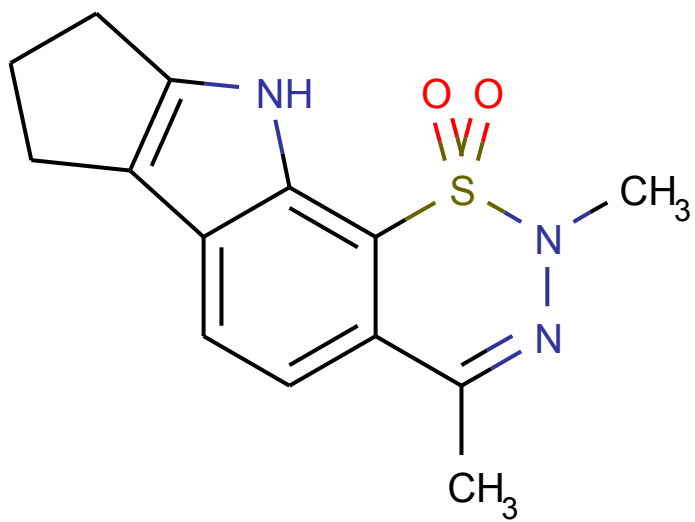


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PGY0784_1A

Submitted by: Pusztai Gyongyver
Operator: Dancso Andras

X-ray Structure Report



November 26, 2024



Fig. 1. The crystal

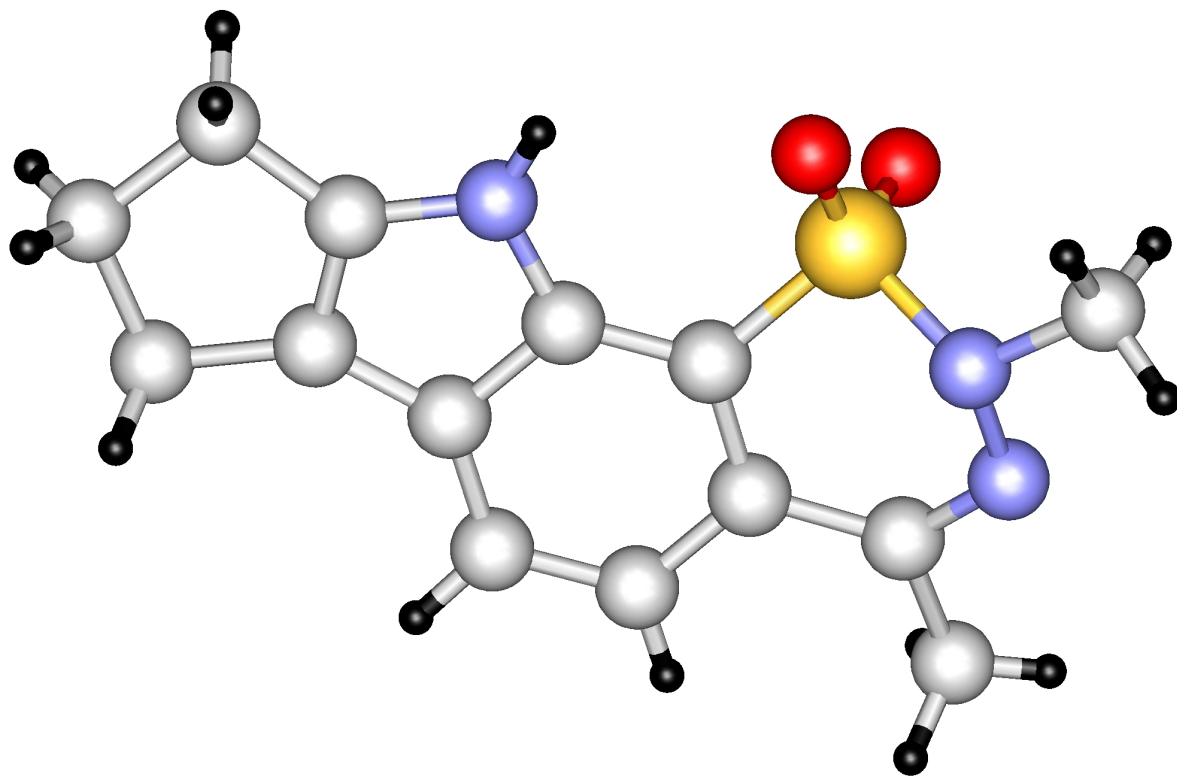


Fig. 2. The molecule

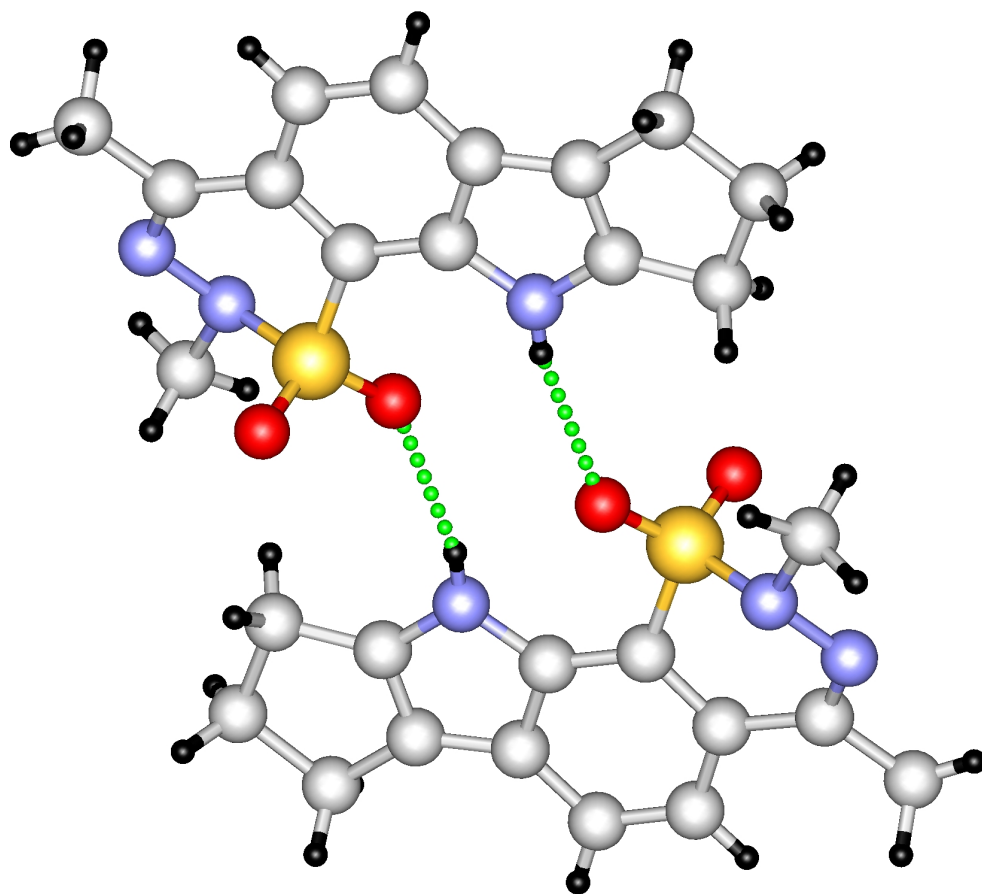


Fig. 3. Hydrogen bonds

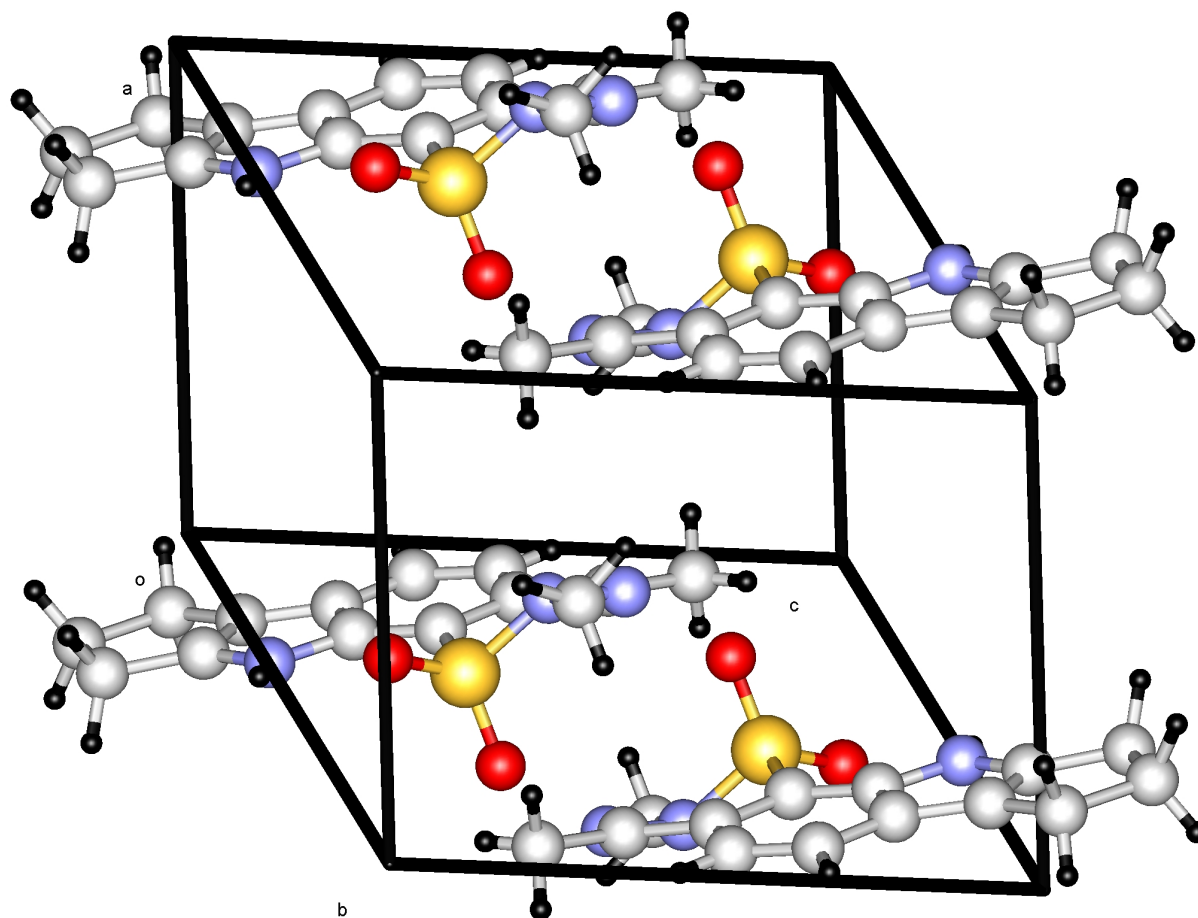


Fig. 4. Packing

Experimental

Data Collection

A colorless chunk crystal of $C_{14}H_{15}N_3O_2S$ having approximate dimensions of 0.31 x 0.20 x 0.11 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 180 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:

$$\begin{aligned}a &= 7.4367(4) \text{ \AA} & \alpha &= 100.874(3)^\circ \\b &= 8.3023(4) \text{ \AA} & \beta &= 104.217(3)^\circ \\c &= 12.3159(7) \text{ \AA} & \gamma &= 109.047(3)^\circ \\V &= 666.11(6) \text{ \AA}^3\end{aligned}$$

