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3-*tert*-Butyl-7,7-dimethyl-1-phenyl-5,6,7,8-tetrahydroimidazo[3,4-*b*]-quinolin-5-one and 2,8,8-trimethyl-5-phenyl-6,7,8,9-tetrahydroimidazo-[2,3-*a*]quinolin-6-one: chains generated by C—H···N hydrogen bonds

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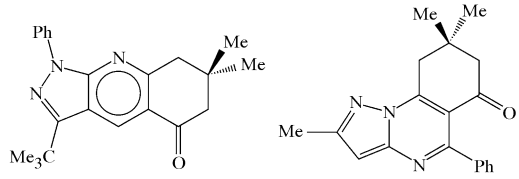
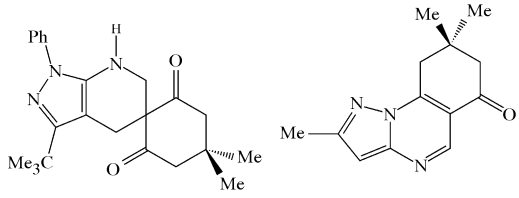
In both 3-*tert*-butyl-7,7-dimethyl-1-phenyl-5,6,7,8-tetrahydroimidazo[3,4-*b*]quinolin-5-one, $C_{22}H_{25}N_3O$, (I), and 2,8,8-trimethyl-5-phenyl-6,7,8,9-tetrahydroimidazo[2,3-*a*]quinolin-6-one, $C_{19}H_{19}N_3O$, (II), the heterobicyclic portions of the molecules are planar, with naphthalene-type delocalization in (II), while the carbocyclic ring in each compound adopts an envelope conformation. In both (I) and (II), the molecules are linked weakly into chains by a single C—H···N hydrogen bond.

Comment

As part of a program for the synthesis of fused pyrazole derivatives (Quiroga *et al.*, 1998, 2001; Cannon *et al.*, 2001*a*,*b*; Low *et al.*, 2001), we have been investigating three-component cyclocondensation reactions induced by microwave irradiation. We report here the molecular and supramolecular structures of two compounds, (I) and (II), obtained from condensation reactions between a substituted aminopyrazole, 5,5-dimethylcyclohexane-1,3-dione (dimedone) and a simple carbonyl compound or its equivalent. Thus, from the reaction involving 5-amino-3-*tert*-butyl-1-phenylpyrazole and formaldehyde, we have now obtained 3-*tert*-butyl-7,7-dimethyl-1-phenyl-5,6,7,8-tetrahydroimidazo[3,4-*b*]quinolin-5-one, (I), in which a single formaldehyde unit has been utilized in the construction of the fused ring system. When two such units are incorporated, spiro compound (III) results (Low *et al.*, 2004). When 5-amino-3-methyl-1*H*-pyrazole is used in combination with orthobenzoic acid trimethyl ester, the product is (II), analogous to the compound, (IV), formed from this pyrazole in the presence of formaldehyde (Low *et al.*, 2004).





In both (I) (Fig. 1) and (II) (Fig. 2), the heterobicyclic portions of the fused ring systems are planar, but the carbocyclic rings are puckered. The ring-puckering parameters (Cremer & Pople, 1975) for (I) [θ = 127.4 (3)° and φ = 353.8 (3)° for the atom sequence C4a—C5—C6—C7—C8—C8a] and (II) [θ = 65.2 (2)° and φ = 174.3 (3)° for the atom

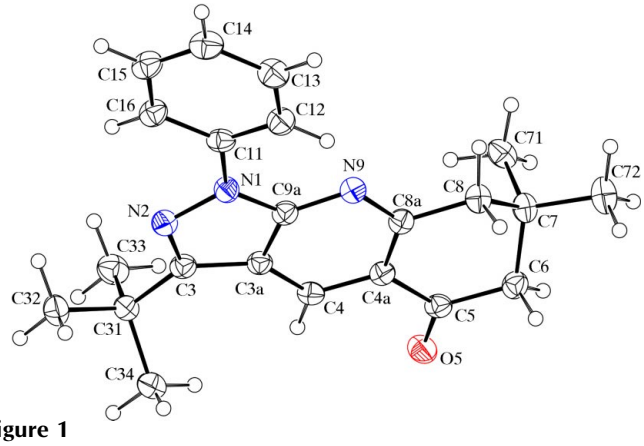


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

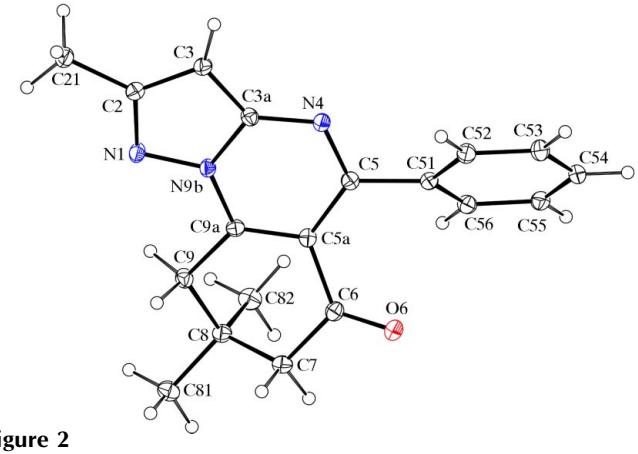


Figure 2
The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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sequence C5a—C6—C7—C8—C9—C9a] indicate envelope conformations for both these rings (Evans & Boeyens, 1989), consistent with the enforced coplanarity of atoms C5, C4a, C8a and C8 in (I), and of atoms C6, C5a, C9a and C9 in (II).

In (I), the C3a—C4 and C4—C4a bonds are of very similar length (Table 1), as are the C8a—N9 and N9—C9a bonds, consistent with aromatic delocalization within the central ring of (I). The formally single C3a—N4 and C9a—N9b bonds in (II) (Table 3) are only slightly longer than the formal double bond N1═C2, although each is significantly longer than the cross-ring C3a—N9b bond, also formally a single bond. The lengths of the C2—C3 and C3═C3a bonds, formally single and double, respectively, differ by less than 0.03 Å. These observations suggest that this heterocyclic system exhibits a degree of naphthalene-type delocalization, involving a peripheral system of ten π electrons but with only modest participation by the cross-ring bond (Glidewell & Lloyd, 1984).

