Supporting Information

Pyrrolo Annelated Tetrathiafulvalenes: The Parent Systems

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Crystal Data, Data Collection, Structure Solution and Refinement Parameters for 22b and 24b

Compound	22b	24b
Empirical formula	C ₂₅ H ₃₁ NO ₂ S ₇	C ₁₄ H ₁₇ N ₁ S ₆
Formula weight	601.93	391.65
Crystal appearance	yellow, needle	yellow, plate
Crystal size	0.28 × 0.07 × 0.03 mm	0.87 × 0.20 × 0.03 mm
Crystal system	monoclinic	monoclinic
Space group	C2/c (#15)	P2 ₁ /a (#14)
<i>a</i> (Å)	47.689(5)	8.626(3)
<i>b</i> (Å)	5.2313(5)	10.507(3)
<i>c</i> (Å)	22.185(2)	20.251(2)
β (deg)	90.990(10)	94.72(2)
<i>V</i> (Å ³)	5533.8(9)	1829.2(7)
<i>Z</i>	8	4
<i>D_c</i> (g cm ⁻³)	1.445	1.422
Abs coeff (mm ⁻¹)	0.595	68.32
<i>F</i> (000)	2528	816
Diffractionmeter	Siemens SMART	Rigaku AFC6S
Temperature (K)	120(2)	296
Radiation λ (Å)	graphite monochromatized <i>Mo Kα</i> (0.71073)	graphite monochromatized <i>Cu Kα</i> (1.54178)
Scan mode	ω	$\omega/2\theta$
$\theta_{\max}$ (deg)	26.40	62.90
Reflections collected	28284	2887
Reflections unique	5685 [<i>R</i> _{int} 0.0847]	2658 [<i>R</i> _{int} 0.019]
Completeness (to $\theta_{\max}$)	99.9%	not relevant
Data reduction	SAINT (Siemens, 1995) ^a	TEXAN ^d
Absorption correction	SADABS (Sheldrick) ^b	empirical ^e
Max and min transmission	0.9853 and 0.8511	0.9971 and 0.3558
Solution method	direct methods (SHELX-97) ^c	direct methods (SIR-97) ^e
Refinement method	full-matrix least-squares on <i>F</i> ²	full-matrix least-squares on <i>F</i> ²
H atom treatment	riding positions	riding positions
Data/restraints/parameters	5685/0 /319	1551/0/190
Goodness-of-fit on <i>F</i> ²	0.989	4.57
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0388, <i>wR</i> ₂ = 0.0840	<i>R</i> ₁ = 0.073, <i>wR</i> ₂ = 0.068
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0808, <i>wR</i> ₂ = 0.1088	not relevant
Largest diff peak and hole (e Å ⁻³)	0.479 and -0.564	0.32 and -0.25

- ^a *SMART and Saint. Area Detector Software*, **1995**. Simens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA
- ^b Sheldrick, G. M. *SADABS*, **1996**. University of Göttingen, Germany
- ^c Sheldrick, G. M. *SHELX-97*, **1996**. University of Göttingen, Germany
- ^d *TEXAN. Crystal Structure Analysis Package*, **1985&1992**. Molecular Structure Corporation, Woodlands, Texas, USA
- ^e Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, M.; Giacovazzo, C.; Guagliardi, A.; Polidori, G. *J. Appl. Cryst.* **1994**, 27, 435

Figure S1. X-ray crystal structure of **22b** showing thermal ellipsoids at the 50% probability level.

Figure S2. X-ray crystal structure of **24b** showing thermal ellipsoids at the 50% probability level.

Table 1. Crystal data and structure refinement for Jb64k.

