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Synthesis, crystal structure and Hirshfeld surface analysis of 7-oxo-6-phenyl-6,7-dihydro-5*H*-thieno-[2,3-*f*]isoindole-8-carboxylic acid

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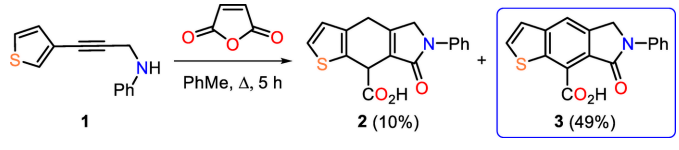
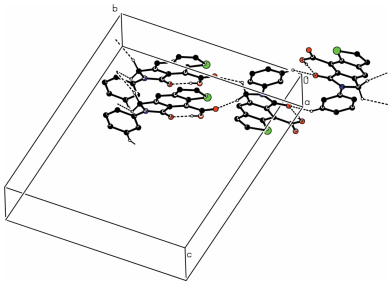
In the title compound, $C_{17}H_{11}NO_3S$, the thieno[2,3-*f*]isoindole ring system and the phenyl ring are oriented at a dihedral angle of 20.57 (13)°. The strong intramolecular O—H···O hydrogen bond partly ensures the coplanarity of the carboxyl group and the ring system. In crystal, the molecules are linked through C—H···O hydrogen bonds, enclosing $R_2^2(14)$ ring motifs, into a three-dimensional architecture. π–π interactions between parallel five-membered and phenyl rings [centroid-to-centroid distances of 3.564 (3) and 3.591 (3) Å] further contribute to the cohesion of the crystal structure. The Hirshfeld surface analysis indicates that the most important contributions for the crystal packing are from H···H (35.5%), H···O/O···H (21.3%), C···C (14.1%) and H···C/C···H (12.8%) interactions.

1. Chemical context

It is widely recognized that in the field of [4 + 2]-cycloaddition chemistry, the terms diene and dienophile are not limited to compounds with only double bonds. In the Diels–Alder reaction, alkynes can also serve as dienophiles, and conjugated 1,3-diynes or 1,3-enynes can act as dienes. Such pericyclic reactions are referred in the literature as dehydro-Diels–Alder reactions (Wessig *et al.*, 2008; Johnson, 2010; Ajaz *et al.*, 2011; Li *et al.*, 2016), and intramolecular dehydro-Diels–Alder (IMDDA) reactions are widely used in organic synthesis to construct polycyclic molecules (Hoye *et al.*, 2012; Brummond *et al.*, 2015; Wang *et al.*, 2016; Rana *et al.*, 2017; Krishna *et al.*, 2022). A special case of this type of transformation is the intramolecular didehydro-Diels–Alder reaction, in which an alkene dienophile reacts with an enyne. There are only a few publications related to the intramolecular dehydro-Diels–Alder reaction of the thiophene series that demonstrate the fundamental possibility of IMDDA transformations (Klemm *et al.*, 1965, 1966; Lu *et al.*, 2005; Bober *et al.*, 2017; Huang *et al.*, 2017).





This work is a continuation of studies on the tandem acylation/[4 + 2] cycloaddition reaction between 3-(thienyl) propargylamines and maleic anhydride as an example of the IMDDA approach (Shelukho *et al.*, 2025). Thienylpropargylamine **1** readily reacts with maleic anhydride to provide a mixture of products, where the product **2** has been published previously (Shelukho *et al.*, 2025), but the major acid **3** could not be isolated and characterized due to the formation of a mixture of products. In this work, we successfully isolated and characterized 7-oxo-6-phenyl-6,7-dihydro-5*H*-thieno[2,3-*f*] isoindole-8-carboxylic acid, **3**. The detection of acid **3** directly confirms the assumption that type **2** dihydroacid is prone to easy oxidation under aerobic conditions. Herein, we report the synthesis and molecular and crystal structure, together with the Hirshfeld surface analysis of the title compound, **3**.

