Structure report of compound 3a \cdot \text{H}_2\text{O}
119217
GYI0161_1
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X-ray Structure Report

September 29, 2016
Fig. 1. The crystal
Fig. 2. The molecule (some hydrogens were generated by the software)
Fig. 4. Hydrogen-bonds
**Experimental**

**Data Collection**

A colorless prism crystal of \( \text{C}_{11}\text{H}_{14}\text{Cl}_{2}\text{N}_{2}\text{O}_{4}\text{S} \) having approximate dimensions of 0.40 x 0.04 x 0.02 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 tetragonal cell (laue class: 4/mmm) with dimensions:

\[
\begin{align*}
\text{a} &= 20.0729(4) \text{ Å} \\
\text{c} &= 7.2451(2) \text{ Å} \\
\text{V} &= 2919.21(12) \text{ Å}^3
\end{align*}
\]

For \( Z = 8 \) and F.W. = 341.21, the calculated density is 1.553 g/cm\(^3\). Based on the systematic absences of:

\[
\begin{align*}
0kl: & \ k \pm 2n \\
hhl: & \ l \pm 2n
\end{align*}
\]

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

\[ \text{P4}_22\text{bc} \ (#106) \]

The data were collected at a temperature of 20 + 1\(^\circ\)C to a maximum 2\( \theta \) value of 143.3\(^\circ\). 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 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 \( \phi = 180.0^\circ \). 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 32203 reflections that were collected, 2821 were unique (R_{int} = 0.086).

The linear absorption coefficient, \( \mu \), for Cu-K\( \alpha \) radiation is 54.845 cm\(^{-1}\). An empirical absorption correction was applied which resulted in transmission factors ranging from 0.640 to 0.877. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods\(^1\) and expanded using Fourier techniques\(^2\). 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\(^3\) on F was based on 20447 observed reflections (I > 2.00\( \sigma \)(I)) and 207 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

\[
R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|} = 0.0612
\]

\[
R_w = \left[ \frac{\sum w (|F_o| - |F_c|)^2}{\sum w F_o^2} \right]^{1/2} = 0.0744
\]

The standard deviation of an observation of unit weight\(^4\) was 3.13. 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 14.10 and -31.00 e\(^{-} \)/Å\(^3\), respectively.

Neutral atom scattering factors were taken from Cromer and Waber\(^5\). Anomalous dispersion effects were included in Fcalc\(^6\); the values for \( \Delta f' \) and \( \Delta f'' \) were those of Creagh and McAuley\(^7\). The values for the mass attenuation coefficients are those of Creagh and Hubbell\(^8\). 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:

$$\sum w(|F_o|-|F_c|)^2$$

where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$\left(\sum w(|F_o|-|F_c|)^2/(N_o-N_v)\right)^{1/2}$$

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/MSC (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)
A. Crystal Data

Empirical Formula: C_{11}H_{14}Cl_{2}N_{2}O_{4}S

Formula Weight: 341.21

Crystal Color, Habit: colorless, prism

Crystal Dimensions: 0.40 X 0.04 X 0.02 mm

Crystal System: tetragonal

Lattice Type: Primitive

Indexing Images: 4 oscillations @ 900.0 seconds

Detector Position: 127.40 mm

Pixel Size: 0.100 mm

Lattice Parameters:
\[ a = 20.0729(4) \, \text{Å} \]
\[ c = 7.2451(2) \, \text{Å} \]
\[ V = 2919.21(12) \, \text{Å}^3 \]

Space Group: P4_{2}bc (\#106)

