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

# 2-{1-[2-((2-Ammonioethyl){2-[1-(5-chloro-2-hydroxyphenyl)ethylideneamino]ethyl}amino)ethyliminio]ethyl}-4-chlorophenolate trifluoroacetate

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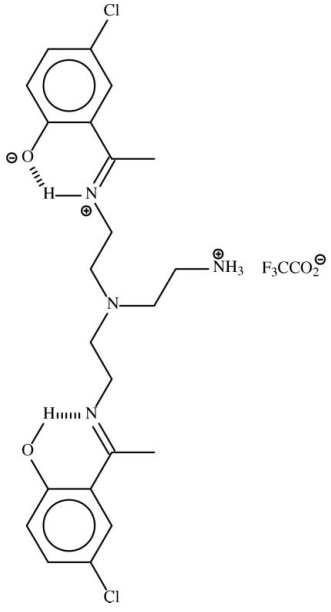
Received 20 January 2009; accepted 23 January 2009

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.048; $wR$ factor = 0.126; data-to-parameter ratio = 16.2.

In the title ion-pair, $C_{22}H_{29}Cl_2N_4O_2^+\cdot C_2F_3O_2^-$, ammonium–carboxylate N—H···O hydrogen bonds link two cations and two anions about a centre of inversion to generate a hydrogen-bonded tetramer. In the cation, one of the imino N atoms is protonated and donates a hydrogen bond to the O atom of the adjacent chlorophenyl ring. The other imino N atom acts as a hydrogen-bond acceptor from a phenolate O atom.

## Related literature

The precursor Schiff base, bis{2-[1-(5-chloro-2-hydroxyphenyl)ethyleneamino]ethyl}{2-[1-(5-chloro-2-phenolate)-ethyleneaminio]ethyl}amine, has one of the three C=N double bonds protonated on the N atom (Lee *et al.*, 2009).




## Experimental

### Crystal data

$C_{22}H_{29}Cl_2N_4O_2^+\cdot C_2F_3O_2^-$
$M_r$ = 565.41
Triclinic, $P\overline{1}$
$a$ = 10.1041 (2) Å
$b$ = 10.6788 (2) Å
$c$ = 12.5241 (3) Å
$\alpha$ = 88.386 (1)°
$\beta$ = 70.743 (1)°
$\gamma$ = 81.234 (2)°
$V$ = 1260.48 (5) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.32 mm$^{-1}$
$T$ = 100 (2) K
0.06 × 0.04 × 0.02 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.981, $T_{max}$ = 0.994
12201 measured reflections
5762 independent reflections
4104 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.126
$S$ = 1.03
5762 reflections
356 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.72 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H21···N2 | 0.85 (1) | 1.68 (2) | 2.493 (2) | 159 (4) |
| N1—H11···O1 | 0.89 (1) | 1.73 (2) | 2.520 (2) | 147 (3) |
| N4—H41···O1 | 0.88 (1) | 1.88 (1) | 2.742 (2) | 168 (2) |
| N4—H42···O3 | 0.89 (1) | 1.89 (1) | 2.769 (3) | 169 (3) |
| N4—H43···O4$^{i}$ | 0.89 (1) | 1.92 (2) | 2.755 (3) | 154 (3) |

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

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: *publCIF* (Westrip, 2009).

The authors thank the University of Malaya (grant Nos. FS339/2008A and PS072/2007C) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2856).

## References

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# supporting information

*Acta Cryst.* (2009). E**65**, o410 [doi:10.1107/S1600536809002943]

## 2-{1-[2-((2-Ammonioethyl){2-[1-(5-chloro-2-hydroxyphenyl)ethylideneamino]-ethyl}amino)ethyliminio]ethyl}-4-chlorophenolate trifluoroacetate

**See Mun Lee, Hapipah Mohd. Ali, Kong Mun Lo and Seik Weng Ng**

### S1. Experimental

Tris(2-aminoethyl)amine (1.46 g m 10 mmol) was condensed with 5-chloro-2-hydroxyacetophenone (5.12 g, 30 mol) in refluxing ethanol (100 ml) to yield the unsolvated Schiff base. The compound (0.64 g, 1 mmol) and trifluoroacetic acid (0.11 g, 1 mmol) were dissolved in a small volume of ethanol. Crystals of the salt separated after several days.

