Supporting Information

N-Nitration of Secondary Amines Using 4-Chloro-5-methoxy-2-nitropyridazin-3-one

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1. Physical, Spectroscopic and Elemental analytical Data for compounds 2a - 2f and 4a - 4l.

General Comments. Melting points were determined with a capillary apparatus and uncorrected. ^1H and ^{13}C NMR spectra were recorded on a 500 MHz spectrometer with chemical shift values reported in δ units (ppm) relative to an internal standard (TMS). X-Ray analysis was carried out on a imaging plate diffractometer with graphite monochromated Mo-K α radiation. Open-bed chromatography was carried out on silica gel 60 (70 ~ 230 mesh, Merck) using the gravity flow. The column was packed as slurries with the elution solvent.

4,5-Dichloro-2-nitropyridazin-3-one (2a): mp 48-50 °C; IR (KBr) 3050, 1720, 1640, 1500, 1360, 1280, 1230, 1000, 920 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.73 (s, 1H); ^{13}C NMR (CDCl_3) δ 133.1, 137.8, 138.4, 149.1; Anal. Calcd for $\text{C}_4\text{HN}_3\text{O}_3\text{Cl}_2$: C, 22.88; H, 0.48; N, 20.01. Found: C, 22.92; H 0.52; N, 20.19.

4-Chloro-5-methoxy-2-nitropyridazin-3-one (2b): mp 84-86 °C; IR (KBr) 3150, 3100, 1720, 1650, 1620, 1520, 1480, 1420, 1330, 1300, 1100 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.21 (s, 3H), 7.80 (s, 1H); ^{13}C NMR (CDCl_3) δ 58.6, 118.5, 124.6, 151.4, 155.8; Anal. Calcd for $\text{C}_5\text{H}_4\text{N}_3\text{O}_4\text{Cl}$: C, 29.22; H, 1.96; N, 20.44. Found: C, 29.28; H 2.01; N, 20.53.

4-Chloro-2-nitro-5-phenoxy pyridazin-3-one (2c): mp 110-112 °C; IR (KBr) 3050, 1720, 1640, 1600, 1500, 1380, 1260, 1180, 1100, 1040 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.13-7.16 (m, 2H), 7.36-7.39 (m, 2H), 7.48-7.53(m, 2H); ^{13}C NMR (CDCl_3) δ 119.8, 121.7, 126.9, 127.0, 130.8, 151.33, 152.9, 154.2; Anal. Calcd for $\text{C}_{10}\text{H}_6\text{N}_3\text{O}_4\text{Cl}$: C, 44.88; H, 2.26; N, 15.70. Found: C, 44.90; H 2.32; N, 15.81.

5-Ethoxy-4-chloro-2-nitropyridazin-3-one (2d): mp 111-113 °C; IR (KBr) 3050, 3000, 1710, 1630, 1600, 1380, 1200, 1020, 820 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.50 (t, 3H, $J = 7.0$ Hz), 4.12 (q, 2H, $J = 7.0$ Hz), 6.18 (s, 1H); ^{13}C NMR (CDCl_3) δ 13.8, 67.1, 105.7, 132.1, 153.9, 157.2. Anal. Calcd for $\text{C}_6\text{H}_6\text{N}_3\text{O}_4\text{Cl}$: C, 32.82; H, 2.75; N, 19.14. Found: C, 32.90; H 2.81; N, 19.20.

5-Azido-4-chloro-2-nitropyridazin-3-one (2e): mp 78-80 °C; IR (KBr) 3050, 2150, 1700, 1640, 1600, 1520, 1400, 1320, 1280, 1240, 1020, 1080 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.51 (s, 1H); ^{13}C NMR (CDCl_3) δ 124.9, 128.0, 141.0, 150.0; Anal. Calcd for $\text{C}_4\text{H}_4\text{N}_6\text{O}_2\text{Cl}$: C, 22.19; H, 0.47; N, 38.81. Found: C, 22.23; H 0.54; N, 38.90.

