Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Silver(I) Catalyzed Intramolecular Cyclization of N-(2-(alk-1-yn-1-yl))Tetrazoles leading to the formation of N-cyano-2-substituted Indoles under ambient conditions

Sangeetha Panaka,[a, b] Rajiv Trivedi,[a, b]* T. Sony,[c] S. Prabhakar,[c] L. Raju Chowhan[d]

[a]Inorganic and Physical Chemistry Division, CSIR-Indian Institute of Chemical Technology, Hyderabad 500007, Telangana, India, trivedi@iict.res.in, Fax: +91-40-2716-0921; Tel: +91-40-2719-1667
[b]Academy of Scientific and Innovative Research, AcSIR CSIR-IICT Campus, Hyderabad, 500007
[c]Analytical Chemistry and Mass spectrometry Division, CSIR-Indian Institute of Chemical Technology, Hyderabad 500007, Telangana, India
[d]School of Chemical Sciences, Central University of Gujarat, Sector 30, Gandhinagar, Gujarat - 382030, India

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X-ray Crystallography Experimental Section

Experimental section for 4ah

Data Collection

A colorless block crystal of $C_{15}H_9FN_2$ having approximate dimensions of 0.500 x 0.480 x 0.320 mm was mounted on a glass fiber. All measurements were made on a Rigaku SCX mini diffractometer using graphite monochromated Mo-Ka radiation.

The crystal-to-detector distance was 52.00 mm.

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

a = 25.51(2) Å

b = 13.881(8) Å

c = 6.794(4) Å

V = 2406(2) $Å^3$

For Z = 8 and F.W. = 236.25, the calculated density is 1.304 g/cm^3. The reflection conditions of:

0kl: k = 2n

h0l: l = 2n

hk0: h = 2n

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of 20 ± 1°C to a maximum 2q value of 55.0°. A total of 540 oscillation images were collected. A sweep of data was done using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./°]. The detector swing angle was -30.80°. A second sweep was performed using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./°]. The detector swing angle was -30.80°. Another sweep was performed using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./°]. The detector swing angle was -30.80°. The crystal-to-detector distance was 52.00 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 20801 reflections that were collected, 2754 were unique (R_{int} = 0.1085); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, m, for Mo-Ka radiation is 0.893 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.568 to 0.972. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2754 observed reflections and 244 variable parameters and converged (largest parameter shift was 0.11 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \, ||Fo| - |Fc|| \, / \, S \, |Fo| = 0.1134$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S \, w(Fo^2)^2]^{1/2} = 0.3222$$

The standard deviation of an observation of unit weight[4] was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.31 and -0.47 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Df' and Df'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(3) Least Squares function minimized: (SHELXL97)

$$\Sigma w(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

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

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

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) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(10) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS OF 4ah

A. Crystal Data

Empirical Formula	$C_{15}H_9FN_2$
Formula Weight	236.25
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.500 X 0.480 X 0.320 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Lattice Parameters	a = 25.51(2) Å b = 13.881(8) Å c = 6.794(4) Å V = 2406(2) Å^3

Space Group	Pbca (#61)
Z value	8
D_{calc}	1.304 g/cm^3
F_{000}	976.00
m(MoKa)	0.893 cm^{-1}

B. Intensity Measurements

Diffractometer	SCX mini
Radiation	MoKa (l = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	20.0^{o}C
Detector Aperture	75 mm (diameter)
Data Images	540 exposures
w oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
w oscillation Range	-120.0 - 60.0^{o}

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Detector Swing Angle	-30.80^{o}
w oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
Detector Position	52.00 mm
Pixel Size	0.146 mm
$2q_{max}$	55.0^{o}
No. of Reflections Measured	Total: 20801 Unique: 2754 (R_{int} = 0.1085)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.568 - 0.972)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.1402 \cdot P)^2 + 2.7382 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2754
No. Variables	244
Reflection/Parameter Ratio	11.29

Residuals: R1 (I>2.00s(I))	0.1134
Residuals: R (All reflections)	0.1474
Residuals: wR2 (All reflections)	0.3222
Goodness of Fit Indicator	1.049
Max Shift/Error in Final Cycle	0.106
Maximum peak in Final Diff. Map	0.31 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.47 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
F1	0.6249(2)	0.1661(5)	0.359(1)	14.3(2)
N1	0.4230(2)	0.1030(3)	0.6720(5)	3.93(7)
N2	0.3927(2)	0.0216(4)	0.9700(6)	6.4(1)
C1	0.3917(2)	0.1332(3)	0.5145(6)	3.87(8)
C2	0.4241(2)	0.1620(3)	0.3718(7)	4.14(9)
C3	0.4769(2)	0.1502(3)	0.4327(6)	3.90(8)
C4	0.5262(3)	0.1671(5)	0.3497(8)	4.6(1)
C5	0.5713(2)	0.1458(3)	0.4486(7)	4.65(9)
C6	0.5679(2)	0.1054(3)	0.6349(8)	4.76(9)
C7	0.5204(2)	0.0878(3)	0.7243(8)	4.48(9)
C8	0.4758(2)	0.1118(3)	0.6202(6)	3.86(8)
C9	0.4064(2)	0.0592(3)	0.8323(6)	4.47(8)
C10	0.3342(2)	0.1283(3)	0.5279(7)	3.98(8)
C11	0.3056(3)	0.0909(4)	0.3747(9)	5.0(1)
C12	0.2515(2)	0.0855(4)	0.384(1)	6.0(2)
C13	0.2252(2)	0.1191(4)	0.544(1)	6.0(2)
C14	0.2534(3)	0.1578(5)	0.697(1)	6.1(2)
C15	0.3074(2)	0.1620(4)	0.6898(8)	5.0(1)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso}/B_{eq} involving hydrogen atoms

