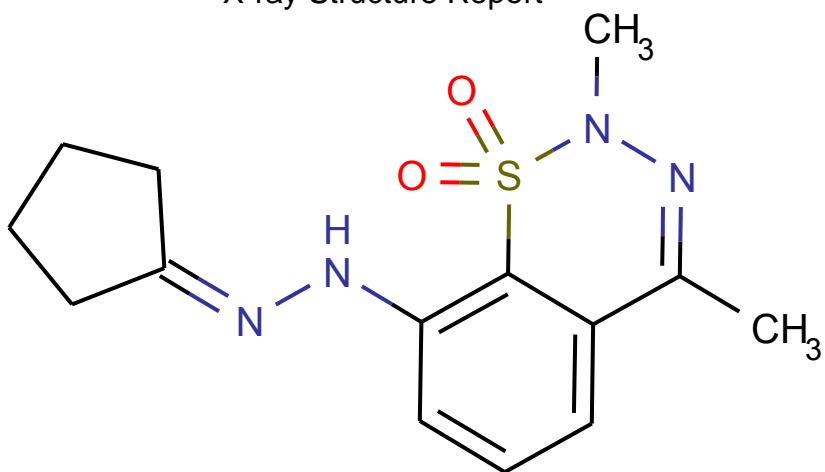


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Submitted by: Pusztai Gyongyver
Operator: Dancso Andras

X-ray Structure Report



December 6, 2024

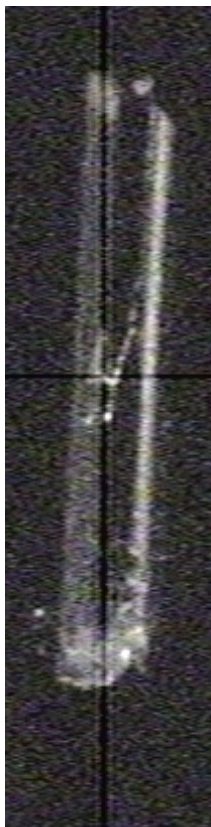


Fig 1. The crystal

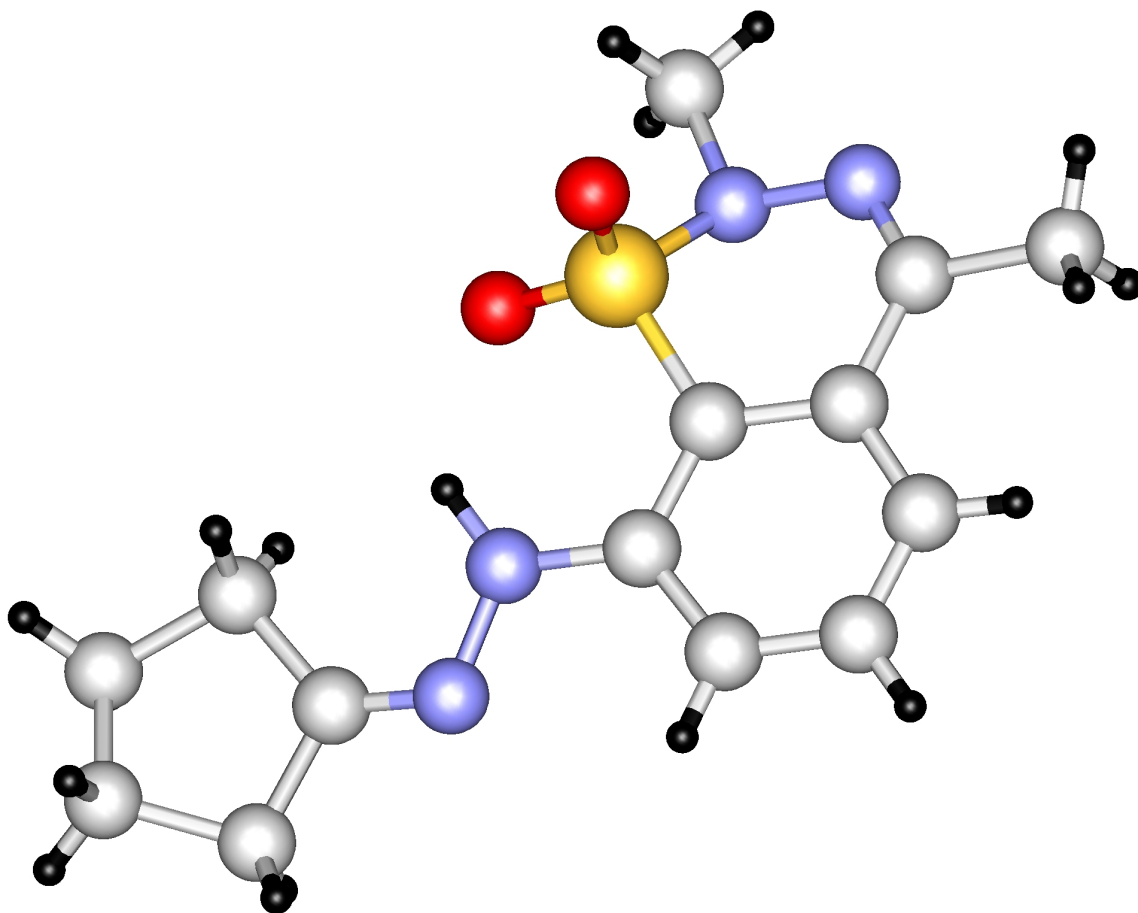


Fig. 2. The molecule (hydrogens were generated by the software)

