

9-Ethyl-3-methyl-1,6-dinitrocarbazole

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The title compound, C₁₅H₁₃N₃O₄, crystallizes in the triclinic space group *P* $\bar{1}$. The 6-nitro and methyl groups are essentially planar with the carbazole moiety, while the 1-nitro group is twisted out of the carbazole plane. Two neighboring molecules are associated with each other through one benzene ring, indicating a weak π – π interaction.

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean σ (C–C) = 0.003 Å

R factor = 0.042

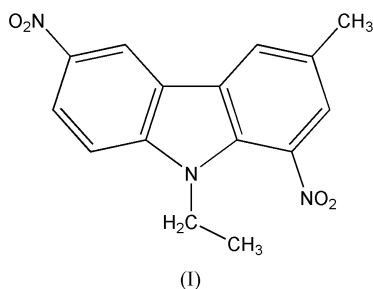
wR factor = 0.116

Data-to-parameter ratio = 12.2

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

Comment

Aminocarbazoles are widely used as intermediates for the preparation of carbazole-based synthetic dyes, agrochemicals, pharmaceuticals, light-sensitive materials, surfactants, and polymers (Shufen *et al.*, 1995). Aminocarbazoles can easily be prepared by the reduction of nitrocarbazoles, while photochemical nitration of carbazoles proceeds readily through an electron-transfer process between the electron donor–acceptor (EDA) complexes of carbazoles with tetranitromethane (TNM) (Iles & Ledwith, 1969). Nitration on the unsubstituted benzene rings of carbazoles proceeds *via* the introduction of the first nitro group mainly at the C3 position. Introduction of the second nitro group at the C6 position on the second benzene ring proceeds more slowly at the same conditions due to the reduced electron density of the π -system due to the presence of the first nitro group. It came to our attention (Asker, 2001) that the existence of an alkyl group at the C3 position on any of the benzene rings significantly activates the C1 position on the same ring. In our attempts to elucidate the nature of the nitration process, we have prepared the title compound, (I), and undertaken a single-crystal X-ray structure determination.



The title compound, (I) (Fig. 1), crystallizes in the space group *P* $\bar{1}$. Bond distances and angles are in agreement with those for related compounds (Baker *et al.*, 1991; Chen *et al.*, 1992). However, compared to unnitrated carbazole rings, the C1–C9A [1.404 (3) Å] and C6–C7 [1.396 (3) Å] bonds at the attachment centers of the nitro groups are found to be slightly longer. Similarly, the C1–C2–C3 [122. (2)°] and C5–C6–C7 [124 (2)°] interior angles are also found to be slightly greater (Table 1).

The 1-nitrated benzene ring of one molecule associates with the 3-nitrated benzene ring of a second molecule in a way that their nitro groups are pointing to the opposite directions indicating a weak π - π interaction while forming C—H...O interactions with a third molecule (Fig. 2).

The 6-nitro substituent is coplanar with the carbazole ring system with the a dihedral angle between the two planes of $5.27 (15)^\circ$, while steric interaction with the ethyl group result in the plane of the 1-nitro group twisting out of the carbazolyl plane with a dihedral angle between the two planes of $37.24 (9)^\circ$. Atoms N1, N6, C10 and C12 deviate from the carbazole best least-squares plane by $0.1750 (24)$, $-0.0950 (25)$, $-0.279 (3)$ and $-0.027 (3)$ Å, respectively.

