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2,3-Bis(4-bromophenyl)quinoxaline

Fang-Fang Jian,* Ke-Fei Wang, Rui-Rui Zhuang and Hai-Lian Xiao

New Materials and Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China
Correspondence e-mail: ffj2003@163169.net

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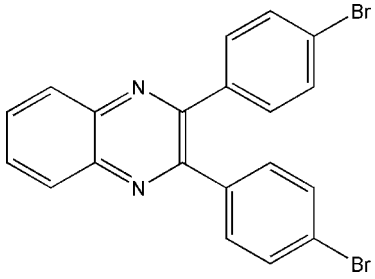
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.045; wR factor = 0.128; data-to-parameter ratio = 13.3.

The title compound, $C_{20}H_{12}Br_2N_2$, was prepared by the reaction of 1-(3-bromophenyl)-2-(4-bromophenyl)ethane-1,2-dione with *o*-phenylenediamine in refluxing ethanol. In the molecule, all bond lengths and angles are within normal ranges. The dihedral angle between the two benzene rings is 34.89 (1)°. The dihedral angles between the benzene rings and the quinoxaline system are 57.23 (1) and 36.75 (1)°. The crystal packing is stabilized by van der Waals forces.

Related literature

For related literature, see: Brock *et al.* (1999); Dailey *et al.* (2001); Guillon *et al.* (1998); Kim *et al.* (1993); Patel *et al.* (2000); Rong *et al.* (2006).



Experimental

Crystal data

$C_{20}H_{12}Br_2N_2$
M_r = 440.14
Triclinic, $P\overline{1}$
a = 6.0830 (12) Å
b = 12.018 (2) Å
c = 12.323 (3) Å
α = 105.47 (3)°
β = 91.89 (3)°
γ = 97.47 (3)°
V = 858.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.72 mm^{-1}
T = 293 (2) K
0.20 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.404, T_{max} = 0.492
3338 measured reflections
2888 independent reflections
1824 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.06
2888 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2324).

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supporting information

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2,3-Bis(4-bromophenyl)quinoxaline

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S1. Comment

Quinoxaline derivatives are an important class of nitrogen containing heterocycles and constitute useful intermediates in organic synthesis which have been reported for their applications in the fields of dyes (Brock *et al.*, 1999) and have also been used as building blocks for the synthesis of organic semiconductors (Dailey *et al.*, 2001). Tetrahydroquinoxaline derivatives are important from a therapeutic point of view since promising anti HIV agents (Patel *et al.*, 2000), glucogen receptor antagonists (Guillon *et al.*, 1998) and angiotens in receptor antagonists (Kim *et al.*, 1993) possess this ring system. The title compound (I) was synthesized as part of our study of these ligands. Here we report the crystal structure of (I).

The structure of (I) is represented in Fig. 1. The bond lengths and angles are usual for this type of compound (Rong *et al.*, 2006). The mean planes p1(C1 - C6) and p2 (N1,N2,C7 - C14) make a dihedral angle of 57.23 (1)°. The dihedral angles formed by phenyl ring(C8 –C13) and phenyl ring (C15 - C20) with p1 are 55.48 (1) and 64.80 (1)°, respectively. The dihedral angles between the benzene rings is 34.89 (1)°. The crystal packing (Fig. 2) is stabilized by van der Waals forces.

S2. Experimental

A mixture of 1-(3-bromophenyl)-2-(4-bromophenyl)ethane-1,2-dione (5.77 g, 0.02 mol) and *o*-phenylene diamine (2.16 g, 0.02 mol) was stirred in refluxing ethanol (30 ml) for 5 h to afford the title compound (3.25 g, yield 74%). Single crystals suitable for X-ray measurements were obtained by recrystallization from THF at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.93–0.96 Å, respectively, and with U_{iso}=1.2–1.5U_{eq} of the parent atoms.

