Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methoxy-3-nitrophenol

David M. Behrman,[a]* E. J. Behrman[b] and Sean Parkin[c]

[a]Somerset Community College, 808 Monticello St., Somerset, KY 42501, USA, [b]Department of Biochemistry, Ohio State University, Columbus, OH 43210, USA, and [c]Department of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA

Correspondence e-mail: david.behrman@kctcs.edu

Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.104
Data-to-parameter ratio = 8.6

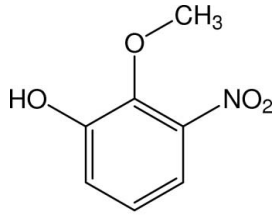
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 19 June 2006
Accepted 21 June 2006

The crystal structure of 2-methoxy-3-nitrophenol, $C_7H_7NO_4$, is stabilized by hydrogen bonds and π–π stacking interactions.

Comment

The title compound, (I), was first synthesized by Oxford (1926) as the only unknown mononitro derivative of guaiacol. It was later reported in a US patent (Peterson, 1946) concerned with the bactericidal properties of mercurated derivatives of mononitroguaiacols. Recently, one of the present authors (EJB) synthesized it as an intermediate in the synthesis of 3,6-dinitrocatechol (Behrman *et al.*, 2002). A new synthesis has just been published (Zhao & Snieckus, 2005) but with an incorrect m.p. (personal communication from the authors). It has been variously called 3-nitroguaiacol and 6-nitroguaiacol. The compound crystallizes from carbon disulfide in beautiful needles which seem limited in length only by the size of the flask. Oxford (1926) was also impressed by the crystals and provides a detailed description of their habit. The present report resulted from a class project for a course in X-ray crystallography. Given the relatively simple molecular formula, two of us were surprised that the crystal structure had not been reported. The crystals are quite fragile, such that attempts to cut the needles invariably led to lengthwise splintering. The crystal packing provides a reasonable explanation for this and thus accounts for the crystal shape.



(I)

Molecules related by the 2_1 screw axis are hydrogen bonded (Table 1), forming chains running parallel to the *c* axis. Within these chains, alternate molecules are arranged into stacks in which the π–π interplanar spacing is 3.315 (3) Å.

Experimental

2-Methoxy-3-nitrophenol was synthesized from *o*-methoxyphenyl acetate by treating with acetyl chloride and silver nitrate following the precise directions of Oxford (1926). Pale-yellow crystals were obtained upon crystallization from carbon disulfide (m.p. 342–343 K). See Zhao & Snieckus (2005) for NMR data.

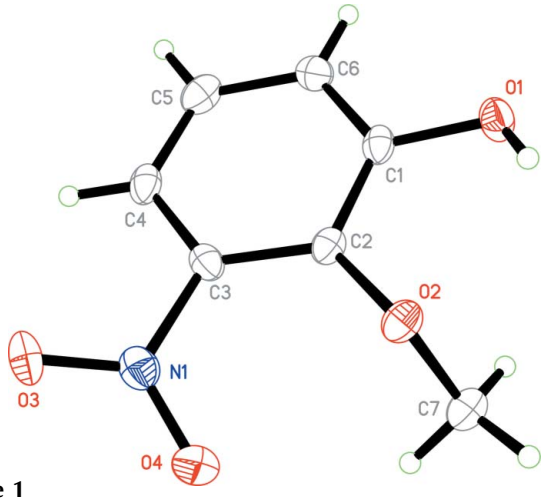


Figure 1
A view of the title compound, with displacement ellipsoids drawn at the 50% probability level.

