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rac-5-Acetyl-6-(4-nitrophenyl)-6,7-dihydro-5*H*-1,3-dioxolo[4,5-*g*]quinoline-8-one: chains of rings built from C—H···O and C—H···π(arene) hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.086
Data-to-parameter ratio = 7.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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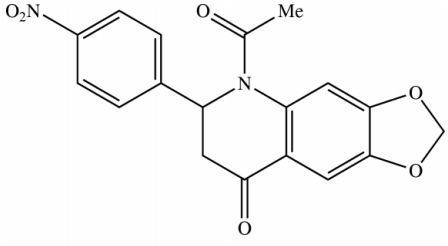
Molecules of the title compound, $C_{18}H_{14}N_2O_6$, are linked into chains by a combination of one C—H···O and one C—H···π(arene) hydrogen bond, augmented by a dipolar carbonyl–carbonyl interaction.

Comment

The title compound, (I), was prepared as an intermediate in the preparation of new bis-amides derived from tetrahydroquinolones, for use as model compounds for DNA intercalating agents (Gamage *et al.*, 1999; Chacón-García & Martínez, 2001; Deady *et al.*, 2001).



(I)

Compound (I) (Fig. 1) crystallizes in the polar space group $Pna2_1$. The molecule contains a stereogenic centre at C6, and the selected reference molecule is of *S* configuration; however, the space group accommodates equal numbers of *R* and *S* enantiomers.

For the heterocyclic ring (N5/C4A/C8A/C8/C7/C6), the ring-puckering parameters (Cremer & Pople, 1975) for this atom sequence, θ = 124.7 (3)° and φ = 120.3 (4)°, indicate an almost pure envelope form (Evans & Boeyens, 1989). The inter-bond angles at N5 (Table 1) indicate that this atom has

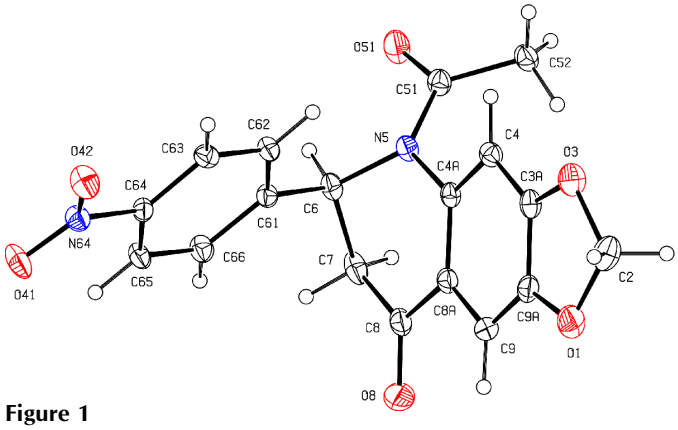


Figure 1
The *S* enantiomer of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

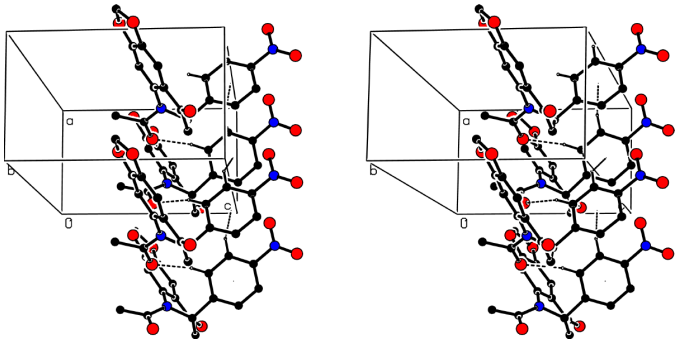


Figure 2
Stereoview of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded chain along [100].

effectively planar coordination, as expected for amidic N, but, unexpectedly, the nitrophenyl substituent at C6 occupies an axial site. Within the fused-ring system, the bond lengths (Table 1) show several unexpected features. In particular, the bonds C3A—C4 and C9—C9A are significantly shorter than the other bonds in the carbocyclic aromatic ring, and the bond C4A—N5 is very long for its type: the mean value for bonds of this type (Allen *et al.*, 1987) is 1.371 Å. It may also be noted that the two carbonyl C═O distances are identical, despite their different local environments.