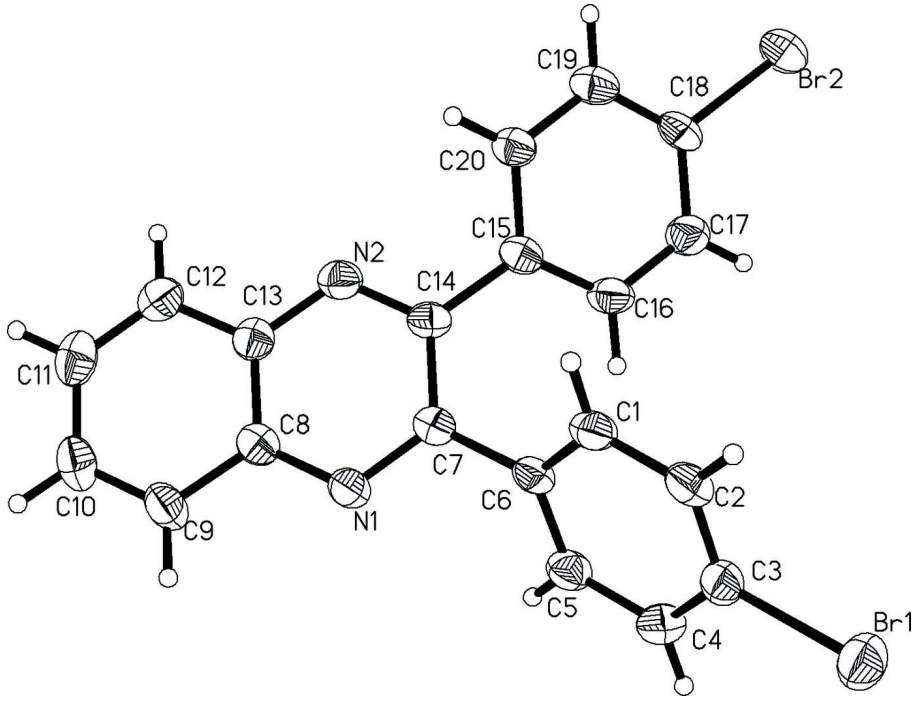

Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

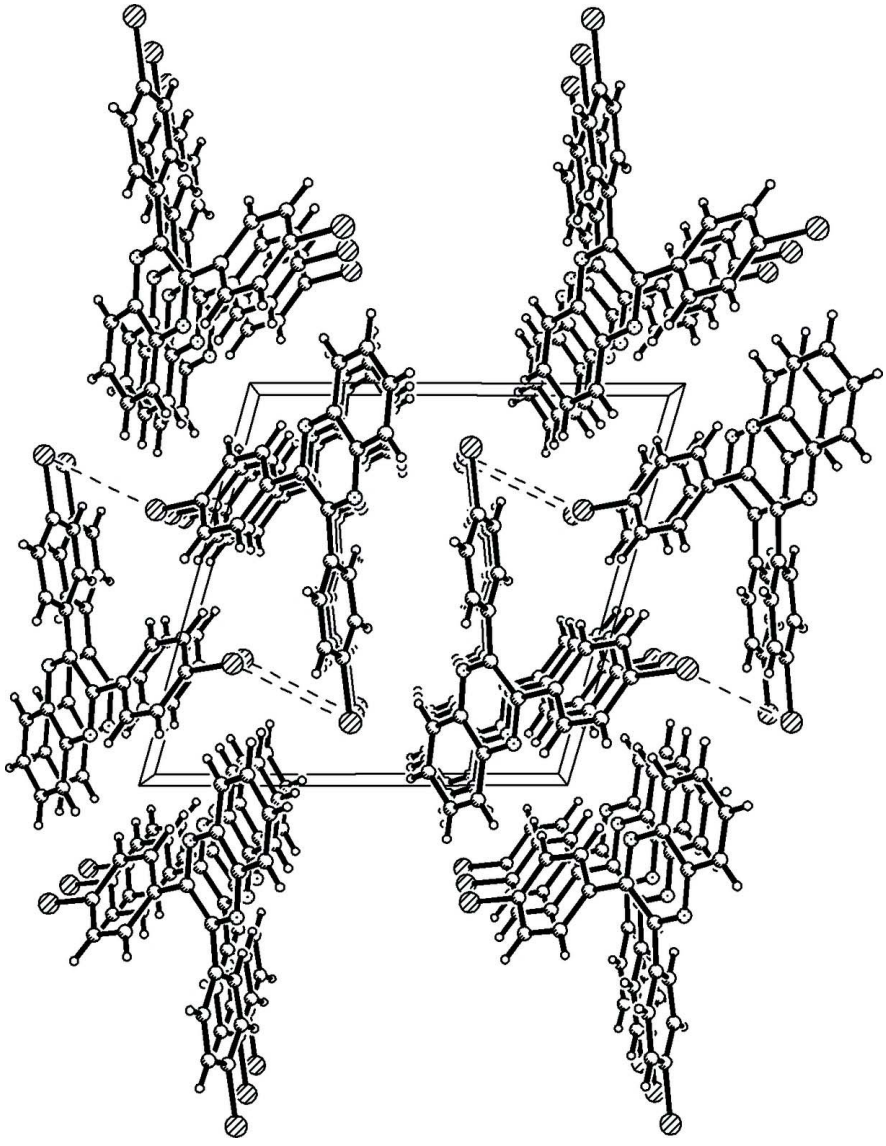

Figure 2

The crystal packing of (I), viewed down the *a* axis.

