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4′-(4-Methoxyphenyl)-1-methylchroman-3-spiro-3′-pyrrolidine-2′-spiro-2″-indan-4,1″,3″-trione

S. Selvanayagam,[a]
D. Velmurugan,[a]*
K. Ravikumar,[b]
J. Jayashankaran[c] and
R. Raghunathan[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.052
wR factor = 0.156
Data-to-parameter ratio = 17.4

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

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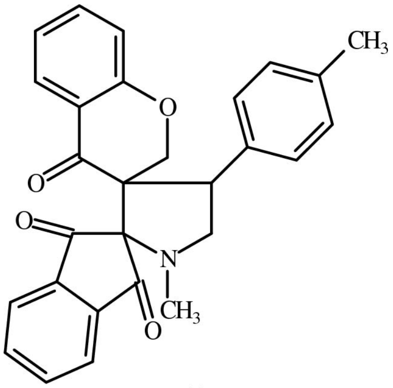
In the title compound, $C_{28}H_{23}NO_4$, the pyrrolidine ring adopts a twist conformation and the pyran ring in the chromanone system adopts a half-chair conformation. The molecules associate *via* C—H···O interactions to form $R_2^2(18)$ dimers.

Comment

The pyrrolidine skeleton occurs in many families of biologically important compounds. The resulting functionality, due to ease of substitution and therefore modification at several positions (Baldwin *et al.*, 1994*a*,*b*), has been utilized to synthesize compounds with various properties. These compounds are found to have antimicrobial and antifungal activity against various pathogens except *Bacillus subtilis* (Amal Raj *et al.*, 2003). Chromanone derivatives possess antiviral activities against HIV and simian immunodeficiency virus (Xu *et al.*, 1998). These derivatives also possess antifungal (Emami *et al.*, 2004; Yang *et al.*, 2002) and anti-inflammatory (Konieczny *et al.*, 1976) activities. In view of its medicinal importance, the crystal and molecular structure determination of (I) was carried out by X-ray diffraction.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1. The C—C bond lengths in the pyrrolidine ring are somewhat longer and the C—N bond lengths are somewhat shorter than normal values (Table 1). A similar effect has been observed in related reported structures (Abdul Ajees *et al.*, 2002; Gzella & Wrzeciono, 1990). The bond lengths in the pyran ring in the chromanone system are also close to those found in related structures (Abdul Ajees *et al.*, 2001; Usha *et al.*, 2003).

The sum of the angles (340.6°) at N1 is in accordance with sp^3 hybridization. The torsion angles C33—C30—C31—C32 [178.5 (2)°] and C33—C30—C29—C28 [−179.0 (2)°] indicate that the methyl group attached to the benzene ring does not deviate significantly from coplanarity with it. The methyl

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group attached to the pyrrolidine ring is in an equatorial position.

The indanedione group is planar with a maximum deviation of 0.087 (1) Å for atom C2. The keto O atoms O15 and O16 deviate from the mean plane of this ring system by 0.176 (1) and 0.251 (1) Å, respectively. The dihedral angle between the fused six-membered and five-membered rings of the indanedione group is 3.0 (1)°. The methylphenyl ring and the indanedione group are oriented at an angle of 53.2 (1)° with respect to each other. The benzene ring of the chromanone system makes a dihedral angle of 79.4 (1)° with the methylphenyl ring and 28.2 (1)° with the indanedione group.

The pyrrolidine ring adopts a twist conformation with puckering parameters q_2 = 0.419 (2) Å and φ = 149.9 (2)° (Cremer & Pople, 1975), the displacement asymmetry parameters being Δ_S(C5) = 0.039 (1) and Δ_2(C3) = 0.049 (1) (Nardelli, 1983). The pyran ring in the chromanone system has a half-chair conformation with the lowest asymmetry parameters of ΔC_2(C3—C25) = 0.012 (1) (Nardelli, 1983).

The molecules of (I) form $R_2^2(18)$ graph-set dimers *via* C—H···O hydrogen-bonding interactions (Fig. 2). The dimers are connected by a further C—H···O hydrogen bond (Table 2).

Experimental

A solution of 3-*p*-methylbenzylidene-4-chromanone (0.5 mmol), ninhydrin (0.5 mmol) and sarcosine (0.5 mmol) in aqueous methanol (20 ml) was refluxed until the disappearance of the starting material. The solvent was removed under reduced pressure and the crude product was purified by column chromatography using petroleum ether and ethyl acetate as eluant to give the title compound. The compound was recrystallized using ethyl acetate and hexane (1:1) by slow evaporation to obtain crystals of good quality.

