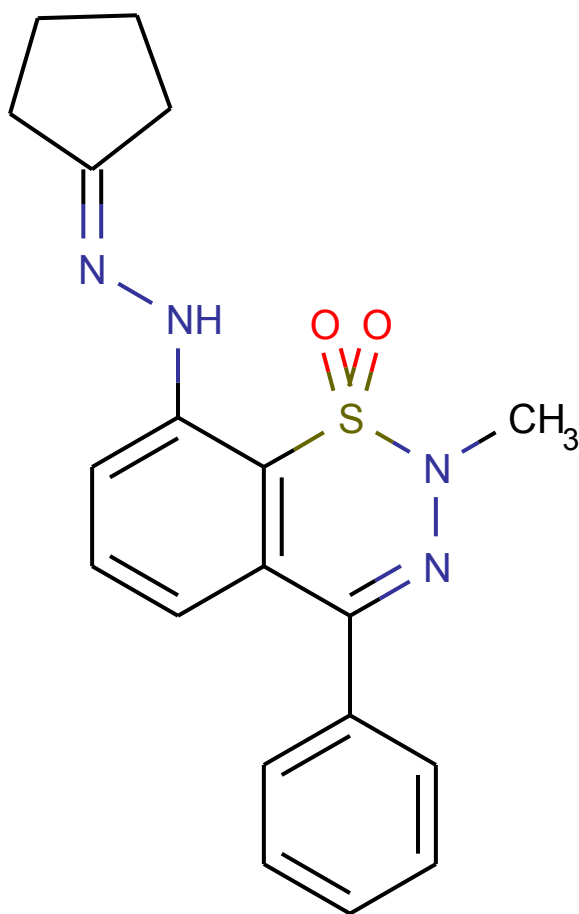


143675

PGY0789_1A

Submitted by: Pusztai Gyongyver
Operator: Dancso Andras

X-ray Structure Report



November 28, 2024



Fig. 1. The crystal

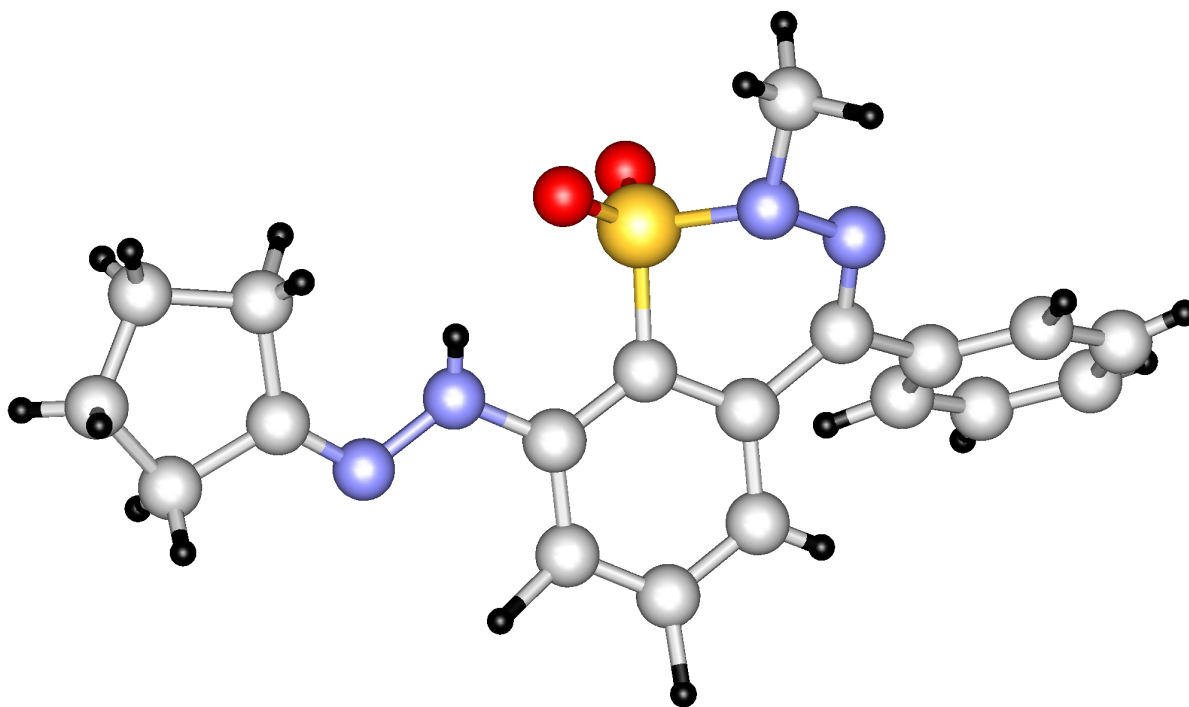


Fig. 2. The molecule (some hydrogens were generated by the software)

