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2-(Naphthalen-2-yloxy)-*N*′-[2-(naphthalen-2-yloxy)acetyl]acetohydrazide monohydrate

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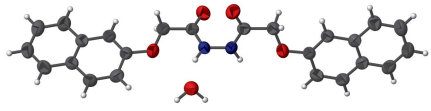
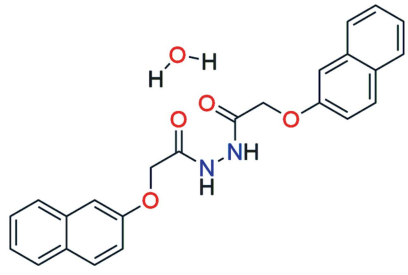
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{24}H_{20}N_2O_4\cdot H_2O$, crystallizes with half a molecule of 2-(naphthalen-2-yloxy)-*N*′-[2-(naphthalen-2-yloxy)acetyl]acetohydrazide and half a water molecule in the asymmetric unit. In the crystal, molecules form planes parallel to (011). Two molecules are connected by water molecules *via* O—H···O and N—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Diacylhydrazines can be used as environmentally friendly insecticides against lepidopteran larvae and ground-dwelling coleopterans (Morou *et al.*, 2013; Suzuki *et al.*, 2017; Wang *et al.*, 2017). In addition, they are precursors in the synthesis of electroluminescent devices (Huang *et al.*, 2009; Wu & Chen, 2009, 2010).

The asymmetric unit comprises half a molecule of 2-(naphthalen-2-yloxy)-*N*′-[2-(naphthalen-2-yloxy)acetyl]acetohydrazide and half a molecule of water, both centred on the twofold rotation axis parallel to the *c* axis. An *ORTEP* representation is shown in Fig. 1. Similar to 2-[(naphthalen-2-yl)oxy]acetamide (Huang *et al.*, 2020), the 2-[(naphthalen-2-yl)oxy]acetamidyl unit of the title compound is planar, and the twist angle between the two halves is 64.9 (1)°.

In the crystal, the molecules form planes parallel to (011) (Fig. 2). Two 2-(naphthalen-2-yloxy)-*N*′-[2-(naphthalen-2-yloxy)acetyl]acetohydrazide molecules are connected by a water molecule *via* O—H···O and N—H···O bonds (Fig. 3, Table 1). One water molecule donates O—H···O hydrogen bonds to two neighbouring molecules (related by

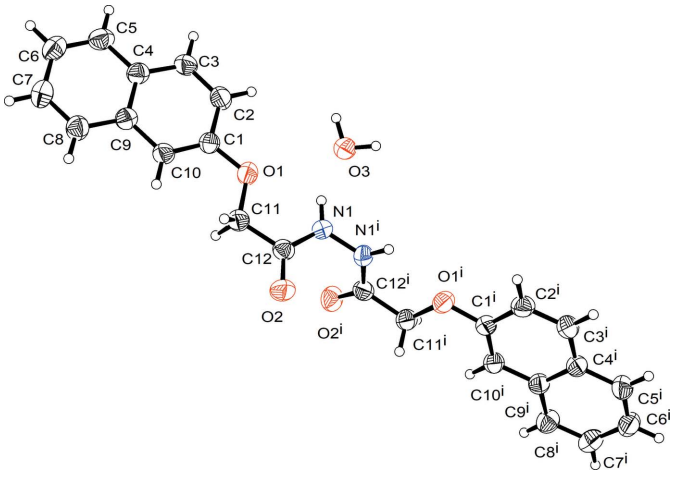


Figure 1
The molecular structure of the title compound showing 50% displacement ellipsoids.

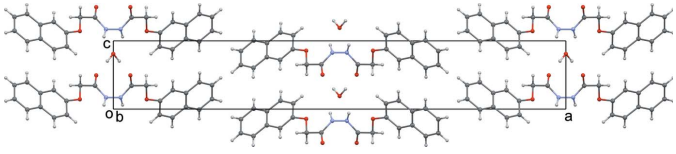


Figure 2
The crystal structure viewed down the *b* axis.

