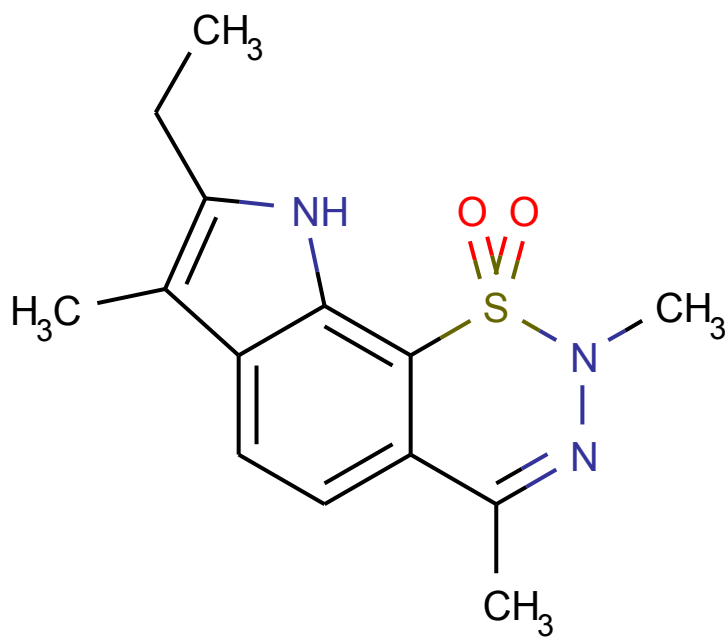


**142611**

**PGY0686\_1B**

Submitted by Pusztai Gyongyver  
Operator: Dancso Andras

X-ray Structure Report



January 16, 2025

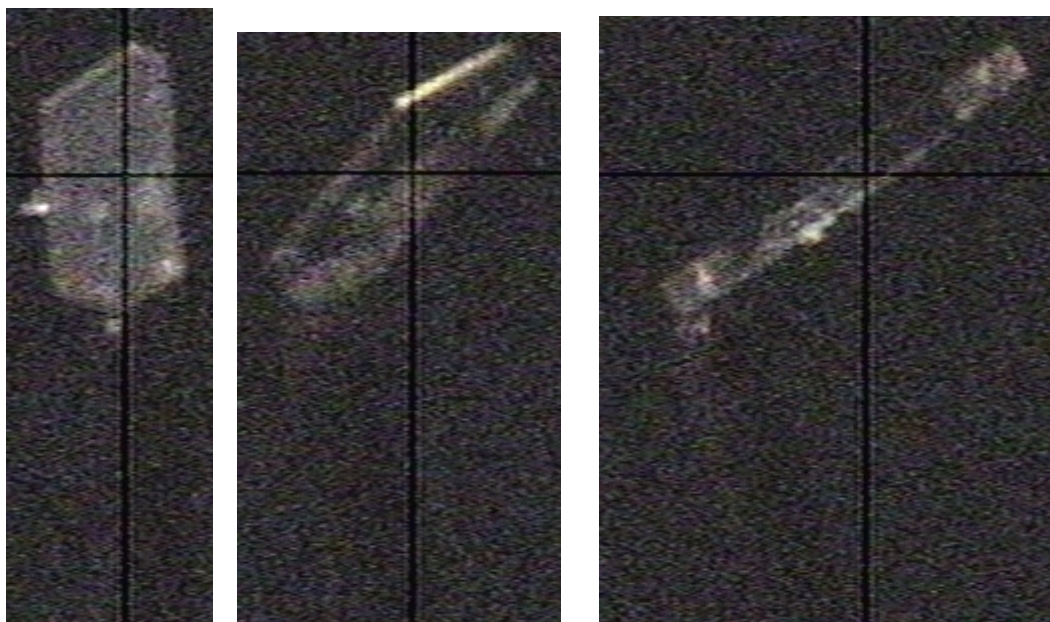


Fig. 1. The crystal

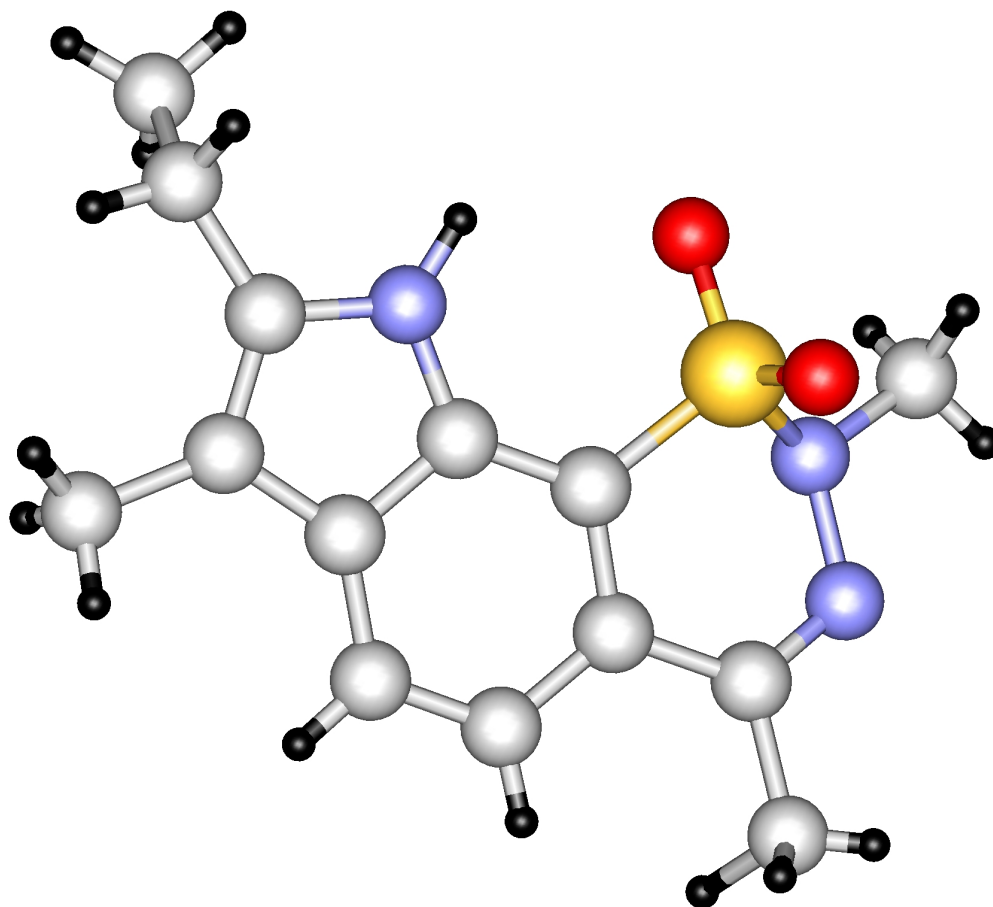


Fig. 2. The molecule (most of the hydrogens were generated by the software)

