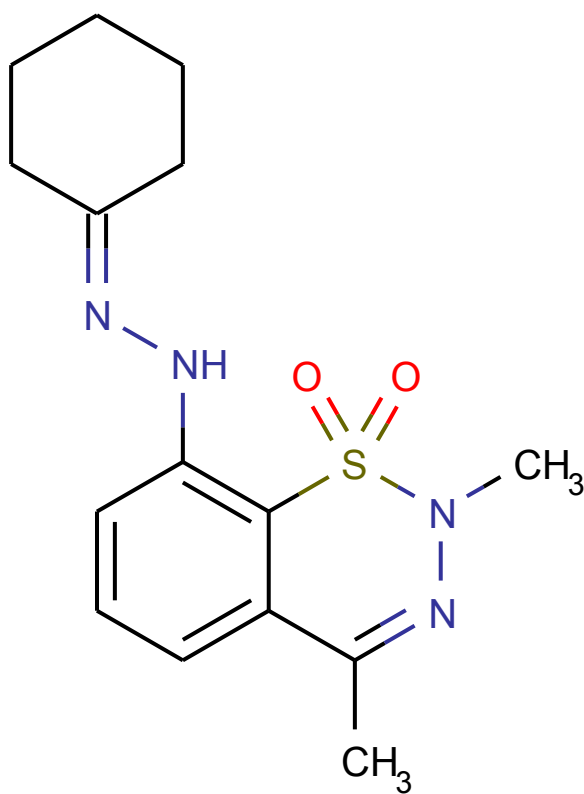


**142695**

**PGY0537\_1A**

Submitted by: Pusztai Gyongyver  
Operator: Dancso Andras

X-ray Structure Report



January 10, 2025



Fig. 1. The crystal

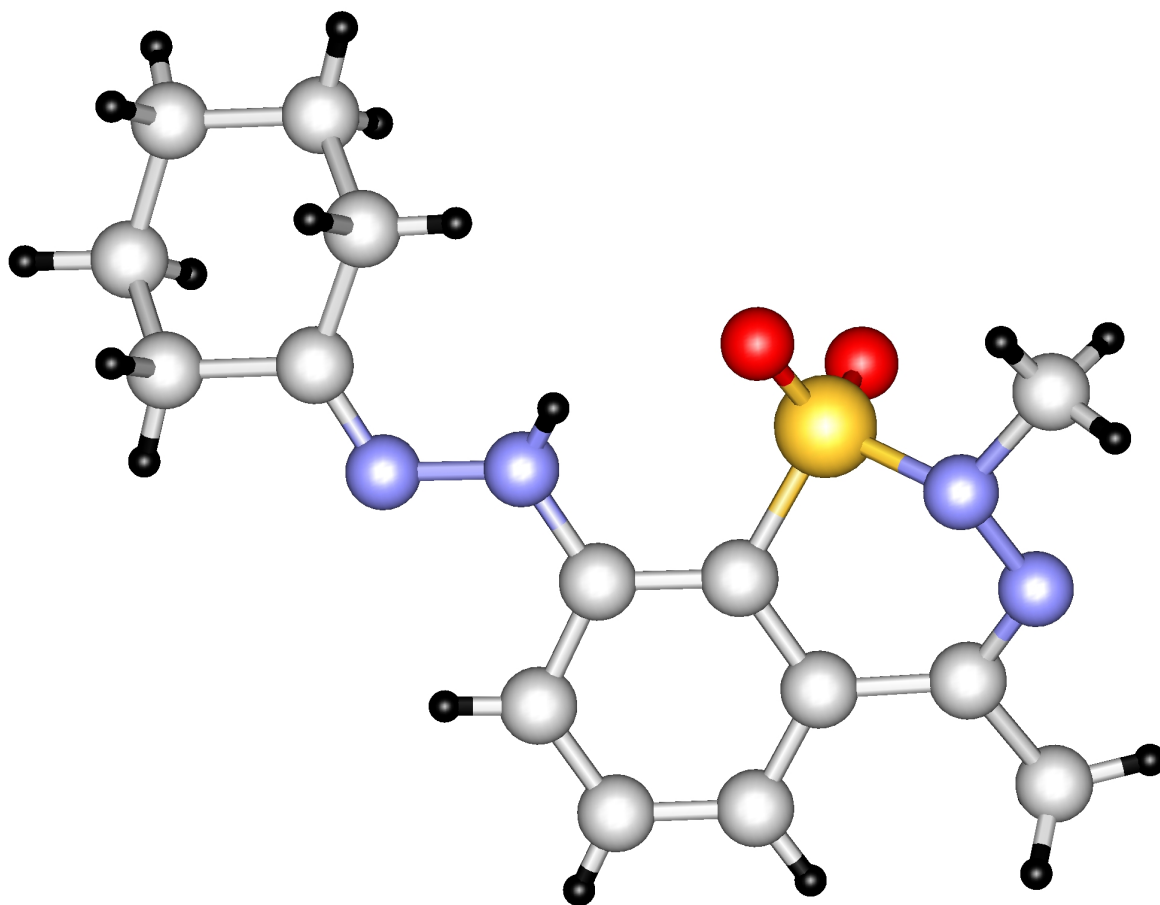


Fig. 2. The molecule

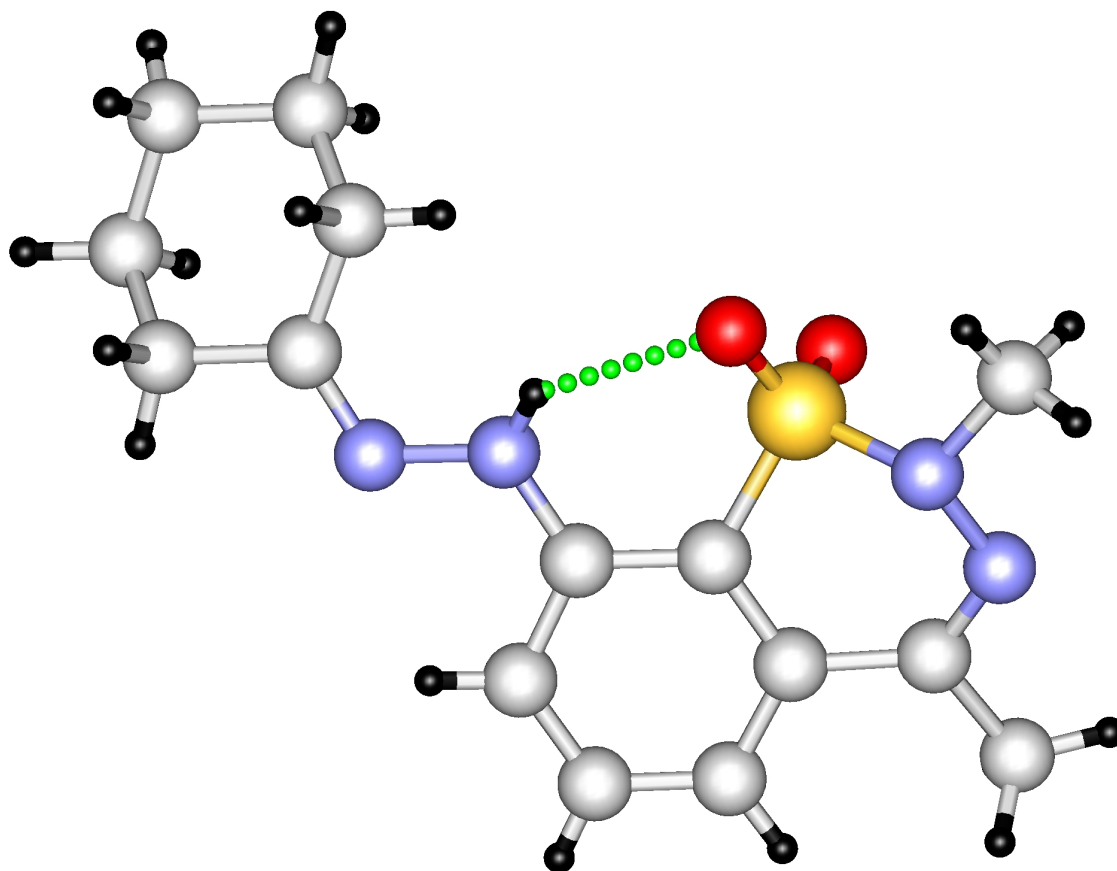


Fig. 3. Hydrogen bond

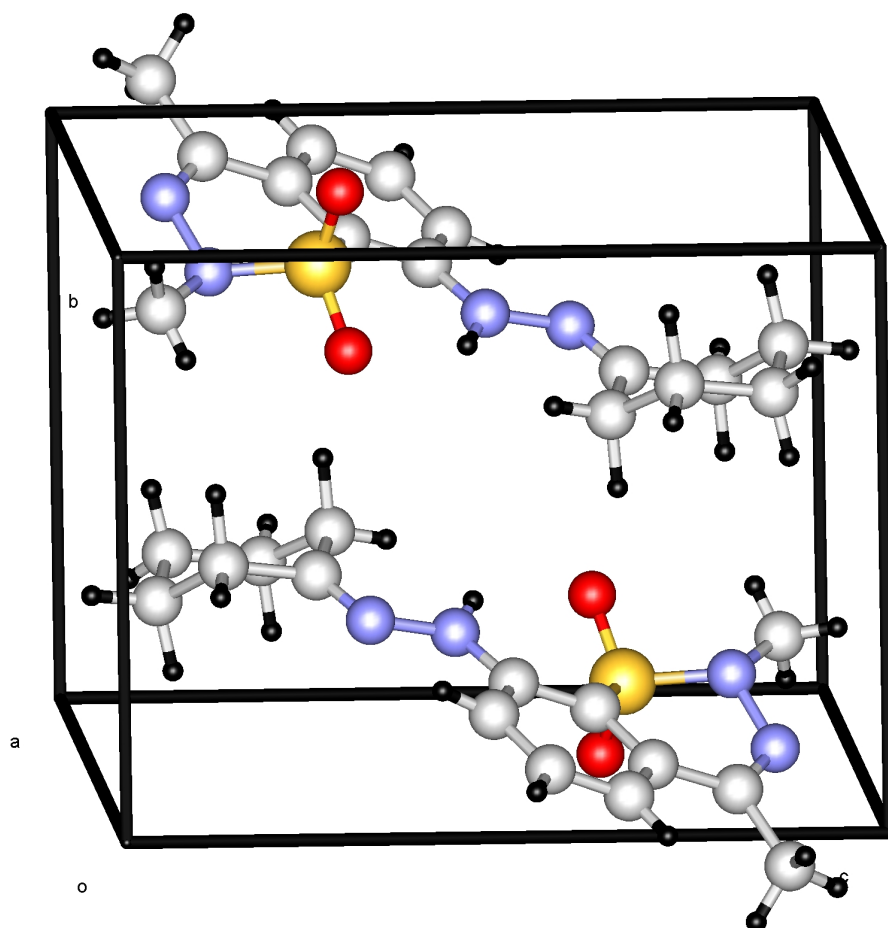


Fig. 4. Packing

## *Experimental*

### Data Collection

A colorless prism crystal of  $C_{15}H_{20}N_4O_2S$  having approximate dimensions of 0.23 x 0.12 x 0.04 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 900 seconds. The crystal-to-detector distance was 127.40 mm.

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

$$\begin{aligned}a &= 7.8403(5) \text{ \AA} & \alpha &= 88.044(5)^\circ \\b &= 8.8939(6) \text{ \AA} & \beta &= 75.471(5)^\circ \\c &= 12.5035(9) \text{ \AA} & \gamma &= 68.340(4)^\circ \\V &= 782.74(9) \text{ \AA}^3\end{aligned}$$

For  $Z = 2$  and F.W. = 320.41, the calculated density is 1.359 g/cm<sup>3</sup>. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

### P-1 (#2)

The data were collected at a temperature of  $20 \pm 1^\circ\text{C}$  to a maximum  $2\theta$  value of  $143.2^\circ$ . A total of 216 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 180.0 [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 180.0 [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 180.0 [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 180.0 [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 180.0 [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 180.0 [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 10710 reflections that were collected, 2706 were unique ( $R_{\text{int}} = 0.041$ ).

The linear absorption coefficient,  $\mu$ , for Cu-K $\alpha$  radiation is 19.501 cm<sup>-1</sup>. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.487 to 0.677. 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. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement<sup>3</sup> on F was based on 9078 observed reflections ( $I > 2.00\sigma(I)$ ) and 279 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.0505$$

$$R_w = [ \sum w (|F_o| - |F_c|)^2 / \sum w F_o^2 ]^{1/2} = 0.0522$$

The standard deviation of an observation of unit weight<sup>4</sup> was 5.24. Unit weights were used. Plots of  $\sum w (|F_o| - |F_c|)^2$  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 3.66 and -2.33 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 Fcalc<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).