The molecules of (I) are linked into chains by a combination of C—H···O and C—H···π(arene) hydrogen bonds, augmented by a dipolar carbonyl–carbonyl interaction. Atoms C62 and C63 in the molecule at (x, y, z) act as hydrogen-bond donors, respectively, to amidic atom O51 and to the ring C61—C66, both in the molecule at ($\frac{1}{2}+x$, $\frac{3}{2}-y$, z), and propagation of these interactions produces a chain of rings running parallel to the [100] direction and generated by the *a* glide plane at y = 0.75 (Fig. 2). In addition, carbonyl atom O51 in the molecule at (x, y, z) forms a short contact with the carbonyl atom C51 in the molecule at ($x-\frac{1}{2}$, $\frac{3}{2}-y$, z): the O···C distance is 3.001 (3) Å, the C—O···C angle is 150 (4)° and the O···C—O angle is 87.4 (2)°, indicative of a type I interaction (Allen *et al.*, 1998), which reinforces the [100] chain.

Two antiparallel chains of this type pass through each unit cell, generated by the *a* glide planes at y = 0.25 and 0.75, but there are no direction-specific interactions between adjacent chains. In particular, there are no π–π stacking interaction and no C—H···O hydrogen bonds involving the nitro O atoms.

Experimental

A mixture of 6-(4-nitrophenyl)-6,7-dihydro-5*H*-[1,3]dioxolo[4,5-*g*]-quinolin-8-one (250 mg, 0.71 mmol) (Donnelly & Farell, 1990) and acetic anhydride (3 ml) was heated at 353 K for 90 min. After reaction was complete (as shown by thin-layer chromatography), the solvent was removed under vacuum and the resulting solid was washed with water and then purified by column chromatography on silica gel with chloroform–ethyl acetate (9:1 *v*/*v*) as eluant, to give a pale yellow solid (60% yield, m.p. 483 K). MS (70 eV): *m*/*e* (%) 354 (40, M^+), 312 (86), 190 (39), 43 (100). Crystals suitable for single-crystal X-ray diffraction were grown from a solution in 96% aqueous ethanol.

Crystal data

$C_{18}H_{14}N_2O_6$
M_r = 354.31
Orthorhombic, $Pna2_1$
a = 6.5253 (2) Å
b = 21.4286 (5) Å
c = 10.8198 (9) Å
V = 1512.91 (14) Å^3
Z = 4
D_x = 1.556 Mg m^{-3}
Mo Kα radiation
Cell parameters from 1952 reflections
θ = 3.3–27.1°
μ = 0.12 mm^{-1}
T = 120 (2) K
Needle, colourless
0.30 × 0.10 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.956, T_{max} = 0.989
15335 measured reflections
1762 independent reflections
1360 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 27.1°
$h = -8 \rightarrow 7$
$k = -27 \rightarrow 27$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.087
S = 1.06
1762 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.0405P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0110 (18)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.431 (4)	C8—C8A	1.480 (4)
C2—O3	1.440 (4)	C8A—C9	1.407 (4)
O3—C3A	1.367 (3)	C9—C9A	1.352 (4)
C3A—C4	1.369 (4)	C9A—O1	1.381 (3)
C4—C4A	1.400 (4)	C3A—C9A	1.385 (4)
C4A—N5	1.425 (3)	C4A—C8A	1.403 (4)
N5—C6	1.472 (4)	N5—C51	1.388 (4)
C6—C7	1.527 (4)	C51—O51	1.223 (3)
C7—C8	1.506 (4)	C8—O8	1.223 (4)
C51—N5—C4A	125.0 (2)	C4A—N5—C6	115.1 (2)
C51—N5—C6	118.1 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C62—H62···O51^{i}	0.95	2.48	3.358 (4)	153
C63—H63···$Cg1^{i}$	0.95	2.98	3.693 (3)	132

Symmetry code: (i) $\frac{1}{2}+x$, $\frac{3}{2}-y$, z. $Cg1$ is the centroid of ring C61–C66

