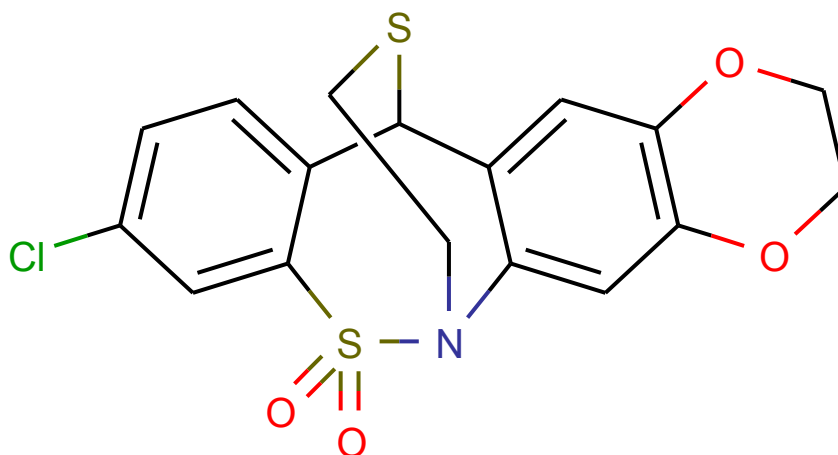


**143527**

**BEG0701\_1C**

Submitted by: Berecz Gabor  
Operator: Dancso Andras

X-ray Structure Report



September 19, 2024

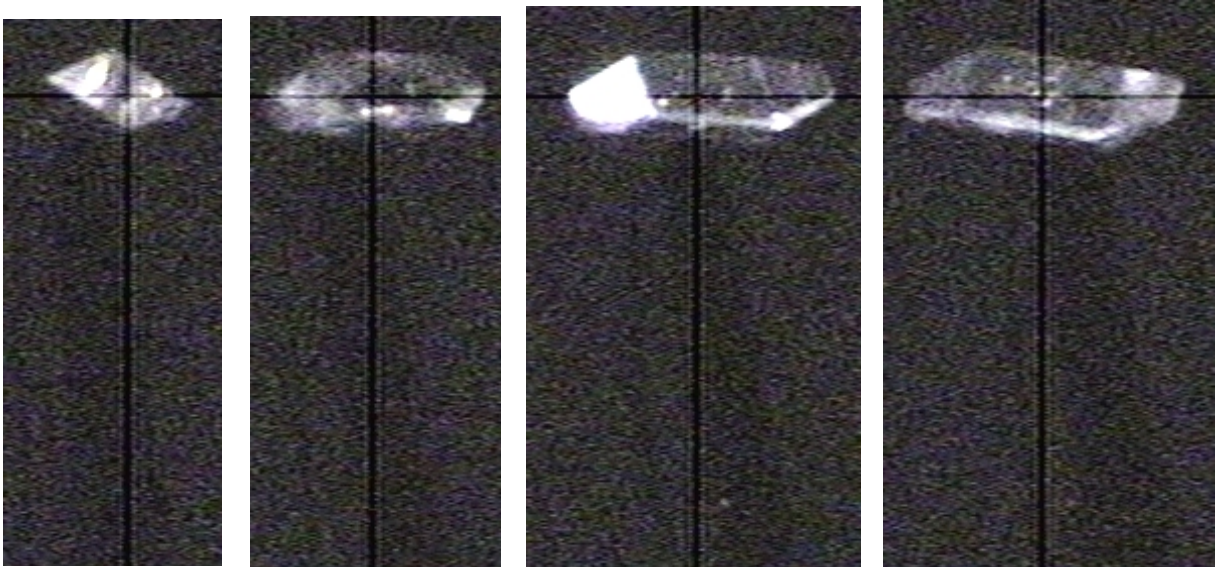


Fig. 1. The crystal

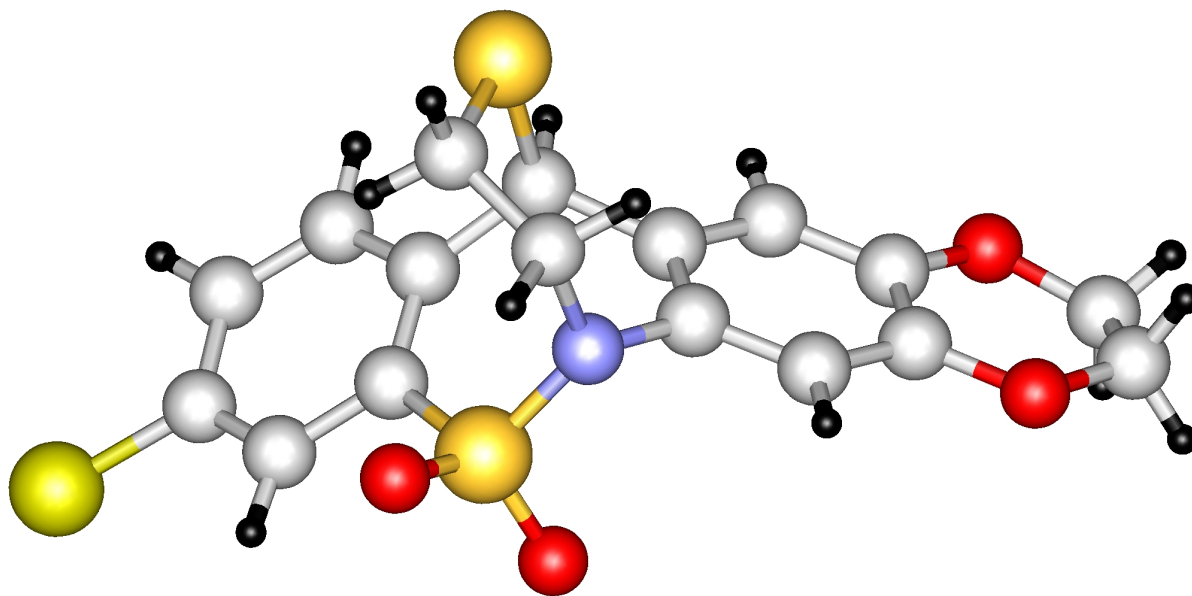


Fig. 2. The molecule

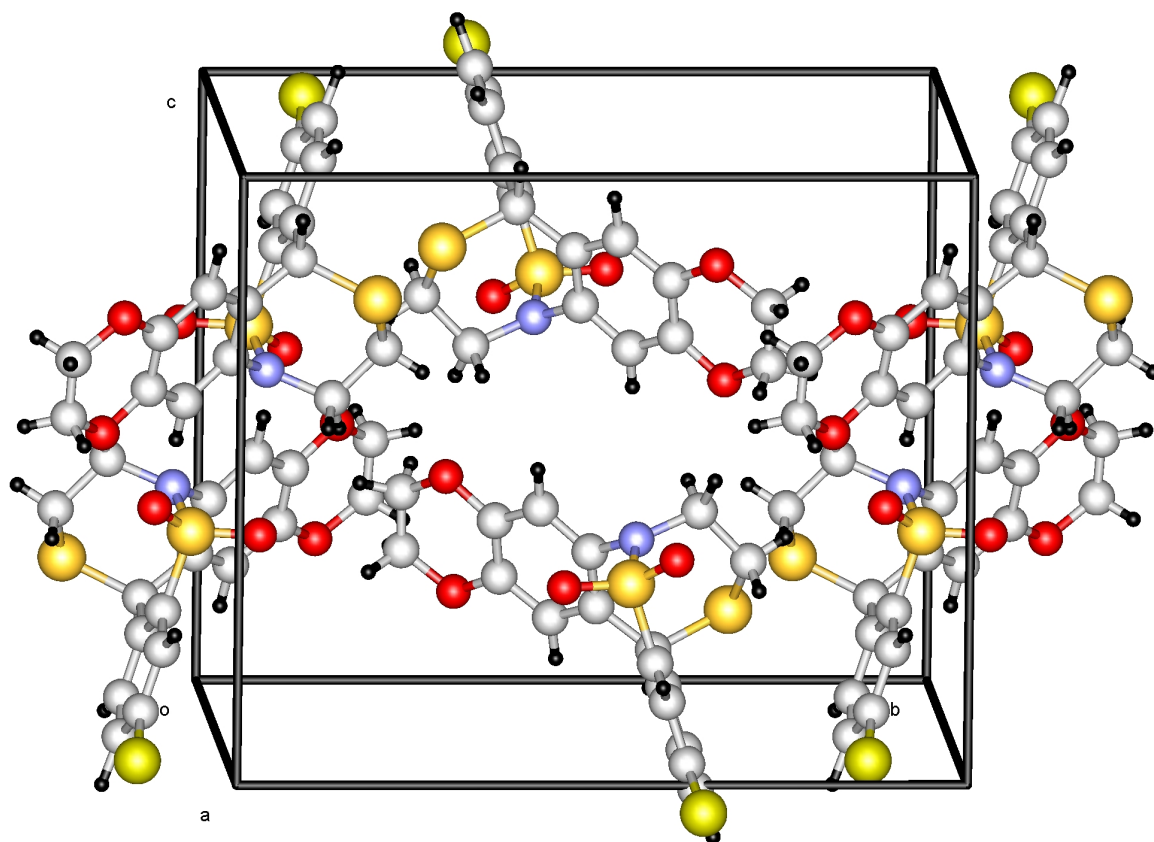


Fig. 3. Packing

## *Experimental*

### Data Collection

A colorless prism crystal of  $C_{17}H_{14}ClNO_4S_2$  having approximate dimensions of 0.41 x 0.13 x 0.10 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 180 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:

$$\begin{aligned}a &= 8.8017(4) \text{ \AA} \\b &= 15.2880(6) \text{ \AA} \quad \beta = 97.430(2)^\circ \\c &= 12.7533(5) \text{ \AA} \\V &= 1701.68(12) \text{ \AA}^3\end{aligned}$$

For  $Z = 4$  and F.W. = 395.87, the calculated density is 1.545 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  $143.4^\circ$ . 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 36.0 [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 36.0 [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 36.0 [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 36.0 [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 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 19802 reflections that were collected, 3204 were unique ( $R_{\text{int}} = 0.061$ ).

