C6—C11	1.379 (9)	C19—C20	1.52 (1)
C7—C8	1.371 (8)	C20—C21	1.52 (1)
C8—C9	1.378 (9)	C21—C22	1.41 (1)
C9—C10	1.382 (8)	C22—C23	1.55 (1)
C9—C12	1.447 (8)		
C5—O2—C6	116.9 (7)	N1—C13—C18	132.7 (8)
C12—O3—C14	104.5 (6)	C14—C13—C18	117.8 (9)
C16—O4—C19	120.8 (7)	O3—C14—C13	107.1 (9)
C12—N1—C13	105.2 (7)	O3—C14—C15	126.8 (8)
O1—C5—O2	123.5 (9)	C13—C14—C15	126.1 (9)
O1—C5—C4	126.2 (9)	C14—C15—C16	113.5 (7)
O2—C5—C4	110.3 (8)	O4—C16—C15	116.0 (7)
O2—C6—C7	120.7 (9)	O4—C16—C17	120.5 (9)
O2—C6—C11	117.8 (9)	C15—C16—C17	123.4 (9)
C7—C6—C11	121.4 (8)	C16—C17—C18	120.5 (8)
C6—C7—C8	118.6 (9)	C13—C18—C17	118.7 (7)
C7—C8—C9	120.2 (8)	O4—C19—O5	122 (1)
C8—C9—C10	120.7 (8)	O4—C19—C20	108.9 (9)
C8—C9—C12	119.9 (9)	O5—C19—C20	129 (1)
C10—C9—C12	119.3 (9)	C1—C2—C3	115. (1)
C9—C10—C11	119.6 (8)	C2—C3—C4	111.4 (9)
C6—C11—C10	119.4 (9)	C3—C4—C5	113.1 (8)
O3—C12—N1	113.8 (8)	C19—C20—C21	116.5 (9)
O3—C12—C9	117.9 (8)	C20—C21—C22	111. (1)
N1—C12—C9	128.2 (9)	C21—C22—C23	109. (1)
N1—C13—C14	109.4 (8)		
C6—O2—C5—O1	2.6 (9)	O2—C6—C11—C10	−179.2 (5)
C6—O2—C5—C4	−177.4 (5)	C7—C8—C9—C12	174.7 (5)
C5—O2—C6—C7	70.9 (7)	C12—C9—C10—C11	−174.2 (5)
C5—O2—C6—C11	−110.7 (6)	C8—C9—C12—O3	1.7 (8)
C19—O4—C16—C15	107.6 (6)	C8—C9—C12—N1	−173.4 (6)
C19—O4—C16—C17	−76.6 (7)	C10—C9—C12—O3	177.7 (5)
C16—O4—C19—O5	−1.8 (9)	C10—C9—C12—N1	2.6 (9)
C16—O4—C19—C20	−179.3 (5)	N1—C13—C18—C17	177.5 (6)
C13—N1—C12—C9	174.4 (5)	C14—C15—C16—O4	171.8 (5)
C1—C2—C3—C4	177.7 (7)	O4—C16—C17—C18	−171.5 (5)
C2—C3—C4—C5	174.1 (6)	O4—C19—C20—C21	−161.6 (5)
C3—C4—C5—O1	−7 (1)	O5—C19—C20—C21	21 (1)
C3—C4—C5—O2	173.5 (6)	C19—C20—C21—C22	72.9 (9)
O2—C6—C7—C8	179.6 (5)	C20—C21—C22—C23	175.4 (8)

H atoms were placed at calculated positions [riding model, C—H = 0.95 Å, $U(\mathrm{H}) = U_{\mathrm{eq}}(\mathrm{C})$]. A refinement against 3037 unique reflections having $I > 0$ gave $R = 0.211$, $wR = 0.091$ and $S = 1.305$ (weights as above), without significant changes in the final structural parameters. Program used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Programs used to refine structure: *SDP* (Enraf-Nonius, 1985). Refinement was by full-matrix least-squares method. All programs were run on a Digital MicroVAX computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71446 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1045]

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Acta Cryst. (1994). C**50**, 263–265

1,3,5-Triethyl-1,3,5-triazine-2,4,6(1*H*,3*H*,5*H*)-trione, $C_9H_{15}N_3O_3$

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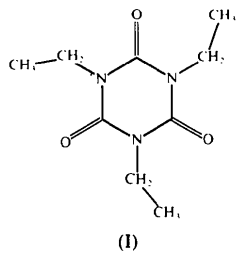
Abstract

The crystal structure of the title compound (triethylisocyanuric acid), which was obtained from the preparation of an ethyl isocyanate derivative of the anti-tumor compound CC-1065, has been determined by X-ray diffraction.

Comment

The title compound (I) was prepared accidentally during the synthesis of a derivative of the known anti-tumor compound CC-1065 (Chidester, Krüger, Mizsak, Duchamp & Martin, 1981). The attempted synthesis of an ethyl isocyanate derivative of CC-1065 yielded the crystalline compound triethylisocyanuric acid. The crystals appeared as clear yellow prisms from a reaction mixture of ethyl isocyanate and pyridine. The perhydrotriazine moiety is essentially planar with the terminal methyl groups lying



(I)

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1.4 Å out of the plane. The molecular packing in the unit cell viewed down the b axis is shown in Fig. 2. The bond angles and distances are in close agreement with those of trimethylisocyanuric acid (Belaj & Nachbaur, 1987).