Experimental

Nitration of 9-ethyl-3-methylcarbazole was performed through a photochemical reaction using TNM as the nitrating agent in dichloromethane. A Westinghouse sun lamp (275 W) was used as the light source. The reaction was carried out in a 25 ml test tube dissolving 100 mg (0.5 mmol) of 9-ethyl-3-methylcarbazole and 500 mg (2.5 mmol) of TNM in 5 ml of dichloromethane. The light source was placed at a distance of approximately 15 cm from the reaction tube and a Corning sharp cutoff UV filter was placed between the light source and the test tube. After 3 h of irradiation, the reaction mixture was extracted with water, the solvent was removed under reduced pressure, and the remaining yellow solid was column chromatographed using basic alumina (80–200 mesh, activity III) and dichloromethane/hexane as the eluting solvents. The title compound, (I), was obtained after recrystallization from CH_2Cl_2 as yellow needles (m.p. 429 K). $^1\text{H NMR}$ (300 MHz, CDCl_3 , p.p.m.): 8.99 (*d*, 2.17 Hz, 1H), 8.45 (*d of d*, 8.96 and 2.19 Hz, 1H), 8.20 (*s*, 1H), 7.88 (*s*, 1H), 7.54 (*d*, 9.14 Hz, 1H), 4.42 (*q*, 7.04 Hz, 2H), 2.61 (*s*, 3H), 1.43 (*t*, 7.13 Hz, 3H).

Crystal data

$\text{C}_{15}\text{H}_{13}\text{N}_3\text{O}_4$	$Z = 2$
$M_r = 299.28$	$D_x = 1.442 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.7094 (4) \text{ \AA}$	Cell parameters from 25 reflections
$b = 8.8147 (6) \text{ \AA}$	$\theta = 6.1\text{--}14.0^\circ$
$c = 12.2004 (9) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$\alpha = 72.908 (6)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 87.720 (6)^\circ$	Slab, yellow
$\gamma = 88.718 (6)^\circ$	$0.51 \times 0.20 \times 0.12 \text{ mm}$
$V = 689.09 (8) \text{ \AA}^3$	

Data collection

Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 25.0^\circ$
ω scans	$h = -7 \rightarrow 7$
Absorption correction: none	$k = 0 \rightarrow 10$
2698 measured reflections	$l = -13 \rightarrow 14$
2442 independent reflections	3 standard reflections
1692 reflections with $I_{\text{net}} > 2\sigma(I_{\text{net}})$	frequency: 120 min
$R_{\text{int}} = 0.007$	intensity decay: 1.2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.1899P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.116$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
2442 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
200 parameters	
H-atom parameters constrained	

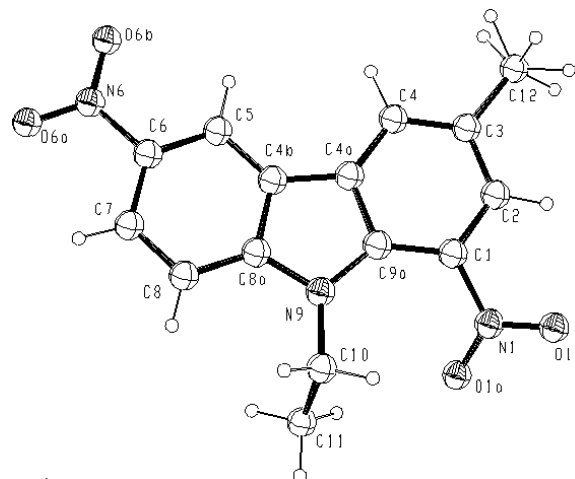


Figure 1
ORTEP (Johnson, 1976) drawing of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. H atoms are represented by small spheres.