The linear absorption coefficient,  $\mu$ , for Cu-K $\alpha$  radiation is 44.912 cm<sup>-1</sup>. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.353 to 0.638. 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 11129 observed reflections ( $I > 2.00\sigma(I)$ ) and 267 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.0445$$

$$R_w = [ \Sigma w (|F_o| - |F_c|)^2 / \Sigma w F_o^2 ]^{1/2} = 0.0488$$

The standard deviation of an observation of unit weight<sup>4</sup> was 3.20. 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 4.89 and -3.40 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).

(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       | $\text{C}_{17}\text{H}_{14}\text{ClNO}_4\text{S}_2$   |
| Formula Weight          | 395.87  |
| Crystal Color, Habit    | colorless, prism  |
| Crystal Dimensions      | 0.41 X 0.13 X 0.10 mm   |
| Crystal System          | monoclinic  |
| Lattice Type            | Primitive   |
| Indexing Images         | 4 oscillations @ 180.0 seconds  |
| Detector Position       | 127.40 mm   |
| Pixel Size              | 0.100 mm  |
| Lattice Parameters      | $a = 8.8017(4) \text{ \AA}$<br>$b = 15.2880(6) \text{ \AA}$<br>$c = 12.7533(5) \text{ \AA}$<br>$\beta = 97.430(2)^\circ$<br>$V = 1701.68(12) \text{ \AA}^3$ |
| Space Group             | $P2_1/a$ (#14)  |
| Z value                 | 4   |
| D <sub>calc</sub>       | 1.545 g/cm <sup>3</sup>   |
| F <sub>000</sub>        | 816.00  |
| $\mu(\text{CuK}\alpha)$ | 44.912 cm <sup>-1</sup>   |



