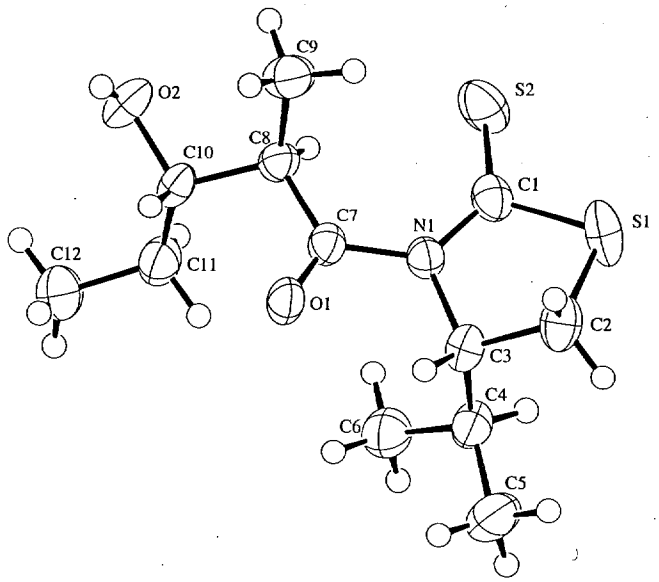

ORTEP Drawing for **10**

The non-hydrogen atoms are represented by 50% probability thermal ellipsoids.
The hydrogen atoms are drawn as circles with an artificial radius.

X-Ray Data for 10.

The data collection crystal was a yellow, rectangular plate. Examination of the diffraction pattern on a Rigaku AFC5S diffractometer indicated an orthorhombic crystal system. Based on the systematic absences, h00, h $\neq$ 2n, 0k0, k $\neq$ 2n, and 00l, l $\neq$ 2n, the space group was uniquely determined as $P2_12_12_1$. Unit cell constants were obtained by a symmetry restricted least-squares fit of the setting angles for 25 reflections in the 2θ range 21 to 28° with MoKα radiation ($\lambda(K\alpha_1) = 0.70930$ Å). Six standard reflections were measured during data collection and indicated that the crystal was stable. Two octants of data were measured: +h, +k, +l and -h, -k, -l. Data reduction was done with the teXsan package[1]. A small analytical absorption correction was applied to the data[2]. Averaging the symmetry equivalent reflections (but not the Friedel pairs) results in an Rint value of 0.029.

The structure was solved by the direct methods procedure in SHELXS-86[3]. The correct enantiomer was chosen based on the known absolute configuration of a stereocenter in the ring. Full-matrix least-squares refinements based on F were performed in teXsan[1]. With the exception of the hydrogen atom bonded to O(2), the hydrogen atoms were included in the model at calculated positions with C-H = 0.98 Å. H(21) bonded to O(2) was located on a difference Fourier map and refined isotropically. The final refinement cycle was based on the 2213 intensities with $I > 1\sigma(I)$ and 158 variables and resulted in final agreement factors of R(F) = 0.051 and Rw(F) = 0.039. The final difference electron density map contains maximum and minimum peak heights of 0.30 and -0.30 e/Å^3. Neutral atom scattering factors were used and include terms for anomalous dispersion[4].

There is an intermolecular hydrogen bond involving the hydroxyl hydrogen atom and it links the molecules into a chain: O(2)...O(2) = 2.797(2)Å and O(2)-H(21)...O(2) = 169(4)°.

References

(1) teXsan: Crystal Structure Analysis Package, version 1.7-2, Molecular Structure Corporation, The Woodlands, TX (1995).

(2) De Meulenaer, J. & Tompa, H., Acta Cryst., (1965), 19, 1014-1018.

(3) SHELXS86: Sheldrick, G. M., Acta Cryst., (1990), A46, 467-473.

(4) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

Table 1. Crystallographic Details for **10**.

Formula	$C_{12}H_{21}NO_2S_2$
Formula wt.	275.42
Space group	$P2_12_12_1$
a, Å	9.173(2)
b, Å	31.304(2)
c, Å	5.041(2)
Volume, Å^3	1447.6(4)
Z	4
Density (calc), g/cm^{-3}	1.26
Bounding planes	{010}, {001}, (110) and (110)
Crystal size, mm	0.10 x 0.30 x 0.25
Radiation	MoKα with graphite monochromator
Linear abs. coeff., cm^{-1}	3.59
Transmissin factors	0.91 to 0.97
Temperature	ambient
2θ limits	$4° \leq 2\theta \leq 55°$
Scan speed	4°/min in ω with maximum of 4 scans/ref.
Background time/scan time	0.5
Scan range	1.30° in ω
Data collected	Two octants: +h, +k, +l and -h, -k, -l
Scan type	ω
Unique data	3379
Unique data, with $F_o^2 > 3\sigma(F_o^2)$	2213
Final number of variables	158
R(F)[a]	0.051
$R_w(F)$[b]	0.039
Error in observation of unit weight, e	1.51