Identification code	jb64k
Empirical formula	C ₂₅ H ₃₁ N O ₂ S ₇
Formula weight	601.93
Temperature	120(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C 1 2/c 1
Unit cell dimensions	a = 47.689(5) Å alpha = 90 deg. b = 5.2313(5) Å beta = 90.990(10) deg. c = 22.185(2) Å gamma = 90 deg.
Volume	5533.8(9) Å ³
Z, Calculated density	8, 1.445 Mg/m ³
Absorption coefficient	0.595 mm ⁻¹
F(000)	2528
Crystal size	0.28 x 0.07 x 0.03 mm
Theta range for data collection	0.85 to 26.40 deg.
Index ranges	-58<=h<=58, -6<=k<=6, -27<=l<=27
Reflections collected / unique	28284 / 5685 [R(int) = 0.0847]
Completeness to 2theta = 26.40	99.9%
Max. and min. transmission	0.9853 and 0.8511
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5685 / 0 / 319
Goodness-of-fit on F ²	0.989
Final R indices [I>2sigma(I)]	R1 = 0.0388, wR2 = 0.0840
R indices (all data)	R1 = 0.0808, wR2 = 0.1088
Largest diff. peak and hole	0.479 and -0.564 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for Jb64k.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
S(1)	1232(1)	8970(2)	772(1)	23(1)
S(2)	907(1)	5237(2)	1641(1)	22(1)
S(3)	553(1)	2302(2)	2505(1)	20(1)
S(4)	1151(1)	-560(2)	2949(1)	19(1)
S(5)	1437(1)	2911(2)	2072(1)	20(1)
S(6)	1786(1)	6820(2)	1209(1)	18(1)
S(7)	2035(1)	14369(2)	-438(1)	15(1)
O(1)	2305(1)	14899(4)	-178(1)	19(1)
O(2)	1839(1)	16363(4)	-575(1)	18(1)
N(1)	1875(1)	12494(5)	59(1)	15(1)
C(1)	1415(1)	6842(6)	1255(1)	17(1)
C(2)	1276(1)	5262(6)	1616(1)	17(1)
C(3)	885(1)	3000(6)	2227(1)	17(1)
C(4)	1126(1)	1900(6)	2418(1)	16(1)
C(5)	1796(1)	9382(6)	705(1)	15(1)
C(6)	2005(1)	10684(6)	430(1)	16(1)
C(7)	1585(1)	12276(6)	95(1)	18(1)
C(8)	1535(1)	10372(6)	495(1)	18(1)
C(9)	2078(1)	12487(6)	-1086(1)	14(1)
C(10)	2279(1)	10553(6)	-1089(1)	19(1)
C(11)	2312(1)	9139(6)	-1605(1)	18(1)
C(12)	2150(1)	9622(6)	-2125(1)	17(1)
C(13)	1950(1)	11529(6)	-2109(1)	18(1)
C(14)	1912(1)	12977(6)	-1593(1)	17(1)
C(15)	2198(1)	8125(6)	-2694(1)	24(1)
C(16)	448(1)	5457(6)	2776(1)	21(1)
C(17)	213(1)	5148(6)	3229(1)	22(1)
C(18)	114(1)	7735(6)	3464(2)	24(1)
C(19)	-122(1)	7506(7)	3911(2)	30(1)
C(20)	-220(1)	10105(8)	4141(2)	44(1)
C(21)	1341(1)	1063(6)	3561(1)	21(1)
C(22)	1191(1)	3376(6)	3810(1)	21(1)
C(23)	931(1)	2812(7)	4167(1)	23(1)
C(24)	802(1)	5218(7)	4430(2)	32(1)
C(25)	558(1)	4660(9)	4843(2)	46(1)

Table 3. Bond lengths [Å] and angles [deg] for Jb64k.

S(1)-C(8)	1.742(3)
S(1)-C(1)	1.766(3)
S(2)-C(3)	1.754(3)
S(2)-C(2)	1.762(3)
S(3)-C(3)	1.750(3)
S(3)-C(16)	1.829(3)
S(4)-C(4)	1.747(3)
S(4)-C(21)	1.827(3)
S(5)-C(2)	1.760(3)
S(5)-C(4)	1.763(3)

S(6)-C(5)	1.746(3)
S(6)-C(1)	1.774(3)
S(7)-O(1)	1.429(2)
S(7)-O(2)	1.430(2)
S(7)-N(1)	1.670(2)
S(7)-C(9)	1.758(3)
N(1)-C(7)	1.389(4)
N(1)-C(6)	1.395(4)
C(1)-C(2)	1.335(4)
C(3)-C(4)	1.348(4)
C(5)-C(6)	1.360(4)
C(5)-C(8)	1.422(4)
C(7)-C(8)	1.357(4)
C(9)-C(14)	1.388(4)
C(9)-C(10)	1.394(4)
C(10)-C(11)	1.376(4)
C(11)-C(12)	1.400(4)
C(12)-C(13)	1.380(4)
C(12)-C(15)	1.507(4)
C(13)-C(14)	1.387(4)
C(16)-C(17)	1.527(4)
C(17)-C(18)	1.528(4)
C(18)-C(19)	1.519(4)
C(19)-C(20)	1.527(5)
C(21)-C(22)	1.514(4)
C(22)-C(23)	1.514(4)
C(23)-C(24)	1.521(5)
C(24)-C(25)	1.522(5)