All H atoms were located in difference maps and subsequently treated as riding atoms, with distances C—H = 0.95 (aromatic), 0.98 (methyl), 0.99 (CH_2) or 1.00 Å (aliphatic CH), and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl group. The value of the Flack parameter [0.2 (13); Flack, 1983] was indeterminate (Flack & Bernardinelli, 2000), and hence the correct orientation of the structure relative to the polar axis direction could not be established (Jones, 1986). Accordingly, the Friedel-equivalent reflections were merged prior to the final refinement.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS*97 (Sheldrick, 1997);

program(s) used to refine structure: *OSCAIL* and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff for all their help and advice. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work. JC thanks the Consejería de Educación y Ciencia (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support; RA thanks Fundación para la Promoción de la Investigación y la Tecnología (Banco de la República) and Universidad del Valle for financial support. PC thanks COLCIENCIAS for a doctoral fellowship.

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Cell parameters from 1952 reflections
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1762 independent reflections
1360 reflections with $I > 2\sigma(I)$
$R_{int} = 0.082$
$\theta_{max} = 27.1°$, $\theta_{min} = 3.3°$
$h = -8 \rightarrow 7$
$k = -27 \rightarrow 27$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.087$
$S = 1.06$
1762 reflections
237 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
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.0405P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0110 (18)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5209 (4)	0.45991 (10)	0.5921 (2)	0.0450 (6)

C2	0.6518 (5)	0.49285 (15)	0.5084 (4)	0.0429 (8)
O3	0.5919 (3)	0.55753 (10)	0.5100 (2)	0.0383 (6)
C3A	0.4110 (4)	0.55944 (14)	0.5736 (3)	0.0278 (7)
C4	0.2903 (4)	0.61069 (13)	0.5940 (3)	0.0265 (6)
C4A	0.1148 (4)	0.60062 (12)	0.6658 (2)	0.0237 (6)
N5	−0.0191 (3)	0.65140 (12)	0.6922 (2)	0.0262 (5)
C51	−0.1005 (4)	0.69175 (13)	0.6047 (3)	0.0291 (7)
O51	−0.2149 (3)	0.73380 (10)	0.63757 (19)	0.0426 (6)
C52	−0.0541 (5)	0.68171 (14)	0.4710 (3)	0.0313 (7)
C6	−0.1136 (4)	0.65011 (13)	0.8158 (3)	0.0278 (6)
C61	0.0432 (4)	0.65684 (13)	0.9186 (3)	0.0249 (6)
C62	0.2377 (4)	0.68212 (13)	0.8979 (3)	0.0268 (6)
C63	0.3740 (4)	0.69024 (13)	0.9951 (3)	0.0273 (6)
C64	0.3144 (4)	0.67328 (12)	1.1127 (2)	0.0246 (6)
N64	0.4578 (4)	0.68102 (11)	1.2154 (2)	0.0298 (6)
O41	0.3963 (3)	0.67045 (11)	1.3207 (2)	0.0451 (6)
O42	0.6331 (3)	0.69795 (11)	1.1920 (2)	0.0410 (6)
C65	0.1243 (4)	0.64875 (13)	1.1358 (3)	0.0292 (7)
C66	−0.0106 (4)	0.64056 (13)	1.0388 (3)	0.0306 (7)
C7	−0.2388 (4)	0.59007 (15)	0.8248 (3)	0.0358 (7)
C8	−0.1070 (5)	0.53365 (14)	0.8006 (3)	0.0347 (7)
O8	−0.1483 (4)	0.48271 (11)	0.8451 (2)	0.0578 (8)
C8A	0.0733 (4)	0.54239 (13)	0.7194 (3)	0.0278 (6)
C9	0.2061 (5)	0.49165 (13)	0.6998 (3)	0.0327 (7)
C9A	0.3695 (4)	0.50171 (13)	0.6255 (3)	0.0307 (7)
H2A	0.6375	0.4756	0.4239	0.051*
H2B	0.7966	0.4886	0.5344	0.051*
H4	0.3235	0.6506	0.5614	0.032*
H52A	−0.1760	0.6913	0.4216	0.047*
H52B	−0.0145	0.6381	0.4577	0.047*
H52C	0.0586	0.7092	0.4460	0.047*
H6	−0.2106	0.6861	0.8215	0.033*
H62	0.2769	0.6939	0.8166	0.032*
H63	0.5064	0.7072	0.9807	0.033*
H65	0.0859	0.6375	1.2175	0.035*
H66	−0.1425	0.6235	1.0544	0.037*
H7A	−0.2999	0.5868	0.9083	0.043*
H7B	−0.3519	0.5914	0.7639	0.043*
H9	0.1822	0.4522	0.7369	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0547 (14)	0.0436 (13)	0.0369 (12)	0.0229 (12)	0.0026 (11)	−0.0028 (11)
C2	0.0360 (17)	0.046 (2)	0.047 (2)	0.0074 (15)	0.0007 (16)	−0.0128 (17)
O3	0.0296 (11)	0.0476 (13)	0.0378 (13)	0.0056 (9)	0.0066 (10)	−0.0047 (10)
C3A	0.0234 (15)	0.0373 (17)	0.0226 (16)	0.0023 (12)	−0.0028 (11)	−0.0051 (12)
C4	0.0256 (14)	0.0304 (15)	0.0235 (15)	−0.0023 (12)	−0.0032 (11)	0.0008 (12)

