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Crystal structure of 4-(4b,8a-dihydro-9*H*-pyrido-[3,4-*b*]indol-1-yl)-7-methyl-2*H*-chromen-2-one

S. Samundeeswari,[a] Manohar V. Kulkarni[a]* and G. N. Anil Kumar[b]

[a]Department of Chemistry, Karnatak University, Dharwad, India, and [b]Department of Physics, M. S. Ramaiah Institute of Technology, Bangalore, India. *Correspondence e-mail: anilgn@msrit.edu

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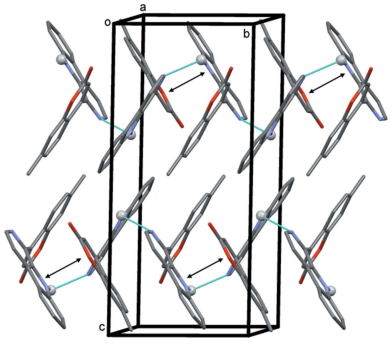
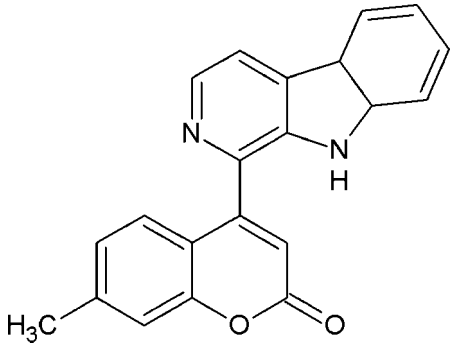
The title compound, $C_{21}H_{14}N_2O_2$, was prepared by Pictet–Spengler cyclization of tryptamine and 4-formyl coumarin. In the molecule, the dihedral angle between the mean planes of the coumarin and β-carboline ring systems is 63.8 (2)°. In the crystal, molecules are linked *via* N—H···N hydrogen bonds, forming chains along the *b*-axis direction. Within the chains, there are a number of offset π–π interactions present [shortest intercentroid distance = 3.457 (2) Å].

1. Chemical context

Naturally occurring coumarins (Murry, 2002) and their derivatives have a vast number of applications in different areas. They are precursor reagents for synthetic anti-coagulants (Bairagi *et al.*, 2012), the most notable being warfarin (Holbrook *et al.*, 2005). Coumarin dyes are also widely used in blue–green organic dyes (Schafer, 1990; Duarte & Hillman, 1990; Duarte, 2003) and in OLED emitters (Duarte *et al.*, 2005). Norharman is a β-carboline alkaloid which has the basic structural unit for a wide range of naturally occurring compounds, and is found in plants, animals and humans (Fekkes *et al.*, 1992). They are used widely as neurotoxins to Parkinson's disease (Kuhn *et al.*, 1996) and as mediators in the mutagenesis of DNA in the presence of another molecule (Mori *et al.*, 1996). Given the ongoing research into the biological functions of norharman and the many related β-carboline derivatives, a single-crystal X-ray structure of norharman would be of use in theoretical modelling and related structural work. Norharman exhibits a one-dimensional herringbone motif (Thatcher & Douthwaite, 2011). Due to their extensive natural occurrence and common biological origin, there are no reports on compounds which contain these two systems in a single molecule. It was hence thought of considerable biological interest to synthesize new molecules which contain both β-carboline and coumarin ring systems.





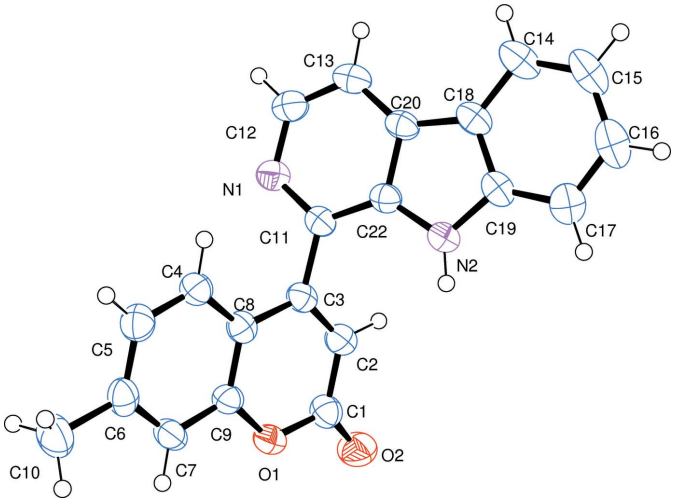


Figure 1
The molecular structure of the title compound, with the atom labelling and displacement ellipsoids drawn at the 50% probability level.

