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Isolation and Magnetic Properties of Heterocycle-Carrying *N*-Alkoxyaminyl Radicals

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Experimental Section

General. The ^1H NMR, ESR, UV-Vis, and SQUID measurements were carried out as previously reported.¹ 2,6-Diarylanilines (**3**) were obtained by the Pd-catalyzed cross-coupling reaction of 2,6-dibromoaniline with arylboronic acid in benzene–water–EtOH in the presence of K_2CO_3 (yields 99%).² Silica gel column chromatography was carried out on silica gel 60N (Kanto Chem. Co., Inc.) and alumina column chromatography on Merck aluminium oxide 90.

4-Bromo-2,6-diphenylaniline (4a). A mixture of 4.44 g (18.1 mmol) of 2,6-diphenylaniline, 9.18 g (23.5 mmol) of benzyltrimethylammonium tribromide³ (BTMA Br_3), and 2.6 g of CaCO_3 in 100 mL of CH_2Cl_2 and 40 mL of MeOH was stirred at room temperature for 2 h. After filtration, the filtrate was washed with 10% NaHSO_3 , extracted with CH_2Cl_2 , dried (MgSO_4), evaporated, and chromatographed on silica gel with 1 : 2 CH_2Cl_2 –hexane, giving a colorless oil in 82% yield. ^1H NMR (CDCl_3) δ 3.82 (s, 2H), 7.23 (s, 2H), 7.34–7.48 (m, 10H). Anal. Calcd for $\text{C}_{18}\text{H}_{14}\text{BrN}$: C, 66.68, H, 4.35; N, 4.32. Found: C, 66.67; H, 4.51; N, 4.06.

4-Bromo-2,6-bis(4-chlorophenyl)aniline (4b). In the same manner as above, **4b** was obtained from 2,6-bis(4-chlorophenyl)aniline with BTMA Br_3 . Colorless needles (from hexane); yield 94%; mp 157–158 °C; ^1H NMR (CDCl_3) δ 3.74 (s, 2H), 7.20 (s, 2H), 7.40 and 7.44 (each d, $J = 8.8$ Hz, 8H). Calcd for $\text{C}_{18}\text{H}_{12}\text{BrCl}_2\text{N}$: C, 55.00; H, 3.08; N, 3.56. Found: C, 54.82; H, 3.21; N, 3.43.

General Procedure for Preparation of 2,6-Diaryl-4-(4-pyridyl)anilines (5). A suspension of 15.4 mmol of **4**, 4.10 g (20 mmol) of 2-(4-pyridyl)-4,4,5,5-tetramethyl-1,3-dioxaborolane,⁴ 2.50 g (7.7 mmol) of tetra-*n*-butylammonium bromide, and 3.50 g of powdered KOH in 200 mL of THF was stirred for 10 min, and 1.80 g (1.54 mmol) of $\text{Pd}(\text{PPh}_3)_4$ was added. The resultant mixture was purged with N_2 and refluxed for 24 h with stirring. After filtration, the filtrate was evaporated, and water was added. The mixture was extracted with CH_2Cl_2 , and the combined extracts were dried (MgSO_4), evaporated, and

chromatographed on alumina with 1:4 hexane-CH₂Cl₂. Crystallization from hexane-ethyl acetate gave **5** as colorless needles.

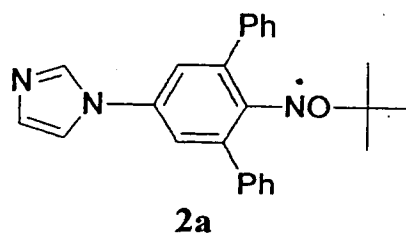
2,6-Diphenyl-4-(4-pyridyl)aniline (5a): yield 81%; mp 175–176 °C; ¹H NMR (CDCl₃) δ 4.06 (s, 2H), 7.40 (t, *J* = 7.3 Hz, 2H), 7.46 (s, 2H), 7.48–7.55 (m, 10H), 8.56 (dd, *J* = 4.9 and 1.5 Hz, 2H). Anal. Calcd for C₂₃H₁₈N₂: C, 85.68; H, 5.63; N, 8.69. Found: C, 85.52; H, 5.78; N, 8.71.

