Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# Crystal structure and theoretical study of *N*,*N*-bis[(5-chloro-2-oxo-2,3-dihydrobenzo[*d*]-oxazol-3-yl)methyl]-2-phenylethanamine

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Received 16 March 2018
Accepted 18 April 2018

Edited by M. Weil, Vienna University of Technology, Austria

**Keywords:** crystal structure; 2,3-dihydro-1,3-benzoxazole ring; semi-empirical *CNDO*/2 method; HOMO; LUMO.

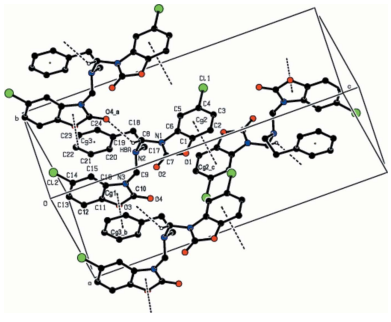
**CCDC reference**: 1838126

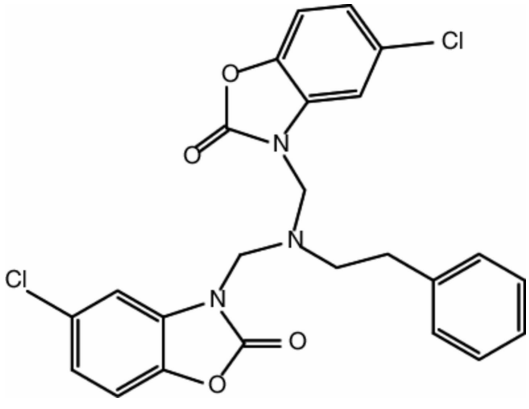
**Supporting information**: this article has supporting information at journals.iucr.org/e

In the molecular structure of the title compound, $C_{24}H_{19}Cl_2N_3O_4$, the three C atoms of the central *N*,*N*-dimethylmethanamine moiety are bonded to the N atoms of the two 5-chloro-1,3-benzoxazol-2(3*H*)-one groups and to the methyl C atom of the methylbenzene group. One of the nine-membered 2,3-dihydro-1,3-benzoxazole rings and the phenyl ring are almost parallel to each other, making a dihedral angle of 5.30 (18)°, but they are almost normal to the mean plane of the other nine-membered 2,3-dihydro-1,3-benzoxazole ring, subtending dihedral angles of 89.29 (16) and 85.41 (18)°, respectively. The crystal structure features C—H···O hydrogen bonds and $\pi$–$\pi$ stacking interactions [centroid-to-centroid distances = 3.5788 (19) Å, slippage = 0.438 and 3.7773 (16) Å, and slippage = 0.716 Å].

## 1. Chemical context

2(3*H*)-Benzoxazolone is a privileged lead molecule for the design of potential bioactive agents, and its derivatives have been shown to posses a broad spectrum of bioactive properties such as anti-HIV (Deng *et al.*, 2006), anticancer (Ivanova *et al.*, 2007), analgesic (Ünlü *et al.*, 2003), anti-inflammatory (Köksal *et al.*, 2005), antinociceptive (Önkol *et al.*, 2001), antimicrobial (Köksal *et al.*, 2002), anticonvulsant (Ucar *et al.*, 1998), anti-malarial (Courtois *et al.*, 2004) and human leukocyte MPO clorinating inhibitor activities (Soyer *et al.*, 2005). In this context, we have investigated another benzoxazolone derivative with formula $C_{24}H_{19}Cl_2N_3O_4$, and report here its synthesis, molecular, crystal and theoretical structures.





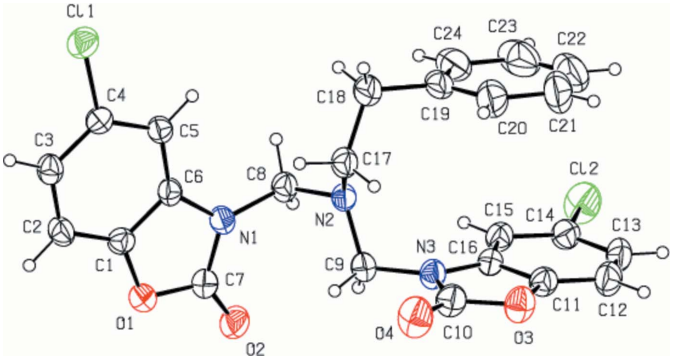



**Figure 1**
View of the title molecule with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

## 2. Structural commentary

The central part of the title molecule (Fig. 1) comprises an *N*,*N*-dimethylmethanamine unit whose three carbon atoms are bonded to the N atoms of the two 5-chloro-1,3-benzoxazol-2(3*H*)-one moieties and to the methyl carbon atom of the methylbenzene group. The nine-membered 2,3-dihydro-1,3-benzoxazole ring (N3/O3/C10–C16) and the phenyl ring (C19–C24) are almost parallel to each other, making a dihedral angle of 5.30 (18)°. These two entities are almost normal to the mean plane of the other 2,3-dihydro-1,3-benzoxazole ring (N1/O1/C1–C7), subtending dihedral angles of 89.29 (16) and 85.41 (18)°, respectively.

