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N-(4-Aminophenyl)-1,8-naphthalimide hemihydrate

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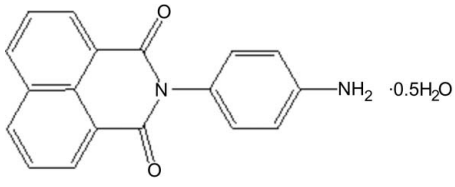
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.008 Å; disorder in solvent or counterion; R factor = 0.069; wR factor = 0.164; data-to-parameter ratio = 6.7.

The title compound, $C_{18}H_{12}N_2O_2\cdot0.5H_2O$, was prepared by the reaction of 1,4-phenylenediamine with 1,8-naphthalic anhydride in refluxing dimethylformamide. The structure is stabilized by N—H···O and O—H···O hydrogen bonds. There are π–π stacking interactions [centroid-centroid distances of 3.718 (2), 3.510 (2) and 3.546 (2) Å] and C—H···π interactions between the molecules. The water molecule lies on a twofold rotation axis. Its two H atoms are disordered equally over two positions.

Related literature

For related literature, see: Ofir (2006); Cederfur *et al.* (2003); Lavin & Shimizu (2006).



Experimental

Crystal data

$C_{18}H_{12}N_2O_2\cdot0.5H_2O$
M_r = 297.31
Orthorhombic, *Aba*2
a = 22.926 (5) Å
b = 17.930 (4) Å
c = 6.836 (1) Å
V = 2810 (1) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
0.40 × 0.40 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
5656 measured reflections
1359 independent reflections
1308 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.164
S = 1.33
1359 reflections
204 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

*Cg*1 is the centroid of the benzene ring C13–C18.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1W^{i}	0.86	2.19	3.046 (6)	172
N2—H2B···O1^{ii}	0.86	2.24	3.099 (5)	178
O1W—H1W1···O1^{iii}	0.85	2.30	2.800 (5)	118
O1W—H2W1···O1^{iv}	0.85	2.12	2.800 (5)	136
C15—H15A···$Cg1^{v}$	0.93	2.96	3.717	140

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2108).

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N-(4-Aminophenyl)-1,8-naphthalimide hemihydrate

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S1. Comment

Recently combinatorial and high throughput stategies have emerged as efficient methods to prepare large numbers of potential receptors (Cederfur *et al.*, 2003). Since the naphthyl group has the potential to be a good receptor (Lavin & Shimizu, 2006), we have studied 1,8-naphthalimide derivatives, such as the pure compound (I) described here.

In the title compound (Fig. 1), the bond lengths and angles in the 1,8-naphthalenedicarboximide group and benzene ring are normal (Ofir, 2006), including the two C?O bond lengths (Table 1). The dihedral angle formed by the benzene ring (C13—C18) and naphthalic ring (N1/C1—C12) is 72.5 (2)°.

The crystal packing is realised by N2—H2A···O1w, N2—H2B···O1, O1W—H11W···O1 and C7- H7A···O2 hydrogen bonds (Table 2 and Fig 2). C—H···π interactions exist between C15—H15A and *Cg*(1)^{i}, the centroid of the benzene ring C13—C18 [symmetry code: (i) –*X*,1/2-Y,1/2+*Z*]. There are π-stacking interactions between the naphthylamide groups, with distances between ring centroids of 3.718 (2), 3.510 (2) and 3.546 (2) Å, for *Cg*(2)···*Cg*(3)^{ii}, *Cg*(3)···*Cg*(2)^{iii} and *Cg*(2)···*Cg*(4)^{ii}, respectively [*Cg*(2) = C2—C6/C11, *Cg*(3) = C1—C2/N1/C10—C12, *Cg*(4)=C6—C11; symmetry codes: (ii) 1/2-*X*,Y,-1/2 + *z*; (iii) 1/2-*X*,Y,1/2 + *z*].

S2. Experimental

The title compound was obtained by reaction of 1,4-phenylenediamine (0.1 mol) with 1,8-naphthalic anhydride (0.1 mol) in refluxing DMF (50 ml) for 4 h. Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from a DMF solution at room temperature.

