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Crystal structure of 4,4-dibutyl-2-phenyl-3,4-dihydroquinazoline

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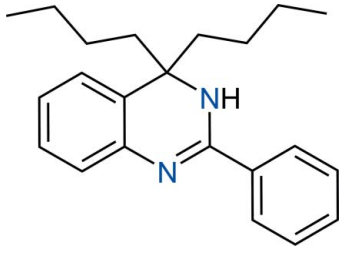
In the title compound, $C_{22}H_{28}N_2$, the dihedral angle between the planes of the phenyl ring and the dihydroquinazoline ring system (r.m.s. deviation = 0.030 Å) is 24.95 (7)° and both *n*-butane chains assume all-*trans* conformations. In the crystal, N—H···N hydrogen bonds link the molecules into *C*(4) chains propagating in the [001] direction.

Keywords: crystal structure; quinazoline; hydrogen bonding.

CCDC reference: 1022964

1. Related literature

For the synthesis of 4,4-dibutyl-2-phenyl-3,4-dihydroquinazoline, see: Smith *et al.* (2005); Plé *et al.* (1997). For the crystal structures of related compounds, see Valkonen *et al.* (2011); Derabli *et al.* (2013).



2. Experimental

2.1. Crystal data

$C_{22}H_{28}N_2$
M_r = 320.46
Monoclinic, $P2_1/c$
a = 19.2953 (8) Å
b = 9.9889 (3) Å
c = 9.6341 (4) Å
β = 96.667 (4)°
V = 1844.31 (12) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 150 K
0.41 × 0.13 × 0.04 mm

2.2. Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.829, T_{max} = 1.000
12894 measured reflections
3657 independent reflections
2866 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 1.04
3657 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N2^{i}	0.88	2.29	3.1239 (16)	157

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001); software used to prepare material for publication: *SHELXL2013*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7281).

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supporting information

Acta Cryst. (2014). E**70**, o1100 [doi:10.1107/S1600536814020017]

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S1. Chemical context

S2. Structural commentary

In the molecule of $C_{22}H_{28}N_2$ (Fig. 1), the phenyl ring is twisted by 24.95 (7) from the plane of the dihydroquinazoline group. Both *n*-butane chains assume all-*trans* conformation. N—H···N hydrogen bonds between neigbouring molecules form chains parallel to the *c*-axis (Fig. 2).

4,4-Dibutyl-2-phenyl-3,4-dihydroquinazoline can be obtained from reaction of two mole equivalents of *n*-butyllithium with 4-(methylthio)-2-phenylquinazoline at –78°C in anhydrous THF [Smith *et al.* (2005); Plé *et al.* (1997)]. The reaction involves initial addition of *n*-butyllithium at the 4-position of quinazoline ring followed by elimination of methanethiolate anion and then further addition of *n*-butyllithium (Smith *et al.*, 2005). For the X-ray structures of related compounds, see Valkonen *et al.* (2011); Derabli *et al.* (2013).

S3. Supramolecular features

S4. Database survey

S5. Synthesis and crystallization

4,4-Dibutyl-2-phenyl-3,4-dihydroquinazoline

A solution of *n*-butyllithium in hexane (1.76 mL, 2.5 M, 4.4 mmol) was added to a cold (–78 ^{o}C), stirred solution of 4-(methylthio)-2-phenylquinazoline (0.50 g, 2.0 mmol) in anhydrous THF (10 mL) under N_2. The reaction mixture was stirred at –78 ^{o}C for 1 h then removed from the cooling bath and allowed to warm to room temperature, diluted with diethyl ether (10 mL), then quenched with aqueous saturated NH_4Cl (10 mL). The organic layer was separated, washed with water (2 x 10 mL), dried ($MgSO_4$), and evaporated under reduced pressure. The residue obtained was purified by column chromatography (silica gel; diethyl ether–hexane, 1:4 by volume) to give 4,4-dibutyl-2-phenyl-3,4-dihydroquinazoline in 96% yield, m.p. 161 ^{o}C [lit. 161 ^{o}C: Smith *et al.* (2005); 154–155 ^{o}C: Plé *et al.* (1997)]. Crystallization from a mixture of ethyl acetate and diethyl ether (1:3 by volume) gave the title compound as colorless crystals. The spectroscopic data for the title compound, including NMR and low and high resolution mass spectra, were consistent with those reported [Smith *et al.* (2005)].

S6. Refinement

H atoms were positioned geometrically and refined using a riding model, with U_{iso}(H) constrained to be 1.2 times U_{eq} for the bonded atom except for methyl groups where U_{iso}(H) was 1.5 times and free rotation about the C—C bond was allowed. Crystal data, data collection and structure refinement details are summarized in the table.