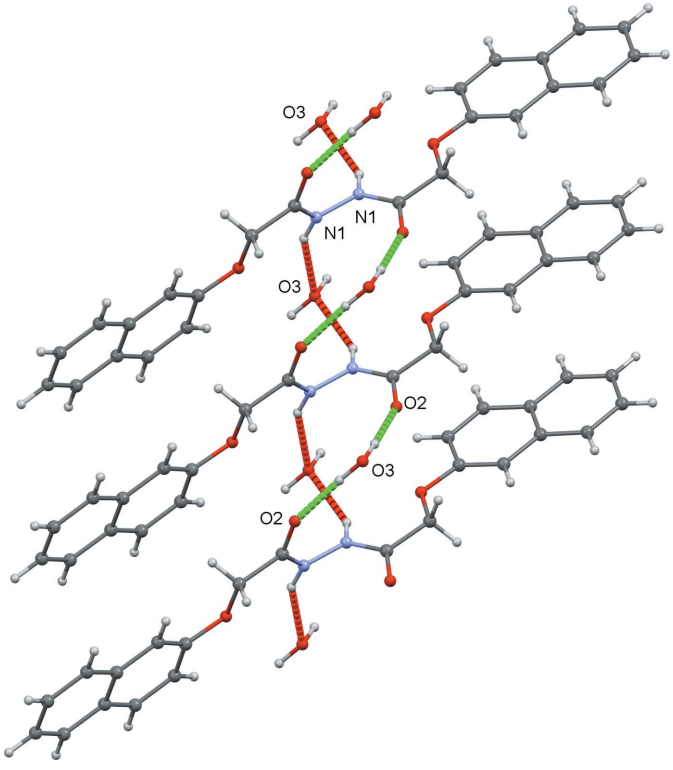


Figure 3
A segment of the crystal structure showing O—H···O hydrogen bonds as green dashed lines and N—H···O interactions as red dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	2.19	2.954 (6)	149
O3—H1*O*···O2^{i}	0.92 (6)	1.87 (6)	2.796 (5)	177 (6)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{20}N_2O_4·H_2O$
M_r	418.43
Crystal system, space group	Orthorhombic, $P2_12_12$
Temperature (K)	293
a, *b*, *c* (Å)	37.196 (4), 4.8441 (4), 5.5840 (4)
V (Å^3)	1006.12 (15)
Z	2
Radiation type	Cu *Kα*
μ (mm^{-1})	0.80
Crystal size (mm)	0.37 × 0.05 × 0.02
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.687, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7482, 2079, 1456
R_{int}	0.063
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.062, 0.172, 1.05
No. of reflections	2079
No. of parameters	145
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.19
Absolute structure	Flack *x* determined using 424 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.1 (3)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2020) and *CHEMDRAW Ultra* (Cambridge Soft, 2016).

twofold rotation), leading to the formation of a helix parallel to the *c* axis (green dashed lines in Fig. 3). The same pair of molecules is also connected by N—H···O bonds, resulting in a second parallel helical arrangement (red dashed lines in Fig. 3).

Synthesis and crystallization

A mixture of ethyl 2-cyano-3-ethoxyacrylate (0.34 g, 2.0 mmol) and 2-(naphthalen-2-yloxy)acetohydrazide (0.43 g, 2.0 mmol) in dry ethanol (10 mL) was heated with stirring under reflux for 2 h. The solid formed on cooling to room temperature. It was collected by filtration, washed with ethanol, dried and recrystallized from dimethylformamide to give colourless crystals, m.p. > 300°C (lit. m.p. > 300°C; Abdel-Wahab *et al.*, 2017) of the title compound in 76% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2021). **6**, x210314 [https://doi.org/10.1107/S2414314621003314X]

