

N1	0.96496 (19)	0.5391 (2)	0.34654 (10)	0.0503 (4)
C1	1.1308 (3)	0.5720 (3)	0.30258 (16)	0.0717 (6)
C2	0.8657 (3)	0.6980 (2)	0.37231 (14)	0.0623 (5)
C3	0.9150 (2)	0.3726 (2)	0.36473 (11)	0.0420 (4)
C4	0.6849 (2)	0.1241 (2)	0.44011 (14)	0.0509 (4)
C5	0.5187 (2)	0.0990 (2)	0.49248 (14)	0.0512 (4)

Table 4. Selected bond lengths (Å) for (II)

S1—C3	1.778 (2)	N1—C2	1.465 (2)
S1—C4	1.812 (2)	N1—C1	1.471 (2)
S2—C3	1.675 (2)	C4—C5	1.529 (2)
N1—C3	1.328 (2)	C5—C5'	1.519 (3)

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

The data collection for (II) covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was -35°. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

Both structures were solved by direct methods and refined by full-matrix least-squares techniques. The H atoms of both (I) and (II) were fixed geometrically and allowed to ride on the attached atoms; rotating-group refinement was used for the methyl groups of both structures.

Data collection: XSCANS (Siemens, 1994) for (I); SMART (Siemens, 1996) for (II). Cell refinement: XSCANS for (I); SAINT (Siemens, 1996) for (II). Data reduction: XSCANS for (I); SAINT for (II). For both compounds, program(s) used to solve structures: SHELXTL (Sheldrick, 1997); program(s) used to refine structures: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1520). Services for accessing these data are described at the back of the journal.

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p-Methoxybenzaldehyde isonicotinoyl-hydrazone monohydrate

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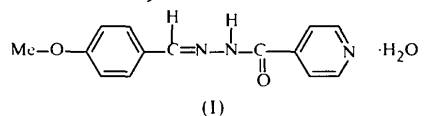
(Received 13 January 1999; accepted 8 February 1999)

Abstract

X-ray analysis reveals that the title compound, C₁₄H₁₃N₃O₂·H₂O, is in keto tautomeric form and the configuration at the azomethine C=N double bond is *E*. The pyridine plane makes a dihedral angle of 39.9(1)° with the plane through the central hydrazone bridge. The crystal structure is stabilized by extensive hydrogen bonding involving the water molecule and the hydrazone bridge.

Comment

Aroylhydrazone compounds containing a pyridyl group have received considerable attention recently. In addition to the existence of a strong coordinating hydrazone group, the pyridyl unit can also serve as a coordination site to form polynuclear complexes. As a continuation of our work on the synthesis and characterization of aroylhydrazone compounds (Fun *et al.*, 1996; Lu, Duan *et al.*, 1996; Lu, Shan *et al.*, 1996; Ma *et al.*, 1994), we report the structure of *p*-methoxybenzaldehyde isonicotinoylhydrazone monohydrate, (I).



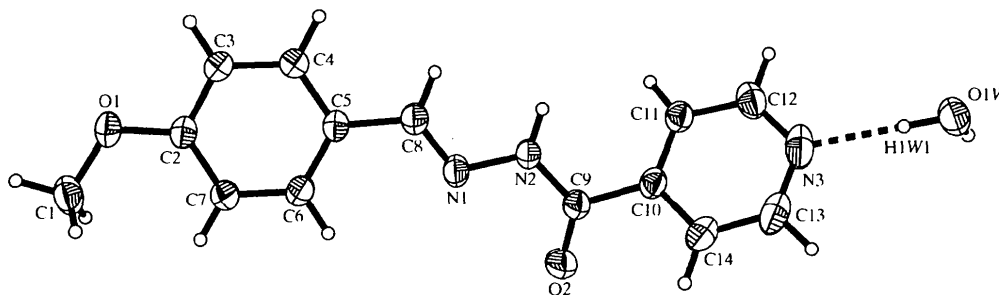


Fig. 1. The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The C9—O2 [1.231 (1) Å] and N1—N2 [1.391 (1) Å] distances, which are consistent with those in the related compound, *p*-methoxybenzaldehyde isonicotinoyl hydrazone dihydrate (Fun *et al.*, 1996), indicate that these bonds correspond to double and single bonds. Thus the molecule is in a keto tautomeric form. It can be seen that the configuration at the N1—C8 is *E*. The C1—O1 and O1—C2 bond lengths [1.432 (2) and 1.370 (1) Å, respectively] in the hydroxyphenyl moiety are consistent with the corresponding ones [1.436 (2) and 1.367 (2) Å, respectively] in the dihydrate. The central hydrazone bridge is planar with torsion angles of $-172.2(1)$ and $176.5(1)^\circ$ about the N2—C9 and N1—N2 bonds, respectively. The O2 atom of the keto group is also in the plane of the central bridge. The dihedral angle between the planes of the pyridine and methoxyphenyl rings is $37.3(1)^\circ$. The methoxyphenyl ring is coplanar with the hydrazone bridge [dihedral angle $8.4(1)^\circ$] and the pyridine ring is twisted by an angle of $39.9(1)^\circ$ with the plane of the same bridge. In *p*-methoxybenzaldehyde nicotinoylhydrazone dihydrate (Fun *et al.*, 1996), the dihedral angle between the planes of the pyridine and methoxyphenyl rings is $9.04(6)^\circ$ and these two planes make angles of $12.15(6)$ and $3.10(6)^\circ$ with the plane of the central hydrazone moiety which shows the almost planar conformation of the molecule. The methoxy group shows a tendency to be coplanar with the phenyl ring [O1 is 0.020 (1) and C1 is 0.013 (2) Å out of plane of the ring] causing enlargement of the angle C7—C2—O1 [$124.8(1)^\circ$] and narrowing of the angle C3—C2—O1 [$115.1(1)^\circ$]. This tendency commonly observed in anisoles, has been justified (Domiano *et al.*, 1979) by some degree of conjugation between O1 and the phenyl ring, giving rise to some shortening of the C_{aryl}—O bond that, in the present case, is C2—O1 = 1.370 (1) Å. The water molecule present in the unit cell deviates from the best plane through the whole molecule by 1.274 (1) and 1.139 (1) Å from the hydrazone group.

