

C1—C2—C3	120.7 (4)	C9—N17—H17N	107 (2)
C2—C1—C11	121.1 (4)	C14—C9—N17	105.3 (3)
C1—C11—C10	125.9 (4)	C9—N17—C16	112.4 (3)
C1—C11—C12	116.2 (4)	C9—N17—C18	113.1 (4)
C2—C3—C4	117.3 (4)	C10—C11—C12	117.7 (4)
C2—C3—O1	126.9 (4)	C11—C12—C13	127.3 (4)
C3—C4—C12	120.6 (4)	C12—C13—C14	108.2 (4)
C3—C4—O2	126.2 (3)	C12—C13—C15	110.5 (4)
C4—C3—O1	115.5 (4)	C14—C13—C15	108.3 (4)
C3—O1—C20	120.7 (4)	C13—C15—C16	111.7 (4)
C4—C12—C11	123.6 (4)	C15—C16—N17	110.8 (4)
C4—C12—C13	108.9 (4)	C16—N17—C18	111.1 (4)
C12—C4—O2	113.2 (3)	C20—C21—C22	109.0 (4)
C4—O2—C5	106.5 (3)	C20—C21—O4	109.9 (4)
C5—C6—C7	114.1 (4)	C21—C20—O1	108.5 (3)
C5—C6—O19	112.2 (3)	C21—C20—O3	109.6 (4)
C6—C5—C13	112.5 (4)	O1—C20—O3	104.3 (3)
C6—C5—O2	110.3 (3)	C20—O3—C24	111.3 (3)
C5—C13—C12	100.6 (3)	C21—C22—C23	110.2 (3)
C5—C13—C14	118.6 (4)	C21—C22—O5	108.2 (3)
C5—C13—C15	110.2 (4)	C22—C21—O4	112.4 (3)
C13—C5—O2	105.7 (3)	C22—C23—C24	108.4 (4)
C6—C7—C8	123.8 (4)	C22—C23—O6	107.8 (3)
C7—C6—O19	109.4 (4)	C23—C22—O5	110.2 (3)
C7—C8—C14	119.1 (4)	C23—C24—C25	112.1 (4)
C8—C14—C9	111.6 (4)	C23—C24—O3	110.0 (3)
C8—C14—C13	109.8 (4)	C24—C23—O6	111.1 (3)
C9—C10—C11	115.1 (4)	C24—C25—O7	117.3 (4)
C10—C9—C14	115.1 (3)	C24—C25—O8	116.3 (4)
C10—C9—N17	112.1 (3)	C25—C24—O3	106.6 (3)
C9—C14—C13	107.8 (3)	O7—C25—O8	126.2 (4)
H1W—O1W—H2W	105 (5)	C16—N17—H17N	112 (2)
H3W—O2W—H4W	82 (5)	C18—N17—H17N	101 (2)
H5W—O3W—H6W	97 (5)	C22—O5—H50	101 (4)
C6—O19—H190	129 (3)	C23—O6—H60	113 (3)

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N17—H17N...O2W	1.09 (4)	1.90 (4)	2.886 (5)	149 (3)
O6—H60...O3W	0.94 (4)	1.73 (4)	2.668 (6)	175 (4)
O1W—H1W...O2	0.95 (6)	2.20 (6)	3.143 (5)	170 (5)
O4—H40...O8 ⁱ	1.02 (4)	1.74 (4)	2.743 (4)	167 (4)
O19—H190...O7 ⁱⁱ	0.92 (5)	1.96 (4)	2.725 (5)	140 (4)
O1W—H2W...O8	1.26 (5)	1.60 (5)	2.860 (5)	175 (4)
O2W—H4W...O8 ⁱⁱⁱ	1.09 (4)	2.37 (5)	2.951 (5)	111 (4)
O3W—H5W...O7 ^{iv}	1.01 (5)	1.84 (5)	2.810 (6)	160 (3)
O3W—H5W...O3	1.01 (5)	2.40 (4)	3.040 (6)	121 (4)
O3W—H6W...O1W	0.88 (6)	2.30 (5)	2.815 (6)	118 (5)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$; (iv) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$.

