listed in Table 1,* and bond lengths and angles are in Table 2.

Related literature. The crystal structure analysis was undertaken to assist in the characterization of the products of the reactions of dialkyl fumarates with amino heterocycles, in which the amino group is attached at the C atom adjacent to a ring N atom (Acheson & Elmore, 1978; Polanc & Colnar, 1984).

The financial support of the Research Community of Slovenia is gratefully acknowledged.

References

Acheson, R. M. & Elmore, V. F. (1978). *Adv. Heterocycl. Chem.* **23**, 263–482.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Polanc, S. & Colnar, C. (1984). *Vestn. Slov. Kem. Drus.* **31**, 115–128.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86*. *Crystallographic Computing* 3, pp. 175–189. Oxford Univ. Press.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected torsion angles and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44240 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Product Predicted with the Felkin–Anh Model

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Abstract. (*Z*)-2-*O*-Benzyl-5,6-*O*-isopropylidene-*N*-methyl-3-*S*-phenyl-3-thio-D-*erythro*-hex-2-enonamide, $C_{23}H_{27}NO_5S$, $M_r = 429{\cdot}54$, monoclinic, $P2_1$, $a = 12{\cdot}524\,(4)$, $b = 7{\cdot}938\,(2)$, $c = 11{\cdot}933\,(2)$ Å, $\beta = 108{\cdot}82\,(2)°$, $V = 1122{\cdot}90\,(96)$ Å^3, $Z = 2$, $D_m = 1{\cdot}28$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}795$ mm^{-1}, $F(000) = 456$, $T = 295$ K, $R = 0{\cdot}039$ for 1379 observed unique reflections. The stereochemistry of the title compound is that predicted with the Felkin–Anh model [Anh (1980). *Top. Curr. Chem.* **88**, 145–166]. Its absolute configuration is inferred from the known configuration of the glyceraldehyde moiety that is introduced into the molecule. Adjacent molecules unite by an intermolecular hydrogen bond between an alcohol function and a carbonyl group, the O···O distance being 2·785 (5) Å.

Experimental. Colorless crystal of dimensions 0·2 × 0·2 × 0·6 mm, D_m by flotation in hexane/methylene chloride. Syntex $P\bar{1}$ diffractometer with incident-beam monochromator, 15 centered reflections within $12{\cdot}5 \le 2\theta \le 25°$ used for determining lattice parameters. Absorption ignored; $(\sin\theta/\lambda)_{\max} = 0{\cdot}557$ Å^{-1}, range of *hkl*: $-13 \le h \le 13$, $0 \le k \le 8$, $-13 \le l \le 13$. Six standard reflections monitored every 100 reflections with random variation of 2·2% over data collection, θ–2θ scans of 1° min^{-1}, 3719 measured reflections, 1758 unique reflections, $R_{\text{int}} = 0{\cdot}039$, 1379 observed $[F_o > 3\sigma(F_o)]$.

Structure solved by direct methods with *MITHRIL* (Gilmore, 1983), *DIRDIF* (Beurskens, 1984) and Fourier procedures. H atoms located in difference maps; constrained to idealized positions with isotropic $B = 1{\cdot}2 \times B$ of bonded atom. H atoms of N and O(1)

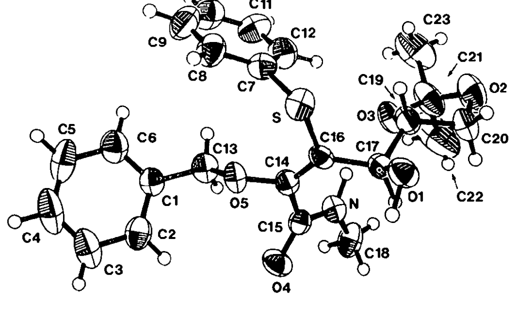

Fig. 1. A drawing of the molecule with thermal ellipsoids scaled at the 50% probability level.

