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2-Butyl-4-chloro-1-(4-nitrobenzyl)-1*H*-imidazole-5-carboxaldehyde

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.057
wR factor = 0.139
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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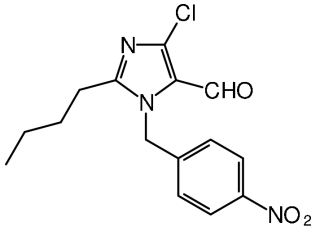
The asymmetric unit of the title compound, $C_{15}H_{16}ClN_3O_3$, comprises two molecules that are each twisted about the benzyl C atom. The second and fourth C atoms of the butyl chain of one molecule are disordered (0.75:0.25 and 0.5:0.5, respectively). The dihedral angles between the imidazole and benzene rings are 76.46 (9) and 76.3 (1)°.

Comment

Imidazole derivatives are reported to be biologically active molecules, and both imidazoles and benzimidazoles are components of larger molecules used in pharmaceuticals, agrochemicals, dyestuffs and high-temperature polymer products (Rasmussen, 1999; Ambalvanan *et al.*, 2003). With this in mind, the title compound, (I), was prepared in a series of syntheses to produce new imidazole derivatives. The Cambridge Structural Database (Version of April 2004; Allen, 2002) reveals that there are currently 42 known structures containing a 3-benzylimidazole moiety, but not yet the title compound.



(I)

The asymmetric unit of (I) comprises two molecules, *A* and *B*, that are each twisted about the benzyl C atom (Fig. 1). The butyl chain of molecule *B* is disordered, with the second and fourth C atoms in the chain occupying two sites each. The second C atom is unequally disordered, with occupancies of 0.75:0.25 for C22*B* and C22*C*, respectively, whereas the fourth C atom is equally disordered across two sites (C24*B* and C24*C*). In early refinements, the third C atom (C23*B*) was split (similar to C22*B*/*C*), but this proved not to be a viable option, with the lesser refining unsatisfactorily. Stable refinement was achieved with C23*B* being treated as a whole atom, even though it displays larger displacement ellipsoids compared with neighbouring atoms. The dihedral angles between the imidazole and benzene rings are 76.46 (9) and 76.3 (1)° for molecules *A* and *B*, respectively.

Experimental

The title compound was prepared by stirring an equimolar mixture of 2-butyl-5-chloro-3*H*-imidazole-4-carboxaldehyde, 4-nitrobenzyl-

bromide and K_2CO_3 in dimethylformamide at room temperature for 6 h. The product was filtered and recrystallized from ethanol to yield colourless plates.

Crystal data

$C_{15}H_{16}ClN_3O_3$
M_r = 321.76
Triclinic, $P\overline{1}$
a = 8.3007 (5) Å
b = 12.2295 (6) Å
c = 16.5605 (10) Å
α = 103.420 (4)°
β = 95.561 (3)°
γ = 106.758 (3)°
V = 1541.35 (15) Å^3
Z = 4
D_x = 1.387 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6811 reflections
θ = 2.9–27.5°
μ = 0.26 mm^{-1}
T = 120 (2) K
Plate, colourless
0.36 × 0.30 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997*b*)
T_{min} = 0.911, T_{max} = 0.990
30 632 measured reflections
6038 independent reflections
3622 reflections with $I > 2\sigma(I)$
R_{int} = 0.098
θ_{max} = 26.0°
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.139
S = 1.04
6038 reflections
418 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

All H atoms were included in the refinement at calculated positions in the riding-model approximation, with C—H distances of 0.95 (aromatic H atoms and CHO H atoms), 0.98 (CH_3 H atoms) and 0.99 Å (CH_2 H atoms). The isotropic displacement parameters were set equal to 1.25U_{eq} of the carrier atom. A high R_{int} was the result of weak high-angle data.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *PLATON*97 (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

