Acta Crystallographica Section E
**Structure Reports Online**

ISSN 1600-5368

# *syn*-3-(4-Chlorobenzyl)-1,5-dimethyl-3,7-diazabicyclo[3.3.1]nonan-9-ol

**Konstantin V. Kudryavtsev,[a,b]* Sergey Z. Vatsadze,[a] Vera S. Semashko[a] and Andrei V. Churakov[c]**

[a]Department of Chemistry, M. V. Lomonosov Moscow State University, Leninskie Gory 1/3, Moscow 119991, Russian Federation, [b]Institute of Physiologically Active Compounds, Russian Academy of Sciences, Chernogolovka 142432, Moscow Region, Russian Federation, and [c]Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii prospekt 31, Moscow 119991, Russian Federation
Correspondence e-mail: kudr@org.chem.msu.ru

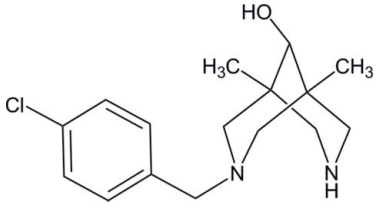
Received 2 July 2012; accepted 3 July 2012

Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.042; $wR$ factor = 0.107; data-to-parameter ratio = 13.7.

In the title compound, $C_{16}H_{23}ClN_2O$, both six-membered rings adopt chair conformations, thus allowing the formation of an intramolecular N—H···N hydrogen bond. In the crystal, adjacent molecules are combined into chains running along the *ac* diagonal *via* O—H···N hydrogen bonds.

## Related literature

For general background to chemistry affording *syn*-3,7-diazabicyclo[3.3.1]nonan-9-ols, see: Vatsadze *et al.* (2006). 3,5,6,7-Tetrasubstituted 3,6-diazabicyclo[3.2.1]octanes, their biological activity as enzyme inhibitors and their X-ray structures have been reported by: Kudryavtsev (2010); Kudryavtsev & Churakov (2012).




## Experimental

### Crystal data

$C_{16}H_{23}ClN_2O$
$M_r$ = 294.81
Monoclinic, $P2_1/n$
$a$ = 7.9739 (4) Å
$b$ = 16.8120 (9) Å
$c$ = 12.1103 (6) Å
$\beta$ = 107.520 (1)°
$V$ = 1548.16 (14) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.25 mm$^{-1}$
$T$ = 120 K
0.25 × 0.20 × 0.20 mm

### Data collection

Bruker SMART 1K diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.941, $T_{max}$ = 0.953
10450 measured reflections
3747 independent reflections
2976 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
$S$ = 1.02
3747 reflections
273 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···N1 | 0.870 (19) | 2.287 (19) | 2.8327 (18) | 120.8 (16) |
| O1—H1···N2$^{i}$ | 0.87 (2) | 1.86 (2) | 2.7242 (17) | 172 (2) |

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This study was partially supported by the Russian Foundation for Basic Research (project Nos. 11-03-00630_a, 11-03-91375-ST_a and 11-03-12101-ofi-m-2011) and RAS Programme OXHM 9 'Medicinal Chemistry'. The authors thank Professor J. A. K. Howard (Durham University) for access to X-ray facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2075).

## References

Bruker (2008). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kudryavtsev, K. V. (2010). *Russ. J. Org. Chem.* **46**, 372–379.
Kudryavtsev, K. V. & Churakov, A. V. (2012). *Acta Cryst.* E**68**, o1718.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Vatsadze, S. Z., Tyurin, V. S., Zatsman, A. I., Manaenkova, M. A., Semashko, V. S., Krut'ko, D. P., Zyk, N. V., Churakov, A. V. & Kuz'mina, L. G. (2006). *Russ. J. Org. Chem.* **42**, 1225–1233.

