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5,5a-Diallyl-5,5a,13,14-tetrahydro-12*H*-di-1,3-benzimidazolo[1,2-*a*;1′,2′-*c*][1,4]diazepine

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The title compound, $C_{23}H_{24}N_4$, was synthesized from 3,3′-diallyl-1,1′-propylenedi(benzimidazole) dibromide and NaH in tetrahydrofuran solution. In the molecule, the diazepine ring exhibits a boat conformation.

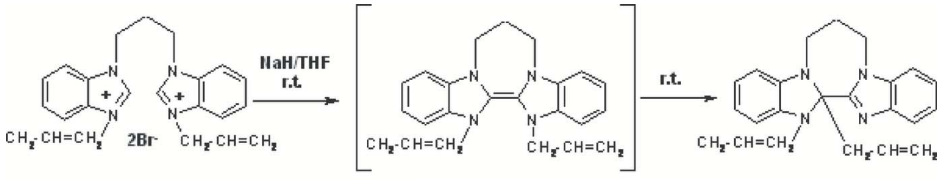
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.089
Data-to-parameter ratio = 10.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Electron-rich olefins have attracted considerable attention in both the organic and inorganic preparative literature as a result of their unique properties as reagents and reaction intermediates (Böhm & Herrmann, 2000). They have been used as powerful reducing agents (Lappert, 1988), sources of carbene transition metal complexes (Küçükbay *et al.*, 1996) and catalysts for acyloin type C—C coupling reactions (Çetinkaya & Küçükbay, 1995). They have an extensive chemistry and, in particular, electron-rich olefins that contain an imidazolidine or benzothiazolidine group have long been known, although there are few studies of electron-rich olefins containing a benzimidazolidine group. Isolation of allyl-, crotyl- or benzyl-substituted electron-rich olefins tends to be difficult because the synthesized olefins spontaneously transform to their [1,3]-sigmatropic rearrangement products. As was previously reported (Baldwin & Walker, 1974; Baldwin *et al.*, 1977; Çetinkaya *et al.*, 1998), we also obtained a [1,3]-sigmatropic rearrangement product, namely 2′,3′-diallyl-2′,3′*H*-dibenzimidazolo[a,c]perhydro-1,4-diazepine, (2), instead of the corresponding electron-rich olefin, (1), from a reaction of 3,3′-bis(allyl)-1,1′-propylendi(benzimidazole) dibromide and NaH in THF solution. The crystal structure of (2) is presented here.



(1) (2)

The molecular structure of (2) is shown in Fig. 1. The geometric parameters in (2) are within the normal ranges (Allen *et al.*, 1987) and agree with those in similar structures reported in the literature (Mague & Eduok, 2000; Akkurt *et al.*, 2006*a*,*b*). The diazepine ring exhibits a boat conformation. The displacements of atoms N3, C17 and C8 from the C1/N2/C9/C10 mean plane are 0.398 (1), 0.214 (2) and 0.562 (2) Å, respectively. The benzimidazole ring systems in (2) are essentially planar and the dihedral angle between them is 75.56 (5)°.

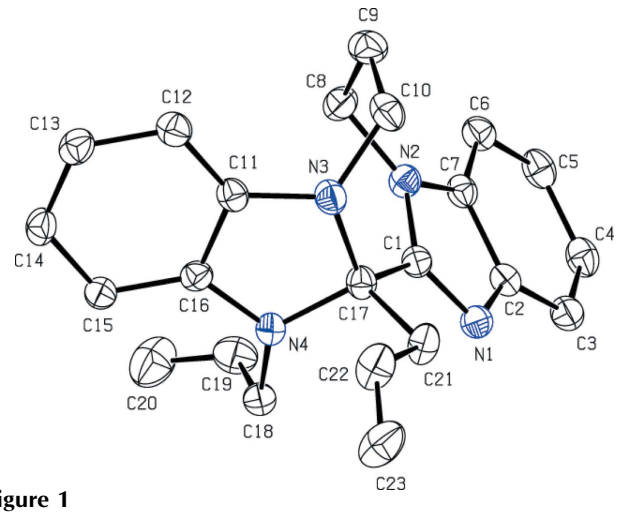


Figure 1
Molecular structure of (2), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. For clarity, H atoms have been omitted.

