

Related literature. The title compound contains four fused heterocyclic ring systems and serves as starting material for new cavitands. The five-membered *cis*-fused imidazolidone rings are distorted envelopes, the puckering parameters (Cremer & Pople, 1975) are $Q = 0.178$ (2) Å and $\varphi = 206.3$ (6)° and the least-squares planes are at an angle of 76.1 (1)°. Each five-membered ring is fused to a six-membered heteroatomic ring that has a chair conformation with puckering parameters $Q = 0.508$ (2) Å, $\theta = 9.9$ (2)° and $\varphi = 3$ (1)°. The molecule has a polar character and the peripheral positions of the O atoms constitute a potential receptor site for e.g. transition metal ammine complexes (Colquhoun, Doughty, Stoddart & Williams, 1984). In the crystal structure there are no intermolecular contacts which are less than the sum of the van der Waals radii.

Acta Cryst. (1990). **C46**, 2486–2487

Structure of Membranolide, a Diterpene from the Antarctic Sponge *Dendrilla membranosa*

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(Received 15 March 1990; accepted 12 April 1990)

Abstract. Methyl 2-[1,3-dihydro-3-oxo-5-(2,3,3-trimethylcyclohexyl)isobenzofuran-4-yl]propionate, $C_{21}H_{28}O_4$, $M_r = 344.45$, orthorhombic, $P2_12_12_1$, $a = 7.040$ (2), $b = 14.238$ (4), $c = 18.810$ (5) Å, $V = 1885.4$ Å³, $Z = 4$, $D_x = 1.214$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.08$ mm⁻¹, $F(000) = 744$, $T = 293$ K, $R = 0.050$ for 1758 unique observed reflections. The structure consists of a cyclohexane ring in the chair conformation which possesses as substituents at C(4) two methyl groups, and at C(10) an equatorial methyl group, and in an axial position an isobenzofuranone ring with an α -(methoxycarbonyl)ethyl side chain. All bond lengths and angles are within the expected ranges.

Experimental. The secondary metabolite membranolide was isolated from an acetonic extract of the Antarctic Sponge *Dendrilla membranosa*. The diter-

References

- COLQUHOUN, H. M., DOUGHTY, S. M., STODDART, J. F. & WILLIAMS, D. J. (1984). *Angew Chem.* **96**, 232–234.
 CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 HIMES, V. L., HUBBARD, C. R., MIGHELL, A. D. & FATIADI, A. J. (1978). *Acta Cryst.* **B34**, 3102–3104.
 NIELE, F. G. M. & NOLTE, R. J. M. (1988). *J. Am. Chem. Soc.* **110**, 172–177.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.
 SPEK, A. L. (1982). *The EUCLID package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.

pene was crystallized from ether/hexane solution. A plate-shaped colourless crystal of dimensions $0.3 \times 0.9 \times 0.2$ mm was used on a Huber four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from 24 centred reflections ($11 < \theta < 16^\circ$). ω scan used for data collection of 1819 unique reflections of which 1758 were observed with $F > 3\sigma(F)$. Three standard reflections ($\bar{3}15$, $1\bar{7}1$, $04\bar{7}$) every 100 measurements show 0.85% variation in intensity. Diffraction intensities were measured up to $\sin \theta/\lambda = 0.70$ Å⁻¹ in the index range $h = 0 \rightarrow 9$, $k = 0 \rightarrow 20$, $l = 0 \rightarrow 26$. Solved by direct phase determination using *SHELXTL-Plus* (Sheldrick, 1987), full-matrix least squares minimized $\sum w(\Delta F)^2$. H-atom positions calculated geometrically and considered isotropically with $U = 1.2U$ of bonded C atom. Positions and thermal parameters of all non-H atoms refined anisotropically giving 227 variables. $R =$

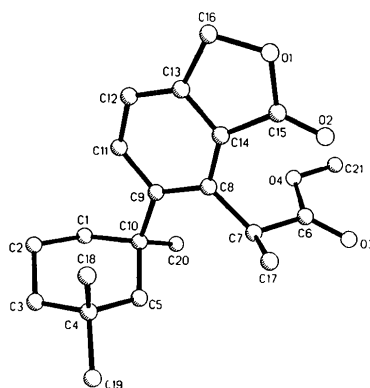


Fig. 1. Perspective drawing of the molecule with the atoms labelled according to Table 1.

