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6-Oxobenz[*de*]isoquinolino[2,1-*a*]-benzimidazolium chloride monohydrate

Fang-Fang Jian,* Li Du and Jing Wang

New Materials and Function Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China
Correspondence e-mail: ffj2003@163169.net

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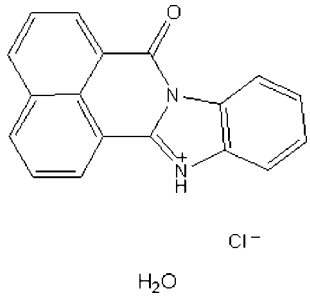
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.112; data-to-parameter ratio = 12.9.

The title compound, $C_{18}H_{11}N_2O^+\cdot Cl^-\cdot H_2O$, was prepared by the reaction of 1,8-naphthalic anhydride with *o*-phenylenediamine in DMF. The dihedral angle formed by the phenyl and naphthalic rings is 177.06°. The structure is stabilized by intramolecular C—H···O hydrogen bonds. There are N—H···Cl, O—H···Cl, C—H···O and C—H···Cl hydrogen bonds in the structure.

Related literature

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



Experimental

Crystal data

$C_{18}H_{11}N_2O^+\cdot Cl^-\cdot H_2O$
M_r = 324.75
Triclinic, $P\overline{1}$
a = 8.9000 (18) Å
b = 8.9440 (18) Å
c = 9.4480 (19) Å
α = 81.50 (3)°
β = 88.76 (3)°
γ = 77.61 (3)°
V = 726.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 295 (2) K
0.3 × 0.25 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
8800 measured reflections
2698 independent reflections
1852 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.02
2698 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1	0.86	2.19	3.044 (2)	175
O2—H2C···Cl1	0.85	2.48	3.253 (3)	153
O2—H2D···Cl1^{i}	0.85	2.35	3.195 (2)	171
C5—H5A···O1	0.93	2.48	2.964 (3)	113
C5—H5A···O1^{ii}	0.93	2.47	3.153 (2)	130
C9—H9A···Cl1	0.93	2.78	3.673 (2)	162
C15—H15A···O2^{iii}	0.93	2.58	3.313 (3)	136

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2048).

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cederfur, J., Pei, Y. X. E. C., Meng, Z. H. & Kempe, M. (2003). *J. Comb. Chem.* **5**, 67–72.
Ofir, Y. (2006). *J. Mater. Chem.* **16**, 2142–2143.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.

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supporting information

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6-Oxobenz[*de*]isoquinolino[2,1-*a*]benzimidazolium chloride monohydrate

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S1. Comment

1,8-Naphthlimide derivatives are an important function material during the recent years and are used as optical characters. 1,8-Naphthalimides exhibit hydrogen-bonding. and cation-dependent fluorescence. The naphthyl group can be used as a good receptor(Cederfur *et al.*, 2003).

In the title compound, the bond lengths and angles are normal in the 1,8-naphthalenedicarboximide and phenyl ring (Ofir, 2006). The dihedral angle formed by the phenyl(C1—C6) ring and naphthalic ring(N1—C18) is 177.06°.

There are π-π stacking interactions in the molecular in the structure. The distance between ring centroids *Cg*(1)—*Cg*(5)(i) are 3.578 (2); *Cg*(2)—*Cg*(2)(i) are 3.648 (2); *Cg*(2)—*Cg*(5)(i) are 3.845 (2) [symmetry code:i=-*x*,1 - *y*,1 - *z*].where *Cg*1 is the centre-of-gravity of the ring defined by atoms (N1—C6—C1—C2—C7), *Cg*2 is the centre-of-gravity of the ring defined by atoms (N1—C7—C8—C17—C16—C18) and *Cg*5 is the centre-of-gravity of the ring defined by atoms (C12—C13—C14—C15—C16—C17).

The crystal packing is stabilized by N2—H2A···Cl,O2—H2C···Cl1, C5—H5A···O1, C9—H9A···Cl1, C15—H15···O2 and O2- H2D···Cl1 hydrogen bonds. (Table 2).

S2. Experimental

The single crystals of the title compound were obtained by reaction *o*-phenylene diamine(0.2 mmol) with 1,8-naphthalic anhydride (0.2 mmol) in refluxing DMF(50 ml). The product (yield 87%) was stirred in the DMF. Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from DMF and HCl 4:1 (*v*/*v*) solution at room temperature.

S3. Refinement

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}. The asymmetric unit comprises a hydrogen bonded unit.

