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4-(Allyloxy)benzohydrazide

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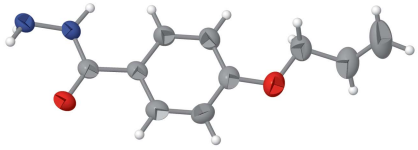
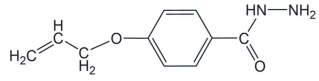
The non-H atoms of the title compound, $C_{10}H_{12}N_2O_2$, are approximately coplanar with the exception of those at the ends: the terminal allyl carbon atom and terminal hydrazide nitrogen atom are displaced from the mean plane by 0.67 (2) and 0.20 (2) Å, respectively. In the crystal, the molecules are linked by N—H···O and N—H···N hydrogen bonds, which give rise a two-dimensional network propagating in the (001) plane.

3D view



Chemical scheme



Structure description

Hydrazides containing an R—C(=O)—NH—NH_2 functional group may act as a pharmacophore and present biological activity (see, for example, Joshi *et al.* 2008). Hydrazide-containing molecules are effective ligands in coordination chemistry (see, for example, Saygıdeğer Demir *et al.*, 2021). As part of our studies in this area, we now describe the synthesis and structure of the title compound (Fig. 1).

The X-ray diffraction analysis revealed that the non-hydrogen atoms are approximately coplanar with the exception of the terminal atoms, which deviate by 0.67 (2) Å for C10 and 0.20 (2) Å for N2. In the crystal, the molecules are connected by N—H···O and N—H···N hydrogen bonds involving the carbohydrazide moieties of symmetry-related molecules (Fig. 2 and Table 1) that form a two-dimensional network propagating in the *ab* plane. This arrangement favours weak aromatic π–stacking interactions of the phenyl rings [centroid-to-centroid distance of 4.092 (3) Å, see Fig. 2].

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	1.02 (7)	2.00 (7)	3.018 (6)	174 (6)
N1—H1B···O1^{ii}	0.96 (7)	2.50 (6)	3.095 (6)	120 (4)
N2—H2···N1^{iii}	0.92 (6)	2.11 (6)	2.964 (6)	153 (4)

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

Synthesis and crystallization

A mixture of ethyl-4-hydroxybenzoate (8.3 g, 50 mmol) and allyl bromide (6.0 g, 50 mmol) in acetone (100 ml) was refluxed for 20 h over anhydrous potassium carbonate (13.8 g, 100 mmol). The filtrate was collected and the solvent removed *in vacuo*. The resulting colourless oily mass was treated with hydrazine hydrate (5.0 g, 100 mmol) and refluxed for 10 h in ethanol (40 ml). The reaction mixture was left overnight and colourless crystals suitable for X-ray characterization were obtained, filtered off and washed with ethanol. Yield: 7.0 g, (73%), melting point: 355–356 K.

FT–IR (KBr), (cm^{-1}): 1650 ν (C═O$_{ester}$), 1621, 1575 ν (C═C), 3328, 3280, 3183 ν (NH—NH$_2$).

^{1}H NMR($CDCl_3$, 400 MHz), δ: 7.72 (*d*, 2H, C-2,6, J = 8.8 Hz), 6.94 (*d*, 2H, C-3,5, J = 8.8 Hz), 7.65 (*s*, NH), 4.13 (*s*, 2H, NH$_2$), 5.42 (*dq*, H$_a$, J = 16 Hz, 1.6 Hz), 5.32 (*dq*, H$_b$, J = 10.4 Hz, 1.2 Hz), 6.05 (*m*, Hc), 4.58 (*dt*, 2H, CH_2O, J = 5.2 Hz, 1.2 Hz).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The absolute structure was indeterminate in the present refinement and the structure was refined as an inversion twin.

