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2-(*p*-Nitrophenoxy)tetrahydropyran

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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.

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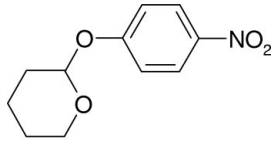
The title compound, $C_{11}H_{13}NO_4$ forms supramolecular sheets parallel to (001) *via* C—H···O hydrogen bonds. Sheets stack along the *c* axis *via* additional C—H···O interactions.

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

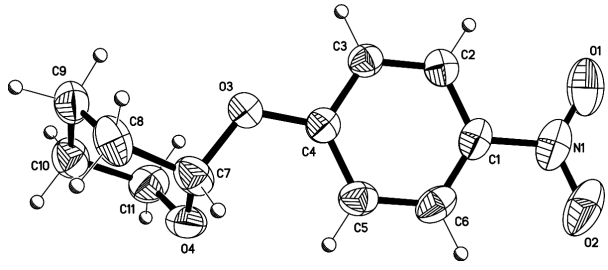


Figure 1
The molecule of (I), showing displacement ellipsoids at the 50% probability level.

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
β = 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
θ = 1.0–27.5°
μ = 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_{int} = 0.034
θ_{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^{i}	0.95	2.40	3.1783 (16)	139
C7—H7···O1^{ii}	1.00	2.41	3.3956 (18)	170
C9—H9*B*···O2^{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_{iso} values for each H atom taken as $1.2U_{eq}$ 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.

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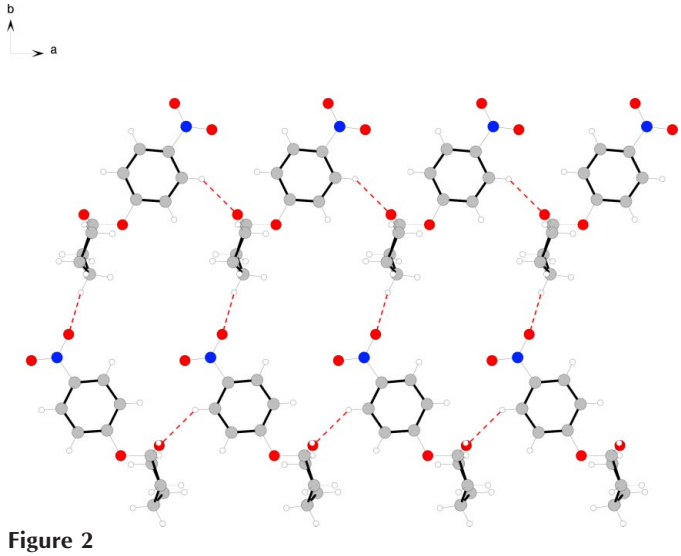

Figure 2
The two-dimensional supramolecular network formed by C—H···O hydrogen bonds (dashed lines) perpendicular to the c axis.

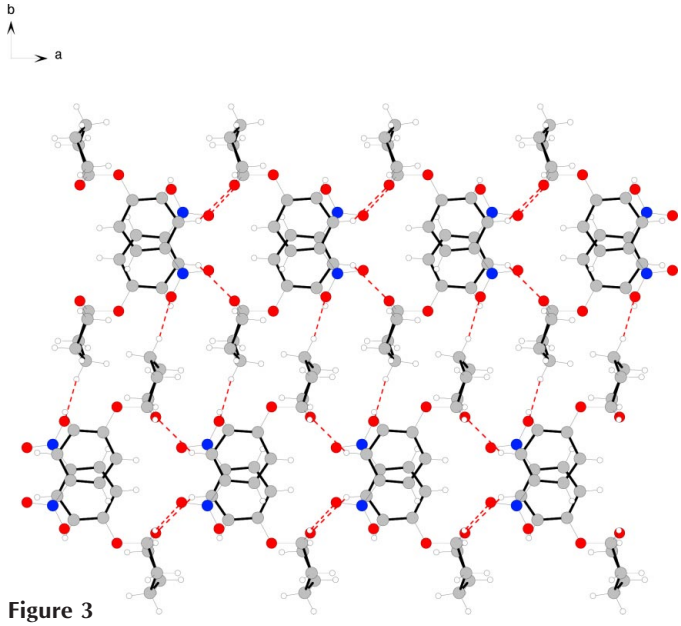

Figure 3
Projection on to (001), showing the two-dimensional networks stacking along the c axis.