The molecular conformation of (2) is stabilized by an intramolecular C—H···N hydrogen-bonding interaction (Table 2).

Experimental

A mixture of 3,3′-bis(allyl)-1,1′-propylendi(benzimidazole) dibromide (5.0 g, 9.7 mmol) and NaH (0.5 g, 21 mmol) in THF (50 ml) was stirred for 10 h at room temperature. Volatiles were eliminated *in vacuo*, toluene (20 ml) was added and the suspension was filtered. The resulting bright-yellow filtrate was concentrated to *ca* 10 ml and *n*-hexane (10 ml) was added. Upon cooling, colourless crystals of (2) (2 g, 53%) were obtained (m.p. 408–409 K). ^{1}H NMR ($CDCl_3$): δ 1.6–1.8 (*m*, –CH_2-bridge, 2H), 3.0–3.2 (*d*, –CH_2–, 2H), 3.7–4.0 (*m*, –N—CH_2-bridge, 4H), 4.2 (*d*, N—CH_2–, 2H), 4.8–5.0 (*q*, ═CH_2, 2H), 5.1–5.3 (*q*, ═CH_2, 2H), 5.4–5.6 (*m*, –CH═, 1H), 5.9–6.1 (*m*, –CH═, 1H), 6.2–7.8 (*m*, Ar—H, 8H). Analysis calculated for $C_{23}H_{24}N_4$: C 77.53, H 6.74, N 15.73%; found: C 76.62, H 6.98, N 16.40%.

Crystal data

$C_{23}H_{24}N_4$
M_r = 356.46
Orthorhombic, $Pca2_1$
a = 25.1810 (13) Å
b = 8.304 (5) Å
c = 8.878 (8) Å
V = 1856 (2) Å^3
Z = 4
D_x = 1.276 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 150 (2) K
Irregular, colourless
0.44 × 0.43 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.967, T_{max} = 0.970
18216 measured reflections
2499 independent reflections
2334 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.02
2499 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.4382P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22···N3	0.93	2.56	2.912 (4)	103

H atoms were placed in geometrically idealized positions and constrained to ride on their parents atoms, with C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *APEXII* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

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5,5a-Diallyl-5,5a,13,14-tetrahydro-12*H*-di-1,3-benzimidazolo[1,2-*a*;1′,2′-*c*][1,4]diazepine

Sema Öztürk Yıldırım, Mehmet Akkurt, Ülkü Yılmaz, Hasan Küçükbay and Vickie McKee

5,5a-Diallyl-5,5a,13,14-tetrahydro-12*H*-di-1,3- benzimidazolo[1,2 - a;1′,2′-c][1,4]diazepine

Crystal data

$C_{23}H_{24}N_4$
M_r = 356.46
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 25.1810 (13) Å
b = 8.304 (5) Å
c = 8.878 (8) Å
V = 1856 (2) Å^3
Z = 4
F(000) = 760

^{1}H NMR ($CDCl_3$): δ 1.6–1.8 (m, –CH_2-bridge, 2H), 3.0–3.2 (d, –CH_2–, 2H), 3.7–4.0 (m, –N—CH_2-bridge, 4H), 4.2 (d, N—CH_2–, 2H), 4.8–5.0 (q, =CH_2, 2H), 5.1–5.3 (q, =CH_2, 2H), 5.4- 5.6 (m, –CH=, 1H), 5.9–6.1 (m, –CH=, 1H), 6.2–7.8 (m, Ar—H, 8H). ^{13}C-NMR ($CDCl_3$): δ 23.71, 39.89, 42.56, 45.29, 87.21, 103.37, 105.54, 109.09, 116.41, 117.31, 118.80, 120.43, 122.13, 123.37, 125.35, 128.27, 129.08, 133.73, 135.14, 138.25, 140.72, 142.13, 152.19.
D_x = 1.276 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 7669 reflections
θ = 2.5–28.4°
μ = 0.08 mm^{-1}
T = 150 K
Irregular, colourless
0.44 × 0.43 × 0.39 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
T_{min} = 0.967, T_{max} = 0.970