C(8)-S(1)-C(1)	94.33(15)
C(3)-S(2)-C(2)	95.68(15)
C(3)-S(3)-C(16)	100.47(15)
C(4)-S(4)-C(21)	100.70(15)
C(2)-S(5)-C(4)	95.53(15)
C(5)-S(6)-C(1)	94.14(15)
O(1)-S(7)-O(2)	121.70(13)
O(1)-S(7)-N(1)	105.54(13)
O(2)-S(7)-N(1)	105.28(13)
O(1)-S(7)-C(9)	108.71(14)
O(2)-S(7)-C(9)	108.67(14)
N(1)-S(7)-C(9)	105.81(13)
C(7)-N(1)-C(6)	110.2(2)
C(7)-N(1)-S(7)	123.6(2)
C(6)-N(1)-S(7)	125.7(2)
C(2)-C(1)-S(1)	120.6(2)
C(2)-C(1)-S(6)	122.5(2)
S(1)-C(1)-S(6)	116.83(17)
C(1)-C(2)-S(5)	124.0(2)
C(1)-C(2)-S(2)	122.0(2)
S(5)-C(2)-S(2)	113.88(17)
C(4)-C(3)-S(3)	125.1(2)
C(4)-C(3)-S(2)	117.2(2)
S(3)-C(3)-S(2)	117.72(19)
C(3)-C(4)-S(4)	125.0(2)
C(3)-C(4)-S(5)	117.1(2)
S(4)-C(4)-S(5)	117.84(18)
C(6)-C(5)-C(8)	108.5(3)
C(6)-C(5)-S(6)	134.5(3)
C(8)-C(5)-S(6)	117.0(2)

C(5)-C(6)-N(1)	106.3(3)
C(8)-C(7)-N(1)	106.6(3)
C(7)-C(8)-C(5)	108.5(3)
C(7)-C(8)-S(1)	134.1(3)
C(5)-C(8)-S(1)	117.3(2)
C(14)-C(9)-C(10)	120.9(3)
C(14)-C(9)-S(7)	119.1(2)
C(10)-C(9)-S(7)	120.0(2)
C(11)-C(10)-C(9)	118.9(3)
C(10)-C(11)-C(12)	121.2(3)
C(13)-C(12)-C(11)	118.8(3)
C(13)-C(12)-C(15)	120.8(3)
C(11)-C(12)-C(15)	120.4(3)
C(12)-C(13)-C(14)	121.2(3)
C(13)-C(14)-C(9)	119.0(3)
C(17)-C(16)-S(3)	109.2(2)
C(16)-C(17)-C(18)	111.4(3)
C(19)-C(18)-C(17)	112.9(3)
C(18)-C(19)-C(20)	112.4(3)
C(22)-C(21)-S(4)	114.4(2)
C(23)-C(22)-C(21)	115.6(3)
C(22)-C(23)-C(24)	112.3(3)
C(23)-C(24)-C(25)	113.0(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Jb64k.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
S(1)	14(1)	34(1)	21(1)	11(1)	0(1)	-3(1)
S(2)	15(1)	28(1)	23(1)	9(1)	2(1)	0(1)
S(3)	16(1)	20(1)	25(1)	1(1)	5(1)	-3(1)
S(4)	22(1)	16(1)	18(1)	2(1)	0(1)	-1(1)
S(5)	15(1)	22(1)	22(1)	6(1)	3(1)	1(1)
S(6)	16(1)	22(1)	17(1)	4(1)	1(1)	0(1)
S(7)	15(1)	18(1)	13(1)	0(1)	1(1)	-3(1)
O(1)	17(1)	24(1)	18(1)	-1(1)	-1(1)	-3(1)
O(2)	20(1)	17(1)	17(1)	1(1)	1(1)	1(1)
N(1)	15(1)	19(1)	11(1)	3(1)	3(1)	-1(1)
C(1)	14(2)	19(2)	17(2)	-2(1)	2(1)	-1(1)
C(2)	15(2)	20(2)	16(2)	-2(1)	1(1)	2(1)
C(3)	19(2)	16(2)	16(2)	3(1)	3(1)	-3(1)
C(4)	17(2)	16(2)	15(2)	-3(1)	2(1)	-3(1)
C(5)	17(2)	18(2)	10(2)	-2(1)	0(1)	-1(1)
C(6)	15(2)	21(2)	11(2)	-1(1)	0(1)	4(1)
C(7)	13(2)	24(2)	17(2)	1(1)	-1(1)	1(2)
C(8)	15(2)	25(2)	13(2)	0(1)	1(1)	-4(2)
C(9)	13(2)	15(2)	15(2)	1(1)	5(1)	-5(1)