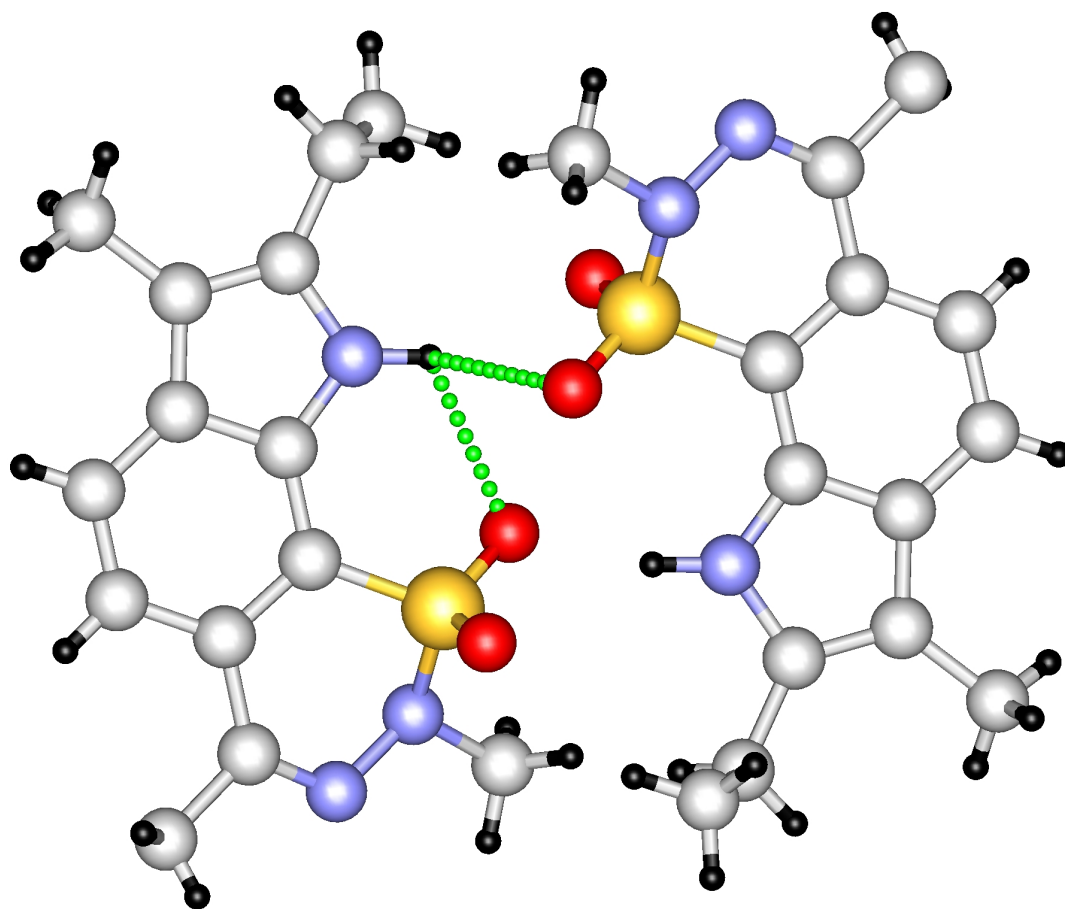


Fig. 3. Hydrogen bonds

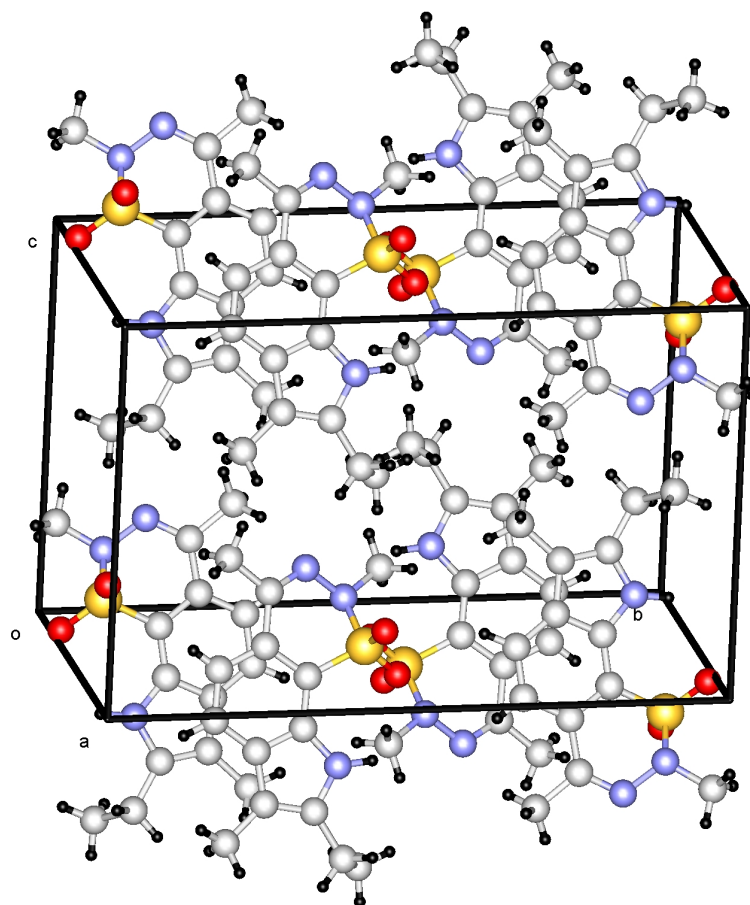


Fig. 4. Packing

## *Experimental*

### Data Collection

A colorless platelet crystal of  $C_{14}H_{17}N_3O_2S$  having approximate dimensions of 0.64 x 0.18 x 0.06 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 $\alpha$  radiation.

