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# Dichlorido(2-{[3-(morpholin-4-ium-4-yl)-propyl]iminomethyl}phenolate)zinc

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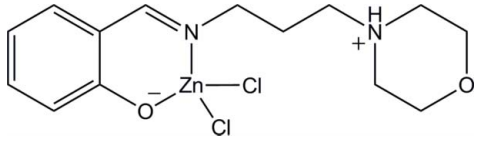
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.020; $wR$ factor = 0.051; data-to-parameter ratio = 19.8.

In the zwitterionic zinc title complex, $[ZnCl_2(C_{14}H_{20}N_2O_2)]$, the $Zn^{II}$ ion is four-coordinated in a distorted tetrahedral geometry. The Schiff base ligand employs its phenolate O and imine N atoms to coordinate the metal atom in a bidentate mode. Two Cl atoms complete the tetrahedral coordination environment. In the crystal, a pair of N—H···O hydrogen bonds connect the molecules into a centrosymmetric dimer. C—H···O, C—H···Cl and C—H···$\pi$ interactions are also observed.

## Related literature

For related structures of similar zwitterionic $ZnCl_2$ complexes, see: Qiu (2006); Ye & You (2008); Zhu (2008).



## Experimental

### Crystal data

$[ZnCl_2(C_{14}H_{20}N_2O_2)]$
$M_r$ = 384.59
Monoclinic, $P2_1/c$
$a$ = 8.11276 (10) Å
$b$ = 11.21021 (13) Å
$c$ = 18.4097 (2) Å
$\beta$ = 92.0168 (6)°
$V$ = 1673.24 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 1.79 mm$^{-1}$
$T$ = 100 K
0.37 × 0.32 × 0.25 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.557, $T_{max}$ = 0.663
14420 measured reflections
3824 independent reflections
3557 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
$S$ = 1.07
3824 reflections
193 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.33 e Å$^{-3}$

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

*Cg*1 is the centroid of the C1–C6 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2*N*···O1$^{i}$ | 0.90 (1) | 1.81 (1) | 2.6954 (14) | 170 (2) |
| C5—H5···O2$^{ii}$ | 0.95 | 2.39 | 3.2161 (16) | 146 |
| C9—H9*A*···Cl1$^{iii}$ | 0.99 | 2.83 | 3.6732 (13) | 144 |
| C10—H10*A*···Cl1$^{i}$ | 0.99 | 2.82 | 3.6905 (13) | 147 |
| C14—H14*A*···Cl2$^{iii}$ | 0.99 | 2.69 | 3.5486 (13) | 146 |
| C14—H14*B*···Cl2$^{iv}$ | 0.99 | 2.78 | 3.6890 (14) | 153 |
| C12—H12*B*···*Cg*1$^{v}$ | 0.99 | 2.57 | 3.4366 (2) | 146 |

Symmetry codes: (i) $-x, -y, -z+2$; (ii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y, -z+2$; (iv) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (v) $x, -y-\frac{1}{2}, z-\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2701).

## References

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# supporting information

*Acta Cryst.* (2011). E**67**, m932 [doi:10.1107/S1600536811022021]

# Dichlorido(2-{[3-(morpholin-4-ium-4-yl)propyl]iminomethyl}phenolate)zinc

**Nurul Azimah Ikmal Hisham, Hamid Khaledi and Hapipah Mohd Ali**

## S1. Comment

The title compound was obtained *via* the complexation of $ZnCl_2$ with the *in situ* prepared Schiff base. The Schiff base ligand coordinates the metal ion *via* its phenolate oxygen and imine nitrogen atoms. The morpholine ring N atom stays away from the coordination and is protonated, implying the zwitterionic nature of the molecule. The tetrahedral geometry around the zinc(II) ion is completed by two Cl atoms. The coordination bond lengths in the complex are comparable to the corresponding values in similar structures (Qiu, 2006; Ye & You, 2008; Zhu, 2008). In the crystal, N—H···O hydrogen bonding connects pairs of the molecules into centrosymmetric dimers. The dimers are linked through C—H···O, C—H···Cl and C—H···$\pi$ interactions into a three-dimensional network.

## S2. Experimental

A mixture of salicylaldehyde (0.20 g, 1.64 mmol) and *N*-(3-aminopropyl)morpholine (0.24 g, 1.64 mmol) in ethanol (20 ml) was refluxed for 2 hr followed by addition of a solution of zinc(II) chloride (0.22 g, 1.64 mmol) in a minimum amount of water. The resulting solution was refluxed for 30 min, then the solvent was removed under reduced pressure. The impure product was recrystallized from methanol to give the yellow crystals of the title compound.