Crystal data

$C_{28}H_{23}NO_4$
M_r = 437.47
Triclinic, $P\overline{1}$
a = 8.0652 (6) Å
b = 8.2977 (6) Å
c = 18.4090 (13) Å
α = 80.057 (1)°
β = 84.796 (1)°
γ = 69.754 (1)°
V = 1137.86 (14) Å^3
Z = 2
D_x = 1.277 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4856 reflections
θ = 2.4–23.8°
μ = 0.09 mm^{-1}
T = 273 (2) K
Block, colourless
0.24 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
13 219 measured reflections
5232 independent reflections
4501 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017
$\theta_{\rm max}$ = 28.0°
h = −10 → 10
k = −10 → 10
l = −23 → 23

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.156
S = 1.04
5232 reflections
300 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0959P)^2 + 0.1809P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.002
$\Delta\rho_{\rm max}$ = 0.35 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.23 e Å^{-3}

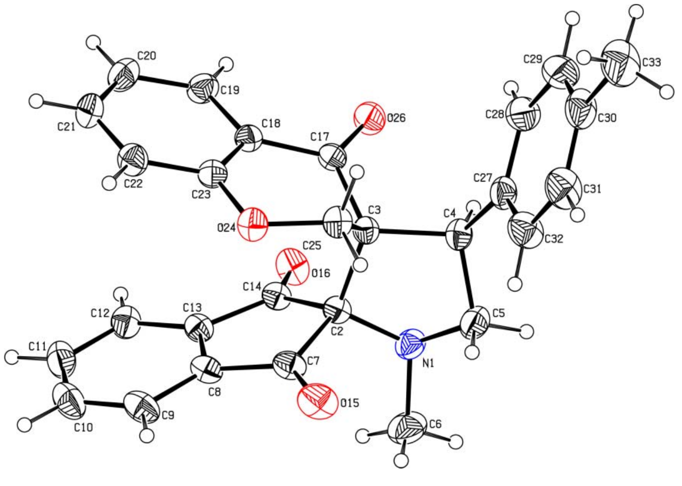

Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.449 (2)	C7—O15	1.208 (2)
N1—C2	1.454 (2)	C14—O16	1.206 (2)
C2—C3	1.586 (2)	C17—O26	1.203 (2)
C3—C4	1.564 (2)	C23—O24	1.367 (2)
C4—C5	1.518 (2)	O24—C25	1.430 (2)
C5—N1—C2	108.2 (1)	C2—N1—C6	116.6 (1)
C5—N1—C6	115.8 (1)		
C6—N1—C2—C3	164.3 (1)	C28—C29—C30—C33	−179.0 (2)
C6—N1—C5—C4	−178.9 (1)	C33—C30—C31—C32	178.5 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20···O16$^{\rm i}$	0.93	2.60	3.526 (2)	179
C9—H9···O26$^{\rm ii}$	0.93	2.47	3.122 (2)	127

Symmetry codes: (i) $-x+2, -y, -z+1$; (ii) $x-1, y+1, z$.

H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.98 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H and 1.2$U_{\rm eq}$(C) for other H atoms. In addition, the methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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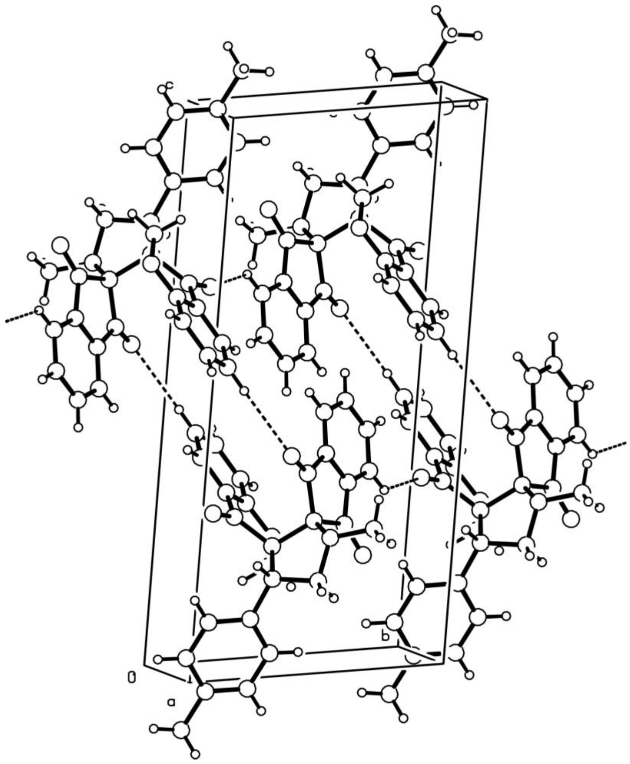

Figure 2
The molecular packing of (I), viewed approximately down the *a* axis. Dashed lines indicate hydrogen bonds.

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