The C7—N1—C8—N2, N2—C9—N3—C16, N2—C17—C18—C19 and C17—C18—C19—C24 torsion angles are −90.7 (3), −75.6 (3), −63.6 (3) and 106.1 (4)°, respectively. The bond lengths and angles of the title molecule (Table 1) are normal and correspond to those observed in related benzoxazolone derivatives (Aydın *et al.*, 2004, 2012, 2017; Allen *et al.*, 1987).

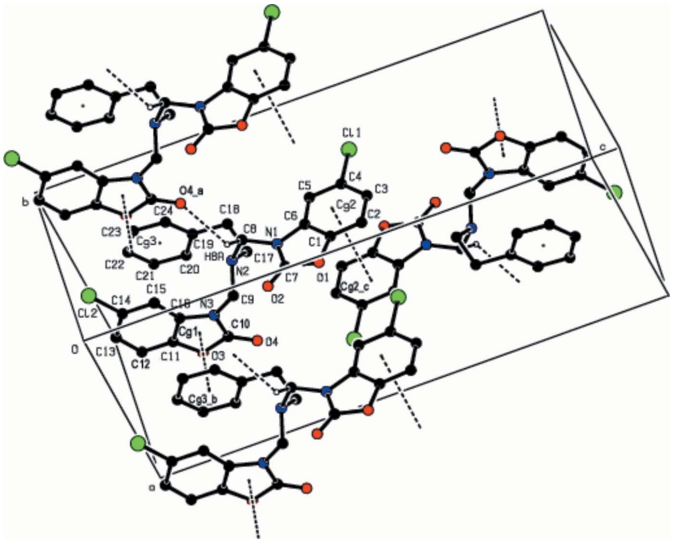



**Figure 2**
A view of the crystal packing in the title structure, showing the C—H···O hydrogen bonding and $\pi$–$\pi$ stacking interactions. H atoms not involved in hydrogen bonds are omitted for the sake of clarity. [Symmetry codes: (*a*) $x-1, y, z$; (*b*) $x, y-1, z$; (*c*) $1-x, 1-y, 1-z$.]

**Table 1**
Comparison of experimental (X-ray) and theoretical (*CNDO*/2) bond lenghts and angles (Å, °) for the title compound.

| Bond | X-ray | *CNDO*/2 |
|---|---|---|
| Cl1—C4 | 1.735 (3) | 1.7379 |
| Cl2—C14 | 1.738 (3) | 1.7382 |
| O1—C1 | 1.382 (3) | 1.3545 |
| O1—C7 | 1.384 (3) | 1.3585 |
| O2—C7 | 1.202 (3) | 1.2091 |
| O3—C10 | 1.371 (3) | 1.3573 |
| O3—C11 | 1.390 (4) | 1.3544 |
| O4—C10 | 1.200 (4) | 1.2090 |
| N1—C6 | 1.398 (3) | 1.3649 |
| N1—C7 | 1.371 (3) | 1.3593 |
| N1—C8 | 1.491 (3) | 1.4587 |
| N2—C8 | 1.430 (4) | 1.4666 |
| N2—C9 | 1.448 (4) | 1.4641 |
| N2—C17 | 1.463 (4) | 1.4672 |
| N3—C9 | 1.444 (3) | 1.4601 |
| N3—C10 | 1.370 (4) | 1.3587 |
| N3—C16 | 1.393 (3) | 1.3663 |
| C17—C18 | 1.520 (4) | 1.5425 |
| C18—C19 | 1.506 (4) | 1.5131 |
| | | |
| C8—N2—C9 | 112.5 (2) | 110.47 |
| C8—N2—C17 | 114.76 (19) | 112.03 |
| C9—N2—C17 | 114.5 (2) | 110.74 |
| Cl1—C4—C3 | 118.03 (19) | 120.09 |
| Cl1—C4—C5 | 118.6 (2) | 119.73 |
| O1—C7—O2 | 123.0 (3) | 124.49 |
| O2—C7—N1 | 129.6 (3) | 127.02 |
| N1—C8—N2 | 115.9 (2) | 112.31 |
| N2—C9—N3 | 111.3 (2) | 111.17 |
| O3—C10—O4 | 123.5 (3) | 124.70 |
| O4—C10—N3 | 128.3 (3) | 126.73 |
| Cl2—C14—C13 | 118.4 (2) | 120.07 |
| Cl2—C14—C15 | 118.6 (2) | 119.71 |
| N2—C17—C18 | 111.8 (2) | 112.23 |
| C17—C18—C19 | 112.9 (2) | 114.03 |
| C18—C19—C20 | 121.2 (3) | 120.60 |
| C18—C19—C24 | 121.2 (3) | 121.28 |

## 3. Supramolecular features

The crystal structure features weak intermolecular C—H···O hydrogen bonds (Table 2, Fig. 2) between a methylene group and a carbonyl O atom of a neighbouring molecule. $\pi$–$\pi$ stacking interactions [$Cg1\cdots Cg3^{ii}$ = 3.5788 (19) Å, slippage = 0.438 Å and $Cg2\cdots Cg2^{iii}$ = 3.7773 (16) Å, slippage = 0.716 Å, symmetry codes: (ii) $x, -1+y, z$, (iii) $1-x, 1-y, 1-z$, where *Cg*1, *Cg*2 and *Cg*3 are the centroids of the O3/N3/C10/C11/C16 2,3-dihydro-1,3-oxazole ring, the C1–C6 benzene ring and the C19–C24 phenyl ring, respectively] additionally consolidate the crystal packing.