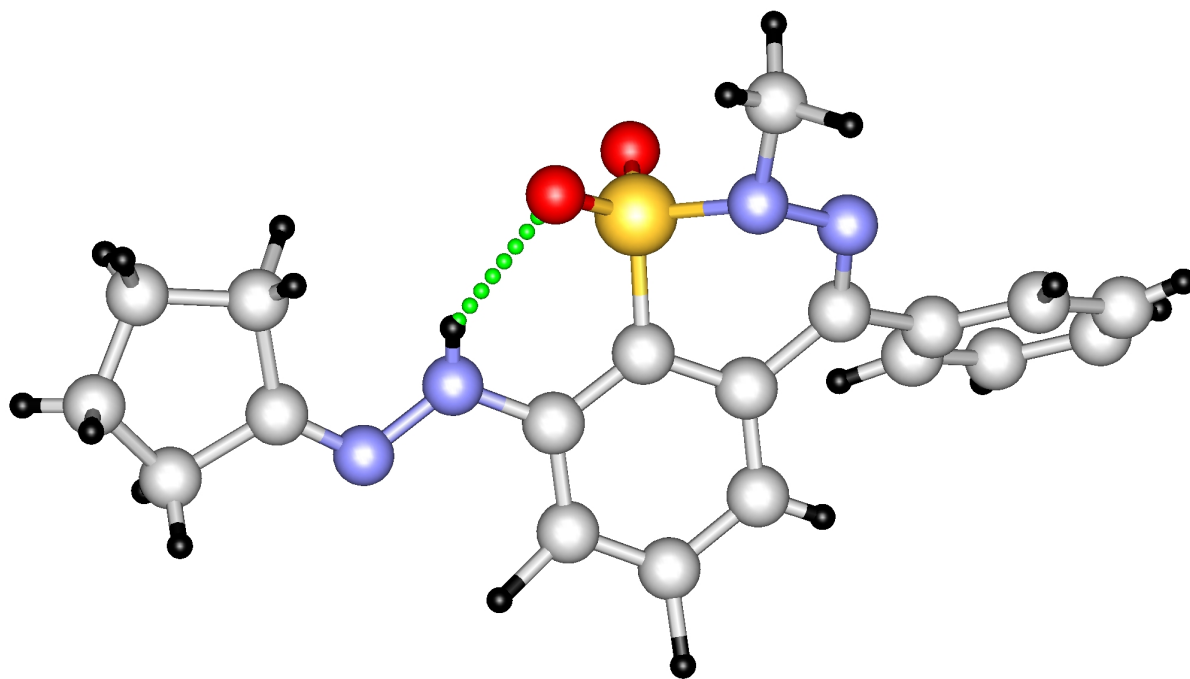


Fig. 3. Hydrogen bond

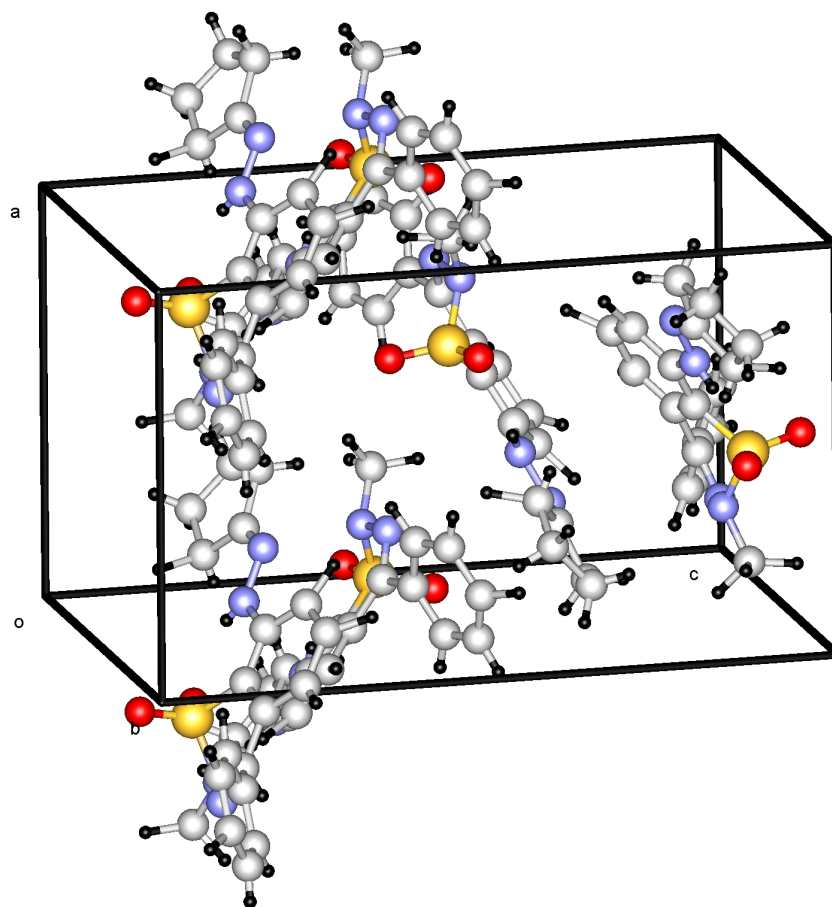


Fig. 4. Packing

Experimental

Data Collection

A colorless chunk crystal of $C_{19}H_{20}N_4O_2S$ having approximate dimensions of 0.66 x 0.20 x 0.14 mm was mounted on a cactus needle. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Cu-K α radiation.

Indexing was performed from 4 oscillations that were exposed for 60 seconds. The crystal-to-detector distance was 127.40 mm.

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

$$\begin{aligned}a &= 10.0402(4) \text{ \AA} \\b &= 10.9583(4) \text{ \AA} \\c &= 16.5846(8) \text{ \AA} \\V &= 1824.70(13) \text{ \AA}^3\end{aligned}$$

For $Z = 4$ and F.W. = 368.45, the calculated density is 1.341 g/cm³. The systematic absences of:

$$\begin{aligned}h00: h \pm 2n \\0k0: k \pm 2n \\00l: l \pm 2n\end{aligned}$$

uniquely determine the space group to be:

$$P2_12_12_1 \text{ (\#19)}$$

The data were collected at a temperature of $20 \pm 1^\circ\text{C}$ to a maximum 2θ value of 143.2° . A total of 180 oscillation images were collected. A sweep of data was done using ω scans from 20.0 to 200.0° in 5.0° step, at $\chi=0.0^\circ$ and $\phi = 0.0^\circ$. The exposure rate was 36.0 [sec./ $^\circ$]. A second sweep was performed using ω scans from 20.0 to 200.0° in 5.0° step, at $\chi=54.0^\circ$ and $\phi = 0.0^\circ$. The exposure rate was 36.0 [sec./ $^\circ$]. Another sweep was performed using ω scans from 20.0 to 200.0° in 5.0° step, at $\chi=54.0^\circ$ and $\phi = 90.0^\circ$. The exposure rate was 36.0 [sec./ $^\circ$]. Another sweep was performed using ω scans from 20.0 to 200.0° in 5.0° step, at $\chi=54.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was 36.0 [sec./ $^\circ$]. Another sweep was performed using ω scans from 20.0 to 200.0° in 5.0° step, at $\chi=54.0^\circ$ and $\phi = 270.0^\circ$. The exposure rate was 36.0 [sec./ $^\circ$]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 21313 reflections that were collected, 3511 were unique ($R_{\text{int}} = 0.053$).

