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Crystal structure of (*E*)-2-(4-methoxystyryl)-2,3-dihydro-1*H*-perimidine acetonitrile monosolvate

A. Manimekalai,[a]* N. Vijayalakshmi[a] and S. Selvanayagam[b]

[a]Department of Chemistry, Faculty of Science, Annamalai University, Annamalainagar 608 002, India, and [b]Department of Physics, Kalasalingam University, Krishnankoil 626 126, India. *Correspondence e-mail: profmeka1@gmail.com

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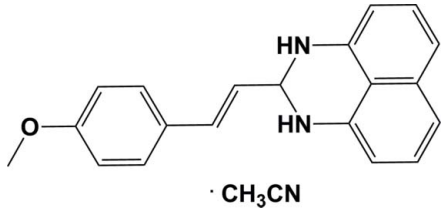
The title compound, $C_{20}H_{18}N_2O\cdot CH_3CN$, a perimidine derivative, crystallized as an acetonitrile monosolvate. The planes of the naphthalene ring system and the methoxyphenyl ring are oriented almost perpendicular to one another, with a dihedral angle of 87.61 (6)°. The conformation about the C=C bond is *E*. The hexahydropyrimidine ring has an envelope conformation, with the methine C atom as the flap. In the crystal, the molecules are linked by N—H···N hydrogen bonds involving the acetonitrile solvent molecule as acceptor, forming zigzag chains propagating along [100].

Keywords: crystal structure; perimidine derivative; bifurcated hydrogen bonding.

CCDC reference: 1015577

1. Related literature

For the diverse range of biological activities of perimidines, see: Bu *et al.* (2001); Ivica *et al.* (2008); Azeez & Salih (2014). For a related structure, see: Maloney *et al.* (2013).



2. Experimental

2.1. Crystal data

$C_{20}H_{18}N_2O\cdot C_2H_3N$ M_r = 343.42

Triclinic, $P\overline{1}$
a = 7.8128 (7) Å
b = 8.4641 (7) Å
c = 14.7427 (14) Å
α = 79.513 (7)°
β = 83.861 (7)°
γ = 77.076 (7)°
V = 932.11 (15) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 292 K
0.30 × 0.30 × 0.19 mm

2.2. Data collection

Oxford Diffraction Gemini/EOS CCD diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2008)
T_{min} = 0.840, T_{max} = 0.890
5843 measured reflections
3569 independent reflections
2940 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.03
3569 reflections
246 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.88 (2)	2.49 (2)	3.305 (3)	154.1 (15)
N2—H2···N3^{ii}	0.84 (2)	2.44 (2)	3.234 (2)	156.8 (16)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2008); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2762).

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supporting information

Acta Cryst. (2014). E**70**, o959 [doi:10.1107/S1600536814017000]

Crystal structure of (*E*)-2-(4-methoxystyryl)-2,3-dihydro-1*H*-perimidine acetonitrile monosolvate

A. Manimekalai, N. Vijayalakshmi and S. Selvanayagam

S1. Synthesis and crystallization

A mixture of 4-methoxy-trans-cinnamaldehyde (0.01 mol), 1,8-diaminonaphthalene (0.01 mol) and a pinch of $MgSO_4.7H_2O$ was finely grinded in a mortor. The mixture was then kept under microwave irradiation (LG Grill, Intellowave 160-800 W, consumption 800 W, output power 320 W and frequency 2450 MHz) operating in a cyclic mode to prevent intense boiling of the sample as well as aggregation of 320 W at 12 min. A brown colour solid separated out which was repeatedly recrystallized using CH_2Cl_2:CH_3CN (1:5) to obtain brown crystals.

S2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Atoms H1 and H2 were located from a difference Fourier map and freely refined. The C-bound H atoms were positioned geometrically and allowed to ride on their parent atoms: C—H = 0.93-0.96 Å with U_{iso}(H) = 1.5U_{eq}(C-methyl) and U_{iso}(H) = 1.2U_{eq} for other C atoms.