In each of (I) and (II), the molecules are linked weakly into chains by means of a single C—H···N hydrogen bond (Tables 2 and 4); the structure of neither compound exhibits any C—H···π(arene) hydrogen bonds or aromatic π–π stacking interactions. In (I), atom C6 in the molecule at (x, y, z) acts as a hydrogen-bond donor, *via* atom H6*B*, to pyridine ring atom N9 in the molecule at ($1+x$, y, z), so generating by translation a $C(6)$ chain (Bernstein *et al.*, 1995) running parallel to the [100] direction (Fig. 3). In (II), aryl atom C54 in the molecule at (x, y, z) acts as a hydrogen-bond donor to pyrazole-ring atom N1 in the molecule at ($1+x$, $\frac{3}{2}-y$, $-\frac{1}{2}+z$),

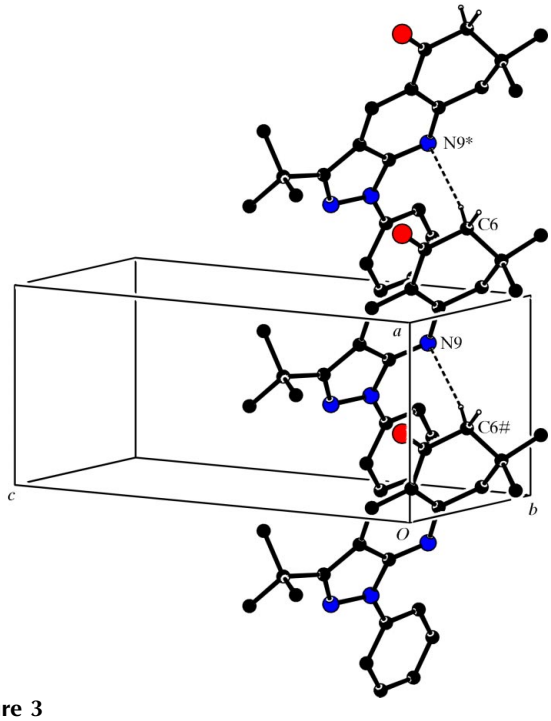

Figure 3
Part of the crystal structure of (I), showing the formation of a $C(6)$ chain along [100]. For clarity, H atoms bonded to C atoms not participating in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($1+x$, y, z) and ($-1+x$, y, z), respectively.

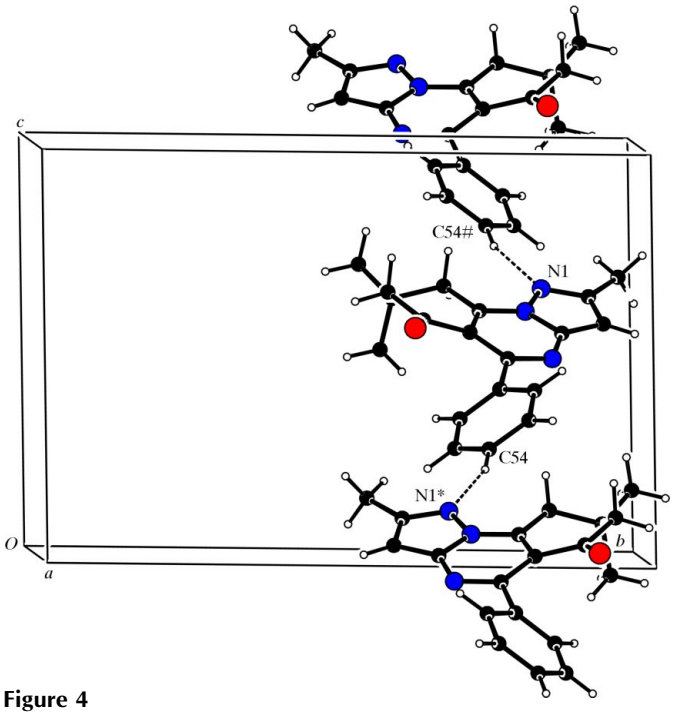

Figure 4
Part of the crystal structure of (II), showing the formation of a $C(10)$ chain along [$20\bar{1}$]. For clarity, H atoms bonded to C atoms not participating in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($1+x$, $\frac{3}{2}-y$, $-\frac{1}{2}+z$) and ($-1+x$, $\frac{3}{2}-y$, $\frac{1}{2}+z$), respectively.

so producing a zigzag $C(10)$ chain running parallel to the [$20\bar{1}$] direction and generated by the *c*-glide plane at $y=\frac{3}{4}$ (Fig. 4).

The constitutions of (II) and (IV) differ only by the presence of the phenyl substituent in (II); however, this difference profoundly influences the differences in the supramolecular structures of these compounds. In (IV), the C—H bond that is replaced by the C–phenyl bond in (II) acts as the sole hydrogen-bond donor, forming, by means of paired C—H···N hydrogen bonds, a centrosymmetric $R_2^2(6)$ dimer. Dimers of this type are then linked into chains by a single π–π stacking interaction (Low *et al.*, 2004).

Experimental

For the synthesis of (I), a mixture of 5-amino-3-*tert*-butyl-1-phenylpyrazole (1 mmol), dimedone (1 mmol) and formaldehyde (3 mmol) was placed in Pyrex-glass open vessels and irradiated in a domestic microwave oven for 4 min (at 600 W). The reaction mixture was extracted with ethanol and the product, (I), was isolated by column chromatography on silica gel, using $CHCl_3$ as eluant, and crystallized from ethanol, yielding crystals suitable for single-crystal X-ray diffraction (m.p. 413 K; yield 41%). Analysis found: C 75.5, H 7.3, N 12.1%; $C_{22}H_{25}N_3O$ requires: C 76.0, H 7.3, N 12.1%. For the synthesis of (II), an equimolar mixture of 5-amino-3-methyl-1*H*-pyrazole, dimedone and orthobenzoic acid trimethyl ester (1 mmol of each) was placed in Pyrex-glass open vessels and irradiated in a domestic microwave oven for 2 min (at 600 W). The reaction mixture was extracted with ethanol and the product, (II), was crystallized from ethanol, producing crystals suitable for single-crystal X-ray diffraction (m.p. 533 K; yield 55%). MS EI (70 eV) *m*/*z* (%): 306 (23), 305 (100, M^+), 304 (60), 291 (13), 290 (54), 250 (14), 249 (73), 248 (14), 220

(13), 153 (11), 127 (16), 126 (10), 77 (29), 66 (10), 55 (10), 53 (16), 52 (13), 351 (17), 42 (20), 41 (34), 39 (35).

