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Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[_h_]cinnolin-2-yl)propionic acid

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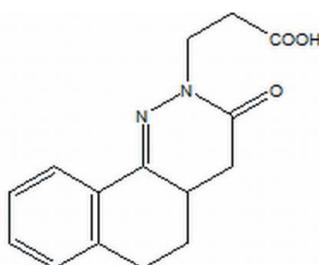
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Acta Cryst. (2014). E**70**   Meneghetti _et al._ • C$_{15}$H$_{16}$N$_2$O$_3$

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Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Fiorella Meneghetti,* Daniela Masciocchi, Arianna Gelain and Stefania Villa

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The asymmetric unit of the title compound, $C_{15}H_{16}N_2O_3$, contains two independent molecules, which present a different conformation of the carboxylic acid side chain [C—C—C—OH torsion angles = 65.3 (7) and −170.1 (5)°]. In both molecules, the dihydropyridazinone ring adopts a geometry intermediate between a twisted-boat and a half-chair conformation, while the central six-membered ring is almost in a half-boat conformation. In the crystal, molecules are linked by O—H⋯O$_k$ (k = ketone) hydrogen bonds, generating [01$\bar{1}$] chains. Aromatic π–π stacking contacts between the benzene and the dihydropyridazinone rings [centroid–centroid distance [3.879 (9) Å] are also observed.

1. Related literature

For background to the bioactivity of pyridazinone derivatives, see: Masciocchi *et al.* (2013). For structural and molecular modeling studies, see: Toma *et al.* (1990). For the chemistry of pyridazinone derivatives, see: Costantino *et al.* (1996).
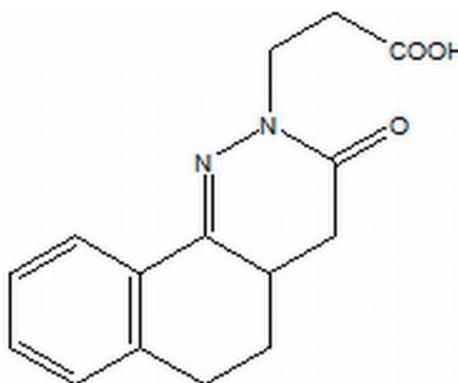
2. Experimental

2.1. Crystal data

$C_{15}H_{16}N_2O_3$	$\gamma = 68.630$ (9)°
$M_r = 272.3$	$V = 1332.6$ (8) Å^3
Triclinic, $P\bar{1}$	$Z = 4$
$a = 11.217$ (4) Å	Mo $K\alpha$ radiation
$b = 11.668$ (4) Å	$\mu = 0.10$ mm^{-1}
$c = 12.110$ (4) Å	$T = 293$ K
$\alpha = 79.22$ (1)°	$0.65 \times 0.45 \times 0.40$ mm
$\beta = 64.62$ (1)°	

2.2. Data collection

Enraf–Nonius TurboCAD-4 diffractometer	1412 reflections with $I > 2\sigma(I)$
5412 measured reflections	$R_{int} = 0.081$
4682 independent reflections	3 standard reflections every 120 min intensity decay: 9%

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.205$	
$S = 0.92$	
4682 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
368 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2A—H2A⋯O1B	0.92 (7)	1.78 (7)	2.651 (6)	158 (6)
O2B—H2B⋯O1A	0.90 (6)	1.75 (6)	2.598 (7)	157 (5)

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

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data reports

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7233).

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supporting information

Crystal structure of 3-(3-oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Fiorella Meneghetti,* Daniela Masciocchi, Arianna Gelain and Stefania Villa