For $Z = 2$ and F.W. = 289.35, the calculated density is 1.443 g/cm³. 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 2θ value of 143.1° . 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 7553 reflections that were collected, 2268 were unique ($R_{\text{int}} = 0.040$).

The linear absorption coefficient, μ , for Cu-K α radiation is 22.116 cm⁻¹. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.637 to 0.781. 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. Hydrogen atoms were refined isotropically. The final cycle of full-matrix least-squares refinement³ on F was based on 4139 observed reflections ($I > 2.00\sigma(I)$) and 241 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.0337$$

$$R_w = [\sum w (|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.0365$$

The standard deviation of an observation of unit weight⁴ was 3.30. 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 0.55 and -1.04 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

(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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(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	$\text{C}_{14}\text{H}_{15}\text{N}_3\text{O}_2\text{S}$
Formula Weight	289.35
Crystal Color, Habit	colorless, chunk
Crystal Dimensions	0.31 X 0.20 X 0.11 mm
Crystal System	triclinic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 180.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 7.4367(4) \text{ \AA}$ $b = 8.3023(4) \text{ \AA}$ $c = 12.3159(7) \text{ \AA}$ $\alpha = 100.874(3)^\circ$ $\beta = 104.217(3)^\circ$ $\gamma = 109.047(3)^\circ$ $V = 666.11(6) \text{ \AA}^3$
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.443 g/cm^3
F_{000}	304.00
$\mu(\text{CuK}\alpha)$	22.116 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°
Exposure Rate	36.0 sec./°
ω oscillation Range (χ =54.0, ϕ =0.0)	20.0 - 200.0°
Exposure Rate	36.0 sec./°
ω oscillation Range (χ =54.0, ϕ =90.0)	20.0 - 200.0°
Exposure Rate	36.0 sec./°
ω oscillation Range (χ =54.0, ϕ =180.0)	20.0 - 200.0°
Exposure Rate	36.0 sec./°
ω oscillation Range (χ =54.0, ϕ =270.0)	20.0 - 200.0°
Exposure Rate	36.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\text{max}}$	143.1°
No. of Reflections Measured	Total: 7553 Unique: 2268 (R_{int} = 0.040)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.637 - 0.781)

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	143.1 $^{\circ}$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 2.00\sigma(I)$)	4139
No. Variables	241
Reflection/Parameter Ratio	17.17
Residuals: R ($I > 2.00\sigma(I)$)	0.0337
Residuals: Rw ($I > 2.00\sigma(I)$)	0.0365
Goodness of Fit Indicator	3.295
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.55 e $^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	-1.04 e $^{-}/\text{\AA}^3$

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

atom	x	y	z	B_{eq}
S(1)	0.86716(12)	0.39937(9)	0.75847(8)	3.78(2)
O(2)	1.0075(2)	0.3287(2)	0.72900(16)	4.84(5)
O(3)	0.8493(2)	0.4072(2)	0.87339(16)	4.50(5)
N(4)	1.1246(3)	0.7989(3)	0.9390(2)	3.43(7)
N(5)	0.6378(3)	0.2929(2)	0.6605(2)	3.88(6)
N(6)	0.6176(3)	0.2911(2)	0.5432(2)	3.92(7)
C(7)	0.9158(4)	0.6053(3)	0.7333(2)	2.83(8)
C(8)	0.7136(4)	0.4398(3)	0.5249(2)	3.28(8)
C(9)	1.0921(4)	0.9307(3)	0.7947(2)	3.25(8)
C(10)	1.0188(4)	0.9271(4)	0.6777(3)	3.78(9)
C(11)	1.2141(4)	1.0638(3)	0.9018(2)	3.30(8)
C(12)	1.0369(4)	0.7636(3)	0.8201(2)	2.90(8)
C(13)	0.8457(4)	0.6053(3)	0.6166(2)	3.05(8)
C(14)	0.9006(4)	0.7702(4)	0.5912(3)	3.72(9)
C(15)	1.2308(4)	0.9811(4)	0.9860(2)	3.29(8)
C(16)	1.4213(5)	1.2863(4)	1.0853(3)	4.24(10)
C(17)	0.6715(6)	0.4281(6)	0.3971(3)	4.51(11)
C(18)	1.3581(5)	1.0996(4)	1.1060(3)	3.72(9)
C(19)	1.3338(5)	1.2612(4)	0.9522(3)	4.36(10)
C(20)	0.5243(7)	0.1128(5)	0.6666(4)	6.04(12)
H(1)	1.106(3)	0.725(2)	0.9773(18)	2.2(6)
H(2)	1.045(2)	1.040(2)	0.6605(17)	3.1(5)
H(3)	0.851(3)	0.766(3)	0.513(2)	3.5(7)
H(4)	1.245(4)	1.326(3)	0.940(2)	7.0(9)
H(5)	1.448(3)	1.298(3)	0.917(2)	6.9(9)
H(6)	1.574(4)	1.343(3)	1.108(2)	7.5(9)
H(7)	1.365(3)	1.359(3)	1.130(2)	6.2(8)
H(8)	1.276(3)	1.087(3)	1.161(2)	5.4(7)
H(9)	1.482(3)	1.070(3)	1.135(2)	5.4(7)
H(10)	0.507(3)	0.130(3)	0.747(2)	7.1(10)
H(11)	0.604(5)	0.039(5)	0.654(3)	14.2(18)
H(12)	0.379(5)	0.065(4)	0.607(2)	11.6(13)
H(13)	0.605(4)	0.318(3)	0.349(2)	7.4(11)
H(14)	0.801(4)	0.468(3)	0.378(2)	8.8(11)
H(15)	0.593(4)	0.504(3)	0.376(2)	8.6(10)