4,5-Dibromo-2-nitropyridazin-3-one (2f): mp 71-73 °C; IR (KBr) 3050, 1720, 1660, 1580, 1520, 1300, 1240, 1200, 1160, 1100, 900, 790, 740 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.71 (s, 1H); ^{13}C NMR (CDCl_3) δ 132.6, 133.7, 134.7, 148.9; Anal. Calcd for $\text{C}_4\text{H}_2\text{N}_4\text{O}_3\text{Br}_2$: C, 16.07; H, 0.34; N, 14.06. Found: C, 16.12; H 0.50; N, 14.13.

N-Methyl-N-nitrobenzylamine (4a): mp oil; $R_f = 0.73$ (CH_2Cl_2); IR (KBr) 3020, 1550, 1500, 1300, 1050, 800, 750, 700, 690 cm^{-1} ; ^1H NMR (CDCl_3) δ 3.35 (s, 3H), 4.98 (s, 2H), 7.26-7.27 (m, 2H), 7.34-7.36 (m, 3H); ^{13}C NMR (CDCl_3) δ 38.1, 56.0, 128.1, 128.5, 129.0, 134.2; Anal. Calcd for $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_2$: C, 57.82; H, 6.07; N, 16.86. Found: C, 57.89; H, 6.23; N, 16.97.

***N*-Isopropyl-*N*-nitrobenzylamine (4b):** mp 45 °C; R_f = 0.71 (CH_2Cl_2); IR (KBr) 3100, 2900, 1500, 1460, 1380, 1300, 1220, 1140, 1060, 950, 770, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.28 (d, 6H, J = 6.6 Hz), 4.86 (s, 2H), 4.92-5.00 (m, 1H), 7.26-7.38 (m, 5H); ^{13}C NMR (CDCl_3) δ 19.5, 49.7, 53.0, 127.0, 127.7, 128.7, 135.8; Anal. Calcd for $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_2$: C, 61.84; H, 7.27; N, 14.42. Found: C, 61.91; H, 7.31; N, 14.31.

***N*-Nitrodiethylamine (4c):** mp oil; R_f = 0.76 (CH_2Cl_2); IR (KBr) 3000, 2900, 1500, 1460, 1380, 1280, 1100 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.29 (t, 6H, J = 6.9 Hz), 3.82 (q, 4H, J = 6.9 Hz); ^{13}C NMR (CDCl_3) δ 11.5, 45.9; Anal. Calcd for $\text{C}_4\text{H}_{10}\text{N}_2\text{O}_2$: C, 40.67; H, 8.53; N, 23.71. Found: C, 40.73; H, 8.65; N, 23.80.

***N*-Nitrodicyclohexylamine (4d):** mp 125-127 °C; R_f = 0.73 ($\text{EtOA}/n\text{-hexane}$ = 1:6, v/v); IR (KBr) 2950, 2890, 1510, 1410, 1300, 1250, 1200, 1090 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.15-1.42 (m, 6H), 1.65-1.88 (m, 14 H), 4.03 (m, 2H); ^{13}C NMR (CDCl_3) δ 25.0, 25.8, 29.3, 59.6; Anal. Calcd for $\text{C}_{12}\text{H}_{22}\text{N}_2\text{O}_2$: C, 63.69; H, 9.80; N, 12.38. Found: C, 63.72; H, 9.97; N, 12.42.

4-Nitromorpholine (4e): mp 50 °C; R_f = 0.77 (CH_2Cl_2); IR (KBr) 3000, 2850, 1510, 1380, 1320, 1260, 1120, 980, 860 cm^{-1} ; ^1H NMR (CDCl_3) δ 3.82 (s, 8H); ^{13}C NMR (CDCl_3) δ 48.8, 65.3; Anal. Calcd for $\text{C}_4\text{H}_8\text{N}_2\text{O}_3$: C, 36.36; H, 6.10; N, 21.20. Found: C, 36.41; H, 6.22; N, 21.31.

