Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

{2,2′-[(5-Bromopyridine-2,3-diyl)bis(nitrilomethylidyne)]diphenolato}-chlorido(*N*,*N*-dimethylformamide)-iron(III)

Ning Sheng[a]* and Zhonghai Ni[b]

[a]Department of Chemistry & Chemical Engineering, Jining University, Qufu 273155, People's Republic of China, and [b]School of Chemistry & Chemical Engineering, Shandong University, Jinan 250100, People's Republic of China
Correspondence e-mail: shengning@mail.sdu.edu.cn

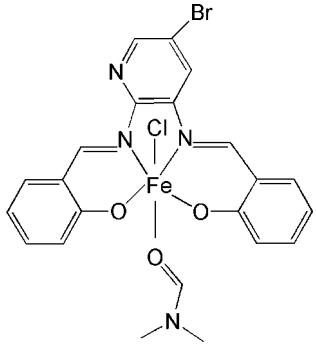
Received 30 July 2009; accepted 10 August 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.039; wR factor = 0.117; data-to-parameter ratio = 15.2.

In the title complex, $[Fe(C_{19}H_{12}BrClN_3O_2)(C_3H_7NO)]$, the Fe^{III} atom is coordinated by an *N*,*N*,*O*,*O*-tetradentate Schiff base ligand and *trans* coordinated by a chloride anion and the O atom of an *N*,*N*-dimethylformamide molecule. The resulting geometry is distorted octahedral within a ClN_2O_3 donor set.

Related literature

For the optical, electronic, magnetic, biological and catalytic properties of complexes containing salicylaldehyde ligands, see: Alam *et al.* (2003); Oshiob *et al.* (2005); Zelewsky & von Knof (1999).



Experimental

Crystal data

$[Fe(C_{19}H_{12}BrClN_3O_2)(C_3H_7NO)]$
M_r = 558.62
Monoclinic, $P2_1/c$
a = 13.1626 (11) Å
b = 15.3553 (13) Å
c = 12.6376 (11) Å
β = 118.186 (1)°
V = 2251.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.60 mm^{-1}
T = 293 K
0.21 × 0.15 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.612, T_{max} = 0.763
11869 measured reflections
4421 independent reflections
3468 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.117
S = 1.04
4421 reflections
291 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

This work was supported by the Postdoctoral Scientific Special Foundation of China (No. 200801414) and the Postdoctoral Scientific Foundation of Shandong Province (No. 200701010). The authors also acknowledge Jining University and Shandong University for support of this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2520).

References

Alam, M. A., Nethaji, M. & Ray, M. (2003). *Angew. Chem. Int. Ed.* **42**, 1940–1942.
Bruker (2004). *APEX2* and *SAINT*-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
Oshiob, H., Nihei, M., Koizumi, S., Shiga, T., Nojiri, H., Nakano, M., Shirakawa, N. & Akatsu, M. (2005). *J. Am. Chem. Soc.* **127**, 4568–4569.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zelewsky, A. & von Knof, U. (1999). *Angew. Chem. Int. Ed.* **38**, 302–322.

supporting information

Acta Cryst. (2009). E**65**, m1202 [doi:10.1107/S1600536809031432]

{2,2′-[(5-Bromopyridine-2,3-diyl)bis(nitrilomethylidyne)]diphenolato}chlorido(*N*,*N*-dimethylformamide)iron(III)

Ning Sheng and Zhonghai Ni

S1. Comment

The synthesis of complexes containing salicylaldehyde ligands has attracted continuous research interest not only because of their appealing structural and topological novelty, but also due to their unusual optical, electronic, magnetic, biological, and catalytic properties (Alam *et al.*, 2003; Zelewsky *et al.*, 1999; Oshiob *et al.*, 2005). In the present paper, we describe the synthesis and structural characterization of the title compound, (I).

