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1-Diazonaphthalen-2(1*H*)-one

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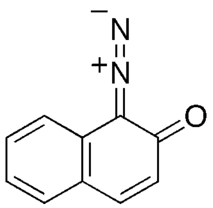
Key indicators: single-crystal X-ray study; *T* = 123 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.044; *wR* factor = 0.119; data-to-parameter ratio = 12.2.

The molecule of the title compound, $C_{10}H_6N_2O$, is nearly planar [maximum deviation = 0.030 (1) Å]. The CN_2 moiety is almost linear, with a C—N—N angle of 175.50 (14)°. A single intermolecular C—H⋯O hydrogen bond is observed in the crystal structure. A π–π interaction is also observed with the shortest distance being 3.6832 (12) Å between the the centroids of the six-membered rings.

Related literature

For the synthesis, see: Kitamura *et al.* (2010). For the crystal structure of related diazonaphthoquinones, see: Seidel *et al.* (1989); Ferreira *et al.* (2006). For an example of the utility of the diazonaphthoquinones, see Reiser *et al.* (1996).



Experimental

Crystal data

$C_{10}H_6N_2O$
$M_r = 170.17$
Orthorhombic, *Pbca*
$a = 11.900$ (2) Å
$b = 9.1978$ (15) Å
$c = 14.521$ (3) Å
$V = 1589.4$ (5) Å^3
$Z = 8$
Cu *Kα* radiation
$\mu = 0.78$ mm^{-1}
$T = 123$ K
0.50 × 0.40 × 0.40 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.566$, $T_{\max} = 0.731$
18038 measured reflections
1456 independent reflections
1359 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.119$
$S = 1.08$
1456 reflections
119 parameters
All H-atom parameters refined
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C6—H3⋯O1$^{\rm i}$	0.95	2.55	3.466 (2)	162

Symmetry code: (i) $-x+\frac{1}{2}, -y+2, z+\frac{1}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku Americas and Rigaku, 2007); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *CrystalStructure* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2420).

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supplementary materials

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1-Diazonaphthalen-2(1*H*)-one

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Comment

1,2-Diazonaphthoquinone derivatives are unique cyclic α-diazocarobonyl compounds that can be drawn as diazonium naphtholate resonance forms, and are exclusively used photoresists such as novolak-diazonaphthoquinone resist (Reiser, *et al.*, 1996). The reports on the X-ray structural data of diazonaphthoquinones are limitted (Seidel *et al.*, 1989; Ferreira *et al.*, 2006). We have synthesized the simplest diazonaphthoquinone, 1-diazo-1*H*-naphthalen-2-one, by the diazo-transfer reaction (Kitamura *et al.*, 2010) and determined its crystal structure which is being reported in this article.

In the structure of the title compound (Fig. 1) the CN_2 moiety is almost linear, with C1—N1—N2 = 175.50 (14)°. The bond length N1—N2 and C1—N2 are 1.1210 (19) and 1.3355 (19) Å. The keto C═O bond length is 1.2474 (19) Å, which is close to a double bond. These data suggest that the structure of the title compound is not diazonium naphtholate form in the solid state.

A single intermolecular hydrogen bond is observed C6—H3···O1^{i} is observed in the crystal structure (Fig. 2). In addition, a π –π interaction is obserbed with the shotest distance 3.6832 (12) Å between the the centroids of the six memberd rings.

Experimental

To a solution of 2-chloro-1,3-dimethylimidazolinium chloride (228 mg, 1.35 mmol) in acetonitrile (2 ml), sodium azide (99.4 mg, 1.5 mmol) and 15-crown-5 ether (0.06 ml, 0.3 mmol) were added at 253 K and the mixture was stirred for 30 min. 2-Naphthol (130 mg, 0.90 mmol) and triethylamine (0.25 ml, 1.8 mmol) in THF (4 ml) were added to the mixture, which was stirred for 20 min. The reaction was quenched with water, and organic materials were extracted three times with CH_2Cl_2. The combined extracts were washed with water and brine, and then, dried over anhydrous sodium sulfate. The solvent was removed *in vacuo* to afford crude compound. The crude material was purified by flash column chromatography (silica gel: hexane/ethyl acetate = 4/1) to give the title compound in 86% yield. Single-crystals suitable for X-ray crystallographic analysis were obtained by recrystallization from a mixture of hexane and ethyl acetate (5:1).