S3. Refinement

The two hydrogen atoms in the water molecule were found to be disordered, each with 50% site occupancies. H atoms were fixed geometrically and allowed to ride on their attached atoms, with N—H=0.86, O—H=0.85, C—H=0.93 Å, and with U_{iso}=1.2U_{eq}. In the absence of significant anomalous scattering effects Friedel pairs have been merged.

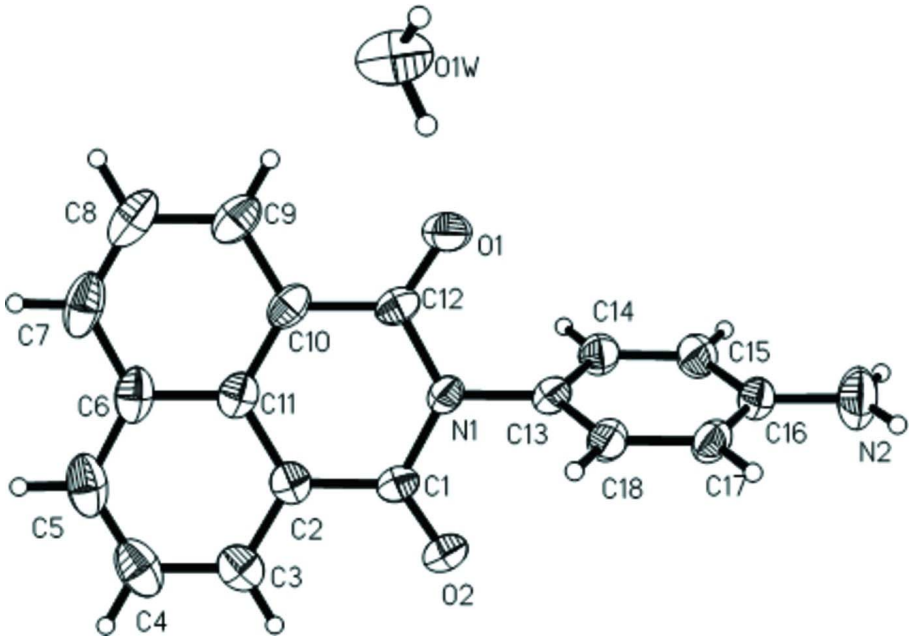

Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level. Only one of the disorder positions is shown for the water H atoms.

