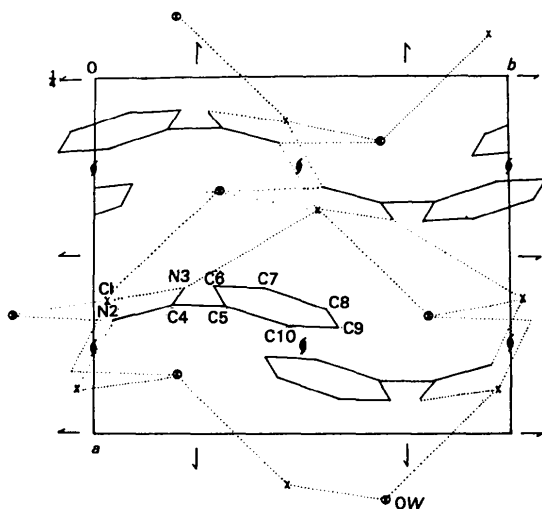


Table 2. *Hydrogen-bond geometry (with e.s.d.'s in parentheses)*

	Symmetry code	$D\cdots A$ (Å)	$D-H$ (Å)	$H\cdots A$ (Å)	$D-H\cdots A$ (°)	$H-D\cdots A$ (°)
N(3)—H(32)⋯Cl	(i)	3.237 (5)	1.19 (5)	2.16 (3)	148 (4)	21 (3)
N(2)—H(22)⋯Cl	(ii)	3.244 (5)	1.01 (5)	2.25 (5)	167 (4)	9 (2)
O(W)—H(W1)⋯Cl	(iii)	3.226 (5)	1.08 (8)	2.17 (10)	163 (6)	11 (5)
O(W)—H(W2)⋯Cl	(iv)	3.218 (5)	0.91 (10)	2.37 (9)	156 (7)	17 (6)
N(3)—H(31)⋯Cl	(v)	3.237 (6)	1.05 (5)	2.20 (5)	170 (4)	7 (3)
N(2)—H(21)⋯O(W)	(vi)	2.840 (6)	0.93 (6)	1.95 (6)	158 (4)	15 (4)

Symmetry code: (i) x, y, z ; (ii) $\frac{1}{2} - x, \bar{y}, \frac{1}{2} + z$; (iii) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$; (iv) $\frac{1}{2} + x, \frac{1}{2} - y, 2 - z$; (v) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (vi) $x - \frac{1}{2}, \frac{1}{2} - y, 2 - z$.

Fig. 2. Packing of the molecule viewed down the c axis.

Packing of the molecule viewed down the c axis is shown in Fig. 2. All the protons available from the amino groups and the water molecule take part in hydrogen bonding. Chlorine is involved in five hydrogen bonds, three $N-H\cdots Cl$ and two $O(W)-H\cdots Cl$ type. The sixth bond is between the water oxygen and N(2). The hydrogen-bond geometry is given in Table 2.

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References

- BEYLER, S. A. & ZANEVELD, L. J. D. (1982). *J. Reprod. Fertil.* **66**(2), 425–431.
- CHANDRASEKAR, K., PATTABHI, V. & RAGHUNATHAN, S. (1982). *Acta Cryst.* **B38**, 2538–2540.
- DEWAR, M. J. S. & SCHMEISING, H. N. (1960). *Tetrahedron*, **11**, 96–120.
- DINIZ, C. R., PEREIRA, A. A., BARROSO, J. & MARES-GUIA, M. (1965). *Biochem. Biophys. Res. Commun.* **21**(5), 448–453.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975). *Acta Cryst.* **B31**, 221–234.
- GANTZEL, K. N., SPARKS, R. A. & TRUEBLOOD, K. N. (1961). *UCLALS1*. Univ. of California.
- HUGHES, E. W. (1941). *J. Am. Chem. Soc.* **63**, 1737–1752.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MARKWARDT, F., LANDMANN, H. & WALSMANN, P. (1968). *Eur. J. Biochem.* **6**(4), 502–506.
- SALUNKE, D. M. & VIJAYAN, M. (1982). *Acta Cryst.* **B38**, 1328–1330.
- THAILAMBAL, V. G., PATTABHI, V., LEE, F. L., LE PAGE, Y. & GABE, E. J. (1985). *Acta Cryst.* **C41**, 105–107.

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(±)-E-6,6,6-Trichloro-3-methoxy-5-methyl-2-hexenoic Acid

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Abstract. $C_8H_{11}Cl_3O_3$, $M_r = 261.52$, triclinic, $P\bar{1}$, $a = 5.926$ (1), $b = 9.641$ (2), $c = 11.254$ (3) Å, $\alpha = 69.57$ (2), $\beta = 80.14$ (2), $\gamma = 75.48$ (2)°, $V = 580.8$ (6) Å³, $Z = 2$, $D_x = 1.50$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 7.7$ cm⁻¹, $F(000) = 268$, $T = 295$ K. Final $R = 0.0601$ for 1336 unique observed reflections.

