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Crystal Studies of Musk Compounds. VII.† Molecular Structures of 1-*tert*-Butyl-2,4,6-trimethyl-3,5-dinitrobenzene, $C_{13}H_{18}N_2O_4$ (2), and 1-Bromo-3,5-dimethyl-2,4,6-trinitrobenzene, $C_8H_6BrN_3O_6$ (4)

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(*Received* 15 *June* 1993; *accepted* 14 *October* 1993)

Abstract

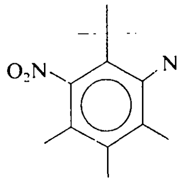
The crystal structures of the title compounds (both non-musks) have been determined by X-ray diffraction. The angles between the nitro groups and the phenyl ring [75.1 (3)-86.6 (3)° for (2) and 67.8 (3)-80.1 (3)° for (4)] are compared with the values found in two strong musk compounds [(1) and (3)]. The phenyl ring in (4) is essentially planar; in (2), significant distortions from planarity are observed.

Comment

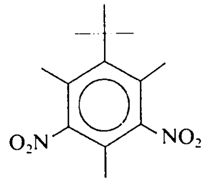
In the first paper of this series (De Ridder, Goubitz & Schenk, 1990), the molecular structure of Musk Ambrette was described. This compound belongs to the class of nitrobenzene musks of which Musk Tibetene (1) and Musk Xylene (3) are two other examples.

In 1977, Beets introduced the pseudo-*meta* and pseudo-*ortho* musks enabling the classification of the nitrobenzene compounds: the prefix pseudo is based on two postulates in which he stated that a nitro group is able to play two different roles, depending on its position in the total structure (Beets, 1957, 1977). According to the first postulate, a nitro group in a sterically unhindered position, permitting its coplanarity with the benzene ring, may act, in the absence of more effective candidates, as a functional group analogous to an acetyl group. The second postulate states that a nitro group, of which the coplanarity with the benzene ring is prevented by one or two adjacent bulky substituents and of which, consequently, the O atoms are forced out of the plane of the benzene ring, may function as a detail of the molecular profile in a way analogous to a tertiary butyl group, of which two methyl groups are necessarily projecting out of the plane of the ring. According to these postulates, (3) is a pseudo-*ortho* musk in which the nitro group in the *para* position with respect to the *tert*-butyl group has the osmophoric function and one of the nitro groups *ortho* to the *tert*-butyl group has the sterical function. These postulates do not explain why (1) is a strong musk, since both nitro groups are not in sterically unhindered positions. Contrary to these postulates, (2), which can easily be classified as a pseudo-*meta* musk, is odourless (Pesaro, 1990).

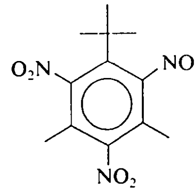
In (4), the *tert*-butyl group of Musk Xylene (3) has been replaced by a Br atom, resulting in a non-musk (Döpp, 1991).



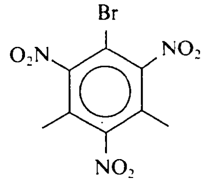
(1)



(2)



(3)



(4)

In the light of these postulates, it was deemed useful to determine the structures of (2) and (4), thus enabling a comparison with the structures of the strong musks (1) and (3).

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

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The phenyl ring of (4) is planar within the limits of accuracy, the maximum deviation of a ring atom from the best plane being 0.008 Å. In molecules of (2), the deviations from planarity are significant: −0.077 (8) and 0.069 (8) Å for C1 of molecules *A* and *B*, respectively. The reason can be found in the presence of the very bulky *tert*-butyl group flanked by two methyl groups, which push each other towards opposite sides of the ring [deviations of C2 and C6 0.048 (8) and 0.043 (9) Å, respectively, for molecule *A*, and −0.042 (9) and −0.046 (9) Å, respectively, for molecule *B*].