2,6-Dimethyl-4-nitromorpholine (4f): mp oil; $R_f = 0.77$ (EtOAc/*n*-hexane = 1:3, v/v); IR (KBr) 3000, 2950, 2900, 1500, 1400, 1350, 1260, 1180, 1080, 1040 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.28 (d, 6H, $J = 6.3$ Hz), 2.83 (dd, 2H, $J = 10.5$ Hz and 12.6 Hz), 3.66-3.77 (m, 2H), 4.41 (dd, 2H, $J = 2.4$ Hz and 13.2 Hz); ^{13}C NMR (CDCl_3) δ 18.5, 53.1, 70.3; Anal. Calcd for $\text{C}_6\text{H}_{12}\text{N}_2\text{O}_3$: C, 44.99; H, 7.55; N, 17.49. Found: C, 45.05; H, 7.62; N, 17.51.

N-Nitro-3-pyrroline (4g): mp 95-96 $^\circ\text{C}$; $R_f = 0.65$ (EtOAc/*n*-hexane = 1:4, v/v); IR (KBr) 3050, 2950, 2900, 1510, 1460, 1380, 1330, 1160, 1010, 960, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 4.61 (s, 4H), 5.91 (s, 2H); ^{13}C NMR (CDCl_3) δ 56.2, 124.5; Anal. Calcd for $\text{C}_4\text{H}_6\text{N}_2\text{O}_2$: C, 42.11; H, 5.30; N, 24.55. Found: C, 42.23; H, 5.41; N, 24.67.

1-Nitrohomopiperazine (4h): mp oil; $R_f = 0.15$ (EtOAc/*n*-hexane = 3:1, v/v); IR (KBr) 3350, 3000, 2900, 1650, 1520, 1480, 1400, 1350, 1300, 1160, 930, 780 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.69-1.75 (m, 3H), 2.69 (t, 2H, $J = 5.5$ Hz), 2.86 (t, 2H, $J = 5.5$ Hz), 3.90-3.99 (m, 4H); ^{13}C NMR (CDCl_3) δ 33.1, 51.7, 53.3, 54.4, 58.2; Anal. Calcd for $\text{C}_5\text{H}_{11}\text{N}_3\text{O}_2$: C, 41.37; H, 7.64; N, 28.95. Found: C, 41.42; H, 7.75; N, 29.03.

2-Methyl-4-nitropiperazine (4i): mp 65-67 $^\circ\text{C}$; $R_f = 0.10$ (EtOAc/*n*-hexane = 3:1, v/v); IR (KBr) 3400, 2950, 2800, 1520, 1400, 1320, 1240, 1120, 1020, 1000 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.04 (d, 3H, $J = 6.0$ Hz), 2.63-2.79 (m, 4H), 2.98-3.14 (m, 2H), 4.26 (t, 2H, $J = 7.8$ Hz); ^{13}C NMR (CDCl_3) δ 18.7, 43.5, 48.3, 49.2, 54.6; Anal. Calcd for $\text{C}_5\text{H}_{11}\text{N}_3\text{O}_2$: C, 41.37; H, 7.64; N, 28.95. Found: C, 41.41; H, 7.72; N, 29.02.

***N*-Nitrodecahydroquinoline (4j):** mp oil; $R_f = 0.66$ (EtOAc/*n*-hexane = 1:3, v/v); IR (KBr) 2900, 2850, 1500, 1450, 1320, 1250, 1000, 750 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.07-1.21 (m, 2H), 1.24-1.50 (m, 2H), 1.57-1.97 (m, 8H), 2.36-2.40 (m, 1H), 3.52-3.69 (m, 2H), 4.47 (m, 1H); ^{13}C NMR (CDCl_3) δ 21.0, 24.9, 25.5, 26.0, 29.7, 32.6, 38.0, 43.7, 66.5; Anal. Calcd for $\text{C}_9\text{H}_{16}\text{N}_2\text{O}_2$: C, 58.67; H, 8.75; N, 15.20. Found: C, 58.74; H, 8.80; N, 15.12.