## S3. Refinement

The C-bound H atoms were placed at calculated positions at distances C—H = 0.95 and 0.99 Å for aryl and methylene type H-atoms, respectively. The N-bound H atom was placed in a difference Fourier map, and was refined with a distance restraint of N—H 0.91 (2) Å. For all hydrogen atoms $U_{iso}$(H) were set to 1.2 times $U_{eq}$(carrier atom).

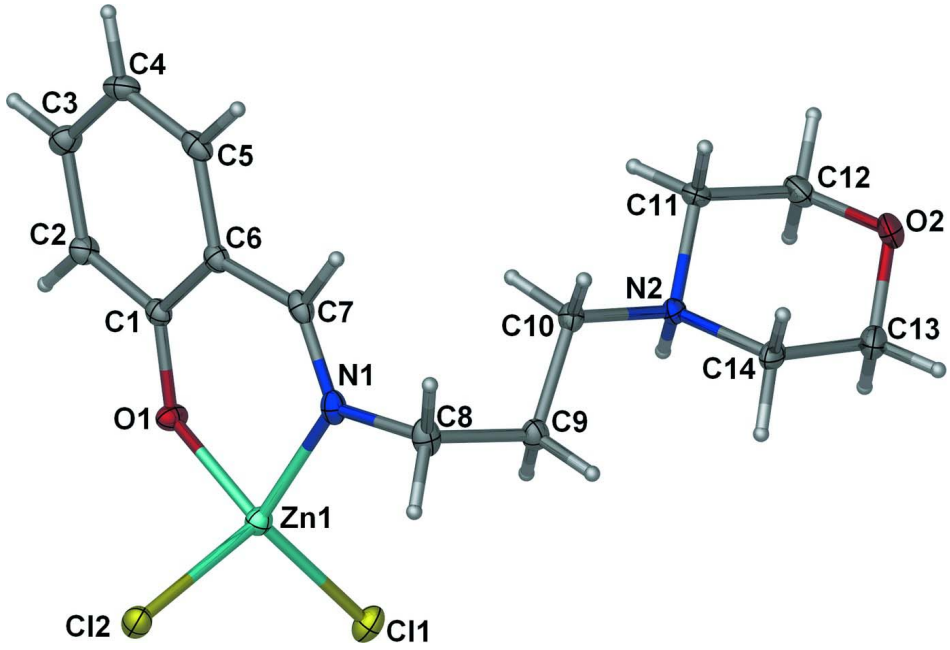


**Figure 1**

Displacement ellipsoid plot of the title compound at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

**Dichlorido(2-{[3-(morpholin-4-ium-4-yl)propyl]iminomethyl}phenolate)zinc**

*Crystal data*

$[ZnCl_2(C_{14}H_{20}N_2O_2)]$
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$c$ = 18.4097 (2) Å
$\beta$ = 92.0168 (6)°
$V$ = 1673.24 (4) Å$^3$
$Z$ = 4
$F(000)$ = 792
$D_x$ = 1.527 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9373 reflections
$\theta$ = 2.2–30.4°
$\mu$ = 1.79 mm$^{-1}$
$T$ = 100 K
Block, yellow
0.37 × 0.32 × 0.25 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.557, $T_{max}$ = 0.663
14420 measured reflections
3824 independent reflections
3557 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 2.1°
$h$ = −10→10
$k$ = −14→14
$l$ = −23→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.051$
$S = 1.07$
3824 reflections
193 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0237P)^2 + 0.6991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.34$ e Å$^{-3}$
$\Delta\rho_{min} = -0.33$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Zn1 | 0.187917 (17) | 0.093296 (13) | 1.107214 (8) | 0.01475 (5) |
| Cl1 | 0.22466 (4) | −0.09919 (3) | 1.084342 (19) | 0.02075 (8) |
| Cl2 | 0.33968 (4) | 0.15593 (3) | 1.205005 (18) | 0.01907 (7) |
| O1 | −0.04186 (11) | 0.13291 (8) | 1.12739 (5) | 0.01608 (18) |
| O2 | 0.13093 (14) | −0.02283 (10) | 0.65039 (5) | 0.0276 (2) |
| N1 | 0.20412 (13) | 0.20247 (10) | 1.02150 (6) | 0.0159 (2) |
| N2 | 0.16476 (13) | 0.05293 (10) | 0.79901 (6) | 0.0135 (2) |
| H2N | 0.1344 (19) | −0.0140 (12) | 0.8218 (8) | 0.016* |
| C1 | −0.12380 (15) | 0.22475 (11) | 1.09867 (7) | 0.0137 (2) |
| C2 | −0.28113 (16) | 0.25169 (12) | 1.12420 (7) | 0.0163 (2) |
| H2 | −0.3253 | 0.2032 | 1.1611 | 0.020* |
| C3 | −0.37327 (16) | 0.34666 (12) | 1.09714 (7) | 0.0186 (3) |
| H3 | −0.4787 | 0.3627 | 1.1158 | 0.022* |
| C4 | −0.31222 (17) | 0.41883 (12) | 1.04277 (8) | 0.0196 (3) |
| H4 | −0.3743 | 0.4849 | 1.0247 | 0.024* |
| C5 | −0.16034 (17) | 0.39287 (12) | 1.01564 (7) | 0.0173 (3) |
| H5 | −0.1203 | 0.4405 | 0.9774 | 0.021* |
| C6 | −0.06264 (15) | 0.29814 (11) | 1.04276 (7) | 0.0142 (2) |
| C7 | 0.09231 (16) | 0.28065 (11) | 1.00702 (7) | 0.0159 (2) |
| H7 | 0.1134 | 0.3336 | 0.9682 | 0.019* |
| C8 | 0.34713 (16) | 0.19638 (13) | 0.97446 (7) | 0.0194 (3) |
| H8A | 0.4473 | 0.1769 | 1.0044 | 0.023* |
| H8B | 0.3641 | 0.2753 | 0.9518 | 0.023* |
| C9 | 0.32253 (16) | 0.10212 (12) | 0.91483 (7) | 0.0170 (3) |