The linear absorption coefficient, μ , for Cu-K α radiation is 17.519 cm⁻¹. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.537 to 0.776. 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. Some hydrogen atoms were refined isotropically and the rest were refined using the riding model. The final cycle of full-matrix least-squares refinement³ on F was based on 17378 observed reflections ($I > 2.00\sigma(I)$) and 294 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.0504$$

$$R_w = [\Sigma w (|F_o| - |F_c|)^2 / \Sigma w F_o^2]^{1/2} = 0.0519$$

The standard deviation of an observation of unit weight⁴ was 2.96. Unit weights were used. Plots of $\Sigma w (|F_o| - |F_c|)^2$ versus $|F_o|$, 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 5.04 and -5.09 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁵. Anomalous dispersion effects were included in Fcalc⁶; 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 Hubbell⁸. All calculations were performed using the CrystalStructure^{9,10} crystallographic software package.

References

- (1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.
- (2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$$\sum w(|F_o| - |F_c|)^2 \quad \text{where } w = \text{Least Squares weights.}$$

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

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

where: N_o = number of observations

N_v = number of variables

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(9) CrystalStructure 3.7.0: Crystal Structure Analysis Package, Rigaku and Rigaku/MSK (2000-2005). 9009 New Trails Dr. The Woodlands TX 77381 USA.

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$\text{C}_{19}\text{H}_{20}\text{N}_4\text{O}_2\text{S}$
Formula Weight	368.45
Crystal Color, Habit	colorless, chunk
Crystal Dimensions	0.66 X 0.20 X 0.14 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 60.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 10.0402(4) \text{ \AA}$ $b = 10.9583(4) \text{ \AA}$ $c = 16.5846(8) \text{ \AA}$ $V = 1824.70(13) \text{ \AA}^3$
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.341 g/cm^3
F_{000}	776.00
$\mu(\text{CuK}\alpha)$	17.519 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	CuK α (λ = 1.54187 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	180 exposures
ω oscillation Range (χ =0.0, ϕ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	36.0 sec./ $^{\circ}$
ω oscillation Range (χ =54.0, ϕ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	36.0 sec./ $^{\circ}$
ω oscillation Range (χ =54.0, ϕ =90.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	36.0 sec./ $^{\circ}$
ω oscillation Range (χ =54.0, ϕ =180.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	36.0 sec./ $^{\circ}$
ω oscillation Range (χ =54.0, ϕ =270.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	36.0 sec./ $^{\circ}$
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\text{max}}$	143.2 $^{\circ}$
No. of Reflections Measured	Total: 21313 Unique: 3511 (R_{int} = 0.053)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.537 - 0.776)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\Sigma w (Fo - Fc)^2$
Least Squares Weights	1
$2\theta_{\text{max}}$ cutoff	143.2 $^{\circ}$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 2.00\sigma(I)$)	17378
No. Variables	294
Reflection/Parameter Ratio	59.11
Residuals: R ($I > 2.00\sigma(I)$)	0.0504
Residuals: Rw ($I > 2.00\sigma(I)$)	0.0519
Goodness of Fit Indicator	2.959
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	5.04 e $^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	-5.09 e $^{-}/\text{\AA}^3$

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

atom	x	y	z	B_{eq}
S(1)	-0.08530(6)	0.84113(5)	0.07196(4)	4.143(14)
O(2)	-0.05504(14)	0.86907(12)	-0.01063(9)	4.87(4)
O(3)	-0.08526(16)	0.71556(12)	0.09528(9)	5.37(4)
N(4)	-0.24639(17)	1.02471(14)	0.08310(11)	3.93(4)
N(5)	-0.23158(18)	0.89895(14)	0.09964(11)	4.25(5)
N(6)	0.2804(2)	0.7192(2)	0.19927(12)	5.26(5)
N(7)	0.1652(2)	0.7622(2)	0.16304(14)	5.35(6)
C(8)	-0.1631(2)	1.2246(2)	0.08012(14)	4.16(6)
C(9)	0.1268(2)	0.8812(2)	0.17411(14)	4.17(6)
C(10)	-0.0242(2)	1.0511(2)	0.14269(13)	3.80(5)
C(11)	0.1654(2)	1.0823(2)	0.23003(17)	5.44(7)
C(12)	0.3005(2)	0.6054(2)	0.19040(16)	4.77(7)
C(13)	-0.2863(2)	1.2815(2)	0.08598(18)	5.30(7)
C(14)	-0.1474(2)	1.09368(19)	0.10326(13)	3.66(5)
C(15)	0.0146(2)	0.9281(2)	0.13570(12)	3.78(5)
C(16)	0.2032(2)	0.9616(2)	0.22111(16)	4.98(7)
C(17)	0.4257(2)	0.5452(2)	0.22257(14)	6.10(8)
C(18)	-0.3005(3)	1.3999(2)	0.0565(2)	6.85(9)
C(19)	-0.1952(3)	1.4612(2)	0.0203(2)	6.79(9)
C(20)	0.0522(2)	1.1268(2)	0.19010(17)	4.94(7)
C(21)	-0.0584(2)	1.2876(2)	0.04478(18)	5.60(7)
C(22)	-0.3503(2)	0.8294(2)	0.0734(2)	5.69(8)
C(23)	0.2166(3)	0.5111(2)	0.1463(2)	5.79(9)
C(24)	-0.0751(3)	1.4043(2)	0.01528(18)	6.80(9)
C(25)	0.2981(3)	0.3969(2)	0.1477(2)	11.35(13)
C(26)	0.4032(3)	0.4122(2)	0.2072(2)	8.41(10)
H(1)	0.283(2)	0.9183(18)	0.2694(12)	6.4(5)
H(2)	0.209(2)	1.132(2)	0.2888(17)	13.1(10)
H(3)	0.0297(16)	1.2057(16)	0.1923(10)	2.8(4)
H(4)	-0.3617(16)	1.2384(15)	0.1136(10)	3.0(4)
H(5)	-0.3813(18)	1.4252(16)	0.0549(11)	3.2(5)
H(6)	-0.216(2)	1.538(2)	-0.0227(15)	10.6(8)
H(7)	0.031(2)	1.2521(18)	0.0461(12)	6.2(6)
H(8)	-0.431(2)	0.869(2)	0.1131(16)	10.8(10)
H(9)	-0.3507(17)	0.7534(16)	0.0917(11)	3.1(5)
H(10)	-0.362(2)	0.826(2)	0.0035(16)	10.1(9)
H(11)	0.1106(16)	0.7184(15)	0.1445(10)	1.8(4)

