$C_7H_9N_2^+.C_{10}H_8NO_4^-$

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

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

	x	y	z	B_{eq}
O(1)	0.1810 (4)	0.1459 (4)	0.5221 (3)	6.6 (1)
O(2)	0.1229 (4)	0.2049 (4)	0.3375 (2)	5.7 (1)
O(3)	0.4900 (4)	−0.1155 (5)	0.7342 (2)	7.2 (1)
O(4)	0.0254 (4)	0.0089 (4)	0.3500 (2)	5.9 (1)
N(1)	−0.0671 (4)	−0.0094 (4)	0.1707 (3)	4.6 (1)
N(2)	−0.0184 (4)	0.2144 (5)	0.1595 (3)	5.3 (1)
N(3)	0.3510 (4)	−0.0089 (5)	0.6250 (3)	4.4 (1)
C(1)	−0.1308 (5)	0.0963 (6)	0.0307 (3)	3.9 (1)
C(2)	−0.1078 (5)	0.1981 (6)	−0.0232 (3)	4.4 (1)
C(3)	−0.1666 (6)	0.1922 (7)	−0.1094 (4)	5.7 (2)
C(4)	−0.2489 (6)	0.0895 (7)	−0.1418 (4)	6.2 (2)
C(5)	−0.2705 (6)	−0.0161 (7)	−0.0888 (4)	5.6 (2)
C(6)	−0.2133 (5)	−0.0109 (6)	−0.0033 (3)	4.9 (2)
C(7)	−0.0695 (5)	0.0982 (5)	0.1235 (3)	4.1 (1)
C(8)	0.3102 (5)	−0.1007 (6)	0.3989 (3)	4.6 (2)
C(9)	0.3981 (5)	−0.2018 (6)	0.4032 (3)	4.9 (2)
C(10)	0.4721 (5)	−0.2398 (6)	0.4812 (4)	5.5 (2)
C(11)	0.4578 (5)	−0.1788 (6)	0.5557 (4)	4.8 (2)
C(12)	0.3705 (5)	−0.0745 (5)	0.5532 (3)	3.8 (1)
C(13)	0.2934 (5)	−0.0345 (5)	0.4727 (3)	3.6 (1)
C(14)	0.1978 (5)	0.0747 (5)	0.4623 (3)	4.4 (1)
C(15)	0.1082 (5)	0.0951 (5)	0.3747 (3)	4.4 (1)
C(16)	0.4099 (5)	−0.0335 (6)	0.7092 (3)	4.7 (2)
C(17)	0.3611 (7)	0.0659 (8)	0.7687 (4)	7.5 (2)

Table 2. Selected geometric parameters (Å, °)