0.050, $wR = 0.043$, $S = 1.92$, where $w^{-1} = \sigma^2(F)$. Final $(\Delta/\sigma)_{\max} = 0.002$, $\Delta\rho_{\max} = 0.20$ and $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$ on final difference Fourier map. Atomic scattering factors were taken from *SHELXTL-Plus* (Sheldrick, 1987). A perspective molecular drawing is shown in Fig. 1 with atoms labelled according to the tables. The absolute configuration was not established. The atomic coordinates are given in Table 1.*

Related literature. Studies by Dayton, Robilliard, Paine & Dayton (1974) revealed that the sponge *Dendrilla membranosa* has a chemical defence system. Molinski & Faulkner (1987) reported the isolation as an oil and the spectroscopic identification of membranolide, a metabolite related to the spongian

* Lists of bond lengths and angles, atomic coordinates of H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53186 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	3005 (5)	9054 (2)	7596 (2)	66 (1)
C(1)	9781 (7)	11206 (3)	5229 (2)	52 (2)
O(2)	4281 (5)	7808 (3)	7060 (2)	68 (1)
C(2)	8495 (7)	11840 (3)	4779 (2)	61 (2)
O(3)	7696 (5)	6803 (2)	6320 (1)	67 (1)
C(3)	8050 (8)	11377 (3)	4071 (2)	64 (2)
O(4)	8654 (4)	8059 (2)	6947 (1)	50 (1)
C(4)	7101 (7)	10411 (3)	4168 (2)	50 (2)
C(5)	8392 (6)	9793 (3)	4634 (2)	46 (2)
C(6)	7754 (7)	7634 (3)	6404 (2)	45 (2)
C(7)	6976 (6)	8347 (3)	5872 (2)	39 (2)
C(8)	6553 (6)	9323 (3)	6179 (2)	33 (2)
C(9)	7414 (6)	10159 (3)	5939 (2)	36 (1)
C(10)	9000 (6)	10197 (3)	5376 (2)	41 (2)
C(11)	6850 (7)	11005 (3)	6275 (2)	46 (2)
C(12)	5454 (7)	11060 (3)	6787 (2)	52 (2)
C(13)	4623 (7)	10246 (3)	7006 (2)	45 (2)
C(14)	5173 (6)	9396 (3)	6714 (2)	36 (2)
C(15)	4191 (8)	8645 (4)	7106 (2)	53 (2)
C(16)	3131 (7)	10059 (3)	7556 (2)	65 (2)
C(17)	5305 (7)	7932 (3)	5457 (2)	56 (2)
C(18)	5083 (7)	10509 (3)	4462 (2)	61 (2)
C(19)	6957 (8)	9929 (3)	3439 (2)	79 (2)
C(20)	10742 (6)	9660 (3)	5673 (2)	58 (2)
C(21)	9544 (7)	7448 (3)	7452 (2)	60 (2)

group of diterpenes, that is likely to be a defensive constituent of *Dendrilla membranosa*. For studies of toxicity on this sponge see McClintock (1987).

References

- DAYTON, P. K., ROBILLIARD, G. A., PAINE, R. T. & DAYTON, L. B. (1974). *Ecol. Monogr.* **44**, 105–128.
 MCCLINTOCK, J. B. (1987). *Mar. Biol.* **94**, 479–487.
 MOLINSKI, T. F. & FAULKNER, D. J. (1987). *J. Org. Chem.* **52**, 296–298.
 SHELDRIK, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Acta Cryst. (1990). **C46**, 2487–2489

Structure of 2-(2,4-Dimethoxybenzoyl)furan-3-carboxylic Acid

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(Received 5 April 1990; accepted 29 May 1990)

Abstract. $\text{C}_{14}\text{H}_{12}\text{O}_6$, $M_r = 276.24$, orthorhombic, $P2_12_12_1$, $a = 18.48$ (1), $b = 9.52$ (1), $c = 7.55$ (1) \AA , $V = 1328.27 \text{ \AA}^3$, $Z = 4$, $D_x = 1.380 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha)$

$= 0.7107 \text{ \AA}$, $\mu = 0.69 \text{ cm}^{-1}$, $F(000) = 576$, $T = 290 \text{ K}$, $R = 0.061$ for 1330 unique observed [$I/\sigma(I) > 3.0$] reflexions. Internal hydrogen bonding between

0108-2701/90/122487-03\$03.00

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