C4A	0.0238 (14)	0.0286 (14)	0.0188 (14)	0.0023 (12)	−0.0033 (11)	−0.0020 (11)
N5	0.0261 (12)	0.0312 (12)	0.0212 (12)	0.0050 (10)	0.0008 (10)	0.0007 (10)
C51	0.0289 (16)	0.0326 (16)	0.0258 (16)	0.0017 (13)	−0.0048 (12)	−0.0009 (13)
O51	0.0499 (13)	0.0473 (13)	0.0306 (12)	0.0214 (11)	−0.0031 (11)	−0.0018 (11)
C52	0.0350 (18)	0.0357 (17)	0.0230 (15)	0.0031 (13)	−0.0029 (12)	0.0026 (13)
C6	0.0272 (15)	0.0352 (15)	0.0211 (14)	0.0055 (12)	0.0016 (12)	−0.0010 (13)
C61	0.0288 (16)	0.0245 (14)	0.0213 (15)	0.0028 (12)	0.0012 (12)	−0.0011 (12)
C62	0.0303 (16)	0.0296 (15)	0.0206 (13)	0.0000 (12)	0.0026 (11)	0.0010 (12)
C63	0.0267 (15)	0.0276 (15)	0.0277 (16)	0.0009 (12)	0.0026 (12)	0.0029 (12)
C64	0.0291 (15)	0.0248 (14)	0.0199 (15)	0.0015 (11)	−0.0017 (12)	−0.0023 (12)
N64	0.0297 (14)	0.0332 (14)	0.0265 (14)	−0.0014 (11)	−0.0012 (11)	0.0013 (11)
O41	0.0463 (14)	0.0687 (16)	0.0204 (12)	−0.0099 (11)	−0.0024 (11)	0.0060 (11)
O42	0.0290 (12)	0.0580 (14)	0.0360 (13)	−0.0080 (10)	−0.0031 (10)	0.0002 (11)
C65	0.0322 (16)	0.0363 (16)	0.0192 (15)	−0.0025 (13)	0.0036 (13)	0.0019 (13)
C66	0.0296 (16)	0.0363 (17)	0.0259 (15)	−0.0023 (12)	0.0028 (12)	−0.0013 (13)
C7	0.0278 (15)	0.058 (2)	0.0216 (14)	−0.0085 (14)	0.0020 (13)	−0.0019 (15)
C8	0.0430 (19)	0.0398 (18)	0.0214 (16)	−0.0138 (14)	0.0013 (13)	−0.0032 (14)
O8	0.0848 (19)	0.0412 (14)	0.0474 (16)	−0.0206 (13)	0.0283 (14)	−0.0019 (13)
C8A	0.0324 (16)	0.0309 (16)	0.0201 (14)	−0.0057 (12)	−0.0012 (12)	−0.0024 (12)
C9	0.0469 (18)	0.0242 (15)	0.0270 (17)	−0.0016 (13)	0.0005 (15)	−0.0001 (13)
C9A	0.0389 (17)	0.0291 (15)	0.0242 (17)	0.0080 (13)	−0.0068 (14)	−0.0056 (13)