18216 measured reflections
2499 independent reflections
2334 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.6°, θ_{min} = 2.5°
h = −33→33
k = −11→11
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.02
2499 reflections
244 parameters
1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.4382P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.10280 (6)	0.84455 (18)	0.66213 (18)	0.0223 (4)
N2	0.05323 (6)	0.71239 (18)	0.83372 (18)	0.0216 (4)
N3	0.14408 (6)	0.70666 (18)	1.04193 (18)	0.0239 (4)
N4	0.17718 (6)	0.57597 (18)	0.83467 (18)	0.0233 (4)
C1	0.10270 (7)	0.7667 (2)	0.7914 (2)	0.0205 (4)
C2	0.05028 (7)	0.8395 (2)	0.6135 (2)	0.0212 (4)
C3	0.02758 (7)	0.8997 (2)	0.4815 (2)	0.0258 (5)
C4	−0.02634 (7)	0.8751 (2)	0.4592 (2)	0.0265 (5)
C5	−0.05748 (7)	0.7954 (2)	0.5675 (2)	0.0260 (5)
C6	−0.03590 (7)	0.7374 (2)	0.7007 (2)	0.0243 (5)
C7	0.01857 (7)	0.7589 (2)	0.7205 (2)	0.0210 (4)
C8	0.04028 (8)	0.6157 (2)	0.9661 (2)	0.0261 (5)
C9	0.04758 (8)	0.7082 (3)	1.1131 (2)	0.0297 (5)
C10	0.10185 (8)	0.7928 (2)	1.1199 (2)	0.0281 (5)
C11	0.16069 (7)	0.5534 (2)	1.0851 (2)	0.0216 (5)
C12	0.16005 (7)	0.4803 (2)	1.2248 (2)	0.0252 (5)
C13	0.18088 (7)	0.3236 (2)	1.2358 (2)	0.0267 (5)
C14	0.20109 (7)	0.2459 (2)	1.1108 (2)	0.0254 (5)
C15	0.20119 (7)	0.3195 (2)	0.9678 (2)	0.0231 (5)
C16	0.18087 (6)	0.4740 (2)	0.9574 (2)	0.0205 (4)
C17	0.15327 (7)	0.7315 (2)	0.8795 (2)	0.0212 (4)
C18	0.17773 (8)	0.5184 (2)	0.6795 (2)	0.0268 (5)
C19	0.12867 (9)	0.4219 (3)	0.6408 (2)	0.0349 (6)
C20	0.12834 (13)	0.2661 (3)	0.6183 (3)	0.0482 (8)
C21	0.19213 (7)	0.8725 (2)	0.8541 (2)	0.0276 (5)
C22	0.24318 (8)	0.8507 (2)	0.9400 (3)	0.0359 (6)
C23	0.28965 (8)	0.8917 (3)	0.8956 (3)	0.0441 (7)
H3	0.04800	0.95460	0.41080	0.0310*
H4	−0.04220	0.91190	0.37110	0.0320*
H5	−0.09350	0.78110	0.54930	0.0310*
H6	−0.05670	0.68680	0.77320	0.0290*
H8A	0.06280	0.52090	0.96750	0.0310*

