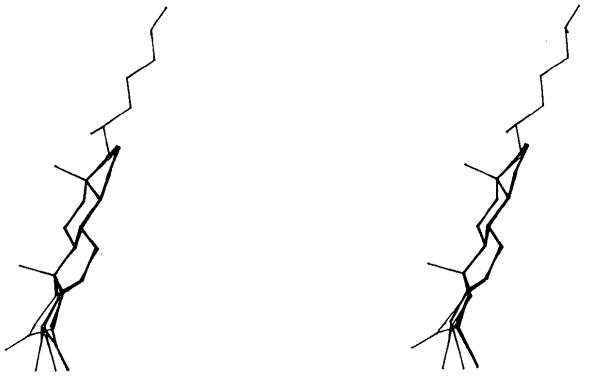

Fig. 3. The superposition of the present molecule (darker line) and the ANDEDP10, HMANDR and MANDIA10 (lighter line) molecules. Atoms C5 through C17 were used to superimpose the molecules.

Ferguson, Marsh, Midgley & Whalley, 1978). The superposition of these three molecules and the title molecule is shown in Fig. 3. Atoms C5 through C17 were used in the program *FITMOL* (Rohrer & Smith, 1980) to superimpose the four molecules. The *B*, *C* and *D* rings of the molecules fit one another very closely but the *A* rings adopt different conformations, ranging from chair to boat.

The side chain at C17 belongs to the most populated conformer *A* (Duax, Griffin, Rohrer & Weeks, 1980).

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References

ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
CARRELL, H. L., GLUSKER, J. P., COVEY, D. F., BATZOLD, F. H. & ROBINSON, C. H. (1978). *J. Am. Chem. Soc.* **100**, 4282–4289.
COX, P. J., MKANDAWIRE, G. J. & MALLISON, P. R. (1981). *Acta Cryst.* B**37**, 727–729.
DUAX, W. L., GRIFFIN, J. F., ROHRER, D. C. & WEEKS, C. M. (1980). *Lipids*, **15**, 783–792.
DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1, p. 18. New York: Plenum.
FERGUSON, G., MARSH, W. C., MIDGLEY, J. M. & WHALLEY, W. B. (1978). *J. Chem. Soc. Perkin Trans.* pp. 272–276.
GRIFFIN, J. F., DUAX, W. L. & WEEKS, G. M. (1984). *Atlas of Steroid Structure*, Vol. 2, p. 21. New York: Plenum.
JOHNSON, C. K. (1970). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
MOTHERWELL, W. D. S. (1976). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.
ROHRER, D. C. & SMITH, G. D. (1980). *FITMOL*. In *PROPHET Molecules*, edited by W. RINDONE & A. KUSH. Cambridge, MA: Bolt Beranek and Newman.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). In *Crystallographic Computing 3*, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

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Structure of an Ascochlorin Derivative (AS-6)

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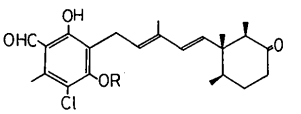
Abstract. 2-Chloro-4-formyl-5-hydroxy-3-methyl-6-{3-methyl-5-[(1*R*,2*S*,6*R*)-1,2,6-trimethyl-3-oxocyclohexyl]-(2*E*,4*E*)-2,4-pentadien-1-yl}phenoxyacetic acid, AS-6 (1), $C_{25}H_{31}ClO_6$, $M_r = 462{\cdot}98$, triclinic, $P\bar{1}$, $a = 11{\cdot}8423\ (7)$, $b = 12{\cdot}9348\ (10)$, $c = 8{\cdot}3598\ (9)$ Å, $\alpha = 103{\cdot}49\ (1)$, $\beta = 101{\cdot}11\ (1)$, $\gamma = 87{\cdot}02\ (1)°$, $V = 1221{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}258$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 16{\cdot}878$ cm^{-1}, $F(000) = 492$, $T = 298$ K, final $R = 0{\cdot}059$ for 3978 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. The asymmetric unit contains two AS-6 molecules, of which conformations are pseudo-mirror symmetric to each other. The molecules are held together by hydrogen bonds between the carboxy groups to form a dimer.