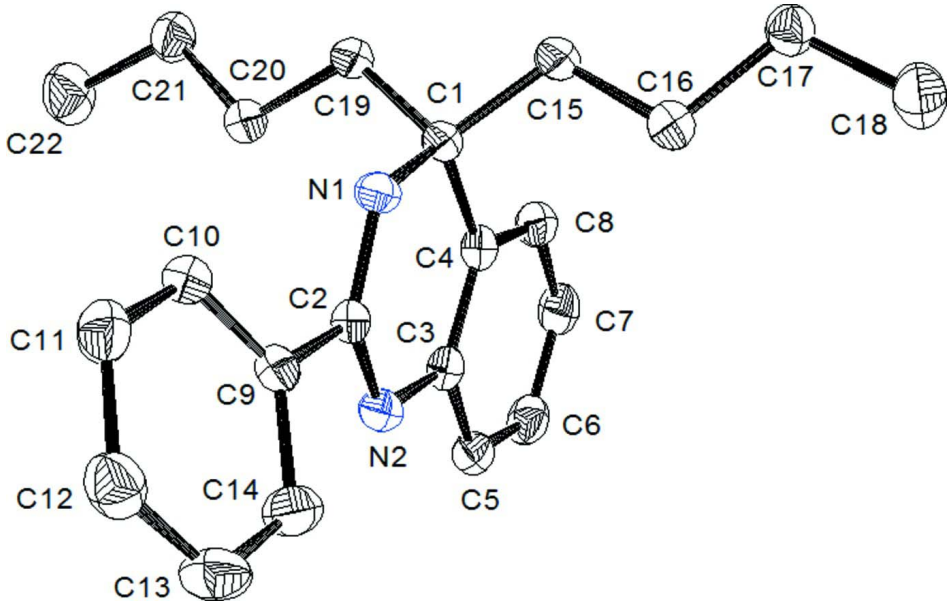

Figure 1

The asymmetric unit of the title compound with 50% probability displacement ellipsoids.

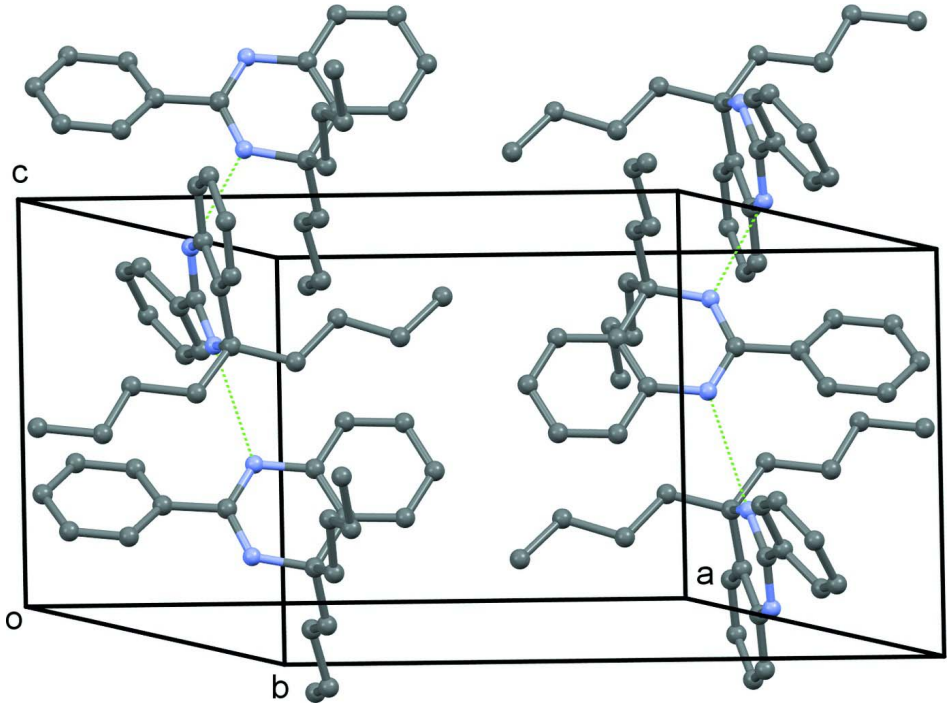

Figure 2

Packing in the crystal structure showing N—H···N contacts as dotted lines with hydrogen atoms omitted for clarity.

