Crystal data

$C_{18}H_{12}Cl_3OP$
M_r = 381.60
Triclinic
$P\bar{1}$
a = 6.1930 (10) Å
b = 9.0770 (10) Å
c = 15.8820 (2) Å
α = 90.690 (10)°
β = 96.390 (10)°
γ = 101.750 (10)°
V = 868.1 (2) Å^3
Z = 2
D_x = 1.460 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 39 reflections
θ = 4.82–12.46°
μ = 0.620 mm^{-1}
T = 293 (2) K
Thin rod
0.46 × 0.22 × 0.22 mm
Light yellow

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical ψ scans (Siemens, 1994)
T_{min} = 0.637, T_{max} = 0.872
5055 measured reflections
3963 independent reflections
2387 reflections with $I > 2\sigma(I)$

R_{int} = 0.019
θ_{max} = 27.49°
$h = -1 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$
3 standard reflections every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.175
S = 0.908
3963 reflections
256 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1125P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

P—O	1.483 (3)	Cl1—C3	1.740 (4)
P—C7	1.810 (3)	Cl2—C9	1.741 (4)
P—C13	1.812 (3)	Cl3—C15	1.744 (4)
P—C1	1.816 (3)		
O—P—C7	111.7 (2)	C6—C1—P	123.9 (3)
O—P—C13	111.88 (14)	C2—C1—P	116.4 (3)
C7—P—C13	108.69 (14)	C12—C7—P	124.7 (3)
O—P—C1	112.35 (14)	C8—C7—P	115.3 (2)
C7—P—C1	105.16 (14)	C18—C13—P	124.3 (3)
C13—P—C1	106.7 (2)	C14—C13—P	115.8 (3)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: direct methods *SHELXTL/PC* (Sheldrick, 1990) and *PARST* (Nardelli, 1983). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1324). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1452–1454

p-Nitrobenzaldehyde Isonicotinoylhydrazone

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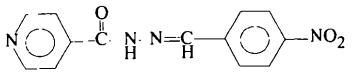
(Received 25 March 1997; accepted 30 May 1997)

Abstract

Molecules of the hydrazone $C_{13}H_{10}N_4O_3$ are planar and exist in the keto tautomeric form. The configuration at the azomethine C=N double bond is *E*. The structure is stabilized by a network of hydrogen bonds.

Comment

Because of their chemical and pharmacological properties, aroylhydrazines and related compounds have been studied extensively (Lu *et al.*, 1994; Sergienko, Abramenko, Minacheva, Porai-Koshits & Sakharova, 1993; Dutta & Hossain, 1985). As part of our work on the synthesis and characterization of new aroylhydrazone complexes, we report here the structure of *p*-nitrobenzaldehyde isonicotinoylhydrazone, (I).



(I)

The hydrazone moiety is in the plane of the phenyl ring (Fig. 1). The pyridine ring and nitro group make angles of 8.13 (6) and 5.2 (1)°, respectively, with the plane of the phenyl ring. The molecule is thus essentially planar. Bond lengths and angles observed here agree well with those found in crystals of *p*-nitrobenzaldehyde nicotinoylhydrazone monohydrate (Lu *et al.*, 1996), which contain molecules isomeric with those of the title compound.

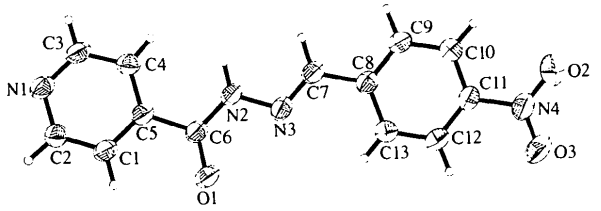


Fig. 1. Structure of title compound showing the numbering scheme and 50% probability ellipsoids.

In the crystal, the molecules pack as a network structure through hydrogen bonds. The pyridine N1 atom is involved in an N—H···N hydrogen bond; it also has close contacts with C4 and C7. The nitro O3 and keto O1 atoms are hydrogen-bonded to C atoms. The details are: C3···O1^{i} 3.365 (2) Å and C3—H3···O1^{i} 150 (1)°, N2···N1^{ii} 3.032 (2) Å and N2—H1N2···N1^{ii} 164 (1)°, C4···N1^{ii} 3.432 (2) Å and C4—H4···N1^{ii} 143 (1)°, C7···N1^{ii} 3.494 (2) Å and C7—H7···N1^{ii} 134 (1)°, and C9···O3^{iii} 3.335 (2) Å and C9—H9···O3^{iii} 141 (1)°; symmetry codes: (i) x, $-y+\frac{1}{2}$, $z-\frac{1}{2}$; (ii) $-x+1$, $y-\frac{1}{2}$, $-z+\frac{3}{2}$; (iii) x, $-y-\frac{1}{2}$, $z-\frac{1}{2}$.