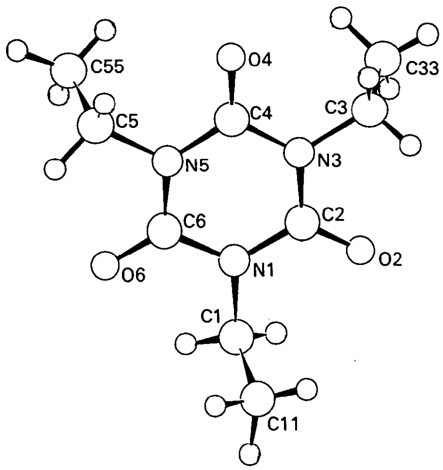

Fig. 1. View of $C_9H_{15}N_3O_3$ showing the labeling of the atoms.

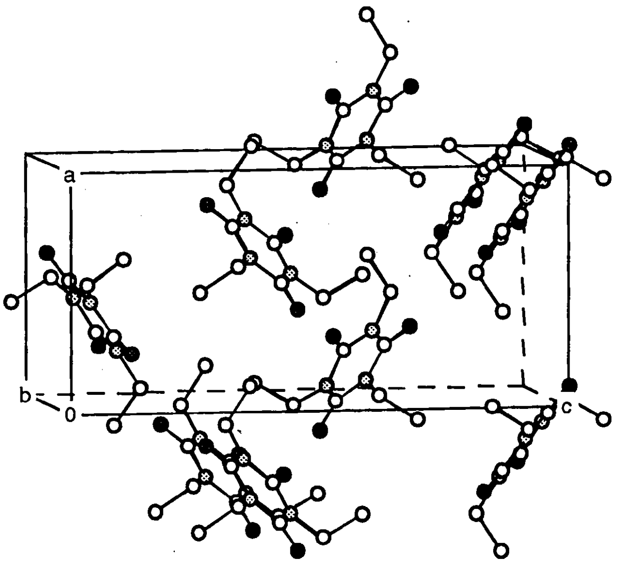

Fig. 2. Packing drawing of the title molecule along the b axis.

Experimental

Crystal data

$C_9H_{15}N_3O_3$
$M_r = 213.24$
Orthorhombic
$P2_12_12_1$
$a = 7.845$ (1) Å
$b = 8.160$ (1) Å
$c = 16.840$ (1) Å
$V = 1078.0$ (1) Å^3
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 60\text{-}69°$
$\mu = 0.748$ mm^{-1}
$T = 123$ K
Thin plate
$Z = 4$
$D_x = 1.31$ Mg m^{-3}
$D_m = 1.25$ Mg m^{-3}
Density measured by flotation in $CsCl_2/H_2O$
$0.50 \times 0.28 \times 0.16$ mm
Clear

Data collection

Siemens $P1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1183 measured reflections
1183 independent reflections
942 observed reflections [$I > 3.0\sigma(I)$]
$\theta_{\max} = 69°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 20$
9 standard reflections monitored every 80 reflections
intensity variation: none

Refinement

Refinement on F^2
$R(F) = 0.035$
$wR(F^2) = 0.078$
$S = 1.57$
1183 reflections
195 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F^2)$
$(\Delta/\sigma)_{\max} = 0.027$
$\Delta\rho_{\max} = 0.172$ e Å^{-3}
$\Delta\rho_{\min} = -0.220$ e Å^{-3}
Atomic scattering factors from Doyle & Turner (1968) (C, N, O) and Stewart, Davidson & Simpson (1965) (H)

Cell refinement: Duchamp (1977). Data reduction: *CRYM* (Duchamp, 1984). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1980). Program(s) used to refine structure: *CRYM*.

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

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

	x	y	z	B_{eq}
N(1)	0.1067 (3)	0.1951 (3)	0.6116 (1)	1.94 (9)
C(1)	0.0574 (5)	0.0214 (4)	0.6212 (2)	2.7 (1)
O(2)	0.3263 (3)	0.1599 (3)	0.6971 (1)	2.90 (9)
C(2)	0.2463 (4)	0.2494 (3)	0.6538 (1)	2.2 (1)
N(3)	0.2890 (3)	0.4127 (3)	0.6441 (1)	1.94 (9)
C(3)	0.4403 (4)	0.4758 (4)	0.6857 (2)	2.5 (1)
O(4)	0.2457 (3)	0.6602 (2)	0.5855 (1)	2.94 (9)
C(4)	0.2024 (4)	0.5188 (4)	0.5940 (1)	2.2 (1)
N(5)	0.0625 (3)	0.4538 (3)	0.5551 (1)	2.05 (9)
C(5)	−0.0273 (4)	0.5627 (4)	0.4988 (2)	2.4 (1)
O(6)	−0.1066 (3)	0.2375 (2)	0.5213 (1)	2.55 (9)
C(6)	0.0114 (4)	0.2915 (3)	0.5595 (1)	2.1 (1)
C(11)	−0.0487 (5)	−0.0049 (5)	0.6958 (2)	3.3 (1)
C(33)	0.5998 (5)	0.4589 (5)	0.6353 (2)	3.4 (2)
C(55)	0.0677 (5)	0.5766 (4)	0.4206 (2)	2.9 (1)