4,4-Dibutyl-2-phenyl-3,4-dihydroquinazoline

Crystal data

$C_{22}H_{28}N_2$
$M_r = 320.46$
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$c = 9.6341$ (4) Å
$\beta = 96.667$ (4)°
$V = 1844.31$ (12) Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.154$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 3898 reflections
$\theta = 4.6$–$73.6°$
$\mu = 0.51$ mm^{-1}
$T = 150$ K
Plate, colourless
0.41 × 0.13 × 0.04 mm

Data collection

SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.829$, $T_{max} = 1.000$
12894 measured reflections
3657 independent reflections
2866 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 73.6°$, $\theta_{min} = 4.6°$
$h = -23 \rightarrow 23$
$k = -12 \rightarrow 12$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.129$
$S = 1.04$
3657 reflections
219 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.3637P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.36.28 (release 01-02-2013 CrysAlis171 .NET) (compiled Feb 1 2013,16:14:44) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.28102 (8)	0.40404 (13)	0.14783 (14)	0.0259 (3)
C2	0.18929 (7)	0.30261 (13)	0.27980 (14)	0.0246 (3)
C3	0.26279 (8)	0.45814 (13)	0.39845 (15)	0.0272 (3)
C4	0.29777 (8)	0.48405 (13)	0.28214 (15)	0.0273 (3)
C5	0.28292 (9)	0.52724 (14)	0.52321 (16)	0.0314 (3)
H5	0.2592	0.5103	0.6025	0.038*
C6	0.33672 (9)	0.61972 (15)	0.53300 (17)	0.0356 (4)
H6	0.3500	0.6650	0.6187	0.043*