atom	x	y	z	B_{eq}
H2	0.413(2)	0.188(3)	0.258(8)	5(1)
H4	0.528(2)	0.191(4)	0.233(9)	6(2)
H6	0.600(2)	0.087(3)	0.694(8)	6(2)
H7	0.521(2)	0.057(3)	0.869(8)	6(2)
H11	0.324(2)	0.071(4)	0.268(8)	6(2)
H12	0.230(2)	0.057(4)	0.280(8)	7(2)
H13	0.186(2)	0.118(4)	0.54(1)	10(2)
H14	0.237(3)	0.187(6)	0.81(2)	12(3)
H15	0.327(2)	0.188(4)	0.806(7)	7(2)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F1	0.119(4)	0.201(5)	0.223(6)	-0.013(4)	0.041(4)	-0.003(5)
N1	0.048(2)	0.053(2)	0.049(2)	-0.001(2)	0.001(2)	0.004(2)
N2	0.072(3)	0.112(4)	0.060(3)	-0.010(3)	0.004(2)	0.015(3)
C1	0.050(2)	0.041(2)	0.056(3)	0.003(2)	-0.004(2)	0.001(2)
C2	0.059(3)	0.046(2)	0.052(3)	0.003(2)	-0.002(2)	0.005(2)
C3	0.053(3)	0.042(2)	0.053(3)	-0.001(2)	0.005(2)	0.001(2)
C4	0.061(3)	0.056(3)	0.058(3)	-0.001(2)	0.012(3)	0.001(3)
C5	0.050(3)	0.049(2)	0.078(3)	-0.002(2)	0.012(2)	-0.002(2)
C6	0.050(3)	0.055(3)	0.076(3)	0.000(2)	-0.003(3)	-0.001(3)
C7	0.054(3)	0.055(3)	0.061(3)	-0.002(2)	-0.004(2)	0.006(2)
C8	0.048(3)	0.044(2)	0.054(3)	-0.002(2)	0.002(2)	0.001(2)
C9	0.054(3)	0.066(3)	0.049(3)	-0.008(2)	0.001(2)	0.002(2)
C10	0.050(3)	0.039(2)	0.062(3)	0.003(2)	-0.004(2)	-0.001(2)
C11	0.057(3)	0.064(3)	0.070(4)	0.004(2)	-0.010(3)	-0.004(3)
C12	0.059(3)	0.073(4)	0.095(4)	0.003(3)	-0.021(3)	-0.005(3)
C13	0.054(3)	0.064(3)	0.109(5)	0.003(3)	-0.007(3)	0.005(3)
C14	0.065(4)	0.071(3)	0.095(4)	0.007(3)	0.016(3)	-0.005(3)
C15	0.053(3)	0.060(3)	0.077(4)	0.002(2)	0.004(3)	-0.013(3)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
F1	C5	1.523(7)	N1	C1	1.400(5)
N1	C8	1.397(5)	N1	C9	1.317(6)
N2	C9	1.128(6)	C1	C2	1.336(6)
C1	C10	1.471(6)	C2	C3	1.420(7)
C3	C4	1.397(8)	C3	C8	1.381(6)
C4	C5	1.365(8)	C5	C6	1.387(7)
C6	C7	1.377(7)	C7	C8	1.381(6)
C10	C11	1.373(7)	C10	C15	1.378(7)
C11	C12	1.383(8)	C12	C13	1.362(9)
C13	C14	1.372(9)	C14	C15	1.380(8)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H2	0.90(5)	C4	H4	0.86(6)
C6	H6	0.96(5)	C7	H7	1.07(5)
C11	H11	0.90(5)	C12	H12	0.97(6)
C13	H13	1.00(5)	C14	H14	0.96(8)
C15	H15	1.00(5)			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	N1	C8	109.4(3)	C1	N1	C9	125.9(4)
C8	N1	C9	124.0(4)	N1	C1	C2	106.9(4)
N1	C1	C10	120.6(4)	C2	C1	C10	132.5(4)
C1	C2	C3	110.0(4)	C2	C3	C4	135.8(5)
C2	C3	C8	107.1(4)	C4	C3	C8	117.1(4)
C3	C4	C5	121.5(5)	F1	C5	C4	121.4(5)
F1	C5	C6	119.7(5)	C4	C5	C6	118.9(5)
C5	C6	C7	122.0(5)	C6	C7	C8	117.2(5)
N1	C8	C3	106.7(4)	N1	C8	C7	130.1(4)
C3	C8	C7	123.2(4)	N1	C9	N2	179.2(5)
C1	C10	C11	120.0(4)	C1	C10	C15	121.9(4)
C11	C10	C15	118.0(4)	C10	C11	C12	121.2(6)
C11	C12	C13	120.5(6)	C12	C13	C14	118.8(6)
C13	C14	C15	120.8(6)	C10	C15	C14	120.6(5)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	C2	H2	124(3)	C3	C2	H2	126(3)
C3	C4	H4	120(3)	C5	C4	H4	119(3)
C5	C6	H6	116(3)	C7	C6	H6	122(3)
C6	C7	H7	118(3)	C8	C7	H7	125(3)
C10	C11	H11	117(3)	C12	C11	H11	122(3)
C11	C12	H12	123(3)	C13	C12	H12	116(3)
C12	C13	H13	119(4)	C14	C13	H13	122(4)
C13	C14	H14	122(4)	C15	C14	H14	117(4)
C10	C15	H15	121(3)	C14	C15	H15	119(3)

