organic compounds

(E)-N'-[5-Chloro-3-methoxy-2-(4-methylphenylsulfonyloxy)benzylidene]isonicotinohydrazide acetic acid solvate: hydrogen-bonded network of alternating $R_4^4(42)$, $R_5^5(33)$ and $R_6^6(40)$ rings

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In the title compound, $C_{21}H_{18}ClN_3O_5S \cdot C_2H_4O_2$, a combination of O—H⋯O, N—H⋯O, C—H⋯O and C—H⋯N hydrogen bonds links the components into a complex network containing alternating $R_4^4(42)$, $R_5^5(33)$ and $R_6^6(40)$ rings.

Comment

There has been a steady growth of interest in the structure and reactivity of Schiff bases owing to their potential biological activities, such as antibacterial and antitumor (Kahwa *et al.*, 1986; Santos *et al.*, 2001). Isonicotinohydrazide forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them have been reported (Wardell, de Souza, Ferreira *et al.*, 2005; Wardell, de Souza, Wardell *et al.*, 2005; Wardell *et al.*, 2006; Low *et al.*, 2006). In order to obtain

(I)
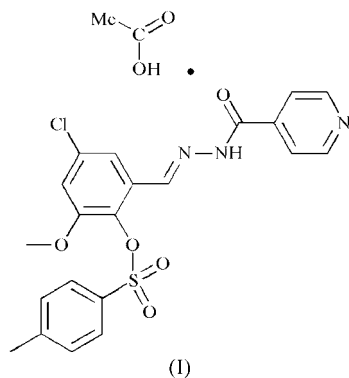
more detailed information on the structural conformation of the molecule, which may be of value in structure–activity analysis, we report here the synthesis and structure of the title compound, (I).

Within the hydrazine component in (I), both N atoms (N17 and N27) have effectively planar coordination, and the N—N bond distance (Table 1) is typical of the value in hydrazines with both N atoms having a planar coordination (the mean value is 1.401 Å; Allen *et al.*, 1987). In addition, the central spacer unit between atoms C17 and C21 (Fig. 1) adopts a nearly planar all-*trans* conformation, as shown by the key torsion angles (Table 1).

In (I) (Fig. 1), the acetic acid molecule is effectively tethered to the hydrazine–carbonyl component by a combination of two independent hydrogen bonds, one of O—H⋯O and one of N—H⋯O type (Fig. 2 and Table 2). Acetic acid atom O41 acts as a hydrogen-bond donor to carbonyl atom O17. At the same time, hydrazine atom N17 in the molecule at (x, y, z) acts as a hydrogen-bond donor to acetic acid atom O42 in the molecule at $(2 - x, 1 - y, -\frac{1}{2} + z)$. In addition, atom C33 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O25 in the molecule at $(x, y, 1 + z)$, so generating by translation a $C_2^2(8)C(9)R_5^5(33)$ chain of fused rings (Bernstein *et al.*, 1995) running parallel to the [001] direction.