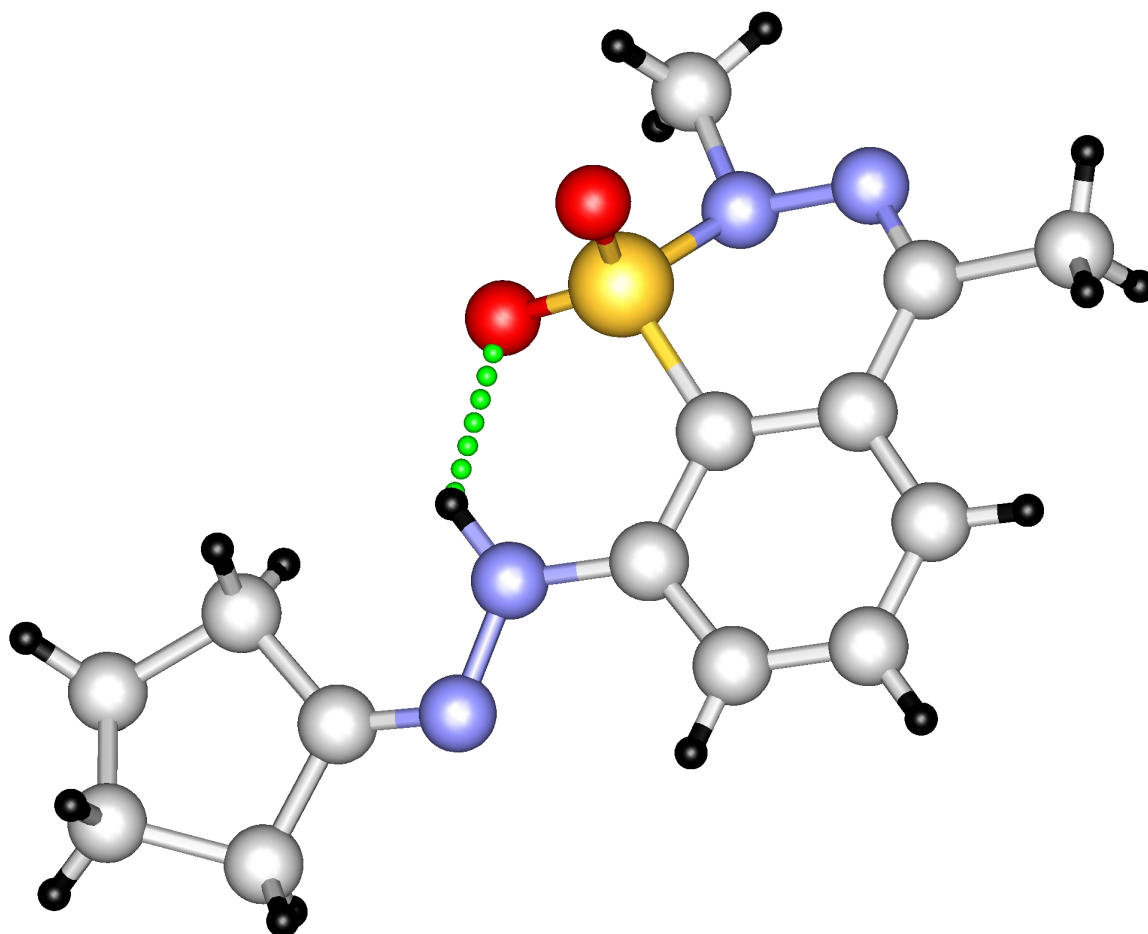


Fig. 3. Hydrogen bond

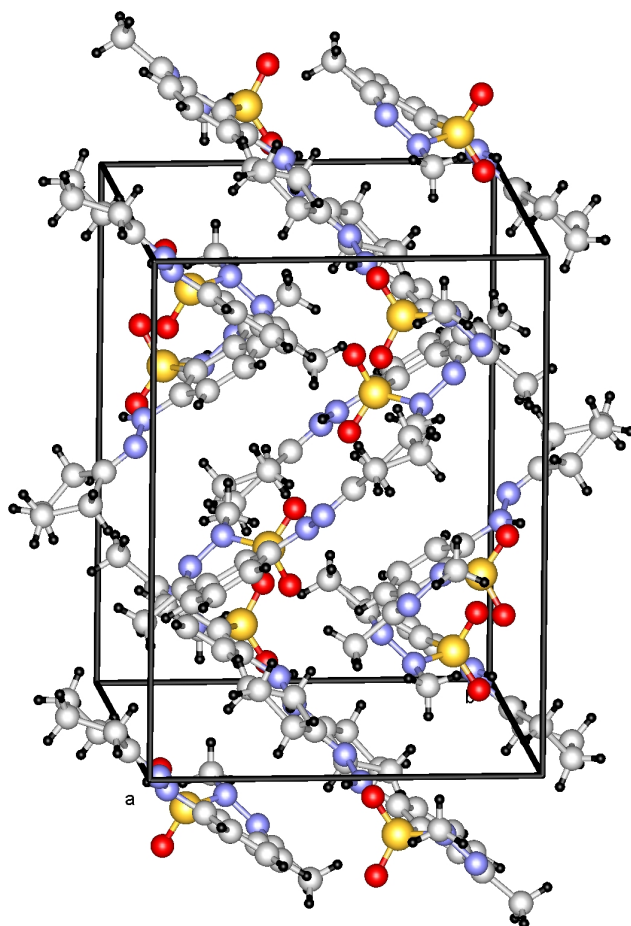


Fig. 4. Packing

Experimental

Data Collection

A colorless prism crystal of $C_{14}H_{18}N_4O_2S$ having approximate dimensions of 0.86 x 0.14 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 α radiation.