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C1	N1	C8	C3	1.7(4)	C1	N1	C8	C7	-177.5(4)
C8	N1	C1	C2	-1.4(4)	C8	N1	C1	C10	178.0(3)
C9	N1	C1	C2	-171.8(4)	C9	N1	C1	C10	7.6(6)
C9	N1	C8	C3	172.3(4)	C9	N1	C8	C7	-6.9(6)
N1	C1	C2	C3	0.6(5)	N1	C1	C10	C11	-133.7(4)
N1	C1	C10	C15	47.5(5)	C2	C1	C10	C11	45.6(6)
C2	C1	C10	C15	-133.2(5)	C10	C1	C2	C3	-178.7(4)
C1	C2	C3	C4	180.0(4)	C1	C2	C3	C8	0.4(5)
C2	C3	C4	C5	-178.9(4)	C2	C3	C8	N1	-1.3(4)
C2	C3	C8	C7	178.0(3)	C4	C3	C8	N1	179.1(4)
C4	C3	C8	C7	-1.7(6)	C8	C3	C4	C5	0.6(7)
C3	C4	C5	F1	-178.8(4)	C3	C4	C5	C6	0.8(7)
F1	C5	C6	C7	178.3(4)	C4	C5	C6	C7	-1.3(7)
C5	C6	C7	C8	0.3(7)	C6	C7	C8	N1	-179.7(4)
C6	C7	C8	C3	1.2(6)	C1	C10	C11	C12	-180.0(4)
C1	C10	C15	C14	179.0(4)	C11	C10	C15	C14	0.2(6)
C15	C10	C11	C12	-1.1(7)	C10	C11	C12	C13	1.2(8)
C11	C12	C13	C14	-0.4(8)	C12	C13	C14	C15	-0.5(9)
C13	C14	C15	C10	0.6(8)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
N1	C4	3.538(7)	N1	C15	3.065(6)
N2	C1	3.461(6)	N2	C8	3.423(6)
N2	C15	3.487(7)	C2	C7	3.583(7)
C2	C9	3.468(7)	C2	C11	3.180(8)
C3	C6	2.767(7)	C3	C9	3.494(6)
C4	C7	2.777(8)	C5	C8	2.742(6)
C7	C9	3.027(7)	C9	C10	2.930(6)
C9	C15	3.058(7)	C10	C13	2.785(7)
C11	C14	2.726(9)	C12	C15	2.736(8)

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H4	2.63(5)	F1	H6	2.60(6)
N1	H2	3.06(5)	N1	H7	2.90(5)
N1	H15	2.88(5)	N2	H7	3.37(5)
N2	H15	3.07(5)	C1	H11	2.56(5)
C1	H15	2.69(5)	C2	H4	2.85(5)
C2	H11	2.94(5)	C3	H7	3.42(5)
C4	H2	2.96(5)	C4	H6	3.21(5)
C5	H7	3.37(5)	C6	H4	3.14(6)
C8	H2	3.12(5)	C8	H4	3.15(6)
C8	H6	3.24(5)	C9	H7	2.93(5)
C9	H15	2.72(5)	C10	H2	2.85(5)
C10	H12	3.29(5)	C10	H14	3.24(8)
C11	H2	3.16(5)	C11	H13	3.28(5)
C11	H15	3.27(5)	C12	H14	3.24(9)
C13	H11	3.20(5)	C13	H15	3.28(5)
C14	H12	3.21(6)	C15	H11	3.16(5)
C15	H13	3.31(5)	H2	H4	2.95(7)
H2	H11	2.81(7)	H6	H7	2.39(7)
H11	H12	2.39(7)	H12	H13	2.28(8)
H13	H14	2.42(10)	H14	H15	2.29(8)

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
F1	N2[1]	3.463(8)	F1	C9[1]	3.481(8)
N1	C2[2]	3.534(6)	N1	C5[1]	3.552(6)
N1	C6[1]	3.572(6)	N2	F1[1]	3.463(8)
N2	C2[3]	3.449(7)	N2	C6[4]	3.365(7)
N2	C7[4]	3.396(7)	C2	N1[5]	3.534(6)
C2	N2[6]	3.449(7)	C3	C7[1]	3.472(6)
C4	C7[5]	3.511(8)	C5	N1[1]	3.552(6)
C5	C9[1]	3.474(6)	C6	N1[1]	3.572(6)
C6	N2[4]	3.365(7)	C7	N2[4]	3.396(7)
C7	C3[1]	3.472(6)	C7	C4[2]	3.511(8)
C9	F1[1]	3.481(8)	C9	C5[1]	3.474(6)

Symmetry Operators:

(1) -X+1,-Y,-Z+1

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

(3) X,Y,Z+1

(4) -X+1,-Y,-Z+2

(5) X,-Y+1/2,Z+1/2-1

(6) X,Y,Z-1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
F1	H12[1]	3.22(5)	F1	H13[2]	3.44(6)
F1	H13[1]	3.22(6)	N1	H2[3]	2.96(5)
N2	H2[4]	3.08(5)	N2	H6[5]	2.74(5)
N2	H7[5]	2.70(5)	N2	H11[4]	2.77(5)
N2	H12[6]	3.56(5)	N2	H13[6]	2.84(5)
C1	H2[3]	3.03(5)	C1	H6[7]	3.37(5)
C1	H15[8]	3.30(5)	C2	H2[3]	3.36(5)
C2	H6[7]	3.54(5)	C2	H15[8]	3.27(5)
C3	H2[3]	3.54(5)	C3	H4[3]	3.28(6)
C3	H7[7]	3.53(5)	C4	H4[3]	3.27(6)
C5	H4[3]	3.18(6)	C6	H4[3]	3.08(5)
C7	H4[3]	3.08(5)	C7	H7[5]	3.57(5)
C8	H2[3]	3.34(5)	C8	H4[3]	3.15(5)
C9	H2[4]	3.41(5)	C9	H2[3]	3.54(5)
C9	H7[5]	3.19(5)	C10	H12[6]	3.50(5)
C10	H15[8]	2.96(5)	C11	H6[7]	3.47(5)
C11	H12[6]	3.56(6)	C11	H14[8]	3.58(7)
C11	H15[8]	3.14(5)	C12	H12[6]	3.38(6)
C12	H14[8]	3.22(8)	C13	H11[6]	3.29(5)
C13	H12[6]	3.14(5)	C13	H14[8]	3.15(8)
C14	H12[6]	3.07(5)	C14	H14[8]	3.43(8)