| | | | | |
|---|---|---|---|---|
| H9A | 0.4296 | 0.0847 | 0.8929 | 0.020* |
| H9B | 0.2812 | 0.0274 | 0.9363 | 0.020* |
| C10 | 0.20034 (15) | 0.14566 (11) | 0.85628 (7) | 0.0152 (2) |
| H10A | 0.0960 | 0.1682 | 0.8790 | 0.018* |
| H10B | 0.2452 | 0.2178 | 0.8331 | 0.018* |
| C11 | 0.02330 (16) | 0.09176 (11) | 0.74944 (7) | 0.0171 (3) |
| H11A | 0.0509 | 0.1679 | 0.7255 | 0.020* |
| H11B | −0.0763 | 0.1045 | 0.7780 | 0.020* |
| C12 | −0.01012 (18) | −0.00338 (13) | 0.69264 (7) | 0.0224 (3) |
| H12A | −0.0405 | −0.0787 | 0.7168 | 0.027* |
| H12B | −0.1042 | 0.0214 | 0.6604 | 0.027* |
| C13 | 0.26442 (19) | −0.06439 (14) | 0.69587 (8) | 0.0253 (3) |
| H13A | 0.3610 | −0.0801 | 0.6659 | 0.030* |
| H13B | 0.2328 | −0.1403 | 0.7190 | 0.030* |
| C14 | 0.31118 (16) | 0.02596 (12) | 0.75431 (7) | 0.0180 (3) |
| H14A | 0.4018 | −0.0062 | 0.7860 | 0.022* |
| H14B | 0.3505 | 0.1002 | 0.7315 | 0.022* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Zn1 | 0.01268 (8) | 0.01573 (8) | 0.01592 (9) | 0.00041 (5) | 0.00142 (6) | −0.00096 (5) |
| Cl1 | 0.01746 (15) | 0.01744 (15) | 0.02752 (17) | 0.00001 (11) | 0.00307 (13) | −0.00615 (12) |
| Cl2 | 0.01577 (14) | 0.02111 (16) | 0.02017 (16) | 0.00145 (11) | −0.00147 (11) | −0.00506 (12) |
| O1 | 0.0137 (4) | 0.0163 (4) | 0.0184 (5) | 0.0015 (3) | 0.0024 (3) | 0.0039 (4) |
| O2 | 0.0390 (6) | 0.0322 (6) | 0.0117 (5) | 0.0108 (5) | 0.0007 (4) | −0.0017 (4) |
| N1 | 0.0148 (5) | 0.0191 (5) | 0.0140 (5) | −0.0042 (4) | 0.0023 (4) | −0.0036 (4) |
| N2 | 0.0153 (5) | 0.0139 (5) | 0.0114 (5) | 0.0016 (4) | 0.0022 (4) | 0.0009 (4) |
| C1 | 0.0147 (6) | 0.0137 (6) | 0.0127 (6) | −0.0014 (4) | −0.0014 (4) | −0.0014 (4) |
| C2 | 0.0153 (6) | 0.0170 (6) | 0.0165 (6) | −0.0005 (5) | 0.0010 (5) | 0.0012 (5) |
| C3 | 0.0137 (6) | 0.0202 (6) | 0.0219 (7) | 0.0011 (5) | −0.0010 (5) | −0.0014 (5) |
| C4 | 0.0192 (6) | 0.0171 (6) | 0.0220 (7) | 0.0014 (5) | −0.0066 (5) | 0.0019 (5) |
| C5 | 0.0219 (6) | 0.0165 (6) | 0.0134 (6) | −0.0039 (5) | −0.0035 (5) | 0.0008 (5) |
| C6 | 0.0164 (6) | 0.0139 (6) | 0.0121 (6) | −0.0019 (4) | −0.0007 (5) | −0.0016 (4) |
| C7 | 0.0205 (6) | 0.0157 (6) | 0.0117 (6) | −0.0056 (5) | 0.0011 (5) | −0.0013 (5) |
| C8 | 0.0141 (6) | 0.0269 (7) | 0.0173 (6) | −0.0057 (5) | 0.0035 (5) | −0.0040 (5) |
| C9 | 0.0135 (6) | 0.0222 (7) | 0.0154 (6) | 0.0004 (5) | 0.0008 (5) | −0.0025 (5) |
| C10 | 0.0165 (6) | 0.0150 (6) | 0.0142 (6) | 0.0001 (5) | 0.0018 (5) | −0.0019 (5) |
| C11 | 0.0183 (6) | 0.0189 (6) | 0.0138 (6) | 0.0042 (5) | −0.0017 (5) | 0.0014 (5) |
| C12 | 0.0284 (7) | 0.0240 (7) | 0.0143 (6) | 0.0020 (6) | −0.0042 (5) | 0.0000 (5) |
| C13 | 0.0333 (8) | 0.0257 (7) | 0.0169 (7) | 0.0105 (6) | 0.0037 (6) | −0.0020 (5) |
| C14 | 0.0199 (6) | 0.0194 (6) | 0.0153 (6) | 0.0055 (5) | 0.0065 (5) | 0.0020 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Zn1—O1 | 1.9644 (9) | C5—H5 | 0.9500 |
| Zn1—N1 | 2.0049 (11) | C6—C7 | 1.4526 (18) |
| Zn1—Cl1 | 2.2206 (3) | C7—H7 | 0.9500 |