Intensities were corrected for Lp effects and variation in the intensity of the standard reflections. The structure was solved by direct methods (SHELXS86; Sheldrick, 1985) and refined by full-matrix least squares minimizing $\sum w(\Delta F)^2$, where $w = 1/\sigma^2(F)$ and σ was obtained from counting statistics (SHELX76; Sheldrick, 1976). All water, hydroxyl and H(N) atoms were found from ΔF synthesis and refined; otherwise, the atomic parameters were recalculated after every six cycles of refinement. The electron-density maximum always accompanied the O2W atom, showing that the second water molecule is slightly disordered. No fluctuations of electron density above that value were observed on the final $\Delta\rho$ maps. All calculations were performed on a PC/486 computer, using the programs quoted above and our own supporting programs.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 1187–1189

3-Acetylamino-1,4-benzoquinone 4-Oxime

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(Received 17 January 1994; accepted 7 November 1994)

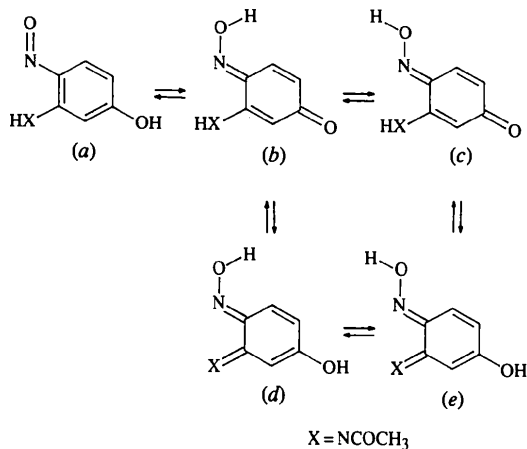
Abstract

In the title compound, $C_8H_8N_2O_3$, the ring system conforms to an almost planar configuration. The mean values of the two short and four long C—C bond distances in the six-membered ring are 1.339 (8) and 1.450 (8) Å, respectively. In the oxime group, the bond dimensions are C—N 1.301 (7), N—O 1.361 (6) and C—N—O 112.5 (4)°. The quinonic C=O bond distance is 1.234 (7) Å, which is similar to the C=O bond length [1.222 (7) Å] of the amide group. The crystal structure is stabilized by intermolecular hydrogen bonding.

Comment

It has been well established that in solution quinone monooximes exhibit nitrosphenolic/quinone oximic tautomerism (Burawoy, Cais, Chamberlain, Liversedge

& Thompson, 1955; French & Perkins, 1937; Hadzi, 1956; Hayashi, Shinra & Shono, 1971; Fischer, Golding & Tennant, 1965). Three tautomers can be written for the title compound and for two of these, *cis/trans* isomers about the oxime N—O bond are possible. Analysis shows the title structure to correspond to (b).



The structures of several 1,2- and 1,4-quinone monooximes have been determined in the solid state by single-crystal X-ray analyses (Charalambous *et al.*, 1991; Bartindale, Crowder & Morley, 1959; Boeyens, 1976; Fischmann & Romers, 1960; Fischmann, Romers & Shoemaker, 1957; Korvenranta, Näsäkkälä & Saari-nen, 1977*a,b*). These studies have all shown that the compounds are essentially quinone oximic in character. This is indicated by the presence of four long and two short C—C ring bond lengths (1.31–1.34 and 1.45–1.47 Å, respectively), and relatively short C—O and C—N bond lengths (1.21–1.28 and 1.28–1.37 Å, respectively). Recently, X-ray studies have shown that the product arising from the nitrosation of 3-hydroxy-2-

methylphenol exists in the 1,4-quinone 4-oximic rather than the 1,2-quinone 2-oximic form (Charalambous *et al.*, 1991).