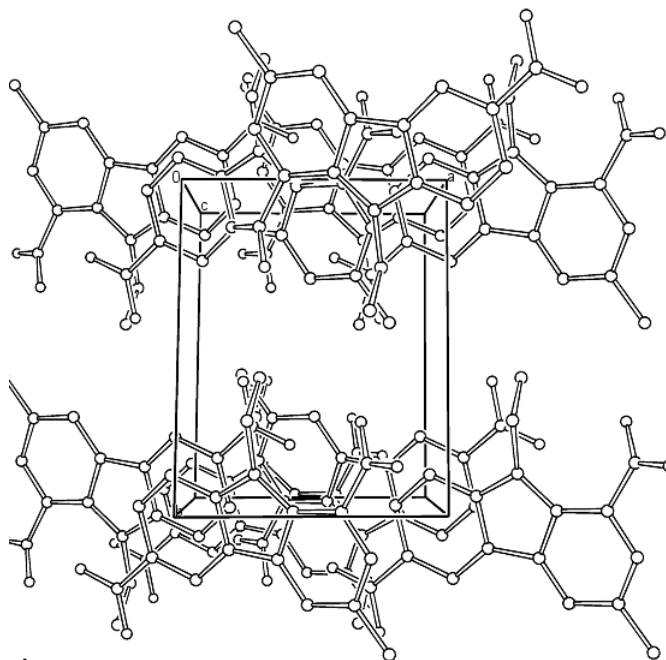


Figure 2
The molecular packing of (I). H atoms have been omitted for clarity.

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.384 (3)	C4B—C5	1.382 (3)
C1—C9A	1.404 (3)	C4B—C8A	1.411 (3)
C2—C3	1.385 (3)	C5—C6	1.374 (3)
C3—C4	1.389 (3)	C6—C7	1.396 (3)
C4—C4A	1.383 (3)	C7—C8	1.374 (3)
C4A—C9A	1.414 (3)	C8—C8A	1.395 (3)
C4A—C4B	1.444 (3)	C10—C11	1.505 (3)
C2—C1—C9A	120.07 (19)	C6—C5—C4B	117.50 (19)
C1—C2—C3	122.4 (2)	C5—C6—C7	123.19 (19)
C2—C3—C4	118.3 (2)	C8—C7—C6	119.64 (19)
C4A—C4—C3	120.1 (2)	C7—C8—C8A	118.28 (19)
C4—C4A—C9A	122.05 (19)	C8—C8A—C4B	121.21 (19)
C5—C4B—C8A	120.16 (18)	C1—C9A—C4A	117.00 (19)

H atoms were located by difference Fourier techniques and allowed to ride on their parent atoms at distances of 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, with $U_{\text{iso}}(\text{H}) = 0.001 \text{ \AA}^2 + U_{\text{eq}}(\text{C})$ of the parent atom. The methyl group shows disorder of the H atoms; it was treated as an idealized disordered methyl group with the site-occupation factors fixed at 0.5.

Data collection: *CAD-4 PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4 PC Software*; data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *NRCVAX SOLVER*; program(s) used to refine structure: *NRCVAX LSTSQ* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) in *NRCVAX*; software used to prepare material for publication: *SHELXL97*.

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supporting information

Acta Cryst. (2004). E60, o1613–o1615 [https://doi.org/10.1107/S1600536804020574]

9-Ethyl-3-methyl-1,6-dinitrocarbazole

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9-Ethyl-3-methyl-1,6-dinitro-9H-carbazole

Crystal data

$C_{15}H_{13}N_3O_4$
 $M_r = 299.28$
 Triclinic, $P\bar{1}$
 Hall symbol: -P 1
 $a = 6.7094$ (4) Å
 $b = 8.8147$ (6) Å
 $c = 12.2004$ (9) Å
 $\alpha = 72.908$ (6)°
 $\beta = 87.720$ (6)°
 $\gamma = 88.718$ (6)°
 $V = 689.09$ (8) Å³
 $Z = 2$

$F(000) = 312$
 ? #Insert any comments here.
 $D_x = 1.442$ Mg m⁻³
 Melting point: 429 K
 Mo $K\alpha$ radiation, $\lambda = 0.70930$ Å
 Cell parameters from 25 reflections
 $\theta = 6.1$ – 14.0 °
 $\mu = 0.11$ mm⁻¹
 $T = 293$ K
 Needle, yellow
 $0.51 \times 0.20 \times 0.12$ mm

Data collection

Nonius CAD-4
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 2698 measured reflections
 2442 independent reflections
 1692 reflections with $I_{net} > 2\sigma(I_{net})$

$R_{int} = 0.007$
 $\theta_{max} = 25.0$ °, $\theta_{min} = 1.7$ °
 $h = -7 \rightarrow 7$
 $k = 0 \rightarrow 10$
 $l = -13 \rightarrow 14$
 3 standard reflections every 120 min
 intensity decay: 1.2%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.116$
 $S = 1.02$
 2442 reflections
 200 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
 $w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.1899P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.14$ e Å⁻³
 $\Delta\rho_{min} = -0.17$ 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.

