

Ethyl 9*H*-1,2,3,4-tetrahydrocarbazole-3-carboxylate

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Key indicators

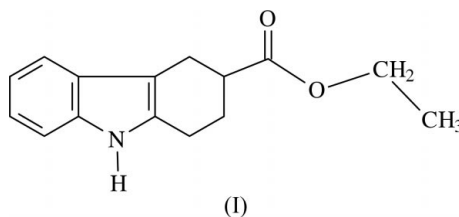
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
R factor = 0.043
wR factor = 0.115
Data-to-parameter ratio = 9.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{15}\text{H}_{17}\text{NO}_2$, consists of a tetrahydrocarbazole skeleton with a carboxyethyl group at position 3. Molecules are linked about inversion centres by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{N}\cdots\text{O}$ 2.908 (4) Å] to form centrosymmetric dimers.

Comment

Tetrahydrocarbazole derivatives can be considered to be synthetic precursors of cyclic indole-type alkaloids of biological interest (Phillipson & Zenk, 1980; Saxton, 1983; Abraham, 1975). They have tricyclic ring systems, as in the strychnose type of indole alkaloids (Bosch & Bonjoch, 1988).

The structures of tetrahydrocarbazole derivatives having different substituents at different positions of the carbazole core have been the subject of much interest in our laboratory. These include ethyl 4-methyl-9*H*-carbazole-3-carboxylate, (II) (Hökelek *et al.*, 2002), 2-(1,2,3,4-tetrahydrocarbazol-2-yl)-butylamine, (III) (Hökelek *et al.*, 2001*a*), 4-methylcarbazole-3-carboxylic acid, (IV) (Hökelek *et al.*, 2001*b*), 1-benzyloxy-1,2,3,4-tetrahydrocarbazole, (V) (Hökelek *et al.*, 2000), *N*-(1,2,3,4-tetrahydrocarbazole-1-yl)-2-methoxyacetamide, (VI) (Hökelek & Patır, 2000*a*), 2,3-dihydro-3-ethyl-9-(phenylsulfonyl)carbazole-4(1*H*)-one, (VII) (Hökelek & Patır, 2000*b*), *N*-(2,2-dimethoxyethyl)-*N*-(9-methoxymethyl-1,2,3,4-tetrahydrospiro[carbazole-1,2'-[1,3]dithiolan]-4-yl)benzamide, (VIII) (Hökelek & Patır, 1999), 9-acetonyl-3-ethylidene-1,2,3,4-tetrahydrospiro[carbazole-1,2'-[1,3]dithiolan]-4-one, (IX) (Hökelek *et al.*, 1999), spiro[carbazole-1(2*H*),2'-[1,3]dithiolan]-4(3*H*)-one, (X) (Hökelek *et al.*, 1998), *N*-(2-methoxymethyl)-*N*-(2,3,4,9-tetrahydrospiro[1*H*-carbazole-1,2-(1,3) dithiolan]-4-yl)benzenesulfonamide, (XI) (Patır *et al.*, 1997), 2,3-dihydro-9-(phenylsulfonyl)carbazole-4(1*H*)-one, (XII) (Hökelek *et al.*, 1994) and 1,2,3,4-tetrahydrocarbazole-1-spiro-2'-[1,3]dithiolane (XIII) (Hökelek *et al.*, 1994).