## B. Intensity Measurements

|   |   |
|---|---|
| Diffractometer  | Rigaku RAXIS-RAPID  |
| Radiation   | CuK $\alpha$ ( $\lambda$ = 1.54187 Å)<br>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°   |
| Exposure Rate   | 36.0 sec./°   |
| $\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =0.0)   | 20.0 - 200.0°   |
| Exposure Rate   | 36.0 sec./°   |
| $\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =90.0)  | 20.0 - 200.0°   |
| Exposure Rate   | 36.0 sec./°   |
| $\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =180.0) | 20.0 - 200.0°   |
| Exposure Rate   | 36.0 sec./°   |
| $\omega$ oscillation Range ( $\chi$ =54.0, $\phi$ =270.0) | 20.0 - 200.0°   |
| Exposure Rate   | 36.0 sec./°   |
| Detector Position   | 127.40 mm   |
| Pixel Size  | 0.100 mm  |
| $2\theta_{\text{max}}$                                    | 143.4°  |
| No. of Reflections Measured                               | Total: 19802<br>Unique: 3204 ( $R_{\text{int}}$ = 0.061)              |
| Corrections   | Lorentz-polarization<br>Absorption<br>(trans. factors: 0.353 - 0.638) |

### 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            | 143.4 $^{\circ}$               |
| Anomalous Dispersion                     | All non-hydrogen atoms         |
| No. Observations ( $I > 2.00\sigma(I)$ ) | 11129                          |
| No. Variables                            | 267                            |
| Reflection/Parameter Ratio               | 41.68                          |
| Residuals: R ( $I > 2.00\sigma(I)$ )     | 0.0445                         |
| Residuals: Rw ( $I > 2.00\sigma(I)$ )    | 0.0488                         |
| Goodness of Fit Indicator                | 3.203                          |
| Max Shift/Error in Final Cycle           | 0.000                          |
| Maximum peak in Final Diff. Map          | 4.89 e $^{-}/\text{\AA}^3$     |
| Minimum peak in Final Diff. Map          | -3.40 e $^{-}/\text{\AA}^3$    |

Table 1. Atomic coordinates and B<sub>iso</sub>/B<sub>eq</sub>

| atom  | x           | y           | z           | B <sub>eq</sub> |
|-------|-------------|-------------|-------------|-----------------|
| Cl(2) | 0.78099(9)  | 0.87645(5)  | 0.00225(6)  | 5.48(2)         |
| S(1)  | 0.48695(9)  | 0.96299(5)  | 0.32842(5)  | 3.089(18)       |
| S(3)  | 0.11651(9)  | 0.80812(5)  | 0.22201(6)  | 5.25(2)         |
| O(1)  | 0.51439(19) | 1.05549(10) | 0.32711(12) | 3.96(4)         |
| O(4)  | -0.0038(2)  | 1.18956(11) | 0.42010(12) | 3.59(5)         |
| O(5)  | -0.1447(2)  | 1.16910(12) | 0.20676(12) | 4.33(5)         |
| O(7)  | 0.59844(19) | 0.90659(11) | 0.38662(12) | 4.16(5)         |
| N(1)  | 0.3253(2)   | 0.94668(12) | 0.37358(13) | 2.76(5)         |
| C(9)  | 0.6069(3)   | 0.91824(16) | 0.1551(2)   | 3.05(8)         |
| C(10) | 0.0122(3)   | 1.04532(18) | 0.1910(2)   | 2.93(7)         |
| C(11) | 0.1559(3)   | 1.06868(17) | 0.3953(2)   | 2.62(7)         |
| C(12) | 0.2005(2)   | 1.00310(16) | 0.3319(2)   | 2.54(7)         |
| C(13) | -0.0327(2)  | 1.11335(18) | 0.2511(2)   | 2.85(7)         |
| C(14) | 0.1709(2)   | 0.91303(14) | 0.16521(19) | 3.09(7)         |
| C(15) | 0.4668(3)   | 0.92710(13) | 0.19389(18) | 2.43(7)         |
| C(16) | 0.1284(2)   | 0.98946(16) | 0.2299(2)   | 2.52(6)         |
| C(17) | 0.3377(3)   | 0.87907(18) | 0.0300(2)   | 3.48(8)         |
| C(18) | 0.6072(3)   | 0.89021(16) | 0.0532(2)   | 3.30(7)         |
| C(19) | 0.3289(3)   | 0.90778(14) | 0.1327(2)   | 2.52(7)         |
| C(20) | 0.0381(3)   | 1.12418(17) | 0.3548(2)   | 2.57(7)         |
| C(21) | 0.4727(4)   | 0.87006(18) | -0.0116(2)  | 3.83(9)         |
| C(22) | -0.1720(5)  | 1.2398(2)   | 0.2704(3)   | 15.67(18)       |
| C(23) | 0.2900(4)   | 0.8589(2)   | 0.4142(2)   | 4.50(10)        |
| C(24) | 0.2685(4)   | 0.7899(2)   | 0.3279(3)   | 5.58(11)        |
| C(25) | -0.1258(4)  | 1.2431(2)   | 0.3737(2)   | 6.20(10)        |
| H(1)  | 0.708(2)    | 0.9296(11)  | 0.1956(14)  | 2.1(5)          |
| H(2)  | 0.476(2)    | 0.8456(13)  | -0.0896(18) | 5.3(6)          |
| H(3)  | 0.244(2)    | 0.8615(13)  | -0.0082(16) | 4.0(7)          |
| H(4)  | 0.212(2)    | 1.0762(10)  | 0.4685(14)  | 1.6(4)          |
| H(5)  | -0.041(2)   | 1.0342(11)  | 0.1145(14)  | 2.2(5)          |
| H(6)  | 0.362(3)    | 0.7801(15)  | 0.2948(19)  | 5.6(8)          |
| H(7)  | 0.254(3)    | 0.7381(19)  | 0.365(2)    | 9.5(11)         |
| H(8)  | 0.387(3)    | 0.8435(16)  | 0.480(2)    | 7.8(9)          |
| H(9)  | 0.188(3)    | 0.8692(15)  | 0.4467(19)  | 6.4(8)          |
| H(10) | 0.0887      | 0.9132      | 0.0879      | 3.69            |
| H(11) | -0.1200     | 1.2873      | 0.2428      | 17.93           |
| H(12) | -0.2793     | 1.2502      | 0.2591      | 17.92           |