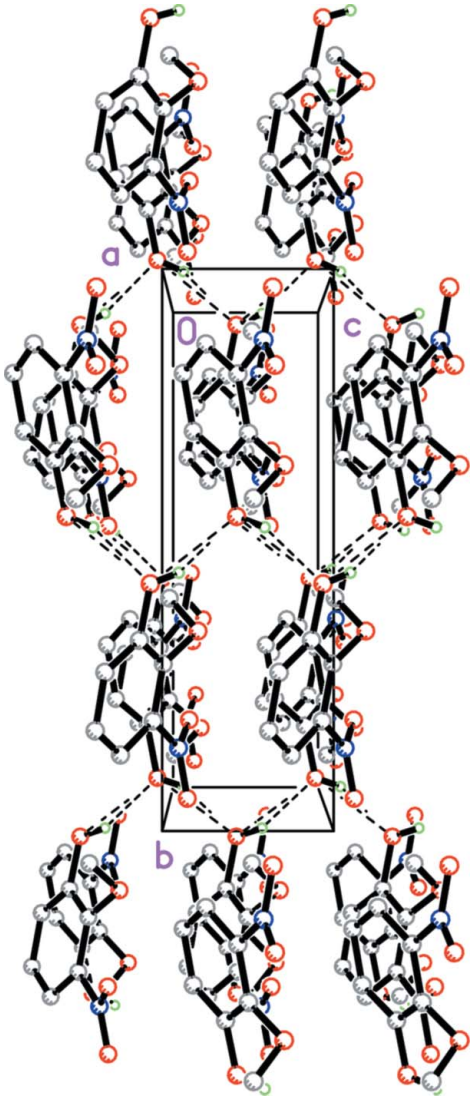


Figure 2
A projection of the crystal structure along the *a* axis, showing the hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

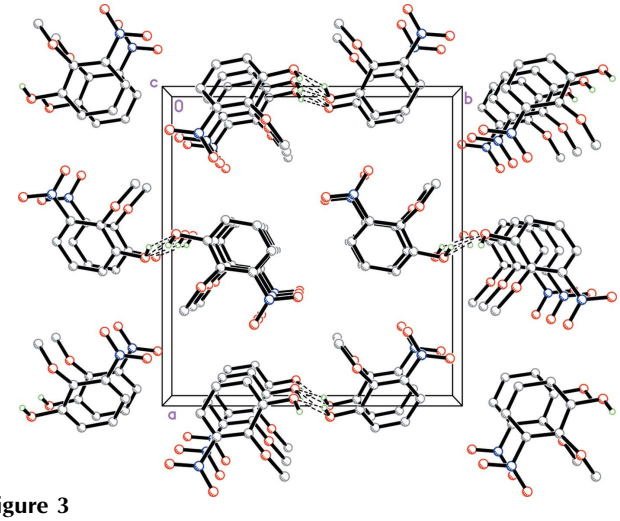


Figure 3
A projection of the crystal structure along the *c* axis, showing the hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Crystal data

$C_7H_7NO_4$
M_r = 169.14
Orthorhombic, $Pna2_1$
a = 13.9581 (2) Å
b = 13.1337 (6) Å
c = 4.0110 (7) Å
V = 735.30 (13) Å^3
Z = 4
D_x = 1.528 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 90.0 (2) K
Needle, pale yellow
0.50 × 0.15 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.94, T_{max} = 0.99
1613 measured reflections
958 independent reflections
751 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 1.06
958 reflections
112 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.017 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1^{i}	0.84	1.97	2.743 (2)	153
O1—H1···O2	0.84	2.29	2.720 (2)	112

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

C-bound H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C). The hydroxy H atom was located in a difference map and refined as riding, with O—H = 0.84 Å and with U_{iso}(H) = 1.5U_{eq}(O).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to

solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97* and local procedures.

References

Behrman, E. C., Chen, S. & Behrman, E. J. (2002). *Tetrahedron Lett.* **43**, 3221–3224.

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Oxford, A. E. (1926). *J. Chem. Soc.* pp. 2004–2011.

Peterson, E. A. (1946). US Patent No. 2 392 801.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1994). *SHELXTL/PC*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

Zhao, Z. & Snieckus, V. (2005). *Org. Lett.* **7**, 2523–2526 supplementary material, p. 8.