Tetrahydrocarbazole derivatives have an important role in the synthesis of indole alkaloids (Fritz *et al.*, 1993; Magnus *et al.*, 1992; Ergün *et al.*, 2000). Synthesizing indole-type alkaloids by substitution at different positions is currently under investigation (Patır *et al.*, 1997). Ellipticine, olivacine and their analogues have attracted much interest due to their anti-

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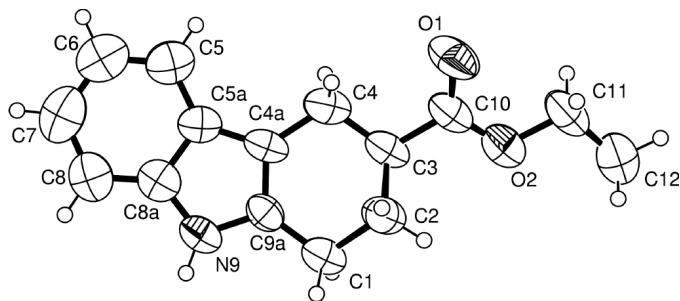


Figure 1

An ORTEP-3 (Farrugia, 1997) drawing of the title molecule, with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

tumour and antileukaemic activities (Svoboda *et al.*, 1968) and many elegant methods for the synthesis of ellipticine and related pyrido-carbazole alkaloids have been reported (Kansal & Potier, 1986; Ishikura *et al.*, 2000; Ergün *et al.*, 1998). The title compound, (I), may be a useful precursor for the synthesis of pyrido-carbazole alkaloids.

The present structure determination of (I) was undertaken in order to understand the effects of the carboxyethyl group on the geometry of the carbazole skeleton, and to compare the results with those obtained for previously reported tetrahydrocarbazole derivatives.

Compound (I) (Fig. 1) contains a tetrahydrocarbazole skeleton with a carboxyethyl group as substituent at position 3. The carboxyethyl group has an electron-withdrawing effect. As can be seen from the packing diagram (Fig. 2), there are intermolecular hydrogen bonds between the carbonyl O atoms and NH groups of neighbouring molecules [O1ⁱ...H9(N9) 2.09 (3) Å and N9—H9...O1ⁱ 168 (3)°; symmetry code: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$]. These intermolecular hydrogen bonds cause dimerization of the substituted carbazole molecules. Dipole-dipole and van der Waals interactions are also effective in the molecular packing. Intermolecular interactions may also cause increases in the angles C5A—C8A—C8 [122.7 (4)°], C1—C9A—N9 [125.1 (4)°], C8—C8A—N9 [130.8 (4)°], C5—C5A—C4A [133.8 (4)°], C4—C4A—C5A [129.1 (4)°], C4—C4A—C9A [123.9 (5)°], C1—C9A—C4A [125.3 (4)°] and decreases in the angles C9A—C1—C2 [108.7 (3)°], C1—C2—C3 [112.6 (3)°], C2—C3—C4 [112.4 (3)°] and C3—C4—C4A [111.2 (4)°].

The absence of any protecting group at atom N9 causes shortening of the C—N bonds [N9—C8A 1.377 (5) and N9—C9A 1.378 (4) Å]. These may be compared with the corresponding values [1.397 (4) and 1.360 (4) Å] in (II), [1.376 (4) and 1.391 (4) Å] in (VIII), [1.377 (2) and 1.396 (2) Å] in (IX), [1.382 (10) and 1.355 (3) Å] in (X), [1.390 (10) and 1.404 (9) Å] in (XI), [1.423 (5) and 1.412 (5) Å] in (XII) and [1.372 (5) and 1.392 (5) Å] in (XIII).

The carboxyethyl group in (I) cause notable changes in the geometry of the carbazole core, leading to increases in the angles C2—C3—C4, C4—C4A—C5A, C3—C4—C4A, C1—C9A—N9, C4A—C5A—C5 and decreases in C4—C4A—C9A,