2,3-Bis(4-bromophenyl)quinoxaline

Crystal data

$C_{20}H_{12}Br_2N_2$
$M_r = 440.14$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.0830\ (12)$ Å
$b = 12.018\ (2)$ Å
$c = 12.323\ (3)$ Å
$\alpha = 105.47\ (3)°$
$\beta = 91.89\ (3)°$
$\gamma = 97.47\ (3)°$
$V = 858.7\ (3)$ Å^3
$Z = 2$
$F(000) = 432$
$D_x = 1.702$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4–14°$
$\mu = 4.72$ mm^{-1}
$T = 293$ K
Block, yellow
0.20 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.404, T_{max} = 0.492
3338 measured reflections
2888 independent reflections
1824 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°, θ_{min} = 1.7°
h = 0→7
k = −14→14
l = −14→14
3 standard reflections every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.06
2888 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.7939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 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}
Br1	0.47031 (12)	0.83130 (5)	0.45247 (6)	0.0741 (3)
Br2	−0.24223 (11)	0.30938 (6)	−0.14438 (6)	0.0753 (3)
N1	0.7857 (7)	0.2921 (4)	0.3229 (4)	0.0493 (11)
N2	0.4951 (8)	0.1176 (4)	0.1656 (4)	0.0542 (12)
C1	0.3718 (9)	0.4705 (5)	0.3533 (5)	0.0526 (14)
H1B	0.2564	0.4116	0.3532	0.063*
C2	0.3425 (9)	0.5854 (5)	0.3977 (5)	0.0529 (14)
H2B	0.2112	0.6041	0.4297	0.064*
C3	0.5130 (10)	0.6720 (5)	0.3936 (5)	0.0522 (14)
C4	0.7127 (10)	0.6469 (5)	0.3496 (5)	0.0589 (15)
H4A	0.8255	0.7065	0.3480	0.071*
C5	0.7412 (9)	0.5309 (5)	0.3078 (5)	0.0544 (14)
H5A	0.8755	0.5129	0.2789	0.065*
C6	0.5720 (9)	0.4407 (4)	0.3082 (4)	0.0461 (13)
C7	0.6106 (9)	0.3173 (4)	0.2700 (4)	0.0462 (13)

