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# 1-[4-(3-{[5-(4-Chlorophenyl)furan-2-yl]-methylideneamino}-2,5-dioxoimidazolidin-1-yl)butyl]-4-methylpiperazine-1,4-diium dichloride hemihydrate

**Yan-Shu Liang,[a] Shuai Mu,[b]* Ying Liu[c] and Deng-Ke Liu[c]**

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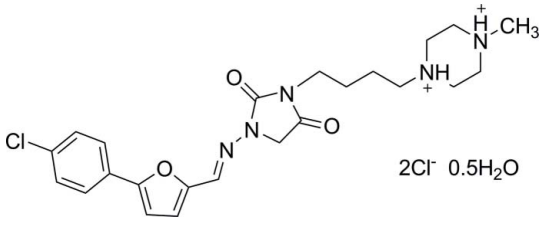
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.045; $wR$ factor = 0.123; data-to-parameter ratio = 14.4.

The title compound, $C_{23}H_{30}ClN_5O_3^{2+}\cdot 2Cl^-\cdot 0.5H_2O$, was synthesized by *N*-alkylation of 1-({[5-(4-chlorophenyl)-2-furanyl]methylene}amino)-2,4-imidazolidinedione with 1-bromo-4-chlorobutane, and *N*-methylpiperazine. In the crystal, the cations, anions and water molecules are linked by O—H···Cl and N—H···Cl hydrogen bonds.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For background to the bioactivity and applications of the title compound, see: Pratt *et al.* (2004). For the preparation of the title compound, see: Matson *et al.* (1999).




## Experimental

### Crystal data

$C_{23}H_{30}ClN_5O_3^{2+}\cdot 2Cl^-\cdot 0.5H_2O$ $M_r$ = 539.88
Monoclinic, $C2/c$
$a$ = 59.563 (12) Å
$b$ = 6.8793 (14) Å
$c$ = 12.831 (2) Å
$\beta$ = 94.402 (4)°
$V$ = 5241.9 (18) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.39 mm$^{-1}$
$T$ = 294 K
0.20 × 0.14 × 0.08 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.725, $T_{max}$ = 1.000
12667 measured reflections
4629 independent reflections
2498 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.059

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
$S$ = 1.00
4629 reflections
321 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4$B$···Cl3 | 0.86 | 2.40 | 3.250 (3) | 169 |
| O4—H4$C$···Cl3$^{i}$ | 0.85 | 2.50 | 3.250 (3) | 148 |
| N5—H5$A$···Cl2$^{ii}$ | 1.05 (4) | 1.95 (4) | 2.995 (3) | 174 (3) |
| N4—H4$A$···Cl3 | 1.04 (4) | 2.00 (4) | 3.035 (3) | 178 (3) |

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank Mr Hai-Bin Song at Nankai University for the X-ray crystallographic determination and helpful suggestions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2297).

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# supporting information

*Acta Cryst.* (2011). E**67**, o320 [doi:10.1107/S1600536811000560]

# 1-[4-(3-{[5-(4-Chlorophenyl)furan-2-yl]methylideneamino}-2,5-dioxoimidazolidin-1-yl)butyl]-4-methylpiperazine-1,4-diium dichloride hemihydrate

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## S1. Comment

The compound, 1-[[[5-(4-chlorophenyl)-2-furanyl]methylene]amino]-3- [4-(4-methyl-1-piperazinyl)butyl]-2, 4-imidazolidinedione dihydrochloride (Azimilide dihydrochloride), (I), is a Vaughan-Williamsclass III antiarrhythmic drug, which is being developed primarily for atrial fibrillation and also as adjunctive antiarrhythmic therapy in patients with implantable cardioverter defibrillators (Pratt *et al.*, 2004). Now, we present the crystal structure of the title compound (Fig. 1).

All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987) except for the N—H bonds involved in hydrogen bonds with charged donors N4—H4A (1.04 Å) and N5—H5A (1.05 Å)of piperazonia ring, and also chloride ions $Cl_2^-$ and $Cl_3^-$ as the acceptors. In the crystal, the imidazolidine-2,4-dione ring (C12/C13/C14/N2/N3/O2/O3) is planar with the r.m.s. deviation of 0.0029 Å. The dihedral angle formed between the imidazolidine-2,4-dione ring plane (A), the benzene ring plane (C1 to C6, B) and the furan ring plane (C7/C8/C9/C10/O1, C) are 28.67 (15) ° (A/B), 12.06 (3) ° (B/C) and 18.73 (18) ° (A/C), respectively. The piperazonia ring adopts a chair conformation. The packing is stabilized by intermolecular O—H···Cl and N—H···Cl hydrogen bonds (Table 1 and Fig. 2).