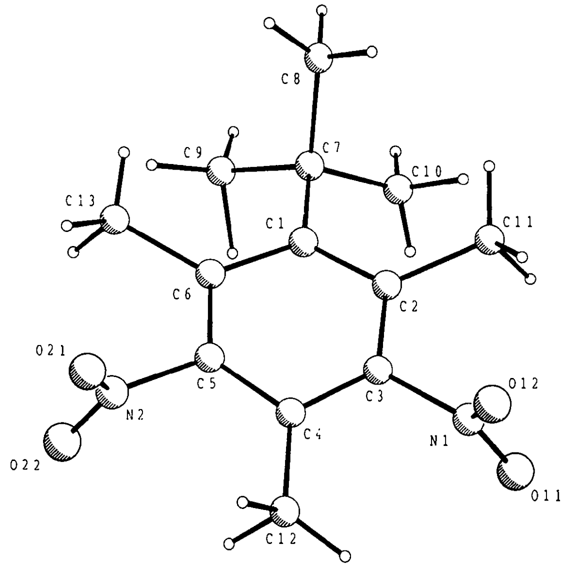

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of (2). The H atoms are shown but not labelled.

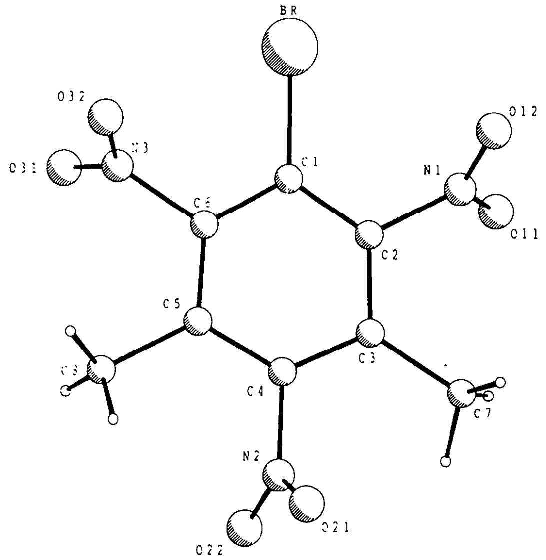

Fig. 2. *PLUTO* (Motherwell & Clegg, 1978) drawing of (4). The H atoms are shown but not labelled.

In (2), the angles between the nitro groups and the aromatic plane [75.1 (3) and 77.8 (3)° for the nitro groups at C3 and C5, respectively, for molecule *A*, and 86.6 (3) and 75.7 (4)°, respectively, for molecule *B*] are comparable to the values found in Musk Xylene and Musk Tibetene, which are in the range 77.4-83.6° (De Ridder, 1992). Most of the structure-activity theories of musk compounds lean heavily on the assumed orientation of the active group(s) (*e.g.* Beets, 1977). Since there is no significant difference between the 'twisting-out' angles of the non-musks compared with the strong musks, the orientation of the active group(s) seems not to be a decisive factor for muskiness. In (4), the angles between the nitro groups and the aromatic plane are 67.8 (3), 75.6 (3) and 80.1 (3)° for the nitro groups attached to C2, C4 and C6, respectively. A survey of the behaviour of the nitro groups with respect to aromatic six-membered rings will be reported elsewhere (De Ridder & Schenk, 1993).

Compound (2) has two molecules in the asymmetric unit. Matching the non-H atoms of molecule *B* with those of molecule *A* resulted in an r.m.s. value of 0.09 Å.

Experimental

In the first step of the synthesis of (2) (Pesaro, 1990), a Grignard reagent was prepared from magnesium turnings and 2,4,6-trimethyl-1-bromobenzene in dry ether. To this solution was added *tert*-butyl chloride, yielding 1-*tert*-butyl-2,4,6-trimethylbenzene. This intermediate was nitrated with a mixture of nitric and sulfuric acid at 0°C. The final product was purified by chromatography, giving colourless crystals of (2). Compound (4) was synthesized in the laboratory of Professor Döpp, according to the method of Blanksma (1907).