C(10)	17(2)	23(2)	17(2)	4(1)	-3(1)	-3(2)
C(11)	17(2)	18(2)	20(2)	3(1)	5(1)	3(1)
C(12)	16(2)	19(2)	16(2)	-2(1)	3(1)	-6(1)
C(13)	15(2)	22(2)	17(2)	2(1)	-1(1)	-5(1)
C(14)	15(2)	18(2)	17(2)	2(1)	1(1)	3(1)
C(15)	25(2)	27(2)	21(2)	-3(2)	4(1)	-2(2)
C(16)	19(2)	20(2)	22(2)	-2(1)	2(1)	-1(2)
C(17)	16(2)	28(2)	21(2)	0(1)	2(1)	-2(2)
C(18)	23(2)	23(2)	26(2)	-3(2)	3(2)	2(2)
C(19)	29(2)	33(2)	29(2)	-2(2)	11(2)	4(2)
C(20)	48(3)	41(3)	45(2)	2(2)	22(2)	11(2)
C(21)	21(2)	25(2)	18(2)	0(1)	-3(1)	-2(2)
C(22)	22(2)	24(2)	17(2)	-3(1)	-2(1)	-1(2)
C(23)	25(2)	26(2)	19(2)	0(2)	-3(1)	1(2)
C(24)	29(2)	38(2)	30(2)	-9(2)	3(2)	3(2)
C(25)	35(2)	67(3)	35(2)	-13(2)	7(2)	4(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Jb64k.

	x	y	z	U(eq)
H(6)	2201	10416	481	19
H(7)	1449	13266	-117	22
H(10)	2391	10218	-740	23
H(11C)	2448	7809	-1610	22
H(13)	1837	11856	-2457	22
H(14)	1774	14284	-1587	20
H(15A)	2155	6318	-2624	36
H(15B)	2394	8295	-2811	36
H(15C)	2076	8787	-3018	36
H(16A)	610	6322	2970	25
H(16B)	382	6521	2433	25
H(17A)	281	4092	3572	26
H(17B)	53	4245	3034	26
H(18A)	275	8624	3660	29
H(18B)	50	8794	3118	29
H(19A)	-58	6453	4257	36
H(19B)	-283	6617	3715	36
H(20A)	-62	10986	4340	67
H(20B)	-370	9860	4430	67
H(20C)	-289	11136	3801	67
H(21A)	1374	-171	3893	25
H(21B)	1526	1609	3413	25
H(22A)	1325	4330	4072	25
H(22B)	1138	4509	3469	25
H(23A)	979	1617	4499	28
H(23B)	790	1961	3901	28
H(24A)	736	6332	4096	39
H(24B)	948	6162	4660	39
H(25A)	420	3580	4631	69
H(25B)	469	6271	4960	69

H (25C)	627	3772	5205	69
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X-ray Structure Report

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Experimental

Data Collection

A yellow plate crystal of $C_{14}H_{17}NS_6$ having approximate dimensions of 0.87 x 0.20 x 0.03 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC6S diffractometer with graphite monochromated Cu-K α radiation.

Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 22 carefully centered reflections in the range $50.09 < 2\theta < 57.11^\circ$ corresponded to a primitive monoclinic cell with dimensions:

$$\begin{aligned} a &= 8.626(3) \text{ \AA} \\ b &= 10.507(3) \text{ \AA} \quad \beta = 94.72(2)^\circ \\ c &= 20.251(2) \text{ \AA} \\ V &= 1829.2(7) \text{ \AA}^3 \end{aligned}$$

For $Z = 4$ and F.W. = 391.66, the calculated density is 1.42 g/cm³. The systematic absences of:

$$\begin{aligned} h0l: h &\neq 2n \\ 0k0: k &\neq 2n \end{aligned}$$

uniquely determine the space group to be:

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

The data were collected at a temperature of $23 \pm 1^\circ\text{C}$ using the ω - 2θ scan technique to a maximum 2θ value of 125.8° . Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.23° with a take-off angle of 6.0° . Scans of $(1.15 + 0.30 \tan \theta)^\circ$ were made at a speed of $4.0^\circ/\text{min}$ (in omega). The weak reflections ($I < 3.0\sigma(I)$) were rescanned (maximum of 1 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 0.8 mm, the crystal to detector distance was 258 mm, and the detector aperture was 6.0 x 6.0 mm (horizontal x vertical).

