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CRYSTAL STRUCTURE OF (3S,6'R,7'R)-6'-(1H-INDOLE-3-CARBONYL)-2-OXO-7'-(PYRIDIN-3-YL)-3',6',7',7A'-TETRAHYDRO-1'H-SPIRO[INDOLINE-3,5'-PYRROLO[1,2-C]THIAZOLE]-6'-CARBONITRILE COMPOUND WITH (METHYLSULFINYL)METHANE

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Abstract: In the title compound, C30 H27 N5 O3 S2, the dihedral angle between the indole ring system (r.m.s. deviation = 0.050 Å) and the thiazole ring is 88.88 (8)º. The latter ring adopts an envelope conformation with the N atom as the flap. The pyrrole ring (N4-C10) has a twisted conformation. In the crystal, molecules associate via N-H…N intermolecular interactions (C-H…N), forming chains propagating along the [100] direction.

***Index Terms*: single-crystal X-ray study; T = 295 K; mean (C–C) = 0.004 A ˚; disorder in solvent or counterion; R factor = 0.0504; wR factor = 0.1510; data-to-parameter ratio = 11.9.**

I. INTRODUCTION

The chemistry of indole has been of increasing interest, since several compounds of this type possess diverse biological activities (Macor et al., 1992)[1]. These derivatives exhibit antibacterial, antifungal (Singh et al., 2000)[2] and antitumour activities (Andreani et al., 2001)[3]. Some of the indole alkaloids extracted from plants possess interesting cytotoxic and antiparasitic properties (Quetin-Leclercq, 1994[4]; Mukhopadhyay et al., 1981)[5]. Indole containing compounds are best known for their medicinal properties in the pharmaceutical industry. In modern times, analogs based on indole are significant players in a diverse array of markets such as dyes, plastics, agriculture, vitamin supplements, over-the-counterdrugs, flavour enhancers and perfumery (Barden, 2011)[6]. Synthesis of spiro compounds has drawn considerable attention from chemists, in view of their very good antimycobacterial activity (Chande et al., 2005)[7]. Oxindole derivatives are known to be potent inhibitors of monoamine oxidase (MAO) in human urine and rat tissues (Glover et al., 1998)[8] and potent antagonists of in vitro receptor binding by atrial natriuretic peptide besides possessing a wide range of central nervous system activities (Bhattacharya et al., 1982)[9]. Oxindole derivatives are of importance in the total synthesis of indole and oxindole alkaloids such as potent inhibitors of monoamine oxidase (MAO) in human urine and rat tissues (Glover et al., 1998)[8].

II. Experimental

A mixture of isatin2a-f (1.0 mmol), sarcosine3 (1.1 mmol) and (pyridin-3-yl)-3',6',7',7a'-tetrahydro-1'H-spiro[indoline-3,5'-pyrrolo[1,2-c]thiazole]-6'-carbonitrile (1.2 mmol) in methanol was stirred at room temperature for 120 min. The solid

precipitated during the reaction mixture was filtered and dried under vacuum to obtain spirooxindoles5a-f in crude form. The resulting crude product was purified by flash column chromatography (mesh 100–200) using hexane/EtOAC (7:3). The solid single product was finally recrystallized from ethanol, giving title compound in good yield as colourless block-like crystals.

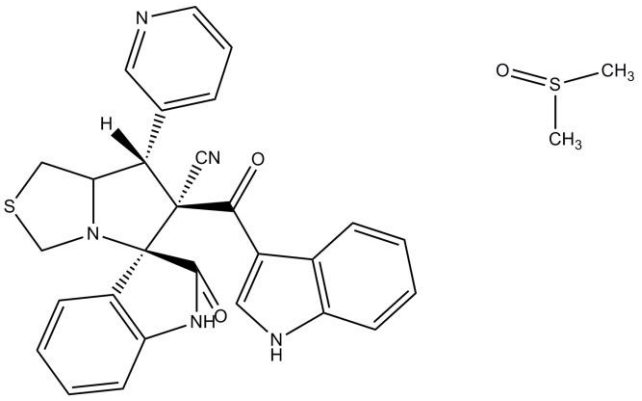

Figure1 Scheme

III. Data collection

APEX2 (Bruker, 2004)[10]; cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT and XPREP (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008)[11]; program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009)[12]; software used to prepare material for publication: SHELXL97 and PLATON[13].

IV. Refinement

The H atoms could all be located in difference electron-density maps. In the final cycles of refinement they were treated as riding atoms and their distances were geometrically constrained: C—H = 0.93 and 0.96 Å for CH and CH3 atoms, respectively, with Uiso(H) = 1.5 Ueq(C– methyl) and = 1.2Ueq(C) for other H atoms.

V. Structural Commentary

The molecular structures of the title compound are illustrated in Figs. 1. The five membered ring (S1-C9) in has a envelope conformation, with puckering (Cremer & J.A. Pople et al.,1975)[14] parameters q_2= 0.485(2) Å ϕ_2= 1.4(3)º. The pyrrole ring (N4-C10) has a twisted conformation. The thiazole and pyrrole rings are inclined at 57.28(2)º, respectively. The two indole rings almost planar, with an interplanar angle of 83.62(10)º the atoms N1 and N2 deviate by -1.944(2) Å and -1.654(2) Å, respectively from the rings to which they are attached. In molecule, the angle between the two disordered solvent compound is 6.2 (5)º. The sulfonyl group, are disorder with s.o.f. vales of 0.589(16) Å and 0.411(16) Å, respectively. Bond lengths and angles are of expected values with the C=N bond length of 1.448(3) Å, clearly indicating a double bond. In addition, the N-C bond [1.331(3) Å] is shortened with respect to the other nitrogen carbon bonds, as is typical for amides. For instance: the 107.46(15)º angle S-O-C at the ring junctions are appreciably less than 120º.