C15	H2[3]	3.44(5)	C15	H12[6]	3.25(5)
C15	H15[8]	3.37(5)	H2	N1[8]	2.96(5)
H2	N2[9]	3.08(5)	H2	C1[8]	3.03(5)
H2	C2[8]	3.36(5)	H2	C3[8]	3.54(5)
H2	C8[8]	3.34(5)	H2	C9[9]	3.41(5)
H2	C9[8]	3.54(5)	H2	C15[8]	3.44(5)
H2	H15[8]	2.81(7)	H4	C3[8]	3.28(6)
H4	C4[8]	3.27(6)	H4	C5[8]	3.18(6)
H4	C6[8]	3.08(5)	H4	C7[8]	3.08(5)
H4	C8[8]	3.15(5)	H4	H7[9]	3.10(8)
H6	N2[5]	2.74(5)	H6	C1[7]	3.37(5)
H6	C2[7]	3.54(5)	H6	C11[7]	3.47(5)
H6	H11[7]	2.94(7)	H6	H13[10]	2.86(8)
H7	N2[5]	2.70(5)	H7	C3[7]	3.53(5)
H7	C7[5]	3.57(5)	H7	C9[5]	3.19(5)
H7	H4[4]	3.10(8)	H7	H7[5]	2.60(7)

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H11	$N2^{9}$	2.77(5)	H11	$C13^{11}$	3.29(5)
H11	$H6^{7}$	2.94(7)	H11	$H13^{11}$	3.05(8)
H11	$H15^{9}$	3.54(7)	H11	$H15^{8}$	3.35(7)
H12	$F1^{12}$	3.22(5)	H12	$N2^{11}$	3.56(5)
H12	$C10^{11}$	3.50(5)	H12	$C11^{11}$	3.56(6)
H12	$C12^{11}$	3.38(6)	H12	$C13^{11}$	3.14(5)
H12	$C14^{11}$	3.07(5)	H12	$C15^{11}$	3.25(5)
H12	$H14^{8}$	3.56(9)	H12	$H14^{11}$	3.49(9)
H13	$F1^{13}$	3.44(6)	H13	$F1^{12}$	3.22(6)
H13	$N2^{11}$	2.84(5)	H13	$H6^{14}$	2.86(8)
H13	$H11^{6}$	3.05(8)	H13	$H14^{8}$	3.40(10)
H14	$C11^{3}$	3.58(7)	H14	$C12^{3}$	3.22(8)
H14	$C13^{3}$	3.15(8)	H14	$C14^{3}$	3.43(8)
H14	$H12^{3}$	3.56(9)	H14	$H12^{6}$	3.49(9)
H14	$H13^{3}$	3.40(10)	H15	$C1^{3}$	3.30(5)
H15	$C2^{3}$	3.27(5)	H15	$C10^{3}$	2.96(5)
H15	$C11^{3}$	3.14(5)	H15	$C15^{3}$	3.37(5)
H15	$H2^{3}$	2.81(7)	H15	$H11^{4}$	3.54(7)
H15	$H11^{3}$	3.35(7)			

Symmetry Operators:

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

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

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

(4) X,Y,Z+1

(5) -X+1,-Y,-Z+2

(6) -X+1/2,-Y,Z+1/2

(7) -X+1,-Y,-Z+1

(8) X,-Y+1/2,Z+1/2-1

(9) X,Y,Z-1

(10) X+1/2,Y,-Z+1/2+1

(11) -X+1/2,-Y,Z+1/2-1

(12) X+1/2-1,Y,-Z+1/2

(13) X+1/2-1,-Y+1/2,-Z+1

(14) X+1/2-1,Y,-Z+1/2+1

Experimental data for 4ch

Data Collection

A colorless chip crystal of $C_{16}H_{12}N_2$ having approximate dimensions of 0.580 x 0.370 x 0.300 mm was mounted on a glass fiber. All measurements were made on a Rigaku SCX mini diffractometer using graphite monochromated Mo-Ka radiation.

The crystal-to-detector distance was 52.00 mm.

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

a = 25.555(2) Å
b = 14.126(1) Å
c = 6.8724(6) Å
V = 2480.8(4) $Å^3$

For Z = 8 and F.W. = 232.27, the calculated density is 1.249 g/cm^3. The reflection conditions of:

0kl: k = 2n
h0l: l = 2n
hk0: h = 2n

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of 20 ± 1°C to a maximum 2q value of 55.0°. A total of 540 oscillation images were collected. A sweep of data was done using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 8.0 [sec./°]. The detector swing angle was -30.80°. A second sweep was performed using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 8.0 [sec./°]. The detector swing angle was -30.80°. Another sweep was performed using w oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 8.0 [sec./°]. The detector swing angle was -30.80°. The crystal-to-detector distance was 52.00 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 23443 reflections that were collected, 2840 were unique (R_{int} = 0.0589); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, m, for Mo-Ka radiation is 0.746 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.766 to 0.978. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2840 observed reflections and 271 variable parameters and converged (largest parameter shift was 2.13 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \, ||Fo| - |Fc|| \, / \, S \, |Fo| = 0.0467$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2) / S \, w(Fo^2)^2]^{1/2} = 0.1541$$

The standard deviation of an observation of unit weight[4] was 0.92. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.11 and -0.20 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Df' and Df'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

References

(1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000.J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.

