Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(4)	H(1)	2.16(1)	75602	C(4)	C(5)	2.496(1)	75602
C(4)	H(4)	2.82(2)	75602	C(4)	H(2)	2.85(1)	75602
C(4)	H(7)	3.41(2)	75602	C(5)	H(5)	3.33(2)	65603
C(5)	H(1)	3.37(1)	75603	C(5)	H(6)	3.44(2)	64607
C(5)	H(8)	3.49(2)	65607	C(6)	H(1)	3.06(1)	45505
C(6)	H(4)	3.32(2)	65607	C(6)	H(5)	3.34(2)	65603
C(6)	H(2)	3.36(1)	65603	C(6)	H(6)	3.38(2)	45505
C(6)	H(8)	3.44(2)	65607	C(6)	H(7)	3.51(2)	4
H(1)	H(1)	2.61(3)	75603	H(1)	H(4)	2.71(2)	75603
H(1)	H(5)	2.86(2)	54505	H(1)	H(8)	2.91(2)	54505
H(1)	H(6)	2.97(2)	75602	H(1)	H(2)	3.23(2)	75602
H(2)	H(5)	2.72(3)	65603	H(2)	H(4)	2.97(2)	4
H(2)	H(7)	3.34(3)	4	H(2)	H(8)	3.43(2)	65603
H(4)	H(8)	2.68(2)	65607	H(5)	H(7)	2.72(3)	65603
H(5)	H(6)	2.76(2)	45505	H(5)	H(5)	2.99(3)	65603
H(5)	H(7)	3.25(3)	4	H(5)	H(6)	3.49(2)	65606
H(6)	H(6)	2.28(3)	75602	H(6)	H(7)	2.55(2)	64607
H(6)	H(6)	3.40(3)	64607	H(7)	H(7)	3.57(4)	64607
H(8)	H(8)	2.78(3)	65607				

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.

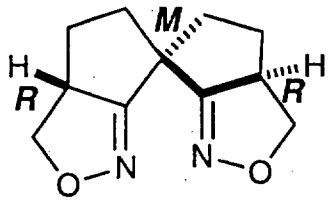
For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and 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.

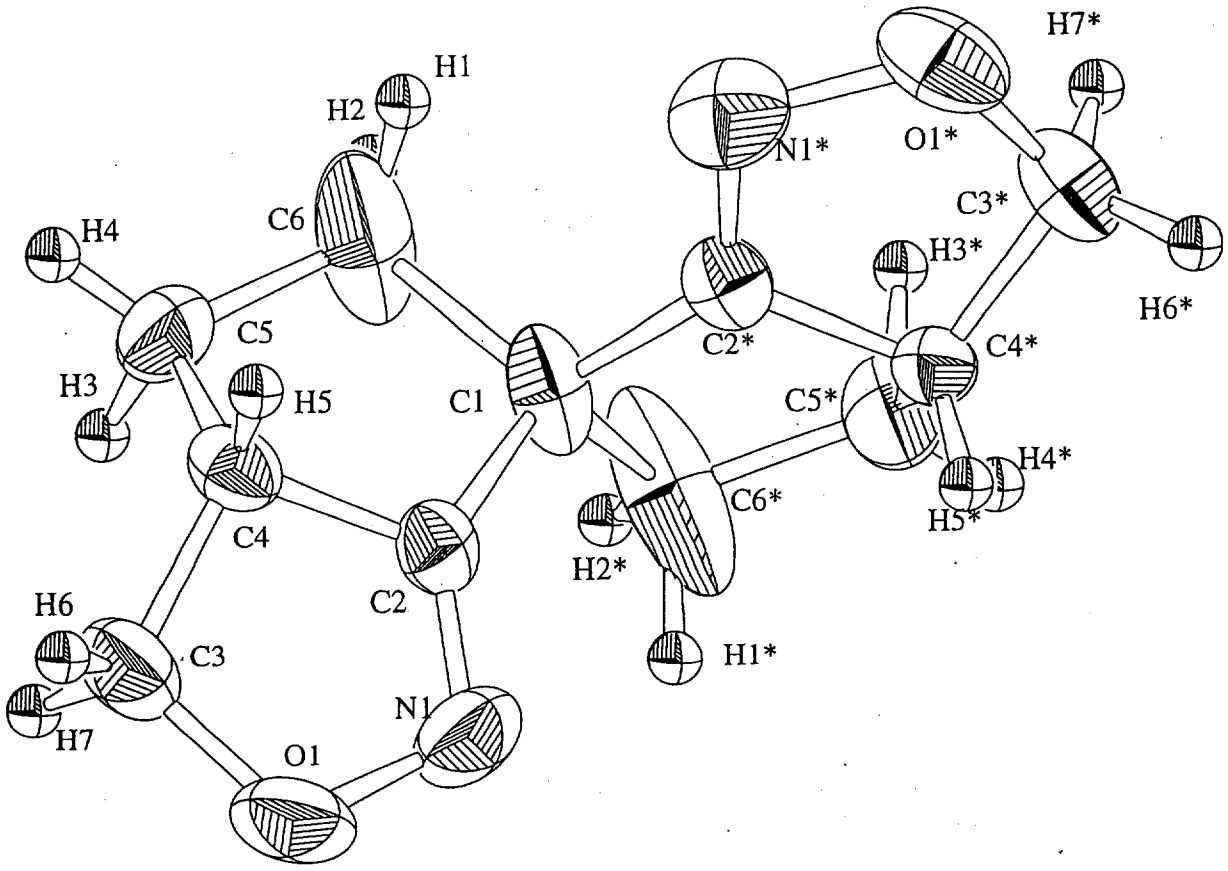
Symmetry Operators:

(1)	X,	Y,	Z	(2)	-X,	Y,	1/2-Z
(3)	-X,	-Y,	-Z	(4)	X,	-Y,	1/2+Z
(5)	1/2+X,	1/2+Y,	Z	(6)	1/2-X,	1/2+Y,	1/2-Z
(7)	1/2-X,	1/2-Y,	-Z	(8)	1/2+X,	1/2-Y,	1/2+Z