Geometric parameters (Å, º)

O1—C2	1.431 (4)	C9—C9A	1.352 (4)
C2—O3	1.440 (4)	C9—H9	0.95
C2—H2A	0.99	C9A—O1	1.381 (3)
C2—H2B	0.99	C3A—C9A	1.385 (4)
O3—C3A	1.367 (3)	C4A—C8A	1.403 (4)
C3A—C4	1.369 (4)	N5—C51	1.388 (4)
C4—C4A	1.400 (4)	C51—O51	1.223 (3)
C4—H4	0.95	C8—O8	1.223 (4)
C4A—N5	1.425 (3)	C61—C66	1.391 (4)
N5—C6	1.472 (4)	C61—C62	1.398 (4)
C51—C52	1.493 (4)	C62—C63	1.388 (4)
C52—H52A	0.98	C62—H62	0.95
C52—H52B	0.98	C63—C64	1.379 (4)
C52—H52C	0.98	C63—H63	0.95
C6—C61	1.519 (4)	C64—C65	1.370 (4)
C6—C7	1.527 (4)	C64—N64	1.463 (4)
C6—H6	1.00	N64—O42	1.226 (3)
C7—C8	1.506 (4)	N64—O41	1.229 (3)
C7—H7A	0.99	C65—C66	1.381 (4)
C7—H7B	0.99	C65—H65	0.95
C8—C8A	1.480 (4)	C66—H66	0.95
C8A—C9	1.407 (4)		
C9A—O1—C2	105.8 (2)	C62—C61—C6	122.1 (2)

O1—C2—O3	107.8 (2)	C63—C62—C61	120.6 (3)
O1—C2—H2A	110.2	C63—C62—H62	119.7
O3—C2—H2A	110.2	C61—C62—H62	119.7
O1—C2—H2B	110.2	C64—C63—C62	119.0 (3)
O3—C2—H2B	110.2	C64—C63—H63	120.5
H2A—C2—H2B	108.5	C62—C63—H63	120.5
C3A—O3—C2	105.6 (2)	C65—C64—C63	121.7 (3)
O3—C3A—C4	127.0 (3)	C65—C64—N64	118.9 (2)
O3—C3A—C9A	110.2 (2)	C63—C64—N64	119.4 (2)
C4—C3A—C9A	122.6 (3)	O42—N64—O41	123.4 (3)
C3A—C4—C4A	115.8 (3)	O42—N64—C64	118.3 (2)
C3A—C4—H4	122.1	O41—N64—C64	118.3 (2)
C4A—C4—H4	122.1	C64—C65—C66	119.1 (2)
C4—C4A—C8A	121.6 (2)	C64—C65—H65	120.4
C4—C4A—N5	119.7 (2)	C66—C65—H65	120.4
C8A—C4A—N5	118.5 (2)	C65—C66—C61	121.2 (3)
C51—N5—C4A	125.0 (2)	C65—C66—H66	119.4
C51—N5—C6	118.1 (2)	C61—C66—H66	119.4
C4A—N5—C6	115.1 (2)	C8—C7—C6	111.1 (2)
O51—C51—N5	119.6 (3)	C8—C7—H7A	109.4
O51—C51—C52	120.8 (3)	C6—C7—H7A	109.4
N5—C51—C52	119.6 (2)	C8—C7—H7B	109.4
C51—C52—H52A	109.5	C6—C7—H7B	109.4
C51—C52—H52B	109.5	H7A—C7—H7B	108.0
H52A—C52—H52B	109.5	O8—C8—C8A	121.4 (3)
C51—C52—H52C	109.5	O8—C8—C7	121.5 (3)
H52A—C52—H52C	109.5	C8A—C8—C7	117.1 (2)
H52B—C52—H52C	109.5	C4A—C8A—C9	120.4 (3)
N5—C6—C61	112.4 (2)	C4A—C8A—C8	120.8 (3)
N5—C6—C7	107.3 (2)	C9—C8A—C8	118.8 (3)
C61—C6—C7	113.2 (2)	C9A—C9—C8A	116.9 (3)
N5—C6—H6	107.9	C9A—C9—H9	121.6
C61—C6—H6	107.9	C8A—C9—H9	121.6
C7—C6—H6	107.9	C9—C9A—O1	128.0 (3)
C66—C61—C62	118.4 (3)	C9—C9A—C3A	122.5 (3)
C66—C61—C6	119.4 (2)	O1—C9A—C3A	109.5 (3)