Indexing was performed from 4 oscillations that were exposed for 600 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:

$$\begin{aligned}a &= 15.4671(4) \text{ \AA} \\b &= 12.1069(4) \text{ \AA} \\c &= 16.1873(5) \text{ \AA} \\V &= 3031.21(16) \text{ \AA}^3\end{aligned}$$

For $Z = 8$ and F.W. = 306.38, the calculated density is 1.343 g/cm³. The systematic absences of:

$$\begin{aligned}0kl: k \pm 2n \\h0l: l \pm 2n \\hk0: h \pm 2n\end{aligned}$$

uniquely determine the space group to be:

Pbca (#61)

The data were collected at a temperature of $20 \pm 1^\circ\text{C}$ to a maximum 2θ value of 149.6° . 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 120.0 [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 120.0 [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 120.0 [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 120.0 [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 120.0 [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 32069 reflections that were collected, 2856 were unique ($R_{\text{int}} = 0.052$).

The linear absorption coefficient, μ , for Cu-K α radiation is 19.902 cm⁻¹. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.408 to 0.891. 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 using the riding model. The final cycle of full-matrix least-squares refinement³ on F was based on 22924 observed reflections ($I > 2.00\sigma(I)$) and 208 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.0793$$

$$R_w = [\Sigma w (|F_o| - |F_c|)^2 / \Sigma w F_o^2]^{1/2} = 0.0927$$

The standard deviation of an observation of unit weight⁴ was 6.13. 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 9.25 and -10.10 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).

(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	$C_{14}H_{18}N_4O_2S$
Formula Weight	306.38
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.86 X 0.14 X 0.06 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	4 oscillations @ 600.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 15.4671(4) \text{ \AA}$ $b = 12.1069(4) \text{ \AA}$ $c = 16.1873(5) \text{ \AA}$ $V = 3031.21(16) \text{ \AA}^3$
Space Group	Pbca (#61)
Z value	8
D_{calc}	1.343 g/cm^3
F_{000}	1296.00
$\mu(\text{CuK}\alpha)$	19.902 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	120.0 sec./°
ω oscillation Range (χ =54.0, ϕ =0.0)	20.0 - 200.0°
Exposure Rate	120.0 sec./°
ω oscillation Range (χ =54.0, ϕ =90.0)	20.0 - 200.0°
Exposure Rate	120.0 sec./°
ω oscillation Range (χ =54.0, ϕ =180.0)	20.0 - 200.0°
Exposure Rate	120.0 sec./°
ω oscillation Range (χ =54.0, ϕ =270.0)	20.0 - 200.0°
Exposure Rate	120.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\text{max}}$	149.6°
No. of Reflections Measured	Total: 32069 Unique: 2856 (R_{int} = 0.052)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.408 - 0.891)

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	149.6 $^{\circ}$
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I > 2.00\sigma(I)$)	22924
No. Variables	208
Reflection/Parameter Ratio	110.21
Residuals: R ($I > 2.00\sigma(I)$)	0.0793
Residuals: Rw ($I > 2.00\sigma(I)$)	0.0927
Goodness of Fit Indicator	6.133
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	9.25 e $^{-}/\text{\AA}^3$
Minimum peak in Final Diff. Map	-10.10 e $^{-}/\text{\AA}^3$