### S2. Refinement

Carbon-bound H atoms were placed in calculated positions (C—H = 0.93–0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C). The methyl H atoms were rotated to fit the electron density.

The iminium/ammonium and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints [N—H = 0.88 (1) and O—H = 0.84 (1) Å; their isotropic displacement parameters were freely refined.

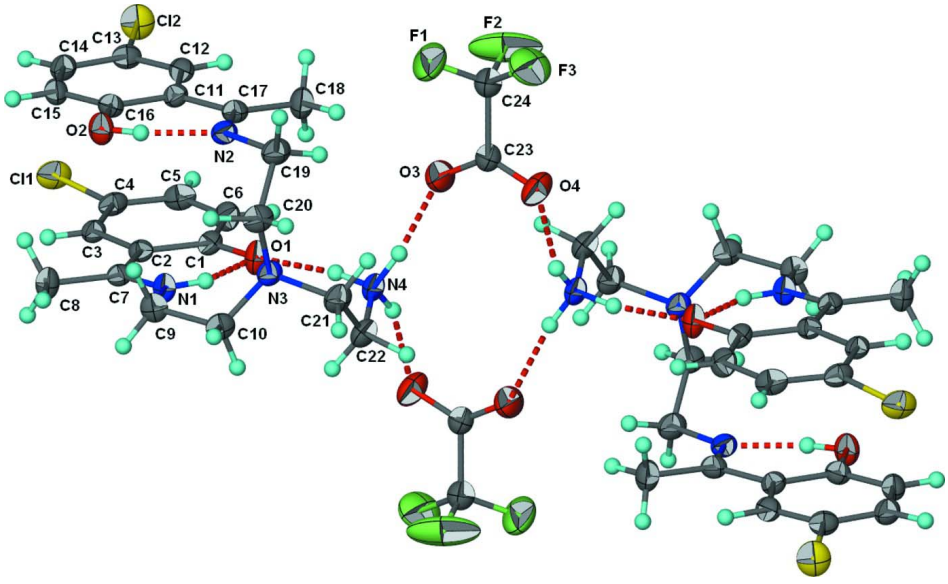


**Figure 1**

Anisotropic displacement ellipsoid plot (Barbour, 2001) of the title compound at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

**2-{1-[2-((2-Ammonioethyl){2-[1-(5-chloro-2- hydroxyphenyl)ethylideneamino]ethyl}amino)ethyliminio]ethyl}-4-chlorophenolate trifluoroacetate**

*Crystal data*

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Triclinic, $P\overline{1}$
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$a$ = 10.1041 (2) Å
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$c$ = 12.5241 (3) Å
$\alpha$ = 88.386 (1)°
$\beta$ = 70.743 (1)°
$\gamma$ = 81.234 (2)°
$V$ = 1260.48 (5) Å$^3$
$Z$ = 2
$F(000)$ = 588
$D_x$ = 1.490 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2402 reflections
$\theta$ = 2.2–27.8°
$\mu$ = 0.32 mm$^{-1}$
$T$ = 100 K
Prism, yellow
0.06 × 0.04 × 0.02 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.981, $T_{max}$ = 0.994
12201 measured reflections
5762 independent reflections
4104 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.032
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.7°
$h$ = −13→13
$k$ = −13→13
$l$ = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.126
$S$ = 1.03
5762 reflections
356 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.6261P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.72 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.10326 (7) | 0.47757 (6) | 1.36535 (5) | 0.03022 (16) |
| Cl2 | 0.46582 (7) | 0.29988 (6) | 1.26264 (5) | 0.03337 (17) |
| O1 | 0.24624 (17) | 0.42151 (15) | 0.87521 (13) | 0.0261 (4) |
| O2 | 0.22931 (18) | −0.00604 (16) | 1.01009 (14) | 0.0258 (4) |
| H21 | 0.278 (4) | 0.005 (4) | 0.9419 (14) | 0.092 (14)* |
| O3 | 0.6309 (2) | 0.30567 (19) | 0.55587 (16) | 0.0445 (5) |
| O4 | 0.7436 (2) | 0.30693 (16) | 0.36975 (14) | 0.0349 (4) |
| N1 | 0.0653 (2) | 0.27355 (18) | 0.90939 (15) | 0.0203 (4) |
| H11 | 0.135 (2) | 0.316 (2) | 0.871 (2) | 0.042 (8)* |
| N2 | 0.39560 (19) | 0.06404 (17) | 0.83106 (15) | 0.0214 (4) |

