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2,6-Bis[1-(2-methylphenylimino)ethyl]-pyridine

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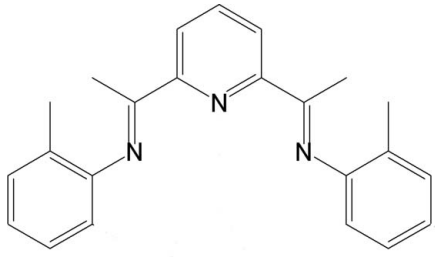
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.157; data-to-parameter ratio = 18.9.

The molecule of the title compound, $C_{23}H_{23}N_3$, which was synthesized by the condensation reaction between 2,6-diacetylpyridine and 2-dimethylaniline, adopts an E configuration about both C=N imine bonds. The dihedral angles formed by the benzene rings with the pyridine ring are 89.68 (5) and 53.62 (6)°.

Related literature

For the applications of pyridine-based ligands in sensor technologies and electro-luminescent devices, see: Tang & Vanslyke (1987); Wang (2001). For the crystal structures of related compounds, see: Mentes *et al.* (2001); Huang *et al.* (2006). For the synthesis, see: Fan *et al.* (2004).



Experimental

Crystal data

$C_{23}H_{23}N_3$
M_r = 341.44
Monoclinic, $P2_1/n$
a = 12.966 (3) Å
b = 11.304 (2) Å
c = 14.767 (3) Å
β = 115.62 (3)°
V = 1951.6 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 193 K
0.56 × 0.41 × 0.36 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.960, T_{max} = 0.972
18526 measured reflections
4435 independent reflections
2804 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.157
S = 1.06
4435 reflections
235 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2329).

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supporting information

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2,6-Bis[1-(2-methylphenylimino)ethyl]pyridine

Rui-Qing Fan, Xiao-Dong Ding, Guang-Peng Zhou and Yu-Lin Yang

S1. Comment

Luminescent coordination compounds based on pyridine-type ligands have attracted intensive attention due to their potential application in areas of sensor technologies and electro-luminescent devices (Tang & Vanslyke, 1987; Wang, 2001). In order to explore potential luminescent complexes of this type, we prepared a series of bis(iminoalkyl)pyridine ligands by the condensation reaction of 2,6-diacetylpyridine with the corresponding aniline in methanol (Fan *et al.*, 2004). It is still challenging to design and rationally synthesize ligands with unique structures and functions. In this regard, we report herein the synthesis and crystal structure of the title compound.

The molecule of the title compound (Fig. 1) possesses an approximate C_s symmetry about a plane bisecting the pyridine ring. The pyridine ring is coplanar with the two imino groups, which show typical C═N double bond character (1.2606 (18) and 1.2674 (19) Å for N1═C1 and N3═C7, respectively). These values are in good agreement with those observed in 2,6-bis[1-(phenylimino)ethyl]pyridine (1.266 (4) Å; Mentes *et al.*, 2001) and in 2,6-bis[1-(2,6-dimethylphenylimino)ethyl]pyridine (1.265 (2) and 1.271 (2) Å; Huang *et al.*, 2006). The dihedral angles between the C10–C15 and C17–C22 benzene rings and the pyridine ring are 89.68 (5) and 53.62 (6)°, respectively. The crystal packing (Fig. 2) is stabilized only by van der Waals interactions.

S2. Experimental

The title compound was synthesized according to the literatute method (Fan *et al.*, 2004). To a solution of 2,6-diacetylpyridine (1.1 g, 6.7 mmol) in absolute methanol (25 ml) was added 2-dimethylaniline (2.2 ml, 20.5 mmol). After the addition of several drops of formic acid, the reaction mixture was refluxed for 24 h and then allowed to cool down to room temperature. The crude product precipitated as yellow powder. Yellow block crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a methanol solution in 85% yield (1.96 g).

S3. Refinement

All H atoms were positioned geometrically with C—H = 0.93–0.96 Å, and allowed to ride on their parent atoms with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms.

