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# Comparison of the crystal structures of 4,4′-bis[3-(4-methylpiperidin-1-yl)prop-1-yn-1-yl]-1,1′-biphenyl and 4,4′-bis[3-(2,2,6,6-tetramethylpiperidin-1-yl)prop-1-yn-1-yl]-1,1′-biphenyl

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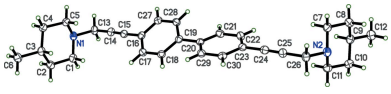
As part of a comprehensive program to discover $\alpha9\alpha10$ nicotinic acetylcholine receptor antagonists, the title compounds $C_{30}H_{36}N_2$, (I), and $C_{36}H_{48}N_2$, (II), were synthesized by coupling 4,4′-bis(3-bromoprop-1-yn-1-yl)-1,1′-biphenyl with 4-methylpiperidine and 2,2,6,6-tetramethylpiperidine, respectively, in acetonitrile at room temperature. In compound (I), the biphenyl system has a twisted conformation with a dihedral angle of 26.57 (6)° between the two phenyl rings of the biphenyl moiety, while in compound (II), the biphenyl moiety sits on a crystallographic inversion centre so the two phenyl rings are exactly coplanar. The terminal piperidine rings in both compound (I) and compound (II) are in the chair conformation. In compound (I), the dihedral angles about the ethynyl groups between the planes of the phenyl rings and the piperidine ring N atoms are 37.16 (16) and 14.20 (17)°. In compound (II), the corresponding dihedral angles are both 61.48 (17)°. There are no noteworthy intermolecular interactions in (I), but in (II) there is a small $\pi$-overlap between inversion-related molecules $(1-x, 1-y, 1-z)$, with an interplanar spacing of 3.553 (3) Å and centroid-to-centroid separation of 3.859 (4) Å.

## 1. Chemical context

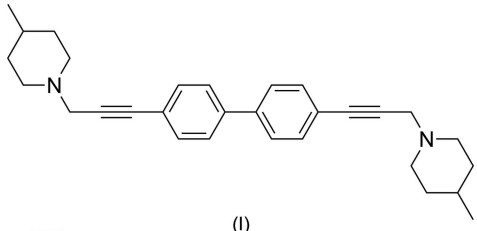
Previous studies have shown that the bis-quaternary ammonium compound 1′-[(1,1′-biphenyl)-4,4′-diylbis(prop-2-yne-3,1-diyl)]bis(3,4-dimethylpyridin-1-ium) bromide (ZZ161C) is a potent and selective $\alpha9\alpha10$ nicotinic acetylcholine receptor antagonist (Zheng *et al.*, 2011). ZZ161C has been reported to have analgesic effects in various animal pain models (Wala *et al.*, 2012). In order to improve the pharmacological and pharmacokinetic profile of ZZ161C, we have replaced the terminal azaaromatic rings with fully reduced piperidine rings to obtain the title compounds (I) and (II). Single-crystal X-ray structure determinations were carried out to determine the conformations of these compounds.



## 2. Structural commentary

The title compounds, $C_{30}H_{36}N_2$ (I) and $C_{36}H_{48}N_2$ (II) are shown in Figs. 1 and 2, respectively. The present X-ray crystallographic study was carried out in order to ascertain the geometry of the piperidine rings and the biphenyl ring systems, as well as to obtain more detailed information about the conformation of the title compounds. Crystals of both (I) and (II) are monoclinic, space group $P2_1/c$, with $Z' = 1$ and 0.5,

respectively. In each compound, individual bond lengths and angles are unremarkable.




The piperidine rings in both of the title molecules are in the chair conformation. In (I), the biphenyl rings (C20-C21-C22-C23-C30-C29) and (C16-C17-C18-C19-C28-C27) are non-coplanar, with a dihedral angle of 26.57 (6)°. For compound (II), however, the biphenyl group is strictly coplanar because the molecule sits on a crystallographic inversion centre. In compound (I), the dihedral angles about the ethynyl groups between the planes of the phenyl rings and the piperidine ring N atoms are 37.16 (16) and 14.20 (17)°. In compound (II), the corresponding dihedral angles are both 61.48 (17)°.

## 3. Supramolecular features

Other than weak van der Waals interactions, there are no noteworthy intermolecular contacts in (I). In (II) there is a small $\pi$-overlap between inversion-related molecules ($1-x$, $1-y$, $1-z$), giving an interplanar spacing of 3.553 (3) Å and centroid-to-centroid separation of 3.859 (4) Å.

## 4. Database survey

A search of the November 2014 release of the Cambridge Structure Database (Groom & Allen, 2014), with updates through May 2015, using the program *Mogul* (Bruno *et al.*, 2004) for 4,4′ substituted biphenyl fragments was conducted. The search was restricted to non-organometallic, solvent-free structures with $R < 5\%$ and Cl as the heaviest element. There were over 1000 hits, which gave a bimodal distribution of biphenyl dihedral angles with a tight peak at 0° and a broader peak centred at 30°. The biphenyl dihedral angles in (I) and (II) are thus not unusual.

## 5. Synthesis and crystallization

In the synthesis of compound (I), 3,3′-[(1,1′-biphenyl)-4,4′-diyl]-bis(prop-2-yn-1-ol) was synthesized by coupling 1,2,4,5-tetraiodobenzene with 4-pentyn-1-ol. Bis-(triphenylphosphine)palladium(II) dichloride and copper(I) iodide were used as catalysts. The mixture was stirred at room temperature for 24 h under argon. The obtained 3,3′-[(1,1′-biphenyl)-4,4′-diyl]-bis(prop-2-yn-1-ol) was converted to 4,4′-bis-(3-bromoprop-1-yn-1-yl)-1,1′-biphenyl using bromomethane and triphenylphosphine in anhydrous methylene chloride at room temperature. To a suspension of the 4,4′-bis(3-bromoprop-1-yn-1-yl)-1,1′-biphenyl (100.0 mg, 0.26 mmol) in acetonitrile (7 mL) was added 4-methylpiperidine (77.2 mg, 0.78 mmol) and the reaction mixture stirred for two hours at room

