4)	-0.009 (4)
C44	0.061 (4)	0.066 (5)	0.069 (5)	-0.008 (3)	-0.006 (4)	-0.009 (4)
C45	0.051 (4)	0.065 (5)	0.073 (5)	-0.009 (3)	0.008 (3)	0.009 (4)
C46	0.063 (4)	0.053 (4)	0.043 (4)	0.001 (3)	0.001 (3)	0.003 (3)
C47	0.041 (3)	0.042 (3)	0.051 (4)	0.009 (3)	-0.004 (3)	0.002 (3)
C48	0.057 (4)	0.041 (3)	0.037 (3)	0.018 (3)	0.000 (3)	-0.005 (3)
N3	0.058 (4)	0.050 (3)	0.066 (4)	-0.005 (3)	0.012 (3)	-0.019 (3)
N4	0.073 (4)	0.061 (4)	0.042 (3)	0.002 (3)	0.008 (3)	-0.018 (3)
N5	0.058 (3)	0.045 (3)	0.058 (4)	0.003 (2)	-0.004 (3)	-0.008 (3)
N6	0.064 (3)	0.047 (3)	0.040 (3)	-0.004 (2)	0.005 (3)	-0.012 (2)
O2	0.090 (3)	0.081 (3)	0.037 (3)	0.003 (2)	0.006 (2)	-0.014 (2)
O3	0.074 (3)	0.068 (3)	0.047 (3)	-0.0139 (19)	0.009 (2)	-0.011 (2)
O5	0.086 (3)	0.064 (3)	0.053 (3)	0.020 (2)	-0.007 (2)	-0.018 (2)
O6	0.079 (3)	0.072 (3)	0.045 (3)	-0.016 (2)	0.012 (2)	-0.017 (2)
S2	0.0958 (14)	0.0883 (13)	0.0779 (13)	0.0070 (10)	0.0068 (11)	-0.0152 (11)
S3	0.0846 (13)	0.0801 (12)	0.0545 (11)	-0.0074 (9)	-0.0003 (10)	-0.0064 (10)
C1	0.071 (5)	0.061 (5)	0.103 (7)	0.009 (3)	0.015 (4)	-0.018 (5)
C2	0.058 (4)	0.057 (5)	0.116 (7)	0.006 (3)	-0.003 (4)	-0.002 (5)
C3	0.062 (4)	0.055 (4)	0.050 (4)	-0.001 (3)	0.009 (3)	0.008 (3)
C4	0.053 (4)	0.040 (4)	0.069 (5)	0.000 (3)	0.008 (3)	-0.006 (3)
C5	0.059 (4)	0.044 (4)	0.052 (4)	-0.001 (3)	-0.002 (3)	-0.002 (3)
C6	0.047 (4)	0.043 (4)	0.055 (4)	-0.006 (3)	-0.001 (3)	-0.002 (3)
C7	0.046 (3)	0.041 (3)	0.032 (4)	0.000 (3)	-0.005 (3)	-0.003 (3)
C8	0.044 (4)	0.052 (4)	0.056 (4)	0.008 (3)	-0.006 (3)	0.004 (3)
C9	0.065 (4)	0.046 (4)	0.050 (4)	0.011 (3)	0.004 (3)	-0.016 (3)
C10	0.040 (4)	0.044 (4)	0.063 (5)	0.001 (3)	0.004 (3)	-0.002 (3)
C11	0.061 (4)	0.069 (5)	0.073 (5)	-0.006 (4)	0.001 (4)	-0.003 (4)

supplementary materials

C12	0.060 (5)	0.070 (5)	0.098 (6)	0.012 (3)	-0.003 (5)	-0.012 (5)
C13	0.062 (5)	0.066 (5)	0.102 (6)	0.003 (3)	-0.010 (4)	0.014 (4)
C14	0.057 (4)	0.053 (4)	0.080 (5)	0.014 (3)	0.003 (3)	-0.009 (4)
C15	0.038 (4)	0.043 (4)	0.058 (5)	0.003 (3)	-0.002 (3)	0.003 (3)
C16	0.047 (3)	0.057 (4)	0.045 (4)	-0.007 (3)	-0.005 (3)	-0.008 (3)
N2	0.061 (3)	0.049 (3)	0.042 (3)	0.002 (2)	-0.005 (3)	-0.009 (3)
O1	0.084 (3)	0.065 (3)	0.047 (3)	0.014 (2)	-0.002 (2)	-0.011 (2)
S1	0.0790 (12)	0.0771 (12)	0.0762 (12)	0.0032 (10)	0.0082 (10)	-0.0121 (11)
N1	0.057 (3)	0.039 (3)	0.057 (3)	0.007 (2)	0.004 (3)	-0.003 (2)
O4	0.065 (3)	0.069 (3)	0.048 (3)	0.010 (2)	0.006 (2)	-0.012 (2)