Indexing was performed from 4 oscillations that were exposed for 720 seconds. The crystal-to-detector distance was 127.40 mm.

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

$$\begin{aligned}a &= 7.6009(5) \text{ \AA} \\b &= 17.1310(10) \text{ \AA} \quad \beta = 95.705(4)^\circ \\c &= 10.9012(8) \text{ \AA} \\V &= 1412.43(16) \text{ \AA}^3\end{aligned}$$

For  $Z = 4$  and F.W. = 291.37, the calculated density is 1.370 g/cm<sup>3</sup>. The systematic absences of:

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

uniquely determine the space group to be:

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

The data were collected at a temperature of  $20 \pm 1^\circ\text{C}$  to a maximum  $2\theta$  value of  $142.8^\circ$ . A total of 180 oscillation images were collected. A sweep of data was done using  $\omega$  scans from  $20.0$  to  $200.0^\circ$  in  $5.0^\circ$  step, at  $\chi=0.0^\circ$  and  $\phi = 0.0^\circ$ . The exposure rate was 144.0 [sec./ $^\circ$ ]. A second sweep was performed using  $\omega$  scans from  $20.0$  to  $200.0^\circ$  in  $5.0^\circ$  step, at  $\chi=54.0^\circ$  and  $\phi = 0.0^\circ$ . The exposure rate was 144.0 [sec./ $^\circ$ ]. Another sweep was performed using  $\omega$  scans from  $20.0$  to  $200.0^\circ$  in  $5.0^\circ$  step, at  $\chi=54.0^\circ$  and  $\phi = 90.0^\circ$ . The exposure rate was 144.0 [sec./ $^\circ$ ]. Another sweep was performed using  $\omega$  scans from  $20.0$  to  $200.0^\circ$  in  $5.0^\circ$  step, at  $\chi=54.0^\circ$  and  $\phi = 180.0^\circ$ . The exposure rate was 144.0 [sec./ $^\circ$ ]. Another sweep was performed using  $\omega$  scans from  $20.0$  to  $200.0^\circ$  in  $5.0^\circ$  step, at  $\chi=54.0^\circ$  and  $\phi = 270.0^\circ$ . The exposure rate was 144.0 [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 16288 reflections that were collected, 2587 were unique ( $R_{\text{int}} = 0.056$ ).

The linear absorption coefficient,  $\mu$ , for Cu-K $\alpha$  radiation is 20.864 cm<sup>-1</sup>. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.605 to 0.882. The data were corrected for Lorentz and polarization effects.

## Structure Solution and Refinement

The structure was solved by direct methods<sup>1</sup> and expanded using Fourier techniques<sup>2</sup>. 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 refinement<sup>3</sup> on F was based on 9573 observed reflections ( $I > 2.00\sigma(I)$ ) and 204 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.0578$$

$$R_w = [ \Sigma w (|F_o| - |F_c|)^2 / \Sigma w F_o^2 ]^{1/2} = 0.0716$$

The standard deviation of an observation of unit weight<sup>4</sup> was 9.81. 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 5.84 and -4.41 e<sup>-</sup>/Å<sup>3</sup>, respectively.

Neutral atom scattering factors were taken from Cromer and Waber<sup>5</sup>. Anomalous dispersion effects were included in  $F_{\text{calc}}$ <sup>6</sup>; the values for  $\Delta f'$  and  $\Delta f''$  were those of Creagh and McAuley<sup>7</sup>. The values for the mass attenuation coefficients are those of Creagh and Hubbell<sup>8</sup>. All calculations were performed using the CrystalStructure<sup>9,10</sup> 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

(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).

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

(10) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)



## EXPERIMENTAL DETAILS

### A. Crystal Data

Empirical Formula	$\text{C}_{14}\text{H}_{17}\text{N}_3\text{O}_2\text{S}$
Formula Weight	291.37
Crystal Color, Habit	colorless, platelet
Crystal Dimensions	0.64 X 0.18 X 0.06 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 720.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 7.6009(5) \text{ \AA}$ $b = 17.1310(10) \text{ \AA}$ $c = 10.9012(8) \text{ \AA}$ $\beta = 95.705(4)^\circ$ $V = 1412.43(16) \text{ \AA}^3$
Space Group	$P2_1/a$ (#14)
Z value	4
$D_{\text{calc}}$	$1.370 \text{ g/cm}^3$
$F_{000}$	616.00
$\mu(\text{CuK}\alpha)$	$20.864 \text{ cm}^{-1}$

## B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	CuK $\alpha$ ( $\lambda$ = 1.54187 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	180 exposures
$\omega$ oscillation Range ( $\chi$ =0.0, $\phi$ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	144.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	144.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =90.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	144.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =180.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	144.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =270.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	144.0 sec./ $^{\circ}$
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	142.8 $^{\circ}$
No. of Reflections Measured	Total: 16288 Unique: 2587 ( $R_{\text{int}}$ = 0.056)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.605 - 0.882)

### 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	142.8 $^{\circ}$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ( $I > 2.00\sigma(I)$ )	9573
No. Variables	204
Reflection/Parameter Ratio	46.93
Residuals: R ( $I > 2.00\sigma(I)$ )	0.0578
Residuals: Rw ( $I > 2.00\sigma(I)$ )	0.0716
Goodness of Fit Indicator	9.813
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	5.84 e $^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	-4.41 e $^{-}/\text{\AA}^3$