Compound (I)

Crystal data

$C_{22}H_{25}N_3O$
M_r = 347.45
Triclinic, $P\overline{1}$
a = 6.1514 (2) Å
b = 10.3171 (5) Å
c = 15.7351 (8) Å
α = 71.722 (2)°
β = 85.780 (3)°
γ = 85.306 (3)°
V = 943.84 (7) Å^3
Z = 2
D_x = 1.223 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4348 reflections
θ = 3.3–27.6°
μ = 0.08 mm^{-1}
T = 120 (2) K
Needle, colourless
0.18 × 0.08 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.964, T_{max} = 0.994
21 211 measured reflections
4348 independent reflections
2666 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
θ_{max} = 27.6°
$h = -7 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.160
S = 1.02
4348 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.3463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected interatomic distances (Å) for (I).

N1—N2	1.384 (2)	C7—C8	1.529 (3)
N2—C3	1.320 (3)	C8—C8a	1.500 (3)
C3—C3a	1.438 (3)	C8a—N9	1.338 (3)
C3a—C4	1.386 (3)	N9—C9a	1.340 (3)
C4—C4a	1.386 (3)	C9a—N1	1.368 (3)
C4a—C5	1.482 (3)	C3a—C9a	1.408 (3)
C5—C6	1.500 (3)	C4a—C8a	1.416 (3)
C6—C7	1.532 (3)		

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

D—H···a	D—H	H···A	D···A	D—H···A
C6—H6B···N9^{i}	0.99	2.56	3.512 (3)	161

Symmetry code: (i) $1 + x, y, z$.

Compound (II)

Crystal data

$C_{19}H_{19}N_3O$
M_r = 305.37
Monoclinic, $P2_1/c$
a = 7.7988 (3) Å
b = 17.0950 (6) Å
c = 12.0231 (3) Å
β = 108.8000 (18)°
V = 1517.41 (9) Å^3
Z = 4
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3477 reflections
θ = 3.0–27.6°
μ = 0.09 mm^{-1}
T = 120 (2) K
Plate, colourless
0.40 × 0.20 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.974, T_{max} = 0.993
21 719 measured reflections
3477 independent reflections
2503 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 27.6°
$h = -9 \rightarrow 10$
$k = -21 \rightarrow 22$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.134
S = 1.03
3477 reflections
212 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.3887P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.027 (3)

Table 3
Selected interatomic distances (Å) for (II).

N1—C2	1.340 (2)	C8—C9	1.535 (2)
C2—C3	1.407 (2)	C9—C9a	1.493 (2)
C3—C3a	1.383 (2)	C9a—N9b	1.355 (2)
C3a—N4	1.358 (2)	N9b—N1	1.3625 (18)
N4—C5	1.325 (2)	C3a—N9b	1.393 (2)
C5—C5a	1.446 (2)	C5a—C9a	1.379 (2)
C5a—C6	1.495 (2)	C7—C8	1.523 (2)
C6—C7	1.513 (2)		

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C54—H54···N1^{ii}	0.95	2.58	3.492 (2)	162

Symmetry code: (ii) $1 + x, \frac{3}{2} - y, z - \frac{1}{2}$.

Crystals of (I) are triclinic; space group $P\overline{1}$ was selected and confirmed by the successful structure analysis. For (II), space group $P2_1/c$ was uniquely determined from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic), 0.98 (CH_3) or 0.99 Å (CH_2), and with U_{iso}(H) values of $1.2U_{eq}$(C) [$1.5U_{eq}$(C) for the methyl groups].

For both compounds, 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 *SHELXS*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 that have provided computing facilities for this work. JC thanks the Consejería de Educación y Ciencia (Junta de Andalucía, Spain) for financial support. JM and JQ thank COLCIENCIAS and the Universidad de Valle for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG1220). Services for accessing these data are described at the back of the journal.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.

Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.

Cannon, D., Quesada, A., Quiroga, J., Mejía, D., Insuasty, B., Abonia, R., Cobo, J., Nogueras, M., Sánchez, A. & Low, J. N. (2001*a*). *Acta Cryst.* E**57**, o151–o153.

Cannon, D., Quesada, A., Quiroga, J., Mejía, D., Insuasty, B., Abonia, R., Cobo, J., Nogueras, M., Sánchez, A. & Low, J. N. (2001*b*). *Acta Cryst.* E**57**, o157–o159.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Evans, D. G. & Boeyens, J. C. A. (1989). *Acta Cryst.* B**45**, 581–590.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.

Glidewell, C. & Lloyd, D. M. G. (1984). *Tetrahedron*, **40**, 4455–4472.

Low, J. N., Cobo, J., Mera, J., Quiroga, J. & Glidewell, C. (2004). *Acta Cryst.* C**60**, o265–o269.

Low, J. N., Cobo, J., Nogueras, M., Sánchez, A., Quiroga, J. & Mejía, D. (2001). *Acta Cryst.* C**57**, 1356–1358.

McArdle, P. (2003). *OSCAIL for Windows. Program for Structure Solution.* Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.

Nonius (1997). *KappaCCD Server Software.* Windows 3.11 Version. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Quiroga, J., Hormaza, A., Insuasty, B., Saitz, C. & Jullian, C. (1998). *J. Heterocycl. Chem.* **35**, 575–578.

Quiroga, J., Mejía, D., Insuasty, B., Abonia, R., Nogueras, M., Sánchez, A., Cobo, J. & Low, J. N. (2001). *Tetrahedron*, **57**, 6947–6953.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2004). C**60**, o479–o482 [doi:10.1107/S0108270104011291]

3-*tert*-Butyl-7,7-dimethyl-1-phenyl-5,6,7,8-tetrahydroimidazo[3,4-*b*]quinolin-5-one and 2,8,8-trimethyl-5-phenyl-6,7,8,9-tetrahydroimidazo[2,3-*a*]quinolin-6-one: chains generated by C—H···N hydrogen bonds

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Computing details

For both compounds, 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 *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXS97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) 3-*tert*-Butyl-7,7-dimethyl-1-phenyl-5,6,7,8- tetrahydroimidazo[3,4-*b*]quinolin-5-one

