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2,3-Bis(4-bromophenyl)quinoxaline

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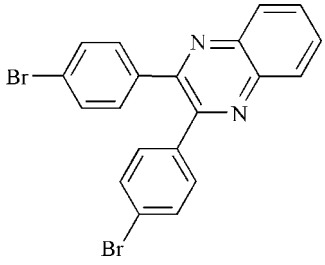
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.047; wR factor = 0.139; data-to-parameter ratio = 13.5.

In the title compound, $C_{20}H_{12}Br_2N_2$, the two bromobenzene rings are inclined at 58.7 (2)° to each other, and at 43.7 (2) and 53.9 (2)° to the quinoxaline ring system.

Related literature

For related literature, see: He *et al.* (2003); Kennedy *et al.* (2004); Loriga *et al.* (1997); Seitz *et al.* (2002).



Experimental

Crystal data

$C_{20}H_{12}Br_2N_2$
M_r = 440.14
Monoclinic, $P2_1/n$
a = 13.5822 (17) Å
b = 7.6918 (13) Å
c = 16.482 (3) Å
β = 104.752 (9)°
V = 1665.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.87 mm^{-1}
T = 298 (2) K
0.35 × 0.26 × 0.18 mm

Data collection

Bruker SMART CCD area detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.281, T_{max} = 0.474
(expected range = 0.246–0.416)
9727 measured reflections
2922 independent reflections
2132 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.139
S = 0.96
2922 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −1.15 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: AT2290).

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supplementary materials

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2,3-Bis(4-bromophenyl)quinoxaline

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Comment

Functionalized quinoxalines represent an important class of nitrogen-containing heterocycle. While rarely described in nature, synthetic quinoxalines are well known in the pharmaceutical industry and have been shown to possess a broad spectrum of biological activities including antiviral, antibacterial and as kinase inhibitors (Loriga *et al.*, 1997; Seitz *et al.*, 2002; He *et al.*, 2003). Recently, we have reported an analogic structure of quinoxaline derivative, namely, 2,3-bis(4-cholrophenyl)quinoxaline. Now we have synthesized a new quinoxaline derivative, 2,3-bis(4-bromophenyl)quinoxaline, (I). We present its crystal structure here.

In the molecular structure of (I) in Fig. 1, the two benzene rings attached to the quinoxaline ring are inclined at an angle of 58.7 (2)°. The quinoxaline ring is approximately planar, with an r. m. s. deviation of 0.033°. The two benzene rings make dihedral angles of 43.7 (2) and 53.9 (2)°, respectively, with the planar quinoxaline ring. The C10—C15 and C9—C173 bond lengths between the benzene rings and the quionxaline ring, [1.499 (5) and 1.480 (5) Å, respectively] are slightly shorter than the general C—C single bond length (Kennedy *et al.*, 2004) as a consequence of the conjugate structure among the aromatic rings.

Experimental

A suspension of 1,2-bis(4-bromophenyl)ethane-1,2-dione (0.8 mmol) and benzene-1,2-diamine (1.0 mmol) in acetic acid (3 ml) was heated to reflux for 0.5 h. The mixture was then poured into ice water and a white precipitate was formed. The mixture was neutralized using saturated $NaHCO_3$ solution. The resulting precipitate was filtered off, washed with water, dried and purified by recrystallization using a mixture of ethyl acetate and petroleum ether (1:5), giving the target product as white needle crystals in 93.3% yield. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform/ethanol (1:1). Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 8.18–8.16 (m, 2H), 7.82–7.80 (m, 2H), 7.52–7.51 (m, 4H), 7.42–7.41 (m, 4H). EI—MS (m/*z*): 440.7 $[M+1]^+$.

Refinement

All H atoms were positioned geometrically and refined as riding (C—H = 0.93%A) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(parent).

