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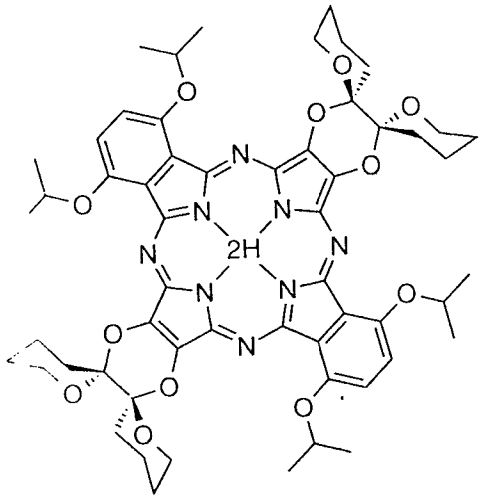

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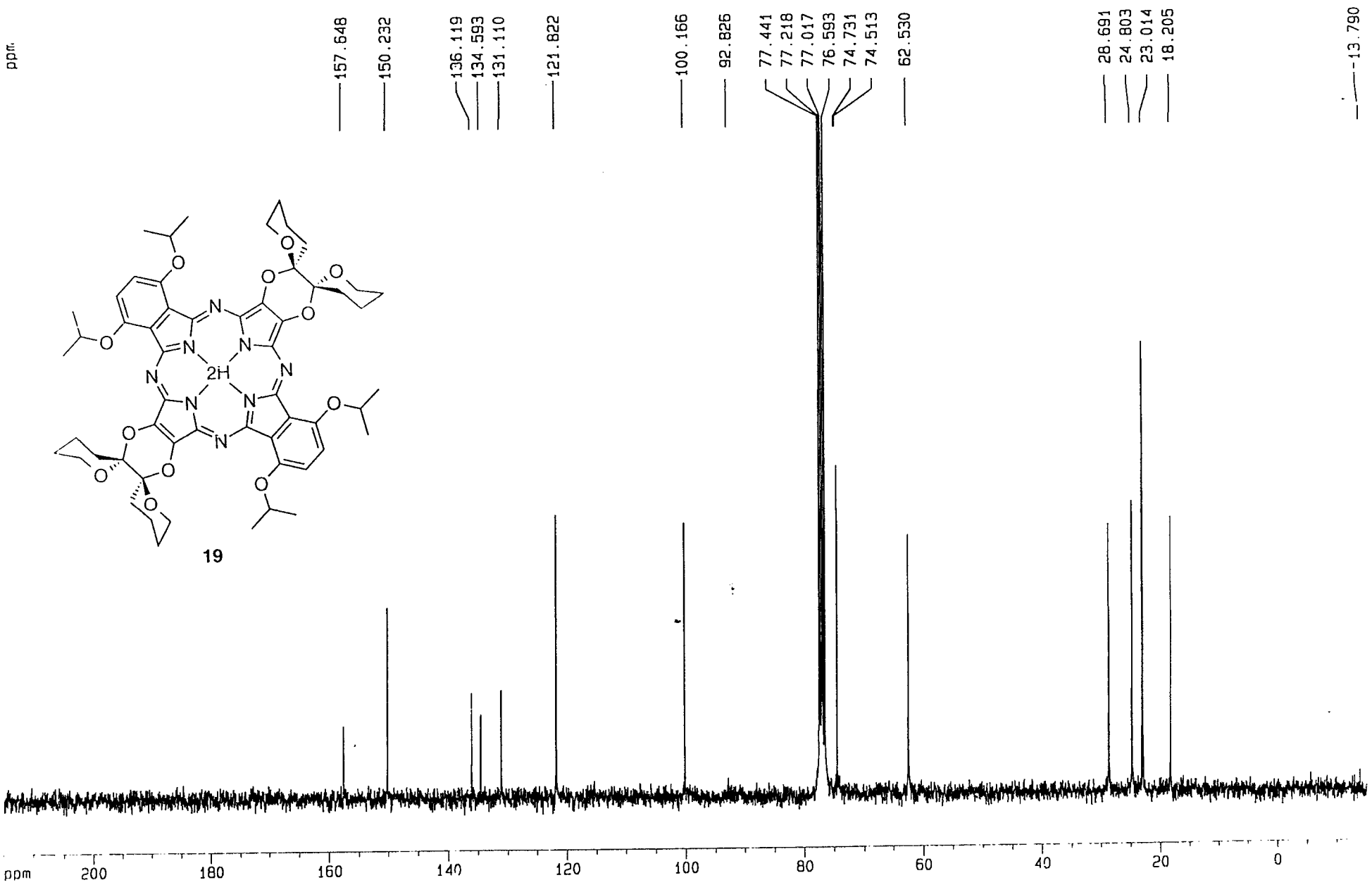
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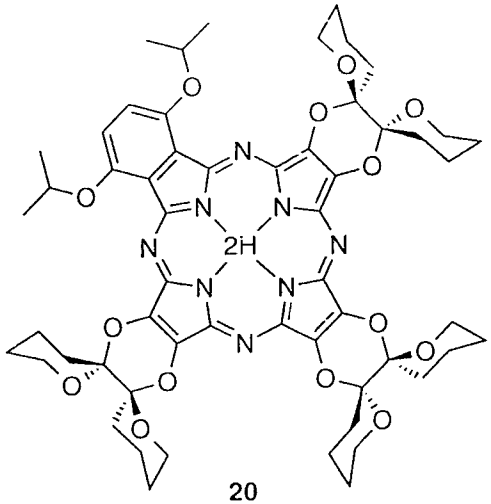