Experimental

The title compound was synthesized by reaction of *p*-nitrobenzaldehyde and isonicotinoyl hydrazine in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{13}H_{10}N_4O_3$
M_r = 270.25
Monoclinic
$P2_1/c$
a = 7.957 (1) Å
b = 10.677 (1) Å
c = 14.909 (2) Å
β = 100.51 (1)°
V = 1245.4 (3) Å^3
Z = 4
D_x = 1.441 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 42 reflections
θ = 5.40–12.47°
μ = 0.107 mm^{-1}
T = 293 (2) K
Rectangular slab
0.52 × 0.38 × 0.28 mm
Yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3846 measured reflections
2871 independent reflections
1756 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.50°
$h = -1 \rightarrow 10$
$k = -1 \rightarrow 13$
$l = -19 \rightarrow 19$
3 standard reflections every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 0.903
2871 reflections
222 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.016 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C6	1.217 (2)	N3—C7	1.266 (2)
O2—N4	1.219 (2)	N4—C11	1.471 (2)
O3—N4	1.213 (2)	C5—C6	1.506 (2)
N2—C6	1.353 (2)	C7—C8	1.467 (2)
N2—N3	1.381 (2)		
C6—N2—N3	118.41 (12)	O1—C6—C5	120.61 (14)
C7—N3—N2	115.78 (12)	N2—C6—C5	116.19 (12)
O1—C6—N2	123.19 (14)	N3—C7—C8	120.93 (13)

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from difference Fourier maps and refined isotropically.

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: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Program used for geometrical calculations: *PARST* (Nardelli, 1995). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1325). Services for accessing these data are described at the back of the journal.

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p-(Dimethylamino)benzaldehyde Benzoylhydrazone Monohydrate

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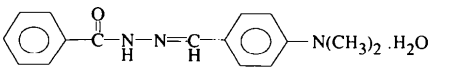
(*Received 26 March 1997; accepted 30 May 1997*)

Abstract

The title compound, $C_{16}H_{17}N_3O.H_2O$, adopts the keto tautomeric form and the azomethine C=N double bond is in the *E* configuration. The crystal structure is stabilized by O—H···O, O—H···N, C—H···O and N—H···O hydrogen bonds between the hydrazone and water molecules.

Comment

In recent years transition metal and lanthanide complexes of aroylhydrazones have been investigated extensively because of their biological activity, especially as potent inhibitors for many enzymes (Ma, Lu, Song & Wu, 1994; Dutta & Hossain, 1985; Han, Jin, Huang & Ma, 1991). As part of our research on the synthesis and characterization of these complexes, we report here the structure of *p*-(dimethylamino)benzaldehyde benzoylhydrazone monohydrate, (I).



(I)

Bond lengths and angles in this structure are comparable with those observed in related structures reported previously (Lu *et al.*, 1995; Fun *et al.*, 1996). The hydrazone moiety is in the plane of the dimethylaminophenyl ring (Fig. 1) and the dihedral angle between the two phenyl rings is 35.76 (9)°. The crystal structure is stabilized by hydrogen bonds (Table 2) between the hydrazone and water molecules, which act as both H-atom acceptors and donors.

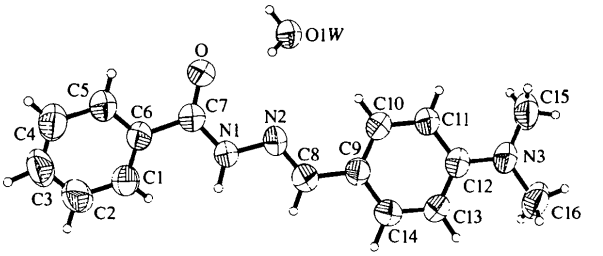


Fig. 1. A view of the title compound showing the numbering scheme and 50% probability ellipsoids.

Experimental

The synthesis of the title compound was carried out by reaction of *p*-(dimethylamino)benzaldehyde and benzoylhydrazine in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{16}H_{17}N_3O.H_2O$
M_r = 285.34
Monoclinic
$P2_1/c$
a = 13.531 (1) Å
b = 11.766 (1) Å
c = 10.272 (3) Å
β = 106.71 (1)°
V = 1566.3 (5) Å^3
Z = 4
D_x = 1.210 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 36 reflections
θ = 5.11–11.90°
μ = 0.082 mm^{-1}
T = 293 (2) K
Needle
0.96 × 0.24 × 0.16 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4590 measured reflections
3600 independent reflections
1294 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.49°
$h = -17 \rightarrow 17$
$k = -15 \rightarrow 1$
$l = -1 \rightarrow 13$
3 standard reflections every 97 reflections
intensity decay: <3%