| | | | | |
|---|---|---|---|---|
| N3 | 0.23242 (19) | 0.19477 (17) | 0.68450 (15) | 0.0207 (4) |
| N4 | 0.3612 (2) | 0.4419 (2) | 0.64538 (17) | 0.0242 (4) |
| H41 | 0.332 (3) | 0.425 (2) | 0.7177 (10) | 0.027 (7)* |
| H42 | 0.4511 (14) | 0.408 (2) | 0.613 (2) | 0.044 (8)* |
| H43 | 0.353 (3) | 0.5260 (10) | 0.641 (2) | 0.038 (8)* |
| C1 | 0.2076 (2) | 0.4375 (2) | 0.98379 (18) | 0.0200 (5) |
| C2 | 0.0963 (2) | 0.37800 (19) | 1.06063 (18) | 0.0177 (4) |
| C3 | 0.0633 (2) | 0.3952 (2) | 1.17817 (18) | 0.0193 (5) |
| H3 | −0.0113 | 0.3569 | 1.2290 | 0.023* |
| C4 | 0.1376 (2) | 0.4664 (2) | 1.21953 (18) | 0.0216 (5) |
| C5 | 0.2434 (2) | 0.5277 (2) | 1.1471 (2) | 0.0247 (5) |
| H5 | 0.2925 | 0.5787 | 1.1773 | 0.030* |
| C6 | 0.2769 (2) | 0.5144 (2) | 1.0326 (2) | 0.0246 (5) |
| H6 | 0.3485 | 0.5578 | 0.9842 | 0.030* |
| C7 | 0.0240 (2) | 0.2942 (2) | 1.01801 (18) | 0.0179 (4) |
| C8 | −0.0912 (2) | 0.2298 (2) | 1.09553 (19) | 0.0250 (5) |
| H8A | −0.1262 | 0.1773 | 1.0509 | 0.037* |
| H8B | −0.1692 | 0.2938 | 1.1394 | 0.037* |
| H8C | −0.0539 | 0.1763 | 1.1471 | 0.037* |
| C9 | 0.0156 (2) | 0.1860 (2) | 0.84896 (19) | 0.0227 (5) |
| H9A | −0.0893 | 0.2016 | 0.8730 | 0.027* |
| H9B | 0.0458 | 0.0976 | 0.8668 | 0.027* |
| C10 | 0.0773 (2) | 0.2056 (2) | 0.72269 (19) | 0.0231 (5) |
| H10A | 0.0499 | 0.1418 | 0.6814 | 0.028* |
| H10B | 0.0368 | 0.2906 | 0.7044 | 0.028* |
| C11 | 0.3915 (2) | 0.1380 (2) | 1.00730 (18) | 0.0193 (5) |
| C12 | 0.4442 (2) | 0.2098 (2) | 1.07147 (19) | 0.0214 (5) |
| H12 | 0.5183 | 0.2569 | 1.0342 | 0.026* |
| C13 | 0.3905 (2) | 0.2132 (2) | 1.18738 (19) | 0.0222 (5) |
| C14 | 0.2819 (2) | 0.1461 (2) | 1.24520 (19) | 0.0237 (5) |
| H14 | 0.2442 | 0.1503 | 1.3255 | 0.028* |
| C15 | 0.2300 (2) | 0.0732 (2) | 1.18385 (19) | 0.0224 (5) |
| H15 | 0.1565 | 0.0261 | 1.2227 | 0.027* |
| C16 | 0.2827 (2) | 0.0670 (2) | 1.06586 (19) | 0.0208 (5) |
| C17 | 0.4476 (2) | 0.1367 (2) | 0.88267 (19) | 0.0214 (5) |
| C18 | 0.5569 (3) | 0.2198 (2) | 0.8265 (2) | 0.0273 (5) |
| H18A | 0.5891 | 0.2051 | 0.7444 | 0.041* |
| H18B | 0.6377 | 0.1995 | 0.8540 | 0.041* |
| H18C | 0.5152 | 0.3089 | 0.8443 | 0.041* |
| C19 | 0.4354 (3) | 0.0499 (2) | 0.70858 (19) | 0.0259 (5) |
| H19A | 0.4932 | −0.0341 | 0.6833 | 0.031* |
| H19B | 0.4927 | 0.1159 | 0.6711 | 0.031* |
| C20 | 0.3015 (2) | 0.0623 (2) | 0.6765 (2) | 0.0247 (5) |
| H20A | 0.3258 | 0.0295 | 0.5981 | 0.030* |
| H20B | 0.2347 | 0.0104 | 0.7273 | 0.030* |
| C21 | 0.2872 (3) | 0.2563 (2) | 0.57570 (19) | 0.0266 (5) |
| H21A | 0.2368 | 0.2340 | 0.5249 | 0.032* |
| H21B | 0.3889 | 0.2223 | 0.5404 | 0.032* |

