Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,3-Dibenzyl-6-bromo-1*H*-imidazo-[4,5-*b*]pyridin-2(3*H*)-one

S. Dahmani,[a] Y. Kandri Rodi,[a] F. Capet,[b] El Mokhtar Essassi[c] and Seik Weng Ng[d]*

[a]Laboratoire de Chimie Organique Appliquée, Faculté des Sciences et Techniques, Université Sidi Mohamed Ben Abdallah, Fés, Morocco, [b]Unité de Catalyse et de Chimie du Solide, Ecole Nationale Supérieure de Chimie de Lille, Lille, France, [c]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

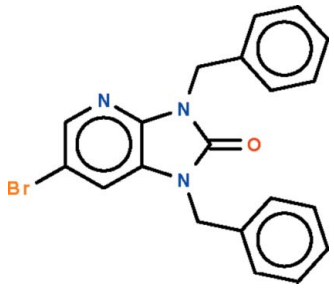
Received 24 February 2010; accepted 1 March 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.041; wR factor = 0.138; data-to-parameter ratio = 17.3.

The imidazopyridine fused-ring in the title compound, $C_{20}H_{16}BrN_3O$, is planar (r.m.s. deviation = 0.011 Å). The phenyl rings of the benzyl substitutents twist away from the central five-membered ring in opposite directions; the rings are aligned at 61.3 (1) and 71.2 (1)° with respect to this ring.

Related literature

For the medicinal applications of 1,3-dihydro-imidazo[4,5-*b*]-pyridin-2-ones, see: Barraclough *et al.* (1990); Cundy *et al.* (1997); Desarro *et al.* (1994); Liu *et al.* (2008); Mader *et al.* (2008); Zaki & Proença (2007). For the product of the reaction of propargyl bromide with 6-bromo-1,3-dihydro-imidazo-[4,5-*b*]pyridin-2-one in DMF at room and high temperatures, see: Dahmani *et al.* (2010*a*,*b*).



Experimental

Crystal data

$C_{20}H_{16}BrN_3O$ M_r = 394.27
Monoclinic, $P2_1/c$
a = 9.1627 (1) Å
b = 25.5071 (3) Å
c = 8.0629 (1) Å
β = 115.571 (1)°
V = 1699.84 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.43 mm^{-1}
T = 293 K
0.42 × 0.18 × 0.13 mm

Data collection

Bruker X8 APEX2 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.428, T_{max} = 0.743
38010 measured reflections
3903 independent reflections
2967 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.138
S = 1.07
3903 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Sidi Mohammed Ben Abdallah, Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2651).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Barraclough, P., Black, J. W., Cambridge, D., Collard, D., Firmin, D., Gerskowitch, V. P., Glen, R. C., Giles, H. & Hill, A. P. (1990). *J. Med. Chem.* **33**, 2231–2239.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cundy, D. J., Holan, G., Otaegui, M. & Simpson, G. W. (1997). *Bioorg. Med. Chem. Lett.* **7**, 669–674.
Dahmani, A. S., Haoudi, A., Capet, F., Essassi, E. M. & Ng, S. W. (2010*a*). *Acta Cryst.* E**66**, o755.
Dahmani, A. S., Haoudi, A., Capet, F., Essassi, E. M. & Ng, S. W. (2010*b*). *Acta Cryst.* E**66**, o756.
Desarro, A., Desarro, G., Chimirri, A., Grasso, S., Monforte, A. M. & Zappala, M. (1994). *Gen. Pharmacol.* **25**,1027–1031.
Liu, L., Xu, P., Zhou, L. & Lei, P. S. (2008). *Chin. Chem. Lett.* **19**, 1–4.
Mader, M., de Dios, A., Shih, C., Bonjouklian, R., Li, T. C., White, W., de Uralde, B. L., Sánchez-Martinez, C., del Prado, M., Jaramillo, C., de Diego, E., Cabrejas, L. M. M., Dominguez, C., Montero, C., Shepherd, T., Dally, R., Toth, J. E., Chatterjee, A., Pleite, S., Blanco-Urgoiti, J., Perez, L., Barberis, M., Lorite, M. J., Jambrina, E., Nevill, C. R., Lee, P. A., Schultz, R. C., Wolos, J. A., Li, L. C., Campbell, R. M. & Anderson, B. D. (2008). *Bioorg. Med. Chem. Lett.* **18**, 179–183.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *publCIF*. In preparation.
Zaki, M. E. A. & Proença, M. F. (2007). *Tetrahedron*, **63**, 3745–3753.