2,6-Bis(4-Chlorophenyl)-4-(4-pyridyl)aniline (5b): yield 94%; 248–250 °C; ¹H NMR (CDCl₃) δ 3.97 (s, 2H), 7.42 (s, 2H), 7.47–7.49 (m, 10H), 8.58 (d, *J* = 4.9 Hz, 2H). Anal. Calcd for C₂₃H₁₆Cl₂N₂: C, 70.60; H, 4.12; N, 7.16. Found: C, 70.73; H, 4.21; N, 7.19.

References

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- (3) Kajigaeshi, S.; Kakinami, T.; Inoue, K.; Kondo, M.; Nakamura, H.; Fujikawa, M.; Okamoto, T. *Bull. Chem. Soc. Jpn.* **1988**, *61*, 597.
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X-ray Crystallographic Data of **2a**



EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₂₅ H ₂₄ ON ₃
Formula Weight	382.48
Crystal Color, Habit	red, needle
Crystal Dimensions	0.35 X 0.08 X 0.06 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2 θ range)	15703 (2.8 - 54.8°)
Indexing Images	2 oscillations at 6.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 14.4575(4) Å b = 24.7605(6) Å c = 5.9936(1) Å β = 99.483(1)° V = 2116.24(9) Å ³
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.200 g/cm ³
F ₀₀₀	812.00
μ (MoK α)	0.74 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoK α (λ = 0.71069 Å) graphite monochromated

Temperature	23.0 °C
Voltage, Current	50 kV, 100 mA
Collimator Size	0.5 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	84 exposures at 4.7 minutes per degree
Oscillation Range ($\phi=0.0^\circ, \chi=45.0^\circ$)	ω 130.0 - 190.0° with 2.0° step
Oscillation Range ($\phi=180.0^\circ, \chi=45.0^\circ$)	ω 0.0 - 162.0° with 3.0° step
Camera Radius	127.40 mm
Pixel Size	0.125 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 18089 Unique: 4682 ($R_{int} = 0.065$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.5634 - 0.9955)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(F_o^2)}$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. of Reflections (All, $2\theta < 54.96^\circ$)	4682
No. Variables	262
Reflection/Parameter Ratio	17.87
Residuals: R; Rw	0.080 ; 0.150
Residuals: R1	0.055

No. of Reflections to calc R1	2647
Goodness of Fit Indicator	1.30
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.40 e^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.47 e^{-}/\text{\AA}^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.1048(1)	-0.19428(6)	0.1898(3)	3.81(4)
N(1)	0.1412(1)	-0.14659(7)	0.2794(3)	3.52(4)
N(2)	0.4871(1)	-0.08318(7)	0.0771(3)	3.48(4)
N(3)	0.5926(2)	-0.06450(9)	-0.1412(4)	5.20(6)
C(1)	0.2308(1)	-0.13863(8)	0.2434(4)	2.95(4)
C(2)	0.2838(1)	-0.17245(8)	0.1163(3)	2.85(4)
C(3)	0.3681(2)	-0.15303(8)	0.0682(4)	3.25(5)
C(4)	0.4021(1)	-0.10243(8)	0.1408(4)	3.16(5)
C(5)	0.3536(2)	-0.07079(8)	0.2743(4)	3.29(5)
C(6)	0.2693(2)	-0.08790(8)	0.3292(4)	3.15(5)
C(7)	0.2545(1)	-0.22822(8)	0.0361(4)	2.94(5)
C(8)	0.2591(2)	-0.27049(9)	0.1887(4)	3.82(5)
C(9)	0.2326(2)	-0.32224(10)	0.1182(5)	4.70(6)
C(10)	0.2017(2)	-0.3324(1)	-0.1043(5)	4.98(7)
C(11)	0.1975(2)	-0.2913(1)	-0.2584(5)	5.23(7)
C(12)	0.2249(2)	-0.23920(10)	-0.1893(4)	4.14(6)
C(13)	0.5140(2)	-0.0895(1)	-0.1284(5)	4.63(6)
C(14)	0.6173(2)	-0.0409(1)	0.0658(5)	4.57(6)
C(15)	0.5541(2)	-0.05129(9)	0.2028(4)	3.95(6)
C(16)	0.2165(2)	-0.05251(9)	0.4651(4)	3.35(5)
C(17)	0.2006(2)	0.0010(1)	0.4044(5)	4.59(6)
C(18)	0.1490(2)	0.0339(1)	0.5265(6)	5.94(8)
C(19)	0.1150(2)	0.0135(1)	0.7100(6)	6.13(8)
C(20)	0.1322(2)	-0.0386(1)	0.7759(5)	5.15(7)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(21)	0.1827(2)	-0.07146(10)	0.6542(4)	4.07(6)
C(22)	0.0047(2)	-0.19908(10)	0.2072(4)	3.64(5)
C(23)	-0.0176(2)	-0.2551(1)	0.1133(5)	4.99(7)
C(24)	-0.0077(2)	-0.1941(1)	0.4512(5)	6.18(8)
C(25)	-0.0484(2)	-0.1558(1)	0.0639(5)	5.54(7)
H(1)	0.3792	-0.0369	0.3283	4.0
H(2)	0.4041	-0.1751	-0.0144	3.9
H(3)	0.1961	-0.1070	0.7033	5.1
H(4)	0.2795	-0.2637	0.3447	4.6
H(5)	0.2356	-0.3508	0.2275	5.8
H(6)	0.2236	-0.2113	-0.2998	5.2
H(7)	0.1831	-0.3682	-0.1562	5.9
H(8)	0.2245	0.0158	0.2786	5.5
H(9)	0.1074	-0.0528	0.9012	6.2
H(10)	0.1375	0.0703	0.4855	7.2
H(11)	0.1765	-0.2986	-0.4144	6.4
H(12)	0.0812	0.0360	0.7957	7.6
H(13)	0.5553	-0.0387	0.3528	4.9
H(14)	0.4801	-0.1101	-0.2493	5.5
H(15)	0.6729	-0.0200	0.1115	5.6
H(16)	0.0218	-0.2808	0.2026	6.0
H(17)	-0.0811	-0.2635	0.1168	6.0
H(18)	-0.0058	-0.2567	-0.0380	6.0
H(19)	-0.0393	-0.1607	-0.0872	6.6