X-ray Structure Report



(*M*, *R*, *R*)-SPRIX



Experimental

Data Collection

A colorless prismatic crystal of $C_{11}H_{14}O_2N_2$ having approximate dimensions of 0.31 x 0.16 x 0.13 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

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 $38.86 < 2\theta < 39.98°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 8.786(2) $\AA$
b = 10.351(2) $\AA$
c = 11.001(2) $\AA$
V = 1000.4(6) $\AA^3$

For Z = 4 and F.W. = 206.24, the calculated density is 1.37 g/cm^3. The systematic absences of:

0kl: l $\neq$ 2n
h0l: l $\neq$ 2n
hk0: h+k $\neq$ 2n

uniquely determine the space group to be:

Pccn (#56)

The data were collected at a temperature of -75 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.18° with a take-off angle of 6.0°. Scans of (1.15 + 0.30 tan θ)° were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 5.0\sigma(I)$) were rescanned (maximum of 10 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

A total of 1361 reflections was collected. The intensities of three representative reflection were measured after every 150 reflections. Over the course of data collection, the standards increased by 0.7%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, μ, for Mo-Kα radiation is 1.0 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging

from 0.93 to 1.00. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 1.54005e-05).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] was based on 817 observed reflections ($I > 2.00\sigma(I)$) and 98 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.063$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.085$$

The standard deviation of an observation of unit weight[4] was 1.88. The weighting scheme was based on counting statistics and included a factor (p = 0.060) to downweight the intense reflections. 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 0.29 and -0.24 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" 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., 27, 435

(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 = $\frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

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	$C_{11}H_{14}O_2N_2$
Formula Weight	206.24
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.31 X 0.16 X 0.13 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (38.9 - 40.0°)
Omega Scan Peak Width at Half-height	0.18°
Lattice Parameters	a = 8.786(2)Å b = 10.351(2) Å c = 11.001(2) Å V = 1000.4(6) Å^3
Space Group	Pccn (#56)
Z value	4
D_{calc}	1.369 g/cm^3
F_{000}	440.00
μ(MoKα)	0.96 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.40, 11.34, 38.03)

Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Temperature	-75.0°C
Scan Type	ω-2θ
Scan Rate	16.0°/min (in ω) (up to 10 scans)
Scan Width	$(1.15 + 0.30 \tan \theta)°$
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 1361
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9287 - 0.9999) Decay (0.74% increase) Secondary Extinction (coefficient: 1.54005e-05)

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	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0600
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	817
No. Variables	98
Reflection/Parameter Ratio	8.34
Residuals: R; Rw	0.063 ; 0.085
Residuals: R1	0.063
No. of Reflections to calc R1	817

Goodness of Fit Indicator	1.88
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	0.29 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.24 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}	occ
O(1)	-0.5150(3)	-0.0130(2)	-0.3008(2)	4.62(5)	1.0000
N(1)	-0.6597(3)	-0.0541(2)	-0.3474(2)	4.15(6)	1.0000
C(1)	-0.7500	-0.2500	-0.4592(3)	3.74(8)	0.5000
C(2)	-0.6447(3)	-0.1680(2)	-0.3837(2)	2.63(5)	1.0000
C(3)	-0.4015(4)	-0.1029(3)	-0.3448(3)	3.93(6)	1.0000
C(4)	-0.4877(3)	-0.2225(2)	-0.3786(2)	2.81(5)	1.0000
C(5)	-0.4748(3)	-0.2797(3)	-0.5073(3)	4.11(7)	1.0000
C(6)	-0.6317(5)	-0.3329(5)	-0.5317(4)	7.9(1)	1.0000
H(1)	-0.640(8)	-0.428(7)	-0.484(7)	16(1)	1.0000
H(2)	-0.657(4)	-0.365(3)	-0.599(4)	6.6(9)	1.0000
H(3)	-0.451(4)	-0.202(4)	-0.571(4)	8(1)	1.0000
H(4)	-0.391(4)	-0.329(3)	-0.517(3)	4.0(6)	1.0000
H(5)	-0.481(3)	-0.284(3)	-0.318(3)	4.3(7)	1.0000
H(6)	-0.328(4)	-0.126(3)	-0.278(4)	7.0(9)	1.0000
H(7)	-0.357(4)	-0.059(3)	-0.424(3)	6.0(8)	1.0000

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.075(2)	0.040(1)	0.060(1)	-0.0157(10)	0.008(1)	-0.0186(9)
N(1)	0.057(2)	0.049(1)	0.052(1)	0.012(1)	0.014(1)	-0.008(1)
C(1)	0.033(2)	0.079(3)	0.030(2)	-0.017(2)	0.0000	0.0000
C(2)	0.034(1)	0.040(1)	0.026(1)	-0.0046(10)	0.0068(9)	0.0030(9)
C(3)	0.047(2)	0.051(2)	0.051(2)	-0.018(1)	0.000(1)	-0.009(1)
C(4)	0.036(1)	0.030(1)	0.040(1)	-0.0041(9)	-0.007(1)	0.0064(10)
C(5)	0.042(2)	0.050(2)	0.065(2)	0.004(1)	0.005(1)	-0.022(1)
C(6)	0.059(2)	0.166(5)	0.076(2)	-0.049(3)	0.030(2)	-0.081(3)

The general temperature factor expression:

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

Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
O(1)	N(1)	1.435(3)	O(1)	C(3)	1.447(4)
N(1)	C(2)	1.252(3)	C(1)	C(2)	1.505(3)
C(1)	C(2)	1.505(3)	C(1)	C(6)	1.566(4)
C(1)	C(6)	1.566(4)	C(2)	C(4)	1.491(3)
C(3)	C(4)	1.498(3)	C(3)	H(6)	1.01(4)
C(3)	H(7)	1.06(4)	C(4)	C(5)	1.539(4)
C(4)	H(5)	0.93(3)	C(5)	C(6)	1.509(5)
C(5)	H(3)	1.08(5)	C(5)	H(4)	0.90(3)
C(6)	H(1)	1.12(8)	C(6)	H(2)	0.84(4)