Z value: 8

D_{calc}: 1.553 g/cm^{3}

F_{000}: 1408.00

\[ \mu(\text{CuK}\alpha) = 54.845 \, \text{cm}^{-1} \]
B. Intensity Measurements

| Parameter                      | Details                                      |
|--------------------------------|----------------------------------------------|
| Diffractometer                 | Rigaku RAXIS-RAPID                           |
| Radiation                      | CuKα (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                  | 180.0 sec./°                                 |
| ω oscillation Range (χ=54.0, φ=0.0) | 20.0 - 200.0°                               |
| Exposure Rate                  | 180.0 sec./°                                 |
| ω oscillation Range (χ=54.0, φ=90.0) | 20.0 - 200.0°                               |
| Exposure Rate                  | 180.0 sec./°                                 |
| ω oscillation Range (χ=54.0, φ=180.0) | 20.0 - 200.0°                               |
| Exposure Rate                  | 180.0 sec./°                                 |
| ω oscillation Range (χ=54.0, φ=270.0) | 20.0 - 200.0°                               |
| Exposure Rate                  | 180.0 sec./°                                 |
| Detector Position              | 127.40 mm                                    |
| Pixel Size                     | 0.100 mm                                     |
| 2θ<sub>max</sub>               | 143.3°                                       |
| No. of Reflections Measured    | Total: 32203                                  |
|                               | Unique: 2821 (R<sub>int</sub> = 0.086)       |
| Corrections                    | Lorentz-polarization                         |
|                               | Absorption                                   |
|                               | (trans. factors: 0.640 - 0.877)              |
C. Structure Solution and Refinement

Structure Solution  
Direct Methods (SIR92)

Refinement  
Full-matrix least-squares on F

Function Minimized  
$\sum w (|F_0| - |F_c|)^2$

Least Squares Weights  
1

$2\theta_{\text{max}}$ cutoff  
143.3°

Anomalous Dispersion  
All non-hydrogen atoms

No. Observations ($I > 2.00 \sigma(I)$)  
20447

No. Variables  
207

Reflection/Parameter Ratio  
98.78

Residuals: R ($I > 2.00 \sigma(I)$)  
0.0612

Residuals: Rw ($I > 2.00 \sigma(I)$)  
0.0744

Goodness of Fit Indicator  
3.128

Max Shift/Error in Final Cycle  
0.000

Maximum peak in Final Diff. Map  
14.10 e⁻/Å³

Minimum peak in Final Diff. Map  
-31.00 e⁻/Å³
Table 1. Atomic coordinates and B_{iso}/B_{eq}

| atom | x      | y      | z      | B_{eq} |
|------|--------|--------|--------|--------|
| Cl(1)| 0.23905(5) | 0.38085(4) | -0.04649(18) | 4.26(2) |
| Cl(2)| 0.10595(4) | 0.30321(4) | -0.07972(18) | 3.96(2) |
| S(1) | 0.10184(5) | 0.14645(5) | -0.00046(16) | 3.22(2) |
| O(1) | 0.35169(14) | 0.04361(16) | 0.3251(4) | 5.55(9) |
| O(2) | 0.2841(2) | 0.04491(18) | -0.3407(5) | 5.97(11) |
| O(3) | 0.10184(5) | 0.14645(5) | -0.00046(16) | 3.22(2) |
| O(4) | 0.11482(14) | 0.07789(14) | 0.1103(4) | 3.53(9) |
| O(5) | 0.29781(19) | 0.05725(18) | 0.0549(4) | 3.26(9) |
| C(9) | 0.17595(16) | 0.19354(18) | 0.0337(4) | 2.08(8) |
| C(10)| 0.23382(19) | 0.16236(17) | 0.0870(4) | 2.43(9) |
| C(12)| 0.17763(18) | 0.26282(18) | -0.0115(4) | 2.59(9) |
| C(13)| 0.29117(18) | 0.1991(2) | 0.1071(4) | 2.75(10) |
| C(14)| 0.3499(2) | 0.0378(2) | 0.1623(7) | 3.84(12) |
| C(15)| 0.29283(19) | 0.26518(19) | 0.0706(4) | 3.02(10) |
| C(16)| 0.17842(18) | 0.04754(17) | 0.0630(5) | 3.41(10) |
| C(17)| 0.23550(19) | 0.29759(17) | 0.0111(4) | 2.64(9) |
| C(18)| 0.2393(2) | 0.0870(2) | 0.1278(5) | 2.60(10) |
| C(19)| 0.09498(19) | 0.07488(19) | 0.3050(5) | 5.12(13) |
| C(20)| 0.40603(18) | 0.0058(2) | 0.0543(7) | 5.83(13) |
| H(1) | 0.2325(12) | 0.0809(12) | 0.257(3) | -0.8(5) |
| H(2) | 0.2710(13) | 0.0070(13) | -0.348(4) | 0.6(7) |
| H(3) | 0.3100(17) | 0.0433(17) | -0.451(5) | 4.3(11) |
| H(4) | 0.3059(12) | 0.0524(11) | -0.064(3) | -2.9(5) |
| H(5) | 0.3306 | 0.1772 | 0.1468 | 3.27 |
| H(6) | 0.3330 | 0.2897 | 0.0842 | 3.65 |
| H(7) | 0.1803 | 0.0041 | 0.1150 | 4.10 |
| H(8) | 0.1800 | 0.0444 | -0.0677 | 4.07 |
| H(9) | 0.1314 | 0.0889 | 0.3790 | 6.17 |
| H(10) | 0.0834 | 0.0304 | 0.3361 | 6.16 |
| H(11) | 0.0579 | 0.1033 | 0.3263 | 6.16 |
| H(12) | 0.4391 | 0.0374 | 0.0204 | 6.98 |
| H(13) | 0.3876 | -0.0137 | -0.0537 | 6.98 |
| H(14) | 0.4256 | -0.0280 | 0.1283 | 7.00 |