S1. Structural commentary

In our previous researches focused on the discovery of new inhibitors targeting aberrant STAT3 signaling for the treatment of human cancers, we evidenced that several pyridazinone derivatives were able to interfer within the STAT3 pathway (Masciocchi *et al.*, 2013). As the size of the central ring plays a main role in determining the conformational properties for this class of compounds, we investigated the extent of planarity of the phenyl with respect to the other cycles by crystallographic analysis determining the molecular structure of the title compound. The asymmetric unit of the title compound (Fig. 1) is characterized by two crystallographically independent molecules (*a* and *b*). The values obtained for the bond length and angles of the two independent molecules are in accordance with each other, whilst at the same time presenting a different conformation of the carboxylic chain linked to N1. This difference is best evidenced by the torsion angles N2—N1—C13—C14 of 76 (1)°[-100 (1)°] and O2—C15—C14—C13 of 65 (1)°[-170 (1)°] (the values in the square brackets refer to the *b* labeled molecule). The tricyclic skeleton of the compound consists of three fused rings slightly twisted with respect to each other. The dihedral angles between their least-square planes α (C1/N1/N2/C4/C3/C2), β (C3/C4/C5/C6/C7/C8) and γ (C5/C6/C12/C11/C10/C9) are: α-β = 5.0 (1)°[3.0 (1)°], α-γ = 11.5 (1)°[10.3 (1)°], β-γ = 6.6 (1)°[7.3 (1)°], respectively. In detail, the dihydropyridazinone ring adopts a geometry intermediate between a twisted-boat and a half-chair conformation quantitatively defined by the parameters QT = 0.321 (6)Å[0.242 (6)Å], φ = -87 (1)°[-83 (1)°] and θ = 113.2 (9)°[114 (1)°], while the central six-membered ring is almost in a half-boat conformation, characterized by the puckering parameters QT = 0.372 (7)Å[0.334 (7)Å], φ = -64 (1)°[-58 (1)°] and θ = 57 (1)°[59 (1)°], with the flap atom C8 out of the best mean plane calculated over the other five carbons by 0.512 (6)Å[0.462 (6)Å]. In the crystal, the *a* and *b* molecules are flattened and lay on planes deviated from that containing the *a* and *c* axes by about 30°. The two conformers interact through π-π contacts between the benzene and the dihydropyridazinone rings, at a centroid-centroid distance of 3.879 (9)Å. In addition, *a* and *b* molecules are interconnected through hydrogen bonds, where the carboxylic oxygen O2 is donor of a proton to the ketonic oxygen O1 of the partner molecule (Fig. 2). The intermolecular contacts involve O2a which is linked to a centrosymmetrically related molecule of *b* (O2a—H···O1b(i) at a distance of 1.78 (7)Å and angle of 158 (6)° [symmetry code: (i) -*x*, 1 - *y*, 1 - *z*)] and O2b that hydrogen bonds a molecule related to *a* by a crystallographic inversion centre (O2b–H···O1a(ii) at a distance of 1.75 (6)Å and angle of 157 (5)° [symmetry code:(ii) at -*x*, 2 - *y*, - *z*)].

S2. Crystallization

After many attempts weakly diffracting yellow prisms were grown by slow evaporation of a 30:70 water/methanol solution.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All non-H-atoms were refined anisotropically. The H-atoms positions bonded to heteroatoms were obtained by a close examination of a final difference Fourier, while the remaining ones were introduced at calculated positions and refined with fixed isotropic thermal parameters (1.2 Ueq of the parent atom).

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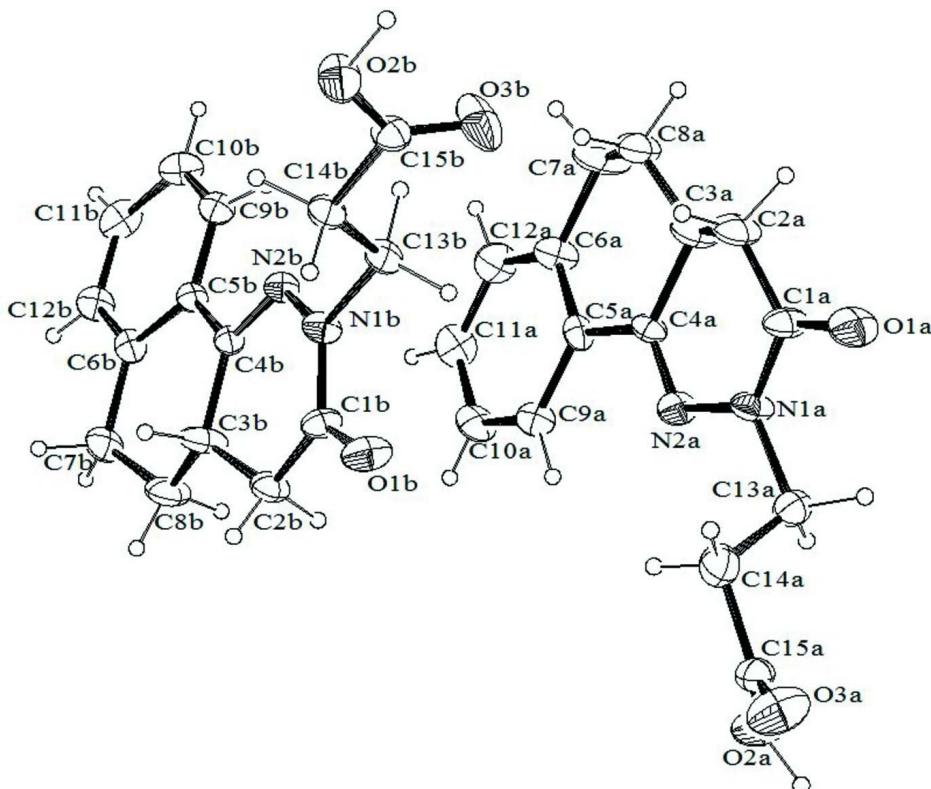
Figure 1

The molecular structure of the asymmetric unit of the title compound, showing displacement ellipsoids for non-H atoms at the 40% probability level.

