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1-Ethoxy-4-nitrobenzene

Brian McBurney, Paul C. D. Foss, Elizabeth M. Reed, Timothy D. Shine, Neil M. Glagovich, Barry L. Westcott, Guy Crundwell, Matthias Zeller and Allen D. Hunter

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Brian McBurney,^a Paul C. D. Foss,^a Elizabeth M. Reed,^a Timothy D. Shine,^a Neil M. Glagovich,^a Barry L. Westcott,^a Guy Crundwell,^{a*} Matthias Zeller^b and Allen D. Hunter^b

^aDepartment of Chemistry, Central Connecticut State University, New Britain, CT 06053, USA, and ^bDepartment of Chemistry, Youngstown State University, One University Plaza, Youngstown, Ohio 44555-3663, USA

Correspondence e-mail:
crundwellg@mail.ccsu.edu

Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.048
wR factor = 0.129
Data-to-parameter ratio = 14.2

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

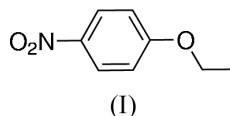
1-Ethoxy-4-nitrobenzene

The title compound, $\text{C}_8\text{H}_9\text{NO}_3$, was synthesized from 4-nitrophenol and ethyl iodide *via* the Williamson ether synthesis. The crystal structure has been determined at 100 K.

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Comment

1-Ethoxy-4-nitrobenzene, (I) (Fig. 1), also known as *p*-nitrophenetole, was synthesized by undergraduate chemistry students as part of an undergraduate laboratory course. The students were preparing a variety of ethers in an effort to design a laboratory that could be incorporated into the undergraduate organic chemistry curriculum. Of the ethers prepared, 1-ethoxy-4-nitrobenzene required the shortest reaction time, gave the best yield, and was the least problematic in terms of isolation. In addition, upon standing, a solution of (I) in ethanol produced very large and well defined crystals. Because the crystal structure of this ether has not been previously reported, it was decided that it would be an interesting extension of the laboratory experience to have the students in the class solve the structure from the X-ray diffraction data.



Experimental

To a 100 ml round-bottomed flask equipped with a reflux condenser were added 15 ml of an 8% (*w/w*) sodium hydroxide solution, *p*-nitrophenol (2.78 g, 19 mmol), ethyl iodide (3.2 g, 21 mmol) and acetone (25 ml). The resulting mixture was heated under reflux for 90 min. After this time, the resulting yellow solution was poured over 75 g of ice, and stirred until the ice melted. The crude yellow solid was collected by vacuum filtration and recrystallized from 95% ethanol to yield *p*-nitrophenetole (yield: 2.64 g, 83%; m.p. 330.2 K). The NMR data agree with the published literature (Heathcock, 1962). IR (CHCl_3): 3115, 3098, 2987, 1609, 1597, 1497, 1474, 1342, 1329, 1302, 1261, 1176, 1090, 1041, 920, 860, 851, 751, 655 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 8.204 (*d*, 2H, $J = 10.5$ Hz), 6.944 (*d*, 2H, $J = 10.5$ Hz), 4.130 (*q*, 2H, $J = 7.0$ Hz), 1.465 (*t*, 3H, $J = 7.0$ Hz); ^{13}C NMR

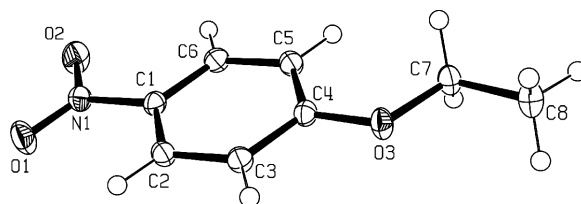


Figure 1
A view of (I) (ORTEP-3; Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level.

(300 MHz, CDCl₃): δ 164.19, 141.39, 125.89, 114.48, 64.53, 14.56; MS (calculated for C₈H₉NO₃): M⁺: 167, measured: 167.

Crystal data

C ₈ H ₉ NO ₃	$D_x = 1.423 \text{ Mg m}^{-3}$
$M_r = 167.16$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6367 reflections
$a = 7.2796 (5) \text{ \AA}$	$\theta = 2.2\text{--}28.3^\circ$
$b = 11.7664 (8) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 9.4285 (6) \text{ \AA}$	$T = 100 (2) \text{ K}$
$\beta = 104.957 (1)^\circ$	Block, colorless
$V = 780.23 (9) \text{ \AA}^3$	$0.45 \times 0.45 \times 0.30 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART APEX diffractometer	1936 independent reflections
ω scans	1894 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$R_{\text{int}} = 0.021$
$T_{\text{min}} = 0.948, T_{\text{max}} = 0.969$	$\theta_{\text{max}} = 28.3^\circ$
8103 measured reflections	$h = -9 \rightarrow 9$
	$k = -15 \rightarrow 15$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.3353P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.129$	$(\Delta/\sigma)_{\text{max}} = 0.020$
$S = 1.22$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
1936 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
136 parameters	
All H-atom parameters refined	

Positional and isotropic displacement parameters for all H atoms were allowed to refine after their location in a difference map.

Data collection: *SMART* (Bruker, 1997–1999); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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