supporting information

Acta Cryst. (2010). E**66**, o754 [doi:10.1107/S1600536810007713]

1,3-Dibenzyl-6-bromo-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

S. Dahmani, Y. Kandri Rodi, F. Capet, El Mokhtar Essassi and Seik Weng Ng

S1. Comment

Imidazo[4,5-*b*]pyridines are precursors for the synthesis of a variety of medicinal agents as compounds having the imidazo[4,5-*b*]pyridine fused-ring system possess a broad range of pharmacological activities (Barraclough *et al.*, 1990; Cundy *et al.*, 1997; Desarro *et al.*, 1994; Liu *et al.*, 2008; Mader *et al.*, 2008; Zaki & Proença, 2007).

The present study represents the synthesis of the substituted an imidazo[4,5-b]pyridin-2-one derivative by the direct action of benzyl chloride on 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one in boiling DMF.

The imidazopyridine fused-ring in $C_{20}H_{16}BrN_3O$ (Scheme I, Fig. 1) is planar (r.m.s. deviation 0.011 Å). The phenyl rings of the benzyl substitutents twist away from the central five-membered ring in opposite directions; the rings are aligned at 61.3 (1) and 71.2 (1) ° with respect to this ring.

The temperature of the reaction, in the case of propargyl bromide, governs the nature of the product (Dahmani *et al.*, 2010*a*, 2010*b*).

S2. Experimental

To a mixture of 6-bromo-1,3-dihydro-imidazo[4,5-*b*]pyridin-2-one (1 mmol), potassium carbonate (4 mmol) and benzyl-tributylammonium chloride (0.1 mmol) in DMF was added benzyl chloride (2.5 mmol). The mixture was stirred for 48 hours. After completion of reaction (monitored by TLC), the inorganic salt was filtered and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel with ethyl acetate/hexane (1/1) as eluent. Colorless crystals were isolated when the solvent was allowed to evaporate.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93-0.97 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C).