Refinement

H atoms were positioned geometrically and were refined in as riding mode on the parent atoms, with C–H distances of 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

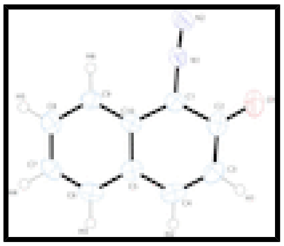

Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

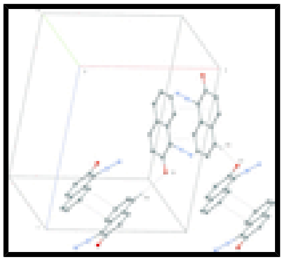

Fig. 2. A unit cell packing diagram showing hydrogen bonds and π–π interaction; H-atoms not involved in H-bonds have been excluded for clarity.

1-Diazonaphthalen-2(1*H*)-one

Crystal data

$C_{10}H_6N_2O$

$M_r = 170.17$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 11.900$ (2) Å

$b = 9.1978$ (15) Å

$c = 14.521$ (3) Å

$V = 1589.4$ (5) Å^3

$Z = 8$

$F(000) = 704.00$

$D_x = 1.422$ Mg m^{-3}

Cu *K*α radiation, λ = 1.54187 Å

Cell parameters from 17442 reflections

θ = 3.0–68.2°

μ = 0.78 mm^{-1}

$T = 123$ K

Prism, brown

0.50 × 0.40 × 0.40 mm

Data collection

Rigaku R-AXIS RAPID diffractometer

Detector resolution: 5.00 pixels mm^{-1}

ω scans

Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.566$, $T_{\max} = 0.731$

18038 measured reflections

1456 independent reflections

1359 reflections with $F^2 > 2\sigma(F^2)$

$R_{\rm int} = 0.018$

$\theta_{\max} = 68.2°$

$h = -14 \rightarrow 14$

$k = -10 \rightarrow 10$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.044$

0 restraints

All H-atom parameters refined

$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.5878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\max} < 0.001$
1456 reflections	$\Delta\rho_{\max} = 0.29$ e Å^{-3}
119 parameters	$\Delta\rho_{\min} = -0.13$ 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 was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
O(1)	0.41204 (11)	1.07612 (13)	0.22608 (7)	0.0476 (3)
N(1)	0.55271 (11)	0.89513 (13)	0.31064 (8)	0.0327 (3)
N(2)	0.63438 (13)	0.87983 (16)	0.27341 (9)	0.0441 (3)
C(1)	0.45523 (13)	0.92398 (16)	0.35238 (10)	0.0336 (3)
C(2)	0.38659 (14)	1.02931 (17)	0.30396 (11)	0.0390 (4)
C(3)	0.28687 (14)	1.07339 (17)	0.35513 (11)	0.0406 (4)
C(4)	0.26340 (13)	1.01769 (18)	0.43965 (12)	0.0419 (4)
C(5)	0.33263 (12)	0.91102 (16)	0.48560 (10)	0.0344 (3)
C(6)	0.30613 (13)	0.85591 (18)	0.57389 (11)	0.0389 (4)
C(7)	0.37387 (14)	0.75293 (19)	0.61482 (11)	0.0395 (4)
C(8)	0.46903 (13)	0.70173 (19)	0.56895 (11)	0.0388 (4)
C(9)	0.49762 (13)	0.75479 (17)	0.48334 (10)	0.0343 (3)
C(10)	0.43117 (12)	0.85986 (16)	0.44128 (10)	0.0310 (3)
H(1)	0.2371	1.1427	0.3288	0.049*
H(2)	0.1977	1.0508	0.4703	0.050*
H(3)	0.2412	0.8901	0.6052	0.047*
H(4)	0.3558	0.7168	0.6743	0.047*
H(5)	0.5146	0.6295	0.5971	0.047*
H(6)	0.5629	0.7195	0.4530	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O(1)	0.0703 (8)	0.0379 (6)	0.0347 (6)	0.0036 (5)	−0.0079 (5)	0.0033 (4)
N(1)	0.0406 (7)	0.0295 (6)	0.0279 (6)	−0.0016 (5)	0.0014 (5)	0.0001 (4)
N(2)	0.0509 (8)	0.0411 (8)	0.0403 (7)	−0.0027 (6)	0.0117 (6)	0.0025 (6)
C(1)	0.0362 (8)	0.0325 (7)	0.0320 (7)	−0.0001 (6)	−0.0012 (6)	−0.0042 (5)
C(2)	0.0501 (9)	0.0313 (8)	0.0355 (8)	−0.0008 (6)	−0.0102 (7)	−0.0042 (6)
C(3)	0.0414 (8)	0.0361 (8)	0.0442 (9)	0.0067 (6)	−0.0124 (7)	−0.0073 (6)