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

atom	x	y	z	B _{eq}
S(1)	0.10168(4)	0.14743(6)	0.62915(4)	2.952(14)
O(2)	0.08464(9)	0.09073(12)	0.70542(10)	3.60(4)
O(3)	0.09632(9)	0.08187(13)	0.55519(10)	4.29(4)
N(4)	0.03741(11)	0.25638(18)	0.61722(12)	3.42(5)
N(5)	0.04097(12)	0.33882(18)	0.67591(12)	3.44(5)
N(6)	0.35237(12)	0.04431(17)	0.52134(12)	3.26(5)
N(7)	0.27723(12)	0.07491(16)	0.56008(12)	3.23(5)
C(8)	0.19977(12)	0.21717(19)	0.63764(12)	2.26(5)
C(9)	0.27756(13)	0.1708(2)	0.60655(13)	2.71(6)
C(10)	0.19892(14)	0.3161(2)	0.68220(12)	2.50(6)
C(11)	0.11559(17)	0.3659(2)	0.70485(13)	3.18(6)
C(12)	0.35371(13)	0.2265(2)	0.62804(16)	3.48(6)
C(13)	0.27746(14)	0.3656(2)	0.69919(13)	3.25(6)
C(14)	0.38950(17)	-0.1958(2)	0.39723(14)	4.42(7)
C(15)	0.26373(14)	-0.1001(2)	0.45085(14)	3.64(7)
C(16)	0.34370(14)	-0.0338(2)	0.47211(14)	3.13(6)
C(17)	0.35345(16)	0.3202(2)	0.67300(16)	3.71(7)
C(18)	0.41962(16)	-0.0778(2)	0.42218(17)	4.77(8)
C(19)	0.29333(16)	-0.1835(2)	0.38341(17)	5.08(8)
C(20)	0.11544(16)	0.4641(2)	0.76745(17)	5.22(8)
C(21)	-0.05214(14)	0.2320(2)	0.59202(17)	5.57(9)
H(1)	0.2264	0.0312	0.5555	3.89
H(2)	0.4065	0.3550	0.6871	4.43
H(3)	0.4075	0.1964	0.6109	4.19
H(4)	0.2783	0.4323	0.7301	3.87
H(5)	0.4178	-0.2199	0.3484	5.32
H(6)	0.4009	-0.2465	0.4407	5.35
H(7)	0.2445	-0.1402	0.4978	4.39
H(8)	0.2186	-0.0533	0.4318	4.37
H(9)	0.4719	-0.0785	0.4530	5.75
H(10)	0.4270	-0.0336	0.3742	5.75
H(11)	0.2825	-0.1522	0.3306	6.15
H(12)	0.2642	-0.2523	0.3878	6.12
H(13)	0.1223	0.5317	0.7383	6.35
H(14)	0.1619	0.4552	0.8053	6.32
H(15)	0.0623	0.4651	0.7968	6.32
H(16)	-0.0554	0.2308	0.5334	6.77

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

atom	x	y	z	B _{eq}
H(17)	-0.0901	0.2869	0.6129	6.80
H(18)	-0.0684	0.1618	0.6132	6.76

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

Table 2. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	0.0290(3)	0.0370(4)	0.0461(3)	-0.0015(3)	0.0012(3)	-0.0010(3)
O(2)	0.0485(10)	0.0366(12)	0.0516(11)	-0.0072(8)	0.0063(8)	0.0145(8)
O(3)	0.0340(10)	0.0619(14)	0.0671(12)	-0.0045(9)	-0.0049(9)	-0.0208(10)
N(4)	0.0275(11)	0.0455(16)	0.0568(15)	0.0024(10)	0.0010(11)	0.0036(11)
N(5)	0.0391(13)	0.0356(16)	0.0559(15)	0.0035(11)	0.0096(10)	-0.0030(12)
N(6)	0.0282(11)	0.0398(16)	0.0559(14)	0.0006(10)	0.0078(10)	-0.0160(11)
N(7)	0.0302(11)	0.0353(15)	0.0572(14)	-0.0080(10)	0.0068(10)	-0.0157(11)
C(8)	0.0238(12)	0.0297(16)	0.0325(13)	-0.0005(11)	-0.0002(10)	0.0050(11)
C(9)	0.0308(14)	0.0281(18)	0.0440(15)	-0.0017(12)	0.0015(11)	0.0064(11)
C(10)	0.0357(14)	0.0249(16)	0.0342(14)	0.0014(12)	0.0036(11)	0.0011(11)
C(11)	0.0498(17)	0.0307(18)	0.0403(15)	0.0030(14)	0.0133(12)	0.0067(13)
C(12)	0.0224(13)	0.044(2)	0.0660(18)	0.0010(12)	0.0034(13)	-0.0062(16)
C(13)	0.0471(16)	0.0288(17)	0.0474(15)	-0.0048(13)	0.0024(12)	-0.0107(13)
C(14)	0.0617(19)	0.060(2)	0.0460(17)	0.0156(16)	0.0063(14)	-0.0145(14)
C(15)	0.0422(16)	0.051(2)	0.0457(16)	-0.0015(14)	-0.0002(12)	-0.0127(14)
C(16)	0.0278(14)	0.043(2)	0.0486(17)	0.0043(13)	0.0057(12)	0.0011(14)
C(17)	0.0324(15)	0.045(2)	0.0637(18)	-0.0044(13)	-0.0047(13)	-0.0143(15)
C(18)	0.0440(17)	0.072(2)	0.065(2)	0.0035(16)	0.0051(14)	-0.0326(17)
C(19)	0.0501(18)	0.070(2)	0.074(2)	-0.0084(16)	0.0013(16)	-0.0271(18)
C(20)	0.0548(19)	0.044(2)	0.099(2)	0.0003(15)	0.0218(17)	-0.0152(17)
C(21)	0.0306(16)	0.085(2)	0.097(2)	0.0010(16)	-0.0127(15)	0.0008(19)