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Crystal data

$C_{24}H_{20}N_2O_4·H_2O$
M_r = 418.43
Orthorhombic, $P2_12_12$
a = 37.196 (4) Å
b = 4.8441 (4) Å
c = 5.5840 (4) Å
V = 1006.12 (15) Å^3
Z = 2
$F(000)$ = 440
D_x = 1.381 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 1274 reflections
θ = 4.7–73.7°
μ = 0.80 mm^{-1}
T = 293 K
Needle, colourless
0.37 × 0.05 × 0.02 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2015)
T_{min} = 0.687, T_{max} = 1.000
7482 measured reflections
2079 independent reflections
1456 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 76.9°, θ_{min} = 4.8°
h = −47→44
k = −6→5
l = −4→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.172
S = 1.05
2079 reflections
145 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.4016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack x determined using 424 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.1 (3)

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. The coordinates for the water hydrogen atom was refined freely. The rest of the hydrogen atoms were positioned geometrically and refined using a riding model. All U_{iso}(H) were constrained to be 1.2 times U_{eq}(C,*N*).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.39660 (13)	0.5528 (10)	0.8789 (8)	0.0464 (10)
C2	0.39748 (15)	0.7245 (12)	1.0831 (9)	0.0555 (12)
H2	0.416807	0.717218	1.188528	0.067*
C3	0.36943 (15)	0.9021 (12)	1.1238 (9)	0.0571 (13)
H3	0.370140	1.016895	1.257183	0.068*
C4	0.33940 (14)	0.9153 (10)	0.9681 (9)	0.0501 (11)
C5	0.31024 (16)	1.0968 (12)	1.0072 (11)	0.0616 (14)
H5	0.310336	1.210702	1.141216	0.074*
C6	0.28194 (15)	1.1080 (12)	0.8520 (11)	0.0635 (14)
H6	0.262865	1.227610	0.880591	0.076*
C7	0.28187 (15)	0.9368 (12)	0.6482 (12)	0.0642 (14)
H7	0.262674	0.944144	0.541710	0.077*
C8	0.30967 (15)	0.7608 (12)	0.6059 (10)	0.0595 (13)
H8	0.309150	0.649189	0.470417	0.071*
C9	0.33923 (14)	0.7443 (11)	0.7632 (9)	0.0480 (11)
C10	0.36861 (14)	0.5619 (11)	0.7222 (9)	0.0488 (11)
H10	0.368669	0.448574	0.587834	0.059*
C11	0.42712 (13)	0.2082 (10)	0.6538 (9)	0.0499 (11)
H11A	0.427237	0.319235	0.509307	0.060*
H11B	0.405620	0.094828	0.651848	0.060*
C12	0.45969 (14)	0.0248 (10)	0.6558 (8)	0.0477 (10)
O2	0.46226 (11)	−0.1567 (8)	0.5050 (8)	0.0639 (10)
O3	0.500000	0.500000	1.1908 (10)	0.0627 (14)
O1	0.42622 (10)	0.3845 (8)	0.8567 (7)	0.0549 (9)
N1	0.48388 (11)	0.0727 (9)	0.8289 (7)	0.0494 (9)
H1	0.479501	0.192285	0.938948	0.059*
H1O	0.4869 (17)	0.609 (14)	1.295 (11)	0.069 (19)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.051 (3)	0.044 (2)	0.045 (2)	0.004 (2)	0.0000 (19)	0.0022 (18)
C2	0.061 (3)	0.055 (3)	0.050 (3)	0.003 (2)	−0.009 (2)	0.000 (2)
C3	0.068 (3)	0.056 (3)	0.047 (3)	0.008 (2)	−0.004 (2)	−0.011 (2)
C4	0.057 (3)	0.043 (2)	0.050 (3)	0.004 (2)	0.004 (2)	0.002 (2)
C5	0.074 (3)	0.053 (3)	0.058 (3)	0.010 (3)	0.011 (3)	−0.003 (3)
C6	0.057 (3)	0.058 (3)	0.075 (4)	0.013 (3)	0.007 (3)	0.004 (3)