H8B	0.00370	0.57980	0.95870	0.0310*
H9A	0.04450	0.63420	1.19720	0.0360*
H9B	0.01960	0.78790	1.12250	0.0360*
H10A	0.09840	0.89930	1.07610	0.0340*
H10B	0.11190	0.80610	1.22470	0.0340*
H12	0.14630	0.53290	1.30870	0.0300*
H13	0.18110	0.27160	1.32860	0.0320*
H14	0.21490	0.14270	1.12120	0.0300*
H15	0.21440	0.26620	0.88360	0.0280*
H18A	0.20890	0.45190	0.66410	0.0320*
H18B	0.18020	0.61000	0.61200	0.0320*
H19	0.09660	0.47690	0.63240	0.0420*
H20A	0.15970	0.20760	0.62590	0.0580*
H20B	0.09670	0.21400	0.59490	0.0580*
H21A	0.20000	0.88120	0.74750	0.0330*
H21B	0.17540	0.97210	0.88560	0.0330*
H22	0.24070	0.80270	1.03450	0.0430*
H23A	0.29410	0.94010	0.80200	0.0530*
H23B	0.31890	0.87320	0.95700	0.0530*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0204 (7)	0.0239 (7)	0.0227 (7)	0.0016 (5)	−0.0006 (6)	0.0021 (6)
N2	0.0200 (7)	0.0226 (7)	0.0222 (7)	0.0004 (5)	−0.0019 (6)	0.0014 (6)
N3	0.0264 (8)	0.0235 (7)	0.0219 (7)	0.0049 (6)	−0.0012 (6)	−0.0004 (6)
N4	0.0258 (7)	0.0206 (7)	0.0235 (7)	0.0037 (6)	0.0024 (6)	0.0028 (6)
C1	0.0215 (8)	0.0194 (7)	0.0205 (8)	0.0015 (6)	−0.0003 (6)	−0.0002 (6)
C2	0.0197 (8)	0.0206 (7)	0.0233 (8)	0.0025 (6)	−0.0001 (6)	−0.0023 (6)
C3	0.0285 (9)	0.0263 (8)	0.0225 (9)	0.0053 (7)	0.0004 (7)	0.0003 (7)
C4	0.0288 (9)	0.0254 (8)	0.0253 (9)	0.0060 (6)	−0.0059 (8)	−0.0025 (8)
C5	0.0211 (8)	0.0239 (8)	0.0330 (10)	0.0027 (7)	−0.0052 (7)	−0.0053 (7)
C6	0.0225 (8)	0.0227 (8)	0.0277 (9)	0.0002 (6)	0.0007 (7)	−0.0028 (7)
C7	0.0236 (8)	0.0195 (7)	0.0200 (8)	0.0025 (6)	−0.0012 (6)	−0.0026 (6)
C8	0.0263 (8)	0.0267 (8)	0.0254 (9)	−0.0010 (7)	0.0011 (7)	0.0054 (8)
C9	0.0295 (9)	0.0381 (10)	0.0215 (8)	0.0062 (8)	0.0020 (7)	0.0022 (8)
C10	0.0313 (9)	0.0285 (9)	0.0246 (9)	0.0086 (7)	−0.0027 (7)	−0.0048 (8)
C11	0.0178 (7)	0.0223 (8)	0.0246 (9)	0.0012 (6)	−0.0023 (6)	−0.0005 (7)
C12	0.0230 (8)	0.0296 (9)	0.0231 (8)	0.0032 (7)	−0.0021 (7)	−0.0002 (7)
C13	0.0252 (8)	0.0304 (9)	0.0245 (9)	0.0009 (7)	−0.0033 (7)	0.0066 (8)
C14	0.0232 (8)	0.0227 (7)	0.0303 (9)	0.0024 (6)	−0.0032 (7)	0.0059 (7)
C15	0.0200 (7)	0.0229 (8)	0.0264 (9)	0.0011 (6)	0.0016 (7)	0.0022 (7)
C16	0.0170 (7)	0.0237 (8)	0.0207 (7)	−0.0005 (6)	−0.0004 (6)	0.0025 (7)
C17	0.0197 (7)	0.0206 (7)	0.0233 (8)	0.0014 (6)	−0.0024 (6)	0.0008 (6)
C18	0.0330 (9)	0.0255 (8)	0.0220 (8)	0.0056 (7)	0.0048 (7)	0.0023 (7)
C19	0.0382 (10)	0.0408 (11)	0.0257 (9)	0.0008 (9)	0.0026 (8)	−0.0058 (8)
C20	0.0705 (17)	0.0399 (12)	0.0343 (11)	−0.0093 (11)	−0.0027 (12)	0.0001 (10)
C21	0.0237 (8)	0.0227 (8)	0.0364 (11)	−0.0025 (6)	−0.0064 (8)	0.0070 (8)