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(1)	O(1)	C(3)	107.5(2)	O(1)	N(1)	C(2)	107.4(2)
C(2)	C(1)	C(2)	113.1(3)	C(2)	C(1)	C(6)	100.5(2)
C(2)	C(1)	C(6)	112.3(2)	C(2)	C(1)	C(6)	112.3(2)
C(2)	C(1)	C(6)	100.5(2)	C(6)	C(1)	C(6)	118.7(5)
N(1)	C(2)	C(1)	130.0(2)	N(1)	C(2)	C(4)	116.2(2)
C(1)	C(2)	C(4)	112.1(2)	O(1)	C(3)	C(4)	105.4(2)
O(1)	C(3)	H(6)	110(2)	O(1)	C(3)	H(7)	104(1)
C(4)	C(3)	H(6)	107(1)	C(4)	C(3)	H(7)	109(1)
H(6)	C(3)	H(7)	118(2)	C(2)	C(4)	C(3)	99.5(2)
C(2)	C(4)	C(5)	100.3(2)	C(2)	C(4)	H(5)	110(1)
C(3)	C(4)	C(5)	120.6(2)	C(3)	C(4)	H(5)	111(1)
C(5)	C(4)	H(5)	113(1)	C(4)	C(5)	C(6)	103.7(2)
C(4)	C(5)	H(3)	108(2)	C(4)	C(5)	H(4)	112(1)
C(6)	C(5)	H(3)	109(2)	C(6)	C(5)	H(4)	121(1)
H(3)	C(5)	H(4)	100(2)	C(1)	C(6)	C(5)	108.4(2)
C(1)	C(6)	H(1)	101(3)	C(1)	C(6)	H(2)	119(2)
C(5)	C(6)	H(1)	107(3)	C(5)	C(6)	H(2)	122(2)
H(1)	C(6)	H(2)	92(4)				

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	N(1)	C(2)	C(1)	-168.3(2)	O(1)	N(1)	C(2)	C(4)	-4.7(3)
O(1)	C(3)	C(4)	C(2)	16.5(3)	O(1)	C(3)	C(4)	C(5)	124.6(3)
N(1)	O(1)	C(3)	C(4)	-20.5(3)	N(1)	C(2)	C(1)	C(2)	-93.1(3)
N(1)	C(2)	C(1)	C(6)	147.0(3)	N(1)	C(2)	C(1)	C(6)	19.8(4)
N(1)	C(2)	C(4)	C(3)	-7.8(3)	N(1)	C(2)	C(4)	C(5)	-131.5(2)
C(1)	C(2)	C(4)	C(3)	158.8(2)	C(1)	C(2)	C(4)	C(5)	35.0(2)
C(1)	C(6)	C(5)	C(4)	29.4(4)	C(2)	N(1)	O(1)	C(3)	15.9(3)
C(2)	C(1)	C(2)	C(4)	102.7(2)	C(2)	C(1)	C(6)	C(5)	-8.3(4)
C(2)	C(1)	C(6)	C(5)	-128.7(3)	C(2)	C(4)	C(5)	C(6)	-37.7(3)
C(3)	C(4)	C(5)	C(6)	-145.3(3)	C(4)	C(2)	C(1)	C(6)	-17.2(3)
C(4)	C(2)	C(1)	C(6)	-144.5(3)	C(5)	C(6)	C(1)	C(6)	114.5(4)

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O(1)	H(3)	2.66(5)	45405	O(1)	H(5)	2.70(3)	45404
O(1)	H(2)	2.84(4)	54508	O(1)	H(6)	3.23(4)	45403
O(1)	H(7)	3.31(4)	45405	O(1)	C(6)	3.515(4)	54508
O(1)	H(1)	3.53(8)	45404	O(1)	N(1)	3.589(3)	55403
O(1)	C(4)	3.597(3)	45404	N(1)	H(6)	2.75(4)	45403
N(1)	H(7)	2.78(4)	45405	N(1)	H(2)	2.85(4)	54508
N(1)	H(3)	2.97(5)	45405	N(1)	C(3)	3.409(4)	45403
N(1)	H(4)	3.43(3)	45406	N(1)	H(5)	3.55(3)	45404
C(1)	H(7)	3.58(3)	45405	C(1)	H(7)	3.58(3)	44406
C(2)	H(2)	3.15(4)	54508	C(2)	H(7)	3.16(4)	45405
C(3)	H(4)	3.27(3)	44502	C(3)	H(2)	3.33(4)	55406
C(3)	H(1)	3.48(7)	55406	C(3)	H(2)	3.53(4)	54508
C(3)	H(6)	3.53(3)	44502	C(3)	H(3)	3.54(4)	45405
C(3)	H(5)	3.57(3)	44502	C(4)	H(6)	3.38(4)	44502
C(4)	H(3)	3.49(4)	54508	C(4)	H(2)	3.53(4)	54508
C(5)	H(1)	3.19(7)	44405	C(5)	H(6)	3.38(4)	54408
C(5)	H(4)	3.41(3)	44502	C(5)	H(5)	3.48(3)	54408
C(5)	H(7)	3.48(3)	44502	C(6)	H(7)	3.11(3)	44406
C(6)	H(1)	3.44(8)	44405	C(6)	H(4)	3.54(3)	44405
H(1)	H(4)	2.53(8)	44405	H(1)	H(7)	2.55(8)	44406
H(1)	H(1)	2.9(1)	44405	H(1)	H(2)	3.50(9)	44405
H(1)	H(6)	3.54(9)	44404	H(2)	H(7)	2.69(5)	44406
H(2)	H(5)	3.25(5)	54408	H(2)	H(6)	3.38(5)	44406
H(2)	H(4)	3.44(5)	44405	H(2)	H(6)	3.49(5)	54408

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
H(3)	H(5)	2.73(6)	54408	H(3)	H(4)	3.08(5)	44502
H(3)	H(6)	3.08(5)	54408	H(3)	H(7)	3.19(5)	45405
H(3)	H(6)	3.44(6)	45407	H(4)	H(7)	2.67(5)	44502
H(4)	H(6)	2.96(5)	54408	H(4)	H(4)	2.97(6)	44502
H(4)	H(6)	3.29(5)	44502	H(4)	H(5)	3.60(4)	54408
H(5)	H(6)	2.90(5)	44502	H(5)	H(7)	3.58(4)	44502
H(6)	H(6)	2.91(7)	44502	H(7)	H(7)	3.25(7)	45405

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.

