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1,3,3-Tribenzylindolin-2-one

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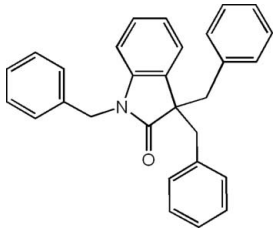
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.033; wR factor = 0.076; data-to-parameter ratio = 7.4.

In the title compound, $C_{29}H_{25}NO$, the dihedral angles between the indolin-2-one ring system and the three benzene rings are 62.78 (9), 31.69 (9) and 80.94 (9)°.

Related literature

For general background to the use of indoline-2-one compounds as precursors for the synthesis of antitumor agents, see: Wang *et al.* (2011). For a related structure, see: Katritzky *et al.* (1997).



Experimental

Crystal data

$C_{29}H_{25}NO$
M_r = 403.50
Monoclinic, $P2_1$
a = 8.3387 (9) Å
b = 9.6266 (10) Å
c = 13.9398 (14) Å
β = 99.442 (2)°
V = 1103.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 296 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.986
4797 measured reflections
2073 independent reflections
1731 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.076
S = 1.05
2073 reflections
281 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5107).

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Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
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Wang, S. B., Zhao, Y. F., Zhang, G. G., Lv, Y. X., Zhang, N. & Gong, P. (2011). *Eur. J. Med. Chem.* **8**, 3509–3518.

supporting information

Acta Cryst. (2011). E**67**, o2998 [doi:10.1107/S1600536811042425]

1,3,3-Tribenzylindolin-2-one

Yufang Liu, Bo Liu, Zhenming Dong and Shuo Jin

S1. Comment

Indoline-2-one compounds have been widely explored as precursors for the synthesis of antitumor agent (Wang *et al.*, 2011). In the course of exploring new antitumor medicine, we obtained a intermediate compound $C_{29}H_{25}NO$ (I), the synthesis and structure of which are reported here.

The title compound contain four ring planes, three benzene rings and one indoline-2-one ring. The interplanar dihedral angle between the indolin-2-one ring plane and the three benzene ring planes are 62.78 (9)°, 31.69 (9)° and 80.94 (9)\ respectively.

The molecules of (I) crystallize in the space group $P2_1$ which is different from that of 3-(1,2-diphenylethylidene)indolin-2-one (*P*-1) (Katritzky *et al.* 1997).

S2. Experimental

Indolin-2-one (0.50 g, 3.76 mmol) was dissolved in THF (20 mL) and KOH (0.80 g, 14.3 mmol) was slowly added. After heating the stirred mixture at reflux temperature for 30 min, a solution of 1-(chloromethyl)benzene (2.00 g, 15.9 mmol) in THF was slowly added and the refluxing continued for 2 h. The mixture was then cooled to 333 K and poured into water (200 mL) and was extracted with chloroform and dried over Na_2SO_4. After removing the solvent, the crude product was purified by column chromatography on silica gel, affording the title compound (yield: 0.23 g, 15%). The compound was then dissolved in THF, and colorless crystals were formed on slow evaporation at room temperature over one week.