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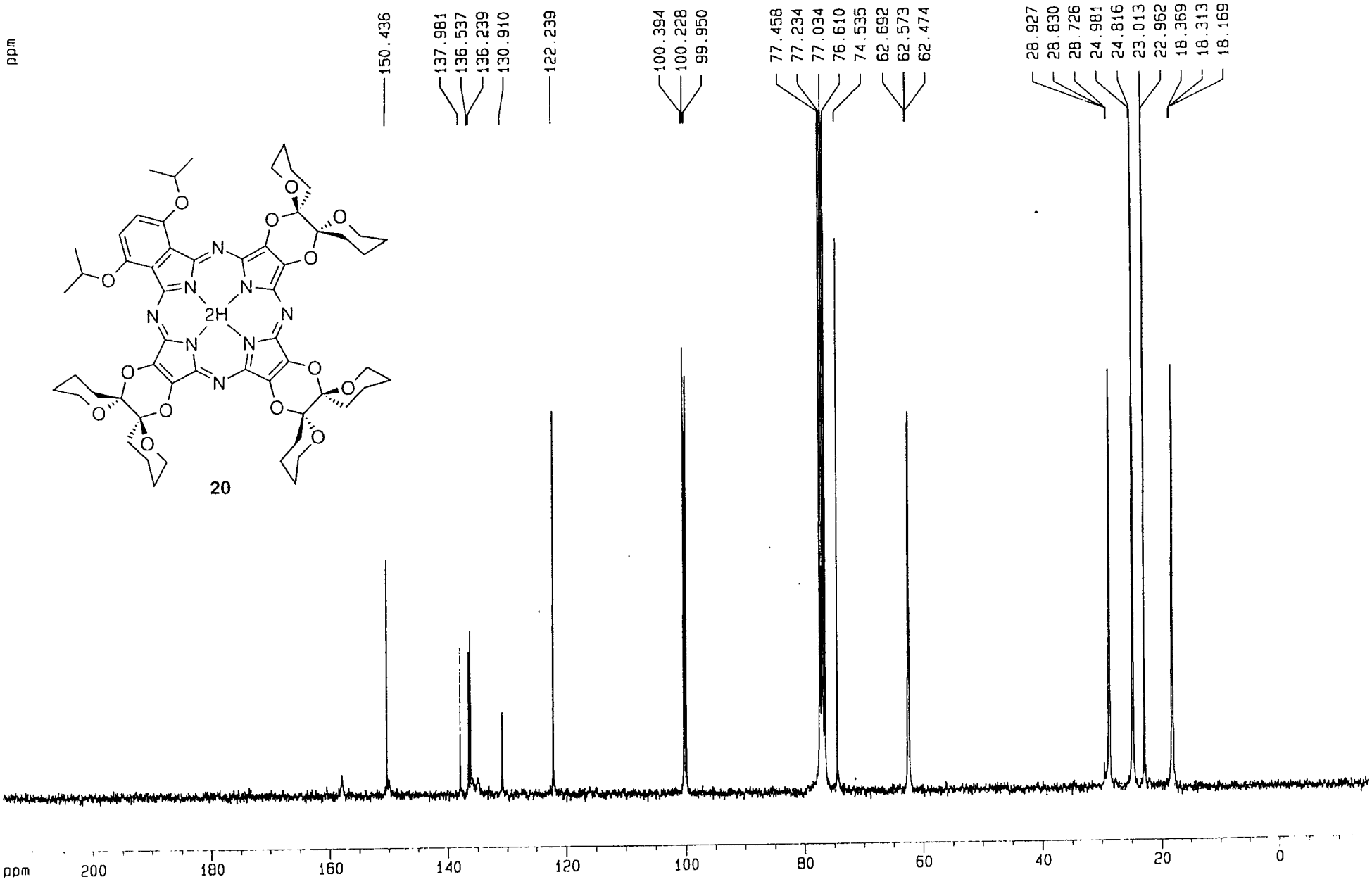
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Crystal Structure Report for Trans-Propyl Porphyrazine (17)

Experimental

Data Collection

A dark, opaque, columnar crystal of $O_4N_8C_{48}H_{62}$ having approximate dimensions of 0.7 x 0.1 x 0.1 mm was mounted using oil. (Paratone-N, Exxon) on a glass fiber. All measurements were made on an Enraf-Nonius CAD4 diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $19.7 < 2\theta < 23.1°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 9.599(3) Å

b = 20.902(3) Å

c = 22.349(3) Å

V = 4484(1) Å^3

For Z = 4 and F.W. = 815.07, the calculated density is 1.21 g/cm^3. The systematic absences of:

h00: h $\neq$ 2n

0k0: k $\neq$ 2n

00l: l $\neq$ 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -120 $\pm$ 1°C using the ω-θ scan technique to a maximum 2θ value of 49.9°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.30° with a take-off angle of 2.8°. Scans of (1.00 + 0.35 tan θ)° were made at a variable speed of 3.0-16.0°/min (in omega). Moving-crystal moving counter background measurements were made by scanning an additional 25% above and below the scan range. The counter aperture consisted of a variable horizontal slit with a width ranging from 2.0 to 2.5 mm and a vertical slit set to 2.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21 cm. For intense reflections an attenuator was automatically inserted in front of the detector.

Data Reduction

A total of 4439 reflections was collected. The intensities of three representative reflection were measured after every 90 minutes of X-ray exposure time. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. An analytical absorption correction was applied which resulted in transmission factors ranging from 0.99 to 0.99. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 2.80793e-07).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. Owing to the Paucity of data, the non-hydrogen atoms were refined isotropically. Hydrogen atoms on the carbon atoms were included in idealized positions but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 1559 observed reflections ($I > 3.00\sigma(I)$) and 242 variable parameters and converged (largest parameter shift was 0.32 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.095$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)} = 0.073$$

The standard deviation of an observation of unit weight[4] was 2.39. The weighting scheme was based on counting statistics. Plots of $\Sigma w(|Fo| - |Fc|)^2$ versus $|Fo|$, 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 0.65 and -0.41 $e^-/\text{Å}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SIR92: Altomare, A., Burla, M.C., Camalli, M., Cascarano, M., Giacovazzo, C., Guagliardi, A., Polidori, G. (1994). J. Appl. Cryst., in preparation.