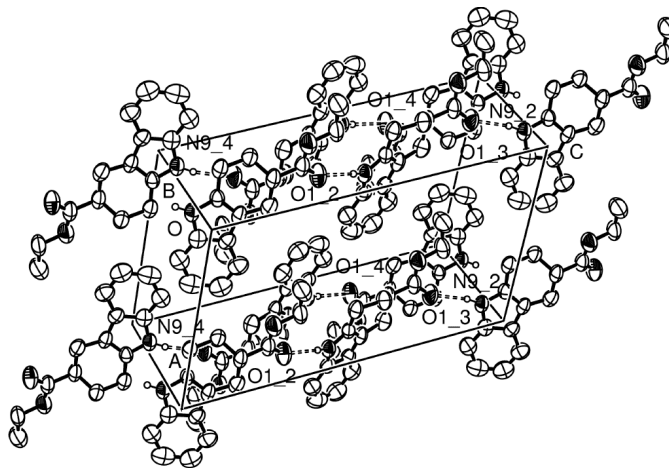


Figure 2

A packing diagram for (I). Hydrogen bonds are shown as dashed lines and H atoms not involved in hydrogen bonding have been omitted.

N9—C8A—C8 angles (Table 1), compared with the corresponding values in compounds (II), (IX), (X), and (XII) (Table 2).

In conclusion, the types of groups, depending on their electron-releasing or electron-donating properties, and their substitution positions, have a significant effect on the geometry of the carbazole system.

An examination of the deviations from the least-squares planes through the individual rings shows that rings A (C5A/C5—C8/C8A) and B (C4A/C5A/C8A/N9/C9A) are nearly planar, while ring C (C1—C4/C4A/C9A) is, of course, non-planar. The dihedral angles between the least-squares planes are A/B = 1.54 (13)°, A/C = 6.72 (12)° and B/C = 5.57 (12)°. Ring C has a local pseudo-twofold axis running through the midpoints of the C2—C3 and C9A—C4A bonds.

Experimental

The title compound, (I), was prepared from an ethyl 4-oxocyclohexane carboxylate (2.5 g, 14.70 mmol) and phenylhydrazine hydrochloride (2.33 g, 16.11 mmol) mixture in absolute ethanol (50 ml), which was refluxed for 6 h under an argon atmosphere. The solvent was removed under reduced pressure. The crude product was dissolved in chloroform and washed with hydrochloric acid (50 ml, 50%) and sodium carbonate (50 ml, 10%). After the organic layer was dried with anhydrous magnesium sulfate, the solvent was evaporated and the residue was crystallized from ethanol (yield 2.80 g, 78%), m.p. 369 K.

Crystal data

C₁₅H₁₇NO₂
M_r = 243.30
 Monoclinic, *P*2₁/*c*
a = 10.2221 (10) Å
b = 7.5133 (10) Å
c = 17.539 (2) Å
 β = 101.603 (9)°
V = 1319.5 (3) Å³
Z = 4

D_x = 1.225 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 10–18°
 μ = 0.08 mm⁻¹
T = 293 (2) K
 Rod, colourless
 0.30 × 0.15 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.024$
Non-profiled ω scans	$\theta_{\text{max}} = 26.3^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -12 \rightarrow 0$
$T_{\text{min}} = 0.976$, $T_{\text{max}} = 0.988$	$k = 0 \rightarrow 9$
2686 measured reflections	$l = -18 \rightarrow 21$
2686 independent reflections	3 standard reflections
653 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 1%

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.115$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2]$
$S = 0.92$	where $P = (F_o^2 + 2F_c^2)/3$
1583 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
167 parameters	$\Delta\rho_{\text{max}} = 0.11 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.15 \text{ e } \text{\AA}^{-3}$

