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Ethyl 4-hydroxy-2,6-diphenyl-1-(2-thiomorpholinopropanoyl)-1,2,5,6-tetrahydropyridine-3-carboxylate

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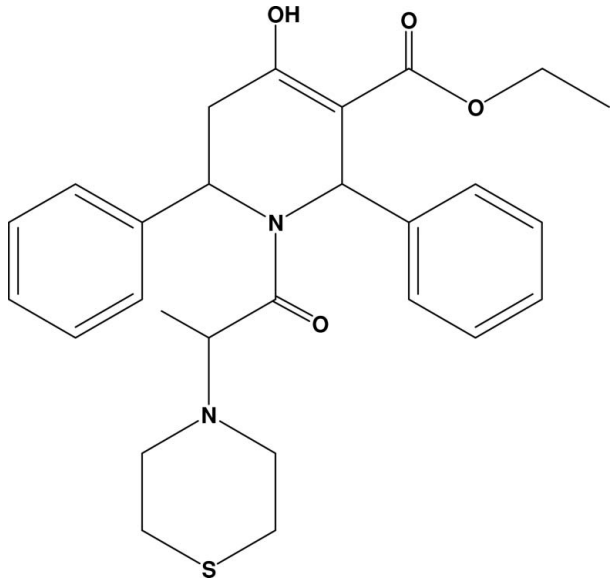
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.117; data-to-parameter ratio = 18.5.

In the title compound, $C_{27}H_{32}N_2O_4S$, the thiomorpholine ring adopts a chair conformation and the tetrahydropyridine ring is in a distorted envelope conformation. The molecular structure is stabilized by an intramolecular O—H···O interaction and the crystal packing is stabilized by an intermolecular C—H···O interaction, generating an $S(6)$ motif and a dimer of the type $R_2^2(18)$, respectively.

Related literature

For the synthesis and biological activity of 2,6-diarylpiperidin-4-one derivatives, see: Aridoss, Balasubramanian, Parthiban, Ramachandran & Kabilan (2007); Aridoss, Balasubramanian, Parthiban & Kabilan (2007); Aridoss, Parthiban *et al.* (2009). For a related structure, see: Aridoss, Gayathri *et al.* (2009). For ring conformational analysis, see: Cremer & Pople (1975); Nardelli (1983).



Experimental

Crystal data

$C_{27}H_{32}N_2O_4S$
M_r = 480.61
Triclinic, $P\overline{1}$
a = 9.904 (3) Å
b = 11.400 (4) Å
c = 12.103 (4) Å
α = 93.908 (18)°
β = 104.941 (15)°
γ = 106.819 (16)°
V = 1248.7 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.952, T_{max} = 0.968
26182 measured reflections
5707 independent reflections
4543 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.117
S = 1.01
5707 reflections
309 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3	0.82	1.85	2.560 (2)	144
C24—H24B···O3^{i}	0.96	2.54	3.285 (2)	135

Symmetry code: (i) $-x+2, -y, -z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2490).

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supporting information

Acta Cryst. (2009). E**65**, o3180–o3181 [doi:10.1107/S1600536809049186]

Ethyl 4-hydroxy-2,6-diphenyl-1-(2-thiomorpholinopropanoyl)-1,2,5,6-tetrahydropyridine-3-carboxylate

G. Aridoss, D. Gayathri, Keun Soo Park, Jong Tae Kim and Yeon Tae Jeong

S1. Comment

Our current research work is committed to find 2,6-diarylpiperidin-4-one based lead drug for the antimicrobial therapy and exploring the stereochemistry of its *N*-acyl derivatives (Aridoss, Balasubramanian, Parthiban, Ramachandran & Kabilan, 2007; Aridoss, Balasubramanian, Parthiban & Kabilan, 2007; Aridoss, Parthiban *et al.*, 2009). Recently we have disclosed the crystal structure of ethyl 1-(2-bromopropanoyl)-4-hydroxy-2,6-diphenyl-1,2,5,6- tetrahydropyridin-3-carboxylate (Aridoss, Gayathri *et al.*, 2009), which crystallizes with two independent molecules per asymmetric unit. Here, the tetrahydropyridine ring adopts a half-chair conformation in one molecule and distorted envelope conformation in other molecule. Thus to understand the change in conformation of the above said compound upon nucleophilic substitution of thiomorpholine in place of bromine, crystal structure of the title compound is determined by X-ray diffraction study and discussed in this paper.

