# organic compounds

# 11-Butyl-3-methoxy-11*H*-benzo[*a*]-carbazole

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The title compound, $C_{21}H_{21}NO$, consists of a carbazole skeleton with a methoxybenzene ring fused to the carbazole, and a butyl group attached to the carbazole N atom. The carbazole skeleton is nearly planar [maximum deviation = 0.078 (2) Å], and it is oriented at a dihedral angle of 4.22 (4)° with respect to the adjacent methoxybenzene ring.

## Related literature

For the biological activity of carbazole derivatives, see: Knölker & Reddy (2002). For the use of carbazole derivatives in the syntheses of indole alkaloids, see: Routier *et al.* (2001). For the use of benzo[*a*]carbazoles in cancer treatment, see: Carini *et al.* (2001). For the antitumor activity of a series of simple benzo[*a*]carbazoles against mammary tumors of rats, leukemia, renal tumors, colon cancer and malignant melanoma tumor cell lines, see: von Angerer & Prekajac (1986); Pindur & Lemster (1997). For the extensive application of benzo[*a*]carbazole derivatives as photographic materials, see: Oliveira *et al.* (2005. For tetrahydrocarbazole systems present in the frameworks of a number of indole-type alkaloids of biological interest, see: Phillipson & Zenk (1980); Saxton (1983); Abraham (1975). For related structures, see: Hökelek *et al.* (1994, 1998, 1999, 2004, 2006); Patır *et al.* (1997); Hökelek & Patır (1999, 2002); Çaylak *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).
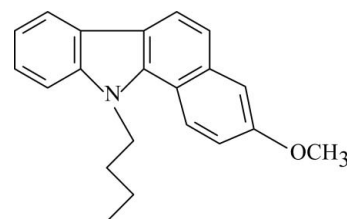
## Experimental

### Crystal data

| | |
|---|---|
| $C_{21}H_{21}NO$ | $V$ = 820.46 (8) Å$^3$ |
| $M_r$ = 303.39 | $Z$ = 2 |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation |
| $a$ = 10.7263 (6) Å | $\mu$ = 0.08 mm$^{-1}$ |
| $b$ = 5.5562 (3) Å | $T$ = 100 K |
| $c$ = 13.8967 (7) Å | 0.48 × 0.39 × 0.35 mm |
| $\beta$ = 97.841 (2)° | |

### Data collection

| | |
|---|---|
| Bruker Kappa APEXII CCD area-detector diffractometer | 7947 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | 2248 independent reflections |
| $T_{min}$ = 0.965, $T_{max}$ = 0.974 | 2001 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.027 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.035 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.091 | |
| $S$ = 1.04 | $\Delta\rho_{max}$ = 0.25 e Å$^{-3}$ |
| 2248 reflections | $\Delta\rho_{min}$ = −0.16 e Å$^{-3}$ |
| 270 parameters | |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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# supporting information

## 11-Butyl-3-methoxy-11*H*-benzo[*a*]carbazole

**Yavuz Ergün, Cevher Gündoğdu, Barış Tercan, Emel Ermiş and Tuncer Hökelek**

### S1. Comment

Carbazole derivatives display a range of biological activities, making them attractive compounds to synthetic and medicinal chemists (Knölker & Reddy, 2002). They also have an important role in the syntheses of indole alkaloids, in which their synthetic derivatives, possessing useful pharmacological properties, are currently under investigation (Routier *et al.*, 2001). Benzo -annulated carbazole ring systems are found only rarely in natural products. The benzo[*a*]carbazoles, containing an aromatic ring fused to the a-face of the carbazole nucleus, are potential candidates for cancer treatment as a result of DNA intercelative binding properties (Carini *et al.*, 2001). A series of simple benzo[*a*]carbazoles have been shown to bind to estrogen receptors and inhibit the growth of mammary tumors of rats (Angerer & Prekajac, 1986). Some benzo[*a*]carbazoles exhibit a pronounced antitumor activity against leukemia, renal tumor, colon cancer, and malignant melanoma tumor cell lines (Pindur & Lemster, 1997). Benzo[*a*]carbazole derivatives have also found extensive application as photographic materials (Oliveira *et al.*, 2005).