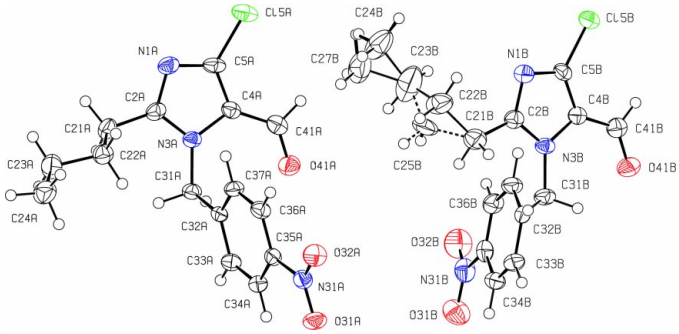


Figure 1
The molecular configurations and atom-numbering schemes for the two independent molecules, *A* and *B*, of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Ambalvanan, P., Palani, K., Ponnuswamy, M. N., Thirumuruhan, R. A., Yathirajan, H. S., Prabhuswamy, B., Raju, C. R., Nagaraja, P. & Mohana, K. N. (2003). *Mol. Cryst. Liq. Cryst.* **393**, 75–82.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rasmussen, P. G. (1999). *J. Chem. Educ.* **76**, 1345.
Sheldrick, G. M. (1997*a*). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.3007\ (5)$ Å
$b = 12.2295\ (6)$ Å
$c = 16.5605\ (10)$ Å
$\alpha = 103.420\ (4)°$
$\beta = 95.561\ (3)°$
$\gamma = 106.758\ (3)°$
$V = 1541.35\ (15)$ Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.387$ Mg m^{-3}
Melting point: 375 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6811 reflections
$\theta = 2.9–27.5°$
$\mu = 0.26$ mm^{-1}
$T = 120$ K
Plate, colourless
0.36 × 0.30 × 0.04 mm

Data collection

Bruker-Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1997b)
$T_{min} = 0.911$, $T_{max} = 0.990$
30632 measured reflections
6038 independent reflections
3622 reflections with $I > 2\sigma(I)$
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$\theta_{max} = 26.0°$, $\theta_{min} = 2.9°$
$h = -10\rightarrow10$
$k = -15\rightarrow15$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.139$
$S = 1.04$
6038 reflections
418 parameters
5 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
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 0.076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Special details

Geometry. Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)
- 6.8962 (0.0067) *x* + 4.9846 (0.0145) *y* - 7.8315 (0.0209) *z* = 1.6003 (0.0120)
* -0.0004 (0.0016) N1A * 0.0000 (0.0016) C2A * 0.0003 (0.0016) N3A * -0.0006 (0.0016) C4A * 0.0006 (0.0017) C5A
Rms deviation of fitted atoms = 0.0004
- 2.8599 (0.0085) *x* - 3.1842 (0.0131) *y* + 15.8848 (0.0052) *z* = 0.0246 (0.0064)
Angle to previous plane (with approximate e.s.d.) = 76.46 (0.09)
* -0.0124 (0.0019) C32A * 0.0090 (0.0019) C33A * 0.0041 (0.0019) C34A * -0.0137 (0.0019) C35A * 0.0100 (0.0019) C36A * 0.0031 (0.0019) C37A
Rms deviation of fitted atoms = 0.0096
7.1630 (0.0060) *x* - 4.4739 (0.0148) *y* + 6.9396 (0.0214) *z* = 6.8216 (0.0014)
Angle to previous plane (with approximate e.s.d.) = 80.19 (0.09)
* 0.0002 (0.0017) N1B * -0.0002 (0.0017) C2B * 0.0002 (0.0016) N3B * 0.0000 (0.0016) C4B * -0.0001 (0.0017) C5B
Rms deviation of fitted atoms = 0.0002
- 2.3192 (0.0090) *x* - 3.5959 (0.0138) *y* + 16.1123 (0.0044) *z* = 1.2353 (0.0111)
Angle to previous plane (with approximate e.s.d.) = 76.31 (0.10)
* 0.0029 (0.0019) C32B * -0.0043 (0.0020) C33B * 0.0014 (0.0020) C34B * 0.0030 (0.0020) C35B * -0.0043 (0.0020) C36B * 0.0014 (0.0020) C37B
Rms deviation of fitted atoms = 0.0031