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

Table 2. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	0.0604(6)	0.0314(4)	0.0457(6)	0.0128(4)	0.0114(5)	0.0149(4)
O(2)	0.0701(14)	0.0490(12)	0.0786(16)	0.0378(11)	0.0239(12)	0.0245(12)
O(3)	0.0794(15)	0.0422(12)	0.0388(14)	0.0107(10)	0.0141(11)	0.0187(11)
N(4)	0.0559(18)	0.0327(17)	0.039(2)	0.0125(14)	0.0128(15)	0.0170(17)
N(5)	0.0524(17)	0.0364(15)	0.0391(18)	0.0025(13)	0.0047(14)	0.0084(15)
N(6)	0.0590(17)	0.0414(16)	0.0351(19)	0.0119(13)	0.0067(14)	0.0074(15)
C(7)	0.0396(19)	0.0305(18)	0.036(2)	0.0114(15)	0.0125(16)	0.0099(19)
C(8)	0.042(2)	0.040(2)	0.041(2)	0.0172(17)	0.0125(18)	0.009(2)
C(9)	0.048(2)	0.0335(19)	0.043(2)	0.0154(16)	0.0151(18)	0.015(2)
C(10)	0.057(2)	0.036(2)	0.050(2)	0.0146(18)	0.0154(19)	0.021(2)
C(11)	0.052(2)	0.035(2)	0.036(2)	0.0149(17)	0.0131(18)	0.011(2)
C(12)	0.043(2)	0.0326(19)	0.035(2)	0.0155(16)	0.0112(17)	0.012(2)
C(13)	0.0422(19)	0.0322(18)	0.033(2)	0.0097(16)	0.0077(16)	0.0062(19)
C(14)	0.057(2)	0.045(2)	0.034(2)	0.0165(18)	0.009(2)	0.016(2)
C(15)	0.043(2)	0.0345(19)	0.035(2)	0.0081(16)	0.0076(18)	0.004(2)
C(16)	0.060(2)	0.041(2)	0.048(3)	0.014(2)	0.010(2)	0.006(2)
C(17)	0.075(3)	0.055(2)	0.026(2)	0.017(2)	0.009(2)	0.001(2)
C(18)	0.052(2)	0.040(2)	0.044(2)	0.0149(19)	0.014(2)	0.010(2)
C(19)	0.069(2)	0.030(2)	0.047(2)	0.008(2)	0.009(2)	0.001(2)
C(20)	0.081(3)	0.051(2)	0.070(3)	-0.008(2)	0.018(2)	0.028(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)	1.439(2)	S(1)	O(3)	1.445(2)
S(1)	N(5)	1.658(2)	S(1)	C(7)	1.732(3)
N(4)	C(12)	1.379(4)	N(4)	C(15)	1.383(3)
N(4)	H(1)	0.84(2)	N(5)	N(6)	1.413(3)
N(5)	C(20)	1.485(4)	N(6)	C(8)	1.301(3)
C(7)	C(12)	1.380(3)	C(7)	C(13)	1.404(4)
C(8)	C(13)	1.455(3)	C(8)	C(17)	1.505(5)
C(9)	C(10)	1.401(5)	C(9)	C(11)	1.411(3)
C(9)	C(12)	1.431(4)	C(10)	C(14)	1.369(3)
C(10)	H(2)	0.97(2)	C(11)	C(15)	1.350(5)
C(11)	C(19)	1.502(3)	C(13)	C(14)	1.414(5)
C(14)	H(3)	0.93(2)	C(15)	C(18)	1.485(4)
C(16)	C(18)	1.556(5)	C(16)	C(19)	1.553(5)
C(16)	H(6)	1.02(2)	C(16)	H(7)	0.99(3)
C(17)	H(13)	0.89(2)	C(17)	H(14)	1.01(3)
C(17)	H(15)	1.01(3)	C(18)	H(8)	1.02(2)
C(18)	H(9)	1.03(2)	C(19)	H(4)	0.98(3)
C(19)	H(5)	1.03(3)	C(20)	H(10)	1.02(3)
C(20)	H(11)	1.00(4)	C(20)	H(12)	1.04(3)