2. Structural commentary

The asymmetric unit of the title compound, $C_{17}H_{11}NO_3S$, contains an essentially planar [r.m.s. deviation = 0.02 (4) Å] thieno[2,3-*f*]isoindole ring system (*A;* S1/C2/C3/C3*A*/C4/C4*A*/C5/N6/C7/C7*A*/C8/C8*A*), and a phenyl ring (*B*; C9–C14) oriented at a dihedral angle of 20.57 (13)°. The carboxyl group (C1/O2/O3/C8) is oriented at a dihedral angle of 0.22 (12)° with respect to the thieno[2,3-*f*]isoindole ring system. Thus, they are almost coplanar, partly as a result of the strong intramolecular O3—H3*O*···O1 hydrogen bond between the carboxyl hydrogen and isoindole oxygen atoms (Table 1, Fig. 1). On the other hand, the dihedral angle between the carboxylate group and ring *B* is 21.2 (4)°. Atom O1 is 0.037 (3) Å away from the best least-squares plane of the thieno[2,3-*f*]isoindole ring system. In the carboxylate group, the O2—C1 and O3—C1 bond lengths are 1.217 (6) and 1.303 (6) Å, respectively. Thus, the C—O bonds in the carboxylate group indicate mainly localized single and double bounds rather than a delocalized bonding arrangement. The O2—C1—O3 [120.9 (4)°] bond angle seems to be decreased compared to that present in a free acid (122.2°; Sim *et al.*, 1955) and compares with corresponding values of 122.42 (14)° in $C_{17}H_{13}NO_2$ (Refcode UYATIZ; Mague *et al.*, 2016), 122.55 (12)° in $C_{14}H_{11}NO_3$ (BIYJEC; El-Mrabet *et al.*, 2023) and 122.70 (12)° in $C_{12}H_{10}ClNO_3$ (PEDKAO; Filali Baba *et al.*, 2022). As indicated by the O2—C1—C8—C7*A* [179.9 (4)°] and O3—C1—C8—C8*A* [179.7 (4)°] torsion angles, the carboxyl group attached to the thieno[2,3-*f*]isoindole ring system is in a *anti* peripheral conformation.

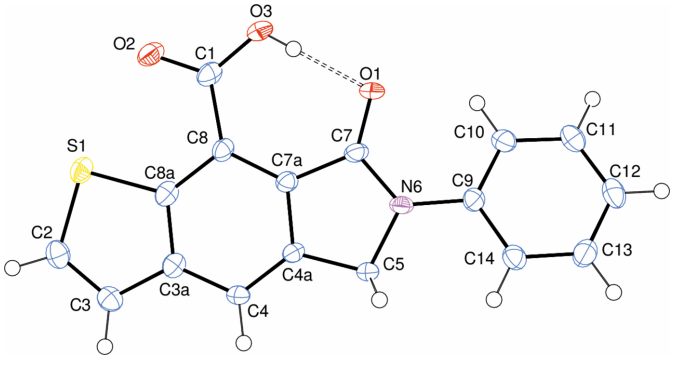


Figure 1
The asymmetric unit of the title compound with the atom-numbering scheme and 50% probability ellipsoids.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*O*···O1	0.74 (8)	1.76 (8)	2.496 (5)	175 (8)
C5—H5*A*···O2^{i}	0.99	2.37	3.343 (6)	167
C5—H5*B*···O2^{ii}	0.99	2.37	3.013 (6)	122
C11—H11···O1^{iii}	0.95	2.50	3.333 (6)	146

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

3. Supramolecular features

In the crystal, the molecules are linked through C—H···O hydrogen bonds, enclosing $R_2^2(14)$ ring motifs, into a three-dimensional architecture (Fig. 2). There are π–π interactions between the parallel five-membered (S1/C2/C3/C3*A*/C8*A* and N6/C5/C4*A*/C7*A*/C7) rings and the phenyl (C3*A*/C4/C4*A*/C7*A*/C8/C8*A*) ring with centroid-to-centroid distances of 3.564 (3) Å (α = 0.92° and slippage = 1.031 Å) and 3.591 (3) Å (α = 0.70° and slippage = 1.041 Å), respectively.

