Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-Phenylcyclohexyl 3-furancarboxylate

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.221
Data-to-parameter ratio = 13.9

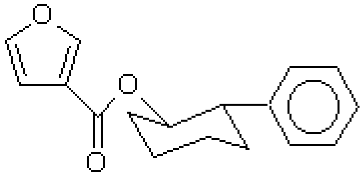
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 July 2001
Accepted 23 July 2001
Online 31 July 2001

The crystal structure of the title compound, $C_{17}H_{18}O_3$, contains molecules that are separated by normal van der Waals distances. The cyclohexyl ring adopts a classical chair conformation, while the furan and phenyl rings are individually planar. The molecular dimensions are as expected.

Comment

The intramolecular Diels–Alder reaction with a furandiene (IMDAF reaction) is a useful reaction for the creation of three rings with up to five stereogenic centers in one reaction (Keay & Hunt, 1999). To date, no-one has reported an asymmetric version of this reaction. *trans*-2-Arylcyclohexanol esters of 3-furoic acid were prepared as potential chiral auxiliaries for studying the asymmetric IMDAF reaction. *AM*1 calculations indicated that the large aryl ring would preferentially block one face of the furandiene thereby leading to high diastereoselectivity in the IMDAF reaction. The structure of 2-phenylcyclohexyl 3-furancarboxylate, (I), was determined to establish if, in the solid state, the large aryl ring would block one face of the furan ring.



(I)

The molecular dimensions are normal and lie within expected values for corresponding bond distances and angles (Orpen *et al.*, 1994). The C1–C6 cyclohexyl ring adopts a classical chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q = 0.568$ (5) Å, $\theta = 177.3$ (5)° and $\omega = 55$ (9)°. The phenyl ring is essentially planar with the maximum deviation of any atom from the mean plane being 0.007 (3) Å. The five-membered furan ring is also planar. The structure is devoid of any undesirable interactions.

Experimental

To a mixture of 3-furoic acid (111 mg, 0.99 mmol) in dry methylene chloride (2 ml) and dry DMF (5 µl) at 273 K under a nitrogen atmosphere, oxalyl chloride (86 µl, 1.1 equivalents) was added. The solution was stirred at 273 K for 15 min, then at room temperature for 3 h. after which the solvent was removed *in vacuo*. After redissolving the residue in dry methylene chloride (2 ml), a solution of *trans*-2-phenylcyclohexanol (164 mg, 0.93 mmol) in dry methylene chloride

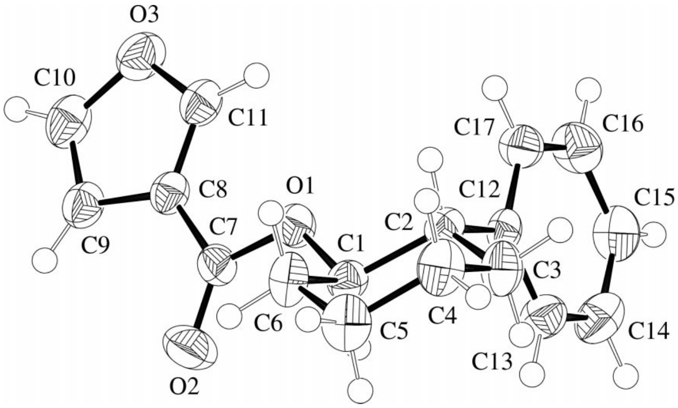


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I). Displacement ellipsoids have been plotted at the 50% probability level.

(2 ml) was added followed by dry pyridine (80 µl). The reaction mixture was stirred at room temperature overnight. After adding CH_2Cl_2 (20 ml), the solution was washed with 5% HCl (25 ml). Et_2O (20 ml) was added, then the organic phase was washed with water (25 ml), dried over $MgSO_4$, then filtered and evaporated *in vacuo* to give the crude product as an oil. Purification *via* a short column (silica gel, EtOAc) followed by radial chromatography (EtOAc/hexanes, 7:1) gave the desired ester, (I).

Crystal data

$C_{17}H_{18}O_3$
$M_r = 270.31$
Monoclinic, $P2_1/n$
$a = 5.5332$ (11) Å
$b = 12.841$ (2) Å
$c = 20.088$ (3) Å
$\beta = 96.52$ (2)°
$V = 1418.1$ (4) Å^3
$Z = 4$

$D_x = 1.266$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0$–$15.0°$
$\mu = 0.09$ mm^{-1}
$T = 200$ (2) K
Block, colourless
0.50 × 0.40 × 0.22 mm

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
2796 measured reflections
2520 independent reflections
1156 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.06$
$\theta_{\rm max} = 25.0°$

$h = 0 \rightarrow 6$
$k = 0 \rightarrow 15$
$l = -23 \rightarrow 23$
3 standard reflections every 200 reflections intensity decay: <1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.221$
$S = 1.02$
2520 reflections
181 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.12P)^2 + 0.066P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.19$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Most of the H atoms were located from difference maps. The H atoms were included at geometrically idealized positions with C—H = 0.95–1.00 Å, in a riding mode with isotropic displacement parameters 1.2 times the displacement parameters of the atoms to which they were attached.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

The authors thank the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary.

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