C22	0.0309 (10)	0.0346 (9)	0.0423 (12)	−0.0052 (8)	−0.0095 (9)	0.0080 (9)
C23	0.0273 (10)	0.0567 (14)	0.0482 (13)	−0.0046 (9)	−0.0082 (10)	0.0249 (12)

Geometric parameters (Å, º)

N1—C1	1.317 (3)	C19—C20	1.309 (4)
N1—C2	1.392 (3)	C21—C22	1.506 (3)
N2—C1	1.377 (3)	C22—C23	1.281 (3)
N2—C7	1.386 (3)	C3—H3	0.9300
N2—C8	1.460 (3)	C4—H4	0.9300
N3—C10	1.457 (3)	C5—H5	0.9300
N3—C11	1.393 (3)	C6—H6	0.9300
N3—C17	1.475 (3)	C8—H8A	0.9700
N4—C16	1.383 (3)	C8—H8B	0.9700
N4—C17	1.480 (3)	C9—H9A	0.9700
N4—C18	1.458 (3)	C9—H9B	0.9700
C1—C17	1.523 (3)	C10—H10A	0.9700
C2—C3	1.396 (3)	C10—H10B	0.9700
C2—C7	1.410 (3)	C12—H12	0.9300
C3—C4	1.387 (3)	C13—H13	0.9300
C4—C5	1.406 (3)	C14—H14	0.9300
C5—C6	1.388 (3)	C15—H15	0.9300
C6—C7	1.394 (3)	C18—H18A	0.9700
C8—C9	1.526 (3)	C18—H18B	0.9700
C9—C10	1.538 (3)	C19—H19	0.9300
C11—C12	1.381 (3)	C20—H20A	0.9300
C11—C16	1.407 (3)	C20—H20B	0.9300
C12—C13	1.406 (3)	C21—H21A	0.9700
C13—C14	1.381 (3)	C21—H21B	0.9700
C14—C15	1.409 (3)	C22—H22	0.9300
C15—C16	1.384 (3)	C23—H23A	0.9300
C17—C21	1.543 (3)	C23—H23B	0.9300
C18—C19	1.512 (3)		
N1···N4	3.291 (4)	C21···H18B	3.0800
N1···C18	3.304 (4)	C21···H10A	3.0800
N2···N3	2.942 (3)	C22···H14^{x}	3.0000
N3···N2	2.942 (3)	C22···H21A^{vi}	3.0900
N3···N4	2.293 (3)	C23···H10B^{i}	2.9900
N4···N3	2.293 (3)	H4···C2^{v}	3.0900
N4···N1	3.291 (4)	H5···C13^{ii}	2.8900
N1···H23B^{i}	2.6900	H5···C14^{ii}	2.7700
N1···H18B	2.7900	H5···C15^{ii}	2.9300
N1···H21B	2.9000	H6···C8	3.0400
N1···H21A	2.5800	H6···H8B	2.4100
N2···H19	2.8700	H6···C12^{ii}	2.9800
N2···H10A	2.8900	H8A···N3	2.6500
N3···H22	2.5600	H8A···C11	2.6900