2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The coumarin (r.m.s. deviation = 0.019 Å) and β-carboline (r.m.s. deviation = 0.034 Å) ring systems exhibit an *s-trans* arrangement across the bridging C7—C6 bond; their mean planes are inclined to one another by 63.8 (2)°.

3. Supramolecular features

In the crystal, molecules are linked *via* N—H···N hydrogen bonds, forming chains along [010]; see Table 1 and Fig. 2. Within the chains there are a number of offset π–π interactions present; the shortest intercentroid distance of 3.457 (2) Å, involves rings N2/C18–C20/C22 of the β-carboline ring system and O1/C1–C3/C8/C9 of the coumarin system.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.37, last update May 2016; Groom *et al.*, 2016) using 4,7-dimethyl-2*H*-chromen-2-one as the main skeleton revealed the presence of 66 structures. However, only six of these structures contain the 7-methyl-4-phenyl-2*H*-chromen-2-one nucleus (refcodes: BUFQUQ, FINNEX, GUFTUY, IFUMED, LENYIO, DUVVIB). There were no structures reported for a search of 7-methyl-4-(pyridin-2-yl)-2*H*-chromen-2-one skeleton.

5. Synthesis and crystallization

Acetic acid (10 ml) was added drop wise, at 273 K, to a mixture of tryptamine (1 eq) and 4-formyl coumarin (1 eq). The reaction mixture was stirred at room temperature for *ca* 12 h. After completion of the reaction, the solid that separated was filtered, washed several times with water and dried (yield >70%) to give the intermediate. This intermediate compound (1 eq) was taken in 10 ml of dry chloroform and 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (2 eq) was added at intervals of 5 min in cold conditions, 273 K. Stirring was continued for *ca* 10 h. The reaction mixture was then quenched using aqueous sodium bicarbonate and extracted with chloroform. The organic layer was washed 2–3 times with sodium bicarbonate, water and brine solution, dried using sodium sulfate, and concentrated to afford the crude title product. It was purified by flash chromatography using 230–400 mesh silica-gels (35% ethyl acetate in hexane mixture; yield 75%). The solid obtained was recrystallized from dichloromethane, giving colourless block-like crystals of the title compound on slow evaporation of the solvent

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1^{i}	0.86	2.47	2.994 (3)	120

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93–0.96 Å, and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{eq}(C,N) for other H atoms.