Figures

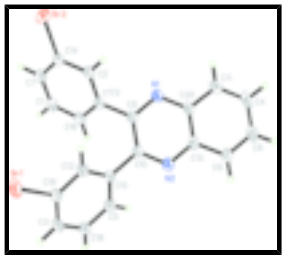


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

2,3-bis(4-bromophenyl)quinoxaline

Crystal data

$C_{20}H_{12}Br_2N_2$	$Z = 4$
$M_r = 440.14$	$F_{000} = 864$
Monoclinic, $P2_1/n$	$D_x = 1.756$ Mg m^{-3}
Hall symbol: -P 2yn	Melting point: 461 K
$a = 13.5822$ (17) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 7.6918$ (13) Å	$\mu = 4.87$ mm^{-1}
$c = 16.482$ (3) Å	$T = 298$ (2) K
$\beta = 104.752$ (9)º	Plate, colourless
$V = 1665.1$ (5) Å^3	0.35 × 0.26 × 0.18 mm

Data collection

Bruker SMART CCD area detector diffractometer	2922 independent reflections
Radiation source: fine-focus sealed tube	2132 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.039$
$T = 293$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan SADABS (Bruker, 1997)	$h = -16 \rightarrow 16$
$T_{min} = 0.281$, $T_{max} = 0.474$	$k = -9 \rightarrow 7$
9727 measured reflections	$l = -19 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.139$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{max} < 0.001$

2922 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
217 parameters	$\Delta\rho_{min} = -1.14$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.08495 (3)	0.95152 (7)	0.11599 (3)	0.0555 (2)
Br2	−0.18098 (4)	1.54598 (9)	−0.17208 (3)	0.0734 (3)
N2	−0.3517 (2)	1.0631 (4)	0.1736 (2)	0.0390 (8)
N1	−0.4070 (2)	1.2803 (5)	0.0336 (2)	0.0370 (8)
C20	−0.4787 (3)	1.1927 (5)	0.0618 (3)	0.0356 (9)
C19	−0.0077 (3)	1.0131 (5)	0.1798 (3)	0.0372 (10)
C18	−0.0427 (3)	1.0550 (6)	0.3121 (3)	0.0477 (12)
H18A	−0.0212	1.0561	0.3703	0.057*
C173	−0.2364 (3)	1.3751 (5)	0.0492 (3)	0.0345 (9)
C16	−0.1655 (3)	1.4680 (5)	0.1092 (3)	0.0410 (10)
H16A	−0.1620	1.4532	0.1659	0.049*
C15	−0.1746 (3)	1.0998 (5)	0.1855 (3)	0.0342 (9)
C14	−0.1739 (3)	1.5134 (6)	−0.0578 (3)	0.0415 (10)
C13	−0.4511 (3)	1.0908 (5)	0.1337 (3)	0.0363 (10)
C12	−0.1065 (3)	1.0592 (5)	0.1387 (3)	0.0362 (10)
H12A	−0.1267	1.0629	0.0804	0.043*
C11	−0.5826 (3)	1.2121 (6)	0.0204 (3)	0.0473 (11)
H11A	−0.6017	1.2783	−0.0283	0.057*
C10	−0.2833 (3)	1.1429 (5)	0.1438 (2)	0.0338 (9)
C9	−0.3111 (3)	1.2620 (5)	0.0751 (2)	0.0338 (9)
C8	−0.6272 (3)	1.0345 (6)	0.1236 (3)	0.0527 (13)
H8A	−0.6779	0.9822	0.1438	0.063*
C7	−0.1039 (3)	1.6059 (6)	0.0012 (3)	0.0479 (11)
H7A	−0.0598	1.6832	−0.0150	0.058*
C6	−0.5277 (3)	1.0109 (6)	0.1654 (3)	0.0465 (11)
H6A	−0.5104	0.9435	0.2138	0.056*
C5	−0.1415 (3)	1.0988 (6)	0.2727 (3)	0.0424 (10)
H5A	−0.1866	1.1280	0.3046	0.051*