S3. Refinement

All H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

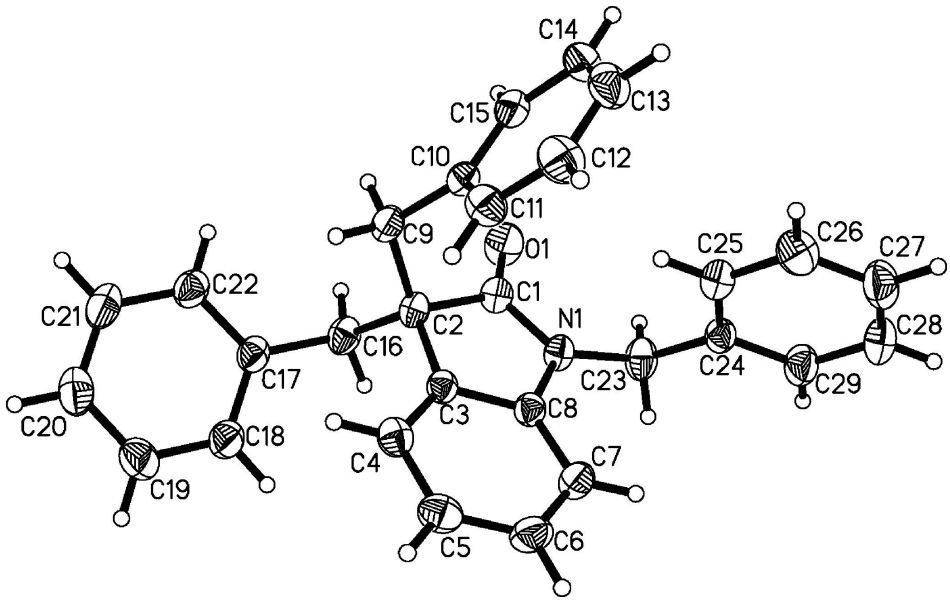

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

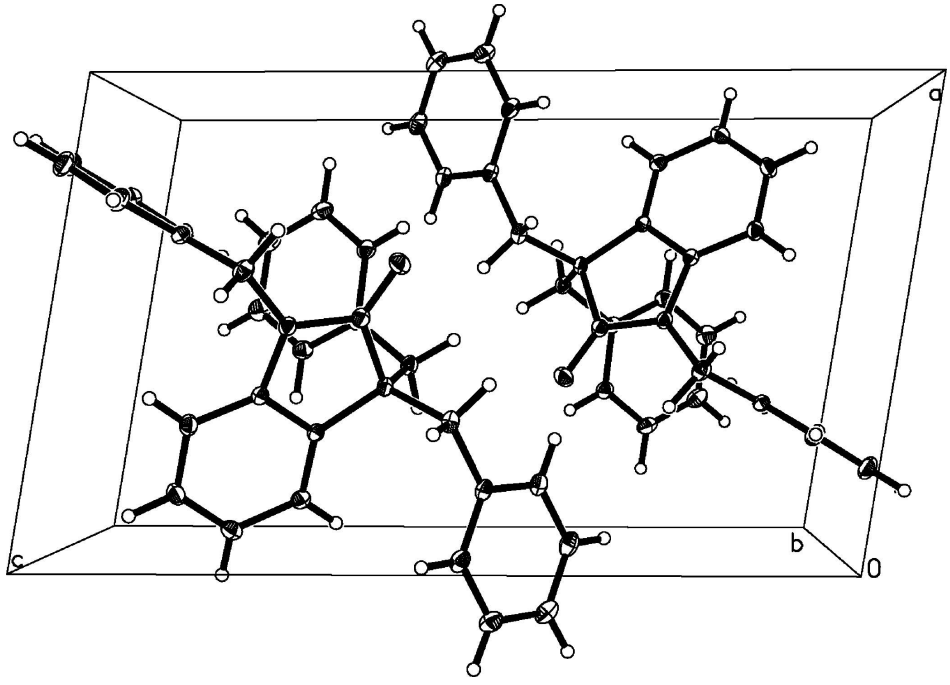

Figure 2

The molecular packing of (I) viewed along the *b* axis.

1,3,3-Tribenzylindolin-2-one

Crystal data

$C_{29}H_{25}NO$
$M_r = 403.50$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 8.3387$ (9) Å
$b = 9.6266$ (10) Å
$c = 13.9398$ (14) Å
$\beta = 99.442$ (2)°
$V = 1103.8$ (2) Å^3
$Z = 2$
$F(000) = 428$
$D_x = 1.214$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2492 reflections
$\theta = 2.9$–$26.3°$
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Block, yellow
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.979$, $T_{max} = 0.986$
4797 measured reflections
2073 independent reflections
1731 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.5°$
$h = -9 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.076$
$S = 1.05$
2073 reflections
281 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.10$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.085 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.3706 (2)	−0.1015 (2)	0.38221 (12)	0.0693 (5)
N1	0.5070 (2)	−0.1315 (2)	0.25382 (12)	0.0454 (5)
C24	0.3103 (3)	−0.1520 (2)	0.09974 (16)	0.0446 (6)