Tetrahydrocarbazole systems are present in the framework of a number of indole-type alkaloids of biological interest (Phillipson & Zenk, 1980; Saxton, 1983; Abraham, 1975). The structures of tricyclic, tetracyclic and pentacyclic ring systems with dithiolane and other substituents of the tetrahydrocarbazole core, have been the subject of much interest in our laboratory. These include 1,2,3,4-tetrahydrocarbazole-1-spiro-2′-[1,3]dithiolane, (II) (Hökelek *et al.*, 1994), *N*-(2-methoxyethyl)-*N*-{2,3,4,9-tetrahydrospiro[1*H*-carbazole-1, 2-(1,3)dithiolane]-4-yl}benzene-sulfonamide, (III) (Patır *et al.*, 1997), spiro[carbazole-1(2*H*),2′-[1,3]-dithiolan]-4(3*H*)-one, (IV) (Hökelek *et al.*, 1998), 9-acetonyl-3-ethyl-idene-1,2,3,4-tetrahydrospiro[carbazole-1,2′-[1,3] dithiolan]-4-one, (V) (Hökelek *et al.*, 1999), *N*-(2,2-dimethoxyethyl)-N-{9-methoxymethyl-1,2,3,4-tetrahydrospiro[carbazole-1,2′-[1,3]dithiolan] -4-yl}benzamide, (VI) (Hökelek & Patır, 1999), 3a,4,10,10*b*-tetrahydro-2H -furo[2,3-*a*]carbazol-5(3*H*)-one, (VII) (Çaylak *et al.*, 2007); also the pentacyclic compounds 6-ethyl-4-(2-methoxyethyl)-2,6-methano-5-oxo-hexahydro- pyrrolo(2,3 - d)carbazole-1-spiro-2′-(1,3)dithiol-ane, (VIII) (Hökelek & Patır, 2002), *N*-(2-benzyloxyethyl)-4,7-dimethyl-6-(1,3-dithiolan-2-yl)-1,2, 3,4,5,6-hexa-hydro-1,5-methano-2-azocino[4,3-*b*]indol-2-one, (IX) (Hökelek *et al.*, 2004) and 4-ethyl-6,6-ethylenedithio-2-(2-meth-oxyethyl)-7-methoxy- methylene-2,3,4,5,6,7-hexahydro-1,5-methano-1*H*-azocino[4,3-*b*]indol-3-one, (*X*) (Hökelek *et al.*, 2006). The title compound, (I), may be considered as a synthetic precursor of tetracyclic indole alkaloids of biological interests. The present study was undertaken to ascertain its crystal structure.

The title compound consists of a carbazole skeleton with a methoxy benzoato group fused to the a-face of the carbazole nucleus, and a butyl group attached to atom N9 (Fig. 1), where the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges, and generally agree with those in compounds (II)-(*X*). In all structures atom N9 is substituted.

An examination of the deviations from the least-squares planes through individual rings shows that rings A (C1—C4/C4a/C9a), B (C4a/C5a/C8a/N9/C9a), C (C5a/C5—C8/C8a) and D (C7/C8/C14—C17) are planar. The carbazole skeleton, containing the rings A, B and C are also nearly planar [with a maximum deviation of 0.078 (2) Å for atom C2] with dihedral angles of A/B = 2.37 (6), A/C = 5.01 (5) and B/C = 2.81 (5) ° Ring D is oriented with respect to the planar

carbazole skeleton at a dihedral angle of 4.22 (4) °. So, it is nearly coplanar with the carbazole skeleton. Atoms O1 and C18 displaced by 0.010 (1) and -0.045 (2) Å from the plane of ring D, respectively, while atom C10 is 0.092 (2) Å away from the plane of the carbazole skeleton.

In the crystal structure, molecules are alongated along the *c* axis and stacked along the *b* axis (Fig. 2).

## S2. Experimental

For the preparation of the title compound, (I), a solution of 3-methoxy-11H -benzo[*a*]carbazole (1.00 g, 4.0 mmol) in dichloromethane (20 ml) was cooled to 273 K, and then sodium hydroxide (2 ml, 50%), tetrabutylammonium hydrogen sulfate (0.10 g) and butyl bromide (0.62 g, 4.5 mmol) were added. The mixture was stirred for 1 h at 273 K, and then 2 h at 298 K. It was washed with hydrochloric acid (50 ml, 10%) and the organic layer was dried with anhydrous magnesium sulfate. The solvent was evaporated under reduced pressure and the residue was crystallized from methanol (yield; 1.15 g, 93%, m.p. 369 K).

