O3*A*—C10*A*—C15*A*	113.4 (4)	O3*B*—C10*B*—C15*B*	115.2 (4)
C12*A*—C13*A*—C16*A*	122.9 (4)	C12*B*—C13*B*—C16*B*	122.7 (5)
C14*A*—C13*A*—C16*A*	117.0 (5)	C14*B*—C13*B*—C16*B*	118.0 (4)
O4*A*—C16*A*—C13*A*	111.9 (5)	O4*B*—C16*B*—C13*B*	112.1 (4)
O4*A*—C17*A*—C18*A*	117.4 (4)	O4*B*—C17*B*—C18*B*	115.0 (4)
O5*A*—C16*A*—C13*A*	125.8 (4)	O5*B*—C16*B*—C13*B*	126.3 (5)
O6*A*—C23*A*—N1*A*	115.4 (3)	O6*B*—C23*B*—N1*B*	114.3 (4)
N1*A*—C23*A*—C20*A*	128.0 (4)	N1*B*—C23*B*—C20*B*	128.7 (5)
N1*A*—C24*A*—C29*A*	109.8 (4)	N1*B*—C24*B*—C29*B*	108.7 (4)
C19*A*—C20*A*—C23*A*	119.4 (4)	C19*B*—C20*B*—C23*B*	121.0 (4)
C21*A*—C20*A*—C23*A*	121.6 (4)	C21*B*—C20*B*—C23*B*	119.3 (4)
C25*A*—C24*A*—C29*A*	119.4 (4)	C25*B*—C24*B*—C29*B*	121.1 (5)
C24*A*—C25*A*—C26*A*	117.5 (4)	C24*B*—C25*B*—C26*B*	116.0 (5)
C25*A*—C26*A*—C27*A*	119.4 (4)	C25*B*—C26*B*—C27*B*	120.2 (4)
C26*A*—C27*A*—C28*A*	125.2 (4)	C26*B*—C27*B*—C28*B*	125.4 (5)
C27*A*—C28*A*—C29*A*	112.6 (4)	C27*B*—C28*B*—C29*B*	112.6 (5)
O6*A*—C29*A*—C24*A*	106.8 (3)	O6*B*—C29*B*—C24*B*	107.9 (4)
C24*A*—C29*A*—C28*A*	125.9 (4)	C24*B*—C29*B*—C28*B*	124.7 (4)

C17*A*—O4*A*—C16*A*—C13*A*	−176.6 (4)
C16*A*—O4*A*—C17*A*—C18*A*	115.2 (5)
O8*A*—N2*A*—C27*A*—C26*A*	−177.4 (5)
C7*A*—C8*A*—C9*A*—O3*A*	60.7 (6)
C14*A*—C13*A*—C16*A*—O4*A*	170.8 (4)
C19*A*—C20*A*—C23*A*—O6*A*	176.3 (4)
C17*B*—O4*B*—C16*B*—C13*B*	174.6 (4)
C16*B*—O4*B*—C17*B*—C18*B*	−150.8 (5)
O8*B*—N2*B*—C27*B*—C26*B*	177.5 (5)
C7*B*—C8*B*—C9*B*—O3*B*	177.4 (4)
C14*B*—C13*B*—C16*B*—O4*B*	172.7 (4)
C21*B*—C20*B*—C23*B*—O6*B*	173.1 (5)

For both compounds, data collection: *SDP* (Enraf–Nonius, 1985); cell refinement: *SDP*; data reduction: *SDP*. Program(s) used to solve structures: *RANTAN* in *MULTAN*11/82 (Main *et al.*, 1982) for (1); *SIR*92 (Altomare *et al.*, 1994) for (2). For both compounds, program(s) used to refine structures: *SDP*; molecular graphics: *ORTEP* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[1,3]Dithiolo[4,5-*b*][1]benzothiophene-2-thione

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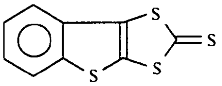
Abstract

The crystals of the title compound, $C_9H_4S_4$, are composed of essentially planar molecules separated by normal van der Waals distances, with S=C and S—C_{sp^2}(mean) bond distances of 1.641 (7) and 1.741 (11) Å, respectively.

Comment

Recently, there has been considerable interest in the area of organic superconductors (Williams *et al.*, 1992). The majority of these incorporate a tetrathiafulvalene (TTF) unit as the electron donor in the conducting salt. Our goal was to a synthesize a novel TTF unit starting from benzo[*b*]thiophene. This was accomplished by a series of reactions starting with the reduction of 2,3-bis(methylthio)benzo[*b*]thiophene, (1) (Clark, Mesher & Primak, 1995). This reaction afforded a yellow oil, (2), that was not amenable to identification and which was reacted further with thiophosgene to afford a yellow solid, (3). Due to the ambiguous identification of (2), compound (3) could not be properly identified. Crystals were thus grown and an X-ray analysis performed, the results of which are described herein.