\[ B_{eq} = \frac{8}{3} \pi^2 (u_{11}(aa^*)^2 + u_{22}(bb^*)^2 + u_{33}(cc^*)^2 + 2u_{12}(aa^*bb^*)\cos \gamma + 2u_{13}(aa^*cc^*)\cos \beta + 2u_{23}(bb^*cc^*)\cos \alpha) \]
Table 2. Anisotropic displacement parameters

| atom | U_{11}   | U_{22}   | U_{33}   | U_{12}   | U_{13}   | U_{23}   |
|------|----------|----------|----------|----------|----------|----------|
| Cl(1)| 0.0698(7)| 0.0337(5)| 0.0582(7)| -0.0086(5)| 0.0123(6)| -0.0004(5)|
| Cl(2)| 0.0439(6)| 0.0441(6)| 0.0626(7)| 0.0093(4) | -0.0038(6)| 0.0116(6)|
| S(1) | 0.0307(6)| 0.0378(6)| 0.0539(7)| -0.0038(5)| -0.0103(5)| -0.0031(6)|
| O(1) | 0.056(2) | 0.106(2) | 0.049(2) | 0.0046(19)| -0.0016(19)| 0.020(2) |
| O(2) | 0.106(3) | 0.055(2) | 0.066(2) | -0.041(2) | 0.017(2) | -0.021(2)|
| O(4) | 0.0285(16)| 0.0364(17)| 0.093(2) | 0.0024(13)| 0.0057(15)| 0.0076(15)|
| O(6) | 0.050(2) | 0.078(2) | 0.069(2) | -0.0148(17)| -0.0268(17)| -0.0141(18)|
| N(1) | 0.021(2) | 0.043(2) | 0.070(2) | 0.0013(16)| 0.0141(19)| -0.0063(19)|
| N(2) | 0.057(2) | 0.051(2) | 0.015(2) | 0.0094(18)| 0.0197(19)| -0.0061(17)|
| C(9) | 0.019(2) | 0.039(2) | 0.021(2) | 0.0034(17)| -0.0023(16)| -0.0026(18)|
| C(10)| 0.046(2) | 0.021(2) | 0.026(2) | -0.0071(19)| 0.0015(19)| 0.0145(17)|
| C(12)| 0.049(2) | 0.036(2) | 0.014(2) | -0.0018(18)| 0.0068(18)| -0.0046(18)|
| C(13)| 0.025(2) | 0.043(2) | 0.036(2) | 0.0065(19)| 0.0092(19)| 0.014(2) |
| C(14)| 0.034(2) | 0.042(3) | 0.069(3) | -0.004(2) | 0.011(2) | 0.024(2) |
| C(15)| 0.038(2) | 0.037(2) | 0.039(2) | -0.012(2) | -0.022(2) | 0.003(2) |
| C(16)| 0.050(2) | 0.021(2) | 0.059(3) | -0.001(2) | 0.009(2) | -0.008(2) |
| C(17)| 0.042(2) | 0.035(2) | 0.024(2) | -0.0097(19)| 0.001(2) | -0.024(2) |
| C(18)| 0.039(2) | 0.035(2) | 0.025(2) | -0.0007(19)| 0.010(2) | 0.004(2) |
| C(19)| 0.070(3) | 0.064(3) | 0.061(3) | 0.025(2) | 0.032(2) | 0.033(2) |
| C(20)| 0.039(3) | 0.056(3) | 0.126(4) | 0.003(2) | 0.003(3) | 0.016(3) |