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)	1.4370(17)	S(1)	O(3)	1.4388(17)
S(1)	N(4)	1.663(2)	S(1)	C(8)	1.742(2)
N(4)	N(5)	1.379(3)	N(4)	C(21)	1.474(2)
N(5)	C(11)	1.288(3)	N(6)	N(7)	1.372(2)
N(6)	C(16)	1.244(3)	N(7)	C(9)	1.383(3)
N(7)	H(1)	0.950	C(8)	C(9)	1.420(3)
C(8)	C(10)	1.398(3)	C(9)	C(12)	1.401(3)
C(10)	C(11)	1.469(3)	C(10)	C(13)	1.382(3)
C(11)	C(20)	1.563(3)	C(12)	C(17)	1.348(3)
C(12)	H(3)	0.950	C(13)	C(17)	1.365(3)
C(13)	H(4)	0.950	C(14)	C(18)	1.556(3)
C(14)	C(19)	1.512(3)	C(14)	H(5)	0.950
C(14)	H(6)	0.950	C(15)	C(16)	1.514(3)
C(15)	C(19)	1.556(3)	C(15)	H(7)	0.950
C(15)	H(8)	0.950	C(16)	C(18)	1.522(3)
C(17)	H(2)	0.950	C(18)	H(9)	0.950
C(18)	H(10)	0.950	C(19)	H(11)	0.950
C(19)	H(12)	0.950	C(20)	H(13)	0.950
C(20)	H(14)	0.950	C(20)	H(15)	0.950
C(21)	H(16)	0.950	C(21)	H(17)	0.950
C(21)	H(18)	0.950			

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

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	S(1)	O(3)	116.15(10)	O(2)	S(1)	N(4)	111.66(10)
O(2)	S(1)	C(8)	108.88(9)	O(3)	S(1)	N(4)	107.85(10)
O(3)	S(1)	C(8)	112.54(9)	N(4)	S(1)	C(8)	98.36(10)
S(1)	N(4)	N(5)	118.05(15)	S(1)	N(4)	C(21)	115.78(17)
N(5)	N(4)	C(21)	111.92(18)	N(4)	N(5)	C(11)	118.1(2)
N(7)	N(6)	C(16)	114.01(19)	N(6)	N(7)	C(9)	118.18(18)
N(6)	N(7)	H(1)	121.0	C(9)	N(7)	H(1)	120.9
S(1)	C(8)	C(9)	121.22(17)	S(1)	C(8)	C(10)	116.60(16)
C(9)	C(8)	C(10)	121.98(19)	N(7)	C(9)	C(8)	121.44(19)
N(7)	C(9)	C(12)	122.8(2)	C(8)	C(9)	C(12)	115.7(2)
C(8)	C(10)	C(11)	119.2(2)	C(8)	C(10)	C(13)	117.8(2)
C(11)	C(10)	C(13)	122.9(2)	N(5)	C(11)	C(10)	126.2(2)
N(5)	C(11)	C(20)	115.3(2)	C(10)	C(11)	C(20)	118.4(2)
C(9)	C(12)	C(17)	122.5(2)	C(9)	C(12)	H(3)	118.6
C(17)	C(12)	H(3)	118.9	C(10)	C(13)	C(17)	121.4(2)
C(10)	C(13)	H(4)	119.0	C(17)	C(13)	H(4)	119.6
C(18)	C(14)	C(19)	104.0(2)	C(18)	C(14)	H(5)	111.1
C(18)	C(14)	H(6)	110.2	C(19)	C(14)	H(5)	111.2
C(19)	C(14)	H(6)	110.8	H(5)	C(14)	H(6)	109.5
C(16)	C(15)	C(19)	105.24(19)	C(16)	C(15)	H(7)	110.2
C(16)	C(15)	H(8)	111.0	C(19)	C(15)	H(7)	108.8
C(19)	C(15)	H(8)	112.0	H(7)	C(15)	H(8)	109.5
N(6)	C(16)	C(15)	129.5(2)	N(6)	C(16)	C(18)	121.5(2)
C(15)	C(16)	C(18)	108.9(2)	C(12)	C(17)	C(13)	120.6(2)
C(12)	C(17)	H(2)	120.0	C(13)	C(17)	H(2)	119.4
C(14)	C(18)	C(16)	103.2(2)	C(14)	C(18)	H(9)	112.5
C(14)	C(18)	H(10)	109.9	C(16)	C(18)	H(9)	112.4
C(16)	C(18)	H(10)	109.2	H(9)	C(18)	H(10)	109.5
C(14)	C(19)	C(15)	104.5(2)	C(14)	C(19)	H(11)	110.2
C(14)	C(19)	H(12)	111.6	C(15)	C(19)	H(11)	108.7
C(15)	C(19)	H(12)	112.2	H(11)	C(19)	H(12)	109.5
C(11)	C(20)	H(13)	109.5	C(11)	C(20)	H(14)	109.3
C(11)	C(20)	H(15)	109.6	H(13)	C(20)	H(14)	109.5
H(13)	C(20)	H(15)	109.5	H(14)	C(20)	H(15)	109.5
N(4)	C(21)	H(16)	109.2	N(4)	C(21)	H(17)	110.0
N(4)	C(21)	H(18)	109.2	H(16)	C(21)	H(17)	109.5
H(16)	C(21)	H(18)	109.5	H(17)	C(21)	H(18)	109.5