(3)

An *ORTEP*II (Johnson, 1976) drawing of the title compound, (3), is presented in Fig. 1, together with the atomic numbering scheme. The molecules are essentially planar, with a maximum deviation of 0.057 (9) Å (for atom C8) from the least-squares plane of the S and C atoms. The mean bond distances are: S=C 1.641 (7), S—C_{sp^2} 1.741 (11), C—$C_{aromatic}$ 1.386 (15), C_{sp^2}—C_{sp^2} 1.458 (9) and C=C 1.335 (8) Å. Similar S—C_{sp^2} bond lengths have been reported for the crystal structures of both 2,3-bis(ethylsulfonyl)benzo[*b*]thiophene [1.725 (1) Å; Parvez, Mesher & Clark, 1996*a*] and 2,3,6,7-tetrakis(methylthio)benzo[*b*]thiophene [1.741 (11) Å; Parvez, Mesher & Clark, 1996*b*].

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The bond angle at the S4 atom, 90.4 (3)°, is significantly smaller than the angles at the S1 and S3 atoms, which have values of 96.4 (3) and 95.8 (3)°, respectively. The molecules are separated by normal van der Waals distances and show no abnormal interactions.

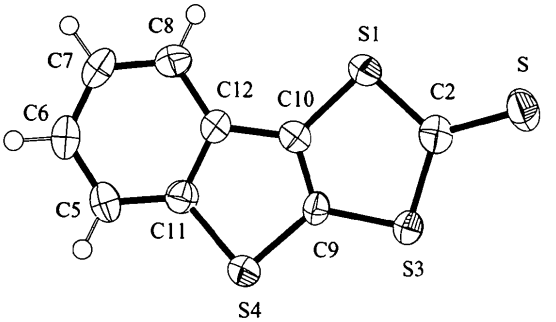

Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids of the non-H atoms are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

Experimental

The title compound was prepared according to Clark, Mesher & Hinman (1996). Crystals in the form of yellow plates were grown from benzene solution on cooling.

Crystal data

$C_9H_4S_4$
$M_r = 240.37$
Monoclinic
$C2/c$
$a = 23.144\,(3)$ Å
$b = 4.036\,(1)$ Å
$c = 20.956\,(3)$ Å
$\beta = 103.89\,(1)^\circ$
$V = 1900.2\,(5)$ Å^3
$Z = 8$
$D_x = 1.680$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–20°
$\mu = 0.940$ mm^{-1}
$T = 296$ K
Plate
0.50 × 0.28 × 0.08 mm
Yellow

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.559$, $T_{\max} = 0.942$
1995 measured reflections
1938 independent reflections

1089 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int} = 0.038$
$\theta_{\max} = 25^\circ$
$h = 0 \rightarrow 26$
$k = 0 \rightarrow 4$
$l = -24 \rightarrow 24$
3 standard reflections monitored every 200 reflections intensity decay: 0.25%

Refinement

Refinement on F
$R = 0.0562$
$wR = 0.0569$

$(\Delta/\sigma)_{\max} = 0.0002$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

$S = 2.856$
1089 reflections
118 parameters
H atoms riding with C—H 0.95 Å
$w = 1/[\sigma^2(F_o) + 0.016(F_o^2)]$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S	0.13766 (9)	−0.2392 (7)	0.65630 (9)	0.0558 (7)
S1	0.08111 (8)	0.0772 (6)	0.75450 (8)	0.0412 (6)
S3	0.20948 (8)	−0.0197 (6)	0.78671 (8)	0.0390 (6)
S4	0.21662 (7)	0.3318 (5)	0.91997 (8)	0.0378 (6)
C2	0.1425 (3)	−0.066 (2)	0.7282 (3)	0.038 (2)
C5	0.1411 (3)	0.632 (2)	0.9903 (3)	0.039 (2)
C6	0.0844 (3)	0.720 (2)	0.9924 (3)	0.046 (2)
C7	0.0364 (3)	0.656 (2)	0.9408 (4)	0.048 (3)
C8	0.0430 (3)	0.501 (2)	0.8849 (3)	0.045 (2)
C9	0.1796 (3)	0.177 (2)	0.8441 (3)	0.033 (2)
C10	0.1208 (3)	0.224 (2)	0.8305 (3)	0.031 (2)
C11	0.1490 (3)	0.470 (2)	0.9343 (3)	0.037 (2)
C12	0.1001 (3)	0.398 (2)	0.8819 (3)	0.030 (2)