(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: $\Sigma w(|Fo| - |Fc|)^2$

where $w = \frac{1}{\sigma^2(Fo)} = \frac{4Fo^2}{\sigma^2(Fo^2)}$

$\sigma^2(Fo^2) = \frac{S^2(C+R^2B)+(pFo^2)^2}{Lp^2}$

S = Scan rate

C = Total integrated peak count

R = Ratio of scan time to background counting time

B = Total background count

Lp = Lorentz-polarization factor

p = p-factor

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

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = 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 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$O_4N_8C_{48}H_{62}$
Formula Weight	815.07
Crystal Color, Habit	dark, columnar
Crystal Dimensions	0.70 X 0.10 X 0.10 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2θ range)	25 (19.7 - 23.1°)
Omega Scan Peak Width	
at Half-height	0.30°
Lattice Parameters	a = 9.599(3) Å b = 20.902(3) Å c = 22.349(3) Å
	V = 4484(1) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.207 g/cm^3
F_{000}	1752.00
μ(MoKα)	0.78 cm^{-1}

B. Intensity Measurements

Diffractometer	CAD4
Radiation	MoKα (λ = 0.71069 Å)

	graphite monochromated
Attenuator	Zr foil (factor = 22.25)
Take-off Angle	2.8°
Detector Aperture	2.0 - 2.5 mm horizontal 2.0 mm vertical
Crystal to Detector Distance	21 mm
Temperature	-120.0°C
Scan Type	ω-θ
Scan Rate	3.0-16.0°/min (in ω) (up to 0 scans)
Scan Width	$(1.00 + 0.35 \tan\theta)°$
$2\theta_{max}$	49.9°
No. of Reflections Measured	Total: 4439
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9879 - 0.9917) Secondary Extinction (coefficient: 2.80793e-07)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$\frac{1}{\sigma^2(Fo)} = \frac{4Fo^2}{\sigma^2(Fo^2)}$
p-factor	0.0000
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I>3.00\sigma(I)$)	1559
No. Variables	242
Reflection/Parameter Ratio	6.44
Residuals: R; Rw	0.095 ; 0.073

Goodness of Fit Indicator	2.39
Max Shift/Error in Final Cycle	0.32
Maximum peak in Final Diff. Map	0.65 e^-/A^3
Minimum peak in Final Diff. Map	-0.41 e^-/A^3

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

atom	x	y	z	B_{eq}
O(1)	1.251(1)	0.3919(5)	0.5694(5)	2.0(3)
O(2)	0.772(1)	0.5393(5)	0.5542(5)	2.4(3)
O(3)	0.498(1)	0.3037(5)	0.1525(5)	1.9(3)
O(4)	0.999(2)	0.1714(6)	0.1604(6)	4.2(4)
N(1)	0.934(1)	0.3993(6)	0.4296(6)	1.5(3)
N(2)	0.737(2)	0.4702(6)	0.4411(6)	1.6(3)
N(3)	0.722(1)	0.4098(6)	0.3488(6)	1.2(3)
N(4)	0.622(2)	0.3705(6)	0.2555(6)	1.4(3)
N(5)	0.842(2)	0.3199(6)	0.2799(6)	1.0(3)
N(6)	1.041(2)	0.2480(7)	0.2676(7)	2.0(4)
N(7)	1.059(1)	0.3086(6)	0.3593(5)	0.6(3)
N(8)	1.155(2)	0.3463(6)	0.4531(6)	1.5(3)
C(1)	1.051(2)	0.3869(8)	0.4675(7)	1.2(4)
C(2)	1.044(2)	0.4244(8)	0.5214(8)	1.4(4)
C(3)	0.918(2)	0.4592(8)	0.5169(7)	1.1(4)
C(4)	0.857(2)	0.4429(8)	0.4586(7)	0.8(4)
C(5)	0.679(2)	0.4531(8)	0.3915(7)	1.1(4)
C(6)	0.549(2)	0.4796(8)	0.3708(7)	1.0(4)
C(7)	0.514(2)	0.4537(8)	0.3181(7)	1.0(4)
C(8)	0.625(2)	0.4086(8)	0.3043(7)	1.3(4)
C(9)	0.726(2)	0.3270(8)	0.2466(8)	1.4(4)
C(10)	0.718(2)	0.2831(7)	0.1975(7)	0.6(3)
C(11)	0.840(2)	0.2481(8)	0.1967(8)	1.9(4)
C(12)	0.915(2)	0.2718(8)	0.2510(8)	1.8(4)

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

atom	x	y	z	B_{eq}
C(13)	1.102(2)	0.2651(8)	0.3161(8)	1.8(4)
C(14)	1.234(2)	0.2357(8)	0.3386(8)	1.7(4)
C(15)	1.264(2)	0.2654(7)	0.3910(7)	0.8(4)
C(16)	1.159(2)	0.3108(8)	0.4040(7)	1.3(4)
C(17)	1.136(2)	0.4298(8)	0.5705(8)	1.6(4)
C(18)	1.096(2)	0.4702(8)	0.6165(8)	1.5(4)
C(19)	0.974(2)	0.5075(8)	0.6116(8)	1.5(4)
C(20)	0.888(2)	0.5051(9)	0.5609(8)	1.8(4)
C(21)	1.354(2)	0.3990(9)	0.6184(9)	3.2(5)
C(22)	1.310(2)	0.3642(10)	0.6717(9)	3.7(5)
C(23)	1.487(2)	0.3737(9)	0.5889(9)	3.8(5)
C(24)	0.724(2)	0.5836(9)	0.6010(8)	2.7(5)
C(25)	0.650(3)	0.548(1)	0.647(1)	6.3(7)
C(26)	0.640(3)	0.6313(9)	0.5652(9)	4.5(6)
C(27)	0.465(2)	0.5282(8)	0.4052(7)	1.5(4)
C(28)	0.408(2)	0.5044(9)	0.4646(8)	2.3(4)
C(29)	0.306(2)	0.5515(9)	0.4949(8)	2.1(4)
C(30)	0.388(2)	0.4628(8)	0.2763(7)	1.4(4)
C(31)	0.265(2)	0.4233(8)	0.2971(8)	2.6(5)
C(32)	0.134(2)	0.4370(8)	0.2569(8)	2.6(5)
C(33)	0.619(2)	0.2673(8)	0.1539(8)	1.8(4)
C(34)	0.643(2)	0.2192(8)	0.1117(8)	1.7(4)
C(35)	0.769(2)	0.1853(8)	0.1138(8)	2.2(4)
C(36)	0.874(2)	0.2017(9)	0.1556(8)	2.2(4)