C7	0.055 (3)	0.060 (3)	0.078 (4)	0.007 (3)	−0.006 (3)	0.008 (3)
C8	0.056 (3)	0.057 (3)	0.066 (3)	0.003 (2)	−0.009 (2)	−0.006 (2)
C9	0.052 (3)	0.042 (2)	0.050 (2)	0.001 (2)	0.0030 (19)	0.0036 (19)
C10	0.056 (3)	0.044 (2)	0.046 (2)	0.004 (2)	−0.0023 (19)	−0.0042 (18)
C11	0.055 (3)	0.047 (2)	0.048 (3)	0.003 (2)	−0.003 (2)	−0.003 (2)
C12	0.055 (3)	0.045 (2)	0.042 (2)	0.001 (2)	0.001 (2)	0.0014 (19)
O2	0.068 (2)	0.060 (2)	0.063 (2)	0.0081 (19)	−0.003 (2)	−0.0190 (19)
O3	0.075 (4)	0.060 (3)	0.053 (3)	0.015 (3)	0.000	0.000
O1	0.0528 (19)	0.0537 (19)	0.058 (2)	0.0103 (15)	−0.0078 (16)	−0.0032 (17)
N1	0.050 (2)	0.054 (2)	0.045 (2)	0.0109 (18)	0.0001 (18)	−0.0065 (18)

Geometric parameters (Å, º)

C1—C10	1.361 (7)	C7—H7	0.9300
C1—O1	1.376 (6)	C8—C9	1.410 (7)
C1—C2	1.412 (7)	C8—H8	0.9300
C2—C3	1.371 (8)	C9—C10	1.424 (7)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.417 (7)	C11—O1	1.419 (6)
C3—H3	0.9300	C11—C12	1.502 (7)
C4—C9	1.413 (7)	C11—H11A	0.9700
C4—C5	1.413 (7)	C11—H11B	0.9700
C5—C6	1.364 (9)	C12—O2	1.221 (6)
C5—H5	0.9300	C12—N1	1.341 (6)
C6—C7	1.408 (9)	O3—H1O	0.92 (6)
C6—H6	0.9300	N1—N1^{i}	1.391 (8)
C7—C8	1.361 (8)	N1—H1	0.8600
C10—C1—O1	125.0 (4)	C7—C8—H8	119.3
C10—C1—C2	121.2 (5)	C9—C8—H8	119.3
O1—C1—C2	113.8 (4)	C8—C9—C4	118.4 (5)
C3—C2—C1	119.1 (5)	C8—C9—C10	122.3 (5)
C3—C2—H2	120.5	C4—C9—C10	119.4 (5)
C1—C2—H2	120.5	C1—C10—C9	120.3 (5)
C2—C3—C4	121.8 (5)	C1—C10—H10	119.9
C2—C3—H3	119.1	C9—C10—H10	119.9
C4—C3—H3	119.1	O1—C11—C12	111.7 (4)
C9—C4—C5	119.1 (5)	O1—C11—H11A	109.3
C9—C4—C3	118.3 (5)	C12—C11—H11A	109.3
C5—C4—C3	122.6 (5)	O1—C11—H11B	109.3
C6—C5—C4	121.3 (5)	C12—C11—H11B	109.3
C6—C5—H5	119.4	H11A—C11—H11B	107.9
C4—C5—H5	119.4	O2—C12—N1	124.7 (5)
C5—C6—C7	119.4 (5)	O2—C12—C11	118.9 (5)
C5—C6—H6	120.3	N1—C12—C11	116.4 (4)
C7—C6—H6	120.3	C1—O1—C11	116.6 (4)
C8—C7—C6	120.5 (5)	C12—N1—N1^{i}	119.4 (4)
C8—C7—H7	119.7	C12—N1—H1	120.3

C6—C7—H7	119.7	N1^{i}—N1—H1	120.3
C7—C8—C9	121.3 (5)		

Symmetry code: (i) −*x*+1, −*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	2.19	2.954 (6)	149
O3—H1*O*···O2^{ii}	0.92 (6)	1.87 (6)	2.796 (5)	177 (6)

Symmetry code: (ii) *x*, *y*+1, *z*+1.