TABLE of LEAST SQUARES PLANES

----- Plane No. 1 -----

Equation of the plane: $3.4329(15)X + 4.087(4)Y + 10.3793(21)Z = 3.4664(9)$

Distances(A) to the plane from the atoms in the plane. N9 - 0.0091 (18) C1 0.0074 (22) C2 - 0.0407 (24) C3 - 0.0330 (24) C4 0.0069 (22) C4a 0.0339 (20) C4b 0.0332 (19) C5 0.0122 (22) C6 - 0.0266 (22) C7 - 0.0258 (23) C8 - 0.0083 (22) C8a 0.0118 (20) C9a 0.0220 (21)

Chi squared for this plane 1568.151

Distances(A) to the plane from the atoms out of the plane. N1 0.1750 (24) N6 - 0.0950 (25) C10 - 0.279 (3) C12 - 0.027 (3)

----- Plane No. 2 -----

Equation of the plane: $5.368(12)X - 0.713(24)Y + 6.931(16)Z = 4.078(13)$

Distances(A) to the plane from the atoms in the plane. N1 0.000 (3) O1a 0.0000 (22) O1b 0.0000 (22)

----- Plane No. 3 -----

Equation of the plane: $3.472(17)X + 3.382(17)Y + 10.562(21)Z = 3.633(21)$

Distances(A) to the plane from the atoms in the plane. N6 0.000 (3) O6a 0.0000 (23) O6b 0.0000 (23)

----- Plane No. 4 -----

Equation of the plane: $6.336(7)X - 0.662(11)Y - 3.55(4)Z = 1.026(10)$

Distances(A) to the plane from the atoms in the plane. N9 0.0000 (22) C10 0.000 (3) C11 0.000 (4)

----- Dihedral angle between planes A and B

A B Angle(°) 1 2 37.24 (9) 1 3 5.27 (15) 1 4 77.86 (17) 2 3 33.58 (10) 2 4 54.63 (23) 3 4 77.85 (25)

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1A	0.6810 (3)	0.2723 (2)	0.08878 (15)	0.0680 (5)	
O1B	0.8095 (3)	0.1447 (2)	-0.02367 (16)	0.0776 (6)	
O6A	-0.4115 (3)	-0.1958 (2)	0.54203 (16)	0.0796 (6)	
O6B	-0.2239 (3)	-0.3960 (2)	0.54437 (17)	0.0837 (6)	
N1	0.7038 (3)	0.1512 (3)	0.05920 (16)	0.0542 (5)	
N6	-0.2610 (3)	-0.2535 (2)	0.51102 (16)	0.0557 (5)	
N9	0.2828 (3)	0.11477 (19)	0.19406 (14)	0.0431 (4)	
C1	0.6113 (3)	0.0053 (2)	0.13046 (17)	0.0441 (5)	
C2	0.7224 (3)	-0.1326 (3)	0.14370 (19)	0.0503 (6)	
H2	0.8401	-0.1286	0.1000	0.060*	
C3	0.6650 (3)	-0.2765 (3)	0.2196 (2)	0.0495 (6)	
C4	0.4884 (3)	-0.2814 (2)	0.28391 (19)	0.0463 (5)	
H4	0.4472	-0.3764	0.3360	0.056*	
C4A	0.3738 (3)	-0.1452 (2)	0.27068 (17)	0.0406 (5)	
C4B	0.1846 (3)	-0.1191 (2)	0.32324 (17)	0.0400 (5)	
C5	0.0552 (3)	-0.2172 (2)	0.40242 (17)	0.0430 (5)	
H5	0.0853	-0.3240	0.4355	0.052*	
C6	-0.1196 (3)	-0.1510 (2)	0.43046 (17)	0.0435 (5)	

