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Ethyl 4,6-dimethyl-3-oxo-2-phenyl-3,7-dihydro-2*H*-pyrazolo[3,4-*b*]pyridine-5-carboxylate monohydrate

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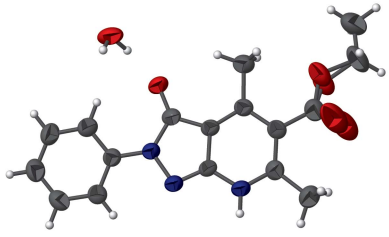
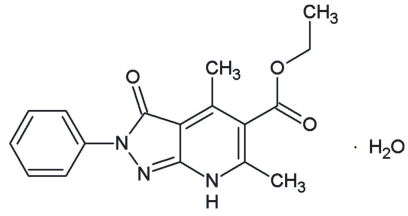
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{17}N_3O_3\cdot H_2O$, the dihedral angle between the 4,6-dimethyl-pyrazolo[3,4-*b*]pyridine-3-one unit [maximum deviation = 0.048 (2) Å] and the phenyl ring is 5.1 (1)°. The structure is characterized by disorder of the carboxylate O atoms, which are split into two parts with a major component of 0.898 (4). In the crystal, the organic molecules and lattice water molecules are linked *via* O—H···O, O—H···N and N—H···O hydrogen bonds. The molecules are also linked by C—H···π and weak offset π–π stacking interactions, forming sheets parallel to (001).

3D view



Chemical scheme



Structure description

The number of substituted pyrazolo[3,4-*b*]pyridines has increased in organic and pharmaceutical chemistry. These heterocyclic systems are found in a number of molecules possessing biological and pharmacological activities such as antimicrobial (Raviraj *et al.*, 2013), anti-inflammatory (Fong & Heymsfield, 2009) and antiviral (Bernardino *et al.*, 2007). Besides their pharmacological properties, pyrazolo[3,4-*b*]pyridines also exhibit corrosion inhibition properties (Gupta *et al.*, 2015). As part of our research on the preparation of new pyrazolo[3,4-*b*]pyridine-3-ones (Fadel *et al.*, 2011), the title compound was isolated and its crystal structure was determined by X-ray diffraction.

The molecular structure is shown in Fig. 1. The dihedral angle between the 4,6-dimethyl-pyrazolo[3,4-*b*]pyridine-3-one unit [maximum deviation = 0.048 (2) Å] and the phenyl ring is 5.1 (1)°. In the crystal, the components are linked through O—H···O, O—

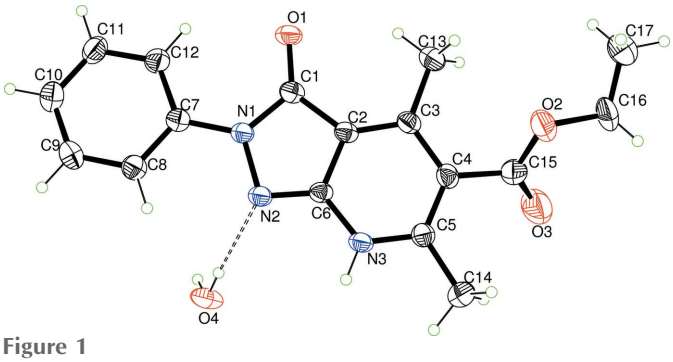


Figure 1
A view of the molecule of the title compound, showing displacement ellipsoids drawn at the 30% probability level. The O—H···N hydrogen bond (see Table 1) is shown as a dashed line. Only atoms of the major component of the disordered carboxylate group have been included.

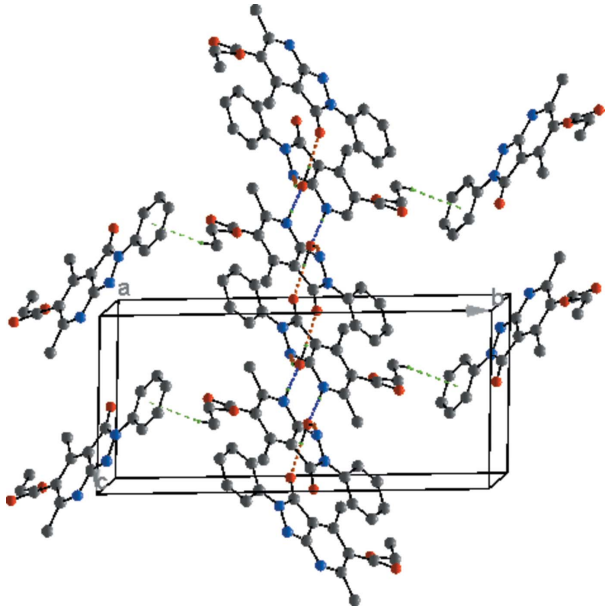


Figure 2
A partial view of the crystal packing of the title compound. O—H···O and O—H···N hydrogen bonds are shown as red dashed lines and N—H···O hydrogen bonds are shown as blue dashed lines while green dashed lines indicate the C—H···π contacts. Atoms not involved in hydrogen bonding have been omitted for clarity.