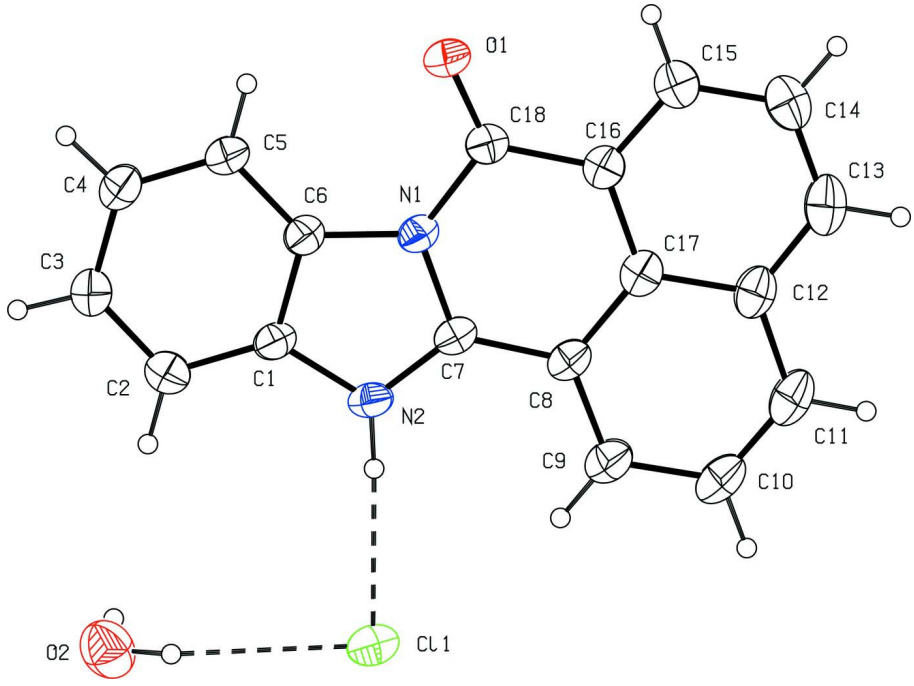

Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

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Crystal data

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$c = 9.4480\ (19)$ Å
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$\beta = 88.76\ (3)°$
$\gamma = 77.61\ (3)°$
$V = 726.5\ (3)$ Å^3
$Z = 2$
$F(000) = 336$
$D_x = 1.485$ Mg m^{-3}
Melting point: 210 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 512 reflections
$\theta = 2–22°$
$\mu = 0.28$ mm^{-1}
$T = 295$ K
Block, colorless
0.3 × 0.25 × 0.2 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
8800 measured reflections
2698 independent reflections
1852 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.2°$
$h = -10\rightarrow10$
$k = -8\rightarrow10$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.112$
$S = 1.02$
2698 reflections
209 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.1865P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.008 (2)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1171 (2)	0.7662 (2)	0.52217 (19)	0.0619 (5)
N1	−0.0148 (2)	0.6439 (2)	0.69142 (19)	0.0415 (5)
N2	−0.1494 (2)	0.5542 (2)	0.8640 (2)	0.0467 (5)
H2A	−0.1834	0.4913	0.9281	0.056*
C1	−0.2061 (3)	0.7127 (3)	0.8379 (2)	0.0444 (6)
C2	−0.3250 (3)	0.8054 (3)	0.8995 (3)	0.0583 (7)
H2B	−0.3835	0.7655	0.9724	0.070*
C3	−0.3523 (3)	0.9601 (3)	0.8470 (3)	0.0614 (7)
H3A	−0.4322	1.0273	0.8850	0.074*
C4	−0.2649 (3)	1.0189 (3)	0.7400 (3)	0.0570 (7)
H4A	−0.2866	1.1249	0.7089	0.068*
C5	−0.1473 (3)	0.9268 (3)	0.6777 (3)	0.0493 (6)
H5A	−0.0893	0.9670	0.6044	0.059*
C6	−0.1196 (2)	0.7714 (3)	0.7295 (2)	0.0428 (6)
C7	−0.0361 (2)	0.5140 (3)	0.7766 (2)	0.0418 (5)
C8	0.0551 (3)	0.3648 (3)	0.7634 (2)	0.0453 (6)
C9	0.0314 (3)	0.2316 (3)	0.8436 (3)	0.0570 (7)
H9A	−0.0465	0.2362	0.9112	0.068*
C10	0.1232 (3)	0.0895 (3)	0.8245 (3)	0.0666 (8)
H10A	0.1067	−0.0004	0.8799	0.080*
C11	0.2356 (3)	0.0807 (3)	0.7269 (3)	0.0650 (8)
H11A	0.2955	−0.0156	0.7155	0.078*
C12	0.2643 (3)	0.2142 (3)	0.6415 (3)	0.0548 (7)