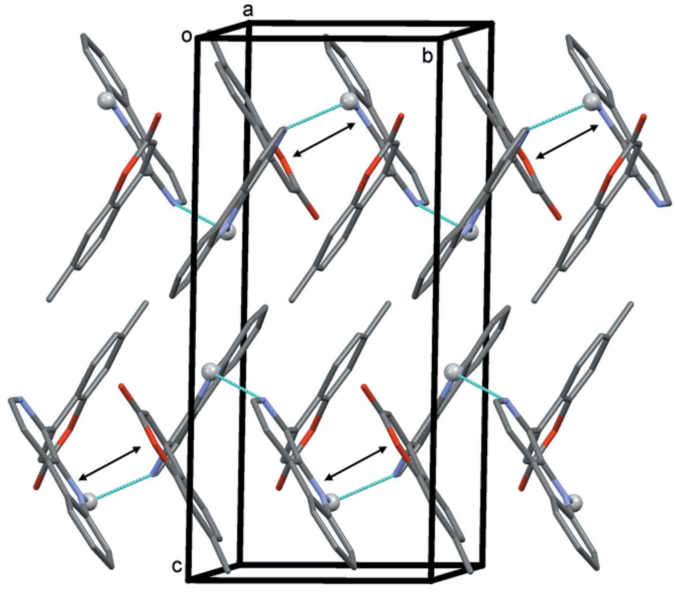


Figure 2
A view along the *a* axis of the crystal packing of the title compound. The N—H···N hydrogen bonds are shown as dashed lines (see Table 1), and the shortest offset π–π interactions by a double-headed arrow. For clarity, only H atom H2 (grey ball) has been included.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{14}N_2O_2$
M_r	326.34
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.6784 (8), 8.0954 (6), 17.9032 (14)
β (°)	98.105 (5)
V (Å^3)	1532.2 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.20 × 0.15 × 0.10
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.941, 0.971
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11601, 2848, 1446
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.060, 0.166, 0.94
No. of reflections	2848
No. of parameters	227
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.27

Computer programs: *SMART* and *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

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Computing details

Data collection: *SMART* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

4-(4b,8a-Dihydro-9*H*-pyrido[3,4-*b*]indol-1-yl)-7-methyl-2*H*-chromen-2-one

Crystal data

$C_{21}H_{14}N_2O_2$
M_r = 326.34
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.6784 (8) Å
b = 8.0954 (6) Å
c = 17.9032 (14) Å
β = 98.105 (5)°
V = 1532.2 (2) Å^3
Z = 4
$F(000)$ = 680
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1990 reflections
θ = 3.3–26.4°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
T_{min} = 0.941, T_{max} = 0.971
11601 measured reflections
2848 independent reflections
1446 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 25.5°, θ_{min} = 1.9°
h = −12→11
k = −9→9
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.166
S = 0.94
2848 reflections
227 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

$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ 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}
O1	0.85044 (16)	0.1787 (2)	0.24656 (10)	0.0445 (6)
O2	0.87191 (18)	0.3169 (3)	0.35363 (12)	0.0602 (7)
N1	0.3818 (2)	0.2835 (3)	0.17822 (12)	0.0413 (7)
N2	0.4360 (2)	0.0447 (3)	0.35591 (12)	0.0407 (7)
H2	0.5149	0.0259	0.3707	0.049*
C1	0.7990 (3)	0.2565 (4)	0.30347 (17)	0.0429 (8)
C2	0.6635 (2)	0.2570 (3)	0.29733 (15)	0.0427 (8)
H2A	0.6264	0.3055	0.3359	0.051*
C3	0.5878 (2)	0.1912 (3)	0.23877 (15)	0.0341 (7)
C4	0.5774 (3)	0.0283 (4)	0.11849 (15)	0.0401 (8)
H4	0.4895	0.0273	0.1126	0.048*
C5	0.6389 (3)	−0.0500 (4)	0.06608 (15)	0.0459 (8)
H5	0.5921	−0.1047	0.0258	0.055*
C6	0.7705 (3)	−0.0486 (4)	0.07241 (15)	0.0427 (8)
C7	0.8384 (3)	0.0291 (4)	0.13369 (15)	0.0431 (8)
H7	0.9263	0.0302	0.1395	0.052*
C8	0.6443 (2)	0.1088 (3)	0.18005 (15)	0.0353 (7)
C9	0.7754 (2)	0.1051 (4)	0.18637 (15)	0.0370 (7)
C10	0.8380 (3)	−0.1277 (4)	0.01293 (17)	0.0635 (10)
H10A	0.924	−0.1509	0.0339	0.095*
H10B	0.8369	−0.0537	−0.0291	0.095*
H10C	0.7961	−0.2287	−0.0038	0.095*
C11	0.4475 (2)	0.2009 (4)	0.23601 (15)	0.0356 (7)
C12	0.2541 (3)	0.2974 (4)	0.17666 (16)	0.0464 (8)
H12	0.2093	0.3581	0.1376	0.056*
C13	0.1874 (3)	0.2280 (4)	0.22862 (17)	0.0449 (8)
H13	0.1	0.2388	0.2242	0.054*
C14	0.1087 (3)	0.0115 (4)	0.37782 (19)	0.0542 (9)
H14	0.0321	0.0482	0.3519	0.065*
C15	0.1115 (3)	−0.0820 (4)	0.44170 (19)	0.0595 (10)
H15	0.0361	−0.1107	0.4587	0.071*
C16	0.2255 (3)	−0.1345 (4)	0.48153 (18)	0.0589 (9)
H16	0.2248	−0.1961	0.5253	0.071*
C17	0.3403 (3)	−0.0978 (4)	0.45785 (16)	0.0489 (9)
H17	0.4166	−0.1329	0.4847	0.059*
C18	0.2223 (3)	0.0506 (4)	0.35234 (15)	0.0411 (8)