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

atom	x	y	z	B_{eq}
C(37)	0.389(2)	0.2883(9)	0.1101(9)	2.6(5)
C(38)	0.254(2)	0.3185(10)	0.1373(9)	4.1(6)
C(39)	0.423(2)	0.3136(10)	0.0478(9)	4.1(5)
C(40)	1.031(3)	0.120(1)	0.113(1)	5.5(7)
C(41)	1.006(3)	0.066(1)	0.149(1)	10.0(9)
C(42)	1.176(3)	0.125(1)	0.096(1)	6.9(8)
C(43)	1.301(3)	0.176(1)	0.308(1)	5.6(7)
C(44)	1.403(4)	0.198(2)	0.273(1)	10(1)
C(45)	1.468(3)	0.131(1)	0.238(1)	9.4(9)
C(46)	1.391(2)	0.2521(8)	0.4293(8)	1.3(4)
C(47)	1.514(2)	0.2983(9)	0.4104(8)	2.9(5)
C(48)	1.643(2)	0.2848(8)	0.4492(8)	2.3(4)
H(1)	1.1522	0.4732	0.6514	1.8224
H(2)	0.9489	0.5346	0.6437	2.1900
H(3)	1.3653	0.1430	0.6273	3.8775
H(4)	1.2243	0.3804	0.6849	4.1620
H(5)	1.3021	0.3201	0.6622	4.1620
H(6)	1.3793	0.3697	0.7018	4.1620
H(7)	1.4715	0.3306	0.5755	4.9870
H(8)	1.5100	0.3993	0.5551	4.9870
H(9)	1.5610	0.3740	0.6167	4.9870
H(10)	0.8025	0.6037	0.6184	3.3880
H(11)	0.6219	0.5759	0.6775	7.5176
H(12)	0.5718	0.5272	0.6296	7.5176

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

atom	x	y	z	B_{eq}
H(13)	0.7112	0.5160	0.6631	7.5176
H(14)	0.6021	0.6622	0.5916	6.2723
H(15)	0.7002	0.6520	0.5373	6.2723
H(16)	0.5681	0.6099	0.5445	6.2723
H(17)	0.5260	0.5635	0.4131	1.8910
H(18)	0.3911	0.5418	0.3807	1.8910
H(19)	0.3593	0.4656	0.4572	2.8364
H(20)	0.4833	0.4968	0.4909	2.8364
H(21)	0.3541	0.5900	0.5038	2.6865
H(22)	0.2306	0.5598	0.4690	2.6865
H(23)	0.2732	0.5327	0.5310	2.6865
H(24)	0.3631	0.5069	0.2778	1.8307
H(25)	0.4126	0.4509	0.2375	1.8307
H(26)	0.2893	0.3794	0.2946	3.1673
H(27)	0.2445	0.4339	0.3376	3.1673
H(28)	0.1098	0.4810	0.2596	3.7125
H(29)	0.1546	0.4265	0.2166	3.7125
H(30)	0.0582	0.1116	0.2705	3.7125
H(31)	0.5759	0.2093	0.0813	2.2762
H(32)	0.7850	0.1509	0.0870	2.9236
H(33)	0.3783	0.2440	0.1083	3.2604
H(34)	0.2635	0.3629	0.1406	4.9043
H(35)	0.1771	0.3078	0.1126	4.9043
H(36)	0.2397	0.3002	0.1762	4.9043

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

atom	x	y	z	B_{eq}
H(37)	0.5075	0.2943	0.0349	4.8118
H(38)	0.3499	0.3022	0.0214	4.8118
H(39)	0.4335	0.3585	0.0491	4.8118
H(40)	0.9698	0.1228	0.0813	6.2369
H(41)	0.9140	0.0661	0.1632	12.3164
H(42)	1.0220	0.0287	0.1252	12.3164
H(43)	1.0695	0.0653	0.1821	12.3164
H(44)	1.2001	0.0875	0.0747	8.6860
H(45)	1.1868	0.1613	0.0712	8.6860
H(46)	1.2326	0.1290	0.1306	8.6860
H(47)	1.3672	0.2592	0.4703	1.8971
H(48)	1.4186	0.2086	0.4245	1.8971
H(49)	1.5368	0.2908	0.3699	3.7970
H(50)	1.4861	0.3411	0.4160	3.7970
H(51)	1.6702	0.2115	0.4449	2.7118
H(52)	1.7170	0.3120	0.4369	2.7118
H(53)	1.6216	0.2932	0.4901	2.7118
H(54)	1.3336	0.1469	0.3372	7.2240
H(55)	1.2332	0.1551	0.2833	7.2240
H(56)	1.3773	0.2285	0.2452	13.2393
H(57)	1.4793	0.2162	0.2978	13.2393
H(58)	1.3941	0.1111	0.2163	11.2695
H(59)	1.5417	0.1409	0.2120	11.2695
H(60)	1.5008	0.1005	0.2675	11.2695