4. Hirshfeld surface analysis

To visualize the intermolecular interactions, a Hirshfeld surface (HS) analysis was carried out using *Crystal Explorer 17.5* (Spackman *et al.*, 2021). In the HS plotted over d_{norm} (Fig. 3), the contact distances equal, shorter and longer with respect to the sum of van der Waals radii are shown in white,

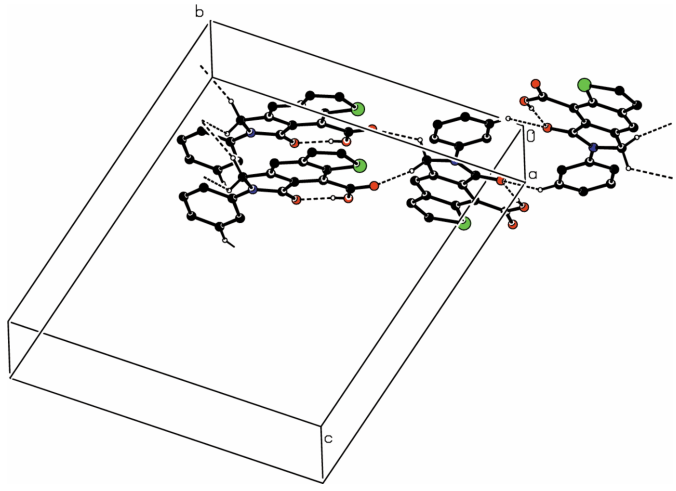


Figure 2
A partial packing diagram of the title compound viewed down the *a*-axis direction. Intramolecular O—H···O and intermolecular C—H···O hydrogen bonds are shown as dashed lines. H atoms not involved in these interactions have been omitted for clarity.

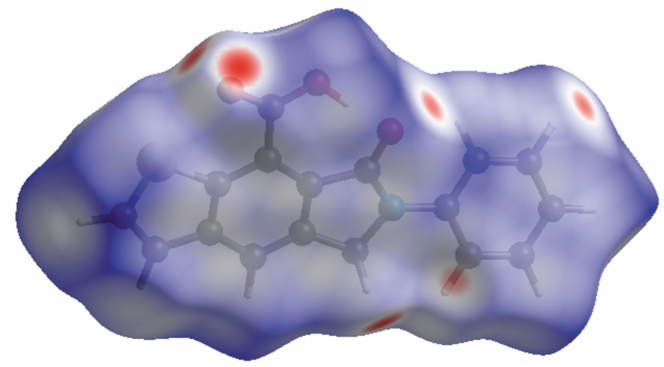


Figure 3
View of the three-dimensional Hirshfeld surface plotted over d_{norm}.

red and blue, respectively. According to the two-dimensional fingerprint plots, H···H, H···O/O···H, C···C and H···C/C···H contacts make the most important contributions to the HS (Fig. 4).

5. Synthesis and crystallization

Maleic anhydride (61.0 mg, 0.62 mmol) was added to *N*-(3-(thiophen-3-yl)prop-2-ynyl)aniline (**1**) (132.3 mg, 0.62 mmol) diluted in $PhCH_3$ (3 mL) at a 5 mL round-bottom flask. The resulting mixture was heated under reflux for 5 h, and then cooled to room temperature. The resulting precipitate was filtered, washed with PhMe (3 mL), Et_2O (2 × 3 mL), and air dried to give acid **2** (20.0 mg, 10%) as a colourless solid (for full characteristics see Shelukho *et al.*, 2025). After cooling mother liquor at 278 K, the precipitate was filtered, washed with mother liquor (3 mL), Et_2O (2 × 3 mL), and air dried to give the title compound **3** as yellow plates (yield 49%, 96.4 mg, m.p. > 523 K). IR (KBr), ν (cm^{-1}): 1704 (CO_2), 1591 (N—C═O). ^{1}H NMR (700.2 MHz, DMSO-d_6): δ (*J*, Hz) 16.45 (*br.s.*, 1H, CO_2H), 8.45 (*s*, 1H, H-Ar), 8.18 (*d*, *J* = 5.5 Hz, 1H, H-2 Thien), 7.91 (*d*, *J* = 7.6 Hz, 2H, H-Ar), 7.70 (*d*, *J* = 5.5 Hz, 1H, H-Thien), 7.56 (*t*, *J* = 7.6 Hz, 2H, H-Ar), 7.37 (*t*, *J* = 7.6 Hz, 1H, H-Ar), 5.33 (*s*, 2H, NCH_2) ppm. ^{13}C {^{1}H} NMR (176.1 MHz, DMSO-d_6): δ 169.1, 165.9, 1447, 142.3, 138.6, 138.1, 136.7, 129.7 (2C), 126.9, 126.8, 123.6, 123.4, 122.6, 122.2 (2C), 52.3 ppm. MS (ESI) *m*/*z*: $[M + H]^+$ 310. Elemental analysis calculated (%) for $C_{17}H_{11}NO_3S$: C 66.01, H 3.58, N 4.53, S 10.36; found: C 65.84, H 3.49, N 4.69, S 10.17.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{11}NO_3S$
M_r	309.33
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	100
a, *b*, *c* (Å)	3.89128 (10), 15.8505 (5), 21.6531 (7)
β (°)	93.596 (3)
V (Å^3)	1332.91 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.28
Crystal size (mm)	0.13 × 0.07 × 0.02
Data collection	
Diffractometer	Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2021).
T_{min}, T_{max}	0.857, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11945, 2775, 2269
R_{int}	0.119
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.097, 0.238, 1.03
No. of reflections	2775
No. of parameters	203
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.94, −0.48