(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) 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	$C_{15}H_{20}N_4O_2S$
Formula Weight	320.41
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.23 X 0.12 X 0.04 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 900.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 7.8403(5) \text{ \AA}$ $b = 8.8939(6) \text{ \AA}$ $c = 12.5035(9) \text{ \AA}$ $\alpha = 88.044(5)^\circ$ $\beta = 75.471(5)^\circ$ $\gamma = 68.340(4)^\circ$ $V = 782.74(9) \text{ \AA}^3$
Space Group	P-1 (#2)
Z value	2
$D_{\text{calc}}$	$1.359 \text{ g/cm}^3$
$F_{000}$	340.00
$\mu(\text{CuK}\alpha)$	$19.501 \text{ cm}^{-1}$

## 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	216 exposures
$\omega$ oscillation Range ( $\chi$ =0.0, $\phi$ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =0.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =90.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =90.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =180.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
$\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =270.0)	20.0 - 200.0 $^{\circ}$
Exposure Rate	180.0 sec./ $^{\circ}$
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	143.2 $^{\circ}$
No. of Reflections Measured	Total: 10710 Unique: 2706 ( $R_{\text{int}}$ = 0.041)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.487 - 0.677)

### 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 ( $I > 2.00\sigma(I)$ )	9078
No. Variables	279
Reflection/Parameter Ratio	32.54
Residuals: R ( $I > 2.00\sigma(I)$ )	0.0505
Residuals: Rw ( $I > 2.00\sigma(I)$ )	0.0522
Goodness of Fit Indicator	5.239
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	3.66 e $^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	-2.33 e $^{-}/\text{\AA}^3$

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

atom	x	y	z	$B_{\text{eq}}$
S(1)	0.23075(8)	0.20671(6)	0.67302(5)	3.379(14)
O(2)	0.37688(19)	0.04720(17)	0.65477(12)	4.34(4)
O(3)	0.2871(2)	0.34178(17)	0.63937(12)	4.15(4)
N(4)	0.1690(2)	0.3324(2)	0.33819(17)	3.78(5)
N(5)	0.1661(2)	0.3106(2)	0.44877(18)	3.91(5)
N(6)	0.0246(2)	0.1407(2)	0.85422(14)	3.81(5)
N(7)	0.1126(2)	0.2470(2)	0.80573(14)	3.80(5)
C(8)	-0.0807(2)	0.1420(2)	0.68498(18)	2.86(5)
C(9)	0.4499(3)	0.4161(3)	0.3104(2)	4.22(7)
C(10)	0.2951(3)	0.3795(2)	0.2788(2)	3.53(6)
C(11)	0.0440(2)	0.2088(2)	0.62050(18)	2.73(5)
C(12)	0.0355(2)	0.2557(2)	0.51343(19)	3.08(5)
C(13)	-0.2179(3)	0.1224(2)	0.6412(2)	3.58(6)
C(14)	-0.0656(2)	0.0938(2)	0.7967(2)	3.27(5)
C(15)	-0.2300(3)	0.1725(2)	0.5374(2)	4.10(6)
C(16)	0.6442(3)	0.3180(3)	0.2362(2)	4.42(7)
C(17)	0.4922(4)	0.2897(3)	0.0863(2)	4.66(7)
C(18)	-0.1621(4)	-0.0147(3)	0.8551(2)	4.76(8)
C(19)	-0.1101(3)	0.2378(3)	0.4747(2)	3.94(6)
C(20)	0.2985(4)	0.3967(3)	0.1575(2)	4.33(7)
C(21)	0.6484(4)	0.3324(4)	0.1133(2)	4.96(8)
C(22)	0.2155(7)	0.2844(6)	0.8784(3)	6.53(12)
H(1)	-0.296(2)	0.073(2)	0.6845(15)	2.8(4)
H(2)	-0.318(2)	0.156(2)	0.5119(15)	3.8(5)
H(3)	-0.119(2)	0.282(2)	0.4071(16)	3.7(5)
H(4)	0.224(3)	0.350(2)	0.4779(18)	5.1(6)
H(5)	0.256(3)	0.529(2)	0.1452(18)	6.9(6)
H(6)	0.195(2)	0.368(2)	0.1446(16)	4.8(5)
H(7)	0.521(2)	0.163(2)	0.1080(17)	6.0(6)
H(8)	0.498(2)	0.299(2)	0.0008(19)	6.4(6)
H(9)	0.623(2)	0.448(2)	0.0919(18)	5.4(6)
H(10)	0.778(3)	0.258(2)	0.074(2)	7.9(8)
H(11)	0.738(2)	0.358(2)	0.2530(16)	5.1(5)
H(12)	0.683(2)	0.188(2)	0.2517(18)	6.5(6)
H(13)	0.410(3)	0.551(3)	0.2994(19)	8.0(7)
H(14)	0.453(2)	0.400(2)	0.3847(17)	4.7(5)
H(15)	0.133(3)	0.319(3)	0.947(2)	8.6(10)

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

atom	x	y	z	$B_{\text{eq}}$
H(16)	0.252(3)	0.362(3)	0.850(2)	7.4(9)
H(17)	0.321(4)	0.186(3)	0.895(2)	11.5(12)
H(18)	-0.122(3)	-0.062(3)	0.921(2)	8.3(8)
H(19)	-0.298(3)	0.035(2)	0.864(2)	7.4(8)
H(20)	-0.129(2)	-0.118(2)	0.8128(18)	5.4(6)

