

***N*-(4-Methoxybenzylidene)-*N'*-(2-pyridyl)hydrazine**Tuncay Tunc,<sup>a\*</sup> Musa Sarı,<sup>a</sup> Rahmi Yagbasan,<sup>a</sup> Habibe Tezcan<sup>a</sup> and Ertan Şahin<sup>b</sup><sup>a</sup>Department of Physics Education, Gazi University, Beşevler, 06500 Ankara, Turkey, and <sup>b</sup>Department of Engineering Physics, Hacettepe University, Beytepe, 06532 Ankara, Turkey

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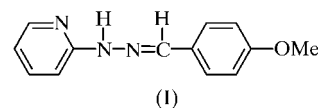
Molecules of the title compound (alternative name *p*-methoxybenzaldehyde 2-pyridylhydrazone), C<sub>13</sub>H<sub>13</sub>N<sub>3</sub>O, adopt an *E* configuration about the azomethine C=N double bond. Molecules are almost planar, the dihedral angle between the pyridine and methoxyphenyl rings being only 6.19 (12)°. Pairwise N—H···N hydrogen bonds [*R*<sub>2</sub><sup>2</sup>(8) in graph-set notation] link centrosymmetrically related molecules into discrete pairs.

**Comment**

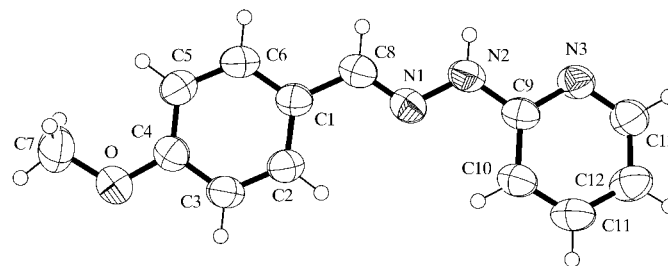
Hydrazones have been widely studied as chelating ligands for the spectrophotometric and fluorimetric determination of trace metal ions (Katyal & Dutt, 1975; Galiano-Roth & Collum, 1988). Many of these compounds have found widespread application in medicine, technology and analytical chemistry (Kitaev, 1977). In recent years, there has been considerable interest in arylhydrazone compounds containing a pyridyl group. Hydrazones are formed when hydrazines condense with aldehydes and ketones, *e.g.* by the condensation reaction of aldehydes (or substituted aldehydes) with phenylhydrazine at a pH of 4–5 (McMurry, 1999), and typically are crystalline compounds with sharp melting points. These compounds can therefore be used to identify the aldehydes and ketones from which the hydrazones have been formed (McMurry, 1999). Hydrazones are frequently more suitable than oximes for this purpose, since the greater molecular weight of the hydrazones causes a lower solubility in most solvents, and they can therefore often be more easily isolated and recrystallized.

Molecules of the title compound, (I), adopt an *E* configuration about the azomethine C=N double bond, with an N2—N1—C8—C1 torsion angle of −179.54 (19)° (Fig. 1). Overall, the molecule is planar, the r.m.s. deviation of the non-H atoms from the least-squares mean plane being only 0.047 Å.

The N1—N2 and N1=C8 bond distances (Table 1) are comparable to those in the related compounds *p*-methoxybenzaldehyde nicotinoylhydrazone dihydrate (Fun *et al.*, 1996) and *p*-methoxybenzaldehyde isonicotinoylhydrazone monohydrate (Shanmuga Sundara Raj *et al.*, 1999, 2000). There is only a slight asymmetry of the exocyclic angles at C1 [C2—C1—C8 = 121.72 (19)° and C6—C1—C8 = 120.84 (19)°], while the asymmetry at C4 is more pronounced [O—C4—C3 = 115.33 (18)° and O—C4—C5 = 125.07 (19)°]. This situation is typical of that found in anisoles and is caused by the tendency of the methoxy group to be coplanar with the phenyl ring through the conjugation of the O atom with the phenyl ring (Domiano *et al.*, 1979). In (I) the methyl C atom shows no significant deviation from the plane of the phenyl ring. There is also asymmetry in the exocyclic angles at C9 (Table 1). The dihedral angle between the planes of the pyridine and methoxyphenyl rings is 6.19 (12)°, and these two planes make dihedral angles with the central hydrazone bridge (N2/N1/C8) of 7.59 (3) and 1.41 (3)°, respectively.