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(1) | C(17) | 1.724(3) | Cl(2) | C(12) | 1.724(3) |
| S(1)  | O(4)  | 1.422(2) | S(1)  | O(6)  | 1.415(3) |
| S(1)  | N(1)  | 1.614(3) | S(1)  | C(9)  | 1.780(3) |
| O(1)  | C(14) | 1.186(6) | O(2)  | H(2)  | 0.81(2)  |
| O(2)  | H(3)  | 0.95(3)  | N(1)  | C(16) | 1.455(4) |
| N(1)  | C(19) | 1.467(5) | N(2)  | C(14) | 1.361(6) |
| N(2)  | C(18) | 1.419(5) | N(2)  | H(4)  | 0.88(2)  |
| C(9)  | C(10) | 1.375(4) | C(9)  | C(12) | 1.429(5) |
| C(10) | C(13) | 1.375(5) | C(10) | C(18) | 1.546(5) |
| C(12) | C(17) | 1.365(5) | C(13) | C(15) | 1.352(5) |
| C(13) | H(5)  | 0.950    | C(14) | C(20) | 1.514(6) |
| C(15) | C(17) | 1.390(5) | C(15) | H(6)  | 0.950    |
| C(16) | C(18) | 1.530(5) | C(16) | H(7)  | 0.950    |
| C(16) | H(8)  | 0.950    | C(18) | H(1)  | 0.95(2)  |
| C(19) | H(9)  | 0.950    | C(19) | H(10) | 0.950    |
| C(19) | H(11) | 0.950    | C(20) | H(12) | 0.950    |
| C(20) | H(13) | 0.950    | C(20) | H(14) | 0.950    |
Table 4. Bond angles (°)