***N*-Nitrosodicyclohexylamine (4k):** mp 107-108 $^\circ\text{C}$; $R_f = 0.63$ (EtOAc/*n*-hexane = 1:6, v/v); IR (KBr) 2950, 2890, 1450, 1400, 1340, 1250, 1180, 1120 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.13-1.46 (m, 9H), 1.70-1.95 (m, 11H), 3.71 (m, 1H), 4.89 (m, 1H); ^{13}C NMR (CDCl_3) δ 25.1, 25.2, 25.4, 26.0, 29.3, 34.3, 52.0, 58.4; Anal. Calcd for $\text{C}_{12}\text{H}_{22}\text{N}_2\text{O}$: C, 68.53; H, 10.54; N, 13.32. Found: C, 68.66; H, 10.62; N, 13.39.

1,4-Dinitrohomopiperazine (4l): mp 147-148 $^\circ\text{C}$; $R_f = 0.49$ (CH_2Cl_2); IR (KBr) 2980, 1770, 1670, 1520, 1400, 1300, 1280, 1050, 940 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.89-1.97 (m, 2H), 4.02 (t, 4H, $J = 5.6$ Hz), 4.22 (s, 4H); ^{13}C NMR (CDCl_3) δ 23.1, 47.8, 49.5; Anal. Calcd for $\text{C}_5\text{H}_{10}\text{N}_4\text{O}_4$: C, 31.58; H, 5.30; N, 29.46. Found: C, 31.63; H, 5.42; N, 29.51.

2. X-Ray analytical Data of *N*-Nitrosodicyclohexylamine (4e)

Experimental

Data Collection

A colorless block crystal of $C_{12}H_{22}N_2O$ having approximate dimensions of 0.20 x 0.20 x 0.07 mm was mounted in a loop. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-K α radiation.

Indexing was performed from 3 oscillations which were exposed for 0.6 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

$$a = 5.762(4) \text{ \AA}$$

$$b = 19.53(2) \text{ \AA} \quad \beta = 94.04(3)^\circ$$

$$c = 10.835(9) \text{ \AA}$$

$$V = 1216(1) \text{ \AA}^3$$

For $Z = 4$ and F.W. = 210.32, the calculated density is 1.15 g/cm³. The systematic absences of:

$$h0l: h+l \pm 2n$$

$$0k0: k \pm 2n$$

uniquely determine the space group to be:

$$P2_1/n \text{ (#14)}$$

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The data were collected at a temperature of $-180 \pm 1^\circ\text{C}$ to a maximum 2θ value of 60.1° . A total of 44 images, corresponding to 220.0° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 0.50 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 14641 reflections which were collected, 3528 were unique ($R_{\text{int}} = 0.119$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 0.7 cm^{-1} . An absorption correction was not applied. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement³ was based on 3527 observed reflections ($I > -3.00\sigma(I)$, $2\theta < 60.07$) and 136 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma (F_o^2 - F_c^2) / \Sigma F_o^2 = 0.113$$

$$R_w = [(\Sigma w (F_o^2 - F_c^2)^2 / \Sigma w (F_o^2)^2)]^{1/2} = 0.125$$

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.068 \quad \text{for } I > 2.0\sigma(I) \text{ data}$$

The standard deviation of an observation of unit weight⁴ was 1.08. The weighting scheme was based on counting statistics and included a factor ($p = 0.003$) to downweight the intense reflections. Plots of $\Sigma w (F_o^2 - F_c^2)^2$ versus F_o^2 -reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.62 and $-0.46\text{ e}^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in F_{calc} ⁶; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁷. The values for the mass attenuation coefficients are those of Creagh and Hubbel⁸. All calculations were performed using the teXsan⁹ crystallographic software package of Molecular Structure Corporation.