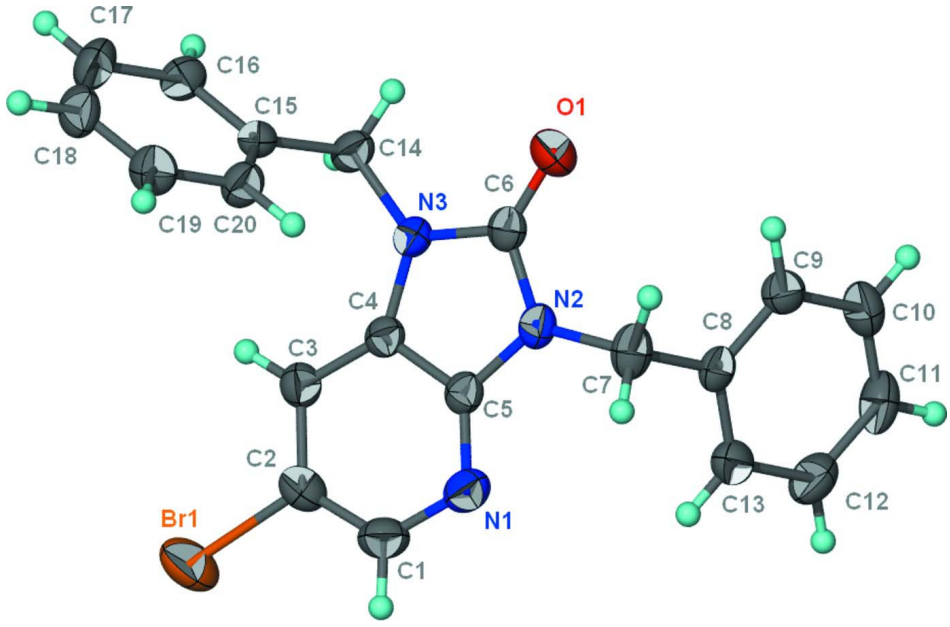


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{20}H_{16}BrN_3O$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius.

1,3-Dibenzyl-6-bromo-1*H*-imidazo[4,5-*b*]pyridin-2(3*H*)-one

Crystal data

$C_{20}H_{16}BrN_3O$
$M_r = 394.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.1627$ (1) Å
$b = 25.5071$ (3) Å
$c = 8.0629$ (1) Å
$\beta = 115.571$ (1)°
$V = 1699.84$ (3) Å^3
$Z = 4$

$F(000) = 800$
$D_x = 1.541$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9890 reflections
$\theta = 2.5$–25.0°
$\mu = 2.43$ mm^{-1}
$T = 293$ K
Prism, colorless
0.42 × 0.18 × 0.13 mm

Data collection

Bruker X8 APEX2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.428$, $T_{max} = 0.743$

38010 measured reflections
3903 independent reflections
2967 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -11 \rightarrow 11$
$k = -33 \rightarrow 33$
$l = -9 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.138$

$S = 1.07$
3903 reflections
226 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
$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 1.1691P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.84$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.75465 (4)	0.225888 (14)	0.48173 (6)	0.07328 (18)
O1	0.2072 (3)	0.45942 (8)	0.3332 (3)	0.0570 (5)
N1	0.3210 (3)	0.28134 (9)	0.4493 (3)	0.0480 (6)
N2	0.2208 (3)	0.36984 (9)	0.3920 (3)	0.0399 (5)
N3	0.4318 (3)	0.40833 (8)	0.3767 (3)	0.0398 (5)
C1	0.4498 (4)	0.25184 (12)	0.4685 (4)	0.0515 (7)
H1	0.4465	0.2160	0.4879	0.062*
C2	0.5858 (4)	0.27231 (11)	0.4606 (4)	0.0473 (7)
C3	0.6008 (3)	0.32553 (11)	0.4321 (4)	0.0428 (6)
H3	0.6926	0.3396	0.4275	0.051*
C4	0.4695 (3)	0.35553 (10)	0.4115 (3)	0.0366 (5)
C5	0.3351 (3)	0.33129 (10)	0.4207 (3)	0.0366 (5)
C6	0.2777 (3)	0.41774 (10)	0.3635 (4)	0.0401 (6)
C7	0.0593 (3)	0.36191 (13)	0.3822 (4)	0.0482 (7)
H7A	0.0226	0.3269	0.3356	0.058*
H7B	−0.0145	0.3867	0.2952	0.058*
C8	0.0513 (3)	0.36830 (10)	0.5637 (4)	0.0376 (5)
C9	0.0052 (3)	0.41597 (11)	0.6102 (4)	0.0448 (6)
H9	−0.0162	0.4444	0.5310	0.054*
C10	−0.0090 (4)	0.42129 (13)	0.7723 (4)	0.0530 (7)
H10	−0.0399	0.4533	0.8023	0.064*
C11	0.0223 (4)	0.37940 (15)	0.8905 (4)	0.0572 (8)
H11	0.0105	0.3829	0.9989	0.069*
C12	0.0711 (4)	0.33231 (14)	0.8480 (5)	0.0581 (8)
H12	0.0946	0.3042	0.9291	0.070*
C13	0.0854 (3)	0.32658 (12)	0.6849 (4)	0.0490 (7)
H13	0.1180	0.2946	0.6565	0.059*
C14	0.5321 (4)	0.44829 (11)	0.3487 (4)	0.0431 (6)
H14A	0.6349	0.4498	0.4570	0.052*
H14B	0.4797	0.4821	0.3362	0.052*
C15	0.5641 (3)	0.43916 (9)	0.1821 (3)	0.0349 (5)
C16	0.6900 (3)	0.46603 (11)	0.1695 (4)	0.0437 (6)
H16	0.7526	0.4889	0.2630	0.052*
C17	0.7240 (4)	0.45935 (14)	0.0203 (4)	0.0549 (8)
H17	0.8092	0.4777	0.0137	0.066*
C18	0.6319 (4)	0.42548 (13)	−0.1197 (4)	0.0533 (7)
H18	0.6559	0.4204	−0.2194	0.064*
C19	0.5046 (4)	0.39937 (12)	−0.1105 (4)	0.0498 (7)