C4	−0.6542 (3)	1.1349 (7)	0.0514 (3)	0.0529 (12)
H4A	−0.7226	1.1492	0.0241	0.063*
C3	0.0247 (3)	1.0097 (6)	0.2658 (3)	0.0437 (11)
H3A	0.0910	0.9772	0.2924	0.052*
C2	−0.2412 (3)	1.4004 (6)	−0.0348 (3)	0.0383 (10)
H2B	−0.2895	1.3415	−0.0757	0.046*
C1	−0.1001 (3)	1.5823 (6)	0.0844 (3)	0.0444 (11)
H1B	−0.0528	1.6441	0.1249	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0302 (3)	0.0757 (4)	0.0622 (4)	0.0013 (2)	0.0149 (2)	−0.0061 (3)
Br2	0.0542 (4)	0.1241 (6)	0.0448 (4)	−0.0103 (3)	0.0177 (3)	0.0214 (3)
N2	0.0288 (18)	0.051 (2)	0.037 (2)	−0.0007 (15)	0.0093 (15)	0.0031 (16)
N1	0.0288 (16)	0.048 (2)	0.0335 (19)	0.0014 (15)	0.0074 (14)	0.0005 (16)
C20	0.0237 (18)	0.043 (2)	0.039 (2)	−0.0001 (17)	0.0067 (17)	−0.0026 (19)
C19	0.026 (2)	0.035 (2)	0.050 (3)	−0.0003 (16)	0.0097 (19)	0.0027 (19)
C18	0.038 (2)	0.068 (3)	0.034 (2)	0.005 (2)	0.002 (2)	0.008 (2)
C173	0.0228 (18)	0.044 (2)	0.037 (2)	0.0026 (17)	0.0079 (17)	0.0021 (19)
C16	0.035 (2)	0.048 (3)	0.038 (2)	0.0028 (19)	0.006 (2)	0.001 (2)
C15	0.0252 (19)	0.037 (2)	0.038 (2)	−0.0039 (16)	0.0042 (17)	0.0057 (18)
C14	0.029 (2)	0.059 (3)	0.039 (2)	0.0045 (19)	0.0119 (19)	0.009 (2)
C13	0.027 (2)	0.043 (2)	0.040 (2)	−0.0046 (17)	0.0112 (18)	−0.0037 (19)
C12	0.031 (2)	0.044 (3)	0.033 (2)	−0.0036 (17)	0.0061 (18)	−0.0017 (18)
C11	0.029 (2)	0.060 (3)	0.051 (3)	0.006 (2)	0.0047 (19)	0.006 (2)
C10	0.0263 (18)	0.043 (2)	0.031 (2)	0.0009 (17)	0.0067 (16)	0.0005 (19)
C9	0.0281 (19)	0.042 (2)	0.032 (2)	−0.0017 (17)	0.0084 (16)	−0.0027 (18)
C8	0.029 (2)	0.074 (3)	0.058 (3)	−0.009 (2)	0.017 (2)	−0.006 (3)
C7	0.030 (2)	0.053 (3)	0.060 (3)	−0.003 (2)	0.012 (2)	0.011 (2)
C6	0.033 (2)	0.062 (3)	0.047 (3)	−0.013 (2)	0.014 (2)	0.005 (2)
C5	0.033 (2)	0.059 (3)	0.036 (2)	−0.001 (2)	0.0100 (19)	0.006 (2)
C4	0.022 (2)	0.072 (3)	0.061 (3)	−0.001 (2)	0.005 (2)	−0.010 (3)
C3	0.028 (2)	0.048 (3)	0.048 (3)	0.0003 (18)	−0.002 (2)	0.004 (2)
C2	0.028 (2)	0.051 (2)	0.036 (2)	−0.0021 (18)	0.0082 (17)	0.002 (2)
C1	0.031 (2)	0.052 (3)	0.047 (3)	−0.0066 (19)	0.0054 (19)	0.001 (2)