References

- (1) SHELXS86: Sheldrick, G.M. (1985). In: "Crystallographic Computing 3" (Eds G.M. Sheldrick, C. Kruger and R. Goddard) Oxford University Press, pp. 175-189.
- (2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\sum w(F_o^2 - F_c^2)^2$ where

$$w = 1/[\sigma^2(F_o^2)] = [\sigma_c^2(F_o^2) + (p (\text{Max}(F_o^2, 0) + 2F_c^2)/3)^2]^{-1}$$

$\sigma_c(F_o^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$[\sum w(|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations

N_v = number of variables

- (5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).
- (6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).
- (7) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).
- (8) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).
- (9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	C ₁₂ H ₂₂ N ₂ O
Formula Weight	210.32
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.20 X 0.20 X 0.07 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2 θ range)	8645 (7.3 - 60.1 $^{\circ}$)
Indexing Images	3 oscillations at 0.6 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 5.762(4) Å b = 19.53(2) Å c = 10.835(9) Å β = 94.04(3) $^{\circ}$ V = 1216(1) Å ³
Space Group	P2 ₁ /n (#14)
Z value	4
D _{calc}	1.149 g/cm ³
F ₀₀₀	464.00
μ (MoK α)	0.73 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Temperature	-180.0 °C
Voltage, Current	60 kV, 90 mA
Collimator Size	0.5 mm
Detector Aperture	280.0 mm x 256.0 mm
Data Images	44 exposures at 0.5 minutes per degree
Oscillation Range ($\phi=0.0^\circ, \chi=45.0^\circ$)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range ($\phi=180.0^\circ, \chi=45.0^\circ$)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\text{max}}$	60.1°
No. of Reflections Measured	Total: 14641 Unique: 3528 ($R_{\text{int}} = 0.119$)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS-86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/\sigma^2(F_o^2)$
p-factor	0.0030
Anomalous Dispersion	All non-hydrogen atoms
No. of Observations ($I > 3.00\sigma(I)$, $2\theta < 60.07^\circ$)	3527
No. Variables	136
Reflection/Parameter Ratio	25.93
Residuals: R; Rw	0.113 ; 0.125
Residuals: R1	0.068
No. of Reflections to calc R1	1735
Goodness of Fit Indicator	1.08
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.62 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.46 \text{ e}^-/\text{\AA}^3$

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

atom	x	y	z	B _{eq}
O(1)	0.1315(3)	0.71326(8)	0.9304(1)	3.17(4)
N(1)	0.2735(3)	0.75341(9)	0.7633(1)	2.12(3)
N(2)	0.1661(3)	0.76487(10)	0.8661(2)	2.71(4)
C(1)	0.3256(3)	0.8158(1)	0.6927(2)	2.10(4)
C(2)	0.1050(4)	0.8513(1)	0.6412(2)	2.77(5)
C(3)	0.1644(4)	0.9147(1)	0.5669(2)	2.95(5)
C(4)	0.3188(4)	0.9637(1)	0.6434(2)	2.91(5)
C(5)	0.5390(4)	0.9280(1)	0.6940(2)	2.85(5)
C(6)	0.4837(4)	0.8644(1)	0.7689(2)	2.45(4)
C(7)	0.3525(3)	0.6851(1)	0.7262(2)	2.19(4)
C(8)	0.2688(4)	0.6681(1)	0.5922(2)	2.54(4)
C(9)	0.3473(4)	0.5958(1)	0.5603(2)	2.81(5)
C(10)	0.6093(4)	0.5871(1)	0.5834(2)	2.87(5)
C(11)	0.6918(4)	0.6059(1)	0.7162(2)	2.63(4)
C(12)	0.6167(3)	0.6784(1)	0.7475(2)	2.39(4)
H(1)	0.4076	0.8016	0.6240	2.5
H(2a)	0.0167	0.8205	0.5888	3.3
H(2b)	0.0162	0.8647	0.7077	3.3
H(3a)	0.2428	0.9007	0.4968	3.6
H(3b)	0.0242	0.9376	0.5400	3.6
H(4a)	0.3594	1.0011	0.5930	3.5
H(4b)	0.2370	0.9803	0.7104	3.5
H(5a)	0.6267	0.9147	0.6269	3.4
H(5b)	0.6283	0.9588	0.7459	3.4