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

Table 2. Anisotropic displacement parameters

atom	U <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)	0.0389(3)	0.0546(3)	0.0432(4)	-0.0258(2)	-0.0124(2)	0.0044(2)
O(2)	0.0367(8)	0.0570(10)	0.0671(12)	-0.0135(8)	-0.0121(8)	0.0051(8)
O(3)	0.0609(10)	0.0669(10)	0.0507(11)	-0.0460(8)	-0.0180(8)	0.0141(8)
N(4)	0.0501(12)	0.0586(12)	0.0343(14)	-0.0206(10)	-0.0097(11)	0.0064(10)
N(5)	0.0572(14)	0.0670(14)	0.0330(14)	-0.0338(11)	-0.0108(12)	0.0065(11)
N(6)	0.0486(11)	0.0629(12)	0.0385(13)	-0.0308(10)	-0.0048(10)	0.0033(10)
N(7)	0.0557(12)	0.0665(13)	0.0346(13)	-0.0373(11)	-0.0101(10)	-0.0003(10)
C(8)	0.0332(12)	0.0369(12)	0.0358(15)	-0.0144(10)	-0.0014(11)	-0.0021(10)
C(9)	0.0668(19)	0.0661(19)	0.0358(18)	-0.0338(15)	-0.0141(15)	0.0059(14)
C(10)	0.0508(15)	0.0428(13)	0.0352(16)	-0.0127(12)	-0.0090(13)	0.0041(11)
C(11)	0.0307(11)	0.0418(12)	0.0316(14)	-0.0159(10)	-0.0047(11)	-0.0008(10)
C(12)	0.0341(12)	0.0431(13)	0.0358(15)	-0.0143(10)	-0.0014(11)	-0.0027(11)
C(13)	0.0317(13)	0.0544(15)	0.0510(19)	-0.0220(12)	-0.0026(13)	-0.0032(13)
C(14)	0.0327(12)	0.0446(13)	0.0423(16)	-0.0171(11)	0.0032(11)	-0.0017(11)
C(15)	0.0330(13)	0.0664(17)	0.057(2)	-0.0178(13)	-0.0119(14)	-0.0129(14)
C(16)	0.0535(17)	0.0692(19)	0.0502(19)	-0.0284(15)	-0.0142(15)	0.0108(15)
C(17)	0.077(2)	0.0705(19)	0.0342(18)	-0.0362(17)	-0.0091(16)	0.0031(15)
C(18)	0.064(2)	0.066(2)	0.060(2)	-0.0409(18)	-0.0056(18)	0.0112(17)
C(19)	0.0401(14)	0.0675(17)	0.0390(18)	-0.0138(13)	-0.0139(14)	-0.0022(14)
C(20)	0.0624(18)	0.0711(19)	0.0408(18)	-0.0330(16)	-0.0194(15)	0.0152(14)
C(21)	0.063(2)	0.077(2)	0.047(2)	-0.0276(18)	-0.0076(16)	0.0042(16)
C(22)	0.114(3)	0.127(3)	0.053(2)	-0.088(3)	-0.036(2)	0.007(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)	1.4382(13)	S(1)	O(3)	1.4418(18)
S(1)	N(7)	1.6626(17)	S(1)	C(11)	1.743(2)
N(4)	N(5)	1.385(3)	N(4)	C(10)	1.273(3)
N(5)	C(12)	1.366(3)	N(5)	H(4)	0.82(2)
N(6)	N(7)	1.404(2)	N(6)	C(14)	1.298(3)
N(7)	C(22)	1.474(6)	C(8)	C(11)	1.400(3)
C(8)	C(13)	1.389(3)	C(8)	C(14)	1.467(3)
C(9)	C(10)	1.509(4)	C(9)	C(16)	1.520(3)
C(9)	H(13)	1.13(2)	C(9)	H(14)	0.94(2)
C(10)	C(20)	1.514(3)	C(11)	C(12)	1.399(3)
C(12)	C(19)	1.407(4)	C(13)	C(15)	1.371(4)
C(13)	H(1)	0.941(19)	C(14)	C(18)	1.500(4)
C(15)	C(19)	1.356(4)	C(15)	H(2)	0.89(2)
C(16)	C(21)	1.530(4)	C(16)	H(11)	0.99(2)
C(16)	H(12)	1.10(2)	C(17)	C(20)	1.522(3)
C(17)	C(21)	1.522(5)	C(17)	H(7)	1.10(2)
C(17)	H(8)	1.06(2)	C(18)	H(18)	0.98(2)
C(18)	H(19)	0.97(2)	C(18)	H(20)	0.99(2)
C(19)	H(3)	0.93(2)	C(20)	H(5)	1.12(2)
C(20)	H(6)	0.98(2)	C(21)	H(9)	1.01(2)
C(21)	H(10)	1.00(2)	C(22)	H(15)	0.92(2)
C(22)	H(16)	0.88(3)	C(22)	H(17)	1.01(2)

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

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	S(1)	O(3)	117.60(9)	O(2)	S(1)	N(7)	111.20(8)
O(2)	S(1)	C(11)	109.90(10)	O(3)	S(1)	N(7)	106.59(9)
O(3)	S(1)	C(11)	111.94(10)	N(7)	S(1)	C(11)	97.75(10)
N(5)	N(4)	C(10)	118.2(2)	N(4)	N(5)	C(12)	119.3(2)
N(4)	N(5)	H(4)	119.5(15)	C(12)	N(5)	H(4)	119.7(15)
N(7)	N(6)	C(14)	117.12(19)	S(1)	N(7)	N(6)	117.13(15)
S(1)	N(7)	C(22)	114.8(2)	N(6)	N(7)	C(22)	111.1(2)
C(11)	C(8)	C(13)	118.8(2)	C(11)	C(8)	C(14)	120.3(2)
C(13)	C(8)	C(14)	121.0(2)	C(10)	C(9)	C(16)	111.4(2)
C(10)	C(9)	H(13)	103.4(14)	C(10)	C(9)	H(14)	114.3(15)
C(16)	C(9)	H(13)	111.6(11)	C(16)	C(9)	H(14)	108.8(11)
H(13)	C(9)	H(14)	107.3(18)	N(4)	C(10)	C(9)	129.4(2)
N(4)	C(10)	C(20)	116.8(2)	C(9)	C(10)	C(20)	113.8(2)
S(1)	C(11)	C(8)	116.37(17)	S(1)	C(11)	C(12)	120.89(17)
C(8)	C(11)	C(12)	122.2(2)	N(5)	C(12)	C(11)	121.5(2)
N(5)	C(12)	C(19)	121.9(2)	C(11)	C(12)	C(19)	116.5(2)
C(8)	C(13)	C(15)	119.1(2)	C(8)	C(13)	H(1)	117.4(13)
C(15)	C(13)	H(1)	123.5(13)	N(6)	C(14)	C(8)	125.1(2)
N(6)	C(14)	C(18)	114.3(2)	C(8)	C(14)	C(18)	120.6(2)
C(13)	C(15)	C(19)	122.4(2)	C(13)	C(15)	H(2)	116.3(12)
C(19)	C(15)	H(2)	121.2(12)	C(9)	C(16)	C(21)	112.1(2)
C(9)	C(16)	H(11)	108.0(9)	C(9)	C(16)	H(12)	110.1(10)
C(21)	C(16)	H(11)	109.7(11)	C(21)	C(16)	H(12)	106.9(12)
H(11)	C(16)	H(12)	110.1(17)	C(20)	C(17)	C(21)	110.6(2)
C(20)	C(17)	H(7)	108.8(10)	C(20)	C(17)	H(8)	111.2(10)
C(21)	C(17)	H(7)	105.4(13)	C(21)	C(17)	H(8)	110.7(14)
H(7)	C(17)	H(8)	110.0(17)	C(14)	C(18)	H(18)	115.9(19)
C(14)	C(18)	H(19)	110.3(15)	C(14)	C(18)	H(20)	114.2(13)
H(18)	C(18)	H(19)	116(2)	H(18)	C(18)	H(20)	97(2)
H(19)	C(18)	H(20)	102(2)	C(12)	C(19)	C(15)	120.9(2)
C(12)	C(19)	H(3)	113.3(15)	C(15)	C(19)	H(3)	125.6(14)
C(10)	C(20)	C(17)	109.8(2)	C(10)	C(20)	H(5)	105.7(12)
C(10)	C(20)	H(6)	107.8(12)	C(17)	C(20)	H(5)	115.3(10)
C(17)	C(20)	H(6)	112.4(10)	H(5)	C(20)	H(6)	105.3(17)
C(16)	C(21)	C(17)	111.5(2)	C(16)	C(21)	H(9)	111.1(13)
C(16)	C(21)	H(10)	104.8(16)	C(17)	C(21)	H(9)	105.3(14)
C(17)	C(21)	H(10)	112.7(19)	H(9)	C(21)	H(10)	111.6(19)