C7	-0.1698 (3)	0.0091 (3)	0.38410 (18)	0.0467 (5)	
H7	-0.2888	0.0491	0.4067	0.056*	
C8	-0.0423 (3)	0.1072 (2)	0.30493 (18)	0.0463 (5)	
H8	-0.0734	0.2141	0.2730	0.056*	
C8A	0.1347 (3)	0.0423 (2)	0.27372 (17)	0.0409 (5)	
C9A	0.4304 (3)	0.0021 (2)	0.19297 (17)	0.0409 (5)	
C10	0.2544 (4)	0.2749 (3)	0.1143 (2)	0.0551 (6)	
H10A	0.1179	0.2864	0.0895	0.066*	
H10B	0.3415	0.2868	0.0470	0.066*	
C11	0.2973 (4)	0.4046 (3)	0.1671 (2)	0.0719 (8)	
H11A	0.2752	0.5061	0.1121	0.108*	
H11B	0.4335	0.3961	0.1896	0.108*	
H11C	0.2104	0.3943	0.2333	0.108*	
C12	0.7964 (4)	-0.4229 (3)	0.2344 (2)	0.0685 (7)	
H12A	0.7346	-0.5116	0.2898	0.103*	0.50
H12B	0.9241	-0.4043	0.2606	0.103*	0.50
H12C	0.8137	-0.4455	0.1623	0.103*	0.50
H12D	0.9136	-0.3960	0.1853	0.103*	0.50
H12E	0.7242	-0.5033	0.2145	0.103*	0.50
H12F	0.8346	-0.4621	0.3128	0.103*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0670 (12)	0.0528 (10)	0.0792 (12)	-0.0106 (9)	0.0062 (9)	-0.0119 (9)
O1B	0.0636 (12)	0.0942 (14)	0.0665 (11)	-0.0116 (10)	0.0259 (10)	-0.0130 (10)
O6A	0.0573 (12)	0.0864 (13)	0.0840 (13)	0.0101 (10)	0.0310 (10)	-0.0129 (10)
O6B	0.0825 (14)	0.0548 (11)	0.0975 (15)	-0.0010 (10)	0.0400 (11)	-0.0026 (10)
N1	0.0390 (11)	0.0673 (14)	0.0512 (11)	-0.0076 (10)	0.0029 (9)	-0.0096 (10)
N6	0.0489 (13)	0.0616 (13)	0.0530 (11)	-0.0010 (10)	0.0117 (10)	-0.0128 (10)
N9	0.0395 (10)	0.0387 (9)	0.0477 (10)	-0.0008 (8)	0.0031 (8)	-0.0079 (8)
C1	0.0372 (12)	0.0493 (13)	0.0455 (12)	-0.0075 (10)	0.0030 (10)	-0.0137 (10)
C2	0.0371 (12)	0.0624 (15)	0.0570 (14)	-0.0021 (11)	0.0050 (10)	-0.0271 (12)
C3	0.0394 (13)	0.0525 (13)	0.0628 (14)	0.0037 (10)	-0.0015 (11)	-0.0269 (11)
C4	0.0419 (13)	0.0413 (12)	0.0566 (13)	-0.0004 (10)	0.0015 (11)	-0.0161 (10)
C4A	0.0361 (11)	0.0402 (11)	0.0468 (12)	-0.0013 (9)	0.0010 (9)	-0.0150 (9)
C4B	0.0362 (12)	0.0406 (11)	0.0434 (11)	-0.0002 (9)	0.0012 (9)	-0.0130 (9)
C5	0.0429 (13)	0.0390 (12)	0.0457 (12)	0.0005 (10)	0.0018 (10)	-0.0107 (9)
C6	0.0383 (12)	0.0484 (12)	0.0429 (11)	-0.0014 (10)	0.0048 (9)	-0.0128 (10)
C7	0.0364 (12)	0.0545 (13)	0.0510 (12)	0.0059 (10)	0.0012 (10)	-0.0192 (10)
C8	0.0444 (13)	0.0401 (12)	0.0545 (13)	0.0069 (10)	-0.0026 (10)	-0.0142 (10)
C8A	0.0365 (12)	0.0423 (11)	0.0449 (12)	-0.0003 (9)	-0.0016 (9)	-0.0142 (9)
C9A	0.0366 (12)	0.0446 (12)	0.0435 (11)	-0.0011 (9)	-0.0016 (9)	-0.0157 (9)
C10	0.0476 (14)	0.0520 (14)	0.0573 (14)	0.0011 (11)	0.0003 (11)	-0.0035 (11)
C11	0.0808 (19)	0.0473 (14)	0.0827 (19)	-0.0008 (13)	0.0083 (15)	-0.0131 (13)
C12	0.0522 (15)	0.0608 (16)	0.099 (2)	0.0120 (12)	0.0028 (14)	-0.0350 (15)

