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***p*-(Dimethylamino)benzaldehyde Benzoylhydrazone Monohydrate**

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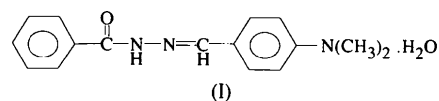
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Abstract

The title compound, C₁₆H₁₇N₃O.H₂O, 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).



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.

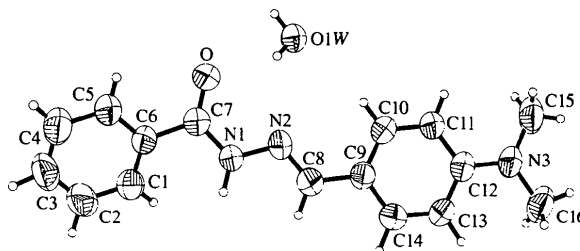


Fig. 1. A view of the title compound showing the numbering scheme and 50% probability ellipsoids.

Experimental

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

Crystal data

C₁₆H₁₇N₃O.H₂O
M_r = 285.34
 Monoclinic
*P*2₁/*c*
a = 13.531 (1) Å
b = 11.766 (1) Å
c = 10.272 (3) Å
 β = 106.71 (1)°
V = 1566.3 (5) Å³
Z = 4
D_x = 1.210 Mg m⁻³
D_m not measured

Mo *K*α 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 P4 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 → 17
k = -15 → 1
l = -1 → 13
 3 standard reflections
 every 97 reflections
 intensity decay: <3%

Refinement

Refinement on F^2	$\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.126$	Extinction correction:
$S = 0.788$	<i>SHELXL93</i> (Sheldrick, 1993)
3600 reflections	Extinction coefficient:
267 parameters	0.0249 (18)
All H atoms refined	Scattering factors from
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$	<i>International Tables for Crystallography</i> (Vol. C)
where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{\max} < 0.001$	

Table 1. Selected geometric parameters (\AA , $^\circ$)

O—C7	1.230 (2)	N3—C15	1.428 (4)
N1—C7	1.344 (3)	N3—C16	1.455 (4)
N1—N2	1.388 (2)	C6—C7	1.497 (3)
N2—C8	1.283 (3)	C8—C9	1.461 (3)
N3—C12	1.362 (3)		
C7—N1—N2	118.9 (2)	O—C7—C6	121.6 (2)
C8—N2—N1	113.8 (2)	N1—C7—C6	114.7 (2)
O—C7—N1	123.7 (2)	N2—C8—C9	122.1 (2)

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1W—H2W \cdots O	0.84 (3)	2.14 (3)	2.898 (2)	149 (2)
O1W—H2W \cdots N2	0.84 (3)	2.50 (3)	3.148 (3)	134 (2)
C8—H8 \cdots O1W'	0.96 (2)	2.57 (2)	3.366 (3)	141 (1)
N1—H1N1 \cdots O1W'	0.94 (3)	1.97 (3)	2.892 (3)	170 (2)
O1W—H1W \cdots O''	0.90 (2)	1.98 (2)	2.881 (3)	170 (2)

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

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: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Program used for geometrical calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1326). Services for accessing these data are described at the back of the journal.

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The Chelating Agent N,N,N',N'',N''',N'''' -Triethylenetetraminehexacetic Acid (TTHA)

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Abstract

The title compound, $C_{18}H_{30}N_4O_{12}$, exists as an unsymmetrical tri-zwitterion. The protonated N atoms form intramolecular hydrogen bonds to carboxylate O atoms. The molecules are linked by strong intermolecular hydrogen bonds formed by three COOH groups at one end of the molecule and three carboxylates at the negative ends of the two nearest neighbors [O \cdots H 1.45 (3) \AA]. The C—N bond lengths are clearly differentiated with respect to the charge on the N atom [C—N 1.459 (6) and C—NH $^+$ 1.502 (9) \AA].

Comment

Metal complexes of polyaminocarboxylates have been studied widely by coordination chemists since the preparation of ethylenediaminetetracetic acid (EDTA); the equilibria and species involved in metal ion/EDTA systems are well understood (Schwarzenbach, 1960). In recent years, there has been a resurgence of interest in this area of chemistry, firstly to provide treatment for heavy-metal intoxication (*e.g.* Tandon & Srivastava, 1985), secondly to model metal-ion sites in biomolecules (Finnen, Pinkerton, Dunham, Sands & Funk, 1991) and thirdly to prepare contrast agents for magnetic resonance imaging (MRI) (Lauffer, 1987; Tweedle, 1989). The search for new and better chelating agents led to linear extension of flexible molecules,