## S2. Experimental

The title compound was prepared according to the method of Matson *et al.* (1999). A three-neck 1 *L* flask fitted with a thermometer, mechanical stirrer, heating mantle, reflux condenser and addition funnel is charged with DMF (240 ml) and heated to 323 K. 1-[[[5-(4-Chlorophenyl)-2-furanyl]methylene]amino]-2,4- imidazolidinedione (29 g) is added and heating is continued. When dissolution is completed, potassium carbonate (14 g) is charged to the flask and heating is continued to 353 K. After 20 minutes, 1-bromo-4-chlorobutane (18.5 g) is added, and heating is continued to approximately 373 K. After 50 minutes, *N*-methylpiperazine (23.5 g) is added, and the mixture is allowed to stir for 2 h at 373 K. The reaction mixture is cooled to approximately 283 K and filtered to remove insolubles. The DMF is removed under reduced pressure at 338–341 K and replaced with absolute ethanol (175 ml). The mixture is heated to dissolve the free base and filtered to remove insolubles. The product is precipitated from ethanol (300 ml total) with the addition of 20 g of concentrated hydrochloric acid and then filtered to give 31 g of Azimilide dihydrochloride. The above dihydrochloride is suspended in 670 ml of refluxing ethanol and 150 ml of water are added to obtain complete solubilization. The mixture was standing under 298 K, then white crystals were grown slowly. The crystals were washed with cold ethanol, yield 25.6 g.

### S3. Refinement

The two water H atoms were located in a difference Fourier map and then refined as riding on the water O atom (0.85 and 0.86 Å). Other H atoms were positioned geometrically and refined using a riding model, with d(C—H) = 0.93 - 0.97 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$($C_{methyl}$).

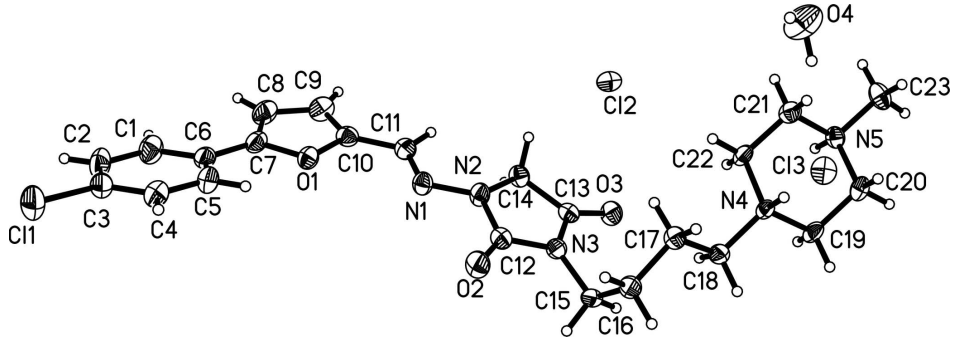


**Figure 1**

The molecular structure of (I) with the atom-numbering scheme and 30% probability displacement ellipsoids.