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1A	0.1941 (3)	0.9197 (2)	0.21017 (15)	0.0320 (6)	
C2A	0.1120 (4)	0.8084 (2)	0.21159 (18)	0.0291 (7)	
C21A	0.0110 (4)	0.7792 (3)	0.27785 (19)	0.0341 (7)	
H21A	−0.1016	0.7203	0.2497	0.043*	
H22A	−0.0099	0.8520	0.3089	0.043*	
C22A	0.0936 (4)	0.7298 (3)	0.34200 (18)	0.0336 (7)	
H23A	0.1042	0.6525	0.3127	0.042*	
H24A	0.2098	0.7850	0.3678	0.042*	
C23A	−0.0126 (4)	0.7134 (3)	0.4106 (2)	0.0452 (9)	
H25A	−0.0219	0.7912	0.4397	0.056*	
H26A	−0.1294	0.6598	0.3840	0.056*	
C24A	0.0598 (5)	0.6628 (3)	0.4755 (2)	0.0473 (9)	
H27A	0.0520	0.5806	0.4490	0.059*	
H28A	−0.0055	0.6649	0.5217	0.059*	
H29A	0.1798	0.7103	0.4977	0.059*	
N3A	0.1292 (3)	0.72747 (19)	0.14485 (14)	0.0260 (5)	
C31A	0.0640 (4)	0.5979 (2)	0.12824 (19)	0.0281 (7)	
H31A	0.0088	0.5623	0.0683	0.035*	
H32A	−0.0239	0.5769	0.1634	0.035*	
C32A	0.2046 (3)	0.5464 (2)	0.14713 (17)	0.0244 (6)	
C33A	0.1713 (4)	0.4240 (2)	0.11796 (17)	0.0280 (7)	
H33A	0.0601	0.3748	0.0892	0.035*	
C34A	0.2970 (4)	0.3735 (2)	0.13014 (17)	0.0278 (7)	
H34A	0.2740	0.2903	0.1099	0.035*	
C35A	0.4576 (3)	0.4473 (2)	0.17274 (17)	0.0256 (6)	
C36A	0.4929 (4)	0.5679 (2)	0.20476 (18)	0.0286 (7)	

H36A	0.6029	0.6164	0.2356	0.036*	
C37A	0.3659 (3)	0.6167 (2)	0.19124 (17)	0.0273 (7)	
H37A	0.3892	0.6999	0.2125	0.034*	
N31A	0.5943 (3)	0.3952 (2)	0.18395 (15)	0.0306 (6)	
O31A	0.5619 (3)	0.28834 (18)	0.15276 (14)	0.0411 (6)	
O32A	0.7355 (3)	0.46121 (18)	0.22401 (13)	0.0389 (5)	
C4A	0.2293 (3)	0.7895 (2)	0.09634 (18)	0.0270 (7)	
C41A	0.2801 (4)	0.7385 (3)	0.01985 (19)	0.0311 (7)	
H41A	0.3513	0.7921	−0.0052	0.039*	
O41A	0.2410 (3)	0.63322 (18)	−0.01605 (13)	0.0373 (5)	
C5A	0.2643 (4)	0.9064 (2)	0.13975 (19)	0.0307 (7)	
Cl5A	0.38198 (10)	1.02856 (6)	0.11284 (5)	0.0410 (2)	
N1B	0.6628 (3)	−0.1150 (2)	0.22474 (15)	0.0313 (6)	
C2B	0.6981 (4)	0.0002 (2)	0.26254 (18)	0.0300 (7)	
C21B	0.6407 (5)	0.0477 (3)	0.3422 (2)	0.0454 (9)	
H21B	0.5462	0.0782	0.3284	0.057*	0.50
H22B	0.7364	0.1153	0.3790	0.057*	0.50
H21C	0.5147	0.0136	0.3336	0.057*	0.50
H22C	0.6689	0.1346	0.3522	0.057*	0.50
C22B	0.5810 (7)	−0.0426 (4)	0.3902 (3)	0.0548 (14)	0.75
H23B	0.5204	−0.0093	0.4332	0.068*	0.75
H24B	0.4953	−0.1133	0.3502	0.068*	0.75
C22C	0.7160 (19)	0.0238 (10)	0.4224 (7)	0.050 (4)	0.25
H23C	0.8403	0.0667	0.4331	0.062*	0.25
H24C	0.6695	0.0639	0.4692	0.062*	0.25
C23B	0.6998 (7)	−0.0824 (5)	0.4313 (3)	0.0957 (18)	
H25B	0.7781	−0.0152	0.4771	0.120*	0.50
H26B	0.7690	−0.1103	0.3907	0.120*	0.50
H25C	0.7749	−0.1123	0.3953	0.120*	0.50
H26C	0.5814	−0.1311	0.4036	0.120*	0.50
C24B	0.6138 (14)	−0.1835 (8)	0.4688 (7)	0.076 (3)	0.50
H27B	0.5378	−0.1587	0.5055	0.096*	0.50
H28B	0.7016	−0.2026	0.5016	0.096*	0.50
H29B	0.5473	−0.2538	0.4231	0.096*	0.50
C24C	0.7279 (15)	−0.1186 (9)	0.5099 (5)	0.077 (3)	0.50
H27C	0.8509	−0.0945	0.5311	0.096*	0.50
H28C	0.6787	−0.2050	0.4979	0.096*	0.50
H29C	0.6728	−0.0802	0.5525	0.096*	0.50
N3B	0.7853 (3)	0.07047 (19)	0.21787 (14)	0.0272 (6)	
C31B	0.8436 (4)	0.2000 (2)	0.24006 (19)	0.0315 (7)	
H31B	0.7715	0.2295	0.2783	0.039*	
H32B	0.8270	0.2255	0.1882	0.039*	
C32B	1.0288 (4)	0.2568 (2)	0.28223 (17)	0.0255 (6)	
C33B	1.0880 (4)	0.3795 (2)	0.31769 (18)	0.0315 (7)	
H33B	1.0113	0.4238	0.3160	0.039*	
C34B	1.2568 (4)	0.4371 (3)	0.35520 (18)	0.0325 (7)	
H34B	1.2970	0.5205	0.3798	0.041*	
C35B	1.3661 (4)	0.3708 (3)	0.35625 (17)	0.0310 (7)	

