$C_{14}H_{28}N_2S_4$

$S = 1.06$
4662 reflections
185 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 1.6893P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

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

S1—C1	1.814 (3)	N1—C2	1.475 (3)
S1—S3	1.997 (1)	N1—C5	1.478 (3)
S2—C1	1.653 (2)	N2—C8	1.337 (3)
S3—C8	1.820 (3)	N2—C9	1.472 (3)
S4—C8	1.646 (3)	N2—C12	1.472 (3)
N1—C1	1.326 (3)		
C1—N1—C2—C3	92.0 (3)	C8—N2—C9—C10	−96.9 (3)
C1—N1—C5—C6	91.4 (3)	C8—N2—C12—C13	−90.0 (3)

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections, and was found to be negligible.

All H atoms were geometrically fixed and allowed to ride on the attached atoms; rotating group refinement was used for the methyl groups. Atoms C4 and C11 of the propyl groups show high thermal vibrations but no indications of disorder were observed.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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p-Hydroxybenzaldehyde nicotinoylhydrazone monohydrate

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Abstract

The title compound, $C_{13}H_{11}N_3O_2 \cdot H_2O$, is in the hydrazide tautomeric form and the configuration of the azomethine C=N double bond [1.275 (3) Å] is *E*. In the solid state, the molecules are stacked parallel to (110) planes and form a hydrogen-bonded column-like supramolecular structure involving two independent water molecules on twofold axes.

Comment

Arylhydrazones have been investigated extensively in recent years because of their biological activity, especially as potent inhibitors for many enzymes (Ma *et al.*, 1994; Lu *et al.*, 1996; Fun, Lu, Duan, Tian, You, Gong & Guo, 1997; Fun, Lu, Duan, Tian, You, Guo & Gong, 1997). As part of our work on the synthesis and characterization of arylhydrazone compounds, we report here the structure of p-hydroxybenzaldehyde nicotinoylhydrazone monohydrate, (I).

(I)
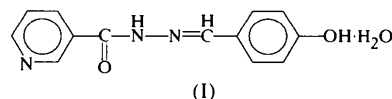
The geometry of the molecule agrees well with those found in related structures (Fun, Lu, Duan, Tian, You,

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Gong & Guo, 1997; Fun, Lu, Duan, Tian, You, Guo & Gong, 1997; Lu *et al.*, 1996). The molecule is in the hydrazide tautomeric form and the N3—C7 bond is in the *E* configuration. The planes of the pyridine and phenyl rings form a dihedral angle of 1.50 (7)° and they make dihedral angles of 7.3 (1) and 8.7 (1)°, respectively, with the central hydrazone linkage. The water of hydration is found in two different special positions, on twofold axes. These two independent water molecules have different surroundings; one has C—H· · ·O and O—H· · ·N contacts while the other has N—H· · ·O and O—H· · ·O contacts.

In the crystal, the inversion-related molecules are stacked parallel to (110) planes. The phenyl and pyridine rings, which are stacked alternately at a perpendicular distance of 3.424 (3) Å, have significant π–π interactions. The stacked molecules are linked by an N—H· · ·O, O—H· · ·N, O—H· · ·O and C—H· · ·O hydrogen-bonding network (Table 2) involving both water molecules to form an infinite column-like structure parallel to the *b* axis (Fig. 2). Since the water O
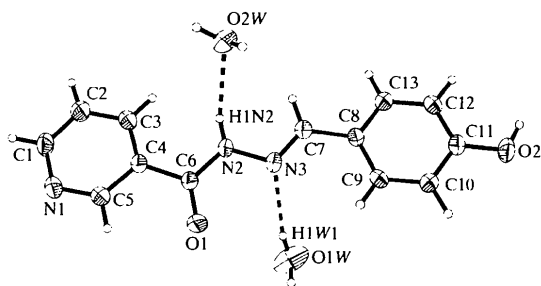

Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. O1*W* and O2*W* lie on the twofold axis and their symmetry generated H atoms are also shown.
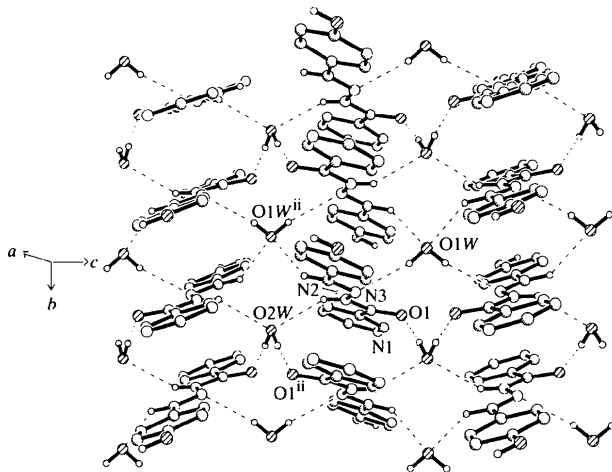

Fig. 2. A view of the hydrogen-bonding network formed by the water molecules.

atoms lie on the crystallographic twofold axis, the column is repeated to form a two-dimensional network. The water O atoms acquire full tetracoordination owing to their involvement in either N—H· · ·O or C—H· · ·O hydrogen bonding. Adjacent two-dimensional network structures are linked by O—H· · ·N hydrogen bonding involving the hydroxyl group to form a supramolecular structure.

