C(1)—C(6)	1.367 (8)	C(18)—C(19)	1.362 (8)
C(1)—C(2)	1.373 (7)	C(18)—C(23)	1.498 (10)
C(2)—C(3)	1.393 (8)	C(19)—C(20)	1.363 (13)
C(2)—C(7)	1.489 (9)	C(20)—C(21)	1.370 (12)
C(3)—C(4)	1.382 (11)	C(21)—C(22)	1.388 (9)
C(4)—C(5)	1.360 (9)	C(23)—C(24)	1.493 (9)
S(1)—Rh—S(2)	92.72 (4)	C(4)—C(3)—C(2)	119.2 (5)
S(1)—Rh—S(3)	93.25 (4)	C(5)—C(4)—C(3)	121.3 (6)
S(1)—Rh—Cl(1)	83.47 (5)	C(4)—C(5)—C(6)	120.1 (7)
S(1)—Rh—Cl(2)	172.96 (5)	C(1)—C(6)—C(5)	118.7 (5)
S(1)—Rh—Cl(3)	93.65 (5)	C(2)—C(7)—C(8)	108.0 (4)
S(2)—Rh—S(3)	172.55 (5)	C(7)—C(8)—S(1)	108.8 (4)
S(2)—Rh—Cl(1)	93.97 (5)	C(14)—C(9)—C(10)	122.6 (4)
S(2)—Rh—Cl(2)	81.80 (4)	C(14)—C(9)—S(2)	124.8 (4)
Cl(3)—Rh—S(2)	86.81 (5)	C(10)—C(9)—S(2)	112.5 (3)
Cl(3)—Rh—S(3)	88.36 (5)	C(11)—C(10)—C(9)	119.7 (4)
Cl(1)—Rh—S(3)	91.14 (5)	C(11)—C(10)—C(15)	126.8 (4)
Cl(2)—Rh—S(3)	92.57 (4)	C(9)—C(10)—C(15)	113.5 (4)
Cl(1)—Rh—Cl(2)	92.46 (5)	C(10)—C(11)—C(12)	118.4 (5)
Cl(1)—Rh—Cl(3)	177.05 (4)	C(13)—C(12)—C(11)	120.6 (4)
Cl(2)—Rh—Cl(3)	90.47 (5)	C(12)—C(13)—C(14)	122.2 (5)
C(1)—S(1)—C(8)	91.1 (2)	C(9)—C(14)—C(13)	116.5 (5)
C(1)—S(1)—Rh	111.3 (1)	C(10)—C(15)—C(16)	105.8 (4)
C(8)—S(1)—Rh	114.6 (2)	C(15)—C(16)—S(2)	106.3 (3)
C(9)—S(2)—C(16)	90.4 (2)	C(18)—C(17)—C(22)	120.8 (5)
C(9)—S(2)—Rh	112.2 (1)	C(18)—C(17)—S(3)	114.5 (5)
C(16)—S(2)—Rh	111.4 (2)	C(22)—C(17)—S(3)	124.6 (4)
C(17)—S(3)—C(24)	89.4 (3)	C(17)—C(18)—C(19)	122.0 (7)
C(17)—S(3)—Rh	109.1 (2)	C(17)—C(18)—C(23)	114.0 (5)
C(24)—S(3)—Rh	110.7 (2)	C(19)—C(18)—C(23)	124.0 (6)
C(6)—C(1)—C(2)	122.8 (5)	C(18)—C(19)—C(20)	118.0 (6)
C(6)—C(1)—S(1)	124.9 (4)	C(19)—C(20)—C(21)	120.8 (7)
C(2)—C(1)—S(1)	112.2 (4)	C(20)—C(21)—C(22)	119.4 (8)
C(1)—C(2)—C(3)	117.9 (6)	C(17)—C(22)—C(21)	118.9 (6)
C(1)—C(2)—C(7)	115.1 (4)	C(24)—C(23)—C(18)	107.1 (5)
C(3)—C(2)—C(7)	127.0 (5)	C(23)—C(24)—S(3)	106.9 (5)

Space group $P2_1/c$ from systematic absences ($0k0$, k odd; $h0l$, l odd). Lorentz–polarization and absorption corrections; no extinction correction. Structure solved by direct methods using *Xtal2.6* (Hall & Stewart, 1989) and refined by full-matrix least-squares calculations. Non-H atoms allowed to refine with anisotropic temperature factors. H atoms included at geometrically idealized positions (C—H 0.95 Å).