## S3. Refinement

H13A, H13B, H13C, H18A, H18B and H18C atoms (for methyl groups) were positioned geometrically with C—H = 0.98 Å and constrained to ride on their parent atoms, $U_{iso}(H) = 1.5U_{eq}(C)$. The remaining H atoms were located in difference Fourier maps and refined isotropically. Friedel pairs were merged because of the weak anomalous scatterer of the compound, and the absolute structure was not determined.
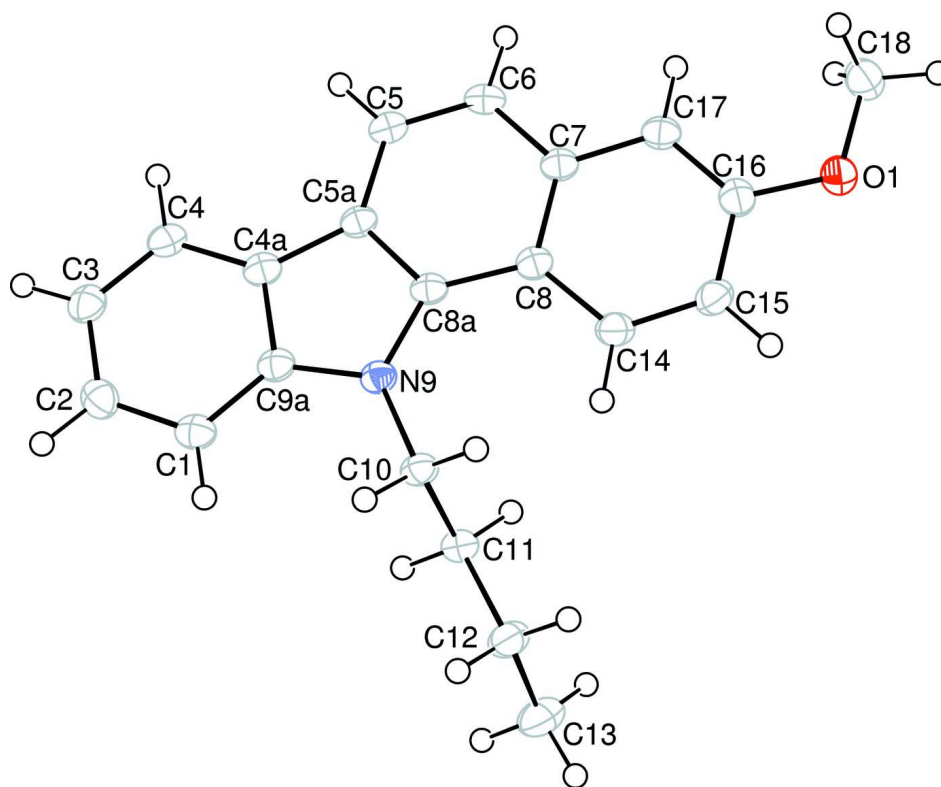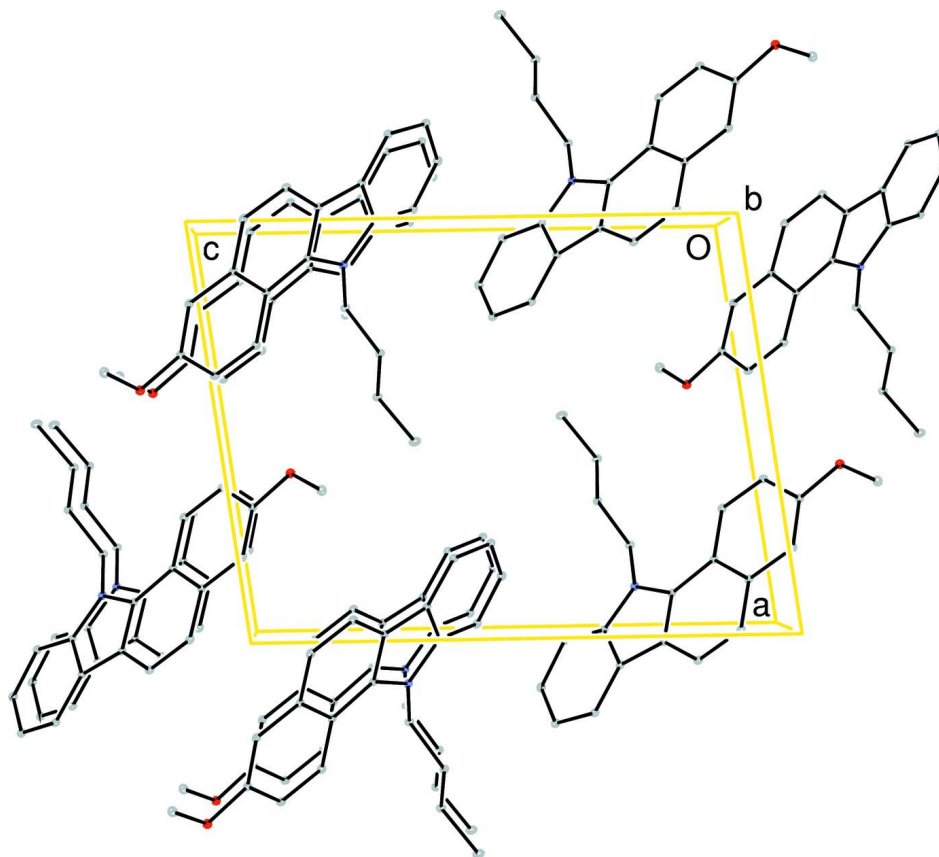