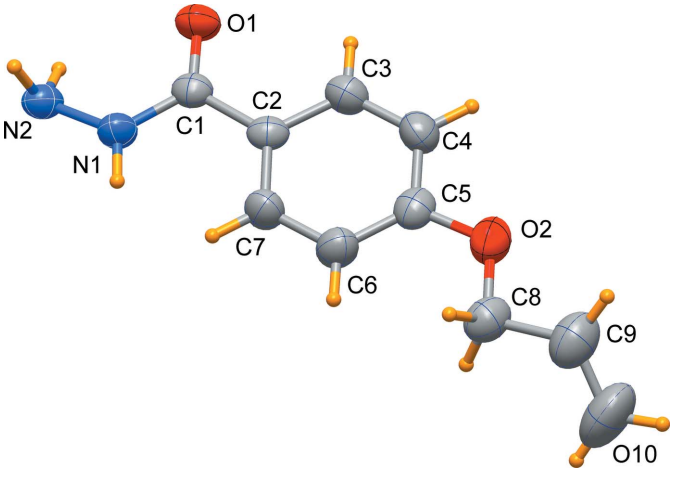


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{12}N_2O_2$
M_r	192.22
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	263
a, b, c (Å)	5.967 (4), 4.092 (3), 20.358 (14)
β (°)	93.080 (18)
V (Å^3)	496.3 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.47 × 0.21 × 0.06
Data collection	
Diffractometer	Rigaku R-AXIS RAPID CCD
Absorption correction	Multi-scan (*ABSCOR*; Rigaku, 1995)
T_{min}, T_{max}	0.299, 0.995
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4612, 2075, 1596
R_{int}	0.073
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.081, 0.228, 1.05
No. of reflections	2075
No. of parameters	139
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.30
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.5

Computer programs: *RAPID-AUTO* (Rigaku, 2010), *SHELXT2014/5* (Sheldrick, 2015*a*) and *SHELXL2019/2* (Sheldrick, 2015*b*) and *DIAMOND* (Brandenburg & Putz, 1999).

Acknowledgements

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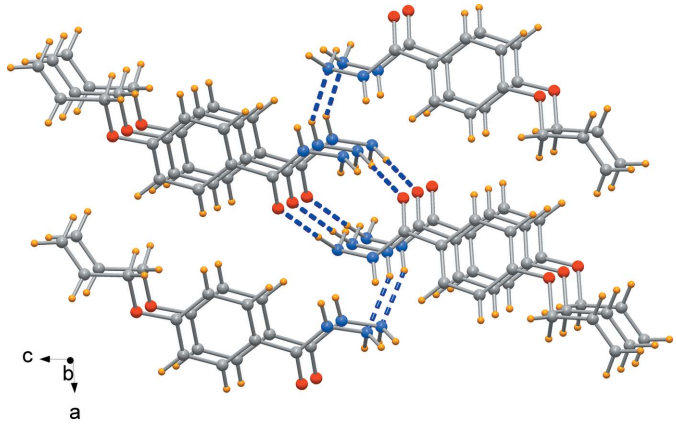

Figure 2
Detail of the crystal packing showing hydrogen-bonding interactions as blue dashed lines.

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full crystallographic data

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4-(Allyloxy)benzohydrazide

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4-(Prop-2-en-1-yloxy)benzohydrazide

Crystal data

$C_{10}H_{12}N_2O_2$
M_r = 192.22
Monoclinic, $P2_1$
a = 5.967 (4) Å
b = 4.092 (3) Å
c = 20.358 (14) Å
β = 93.080 (18)°
V = 496.3 (6) Å^3
Z = 2
$F(000)$ = 204
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 702 reflections
θ = 3.9–27.4°
μ = 0.09 mm^{-1}
T = 263 K
Plate, colorless
0.47 × 0.21 × 0.06 mm

Data collection

Rigaku R-AXIS RAPID CCD diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
T_{min} = 0.299, T_{max} = 0.995
4612 measured reflections
2075 independent reflections
1596 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −7→7
k = −5→4
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.228
S = 1.05
2075 reflections
139 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1107P)^2 + 0.1955P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.043
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.5

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a 2-component perfect inversion twin