Compound (2)

Crystal data

$C_{13}H_{18}N_2O_4$
$M_r = 266.30$
Triclinic
$P\bar{1}$
$a = 9.932$ (3) Å
$b = 11.20$ (1) Å
$c = 14.08$ (1) Å
$\alpha = 74.10$ (5)°
$\beta = 75.54$ (4)°
$\gamma = 71.53$ (3)°
$V = 1407$ (7) Å^3
$Z = 4$

$D_x = 1.258$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 23 reflections
$\theta = 20.9$-$26.9°$
$\mu = 0.742$ mm^{-1}
$T = 298$ K
Block
0.75 × 0.20 × 0.05 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.82$, $T_{\max} = 1.21$
4884 measured reflections
4762 independent reflections

1935 observed reflections [$I_{\rm net} > 2.5\sigma(I_{\rm net})$]
$\theta_{\max} = 65.02°$
$h = 0 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
$R = 0.061$
$wR = 0.089$
$S = 0.281$
1935 reflections
488 parameters
All H-atom parameters refined
$w = 1/(5.62 + F_o + 0.013F_o^2)$
$(\Delta/\sigma)_{\max} = 0.844$
$\Delta\rho_{\max} = 0.183$ e Å^{-3}
$\Delta\rho_{\min} = -0.173$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: $g = 3(1) \times 10^{-6}$
Atomic scattering factors from Cromer & Mann (1968) (C, O, N) and Stewart, Davidson & Simpson (1965) (H)

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

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

	x	y	z	U_{eq}
C1*A*	0.9347 (7)	0.3659 (6)	0.4152 (4)	0.047 (6)
C2*A*	0.9475 (7)	0.4771 (6)	0.3438 (4)	0.046 (6)
C3*A*	1.0851 (7)	0.4907 (7)	0.2948 (5)	0.051 (7)
C4*A*	1.2134 (7)	0.4044 (6)	0.3155 (5)	0.049 (7)
C6*A*	1.0624 (7)	0.2826 (6)	0.4501 (5)	0.052 (7)
C5*A*	1.1929 (7)	0.3057 (6)	0.3975 (5)	0.049 (6)
C7*A*	0.7878 (7)	0.3283 (7)	0.4513 (5)	0.061 (8)
C8*A*	0.700 (1)	0.387 (1)	0.5420 (8)	0.09 (1)
C9*A*	0.807 (1)	0.1826 (9)	0.479 (1)	0.10 (1)
C10*A*	0.699 (1)	0.377 (2)	0.367 (1)	0.10 (2)
C11*A*	0.821 (1)	0.5949 (9)	0.3217 (8)	0.069 (9)
C12*A*	1.360 (1)	0.418 (1)	0.2546 (7)	0.07 (1)
C13*A*	1.067 (1)	0.1788 (9)	0.5440 (6)	0.07 (1)
N1*A*	1.0963 (7)	0.6056 (6)	0.2134 (5)	0.065 (7)
N2*A*	1.3263 (6)	0.2159 (7)	0.4321 (5)	0.067 (7)
O11*A*	1.0744 (7)	0.6086 (6)	0.1320 (4)	0.097 (8)
O12*A*	1.1261 (8)	0.6923 (6)	0.2333 (5)	0.102 (8)
O21*A*	1.3836 (6)	0.2494 (6)	0.4832 (5)	0.096 (8)
O22*A*	1.3735 (8)	0.1145 (7)	0.4066 (6)	0.13 (1)
C1*B*	0.5457 (7)	0.0980 (6)	0.1194 (5)	0.049 (6)
C2*B*	0.4317 (7)	0.0432 (6)	0.1771 (4)	0.050 (7)
C3*B*	0.2914 (7)	0.1112 (7)	0.1653 (5)	0.052 (7)
C4*B*	0.2512 (7)	0.2260 (7)	0.0992 (5)	0.054 (7)
C5*B*	0.3660 (8)	0.2662 (6)	0.0373 (5)	0.054 (7)
C6*B*	0.5122 (7)	0.2052 (6)	0.0419 (4)	0.051 (7)
C7*B*	0.7044 (7)	0.0443 (7)	0.1440 (6)	0.065 (8)
C8*B*	0.791 (1)	−0.062 (1)	0.0878 (9)	0.10 (1)
C9*B*	0.781 (1)	0.154 (1)	0.118 (1)	0.12 (2)
C10*B*	0.699 (1)	−0.008 (2)	0.2591 (8)	0.11 (2)
C11*B*	0.452 (1)	−0.0878 (9)	0.2461 (8)	0.08 (1)
C12*B*	0.095 (1)	0.299 (1)	0.0945 (9)	0.09 (1)
C13*B*	0.624 (1)	0.252 (1)	−0.0451 (7)	0.08 (1)
N1*B*	0.1735 (7)	0.0566 (7)	0.2283 (5)	0.070 (7)
N2*B*	0.3333 (8)	0.3845 (7)	−0.0404 (6)	0.081 (9)
O11*B*	0.1329 (7)	−0.0130 (6)	0.1949 (5)	0.099 (8)
O12*B*	0.1198 (8)	0.0836 (8)	0.3085 (5)	0.122 (9)
O21*B*	0.301 (1)	0.3752 (8)	−0.1135 (6)	0.17 (1)
O22*B*	0.3414 (8)	0.4833 (6)	−0.0273 (5)	0.110 (9)

