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(3*RS*,4*RS*)-3-(2-Furyl)-2-phenethyl-4-(pyrrolidin-1-ylcarbonyl)-3,4-dihydroisoquinolin-1(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.010 Å
R factor = 0.055
wR factor = 0.129
Data-to-parameter ratio = 7.8

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

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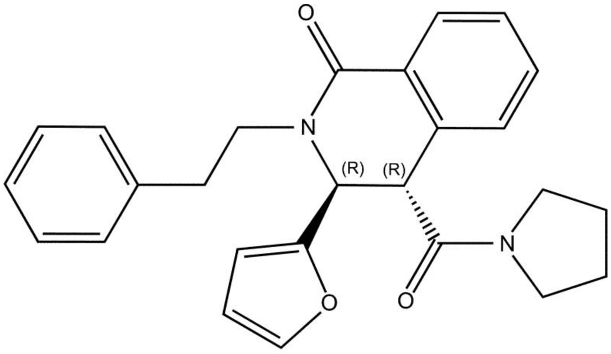
The title compound, $C_{26}H_{26}N_2O_3$, crystallizes as a racemic mixture of *R*,*R* and *S*,*S* enantiomers. The two independent molecules show a similar conformation. They are connected *via* C—H···O and C—H···π interactions to form ribbons along the *a* axis.

Comment

2,3,4-trisubstituted tetrahydroisoquinolines are important from both a technological and an applied point of view as part of a significant class of compounds with a variety of pharmacological applications (Jayaraman *et al.*, 2000; Zhang *et al.*, 1997; Bernan *et al.*, 1994; Koleva *et al.*, 1998). In the course of our research on the crystal structure and biological activity of heterocyclic molecules, the title compound, (I), was synthesized. Although the presence of pyrrolidine and furyl substituents suggests biological activity of this compound it has not been confirmed experimentally. In this paper, we describe the crystal structure of (I).



(I)

There are two independent molecules, *A* and *B* in the asymmetric unit of (I) (Fig. 1). The chemical scheme corresponds to the *R*,*R* isomer. All intramolecular bond distances and angles are within expected ranges (Gzella *et al.*, 2002; Georgieva *et al.*, 1994). In molecules *A* and *B*, the aromatic and furyl rings are nearly planar, the pyridinone rings adopt a twist conformation, and the pyrrolidine rings show an envelope conformation (Table 1). At the same time, according to IUPAC terminology (Moss, 1996), both furyl and pyrrolidine groups are in pseudo-axial positions with respect to the dihydroisoquinoline group.

Molecules *A* and *B* participate in similar three-dimensional molecular packing interactions (Fig. 2). Symmetry-equivalent molecules are linked *via* weak C—H···O interactions (Table 2) between one of the furyl C atoms and the dihydroisoquinolinone O atom, forming zigzag chains along the *a* axis.

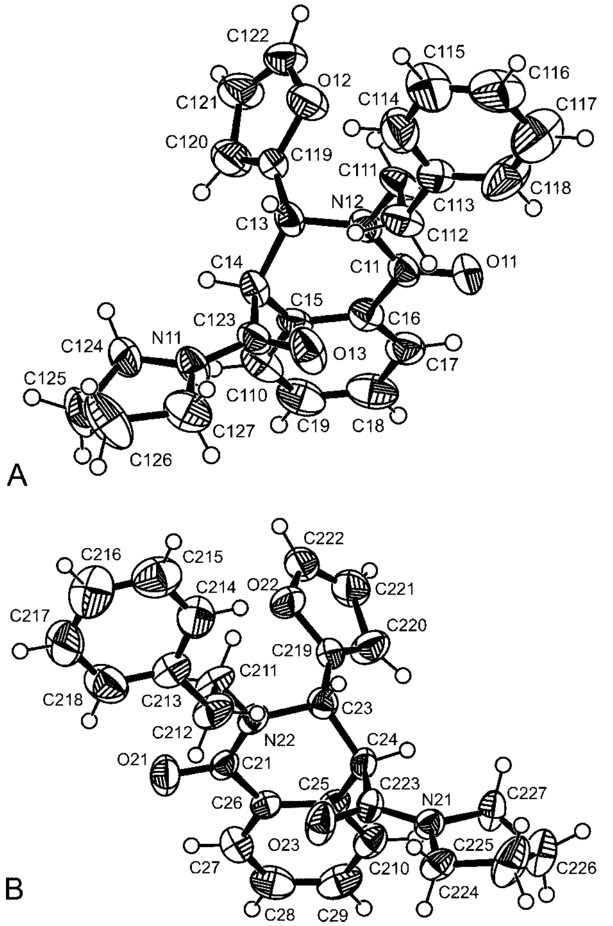


Figure 1
The structures of the two independent molecules, *A* and *B*, in (I), showing 50% probability displacement ellipsoids.