The observed bond lengths are in good agreement with the corresponding ones reported earlier for both 3-hydroxy-2-methyl-1,4-benzoquinone 4-oxime (Charalambous *et al.*, 1991) and 3-chloro-1,4-benzoquinone 4-oxime (Romers, Shoemaker & Fischmann, 1957), and also with those given by Allen *et al.* (1987). Pronounced double-bond character is shown by C(1)—N(2), C(4)—O(1), C(7)—O(3), C(2)—C(3) and C(6)—C(5). Atom O(2) of the oxime group lies 0.12 (2) Å above the plane of the C(1)—C(6) ring. As with the structures of other 1,4-quinone monooximes, the OH of the NOH group is *anti* to the group closest to it, *i.e.* the acetyl group.

Experimental

3-Acetylamino-1,4-benzoquinone 4-oxime was obtained from the nitrosation of 3-acetylamino-phenol in the presence of nickel(II) chloride hexahydrate together with bis(5-acetylamino-1,2-benzoquinone 2-oximato)nickel(II) hexahydrate, and was recrystallized from ethyl acetate (Barjesteh, Charalambous & Pathirana, 1993).

Crystal data

C₈H₈N₂O₃
M_r = 180.16
 Monoclinic
*P*2₁/*n*
a = 16.652 (3) Å
b = 12.754 (3) Å
c = 3.841 (2) Å
 β = 94.39 (2)°
V = 813.4 (5) Å³
Z = 4
D_x = 1.47 Mg m⁻³

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 3–25°
 μ = 0.107 mm⁻¹
T = 295 K
 Needle-like
 0.30 × 0.16 × 0.12 mm
 Yellow

Data collection

Philips PW1100 diffractometer
 ω -2 θ scans
 Absorption correction: none
 1684 measured reflections
 1341 independent reflections
 548 observed reflections
 [*I* > 3 σ (*I*)]

*R*_{int} = 0.001
 θ _{max} = 25°
h = -17 → 19
k = 0 → 14
l = 0 → 4
 3 standard reflections
 frequency: 300 min
 intensity decay: <3%

Refinement

Refinement on *F*
R = 0.0560
wR = 0.0545
S = 1.37
 548 reflections
 79 parameters
 H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
 $(\Delta/\sigma)_{\max} = 0.02$
 $\Delta\rho_{\max} = 0.3 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = 0.1 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors from SHELX76 (Sheldrick, 1976)

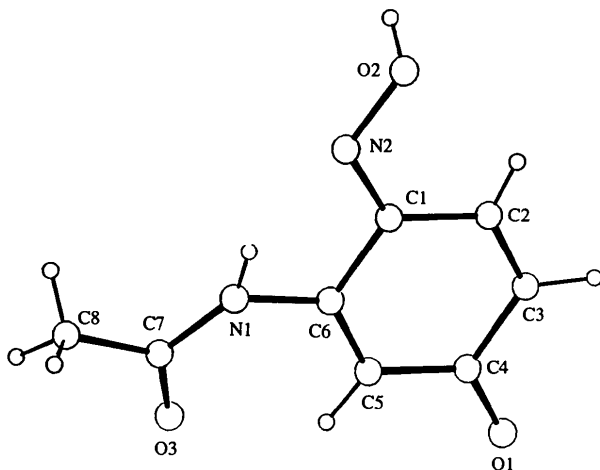


Fig. 1. Perspective view of the title molecule showing the atom-numbering scheme.

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for C atoms; $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^*$ for O and N.