The sum of the angles at N1 [358.6 (3)°] and N2 [336.1 (3)°] are in accordance with sp^2 and sp^3 hybridization, respectively. The dihedral angle between the two phenyl rings attached to the pyridine moiety is 21.8 (1)°. The thiomorpholine ring adopts chair conformation with atoms C9 and C11 deviating by 0.758 (2) and -0.673 (2) Å, respectively, from the least squares plane defined by atoms N2/C8/S1/C10. The tetrahydropyridine ring adops distorted envelope conformation. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for thiomorpholine and tetrahydropyridine rings are q_2 = 0.065 (1), 0.394 (1) Å, q_3 = 0.639 (2), 0.294 (1) Å; Q_T = 0.642 (2), 0.491 (1) Å and θ = 5.7 (1), 53.3 (2)°, respectively.

The molecular structure and the crystal packing are stabilized by O—H···O intramolecular and C—H···O intermolecular interactions, respectively, with atom O3 acting as bifurcated acceptor. In intramolecular interaction, atom O2 acts as a donor to O3 generating an S(6) motif and in intermolecular interaction, atom C24 acts as a donor to atom O3 at (2 - *x*, -*y*, -*z*), generating a dimer of the type $R_2^2(18)$.

S2. Experimental

To a solution of thiomorpholine (1 equiv.) and dry K_2CO_3 in benzene, ethyl 1-(2-bromopropanoyl)-4-hydroxy-2,6-diphenyl-1,2,5,6-tetrahydropyridin-3- carboxylate (1 equiv.; Aridoss, Gayathri *et al.*, 2009) in benzene was added slowly over a period of 15 minutes and refluxed over night. After the completion of reaction, the contents were poured into water and extracted twice with ethyl acetate. The combined organic extracts were then washed well with brine and dried over anhydrous sodium sulfate. This upon evaporation, column purification and subsequent recrystallization in distilled ethanol afforded fine white crystals appropriate for X-ray diffraction study.

S3. Refinement

All H atoms were refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic, C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3, and O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O) for the OH group.

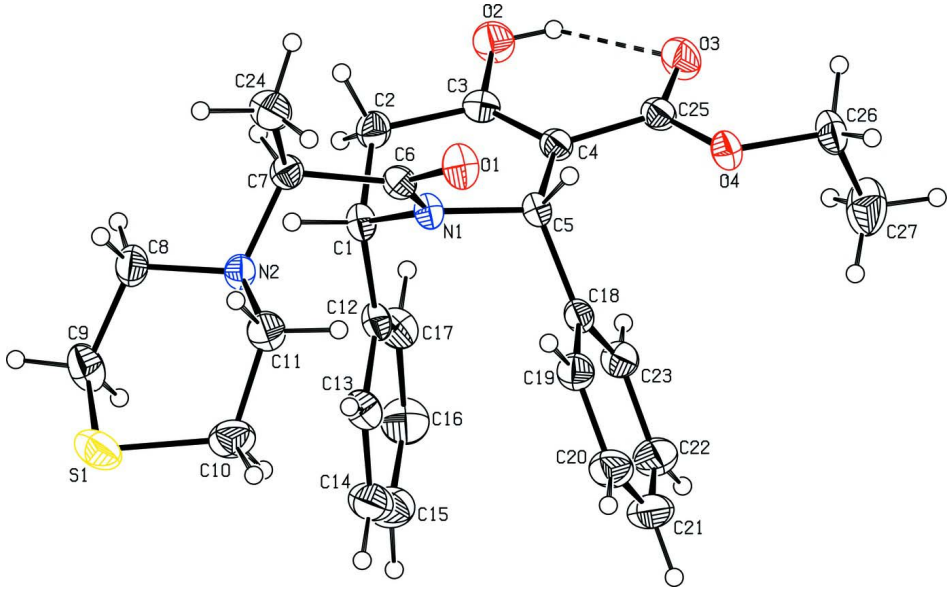

Figure 1

The molecular structure of title compound, showing 30% probability displacement ellipsoids.