Crystal data

$C_{22}H_{25}N_3O$
$M_r = 347.45$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.1514\ (2)$ Å
$b = 10.3171\ (5)$ Å
$c = 15.7351\ (8)$ Å
$\alpha = 71.722\ (2)°$
$\beta = 85.780\ (3)°$
$\gamma = 85.306\ (3)°$
$V = 943.84\ (7)$ Å^3
$Z = 2$
$F(000) = 372$
$D_x = 1.223$ Mg m^{-3}
Melting point: 413 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4348 reflections
$\theta = 3.3–27.6°$
$\mu = 0.08$ mm^{-1}
$T = 120$ K
Needle, colourless
0.18 × 0.08 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: rotating anode
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{min} = 0.964$, $T_{max} = 0.994$
21211 measured reflections
4348 independent reflections
2666 reflections with $I > 2\sigma(I)$
$R_{int} = 0.105$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.3°$
$h = -7\rightarrow8$
$k = -13\rightarrow13$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.160$
$S = 1.02$
4348 reflections
240 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
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.3463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4926 (3)	0.63590 (17)	0.29289 (11)	0.0294 (4)
C11	0.3406 (3)	0.7490 (2)	0.28932 (14)	0.0300 (5)
C12	0.3974 (4)	0.8816 (2)	0.24695 (15)	0.0352 (5)
C13	0.2449 (4)	0.9890 (2)	0.24520 (16)	0.0401 (6)
C14	0.0383 (4)	0.9640 (3)	0.28538 (16)	0.0408 (6)
C15	−0.0151 (4)	0.8318 (2)	0.32808 (15)	0.0381 (5)
C16	0.1342 (3)	0.7228 (2)	0.33087 (15)	0.0342 (5)
N2	0.4543 (3)	0.51076 (18)	0.35583 (12)	0.0318 (4)
C3	0.6170 (3)	0.4233 (2)	0.34732 (14)	0.0295 (5)
C31	0.6163 (3)	0.2757 (2)	0.40451 (14)	0.0326 (5)
C32	0.4554 (4)	0.2600 (3)	0.48550 (16)	0.0427 (6)
C33	0.5456 (4)	0.1925 (2)	0.34698 (17)	0.0427 (6)
C34	0.8452 (4)	0.2235 (3)	0.43831 (17)	0.0465 (6)
C4A	1.0614 (3)	0.5607 (2)	0.16742 (13)	0.0269 (5)
C3A	0.7706 (3)	0.4904 (2)	0.27701 (13)	0.0279 (5)
C4	0.9659 (3)	0.4585 (2)	0.23619 (13)	0.0285 (5)
C5	1.2720 (3)	0.5296 (2)	0.12364 (14)	0.0294 (5)
O5	1.3606 (2)	0.41429 (16)	0.14590 (10)	0.0391 (4)
C6	1.3728 (3)	0.6460 (2)	0.05286 (14)	0.0330 (5)
C7	1.2080 (3)	0.7542 (2)	−0.00153 (14)	0.0310 (5)
C71	1.0758 (4)	0.6924 (2)	−0.05632 (15)	0.0381 (5)
C72	1.3302 (4)	0.8716 (2)	−0.06540 (16)	0.0429 (6)
C8	1.0557 (3)	0.8051 (2)	0.06407 (14)	0.0322 (5)
C8A	0.9560 (3)	0.6933 (2)	0.13889 (14)	0.0281 (5)
N9	0.7658 (3)	0.72708 (17)	0.17598 (11)	0.0290 (4)
C9A	0.6834 (3)	0.6263 (2)	0.24410 (14)	0.0281 (5)
H12	0.5392	0.8990	0.2194	0.042*
H13	0.2826	1.0804	0.2162	0.048*
H14	−0.0660	1.0380	0.2834	0.049*
H15	−0.1566	0.8149	0.3561	0.046*
H16	0.0963	0.6316	0.3607	0.041*
H32A	0.4968	0.3163	0.5209	0.064*
H32B	0.4586	0.1639	0.5226	0.064*
H32C	0.3077	0.2897	0.4648	0.064*
H33A	0.3961	0.2229	0.3288	0.064*
H33B	0.5512	0.0953	0.3818	0.064*
H33C	0.6443	0.2062	0.2935	0.064*
H34A	0.9478	0.2264	0.3871	0.070*

H34B	0.8406	0.1292	0.4782	0.070*
H34C	0.8933	0.2817	0.4712	0.070*
H4	1.0330	0.3685	0.2549	0.034*
H6A	1.4670	0.6087	0.0112	0.040*
H6B	1.4675	0.6910	0.0819	0.040*
H71A	0.9769	0.7637	−0.0933	0.057*
H71B	0.9905	0.6197	−0.0157	0.057*
H71C	1.1755	0.6541	−0.0951	0.057*
H72A	1.4173	0.9110	−0.0310	0.064*
H72B	1.2251	0.9420	−0.0992	0.064*
H72C	1.4269	0.8374	−0.1070	0.064*
H8A	1.1388	0.8603	0.0902	0.039*
H8B	0.9369	0.8658	0.0305	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0281 (9)	0.0301 (10)	0.0288 (10)	0.0010 (7)	0.0018 (7)	−0.0089 (8)
C11	0.0303 (11)	0.0353 (12)	0.0266 (11)	0.0046 (9)	−0.0054 (8)	−0.0133 (10)
C12	0.0328 (12)	0.0357 (13)	0.0364 (13)	0.0024 (9)	−0.0001 (9)	−0.0119 (10)
C13	0.0425 (14)	0.0372 (13)	0.0396 (14)	0.0068 (10)	−0.0008 (10)	−0.0132 (11)
C14	0.0386 (13)	0.0461 (15)	0.0409 (14)	0.0124 (11)	−0.0029 (10)	−0.0215 (12)
C15	0.0295 (12)	0.0505 (15)	0.0378 (13)	0.0041 (10)	0.0005 (9)	−0.0211 (12)
C16	0.0305 (12)	0.0399 (13)	0.0348 (12)	0.0000 (10)	−0.0001 (9)	−0.0159 (10)
N2	0.0304 (10)	0.0340 (10)	0.0299 (10)	−0.0015 (8)	−0.0013 (7)	−0.0084 (8)
C3	0.0280 (11)	0.0344 (12)	0.0278 (11)	−0.0008 (9)	−0.0029 (8)	−0.0121 (9)
C31	0.0303 (11)	0.0343 (12)	0.0293 (12)	−0.0003 (9)	−0.0011 (9)	−0.0047 (9)
C32	0.0394 (13)	0.0441 (14)	0.0367 (14)	−0.0045 (11)	0.0056 (10)	−0.0026 (11)
C33	0.0480 (14)	0.0329 (13)	0.0457 (15)	0.0006 (10)	−0.0054 (11)	−0.0099 (11)
C34	0.0377 (13)	0.0521 (16)	0.0377 (14)	0.0019 (11)	−0.0042 (10)	0.0026 (12)
C4A	0.0262 (10)	0.0310 (11)	0.0259 (11)	−0.0005 (8)	−0.0018 (8)	−0.0126 (9)
C3A	0.0272 (10)	0.0313 (11)	0.0259 (11)	−0.0009 (8)	−0.0037 (8)	−0.0096 (9)
C4	0.0292 (11)	0.0292 (11)	0.0276 (11)	0.0032 (9)	−0.0048 (8)	−0.0100 (9)
C5	0.0291 (11)	0.0369 (13)	0.0245 (11)	0.0012 (9)	−0.0035 (8)	−0.0131 (10)
O5	0.0382 (9)	0.0386 (9)	0.0342 (9)	0.0096 (7)	0.0027 (7)	−0.0064 (7)
C6	0.0292 (11)	0.0376 (12)	0.0328 (12)	−0.0021 (9)	0.0021 (9)	−0.0127 (10)
C7	0.0369 (12)	0.0251 (11)	0.0321 (12)	−0.0050 (9)	0.0037 (9)	−0.0109 (9)
C71	0.0507 (14)	0.0336 (12)	0.0295 (12)	−0.0035 (10)	−0.0047 (10)	−0.0085 (10)
C72	0.0518 (15)	0.0335 (13)	0.0423 (14)	−0.0092 (11)	0.0136 (11)	−0.0122 (11)
C8	0.0353 (12)	0.0273 (11)	0.0347 (12)	−0.0023 (9)	0.0026 (9)	−0.0114 (10)
C8A	0.0291 (11)	0.0304 (11)	0.0274 (11)	−0.0033 (9)	−0.0018 (8)	−0.0123 (9)
N9	0.0301 (9)	0.0292 (10)	0.0292 (10)	−0.0009 (7)	−0.0008 (7)	−0.0117 (8)
C9A	0.0267 (11)	0.0334 (11)	0.0267 (11)	−0.0016 (9)	−0.0025 (8)	−0.0127 (9)

