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5-(2-Hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)-3-methyl-2-(methylsulfanyl)-6-phenyl-7*H*-pyrrolo[2,3-*d*]pyrimidin-4(3*H*)-one monohydrate: complex sheets generated by multiple hydrogen bonds

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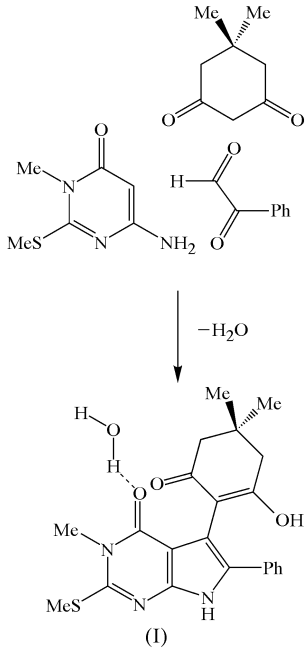
In the title compound, $C_{22}H_{23}N_3O_3S\cdot H_2O$, the non-aromatic carbocyclic ring adopts a half-chair conformation. The molecules are linked into complex sheets by a combination of one N—H···O hydrogen bond and three O—H···O hydrogen bonds.

Comment

We have recently reported the preparation of new fused heterocyclic pyrimidine derivatives, such as pyrimido[4,5-*b*]quinolines, by multicomponent reactions between 6-aminopyrimidine derivatives, 5,5-dimethylcyclohexane-1,3-dione (dimedone) and aryl aldehydes (Quiroga *et al.*, 2006). The extension of this method, with replacement of the aldehyde component by a glyoxal derivative (see scheme), has now provided the title pyrrolo[2,3-*d*]pyrimidine compound, (I) (Fig. 1), whose molecular and supramolecular structures are reported here.

The bond distances (Table 1) show evidence for strong bond fixation, both within the heterocyclic rings and in the non-aromatic carbocyclic ring; for the atom sequence C51–C56 within this ring, the ring-puckering parameters (Cremer & Pople, 1975) are θ = 52.2 (3)° and φ = 154.4 (4)°. These parameters are very close to the ideal values for the half-chair conformation, *viz.* θ = 50.8° and φ = (60*n* + 30)°. Atoms C5, C51, C52, C55 and C56 are almost coplanar, but atoms C53 and C54 deviate from this plane by 0.345 (2) and 0.352 (2) Å, respectively, on opposite sides of the reference plane. The aryl ring makes a dihedral angle of 16.0 (2)° with the pyrrole ring, while methyl atom C21 is almost coplanar with the adjacent pyrimidine ring.



(I)

Within the selected asymmetric unit (Fig. 1), the molecular components are linked by an O—H···O hydrogen bond. These two-component aggregates are linked into complex sheets by a combination of two further O—H···O hydrogen bonds and one N—H···O hydrogen bond (Table 2), each of which, considered in isolation, links pairs of aggregates into centrosymmetric motifs. Each pairwise combination of two

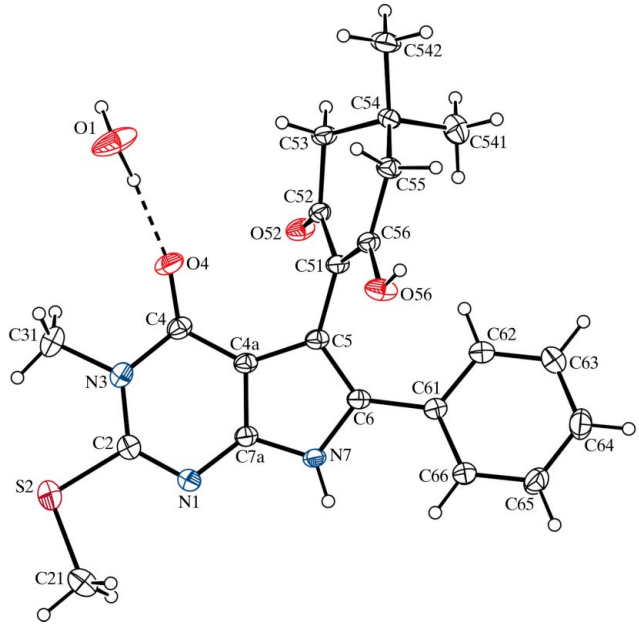


Figure 1
The independent molecular components of (I), showing the atom-labelling scheme and the O—H···O hydrogen bond within the selected asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level.