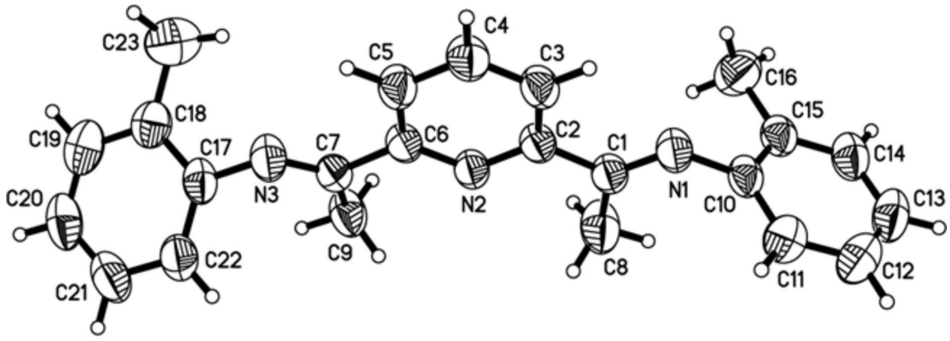

Figure 1

The molecular structire of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

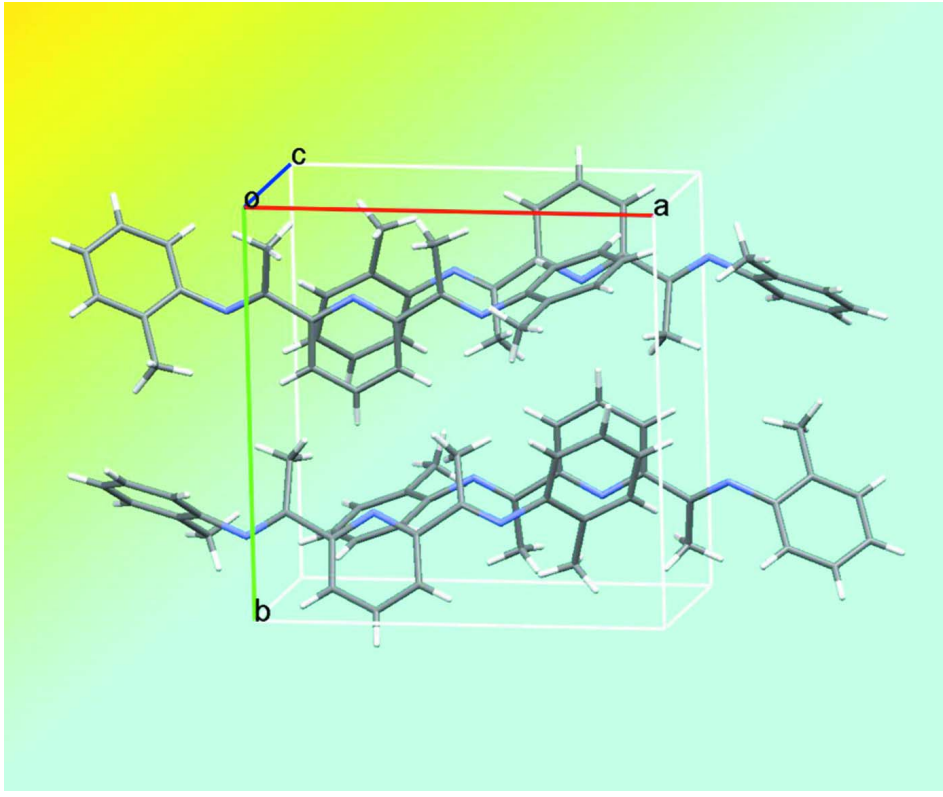

Figure 2

Packing diagram of the title compound viewed along the *c* axis.