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Table 1. *Positional parameters and equivalent isotropic temperature factors* (*Hamilton,* 1959)

	x	y	z	B_{eq}
S	0·3059 (1)	0·9120	0·4118 (1)	4·39 (6)
O1	0·2822 (3)	1·1345 (5)	0·6071 (3)	4·8 (2)
O2	0·4610 (4)	0·7991 (8)	0·8847 (3)	7·4 (2)
O3	0·3273 (3)	0·7086 (5)	0·7169 (3)	4·6 (2)
O4	−0·0682 (3)	0·7792 (5)	0·4637 (3)	5·1 (2)
O5	0·0810 (3)	0·7534 (4)	0·3322 (3)	4·0 (2)
N	0·0765 (3)	0·6921 (6)	0·6213 (3)	4·0 (2)
C1	−0·0275 (5)	0·5773 (7)	0·1741 (4)	4·1 (2)
C2	−0·1301 (6)	0·6608 (8)	0·1388 (4)	4·9 (3)
C3	−0·1979 (6)	0·656 (1)	0·0229 (5)	6·3 (3)
C4	−0·1666 (8)	0·568 (1)	−0·0580 (5)	7·2 (4)
C5	−0·0650 (8)	0·485 (1)	−0·0253 (5)	6·7 (4)
C6	0·0042 (5)	0·4899 (8)	0·0917 (4)	5·1 (3)
C7	0·3229 (4)	0·7002 (7)	0·3710 (4)	4·0 (2)
C8	0·3101 (6)	0·6637 (9)	0·2550 (5)	5·5 (3)
C9	0·3233 (6)	0·499 (1)	0·2223 (5)	6·3 (3)
C10	0·3488 (6)	0·3738 (8)	0·3057 (7)	6·2 (3)
C11	0·3659 (6)	0·4080 (9)	0·4225 (5)	5·8 (3)
C12	0·3535 (6)	0·5725 (8)	0·4550 (5)	5·2 (3)
C13	0·0452 (5)	0·5806 (7)	0·3010 (4)	4·2 (2)
C14	0·1173 (5)	0·7945 (7)	0·4505 (4)	3·6 (2)
C15	0·0339 (5)	0·7540 (7)	0·5132 (4)	3·7 (2)
C16	0·2123 (4)	0·8800 (6)	0·4959 (4)	3·4 (2)
C17	0·2520 (4)	0·9632 (7)	0·6165 (4)	3·8 (2)
C18	0·0095 (6)	0·6479 (8)	0·6974 (5)	5·4 (3)
C19	0·3565 (5)	0·8780 (7)	0·6963 (4)	4·3 (2)
C20	0·4038 (5)	0·943 (1)	0·8214 (5)	6·2 (3)
C21	0·3946 (6)	0·656 (1)	0·8329 (5)	6·1 (3)
C22	0·3172 (8)	0·608 (2)	0·8978 (6)	13·5 (7)
C23	0·4713 (8)	0·517 (1)	0·8253 (7)	8·9 (5)

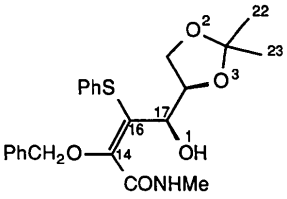
Table 2. *Bond lengths* (Å) *and bond angles* (°)

S—C7	1·782 (5)	C3—C4	1·35 (1)
S—C16	1·790 (5)	C4—C5	1·37 (1)
O1—C17	1·426 (6)	C5—C6	1·386 (8)
O2—C21	1·423 (8)	C7—C8	1·372 (7)
O2—C20	1·430 (8)	C7—C12	1·390 (7)
O3—C21	1·431 (6)	C8—C9	1·389 (9)
O3—C19	1·436 (6)	C9—C10	1·369 (9)
O4—C15	1·240 (6)	C10—C11	1·368 (9)
O5—C14	1·376 (5)	C11—C12	1·385 (8)
O5—C13	1·454 (6)	C14—C16	1·324 (6)
N—C15	1·322 (6)	C14—C15	1·502 (8)
N—C18	1·464 (7)	C16—C17	1·514 (6)
C1—C6	1·362 (7)	C17—C19	1·508 (7)
C1—C2	1·385 (8)	C19—C20	1·509 (7)
C1—C13	1·495 (7)	C21—C22	1·47 (1)
C2—C3	1·371 (8)	C21—C23	1·49 (1)