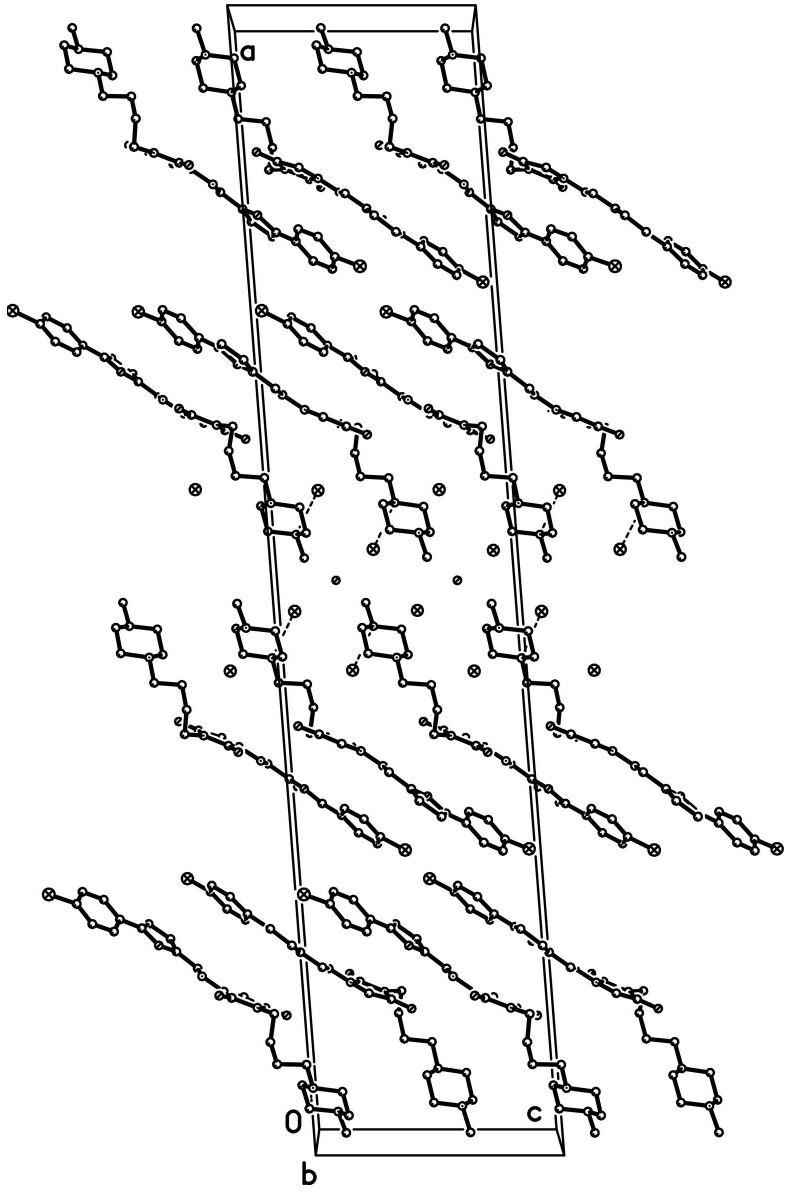


**Figure 2**

Packing diagram for (I) with hydrogen bonds drawn by dashed lines.

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*Crystal data*

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$M_r = 539.88$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 59.563\ (12)$ Å
$b = 6.8793\ (14)$ Å
$c = 12.831\ (2)$ Å
$\beta = 94.402\ (4)°$
$V = 5241.9\ (18)$ Å$^3$
$Z = 8$
$F(000) = 2264$
$D_x = 1.368$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2226 reflections
$\theta = 2.7–27.7°$
$\mu = 0.39$ mm$^{-1}$

$T$ = 294 K
Prism, white

0.20 × 0.14 × 0.08 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.725, $T_{max}$ = 1.000

12667 measured reflections
4629 independent reflections
2498 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.059
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 0.7°
$h$ = −70→70
$k$ = −3→8
$l$ = −14→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
$S$ = 1.00
4629 reflections
321 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 5.8309P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 0.23396 (2) | 0.4537 (2) | −0.45468 (9) | 0.0911 (5) | |
| O1 | 0.18447 (4) | 0.8711 (4) | −0.05974 (19) | 0.0462 (7) | |
| O2 | 0.14878 (5) | 0.4220 (4) | 0.1843 (2) | 0.0589 (8) | |
| O3 | 0.12471 (4) | 0.8338 (4) | 0.43121 (19) | 0.0515 (7) | |
| N1 | 0.15913 (5) | 0.7902 (5) | 0.1069 (2) | 0.0440 (8) | |
| N2 | 0.14652 (5) | 0.7590 (5) | 0.1904 (2) | 0.0448 (8) | |
| N3 | 0.13558 (5) | 0.5894 (4) | 0.3246 (2) | 0.0400 (8) | |
| N4 | 0.06729 (5) | 0.5843 (4) | 0.5197 (2) | 0.0337 (7) | |
| N5 | 0.04067 (5) | 0.8887 (5) | 0.6061 (2) | 0.0390 (8) | |
| C1 | 0.22367 (7) | 0.9011 (7) | −0.2680 (3) | 0.0650 (13) | |
| H1 | 0.2290 | 1.0276 | −0.2592 | 0.078* | |
| C2 | 0.23197 (7) | 0.7838 (9) | −0.3432 (3) | 0.0690 (14) | |
| H2 | 0.2430 | 0.8308 | −0.3840 | 0.083* | |