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such motifs generates a chain of edge-fused rings, and the combination of all three chains generates a complex sheet.

We analyse, firstly, the formation of the three finite zero-dimensional substructures, and then their combinations to form three one-dimensional substructures. Water atom O1 at (x, y, z) acts as a hydrogen-bond donor, *via* H1*B*, to carbonyl atom O52 at $(1-x, 1-y, 1-z)$, so generating by inversion an $R_4^4(20)$ (Bernstein *et al.*, 1995) ring centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, which we denote motif *A*. Hydroxy atom O56 at (x, y, z) acts as a hydrogen-bond donor to water atom O1 at $(-x, 1-y, 1-z)$, so generating by inversion a second and distinct $R_4^4(20)$ motif, this time centred at $(0, \frac{1}{2}, \frac{1}{2})$, which we denote motif *B*. Finally, pyrrole atom N7 at (x, y, z) acts as a hydrogen-bond donor to carbonyl atom O52 at $(-x, 1-y, -z)$, so generating by inversion an $R_2^2(14)$ motif centred at $(0, \frac{1}{2}, 0)$, denoted motif *C*.

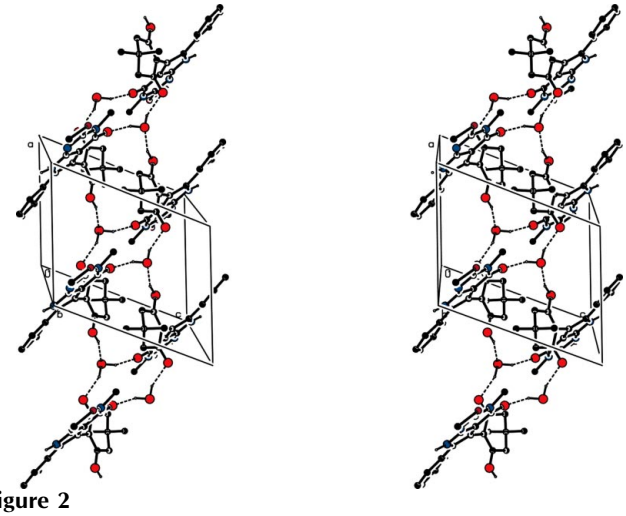

Figure 2
A stereoview of part of the crystal structure of (I), showing the formation of a chain of $R_4^4(20)$ rings along [100] and built from O—H···O hydrogen bonds only. For the sake of clarity, H atoms bonded to C atoms have been omitted.

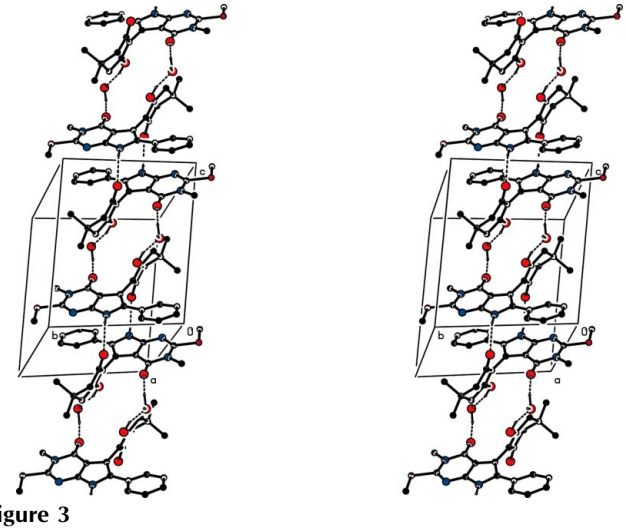

Figure 3
A stereoview of part of the crystal structure of (I), showing the formation of a chain of alternating $R_2^2(14)$ and $R_4^4(20)$ rings along [001] and built from O—H···O and N—H···O hydrogen bonds. For the sake of clarity, H atoms bonded to C atoms have been omitted.