Table 1

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

C9A–C4A	1.352 (4)	C3–C2	1.516 (4)
C9A–N9	1.378 (4)	C8A–C8	1.381 (5)
C9A–C1	1.478 (4)	C8A–C5A	1.407 (5)
O2–C10	1.329 (4)	C4A–C5A	1.419 (5)
O2–C11	1.448 (4)	C4A–C4	1.477 (4)
N9–C8A	1.377 (5)	C5A–C5	1.391 (5)
C10–O1	1.206 (4)	C5–C6	1.372 (5)
C10–C3	1.503 (5)	C11–C12	1.475 (5)
C1–C2	1.528 (4)	C7–C8	1.384 (5)
C3–C4	1.516 (5)	C7–C6	1.400 (5)
C4A–C9A–C1	125.3 (4)	C9A–C4A–C4	123.9 (5)
N9–C9A–C1	125.1 (4)	C5A–C4A–C4	129.1 (4)
C9A–C1–C2	108.7 (3)	C4A–C4–C3	111.2 (4)
C4–C3–C2	112.4 (3)	C3–C2–C1	112.6 (3)
N9–C8A–C8	130.8 (4)	C5–C5A–C8A	118.4 (5)
C8–C8A–C5A	122.7 (4)	C5–C5A–C4A	133.8 (4)
C9A–C4A–C5A	107.0 (4)	C8A–C5A–C4A	107.8 (4)
C4A–C9A–C1–C2	17.6 (4)	C1–C9A–C4A–C4	–0.6 (5)
O1–C10–C3–C4	8.8 (5)	C9A–C4A–C4–C3	11.7 (4)
O2–C10–C3–C4	–172.3 (3)	C2–C3–C4–C4A	–40.3 (4)
O1–C10–C3–C2	–118.6 (4)	C4–C3–C2–C1	60.3 (4)
O2–C10–C3–C2	60.4 (4)	C9A–C1–C2–C3	–46.1 (4)

Table 2

Comparison of the bond angles ($^\circ$) in the carbazole core of (I) with the corresponding values in the related compounds (II), (IX), (X) and (XII).

Angles	(I)	(II)	(IX)	(X)	(XII)
C2–C3–C4	112.4 (3)	119.4 (3)	115.1 (2)	114.7 (2)	114.6 (5)
C4–C4A–C5A	129.1 (4)	133.6 (2)	127.5 (2)	130.9 (2)	130.4 (4)
C3–C4–C4A	111.2 (4)	117.6 (2)	114.6 (2)	115.9 (2)	116.5 (4)
C1–C9A–N9	125.1 (4)	128.7 (3)	127.5 (2)	126.4 (2)	126.8 (4)
C4A–C5A–C5	133.8 (4)	135.1 (3)	134.0 (3)	134.7 (2)	132.2 (4)
C4–C4A–C9A	123.9 (5)	120.8 (3)	124.5 (2)	122.0 (2)	121.5 (4)
N9–C8A–C8	130.8 (4)	128.1 (3)	129.4 (3)	129.8 (2)	131.0 (4)

Atom H9 was located in a difference map and refined isotropically; the positions of the other H atoms were calculated geometrically at distances of 0.93 and 0.98 (CH), 0.97 (CH₂) and 0.96 (CH₃) Å from the attached C atoms, and a riding model was used during the refinement process.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX publication routines (Farrugia, 1999).

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supporting information

Acta Cryst. (2002). E58, o1255–o1257 [https://doi.org/10.1107/S1600536802018871]

Ethyl 9*H*-1,2,3,4-tetrahydrocarbazole-3-carboxylate

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(I)

Crystal data

C₁₅H₁₇NO₂

M_r = 243.30

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 10.2221 (10) Å

b = 7.5133 (10) Å

c = 17.539 (2) Å

β = 101.603 (9)°

V = 1319.5 (3) Å³

Z = 4

F(000) = 520

D_x = 1.225 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10–18°

μ = 0.08 mm⁻¹

T = 293 K

Rod, colourless

0.30 × 0.15 × 0.15 mm

Data collection

Enraf Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

non-profiled ω scans

Absorption correction: ψ scan

(North et al., 1968)

T_{min} = 0.976, *T_{max}* = 0.988

2686 measured reflections

2686 independent reflections

653 reflections with *I* > 2σ(*I*)

R_{int} = 0.024

θ_{max} = 26.3°, θ_{min} = 2.4°

h = -12→0

k = 0→9

l = -18→21

3 standard reflections every 120 min

intensity decay: 1%

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.043

wR(*F*²) = 0.115

S = 0.92

1583 reflections

167 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 atoms treated by a mixture of independent
and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0459*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.11 e Å⁻³

