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(3*E*)-4-[(Naphthalen-1-yl)amino]pent-3-en-2-one hemihydrate

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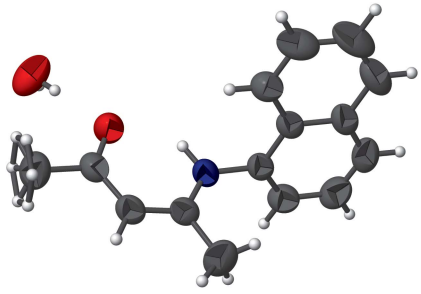
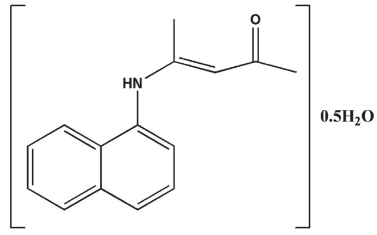
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{15}NO\cdot0.5H_2O$, was prepared from α-naphthylamine and 2,4-pentanedione in a 1:1 ratio. An intramolecular N—H···O hydrogen bond in the *N*-naphthylpent-3-en-2-one molecule involving the amine and carbonyl groups strengthens the structure. The water molecule interacts with two symmetry-related *N*-naphthylpent-3-en-2-one molecules *via*by O—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Enaminones, which consist of an amino group linked by a carbon–carbon double bond to a carbonyl group, is an area of considerable opportunity (Montanile, 2003). In fact, enaminones are used in the synthesis of different heterocycles and biologically active analogues and also in the development of pharmaceuticals because of their role as organic intermediates (Esmaiel *et al.*, 2018). It should be noted that the biological activity of enaminone compounds is attributed to the presence of the active N—C═C—C═O group within a ring system (Kale, 2016). Much work has been undertaken to explore new routes for the synthesis of enaminones. Azzoro *et al.* (1981) reported a simple method using amines and 1,3-diketones. Eddington *et al.* (2000) reported on the synthesis and anticonvulsant evaluations of some enaminones. In another method, β-chloro vinyl ketone was reacted with amines to synthesize these compounds (Pohland & Benson, 1966). Other methods of preparation include reactions between formamide dimethyl acetate and ketones (Abdulla & Brinkmeyer, 1979), acid chlorides with terminal alkynes and triethylamine (Karpov & Müller, 2003), primary amines and β-dicarbonyl compounds catalysed by copper nanoparticles (Kidwai *et al.*, 2009). Lue & Greenhill

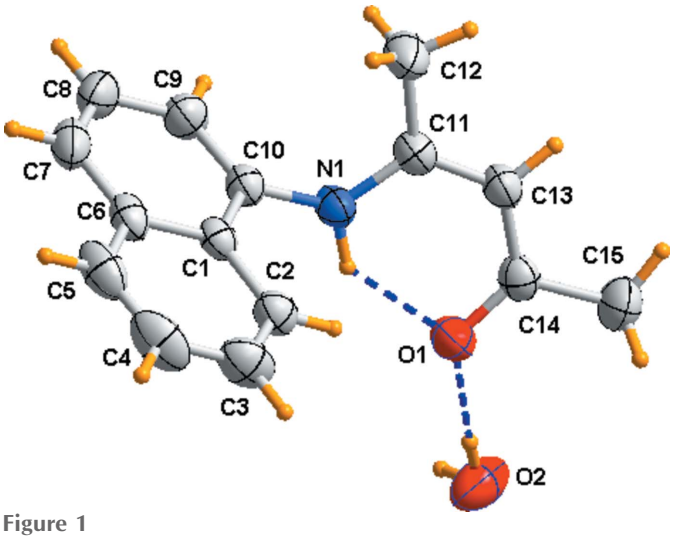


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 35% probability level. Hydrogen bonds are shown as light-blue dashed lines.

(1996) functionalized enaminones by introducing different substituents on the nitrogen, α- and β-carbon atoms to the carbonyl group. These derivatives are used extensively for preserving natural products and analogues. Enaminones are also considered to be good chelating ligands for transition metals in coordination chemistry (Esmaiel *et al.*, 2018). The anions produced from enaminone offer potential isoelectronic alternatives to cyclopentadienyl-based anions and therefore their transition-metal complexes can act as possible alternative catalysts for olefinic polymerization (Pešková *et al.*, 2006). Imada *et al.* (1996) transformed β-amino ketones to enaminones using palladium while Tan *et al.* (2008) reported enaminone applications of rhodium compounds containing bidentate ligand systems.