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

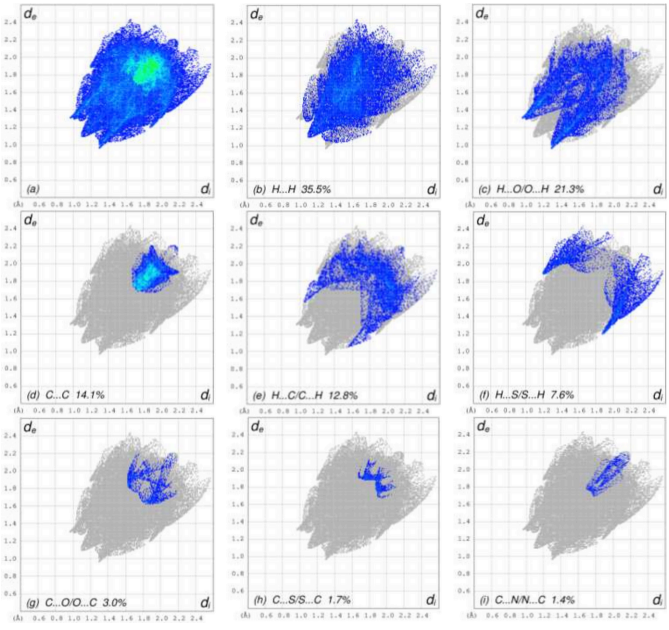


Figure 4
The full two-dimensional fingerprint plots showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) H···O/O···H, (*d*) C···C, (*e*) H···C/C···H, (*f*) H···S/S···H, (*g*) C···O/O···C, (*h*) C···S/S···C, (*i*) C···N/N···C, (*j*) O···S/S···O, (*k*) O···O, (*l*) H···N/N···H, (*m*) S···S and (*n*) N···N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxy hydrogen atom was located in a difference Fourier map and refined isotropically. The C-bound hydrogen-atom positions were calculated geometrically at distances of 0.95 Å (for aromatic CH) and 0.99 Å (for CH_2) and refined using a riding model by applying the constraint $U_{iso}(H) = 1.2U_{eq}(C)$.

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The authors' contributions are as follows. Conceptualization, TH and MHAD; synthesis, ERS and VPZ; X-ray analysis, VNK and NAG; Hirshfeld surface analysis, TH; writing (review and editing of the manuscript), TH, KIH and TAJ; supervision, TH and MHAD.

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Synthesis, crystal structure and Hirshfeld surface analysis of 7-oxo-6-phenyl-6,7-dihydro-5*H*-thieno[2,3-*f*]isoindole-8-carboxylic acid

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Computing details

7-Oxo-6-phenyl-6,7-dihydro-5*H*-thieno[2,3-*f*]isoindole-8-carboxylic acid

Crystal data

$C_{17}H_{11}NO_3S$
$M_r = 309.33$
Monoclinic, $P2_1/n$
$a = 3.89128$ (10) Å
$b = 15.8505$ (5) Å
$c = 21.6531$ (7) Å
$\beta = 93.596$ (3)°
$V = 1332.91$ (7) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.541$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3873 reflections
$\theta = 3.5$–$78.8°$
$\mu = 2.28$ mm^{-1}
$T = 100$ K
Plate, yellow
0.13 × 0.07 × 0.02 mm

