

Crystal structure of (*E*)-2-(*tert*-butylamino)-4-(*tert*-butylimino)naphthalen-1(4*H*)-one

Guy Lamoureux,^{a,b,*} Mónica Alvarado-Rojas^{a,b} and Leslie W. Pineda^{a,c}

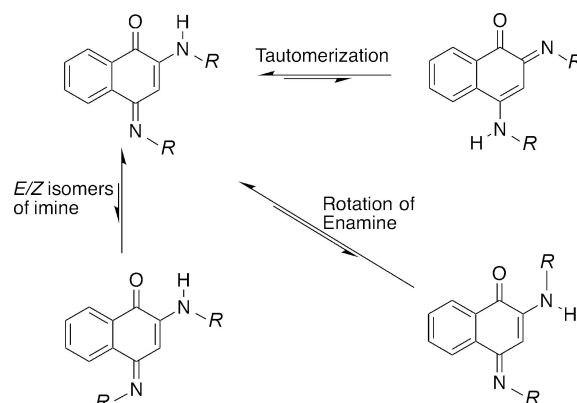
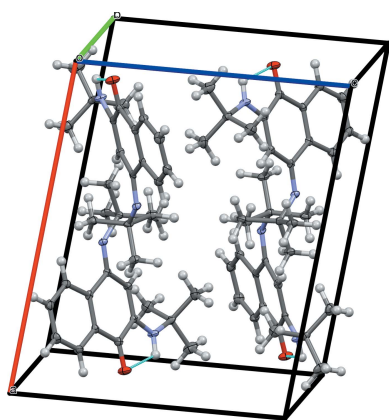
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The title compound, C₁₈H₂₄N₂O, is the first example of a naphthoquinone imine derivative isolated in the 4-imine/2-amine tautomeric form having bulky alkyl substituents at the N atoms. The molecular conformation is stabilized by an intramolecular hydrogen bond between the amine and a carbonyl group and by London attraction between the two *tert*-butyl groups. Only van der Waals interactions were identified in the crystal packing.

1. Chemical context

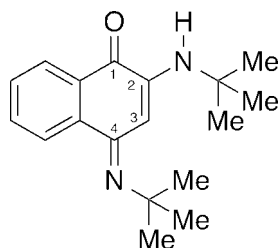
Naphthoquinones (naphthalenediones) form an important part of some pharmacophores in medicinal chemistry (López *et al.*, 2015). During an exploration of antimalarial drugs, Fieser (Fieser & Fieser, 1935) indicated that aminoiminonaphthoquinones, although difficult to form, had interesting medicinal properties. Bullock *et al.* (1969) provided more efficient ways to synthesize a series of these compounds and further investigated their properties as antiprotozoal agents (Bullock *et al.*, 1970).

Naturally occurring compounds with a similar structure to these aminoiminonaphthoquinones are known as hydrolytically stable pigments. Recently, several natural products containing a rigid aminoiminoquinone structure have been isolated and identified: macrophilone A (Zlotkowski *et al.*, 2017), makaluvamines (Radisky *et al.*, 1993), isobatzelline (Stierle & Faulkner, 1991), prianosin (Cheng *et al.*, 1988), epinardin (D'Ambrosio *et al.*, 1996), and discorhabdin (Harayama & Kita, 2005) families. These alkaloid secondary metabolites from marine organisms were found to possess cytotoxic antitumor properties. It has been reported that the aminoiminoquinone system may contribute to the cytotoxic activity (LaBarbera & Skibo, 2013).



Although the 4-imine/2-amine structure was thought to be the most stable, there is evidence for multiple equilibria of these compounds in solution (see reaction scheme). For example, in the case of the methyl derivative ($R = \text{Me}$), NMR evidence at room temperature shows a mixture of tautomers (Bullock *et al.*, 1969). This equilibrium, and in particular the possibility of tautomers, is important since the biological activity of these compounds depends on which tautomer is more stable (Hatfield *et al.*, 2017).