[a] $R(F) = \Sigma||F_o|-|F_c||/\Sigma|F_o|$

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

Table 2. Bond Lengths for **10**

atom	atom	distance	atom	atom	distance
S(1)	C(1)	1.740(3)	C(3)	C(4)	1.541(4)
S(1)	C(2)	1.800(4)	C(4)	C(5)	1.525(5)
S(2)	C(1)	1.643(4)	C(4)	C(6)	1.506(4)
O(1)	C(7)	1.201(4)	C(7)	C(8)	1.504(4)
O(2)	C(10)	1.438(4)	C(8)	C(9)	1.534(5)
N(1)	C(1)	1.374(4)	C(8)	C(10)	1.542(4)
N(1)	C(3)	1.501(4)	C(10)	C(11)	1.516(4)
N(1)	C(7)	1.437(4)	C(11)	C(12)	1.517(5)
C(2)	C(3)	1.523(4)			

Distances are in angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

Table 3. Bond Angles for **10**

atom	atom	atom	angle	atom	atom	atom	angle
C(1)	S(1)	C(2)	94.0(2)	C(3)	C(4)	C(6)	114.4(3)
C(1)	N(1)	C(3)	114.0(2)	C(5)	C(4)	C(6)	110.2(3)
C(1)	N(1)	C(7)	129.9(3)	O(1)	C(7)	N(1)	117.5(3)
C(3)	N(1)	C(7)	115.9(2)	O(1)	C(7)	C(8)	122.1(3)
S(1)	C(1)	S(2)	117.8(2)	N(1)	C(7)	C(8)	120.4(3)
S(1)	C(1)	N(1)	110.4(2)	C(7)	C(8)	C(9)	108.6(3)
S(2)	C(1)	N(1)	131.7(3)	C(7)	C(8)	C(10)	109.0(2)
S(1)	C(2)	C(3)	104.5(2)	C(9)	C(8)	C(10)	110.6(3)
N(1)	C(3)	C(2)	105.4(3)	O(2)	C(10)	C(8)	109.1(3)
N(1)	C(3)	C(4)	112.0(3)	O(2)	C(10)	C(11)	107.7(3)
C(2)	C(3)	C(4)	112.2(3)	C(8)	C(10)	C(11)	113.8(3)
C(3)	C(4)	C(5)	108.3(3)	C(10)	C(11)	C(12)	113.0(3)

Angles are in degrees. Estimated standard deviations in the least significant figure are given in parentheses.

Table 4. Bond Lengths Involving the Hydrogen Atoms for **10**

atom	atom	distance	atom	atom	distance
O(2)	H(21)	0.75(3)	C(8)	H(14)	0.980
C(2)	H(10)	0.979	C(9)	H(7)	0.981
C(2)	H(11)	0.981	C(9)	H(8)	0.980
C(3)	H(12)	0.981	C(9)	H(9)	0.980
C(4)	H(13)	0.980	C(10)	H(15)	0.979
C(5)	H(4)	0.980	C(11)	H(16)	0.980
C(5)	H(5)	0.979	C(11)	H(17)	0.980
C(5)	H(6)	0.980	C(12)	H(18)	0.979
C(6)	H(1)	0.979	C(12)	H(19)	0.981
C(6)	H(2)	0.981	C(12)	H(20)	0.980
C(6)	H(3)	0.980			

Distances are in angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

Table 5. Positional Parameters and B(eq) Values for **10**

atom	x	y	z	B(eq), $Å^2$
S(1)	0.9800(1)	0.23253(3)	0.3644(2)	4.77(2)
S(2)	1.1610(1)	0.17143(3)	0.6433(2)	4.66(2)
O(1)	0.9120(3)	0.09405(6)	0.0134(5)	3.77(6)
O(2)	1.2014(3)	0.01312(8)	0.3818(6)	3.95(7)
N(1)	0.9546(3)	0.15253(8)	0.2640(5)	2.67(6)
C(1)	1.0337(4)	0.18000(10)	0.4202(7)	3.33(8)
C(2)	0.8611(4)	0.2189(1)	0.0934(7)	3.86(9)
C(3)	0.8214(3)	0.17229(9)	0.1421(7)	3.04(7)
C(4)	0.6864(4)	0.16758(10)	0.3214(7)	3.51(8)
C(5)	0.5519(4)	0.1799(1)	0.161(1)	5.3(1)
C(6)	0.6671(4)	0.1238(1)	0.4394(8)	4.4(1)
C(7)	0.9852(3)	0.10927(9)	0.1869(7)	2.73(7)
C(8)	1.1070(3)	0.08483(10)	0.3171(7)	2.89(7)
C(9)	1.2506(4)	0.0964(1)	0.1788(9)	4.56(10)
C(10)	1.0760(3)	0.03660(10)	0.2918(6)	2.90(7)
C(11)	0.9457(4)	0.0216(1)	0.4517(7)	3.77(9)
C(12)	0.9050(4)	-0.0245(1)	0.397(1)	6.0(1)