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2021).
$T_{min} = 0.857$, $T_{max} = 1.000$
11945 measured reflections
2775 independent reflections
2269 reflections with $I > 2\sigma(I)$
$R_{int} = 0.119$
$\theta_{max} = 79.8°$, $\theta_{min} = 3.5°$
$h = -3 \rightarrow 4$
$k = -19 \rightarrow 19$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.097$
$wR(F^2) = 0.238$
$S = 1.03$
2775 reflections
203 parameters
0 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.088P)^2 + 6.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.94$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.1382 (3)	0.55415 (8)	0.87334 (5)	0.0250 (3)
O1	0.7474 (9)	0.5121 (2)	0.63096 (15)	0.0265 (8)
O2	0.4093 (9)	0.6550 (2)	0.79077 (16)	0.0276 (8)
O3	0.6243 (11)	0.6318 (2)	0.70111 (19)	0.0332 (9)
H3O	0.671 (19)	0.596 (5)	0.681 (4)	0.05 (2)*
C1	0.4827 (12)	0.6057 (3)	0.7506 (2)	0.0239 (10)
C2	−0.0144 (12)	0.4688 (3)	0.9129 (2)	0.0278 (10)
H2	−0.1150	0.4741	0.9516	0.033*
C3	0.0211 (12)	0.3938 (3)	0.8842 (2)	0.0249 (10)
H3	−0.0494	0.3413	0.9004	0.030*
C3A	0.1779 (11)	0.4030 (3)	0.8264 (2)	0.0216 (9)
C4	0.2556 (11)	0.3396 (3)	0.7848 (2)	0.0213 (9)
H4	0.2094	0.2821	0.7932	0.026*
C4A	0.4011 (11)	0.3632 (3)	0.7312 (2)	0.0181 (9)
C5	0.5110 (12)	0.3077 (3)	0.6796 (2)	0.0219 (9)
H5A	0.6944	0.2680	0.6945	0.026*
H5B	0.3141	0.2753	0.6607	0.026*
N6	0.6397 (9)	0.3689 (2)	0.63574 (17)	0.0204 (8)
C7	0.6339 (12)	0.4491 (3)	0.6577 (2)	0.0218 (9)
C7A	0.4790 (11)	0.4474 (3)	0.7184 (2)	0.0205 (9)
C8	0.4125 (11)	0.5128 (3)	0.7595 (2)	0.0209 (9)
C8A	0.2606 (11)	0.4884 (3)	0.8140 (2)	0.0219 (9)
C9	0.7463 (11)	0.3427 (3)	0.5766 (2)	0.0213 (9)
C10	0.7616 (12)	0.3990 (3)	0.5276 (2)	0.0272 (10)
H10	0.7054	0.4567	0.5330	0.033*
C11	0.8587 (13)	0.3705 (3)	0.4710 (2)	0.0292 (11)
H11	0.8698	0.4092	0.4377	0.035*
C12	0.9398 (12)	0.2871 (4)	0.4619 (2)	0.0297 (11)
H12	1.0073	0.2684	0.4228	0.036*
C13	0.9218 (13)	0.2306 (3)	0.5106 (2)	0.0299 (11)
H13	0.9781	0.1730	0.5046	0.036*
C14	0.8225 (12)	0.2572 (3)	0.5679 (2)	0.0249 (10)
H14	0.8065	0.2180	0.6008	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0238 (6)	0.0237 (6)	0.0280 (6)	0.0030 (4)	0.0046 (4)	−0.0064 (4)
O1	0.0360 (19)	0.0144 (16)	0.0301 (17)	−0.0061 (13)	0.0105 (14)	0.0027 (13)