| | | | | |
|---|---|---|---|---|
| C3 | 0.22404 (7) | 0.5989 (8) | −0.3580 (3) | 0.0594 (12) |
| C4 | 0.20785 (7) | 0.5277 (7) | −0.2977 (3) | 0.0630 (13) |
| H4 | 0.2023 | 0.4022 | −0.3083 | 0.076* |
| C5 | 0.19981 (7) | 0.6438 (7) | −0.2213 (3) | 0.0573 (12) |
| H5 | 0.1890 | 0.5946 | −0.1797 | 0.069* |
| C6 | 0.20750 (6) | 0.8320 (7) | −0.2054 (3) | 0.0485 (11) |
| C7 | 0.19819 (7) | 0.9568 (6) | −0.1273 (3) | 0.0478 (11) |
| C8 | 0.19909 (7) | 1.1508 (7) | −0.1081 (3) | 0.0600 (12) |
| H8 | 0.2073 | 1.2418 | −0.1429 | 0.072* |
| C9 | 0.18542 (7) | 1.1891 (7) | −0.0263 (3) | 0.0574 (12) |
| H9 | 0.1827 | 1.3098 | 0.0030 | 0.069* |
| C10 | 0.17690 (7) | 1.0172 (6) | 0.0018 (3) | 0.0452 (10) |
| C11 | 0.16305 (6) | 0.9676 (6) | 0.0845 (3) | 0.0444 (10) |
| H11 | 0.1568 | 1.0662 | 0.1225 | 0.053* |
| C12 | 0.14429 (6) | 0.5725 (6) | 0.2266 (3) | 0.0439 (10) |
| C13 | 0.13208 (6) | 0.7804 (6) | 0.3510 (3) | 0.0394 (9) |
| C14 | 0.13935 (6) | 0.9033 (5) | 0.2631 (3) | 0.0422 (10) |
| H14A | 0.1270 | 0.9805 | 0.2321 | 0.051* |
| H14B | 0.1517 | 0.9888 | 0.2867 | 0.051* |
| C15 | 0.13341 (6) | 0.4226 (6) | 0.3940 (3) | 0.0461 (10) |
| H15A | 0.1346 | 0.4677 | 0.4658 | 0.055* |
| H15B | 0.1458 | 0.3340 | 0.3857 | 0.055* |
| C16 | 0.11156 (6) | 0.3135 (6) | 0.3737 (3) | 0.0464 (10) |
| H16A | 0.1118 | 0.2014 | 0.4197 | 0.056* |
| H16B | 0.1106 | 0.2658 | 0.3024 | 0.056* |
| C17 | 0.09061 (6) | 0.4316 (6) | 0.3897 (3) | 0.0424 (10) |
| H17A | 0.0774 | 0.3566 | 0.3660 | 0.051* |
| H17B | 0.0907 | 0.5486 | 0.3475 | 0.051* |
| C18 | 0.08908 (6) | 0.4875 (6) | 0.5033 (3) | 0.0413 (10) |
| H18A | 0.0906 | 0.3719 | 0.5464 | 0.050* |
| H18B | 0.1014 | 0.5747 | 0.5248 | 0.050* |
| C19 | 0.06381 (6) | 0.5970 (6) | 0.6343 (2) | 0.0426 (10) |
| H19A | 0.0643 | 0.4676 | 0.6644 | 0.051* |
| H19B | 0.0758 | 0.6730 | 0.6695 | 0.051* |
| C20 | 0.04159 (6) | 0.6894 (6) | 0.6511 (3) | 0.0448 (10) |
| H20A | 0.0295 | 0.6113 | 0.6179 | 0.054* |
| H20B | 0.0396 | 0.6956 | 0.7253 | 0.054* |
| C21 | 0.04333 (7) | 0.8767 (6) | 0.4918 (3) | 0.0463 (10) |
| H21A | 0.0426 | 1.0061 | 0.4617 | 0.056* |
| H21B | 0.0312 | 0.8002 | 0.4581 | 0.056* |
| C22 | 0.06551 (6) | 0.7845 (5) | 0.4732 (3) | 0.0413 (10) |
| H22A | 0.0776 | 0.8647 | 0.5041 | 0.050* |
| H22B | 0.0671 | 0.7767 | 0.3987 | 0.050* |
| C23 | 0.01982 (7) | 0.9928 (7) | 0.6300 (3) | 0.0593 (12) |
| H23A | 0.0195 | 1.1188 | 0.5977 | 0.089* |
| H23B | 0.0195 | 1.0073 | 0.7043 | 0.089* |
| H23C | 0.0069 | 0.9196 | 0.6034 | 0.089* |
| Cl2 | 0.080376 (16) | 0.89703 (15) | 0.20687 (7) | 0.0483 (3) |