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supporting information

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2-(*p*-Nitrophenoxy)tetrahydropyran

Reenu Chopra, Ning Shan, W. D. Sam Motherwell and William Jones

2-(*p*-Nitrophenoxy)terahydropyran

Crystal data

$C_{11}H_{13}NO_4$
$M_r = 223.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.4772$ (1) Å
$b = 21.9462$ (4) Å
$c = 6.7828$ (1) Å
$\beta = 102.491$ (1)°
$V = 1086.69$ (3) Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.364$ Mg m^{-3}
Melting point: 332 K
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 12872 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.11$ mm^{-1}
$T = 180$ K
Plate, pale yellow
0.46 × 0.23 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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 $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.6°$
$h = -9 \rightarrow 9$
$k = -28 \rightarrow 28$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.121$
$S = 1.08$
2476 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.061 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.0794 (2)	0.31572 (6)	0.83905 (18)	0.0541 (4)
O1	−0.08743 (19)	0.30900 (6)	0.81357 (19)	0.0712 (4)
O2	0.1547 (2)	0.36578 (5)	0.8542 (2)	0.0771 (4)
O3	0.49426 (12)	0.10316 (4)	0.89300 (14)	0.0412 (3)
O4	0.74541 (13)	0.12513 (5)	0.75296 (16)	0.0515 (3)
C1	0.19406 (19)	0.26134 (6)	0.85389 (18)	0.0395 (3)
C2	0.10857 (18)	0.20498 (6)	0.83302 (19)	0.0382 (3)
H2	−0.0211	0.2020	0.8097	0.046*
C3	0.21496 (17)	0.15338 (6)	0.84666 (19)	0.0364 (3)
H3	0.1585	0.1144	0.8331	0.044*
C4	0.40482 (17)	0.15789 (6)	0.88016 (18)	0.0350 (3)
C5	0.48881 (18)	0.21476 (6)	0.8987 (2)	0.0423 (3)
H5	0.6183	0.2179	0.9199	0.051*
C6	0.3819 (2)	0.26676 (6)	0.8859 (2)	0.0444 (3)
H6	0.4376	0.3059	0.8992	0.053*
C7	0.69021 (17)	0.10322 (7)	0.9249 (2)	0.0462 (4)
H7	0.7411	0.1306	1.0414	0.055*
C8	0.7531 (2)	0.03892 (8)	0.9800 (2)	0.0529 (4)
H8A	0.6943	0.0240	1.0882	0.063*
H8B	0.8873	0.0389	1.0332	0.063*
C9	0.7073 (2)	−0.00382 (7)	0.8016 (2)	0.0486 (4)
H9A	0.5732	−0.0103	0.7642	0.058*
H9B	0.7666	−0.0438	0.8384	0.058*
C10	0.7730 (2)	0.02266 (7)	0.6232 (2)	0.0504 (4)
H10A	0.9085	0.0243	0.6542	0.060*
H10B	0.7325	−0.0037	0.5033	0.060*
C11	0.6961 (2)	0.08599 (7)	0.5787 (2)	0.0506 (4)
H11A	0.7432	0.1036	0.4656	0.061*
H11B	0.5609	0.0837	0.5366	0.061*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0908 (10)	0.0414 (7)	0.0316 (6)	0.0167 (7)	0.0162 (6)	0.0050 (5)
O1	0.0788 (9)	0.0680 (8)	0.0628 (8)	0.0356 (7)	0.0064 (6)	0.0026 (6)
O2	0.1359 (13)	0.0348 (6)	0.0680 (8)	0.0119 (6)	0.0382 (8)	0.0075 (5)

O3	0.0356 (5)	0.0401 (5)	0.0492 (6)	0.0012 (3)	0.0121 (4)	0.0002 (4)
O4	0.0435 (5)	0.0500 (6)	0.0657 (7)	−0.0119 (4)	0.0219 (5)	−0.0089 (5)
C1	0.0592 (8)	0.0350 (7)	0.0253 (6)	0.0061 (5)	0.0113 (5)	0.0018 (5)
C2	0.0420 (7)	0.0421 (7)	0.0309 (6)	0.0036 (5)	0.0085 (5)	0.0027 (5)
C3	0.0399 (6)	0.0354 (6)	0.0345 (6)	−0.0028 (5)	0.0094 (5)	0.0009 (5)
C4	0.0405 (6)	0.0366 (6)	0.0291 (6)	−0.0004 (5)	0.0101 (5)	−0.0007 (5)
C5	0.0431 (7)	0.0464 (8)	0.0386 (7)	−0.0091 (5)	0.0117 (5)	−0.0057 (6)
C6	0.0657 (9)	0.0362 (7)	0.0332 (7)	−0.0112 (6)	0.0151 (6)	−0.0037 (5)
C7	0.0338 (7)	0.0586 (9)	0.0446 (8)	−0.0008 (6)	0.0052 (5)	−0.0105 (6)
C8	0.0450 (7)	0.0688 (10)	0.0434 (8)	0.0164 (7)	0.0063 (6)	0.0034 (7)
C9	0.0473 (7)	0.0473 (8)	0.0522 (8)	0.0113 (6)	0.0133 (6)	0.0061 (6)
C10	0.0499 (8)	0.0542 (9)	0.0499 (8)	0.0040 (6)	0.0171 (6)	−0.0048 (7)
C11	0.0597 (9)	0.0501 (8)	0.0467 (8)	−0.0045 (6)	0.0218 (7)	0.0014 (6)