Δρ_{min} = -0.15 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 of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0775 (3)	0.6499 (3)	0.17629 (14)	0.1011 (11)
O2	0.2895 (4)	0.6089 (3)	0.23494 (18)	0.0929 (11)
N9	-0.0190 (4)	0.7672 (4)	0.5127 (2)	0.0670 (11)
C1	0.1837 (4)	0.7710 (4)	0.44976 (19)	0.0691 (12)
C2	0.2005 (4)	0.7823 (4)	0.36529 (18)	0.0754 (12)
C3	0.1295 (4)	0.6329 (4)	0.31537 (19)	0.0705 (13)
C4	-0.0198 (4)	0.6330 (5)	0.3125 (2)	0.0777 (14)
C4A	-0.0497 (4)	0.6740 (4)	0.3896 (2)	0.0624 (13)
C5	-0.3015 (5)	0.6110 (5)	0.3785 (3)	0.0861 (15)
C5A	-0.1733 (4)	0.6632 (4)	0.4144 (2)	0.0642 (12)
C6	-0.4034 (5)	0.6221 (6)	0.4186 (3)	0.1003 (18)
C7	-0.3807 (5)	0.6852 (5)	0.4953 (4)	0.1016 (16)
C8	-0.2541 (6)	0.7371 (5)	0.5327 (2)	0.0830 (14)
C8A	-0.1523 (5)	0.7238 (4)	0.4918 (3)	0.0674 (13)
C9A	0.0416 (4)	0.7363 (4)	0.4505 (2)	0.0572 (12)
C10	0.1605 (5)	0.6309 (5)	0.2352 (2)	0.0744 (15)
C11	0.3266 (5)	0.6034 (5)	0.1596 (2)	0.0920 (16)
C12	0.4729 (4)	0.5831 (6)	0.1724 (3)	0.1178 (19)
H1A	0.2387	0.6758	0.4764	0.083*
H1B	0.2115	0.8818	0.4765	0.083*
H2A	0.1658	0.8955	0.3435	0.091*
H2B	0.2949	0.7782	0.3641	0.091*
H3	0.1642	0.5212	0.3405	0.085*
H4A	-0.0621	0.7209	0.2750	0.093*
H4B	-0.0563	0.5173	0.2954	0.093*
H5	-0.3179	0.5688	0.3276	0.103*
H6	-0.4890	0.5871	0.3945	0.120*
H7	-0.4514	0.6922	0.5213	0.122*
H8	-0.2382	0.7794	0.5836	0.100*
H9	0.014 (4)	0.805 (4)	0.5572 (19)	0.097 (16)*
H11A	0.2993	0.7123	0.1311	0.110*
H11B	0.2830	0.5040	0.1294	0.110*
H12A	0.5000	0.5791	0.1231	0.177*
H12B	0.4989	0.4747	0.2003	0.177*
H12C	0.5151	0.6822	0.2021	0.177*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.126 (3)	0.122 (2)	0.0495 (15)	0.0113 (18)	0.0042 (19)	0.0045 (14)
O2	0.104 (3)	0.115 (2)	0.060 (2)	-0.0009 (18)	0.017 (3)	-0.0015 (15)
N9	0.088 (3)	0.065 (2)	0.047 (2)	-0.001 (2)	0.009 (3)	-0.0025 (17)
C1	0.090 (3)	0.061 (2)	0.052 (2)	-0.004 (2)	0.005 (3)	-0.0016 (17)
C2	0.088 (3)	0.080 (3)	0.057 (2)	-0.014 (2)	0.009 (2)	-0.0052 (19)
C3	0.098 (3)	0.066 (2)	0.043 (2)	-0.004 (2)	0.006 (3)	-0.0032 (18)
C4	0.090 (4)	0.075 (2)	0.061 (3)	-0.011 (2)	0.000 (3)	-0.0052 (19)
C4A	0.084 (3)	0.053 (2)	0.044 (2)	-0.005 (2)	-0.001 (3)	-0.0011 (17)
C5	0.087 (3)	0.076 (3)	0.091 (3)	-0.007 (3)	0.008 (4)	0.007 (2)
C5A	0.073 (3)	0.051 (2)	0.064 (3)	-0.006 (2)	0.003 (3)	0.0044 (19)
C6	0.092 (4)	0.084 (3)	0.119 (5)	-0.001 (3)	0.008 (4)	0.007 (3)
C7	0.091 (4)	0.073 (3)	0.147 (5)	0.009 (3)	0.039 (4)	0.015 (3)
C8	0.104 (4)	0.058 (2)	0.092 (3)	0.007 (3)	0.030 (4)	0.003 (2)
C8A	0.084 (4)	0.046 (2)	0.071 (3)	0.009 (2)	0.012 (3)	0.005 (2)
C9A	0.075 (3)	0.052 (2)	0.045 (2)	0.002 (2)	0.013 (3)	0.0039 (18)
C10	0.108 (4)	0.068 (2)	0.046 (3)	-0.005 (3)	0.013 (4)	-0.001 (2)
C11	0.132 (5)	0.095 (3)	0.054 (3)	-0.007 (3)	0.030 (4)	-0.009 (2)
C12	0.112 (4)	0.160 (4)	0.090 (3)	-0.001 (3)	0.039 (4)	-0.007 (3)