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

atom	x	y	z	$B_{eq}$
S(1)	0.29792(12)	0.06756(4)	1.10979(8)	2.64(2)
O(2)	0.4560(2)	0.05483(10)	1.19017(18)	3.13(5)
O(3)	0.2488(2)	0.00721(11)	1.02102(19)	3.46(5)
N(4)	0.3983(2)	0.11418(12)	0.8354(2)	2.35(6)
N(5)	0.1370(3)	0.14898(14)	1.2686(2)	2.83(7)
N(6)	0.1251(3)	0.08449(12)	1.1872(2)	2.92(7)
C(7)	0.3558(3)	0.16761(16)	0.9214(3)	2.05(8)
C(8)	0.2834(4)	0.29859(18)	1.0571(3)	2.68(9)
C(9)	0.3326(4)	0.30778(18)	0.9411(3)	2.57(9)
C(10)	0.3696(3)	0.24272(16)	0.8700(3)	2.18(7)
C(11)	0.2673(3)	0.22429(16)	1.1091(2)	2.06(7)
C(12)	0.4221(3)	0.23263(17)	0.7490(3)	2.51(8)
C(13)	0.4554(4)	0.29644(16)	0.6614(2)	3.95(9)
C(14)	0.4828(4)	0.10926(17)	0.6200(2)	3.46(9)
C(15)	0.3064(3)	0.15824(16)	1.0399(2)	2.12(7)
C(16)	0.2022(3)	0.21348(17)	1.2300(3)	2.52(8)
C(17)	0.2024(3)	0.28092(16)	1.3179(2)	3.31(9)
C(18)	0.4380(3)	0.15384(17)	0.7309(2)	2.50(8)
C(19)	0.0340(4)	0.01740(16)	1.2347(2)	4.06(9)
C(20)	0.3229(4)	0.0659(2)	0.5539(3)	6.03(12)
H(1)	0.348(3)	0.3583(14)	0.909(2)	2.9(6)
H(2)	0.276(2)	0.3402(13)	1.106(2)	1.7(6)
H(3)	0.3985	0.0591	0.8454	2.92
H(4)	0.3482	0.3088	0.6130	4.89
H(5)	0.5421	0.2815	0.6090	4.86
H(6)	0.4956	0.3408	0.7083	4.89
H(7)	0.5263	0.1446	0.5630	4.29
H(8)	0.5722	0.0724	0.6460	4.28
H(9)	0.0945	0.3086	1.3002	4.06
H(10)	0.2990	0.3148	1.3080	4.06
H(11)	0.2105	0.2624	1.4004	4.06
H(12)	-0.0577	0.0017	1.1741	5.03
H(13)	-0.0157	0.0316	1.3082	5.01
H(14)	0.1144	-0.0246	1.2515	5.04
H(15)	0.3168	0.0151	0.5883	7.15
H(16)	0.3374	0.0618	0.4686	7.13
H(17)	0.2168	0.0934	0.5641	7.13

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

Table 2. Anisotropic displacement parameters

atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>12</sub>	U <sub>13</sub>	U <sub>23</sub>
S(1)	0.0410(5)	0.0240(4)	0.0358(5)	0.0005(4)	0.0070(4)	0.0026(4)
O(2)	0.0382(13)	0.0366(12)	0.0422(15)	0.0100(10)	-0.0047(11)	0.0081(11)
O(3)	0.0685(16)	0.0242(11)	0.0409(16)	-0.0071(10)	0.0149(12)	-0.0051(11)
N(4)	0.0443(17)	0.0195(13)	0.0273(19)	-0.0023(12)	0.0132(14)	-0.0025(13)
N(5)	0.0442(18)	0.0351(15)	0.0293(19)	0.0038(14)	0.0086(14)	-0.0028(14)
N(6)	0.0450(18)	0.0304(15)	0.037(2)	-0.0048(13)	0.0137(15)	0.0034(13)
C(7)	0.030(2)	0.0238(17)	0.025(2)	0.0010(14)	0.0068(16)	-0.0005(16)
C(8)	0.047(2)	0.0193(19)	0.036(2)	-0.0010(16)	0.0038(19)	-0.0088(18)
C(9)	0.038(2)	0.0176(18)	0.041(2)	-0.0004(16)	0.0035(19)	0.0037(18)
C(10)	0.0293(18)	0.0230(17)	0.030(2)	0.0001(14)	0.0019(16)	0.0055(16)
C(11)	0.0221(19)	0.0318(18)	0.024(2)	0.0008(14)	0.0014(16)	-0.0026(17)
C(12)	0.037(2)	0.0285(18)	0.031(2)	0.0002(15)	0.0064(18)	0.0034(17)
C(13)	0.072(2)	0.039(2)	0.042(2)	0.0057(18)	0.021(2)	0.0058(19)
C(14)	0.064(2)	0.040(2)	0.031(2)	0.0072(19)	0.025(2)	0.0034(17)
C(15)	0.0279(19)	0.0180(16)	0.034(2)	-0.0019(13)	0.0024(16)	0.0019(15)
C(16)	0.031(2)	0.0306(19)	0.033(2)	0.0088(15)	-0.0003(17)	-0.0007(17)
C(17)	0.058(2)	0.040(2)	0.030(2)	0.0037(17)	0.0148(18)	-0.0058(17)
C(18)	0.037(2)	0.0345(19)	0.025(2)	0.0001(16)	0.0121(17)	-0.0006(17)
C(19)	0.056(2)	0.043(2)	0.058(2)	-0.0147(19)	0.021(2)	0.0078(19)
C(20)	0.059(2)	0.105(3)	0.062(3)	0.022(2)	-0.012(2)	-0.049(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)	1.432(2)	S(1)	O(3)	1.440(2)
S(1)	N(6)	1.656(2)	S(1)	C(15)	1.734(2)
N(4)	C(7)	1.372(3)	N(4)	C(18)	1.385(4)
N(4)	H(3)	0.950	N(5)	N(6)	1.415(3)
N(5)	C(16)	1.299(3)	N(6)	C(19)	1.463(3)
C(7)	C(10)	1.412(4)	C(7)	C(15)	1.390(4)
C(8)	C(9)	1.363(5)	C(8)	C(11)	1.403(4)
C(8)	H(2)	0.90(2)	C(9)	C(10)	1.402(4)
C(9)	H(1)	0.94(2)	C(10)	C(12)	1.426(4)
C(11)	C(15)	1.408(4)	C(11)	C(16)	1.465(4)
C(12)	C(13)	1.489(4)	C(12)	C(18)	1.371(4)
C(13)	H(4)	0.950	C(13)	H(5)	0.950
C(13)	H(6)	0.950	C(14)	C(18)	1.498(4)
C(14)	C(20)	1.542(4)	C(14)	H(7)	0.950
C(14)	H(8)	0.950	C(16)	C(17)	1.501(4)
C(17)	H(9)	0.950	C(17)	H(10)	0.950
C(17)	H(11)	0.950	C(19)	H(12)	0.950
C(19)	H(13)	0.950	C(19)	H(14)	0.950
C(20)	H(15)	0.950	C(20)	H(16)	0.950
C(20)	H(17)	0.950			