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H8*A*···O4$^{i}$ | 0.97 | 2.51 | 3.037 (4) | 114 |

Symmetry code: (i) $x-1, y, z$.

## 4. Theoretical calculations

Semi-empirical molecular orbital (MO) calculations of the title molecule were carried out using the *CNDO/2* method (Pople & Segal, 1966). It is based on the *Complete Neglect of Differential Overlap* integral approximation. The semi-empirical *CNDO/2* parameterization is widely used to derive bond lengths, bond angles, torsion angles, atom charges, *HOMO* and *LUMO* energy levels, dipole moments, polarizability, *etc*. The spatial view of the title compound calculated as a closed-shell in a vacuum at 0 K is shown in Fig. 3.

In the title molecule, the calculated charges on the Cl1, Cl2, O1, O2, O3, O4, N1, N2 and N3 atoms are −0.164, −0.226, −0.424, −0.228, −0.431, −0.117, −0.187 and −0.112 e$^-$, respectively. The calculated dipole moment is about 2.122 Debye. The *HOMO* and *LUMO* energy levels are −10.7480 and 3.4691 eV, respectively.

The calculated bond lengths and angles of the title molecule are consistent with those obtained by X-ray structure determination within error limits (Table 1). Looking at Figs. 1 and 3, the experimental and calculated conformations appear to be quite different. This is supported by the torsion angles N1—C8—N2—C17 [experimental 70.5 (3), calculated 58.25°], N1—C8—N2—C9 [−62.8 (3), −177.78°], N2—C17—C18—C19 [−63.6 (3), −150.35°], C18—C17—N2—C8 [84.1 (3), −95.53°] and C9—N2—C17—18 [143.5 (2), −140.65°]. The small differences between the theoretical and experimental results are due to the calculations being in a vacuum and at 0 K.

## 5. Synthesis and crystallization

4-Chloro-2-aminophenol (10 mmol), urea (50 mmol) and 37%$_{wt}$ HCl (2.5 ml) were irradiated (300 W, 413 K) for 15 min in a microwave oven. After completion of the reaction (monitored with TLC), water (10 ml) was added to the reaction mixture and stirred at room temperature for 1 h. The resulting precipitate was filtered and washed with water. After drying the precipitate, crystallization from ethanol–water (1:1 *v*/*v*) yielded 5-chloro-2(3*H*)-benzoxazolone. This compound (2 mmol) was dissolved in methanol (5 ml). Phenethylamine (2 mmol) and 37%$_{wt}$ formalin (2.5 mmol) were added to this solution. The mixture was then stirred vigorously for 1 h. The resulting precipitate was filtered and washed with cold methanol. The crude product was recrystallized from methanol, yield 40%; m.p. 427 K.

IR $\upsilon_{max}$ (FTIR/ATR): 3062, 2862, 1769, 1038 cm$^{-1}$; $^1$H NMR ($CDCl_3$): $\delta$ 2.79 (2H, *t*, *J* = 6.8 Hz, $NCH_2CH_2$), 3.14 (2H, *t*, *J* = 7.0 Hz, $CH_2CH_2$-phenyl) 4.90 (4H, *s*, 2 × $CH_2$), 6.88–7.16 (11H, *m*, Ar-H) ppm; MS (ESI) *m*/*z* (%): 315 (100), 317 (37), 484 (*M* + H, 3), 486 (*M* + H + 2, 1).