Geometric parameters (Å, º)

N1—N2	1.384 (2)	C3—C31	1.506 (3)
N2—C3	1.320 (3)	C31—C32	1.530 (3)

C3—C3A	1.438 (3)	C31—C33	1.534 (3)
C3A—C4	1.386 (3)	C31—C34	1.537 (3)
C4—C4A	1.386 (3)	C32—H32A	0.98
C4A—C5	1.482 (3)	C32—H32B	0.98
C5—C6	1.500 (3)	C32—H32C	0.98
C6—C7	1.532 (3)	C33—H33A	0.98
C7—C8	1.529 (3)	C33—H33B	0.98
C8—C8A	1.500 (3)	C33—H33C	0.98
C8A—N9	1.338 (3)	C34—H34A	0.98
N9—C9A	1.340 (3)	C34—H34B	0.98
C9A—N1	1.368 (3)	C34—H34C	0.98
C3A—C9A	1.408 (3)	C4—H4	0.95
C4A—C8A	1.416 (3)	C5—O5	1.223 (2)
N1—C11	1.424 (3)	C6—H6A	0.99
C11—C12	1.383 (3)	C6—H6B	0.99
C11—C16	1.391 (3)	C7—C72	1.522 (3)
C12—C13	1.386 (3)	C7—C71	1.531 (3)
C12—H12	0.95	C71—H71A	0.98
C13—C14	1.383 (3)	C71—H71B	0.98
C13—H13	0.95	C71—H71C	0.98
C14—C15	1.373 (3)	C72—H72A	0.98
C14—H14	0.95	C72—H72B	0.98
C15—C16	1.384 (3)	C72—H72C	0.98
C15—H15	0.95	C8—H8A	0.99
C16—H16	0.95	C8—H8B	0.99

C9A—N1—N2	110.12 (16)	C4—C4A—C8A	119.88 (18)
C9A—N1—C11	131.16 (18)	C4—C4A—C5	119.54 (18)
N2—N1—C11	118.72 (16)	C8A—C4A—C5	120.58 (18)
C12—C11—C16	120.7 (2)	C4—C3A—C9A	116.53 (19)
C12—C11—N1	120.93 (19)	C4—C3A—C3	138.5 (2)
C16—C11—N1	118.3 (2)	C9A—C3A—C3	104.99 (17)
C11—C12—C13	119.2 (2)	C4A—C4—C3A	118.51 (19)
C11—C12—H12	120.4	C4A—C4—H4	120.7
C13—C12—H12	120.4	C3A—C4—H4	120.7
C14—C13—C12	120.6 (2)	O5—C5—C4A	121.01 (19)
C14—C13—H13	119.7	O5—C5—C6	121.91 (19)
C12—C13—H13	119.7	C4A—C5—C6	117.06 (18)
C15—C14—C13	119.6 (2)	C5—C6—C7	114.52 (17)
C15—C14—H14	120.2	C5—C6—H6A	108.6
C13—C14—H14	120.2	C7—C6—H6A	108.6
C14—C15—C16	121.0 (2)	C5—C6—H6B	108.6
C14—C15—H15	119.5	C7—C6—H6B	108.6
C16—C15—H15	119.5	H6A—C6—H6B	107.6
C15—C16—C11	118.9 (2)	C72—C7—C8	110.44 (17)
C15—C16—H16	120.5	C72—C7—C71	108.81 (18)
C11—C16—H16	120.5	C8—C7—C71	109.99 (18)
C3—N2—N1	107.77 (17)	C72—C7—C6	109.24 (18)

N2—C3—C3A	109.97 (19)	C8—C7—C6	108.03 (18)
N2—C3—C31	120.21 (19)	C71—C7—C6	110.32 (17)
C3A—C3—C31	129.76 (18)	C7—C71—H71A	109.5
C3—C31—C32	110.35 (18)	C7—C71—H71B	109.5
C3—C31—C33	107.90 (18)	H71A—C71—H71B	109.5
C32—C31—C33	109.42 (19)	C7—C71—H71C	109.5
C3—C31—C34	110.33 (18)	H71A—C71—H71C	109.5
C32—C31—C34	108.67 (18)	H71B—C71—H71C	109.5
C33—C31—C34	110.15 (19)	C7—C72—H72A	109.5
C31—C32—H32A	109.5	C7—C72—H72B	109.5
C31—C32—H32B	109.5	H72A—C72—H72B	109.5
H32A—C32—H32B	109.5	C7—C72—H72C	109.5
C31—C32—H32C	109.5	H72A—C72—H72C	109.5
H32A—C32—H32C	109.5	H72B—C72—H72C	109.5
H32B—C32—H32C	109.5	C8A—C8—C7	114.17 (17)
C31—C33—H33A	109.5	C8A—C8—H8A	108.7
C31—C33—H33B	109.5	C7—C8—H8A	108.7
H33A—C33—H33B	109.5	C8A—C8—H8B	108.7
C31—C33—H33C	109.5	C7—C8—H8B	108.7
H33A—C33—H33C	109.5	H8A—C8—H8B	107.6
H33B—C33—H33C	109.5	N9—C8A—C4A	123.13 (19)
C31—C34—H34A	109.5	N9—C8A—C8	116.16 (18)
C31—C34—H34B	109.5	C4A—C8A—C8	120.71 (18)
H34A—C34—H34B	109.5	C8A—N9—C9A	114.87 (18)
C31—C34—H34C	109.5	N9—C9A—N1	125.79 (19)
H34A—C34—H34C	109.5	N9—C9A—C3A	127.03 (18)
H34B—C34—H34C	109.5	N1—C9A—C3A	107.15 (18)