Table 4. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	S(1)	O(3)	116.59(11)	O(2)	S(1)	N(6)	112.03(12)
O(2)	S(1)	C(15)	109.86(12)	O(3)	S(1)	N(6)	107.60(12)
O(3)	S(1)	C(15)	111.49(13)	N(6)	S(1)	C(15)	97.65(13)
C(7)	N(4)	C(18)	108.7(2)	C(7)	N(4)	H(3)	125.6
C(18)	N(4)	H(3)	125.7	N(6)	N(5)	C(16)	117.6(2)
S(1)	N(6)	N(5)	116.87(18)	S(1)	N(6)	C(19)	118.06(18)
N(5)	N(6)	C(19)	113.2(2)	N(4)	C(7)	C(10)	107.7(2)
N(4)	C(7)	C(15)	131.5(2)	C(10)	C(7)	C(15)	120.8(2)
C(9)	C(8)	C(11)	121.5(3)	C(9)	C(8)	H(2)	119.9(15)
C(11)	C(8)	H(2)	118.0(15)	C(8)	C(9)	C(10)	120.7(2)
C(8)	C(9)	H(1)	120.3(16)	C(10)	C(9)	H(1)	119.0(16)
C(7)	C(10)	C(9)	118.5(3)	C(7)	C(10)	C(12)	107.2(2)
C(9)	C(10)	C(12)	134.3(2)	C(8)	C(11)	C(15)	118.8(3)
C(8)	C(11)	C(16)	121.9(2)	C(15)	C(11)	C(16)	119.2(2)
C(10)	C(12)	C(13)	125.8(2)	C(10)	C(12)	C(18)	106.8(2)
C(13)	C(12)	C(18)	127.4(3)	C(12)	C(13)	H(4)	109.2
C(12)	C(13)	H(5)	111.2	C(12)	C(13)	H(6)	108.0
H(4)	C(13)	H(5)	109.5	H(4)	C(13)	H(6)	109.5
H(5)	C(13)	H(6)	109.5	C(18)	C(14)	C(20)	113.0(2)
C(18)	C(14)	H(7)	109.0	C(18)	C(14)	H(8)	108.1
C(20)	C(14)	H(7)	108.1	C(20)	C(14)	H(8)	109.1
H(7)	C(14)	H(8)	109.5	S(1)	C(15)	C(7)	122.5(2)
S(1)	C(15)	C(11)	117.7(2)	C(7)	C(15)	C(11)	119.7(2)
N(5)	C(16)	C(11)	125.0(2)	N(5)	C(16)	C(17)	115.0(2)
C(11)	C(16)	C(17)	119.9(2)	C(16)	C(17)	H(9)	107.9
C(16)	C(17)	H(10)	110.4	C(16)	C(17)	H(11)	110.1
H(9)	C(17)	H(10)	109.5	H(9)	C(17)	H(11)	109.5
H(10)	C(17)	H(11)	109.5	N(4)	C(18)	C(12)	109.6(2)
N(4)	C(18)	C(14)	119.9(2)	C(12)	C(18)	C(14)	130.4(2)
N(6)	C(19)	H(12)	108.4	N(6)	C(19)	H(13)	109.8
N(6)	C(19)	H(14)	110.2	H(12)	C(19)	H(13)	109.5
H(12)	C(19)	H(14)	109.5	H(13)	C(19)	H(14)	109.5
C(14)	C(20)	H(15)	108.9	C(14)	C(20)	H(16)	109.3
C(14)	C(20)	H(17)	110.2	H(15)	C(20)	H(16)	109.5
H(15)	C(20)	H(17)	109.5	H(16)	C(20)	H(17)	109.5