As part of our work on the synthesis and properties of naphthoquinones (Lamoureux *et al.*, 2008), we isolated the title compound as a minor product and predicted that the 4-imine/2-amine tautomeric form would not form because of the presence of bulky R groups. Much to our surprise, (*E*)-2-(*tert*-butylamino)-4-(*tert*-butylimino)naphthalen-1(*4H*)-one is the first compound isolated and structurally characterized of this type with a tertiary alkyl group.



2. Structural commentary

In the molecule of the title compound (Fig. 1), the imine $\text{C}=\text{N}$ bond length at the 4-position [$\text{C8}-\text{N19} = 1.291(3) \text{ \AA}$] is shorter than the enamine $\text{C}-\text{N}$ bond length at the 2-position [$\text{C10}-\text{N20} = 1.353(3) \text{ \AA}$], reflecting the greater double-bond character. The distance between the enamine N-atom and the

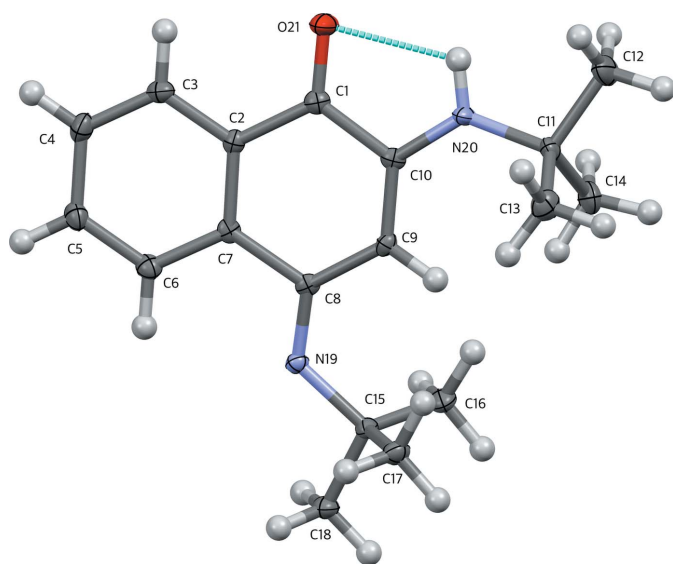


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. The intramolecular hydrogen bond is shown as a dotted line.

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N20}-\text{H20}\cdots\text{O21}$	0.88	2.20	2.629 (3)	109

t-butyl C-atom [$\text{N20}-\text{C11} = 1.476(3) \text{ \AA}$] is slightly shorter than the corresponding bond involving the imino group [$\text{N19}-\text{C15} = 1.485(3) \text{ \AA}$], possibly as a result of steric compression at the imine. However, the bond angles around the two nitrogen atoms [$\text{C8}-\text{N19}-\text{C15} = 124.1(2)^\circ$; $\text{C10}-\text{N20}-\text{C11} = 129.2(2)^\circ$] are similar because of the delocalization of π electrons between the two nitrogen atoms. This system can be considered to be a type of vinylogous amidine (Shriner & Neumann, 1944), both nitrogen atoms having a trigonal-planar geometry. The fused iminoquinone ring adopts a flattened envelope conformation, with the flap atom C8 displaced by $0.112(2) \text{ \AA}$ from the mean plane through C1/C2/C7/C9/C10, and with the $\text{C7}-\text{C8}-\text{C9}$ angle of $116.9(2)^\circ$ showing the largest deviation from the ideal value of 120° .

The title compound possesses an intramolecular hydrogen bond between the imine $\text{N}-\text{H}$ and carbonyl groups (Table 1), forming a ring with $S(5)$ graph-set motif. The distance between the donor H atom and the acceptor carbonyl oxygen atom of 2.20 \AA is shorter than expected as a result of the bulkiness of the *tert*-butyl group (*vide infra*). These *tert*-butyl groups also shield the nitrogen atoms and provide a hydrophobic environment on the side of the naphthalen-1-one ring system. The shortest $\text{C}\cdots\text{C}$ separations between carbon atoms of the *tert*-butyl groups are in the range $4.228(4)-4.825(4) \text{ \AA}$, bringing

