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 $r_{B,\mathrm{calc}} = 2(I_{\mathrm{calc}+} - I_{\mathrm{calc}-})/(I_{\mathrm{calc}-})/(I_{\mathrm{calc}+} + I_{\mathrm{calc}-})$. 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 $r_{B,\mathrm{obs}}$. 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).

The authors are indebted to the Co-editor Dr Håkon Hope for his help with the absolute-configuration determination.

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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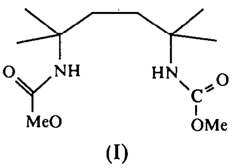
Abstract

There are two nearly planar NHCOO groups in the title molecule (dimethyl 2,5-dimethyl-2,5-hexanediyldicarbamate, $C_{12}H_{24}N_2O_4$). 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 considerably from their associated planes, with out-of-plane distances of 0.061 (4) and 0.109 (4) Å, respectively. The bond distances and angles are close to those observed in other carbamate compounds (Bracher & Small, 1967; Sepehrnia, Ruble & Jeffrey, 1987). The molecules are hydrogen bonded (NH···O=C interactions) as illustrated in Fig. 2. The hydrogen-bond lengths and angles are shown in Table 3.

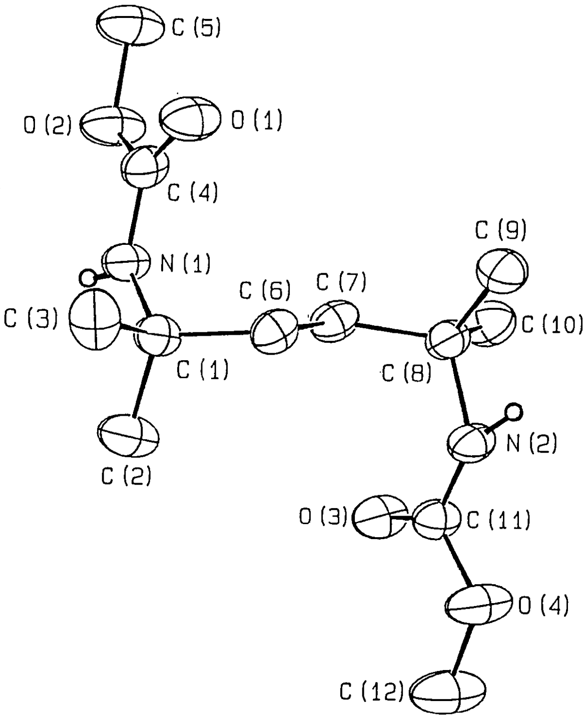


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title molecule showing the atomic numbering scheme.

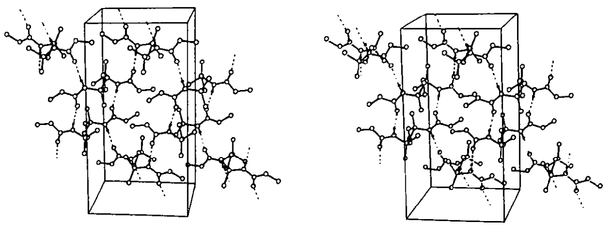


Fig. 2. Stereoview of the packing of the molecules in the unit cell. The hydrogen bonds are drawn as dotted lines. The origin is in the lower back left hand corner, the *b* axis is horizontal and the *c* axis is vertical.

Experimental

Crystal data

$C_{12}H_{24}N_2O_4$
$M_r = 260.33$
Monoclinic
$P2_1/n$
$a = 9.265$ (1) Å
$b = 9.354$ (1) Å
$c = 17.399$ (2) Å
$\beta = 94.39$ (1)°
$V = 1503.5$ (5) Å^3
$Z = 4$
$D_x = 1.150$ Mg m^{-3}
$D_m = 1.151$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{–}15°$
$\mu = 0.08$ mm^{-1}
Prism
0.28 × 0.21 × 0.07 mm
Colourless
Crystal source: recrystallized from methanol