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

atom	atom	atom	angle	atom	atom	atom	angle
N(7)	C(22)	H(15)	108(2)	N(7)	C(22)	H(16)	107(2)
N(7)	C(22)	H(17)	114(2)	H(15)	C(22)	H(16)	110(2)
H(15)	C(22)	H(17)	103(2)	H(16)	C(22)	H(17)	115(2)

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

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)	S(1)	N(7)	N(6)	-57.56(18)	O(2)	S(1)	N(7)	C(22)	75.4(2)
O(2)	S(1)	C(11)	C(8)	77.82(15)	O(2)	S(1)	C(11)	C(12)	-94.18(16)
O(3)	S(1)	N(7)	N(6)	173.08(14)	O(3)	S(1)	N(7)	C(22)	-54.0(2)
O(3)	S(1)	C(11)	C(8)	-149.56(13)	O(3)	S(1)	C(11)	C(12)	38.44(17)
N(7)	S(1)	C(11)	C(8)	-38.12(15)	N(7)	S(1)	C(11)	C(12)	149.88(15)
C(11)	S(1)	N(7)	N(6)	57.35(16)	C(11)	S(1)	N(7)	C(22)	-169.7(2)
N(5)	N(4)	C(10)	C(9)	0.5(3)	N(5)	N(4)	C(10)	C(20)	177.98(17)
C(10)	N(4)	N(5)	C(12)	-178.15(17)	N(4)	N(5)	C(12)	C(11)	170.42(16)
N(4)	N(5)	C(12)	C(19)	-7.3(2)	N(7)	N(6)	C(14)	C(8)	0.7(2)
N(7)	N(6)	C(14)	C(18)	-177.89(16)	C(14)	N(6)	N(7)	S(1)	-43.1(2)
C(14)	N(6)	N(7)	C(22)	-177.7(2)	C(11)	C(8)	C(13)	C(15)	-1.5(2)
C(13)	C(8)	C(11)	S(1)	-172.22(14)	C(13)	C(8)	C(11)	C(12)	-0.3(2)
C(11)	C(8)	C(14)	N(6)	17.1(2)	C(11)	C(8)	C(14)	C(18)	-164.42(18)
C(14)	C(8)	C(11)	S(1)	7.8(2)	C(14)	C(8)	C(11)	C(12)	179.65(16)
C(13)	C(8)	C(14)	N(6)	-162.90(18)	C(13)	C(8)	C(14)	C(18)	15.6(2)
C(14)	C(8)	C(13)	C(15)	178.47(18)	C(10)	C(9)	C(16)	C(21)	50.8(3)
C(16)	C(9)	C(10)	N(4)	124.8(2)	C(16)	C(9)	C(10)	C(20)	-52.8(2)
N(4)	C(10)	C(20)	C(17)	-122.0(2)	C(9)	C(10)	C(20)	C(17)	55.9(3)
S(1)	C(11)	C(12)	N(5)	-4.0(2)	S(1)	C(11)	C(12)	C(19)	173.82(15)
C(8)	C(11)	C(12)	N(5)	-175.55(17)	C(8)	C(11)	C(12)	C(19)	2.3(2)
N(5)	C(12)	C(19)	C(15)	175.4(2)	C(11)	C(12)	C(19)	C(15)	-2.5(3)
C(8)	C(13)	C(15)	C(19)	1.4(3)	C(13)	C(15)	C(19)	C(12)	0.7(3)
C(9)	C(16)	C(21)	C(17)	-53.7(3)	C(20)	C(17)	C(21)	C(16)	56.9(3)
C(21)	C(17)	C(20)	C(10)	-57.0(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(1) <sup>11</sup>	3.490(19)	S(1)	H(2) <sup>11</sup>	3.478(18)
S(1)	H(12) <sup>21</sup>	3.47(2)	O(2)	C(13) <sup>11</sup>	3.444(3)
O(2)	C(16) <sup>21</sup>	3.524(3)	O(2)	H(1) <sup>11</sup>	2.77(2)
O(2)	H(2) <sup>11</sup>	3.05(2)	O(2)	H(2) <sup>31</sup>	3.03(2)
O(2)	H(7) <sup>21</sup>	3.56(2)	O(2)	H(12) <sup>21</sup>	2.50(2)
O(3)	C(15) <sup>11</sup>	3.425(2)	O(3)	H(1) <sup>11</sup>	3.431(17)
O(3)	H(2) <sup>11</sup>	2.965(16)	O(3)	H(3) <sup>41</sup>	3.208(18)
O(3)	H(11) <sup>51</sup>	2.95(2)	O(3)	H(13) <sup>51</sup>	3.13(3)
O(3)	H(14) <sup>51</sup>	3.55(2)	N(4)	H(1) <sup>31</sup>	3.365(17)
N(4)	H(20) <sup>31</sup>	2.88(2)	N(5)	N(5) <sup>41</sup>	3.452(2)
N(5)	C(12) <sup>41</sup>	3.585(2)	N(5)	H(1) <sup>31</sup>	3.521(18)
N(5)	H(4) <sup>41</sup>	3.357(18)	N(6)	H(5) <sup>41</sup>	2.95(2)
N(6)	H(10) <sup>61</sup>	2.90(2)	N(6)	H(10) <sup>21</sup>	3.49(2)
N(6)	H(12) <sup>21</sup>	3.060(19)	N(6)	H(18) <sup>71</sup>	3.09(2)
N(7)	C(20) <sup>41</sup>	3.530(2)	N(7)	H(5) <sup>41</sup>	2.765(19)
N(7)	H(6) <sup>41</sup>	3.351(17)	C(8)	C(19) <sup>31</sup>	3.587(3)
C(8)	H(5) <sup>41</sup>	3.31(2)	C(8)	H(13) <sup>41</sup>	2.96(2)
C(10)	H(20) <sup>31</sup>	3.41(2)	C(11)	C(15) <sup>31</sup>	3.591(3)
C(11)	H(2) <sup>31</sup>	3.365(16)	C(11)	H(5) <sup>41</sup>	3.54(2)
C(11)	H(13) <sup>41</sup>	3.31(2)	C(12)	N(5) <sup>41</sup>	3.585(2)
C(12)	C(13) <sup>31</sup>	3.549(3)	C(12)	C(15) <sup>31</sup>	3.553(3)
C(12)	H(1) <sup>31</sup>	3.518(16)	C(12)	H(2) <sup>31</sup>	3.487(16)
C(12)	H(4) <sup>41</sup>	3.330(19)	C(12)	H(13) <sup>41</sup>	3.518(19)
C(13)	O(2) <sup>81</sup>	3.444(3)	C(13)	C(12) <sup>31</sup>	3.549(3)
C(13)	C(19) <sup>31</sup>	3.367(2)	C(13)	H(3) <sup>31</sup>	3.548(16)
C(13)	H(13) <sup>41</sup>	2.76(2)	C(14)	H(5) <sup>41</sup>	3.15(2)
C(14)	H(8) <sup>61</sup>	3.591(18)	C(14)	H(10) <sup>61</sup>	3.54(2)
C(14)	H(12) <sup>21</sup>	3.050(18)	C(15)	O(3) <sup>81</sup>	3.425(2)
C(15)	C(11) <sup>31</sup>	3.591(3)	C(15)	C(12) <sup>31</sup>	3.553(3)
C(15)	H(13) <sup>41</sup>	2.92(2)	C(15)	H(14) <sup>81</sup>	3.51(2)
C(16)	O(2) <sup>21</sup>	3.524(3)	C(16)	H(3) <sup>11</sup>	3.10(2)
C(16)	H(16) <sup>51</sup>	3.31(3)	C(16)	H(20) <sup>21</sup>	3.45(2)
C(17)	H(9) <sup>91</sup>	3.21(2)	C(17)	H(15) <sup>101</sup>	3.60(3)
C(17)	H(17) <sup>101</sup>	3.33(3)	C(17)	H(19) <sup>111</sup>	3.30(2)
C(18)	H(6) <sup>31</sup>	3.25(2)	C(18)	H(7) <sup>61</sup>	3.488(18)
C(18)	H(7) <sup>31</sup>	3.45(2)	C(18)	H(8) <sup>61</sup>	3.262(18)
C(18)	H(10) <sup>61</sup>	3.54(2)	C(18)	H(10) <sup>21</sup>	3.31(2)