(2) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(3) Least Squares function minimized: (SHELXL97)

$S w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

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

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

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) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(10) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS OF 4ch

A. Crystal Data

Empirical Formula	$C_{16}H_{12}N_2$
Formula Weight	232.27
Crystal Color, Habit	colorless, chip
Crystal Dimensions	0.580 X 0.370 X 0.300 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Lattice Parameters	a = 25.555(2) Å b = 14.126(1) Å c = 6.8724(6) Å V = 2480.8(4) $Å^3$
Space Group	Pbca (#61)
Z value	8
D_{calc}	1.249 g/cm^3
F_{000}	984.00
m(MoKa)	0.746 cm^{-1}

B. Intensity Measurements

Diffractometer	SCX mini
Radiation	MoKa (l = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	20.0^{o}C
Detector Aperture	75 mm (diameter)
Data Images	540 exposures
w oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
w oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
w oscillation Range	-120.0 - 60.0^{o}
Exposure Rate	8.0 sec./^{o}
Detector Swing Angle	-30.80^{o}
Detector Position	52.00 mm
Pixel Size	0.146 mm
$2q_{max}$	55.0^{o}
No. of Reflections Measured	Total: 23443 Unique: 2840 (R_{int} = 0.0589)

Corrections	Lorentz-polarization Absorption (trans. factors: 0.766 - 0.978)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.1000 \cdot P)^2 + 0.0000 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2840
No. Variables	271
Reflection/Parameter Ratio	10.48
Residuals: R1 (I>2.00s(I))	0.0467
Residuals: R (All reflections)	0.0926
Residuals: wR2 (All reflections)	0.1541
Goodness of Fit Indicator	0.920
Max Shift/Error in Final Cycle	2.128
Maximum peak in Final Diff. Map	$0.11\ e^-/Å^3$
Minimum peak in Final Diff. Map	$-0.20\ e^-/Å^3$

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

atom	x	y	z	B_{eq}
N1	0.57703(5)	0.10275(9)	0.3268(2)	3.84(3)
N2	0.60721(6)	0.0217(2)	0.0299(3)	6.41(5)
C1	0.60847(6)	0.1329(1)	0.4853(3)	3.80(4)
C2	0.57606(7)	0.1624(2)	0.6299(3)	4.04(4)
C3	0.52275(6)	0.1501(1)	0.5674(3)	3.85(4)
C4	0.47390(7)	0.1672(2)	0.6527(3)	4.29(4)
C5	0.42877(7)	0.1454(2)	0.5512(3)	4.53(4)
C6	0.43227(8)	0.1053(2)	0.3650(4)	4.59(4)
C7	0.47961(8)	0.0879(2)	0.2770(4)	4.33(4)
C8	0.52387(6)	0.1120(1)	0.3795(3)	3.69(4)
C9	0.3761(1)	0.1662(3)	0.6392(6)	6.22(8)
C10	0.66575(6)	0.1284(1)	0.4723(3)	4.03(4)
C11	0.69422(8)	0.0903(2)	0.6279(4)	4.93(5)
C12	0.74805(9)	0.0855(2)	0.6174(4)	6.01(6)
C13	0.7744(1)	0.1187(2)	0.4549(5)	6.19(6)
C14	0.74675(9)	0.1577(2)	0.3035(5)	5.90(5)
C15	0.69250(8)	0.1620(2)	0.3111(4)	5.04(5)
C16	0.59357(7)	0.0590(2)	0.1688(3)	4.26(4)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso}/B_{eq} involving hydrogen atoms

atom	x	y	z	B_{eq}
H2	0.5893(8)	0.190(2)	0.760(3)	4.9(5)
H4	0.4723(7)	0.192(2)	0.784(3)	5.2(5)
H6	0.3987(7)	0.089(2)	0.292(3)	5.1(5)
H7	0.4808(8)	0.061(2)	0.161(3)	5.3(5)
H9A	0.366(2)	0.230(3)	0.64(1)	17(2)
H9B	0.355(2)	0.123(4)	0.619(7)	14(2)
H9C	0.375(2)	0.162(5)	0.773(4)	12(2)
H11	0.6744(8)	0.067(2)	0.751(3)	6.3(6)
H12	0.7694(8)	0.058(2)	0.727(3)	7.1(6)
H13	0.8140(9)	0.115(2)	0.442(4)	9.8(7)
H14	0.7656(9)	0.180(2)	0.197(3)	7.4(7)
H15	0.6732(8)	0.189(2)	0.205(3)	5.8(5)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N1	0.0414(8)	0.0537(9)	0.0507(9)	0.0029(7)	0.0003(7)	-0.0019(7)
N2	0.068(2)	0.113(2)	0.063(1)	0.009(1)	0.0034(9)	-0.014(1)
C1	0.045(1)	0.0425(9)	0.057(1)	-0.0020(7)	-0.0036(8)	0.0031(8)
C2	0.048(1)	0.048(1)	0.057(2)	-0.0003(8)	-0.0002(9)	-0.0034(9)
C3	0.048(1)	0.0406(9)	0.057(1)	0.0004(8)	0.0014(8)	-0.0008(8)
C4	0.053(2)	0.052(1)	0.058(2)	0.0038(8)	0.009(1)	-0.0016(9)
C5	0.045(1)	0.048(1)	0.080(2)	0.0014(8)	0.008(1)	0.006(1)
C6	0.044(1)	0.054(1)	0.076(2)	-0.0001(9)	-0.005(1)	0.001(1)
C7	0.048(1)	0.056(2)	0.060(2)	0.0007(9)	-0.006(1)	-0.006(1)
C8	0.043(1)	0.0429(9)	0.055(1)	0.0028(7)	0.0017(8)	0.0005(8)
C9	0.051(2)	0.085(2)	0.100(3)	0.007(2)	0.018(2)	0.005(2)
C10	0.043(1)	0.0414(9)	0.069(2)	-0.0022(7)	-0.0025(9)	0.0008(9)
C11	0.052(2)	0.064(2)	0.071(2)	-0.0057(9)	-0.013(1)	0.004(1)
C12	0.052(2)	0.077(2)	0.099(2)	-0.005(1)	-0.021(2)	0.006(2)
C13	0.046(2)	0.066(2)	0.124(2)	-0.004(1)	-0.007(2)	-0.005(2)
C14	0.054(2)	0.071(2)	0.098(2)	-0.007(1)	0.016(2)	0.007(2)
C15	0.050(2)	0.064(2)	0.077(2)	-0.0008(9)	0.004(1)	0.013(2)
C16	0.047(1)	0.067(2)	0.048(1)	0.0052(9)	0.0012(9)	-0.0012(9)