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The compound was prepared by synthesis as an intermediate for the construction of halogenated marine natural products. A least-squares plane through OCCCCO shows the α,β -unsaturated carboxylic acid to be essentially planar. The carboxylic acid functionality lies near a crystallographic inversion center and is hydrogen bonded to a carboxylic acid group of an adjacent molecule.

Table 1. Atom coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

	x	y	z	U_{eq}^*
C(1)	7330 (8)	4701 (4)	1276 (4)	47 (2)
C(2)	5373 (7)	4402 (4)	2212 (4)	46 (2)
C(3)	4347 (7)	5221 (4)	3004 (4)	44 (2)
C(4)	5014 (7)	6570 (4)	3076 (4)	45 (2)
C(5)	3183 (8)	8023 (4)	2518 (4)	50 (2)
C(6)	3419 (8)	9316 (5)	2924 (5)	55 (2)
C(7)	1473 (10)	3674 (5)	3913 (5)	66 (2)
C(8)	3210 (13)	8432 (6)	1103 (5)	81 (3)
Cl(1)	6264 (2)	9727 (2)	2504 (1)	78 (1)
Cl(2)	2890 (3)	8878 (2)	4610 (1)	83 (1)
Cl(3)	1359 (3)	10981 (1)	2260 (2)	87 (1)
O(1)	8443 (6)	5700 (4)	1087 (3)	68 (2)
O(2)	7879 (6)	3790 (4)	594 (3)	63 (1)
O(3)	2517 (5)	4898 (3)	3851 (3)	56 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Bond lengths (\AA) and angles ($^\circ$)

C(1)–C(2)	1.440 (6)	C(1)–O(1)	1.240 (7)
C(1)–O(2)	1.307 (6)	C(2)–C(3)	1.361 (6)
C(3)–C(4)	1.481 (7)	C(3)–O(3)	1.338 (5)
C(4)–C(5)	1.547 (5)	C(5)–C(6)	1.513 (8)
C(5)–C(8)	1.498 (7)	C(6)–Cl(1)	1.776 (5)
C(6)–Cl(2)	1.783 (6)	C(6)–Cl(3)	1.766 (5)
C(7)–O(3)	1.440 (7)		
C(2)–C(1)–O(1)	126.3 (5)	C(2)–C(1)–O(2)	113.0 (4)
O(1)–C(1)–O(2)	120.7 (4)	C(1)–C(2)–C(3)	125.7 (5)
C(2)–C(3)–C(4)	127.1 (4)	C(2)–C(3)–O(3)	123.2 (4)
C(4)–C(3)–O(3)	109.7 (4)	C(3)–C(4)–C(5)	111.1 (4)
C(4)–C(5)–C(6)	111.8 (4)	C(4)–C(5)–C(8)	111.8 (4)
C(6)–C(5)–C(8)	112.9 (4)	C(5)–C(6)–Cl(1)	112.6 (3)
C(5)–C(6)–Cl(2)	110.9 (3)	Cl(1)–C(6)–Cl(2)	106.5 (3)
C(5)–C(6)–Cl(3)	111.6 (4)	Cl(1)–C(6)–Cl(3)	108.5 (2)
Cl(2)–C(6)–Cl(3)	106.5 (3)	C(3)–O(3)–C(7)	119.9 (4)

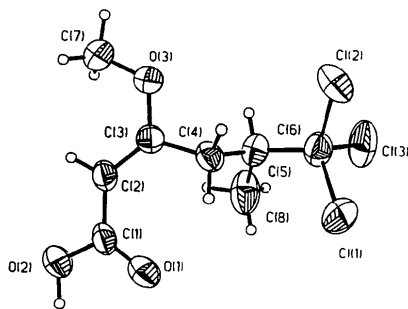


Fig. 1. Molecular structure and numbering scheme.

Introduction. The title compound was prepared as part of a total synthesis of the novel marine natural product, dysidin (Williard & de Laszlo, 1984; Hofheinz & Oberhansli, 1977). An X-ray crystal-structure determination of this substance was undertaken to define unambiguously all aspects of its stereochemistry.