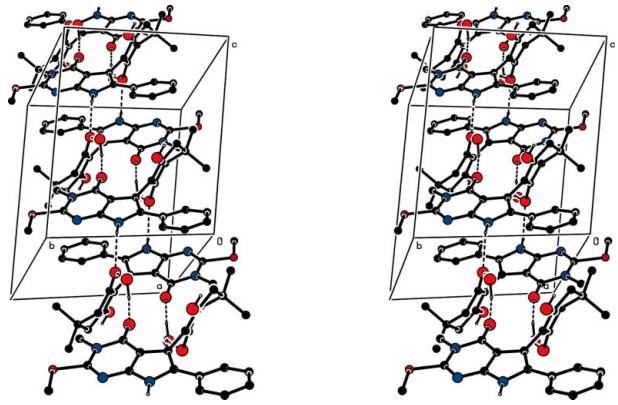

Figure 4
A stereoview of part of the crystal structure of (I), showing the formation of a chain of alternating $R_2^2(14)$ and $R_4^4(20)$ rings along [101] and built from O—H···O and N—H···O hydrogen bonds. For the sake of clarity, H atoms bonded to C atoms have been omitted.

The combination of motifs *A* and *B* generates a chain of edge-fused rings, containing two types of $R_4^4(20)$ ring, running parallel to the [100] direction (Fig. 2). The combination of motifs *B* and *C* generates a chain of alternating $R_2^2(14)$ and $R_4^4(20)$ rings running parallel to the [001] direction (Fig. 3). Finally, the combination of motifs *A* and *C* generates a second chain of $R_2^2(14)$ and $R_4^4(20)$ rings, this time running parallel to the [101] direction (Fig. 4). The combination of any two of the [100], [101] and [001] chains suffices to generate a sheet parallel to (010). There are no direction-specific interactions between adjacent sheets.

Experimental

Equimolar quantities (1 mmol of each component) of 6-amino-3-methyl-2-(methylsulfanyl)pyrimidin-4(3*H*)-one, 5,5-dimethylcyclohexane-1,3-dione and phenylglyoxal hydrate were mixed, and the mixture was then placed in an open Pyrex-glass vessel and irradiated in a domestic microwave oven for 5 min at 600 W. The product mixture was extracted with ethanol and, after removal of the solvent, the product was recrystallized from ethanol to give crystals of (I) suitable for single-crystal X-ray diffraction (m.p. 565–567 K, yield 45%). MS (EI 70 eV) *m*/*z* (%): 410 (27), 409 (M^+, 100), 395 (19), 394 (75), 311 (27), 284 (43), 264 (13), 236 (9), 88 (19).

Crystal data

$C_{22}H_{23}N_3O_3S\cdot H_2O$
$M_r = 427.51$
Triclinic, $P\overline{1}$
$a = 9.11880\ (12)$ Å
$b = 11.3095\ (2)$ Å
$c = 11.6526\ (2)$ Å
$\alpha = 97.5471\ (10)^\circ$
$\beta = 110.5868\ (10)^\circ$
$\gamma = 104.4677\ (11)^\circ$
$V = 1056.98\ (3)$ Å^3
$Z = 2$
$D_x = 1.343$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.19$ mm^{-1}
$T = 298\ (2)$ K
Lath, colourless
0.22 × 0.14 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.949$, $T_{max} = 0.982$
26102 measured reflections
4836 independent reflections
3874 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 0.7123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$(\Delta/\sigma)_{max} = 0.002$
$wR(F^2) = 0.144$	$\Delta\rho_{max} = 0.71$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.59$ e Å^{-3}
4836 reflections	
272 parameters	
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

N1—C2	1.302 (3)	C2—S2	1.762 (2)
C2—N3	1.381 (3)	S2—C21	1.786 (3)
N3—C4	1.414 (3)	C51—C52	1.446 (2)
C4—C4a	1.422 (3)	C52—C53	1.511 (2)
C4a—C5	1.427 (3)	C53—C54	1.534 (3)
C5—C6	1.382 (2)	C54—C55	1.532 (3)
C6—N7	1.395 (2)	C55—C56	1.494 (2)
N7—C7a	1.353 (2)	C56—C51	1.362 (2)
C7a—N1	1.365 (2)	C52—O52	1.236 (2)
C4a—C7a	1.386 (2)	C56—O56	1.328 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O4	0.98	1.76	2.688 (3)	157
O1—H1B···O52^{i}	0.98	1.90	2.762 (3)	145
N7—H7···O52^{ii}	0.86	2.24	2.974 (2)	143
O56—H56···O1^{iii}	0.82	1.78	2.560 (3)	159