In the crystal, the hydrazone molecules lie parallel to one another in the crystal lattice and are aligned perpendicular to the *a* axis as observed in the related compound (Fun *et al.*, 1996) with two water molecules. The water and hydroxyl group are involved in a variety of N—H...O and O—H...N hydrogen bonds.

Experimental

The synthesis of the compound was carried out by the reaction of *p*-methoxybenzaldehyde and isonicotinoylhydrazone in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol. The water molecule in the crystal lattice may have come from the ethanol used for the synthesis and recrystallization. The difference in the water content of this compound and *p*-methoxybenzaldehyde nicotinoylhydrazone dihydrate (Fun *et al.*, 1996) may be due to the water content of the ethanol solvent.

Crystal data

C₁₄H₁₃N₃O₂·H₂O
M_r = 273.29
 Monoclinic
*P*2₁/*c*
a = 7.3875 (2) Å
b = 12.4718 (3) Å
c = 14.8382 (4) Å
 β = 96.727 (1) $^\circ$
V = 1357.71 (6) Å³
Z = 4
D_x = 1.337 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 6564 reflections
 θ = 2.76–33.24 $^\circ$
 μ = 0.096 mm⁻¹
T = 293 (2) K
 Parallelepiped
 0.50 × 0.42 × 0.32 mm
 Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: none
 12180 measured reflections
 5003 independent reflections

3596 reflections with $I > 2\sigma(I)$
*R*_{int} = 0.021
 θ_{\max} = 33.09 $^\circ$
h = $-11 \rightarrow 10$
k = $-19 \rightarrow 8$
l = $-21 \rightarrow 22$

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.049
 $wR(F^2)$ = 0.141
S = 1.056
 5003 reflections
 241 parameters
 All H-parameters atom refined

$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 0.1724P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.313 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.169 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

N1—C8	1.281 (2)	O1—C1	1.432 (2)
N1—N2	1.391 (1)	O2—C9	1.231 (1)
N2—C9	1.349 (1)	C5—C8	1.466 (1)
O1—C2	1.370 (1)	C9—C10	1.509 (2)
C8—N1—N2	113.59 (9)	O2—C9—C10	121.3 (1)
C9—N2—N1	119.45 (9)	N2—C9—C10	114.27 (9)
O2—C9—N2	124.4 (1)		
C8—N1—N2—C9	176.5 (1)	N1—N2—C9—O2	5.0 (2)
N2—N1—C8—C5	-175.9 (1)	N1—N2—C9—C10	-172.2 (1)

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

D—H...A	D—H	H...A	D...A	D—H...A
O1W—H1W1...N3	0.90 (2)	1.92 (2)	2.820 (2)	179 (2)
N2—H1N2...O1W ^a	0.92 (2)	2.04 (2)	2.945 (1)	171 (1)
O1W—H2W1...O2 ^b	0.88 (2)	2.00 (2)	2.886 (2)	177 (2)

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

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 10 s covered 0.3° in ω . The crystal-to-detector distance was 4 cm and the detector swing angle was -35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1409). Services for accessing these data are described at the back of the journal.

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2-Acetyl-6-phenyl-5,6,7,8-tetrahydro-1-naphthyl *p*-toluenesulfonate

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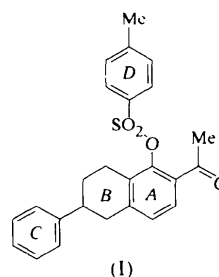
(Received 23 November 1998; accepted 28 January 1999)

Abstract

In the title compound, C₂₅H₂₄O₄S, the S atom is in a distorted tetrahedral geometry. The cyclohexene ring in the structure adopts the sofa conformation. The acetyl group is twisted from the benzene ring by 37.4 (5)°. The equatorially attached phenyl ring forms a dihedral angle of 68.1 (2)° with the cyclohexene ring.

Comment

Naphthalene derivatives are widely used as intermediates in the synthesis of several polycyclic phenols which are useful as antifibrillatory agents, disinfectants and water softeners (Hauck *et al.*, 1977). β -Keto derivatives of naphthalene serve as useful synthetic intermediates in the synthesis of the subunits of daunomycinone and adiramycin, which are important anticancer drugs (Crouse *et al.*, 1981). The structure determination of the title compound, (I), was carried out in order to elucidate its molecular conformation.



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