**Figure 1**
The molecular structure of the title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A partial packing diagram.

**11-Butyl-3-methoxy-11*H*-benzo[*a*]carbazole**

*Crystal data*

| | |
|---|---|
| $C_{21}H_{21}NO$ | $F(000) = 324$ |
| $M_r = 303.39$ | $D_x = 1.228$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: P 2yb | Cell parameters from 3840 reflections |
| $a = 10.7263$ (6) Å | $\theta = 2.3–28.4°$ |
| $b = 5.5562$ (3) Å | $\mu = 0.08$ mm$^{-1}$ |
| $c = 13.8967$ (7) Å | $T = 100$ K |
| $\beta = 97.841$ (2)° | Block, colorless |
| $V = 820.46$ (8) Å$^3$ | $0.48 \times 0.39 \times 0.35$ mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD area-detector diffractometer | 7947 measured reflections |
| | 2248 independent reflections |
| Radiation source: fine-focus sealed tube | 2001 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.027$ |
| $\varphi$ and $\omega$ scans | $\theta_{max} = 28.4°$, $\theta_{min} = 1.5°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2005) | $h = -13 \rightarrow 14$ |
| | $k = -7 \rightarrow 6$ |
| $T_{min} = 0.965$, $T_{max} = 0.974$ | $l = -17 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.035$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.091$ | |
| $S = 1.04$ | H atoms treated by a mixture of independent and constrained refinement |
| 2248 reflections | |
| 270 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.1088P]$ |
| Primary atom site location: structure-invariant direct methods | where $P = (F_o^2 + 2F_c^2)/3$ |
| | $(\Delta/\sigma)_{max} < 0.001$ |
| | $\Delta\rho_{max} = 0.25$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.16$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.59965 (11) | 0.9420 (3) | −0.11895 (9) | 0.0299 (3) |
| C1 | 1.02958 (17) | 0.2221 (4) | 0.41676 (13) | 0.0246 (4) |
| H1 | 0.9764 (19) | 0.094 (5) | 0.4258 (15) | 0.030 (6)* |
| C2 | 1.14448 (17) | 0.2475 (4) | 0.47538 (13) | 0.0276 (4) |
| H2 | 1.166 (2) | 0.136 (5) | 0.5266 (17) | 0.042 (7)* |
| C3 | 1.22609 (17) | 0.4381 (4) | 0.46185 (13) | 0.0271 (4) |
| H3 | 1.3035 (19) | 0.447 (4) | 0.5040 (14) | 0.022 (5)* |
| C4 | 1.19492 (16) | 0.6061 (4) | 0.38921 (13) | 0.0245 (4) |
| H4 | 1.2520 (17) | 0.742 (4) | 0.3796 (13) | 0.021 (5)* |
| C4A | 1.08080 (15) | 0.5806 (4) | 0.32759 (12) | 0.0217 (4) |
| C5 | 1.06152 (16) | 0.9086 (4) | 0.19246 (13) | 0.0232 (4) |
| H5 | 1.1411 (18) | 0.992 (4) | 0.2181 (13) | 0.020 (5)* |
| C5A | 1.02208 (15) | 0.7091 (3) | 0.24338 (12) | 0.0210 (4) |
| C6 | 0.98899 (16) | 0.9848 (4) | 0.10954 (13) | 0.0238 (4) |
| H6 | 1.0136 (18) | 1.120 (5) | 0.0755 (14) | 0.025 (5)* |
| C7 | 0.87121 (15) | 0.8724 (4) | 0.07581 (12) | 0.0215 (4) |
| C8 | 0.82600 (15) | 0.6754 (4) | 0.12711 (12) | 0.0205 (4) |
| C8A | 0.90768 (15) | 0.5921 (4) | 0.21092 (12) | 0.0197 (3) |
| C9A | 0.99973 (15) | 0.3887 (4) | 0.34259 (12) | 0.0214 (4) |
| N9 | 0.89451 (13) | 0.3987 (3) | 0.27259 (10) | 0.0209 (3) |
| C10 | 0.79576 (15) | 0.2165 (4) | 0.26464 (13) | 0.0221 (4) |
| H101 | 0.8331 (18) | 0.076 (5) | 0.2927 (14) | 0.021 (5)* |
| H102 | 0.7675 (17) | 0.185 (4) | 0.1949 (14) | 0.021 (5)* |
| C11 | 0.68345 (16) | 0.2861 (4) | 0.31519 (13) | 0.0228 (4) |