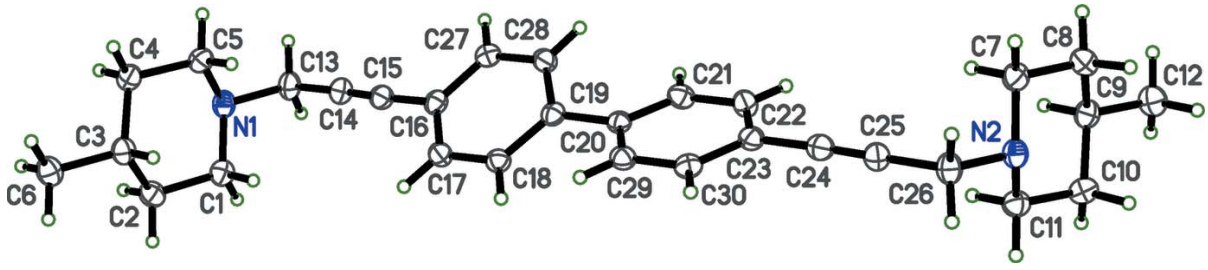



**Figure 1**
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

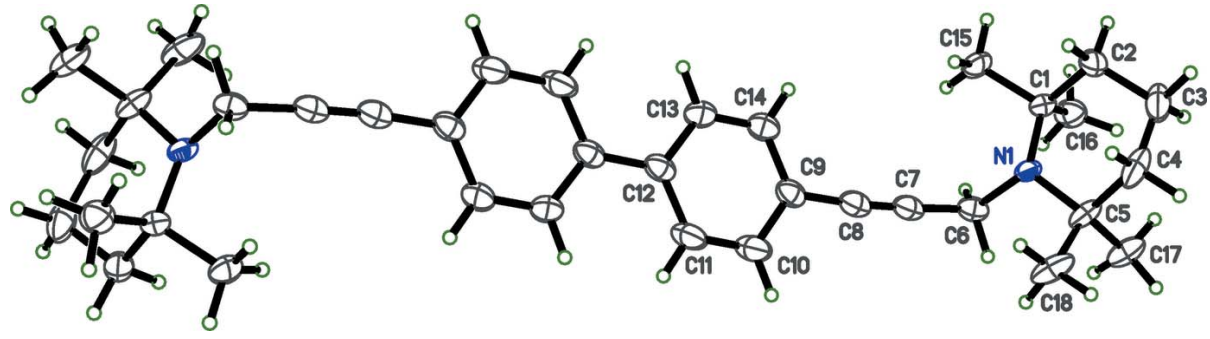



**Figure 2**
The molecular structure of (II), with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are generated by the symmetry operator ($1-x$, $2-y$, $1-z$).

**Table 1**
Experimental details.

| | (I) | (II) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{30}H_{36}N_2$ | $C_{36}H_{48}N_2$ |
| $M_r$ | 424.61 | 508.76 |
| Crystal system, space group | Monoclinic, $P2_1/c$ | Monoclinic, $P2_1/c$ |
| Temperature (K) | 90 | 90 |
| $a, b, c$ (Å) | 21.9870 (6), 7.0390 (3), 15.7840 (11) | 16.0591 (3), 6.2267 (1), 15.5921 (3) |
| $\beta$ (°) | 99.0310 (19) | 100.895 (1) |
| $V$ (Å$^3$) | 2412.6 (2) | 1531.03 (5) |
| $Z$ | 4 | 2 |
| Radiation type | Mo $K\alpha$ | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.07 | 0.47 |
| Crystal size (mm) | 0.32 × 0.30 × 0.03 | 0.22 × 0.04 × 0.03 |
| Data collection | | |
| Diffractometer | Nonius KappaCCD | Bruker X8 Proteum |
| Absorption correction | Multi-scan (*SADABS*; Sheldrick, 2008*a*) | Multi-scan (*SADABS*; Bruker, 2006) |
| $T_{min}$, $T_{max}$ | 0.764, 0.958 | 0.767, 0.929 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 54455, 5546, 3347 | 19631, 2797, 2405 |
| $R_{int}$ | 0.066 | 0.053 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.650 | 0.602 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.049, 0.144, 1.02 | 0.050, 0.137, 1.05 |
| No. of reflections | 5546 | 2797 |
| No. of parameters | 291 | 176 |
| H-atom treatment | H-atom parameters constrained | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.22, −0.22 | 0.24, −0.24 |

Computer programs: *COLLECT* (Nonius, 1998), *APEX2* and *SAINT* (Bruker, 2006), *SCALEPACK* and *DENZO-SMN* (Otwinowski & Minor, 2006), *SHELXS97*, *SHELXTL* and *XP* in *SHELXTL* (Sheldrick, 2008*b*), *SHELXL2014* (Sheldrick, 2015) and *CIFFIX* (Parkin, 2013).

temperature to obtain compound (I). Acetonitrile was removed from the reaction mixture under reduced pressure and the resulting residue was partitioned between water and dichloromethane. The organic layers were collected and combined. The extract (organic layer) was dried over anhydrous sodium sulfate, filtered, and the filtrate concentrated under reduced pressure. The resulting crude sample of compound (I) was purified by column chromatography (dichloromethane/methanol, 100:2 *v*/*v*). Yield: 80%.

A crude sample of compound (II) was prepared using the same experimental conditions for the preparation of compound (I) but utilizing 2,2,6,6-tetramethylpiperidine (110.0 mg, 0.78 mmol) instead of 4-methylpiperidine. Column chromatography (dichlormethane/methanol 100:2 *v*/*v*) was then used for purification of (II). Yield: 80%.

Compound (I) and (II) were each dissolved separately in a mixture of dichloromethane/methanol (2:1 *v*/*v*). Yellow crystals of both compounds were obtained by slow evaporation of the solution at room temperature over 24 h.

Compound (I) $^1$H-NMR (400 Mz, $CDCl_3$): $\delta$ 7.49 (*q*, 8H), 3.52 (*s*, 4H), 2.97 (*d*, 4H), 2.26 (*t*, 4H) p.p.m.; $^{13}$C-NMR (100 Mz, $CDCl_3$): $\delta$ 132.92, 132.19, 126.76, 122.36, 85.13, 52.83, 48.09, 34.02, 30.20, 21.74 p.p.m.

Compound (II) $^1$H-NMR (400 Mz, $CDCl_3$): $\delta$ 7.50 (*q*, 8H), 3.62 (*s*, 4H), 1.61–1.60 (*m*, 8H), 1.52–1.51 (*m*, 4H), 1.22 (*s*, 24H) p.p.m. $^{13}$C-NMR (100 Mz, $CDCl_3$): $\delta$ 139.47, 131.88, 126.61, 123.42, 94.00, 80.78, 55.00, 41.16, 33.87, 27.49, 17.81 p.p.m.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were found in difference Fourier maps, but subsequently included in the refinement using riding models, with constrained distances set to 0.95 Å ($CSp^2H$), 0.98 Å ($RCH_3$), 0.99 Å ($R_2CH_2$) and 1.00 Å ($R_3CH$). $U_{iso}$(H) parameters were set to values of either $1.2U_{eq}$(C) or $1.5U_{eq}$(C) ($RCH_3$ only) of the attached atom. The final models were checked using an *R*-tensor (Parkin, 2000) and by *PLATON* (Spek, 2009).

## Acknowledgements

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# supporting information

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## Computing details

Data collection: *COLLECT* (Nonius, 1998) for (I); *APEX2* (Bruker, 2006) for (II). Cell refinement: *SCALEPACK* (Otwinowski & Minor, 2006) for (I); *SAINT* (Bruker, 2006) for (II). Data reduction: *DENZO-SMN* (Otwinowski & Minor, 2006) for (I); *SAINT* (Bruker, 2006) for (II). For both compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008b); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *XP in SHELXTL* (Sheldrick, 2008b); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008b) and *CIFFIX* (Parkin, 2013).