Geometric parameters (\AA , $^\circ$)

C9A—C4A	1.352 (4)	C4—H4A	0.9700
C9A—N9	1.378 (4)	C4—H4B	0.9700
C9A—C1	1.478 (4)	C2—H2A	0.9700
O2—C10	1.329 (4)	C2—H2B	0.9700
O2—C11	1.448 (4)	C5A—C5	1.391 (5)
N9—C8A	1.377 (5)	C5—C6	1.372 (5)
N9—H9	0.84 (3)	C5—H5	0.9300
C10—O1	1.206 (4)	C11—C12	1.475 (5)
C10—C3	1.503 (5)	C11—H11A	0.9700
C1—C2	1.528 (4)	C11—H11B	0.9700
C1—H1A	0.9700	C7—C8	1.384 (5)
C1—H1B	0.9700	C7—C6	1.400 (5)
C3—C4	1.516 (5)	C7—H7	0.9300
C3—C2	1.516 (4)	C6—H6	0.9300
C3—H3	0.9800	C8—H8	0.9300
C8A—C8	1.381 (5)	C12—H12A	0.9600
C8A—C5A	1.407 (5)	C12—H12B	0.9600
C4A—C5A	1.419 (5)	C12—H12C	0.9600
C4A—C4	1.477 (4)		
C4A—C9A—N9	109.6 (4)	C3—C2—C1	112.6 (3)
C4A—C9A—C1	125.3 (4)	C3—C2—H2A	109.1
N9—C9A—C1	125.1 (4)	C1—C2—H2A	109.1
C10—O2—C11	116.7 (4)	C3—C2—H2B	109.1