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

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	S(1)	O(3)	117.24(13)	O(2)	S(1)	N(5)	111.80(11)
O(2)	S(1)	C(7)	109.49(14)	O(3)	S(1)	N(5)	107.89(12)
O(3)	S(1)	C(7)	111.01(14)	N(5)	S(1)	C(7)	97.67(13)
C(12)	N(4)	C(15)	107.8(2)	C(12)	N(4)	H(1)	126.1(11)
C(15)	N(4)	H(1)	125.8(12)	S(1)	N(5)	N(6)	117.12(18)
S(1)	N(5)	C(20)	115.2(2)	N(6)	N(5)	C(20)	110.7(2)
N(5)	N(6)	C(8)	117.3(2)	S(1)	C(7)	C(12)	122.7(2)
S(1)	C(7)	C(13)	116.89(19)	C(12)	C(7)	C(13)	120.1(2)
N(6)	C(8)	C(13)	124.8(3)	N(6)	C(8)	C(17)	113.5(2)
C(13)	C(8)	C(17)	121.7(3)	C(10)	C(9)	C(11)	135.8(3)
C(10)	C(9)	C(12)	117.1(2)	C(11)	C(9)	C(12)	107.0(3)
C(9)	C(10)	C(14)	121.4(3)	C(9)	C(10)	H(2)	117.9(11)
C(14)	C(10)	H(2)	120.5(11)	C(9)	C(11)	C(15)	107.2(2)
C(9)	C(11)	C(19)	141.8(3)	C(15)	C(11)	C(19)	110.9(2)
N(4)	C(12)	C(7)	131.2(3)	N(4)	C(12)	C(9)	107.1(2)
C(7)	C(12)	C(9)	121.6(3)	C(7)	C(13)	C(8)	120.5(2)
C(7)	C(13)	C(14)	118.5(2)	C(8)	C(13)	C(14)	121.0(2)
C(10)	C(14)	C(13)	121.3(3)	C(10)	C(14)	H(3)	122.2(14)
C(13)	C(14)	H(3)	116.5(13)	N(4)	C(15)	C(11)	110.8(2)
N(4)	C(15)	C(18)	133.8(3)	C(11)	C(15)	C(18)	115.4(2)
C(18)	C(16)	C(19)	108.5(2)	C(18)	C(16)	H(6)	109.5(18)
C(18)	C(16)	H(7)	108.6(17)	C(19)	C(16)	H(6)	107.0(17)
C(19)	C(16)	H(7)	110.1(16)	H(6)	C(16)	H(7)	113(2)
C(8)	C(17)	H(13)	115(2)	C(8)	C(17)	H(14)	111.3(16)
C(8)	C(17)	H(15)	109.3(18)	H(13)	C(17)	H(14)	102(2)
H(13)	C(17)	H(15)	109(2)	H(14)	C(17)	H(15)	111(2)
C(15)	C(18)	C(16)	101.7(3)	C(15)	C(18)	H(8)	110.0(10)
C(15)	C(18)	H(9)	109.9(12)	C(16)	C(18)	H(8)	113.0(15)
C(16)	C(18)	H(9)	110.5(13)	H(8)	C(18)	H(9)	111(2)
C(11)	C(19)	C(16)	103.4(2)	C(11)	C(19)	H(4)	110.4(12)
C(11)	C(19)	H(5)	109.1(14)	C(16)	C(19)	H(4)	109.3(16)
C(16)	C(19)	H(5)	110.8(14)	H(4)	C(19)	H(5)	113(2)
N(5)	C(20)	H(10)	106.4(14)	N(5)	C(20)	H(11)	108(2)
N(5)	C(20)	H(12)	108(2)	H(10)	C(20)	H(11)	113(3)
H(10)	C(20)	H(12)	105(2)	H(11)	C(20)	H(12)	116(2)

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

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)	S(1)	N(5)	N(6)	-57.9(2)	O(2)	S(1)	N(5)	C(20)	74.9(3)
O(2)	S(1)	C(7)	C(12)	-94.4(3)	O(2)	S(1)	C(7)	C(13)	78.3(2)
O(3)	S(1)	N(5)	N(6)	171.8(2)	O(3)	S(1)	N(5)	C(20)	-55.4(3)
O(3)	S(1)	C(7)	C(12)	36.6(3)	O(3)	S(1)	C(7)	C(13)	-150.7(2)
N(5)	S(1)	C(7)	C(12)	149.1(3)	N(5)	S(1)	C(7)	C(13)	-38.2(2)
C(7)	S(1)	N(5)	N(6)	56.7(2)	C(7)	S(1)	N(5)	C(20)	-170.5(3)
C(12)	N(4)	C(15)	C(11)	0.2(4)	C(12)	N(4)	C(15)	C(18)	-178.6(4)
C(15)	N(4)	C(12)	C(7)	-179.7(3)	C(15)	N(4)	C(12)	C(9)	-0.2(3)
S(1)	N(5)	N(6)	C(8)	-43.6(3)	C(20)	N(5)	N(6)	C(8)	-178.5(3)
N(5)	N(6)	C(8)	C(13)	2.5(4)	N(5)	N(6)	C(8)	C(17)	-176.0(3)
S(1)	C(7)	C(12)	N(4)	-7.2(5)	S(1)	C(7)	C(12)	C(9)	173.3(2)
S(1)	C(7)	C(13)	C(8)	8.7(4)	S(1)	C(7)	C(13)	C(14)	-173.5(2)
C(12)	C(7)	C(13)	C(8)	-178.4(3)	C(12)	C(7)	C(13)	C(14)	-0.5(5)
C(13)	C(7)	C(12)	N(4)	-179.7(3)	C(13)	C(7)	C(12)	C(9)	0.8(5)
N(6)	C(8)	C(13)	C(7)	15.0(5)	N(6)	C(8)	C(13)	C(14)	-162.8(3)
C(17)	C(8)	C(13)	C(7)	-166.7(3)	C(17)	C(8)	C(13)	C(14)	15.5(5)
C(10)	C(9)	C(11)	C(15)	-180(179)	C(10)	C(9)	C(11)	C(19)	-2.4(8)
C(11)	C(9)	C(10)	C(14)	179.5(4)	C(10)	C(9)	C(12)	N(4)	-180.0(2)
C(10)	C(9)	C(12)	C(7)	-0.4(5)	C(12)	C(9)	C(10)	C(14)	-0.4(5)
C(11)	C(9)	C(12)	N(4)	0.1(3)	C(11)	C(9)	C(12)	C(7)	179.7(3)
C(12)	C(9)	C(11)	C(15)	0.0(3)	C(12)	C(9)	C(11)	C(19)	177.6(4)
C(9)	C(10)	C(14)	C(13)	0.7(5)	C(9)	C(11)	C(15)	N(4)	-0.1(3)
C(9)	C(11)	C(15)	C(18)	178.9(3)	C(9)	C(11)	C(19)	C(16)	-179.8(3)
C(15)	C(11)	C(19)	C(16)	-2.3(4)	C(19)	C(11)	C(15)	N(4)	-178.5(3)
C(19)	C(11)	C(15)	C(18)	0.5(4)	C(7)	C(13)	C(14)	C(10)	-0.2(5)
C(8)	C(13)	C(14)	C(10)	177.6(3)	N(4)	C(15)	C(18)	C(16)	-179.7(3)
C(11)	C(15)	C(18)	C(16)	1.5(4)	C(18)	C(16)	C(19)	C(11)	3.2(4)
C(19)	C(16)	C(18)	C(15)	-2.9(4)					