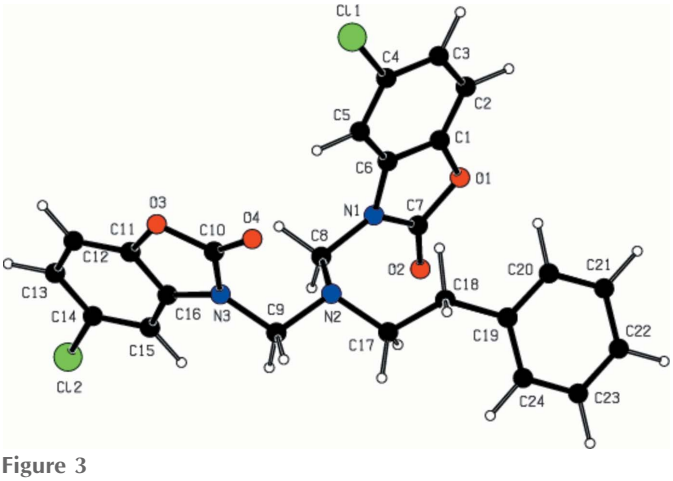



**Figure 3**
The molecular structure of the title compound calculated using the *CNDO/2* method.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{24}H_{19}Cl_2N_3O_4$ |
| $M_r$ | 484.32 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 296 |
| *a*, *b*, *c* (Å) | 7.4028 (5), 7.4432 (5), 22.4616 (15) |
| $\alpha$, $\beta$, $\gamma$ (°) | 97.255 (5), 90.274 (5), 114.784 (5) |
| *V* (Å$^3$) | 1112.36 (14) |
| *Z* | 2 |
| Radiation type | Mo *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.33 |
| Crystal size (mm) | 0.61 × 0.26 × 0.04 |
| | |
| Data collection | |
| Diffractometer | Stoe IPDS 2 |
| Absorption correction | Integration (*X-RED32*; Stoe & Cie, 2002) |
| $T_{min}$, $T_{max}$ | 0.901, 0.987 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15409, 4604, 2261 |
| $R_{int}$ | 0.083 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.628 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.043, 0.089, 0.87 |
| No. of reflections | 4604 |
| No. of parameters | 298 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.13, −0.17 |

Computer programs: *X-AREA* and *X-RED32* (Stoe & Cie, 2002), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 (aromatic) and 0.97 (methylene) Å and $U_{iso}$ = 1.2$U_{eq}$(C).

## Acknowledgements

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS 2 diffractometer (purchased under grant F.279 of the University Research Fund).

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Ünlü, S., Önkol, T., Dündar, Y., Ökçelik, B., Küpeli, E., Yeşilada, E., Noyanalpan, N. & Şahin, M. F. (2003). *Arch. Pharm. Pharm. Med. Chem.* **336**, 353–361.

# supporting information

*Acta Cryst.* (2018). E**74**, 757-760 [https://doi.org/10.1107/S2056989018005984]

# Crystal structure and theoretical study of *N*,*N*-bis[(5-chloro-2-oxo-2,3-dihydrobenzo[*d*]oxazol-3-yl)methyl]-2-phenylethanamine

**Abdullah Aydın, Zeynep Soyer, Mehmet Akkurt and Orhan Büyükgüngör**

## Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); 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, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

## *N*,*N*-Bis[(5-chloro-2-oxo-2,3-dihydrobenzo[*d*]oxazol-3-yl)methyl]-2-phenylethanamine

*Crystal data*

$C_{24}H_{19}Cl_2N_3O_4$
$M_r$ = 484.32
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 7.4028 (5) Å
$b$ = 7.4432 (5) Å
$c$ = 22.4616 (15) Å
$\alpha$ = 97.255 (5)°
$\beta$ = 90.274 (5)°
$\gamma$ = 114.784 (5)°
$V$ = 1112.36 (14) Å$^3$
$Z$ = 2
$F(000)$ = 500
$D_x$ = 1.446 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 12292 reflections
$\theta$ = 1.8–27.2°
$\mu$ = 0.33 mm$^{-1}$
$T$ = 296 K
Plate, light yellow
0.61 × 0.26 × 0.04 mm

*Data collection*

Stoe IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: integration (XRED-32; Stoe & Cie, 2002)
$T_{min}$ = 0.901, $T_{max}$ = 0.987
15409 measured reflections
4604 independent reflections
2261 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.083
$\theta_{max}$ = 26.5°, $\theta_{min}$ = 1.8°
$h$ = −9→9
$k$ = −9→9
$l$ = −28→28

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.089
$S$ = 0.87
4604 reflections
298 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