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(20)	-0.1120	-0.1580	0.0762	6.6
H(21)	-0.0236	-0.1216	0.1178	6.6
H(22)	0.0164	-0.1595	0.5087	7.8
H(23)	-0.0712	-0.1966	0.4646	7.8
H(24)	0.0275	-0.2215	0.5389	7.8

$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^* \cos \gamma + 2U_{13}aa^*cc^* \cos \beta + 2U_{23}bb^*cc^* \cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.0352(8)	0.0408(9)	0.073(1)	-0.0093(7)	0.0213(8)	-0.0146(8)
N(1)	0.0385(10)	0.040(1)	0.057(1)	-0.0075(9)	0.0137(9)	-0.0111(9)
N(2)	0.039(1)	0.0315(10)	0.065(1)	-0.0054(8)	0.0179(10)	-0.0021(9)
N(3)	0.061(1)	0.052(1)	0.093(2)	-0.013(1)	0.039(1)	0.001(1)
C(1)	0.037(1)	0.032(1)	0.045(1)	-0.0026(10)	0.0109(10)	-0.0014(10)
C(2)	0.035(1)	0.030(1)	0.043(1)	-0.0015(9)	0.0067(10)	-0.0023(9)
C(3)	0.040(1)	0.032(1)	0.054(1)	-0.0012(10)	0.017(1)	-0.006(1)
C(4)	0.036(1)	0.033(1)	0.053(1)	-0.0050(10)	0.012(1)	0.000(1)
C(5)	0.040(1)	0.030(1)	0.056(1)	-0.0057(10)	0.011(1)	-0.006(1)
C(6)	0.040(1)	0.033(1)	0.047(1)	-0.0019(10)	0.010(1)	-0.0042(10)
C(7)	0.030(1)	0.032(1)	0.052(1)	-0.0017(9)	0.0133(10)	-0.006(1)
C(8)	0.049(1)	0.037(1)	0.057(1)	-0.005(1)	0.004(1)	0.000(1)
C(9)	0.060(2)	0.035(1)	0.084(2)	-0.004(1)	0.012(1)	0.003(1)
C(10)	0.059(2)	0.040(1)	0.092(2)	-0.015(1)	0.018(2)	-0.020(2)
C(11)	0.067(2)	0.069(2)	0.063(2)	-0.015(2)	0.010(1)	-0.028(2)
C(12)	0.060(2)	0.049(1)	0.049(1)	-0.010(1)	0.011(1)	-0.007(1)
C(13)	0.064(2)	0.046(1)	0.074(2)	-0.013(1)	0.033(1)	-0.007(1)
C(14)	0.040(1)	0.043(1)	0.093(2)	-0.005(1)	0.015(1)	0.010(1)
C(15)	0.038(1)	0.039(1)	0.072(2)	-0.006(1)	0.007(1)	-0.001(1)
C(16)	0.038(1)	0.036(1)	0.053(1)	-0.0031(10)	0.010(1)	-0.012(1)
C(17)	0.062(2)	0.040(1)	0.076(2)	-0.001(1)	0.021(1)	-0.007(1)
C(18)	0.079(2)	0.040(1)	0.110(3)	0.011(1)	0.026(2)	-0.012(2)
C(19)	0.070(2)	0.064(2)	0.107(3)	0.005(2)	0.039(2)	-0.031(2)
C(20)	0.065(2)	0.067(2)	0.068(2)	-0.005(1)	0.026(1)	-0.021(2)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(21)	0.056(1)	0.044(1)	0.056(1)	0.001(1)	0.013(1)	-0.008(1)
C(22)	0.031(1)	0.052(1)	0.057(1)	-0.007(1)	0.014(1)	-0.005(1)
C(23)	0.045(1)	0.054(2)	0.092(2)	-0.015(1)	0.013(1)	-0.003(1)
C(24)	0.051(2)	0.124(3)	0.064(2)	-0.021(2)	0.022(1)	-0.005(2)
C(25)	0.055(2)	0.062(2)	0.093(2)	0.002(1)	0.007(2)	-0.008(2)