C8	0.6583 (2)	−0.0925 (2)	0.23014 (14)	0.0415 (5)
C3	0.7426 (3)	−0.0128 (2)	0.30485 (14)	0.0393 (5)
C16	0.7158 (3)	−0.0745 (3)	0.48023 (15)	0.0519 (6)
H16A	0.6343	−0.0772	0.5225	0.062*
H16B	0.7383	−0.1698	0.4640	0.062*
C10	0.4948 (3)	0.2266 (2)	0.32102 (16)	0.0464 (6)
C15	0.3277 (3)	0.2368 (3)	0.31634 (18)	0.0558 (7)
H15	0.2784	0.1947	0.3640	0.067*
C4	0.8954 (3)	0.0370 (3)	0.29641 (17)	0.0503 (6)
H4	0.9531	0.0917	0.3452	0.060*
C2	0.6413 (3)	0.0012 (2)	0.38491 (15)	0.0437 (6)
C1	0.4884 (3)	−0.0807 (3)	0.34282 (16)	0.0468 (6)
C25	0.3007 (3)	−0.0100 (3)	0.08741 (17)	0.0524 (6)
H25	0.3496	0.0480	0.1371	0.063*
C17	0.8693 (3)	−0.0135 (2)	0.53710 (15)	0.0488 (6)
C23	0.3890 (3)	−0.2184 (3)	0.19369 (17)	0.0574 (7)
H23A	0.3046	−0.2437	0.2308	0.069*
H23B	0.4424	−0.3033	0.1787	0.069*
C18	1.0215 (3)	−0.0589 (3)	0.52212 (17)	0.0612 (7)
H18	1.0286	−0.1267	0.4755	0.073*
C9	0.5968 (3)	0.1523 (3)	0.40544 (16)	0.0504 (6)
H9A	0.5382	0.1527	0.4601	0.060*
H9B	0.6965	0.2043	0.4247	0.060*
C22	0.8643 (3)	0.0867 (3)	0.60759 (16)	0.0566 (7)
H22	0.7641	0.1195	0.6188	0.068*
C6	0.8748 (3)	−0.0747 (3)	0.14105 (18)	0.0610 (7)
H6	0.9211	−0.0958	0.0865	0.073*
C29	0.2389 (3)	−0.2353 (3)	0.02380 (18)	0.0629 (7)
H29	0.2455	−0.3314	0.0302	0.075*
C26	0.2199 (3)	0.0472 (3)	0.0027 (2)	0.0668 (8)
H26	0.2133	0.1432	−0.0042	0.080*
C7	0.7211 (3)	−0.1231 (3)	0.14706 (16)	0.0547 (6)
H7	0.6619	−0.1745	0.0969	0.066*
C11	0.5633 (3)	0.2891 (3)	0.24806 (17)	0.0578 (7)
H11	0.6752	0.2844	0.2500	0.069*
C21	1.0043 (4)	0.1389 (3)	0.66143 (19)	0.0682 (8)
H21	0.9976	0.2061	0.7085	0.082*
C12	0.4683 (3)	0.3586 (3)	0.1721 (2)	0.0697 (8)
H12	0.5161	0.3982	0.1229	0.084*
C20	1.1531 (4)	0.0928 (3)	0.6464 (2)	0.0698 (8)
H20	1.2474	0.1271	0.6838	0.084*
C5	0.9610 (3)	0.0040 (3)	0.21408 (18)	0.0580 (7)
H5	1.0644	0.0355	0.2083	0.070*
C28	0.1581 (3)	−0.1783 (4)	−0.0613 (2)	0.0754 (9)
H28	0.1100	−0.2359	−0.1114	0.090*
C14	0.2335 (3)	0.3088 (3)	0.2416 (2)	0.0639 (7)
H14	0.1220	0.3166	0.2403	0.077*
C13	0.3036 (3)	0.3686 (3)	0.1697 (2)	0.0684 (8)