Geometric parameters (Å, °)

C17—C18	1.306 (8)	C42—C47	1.400 (7)
C17—S2	1.695 (6)	C43—C44	1.337 (7)
C17—H17	0.9300	C43—H43	0.9300
C18—C19	1.398 (6)	C44—C45	1.398 (7)
C18—H18	0.9300	C44—H44	0.9300
C19—C20	1.409 (7)	C45—C46	1.355 (6)
C19—H19	0.9300	C45—H45	0.9300
C20—C21	1.442 (7)	C46—C47	1.407 (6)
C20—S2	1.694 (6)	C46—H46	0.9300
C21—N3	1.274 (6)	C47—C48	1.427 (6)
C21—H21	0.9300	C48—H48	0.9300
C22—O2	1.225 (6)	N3—N4	1.392 (5)
C22—N4	1.323 (6)	N4—H53	0.8600
C22—C23	1.510 (7)	N5—N6	1.379 (5)
C23—C32	1.369 (6)	N6—H54	0.8600
C23—C24	1.408 (7)	O5—O1	2.682 (5)
C24—O5	1.382 (6)	O5—H50	0.8199
C24—C25	1.370 (6)	O6—H51	0.8200
C25—C26	1.387 (7)	C1—C2	1.332 (7)
C25—H25	0.9300	C1—S1	1.702 (6)
C26—C31	1.393 (7)	C1—H1	0.9300
C26—C27	1.415 (7)	C2—C3	1.409 (7)
C27—C28	1.336 (8)	C2—H2	0.9300
C27—H27	0.9300	C3—C4	1.376 (6)
C28—C29	1.409 (9)	C3—H3	0.9300
C28—H28	0.9300	C4—C5	1.459 (7)
C29—C30	1.353 (8)	C4—S1	1.673 (6)
C29—H29	0.9300	C5—N1	1.281 (6)
C30—C31	1.387 (7)	C5—H5	0.9300
C30—H30	0.9300	C6—O1	1.211 (6)
C31—C32	1.398 (6)	C6—N2	1.353 (6)
C32—H32	0.9300	C6—C7	1.493 (7)
C33—C34	1.365 (8)	C7—C16	1.334 (6)
C33—S3	1.713 (6)	C7—C8	1.418 (7)
C33—H33	0.9300	C8—C9	1.356 (6)
C34—C35	1.376 (7)	C8—O4	1.396 (5)