| atom | atom | atom | angle  | atom | atom | atom | angle  |
|------|------|------|--------|------|------|------|--------|
| O(4) | S(1) | O(6) | 118.44(17) | O(4) | S(1) | N(1) | 106.61(16) |
| O(4) | S(1) | C(9) | 111.35(15) | O(6) | S(1) | N(1) | 109.09(17) |
| O(6) | S(1) | C(9) | 106.09(17) | N(1) | S(1) | C(9) | 104.40(16) |
| H(2) | O(2) | H(3) | 95(3) | S(1) | N(1) | C(16) | 112.4(2) |
| S(1) | N(1) | C(19) | 118.0(2) | C(16) | N(1) | C(19) | 116.6(3) |
| C(14) | N(2) | C(18) | 123.0(3) | C(14) | N(2) | H(4) | 112.6(16) |
| C(18) | N(2) | H(4) | 124.2(16) | S(1) | C(9) | C(10) | 112.2(3) |
| S(1) | C(9) | C(12) | 120.3(2) | C(10) | C(9) | C(12) | 119.2(3) |
| C(9) | C(10) | C(13) | 119.5(3) | C(9) | C(10) | C(12) | 124.0(3) |
| C(13) | C(10) | C(18) | 116.5(3) | C(12) | C(9) | C(12) | 120.2(2) |
| Cl(2) | C(12) | C(17) | 120.3(2) | C(9) | C(12) | C(17) | 119.4(3) |
| C(10) | C(13) | C(15) | 121.8(3) | C(10) | C(13) | H(5) | 118.7 |
| C(15) | C(13) | H(5) | 119.6 | O(1) | C(14) | N(2) | 124.2(4) |
| O(1) | C(14) | C(20) | 122.3(4) | N(2) | C(14) | C(20) | 113.5(4) |
| C(13) | C(15) | C(17) | 119.9(3) | C(13) | C(15) | H(6) | 120.5 |
| C(17) | C(15) | H(6) | 119.5 | N(1) | C(16) | C(18) | 114.4(2) |
| N(1) | C(16) | H(7) | 109.0 | N(1) | C(16) | H(8) | 107.0 |
| C(18) | C(16) | H(7) | 108.8 | C(18) | C(16) | H(8) | 108.2 |
| H(7) | C(16) | H(8) | 109.5 | Cl(1) | C(17) | C(12) | 120.1(2) |
| Cl(1) | C(17) | C(15) | 119.6(2) | C(12) | C(17) | C(15) | 120.1(3) |
| N(2) | C(18) | C(10) | 113.6(3) | N(2) | C(18) | C(16) | 109.2(3) |
| N(2) | C(18) | H(1) | 115.5(15) | C(10) | C(18) | C(16) | 113.0(3) |
| C(10) | C(18) | H(1) | 107.5(15) | C(16) | C(18) | H(1) | 96.9(15) |
| N(1) | C(19) | H(9) | 108.8 | N(1) | C(19) | H(10) | 109.5 |
| N(1) | C(19) | H(11) | 110.1 | H(9) | C(19) | H(10) | 109.5 |
| H(9) | C(19) | H(11) | 109.5 | H(10) | C(19) | H(11) | 109.5 |
| C(14) | C(20) | H(12) | 111.7 | C(14) | C(20) | H(13) | 108.1 |
| C(14) | C(20) | H(14) | 108.6 | H(12) | C(20) | H(13) | 109.5 |
| H(12) | C(20) | H(14) | 109.5 | H(13) | C(20) | H(14) | 109.5 |
Table 5. Torsion Angles(°)