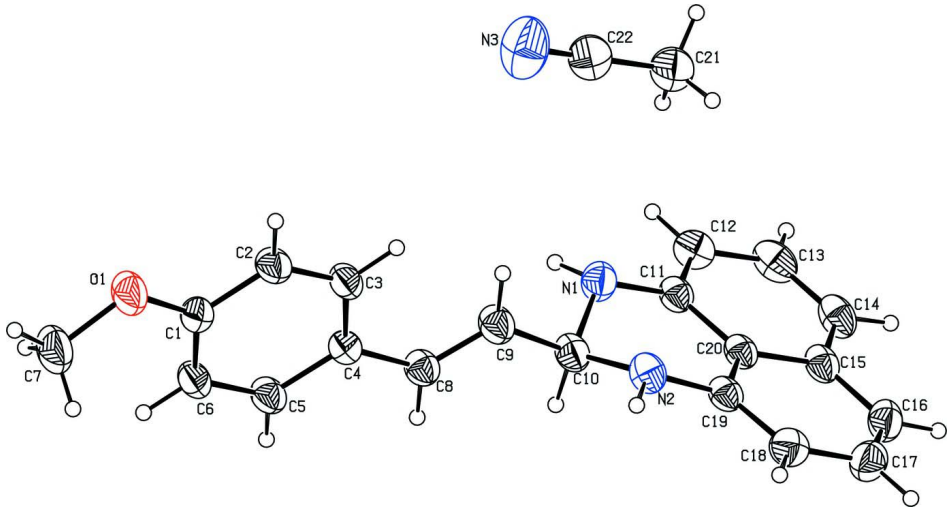

Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

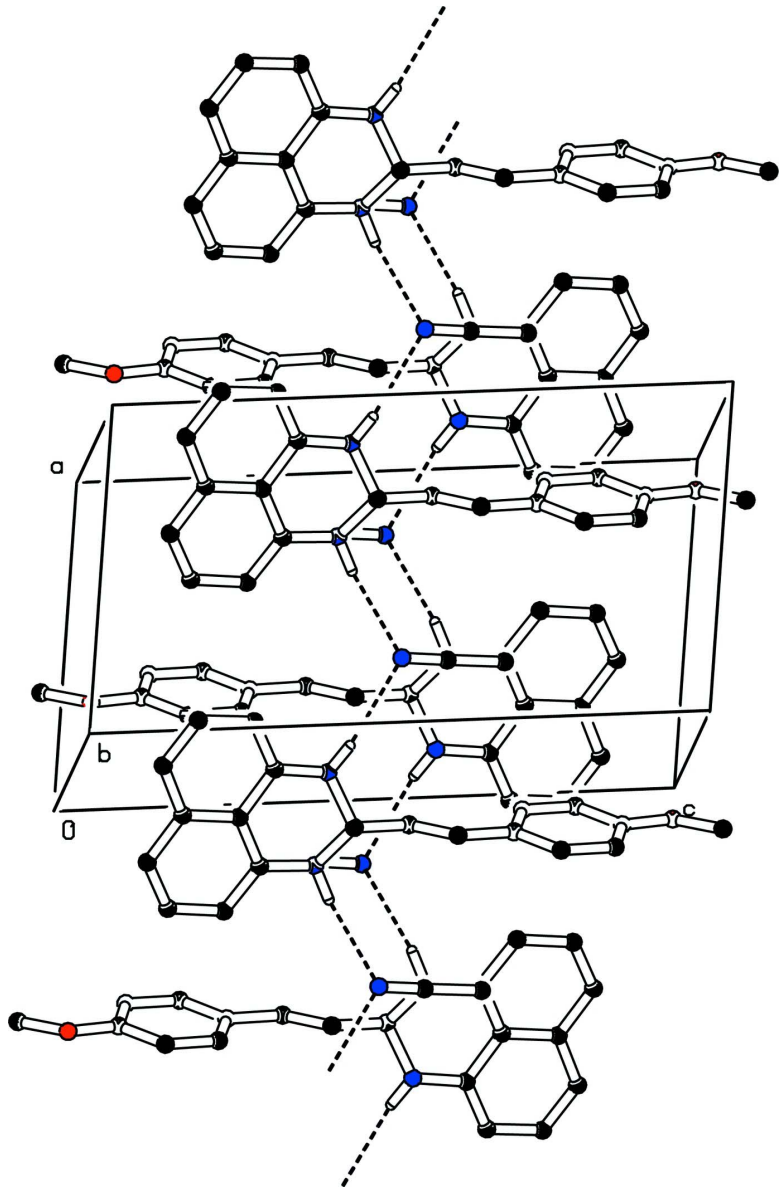

Figure 2

Crystal packing of the title compound, viewed along the b axis, showing the hydrogen bonds as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity).