$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.13$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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 | 0.33895 (11) | 0.91821 (11) | 0.55979 (3) | 0.0695 (3) |
| Cl2 | −0.03618 (13) | 0.22527 (15) | 0.03733 (4) | 0.0993 (4) |
| O1 | 0.1417 (3) | 0.1051 (3) | 0.45148 (8) | 0.0614 (7) |
| O2 | 0.0466 (3) | −0.0234 (3) | 0.35320 (9) | 0.0774 (8) |
| O3 | 0.7070 (3) | 0.3740 (3) | 0.16410 (8) | 0.0729 (8) |
| O4 | 0.7524 (3) | 0.3741 (4) | 0.26394 (9) | 0.0931 (10) |
| N1 | 0.1491 (3) | 0.3147 (3) | 0.38837 (9) | 0.0519 (7) |
| N2 | 0.2740 (3) | 0.4370 (3) | 0.29248 (8) | 0.0487 (8) |
| N3 | 0.4467 (3) | 0.3171 (3) | 0.21984 (9) | 0.0542 (8) |
| C1 | 0.1949 (3) | 0.2919 (4) | 0.48420 (11) | 0.0502 (9) |
| C2 | 0.2325 (4) | 0.3444 (4) | 0.54484 (11) | 0.0567 (10) |
| C3 | 0.2776 (3) | 0.5405 (4) | 0.56779 (11) | 0.0548 (9) |
| C4 | 0.2845 (3) | 0.6740 (4) | 0.52899 (11) | 0.0490 (9) |
| C5 | 0.2442 (3) | 0.6204 (4) | 0.46734 (11) | 0.0487 (9) |
| C6 | 0.2011 (3) | 0.4255 (4) | 0.44584 (10) | 0.0465 (9) |
| C7 | 0.1063 (4) | 0.1186 (4) | 0.39202 (12) | 0.0598 (11) |
| C8 | 0.1117 (4) | 0.3782 (4) | 0.33094 (10) | 0.0548 (9) |
| C9 | 0.3282 (4) | 0.2752 (4) | 0.27152 (11) | 0.0560 (10) |
| C10 | 0.6446 (4) | 0.3553 (4) | 0.22130 (13) | 0.0671 (11) |
| C11 | 0.5420 (4) | 0.3407 (4) | 0.12702 (12) | 0.0570 (10) |
| C12 | 0.5348 (4) | 0.3392 (5) | 0.06646 (13) | 0.0723 (11) |
| C13 | 0.3512 (5) | 0.2999 (5) | 0.03966 (12) | 0.0706 (11) |
| C14 | 0.1891 (4) | 0.2687 (4) | 0.07364 (12) | 0.0622 (11) |
| C15 | 0.1980 (4) | 0.2725 (4) | 0.13546 (12) | 0.0575 (10) |
| C16 | 0.3809 (4) | 0.3077 (4) | 0.16085 (10) | 0.0507 (9) |
| C17 | 0.4427 (4) | 0.6267 (4) | 0.31468 (11) | 0.0577 (10) |
| C18 | 0.4109 (5) | 0.8042 (4) | 0.29870 (11) | 0.0722 (11) |
| C19 | 0.3978 (5) | 0.8067 (4) | 0.23191 (13) | 0.0697 (11) |
| C20 | 0.5647 (6) | 0.8579 (5) | 0.19940 (15) | 0.0932 (14) |
| C21 | 0.5538 (8) | 0.8637 (6) | 0.13801 (19) | 0.122 (2) |
| C22 | 0.3763 (11) | 0.8152 (7) | 0.1095 (2) | 0.136 (3) |
| C23 | 0.2082 (9) | 0.7639 (6) | 0.1404 (2) | 0.121 (2) |