Centrosymmetrically related molecules are linked into discrete pairs by pairwise N2—H2A···N3<sup>i</sup> hydrogen bonds [symmetry code: (i) 2 − *x*, 2 − *y*, −*z*; *R*<sub>2</sub><sup>2</sup>(8) in graph-set

**Figure 1**

The molecular structure and atom-labelling scheme of (I) (ORTEP-3; Farrugia, 1997). Displacement ellipsoids are shown at the 50% probability level.

notation]. The N—H···N and N···N distances (Table 2) are short compared with the sums of the relevant van der Waals radii (Bondi, 1964).

**Experimental**

A solution of 2-hydrazinopyridine (3.32 g, 0.030 mol) in hot methanol (25 ml) was added dropwise to a solution of 4-methoxybenzaldehyde (4.10 g, 0.030 mol) in hot methanol (22 ml) at 298–303 K. The resulting yellow solid was filtered off, dried and recrystallized after refluxing in hot methanol for 4 h. The yellow crystals thus obtained were filtered off and dried in air.

## Crystal data

$C_{13}H_{13}N_3O$	$D_x = 1.283 \text{ Mg m}^{-3}$
$M_r = 227.26$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 5.5633 (16) \text{ \AA}$	$\theta = 2.2\text{--}25.7^\circ$
$b = 10.4599 (11) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 20.314 (4) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 95.51 (2)^\circ$	Block, yellow
$V = 1176.6 (4) \text{ \AA}^3$	$0.30 \times 0.27 \times 0.21 \text{ mm}$
$Z = 4$	

## Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{\max} = 25.7^\circ$
$\omega/2\theta$ scans	$h = -6 \rightarrow 0$
2461 measured reflections	$k = 0 \rightarrow 12$
2223 independent reflections	$l = -24 \rightarrow 24$
1177 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{\text{int}} = 0.021$	frequency: 120 min
	intensity decay: 3%

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.057P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 0.99$	$\Delta\rho_{\max} = 0.11 \text{ e \AA}^{-3}$
2223 reflections	$\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
154 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O—C4	1.367 (2)	N2—C9	1.374 (2)
O—C7	1.415 (2)	N3—C13	1.340 (2)
N1—C8	1.270 (2)	N3—C9	1.336 (2)
N1—N2	1.367 (2)	C1—C8	1.459 (3)
C4—O—C7	119.11 (17)	O—C4—C3	115.33 (18)
C8—N1—N2	118.18 (18)	C3—C4—C5	119.59 (19)
N1—N2—C9	118.12 (19)	N1—C8—C1	121.00 (19)
C9—N3—C13	116.38 (18)	N2—C9—C10	121.74 (18)
C2—C1—C8	121.72 (19)	N3—C9—N2	115.37 (17)
O—C4—C5	125.07 (19)		

H atoms were placed geometrically at distances of 0.86, 0.93 and 0.96  $\text{\AA}$  from their parent atoms for N—H, methyl C—H and  $sp^2$  C—H bonds, respectively. In the refinement, a riding model was used for all H atoms, with  $U_{\text{iso}}(\text{H})$  equal to  $1.3U_{\text{eq}}(\text{C,N})$ .

Table 2

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{N2---H2A}\cdots\text{N3}^i$	0.86	2.27	3.119 (4)	169

Symmetry code: (i)  $2 - x, 2 - y, -z$ .

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1525). Services for accessing these data are described at the back of the journal.

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