C(4)	0.0327 (7)	0.0423 (8)	0.0506 (9)	0.0021 (6)	−0.0036 (6)	−0.0167 (7)
C(5)	0.0317 (7)	0.0345 (8)	0.0371 (8)	−0.0045 (6)	−0.0059 (6)	−0.0105 (6)
C(6)	0.0305 (7)	0.0483 (9)	0.0379 (8)	−0.0096 (6)	0.0041 (6)	−0.0150 (7)
C(7)	0.0428 (8)	0.0484 (9)	0.0274 (7)	−0.0132 (7)	0.0011 (6)	−0.0029 (6)
C(8)	0.0388 (8)	0.0428 (9)	0.0349 (8)	−0.0056 (6)	−0.0043 (6)	0.0004 (6)
C(9)	0.0300 (6)	0.0384 (8)	0.0344 (8)	−0.0019 (6)	−0.0001 (6)	−0.0027 (6)
C(10)	0.0308 (7)	0.0336 (7)	0.0288 (7)	−0.0074 (5)	−0.0015 (5)	−0.0071 (5)

Geometric parameters (Å, °)

O(1)—C(2)	1.2474 (19)	C(6)—C(7)	1.378 (2)
N(1)—N(2)	1.1210 (19)	C(7)—C(8)	1.396 (2)
N(1)—C(1)	1.3355 (19)	C(8)—C(9)	1.378 (2)
C(1)—C(2)	1.449 (2)	C(9)—C(10)	1.390 (2)
C(1)—C(10)	1.448 (2)	C(3)—H(1)	0.950
C(2)—C(3)	1.458 (2)	C(4)—H(2)	0.950
C(3)—C(4)	1.359 (2)	C(6)—H(3)	0.950
C(4)—C(5)	1.444 (2)	C(7)—H(4)	0.950
C(5)—C(6)	1.414 (2)	C(8)—H(5)	0.950
C(5)—C(10)	1.418 (2)	C(9)—H(6)	0.950
N(2)—N(1)—C(1)	175.50 (14)	C(1)—C(10)—C(5)	115.66 (12)
N(1)—C(1)—C(2)	113.72 (13)	C(1)—C(10)—C(9)	124.23 (13)
N(1)—C(1)—C(10)	119.70 (13)	C(5)—C(10)—C(9)	120.11 (13)
C(2)—C(1)—C(10)	126.40 (13)	C(2)—C(3)—H(1)	119.2
O(1)—C(2)—C(1)	122.26 (14)	C(4)—C(3)—H(1)	119.2
O(1)—C(2)—C(3)	124.32 (14)	C(3)—C(4)—H(2)	118.1
C(1)—C(2)—C(3)	113.42 (13)	C(5)—C(4)—H(2)	118.1
C(2)—C(3)—C(4)	121.52 (14)	C(5)—C(6)—H(3)	119.8
C(3)—C(4)—C(5)	123.78 (14)	C(7)—C(6)—H(3)	119.8
C(4)—C(5)—C(6)	122.35 (13)	C(6)—C(7)—H(4)	120.0
C(4)—C(5)—C(10)	119.17 (13)	C(8)—C(7)—H(4)	120.0
C(6)—C(5)—C(10)	118.48 (13)	C(7)—C(8)—H(5)	119.6
C(5)—C(6)—C(7)	120.45 (14)	C(9)—C(8)—H(5)	119.6
C(6)—C(7)—C(8)	120.04 (14)	C(8)—C(9)—H(6)	119.9
C(7)—C(8)—C(9)	120.75 (15)	C(10)—C(9)—H(6)	119.9
C(8)—C(9)—C(10)	120.14 (14)		
N(2)—N(1)—C(1)—C(2)	29.7 (19)	C(3)—C(4)—C(5)—C(6)	179.60 (15)
N(2)—N(1)—C(1)—C(10)	−145.8 (18)	C(3)—C(4)—C(5)—C(10)	−0.0 (2)
N(1)—C(1)—C(2)—O(1)	7.0 (2)	C(4)—C(5)—C(6)—C(7)	179.26 (15)
N(1)—C(1)—C(2)—C(3)	−173.04 (13)	C(4)—C(5)—C(10)—C(1)	1.5 (2)
N(1)—C(1)—C(10)—C(5)	172.20 (13)	C(4)—C(5)—C(10)—C(9)	−178.53 (14)
N(1)—C(1)—C(10)—C(9)	−7.8 (2)	C(6)—C(5)—C(10)—C(1)	−178.15 (13)
C(2)—C(1)—C(10)—C(5)	−2.6 (2)	C(6)—C(5)—C(10)—C(9)	1.8 (2)
C(2)—C(1)—C(10)—C(9)	177.38 (15)	C(10)—C(5)—C(6)—C(7)	−1.1 (2)
C(10)—C(1)—C(2)—O(1)	−177.92 (14)	C(5)—C(6)—C(7)—C(8)	−0.3 (2)
C(10)—C(1)—C(2)—C(3)	2.1 (2)	C(6)—C(7)—C(8)—C(9)	1.1 (2)
O(1)—C(2)—C(3)—C(4)	179.61 (15)	C(7)—C(8)—C(9)—C(10)	−0.4 (2)
C(1)—C(2)—C(3)—C(4)	−0.4 (2)	C(8)—C(9)—C(10)—C(1)	178.89 (14)
C(2)—C(3)—C(4)—C(5)	−0.6 (2)	C(8)—C(9)—C(10)—C(5)	−1.1 (2)