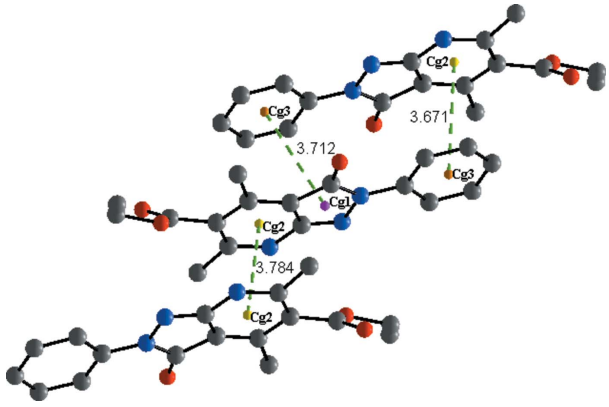


Figure 3
Stacking interactions. *Cg*1, *Cg*2 and *Cg*3 are the centroids of the N1/N2/C1/C2/C6, N3/C2/C3/C4/C5/C6 and C7–C12 rings, respectively, with centroids shown as colored spheres and *Cg*···*Cg* contacts drawn as green dashed lines.

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

*Cg*3 is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O4^{i}	0.86	1.86	2.718 (3)	177
O4—H1*W*···N2^{ii}	0.86 (5)	2.07 (5)	2.899 (3)	164 (5)
O4—H2*W*···O1	0.90 (5)	1.84 (5)	2.739 (3)	172 (4)
C17—H17*A*···*Cg*3^{iii}	0.96	2.79	3.717 (4)	163

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{17}N_3O_3{\cdot}H_2O$
M_r	329.35
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	10.5321 (15), 18.794 (3), 9.0491 (12)
β (°)	113.903 (5)
V (Å^3)	1637.6 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.32 × 0.24 × 0.15
Data collection	
Diffractometer	Bruker DUO APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.696, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15373, 2879, 1709
R_{int}	0.056
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.166, 0.96
No. of reflections	2879
No. of parameters	235
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.24

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg *et al.*, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

H···N and N—H···O hydrogen bonds (Table 1, Fig. 2). The cohesion of the crystal structure is enhanced by three π–π stacking interactions between the rings, Fig. 3, with centroid–centroid distances in the range 3.671 (2) to 3.784 (2) Å, forming molecular layers along the *c*-axis direction. These sheets are linked together by C17—H17*A*···π interactions (Table 1, Fig. 2).

Synthesis and crystallization

A solution of 3-amino-1-phenyl-1*H*-pyrazol-5(4*H*)-one (875 mg, 5 mmol) and ethyl acetoacetate (3,19 ml, 25 mmol) was refluxed in a sand bath (180–185° C) for 6 h. The reaction mixture was left overnight at room temperature. The grey crystals formed were collected by filtration and washed with

diethyl ether. The dried orange crystals were obtained with 42% yield and melting point around 534 K.

^{1}H NMR (300 MHz, $CDCl_3$, δ p.p.m.): 1.39 (3*H*, H17), 2.28 (3*H*, H14), 2.77 (3*H*, H13), 4.40 (2*H*, H16), 7.21 (1*H*, H10), 7.40 (2*H*, H9 and H11), 7.85 (2*H*, H8 and H12);

^{13}C NMR (75 MHz, $CDCl_3$, δ p.p.m.): 14.56 (CH3), 22.16 (C13, C14), 61.73 (C16), 108.99 (C2, C4), 122.30 (C10), 125.72 (C9, C11), 129.12 (C9, C11), 137.40 (C7), 150.92 (C3), 153.73 (C6), 157.34 (C5), 159.16 (C15), 167.30 (C1).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The reflection ($\bar{3}$33) affected by the beam-stop was removed during refinement. The O atoms of the carboxylate group are disordered over two sets of sites, with a refined occupancy ratio of 0.898 (4):0.102 (4).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161619 [https://doi.org/10.1107/S2414314616016199]