2,6-bis[1-(2-methylphenylimino)ethyl]pyridine

Crystal data

$C_{23}H_{23}N_3$
$M_r = 341.44$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.966$ (3) Å
$b = 11.304$ (2) Å
$c = 14.767$ (3) Å
$\beta = 115.62$ (3)°
$V = 1951.6$ (8) Å^3
$Z = 4$
$F(000) = 728$
$D_x = 1.162$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 18526 reflections

θ = 3.1–27.5°
μ = 0.07 mm^{-1}
T = 193 K

Block, yellow
0.56 × 0.41 × 0.36 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.960, T_{max} = 0.972

18526 measured reflections
4435 independent reflections
2804 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −16→16
k = −14→13
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.157
S = 1.06
4435 reflections
235 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
$w = 1/[\sigma^2(F_o^2) + (0.0848P)^2 + 0.041P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N2	0.27847 (10)	0.74889 (10)	0.16139 (9)	0.0458 (3)
N1	−0.01920 (11)	0.79913 (11)	0.03667 (10)	0.0527 (3)
C6	0.37804 (12)	0.80381 (12)	0.18433 (11)	0.0448 (4)
N3	0.57761 (11)	0.77842 (11)	0.25205 (11)	0.0565 (4)
C2	0.18145 (13)	0.80916 (12)	0.10943 (11)	0.0457 (4)
C1	0.07290 (13)	0.74430 (12)	0.08606 (11)	0.0476 (4)
C7	0.48407 (13)	0.73369 (12)	0.24317 (11)	0.0466 (4)
C3	0.18154 (13)	0.92576 (13)	0.08007 (12)	0.0524 (4)
H3B	0.1130	0.9656	0.0444	0.063*
C5	0.38449 (14)	0.92051 (13)	0.15758 (13)	0.0543 (4)
H5A	0.4550	0.9567	0.1751	0.065*
C10	−0.12858 (13)	0.74706 (12)	0.00704 (12)	0.0489 (4)
C17	0.68397 (13)	0.72253 (14)	0.30780 (13)	0.0523 (4)

C15	−0.18677 (13)	0.75912 (12)	0.06699 (12)	0.0522 (4)
C4	0.28403 (14)	0.98142 (13)	0.10444 (13)	0.0586 (4)
H4A	0.2858	1.0595	0.0853	0.070*
C14	−0.29933 (15)	0.72146 (15)	0.02802 (15)	0.0620 (5)
H14A	−0.3395	0.7298	0.0668	0.074*
C22	0.71360 (14)	0.61751 (15)	0.27656 (14)	0.0633 (5)
H22A	0.6602	0.5788	0.2202	0.076*
C9	0.47073 (15)	0.62046 (14)	0.28932 (15)	0.0633 (5)
H9A	0.5443	0.5840	0.3248	0.095*
H9B	0.4213	0.5681	0.2375	0.095*
H9C	0.4381	0.6366	0.3353	0.095*
C18	0.76376 (15)	0.78082 (15)	0.39198 (13)	0.0579 (4)
C8	0.08130 (16)	0.61968 (15)	0.12366 (17)	0.0764 (6)
H8A	0.0058	0.5882	0.1037	0.115*
H8B	0.1217	0.6191	0.1956	0.115*
H8C	0.1217	0.5721	0.0958	0.115*
C13	−0.35352 (16)	0.67238 (16)	−0.06562 (16)	0.0697 (5)
H13A	−0.4295	0.6487	−0.0902	0.084*
C11	−0.18236 (16)	0.69527 (15)	−0.08650 (14)	0.0634 (5)
H11A	−0.1426	0.6850	−0.1254	0.076*
C20	0.90039 (16)	0.62620 (19)	0.41061 (16)	0.0742 (6)
H20A	0.9733	0.5946	0.4454	0.089*
C12	−0.29465 (17)	0.65852 (17)	−0.12285 (15)	0.0721 (5)
H12A	−0.3304	0.6243	−0.1862	0.087*
C19	0.87169 (16)	0.72988 (18)	0.44214 (14)	0.0708 (5)
H19A	0.9259	0.7674	0.4988	0.085*
C16	−0.12855 (18)	0.81252 (18)	0.17075 (15)	0.0768 (6)
H16A	−0.1809	0.8138	0.2009	0.115*
H16B	−0.1049	0.8918	0.1659	0.115*
H16C	−0.0629	0.7659	0.2114	0.115*
C21	0.82176 (17)	0.56955 (18)	0.32815 (17)	0.0744 (5)
H21A	0.8407	0.4990	0.3066	0.089*
C23	0.7334 (2)	0.89394 (18)	0.42839 (17)	0.0874 (6)
H23A	0.6561	0.9154	0.3847	0.131*
H23B	0.7842	0.9558	0.4283	0.131*
H23C	0.7407	0.8829	0.4953	0.131*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0389 (7)	0.0437 (7)	0.0531 (7)	0.0021 (5)	0.0183 (6)	0.0030 (5)
N1	0.0407 (7)	0.0476 (7)	0.0659 (9)	0.0017 (5)	0.0193 (6)	0.0079 (6)
C6	0.0412 (8)	0.0442 (8)	0.0508 (8)	0.0034 (6)	0.0217 (7)	0.0030 (6)
N3	0.0409 (8)	0.0550 (7)	0.0719 (9)	0.0030 (6)	0.0227 (7)	0.0109 (6)
C2	0.0413 (8)	0.0460 (8)	0.0493 (8)	0.0043 (6)	0.0191 (7)	0.0026 (6)
C1	0.0423 (8)	0.0458 (8)	0.0533 (9)	0.0019 (6)	0.0193 (7)	0.0031 (6)
C7	0.0421 (8)	0.0441 (7)	0.0537 (9)	0.0027 (6)	0.0208 (7)	−0.0005 (6)
C3	0.0439 (9)	0.0460 (8)	0.0631 (10)	0.0072 (6)	0.0191 (7)	0.0075 (7)