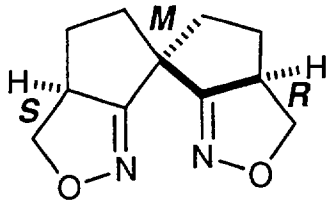
For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and 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.

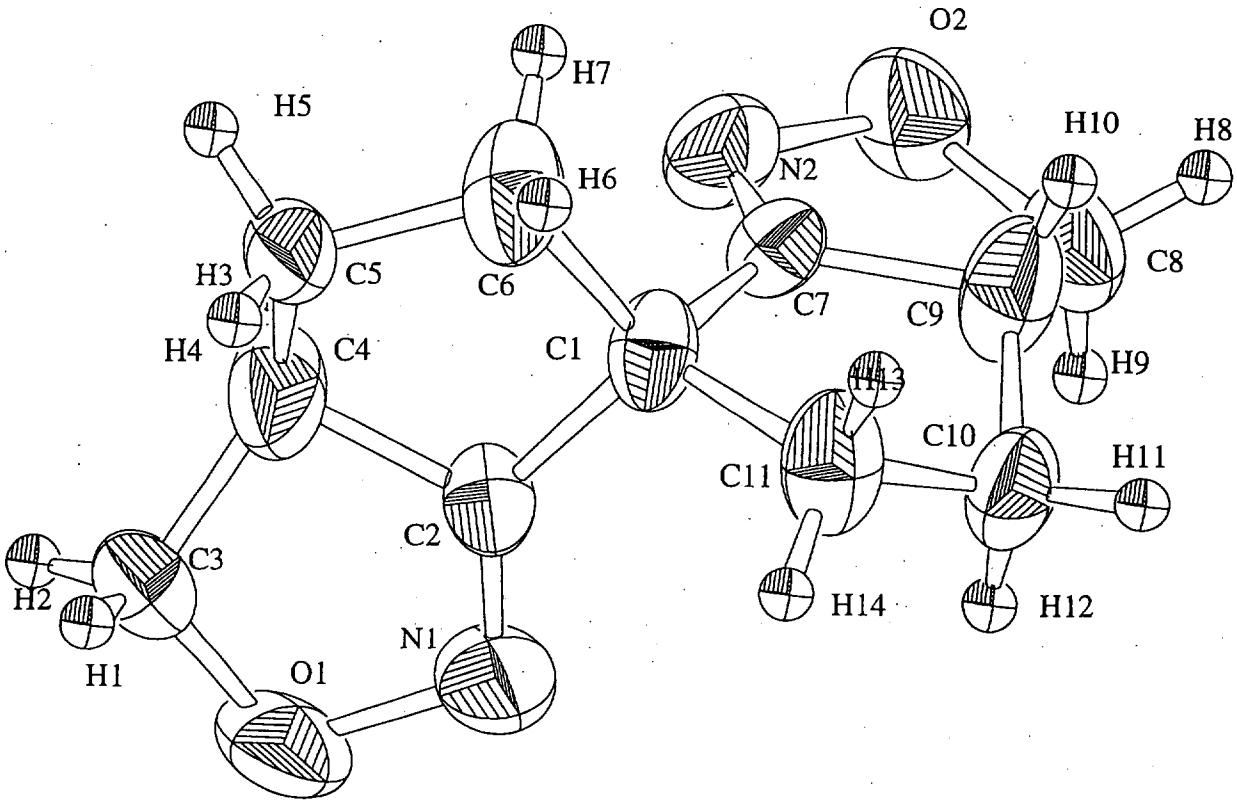
Symmetry Operators:

(1)	X,	Y,	Z	(2)	1/2-X,	1/2-Y,	Z
(3)	1/2+X,	-Y,	1/2-Z	(4)	-X,	1/2+Y,	1/2-Z
(5)	-X,	-Y,	-Z	(6)	1/2+X,	1/2+Y,	-Z
(7)	1/2-X,	Y,	1/2+Z	(8)	X,	1/2-Y,	1/2+Z

X-ray Structure Report



(*M*, *S*, *R*)-SPRIX



Experimental

Data Collection

A colorless prismatic crystal of $C_{11}H_{14}O_2N_2$ having approximate dimensions of 0.95 x 0.75 x 0.43 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

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 $39.06 < 2\theta < 40.02°$ corresponded to a primitive orthorhombic cell with dimensions:

a = 10.461(2) $\AA$
b = 11.285(2) $\AA$
c = 8.660(1) $\AA$
V = 1022.3(3) $\AA^3$

For Z = 4 and F.W. = 206.24, the calculated density is 1.34 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 -75 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.26° with a take-off angle of 6.0°. Scans of $(1.15 + 0.30 \tan \theta)°$ were made at a speed of 16.0°/min (in omega). The weak reflections ($I < 5.0\sigma(I)$) were rescanned (maximum of 10 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

A total of 1368 reflections was collected. The intensities of three representative reflection were measured after every 150 reflections. Over the course of data collection, the standards increased by 0.7%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.9 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.95 to 1.00. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement[3] was based on 1077 observed reflections ($I > 3.00\sigma(I)$) and 192 variable parameters and converged (largest parameter shift was 1.89 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.083$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2} = 0.116$$