Data Reduction

Of the 2887 reflections which were collected, 2658 were unique ($R_{int} = 0.019$). The intensities of three representative reflection were measured after every 150 reflections. No decay correction was applied.

The linear absorption coefficient, μ , for Cu-K α radiation is 68.3 cm^{-1} . An empirical absorption correction based on azimuthal scans of several reflections was applied which resulted in transmission factors ranging from 0.36 to 1.00. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods¹ and expanded using Fourier techniques². The non-hydrogen atoms were refined anisotropically. The final cycle of full-matrix least-squares refinement³ was based on 1551 observed reflections ($I > 3.00\sigma(I)$) and 190 variable parameters and converged (largest parameter shift was 0.41 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.073$$

$$R_w = \sqrt{\Sigma w(|Fo| - |Fc|)^2 / \Sigma w Fo^2} = 0.068$$

The standard deviation of an observation of unit weight⁴ was 4.57. The weighting scheme was based on counting statistics and included a factor ($p = 0.008$) 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 0.32 and -0.25 $e^-/\text{\AA}^3$, respectively.

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

References

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

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

$p = \text{p-factor}$

(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).

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(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 ₁₄ H ₁₇ NS ₆
Formula Weight	391.66
Crystal Color, Habit	yellow, plate
Crystal Dimensions	0.87 X 0.20 X 0.03 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2 θ range)	22 (50.1 - 57.1°)
Omega Scan Peak Width at Half-height	0.23°
Lattice Parameters	$a = 8.626(3) \text{ \AA}$ $b = 10.507(3) \text{ \AA}$ $c = 20.251(2) \text{ \AA}$ $\beta = 94.72(2)^\circ$
	$V = 1829.2(7) \text{ \AA}^3$
Space Group	P2 ₁ /a (#14)
Z value	4
D _{calc}	1.422 g/cm ³
F ₀₀₀	816.00
$\mu(\text{CuK}\alpha)$	68.32 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku AFC6S
Radiation	CuK α ($\lambda = 1.54178 \text{ \AA}$) graphite monochromated

Take-off Angle	6.0°
Detector Aperture	6.0 mm horizontal 6.0 mm vertical
Crystal to Detector Distance	258 mm
Voltage, Current	40kV, 40mA
Temperature	23.0°C
Scan Type	ω -2 θ
Scan Rate	4.0°/min (in ω)
Scan Width	(1.15 + 0.30 tan θ)°
2 θ_{max}	125.8°
No. of Reflections Measured	Total: 2887 Unique: 2658 ($R_{int} = 0.019$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.3558 - 0.9971)

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	0.0080
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 3.00\sigma(I)$)	1551
No. Variables	190
Reflection/Parameter Ratio	8.16
Residuals: R; Rw	0.073 ; 0.068
Residuals: R1	0.073
No. of Reflections to calc R1	1551

Goodness of Fit Indicator	4.57
Max Shift/Error in Final Cycle	0.41
Maximum peak in Final Diff. Map	$0.32\ e^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.25\ e^-/\text{\AA}^3$

