organic papers

2-(1-Naphthyl)cyclohexyl 2-methylfuran-3-carboxylate

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The crystal structure of the title compound, $C_{22}H_{22}O_3$, contains molecules that are separated by normal van der Waals distances. The cyclohexyl ring adopts a classical chair conformation, while the furan and naphthyl rings are individually planar. The molecular dimensions are as expected.

Comment

In our continuing study to develop an asymmetric version of an intramolecular Diels–Alder reaction with a furandiene (IMDAF; Keay & Hunt, 1999), we required the synthesis of a variety of 3-furoate *trans*-2-arylcyclohexanol esters. *AM*1 calculations had indicated that the aryl group should block one face of the furan diene and we hoped an X-ray crystal structure would verify this observation. Thus, the structure of 2-(1-naphthyl)cyclohexyl 2-methylfuran-3-carboxylate, (I), was determined and is presented here.
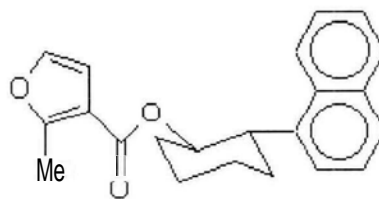
(I)

The crystal structure of (I) is composed of molecules (Fig. 1) that 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) $Q = 0.574$ (3) Å, $\vartheta = 1.3$ (3)° and $\omega = 246$ (11)°. The naphthyl ring is essentially planar, with the maximum deviation of any atom from the mean plane being 0.034 (3) Å. The five-membered furan ring is also planar. The structure is devoid of any undesirable interactions.

Experimental

A mixture of *trans*-2-(1-naphthyl)cyclohexanol (50 mg, 0.22 mmol), distilled 2-methyl-3-furoic acid (28 mg, 0.22 mmol), DCC (50 mg, 1.1 equivalents), DMAP (3 mg, 10 mol%) and PPTS (3 mg, 5 mol%) in dry CH_2Cl_2 (1 ml) was stirred at room temperature under a nitrogen atmosphere for 48 h. After adding more CH_2Cl_2, the mixture was filtered through a silica pad, then washed with 5% HCl (10 ml). The organic phase was dried over $MgSO_4$, then filtered and the solvent
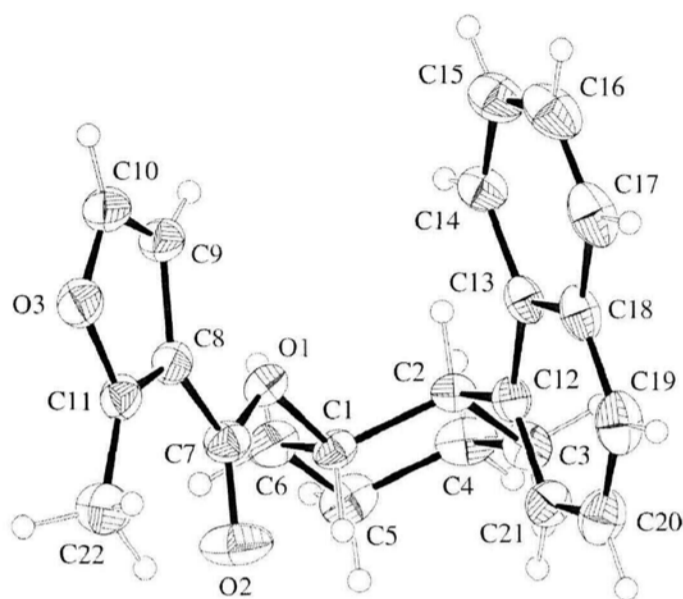
Figure 1
ORTEPll (Johnson, 1976) drawing of (I). Displacement ellipsoids have been plotted at 30% probability level.

removed *in vacuo* to give the title compound, (I), in the form of an oil that crystallized on standing in the freezer. X-ray quality crystals of (I) were grown from hexane.

Crystal data

$C_{22}H_{22}O_3$	$D_x = 1.226$ Mg m^{-3}
$M_r = 334.40$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25
$a = 21.774$ (3) Å	reflections
$b = 10.694$ (2) Å	$\theta = 10.0–15.0°$
$c = 18.430$ (3) Å	$\mu = 0.08$ mm^{-1}
$\beta = 122.380$ (10)°	$T = 200$ (2) K
$V = 3624.2$ (10) Å^3	Prism, colourless
$Z = 8$	0.60 x 0.50 x 0.30 mm

Data collection

Rigaku AFC-6S diffractometer	$h = -25 \rightarrow 0$
$\omega/2\theta$ scans	$k = -12 \rightarrow 0$
3299 measured reflections	$l = -18 \rightarrow 21$
3209 independent reflections	3 standard reflections
1641 reflections with $I > 2\sigma(I)$	every 200 reflections
$R_{int} = 0.03$	intensity decay: <0.1%
$\theta_{max} = 25.0°$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.051P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$+ 3.6P]$
$wR(F^2) = 0.146$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
3209 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
227 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	

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 (non-methyl) and 1.5 (methyl) times the displacement parameters of the atoms to which they were attached and a torsional parameter was refined for the methyl group.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSCIAFC 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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