fig2.tif
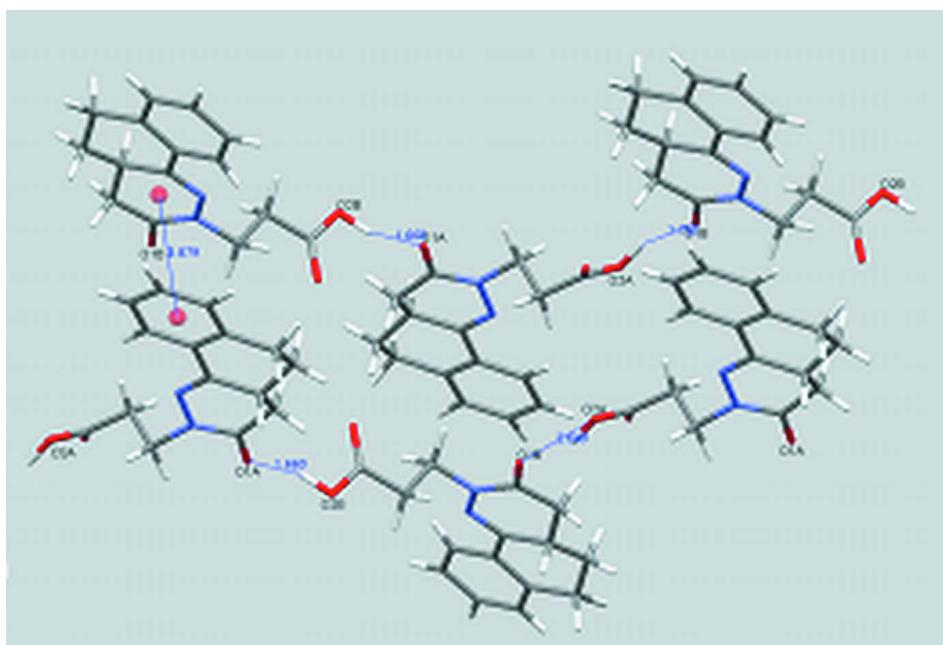
Figure 2

Intermolecular interactions of the title compound. Hydrogen bonds are shown as dashed lines.

3-(3-Oxo-2,3,4,4a,5,6-hexahydrobenzo[*h*]cinnolin-2-yl)propionic acid

Crystal data

$C_{15}H_{16}N_2O_3$	$Z = 4$
$M_r = 272.3$	$F(000) = 576$
Triclinic, $P\bar{1}$	$D_x = 1.357$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 11.217$ (4) Å	Cell parameters from 25 reflections
$b = 11.668$ (4) Å	$\theta = 9–10°$
$c = 12.110$ (4) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 79.22$ (1)°	$T = 293$ K
$\beta = 64.62$ (1)°	Prism, yellow
$\gamma = 68.630$ (9)°	$0.65 \times 0.45 \times 0.40$ mm
$V = 1332.6$ (8) Å^3	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	$R_{int} = 0.081$
	$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
Radiation source: fine-focus sealed tube	$h = -12 \rightarrow 13$
Graphite monochromator	$k = -13 \rightarrow 13$
non–profiled $\omega/2\theta$ scans	$l = -1 \rightarrow 14$
5412 measured reflections	3 standard reflections every 120 min
4682 independent reflections	intensity decay: 9%
1412 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	
$wR(F^2) = 0.205$	
$S = 0.92$	H atoms treated by a mixture of independent and constrained refinement
4682 reflections	
368 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2]$
0 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.28$ e Å⁻³
	$\Delta\rho_{min} = -0.22$ 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}
O1A	0.0119 (4)	0.6619 (4)	0.0134 (4)	0.0688 (13)
O2A	−0.0208 (4)	0.3563 (4)	0.4257 (4)	0.0671 (14)
H2A	−0.067 (6)	0.299 (6)	0.451 (6)	0.08 (2)*
O3A	−0.2262 (5)	0.4797 (4)	0.4325 (5)	0.0882 (16)
N1A	0.1447 (4)	0.5851 (4)	0.1231 (4)	0.0496 (13)
N2A	0.2481 (5)	0.5828 (4)	0.1591 (4)	0.0471 (13)
C1A	0.1087 (6)	0.6616 (5)	0.0356 (6)	0.0530 (17)
C2A	0.1924 (6)	0.7439 (6)	−0.0252 (6)	0.068 (2)
H2A1	0.2002	0.7583	−0.1092	0.081*
H2A2	0.1415	0.8224	0.0140	0.081*
C3A	0.3308 (6)	0.7041 (6)	−0.0269 (6)	0.0621 (18)
H3A	0.3821	0.6377	−0.0852	0.075*
C4A	0.3353 (6)	0.6383 (5)	0.0923 (5)	0.0383 (14)
C5A	0.4477 (6)	0.6302 (5)	0.1270 (5)	0.0408 (14)
C6A	0.5553 (5)	0.6744 (5)	0.0508 (5)	0.0520 (17)
C7A	0.5541 (6)	0.7381 (7)	−0.0711 (6)	0.081 (2)
H7A1	0.5920	0.8051	−0.0879	0.097*
H7A2	0.6149	0.6797	−0.1354	0.097*
C8A	0.4166 (6)	0.7870 (6)	−0.0752 (6)	0.067 (2)
H8A1	0.4281	0.8098	−0.1595	0.080*
H8A2	0.3654	0.8618	−0.0294	0.080*
C9A	0.4508 (6)	0.5720 (5)	0.2382 (5)	0.0484 (16)
H9A	0.3789	0.5420	0.2913	0.058*
C10A	0.5593 (6)	0.5589 (5)	0.2699 (6)	0.0576 (18)