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

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)	S(1)	N(6)	N(5)	58.5(2)	O(2)	S(1)	N(6)	C(19)	-81.9(2)
O(2)	S(1)	C(15)	C(7)	98.4(2)	O(2)	S(1)	C(15)	C(11)	-79.3(2)
O(3)	S(1)	N(6)	N(5)	-172.10(18)	O(3)	S(1)	N(6)	C(19)	47.5(2)
O(3)	S(1)	C(15)	C(7)	-32.4(2)	O(3)	S(1)	C(15)	C(11)	149.9(2)
N(6)	S(1)	C(15)	C(7)	-144.8(2)	N(6)	S(1)	C(15)	C(11)	37.5(2)
C(15)	S(1)	N(6)	N(5)	-56.6(2)	C(15)	S(1)	N(6)	C(19)	163.0(2)
C(7)	N(4)	C(18)	C(12)	0.2(2)	C(7)	N(4)	C(18)	C(14)	-177.8(2)
C(18)	N(4)	C(7)	C(10)	-0.1(2)	C(18)	N(4)	C(7)	C(15)	179.9(2)
N(6)	N(5)	C(16)	C(11)	-1.1(4)	N(6)	N(5)	C(16)	C(17)	176.9(2)
C(16)	N(5)	N(6)	S(1)	43.5(3)	C(16)	N(5)	N(6)	C(19)	-174.3(2)
N(4)	C(7)	C(10)	C(9)	-179.9(2)	N(4)	C(7)	C(10)	C(12)	0.1(2)
N(4)	C(7)	C(15)	S(1)	3.1(4)	N(4)	C(7)	C(15)	C(11)	-179.2(2)
C(10)	C(7)	C(15)	S(1)	-176.8(2)	C(10)	C(7)	C(15)	C(11)	0.9(4)
C(15)	C(7)	C(10)	C(9)	0.1(3)	C(15)	C(7)	C(10)	C(12)	-180.0(2)
C(9)	C(8)	C(11)	C(15)	1.2(4)	C(9)	C(8)	C(11)	C(16)	-175.5(2)
C(11)	C(8)	C(9)	C(10)	-0.3(4)	C(8)	C(9)	C(10)	C(7)	-0.4(4)
C(8)	C(9)	C(10)	C(12)	179.7(3)	C(7)	C(10)	C(12)	C(13)	-179.0(2)
C(7)	C(10)	C(12)	C(18)	0.0(2)	C(9)	C(10)	C(12)	C(13)	0.9(5)
C(9)	C(10)	C(12)	C(18)	180(179)	C(8)	C(11)	C(15)	S(1)	176.3(2)
C(8)	C(11)	C(15)	C(7)	-1.5(4)	C(8)	C(11)	C(16)	N(5)	159.4(2)
C(8)	C(11)	C(16)	C(17)	-18.4(4)	C(15)	C(11)	C(16)	N(5)	-17.3(4)
C(15)	C(11)	C(16)	C(17)	164.9(2)	C(16)	C(11)	C(15)	S(1)	-6.9(3)
C(16)	C(11)	C(15)	C(7)	175.3(2)	C(10)	C(12)	C(18)	N(4)	-0.1(2)
C(10)	C(12)	C(18)	C(14)	177.5(2)	C(13)	C(12)	C(18)	N(4)	178.9(2)
C(13)	C(12)	C(18)	C(14)	-3.4(5)	C(20)	C(14)	C(18)	N(4)	69.9(3)
C(20)	C(14)	C(18)	C(12)	-107.6(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(3) <sup>11</sup>	3.170	O(2)	O(3) <sup>11</sup>	3.534(2)
O(2)	N(4) <sup>11</sup>	3.122(2)	O(2)	C(14) <sup>11</sup>	3.494(3)
O(2)	C(17) <sup>21</sup>	3.584(3)	O(2)	H(2) <sup>21</sup>	3.23(2)
O(2)	H(3) <sup>11</sup>	2.295	O(2)	H(8) <sup>11</sup>	2.839
O(2)	H(9) <sup>21</sup>	2.788	O(2)	H(10) <sup>21</sup>	3.574
O(2)	H(15) <sup>11</sup>	3.069	O(2)	H(16) <sup>31</sup>	3.253
O(3)	O(2) <sup>11</sup>	3.534(2)	O(3)	C(9) <sup>41</sup>	3.504(3)
O(3)	C(19) <sup>51</sup>	3.373(3)	O(3)	H(1) <sup>41</sup>	2.78(2)
O(3)	H(2) <sup>41</sup>	3.18(2)	O(3)	H(3) <sup>11</sup>	3.134
O(3)	H(12) <sup>51</sup>	2.459	N(4)	O(2) <sup>11</sup>	3.122(2)
N(4)	H(1) <sup>21</sup>	3.46(2)	N(4)	H(6) <sup>61</sup>	3.322
N(4)	H(12) <sup>51</sup>	3.256	N(5)	C(8) <sup>61</sup>	3.481(4)
N(5)	C(20) <sup>31</sup>	3.581(4)	N(5)	H(2) <sup>61</sup>	3.12(2)
N(5)	H(9) <sup>21</sup>	3.536	N(5)	H(10) <sup>61</sup>	2.718
N(5)	H(16) <sup>31</sup>	2.940	N(5)	H(17) <sup>31</sup>	3.355
N(6)	C(8) <sup>61</sup>	3.469(3)	N(6)	H(2) <sup>61</sup>	3.00(2)
N(6)	H(10) <sup>61</sup>	3.392	N(6)	H(16) <sup>31</sup>	3.344
C(7)	C(8) <sup>21</sup>	3.483(4)	C(7)	H(6) <sup>61</sup>	3.413
C(8)	N(5) <sup>21</sup>	3.481(4)	C(8)	N(6) <sup>21</sup>	3.469(3)
C(8)	C(7) <sup>61</sup>	3.483(4)	C(8)	C(16) <sup>21</sup>	3.541(4)
C(9)	O(3) <sup>71</sup>	3.504(3)	C(10)	C(12) <sup>61</sup>	3.549(3)
C(10)	H(6) <sup>61</sup>	3.498	C(11)	C(16) <sup>21</sup>	3.597(3)
C(11)	H(9) <sup>21</sup>	3.133	C(12)	C(10) <sup>21</sup>	3.549(3)
C(12)	H(5) <sup>61</sup>	3.139	C(12)	H(6) <sup>61</sup>	3.464
C(13)	H(4) <sup>21</sup>	3.572	C(13)	H(5) <sup>61</sup>	3.409
C(13)	H(7) <sup>61</sup>	3.480	C(13)	H(11) <sup>81</sup>	3.294
C(13)	H(14) <sup>71</sup>	3.268	C(13)	H(17) <sup>21</sup>	3.010
C(14)	O(2) <sup>11</sup>	3.494(3)	C(14)	H(4) <sup>21</sup>	3.119
C(14)	H(14) <sup>11</sup>	3.548	C(14)	H(15) <sup>91</sup>	3.563
C(14)	H(16) <sup>91</sup>	3.413	C(15)	H(2) <sup>21</sup>	3.57(2)
C(15)	H(9) <sup>21</sup>	3.455	C(16)	C(8) <sup>61</sup>	3.541(4)
C(16)	C(11) <sup>61</sup>	3.597(3)	C(16)	H(2) <sup>61</sup>	3.51(2)
C(16)	H(9) <sup>21</sup>	3.028	C(16)	H(10) <sup>61</sup>	3.297
C(17)	O(2) <sup>61</sup>	3.584(3)	C(17)	H(4) <sup>31</sup>	3.330
C(17)	H(7) <sup>101</sup>	3.357	C(17)	H(9) <sup>21</sup>	3.374
C(17)	H(10) <sup>61</sup>	3.469	C(18)	H(1) <sup>21</sup>	3.51(2)
C(18)	H(4) <sup>21</sup>	3.546	C(18)	H(5) <sup>61</sup>	3.354