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

Crystals of (I) are triclinic; the space group $P\overline{1}$ was selected and confirmed by the structure analysis. All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.93 (aromatic), 0.96 (CH_3) or 0.97 Å (CH_2), and O—H distances of 0.82 (hydroxy) or 0.98 Å (water), and with U_{iso}(H) = kU_{eq}(C,O), where k = 1.5 for O-bound and methyl H atoms, and 1.2 for all other H atoms.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC National X-ray Crystallography Service, University of Southampton, England. The authors thank the staff for all their help and advice. JC and JMT thank the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain), and the Universidad de Jaén for financial support; JMT also thanks the Universidad de Jaén for a scholarship grant supporting a short stay at the EPSRC National X-ray Crystallography Service. JQ and SC thank COLCIENCIAS, UNIVALLE (Universidad del Valle, Colombia) and UDENAR (Universidad de Nariño, Colombia) for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3043). Services for accessing these data are described at the back of the journal.

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supporting information

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Computing details

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Crystal data

$C_{22}H_{23}N_3O_3S\cdot H_2O$
$M_r = 427.51$
Triclinic$P\overline{1}$
Hall symbol: -P 1
$a = 9.11880\ (12)$ Å
$b = 11.3095\ (2)$ Å
$c = 11.6526\ (2)$ Å
$\alpha = 97.5471\ (10)°$
$\beta = 110.5868\ (10)°$
$\gamma = 104.4677\ (11)°$
$V = 1056.98\ (3)$ Å^3
$Z = 2$
$F(000) = 452$
$D_x = 1.343$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4836 reflections
$\theta = 3.6–27.5°$
$\mu = 0.19$ mm^{-1}
$T = 298$ K
Lath, colourless
0.22 × 0.14 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.949$, $T_{max} = 0.982$
26102 measured reflections
4836 independent reflections
3874 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.6°$
$h = -11\rightarrow11$
$k = -14\rightarrow14$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.144$
$S = 1.01$
4836 reflections
272 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
$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 0.7123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.1189 (2)	0.81065 (15)	0.11953 (16)	0.0356 (4)
C2	0.2635 (3)	0.86395 (19)	0.2121 (2)	0.0376 (4)
S2	0.37205 (8)	1.02277 (6)	0.23139 (7)	0.0575 (2)
C21	0.2266 (4)	1.0633 (2)	0.1072 (3)	0.0620 (7)
N3	0.3391 (2)	0.80628 (16)	0.30260 (16)	0.0380 (4)
C31	0.5122 (3)	0.8652 (2)	0.3939 (2)	0.0544 (6)
C4	0.2596 (2)	0.6850 (2)	0.31056 (18)	0.0345 (4)
O4	0.33147 (19)	0.64058 (16)	0.39625 (14)	0.0490 (4)
C4A	0.0998 (2)	0.62665 (18)	0.21189 (17)	0.0294 (4)
C5	−0.0234 (2)	0.50737 (18)	0.17936 (17)	0.0281 (4)
C51	−0.0167 (2)	0.41820 (17)	0.26267 (17)	0.0287 (4)
C52	0.1147 (2)	0.36266 (17)	0.29283 (17)	0.0287 (4)
O52	0.20445 (16)	0.36641 (14)	0.23397 (13)	0.0382 (3)
C53	0.1429 (2)	0.30061 (19)	0.40243 (19)	0.0347 (4)
C54	−0.0187 (2)	0.22362 (18)	0.40657 (19)	0.0330 (4)
C541	−0.1082 (3)	0.1113 (2)	0.2922 (2)	0.0529 (6)
C542	0.0184 (3)	0.1774 (2)	0.5281 (2)	0.0505 (6)
C55	−0.1254 (2)	0.30910 (19)	0.40595 (19)	0.0341 (4)
C56	−0.1262 (2)	0.39428 (17)	0.31816 (17)	0.0296 (4)
O56	−0.24067 (18)	0.45137 (15)	0.29545 (14)	0.0431 (4)
C6	−0.1488 (2)	0.50179 (17)	0.06727 (17)	0.0285 (4)
C61	−0.3104 (2)	0.40672 (17)	−0.00802 (17)	0.0287 (4)
C62	−0.3469 (3)	0.2851 (2)	0.0104 (2)	0.0399 (5)
C63	−0.5036 (3)	0.1991 (2)	−0.0531 (2)	0.0485 (5)
C64	−0.6270 (3)	0.2314 (2)	−0.1374 (2)	0.0501 (6)
C65	−0.5923 (3)	0.3491 (2)	−0.1607 (2)	0.0479 (5)
C66	−0.4359 (2)	0.43556 (19)	−0.0971 (2)	0.0383 (5)
N7	−0.10567 (18)	0.61512 (14)	0.03326 (14)	0.0296 (3)
C7A	0.0434 (2)	0.69055 (18)	0.12059 (17)	0.0294 (4)
O1	0.4787 (2)	0.6388 (3)	0.64016 (18)	0.0838 (8)
H21A	0.2717	1.1497	0.1074	0.093*
H21B	0.1261	1.0518	0.1201	0.093*
H21C	0.2038	1.0102	0.0276	0.093*
H31C	0.5735	0.8082	0.3893	0.082*

