

D—H···A      D—H    H···A      D···A      D—H···A  
 O4—H8···O1      0.91    2.02      2.836 (4)      148

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

The H8 atom was fixed at its difference Fourier map position. The remaining H atoms were placed at assumed positions (C—H 0.95 Å,  $U = 1.2U_{eq}$  of the associated atom) and fixed.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP* (Johnson, 1965) in *TEXSAN*. Software used to prepare material for publication: *TEXSAN FINISH*; *PLATON* (Spek, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1127). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *p*-Methoxybenzaldehyde Nicotinoylhydrazone Dihydrate

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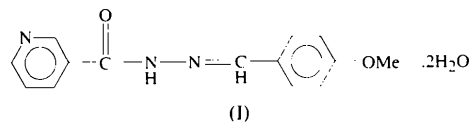
(Received 19 October 1995; accepted 10 November 1995)

### Abstract

The nearly planar aroylhydrazone molecule of the title compound, C<sub>14</sub>H<sub>13</sub>N<sub>3</sub>O<sub>2</sub>·2H<sub>2</sub>O, exists as the keto tautomer in the solid state with an *E* configuration across the C=N bond. The crystal structure is stabilized by extensive hydrogen bonding involving the water and the hydrazone molecules.

### Comment

In recent years there has been considerable interest in the chemistry of aroylhydrazone compounds because of their ability to chelate transition and lanthanide metal ions (Dutta & Hossain, 1985; Sergienko, Abramenko, Minacheva, Porai-Koshits & Sakharova, 1993; Rao & Gopal, 1991) and their medical properties (Constable & Holmes, 1987; Girges, Hanna, Berghot & Rassala, 1992). As part of our studies on the synthesis and characterization of these compounds, we determined the structure of the title compound, (I).



The C6—O1 [1.224 (2) Å] and N2—N3 [1.382 (2) Å] bond lengths in (I) (Fig. 1) indicate bond orders of two and one, respectively, confirming that the crystal contains the keto tautomer. The hydrazone moiety has an *E* configuration at the N3=C7 double bond. The dihedral angle between the planes of the pyridine and methoxyphenyl rings is 9.04 (6)° and these two planes make angles of 12.15 (6)° and 3.10 (6)° with the plane of the central hydrazone moiety; the methoxy group C

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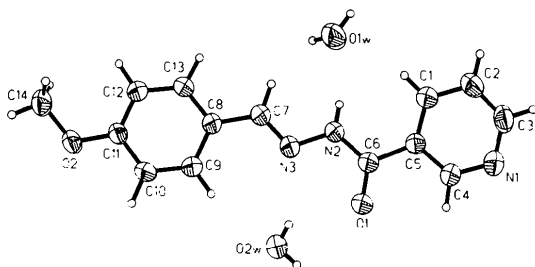


Fig. 1. Displacement ellipsoid plot (50% probability) of the title compound showing the atomic numbering scheme.

atom, C14, deviates by 0.316(3) Å from the plane of the phenyl ring. Thus the molecule is roughly planar.

The hydrazone molecules lie parallel to one another in the crystal lattice and are aligned perpendicular to the *a* axis. The two water molecules present are involved in a variety of N—H...O, O—H...O and C—H...O hydrogen bonds (Table 3). The water molecules (W) link hydrazone molecules (Hyd) related by centres of inversion through HydA...W1A...W2A...HydB...W1B...W2B...HydA loops of hydrogen bonds (Fig. 2).

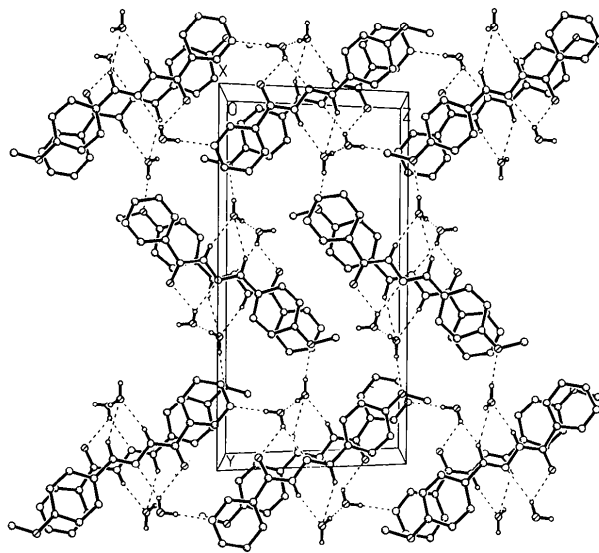


Fig. 2. Packing of the molecules viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