(*E*)-2-(4-Methoxystyryl)-2,3-dihydro-1*H*-perimidine acetonitrile monosolvate

Crystal data

$C_{20}H_{18}N_2O\cdot C_2H_3N$
M_r = 343.42
Triclinic, $P\overline{1}$
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α = 79.513 (7)°
β = 83.861 (7)°
γ = 77.076 (7)°
V = 932.11 (15) Å^3
Z = 2
$F(000)$ = 364
D_x = 1.224 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2420 reflections
θ = 3.8–26.7°

μ = 0.60 mm^{-1}
T = 292 K

Block, colourless
0.30 × 0.30 × 0.19 mm

Data collection

Oxford Diffraction Gemini/EOS CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2008)
T_{min} = 0.840, T_{max} = 0.890
5843 measured reflections

3569 independent reflections
2940 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 72.0°, θ_{min} = 3.1°
h = −8→9
k = −9→10
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.03
3569 reflections
246 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 0.0999P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.038 (2)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.19042 (19)	0.58843 (14)	0.01925 (7)	0.0797 (4)
N1	0.36331 (18)	0.27710 (16)	0.60754 (8)	0.0591 (3)
H1	0.465 (2)	0.295 (2)	0.5801 (12)	0.070 (5)*
N2	0.10070 (17)	0.19149 (16)	0.60104 (8)	0.0565 (3)
H2	0.027 (2)	0.170 (2)	0.5698 (12)	0.066 (5)*
N3	0.2463 (3)	0.7821 (3)	0.50695 (14)	0.1157 (7)
C1	0.2221 (2)	0.49942 (17)	0.10443 (9)	0.0541 (3)
C2	0.1430 (2)	0.57618 (17)	0.17864 (10)	0.0579 (4)
H2A	0.0739	0.6816	0.1677	0.069*
C3	0.16593 (19)	0.49800 (17)	0.26764 (9)	0.0534 (3)
H3	0.1121	0.5512	0.3164	0.064*
C4	0.26887 (17)	0.33940 (16)	0.28649 (9)	0.0469 (3)
C5	0.34737 (19)	0.26561 (17)	0.21182 (9)	0.0550 (4)
H5	0.4168	0.1603	0.2227	0.066*
C6	0.3260 (2)	0.34347 (18)	0.12137 (10)	0.0577 (4)
H6	0.3811	0.2912	0.0725	0.069*