The authors wish to thank the Natural Sciences and Engineering Research Council of Canada and Alberta Sulfur Research Ltd, Calgary, Alberta, for financial support.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55522 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1009]

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Acta Cryst. (1993). C**49**, 385–387

Structure of 6-Methyl-2-oxabicyclo[4.3.0]non-4-ene-3,8-dione

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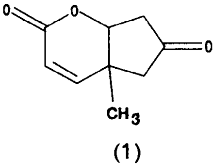
(*Received* 2 *June* 1992; *accepted* 6 *August* 1992)

Abstract

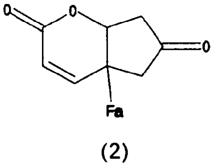
The pyrone ring adopts a C(9)-sofa conformation and the *cis*-fused five-membered ring is in a C(5)-envelope conformation. The methyl group is in a pseudo-axial position. The molecular packing involves C—H···O contacts.

Comment

During the studies of seeds of *Otoba parvifolia* (Mkfg) A. Gentry (Ferreira, 1985), some novel compounds were isolated and identified. The spectroscopic data for the bicyclic lactone (2) raised some doubts about the assignments. In order to explain the data for the natural product, the model compound (1) (in which the farnesyl group is substituted by a methyl group) was synthesized and a crystal structure determination undertaken to determine its stereochemistry unambiguously.



(1)



(2)

Fa = farnesyl

The pyrone ring is in a sofa conformation with C(9) 0.540 (3) Å out of the plane formed by the other five atoms; the carbonyl O atom O(2) lies 0.217 (3) Å out of the plane. As in other pyrones (Selladurai & Subramanian, 1992, and references therein) the angle O(2)—C(2)—O(1) is smaller than O(2)—C(2)—O(3).

The five-membered ring is in an envelope conformation with C(6), C(7), C(8), C(9) coplanar to within experimental error and C(5) lying 0.527 (3) Å out of the plane. The dihedral angle between the best least-squares planes through the rings is 63.3 (1)°.

The molecular packing involves intermolecular C—H···O interactions as listed in Table 2.

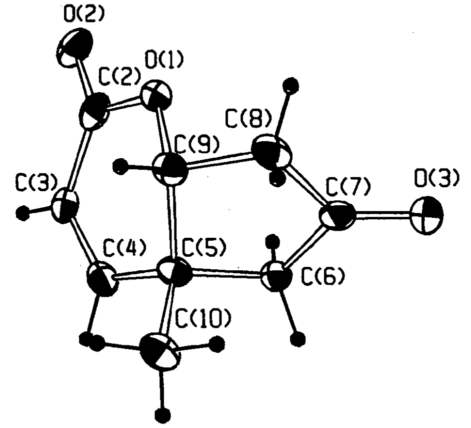


Fig. 1. The molecular structure of $C_9H_{10}O_3$ showing the atom labelling. 50% thermal ellipsoids are shown for non-H atoms.

Experimental

Crystal data

$C_9H_{10}O_3$
$M_r = 166.18$
Orthorhombic
$Pca2_1$
$a = 6.610\ (5)$ Å
$b = 11.272\ (3)$ Å
$c = 10.738\ (3)$ Å
$V = 800\ (1)$ Å^3
$Z = 4$
$D_x = 1.380$ Mg m^{-3}
Mo $K\alpha$ radiation

$\lambda = 0.70926$ Å
Cell parameters from 25 reflections
$\theta = 8$–19°
$\mu = 0.97$ mm^{-1}
$T = 83$ K
Irregular
0.38 × 0.16 × 0.05 mm
Colourless
Crystal source: from 1:9 ethyl acetate/*n*-hexane

Data collection

Rigaku AFC-5R diffractometer
ω scans
Absorption correction: none
1123 measured reflections
1123 independent reflections
799 observed reflections [$I>3\sigma(I)$]

$\theta_{\max} = 25°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 15 min
intensity variation: 0.5%