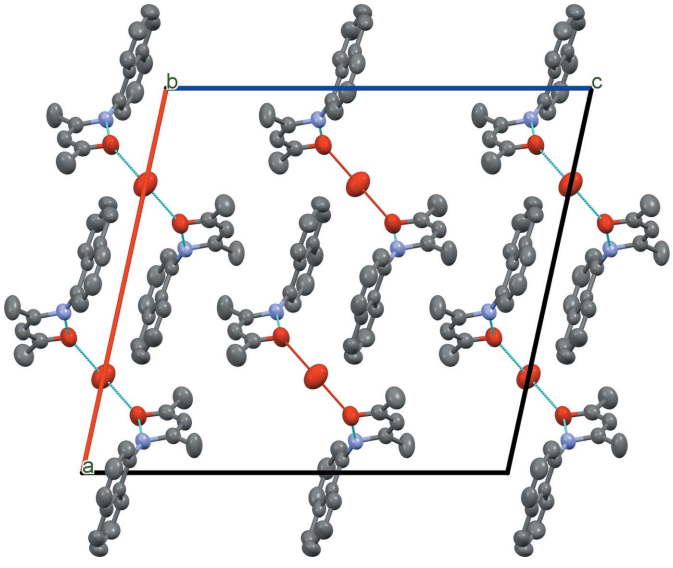


Figure 2
Crystal packing of the title compound viewed down the *b* axis. O—H···O hydrogen bonds are shown as light-green dashed lines. H atoms are omitted.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.905 (17)	1.933 (17)	2.6765 (17)	138.2 (15)
O2—H2A···O1	0.85 (3)	2.02 (3)	2.8678 (15)	176 (3)

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{15}NO{\cdot}0.5H_2O$
M_r	234.29
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	295
a, *b*, *c* (Å)	17.1405 (8), 8.3052 (4), 18.5156 (8)
β (°)	102.347 (4)
V (Å^3)	2574.8 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.88 × 0.48 × 0.27
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Ruby, Gemini Ultra
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.952, 0.981
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6527, 2637, 2045
R_{int}	0.013
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.123, 1.05
No. of reflections	2637
No. of parameters	168
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.13, −0.16

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*a*) and *SHELXL2018/3* (Sheldrick, 2015*b*).

In the title compound (Fig. 1), the dihedral angle formed by the mean planes through the naphthalene ring system and the aminopentanone group is 69.66 (9)°. This angle is determined by crystal-packing requirements. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond. The water molecule, located on a crystallographic twofold axis, is linked to two symmetry-related *N*-naphthylpent-3-en-2-one molecules *via* O—H···O hydrogen bonds (Fig. 2 and Table 1). In addition, In addition, there are π–π interactions [centroid-to-centroid distance of 3.7975 (10) Å] between the naphthalene ring systems of symmetry-related molecules, generating chains of molecules running in the [100] direction.

Synthesis and crystallization

A mixture of α-naphthylamine (0,143 g; 1 mmol) and 2,4-pentanedione (0,100 g; 1 mmol) in ethanol solvent was stirred for about 4 h and then filtered. Slow evaporation of the solution at room temperature was carried out, leading to grey

crystals suitable for a single-crystal X-ray diffraction study (yield 63%).

Refinement

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

Acknowledgements

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References

Abdulla, R. & Brinkmeyer, R. S. (1979). *Tetrahedron*, **35**, 1675–1735.
Azzoro, M., Geribaldi, S. & Videau, B. (1981). *Organic Chemistry Portable*, **11**, 880–881.
Eddington, N., Cox, D., Roberts, R., Stables, J., Powell, C. & Scott, K. (2000). *Curr. Med. Chem.* **7**, 417–436.
Esmaiel, E., Mohamad, Z. & Peter, T. C. (2018). ACS *Appl. Mater. Interfaces*, 44, 1–13.
Imada, Y., Mitsue, Y., Ike, K., Washizuka, K. & Murahashi, S. (1996). *Bull. Chem. Soc. Jpn*, **69**, 2079–2090.
Kale, A. A. (2016). Int. J. Sci. Eng. Res. **7**,873–880.
Karpov, A. & Müller, T. (2003). *Organic Chemistry Portal*, **18**, 2815–2826.
Kidwai, M., Bhardwaj, S., Mishra, N., Bansal, V., Kumar, A. & Mozumdar, S. (2009). *Catal. Commun.* **10**, 1514–1517.
Lue, P. & Greenhill, J. (1996). *Adv. Heterocycl. Chem.* **67**, 207–343.
Montanile, C. (2003). *J. Braz. Chem. Soc.* **14**(6), 945–969.
Pešková, M., Šimůnek, P., Bertolasi, V., Macháček, V. & Lyčka, A. (2006). *Organometallics*, **25**, 2025–2030.
Pohland, A. & Benson, W. (1966). *Chem. Rev.* **66**, 161–197.
Rigaku OD (2018). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Tan, H. Y., Loke, W. K., Tan, Y. T. & Nguyen, N. T. (2008). *Lab Chip*, **8**, 885–891.