Geometric parameters (Å, º)

N1—O2	1.2284 (18)	C5—H5	0.9500
N1—O1	1.2308 (19)	C6—H6	0.9500
N1—C1	1.4600 (17)	C7—C8	1.509 (2)
O3—C4	1.3684 (15)	C7—H7	1.0000
O3—C7	1.4340 (15)	C8—C9	1.511 (2)
O4—C7	1.4033 (18)	C8—H8A	0.9900
O4—C11	1.4430 (18)	C8—H8B	0.9900
C1—C6	1.379 (2)	C9—C10	1.517 (2)
C1—C2	1.3855 (18)	C9—H9A	0.9900
C2—C3	1.3754 (17)	C9—H9B	0.9900
C2—H2	0.9500	C10—C11	1.510 (2)
C3—C4	1.3918 (17)	C10—H10A	0.9900
C3—H3	0.9500	C10—H10B	0.9900
C4—C5	1.3905 (18)	C11—H11A	0.9900
C5—C6	1.385 (2)	C11—H11B	0.9900
O2—N1—O1	123.43 (14)	O3—C7—H7	108.7
O2—N1—C1	118.27 (15)	C8—C7—H7	108.7
O1—N1—C1	118.29 (13)	C7—C8—C9	112.19 (12)
C4—O3—C7	118.57 (10)	C7—C8—H8A	109.2
C7—O4—C11	114.06 (11)	C9—C8—H8A	109.2
C6—C1—C2	121.65 (12)	C7—C8—H8B	109.2
C6—C1—N1	120.21 (13)	C9—C8—H8B	109.2
C2—C1—N1	118.14 (13)	H8A—C8—H8B	107.9
C3—C2—C1	118.73 (12)	C8—C9—C10	110.15 (13)
C3—C2—H2	120.6	C8—C9—H9A	109.6
C1—C2—H2	120.6	C10—C9—H9A	109.6
C2—C3—C4	120.47 (12)	C8—C9—H9B	109.6
C2—C3—H3	119.8	C10—C9—H9B	109.6
C4—C3—H3	119.8	H9A—C9—H9B	108.1
O3—C4—C5	125.25 (11)	C11—C10—C9	109.71 (12)
O3—C4—C3	114.53 (11)	C11—C10—H10A	109.7

C5—C4—C3	120.22 (12)	C9—C10—H10A	109.7
C6—C5—C4	119.37 (13)	C11—C10—H10B	109.7
C6—C5—H5	120.3	C9—C10—H10B	109.7
C4—C5—H5	120.3	H10A—C10—H10B	108.2
C1—C6—C5	119.54 (12)	O4—C11—C10	111.43 (13)
C1—C6—H6	120.2	O4—C11—H11A	109.3
C5—C6—H6	120.2	C10—C11—H11A	109.3
O4—C7—O3	110.46 (11)	O4—C11—H11B	109.3
O4—C7—C8	113.22 (12)	C10—C11—H11B	109.3
O3—C7—C8	106.88 (12)	H11A—C11—H11B	108.0
O4—C7—H7	108.7		

O2—N1—C1—C6	0.57 (18)	C2—C1—C6—C5	−0.31 (19)
O1—N1—C1—C6	−178.88 (12)	N1—C1—C6—C5	−179.69 (11)
O2—N1—C1—C2	−178.83 (12)	C4—C5—C6—C1	−0.4 (2)
O1—N1—C1—C2	1.72 (18)	C11—O4—C7—O3	−66.70 (15)
C6—C1—C2—C3	0.60 (19)	C11—O4—C7—C8	53.12 (15)
N1—C1—C2—C3	179.99 (11)	C4—O3—C7—O4	−69.12 (14)
C1—C2—C3—C4	−0.21 (19)	C4—O3—C7—C8	167.31 (11)
C7—O3—C4—C5	−1.07 (18)	O4—C7—C8—C9	−50.17 (16)
C7—O3—C4—C3	179.24 (10)	O3—C7—C8—C9	71.68 (15)
C2—C3—C4—O3	179.24 (11)	C7—C8—C9—C10	50.86 (16)
C2—C3—C4—C5	−0.47 (19)	C8—C9—C10—C11	−54.19 (16)
O3—C4—C5—C6	−178.91 (11)	C7—O4—C11—C10	−57.01 (15)
C3—C4—C5—C6	0.77 (19)	C9—C10—C11—O4	56.86 (16)

Hydrogen-bond geometry (Å, º)

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

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) *x*+1, −*y*+1/2, *z*+1/2; (iii) −*x*+1, *y*−1/2, −*z*+3/2.