C19	0.3358 (3)	−0.0063 (4)	0.39225 (15)	0.0403 (7)
C20	0.2544 (2)	0.1410 (3)	0.28810 (16)	0.0370 (7)
C22	0.3865 (2)	0.1313 (3)	0.29183 (15)	0.0342 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0319 (11)	0.0588 (15)	0.0433 (12)	−0.0005 (10)	0.0069 (9)	−0.0029 (11)
O2	0.0450 (13)	0.0760 (18)	0.0575 (14)	−0.0050 (12)	−0.0001 (11)	−0.0134 (13)
N1	0.0356 (14)	0.0428 (17)	0.0454 (15)	0.0022 (12)	0.0056 (11)	0.0007 (13)
N2	0.0321 (13)	0.0503 (18)	0.0406 (14)	0.0004 (12)	0.0083 (11)	0.0003 (13)
C1	0.0423 (18)	0.044 (2)	0.0434 (18)	−0.0038 (16)	0.0090 (15)	−0.0018 (16)
C2	0.0376 (17)	0.049 (2)	0.0419 (18)	0.0018 (15)	0.0083 (14)	−0.0027 (16)
C3	0.0332 (15)	0.0306 (19)	0.0397 (16)	0.0001 (13)	0.0086 (13)	0.0041 (14)
C4	0.0372 (16)	0.043 (2)	0.0409 (17)	−0.0026 (15)	0.0071 (13)	0.0029 (15)
C5	0.048 (2)	0.048 (2)	0.0419 (18)	−0.0040 (16)	0.0066 (15)	0.0008 (16)
C6	0.055 (2)	0.037 (2)	0.0376 (17)	0.0076 (16)	0.0135 (15)	0.0056 (15)
C7	0.0359 (16)	0.049 (2)	0.0468 (18)	0.0071 (15)	0.0150 (14)	0.0058 (16)
C8	0.0385 (17)	0.0310 (19)	0.0373 (16)	−0.0013 (14)	0.0086 (13)	0.0032 (14)
C9	0.0311 (16)	0.041 (2)	0.0393 (17)	−0.0008 (14)	0.0060 (13)	0.0051 (15)
C10	0.070 (2)	0.071 (3)	0.054 (2)	0.0156 (19)	0.0223 (17)	−0.0037 (19)
C11	0.0333 (16)	0.036 (2)	0.0382 (16)	−0.0008 (14)	0.0086 (13)	−0.0025 (14)
C12	0.0383 (18)	0.050 (2)	0.0500 (19)	0.0041 (16)	0.0011 (15)	−0.0005 (16)
C13	0.0320 (16)	0.045 (2)	0.058 (2)	0.0013 (15)	0.0074 (15)	−0.0065 (17)
C14	0.0461 (19)	0.048 (2)	0.074 (2)	0.0008 (17)	0.0276 (17)	−0.0005 (19)
C15	0.064 (2)	0.051 (2)	0.073 (2)	−0.0048 (19)	0.0414 (19)	−0.001 (2)
C16	0.078 (2)	0.049 (2)	0.056 (2)	−0.006 (2)	0.0313 (19)	0.0005 (18)
C17	0.059 (2)	0.041 (2)	0.0487 (19)	0.0011 (17)	0.0127 (16)	0.0001 (16)
C18	0.0375 (17)	0.037 (2)	0.0509 (19)	−0.0006 (15)	0.0157 (14)	−0.0048 (16)
C19	0.0418 (17)	0.0379 (19)	0.0440 (18)	−0.0030 (15)	0.0157 (14)	−0.0038 (15)
C20	0.0310 (16)	0.036 (2)	0.0445 (17)	0.0027 (14)	0.0093 (13)	−0.0075 (15)
C22	0.0324 (16)	0.033 (2)	0.0372 (17)	0.0017 (13)	0.0059 (13)	−0.0078 (14)