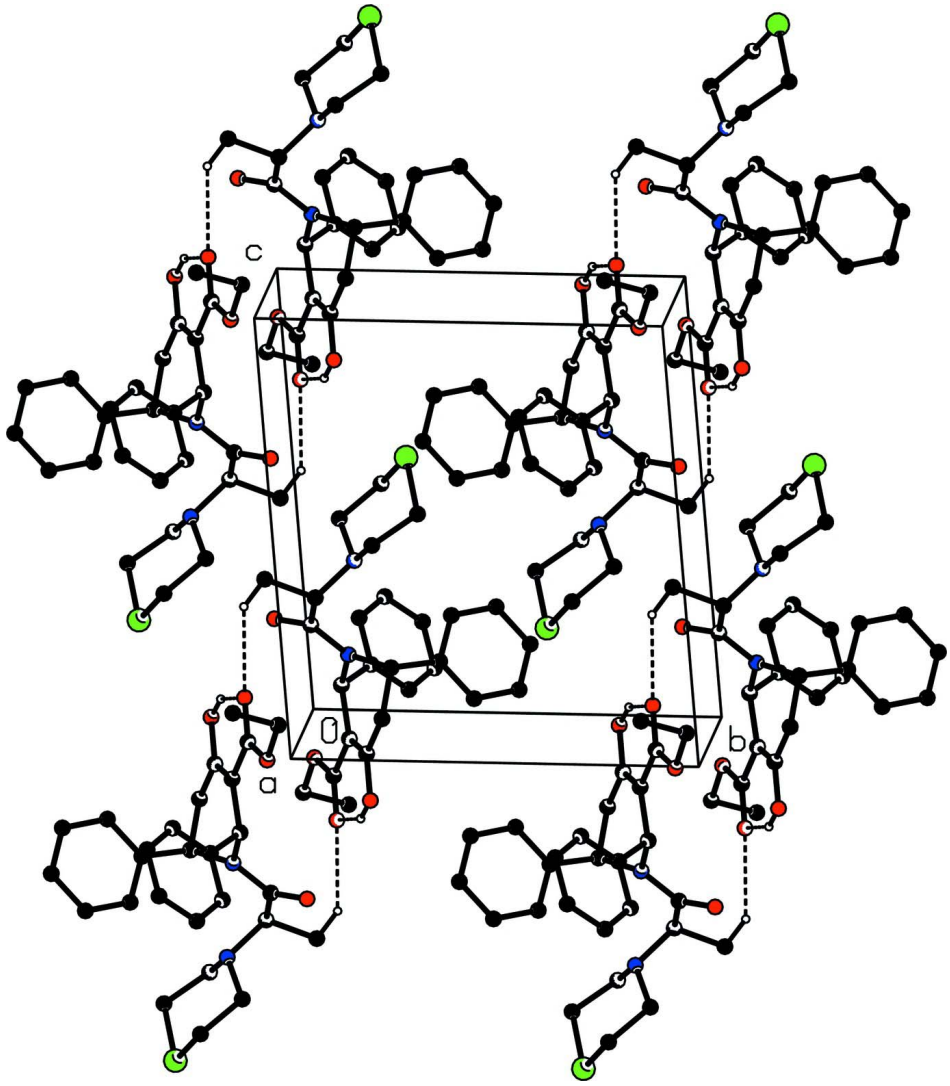

Figure 2

The molecular packing of (I). For clarity, hydrogen atoms which are not involved in hydrogen bonding are omitted.

Ethyl 4-hydroxy-2,6-diphenyl-1-(2-thiomorpholinopropanoyl)- 1,2,5,6-tetrahydropyridine-3-carboxylate