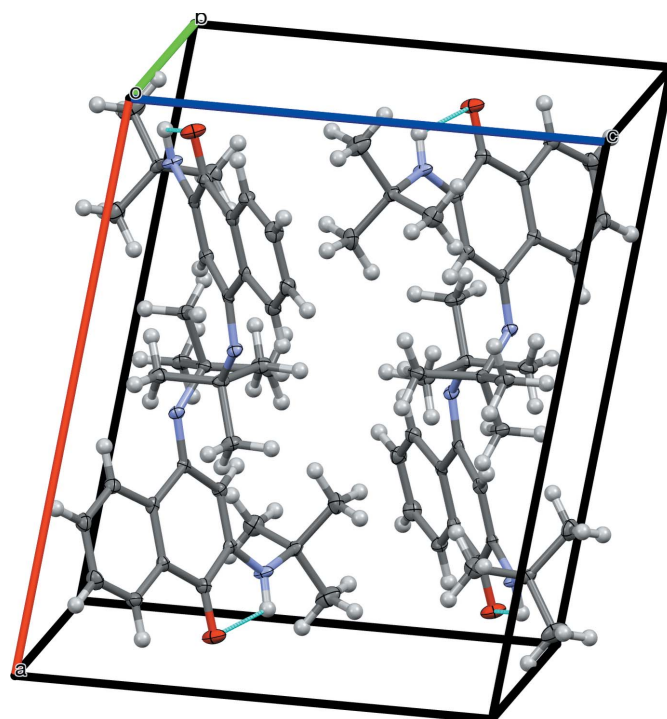


Figure 2
Unit-cell contents of the title compound. Intramolecular hydrogen bonds are shown in turquoise.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₈ H ₂₄ N ₂ O
<i>M_r</i>	284.39
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	14.2792 (18), 9.8936 (13), 11.4978 (13)
β (°)	97.539 (4)
<i>V</i> (Å ³)	1610.3 (3)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.07
Crystal size (mm)	0.50 × 0.50 × 0.10
Data collection	
Diffraction	Bruker D8 Venture
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.688, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	42010, 3732, 1927
<i>R_{int}</i>	0.213
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.652
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.079, 0.143, 1.01
No. of reflections	3732
No. of parameters	196
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.27, -0.29

Computer programs: *APEX3* (Bruker, 2015), *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2006).

them within distance of London attraction (Wagner & Schreiner, 2015).

3. Supramolecular features

In the crystal structure of the title compound (Fig. 2), the *tert*-butyl groups are oriented toward the centre of the unit cell. There are no intermolecular hydrogen bonds, as seen in a similar structure with *n*-butyl groups (see below); the *tert*-butyl groups are shielding the nitrogen atoms and preventing close approach of the supramolecular donors and acceptors. There are no π - π stacking interactions present, the aromatic rings being separated by more than 6 Å.

4. Database survey

A search of the Cambridge Structural Database (Version 5.39, update February 2018; Groom *et al.*, 2016) for the substructure 2-(alkylamino)-4-(alkylimino)naphthalen-1(4*H*)-one yielded three hits. Two of the structures, ESOFID (Schweinfurth *et al.*, 2016) and UDAZEF (Singh *et al.*, 2007) have aromatic amines (aniline or substituted aniline) as the amine moiety. Only one structure, UDAZIJ (Singh *et al.*, 2007), has an aliphatic primary amine (*n*-butylamine) at positions 2 and 4. The structure of UDAZIJ is noteworthy because the intramolecular N—H...O separation of 2.34 Å is much longer than that observed in the title compound, and because in the crystal lattice a dimeric assembly forms, held together by pairs of

intermolecular hydrogen-bonding interactions between the N—H and carbonyl groups of centrosymmetrically -related molecules.