### (I) 4,4′-Bis[3-(4-methylpiperidin-1-yl)prop-1-yn-1-yl]-1,1′-biphenyl

*Crystal data*

$C_{30}H_{36}N_2$
$M_r$ = 424.61
Monoclinic, $P2_1/c$
$a$ = 21.9870 (6) Å
$b$ = 7.0390 (3) Å
$c$ = 15.7840 (11) Å
$\beta$ = 99.0310 (19)°
$V$ = 2412.6 (2) Å$^3$
$Z$ = 4
$F(000)$ = 920
$D_x$ = 1.169 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5987 reflections
$\theta$ = 1.0–27.5°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 90 K
Plate, colourless
0.32 × 0.30 × 0.03 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed-tube
Detector resolution: 9.1 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans at fixed $\chi$ = 55°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008a)
$T_{min}$ = 0.764, $T_{max}$ = 0.958
54455 measured reflections
5546 independent reflections
3347 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.066
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.9°
$h$ = −28→28
$k$ = −9→9
$l$ = −20→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.144
$S$ = 1.02
5546 reflections
291 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

| | |
|---|---|
| Secondary atom site location: difference Fourier map | $w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.3882P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| Hydrogen site location: difference Fourier map | $(\Delta/\sigma)_{max} < 0.001$ |
| H-atom parameters constrained | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |

*Special details*

**Experimental**. The crystal was mounted with polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid nitrogen based cryostat, according to published methods (Hope, 1994; Parkin & Hope, 1998).
Diffraction data were collected with the crystal at 90 K, which is standard practice in this laboratory for the majority of flash-cooled crystals.

**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 progress was checked using *PLATON* (Spek, 2009) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.20730 (5) | 0.24205 (18) | 0.41351 (8) | 0.0245 (3) |
| N2 | 0.79153 (5) | 0.22850 (18) | −0.16448 (8) | 0.0234 (3) |
| C1 | 0.19401 (7) | 0.4165 (2) | 0.36333 (10) | 0.0255 (4) |
| H1A | 0.2171 | 0.4153 | 0.3143 | 0.031* |
| H1B | 0.2080 | 0.5277 | 0.3996 | 0.031* |
| C2 | 0.12538 (7) | 0.4349 (2) | 0.33012 (10) | 0.0268 (4) |
| H2A | 0.1179 | 0.5510 | 0.2946 | 0.032* |
| H2B | 0.1027 | 0.4478 | 0.3793 | 0.032* |
| C3 | 0.10128 (7) | 0.2625 (2) | 0.27674 (10) | 0.0246 (4) |
| H3A | 0.1231 | 0.2569 | 0.2258 | 0.030* |
| C4 | 0.11755 (6) | 0.0832 (2) | 0.32978 (10) | 0.0259 (4) |
| H4A | 0.0947 | 0.0822 | 0.3791 | 0.031* |
| H4B | 0.1049 | −0.0300 | 0.2941 | 0.031* |
| C5 | 0.18653 (6) | 0.0736 (2) | 0.36255 (10) | 0.0250 (4) |
| H5A | 0.1957 | −0.0416 | 0.3982 | 0.030* |
| H5B | 0.2092 | 0.0644 | 0.3132 | 0.030* |
| C6 | 0.03225 (7) | 0.2769 (2) | 0.24399 (10) | 0.0304 (4) |
| H6A | 0.0188 | 0.1650 | 0.2091 | 0.046* |
| H6B | 0.0239 | 0.3918 | 0.2090 | 0.046* |
| H6C | 0.0098 | 0.2833 | 0.2929 | 0.046* |
| C7 | 0.80978 (7) | 0.0602 (2) | −0.11219 (10) | 0.0251 (4) |
| H7A | 0.7993 | −0.0555 | −0.1470 | 0.030* |
| H7B | 0.7867 | 0.0566 | −0.0631 | 0.030* |
| C8 | 0.87867 (7) | 0.0625 (2) | −0.07886 (10) | 0.0263 (4) |
| H8A | 0.9017 | 0.0561 | −0.1279 | 0.032* |
| H8B | 0.8897 | −0.0505 | −0.0424 | 0.032* |
| C9 | 0.89727 (7) | 0.2419 (2) | −0.02685 (10) | 0.0248 (4) |
| H9A | 0.8756 | 0.2410 | 0.0243 | 0.030* |

