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4′-(4-Chlorophenyl)-2′-[1-(4-methoxyphenyl)-4-oxo-3-phenylazetidin-2-yl]-1′-methyl-2-phenylspiro[1,3-oxazole-4(5*H*),3′-pyrrolidin]-5-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.059
wR factor = 0.193
Data-to-parameter ratio = 17.2

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

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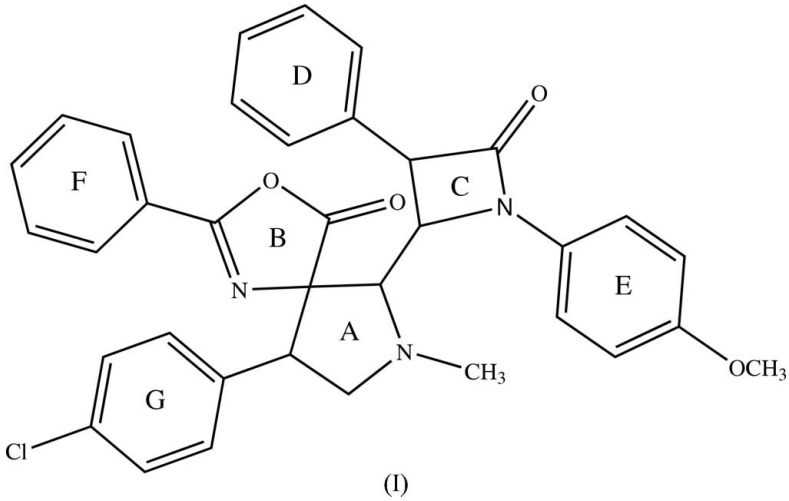
The pyrrolidine ring of the title molecule, $C_{35}H_{30}ClN_3O_4$, adopts a half-chair conformation; the oxazole and azetidine rings are essentially planar. Intermolecular C—H···O interactions are involved in the formation of dimers which are connected into a chain running along the [010] direction.

Comment

The spiro-ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids. Pyrrolidine derivatives possess anti-influenza virus (Stylianakis *et al.*, 2003) and anticonvulsant (Obniska *et al.*, 2002) activity. Substituted pyrrolidine compounds have been found to have antimicrobial and antifungal activity against various pathogens (Amalraj *et al.*, 2003). Spiro-oxazole derivatives have been used in many natural product syntheses and have also been proved to be efficient precursors for many synthetic intermediates including γ-aminoalcohols, β-hydroxyketones, *etc*. (Kozikowski, 1984; Kanemasa & Tsuge, 1990). Spiro-oxazole compounds display interesting biological properties such as herbicidal, plant-growth regulatory and antiumour activities (Howe & Shelton 1990; De Amici *et al.*, 1990; Smietana *et al.*, 1999). The title compound, (I), was chosen for crystallographic study to establish its structure.



(I)

Fig. 1 shows a displacement ellipsoid plot of the title molecule, with the atomic numbering scheme. The pyrrolidine ring is in a half-chair conformation, with lowest displacement asymmetry parameter (Nardelli, 1983) ΔC_2(C1) = 0.009 (1), and puckering parameters (Cremer & Pople, 1975) q_2 = 0.425 (2) Å and φ = 123.2 (2)°. The oxazole and azetidine rings are essentially planar, with maximum deviations of 0.043 Å for

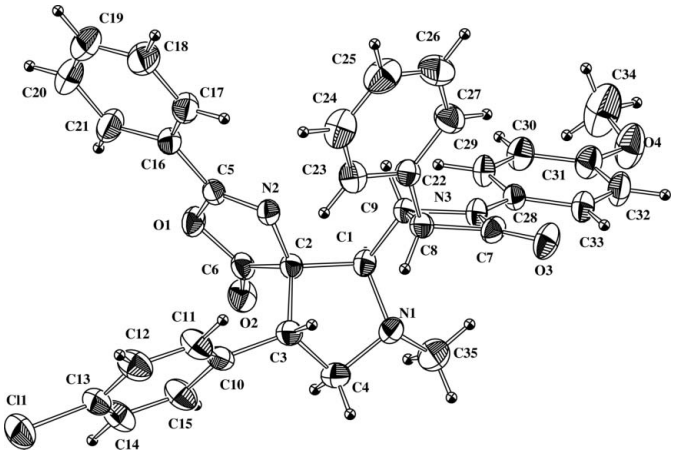


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

C2 and 0.023 (2) Å for N3. The oxazole ring makes a dihedral angle of 2.1 (1)° with the attached phenyl ring.

The methoxy group is almost coplanar with the attached phenyl ring. The ketone atoms O2 and O3 deviate by 0.164 (2) and 0.046 (2) Å, respectively, from the least-squares planes of the oxazole and azetidine rings.

In the crystal structure, there is an intermolecular C32—H32···O4^{i} hydrogen bond (symmetry code as in Table 2), resulting in the formation of dimers with graph-set motif $R_2^2(8)$ (Bernstein *et al.*, 1995). The dimers are linked by the C19—H19···O3^{ii} hydrogen bond (Table 2), forming $C(13)$ chains running along the *b*-axis direction. A noteworthy feature of the crystal structure is the existence of a short intermolecular halogen–carbon contact, *i.e.* Cl1···C17^{iii} 3.226 (3) Å [symmetry code: (iii) $x-1, y, z$].

Experimental

A mixture of *cis*-4-formyl-2-azetidinone (1 mmol), sarcosine (1 mmol) and 4-(*p*-chlorobenzylidene-2-phenyloxazol)-5-one (1 mmol) in dry toluene (15 ml) was refluxed for 12 h at 383 K, using a Dean–Stark apparatus. After completion of the reaction, as evidenced by thin-layer chromatography, the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using hexane–ethyl acetate (8.5:1.5) as eluent. The product was recrystallized from $CHCl_3$–hexane (1:2) by slow evaporation (m.p. 518 K).

Crystal data

$C_{35}H_{30}ClN_3O_4$
M_r = 592.07
Triclinic, $P\overline{1}$
a = 10.0409 (10) Å
b = 13.2335 (13) Å
c = 13.4951 (13) Å
α = 117.751 (2)°
β = 94.976 (2)°
γ = 98.865 (2)°
V = 1542.0 (3) Å^3
Z = 2
D_x = 1.275 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 293 (2) K
Block, colourless
0.38 × 0.25 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
9969 measured reflections
6680 independent reflections
4825 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.193
S = 0.91
6680 reflections
388 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.15P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected torsion angles (°).

C35—N1—C4—C3	−164.65 (18)	C34—O4—C31—C32	−171.2 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C32—H32···O4^{i}	0.93	2.58	3.476 (3)	161
C19—H19···O3^{ii}	0.93	2.59	3.295 (3)	133
C21—H21···O1	0.93	2.43	2.764 (3)	101
C33—H33···O3	0.93	2.51	3.114 (3)	123

Symmetry codes: (i) $-x+3, -y, -z$; (ii) $x, y+1, z$.

All H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(methyl C) or 1.2U_{eq}(C).

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: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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