C8	0.8169 (9)	0.1783 (5)	0.2990 (5)	0.0493 (13)
C9	0.9999 (10)	0.1469 (6)	0.3534 (5)	0.0652 (17)
H9A	1.1007	0.2046	0.4025	0.078*
C10	1.0281 (12)	0.0335 (6)	0.3342 (6)	0.0709 (18)
H10A	1.1480	0.0130	0.3695	0.085*
C11	0.8729 (12)	−0.0534 (6)	0.2597 (6)	0.0735 (19)
H11A	0.8904	−0.1314	0.2479	0.088*
C12	0.7000 (11)	−0.0263 (5)	0.2052 (6)	0.0694 (18)
H12A	0.6031	−0.0852	0.1551	0.083*
C13	0.6657 (9)	0.0901 (5)	0.2238 (5)	0.0505 (13)
C14	0.4677 (8)	0.2285 (4)	0.1856 (5)	0.0472 (13)
C15	0.2918 (9)	0.2540 (5)	0.1121 (4)	0.0477 (13)
C16	0.3189 (10)	0.3520 (5)	0.0707 (5)	0.0584 (15)
H16A	0.4455	0.4071	0.0940	0.070*
C17	0.1619 (10)	0.3685 (5)	−0.0041 (5)	0.0611 (16)
H17A	0.1836	0.4330	−0.0326	0.073*
C18	−0.0301 (9)	0.2875 (5)	−0.0366 (5)	0.0545 (14)
C19	−0.0638 (10)	0.1897 (5)	0.0024 (5)	0.0576 (15)
H19A	−0.1927	0.1362	−0.0200	0.069*
C20	0.0981 (9)	0.1728 (5)	0.0755 (5)	0.0516 (14)
H20A	0.0783	0.1062	0.1010	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0889 (5)	0.0564 (4)	0.0746 (5)	0.0182 (3)	0.0012 (4)	0.0107 (3)
Br2	0.0645 (4)	0.0854 (5)	0.0725 (5)	0.0068 (3)	−0.0241 (3)	0.0212 (4)
N1	0.044 (3)	0.053 (3)	0.049 (3)	0.009 (2)	−0.002 (2)	0.009 (2)
N2	0.048 (3)	0.056 (3)	0.057 (3)	−0.001 (2)	0.002 (2)	0.016 (2)
C1	0.040 (3)	0.056 (3)	0.057 (4)	0.001 (3)	−0.004 (3)	0.011 (3)
C2	0.039 (3)	0.065 (4)	0.052 (3)	0.011 (3)	−0.002 (3)	0.010 (3)
C3	0.059 (4)	0.051 (3)	0.045 (3)	0.010 (3)	−0.006 (3)	0.012 (3)
C4	0.050 (4)	0.056 (3)	0.066 (4)	−0.004 (3)	−0.001 (3)	0.014 (3)
C5	0.039 (3)	0.061 (3)	0.061 (4)	0.007 (3)	0.002 (3)	0.013 (3)
C6	0.041 (3)	0.053 (3)	0.041 (3)	0.004 (2)	−0.008 (2)	0.010 (2)
C7	0.041 (3)	0.053 (3)	0.044 (3)	0.004 (2)	0.001 (2)	0.013 (2)
C8	0.044 (3)	0.058 (3)	0.047 (3)	0.009 (3)	0.007 (3)	0.014 (3)
C9	0.059 (4)	0.073 (4)	0.064 (4)	0.022 (3)	−0.010 (3)	0.016 (3)
C10	0.076 (5)	0.075 (4)	0.072 (4)	0.033 (4)	0.001 (4)	0.027 (4)
C11	0.084 (5)	0.059 (4)	0.087 (5)	0.027 (4)	0.018 (4)	0.027 (4)
C12	0.065 (4)	0.054 (3)	0.087 (5)	0.003 (3)	0.000 (4)	0.019 (3)
C13	0.047 (3)	0.052 (3)	0.054 (3)	0.007 (3)	0.010 (3)	0.017 (3)
C14	0.037 (3)	0.051 (3)	0.050 (3)	0.000 (2)	0.004 (2)	0.011 (3)
C15	0.042 (3)	0.058 (3)	0.041 (3)	0.005 (2)	0.001 (2)	0.011 (3)
C16	0.051 (4)	0.061 (4)	0.057 (4)	−0.008 (3)	−0.010 (3)	0.015 (3)
C17	0.063 (4)	0.059 (3)	0.061 (4)	−0.002 (3)	−0.009 (3)	0.021 (3)
C18	0.043 (3)	0.071 (4)	0.045 (3)	0.008 (3)	−0.011 (3)	0.011 (3)
C19	0.049 (3)	0.070 (4)	0.048 (3)	−0.002 (3)	−0.004 (3)	0.013 (3)

C20	0.047 (3)	0.057 (3)	0.049 (3)	0.002 (3)	−0.002 (3)	0.014 (3)

Geometric parameters (Å, º)

Br1—C3	1.913 (5)	C9—C10	1.355 (8)
Br2—C18	1.913 (5)	C9—H9A	0.9300
N1—C7	1.339 (6)	C10—C11	1.414 (9)
N1—C8	1.360 (7)	C10—H10A	0.9300
N2—C14	1.322 (7)	C11—C12	1.350 (9)
N2—C13	1.367 (7)	C11—H11A	0.9300
C1—C2	1.380 (8)	C12—C13	1.400 (8)
C1—C6	1.404 (8)	C12—H12A	0.9300
C1—H1B	0.9300	C14—C15	1.493 (7)
C2—C3	1.383 (8)	C15—C16	1.397 (8)
C2—H2B	0.9300	C15—C20	1.405 (7)
C3—C4	1.383 (8)	C16—C17	1.374 (8)
C4—C5	1.388 (8)	C16—H16A	0.9300
C4—H4A	0.9300	C17—C18	1.392 (8)
C5—C6	1.395 (7)	C17—H17A	0.9300
C5—H5A	0.9300	C18—C19	1.378 (8)
C6—C7	1.484 (7)	C19—C20	1.384 (7)
C7—C14	1.443 (7)	C19—H19A	0.9300
C8—C13	1.412 (7)	C20—H20A	0.9300
C8—C9	1.424 (7)		
C7—N1—C8	117.5 (4)	C11—C10—H10A	120.4
C14—N2—C13	118.5 (4)	C12—C11—C10	121.8 (6)
C2—C1—C6	121.4 (5)	C12—C11—H11A	119.1
C2—C1—H1B	119.3	C10—C11—H11A	119.1
C6—C1—H1B	119.3	C11—C12—C13	120.4 (6)
C1—C2—C3	118.5 (5)	C11—C12—H12A	119.8
C1—C2—H2B	120.8	C13—C12—H12A	119.8
C3—C2—H2B	120.8	N2—C13—C12	120.2 (5)
C2—C3—C4	122.1 (5)	N2—C13—C8	120.9 (5)
C2—C3—Br1	118.5 (4)	C12—C13—C8	118.8 (5)
C4—C3—Br1	119.3 (4)	N2—C14—C7	120.4 (5)
C3—C4—C5	118.6 (5)	N2—C14—C15	115.8 (4)
C3—C4—H4A	120.7	C7—C14—C15	123.7 (5)
C5—C4—H4A	120.7	C16—C15—C20	118.0 (5)
C4—C5—C6	121.2 (5)	C16—C15—C14	122.4 (5)
C4—C5—H5A	119.4	C20—C15—C14	119.4 (5)
C6—C5—H5A	119.4	C17—C16—C15	121.2 (5)
C5—C6—C1	118.1 (5)	C17—C16—H16A	119.4
C5—C6—C7	120.4 (5)	C15—C16—H16A	119.4
C1—C6—C7	121.3 (5)	C16—C17—C18	119.2 (5)
N1—C7—C14	121.5 (5)	C16—C17—H17A	120.4
N1—C7—C6	114.9 (4)	C18—C17—H17A	120.4
C14—C7—C6	123.5 (5)	C19—C18—C17	121.5 (5)