| | | | | | |
|---|---|---|---|---|---|
| Cl3 | 0.027191 (17) | 0.36360 (15) | 0.41881 (7) | 0.0530 (3) | |
| H4A | 0.0538 (6) | 0.507 (5) | 0.485 (3) | 0.055 (12)* | |
| H5A | 0.0542 (7) | 0.972 (6) | 0.638 (3) | 0.069 (13)* | |
| O4 | 0.0000 | 0.6464 (6) | 0.2500 | 0.0960 (16) | |
| H4C | −0.0016 | 0.5712 | 0.1973 | 0.115* | 0.50 |
| H4B | 0.0068 | 0.5826 | 0.3011 | 0.115* | 0.50 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0934 (10) | 0.1185 (12) | 0.0647 (8) | 0.0178 (9) | 0.0280 (7) | −0.0075 (8) |
| O1 | 0.0471 (16) | 0.0461 (17) | 0.0460 (15) | −0.0059 (14) | 0.0077 (13) | 0.0043 (14) |
| O2 | 0.081 (2) | 0.0401 (17) | 0.0583 (18) | 0.0067 (16) | 0.0215 (15) | −0.0113 (15) |
| O3 | 0.0632 (18) | 0.0497 (18) | 0.0428 (15) | 0.0052 (15) | 0.0115 (14) | −0.0118 (14) |
| N1 | 0.049 (2) | 0.043 (2) | 0.0403 (18) | −0.0018 (18) | 0.0086 (16) | −0.0052 (17) |
| N2 | 0.059 (2) | 0.0347 (19) | 0.0426 (18) | 0.0017 (18) | 0.0164 (16) | −0.0054 (17) |
| N3 | 0.0466 (19) | 0.0356 (19) | 0.0383 (17) | 0.0059 (16) | 0.0072 (15) | −0.0029 (16) |
| N4 | 0.044 (2) | 0.0320 (18) | 0.0260 (15) | 0.0009 (17) | 0.0065 (14) | 0.0021 (14) |
| N5 | 0.0400 (19) | 0.037 (2) | 0.0403 (18) | −0.0005 (17) | 0.0081 (15) | −0.0068 (16) |
| C1 | 0.057 (3) | 0.077 (4) | 0.062 (3) | −0.017 (3) | 0.011 (2) | 0.005 (3) |
| C2 | 0.055 (3) | 0.099 (4) | 0.055 (3) | −0.012 (3) | 0.021 (2) | 0.003 (3) |
| C3 | 0.053 (3) | 0.078 (4) | 0.048 (3) | 0.011 (3) | 0.009 (2) | 0.003 (3) |
| C4 | 0.069 (3) | 0.065 (3) | 0.056 (3) | 0.001 (3) | 0.014 (2) | 0.010 (3) |
| C5 | 0.057 (3) | 0.066 (3) | 0.051 (3) | −0.003 (3) | 0.017 (2) | 0.007 (2) |
| C6 | 0.038 (2) | 0.063 (3) | 0.044 (2) | 0.000 (2) | 0.0021 (19) | 0.011 (2) |
| C7 | 0.045 (3) | 0.055 (3) | 0.043 (2) | −0.007 (2) | 0.002 (2) | 0.012 (2) |
| C8 | 0.063 (3) | 0.055 (3) | 0.062 (3) | −0.016 (3) | 0.004 (2) | 0.015 (3) |
| C9 | 0.063 (3) | 0.049 (3) | 0.060 (3) | −0.011 (2) | 0.000 (2) | 0.000 (2) |
| C10 | 0.045 (3) | 0.047 (3) | 0.043 (2) | 0.004 (2) | −0.0029 (19) | −0.003 (2) |
| C11 | 0.045 (2) | 0.046 (3) | 0.041 (2) | 0.000 (2) | 0.0013 (19) | −0.003 (2) |
| C12 | 0.042 (2) | 0.044 (3) | 0.046 (2) | 0.003 (2) | 0.0045 (19) | −0.002 (2) |
| C13 | 0.037 (2) | 0.039 (2) | 0.043 (2) | 0.005 (2) | 0.0008 (18) | −0.004 (2) |
| C14 | 0.046 (2) | 0.037 (2) | 0.044 (2) | 0.000 (2) | 0.0066 (18) | −0.008 (2) |
| C15 | 0.048 (3) | 0.048 (3) | 0.043 (2) | 0.018 (2) | 0.0067 (19) | 0.008 (2) |
| C16 | 0.055 (3) | 0.037 (2) | 0.049 (2) | 0.007 (2) | 0.013 (2) | −0.002 (2) |
| C17 | 0.047 (2) | 0.042 (2) | 0.039 (2) | 0.001 (2) | 0.0049 (18) | −0.0055 (19) |
| C18 | 0.043 (2) | 0.046 (2) | 0.036 (2) | 0.007 (2) | 0.0035 (18) | 0.0026 (19) |