In (I), the Fe^{III} atom is tetracoordinated by Schiff base ligand *via* two N and two O atoms, Fig. 1. In addition the metal centre is coordinated by a Cl anion and the O atom of a *N*,*N*-dimethylformamide molecule. The resulting coordination geometry is based on a distorted octahedron in which the Cl and *N*,*N*-dimethylformamide-O atoms define axial sites.

S2. Experimental

Condensation of 4-bromo-*o*-phenylenediamine with salicylaldehyde in a 1:2 molar ratio in ethanol gave the Schiff base ligand. $FeCl_3$ (0.1 mmol) was added dropwise to a solution of the Schiff base (0.1 mmol) in methanol. The resulting solution was stirred at room temperature for 30 minutes. After filtering, the insoluble solids were dissolved in DMF and ether. The product was isolated red-brown crystals in a yield of 45% after a few weeks.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.93–0.96 Å) and were treated as riding on their parent atoms with U(H) set to 1.2U_{eq}(C). The maximum and minimum residual electron density peaks of 1.35 and 0.39 eÅ^{-3}, respectively, were located 1.07 Å and 0.69 Å from the H5 and Br1 atoms, respectively.

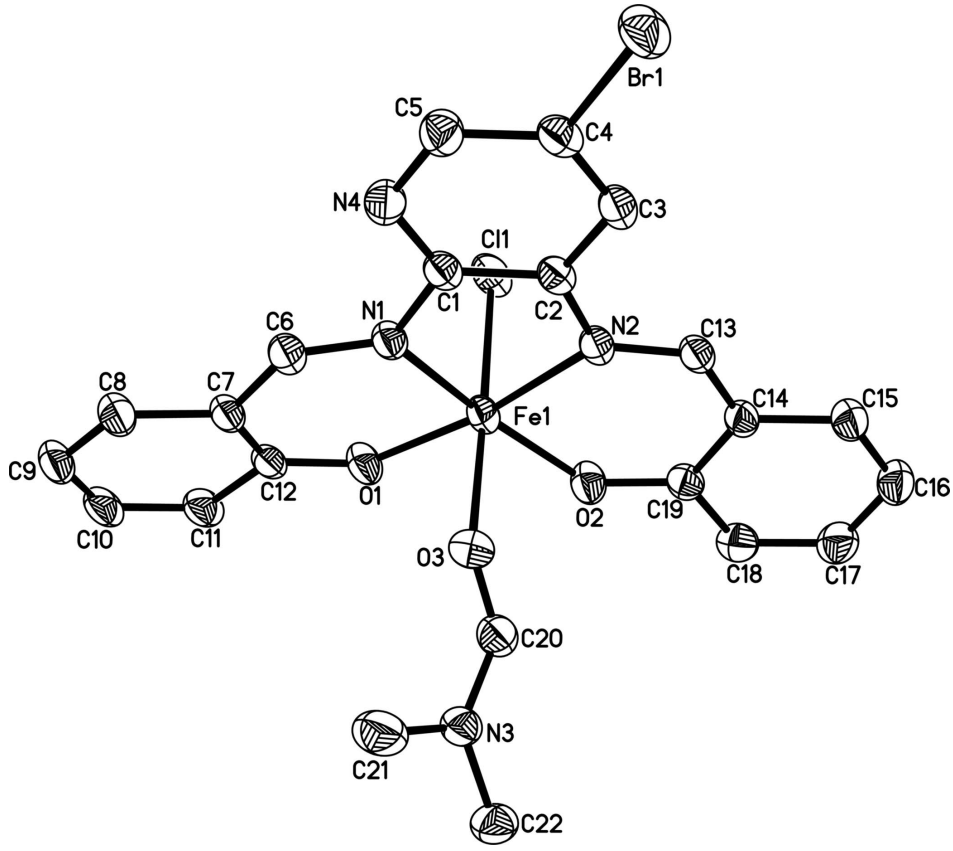


Figure 1

The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity.