The standard deviation of an observation of unit weight[4] was 2.70. The weighting scheme was based on counting statistics and included a factor (p = 0.060) to downweight the intense reflections. 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 0.78 and -0.27 $e^-/\AA^3$, 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., 27, 435

(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 $w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$

$\sigma_c(Fo)$ = e.s.d. based on counting statistics

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	$C_{11}H_{14}O_2N_2$
Formula Weight	206.24
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.95 X 0.75 X 0.43 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (39.1 - 40.0°)
Omega Scan Peak Width at Half-height	0.26°
Lattice Parameters	a = 10.461(2)Å b = 11.285(2) Å c = 8.660(1) Å V = 1022.3(3) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.340 g/cm^3
F_{000}	440.00
μ(MoKα)	0.94 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factors = 1.00, 3.40, 11.34, 38.03)

Take-off Angle	6.0°
Detector Aperture	9.0 mm horizontal 13.0 mm vertical
Crystal to Detector Distance	258 mm
Temperature	-75.0°C
Scan Type	ω-2θ
Scan Rate	16.0°/min (in ω) (up to 10 scans)
Scan Width	$(1.15 + 0.30 \tan\theta)°$
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 1368
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9489 - 0.9969) Decay (0.70% increase)

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	$w = \frac{1}{\sigma^2(Fo)} = [\sigma_c^2(Fo) + \frac{p^2}{4}Fo^2]^{-1}$
p-factor	0.0600
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	1077
No. Variables	192
Reflection/Parameter Ratio	5.61
Residuals: R; Rw	0.083 ; 0.116
Residuals: R1	0.084
No. of Reflections to calc R1	1077
Goodness of Fit Indicator	2.70

Max Shift/Error in Final Cycle	1.89
Maximum peak in Final Diff. Map	0.78 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.27 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
O(1)	-0.6630(4)	0.0479(4)	-0.4160(5)	5.51(10)
O(2)	-0.2391(4)	-0.0814(4)	-0.8325(6)	5.59(10)
N(1)	-0.6296(5)	-0.0097(4)	-0.5601(6)	4.8(1)
N(2)	-0.2978(5)	-0.1194(5)	-0.6904(6)	5.2(1)
C(1)	-0.5144(5)	-0.1973(4)	-0.6223(5)	3.43(10)
C(2)	-0.5803(5)	-0.1052(4)	-0.5264(6)	3.27(9)
C(3)	-0.5991(6)	-0.0151(6)	-0.2931(7)	5.0(1)
C(4)	-0.5760(7)	-0.1322(5)	-0.3547(6)	5.3(1)
C(5)	-0.4599(6)	-0.2095(5)	-0.3463(6)	4.3(1)
C(6)	-0.4657(8)	-0.2819(5)	-0.4943(7)	4.9(1)
C(7)	-0.4087(5)	-0.1430(4)	-0.7178(5)	3.44(9)
C(8)	-0.3277(6)	-0.1085(6)	-0.9566(7)	4.5(1)
C(9)	-0.4571(7)	-0.1229(7)	-0.8806(7)	6.1(2)
C(10)	-0.5471(6)	-0.2176(8)	-0.9024(6)	5.7(2)
C(11)	-0.6017(7)	-0.2530(6)	-0.7506(7)	5.1(1)
H(1)	-0.521(7)	0.023(6)	-0.267(9)	6(1)
H(2)	-0.652(7)	-0.019(6)	-0.204(9)	6(1)
H(3)	-0.648(7)	-0.180(6)	-0.331(9)	6(1)
H(4)	-0.384(7)	-0.163(6)	-0.343(9)	5(1)
H(5)	-0.463(7)	-0.260(6)	-0.258(9)	5(1)
H(6)	-0.523(7)	-0.347(6)	-0.482(9)	5(1)
H(7)	-0.383(7)	-0.311(6)	-0.520(9)	5(1)
H(9)	-0.501(7)	-0.049(6)	-0.884(9)	7(1)
H(10)	-0.614(7)	-0.192(6)	-0.969(9)	6(1)

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

atom	x	y	z	B_{eq}
H(11)	-0.505(7)	-0.284(6)	-0.948(9)	6(1)
H(12)	-0.602(7)	-0.337(6)	-0.742(9)	6(1)
H(13)	-0.687(7)	-0.224(6)	-0.741(9)	6(1)
H(14)	-0.330(7)	-0.046(6)	-1.029(9)	5(1)
H(15)	-0.303(7)	-0.180(6)	-1.007(9)	5(1)

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.073(3)	0.067(2)	0.069(3)	0.031(2)	0.017(2)	0.001(2)
O(2)	0.043(2)	0.086(3)	0.083(3)	-0.021(2)	0.012(2)	-0.006(3)
N(1)	0.062(3)	0.064(3)	0.055(3)	0.022(2)	-0.008(3)	0.004(2)
N(2)	0.051(3)	0.089(3)	0.059(3)	-0.022(3)	-0.001(2)	-0.013(3)
C(1)	0.062(3)	0.036(2)	0.033(2)	-0.008(2)	-0.002(2)	-0.003(2)
C(2)	0.045(2)	0.039(2)	0.040(2)	0.005(2)	-0.008(2)	0.000(2)
C(3)	0.071(4)	0.071(4)	0.048(3)	0.017(3)	0.016(3)	-0.008(3)
C(4)	0.097(5)	0.064(3)	0.040(3)	0.026(3)	-0.006(3)	-0.006(3)
C(5)	0.069(3)	0.056(3)	0.037(2)	0.015(3)	-0.004(3)	0.007(2)
C(6)	0.095(5)	0.044(3)	0.047(3)	0.017(3)	0.006(3)	0.006(3)
C(7)	0.053(3)	0.042(2)	0.036(2)	-0.018(2)	0.002(2)	-0.004(2)
C(8)	0.048(3)	0.074(4)	0.051(3)	-0.006(3)	0.012(3)	0.003(3)
C(9)	0.077(4)	0.109(5)	0.044(3)	-0.045(4)	-0.002(3)	0.015(3)
C(10)	0.045(3)	0.138(6)	0.033(3)	-0.027(4)	-0.001(2)	-0.012(3)
C(11)	0.089(4)	0.062(3)	0.041(3)	-0.034(3)	-0.002(3)	-0.009(2)