| atom1 | atom2 | atom3 | atom4 | angle   | atom1 | atom2 | atom3 | atom4 | angle   |
|-------|-------|-------|-------|---------|-------|-------|-------|-------|---------|
| O(4)  | S(1)  | N(1)  | C(16) | -168.2(2) | O(4)  | S(1)  | N(1)  | C(19) | -28.1(3) |
| O(4)  | S(1)  | C(9)  | C(10) | 134.1(2)  | O(4)  | S(1)  | C(9)  | C(12) | -52.2(3) |
| O(6)  | S(1)  | N(1)  | C(16) | 62.9(2)   | O(6)  | S(1)  | N(1)  | C(19) | -157.1(2)|
| O(6)  | S(1)  | C(9)  | C(10) | -95.8(3)  | O(6)  | S(1)  | C(9)  | C(12) | 78.0(3) |
| N(1)  | S(1)  | C(9)  | C(10) | 19.4(3)   | N(1)  | S(1)  | C(9)  | C(12) | -166.8(2)|
| C(9)  | S(1)  | N(1)  | C(16) | -50.2(2)  | C(9)  | S(1)  | N(1)  | C(19) | 89.9(2) |
| S(1)  | N(1)  | C(16) | C(18) | 67.9(3)   | C(19) | N(1)  | C(16) | C(18) | -72.8(4) |
| C(14) | N(2)  | C(18) | C(10) | 107.2(4)  | C(14) | N(2)  | C(18) | C(16) | -125.6(4)|
| C(18) | N(2)  | C(14) | O(1)  | -0.0(6)   | C(18) | N(2)  | C(14) | C(20) | 178.5(3) |
| S(1)  | C(9)  | C(10) | C(13) | 177.9(2)  | S(1)  | C(9)  | C(10) | C(18) | -2.3(4) |
| S(1)  | C(9)  | C(12) | C(2)  | 5.9(4)    | S(1)  | C(9)  | C(12) | C(17) | -178.2(2)|
| C(10) | C(9)  | C(12) | C(2)  | 179.7(2)  | C(10) | C(9)  | C(12) | C(17) | -4.4(5) |
| C(12) | C(9)  | C(10) | C(13) | 4.1(5)    | C(12) | C(9)  | C(10) | C(18) | -176.1(3)|
| C(9)  | C(10) | C(13) | C(15) | -1.9(5)   | C(9)  | C(10) | C(18) | N(2)  | 137.8(3) |
| C(9)  | C(10) | C(18) | C(16) | 12.6(5)   | C(13) | C(10) | C(18) | N(2)  | -42.4(4) |
| C(13) | C(10) | C(18) | C(16) | -167.6(3) | C(18) | C(10) | C(13) | C(15) | 178.2(3) |
| C(2)  | C(12) | C(17) | C(1)  | -5.3(4)   | C(2)  | C(12) | C(17) | C(15) | 178.4(2) |
| C(9)  | C(12) | C(17) | C(1)  | 178.9(2)  | C(9)  | C(12) | C(17) | C(15) | 2.5(5)   |
| C(10) | C(13) | C(15) | C(17) | -0.0(4)   | C(13) | C(15) | C(17) | C(1)  | -176.7(2)|
| C(13) | C(15) | C(17) | C(12) | -0.3(5)   | N(1)  | C(16) | C(18) | N(2)  | -172.9(3)|
| N(1)  | C(16) | C(18) | C(10) | -45.4(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(1) | Cl(2) | 3.4966(18) | Cl(1) | O(4)  | 3.478(2)  |
| Cl(1) | C(9)  | 3.519(3)  | Cl(1) | C(12) | 3.568(3)  |
| Cl(1) | H(2)  | 3.35(2)   | Cl(1) | H(4)  | 3.56(2)   |
| Cl(1) | H(7)  | 3.181     | Cl(1) | H(9)  | 3.504     |
| Cl(1) | H(11) | 3.518     | Cl(1) | H(13) | 3.308     |
| Cl(2) | Cl(1) | 3.4966(18) | Cl(2) | O(1)  | 3.230(3)  |
| Cl(2) | O(2)  | 3.510(4)  | Cl(2) | C(14) | 3.563(4)  |
| Cl(2) | H(2)  | 2.90(2)   | Cl(2) | H(3)  | 3.14(3)   |
| S(1)  | H(2)  | 3.50(2)   | S(1)  | H(7)  | 3.570     |
| S(1)  | H(10) | 3.170     | O(1)  | Cl(2) | 3.230(3)  |
| O(1)  | O(2)  | 2.775(4)  | O(1)  | C(20) | 3.459(5)  |
| O(1)  | H(2)  | 2.96(3)   | O(1)  | H(3)  | 1.83(3)   |
| O(1)  | H(5)  | 3.485     | O(1)  | H(12) | 2.661     |
| O(2)  | Cl(2) | 3.510(3)  | O(2)  | O(1)  | 2.776(4)  |
| O(2)  | O(4)  | 2.864(4)  | O(2)  | N(2)  | 2.890(5)  |
| O(2)  | C(13) | 3.329(5)  | O(2)  | C(15) | 3.464(5)  |
| O(2)  | H(1)  | 3.18(2)   | O(2)  | H(4)  | 2.06(2)   |
| O(2)  | H(5)  | 2.619     | O(2)  | H(6)  | 2.914     |
| O(2)  | H(8)  | 2.876     | O(2)  | H(13) | 3.165     |
| O(4)  | Cl(1) | 3.478(2)  | O(4)  | O(2)  | 2.864(4)  |
| O(4)  | C(19) | 3.571(4)  | O(4)  | H(2)  | 2.21(2)   |
| O(4)  | H(3)  | 3.22(3)   | O(4)  | H(7)  | 3.549     |
| O(4)  | H(8)  | 3.106     | O(4)  | H(9)  | 3.228     |
| O(4)  | H(10) | 3.029     | O(6)  | C(15) | 3.558(4)  |
| O(6)  | C(16) | 3.553(4)  | O(6)  | C(19) | 3.566(4)  |
| O(6)  | H(6)  | 2.851     | O(6)  | H(7)  | 2.671     |
| O(6)  | H(9)  | 3.297     | O(6)  | H(10) | 2.782     |
| N(1)  | H(10) | 3.529     | N(1)  | H(11) | 3.423     |
| N(2)  | O(2)  | 2.890(5)  | N(2)  | H(2)  | 3.13(3)   |
| C(9)  | Cl(1) | 3.519(3)  | C(9)  | C(15) | 3.568(4)  |
| C(9)  | H(6)  | 3.372     | C(12) | Cl(1) | 3.568(3)  |
| C(12) | C(15) | 3.426(4)  | C(12) | C(17) | 3.495(4)  |
| C(12) | H(6)  | 3.566     | C(13) | O(2)  | 3.329(5)  |
| C(13) | C(15) | 3.547(4)  | C(13) | H(3)  | 3.35(3)   |
| C(13) | H(5)  | 3.446     | C(14) | Cl(2) | 3.563(4)  |
| C(14) | H(3)  | 2.92(3)   | C(14) | H(12) | 3.473     |
| C(15) | O(2)  | 3.464(5)  | C(15) | O(6)  | 3.558(4)  |
Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

