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Crystal Studies of Musk Compounds. IX.† 4-Acetyl-1,1,3,3,5,6-hexamethylindan

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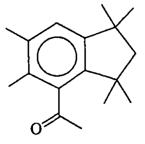
Abstract

The structure of the title compound, $C_{17}H_{24}O$, which is a weak musk, has been determined by X-ray diffraction. The acetyl group is nearly perpendicular to the aromatic ring. The molecular dimensions are discussed with respect to other musk compounds belonging to the indan family.

Comment

The title compound (I) is a structural isomer of Musk Phantolid (6-acetyl-1,1,2,3,3,5-hexamethylindan), the crystal structure of which has been described (De Ridder & Schenk, 1991*a*). (I) is only a weak musk compared to Musk Phantolid (Weber, Kleipool & Spoelstra, 1957). In contrast to Musk Phantolid and the musk compounds discussed in part VIII of this series (De Ridder, Čapková, Hatjisymeon, Fraanje & Schenk, 1994), the title compound carries an acetyl group at the C4 atom of the indan moiety.



(I)

The aromatic ring is essentially planar, the maximum deviation of a ring atom from the best plane through its six atoms being 0.008 (2) Å. The distance of atom C2 from the best plane through the atoms C1, C8, C9, C3 [0.367 (3) Å] and the dihedral angle between this plane and the plane through C1, C2, C3 [24.0 (2)°] are comparable to the corresponding values found in Musk Phantolid (De Ridder & Schenk, 1991*a*). The five-membered ring adopts an almost ideal C2 envelope conformation; the asymmetry parameter $\Delta_s^2 = 1.3°$ (Duax, Weeks & Rohrer, 1976).

The angle of the acetyl group to the aromatic plane is 78.86 (9)°, which is significantly larger than in Musk Phantolid or its homologues. Since this compound still has a weak musk odour while the osmophoric group is almost perpendicular to the aromatic ring, this is again an indication that the coplanarity of the active group with a phenyl ring is not essential for the occurrence of musk odour [see part VIII of this series (De Ridder, Čapková, Hatjisymeon, Fraanje & Schenk, 1994)].

The dimensions of a triangle defined by the two quaternary C atoms of the non-aromatic ring and the sp^2-C atom of the acetyl group (or the N atom of a nitro group in the same position) attached to the aromatic ring have been compared for a number of indans and tetralins in both acetyl and nitro compounds (De Ridder & Schenk, 1991*b*). The distances between the quaternary atoms were 2.51-2.54 and 3.05-3.09 Å for the indans and tetralins, respectively, the former being comparable to the distance in the title compound [C1···C3 2.512 (3) Å]. However, the two other dimensions of the triangle, in the ranges 4.91-5.09 and 5.72-5.85 Å, respectively, are significantly larger than found in the title compound [C3···C14 3.156 (3) and C1···C14 4.943 (3) Å]. These different molecular dimensions may explain why the title compound is only a weak musk.

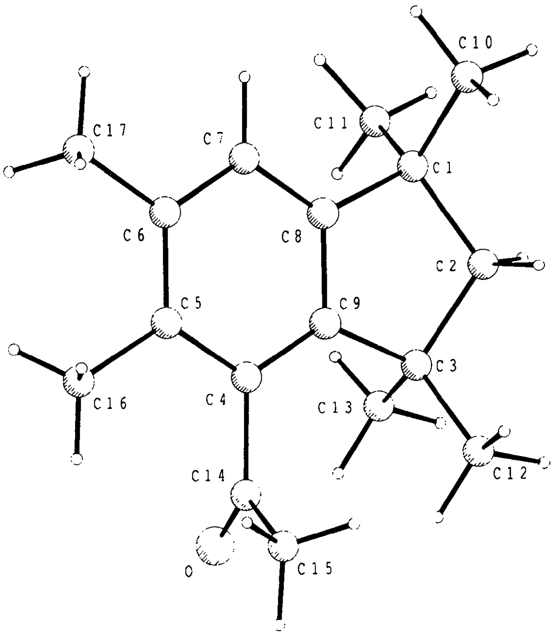


Fig. 1. *PLUTO* drawing (Motherwell & Clegg, 1978) of the title compound. The H atoms (not labelled) are shown as spheres of arbitrary size.