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega\text{–}2\theta$ scans
Absorption correction: none
2316 measured reflections
2104 independent reflections
1067 observed reflections $[F_o > 3\sigma(F_o)]$

$R_{\text{int}} = 0.022$
$\theta_{\max} = 22.5°$
$h = -9 \rightarrow 0$
$k = 0 \rightarrow 10$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity variation: 0.3%

Refinement

Refinement on F
$R = 0.048$
$wR = 0.055$
$S = 1.33$
1067 reflections
169 parameters
Only coordinates of H atoms refined for HN, H-atom parameters not refined for other H atoms

$w = 4F_o^2/[\sigma^2(F_o^2) + (0.055F_o^2)^2]$
$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{\text{eq}} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.4637 (3)	0.4017 (3)	0.6901 (1)	5.47 (6)
O(2)	0.3504 (3)	0.4420 (3)	0.5733 (2)	6.27 (7)
O(3)	0.7705 (3)	−0.1322 (3)	0.5413 (1)	5.38 (6)
O(4)	0.8961 (3)	−0.2645 (3)	0.6313 (1)	6.21 (7)
N(1)	0.3782 (3)	0.2197 (3)	0.6125 (2)	4.27 (7)
N(2)	0.8774 (3)	−0.0343 (3)	0.6515 (2)	4.17 (7)
C(1)	0.4273 (4)	0.0975 (4)	0.6615 (2)	3.99 (8)
C(2)	0.3901 (5)	−0.0373 (4)	0.6141 (2)	6.0 (1)
C(3)	0.3444 (4)	0.0948 (5)	0.7339 (2)	5.8 (1)
C(4)	0.4020 (4)	0.3562 (4)	0.6316 (2)	4.31 (9)
C(5)	0.3718 (5)	0.5928 (4)	0.5841 (3)	7.1 (1)
C(6)	0.5894 (4)	0.1046 (4)	0.6842 (2)	4.00 (8)
C(7)	0.6832 (4)	0.1441 (4)	0.6187 (2)	4.21 (9)
C(8)	0.8456 (4)	0.1188 (4)	0.6360 (2)	4.10 (8)
C(9)	0.9023 (4)	0.1994 (4)	0.7078 (2)	5.4 (1)
C(10)	0.9265 (5)	0.1675 (4)	0.5673 (2)	5.7 (1)
C(11)	0.8407 (4)	−0.1402 (4)	0.6026 (2)	4.20 (9)
C(12)	0.8632 (6)	−0.3912 (4)	0.5877 (3)	7.8 (1)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

O(1)—C(4)	1.207 (4)	N(2)—C(11)	1.333 (4)
O(2)—C(4)	1.352 (4)	C(1)—C(2)	1.531 (5)
O(2)—C(5)	1.435 (5)	C(1)—C(3)	1.527 (5)
O(3)—C(11)	1.208 (5)	C(1)—C(6)	1.525 (5)
O(4)—C(11)	1.351 (4)	C(6)—C(7)	1.530 (5)
O(4)—C(12)	1.428 (5)	C(7)—C(8)	1.530 (5)