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

	Molecule *A*	Molecule *B*
C1—C2	1.389 (8)	1.42 (1)
C1—C6	1.425 (9)	1.397 (8)
C1—C7	1.57 (1)	1.59 (1)
C2—C3	1.403 (9)	1.387 (9)
C2—C11	1.53 (1)	1.51 (1)
C3—C4	1.377 (9)	1.372 (9)
C3—N1	1.483 (9)	1.47 (1)
C4—C5	1.387 (8)	1.37 (1)
C4—C12	1.52 (1)	1.52 (1)
C5—C6	1.39 (1)	1.40 (1)
C5—N2	1.489 (9)	1.473 (9)
C6—C13	1.50 (1)	1.53 (1)
C7—C8	1.54 (1)	1.52 (1)
C7—C9	1.53 (1)	1.56 (2)
C7—C10	1.54 (2)	1.56 (1)
N1—O11	1.21 (1)	1.22 (1)
N1—O12	1.22 (1)	1.20 (1)
N2—O21	1.21 (1)	1.19 (1)
N2—O22	1.20 (1)	1.20 (1)
C2—C1—C6	117.4 (6)	117.5 (6)
C2—C1—C7	121.6 (5)	121.9 (5)
C6—C1—C7	120.9 (5)	120.5 (6)
C1—C2—C3	119.5 (5)	118.2 (5)
C1—C2—C11	124.2 (6)	124.8 (7)
C3—C2—C11	116.0 (6)	116.8 (7)
C2—C3—C4	125.1 (6)	125.8 (6)
C2—C3—N1	118.8 (5)	118.0 (5)
C4—C3—N1	116.2 (6)	116.2 (6)
C3—C4—C5	112.1 (6)	113.3 (6)
C3—C4—C12	123.1 (6)	123.1 (7)
C5—C4—C12	124.8 (6)	123.6 (6)
C4—C5—C6	127.1 (6)	125.7 (6)
C4—C5—N2	115.8 (6)	117.2 (6)
C6—C5—N2	117.1 (5)	117.1 (6)
C1—C6—C5	117.3 (5)	118.2 (6)
C1—C6—C13	125.5 (7)	124.3 (6)
C5—C6—C13	117.0 (7)	117.2 (6)
C1—C7—C8	110.0 (8)	109.9 (8)
C1—C7—C9	113.3 (6)	111.1 (7)
C1—C7—C10	110.8 (7)	110.1 (6)
C8—C7—C9	109.2 (8)	111.3 (8)
C8—C7—C10	109.0 (8)	110.1 (8)
C9—C7—C10	104.0 (1)	104.0 (1)
C3—N1—O11	118.7 (8)	117.8 (7)
C3—N1—O12	117.6 (7)	119.4 (9)
O11—N1—O12	123.7 (7)	122.8 (8)
C5—N2—O21	118.5 (7)	117.0 (8)
C5—N2—O22	118.4 (8)	119.0 (8)
O21—N2—O22	123.1 (7)	124.0 (8)