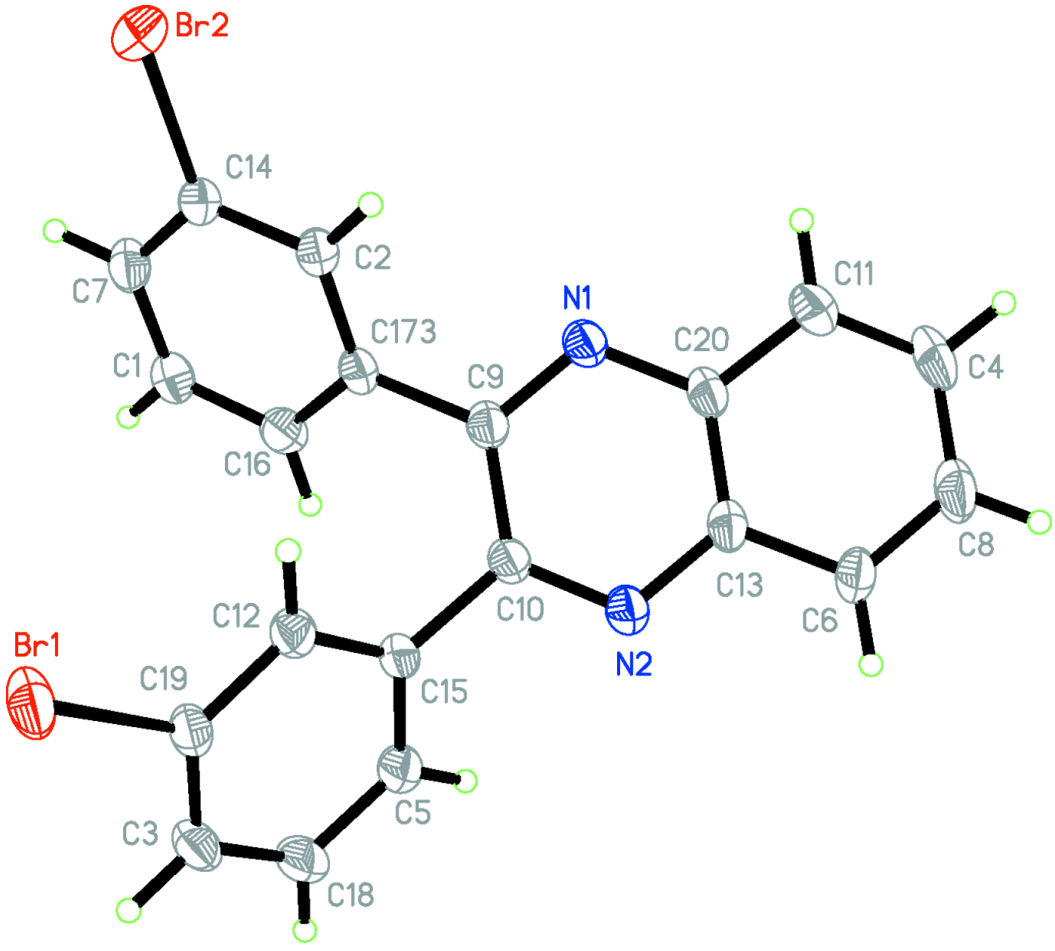
Geometric parameters (Å, °)

Br1—C19	1.894 (4)	C15—C10	1.499 (5)
Br2—C14	1.879 (4)	C14—C7	1.373 (6)
N2—C10	1.309 (5)	C14—C2	1.382 (6)
N2—C13	1.359 (5)	C13—C6	1.418 (6)
N1—C9	1.316 (4)	C12—H12A	0.9300
N1—C20	1.360 (5)	C11—C4	1.346 (6)
C20—C13	1.391 (6)	C11—H11A	0.9300
C20—C11	1.410 (5)	C10—C9	1.431 (5)
C19—C3	1.374 (6)	C8—C6	1.363 (6)
C19—C12	1.387 (6)	C8—C4	1.387 (7)