full crystallographic data

IUCrData (2021). **6**, x210989 [https://doi.org/10.1107/S2414314621009895]

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Crystal data

$C_{15}H_{15}NO{\cdot}0.5H_2O$
M_r = 234.29
Monoclinic, $I2/a$
a = 17.1405 (8) Å
b = 8.3052 (4) Å
c = 18.5156 (8) Å
β = 102.347 (4)°
V = 2574.8 (2) Å^3
Z = 8
$F(000)$ = 1000
D_x = 1.209 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2470 reflections
θ = 2.9–30.5°
μ = 0.08 mm^{-1}
T = 295 K
Block, colourless
0.88 × 0.48 × 0.27 mm

Data collection

Oxford Diffraction Xcalibur, Ruby, Gemini Ultra diffractometer
Graphite monochromator
Detector resolution: 10.3712 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlisPro; Rigaku OD, 2018)
T_{min} = 0.952, T_{max} = 0.981
6527 measured reflections
2637 independent reflections
2045 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 26.4°, θ_{min} = 2.3°
h = −21→13
k = −10→10
l = −21→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.05
2637 reflections
168 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: dual
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.6014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.35246 (7)	0.84680 (13)	0.60203 (6)	0.0697 (3)	
N1	0.41810 (7)	0.55405 (15)	0.62641 (6)	0.0554 (3)	
H1	0.4037 (10)	0.641 (2)	0.5971 (9)	0.067*	
C1	0.54448 (8)	0.47503 (16)	0.59592 (7)	0.0487 (3)	
C2	0.57433 (9)	0.63404 (19)	0.60622 (8)	0.0613 (4)	
H2	0.542714	0.714959	0.619655	0.074*	
C3	0.64878 (11)	0.6695 (3)	0.59665 (10)	0.0820 (5)	
H3	0.667331	0.774862	0.602925	0.098*	
C4	0.69744 (11)	0.5507 (3)	0.57766 (10)	0.0914 (6)	
H4	0.748515	0.576743	0.571849	0.110*	
C5	0.67113 (10)	0.3970 (3)	0.56749 (9)	0.0787 (5)	
H5	0.704585	0.318524	0.554932	0.094*	
C6	0.59393 (9)	0.35372 (18)	0.57560 (7)	0.0581 (4)	
C7	0.56410 (11)	0.1952 (2)	0.56424 (9)	0.0716 (5)	
H7	0.596165	0.114794	0.551045	0.086*	
C8	0.48942 (12)	0.15854 (19)	0.57231 (10)	0.0747 (5)	
H8	0.470690	0.053490	0.564663	0.090*	
C9	0.44020 (10)	0.27831 (19)	0.59215 (8)	0.0654 (4)	
H9	0.388734	0.252280	0.596803	0.079*	
C10	0.46696 (8)	0.43196 (16)	0.60462 (7)	0.0511 (3)	
C11	0.40408 (8)	0.57179 (17)	0.69445 (7)	0.0545 (3)	
C12	0.42920 (14)	0.4398 (2)	0.74914 (10)	0.0870 (6)	
H12A	0.395859	0.347308	0.734921	0.131*	
H12B	0.424079	0.475343	0.797244	0.131*	
H12C	0.483825	0.411805	0.750444	0.131*	
C13	0.36721 (9)	0.70740 (18)	0.71367 (8)	0.0583 (4)	
H13	0.357332	0.711308	0.761114	0.070*	
C14	0.34324 (8)	0.84062 (17)	0.66740 (8)	0.0566 (4)	
C15	0.30550 (13)	0.9813 (2)	0.69784 (11)	0.0864 (5)	
H15A	0.297561	1.067498	0.662469	0.130*	0.5
H15B	0.339877	1.017109	0.742911	0.130*	0.5
H15C	0.254936	0.949261	0.707604	0.130*	0.5
H15D	0.297355	0.955081	0.746187	0.130*	0.5
H15E	0.255039	1.005469	0.665745	0.130*	0.5
H15F	0.339980	1.073318	0.701052	0.130*	0.5
O2	0.250000	1.0550 (2)	0.500000	0.1027 (7)	
H2A	0.2805 (17)	0.990 (3)	0.5285 (16)	0.151 (11)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0799 (8)	0.0721 (7)	0.0613 (7)	0.0141 (5)	0.0242 (5)	0.0081 (5)
N1	0.0566 (7)	0.0623 (7)	0.0506 (7)	0.0104 (5)	0.0184 (5)	0.0042 (5)
C1	0.0496 (7)	0.0604 (8)	0.0356 (6)	0.0035 (6)	0.0080 (5)	−0.0002 (6)
C2	0.0641 (9)	0.0675 (9)	0.0528 (8)	−0.0044 (7)	0.0141 (7)	−0.0035 (7)

