

2-Benzoyl-3-cyclohexyl-4-(*p*-methoxyphenyl)-5-phenylpyrrolidine-3-spiro-3'-chroman-4'-oneG. Usha,<sup>a</sup> S. Selvanayagam,<sup>a</sup>  
M. Yogavel,<sup>a</sup> D. Velmurugan,<sup>a\*</sup>  
A. Amalraj,<sup>b</sup> R. Raghunathan,<sup>b</sup>  
S. Shanmuga Sundara Raj<sup>c</sup> and  
H.-K. Fun<sup>d</sup><sup>a</sup>Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, <sup>b</sup>Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, <sup>c</sup>B3121 Medical Centre North, VanderBelt University, Department of Medicine-Nephrology, Nashville, USA, and <sup>d</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: d\_velu@yahoo.com

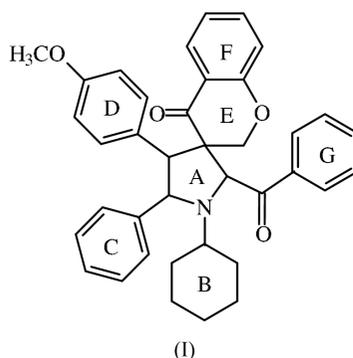
## Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
R factor = 0.047  
wR factor = 0.137  
Data-to-parameter ratio = 10.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{38}\text{H}_{37}\text{NO}_4$ , crystallizes with two molecules in the asymmetric unit. The molecules have similar geometric parameters and represent opposite enantiomers of the title compound. In both molecules, the pyrrolidine ring adopts an envelope conformation; the pyran ring in the chromanone moiety has a half-chair conformation. The molecular packing in the crystal is stabilized by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions, in addition to van der Waals forces.

## Comment

Substituted pyrrolidines have gained much importance in recent years, since they are the basic structural elements of many alkaloids and pharmacologically active compounds (Jeyabharathi *et al.*, 2002). Pyrrolidine derivatives possess anti-influenza virus (Stylianakis *et al.*, 2003) and anti-oncogenic (Obniska *et al.*, 2002) activity. The title compound, (I), a pyrrolidine derivative, was chosen for crystallographic study to determine its structure and function.



The asymmetric unit of (I) contains two molecules (Fig. 1); their corresponding bond lengths and bond angles are in good agreement.

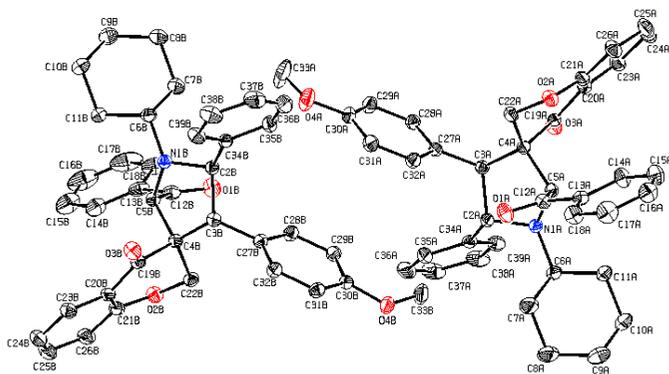
All the  $\text{C}-\text{C}$  bond lengths in the aromatic rings (C, D, F and G) and the cyclohexyl ring (B) are comparable with the corresponding literature values of 1.384 (13) and 1.539 (16)  $\text{Å}$ , respectively (Allen *et al.*, 1987). The bond lengths in the pyrrolidine ring (A) are comparable with those reported earlier (Jeyabharathi *et al.*, 2001, 2002; Abdul Ajees *et al.*, 2002; Gzella & Wrzeciono, 1990). The bond lengths in the pyran ring (E) in the chromanone moiety are also close to those found in related structures (Jeyabharathi *et al.*, 2002; Abdul Ajees *et al.*, 2001).

The title compound has four chiral centres and crystallizes in the non-centrosymmetric space group  $P2_12_12_1$ . The lack of

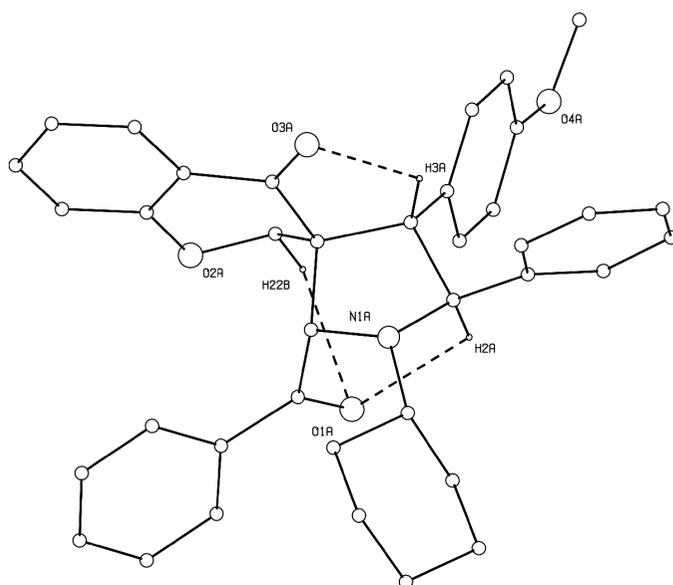
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**Figure 1**  
The molecular structure of the title compound, showing 30% probability displacement ellipsoids; H atoms have been omitted for clarity.