C5	0.0434 (9)	0.0472 (8)	0.0721 (11)	−0.0002 (6)	0.0247 (8)	0.0068 (7)
C10	0.0394 (8)	0.0399 (7)	0.0619 (9)	0.0040 (6)	0.0166 (7)	0.0108 (6)
C17	0.0384 (8)	0.0541 (9)	0.0654 (10)	0.0012 (7)	0.0234 (8)	0.0113 (7)
C15	0.0446 (9)	0.0435 (8)	0.0658 (10)	0.0024 (6)	0.0212 (8)	0.0074 (7)
C4	0.0521 (10)	0.0421 (8)	0.0790 (11)	0.0036 (7)	0.0260 (8)	0.0122 (7)
C14	0.0454 (9)	0.0622 (10)	0.0774 (12)	0.0018 (8)	0.0256 (9)	0.0112 (9)
C22	0.0455 (9)	0.0639 (10)	0.0785 (12)	0.0029 (8)	0.0248 (9)	0.0004 (8)
C9	0.0487 (10)	0.0548 (9)	0.0876 (13)	0.0091 (7)	0.0305 (9)	0.0182 (8)
C18	0.0542 (10)	0.0614 (9)	0.0583 (10)	−0.0042 (8)	0.0245 (8)	0.0091 (7)
C8	0.0511 (10)	0.0542 (10)	0.1118 (16)	0.0021 (8)	0.0239 (10)	0.0249 (10)
C13	0.0429 (9)	0.0704 (11)	0.0806 (13)	−0.0085 (8)	0.0125 (9)	0.0122 (10)
C11	0.0589 (11)	0.0667 (10)	0.0621 (11)	−0.0051 (8)	0.0238 (9)	0.0013 (8)
C20	0.0415 (10)	0.0890 (14)	0.0863 (14)	0.0123 (9)	0.0222 (10)	0.0223 (11)
C12	0.0647 (12)	0.0717 (11)	0.0630 (11)	−0.0148 (9)	0.0116 (10)	−0.0010 (9)
C19	0.0518 (11)	0.0886 (13)	0.0613 (11)	−0.0094 (9)	0.0143 (9)	0.0121 (9)
C16	0.0730 (13)	0.0819 (13)	0.0805 (14)	−0.0175 (10)	0.0380 (11)	−0.0189 (10)
C21	0.0567 (11)	0.0729 (11)	0.1024 (15)	0.0155 (9)	0.0428 (11)	0.0108 (11)
C23	0.1025 (18)	0.0732 (12)	0.0791 (14)	−0.0003 (12)	0.0323 (13)	−0.0034 (10)