C8A—N9—C9A	109.2 (4)	C1—C2—H2B	109.1
C8A—N9—H9	122 (3)	H2A—C2—H2B	107.8
C9A—N9—H9	129 (3)	C5—C5A—C8A	118.4 (5)
O1—C10—O2	122.6 (5)	C5—C5A—C4A	133.8 (4)
O1—C10—C3	123.7 (5)	C8A—C5A—C4A	107.8 (4)
O2—C10—C3	113.6 (4)	C6—C5—C5A	119.6 (4)
C9A—C1—C2	108.7 (3)	C6—C5—H5	120.2
C9A—C1—H1A	109.9	C5A—C5—H5	120.2
C2—C1—H1A	109.9	O2—C11—C12	107.9 (4)
C9A—C1—H1B	109.9	O2—C11—H11A	110.1
C2—C1—H1B	109.9	C12—C11—H11A	110.1
H1A—C1—H1B	108.3	O2—C11—H11B	110.1
C10—C3—C4	111.7 (4)	C12—C11—H11B	110.1
C10—C3—C2	112.4 (3)	H11A—C11—H11B	108.4
C4—C3—C2	112.4 (3)	C8—C7—C6	120.6 (5)
C10—C3—H3	106.6	C8—C7—H7	119.7
C4—C3—H3	106.6	C6—C7—H7	119.7
C2—C3—H3	106.6	C5—C6—C7	121.1 (5)
N9—C8A—C8	130.8 (4)	C5—C6—H6	119.4
N9—C8A—C5A	106.5 (4)	C7—C6—H6	119.4
C8—C8A—C5A	122.7 (4)	C8A—C8—C7	117.6 (4)
C9A—C4A—C5A	107.0 (4)	C8A—C8—H8	121.2
C9A—C4A—C4	123.9 (5)	C7—C8—H8	121.2
C5A—C4A—C4	129.1 (4)	C11—C12—H12A	109.5
C4A—C4—C3	111.2 (4)	C11—C12—H12B	109.5
C4A—C4—H4A	109.4	H12A—C12—H12B	109.5
C3—C4—H4A	109.4	C11—C12—H12C	109.5
C4A—C4—H4B	109.4	H12A—C12—H12C	109.5
C3—C4—H4B	109.4	H12B—C12—H12C	109.5
H4A—C4—H4B	108.0		
C4A—C9A—N9—C8A	0.2 (3)	C10—C3—C2—C1	-172.7 (3)
C1—C9A—N9—C8A	178.5 (3)	C4—C3—C2—C1	60.3 (4)
C11—O2—C10—O1	-1.8 (5)	C9A—C1—C2—C3	-46.1 (4)
C11—O2—C10—C3	179.2 (3)	N9—C8A—C5A—C5	-179.7 (3)
C4A—C9A—C1—C2	17.6 (4)	C8—C8A—C5A—C5	-1.5 (5)
N9—C9A—C1—C2	-160.4 (3)	N9—C8A—C5A—C4A	-0.7 (3)
O1—C10—C3—C4	8.8 (5)	C8—C8A—C5A—C4A	177.4 (3)
O2—C10—C3—C4	-172.3 (3)	C9A—C4A—C5A—C5	179.5 (4)
O1—C10—C3—C2	-118.6 (4)	C4—C4A—C5A—C5	1.3 (6)
O2—C10—C3—C2	60.4 (4)	C9A—C4A—C5A—C8A	0.8 (4)
C9A—N9—C8A—C8	-177.6 (3)	C4—C4A—C5A—C8A	-177.4 (3)
C9A—N9—C8A—C5A	0.4 (3)	C8A—C5A—C5—C6	0.9 (5)
N9—C9A—C4A—C5A	-0.6 (4)	C4A—C5A—C5—C6	-177.6 (4)
C1—C9A—C4A—C5A	-178.9 (3)	C10—O2—C11—C12	178.5 (3)
N9—C9A—C4A—C4	177.7 (3)	C5A—C5—C6—C7	0.0 (6)
C1—C9A—C4A—C4	-0.6 (5)	C8—C7—C6—C5	-0.4 (6)
C9A—C4A—C4—C3	11.7 (4)	N9—C8A—C8—C7	178.8 (3)

C5A—C4A—C4—C3	-170.3 (3)	C5A—C8A—C8—C7	1.1 (5)
C10—C3—C4—C4A	-167.7 (3)	C6—C7—C8—C8A	-0.1 (5)
C2—C3—C4—C4A	-40.3 (4)		