C9A—N1—C11—C12	−17.0 (3)	C4—C4A—C5—O5	1.7 (3)
N2—N1—C11—C12	161.83 (19)	C8A—C4A—C5—O5	−177.62 (19)
C9A—N1—C11—C16	164.1 (2)	C4—C4A—C5—C6	−176.99 (18)
N2—N1—C11—C16	−17.1 (3)	C8A—C4A—C5—C6	3.7 (3)
C16—C11—C12—C13	−0.9 (3)	O5—C5—C6—C7	148.82 (19)
N1—C11—C12—C13	−179.79 (19)	C4A—C5—C6—C7	−32.5 (3)
C11—C12—C13—C14	0.1 (3)	C5—C6—C7—C72	175.44 (18)
C12—C13—C14—C15	0.6 (3)	C5—C6—C7—C8	55.3 (2)
C13—C14—C15—C16	−0.6 (3)	C5—C6—C7—C71	−65.0 (2)
C14—C15—C16—C11	−0.2 (3)	C72—C7—C8—C8A	−170.53 (18)
C12—C11—C16—C15	0.9 (3)	C71—C7—C8—C8A	69.4 (2)
N1—C11—C16—C15	179.86 (18)	C6—C7—C8—C8A	−51.1 (2)
C9A—N1—N2—C3	0.0 (2)	C4—C4A—C8A—N9	0.7 (3)
C11—N1—N2—C3	−179.07 (17)	C5—C4A—C8A—N9	−179.98 (18)
N1—N2—C3—C3A	0.2 (2)	C4—C4A—C8A—C8	−179.51 (18)
N1—N2—C3—C31	−177.34 (17)	C5—C4A—C8A—C8	−0.2 (3)
N2—C3—C31—C32	−17.7 (3)	C7—C8—C8A—N9	−154.76 (18)
C3A—C3—C31—C32	165.3 (2)	C7—C8—C8A—C4A	25.5 (3)
N2—C3—C31—C33	101.8 (2)	C4A—C8A—N9—C9A	1.4 (3)
C3A—C3—C31—C33	−75.2 (3)	C8—C8A—N9—C9A	−178.38 (17)

N2—C3—C31—C34	−137.9 (2)	C8A—N9—C9A—N1	179.57 (18)
C3A—C3—C31—C34	45.2 (3)	C8A—N9—C9A—C3A	−2.8 (3)
N2—C3—C3A—C4	−179.8 (2)	N2—N1—C9A—N9	177.88 (18)
C31—C3—C3A—C4	−2.6 (4)	C11—N1—C9A—N9	−3.2 (3)
N2—C3—C3A—C9A	−0.3 (2)	N2—N1—C9A—C3A	−0.1 (2)
C31—C3—C3A—C9A	176.94 (19)	C11—N1—C9A—C3A	178.75 (19)
C8A—C4A—C4—C3A	−1.6 (3)	C4—C3A—C9A—N9	1.9 (3)
C5—C4A—C4—C3A	179.07 (18)	C3—C3A—C9A—N9	−177.76 (19)
C9A—C3A—C4—C4A	0.4 (3)	C4—C3A—C9A—N1	179.92 (17)
C3—C3A—C4—C4A	180.0 (2)	C3—C3A—C9A—N1	0.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···N9^{i}	0.99	2.56	3.512 (3)	161

Symmetry code: (i) *x*+1, *y*, *z*.