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

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

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O(2)	S(1)	N(4)	N(5)	59.82(18)	O(2)	S(1)	N(4)	C(21)	-76.82(19)
O(2)	S(1)	C(8)	C(9)	97.33(19)	O(2)	S(1)	C(8)	C(10)	-77.67(19)
O(3)	S(1)	N(4)	N(5)	-171.42(15)	O(3)	S(1)	N(4)	C(21)	51.95(19)
O(3)	S(1)	C(8)	C(9)	-32.9(2)	O(3)	S(1)	C(8)	C(10)	152.07(16)
N(4)	S(1)	C(8)	C(9)	-146.28(18)	N(4)	S(1)	C(8)	C(10)	38.72(19)
C(8)	S(1)	N(4)	N(5)	-54.40(18)	C(8)	S(1)	N(4)	C(21)	168.96(17)
S(1)	N(4)	N(5)	C(11)	39.9(2)	C(21)	N(4)	N(5)	C(11)	178.1(2)
N(4)	N(5)	C(11)	C(10)	-0.9(3)	N(4)	N(5)	C(11)	C(20)	176.8(2)
N(7)	N(6)	C(16)	C(15)	-1.5(3)	N(7)	N(6)	C(16)	C(18)	178.8(2)
C(16)	N(6)	N(7)	C(9)	-170.7(2)	N(6)	N(7)	C(9)	C(8)	170.0(2)
N(6)	N(7)	C(9)	C(12)	-11.9(3)	S(1)	C(8)	C(9)	N(7)	7.6(3)
S(1)	C(8)	C(9)	C(12)	-170.69(18)	S(1)	C(8)	C(10)	C(11)	-11.6(2)
S(1)	C(8)	C(10)	C(13)	171.67(17)	C(9)	C(8)	C(10)	C(11)	173.4(2)
C(9)	C(8)	C(10)	C(13)	-3.3(3)	C(10)	C(8)	C(9)	N(7)	-177.7(2)
C(10)	C(8)	C(9)	C(12)	4.0(3)	N(7)	C(9)	C(12)	C(17)	179.7(2)
C(8)	C(9)	C(12)	C(17)	-2.1(3)	C(8)	C(10)	C(11)	N(5)	-13.1(3)
C(8)	C(10)	C(11)	C(20)	169.3(2)	C(8)	C(10)	C(13)	C(17)	0.5(3)
C(11)	C(10)	C(13)	C(17)	-176.1(2)	C(13)	C(10)	C(11)	N(5)	163.4(2)
C(13)	C(10)	C(11)	C(20)	-14.1(3)	C(9)	C(12)	C(17)	C(13)	-0.5(4)
C(10)	C(13)	C(17)	C(12)	1.4(3)	C(18)	C(14)	C(19)	C(15)	-37.0(2)
C(19)	C(14)	C(18)	C(16)	35.3(2)	C(16)	C(15)	C(19)	C(14)	24.4(2)
C(19)	C(15)	C(16)	N(6)	178.2(2)	C(19)	C(15)	C(16)	C(18)	-2.0(2)
N(6)	C(16)	C(18)	C(14)	159.5(2)	C(15)	C(16)	C(18)	C(14)	-20.3(2)

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(4) ¹⁾	3.592	S(1)	H(6) ²⁾	3.310
S(1)	H(15) ³⁾	3.569	O(2)	C(13) ¹⁾	3.463(2)
O(2)	C(14) ⁴⁾	3.379(2)	O(2)	C(17) ¹⁾	3.452(3)
O(2)	C(18) ⁴⁾	3.513(3)	O(2)	C(20) ³⁾	3.481(2)
O(2)	H(2) ¹⁾	2.873	O(2)	H(4) ¹⁾	2.887
O(2)	H(5) ⁴⁾	2.793	O(2)	H(10) ⁴⁾	2.824
O(2)	H(11) ⁴⁾	2.980	O(2)	H(13) ³⁾	3.403
O(2)	H(15) ³⁾	2.734	O(3)	H(2) ¹⁾	3.480
O(3)	H(6) ²⁾	2.785	N(4)	H(6) ²⁾	3.013
N(4)	H(9) ²⁾	3.329	N(5)	H(2) ⁵⁾	3.046
N(5)	H(5) ⁴⁾	3.205	N(5)	H(10) ⁶⁾	3.054
N(6)	C(21) ⁷⁾	3.589(3)	N(6)	H(9) ⁸⁾	2.781
N(6)	H(13) ¹⁾	3.538	N(6)	H(16) ⁷⁾	3.199
N(6)	H(17) ⁷⁾	3.113	N(7)	C(13) ¹⁾	3.494(3)
N(7)	H(4) ¹⁾	3.360	N(7)	H(12) ²⁾	3.545
N(7)	H(13) ¹⁾	3.319	C(8)	H(6) ²⁾	3.575
C(8)	H(7) ²⁾	2.975	C(8)	H(11) ⁴⁾	3.232
C(9)	H(7) ²⁾	2.907	C(9)	H(13) ¹⁾	3.128
C(10)	H(1) ²⁾	3.511	C(10)	H(5) ⁴⁾	3.443
C(10)	H(7) ²⁾	3.156	C(10)	H(11) ⁴⁾	3.129
C(10)	H(12) ⁴⁾	3.463	C(11)	H(5) ⁴⁾	2.965
C(12)	H(5) ⁸⁾	3.555	C(12)	H(7) ²⁾	3.058
C(12)	H(9) ⁸⁾	3.494	C(12)	H(13) ¹⁾	2.982
C(12)	H(16) ⁷⁾	3.012	C(13)	O(2) ²⁾	3.463(2)
C(13)	N(7) ²⁾	3.494(3)	C(13)	H(1) ²⁾	3.072
C(13)	H(7) ²⁾	3.278	C(13)	H(11) ⁴⁾	3.473
C(13)	H(12) ⁴⁾	3.408	C(14)	O(2) ⁹⁾	3.379(2)
C(14)	H(3) ⁸⁾	3.143	C(14)	H(14) ⁹⁾	3.565
C(14)	H(16) ¹⁾	3.498	C(15)	H(14) ¹⁰⁾	3.333
C(15)	H(16) ¹¹⁾	3.599	C(15)	H(18) ¹¹⁾	3.280
C(16)	H(9) ⁸⁾	3.384	C(16)	H(17) ⁷⁾	3.446
C(17)	O(2) ²⁾	3.452(3)	C(17)	H(1) ²⁾	3.416
C(17)	H(7) ²⁾	3.250	C(17)	H(17) ¹²⁾	3.596
C(18)	O(2) ⁹⁾	3.513(3)	C(18)	H(3) ⁸⁾	3.082
C(18)	H(9) ⁸⁾	3.237	C(18)	H(17) ⁷⁾	3.570
C(19)	H(14) ⁹⁾	3.592	C(19)	H(17) ¹¹⁾	3.384
C(19)	H(18) ¹¹⁾	3.489	C(20)	O(2) ¹³⁾	3.481(2)