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

atom	x	y	z	B_{eq}
H(12)	0.1464(18)	0.5044(19)	0.1626(13)	3.3(6)
H(13)	0.196(2)	0.5404(19)	0.0753(14)	6.7(6)
H(14)	0.5025	0.5740	0.1950	7.31
H(15)	0.4352	0.5601	0.2787	7.31
H(16)	-0.0024	1.4489	-0.0065	8.15
H(17)	0.2443	0.3276	0.1588	13.63
H(18)	0.3378	0.3881	0.0960	13.69
H(19)	0.4820	0.3724	0.1895	10.12
H(20)	0.3734	0.3760	0.2560	10.16

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

Table 2. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	0.0601(3)	0.0402(2)	0.0571(4)	0.0024(3)	-0.0095(3)	-0.0042(3)
O(2)	0.0714(11)	0.0628(10)	0.0508(10)	0.0116(8)	-0.0053(9)	-0.0094(8)
O(3)	0.0786(10)	0.0382(8)	0.0872(13)	-0.0057(9)	-0.0197(11)	0.0014(8)
N(4)	0.0474(12)	0.0436(10)	0.0582(13)	-0.0005(9)	0.0004(10)	-0.0043(10)
N(5)	0.0495(11)	0.0429(11)	0.0690(15)	-0.0076(9)	0.0006(11)	0.0032(10)
N(6)	0.0600(13)	0.0598(13)	0.0802(17)	0.0105(13)	-0.0204(13)	-0.0029(13)
N(7)	0.0630(15)	0.0526(15)	0.0876(19)	0.0017(12)	-0.0285(14)	-0.0053(13)
C(8)	0.0550(14)	0.0465(13)	0.0565(17)	0.0011(12)	-0.0021(14)	-0.0104(13)
C(9)	0.0552(16)	0.0488(14)	0.0546(17)	0.0039(12)	-0.0041(13)	0.0011(13)
C(10)	0.0511(14)	0.0437(14)	0.0495(16)	-0.0018(11)	-0.0021(12)	-0.0044(12)
C(11)	0.0729(19)	0.0704(19)	0.063(2)	-0.0015(16)	-0.0184(16)	-0.0115(15)
C(12)	0.0591(17)	0.0649(18)	0.0572(19)	0.0107(15)	-0.0024(15)	0.0071(15)
C(13)	0.0663(18)	0.0526(15)	0.083(2)	-0.0007(15)	0.0125(18)	0.0002(16)
C(14)	0.0540(15)	0.0385(13)	0.0464(16)	0.0016(11)	0.0016(12)	-0.0081(11)
C(15)	0.0515(14)	0.0438(13)	0.0482(15)	-0.0036(11)	-0.0063(12)	-0.0040(12)
C(16)	0.0655(18)	0.0610(18)	0.0628(19)	0.0072(15)	-0.0212(15)	-0.0056(14)
C(17)	0.0776(19)	0.0762(19)	0.078(2)	0.0210(18)	-0.0134(17)	0.0044(16)
C(18)	0.079(2)	0.058(2)	0.123(3)	0.0222(18)	-0.011(2)	-0.0044(19)
C(19)	0.107(2)	0.0490(18)	0.102(2)	-0.0063(19)	-0.022(2)	0.0017(18)
C(20)	0.0712(19)	0.0451(16)	0.071(2)	0.0006(14)	-0.0033(15)	-0.0136(14)
C(21)	0.0576(18)	0.0549(16)	0.100(2)	-0.0006(14)	-0.0011(16)	0.0080(15)
C(22)	0.0733(19)	0.0523(17)	0.090(2)	-0.0188(16)	-0.014(2)	0.008(2)
C(23)	0.059(2)	0.0601(19)	0.101(3)	0.0061(18)	-0.006(2)	-0.0024(18)
C(24)	0.092(2)	0.0609(19)	0.106(2)	-0.0172(18)	-0.010(2)	0.0189(17)
C(25)	0.131(3)	0.065(2)	0.234(4)	0.026(2)	-0.075(3)	-0.004(2)
C(26)	0.110(2)	0.085(2)	0.124(3)	0.043(2)	0.006(2)	0.005(2)

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

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
S(1)	O(2)	1.4360(16)	S(1)	O(3)	1.4294(14)
S(1)	N(5)	1.6641(18)	S(1)	C(15)	1.742(2)
N(4)	N(5)	1.413(2)	N(4)	C(14)	1.293(2)
N(5)	C(22)	1.481(3)	N(6)	N(7)	1.386(3)
N(6)	C(12)	1.271(3)	N(7)	C(9)	1.372(3)
N(7)	H(11)	0.790(16)	C(8)	C(13)	1.389(3)
C(8)	C(14)	1.493(3)	C(8)	C(21)	1.387(3)
C(9)	C(15)	1.392(3)	C(9)	C(16)	1.404(3)
C(10)	C(14)	1.475(3)	C(10)	C(15)	1.407(3)
C(10)	C(20)	1.376(3)	C(11)	C(16)	1.384(3)
C(11)	C(20)	1.403(3)	C(11)	H(2)	1.20(2)
C(12)	C(17)	1.517(3)	C(12)	C(23)	1.520(4)
C(13)	C(18)	1.394(4)	C(13)	H(4)	1.003(16)
C(16)	H(1)	1.23(2)	C(17)	C(26)	1.496(3)
C(17)	H(14)	0.950	C(17)	H(15)	0.950
C(18)	C(19)	1.389(5)	C(18)	H(5)	0.858(18)
C(19)	C(24)	1.359(4)	C(19)	H(6)	1.13(2)
C(20)	H(3)	0.895(17)	C(21)	C(24)	1.380(3)
C(21)	H(7)	0.98(2)	C(22)	H(8)	1.13(2)
C(22)	H(9)	0.886(18)	C(22)	H(10)	1.16(2)
C(23)	C(25)	1.496(4)	C(23)	H(12)	0.758(19)
C(23)	H(13)	1.24(2)	C(24)	H(16)	0.950
C(25)	C(26)	1.454(5)	C(25)	H(17)	0.950
C(25)	H(18)	0.950	C(26)	H(19)	0.950
C(26)	H(20)	0.950			