(II) 2,8,8-Trimethyl-5-phenyl-6,7,8,9-tetrahydroimidazo[2,3-*a*]quinolin-6-one

Crystal data

$C_{19}H_{19}N_3O$
M_r = 305.37
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.7988 (3) Å
b = 17.0950 (6) Å
c = 12.0231 (3) Å
β = 108.8000 (18)°
V = 1517.41 (9) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.337 Mg m^{-3}
Melting point: 533 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3477 reflections
θ = 3.0–27.6°
μ = 0.09 mm^{-1}
T = 120 K
Plate, colourless
0.40 × 0.20 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: rotating anode
Graphite monochromator
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.974, T_{max} = 0.993
21719 measured reflections
3477 independent reflections
2503 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 27.6°, θ_{min} = 3.0°
h = −9→10
k = −21→22
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.134
S = 1.03
3477 reflections
212 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
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.3887P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Extinction correction: *SHELXL97*, $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.027 (3)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.20587 (18)	0.84734 (8)	0.64251 (12)	0.0212 (3)
C2	0.2151 (2)	0.92440 (9)	0.62466 (14)	0.0208 (4)
C3	0.3362 (2)	0.94346 (10)	0.56381 (14)	0.0223 (4)
C3A	0.4062 (2)	0.87260 (9)	0.54327 (14)	0.0204 (4)
N4	0.52405 (18)	0.85190 (8)	0.48631 (12)	0.0211 (3)
C5	0.5692 (2)	0.77709 (9)	0.48729 (14)	0.0201 (4)
C5A	0.4993 (2)	0.71800 (9)	0.54723 (13)	0.0195 (4)
C6	0.5815 (2)	0.63875 (9)	0.57918 (14)	0.0217 (4)
O6	0.72358 (17)	0.61996 (7)	0.56513 (11)	0.0311 (3)
C7	0.4900 (2)	0.58442 (9)	0.64206 (15)	0.0244 (4)
C8	0.2888 (2)	0.59886 (9)	0.61866 (14)	0.0223 (4)
C9	0.2671 (2)	0.68471 (9)	0.64919 (15)	0.0223 (4)
C9A	0.3664 (2)	0.73904 (9)	0.59422 (13)	0.0189 (4)
N9B	0.32353 (18)	0.81598 (7)	0.59205 (11)	0.0192 (3)
C21	0.0981 (2)	0.97774 (9)	0.66769 (15)	0.0249 (4)
C51	0.6914 (2)	0.75871 (10)	0.41790 (14)	0.0208 (4)
C52	0.8311 (2)	0.81025 (10)	0.42116 (15)	0.0239 (4)
C53	0.9428 (2)	0.79628 (10)	0.35357 (15)	0.0271 (4)
C54	0.9150 (2)	0.73119 (10)	0.28171 (15)	0.0273 (4)
C55	0.7744 (2)	0.68033 (10)	0.27692 (14)	0.0253 (4)
C56	0.6630 (2)	0.69345 (10)	0.34422 (14)	0.0232 (4)
C81	0.2181 (3)	0.54709 (10)	0.69784 (16)	0.0298 (4)
C82	0.1801 (2)	0.58207 (10)	0.49040 (15)	0.0283 (4)
H21A	0.0151	1.0060	0.6010	0.037*
H21B	0.1745	1.0153	0.7236	0.037*
H91C	0.0282	0.9469	0.7067	0.037*
H3	0.3639	0.9940	0.5416	0.027*
H52	0.8503	0.8553	0.4699	0.029*
H53	1.0384	0.8315	0.3568	0.032*
H54	0.9917	0.7214	0.2359	0.033*
H55	0.7544	0.6359	0.2268	0.030*
H56	0.5672	0.6581	0.3403	0.028*
H7A	0.5530	0.5890	0.7276	0.029*
H7B	0.5060	0.5300	0.6190	0.029*
H81A	0.2314	0.4920	0.6798	0.045*
H81B	0.0899	0.5587	0.6843	0.045*
H81C	0.2875	0.5574	0.7803	0.045*
H82A	0.2232	0.6159	0.4391	0.042*
H82B	0.0515	0.5924	0.4774	0.042*
H82C	0.1961	0.5272	0.4725	0.042*
H9A	0.1369	0.6985	0.6220	0.027*
H9B	0.3132	0.6913	0.7356	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0201 (7)	0.0215 (7)	0.0249 (7)	0.0016 (6)	0.0112 (6)	−0.0020 (6)
C2	0.0203 (8)	0.0194 (8)	0.0223 (8)	0.0005 (6)	0.0061 (7)	−0.0002 (6)
C3	0.0239 (9)	0.0177 (8)	0.0274 (8)	0.0014 (6)	0.0113 (7)	0.0018 (7)
C3A	0.0200 (8)	0.0209 (8)	0.0217 (8)	−0.0016 (7)	0.0086 (7)	0.0019 (6)
N4	0.0213 (7)	0.0198 (7)	0.0244 (7)	0.0018 (6)	0.0106 (6)	0.0012 (6)
C5	0.0189 (8)	0.0214 (8)	0.0197 (8)	−0.0006 (6)	0.0058 (7)	0.0000 (6)
C5A	0.0190 (8)	0.0196 (8)	0.0202 (8)	−0.0013 (6)	0.0069 (7)	−0.0017 (6)
C6	0.0224 (9)	0.0210 (8)	0.0219 (8)	0.0005 (7)	0.0072 (7)	−0.0030 (7)
O6	0.0319 (7)	0.0291 (7)	0.0385 (7)	0.0095 (6)	0.0202 (6)	0.0059 (5)
C7	0.0272 (9)	0.0196 (8)	0.0273 (9)	0.0013 (7)	0.0100 (7)	0.0034 (7)
C8	0.0246 (9)	0.0169 (8)	0.0268 (9)	−0.0008 (7)	0.0102 (7)	0.0004 (7)
C9	0.0247 (9)	0.0196 (8)	0.0259 (8)	−0.0028 (7)	0.0126 (7)	−0.0017 (7)
C9A	0.0199 (8)	0.0162 (8)	0.0203 (8)	−0.0011 (6)	0.0059 (7)	−0.0003 (6)
N9B	0.0185 (7)	0.0183 (7)	0.0230 (7)	−0.0004 (5)	0.0101 (6)	−0.0004 (5)
C21	0.0260 (9)	0.0220 (8)	0.0294 (9)	0.0014 (7)	0.0129 (8)	−0.0012 (7)
C51	0.0192 (9)	0.0223 (8)	0.0214 (8)	0.0029 (7)	0.0070 (7)	0.0031 (6)
C52	0.0242 (9)	0.0225 (9)	0.0267 (9)	−0.0001 (7)	0.0104 (7)	−0.0006 (7)
C53	0.0210 (9)	0.0297 (9)	0.0336 (10)	−0.0003 (7)	0.0132 (8)	0.0039 (8)
C54	0.0286 (10)	0.0302 (10)	0.0284 (9)	0.0073 (8)	0.0163 (8)	0.0077 (7)
C55	0.0304 (10)	0.0242 (9)	0.0222 (8)	0.0055 (7)	0.0100 (8)	0.0013 (7)
C56	0.0221 (9)	0.0239 (9)	0.0238 (8)	0.0007 (7)	0.0078 (7)	0.0028 (7)
C81	0.0343 (10)	0.0232 (9)	0.0349 (10)	−0.0028 (7)	0.0155 (8)	0.0039 (7)
C82	0.0299 (10)	0.0245 (9)	0.0292 (9)	−0.0051 (7)	0.0076 (8)	−0.0029 (7)

Geometric parameters (Å, º)

N1—C2	1.340 (2)	C52—H52	0.95
C2—C3	1.407 (2)	C53—C54	1.382 (3)
C3—C3A	1.383 (2)	C53—H53	0.95
C3A—N4	1.358 (2)	C54—C55	1.385 (3)
N4—C5	1.325 (2)	C54—H54	0.95
C5—C5A	1.446 (2)	C55—C56	1.383 (2)
C5A—C6	1.495 (2)	C55—H55	0.95
C6—C7	1.513 (2)	C56—H56	0.95
C8—C9	1.535 (2)	C6—O6	1.217 (2)
C9—C9A	1.493 (2)	C7—C8	1.523 (2)
C9A—N9B	1.355 (2)	C7—H7A	0.99
N9B—N1	1.3625 (18)	C7—H7B	0.99
C3A—N9B	1.393 (2)	C8—C81	1.527 (2)
C5A—C9A	1.379 (2)	C8—C82	1.528 (2)
C2—C21	1.495 (2)	C81—H81A	0.98
C21—H21A	0.98	C81—H81B	0.98
C21—H21B	0.98	C81—H81C	0.98
C21—H91C	0.98	C82—H82A	0.98
C3—H3	0.95	C82—H82B	0.98