Crystal data

$C_{27}H_{32}N_2O_4S$
$M_r = 480.61$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.904$ (3) Å
$b = 11.400$ (4) Å
$c = 12.103$ (4) Å
$\alpha = 93.908$ (18)°
$\beta = 104.941$ (15)°
$\gamma = 106.819$ (16)°
$V = 1248.7$ (7) Å^3
$Z = 2$
$F(000) = 512$
$D_x = 1.278$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5338 reflections
$\theta = 2.3$–27.8°
$\mu = 0.17$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.952, T_{max} = 0.968
26182 measured reflections
5707 independent reflections
4543 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.8°, θ_{min} = 2.3°
h = −12→12
k = −14→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.117
S = 1.01
5707 reflections
309 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
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.2715P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 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}
C1	1.19148 (14)	0.27730 (12)	0.23357 (11)	0.0347 (3)
H1	1.2819	0.2883	0.2959	0.042*
C2	1.22176 (15)	0.24683 (13)	0.12043 (12)	0.0400 (3)
H2A	1.2817	0.3218	0.1013	0.048*
H2B	1.2769	0.1885	0.1293	0.048*
C3	1.08292 (15)	0.19255 (12)	0.02448 (11)	0.0371 (3)
C4	0.95026 (14)	0.14479 (11)	0.04268 (10)	0.0331 (3)
C5	0.93213 (13)	0.13505 (11)	0.16238 (10)	0.0310 (3)
H5	0.8828	0.0472	0.1626	0.037*
C6	1.10345 (14)	0.08311 (12)	0.31764 (10)	0.0337 (3)
C7	1.25989 (15)	0.11016 (13)	0.39709 (11)	0.0357 (3)
H7	1.3274	0.1358	0.3501	0.043*
C8	1.45086 (15)	0.27218 (15)	0.54032 (13)	0.0478 (4)
H8A	1.4994	0.2871	0.4801	0.057*
H8B	1.4889	0.2154	0.5853	0.057*
C9	1.4863 (2)	0.39292 (17)	0.61776 (16)	0.0611 (4)

H9A	1.4450	0.4485	0.5733	0.073*
H9B	1.5924	0.4315	0.6454	0.073*
C10	1.2279 (2)	0.29267 (17)	0.65584 (14)	0.0574 (4)
H10A	1.1697	0.2655	0.7082	0.069*
H10B	1.1886	0.3499	0.6126	0.069*
C11	1.21338 (17)	0.18191 (15)	0.57289 (12)	0.0457 (3)
H11A	1.2508	0.1239	0.6163	0.055*
H11B	1.1099	0.1405	0.5329	0.055*
C12	1.15095 (14)	0.39523 (12)	0.24545 (11)	0.0358 (3)
C13	1.12638 (16)	0.43177 (14)	0.34780 (13)	0.0448 (3)
H13	1.1291	0.3814	0.4051	0.054*
C14	1.0980 (2)	0.54136 (16)	0.36610 (16)	0.0577 (4)
H14	1.0809	0.5643	0.4352	0.069*
C15	1.0948 (2)	0.61727 (16)	0.28258 (18)	0.0640 (5)
H15	1.0773	0.6922	0.2955	0.077*
C16	1.1173 (2)	0.58217 (16)	0.18069 (17)	0.0608 (4)
H16	1.1135	0.6327	0.1235	0.073*
C17	1.14591 (18)	0.47184 (13)	0.16176 (13)	0.0471 (3)
H17	1.1618	0.4490	0.0921	0.057*
C18	0.83851 (13)	0.20571 (12)	0.19968 (11)	0.0332 (3)
C19	0.80193 (16)	0.18358 (14)	0.30119 (12)	0.0425 (3)
H19	0.8339	0.1262	0.3429	0.051*
C20	0.71920 (19)	0.24515 (16)	0.34109 (15)	0.0547 (4)
H20	0.6955	0.2294	0.4093	0.066*
C21	0.6719 (2)	0.32964 (16)	0.28039 (16)	0.0597 (4)
H21	0.6165	0.3720	0.3075	0.072*
C22	0.7062 (2)	0.35187 (16)	0.17939 (16)	0.0555 (4)
H22	0.6733	0.4090	0.1379	0.067*
C23	0.78906 (16)	0.29013 (13)	0.13889 (12)	0.0423 (3)
H23	0.8116	0.3057	0.0702	0.051*
C24	1.2782 (2)	−0.00907 (15)	0.43919 (14)	0.0524 (4)
H24A	1.3719	0.0096	0.4965	0.079*
H24B	1.2733	−0.0661	0.3751	0.079*
H24C	1.2008	−0.0457	0.4725	0.079*
C25	0.82265 (15)	0.08996 (12)	−0.05693 (11)	0.0369 (3)
C26	0.56361 (17)	−0.00482 (15)	−0.12584 (13)	0.0494 (4)
H26A	0.4891	−0.0668	−0.1038	0.059*
H26B	0.5820	−0.0430	−0.1922	0.059*
C27	0.5094 (2)	0.1005 (2)	−0.15733 (19)	0.0757 (6)
H27A	0.4967	0.1411	−0.0904	0.114*
H27B	0.4167	0.0701	−0.2167	0.114*
H27C	0.5797	0.1585	−0.1852	0.114*
N1	1.07728 (11)	0.16791 (10)	0.24818 (9)	0.0326 (2)
N2	1.29325 (11)	0.21563 (10)	0.48737 (9)	0.0336 (2)
O1	1.00568 (11)	−0.01107 (9)	0.31843 (9)	0.0454 (2)
O2	1.10662 (12)	0.19398 (11)	−0.07897 (9)	0.0520 (3)
H2	1.0283	0.1614	−0.1296	0.078*
O3	0.82742 (12)	0.08937 (11)	−0.15678 (8)	0.0539 (3)