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Crystal data

$C_{17}H_{17}N_3O_3·H_2O$
M_r = 329.35
Monoclinic, $P2_1/c$
a = 10.5321 (15) Å
b = 18.794 (3) Å
c = 9.0491 (12) Å
β = 113.903 (5)°
V = 1637.6 (4) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.336 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2879 reflections
θ = 2.1–25.0°
μ = 0.10 mm^{-1}
T = 293 K
Block, orange
0.32 × 0.24 × 0.15 mm

Data collection

Bruker DUO APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.696, T_{max} = 0.746
15373 measured reflections
2879 independent reflections
1709 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 25.0°, θ_{min} = 2.1°
h = −12→11
k = −22→22
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.166
S = 0.96
2879 reflections
235 parameters
2 restraints
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.5493P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 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}	Occ. (<1)
O1	0.2341 (2)	−0.04229 (12)	0.5100 (2)	0.0593 (6)	
O2	−0.1524 (3)	0.14716 (15)	0.0298 (4)	0.0671 (8)	0.898 (4)
O3	−0.0117 (3)	0.21966 (17)	−0.0281 (4)	0.1033 (12)	0.898 (4)
O2A	−0.1029 (19)	0.1830 (15)	0.092 (3)	0.0671 (8)	0.102 (4)
O3A	−0.121 (2)	0.1625 (14)	−0.133 (2)	0.1033 (12)	0.102 (4)
N1	0.3718 (2)	−0.06247 (12)	0.3654 (3)	0.0446 (6)	
N2	0.3789 (2)	−0.04097 (13)	0.2189 (3)	0.0453 (6)	
C1	0.2639 (3)	−0.03131 (16)	0.3926 (3)	0.0451 (7)	
C2	0.1975 (3)	0.01464 (15)	0.2528 (3)	0.0419 (7)	
C3	0.0919 (3)	0.06363 (15)	0.2102 (3)	0.0455 (7)	
C4	0.0624 (3)	0.10151 (16)	0.0640 (3)	0.0476 (7)	
C5	0.1344 (3)	0.08678 (16)	−0.0323 (3)	0.0481 (7)	
C6	0.2745 (3)	0.00291 (14)	0.1589 (3)	0.0408 (7)	
C7	0.4740 (3)	−0.11041 (15)	0.4645 (3)	0.0443 (7)	
C15	−0.0376 (4)	0.1618 (2)	0.0152 (4)	0.0646 (9)	
N3	0.2371 (2)	0.03840 (12)	0.0153 (3)	0.0458 (6)	
H3N	0.2801	0.0295	−0.0456	0.055*	
C8	0.5713 (3)	−0.13804 (16)	0.4129 (4)	0.0508 (8)	
H8	0.5677	−0.1259	0.3117	0.061*	
C9	0.6732 (3)	−0.18347 (16)	0.5113 (4)	0.0556 (8)	
H9	0.7381	−0.2015	0.4758	0.067*	
C10	0.6803 (3)	−0.20245 (17)	0.6610 (4)	0.0616 (9)	
H10	0.7498	−0.2328	0.7271	0.074*	
C11	0.5833 (4)	−0.17590 (17)	0.7113 (4)	0.0634 (9)	
H11	0.5869	−0.1889	0.8121	0.076*	
C12	0.4808 (3)	−0.13044 (16)	0.6156 (4)	0.0562 (8)	
H12	0.4158	−0.1130	0.6518	0.067*	
C13	0.0193 (3)	0.07762 (19)	0.3210 (4)	0.0654 (10)	
H13A	0.0138	0.1280	0.3351	0.098*	
H13B	0.0706	0.0558	0.4241	0.098*	
H13C	−0.0727	0.0580	0.2744	0.098*	
C14	0.1036 (4)	0.12011 (18)	−0.1943 (4)	0.0657 (9)	