{2,2′-[(5-Bromopyridine-2,3- diyl)bis(nitrilomethylidyne)]diphenolato}chlorido(*N*,*N*-dimethylformamide)iron(III)

Crystal data

$[Fe(C_{19}H_{12}BrClN_3O_2)(C_3H_7NO)]$
M_r = 558.62
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.1626 (11) Å
b = 15.3553 (13) Å
c = 12.6376 (11) Å
β = 118.186 (1)°
V = 2251.4 (3) Å^3
Z = 4
$F(000)$ = 1122
D_x = 1.647 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
μ = 2.60 mm^{-1}
T = 293 K
Block, red-brown
0.21 × 0.15 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.612, T_{max} = 0.763
11869 measured reflections
4421 independent reflections
3468 reflections with $I > 2\sigma(I)$

R_{int} = 0.023
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −16→16
k = −14→18
l = −15→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.117
S = 1.04
4421 reflections
291 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
$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 1.0974P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 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}
Fe1	0.81172 (4)	0.45734 (3)	0.12689 (4)	0.04193 (15)
Br1	0.45611 (3)	0.63148 (3)	0.36373 (4)	0.06309 (16)
Cl1	0.68099 (7)	0.34010 (6)	0.05361 (9)	0.0584 (3)
O1	0.94591 (19)	0.38509 (16)	0.1821 (2)	0.0511 (6)
O2	0.78233 (19)	0.49869 (16)	−0.0266 (2)	0.0505 (6)
O3	0.9319 (2)	0.56438 (16)	0.2007 (2)	0.0555 (6)
N1	0.8280 (2)	0.45132 (16)	0.3011 (2)	0.0412 (6)
N2	0.6755 (2)	0.54061 (16)	0.1157 (2)	0.0401 (6)
N3	1.0807 (2)	0.63665 (18)	0.1998 (3)	0.0498 (7)
N4	0.7329 (2)	0.48025 (19)	0.4177 (3)	0.0501 (7)
C1	0.7382 (3)	0.4908 (2)	0.3158 (3)	0.0410 (7)
C2	0.6585 (3)	0.53962 (19)	0.2178 (3)	0.0394 (7)
C3	0.5712 (3)	0.5822 (2)	0.2311 (3)	0.0450 (7)
H3	0.5163	0.6158	0.1694	0.054*
C4	0.5694 (3)	0.5727 (2)	0.3380 (3)	0.0472 (8)
C5	0.6484 (3)	0.5206 (2)	0.4282 (3)	0.0515 (8)
H5	0.6426	0.5135	0.4982	0.062*
C6	0.9158 (3)	0.4183 (2)	0.3952 (3)	0.0452 (7)
H6	0.9172	0.4251	0.4690	0.054*
C7	1.0095 (3)	0.3728 (2)	0.3937 (3)	0.0454 (8)
C8	1.0937 (3)	0.3383 (2)	0.5057 (3)	0.0551 (9)
H8	1.0876	0.3493	0.5748	0.066*