Table 4. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	S(1)	O(3)	117.60(9)	O(2)	S(1)	N(5)	111.63(9)
O(2)	S(1)	C(15)	109.90(9)	O(3)	S(1)	N(5)	106.99(9)
O(3)	S(1)	C(15)	111.26(10)	N(5)	S(1)	C(15)	97.62(9)
N(5)	N(4)	C(14)	116.04(17)	S(1)	N(5)	N(4)	114.25(13)
S(1)	N(5)	C(22)	115.69(16)	N(4)	N(5)	C(22)	111.12(18)
N(7)	N(6)	C(12)	114.6(2)	N(6)	N(7)	C(9)	120.0(2)
N(6)	N(7)	H(11)	122.7(12)	C(9)	N(7)	H(11)	115.8(12)
C(13)	C(8)	C(14)	120.5(2)	C(13)	C(8)	C(21)	118.7(2)
C(14)	C(8)	C(21)	120.5(2)	N(7)	C(9)	C(15)	121.1(2)
N(7)	C(9)	C(16)	121.1(2)	C(15)	C(9)	C(16)	117.7(2)
C(14)	C(10)	C(15)	119.92(19)	C(14)	C(10)	C(20)	122.0(2)
C(15)	C(10)	C(20)	118.0(2)	C(16)	C(11)	C(20)	120.2(2)
C(16)	C(11)	H(2)	115.1(14)	C(20)	C(11)	H(2)	121.3(14)
N(6)	C(12)	C(17)	121.2(2)	N(6)	C(12)	C(23)	129.3(2)
C(17)	C(12)	C(23)	109.4(2)	C(8)	C(13)	C(18)	119.0(2)
C(8)	C(13)	H(4)	119.5(9)	C(18)	C(13)	H(4)	121.4(9)
N(4)	C(14)	C(8)	114.5(2)	N(4)	C(14)	C(10)	125.06(19)
C(8)	C(14)	C(10)	120.44(19)	S(1)	C(15)	C(9)	122.80(17)
S(1)	C(15)	C(10)	114.49(16)	C(9)	C(15)	C(10)	122.7(2)
C(9)	C(16)	C(11)	120.6(2)	C(9)	C(16)	H(1)	118.4(9)
C(11)	C(16)	H(1)	118.5(9)	C(12)	C(17)	C(26)	103.8(2)
C(12)	C(17)	H(14)	111.0	C(12)	C(17)	H(15)	110.6
C(26)	C(17)	H(14)	111.4	C(26)	C(17)	H(15)	110.5
H(14)	C(17)	H(15)	109.5	C(13)	C(18)	C(19)	121.6(3)
C(13)	C(18)	H(5)	114.1(12)	C(19)	C(18)	H(5)	123.4(12)
C(18)	C(19)	C(24)	118.6(2)	C(18)	C(19)	H(6)	119.8(13)
C(24)	C(19)	H(6)	118.0(13)	C(10)	C(20)	C(11)	120.8(2)
C(10)	C(20)	H(3)	117.8(11)	C(11)	C(20)	H(3)	121.4(11)
C(8)	C(21)	C(24)	121.3(2)	C(8)	C(21)	H(7)	119.2(12)
C(24)	C(21)	H(7)	119.2(12)	N(5)	C(22)	H(8)	102.1(14)
N(5)	C(22)	H(9)	112.7(11)	N(5)	C(22)	H(10)	112.9(12)
H(8)	C(22)	H(9)	98.9(18)	H(8)	C(22)	H(10)	121.5(18)
H(9)	C(22)	H(10)	108.1(17)	C(12)	C(23)	C(25)	104.9(2)
C(12)	C(23)	H(12)	114.3(16)	C(12)	C(23)	H(13)	112.0(10)
C(25)	C(23)	H(12)	114.9(16)	C(25)	C(23)	H(13)	108.8(10)
H(12)	C(23)	H(13)	102.1(19)	C(19)	C(24)	C(21)	120.8(2)
C(19)	C(24)	H(16)	118.0	C(21)	C(24)	H(16)	121.2

Table 4. Bond angles ($^{\circ}$) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(23)	C(25)	C(26)	108.1(2)	C(23)	C(25)	H(17)	111.1
C(23)	C(25)	H(18)	107.5	C(26)	C(25)	H(17)	111.9
C(26)	C(25)	H(18)	108.6	H(17)	C(25)	H(18)	109.5
C(17)	C(26)	C(25)	109.7(2)	C(17)	C(26)	H(19)	111.9
C(17)	C(26)	H(20)	108.0	C(25)	C(26)	H(19)	110.0
C(25)	C(26)	H(20)	107.5	H(19)	C(26)	H(20)	109.5