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

atom	x	y	z	B_{eq}

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

Table 2. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(17)	1.36(2)	O(1)	C(21)	1.48(2)
O(2)	C(20)	1.34(2)	O(2)	C(24)	1.47(2)
O(3)	C(33)	1.39(2)	O(3)	C(37)	1.45(2)
O(4)	C(36)	1.36(2)	O(4)	C(40)	1.53(2)
N(1)	C(1)	1.43(2)	N(1)	C(4)	1.34(2)
N(2)	C(4)	1.34(2)	N(2)	C(5)	1.29(2)
N(3)	C(5)	1.38(2)	N(3)	C(8)	1.37(2)
N(4)	C(8)	1.35(2)	N(4)	C(9)	1.36(2)
N(5)	C(9)	1.35(2)	N(5)	C(12)	1.38(2)
N(6)	C(12)	1.36(2)	N(6)	C(13)	1.28(2)
N(7)	C(13)	1.39(2)	N(7)	C(16)	1.39(2)
N(8)	C(1)	1.35(2)	N(8)	C(16)	1.32(2)
C(1)	C(2)	1.44(2)	C(2)	C(3)	1.41(2)
C(2)	C(17)	1.42(2)	C(3)	C(4)	1.47(2)
C(3)	C(20)	1.40(2)	C(5)	C(6)	1.44(2)
C(6)	C(7)	1.34(2)	C(6)	C(27)	1.50(2)
C(7)	C(8)	1.45(2)	C(7)	C(30)	1.54(2)
C(9)	C(10)	1.43(2)	C(10)	C(11)	1.38(2)
C(10)	C(33)	1.40(2)	C(11)	C(12)	1.49(2)
C(11)	C(36)	1.37(2)	C(13)	C(14)	1.50(2)
C(14)	C(15)	1.36(2)	C(14)	C(43)	1.56(3)
C(15)	C(16)	1.42(2)	C(15)	C(46)	1.51(2)
C(17)	C(18)	1.38(2)	C(18)	C(19)	1.41(2)
C(19)	C(20)	1.40(2)	C(21)	C(22)	1.46(2)

Table 2. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(21)	C(23)	1.53(3)	C(24)	C(25)	1.45(3)
C(24)	C(26)	1.51(3)	C(27)	C(28)	1.52(2)
C(28)	C(29)	1.54(2)	C(30)	C(31)	1.51(2)
C(31)	C(32)	1.57(3)	C(33)	C(34)	1.40(2)
C(34)	C(35)	1.41(2)	C(35)	C(36)	1.41(2)
C(37)	C(38)	1.56(3)	C(37)	C(39)	1.52(2)
C(40)	C(41)	1.42(3)	C(40)	C(42)	1.45(3)
C(43)	C(44)	1.34(3)	C(44)	C(45)	1.73(4)
C(46)	C(47)	1.58(2)	C(47)	C(48)	1.54(3)

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
C(18)	H(1)	0.95	C(19)	H(2)	0.95
C(21)	H(3)	0.95	C(22)	H(4)	0.94
C(22)	H(5)	0.95	C(22)	H(6)	0.95
C(23)	H(7)	0.96	C(23)	H(8)	0.95
C(23)	H(9)	0.94	C(24)	H(10)	0.95
C(25)	H(11)	0.95	C(25)	H(12)	0.94
C(25)	H(13)	0.96	C(26)	H(14)	0.95
C(26)	H(15)	0.95	C(26)	H(16)	0.94
C(27)	H(17)	0.96	C(27)	H(18)	0.94
C(28)	H(19)	0.95	C(28)	H(20)	0.94
C(29)	H(21)	0.95	C(29)	H(22)	0.94
C(29)	H(23)	0.95	C(30)	H(24)	0.95
C(30)	H(25)	0.93	C(31)	H(26)	0.95
C(31)	H(27)	0.95	C(32)	H(28)	0.95
C(32)	H(29)	0.95	C(32)	H(30)	0.95
C(34)	H(31)	0.95	C(35)	H(32)	0.95
C(37)	H(33)	0.93	C(38)	H(34)	0.93
C(38)	H(35)	0.95	C(38)	H(36)	0.96
C(39)	H(37)	0.95	C(39)	H(38)	0.95
C(39)	H(39)	0.94	C(40)	H(40)	0.92
C(41)	H(41)	0.94	C(41)	H(42)	0.95
C(41)	H(43)	0.96	C(42)	H(44)	0.95
C(42)	H(45)	0.95	C(42)	H(46)	0.94
C(43)	H(54)	0.94	C(43)	H(55)	0.96

Table 3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(44)	H(56)	0.93	C(44)	H(57)	0.99
C(45)	H(58)	0.95	C(45)	H(59)	0.94
C(45)	H(60)	0.97	C(46)	H(47)	0.96
C(46)	H(48)	0.95	C(47)	H(49)	0.94
C(47)	H(50)	0.94	C(48)	H(51)	0.95
C(48)	H(52)	0.95	C(48)	H(53)	0.95