O2	0.0290 (18)	0.0184 (16)	0.0359 (18)	0.0002 (13)	0.0047 (14)	−0.0047 (14)
O3	0.050 (2)	0.0135 (17)	0.038 (2)	−0.0018 (15)	0.0152 (17)	−0.0018 (15)
C1	0.019 (2)	0.019 (2)	0.034 (2)	0.0045 (17)	0.0034 (18)	−0.0033 (18)
C2	0.025 (2)	0.034 (3)	0.025 (2)	0.002 (2)	0.0050 (18)	0.000 (2)
C3	0.021 (2)	0.028 (3)	0.025 (2)	−0.0034 (19)	0.0008 (17)	0.0016 (19)
C3A	0.016 (2)	0.023 (2)	0.026 (2)	0.0028 (17)	0.0013 (16)	−0.0021 (18)
C4	0.024 (2)	0.016 (2)	0.024 (2)	−0.0030 (17)	0.0043 (17)	0.0020 (17)
C4A	0.016 (2)	0.015 (2)	0.024 (2)	−0.0001 (16)	0.0024 (15)	−0.0014 (16)
C5	0.027 (2)	0.015 (2)	0.024 (2)	−0.0025 (17)	0.0071 (17)	0.0010 (17)
N6	0.0211 (19)	0.0150 (18)	0.0255 (19)	−0.0024 (14)	0.0048 (14)	0.0036 (15)
C7	0.023 (2)	0.014 (2)	0.029 (2)	0.0023 (17)	0.0051 (17)	0.0021 (18)
C7A	0.020 (2)	0.017 (2)	0.024 (2)	0.0016 (17)	0.0041 (16)	0.0001 (17)
C8	0.018 (2)	0.020 (2)	0.025 (2)	0.0016 (17)	0.0037 (16)	−0.0037 (17)
C8A	0.014 (2)	0.023 (2)	0.028 (2)	0.0016 (17)	0.0018 (16)	−0.0050 (18)
C9	0.017 (2)	0.023 (2)	0.025 (2)	−0.0015 (17)	0.0050 (16)	−0.0021 (18)
C10	0.028 (3)	0.023 (2)	0.031 (2)	−0.0008 (19)	0.0091 (19)	0.003 (2)
C11	0.023 (2)	0.035 (3)	0.030 (2)	−0.003 (2)	0.0088 (18)	0.002 (2)
C12	0.023 (2)	0.041 (3)	0.026 (2)	−0.005 (2)	0.0077 (18)	−0.003 (2)
C13	0.032 (3)	0.027 (3)	0.031 (3)	0.002 (2)	0.004 (2)	−0.007 (2)
C14	0.023 (2)	0.028 (3)	0.025 (2)	−0.0024 (19)	0.0054 (18)	0.0005 (19)

Geometric parameters (Å, º)

S1—C2	1.726 (5)	C5—H5A	0.9900
S1—C8A	1.745 (5)	C5—H5B	0.9900
O1—C7	1.249 (6)	N6—C7	1.358 (6)
O2—C1	1.217 (6)	N6—C9	1.431 (6)
O3—C1	1.303 (6)	C7—C7A	1.479 (6)
O3—H3O	0.74 (8)	C7A—C8	1.402 (6)
C1—C8	1.512 (7)	C8—C8A	1.406 (6)
C2—C3	1.353 (7)	C9—C10	1.392 (7)
C2—H2	0.9500	C9—C14	1.402 (7)
C3—C3A	1.434 (6)	C10—C11	1.380 (7)
C3—H3	0.9500	C10—H10	0.9500
C3A—C4	1.396 (6)	C11—C12	1.376 (8)
C3A—C8A	1.420 (7)	C11—H11	0.9500
C4—C4A	1.373 (6)	C12—C13	1.387 (8)
C4—H4	0.9500	C12—H12	0.9500
C4A—C7A	1.400 (6)	C13—C14	1.388 (7)
C4A—C5	1.505 (6)	C13—H13	0.9500
C5—N6	1.467 (6)	C14—H14	0.9500

C2—S1—C8A	90.9 (2)	O1—C7—C7A	127.0 (4)
C1—O3—H3O	112 (6)	N6—C7—C7A	108.1 (4)
O2—C1—O3	120.9 (4)	C4A—C7A—C8	121.8 (4)
O2—C1—C8	118.8 (4)	C4A—C7A—C7	107.4 (4)
O3—C1—C8	120.3 (4)	C8—C7A—C7	130.7 (4)
C3—C2—S1	114.4 (4)	C7A—C8—C8A	115.7 (4)

C3—C2—H2	122.8	C7A—C8—C1	126.7 (4)
S1—C2—H2	122.8	C8A—C8—C1	117.7 (4)
C2—C3—C3A	111.9 (4)	C8—C8A—C3A	122.3 (4)
C2—C3—H3	124.1	C8—C8A—S1	127.0 (4)
C3A—C3—H3	124.1	C3A—C8A—S1	110.7 (3)
C4—C3A—C8A	120.1 (4)	C10—C9—C14	119.8 (4)
C4—C3A—C3	127.7 (4)	C10—C9—N6	121.7 (4)
C8A—C3A—C3	112.2 (4)	C14—C9—N6	118.4 (4)
C4A—C4—C3A	117.9 (4)	C11—C10—C9	119.7 (5)
C4A—C4—H4	121.1	C11—C10—H10	120.2
C3A—C4—H4	121.1	C9—C10—H10	120.2
C4—C4A—C7A	122.2 (4)	C12—C11—C10	121.3 (5)
C4—C4A—C5	128.3 (4)	C12—C11—H11	119.4
C7A—C4A—C5	109.5 (4)	C10—C11—H11	119.4
N6—C5—C4A	102.7 (4)	C11—C12—C13	119.2 (5)
N6—C5—H5A	111.2	C11—C12—H12	120.4
C4A—C5—H5A	111.2	C13—C12—H12	120.4
N6—C5—H5B	111.2	C12—C13—C14	120.9 (5)
C4A—C5—H5B	111.2	C12—C13—H13	119.5
H5A—C5—H5B	109.1	C14—C13—H13	119.5
C7—N6—C9	126.7 (4)	C13—C14—C9	119.1 (5)
C7—N6—C5	112.2 (4)	C13—C14—H14	120.4
C9—N6—C5	121.1 (4)	C9—C14—H14	120.4
O1—C7—N6	124.9 (4)		