# supporting information

*Acta Cryst.* (2012). E**68**, o2373 [https://doi.org/10.1107/S1600536812030310]

## *syn*-3-(4-Chlorobenzyl)-1,5-dimethyl-3,7-diazabicyclo[3.3.1]nonan-9-ol

**Konstantin V. Kudryavtsev, Sergey Z. Vatsadze, Vera S. Semashko and Andrei V. Churakov**

### S1. Comment

In the title compound, both six-membered cycles adopt chair conformation (Fig. 1). The latter allows the formation of intramolecular N—H···N hydrogen bond. The hydroxyl group at the 9th position of the heterocyclic scaffold is situated closer to the tertiary N-atom. In crystal, the adjacent molecules are combined in chains running along *ac*-diagonal *via* O—H···N hydrogen bonds (Fig. 2).

*syn*-3,7-Diazabicyclo[3.3.1]nonan-9-ols are of interest as a structural motif for enzymes inhibitors. Inhibition of thrombin by substituted 3,6-diazabicyclo[3.2.1]octanes is reported (Kudryavtsev (2010)).

### S2. Experimental

The solution of 2.23 g (6.9 mmol) of *anti*-5-(4-chlorobenzyl)-3-formyl- 1,5-dimethyl-3,7-diazabicyclo[3.3.1]nonan-9-ol (Vatsadze *et al.*(2006)) in conc. HCl (molar ratio *ca* 1:120) was refluxed for 21 h. The cooled solution was neutralized with NaOH to the pH 9–10. The precipitate formed was filtered off, washed with water to neutral pH of mother liquor, then once with ether, recrystallized from EtOH. *syn*-3-(4-Chlorobenzyl)-1,5-dimethyl-3,7-diazabicyclo[3.3.1]nonan-9-ol. Yield 1.50 g (74%), colourless crystals, sublimated at 473 K. $^1$H NMR (dmso-$d_6$): $\delta$ 0.63 (s, 6H, 2$CH_3$); 2.26–2.41 (m, 6H, H-6a, H-8a, H-4 e, H-4a, H-2 e, H-2a); 2.72 (d, 2H, H-6 e, H-8 e, *J* 13.3); 2.98 (d, 1H, H-9, *J* 4.5); 3.27 (s, 2H, $CH_2$Ar); 4.74 (d, 1H, OH, *J* 5.0); 7.28, 7.38 (both d, both 2H, CH(3,5)(Ar), CH(2,6)(Ar), *J* 8.3). $^1$H NMR (dmso-$d_6$ + $CF_3COOH$): $\delta$ 0.77 (s, 6H, 2$CH_3$); 2.36 (d, 2H, H-2a, H-4a, *J* 10.6); 2.48 (d, 2H, H-2 e, H-4 e, *J* 12.4); 2.86 (d, 2H, H-6a, H-8a, *J* 11.9); 3.21 (d, 2H, H-6 e, H-8 e, *J* 12.6); 3.24 (s, 1H, H-9); 3.43 (s, 2H, $CH_2$Ar); 7.34–7.43 (m, 4H, H(Ar)). $^{13}$C NMR (dmso-$d_6$ + $CF_3COOH$): $\delta$ 20.3 (2$CH_3$); 35.4 (C-1, C-5); 53.7 (C-6, C-8); 56.5 (C-2, C-4); 61.2 ($CH_2$Ar); 73.4 (C-9); 117.4, 128.7, 131.7, 136.0 (C(Ph)). Anal. Calcd. for $C_{16}H_{23}ClN_2O$: C, 65.20; H, 7.81; N, 9.51. Found: C, 65.52; H, 7.98; N, 9.35.

### S3. Refinement

All hydrogen atoms were located in a difference Fourier map and refined isotropically.

