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Acta Cryst. (1993). C**49**, 387–388

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

D. Boys

Departamento de Física, Facultad de Ciencias Físicas y Matemáticas, Casilla 487-3, *Universidad de Chile, Santiago, Chile*

V. Manríquez and B. K. Cassels

Departamento de Química, Facultad de Ciencias, Casilla 653, *Universidad de Chile, Santiago, Chile*

(*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).

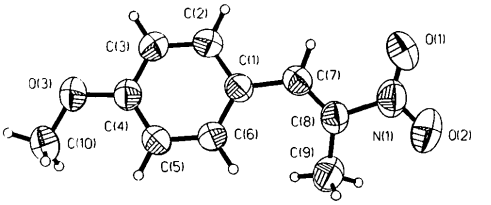

Fig. 1. Molecular structure with 50% probability ellipsoids, showing atom-numbering scheme. H atoms are drawn as circles of arbitrary radii.

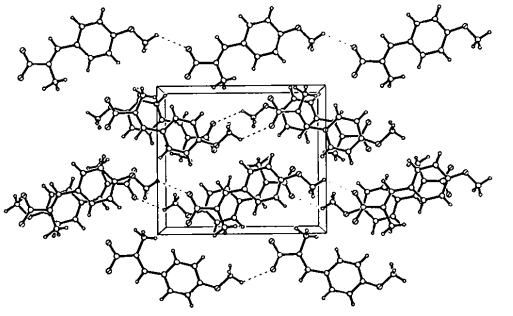

Fig. 2. Molecular packing viewed along the *a* axis, with short C—H···O contacts represented by dashed lines.

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 $K\alpha$ 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

Data collection

Siemens *R3m/V* diffractometer	$R_{int} = 0.0172$
2θ–θ scans	$\theta_{max} = 25.5°$
Absorption correction: none	$h = 0 \rightarrow 8$
2064 measured reflections	$k = 0 \rightarrow 12$
1027 independent reflections	$l = 0 \rightarrow 14$
879 observed reflections $[F > 3\sigma(F)]$	2 standard reflections monitored every 98 reflections

Refinement

Refinement on F	$w = [\sigma^2(F) + 0.0024F^2]^{-1}$
Final $R = 0.039$	$(\Delta/\sigma)_{max} = 0.030$
$wR = 0.055$	$\Delta\rho_{max} = 0.11$ e Å^{-3}
$S = 0.99$	$\Delta\rho_{min} = -0.19$ e Å^{-3}
156 parameters	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
Riding model, C—H 0.96 Å, free isotropic U; methyl H atoms located from ΔF map, refined isotropically	

Program used to solve and refine structure: *SHELXTL PLUS* (Sheldrick, 1990).

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

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
N(1)	0.1550 (4)	0.3321 (3)	1.2706 (2)	0.079 (1)
O(1)	0.1926 (5)	0.2342 (3)	1.3158 (2)	0.120 (1)
O(2)	0.1345 (5)	0.4289 (3)	1.3207 (2)	0.113 (1)
O(3)	0.1242 (3)	0.1017 (2)	0.6575 (1)	0.078 (1)
C(1)	0.1490 (4)	0.2024 (2)	0.9839 (2)	0.053 (1)
C(2)	0.1054 (4)	0.0821 (3)	0.9500 (2)	0.062 (1)
C(3)	0.0960 (3)	0.0509 (3)	0.8412 (2)	0.064 (1)
C(4)	0.1345 (3)	0.1411 (3)	0.7635 (2)	0.059 (1)
C(5)	0.1793 (3)	0.2603 (2)	0.7943 (2)	0.061 (1)
C(6)	0.1863 (3)	0.2902 (2)	0.9041 (2)	0.058 (1)
C(7)	0.1581 (3)	0.2271 (3)	1.1009 (2)	0.059 (1)
C(8)	0.1383 (3)	0.3352 (3)	1.1515 (2)	0.060 (1)
C(9)	0.0907 (5)	0.4600 (3)	1.1072 (3)	0.077 (1)
C(10)	0.1574 (6)	0.1915 (3)	0.5759 (2)	0.085 (1)
H(9A)	0.0252 (49)	0.4492 (36)	1.0402 (32)	0.105 (12)
H(9B)	0.1874 (70)	0.5031 (44)	1.1056 (46)	0.147 (20)
H(9C)	0.0136 (47)	0.5090 (30)	1.1650 (27)	0.099 (10)
H(10A)	0.1416 (52)	0.1415 (33)	0.5080 (29)	0.107 (12)
H(10B)	0.2884 (59)	0.2267 (38)	0.5831 (36)	0.119 (14)
H(10C)	0.0665 (50)	0.2533 (31)	0.5780 (31)	0.091 (11)