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

atom	atom	distance	atom	atom	distance
C(18)	H(11) <sup>2j</sup>	3.571(16)	C(18)	H(12) <sup>2j</sup>	3.409(19)
C(18)	H(15) <sup>7j</sup>	3.59(2)	C(18)	H(17) <sup>7j</sup>	3.57(3)
C(19)	C(8) <sup>3j</sup>	3.587(3)	C(19)	C(13) <sup>3j</sup>	3.367(2)
C(19)	H(1) <sup>3j</sup>	3.527(14)	C(19)	H(4) <sup>4j</sup>	3.47(2)
C(19)	H(11) <sup>8j</sup>	3.28(2)	C(19)	H(13) <sup>4j</sup>	3.29(2)
C(19)	H(14) <sup>4j</sup>	3.512(17)	C(20)	N(7) <sup>4j</sup>	3.530(2)
C(20)	H(9) <sup>9j</sup>	3.37(2)	C(20)	H(15) <sup>10j</sup>	3.40(3)
C(20)	H(20) <sup>3j</sup>	3.19(2)	C(21)	H(5) <sup>9j</sup>	3.42(2)
C(21)	H(8) <sup>9j</sup>	3.44(2)	C(21)	H(16) <sup>5j</sup>	3.17(3)
C(21)	H(18) <sup>2j</sup>	3.53(2)	C(22)	H(5) <sup>4j</sup>	3.54(2)
C(22)	H(6) <sup>12j</sup>	3.39(2)	C(22)	H(8) <sup>12j</sup>	3.03(2)
C(22)	H(9) <sup>5j</sup>	3.15(2)	C(22)	H(18) <sup>7j</sup>	3.25(2)
C(22)	H(19) <sup>1j</sup>	3.60(2)	H(1)	S(1) <sup>8j</sup>	3.490(19)
H(1)	O(2) <sup>8j</sup>	2.77(2)	H(1)	O(3) <sup>8j</sup>	3.431(17)
H(1)	N(4) <sup>3j</sup>	3.365(17)	H(1)	N(5) <sup>3j</sup>	3.521(18)
H(1)	C(12) <sup>3j</sup>	3.518(16)	H(1)	C(19) <sup>3j</sup>	3.527(14)
H(1)	H(3) <sup>3j</sup>	3.56(2)	H(1)	H(13) <sup>4j</sup>	3.13(3)
H(1)	H(17) <sup>8j</sup>	3.30(3)	H(2)	S(1) <sup>8j</sup>	3.478(18)
H(2)	O(2) <sup>8j</sup>	3.05(2)	H(2)	O(2) <sup>3j</sup>	3.03(2)
H(2)	O(3) <sup>8j</sup>	2.965(16)	H(2)	C(11) <sup>3j</sup>	3.365(16)
H(2)	C(12) <sup>3j</sup>	3.487(16)	H(2)	H(4) <sup>8j</sup>	3.49(3)
H(2)	H(12) <sup>8j</sup>	3.26(3)	H(2)	H(13) <sup>4j</sup>	3.32(3)
H(2)	H(14) <sup>8j</sup>	2.96(2)	H(3)	O(3) <sup>4j</sup>	3.208(18)
H(3)	C(13) <sup>3j</sup>	3.548(16)	H(3)	C(16) <sup>8j</sup>	3.10(2)
H(3)	H(1) <sup>3j</sup>	3.56(2)	H(3)	H(4) <sup>4j</sup>	3.35(3)
H(3)	H(11) <sup>8j</sup>	2.42(3)	H(3)	H(12) <sup>8j</sup>	3.07(3)
H(3)	H(14) <sup>8j</sup>	3.21(3)	H(3)	H(20) <sup>3j</sup>	2.99(2)
H(4)	N(5) <sup>4j</sup>	3.357(18)	H(4)	C(12) <sup>4j</sup>	3.330(19)
H(4)	C(19) <sup>4j</sup>	3.47(2)	H(4)	H(2) <sup>1j</sup>	3.49(3)
H(4)	H(3) <sup>4j</sup>	3.35(3)	H(4)	H(4) <sup>4j</sup>	3.46(2)
H(5)	N(6) <sup>4j</sup>	2.95(2)	H(5)	N(7) <sup>4j</sup>	2.765(19)
H(5)	C(8) <sup>4j</sup>	3.31(2)	H(5)	C(11) <sup>4j</sup>	3.54(2)
H(5)	C(14) <sup>4j</sup>	3.15(2)	H(5)	C(21) <sup>9j</sup>	3.42(2)
H(5)	C(22) <sup>4j</sup>	3.54(2)	H(5)	H(8) <sup>9j</sup>	3.10(3)
H(5)	H(9) <sup>9j</sup>	2.90(3)	H(5)	H(10) <sup>9j</sup>	3.28(3)
H(5)	H(15) <sup>4j</sup>	3.33(3)	H(6)	N(7) <sup>4j</sup>	3.351(17)