Compound (4)

Crystal data

$C_8H_6BrN_3O_6$
$M_r = 320.06$
Triclinic
$P\bar{1}$
$a = 5.4224$ (9) Å
$b = 8.975$ (1) Å
$c = 12.094$ (2) Å
$\alpha = 77.820$ (9)°
$\beta = 84.19$ (2)°
$\gamma = 76.46$ (1)°
$V = 558.5$ (2) Å^3
$Z = 2$
$D_x = 1.903$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 23 reflections
$\theta = 40.0$-44.8°
$\mu = 5.34$ mm^{-1}
$T = 298$ K
Block
0.6 × 0.3 × 0.25 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.51$, $T_{\max} = 1.97$
2364 measured reflections
2288 independent reflections
2072 observed reflections [$I_{\text{net}} > 2.5\sigma(I_{\text{net}})$]
$\theta_{\max} = 74.67$°
$h = 0 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$
2 standard reflections frequency: 60 min intensity variation: none

Refinement

Refinement on F
$R = 0.073$
$\Delta\rho_{\max} = 1.326$ e Å^{-3}
$\Delta\rho_{\min} = -1.556$ e Å^{-3}

$wR = 0.095$
$S = 0.286$
2072 reflections
188 parameters
All H-atom parameters refined
$w = 1/(4.50 + F_o + 0.0062F_o^2)$
$(\Delta/\sigma)_{\max} = 0.68$

Extinction correction: Zachariasen (1967)
Extinction coefficient: $g = 4.3\ (7) \times 10^{-6}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (4)

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

	x	y	z	$U_{\rm eq}$
Br	0.7443 (1)	1.02781 (8)	0.10859 (6)	0.0545 (7)
C1	0.603 (1)	0.8646 (6)	0.1905 (4)	0.033 (3)
C2	0.6601 (9)	0.7178 (6)	0.1632 (4)	0.031 (3)
C3	0.5540 (9)	0.5934 (5)	0.2211 (4)	0.030 (3)
C4	0.3778 (9)	0.6288 (5)	0.3092 (4)	0.029 (3)
C5	0.3061 (9)	0.7727 (6)	0.3419 (4)	0.032 (3)
C6	0.425 (1)	0.8870 (6)	0.2806 (4)	0.033 (3)
C7	0.624 (1)	0.4310 (6)	0.1903 (5)	0.038 (4)
C8	0.106 (1)	0.8037 (6)	0.4427 (4)	0.036 (4)
N1	0.8471 (9)	0.6899 (6)	0.0679 (4)	0.038 (3)
N2	0.2556 (9)	0.5040 (5)	0.3717 (4)	0.036 (3)
N3	0.363 (1)	1.0421 (5)	0.3127 (4)	0.042 (4)
O11	0.770 (1)	0.6679 (8)	−0.0167 (4)	0.067 (4)
O12	1.0666 (8)	0.6911 (7)	0.0793 (5)	0.064 (4)
O21	0.3751 (9)	0.4008 (5)	0.4414 (4)	0.053 (3)
O22	0.0363 (9)	0.5127 (7)	0.3532 (5)	0.069 (4)
O31	0.173 (1)	1.1311 (6)	0.2803 (6)	0.080 (5)
O32	0.512 (1)	1.0703 (7)	0.3700 (6)	0.077 (5)

Table 4. *Selected geometric parameters* (Å, °) *for* (4)