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

atom	x	y	z	B _{eq}
H(6a)	0.4086	0.8781	0.8402	3.0
H(6b)	0.6248	0.8414	0.7936	3.0
H(7)	0.2848	0.6525	0.7779	2.6
H(8a)	0.1037	0.6706	0.5831	3.1
H(8b)	0.3329	0.7001	0.5379	3.1
H(9a)	0.2716	0.5638	0.6100	3.4
H(9b)	0.3048	0.5870	0.4755	3.4
H(10a)	0.6489	0.5407	0.5686	3.5
H(10b)	0.6851	0.6159	0.5281	3.5
H(11a)	0.6269	0.5745	0.7712	3.2
H(11b)	0.8568	0.6031	0.7253	3.2
H(12a)	0.6895	0.7101	0.6962	2.9
H(12b)	0.6620	0.6878	0.8318	2.9

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.0438(9)	0.0511(10)	0.0261(7)	-0.0013(8)	0.0077(7)	0.0073(7)
N(1)	0.0276(9)	0.0326(9)	0.0207(8)	0.0016(8)	0.0026(7)	0.0020(7)
N(2)	0.033(1)	0.047(1)	0.0230(9)	-0.0019(8)	0.0031(8)	0.0040(8)
C(1)	0.029(1)	0.029(1)	0.0221(9)	0.0012(9)	0.0040(8)	0.0003(8)
C(2)	0.031(1)	0.041(1)	0.032(1)	-0.0007(10)	-0.0044(9)	0.0037(10)
C(3)	0.034(1)	0.041(1)	0.036(1)	0.003(1)	-0.0034(10)	0.010(1)
C(4)	0.045(1)	0.034(1)	0.032(1)	0.002(1)	0.006(1)	0.0031(9)
C(5)	0.039(1)	0.036(1)	0.033(1)	-0.005(1)	-0.0039(10)	0.0008(9)
C(6)	0.031(1)	0.038(1)	0.0239(10)	0.0007(9)	-0.0035(9)	0.0019(9)
C(7)	0.029(1)	0.030(1)	0.0238(9)	-0.0029(9)	0.0002(8)	0.0010(8)
C(8)	0.035(1)	0.036(1)	0.025(1)	0.0003(10)	-0.0051(9)	-0.0019(9)
C(9)	0.045(1)	0.038(1)	0.0230(10)	0.000(1)	-0.0091(9)	-0.0031(9)
C(10)	0.050(1)	0.032(1)	0.027(1)	0.006(1)	-0.0002(10)	-0.0024(9)
C(11)	0.030(1)	0.039(1)	0.030(1)	0.0030(10)	-0.0019(9)	0.0007(9)
C(12)	0.032(1)	0.034(1)	0.0238(9)	0.0007(9)	-0.0051(8)	-0.0020(9)

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(2)	1.249(2)	N(1)	N(2)	1.331(2)
N(1)	C(1)	1.481(3)	N(1)	C(7)	1.474(3)
C(1)	C(2)	1.519(3)	C(1)	C(6)	1.518(3)
C(2)	C(3)	1.529(3)	C(3)	C(4)	1.514(3)
C(4)	C(5)	1.517(3)	C(5)	C(6)	1.529(3)
C(7)	C(8)	1.533(3)	C(7)	C(12)	1.529(3)
C(8)	C(9)	1.530(3)	C(9)	C(10)	1.522(3)
C(10)	C(11)	1.528(3)	C(11)	C(12)	1.525(3)

Table 4. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.95	C(2)	H(2a)	0.95
C(2)	H(2b)	0.95	C(3)	H(3a)	0.95
C(3)	H(3b)	0.95	C(4)	H(4a)	0.95
C(4)	H(4b)	0.95	C(5)	H(5a)	0.95
C(5)	H(5b)	0.95	C(6)	H(6a)	0.95
C(6)	H(6b)	0.95	C(7)	H(7)	0.95
C(8)	H(8a)	0.95	C(8)	H(8b)	0.95
C(9)	H(9a)	0.95	C(9)	H(9b)	0.95
C(10)	H(10a)	0.95	C(10)	H(10b)	0.95
C(11)	H(11a)	0.95	C(11)	H(11b)	0.95
C(12)	H(12a)	0.95	C(12)	H(12b)	0.95

