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3-Oxo-2-(phenylhydrazono)butanoic acid

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.183
Data-to-parameter ratio = 13.8

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

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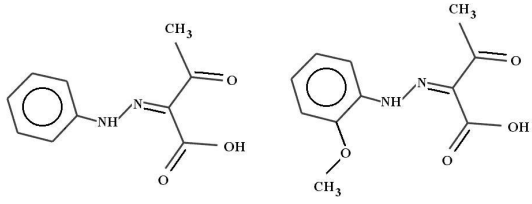
The non-hydrogen skeleton of the title molecule, $C_{10}H_{10}N_2O_3$, is planar and intramolecular N—H···O and O—H···O hydrogen bonds contribute to this planarity. The molecule exists in the space isomeric *Z* stereoisomer.

Comment

As a part of our project to study the crystal structures of a series of phenylhydrazones and their stereochemistry, the crystal structure of the title compound, (I), has been determined. Earlier we reported the structure of 2-(2-methoxyphenylhydrazono)-3-oxobutanoic acid, (II) (Rani *et al.*, 2002). The bond lengths and angles in (I) show normal values and are comparable with those observed for (II). The molecule is planar, with atom O1 deviating by a maximum of 0.066 (2) Å. The planarity of the molecule is facilitated by intramolecular hydrogen bonds. The NH group forms an intramolecular



N—H···O hydrogen bond with the carbonyl O atom of the carboxyl group. The carbonyl O atom of the carbomethoxy group is also involved in an intramolecular O—H···O hydrogen bond with the carboxyl group (Fig. 1). In the crystal, the molecules exit as centrosymmetrically hydrogen-bonded dimers [N1—H1N···O2($1-x, 1-y, 2-z$)] (Table 2). Along the *a*-cell direction, the molecules related by inversion are stacked alternately 3.412 and 3.501 Å apart; these are optimum distances for π–π stacking interactions. The short contacts observed in the structure are listed in Table 3.

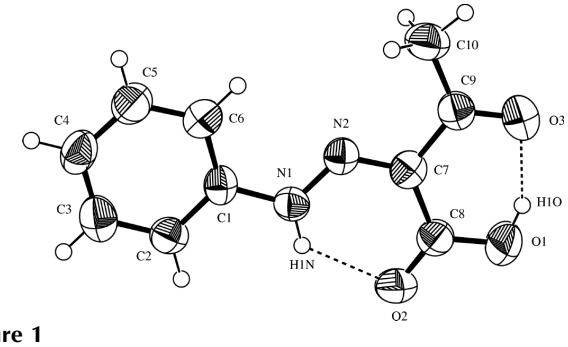


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title compound was prepared by partial hydrolysis of ethyl 3-oxo-2-(2-phenylhydrazono)butyrate by the action of strong acids (Prasad *et al.*, 1994) and was recrystallized from methanol at room temperature.

Crystal data

$C_{10}H_{10}N_2O_3$
M_r = 206.20
Monoclinic, $P2_1/n$
a = 6.9138 (10) Å
b = 21.309 (2) Å
c = 7.0783 (10) Å
β = 108.917 (10)°
V = 986.5 (2) Å^3
Z = 4
D_x = 1.388 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 16.7–49.7°
μ = 0.88 mm^{-1}
T = 293 (2) K
Needle (cut from a larger crystal), colourless
0.20 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2026 measured reflections
1878 independent reflections
1230 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 70.1°
$h = -8 \rightarrow 7$
$k = 0 \rightarrow 25$
$l = 0 \rightarrow 8$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.183
S = 1.08
1878 reflections
136 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1093P)^2 + 0.0037P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.309 (2)	N2—C7	1.311 (3)
O2—C8	1.227 (2)	C7—C9	1.464 (3)
O3—C9	1.229 (3)	C7—C8	1.475 (3)
N1—N2	1.316 (2)	C9—C10	1.475 (3)
N1—C1	1.406 (2)		
N2—N1—C1	118.36 (16)	O2—C8—O1	120.6 (2)
C7—N2—N1	122.36 (17)	O2—C8—C7	122.23 (18)
C2—C1—N1	117.77 (18)	O1—C8—C7	117.16 (19)
C6—C1—N1	121.75 (19)	O3—C9—C7	120.1 (2)
N2—C7—C9	114.78 (19)	O3—C9—C10	120.5 (2)
N2—C7—C8	124.22 (18)	C7—C9—C10	119.38 (19)
C9—C7—C8	121.00 (19)		
C1—N1—N2—C7	178.89 (16)	C9—C7—C8—O2	−176.7 (2)
N2—N1—C1—C2	177.63 (15)	N2—C7—C8—O1	−178.66 (19)
N2—N1—C1—C6	−3.9 (3)	C9—C7—C8—O1	0.5 (3)
N1—C1—C2—C3	178.86 (16)	N2—C7—C9—O3	179.73 (18)
N1—C1—C6—C5	−179.66 (19)	C8—C7—C9—O3	0.5 (3)
N1—N2—C7—C9	−177.78 (14)	N2—C7—C9—C10	−2.2 (3)
N1—N2—C7—C8	1.5 (3)	C8—C7—C9—C10	178.5 (2)
N2—C7—C8—O2	4.1 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O2	0.86	2.03	2.661 (2)	130
N1—H1N···O2^{i}	0.86	2.54	3.270 (2)	143
O1—H1O···O3	0.82	1.76	2.518 (2)	152

Symmetry code: (i) $1-x, 1-y, 2-z$.

Table 3
Contact distances (Å).

O1···C2^{i}	3.487 (3)	C3···C9^{iv}	3.423 (3)
O1···C10^{ii}	3.366 (3)	C4···C7^{iv}	3.587 (4)
O2···O2^{i}	2.907 (2)	C5···C8^{iii}	3.588 (4)
O2···C2^{i}	3.453 (3)	C5···C8^{iv}	3.390 (4)
C3···C9^{iii}	3.507 (3)		

Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $x, y, 1+z$; (iii) $-x, 1-y, 1-z$; (iv) $1-x, 1-y, 1-z$.

After location in a difference Fourier map, all the H atoms were placed at calculated positions and were allowed to ride on their respective parent atoms using *SHELXL*97 (Sheldrick, 1997) defaults.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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