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

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)	S(1)	N(5)	N(4)	-51.96(16)	O(2)	S(1)	N(5)	C(22)	79.0(2)
O(2)	S(1)	C(15)	C(9)	-101.6(2)	O(2)	S(1)	C(15)	C(10)	75.93(18)
O(3)	S(1)	N(5)	N(4)	178.08(13)	O(3)	S(1)	N(5)	C(22)	-51.0(2)
O(3)	S(1)	C(15)	C(9)	30.4(2)	O(3)	S(1)	C(15)	C(10)	-152.04(16)
N(5)	S(1)	C(15)	C(9)	142.00(19)	N(5)	S(1)	C(15)	C(10)	-40.42(18)
C(15)	S(1)	N(5)	N(4)	63.03(15)	C(15)	S(1)	N(5)	C(22)	-166.05(19)
N(5)	N(4)	C(14)	C(8)	176.30(18)	N(5)	N(4)	C(14)	C(10)	-1.1(3)
C(14)	N(4)	N(5)	S(1)	-46.4(2)	C(14)	N(4)	N(5)	C(22)	-179.5(2)
N(7)	N(6)	C(12)	C(17)	176.7(2)	N(7)	N(6)	C(12)	C(23)	-0.2(4)
C(12)	N(6)	N(7)	C(9)	174.5(2)	N(6)	N(7)	C(9)	C(15)	177.3(2)
N(6)	N(7)	C(9)	C(16)	0.7(3)	C(13)	C(8)	C(14)	N(4)	38.4(3)
C(13)	C(8)	C(14)	C(10)	-144.0(2)	C(14)	C(8)	C(13)	C(18)	-173.8(2)
C(13)	C(8)	C(21)	C(24)	-0.4(4)	C(21)	C(8)	C(13)	C(18)	-0.3(4)
C(14)	C(8)	C(21)	C(24)	173.1(2)	C(21)	C(8)	C(14)	N(4)	-135.0(2)
C(21)	C(8)	C(14)	C(10)	42.6(3)	N(7)	C(9)	C(15)	S(1)	0.4(3)
N(7)	C(9)	C(15)	C(10)	-177.0(2)	N(7)	C(9)	C(16)	C(11)	178.2(2)
C(15)	C(9)	C(16)	C(11)	1.5(3)	C(16)	C(9)	C(15)	S(1)	177.09(18)
C(16)	C(9)	C(15)	C(10)	-0.3(3)	C(14)	C(10)	C(15)	S(1)	5.0(2)
C(14)	C(10)	C(15)	C(9)	-177.4(2)	C(15)	C(10)	C(14)	N(4)	22.7(3)
C(15)	C(10)	C(14)	C(8)	-154.6(2)	C(14)	C(10)	C(20)	C(11)	176.9(2)
C(20)	C(10)	C(14)	N(4)	-154.0(2)	C(20)	C(10)	C(14)	C(8)	28.7(3)
C(15)	C(10)	C(20)	C(11)	0.1(3)	C(20)	C(10)	C(15)	S(1)	-178.09(19)
C(20)	C(10)	C(15)	C(9)	-0.5(3)	C(16)	C(11)	C(20)	C(10)	1.1(4)
C(20)	C(11)	C(16)	C(9)	-1.9(4)	N(6)	C(12)	C(17)	C(26)	174.4(2)
N(6)	C(12)	C(23)	C(25)	173.3(3)	C(17)	C(12)	C(23)	C(25)	-4.0(3)
C(23)	C(12)	C(17)	C(26)	-8.1(3)	C(8)	C(13)	C(18)	C(19)	1.2(4)
C(12)	C(17)	C(26)	C(25)	17.8(3)	C(13)	C(18)	C(19)	C(24)	-1.4(5)
C(18)	C(19)	C(24)	C(21)	0.7(4)	C(8)	C(21)	C(24)	C(19)	0.2(3)
C(12)	C(23)	C(25)	C(26)	15.0(3)	C(23)	C(25)	C(26)	C(17)	-21.1(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 6. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
S(1)	H(2) ¹¹	3.48(2)	S(1)	H(10) ²¹	3.15(2)
S(1)	H(13) ³¹	3.53(2)	O(2)	C(12) ³¹	3.327(3)
O(2)	C(22) ²¹	3.168(3)	O(2)	C(23) ³¹	3.471(3)
O(2)	H(5) ⁴¹	2.943(18)	O(2)	H(6) ⁴¹	3.60(2)
O(2)	H(8) ²¹	3.35(2)	O(2)	H(9) ²¹	2.796(17)
O(2)	H(10) ²¹	2.89(2)	O(2)	H(13) ³¹	2.89(2)
O(2)	H(14) ³¹	3.174	O(2)	H(18) ³¹	3.200
O(3)	C(11) ¹¹	3.343(3)	O(3)	C(19) ⁵¹	3.246(3)
O(3)	H(2) ¹¹	2.46(2)	O(3)	H(3) ¹¹	3.568(17)
O(3)	H(6) ⁵¹	3.05(2)	O(3)	H(10) ²¹	2.82(2)
O(3)	H(16) ⁵¹	3.475	N(4)	H(13) ³¹	2.78(2)
N(4)	H(15) ⁶¹	2.999	N(4)	H(16) ⁷¹	2.882
N(4)	H(18) ³¹	3.233	N(4)	H(20) ⁶¹	3.377
N(5)	H(2) ¹¹	3.47(3)	N(5)	H(13) ³¹	3.06(2)
N(5)	H(15) ⁶¹	3.371	N(5)	H(16) ⁷¹	3.544
N(5)	H(20) ⁶¹	2.797	N(6)	H(3) ¹¹	3.598(17)
N(6)	H(4) ¹¹	3.216(17)	N(6)	H(19) ⁸¹	3.451
N(7)	C(20) ¹¹	3.591(3)	N(7)	H(3) ¹¹	3.157(17)
N(7)	H(6) ⁴¹	3.41(2)	N(7)	H(10) ²¹	2.94(2)
C(8)	H(1) ⁶¹	3.49(2)	C(8)	H(18) ³¹	3.172
C(9)	H(3) ¹¹	3.328(17)	C(9)	H(6) ⁴¹	3.09(2)
C(10)	H(5) ⁴¹	3.586(18)	C(10)	H(12) ⁶¹	3.49(2)
C(11)	O(3) ⁶¹	3.343(3)	C(11)	H(14) ⁸¹	3.560
C(11)	H(17) ⁹¹	3.041	C(12)	O(2) ²¹	3.327(3)
C(13)	C(21) ⁷¹	3.569(4)	C(13)	H(1) ⁶¹	2.83(2)
C(13)	H(7) ⁷¹	2.88(2)	C(13)	H(16) ⁷¹	3.580
C(13)	H(19) ¹⁰¹	3.058	C(14)	H(15) ⁶¹	3.509
C(14)	H(18) ³¹	3.314	C(15)	H(6) ⁴¹	3.31(2)
C(16)	H(6) ⁴¹	3.39(2)	C(16)	H(14) ⁸¹	3.491
C(17)	H(1) ¹¹¹	3.24(2)	C(17)	H(4) ¹¹	3.504(17)
C(17)	H(8) ¹¹	3.34(2)	C(18)	H(1) ⁶¹	2.90(2)
C(18)	H(7) ⁷¹	2.92(2)	C(18)	H(14) ¹⁰¹	3.582
C(18)	H(19) ¹⁰¹	3.118	C(19)	O(3) ⁹¹	3.246(3)
C(20)	N(7) ⁶¹	3.591(3)	C(20)	H(11) ⁶¹	3.347(17)
C(20)	H(12) ⁶¹	3.43(2)	C(20)	H(17) ⁹¹	2.972
C(21)	C(13) ⁴¹	3.569(4)	C(21)	H(4) ⁴¹	3.299(17)
C(21)	H(5) ⁴¹	3.366(18)	C(21)	H(18) ³¹	3.201

Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(22)	O(2) ^{3j}	3.168(3)	C(22)	H(2) ^{1j}	3.45(2)
C(22)	H(13) ^{3j}	2.89(2)	C(22)	H(16) ^{7j}	3.076
C(22)	H(20) ^{6j}	2.885	C(23)	O(2) ^{2j}	3.471(3)
C(23)	H(10) ^{2j}	3.16(2)	C(23)	H(16) ^{5j}	3.424
C(24)	H(4) ^{4j}	3.407(16)	C(24)	H(8) ^{4j}	3.58(2)
C(24)	H(12) ^{9j}	3.48(2)	C(24)	H(13) ^{9j}	3.26(2)
C(25)	H(3) ^{5j}	3.493(17)	C(25)	H(5) ^{12j}	3.581(18)
C(25)	H(7) ^{5j}	3.54(2)	C(26)	H(1) ^{11j}	3.18(2)
C(26)	H(4) ^{12j}	3.407(16)	C(26)	H(5) ^{12j}	3.329(18)
C(26)	H(8) ^{1j}	3.03(2)	H(1)	C(8) ^{1j}	3.49(2)
H(1)	C(13) ^{1j}	2.83(2)	H(1)	C(17) ^{8j}	3.24(2)
H(1)	C(18) ^{1j}	2.90(2)	H(1)	C(26) ^{8j}	3.18(2)
H(1)	H(4) ^{1j}	2.88(2)	H(1)	H(5) ^{1j}	3.08(2)
H(1)	H(14) ^{8j}	2.813	H(1)	H(15) ^{8j}	3.328
H(1)	H(19) ^{8j}	2.509	H(1)	H(20) ^{8j}	3.509
H(2)	S(1) ^{6j}	3.48(2)	H(2)	O(3) ^{6j}	2.46(2)
H(2)	N(5) ^{6j}	3.47(2)	H(2)	C(22) ^{6j}	3.45(2)
H(2)	H(9) ^{6j}	2.78(3)	H(2)	H(11) ^{6j}	3.52(3)
H(2)	H(14) ^{8j}	2.980	H(2)	H(17) ^{9j}	3.058
H(2)	H(20) ^{9j}	3.187	H(3)	O(3) ^{6j}	3.568(17)
H(3)	N(6) ^{6j}	3.598(17)	H(3)	N(7) ^{6j}	3.157(17)
H(3)	C(9) ^{6j}	3.328(17)	H(3)	C(25) ^{9j}	3.493(17)
H(3)	H(11) ^{6j}	3.05(2)	H(3)	H(12) ^{9j}	3.51(2)
H(3)	H(17) ^{9j}	2.595	H(4)	N(6) ^{6j}	3.216(17)
H(4)	C(17) ^{6j}	3.504(17)	H(4)	C(21) ^{7j}	3.299(17)
H(4)	C(24) ^{7j}	3.407(16)	H(4)	C(26) ^{10j}	3.407(16)
H(4)	H(1) ^{6j}	2.88(2)	H(4)	H(7) ^{7j}	2.86(2)
H(4)	H(15) ^{6j}	2.748	H(4)	H(16) ^{7j}	3.061
H(4)	H(18) ^{10j}	3.447	H(4)	H(19) ^{10j}	2.490
H(5)	O(2) ^{7j}	2.943(18)	H(5)	C(10) ^{7j}	3.586(18)
H(5)	C(21) ^{7j}	3.366(18)	H(5)	C(25) ^{10j}	3.581(18)
H(5)	C(26) ^{10j}	3.329(18)	H(5)	H(1) ^{6j}	3.08(2)
H(5)	H(7) ^{7j}	2.71(2)	H(5)	H(14) ^{10j}	3.070
H(5)	H(18) ^{10j}	2.930	H(5)	H(19) ^{10j}	2.684
H(6)	O(2) ^{7j}	3.60(2)	H(6)	O(3) ^{9j}	3.05(2)
H(6)	N(7) ^{7j}	3.41(2)	H(6)	C(9) ^{7j}	3.09(2)
H(6)	C(15) ^{7j}	3.31(2)	H(6)	C(16) ^{7j}	3.39(2)

Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
H(6)	H(8) ^{4j}	3.39(3)	H(6)	H(9) ^{9j}	3.32(3)
H(6)	H(10) ^{9j}	3.50(3)	H(7)	C(13) ^{4j}	2.88(2)
H(7)	C(18) ^{4j}	2.92(2)	H(7)	C(25) ^{9j}	3.54(2)
H(7)	H(4) ^{4j}	2.86(2)	H(7)	H(5) ^{4j}	2.71(2)
H(7)	H(12) ^{9j}	3.57(2)	H(7)	H(13) ^{9j}	3.60(2)
H(7)	H(17) ^{9j}	2.959	H(7)	H(18) ^{9j}	3.518
H(7)	H(18) ^{3j}	3.419	H(8)	O(2) ^{3j}	3.35(2)
H(8)	C(17) ^{6j}	3.34(2)	H(8)	C(24) ^{7j}	3.58(2)
H(8)	C(26) ^{6j}	3.03(2)	H(8)	H(6) ^{7j}	3.39(3)
H(8)	H(13) ^{3j}	3.52(3)	H(8)	H(14) ^{13j}	3.566
H(8)	H(15) ^{6j}	2.761	H(8)	H(16) ^{7j}	2.764
H(8)	H(19) ^{6j}	3.313	H(8)	H(20) ^{6j}	2.248
H(9)	O(2) ^{3j}	2.796(17)	H(9)	H(2) ^{1j}	2.78(3)
H(9)	H(6) ^{5j}	3.32(3)	H(9)	H(14) ^{13j}	2.996
H(9)	H(20) ^{6j}	2.870	H(10)	S(1) ^{3j}	3.15(2)
H(10)	O(2) ^{3j}	2.89(2)	H(10)	O(3) ^{3j}	2.82(2)
H(10)	N(7) ^{3j}	2.94(2)	H(10)	C(23) ^{3j}	3.16(2)
H(10)	H(6) ^{5j}	3.50(3)	H(10)	H(11) ^{3j}	2.52(3)
H(10)	H(12) ^{3j}	3.32(3)	H(10)	H(13) ^{3j}	2.05(3)
H(10)	H(16) ^{7j}	2.843	H(11)	C(20) ^{1j}	3.347(17)
H(11)	H(2) ^{1j}	3.52(3)	H(11)	H(3) ^{1j}	3.05(2)
H(11)	H(10) ^{2j}	2.52(3)	H(12)	C(10) ^{1j}	3.49(2)
H(12)	C(20) ^{1j}	3.43(2)	H(12)	C(24) ^{5j}	3.48(2)
H(12)	H(3) ^{5j}	3.51(2)	H(12)	H(7) ^{5j}	3.57(2)
H(12)	H(10) ^{2j}	3.32(3)	H(12)	H(16) ^{5j}	3.234
H(13)	S(1) ^{2j}	3.53(2)	H(13)	O(2) ^{2j}	2.89(2)
H(13)	N(4) ^{2j}	2.78(2)	H(13)	N(5) ^{2j}	3.06(2)
H(13)	C(22) ^{2j}	2.89(2)	H(13)	C(24) ^{5j}	3.26(2)
H(13)	H(7) ^{5j}	3.60(2)	H(13)	H(8) ^{2j}	3.52(3)
H(13)	H(10) ^{2j}	2.05(3)	H(13)	H(16) ^{5j}	2.610
H(14)	O(2) ^{2j}	3.174	H(14)	C(11) ^{11j}	3.560
H(14)	C(16) ^{11j}	3.491	H(14)	C(18) ^{12j}	3.582
H(14)	H(1) ^{11j}	2.813	H(14)	H(2) ^{11j}	2.980
H(14)	H(5) ^{12j}	3.070	H(14)	H(8) ^{14j}	3.566
H(14)	H(9) ^{14j}	2.996	H(15)	N(4) ^{1j}	2.999
H(15)	N(5) ^{1j}	3.371	H(15)	C(14) ^{1j}	3.509
H(15)	H(1) ^{11j}	3.328	H(15)	H(4) ^{1j}	2.748

Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
H(15)	H(8) ¹¹	2.761	H(15)	H(19) ⁸	3.561
H(16)	O(3) ⁹	3.475	H(16)	N(4) ⁴	2.882
H(16)	N(5) ⁴	3.544	H(16)	C(13) ⁴	3.580
H(16)	C(22) ⁴	3.076	H(16)	C(23) ⁹	3.424
H(16)	H(4) ⁴	3.061	H(16)	H(8) ⁴	2.764
H(16)	H(10) ⁴	2.843	H(16)	H(12) ⁹	3.234
H(16)	H(13) ⁹	2.610	H(17)	C(11) ⁵	3.041
H(17)	C(20) ⁵	2.972	H(17)	H(2) ⁵	3.058
H(17)	H(3) ⁵	2.595	H(17)	H(7) ⁵	2.959
H(18)	O(2) ²	3.200	H(18)	N(4) ²	3.233
H(18)	C(8) ²	3.172	H(18)	C(14) ²	3.314
H(18)	C(21) ²	3.201	H(18)	H(4) ¹²	3.447
H(18)	H(5) ¹²	2.930	H(18)	H(7) ⁵	3.518
H(18)	H(7) ²	3.419	H(19)	N(6) ¹¹	3.451
H(19)	C(13) ¹²	3.058	H(19)	C(18) ¹²	3.118
H(19)	H(1) ¹¹	2.509	H(19)	H(4) ¹²	2.490
H(19)	H(5) ¹²	2.684	H(19)	H(8) ¹¹	3.313
H(19)	H(15) ¹¹	3.561	H(20)	N(4) ¹¹	3.377
H(20)	N(5) ¹¹	2.797	H(20)	C(22) ¹¹	2.885
H(20)	H(1) ¹¹	3.509	H(20)	H(2) ⁵	3.187
H(20)	H(8) ¹¹	2.248	H(20)	H(9) ¹¹	2.870

Symmetry Operators:

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

Table 7. Intramolecular and Intermolecular Hydrogen bonds

D	H	A	D...A	D-H	H...A	D-H...A
N(7)	H(11)	O(3)	2.801(2)	0.790(16)	2.130(17)	142.9(16)

- Note) 1. The symmetry operations are applied to the acceptors.
 2. Estimated standard deviations (esd's) are shown in the parentheses.
 They are not calculated when all atoms have an esd=0.0.