C9	1.1837 (3)	0.2891 (3)	0.5130 (4)	0.0632 (11)
H9	1.2383	0.2667	0.5864	0.076*
C10	1.1924 (3)	0.2731 (2)	0.4092 (4)	0.0612 (10)
H10	1.2529	0.2392	0.4141	0.073*
C11	1.1146 (3)	0.3058 (2)	0.3007 (4)	0.0547 (9)
H11	1.1235	0.2947	0.2332	0.066*
C12	1.0196 (3)	0.3567 (2)	0.2898 (3)	0.0448 (8)
C13	0.6053 (3)	0.5821 (2)	0.0183 (3)	0.0414 (7)
H13	0.5427	0.6095	0.0191	0.050*
C14	0.6146 (3)	0.5897 (2)	−0.0883 (3)	0.0424 (7)
C15	0.5322 (3)	0.6421 (2)	−0.1808 (3)	0.0524 (9)
H15	0.4718	0.6658	−0.1716	0.063*
C16	0.5389 (3)	0.6588 (3)	−0.2836 (3)	0.0594 (10)
H16	0.4838	0.6936	−0.3434	0.071*
C17	0.6287 (3)	0.6233 (3)	−0.2976 (4)	0.0604 (10)
H17	0.6344	0.6355	−0.3667	0.072*
C18	0.7094 (3)	0.5704 (2)	−0.2111 (3)	0.0539 (9)
H18	0.7687	0.5475	−0.2229	0.065*
C19	0.7045 (3)	0.5504 (2)	−0.1065 (3)	0.0439 (7)
C20	0.9823 (3)	0.5955 (2)	0.1477 (3)	0.0512 (8)
H20	0.9474	0.5890	0.0647	0.061*
C21	1.1401 (4)	0.6472 (3)	0.3283 (4)	0.0786 (13)
H21A	1.1111	0.6978	0.3498	0.118*
H21B	1.2212	0.6541	0.3553	0.118*
H21C	1.1277	0.5967	0.3654	0.118*
C22	1.1359 (3)	0.6718 (3)	0.1329 (4)	0.0615 (10)
H22A	1.0840	0.6676	0.0484	0.092*
H22B	1.2047	0.6394	0.1519	0.092*
H22C	1.1553	0.7318	0.1542	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0323 (2)	0.0442 (3)	0.0446 (3)	0.00752 (19)	0.0144 (2)	0.0030 (2)
Br1	0.0530 (2)	0.0760 (3)	0.0671 (3)	0.01648 (18)	0.0340 (2)	0.00264 (19)
Cl1	0.0388 (4)	0.0490 (5)	0.0738 (6)	0.0018 (4)	0.0156 (4)	−0.0049 (4)
O1	0.0363 (12)	0.0610 (15)	0.0522 (14)	0.0125 (10)	0.0177 (11)	0.0032 (11)
O2	0.0456 (13)	0.0555 (14)	0.0484 (13)	0.0124 (11)	0.0205 (11)	0.0053 (11)
O3	0.0466 (13)	0.0568 (15)	0.0615 (15)	−0.0083 (11)	0.0242 (12)	−0.0003 (12)
N1	0.0337 (13)	0.0393 (14)	0.0467 (15)	0.0040 (11)	0.0158 (12)	0.0030 (11)
N2	0.0331 (13)	0.0386 (14)	0.0421 (14)	0.0028 (11)	0.0124 (11)	0.0006 (11)
N3	0.0424 (15)	0.0472 (16)	0.0608 (19)	0.0015 (12)	0.0252 (14)	0.0027 (13)
N4	0.0489 (16)	0.0571 (17)	0.0465 (16)	0.0089 (14)	0.0244 (14)	0.0070 (13)
C1	0.0333 (16)	0.0375 (16)	0.0491 (18)	0.0010 (13)	0.0169 (14)	−0.0013 (13)
C2	0.0326 (15)	0.0386 (16)	0.0434 (17)	−0.0005 (12)	0.0151 (13)	−0.0031 (13)
C3	0.0350 (16)	0.0421 (17)	0.0494 (19)	0.0038 (13)	0.0131 (14)	0.0007 (14)
C4	0.0375 (17)	0.0488 (19)	0.057 (2)	0.0019 (14)	0.0239 (16)	−0.0022 (16)
C5	0.053 (2)	0.055 (2)	0.051 (2)	0.0086 (17)	0.0283 (17)	0.0060 (16)