| C19 | 0.061 (3) | 0.043 (2) | 0.0250 (18) | 0.003 (2) | 0.0079 (17) | 0.0050 (18) |
| C20 | 0.056 (3) | 0.045 (3) | 0.035 (2) | −0.007 (2) | 0.0135 (19) | −0.001 (2) |
| C21 | 0.060 (3) | 0.042 (3) | 0.037 (2) | 0.007 (2) | 0.0073 (19) | −0.001 (2) |
| C22 | 0.058 (3) | 0.033 (2) | 0.034 (2) | 0.002 (2) | 0.0128 (18) | 0.0069 (18) |
| C23 | 0.049 (3) | 0.066 (3) | 0.064 (3) | 0.008 (2) | 0.017 (2) | −0.016 (3) |
| Cl2 | 0.0508 (6) | 0.0441 (6) | 0.0489 (6) | 0.0031 (5) | −0.0043 (5) | 0.0066 (5) |
| Cl3 | 0.0545 (7) | 0.0547 (7) | 0.0496 (6) | −0.0077 (6) | 0.0021 (5) | −0.0077 (5) |
| O4 | 0.156 (5) | 0.046 (3) | 0.086 (3) | 0.000 | 0.014 (3) | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C3 | 1.732 (4) | C9—C10 | 1.347 (5) |
| O1—C7 | 1.370 (4) | C9—H9 | 0.9300 |
| O1—C10 | 1.375 (4) | C10—C11 | 1.434 (5) |
| O2—C12 | 1.208 (4) | C11—H11 | 0.9300 |
| O3—C13 | 1.207 (4) | C13—C14 | 1.499 (5) |
| N1—C11 | 1.279 (5) | C14—H14A | 0.9700 |
| N1—N2 | 1.372 (4) | C14—H14B | 0.9700 |
| N2—C12 | 1.374 (5) | C15—C16 | 1.507 (5) |
| N2—C14 | 1.449 (4) | C15—H15A | 0.9700 |
| N3—C13 | 1.376 (5) | C15—H15B | 0.9700 |
| N3—C12 | 1.402 (4) | C16—C17 | 1.516 (5) |
| N3—C15 | 1.464 (4) | C16—H16A | 0.9700 |
| N4—C18 | 1.488 (4) | C16—H16B | 0.9700 |
| N4—C22 | 1.501 (4) | C17—C18 | 1.517 (4) |
| N4—C19 | 1.504 (4) | C17—H17A | 0.9700 |
| N4—H4A | 1.04 (4) | C17—H17B | 0.9700 |
| N5—C23 | 1.486 (4) | C18—H18A | 0.9700 |
| N5—C20 | 1.487 (5) | C18—H18B | 0.9700 |
| N5—C21 | 1.489 (4) | C19—C20 | 1.498 (5) |
| N5—H5A | 1.05 (4) | C19—H19A | 0.9700 |
| C1—C2 | 1.378 (6) | C19—H19B | 0.9700 |
| C1—C6 | 1.385 (5) | C20—H20A | 0.9700 |
| C1—H1 | 0.9300 | C20—H20B | 0.9700 |
| C2—C3 | 1.365 (6) | C21—C22 | 1.501 (5) |
| C2—H2 | 0.9300 | C21—H21A | 0.9700 |
| C3—C4 | 1.372 (6) | C21—H21B | 0.9700 |
| C4—C5 | 1.378 (6) | C22—H22A | 0.9700 |
| C4—H4 | 0.9300 | C22—H22B | 0.9700 |
| C5—C6 | 1.384 (6) | C23—H23A | 0.9600 |
| C5—H5 | 0.9300 | C23—H23B | 0.9600 |
| C6—C7 | 1.460 (5) | C23—H23C | 0.9600 |
| C7—C8 | 1.357 (6) | O4—H4C | 0.8500 |
| C8—C9 | 1.402 (5) | O4—H4B | 0.8627 |
| C8—H8 | 0.9300 | | |
| | | | |
| C7—O1—C10 | 106.7 (3) | N2—C14—H14A | 111.3 |
| C11—N1—N2 | 116.5 (3) | C13—C14—H14A | 111.3 |
| N1—N2—C12 | 118.9 (3) | N2—C14—H14B | 111.3 |
| N1—N2—C14 | 127.0 (3) | C13—C14—H14B | 111.3 |
| C12—N2—C14 | 112.4 (3) | H14A—C14—H14B | 109.2 |
| C13—N3—C12 | 111.9 (3) | N3—C15—C16 | 113.7 (3) |
| C13—N3—C15 | 125.2 (3) | N3—C15—H15A | 108.8 |
| C12—N3—C15 | 122.4 (3) | C16—C15—H15A | 108.8 |
| C18—N4—C22 | 113.0 (3) | N3—C15—H15B | 108.8 |
| C18—N4—C19 | 110.6 (3) | C16—C15—H15B | 108.8 |
| C22—N4—C19 | 108.9 (3) | H15A—C15—H15B | 107.7 |

