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Structure of (E)-1-(4-Methoxyphenyl)-2-Nitropropene

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Abstract

An important feature of the molecule is its non-planarity, the nitropropane and methoxyphenyl groups exhibiting a dihedral angle of 27.1° and torsion angles about the C(1)-C(7) bond of 26.6 and -155.8°. 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 2.528(4)Å and O-H-C angles of 132.0(3)°. All intramolecular bonds and angles are within the expected range.

Comment

Studies by Doré & Viel (1972) have indicated that a number of ß-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 ß-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 has been discussed by Rubinchik & Tolkachev (1976).

The present compound was prepared by Knoevenagel condensation of 4-methoxybenzaldehyde and nitroethane at reflux in acetic acid, catalyzed by butylamine; crystals were grown in methanol. Structural work on closely related compounds: (E)-1-(4-Hydroxy-3-Methoxyphenyl)-2-Nitropropene by Zabel, Watson, Cassels & Langs (1980), and (E)-1-(4-Dimethylaminophenyl)-2-Nitropropene by Brito, Manríquez, Reyes-Parada, Cassels & Rodríguez (1991).

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Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No SUP.... (4 pp). Copies may be obtained through the Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2H2, England.

Experimental

Crystal Data

Empirical Formula	$C_{10} H_{11} N O_3$
Color; Habit	yellow parallelepiped
Crystal size (mm)	0.44 x 0.34 x 0.55
Crystal System	Orthorhombic
Space Group	$P2_12_12_1$
Unit Cell Dimensions	a = 7.387(1) Å
	b = 10.719(2) Å
	c = 12.325(2) Å
Cell Parameters from	24 reflections, $6.6 \leq 2\theta \leq 22.0^\circ$
Volume	975.9(3) $Å^3$
Z	4
Formula weight	193.2
Density(calc.)	1.315 Mg/m^3
Absorption Coefficient	0.092 mm^{-1}
F(000)	408

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	293
Monochromator	Highly oriented graphite crystal
2θ Range	3.0 to 51.0°
Scan Type	2θ-θ
Scan Speed	Variable; 4.19 to 29.30°/min. in ω
Standard Reflections	2 measured every 98 reflections
Correction Factors Applied from Standards	1.0000 - 0.9543
Index Ranges	$0 \leq h \leq 8$, $0 \leq k \leq 12$, $0 \leq \ell \leq 14$, and Friedel opposites
Reflections Collected	2064
Independent Reflections	1027 (R_{int} = 1.72%)
Observed Reflections	879 ($F > 3.0\sigma(F)$)
Absorption Correction	No

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version) (Sheldrick,1990)
Solution	Direct Methods
Refinement Method	on F; Full-Matrix Least-Squares
Quantity Minimized	$\Sigma w(F_o - F_c)^2$
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0026F^2$
Hydrogen atoms	Riding model, C - H = 0.96Å, free isotropic U
Number of Parameters Refined	138
Final R Indices (obs. data)	R = 0.043 , wR = 0.065
R Indices (all data)	R = 0.049 , wR = 0.069
Goodness-of-Fit S	1.13
Largest and Mean Δ/σ	0.017, 0.004
Largest Difference Peak,Hole	0.15 $eÅ^{-3}$, -0.16 $eÅ^{-3}$

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TABLE CAPTIONS

Table 1. Atomic coordinates and equivalent isotropic temperature factors ($Å^2$).

Table 2. Geometric parameters (Å, °).

FIGURE CAPTIONS

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

Fig. 2. Molecular packing viewed along the a axis, with H-bonds in dotted lines.