C36B	1.3121 (4)	0.2497 (3)	0.32099 (19)	0.0346 (7)
H36B	1.3899	0.2060	0.3219	0.043*
C37B	1.1418 (4)	0.1932 (3)	0.28422 (19)	0.0329 (7)
H37B	1.1022	0.1097	0.2601	0.041*
N31B	1.5466 (3)	0.4314 (3)	0.39535 (16)	0.0411 (7)
O31B	1.5914 (3)	0.5382 (2)	0.42867 (15)	0.0552 (7)
O32B	1.6441 (3)	0.3719 (2)	0.39210 (16)	0.0591 (7)
C4B	0.8079 (3)	−0.0049 (2)	0.14593 (17)	0.0266 (7)
C41B	0.8932 (4)	0.0287 (3)	0.07954 (19)	0.0342 (7)
H41B	0.8993	−0.0338	0.0349	0.043*
O41B	0.9582 (3)	0.12965 (18)	0.07510 (13)	0.0424 (6)
C5B	0.7303 (3)	−0.1171 (2)	0.15369 (17)	0.0274 (7)
Cl5B	0.71564 (10)	−0.24842 (6)	0.08330 (5)	0.0357 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0331 (15)	0.0262 (14)	0.0401 (16)	0.0145 (11)	0.0045 (12)	0.0103 (11)
C2A	0.0258 (17)	0.0268 (16)	0.0372 (18)	0.0137 (13)	0.0027 (13)	0.0081 (14)
C21A	0.0330 (18)	0.0349 (17)	0.0386 (18)	0.0177 (14)	0.0104 (15)	0.0079 (14)
C22A	0.0310 (18)	0.0322 (17)	0.0368 (18)	0.0106 (14)	0.0047 (14)	0.0076 (14)
C23A	0.046 (2)	0.051 (2)	0.046 (2)	0.0214 (17)	0.0163 (17)	0.0166 (17)
C24A	0.064 (2)	0.043 (2)	0.037 (2)	0.0204 (18)	0.0147 (17)	0.0091 (16)
N3A	0.0208 (13)	0.0216 (12)	0.0368 (14)	0.0084 (10)	0.0040 (11)	0.0089 (11)
C31A	0.0244 (16)	0.0208 (15)	0.0366 (17)	0.0043 (12)	0.0047 (13)	0.0067 (13)
C32A	0.0247 (16)	0.0205 (14)	0.0307 (16)	0.0071 (12)	0.0088 (13)	0.0107 (12)
C33A	0.0266 (17)	0.0240 (15)	0.0295 (16)	0.0044 (13)	0.0007 (13)	0.0060 (13)
C34A	0.0302 (17)	0.0236 (15)	0.0308 (17)	0.0094 (13)	0.0060 (13)	0.0084 (13)
C35A	0.0228 (16)	0.0262 (15)	0.0329 (16)	0.0106 (13)	0.0085 (13)	0.0128 (13)
C36A	0.0238 (16)	0.0258 (15)	0.0342 (17)	0.0048 (13)	0.0022 (13)	0.0096 (13)
C37A	0.0264 (17)	0.0185 (14)	0.0331 (17)	0.0052 (12)	0.0024 (13)	0.0036 (12)
N31A	0.0307 (15)	0.0309 (15)	0.0372 (15)	0.0138 (12)	0.0091 (12)	0.0161 (12)
O31A	0.0392 (13)	0.0263 (12)	0.0652 (16)	0.0170 (10)	0.0119 (11)	0.0170 (11)
O32A	0.0270 (13)	0.0405 (13)	0.0488 (14)	0.0119 (10)	−0.0011 (10)	0.0133 (11)
C4A	0.0207 (15)	0.0256 (15)	0.0360 (18)	0.0069 (12)	0.0036 (13)	0.0124 (13)
C41A	0.0225 (16)	0.0327 (18)	0.0392 (19)	0.0073 (13)	0.0012 (14)	0.0156 (15)
O41A	0.0344 (13)	0.0310 (12)	0.0399 (13)	0.0063 (10)	0.0046 (10)	0.0032 (10)
C5A	0.0289 (17)	0.0207 (15)	0.0433 (19)	0.0079 (13)	−0.0002 (14)	0.0130 (14)
Cl5A	0.0365 (5)	0.0269 (4)	0.0585 (5)	0.0048 (3)	0.0017 (4)	0.0192 (4)
N1B	0.0314 (15)	0.0275 (14)	0.0336 (15)	0.0085 (11)	0.0059 (12)	0.0069 (11)
C2B	0.0283 (17)	0.0290 (17)	0.0329 (17)	0.0103 (13)	0.0039 (14)	0.0083 (14)
C21B	0.063 (2)	0.0383 (19)	0.043 (2)	0.0260 (18)	0.0199 (18)	0.0098 (16)
C22B	0.079 (4)	0.040 (3)	0.060 (3)	0.028 (3)	0.041 (3)	0.019 (3)
C22C	0.065 (11)	0.023 (7)	0.046 (9)	−0.002 (7)	0.019 (8)	−0.003 (6)
C23B	0.156 (5)	0.123 (4)	0.071 (3)	0.104 (4)	0.058 (3)	0.055 (3)
C24B	0.117 (10)	0.084 (8)	0.079 (8)	0.073 (7)	0.059 (7)	0.052 (6)
C24C	0.125 (10)	0.092 (8)	0.049 (6)	0.068 (7)	0.036 (6)	0.033 (6)
N3B	0.0242 (13)	0.0220 (12)	0.0336 (14)	0.0086 (10)	−0.0008 (11)	0.0048 (11)