O4	0.69839 (10)	0.03850 (9)	−0.03050 (8)	0.0406 (2)
S1	1.41530 (6)	0.37181 (4)	0.73873 (4)	0.06261 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0296 (6)	0.0326 (6)	0.0350 (6)	0.0045 (5)	0.0035 (5)	0.0060 (5)
C2	0.0337 (7)	0.0390 (7)	0.0460 (8)	0.0076 (5)	0.0137 (6)	0.0064 (6)
C3	0.0431 (7)	0.0354 (7)	0.0351 (7)	0.0139 (6)	0.0140 (6)	0.0047 (5)
C4	0.0358 (6)	0.0322 (6)	0.0299 (6)	0.0116 (5)	0.0070 (5)	0.0025 (5)
C5	0.0288 (6)	0.0298 (6)	0.0289 (6)	0.0052 (5)	0.0041 (5)	0.0032 (5)
C6	0.0370 (7)	0.0346 (7)	0.0286 (6)	0.0105 (5)	0.0090 (5)	0.0055 (5)
C7	0.0362 (7)	0.0409 (7)	0.0306 (6)	0.0146 (5)	0.0078 (5)	0.0069 (5)
C8	0.0320 (7)	0.0619 (10)	0.0434 (8)	0.0100 (7)	0.0079 (6)	0.0019 (7)
C9	0.0495 (9)	0.0547 (10)	0.0591 (10)	−0.0010 (8)	0.0049 (8)	−0.0027 (8)
C10	0.0655 (11)	0.0708 (11)	0.0464 (9)	0.0303 (9)	0.0250 (8)	0.0069 (8)
C11	0.0438 (8)	0.0530 (9)	0.0390 (7)	0.0094 (7)	0.0169 (6)	0.0063 (6)
C12	0.0307 (6)	0.0302 (6)	0.0368 (7)	0.0014 (5)	0.0032 (5)	0.0031 (5)
C13	0.0459 (8)	0.0404 (7)	0.0407 (7)	0.0057 (6)	0.0097 (6)	0.0036 (6)
C14	0.0615 (10)	0.0485 (9)	0.0599 (10)	0.0124 (8)	0.0215 (8)	−0.0046 (8)
C15	0.0691 (12)	0.0387 (9)	0.0863 (13)	0.0184 (8)	0.0259 (10)	0.0066 (8)
C16	0.0725 (11)	0.0420 (9)	0.0709 (11)	0.0196 (8)	0.0213 (9)	0.0216 (8)
C17	0.0546 (9)	0.0389 (8)	0.0446 (8)	0.0112 (7)	0.0125 (7)	0.0101 (6)
C18	0.0293 (6)	0.0327 (6)	0.0313 (6)	0.0049 (5)	0.0051 (5)	0.0003 (5)
C19	0.0429 (7)	0.0467 (8)	0.0379 (7)	0.0131 (6)	0.0130 (6)	0.0062 (6)
C20	0.0576 (10)	0.0613 (10)	0.0484 (9)	0.0173 (8)	0.0248 (8)	0.0013 (7)
C21	0.0619 (10)	0.0562 (10)	0.0693 (11)	0.0254 (8)	0.0285 (9)	−0.0019 (8)
C22	0.0611 (10)	0.0474 (9)	0.0641 (10)	0.0279 (8)	0.0167 (8)	0.0086 (8)
C23	0.0455 (8)	0.0413 (7)	0.0403 (7)	0.0150 (6)	0.0117 (6)	0.0060 (6)
C24	0.0642 (10)	0.0485 (9)	0.0429 (8)	0.0280 (8)	0.0018 (7)	0.0064 (7)
C25	0.0422 (7)	0.0358 (7)	0.0311 (6)	0.0146 (6)	0.0065 (5)	0.0017 (5)
C26	0.0395 (8)	0.0513 (9)	0.0411 (8)	0.0069 (6)	−0.0052 (6)	−0.0004 (6)
C27	0.0595 (11)	0.0746 (13)	0.0794 (13)	0.0234 (10)	−0.0081 (10)	0.0230 (11)
N1	0.0296 (5)	0.0308 (5)	0.0314 (5)	0.0059 (4)	0.0030 (4)	0.0054 (4)
N2	0.0291 (5)	0.0398 (6)	0.0302 (5)	0.0091 (4)	0.0077 (4)	0.0055 (4)
O1	0.0431 (5)	0.0415 (5)	0.0446 (5)	0.0043 (4)	0.0088 (4)	0.0161 (4)
O2	0.0516 (6)	0.0649 (7)	0.0388 (5)	0.0120 (5)	0.0204 (5)	0.0043 (5)
O3	0.0537 (6)	0.0723 (8)	0.0297 (5)	0.0162 (6)	0.0084 (4)	0.0000 (5)
O4	0.0352 (5)	0.0447 (5)	0.0327 (5)	0.0077 (4)	0.0010 (4)	0.0018 (4)
S1	0.0835 (3)	0.0571 (3)	0.0407 (2)	0.0286 (2)	0.0031 (2)	−0.00484 (18)