C3	0.0744 (12)	0.0982 (13)	0.0744 (11)	−0.0283 (10)	0.0180 (9)	−0.0020 (9)
C4	0.0535 (10)	0.148 (2)	0.0751 (12)	−0.0135 (11)	0.0187 (8)	−0.0011 (12)
C5	0.0573 (10)	0.1225 (16)	0.0582 (9)	0.0216 (10)	0.0164 (7)	0.0010 (10)
C6	0.0596 (8)	0.0751 (10)	0.0395 (7)	0.0158 (7)	0.0108 (6)	0.0001 (6)
C7	0.0924 (13)	0.0661 (10)	0.0562 (9)	0.0226 (9)	0.0157 (8)	−0.0059 (7)
C8	0.1019 (14)	0.0539 (9)	0.0677 (10)	−0.0024 (8)	0.0170 (9)	−0.0089 (7)
C9	0.0687 (10)	0.0653 (9)	0.0634 (9)	−0.0095 (7)	0.0166 (7)	−0.0046 (7)
C10	0.0530 (8)	0.0566 (8)	0.0443 (7)	0.0043 (6)	0.0121 (6)	−0.0005 (6)
C11	0.0550 (8)	0.0613 (8)	0.0484 (7)	−0.0003 (6)	0.0137 (6)	−0.0007 (6)
C12	0.1277 (16)	0.0789 (11)	0.0574 (10)	0.0258 (11)	0.0262 (10)	0.0092 (9)
C13	0.0631 (9)	0.0657 (9)	0.0493 (8)	0.0026 (7)	0.0195 (6)	−0.0032 (7)
C14	0.0510 (8)	0.0618 (8)	0.0584 (9)	−0.0019 (6)	0.0151 (6)	−0.0055 (7)
C15	0.1069 (15)	0.0715 (10)	0.0870 (12)	0.0205 (10)	0.0350 (11)	−0.0041 (9)
O2	0.1283 (19)	0.0622 (11)	0.1037 (16)	0.000	−0.0062 (13)	0.000

Geometric parameters (Å, º)

O1—C14	1.2548 (17)	C8—H8	0.9300
N1—C11	1.3403 (17)	C9—C10	1.359 (2)
N1—C10	1.4271 (17)	C9—H9	0.9300
N1—H1	0.904 (17)	C11—C13	1.375 (2)
C1—C2	1.414 (2)	C11—C12	1.492 (2)
C1—C10	1.4180 (18)	C12—H12A	0.9600
C1—C6	1.4183 (18)	C12—H12B	0.9600
C2—C3	1.358 (2)	C12—H12C	0.9600
C2—H2	0.9300	C13—C14	1.406 (2)
C3—C4	1.385 (3)	C13—H13	0.9300
C3—H3	0.9300	C14—C15	1.503 (2)
C4—C5	1.354 (3)	C15—H15A	0.9600
C4—H4	0.9300	C15—H15B	0.9600
C5—C6	1.410 (2)	C15—H15C	0.9600
C5—H5	0.9300	C15—H15D	0.9600
C6—C7	1.412 (2)	C15—H15E	0.9600
C7—C8	1.355 (2)	C15—H15F	0.9600
C7—H7	0.9300	O2—H2A	0.85 (3)
C8—C9	1.403 (2)	O2—H2A^{i}	0.85 (3)
C11—N1—C10	125.30 (12)	C13—C11—C12	120.54 (13)
C11—N1—H1	113.3 (10)	C11—C12—H12A	109.5
C10—N1—H1	119.8 (10)	C11—C12—H12B	109.5
C2—C1—C10	122.75 (12)	H12A—C12—H12B	109.5
C2—C1—C6	118.65 (13)	C11—C12—H12C	109.5
C10—C1—C6	118.60 (13)	H12A—C12—H12C	109.5
C3—C2—C1	120.52 (15)	H12B—C12—H12C	109.5
C3—C2—H2	119.7	C11—C13—C14	125.27 (13)
C1—C2—H2	119.7	C11—C13—H13	117.4
C2—C3—C4	120.85 (18)	C14—C13—H13	117.4
C2—C3—H3	119.6	O1—C14—C13	122.64 (13)