H19	0.4411	0.3770	−0.2055	0.060*
C20	0.4702 (3)	0.40613 (11)	0.0401 (4)	0.0445 (6)
H20	0.3836	0.3884	0.0453	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0600 (2)	0.0554 (2)	0.0961 (3)	0.01808 (15)	0.0259 (2)	0.00089 (17)
O1	0.0630 (13)	0.0486 (12)	0.0681 (14)	0.0147 (10)	0.0364 (11)	0.0062 (10)
N1	0.0528 (14)	0.0437 (13)	0.0526 (14)	−0.0072 (10)	0.0274 (12)	0.0003 (10)
N2	0.0410 (11)	0.0455 (12)	0.0394 (12)	−0.0017 (9)	0.0233 (10)	−0.0020 (9)
N3	0.0454 (12)	0.0371 (11)	0.0455 (12)	−0.0015 (9)	0.0277 (10)	0.0009 (9)
C1	0.0595 (18)	0.0372 (15)	0.0584 (18)	−0.0029 (13)	0.0261 (15)	0.0018 (13)
C2	0.0459 (15)	0.0424 (15)	0.0494 (16)	0.0071 (12)	0.0166 (13)	−0.0007 (12)
C3	0.0398 (13)	0.0436 (14)	0.0478 (15)	0.0001 (11)	0.0214 (12)	−0.0026 (12)
C4	0.0424 (13)	0.0371 (12)	0.0336 (12)	−0.0029 (10)	0.0194 (11)	−0.0028 (10)
C5	0.0392 (13)	0.0400 (13)	0.0336 (12)	−0.0026 (10)	0.0186 (10)	−0.0031 (10)
C6	0.0482 (15)	0.0438 (14)	0.0346 (13)	0.0014 (12)	0.0239 (11)	−0.0020 (11)
C7	0.0359 (13)	0.0663 (19)	0.0426 (15)	−0.0056 (12)	0.0172 (12)	−0.0081 (13)
C8	0.0291 (11)	0.0463 (14)	0.0404 (13)	−0.0053 (10)	0.0177 (10)	−0.0025 (11)
C9	0.0402 (14)	0.0445 (15)	0.0506 (16)	0.0020 (11)	0.0204 (12)	0.0050 (12)
C10	0.0482 (16)	0.0594 (18)	0.0570 (18)	0.0009 (14)	0.0279 (14)	−0.0135 (15)
C11	0.0468 (16)	0.090 (2)	0.0415 (16)	−0.0001 (16)	0.0251 (13)	−0.0038 (16)
C12	0.0531 (18)	0.069 (2)	0.0566 (19)	0.0009 (15)	0.0276 (15)	0.0177 (16)
C13	0.0460 (15)	0.0441 (15)	0.0637 (18)	0.0007 (12)	0.0302 (14)	0.0014 (13)
C14	0.0544 (16)	0.0379 (13)	0.0447 (15)	−0.0103 (12)	0.0287 (13)	−0.0057 (11)
C15	0.0382 (12)	0.0309 (12)	0.0383 (13)	0.0023 (10)	0.0190 (11)	0.0031 (10)
C16	0.0412 (14)	0.0442 (15)	0.0437 (15)	−0.0059 (11)	0.0166 (12)	0.0001 (11)
C17	0.0486 (17)	0.066 (2)	0.0605 (19)	−0.0051 (14)	0.0338 (16)	0.0068 (15)
C18	0.0600 (18)	0.0642 (19)	0.0467 (16)	0.0081 (15)	0.0333 (15)	0.0053 (14)
C19	0.0571 (17)	0.0519 (16)	0.0401 (15)	−0.0015 (13)	0.0206 (13)	−0.0074 (12)
C20	0.0460 (15)	0.0461 (15)	0.0463 (15)	−0.0085 (12)	0.0245 (13)	−0.0048 (12)