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

atom	atom	distance	atom	atom	distance
C(18)	H(6) <sup>6j</sup>	3.348	C(19)	O(3) <sup>5j</sup>	3.373(3)
C(19)	H(1) <sup>4j</sup>	3.31(2)	C(19)	H(2) <sup>6j</sup>	3.35(2)
C(19)	H(3) <sup>5j</sup>	3.565	C(19)	H(6) <sup>4j</sup>	3.100
C(19)	H(8) <sup>1j</sup>	3.499	C(19)	H(10) <sup>6j</sup>	3.518
C(19)	H(15) <sup>5j</sup>	3.488	C(19)	H(16) <sup>3j</sup>	3.351
C(19)	H(17) <sup>5j</sup>	3.588	C(20)	N(5) <sup>8j</sup>	3.581(4)
C(20)	H(5) <sup>6j</sup>	3.465	C(20)	H(6) <sup>6j</sup>	3.522
C(20)	H(8) <sup>9j</sup>	3.368	C(20)	H(13) <sup>8j</sup>	3.573
C(20)	H(13) <sup>5j</sup>	3.346	C(20)	H(15) <sup>9j</sup>	3.559
C(20)	H(16) <sup>9j</sup>	3.411	H(1)	O(3) <sup>7j</sup>	2.78(2)
H(1)	N(4) <sup>6j</sup>	3.46(2)	H(1)	C(18) <sup>6j</sup>	3.51(2)
H(1)	C(19) <sup>7j</sup>	3.31(2)	H(1)	H(8) <sup>6j</sup>	3.581
H(1)	H(12) <sup>7j</sup>	3.114	H(1)	H(14) <sup>7j</sup>	2.697
H(2)	O(2) <sup>6j</sup>	3.23(2)	H(2)	O(3) <sup>7j</sup>	3.18(2)
H(2)	N(5) <sup>2j</sup>	3.12(2)	H(2)	N(6) <sup>2j</sup>	3.00(2)
H(2)	C(15) <sup>6j</sup>	3.57(2)	H(2)	C(16) <sup>2j</sup>	3.51(2)
H(2)	C(19) <sup>2j</sup>	3.35(2)	H(2)	H(12) <sup>2j</sup>	3.047
H(2)	H(13) <sup>2j</sup>	3.389	H(3)	S(1) <sup>1j</sup>	3.170
H(3)	O(2) <sup>1j</sup>	2.295	H(3)	O(3) <sup>1j</sup>	3.134
H(3)	C(19) <sup>5j</sup>	3.565	H(3)	H(12) <sup>5j</sup>	2.780
H(3)	H(13) <sup>5j</sup>	3.566	H(4)	C(13) <sup>6j</sup>	3.572
H(4)	C(14) <sup>6j</sup>	3.119	H(4)	C(17) <sup>8j</sup>	3.330
H(4)	C(18) <sup>6j</sup>	3.546	H(4)	H(5) <sup>6j</sup>	2.791
H(4)	H(7) <sup>6j</sup>	2.579	H(4)	H(8) <sup>6j</sup>	2.970
H(4)	H(10) <sup>8j</sup>	3.310	H(4)	H(11) <sup>8j</sup>	2.572
H(4)	H(14) <sup>7j</sup>	3.214	H(4)	H(17) <sup>2j</sup>	3.353
H(5)	C(12) <sup>2j</sup>	3.139	H(5)	C(13) <sup>2j</sup>	3.409
H(5)	C(18) <sup>2j</sup>	3.354	H(5)	C(20) <sup>2j</sup>	3.465
H(5)	H(4) <sup>2j</sup>	2.791	H(5)	H(11) <sup>8j</sup>	3.239
H(5)	H(11) <sup>11j</sup>	2.820	H(5)	H(17) <sup>2j</sup>	2.593
H(6)	N(4) <sup>2j</sup>	3.322	H(6)	C(7) <sup>2j</sup>	3.413
H(6)	C(10) <sup>2j</sup>	3.498	H(6)	C(12) <sup>2j</sup>	3.464
H(6)	C(18) <sup>2j</sup>	3.348	H(6)	C(19) <sup>7j</sup>	3.100
H(6)	C(20) <sup>2j</sup>	3.522	H(6)	H(8) <sup>6j</sup>	3.549
H(6)	H(12) <sup>7j</sup>	3.056	H(6)	H(13) <sup>7j</sup>	3.277
H(6)	H(14) <sup>7j</sup>	2.506	H(6)	H(17) <sup>2j</sup>	2.663
H(7)	C(13) <sup>2j</sup>	3.480	H(7)	C(17) <sup>11j</sup>	3.357