C31B	0.0316 (18)	0.0231 (15)	0.0405 (18)	0.0128 (13)	0.0006 (14)	0.0070 (13)
C32B	0.0293 (17)	0.0260 (15)	0.0226 (15)	0.0100 (13)	0.0032 (12)	0.0084 (12)
C33B	0.0328 (18)	0.0269 (16)	0.0384 (18)	0.0136 (14)	0.0083 (14)	0.0101 (14)
C34B	0.0360 (19)	0.0247 (15)	0.0344 (18)	0.0088 (14)	0.0073 (14)	0.0042 (13)
C35B	0.0261 (17)	0.0408 (18)	0.0256 (16)	0.0089 (14)	0.0037 (13)	0.0106 (14)
C36B	0.0330 (19)	0.0356 (18)	0.0393 (19)	0.0169 (15)	0.0024 (15)	0.0122 (14)
C37B	0.0351 (19)	0.0239 (15)	0.0381 (18)	0.0115 (14)	−0.0008 (14)	0.0057 (13)
N31B	0.0331 (17)	0.0472 (18)	0.0363 (16)	0.0033 (15)	0.0035 (13)	0.0119 (14)
O31B	0.0392 (14)	0.0497 (16)	0.0583 (16)	−0.0034 (12)	0.0022 (12)	0.0049 (13)
O32B	0.0347 (15)	0.0721 (18)	0.0692 (18)	0.0201 (14)	−0.0042 (12)	0.0184 (14)
C4B	0.0228 (16)	0.0262 (16)	0.0295 (17)	0.0079 (12)	0.0018 (13)	0.0065 (13)
C41B	0.0315 (18)	0.0335 (18)	0.0375 (19)	0.0104 (14)	0.0036 (14)	0.0103 (14)
O41B	0.0462 (14)	0.0311 (12)	0.0479 (14)	0.0051 (10)	0.0115 (11)	0.0148 (10)
C5B	0.0257 (16)	0.0254 (15)	0.0262 (16)	0.0074 (13)	−0.0013 (13)	0.0010 (12)
Cl5B	0.0403 (5)	0.0268 (4)	0.0343 (4)	0.0076 (3)	0.0046 (3)	0.0026 (3)

Geometric parameters (Å, º)

