

3,4,5-Trimethoxybenzohydrazide hemihydrate

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Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.048
 wR factor = 0.134
Data-to-parameter ratio = 16.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

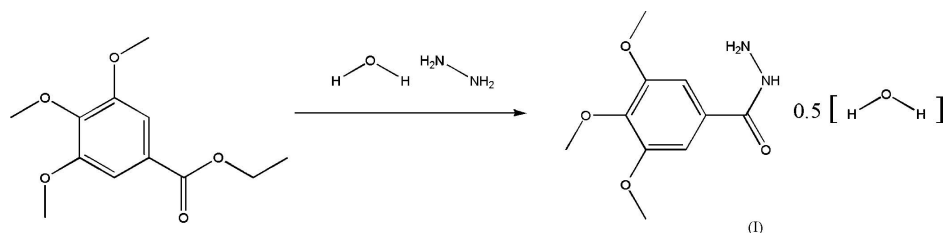
The title compound, $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_4 \cdot 0.5\text{H}_2\text{O}$, is an important intermediate for the synthesis of biologically active heterocyclic compounds. The hydrazide group is nearly coplanar with the benzene ring. The water molecule possesses crystallographically imposed C_2 symmetry. The crystal packing is stabilized by $\text{N}-\text{H} \cdots \text{O}$, $\text{C}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{N}$ hydrogen-bond interactions.

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Comment

Aromatic hydrazides are important intermediates in heterocyclic chemistry and have been used for the synthesis of various biologically active five-membered heterocycles such as 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In view of the versatility of these compounds, we have synthesized the title compound, (I), by reaction of the ethyl ester of 3,4,5-trimethoxybenzoic acid with hydrazine hydrate using a reported procedure (Furniss *et al.*, 1978). Here we report its crystal structure.



The molecular structure of the title compound is shown in Fig. 1. In (I), bond distances and angles are within expected ranges (Allen *et al.*, 1987). The hydrazide group $\text{C}10/\text{O}4/\text{N}1/\text{N}2$ is planar [maximum displacement of 0.007 (3) Å for C10] and nearly coplanar with the benzene ring, the dihedral angle they form being 9.27 (10)°. Atom O5 of the water molecule lies on a twofold rotation axis. The crystal packing is stabilized by $\text{N}-\text{H} \cdots \text{O}$, $\text{C}-\text{H} \cdots \text{O}$, $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{N}$ intra- and intermolecular hydrogen-bond interactions (Table 1), forming an extended three-dimensional network (Fig. 2).

Experimental

A mixture of ethyl 3,4,5-trimethoxybenzoate (2.26 g, 10 mmol) and hydrazine hydrate (15 ml, 80%) in absolute ethanol (50 ml) was refluxed for 5 h at 413–423 K. The excess solvent was removed by distillation. The solid residue was filtered off, washed with water, and recrystallized from ethanol (30%) to give the title compound (yield

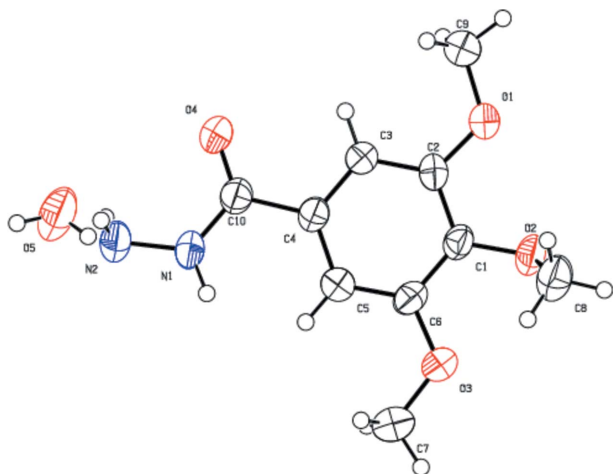


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

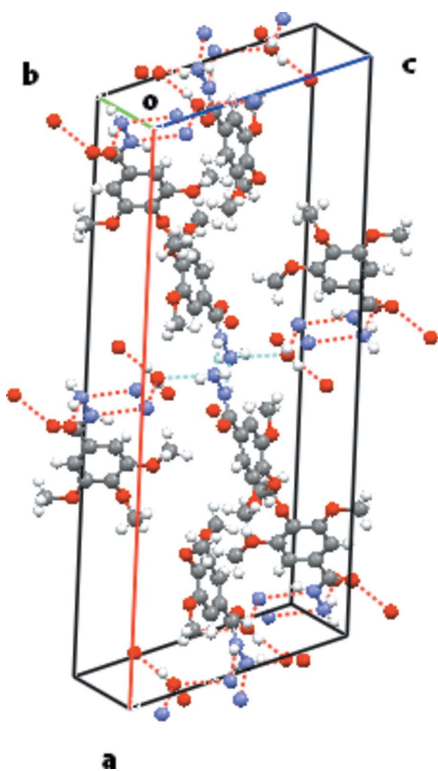


Figure 2
The crystal packing of the title compound. Hydrogen bonds are shown as dotted lines.

89%). Colorless block-like single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_{10}H_{14}N_2O_4 \cdot 0.5H_2O$ $Z = 8$
 $M_r = 235.24$ $D_x = 1.368 \text{ Mg m}^{-3}$
 Monoclinic, $C2/c$ Mo $K\alpha$ radiation
 $a = 33.253 (6) \text{ \AA}$ $\mu = 0.11 \text{ mm}^{-1}$
 $b = 4.8362 (8) \text{ \AA}$ $T = 273 (2) \text{ K}$
 $c = 14.227 (3) \text{ \AA}$ Block, colorless
 $\beta = 93.074 (16)^\circ$ $0.25 \times 0.15 \times 0.13 \text{ mm}$
 $V = 2284.7 (7) \text{ \AA}^3$

Data collection

Bruker SMART CCD area-detector 2843 independent reflections
 diffractometer 1054 reflections with $I > 2\sigma(I)$
 φ and ω scans $R_{int} = 0.062$
 Absorption correction: none $\theta_{max} = 28.3^\circ$
 12158 measured reflections

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.134$
 $S = 0.96$
 2843 reflections
 169 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0544P)^2 + 0.0583P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.20 \text{ e \AA}^{-3}$

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2A \cdots O5$	0.79 (3)	2.27 (3)	2.991 (3)	153 (3)
$C8-H8C \cdots O3$	0.96	2.57	3.080 (3)	114
$O5-H51 \cdots O4^i$	0.83 (1)	1.92 (2)	2.722 (2)	163 (1)
$N1-H1 \cdots N2^{ii}$	0.91 (3)	2.21 (3)	3.004 (4)	145.4 (5)

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

The H atoms bound to C atoms were placed in idealized positions and constrained to ride on their parent atoms, with $C-H = 0.93-0.96 \text{ \AA}$ and $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$ for aromatic or methyl H atoms, respectively. The H atoms bound to N and O atoms were located in a difference Fourier map and refined freely. The O-H and H-H distances within the water molecule were restrained to be 0.83 (1) and 1.35 (1) \AA , respectively. The best crystals that could be obtained diffracted weakly, which could account for the rather low observed:unique reflection ratio (37%).

Data collection: SMART (Bruker, 1998); 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: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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