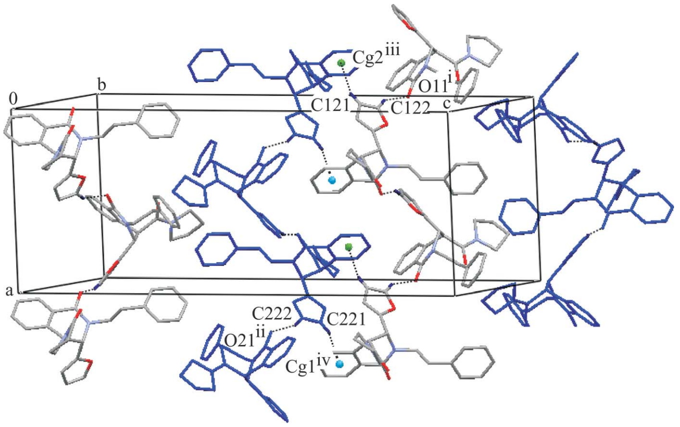


Figure 2
The molecular packing, with *B* molecules colored in blue. The dotted lines indicate C—H···π and C—H···O interactions. [Symmetry codes: (i) $x - \frac{1}{2}$, $2 - y$, z; (ii) $\frac{1}{2} + x$, $1 - y$, z; (iii) $x - 1$, y, z; (iv) $1 + x$, y, z.]

Molecules *A* and *B* are connected *via* intermolecular C—H$_{\rm furyl}$···π interactions (Table 2) to form almost-centrosymmetric dimer units. The angles between the normal to the plane of the aromatic unit and the line linking the C atom and the centroid of the ring are 22.1 (2) and 21.0 (1)° in molecules *A* and *B*, respectively.

Experimental

Compound (I) was synthesized by the well known reaction between homophthalic anhydride and imine (Haimova *et al.*, 1977; Kozekov *et al.*, 2002; Stoyanova *et al.*, 2003), leading to the corresponding *trans*- and *cis*-carboxylic acids and their subsequent transformations by analogy to Haimova *et al.* (1984). The product was characterized by IR, ^{1}H and ^{13}C NMR spectra. Crystals of (I) were obtained by slow evaporation (about five months) of a dimethylformamide solution.

Crystal data

$C_{26}H_{26}N_2O_3$
$M_r = 414.49$
Orthorhombic, $Pca2_1$
$a = 11.9675$ (15) Å
$b = 11.066$ (2) Å
$c = 32.7891$ (14) Å
$V = 4342.3$ (10) Å^3
$Z = 8$
$D_x = 1.268$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 reflections
$\theta = 16.1$–17.3°
$\mu = 0.08$ mm^{-1}
$T = 290$ (2) K
Prism, yellow
0.26 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
non–profiled $\omega/2\theta$ scans
Absorption correction: none
8655 measured reflections
4339 independent reflections
2189 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.076$
$\theta_{\rm max} = 26.0°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 13$
$l = -40 \rightarrow 40$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.129$
$S = 0.98$
4339 reflections
559 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}

Table 1
Selected torsion angles (°).

C14—C13—N12—C11	31.5 (8)	C24—C23—N22—C21	−36.8 (7)
N12—C13—C14—C15	−46.2 (6)	N22—C23—C24—C25	49.4 (7)
C13—C14—C15—C16	33.9 (7)	C23—C24—C25—C26	−34.0 (7)
C124—C125—C126—C127	−36.0 (10)	N21—C224—C225—C226	−28.4 (8)
C125—C126—C127—N11	28.4 (9)	C224—C225—C226—C227	33.2 (8)

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the dihydroisoquinoline aromatic rings of molecules *A* and *B*, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C122—H122···O11^{i}	0.93	2.49	3.342 (8)	153
C222—H222···O21^{ii}	0.93	2.57	3.454 (8)	160
C121—H121···*Cg*2^{iii}	0.93	2.89	3.681 (6)	147
C221—H221···*Cg*1^{iv}	0.93	2.92	3.756 (5)	149

Symmetry codes: (i) $x - \frac{1}{2}, -y + 2, z$; (ii) $x + \frac{1}{2}, -y + 1, z$; (iii) $x - 1, y, z$; (iv) $x + 1, y, z$.

The H atoms were placed in idealized positions (C—H = 0.93–0.97 Å) and were constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Friedel-pair reflections were merged, since anormalous scattering effects were negligible.

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Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997) *MERCURY* (Version 1.3; Bruno *et al.*, 2002); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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