C6	0.0380 (17)	0.0472 (18)	0.0455 (19)	0.0030 (14)	0.0157 (15)	0.0015 (14)
C7	0.0305 (16)	0.0424 (18)	0.054 (2)	0.0017 (13)	0.0119 (14)	0.0072 (14)
C8	0.0396 (18)	0.056 (2)	0.055 (2)	0.0002 (16)	0.0102 (16)	0.0072 (17)
C9	0.0354 (18)	0.057 (2)	0.074 (3)	0.0052 (16)	0.0073 (18)	0.019 (2)
C10	0.0312 (17)	0.051 (2)	0.090 (3)	0.0099 (15)	0.0195 (19)	0.0130 (19)
C11	0.0398 (18)	0.0478 (19)	0.076 (3)	0.0064 (15)	0.0266 (18)	0.0050 (17)
C12	0.0274 (15)	0.0389 (16)	0.060 (2)	0.0006 (12)	0.0140 (15)	0.0058 (14)
C13	0.0313 (15)	0.0369 (16)	0.0504 (19)	0.0021 (13)	0.0145 (14)	0.0007 (14)
C14	0.0333 (15)	0.0376 (16)	0.0442 (18)	−0.0038 (13)	0.0084 (14)	0.0019 (13)
C15	0.0421 (19)	0.053 (2)	0.052 (2)	0.0020 (15)	0.0136 (16)	0.0062 (16)
C16	0.054 (2)	0.059 (2)	0.051 (2)	0.0041 (18)	0.0129 (18)	0.0119 (17)
C17	0.061 (2)	0.068 (2)	0.046 (2)	−0.0066 (19)	0.0198 (18)	0.0068 (17)
C18	0.052 (2)	0.059 (2)	0.052 (2)	−0.0045 (17)	0.0257 (18)	−0.0023 (17)
C19	0.0378 (17)	0.0435 (18)	0.0429 (18)	−0.0063 (13)	0.0128 (14)	−0.0031 (14)
C20	0.0449 (19)	0.051 (2)	0.053 (2)	−0.0002 (16)	0.0191 (17)	−0.0008 (16)
C21	0.061 (3)	0.101 (3)	0.064 (3)	−0.027 (2)	0.022 (2)	−0.004 (2)
C22	0.056 (2)	0.062 (2)	0.080 (3)	0.0015 (18)	0.043 (2)	0.008 (2)

Geometric parameters (Å, º)

Fe1—O2	1.900 (2)	C7—C8	1.424 (5)
Fe1—O1	1.917 (2)	C8—C9	1.371 (5)
Fe1—N1	2.110 (3)	C8—H8	0.9300
Fe1—N2	2.152 (3)	C9—C10	1.391 (6)
Fe1—O3	2.164 (2)	C9—H9	0.9300
Fe1—Cl1	2.3566 (10)	C10—C11	1.361 (5)
Br1—C4	1.899 (3)	C10—H10	0.9300
O1—C12	1.317 (4)	C11—C12	1.425 (5)
O2—C19	1.312 (4)	C11—H11	0.9300
O3—C20	1.238 (4)	C13—C14	1.414 (5)
N1—C6	1.307 (4)	C13—H13	0.9300
N1—C1	1.416 (4)	C14—C15	1.412 (5)
N2—C13	1.304 (4)	C14—C19	1.441 (5)
N2—C2	1.409 (4)	C15—C16	1.367 (5)
N3—C20	1.306 (4)	C15—H15	0.9300
N3—C21	1.441 (5)	C16—C17	1.388 (6)
N3—C22	1.454 (5)	C16—H16	0.9300
N4—C1	1.332 (4)	C17—C18	1.373 (5)
N4—C5	1.334 (4)	C17—H17	0.9300
C1—C2	1.403 (4)	C18—C19	1.388 (5)
C2—C3	1.399 (4)	C18—H18	0.9300
C3—C4	1.370 (5)	C20—H20	0.9300
C3—H3	0.9300	C21—H21A	0.9600
C4—C5	1.378 (5)	C21—H21B	0.9600
C5—H5	0.9300	C21—H21C	0.9600
C6—C7	1.426 (5)	C22—H22A	0.9600
C6—H6	0.9300	C22—H22B	0.9600
C7—C12	1.404 (5)	C22—H22C	0.9600