Refinement

Refinement on F^2
Final $R = 0.035$
$wR = 0.036$
$S = 1.08$
799 reflections
110 parameters
$w = [\sigma^2(|F_o|)+0.0005|F_o|^2]^{-1}$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

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

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.5156 (4)	0.6055 (2)	0.1907 (3)	1.76 (8)
O(2)	0.5057 (4)	0.4198 (2)	0.1303 (3)	2.40 (9)
O(3)	0.1608 (5)	0.9023 (2)	0.2459 (3)	2.64 (9)
C(2)	0.5265 (5)	0.5223 (3)	0.1010 (4)	1.9 (1)
C(3)	0.5492 (6)	0.5623 (3)	−0.0281 (4)	1.7 (1)
C(4)	0.5342 (6)	0.6758 (3)	−0.0584 (4)	1.9 (1)
C(5)	0.5146 (5)	0.7701 (3)	0.0377 (3)	1.5 (1)
C(6)	0.2946 (6)	0.8075 (3)	0.0627 (4)	1.7 (1)
C(7)	0.2927 (6)	0.8458 (3)	0.1961 (4)	1.8 (1)
C(8)	0.4837 (6)	0.8035 (3)	0.2580 (3)	1.9 (1)
C(9)	0.5858 (5)	0.7251 (3)	0.1652 (3)	1.7 (1)
C(10)	0.6425 (6)	0.8793 (3)	0.0039 (4)	2.1 (1)

Table 2. *Bond lengths* (Å), *valence angles* (°) *and geometrical parameters of selected intermolecular interactions* (Å, °)

O(1)—C(2)	1.346 (5)	O(1)—C(9)	1.452 (4)
O(2)—C(2)	1.205 (4)	O(3)—C(7)	1.205 (5)
C(2)—C(3)	1.465 (6)	C(3)—C(4)	1.324 (5)
C(4)—C(5)	1.487 (5)	C(5)—C(6)	1.538 (5)
C(5)—C(9)	1.534 (5)	C(5)—C(10)	1.537 (5)
C(6)—C(7)	1.496 (6)	C(7)—C(8)	1.504 (6)
C(8)—C(9)	1.493 (5)		
C(2)—O(1)—C(9)	119.7 (3)	O(1)—C(2)—O(2)	118.4 (3)
O(1)—C(2)—C(3)	117.9 (3)	O(2)—C(2)—C(3)	123.6 (4)
C(2)—C(3)—C(4)	121.5 (4)	C(3)—C(4)—C(5)	121.8 (3)
C(4)—C(5)—C(6)	113.5 (3)	C(4)—C(5)—C(9)	110.9 (3)
C(4)—C(5)—C(10)	111.1 (3)	C(6)—C(5)—C(9)	103.0 (3)
C(6)—C(5)—C(10)	110.0 (3)	C(9)—C(5)—C(10)	107.9 (3)
C(5)—C(6)—C(7)	104.7 (3)	O(3)—C(7)—C(6)	125.7 (3)
O(3)—C(7)—C(8)	125.4 (3)	C(6)—C(7)—C(8)	108.9 (3)
C(7)—C(8)—C(9)	105.8 (3)	O(1)—C(9)—C(5)	112.2 (3)
O(1)—C(9)—C(8)	106.2 (3)	C(5)—C(9)—C(8)	105.1 (3)

D—H···A	D···A	D—H	H···A	D—H···A
C(6)—H(C6)···O(2)^{i}	3.276 (5)	1.00 (4)	2.34 (4)	157 (1)
C(9)—H(C9)···O(2)^{ii}	3.243 (5)	0.90 (4)	2.55 (4)	134 (1)

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

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. H atoms were found in a difference synthesis and included as fixed contributors with an overall isotropic thermal parameter [$U = 0.0233(3)$ Å^2]. The programs used were *SHELXS86* (Sheldrick, 1985), *SHELX76* and *ORTEP* (Johnson, 1965). The refinement was by blocked-matrix least-squares methods. Most of the calculations were performed on a VAX 6420 computer at the Instituto de Física e Química de São Carlos.