| | | | |
|---|---|---|---|
| Zn1—Cl2 | 2.2570 (3) | C8—C9 | 1.5318 (18) |
| O1—C1 | 1.3254 (15) | C8—H8A | 0.9900 |
| O2—C12 | 1.4230 (17) | C8—H8B | 0.9900 |
| O2—C13 | 1.4238 (18) | C9—C10 | 1.5190 (18) |
| N1—C7 | 1.2825 (17) | C9—H9A | 0.9900 |
| N1—C8 | 1.4736 (16) | C9—H9B | 0.9900 |
| N2—C14 | 1.4995 (16) | C10—H10A | 0.9900 |
| N2—C10 | 1.5012 (16) | C10—H10B | 0.9900 |
| N2—C11 | 1.5052 (16) | C11—C12 | 1.5114 (18) |
| N2—H2N | 0.898 (13) | C11—H11A | 0.9900 |
| C1—C2 | 1.4084 (17) | C11—H11B | 0.9900 |
| C1—C6 | 1.4206 (17) | C12—H12A | 0.9900 |
| C2—C3 | 1.3836 (18) | C12—H12B | 0.9900 |
| C2—H2 | 0.9500 | C13—C14 | 1.516 (2) |
| C3—C4 | 1.392 (2) | C13—H13A | 0.9900 |
| C3—H3 | 0.9500 | C13—H13B | 0.9900 |
| C4—C5 | 1.377 (2) | C14—H14A | 0.9900 |
| C4—H4 | 0.9500 | C14—H14B | 0.9900 |
| C5—C6 | 1.4062 (18) | | |