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

N(1)—C(1)	1.478 (4)	C(3)—C(33)	1.518 (5)
N(1)—C(2)	1.378 (4)	O(4)—C(4)	1.211 (4)
N(1)—C(6)	1.396 (4)	C(4)—N(5)	1.383 (4)
C(1)—C(11)	1.523 (5)	N(5)—C(5)	1.478 (4)
O(2)—C(2)	1.208 (3)	N(5)—C(6)	1.386 (4)
C(2)—N(3)	1.384 (4)	C(5)—C(55)	1.518 (4)
N(3)—C(3)	1.471 (4)	O(6)—C(6)	1.210 (3)
N(3)—C(4)	1.386 (4)		
C(1)—N(1)—C(2)	117.4 (2)	N(3)—C(4)—O(4)	121.9 (3)
C(1)—N(1)—C(6)	118.0 (2)	N(3)—C(4)—N(5)	116.0 (2)
C(2)—N(1)—C(6)	124.6 (2)	O(4)—C(4)—N(5)	122.1 (3)
N(1)—C(1)—C(11)	111.7 (3)	C(4)—N(5)—C(5)	116.8 (2)
N(1)—C(2)—O(2)	122.0 (3)	C(4)—N(5)—C(6)	124.8 (2)
N(1)—C(2)—N(3)	116.2 (2)	C(5)—N(5)—C(6)	118.1 (2)

O(2)—C(2)—N(3)	121.9 (3)	N(5)—C(5)—C(55)	111.5 (3)
C(2)—N(3)—C(3)	118.4 (2)	N(1)—C(6)—N(5)	114.6 (2)
C(2)—N(3)—C(4)	123.7 (2)	N(1)—C(6)—O(6)	122.6 (3)
C(3)—N(3)—C(4)	117.8 (2)	N(5)—C(6)—O(6)	122.8 (3)
N(3)—C(3)—C(33)	111.5 (2)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71553 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1063]

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Crystal Studies of Musk Compounds. V.† Structure of 1-(1,2,6,7,8,8a-Hexahydro-3,6,6,8a-tetramethylacenaphthylen-4-yl)ethan-1-one

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Abstract

The acetyl group in the title compound, $C_{18}H_{24}O$, is twisted by 31.6 (1)° out of the plane of the aromatic ring with its O atom facing the neighbouring methyl group. The aromatic ring is essentially planar; due to the strain induced by the fused rings, the non-aromatic six- and five-membered rings adopt deformed half-chair and envelope conformations, respectively.

Comment

This paper reports the only tricyclic structure determined in this series dealing with crystal studies of musk compounds (Part IV: De Ridder & Schenk, 1992).

Recently, Fehr, Galindo, Haubrichs & Perret (1989) used the results of the structure-activity relationship studies of Beets (1978) and those of Theimer & Davies (1967) to construct new strong musks. Fehr and co-workers arrived at the basic musk skeleton given in Fig. 1.

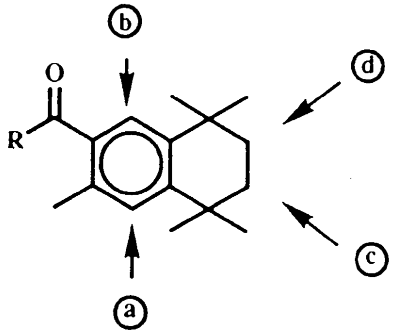
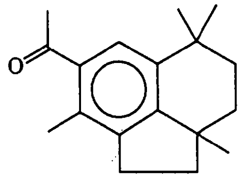


Fig. 1. Basic musk skeleton structure, $R = H, CH_3$ (reprinted from Fehr *et al.*, 1989).

This skeleton is represented in a musk compound described in the second paper of this series (De Ridder, Goubitz & Schenk, 1990): incorporation of a methyl group at the position marked 'c' leads to musk tonalid ($R = CH_3$). By the appropriate structural modification of known musk odorants, Fehr, Galindo, Haubrichs & Perret (1989) manufactured a number of new compounds. According to these authors, the incorporation of an additional CH_3 or CH_2 group into the basic musk skeleton of Fig. 1 modifies only slightly the global shape of the molecule, but leads to densely packed structures of enhanced lipophilicity. *Via* the incorporation of a methyl group at the position marked 'a', a tricyclic musk (I) was obtained, the crystal structure of which is described in this paper.



(I)

The title compound is a strong musk and was obtained from the Firmenich laboratories.

For convenience, the six-membered rings are numbered analogously to the tetralin structures described by De Ridder, Goubitz & Schenk (1990) (see Fig. 2). This numbering is different to the commonly used IUPAC system.

The phenyl ring is practically planar; the atoms directly attached to the benzene ring show considerable devia-

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† This work forms part of a thesis by De Ridder (1992).

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