Experimental

The synthesis of the title compound was carried out by reaction of *o*-chlorobenzaldehyde and nicotinoylhydrazine in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol. The water molecules in the crystal lattice may come from the ethanol used for the synthesis and recrystallization. The melting point of the compound is 571–573 K.

Crystal data

$C_{13}H_{11}N_3O_2 \cdot H_2O$	Mo $K\alpha$ radiation
$M_r = 259.26$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 3324
$C2/c$	reflections
$a = 27.775\,(1)$ Å	$\theta = 3.34–30.00°$
$b = 7.3270\,(3)$ Å	$\mu = 0.103$ mm^{-1}
$c = 11.9575\,(5)$ Å	$T = 293\,(2)$ K
$\beta = 91.126\,(1)°$	Plate
$V = 2432.9\,(2)$ Å^3	$0.52 \times 0.28 \times 0.04$ mm
$Z = 8$	Yellow
$D_x = 1.416$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART CCD area-detector diffractometer	2101 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.044$
Absorption correction: none	$\theta_{max} = 30°$
8816 measured reflections	$h = -38 \rightarrow 38$
3479 independent reflections	$k = 0 \rightarrow 10$
	$l = 0 \rightarrow 16$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.064$	$\Delta\rho_{max} = 0.285$ e Å^{-3}
$wR(F^2) = 0.146$	$\Delta\rho_{min} = -0.210$ e Å^{-3}
$S = 1.058$	Extinction correction:
3479 reflections	*SHELXTL* (Sheldrick,
226 parameters	1996)
All H-atom parameters refined	Extinction coefficient: 0.0014 (3)
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 3.0789P]$	Scattering factors from *International Tables for Crystallography* (Vol. C)
where $P = (F_o^2 + 2F_c^2)/3$	

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

O1—C6	1.226 (2)	N2—C6	1.342 (3)
O2—C11	1.354 (2)	N2—N3	1.389 (2)
N1—C5	1.337 (3)	N3—C7	1.275 (3)
N1—C1	1.338 (3)		

C6—N2—N3—C7	177.4 (2)	
N3—N2—C6—C4	−177.01 (18)	
C5—C4—C6—O1	5.6 (3)	
C3—C4—C6—O1	−171.7 (2)	
N2—N3—C7—C8	178.41 (19)	
N3—C7—C8—C13	176.0 (2)	
N3—C7—C8—C9	−6.4 (3)	

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1N2···O2W	0.87 (2)	2.28 (3)	3.011 (2)	142 (2)
O1W—H1W1···N3	0.85 (4)	2.25 (4)	3.088 (3)	167 (4)
O2—H1O2···N1^i	0.89 (3)	1.87 (3)	2.759 (3)	170 (3)
O2W—H1W2···O1^{ii}	0.89 (3)	1.92 (3)	2.807 (2)	173 (3)
C7—H7···O1W^{iii}	0.99 (2)	2.50 (2)	3.351 (3)	144 (2)

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

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The H atoms were refined because of their involvement in the formation of the supramolecular structure. C—H distances range from 0.94 (2) to 1.01 (2) Å and U_{iso} values for H atoms range from 0.029 (6) to 0.15 (2) Å^2.

Data collection: *SMART* (Siemens, 1996a). Cell refinement: *SAINT* (Siemens, 1996b). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Tamarixetin monohydrate

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Abstract

In the title compound, 3,5,7,3'-tetrahydroxy-4'-methoxy-flavone monohydrate [or 3,5,7-trihydroxy-2-(3-hydroxy-4-methoxyphenyl)-4H-1-benzopyran-4-one monohydrate], $C_{16}H_{12}O_7 \cdot H_2O$, there are two independent molecules in the asymmetric unit, linked through OH groups to form an O—H···O hydrogen-bonded dimer. The benzo-pyran rings are slightly distorted from planarity; in one of the molecules the phenyl ring is coplanar with the pyrone ring, while in the other it is twisted by 4.8 (1)°. The hydroxyl groups are involved in various O—H···O and C—H···O hydrogen bonds and the molecules are stacked parallel to the (220) plane.

Comment

Chromolaena odorata King and Robinson [formerly *Eupatorium odoratum* (L.)] is a perennial shrub native to Central and South America. It has, however, found its way as a troublesome weed into practically every tropical region. In some of these areas, it has acquired a reputation as a medicinal herb for a variety of ailments, including malaria, fever and infections, and as a haemostatic agent. A blood anticoagulating effect in the leaf extract was discovered by us and we speculated that this could be a consequence of the calcium-ion complexing ability of the 3-flavonols found in the extract. It was therefore an important extension of this study that the configurations of some of these

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