Geometric parameters (Å, º)

N2—C6	1.3374 (18)	C22—H22A	0.9300
N2—C2	1.3420 (18)	C9—H9A	0.9600
N1—C1	1.2606 (18)	C9—H9B	0.9600
N1—C10	1.4187 (19)	C9—H9C	0.9600
C6—C5	1.390 (2)	C18—C19	1.394 (3)
C6—C7	1.497 (2)	C18—C23	1.504 (3)
N3—C7	1.2674 (19)	C8—H8A	0.9600
N3—C17	1.413 (2)	C8—H8B	0.9600
C2—C3	1.388 (2)	C8—H8C	0.9600
C2—C1	1.489 (2)	C13—C12	1.371 (3)
C1—C8	1.501 (2)	C13—H13A	0.9300
C7—C9	1.495 (2)	C11—C12	1.380 (3)
C3—C4	1.370 (2)	C11—H11A	0.9300
C3—H3B	0.9300	C20—C21	1.363 (3)
C5—C4	1.379 (2)	C20—C19	1.371 (3)
C5—H5A	0.9300	C20—H20A	0.9300
C10—C11	1.380 (2)	C12—H12A	0.9300
C10—C15	1.396 (2)	C19—H19A	0.9300
C17—C22	1.387 (2)	C16—H16A	0.9600
C17—C18	1.392 (2)	C16—H16B	0.9600
C15—C14	1.384 (2)	C16—H16C	0.9600
C15—C16	1.511 (3)	C21—H21A	0.9300
C4—H4A	0.9300	C23—H23A	0.9600
C14—C13	1.369 (3)	C23—H23B	0.9600
C14—H14A	0.9300	C23—H23C	0.9600
C22—C21	1.385 (2)		

C6—N2—C2	118.22 (12)	C7—C9—H9C	109.5
C1—N1—C10	123.03 (12)	H9A—C9—H9C	109.5
N2—C6—C5	122.59 (13)	H9B—C9—H9C	109.5
N2—C6—C7	116.43 (12)	C17—C18—C19	117.84 (17)
C5—C6—C7	120.96 (13)	C17—C18—C23	120.89 (17)
C7—N3—C17	122.08 (13)	C19—C18—C23	121.26 (18)
N2—C2—C3	122.25 (14)	C1—C8—H8A	109.5
N2—C2—C1	116.17 (12)	C1—C8—H8B	109.5
C3—C2—C1	121.58 (13)	H8A—C8—H8B	109.5
N1—C1—C2	117.13 (13)	C1—C8—H8C	109.5
N1—C1—C8	125.07 (14)	H8A—C8—H8C	109.5
C2—C1—C8	117.79 (13)	H8B—C8—H8C	109.5
N3—C7—C9	126.06 (14)	C14—C13—C12	119.32 (17)
N3—C7—C6	116.53 (13)	C14—C13—H13A	120.3
C9—C7—C6	117.37 (13)	C12—C13—H13A	120.3
C4—C3—C2	119.07 (14)	C10—C11—C12	120.57 (19)
C4—C3—H3B	120.5	C10—C11—H11A	119.7
C2—C3—H3B	120.5	C12—C11—H11A	119.7
C4—C5—C6	118.51 (14)	C21—C20—C19	119.81 (17)
C4—C5—H5A	120.7	C21—C20—H20A	120.1
C6—C5—H5A	120.7	C19—C20—H20A	120.1
C11—C10—C15	119.90 (15)	C13—C12—C11	120.05 (18)
C11—C10—N1	119.33 (16)	C13—C12—H12A	120.0
C15—C10—N1	120.41 (15)	C11—C12—H12A	120.0
C22—C17—C18	119.76 (15)	C20—C19—C18	122.03 (18)
C22—C17—N3	121.99 (15)	C20—C19—H19A	119.0
C18—C17—N3	118.04 (15)	C18—C19—H19A	119.0
C14—C15—C10	117.91 (16)	C15—C16—H16A	109.5
C14—C15—C16	121.28 (17)	C15—C16—H16B	109.5
C10—C15—C16	120.81 (15)	H16A—C16—H16B	109.5
C3—C4—C5	119.35 (14)	C15—C16—H16C	109.5
C3—C4—H4A	120.3	H16A—C16—H16C	109.5
C5—C4—H4A	120.3	H16B—C16—H16C	109.5
C13—C14—C15	122.21 (18)	C20—C21—C22	119.73 (18)
C13—C14—H14A	118.9	C20—C21—H21A	120.1
C15—C14—H14A	118.9	C22—C21—H21A	120.1
C21—C22—C17	120.83 (17)	C18—C23—H23A	109.5
C21—C22—H22A	119.6	C18—C23—H23B	109.5
C17—C22—H22A	119.6	H23A—C23—H23B	109.5
C7—C9—H9A	109.5	C18—C23—H23C	109.5
C7—C9—H9B	109.5	H23A—C23—H23C	109.5
H9A—C9—H9B	109.5	H23B—C23—H23C	109.5

