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7″-Benzyl-9″-benzylidene-4′-[4-(dimethylamino)-phenyl]-1′-methyl-5″-phenyl- 2″,3″,6″,7″,8″,9″-hexahydro-1*H*-indole-3(2*H*)-spiro-2′-pyrrolidine-3′-spiro-2″-pyrido[4,3-*d*]thiazolo[3,2-*a*]pyrimidine-2,2″-dione acetonitrile disolvate

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Key indicators

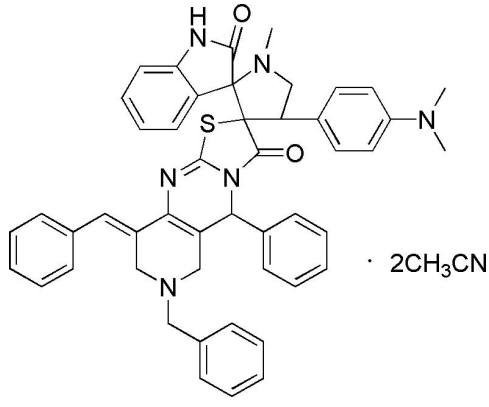
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.157
Data-to-parameter ratio = 12.7

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

The title compound, $C_{48}H_{44}N_6O_2S\cdot 2C_2H_3N$, was synthesized by the intermolecular [3+2]-cycloaddition of an azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 7-benzyl-9-benzylidene-2-[4-(dimethylamino)-benzylidene]-5-phenyl-2,3,6,7,8,9-hexahydro-5*H*-pyrido-[4,3-*d*]thiazolo[3,2-*a*]pyrimidin-3-one. In the molecule, the two spiro junctions link a planar 2-oxindole system, a pyrrolidine ring in an envelope conformation and a thiazolidone ring of the fused pyrimidine ring system. The packing of the molecules in the crystal structure is mainly stabilized by N—H···O hydrogen bonds.

Comment

The intermolecular 1,3-dipolar cycloaddition reaction of azomethine ylides with exocyclic double bonds provides the spiropyrrolidine framework, which occurs widely in natural substances and is characterized by highly pronounced biological properties (Raj *et al.*, 2003; Mishriky *et al.*, 1997). In this paper, the structure of the title compound, (I), is reported. The compound was synthesized by the intermolecular [3+2]-cycloaddition of an azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 7-benzyl-9-benzylidene-2-[4′-(dimethylamino)benzylidene]-5-phenyl-2,3,6,7,8,9-hexahydro-5*H*-pyrido[4,3-*d*]thiazolo[3,2-*a*]pyrimidin-3-one (Hammam *et al.*, 2001).



(I)

The structure of (I) is shown in Fig. 1. The pyrrolidine ring (N5/C30/C29/C40/C39) is not planar and adopts an envelope conformation. Atoms C30, C29, C40 and C39 are almost coplanar, the mean deviation from this plane being 0.026 (3) Å. Atom N5 lies 0.594 (3) Å above the C30/C29/C40/C39 plane of the pyrrolidine ring, forming the flap of the

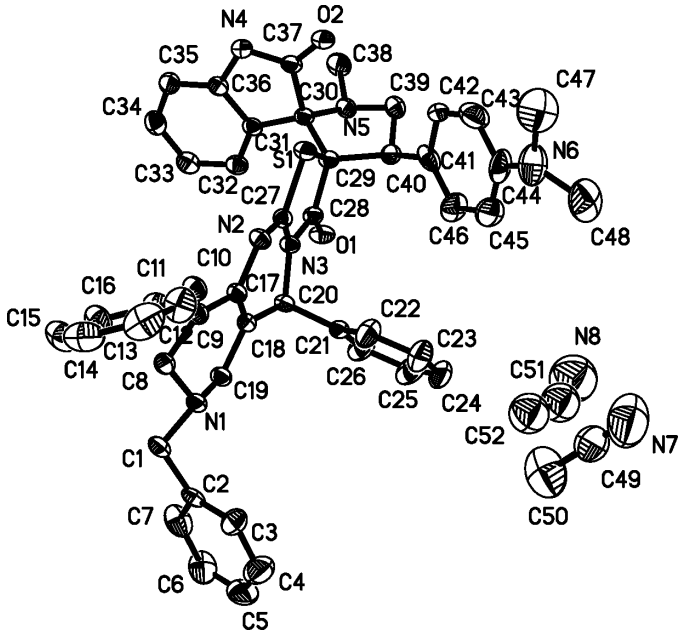


Figure 1
The molecular structure of (I), showing the atom numbering and 30% probability ellipsoids. H atoms have been omitted for clarity.