C7	0.2448 (3)	0.5081 (3)	−0.05893 (11)	0.0848 (6)
H7A	0.3710	0.4773	−0.0645	0.127*
H7B	0.2041	0.5813	−0.1137	0.127*
H7C	0.1961	0.4116	−0.0514	0.127*
C8	0.29403 (18)	0.25000 (17)	0.38074 (9)	0.0518 (3)
H8	0.3531	0.1408	0.3858	0.062*
C9	0.2431 (2)	0.30587 (18)	0.45874 (10)	0.0599 (4)
H9	0.1876	0.4158	0.4560	0.072*
C10	0.26927 (19)	0.20271 (18)	0.55165 (9)	0.0550 (4)
H10	0.3362	0.0925	0.5446	0.066*
C11	0.37511 (18)	0.20595 (16)	0.70001 (9)	0.0495 (3)
C12	0.5038 (2)	0.22395 (19)	0.75213 (11)	0.0616 (4)
H12	0.5903	0.2804	0.7247	0.074*
C13	0.5045 (2)	0.1575 (2)	0.84619 (11)	0.0668 (4)
H13	0.5919	0.1710	0.8805	0.080*
C14	0.3811 (2)	0.0739 (2)	0.88851 (10)	0.0625 (4)
H14	0.3848	0.0307	0.9511	0.075*
C15	0.24639 (18)	0.05219 (16)	0.83788 (9)	0.0526 (3)
C16	0.1127 (2)	−0.03118 (19)	0.87873 (11)	0.0644 (4)
H16	0.1144	−0.0795	0.9407	0.077*
C17	−0.0188 (2)	−0.0412 (2)	0.82776 (12)	0.0684 (4)
H17	−0.1062	−0.0964	0.8555	0.082*
C18	−0.02515 (19)	0.03028 (19)	0.73418 (11)	0.0608 (4)
H18	−0.1172	0.0235	0.7009	0.073*
C19	0.10345 (17)	0.10989 (16)	0.69160 (9)	0.0489 (3)
C20	0.24349 (16)	0.12050 (15)	0.74293 (9)	0.0460 (3)
C21	0.2512 (3)	0.6594 (2)	0.67886 (14)	0.0835 (5)
H21A	0.3480	0.5668	0.6877	0.125*
H21B	0.1430	0.6246	0.7000	0.125*
H21C	0.2655	0.7410	0.7134	0.125*
C22	0.2473 (2)	0.7278 (2)	0.58287 (14)	0.0749 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1225 (10)	0.0607 (7)	0.0433 (6)	−0.0013 (6)	0.0027 (6)	−0.0027 (5)
N1	0.0629 (8)	0.0651 (8)	0.0494 (7)	−0.0174 (6)	−0.0017 (6)	−0.0063 (6)
N2	0.0556 (7)	0.0655 (8)	0.0475 (7)	−0.0064 (5)	−0.0112 (5)	−0.0100 (5)
N3	0.1015 (14)	0.168 (2)	0.0829 (13)	−0.0474 (14)	−0.0207 (10)	−0.0021 (13)
C1	0.0677 (8)	0.0491 (7)	0.0437 (7)	−0.0121 (6)	0.0009 (6)	−0.0056 (6)
C2	0.0710 (9)	0.0436 (7)	0.0528 (8)	0.0004 (6)	−0.0005 (7)	−0.0093 (6)
C3	0.0616 (8)	0.0503 (7)	0.0460 (7)	−0.0042 (6)	0.0024 (6)	−0.0147 (6)
C4	0.0472 (7)	0.0477 (7)	0.0460 (7)	−0.0083 (5)	−0.0015 (5)	−0.0112 (5)
C5	0.0619 (8)	0.0473 (7)	0.0508 (8)	0.0012 (6)	−0.0017 (6)	−0.0116 (6)
C6	0.0690 (9)	0.0543 (8)	0.0473 (7)	−0.0054 (6)	0.0059 (6)	−0.0168 (6)
C7	0.1177 (16)	0.0847 (12)	0.0441 (9)	−0.0088 (11)	0.0010 (9)	−0.0093 (8)
C8	0.0534 (7)	0.0503 (7)	0.0492 (7)	−0.0039 (6)	−0.0049 (6)	−0.0095 (6)
C9	0.0728 (9)	0.0531 (8)	0.0477 (8)	−0.0005 (7)	−0.0048 (7)	−0.0079 (6)

C10	0.0624 (8)	0.0534 (8)	0.0449 (7)	−0.0015 (6)	−0.0043 (6)	−0.0096 (6)
C11	0.0507 (7)	0.0481 (7)	0.0464 (7)	−0.0012 (5)	−0.0034 (5)	−0.0108 (5)
C12	0.0558 (8)	0.0629 (9)	0.0677 (9)	−0.0096 (7)	−0.0083 (7)	−0.0157 (7)
C13	0.0647 (9)	0.0715 (10)	0.0651 (9)	0.0021 (8)	−0.0226 (7)	−0.0230 (8)
C14	0.0692 (9)	0.0649 (9)	0.0459 (7)	0.0092 (7)	−0.0135 (7)	−0.0127 (6)
C15	0.0569 (8)	0.0471 (7)	0.0455 (7)	0.0080 (6)	−0.0027 (6)	−0.0097 (5)
C16	0.0738 (10)	0.0573 (8)	0.0506 (8)	−0.0005 (7)	0.0061 (7)	−0.0015 (6)
C17	0.0660 (9)	0.0624 (9)	0.0722 (10)	−0.0144 (7)	0.0123 (8)	−0.0081 (8)
C18	0.0531 (8)	0.0610 (9)	0.0688 (9)	−0.0100 (6)	−0.0021 (7)	−0.0154 (7)
C19	0.0499 (7)	0.0450 (7)	0.0481 (7)	0.0011 (5)	−0.0038 (5)	−0.0119 (5)
C20	0.0474 (7)	0.0419 (6)	0.0443 (7)	0.0033 (5)	−0.0029 (5)	−0.0111 (5)
C21	0.0940 (13)	0.0738 (11)	0.0794 (12)	−0.0137 (10)	0.0011 (10)	−0.0126 (9)
C22	0.0647 (10)	0.0877 (12)	0.0764 (12)	−0.0204 (9)	−0.0083 (8)	−0.0161 (10)