N3···H8A	2.6500	H8A···C16	3.0000
C1···C19	3.227 (4)	H8A···C17	2.9800
C5···C13^{ii}	3.587 (4)	H8A···C5^{iii}	2.7800
C5···C8^{ii}	3.557 (4)	H8A···C6^{iii}	3.0600
C6···C8^{ii}	3.598 (4)	H8B···C6	2.8200
C8···C6^{iii}	3.598 (4)	H8B···H6	2.4100
C8···C11	3.252 (4)	H9A···C6^{iii}	3.0900
C8···C5^{iii}	3.557 (4)	H10A···N2	2.8900
C9···C12	3.548 (4)	H10A···C1	2.7600
C11···C8	3.252 (4)	H10A···C21	3.0800
C11···C22	3.474 (4)	H10A···C4^{iv}	2.8100
C12···C9	3.548 (4)	H10A···C5^{iv}	2.7400
C13···C5^{iii}	3.587 (4)	H10B···C12	2.9600
C15···C19	3.534 (4)	H10B···H12	2.5400
C16···C22	3.503 (4)	H10B···C23^{vi}	2.9900
C18···N1	3.304 (4)	H12···C10	2.9500
C19···C15	3.534 (4)	H12···H10B	2.5400
C19···C1	3.227 (4)	H13···C20^{xi}	2.9000
C22···C16	3.503 (4)	H14···C22^{viii}	3.0000
C22···C11	3.474 (4)	H14···H23A^{vii}	2.3400
C1···H18B	2.8400	H15···C18	2.9200
C1···H10A	2.7600	H15···H18A	2.4900
C1···H19	2.7900	H15···C13^{i}	2.9800
C2···H4^{iv}	3.0900	H18A···C15	2.9200
C4···H10A^{v}	2.8100	H18A···H15	2.4900
C5···H10A^{v}	2.7400	H18A···H20A	2.4000
C5···H8A^{ii}	2.7800	H18A···C13^{i}	3.0400
C6···H8A^{ii}	3.0600	H18A···C14^{i}	2.8800
C6···H9A^{ii}	3.0900	H18A···C15^{i}	3.0600
C6···H8B	2.8200	H18B···N1	2.7900
C8···H6	3.0400	H18B···C1	2.8400
C10···H12	2.9500	H18B···C21	3.0800
C11···H22	2.9200	H18B···H23B^{i}	2.5800
C11···H8A	2.6900	H19···N2	2.8700
C12···H6^{iii}	2.9800	H19···C1	2.7900
C12···H10B	2.9600	H20A···H18A	2.4000
C13···H15^{vi}	2.9800	H21A···N1	2.5800
C13···H18A^{vi}	3.0400	H21A···H23A	2.4700
C13···H5^{iii}	2.8900	H21A···C22^{i}	3.0900
C14···H23A^{vii}	3.0600	H21A···H22^{i}	2.5000
C14···H21B^{viii}	3.1000	H21B···N1	2.9000
C14···H18A^{vi}	2.8800	H21B···C14^{x}	3.1000
C14···H5^{iii}	2.7700	H21B···C15^{x}	3.0500
C15···H5^{iii}	2.9300	H22···N3	2.5600
C15···H21B^{viii}	3.0500	H22···C11	2.9200
C15···H18A	2.9200	H22···H21A^{vi}	2.5000
C15···H18A^{vi}	3.0600	H23A···H21A	2.4700
C16···H8A	3.0000	H23A···C14^{xii}	3.0600