| | | | | |
|---|---|---|---|---|
| C22 | 0.2712 (3) | 0.3986 (2) | 0.5850 (2) | 0.0274 (5) |
| H22A | 0.2968 | 0.4336 | 0.5081 | 0.033* |
| H22B | 0.1706 | 0.4325 | 0.6256 | 0.033* |
| C23 | 0.7267 (2) | 0.2710 (2) | 0.4670 (2) | 0.0245 (5) |
| C24 | 0.8459 (3) | 0.1700 (2) | 0.4815 (2) | 0.0281 (5) |
| F1 | 0.79561 (19) | 0.07794 (17) | 0.54812 (16) | 0.0644 (6) |
| F2 | 0.9303 (2) | 0.21998 (18) | 0.5210 (2) | 0.0813 (8) |
| F3 | 0.92729 (18) | 0.11073 (17) | 0.38502 (15) | 0.0550 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0342 (3) | 0.0384 (4) | 0.0199 (3) | 0.0023 (3) | −0.0144 (3) | −0.0062 (2) |
| Cl2 | 0.0386 (4) | 0.0377 (4) | 0.0288 (3) | −0.0110 (3) | −0.0150 (3) | −0.0050 (3) |
| O1 | 0.0300 (9) | 0.0305 (9) | 0.0170 (8) | −0.0130 (7) | −0.0031 (7) | 0.0024 (7) |
| O2 | 0.0267 (9) | 0.0292 (9) | 0.0234 (9) | −0.0125 (7) | −0.0069 (8) | 0.0015 (7) |
| O3 | 0.0322 (10) | 0.0547 (13) | 0.0297 (10) | 0.0114 (9) | 0.0037 (8) | 0.0106 (9) |
| O4 | 0.0475 (11) | 0.0331 (10) | 0.0223 (9) | −0.0028 (8) | −0.0111 (8) | 0.0062 (7) |
| N1 | 0.0228 (10) | 0.0221 (10) | 0.0169 (9) | −0.0087 (8) | −0.0054 (8) | −0.0002 (7) |
| N2 | 0.0216 (10) | 0.0239 (10) | 0.0186 (9) | −0.0002 (8) | −0.0078 (8) | 0.0011 (8) |
| N3 | 0.0235 (10) | 0.0230 (10) | 0.0149 (9) | −0.0012 (8) | −0.0065 (8) | −0.0002 (7) |
| N4 | 0.0251 (11) | 0.0292 (12) | 0.0153 (10) | −0.0029 (9) | −0.0034 (9) | 0.0054 (8) |
| C1 | 0.0233 (11) | 0.0173 (11) | 0.0197 (11) | −0.0029 (9) | −0.0076 (9) | 0.0027 (8) |
| C2 | 0.0183 (11) | 0.0156 (10) | 0.0199 (11) | −0.0020 (8) | −0.0075 (9) | 0.0006 (8) |
| C3 | 0.0202 (11) | 0.0190 (11) | 0.0165 (11) | 0.0015 (9) | −0.0050 (9) | 0.0007 (8) |
| C4 | 0.0269 (12) | 0.0216 (11) | 0.0174 (11) | 0.0016 (9) | −0.0110 (10) | −0.0022 (9) |
| C5 | 0.0286 (12) | 0.0202 (12) | 0.0312 (13) | −0.0046 (9) | −0.0170 (11) | −0.0014 (9) |
| C6 | 0.0268 (12) | 0.0207 (12) | 0.0282 (13) | −0.0095 (9) | −0.0094 (10) | 0.0035 (9) |
| C7 | 0.0191 (11) | 0.0159 (10) | 0.0186 (11) | −0.0005 (8) | −0.0071 (9) | 0.0003 (8) |
| C8 | 0.0282 (12) | 0.0281 (13) | 0.0196 (11) | −0.0121 (10) | −0.0058 (10) | 0.0021 (9) |