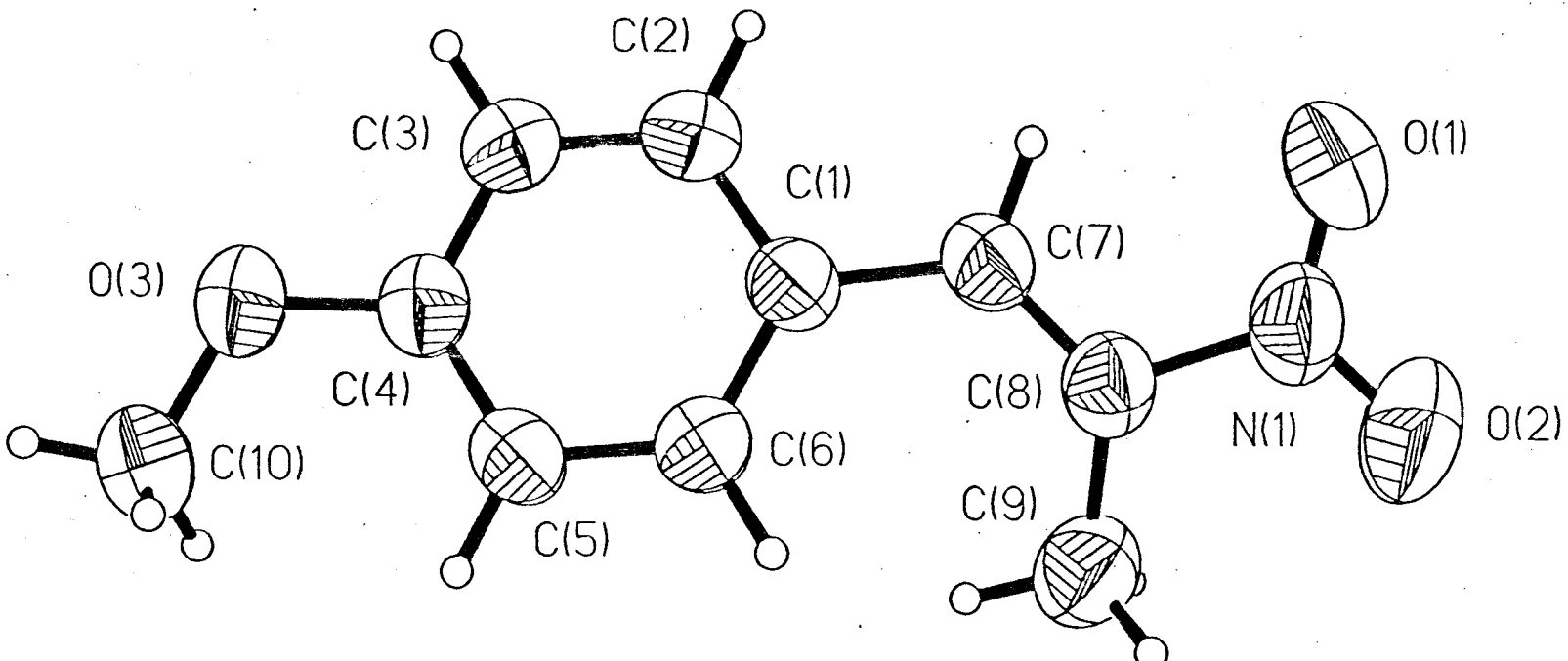
	x	y	z	U(eq)*
N(1)	0.1549(4)	0.3322(3)	1.2701(2)	0.078(1)
O(1)	0.1921(6)	0.2338(3)	1.3156(2)	0.120(1)
O(2)	0.1336(5)	0.4288(3)	1.3208(2)	0.115(1)
O(3)	0.1250(3)	0.1018(2)	0.6573(2)	0.077(1)
C(1)	0.1487(4)	0.2026(3)	0.9836(2)	0.053(1)
C(2)	0.1054(4)	0.0820(3)	0.9502(2)	0.062(1)
C(3)	0.0966(4)	0.0512(3)	0.8411(2)	0.063(1)
C(4)	0.1347(4)	0.1415(3)	0.7635(2)	0.059(1)
C(5)	0.1796(4)	0.2605(3)	0.7943(2)	0.060(1)
C(6)	0.1864(4)	0.2902(3)	0.9041(2)	0.059(1)
C(7)	0.1582(4)	0.2276(3)	1.1011(2)	0.058(1)
C(8)	0.1376(4)	0.3351(3)	1.1511(2)	0.060(1)
C(9)	0.0907(5)	0.4601(3)	1.1074(3)	0.078(1)
C(10)	0.1576(6)	0.1911(3)	0.5758(2)	0.085(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

N(1)-O(1)	1.226(5)	1.279*	N(1)-O(2)	1.220(5)	1.266*
N(1)-C(8)	1.473(4)		O(3)-C(4)	1.378(3)	
O(3)-C(10)	1.408(4)		C(1)-C(2)	1.394(4)	
C(1)-C(6)	1.386(4)		C(1)-C(7)	1.474(4)	
C(2)-C(3)	1.386(4)		C(3)-C(4)	1.390(4)	
C(4)-C(5)	1.372(4)		C(5)-C(6)	1.391(4)	
C(7)-C(8)	1.316(4)		C(8)-C(9)	1.485(5)	

O(1)-N(1)-O(2)	121.7(3)	O(1)-N(1)-C(8)	119.6(3)
O(2)-N(1)-C(8)	118.7(3)	C(4)-O(3)-C(10)	117.3(3)
C(2)-C(1)-C(6)	117.7(2)	C(2)-C(1)-C(7)	118.1(2)
C(6)-C(1)-C(7)	124.2(3)	C(1)-C(2)-C(3)	121.3(3)
C(2)-C(3)-C(4)	119.5(3)	O(3)-C(4)-C(3)	115.3(3)
O(3)-C(4)-C(5)	124.3(3)	C(3)-C(4)-C(5)	120.4(3)
C(4)-C(5)-C(6)	119.4(3)	C(1)-C(6)-C(5)	121.8(3)
C(1)-C(7)-C(8)	127.9(3)	N(1)-C(8)-C(7)	115.9(3)
N(1)-C(8)-C(9)	113.7(3)	C(7)-C(8)-C(9)	130.3(3)

* Corrected bond length for riding motion


C(2)
C(3)
C(1)
O(1)
C(7)
O(3)
C(4)
C(8)
N(1)
O(2)
C(10)
C(6)
C(9)
C(5)