| | | | | |
|---|---|---|---|---|
| C24 | 0.2183 (6) | 0.7582 (5) | 0.20128 (15) | 0.0887 (14) |
| H2 | 0.22790 | 0.25200 | 0.56970 | 0.0680* |
| H3 | 0.30310 | 0.58280 | 0.60900 | 0.0660* |
| H5 | 0.24630 | 0.71120 | 0.44220 | 0.0580* |
| H8A | −0.00220 | 0.26870 | 0.30880 | 0.0660* |
| H8B | 0.07580 | 0.48940 | 0.34060 | 0.0660* |
| H9A | 0.40320 | 0.25540 | 0.30360 | 0.0670* |
| H9B | 0.20820 | 0.15270 | 0.26090 | 0.0670* |
| H12 | 0.64670 | 0.36320 | 0.04440 | 0.0870* |
| H13 | 0.33700 | 0.29450 | −0.00180 | 0.0850* |
| H15 | 0.08810 | 0.25270 | 0.15810 | 0.0690* |
| H17A | 0.56300 | 0.62780 | 0.29750 | 0.0690* |
| H17B | 0.46100 | 0.63870 | 0.35800 | 0.0690* |
| H18A | 0.28880 | 0.80080 | 0.31520 | 0.0870* |
| H18B | 0.52020 | 0.92670 | 0.31720 | 0.0870* |
| H20 | 0.68740 | 0.88940 | 0.21890 | 0.1120* |
| H21 | 0.66850 | 0.90090 | 0.11680 | 0.1460* |
| H22 | 0.36810 | 0.81670 | 0.06830 | 0.1640* |
| H23 | 0.08630 | 0.73270 | 0.12030 | 0.1450* |
| H24 | 0.10250 | 0.72110 | 0.22200 | 0.1070* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0777 (5) | 0.0615 (5) | 0.0641 (5) | 0.0269 (4) | 0.0050 (4) | −0.0006 (4) |
| Cl2 | 0.0934 (6) | 0.1463 (9) | 0.0720 (6) | 0.0683 (6) | −0.0183 (5) | 0.0007 (5) |
| O1 | 0.0774 (12) | 0.0546 (12) | 0.0547 (12) | 0.0289 (10) | 0.0104 (9) | 0.0128 (10) |
| O2 | 0.1047 (16) | 0.0566 (13) | 0.0598 (13) | 0.0252 (12) | 0.0167 (11) | 0.0014 (11) |
| O3 | 0.0563 (11) | 0.1035 (16) | 0.0651 (13) | 0.0394 (12) | 0.0080 (10) | 0.0133 (12) |
| O4 | 0.0797 (15) | 0.142 (2) | 0.0701 (14) | 0.0601 (15) | −0.0131 (12) | 0.0120 (14) |
| N1 | 0.0590 (13) | 0.0511 (13) | 0.0451 (12) | 0.0215 (11) | 0.0062 (10) | 0.0117 (10) |
| N2 | 0.0526 (13) | 0.0542 (14) | 0.0412 (12) | 0.0238 (12) | 0.0059 (10) | 0.0094 (10) |
| N3 | 0.0562 (14) | 0.0671 (15) | 0.0434 (13) | 0.0297 (12) | 0.0032 (10) | 0.0090 (11) |
| C1 | 0.0504 (15) | 0.0532 (17) | 0.0506 (16) | 0.0255 (14) | 0.0034 (12) | 0.0068 (13) |
| C2 | 0.0639 (17) | 0.0652 (19) | 0.0477 (16) | 0.0313 (15) | 0.0018 (13) | 0.0170 (14) |
| C3 | 0.0542 (15) | 0.0677 (19) | 0.0441 (14) | 0.0268 (15) | 0.0000 (12) | 0.0100 (14) |
| C4 | 0.0458 (15) | 0.0503 (16) | 0.0505 (16) | 0.0204 (13) | 0.0030 (12) | 0.0051 (13) |
| C5 | 0.0485 (14) | 0.0529 (16) | 0.0472 (15) | 0.0224 (13) | 0.0068 (11) | 0.0127 (12) |
| C6 | 0.0449 (14) | 0.0559 (17) | 0.0414 (15) | 0.0226 (13) | 0.0077 (11) | 0.0119 (13) |
| C7 | 0.0707 (19) | 0.061 (2) | 0.0515 (18) | 0.0301 (17) | 0.0141 (14) | 0.0125 (15) |
| C8 | 0.0573 (16) | 0.0670 (18) | 0.0434 (14) | 0.0294 (15) | 0.0018 (12) | 0.0086 (13) |
| C9 | 0.0674 (17) | 0.0600 (18) | 0.0432 (15) | 0.0283 (15) | 0.0062 (13) | 0.0117 (13) |
| C10 | 0.0627 (19) | 0.084 (2) | 0.0610 (19) | 0.0375 (17) | 0.0037 (15) | 0.0097 (17) |
| C11 | 0.0551 (17) | 0.0645 (18) | 0.0553 (17) | 0.0290 (15) | 0.0049 (13) | 0.0084 (14) |
| C12 | 0.074 (2) | 0.097 (2) | 0.0583 (19) | 0.0455 (19) | 0.0214 (15) | 0.0207 (17) |
| C13 | 0.090 (2) | 0.092 (2) | 0.0431 (16) | 0.0499 (19) | 0.0109 (16) | 0.0146 (15) |
| C14 | 0.0710 (19) | 0.072 (2) | 0.0510 (17) | 0.0381 (17) | −0.0010 (14) | 0.0069 (15) |
| C15 | 0.0579 (16) | 0.0672 (19) | 0.0540 (16) | 0.0325 (15) | 0.0094 (13) | 0.0097 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0576 (16) | 0.0556 (16) | 0.0438 (15) | 0.0283 (14) | 0.0075 (13) | 0.0087 (12) |
| C17 | 0.0647 (17) | 0.0614 (18) | 0.0443 (15) | 0.0243 (16) | 0.0019 (13) | 0.0070 (13) |
| C18 | 0.102 (2) | 0.0616 (19) | 0.0517 (17) | 0.0336 (18) | 0.0064 (16) | 0.0067 (15) |
| C19 | 0.107 (2) | 0.0455 (17) | 0.0530 (18) | 0.0285 (18) | 0.0043 (18) | 0.0077 (14) |
| C20 | 0.121 (3) | 0.075 (2) | 0.071 (2) | 0.028 (2) | 0.017 (2) | 0.0145 (19) |
| C21 | 0.188 (5) | 0.091 (3) | 0.073 (3) | 0.041 (3) | 0.045 (3) | 0.029 (2) |
| C22 | 0.254 (7) | 0.086 (3) | 0.066 (3) | 0.069 (4) | −0.008 (4) | 0.013 (2) |
| C23 | 0.184 (5) | 0.090 (3) | 0.088 (3) | 0.061 (3) | −0.041 (3) | 0.002 (3) |
| C24 | 0.123 (3) | 0.070 (2) | 0.074 (2) | 0.042 (2) | −0.011 (2) | 0.0095 (18) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.735 (3) | C15—C16 | 1.373 (4) |
| Cl2—C14 | 1.738 (3) | C17—C18 | 1.520 (4) |
| O1—C1 | 1.382 (3) | C18—C19 | 1.506 (4) |
| O1—C7 | 1.384 (3) | C19—C20 | 1.375 (6) |
| O2—C7 | 1.202 (3) | C19—C24 | 1.377 (6) |
| O3—C10 | 1.371 (3) | C20—C21 | 1.388 (5) |
| O3—C11 | 1.390 (4) | C21—C22 | 1.343 (10) |
| O4—C10 | 1.200 (4) | C22—C23 | 1.361 (10) |
| N1—C6 | 1.398 (3) | C23—C24 | 1.376 (6) |
| N1—C7 | 1.371 (3) | C2—H2 | 0.9300 |
| N1—C8 | 1.491 (3) | C3—H3 | 0.9300 |
| N2—C8 | 1.430 (4) | C5—H5 | 0.9300 |
| N2—C9 | 1.448 (4) | C8—H8A | 0.9700 |
| N2—C17 | 1.463 (4) | C8—H8B | 0.9700 |
| N3—C9 | 1.444 (3) | C9—H9A | 0.9700 |
| N3—C10 | 1.370 (4) | C9—H9B | 0.9700 |
| N3—C16 | 1.393 (3) | C12—H12 | 0.9300 |
| C1—C2 | 1.362 (3) | C13—H13 | 0.9300 |
| C1—C6 | 1.383 (4) | C15—H15 | 0.9300 |
| C2—C3 | 1.381 (4) | C17—H17A | 0.9700 |
| C3—C4 | 1.388 (4) | C17—H17B | 0.9700 |
| C4—C5 | 1.387 (3) | C18—H18A | 0.9700 |
| C5—C6 | 1.369 (4) | C18—H18B | 0.9700 |
| C11—C12 | 1.360 (4) | C20—H20 | 0.9300 |
| C11—C16 | 1.368 (4) | C21—H21 | 0.9300 |
| C12—C13 | 1.382 (5) | C22—H22 | 0.9300 |
| C13—C14 | 1.377 (5) | C23—H23 | 0.9300 |
| C14—C15 | 1.386 (4) | C24—H24 | 0.9300 |
| | | | |
| C1—O1—C7 | 107.7 (2) | C19—C20—C21 | 121.3 (4) |
| C10—O3—C11 | 107.1 (2) | C20—C21—C22 | 119.5 (5) |
| C6—N1—C7 | 109.6 (2) | C21—C22—C23 | 120.6 (4) |
| C6—N1—C8 | 128.6 (2) | C22—C23—C24 | 120.1 (6) |
| C7—N1—C8 | 121.2 (2) | C19—C24—C23 | 120.8 (4) |
| C8—N2—C9 | 112.5 (2) | C1—C2—H2 | 121.00 |
| C8—N2—C17 | 114.76 (19) | C3—C2—H2 | 121.00 |