C5—C51	1.488 (2)	C82—H82C	0.98
C51—C52	1.392 (2)	C9—H9A	0.99
C51—C56	1.397 (2)	C9—H9B	0.99
C52—C53	1.390 (2)		

C2—N1—N9B	103.69 (12)	C5—C5A—C6	124.20 (14)
N1—C2—C3	113.00 (14)	O6—C6—C5A	122.53 (15)
N1—C2—C21	118.27 (14)	O6—C6—C7	120.29 (15)
C3—C2—C21	128.72 (15)	C5A—C6—C7	116.90 (14)
C2—C21—H21A	109.5	C6—C7—C8	115.59 (14)
C2—C21—H21B	109.5	C6—C7—H7A	108.4
H21A—C21—H21B	109.5	C8—C7—H7A	108.4
C2—C21—H91C	109.5	C6—C7—H7B	108.4
H21A—C21—H91C	109.5	C8—C7—H7B	108.4
H21B—C21—H91C	109.5	H7A—C7—H7B	107.4
C3A—C3—C2	105.04 (14)	C7—C8—C81	110.40 (14)
C3A—C3—H3	127.5	C7—C8—C82	111.05 (14)
C2—C3—H3	127.5	C81—C8—C82	109.03 (14)
N4—C3A—C3	133.49 (15)	C7—C8—C9	107.28 (13)
N4—C3A—N9B	120.76 (14)	C81—C8—C9	108.38 (13)
C3—C3A—N9B	105.71 (13)	C82—C8—C9	110.66 (14)
C5—N4—C3A	117.91 (13)	C8—C81—H81A	109.5
N4—C5—C5A	122.46 (14)	C8—C81—H81B	109.5
N4—C5—C51	114.53 (14)	H81A—C81—H81B	109.5
C5A—C5—C51	122.97 (14)	C8—C81—H81C	109.5
C52—C51—C56	119.07 (15)	H81A—C81—H81C	109.5
C52—C51—C5	119.17 (15)	H81B—C81—H81C	109.5
C56—C51—C5	121.66 (14)	C8—C82—H82A	109.5
C53—C52—C51	120.47 (16)	C8—C82—H82B	109.5
C53—C52—H52	119.8	H82A—C82—H82B	109.5
C51—C52—H52	119.8	C8—C82—H82C	109.5
C54—C53—C52	120.12 (16)	H82A—C82—H82C	109.5
C54—C53—H53	119.9	H82B—C82—H82C	109.5
C52—C53—H53	119.9	C9A—C9—C8	112.09 (13)
C53—C54—C55	119.61 (15)	C9A—C9—H9A	109.2
C53—C54—H54	120.2	C8—C9—H9A	109.2
C55—C54—H54	120.2	C9A—C9—H9B	109.2
C56—C55—C54	120.76 (16)	C8—C9—H9B	109.2
C56—C55—H55	119.6	H9A—C9—H9B	107.9
C54—C55—H55	119.6	N9B—C9A—C5A	117.30 (14)
C55—C56—C51	119.95 (16)	N9B—C9A—C9	116.90 (13)
C55—C56—H56	120.0	C5A—C9A—C9	125.79 (14)
C51—C56—H56	120.0	C9A—N9B—N1	124.80 (13)
C9A—C5A—C5	118.42 (14)	C9A—N9B—C3A	122.53 (13)
C9A—C5A—C6	116.60 (14)	N1—N9B—C3A	112.55 (13)

N9B—N1—C2—C3	−0.02 (18)	C5—C5A—C6—O6	6.0 (2)
N9B—N1—C2—C21	178.86 (14)	C9A—C5A—C6—C7	10.2 (2)

N1—C2—C3—C3A	−0.11 (19)	C5—C5A—C6—C7	179.90 (15)
C21—C2—C3—C3A	−178.85 (16)	O6—C6—C7—C8	−159.59 (15)
C2—C3—C3A—N4	177.79 (17)	C5A—C6—C7—C8	26.4 (2)
C2—C3—C3A—N9B	0.18 (18)	C6—C7—C8—C81	−173.26 (14)
C3—C3A—N4—C5	177.12 (18)	C6—C7—C8—C82	65.68 (18)
N9B—C3A—N4—C5	−5.6 (2)	C6—C7—C8—C9	−55.35 (18)
C3A—N4—C5—C5A	−0.7 (2)	C7—C8—C9—C9A	49.05 (18)
C3A—N4—C5—C51	177.04 (14)	C81—C8—C9—C9A	168.26 (14)
N4—C5—C51—C52	39.9 (2)	C82—C8—C9—C9A	−72.23 (18)
C5A—C5—C51—C52	−142.40 (16)	C5—C5A—C9A—N9B	−6.9 (2)
N4—C5—C51—C56	−136.28 (16)	C6—C5A—C9A—N9B	163.46 (14)
C5A—C5—C51—C56	41.4 (2)	C5—C5A—C9A—C9	174.13 (15)
C56—C51—C52—C53	−1.1 (2)	C6—C5A—C9A—C9	−15.5 (2)
C5—C51—C52—C53	−177.37 (15)	C8—C9—C9A—N9B	165.06 (14)
C51—C52—C53—C54	0.5 (3)	C8—C9—C9A—C5A	−16.0 (2)
C52—C53—C54—C55	0.4 (3)	C5A—C9A—N9B—N1	−174.75 (14)
C53—C54—C55—C56	−0.7 (3)	C9—C9A—N9B—N1	4.3 (2)
C54—C55—C56—C51	0.1 (3)	C5A—C9A—N9B—C3A	1.0 (2)
C52—C51—C56—C55	0.8 (2)	C9—C9A—N9B—C3A	−179.96 (14)
C5—C51—C56—C55	176.97 (15)	C2—N1—N9B—C9A	176.24 (14)
N4—C5—C5A—C9A	7.1 (2)	C2—N1—N9B—C3A	0.14 (17)
C51—C5—C5A—C9A	−170.43 (15)	N4—C3A—N9B—C9A	5.6 (2)
N4—C5—C5A—C6	−162.50 (15)	C3—C3A—N9B—C9A	−176.41 (14)
C51—C5—C5A—C6	20.0 (2)	N4—C3A—N9B—N1	−178.19 (14)
C9A—C5A—C6—O6	−163.75 (16)	C3—C3A—N9B—N1	−0.21 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C54—H54···N1^{i}	0.95	2.58	3.492 (2)	162

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