The general temperature factor expression:

$$\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	N(1)	1.366(2)	O(1)	C(22)	1.473(2)
N(1)	C(1)	1.362(3)	N(2)	C(4)	1.427(3)
N(2)	C(13)	1.360(3)	N(2)	C(15)	1.374(3)
N(3)	C(13)	1.307(3)	N(3)	C(14)	1.365(4)
C(1)	C(2)	1.436(3)	C(1)	C(6)	1.434(3)
C(2)	C(3)	1.384(3)	C(2)	C(7)	1.500(3)
C(3)	C(4)	1.389(3)	C(4)	C(5)	1.389(3)
C(5)	C(6)	1.381(3)	C(6)	C(16)	1.490(3)
C(7)	C(8)	1.384(3)	C(7)	C(12)	1.375(3)
C(8)	C(9)	1.383(3)	C(9)	C(10)	1.358(4)
C(10)	C(11)	1.368(4)	C(11)	C(12)	1.393(4)
C(14)	C(15)	1.350(3)	C(16)	C(17)	1.382(3)
C(16)	C(21)	1.388(3)	C(17)	C(18)	1.392(3)
C(18)	C(19)	1.373(4)	C(19)	C(20)	1.360(4)
C(20)	C(21)	1.379(3)	C(22)	C(23)	1.512(3)
C(22)	C(24)	1.508(4)	C(22)	C(25)	1.502(4)

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(3)	H(2)	0.95	C(5)	H(1)	0.95
C(8)	H(4)	0.95	C(9)	H(5)	0.96
C(10)	H(7)	0.96	C(11)	H(11)	0.95
C(12)	H(6)	0.96	C(13)	H(14)	0.95
C(14)	H(15)	0.96	C(15)	H(13)	0.95
C(17)	H(8)	0.95	C(18)	H(10)	0.94
C(19)	H(12)	0.95	C(20)	H(9)	0.95
C(21)	H(3)	0.94	C(23)	H(16)	0.96
C(23)	H(17)	0.94	C(23)	H(18)	0.95
C(24)	H(22)	0.97	C(24)	H(23)	0.94
C(24)	H(24)	0.95	C(25)	H(19)	0.94
C(25)	H(20)	0.94	C(25)	H(21)	0.96