Table 2. *Selected geometric parameters (Å, °)*

S—C2	1.641 (7)	S4—C9	1.730 (7)
S1—C2	1.740 (7)	S4—C11	1.754 (7)
S1—C10	1.740 (6)	C9—C10	1.335 (8)
S3—C2	1.741 (7)	C10—C12	1.458 (9)
S3—C9	1.720 (7)		
C2—S1—C10	96.4 (3)	S4—C9—C10	113.6 (5)
C2—S3—C9	95.8 (3)	S1—C10—C9	115.8 (5)
C9—S4—C11	90.4 (3)	S1—C10—C12	130.1 (5)
S—C2—S1	123.5 (4)	C9—C10—C12	114.1 (6)
S—C2—S3	123.0 (4)	S4—C11—C5	126.6 (5)
S1—C2—S3	113.5 (4)	S4—C11—C12	112.6 (5)
S3—C9—S4	128.0 (4)	C8—C12—C10	131.0 (6)
S3—C9—C10	118.4 (5)	C10—C12—C11	109.4 (6)

The space group was determined to be *C2/c* or *Cc* from the systematic absences: *hkl*, $h + k = 2n + 1$; *h*0*l*, $l = 2n + 1$. The former was chosen on the basis of a statistical analysis of intensity distribution and confirmed by the successful solution and refinement of the structure.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with a stereoview of the unit cell, have been deposited with the IUCr (Reference: FG1109). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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p-(Dimethylamino)benzaldehyde Isonicotinoylhydrazone Monohydrate

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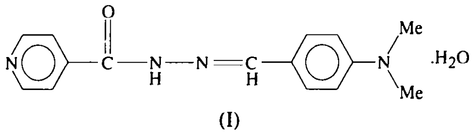
Abstract

The single-crystal X-ray structure determination of the title compound, $C_{15}H_{16}N_4O.H_2O$, reveals that the molecule is in the keto tautomeric form and that the azomethine C=N double bond has the *E* configuration. The water molecule is hydrogen bonded to the hydrazone molecules to form a network structure in the crystal lattice. The crystal structure is stabilized by N—H···O, O—H···O and C—H···O hydrogen bonds.

Comment

The chemical and pharmacological properties of aroylhydrazones have been investigated extensively, owing to their chelating ability with metal ions and to their antineoplastic, antiviral and anti-inflammatory activities (Lu, Wu, Liang, Song & Ma, 1994; Dutta & Hossain, 1985; Rao & Gopal, 1991; Constable & Holmes, 1987; Wester & Palenik, 1973). In this paper we report the structure of *p*-(dimethylamino)benzaldehyde isonicotinoylhydrazone monohydrate, (I).



(I)

Fig. 1 shows an *ORTEP*II (Johnson, 1976) plot of the title compound with the atom-numbering scheme. It can be seen that the molecule is in the keto tautomeric form and that the configuration of the N3—C7 bond is *E*. The bond lengths and angles in the pyridine and dimethylaminophenyl rings are in agreement with those of related structures reported earlier (Wang, Deng, Wu & Chen, 1992; Lu *et al.*, 1995). While the hydrazone moiety is in the plane of the phenyl ring, the pyridine ring makes an angle of 48.08 (7)° with the phenyl ring, and the dimethylamino group is tilted 4.3 (2)° from the plane of the phenyl ring. As a whole, the molecule has an almost planar conformation with the plane of the molecule parallel to the *bc* plane and its axis parallel to the *c* axis. The water molecule lies almost in the plane of the central hydrazone group.

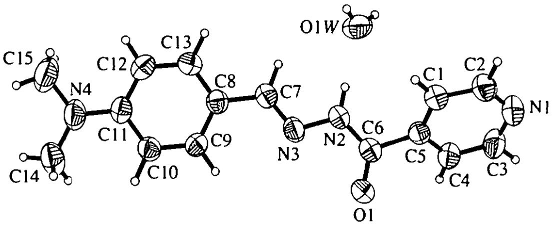


Fig. 1. Displacement ellipsoid plot (50% probability) of the title compound with numbering scheme.

In the crystal lattice, the molecules create a network structure through hydrogen bonds. The water molecule acts as both acceptor and donor in the hydrogen bonds. The keto O1 atom is involved in both O—H···O and C—H···O hydrogen bonds. The details are: N2···O1*W*$^{\rm i}$ 2.749 (3) Å, N2—H2N···O1*W*$^{\rm i}$ 172 (2)°; O1*W*···O1$^{\rm ii}$ 2.741 (3) Å, O1*W*—H1*W*···O1$^{\rm ii}$ 150 (4)°; O1*W*···N1$^{\rm iii}$ 2.839 (3) Å, O1*W*—H2*W*···N1 170 (4)°;

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