Geometric parameters (Å, º)

Br1—C2	1.897 (3)	C9—H9	0.9300
O1—C6	1.213 (3)	C10—C11	1.377 (5)
N1—C5	1.311 (3)	C10—H10	0.9300
N1—C1	1.351 (4)	C11—C12	1.376 (5)
N2—C5	1.382 (3)	C11—H11	0.9300
N2—C6	1.386 (3)	C12—C13	1.384 (5)
N2—C7	1.462 (3)	C12—H12	0.9300
N3—C4	1.389 (3)	C13—H13	0.9300
N3—C6	1.391 (3)	C14—C15	1.512 (4)
N3—C14	1.453 (3)	C14—H14A	0.9700
C1—C2	1.377 (4)	C14—H14B	0.9700
C1—H1	0.9300	C15—C20	1.382 (4)
C2—C3	1.393 (4)	C15—C16	1.383 (4)

C3—C4	1.374 (4)	C16—C17	1.377 (4)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.408 (4)	C17—C18	1.382 (5)
C7—C8	1.505 (4)	C17—H17	0.9300
C7—H7A	0.9700	C18—C19	1.372 (5)
C7—H7B	0.9700	C18—H18	0.9300
C8—C13	1.386 (4)	C19—C20	1.391 (4)
C8—C9	1.390 (4)	C19—H19	0.9300
C9—C10	1.376 (4)	C20—H20	0.9300

C5—N1—C1	114.4 (2)	C9—C10—C11	120.3 (3)
C5—N2—C6	110.0 (2)	C9—C10—H10	119.9
C5—N2—C7	126.0 (2)	C11—C10—H10	119.9
C6—N2—C7	123.9 (2)	C12—C11—C10	119.9 (3)
C4—N3—C6	109.8 (2)	C12—C11—H11	120.1
C4—N3—C14	126.4 (2)	C10—C11—H11	120.1
C6—N3—C14	123.8 (2)	C11—C12—C13	120.2 (3)
N1—C1—C2	123.1 (3)	C11—C12—H12	119.9
N1—C1—H1	118.4	C13—C12—H12	119.9
C2—C1—H1	118.4	C12—C13—C8	120.2 (3)
C1—C2—C3	122.2 (3)	C12—C13—H13	119.9
C1—C2—Br1	118.6 (2)	C8—C13—H13	119.9
C3—C2—Br1	119.2 (2)	N3—C14—C15	114.1 (2)
C4—C3—C2	114.8 (3)	N3—C14—H14A	108.7
C4—C3—H3	122.6	C15—C14—H14A	108.7
C2—C3—H3	122.6	N3—C14—H14B	108.7
C3—C4—N3	133.9 (2)	C15—C14—H14B	108.7
C3—C4—C5	119.3 (2)	H14A—C14—H14B	107.6
N3—C4—C5	106.9 (2)	C20—C15—C16	118.8 (2)
N1—C5—N2	126.6 (2)	C20—C15—C14	122.7 (2)
N1—C5—C4	126.2 (3)	C16—C15—C14	118.5 (2)
N2—C5—C4	107.2 (2)	C17—C16—C15	120.9 (3)
O1—C6—N2	126.9 (3)	C17—C16—H16	119.6
O1—C6—N3	127.0 (3)	C15—C16—H16	119.6
N2—C6—N3	106.1 (2)	C16—C17—C18	120.1 (3)
N2—C7—C8	113.9 (2)	C16—C17—H17	120.0
N2—C7—H7A	108.8	C18—C17—H17	120.0
C8—C7—H7A	108.8	C19—C18—C17	119.6 (3)
N2—C7—H7B	108.8	C19—C18—H18	120.2
C8—C7—H7B	108.8	C17—C18—H18	120.2
H7A—C7—H7B	107.7	C18—C19—C20	120.3 (3)
C13—C8—C9	118.9 (2)	C18—C19—H19	119.8
C13—C8—C7	120.7 (3)	C20—C19—H19	119.8
C9—C8—C7	120.3 (3)	C15—C20—C19	120.3 (3)
C10—C9—C8	120.4 (3)	C15—C20—H20	119.9
C10—C9—H9	119.8	C19—C20—H20	119.9
C8—C9—H9	119.8		