C18—C5	1.376 (6)	C8—H8A	0.9300
C18—C3	1.376 (7)	C7—C1	1.372 (6)
C18—H18A	0.9300	C7—H7A	0.9300
C173—C16	1.390 (6)	C6—H6A	0.9300
C173—C2	1.383 (6)	C5—H5A	0.9300
C173—C9	1.480 (5)	C4—H4A	0.9300
C16—C1	1.383 (6)	C3—H3A	0.9300
C16—H16A	0.9300	C2—H2B	0.9300
C15—C12	1.384 (6)	C1—H1B	0.9300
C15—C5	1.391 (6)		
C10—N2—C13	117.3 (3)	C20—C11—H11A	120.0
C9—N1—C20	118.0 (3)	N2—C10—C9	121.8 (3)
N1—C20—C13	120.6 (3)	N2—C10—C15	115.8 (3)
N1—C20—C11	119.9 (4)	C9—C10—C15	122.4 (3)
C13—C20—C11	119.4 (4)	N1—C9—C10	120.5 (3)
C3—C19—C12	121.7 (4)	N1—C9—C173	116.6 (3)
C3—C19—Br1	119.0 (3)	C10—C9—C173	122.9 (3)
C12—C19—Br1	119.3 (3)	C6—C8—C4	121.3 (4)
C5—C18—C3	120.5 (4)	C6—C8—H8A	119.4
C5—C18—H18A	119.8	C4—C8—H8A	119.4
C3—C18—H18A	119.8	C1—C7—C14	118.8 (4)
C16—C173—C2	119.2 (4)	C1—C7—H7A	120.6
C16—C173—C9	119.9 (4)	C14—C7—H7A	120.6
C2—C173—C9	120.7 (3)	C8—C6—C13	118.8 (4)
C173—C16—C1	119.8 (4)	C8—C6—H6A	120.6
C173—C16—H16A	120.1	C13—C6—H6A	120.6
C1—C16—H16A	120.1	C18—C5—C15	120.5 (4)
C12—C15—C5	119.3 (4)	C18—C5—H5A	119.7
C12—C15—C10	121.0 (4)	C15—C5—H5A	119.7
C5—C15—C10	119.7 (4)	C11—C4—C8	120.8 (4)
C7—C14—C2	121.3 (4)	C11—C4—H4A	119.6
C7—C14—Br2	119.3 (3)	C8—C4—H4A	119.6
C2—C14—Br2	119.3 (3)	C19—C3—C18	118.9 (4)
N2—C13—C20	121.3 (3)	C19—C3—H3A	120.5
N2—C13—C6	119.1 (4)	C18—C3—H3A	120.5
C20—C13—C6	119.6 (4)	C14—C2—C173	119.8 (4)
C19—C12—C15	119.1 (4)	C14—C2—H2B	120.1
C19—C12—H12A	120.5	C173—C2—H2B	120.1
C15—C12—H12A	120.5	C7—C1—C16	121.1 (4)
C4—C11—C20	120.0 (4)	C7—C1—H1B	119.5
C4—C11—H11A	120.0	C16—C1—H1B	119.5
C9—N1—C20—C13	1.7 (6)	N2—C10—C9—C173	169.7 (4)
C9—N1—C20—C11	178.4 (4)	C15—C10—C9—C173	−11.1 (6)
C2—C173—C16—C1	−0.8 (6)	C16—C173—C9—N1	130.9 (4)
C9—C173—C16—C1	−175.8 (4)	C2—C173—C9—N1	−44.1 (5)
C10—N2—C13—C20	3.2 (6)	C16—C173—C9—C10	−46.5 (6)
C10—N2—C13—C6	−178.2 (4)	C2—C173—C9—C10	138.5 (4)
N1—C20—C13—N2	−5.9 (6)	C2—C14—C7—C1	1.2 (7)

C11—C20—C13—N2	177.4 (4)	Br2—C14—C7—C1	179.4 (3)
N1—C20—C13—C6	175.4 (4)	C4—C8—C6—C13	−0.2 (7)
C11—C20—C13—C6	−1.3 (6)	N2—C13—C6—C8	−177.9 (4)
C3—C19—C12—C15	−1.2 (6)	C20—C13—C6—C8	0.8 (7)
Br1—C19—C12—C15	178.4 (3)	C3—C18—C5—C15	−0.7 (7)
C5—C15—C12—C19	2.1 (6)	C12—C15—C5—C18	−1.1 (6)
C10—C15—C12—C19	−177.3 (4)	C10—C15—C5—C18	178.3 (4)
N1—C20—C11—C4	−175.5 (4)	C20—C11—C4—C8	−0.6 (7)
C13—C20—C11—C4	1.2 (7)	C6—C8—C4—C11	0.1 (8)
C13—N2—C10—C9	3.3 (6)	C12—C19—C3—C18	−0.7 (6)
C13—N2—C10—C15	−175.9 (3)	Br1—C19—C3—C18	179.8 (3)
C12—C15—C10—N2	132.3 (4)	C5—C18—C3—C19	1.6 (7)
C5—C15—C10—N2	−47.2 (5)	C7—C14—C2—C173	−2.0 (6)
C12—C15—C10—C9	−47.0 (6)	Br2—C14—C2—C173	179.8 (3)
C5—C15—C10—C9	133.6 (4)	C16—C173—C2—C14	1.8 (6)
C20—N1—C9—C10	4.7 (6)	C9—C173—C2—C14	176.7 (4)
C20—N1—C9—C173	−172.8 (4)	C14—C7—C1—C16	−0.2 (7)
N2—C10—C9—N1	−7.6 (6)	C173—C16—C1—C7	0.0 (7)
C15—C10—C9—N1	171.6 (4)		

Fig. 1


Br2
C14
C2
C7
C1
C173
C9
N1
C20
C11
C4
C16
C8
C13
C10
C12
C6
Br1
C19
N2
C15
C5
C3
C18