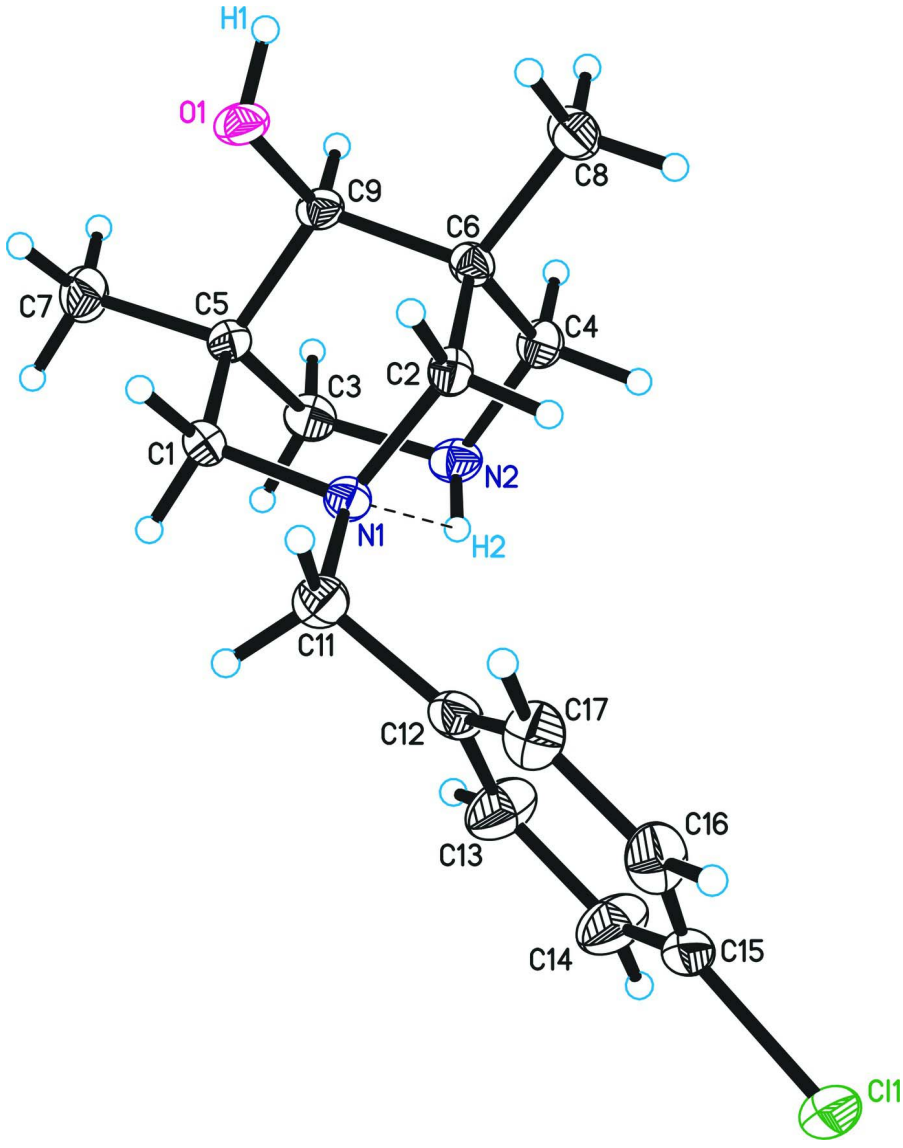



**Figure 1**

The molecular structure of the title compound, showing the numbering scheme adopted. Displacement ellipsoids are shown at the 50% probability level. Intramolecular hydrogen bond is drawn as dashed line.