The form of the equivalent isotropic thermal parameter is:

$$Beq = \frac{8}{3}\pi^2 \sum\sum_{ij} U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

Table 6. Anisotropic Displacement Parameters for **10**

atom	U11	U22	U33	U12	U13	U23
S(1)	0.0974(8)	0.0299(4)	0.0538(6)	-0.0111(5)	-0.0025(7)	-0.0005(5)
S(2)	0.0653(7)	0.0575(6)	0.0541(6)	-0.0127(5)	-0.0149(7)	-0.0098(6)
O(1)	0.054(2)	0.035(1)	0.054(2)	0.010(1)	-0.019(1)	-0.012(1)
O(2)	0.050(2)	0.058(2)	0.042(2)	0.027(1)	-0.002(1)	0.000(1)
N(1)	0.037(2)	0.031(1)	0.034(1)	0.000(1)	-0.002(1)	-0.001(1)
C(1)	0.047(2)	0.037(2)	0.042(2)	-0.005(2)	0.010(2)	-0.003(2)
C(2)	0.068(3)	0.037(2)	0.042(2)	0.006(2)	0.003(2)	0.006(2)
C(3)	0.047(2)	0.032(2)	0.036(2)	0.007(2)	0.001(2)	0.001(2)
C(4)	0.047(2)	0.036(2)	0.050(2)	0.007(2)	0.003(2)	-0.001(2)
C(5)	0.052(2)	0.062(2)	0.089(3)	0.016(2)	-0.002(3)	0.007(3)
C(6)	0.052(3)	0.053(2)	0.063(3)	0.001(2)	0.009(2)	0.002(2)
C(7)	0.036(2)	0.031(2)	0.037(2)	0.002(2)	0.004(2)	-0.002(2)
C(8)	0.034(2)	0.039(2)	0.037(2)	0.004(1)	-0.004(2)	-0.004(2)
C(9)	0.039(2)	0.058(2)	0.076(3)	0.003(2)	0.001(2)	0.006(2)
C(10)	0.041(2)	0.035(2)	0.035(2)	0.014(2)	-0.003(2)	0.000(2)
C(11)	0.050(2)	0.039(2)	0.055(2)	0.006(2)	0.002(2)	0.003(2)
C(12)	0.065(3)	0.040(2)	0.122(4)	-0.003(2)	-0.002(3)	0.010(3)

The form of the anisotropic thermal ellipsoid is:

$$\exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2\,U_{12}hka^*b^* + 2\,U_{13}hla^*c^* + 2\,U_{23}klb^*c^*)\}$$

Table 7. Positional Parameters for the Hydrogen Atoms of **10**

atom	x	y	z	B, Å^2
H(1)	0.7548	0.1159	0.5387	5.3
H(2)	0.5828	0.1239	0.5590	5.3
H(3)	0.6507	0.1030	0.2969	5.3
H(4)	0.5360	0.1588	0.0204	6.4
H(5)	0.4667	0.1808	0.2775	6.4
H(6)	0.5669	0.2081	0.0808	6.4
H(7)	1.2584	0.1275	0.1635	5.5
H(8)	1.3327	0.0855	0.2830	5.5
H(9)	1.2526	0.0836	0.0013	5.5
H(10)	0.7738	0.2369	0.0946	4.6
H(11)	0.9118	0.2221	-0.0768	4.6
H(12)	0.8019	0.1584	-0.0287	3.7
H(13)	0.6960	0.1880	0.4677	4.2
H(14)	1.1127	0.0926	0.5049	3.5
H(15)	1.0593	0.0299	0.1045	3.5
H(16)	0.9686	0.0245	0.6408	4.5
H(17)	0.8621	0.0398	0.4080	4.5
H(18)	0.8889	-0.0284	0.2064	7.2
H(19)	0.8156	-0.0317	0.4937	7.2
H(20)	0.9842	-0.0433	0.4557	7.2
H(21)	1.238(4)	0.005(1)	0.257(6)	3.5(9)*

*Refined isotropically