The general temperature factor expression:

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

Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
O(1)	N(1)	1.454(8)	O(1)	C(3)	1.444(9)
O(2)	N(2)	1.443(8)	O(2)	C(8)	1.451(9)
N(1)	C(2)	1.232(8)	N(2)	C(7)	1.212(8)
C(1)	C(2)	1.496(8)	C(1)	C(6)	1.549(8)
C(1)	C(7)	1.510(8)	C(1)	C(11)	1.571(9)
C(2)	C(4)	1.523(9)	C(3)	C(4)	1.454(10)
C(3)	H(2)	0.98(7)	C(4)	C(5)	1.497(10)
C(5)	C(6)	1.520(9)	C(5)	H(4)	1.1(1)
C(5)	H(5)	0.86(8)	C(6)	H(6)	0.78(8)
C(6)	H(7)	1.1(1)	C(7)	C(9)	1.518(9)
C(8)	C(9)	1.522(10)	C(8)	H(8)	0.92(8)
C(8)	H(9)	0.89(10)	C(9)	C(10)	1.44(1)
C(10)	C(11)	1.490(10)	C(10)	H(11)	0.88(8)
C(10)	H(12)	0.9(1)	C(11)	H(13)	0.8(1)

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
N(1)	O(1)	C(3)	107.7(4)	N(2)	O(2)	C(8)	107.3(4)
O(1)	N(1)	C(2)	106.7(5)	O(2)	N(2)	C(7)	107.8(5)
C(2)	C(1)	C(6)	100.4(4)	C(2)	C(1)	C(7)	111.2(4)
C(2)	C(1)	C(11)	113.6(6)	C(6)	C(1)	C(7)	113.6(6)
C(6)	C(1)	C(11)	116.8(5)	C(7)	C(1)	C(11)	101.7(4)
N(1)	C(2)	C(1)	132.0(5)	N(1)	C(2)	C(4)	115.0(6)
C(1)	C(2)	C(4)	112.9(4)	O(1)	C(3)	C(4)	104.7(6)
O(1)	C(3)	H(2)	105(3)	C(4)	C(3)	H(2)	130(3)
C(2)	C(4)	C(3)	99.6(5)	C(2)	C(4)	C(5)	100.5(5)
C(3)	C(4)	C(5)	130.0(7)	C(4)	C(5)	C(6)	103.8(6)
C(4)	C(5)	H(4)	98(5)	C(4)	C(5)	H(5)	117(5)
C(6)	C(5)	H(4)	109(5)	C(6)	C(5)	H(5)	113(5)
H(4)	C(5)	H(5)	113(7)	C(1)	C(6)	C(5)	106.6(5)
C(1)	C(6)	H(6)	118(6)	C(1)	C(6)	H(7)	108(6)
C(5)	C(6)	H(6)	118(6)	C(5)	C(6)	H(7)	96(6)
H(6)	C(6)	H(7)	106(8)	N(2)	C(7)	C(1)	132.9(6)
N(2)	C(7)	C(9)	118.2(6)	C(1)	C(7)	C(9)	108.9(5)
O(2)	C(8)	C(9)	105.7(5)	O(2)	C(8)	H(8)	112(5)
O(2)	C(8)	H(9)	115(6)	C(9)	C(8)	H(8)	120(5)
C(9)	C(8)	H(9)	97(6)	H(8)	C(8)	H(9)	103(7)
C(7)	C(9)	C(8)	96.6(6)	C(7)	C(9)	C(10)	103.0(6)
C(8)	C(9)	C(10)	126.8(8)	C(9)	C(10)	C(11)	109.4(6)
C(9)	C(10)	H(11)	124(5)	C(9)	C(10)	H(12)	106(8)
C(11)	C(10)	H(11)	112(5)	C(11)	C(10)	H(12)	99(8)

Table 4. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(11)	C(10)	H(12)	101(8)	C(1)	C(11)	C(10)	107.0(5)
C(1)	C(11)	H(13)	95(9)	C(10)	C(11)	H(13)	108(9)

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	N(1)	C(2)	C(1)	-172.5(6)	O(1)	N(1)	C(2)	C(4)	2.9(8)
O(1)	C(3)	C(4)	C(2)	23.3(8)	O(1)	C(3)	C(4)	C(5)	136.0(7)
O(2)	N(2)	C(7)	C(1)	173.7(6)	O(2)	N(2)	C(7)	C(9)	-3.4(9)
O(2)	C(8)	C(9)	C(7)	-19.5(7)	O(2)	C(8)	C(9)	C(10)	-131.1(8)
N(1)	O(1)	C(3)	C(4)	-24.0(8)	N(1)	C(2)	C(1)	C(6)	173.7(7)
N(1)	C(2)	C(1)	C(7)	53.1(9)	N(1)	C(2)	C(1)	C(11)	-60.8(9)
N(1)	C(2)	C(4)	C(3)	-17.4(9)	N(1)	C(2)	C(4)	C(5)	-151.4(6)
N(2)	O(2)	C(8)	C(9)	20.0(8)	N(2)	C(7)	C(1)	C(2)	84.1(9)
N(2)	C(7)	C(1)	C(6)	-28.3(9)	N(2)	C(7)	C(1)	C(11)	-154.7(8)
N(2)	C(7)	C(9)	C(8)	15.0(9)	N(2)	C(7)	C(9)	C(10)	145.2(7)
C(1)	C(2)	C(4)	C(3)	159.0(6)	C(1)	C(2)	C(4)	C(5)	24.9(8)
C(1)	C(6)	C(5)	C(4)	38.9(8)	C(1)	C(7)	C(9)	C(8)	-162.8(5)
C(1)	C(7)	C(9)	C(10)	-32.5(8)	C(1)	C(11)	C(10)	C(9)	-15(1)
C(2)	N(1)	O(1)	C(3)	13.2(7)	C(2)	C(1)	C(6)	C(5)	-22.2(8)
C(2)	C(1)	C(7)	C(9)	-98.6(6)	C(2)	C(1)	C(11)	C(10)	114.7(8)
C(2)	C(4)	C(5)	C(6)	-37.4(7)	C(3)	C(4)	C(5)	C(6)	-149.6(8)
C(4)	C(2)	C(1)	C(6)	-1.9(8)	C(4)	C(2)	C(1)	C(7)	-122.4(6)
C(4)	C(2)	C(1)	C(11)	123.7(6)	C(5)	C(6)	C(1)	C(7)	96.6(7)
C(5)	C(6)	C(1)	C(11)	-145.5(7)	C(6)	C(1)	C(7)	C(9)	148.9(6)
C(6)	C(1)	C(11)	C(10)	-129.0(8)	C(7)	N(2)	O(2)	C(8)	-10.9(8)
C(7)	C(1)	C(11)	C(10)	-4.8(8)	C(7)	C(9)	C(10)	C(11)	28.9(10)
C(8)	C(9)	C(10)	C(11)	137.5(9)	C(9)	C(7)	C(1)	C(11)	22.6(7)