H31B	0.5156	0.8845	0.4777	0.082*
H31A	0.5603	0.9413	0.3741	0.082*
H53A	0.2063	0.3650	0.4807	0.042*
H53B	0.2076	0.2457	0.3962	0.042*
H54A	−0.1313	0.1407	0.2162	0.079*
H54B	−0.2101	0.0636	0.2942	0.079*
H54C	−0.0397	0.0588	0.2943	0.079*
H54D	0.0749	0.2483	0.5998	0.076*
H54E	0.0868	0.1248	0.5304	0.076*
H54F	−0.0833	0.1300	0.5305	0.076*
H55A	−0.2381	0.2567	0.3831	0.041*
H55B	−0.0862	0.3602	0.4910	0.041*
H56	−0.2987	0.4295	0.3339	0.065*
H62	−0.2643	0.2615	0.0665	0.048*
H63	−0.5256	0.1189	−0.0387	0.058*
H64	−0.7332	0.1741	−0.1783	0.060*
H65	−0.6744	0.3705	−0.2196	0.057*
H66	−0.4142	0.5147	−0.1142	0.046*
H7	−0.1646	0.6341	−0.0329	0.036*
H1A	0.4434	0.6274	0.5486	0.126*
H1B	0.5693	0.6029	0.6611	0.126*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0341 (9)	0.0334 (9)	0.0373 (9)	0.0087 (7)	0.0118 (7)	0.0135 (7)
C2	0.0364 (10)	0.0356 (10)	0.0388 (11)	0.0089 (8)	0.0141 (9)	0.0100 (9)
S2	0.0515 (4)	0.0375 (3)	0.0631 (4)	0.0010 (3)	0.0090 (3)	0.0105 (3)
C21	0.0691 (17)	0.0419 (13)	0.0693 (17)	0.0129 (12)	0.0215 (14)	0.0221 (12)
N3	0.0319 (8)	0.0402 (9)	0.0338 (9)	0.0079 (7)	0.0071 (7)	0.0067 (7)
C31	0.0378 (12)	0.0564 (15)	0.0457 (13)	0.0042 (11)	0.0003 (10)	0.0041 (11)
C4	0.0327 (10)	0.0443 (11)	0.0278 (9)	0.0133 (9)	0.0120 (8)	0.0115 (8)
O4	0.0433 (8)	0.0625 (10)	0.0339 (8)	0.0164 (8)	0.0039 (7)	0.0216 (7)
C4A	0.0283 (9)	0.0367 (10)	0.0285 (9)	0.0130 (8)	0.0135 (7)	0.0139 (8)
C5	0.0260 (8)	0.0362 (10)	0.0296 (9)	0.0146 (7)	0.0141 (7)	0.0144 (8)
C51	0.0271 (9)	0.0344 (9)	0.0289 (9)	0.0129 (7)	0.0113 (7)	0.0155 (8)
C52	0.0242 (8)	0.0326 (9)	0.0296 (9)	0.0094 (7)	0.0091 (7)	0.0121 (7)
O52	0.0319 (7)	0.0566 (9)	0.0381 (8)	0.0231 (6)	0.0177 (6)	0.0220 (7)
C53	0.0286 (9)	0.0424 (11)	0.0375 (10)	0.0157 (8)	0.0112 (8)	0.0209 (9)
C54	0.0314 (9)	0.0306 (9)	0.0380 (10)	0.0092 (8)	0.0124 (8)	0.0168 (8)
C541	0.0554 (14)	0.0361 (12)	0.0583 (15)	0.0087 (10)	0.0180 (12)	0.0070 (11)
C542	0.0508 (13)	0.0543 (14)	0.0556 (14)	0.0198 (11)	0.0213 (11)	0.0375 (12)
C55	0.0330 (9)	0.0398 (10)	0.0367 (10)	0.0130 (8)	0.0181 (8)	0.0187 (9)
C56	0.0276 (9)	0.0332 (9)	0.0303 (9)	0.0122 (7)	0.0113 (7)	0.0116 (8)
O56	0.0430 (8)	0.0574 (9)	0.0561 (9)	0.0322 (7)	0.0327 (7)	0.0345 (8)
C6	0.0278 (9)	0.0344 (9)	0.0299 (9)	0.0140 (8)	0.0140 (7)	0.0147 (8)
C61	0.0285 (9)	0.0339 (9)	0.0279 (9)	0.0129 (8)	0.0130 (7)	0.0100 (7)
C62	0.0400 (11)	0.0385 (11)	0.0394 (11)	0.0145 (9)	0.0102 (9)	0.0157 (9)