Br—C1	1.863 (5)	C5—C6	1.381 (7)
C1—C2	1.383 (8)	C5—C8	1.572 (7)
C1—C6	1.397 (7)	C6—N3	1.477 (7)
C2—C3	1.399 (7)	N1—O11	1.213 (8)
C2—N1	1.477 (7)	N1—O12	1.215 (7)
C3—C4	1.394 (7)	N2—O21	1.221 (6)
C3—C7	1.533 (8)	N2—O22	1.214 (7)
C4—C5	1.388 (7)	N3—O31	1.192 (7)
C4—N2	1.468 (7)	N3—O32	1.22 (1)
Br—C1—C2	122.4 (4)	C6—C5—C8	122.1 (5)
Br—C1—C6	121.1 (4)	C1—C6—C5	124.0 (5)
C2—C1—C6	116.5 (5)	C1—C6—N3	117.6 (5)
C1—C2—C3	124.0 (4)	C5—C6—N3	118.4 (5)
C1—C2—N1	118.0 (5)	C2—N1—O11	117.6 (5)
C3—C2—N1	117.9 (5)	C2—N1—O12	118.0 (5)
C2—C3—C4	114.7 (5)	O11—N1—O12	124.4 (5)
C2—C3—C7	122.9 (4)	C4—N2—O21	118.9 (5)
C4—C3—C7	122.4 (4)	C4—N2—O22	117.4 (4)
C3—C4—C5	125.5 (5)	O21—N2—O22	123.6 (5)
C3—C4—N2	117.3 (4)	C6—N3—O31	118.1 (6)
C5—C4—N2	117.2 (4)	C6—N3—O32	117.4 (5)
C4—C5—C6	115.4 (4)	O31—N3—O32	124.5 (6)
C4—C5—C8	122.6 (4)		

For both compounds, considering the values of Z and the statistics of the E values, the structures were refined in $P\bar{1}$. Compound (2) has two molecules in the asymmetric unit. The program *MISSYM* (Le Page, 1987), with a search on all non-H atoms, did not indicate any additional crystallographic symmetry. The structures were determined by direct methods. H-atom positions were calculated on the basis of standard geometry. For compound (4), the H atoms were restrained in such a way that the distances to their respective carrier atoms remained constant (at approximately 1.09 Å). A dispersion correction was applied for compound (4). For both compounds, full-matrix least-squares refinements were performed on F, anisotropic for the non-H atoms and isotropic for the H atoms. Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON*, comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal 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: *Xtal BONDLA* (Dreissig, Doherty, Stewart & Hall, 1990) and *Xtal CIFIO* (Hall, 1990).

The authors gratefully acknowledge the provision of crystals of (2) by Dr M. Pesaro of Givaudan-Roure-Dübendorf (Switzerland), and crystals of (4) by Professor Dr D. Döpp (University of Duisburg, Germany). Parts of the calculations for (2) were performed by Drs M. Numan.

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 71768 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1072]

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

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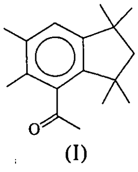
(*Received* 21 *September* 1993; *accepted* 4 *November* 1993)

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.

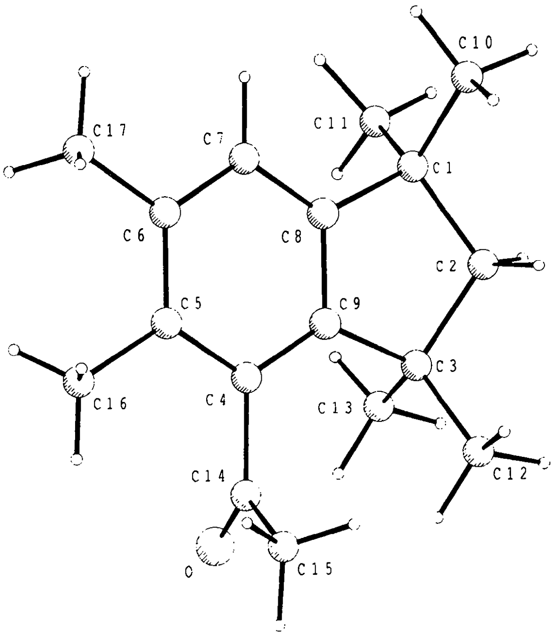


Fig. 1. *PLUTO* drawing (Motherwell & Clegg, 1978) of the title compound. The H atoms (not labelled) are shown as spheres of arbitrary size.

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

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Acta Crystallographica Section C
ISSN 0108-2701 © 1994