Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-(*p*-Nitrophenoxy)tetrahydropyran

Reenu Chopra,[a]* Ning Shan,[a] W. D. Sam Motherwell[b] and William Jones[a]

[a]Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, England, and [b]Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, England

Correspondence e-mail: rc305@cam.ac.uk

Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.121
Data-to-parameter ratio = 17.0

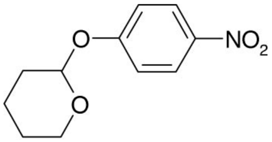
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 September 2004
Accepted 23 September 2004
Online 9 October 2004

The title compound, $C_{11}H_{13}NO_4$ forms supramolecular sheets parallel to (001) *via* C—H···O hydrogen bonds. Sheets stack along the *c* axis *via* additional C—H···O interactions.

Comment

As part of a continuing study of the decomposition kinetics of 2-(*p*-nitrophenoxy)tetrahydropyran, (I), in amorphous saccharides, we have determined the crystal structure of (I) at 180 K. Compound (I) was synthesized by a modification of the procedure of Fife & Jao (1968) (see *Experimental*). Crystals of (I), as a racemic mixture, were obtained from its solution in hexane at room temperature.



(I)

The asymmetric unit of (I) consists of only one molecule. Two-dimensional networks (Fig. 2) perpendicular to the *c* axis are formed *via* C2—H2···O4 and C9—H9*B*···O2 hydrogen bonds (Table 1). These two-dimensional networks then stack along the *c* axis, linked by further C7—H7···O1 interactions.

Experimental

3,4-Dihydro-2*H*-pyran and *p*-nitrophenol were obtained from Aldrich and Avocado, respectively, and were used without further purification. Toluene, bought from Aldrich, was further dried over sodium wire. *p*-Nitrophenol (0.1 mol) was dissolved in dry toluene (100 ml) and an excess of 3,4 dihydro-2*H*-pyran (30 ml) was added to the solution. The resulting solution was stirred under reflux at 378 K for 3 d. The reaction mixture was then diluted with ether, followed by washing with 2% NaOH several times to remove the unreacted *p*-nitrophenol. The organic layer, dried over Na_2SO_4, was then filtered

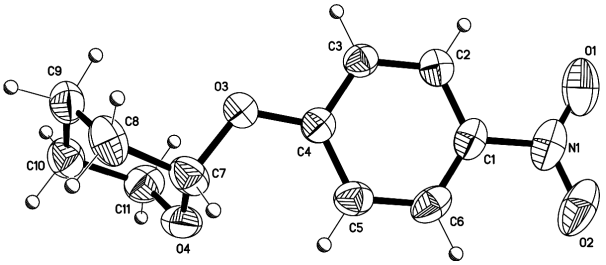


Figure 1
The molecule of (I), showing displacement ellipsoids at the 50% probability level.

and evaporated. Crystals of (I) were obtained by dissolving the crude sample in hexane followed by slow evaporation at room temperature.

Crystal data

$C_{11}H_{13}NO_4$
$M_r = 223.22$
Monoclinic, $P2_1/c$
$a = 7.4772$ (1) Å
$b = 21.9462$ (4) Å
$c = 6.7828$ (1) Å
$\beta = 102.491$ (1)°
$V = 1086.69$ (3) Å^3
$Z = 4$
$D_x = 1.364$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12872 reflections
$\theta = 1.0$–27.5°
$\mu = 0.11$ mm^{-1}
$T = 180$ (2) K
Block, pale yellow
0.46 × 0.23 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.891$, $T_{\max} = 0.984$
13336 measured reflections
2476 independent reflections
1970 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -28 \rightarrow 28$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.121$
$S = 1.08$
2476 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.2284P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.061 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4$^{\rm i}$	0.95	2.40	3.1783 (16)	139
C7—H7···O1$^{\rm ii}$	1.00	2.41	3.3956 (18)	170
C9—H9*B*···O2$^{\rm iii}$	0.99	2.52	3.2930 (19)	135

Symmetry codes: (i) $x-1, y, z$; (ii) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined using a riding model, with the $U_{\rm iso}$ values for each H atom taken as $1.2U_{\rm eq}$ of the carrier atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL*97.

The authors are grateful to the Pfizer Institute for Pharmaceutical Materials Science for funding the research. RC also acknowledges a Cambridge Commonwealth Trust and ORS Award. We thank Dr John Davies for assistance with data collection and structure solution and the EPSRC for financial assistance towards the purchase of the Nonius KappaCCD diffractometer.

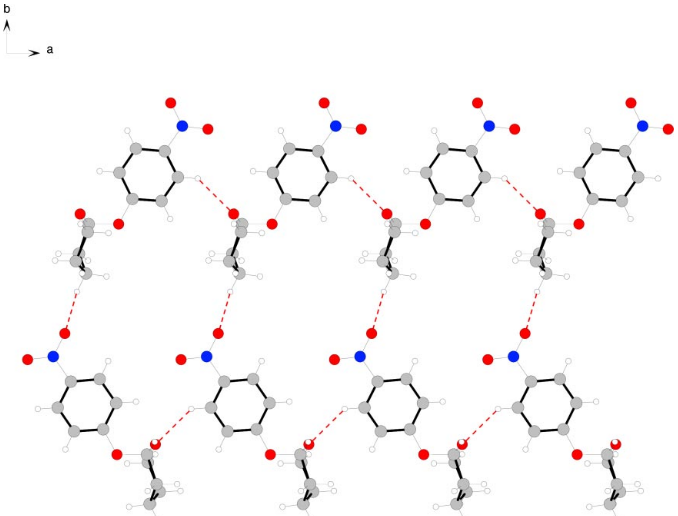


Figure 2
The two-dimensional supramolecular network formed by C—H···O hydrogen bonds (dashed lines) perpendicular to the *c* axis.

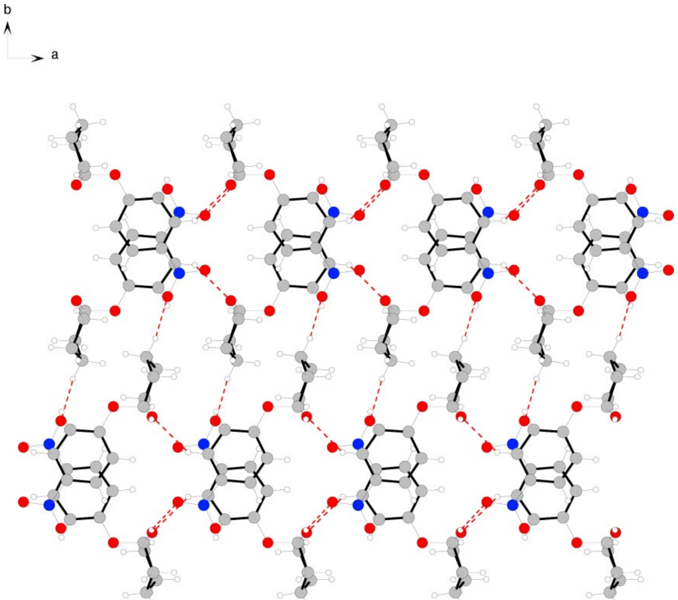


Figure 3
Projection on to (001), showing the two-dimensional networks stacking along the *c* axis.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Brandenburg, K. (1999). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.
Fife, T. H. & Jao, L. K. (1968). *J. Am. Chem. Soc.* **90**, 4081–4085.
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 and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1993). *XP*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

o1924 Reenu Chopra *et al.* • $C_{11}H_{13}NO_4$ *Acta Cryst.* (2004). E**60**, o1923–o1924