N1A—C2A	1.341 (3)	C2B—C21B	1.494 (4)
N1A—C5A	1.351 (4)	C21B—C22B	1.504 (5)
C2A—N3A	1.351 (4)	C21B—H21B	0.99
C2A—C21A	1.491 (4)	C21B—H22B	0.99
C21A—C22A	1.537 (4)	C22B—C23B	1.405 (6)
C21A—H21A	0.99	C22B—H23B	0.99
C21A—H22A	0.99	C22B—H24B	0.99
C22A—C23A	1.518 (4)	C23B—C24B	1.534 (9)
C22A—H23A	0.99	C23B—H25B	0.99
C22A—H24A	0.99	C23B—H26B	0.99
C23A—C24A	1.515 (4)	C24B—H27B	0.98
C23A—H25A	0.99	C24B—H28B	0.98
C23A—H26A	0.99	C24B—H29B	0.98
C24A—H27A	0.98	C22C—H23C	0.99
C24A—H28A	0.98	C22C—H24C	0.99
C24A—H29A	0.98	C24C—H27C	0.98
N3A—C4A	1.400 (3)	C24C—H28C	0.98
N3A—C31A	1.467 (3)	C24C—H29C	0.98
C31A—C32A	1.518 (4)	N3B—C4B	1.392 (3)
C31A—H31A	0.99	N3B—C31B	1.460 (3)
C31A—H32A	0.99	C31B—C32B	1.511 (4)
C32A—C37A	1.386 (4)	C31B—H31B	0.99
C32A—C33A	1.395 (4)	C31B—H32B	0.99
C33A—C34A	1.378 (4)	C32B—C37B	1.383 (4)
C33A—H33A	0.95	C32B—C33B	1.396 (4)
C34A—C35A	1.385 (4)	C33B—C34B	1.380 (4)
C34A—H34A	0.95	C33B—H33B	0.95
C35A—C36A	1.378 (4)	C34B—C35B	1.381 (4)
C35A—N31A	1.469 (3)	C34B—H34B	0.95
C36A—C37A	1.378 (4)	C35B—C36B	1.379 (4)

C36A—H36A	0.95	C35B—N31B	1.470 (4)
C37A—H37A	0.95	C36B—C37B	1.386 (4)
N31A—O31A	1.227 (3)	C36B—H36B	0.95
N31A—O32A	1.233 (3)	C37B—H37B	0.95
C4A—C5A	1.373 (4)	N31B—O31B	1.222 (3)
C4A—C41A	1.436 (4)	N31B—O32B	1.233 (3)
C41A—O41A	1.217 (3)	C4B—C5B	1.379 (4)
C41A—H41A	0.95	C4B—C41B	1.439 (4)
C5A—Cl5A	1.714 (3)	C41B—O41B	1.219 (3)
N1B—C2B	1.334 (3)	C41B—H41B	0.95
N1B—C5B	1.349 (4)	C5B—Cl5B	1.716 (3)
C2B—N3B	1.360 (3)		

C2A—N1A—C5A	104.4 (2)	C4A—C5A—Cl5A	126.9 (2)
N1A—C2A—N3A	111.6 (3)	C2B—N1B—C5B	104.4 (2)
N1A—C2A—C21A	123.6 (3)	N1B—C2B—N3B	112.2 (2)
N3A—C2A—C21A	124.7 (2)	N1B—C2B—C21B	124.4 (3)
C2A—C21A—C22A	115.5 (2)	N3B—C2B—C21B	123.3 (2)
C2A—C21A—H21A	108.4	C2B—N3B—C4B	106.8 (2)
C22A—C21A—H21A	108.4	C2B—N3B—C31B	127.0 (2)
C2A—C21A—H22A	108.4	C4B—N3B—C31B	126.1 (2)
C22A—C21A—H22A	108.4	N3B—C31B—C32B	114.0 (2)
H21A—C21A—H22A	107.5	N3B—C31B—H31B	108.8
C23A—C22A—C21A	110.9 (2)	C32B—C31B—H31B	108.8
C23A—C22A—H23A	109.5	N3B—C31B—H32B	108.8
C21A—C22A—H23A	109.5	C32B—C31B—H32B	108.8
C23A—C22A—H24A	109.5	H31B—C31B—H32B	107.7
C21A—C22A—H24A	109.5	C37B—C32B—C33B	119.2 (3)
H23A—C22A—H24A	108.0	C37B—C32B—C31B	122.9 (2)
C24A—C23A—C22A	113.9 (3)	C33B—C32B—C31B	117.8 (2)
C24A—C23A—H25A	108.8	C34B—C33B—C32B	120.7 (3)
C22A—C23A—H25A	108.8	C34B—C33B—H33B	119.7
C24A—C23A—H26A	108.8	C32B—C33B—H33B	119.7
C22A—C23A—H26A	108.8	C33B—C34B—C35B	118.6 (3)
H25A—C23A—H26A	107.7	C33B—C34B—H34B	120.7
C23A—C24A—H27A	109.5	C35B—C34B—H34B	120.7
C23A—C24A—H28A	109.5	C36B—C35B—C34B	122.2 (3)
H27A—C24A—H28A	109.5	C36B—C35B—N31B	118.8 (3)
C23A—C24A—H29A	109.5	C34B—C35B—N31B	119.0 (3)
H27A—C24A—H29A	109.5	C35B—C36B—C37B	118.4 (3)
H28A—C24A—H29A	109.5	C35B—C36B—H36B	120.8
C2A—N3A—C4A	107.6 (2)	C37B—C36B—H36B	120.8
C2A—N3A—C31A	126.6 (2)	C32B—C37B—C36B	120.9 (3)
C4A—N3A—C31A	125.7 (2)	C32B—C37B—H37B	119.6
N3A—C31A—C32A	112.1 (2)	C36B—C37B—H37B	119.6
N3A—C31A—H31A	109.2	O31B—N31B—O32B	123.8 (3)
C32A—C31A—H31A	109.2	O31B—N31B—C35B	118.0 (3)
N3A—C31A—H32A	109.2	O32B—N31B—C35B	118.2 (3)