C2—N2—C6—C5	0.8 (2)	C11—C10—C15—C14	−2.1 (2)
C2—N2—C6—C7	179.43 (13)	N1—C10—C15—C14	170.93 (13)
C6—N2—C2—C3	−0.6 (2)	C11—C10—C15—C16	178.34 (15)
C6—N2—C2—C1	−179.79 (13)	N1—C10—C15—C16	−8.6 (2)
C10—N1—C1—C2	178.32 (14)	C2—C3—C4—C5	−0.1 (3)

C10—N1—C1—C8	−2.3 (3)	C6—C5—C4—C3	0.3 (3)
N2—C2—C1—N1	−179.50 (14)	C10—C15—C14—C13	0.8 (2)
C3—C2—C1—N1	1.3 (2)	C16—C15—C14—C13	−179.72 (16)
N2—C2—C1—C8	1.0 (2)	C18—C17—C22—C21	0.1 (3)
C3—C2—C1—C8	−178.12 (16)	N3—C17—C22—C21	174.74 (16)
C17—N3—C7—C9	1.0 (3)	C22—C17—C18—C19	−0.2 (2)
C17—N3—C7—C6	178.40 (14)	N3—C17—C18—C19	−175.06 (15)
N2—C6—C7—N3	169.72 (14)	C22—C17—C18—C23	−179.14 (17)
C5—C6—C7—N3	−11.6 (2)	N3—C17—C18—C23	6.0 (2)
N2—C6—C7—C9	−12.7 (2)	C15—C14—C13—C12	0.7 (3)
C5—C6—C7—C9	165.96 (16)	C15—C10—C11—C12	2.1 (2)
N2—C2—C3—C4	0.3 (2)	N1—C10—C11—C12	−171.07 (15)
C1—C2—C3—C4	179.40 (15)	C14—C13—C12—C11	−0.8 (3)
N2—C6—C5—C4	−0.6 (2)	C10—C11—C12—C13	−0.5 (3)
C7—C6—C5—C4	−179.19 (15)	C21—C20—C19—C18	−0.4 (3)
C1—N1—C10—C11	−93.60 (19)	C17—C18—C19—C20	0.4 (3)
C1—N1—C10—C15	93.31 (19)	C23—C18—C19—C20	179.31 (19)
C7—N3—C17—C22	68.2 (2)	C19—C20—C21—C22	0.3 (3)
C7—N3—C17—C18	−117.05 (18)	C17—C22—C21—C20	−0.1 (3)