Geometric parameters (Å, º)

C1—N1	1.4773 (16)	C12—C13	1.382 (2)
C1—C2	1.5142 (19)	C13—C14	1.373 (2)
C1—C12	1.5182 (19)	C13—H13	0.9300
C1—H1	0.9800	C14—C15	1.375 (3)
C2—C3	1.4861 (19)	C14—H14	0.9300

C2—H2A	0.9700	C15—C16	1.363 (3)
C2—H2B	0.9700	C15—H15	0.9300
C3—O2	1.3323 (16)	C16—C17	1.384 (2)
C3—C4	1.3501 (19)	C16—H16	0.9300
C4—C25	1.4477 (18)	C17—H17	0.9300
C4—C5	1.5126 (17)	C18—C23	1.375 (2)
C5—N1	1.4665 (16)	C18—C19	1.3867 (19)
C5—C18	1.5202 (18)	C19—C20	1.374 (2)
C5—H5	0.9800	C19—H19	0.9300
C6—O1	1.2227 (16)	C20—C21	1.367 (3)
C6—N1	1.3609 (17)	C20—H20	0.9300
C6—C7	1.5281 (18)	C21—C22	1.371 (2)
C7—N2	1.4675 (18)	C21—H21	0.9300
C7—C24	1.526 (2)	C22—C23	1.380 (2)
C7—H7	0.9800	C22—H22	0.9300
C8—N2	1.4520 (18)	C23—H23	0.9300
C8—C9	1.507 (2)	C24—H24A	0.9600
C8—H8A	0.9700	C24—H24B	0.9600
C8—H8B	0.9700	C24—H24C	0.9600
C9—S1	1.782 (2)	C25—O3	1.2212 (17)
C9—H9A	0.9700	C25—O4	1.3310 (17)
C9—H9B	0.9700	C26—O4	1.4505 (16)
C10—C11	1.507 (2)	C26—C27	1.485 (3)
C10—S1	1.792 (2)	C26—H26A	0.9700
C10—H10A	0.9700	C26—H26B	0.9700
C10—H10B	0.9700	C27—H27A	0.9600
C11—N2	1.4647 (17)	C27—H27B	0.9600
C11—H11A	0.9700	C27—H27C	0.9600
C11—H11B	0.9700	O2—H2	0.8200
C12—C17	1.382 (2)		