| | | | |
|---|---|---|---|
| C18—N4—H4A | 111 (2) | C15—C16—C17 | 114.7 (3) |
| C22—N4—H4A | 106 (2) | C15—C16—H16A | 108.6 |
| C19—N4—H4A | 107.1 (18) | C17—C16—H16A | 108.6 |
| C23—N5—C20 | 111.8 (3) | C15—C16—H16B | 108.6 |
| C23—N5—C21 | 112.6 (3) | C17—C16—H16B | 108.6 |
| C20—N5—C21 | 109.1 (3) | H16A—C16—H16B | 107.6 |
| C23—N5—H5A | 107 (2) | C16—C17—C18 | 112.1 (3) |
| C20—N5—H5A | 110 (2) | C16—C17—H17A | 109.2 |
| C21—N5—H5A | 106 (2) | C18—C17—H17A | 109.2 |
| C2—C1—C6 | 120.6 (5) | C16—C17—H17B | 109.2 |
| C2—C1—H1 | 119.7 | C18—C17—H17B | 109.2 |
| C6—C1—H1 | 119.7 | H17A—C17—H17B | 107.9 |
| C3—C2—C1 | 120.3 (4) | N4—C18—C17 | 111.5 (3) |
| C3—C2—H2 | 119.9 | N4—C18—H18A | 109.3 |
| C1—C2—H2 | 119.9 | C17—C18—H18A | 109.3 |
| C2—C3—C4 | 120.3 (4) | N4—C18—H18B | 109.3 |
| C2—C3—Cl1 | 120.2 (4) | C17—C18—H18B | 109.3 |
| C4—C3—Cl1 | 119.5 (4) | H18A—C18—H18B | 108.0 |
| C3—C4—C5 | 119.5 (5) | C20—C19—N4 | 110.8 (3) |
| C3—C4—H4 | 120.3 | C20—C19—H19A | 109.5 |
| C5—C4—H4 | 120.3 | N4—C19—H19A | 109.5 |
| C4—C5—C6 | 121.2 (4) | C20—C19—H19B | 109.5 |
| C4—C5—H5 | 119.4 | N4—C19—H19B | 109.5 |
| C6—C5—H5 | 119.4 | H19A—C19—H19B | 108.1 |
| C5—C6—C1 | 118.2 (4) | N5—C20—C19 | 110.0 (3) |
| C5—C6—C7 | 120.8 (4) | N5—C20—H20A | 109.7 |
| C1—C6—C7 | 120.9 (4) | C19—C20—H20A | 109.7 |
| C8—C7—O1 | 109.0 (4) | N5—C20—H20B | 109.7 |
| C8—C7—C6 | 133.7 (4) | C19—C20—H20B | 109.7 |
| O1—C7—C6 | 117.2 (4) | H20A—C20—H20B | 108.2 |
| C7—C8—C9 | 107.7 (4) | N5—C21—C22 | 109.9 (3) |
| C7—C8—H8 | 126.1 | N5—C21—H21A | 109.7 |
| C9—C8—H8 | 126.1 | C22—C21—H21A | 109.7 |
| C10—C9—C8 | 106.7 (4) | N5—C21—H21B | 109.7 |
| C10—C9—H9 | 126.7 | C22—C21—H21B | 109.7 |
| C8—C9—H9 | 126.7 | H21A—C21—H21B | 108.2 |
| C9—C10—O1 | 110.0 (3) | C21—C22—N4 | 111.2 (3) |
| C9—C10—C11 | 131.0 (4) | C21—C22—H22A | 109.4 |
| O1—C10—C11 | 118.9 (4) | N4—C22—H22A | 109.4 |
| N1—C11—C10 | 121.3 (4) | C21—C22—H22B | 109.4 |
| N1—C11—H11 | 119.4 | N4—C22—H22B | 109.4 |
| C10—C11—H11 | 119.4 | H22A—C22—H22B | 108.0 |
| O2—C12—N2 | 128.1 (4) | N5—C23—H23A | 109.5 |
| O2—C12—N3 | 125.8 (4) | N5—C23—H23B | 109.5 |
| N2—C12—N3 | 106.1 (3) | H23A—C23—H23B | 109.5 |
| O3—C13—N3 | 124.9 (4) | N5—C23—H23C | 109.5 |
| O3—C13—C14 | 127.9 (4) | H23A—C23—H23C | 109.5 |
| N3—C13—C14 | 107.2 (3) | H23B—C23—H23C | 109.5 |