Geometric parameters (Å, º)

O1—C1	1.377 (3)	C8—C9	1.389 (3)
O1—C9	1.383 (3)	C10—H10A	0.96
O2—C1	1.206 (3)	C10—H10B	0.96
N1—C11	1.344 (3)	C10—H10C	0.96
N1—C12	1.365 (3)	C11—C22	1.388 (3)
N2—C22	1.384 (3)	C12—C13	1.369 (4)
N2—C19	1.391 (3)	C12—H12	0.93
N2—H2	0.86	C13—C20	1.388 (4)
C1—C2	1.435 (4)	C13—H13	0.93
C2—C3	1.341 (3)	C14—C15	1.368 (4)
C2—H2A	0.93	C14—C18	1.392 (4)
C3—C8	1.447 (3)	C14—H14	0.93
C3—C11	1.494 (3)	C15—C16	1.388 (4)

C4—C5	1.374 (4)	C15—H15	0.93
C4—C8	1.388 (3)	C16—C17	1.385 (4)
C4—H4	0.93	C16—H16	0.93
C5—C6	1.394 (4)	C17—C19	1.384 (4)
C5—H5	0.93	C17—H17	0.93
C6—C7	1.379 (4)	C18—C19	1.396 (4)
C6—C10	1.510 (4)	C18—C20	1.445 (4)
C7—C9	1.378 (4)	C20—C22	1.405 (3)
C7—H7	0.93		

C1—O1—C9	121.7 (2)	H10A—C10—H10C	109.5
C11—N1—C12	117.9 (2)	H10B—C10—H10C	109.5
C22—N2—C19	108.0 (2)	N1—C11—C22	120.6 (2)
C22—N2—H2	126	N1—C11—C3	117.7 (2)
C19—N2—H2	126	C22—C11—C3	121.7 (2)
O2—C1—O1	117.0 (3)	C13—C12—N1	124.5 (3)
O2—C1—C2	126.4 (3)	C13—C12—H12	117.8
O1—C1—C2	116.6 (3)	N1—C12—H12	117.8
C3—C2—C1	123.3 (3)	C12—C13—C20	117.9 (3)
C3—C2—H2A	118.4	C12—C13—H13	121
C1—C2—H2A	118.4	C20—C13—H13	121
C2—C3—C8	119.0 (2)	C15—C14—C18	118.8 (3)
C2—C3—C11	119.7 (3)	C15—C14—H14	120.6
C8—C3—C11	121.3 (2)	C18—C14—H14	120.6
C5—C4—C8	121.1 (3)	C14—C15—C16	120.9 (3)
C5—C4—H4	119.4	C14—C15—H15	119.6
C8—C4—H4	119.4	C16—C15—H15	119.6
C4—C5—C6	121.0 (3)	C17—C16—C15	121.8 (3)
C4—C5—H5	119.5	C17—C16—H16	119.1
C6—C5—H5	119.5	C15—C16—H16	119.1
C7—C6—C5	118.6 (3)	C16—C17—C19	116.6 (3)
C7—C6—C10	120.3 (3)	C16—C17—H17	121.7
C5—C6—C10	121.0 (3)	C19—C17—H17	121.7
C6—C7—C9	119.7 (3)	C14—C18—C19	119.5 (3)
C6—C7—H7	120.1	C14—C18—C20	133.8 (3)
C9—C7—H7	120.1	C19—C18—C20	106.7 (2)
C9—C8—C4	117.0 (3)	C17—C19—N2	128.3 (3)
C9—C8—C3	118.0 (2)	C17—C19—C18	122.3 (3)
C4—C8—C3	124.9 (2)	N2—C19—C18	109.4 (2)
C7—C9—O1	116.1 (2)	C13—C20—C22	118.0 (3)
C7—C9—C8	122.5 (3)	C13—C20—C18	135.5 (3)
O1—C9—C8	121.4 (2)	C22—C20—C18	106.4 (2)
C6—C10—H10A	109.5	C11—C22—N2	129.7 (2)
C6—C10—H10B	109.5	C11—C22—C20	120.9 (3)
H10A—C10—H10B	109.5	N2—C22—C20	109.4 (2)
C6—C10—H10C	109.5		