The general temperature factor expression: $\exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
N1	C1	1.419(3)	N1	C8	1.412(2)
N1	C16	1.319(3)	N2	C16	1.145(3)
C1	C2	1.360(3)	C1	C10	1.468(3)
C2	C3	1.439(3)	C3	C4	1.401(3)
C3	C8	1.399(3)	C4	C5	1.383(3)
C5	C6	1.402(3)	C5	C9	1.504(4)
C6	C7	1.374(3)	C7	C8	1.375(3)
C10	C11	1.401(3)	C10	C15	1.385(3)
C11	C12	1.379(3)	C12	C13	1.386(4)
C13	C14	1.372(4)	C14	C15	1.389(3)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H2	1.031(16)	C4	H4	0.967(18)
C6	H6	1.019(18)	C7	H7	0.89(2)
C9	H9A	0.94(5)	C9	H9B	0.84(5)
C9	H9C	0.92(3)	C11	H11	1.040(18)
C12	H12	1.01(2)	C13	H13	1.02(3)
C14	H14	0.93(2)	C15	H15	0.961(18)

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	N1	C8	108.68(13)	C1	N1	C16	126.23(14)
C8	N1	C16	124.24(15)	N1	C1	C2	107.96(14)
N1	C1	C10	120.36(15)	C2	C1	C10	131.68(17)
C1	C2	C3	108.73(16)	C2	C3	C4	134.24(18)
C2	C3	C8	107.63(15)	C4	C3	C8	118.13(16)
C3	C4	C5	119.55(19)	C4	C5	C6	119.84(18)
C4	C5	C9	119.9(3)	C6	C5	C9	120.2(3)
C5	C6	C7	121.98(19)	C6	C7	C8	117.1(2)
N1	C8	C3	106.96(14)	N1	C8	C7	129.61(17)
C3	C8	C7	123.41(16)	C1	C10	C11	119.20(17)
C1	C10	C15	121.74(17)	C11	C10	C15	119.06(17)
C10	C11	C12	119.8(2)	C11	C12	C13	120.7(3)
C12	C13	C14	119.8(3)	C13	C14	C15	120.2(3)
C10	C15	C14	120.5(2)	N1	C16	N2	178.8(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	C2	H2	123.2(11)	C3	C2	H2	128.0(11)
C3	C4	H4	119.3(11)	C5	C4	H4	121.1(11)
C5	C6	H6	119.0(11)	C7	C6	H6	119.0(11)
C6	C7	H7	120.3(13)	C8	C7	H7	122.6(13)
C5	C9	H9A	117(3)	C5	C9	H9B	112(4)
C5	C9	H9C	115(3)	H9A	C9	H9B	121(4)
H9A	C9	H9C	92(6)	H9B	C9	H9C	96(5)
C10	C11	H11	119.4(11)	C12	C11	H11	120.8(11)
C11	C12	H12	121.4(12)	C13	C12	H12	118.0(12)
C12	C13	H13	122.5(14)	C14	C13	H13	117.7(14)
C13	C14	H14	117.9(14)	C15	C14	H14	122.0(14)
C10	C15	H15	119.4(12)	C14	C15	H15	120.1(12)