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

atom	atom	distance	atom	atom	distance
H(7)	H(4) <sup>2)</sup>	2.579	H(7)	H(9) <sup>11)</sup>	3.069
H(7)	H(11) <sup>8)</sup>	3.483	H(7)	H(11) <sup>11)</sup>	2.852
H(7)	H(15) <sup>9)</sup>	3.465	H(8)	O(2) <sup>1)</sup>	2.839
H(8)	C(19) <sup>1)</sup>	3.499	H(8)	C(20) <sup>9)</sup>	3.368
H(8)	H(1) <sup>2)</sup>	3.581	H(8)	H(4) <sup>2)</sup>	2.970
H(8)	H(6) <sup>2)</sup>	3.549	H(8)	H(14) <sup>1)</sup>	2.658
H(8)	H(15) <sup>9)</sup>	3.148	H(8)	H(16) <sup>9)</sup>	2.736
H(9)	O(2) <sup>6)</sup>	2.788	H(9)	N(5) <sup>6)</sup>	3.536
H(9)	C(11) <sup>6)</sup>	3.133	H(9)	C(15) <sup>6)</sup>	3.455
H(9)	C(16) <sup>6)</sup>	3.028	H(9)	C(17) <sup>6)</sup>	3.374
H(9)	H(7) <sup>10)</sup>	3.069	H(9)	H(10) <sup>6)</sup>	3.092
H(9)	H(11) <sup>6)</sup>	3.439	H(9)	H(16) <sup>10)</sup>	3.583
H(10)	O(2) <sup>6)</sup>	3.574	H(10)	N(5) <sup>2)</sup>	2.718
H(10)	N(6) <sup>2)</sup>	3.392	H(10)	C(16) <sup>2)</sup>	3.297
H(10)	C(17) <sup>2)</sup>	3.469	H(10)	C(19) <sup>2)</sup>	3.518
H(10)	H(4) <sup>3)</sup>	3.310	H(10)	H(9) <sup>2)</sup>	3.092
H(10)	H(11) <sup>2)</sup>	3.453	H(10)	H(13) <sup>2)</sup>	2.985
H(11)	C(13) <sup>3)</sup>	3.294	H(11)	H(4) <sup>3)</sup>	2.572
H(11)	H(5) <sup>3)</sup>	3.239	H(11)	H(5) <sup>10)</sup>	2.820
H(11)	H(7) <sup>3)</sup>	3.483	H(11)	H(7) <sup>10)</sup>	2.852
H(11)	H(9) <sup>2)</sup>	3.439	H(11)	H(10) <sup>6)</sup>	3.453
H(11)	H(17) <sup>3)</sup>	3.399	H(12)	O(3) <sup>5)</sup>	2.459
H(12)	N(4) <sup>5)</sup>	3.256	H(12)	H(1) <sup>4)</sup>	3.114
H(12)	H(2) <sup>6)</sup>	3.047	H(12)	H(3) <sup>5)</sup>	2.780
H(12)	H(6) <sup>4)</sup>	3.056	H(12)	H(15) <sup>5)</sup>	3.418
H(12)	H(17) <sup>5)</sup>	3.598	H(13)	C(20) <sup>3)</sup>	3.573
H(13)	C(20) <sup>5)</sup>	3.346	H(13)	H(2) <sup>6)</sup>	3.389
H(13)	H(3) <sup>5)</sup>	3.566	H(13)	H(6) <sup>4)</sup>	3.277
H(13)	H(10) <sup>6)</sup>	2.985	H(13)	H(15) <sup>5)</sup>	2.769
H(13)	H(16) <sup>3)</sup>	3.097	H(13)	H(17) <sup>3)</sup>	3.323
H(13)	H(17) <sup>5)</sup>	3.046	H(14)	C(13) <sup>4)</sup>	3.268
H(14)	C(14) <sup>1)</sup>	3.548	H(14)	H(1) <sup>4)</sup>	2.697
H(14)	H(4) <sup>4)</sup>	3.214	H(14)	H(6) <sup>4)</sup>	2.506
H(14)	H(8) <sup>1)</sup>	2.658	H(14)	H(16) <sup>3)</sup>	3.141
H(14)	H(17) <sup>5)</sup>	3.574	H(15)	O(2) <sup>1)</sup>	3.069
H(15)	C(14) <sup>9)</sup>	3.563	H(15)	C(19) <sup>5)</sup>	3.488
H(15)	C(20) <sup>9)</sup>	3.559	H(15)	H(7) <sup>9)</sup>	3.465

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

atom	atom	distance	atom	atom	distance
H(15)	H(8) <sup>9j</sup>	3.148	H(15)	H(12) <sup>5j</sup>	3.418
H(15)	H(13) <sup>5j</sup>	2.769	H(15)	H(15) <sup>9j</sup>	3.576
H(15)	H(16) <sup>9j</sup>	3.057	H(16)	O(2) <sup>8j</sup>	3.253
H(16)	N(5) <sup>8j</sup>	2.940	H(16)	N(6) <sup>8j</sup>	3.344
H(16)	C(14) <sup>9j</sup>	3.413	H(16)	C(19) <sup>8j</sup>	3.351
H(16)	C(20) <sup>9j</sup>	3.411	H(16)	H(8) <sup>9j</sup>	2.736
H(16)	H(9) <sup>11j</sup>	3.583	H(16)	H(13) <sup>8j</sup>	3.097
H(16)	H(14) <sup>8j</sup>	3.141	H(16)	H(15) <sup>9j</sup>	3.057
H(16)	H(16) <sup>9j</sup>	3.274	H(17)	N(5) <sup>8j</sup>	3.355
H(17)	C(13) <sup>6j</sup>	3.010	H(17)	C(19) <sup>5j</sup>	3.588
H(17)	H(4) <sup>6j</sup>	3.353	H(17)	H(5) <sup>6j</sup>	2.593
H(17)	H(6) <sup>6j</sup>	2.663	H(17)	H(11) <sup>8j</sup>	3.399
H(17)	H(12) <sup>5j</sup>	3.598	H(17)	H(13) <sup>8j</sup>	3.323
H(17)	H(13) <sup>5j</sup>	3.046	H(17)	H(14) <sup>5j</sup>	3.574

Symmetry Operators:

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

Table 7. Intramolecular and Intermolecular Hydrogen bonds

D	H	A	D...A	D-H	H...A	D-H...A
N(4)	H(3)	O(2)[3:1:0:2]	3.122(2)	0.950	2.295	145.1
N(4)	H(3)	O(3)	3.034(3)	0.950	2.486	116.6

- 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.