Geometric parameters (Å, °)

O1A—N1	1.228 (2)	C4B—C8A	1.411 (3)
O1B—N1	1.226 (2)	C5—C6	1.374 (3)
O6A—N6	1.217 (2)	C5—H5	0.9300
O6B—N6	1.224 (2)	C6—C7	1.396 (3)
N1—C1	1.460 (3)	C7—C8	1.374 (3)
N6—C6	1.458 (3)	C7—H7	0.9300
N9—C8A	1.388 (3)	C8—C8A	1.395 (3)
N9—C9A	1.390 (3)	C8—H8	0.9300
N9—C10	1.473 (3)	C10—C11	1.505 (3)
C1—C2	1.384 (3)	C10—H10A	0.9700
C1—C9A	1.404 (3)	C10—H10B	0.9700
C2—C3	1.385 (3)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C3—C4	1.389 (3)	C11—H11C	0.9600
C3—C12	1.516 (3)	C12—H12A	0.9600
C4—C4A	1.383 (3)	C12—H12B	0.9600
C4—H4	0.9300	C12—H12C	0.9600
C4A—C9A	1.414 (3)	C12—H12D	0.9600
C4A—C4B	1.444 (3)	C12—H12E	0.9600
C4B—C5	1.382 (3)	C12—H12F	0.9600
O1B—N1—O1A	123.2 (2)	N9—C8A—C8	129.08 (19)
O1B—N1—C1	118.3 (2)	N9—C8A—C4B	109.70 (17)
O1A—N1—C1	118.41 (19)	C8—C8A—C4B	121.21 (19)
O6A—N6—O6B	122.3 (2)	N9—C9A—C1	133.76 (19)
O6A—N6—C6	119.5 (2)	N9—C9A—C4A	109.20 (17)
O6B—N6—C6	118.22 (18)	C1—C9A—C4A	117.00 (19)
C8A—N9—C9A	108.03 (16)	N9—C10—C11	112.8 (2)
C8A—N9—C10	121.53 (17)	N9—C10—H10A	109.0
C9A—N9—C10	129.51 (18)	C11—C10—H10A	109.0
C2—C1—C9A	120.07 (19)	N9—C10—H10B	109.0
C2—C1—N1	116.07 (19)	C11—C10—H10B	109.0
C9A—C1—N1	123.54 (19)	H10A—C10—H10B	107.8
C1—C2—C3	122.4 (2)	C10—C11—H11A	109.5
C1—C2—H2	118.8	C10—C11—H11B	109.5
C3—C2—H2	118.8	H11A—C11—H11B	109.5
C2—C3—C4	118.3 (2)	C10—C11—H11C	109.5
C2—C3—C12	120.4 (2)	H11A—C11—H11C	109.5
C4—C3—C12	121.3 (2)	H11B—C11—H11C	109.5
C4A—C4—C3	120.1 (2)	C3—C12—H12A	109.5
C4A—C4—H4	119.9	C3—C12—H12B	109.5
C3—C4—H4	119.9	H12A—C12—H12B	109.5
C4—C4A—C9A	122.05 (19)	C3—C12—H12C	109.5
C4—C4A—C4B	131.18 (19)	H12A—C12—H12C	109.5
C9A—C4A—C4B	106.74 (18)	H12B—C12—H12C	109.5
C5—C4B—C8A	120.16 (18)	C3—C12—H12D	109.5