Atom C24 in the molecule at $(1 + x, y, z)$ acts as a hydrogen-bond donor to pyridyl atom N11 in the molecule at (x, y, z), so forming a $C(13)$ chain running parallel to the [100] direction. In addition, atom C33 in the molecule at $(x, y, -1 + z)$ in turn acts as a hydrogen-bond donor to atom O25 in the molecule at (x, y, z), so forming a $C(9)$ chain running parallel to the [001] direction. The combination of the [100] and [001] chains then generate a sheet parallel to (010) containing $R_4^4(42)$ rings (Fig. 3). In combination with the $C_2^2(8)C(9)R_5^5(33)$ chain of rings (Fig. 2), these hydrogen bonds then generate a complex network containing alternating $R_4^4(42)$, $R_5^5(33)$ and $R_6^6(40)$ rings (Fig. 4).
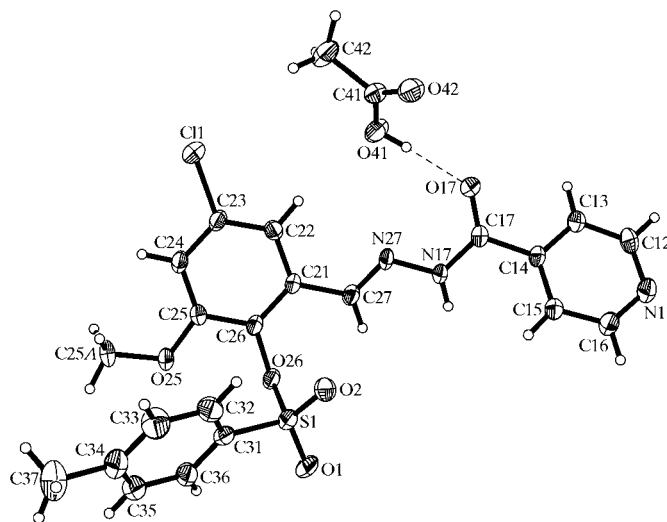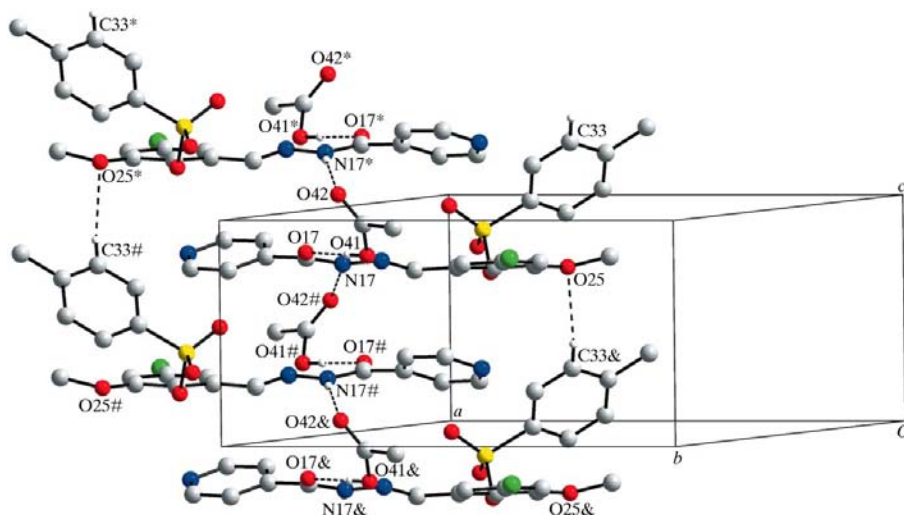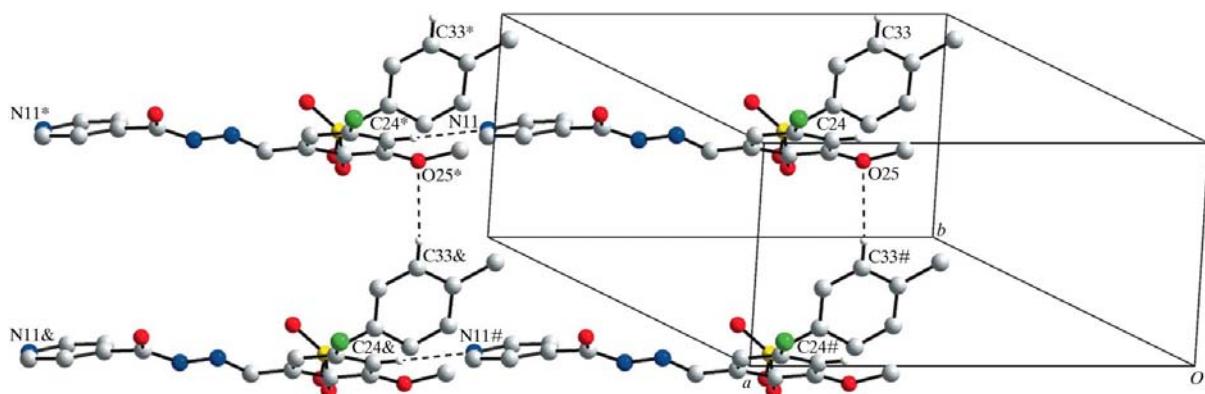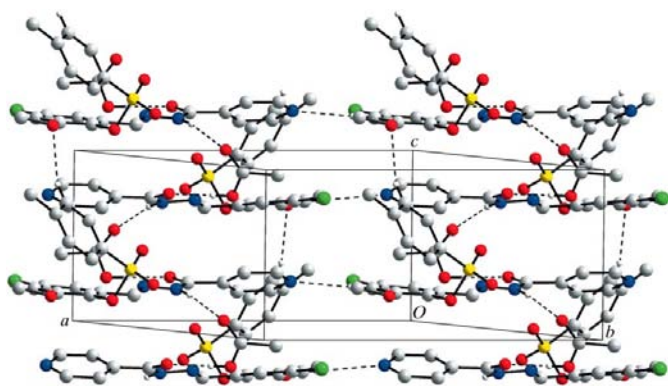

Figure 1
The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Figure 2
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded chain of rings along [001]. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(2 - x, 1 - y, \frac{1}{2} + z)$, $(2 - x, 1 - y, -\frac{1}{2} + z)$ and $(x, y, -1 + z)$, respectively.