5. Synthesis and crystallization

The synthesis of the title compound was based on a new procedure (complete publication in progress). 192 mg (1.00 mmol) of 4-chloronaphthalene-1,2-dione and 211 μ L (2.00 mmol, 2 equiv.) of *tert*-butylamine were dissolved in *tert*-amyl alcohol (3.0 mL). This solution was stirred at 383 K under a nitrogen atmosphere for 2 h. After being allowed to cool to room temperature, the green-brownish solution (originally yellow) was diluted with saline water (30 mL) and extracted with ethyl acetate (3 × 20 mL). The combined organic layers were dried over Na₂SO₄, filtered, and then concentrated under reduced pressure. The crude brown-dark solid material (249 mg) was separated by silica gel column chromatography using ethyl acetate as eluent to obtain the title compound as secondary product in the form of a dark-brown oily solid (119 mg). The compound was further purified by column chromatography over silica gel with gradient solvent elution [100% dichloromethane (CH₂Cl₂) and then 100% methyl *tert*-butyl ether (C₅H₁₂O)], and the fractions were dried under vacuum to yield 14 mg of the pure product (5% yield) as a yellow oily solid. Part of the purified product was redissolved in methanol with a few drops of water and placed at room temperature for slow evaporation. After several days, yellow crystal plates suitable for X-ray analysis were obtained. M.p. 377–388 K using a Fisher–Johns melting-point apparatus with calibrated thermometer. ¹H NMR (600 MHz, CDCl₃) δ 8.46–8.48 (*dd*, *J* = 7.8, 1.3 Hz, 1 H), 8.09–8.12 (*dd*, *J* = 7.8, 1.3 Hz, 1 H), 7.61–7.64 (*td*, *J* = 7.8, 1.3 Hz, 1 H), 7.48–7.52 (*td*, *J* = 7.8, 1.2 Hz, 1 H), 6.36 (*s*, 1 H), 5.53 (*br s*, 1 H), 1.56 (*s*, 9 H), 1.47 (*s*, 9 H).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms are placed in calculated positions with N—H = 0.88 Å, C—H = 0.95–0.98 Å, and with $U_{iso}(\text{H}) = 1.2U_{eq}(\text{C}, \text{N})$ or $1.5U_{eq}(\text{C})$ for methyl H atoms. A rotating model was used for the methyl groups.

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supporting information

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Computing details

Data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINTE* (Bruker, 2015); data reduction: *SAINTE* (Bruker, 2015); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b).

(*E*)-2-(*tert*-Butylamino)-4-(*tert*-butylimino)naphthalen-1(*4H*)-one

Crystal data

C₁₈H₂₄N₂O

M_r = 284.39

Monoclinic, *P2₁/c*

a = 14.2792 (18) Å

b = 9.8936 (13) Å

c = 11.4978 (13) Å

β = 97.539 (4)°

V = 1610.3 (3) Å³

Z = 4

F(000) = 616

D_x = 1.173 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 68 reflections

θ = 8.7–51.9°

μ = 0.07 mm⁻¹

T = 100 K

Plate, clear light yellow

0.50 × 0.50 × 0.10 mm

Data collection

Bruker D8 Venture
diffractometer

Radiation source: Incoatec Microsource

Mirrors monochromator

Detector resolution: 10.4167 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2015)

T_{min} = 0.688, *T_{max}* = 0.746

42010 measured reflections

3732 independent reflections

1927 reflections with *I* > 2σ(*I*)