C32A—C31A—H32A	109.2	C5B—C4B—N3B	104.0 (2)
H31A—C31A—H32A	107.9	C5B—C4B—C41B	128.8 (3)
C37A—C32A—C33A	118.8 (3)	N3B—C4B—C41B	127.2 (2)
C37A—C32A—C31A	122.3 (2)	O41B—C41B—C4B	126.3 (3)
C33A—C32A—C31A	118.9 (2)	O41B—C41B—H41B	116.8
C34A—C33A—C32A	121.1 (3)	C4B—C41B—H41B	116.8
C34A—C33A—H33A	119.4	N1B—C5B—C4B	112.6 (2)
C32A—C33A—H33A	119.4	N1B—C5B—Cl5B	121.4 (2)
C33A—C34A—C35A	118.2 (3)	C4B—C5B—Cl5B	126.0 (2)
C33A—C34A—H34A	120.9	C2B—C21B—C22B	113.8 (3)
C35A—C34A—H34A	120.9	C2B—C21B—H21B	108.8
C36A—C35A—C34A	122.0 (3)	C22B—C21B—H21B	108.8
C36A—C35A—N31A	119.1 (2)	C2B—C21B—H22B	108.8
C34A—C35A—N31A	118.9 (2)	C22B—C21B—H22B	108.8
C37A—C36A—C35A	118.8 (3)	H21B—C21B—H22B	107.7
C37A—C36A—H36A	120.6	C23B—C22B—C21B	120.0 (4)
C35A—C36A—H36A	120.6	C23B—C22B—H23B	107.3
C36A—C37A—C32A	121.0 (3)	C21B—C22B—H23B	107.3
C36A—C37A—H37A	119.5	C23B—C22B—H24B	107.3
C32A—C37A—H37A	119.5	C21B—C22B—H24B	107.3
O31A—N31A—O32A	123.5 (2)	H23B—C22B—H24B	106.9
O31A—N31A—C35A	118.2 (2)	C22B—C23B—C24B	112.5 (6)
O32A—N31A—C35A	118.3 (2)	C22B—C23B—H25B	109.1
C5A—C4A—N3A	103.4 (2)	C24B—C23B—H25B	109.1
C5A—C4A—C41A	130.2 (3)	C22B—C23B—H26B	109.1
N3A—C4A—C41A	126.4 (2)	C24B—C23B—H26B	109.1
O41A—C41A—C4A	126.4 (3)	H25B—C23B—H26B	107.8
O41A—C41A—H41A	116.8	H23C—C22C—H24C	106.4
C4A—C41A—H41A	116.8	H27C—C24C—H28C	109.5
N1A—C5A—C4A	112.9 (2)	H27C—C24C—H29C	109.5
N1A—C5A—Cl5A	120.2 (2)	H28C—C24C—H29C	109.5