Experimental. Colorless plates of title compound were grown from benzene/cyclohexane (56:44 *v*/*v*) solution. Crystal size 0·50 × 0·45 × 0·13 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$



(1) R= CH_2COOH
(2) R= H

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
Cl	0·799	0·753	0·176	5·27 (4)
C(1)	0·6143 (5)	0·5453 (5)	0·3074 (7)	3·7 (1)
C(2)	0·5026 (5)	0·5886 (5)	0·2747 (7)	3·8 (1)
C(3)	0·4804 (5)	0·6809 (5)	0·2143 (7)	3·8 (1)
C(4)	0·5753 (5)	0·7290 (5)	0·1818 (7)	3·6 (1)
C(5)	0·6871 (5)	0·6856 (4)	0·2172 (7)	3·3 (1)
C(6)	0·7065 (5)	0·5954 (5)	0·2748 (7)	4·0 (1)
C(7)	0·6279 (7)	0·4487 (5)	0·3664 (9)	5·2 (2)
O(8)	0·5499 (6)	0·4013 (5)	0·3931 (7)	8·1 (2)
O(9)	0·4130 (4)	0·5432 (4)	0·3014 (6)	5·4 (1)
O(10)	0·5606 (4)	0·8154 (3)	0·1145 (6)	4·8 (1)
C(11)	0·5848 (7)	0·9155 (5)	0·2290 (9)	4·8 (2)
C(12)	0·8275 (6)	0·5488 (6)	0·3089 (9)	5·2 (2)
C(13)	0·3622 (5)	0·7227 (6)	0·1720 (8)	4·6 (2)
C(14)	0·3162 (5)	0·7889 (6)	0·3208 (8)	4·7 (2)
C(15)	0·2186 (5)	0·7723 (5)	0·3689 (7)	3·5 (1)
C(16)	0·1821 (5)	0·8503 (6)	0·5070 (7)	4·1 (1)
C(17)	0·0862 (5)	0·8468 (5)	0·5649 (7)	3·6 (1)
C(18)	0·0406 (5)	0·9255 (5)	0·7037 (8)	3·8 (1)
C(19)	−0·0756 (6)	0·9689 (6)	0·6330 (8)	4·9 (2)
C(20)	−0·1277 (7)	1·0403 (8)	0·771 (1)	7·7 (2)
C(21)	−0·1469 (7)	0·9853 (9)	0·907 (1)	7·2 (2)
C(22)	−0·0428 (7)	0·9251 (8)	0·9620 (8)	6·7 (2)
C(23)	0·0217 (5)	0·8588 (6)	0·8280 (7)	4·6 (1)
C(24)	0·1410 (6)	0·6751 (7)	0·2895 (9)	6·3 (2)
C(25)	0·1255 (6)	1·0128 (6)	0·7886 (9)	5·0 (2)
C(26)	−0·0676 (9)	1·0345 (8)	0·503 (1)	9·8 (3)
O(27)	−0·0099 (6)	0·9239 (7)	1·1082 (7)	9·5 (2)
C(28)	0·1298 (8)	0·8128 (7)	0·9125 (9)	6·8 (2)
C(29)	0·5919 (6)	0·9964 (5)	0·1301 (9)	4·9 (2)