Table 1. Atomic coordinates and B<sub>iso</sub>/B<sub>eq</sub> (continued)

| atom  | x       | y      | z      | B <sub>eq</sub> |
|-------|---------|--------|--------|-----------------|
| H(13) | -0.2132 | 1.2261 | 0.4054 | 7.50            |
| H(14) | -0.0990 | 1.3016 | 0.3931 | 7.49            |

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

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> |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Cl(2) | 0.0510(6)       | 0.0913(6)       | 0.0709(5)       | -0.0025(4)      | 0.0260(4)       | -0.0142(4)      |
| S(1)  | 0.0374(5)       | 0.0420(4)       | 0.0360(4)       | 0.0030(4)       | -0.0031(3)      | -0.0053(3)      |
| S(3)  | 0.0592(6)       | 0.0454(5)       | 0.0977(6)       | -0.0192(4)      | 0.0215(5)       | -0.0149(4)      |
| O(1)  | 0.0505(13)      | 0.0368(11)      | 0.0627(12)      | -0.0097(9)      | 0.0054(10)      | -0.0173(9)      |
| O(4)  | 0.0417(13)      | 0.0483(12)      | 0.0454(12)      | 0.0132(10)      | 0.0013(10)      | -0.0094(10)     |
| O(5)  | 0.0476(14)      | 0.0671(14)      | 0.0456(12)      | 0.0272(11)      | -0.0102(10)     | -0.0113(10)     |
| O(7)  | 0.0425(13)      | 0.0703(13)      | 0.0420(11)      | 0.0190(11)      | -0.0074(9)      | -0.0049(9)      |
| N(1)  | 0.0350(16)      | 0.0368(14)      | 0.0330(13)      | 0.0039(11)      | 0.0034(11)      | 0.0049(10)      |
| C(9)  | 0.039(2)        | 0.0384(17)      | 0.0369(19)      | -0.0074(16)     | -0.0006(17)     | -0.0006(13)     |
| C(10) | 0.0263(19)      | 0.0517(19)      | 0.0323(18)      | 0.0012(15)      | -0.0001(14)     | -0.0087(15)     |
| C(11) | 0.031(2)        | 0.0385(17)      | 0.0291(17)      | -0.0031(14)     | -0.0004(14)     | -0.0037(13)     |
| C(12) | 0.0262(19)      | 0.0329(17)      | 0.0376(17)      | 0.0008(13)      | 0.0052(14)      | 0.0044(13)      |
| C(13) | 0.0263(19)      | 0.0446(19)      | 0.0356(17)      | 0.0042(15)      | -0.0021(14)     | 0.0016(14)      |
| C(14) | 0.034(2)        | 0.0405(18)      | 0.0426(17)      | -0.0093(14)     | 0.0025(14)      | -0.0126(13)     |
| C(15) | 0.036(2)        | 0.0283(15)      | 0.0280(16)      | 0.0017(13)      | 0.0036(14)      | -0.0016(11)     |
| C(16) | 0.0255(19)      | 0.0363(16)      | 0.0336(16)      | -0.0050(14)     | 0.0019(13)      | -0.0069(13)     |
| C(17) | 0.029(2)        | 0.056(2)        | 0.046(2)        | 0.0023(17)      | -0.0019(17)     | -0.0127(15)     |
| C(18) | 0.037(2)        | 0.0412(18)      | 0.051(2)        | 0.0069(15)      | 0.0195(16)      | 0.0088(15)      |
| C(19) | 0.035(2)        | 0.0288(16)      | 0.0317(17)      | 0.0016(13)      | 0.0027(14)      | -0.0028(12)     |
| C(20) | 0.0303(19)      | 0.0304(16)      | 0.0386(17)      | -0.0028(13)     | 0.0102(14)      | -0.0044(13)     |
| C(21) | 0.048(2)        | 0.058(2)        | 0.037(2)        | 0.0061(17)      | -0.0013(18)     | -0.0063(16)     |
| C(22) | 0.237(5)        | 0.236(5)        | 0.094(3)        | 0.213(4)        | -0.085(3)       | -0.099(3)       |
| C(23) | 0.073(3)        | 0.041(2)        | 0.061(2)        | 0.0123(19)      | 0.024(2)        | 0.0224(18)      |
| C(24) | 0.072(3)        | 0.042(2)        | 0.103(3)        | 0.002(2)        | 0.030(2)        | 0.023(2)        |
| C(25) | 0.100(3)        | 0.074(2)        | 0.060(2)        | 0.042(2)        | 0.006(2)        | 0.0045(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   |
|-------|-------|------------|-------|-------|------------|
| Cl(2) | C(18) | 1.750(3)   | S(1)  | O(1)  | 1.4351(17) |
| S(1)  | O(7)  | 1.4384(17) | S(1)  | N(1)  | 1.621(2)   |
| S(1)  | C(15) | 1.788(2)   | S(3)  | C(14) | 1.848(2)   |
| S(3)  | C(24) | 1.795(3)   | O(4)  | C(20) | 1.382(3)   |
| O(4)  | C(25) | 1.417(3)   | O(5)  | C(13) | 1.369(3)   |
| O(5)  | C(22) | 1.392(4)   | N(1)  | C(12) | 1.443(3)   |
| N(1)  | C(23) | 1.486(3)   | C(9)  | C(15) | 1.394(4)   |
| C(9)  | C(18) | 1.370(4)   | C(9)  | H(1)  | 0.987(19)  |
| C(10) | C(13) | 1.380(3)   | C(10) | C(16) | 1.375(3)   |
| C(10) | H(5)  | 1.041(17)  | C(11) | C(12) | 1.377(3)   |
| C(11) | C(20) | 1.387(3)   | C(11) | H(4)  | 1.003(17)  |
| C(12) | C(16) | 1.387(3)   | C(13) | C(20) | 1.397(3)   |
| C(14) | C(16) | 1.505(3)   | C(14) | C(19) | 1.503(3)   |
| C(14) | H(10) | 1.145      | C(15) | C(19) | 1.387(3)   |
| C(17) | C(19) | 1.393(4)   | C(17) | C(21) | 1.369(5)   |
| C(17) | H(3)  | 0.94(2)    | C(18) | C(21) | 1.388(4)   |
| C(21) | H(2)  | 1.07(2)    | C(22) | C(25) | 1.328(5)   |
| C(22) | H(11) | 0.950      | C(22) | H(12) | 0.950      |
| C(23) | C(24) | 1.519(5)   | C(23) | H(8)  | 1.14(2)    |
| C(23) | H(9)  | 1.05(2)    | C(24) | H(6)  | 0.98(2)    |
| C(24) | H(7)  | 0.94(3)    | C(25) | H(13) | 0.950      |
| C(25) | H(14) | 0.950      |       |       |            |