| | | | | |
|---|---|---|---|---|
| C10 | 0.87516 (7) | 0.4146 (2) | −0.08094 (10) | 0.0264 (4) |
| H10A | 0.8981 | 0.4233 | −0.1300 | 0.032* |
| H10B | 0.8838 | 0.5309 | −0.0459 | 0.032* |
| C11 | 0.80634 (7) | 0.4027 (2) | −0.11454 (10) | 0.0253 (4) |
| H11A | 0.7831 | 0.4048 | −0.0657 | 0.030* |
| H11B | 0.7936 | 0.5146 | −0.1511 | 0.030* |
| C12 | 0.96641 (7) | 0.2486 (2) | 0.00546 (10) | 0.0308 (4) |
| H12A | 0.9786 | 0.1362 | 0.0407 | 0.046* |
| H12B | 0.9763 | 0.3635 | 0.0400 | 0.046* |
| H12C | 0.9888 | 0.2505 | −0.0436 | 0.046* |
| C13 | 0.27358 (6) | 0.2281 (2) | 0.44724 (10) | 0.0266 (4) |
| H13A | 0.2807 | 0.1132 | 0.4836 | 0.032* |
| H13B | 0.2858 | 0.3397 | 0.4842 | 0.032* |
| C14 | 0.31350 (7) | 0.2189 (2) | 0.38025 (10) | 0.0254 (4) |
| C15 | 0.34323 (7) | 0.2140 (2) | 0.32287 (10) | 0.0237 (3) |
| C16 | 0.38357 (6) | 0.2046 (2) | 0.25885 (9) | 0.0215 (3) |
| C17 | 0.37212 (6) | 0.3064 (2) | 0.18216 (9) | 0.0229 (3) |
| H17A | 0.3354 | 0.3788 | 0.1691 | 0.027* |
| C18 | 0.41397 (6) | 0.3028 (2) | 0.12486 (9) | 0.0214 (3) |
| H18A | 0.4054 | 0.3731 | 0.0730 | 0.026* |
| C19 | 0.46856 (6) | 0.1975 (2) | 0.14209 (9) | 0.0198 (3) |
| C20 | 0.51543 (6) | 0.2019 (2) | 0.08399 (9) | 0.0199 (3) |
| C21 | 0.57773 (6) | 0.1686 (2) | 0.11555 (9) | 0.0236 (4) |
| H21A | 0.5898 | 0.1423 | 0.1748 | 0.028* |
| C22 | 0.62205 (7) | 0.1732 (2) | 0.06218 (9) | 0.0250 (4) |
| H22A | 0.6640 | 0.1505 | 0.0852 | 0.030* |
| C23 | 0.60560 (7) | 0.2107 (2) | −0.02488 (10) | 0.0225 (3) |
| C24 | 0.64979 (7) | 0.2140 (2) | −0.08346 (10) | 0.0247 (4) |
| C25 | 0.68349 (7) | 0.2177 (2) | −0.13613 (10) | 0.0244 (4) |
| C26 | 0.72570 (6) | 0.2217 (2) | −0.20057 (9) | 0.0256 (4) |
| H26A | 0.7181 | 0.1073 | −0.2372 | 0.031* |
| H26B | 0.7157 | 0.3341 | −0.2379 | 0.031* |
| C27 | 0.43683 (6) | 0.0935 (2) | 0.27488 (9) | 0.0226 (3) |
| H27A | 0.4446 | 0.0193 | 0.3257 | 0.027* |
| C28 | 0.47825 (6) | 0.0904 (2) | 0.21758 (9) | 0.0223 (3) |
| H28A | 0.5141 | 0.0138 | 0.2297 | 0.027* |
| C29 | 0.49939 (7) | 0.2415 (2) | −0.00348 (10) | 0.0214 (3) |
| H29A | 0.4576 | 0.2659 | −0.0266 | 0.026* |
| C30 | 0.54362 (7) | 0.2455 (2) | −0.05680 (9) | 0.0221 (3) |
| H30A | 0.5317 | 0.2723 | −0.1161 | 0.027* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0214 (6) | 0.0309 (8) | 0.0216 (7) | −0.0012 (5) | 0.0049 (5) | −0.0001 (6) |
| N2 | 0.0216 (6) | 0.0271 (8) | 0.0218 (7) | −0.0004 (5) | 0.0045 (5) | −0.0014 (5) |
| C1 | 0.0259 (8) | 0.0268 (9) | 0.0243 (8) | −0.0011 (7) | 0.0061 (7) | −0.0036 (7) |
| C2 | 0.0278 (8) | 0.0273 (9) | 0.0255 (8) | 0.0033 (7) | 0.0053 (7) | −0.0013 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.0228 (8) | 0.0299 (9) | 0.0216 (8) | 0.0002 (6) | 0.0051 (6) | −0.0005 (7) |
| C4 | 0.0244 (8) | 0.0274 (9) | 0.0257 (8) | −0.0029 (7) | 0.0037 (7) | 0.0010 (7) |
| C5 | 0.0256 (8) | 0.0253 (9) | 0.0247 (8) | −0.0018 (6) | 0.0053 (7) | 0.0016 (7) |
| C6 | 0.0267 (8) | 0.0352 (10) | 0.0284 (9) | 0.0020 (7) | 0.0014 (7) | 0.0009 (7) |
| C7 | 0.0271 (8) | 0.0243 (9) | 0.0250 (8) | −0.0010 (7) | 0.0071 (7) | −0.0026 (7) |
| C8 | 0.0263 (8) | 0.0263 (9) | 0.0263 (9) | 0.0036 (7) | 0.0044 (7) | −0.0008 (7) |
| C9 | 0.0262 (8) | 0.0287 (9) | 0.0202 (8) | −0.0008 (7) | 0.0059 (6) | −0.0006 (7) |
| C10 | 0.0294 (8) | 0.0242 (9) | 0.0252 (8) | −0.0028 (7) | 0.0028 (7) | 0.0013 (7) |
| C11 | 0.0284 (8) | 0.0234 (9) | 0.0244 (8) | 0.0008 (7) | 0.0048 (7) | 0.0014 (7) |
| C12 | 0.0283 (8) | 0.0347 (10) | 0.0285 (9) | 0.0005 (7) | 0.0024 (7) | −0.0014 (7) |
| C13 | 0.0210 (8) | 0.0367 (10) | 0.0226 (8) | −0.0013 (7) | 0.0048 (6) | 0.0010 (7) |
| C14 | 0.0227 (8) | 0.0276 (9) | 0.0257 (8) | −0.0022 (7) | 0.0036 (7) | −0.0007 (7) |
| C15 | 0.0219 (8) | 0.0221 (8) | 0.0263 (8) | −0.0016 (6) | 0.0014 (7) | −0.0010 (7) |
| C16 | 0.0219 (7) | 0.0205 (8) | 0.0222 (8) | −0.0036 (6) | 0.0040 (6) | −0.0024 (6) |
| C17 | 0.0200 (7) | 0.0214 (8) | 0.0270 (8) | 0.0011 (6) | 0.0031 (6) | −0.0007 (7) |
| C18 | 0.0224 (7) | 0.0207 (8) | 0.0207 (8) | −0.0002 (6) | 0.0019 (6) | 0.0020 (6) |
| C19 | 0.0209 (7) | 0.0180 (8) | 0.0206 (8) | −0.0032 (6) | 0.0033 (6) | −0.0027 (6) |
| C20 | 0.0219 (7) | 0.0152 (8) | 0.0232 (8) | −0.0004 (6) | 0.0053 (6) | −0.0018 (6) |
| C21 | 0.0261 (8) | 0.0231 (9) | 0.0213 (8) | 0.0035 (6) | 0.0030 (7) | 0.0015 (7) |
| C22 | 0.0210 (8) | 0.0269 (9) | 0.0271 (8) | 0.0023 (6) | 0.0041 (7) | 0.0001 (7) |
| C23 | 0.0250 (8) | 0.0181 (8) | 0.0257 (8) | 0.0013 (6) | 0.0083 (7) | −0.0008 (7) |
| C24 | 0.0252 (8) | 0.0225 (9) | 0.0260 (8) | 0.0009 (6) | 0.0030 (7) | 0.0004 (7) |
| C25 | 0.0221 (8) | 0.0254 (9) | 0.0259 (8) | −0.0001 (6) | 0.0044 (7) | 0.0000 (7) |
| C26 | 0.0231 (8) | 0.0320 (9) | 0.0224 (8) | −0.0001 (7) | 0.0059 (6) | −0.0020 (7) |
| C27 | 0.0251 (8) | 0.0213 (9) | 0.0213 (8) | −0.0012 (6) | 0.0034 (6) | 0.0012 (6) |
| C28 | 0.0213 (7) | 0.0205 (8) | 0.0247 (8) | 0.0018 (6) | 0.0026 (6) | −0.0004 (6) |
| C29 | 0.0217 (7) | 0.0178 (8) | 0.0240 (8) | 0.0001 (6) | 0.0019 (6) | −0.0015 (6) |
| C30 | 0.0267 (8) | 0.0197 (8) | 0.0199 (8) | −0.0007 (6) | 0.0034 (6) | 0.0009 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C5 | 1.4647 (19) | C11—H11B | 0.9900 |
| N1—C1 | 1.4657 (19) | C12—H12A | 0.9800 |
| N1—C13 | 1.4741 (18) | C12—H12B | 0.9800 |
| N2—C7 | 1.4631 (19) | C12—H12C | 0.9800 |
| N2—C11 | 1.4666 (19) | C13—C14 | 1.478 (2) |
| N2—C26 | 1.4707 (18) | C13—H13A | 0.9900 |
| C1—C2 | 1.523 (2) | C13—H13B | 0.9900 |
| C1—H1A | 0.9900 | C14—C15 | 1.198 (2) |
| C1—H1B | 0.9900 | C15—C16 | 1.447 (2) |
| C2—C3 | 1.524 (2) | C16—C17 | 1.395 (2) |
| C2—H2A | 0.9900 | C16—C27 | 1.398 (2) |
| C2—H2B | 0.9900 | C17—C18 | 1.3880 (19) |
| C3—C4 | 1.526 (2) | C17—H17A | 0.9500 |
| C3—C6 | 1.5280 (19) | C18—C19 | 1.401 (2) |
| C3—H3A | 1.0000 | C18—H18A | 0.9500 |
| C4—C5 | 1.5252 (19) | C19—C28 | 1.3976 (19) |
| C4—H4A | 0.9900 | C19—C20 | 1.483 (2) |