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C1	N1	C8	C3	1.64(16)	C1	N1	C8	C7	-176.95(14)
C8	N1	C1	C2	-1.67(16)	C8	N1	C1	C10	178.39(12)
C16	N1	C1	C2	-171.45(15)	C16	N1	C1	C10	8.6(3)
C16	N1	C8	C3	171.67(14)	C16	N1	C8	C7	-6.9(3)
N1	C1	C2	C3	1.03(18)	N1	C1	C10	C11	-133.46(15)
N1	C1	C10	C15	47.3(2)	C2	C1	C10	C11	46.6(3)
C2	C1	C10	C15	-132.63(19)	C10	C1	C2	C3	-179.04(15)
C1	C2	C3	C4	179.63(16)	C1	C2	C3	C8	-0.02(18)
C2	C3	C4	C5	-178.95(16)	C2	C3	C8	N1	-1.00(17)
C2	C3	C8	C7	177.70(14)	C4	C3	C8	N1	179.28(14)
C4	C3	C8	C7	-2.0(3)	C8	C3	C4	C5	0.7(3)
C3	C4	C5	C6	0.8(3)	C3	C4	C5	C9	-178.19(14)
C4	C5	C6	C7	-1.1(3)	C9	C5	C6	C7	177.9(3)
C5	C6	C7	C8	-0.2(3)	C6	C7	C8	N1	-179.86(16)
C6	C7	C8	C3	1.7(3)	C1	C10	C11	C12	179.83(14)
C1	C10	C15	C14	179.43(15)	C11	C10	C15	C14	0.2(3)
C15	C10	C11	C12	-0.9(3)	C10	C11	C12	C13	0.5(3)
C11	C12	C13	C14	0.6(4)	C12	C13	C14	C15	-1.3(4)
C13	C14	C15	C10	0.9(4)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
N1	C4	3.577(3)	N1	C15	3.069(3)
N2	C1	3.502(3)	N2	C8	3.454(3)
N2	C15	3.523(3)	C2	C11	3.187(3)
C2	C16	3.518(3)	C3	C6	2.771(3)
C3	C16	3.526(3)	C4	C7	2.818(3)
C5	C8	2.743(3)	C7	C16	3.033(3)
C10	C13	2.782(3)	C10	C16	2.952(3)
C11	C14	2.771(4)	C12	C15	2.759(4)
C15	C16	3.077(3)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
N1	H2	3.236(16)	N1	H7	2.78(2)
N1	H15	2.87(2)	N2	H7	3.40(2)
N2	H15	3.14(2)	C1	H11	2.655(19)
C1	H15	2.661(18)	C2	H4	2.885(18)
C2	H11	2.97(2)	C3	H7	3.25(2)
C4	H2	3.057(19)	C4	H6	3.324(18)
C4	H9A	2.90(4)	C4	H9B	3.12(4)
C4	H9C	2.66(4)	C5	H7	3.23(2)
C6	H4	3.292(18)	C6	H9A	3.11(5)
C6	H9B	2.66(5)	C6	H9C	3.26(3)
C8	H2	3.293(17)	C8	H4	3.277(18)
C8	H6	3.270(17)	C9	H4	2.677(18)
C9	H6	2.685(19)	C10	H2	2.910(17)
C10	H12	3.33(2)	C10	H14	3.26(2)
C11	H2	3.160(18)	C11	H13	3.34(3)
C11	H15	3.270(17)	C12	H14	3.22(2)
C13	H11	3.347(19)	C13	H15	3.260(19)
C14	H12	3.284(19)	C15	H11	3.341(18)
C15	H13	3.30(3)	C16	H7	2.88(2)
C16	H15	2.75(2)	H2	H4	2.99(3)
H2	H11	2.78(3)	H4	H9A	2.94(5)
H4	H9B	3.36(5)	H4	H9C	2.53(4)
H6	H7	2.32(3)	H6	H9A	3.24(6)
H6	H9B	2.56(5)	H6	H9C	3.51(4)
H11	H12	2.44(3)	H12	H13	2.41(3)
H13	H14	2.28(3)	H14	H15	2.37(3)

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
N1	C2[1]	3.582(3)	N2	C2[2]	3.485(3)
N2	C6[3]	3.406(3)	N2	C7[3]	3.431(3)
N2	C9[4]	3.521(5)	C2	N1[5]	3.582(3)
C2	N2[6]	3.485(3)	C3	C7[4]	3.529(3)
C4	C7[5]	3.565(3)	C5	C16[4]	3.517(3)
C6	N2[3]	3.406(3)	C7	N2[3]	3.431(3)
C7	C3[4]	3.529(3)	C7	C4[1]	3.565(3)
C9	N2[4]	3.521(5)	C9	C16[4]	3.530(5)
C16	C5[4]	3.517(3)	C16	C9[4]	3.530(5)

Symmetry Operators:

(1) X,-Y+1/2,Z+1/2-1
(2) X,Y,Z-1
(3) -X+1,-Y,-Z
(4) -X+1,-Y,-Z+1
(5) X,-Y+1/2,Z+1/2
(6) X,Y,Z+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
N1	H2[1]	2.981(18)	N2	H2[2]	3.049(17)
N2	H6[3]	2.715(19)	N2	H7[3]	2.85(2)
N2	H9B[4]	3.31(5)	N2	H9C[4]	2.96(6)
N2	H11[2]	2.651(19)	N2	H13[5]	2.86(3)
C1	H2[1]	2.985(17)	C1	H6[4]	3.494(19)
C1	H15[6]	3.37(2)	C2	H2[1]	3.307(17)
C2	H15[6]	3.29(2)	C3	H2[1]	3.531(17)
C3	H4[1]	3.228(19)	C3	H7[4]	3.52(2)
C4	H4[1]	3.220(19)	C5	H4[1]	3.142(19)
C6	H4[1]	3.09(2)	C6	H9A[1]	3.26(5)
C7	H4[1]	3.11(2)	C8	H2[1]	3.363(18)
C8	H4[1]	3.13(2)	C9	H9C[1]	3.50(5)
C9	H12[7]	3.26(2)	C9	H13[8]	3.52(3)
C9	H13[7]	3.37(3)	C10	H2[1]	3.540(18)
C10	H12[5]	3.54(3)	C10	H15[6]	3.04(2)
C11	H6[4]	3.516(18)	C11	H12[5]	3.58(2)
C11	H15[6]	3.21(2)	C12	H9B[9]	3.31(5)
C12	H9C[9]	3.50(4)	C12	H12[5]	3.39(2)
C12	H14[6]	3.38(3)	C13	H9A[10]	3.24(4)
C13	H9B[9]	3.58(5)	C13	H9C[9]	3.24(4)
C13	H11[5]	3.25(2)	C13	H12[5]	3.15(3)
C13	H14[6]	3.30(3)	C14	H9A[10]	3.45(4)
C14	H12[5]	3.12(3)	C14	H14[6]	3.57(2)
C15	H2[1]	3.384(18)	C15	H12[5]	3.31(3)
C15	H15[6]	3.462(18)	C16	H2[2]	3.366(16)
C16	H7[3]	3.40(2)	C16	H9B[4]	3.24(6)
C16	H9C[4]	3.25(6)	C16	H11[2]	3.538(18)
H2	N1[6]	2.981(18)	H2	N2[11]	3.049(17)
H2	C1[6]	2.985(17)	H2	C2[6]	3.307(17)
H2	C3[6]	3.531(17)	H2	C8[6]	3.363(18)
H2	C10[6]	3.540(18)	H2	C15[6]	3.384(18)
H2	C16[11]	3.366(16)	H2	H15[6]	2.77(3)
H4	C3[6]	3.228(19)	H4	C4[6]	3.220(19)
H4	C5[6]	3.142(19)	H4	C6[6]	3.09(2)
H4	C7[6]	3.11(2)	H4	C8[6]	3.13(2)
H4	H7[11]	3.20(3)	H4	H7[6]	3.60(3)
H6	N2[3]	2.715(19)	H6	C1[4]	3.494(19)