H13	0.2396	0.4160	0.1192	0.082*
C19	1.1621 (3)	−0.0052 (3)	0.5751 (2)	0.0710 (8)
H19	1.2629	−0.0350	0.5628	0.085*
C27	0.1485 (3)	−0.0374 (4)	−0.0721 (2)	0.0730 (9)
H27	0.0942	0.0012	−0.1296	0.088*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0624 (10)	0.0862 (14)	0.0635 (10)	−0.0224 (10)	0.0228 (9)	−0.0056 (11)
N1	0.0469 (11)	0.0475 (12)	0.0406 (10)	−0.0077 (9)	0.0037 (8)	−0.0046 (9)
C24	0.0411 (12)	0.0487 (15)	0.0428 (13)	−0.0069 (11)	0.0039 (10)	−0.0058 (11)
C8	0.0445 (12)	0.0402 (13)	0.0387 (11)	0.0010 (11)	0.0037 (10)	0.0007 (11)
C3	0.0425 (12)	0.0363 (12)	0.0375 (11)	0.0003 (10)	0.0016 (9)	0.0013 (10)
C16	0.0670 (15)	0.0468 (14)	0.0406 (12)	−0.0041 (13)	0.0048 (11)	0.0017 (12)
C10	0.0506 (14)	0.0403 (13)	0.0462 (13)	0.0024 (11)	0.0014 (11)	−0.0099 (11)
C15	0.0539 (15)	0.0581 (16)	0.0539 (14)	−0.0019 (13)	0.0041 (12)	−0.0131 (14)
C4	0.0485 (14)	0.0491 (15)	0.0516 (14)	−0.0032 (11)	0.0027 (11)	0.0018 (12)
C2	0.0498 (13)	0.0458 (14)	0.0346 (11)	−0.0052 (11)	0.0048 (10)	−0.0015 (11)
C1	0.0499 (13)	0.0468 (14)	0.0434 (12)	−0.0058 (12)	0.0067 (11)	0.0027 (12)
C25	0.0567 (15)	0.0540 (16)	0.0462 (14)	−0.0064 (12)	0.0073 (11)	−0.0014 (13)
C17	0.0633 (15)	0.0434 (14)	0.0367 (11)	0.0057 (12)	−0.0003 (11)	0.0033 (11)
C23	0.0633 (16)	0.0510 (16)	0.0540 (14)	−0.0171 (13)	−0.0019 (12)	−0.0020 (13)
C18	0.0718 (17)	0.0545 (16)	0.0521 (15)	0.0139 (14)	−0.0049 (13)	−0.0046 (13)
C9	0.0571 (14)	0.0504 (15)	0.0425 (13)	0.0005 (12)	0.0047 (11)	−0.0086 (12)
C22	0.0729 (17)	0.0547 (16)	0.0402 (13)	0.0072 (13)	0.0035 (12)	−0.0010 (13)
C6	0.0639 (16)	0.0716 (18)	0.0511 (13)	0.0114 (15)	0.0203 (13)	−0.0025 (14)
C29	0.0678 (17)	0.0570 (17)	0.0587 (16)	−0.0054 (14)	−0.0047 (13)	−0.0108 (14)
C26	0.0690 (17)	0.0654 (18)	0.0652 (18)	−0.0027 (15)	0.0081 (14)	0.0138 (15)
C7	0.0608 (15)	0.0583 (16)	0.0446 (12)	0.0040 (13)	0.0075 (11)	−0.0074 (13)
C11	0.0532 (15)	0.0559 (16)	0.0630 (16)	−0.0005 (13)	0.0061 (13)	0.0048 (14)
C21	0.095 (2)	0.0543 (16)	0.0498 (15)	−0.0009 (16)	−0.0057 (15)	−0.0088 (14)
C12	0.0756 (19)	0.0597 (17)	0.0717 (17)	0.0039 (15)	0.0063 (15)	0.0158 (15)
C20	0.0749 (19)	0.0567 (18)	0.0686 (18)	−0.0057 (15)	−0.0152 (15)	0.0032 (16)
C5	0.0512 (14)	0.0616 (17)	0.0641 (16)	0.0033 (13)	0.0176 (13)	0.0075 (14)
C28	0.077 (2)	0.089 (2)	0.0528 (17)	−0.0057 (18)	−0.0112 (14)	−0.0182 (17)
C14	0.0502 (15)	0.0652 (17)	0.0713 (17)	0.0067 (14)	−0.0049 (13)	−0.0105 (15)
C13	0.0723 (19)	0.0517 (16)	0.0732 (18)	0.0061 (15)	−0.0120 (15)	0.0052 (16)
C19	0.0628 (17)	0.0706 (19)	0.0736 (17)	0.0133 (15)	−0.0062 (14)	0.0036 (17)
C27	0.0704 (19)	0.097 (3)	0.0476 (15)	0.0017 (17)	−0.0012 (13)	0.0128 (17)

Geometric parameters (Å, º)

O1—C1	1.218 (3)	C18—C19	1.380 (4)
N1—C1	1.366 (3)	C18—H18	0.9300
N1—C8	1.407 (3)	C9—H9A	0.9700
N1—C23	1.449 (3)	C9—H9B	0.9700
C24—C25	1.379 (3)	C22—C21	1.375 (3)

C24—C29	1.383 (3)	C22—H22	0.9300
C24—C23	1.507 (3)	C6—C5	1.374 (4)
C8—C7	1.379 (3)	C6—C7	1.380 (3)
C8—C3	1.388 (3)	C6—H6	0.9300
C3—C4	1.384 (3)	C29—C28	1.379 (4)
C3—C2	1.512 (3)	C29—H29	0.9300
C16—C17	1.510 (3)	C26—C27	1.379 (4)
C16—C2	1.553 (3)	C26—H26	0.9300
C16—H16A	0.9700	C7—H7	0.9300
C16—H16B	0.9700	C11—C12	1.386 (3)
C10—C11	1.383 (3)	C11—H11	0.9300
C10—C15	1.388 (3)	C21—C20	1.366 (4)
C10—C9	1.514 (3)	C21—H21	0.9300
C15—C14	1.384 (3)	C12—C13	1.372 (4)
C15—H15	0.9300	C12—H12	0.9300
C4—C5	1.387 (3)	C20—C19	1.381 (4)
C4—H4	0.9300	C20—H20	0.9300
C2—C1	1.532 (3)	C5—H5	0.9300
C2—C9	1.539 (3)	C28—C27	1.365 (4)
C25—C26	1.375 (3)	C28—H28	0.9300
C25—H25	0.9300	C14—C13	1.367 (4)
C17—C22	1.383 (3)	C14—H14	0.9300
C17—C18	1.389 (3)	C13—H13	0.9300
C23—H23A	0.9700	C19—H19	0.9300
C23—H23B	0.9700	C27—H27	0.9300