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O(1)	H(14)	2.4(1)	35502	O(1)	H(10)	2.8(2)	35502
O(1)	H(12)	2.9(1)	45304	O(1)	H(5)	3.00(8)	45404
O(1)	H(9)	3.16(10)	45304	O(1)	H(11)	3.17(8)	35502
O(1)	H(2)	3.26(7)	35402	O(1)	C(10)	3.59(1)	35502
O(2)	H(1)	2.4(2)	45402	O(2)	H(3)	2.8(2)	54403
O(2)	H(4)	2.8(1)	45402	O(2)	H(8)	3.04(9)	45502
O(2)	H(11)	3.11(8)	54303	O(2)	H(13)	3.5(1)	45304
N(1)	H(2)	2.67(7)	35402	N(1)	H(12)	2.9(2)	45304
N(1)	H(7)	3.3(1)	44403	N(1)	H(10)	3.4(2)	35502
N(1)	C(3)	3.490(9)	35402	N(1)	H(13)	3.6(1)	45304
N(2)	H(3)	2.3(2)	54403	N(2)	H(8)	2.69(9)	45502
N(2)	H(1)	3.2(2)	45402	N(2)	C(8)	3.526(9)	45502
N(2)	H(6)	3.56(8)	54403	C(1)	H(3)	3.6(1)	54403
C(2)	H(7)	3.2(1)	44403	C(2)	H(2)	3.44(7)	35402
C(3)	H(14)	3.0(2)	35502	C(3)	H(5)	3.05(8)	45404
C(3)	H(10)	3.1(2)	55601	C(3)	H(6)	3.26(8)	45404
C(3)	H(11)	3.59(8)	55601	C(4)	H(7)	3.3(1)	44403
C(4)	H(11)	3.43(8)	55601	C(5)	H(14)	3.0(1)	54403
C(5)	H(1)	3.3(1)	44404	C(5)	H(2)	3.36(7)	44404
C(5)	H(9)	3.37(10)	55601	C(5)	H(8)	3.47(8)	55601
C(5)	H(11)	3.54(8)	55601	C(6)	H(10)	3.0(2)	44304
C(6)	H(2)	3.09(7)	44404	C(6)	H(3)	3.3(1)	54403
C(6)	H(1)	3.3(1)	44404	C(6)	H(14)	3.5(1)	54403
C(7)	H(3)	2.9(2)	54403	C(8)	H(11)	3.08(8)	54303

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(8)	H(5)	3.38(8)	55401	C(8)	H(3)	3.4(2)	54403
C(8)	H(14)	3.5(1)	54303	C(8)	H(6)	3.53(8)	45304
C(8)	H(4)	3.5(1)	55401	C(8)	H(1)	3.6(1)	55401
C(9)	H(6)	3.35(8)	45304	C(9)	H(13)	3.6(1)	45304
C(10)	H(9)	3.21(9)	44303	C(10)	H(5)	3.33(8)	55401
C(11)	H(9)	3.26(10)	44303	C(11)	H(4)	3.3(1)	44403
C(11)	H(7)	3.6(1)	44403	C(11)	H(8)	3.57(9)	44303
H(1)	H(5)	2.7(2)	45404	H(1)	H(6)	2.8(2)	45404
H(1)	H(8)	2.8(2)	55601	H(1)	H(10)	3.3(2)	55601
H(1)	H(12)	3.3(2)	45304	H(2)	H(10)	2.5(2)	55601
H(2)	H(14)	2.6(2)	35502	H(2)	H(6)	2.7(1)	45404
H(2)	H(7)	2.8(1)	45404	H(2)	H(5)	2.9(1)	45404
H(2)	H(11)	3.26(10)	55601	H(3)	H(7)	2.4(2)	44403
H(3)	H(11)	3.0(1)	55601	H(3)	H(9)	3.2(2)	44403
H(4)	H(14)	2.9(2)	54403	H(4)	H(8)	3.0(1)	55601
H(4)	H(9)	3.2(1)	55601	H(4)	H(13)	3.3(2)	54403
H(4)	H(8)	3.5(2)	45502	H(5)	H(14)	2.6(2)	54403
H(5)	H(9)	2.8(1)	55601	H(5)	H(11)	3.0(1)	55601
H(5)	H(12)	3.0(2)	55601	H(5)	H(8)	3.2(1)	55601
H(6)	H(10)	2.4(2)	44304	H(6)	H(8)	3.1(1)	44304
H(7)	H(14)	2.8(2)	54403	H(7)	H(10)	2.9(2)	44304
H(8)	H(11)	3.3(1)	54303	H(8)	H(13)	3.4(2)	54303
H(8)	H(14)	3.5(2)	54303	H(9)	H(11)	2.5(1)	54303
H(9)	H(14)	2.8(2)	54303	H(9)	H(13)	3.4(2)	54303

Table 6. Non-bonded Contacts out to 3.60 $\AA$ (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
H(9)	H(12)	3.5(2)	54303	H(10)	H(13)	3.3(2)	45304

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.