O(30)	0·6324 (5)	1·0871 (4)	0·2278 (7)	6·8 (1)
O(31)	0·5668 (5)	0·9785 (4)	−0·0204 (6)	6·4 (1)
Cl*	−0·5877 (2)	0·4522 (2)	0·8488 (2)	6·14 (4)
C(1*)	−0·4050 (5)	0·6591 (5)	0·7177 (7)	3·8 (1)
C(2*)	−0·2939 (5)	0·6163 (6)	0·7491 (7)	3·9 (1)
C(3*)	−0·2741 (5)	0·5256 (5)	0·8102 (7)	3·7 (1)
C(4*)	−0·3673 (5)	0·4781 (5)	0·8369 (7)	3·6 (1)
C(5*)	−0·4783 (5)	0·5158 (6)	0·8034 (8)	4·2 (1)
C(6*)	−0·5021 (5)	0·6090 (5)	0·7462 (7)	3·5 (1)
C(7*)	−0·4223 (7)	0·7578 (7)	0·6563 (9)	5·8 (2)
O(8*)	−0·3429 (5)	0·8018 (4)	0·6191 (6)	6·0 (1)
O(9*)	−0·2013 (4)	0·6642 (4)	0·7190 (6)	5·4 (1)
O(10*)	−0·3454 (5)	0·3912 (4)	0·9085 (6)	5·2 (1)
C(11*)	−0·3700 (7)	0·2941 (6)	0·8032 (9)	6·7 (2)
C(12*)	−0·6209 (6)	0·6482 (8)	0·7139 (9)	6·4 (2)
C(13*)	−0·1499 (6)	0·4865 (7)	0·8556 (8)	5·4 (2)
C(14*)	−0·1019 (5)	0·4192 (5)	0·7108 (7)	3·8 (1)
C(15*)	−0·0023 (5)	0·4373 (5)	0·6607 (7)	4·3 (1)
C(16*)	0·0308 (5)	0·3564 (5)	0·5214 (8)	3·9 (1)
C(17*)	0·1312 (5)	0·3548 (6)	0·4687 (8)	4·3 (1)
C(18*)	0·1666 (5)	0·2752 (5)	0·3234 (7)	3·7 (1)
C(19*)	0·1902 (6)	0·3388 (5)	0·1946 (8)	4·1 (1)
C(20*)	0·2484 (6)	0·2741 (5)	0·0588 (8)	4·6 (1)
C(21*)	0·3547 (6)	0·2152 (6)	0·127 (1)	6·5 (2)
C(22*)	0·3404 (6)	0·1589 (6)	0·2551 (9)	5·8 (2)
C(23*)	0·2876 (6)	0·2279 (6)	0·4008 (9)	5·3 (2)
C(24*)	0·0692 (6)	0·5291 (6)	0·7393 (9)	4·9 (2)
C(25*)	0·0797 (7)	0·1863 (6)	0·2410 (9)	5·6 (2)
C(26*)	0·0798 (8)	0·3932 (7)	0·120 (1)	6·4 (2)
O(27*)	0·3565 (6)	0·0644 (5)	0·2450 (9)	9·3 (2)
C(28*)	0·2806 (8)	0·1706 (7)	0·534 (1)	6·8 (2)
C(29*)	−0·3832 (6)	0·2129 (6)	0·8948 (9)	5·4 (2)
O(30*)	−0·4222 (5)	0·1230 (4)	0·8067 (6)	6·6 (1)
O(31*)	−0·3501 (6)	0·2303 (5)	1·0494 (7)	7·1 (2)