C17···H8A	2.9800	H23A···H14^{xii}	2.3400
C18···H15	2.9200	H23B···N1^{vi}	2.6900
C20···H13^{ix}	2.9000	H23B···H18B^{vi}	2.5800
C1—N1—C2	104.69 (15)	C5—C4—H4	119.00
C1—N2—C7	106.29 (15)	C4—C5—H5	119.00
C1—N2—C8	126.99 (15)	C6—C5—H5	119.00
C7—N2—C8	126.60 (15)	C5—C6—H6	122.00
C10—N3—C11	122.48 (15)	C7—C6—H6	122.00
C10—N3—C17	120.70 (14)	N2—C8—H8A	109.00
C11—N3—C17	110.46 (14)	N2—C8—H8B	109.00
C16—N4—C17	110.48 (14)	C9—C8—H8A	109.00
C16—N4—C18	122.88 (14)	C9—C8—H8B	109.00
C17—N4—C18	122.97 (14)	H8A—C8—H8B	108.00
N1—C1—N2	113.59 (16)	C8—C9—H9A	109.00
N1—C1—C17	122.69 (16)	C8—C9—H9B	109.00
N2—C1—C17	123.61 (15)	C10—C9—H9A	109.00
N1—C2—C3	129.68 (16)	C10—C9—H9B	109.00
N1—C2—C7	110.09 (15)	H9A—C9—H9B	108.00
C3—C2—C7	120.23 (16)	N3—C10—H10A	109.00
C2—C3—C4	117.89 (16)	N3—C10—H10B	109.00
C3—C4—C5	121.17 (16)	C9—C10—H10A	109.00
C4—C5—C6	121.85 (16)	C9—C10—H10B	109.00
C5—C6—C7	116.63 (16)	H10A—C10—H10B	108.00
N2—C7—C2	105.32 (15)	C11—C12—H12	121.00
N2—C7—C6	132.47 (16)	C13—C12—H12	121.00
C2—C7—C6	122.21 (16)	C12—C13—H13	120.00
N2—C8—C9	112.63 (15)	C14—C13—H13	120.00
C8—C9—C10	111.76 (15)	C13—C14—H14	119.00
N3—C10—C9	113.94 (15)	C15—C14—H14	119.00
N3—C11—C12	130.17 (16)	C14—C15—H15	121.00
N3—C11—C16	108.36 (15)	C16—C15—H15	121.00
C12—C11—C16	121.47 (16)	N4—C18—H18A	109.00
C11—C12—C13	117.69 (16)	N4—C18—H18B	109.00
C12—C13—C14	120.93 (16)	C19—C18—H18A	109.00
C13—C14—C15	121.47 (16)	C19—C18—H18B	109.00
C14—C15—C16	117.48 (16)	H18A—C18—H18B	108.00
N4—C16—C11	108.89 (14)	C18—C19—H19	118.00
N4—C16—C15	130.15 (16)	C20—C19—H19	118.00
C11—C16—C15	120.95 (16)	C19—C20—H20A	120.00
N3—C17—N4	101.82 (13)	C19—C20—H20B	120.00
N3—C17—C1	113.44 (14)	H20A—C20—H20B	120.00
N3—C17—C21	110.40 (14)	C17—C21—H21A	109.00
N4—C17—C1	111.70 (14)	C17—C21—H21B	109.00
N4—C17—C21	111.42 (14)	C22—C21—H21A	109.00
C1—C17—C21	108.04 (14)	C22—C21—H21B	109.00
N4—C18—C19	112.37 (15)	H21A—C21—H21B	108.00
C18—C19—C20	124.3 (2)	C21—C22—H22	117.00

C17—C21—C22	112.11 (14)	C23—C22—H22	117.00
C21—C22—C23	126.3 (2)	C22—C23—H23A	120.00
C2—C3—H3	121.00	C22—C23—H23B	120.00
C4—C3—H3	121.00	H23A—C23—H23B	120.00
C3—C4—H4	119.00		