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

atom	x	y	z	B_{eq}
S(1)	0.3411(3)	0.2523(3)	0.1412(1)	5.31(7)
S(2)	0.1237(3)	0.4699(3)	0.1114(1)	5.47(8)
S(3)	0.3113(4)	0.2920(3)	0.2972(1)	6.75(9)
S(4)	0.0869(4)	0.4974(3)	0.2689(2)	7.46(10)
S(5)	0.3812(6)	0.3640(5)	0.4392(2)	12.2(2)
S(6)	0.1324(6)	0.6083(5)	0.4062(2)	13.3(2)
N(1)	0.370(1)	0.3888(9)	-0.0396(5)	5.9(3)
C(1)	0.478(2)	0.374(2)	-0.2402(10)	15.0(8)
C(2)	0.359(2)	0.417(3)	-0.235(1)	17.3(9)
C(3)	0.311(1)	0.360(1)	-0.1572(6)	8.3(4)
C(4)	0.436(1)	0.406(1)	-0.1065(5)	6.9(4)
C(5)	0.425(1)	0.2988(10)	0.0074(5)	4.9(3)
C(6)	0.258(1)	0.467(1)	-0.0157(5)	5.5(3)
C(7)	0.343(1)	0.3212(9)	0.0619(4)	4.1(3)
C(8)	0.242(1)	0.4248(9)	0.0485(5)	4.3(3)
C(9)	0.224(1)	0.3730(9)	0.1713(5)	4.4(3)
C(10)	0.208(1)	0.3863(9)	0.2355(5)	5.2(3)
C(11)	0.282(1)	0.397(1)	0.3633(6)	7.2(4)
C(12)	0.183(2)	0.488(1)	0.3503(6)	7.6(4)
C(13)	0.575(2)	0.388(2)	0.4244(6)	13.0(6)
C(14)	-0.056(2)	0.592(2)	0.4076(9)	19.5(9)

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

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	0.088(2)	0.057(2)	0.055(2)	0.014(2)	0.000(2)	0.002(2)
S(2)	0.064(2)	0.071(2)	0.071(2)	0.013(2)	-0.005(2)	0.001(2)
S(3)	0.101(3)	0.092(2)	0.063(2)	0.007(2)	0.000(2)	0.004(2)
S(4)	0.088(3)	0.112(3)	0.083(2)	0.018(2)	0.008(2)	-0.025(2)
S(5)	0.183(5)	0.224(5)	0.053(2)	0.003(4)	-0.009(3)	0.004(3)
S(6)	0.171(5)	0.215(5)	0.123(3)	0.004(4)	0.034(3)	-0.089(4)
N(1)	0.069(7)	0.078(7)	0.077(7)	-0.002(6)	0.001(6)	0.003(6)
C(1)	0.19(2)	0.24(2)	0.14(2)	0.00(2)	0.00(2)	-0.04(2)
C(2)	0.07(1)	0.45(4)	0.14(2)	0.06(2)	0.01(1)	-0.10(2)
C(3)	0.09(1)	0.16(1)	0.066(8)	-0.020(9)	0.007(8)	-0.008(9)
C(4)	0.084(9)	0.14(1)	0.044(6)	-0.031(8)	0.009(7)	0.007(8)
C(5)	0.059(8)	0.072(8)	0.053(6)	-0.007(6)	-0.005(6)	0.004(7)
C(6)	0.066(8)	0.074(9)	0.067(8)	-0.012(7)	-0.005(7)	0.003(7)
C(7)	0.061(8)	0.045(6)	0.046(6)	0.004(6)	-0.005(6)	0.001(6)
C(8)	0.045(7)	0.056(7)	0.061(7)	-0.004(6)	-0.007(6)	-0.001(6)
C(9)	0.045(7)	0.063(7)	0.059(7)	0.004(6)	-0.002(6)	-0.003(6)
C(10)	0.061(8)	0.071(8)	0.066(7)	0.007(6)	-0.001(6)	-0.014(7)
C(11)	0.10(1)	0.12(1)	0.056(8)	0.010(9)	0.008(8)	-0.009(8)
C(12)	0.10(1)	0.12(1)	0.064(8)	-0.007(9)	0.013(8)	-0.029(9)
C(13)	0.09(1)	0.30(2)	0.10(1)	0.07(1)	-0.008(9)	-0.03(1)
C(14)	0.14(2)	0.41(3)	0.19(2)	0.04(2)	0.03(2)	-0.14(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(Å)

atom	atom	distance	atom	atom	distance
S(1)	C(7)	1.763(9)	S(1)	C(9)	1.761(10)
S(2)	C(8)	1.758(10)	S(2)	C(9)	1.755(10)
S(3)	C(10)	1.78(1)	S(3)	C(11)	1.77(1)
S(4)	C(10)	1.738(10)	S(4)	C(12)	1.79(1)
S(5)	C(11)	1.73(1)	S(5)	C(13)	1.74(1)
S(6)	C(12)	1.77(1)	S(6)	C(14)	1.63(2)
N(1)	C(4)	1.52(1)	N(1)	C(5)	1.40(1)
N(1)	C(6)	1.39(1)	C(1)	C(2)	1.13(2)
C(2)	C(3)	1.77(3)	C(3)	C(4)	1.51(1)
C(5)	C(7)	1.38(1)	C(6)	C(8)	1.39(1)
C(7)	C(8)	1.41(1)	C(9)	C(10)	1.33(1)
C(11)	C(12)	1.30(1)			