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(17)	O(1)	C(21)	118(1)	C(20)	O(2)	C(24)	121(1)
C(33)	O(3)	C(37)	119(1)	C(36)	O(4)	C(40)	116(1)
C(1)	N(1)	C(4)	105(1)	C(4)	N(2)	C(5)	120(1)
C(5)	N(3)	C(8)	107(1)	C(8)	N(4)	C(9)	119(1)
C(9)	N(5)	C(12)	103(1)	C(12)	N(6)	C(13)	122(1)
C(13)	N(7)	C(16)	108(1)	C(1)	N(8)	C(16)	124(1)
N(1)	C(1)	N(8)	123(1)	N(1)	C(1)	C(2)	111(1)
N(8)	C(1)	C(2)	125(1)	C(1)	C(2)	C(3)	105(1)
C(1)	C(2)	C(17)	131(1)	C(3)	C(2)	C(17)	123(1)
C(2)	C(3)	C(4)	106(1)	C(2)	C(3)	C(20)	118(1)
C(4)	C(3)	C(20)	134(1)	N(1)	C(4)	N(2)	128(1)
N(1)	C(4)	C(3)	111(1)	N(2)	C(4)	C(3)	120(1)
N(2)	C(5)	N(3)	130(1)	N(2)	C(5)	C(6)	122(1)
N(3)	C(5)	C(6)	107(1)	C(5)	C(6)	C(7)	109(1)
C(5)	C(6)	C(27)	123(1)	C(7)	C(6)	C(27)	126(1)
C(6)	C(7)	C(8)	105(1)	C(6)	C(7)	C(30)	132(1)
C(8)	C(7)	C(30)	121(1)	N(3)	C(8)	N(4)	127(1)
N(3)	C(8)	C(7)	109(1)	N(4)	C(8)	C(7)	122(1)
N(4)	C(9)	N(5)	126(1)	N(4)	C(9)	C(10)	119(1)
N(5)	C(9)	C(10)	113(1)	C(9)	C(10)	C(11)	107(1)
C(9)	C(10)	C(33)	136(1)	C(11)	C(10)	C(33)	116(1)
C(10)	C(11)	C(12)	102(1)	C(10)	C(11)	C(36)	125(1)
C(12)	C(11)	C(36)	131(1)	N(5)	C(12)	N(6)	125(1)
N(5)	C(12)	C(11)	112(1)	N(6)	C(12)	C(11)	121(1)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
N(6)	C'(13)	N(7)	129(1)	N(6)	C'(13)	C'(14)	123(1)
N(7)	C'(13)	C'(14)	106(1)	C'(13)	C'(14)	C'(15)	106(1)
C'(13)	C'(14)	C(43)	122(1)	C'(15)	C'(14)	C(43)	131(1)
C'(14)	C'(15)	C'(16)	109(1)	C'(14)	C'(15)	C'(46)	124(1)
C'(16)	C'(15)	C(46)	125(1)	N(7)	C'(16)	N(8)	126(1)
N(7)	C'(16)	C'(15)	108(1)	N(8)	C'(16)	C'(15)	124(1)
O(1)	C'(17)	C(2)	116(1)	O(1)	C'(17)	C'(18)	126(1)
C(2)	C'(17)	C'(18)	116(1)	C'(17)	C'(18)	C'(19)	120(1)
C'(18)	C'(19)	C'(20)	122(1)	O(2)	C'(20)	C(3)	117(1)
O(2)	C'(20)	C'(19)	124(1)	C'(3)	C'(20)	C'(19)	118(1)
O(1)	C'(21)	C'(22)	111(1)	O(1)	C'(21)	C'(23)	101(1)
C'(22)	C'(21)	C'(23)	114(1)	O(2)	C'(24)	C'(25)	109(1)
O(2)	C'(24)	C'(26)	101(1)	C'(25)	C'(24)	C'(26)	116(2)
C'(6)	C'(27)	C'(28)	114(1)	C'(27)	C'(28)	C'(29)	113(1)
C'(7)	C'(30)	C'(31)	110(1)	C'(30)	C'(31)	C'(32)	110(1)
O(3)	C'(33)	C'(10)	117(1)	O(3)	C'(33)	C'(34)	120(1)
C'(10)	C'(33)	C'(34)	122(1)	C'(33)	C'(34)	C'(35)	118(1)
C'(34)	C'(35)	C'(36)	120(1)	O(4)	C'(36)	C'(11)	118(1)
O(4)	C'(36)	C'(35)	124(1)	C'(11)	C'(36)	C'(35)	116(1)
O(3)	C'(37)	C'(38)	104(1)	O(3)	C'(37)	C'(39)	111(1)
C'(38)	C'(37)	C'(39)	113(1)	O(4)	C'(40)	C'(41)	97(1)
O(4)	C'(40)	C'(42)	108(2)	C'(41)	C'(40)	C'(42)	110(2)
C'(14)	C'(43)	C'(44)	105(2)	C'(43)	C'(44)	C'(45)	104(2)
C'(15)	C'(46)	C'(47)	109(1)	C'(46)	C'(47)	C'(48)	109(1)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(17)	C(18)	H(1)	119.7	C(19)	C(18)	H(1)	119.7
C(18)	C(19)	H(2)	119.1	C(20)	C(19)	H(2)	118.8
O(1)	C(21)	H(3)	109.3	C(22)	C(21)	H(3)	110.2
C(23)	C(21)	H(3)	109.1	C(21)	C(22)	H(4)	109.3
C(21)	C(22)	H(5)	108.8	C(21)	C(22)	H(6)	108.4
H(4)	C(22)	H(5)	110.6	H(4)	C(22)	H(6)	110.3
H(5)	C(22)	H(6)	109.3	C(21)	C(23)	H(7)	109.1
C(21)	C(23)	H(8)	109.9	C(21)	C(23)	H(9)	110.1
H(7)	C(23)	H(8)	108.5	H(7)	C(23)	H(9)	109.2
H(8)	C(23)	H(9)	110.1	O(2)	C(24)	H(10)	108.9
C(25)	C(24)	H(10)	109.3	C(26)	C(24)	H(10)	110.4
C(24)	C(25)	H(11)	109.2	C(24)	C(25)	H(12)	110.0
C(24)	C(25)	H(13)	109.4	H(11)	C(25)	H(12)	110.1
H(11)	C(25)	H(13)	108.8	H(12)	C(25)	H(13)	109.3
C(24)	C(26)	H(14)	109.0	C(24)	C(26)	H(15)	108.8
C(24)	C(26)	H(16)	109.8	H(14)	C(26)	H(15)	109.3
H(14)	C(26)	H(16)	110.3	H(15)	C(26)	H(16)	109.6
C(6)	C(27)	H(17)	107.0	C(6)	C(27)	H(18)	108.1
C(28)	C(27)	H(17)	108.1	C(28)	C(27)	H(18)	109.3
H(17)	C(27)	H(18)	109.6	C(27)	C(28)	H(19)	107.7
C(27)	C(28)	H(20)	108.7	C(29)	C(28)	H(19)	108.0
C(29)	C(28)	H(20)	108.6	H(19)	C(28)	H(20)	110.0
C(28)	C(29)	H(21)	109.1	C(28)	C(29)	H(22)	109.7
C(28)	C(29)	H(23)	108.7	H(21)	C(29)	H(22)	110.2