This work has received partial support from FARESP, CNPq, CAPES and FINEP. One of us (JZ-S) would like to thank the Associação de Amigos do Instituto Weizmann de São Paulo for a scholarship, The Weizmann Institute of Science for providing the X-ray facilities and Dr F. Frolow for collecting the data.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55410 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1018]

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Acta Cryst. (1993). **C49**, 387–388

Structure of (*E*)-1-(4-Methoxyphenyl)-2-nitropropene

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(*Received* 16 *March* 1992; *accepted* 27 *August* 1992)

Abstract

An important feature of the molecule is its non-planarity, the nitropropene and methoxyphenyl groups exhibiting a dihedral angle of 27.1 (2)° and torsion angles about the C(1)—C(7) bond of 25.9 (4) and − 156.1 (3)°. The molecules pack to form chains with head-to-tail short contacts between the methyl and nitro groups of neighbouring molecules with O···H distances of 2.60 (4) Å and O···H—C angles of 123 (3)°. All intramolecular bonds and angles are within the expected range.

Comment

Studies by Doré & Viel (1972) have indicated that a number of *β*-nitrostyrene derivatives are cytotoxic and some of them inhibit Krebs II ascitic carcinoma in mice. Later studies by Cassels *et al.* (1982) have shown that some *β*-nitrostyrenes possess reproducible antitumor activity in the P-388 murine lymphocytic leukemia assay. Cytotoxicity of these substances has been related to the electrophilicity (Cavier *et al.*, 1978) and the Hückel bond index of the nitrovinyl double bond (Doré, Chalvet & Viel, 1976). Fungistatic actions and toxicity have been discussed by Rubinchik & Tolkachev (1976).

The title compound was prepared by Knoevenagel condensation of 4-methoxybenzaldehyde and nitroethane at reflux in acetic acid, catalyzed by butylamine; crystals were grown in methanol. Molecules are linked by C—H···O interactions. The strongest of these interactions involves the methoxy and nitro groups with a short contact C···O = 3.249 (4) and O···H = 2.60 (4) Å. Different substituents on the benzene ring significantly affect the coplanarity of the molecule. The dihedral angle of 27.1 (2)° between the methoxyphenyl and nitropropene groups in the present compound may be compared with the dihedral angles of 23.7 and 12.2° in the 4-hydroxy-3-methoxy (Zabel, Watson, Cassels & Langs, 1980) and the 3,4,5-trimethoxy (Mascarenhas, 1989) analogs respectively. In contrast the 4-dimethylamine analog exhibits a dihedral angle of only 4.8° (Brito, Manríquez, Reyes-Parada, Cassels & Rodríguez, 1991).

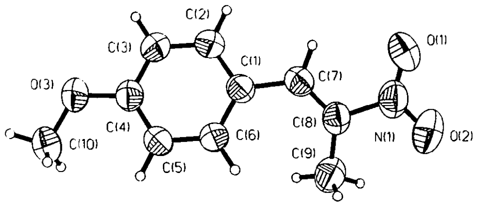

Fig. 1. Molecular structure with 50% probability ellipsoids, showing atom-numbering scheme. H atoms are drawn as circles of arbitrary radii.

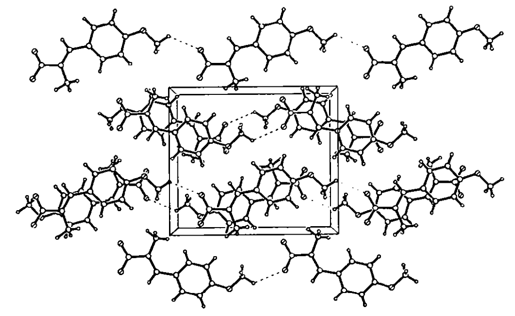

Fig. 2. Molecular packing viewed along the *a* axis, with short C—H···O contacts represented by dashed lines.

Experimental

Crystal data

$C_{10}H_{11}NO_3$
M_r = 193.2
Orthorhombic
$P2_12_12_1$
a = 7.387 (1) Å
b = 10.719 (2) Å
c = 12.325 (2) Å
V = 975.9 (3) Å^3
Z = 4
D_x = 1.315 Mg m^{-3}

Mo *Kα* radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 3.3–11.0°
μ = 0.092 mm^{-1}
T = 293 K
Parallelepiped
0.55 × 0.44 × 0.34 mm
Yellow

0108-2701/93/020387-02$06.00