Table 4. Bond angles (°)

| atom  | atom  | atom  | angle      | atom  | atom  | atom  | angle      |
|-------|-------|-------|------------|-------|-------|-------|------------|
| O(1)  | S(1)  | O(7)  | 119.48(10) | O(1)  | S(1)  | N(1)  | 108.20(10) |
| O(1)  | S(1)  | C(15) | 106.69(10) | O(7)  | S(1)  | N(1)  | 107.21(10) |
| O(7)  | S(1)  | C(15) | 106.72(11) | N(1)  | S(1)  | C(15) | 108.10(12) |
| C(14) | S(3)  | C(24) | 102.95(14) | C(20) | O(4)  | C(25) | 114.37(19) |
| C(13) | O(5)  | C(22) | 114.4(2)   | S(1)  | N(1)  | C(12) | 116.00(16) |
| S(1)  | N(1)  | C(23) | 119.7(2)   | C(12) | N(1)  | C(23) | 119.0(2)   |
| C(15) | C(9)  | C(18) | 118.5(2)   | C(15) | C(9)  | H(1)  | 125.4(12)  |
| C(18) | C(9)  | H(1)  | 116.1(12)  | C(13) | C(10) | C(16) | 121.4(2)   |
| C(13) | C(10) | H(5)  | 121.1(10)  | C(16) | C(10) | H(5)  | 117.5(10)  |
| C(12) | C(11) | C(20) | 118.9(2)   | C(12) | C(11) | H(4)  | 119.2(10)  |
| C(20) | C(11) | H(4)  | 121.9(10)  | N(1)  | C(12) | C(11) | 118.7(2)   |
| N(1)  | C(12) | C(16) | 119.3(2)   | C(11) | C(12) | C(16) | 122.0(2)   |
| O(5)  | C(13) | C(10) | 118.9(2)   | O(5)  | C(13) | C(20) | 121.8(2)   |
| C(10) | C(13) | C(20) | 119.4(2)   | S(3)  | C(14) | C(16) | 111.30(18) |
| S(3)  | C(14) | C(19) | 110.95(16) | S(3)  | C(14) | H(10) | 99.9       |
| C(16) | C(14) | C(19) | 119.8(2)   | C(16) | C(14) | H(10) | 107.2      |
| C(19) | C(14) | H(10) | 105.5      | S(1)  | C(15) | C(9)  | 112.79(19) |
| S(1)  | C(15) | C(19) | 125.2(2)   | C(9)  | C(15) | C(19) | 122.0(2)   |
| C(10) | C(16) | C(12) | 118.3(2)   | C(10) | C(16) | C(14) | 120.7(2)   |
| C(12) | C(16) | C(14) | 121.0(2)   | C(19) | C(17) | C(21) | 123.6(2)   |
| C(19) | C(17) | H(3)  | 115.2(14)  | C(21) | C(17) | H(3)  | 121.1(14)  |
| Cl(2) | C(18) | C(9)  | 119.9(2)   | Cl(2) | C(18) | C(21) | 118.2(2)   |
| C(9)  | C(18) | C(21) | 121.9(2)   | C(14) | C(19) | C(15) | 127.3(2)   |
| C(14) | C(19) | C(17) | 116.2(2)   | C(15) | C(19) | C(17) | 116.4(2)   |
| O(4)  | C(20) | C(11) | 117.6(2)   | O(4)  | C(20) | C(13) | 122.4(2)   |
| C(11) | C(20) | C(13) | 120.1(2)   | C(17) | C(21) | C(18) | 117.6(2)   |
| C(17) | C(21) | H(2)  | 121.7(12)  | C(18) | C(21) | H(2)  | 120.7(12)  |
| O(5)  | C(22) | C(25) | 123.6(3)   | O(5)  | C(22) | H(11) | 104.5      |
| O(5)  | C(22) | H(12) | 106.6      | C(25) | C(22) | H(11) | 103.8      |
| C(25) | C(22) | H(12) | 108.4      | H(11) | C(22) | H(12) | 109.5      |
| N(1)  | C(23) | C(24) | 112.8(2)   | N(1)  | C(23) | H(8)  | 105.9(13)  |
| N(1)  | C(23) | H(9)  | 103.4(13)  | C(24) | C(23) | H(8)  | 113.6(13)  |
| C(24) | C(23) | H(9)  | 110.9(13)  | H(8)  | C(23) | H(9)  | 109.7(19)  |
| S(3)  | C(24) | C(23) | 116.6(2)   | S(3)  | C(24) | H(6)  | 106.4(14)  |
| S(3)  | C(24) | H(7)  | 111.7(17)  | C(23) | C(24) | H(6)  | 112.6(14)  |
| C(23) | C(24) | H(7)  | 103.4(18)  | H(6)  | C(24) | H(7)  | 106(2)     |
| O(4)  | C(25) | C(22) | 120.7(3)   | O(4)  | C(25) | H(13) | 106.0      |