N(1)—C(7)	1.289 (7)	O(4)—C(15)	1.222 (6)
N(2)—C(7)	1.329 (7)	N(3)—C(12)	1.379 (7)
C(1)—C(2)	1.378 (8)	N(3)—C(16)	1.371 (6)
C(1)—C(6)	1.396 (7)	C(8)—C(9)	1.358 (8)
C(1)—C(7)	1.479 (7)	C(8)—C(13)	1.401 (8)
C(2)—C(3)	1.380 (7)	C(9)—C(10)	1.366 (8)
C(3)—C(4)	1.356 (9)	C(10)—C(11)	1.379 (8)
C(4)—C(5)	1.390 (9)	C(11)—C(12)	1.381 (8)
C(5)—C(6)	1.365 (7)	C(12)—C(13)	1.415 (6)
O(1)—C(14)	1.234 (7)	C(13)—C(14)	1.464 (7)
O(2)—C(15)	1.254 (6)	C(14)—C(15)	1.517 (7)
O(3)—C(16)	1.176 (7)	C(16)—C(17)	1.541 (9)
C(2)—C(1)—C(6)	119.1 (5)	N(3)—C(12)—C(11)	123.9 (4)
C(2)—C(1)—C(7)	121.4 (5)	N(3)—C(12)—C(13)	117.6 (5)
C(6)—C(1)—C(7)	119.5 (5)	C(11)—C(12)—C(13)	118.4 (5)
C(1)—C(2)—C(3)	119.6 (5)	C(8)—C(13)—C(12)	118.7 (5)
C(2)—C(3)—C(4)	121.1 (6)	C(8)—C(13)—C(14)	118.1 (4)
C(3)—C(4)—C(5)	120.1 (6)	C(12)—C(13)—C(14)	123.2 (5)
C(4)—C(5)—C(6)	119.2 (5)	O(1)—C(14)—C(13)	123.5 (4)
C(1)—C(6)—C(5)	120.8 (5)	O(1)—C(14)—C(15)	117.9 (5)
N(1)—C(7)—N(2)	119.1 (5)	C(13)—C(14)—C(15)	118.4 (5)
N(1)—C(7)—C(1)	121.6 (4)	O(2)—C(15)—O(4)	126.7 (5)
N(2)—C(7)—C(1)	119.2 (5)	O(2)—C(15)—C(14)	115.4 (4)
C(12)—N(3)—C(16)	128.2 (5)	O(4)—C(15)—C(14)	117.9 (5)
C(9)—C(8)—C(13)	121.5 (5)	O(3)—C(16)—N(3)	125.7 (5)
C(8)—C(9)—C(10)	119.4 (5)	O(3)—C(16)—C(17)	123.4 (5)
C(9)—C(10)—C(11)	121.1 (5)	N(3)—C(16)—C(17)	110.9 (5)
C(10)—C(11)—C(12)	120.9 (5)		

Table 3. Hydrogen bonds (Å)

N(1)···O(4)	2.819 (5)	N(2)···O(4^{ii})	2.868 (6)
N(1)···O(2^i)	2.839 (5)	N(2)···O(2)	2.901 (5)

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $-x, y+\frac{1}{2}, \frac{1}{2}-z$.

The data were corrected for Lorentz and polarization effects. There was no crystal decay and no absorption correction was made. The structure was solved by routine direct methods and refined using a full-matrix least-squares method with non-H atoms anisotropic. H atoms were located on a difference map

and refined with isotropic temperature factors. Programs from the *SDP-Plus* package (B. A. Frenz & Associates, Inc., 1983) were used. All calculations were carried out on a a PDP11/73 computer.

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Salvireptanolide

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Abstract

The whole molecule of (7R,8S,9R)-5,6-*seco-neo*-clerodane-1,3,5(10),13(16),14-pentaene-12-oxo-15,16-epoxy-18,19;20(7)-diolide, $C_{20}H_{18}O_6$, adopts a T-

shaped conformation. The methyl groups on the γ-lactone ring are related synclinally and the crystal packing is entirely the result of van der Waals forces.

Comment

The structure determination of the title compound, (I), was undertaken to establish the absolute configuration and the orientation of the methyl groups as part of the structural study of the diterpenoid constituents isolated from *Salvia reptans*.
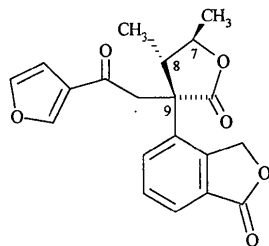
(I)