In our previous studies we observed that aroylhydrazones usually crystallize as monohydrates (Fun *et al.*, 1996; Lu *et al.*, 1996), though (I) is a dihydrate. In all cases, N2—H is hydrogen-bonded to a water molecule.

## Experimental

The title compound was prepared by the reaction of *p*-methoxybenzaldehyde and nicotinoylhydrazine under reflux in ethanol solution for 3 h. Single crystals were obtained by recrystallization from ethanol. The water molecules in the crystal lattice may have come from the ethanol used for the synthesis and recrystallization.

### Crystal data

C<sub>14</sub>H<sub>13</sub>N<sub>3</sub>O<sub>2</sub>·2H<sub>2</sub>O

*M<sub>r</sub>* = 291.31

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 7.162(1) Å

*b* = 20.033(2) Å

*c* = 10.611(1) Å

β = 107.21(1)°

*V* = 1454.3(3) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.331 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 8–25°

μ = 0.099 mm<sup>-1</sup>

*T* = 293(2) K

Needle

0.58 × 0.38 × 0.18 mm

Colourless

### Data collection

Siemens *P4* diffractometer

θ/2θ scans

Absorption correction:

none

4351 measured reflections

3333 independent reflections

2464 observed reflections

[*I* > 2σ(*I*)]

*R<sub>int</sub>* = 0.0166

θ<sub>max</sub> = 27.5°

*h* = -1 → 9

*k* = -1 → 26

*l* = -13 → 13

3 standard reflections

monitored every 97

reflections

intensity decay: <3%

### Refinement

Refinement on *F*<sup>2</sup>

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.0466

*wR*(*F*<sup>2</sup>) = 0.1426

*S* = 1.063

3333 reflections

259 parameters

All H-atom parameters

refined

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0870*P*)<sup>2</sup> + 0.0425*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> < 0.001

Δρ<sub>max</sub> = 0.22 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.19 e Å<sup>-3</sup>

Extinction correction:

*SHELXL93* (Sheldrick, 1993)

Extinction coefficient:

0.0113(24)

Atomic scattering factors

from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
O1	0.8066 (2)	0.48349 (5)	0.29452 (10)	0.0616 (3)
O2	0.6865 (2)	0.31441 (5)	-0.48228 (10)	0.0589 (3)
N1	0.8651 (2)	0.64690 (7)	0.53884 (13)	0.0628 (4)
N2	0.7599 (2)	0.54708 (6)	0.11249 (11)	0.0448 (3)
N3	0.7613 (2)	0.49208 (6)	0.03411 (11)	0.0460 (3)
C1	0.7330 (3)	0.66345 (7)	0.2679 (2)	0.0543 (4)
C2	0.7432 (3)	0.71686 (8)	0.3522 (2)	0.0636 (5)
C3	0.8093 (3)	0.70620 (9)	0.4850 (2)	0.0630 (5)
C4	0.8536 (3)	0.59547 (8)	0.45646 (15)	0.0521 (4)
C5	0.7896 (2)	0.60090 (6)	0.32062 (13)	0.0407 (3)
C6	0.7864 (2)	0.53857 (6)	0.24237 (13)	0.0423 (3)

C7	0.7265 (2)	0.50523 (7)	-0.08808 (14)	0.0473 (3)
C8	0.7248 (2)	0.45456 (6)	-0.18741 (13)	0.0415 (3)
C9	0.7567 (2)	0.38670 (7)	-0.15955 (14)	0.0490 (4)
C10	0.7451 (3)	0.34165 (7)	-0.25929 (14)	0.0510 (4)
C11	0.7009 (2)	0.36315 (6)	-0.38967 (13)	0.0426 (3)
C12	0.6767 (2)	0.43024 (7)	-0.41855 (13)	0.0469 (3)
C13	0.6896 (2)	0.47504 (7)	-0.31701 (14)	0.0474 (4)
C14	0.6092 (3)	0.33325 (9)	-0.6184 (2)	0.0638 (5)
O1W	0.7643 (3)	0.67018 (7)	-0.04812 (14)	0.0793 (5)
O2W	0.9830 (3)	0.37037 (6)	0.18312 (14)	0.0723 (4)