| | | | |
|---|---|---|---|
| C4—H4B | 0.9900 | C20—C29 | 1.398 (2) |
| C5—H5A | 0.9900 | C20—C21 | 1.4019 (19) |
| C5—H5B | 0.9900 | C21—C22 | 1.3848 (19) |
| C6—H6A | 0.9800 | C21—H21A | 0.9500 |
| C6—H6B | 0.9800 | C22—C23 | 1.390 (2) |
| C6—H6C | 0.9800 | C22—H22A | 0.9500 |
| C7—C8 | 1.523 (2) | C23—C30 | 1.3984 (19) |
| C7—H7A | 0.9900 | C23—C24 | 1.443 (2) |
| C7—H7B | 0.9900 | C24—C25 | 1.197 (2) |
| C8—C9 | 1.527 (2) | C25—C26 | 1.481 (2) |
| C8—H8A | 0.9900 | C26—H26A | 0.9900 |
| C8—H8B | 0.9900 | C26—H26B | 0.9900 |
| C9—C10 | 1.520 (2) | C27—C28 | 1.3807 (19) |
| C9—C12 | 1.526 (2) | C27—H27A | 0.9500 |
| C9—H9A | 1.0000 | C28—H28A | 0.9500 |
| C10—C11 | 1.525 (2) | C29—C30 | 1.383 (2) |
| C10—H10A | 0.9900 | C29—H29A | 0.9500 |
| C10—H10B | 0.9900 | C30—H30A | 0.9500 |
| C11—H11A | 0.9900 | | |

| | | | |
|---|---|---|---|
| C5—N1—C1 | 111.28 (11) | H10A—C10—H10B | 108.0 |
| C5—N1—C13 | 110.47 (12) | N2—C11—C10 | 110.91 (12) |
| C1—N1—C13 | 110.65 (12) | N2—C11—H11A | 109.5 |
| C7—N2—C11 | 110.83 (11) | C10—C11—H11A | 109.5 |
| C7—N2—C26 | 110.99 (12) | N2—C11—H11B | 109.5 |
| C11—N2—C26 | 110.93 (12) | C10—C11—H11B | 109.5 |
| N1—C1—C2 | 111.08 (12) | H11A—C11—H11B | 108.0 |
| N1—C1—H1A | 109.4 | C9—C12—H12A | 109.5 |
| C2—C1—H1A | 109.4 | C9—C12—H12B | 109.5 |
| N1—C1—H1B | 109.4 | H12A—C12—H12B | 109.5 |
| C2—C1—H1B | 109.4 | C9—C12—H12C | 109.5 |
| H1A—C1—H1B | 108.0 | H12A—C12—H12C | 109.5 |
| C1—C2—C3 | 111.25 (12) | H12B—C12—H12C | 109.5 |
| C1—C2—H2A | 109.4 | N1—C13—C14 | 114.15 (12) |
| C3—C2—H2A | 109.4 | N1—C13—H13A | 108.7 |
| C1—C2—H2B | 109.4 | C14—C13—H13A | 108.7 |
| C3—C2—H2B | 109.4 | N1—C13—H13B | 108.7 |
| H2A—C2—H2B | 108.0 | C14—C13—H13B | 108.7 |
| C2—C3—C4 | 108.91 (12) | H13A—C13—H13B | 107.6 |
| C2—C3—C6 | 112.03 (13) | C15—C14—C13 | 176.56 (16) |
| C4—C3—C6 | 112.04 (13) | C14—C15—C16 | 175.24 (15) |
| C2—C3—H3A | 107.9 | C17—C16—C27 | 118.45 (13) |
| C4—C3—H3A | 107.9 | C17—C16—C15 | 122.39 (13) |
| C6—C3—H3A | 107.9 | C27—C16—C15 | 119.14 (13) |
| C5—C4—C3 | 110.95 (12) | C18—C17—C16 | 120.54 (13) |
| C5—C4—H4A | 109.4 | C18—C17—H17A | 119.7 |
| C3—C4—H4A | 109.4 | C16—C17—H17A | 119.7 |
| C5—C4—H4B | 109.4 | C17—C18—C19 | 121.25 (14) |

