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2-(1-Naphthyl)cyclohexyl 3-furancarboxylate

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Key indicators

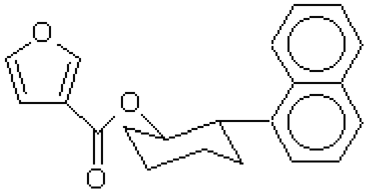
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.009 Å
R factor = 0.053
wR factor = 0.242
Data-to-parameter ratio = 14.0

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

The crystal structure of the title compound, $C_{21}H_{20}O_3$, contains molecules separated by normal van der Waals distances. The cyclohexyl ring adopts a classical chair conformation, while the furan and naphthyl rings are individually planar. The molecular dimensions are as expected.

Comment

3-Furoate *trans*-2-arylcyclohexanol esters were prepared as model compounds as part of an investigation to create a chiral auxiliary for use in an asymmetric intramolecular Diels–Alder reaction with a furandiene (IMDAF reaction) (Keay & Hunt, 1999). Molecular modeling had suggested that such systems might be able to block one face of the furandiene. The model compound, 2-(1-naphthyl)cyclohexyl 3-furancarboxylate, (I), was prepared and the structure determined in order to verify the molecular modeling results.



(I)

The crystal structure of (I) is composed of molecules (Fig. 1) which are separated by normal van der Waals distances. 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 = 0.563 (6) Å, θ = 177.7 (6)° and ω = 123 (21)°. The naphthyl ring is essentially planar, with the maximum deviation of any atom from the mean plane being 0.027 (4) Å. The five-membered furan ring is also planar.

Experimental

To a solution of 3-furoic acid (76 mg, 0.68 mmol) in dry methylene chloride (2.5 ml) and dry DMF (5 µl), oxalyl chloride (120 µl, 1.36 mmol) was added. The solution was refluxed overnight, then cooled and the solvent removed in vacuo. After redissolving the residue in dry methylene chloride (2.5 ml), a solution of *trans*-2-(1-naphthyl)cyclohexanol (136 mg, 0.9 equivalents) in dry methylene chloride (2 ml) was added followed by dry triethylamine (189 µl) and DMAP (9 mg). The reaction was stirred at room temperature for 36 h. After adding Et_2O (25 ml), the solution was washed with 5%

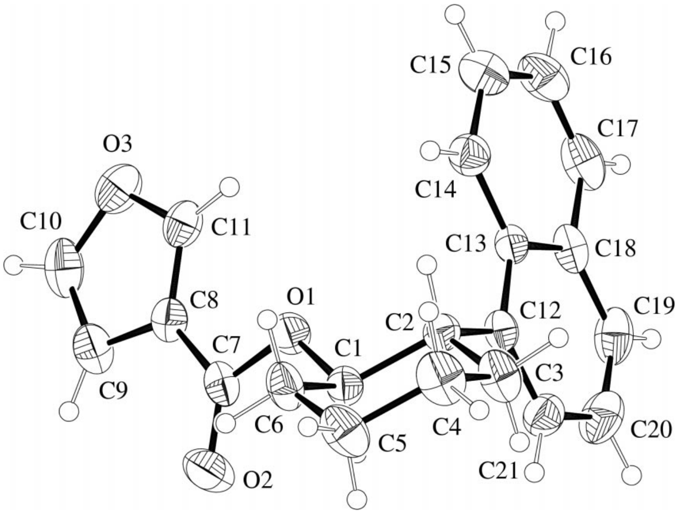


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I). Displacement ellipsoids have been plotted at the 30% probability level.

HCl (10 ml) and water (20 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, 20:1 to 5:1), gave recovered *trans*-2-(1-naphthyl)cyclohexanol (41 mg) and the title product (I) (135 mg) as a white crystalline solid (m.p. 370 K).

Crystal data

$C_{21}H_{20}O_3$
$M_r = 320.37$
Monoclinic, $P2_1/c$
$a = 9.1991$ (11) Å
$b = 7.3840$ (5) Å
$c = 25.694$ (3) Å
$\beta = 100.030$ (9)°
$V = 1718.6$ (3) Å^3
$Z = 4$

$D_x = 1.238$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0$–15.0°
$\mu = 0.08$ mm^{-1}
$T = 296$ (2) K
Prism, colourless
0.40 × 0.30 × 0.24 mm

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
3245 measured reflections
3043 independent reflections
917 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.08$
$\theta_{\rm max} = 25.0°$

$h = 0 \rightarrow 10$
$k = 0 \rightarrow 8$
$l = -30 \rightarrow 30$
3 standard reflections every 200 reflections intensity decay: <0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.242$
$S = 0.96$
3043 reflections
217 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1064P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ 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 = 0.93–0.98 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).

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