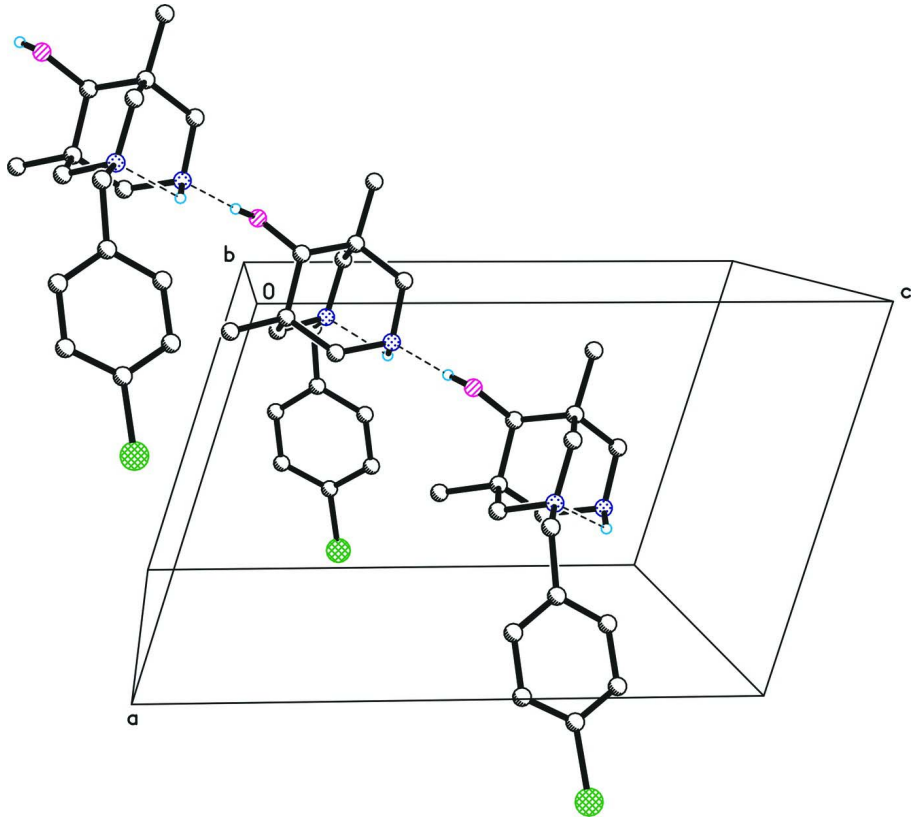



**Figure 2**

Hydrogen bonded chains spreads along *ac*-diagonal. Hydrogen bonds are drawn as dashed lines.

**syn-3-(4-Chlorobenzyl)-1,5-dimethyl-3,7-diazabicyclo[3.3.1]nonan-9-ol**

*Crystal data*

$C_{16}H_{23}ClN_2O$
$M_r = 294.81$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.9739\ (4)$ Å
$b = 16.8120\ (9)$ Å
$c = 12.1103\ (6)$ Å
$\beta = 107.520\ (1)°$
$V = 1548.16\ (14)$ Å$^3$
$Z = 4$
$F(000) = 632$
$D_x = 1.265$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4857 reflections
$\theta = 2.7–30.0°$
$\mu = 0.25$ mm$^{-1}$
$T = 120$ K
Prism, colourless
0.25 × 0.20 × 0.20 mm

*Data collection*

Bruker SMART 1K **[or APEXII?]** diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.941$, $T_{max} = 0.953$
10450 measured reflections
3747 independent reflections
2976 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.1°$
$h = -10\rightarrow9$
$k = -22\rightarrow14$
$l = -15\rightarrow15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.107$
$S = 1.02$
3747 reflections
273 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.4862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.36$ e Å$^{-3}$