| | | | | |
|---|---|---|---|---|
| H111 | 0.7121 (18) | 0.304 (4) | 0.3868 (14) | 0.023 (5)* |
| H112 | 0.6483 (19) | 0.442 (5) | 0.2886 (15) | 0.027 (6)* |
| C12 | 0.58043 (17) | 0.0964 (4) | 0.29886 (15) | 0.0290 (4) |
| H121 | 0.6196 (19) | −0.064 (5) | 0.3171 (14) | 0.025 (5)* |
| H122 | 0.5517 (19) | 0.077 (5) | 0.2304 (17) | 0.036 (6)* |
| C13 | 0.47038 (18) | 0.1518 (5) | 0.35479 (16) | 0.0397 (5) |
| H13A | 0.4050 | 0.0290 | 0.3400 | 0.060* |
| H13B | 0.4355 | 0.3104 | 0.3355 | 0.060* |
| H13C | 0.5002 | 0.1516 | 0.4247 | 0.060* |
| C14 | 0.70388 (16) | 0.5850 (4) | 0.09343 (13) | 0.0248 (4) |
| H14 | 0.668 (2) | 0.458 (5) | 0.1282 (15) | 0.032 (6)* |
| C15 | 0.63327 (16) | 0.6779 (4) | 0.01272 (13) | 0.0264 (4) |
| H15 | 0.549 (2) | 0.613 (5) | −0.0092 (17) | 0.044 (7)* |
| C16 | 0.68014 (16) | 0.8672 (4) | −0.03944 (12) | 0.0242 (4) |
| C17 | 0.79658 (16) | 0.9641 (4) | −0.00876 (12) | 0.0235 (4) |
| H17 | 0.8282 (16) | 1.097 (4) | −0.0415 (13) | 0.015 (5)* |
| C18 | 0.63988 (18) | 1.1414 (4) | −0.17108 (14) | 0.0325 (5) |
| H18A | 0.5741 | 1.1833 | −0.2245 | 0.049* |
| H18B | 0.7172 | 1.0988 | −0.1974 | 0.049* |
| H18C | 0.6561 | 1.2794 | −0.1272 | 0.049* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0241 (6) | 0.0399 (9) | 0.0257 (6) | 0.0017 (6) | 0.0027 (5) | 0.0051 (7) |
| C1 | 0.0267 (9) | 0.0213 (10) | 0.0275 (8) | 0.0008 (8) | 0.0096 (7) | −0.0004 (8) |
| C2 | 0.0292 (9) | 0.0284 (11) | 0.0259 (9) | 0.0058 (8) | 0.0057 (7) | 0.0023 (8) |
| C3 | 0.0228 (8) | 0.0342 (12) | 0.0250 (8) | 0.0021 (8) | 0.0062 (7) | −0.0029 (9) |
| C4 | 0.0212 (8) | 0.0280 (11) | 0.0253 (8) | −0.0024 (8) | 0.0071 (6) | −0.0025 (8) |
| C4A | 0.0222 (8) | 0.0219 (9) | 0.0223 (8) | 0.0003 (7) | 0.0077 (6) | −0.0039 (8) |
| C5 | 0.0215 (8) | 0.0229 (9) | 0.0263 (8) | −0.0032 (7) | 0.0075 (7) | −0.0025 (8) |