| atom         | atom      | distance  | atom         | atom      | distance  |
|--------------|-----------|-----------|--------------|-----------|-----------|
| C(15)        | C(9)      | 3.568(4)  | C(15)        | C(12)     | 3.426(4)  |
| C(15)        | C(13)     | 3.547(4)  | C(15)        | H(1)      | 3.40(2)   |
| C(15)        | H(9)      | 3.445     | C(16)        | O(6)      | 3.553(4)  |
| C(16)        | H(11)     | 3.114     | C(17)        | C(12)     | 3.495(4)  |
| C(18)        | H(3)      | 3.48(3)   | C(19)        | O(4)      | 3.571(4)  |
| C(19)        | O(6)      | 3.566(4)  | C(19)        | H(6)      | 3.588     |
| C(20)        | O(1)      | 3.459(5)  | C(20)        | H(12)     | 3.237     |
| C(20)        | H(14)     | 3.450     | H(1)         | O(2)      | 3.18(2)   |
| H(1)         | C(15)     | 3.40(2)   | H(1)         | H(2)      | 3.32(3)   |
| H(1)         | H(3)      | 2.73(4)   | H(1)         | H(6)      | 2.967     |
| H(2)         | Cl(1)     | 3.35(2)   | H(2)         | Cl(2)     | 2.90(2)   |
| H(2)         | S(1)      | 3.50(2)   | H(2)         | O(1)      | 2.96(3)   |
| H(2)         | O(4)      | 2.21(2)   | H(2)         | N(2)      | 3.13(3)   |
| H(2)         | H(1)      | 3.32(3)   | H(2)         | H(4)      | 2.36(4)   |
| H(2)         | H(5)      | 3.422     | H(2)         | H(6)      | 3.470     |
| H(2)         | H(8)      | 2.831     | H(2)         | H(13)     | 3.191     |
| H(3)         | Cl(2)     | 3.14(3)   | H(3)         | O(1)      | 1.83(3)   |
| H(3)         | O(4)      | 3.22(3)   | H(3)         | C(13)     | 3.35(3)   |
| H(3)         | C(14)     | 2.92(3)   | H(3)         | C(18)     | 3.48(3)   |
| H(3)         | H(1)      | 2.73(4)   | H(3)         | H(4)      | 2.81(4)   |
| H(3)         | H(5)      | 2.641     | H(3)         | H(6)      | 3.199     |
| H(3)         | H(12)     | 3.091     | H(3)         | H(13)     | 3.465     |
| H(4)         | Cl(1)     | 3.56(2)   | H(4)         | O(2)      | 2.06(2)   |
| H(4)         | H(2)      | 2.36(4)   | H(4)         | H(3)      | 2.81(4)   |
| H(4)         | H(5)      | 3.166     | H(5)         | O(1)      | 3.485     |
| H(5)         | O(2)      | 2.619     | H(5)         | C(13)     | 3.446     |
| H(5)         | H(2)      | 3.422     | H(5)         | H(3)      | 2.641     |
| H(5)         | H(4)      | 3.166     | H(6)         | O(2)      | 2.914     |
| H(6)         | O(6)      | 2.851     | H(6)         | C(9)      | 3.372     |
| H(6)         | C(12)     | 3.566     | H(6)         | C(19)     | 3.588     |
| H(6)         | H(1)      | 2.967     | H(6)         | H(2)      | 3.470     |
| H(6)         | H(3)      | 3.199     | H(6)         | H(8)      | 3.575     |
| H(6)         | H(9)      | 2.678     | H(7)         | Cl(1)     | 3.181     |
| H(7)         | S(1)      | 3.570     | H(7)         | O(4)      | 3.549     |
| H(7)         | O(6)      | 2.671     | H(7)         | H(11)     | 2.883     |
| H(8)         | O(2)      | 2.876     | H(8)         | O(4)      | 3.106     |
| H(8)         | H(2)      | 2.831     | H(8)         | H(6)      | 3.575     |
Table 6. Distances beyond the asymmetric unit out to 3.60 Å (continued)

