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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C})$ = 0.003 Å
R factor = 0.041
wR factor = 0.101
Data-to-parameter ratio = 11.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The Schiff base 3,4-methylenedioxybenzaldehyde semicarbazone

The synthesis of the title compound, $\text{C}_9\text{H}_9\text{N}_3\text{O}_3$, was achieved by the condensation of 3,4-methylenedioxybenzaldehyde with semicarbazide hydrochloride. The molecule is approximately planar: the dihedral angles of the 3,4-methylenedioxybenzene and amide moieties with the hydrazone moiety are 10.2 (2) and 8.9 (3)°, respectively. Intra- and intermolecular hydrogen bonds generate a two-dimensional network. Aromatic stacking interactions are also found in the crystal structure.

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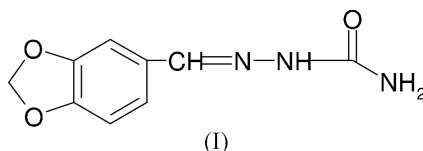
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Comment

Various benzoylhydrazones, derived from aryl aldehydes, phenylalkyl aldehydes and phenylalkyl ketones and some related compounds, have been evaluated for anticonvulsant activity (He *et al.*, 2002). Most of the compounds displayed anticonvulsant activity in the maximal electroshock (MES) and subcutaneous pentylenetetrazole (Sc PTZ) screens, accompanied by neurotoxicity, when given to mice by the intraperitoneal route (Desiraju & Kishan, 1989).

The title compound, (I), prepared by the condensation of 3,4-methylenedioxybenzaldehyde and semicarbazide hydrochloride, is neutral. A view of the molecular structure is shown in Fig. 1.



The molecule of (I) consists of 3,4-methylenedioxybenzene, amide and hydrazone groups, and is approximately planar. The largest deviation from the 3,4-methylenedioxybenzene plane is 0.0137 (2) Å, for atom C10. The dihedral angles formed by the 3,4-methylenedioxybenzene and amide groups with the hydrazone group are 10.2 (2) and 8.9 (3)°, respectively. The N1–C1 bond length 1.274 (2) Å is close to the value of 1.280 (5) Å in *p*-dimethylaminebenzaldehyde 2,4-dinitrobenzaldehyde hydrazone (Wang *et al.*, 2004) and smaller than the value of 1.337 (2) Å for the C–N single bond in the 1:1 complex of 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone and nicotinoylhydrazine (Liu *et al.*, 2001). The C1–N2 and N2–N3 bond lengths are 1.362 (2) and 1.3792 (19) Å, respectively, similar to the values of 1.344 (2) and 1.388 (2) Å in *p*-(dimethylamino)benzaldehyde benzoylhydrazone (Fun *et al.*, 1997); these lie between the classical single- and double-bond lengths and indicate a partial electron delocalization over this moiety. The C=O and C–N bond lengths in the amide group are 1.2424 (12) and 1.334 (2) Å, respectively, and are normal.

The separation distance between adjacent parallel benzene rings, 3.582 Å, indicates an aromatic stacking interaction, as in

acetophenone 2,4-dinitrophenylhydrazone (Shan *et al.*, 2002). The molecules of (I) stack in the crystal structure to give chains. Intra- and intermolecular hydrogen bonds are observed (Table 1), linking the molecules in a two-dimensional network.

Experimental

An anhydrous ethanol solution of semicarbazide hydrochloride (20 ml, 0.01 mol) was added to an anhydrous ethanol solution of 3,4-methylenedioxybenzaldehyde (20 ml, 0.01 mol), and the mixture was stirred at 343–353 K for about 2 h. Concentrated hydrochloric acid (10 ml) was added, and a yellow precipitate appeared. After 3–4 h of further stirring at the same temperature, water (20 ml) was added and the colour of the solution changed to pink. The crude product was isolated when the solvent had partially evaporated. The bright pink product was collected by filtration and washed several times with cold anhydrous ethanol, then dried in a vacuum desiccator over CaCl_2 . Bright pink single crystals of (I) suitable for X-ray analysis were obtained by slowly cooling a warmed solution in dimethylsulfoxide.

Crystal data

$\text{C}_9\text{H}_9\text{N}_3\text{O}_3$
 $M_r = 207.19$
 Monoclinic, $P2_1/c$
 $a = 17.698$ (7) Å
 $b = 4.475$ (2) Å
 $c = 12.084$ (5) Å
 $\beta = 101.353$ (6)°
 $V = 938.2$ (7) Å³
 $Z = 4$

$D_x = 1.467$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 820 reflections
 $\theta = 2.0$ – 21.1 °
 $\mu = 0.11$ mm⁻¹
 $T = 293$ (2) K
 Prism, pink
 $0.26 \times 0.25 \times 0.20$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 5042 measured reflections
 1916 independent reflections

1265 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 26.4$ °
 $h = -15 \rightarrow 22$
 $k = -5 \rightarrow 5$
 $l = -15 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.101$
 $S = 1.03$
 1916 reflections
 173 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.0571P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.010 (2)

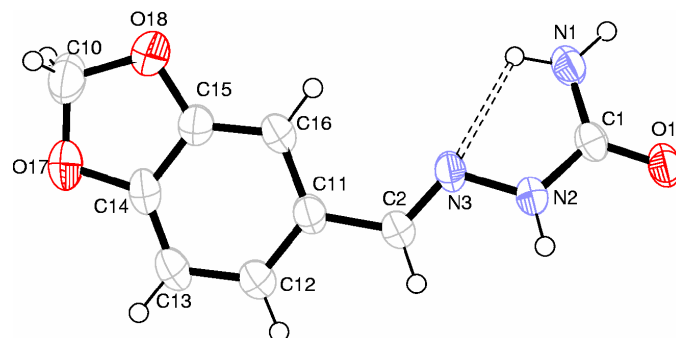


Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids. The intramolecular hydrogen bond is shown dashed.

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1B} \cdots \text{N3}$	0.90 (2)	2.27 (2)	2.666 (2)	106.2 (15)
$\text{N1}-\text{H1A} \cdots \text{O1}^{\text{i}}$	0.90 (2)	2.05 (2)	2.922 (2)	163.5 (17)
$\text{N2}-\text{H2} \cdots \text{O1}^{\text{ii}}$	0.91 (2)	2.01 (2)	2.925 (2)	175.9 (17)

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

All H atoms were located in a difference Fourier map and refined freely.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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