| C5A | 0.0197 (7) | 0.0218 (9) | 0.0229 (8) | 0.0002 (7) | 0.0072 (6) | −0.0024 (7) |
| C6 | 0.0262 (8) | 0.0209 (9) | 0.0261 (9) | −0.0032 (8) | 0.0100 (7) | 0.0006 (8) |
| C7 | 0.0215 (8) | 0.0226 (10) | 0.0220 (8) | 0.0009 (7) | 0.0088 (7) | −0.0025 (7) |
| C8 | 0.0207 (7) | 0.0203 (9) | 0.0215 (8) | 0.0008 (7) | 0.0065 (6) | −0.0024 (7) |
| C8A | 0.0203 (7) | 0.0178 (9) | 0.0225 (8) | 0.0001 (7) | 0.0081 (6) | −0.0021 (7) |
| C9A | 0.0199 (7) | 0.0217 (9) | 0.0239 (8) | 0.0016 (7) | 0.0076 (6) | −0.0015 (7) |
| N9 | 0.0204 (6) | 0.0186 (8) | 0.0240 (7) | −0.0017 (6) | 0.0045 (5) | 0.0000 (6) |
| C10 | 0.0210 (8) | 0.0189 (9) | 0.0269 (9) | −0.0034 (7) | 0.0051 (7) | −0.0001 (8) |
| C11 | 0.0212 (8) | 0.0226 (10) | 0.0252 (9) | −0.0026 (7) | 0.0054 (6) | 0.0003 (7) |
| C12 | 0.0230 (8) | 0.0338 (12) | 0.0307 (10) | −0.0073 (8) | 0.0051 (7) | −0.0015 (9) |
| C13 | 0.0246 (9) | 0.0471 (15) | 0.0491 (12) | −0.0051 (10) | 0.0112 (8) | 0.0041 (11) |
| C14 | 0.0247 (8) | 0.0265 (10) | 0.0240 (8) | −0.0033 (8) | 0.0062 (6) | 0.0006 (8) |
| C15 | 0.0201 (8) | 0.0323 (11) | 0.0272 (9) | −0.0018 (8) | 0.0049 (7) | −0.0012 (8) |
| C16 | 0.0235 (8) | 0.0286 (10) | 0.0211 (8) | 0.0045 (7) | 0.0051 (7) | −0.0009 (8) |
| C17 | 0.0246 (8) | 0.0240 (10) | 0.0234 (8) | 0.0005 (7) | 0.0087 (6) | −0.0008 (8) |
| C18 | 0.0324 (9) | 0.0366 (12) | 0.0283 (9) | 0.0040 (9) | 0.0035 (8) | 0.0064 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C16 | 1.371 (2) | C10—C11 | 1.525 (2) |
| O1—C18 | 1.422 (3) | C10—H101 | 0.94 (2) |
| C1—C2 | 1.389 (3) | C10—H102 | 0.991 (19) |
| C1—H1 | 0.93 (2) | C11—C12 | 1.521 (3) |
| C2—C3 | 1.403 (3) | C11—H111 | 1.006 (19) |
| C2—H2 | 0.95 (3) | C11—H112 | 0.99 (3) |
| C3—C4 | 1.382 (3) | C12—C13 | 1.530 (3) |
| C3—H3 | 0.95 (2) | C12—H121 | 1.00 (2) |