N1—C8—C13	120.9 (5)	C19—C18—Br2	119.8 (4)
N1—C8—C9	119.7 (5)	C17—C18—Br2	118.6 (5)
C13—C8—C9	119.4 (5)	C18—C19—C20	118.6 (5)
C10—C9—C8	120.4 (6)	C18—C19—H19A	120.7
C10—C9—H9A	119.8	C20—C19—H19A	120.7
C8—C9—H9A	119.8	C19—C20—C15	121.5 (5)
C9—C10—C11	119.1 (6)	C19—C20—H20A	119.3
C9—C10—H10A	120.4	C15—C20—H20A	119.3

C6—C1—C2—C3	2.2 (8)	C11—C12—C13—C8	1.0 (9)
C1—C2—C3—C4	−1.9 (8)	N1—C8—C13—N2	5.8 (8)
C1—C2—C3—Br1	178.8 (4)	C9—C8—C13—N2	−176.5 (5)
C2—C3—C4—C5	0.4 (9)	N1—C8—C13—C12	−177.5 (5)
Br1—C3—C4—C5	179.7 (4)	C9—C8—C13—C12	0.2 (8)
C3—C4—C5—C6	0.9 (9)	C13—N2—C14—C7	−2.2 (8)
C4—C5—C6—C1	−0.6 (8)	C13—N2—C14—C15	174.7 (5)
C4—C5—C6—C7	−176.0 (5)	N1—C7—C14—N2	6.1 (8)
C2—C1—C6—C5	−1.0 (8)	C6—C7—C14—N2	−171.5 (5)
C2—C1—C6—C7	174.4 (5)	N1—C7—C14—C15	−170.5 (5)
C8—N1—C7—C14	−3.8 (8)	C6—C7—C14—C15	11.9 (8)
C8—N1—C7—C6	174.0 (5)	N2—C14—C15—C16	−140.4 (6)
C5—C6—C7—N1	54.7 (7)	C7—C14—C15—C16	36.3 (8)
C1—C6—C7—N1	−120.5 (6)	N2—C14—C15—C20	34.6 (7)
C5—C6—C7—C14	−127.6 (6)	C7—C14—C15—C20	−148.6 (6)
C1—C6—C7—C14	57.2 (7)	C20—C15—C16—C17	−0.3 (9)
C7—N1—C8—C13	−1.9 (8)	C14—C15—C16—C17	174.8 (5)
C7—N1—C8—C9	−179.6 (5)	C15—C16—C17—C18	1.7 (9)
N1—C8—C9—C10	177.2 (6)	C16—C17—C18—C19	−1.6 (9)
C13—C8—C9—C10	−0.5 (9)	C16—C17—C18—Br2	−178.0 (5)
C8—C9—C10—C11	−0.3 (10)	C17—C18—C19—C20	0.1 (9)
C9—C10—C11—C12	1.5 (11)	Br2—C18—C19—C20	176.5 (4)
C10—C11—C12—C13	−1.9 (11)	C18—C19—C20—C15	1.3 (9)
C14—N2—C13—C12	179.9 (6)	C16—C15—C20—C19	−1.2 (8)
C14—N2—C13—C8	−3.5 (8)	C14—C15—C20—C19	−176.5 (5)
C11—C12—C13—N2	177.7 (6)		