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

atom	atom	distance	atom	atom	distance
C(20)	H(2) ⁵⁾	3.568	C(20)	H(5) ⁴⁾	3.275
C(20)	H(8) ¹⁴⁾	3.285	C(20)	H(11) ¹⁴⁾	3.593
C(20)	H(18) ¹³⁾	3.161	C(21)	N(6) ⁶⁾	3.589(3)
C(21)	H(3) ⁶⁾	3.454	C(21)	H(6) ²⁾	3.397
C(21)	H(7) ¹¹⁾	3.492	C(21)	H(8) ¹¹⁾	3.384
C(21)	H(9) ²⁾	3.445	C(21)	H(12) ¹¹⁾	3.306
H(1)	C(10) ¹⁾	3.511	H(1)	C(13) ¹⁾	3.072
H(1)	C(17) ¹⁾	3.416	H(1)	H(4) ¹⁾	3.071
H(2)	O(2) ²⁾	2.873	H(2)	O(3) ²⁾	3.480
H(2)	N(5) ¹²⁾	3.046	H(2)	C(20) ¹²⁾	3.568
H(2)	H(5) ⁸⁾	3.223	H(2)	H(15) ¹²⁾	2.766
H(2)	H(17) ¹²⁾	3.340	H(3)	C(14) ⁸⁾	3.143
H(3)	C(18) ⁸⁾	3.082	H(3)	C(21) ⁷⁾	3.454
H(3)	H(5) ⁸⁾	2.796	H(3)	H(6) ⁸⁾	3.139
H(3)	H(7) ²⁾	3.576	H(3)	H(9) ⁸⁾	2.566
H(3)	H(10) ⁸⁾	3.240	H(3)	H(13) ¹⁾	2.906
H(3)	H(16) ⁷⁾	2.562	H(4)	S(1) ²⁾	3.592
H(4)	O(2) ²⁾	2.887	H(4)	N(7) ²⁾	3.360
H(4)	H(1) ²⁾	3.071	H(4)	H(10) ¹⁴⁾	3.498
H(4)	H(11) ¹⁴⁾	3.121	H(4)	H(12) ⁴⁾	3.420
H(5)	O(2) ⁹⁾	2.793	H(5)	N(5) ⁹⁾	3.205
H(5)	C(10) ⁹⁾	3.443	H(5)	C(11) ⁹⁾	2.965
H(5)	C(12) ⁸⁾	3.555	H(5)	C(20) ⁹⁾	3.275
H(5)	H(2) ⁸⁾	3.223	H(5)	H(3) ⁸⁾	2.796
H(5)	H(14) ⁹⁾	3.182	H(5)	H(15) ⁹⁾	3.100
H(6)	S(1) ¹⁾	3.310	H(6)	O(3) ¹⁾	2.785
H(6)	N(4) ¹⁾	3.013	H(6)	C(8) ¹⁾	3.575
H(6)	C(21) ¹⁾	3.397	H(6)	H(3) ⁸⁾	3.139
H(6)	H(14) ⁹⁾	3.483	H(6)	H(15) ⁹⁾	3.572
H(6)	H(16) ¹⁾	2.836	H(7)	C(8) ¹⁾	2.975
H(7)	C(9) ¹⁾	2.907	H(7)	C(10) ¹⁾	3.156
H(7)	C(12) ¹⁾	3.058	H(7)	C(13) ¹⁾	3.278
H(7)	C(17) ¹⁾	3.250	H(7)	C(21) ¹¹⁾	3.492
H(7)	H(3) ¹⁾	3.576	H(7)	H(16) ¹¹⁾	3.164
H(7)	H(17) ¹¹⁾	3.474	H(7)	H(18) ¹¹⁾	3.273
H(8)	C(20) ¹⁰⁾	3.285	H(8)	C(21) ¹¹⁾	3.384
H(8)	H(13) ¹⁰⁾	3.478	H(8)	H(14) ¹⁰⁾	2.525