Table 4. Bond angles (°) (continued)

| atom  | atom  | atom  | angle | atom  | atom  | atom  | angle |
|-------|-------|-------|-------|-------|-------|-------|-------|
| O(4)  | C(25) | H(14) | 106.6 | C(22) | C(25) | H(13) | 104.6 |
| C(22) | C(25) | H(14) | 109.1 | H(13) | C(25) | H(14) | 109.5 |

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

| atom1 | atom2 | atom3 | atom4 | angle       | atom1 | atom2 | atom3 | atom4 | angle       |
|-------|-------|-------|-------|-------------|-------|-------|-------|-------|-------------|
| O(1)  | S(1)  | N(1)  | C(12) | 44.51(18)   | O(1)  | S(1)  | N(1)  | C(23) | -161.48(19) |
| O(1)  | S(1)  | C(15) | C(9)  | 78.39(19)   | O(1)  | S(1)  | C(15) | C(19) | -102.9(2)   |
| O(7)  | S(1)  | N(1)  | C(12) | 174.61(15)  | O(7)  | S(1)  | N(1)  | C(23) | -31.4(2)    |
| O(7)  | S(1)  | C(15) | C(9)  | -50.40(19)  | O(7)  | S(1)  | C(15) | C(19) | 128.4(2)    |
| N(1)  | S(1)  | C(15) | C(9)  | -165.43(17) | N(1)  | S(1)  | C(15) | C(19) | 13.3(2)     |
| C(15) | S(1)  | N(1)  | C(12) | -70.67(18)  | C(15) | S(1)  | N(1)  | C(23) | 83.3(2)     |
| C(14) | S(3)  | C(24) | C(23) | 60.1(3)     | C(24) | S(3)  | C(14) | C(16) | -79.9(2)    |
| C(24) | S(3)  | C(14) | C(19) | 56.2(2)     | C(20) | O(4)  | C(25) | C(22) | -12.5(4)    |
| C(25) | O(4)  | C(20) | C(11) | -179.4(2)   | C(25) | O(4)  | C(20) | C(13) | 1.1(3)      |
| C(13) | O(5)  | C(22) | C(25) | -16.7(5)    | C(22) | O(5)  | C(13) | C(10) | -174.5(2)   |
| C(22) | O(5)  | C(13) | C(20) | 4.7(3)      | S(1)  | N(1)  | C(12) | C(11) | -105.9(2)   |
| S(1)  | N(1)  | C(12) | C(16) | 74.6(2)     | S(1)  | N(1)  | C(23) | C(24) | -67.3(3)    |
| C(12) | N(1)  | C(23) | C(24) | 86.0(3)     | C(23) | N(1)  | C(12) | C(11) | 99.9(2)     |
| C(23) | N(1)  | C(12) | C(16) | -79.6(3)    | C(15) | C(9)  | C(18) | Cl(2) | -178.91(18) |
| C(15) | C(9)  | C(18) | C(21) | -0.4(3)     | C(18) | C(9)  | C(15) | S(1)  | 179.32(19)  |
| C(18) | C(9)  | C(15) | C(19) | 0.5(3)      | C(13) | C(10) | C(16) | C(12) | -0.1(3)     |
| C(13) | C(10) | C(16) | C(14) | 177.1(2)    | C(16) | C(10) | C(13) | O(5)  | 177.3(2)    |
| C(16) | C(10) | C(13) | C(20) | -1.9(4)     | C(12) | C(11) | C(20) | O(4)  | -179.8(2)   |
| C(12) | C(11) | C(20) | C(13) | -0.3(4)     | C(20) | C(11) | C(12) | N(1)  | 178.7(2)    |
| C(20) | C(11) | C(12) | C(16) | -1.8(4)     | N(1)  | C(12) | C(16) | C(10) | -178.4(2)   |
| N(1)  | C(12) | C(16) | C(14) | 4.4(3)      | C(11) | C(12) | C(16) | C(10) | 2.0(4)      |
| C(11) | C(12) | C(16) | C(14) | -175.1(2)   | O(5)  | C(13) | C(20) | O(4)  | 2.4(4)      |
| O(5)  | C(13) | C(20) | C(11) | -177.1(2)   | C(10) | C(13) | C(20) | O(4)  | -178.4(2)   |
| C(10) | C(13) | C(20) | C(11) | 2.1(4)      | S(3)  | C(14) | C(16) | C(10) | -110.2(2)   |
| S(3)  | C(14) | C(16) | C(12) | 66.9(2)     | S(3)  | C(14) | C(19) | C(15) | -90.0(2)    |
| S(3)  | C(14) | C(19) | C(17) | 89.0(2)     | C(16) | C(14) | C(19) | C(15) | 41.9(3)     |
| C(16) | C(14) | C(19) | C(17) | -139.1(2)   | C(19) | C(14) | C(16) | C(10) | 118.1(2)    |
| C(19) | C(14) | C(16) | C(12) | -64.8(3)    | S(1)  | C(15) | C(19) | C(14) | 0.3(3)      |
| S(1)  | C(15) | C(19) | C(17) | -178.74(18) | C(9)  | C(15) | C(19) | C(14) | 178.9(2)    |
| C(9)  | C(15) | C(19) | C(17) | -0.1(2)     | C(19) | C(17) | C(21) | C(18) | 0.5(4)      |
| C(21) | C(17) | C(19) | C(14) | -179.6(2)   | C(21) | C(17) | C(19) | C(15) | -0.4(3)     |
| Cl(2) | C(18) | C(21) | C(17) | 178.4(2)    | C(9)  | C(18) | C(21) | C(17) | -0.1(3)     |