C5A—N1A—C2A—N3A	0.0 (3)	C5B—N1B—C2B—N3B	0.0 (3)
C5A—N1A—C2A—C21A	−178.5 (3)	C5B—N1B—C2B—C21B	176.7 (3)
N1A—C2A—C21A—C22A	−106.8 (3)	N1B—C2B—N3B—C4B	0.0 (3)
N3A—C2A—C21A—C22A	74.9 (4)	C21B—C2B—N3B—C4B	−176.8 (3)
C2A—C21A—C22A—C23A	175.1 (3)	N1B—C2B—N3B—C31B	179.6 (2)
C21A—C22A—C23A—C24A	179.0 (3)	C21B—C2B—N3B—C31B	2.8 (4)
N1A—C2A—N3A—C4A	0.0 (3)	C2B—N3B—C31B—C32B	97.8 (3)
C21A—C2A—N3A—C4A	178.5 (3)	C4B—N3B—C31B—C32B	−82.7 (3)
N1A—C2A—N3A—C31A	176.8 (2)	N3B—C31B—C32B—C37B	12.5 (4)
C21A—C2A—N3A—C31A	−4.7 (4)	N3B—C31B—C32B—C33B	−170.0 (2)
C2A—N3A—C31A—C32A	−104.2 (3)	C37B—C32B—C33B—C34B	−0.7 (4)
C4A—N3A—C31A—C32A	71.9 (3)	C31B—C32B—C33B—C34B	−178.3 (3)
N3A—C31A—C32A—C37A	12.6 (4)	C32B—C33B—C34B—C35B	0.5 (4)
N3A—C31A—C32A—C33A	−166.4 (2)	C33B—C34B—C35B—C36B	0.2 (4)
C37A—C32A—C33A—C34A	−2.0 (4)	C33B—C34B—C35B—N31B	179.4 (3)
C31A—C32A—C33A—C34A	177.0 (2)	C34B—C35B—C36B—C37B	−0.7 (4)

C32A—C33A—C34A—C35A	0.4 (4)	N31B—C35B—C36B—C37B	−180.0 (3)
C33A—C34A—C35A—C36A	1.8 (4)	C33B—C32B—C37B—C36B	0.1 (4)
C33A—C34A—C35A—N31A	−178.2 (2)	C31B—C32B—C37B—C36B	177.6 (3)
C34A—C35A—C36A—C37A	−2.4 (4)	C35B—C36B—C37B—C32B	0.5 (4)
N31A—C35A—C36A—C37A	177.6 (2)	C36B—C35B—N31B—O31B	−177.7 (3)
C35A—C36A—C37A—C32A	0.7 (4)	C34B—C35B—N31B—O31B	3.0 (4)
C33A—C32A—C37A—C36A	1.4 (4)	C36B—C35B—N31B—O32B	2.8 (4)
C31A—C32A—C37A—C36A	−177.6 (3)	C34B—C35B—N31B—O32B	−176.5 (3)
C36A—C35A—N31A—O31A	−177.8 (2)	C2B—N3B—C4B—C5B	0.0 (3)
C34A—C35A—N31A—O31A	2.2 (4)	C31B—N3B—C4B—C5B	−179.6 (2)
C36A—C35A—N31A—O32A	2.1 (4)	C2B—N3B—C4B—C41B	180.0 (3)
C34A—C35A—N31A—O32A	−178.0 (2)	C31B—N3B—C4B—C41B	0.4 (4)
C2A—N3A—C4A—C5A	−0.1 (3)	C5B—C4B—C41B—O41B	179.0 (3)
C31A—N3A—C4A—C5A	−176.9 (2)	N3B—C4B—C41B—O41B	−0.9 (5)
C2A—N3A—C4A—C41A	179.7 (3)	C2B—N1B—C5B—C4B	0.0 (3)
C31A—N3A—C4A—C41A	2.9 (4)	C2B—N1B—C5B—Cl5B	−179.8 (2)
C5A—C4A—C41A—O41A	−178.4 (3)	N3B—C4B—C5B—N1B	0.0 (3)
N3A—C4A—C41A—O41A	1.8 (5)	C41B—C4B—C5B—N1B	180.0 (3)
C2A—N1A—C5A—C4A	−0.1 (3)	N3B—C4B—C5B—Cl5B	179.8 (2)
C2A—N1A—C5A—Cl5A	179.7 (2)	C41B—C4B—C5B—Cl5B	−0.2 (4)
N3A—C4A—C5A—N1A	0.1 (3)	N1B—C2B—C21B—C22B	16.0 (5)
C41A—C4A—C5A—N1A	−179.7 (3)	N3B—C2B—C21B—C22B	−167.5 (3)
N3A—C4A—C5A—Cl5A	−179.6 (2)	C2B—C21B—C22B—C23B	70.9 (6)
C41A—C4A—C5A—Cl5A	0.6 (5)	C21B—C22B—C23B—C24B	−173.9 (5)