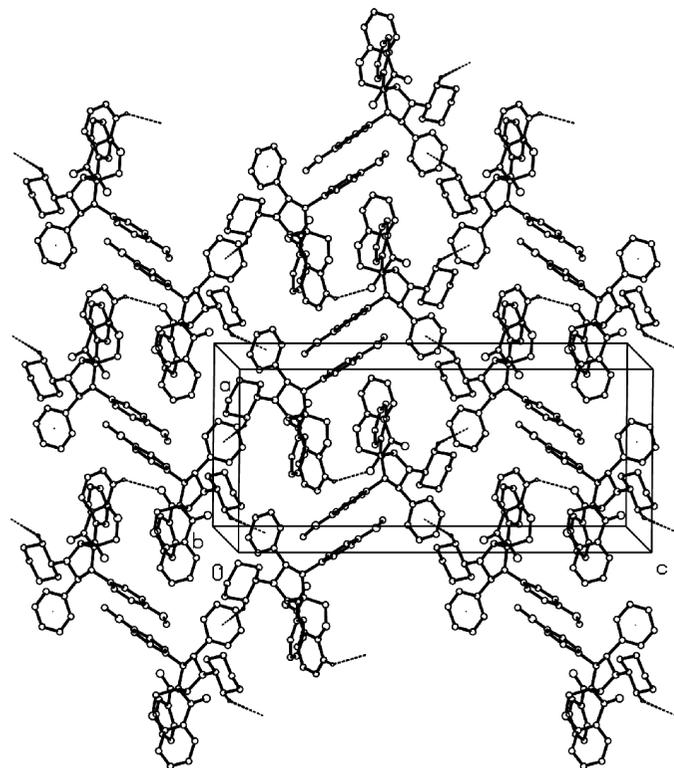


**Figure 2**  
The C—H...O intramolecular interactions in molecule A.

effective anomalous scatterers did not allow us to determine the Flack (1983) parameter. It is noteworthy that the two independent molecules in the crystal structure of (I) are opposite enantiomers (Fig. 1, Table 1). The title compound is thus racemic, but crystallizes in a space group usually adopted by single enantiomers of chiral molecules.

The conformations of both molecules are quite similar. The dihedral angle between rings C and D is 56.5 (1)° for molecule A and 65.7 (1)° for molecule B. The dihedral angle between rings F and G is 30.7 (1)° for molecule A and 42.0 (1)° for molecule B.

The pyrrolidine ring (A) is in an envelope conformation with puckering parameters (Cremer & Pople, 1975)  $q_2 = 0.370$  (2) Å and  $\varphi = -177.7$  (3)° for molecule A, and  $q_2 = 0.409$  (2) Å and  $\varphi = 2.5$  (4)° for molecule B. Atom N1 deviates by 0.544 (2) and 0.598 (2) Å from the least-squares plane through the remaining four atoms (C2–C5) of that of ring for molecules A and B, respectively. The cyclohexyl ring adopts a chair conformation. This is confirmed by the puckering para-



**Figure 3**  
Packing of the molecules in the crystal of (I), viewed down the *b* axis.

eters  $q_2 = 0.038$  (4) Å,  $q_3 = 0.574$  (4) Å =  $Q_T$ ,  $\theta = 3.8$  (4)° and  $\varphi = 10$  (7)° for molecule A, and  $q_2 = 0.031$  (2) Å,  $q_3 = -0.580$  (3) Å =  $Q_T$ ,  $\theta = 176.9$  (4)° and  $\varphi = -165$  (5)° for molecule B. The pyran ring in the chromanone moiety has a half-chair conformation with the lowest asymmetry parameters of  $\Delta C_2(C21-C20) = 0.059$  (2) for molecule A and 0.050 (1) for molecule B (Nardelli, 1983).