C13	0.3794 (3)	0.2111 (4)	0.5386 (3)	0.0673 (8)
H13A	0.4432	0.1167	0.5273	0.081*
C14	0.4013 (3)	0.3414 (4)	0.4543 (3)	0.0632 (7)
H14A	0.4777	0.3354	0.3854	0.076*
C15	0.3092 (3)	0.4827 (3)	0.4716 (3)	0.0535 (6)
H15A	0.3238	0.5719	0.4136	0.064*
C16	0.1967 (3)	0.4934 (3)	0.5731 (2)	0.0453 (6)
C17	0.1714 (3)	0.3589 (3)	0.6603 (3)	0.0466 (6)
C18	0.1035 (3)	0.6455 (3)	0.5888 (2)	0.0451 (6)
Cl1	−0.26953 (7)	0.34446 (8)	1.10307 (7)	0.0603 (2)
O2	−0.5321 (2)	0.6341 (3)	1.1799 (2)	0.0821 (6)
H2C	−0.4470	0.5687	1.1836	0.098*
H2D	−0.5787	0.6295	1.1035	0.098*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0707 (11)	0.0447 (11)	0.0648 (12)	−0.0123 (9)	0.0176 (9)	0.0077 (9)
N1	0.0449 (10)	0.0346 (11)	0.0446 (11)	−0.0109 (9)	−0.0005 (8)	−0.0005 (9)
N2	0.0538 (11)	0.0415 (12)	0.0434 (11)	−0.0150 (9)	0.0040 (9)	0.0047 (9)
C1	0.0478 (13)	0.0382 (14)	0.0448 (13)	−0.0101 (11)	−0.0010 (11)	0.0030 (11)
C2	0.0559 (15)	0.0577 (18)	0.0587 (16)	−0.0110 (13)	0.0133 (13)	−0.0033 (14)
C3	0.0596 (16)	0.0516 (17)	0.0666 (18)	−0.0002 (13)	0.0115 (13)	−0.0064 (14)
C4	0.0608 (16)	0.0401 (15)	0.0655 (17)	−0.0050 (12)	0.0016 (13)	−0.0022 (13)
C5	0.0554 (14)	0.0404 (14)	0.0505 (14)	−0.0126 (12)	0.0036 (12)	0.0013 (11)
C6	0.0422 (12)	0.0393 (14)	0.0462 (14)	−0.0092 (11)	−0.0014 (10)	−0.0030 (11)
C7	0.0457 (12)	0.0367 (13)	0.0429 (13)	−0.0125 (11)	−0.0044 (11)	0.0003 (11)
C8	0.0524 (13)	0.0368 (14)	0.0467 (14)	−0.0119 (11)	−0.0074 (11)	−0.0018 (11)
C9	0.0677 (16)	0.0435 (16)	0.0590 (16)	−0.0141 (13)	−0.0031 (13)	−0.0008 (13)
C10	0.0822 (19)	0.0381 (16)	0.077 (2)	−0.0138 (14)	−0.0093 (16)	0.0011 (14)
C11	0.0748 (18)	0.0391 (16)	0.080 (2)	−0.0058 (14)	−0.0160 (16)	−0.0138 (15)
C12	0.0559 (15)	0.0441 (16)	0.0649 (17)	−0.0056 (12)	−0.0109 (13)	−0.0148 (13)
C13	0.0624 (17)	0.0588 (19)	0.082 (2)	−0.0009 (14)	−0.0076 (16)	−0.0301 (17)
C14	0.0557 (16)	0.072 (2)	0.0666 (18)	−0.0134 (15)	0.0056 (13)	−0.0271 (16)
C15	0.0512 (14)	0.0576 (17)	0.0539 (15)	−0.0125 (13)	−0.0013 (12)	−0.0139 (13)
C16	0.0453 (13)	0.0443 (15)	0.0474 (14)	−0.0105 (11)	−0.0028 (11)	−0.0083 (11)
C17	0.0481 (13)	0.0444 (15)	0.0489 (14)	−0.0100 (11)	−0.0100 (11)	−0.0100 (11)
C18	0.0454 (13)	0.0451 (15)	0.0442 (14)	−0.0112 (11)	−0.0011 (11)	−0.0027 (12)
Cl1	0.0674 (4)	0.0554 (5)	0.0554 (4)	−0.0173 (3)	−0.0016 (3)	0.0071 (3)
O2	0.0807 (13)	0.0973 (17)	0.0678 (13)	−0.0119 (12)	0.0008 (11)	−0.0218 (12)