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

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Experimental

Crystal data

$C_{17}H_{24}O$
M_r = 244.38
Monoclinic
$P2_1/c$
a = 10.213 (2) Å
b = 8.151 (1) Å
c = 17.671 (3) Å
β = 94.83 (2)°
V = 1465.8 (4) Å^3
Z = 4

D_x = 1.107 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 22 reflections
θ = 35.5-39.7°
μ = 0.475 mm^{-1}
T = 196 K
Block shaped
0.6 × 0.6 × 0.5 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min}$ = 0.67, $T_{\max}$ = 1.19
3333 measured reflections
3024 independent reflections

2371 observed reflections $[I_{\rm net} > 2.5\sigma(I_{\rm net})]$
$\theta_{\max}$ = 75.03°
$h = -12 \rightarrow 0$
$k = 0 \rightarrow 10$
$l = -21 \rightarrow 22$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
R = 0.05
wR = 0.064
S = 0.159
2371 reflections
260 parameters
H-atoms refined at calculated positions
$w = 1/(6.08 + F_o + 0.0050F_o^2)$
$(\Delta/\sigma)_{\max}$ = 0.135

$\Delta\rho_{\max}$ = 0.226 e Å^{-3}
$\Delta\rho_{\min}$ = -0.204 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 5 (1) × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

	x	y	z	$U_{\rm eq}$
C1	0.2786 (2)	0.4418 (2)	0.2120 (1)	0.0376 (9)
C2	0.1728 (2)	0.3058 (3)	0.2067 (1)	0.043 (1)
C3	0.1687 (2)	0.2267 (2)	0.1269 (1)	0.0367 (9)
C4	0.2279 (2)	0.3812 (2)	0.00258 (9)	0.0317 (8)
C5	0.2922 (2)	0.5153 (2)	−0.02779 (9)	0.0336 (8)
C6	0.3555 (2)	0.6325 (2)	0.0209 (1)	0.0343 (8)
C7	0.3511 (2)	0.6142 (2)	0.0988 (1)	0.0345 (8)
C8	0.2888 (2)	0.4812 (2)	0.12880 (9)	0.0311 (8)
C9	0.2266 (2)	0.3636 (2)	0.08118 (9)	0.0300 (8)
C10	0.2371 (2)	0.5910 (3)	0.2567 (1)	0.049 (1)
C11	0.4114 (2)	0.3815 (3)	0.2491 (1)	0.050 (1)
C12	0.0276 (2)	0.1792 (3)	0.0994 (1)	0.049 (1)
C13	0.2549 (3)	0.0718 (3)	0.1267 (1)	0.052 (1)
C14	0.1609 (2)	0.2578 (2)	−0.0514 (1)	0.0394 (9)
C15	0.0291 (2)	0.3042 (3)	−0.0898 (1)	0.054 (1)
C16	0.2994 (2)	0.5305 (3)	−0.1128 (1)	0.047 (1)
C17	0.4269 (2)	0.7762 (3)	−0.0102 (1)	0.048 (1)
O	0.2124 (2)	0.1282 (2)	−0.06377 (9)	0.0557 (9)

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

C1—C2	1.545 (3)	C4—C14	1.510 (3)
C1—C8	1.517 (2)	C5—C6	1.406 (2)
C1—C10	1.529 (3)	C5—C16	1.514 (3)
C1—C11	1.537 (3)	C6—C7	1.389 (3)
C2—C3	1.547 (3)	C6—C17	1.509 (3)
C3—C9	1.526 (3)	C7—C8	1.385 (3)
C3—C12	1.531 (3)	C8—C9	1.393 (2)
C3—C13	1.540 (3)	C14—C15	1.504 (3)
C4—C5	1.405 (3)	C14—O	1.208 (3)
C4—C9	1.398 (2)		
C2—C1—C8	101.3 (1)	C4—C5—C6	120.0 (2)
C2—C1—C10	112.3 (2)	C4—C5—C16	120.4 (2)
C2—C1—C11	112.6 (2)	C6—C5—C16	119.6 (2)
C8—C1—C10	112.1 (2)	C5—C6—C7	118.9 (2)
C8—C1—C11	110.6 (2)	C5—C6—C17	121.0 (2)
C10—C1—C11	107.9 (2)	C7—C6—C17	120.1 (2)
C1—C2—C3	108.7 (2)	C6—C7—C8	121.1 (2)
C2—C3—C9	101.3 (1)	C1—C8—C7	127.4 (2)
C2—C3—C12	110.4 (2)	C1—C8—C9	112.0 (2)
C2—C3—C13	111.8 (2)	C7—C8—C9	120.6 (2)
C9—C3—C12	114.4 (2)	C3—C9—C4	129.6 (2)
C9—C3—C13	110.5 (2)	C3—C9—C8	111.1 (1)
C12—C3—C13	108.3 (2)	C4—C9—C8	119.2 (2)
C5—C4—C9	120.2 (2)	C4—C14—C15	117.3 (2)
C5—C4—C14	118.6 (2)	C4—C14—O	121.0 (2)
C9—C4—C14	121.2 (2)	C15—C14—O	121.7 (2)