N(1)—C(1)	1.476 (4)	C(8)—C(9)	1.518 (5)
N(1)—C(4)	1.333 (5)	C(8)—C(10)	1.528 (5)
N(2)—C(8)	1.483 (5)		
C(4)—O(2)—C(5)	116.6 (3)	O(2)—C(4)—N(1)	109.9 (3)
C(11)—O(4)—C(12)	117.2 (3)	C(1)—C(6)—C(7)	114.9 (3)
C(1)—N(1)—C(4)	124.1 (3)	C(6)—C(7)—C(8)	115.1 (3)
C(8)—N(2)—C(11)	124.3 (3)	N(2)—C(8)—C(7)	111.4 (3)
N(1)—C(1)—C(2)	106.3 (3)	N(2)—C(8)—C(9)	106.1 (3)
N(1)—C(1)—C(3)	109.7 (3)	N(2)—C(8)—C(10)	109.1 (3)
N(1)—C(1)—C(6)	111.7 (3)	C(7)—C(8)—C(9)	111.0 (3)
C(2)—C(1)—C(3)	108.9 (3)	C(7)—C(8)—C(10)	109.5 (3)
C(2)—C(1)—C(6)	110.6 (4)	C(9)—C(8)—C(10)	109.6 (4)
C(3)—C(1)—C(6)	109.6 (3)	O(3)—C(11)—O(4)	123.0 (3)
O(1)—C(4)—O(2)	122.7 (3)	O(3)—C(11)—N(2)	127.8 (3)
O(1)—C(4)—N(1)	127.3 (3)	O(4)—C(11)—N(2)	109.4 (3)
C(5)—O(2)—C(4)—O(1)	−0.2 (5)	C(4)—N(1)—C(1)—C(2)	−174.6 (3)
C(5)—O(2)—C(4)—N(1)	−178.2 (3)	C(4)—N(1)—C(1)—C(3)	67.9 (4)
C(12)—O(4)—C(11)—O(3)	−2.9 (5)	C(4)—N(1)—C(1)—C(6)	−53.8 (4)
C(12)—O(4)—C(11)—N(2)	177.8 (3)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H(N1)···O(3^{i})	0.86 (1)	2.171 (5)	3.027 (5)	172.6 (4)
N(2)—H(N2)···O(1^{ii})	0.85 (1)	2.198 (5)	3.082 (5)	175.1 (4)

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$.

Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares with weighting scheme using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAX computer. Anisotropic displacement parameters were used for all the non-H atoms. At an intermediate stage of refinement, difference Fourier maps showed maxima in positions consistent with the locations of all H atoms. The HN atoms were included in the least-squares refinement while all other H atoms were placed in their idealized positions (C—H 0.96 Å) and included as riding atoms in the structure-factor calculations, but were not refined.

The author thanks the University of Hong Kong for support of this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: HL1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Octahydroacridines

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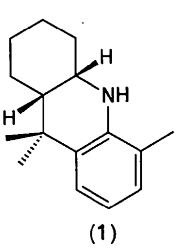
Abstract

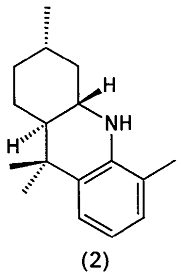
A *cis*-decalin conformation with congestion at the central heterocyclic ring is found for *cis*-5,9,9-trimethyloctahydroacridine, $C_{16}H_{23}N$, and a slightly twisted *trans*-decalin conformation is found for (3*RS*,4a*RS*,9a*SR*)-3,5,9,9-tetramethyloctahydroacridine, $C_{17}H_{25}N$.

Comment

We recently reported the convenient preparation of substituted octahydroacridine derivatives by intramolecular Lewis acid-catalyzed hetero-Diels–Alder reaction of *N*-arylimines, which are tethered to an alkene moiety (Laschat & Lauterwein, 1993). The *cis*/*trans* ratio was found to be mainly dependent on the substituents at C(3) of the octahydroacridine system. Determination of the stereochemistry of the ring fusion by NMR experiments led to the conclusion that the *cis* isomer prefers a *cis*-decalin conformation, whereas the *trans*-decalin conformation is favoured by the *trans* isomer. This assumption is now confirmed by an X-ray crystallographic analysis of *cis*-5,9,9-trimethyloctahydroacridine, (1) (Fig. 1), and (3*RS*,4a*RS*,9a*SR*)-3,5,9,9-tetramethyloctahydroacridine, (2) (Fig. 2). In addition, the relative *cis* configuration between H(3) and H(4*a*) in (2) can be established from the X-ray data.



(1)



(2)

Comparison of the important structural features of (1) and (2) with those of tricarbonyl(*trans*-3,3,5,9,9-pentamethyloctahydroacridine)chromium (Laschat, Noe, Riedel & Krüger, 1993) shows the following differences. The aromatic bond lengths of (1) and (2) are very