H(6)	C(18) <sup>3j</sup>	3.25(2)	H(6)	C(22) <sup>10j</sup>	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(9) <sup>9j</sup>	3.56(3)	H(6)	H(11) <sup>8j</sup>	3.54(3)
H(6)	H(15) <sup>10j</sup>	2.72(4)	H(6)	H(15) <sup>4j</sup>	3.42(3)
H(6)	H(16) <sup>4j</sup>	3.43(3)	H(6)	H(17) <sup>10j</sup>	3.32(3)
H(6)	H(18) <sup>3j</sup>	3.15(3)	H(6)	H(19) <sup>3j</sup>	3.38(3)
H(6)	H(20) <sup>3j</sup>	2.48(3)	H(7)	O(2) <sup>2j</sup>	3.56(2)
H(7)	C(18) <sup>11j</sup>	3.488(18)	H(7)	C(18) <sup>3j</sup>	3.45(2)
H(7)	H(17) <sup>10j</sup>	3.38(4)	H(7)	H(17) <sup>2j</sup>	2.88(3)
H(7)	H(18) <sup>11j</sup>	3.20(2)	H(7)	H(19) <sup>11j</sup>	3.08(3)
H(7)	H(19) <sup>3j</sup>	2.87(4)	H(7)	H(20) <sup>3j</sup>	3.15(3)
H(8)	C(14) <sup>11j</sup>	3.591(18)	H(8)	C(18) <sup>11j</sup>	3.262(18)
H(8)	C(21) <sup>9j</sup>	3.44(2)	H(8)	C(22) <sup>10j</sup>	3.03(2)
H(8)	H(5) <sup>9j</sup>	3.10(3)	H(8)	H(8) <sup>9j</sup>	3.58(3)
H(8)	H(9) <sup>9j</sup>	2.47(3)	H(8)	H(15) <sup>10j</sup>	3.04(4)
H(8)	H(16) <sup>10j</sup>	2.92(4)	H(8)	H(17) <sup>10j</sup>	2.60(4)
H(8)	H(18) <sup>11j</sup>	3.45(2)	H(8)	H(19) <sup>11j</sup>	2.67(2)
H(9)	C(17) <sup>9j</sup>	3.21(2)	H(9)	C(20) <sup>9j</sup>	3.37(2)
H(9)	C(22) <sup>5j</sup>	3.15(2)	H(9)	H(5) <sup>9j</sup>	2.90(3)
H(9)	H(6) <sup>9j</sup>	3.56(3)	H(9)	H(8) <sup>9j</sup>	2.47(3)
H(9)	H(9) <sup>9j</sup>	3.26(3)	H(9)	H(15) <sup>5j</sup>	3.25(4)
H(9)	H(16) <sup>5j</sup>	2.45(4)	H(9)	H(17) <sup>5j</sup>	3.46(4)
H(10)	N(6) <sup>11j</sup>	2.90(2)	H(10)	N(6) <sup>2j</sup>	3.49(2)
H(10)	C(14) <sup>11j</sup>	3.54(2)	H(10)	C(18) <sup>11j</sup>	3.54(2)
H(10)	C(18) <sup>2j</sup>	3.31(2)	H(10)	H(5) <sup>9j</sup>	3.28(3)
H(10)	H(15) <sup>11j</sup>	3.07(4)	H(10)	H(16) <sup>5j</sup>	3.45(4)
H(10)	H(18) <sup>11j</sup>	3.22(3)	H(10)	H(18) <sup>2j</sup>	2.63(3)
H(10)	H(20) <sup>2j</sup>	3.23(3)	H(11)	O(3) <sup>5j</sup>	2.95(2)
H(11)	C(18) <sup>2j</sup>	3.571(16)	H(11)	C(19) <sup>1j</sup>	3.28(2)
H(11)	H(3) <sup>1j</sup>	2.42(3)	H(11)	H(6) <sup>1j</sup>	3.54(3)
H(11)	H(16) <sup>5j</sup>	2.78(3)	H(11)	H(18) <sup>2j</sup>	3.46(2)
H(11)	H(20) <sup>2j</sup>	2.94(2)	H(12)	S(1) <sup>2j</sup>	3.47(2)
H(12)	O(2) <sup>2j</sup>	2.50(2)	H(12)	N(6) <sup>2j</sup>	3.060(19)
H(12)	C(14) <sup>2j</sup>	3.050(18)	H(12)	C(18) <sup>2j</sup>	3.409(19)
H(12)	H(2) <sup>1j</sup>	3.26(3)	H(12)	H(3) <sup>1j</sup>	3.07(3)
H(12)	H(18) <sup>2j</sup>	3.37(3)	H(12)	H(20) <sup>2j</sup>	3.21(3)
H(13)	O(3) <sup>5j</sup>	3.13(3)	H(13)	C(8) <sup>4j</sup>	2.96(2)
H(13)	C(11) <sup>4j</sup>	3.31(2)	H(13)	C(12) <sup>4j</sup>	3.518(19)
H(13)	C(13) <sup>4j</sup>	2.76(2)	H(13)	C(15) <sup>4j</sup>	2.92(2)