104	H10A	0.5600	0.5200	0.3442	0.069*
105	C11A	0.6657 (6)	0.6025 (6)	0.1934 (7)	0.068 (2)
106	H11A	0.7392	0.5929	0.2148	0.081*
107	C12A	0.6632 (6)	0.6610 (6)	0.0840 (6)	0.0662 (19)
108	H12A	0.7350	0.6917	0.0320	0.079*
109	C13A	0.0675 (6)	0.4990 (5)	0.1958 (6)	0.0529 (16)
110	H13A	0.1326	0.4197	0.2051	0.063*
111	H13B	0.0167	0.4871	0.1534	0.063*
112	C14A	−0.0311 (6)	0.5484 (5)	0.3179 (6)	0.0610 (18)
113	H14A	−0.0990	0.6255	0.3081	0.073*
114	H14B	0.0194	0.5652	0.3576	0.073*
115	C15A	−0.1058 (7)	0.4600 (5)	0.3977 (6)	0.0529 (16)
116	O1B	0.1349 (4)	0.8168 (3)	0.4536 (4)	0.0599 (12)
117	O2B	0.0424 (5)	1.2275 (4)	0.1748 (4)	0.0815 (16)
118	H2B	0.038 (6)	1.246 (5)	0.101 (5)	0.070*
119	O3B	0.1379 (6)	1.0503 (4)	0.0884 (5)	0.0973 (19)
120	N1B	0.3133 (4)	0.8868 (4)	0.3469 (4)	0.0422 (12)
121	N2B	0.4445 (4)	0.8964 (4)	0.3189 (4)	0.0374 (11)
122	C1B	0.2460 (6)	0.8252 (5)	0.4432 (5)	0.0435 (15)
123	C2B	0.3141 (6)	0.7674 (5)	0.5298 (5)	0.0506 (16)
124	H2B1	0.2426	0.7792	0.6121	0.061*
125	H2B2	0.3534	0.6795	0.5173	0.061*
126	C3B	0.4248 (6)	0.8110 (6)	0.5232 (5)	0.0609 (19)
127	H3B	0.3709	0.8868	0.5689	0.073*
128	C4B	0.4978 (5)	0.8586 (4)	0.3997 (5)	0.0347 (13)
129	C5B	0.6346 (5)	0.8694 (4)	0.3670 (5)	0.0355 (14)
130	C6B	0.6924 (5)	0.8414 (5)	0.4544 (5)	0.0425 (15)
131	C7B	0.6210 (6)	0.7916 (5)	0.5799 (5)	0.0535 (17)
132	H7B1	0.6902	0.7264	0.6035	0.064*
133	H7B2	0.5783	0.8566	0.6376	0.064*
134	C8B	0.5143 (7)	0.7434 (6)	0.5873 (6)	0.069 (2)
135	H8B1	0.5608	0.6606	0.5571	0.083*
136	H8B2	0.4553	0.7367	0.6731	0.083*
137	C9B	0.7096 (5)	0.9085 (5)	0.2506 (5)	0.0453 (15)
138	H9B	0.6733	0.9244	0.1912	0.054*
139	C10B	0.8366 (6)	0.9246 (5)	0.2198 (6)	0.0570 (18)
140	H10B	0.8844	0.9527	0.1418	0.068*
141	C11B	0.8898 (6)	0.8975 (5)	0.3091 (6)	0.0593 (19)
142	H11B	0.9754	0.9066	0.2904	0.071*
143	C12B	0.8196 (6)	0.8580 (5)	0.4235 (6)	0.0489 (16)
144	H12B	0.8572	0.8418	0.4820	0.059*
145	C13B	0.2663 (5)	0.9335 (5)	0.2464 (5)	0.0463 (15)
146	H13C	0.2232	0.8791	0.2377	0.056*
147	H13D	0.3459	0.9344	0.1705	0.056*
148	C14B	0.1637 (6)	1.0613 (5)	0.2699 (5)	0.0510 (16)
149	H14C	0.2069	1.1154	0.2791	0.061*
150	H14D	0.0842	1.0601	0.3458	0.061*
151	C15B	0.1141 (6)	1.1115 (6)	0.1669 (6)	0.0479 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.069 (3)	0.075 (3)	0.081 (3)	−0.027 (2)	−0.057 (3)	0.031 (2)
O2A	0.051 (3)	0.069 (3)	0.083 (3)	−0.025 (3)	−0.037 (3)	0.031 (3)
O3A	0.063 (3)	0.064 (3)	0.130 (5)	−0.018 (3)	−0.044 (3)	0.025 (3)