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle
H(21)	C(29)	H(23)	109.3
C(7)	C(30)	H(24)	107.2
C(31)	C(30)	H(24)	108.6
H(24)	C(30)	H(25)	110.7
C(30)	C(31)	H(27)	109.2
C(32)	C(31)	H(27)	109.4
C(31)	C(32)	H(28)	109.6
C(31)	C(32)	H(30)	109.3
H(28)	C(32)	H(30)	109.4
C(33)	C(34)	H(31)	121.9
C(34)	C(35)	H(32)	119.7
O(3)	C(37)	H(33)	109.0
C(39)	C(37)	H(33)	109.2
C(37)	C(38)	H(35)	108.8
H(34)	C(38)	H(35)	110.7
H(35)	C(38)	H(36)	108.3
C(37)	C(39)	H(38)	108.9
H(37)	C(39)	H(38)	109.6
H(38)	C(39)	H(39)	110.1
C(41)	C(40)	H(40)	112.0
C(40)	C(41)	H(41)	110.2
C(40)	C(41)	H(43)	110.1
H(41)	C(41)	H(43)	109.8
C(40)	C(42)	H(44)	108.2
H(22)	C(29)	H(23)	109.8
C(7)	C(30)	H(25)	109.6
C(31)	C(30)	H(25)	109.7
C(30)	C(31)	H(26)	108.7
C(32)	C(31)	H(26)	109.7
H(26)	C(31)	H(27)	109.3
C(31)	C(32)	H(29)	109.4
H(28)	C(32)	H(29)	109.6
H(29)	C(32)	H(30)	109.5
C(35)	C(34)	H(31)	119.7
C(36)	C(35)	H(32)	119.4
C(38)	C(37)	H(33)	109.3
C(37)	C(38)	H(34)	110.8
C(37)	C(38)	H(36)	108.2
H(34)	C(38)	H(36)	109.8
C(37)	C(39)	H(37)	108.2
C(37)	C(39)	H(39)	109.9
H(37)	C(39)	H(39)	109.7
O(4)	C(40)	H(40)	111.6
C(42)	C(40)	H(40)	114.5
C(40)	C(41)	H(42)	108.2
H(41)	C(41)	H(42)	110.0
H(42)	C(41)	H(43)	108.5
C(40)	C(42)	H(45)	107.8

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(40)	C(42)	H(46)	110.8	H(44)	C(42)	H(45)	109.4
H(44)	C(42)	H(46)	110.5	H(45)	C(42)	H(46)	110.1
C(14)	C(43)	H(54)	110.7	C(14)	C(43)	H(55)	109.7
C(44)	C(43)	H(54)	112.4	C(44)	C(43)	H(55)	108.1
H(54)	C(43)	H(55)	110.0	C(43)	C(44)	H(56)	116.2
C(43)	C(44)	H(57)	110.6	C(45)	C(44)	H(56)	110.1
C(45)	C(44)	H(57)	107.4	H(56)	C(44)	H(57)	108.1
C(44)	C(45)	H(58)	108.5	C(44)	C(45)	H(59)	111.4
C(44)	C(45)	H(60)	109.6	H(58)	C(45)	H(59)	110.5
H(58)	C(45)	H(60)	108.2	H(59)	C(45)	H(60)	108.6
C(15)	C(46)	H(47)	108.9	C(15)	C(46)	H(48)	109.8
C(47)	C(46)	H(47)	109.7	C(47)	C(46)	H(48)	110.0
H(47)	C(46)	H(48)	108.8	C(46)	C(47)	H(49)	109.2
C(46)	C(47)	H(50)	109.4	C(48)	C(47)	H(49)	108.9
C(48)	C(47)	H(50)	109.1	H(49)	C(47)	H(50)	110.5
C(47)	C(48)	H(51)	110.0	C(47)	C(48)	H(52)	109.3
C(47)	C(48)	H(53)	109.8	H(51)	C(48)	H(52)	109.2
H(51)	C(48)	H(53)	109.1	H(52)	C(48)	H(53)	109.0

Table 6. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(17)	C(2)	C(1)	-2(2)	O(1)	C(17)	C(2)	C(3)	177(1)
O(1)	C(17)	C(18)	C(19)	179(1)	O(2)	C(20)	C(3)	C(2)	177(1)
O(2)	C(20)	C(3)	C(4)	8(3)	O(2)	C(20)	C(19)	C(18)	179(1)
O(3)	C(33)	C(10)	C(9)	4(3)	O(3)	C(33)	C(10)	C(11)	-177(1)
O(3)	C(33)	C(34)	C(35)	177(1)	O(4)	C(36)	C(11)	C(10)	179(1)
O(4)	C(36)	C(11)	C(12)	-4(3)	O(4)	C(36)	C(35)	C(34)	-179(1)
N(1)	C(1)	N(8)	C(16)	-3(2)	N(1)	C(1)	C(2)	C(3)	1(1)
N(1)	C(1)	C(2)	C(17)	-177(1)	N(1)	C(4)	N(2)	C(5)	2(2)
N(1)	C(4)	C(3)	C(2)	2(1)	N(1)	C(4)	C(3)	C(20)	172(1)
N(2)	C(4)	N(1)	C(1)	179(1)	N(2)	C(4)	C(3)	C(2)	-178(1)
N(2)	C(4)	C(3)	C(20)	-8(3)	N(2)	C(5)	N(3)	C(8)	-179(1)
N(2)	C(5)	C(6)	C(7)	179(1)	N(2)	C(5)	C(6)	C(27)	-1(2)
N(3)	C(5)	N(2)	C(4)	-2(2)	N(3)	C(5)	C(6)	C(7)	0(1)
N(3)	C(5)	C(6)	C(27)	179(1)	N(3)	C(8)	N(4)	C(9)	0(2)
N(3)	C(8)	C(7)	C(6)	0(1)	N(3)	C(8)	C(7)	C(30)	-179(1)
N(4)	C(8)	N(3)	C(5)	-176(1)	N(4)	C(8)	C(7)	C(6)	176(1)
N(4)	C(8)	C(7)	C(30)	-2(2)	N(4)	C(9)	N(5)	C(12)	-177(1)
N(4)	C(9)	C(10)	C(11)	175(1)	N(4)	C(9)	C(10)	C(33)	-6(3)
N(5)	C(9)	N(4)	C(8)	-5(2)	N(5)	C(9)	C(10)	C(11)	-3(2)
N(5)	C(9)	C(10)	C(33)	174(1)	N(5)	C(12)	N(6)	C(13)	6(2)
N(5)	C(12)	C(11)	C(10)	-2(2)	N(5)	C(12)	C(11)	C(36)	-179(1)
N(6)	C(12)	N(5)	C(9)	179(1)	N(6)	C(12)	C(11)	C(10)	178(1)
N(6)	C(12)	C(11)	C(36)	1(3)	N(6)	C(13)	N(7)	C(16)	179(1)
N(6)	C(13)	C(14)	C(15)	-178(1)	N(6)	C(13)	C(14)	C(43)	-7(2)