C34—H34	0.9300	C9—C10	1.389 (7)
C35—C36	1.365 (6)	C9—H9	0.9300
C35—H35	0.9300	C10—C15	1.409 (7)
C36—C37	1.437 (6)	C10—C11	1.430 (7)
C36—S3	1.682 (5)	C11—C12	1.338 (7)
C37—N5	1.280 (6)	C11—H11	0.9300
C37—H37	0.9300	C12—C13	1.418 (8)
C38—O3	1.245 (6)	C12—H12	0.9300
C38—N6	1.332 (6)	C13—C14	1.350 (7)
C38—C39	1.478 (6)	C13—H13	0.9300
C39—C48	1.373 (6)	C14—C15	1.370 (7)
C39—C40	1.406 (6)	C14—H14	0.9300
C40—C41	1.356 (6)	C15—C16	1.415 (6)
C40—O6	1.370 (5)	C16—H16	0.9300
C41—C42	1.420 (7)	N2—N1	1.397 (5)
C41—H41	0.9300	N2—H2A	0.8600
C42—C43	1.404 (7)	O4—H4	0.8200
C18—C17—S2	111.8 (5)	C45—C44—H44	119.8
C18—C17—H17	124.1	C46—C45—C44	121.5 (6)
S2—C17—H17	124.1	C46—C45—H45	119.3
C17—C18—C19	116.8 (6)	C44—C45—H45	119.3
C17—C18—H18	121.6	C45—C46—C47	118.7 (5)
C19—C18—H18	121.6	C45—C46—H46	120.7
C18—C19—C20	107.5 (6)	C47—C46—H46	120.7
C18—C19—H19	126.2	C42—C47—C46	120.1 (5)
C20—C19—H19	126.2	C42—C47—C48	118.9 (5)
C19—C20—C21	128.1 (6)	C46—C47—C48	121.0 (5)
C19—C20—S2	112.6 (4)	C39—C48—C47	121.8 (5)
C21—C20—S2	119.3 (5)	C39—C48—H48	119.1
N3—C21—C20	121.8 (6)	C47—C48—H48	119.1
N3—C21—H21	119.1	C21—N3—N4	114.5 (5)
C20—C21—H21	119.1	C22—N4—N3	121.2 (5)
O2—C22—N4	122.9 (5)	C22—N4—H53	119.4
O2—C22—C23	121.1 (5)	N3—N4—H53	119.4
N4—C22—C23	115.9 (5)	C37—N5—N6	114.5 (5)
C32—C23—C24	117.7 (5)	C38—N6—N5	120.7 (4)
C32—C23—C22	117.2 (5)	C38—N6—H54	119.6
C24—C23—C22	125.1 (5)	N5—N6—H54	119.6
O5—C24—C25	120.6 (5)	C24—O5—O1	103.9 (3)
O5—C24—C23	119.6 (5)	C24—O5—H50	109.6
C25—C24—C23	119.8 (6)	O1—O5—H50	7.9
C24—C25—C26	121.9 (6)	C40—O6—H51	109.5
C24—C25—H25	119.0	C17—S2—C20	91.1 (3)
C26—C25—H25	119.0	C36—S3—C33	91.8 (3)
C25—C26—C31	119.4 (5)	C2—C1—S1	110.9 (5)
C25—C26—C27	121.8 (6)	C2—C1—H1	124.6
C31—C26—C27	118.9 (6)	S1—C1—H1	124.6
C28—C27—C26	120.3 (7)	C1—C2—C3	113.7 (7)
C28—C27—H27	119.9	C1—C2—H2	123.1

supplementary materials

C26—C27—H27	119.9	C3—C2—H2	123.1
C27—C28—C29	121.4 (7)	C4—C3—C2	111.2 (6)
C27—C28—H28	119.3	C4—C3—H3	124.4
C29—C28—H28	119.3	C2—C3—H3	124.4
C30—C29—C28	118.3 (7)	C3—C4—C5	125.3 (5)
C30—C29—H29	120.8	C3—C4—S1	111.4 (4)
C28—C29—H29	120.8	C5—C4—S1	123.3 (5)
C29—C30—C31	122.2 (7)	N1—C5—C4	119.0 (5)
C29—C30—H30	118.9	N1—C5—H5	120.5
C31—C30—H30	118.9	C4—C5—H5	120.5
C30—C31—C26	118.9 (6)	O1—C6—N2	122.2 (5)
C30—C31—C32	123.3 (6)	O1—C6—C7	122.1 (5)
C26—C31—C32	117.8 (6)	N2—C6—C7	115.6 (5)
C23—C32—C31	123.4 (6)	C16—C7—C8	116.6 (5)
C23—C32—H32	118.3	C16—C7—C6	117.0 (5)
C31—C32—H32	118.3	C8—C7—C6	126.4 (5)
C34—C33—S3	111.7 (5)	C9—C8—O4	118.8 (5)
C34—C33—H33	124.2	C9—C8—C7	120.9 (5)
S3—C33—H33	124.2	O4—C8—C7	120.2 (5)
C33—C34—C35	111.1 (6)	C8—C9—C10	121.7 (5)
C33—C34—H34	124.4	C8—C9—H9	119.1
C35—C34—H34	124.4	C10—C9—H9	119.1
C36—C35—C34	114.6 (6)	C9—C10—C15	119.1 (5)
C36—C35—H35	122.7	C9—C10—C11	121.7 (6)
C34—C35—H35	122.7	C15—C10—C11	119.1 (5)
C35—C36—C37	124.8 (5)	C12—C11—C10	119.6 (6)
C35—C36—S3	110.7 (4)	C12—C11—H11	120.2
C37—C36—S3	124.5 (4)	C10—C11—H11	120.2
N5—C37—C36	118.6 (5)	C11—C12—C13	120.6 (6)
N5—C37—H37	120.7	C11—C12—H12	119.7
C36—C37—H37	120.7	C13—C12—H12	119.7
O3—C38—N6	120.7 (5)	C14—C13—C12	120.0 (6)
O3—C38—C39	120.8 (5)	C14—C13—H13	120.0
N6—C38—C39	118.5 (5)	C12—C13—H13	120.0
C48—C39—C40	118.8 (5)	C13—C14—C15	121.6 (6)
C48—C39—C38	115.6 (5)	C13—C14—H14	119.2
C40—C39—C38	125.6 (5)	C15—C14—H14	119.2
C41—C40—O6	121.0 (5)	C14—C15—C10	119.2 (5)
C41—C40—C39	120.0 (5)	C14—C15—C16	124.5 (6)
O6—C40—C39	119.0 (5)	C10—C15—C16	116.3 (5)
C40—C41—C42	122.8 (5)	C7—C16—C15	125.3 (5)
C40—C41—H41	118.6	C7—C16—H16	117.4
C42—C41—H41	118.6	C15—C16—H16	117.4
C43—C42—C47	118.8 (5)	C6—N2—N1	119.3 (5)
C43—C42—C41	123.6 (5)	C6—N2—H2A	120.4
C47—C42—C41	117.5 (5)	N1—N2—H2A	120.4
C44—C43—C42	120.6 (6)	C6—O1—O5	143.8 (4)
C44—C43—H43	119.7	C4—S1—C1	92.8 (3)
C42—C43—H43	119.7	C5—N1—N2	112.8 (5)