C4—C3—H3	119.6	O1—C14—C15	118.90 (14)
C5—C4—C3	120.41 (16)	C13—C14—C15	118.46 (13)
C5—C4—H4	119.8	C14—C15—H15A	109.5
C3—C4—H4	119.8	C14—C15—H15B	109.5
C4—C5—C6	121.21 (17)	H15A—C15—H15B	109.5
C4—C5—H5	119.4	C14—C15—H15C	109.5
C6—C5—H5	119.4	H15A—C15—H15C	109.5
C5—C6—C7	122.70 (15)	H15B—C15—H15C	109.5
C5—C6—C1	118.35 (15)	C14—C15—H15D	109.5
C7—C6—C1	118.95 (14)	H15A—C15—H15D	141.1
C8—C7—C6	120.91 (14)	H15B—C15—H15D	56.3
C8—C7—H7	119.5	H15C—C15—H15D	56.3
C6—C7—H7	119.5	C14—C15—H15E	109.5
C7—C8—C9	120.32 (15)	H15A—C15—H15E	56.3
C7—C8—H8	119.8	H15B—C15—H15E	141.1
C9—C8—H8	119.8	H15C—C15—H15E	56.3
C10—C9—C8	120.68 (15)	H15D—C15—H15E	109.5
C10—C9—H9	119.7	C14—C15—H15F	109.5
C8—C9—H9	119.7	H15A—C15—H15F	56.3
C9—C10—C1	120.52 (13)	H15B—C15—H15F	56.3
C9—C10—N1	121.20 (13)	H15C—C15—H15F	141.1
C1—C10—N1	118.27 (12)	H15D—C15—H15F	109.5
N1—C11—C13	121.20 (13)	H15E—C15—H15F	109.5
N1—C11—C12	118.26 (13)	H2A—O2—H2A^{i}	101 (4)

C10—C1—C2—C3	−179.47 (14)	C8—C9—C10—C1	1.7 (2)
C6—C1—C2—C3	0.1 (2)	C8—C9—C10—N1	−178.61 (14)
C1—C2—C3—C4	−0.9 (3)	C2—C1—C10—C9	178.20 (13)
C2—C3—C4—C5	0.7 (3)	C6—C1—C10—C9	−1.40 (19)
C3—C4—C5—C6	0.2 (3)	C2—C1—C10—N1	−1.53 (19)
C4—C5—C6—C7	178.95 (16)	C6—C1—C10—N1	178.86 (11)
C4—C5—C6—C1	−1.0 (2)	C11—N1—C10—C9	76.91 (19)
C2—C1—C6—C5	0.79 (19)	C11—N1—C10—C1	−103.36 (16)
C10—C1—C6—C5	−179.59 (12)	C10—N1—C11—C13	169.20 (13)
C2—C1—C6—C7	−179.15 (13)	C10—N1—C11—C12	−11.1 (2)
C10—C1—C6—C7	0.47 (19)	N1—C11—C13—C14	−2.3 (2)
C5—C6—C7—C8	−179.74 (15)	C12—C11—C13—C14	178.05 (16)
C1—C6—C7—C8	0.2 (2)	C11—C13—C14—O1	1.2 (2)
C6—C7—C8—C9	0.0 (3)	C11—C13—C14—C15	−178.45 (16)
C7—C8—C9—C10	−1.0 (3)		

Symmetry code: (i) −*x*+1/2, *y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.905 (17)	1.933 (17)	2.6765 (17)	138.2 (15)
O2—H2*A*···O1	0.85 (3)	2.02 (3)	2.8678 (15)	176 (3)