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

atom	atom	distance	atom	atom	distance
H(8)	H(15) ^{10j}	3.430	H(8)	H(16) ^{11j}	3.362
H(8)	H(17) ^{11j}	3.531	H(8)	H(18) ^{11j}	2.766
H(9)	N(4) ^{1j}	3.329	H(9)	N(6) ^{8j}	2.781
H(9)	C(12) ^{8j}	3.494	H(9)	C(16) ^{8j}	3.384
H(9)	C(18) ^{8j}	3.237	H(9)	C(21) ^{1j}	3.445
H(9)	H(3) ^{8j}	2.566	H(9)	H(9) ^{8j}	2.586
H(9)	H(10) ^{8j}	3.481	H(9)	H(16) ^{1j}	2.948
H(9)	H(17) ^{1j}	3.564	H(10)	O(2) ^{9j}	2.824
H(10)	N(5) ^{7j}	3.054	H(10)	H(3) ^{8j}	3.240
H(10)	H(4) ^{10j}	3.498	H(10)	H(9) ^{8j}	3.481
H(10)	H(13) ^{7j}	3.528	H(10)	H(15) ^{7j}	3.568
H(10)	H(17) ^{7j}	3.005	H(11)	O(2) ^{9j}	2.980
H(11)	C(8) ^{9j}	3.232	H(11)	C(10) ^{9j}	3.129
H(11)	C(13) ^{9j}	3.473	H(11)	C(20) ^{10j}	3.593
H(11)	H(4) ^{10j}	3.121	H(11)	H(13) ^{10j}	3.241
H(11)	H(14) ^{10j}	3.056	H(11)	H(17) ^{11j}	3.515
H(11)	H(18) ^{11j}	3.436	H(12)	N(7) ^{1j}	3.545
H(12)	C(10) ^{9j}	3.463	H(12)	C(13) ^{9j}	3.408
H(12)	C(21) ^{11j}	3.306	H(12)	H(4) ^{9j}	3.420
H(12)	H(14) ^{9j}	3.020	H(12)	H(16) ^{11j}	3.483
H(12)	H(17) ^{11j}	2.725	H(12)	H(18) ^{11j}	3.221
H(13)	O(2) ^{13j}	3.403	H(13)	N(6) ^{2j}	3.538
H(13)	N(7) ^{2j}	3.319	H(13)	C(9) ^{2j}	3.128
H(13)	C(12) ^{2j}	2.982	H(13)	H(3) ^{2j}	2.906
H(13)	H(8) ^{14j}	3.478	H(13)	H(10) ^{6j}	3.528
H(13)	H(11) ^{14j}	3.241	H(13)	H(18) ^{13j}	2.992
H(14)	C(14) ^{4j}	3.565	H(14)	C(15) ^{14j}	3.333
H(14)	C(19) ^{4j}	3.592	H(14)	H(5) ^{4j}	3.182
H(14)	H(6) ^{4j}	3.483	H(14)	H(8) ^{14j}	2.525
H(14)	H(11) ^{14j}	3.056	H(14)	H(12) ^{4j}	3.020
H(14)	H(18) ^{13j}	3.176	H(15)	S(1) ^{13j}	3.569
H(15)	O(2) ^{13j}	2.734	H(15)	H(2) ^{5j}	2.766
H(15)	H(5) ^{4j}	3.100	H(15)	H(6) ^{4j}	3.572
H(15)	H(8) ^{14j}	3.430	H(15)	H(10) ^{6j}	3.568
H(15)	H(18) ^{13j}	2.793	H(16)	N(6) ^{6j}	3.199
H(16)	C(12) ^{6j}	3.012	H(16)	C(14) ^{2j}	3.498
H(16)	C(15) ^{11j}	3.599	H(16)	H(3) ^{6j}	2.562

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

atom	atom	distance	atom	atom	distance
H(16)	H(6) ²⁾	2.836	H(16)	H(7) ¹¹⁾	3.164
H(16)	H(8) ¹¹⁾	3.362	H(16)	H(9) ²⁾	2.948
H(16)	H(12) ¹¹⁾	3.483	H(17)	N(6) ⁶⁾	3.113
H(17)	C(16) ⁶⁾	3.446	H(17)	C(17) ⁵⁾	3.596
H(17)	C(18) ⁶⁾	3.570	H(17)	C(19) ¹¹⁾	3.384
H(17)	H(2) ⁵⁾	3.340	H(17)	H(7) ¹¹⁾	3.474
H(17)	H(8) ¹¹⁾	3.531	H(17)	H(9) ²⁾	3.564
H(17)	H(10) ⁶⁾	3.005	H(17)	H(11) ¹¹⁾	3.515
H(17)	H(12) ¹¹⁾	2.725	H(18)	C(15) ¹¹⁾	3.280
H(18)	C(19) ¹¹⁾	3.489	H(18)	C(20) ³⁾	3.161
H(18)	H(7) ¹¹⁾	3.273	H(18)	H(8) ¹¹⁾	2.766
H(18)	H(11) ¹¹⁾	3.436	H(18)	H(12) ¹¹⁾	3.221
H(18)	H(13) ³⁾	2.992	H(18)	H(14) ³⁾	3.176
H(18)	H(15) ³⁾	2.793			

Symmetry Operators:

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

Table 7. Intramolecular and Intermolecular Hydrogen bonds

D	H	A	D...A	D-H	H...A	D-H...A
N(7)	H(1)	O(3)	2.801(2)	0.950	2.104	129.0

- 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.