crystal structural studies on (a) sulfamides, (b) sulfamates and (c) sulfonamides are the following: (a) Trueblood & Mayer (1956); Jordan, Smith, Lohr & Lipscomb (1963); Atwood, Cowley, Hunter & Mehrotra (1982); (b) Morris, Kennard, Hall, Smith & White (1982, 1983); Manickkavachagam & Rajaram (1984); (c) Singh, Tiwari & Singh (1985); Rambaud, Maury, Pauvert, Berge, Audran, Lassere & Declercq (1985).

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(3*S*,4*S*)-4-(2-Methoxycarbonylethyl)-3-methoxycarbonylmethyl-3-methyl-2,5-dithioxopyrrolidine

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Abstract. $C_{12}H_{17}NO_4S_2$, $M_r = 303{\cdot}39$, monoclinic, $P2_1$, $a = 7{\cdot}257\,(1)$, $b = 12{\cdot}178\,(2)$, $c = 8{\cdot}650\,(1)$ Å, $\beta = 99{\cdot}70\,(1)°$, $V = 753{\cdot}4$ Å^3, $D_x = 1{\cdot}34$ Mg m^{-3}, $Z = 2$, $\bar{\lambda}$(Cu $K\alpha$) = 1·54178 Å, $\mu = 3{\cdot}24$ mm^{-1}, $F(000) = 320$, $T = 293$ K. Final $R = 0{\cdot}036$ for 1054 observed reflections [$F_o > 4\sigma(F_o)$] and 179 parameters. The absolute configuration was established by η refinement, $\eta = 1{\cdot}01\,(11)$. The 2,5-dithioxopyrrolidine ring is crystallographically novel and exhibits approximate C_2 symmetry through N. The C=S bond lengths are identical at 1·630 (4) Å, with C—N at 1·360 (5), 1·367 (5) Å. Molecules are linked by O(1)···H(1^i)—N(1^i) [(i): $2-x$, $0{\cdot}5+y$, $-z$] hydrogen bonds with O1···N(1^i) = 2·899 (6), O(1)···H(1^i) = 2·04 (4) Å and O(1)···H(1^i)—N(1^i) = 159 (3)°.

Experimental. The dithioimide (I) was prepared from the corresponding imide by treatment with Lawesson's reagent (Scheibye, Pedersen & Lawesson, 1978). A pale yellow plate of size 0·08 × 0·28 × 0·36 mm was selected for the analysis. Data collection on a Nicolet *R3mμ* diffractometer with graphite-monochromatized Cu $K\alpha$ radiation and ω–2θ scan range from 0·9° below α_1 to 0·9° above α_2. Cell dimensions obtained by a least-squares procedure based on setting angles for 25 centred reflections with $50 \le 2\theta \le 60°$. Intensities of 1228 reflections were measured within $5 \le 2\theta \le 116°$ (h 0/8, k 0/14, l −10/10). Three check reflections (040, 002, 22$\bar{1}$) measured in every 100 showed no significant variation in net intensity. Of 1083 unique reflections ($R_{int} = 0{\cdot}015$) 1056 with $F_o > 4\sigma(F_o)$ were used in the analysis. An empirical absorption correction was applied, based on an ellipsoid model and 400 azimuthal scan data; maximum and minimum transmission factors 0·47 and 0·35. Structure solved by direct methods and refined by blocked-cascade least squares based on F. Non-hydrogen atoms were refined with anisotropic thermal parameters; H atoms riding on C with C—H = 0·96 Å and separate isotropic U's for different H types; H1(N) located on difference Fourier map and x, y, z, U refined freely. Two reflections (121, 020) affected by extinction omitted in final cycles. For 179 parameters $R = 0{\cdot}036$, $wR = 0{\cdot}051$ (observed data), $R = 0{\cdot}038$, $wR = 0{\cdot}052$ (all data), $w^{-1} =$

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Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	U_{eq}*
C(1)	9907 (5)	1690 (3)	−1025 (4)	41 (1)
C(2)	8530 (5)	2422†	−2065 (4)	39 (1)
C(3)	7684 (5)	3347 (3)	−1233 (4)	44 (1)
C(4)	6012 (5)	2968 (4)	−534 (5)	49 (1)
C(5)	5365 (5)	3776 (3)	555 (5)	44 (1)
C(6)	3041 (7)	4126 (5)	2110 (6)	74 (2)
C(7)	11108 (5)	1864 (4)	−3316 (5)	47 (1)
C(8)	9613 (5)	2738 (3)	−3412 (4)	41 (1)
C(9)	8357 (6)	2784 (4)	−5020 (4)	49 (1)
C(10)	7397 (5)	1717 (4)	−5536 (4)	44 (1)
C(11)	5186 (6)	882 (4)	−7481 (5)	64 (2)
C(12)	10632 (6)	3842 (3)	−3102 (5)	53 (1)
N(1)	11200 (4)	1355 (3)	−1906 (4)	47 (1)
S(1)	9928 (1)	1332 (2)	795 (1)	62 (1)
S(2)	12500 (2)	1613 (3)	−4570 (2)	79 (1)
O(1)	6185 (5)	4566 (3)	1080 (4)	76 (1)
O(2)	3706 (4)	3481 (3)	911 (3)	55 (1)
O(3)	7710 (4)	855 (3)	−4893 (3)	59 (1)
O(4)	6136 (3)	1869 (3)	−6821 (3)	55 (1)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.
† Fixed origin on 2_1 axis.