N1—C1—C2	107.66 (11)	C12—C13—H13	119.5
N1—C1—C12	112.16 (11)	C13—C14—C15	120.20 (16)
C2—C1—C12	114.82 (11)	C13—C14—H14	119.9
N1—C1—H1	107.3	C15—C14—H14	119.9
C2—C1—H1	107.3	C16—C15—C14	119.63 (16)
C12—C1—H1	107.3	C16—C15—H15	120.2
C3—C2—C1	111.58 (11)	C14—C15—H15	120.2
C3—C2—H2A	109.3	C15—C16—C17	120.40 (16)
C1—C2—H2A	109.3	C15—C16—H16	119.8
C3—C2—H2B	109.3	C17—C16—H16	119.8
C1—C2—H2B	109.3	C12—C17—C16	120.56 (15)
H2A—C2—H2B	108.0	C12—C17—H17	119.7
O2—C3—C4	125.09 (13)	C16—C17—H17	119.7
O2—C3—C2	112.09 (12)	C23—C18—C19	118.54 (13)
C4—C3—C2	122.79 (12)	C23—C18—C5	123.75 (12)
C3—C4—C25	118.29 (12)	C19—C18—C5	117.71 (12)
C3—C4—C5	122.64 (11)	C20—C19—C18	120.98 (14)

C25—C4—C5	118.79 (11)	C20—C19—H19	119.5
N1—C5—C4	109.82 (10)	C18—C19—H19	119.5
N1—C5—C18	110.60 (10)	C21—C20—C19	119.88 (15)
C4—C5—C18	116.73 (10)	C21—C20—H20	120.1
N1—C5—H5	106.3	C19—C20—H20	120.1
C4—C5—H5	106.3	C20—C21—C22	119.83 (15)
C18—C5—H5	106.3	C20—C21—H21	120.1
O1—C6—N1	121.77 (12)	C22—C21—H21	120.1
O1—C6—C7	120.49 (12)	C21—C22—C23	120.49 (15)
N1—C6—C7	117.73 (11)	C21—C22—H22	119.8
N2—C7—C24	116.11 (11)	C23—C22—H22	119.8
N2—C7—C6	109.40 (10)	C18—C23—C22	120.26 (14)
C24—C7—C6	109.39 (12)	C18—C23—H23	119.9
N2—C7—H7	107.2	C22—C23—H23	119.9
C24—C7—H7	107.2	C7—C24—H24A	109.5
C6—C7—H7	107.2	C7—C24—H24B	109.5
N2—C8—C9	111.80 (13)	H24A—C24—H24B	109.5
N2—C8—H8A	109.3	C7—C24—H24C	109.5
C9—C8—H8A	109.3	H24A—C24—H24C	109.5
N2—C8—H8B	109.3	H24B—C24—H24C	109.5
C9—C8—H8B	109.3	O3—C25—O4	122.39 (12)
H8A—C8—H8B	107.9	O3—C25—C4	123.53 (13)
C8—C9—S1	112.18 (12)	O4—C25—C4	114.08 (11)
C8—C9—H9A	109.2	O4—C26—C27	110.13 (13)
S1—C9—H9A	109.2	O4—C26—H26A	109.6
C8—C9—H9B	109.2	C27—C26—H26A	109.6
S1—C9—H9B	109.2	O4—C26—H26B	109.6
H9A—C9—H9B	107.9	C27—C26—H26B	109.6
C11—C10—S1	112.47 (12)	H26A—C26—H26B	108.1
C11—C10—H10A	109.1	C26—C27—H27A	109.5
S1—C10—H10A	109.1	C26—C27—H27B	109.5
C11—C10—H10B	109.1	H27A—C27—H27B	109.5
S1—C10—H10B	109.1	C26—C27—H27C	109.5
H10A—C10—H10B	107.8	H27A—C27—H27C	109.5
N2—C11—C10	112.61 (13)	H27B—C27—H27C	109.5
N2—C11—H11A	109.1	C6—N1—C5	117.56 (10)
C10—C11—H11A	109.1	C6—N1—C1	125.05 (11)
N2—C11—H11B	109.1	C5—N1—C1	115.92 (10)
C10—C11—H11B	109.1	C8—N2—C11	112.46 (11)
H11A—C11—H11B	107.8	C8—N2—C7	111.81 (11)
C17—C12—C13	118.22 (14)	C11—N2—C7	111.79 (11)
C17—C12—C1	123.25 (13)	C3—O2—H2	109.5
C13—C12—C1	118.41 (12)	C25—O4—C26	116.77 (11)
C14—C13—C12	120.97 (15)	C9—S1—C10	95.98 (8)
C14—C13—H13	119.5		