Figure 3
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded (010) sheet. For the sake of clarity, H atoms not involved in the motifs shown have been omitted. Atoms marked with an asterisk (*), a hash (#) or an ampersand (&) are at the symmetry positions $(1 + x, y, z)$, $(x, y, -1 + z)$ and $(1 + x, y, -1 + z)$, respectively.

Figure 4
Part of the crystal structure of (I), showing the complex network containing alternating $R_4^4(42)$, $R_5^5(33)$ and $R_6^6(40)$ rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

Experimental

An anhydrous ethanol solution (50 ml) of 4-chloro-2-formyl-6-methoxyphenyl 4-methylbenzenesulfonate (3.41 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of isonicotino-hydrazide (1.37 g, 10 mmol) and the mixture was stirred at 350 K for 3 h under N_2, giving a white precipitate. The product was isolated, recrystallized from ethanol and then dried in a vacuum to give pure compound (I) in 72% yield. Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a solution in ethanol and acetic acid (80:20 v/v).

Crystal data

$C_{21}H_{18}ClN_3O_5S \cdot C_2H_4O_2$	$V = 2438.5 (8)$ Å^3
$M_r = 519.96$	$Z = 4$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 14.886 (3)$ Å	$\mu = 0.29$ mm^{-1}
$b = 23.190 (4)$ Å	$T = 294 (2)$ K
$c = 7.0638 (14)$ Å	$0.26 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	13425 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	4793 independent reflections
$T_{min} = 0.912$, $T_{max} = 0.955$	2849 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.069$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	H-atom parameters constrained
$wR(F^2) = 0.092$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 0.97$	$\Delta\rho_{min} = -0.20$ e Å^{-3}
4793 reflections	Absolute structure: Flack (1983), 2080 Friedel pairs
322 parameters	Flack parameter: -0.01 (7)
1 restraint	

Table 1
Selected geometric parameters (Å, °).

N17—N27	1.377 (3)		
O1—S1—O2	120.13 (15)	C17—N17—N27	118.9 (3)
O1—S1—O26	102.53 (17)	C26—O26—S1	118.4 (2)
O2—S1—O26	107.68 (13)	C27—N27—N17	115.9 (3)
O1—S1—C31	110.46 (16)	N27—C27—C21	120.0 (3)
O2—S1—C31	109.27 (19)	O17—C17—N17	123.3 (3)
O26—S1—C31	105.61 (14)		
O1—S1—O26—C26	−174.7 (2)	C26—C21—C27—N27	−164.9 (3)
O2—S1—O26—C26	−47.0 (2)	C22—C21—C27—N27	14.5 (5)
C31—S1—O26—C26	69.6 (2)	N27—N17—C17—O17	−5.1 (6)
C17—N17—N27—C27	174.6 (3)	N27—N17—C17—C14	173.3 (3)
N17—N27—C27—C21	179.3 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O41—H41$\cdots$O17	0.82	1.83	2.649 (3)	177
N17—H17$\cdots$O42^i	0.86	2.16	2.992 (4)	163
C33—H33$\cdots$O25^{ii}	0.93	2.50	3.378 (6)	157
C24—H24$\cdots$N11^{iii}	0.93	2.59	3.521 (4)	178

Symmetry codes: (i) $-x + 2, -y + 1, z - \frac{1}{2}$; (ii) $x, y, z + 1$; (iii) $x - 1, y, z$.

H atoms were included in calculated positions and refined using a riding-model approximation [constrained C—H and N—H bond lengths and U_{iso}(H) parameters: 0.93 Å and $1.2U_{eq}$(C) for Csp^2 H atoms, 0.96 Å and $1.5U_{eq}$(C) for methyl H atoms, 0.82 Å and $1.5U_{eq}$(O) for the hydroxy H atom, and 0.86 Å and $1.2U_{eq}$(N) for the imine H atom].

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

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