| | | | |
|---|---|---|---|
| C3—C4—H4B | 109.4 | C17—C18—H18A | 119.4 |
| H4A—C4—H4B | 108.0 | C19—C18—H18A | 119.4 |
| N1—C5—C4 | 110.99 (12) | C28—C19—C18 | 117.53 (13) |
| N1—C5—H5A | 109.4 | C28—C19—C20 | 120.70 (13) |
| C4—C5—H5A | 109.4 | C18—C19—C20 | 121.75 (13) |
| N1—C5—H5B | 109.4 | C29—C20—C21 | 117.70 (13) |
| C4—C5—H5B | 109.4 | C29—C20—C19 | 121.51 (13) |
| H5A—C5—H5B | 108.0 | C21—C20—C19 | 120.79 (13) |
| C3—C6—H6A | 109.5 | C22—C21—C20 | 121.44 (14) |
| C3—C6—H6B | 109.5 | C22—C21—H21A | 119.3 |
| H6A—C6—H6B | 109.5 | C20—C21—H21A | 119.3 |
| C3—C6—H6C | 109.5 | C21—C22—C23 | 120.46 (14) |
| H6A—C6—H6C | 109.5 | C21—C22—H22A | 119.8 |
| H6B—C6—H6C | 109.5 | C23—C22—H22A | 119.8 |
| N2—C7—C8 | 110.85 (12) | C22—C23—C30 | 118.49 (13) |
| N2—C7—H7A | 109.5 | C22—C23—C24 | 122.66 (13) |
| C8—C7—H7A | 109.5 | C30—C23—C24 | 118.85 (14) |
| N2—C7—H7B | 109.5 | C25—C24—C23 | 175.97 (16) |
| C8—C7—H7B | 109.5 | C24—C25—C26 | 179.41 (16) |
| H7A—C7—H7B | 108.1 | N2—C26—C25 | 114.80 (12) |
| C7—C8—C9 | 111.25 (12) | N2—C26—H26A | 108.6 |
| C7—C8—H8A | 109.4 | C25—C26—H26A | 108.6 |
| C9—C8—H8A | 109.4 | N2—C26—H26B | 108.6 |
| C7—C8—H8B | 109.4 | C25—C26—H26B | 108.6 |
| C9—C8—H8B | 109.4 | H26A—C26—H26B | 107.5 |
| H8A—C8—H8B | 108.0 | C28—C27—C16 | 120.67 (14) |
| C10—C9—C12 | 112.14 (13) | C28—C27—H27A | 119.7 |
| C10—C9—C8 | 108.91 (12) | C16—C27—H27A | 119.7 |
| C12—C9—C8 | 111.96 (12) | C27—C28—C19 | 121.47 (14) |
| C10—C9—H9A | 107.9 | C27—C28—H28A | 119.3 |
| C12—C9—H9A | 107.9 | C19—C28—H28A | 119.3 |
| C8—C9—H9A | 107.9 | C30—C29—C20 | 120.84 (14) |
| C9—C10—C11 | 111.35 (12) | C30—C29—H29A | 119.6 |
| C9—C10—H10A | 109.4 | C20—C29—H29A | 119.6 |
| C11—C10—H10A | 109.4 | C29—C30—C23 | 121.06 (14) |
| C9—C10—H10B | 109.4 | C29—C30—H30A | 119.5 |
| C11—C10—H10B | 109.4 | C23—C30—H30A | 119.5 |

| | | | |
|---|---|---|---|
| C5—N1—C1—C2 | 58.26 (15) | C16—C17—C18—C19 | 0.1 (2) |
| C13—N1—C1—C2 | −178.52 (12) | C17—C18—C19—C28 | 2.4 (2) |
| N1—C1—C2—C3 | −56.80 (17) | C17—C18—C19—C20 | −176.17 (13) |
| C1—C2—C3—C4 | 54.60 (16) | C28—C19—C20—C29 | 154.84 (14) |
| C1—C2—C3—C6 | 179.11 (12) | C18—C19—C20—C29 | −26.7 (2) |
| C2—C3—C4—C5 | −54.81 (16) | C28—C19—C20—C21 | −26.0 (2) |
| C6—C3—C4—C5 | −179.30 (12) | C18—C19—C20—C21 | 152.48 (14) |
| C1—N1—C5—C4 | −58.59 (15) | C29—C20—C21—C22 | −0.5 (2) |
| C13—N1—C5—C4 | 178.08 (12) | C19—C20—C21—C22 | −179.67 (14) |
| C3—C4—C5—N1 | 57.36 (17) | C20—C21—C22—C23 | −0.2 (2) |

| | | | |
|---|---|---|---|
| C11—N2—C7—C8 | −59.31 (15) | C21—C22—C23—C30 | 0.7 (2) |
| C26—N2—C7—C8 | 176.95 (12) | C21—C22—C23—C24 | −178.97 (14) |
| N2—C7—C8—C9 | 57.45 (17) | C7—N2—C26—C25 | 61.28 (17) |
| C7—C8—C9—C10 | −54.19 (16) | C11—N2—C26—C25 | −62.40 (17) |
| C7—C8—C9—C12 | −178.78 (12) | C17—C16—C27—C28 | 2.4 (2) |
| C12—C9—C10—C11 | 178.51 (12) | C15—C16—C27—C28 | −176.02 (13) |
| C8—C9—C10—C11 | 54.03 (16) | C16—C27—C28—C19 | 0.0 (2) |
| C7—N2—C11—C10 | 59.11 (15) | C18—C19—C28—C27 | −2.4 (2) |
| C26—N2—C11—C10 | −177.12 (12) | C20—C19—C28—C27 | 176.14 (13) |
| C9—C10—C11—N2 | −57.14 (17) | C21—C20—C29—C30 | 0.7 (2) |
| C5—N1—C13—C14 | 61.90 (17) | C19—C20—C29—C30 | 179.85 (13) |
| C1—N1—C13—C14 | −61.79 (17) | C20—C29—C30—C23 | −0.2 (2) |
| C27—C16—C17—C18 | −2.5 (2) | C22—C23—C30—C29 | −0.5 (2) |
| C15—C16—C17—C18 | 175.94 (14) | C24—C23—C30—C29 | 179.17 (13) |

### (II) 4,4′-Bis[3-(2,2,6,6-tetramethylpiperidin-1-yl)prop-1-yn-1-yl]-1,1′-biphenyl

*Crystal data*

$C_{36}H_{48}N_2$
$M_r$ = 508.76
Monoclinic, $P2_1/c$
$a$ = 16.0591 (3) Å
$b$ = 6.2267 (1) Å
$c$ = 15.5921 (3) Å
$\beta$ = 100.895 (1)°
$V$ = 1531.03 (5) Å$^3$
$Z$ = 2
$F(000)$ = 556
$D_x$ = 1.104 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 8231 reflections
$\theta$ = 2.8–67.9°
$\mu$ = 0.47 mm$^{-1}$
$T$ = 90 K
Needle, colourless
0.22 × 0.04 × 0.03 mm