| C9 | 0.0248 (12) | 0.0242 (12) | 0.0215 (11) | −0.0074 (9) | −0.0088 (10) | −0.0031 (9) |
| C10 | 0.0271 (12) | 0.0241 (12) | 0.0203 (11) | −0.0028 (9) | −0.0110 (10) | −0.0021 (9) |
| C11 | 0.0205 (11) | 0.0179 (11) | 0.0191 (11) | −0.0013 (9) | −0.0067 (9) | 0.0018 (8) |
| C12 | 0.0200 (11) | 0.0190 (11) | 0.0255 (12) | −0.0039 (9) | −0.0075 (10) | 0.0032 (9) |
| C13 | 0.0236 (12) | 0.0211 (12) | 0.0237 (12) | −0.0011 (9) | −0.0108 (10) | −0.0017 (9) |
| C14 | 0.0245 (12) | 0.0256 (12) | 0.0179 (11) | 0.0009 (9) | −0.0050 (9) | 0.0011 (9) |
| C15 | 0.0200 (11) | 0.0212 (11) | 0.0231 (12) | −0.0034 (9) | −0.0033 (9) | 0.0034 (9) |
| C16 | 0.0179 (11) | 0.0207 (11) | 0.0234 (12) | −0.0015 (9) | −0.0069 (9) | 0.0012 (9) |
| C17 | 0.0192 (11) | 0.0199 (11) | 0.0251 (12) | −0.0013 (9) | −0.0083 (10) | 0.0029 (9) |
| C18 | 0.0277 (13) | 0.0311 (13) | 0.0216 (12) | −0.0091 (10) | −0.0046 (10) | 0.0049 (10) |
| C19 | 0.0283 (13) | 0.0291 (13) | 0.0182 (11) | 0.0023 (10) | −0.0075 (10) | −0.0010 (9) |
| C20 | 0.0302 (13) | 0.0238 (12) | 0.0209 (12) | −0.0009 (10) | −0.0106 (10) | −0.0035 (9) |
| C21 | 0.0310 (13) | 0.0331 (13) | 0.0150 (11) | −0.0018 (10) | −0.0078 (10) | 0.0008 (9) |
| C22 | 0.0313 (13) | 0.0306 (13) | 0.0209 (12) | −0.0048 (10) | −0.0097 (10) | 0.0075 (10) |
| C23 | 0.0244 (12) | 0.0243 (12) | 0.0239 (12) | −0.0046 (9) | −0.0069 (10) | 0.0038 (9) |
| C24 | 0.0271 (13) | 0.0301 (13) | 0.0257 (13) | −0.0042 (10) | −0.0072 (11) | 0.0029 (10) |
| F1 | 0.0543 (11) | 0.0495 (11) | 0.0659 (13) | 0.0096 (9) | 0.0014 (10) | 0.0328 (10) |
| F2 | 0.0898 (15) | 0.0505 (12) | 0.143 (2) | 0.0103 (10) | −0.0980 (16) | −0.0211 (12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| F3 | 0.0445 (10) | 0.0601 (12) | 0.0440 (10) | 0.0166 (8) | −0.0026 (8) | −0.0095 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.747 (2) | C9—C10 | 1.518 (3) |
| Cl2—C13 | 1.749 (2) | C9—H9A | 0.9900 |
| O1—C1 | 1.294 (3) | C9—H9B | 0.9900 |
| O2—C16 | 1.339 (3) | C10—H10A | 0.9900 |
| O2—H21 | 0.847 (10) | C10—H10B | 0.9900 |
| O3—C23 | 1.231 (3) | C11—C12 | 1.400 (3) |
| O4—C23 | 1.231 (3) | C11—C16 | 1.419 (3) |