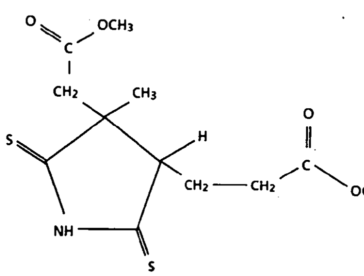
Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

C(1)–C(2)	1·516 (4)	C(1)–N(1)	1·367 (5)
C(1)–S(1)	1·631 (4)	C(2)–C(3)	1·521 (5)
C(2)–C(8)	1·559 (5)	C(3)–C(4)	1·515 (6)
C(4)–C(5)	1·492 (6)	C(5)–O(1)	1·181 (5)
C(5)–O(2)	1·341 (5)	C(6)–O(2)	1·447 (7)
C(7)–C(8)	1·512 (5)	C(7)–N(1)	1·360 (5)
C(7)–S(2)	1·631 (4)	C(8)–C(9)	1·531 (5)
C(8)–C(12)	1·536 (6)	C(9)–C(10)	1·506 (6)
C(10)–O(3)	1·191 (5)	C(10)–O(4)	1·328 (4)
C(11)–O(4)	1·453 (6)		

C(2)–C(1)–N(1)	106·9 (3)	C(2)–C(1)–S(1)	128·9 (3)
N(1)–C(1)–S(1)	124·2 (3)	C(1)–C(2)–C(3)	115·6 (3)
C(1)–C(2)–C(8)	102·9 (3)	C(3)–C(2)–C(8)	117·7 (2)
C(2)–C(3)–C(4)	112·2 (3)	C(3)–C(4)–C(5)	114·1 (4)
C(4)–C(5)–O(1)	126·1 (4)	C(4)–C(5)–O(2)	111·0 (3)
O(1)–C(5)–O(2)	122·9 (4)	C(8)–C(7)–N(1)	107·2 (3)
C(8)–C(7)–S(2)	127·9 (3)	N(1)–C(7)–S(2)	124·7 (3)
C(2)–C(8)–C(7)	103·4 (3)	C(2)–C(8)–C(9)	113·0 (3)
C(7)–C(8)–C(9)	112·8 (3)	C(2)–C(8)–C(12)	112·0 (3)
C(7)–C(8)–C(12)	106·6 (3)	C(9)–C(8)–C(12)	108·8 (3)
C(8)–C(9)–C(10)	114·4 (3)	C(9)–C(10)–O(3)	125·4 (3)
C(9)–C(10)–O(4)	110·3 (3)	O(3)–C(10)–O(4)	124·3 (4)
C(1)–N(1)–C(7)	115·4 (3)	C(5)–O(2)–C(6)	116·1 (3)
C(10)–O(4)–C(11)	115·4 (3)		

C(1)–N(1)–C(7)–C(8)	9·4 (4)	C(8)–C(2)–C(1)–N(1)	19·5 (4)
N(1)–C(7)–C(8)–C(2)	4·0 (4)	C(2)–C(1)–N(1)–C(7)	−17·9 (4)
C(7)–C(8)–C(2)–C(1)	−15·0 (4)		

$[\sigma^2(F_o) + 0.0005F_o^2]$, $S = 1.97$; $\Delta/\sigma = 0.31$ (max) and 0·06 (mean). $\Delta\rho$ within +0·26 and −0·19 e Å^{-3}. Refinement of the Rogers (1981) η parameter [η = 1·01 (11)] shows conclusively that the enantiomer depicted in Fig. 1 represents the correct absolute configuration. The *SHELXTL* program system (Sheldrick, 1983) was used throughout with scattering factors taken from *International Tables for X-ray Crystallography* (1974).



(I)

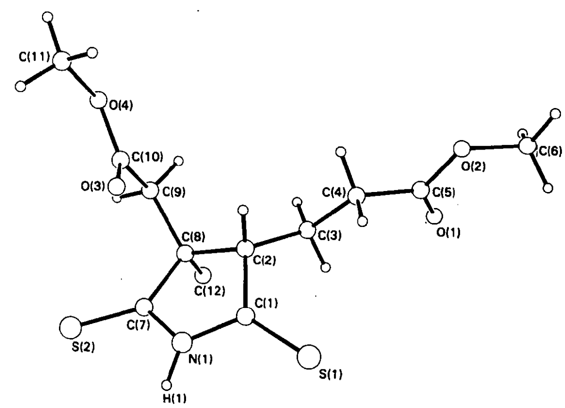


Fig. 1. A perspective view of the molecule showing atomic nomenclature.

Final atomic parameters are listed in Table 1* and molecular geometry in Table 2. The molecular structure and atomic numbering are illustrated in Fig. 1.

The compound was synthesized as part of a study of the conformation of thiazolium rings and their analogues.

* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51522 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester, CH1 2HU, England.

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