| atom  | atom | distance | atom  | atom | distance |
|-------|------|----------|-------|------|----------|
| H(8)  | H(11) | 2.680    | H(9)  | Cl(1) | 3.504    |
| H(9)  | O(4)  | 3.228    | H(9)  | O(6)  | 3.297    |
| H(9)  | C(15) | 3.445    | H(9)  | H(6)  | 2.678    |
| H(10) | S(1)  | 3.170    | H(10) | O(4)  | 3.029    |
| H(10) | O(6)  | 2.782    | H(10) | N(1)  | 3.529    |
| H(10) | H(10) | 3.563    | H(11) | Cl(1) | 3.518    |
| H(11) | N(1)  | 3.423    | H(11) | C(16) | 3.114    |
| H(11) | H(7)  | 2.883    | H(11) | H(8)  | 2.680    |
| H(12) | O(1)  | 2.661    | H(12) | C(14) | 3.473    |
| H(12) | C(20) | 3.237    | H(12) | H(3)  | 3.091    |
| H(12) | H(12) | 2.870    | H(12) | H(13) | 3.553    |
| H(12) | H(14) | 2.832    | H(12) | H(14) | 3.436    |
| H(13) | Cl(1) | 3.308    | H(13) | O(2)  | 3.165    |
| H(13) | H(2)  | 3.191    | H(13) | H(3)  | 3.465    |
| H(13) | H(12) | 3.553    | H(13) | H(14) | 3.108    |
| H(14) | C(20) | 3.450    | H(14) | H(12) | 2.832    |
| H(14) | H(12) | 3.436    | H(14) | H(13) | 3.108    |
| H(14) | H(14) | 3.190    |  |  |  |

Symmetry Operators:

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

Intramolecular and Intermolecular Hydrogen bonds

| D     | H     | A          | D...A   | D-H    | H...A   | D-H...A |
|-------|-------|------------|---------|--------|---------|---------|
| O(2)  | H(2)  | O(4)[4:0:0:-1] | 2.864(4) | 0.81(2) | 2.21(2) | 138(2)  |
| O(2)  | H(3)  | O(1)[1:0:0:-1] | 2.776(4) | 0.95(3) | 1.83(3) | 174(3)  |
| N(2)  | H(4)  | O(2)       | 2.890(5) | 0.88(2) | 2.06(2) | 157(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.