H14A	0.1171	0.1706	−0.1817	0.099*	
H14B	0.0092	0.1103	−0.2659	0.099*	
H14C	0.1650	0.1007	−0.2386	0.099*	
C16	−0.2515 (4)	0.2055 (2)	0.0019 (6)	0.0954 (14)	
H16A	−0.2026	0.2497	0.0444	0.115*	
H16B	−0.3065	0.2115	−0.1130	0.115*	
C17	−0.3398 (5)	0.1878 (2)	0.0831 (6)	0.1029 (15)	
H17A	−0.4087	0.2242	0.0629	0.154*	
H17B	−0.2849	0.1842	0.1972	0.154*	
H17C	−0.3847	0.1431	0.0432	0.154*	
O4	0.3690 (3)	0.00542 (15)	0.8210 (3)	0.0673 (7)	
H1W	0.443 (5)	0.023 (3)	0.819 (6)	0.131 (19)*	
H2W	0.317 (4)	−0.010 (2)	0.720 (6)	0.114 (16)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0515 (13)	0.0909 (16)	0.0426 (11)	0.0058 (12)	0.0263 (10)	0.0118 (11)
O2	0.0455 (16)	0.0638 (18)	0.092 (2)	0.0074 (14)	0.0281 (15)	0.0040 (15)
O3	0.088 (2)	0.078 (2)	0.167 (3)	0.0230 (17)	0.076 (2)	0.048 (2)
O2A	0.0455 (16)	0.0638 (18)	0.092 (2)	0.0074 (14)	0.0281 (15)	0.0040 (15)
O3A	0.088 (2)	0.078 (2)	0.167 (3)	0.0230 (17)	0.076 (2)	0.048 (2)
N1	0.0414 (14)	0.0569 (15)	0.0391 (13)	0.0032 (12)	0.0202 (11)	0.0044 (11)
N2	0.0426 (14)	0.0588 (15)	0.0386 (13)	0.0019 (13)	0.0208 (12)	0.0017 (11)
C1	0.0382 (16)	0.0599 (18)	0.0377 (15)	−0.0039 (14)	0.0160 (14)	−0.0036 (14)
C2	0.0345 (16)	0.0557 (17)	0.0366 (14)	−0.0047 (14)	0.0155 (13)	−0.0040 (13)
C3	0.0386 (17)	0.0561 (17)	0.0436 (16)	−0.0014 (14)	0.0183 (14)	−0.0029 (14)
C4	0.0374 (17)	0.0568 (18)	0.0478 (17)	−0.0009 (15)	0.0164 (14)	−0.0026 (14)
C5	0.0372 (17)	0.0579 (18)	0.0460 (16)	−0.0027 (15)	0.0136 (14)	0.0017 (14)
C6	0.0392 (16)	0.0506 (17)	0.0333 (14)	−0.0037 (14)	0.0154 (13)	−0.0025 (13)
C7	0.0437 (17)	0.0467 (16)	0.0410 (16)	−0.0011 (14)	0.0156 (14)	−0.0019 (13)
C15	0.052 (2)	0.078 (3)	0.064 (2)	0.011 (2)	0.0243 (18)	0.0137 (19)
N3	0.0430 (14)	0.0613 (15)	0.0387 (13)	0.0014 (13)	0.0221 (11)	0.0008 (12)
C8	0.0470 (18)	0.0544 (18)	0.0496 (18)	−0.0001 (15)	0.0183 (15)	−0.0016 (15)
C9	0.0482 (19)	0.0523 (18)	0.065 (2)	0.0031 (16)	0.0216 (17)	−0.0080 (16)
C10	0.062 (2)	0.0532 (19)	0.062 (2)	0.0086 (17)	0.0168 (18)	0.0018 (16)
C11	0.079 (2)	0.057 (2)	0.0509 (19)	0.0140 (19)	0.0230 (18)	0.0108 (16)
C12	0.067 (2)	0.0563 (19)	0.0494 (17)	0.0076 (17)	0.0274 (17)	0.0025 (15)
C13	0.054 (2)	0.092 (3)	0.0580 (19)	0.0108 (18)	0.0308 (17)	0.0053 (18)
C14	0.066 (2)	0.077 (2)	0.056 (2)	0.0057 (18)	0.0266 (18)	0.0152 (17)
C16	0.070 (3)	0.102 (3)	0.125 (3)	0.042 (2)	0.051 (3)	0.037 (3)
C17	0.107 (3)	0.113 (3)	0.112 (3)	0.046 (3)	0.068 (3)	0.021 (3)
O4	0.0592 (16)	0.102 (2)	0.0534 (15)	−0.0104 (14)	0.0353 (13)	−0.0105 (13)