C63	0.0506 (13)	0.0340 (11)	0.0529 (13)	0.0064 (10)	0.0155 (11)	0.0128 (10)
C64	0.0357 (11)	0.0410 (12)	0.0565 (14)	0.0031 (9)	0.0084 (10)	0.0030 (10)
C65	0.0357 (11)	0.0434 (12)	0.0502 (13)	0.0145 (9)	0.0006 (9)	0.0075 (10)
C66	0.0343 (10)	0.0344 (10)	0.0412 (11)	0.0121 (8)	0.0074 (9)	0.0130 (9)
N7	0.0269 (7)	0.0350 (8)	0.0283 (8)	0.0117 (6)	0.0085 (6)	0.0153 (6)
C7A	0.0273 (9)	0.0351 (10)	0.0291 (9)	0.0118 (7)	0.0121 (7)	0.0125 (8)
O1	0.0399 (9)	0.183 (2)	0.0625 (12)	0.0545 (13)	0.0307 (9)	0.0738 (14)

Geometric parameters (Å, º)

N1—C2	1.302 (3)	C53—H53A	0.97
C2—N3	1.381 (3)	C53—H53B	0.97
N3—C4	1.414 (3)	C54—C541	1.526 (3)
C4—C4A	1.422 (3)	C54—C542	1.530 (3)
C4A—C5	1.427 (3)	C541—H54A	0.96
C5—C6	1.382 (2)	C541—H54B	0.96
C6—N7	1.395 (2)	C541—H54C	0.96
N7—C7A	1.353 (2)	C542—H54D	0.96
C7A—N1	1.365 (2)	C542—H54E	0.96
C4A—C7A	1.386 (2)	C542—H54F	0.96
C2—S2	1.762 (2)	C55—H55A	0.97
S2—C21	1.786 (3)	C55—H55B	0.97
C21—H21A	0.96	O56—H56	0.82
C21—H21B	0.96	C6—C61	1.466 (3)
C21—H21C	0.96	C61—C66	1.391 (3)
N3—C31	1.475 (3)	C61—C62	1.396 (3)
C31—H31C	0.96	C62—C63	1.381 (3)
C31—H31B	0.96	C62—H62	0.93
C31—H31A	0.96	C63—C64	1.374 (3)
C4—O4	1.231 (2)	C63—H63	0.93
C5—C51	1.486 (2)	C64—C65	1.375 (3)
C51—C52	1.446 (2)	C64—H64	0.93
C52—C53	1.511 (2)	C65—C66	1.382 (3)
C53—C54	1.534 (3)	C65—H65	0.93
C54—C55	1.532 (3)	C66—H66	0.93
C55—C56	1.494 (2)	N7—H7	0.86
C56—C51	1.362 (2)	O1—H1A	0.9799
C52—O52	1.236 (2)	O1—H1B	0.9798
C56—O56	1.328 (2)		