C2—N1—C1—N2	−1.1 (2)	C16—N4—C17—C21	118.01 (15)
C2—N1—C1—C17	175.15 (15)	N1—C1—C17—N4	−89.5 (2)
C1—N1—C2—C3	−178.33 (18)	N1—C1—C17—N3	156.09 (16)
C1—N1—C2—C7	1.39 (19)	N2—C1—C17—N4	86.4 (2)
C7—N2—C1—C17	−175.80 (15)	N1—C1—C17—C21	33.4 (2)
C7—N2—C1—N1	0.4 (2)	N2—C1—C17—N3	−28.0 (2)
C8—N2—C1—N1	176.68 (16)	N2—C1—C17—C21	−150.75 (16)
C1—N2—C8—C9	68.4 (2)	N1—C2—C7—C6	179.59 (16)
C7—N2—C8—C9	−116.2 (2)	C7—C2—C3—C4	−1.0 (2)
C8—N2—C1—C17	0.4 (3)	C3—C2—C7—N2	178.60 (15)
C1—N2—C7—C2	0.45 (18)	N1—C2—C3—C4	178.69 (17)
C8—N2—C7—C2	−175.81 (16)	C3—C2—C7—C6	−0.7 (3)
C1—N2—C7—C6	179.60 (18)	N1—C2—C7—N2	−1.15 (19)
C8—N2—C7—C6	3.3 (3)	C2—C3—C4—C5	1.5 (3)
C11—N3—C17—N4	−0.25 (18)	C3—C4—C5—C6	−0.2 (3)
C10—N3—C17—N4	−152.88 (15)	C4—C5—C6—C7	−1.4 (2)
C17—N3—C10—C9	86.92 (19)	C5—C6—C7—N2	−177.21 (18)
C10—N3—C11—C16	152.15 (16)	C5—C6—C7—C2	1.8 (3)
C11—N3—C10—C9	−62.4 (2)	N2—C8—C9—C10	−49.2 (2)
C11—N3—C17—C21	−118.67 (15)	C8—C9—C10—N3	−32.5 (2)
C10—N3—C17—C1	−32.7 (2)	N3—C11—C16—N4	0.1 (2)
C17—N3—C11—C16	0.1 (2)	N3—C11—C16—C15	178.87 (15)
C17—N3—C11—C12	179.32 (18)	C12—C11—C16—C15	−0.4 (3)
C10—N3—C17—C21	88.71 (18)	N3—C11—C12—C13	−178.45 (17)
C10—N3—C11—C12	−28.6 (3)	C16—C11—C12—C13	0.7 (3)
C11—N3—C17—C1	119.91 (16)	C12—C11—C16—N4	−179.19 (16)
C16—N4—C17—C1	−121.06 (15)	C11—C12—C13—C14	−0.2 (3)
C18—N4—C17—N3	159.23 (15)	C12—C13—C14—C15	−0.6 (3)
C17—N4—C16—C11	−0.28 (19)	C13—C14—C15—C16	0.9 (3)
C18—N4—C17—C1	37.9 (2)	C14—C15—C16—C11	−0.3 (2)
C16—N4—C17—N3	0.32 (17)	C14—C15—C16—N4	178.12 (17)
C16—N4—C18—C19	67.5 (2)	C1—C17—C21—C22	178.67 (16)
C17—N4—C18—C19	−88.8 (2)	N3—C17—C21—C22	54.1 (2)
C18—N4—C16—C15	22.2 (3)	N4—C17—C21—C22	−58.3 (2)
C17—N4—C16—C15	−178.89 (17)	N4—C18—C19—C20	−109.4 (2)
C18—N4—C17—C21	−83.1 (2)	C17—C21—C22—C23	143.9 (2)
C18—N4—C16—C11	−159.21 (16)		

Symmetry codes: (i) −*x*+1/2, *y*, *z*−1/2; (ii) −*x*, −*y*+1, *z*−1/2; (iii) −*x*, −*y*+1, *z*+1/2; (iv) −*x*, −*y*+2, *z*+1/2; (v) −*x*, −*y*+2, *z*−1/2; (vi) −*x*+1/2, *y*, *z*+1/2; (vii) −*x*+1/2, *y*−1, *z*+1/2; (viii) *x*, *y*−1, *z*; (ix) *x*, *y*, *z*−1; (x) *x*, *y*+1, *z*; (xi) *x*, *y*, *z*+1; (xii) −*x*+1/2, *y*+1, *z*−1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···N3	0.93	2.56	2.912 (4)	103