| C4—H4 | 0.99 (2) | C12—H122 | 0.97 (2) |
| C4A—C4 | 1.402 (2) | C13—H13A | 0.9800 |
| C4A—C9A | 1.410 (3) | C13—H13B | 0.9800 |
| C5—C5A | 1.411 (3) | C13—H13C | 0.9800 |
| C5—H5 | 1.00 (2) | C14—C8 | 1.421 (2) |
| C5A—C4A | 1.440 (2) | C14—H14 | 0.97 (2) |
| C6—C5 | 1.367 (3) | C15—C14 | 1.366 (3) |
| C6—H6 | 0.95 (2) | C15—H15 | 0.98 (2) |
| C7—C6 | 1.430 (2) | C16—C15 | 1.408 (3) |
| C8—C7 | 1.427 (2) | C16—C17 | 1.373 (3) |
| C8—C8A | 1.435 (2) | C17—C7 | 1.423 (3) |
| C8A—C5A | 1.408 (2) | C17—H17 | 0.96 (2) |
| C9A—C1 | 1.390 (3) | C18—H18A | 0.9800 |
| N9—C8A | 1.393 (2) | C18—H18B | 0.9800 |
| N9—C9A | 1.387 (2) | C18—H18C | 0.9800 |
| N9—C10 | 1.458 (2) | | |
| | | | |
| C16—O1—C18 | 116.45 (15) | N9—C10—H101 | 106.4 (12) |
| C2—C1—C9A | 117.62 (18) | N9—C10—H102 | 108.7 (12) |
| C2—C1—H1 | 120.6 (13) | C11—C10—H101 | 109.8 (12) |
| C9A—C1—H1 | 121.7 (13) | C11—C10—H102 | 109.9 (11) |
| C1—C2—C3 | 121.20 (18) | H101—C10—H102 | 108.3 (19) |
| C1—C2—H2 | 119.0 (15) | C10—C11—H111 | 109.1 (11) |
| C3—C2—H2 | 119.8 (15) | C10—C11—H112 | 109.4 (12) |
| C2—C3—H3 | 117.8 (14) | C12—C11—C10 | 111.04 (16) |
| C4—C3—C2 | 120.97 (17) | C12—C11—H111 | 109.5 (13) |
| C4—C3—H3 | 121.2 (14) | C12—C11—H112 | 108.4 (13) |
| C3—C4—C4A | 118.82 (18) | H112—C11—H111 | 109.4 (18) |
| C3—C4—H4 | 121.4 (11) | C11—C12—C13 | 112.32 (18) |
| C4A—C4—H4 | 119.8 (11) | C11—C12—H121 | 107.9 (12) |
| C4—C4A—C5A | 134.01 (18) | C11—C12—H122 | 110.6 (15) |
| C4—C4A—C9A | 119.40 (17) | C13—C12—H121 | 112.1 (12) |
| C9A—C4A—C5A | 106.56 (14) | C13—C12—H122 | 111.0 (13) |
| C5A—C5—H5 | 119.3 (12) | H121—C12—H122 | 103 (2) |
| C6—C5—C5A | 119.41 (17) | C12—C13—H13A | 109.5 |
| C6—C5—H5 | 121.3 (12) | C12—C13—H13B | 109.5 |
| C5—C5A—C4A | 132.01 (16) | C12—C13—H13C | 109.5 |
| C8A—C5A—C4A | 107.26 (16) | H13A—C13—H13B | 109.5 |