*Data collection*

Bruker X8 Proteum diffractometer
Radiation source: fine-focus rotating anode
Detector resolution: 5.6 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min}$ = 0.767, $T_{max}$ = 0.929
19631 measured reflections
2797 independent reflections
2405 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.053
$\theta_{max}$ = 68.2°, $\theta_{min}$ = 2.8°
$h$ = −19→18
$k$ = −7→7
$l$ = −7→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
$S$ = 1.05
2797 reflections
176 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.6795P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 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 progress was checked using *PLATON* (Spek, 2009) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.78341 (8) | 0.0593 (2) | 0.79190 (7) | 0.0251 (3) |
| C1 | 0.83571 (10) | −0.0569 (3) | 0.73835 (10) | 0.0282 (4) |
| C2 | 0.92701 (10) | 0.0213 (3) | 0.76309 (11) | 0.0362 (4) |
| H2A | 0.9634 | −0.0696 | 0.7333 | 0.043* |
| H2B | 0.9303 | 0.1703 | 0.7418 | 0.043* |
| C3 | 0.96116 (11) | 0.0163 (3) | 0.86058 (12) | 0.0459 (5) |
| H3A | 1.0197 | 0.0734 | 0.8732 | 0.055* |
| H3B | 0.9623 | −0.1334 | 0.8822 | 0.055* |
| C4 | 0.90420 (12) | 0.1523 (3) | 0.90597 (11) | 0.0450 (5) |
| H4A | 0.9072 | 0.3034 | 0.8869 | 0.054* |
| H4B | 0.9258 | 0.1472 | 0.9698 | 0.054* |
| C5 | 0.81150 (11) | 0.0802 (3) | 0.88750 (9) | 0.0344 (4) |
| C6 | 0.69196 (10) | 0.0225 (3) | 0.76398 (11) | 0.0311 (4) |
| H6A | 0.6669 | −0.0110 | 0.8158 | 0.037* |
| H6B | 0.6829 | −0.1031 | 0.7244 | 0.037* |
| C7 | 0.64850 (9) | 0.2102 (3) | 0.71880 (10) | 0.0315 (4) |
| C8 | 0.61556 (10) | 0.3635 (3) | 0.68003 (10) | 0.0321 (4) |
| C9 | 0.58105 (9) | 0.5448 (3) | 0.62925 (10) | 0.0306 (4) |
| C10 | 0.52059 (11) | 0.6782 (3) | 0.65485 (12) | 0.0408 (4) |
| H10A | 0.5007 | 0.6487 | 0.7073 | 0.049* |
| C11 | 0.48936 (11) | 0.8531 (3) | 0.60457 (12) | 0.0401 (4) |
| H11A | 0.4478 | 0.9409 | 0.6232 | 0.048* |
| C12 | 0.51674 (9) | 0.9061 (3) | 0.52692 (10) | 0.0301 (4) |
| C13 | 0.57751 (13) | 0.7705 (4) | 0.50380 (12) | 0.0497 (5) |
| H13A | 0.5981 | 0.7997 | 0.4517 | 0.060* |
| C14 | 0.60900 (12) | 0.5960 (4) | 0.55329 (12) | 0.0478 (5) |
| H14A | 0.6509 | 0.5086 | 0.5349 | 0.057* |
| C15 | 0.80502 (11) | 0.0062 (3) | 0.64265 (10) | 0.0361 (4) |
| H15A | 0.8023 | 0.1630 | 0.6377 | 0.054* |
| H15B | 0.7485 | −0.0545 | 0.6217 | 0.054* |
| H15C | 0.8446 | −0.0496 | 0.6073 | 0.054* |
| C16 | 0.83244 (12) | −0.3037 (3) | 0.74548 (12) | 0.0384 (4) |
| H16A | 0.8630 | −0.3487 | 0.8031 | 0.058* |
| H16B | 0.8590 | −0.3685 | 0.7001 | 0.058* |
| H16C | 0.7732 | −0.3505 | 0.7378 | 0.058* |
| C17 | 0.80193 (14) | −0.1264 (3) | 0.93930 (11) | 0.0456 (5) |
| H17A | 0.8451 | −0.2307 | 0.9301 | 0.068* |

| | | | | |
|---|---|---|---|---|
| H17B | 0.7454 | −0.1875 | 0.9190 | 0.068* |
| H17C | 0.8092 | −0.0922 | 1.0016 | 0.068* |
| C18 | 0.75837 (15) | 0.2577 (3) | 0.91871 (11) | 0.0477 (5) |
| H18A | 0.7586 | 0.3850 | 0.8818 | 0.072* |
| H18B | 0.7825 | 0.2945 | 0.9794 | 0.072* |
| H18C | 0.7000 | 0.2071 | 0.9149 | 0.072* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0261 (6) | 0.0330 (7) | 0.0174 (6) | −0.0037 (5) | 0.0076 (5) | 0.0001 (5) |
| C1 | 0.0288 (8) | 0.0347 (8) | 0.0231 (7) | 0.0026 (6) | 0.0103 (6) | 0.0023 (6) |
| C2 | 0.0262 (8) | 0.0442 (10) | 0.0398 (9) | 0.0021 (7) | 0.0102 (7) | 0.0145 (8) |
| C3 | 0.0311 (9) | 0.0553 (12) | 0.0456 (10) | −0.0071 (8) | −0.0075 (8) | 0.0224 (9) |
| C4 | 0.0568 (11) | 0.0472 (11) | 0.0241 (8) | −0.0167 (9) | −0.0104 (7) | 0.0098 (7) |
| C5 | 0.0508 (10) | 0.0364 (9) | 0.0169 (7) | −0.0088 (7) | 0.0088 (6) | 0.0027 (6) |
| C6 | 0.0267 (8) | 0.0330 (8) | 0.0359 (8) | −0.0049 (6) | 0.0120 (6) | −0.0015 (7) |
| C7 | 0.0241 (7) | 0.0389 (9) | 0.0335 (8) | −0.0034 (7) | 0.0103 (6) | −0.0075 (7) |
| C8 | 0.0243 (7) | 0.0387 (9) | 0.0347 (8) | −0.0037 (7) | 0.0092 (6) | −0.0092 (7) |
| C9 | 0.0217 (7) | 0.0372 (9) | 0.0325 (8) | −0.0017 (6) | 0.0040 (6) | −0.0091 (7) |
| C10 | 0.0374 (9) | 0.0434 (10) | 0.0475 (10) | 0.0033 (8) | 0.0227 (8) | −0.0001 (8) |
| C11 | 0.0343 (9) | 0.0417 (10) | 0.0500 (10) | 0.0072 (7) | 0.0222 (8) | −0.0021 (8) |
| C12 | 0.0222 (7) | 0.0376 (9) | 0.0306 (8) | −0.0012 (6) | 0.0048 (6) | −0.0111 (7) |
| C13 | 0.0514 (11) | 0.0697 (14) | 0.0335 (9) | 0.0281 (10) | 0.0221 (8) | 0.0066 (9) |
| C14 | 0.0464 (11) | 0.0649 (13) | 0.0363 (9) | 0.0264 (9) | 0.0186 (8) | 0.0009 (9) |
| C15 | 0.0453 (10) | 0.0444 (10) | 0.0203 (7) | 0.0054 (8) | 0.0105 (7) | −0.0009 (7) |
| C16 | 0.0436 (10) | 0.0342 (9) | 0.0400 (9) | 0.0042 (7) | 0.0148 (7) | 0.0009 (7) |
| C17 | 0.0688 (13) | 0.0434 (11) | 0.0286 (8) | −0.0070 (9) | 0.0193 (8) | 0.0090 (8) |
| C18 | 0.0838 (15) | 0.0393 (10) | 0.0254 (8) | −0.0073 (10) | 0.0239 (9) | −0.0047 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C6 | 1.4688 (19) | C9—C10 | 1.392 (2) |
| N1—C5 | 1.4793 (18) | C10—C11 | 1.380 (3) |
| N1—C1 | 1.4804 (19) | C10—H10A | 0.9500 |
| C1—C2 | 1.524 (2) | C11—C12 | 1.403 (2) |
| C1—C15 | 1.532 (2) | C11—H11A | 0.9500 |
| C1—C16 | 1.542 (2) | C12—C13 | 1.389 (2) |
| C2—C3 | 1.516 (2) | C12—C12$^{i}$ | 1.480 (3) |
| C2—H2A | 0.9900 | C13—C14 | 1.373 (3) |
| C2—H2B | 0.9900 | C13—H13A | 0.9500 |
| C3—C4 | 1.517 (3) | C14—H14A | 0.9500 |
| C3—H3A | 0.9900 | C15—H15A | 0.9800 |
| C3—H3B | 0.9900 | C15—H15B | 0.9800 |
| C4—C5 | 1.529 (2) | C15—H15C | 0.9800 |
| C4—H4A | 0.9900 | C16—H16A | 0.9800 |
| C4—H4B | 0.9900 | C16—H16B | 0.9800 |
| C5—C18 | 1.531 (3) | C16—H16C | 0.9800 |