The methyl groups have a *synclinal* relationship $\{\tau[C(6)—C(7)—C(8)—C(17)] = 88.3\ (4)°\}$, contrary to that expected from NMR evidence (Esquivel, Esquivel, Cárdenas, Sánchez, Ramamoorthy, Toscano & Rodríguez-Hahn, 1991). The saturated γ-lactone ring is in an envelope conformation with C(7) out of the plane. Intra-annular torsion angles of the five-membered ring [starting with C(9)—C(20)—O(1)—C(7) and proceeding around the ring] are −14.6 (4), 25.0 (4), −24.7 (4), 16.3 (4) and −1.6 (4)°. The angle between the least-squares plane through the atoms of the benzofuranone moiety and the plane of the furanylketone is 10.2 (1)°.
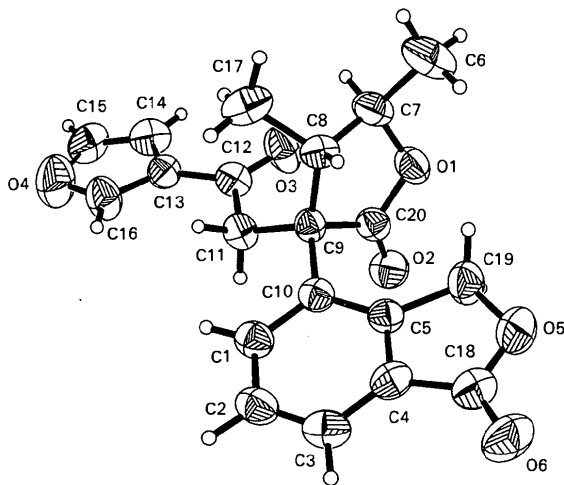

Fig. 1. The molecular structure of the title compound showing the atom labelling and absolute configuration.

Experimental

The compound was obtained from extract of *Salvia reptans*. Crystals were obtained from ethyl acetate solution of the compound.

Crystal data

$C_{20}H_{18}O_6$	Cu $K\alpha$ radiation
$M_r = 354.36$	$\lambda = 1.54178$ Å
Orthorhombic	Cell parameters from 25
$P2_12_12_1$	reflections
$a = 9.439\ (3)$ Å	$\theta = 4.69–16.6°$
$b = 12.979\ (4)$ Å	$\mu = 0.82$ mm^{-1}
$c = 13.773\ (5)$ Å	$T = 298$ K
$V = 1687.2\ (5)$ Å^3	Prismatic
$Z = 4$	$0.4 \times 0.26 \times 0.22$ mm
$D_x = 1.395$ Mg m^{-3}	Colorless

Data collection

Nicolet *P3F* diffractometer	$\theta_{max} = 52.4°$
$2\theta–\theta$ scans	$h = 0 \rightarrow 9$
Absorption correction:	$k = 0 \rightarrow 13$
none	$l = 0 \rightarrow 14$
1261 measured reflections	2 standard reflections
1136 independent reflections	monitored every 50
1115 observed reflections	reflections
$[I > 2.5\sigma(I)]$	intensity variation: ±3%
$R_{int} = 0.035$	

Refinement

Refinement on F	$\Delta\rho_{max} = 0.45$ e Å^{-3}
$R = 0.039$	$\Delta\rho_{min} = -0.43$ e Å^{-3}
$wR = 0.045$	Extinction correction:
$S = 1.08$	Larson (1967)
1115 reflections	Extinction coefficient:
237 parameters	2.68 (4)
H atoms refined using	Atomic scattering factors
a riding model	from *International Tables*
Weights based on counting	*for X-ray Crystallography*
statistics	(1974, Vol. IV)
$(\Delta/\sigma)_{max} = 0.002$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	−0.2724 (3)	0.4931 (2)	0.0559 (2)	0.051 (1)
O(2)	−0.1753 (3)	0.6353 (2)	0.1145 (2)	0.056 (1)
O(3)	−0.3505 (3)	0.6576 (2)	−0.0826 (2)	0.067 (1)
O(4)	−0.2159 (5)	0.8621 (3)	−0.3342 (3)	0.099 (2)
O(5)	0.1348 (3)	0.3710 (2)	0.1994 (2)	0.062 (1)
O(6)	0.3701 (3)	0.3621 (3)	0.2092 (2)	0.071 (1)
C(1)	0.1810 (4)	0.6065 (3)	−0.0504 (3)	0.045 (1)
C(2)	0.3234 (4)	0.5938 (3)	−0.0247 (3)	0.049 (1)
C(3)	0.3624 (4)	0.5279 (3)	0.0482 (3)	0.051 (1)
C(4)	0.2542 (4)	0.4755 (3)	0.0950 (3)	0.044 (1)
C(5)	0.1139 (4)	0.4884 (2)	0.0720 (2)	0.040 (1)
C(6)	−0.3081 (6)	0.3268 (3)	−0.0208 (4)	0.083 (2)
C(7)	−0.2765 (4)	0.4397 (3)	−0.0382 (3)	0.054 (1)
C(8)	−0.1333 (4)	0.4619 (3)	−0.0849 (3)	0.046 (1)
C(9)	−0.0841 (3)	0.5637 (2)	−0.0373 (2)	0.038 (1)
C(10)	0.0731 (3)	0.5546 (2)	−0.0038 (2)	0.039 (1)
C(11)	−0.0998 (4)	0.6602 (3)	−0.0996 (3)	0.044 (1)
C(12)	−0.2511 (4)	0.6891 (2)	−0.1267 (3)	0.044 (1)