Table 2. *Selected bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(2)—C(13)	1·483 (9)	C(3*)—C(13*)	1·537 (9)
O(10)—C(11)	1·424 (7)	O(10*)—C(11*)	1·364 (8)
C(11)—C(29)	1·491 (11)	C(11*)—C(29*)	1·467 (13)
C(13)—C(14)	1·515 (9)	C(13*)—C(14*)	1·506 (9)
C(14)—C(15)	1·336 (9)	C(14*)—C(15*)	1·373 (10)
C(15)—C(16)	1·463 (8)	C(15*)—C(16*)	1·473 (8)
C(16)—C(17)	1·324 (9)	C(16*)—C(17*)	1·343 (9)
C(17)—C(18)	1·519 (8)	C(17*)—C(18*)	1·510 (8)
C(29)—O(30)	1·317 (7)	C(29*)—O(30*)	1·246 (9)
C(29)—O(31)	1·205 (8)	C(29*)—O(31*)	1·278 (9)

O(10)—C(11)—C(29)	107·9 (6)	O(10*)—C(11*)—C(29*)	111·7 (6)
C(3)—C(13)—C(14)	114·2 (5)	C(3*)—C(13*)—C(14*)	114·8 (5)
C(13)—C(14)—C(15)	126·4 (6)	C(13*)—C(14*)—C(15*)	126·4 (6)
C(14)—C(15)—C(16)	118·9 (6)	C(14*)—C(15*)—C(16*)	116·5 (6)
C(15)—C(16)—C(17)	125·9 (6)	C(15*)—C(16*)—C(17*)	124·5 (6)
C(16)—C(17)—C(18)	129·2 (5)	C(16*)—C(17*)—C(18*)	126·0 (6)
C(11)—C(29)—O(30)	111·3 (6)	C(11*)—C(29*)—O(30*)	120·5 (7)
C(11)—C(29)—O(31)	123·5 (6)	C(11*)—C(29*)—O(31*)	116·5 (6)
O(30)—C(29)—O(31)	125·1 (7)	O(30*)—C(29*)—O(31*)	122·8 (8)

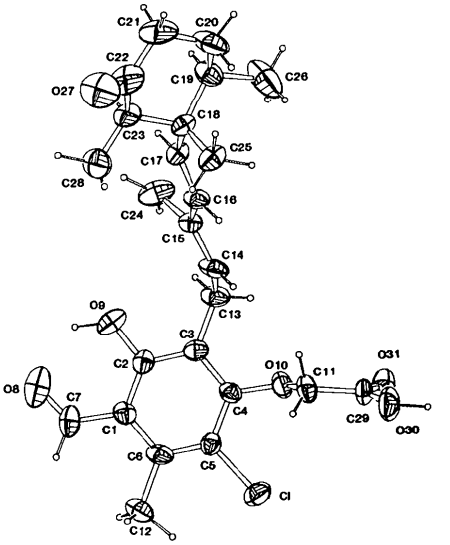

Fig. 1. A perspective view of the molecule with numbering scheme, showing the correct absolute configuration.

radiation, graphite monochromator, θ–2θ scan with scan speed 3·30–5·49° min^{-1} in θ, scan width (0·7 + 0·14tanθ)°. Range of indices, $-14 \le h \le 14$, $-16 \le k \le 16$, $0 \le l \le 10$ ($2\theta < 150°$). Lattice constants determined based on 25 2θ values ($27 < 2\theta < 50°$). Variation of standard < 1·3%; 5037 unique reflections measured; 3978 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. No corrections for absorption. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refined by full-matrix least squares on F. The locations of the carboxyl, hydroxyl and methyl H atoms were found on difference Fourier maps. Those of the other H atoms were calculated stereochemically. Non-H atoms refined with anisotropic thermal parameters. H atoms with the isotropic thermal parameters (B = 5·0 Å^2) were added in the calculation of structure factors. $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1·0$ for $|F_o| < 89·27$, $w = (89·27/F_o)^2$ for $|F_o| \ge 89·27$. Final R = 0·059, wR = 0·60, S = 9·49 for 578 variables, secondary-extinction factor (g) 3·40 (9) × 10^{-6} [$|F_o| = |F_c|/(1 + gIc)$]; $(\Delta/\sigma) <$ 0·46, largest peak in final ΔF map +0·42 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV); programs

used: Enraf–Nonius *SDP* (Frenz, 1980), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, selected bond distances and angles are listed in Table 2.*

Related literature. Ascochlorin (2) is a terpenoid antibiotic (Tamura, Suzuki, Takatsuki, Ando & Arima, 1968), and the structure was elucidated by X-ray analysis (Nawata, Ando, Tamura, Arima & Iitaka, 1969; Nawata & Iitaka, 1971). The title compound is a 4-*O*-carboxymethyl derivative of ascochlorin (Hosokawa, Matsuura, Takahashi, Ando & Tamura, 1990) and is an antidiabetic agent (Hosokawa, Ando & Tamura, 1985).