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(7)	S(1)	C(9)	93.9(5)	C(8)	S(2)	C(9)	93.6(5)
C(10)	S(3)	C(11)	95.2(5)	C(10)	S(4)	C(12)	94.3(6)
C(11)	S(5)	C(13)	103.3(6)	C(12)	S(6)	C(14)	103.6(8)
C(4)	N(1)	C(5)	123.7(10)	C(4)	N(1)	C(6)	124.3(10)
C(5)	N(1)	C(6)	111.8(9)	C(1)	C(2)	C(3)	102(2)
C(2)	C(3)	C(4)	106(1)	N(1)	C(4)	C(3)	105.9(9)
N(1)	C(5)	C(7)	105.1(9)	N(1)	C(6)	C(8)	105.3(9)
S(1)	C(7)	C(5)	134.6(9)	S(1)	C(7)	C(8)	116.1(8)
C(5)	C(7)	C(8)	109.2(9)	S(2)	C(8)	C(6)	134.4(9)
S(2)	C(8)	C(7)	117.1(8)	C(6)	C(8)	C(7)	108.4(9)
S(1)	C(9)	S(2)	116.3(6)	S(1)	C(9)	C(10)	121.6(8)
S(2)	C(9)	C(10)	122.0(8)	S(3)	C(10)	S(4)	112.5(6)
S(3)	C(10)	C(9)	122.9(8)	S(4)	C(10)	C(9)	124.6(8)
S(3)	C(11)	S(5)	117.3(8)	S(3)	C(11)	C(12)	116.0(10)
S(5)	C(11)	C(12)	126(1)	S(4)	C(12)	S(6)	115.4(8)
S(4)	C(12)	C(11)	118.4(10)	S(6)	C(12)	C(11)	126(1)

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
S(1)	C(7)	C(5)	N(1)	-179.7(8)	S(1)	C(7)	C(8)	S(2)	-1(1)
S(1)	C(7)	C(8)	C(6)	179.9(7)	S(1)	C(9)	S(2)	C(8)	-17.2(6)
S(1)	C(9)	C(10)	S(3)	4(1)	S(1)	C(9)	C(10)	S(4)	-176.6(5)
S(2)	C(8)	C(6)	N(1)	-179.0(8)	S(2)	C(8)	C(7)	C(5)	179.7(7)
S(2)	C(9)	S(1)	C(7)	16.8(6)	S(2)	C(9)	C(10)	S(3)	-178.4(5)
S(2)	C(9)	C(10)	S(4)	0(1)	S(3)	C(10)	S(4)	C(12)	17.8(7)
S(3)	C(11)	S(5)	C(13)	66.6(10)	S(3)	C(11)	C(12)	S(4)	0(1)
S(3)	C(11)	C(12)	S(6)	-179.2(7)	S(4)	C(10)	S(3)	C(11)	-17.9(7)
S(4)	C(12)	S(6)	C(14)	57(1)	S(4)	C(12)	C(11)	S(5)	-176.4(7)
S(5)	C(11)	S(3)	C(10)	-172.3(8)	S(5)	C(11)	C(12)	S(6)	4(2)
S(6)	C(12)	S(4)	C(10)	168.2(7)	N(1)	C(4)	C(3)	C(2)	164(1)
N(1)	C(5)	C(7)	C(8)	0(1)	N(1)	C(6)	C(8)	C(7)	0(1)
C(1)	C(2)	C(3)	C(4)	59(2)	C(3)	C(4)	N(1)	C(5)	110(1)
C(3)	C(4)	N(1)	C(6)	-74(1)	C(4)	N(1)	C(5)	C(7)	175.9(9)
C(4)	N(1)	C(6)	C(8)	-175.4(9)	C(5)	N(1)	C(6)	C(8)	0(1)
C(5)	C(7)	S(1)	C(9)	169(1)	C(5)	C(7)	C(8)	C(6)	0(1)
C(6)	N(1)	C(5)	C(7)	0(1)	C(6)	C(8)	S(2)	C(9)	-170(1)
C(7)	S(1)	C(9)	C(10)	-165.9(9)	C(7)	C(8)	S(2)	C(9)	10.9(8)
C(8)	S(2)	C(9)	C(10)	165.4(9)	C(8)	C(7)	S(1)	C(9)	-9.0(8)
C(9)	C(10)	S(3)	C(11)	161.2(10)	C(9)	C(10)	S(4)	C(12)	-161(1)
C(10)	S(3)	C(11)	C(12)	10(1)	C(10)	S(4)	C(12)	C(11)	-11(1)
C(11)	C(12)	S(6)	C(14)	-122(1)	C(12)	C(11)	S(5)	C(13)	-116(1)