C9—O1—C1—O2	179.9 (2)	C11—N1—C12—C13	−2.4 (4)

C9—O1—C1—C2	0.2 (4)	N1—C12—C13—C20	1.7 (4)
O2—C1—C2—C3	178.1 (3)	C18—C14—C15—C16	1.3 (5)
O1—C1—C2—C3	−2.2 (4)	C14—C15—C16—C17	−1.2 (5)
C1—C2—C3—C8	2.8 (4)	C15—C16—C17—C19	−0.2 (4)
C1—C2—C3—C11	−178.5 (3)	C15—C14—C18—C19	0.1 (4)
C8—C4—C5—C6	−1.1 (4)	C15—C14—C18—C20	178.5 (3)
C4—C5—C6—C7	1.8 (4)	C16—C17—C19—N2	−179.0 (3)
C4—C5—C6—C10	−177.1 (3)	C16—C17—C19—C18	1.6 (4)
C5—C6—C7—C9	−0.9 (4)	C22—N2—C19—C17	179.2 (3)
C10—C6—C7—C9	178.0 (3)	C22—N2—C19—C18	−1.3 (3)
C5—C4—C8—C9	−0.5 (4)	C14—C18—C19—C17	−1.6 (4)
C5—C4—C8—C3	−178.6 (3)	C20—C18—C19—C17	179.7 (3)
C2—C3—C8—C9	−1.4 (4)	C14—C18—C19—N2	178.9 (3)
C11—C3—C8—C9	179.8 (2)	C20—C18—C19—N2	0.1 (3)
C2—C3—C8—C4	176.6 (3)	C12—C13—C20—C22	0.7 (4)
C11—C3—C8—C4	−2.2 (4)	C12—C13—C20—C18	−179.2 (3)
C6—C7—C9—O1	179.3 (2)	C14—C18—C20—C13	2.6 (6)
C6—C7—C9—C8	−0.8 (4)	C19—C18—C20—C13	−178.9 (3)
C1—O1—C9—C7	−179.1 (2)	C14—C18—C20—C22	−177.4 (3)
C1—O1—C9—C8	1.0 (4)	C19—C18—C20—C22	1.1 (3)
C4—C8—C9—C7	1.5 (4)	N1—C11—C22—N2	−178.8 (2)
C3—C8—C9—C7	179.7 (2)	C3—C11—C22—N2	−0.5 (4)
C4—C8—C9—O1	−178.6 (2)	N1—C11—C22—C20	2.0 (4)
C3—C8—C9—O1	−0.4 (4)	C3—C11—C22—C20	−179.7 (2)
C12—N1—C11—C22	0.4 (4)	C19—N2—C22—C11	−177.3 (3)
C12—N1—C11—C3	−177.9 (2)	C19—N2—C22—C20	2.0 (3)
C2—C3—C11—N1	118.4 (3)	C13—C20—C22—C11	−2.5 (4)
C8—C3—C11—N1	−62.9 (3)	C18—C20—C22—C11	177.4 (2)
C2—C3—C11—C22	−60.0 (4)	C13—C20—C22—N2	178.1 (2)
C8—C3—C11—C22	118.8 (3)	C18—C20—C22—N2	−1.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.86	2.47	2.994 (3)	120

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+1/2.