C9A—O1—C2—O3	9.3 (3)	C63—C64—N64—O42	−5.6 (4)
O1—C2—O3—C3A	−10.7 (3)	C65—C64—N64—O41	−6.4 (4)
C2—O3—C3A—C4	−176.6 (3)	C63—C64—N64—O41	174.2 (3)
C2—O3—C3A—C9A	8.0 (3)	C63—C64—C65—C66	0.3 (4)
O3—C3A—C4—C4A	−178.0 (3)	N64—C64—C65—C66	−179.1 (2)
C9A—C3A—C4—C4A	−3.1 (4)	C64—C65—C66—C61	−0.1 (4)
C3A—C4—C4A—C8A	3.8 (4)	C62—C61—C66—C65	−0.3 (4)
C3A—C4—C4A—N5	179.6 (2)	C6—C61—C66—C65	−176.9 (3)
C4—C4A—N5—C51	51.2 (4)	N5—C6—C7—C8	56.7 (3)
C8A—C4A—N5—C51	−132.8 (3)	C61—C6—C7—C8	−67.9 (3)
C4—C4A—N5—C6	−144.3 (3)	C6—C7—C8—O8	152.7 (3)

C8A—C4A—N5—C6	31.7 (3)	C6—C7—C8—C8A	−28.0 (4)
C4A—N5—C51—O51	179.7 (3)	C4—C4A—C8A—C9	−1.5 (4)
C6—N5—C51—O51	15.7 (4)	N5—C4A—C8A—C9	−177.4 (3)
C4A—N5—C51—C52	2.2 (4)	C4—C4A—C8A—C8	176.6 (3)
C6—N5—C51—C52	−161.9 (3)	N5—C4A—C8A—C8	0.7 (4)
C51—N5—C6—C61	−129.5 (3)	O8—C8—C8A—C4A	177.9 (3)
C4A—N5—C6—C61	64.8 (3)	C7—C8—C8A—C4A	−1.5 (4)
C51—N5—C6—C7	105.4 (3)	O8—C8—C8A—C9	−3.9 (4)
C4A—N5—C6—C7	−60.2 (3)	C7—C8—C8A—C9	176.7 (3)
N5—C6—C61—C66	−163.4 (2)	C4A—C8A—C9—C9A	−1.5 (4)
C7—C6—C61—C66	−41.6 (4)	C8—C8A—C9—C9A	−179.7 (3)
N5—C6—C61—C62	20.1 (4)	C8A—C9—C9A—O1	179.9 (3)
C7—C6—C61—C62	141.9 (3)	C8A—C9—C9A—C3A	2.2 (4)
C66—C61—C62—C63	0.5 (4)	C2—O1—C9A—C9	177.5 (3)
C6—C61—C62—C63	177.0 (3)	C2—O1—C9A—C3A	−4.5 (3)
C61—C62—C63—C64	−0.3 (4)	O3—C3A—C9A—C9	175.8 (3)
C62—C63—C64—C65	−0.1 (4)	C4—C3A—C9A—C9	0.2 (4)
C62—C63—C64—N64	179.3 (2)	O3—C3A—C9A—O1	−2.3 (3)
C65—C64—N64—O42	173.8 (2)	C4—C3A—C9A—O1	−177.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C62—H62···O51^{i}	0.95	2.48	3.358 (4)	153
C63—H63···*Cg*1^{i}	0.95	2.98	3.693 (3)	132

Symmetry code: (i) *x*+1/2, −*y*+3/2, *z*.