Intramolecular C—H...O interactions stabilize the conformation of the molecules (Fig. 2), whereas the crystal structure is stabilized by intermolecular C—H...O interactions (Table 2 and Fig. 3). In addition to this, the molecules in the crystal are also interlinked by C—H... $\pi$  interactions, so that H11B (bonded to C11A) is 2.83 Å from the centroid of phenyl ring C of molecule B transformed by  $(\frac{3}{2} - x, 2 - y, z - \frac{1}{2})$ , with a C11A—H11B...centroid angle of 158° and a C11A...centroid distance of 3.751 (4) Å; H11D (bonded to C11B) is 2.87 Å from the centroid of phenyl ring C of molecule A transformed by  $(\frac{3}{2} - x, 2 - y, \frac{1}{2} + z)$ , with a C11B—H11D...centroid angle of 152° and a C11B...centroid distance of 3.750 (3) Å.

## Experimental

A mixture of (*E*)-3-(*p*-methylbenzylidene)-4-chromanone and *cis*-1-cyclohexyl-2-phenyl-3-benzoylaziridine was refluxed in toluene under a nitrogen atmosphere for 48 h. After the reaction was complete, the solvent was evaporated *in vacuo* and the resulting crude product was purified by column chromatography using a hexane–benzene mixture (7:3) as eluant. The product was crystallized from ethanol (Amalraj *et al.*, 1999).

Crystal data

C<sub>38</sub>H<sub>37</sub>NO<sub>4</sub>  
*M<sub>r</sub>* = 571.69  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 11.8945 (3) Å  
*b* = 19.2698 (4) Å  
*c* = 27.0279 (6) Å  
*V* = 6194.9 (2) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.226 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 24284 reflections  
 $\theta = 2.0\text{--}28.2^\circ$   
 $\mu = 0.08\text{ mm}^{-1}$   
*T* = 293 (2) K  
 Block, yellow  
 0.24 × 0.20 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 42360 measured reflections  
 8348 independent reflections

6500 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.037  
 $\theta_{\text{max}} = 28.3^\circ$   
*h* = -10 → 15  
*k* = -25 → 23  
*l* = -35 → 32

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.047  
*wR* (*F*<sup>2</sup>) = 0.137  
*S* = 1.07  
 8348 reflections  
 775 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 1.0304P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.28\text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.17\text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N1A—C2A	1.461 (3)	N1B—C2B	1.464 (3)
N1A—C5A	1.464 (3)	N1B—C5B	1.465 (3)
C2A—C3A	1.557 (4)	C2B—C3B	1.559 (4)
C3A—C4A	1.562 (4)	C3B—C4B	1.564 (4)
C4A—C5A	1.576 (4)	C4B—C5B	1.568 (4)
C4A—C22A	1.536 (3)	C4B—C22B	1.530 (3)
C19A—O3A	1.219 (3)	C19B—O3B	1.212 (3)
C19A—C20A	1.477 (4)	C19B—C20B	1.484 (4)
C20A—C21A	1.395 (4)	C20B—C21B	1.385 (4)
C20A—C23A	1.409 (4)	C20B—C23B	1.403 (4)
C21A—O2A	1.365 (3)	C21B—O2B	1.374 (4)
C22A—O2A	1.435 (3)	C22B—O2B	1.438 (4)
C27A—C3A—C4A—C19A	-114.0 (2)	C27B—C3B—C4B—C19B	115.4 (2)
C2A—C3A—C4A—C19A	116.9 (2)	C2B—C3B—C4B—C19B	-115.3 (2)
C19A—C4A—C5A—N1A	-96.3 (2)	C19B—C4B—C5B—N1B	93.5 (2)
C19A—C4A—C5A—C12A	141.4 (2)	C19B—C4B—C5B—C12B	-145.7 (2)
C19A—C4A—C22A—O2A	-60.5 (3)	C19B—C4B—C22B—O2B	62.4 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3A—H3A...O3A	0.98	2.26	2.813 (3)	115
C3B—H3B...O3B	0.98	2.27	2.822 (3)	114
C2A—H2A...O1A	0.98	2.49	3.028 (3)	114
C2B—H2B...O1B	0.98	2.39	2.969 (3)	118
C22A—H22B...O1A	0.97	2.53	3.120 (4)	119
C22B—H22C...O1B	0.97	2.52	3.089 (4)	118
C26A—H26A...O1B <sup>i</sup>	0.93	2.59	3.310 (4)	134

Symmetry code: (i) *x* - 1, *y*, *z*.

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å, methine C—H distances of 0.98 Å and methylene C—H distances of 0.97 Å, and with *U*<sub>iso</sub> = 1.5*U*<sub>eq</sub>(C) for methyl H atoms and 1.2*U*<sub>eq</sub>(C) for other H atoms. Due to the lack of anomalous scatterers, the absolute structure was not determined from the X-ray diffraction data and Friedel pairs were merged.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 1990); software used to prepare material for publication: SHELXL97 and PARST (Nardelli, 1995).

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