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) ¹¹	3.57(2)	S(1)	H(4) ²¹	3.43(3)
S(1)	H(7) ³¹	3.36(3)	S(1)	H(14) ⁴¹	3.33(3)
O(2)	C(10) ²¹	3.308(4)	O(2)	C(17) ⁴¹	3.471(5)
O(2)	C(19) ²¹	3.457(4)	O(2)	H(2) ²¹	2.51(2)
O(2)	H(3) ⁴¹	3.44(2)	O(2)	H(4) ²¹	2.77(2)
O(2)	H(6) ⁵¹	3.23(2)	O(2)	H(14) ⁴¹	2.61(3)
O(2)	H(15) ⁴¹	3.51(3)	O(3)	O(3) ¹¹	3.102(2)
O(3)	N(4) ¹¹	3.130(3)	O(3)	H(1) ¹¹	2.32(2)
O(3)	H(4) ²¹	3.18(3)	O(3)	H(4) ³¹	3.25(3)
O(3)	H(5) ⁶¹	3.04(3)	O(3)	H(7) ³¹	2.89(3)
N(4)	O(3) ¹¹	3.130(3)	N(4)	H(4) ³¹	3.40(3)
N(4)	H(6) ⁵¹	2.98(3)	N(4)	H(7) ³¹	3.27(2)
N(4)	H(8) ³¹	3.43(2)	N(4)	H(9) ⁵¹	3.19(2)
N(5)	H(3) ⁷¹	3.56(2)	N(5)	H(7) ³¹	3.51(2)
N(5)	H(15) ⁷¹	2.79(3)	N(6)	C(20) ⁸¹	3.480(4)
N(6)	H(3) ⁷¹	3.23(2)	N(6)	H(11) ⁸¹	2.95(3)
N(6)	H(12) ⁸¹	3.19(3)	N(6)	H(15) ⁷¹	2.86(3)
C(7)	H(7) ³¹	3.04(3)	C(7)	H(8) ³¹	3.50(2)
C(7)	H(14) ⁴¹	2.94(3)	C(7)	H(15) ⁷¹	3.43(2)
C(8)	C(8) ⁷¹	3.580(4)	C(8)	H(14) ⁴¹	3.28(3)
C(8)	H(15) ⁷¹	2.95(3)	C(9)	H(8) ³¹	2.88(2)
C(9)	H(9) ⁵¹	3.07(2)	C(9)	H(10) ⁹¹	3.25(3)
C(9)	H(12) ⁹¹	3.60(4)	C(10)	O(2) ¹⁰¹	3.308(4)
C(10)	H(8) ³¹	3.29(2)	C(10)	H(10) ⁹¹	3.27(2)
C(10)	H(11) ¹⁰¹	3.46(4)	C(10)	H(12) ⁹¹	2.96(4)
C(11)	H(8) ³¹	3.27(2)	C(11)	H(9) ⁵¹	2.92(3)
C(11)	H(10) ⁹¹	3.22(3)	C(12)	H(6) ⁵¹	3.26(3)
C(12)	H(7) ³¹	3.09(3)	C(12)	H(8) ³¹	3.01(3)
C(12)	H(9) ⁵¹	3.24(2)	C(12)	H(14) ⁴¹	3.50(3)
C(13)	H(14) ⁴¹	2.87(3)	C(13)	H(15) ⁷¹	3.11(3)
C(14)	H(12) ⁹¹	3.53(3)	C(14)	H(12) ⁷¹	3.58(4)
C(14)	H(14) ⁴¹	3.42(3)	C(15)	H(6) ⁵¹	3.59(3)
C(15)	H(8) ³¹	3.56(2)	C(15)	H(9) ⁵¹	2.98(3)
C(16)	C(17) ¹¹¹	3.599(5)	C(16)	H(4) ¹²¹	3.49(2)
C(16)	H(5) ¹²¹	3.28(2)	C(16)	H(13) ¹¹¹	3.12(3)
C(16)	H(15) ¹¹¹	3.40(2)	C(17)	O(2) ⁴¹	3.471(5)
C(17)	C(16) ¹³¹	3.599(5)	C(17)	H(6) ¹³¹	3.34(2)