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(1)	O(1)	C(22)	111.2(2)	O(1)	N(1)	C(1)	112.2(2)
C(4)	N(2)	C(13)	126.3(2)	C(4)	N(2)	C(15)	127.2(2)
C(13)	N(2)	C(15)	106.3(2)	C(13)	N(3)	C(14)	104.4(2)
N(1)	C(1)	C(2)	127.2(2)	N(1)	C(1)	C(6)	113.2(2)
C(2)	C(1)	C(6)	119.4(2)	C(1)	C(2)	C(3)	118.3(2)
C(1)	C(2)	C(7)	124.0(2)	C(3)	C(2)	C(7)	117.7(2)
C(2)	C(3)	C(4)	121.8(2)	N(2)	C(4)	C(3)	119.6(2)
N(2)	C(4)	C(5)	120.3(2)	C(3)	C(4)	C(5)	120.2(2)
C(4)	C(5)	C(6)	120.9(2)	C(1)	C(6)	C(5)	119.3(2)
C(1)	C(6)	C(16)	120.5(2)	C(5)	C(6)	C(16)	120.1(2)
C(2)	C(7)	C(8)	120.2(2)	C(2)	C(7)	C(12)	121.5(2)
C(8)	C(7)	C(12)	118.2(2)	C(7)	C(8)	C(9)	121.2(2)
C(8)	C(9)	C(10)	120.0(2)	C(9)	C(10)	C(11)	119.8(2)
C(10)	C(11)	C(12)	120.6(2)	C(7)	C(12)	C(11)	120.2(2)
N(2)	C(13)	N(3)	112.4(2)	N(3)	C(14)	C(15)	111.7(2)
N(2)	C(15)	C(14)	105.2(2)	C(6)	C(16)	C(17)	119.8(2)
C(6)	C(16)	C(21)	121.9(2)	C(17)	C(16)	C(21)	118.2(2)
C(16)	C(17)	C(18)	120.2(3)	C(17)	C(18)	C(19)	120.0(3)
C(18)	C(19)	C(20)	120.6(2)	C(19)	C(20)	C(21)	119.6(3)
C(16)	C(21)	C(20)	121.3(2)	O(1)	C(22)	C(23)	101.5(2)
O(1)	C(22)	C(24)	109.7(2)	O(1)	C(22)	C(25)	108.5(2)
C(23)	C(22)	C(24)	112.5(2)	C(23)	C(22)	C(25)	112.5(2)
C(24)	C(22)	C(25)	111.4(2)				

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(3)	H(2)	119.1	C(4)	C(3)	H(2)	119.1
C(4)	C(5)	H(1)	119.2	C(6)	C(5)	H(1)	119.9
C(7)	C(8)	H(4)	119.4	C(9)	C(8)	H(4)	119.4
C(8)	C(9)	H(5)	119.7	C(10)	C(9)	H(5)	120.3
C(9)	C(10)	H(7)	121.1	C(11)	C(10)	H(7)	119.2
C(10)	C(11)	H(11)	119.6	C(12)	C(11)	H(11)	119.8
C(7)	C(12)	H(6)	120.4	C(11)	C(12)	H(6)	119.5
N(2)	C(13)	H(14)	124.1	N(3)	C(13)	H(14)	123.5
N(3)	C(14)	H(15)	124.9	C(15)	C(14)	H(15)	123.3
N(2)	C(15)	H(13)	127.5	C(14)	C(15)	H(13)	127.2
C(16)	C(17)	H(8)	120.7	C(18)	C(17)	H(8)	119.1
C(17)	C(18)	H(10)	120.7	C(19)	C(18)	H(10)	119.3
C(18)	C(19)	H(12)	120.2	C(20)	C(19)	H(12)	119.2
C(19)	C(20)	H(9)	120.4	C(21)	C(20)	H(9)	119.9
C(16)	C(21)	H(3)	119.4	C(20)	C(21)	H(3)	119.2
C(22)	C(23)	H(16)	109.3	C(22)	C(23)	H(17)	110.0
C(22)	C(23)	H(18)	109.3	H(16)	C(23)	H(17)	109.4
H(16)	C(23)	H(18)	108.8	H(17)	C(23)	H(18)	109.9
C(22)	C(24)	H(22)	108.9	C(22)	C(24)	H(23)	110.7
C(22)	C(24)	H(24)	109.8	H(22)	C(24)	H(23)	109.1
H(22)	C(24)	H(24)	107.9	H(23)	C(24)	H(24)	110.4
C(22)	C(25)	H(19)	108.6	C(22)	C(25)	H(20)	109.3
C(22)	C(25)	H(21)	108.1	H(19)	C(25)	H(20)	111.2
H(19)	C(25)	H(21)	109.4	H(20)	C(25)	H(21)	110.1