Geometric parameters (Å, º)

O1—C1	1.3581 (16)	C9—C10	1.4917 (19)
O1—C7	1.422 (2)	C9—H9	0.9300
N1—C11	1.3902 (17)	C10—H10	0.9800
N1—C10	1.4630 (19)	C11—C12	1.379 (2)
N1—H1	0.882 (18)	C11—C20	1.4170 (19)
N2—C19	1.3869 (17)	C12—C13	1.398 (2)
N2—C10	1.4489 (19)	C12—H12	0.9300
N2—H2	0.842 (18)	C13—C14	1.355 (2)
N3—C22	1.130 (2)	C13—H13	0.9300
C1—C6	1.381 (2)	C14—C15	1.415 (2)
C1—C2	1.3913 (19)	C14—H14	0.9300
C2—C3	1.3685 (19)	C15—C16	1.412 (2)
C2—H2A	0.9300	C15—C20	1.4144 (18)
C3—C4	1.3983 (19)	C16—C17	1.362 (2)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.3830 (18)	C17—C18	1.403 (2)
C4—C8	1.4666 (18)	C17—H17	0.9300
C5—C6	1.3852 (19)	C18—C19	1.368 (2)
C5—H5	0.9300	C18—H18	0.9300
C6—H6	0.9300	C19—C20	1.4226 (19)
C7—H7A	0.9600	C21—C22	1.429 (3)
C7—H7B	0.9600	C21—H21A	0.9600
C7—H7C	0.9600	C21—H21B	0.9600
C8—C9	1.312 (2)	C21—H21C	0.9600
C8—H8	0.9300		
C1—O1—C7	118.12 (13)	N2—C10—H10	109.7
C11—N1—C10	116.49 (12)	N1—C10—H10	109.7
C11—N1—H1	113.1 (11)	C9—C10—H10	109.7
C10—N1—H1	112.6 (11)	C12—C11—N1	122.29 (14)
C19—N2—C10	116.97 (11)	C12—C11—C20	119.28 (13)
C19—N2—H2	114.5 (12)	N1—C11—C20	118.32 (12)

