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trans-(4-Phenyltetrahydro-2-furyl)methyl *p*-nitrobenzoate

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Key indicators

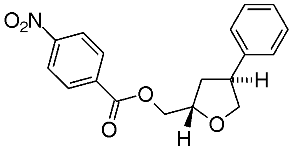
Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.005 Å
R factor = 0.074
wR factor = 0.244
Data-to-parameter ratio = 14.9

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

The tetrahydrofuran ring in *trans*-4*R*(*S*)-phenyltetrahydro-2*R*(*S*)-furylmethyl *p*-nitrobenzoate, $C_{18}H_{17}NO_5$, adopts a $_1T^5$ conformation in the solid state. The phenyl group and the functionalized CH_2 substituent are located in pseudo-equatorial positions.

Comment

trans-4-Phenyltetrahydrofuryl-2-methyl *p*-nitrobenzoate, (I), has two asymmetrically substituted C atoms, C2 and C4. The racemate of (I) has been prepared and investigated (Fig. 1).



(I)

The tetrahydrofuran ring in (I) adopts a twist conformation, with atoms O1 and C5 displaced in opposite directions from the plane defined by atoms C2, C3, and C4 [0.16 (1) Å for O1 and −0.40 (1) Å for C5], leading to a $_1T^5$ arrangement in the solid state [O1—C2—C3—C4 6.7 (5)° and C5—C4—C3—C2 16.0 (4)°]. This geometry is not expected to be an energetic minimum in the liquid phase, predominantly for reasons of torsional strain (Romers *et al.*, 1969).

The observed tetrahydrofuran conformation positions the phenyl and the functionalized CH_2 substituent into pseudo-equatorial locations (Hartung, Kneuer *et al.*, 2003; Fuchs, 1978) [O1—C5—C4—C7 −166.6 (3)° and C4—C3—C2—C6 127.6 (4)°]. The O1—C2—C6—O2 torsion angle of −57.6 (5)° is in agreement with the preferred *gauche* arrangement of diacceptor-substituted ethane entities (Kirby, 1983). The ester functionality in (I) exhibits *Z* geometry [O3—C13—O2—C6 2.9 (5)°].

The C2—C6 bond appears to be unusually short for a single C—C bond [1.408 (6) Å] due to the intense thermal motion. The ^{1}H and ^{13}C NMR spectra, though, leave no doubt as to the sp^3 character of both C atoms.

Experimental

p-Nitrobenzoyl chloride (91.0 mg, 0.49 mmol) was added at 273 K to 53.0 mg (0.33 mmol) of a solution of 2-hydroxymethyl-4-phenyltetrahydrofuran [50:50 mixture of *cis*/*trans*-diastereomers (Hartung, Drees *et al.*, 2003)] and 73.3 mg (0.65 mmol) of 1,4-diazabicyclo[2.2.2]octane in 5 ml of CH_2Cl_2 (Hartung *et al.*, 1997). The reaction mixture was stirred for 10 min. The precipitate was filtered off and the filtrate was washed with dilute HCl (2 × 2 ml). The aqueous phase was extracted with CH_2Cl_2 (2 × 2 ml). The combined organic phases were dried ($MgSO_4$) and concentrated under reduced pressure. The residue was purified by chromatography [SiO_2, petroleum ether/acetone = 3:2 (*v*/*v*)] to furnish 101 mg (94%) of 4-phenyl-2-tetrahydrofurylmethyl *p*-nitrobenzoate as a colourless oil (*cis*:*trans* = 50:50). This material was crystallized from petroleum ether (2 ml) and diethyl ether (0.2 ml) to afford 38 mg (24%) of compound (I) as a diastereomerically pure product (colourless prisms, m.p. 324 K). The stereochemical purity of this material was verified by analyzing *ca* 20 mg of (I) by ^{1}H and ^{13}C NMR spectroscopy, which confirmed the presence of a single diastereomer in solution. The relative *trans* configuration of this compound was derived from additional results of nuclear Overhauser effect experiments. Analytical data for *trans*-4-phenyl-2-tetrahydrofurylmethyl *p*-nitrobenzoate, (I), calculated: C 66.04, H 5.23, N 4.28; found: C 66.11, H 5.25, N 4.23; ^{1}H NMR (250 MHz, $CDCl_3$, δ_H, p.p.m.): 2.25 (*dd*, J = 7.0 and 7.9 Hz, 2H), 3.54 (m_c, 1H), 3.87 (*dd*, J = 6.3 and 8.6 Hz, 1H), 4.31 (*dd*, J = 7.0 and 8.6 Hz, 1H), 4.46 (m_c, 2H), 4.55 (m_c, 1H), 7.21–7.39 (*m*, 5H), 8.18–8.37 (*m*, 4H); ^{13}C NMR (63 MHz, $CDCl_3$, δ_C, p.p.m.): 36.3, 44.5, 67.8, 74.9, 77.4, 123.6, 126.7, 126.9, 127.1, 127.2, 128.7, 130.8, 141.5, 164.6.

Crystal data

$C_{18}H_{17}NO_5$
M_r = 327.33
Monoclinic, $P2_1/c$
a = 6.993 (1) Å
b = 14.490 (2) Å
c = 16.274 (3) Å
β = 96.07 (2)°
V = 1639.8 (4) Å^3
Z = 4

D_x = 1.326 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 3.6–12.8°
μ = 0.10 mm^{-1}
T = 299 (2) K
Prism, colourless
0.75 × 0.60 × 0.43 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3886 measured reflections
3212 independent reflections
2250 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

θ_{max} = 26.0°
$h = -8 \rightarrow 1$
$k = -17 \rightarrow 0$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity decay: 2.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.244
S = 1.06
3212 reflections
215 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1114P)^2 + 0.9016P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

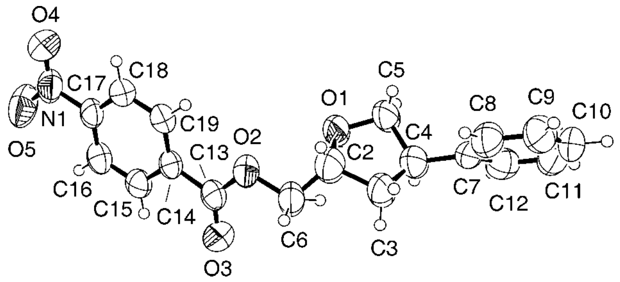


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

All H atoms were positioned geometrically and included in the refinement in a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$ of the corresponding carrier atom.

Data collection: *CAD*-4 *Diffractometer Control Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *Diffractometer Control Software*; data reduction: *CAD*-4 *Diffractometer Control Software;* program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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