C7	0.37112 (9)	0.64606 (15)	0.41752 (18)	0.0358 (4)
H7	0.4078	0.7099	0.4232	0.043*
C8	0.35142 (8)	0.57829 (15)	0.29334 (17)	0.0326 (3)
H8	0.3750	0.5966	0.2142	0.039*
C9	0.12820 (8)	0.20985 (13)	0.27592 (14)	0.0259 (3)
C10	0.11781 (8)	0.10256 (14)	0.18380 (16)	0.0293 (3)
H10	0.1501	0.0869	0.1183	0.035*
C11	0.06087 (9)	0.01805 (15)	0.18637 (17)	0.0347 (4)
H11	0.0544	−0.0546	0.1225	0.042*
C12	0.01348 (9)	0.03893 (17)	0.28127 (18)	0.0369 (4)
H12	−0.0251	−0.0198	0.2837	0.044*
C13	0.02269 (9)	0.14620 (18)	0.37290 (18)	0.0399 (4)
H13	−0.0099	0.1616	0.4378	0.048*
C14	0.07937 (9)	0.23099 (16)	0.36999 (17)	0.0346 (4)
H14	0.0851	0.3045	0.4328	0.041*
C15	0.26253 (8)	0.49478 (14)	0.01923 (15)	0.0286 (3)
H15A	0.3044	0.5479	0.0049	0.034*
H15B	0.2520	0.4366	−0.0637	0.034*
C16	0.20181 (9)	0.59109 (15)	0.02432 (16)	0.0322 (3)
H16A	0.2107	0.6483	0.1084	0.039*
H16B	0.1586	0.5394	0.0319	0.039*
C17	0.19140 (8)	0.67945 (14)	−0.10553 (16)	0.0309 (3)
H17A	0.2352	0.7288	−0.1141	0.037*
H17B	0.1817	0.6217	−0.1891	0.037*
C18	0.13220 (9)	0.77946 (17)	−0.10306 (19)	0.0410 (4)
H18A	0.0880	0.7313	−0.1029	0.061*
H18B	0.1300	0.8369	−0.1859	0.061*
H18C	0.1404	0.8347	−0.0187	0.061*
C19	0.34439 (8)	0.31704 (14)	0.12029 (15)	0.0284 (3)
H19A	0.3305	0.2605	0.0373	0.034*
H19B	0.3820	0.3773	0.0966	0.034*
C20	0.37407 (8)	0.22668 (15)	0.23908 (16)	0.0322 (3)
H20A	0.3375	0.1632	0.2613	0.039*
H20B	0.3877	0.2818	0.3233	0.039*
C21	0.43742 (9)	0.14803 (16)	0.20321 (18)	0.0380 (4)
H21A	0.4249	0.0997	0.1141	0.046*
H21B	0.4755	0.2115	0.1894	0.046*
C22	0.46367 (11)	0.0478 (2)	0.3163 (2)	0.0534 (5)
H22A	0.4806	0.0958	0.4023	0.080*
H22B	0.5018	−0.0049	0.2851	0.080*
H22C	0.4255	−0.0121	0.3343	0.080*
N1	0.22079 (6)	0.31594 (12)	0.16295 (12)	0.0269 (3)
H1	0.2042	0.2684	0.0897	0.032*
N2	0.20764 (7)	0.36574 (12)	0.39726 (12)	0.0281 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0316 (8)	0.0222 (6)	0.0253 (7)	−0.0012 (5)	0.0088 (6)	0.0013 (5)
C2	0.0291 (7)	0.0207 (6)	0.0248 (7)	0.0044 (5)	0.0060 (5)	0.0031 (5)
C3	0.0328 (8)	0.0205 (6)	0.0285 (7)	0.0035 (5)	0.0049 (6)	0.0009 (5)
C4	0.0322 (7)	0.0201 (6)	0.0297 (7)	0.0024 (5)	0.0045 (6)	0.0022 (5)
C5	0.0408 (9)	0.0265 (7)	0.0273 (7)	0.0023 (6)	0.0054 (6)	−0.0007 (6)
C6	0.0445 (9)	0.0260 (7)	0.0349 (8)	0.0018 (6)	−0.0013 (7)	−0.0062 (6)
C7	0.0369 (9)	0.0247 (7)	0.0456 (9)	−0.0037 (6)	0.0032 (7)	−0.0020 (6)
C8	0.0376 (8)	0.0256 (7)	0.0357 (8)	−0.0009 (6)	0.0089 (7)	0.0010 (6)
C9	0.0297 (7)	0.0240 (6)	0.0243 (7)	0.0022 (5)	0.0049 (6)	0.0050 (5)
C10	0.0344 (8)	0.0264 (7)	0.0287 (7)	0.0016 (6)	0.0104 (6)	0.0019 (5)
C11	0.0389 (9)	0.0254 (7)	0.0404 (9)	−0.0018 (6)	0.0070 (7)	−0.0001 (6)
C12	0.0320 (8)	0.0353 (8)	0.0440 (9)	−0.0029 (6)	0.0068 (7)	0.0066 (7)
C13	0.0338 (9)	0.0490 (10)	0.0398 (9)	−0.0008 (7)	0.0168 (7)	−0.0003 (7)
C14	0.0366 (9)	0.0372 (8)	0.0313 (8)	0.0006 (6)	0.0101 (6)	−0.0039 (6)
C15	0.0349 (8)	0.0257 (7)	0.0265 (7)	−0.0016 (6)	0.0092 (6)	0.0023 (5)
C16	0.0378 (8)	0.0286 (7)	0.0313 (8)	0.0019 (6)	0.0083 (6)	0.0035 (6)
C17	0.0358 (8)	0.0264 (7)	0.0305 (8)	−0.0011 (6)	0.0036 (6)	0.0011 (6)
C18	0.0396 (9)	0.0356 (8)	0.0477 (10)	0.0043 (7)	0.0053 (7)	0.0087 (7)
C19	0.0334 (8)	0.0265 (7)	0.0267 (7)	−0.0005 (6)	0.0094 (6)	0.0009 (5)
C20	0.0380 (8)	0.0292 (7)	0.0301 (8)	0.0036 (6)	0.0075 (6)	0.0020 (6)
C21	0.0387 (9)	0.0322 (8)	0.0443 (9)	0.0047 (7)	0.0095 (7)	0.0024 (7)
C22	0.0478 (11)	0.0497 (11)	0.0628 (12)	0.0166 (9)	0.0060 (9)	0.0133 (9)
N1	0.0345 (7)	0.0247 (6)	0.0227 (6)	−0.0036 (5)	0.0084 (5)	−0.0009 (4)
N2	0.0352 (7)	0.0253 (6)	0.0248 (6)	−0.0012 (5)	0.0079 (5)	−0.0002 (5)

Geometric parameters (Å, º)

C1—N1	1.4783 (18)	C13—H13	0.9500
C1—C4	1.523 (2)	C14—H14	0.9500
C1—C15	1.5432 (19)	C15—C16	1.521 (2)
C1—C19	1.5480 (19)	C15—H15A	0.9900
C2—N2	1.3079 (19)	C15—H15B	0.9900
C2—N1	1.3466 (18)	C16—C17	1.525 (2)
C2—C9	1.496 (2)	C16—H16A	0.9900
C3—C4	1.398 (2)	C16—H16B	0.9900
C3—C5	1.401 (2)	C17—C18	1.520 (2)
C3—N2	1.4078 (19)	C17—H17A	0.9900
C4—C8	1.394 (2)	C17—H17B	0.9900
C5—C6	1.385 (2)	C18—H18A	0.9800
C5—H5	0.9500	C18—H18B	0.9800
C6—C7	1.385 (2)	C18—H18C	0.9800
C6—H6	0.9500	C19—C20	1.517 (2)
C7—C8	1.389 (2)	C19—H19A	0.9900
C7—H7	0.9500	C19—H19B	0.9900
C8—H8	0.9500	C20—C21	1.526 (2)