C5—N1—C1—C2	0.6 (4)	C4—N3—C6—N2	0.4 (3)
N1—C1—C2—C3	0.0 (5)	C14—N3—C6—N2	177.7 (2)
N1—C1—C2—Br1	−178.0 (2)	C5—N2—C7—C8	92.2 (3)
C1—C2—C3—C4	−0.4 (4)	C6—N2—C7—C8	−91.5 (3)
Br1—C2—C3—C4	177.6 (2)	N2—C7—C8—C13	−86.0 (3)
C2—C3—C4—N3	−178.0 (3)	N2—C7—C8—C9	95.7 (3)
C2—C3—C4—C5	0.1 (4)	C13—C8—C9—C10	−1.1 (4)
C6—N3—C4—C3	178.2 (3)	C7—C8—C9—C10	177.2 (3)
C14—N3—C4—C3	0.9 (5)	C8—C9—C10—C11	0.0 (4)
C6—N3—C4—C5	−0.1 (3)	C9—C10—C11—C12	1.3 (5)
C14—N3—C4—C5	−177.4 (2)	C10—C11—C12—C13	−1.4 (5)
C1—N1—C5—N2	178.3 (3)	C11—C12—C13—C8	0.3 (5)
C1—N1—C5—C4	−1.0 (4)	C9—C8—C13—C12	1.0 (4)
C6—N2—C5—N1	−178.9 (3)	C7—C8—C13—C12	−177.3 (3)
C7—N2—C5—N1	−2.1 (4)	C4—N3—C14—C15	62.6 (4)
C6—N2—C5—C4	0.5 (3)	C6—N3—C14—C15	−114.3 (3)
C7—N2—C5—C4	177.3 (2)	N3—C14—C15—C20	18.6 (4)
C3—C4—C5—N1	0.6 (4)	N3—C14—C15—C16	−163.1 (2)
N3—C4—C5—N1	179.2 (3)	C20—C15—C16—C17	−1.3 (4)
C3—C4—C5—N2	−178.8 (2)	C14—C15—C16—C17	−179.7 (3)
N3—C4—C5—N2	−0.2 (3)	C15—C16—C17—C18	0.1 (5)
C5—N2—C6—O1	179.4 (3)	C16—C17—C18—C19	1.2 (5)
C7—N2—C6—O1	2.6 (4)	C17—C18—C19—C20	−1.2 (5)
C5—N2—C6—N3	−0.5 (3)	C16—C15—C20—C19	1.4 (4)
C7—N2—C6—N3	−177.4 (2)	C14—C15—C20—C19	179.7 (3)
C4—N3—C6—O1	−179.6 (3)	C18—C19—C20—C15	−0.1 (5)
C14—N3—C6—O1	−2.2 (4)		