C2—N1—C7A	113.72 (17)	H54A—C541—H54B	109.5
N1—C2—N3	124.66 (18)	C54—C541—H54C	109.5
N1—C2—S2	119.72 (16)	H54A—C541—H54C	109.5
N3—C2—S2	115.58 (15)	H54B—C541—H54C	109.5
C2—S2—C21	101.03 (11)	C54—C542—H54D	109.5
S2—C21—H21A	109.5	C54—C542—H54E	109.5
S2—C21—H21B	109.5	H54D—C542—H54E	109.5
H21A—C21—H21B	109.5	C54—C542—H54F	109.5

S2—C21—H21C	109.5	H54D—C542—H54F	109.5
H21A—C21—H21C	109.5	H54E—C542—H54F	109.5
H21B—C21—H21C	109.5	C56—C55—C54	114.89 (15)
C2—N3—C4	122.69 (17)	C56—C55—H55A	108.5
C2—N3—C31	121.82 (18)	C54—C55—H55A	108.5
C4—N3—C31	115.43 (17)	C56—C55—H55B	108.5
N3—C31—H31C	109.5	C54—C55—H55B	108.5
N3—C31—H31B	109.5	H55A—C55—H55B	107.5
H31C—C31—H31B	109.5	O56—C56—C51	118.66 (16)
N3—C31—H31A	109.5	O56—C56—C55	116.90 (15)
H31C—C31—H31A	109.5	C51—C56—C55	124.43 (16)
H31B—C31—H31A	109.5	C56—O56—H56	109.5
O4—C4—N3	119.56 (18)	C5—C6—N7	108.05 (16)
O4—C4—C4A	127.26 (19)	C5—C6—C61	131.68 (16)
N3—C4—C4A	113.17 (16)	N7—C6—C61	120.09 (15)
C7A—C4A—C4	118.42 (17)	C66—C61—C62	117.25 (17)
C7A—C4A—C5	107.83 (16)	C66—C61—C6	121.31 (17)
C4—C4A—C5	133.70 (17)	C62—C61—C6	121.39 (16)
C6—C5—C4A	106.47 (15)	C63—C62—C61	121.15 (19)
C6—C5—C51	129.57 (17)	C63—C62—H62	119.4
C4A—C5—C51	123.63 (16)	C61—C62—H62	119.4
C56—C51—C52	119.12 (16)	C64—C63—C62	120.4 (2)
C56—C51—C5	120.88 (16)	C64—C63—H63	119.8
C52—C51—C5	119.86 (15)	C62—C63—H63	119.8
O52—C52—C51	122.41 (16)	C63—C64—C65	119.4 (2)
O52—C52—C53	119.86 (16)	C63—C64—H64	120.3
C51—C52—C53	117.72 (15)	C65—C64—H64	120.3
C52—C53—C54	112.82 (15)	C64—C65—C66	120.4 (2)
C52—C53—H53A	109.0	C64—C65—H65	119.8
C54—C53—H53A	109.0	C66—C65—H65	119.8
C52—C53—H53B	109.0	C65—C66—C61	121.31 (19)
C54—C53—H53B	109.0	C65—C66—H66	119.3
H53A—C53—H53B	107.8	C61—C66—H66	119.3
C541—C54—C542	109.74 (18)	C7A—N7—C6	109.42 (14)
C541—C54—C55	110.70 (17)	C7A—N7—H7	125.3
C542—C54—C55	108.64 (17)	C6—N7—H7	125.3
C541—C54—C53	109.43 (18)	N7—C7A—N1	124.78 (16)
C542—C54—C53	109.92 (16)	N7—C7A—C4A	108.20 (16)
C55—C54—C53	108.39 (15)	N1—C7A—C4A	126.96 (17)
C54—C541—H54A	109.5	H1A—O1—H1B	101.7
C54—C541—H54B	109.5		