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
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Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	N(1)	C(1)	C(2)	3.8(3)	O(1)	N(1)	C(1)	C(6)	178.0(2)
N(1)	O(1)	C(22)	C(23)	-176.6(2)	N(1)	O(1)	C(22)	C(24)	-57.3(3)
N(1)	O(1)	C(22)	C(25)	64.6(2)	N(1)	C(1)	C(2)	C(3)	169.6(2)
N(1)	C(1)	C(2)	C(7)	-11.7(4)	N(1)	C(1)	C(6)	C(5)	-170.1(2)
N(1)	C(1)	C(6)	C(16)	6.5(3)	N(2)	C(4)	C(3)	C(2)	-176.9(2)
N(2)	C(4)	C(5)	C(6)	177.2(2)	N(2)	C(13)	N(3)	C(14)	-0.2(3)
N(2)	C(15)	C(14)	N(3)	0.5(3)	N(3)	C(13)	N(2)	C(4)	175.9(2)
N(3)	C(13)	N(2)	C(15)	0.5(3)	C(1)	N(1)	O(1)	C(22)	-173.4(2)
C(1)	C(2)	C(3)	C(4)	0.5(3)	C(1)	C(2)	C(7)	C(8)	-72.2(3)
C(1)	C(2)	C(7)	C(12)	109.6(3)	C(1)	C(6)	C(5)	C(4)	-1.1(3)
C(1)	C(6)	C(16)	C(17)	-127.4(3)	C(1)	C(6)	C(16)	C(21)	53.0(3)
C(2)	C(1)	C(6)	C(5)	4.6(3)	C(2)	C(1)	C(6)	C(16)	-178.9(2)
C(2)	C(3)	C(4)	C(5)	3.1(4)	C(2)	C(7)	C(8)	C(9)	-179.8(2)
C(2)	C(7)	C(12)	C(11)	-179.6(2)	C(3)	C(2)	C(1)	C(6)	-4.2(3)
C(3)	C(2)	C(7)	C(8)	106.5(2)	C(3)	C(2)	C(7)	C(12)	-71.7(3)
C(3)	C(4)	N(2)	C(13)	39.7(3)	C(3)	C(4)	N(2)	C(15)	-145.7(2)
C(3)	C(4)	C(5)	C(6)	-2.7(4)	C(4)	N(2)	C(15)	C(14)	-175.9(2)
C(4)	C(3)	C(2)	C(7)	-178.3(2)	C(4)	C(5)	C(6)	C(16)	-177.6(2)
C(5)	C(4)	N(2)	C(13)	-140.2(2)	C(5)	C(4)	N(2)	C(15)	34.4(3)
C(5)	C(6)	C(16)	C(17)	49.1(3)	C(5)	C(6)	C(16)	C(21)	-130.5(2)
C(6)	C(1)	C(2)	C(7)	174.4(2)	C(6)	C(16)	C(17)	C(18)	178.2(3)
C(6)	C(16)	C(21)	C(20)	-178.6(2)	C(7)	C(8)	C(9)	C(10)	0.3(4)
C(7)	C(12)	C(11)	C(10)	-1.6(4)	C(8)	C(7)	C(12)	C(11)	2.3(3)
C(8)	C(9)	C(10)	C(11)	0.5(4)	C(9)	C(8)	C(7)	C(12)	-1.6(3)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(9)	C(10)	C(11)	C(12)	0.2(4)	C(13)	N(2)	C(15)	C(14)	-0.5(3)
C(13)	N(3)	C(14)	C(15)	-0.2(3)	C(16)	C(17)	C(18)	C(19)	1.0(5)
C(16)	C(21)	C(20)	C(19)	0.0(4)	C(17)	C(16)	C(21)	C(20)	1.8(4)
C(17)	C(18)	C(19)	C(20)	0.9(5)	C(18)	C(17)	C(16)	C(21)	-2.2(4)
C(18)	C(19)	C(20)	C(21)	-1.4(5)					