Geometric parameters (Å, º)

O1—C1	1.241 (3)	C8—C9	1.378 (4)
O2—C15	1.299 (4)	C8—H8	0.9300
O2—C16	1.464 (4)	C9—C10	1.373 (4)
O3—C15	1.223 (4)	C9—H9	0.9300
O2A—C15	1.23 (2)	C10—C11	1.370 (4)
O2A—C16	1.504 (16)	C10—H10	0.9300
O3A—C15	1.272 (18)	C11—C12	1.374 (4)
N1—C1	1.386 (4)	C11—H11	0.9300
N1—C7	1.412 (4)	C12—H12	0.9300
N1—N2	1.417 (3)	C13—H13A	0.9600
N2—C6	1.303 (3)	C13—H13B	0.9600
C1—C2	1.455 (4)	C13—H13C	0.9600
C2—C3	1.373 (4)	C14—H14A	0.9600
C2—C6	1.411 (4)	C14—H14B	0.9600
C3—C4	1.421 (4)	C14—H14C	0.9600
C3—C13	1.511 (4)	C16—C17	1.439 (5)

C4—C5	1.396 (4)	C16—H16A	0.9700
C4—C15	1.487 (4)	C16—H16B	0.9700
C5—N3	1.343 (4)	C17—H17A	0.9600
C5—C14	1.503 (4)	C17—H17B	0.9600
C6—N3	1.368 (3)	C17—H17C	0.9600
C7—C8	1.387 (4)	O4—H1W	0.86 (5)
C7—C12	1.392 (4)	O4—H2W	0.90 (5)
N3—H3N	0.8600		

C15—O2—C16	116.8 (3)	C10—C9—C8	121.0 (3)
C15—O2A—C16	118.7 (19)	C10—C9—H9	119.5
C1—N1—C7	128.7 (2)	C8—C9—H9	119.5
C1—N1—N2	113.8 (2)	C11—C10—C9	118.9 (3)
C7—N1—N2	117.4 (2)	C11—C10—H10	120.6
C6—N2—N1	102.4 (2)	C9—C10—H10	120.6
O1—C1—N1	126.3 (3)	C10—C11—C12	121.2 (3)
O1—C1—C2	130.1 (3)	C10—C11—H11	119.4
N1—C1—C2	103.7 (2)	C12—C11—H11	119.4
C3—C2—C6	122.2 (2)	C11—C12—C7	120.1 (3)
C3—C2—C1	133.8 (2)	C11—C12—H12	119.9
C6—C2—C1	103.9 (2)	C7—C12—H12	119.9
C2—C3—C4	116.4 (2)	C3—C13—H13A	109.5
C2—C3—C13	120.0 (3)	C3—C13—H13B	109.5
C4—C3—C13	123.5 (3)	H13A—C13—H13B	109.5
C5—C4—C3	120.9 (3)	C3—C13—H13C	109.5
C5—C4—C15	117.7 (3)	H13A—C13—H13C	109.5
C3—C4—C15	121.3 (3)	H13B—C13—H13C	109.5
N3—C5—C4	120.3 (2)	C5—C14—H14A	109.5
N3—C5—C14	115.0 (3)	C5—C14—H14B	109.5
C4—C5—C14	124.7 (3)	H14A—C14—H14B	109.5
N2—C6—N3	125.0 (2)	C5—C14—H14C	109.5
N2—C6—C2	116.2 (2)	H14A—C14—H14C	109.5
N3—C6—C2	118.7 (3)	H14B—C14—H14C	109.5
C8—C7—C12	118.6 (3)	C17—C16—O2	107.5 (3)
C8—C7—N1	120.2 (2)	C17—C16—O2A	113.7 (11)
C12—C7—N1	121.3 (3)	C17—C16—H16A	110.2
O2A—C15—O3A	107.0 (15)	O2—C16—H16A	110.2
O3—C15—O2	124.0 (3)	C17—C16—H16B	110.2
O3—C15—C4	123.4 (3)	O2—C16—H16B	110.2
O2A—C15—C4	124.8 (11)	H16A—C16—H16B	108.5
O3A—C15—C4	115.0 (13)	C16—C17—H17A	109.5
O2—C15—C4	112.6 (3)	C16—C17—H17B	109.5
C5—N3—C6	121.3 (2)	H17A—C17—H17B	109.5
C5—N3—H3N	119.3	C16—C17—H17C	109.5
C6—N3—H3N	119.3	H17A—C17—H17C	109.5
C9—C8—C7	120.2 (3)	H17B—C17—H17C	109.5
C9—C8—H8	119.9	H1W—O4—H2W	107 (4)
C7—C8—H8	119.9		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O4^{i}	0.86	1.86	2.718 (3)	177
O4—H1*W*···N2^{ii}	0.86 (5)	2.07 (5)	2.899 (3)	164 (5)
O4—H2*W*···O1	0.90 (5)	1.84 (5)	2.739 (3)	172 (4)
C17—H17*A*···*Cg*3^{iii}	0.96	2.79	3.717 (4)	163

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