Table 2. *Geometric parameters* (Å, °)

N(1)—O(1)	1.221 (4)	N(1)—O(2)	1.217 (4)
N(1)—C(8)	1.474 (3)	O(3)—C(4)	1.375 (3)
O(3)—C(10)	1.414 (4)	C(1)—C(2)	1.393 (4)
C(1)—C(6)	1.389 (3)	C(1)—C(7)	1.467 (3)
C(2)—C(3)	1.384 (4)	C(3)—C(4)	1.390 (4)
C(4)—C(5)	1.373 (4)	C(5)—C(6)	1.392 (3)
C(7)—C(8)	1.324 (4)	C(8)—C(9)	1.486 (4)
C(9)—H(9A)	0.96 (4)	C(10)—H(10A)	1.00 (4)
C(9)—H(9B)	0.85 (5)	C(10)—H(10B)	1.04 (4)
C(9)—H(9C)	1.05 (3)	C(10)—H(10C)	0.94 (4)
O(1)—N(1)—O(2)	122.0 (3)	O(1)—N(1)—C(8)	119.6 (3)
O(2)—N(1)—C(8)	118.4 (3)	C(4)—O(3)—C(10)	117.2 (2)
C(2)—C(1)—C(6)	117.4 (2)	C(2)—C(1)—C(7)	118.2 (2)
C(6)—C(1)—C(7)	124.4 (2)	C(1)—C(2)—C(3)	121.7 (2)
C(2)—C(3)—C(4)	119.3 (3)	O(3)—C(4)—C(3)	115.4 (2)
O(3)—C(4)—C(5)	124.1 (2)	C(3)—C(4)—C(5)	120.4 (2)
C(4)—C(5)—C(6)	119.5 (2)	C(1)—C(6)—C(5)	121.7 (2)
C(1)—C(7)—C(8)	128.0 (2)	N(1)—C(8)—C(7)	116.1 (2)
N(1)—C(8)—C(9)	113.9 (2)	C(7)—C(8)—C(9)	129.8 (2)
H(9A)—C(9)—H(9B)	118 (4)	H(10A)—C(10)—H(10B)	112 (3)
H(9A)—C(9)—H(9C)	112 (3)	H(10A)—C(10)—H(10C)	108 (3)
H(9B)—C(9)—H(9C)	102 (4)	H(10B)—C(10)—H(10C)	114 (3)

One of the authors (DB) thanks Fundación Andes for funding the purchase of a single-crystal diffractometer.

Lists of structure factors, anisotropic thermal parameters and complete H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55486 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1007]

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Acta Cryst. (1993). C**49**, 388–391

Structure of Ethyltriphenylphosphonium Triiodide, $[P(C_2H_5)(C_6H_5)_3]I_3$

SUZANNE BÉLANGER AND ANDRÉ L. BEAUCHAMP

Département de chimie, Université de Montréal, CP 6128, *Succ. A, Montréal, Québec, Canada H3C 3J7*

(*Received* 14 *April* 1992; *accepted* 27 *August* 1992)

Abstract

The title compound was obtained unexpectedly from a mixture of $KReO_4$, triphenylphosphine and excess HI in ethanol. The crystal contains the tetrahedral $[P(C_2H_5)(C_6H_5)_3]^+$ cation in which the P—C distances range from 1.789 (13) to 1.819 (14) Å and the C—P—C angles from 108.3 (6) to 110.5 (6)°. The I_3^-

0108-2701/93/020388-04$06.00