Hydrogen-bond geometry (°)

D—H···*A*
—···

Fig. 1

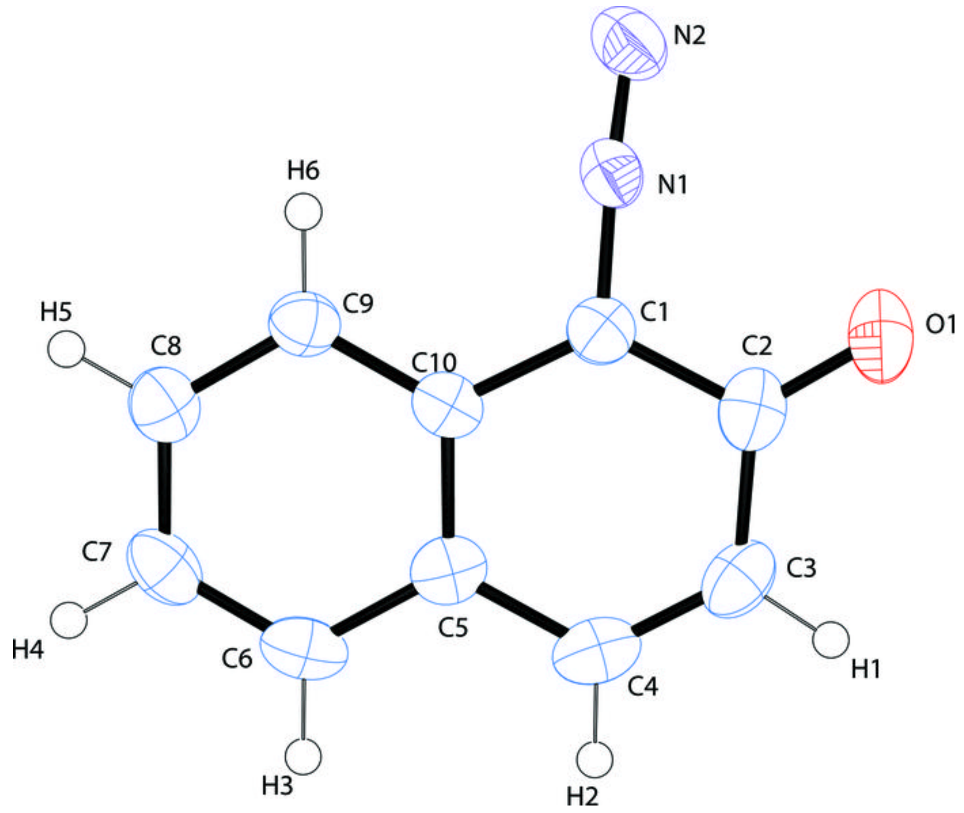

Fig. 2