N1—C1—C2—C3	48.04 (14)	C18—C19—C20—C21	0.0 (2)
C12—C1—C2—C3	−77.64 (14)	C19—C20—C21—C22	0.5 (3)

C1—C2—C3—O2	164.35 (11)	C20—C21—C22—C23	−0.4 (3)
C1—C2—C3—C4	−17.41 (18)	C19—C18—C23—C22	0.6 (2)
O2—C3—C4—C25	0.3 (2)	C5—C18—C23—C22	−179.08 (13)
C2—C3—C4—C25	−177.71 (12)	C21—C22—C23—C18	−0.2 (2)
O2—C3—C4—C5	174.15 (12)	C3—C4—C25—O3	−3.3 (2)
C2—C3—C4—C5	−3.9 (2)	C5—C4—C25—O3	−177.40 (13)
C3—C4—C5—N1	−7.76 (17)	C3—C4—C25—O4	176.05 (12)
C25—C4—C5—N1	166.06 (11)	C5—C4—C25—O4	1.95 (17)
C3—C4—C5—C18	119.13 (14)	O1—C6—N1—C5	−8.07 (18)
C25—C4—C5—C18	−67.04 (15)	C7—C6—N1—C5	172.90 (10)
O1—C6—C7—N2	−110.36 (14)	O1—C6—N1—C1	−173.66 (12)
N1—C6—C7—N2	68.68 (14)	C7—C6—N1—C1	7.31 (18)
O1—C6—C7—C24	17.86 (18)	C4—C5—N1—C6	−123.91 (12)
N1—C6—C7—C24	−163.10 (12)	C18—C5—N1—C6	105.82 (13)
N2—C8—C9—S1	64.32 (17)	C4—C5—N1—C1	42.99 (14)
S1—C10—C11—N2	−61.25 (16)	C18—C5—N1—C1	−87.27 (13)
N1—C1—C12—C17	−125.18 (14)	C2—C1—N1—C6	101.21 (14)
C2—C1—C12—C17	−1.86 (18)	C12—C1—N1—C6	−131.53 (13)
N1—C1—C12—C13	58.79 (15)	C2—C1—N1—C5	−64.58 (14)
C2—C1—C12—C13	−177.89 (12)	C12—C1—N1—C5	62.67 (14)
C17—C12—C13—C14	−0.1 (2)	C9—C8—N2—C11	−63.15 (17)
C1—C12—C13—C14	176.10 (14)	C9—C8—N2—C7	170.12 (13)
C12—C13—C14—C15	−0.5 (3)	C10—C11—N2—C8	61.90 (17)
C13—C14—C15—C16	1.1 (3)	C10—C11—N2—C7	−171.36 (12)
C14—C15—C16—C17	−1.1 (3)	C24—C7—N2—C8	75.03 (16)
C13—C12—C17—C16	0.2 (2)	C6—C7—N2—C8	−160.59 (11)
C1—C12—C17—C16	−175.88 (14)	C24—C7—N2—C11	−52.06 (16)
C15—C16—C17—C12	0.5 (3)	C6—C7—N2—C11	72.31 (14)
N1—C5—C18—C23	116.73 (13)	O3—C25—O4—C26	−7.80 (19)
C4—C5—C18—C23	−9.78 (18)	C4—C25—O4—C26	172.84 (11)
N1—C5—C18—C19	−62.99 (15)	C27—C26—O4—C25	−81.20 (18)
C4—C5—C18—C19	170.50 (11)	C8—C9—S1—C10	−55.24 (14)
C23—C18—C19—C20	−0.6 (2)	C11—C10—S1—C9	53.83 (14)
C5—C18—C19—C20	179.18 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.82	1.85	2.560 (2)	144
C24—H24*B*···O3^{i}	0.96	2.54	3.285 (2)	135

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