N1A	0.044 (3)	0.063 (3)	0.046 (3)	−0.024 (3)	−0.025 (3)	0.022 (3)
N2A	0.046 (3)	0.051 (3)	0.057 (3)	−0.017 (3)	−0.037 (3)	0.017 (3)
C1A	0.055 (4)	0.046 (4)	0.055 (4)	−0.016 (3)	−0.028 (4)	0.021 (3)
C2A	0.073 (5)	0.080 (5)	0.065 (5)	−0.035 (4)	−0.049 (4)	0.039 (4)
C3A	0.056 (4)	0.076 (4)	0.052 (4)	−0.021 (4)	−0.031 (4)	0.026 (4)
C4A	0.040 (3)	0.039 (3)	0.032 (3)	−0.008 (3)	−0.019 (3)	0.009 (3)
C5A	0.043 (3)	0.036 (3)	0.043 (4)	0.000 (3)	−0.026 (3)	−0.006 (3)
C6A	0.030 (3)	0.067 (4)	0.048 (4)	−0.011 (3)	−0.014 (3)	0.012 (3)
C7A	0.049 (4)	0.117 (6)	0.060 (5)	−0.039 (4)	−0.013 (4)	0.038 (4)
C8A	0.078 (5)	0.078 (5)	0.055 (4)	−0.041 (4)	−0.036 (4)	0.033 (4)
C9A	0.041 (4)	0.044 (3)	0.049 (4)	−0.002 (3)	−0.020 (3)	0.004 (3)
C10A	0.061 (4)	0.054 (4)	0.059 (5)	−0.001 (3)	−0.042 (4)	0.006 (3)
C11A	0.046 (4)	0.077 (5)	0.081 (6)	−0.008 (4)	−0.032 (4)	−0.010 (4)
C12A	0.044 (4)	0.087 (5)	0.067 (5)	−0.025 (4)	−0.019 (4)	−0.001 (4)
C13A	0.051 (4)	0.050 (4)	0.064 (5)	−0.019 (3)	−0.029 (4)	0.007 (3)
C14A	0.067 (4)	0.056 (4)	0.063 (5)	−0.019 (4)	−0.027 (4)	−0.006 (4)
C15A	0.066 (4)	0.054 (4)	0.052 (4)	−0.030 (4)	−0.033 (4)	0.016 (3)
O1B	0.046 (2)	0.061 (3)	0.078 (3)	−0.023 (2)	−0.036 (2)	0.026 (2)
O2B	0.128 (4)	0.046 (3)	0.085 (4)	0.002 (3)	−0.082 (4)	0.004 (3)
O3B	0.139 (5)	0.066 (3)	0.081 (4)	0.026 (3)	−0.081 (4)	−0.018 (3)
N1B	0.032 (3)	0.052 (3)	0.044 (3)	−0.009 (2)	−0.026 (2)	0.012 (3)
N2B	0.031 (3)	0.038 (3)	0.040 (3)	−0.008 (2)	−0.017 (2)	0.004 (2)
C1B	0.037 (4)	0.039 (3)	0.055 (4)	−0.012 (3)	−0.025 (3)	0.016 (3)
C2B	0.057 (4)	0.060 (4)	0.042 (4)	−0.025 (3)	−0.029 (3)	0.018 (3)
C3B	0.056 (4)	0.079 (5)	0.050 (4)	−0.032 (4)	−0.029 (4)	0.035 (4)
C4B	0.040 (3)	0.028 (3)	0.037 (4)	−0.006 (2)	−0.020 (3)	0.001 (3)
C5B	0.036 (3)	0.034 (3)	0.039 (4)	−0.006 (3)	−0.023 (3)	0.003 (3)
C6B	0.040 (4)	0.038 (3)	0.046 (4)	−0.001 (3)	−0.023 (3)	−0.003 (3)
C7B	0.054 (4)	0.058 (4)	0.053 (4)	−0.010 (3)	−0.036 (3)	0.007 (3)
C8B	0.090 (5)	0.083 (5)	0.055 (5)	−0.047 (4)	−0.047 (4)	0.037 (4)
C9B	0.040 (4)	0.042 (3)	0.047 (4)	−0.003 (3)	−0.023 (3)	0.006 (3)
C10B	0.036 (3)	0.056 (4)	0.062 (5)	−0.012 (3)	−0.011 (3)	0.012 (3)
C11B	0.037 (4)	0.056 (4)	0.085 (6)	−0.010 (3)	−0.031 (4)	0.004 (4)
C12B	0.039 (4)	0.051 (4)	0.058 (5)	−0.003 (3)	−0.032 (4)	0.002 (3)
C13B	0.037 (3)	0.055 (4)	0.046 (4)	−0.009 (3)	−0.023 (3)	0.004 (3)
C14B	0.057 (4)	0.051 (4)	0.047 (4)	−0.011 (3)	−0.032 (3)	0.009 (3)
C15B	0.052 (4)	0.039 (4)	0.049 (4)	−0.005 (3)	−0.026 (3)	0.003 (3)