Geometric parameters (Å, º)

O1—C18	1.195 (3)	C8—C17	1.404 (3)
N1—C7	1.359 (3)	C9—C10	1.389 (4)
N1—C6	1.397 (3)	C9—H9A	0.9300
N1—C18	1.416 (3)	C10—C11	1.344 (4)
N2—C7	1.312 (3)	C10—H10A	0.9300

N2—C1	1.385 (3)	C11—C12	1.407 (4)
N2—H2A	0.8600	C11—H11A	0.9300
C1—C2	1.373 (3)	C12—C13	1.396 (4)
C1—C6	1.377 (3)	C12—C17	1.411 (3)
C2—C3	1.370 (4)	C13—C14	1.359 (4)
C2—H2B	0.9300	C13—H13A	0.9300
C3—C4	1.374 (4)	C14—C15	1.381 (4)
C3—H3A	0.9300	C14—H14A	0.9300
C4—C5	1.366 (4)	C15—C16	1.369 (3)
C4—H4A	0.9300	C15—H15A	0.9300
C5—C6	1.375 (3)	C16—C17	1.412 (3)
C5—H5A	0.9300	C16—C18	1.459 (3)
C7—C8	1.427 (3)	O2—H2C	0.8500
C8—C9	1.369 (3)	O2—H2D	0.8500

C7—N1—C6	108.91 (18)	C8—C9—H9A	119.9
C7—N1—C18	123.8 (2)	C10—C9—H9A	119.9
C6—N1—C18	127.18 (19)	C11—C10—C9	120.7 (3)
C7—N2—C1	110.12 (19)	C11—C10—H10A	119.6
C7—N2—H2A	124.9	C9—C10—H10A	119.6
C1—N2—H2A	124.9	C10—C11—C12	121.2 (3)
C2—C1—C6	122.1 (2)	C10—C11—H11A	119.4
C2—C1—N2	130.9 (2)	C12—C11—H11A	119.4
C6—C1—N2	107.0 (2)	C13—C12—C11	123.4 (3)
C3—C2—C1	116.0 (2)	C13—C12—C17	118.2 (3)
C3—C2—H2B	122.0	C11—C12—C17	118.4 (3)
C1—C2—H2B	122.0	C14—C13—C12	122.2 (3)
C2—C3—C4	121.9 (2)	C14—C13—H13A	118.9
C2—C3—H3A	119.1	C12—C13—H13A	118.9
C4—C3—H3A	119.1	C13—C14—C15	119.5 (3)
C5—C4—C3	122.3 (2)	C13—C14—H14A	120.2
C5—C4—H4A	118.9	C15—C14—H14A	120.2
C3—C4—H4A	118.9	C16—C15—C14	120.9 (3)
C4—C5—C6	116.1 (2)	C16—C15—H15A	119.6
C4—C5—H5A	121.9	C14—C15—H15A	119.6
C6—C5—H5A	121.9	C15—C16—C17	120.2 (2)
C5—C6—C1	121.6 (2)	C15—C16—C18	119.0 (2)
C5—C6—N1	132.6 (2)	C17—C16—C18	120.8 (2)
C1—C6—N1	105.75 (19)	C8—C17—C12	119.2 (2)
N2—C7—N1	108.2 (2)	C8—C17—C16	121.9 (2)
N2—C7—C8	129.9 (2)	C12—C17—C16	118.9 (2)
N1—C7—C8	121.9 (2)	O1—C18—N1	119.1 (2)
C9—C8—C17	120.3 (2)	O1—C18—C16	126.1 (2)
C9—C8—C7	123.0 (2)	N1—C18—C16	114.8 (2)
C17—C8—C7	116.7 (2)	H2C—O2—H2D	107.7
C8—C9—C10	120.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1	0.86	2.19	3.044 (2)	175
O2—H2*C*···Cl1	0.85	2.48	3.253 (3)	153
O2—H2*D*···Cl1^{i}	0.85	2.35	3.195 (2)	171
C5—H5*A*···O1	0.93	2.48	2.964 (3)	113
C5—H5*A*···O1^{ii}	0.93	2.47	3.153 (2)	130
C9—H9*A*···Cl1	0.93	2.78	3.673 (2)	162
C15—H15*A*···O2^{iii}	0.93	2.58	3.313 (3)	136

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