| O(5)  | C(22) | C(25) | O(4)  | 21.4(5)     | N(1)  | C(23) | C(24) | S(3)  | -60.4(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  |
|-------|----------------------|-----------|-------|----------------------|-----------|
| Cl(2) | C(10) <sup>11</sup>  | 3.461(2)  | Cl(2) | H(5) <sup>21</sup>   | 3.120(17) |
| Cl(2) | H(5) <sup>11</sup>   | 3.195(19) | Cl(2) | H(10) <sup>21</sup>  | 2.843     |
| Cl(2) | H(11) <sup>31</sup>  | 3.534     | S(1)  | H(4) <sup>41</sup>   | 3.511(17) |
| S(3)  | C(9) <sup>51</sup>   | 3.563(2)  | S(3)  | H(6) <sup>51</sup>   | 2.87(2)   |
| O(1)  | C(22) <sup>61</sup>  | 3.564(4)  | O(1)  | C(25) <sup>61</sup>  | 3.398(3)  |
| O(1)  | H(2) <sup>11</sup>   | 3.40(2)   | O(1)  | H(8) <sup>41</sup>   | 2.94(2)   |
| O(1)  | H(11) <sup>61</sup>  | 2.828     | O(1)  | H(13) <sup>21</sup>  | 3.596     |
| O(1)  | H(14) <sup>61</sup>  | 2.588     | O(4)  | C(23) <sup>71</sup>  | 3.569(4)  |
| O(4)  | H(7) <sup>81</sup>   | 3.37(2)   | O(4)  | H(8) <sup>81</sup>   | 2.81(2)   |
| O(4)  | H(9) <sup>71</sup>   | 2.65(2)   | O(4)  | H(12) <sup>61</sup>  | 3.164     |
| O(4)  | H(13) <sup>61</sup>  | 2.892     | O(5)  | C(17) <sup>91</sup>  | 3.350(3)  |
| O(5)  | H(2) <sup>101</sup>  | 3.51(2)   | O(5)  | H(2) <sup>91</sup>   | 3.11(2)   |
| O(5)  | H(3) <sup>91</sup>   | 2.61(2)   | O(5)  | H(12) <sup>61</sup>  | 3.429     |
| O(7)  | C(11) <sup>41</sup>  | 3.316(2)  | O(7)  | C(24) <sup>111</sup> | 3.480(4)  |
| O(7)  | H(4) <sup>41</sup>   | 2.341(17) | O(7)  | H(7) <sup>111</sup>  | 2.63(3)   |
| O(7)  | H(14) <sup>121</sup> | 3.235     | C(9)  | S(3) <sup>111</sup>  | 3.563(2)  |
| C(10) | Cl(2) <sup>11</sup>  | 3.461(2)  | C(10) | H(1) <sup>131</sup>  | 3.21(2)   |
| C(10) | H(3) <sup>91</sup>   | 3.35(2)   | C(11) | O(7) <sup>41</sup>   | 3.316(2)  |
| C(11) | C(25) <sup>61</sup>  | 3.491(4)  | C(11) | H(12) <sup>61</sup>  | 3.356     |
| C(11) | H(13) <sup>61</sup>  | 3.339     | C(11) | H(14) <sup>61</sup>  | 2.934     |
| C(12) | H(14) <sup>61</sup>  | 3.505     | C(13) | H(3) <sup>91</sup>   | 3.42(2)   |
| C(13) | H(12) <sup>61</sup>  | 3.046     | C(17) | O(5) <sup>91</sup>   | 3.350(3)  |
| C(17) | H(5) <sup>91</sup>   | 3.275(17) | C(20) | C(22) <sup>61</sup>  | 3.563(5)  |
| C(20) | C(25) <sup>61</sup>  | 3.570(4)  | C(20) | H(9) <sup>71</sup>   | 3.42(2)   |
| C(20) | H(12) <sup>61</sup>  | 2.874     | C(20) | H(13) <sup>61</sup>  | 3.175     |
| C(20) | H(14) <sup>61</sup>  | 3.365     | C(22) | O(1) <sup>141</sup>  | 3.564(4)  |
| C(22) | C(20) <sup>141</sup> | 3.563(5)  | C(22) | H(2) <sup>101</sup>  | 3.46(2)   |
| C(22) | H(2) <sup>91</sup>   | 3.55(2)   | C(22) | H(12) <sup>61</sup>  | 3.481     |
| C(23) | O(4) <sup>71</sup>   | 3.569(4)  | C(23) | C(25) <sup>71</sup>  | 3.585(5)  |
| C(23) | H(13) <sup>71</sup>  | 2.800     | C(23) | H(14) <sup>121</sup> | 3.532     |
| C(24) | O(7) <sup>51</sup>   | 3.480(4)  | C(24) | H(13) <sup>71</sup>  | 3.506     |
| C(25) | O(1) <sup>141</sup>  | 3.398(3)  | C(25) | C(11) <sup>141</sup> | 3.491(4)  |
| C(25) | C(20) <sup>141</sup> | 3.570(4)  | C(25) | C(23) <sup>71</sup>  | 3.585(5)  |
| C(25) | H(4) <sup>141</sup>  | 3.404(17) | C(25) | H(8) <sup>81</sup>   | 3.04(2)   |
| C(25) | H(8) <sup>71</sup>   | 3.41(2)   | C(25) | H(9) <sup>71</sup>   | 2.97(2)   |
| C(25) | H(12) <sup>61</sup>  | 3.549     | H(1)  | C(10) <sup>21</sup>  | 3.21(2)   |
| H(1)  | H(5) <sup>21</sup>   | 3.01(2)   | H(1)  | H(7) <sup>111</sup>  | 3.34(3)   |



