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.

Acta Cryst. (1987). C**43**, 2459–2460

Cyhalothric Acid*

BY ERNST HORN, MICHAEL R. SNOW AND EDWARD R. T. TIEKINK

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

(*Received* 8 *June* 1987; *accepted* 15 *July* 1987)

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}, λ(Mo $K\bar{\alpha}$) = 0·7107 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; ω:2θ 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 ($R_{int} = 0{\cdot}040$), 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)

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

	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

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

0108-2701/87/122459-02$01.50

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

C(1)–F(1)	1·318 (5)	C(1)–F(2)	1·318 (5)
C(1)–F(3)	1·333 (5)	C(1)–C(2)	1·496 (6)
C(2)–Cl(1)	1·719 (4)	C(2)–C(3)	1·317 (5)
C(3)–C(4)	1·467 (5)	C(4)–C(5)	1·530 (5)
C(4)–C(7)	1·515 (5)	C(5)–C(6)	1·472 (5)
C(5)–C(7)	1·511 (6)	C(6)–O(1)	1·226 (4)
C(6)–O(2)	1·305 (5)	C(7)–C(8)	1·517 (5)
C(7)–C(9)	1·506 (6)		

F(1)–C(1)–F(2)	107·9 (4)	F(1)–C(1)–F(3)	106·7 (4)
F(2)–C(1)–F(3)	105·9 (4)	F(1)–C(1)–C(2)	112·0 (3)
F(2)–C(1)–C(2)	111·7 (4)	F(3)–C(1)–C(2)	112·3 (4)
Cl(1)–C(2)–C(1)	112·5 (3)	Cl(1)–C(2)–C(3)	123·5 (3)
C(1)–C(2)–C(3)	124·1 (4)	C(2)–C(3)–C(4)	125·7 (4)
C(3)–C(4)–C(5)	121·3 (3)	C(3)–C(4)–C(7)	122·3 (3)
C(5)–C(4)–C(7)	59·5 (2)	C(4)–C(5)–C(6)	121·2 (3)
C(4)–C(5)–C(7)	59·7 (3)	C(7)–C(5)–C(6)	124·6 (4)
C(5)–C(6)–O(1)	124·4 (4)	C(5)–C(6)–O(2)	112·2 (4)
O(1)–C(6)–O(2)	123·4 (4)	C(4)–C(7)–C(5)	60·7 (2)
C(4)–C(7)–C(8)	115·8 (3)	C(4)–C(7)–C(9)	120·6 (3)
C(5)–C(7)–C(8)	113·7 (3)	C(5)–C(7)–C(9)	121·1 (3)
C(8)–C(7)–C(9)	114·6 (3)		

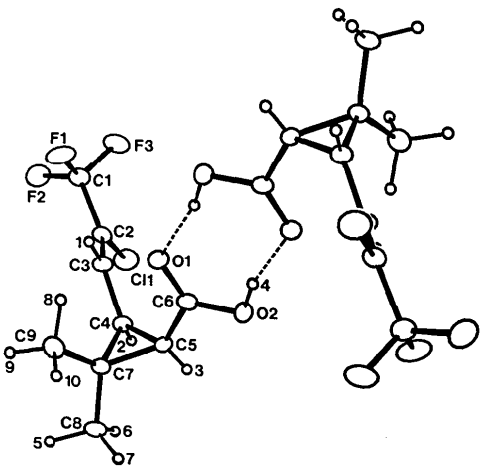

Fig. 1. Crystallographic numbering scheme for cyhalothric acid showing the association between centrosymmetrically related molecules (*ORTEP*, Johnson, 1971). Atoms otherwise not indicated are H atoms.

laboratory μ-VAX computer system. Atomic parameters given in Table 1, selected bond distances and angles in Table 2,* the numbering scheme used is shown in Fig. 1.

Related literature. Cyhalothric acid is an intermediate in the industrial preparation of the cyano(3-phenoxyphenyl)methyl ester of cyhalothric acid, cyhalothrin, one of the synthetic pyrethrins. The spectral and X-ray analyses reported here have shown that the *cis*-cyhalothric acid used in this preparation exists exclusively as the *Z* isomer.

The Australian Research Grants Scheme is thanked for support and Dr E. J. McGarry (ICI) is thanked for the gift of cyhalothric acid.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44260 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

DE BOER, J. L. & DUISENBERG, A. J. M. (1984). Enraf–Nonius CAD-4*F* diffractometer software update February 1984. Groningen and Utrecht, The Netherlands.

GILMORE, C. J. (1984). *J. Appl. Cryst.* 17, 42–46.

JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1987). C**43**, 2460–2462

Structure of *N*-[2-(Nitrooxy)ethyl]nicotinamide (SG 75)

BY Y. NAWATA,* N. TERAO, T. TERAZONO, K. IGUSA, Y. YUTANI AND K. OCHI

Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, *Japan*

(*Received* 18 *June* 1987; *accepted* 31 *July* 1987)

Abstract. $C_8H_9N_3O_4$, $M_r = 211{\cdot}18$, monoclinic, $P2_1/a$, $a = 9{\cdot}519$ (1), $b = 19{\cdot}498$ (2), $c = 5{\cdot}230$ (1) Å, $\beta = 102{\cdot}10(2)°$, $V = 954{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}478$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 9{\cdot}9$ cm^{-1}, $F(000) = 440$, $T = 298$ K, final $R = 0{\cdot}052$ for 1327 unique reflections [$F_o^2 > 2{\cdot}0\,\sigma(F_o^2)$]. The SG 75 molecule adopts a folded conformation, which is stabilized by intramolecular van der Waals contacts between the carbonyl O atom and the nitro group. Intermolecular H bonds and short contacts are observed.

* To whom all correspondence should be addressed.

0108-2701/87/122460-03$01.50