Table 2. Selected bond lengths (Å)

O1—C6	1.224 (2)	C2—C3	1.363 (3)
O2—C11	1.367 (2)	C4—C5	1.382 (2)
O2—C14	1.436 (2)	C5—C6	1.496 (2)
N1—C3	1.328 (2)	C7—C8	1.461 (2)
N1—C4	1.338 (2)	C8—C13	1.385 (2)
N2—C6	1.345 (2)	C8—C9	1.396 (2)
N2—N3	1.382 (2)	C9—C10	1.375 (2)
N3—C7	1.273 (2)	C10—C11	1.393 (2)
C1—C2	1.382 (2)	C11—C12	1.378 (2)
C1—C5	1.383 (2)	C12—C13	1.384 (2)

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O1W—H1W1...O2W <sup>i</sup>	0.94 (3)	1.81 (3)	2.743 (3)	174 (2)
O1W—H2W1...O2 <sup>ii</sup>	0.88 (3)	2.05 (3)	2.917 (2)	169 (3)
O2W—H1W2...O1	0.89 (3)	2.31 (4)	3.002 (2)	135 (3)
O2W—H1W2...N3	0.89 (3)	2.29 (3)	3.079 (2)	148 (3)
O2W—H2W2...N1 <sup>iii</sup>	0.82 (3)	2.03 (3)	2.847 (2)	177 (3)
N2—H2N...O1W	0.84 (2)	2.18 (2)	3.003 (2)	164 (2)
C1—H1...O1W	0.94 (2)	2.61 (3)	3.429 (2)	146 (2)
C2—H2...O2W <sup>iv</sup>	0.95 (2)	2.62 (2)	3.444 (2)	145 (2)
C7—H7...O1W	0.98 (2)	2.47 (2)	3.332 (2)	146 (2)
C9—H9...O2W	0.97 (2)	2.63 (2)	3.531 (2)	156 (2)

Symmetry codes: (i)  $2 - x, 1 - y, -z$ ; (ii)  $\frac{3}{2} - x, \frac{1}{2} + y, -\frac{1}{2} - z$ ; (iii)  $2 - x, 1 - y, 1 - z$ ; (iv)  $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ .

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: ORTEPII (Johnson, 1976) and SHELXTL/PC. Software used to prepare material for publication: SHELXL93. Geometrical calculations: PARST (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1230). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## (2R,3R)-3-Ammonio-1-(1-methoxycarbonyl-2-methyl-1-propenyl)-4-oxoazetidene-2-sulfonate Monohydrate: a New Chiral $\beta$ -Lactam Synthone

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## Abstract

The title compound, a novel monocyclic  $\beta$ -lactam, was prepared and chemically characterized by IR and <sup>1</sup>H NMR spectroscopy and X-ray structure analysis. C<sub>9</sub>H<sub>14</sub>N<sub>2</sub>O<sub>6</sub>S·H<sub>2</sub>O exists as a zwitterion crystallized with a water molecule. The four-membered lactam ring is slightly puckered, the ammonio and sulfonate groups are *cis*, and the aliphatic substituent is *trans* to these polar groups. Hydrogen bonds link the zwitterions and the water molecules into spirals parallel to the *b* axis, forming a two-dimensional network with base vectors along (100) and (010).

## Comment

A foamy sample of 3-phenoxyacetamino-4-oxoazetidene-2-sulfonic acid methyl ester, (1), prepared as described earlier (Kovačević *et al.*, 1992) changed colour and form on standing at room temperature. The formation of three new compounds was detected by monitoring the change by TLC over approximately two weeks. The most polar product was precipitated out by acetone and isolated by filtration. IR spectroscopy showed