| | | | |
|---|---|---|---|
| N2—C14—C13 | 102.4 (3) | H4C—O4—H4B | 108.2 |
| | | | |
| C11—N1—N2—C12 | −171.5 (4) | N1—N2—C12—N3 | 166.0 (3) |
| C11—N1—N2—C14 | −7.7 (5) | C14—N2—C12—N3 | −0.1 (4) |
| C6—C1—C2—C3 | 1.1 (7) | C13—N3—C12—O2 | −179.5 (4) |
| C1—C2—C3—C4 | −0.4 (7) | C15—N3—C12—O2 | 7.6 (6) |
| C1—C2—C3—Cl1 | 178.4 (3) | C13—N3—C12—N2 | 0.5 (4) |
| C2—C3—C4—C5 | −0.6 (7) | C15—N3—C12—N2 | −172.3 (3) |
| Cl1—C3—C4—C5 | −179.4 (3) | C12—N3—C13—O3 | −180.0 (4) |
| C3—C4—C5—C6 | 1.0 (7) | C15—N3—C13—O3 | −7.4 (6) |
| C4—C5—C6—C1 | −0.4 (6) | C12—N3—C13—C14 | −0.7 (4) |
| C4—C5—C6—C7 | 177.3 (4) | C15—N3—C13—C14 | 171.9 (3) |
| C2—C1—C6—C5 | −0.7 (6) | N1—N2—C14—C13 | −165.1 (3) |
| C2—C1—C6—C7 | −178.4 (4) | C12—N2—C14—C13 | −0.3 (4) |
| C10—O1—C7—C8 | 0.1 (4) | N3—C13—C14—N2 | 0.6 (4) |
| C10—O1—C7—C6 | −176.9 (3) | C13—N3—C15—C16 | 100.9 (4) |
| C5—C6—C7—C8 | −165.5 (5) | C12—N3—C15—C16 | −87.3 (4) |
| C1—C6—C7—C8 | 12.2 (7) | N3—C15—C16—C17 | −61.7 (4) |
| C5—C6—C7—O1 | 10.5 (6) | C15—C16—C17—C18 | −66.6 (4) |
| C1—C6—C7—O1 | −171.9 (4) | C22—N4—C18—C17 | −70.6 (4) |
| O1—C7—C8—C9 | −0.4 (5) | C19—N4—C18—C17 | 167.0 (3) |
| C6—C7—C8—C9 | 175.8 (4) | C16—C17—C18—N4 | −173.7 (3) |
| C7—C8—C9—C10 | 0.6 (5) | C18—N4—C19—C20 | −178.4 (3) |
| C8—C9—C10—O1 | −0.6 (5) | C22—N4—C19—C20 | 56.9 (4) |
| C8—C9—C10—C11 | 175.8 (4) | C23—N5—C20—C19 | −174.5 (3) |
| C7—O1—C10—C9 | 0.3 (4) | C21—N5—C20—C19 | 60.4 (4) |
| C7—O1—C10—C11 | −176.6 (3) | N4—C19—C20—N5 | −59.7 (4) |
| N2—N1—C11—C10 | 177.1 (3) | C23—N5—C21—C22 | 175.5 (3) |
| C9—C10—C11—N1 | −170.6 (4) | C20—N5—C21—C22 | −59.8 (4) |
| O1—C10—C11—N1 | 5.5 (6) | N5—C21—C22—N4 | 58.8 (4) |
| N1—N2—C12—O2 | −13.9 (6) | C18—N4—C22—C21 | −179.9 (3) |
| C14—N2—C12—O2 | 179.9 (4) | C19—N4—C22—C21 | −56.6 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4*B*···Cl3 | 0.86 | 2.40 | 3.250 (3) | 169 |
| O4—H4*C*···Cl3$^{i}$ | 0.85 | 2.50 | 3.250 (3) | 148 |
| N5—H5*A*···Cl2$^{ii}$ | 1.05 (4) | 1.95 (4) | 2.995 (3) | 174 (3) |
| N4—H4*A*···Cl3 | 1.04 (4) | 2.00 (4) | 3.035 (3) | 178 (3) |

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