VI. Superamolecular features

In the crystal, molecules associate via N-H…N intermolecular interactions (N2-H2A…N3), forming chains propagating along the [100] direction, see Fig.2. In addition to this inversion related molecules are linked into chains by C-H…π interactions [C29-H29C… Cg7], where cg is the centroid of the (C23-C28) benzene ring. Intra- and intermolecular interactions observed are C-H…N type hydrogen bonds and parallel partial pi-pi stacking between the fused aromatic rings of the core of the molecules within each unit, and also connecting to molecules with translational periodicity in a a-axis direction in what can be described as columns of stacked molecules wit alternating chirality (Fig.1). In addition to this weak C-H…π interactions are also observed.

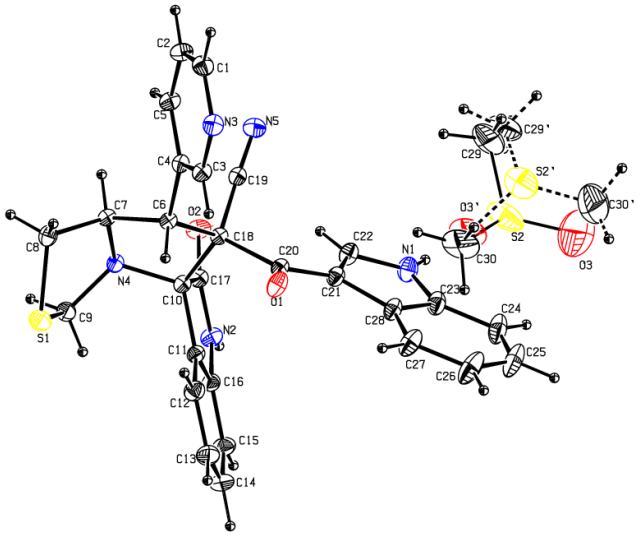

Figure 2 The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

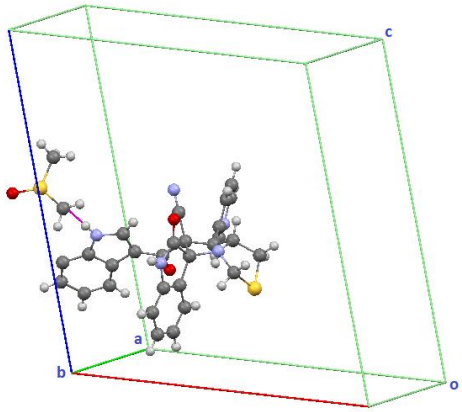

Figure 3 A partial view along the a axis of the crystal packing of the title compound. The N-H…C hydrogen bonds are shown as dashed lines (see Table 2 for details).

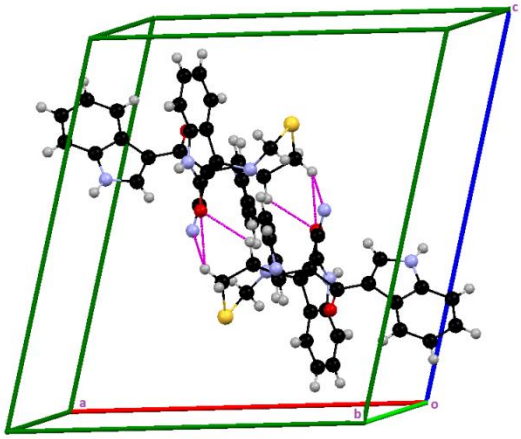

Figure 2 A views along the c axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 2)

VII. Table: 1

Crystal Data and Details of the Structure Determination

Parameters	**Title of compound**
Empirical formula	C30 H27 N5 O3 S2
Formula weight	569.69
Wavelength	0.71073 Å
Crystal system space group	Monoclinic P21/c
Unit cell dimensions	a = 15.6743(8)Å
	b = 10.9532(6) Å
	c = 17.2489(8) Å
	α = 90°
	β 104.833(2)°
	γ = 90°
Volume	2862.7(3) $Å^3$
Z, Calculated density	4, 1.3218(1)Mg/m^3
Absorption coefficient	0.185 mm^{-1}
F(000)	1192
Crystal size	0.15 x 0.20 x 0.25 mm
θ range	1.43 to 28.41°
Index ranges	-18 ≤ h ≤ 18 -13 ≤ k ≤ 13 -20 ≤ l ≤ 20
Reflections collected / unique	43661 / 5329 [R(int) = 0.041]
Completeness to theta	99.1 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5329/ 0 / 392
Goodness-of-fit on F^2	1.071
Final R indices [I>2sigma(I)]	R1 = 0.0504 wR2 = 0.1514
R indices (all data)	R1 = 0.1083 wR2 = 0.1822
Largest diff. peak and hole	0.46 and -0.44 e.$Å^{-3}$

VIII. Table 2

Hydrogen-bond geometry (Å , °).

D-H…A	**D-H**	**H…A**	**D…A**	**D-H…A**
N2-H2A…N3	0.86	2.11	2.911(3)	154
C8-H8A…N5	0.97	2.62	3.404(2)	138

Symmetry codes: (i) x,-1+y, z
(ii) 1-x,-y,-z

IX. ACKNOWLEDGMENT

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X. References

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