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.

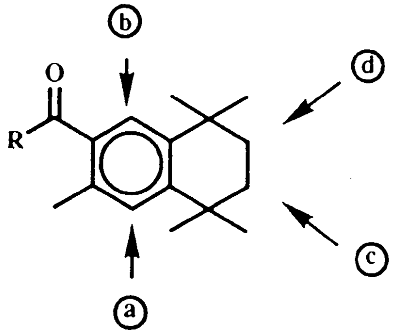
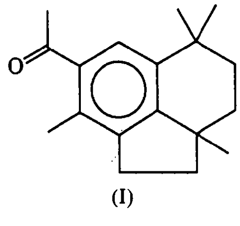


Fig. 1. Basic musk skeleton structure, $R = H, CH_3$ (reprinted from Fehr *et al.*, 1989).

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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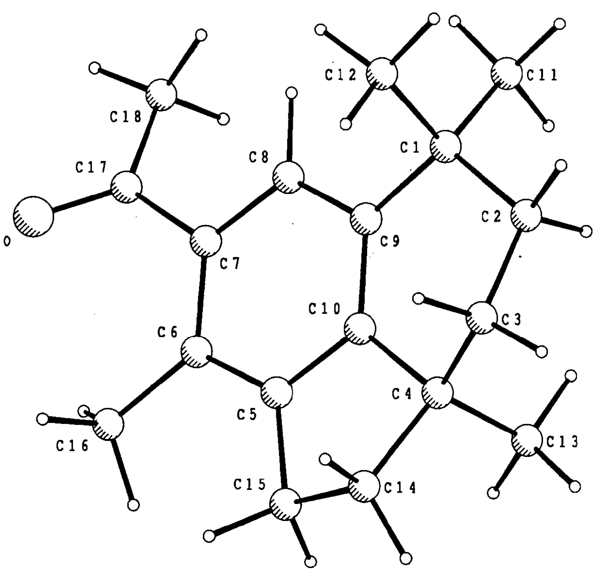

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) drawing of the title compound. The H atoms are shown but not labelled.

tions from the plane of the ring (see Table 3) because of the strain induced by the fused rings. Therefore, the non-aromatic six-membered ring adopts a somewhat deformed half-chair conformation and the five-membered ring a deformed envelope conformation. This is reflected in the distances of C2 and C3 from the best plane through C1, C9, C10 and C4 [−0.118 (4) and 0.560 (5) Å, respectively] and in the distance of atom C14 from the best plane through C4, C10, C5 and C15 [0.499 (4) Å]. In the title compound, the O atom is facing the methyl group at C6, analogous to musk tonalid (De Ridder, Goubitz & Schenk, 1990) and musk phantolid (De Ridder & Schenk, 1991). The angle between the acetyl group and the phenyl ring is 31.6 (1)°.

Experimental

Crystal data

$C_{18}H_{24}O$
$M_r = 256.39$
Monoclinic
$P2_1/a$
$a = 10.904$ (1) Å
$b = 10.3686$ (8) Å
$c = 13.887$ (2) Å
$\beta = 102.132$ (7)°
$V = 1535.0$ (3) Å^3
$Z = 4$
$D_x = 1.109$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 23 reflections
$\theta = 40$–$44.5°$
$\mu = 0.475$ mm^{-1}
$T = 251$ K
Block-shaped crystals
0.8 × 0.5 × 0.4 mm
Colourless
Crystal source: slow evaporation of ethanol

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.72$, $T_{\max} = 1.34$
2849 measured reflections
2603 independent reflections

2297 observed reflections $[I_{\rm net} > 2.5\sigma(I_{\rm net})]$
$\theta_{\max} = 64.81°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 12$
$l = -16 \rightarrow 15$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
$R = 0.057$
$wR = 0.067$
$S = 0.14$
2297 reflections
269 parameters
H atoms refined, restrained at 1.09 Å from their carrier atom
$w = 1/(10.0 + F_o + 0.0033F_o^2)$
$(\Delta/\sigma)_{\max} = 0.153$

$\Delta\rho_{\max} = 0.198$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 1.7 (4) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* program comparable to *Xtal LATCON*. Data reduction: *Xtal SORTRF* (Hall, Spadaccini & Stewart, 1990) and *ADDREF* (Davenport & Hall, 1990). Program(s) used to solve structure: *Xtal SIMPEL* (Schenk & Hall, 1990). Program(s) used to refine structure: *Xtal CRYLSQ* (Olthof-Hazekamp, 1990). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *BONDLA* (Dreissig, Doherty, Stewart & Hall, 1990) and *CIFIO* (Hall, 1990).