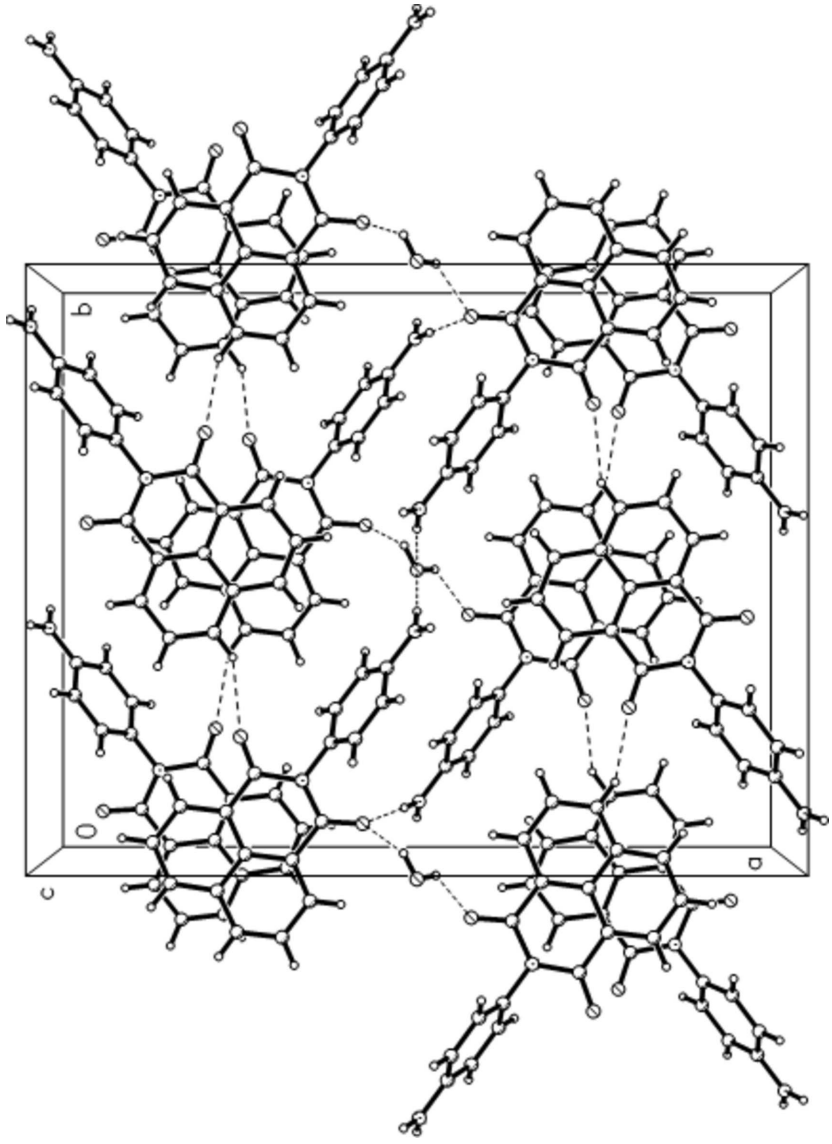

Figure 2

The packing of (I), viewed down the *c* axis, showing one layer of molecules connected by N—H···O and O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

N-(4-Aminophenyl)-1,8-naphthalimide hemihydrate

Crystal data

$C_{18}H_{12}N_2O_2{\cdot}0.5H_2O$
$M_r = 297.31$
Orthorhombic, *Aba*2
Hall symbol: A2 -2ac
$a = 22.926\ (5)$ Å
$b = 17.930\ (4)$ Å
$c = 6.836\ (1)$ Å
$V = 2810\ (1)$ Å^3
$Z = 8$
$F(000) = 1240$
$D_x = 1.406$ Mg m^{-3}
Melting point: 286 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 512 reflections
$\theta = 2$–22°
$\mu = 0.10$ mm^{-1}
$T = 295$ K
Block, yellow
0.40 × 0.40 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
5656 measured reflections
1359 independent reflections
1308 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -27 \rightarrow 23$
$k = -20 \rightarrow 21$
$l = -8 \rightarrow 7$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.164$
$S = 1.33$
1359 reflections
204 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 2.2916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.42928 (16)	0.07821 (19)	0.8275 (7)	0.0704 (12)	
O2	0.27279 (15)	0.22571 (18)	0.9326 (8)	0.0675 (12)	
N1	0.35109 (17)	0.15181 (19)	0.8761 (6)	0.0447 (10)	
N2	0.4972 (2)	0.3999 (3)	0.9511 (11)	0.095 (2)	
H2A	0.5002	0.4314	0.8566	0.115*	
H2B	0.5170	0.4064	1.0567	0.115*	
C1	0.2903 (2)	0.1646 (3)	0.8953 (8)	0.0502 (13)	
C2	0.2527 (2)	0.0997 (2)	0.8651 (7)	0.0481 (12)	
C3	0.1932 (2)	0.1090 (3)	0.8646 (8)	0.0609 (15)	
H3A	0.1772	0.1563	0.8800	0.073*	
C4	0.1569 (3)	0.0475 (4)	0.8411 (10)	0.080 (2)	
H4A	0.1167	0.0543	0.8394	0.096*	
C5	0.1787 (3)	−0.0214 (4)	0.8208 (10)	0.080 (2)	
H5A	0.1533	−0.0616	0.8090	0.096*	
C6	0.2396 (3)	−0.0341 (3)	0.8168 (9)	0.0641 (16)	
C7	0.2649 (4)	−0.1041 (3)	0.7944 (10)	0.082 (2)	