C(13)	−0.2667 (4)	0.7607 (3)	−0.2091 (2)	0.044 (1)
C(14)	−0.3919 (5)	0.8088 (3)	−0.2391 (3)	0.059 (1)
C(15)	−0.3614 (5)	0.8683 (3)	−0.3124 (3)	0.066 (2)
C(16)	−0.1638 (5)	0.7963 (3)	−0.2692 (3)	0.064 (1)
C(17)	−0.1416 (5)	0.4602 (4)	−0.1956 (3)	0.065 (1)
C(18)	0.2663 (4)	0.3999 (3)	0.1725 (3)	0.053 (1)
C(19)	0.0294 (4)	0.4215 (3)	0.1415 (3)	0.053 (1)
C(20)	−0.1806 (3)	0.5716 (3)	0.0516 (3)	0.042 (1)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(7)	1.472 (5)	O(1)—C(20)	1.338 (4)	
O(2)—C(20)	1.201 (4)	O(3)—C(12)	1.190 (5)	
O(4)—C(15)	1.408 (7)	O(4)—C(16)	1.333 (6)	
O(5)—C(18)	1.348 (5)	O(5)—C(19)	1.434 (5)	
O(6)—C(18)	1.207 (5)	C(1)—C(2)	1.398 (5)	
C(1)—C(10)	1.379 (5)	C(2)—C(3)	1.371 (5)	
C(3)—C(4)	1.387 (5)	C(4)—C(5)	1.371 (5)	
C(4)—C(18)	1.457 (5)	C(5)—C(10)	1.408 (5)	
C(5)—C(19)	1.520 (5)	C(6)—C(7)	1.515 (6)	
C(7)—C(8)	1.525 (5)	C(8)—C(9)	1.547 (5)	
C(8)—C(17)	1.529 (5)	C(9)—C(10)	1.557 (5)	
C(9)—C(11)	1.527 (5)	C(9)—C(20)	1.530 (5)	
C(11)—C(12)	1.522 (5)	C(12)—C(13)	1.475 (5)	
C(13)—C(14)	1.399 (6)	C(13)—C(16)	1.358 (6)	
C(14)—C(15)	1.305 (6)			
C(7)—O(1)—C(20)	109.7 (3)	C(15)—O(4)—C(16)	104.7 (4)	
C(18)—O(5)—C(19)	111.0 (3)	C(2)—C(1)—C(10)	122.3 (3)	
C(1)—C(2)—C(3)	121.1 (4)	C(2)—C(3)—C(4)	116.7 (3)	
C(3)—C(4)—C(5)	122.9 (3)	C(3)—C(4)—C(18)	127.9 (3)	
C(5)—C(4)—C(18)	109.2 (3)	C(4)—C(5)—C(10)	120.7 (3)	
(4)—C(5)—C(19)	106.9 (3)	C(10)—C(5)—C(19)	132.4 (3)	
O(1)—C(7)—C(6)	108.8 (3)	O(1)—C(7)—C(8)	105.1 (3)	
C(6)—C(7)—C(8)	115.1 (3)	C(7)—C(8)—C(9)	104.3 (3)	
C(7)—C(8)—C(17)	111.9 (3)	C(9)—C(8)—C(17)	116.9 (3)	
C(8)—C(9)—C(10)	110.3 (3)	C(8)—C(9)—C(11)	115.6 (3)	
C(10)—C(9)—C(11)	108.7 (3)	C(8)—C(9)—C(20)	102.6 (3)	
C(10)—C(9)—C(20)	109.5 (3)	C(11)—C(9)—C(20)	109.8 (3)	
C(1)—C(10)—C(5)	116.3 (3)	C(1)—C(10)—C(9)	121.9 (3)	
C(5)—C(10)—C(9)	121.7 (3)	C(9)—C(11)—C(12)	115.6 (3)	
O(3)—C(12)—C(11)	121.9 (3)	O(3)—C(12)—C(13)	122.1 (3)	
C(11)—C(12)—C(13)	116.0 (3)	C(12)—C(13)—C(14)	126.4 (3)	
C(12)—C(13)—C(16)	127.9 (3)	C(14)—C(13)—C(16)	105.7 (3)	
C(13)—C(14)—C(15)	107.9 (4)	O(4)—C(15)—C(14)	110.3 (4)	
O(4)—C(16)—C(13)	111.4 (4)	O(5)—C(18)—O(6)	121.2 (4)	
O(5)—C(18)—C(4)	108.5 (3)	O(6)—C(18)—C(4)	130.3 (4)	
O(5)—C(19)—C(5)	104.4 (3)	O(1)—C(20)—O(2)	121.3 (3)	
O(1)—C(20)—C(9)	111.7 (3)	O(2)—C(20)—C(9)	126.9 (3)	