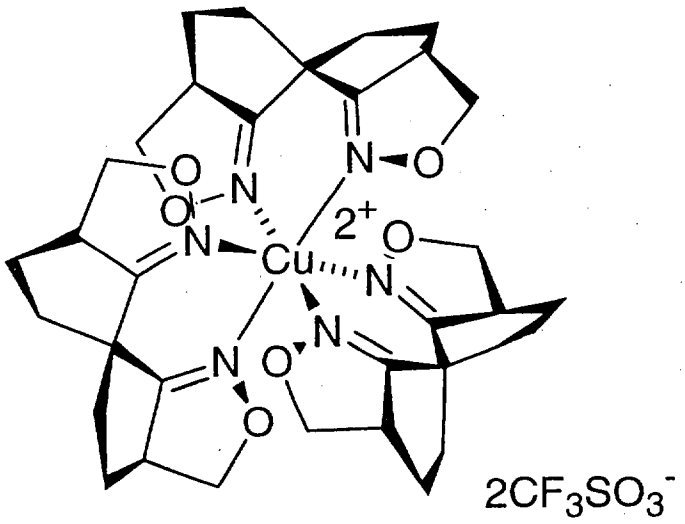
For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and 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.

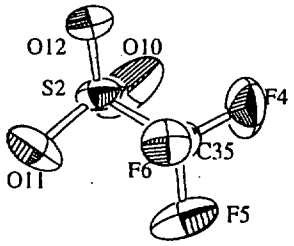
Symmetry Operators:

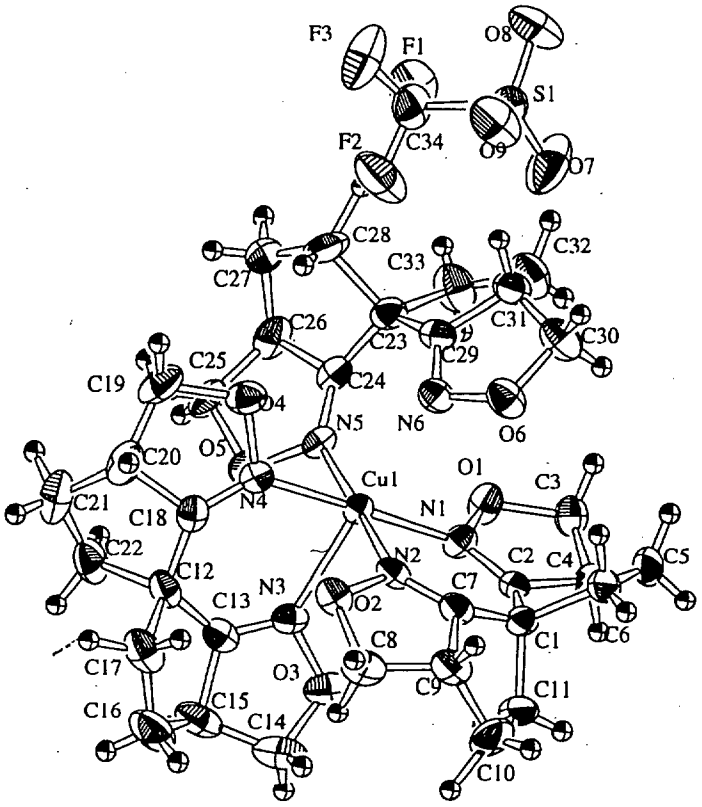
(1)	X,	Y,	Z	(2)	1/2-X,	-Y,	1/2+Z
(3)	1/2+X,	1/2-Y,	-Z	(4)	-X,	1/2+Y,	1/2-Z

X-ray Structure Report



A Hexacoordinated Cu Complex of (*M,S,S*)-SPRIX


O12
O10
S2
F4
C35
F6
O11
F5


F3
F1
O8
S1
F2
C34
O9
O7
C28
C32
C33
C27
C31
C26
C23
C29
C30
C25
C24
C19
O4
N5
N6
O6
C20
O5
O1
Cu1
C3
C21
N4
N1
C18
C22
N2
C2
C4
C5
C12
C7
N3
C13
O2
C1
C6
C8
C17
O3
C9
C11
C16
C15
C14
C10

Experimental

Data Collection

A green plate crystal of $C_{35}H_{42}O_{12}N_6CuF_6S_2$ having approximate dimensions of 0.33 x 0.75 x 0.13 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Mo-Kα radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 24 carefully centered reflections in the range $38.75 < 2\theta < 39.91°$ corresponded to amonoclinic cell with dimensions:

a = 7.493(3) $\AA$

b = 13.001(4) $\AA$ $\quad \beta = 95.77(2)°$

c = 20.876(2) $\AA$

V = 2023.3(10) $\AA^3$

For Z = 4 and F.W. = 980.41, the calculated density is 3.22 g/cm^3. Based on the systematic absences of:

0k0: k $\neq$ 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of -100 $\pm$ 1°C using the ω-2θ scan technique to a maximum 2θ value of 55.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.31° with a take-off angle of 6.0°. Scans of $(1.10 + 0.30 \tan \theta)°$ were made at a speed of 8.0°/min (in omega). The weak reflections ($I < 5.0\sigma(I)$) were rescanned (maximum of 10 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 258 mm, and the detector aperture was 9.0 x 13.0 mm (horizontal x vertical).

Data Reduction

Of the 10426 reflections which were collected, 4850 were unique (R_{int} = 0.048). The intensities of three representative reflection were measured after every 150 reflections. Over the course of data collection, the standards decreased by 3.7%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, μ, for Mo-Kα radiation is 14.8 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.85 to 1.00. The data were corrected for Lorentz and polarization effects.