*Special details*

**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.

**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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.97589 (5) | 1.10266 (3) | 0.39054 (4) | 0.03592 (14) |
| O1 | −0.15380 (14) | 0.82506 (6) | −0.00656 (9) | 0.0220 (2) |
| N1 | 0.16827 (16) | 0.92965 (7) | 0.19544 (10) | 0.0176 (3) |
| N2 | 0.22587 (17) | 0.78671 (8) | 0.32531 (11) | 0.0211 (3) |
| C1 | −0.01659 (19) | 0.92287 (8) | 0.19298 (13) | 0.0180 (3) |
| C2 | 0.20407 (19) | 0.88120 (9) | 0.10373 (13) | 0.0176 (3) |
| C3 | 0.0379 (2) | 0.79594 (9) | 0.31288 (13) | 0.0216 (3) |
| C4 | 0.2528 (2) | 0.75254 (9) | 0.22011 (13) | 0.0204 (3) |
| C5 | −0.07319 (18) | 0.83646 (8) | 0.20054 (12) | 0.0173 (3) |
| C6 | 0.14948 (19) | 0.79358 (8) | 0.10650 (12) | 0.0168 (3) |
| C7 | −0.2661 (2) | 0.83514 (11) | 0.19815 (17) | 0.0265 (3) |
| C8 | 0.1851 (2) | 0.74991 (10) | 0.00522 (14) | 0.0244 (3) |
| C9 | −0.04692 (19) | 0.79066 (8) | 0.09780 (12) | 0.0173 (3) |
| C11 | 0.2140 (2) | 1.01297 (9) | 0.18344 (15) | 0.0224 (3) |
| C12 | 0.4088 (2) | 1.02924 (8) | 0.23069 (14) | 0.0212 (3) |
| C13 | 0.4991 (2) | 1.01062 (12) | 0.34495 (15) | 0.0332 (4) |
| C14 | 0.6739 (2) | 1.03189 (12) | 0.39435 (16) | 0.0350 (4) |
| C15 | 0.7590 (2) | 1.07151 (9) | 0.32730 (15) | 0.0249 (3) |
| C16 | 0.6764 (2) | 1.08801 (10) | 0.21243 (16) | 0.0284 (4) |
| C17 | 0.5000 (2) | 1.06683 (10) | 0.16459 (15) | 0.0267 (4) |
| H1 | −0.185 (3) | 0.7863 (13) | −0.056 (2) | 0.047 (6)* |
| H2 | 0.275 (2) | 0.8334 (12) | 0.3320 (17) | 0.029 (5)* |
| H9 | −0.082 (2) | 0.7325 (11) | 0.1019 (15) | 0.024 (5)* |
| H11 | −0.029 (2) | 0.9525 (9) | 0.2591 (14) | 0.014 (4)* |
| H12 | −0.097 (2) | 0.9484 (10) | 0.1210 (15) | 0.022 (4)* |