C10—N2—H2	114.7 (12)	C11—C12—C13	120.18 (15)
O1—C1—C6	125.11 (13)	C11—C12—H12	119.9
O1—C1—C2	115.59 (13)	C13—C12—H12	119.9
C6—C1—C2	119.30 (13)	C14—C13—C12	121.58 (14)
C3—C2—C1	120.63 (13)	C14—C13—H13	119.2
C3—C2—H2A	119.7	C12—C13—H13	119.2
C1—C2—H2A	119.7	C13—C14—C15	120.31 (14)
C2—C3—C4	121.10 (12)	C13—C14—H14	119.8
C2—C3—H3	119.5	C15—C14—H14	119.8
C4—C3—H3	119.5	C16—C15—C20	118.76 (13)
C5—C4—C3	117.41 (12)	C16—C15—C14	122.73 (14)
C5—C4—C8	119.73 (12)	C20—C15—C14	118.49 (14)
C3—C4—C8	122.85 (12)	C17—C16—C15	120.21 (14)
C4—C5—C6	122.15 (12)	C17—C16—H16	119.9
C4—C5—H5	118.9	C15—C16—H16	119.9
C6—C5—H5	118.9	C16—C17—C18	121.29 (15)
C1—C6—C5	119.41 (13)	C16—C17—H17	119.4
C1—C6—H6	120.3	C18—C17—H17	119.4
C5—C6—H6	120.3	C19—C18—C17	120.33 (14)
O1—C7—H7A	109.5	C19—C18—H18	119.8
O1—C7—H7B	109.5	C17—C18—H18	119.8
H7A—C7—H7B	109.5	C18—C19—N2	123.57 (13)
O1—C7—H7C	109.5	C18—C19—C20	119.56 (13)
H7A—C7—H7C	109.5	N2—C19—C20	116.74 (12)
H7B—C7—H7C	109.5	C15—C20—C11	120.15 (12)
C9—C8—C4	127.69 (13)	C15—C20—C19	119.81 (13)
C9—C8—H8	116.2	C11—C20—C19	119.98 (12)
C4—C8—H8	116.2	C22—C21—H21A	109.5
C8—C9—C10	123.58 (14)	C22—C21—H21B	109.5
C8—C9—H9	118.2	H21A—C21—H21B	109.5
C10—C9—H9	118.2	C22—C21—H21C	109.5
N2—C10—N1	106.95 (11)	H21A—C21—H21C	109.5
N2—C10—C9	110.23 (12)	H21B—C21—H21C	109.5
N1—C10—C9	110.56 (12)	N3—C22—C21	179.1 (2)

C7—O1—C1—C6	10.1 (3)	C11—C12—C13—C14	0.2 (2)
C7—O1—C1—C2	−170.59 (16)	C12—C13—C14—C15	−0.2 (2)
O1—C1—C2—C3	−179.98 (14)	C13—C14—C15—C16	−178.84 (14)
C6—C1—C2—C3	−0.7 (2)	C13—C14—C15—C20	−0.5 (2)
C1—C2—C3—C4	−0.1 (2)	C20—C15—C16—C17	−1.8 (2)
C2—C3—C4—C5	0.5 (2)	C14—C15—C16—C17	176.61 (14)
C2—C3—C4—C8	−178.77 (13)	C15—C16—C17—C18	0.0 (2)
C3—C4—C5—C6	−0.2 (2)	C16—C17—C18—C19	1.0 (2)
C8—C4—C5—C6	179.08 (13)	C17—C18—C19—N2	−176.13 (13)
O1—C1—C6—C5	−179.82 (14)	C17—C18—C19—C20	−0.3 (2)
C2—C1—C6—C5	0.9 (2)	C10—N2—C19—C18	−152.05 (14)
C4—C5—C6—C1	−0.5 (2)	C10—N2—C19—C20	31.99 (17)
C5—C4—C8—C9	173.37 (16)	C16—C15—C20—C11	179.55 (12)

C3—C4—C8—C9	−7.4 (2)	C14—C15—C20—C11	1.10 (19)
C4—C8—C9—C10	177.61 (13)	C16—C15—C20—C19	2.48 (18)
C19—N2—C10—N1	−54.88 (16)	C14—C15—C20—C19	−175.97 (12)
C19—N2—C10—C9	−175.12 (11)	C12—C11—C20—C15	−1.08 (19)
C11—N1—C10—N2	51.33 (16)	N1—C11—C20—C15	−177.36 (11)
C11—N1—C10—C9	171.35 (12)	C12—C11—C20—C19	175.98 (12)
C8—C9—C10—N2	−116.90 (17)	N1—C11—C20—C19	−0.30 (19)
C8—C9—C10—N1	125.05 (16)	C18—C19—C20—C15	−1.48 (19)
C10—N1—C11—C12	157.87 (14)	N2—C19—C20—C15	174.65 (11)
C10—N1—C11—C20	−25.97 (18)	C18—C19—C20—C11	−178.55 (12)
N1—C11—C12—C13	176.53 (13)	N2—C19—C20—C11	−2.42 (18)
C20—C11—C12—C13	0.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.88 (2)	2.49 (2)	3.305 (3)	154.1 (15)
N2—H2···N3^{ii}	0.84 (2)	2.44 (2)	3.234 (2)	156.8 (16)

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