Table 6. Torsion Angles (°) continued

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
N(7)	C(13)	N(6)	C(12)	-1(3)	N(7)	C(13)	C(14)	C(15)	-3(2)
N(7)	C(13)	C(14)	C(43)	169(2)	N(7)	C(16)	N(8)	C(1)	0(3)
N(7)	C(16)	C(15)	C(14)	1(2)	N(7)	C(16)	C(15)	C(46)	-179(2)
N(8)	C(1)	N(1)	C(4)	-178(2)	N(8)	C(1)	C(2)	C(3)	180(2)
N(8)	C(1)	C(2)	C(17)	1(3)	N(8)	C(16)	N(7)	C(13)	-179(2)
N(8)	C(16)	C(15)	C(14)	178(2)	N(8)	C(16)	C(15)	C(46)	-3(3)
C(1)	N(1)	C(4)	C(3)	-2(2)	C(1)	N(8)	C(16)	C(15)	-176(2)
C(1)	C(2)	C(3)	C(4)	-2(2)	C(1)	C(2)	C(3)	C(20)	-174(2
C(1)	C(2)	C(17)	C(18)	-179(2)	C(2)	C(1)	N(1)	C(4)	0(2)
C(2)	C(1)	N(8)	C(16)	178(2)	C(2)	C(3)	C(20)	C(19)	-9(3)
C(2)	C(17)	O(1)	C(21)	177(1)	C(2)	C(17)	C(18)	C(19)	-5(3)
C(3)	C(2)	C(17)	C(18)	2(3)	C(3)	C(4)	N(2)	C(5)	-176(2)
C(3)	C(20)	O(2)	C(24)	173(2)	C(3)	C(20)	C(19)	C(18)	7(3)
C(4)	N(2)	C(5)	C(6)	179(2)	C(4)	C(3)	C(2)	C(17)	177(2)
C(4)	C(3)	C(20)	C(19)	-178(2)	C(5)	N(3)	C(8)	C(7)	1(2)
C(5)	C(6)	C(7)	C(30)	179(2)	C(5)	C(6)	C(7)	C(8)	0(2)
C(6)	C(5)	N(3)	C(8)	-1(2)	C(6)	C(7)	C(30)	C(31)	-82(2)
C(6)	C(27)	C(28)	C(29)	-172(2)	C(7)	C(6)	C(27)	C(28)	114(2)
C(7)	C(8)	N(4)	C(9)	-177(2)	C(7)	C(30)	C(31)	C(32)	176(1)
C(8)	N(4)	C(9)	C(10)	175(2)	C(8)	C(7)	C(6)	C(27)	-178(2)
C(8)	C(7)	C(30)	C(31)	97(2)	C(9)	N(5)	C(12)	C(11)	1(2)
C(9)	C(10)	C(11)	C(12)	4(2)	C(9)	C(10)	C(11)	C(36)	-179(2)
C(9)	C(10)	C(33)	C(34)	-178(2)	C(10)	C(9)	N(5)	C(12)	2(2)
C(10)	C(11)	C(36)	C(35)	-5(3)	C(10)	C(33)	O(3)	C(37)	-177(1)

Table 6. Torsion Angles (°) continued

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(10)	C(33)	C(34)	C(35)	1(3)	C(11)	C(10)	C(33)	C(34)	0(3)
C(11)	C(12)	N(6)	C(13)	-176(2)	C(11)	C(36)	O(4)	C(40)	-179(2)
C(11)	C(36)	C(35)	C(34)	5(3)	C(12)	N(6)	C(13)	C(14)	174(2)
C(12)	C(11)	C(10)	C(33)	-175(1)	C(12)	C(11)	C(36)	C(35)	171(2
C(13)	N(7)	C(16)	C(15)	-3(2)	C(13)	C(14)	C(15)	C(16)	1(2)
C(13)	C(14)	C(15)	C(46)	-179(2)	C(13)	C(14)	C(43)	C(44)	100(3)
C(14)	C(13)	N(7)	C(16)	4(2)	C(14)	C(15)	C(46)	C(47)	91(2)
C(14)	C(43)	C(44)	C(45)	-177(2)	C(15)	C(14)	C(43)	C(44)	-91(3)
C(15)	C(46)	C(47)	C(48)	180(1)	C(16)	C(15)	C(14)	C(43)	-170(2)
C(16)	C(15)	C(46)	C(47)	-89(2)	C(17)	O(1)	C(21)	C(22)	81(2)
C(17)	O(1)	C(21)	C(23)	-156(2)	C(17)	C(2)	C(3)	C(20)	5(3)
C(17)	C(18)	C(19)	C(20)	1(3)	C(18)	C(17)	O(1)	C(21)	-8(3)
C(19)	C(20)	O(2)	C(24)	0(3)	C(20)	O(2)	C(24)	C(25)	-82(2)
C(20)	O(2)	C(24)	C(26)	154(2)	C(27)	C(6)	C(7)	C(30)	0(3)
C(33)	O(3)	C(37)	C(38)	158(2)	C(33)	O(3)	C(37)	C(39)	-79(2)
C(33)	C(10)	C(11)	C(36)	2(3)	C(33)	C(34)	C(35)	C(36)	-3(3)
C(34)	C(33)	O(3)	C(37)	5(2)	C(35)	C(36)	O(4)	C(40)	5(2)
C(36)	O(4)	C(40)	C(42)	140(2)	C(36)	O(4)	C(40)	C(41)	-103(2)
C(43)	C(14)	C(15)	C(46)	11(3)					

Table 7. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
N(2)	C(42)	3.44(3)	75504	N(3)	C(47)	3.37(2)	45501
N(3)	C(48)	3.53(2)	45501	N(6)	C(22)	3.50(3)	45603
N(7)	C(31)	3.41(2)	65501	N(8)	C(48)	3.51(2)	45603
C(8)	C(47)	3.47(3)	45501	C(10)	C(24)	3.57(2)	66402
C(12)	C(22)	3.47(3)	45603	C(16)	C(31)	3.51(2)	65501
C(17)	C(29)	3.46(2)	65501				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

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