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

atom	atom	distance	atom	atom	distance
C(17)	H(7) ¹³	3.33(2)	C(17)	H(8) ¹³	3.519(18)
C(17)	H(9) ¹³	3.56(2)	C(17)	H(11) ⁸	3.59(3)
C(18)	H(10) ¹¹	3.10(3)	C(18)	H(11) ¹¹	3.35(4)
C(18)	H(13) ¹¹	2.95(2)	C(19)	O(2) ¹⁰	3.457(4)
C(19)	H(4) ¹²	3.54(2)	C(19)	H(5) ¹²	3.35(2)
C(19)	H(6) ¹²	3.39(3)	C(19)	H(9) ⁵	3.56(2)
C(19)	H(10) ⁹	3.27(3)	C(20)	N(6) ⁸	3.480(4)
C(20)	H(2) ⁶	3.40(2)	C(20)	H(5) ⁶	3.44(3)
C(20)	H(8) ¹¹	3.29(2)	C(20)	H(9) ¹¹	3.12(2)
C(20)	H(13) ⁸	3.34(3)	H(1)	S(1) ¹¹	3.57(2)
H(1)	O(3) ¹¹	2.32(2)	H(1)	H(4) ³	2.96(4)
H(1)	H(5) ⁵	3.33(3)	H(1)	H(6) ⁵	3.00(4)
H(1)	H(7) ³	3.21(3)	H(1)	H(10) ¹¹	3.56(3)
H(2)	O(2) ¹⁰	2.51(2)	H(2)	C(20) ⁹	3.40(2)
H(2)	H(3) ¹⁴	3.01(3)	H(2)	H(10) ⁹	3.12(3)
H(2)	H(11) ¹⁰	3.26(5)	H(2)	H(12) ⁹	2.67(4)
H(3)	O(2) ⁴	3.44(2)	H(3)	N(5) ⁷	3.56(2)
H(3)	N(6) ⁷	3.23(2)	H(3)	H(2) ¹⁴	3.01(3)
H(3)	H(12) ⁷	2.86(4)	H(4)	S(1) ¹⁰	3.43(3)
H(4)	O(2) ¹⁰	2.77(2)	H(4)	O(3) ¹⁰	3.18(3)
H(4)	O(3) ³	3.25(3)	H(4)	N(4) ³	3.40(3)
H(4)	C(16) ¹²	3.49(2)	H(4)	C(19) ¹²	3.54(2)
H(4)	H(1) ³	2.96(4)	H(4)	H(5) ¹²	3.06(2)
H(4)	H(6) ¹²	2.88(4)	H(5)	O(3) ⁹	3.04(3)
H(5)	C(16) ¹²	3.28(2)	H(5)	C(19) ¹²	3.35(2)
H(5)	C(20) ⁹	3.44(3)	H(5)	H(1) ⁵	3.33(3)
H(5)	H(4) ¹²	3.06(2)	H(5)	H(5) ¹²	3.32(3)
H(5)	H(6) ¹²	3.10(4)	H(5)	H(7) ¹²	2.97(3)
H(5)	H(9) ⁵	3.24(4)	H(5)	H(10) ⁹	2.50(4)
H(6)	O(2) ⁵	3.23(2)	H(6)	N(4) ⁵	2.98(3)
H(6)	C(12) ⁵	3.26(3)	H(6)	C(15) ⁵	3.59(3)
H(6)	C(17) ¹¹	3.34(2)	H(6)	C(19) ¹²	3.39(3)
H(6)	H(1) ⁵	3.00(4)	H(6)	H(4) ¹²	2.88(4)
H(6)	H(5) ¹²	3.10(4)	H(6)	H(13) ¹¹	2.97(4)
H(6)	H(14) ¹¹	3.12(3)	H(6)	H(15) ¹¹	3.27(4)
H(7)	S(1) ³	3.36(3)	H(7)	O(3) ³	2.89(3)
H(7)	N(4) ³	3.27(2)	H(7)	N(5) ³	3.51(2)