	x	y	z	$U_{\text{iso}}/U_{\text{eq}}$
O(1)	0.1333 (2)	0.6175 (3)	-0.4465 (13)	0.059 (3)
O(2)	0.0963 (2)	1.0774 (3)	-0.0783 (13)	0.058 (3)
O(3)	0.3630 (2)	0.7528 (3)	0.2234 (13)	0.054 (3)
N(1)	0.2920 (2)	0.8976 (3)	0.0772 (12)	0.032 (3)
N(2)	0.1632 (3)	1.0167 (4)	-0.0211 (15)	0.041 (3)
C(1)	0.1524 (3)	0.9219 (4)	-0.1408 (15)	0.030 (1)
C(2)	0.0788 (3)	0.8823 (4)	-0.3181 (18)	0.041 (2)
C(3)	0.0734 (3)	0.7819 (4)	-0.4186 (17)	0.040 (2)
C(4)	0.1406 (4)	0.7099 (5)	-0.3545 (18)	0.040 (2)
C(5)	0.2142 (3)	0.7503 (5)	-0.1840 (16)	0.037 (2)
C(6)	0.2212 (3)	0.8508 (4)	-0.0862 (16)	0.030 (1)
C(7)	0.3572 (3)	0.8484 (4)	0.2245 (17)	0.032 (1)
C(8)	0.4216 (3)	0.9176 (5)	0.3818 (17)	0.044 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O(1)—C(4)	1.234 (7)	O(2)—N(2)	1.361 (6)
O(3)—C(7)	1.222 (7)	N(1)—C(6)	1.424 (7)
N(1)—C(7)	1.341 (7)	N(2)—C(1)	1.301 (7)
C(1)—C(2)	1.446 (8)	C(1)—C(6)	1.462 (7)
C(2)—C(3)	1.339 (8)	C(3)—C(4)	1.453 (8)
C(4)—C(5)	1.440 (8)	C(5)—C(6)	1.339 (8)
C(7)—C(8)	1.482 (8)		
C(7)—N(1)—C(6)	127.4 (5)	C(1)—N(2)—O(2)	112.5 (4)
C(2)—C(1)—N(2)	125.4 (5)	C(6)—C(1)—N(2)	116.1 (5)
C(6)—C(1)—C(2)	118.5 (5)	C(3)—C(2)—C(1)	120.3 (5)
C(4)—C(3)—C(2)	121.4 (5)	C(3)—C(4)—O(1)	119.8 (5)
C(5)—C(4)—O(1)	122.3 (5)	C(5)—C(4)—C(3)	118.0 (5)
C(6)—C(5)—C(4)	121.5 (5)	C(1)—C(6)—N(1)	114.6 (5)
C(5)—C(6)—N(1)	125.1 (5)	C(5)—C(6)—C(1)	120.2 (5)
N(1)—C(7)—O(3)	121.8 (5)	C(8)—C(7)—O(3)	122.8 (5)
C(8)—C(7)—N(1)	115.5 (5)		

The methods of data collection and data processing were similar to those described previously (Adams *et al.*, 1980). The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985) and difference Fourier maps. The positions of the H atoms were found from difference Fourier maps using $\sin \theta < 0.35$. The displacement parameters of the atoms were fixed at 0.07\AA^2 and their parameters were used in the calculation of standard factors but were not refined. During the final stages of structure solution, anisotropic displacement parameters were assigned to the three O and the two N atoms of the molecule. Full-matrix refinement was carried out on the parameters of all non-H atoms, with weights assigned to individual reflections. The structure was refined using *SHELX76* (Sheldrick, 1976). Molecular graphics were prepared using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including inter- and intramolecular contact distances and least-squares-planes data, have been deposited with the IUCr (Reference: HA1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 1189–1193

Two Related Potent Antiviral Compounds: 3-Bromo-*N*-butyl-4-butylamino-1,8-naphthalenedicarboximide (1) and 4-Amino-3-bromo-*N*-butyl-1,8-naphthalenedicarboximide (2)

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(Received 21 December 1993; accepted 26 September 1994)

Abstract

The crystal structures of two brominated derivatives of 4-amino-*N*-butyl-1,8-naphthalenedicarboximide [alternative systematic names: 8-bromo-2-butyl-7-butylamino-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione, $\text{C}_{20}\text{H}_{23}\text{BrN}_2\text{O}_2$, and 7-amino-8-bromo-2-butyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione, $\text{C}_{16}\text{H}_{15}\text{BrN}_2\text{O}_2$] are reported. The *N*-butyl groups, which are common to both compounds, are perpendicular to the plane of the naphthalenedicarboximide ring system. The butylamino