Geometric parameters (Å, º)

O1A—C1A	1.225 (6)	O1B—C1B	1.236 (6)
O2A—C15A	1.330 (7)	O2B—C15B	1.295 (7)

197	O2A—H2A	0.92 (6)	O2B—H2B	0.90 (6)
198	O3A—C15A	1.174 (6)	O3B—C15B	1.175 (6)
199	N1A—C1A	1.353 (7)	N1B—C1B	1.332 (6)
200	N1A—N2A	1.395 (5)	N1B—N2B	1.403 (5)
201	N1A—C13A	1.477 (6)	N1B—C13B	1.466 (6)
202	N2A—C4A	1.272 (6)	N2B—C4B	1.287 (6)
203	C1A—C2A	1.465 (8)	C1B—C2B	1.478 (7)
204	C2A—C3A	1.441 (7)	C2B—C3B	1.472 (7)
205	C2A—H2A1	0.9700	C2B—H2B1	0.9700
206	C2A—H2A2	0.9700	C2B—H2B2	0.9700
207	C3A—C8A	1.480 (8)	C3B—C8B	1.448 (7)
208	C3A—C4A	1.515 (7)	C3B—C4B	1.478 (7)
209	C3A—H3A	0.9800	C3B—H3B	0.9800
210	C4A—C5A	1.459 (7)	C4B—C5B	1.459 (7)
211	C5A—C6A	1.377 (7)	C5B—C9B	1.388 (7)
212	C5A—C9A	1.399 (7)	C5B—C6B	1.397 (7)
213	C6A—C12A	1.380 (7)	C6B—C12B	1.389 (7)
214	C6A—C7A	1.526 (8)	C6B—C7B	1.501 (7)
215	C7A—C8A	1.457 (7)	C7B—C8B	1.461 (7)
216	C7A—H7A1	0.9700	C7B—H7B1	0.9700
217	C7A—H7A2	0.9700	C7B—H7B2	0.9700
218	C8A—H8A1	0.9700	C8B—H8B1	0.9700
219	C8A—H8A2	0.9700	C8B—H8B2	0.9700
220	C9A—C10A	1.376 (7)	C9B—C10B	1.384 (7)
221	C9A—H9A	0.9300	C9B—H9B	0.9300
222	C10A—C11A	1.365 (8)	C10B—C11B	1.384 (8)
223	C10A—H10A	0.9300	C10B—H10B	0.9300
224	C11A—C12A	1.378 (8)	C11B—C12B	1.357 (8)
225	C11A—H11A	0.9300	C11B—H11B	0.9300
226	C12A—H12A	0.9300	C12B—H12B	0.9300
227	C13A—C14A	1.481 (8)	C13B—C14B	1.503 (7)
228	C13A—H13A	0.9700	C13B—H13C	0.9700
229	C13A—H13B	0.9700	C13B—H13D	0.9700
230	C14A—C15A	1.504 (8)	C14B—C15B	1.514 (7)
231	C14A—H14A	0.9700	C14B—H14C	0.9700
232	C14A—H14B	0.9700	C14B—H14D	0.9700
233				
234	C15A—O2A—H2A	107 (4)	C15B—O2B—H2B	102 (4)
235	C1A—N1A—N2A	125.5 (5)	C1B—N1B—N2B	125.6 (4)
236	C1A—N1A—C13A	120.9 (5)	C1B—N1B—C13B	121.2 (4)
237	N2A—N1A—C13A	113.5 (4)	N2B—N1B—C13B	112.4 (4)
238	C4A—N2A—N1A	119.2 (4)	C4B—N2B—N1B	118.6 (4)
239	O1A—C1A—N1A	121.5 (5)	O1B—C1B—N1B	119.5 (5)
240	O1A—C1A—C2A	124.3 (5)	O1B—C1B—C2B	124.2 (5)
241	N1A—C1A—C2A	114.2 (5)	N1B—C1B—C2B	116.2 (5)
242	C3A—C2A—C1A	117.4 (5)	C3B—C2B—C1B	117.4 (5)
243	C3A—C2A—H2A1	107.9	C3B—C2B—H2B1	108.0
244	C1A—C2A—H2A1	107.9	C1B—C2B—H2B1	108.0