C9—C10	1.391 (2)	C20—H20A	0.9900
C9—C14	1.397 (2)	C20—H20B	0.9900
C10—C11	1.388 (2)	C21—C22	1.523 (2)
C10—H10	0.9500	C21—H21A	0.9900
C11—C12	1.382 (2)	C21—H21B	0.9900
C11—H11	0.9500	C22—H22A	0.9800
C12—C13	1.387 (2)	C22—H22B	0.9800
C12—H12	0.9500	C22—H22C	0.9800
C13—C14	1.386 (2)	N1—H1	0.8800

N1—C1—C4	108.69 (11)	C1—C15—H15B	108.1
N1—C1—C15	108.52 (12)	H15A—C15—H15B	107.3
C4—C1—C15	112.35 (11)	C15—C16—C17	111.60 (12)
N1—C1—C19	109.17 (11)	C15—C16—H16A	109.3
C4—C1—C19	110.25 (12)	C17—C16—H16A	109.3
C15—C1—C19	107.80 (11)	C15—C16—H16B	109.3
N2—C2—N1	124.93 (13)	C17—C16—H16B	109.3
N2—C2—C9	116.96 (12)	H16A—C16—H16B	108.0
N1—C2—C9	118.10 (12)	C18—C17—C16	113.28 (13)
C4—C3—C5	119.01 (14)	C18—C17—H17A	108.9
C4—C3—N2	123.35 (13)	C16—C17—H17A	108.9
C5—C3—N2	117.64 (13)	C18—C17—H17B	108.9
C8—C4—C3	119.10 (14)	C16—C17—H17B	108.9
C8—C4—C1	120.17 (13)	H17A—C17—H17B	107.7
C3—C4—C1	120.61 (13)	C17—C18—H18A	109.5
C6—C5—C3	121.19 (14)	C17—C18—H18B	109.5
C6—C5—H5	119.4	H18A—C18—H18B	109.5
C3—C5—H5	119.4	C17—C18—H18C	109.5
C5—C6—C7	119.83 (15)	H18A—C18—H18C	109.5
C5—C6—H6	120.1	H18B—C18—H18C	109.5
C7—C6—H6	120.1	C20—C19—C1	116.19 (12)
C6—C7—C8	119.38 (15)	C20—C19—H19A	108.2
C6—C7—H7	120.3	C1—C19—H19A	108.2
C8—C7—H7	120.3	C20—C19—H19B	108.2
C7—C8—C4	121.49 (14)	C1—C19—H19B	108.2
C7—C8—H8	119.3	H19A—C19—H19B	107.4
C4—C8—H8	119.3	C19—C20—C21	112.19 (12)
C10—C9—C14	118.18 (14)	C19—C20—H20A	109.2
C10—C9—C2	123.21 (12)	C21—C20—H20A	109.2
C14—C9—C2	118.61 (13)	C19—C20—H20B	109.2
C11—C10—C9	120.86 (13)	C21—C20—H20B	109.2
C11—C10—H10	119.6	H20A—C20—H20B	107.9
C9—C10—H10	119.6	C22—C21—C20	112.66 (14)
C12—C11—C10	120.39 (15)	C22—C21—H21A	109.1
C12—C11—H11	119.8	C20—C21—H21A	109.1
C10—C11—H11	119.8	C22—C21—H21B	109.1
C11—C12—C13	119.48 (15)	C20—C21—H21B	109.1
C11—C12—H12	120.3	H21A—C21—H21B	107.8

C13—C12—H12	120.3	C21—C22—H22A	109.5
C14—C13—C12	120.16 (14)	C21—C22—H22B	109.5
C14—C13—H13	119.9	H22A—C22—H22B	109.5
C12—C13—H13	119.9	C21—C22—H22C	109.5
C13—C14—C9	120.92 (15)	H22A—C22—H22C	109.5
C13—C14—H14	119.5	H22B—C22—H22C	109.5
C9—C14—H14	119.5	C2—N1—C1	125.27 (12)
C16—C15—C1	116.92 (12)	C2—N1—H1	117.4
C16—C15—H15A	108.1	C1—N1—H1	117.4
C1—C15—H15A	108.1	C2—N2—C3	116.84 (12)
C16—C15—H15B	108.1		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2^{i}	0.88	2.29	3.1239 (16)	157

Symmetry code: (i) x, $-y+1/2$, $z-1/2$.