| | | | |
|---|---|---|---|
| C8A—C5A—C5 | 120.68 (16) | H13A—C13—H13C | 109.5 |
| C5—C6—C7 | 121.13 (17) | H13B—C13—H13C | 109.5 |
| C5—C6—H6 | 120.2 (12) | C8—C14—H14 | 120.6 (13) |
| C7—C6—H6 | 118.5 (12) | C15—C14—C8 | 121.32 (18) |
| C8—C7—C6 | 121.11 (16) | C15—C14—H14 | 118.1 (13) |
| C17—C7—C6 | 119.12 (17) | C14—C15—C16 | 120.52 (17) |
| C17—C7—C8 | 119.75 (16) | C14—C15—H15 | 120.0 (16) |
| C7—C8—C8A | 116.20 (15) | C16—C15—H15 | 119.5 (15) |
| C14—C8—C7 | 117.85 (17) | O1—C16—C15 | 114.31 (16) |
| C14—C8—C8A | 125.91 (17) | O1—C16—C17 | 125.26 (18) |
| C5A—C8A—C8 | 121.33 (16) | C17—C16—C15 | 120.42 (17) |
| N9—C8A—C5A | 108.44 (15) | C7—C17—H17 | 118.4 (11) |
| N9—C8A—C8 | 130.23 (16) | C16—C17—C7 | 120.08 (18) |
| C1—C9A—C4A | 121.96 (16) | C16—C17—H17 | 121.5 (11) |
| N9—C9A—C1 | 129.06 (17) | O1—C18—H18A | 109.5 |
| N9—C9A—C4A | 108.98 (15) | O1—C18—H18B | 109.5 |
| C8A—N9—C10 | 128.47 (14) | O1—C18—H18C | 109.5 |
| C9A—N9—C8A | 108.74 (14) | H18A—C18—H18B | 109.5 |
| C9A—N9—C10 | 122.57 (15) | H18A—C18—H18C | 109.5 |
| N9—C10—C11 | 113.60 (15) | H18B—C18—H18C | 109.5 |
| | | | |
| C18—O1—C16—C15 | 176.75 (16) | N9—C8A—C5A—C4A | 0.40 (19) |
| C18—O1—C16—C17 | −2.2 (3) | N9—C8A—C5A—C5 | 178.18 (16) |
| C9A—C1—C2—C3 | 1.6 (3) | C8—C8A—C5A—C4A | −179.64 (15) |
| C1—C2—C3—C4 | −0.4 (3) | C8—C8A—C5A—C5 | −1.9 (3) |
| C2—C3—C4—C4A | −1.2 (3) | N9—C9A—C1—C2 | 177.47 (17) |
| C5A—C4A—C4—C3 | −176.09 (19) | C4A—C9A—C1—C2 | −1.3 (3) |
| C9A—C4A—C4—C3 | 1.5 (2) | C9A—N9—C8A—C5A | −1.06 (18) |
| C4—C4A—C9A—N9 | −179.24 (16) | C9A—N9—C8A—C8 | 178.97 (17) |
| C4—C4A—C9A—C1 | −0.2 (2) | C10—N9—C8A—C5A | −175.70 (16) |
| C5A—C4A—C9A—N9 | −1.06 (18) | C10—N9—C8A—C8 | 4.3 (3) |
| C5A—C4A—C9A—C1 | 177.96 (16) | C8A—N9—C9A—C1 | −177.60 (17) |
| C6—C5—C5A—C4A | 175.63 (18) | C8A—N9—C9A—C4A | 1.33 (18) |
| C6—C5—C5A—C8A | −1.5 (3) | C10—N9—C9A—C1 | −2.6 (3) |
| C5—C5A—C4A—C4 | 0.8 (3) | C10—N9—C9A—C4A | 176.34 (15) |
| C5—C5A—C4A—C9A | −177.03 (18) | C9A—N9—C10—C11 | 96.69 (19) |
| C8A—C5A—C4A—C4 | 178.20 (19) | C8A—N9—C10—C11 | −89.3 (2) |
| C8A—C5A—C4A—C9A | 0.40 (19) | N9—C10—C11—C12 | 176.39 (15) |
| C7—C6—C5—C5A | 2.7 (3) | C10—C11—C12—C13 | 175.83 (17) |
| C8—C7—C6—C5 | −0.6 (3) | C15—C14—C8—C7 | 2.1 (3) |
| C17—C7—C6—C5 | 177.50 (17) | C15—C14—C8—C8A | 179.73 (17) |
| C8A—C8—C7—C6 | −2.6 (2) | C16—C15—C14—C8 | −0.1 (3) |
| C8A—C8—C7—C17 | 179.29 (16) | O1—C16—C15—C14 | 179.77 (17) |
| C14—C8—C7—C6 | 175.24 (16) | C17—C16—C15—C14 | −1.3 (3) |
| C14—C8—C7—C17 | −2.8 (2) | O1—C16—C17—C7 | 179.31 (16) |
| C7—C8—C8A—N9 | −176.22 (16) | C15—C16—C17—C7 | 0.5 (3) |
| C7—C8—C8A—C5A | 3.8 (2) | C16—C17—C7—C6 | −176.50 (16) |
| C14—C8—C8A—N9 | 6.1 (3) | C16—C17—C7—C8 | 1.6 (3) |

C14—C8—C8A—C5A     −173.86 (17)