245	C3A—C2A—H2A2	107.9	C3B—C2B—H2B2	108.0
246	C1A—C2A—H2A2	107.9	C1B—C2B—H2B2	108.0
247	H2A1—C2A—H2A2	107.2	H2B1—C2B—H2B2	107.2
248	C2A—C3A—C8A	120.7 (5)	C8B—C3B—C2B	120.4 (5)
249	C2A—C3A—C4A	111.5 (5)	C8B—C3B—C4B	114.2 (5)
250	C8A—C3A—C4A	112.6 (5)	C2B—C3B—C4B	112.9 (5)
251	C2A—C3A—H3A	103.2	C8B—C3B—H3B	101.9
252	C8A—C3A—H3A	103.2	C2B—C3B—H3B	101.9
253	C4A—C3A—H3A	103.2	C4B—C3B—H3B	101.9
254	N2A—C4A—C5A	119.0 (5)	N2B—C4B—C5B	117.1 (5)
255	N2A—C4A—C3A	121.2 (5)	N2B—C4B—C3B	123.2 (5)
256	C5A—C4A—C3A	119.6 (5)	C5B—C4B—C3B	119.7 (5)
257	C6A—C5A—C9A	118.3 (5)	C9B—C5B—C6B	118.4 (5)
258	C6A—C5A—C4A	121.7 (5)	C9B—C5B—C4B	121.7 (5)
259	C9A—C5A—C4A	119.9 (5)	C6B—C5B—C4B	119.9 (5)
260	C5A—C6A—C12A	120.4 (5)	C12B—C6B—C5B	119.1 (5)
261	C5A—C6A—C7A	119.2 (5)	C12B—C6B—C7B	119.6 (5)
262	C12A—C6A—C7A	120.4 (6)	C5B—C6B—C7B	121.3 (5)
263	C8A—C7A—C6A	114.2 (5)	C8B—C7B—C6B	113.1 (5)
264	C8A—C7A—H7A1	108.7	C8B—C7B—H7B1	109.0
265	C6A—C7A—H7A1	108.7	C6B—C7B—H7B1	109.0
266	C8A—C7A—H7A2	108.7	C8B—C7B—H7B2	109.0
267	C6A—C7A—H7A2	108.7	C6B—C7B—H7B2	109.0
268	H7A1—C7A—H7A2	107.6	H7B1—C7B—H7B2	107.8
269	C7A—C8A—C3A	116.2 (5)	C3B—C8B—C7B	118.4 (5)
270	C7A—C8A—H8A1	108.2	C3B—C8B—H8B1	107.7
271	C3A—C8A—H8A1	108.2	C7B—C8B—H8B1	107.7
272	C7A—C8A—H8A2	108.2	C3B—C8B—H8B2	107.7
273	C3A—C8A—H8A2	108.2	C7B—C8B—H8B2	107.7
274	H8A1—C8A—H8A2	107.4	H8B1—C8B—H8B2	107.1
275	C10A—C9A—C5A	120.6 (6)	C10B—C9B—C5B	122.4 (5)
276	C10A—C9A—H9A	119.7	C10B—C9B—H9B	118.8
277	C5A—C9A—H9A	119.7	C5B—C9B—H9B	118.8
278	C11A—C10A—C9A	120.5 (6)	C11B—C10B—C9B	117.6 (6)
279	C11A—C10A—H10A	119.7	C11B—C10B—H10B	121.2
280	C9A—C10A—H10A	119.7	C9B—C10B—H10B	121.2
281	C10A—C11A—C12A	119.4 (6)	C12B—C11B—C10B	121.4 (6)
282	C10A—C11A—H11A	120.3	C12B—C11B—H11B	119.3
283	C12A—C11A—H11A	120.3	C10B—C11B—H11B	119.3
284	C11A—C12A—C6A	120.8 (6)	C11B—C12B—C6B	121.0 (5)
285	C11A—C12A—H12A	119.6	C11B—C12B—H12B	119.5
286	C6A—C12A—H12A	119.6	C6B—C12B—H12B	119.5
287	N1A—C13A—C14A	110.2 (5)	N1B—C13B—C14B	110.8 (4)
288	N1A—C13A—H13A	109.6	N1B—C13B—H13C	109.5
289	C14A—C13A—H13A	109.6	C14B—C13B—H13C	109.5
290	N1A—C13A—H13B	109.6	N1B—C13B—H13D	109.5
291	C14A—C13A—H13B	109.6	C14B—C13B—H13D	109.5
292	H13A—C13A—H13B	108.1	H13C—C13B—H13D	108.1