C8A—S1—C2—C3	0.0 (4)	C7—C7A—C8—C1	1.0 (8)
S1—C2—C3—C3A	−0.4 (5)	O2—C1—C8—C7A	179.9 (4)
C2—C3—C3A—C4	179.7 (5)	O3—C1—C8—C7A	−1.2 (7)
C2—C3—C3A—C8A	0.7 (6)	O2—C1—C8—C8A	0.8 (6)
C8A—C3A—C4—C4A	−2.3 (6)	O3—C1—C8—C8A	179.7 (4)
C3—C3A—C4—C4A	178.8 (4)	C7A—C8—C8A—C3A	−0.3 (6)
C3A—C4—C4A—C7A	1.5 (7)	C1—C8—C8A—C3A	178.8 (4)
C3A—C4—C4A—C5	179.4 (4)	C7A—C8—C8A—S1	−178.6 (3)
C4—C4A—C5—N6	179.1 (4)	C1—C8—C8A—S1	0.5 (6)
C7A—C4A—C5—N6	−2.8 (5)	C4—C3A—C8A—C8	1.8 (7)
C4A—C5—N6—C7	3.5 (5)	C3—C3A—C8A—C8	−179.2 (4)
C4A—C5—N6—C9	−174.9 (4)	C4—C3A—C8A—S1	−179.7 (3)
C9—N6—C7—O1	−5.8 (8)	C3—C3A—C8A—S1	−0.6 (5)
C5—N6—C7—O1	175.8 (4)	C2—S1—C8A—C8	178.8 (4)
C9—N6—C7—C7A	175.4 (4)	C2—S1—C8A—C3A	0.3 (3)
C5—N6—C7—C7A	−3.0 (5)	C7—N6—C9—C10	−19.4 (7)
C4—C4A—C7A—C8	−0.1 (7)	C5—N6—C9—C10	158.8 (4)
C5—C4A—C7A—C8	−178.4 (4)	C7—N6—C9—C14	163.3 (4)
C4—C4A—C7A—C7	179.5 (4)	C5—N6—C9—C14	−18.5 (6)
C5—C4A—C7A—C7	1.2 (5)	C14—C9—C10—C11	−1.3 (7)
O1—C7—C7A—C4A	−177.7 (5)	N6—C9—C10—C11	−178.6 (4)
N6—C7—C7A—C4A	1.0 (5)	C9—C10—C11—C12	0.3 (8)
O1—C7—C7A—C8	1.8 (8)	C10—C11—C12—C13	0.3 (8)

N6—C7—C7A—C8	−179.5 (5)	C11—C12—C13—C14	0.2 (8)
C4A—C7A—C8—C8A	−0.5 (6)	C12—C13—C14—C9	−1.2 (7)
C7—C7A—C8—C8A	−179.9 (4)	C10—C9—C14—C13	1.8 (7)
C4A—C7A—C8—C1	−179.6 (4)	N6—C9—C14—C13	179.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*O*···O1	0.74 (8)	1.76 (8)	2.496 (5)	175 (8)
C5—H5*A*···O2^{i}	0.99	2.37	3.343 (6)	167
C5—H5*B*···O2^{ii}	0.99	2.37	3.013 (6)	122
C11—H11···O1^{iii}	0.95	2.50	3.333 (6)	146

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+3/2; (ii) −*x*+1/2, *y*−1/2, −*z*+3/2; (iii) −*x*+2, −*y*+1, −*z*+1.