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

| atom  | atom                 | distance  | atom  | atom                 | distance  |
|-------|----------------------|-----------|-------|----------------------|-----------|
| H(2)  | O(1) <sup>11</sup>   | 3.40(2)   | H(2)  | O(5) <sup>33</sup>   | 3.51(2)   |
| H(2)  | O(5) <sup>93</sup>   | 3.11(2)   | H(2)  | C(22) <sup>33</sup>  | 3.46(2)   |
| H(2)  | C(22) <sup>93</sup>  | 3.55(2)   | H(2)  | H(11) <sup>33</sup>  | 2.621     |
| H(2)  | H(12) <sup>93</sup>  | 2.974     | H(3)  | O(5) <sup>93</sup>   | 2.61(2)   |
| H(3)  | C(10) <sup>93</sup>  | 3.35(2)   | H(3)  | C(13) <sup>93</sup>  | 3.42(2)   |
| H(3)  | H(5) <sup>93</sup>   | 2.63(2)   | H(4)  | S(1) <sup>43</sup>   | 3.511(16) |
| H(4)  | O(7) <sup>43</sup>   | 2.341(16) | H(4)  | C(25) <sup>63</sup>  | 3.404(17) |
| H(4)  | H(7) <sup>83</sup>   | 3.25(3)   | H(4)  | H(13) <sup>63</sup>  | 3.218     |
| H(4)  | H(14) <sup>63</sup>  | 2.758     | H(5)  | Cl(2) <sup>133</sup> | 3.120(17) |
| H(5)  | Cl(2) <sup>13</sup>  | 3.195(19) | H(5)  | C(17) <sup>93</sup>  | 3.275(17) |
| H(5)  | H(1) <sup>133</sup>  | 3.01(2)   | H(5)  | H(3) <sup>93</sup>   | 2.63(2)   |
| H(5)  | H(5) <sup>93</sup>   | 3.27(2)   | H(5)  | H(10) <sup>93</sup>  | 2.684     |
| H(6)  | S(3) <sup>113</sup>  | 2.87(2)   | H(6)  | H(7) <sup>113</sup>  | 3.46(3)   |
| H(7)  | O(4) <sup>123</sup>  | 3.37(2)   | H(7)  | O(7) <sup>53</sup>   | 2.63(3)   |
| H(7)  | H(1) <sup>53</sup>   | 3.34(3)   | H(7)  | H(4) <sup>123</sup>  | 3.25(3)   |
| H(7)  | H(6) <sup>53</sup>   | 3.46(3)   | H(7)  | H(13) <sup>73</sup>  | 3.044     |
| H(7)  | H(14) <sup>73</sup>  | 3.581     | H(8)  | O(1) <sup>43</sup>   | 2.94(2)   |
| H(8)  | O(4) <sup>123</sup>  | 2.81(2)   | H(8)  | C(25) <sup>123</sup> | 3.04(2)   |
| H(8)  | C(25) <sup>73</sup>  | 3.41(2)   | H(8)  | H(13) <sup>123</sup> | 3.537     |
| H(8)  | H(13) <sup>73</sup>  | 2.485     | H(8)  | H(14) <sup>123</sup> | 2.393     |
| H(9)  | O(4) <sup>73</sup>   | 2.65(2)   | H(9)  | C(20) <sup>73</sup>  | 3.42(2)   |
| H(9)  | C(25) <sup>73</sup>  | 2.97(2)   | H(9)  | H(13) <sup>73</sup>  | 2.371     |
| H(9)  | H(14) <sup>73</sup>  | 3.466     | H(10) | Cl(2) <sup>133</sup> | 2.843     |
| H(10) | H(5) <sup>93</sup>   | 2.684     | H(11) | Cl(2) <sup>103</sup> | 3.534     |
| H(11) | O(1) <sup>143</sup>  | 2.828     | H(11) | H(2) <sup>103</sup>  | 2.621     |
| H(11) | H(12) <sup>63</sup>  | 3.034     | H(12) | O(4) <sup>143</sup>  | 3.164     |
| H(12) | O(5) <sup>143</sup>  | 3.429     | H(12) | C(11) <sup>143</sup> | 3.356     |
| H(12) | C(13) <sup>143</sup> | 3.046     | H(12) | C(20) <sup>143</sup> | 2.874     |
| H(12) | C(22) <sup>143</sup> | 3.481     | H(12) | C(25) <sup>143</sup> | 3.549     |
| H(12) | H(2) <sup>93</sup>   | 2.974     | H(12) | H(11) <sup>143</sup> | 3.034     |
| H(12) | H(14) <sup>143</sup> | 3.564     | H(13) | O(1) <sup>133</sup>  | 3.596     |
| H(13) | O(4) <sup>143</sup>  | 2.892     | H(13) | C(11) <sup>143</sup> | 3.339     |
| H(13) | C(20) <sup>143</sup> | 3.175     | H(13) | C(23) <sup>73</sup>  | 2.800     |
| H(13) | C(24) <sup>73</sup>  | 3.506     | H(13) | H(4) <sup>143</sup>  | 3.218     |
| H(13) | H(7) <sup>73</sup>   | 3.044     | H(13) | H(8) <sup>83</sup>   | 3.537     |
| H(13) | H(8) <sup>73</sup>   | 2.485     | H(13) | H(9) <sup>73</sup>   | 2.371     |
| H(13) | H(14) <sup>143</sup> | 3.406     | H(14) | O(1) <sup>143</sup>  | 2.588     |

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

| atom  | atom                 | distance | atom  | atom                 | distance |
|-------|----------------------|----------|-------|----------------------|----------|
| H(14) | O(7) <sup>8)</sup>   | 3.235    | H(14) | C(11) <sup>14)</sup> | 2.934    |
| H(14) | C(12) <sup>14)</sup> | 3.505    | H(14) | C(20) <sup>14)</sup> | 3.365    |
| H(14) | C(23) <sup>8)</sup>  | 3.532    | H(14) | H(4) <sup>14)</sup>  | 2.758    |
| H(14) | H(7) <sup>7)</sup>   | 3.581    | H(14) | H(8) <sup>8)</sup>   | 2.393    |
| H(14) | H(9) <sup>7)</sup>   | 3.466    | H(14) | H(12) <sup>6)</sup>  | 3.564    |
| H(14) | H(13) <sup>6)</sup>  | 3.406    |       |                      |          |

Symmetry Operators:

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