| | | | | |
|---|---|---|---|---|
| H21 | 0.328 (2) | 0.8846 (10) | 0.1130 (14) | 0.017 (4)* |
| H22 | 0.144 (2) | 0.9043 (10) | 0.0278 (15) | 0.018 (4)* |
| H31 | −0.013 (2) | 0.7404 (11) | 0.3163 (16) | 0.027 (5)* |
| H32 | 0.032 (2) | 0.8250 (10) | 0.3794 (16) | 0.021 (4)* |
| H41 | 0.219 (2) | 0.6942 (10) | 0.2163 (14) | 0.017 (4)* |
| H42 | 0.382 (2) | 0.7540 (10) | 0.2280 (14) | 0.018 (4)* |
| H13 | 0.438 (3) | 0.9822 (12) | 0.3919 (18) | 0.041 (6)* |
| H14 | 0.737 (3) | 1.0203 (13) | 0.4736 (19) | 0.043 (6)* |
| H16 | 0.736 (3) | 1.1168 (14) | 0.168 (2) | 0.054 (7)* |
| H17 | 0.438 (3) | 1.0795 (12) | 0.0842 (19) | 0.043 (6)* |
| H71 | −0.343 (3) | 0.8633 (12) | 0.1288 (18) | 0.040 (6)* |
| H72 | −0.309 (3) | 0.7796 (13) | 0.1971 (19) | 0.046 (6)* |
| H73 | −0.279 (3) | 0.8637 (13) | 0.2655 (19) | 0.046 (6)* |
| H81 | 0.309 (3) | 0.7521 (12) | 0.0095 (17) | 0.036 (5)* |
| H82 | 0.116 (2) | 0.7740 (11) | −0.0672 (17) | 0.029 (5)* |
| H83 | 0.151 (2) | 0.6936 (12) | 0.0048 (15) | 0.027 (5)* |
| H111 | 0.153 (2) | 1.0450 (11) | 0.2285 (16) | 0.029 (5)* |
| H112 | 0.172 (2) | 1.0293 (11) | 0.1034 (17) | 0.032 (5)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0236 (2) | 0.0411 (3) | 0.0449 (3) | −0.01249 (17) | 0.01322 (18) | −0.0198 (2) |
| O1 | 0.0232 (6) | 0.0192 (5) | 0.0174 (5) | 0.0022 (4) | −0.0034 (4) | −0.0017 (4) |
| N1 | 0.0177 (6) | 0.0145 (6) | 0.0201 (6) | −0.0009 (4) | 0.0047 (5) | −0.0012 (5) |
| N2 | 0.0240 (7) | 0.0205 (6) | 0.0159 (6) | −0.0001 (5) | 0.0017 (5) | 0.0010 (5) |
| C1 | 0.0180 (7) | 0.0165 (7) | 0.0196 (7) | 0.0016 (5) | 0.0059 (6) | −0.0023 (6) |
| C2 | 0.0162 (7) | 0.0189 (7) | 0.0177 (7) | 0.0014 (5) | 0.0051 (6) | 0.0010 (5) |
| C3 | 0.0262 (8) | 0.0226 (8) | 0.0167 (7) | −0.0007 (6) | 0.0074 (6) | 0.0013 (6) |
| C4 | 0.0203 (7) | 0.0194 (7) | 0.0191 (7) | 0.0041 (6) | 0.0026 (6) | 0.0026 (6) |
| C5 | 0.0162 (7) | 0.0168 (7) | 0.0184 (7) | −0.0009 (5) | 0.0046 (6) | −0.0012 (5) |
| C6 | 0.0192 (7) | 0.0157 (7) | 0.0152 (7) | 0.0023 (5) | 0.0045 (6) | 0.0001 (5) |
| C7 | 0.0201 (8) | 0.0281 (9) | 0.0335 (9) | −0.0020 (6) | 0.0115 (7) | −0.0024 (7) |
| C8 | 0.0279 (9) | 0.0247 (8) | 0.0215 (8) | 0.0045 (6) | 0.0086 (7) | −0.0019 (6) |
| C9 | 0.0185 (7) | 0.0146 (7) | 0.0159 (7) | 0.0008 (5) | 0.0007 (5) | 0.0003 (5) |
| C11 | 0.0230 (8) | 0.0158 (7) | 0.0267 (8) | −0.0008 (6) | 0.0048 (6) | 0.0015 (6) |
| C12 | 0.0239 (8) | 0.0152 (7) | 0.0253 (8) | −0.0020 (6) | 0.0084 (6) | −0.0038 (6) |
| C13 | 0.0274 (9) | 0.0496 (11) | 0.0237 (9) | −0.0135 (8) | 0.0093 (7) | −0.0002 (8) |
| C14 | 0.0277 (9) | 0.0543 (12) | 0.0217 (9) | −0.0116 (8) | 0.0053 (7) | −0.0041 (8) |
| C15 | 0.0203 (7) | 0.0236 (8) | 0.0329 (9) | −0.0061 (6) | 0.0108 (7) | −0.0114 (7) |
| C16 | 0.0287 (9) | 0.0229 (8) | 0.0379 (9) | −0.0015 (6) | 0.0166 (8) | 0.0046 (7) |
| C17 | 0.0271 (8) | 0.0238 (8) | 0.0294 (9) | 0.0018 (6) | 0.0087 (7) | 0.0064 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C15 | 1.7476 (16) | C6—C8 | 1.528 (2) |
| O1—C9 | 1.4175 (17) | C6—C9 | 1.538 (2) |
| O1—H1 | 0.87 (2) | C7—H71 | 1.00 (2) |

| | | | |
|---|---|---|---|
| N1—C11 | 1.4656 (19) | C7—H72 | 0.99 (2) |
| N1—C1 | 1.4698 (19) | C7—H73 | 0.98 (2) |
| N1—C2 | 1.4729 (19) | C8—H81 | 0.97 (2) |
| N2—C3 | 1.469 (2) | C8—H82 | 0.971 (19) |
| N2—C4 | 1.471 (2) | C8—H83 | 0.984 (19) |
| N2—H2 | 0.870 (19) | C9—H9 | 1.021 (18) |
| C1—C5 | 1.532 (2) | C11—C12 | 1.510 (2) |
| C1—H11 | 0.972 (16) | C11—H111 | 0.993 (19) |
| C1—H12 | 1.010 (18) | C11—H112 | 0.97 (2) |
| C2—C6 | 1.539 (2) | C12—C17 | 1.386 (2) |
| C2—H21 | 0.962 (17) | C12—C13 | 1.390 (2) |
| C2—H22 | 0.980 (17) | C13—C14 | 1.388 (2) |
| C3—C5 | 1.541 (2) | C13—H13 | 0.98 (2) |
| C3—H31 | 1.026 (19) | C14—C15 | 1.377 (2) |
| C3—H32 | 0.955 (18) | C14—H14 | 0.96 (2) |
| C4—C6 | 1.5381 (19) | C15—C16 | 1.376 (3) |
| C4—H41 | 1.014 (17) | C16—C17 | 1.397 (2) |
| C4—H42 | 1.005 (17) | C16—H16 | 0.95 (2) |
| C5—C7 | 1.530 (2) | C17—H17 | 0.97 (2) |
| C5—C9 | 1.530 (2) | | |