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

atom	atom	distance	atom	atom	distance
H(7)	C(7) ^{3j}	3.04(3)	H(7)	C(12) ^{3j}	3.09(3)
H(7)	C(17) ^{11j}	3.33(2)	H(7)	H(1) ^{3j}	3.21(3)
H(7)	H(5) ^{12j}	2.97(3)	H(7)	H(13) ^{11j}	2.99(4)
H(7)	H(14) ^{11j}	3.58(3)	H(7)	H(15) ^{11j}	2.88(3)
H(8)	N(4) ^{3j}	3.43(2)	H(8)	C(7) ^{3j}	3.50(2)
H(8)	C(9) ^{3j}	2.88(2)	H(8)	C(10) ^{3j}	3.29(2)
H(8)	C(11) ^{3j}	3.27(2)	H(8)	C(12) ^{3j}	3.01(3)
H(8)	C(15) ^{3j}	3.56(2)	H(8)	C(17) ^{11j}	3.519(18)
H(8)	C(20) ^{1j}	3.29(2)	H(8)	H(10) ^{1j}	2.99(4)
H(8)	H(11) ^{1j}	2.75(5)	H(8)	H(13) ^{11j}	2.74(3)
H(8)	H(15) ^{11j}	3.56(3)	H(9)	N(4) ^{5j}	3.19(2)
H(9)	C(9) ^{5j}	3.07(2)	H(9)	C(11) ^{5j}	2.92(3)
H(9)	C(12) ^{5j}	3.24(2)	H(9)	C(15) ^{5j}	2.98(3)
H(9)	C(17) ^{11j}	3.56(2)	H(9)	C(19) ^{5j}	3.56(2)
H(9)	C(20) ^{1j}	3.12(2)	H(9)	H(5) ^{5j}	3.24(4)
H(9)	H(9) ^{5j}	3.41(3)	H(9)	H(10) ^{1j}	2.42(4)
H(9)	H(11) ^{1j}	3.06(5)	H(9)	H(12) ^{1j}	3.60(4)
H(9)	H(13) ^{11j}	2.75(3)	H(10)	C(9) ^{6j}	3.25(3)
H(10)	C(10) ^{6j}	3.27(2)	H(10)	C(11) ^{6j}	3.22(3)
H(10)	C(18) ^{1j}	3.10(3)	H(10)	C(19) ^{6j}	3.27(3)
H(10)	H(1) ^{1j}	3.56(3)	H(10)	H(2) ^{6j}	3.12(3)
H(10)	H(5) ^{6j}	2.50(4)	H(10)	H(8) ^{1j}	2.99(4)
H(10)	H(9) ^{1j}	2.42(4)	H(10)	H(13) ^{8j}	3.44(4)
H(11)	N(6) ^{8j}	2.95(3)	H(11)	C(10) ^{2j}	3.46(4)
H(11)	C(17) ^{8j}	3.59(3)	H(11)	C(18) ^{1j}	3.35(4)
H(11)	H(2) ^{2j}	3.26(5)	H(11)	H(8) ^{1j}	2.75(5)
H(11)	H(9) ^{1j}	3.06(5)	H(11)	H(11) ^{8j}	3.57(5)
H(11)	H(12) ^{8j}	3.22(5)	H(11)	H(13) ^{8j}	2.85(4)
H(12)	N(6) ^{8j}	3.19(3)	H(12)	C(9) ^{6j}	3.60(4)
H(12)	C(10) ^{6j}	2.96(4)	H(12)	C(14) ^{6j}	3.53(3)
H(12)	C(14) ^{7j}	3.58(4)	H(12)	H(2) ^{6j}	2.67(4)
H(12)	H(3) ^{7j}	2.86(4)	H(12)	H(9) ^{1j}	3.60(4)
H(12)	H(11) ^{8j}	3.22(5)	H(12)	H(13) ^{8j}	3.36(5)
H(12)	H(15) ^{7j}	3.48(4)	H(13)	C(16) ^{13j}	3.12(3)
H(13)	C(18) ^{13j}	2.95(2)	H(13)	C(20) ^{8j}	3.34(3)
H(13)	H(6) ^{13j}	2.97(4)	H(13)	H(7) ^{13j}	2.99(4)
H(13)	H(8) ^{13j}	2.74(3)	H(13)	H(9) ^{13j}	2.75(3)

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

atom	atom	distance	atom	atom	distance
H(13)	H(10) ⁽⁸⁾	3.44(4)	H(13)	H(11) ⁽⁸⁾	2.85(4)
H(13)	H(12) ⁽⁸⁾	3.36(5)	H(14)	S(1) ⁽⁴⁾	3.33(3)
H(14)	O(2) ⁽⁴⁾	2.61(3)	H(14)	C(7) ⁽⁴⁾	2.94(3)
H(14)	C(8) ⁽⁴⁾	3.28(3)	H(14)	C(12) ⁽⁴⁾	3.50(3)
H(14)	C(13) ⁽⁴⁾	2.87(3)	H(14)	C(14) ⁽⁴⁾	3.42(3)
H(14)	H(6) ⁽¹³⁾	3.12(3)	H(14)	H(7) ⁽¹³⁾	3.58(3)
H(14)	H(14) ⁽⁴⁾	3.48(4)	H(15)	O(2) ⁽⁴⁾	3.51(3)
H(15)	N(5) ⁽⁷⁾	2.79(3)	H(15)	N(6) ⁽⁷⁾	2.86(3)
H(15)	C(7) ⁽⁷⁾	3.43(2)	H(15)	C(8) ⁽⁷⁾	2.95(3)
H(15)	C(13) ⁽⁷⁾	3.11(3)	H(15)	C(16) ⁽¹³⁾	3.40(2)
H(15)	H(6) ⁽¹³⁾	3.27(4)	H(15)	H(7) ⁽¹³⁾	2.88(3)
H(15)	H(8) ⁽¹³⁾	3.56(3)	H(15)	H(12) ⁽⁷⁾	3.48(4)

Symmetry Operators:

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

Tabel 7. Intramolecular and Intermolecular Hydrogen bonds

D	H	A	D...A	D-H	H...A	D-H...A
N(4)	H(1)	O(3)	3.045(2)	0.84(2)	2.545(17)	119.3(14)
N(4)	H(1)	O(3)[2:2:1:2]	3.130(3)	0.84(2)	2.32(2)	161.2(14)

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.