R_{int} = 0.213

θ_{max} = 27.6°, θ_{min} = 2.5°

h = -18→18

k = -12→12

l = -14→14

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.079

wR(*F*²) = 0.143

S = 1.01

3732 reflections

196 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

$$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.5846P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O21	0.96284 (12)	0.43511 (18)	0.34929 (16)	0.0228 (5)
N19	0.59245 (14)	0.4688 (2)	0.17726 (17)	0.0128 (5)
N20	0.87654 (14)	0.6571 (2)	0.40230 (18)	0.0158 (5)
H20	0.9346	0.6315	0.4279	0.019*
C1	0.88028 (17)	0.4461 (3)	0.3057 (2)	0.0143 (6)
C2	0.83366 (17)	0.3446 (2)	0.2234 (2)	0.0127 (6)
C3	0.88527 (18)	0.2349 (3)	0.1908 (2)	0.0166 (6)
H3	0.9494	0.2242	0.224	0.02*
C4	0.84444 (18)	0.1415 (3)	0.1110 (2)	0.0198 (7)
H4	0.8799	0.0664	0.0895	0.024*
C5	0.75077 (18)	0.1583 (3)	0.0622 (2)	0.0189 (6)
H5	0.7226	0.0953	0.0058	0.023*
C6	0.69833 (18)	0.2659 (3)	0.0950 (2)	0.0166 (6)
H6	0.6342	0.2755	0.0617	0.02*
C7	0.73876 (17)	0.3605 (2)	0.1764 (2)	0.0116 (6)
C8	0.68184 (17)	0.4750 (2)	0.2136 (2)	0.0114 (6)
C9	0.73246 (17)	0.5786 (2)	0.2858 (2)	0.0126 (6)
H9	0.6993	0.6582	0.302	0.015*
C10	0.82484 (16)	0.5677 (2)	0.3316 (2)	0.0116 (6)
C11	0.84947 (17)	0.7914 (3)	0.4426 (2)	0.0152 (6)
C12	0.93401 (19)	0.8415 (3)	0.5258 (3)	0.0283 (7)
H12A	0.9899	0.8441	0.4845	0.042*
H12B	0.9457	0.7801	0.593	0.042*
H12C	0.921	0.9324	0.5533	0.042*
C13	0.83000 (19)	0.8877 (3)	0.3390 (2)	0.0227 (7)
H13A	0.8865	0.8949	0.2993	0.034*
H13B	0.8137	0.9771	0.3671	0.034*
H13C	0.7773	0.8531	0.2839	0.034*
C14	0.76476 (18)	0.7821 (3)	0.5099 (2)	0.0232 (7)
H14A	0.7087	0.7557	0.4562	0.035*
H14B	0.7538	0.8703	0.5446	0.035*
H14C	0.7772	0.7144	0.5722	0.035*
C15	0.52276 (17)	0.5718 (3)	0.2041 (2)	0.0142 (6)
C16	0.52078 (18)	0.5907 (3)	0.3365 (2)	0.0197 (6)
H16A	0.4676	0.6492	0.3491	0.03*
H16B	0.58	0.6326	0.3717	0.03*

H16C	0.5135	0.5026	0.3731	0.03*
C17	0.53815 (18)	0.7053 (3)	0.1418 (2)	0.0189 (6)
H17A	0.5372	0.6887	0.0576	0.028*
H17B	0.5994	0.7437	0.174	0.028*
H17C	0.4877	0.7689	0.1539	0.028*
C18	0.42656 (17)	0.5160 (3)	0.1510 (2)	0.0187 (6)
H18A	0.4251	0.5067	0.0659	0.028*
H18B	0.3766	0.5783	0.1677	0.028*
H18C	0.4164	0.4274	0.1854	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O21	0.0141 (11)	0.0190 (11)	0.0329 (12)	0.0019 (8)	-0.0057 (9)	-0.0047 (9)
N19	0.0124 (12)	0.0116 (11)	0.0140 (12)	0.0017 (9)	0.0003 (9)	0.0013 (10)
N20	0.0111 (12)	0.0129 (12)	0.0215 (13)	0.0013 (9)	-0.0049 (10)	-0.0031 (10)
C1	0.0143 (15)	0.0139 (15)	0.0150 (14)	-0.0006 (11)	0.0027 (12)	0.0023 (12)
C2	0.0155 (14)	0.0113 (14)	0.0118 (14)	0.0002 (12)	0.0036 (11)	0.0007 (11)
C3	0.0131 (14)	0.0200 (15)	0.0165 (15)	0.0021 (12)	0.0012 (11)	0.0025 (13)
C4	0.0247 (16)	0.0149 (15)	0.0204 (15)	0.0039 (12)	0.0052 (13)	-0.0018 (12)
C5	0.0209 (16)	0.0197 (16)	0.0163 (15)	-0.0018 (13)	0.0036 (12)	-0.0043 (12)
C6	0.0151 (14)	0.0183 (15)	0.0168 (15)	-0.0018 (12)	0.0029 (11)	-0.0007 (13)
C7	0.0157 (14)	0.0085 (14)	0.0108 (14)	-0.0026 (11)	0.0024 (11)	0.0036 (11)
C8	0.0130 (14)	0.0102 (14)	0.0112 (14)	0.0002 (11)	0.0029 (11)	0.0045 (11)
C9	0.0148 (14)	0.0096 (13)	0.0138 (14)	0.0015 (11)	0.0034 (11)	0.0013 (11)
C10	0.0123 (14)	0.0119 (14)	0.0106 (13)	-0.0013 (11)	0.0017 (11)	0.0039 (11)
C11	0.0158 (14)	0.0133 (14)	0.0154 (15)	-0.0005 (12)	-0.0025 (11)	-0.0036 (11)
C12	0.0288 (18)	0.0173 (16)	0.0353 (18)	0.0003 (13)	-0.0085 (14)	-0.0080 (14)
C13	0.0268 (16)	0.0162 (15)	0.0248 (16)	0.0000 (13)	0.0025 (13)	0.0016 (13)
C14	0.0277 (17)	0.0252 (16)	0.0169 (15)	0.0006 (14)	0.0033 (12)	-0.0065 (13)
C15	0.0110 (13)	0.0149 (14)	0.0166 (14)	0.0010 (11)	0.0014 (11)	-0.0029 (12)
C16	0.0134 (14)	0.0232 (16)	0.0227 (16)	0.0018 (12)	0.0032 (12)	-0.0034 (13)
C17	0.0166 (15)	0.0151 (15)	0.0241 (15)	0.0033 (12)	-0.0005 (12)	0.0019 (13)
C18	0.0133 (14)	0.0191 (15)	0.0228 (16)	0.0015 (12)	-0.0013 (12)	-0.0016 (13)