| | | | |
|---|---|---|---|
| C9—O1—H1 | 106.2 (15) | C5—C7—H71 | 111.5 (12) |
| C11—N1—C1 | 110.47 (11) | C5—C7—H72 | 110.7 (12) |
| C11—N1—C2 | 110.08 (12) | H71—C7—H72 | 108.4 (17) |
| C1—N1—C2 | 111.27 (11) | C5—C7—H73 | 109.4 (12) |
| C3—N2—C4 | 111.30 (12) | H71—C7—H73 | 106.2 (17) |
| C3—N2—H2 | 109.3 (13) | H72—C7—H73 | 110.6 (18) |
| C4—N2—H2 | 104.8 (13) | C6—C8—H81 | 111.8 (12) |
| N1—C1—C5 | 112.58 (11) | C6—C8—H82 | 109.5 (11) |
| N1—C1—H11 | 106.9 (9) | H81—C8—H82 | 108.6 (16) |
| C5—C1—H11 | 109.4 (9) | C6—C8—H83 | 110.5 (11) |
| N1—C1—H12 | 111.0 (10) | H81—C8—H83 | 108.0 (16) |
| C5—C1—H12 | 109.4 (10) | H82—C8—H83 | 108.5 (15) |
| H11—C1—H12 | 107.3 (13) | O1—C9—C5 | 109.08 (11) |
| N1—C2—C6 | 113.11 (12) | O1—C9—C6 | 111.99 (12) |
| N1—C2—H21 | 107.7 (10) | C5—C9—C6 | 108.29 (11) |
| C6—C2—H21 | 110.0 (10) | O1—C9—H9 | 109.6 (10) |
| N1—C2—H22 | 109.7 (10) | C5—C9—H9 | 109.6 (10) |
| C6—C2—H22 | 109.8 (10) | C6—C9—H9 | 108.2 (10) |
| H21—C2—H22 | 106.2 (14) | N1—C11—C12 | 113.18 (12) |
| N2—C3—C5 | 115.77 (12) | N1—C11—H111 | 106.7 (11) |
| N2—C3—H31 | 107.9 (10) | C12—C11—H111 | 108.2 (11) |
| C5—C3—H31 | 107.9 (10) | N1—C11—H112 | 110.5 (11) |
| N2—C3—H32 | 106.1 (11) | C12—C11—H112 | 109.6 (11) |
| C5—C3—H32 | 110.9 (10) | H111—C11—H112 | 108.5 (15) |
| H31—C3—H32 | 108.0 (15) | C17—C12—C13 | 118.22 (15) |
| N2—C4—C6 | 114.89 (12) | C17—C12—C11 | 121.53 (15) |
| N2—C4—H41 | 108.1 (9) | C13—C12—C11 | 120.16 (14) |