| | | | |
|---|---|---|---|
| C5—C17 | 1.542 (2) | C17—H17A | 0.9800 |
| C6—C7 | 1.471 (2) | C17—H17B | 0.9800 |
| C6—H6A | 0.9900 | C17—H17C | 0.9800 |
| C6—H6B | 0.9900 | C18—H18A | 0.9800 |
| C7—C8 | 1.198 (2) | C18—H18B | 0.9800 |
| C8—C9 | 1.429 (2) | C18—H18C | 0.9800 |
| C9—C14 | 1.381 (2) | | |

| | | | |
|---|---|---|---|
| C6—N1—C5 | 114.19 (12) | C14—C9—C8 | 120.16 (15) |
| C6—N1—C1 | 113.49 (12) | C10—C9—C8 | 122.07 (15) |
| C5—N1—C1 | 120.92 (13) | C11—C10—C9 | 120.43 (16) |
| N1—C1—C2 | 108.79 (13) | C11—C10—H10A | 119.8 |
| N1—C1—C15 | 108.16 (12) | C9—C10—H10A | 119.8 |
| C2—C1—C15 | 106.36 (13) | C10—C11—C12 | 122.43 (15) |
| N1—C1—C16 | 114.57 (13) | C10—C11—H11A | 118.8 |
| C2—C1—C16 | 110.15 (14) | C12—C11—H11A | 118.8 |
| C15—C1—C16 | 108.47 (14) | C13—C12—C11 | 115.55 (16) |
| C3—C2—C1 | 113.30 (13) | C13—C12—C12$^{i}$ | 122.06 (18) |
| C3—C2—H2A | 108.9 | C11—C12—C12$^{i}$ | 122.39 (17) |
| C1—C2—H2A | 108.9 | C14—C13—C12 | 122.53 (16) |
| C3—C2—H2B | 108.9 | C14—C13—H13A | 118.7 |
| C1—C2—H2B | 108.9 | C12—C13—H13A | 118.7 |
| H2A—C2—H2B | 107.7 | C13—C14—C9 | 121.32 (16) |
| C2—C3—C4 | 108.70 (14) | C13—C14—H14A | 119.3 |
| C2—C3—H3A | 109.9 | C9—C14—H14A | 119.3 |
| C4—C3—H3A | 109.9 | C1—C15—H15A | 109.5 |
| C2—C3—H3B | 109.9 | C1—C15—H15B | 109.5 |
| C4—C3—H3B | 109.9 | H15A—C15—H15B | 109.5 |
| H3A—C3—H3B | 108.3 | C1—C15—H15C | 109.5 |
| C3—C4—C5 | 113.55 (15) | H15A—C15—H15C | 109.5 |
| C3—C4—H4A | 108.9 | H15B—C15—H15C | 109.5 |
| C5—C4—H4A | 108.9 | C1—C16—H16A | 109.5 |
| C3—C4—H4B | 108.9 | C1—C16—H16B | 109.5 |
| C5—C4—H4B | 108.9 | H16A—C16—H16B | 109.5 |
| H4A—C4—H4B | 107.7 | C1—C16—H16C | 109.5 |
| N1—C5—C4 | 108.43 (12) | H16A—C16—H16C | 109.5 |
| N1—C5—C18 | 107.54 (14) | H16B—C16—H16C | 109.5 |
| C4—C5—C18 | 108.02 (15) | C5—C17—H17A | 109.5 |
| N1—C5—C17 | 114.40 (14) | C5—C17—H17B | 109.5 |
| C4—C5—C17 | 109.65 (15) | H17A—C17—H17B | 109.5 |
| C18—C5—C17 | 108.61 (14) | C5—C17—H17C | 109.5 |
| N1—C6—C7 | 112.04 (13) | H17A—C17—H17C | 109.5 |
| N1—C6—H6A | 109.2 | H17B—C17—H17C | 109.5 |
| C7—C6—H6A | 109.2 | C5—C18—H18A | 109.5 |
| N1—C6—H6B | 109.2 | C5—C18—H18B | 109.5 |
| C7—C6—H6B | 109.2 | H18A—C18—H18B | 109.5 |
| H6A—C6—H6B | 107.9 | C5—C18—H18C | 109.5 |
| C8—C7—C6 | 177.40 (16) | H18A—C18—H18C | 109.5 |

| | | | |
|---|---|---|---|
| C7—C8—C9 | 175.42 (16) | H18B—C18—H18C | 109.5 |
| C14—C9—C10 | 117.74 (16) | | |
| | | | |
| C6—N1—C1—C2 | 170.72 (12) | C1—N1—C5—C17 | −75.06 (19) |
| C5—N1—C1—C2 | −47.93 (18) | C3—C4—C5—N1 | −50.81 (19) |
| C6—N1—C1—C15 | 55.59 (17) | C3—C4—C5—C18 | −167.07 (14) |
| C5—N1—C1—C15 | −163.06 (14) | C3—C4—C5—C17 | 74.74 (17) |
| C6—N1—C1—C16 | −65.52 (17) | C5—N1—C6—C7 | 109.77 (15) |
| C5—N1—C1—C16 | 75.83 (18) | C1—N1—C6—C7 | −106.21 (15) |
| N1—C1—C2—C3 | 51.04 (19) | C14—C9—C10—C11 | 1.0 (3) |
| C15—C1—C2—C3 | 167.34 (15) | C8—C9—C10—C11 | 179.33 (16) |
| C16—C1—C2—C3 | −75.31 (19) | C9—C10—C11—C12 | −0.6 (3) |
| C1—C2—C3—C4 | −57.6 (2) | C10—C11—C12—C13 | 0.0 (3) |
| C2—C3—C4—C5 | 57.58 (19) | C10—C11—C12—C12$^{i}$ | −179.94 (18) |
| C6—N1—C5—C4 | −171.25 (14) | C11—C12—C13—C14 | 0.0 (3) |
| C1—N1—C5—C4 | 47.65 (19) | C12$^{i}$—C12—C13—C14 | 180.0 (2) |
| C6—N1—C5—C18 | −54.69 (17) | C12—C13—C14—C9 | 0.5 (3) |
| C1—N1—C5—C18 | 164.21 (14) | C10—C9—C14—C13 | −0.9 (3) |
| C6—N1—C5—C17 | 66.04 (19) | C8—C9—C14—C13 | −179.33 (18) |

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