O2—Fe1—O1	105.85 (10)	C9—C8—H8	119.5
O2—Fe1—N1	162.06 (10)	C7—C8—H8	119.5
O1—Fe1—N1	88.45 (10)	C8—C9—C10	119.1 (3)
O2—Fe1—N2	88.45 (10)	C8—C9—H9	120.5
O1—Fe1—N2	164.57 (11)	C10—C9—H9	120.5
N1—Fe1—N2	76.44 (10)	C11—C10—C9	121.9 (3)
O2—Fe1—O3	86.61 (10)	C11—C10—H10	119.1
O1—Fe1—O3	85.54 (10)	C9—C10—H10	119.1
N1—Fe1—O3	83.71 (10)	C10—C11—C12	120.5 (4)
N2—Fe1—O3	89.66 (10)	C10—C11—H11	119.7
O2—Fe1—Cl1	95.47 (8)	C12—C11—H11	119.7
O1—Fe1—Cl1	94.43 (8)	O1—C12—C7	124.0 (3)
N1—Fe1—Cl1	94.13 (8)	O1—C12—C11	117.8 (3)
N2—Fe1—Cl1	89.83 (7)	C7—C12—C11	118.2 (3)
O3—Fe1—Cl1	177.85 (8)	N2—C13—C14	126.8 (3)
C12—O1—Fe1	132.1 (2)	N2—C13—H13	116.6
C19—O2—Fe1	134.1 (2)	C14—C13—H13	116.6
C20—O3—Fe1	121.9 (2)	C15—C14—C13	117.3 (3)
C6—N1—C1	118.7 (3)	C15—C14—C19	118.3 (3)
C6—N1—Fe1	125.2 (2)	C13—C14—C19	124.4 (3)
C1—N1—Fe1	116.0 (2)	C16—C15—C14	121.7 (4)
C13—N2—C2	121.4 (3)	C16—C15—H15	119.2
C13—N2—Fe1	123.3 (2)	C14—C15—H15	119.2
C2—N2—Fe1	114.89 (19)	C15—C16—C17	119.3 (4)
C20—N3—C21	120.2 (3)	C15—C16—H16	120.4
C20—N3—C22	122.4 (3)	C17—C16—H16	120.4
C21—N3—C22	117.3 (3)	C18—C17—C16	121.1 (4)
C1—N4—C5	117.8 (3)	C18—C17—H17	119.5
N4—C1—C2	124.0 (3)	C16—C17—H17	119.5
N4—C1—N1	120.2 (3)	C17—C18—C19	121.5 (4)
C2—C1—N1	115.8 (3)	C17—C18—H18	119.2
C3—C2—C1	117.2 (3)	C19—C18—H18	119.2
C3—C2—N2	126.8 (3)	O2—C19—C18	119.8 (3)
C1—C2—N2	116.0 (3)	O2—C19—C14	122.1 (3)
C4—C3—C2	117.8 (3)	C18—C19—C14	118.1 (3)
C4—C3—H3	121.1	O3—C20—N3	124.8 (4)
C2—C3—H3	121.1	O3—C20—H20	117.6
C3—C4—C5	121.3 (3)	N3—C20—H20	117.6
C3—C4—Br1	120.0 (3)	N3—C21—H21A	109.5
C5—C4—Br1	118.7 (3)	N3—C21—H21B	109.5
N4—C5—C4	121.8 (3)	H21A—C21—H21B	109.5
N4—C5—H5	119.1	N3—C21—H21C	109.5
C4—C5—H5	119.1	H21A—C21—H21C	109.5
N1—C6—C7	125.2 (3)	H21B—C21—H21C	109.5
N1—C6—H6	117.4	N3—C22—H22A	109.5
C7—C6—H6	117.4	N3—C22—H22B	109.5
C12—C7—C8	119.4 (3)	H22A—C22—H22B	109.5

C12—C7—C6	124.3 (3)	N3—C22—H22C	109.5
C8—C7—C6	116.2 (3)	H22A—C22—H22C	109.5
C9—C8—C7	120.9 (4)	H22B—C22—H22C	109.5