| | | | |
|---|---|---|---|
| C9—N2—C17 | 114.5 (2) | C2—C3—H3 | 120.00 |
| C9—N3—C10 | 123.4 (2) | C4—C3—H3 | 120.00 |
| C9—N3—C16 | 127.5 (2) | C4—C5—H5 | 122.00 |
| C10—N3—C16 | 108.9 (2) | C6—C5—H5 | 122.00 |
| O1—C1—C2 | 127.6 (2) | N1—C8—H8A | 108.00 |
| O1—C1—C6 | 109.5 (2) | N1—C8—H8B | 108.00 |
| C2—C1—C6 | 122.9 (2) | N2—C8—H8A | 108.00 |
| C1—C2—C3 | 117.2 (2) | N2—C8—H8B | 108.00 |
| C2—C3—C4 | 119.5 (2) | H8A—C8—H8B | 107.00 |
| Cl1—C4—C3 | 118.03 (19) | N2—C9—H9A | 109.00 |
| Cl1—C4—C5 | 118.6 (2) | N2—C9—H9B | 109.00 |
| C3—C4—C5 | 123.3 (2) | N3—C9—H9A | 109.00 |
| C4—C5—C6 | 115.8 (2) | N3—C9—H9B | 109.00 |
| N1—C6—C1 | 105.7 (2) | H9A—C9—H9B | 108.00 |
| N1—C6—C5 | 133.2 (2) | C11—C12—H12 | 122.00 |
| C1—C6—C5 | 121.1 (2) | C13—C12—H12 | 122.00 |
| O1—C7—O2 | 123.0 (3) | C12—C13—H13 | 120.00 |
| O1—C7—N1 | 107.5 (2) | C14—C13—H13 | 120.00 |
| O2—C7—N1 | 129.6 (3) | C14—C15—H15 | 122.00 |
| N1—C8—N2 | 115.9 (2) | C16—C15—H15 | 122.00 |
| N2—C9—N3 | 111.3 (2) | N2—C17—H17A | 109.00 |
| O3—C10—O4 | 123.5 (3) | N2—C17—H17B | 109.00 |
| O3—C10—N3 | 108.3 (2) | C18—C17—H17A | 109.00 |
| O4—C10—N3 | 128.3 (3) | C18—C17—H17B | 109.00 |
| O3—C11—C12 | 127.0 (3) | H17A—C17—H17B | 108.00 |
| O3—C11—C16 | 109.4 (2) | C17—C18—H18A | 109.00 |
| C12—C11—C16 | 123.5 (3) | C17—C18—H18B | 109.00 |
| C11—C12—C13 | 115.9 (3) | C19—C18—H18A | 109.00 |
| C12—C13—C14 | 120.7 (3) | C19—C18—H18B | 109.00 |
| Cl2—C14—C13 | 118.4 (2) | H18A—C18—H18B | 108.00 |
| Cl2—C14—C15 | 118.6 (2) | C19—C20—H20 | 119.00 |
| C13—C14—C15 | 123.0 (3) | C21—C20—H20 | 119.00 |
| C14—C15—C16 | 115.1 (3) | C20—C21—H21 | 120.00 |
| N3—C16—C11 | 106.3 (3) | C22—C21—H21 | 120.00 |
| N3—C16—C15 | 132.1 (3) | C21—C22—H22 | 120.00 |
| C11—C16—C15 | 121.6 (2) | C23—C22—H22 | 120.00 |
| N2—C17—C18 | 111.8 (2) | C22—C23—H23 | 120.00 |
| C17—C18—C19 | 112.9 (2) | C24—C23—H23 | 120.00 |
| C18—C19—C20 | 121.2 (3) | C19—C24—H24 | 120.00 |
| C18—C19—C24 | 121.2 (3) | C23—C24—H24 | 120.00 |
| C20—C19—C24 | 117.7 (3) | | |