* Lists of structure factors, anisotropic thermal parameters, bond lengths, bond angles, torsion angles, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53532 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

FRENZ, B. A. (1980). Enraf–Nonius structure determination package. Version 17. College Station, Texas, USA.

HOSOKAWA, T., ANDO, K. & TAMURA, G. (1985). *Diabetes*, **34**, 267–274.

HOSOKAWA, T., MATSUURA, I., TAKAHASHI, H., ANDO, K. & TAMURA, G. (1970). Japanese Patent No. 1556883.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

NAWATA, Y., ANDO, K., TAMURA, G., ARIMA, K. & IITAKA, Y. (1969). *J. Antibiot.* **22**, 511–512.

NAWATA, Y. & IITAKA, Y. (1971). *Bull. Chem. Soc. Jpn*, **44**, 2652–2660.

TAMURA, G., SUZUKI, S., TAKATSUKI, A., ANDO, K. & ARIMA, K. (1968). *J. Antibiot.* **21**, 539–544.

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Triethylammonium 3,3,6,6-tetrathioxocyclodi(phosphadithianate) at 178 K

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Abstract. $[(C_2H_5)_3NH]_2[P_2S_8]$, $M_r = 522{\cdot}9$, monoclinic, $P2_1/c$, $a = 6{\cdot}929\ (3)$, $b = 13{\cdot}157\ (7)$, $c = 13{\cdot}447\ (5)$ Å, $\beta = 98{\cdot}07\ (3)°$, $V = 1213{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}43$ Mg m^{-3}, $F(000) = 520$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}75$ mm^{-1}, $T = 178$ K, $R = 0{\cdot}026$ for 2396 reflections. The anion P_2S_4 ring is exactly centrosymmetric and possesses a chair conformation. The axial P—S bond is appreciably shorter than the equatorial (1·952, 1·987 Å). The anion and cation are connected by a hydrogen bond from the equatorial exocyclic S atom, S3···N 3·30 Å.

Experimental. A colourless prism 0·8 × 0·4 × 0·3 mm, obtained from acetonitrile solution, was mounted in inert oil and transferred to the cold gas stream of the diffractometer (Siemens *R*3 with LT-2 low temperature attachment). 4256 intensities were registered to $2\theta_{max} = 55°$ using monochromated Mo $K\alpha$ radiation (ω scans, width 1·1°, constant speed 7·3° min^{-1}, quadrant $-h+k+l$ and some $-k$ equivalents, index ranges h −8→0, k −16→16, l −17→17). Merging equivalents gave 2779 unique reflections (R_{int} 0·026), of which 2396 with $F > 4\sigma(F)$ were used for all calculations (program system Siemens *SHELXTL-Plus*). Three check reflections showed no significant intensity change. No absorp-

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^4)

	x	y	z	U_{eq}*
N	8372 (2)	1662 (1)	2141 (1)	206 (4)
C(1)	9529 (2)	1810 (1)	1287 (1)	253 (5)
C(2)	11094 (3)	1016 (2)	1251 (1)	346 (6)
C(3)	9511 (3)	1911 (1)	3153 (1)	258 (5)
C(4)	9843 (3)	3034 (1)	3310 (1)	325 (6)
C(5)	7442 (3)	630 (1)	2169 (1)	265 (5)
C(6)	6311 (3)	319 (1)	1170 (1)	335 (6)
P(1)	4783 (1)	3651 (1)	594 (1)	177 (1)
S(1)	2356 (1)	4653 (1)	298 (1)	202 (1)
S(2)	7176 (1)	4655 (1)	1022 (1)	209 (1)
S(3)	4445 (1)	3104 (1)	1933 (1)	244 (1)
S(4)	5089 (1)	2799 (1)	−559 (1)	263 (1)

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

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