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.0223 (5)	0.0117 (11)	0.59637 (15)	0.0501 (10)
O2	0.6099 (6)	0.6905 (12)	0.84332 (16)	0.0576 (11)
N1	0.7352 (6)	0.0258 (13)	0.48583 (18)	0.0420 (10)
H1A	0.810 (10)	0.20 (2)	0.459 (3)	0.070 (18)*
H1B	0.828 (9)	−0.162 (18)	0.495 (3)	0.053 (15)*
N2	0.6987 (6)	0.1941 (12)	0.54599 (17)	0.0422 (10)
H2	0.568 (9)	0.310 (17)	0.551 (2)	0.053 (15)*
C1	0.8432 (7)	0.1605 (13)	0.5991 (2)	0.0381 (10)
C2	0.7721 (7)	0.3101 (12)	0.6615 (2)	0.0368 (10)
C3	0.9130 (7)	0.2748 (14)	0.7179 (2)	0.0466 (13)
H3	1.048347	0.163742	0.715485	0.056*
C4	0.8538 (8)	0.4031 (15)	0.7774 (2)	0.0512 (14)
H4	0.948590	0.375286	0.814732	0.061*
C5	0.6535 (8)	0.5734 (13)	0.7817 (2)	0.0450 (12)
C6	0.5104 (9)	0.6088 (14)	0.7266 (2)	0.0499 (14)
H6	0.374650	0.718525	0.729247	0.060*
C7	0.5717 (8)	0.4782 (15)	0.6669 (2)	0.0476 (13)
H7	0.475950	0.504343	0.629677	0.057*
C8	0.4030 (9)	0.8680 (17)	0.8490 (2)	0.0558 (14)
H8A	0.275903	0.726145	0.838444	0.067*
H8B	0.396374	1.050785	0.818586	0.067*
C9	0.3954 (12)	0.9884 (19)	0.9178 (3)	0.0743 (19)
H9	0.525899	1.076453	0.937629	0.089*
C10	0.2180 (15)	0.978 (3)	0.9519 (3)	0.105 (3)
H10A	0.084985	0.891368	0.933402	0.126*
H10B	0.223805	1.057220	0.994739	0.126*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0359 (16)	0.058 (2)	0.0565 (18)	0.0103 (18)	0.0023 (12)	−0.0034 (17)
O2	0.064 (2)	0.064 (3)	0.0447 (18)	−0.003 (2)	0.0052 (14)	−0.0052 (17)
N1	0.033 (2)	0.045 (3)	0.048 (2)	0.0006 (19)	0.0049 (14)	−0.0047 (19)
N2	0.0352 (19)	0.048 (3)	0.0436 (19)	0.007 (2)	0.0032 (14)	−0.0050 (17)
C1	0.034 (2)	0.035 (2)	0.046 (2)	−0.004 (2)	0.0044 (16)	0.0039 (19)
C2	0.032 (2)	0.031 (3)	0.048 (2)	−0.0047 (19)	0.0051 (15)	0.0019 (18)
C3	0.041 (2)	0.047 (3)	0.052 (3)	0.001 (2)	−0.0016 (18)	0.002 (2)
C4	0.046 (3)	0.061 (4)	0.046 (2)	−0.005 (3)	−0.0056 (19)	0.000 (2)
C5	0.050 (3)	0.039 (3)	0.046 (2)	−0.010 (2)	0.0081 (18)	0.000 (2)
C6	0.047 (3)	0.055 (4)	0.048 (2)	0.008 (3)	0.006 (2)	0.001 (2)
C7	0.044 (3)	0.055 (4)	0.044 (2)	0.009 (3)	−0.0007 (17)	0.003 (2)
C8	0.061 (3)	0.050 (3)	0.058 (3)	−0.004 (3)	0.014 (2)	−0.007 (2)
C9	0.094 (4)	0.066 (5)	0.065 (3)	−0.006 (4)	0.020 (3)	−0.009 (3)
C10	0.131 (6)	0.117 (8)	0.072 (4)	0.017 (7)	0.046 (4)	−0.006 (5)