The absolute configuration was determined by Bijvoet analysis of 20 pairs with the largest Bijvoet ratios $r_{B,calc} = 2(I_{calc+} - I_{calc-})/(I_{calc-})/(I_{calc+} + I_{calc-})$. For each *hkl* all symmetry equivalents were measured with high precision (maximum scan speed $0.25°$ min^{-1}). Bijvoet-equivalent intensities were summed and the sums used to obtain $r_{B,obs}$. Of the 20 pairs, 17 indicated the absolute configuration given in the *Abstract*, while three were ambiguous. An isotropic *U* value, 1.2 times that of the parent C atom, was used for each H atom.

Diagrams and most calculations were performed with the PC version of the *NRCVAX* package (Gabe, Le Page, Charland, Lee & White, 1989). The absolute configuration determined for salvireptanolide is in agreement with that observed in most of the *neo*-clerodane diterpenoids isolated from *Salvia spp.*, following the configuration found in clerodine, in which the methyl group at C(8) is α-equatorial and the atom C(20) α-axial (Rodriguez-Hahn, Esquivel & Cárdenas, 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, Bijvoet difference analysis and most sensitive Friedel pairs have been deposited with the IUCr (Reference: HH1055). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,N'-Bis(methoxycarbonyl)-2,5-dimethylhexane-2,5-diamine

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Abstract

There are two nearly planar NHCOO groups in the title molecule (dimethyl 2,5-dimethyl-2,5-hexanediyl-dicarbamate, $C_{12}H_{24}N_2O_4$). The individual molecules in the crystal are linked by hydrogen bonds.

Comment

The title compound (I) is a product of the reaction of $KAuCl_4$ and 2,5-diisocyano-2,5-dimethylhexane in the presence of MeOH at room temperature. The full characterization of (I) is important for the study of the mechanism of the above reaction (Che, Yip, Wong & Lai, 1992). An X-ray structure determination was therefore undertaken.

(I)
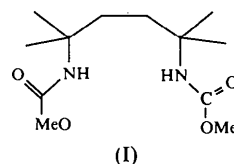
The numbering of the atoms is shown in Fig. 1. The O—C(=O)—N groups are planar to within 0.011 (4) Å. The atoms C(1) and C(8) deviate con-