C5—C4B—C4A	133.50 (19)	H12A—C12—H12D	141.1
C8A—C4B—C4A	106.30 (18)	H12B—C12—H12D	56.3
C6—C5—C4B	117.50 (19)	H12C—C12—H12D	56.3
C6—C5—H5	121.3	C3—C12—H12E	109.5
C4B—C5—H5	121.3	H12A—C12—H12E	56.3
C5—C6—C7	123.19 (19)	H12B—C12—H12E	141.1
C5—C6—N6	118.38 (19)	H12C—C12—H12E	56.3
C7—C6—N6	118.42 (19)	H12D—C12—H12E	109.5
C8—C7—C6	119.64 (19)	C3—C12—H12F	109.5
C8—C7—H7	120.2	H12A—C12—H12F	56.3
C6—C7—H7	120.2	H12B—C12—H12F	56.3
C7—C8—C8A	118.28 (19)	H12C—C12—H12F	141.1
C7—C8—H8	120.9	H12D—C12—H12F	109.5
C8A—C8—H8	120.9	H12E—C12—H12F	109.5
O1B—N1—C1—C2	34.9 (3)	C6—C7—C8—C8A	-0.1 (3)
O1A—N1—C1—C2	-141.1 (2)	C9A—N9—C8A—C8	179.9 (2)
O1B—N1—C1—C9A	-151.7 (2)	C10—N9—C8A—C8	-10.1 (3)
O1A—N1—C1—C9A	32.3 (3)	C9A—N9—C8A—C4B	-1.1 (2)
C9A—C1—C2—C3	-1.7 (3)	C10—N9—C8A—C4B	168.90 (18)
N1—C1—C2—C3	172.0 (2)	C7—C8—C8A—N9	177.8 (2)
C1—C2—C3—C4	0.3 (3)	C7—C8—C8A—C4B	-1.1 (3)
C1—C2—C3—C12	-177.5 (2)	C5—C4B—C8A—N9	-177.66 (18)
C2—C3—C4—C4A	0.6 (3)	C4A—C4B—C8A—N9	0.2 (2)
C12—C3—C4—C4A	178.4 (2)	C5—C4B—C8A—C8	1.4 (3)
C3—C4—C4A—C9A	-0.2 (3)	C4A—C4B—C8A—C8	179.30 (19)
C3—C4—C4A—C4B	177.8 (2)	C8A—N9—C9A—C1	179.2 (2)
C4—C4A—C4B—C5	-0.1 (4)	C10—N9—C9A—C1	10.3 (4)
C9A—C4A—C4B—C5	178.2 (2)	C8A—N9—C9A—C4A	1.5 (2)
C4—C4A—C4B—C8A	-177.6 (2)	C10—N9—C9A—C4A	-167.38 (19)
C9A—C4A—C4B—C8A	0.7 (2)	C2—C1—C9A—N9	-175.6 (2)
C8A—C4B—C5—C6	-0.4 (3)	N1—C1—C9A—N9	11.2 (4)
C4A—C4B—C5—C6	-177.6 (2)	C2—C1—C9A—C4A	2.0 (3)
C4B—C5—C6—C7	-0.8 (3)	N1—C1—C9A—C4A	-171.21 (18)
C4B—C5—C6—N6	178.08 (19)	C4—C4A—C9A—N9	177.09 (19)
O6A—N6—C6—C5	175.9 (2)	C4B—C4A—C9A—N9	-1.4 (2)
O6B—N6—C6—C5	-4.7 (3)	C4—C4A—C9A—C1	-1.1 (3)
O6A—N6—C6—C7	-5.2 (3)	C4B—C4A—C9A—C1	-179.53 (18)
O6B—N6—C6—C7	174.2 (2)	C8A—N9—C10—C11	83.1 (3)
C5—C6—C7—C8	1.1 (3)	C9A—N9—C10—C11	-109.3 (3)
N6—C6—C7—C8	-177.8 (2)		