Table 5. Bond Angles($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
N(2)	N(1)	C(1)	114.6(2)	N(2)	N(1)	C(7)	123.5(2)
C(1)	N(1)	C(7)	121.7(1)	O(1)	N(2)	N(1)	115.7(2)
N(1)	C(1)	C(2)	111.8(2)	N(1)	C(1)	C(6)	111.6(2)
C(2)	C(1)	C(6)	111.7(2)	C(1)	C(2)	C(3)	110.5(2)
C(2)	C(3)	C(4)	111.6(2)	C(3)	C(4)	C(5)	110.6(2)
C(4)	C(5)	C(6)	111.4(2)	C(1)	C(6)	C(5)	111.0(2)
N(1)	C(7)	C(8)	111.8(2)	N(1)	C(7)	C(12)	111.1(2)
C(8)	C(7)	C(12)	111.5(2)	C(7)	C(8)	C(9)	109.6(2)
C(8)	C(9)	C(10)	111.8(2)	C(9)	C(10)	C(11)	111.5(2)
C(10)	C(11)	C(12)	110.9(2)	C(7)	C(12)	C(11)	110.0(2)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(1)	C(1)	H(1)	107.1	C(2)	C(1)	H(1)	107.2
C(6)	C(1)	H(1)	107.2	C(1)	C(2)	H(2a)	109.2
C(1)	C(2)	H(2b)	109.2	C(3)	C(2)	H(2a)	109.2
C(3)	C(2)	H(2b)	109.2	H(2a)	C(2)	H(2b)	109.5
C(2)	C(3)	H(3a)	108.9	C(2)	C(3)	H(3b)	108.9
C(4)	C(3)	H(3a)	109.0	C(4)	C(3)	H(3b)	109.0
H(3a)	C(3)	H(3b)	109.4	C(3)	C(4)	H(4a)	109.2
C(3)	C(4)	H(4b)	109.3	C(5)	C(4)	H(4a)	109.2
C(5)	C(4)	H(4b)	109.2	H(4a)	C(4)	H(4b)	109.4
C(4)	C(5)	H(5a)	109.0	C(4)	C(5)	H(5b)	109.0
C(6)	C(5)	H(5a)	109.0	C(6)	C(5)	H(5b)	109.0
H(5a)	C(5)	H(5b)	109.4	C(1)	C(6)	H(6a)	109.1
C(1)	C(6)	H(6b)	109.1	C(5)	C(6)	H(6a)	109.1
C(5)	C(6)	H(6b)	109.1	H(6a)	C(6)	H(6b)	109.4
N(1)	C(7)	H(7)	107.4	C(8)	C(7)	H(7)	107.3
C(12)	C(7)	H(7)	107.4	C(7)	C(8)	H(8a)	109.4
C(7)	C(8)	H(8b)	109.5	C(9)	C(8)	H(8a)	109.4
C(9)	C(8)	H(8b)	109.5	H(8a)	C(8)	H(8b)	109.4
C(8)	C(9)	H(9a)	108.9	C(8)	C(9)	H(9b)	108.9
C(10)	C(9)	H(9a)	108.9	C(10)	C(9)	H(9b)	108.9
H(9a)	C(9)	H(9b)	109.5	C(9)	C(10)	H(10a)	109.0
C(9)	C(10)	H(10b)	109.0	C(11)	C(10)	H(10a)	108.9
C(11)	C(10)	H(10b)	109.0	H(10a)	C(10)	H(10b)	109.4
C(10)	C(11)	H(11a)	109.1	C(10)	C(11)	H(11b)	109.0

Table 6. Bond Angles($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(12)	C(11)	H(11a)	109.2	C(12)	C(11)	H(11b)	109.1
H(11a)	C(11)	H(11b)	109.5	C(7)	C(12)	H(12a)	109.3
C(7)	C(12)	H(12b)	109.3	C(11)	C(12)	H(12a)	109.4
C(11)	C(12)	H(12b)	109.3	H(12a)	C(12)	H(12b)	109.4

Table 7. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	C(1) ¹⁾	3.495(2)	O(1)	C(12) ²⁾	3.516(3)

Symmetry operations

(1) $X-1/2, -Y+3/2, Z+1/2$ (2) $X-1, Y, Z$