| | | | |
|---|---|---|---|
| C7—O1—C1—C2 | 176.3 (3) | O1—C1—C2—C3 | −178.1 (3) |
| C7—O1—C1—C6 | −2.3 (3) | C2—C1—C6—N1 | −178.4 (3) |
| C1—O1—C7—N1 | 3.4 (3) | C2—C1—C6—C5 | −0.7 (4) |
| C1—O1—C7—O2 | −175.8 (3) | O1—C1—C6—C5 | 178.0 (2) |
| C11—O3—C10—N3 | −1.7 (3) | C1—C2—C3—C4 | −0.6 (4) |
| C11—O3—C10—O4 | 179.4 (3) | C2—C3—C4—Cl1 | 179.3 (2) |

| | | | |
|---|---|---|---|
| C10—O3—C11—C16 | 1.8 (3) | C2—C3—C4—C5 | 1.3 (4) |
| C10—O3—C11—C12 | −178.1 (3) | C3—C4—C5—C6 | −1.6 (4) |
| C8—N1—C7—O1 | −175.2 (2) | Cl1—C4—C5—C6 | −179.6 (2) |
| C6—N1—C7—O1 | −3.3 (3) | C4—C5—C6—N1 | 178.3 (3) |
| C6—N1—C8—N2 | 99.0 (3) | C4—C5—C6—C1 | 1.3 (4) |
| C7—N1—C8—N2 | −90.7 (3) | C12—C11—C16—C15 | −0.8 (5) |
| C7—N1—C6—C1 | 1.9 (3) | C12—C11—C16—N3 | 178.7 (3) |
| C8—N1—C6—C1 | 173.1 (3) | O3—C11—C12—C13 | 179.3 (3) |
| C7—N1—C6—C5 | −175.4 (3) | C16—C11—C12—C13 | −0.6 (5) |
| C8—N1—C6—C5 | −4.3 (5) | O3—C11—C16—N3 | −1.1 (3) |
| C6—N1—C7—O2 | 175.9 (3) | O3—C11—C16—C15 | 179.4 (2) |
| C8—N1—C7—O2 | 3.9 (5) | C11—C12—C13—C14 | 1.2 (5) |
| C17—N2—C8—N1 | −70.5 (3) | C12—C13—C14—C15 | −0.6 (5) |
| C9—N2—C17—C18 | 143.5 (2) | C12—C13—C14—Cl2 | 178.7 (3) |
| C8—N2—C9—N3 | 162.6 (2) | Cl2—C14—C15—C16 | −180.0 (2) |
| C9—N2—C8—N1 | 62.8 (3) | C13—C14—C15—C16 | −0.7 (4) |
| C8—N2—C17—C18 | −84.1 (3) | C14—C15—C16—C11 | 1.4 (4) |
| C17—N2—C9—N3 | −64.0 (3) | C14—C15—C16—N3 | −178.0 (3) |
| C9—N3—C10—O3 | 175.6 (2) | N2—C17—C18—C19 | −63.6 (3) |
| C16—N3—C10—O3 | 1.1 (3) | C17—C18—C19—C20 | −74.1 (4) |
| C10—N3—C16—C11 | 0.0 (3) | C17—C18—C19—C24 | 106.1 (4) |
| C9—N3—C16—C15 | 5.2 (5) | C18—C19—C20—C21 | −178.9 (3) |
| C9—N3—C10—O4 | −5.6 (5) | C24—C19—C20—C21 | 1.0 (5) |
| C16—N3—C10—O4 | 179.9 (3) | C18—C19—C24—C23 | 179.0 (3) |
| C10—N3—C16—C15 | 179.5 (3) | C20—C19—C24—C23 | −0.9 (5) |
| C10—N3—C9—N2 | 110.9 (3) | C19—C20—C21—C22 | −1.1 (6) |
| C16—N3—C9—N2 | −75.6 (3) | C20—C21—C22—C23 | 1.1 (7) |
| C9—N3—C16—C11 | −174.2 (2) | C21—C22—C23—C24 | −1.0 (7) |
| C6—C1—C2—C3 | 0.3 (4) | C22—C23—C24—C19 | 0.9 (6) |
| O1—C1—C6—N1 | 0.3 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H8*A*···O4$^{i}$ | 0.97 | 2.51 | 3.037 (4) | 114 |
| C9—H9*A*···O4 | 0.97 | 2.56 | 2.921 (4) | 102 |
| C17—H17*A*···N3 | 0.97 | 2.54 | 2.944 (3) | 105 |

Symmetry code: (i) *x*−1, *y*, *z*.