Experimental. Clear, colorless crystal of the carboxylic acid (0.30 \times 0.20 \times 0.35 mm), m.p. 409–410 K, aligned on Nicolet R3m/E diffractometer with an Mo target, normal focus tube, a graphite monochromator and 0.5 mm pinhole collimator. Lattice parameters calculated from 25 reflections with $29 \leq 2\theta \leq 31^\circ$. No systematic absences found. Total of 1819 reflections measured over a period of 33 h utilizing $\theta/2\theta$ scan technique within ranges $3.5 \leq 2\theta \leq 45^\circ$, $0 \leq h \leq 7$, $-11 \leq k \leq 11$, $-13 \leq l \leq 13$. Intensities of three standard reflections (223, 023, 036) recorded after every 45 reflections. Total of 98 standard reflections decreasing linearly by 2.3%, used to correct remaining 1336 unique, observed reflections [$F_o > 2.5\sigma(F_o)$]. Data corrected for Lorentz and polarization effects. No absorbance correction applied. All non-H atoms immediately located by direct-methods structure solution *SHELXTL* (Nicolet, 1982). Remaining 11 H atoms located in subsequent difference Fourier syntheses. Structure refined by block-diagonal least squares with anisotropic thermal parameters for all atoms except H to minimize $\sum w(|F_c - F_o|^2) / \sum F_o^2$. H-atom thermal parameters taken as 1.2 times equivalent isotropic parameter of the bonded atom. H-atom position parameters fixed in calculated positions in final stages of refinement. Final $R = 0.0601$ and $wR = 0.0960$ for 160 variables with $w = 1/[\sigma(F_o)^2 + 0.0001(F_o)^2]$ and $S = 1.26$. Maximum $\Delta/\sigma = 0.14$ in final refinement cycle. Residual electron density in final Fourier map concentrated in positions near the trichloromethyl group indicative of a rotamer; otherwise no significant peaks. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All computer programs belong to the *SHELXTL* 4.0 system (Nicolet, 1982).

Discussion. Final positional and isotropic thermal parameters for the non-H atoms are given in Table 1 and the bond lengths and angles are given in Table 2.* The corresponding e.s.d.'s for the least-significant digits are shown in parentheses. A computer-generated plot in Fig. 1 shows the molecular structure and the numbering scheme.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42653 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

A least-squares plane [$3.494x - 2.667y + 6.700z = 2.1565$] through the six atoms O(1), O(2), C(3), C(4), C(5) and O(3) reveals that the α,β -unsaturated carboxylic acid is essentially planar. Two features of this structure are somewhat curious: the C(7) methyl carbon is nearly in the plane (-0.0002 \AA) described above and also the double bond between C(2) and C(3) is *cis* to the shorter C—O carboxylic acid bond, *i.e.* C(1)—O(1).

The carboxyl end of this structure is close to a crystallographic inversion center so that a hydrogen-bonded dimer is a more accurate representation of the solid.

All C—C bond lengths are equivalent to one another within experimental error and these lengths agree well with what is expected (Kennard, 1962). From the final residual electron density map it is clear that there is some disorder in the crystal due to different rotamers of the trichloromethyl group. We believe that this fact accounts for the slightly high final wR and residual electron density.

A view of the packing is given in Fig. 2. Intermolecular hydrogen bonds holding two asymmetric units together are shown with dashed lines. No other unusual short intermolecular contacts were noted.

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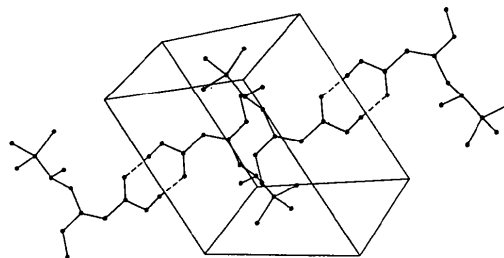


Fig. 2. A view of the packing.

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References

- HOFHEINZ, W. & OBERHÄNSLI, W. E. (1977). *Helv. Chim. Acta*, **60**, 660–669.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- KENNARD, O. (1962). In *International Tables for X-ray Crystallography*. Vol. III, pp. 275–276. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- Nicolet (1982). *SHELXTL*. Nicolet XRD Corporation, Madison, WI 53711, USA.
- WILLIARD, P. G. & DE LASZLO, S. E. (1984). *J. Org. Chem.* **49**, 3489–3493.

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Structure of 5-Fluoroarabinosylcytosine

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Abstract. $C_9H_{12}FN_3O_5$, $M_r = 261.2$, orthorhombic, $P2_12_12_1$, $a = 5.927(2)$, $b = 7.882(1)$, $c = 23.069(4) \text{ \AA}$, $U = 1077.7 \text{ \AA}^3$, $Z = 4$, $D_x = 1.61 \text{ g cm}^{-3}$, $Mo K\alpha$, $\lambda = 0.71073 \text{ \AA}$, $\mu = 1.35 \text{ cm}^{-1}$, $F(000) = 544$, $T = 296 \text{ K}$, $R = 0.030$ for 1077 unique observed reflections. The *N*-glycosidic torsion angle, χ ,

is $164.0(2)^\circ$, in the *anti* range; the sugar pucker is 2E , $P = 165.4(2)^\circ$, $\psi_m = 37.3(2)^\circ$; the C(4')—C(5') conformation is *ap* with $\gamma = 173.8(2)^\circ$. These are typical values for arabinosyl nucleosides. The *ap* conformation about the C(4')—C(5') bond precludes the formation of an intramolecular hydrogen bond between O(5') and