C7—S—C16	100·2 (2)	C16—C14—O5	119·8 (5)
C21—O2—C20	106·5 (4)	C16—C14—C15	125·7 (4)
C21—O3—C19	109·5 (5)	O5—C14—C15	114·0 (4)
C14—O5—C13	117·3 (4)	O4—C15—N	123·7 (5)
C15—N—C18	124·3 (5)	O4—C15—C14	120·3 (5)
C6—C1—C2	118·8 (5)	N—C15—C14	116·0 (5)
C6—C1—C13	121·1 (6)	C14—C16—C17	126·0 (5)
C2—C1—C13	120·1 (5)	C14—C16—S	120·5 (4)
C3—C2—C1	120·3 (6)	C17—C16—S	113·4 (4)
C4—C3—C2	120·7 (7)	O1—C17—C19	106·2 (4)
C3—C4—C5	120·1 (6)	O1—C17—C16	110·8 (4)
C4—C5—C6	119·5 (6)	C19—C17—C16	110·9 (4)
C1—C6—C5	120·6 (6)	O3—C19—C17	108·1 (5)
C8—C7—C12	119·3 (5)	O3—C19—C20	101·1 (5)
C8—C7—S	119·3 (4)	C17—C19—C20	117·0 (5)
C12—C7—S	121·3 (4)	O2—C20—C19	102·6 (5)
C7—C8—C9	119·8 (6)	O2—C21—O3	105·6 (5)
C10—C9—C8	120·0 (5)	O2—C21—C22	111·8 (7)
C11—C10—C9	121·3 (6)	O2—C21—C23	108·7 (6)
C10—C11—C12	118·5 (6)	O3—C21—C22	107·6 (6)
C11—C12—C7	121·0 (5)	O3—C21—C23	110·3 (6)
O5—C13—C1	108·2 (4)	C22—C21—C23	112·7 (8)

constrained to positions found on difference map and isotropic $B = 1{\cdot}2 \times B$ of N or O(1). $\sum w(F_o - F_c)^2$ minimized where $w = 1/\sigma^2(F_o)$. 271 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, $(\Delta/\sigma)_{\max} = 0{\cdot}07$, $R = 0{\cdot}039$, $wR = 0{\cdot}044$, $S = 1{\cdot}24$. Final difference electron density excursions −0·22 and 0·19 e Å^{-3}. The enantiomorphic form of the molecule is deduced from correlations in the synthetic procedure. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) and programs used were those from the *Texray* Crystallographic Software Package (Molecular Structure Corporation, 1985). Atom numbering for Tables 1 and 2 (atom coordinates, and bond distances and bond angles, respectively) follows that shown in Fig. 1.*

Related literature. For preparation of the title compound (I) see Pallenberg & White (1986); it represents a stage in the synthesis and determination of the absolute configuration of (+)-leptosphaerin, a metabolite of the marine ascomycete *Leptosphaeria oraemaris*. For a structure of the latter see Schiehser, White, Matsumoto, Pezzanite & Clardy (1986).



(I)

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References

Anh, N. T. (1980). *Top. Curr. Chem.* **88**, 145–166.

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures. An Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Tech. Rep. 1. Crystallography Laboratory, Toernooiveld, 6525 Ed Nijmegen, The Netherlands.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and intermolecular distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44232 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Gilmore, G. J. (1983). *MITHRIL. A Computer Program for the Automatic Solution of Crystal Structures from X-ray Data.* Univ. of Glasgow, Scotland.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Molecular Structure Corporation (1985). *TEXSAN. Structure Analysis Package.* MSC, 3304 Longmuire Drive, College Station, TX 77840, USA.

Pallenberg, A. J. & White, J. D. (1986). *Tetrahedron Lett.* **27**, 5591–5594.

Schiehser, G. A., White, J. D., Matsumoto, G., Pezzanite, J. O. & Clardy, J. (1986). *Tetrahedron Lett.* **27**, 5587–5590.

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Cyhalothric Acid*

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Abstract. $C_9H_{10}ClF_3O_2$, $M_r = 242{\cdot}6$, monoclinic, $P2_1/n$, $a = 9{\cdot}519\,(4)$, $b = 8{\cdot}017\,(1)$, $c = 14{\cdot}131\,(3)$ Å, $\beta = 95{\cdot}65\,(5)°$, $V = 1073\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}51\,(1)$, $D_x = 1{\cdot}502$ Mg m^{-3}, $\lambda(\text{Mo }K\bar{\alpha}) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}321$ mm^{-1}, $F(000) = 496$, $T = 295\,(2)$ K, $R = 0{\cdot}061$ for 1442 reflections with $I \geq 2{\cdot}5\sigma(I)$. In the title compound the carboxylic acid and substituted propylene functions are *cis* to each other [torsion angle C(3)—C(4)—C(5)—C(6): 3·0 (6)°] and the configuration about C(2)=C(3) is *Z*. In the crystal lattice, centrosymmetrically related molecules associate *via* hydrogen-bonding contacts involving the carboxylic acid groups: O(2)—H(4)···O(1′) 0·86 (6), 1·80 (6) Å and 172 (4)°.