Geometric parameters (Å, º)

O1—C1	1.234 (6)	C4—C5	1.390 (7)
O2—C5	1.380 (6)	C4—H4	0.9300
O2—C8	1.442 (7)	C5—C6	1.381 (7)
N1—N2	1.432 (5)	C6—C7	1.395 (7)
N1—H1A	1.02 (7)	C6—H6	0.9300
N1—H1B	0.96 (7)	C7—H7	0.9300
N2—C1	1.352 (6)	C8—C9	1.488 (8)
N2—H2	0.92 (6)	C8—H8A	0.9700
C1—C2	1.493 (6)	C8—H8B	0.9700
C2—C3	1.393 (6)	C9—C10	1.297 (9)
C2—C7	1.389 (6)	C9—H9	0.9300
C3—C4	1.383 (7)	C10—H10A	0.9300
C3—H3	0.9300	C10—H10B	0.9300
C5—O2—C8	116.8 (4)	C6—C5—C4	119.8 (4)
N2—N1—H1A	102 (4)	O2—C5—C4	115.8 (4)
N2—N1—H1B	109 (3)	C5—C6—C7	119.2 (5)
H1A—N1—H1B	114 (5)	C5—C6—H6	120.4
C1—N2—N1	121.0 (4)	C7—C6—H6	120.4
C1—N2—H2	118 (3)	C2—C7—C6	121.7 (4)
N1—N2—H2	121 (3)	C2—C7—H7	119.1
O1—C1—N2	122.1 (4)	C6—C7—H7	119.1
O1—C1—C2	121.8 (4)	O2—C8—C9	108.1 (5)
N2—C1—C2	116.1 (4)	O2—C8—H8A	110.1
C3—C2—C7	118.1 (4)	C9—C8—H8A	110.1
C3—C2—C1	118.2 (4)	O2—C8—H8B	110.1
C7—C2—C1	123.7 (4)	C9—C8—H8B	110.1
C4—C3—C2	120.7 (5)	H8A—C8—H8B	108.4
C4—C3—H3	119.7	C10—C9—C8	124.0 (8)
C2—C3—H3	119.7	C10—C9—H9	118.0
C3—C4—C5	120.4 (4)	C8—C9—H9	118.0
C3—C4—H4	119.8	C9—C10—H10A	120.0
C5—C4—H4	119.8	C9—C10—H10B	120.0
C6—C5—O2	124.3 (5)	H10A—C10—H10B	120.0
N1—N2—C1—O1	7.3 (8)	C8—O2—C5—C4	180.0 (5)
N1—N2—C1—C2	−171.9 (4)	C3—C4—C5—C6	1.4 (8)
O1—C1—C2—C3	−0.8 (7)	C3—C4—C5—O2	179.8 (5)
N2—C1—C2—C3	178.5 (5)	O2—C5—C6—C7	−179.6 (5)
O1—C1—C2—C7	−179.7 (5)	C4—C5—C6—C7	−1.3 (8)
N2—C1—C2—C7	−0.5 (7)	C3—C2—C7—C6	−0.1 (8)
C7—C2—C3—C4	0.2 (7)	C1—C2—C7—C6	178.8 (5)
C1—C2—C3—C4	−178.8 (5)	C5—C6—C7—C2	0.7 (8)
C2—C3—C4—C5	−0.8 (8)	C5—O2—C8—C9	−176.7 (5)
C8—O2—C5—C6	−1.7 (8)	O2—C8—C9—C10	−137.3 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	1.02 (7)	2.00 (7)	3.018 (6)	174 (6)
N1—H1*B*···O1^{ii}	0.96 (7)	2.50 (6)	3.095 (6)	120 (4)
N2—H2···N1^{iii}	0.92 (6)	2.11 (6)	2.964 (6)	153 (4)

Symmetry codes: (i) −*x*+2, *y*+1/2, −*z*+1; (ii) −*x*+2, *y*−1/2, −*z*+1; (iii) −*x*+1, *y*+1/2, −*z*+1.