Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 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.

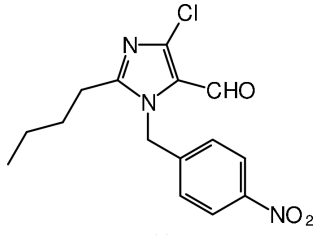


Received 25 November 2004
Accepted 26 November 2004
Online 30 November 2004

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
$\alpha$ = 103.420 (4)°
$\beta$ = 95.561 (3)°
$\gamma$ = 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
$\theta$ = 2.9–27.5°
$\mu$ = 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
$\varphi$ and $\omega$ 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
$\theta_{max}$ = 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 1.25$U_{eq}$ 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.

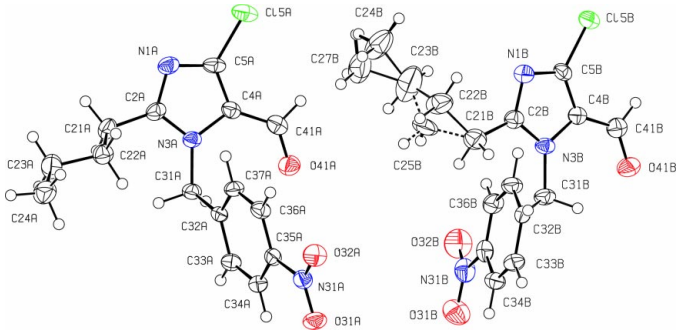



**Figure 1**
The molecular configurations and atom-numbering schemes for the two independent molecules, *A* and *B*, of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

The authors thank the EPSRC National Crystallography Service (Southampton, England) and acknowledge the use of the EPSRC's Chemical Database Service at Daresbury.

## 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.

# supporting information

*Acta Cryst.* (2004). E**60**, o2520–o2521 [https://doi.org/10.1107/S1600536804031162]

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*Crystal data*

$C_{15}H_{16}ClN_3O_3$
$M_r = 321.76$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.3007\ (5)$ Å
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$c = 16.5605\ (10)$ Å
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$\beta = 95.561\ (3)°$
$\gamma = 106.758\ (3)°$
$V = 1541.35\ (15)$ 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$^{-1}$
$\varphi$ and $\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.098$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.9°$
$h = -10\rightarrow10$
$k = -15\rightarrow15$
$l = -20\rightarrow20$

*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) |

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|---|---|---|---|---|---|---|
| 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) |

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|---|---|---|---|
| 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) | | |

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|---|---|---|---|
| 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 |

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|---|---|---|---|
| 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) |

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|---|---|---|---|
| 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) |