293	C13A—C14A—C15A	111.9 (5)	C13B—C14B—C15B	111.7 (5)
294	C13A—C14A—H14A	109.2	C13B—C14B—H14C	109.3
295	C15A—C14A—H14A	109.2	C15B—C14B—H14C	109.3
296	C13A—C14A—H14B	109.2	C13B—C14B—H14D	109.3
297	C15A—C14A—H14B	109.2	C15B—C14B—H14D	109.3
298	H14A—C14A—H14B	107.9	H14C—C14B—H14D	107.9
299	O3A—C15A—O2A	123.1 (6)	O3B—C15B—O2B	123.7 (6)
300	O3A—C15A—C14A	124.0 (6)	O3B—C15B—C14B	122.9 (6)
301	O2A—C15A—C14A	112.9 (6)	O2B—C15B—C14B	113.3 (6)
302				
303	C1A—N1A—N2A—C4A	−14.3 (8)	C1B—N1B—N2B—C4B	−10.7 (7)
304	C13A—N1A—N2A—C4A	169.3 (5)	C13B—N1B—N2B—C4B	179.9 (4)
305	N2A—N1A—C1A—O1A	−176.4 (5)	N2B—N1B—C1B—O1B	−175.2 (5)
306	C13A—N1A—C1A—O1A	−0.3 (9)	C13B—N1B—C1B—O1B	−6.7 (8)
307	N2A—N1A—C1A—C2A	2.0 (8)	N2B—N1B—C1B—C2B	3.0 (8)
308	C13A—N1A—C1A—C2A	178.1 (5)	C13B—N1B—C1B—C2B	171.5 (5)
309	O1A—C1A—C2A—C3A	−156.2 (7)	O1B—C1B—C2B—C3B	−164.7 (6)
310	N1A—C1A—C2A—C3A	25.5 (9)	N1B—C1B—C2B—C3B	17.2 (8)
311	C1A—C2A—C3A—C8A	−173.7 (6)	C1B—C2B—C3B—C8B	−167.9 (6)
312	C1A—C2A—C3A—C4A	−38.1 (9)	C1B—C2B—C3B—C4B	−28.0 (8)
313	N1A—N2A—C4A—C5A	−176.8 (5)	N1B—N2B—C4B—C5B	179.7 (4)
314	N1A—N2A—C4A—C3A	−1.5 (8)	N1B—N2B—C4B—C3B	−3.0 (7)
315	C2A—C3A—C4A—N2A	26.7 (8)	C8B—C3B—C4B—N2B	164.1 (5)
316	C8A—C3A—C4A—N2A	166.0 (5)	C2B—C3B—C4B—N2B	21.7 (8)
317	C2A—C3A—C4A—C5A	−158.1 (6)	C8B—C3B—C4B—C5B	−18.6 (8)
318	C8A—C3A—C4A—C5A	−18.7 (8)	C2B—C3B—C4B—C5B	−161.0 (5)
319	N2A—C4A—C5A—C6A	171.5 (5)	N2B—C4B—C5B—C9B	−5.4 (7)
320	C3A—C4A—C5A—C6A	−3.9 (8)	C3B—C4B—C5B—C9B	177.1 (5)
321	N2A—C4A—C5A—C9A	−6.1 (8)	N2B—C4B—C5B—C6B	173.8 (5)
322	C3A—C4A—C5A—C9A	178.6 (5)	C3B—C4B—C5B—C6B	−3.7 (7)
323	C9A—C5A—C6A—C12A	0.2 (8)	C9B—C5B—C6B—C12B	2.4 (8)
324	C4A—C5A—C6A—C12A	−177.4 (5)	C4B—C5B—C6B—C12B	−176.8 (5)
325	C9A—C5A—C6A—C7A	179.9 (5)	C9B—C5B—C6B—C7B	−176.4 (5)
326	C4A—C5A—C6A—C7A	2.4 (8)	C4B—C5B—C6B—C7B	4.4 (7)
327	C5A—C6A—C7A—C8A	22.2 (9)	C12B—C6B—C7B—C8B	−162.0 (5)
328	C12A—C6A—C7A—C8A	−158.0 (6)	C5B—C6B—C7B—C8B	16.8 (8)
329	C6A—C7A—C8A—C3A	−46.2 (9)	C2B—C3B—C8B—C7B	−178.9 (6)
330	C2A—C3A—C8A—C7A	179.2 (7)	C4B—C3B—C8B—C7B	41.7 (8)
331	C4A—C3A—C8A—C7A	44.0 (8)	C6B—C7B—C8B—C3B	−40.6 (8)
332	C6A—C5A—C9A—C10A	−0.4 (8)	C6B—C5B—C9B—C10B	−2.3 (8)
333	C4A—C5A—C9A—C10A	177.2 (5)	C4B—C5B—C9B—C10B	176.9 (5)
334	C5A—C9A—C10A—C11A	0.1 (9)	C5B—C9B—C10B—C11B	1.5 (8)
335	C9A—C10A—C11A—C12A	0.5 (10)	C9B—C10B—C11B—C12B	−0.8 (9)
336	C10A—C11A—C12A—C6A	−0.7 (10)	C10B—C11B—C12B—C6B	1.0 (9)
337	C5A—C6A—C12A—C11A	0.4 (10)	C5B—C6B—C12B—C11B	−1.8 (8)
338	C7A—C6A—C12A—C11A	−179.4 (6)	C7B—C6B—C12B—C11B	177.0 (5)
339	C1A—N1A—C13A—C14A	−101.3 (6)	C1B—N1B—C13B—C14B	89.7 (6)
340	N2A—N1A—C13A—C14A	75.3 (6)	N2B—N1B—C13B—C14B	−100.4 (5)

N1A—C13A—C14A—C15A	−176.6 (5)
C13A—C14A—C15A—O3A	−115.0 (7)
C13A—C14A—C15A—O2A	65.3 (7)

N1B—C13B—C14B—C15B	179.8 (4)
C13B—C14B—C15B—O3B	10.4 (9)
C13B—C14B—C15B—O2B	−170.1 (5)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H2A···O1B	0.92 (7)	1.78 (7)	2.651 (6)	158 (6)
O2B—H2B···O1A	0.90 (6)	1.75 (6)	2.598 (7)	157 (5)

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