H7A	0.2409	−0.1457	0.7839	0.099*	
C8	0.3234 (4)	−0.1134 (3)	0.7875 (11)	0.083 (2)	
H8A	0.3391	−0.1610	0.7732	0.100*	
C9	0.3604 (3)	−0.0517 (3)	0.8017 (9)	0.0669 (16)	
H9A	0.4005	−0.0581	0.7927	0.080*	
C10	0.3376 (3)	0.0183 (3)	0.8289 (8)	0.0538 (13)	
C11	0.2765 (2)	0.0281 (3)	0.8369 (8)	0.0509 (12)	
C12	0.3765 (2)	0.0826 (2)	0.8410 (8)	0.0512 (13)	
C13	0.3889 (2)	0.2158 (3)	0.8965 (9)	0.0522 (14)	
C14	0.4212 (2)	0.2260 (3)	1.0623 (10)	0.0602 (15)	
H14A	0.4190	0.1914	1.1632	0.072*	
C15	0.4569 (3)	0.2869 (3)	1.0813 (11)	0.0629 (15)	
H15A	0.4787	0.2932	1.1949	0.075*	
C16	0.4609 (2)	0.3390 (3)	0.9330 (10)	0.0577 (14)	
C17	0.4289 (2)	0.3279 (3)	0.7643 (9)	0.0565 (14)	
H17A	0.4319	0.3619	0.6619	0.068*	
C18	0.3928 (2)	0.2670 (3)	0.7466 (9)	0.0544 (14)	
H18A	0.3710	0.2603	0.6332	0.065*	
O1W	0.5000	0.0000	0.0917 (13)	0.089 (2)	
H1W1	0.4830	0.0421	0.0966	0.107*	0.50
H2W1	0.5239	0.0012	−0.0033	0.107*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.055 (2)	0.063 (2)	0.094 (3)	0.0129 (17)	0.011 (3)	0.010 (2)
O2	0.067 (2)	0.0447 (18)	0.091 (3)	0.0124 (16)	−0.001 (3)	0.000 (2)
N1	0.053 (2)	0.0357 (18)	0.046 (2)	−0.0006 (16)	−0.004 (2)	0.0032 (18)
N2	0.102 (4)	0.079 (3)	0.106 (5)	−0.039 (3)	−0.022 (4)	0.011 (4)
C1	0.055 (3)	0.045 (2)	0.050 (3)	0.009 (2)	−0.003 (3)	0.005 (2)
C2	0.055 (3)	0.055 (2)	0.034 (3)	−0.004 (2)	−0.004 (2)	0.010 (2)
C3	0.063 (3)	0.078 (4)	0.042 (3)	−0.004 (3)	−0.004 (3)	0.009 (3)
C4	0.065 (4)	0.116 (5)	0.060 (4)	−0.028 (4)	−0.001 (3)	0.021 (4)
C5	0.097 (5)	0.087 (4)	0.056 (4)	−0.044 (4)	−0.012 (4)	0.018 (4)
C6	0.090 (5)	0.059 (3)	0.043 (3)	−0.027 (3)	−0.005 (3)	0.013 (3)
C7	0.136 (7)	0.052 (3)	0.058 (4)	−0.030 (4)	−0.018 (4)	0.013 (3)
C8	0.142 (7)	0.042 (3)	0.065 (4)	0.002 (4)	−0.006 (5)	−0.001 (3)
C9	0.102 (4)	0.047 (3)	0.052 (4)	0.008 (3)	0.001 (3)	0.001 (3)
C10	0.079 (4)	0.037 (2)	0.045 (3)	0.003 (2)	0.006 (3)	0.011 (2)
C11	0.072 (3)	0.048 (3)	0.032 (2)	−0.007 (2)	−0.002 (3)	0.008 (2)
C12	0.060 (3)	0.043 (3)	0.050 (3)	0.010 (2)	0.001 (3)	0.007 (2)
C13	0.053 (3)	0.038 (2)	0.066 (4)	0.004 (2)	0.001 (3)	0.004 (3)
C14	0.063 (3)	0.055 (3)	0.063 (4)	−0.002 (3)	−0.015 (3)	0.012 (3)
C15	0.061 (3)	0.058 (3)	0.071 (4)	−0.006 (3)	−0.016 (3)	−0.003 (3)
C16	0.053 (3)	0.045 (3)	0.075 (4)	−0.007 (2)	0.000 (3)	−0.007 (3)
C17	0.067 (3)	0.039 (2)	0.063 (4)	−0.003 (2)	−0.006 (3)	0.012 (3)
C18	0.059 (3)	0.044 (3)	0.060 (4)	0.000 (2)	−0.007 (3)	0.001 (3)
O1W	0.098 (5)	0.093 (4)	0.078 (4)	0.028 (4)	0.000	0.000