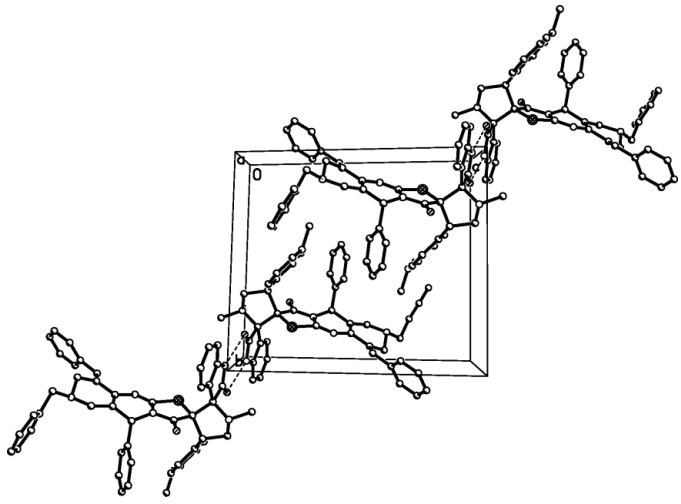


Figure 2
The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

envelope. The dihedral angle between the C30/N5/C39 and C30/C29/C40/C39 planes is 42.8 (2)°. The 4-dimethylaminophenyl group is rotated by 76.5 (2)° with respect to the C30/C29/C40/C39 plane, as a result of steric hindrance. The 2-oxoindole system (C30/C37/N4/C36/C31–C35) is nearly planar [mean deviation 0.013 (3) Å] and makes a dihedral angle of 74.8 (2)° with the C30/C29/C40/C39 plane. The five-membered thiazolidone ring (S1/C27/N3/C28/C29), fused to the pyrimidine ring, is nearly planar [mean deviation 0.017 (3) Å]. The S1/C27/N3/C28/C29 mean plane makes a dihedral angle of 94.0 (2)° with the adjacent C30/C29/C40/C39 plane. Atoms N3/C27/N2/C17/C18 of the six-membered dihydropyrimidine ring are almost coplanar, as a result of electron delocalization [mean deviation 0.031 (3) Å]. Atom C20 lies 0.342 (3) Å above this plane. The N3/C27/N2/C17/C18 plane makes dihedral angles of 2.3 (2) and 91.1 (2)°, respectively, with the S1/C27/N3/C28/C29 plane and the C21–C26 phenyl ring. The piperidine ring adopts a chair conformation. The crystal packing (Fig. 2) is characterized by layers parallel to the *bc* plane. Pairs of molecules in these layers are connected by two intermolecular N—H···O hydrogen bonds (Table 1).

Experimental

A mixture of 7-benzyl-9-benzylidene-2-[4′-(dimethylamino)benzyl idene]-5-phenyl-2,3,6,7,8,9-hexahydro-5*H*-pyrido[4,3-*d*]thiazolo[3,2-*a*]pyrimidin-3-one (1 mmol), isatin (1.2 mmol) and sarcosine (1.2 mmol) was refluxed in acetonitrile (80 ml) until the disappearance of the starting materials, as evidenced by thin-layer chromatography. The solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate, 5:1) to give the unsolvated compound. M.p. 473 K. IR (KBr, cm^{-1}): 3419.9 (–NH), 1719.9 (C═O), 1367.5 ($-CH_3$); ^{1}H NMR (p.p.m.): δ 2.20 (*s*, 3H, $-NCH_3$), 2.72 (*d*, *J* = 17.0 Hz, 1H, piperidyl), 2.96 (*s*, 6H, $-NCH_3$), 3.04 (*d*, *J* = 17.0 Hz, 1H, piperidyl), 3.32 (*d*, *J* = 13.0 Hz, 1H, piperidyl), 3.37 (*d*, *J* = 14.5 Hz, 1H, piperidyl), 3.39 (*dd*, *J* = 7.5, 9.0 Hz, 1H, –CH), 3.45 (*d*, *J* = 13.0 Hz, 1H, piperidyl), 3.74 (*d*, *J* = 14.5 Hz, 1H, piperidyl), 3.95–3.99 (*m*, 1H, –CH), 4.05 (*dd*, *J* = 7.5 and 10.5 Hz, 1H, –CH), 5.18 (*s*, 1H, –CH), 6.59–7.46 (*m*, 25H, –CH, –ArH and –NH). This product (20 mg) was dissolved in acetonitrile (15 ml); the solution was kept at room temperature for 20 d to give colourless single crystals of (I), suitable for X-ray analysis.

Crystal data

$C_{48}H_{44}N_6O_2S\cdot 2C_2H_3N$	Z = 2
M_r = 851.06	D_x = 1.218 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 12.4130 (14) Å	Cell parameters from 3369 reflections
b = 13.1265 (15) Å	θ = 2.3–24.0°
c = 15.1590 (18) Å	μ = 0.12 mm^{-1}
α = 87.559 (2)°	T = 294 (2) K
β = 74.392 (2)°	Block, colourless
γ = 77.313 (2)°	0.26 × 0.22 × 0.20 mm
V = 2320.5 (5) $Å^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	8127 independent reflections
φ and ω scans	5109 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.021
T_{min} = 0.960, T_{max} = 0.972	θ_{max} = 25.0°
11 894 measured reflections	$h = -14 \rightarrow 13$
	$k = -13 \rightarrow 15$
	$l = -18 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 1.1607P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.053	
$wR(F^2)$ = 0.157	
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.003
8127 reflections	$\Delta\rho_{max}$ = 0.43 e $Å^{-3}$
642 parameters	$\Delta\rho_{min}$ = −0.32 e $Å^{-3}$
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O2^{i}	0.83 (3)	2.06 (3)	2.885 (3)	169 (3)

Symmetry code: (i) $-x+1, -y+2, -z$.

C-bound H atoms were positioned geometrically and refined in the riding-model approximation [C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C)]. The N-bound H atom (H4) was located from a difference map and refined freely. Atoms C51, C52 and N8 of one of the acetonitrile solvent molecules are disordered over two sites and were refined isotropically with their bond lengths restrained [C—C = 1.5 (1) Å and C—N = 1.1 (1) Å]. The atoms of the C2–C7 phenyl ring and the 4-dimethyaminophenyl group (C41–C48/N6) are disordered over two sites. The ratios of site occupancies from the refinement were 0.48 (2):0.52 (2) and 0.52 (2):0.48 (2), respectively. The disordered benzene ring was constrained to have the geometry of a regular hexagon. The other disordered atoms were restrained with C—N bond lengths of 1.48 (1) Å, C—C bond lengths of 1.52 (1) Å and C═C bond lengths of 1.34 (1) Å.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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