Table 6. Non-bonded Contacts out to 4.23 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
S(1)	S(2)	3.464(4)	4	S(1)	C(9)	3.560(10)	4
S(1)	C(10)	3.84(1)	4	S(1)	C(6)	3.98(1)	54502
S(1)	S(4)	4.143(4)	4	S(1)	C(4)	4.16(1)	66503
S(2)	C(6)	3.74(1)	56503	S(2)	C(5)	3.84(1)	45504
S(2)	C(7)	3.977(10)	45504	S(2)	C(4)	4.03(1)	66503
S(2)	C(5)	4.21(1)	2	S(2)	C(3)	4.22(1)	2
S(3)	C(13)	3.90(1)	45504	S(3)	S(4)	3.931(5)	4
S(3)	C(1)	4.16(2)	66503	S(3)	C(10)	4.18(1)	4
S(4)	C(2)	3.96(2)	56503	S(4)	C(1)	4.03(2)	2
S(4)	C(1)	4.08(2)	66503	S(4)	C(3)	4.23(1)	56503
S(5)	C(13)	3.73(1)	45504	S(5)	C(13)	3.80(1)	66603
S(5)	S(6)	4.135(6)	54602	S(5)	S(5)	4.195(9)	66603
S(6)	C(13)	4.09(1)	66603	S(6)	C(14)	4.14(2)	56504
N(1)	N(1)	3.53(2)	66503	N(1)	C(6)	3.63(1)	66503
N(1)	C(5)	3.76(1)	66503	N(1)	C(8)	3.89(1)	66503
N(1)	C(4)	3.92(1)	66503	N(1)	C(7)	3.97(1)	66503
C(1)	C(10)	3.70(2)	66503	C(1)	C(9)	3.88(2)	66503
C(1)	C(3)	4.04(2)	4	C(1)	C(12)	4.09(2)	66503
C(1)	C(11)	4.14(3)	66503	C(2)	C(14)	4.19(3)	56503
C(4)	C(8)	3.43(1)	66503	C(4)	C(7)	3.52(1)	66503
C(4)	C(6)	3.71(1)	66503	C(4)	C(5)	3.83(2)	66503
C(4)	C(9)	4.05(1)	66503	C(5)	C(8)	3.64(1)	4
C(5)	C(6)	3.67(1)	66503	C(5)	C(6)	3.84(1)	54502
C(5)	C(7)	3.89(1)	4	C(5)	C(6)	4.06(1)	4

Table 6. Non-bonded Contacts out to 4.23 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(6)	C(7)	3.92(1)	2	C(6)	C(6)	4.22(2)	66503
C(11)	C(13)	3.74(2)	45504	C(13)	C(14)	3.88(3)	65501
C(13)	C(13)	4.16(3)	66603				

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

Table 7. Least Squares Planes

Plane number 1

Atoms defining plane	Distance
S(1)	0.000(3)
S(2)	-0.001(3)
C(5)	0.000(10)
C(6)	0.00(1)
C(7)	-0.006(9)
C(8)	0.010(9)
N(1)	-0.004(8)

Plane number 2

Atoms defining plane	Distance
S(1)	-0.031(3)
S(2)	0.024(3)
S(3)	0.035(3)
S(4)	-0.040(3)
C(9)	0.019(9)
C(10)	0.00(1)

Plane number 3

Atoms defining plane	Distance
S(3)	0.016(3)
S(4)	-0.020(3)
S(5)	-0.035(5)
S(6)	0.037(5)
C(11)	0.02(1)
C(12)	0.01(1)

Summary

plane	mean deviation	χ^2
1	0.0036	2.2
2	0.0249	412.0
3	0.0237	159.6

Dihedral angles between planes (°)

plane	1	2
2	17.63	
3	36.96	19.33