| | | | |
|---|---|---|---|
| C6—C4—H41 | 109.2 (9) | C14—C13—C12 | 121.57 (16) |
| N2—C4—H42 | 108.7 (10) | C14—C13—H13 | 119.2 (12) |
| C6—C4—H42 | 109.4 (10) | C12—C13—H13 | 119.2 (12) |
| H41—C4—H42 | 106.2 (13) | C15—C14—C13 | 118.67 (17) |
| C7—C5—C9 | 111.12 (12) | C15—C14—H14 | 118.9 (13) |
| C7—C5—C1 | 108.97 (12) | C13—C14—H14 | 122.4 (13) |
| C9—C5—C1 | 108.28 (12) | C16—C15—C14 | 121.53 (16) |
| C7—C5—C3 | 108.52 (13) | C16—C15—Cl1 | 119.45 (13) |
| C9—C5—C3 | 108.21 (12) | C14—C15—Cl1 | 119.01 (14) |
| C1—C5—C3 | 111.76 (12) | C15—C16—C17 | 118.92 (16) |
| C8—C6—C4 | 108.87 (12) | C15—C16—H16 | 120.4 (15) |
| C8—C6—C9 | 111.18 (12) | C17—C16—H16 | 120.5 (14) |
| C4—C6—C9 | 107.90 (12) | C12—C17—C16 | 121.00 (16) |
| C8—C6—C2 | 108.67 (12) | C12—C17—H17 | 118.8 (13) |
| C4—C6—C2 | 111.71 (12) | C16—C17—H17 | 120.2 (13) |
| C9—C6—C2 | 108.54 (11) | | |

| | | | |
|---|---|---|---|
| C11—N1—C1—C5 | 177.79 (12) | C3—C5—C9—C6 | −60.28 (14) |
| C2—N1—C1—C5 | 55.20 (16) | C8—C6—C9—O1 | −58.82 (15) |
| C11—N1—C2—C6 | −176.75 (12) | C4—C6—C9—O1 | −178.14 (11) |
| C1—N1—C2—C6 | −53.93 (15) | C2—C6—C9—O1 | 60.65 (15) |
| C4—N2—C3—C5 | −48.59 (17) | C8—C6—C9—C5 | −179.14 (12) |
| C3—N2—C4—C6 | 49.63 (17) | C4—C6—C9—C5 | 61.55 (14) |
| N1—C1—C5—C7 | 179.66 (13) | C2—C6—C9—C5 | −59.66 (14) |
| N1—C1—C5—C9 | −59.36 (15) | C1—N1—C11—C12 | 157.40 (13) |
| N1—C1—C5—C3 | 59.73 (16) | C2—N1—C11—C12 | −79.32 (16) |
| N2—C3—C5—C7 | 175.66 (13) | N1—C11—C12—C17 | 127.48 (16) |
| N2—C3—C5—C9 | 54.98 (16) | N1—C11—C12—C13 | −56.2 (2) |
| N2—C3—C5—C1 | −64.15 (16) | C17—C12—C13—C14 | 2.9 (3) |
| N2—C4—C6—C8 | −177.88 (13) | C11—C12—C13—C14 | −173.55 (17) |
| N2—C4—C6—C9 | −57.11 (16) | C12—C13—C14—C15 | −0.8 (3) |
| N2—C4—C6—C2 | 62.10 (17) | C13—C14—C15—C16 | −2.1 (3) |
| N1—C2—C6—C8 | 177.65 (12) | C13—C14—C15—Cl1 | 177.03 (15) |
| N1—C2—C6—C4 | −62.22 (16) | C14—C15—C16—C17 | 2.7 (3) |
| N1—C2—C6—C9 | 56.62 (15) | Cl1—C15—C16—C17 | −176.40 (13) |
| C7—C5—C9—O1 | 58.55 (16) | C13—C12—C17—C16 | −2.2 (2) |
| C1—C5—C9—O1 | −61.09 (15) | C11—C12—C17—C16 | 174.16 (15) |
| C3—C5—C9—O1 | 177.60 (12) | C15—C16—C17—C12 | −0.5 (3) |
| C7—C5—C9—C6 | −179.33 (12) | C5—C9—O1—H1 | −147.5 (15) |
| C1—C5—C9—C6 | 61.02 (14) | C6—C9—O1—H1 | 92.6 (15) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···N1 | 0.870 (19) | 2.287 (19) | 2.8327 (18) | 120.8 (16) |
| O1—H1···N2$^{i}$ | 0.87 (2) | 1.86 (2) | 2.7242 (17) | 172 (2) |

Symmetry code: (i) $x-1/2, -y+3/2, z-1/2$.