C43—C44—C45	120.4 (6)	C8—O4—H4	109.5
C43—C44—H44	119.8		
S2—C17—C18—C19	2.5 (8)	C38—C39—C48—C47	179.6 (4)
C17—C18—C19—C20	-3.3 (8)	C42—C47—C48—C39	-0.1 (7)
C18—C19—C20—C21	-176.7 (6)	C46—C47—C48—C39	-178.8 (5)
C18—C19—C20—S2	2.7 (6)	C20—C21—N3—N4	178.5 (5)
C19—C20—C21—N3	-176.1 (6)	O2—C22—N4—N3	2.8 (8)
S2—C20—C21—N3	4.5 (8)	C23—C22—N4—N3	179.8 (4)
O2—C22—C23—C32	-10.7 (8)	C21—N3—N4—C22	175.6 (5)
N4—C22—C23—C32	172.2 (5)	C36—C37—N5—N6	-178.1 (4)
O2—C22—C23—C24	170.0 (5)	O3—C38—N6—N5	3.0 (7)
N4—C22—C23—C24	-7.1 (8)	C39—C38—N6—N5	-179.2 (4)
C32—C23—C24—O5	-177.9 (5)	C37—N5—N6—C38	-177.3 (5)
C22—C23—C24—O5	1.4 (8)	C25—C24—O5—O1	21.7 (6)
C32—C23—C24—C25	1.7 (8)	C23—C24—O5—O1	-158.7 (4)
C22—C23—C24—C25	-179.0 (5)	C18—C17—S2—C20	-0.6 (6)
O5—C24—C25—C26	178.6 (5)	C19—C20—S2—C17	-1.3 (5)
C23—C24—C25—C26	-1.0 (8)	C21—C20—S2—C17	178.1 (5)
C24—C25—C26—C31	0.0 (8)	C35—C36—S3—C33	0.0 (4)
C24—C25—C26—C27	179.5 (5)	C37—C36—S3—C33	179.9 (4)
C25—C26—C27—C28	-179.7 (6)	C34—C33—S3—C36	-1.4 (5)
C31—C26—C27—C28	-0.2 (9)	S1—C1—C2—C3	-1.8 (8)
C26—C27—C28—C29	-0.2 (10)	C1—C2—C3—C4	1.5 (8)
C27—C28—C29—C30	-0.6 (10)	C2—C3—C4—C5	177.8 (5)
C28—C29—C30—C31	1.9 (10)	C2—C3—C4—S1	-0.4 (6)
C29—C30—C31—C26	-2.2 (9)	C3—C4—C5—N1	-178.9 (5)
C29—C30—C31—C32	178.4 (6)	S1—C4—C5—N1	-0.9 (7)
C25—C26—C31—C30	-179.1 (5)	O1—C6—C7—C16	-4.1 (8)
C27—C26—C31—C30	1.4 (8)	N2—C6—C7—C16	174.5 (5)
C25—C26—C31—C32	0.2 (8)	O1—C6—C7—C8	176.8 (5)
C27—C26—C31—C32	-179.3 (5)	N2—C6—C7—C8	-4.7 (7)
C24—C23—C32—C31	-1.5 (8)	C16—C7—C8—C9	-1.4 (7)
C22—C23—C32—C31	179.2 (5)	C6—C7—C8—C9	177.7 (5)
C30—C31—C32—C23	179.9 (5)	C16—C7—C8—O4	179.0 (4)
C26—C31—C32—C23	0.5 (8)	C6—C7—C8—O4	-1.9 (8)
S3—C33—C34—C35	2.3 (7)	O4—C8—C9—C10	-179.9 (5)
C33—C34—C35—C36	-2.4 (8)	C7—C8—C9—C10	0.5 (8)
C34—C35—C36—C37	-178.6 (5)	C8—C9—C10—C15	1.3 (8)
C34—C35—C36—S3	1.4 (6)	C8—C9—C10—C11	178.1 (5)
C35—C36—C37—N5	-178.9 (5)	C9—C10—C11—C12	-178.3 (5)
S3—C36—C37—N5	1.1 (7)	C15—C10—C11—C12	-1.6 (8)
O3—C38—C39—C48	1.5 (7)	C10—C11—C12—C13	1.4 (9)
N6—C38—C39—C48	-176.4 (5)	C11—C12—C13—C14	-1.2 (9)
O3—C38—C39—C40	-177.1 (5)	C12—C13—C14—C15	1.0 (9)
N6—C38—C39—C40	5.0 (7)	C13—C14—C15—C10	-1.2 (8)
C48—C39—C40—C41	0.8 (8)	C13—C14—C15—C16	179.2 (5)
C38—C39—C40—C41	179.3 (5)	C9—C10—C15—C14	178.3 (5)
C48—C39—C40—O6	-178.4 (4)	C11—C10—C15—C14	1.5 (8)
C38—C39—C40—O6	0.2 (7)	C9—C10—C15—C16	-2.1 (7)