Geometric parameters (Å, °)

O21—C1	1.224 (3)	C11—C13	1.522 (4)
N19—C8	1.291 (3)	C11—C14	1.522 (4)
N19—C15	1.485 (3)	C12—H12A	0.98
N20—C10	1.353 (3)	C12—H12B	0.98
N20—C11	1.476 (3)	C12—H12C	0.98
N20—H20	0.88	C13—H13A	0.98
C1—C2	1.477 (3)	C13—H13B	0.98
C1—C10	1.491 (3)	C13—H13C	0.98
C2—C3	1.391 (3)	C14—H14A	0.98
C2—C7	1.400 (3)	C14—H14B	0.98
C3—C4	1.376 (3)	C14—H14C	0.98

C3—H3	0.95	C15—C18	1.531 (3)
C4—C5	1.391 (3)	C15—C17	1.533 (3)
C4—H4	0.95	C15—C16	1.537 (3)
C5—C6	1.382 (3)	C16—H16A	0.98
C5—H5	0.95	C16—H16B	0.98
C6—C7	1.394 (3)	C16—H16C	0.98
C6—H6	0.95	C17—H17A	0.98
C7—C8	1.489 (3)	C17—H17B	0.98
C8—C9	1.451 (3)	C17—H17C	0.98
C9—C10	1.359 (3)	C18—H18A	0.98
C9—H9	0.95	C18—H18B	0.98
C11—C12	1.521 (3)	C18—H18C	0.98
C8—N19—C15	124.1 (2)	C11—C12—H12B	109.5
C10—N20—C11	129.2 (2)	H12A—C12—H12B	109.5
C10—N20—H20	115.4	C11—C12—H12C	109.5
C11—N20—H20	115.4	H12A—C12—H12C	109.5
O21—C1—C2	122.1 (2)	H12B—C12—H12C	109.5
O21—C1—C10	119.9 (2)	C11—C13—H13A	109.5
C2—C1—C10	118.0 (2)	C11—C13—H13B	109.5
C3—C2—C7	120.2 (2)	H13A—C13—H13B	109.5
C3—C2—C1	119.5 (2)	C11—C13—H13C	109.5
C7—C2—C1	120.2 (2)	H13A—C13—H13C	109.5
C4—C3—C2	120.7 (2)	H13B—C13—H13C	109.5
C4—C3—H3	119.7	C11—C14—H14A	109.5
C2—C3—H3	119.7	C11—C14—H14B	109.5
C3—C4—C5	119.3 (2)	H14A—C14—H14B	109.5
C3—C4—H4	120.3	C11—C14—H14C	109.5
C5—C4—H4	120.3	H14A—C14—H14C	109.5
C6—C5—C4	120.6 (3)	H14B—C14—H14C	109.5
C6—C5—H5	119.7	N19—C15—C18	105.09 (19)
C4—C5—H5	119.7	N19—C15—C17	110.7 (2)
C5—C6—C7	120.6 (2)	C18—C15—C17	107.5 (2)
C5—C6—H6	119.7	N19—C15—C16	112.9 (2)
C7—C6—H6	119.7	C18—C15—C16	107.9 (2)
C6—C7—C2	118.6 (2)	C17—C15—C16	112.3 (2)
C6—C7—C8	120.6 (2)	C15—C16—H16A	109.5
C2—C7—C8	120.8 (2)	C15—C16—H16B	109.5
N19—C8—C9	128.0 (2)	H16A—C16—H16B	109.5
N19—C8—C7	115.1 (2)	C15—C16—H16C	109.5
C9—C8—C7	116.9 (2)	H16A—C16—H16C	109.5
C10—C9—C8	123.4 (2)	H16B—C16—H16C	109.5
C10—C9—H9	118.3	C15—C17—H17A	109.5
C8—C9—H9	118.3	C15—C17—H17B	109.5
N20—C10—C9	127.3 (2)	H17A—C17—H17B	109.5
N20—C10—C1	112.7 (2)	C15—C17—H17C	109.5
C9—C10—C1	120.0 (2)	H17A—C17—H17C	109.5
N20—C11—C12	105.8 (2)	H17B—C17—H17C	109.5