| | | | |
|---|---|---|---|
| O1—Zn1—N1 | 95.72 (4) | C9—C8—H8A | 109.3 |
| O1—Zn1—Cl1 | 112.96 (3) | N1—C8—H8B | 109.3 |
| N1—Zn1—Cl1 | 115.53 (3) | C9—C8—H8B | 109.3 |
| O1—Zn1—Cl2 | 105.87 (3) | H8A—C8—H8B | 108.0 |
| N1—Zn1—Cl2 | 112.88 (3) | C10—C9—C8 | 110.63 (11) |
| Cl1—Zn1—Cl2 | 112.363 (13) | C10—C9—H9A | 109.5 |
| C1—O1—Zn1 | 124.48 (8) | C8—C9—H9A | 109.5 |
| C12—O2—C13 | 109.77 (10) | C10—C9—H9B | 109.5 |
| C7—N1—C8 | 118.38 (11) | C8—C9—H9B | 109.5 |
| C7—N1—Zn1 | 121.01 (9) | H9A—C9—H9B | 108.1 |
| C8—N1—Zn1 | 120.60 (9) | N2—C10—C9 | 112.36 (10) |
| C14—N2—C10 | 112.86 (10) | N2—C10—H10A | 109.1 |
| C14—N2—C11 | 109.11 (10) | C9—C10—H10A | 109.1 |
| C10—N2—C11 | 110.44 (10) | N2—C10—H10B | 109.1 |
| C14—N2—H2N | 108.8 (10) | C9—C10—H10B | 109.1 |
| C10—N2—H2N | 107.5 (10) | H10A—C10—H10B | 107.9 |
| C11—N2—H2N | 108.0 (10) | N2—C11—C12 | 109.25 (10) |
| O1—C1—C2 | 118.76 (11) | N2—C11—H11A | 109.8 |
| O1—C1—C6 | 123.77 (11) | C12—C11—H11A | 109.8 |
| C2—C1—C6 | 117.47 (11) | N2—C11—H11B | 109.8 |
| C3—C2—C1 | 121.96 (12) | C12—C11—H11B | 109.8 |
| C3—C2—H2 | 119.0 | H11A—C11—H11B | 108.3 |
| C1—C2—H2 | 119.0 | O2—C12—C11 | 110.99 (12) |
| C2—C3—C4 | 120.32 (13) | O2—C12—H12A | 109.4 |
| C2—C3—H3 | 119.8 | C11—C12—H12A | 109.4 |
| C4—C3—H3 | 119.8 | O2—C12—H12B | 109.4 |
| C5—C4—C3 | 118.93 (12) | C11—C12—H12B | 109.4 |
| C5—C4—H4 | 120.5 | H12A—C12—H12B | 108.0 |

| | | | |
|---|---|---|---|
| C3—C4—H4 | 120.5 | O2—C13—C14 | 111.40 (11) |
| C4—C5—C6 | 122.09 (12) | O2—C13—H13A | 109.3 |
| C4—C5—H5 | 119.0 | C14—C13—H13A | 109.3 |
| C6—C5—H5 | 119.0 | O2—C13—H13B | 109.3 |
| C5—C6—C1 | 119.19 (12) | C14—C13—H13B | 109.3 |
| C5—C6—C7 | 115.28 (12) | H13A—C13—H13B | 108.0 |
| C1—C6—C7 | 125.44 (12) | N2—C14—C13 | 109.93 (11) |
| N1—C7—C6 | 127.96 (12) | N2—C14—H14A | 109.7 |
| N1—C7—H7 | 116.0 | C13—C14—H14A | 109.7 |
| C6—C7—H7 | 116.0 | N2—C14—H14B | 109.7 |
| N1—C8—C9 | 111.57 (10) | C13—C14—H14B | 109.7 |
| N1—C8—H8A | 109.3 | H14A—C14—H14B | 108.2 |

*Hydrogen-bond geometry (Å, º)*

*Cg*1 is the centroid of the C1–C6 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*N*···O1$^{i}$ | 0.90 (1) | 1.81 (1) | 2.6954 (14) | 170 (2) |
| C5—H5···O2$^{ii}$ | 0.95 | 2.39 | 3.2161 (16) | 146 |
| C9—H9*A*···Cl1$^{iii}$ | 0.99 | 2.83 | 3.6732 (13) | 144 |
| C10—H10*A*···Cl1$^{i}$ | 0.99 | 2.82 | 3.6905 (13) | 147 |
| C14—H14*A*···Cl2$^{iii}$ | 0.99 | 2.69 | 3.5486 (13) | 146 |
| C14—H14*B*···Cl2$^{iv}$ | 0.99 | 2.78 | 3.6890 (14) | 153 |
| C12—H12*B*···*Cg*1$^{v}$ | 0.99 | 2.57 | 3.4366 (2) | 146 |

Symmetry codes: (i) −*x*, −*y*, −*z*+2; (ii) −*x*, *y*+1/2, −*z*+3/2; (iii) −*x*+1, −*y*, −*z*+2; (iv) *x*, −*y*+1/2, *z*−1/2; (v) *x*, −*y*−1/2, *z*−3/2.