Experimental. Crude cyhalothric acid was obtained from ICI Australia Operations Pty Ltd (Deer Park, Victoria, Australia). The observation of nine distinct resonances in the ^{13}C NMR spectra of the bulk sample and of the recrystallized material is consistent with the presence of only one isomer of cyhalothric acid. Crystals suitable for X-ray analysis (m.p. 383–386 K) were grown by the slow evaporation of an ammonium hydroxide solution of the acid under a stream of air. Density measured by flotation. Enraf–Nonius CAD-4*F* diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega{:}2\theta$ scan technique. Cell parameters on crystal 0·16 × 0·40 × 0·40 mm from least-squares procedure (De Boer & Duisenberg, 1984) on 25 reflections ($11 \leq \theta \leq 16°$). No absorption correction. Total of 2168 reflections ($1 \leq \theta \leq 25{\cdot}0°$) measured in the range $-11 \leq h \leq 11$, $-9 \leq k \leq 0$, $-16 \leq l \leq 1$. No significant variation in the intensities of three standards (423, 333, 431) monitored every 3600 s. 1899 unique reflections ($R_{\text{int}} = 0{\cdot}040$), 1442 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984), full-matrix least-squares refinement of 158 parameters based on *F* (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, methyl-group H atoms included in the model at their calculated positions and remaining H atoms located from difference map and refined. At convergence $R = 0{\cdot}061$, $wR = 0{\cdot}069$, $w = 18{\cdot}4/[\sigma^2(F) + 0{\cdot}0001F^2]$, $S = 17$, $(\Delta/\sigma)_{\max} \leq 0{\cdot}001$, $\Delta\rho_{\max} = 0{\cdot}33$, $\Delta\rho_{\min} = -0{\cdot}42$ e Å^{-3}; no extinction correction. It should be noted that the structure determination of this sample comprised three data collections and refinements and that in each case unsatisfactorily high values for *k*, in the weighting scheme, and for *S* were obtained. The best model of the three analyses is presented here. Scattering factors for all atoms given in *SHELX*76 (Sheldrick, 1976), all calculations on

Table 1. *Fractional atomic coordinates and* B_{eq} *values* (Å^2)

$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	*y*	*z*	B_{eq}
Cl(1)	0·0287 (1)	−0·1199 (2)	0·9049 (1)	5·87
F(1)	0·2535 (3)	0·0185 (5)	0·7059 (2)	7·04
F(2)	0·2311 (3)	0·1354 (4)	0·8391 (3)	7·15
F(3)	0·3167 (3)	−0·1078 (4)	0·8355 (2)	6·99
O(1)	−0·0276 (3)	−0·3109 (4)	0·5405 (2)	4·12
O(2)	−0·1449 (3)	−0·5424 (4)	0·5706 (2)	4·34
C(1)	0·2185 (4)	−0·0073 (6)	0·7927 (3)	4·73
C(2)	0·0731 (4)	−0·0782 (5)	0·7923 (3)	3·79
C(3)	−0·0129 (4)	−0·1032 (5)	0·7147 (3)	3·64
C(4)	−0·1586 (4)	−0·1640 (5)	0·7117 (3)	3·44
C(5)	−0·2132 (4)	−0·3014 (5)	0·6425 (3)	3·31
C(6)	−0·1195 (4)	−0·3830 (5)	0·5797 (3)	3·35
C(7)	−0·2686 (4)	−0·1253 (5)	0·6297 (3)	3·58
C(8)	−0·4185 (4)	−0·1038 (6)	0·6556 (4)	5·05
C(9)	−0·2319 (5)	−0·0189 (6)	0·5478 (3)	4·82

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* 3-(2-Chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylic acid.

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