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H6	C11[4]	3.516(18)	H6	H9A[1]	2.88(5)
H6	H9C[1]	3.57(7)	H6	H11[4]	2.91(3)
H6	H13[12]	2.72(3)	H7	N2[3]	2.85(2)
H7	C3[4]	3.52(2)	H7	C16[3]	3.40(2)
H7	H4[2]	3.20(3)	H7	H4[1]	3.60(3)
H7	H7[3]	2.96(3)	H9A	C6[6]	3.26(5)
H9A	C13[8]	3.24(4)	H9A	C14[8]	3.45(4)
H9A	H6[6]	2.88(5)	H9A	H9A[1]	3.48(9)
H9A	H9A[6]	3.48(9)	H9A	H9C[1]	2.98(7)
H9A	H12[7]	3.57(5)	H9A	H13[8]	2.62(5)
H9A	H13[7]	3.54(6)	H9A	H14[8]	3.06(5)
H9A	H14[12]	3.53(6)	H9B	N2[4]	3.31(5)
H9B	C12[7]	3.31(5)	H9B	C13[7]	3.58(5)
H9B	C16[4]	3.24(6)	H9B	H12[7]	2.59(5)
H9B	H13[7]	3.19(6)	H9B	H14[12]	3.24(5)
H9C	N2[4]	2.96(6)	H9C	C9[6]	3.50(5)
H9C	C12[7]	3.50(4)	H9C	C13[7]	3.24(4)
H9C	C16[4]	3.25(6)	H9C	H6[6]	3.57(7)
H9C	H9A[6]	2.98(7)	H9C	H12[7]	3.07(5)
H9C	H13[7]	2.59(4)	H9C	H14[8]	3.58(6)
H11	N2[11]	2.651(19)	H11	C13[13]	3.25(2)
H11	C16[11]	3.538(18)	H11	H6[4]	2.91(3)
H11	H13[13]	2.90(3)	H11	H15[11]	3.56(3)
H11	H15[6]	3.46(3)	H12	C9[9]	3.26(2)
H12	C10[13]	3.54(3)	H12	C11[13]	3.58(2)
H12	C12[13]	3.39(2)	H12	C13[13]	3.15(3)
H12	C14[13]	3.12(3)	H12	C15[13]	3.31(3)
H12	H9A[9]	3.57(5)	H12	H9B[9]	2.59(5)
H12	H9C[9]	3.07(5)	H12	H13[13]	3.57(3)
H12	H14[13]	3.49(4)	H13	N2[13]	2.86(3)
H13	C9[10]	3.52(3)	H13	C9[9]	3.37(3)
H13	H6[14]	2.72(3)	H13	H9A[10]	2.62(5)
H13	H9A[9]	3.54(6)	H13	H9B[9]	3.19(6)
H13	H9C[9]	2.59(4)	H13	H11[5]	2.90(3)
H13	H12[5]	3.57(3)	H13	H14[6]	3.60(4)
H14	C12[1]	3.38(3)	H14	C13[1]	3.30(3)
H14	C14[1]	3.57(2)	H14	H9A[10]	3.06(5)

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H14	H9A[14]	3.53(6)	H14	H9B[14]	3.24(5)
H14	H9C[10]	3.58(6)	H14	H12[5]	3.49(4)
H14	H13[1]	3.60(4)	H15	C1[1]	3.37(2)
H15	C2[1]	3.29(2)	H15	C10[1]	3.04(2)
H15	C11[1]	3.21(2)	H15	C15[1]	3.462(18)
H15	H2[1]	2.77(3)	H15	H11[2]	3.56(3)
H15	H11[1]	3.46(3)			

Symmetry Operators:

(1) X,-Y+1/2,Z+1/2-1
(2) X,Y,Z-1
(3) -X+1,-Y,-Z
(4) -X+1,-Y,-Z+1
(5) -X+1/2+1,-Y,Z+1/2-1
(6) X,-Y+1/2,Z+1/2
(7) X+1/2-1,Y,-Z+1/2+1
(8) X+1/2-1,-Y+1/2,-Z+1
(9) X+1/2,Y,-Z+1/2+1
(10) X+1/2,-Y+1/2,-Z+1
(11) X,Y,Z+1
(12) X+1/2-1,Y,-Z+1/2
(13) -X+1/2+1,-Y,Z+1/2
(14) X+1/2,Y,-Z+1/2