N20—C11—C13	110.3 (2)	C15—C18—H18A	109.5
C12—C11—C13	109.7 (2)	C15—C18—H18B	109.5
N20—C11—C14	111.3 (2)	H18A—C18—H18B	109.5
C12—C11—C14	108.5 (2)	C15—C18—H18C	109.5
C13—C11—C14	111.1 (2)	H18A—C18—H18C	109.5
C11—C12—H12A	109.5	H18B—C18—H18C	109.5
O21—C1—C2—C3	-2.0 (4)	C6—C7—C8—C9	-171.7 (2)
C10—C1—C2—C3	175.5 (2)	C2—C7—C8—C9	9.0 (3)
O21—C1—C2—C7	179.4 (2)	N19—C8—C9—C10	171.5 (2)
C10—C1—C2—C7	-3.1 (3)	C7—C8—C9—C10	-8.2 (3)
C7—C2—C3—C4	0.9 (4)	C11—N20—C10—C9	-4.0 (4)
C1—C2—C3—C4	-177.7 (2)	C11—N20—C10—C1	176.4 (2)
C2—C3—C4—C5	0.4 (4)	C8—C9—C10—N20	-177.9 (2)
C3—C4—C5—C6	-1.3 (4)	C8—C9—C10—C1	1.7 (4)
C4—C5—C6—C7	0.9 (4)	O21—C1—C10—N20	1.4 (3)
C5—C6—C7—C2	0.5 (4)	C2—C1—C10—N20	-176.2 (2)
C5—C6—C7—C8	-178.8 (2)	O21—C1—C10—C9	-178.3 (2)
C3—C2—C7—C6	-1.4 (4)	C2—C1—C10—C9	4.1 (3)
C1—C2—C7—C6	177.2 (2)	C10—N20—C11—C12	176.7 (2)
C3—C2—C7—C8	177.9 (2)	C10—N20—C11—C13	-64.8 (3)
C1—C2—C7—C8	-3.5 (3)	C10—N20—C11—C14	59.0 (3)
C15—N19—C8—C9	0.2 (4)	C8—N19—C15—C18	-175.8 (2)
C15—N19—C8—C7	179.9 (2)	C8—N19—C15—C17	68.5 (3)
C6—C7—C8—N19	8.6 (3)	C8—N19—C15—C16	-58.5 (3)
C2—C7—C8—N19	-170.7 (2)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N20—H20...O21	0.88	2.20	2.629 (3)	109