C1—N1—C8	110.93 (17)	C10—C9—C2	115.07 (18)
C1—N1—C23	124.39 (19)	C10—C9—H9A	108.5
C8—N1—C23	124.65 (19)	C2—C9—H9A	108.5
C25—C24—C29	118.1 (2)	C10—C9—H9B	108.5
C25—C24—C23	122.5 (2)	C2—C9—H9B	108.5
C29—C24—C23	119.4 (2)	H9A—C9—H9B	107.5
C7—C8—C3	122.2 (2)	C21—C22—C17	121.4 (3)
C7—C8—N1	128.2 (2)	C21—C22—H22	119.3
C3—C8—N1	109.58 (17)	C17—C22—H22	119.3
C4—C3—C8	119.18 (19)	C5—C6—C7	121.3 (2)
C4—C3—C2	131.7 (2)	C5—C6—H6	119.3
C8—C3—C2	109.10 (17)	C7—C6—H6	119.3
C17—C16—C2	116.90 (19)	C28—C29—C24	121.1 (3)
C17—C16—H16A	108.1	C28—C29—H29	119.5
C2—C16—H16A	108.1	C24—C29—H29	119.5
C17—C16—H16B	108.1	C25—C26—C27	120.2 (3)
C2—C16—H16B	108.1	C25—C26—H26	119.9
H16A—C16—H16B	107.3	C27—C26—H26	119.9
C11—C10—C15	117.9 (2)	C8—C7—C6	117.6 (2)
C11—C10—C9	122.0 (2)	C8—C7—H7	121.2
C15—C10—C9	120.1 (2)	C6—C7—H7	121.2
C14—C15—C10	120.8 (3)	C10—C11—C12	121.2 (2)

C14—C15—H15	119.6	C10—C11—H11	119.4
C10—C15—H15	119.6	C12—C11—H11	119.4
C3—C4—C5	119.0 (2)	C20—C21—C22	120.6 (3)
C3—C4—H4	120.5	C20—C21—H21	119.7
C5—C4—H4	120.5	C22—C21—H21	119.7
C3—C2—C1	101.66 (17)	C13—C12—C11	119.8 (3)
C3—C2—C9	113.73 (18)	C13—C12—H12	120.1
C1—C2—C9	110.30 (19)	C11—C12—H12	120.1
C3—C2—C16	113.32 (18)	C21—C20—C19	119.4 (3)
C1—C2—C16	106.07 (17)	C21—C20—H20	120.3
C9—C2—C16	111.05 (18)	C19—C20—H20	120.3
O1—C1—N1	124.8 (2)	C6—C5—C4	120.7 (2)
O1—C1—C2	126.4 (2)	C6—C5—H5	119.7
N1—C1—C2	108.73 (19)	C4—C5—H5	119.7
C26—C25—C24	120.9 (2)	C27—C28—C29	120.2 (3)
C26—C25—H25	119.5	C27—C28—H28	119.9
C24—C25—H25	119.5	C29—C28—H28	119.9
C22—C17—C18	117.4 (2)	C13—C14—C15	120.3 (2)
C22—C17—C16	121.5 (2)	C13—C14—H14	119.8
C18—C17—C16	121.0 (2)	C15—C14—H14	119.8
N1—C23—C24	114.57 (19)	C14—C13—C12	119.9 (3)
N1—C23—H23A	108.6	C14—C13—H13	120.0
C24—C23—H23A	108.6	C12—C13—H13	120.0
N1—C23—H23B	108.6	C20—C19—C18	119.9 (3)
C24—C23—H23B	108.6	C20—C19—H19	120.0
H23A—C23—H23B	107.6	C18—C19—H19	120.0
C19—C18—C17	121.3 (3)	C28—C27—C26	119.5 (3)
C19—C18—H18	119.4	C28—C27—H27	120.2
C17—C18—H18	119.4	C26—C27—H27	120.2