supplementary materials

O6—C40—C41—C42	-178.9 (5)	C11—C10—C15—C16	-178.9 (4)
C39—C40—C41—C42	2.0 (8)	C8—C7—C16—C15	0.6 (7)
C40—C41—C42—C43	179.1 (5)	C6—C7—C16—C15	-178.6 (5)
C40—C41—C42—C47	-3.7 (8)	C14—C15—C16—C7	-179.2 (5)
C47—C42—C43—C44	1.5 (9)	C10—C15—C16—C7	1.2 (8)
C41—C42—C43—C44	178.7 (6)	O1—C6—N2—N1	-2.4 (8)
C42—C43—C44—C45	-1.4 (9)	C7—C6—N2—N1	179.1 (4)
C43—C44—C45—C46	1.0 (9)	N2—C6—O1—O5	-46.8 (9)
C44—C45—C46—C47	-0.8 (8)	C7—C6—O1—O5	131.6 (5)
C43—C42—C47—C46	-1.3 (7)	C24—O5—O1—C6	-136.0 (7)
C41—C42—C47—C46	-178.6 (5)	C3—C4—S1—C1	-0.5 (4)
C43—C42—C47—C48	180.0 (5)	C5—C4—S1—C1	-178.8 (5)
C41—C42—C47—C48	2.7 (7)	C2—C1—S1—C4	1.3 (5)
C45—C46—C47—C42	0.9 (7)	C4—C5—N1—N2	177.6 (4)
C45—C46—C47—C48	179.6 (5)	C6—N2—N1—C5	176.4 (5)
C40—C39—C48—C47	-1.7 (7)		

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O6—H51 \cdots O2	0.82	2.03	2.771 (5)	149
N6—H54 \cdots O6	0.86	1.96	2.641 (5)	135
N2—H2A \cdots O4	0.86	1.96	2.660 (5)	138
N4—H53 \cdots O5	0.86	1.90	2.614 (5)	139
O5—H50 \cdots O1	0.82	1.87	2.682 (5)	169
O4—H4 \cdots O3 ⁱ	0.82	2.40	2.688 (5)	102

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

Fig. 1