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

atom	atom	distance	atom	atom	distance
H(13)	C(19) <sup>4)</sup>	3.29(2)	H(13)	H(1) <sup>4)</sup>	3.13(3)
H(13)	H(2) <sup>4)</sup>	3.32(3)	H(13)	H(16) <sup>5)</sup>	3.17(3)
H(14)	O(3) <sup>5)</sup>	3.55(2)	H(14)	C(15) <sup>1)</sup>	3.51(2)
H(14)	C(19) <sup>4)</sup>	3.512(17)	H(14)	H(2) <sup>1)</sup>	2.96(2)
H(14)	H(3) <sup>1)</sup>	3.21(3)	H(15)	C(17) <sup>12)</sup>	3.60(3)
H(15)	C(18) <sup>7)</sup>	3.59(2)	H(15)	C(20) <sup>12)</sup>	3.40(3)
H(15)	H(5) <sup>4)</sup>	3.33(3)	H(15)	H(6) <sup>12)</sup>	2.72(4)
H(15)	H(6) <sup>4)</sup>	3.42(3)	H(15)	H(8) <sup>12)</sup>	3.04(4)
H(15)	H(9) <sup>5)</sup>	3.25(4)	H(15)	H(10) <sup>6)</sup>	3.07(4)
H(15)	H(15) <sup>13)</sup>	3.23(3)	H(15)	H(18) <sup>7)</sup>	2.79(4)
H(15)	H(20) <sup>7)</sup>	3.44(3)	H(16)	C(16) <sup>5)</sup>	3.31(3)
H(16)	C(21) <sup>5)</sup>	3.17(3)	H(16)	H(6) <sup>4)</sup>	3.43(3)
H(16)	H(8) <sup>12)</sup>	2.92(4)	H(16)	H(9) <sup>5)</sup>	2.45(4)
H(16)	H(10) <sup>5)</sup>	3.45(4)	H(16)	H(11) <sup>5)</sup>	2.78(3)
H(16)	H(13) <sup>5)</sup>	3.17(3)	H(17)	C(17) <sup>12)</sup>	3.33(3)
H(17)	C(18) <sup>7)</sup>	3.57(3)	H(17)	H(1) <sup>1)</sup>	3.30(3)
H(17)	H(6) <sup>12)</sup>	3.32(3)	H(17)	H(7) <sup>12)</sup>	3.38(4)
H(17)	H(7) <sup>2)</sup>	2.88(3)	H(17)	H(8) <sup>12)</sup>	2.60(4)
H(17)	H(9) <sup>5)</sup>	3.46(4)	H(17)	H(18) <sup>7)</sup>	2.87(4)
H(17)	H(19) <sup>1)</sup>	2.72(4)	H(17)	H(19) <sup>7)</sup>	3.55(4)
H(18)	N(6) <sup>7)</sup>	3.09(2)	H(18)	C(21) <sup>2)</sup>	3.53(2)
H(18)	C(22) <sup>7)</sup>	3.25(2)	H(18)	H(6) <sup>3)</sup>	3.15(3)
H(18)	H(7) <sup>6)</sup>	3.20(2)	H(18)	H(8) <sup>6)</sup>	3.45(2)
H(18)	H(10) <sup>6)</sup>	3.22(3)	H(18)	H(10) <sup>2)</sup>	2.63(3)
H(18)	H(11) <sup>2)</sup>	3.46(2)	H(18)	H(12) <sup>2)</sup>	3.37(3)
H(18)	H(15) <sup>7)</sup>	2.79(4)	H(18)	H(17) <sup>7)</sup>	2.87(4)
H(18)	H(18) <sup>7)</sup>	3.53(4)	H(19)	C(17) <sup>6)</sup>	3.30(2)
H(19)	C(22) <sup>8)</sup>	3.60(2)	H(19)	H(6) <sup>3)</sup>	3.38(3)
H(19)	H(7) <sup>6)</sup>	3.08(3)	H(19)	H(7) <sup>3)</sup>	2.87(4)
H(19)	H(8) <sup>6)</sup>	2.67(2)	H(19)	H(17) <sup>8)</sup>	2.72(4)
H(19)	H(17) <sup>7)</sup>	3.55(4)	H(20)	N(4) <sup>3)</sup>	2.88(2)
H(20)	C(10) <sup>3)</sup>	3.41(2)	H(20)	C(16) <sup>2)</sup>	3.45(2)
H(20)	C(20) <sup>3)</sup>	3.19(2)	H(20)	H(3) <sup>3)</sup>	2.99(2)
H(20)	H(6) <sup>3)</sup>	2.48(3)	H(20)	H(7) <sup>3)</sup>	3.15(3)
H(20)	H(10) <sup>2)</sup>	3.23(3)	H(20)	H(11) <sup>2)</sup>	2.94(2)
H(20)	H(12) <sup>2)</sup>	3.21(3)	H(20)	H(15) <sup>7)</sup>	3.44(3)

Symmetry Operators:

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

Table 7. Intramolecular and Intermolecular Hydrogen bonds

D	H	A	D...A	D-H	H...A	D-H...A
N(5)	H(4)	O(3)	2.834(3)	0.82(2)	2.19(2)	136(2)

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.