Geometric parameters (Å, º)

O1—C12	1.217 (6)	C7—H7A	0.9300
O2—C1	1.194 (6)	C8—C9	1.397 (9)
N1—C12	1.391 (6)	C8—H8A	0.9300
N1—C1	1.419 (6)	C9—C10	1.372 (7)
N1—C13	1.445 (6)	C9—H9A	0.9300
N2—C16	1.379 (6)	C10—C11	1.413 (7)
N2—H2A	0.8600	C10—C12	1.461 (7)
N2—H2B	0.8600	C13—C14	1.366 (8)
C1—C2	1.462 (7)	C13—C18	1.379 (7)
C2—C3	1.375 (8)	C14—C15	1.371 (7)
C2—C11	1.408 (7)	C14—H14A	0.9300
C3—C4	1.391 (8)	C15—C16	1.381 (8)
C3—H3A	0.9300	C15—H15A	0.9300
C4—C5	1.340 (9)	C16—C17	1.381 (8)
C4—H4A	0.9300	C17—C18	1.375 (7)
C5—C6	1.416 (9)	C17—H17A	0.9300
C5—H5A	0.9300	C18—H18A	0.9300
C6—C7	1.391 (8)	O1W—H1W1	0.8501
C6—C11	1.406 (6)	O1W—H2W1	0.8501
C7—C8	1.353 (10)		
C12—N1—C1	124.8 (4)	C10—C9—H9A	119.9
C12—N1—C13	118.3 (4)	C8—C9—H9A	119.9
C1—N1—C13	116.9 (4)	C9—C10—C11	119.8 (5)
C16—N2—H2A	120.0	C9—C10—C12	119.8 (5)
C16—N2—H2B	120.0	C11—C10—C12	120.3 (4)
H2A—N2—H2B	120.0	C6—C11—C2	120.3 (5)
O2—C1—N1	119.8 (4)	C6—C11—C10	119.5 (5)
O2—C1—C2	124.2 (5)	C2—C11—C10	120.2 (5)
N1—C1—C2	115.9 (4)	O1—C12—N1	119.2 (4)
C3—C2—C11	119.6 (5)	O1—C12—C10	123.5 (4)
C3—C2—C1	119.2 (5)	N1—C12—C10	117.3 (4)
C11—C2—C1	121.1 (5)	C14—C13—C18	119.5 (5)
C2—C3—C4	119.9 (6)	C14—C13—N1	120.8 (5)
C2—C3—H3A	120.1	C18—C13—N1	119.7 (5)
C4—C3—H3A	120.1	C13—C14—C15	120.6 (6)
C5—C4—C3	121.3 (6)	C13—C14—H14A	119.7
C5—C4—H4A	119.4	C15—C14—H14A	119.7
C3—C4—H4A	119.4	C14—C15—C16	120.5 (6)
C4—C5—C6	121.2 (6)	C14—C15—H15A	119.7
C4—C5—H5A	119.4	C16—C15—H15A	119.7
C6—C5—H5A	119.4	N2—C16—C17	120.6 (6)
C7—C6—C11	118.5 (6)	N2—C16—C15	120.6 (6)
C7—C6—C5	123.9 (6)	C17—C16—C15	118.8 (5)
C11—C6—C5	117.6 (5)	C18—C17—C16	120.4 (5)
C8—C7—C6	121.9 (6)	C18—C17—H17A	119.8

C8—C7—H7A	119.1	C16—C17—H17A	119.8
C6—C7—H7A	119.1	C17—C18—C13	120.2 (5)
C7—C8—C9	120.1 (6)	C17—C18—H18A	119.9
C7—C8—H8A	120.0	C13—C18—H18A	119.9
C9—C8—H8A	120.0	H1W1—O1W—H2W1	107.7
C10—C9—C8	120.2 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1*W*^{i}	0.86	2.19	3.046 (6)	172
N2—H2*B*···O1^{ii}	0.86	2.24	3.099 (5)	178
O1*W*—H1*W*1···O1^{iii}	0.85	2.30	2.800 (5)	118
O1*W*—H2*W*1···O1^{iv}	0.85	2.12	2.800 (5)	136
C15—H15*A*···*Cg*1^{v}	0.93	2.96		140

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