C7A—N1—C2—N3	1.6 (3)	C542—C54—C55—C56	160.74 (17)
C7A—N1—C2—S2	−175.66 (14)	C53—C54—C55—C56	41.3 (2)
N1—C2—S2—C21	2.6 (2)	C52—C51—C56—O56	176.22 (17)
N3—C2—S2—C21	−174.90 (17)	C5—C51—C56—O56	0.6 (3)
N1—C2—N3—C4	−5.8 (3)	C52—C51—C56—C55	−2.6 (3)
S2—C2—N3—C4	171.58 (15)	C5—C51—C56—C55	−178.16 (18)

N1—C2—N3—C31	171.1 (2)	C54—C55—C56—O56	167.00 (17)
S2—C2—N3—C31	−11.5 (3)	C54—C55—C56—C51	−14.2 (3)
C2—N3—C4—O4	−177.36 (19)	C4A—C5—C6—N7	−1.2 (2)
C31—N3—C4—O4	5.5 (3)	C51—C5—C6—N7	172.33 (17)
C2—N3—C4—C4A	3.7 (3)	C4A—C5—C6—C61	−176.16 (18)
C31—N3—C4—C4A	−173.37 (18)	C51—C5—C6—C61	−2.6 (3)
O4—C4—C4A—C7A	−177.1 (2)	C5—C6—C61—C66	161.0 (2)
N3—C4—C4A—C7A	1.7 (3)	N7—C6—C61—C66	−13.5 (3)
O4—C4—C4A—C5	0.1 (4)	C5—C6—C61—C62	−16.3 (3)
N3—C4—C4A—C5	178.89 (19)	N7—C6—C61—C62	169.26 (17)
C7A—C4A—C5—C6	1.5 (2)	C66—C61—C62—C63	−2.9 (3)
C4—C4A—C5—C6	−175.9 (2)	C6—C61—C62—C63	174.44 (19)
C7A—C4A—C5—C51	−172.47 (16)	C61—C62—C63—C64	0.7 (4)
C4—C4A—C5—C51	10.1 (3)	C62—C63—C64—C65	1.8 (4)
C6—C5—C51—C56	−61.4 (3)	C63—C64—C65—C66	−2.0 (4)
C4A—C5—C51—C56	111.2 (2)	C64—C65—C66—C61	−0.3 (4)
C6—C5—C51—C52	123.1 (2)	C62—C61—C66—C65	2.7 (3)
C4A—C5—C51—C52	−64.4 (2)	C6—C61—C66—C65	−174.7 (2)
C56—C51—C52—O52	170.12 (18)	C5—C6—N7—C7A	0.4 (2)
C5—C51—C52—O52	−14.3 (3)	C61—C6—N7—C7A	176.08 (16)
C56—C51—C52—C53	−11.1 (3)	C6—N7—C7A—N1	−176.78 (17)
C5—C51—C52—C53	164.49 (17)	C6—N7—C7A—C4A	0.6 (2)
O52—C52—C53—C54	−140.24 (19)	C2—N1—C7A—N7	−178.74 (18)
C51—C52—C53—C54	41.0 (2)	C2—N1—C7A—C4A	4.4 (3)
C52—C53—C54—C541	66.4 (2)	C4—C4A—C7A—N7	176.59 (16)
C52—C53—C54—C542	−172.99 (18)	C5—C4A—C7A—N7	−1.3 (2)
C52—C53—C54—C55	−54.4 (2)	C4—C4A—C7A—N1	−6.1 (3)
C541—C54—C55—C56	−78.7 (2)	C5—C4A—C7A—N1	175.97 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O4	0.98	1.76	2.688 (3)	157
O1—H1*B*···O52^{i}	0.98	1.90	2.762 (3)	145
N7—H7···O52^{ii}	0.86	2.24	2.974 (2)	143
O56—H56···O1^{iii}	0.82	1.78	2.560 (3)	159

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