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

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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 71813 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1084]

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Acta Cryst. (1994). C**50**, 755–757

Crystal Studies of Musk Compounds. X.†‡ 1,1,2,3,3-Pentamethyl-2,3,4,5,6,7-hexahydroinden-4-one

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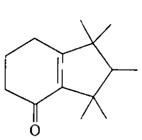
Abstract

The structure of the title compound, $C_{14}H_{22}O$, which is a strong musk, has been established by X-ray diffraction. The five- and six-membered rings adopt envelope and sofa conformations, respectively. The molecular dimensions are discussed with respect to the known structure-activity relationships of musk compounds.

Comment

The title compound (I) is the main component of the commercial product *Cashmeran* and has a strong musk odour (International Flavors & Fragrances Inc., 1973). It is one of the most intriguing musk compounds, since it is, as yet, the only known bicyclic musk compound without an aromatic ring and may be the first example of a new series of musk compounds. The crystal structures of a number of bicyclic compounds belonging to the indan family (*i.e.* an aromatic six-membered ring fused with a non-aromatic five-membered ring) have been discussed in part III (De Ridder & Schenk, 1991*a*) and part VIII (De Ridder, Čapková, Hatjisymeon, Fraanje & Schenk, 1994) of this series.



(I)

In this compound the methyl group attached to C2 is found to be in the equatorial position, analogous to the indan compounds described previously. The six-membered ring has two conjugated double bonds which constrain atoms C1, C3, C4, C5, C7, C8, C9 and O to be coplanar or nearly so [the maximum deviation from the least-squares plane through these atoms is 0.046 (5) Å]. As a result, the six-membered ring has to adopt a C6-sofa conformation and the five-membered ring a C2-envelope conformation; the ring-displacement asymmetry parameters (Duax, Weeks & Rohrer, 1976) are Δ_s^6 = 4.3° and Δ_s^2 = 0.7°, respectively.

The cyclopentene ring shows the same type of disorder as discussed in part VIII (De Ridder, Čapková, Hatjisymeon, Fraanje & Schenk, 1994) of this series. The quantities defined in part VIII are as follows for the title compound: the dihedral angle between the least-squares planes through atoms C1, C8, C9, C3 and atoms C1, C2, C3 is 7.1 (8)°; the distance of atom C2 from the former plane is 0.11 (1) Å; the sum of the angles around atom C2 is 358.7°. For comparison, the average values for four compounds not having a disordered cyclopentene ring are 25.3°, 0.39 Å and 341°, respectively. This corroborates with the high displacement parameter observed for C2.

For a number of indans and tetralins (both acetyl and nitro compounds), the dimensions of a triangle defined by the two quaternary C atoms of the non-aromatic ring and the sp^2-C atom of the acetyl group (or the N atom of a nitro group in the same position) attached to the aromatic ring, have been compared (De Ridder & Schenk, 1991*b*). The distances between the quaternary atoms were 2.51-2.54 and 3.05-3.09 Å for the indans and tetralins, respectively, the former being comparable to the distance in the title compound [C1···C3 2.497 (6) Å]. However, the two other dimensions of the triangle, in the ranges 4.91-5.09

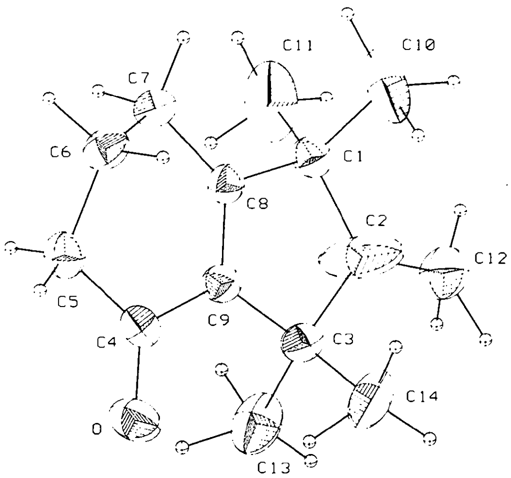


Fig. 1. *PEANUT* drawing (Hummel, Hauser & Bürgi, 1990) of the title compound. Displacement ellipsoids are shown at the 30% probability level; H atoms (not labelled) are drawn as spheres of arbitrary size.

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

‡ Part IX: De Ridder, Fraanje & Schenk (1994).

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