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

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

	x	y	z	$U_{\rm eq}$
C1	0.4525 (2)	0.4376 (2)	0.7564 (2)	0.049 (1)
C2	0.3513 (3)	0.4098 (3)	0.6624 (2)	0.067 (1)
C3	0.2477 (2)	0.5107 (3)	0.6377 (2)	0.069 (1)
C4	0.3025 (2)	0.6417 (2)	0.6216 (2)	0.055 (1)
C5	0.4132 (2)	0.8027 (2)	0.7307 (1)	0.047 (1)
C6	0.5037 (2)	0.8534 (2)	0.8072 (2)	0.047 (1)
C7	0.5876 (2)	0.7634 (2)	0.8633 (1)	0.0426 (9)
C8	0.5740 (2)	0.6315 (2)	0.8442 (1)	0.043 (1)
C9	0.4802 (2)	0.5812 (2)	0.7683 (1)	0.043 (1)
C10	0.4058 (2)	0.6717 (2)	0.7101 (1)	0.045 (1)
C11	0.5718 (2)	0.3615 (2)	0.7504 (2)	0.060 (1)
C12	0.4039 (2)	0.3918 (3)	0.8472 (2)	0.061 (1)
C13	0.3559 (3)	0.6436 (3)	0.5265 (2)	0.067 (1)
C14	0.2176 (2)	0.7609 (3)	0.6216 (2)	0.069 (1)
C15	0.3066 (2)	0.8717 (3)	0.6625 (2)	0.064 (1)
C16	0.5072 (3)	0.9963 (2)	0.8267 (2)	0.064 (1)
C17	0.6898 (2)	0.8055 (2)	0.9477 (2)	0.050 (1)
C18	0.8072 (2)	0.7289 (3)	0.9719 (2)	0.064 (1)
O	0.6776 (2)	0.9006 (2)	0.9965 (1)	0.087 (1)

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

C1—C2	1.550 (3)	C5—C15	1.516 (3)
C1—C9	1.521 (3)	C6—C7	1.419 (3)
C1—C11	1.538 (3)	C6—C16	1.506 (3)
C1—C12	1.542 (4)	C7—C8	1.396 (3)
C2—C3	1.524 (4)	C7—C17	1.503 (3)
C3—C4	1.519 (4)	C8—C9	1.405 (3)
C4—C10	1.514 (3)	C9—C10	1.384 (3)
C4—C13	1.552 (4)	C14—C15	1.533 (4)

C4—C14	1.544 (4)	C17—C18	1.484 (3)
C5—C6	1.392 (3)	C17—O	1.219 (3)
C5—C10	1.386 (3)		
C2—C1—C9	111.3 (2)	C5—C6—C16	119.8 (2)
C2—C1—C11	108.7 (2)	C7—C6—C16	124.0 (2)
C2—C1—C12	109.0 (2)	C6—C7—C8	120.7 (2)
C9—C1—C11	110.9 (2)	C6—C7—C17	121.6 (2)
C9—C1—C12	107.9 (2)	C8—C7—C17	117.7 (2)
C11—C1—C12	109.0 (2)	C7—C8—C9	122.5 (2)
C1—C2—C3	115.2 (2)	C1—C9—C8	122.7 (2)
C2—C3—C4	110.5 (2)	C1—C9—C10	121.4 (2)
C3—C4—C10	108.3 (2)	C8—C9—C10	115.6 (2)
C3—C4—C13	111.5 (2)	C4—C10—C5	111.7 (2)
C3—C4—C14	117.5 (2)	C4—C10—C9	125.4 (2)
C10—C4—C13	109.9 (2)	C5—C10—C9	122.8 (2)
C10—C4—C14	100.3 (2)	C4—C14—C15	105.5 (2)
C13—C4—C14	108.8 (2)	C5—C15—C14	102.7 (2)
C6—C5—C10	121.9 (2)	C7—C17—C18	118.9 (2)
C6—C5—C15	128.7 (2)	C7—C17—O	121.2 (2)
C10—C5—C15	109.3 (2)	C18—C17—O	119.8 (2)
C5—C6—C7	116.2 (2)		

Table 3. *Distances from the best plane through the phenyl ring* (Å)

	Ring atoms	Attached atoms
C5	0.012 (3)	0.134 (4)
C6	0.019 (2)	0.081 (4)
C7	−0.021 (2)	−0.021 (3)
C8	−0.000 (2)	-
C9	0.026 (2)	0.253 (3)
C10	−0.036 (2)	−0.080 (4)

The crystals were recrystallized by slow evaporation from ethanol. The structure was determined by direct methods (*SIMPEL*; Schenk & Hall, 1990). H-atom positions were calculated on the basis of standard geometry. Full-matrix least-squares refinement on *F*, anisotropic for non-H atoms and isotropic for H atoms.

The authors gratefully acknowledge the provision of the crystals by Dr C. Fehr of Firmenich-Geneva (Switzerland). We wish to thank Ing. Jan Fraanje for collecting the X-ray data.

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 71502 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1068]

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Acta Cryst. (1994). **C50**, 267–269

2,2′-Biadamantane

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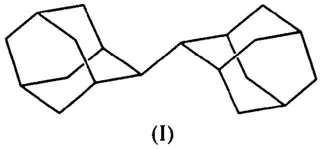
Abstract

The structure of 2,2′-biadamantane, $C_{20}H_{30}$, (space group *C*2/*c*) has been determined by single-crystal diffractometry. The central C—C linkage was found to be 1.542 (2) Å and is perfectly staggered (crystallographic center of symmetry). The bridging induces a small distortion of the adamantane moieties [average C—C 1.533 (5) Å, average C—C—C 109.6 (6)°].

Comment

Few structures of compounds involving the 2,2′-biadamantane skeleton are known (*e.g.* Watson & Nagl, 1988; Hess & Vos, 1977; Slebocka-Tilk, Ball & Brown, 1985). While investigating mechanistic aspects of a modified Barbier reaction (Mielke, 1993) we obtained the parent hydrocarbon 2,2′-biadamantane (I) (van Zorge, Strating



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