| N1—C7 | 1.299 (3) | C11—C17 | 1.474 (3) |
| N1—C9 | 1.460 (3) | C12—C13 | 1.371 (3) |
| N1—H11 | 0.889 (10) | C12—H12 | 0.9500 |
| N2—C17 | 1.293 (3) | C13—C14 | 1.389 (3) |
| N2—C19 | 1.458 (3) | C14—C15 | 1.377 (3) |
| N3—C10 | 1.467 (3) | C14—H14 | 0.9500 |
| N3—C20 | 1.469 (3) | C15—C16 | 1.396 (3) |
| N3—C21 | 1.469 (3) | C15—H15 | 0.9500 |
| N4—C22 | 1.487 (3) | C17—C18 | 1.501 (3) |
| N4—H41 | 0.878 (10) | C18—H18A | 0.9800 |
| N4—H42 | 0.886 (10) | C18—H18B | 0.9800 |
| N4—H43 | 0.892 (10) | C18—H18C | 0.9800 |
| C1—C6 | 1.423 (3) | C19—C20 | 1.520 (3) |
| C1—C2 | 1.436 (3) | C19—H19A | 0.9900 |
| C2—C3 | 1.409 (3) | C19—H19B | 0.9900 |
| C2—C7 | 1.450 (3) | C20—H20A | 0.9900 |
| C3—C4 | 1.365 (3) | C20—H20B | 0.9900 |
| C3—H3 | 0.9500 | C21—C22 | 1.507 (3) |
| C4—C5 | 1.391 (3) | C21—H21A | 0.9900 |
| C5—C6 | 1.367 (3) | C21—H21B | 0.9900 |
| C5—H5 | 0.9500 | C22—H22A | 0.9900 |
| C6—H6 | 0.9500 | C22—H22B | 0.9900 |
| C7—C8 | 1.494 (3) | C23—C24 | 1.545 (3) |
| C8—H8A | 0.9800 | C24—F2 | 1.299 (3) |
| C8—H8B | 0.9800 | C24—F1 | 1.322 (3) |
| C8—H8C | 0.9800 | C24—F3 | 1.329 (3) |
| | | | |
| C16—O2—H21 | 102 (3) | C13—C12—H12 | 119.5 |
| C7—N1—C9 | 127.14 (19) | C11—C12—H12 | 119.5 |
| C7—N1—H11 | 113.5 (19) | C12—C13—C14 | 121.3 (2) |
| C9—N1—H11 | 119.3 (19) | C12—C13—Cl2 | 118.69 (18) |
| C17—N2—C19 | 124.9 (2) | C14—C13—Cl2 | 119.96 (18) |
| C10—N3—C20 | 112.17 (18) | C15—C14—C13 | 118.7 (2) |
| C10—N3—C21 | 111.60 (18) | C15—C14—H14 | 120.7 |
| C20—N3—C21 | 109.93 (18) | C13—C14—H14 | 120.7 |
| C22—N4—H41 | 112.4 (17) | C14—C15—C16 | 121.5 (2) |
| C22—N4—H42 | 110.4 (19) | C14—C15—H15 | 119.2 |

| | | | |
|---|---|---|---|
| H41—N4—H42 | 110 (3) | C16—C15—H15 | 119.2 |
| C22—N4—H43 | 107.7 (18) | O2—C16—C15 | 119.2 (2) |
| H41—N4—H43 | 106 (2) | O2—C16—C11 | 121.3 (2) |
| H42—N4—H43 | 109 (3) | C15—C16—C11 | 119.5 (2) |
| O1—C1—C6 | 120.6 (2) | N2—C17—C11 | 116.1 (2) |
| O1—C1—C2 | 122.6 (2) | N2—C17—C18 | 125.6 (2) |
| C6—C1—C2 | 116.8 (2) | C11—C17—C18 | 118.3 (2) |
| C3—C2—C1 | 119.7 (2) | C17—C18—H18A | 109.5 |
| C3—C2—C7 | 119.8 (2) | C17—C18—H18B | 109.5 |
| C1—C2—C7 | 120.32 (19) | H18A—C18—H18B | 109.5 |
| C4—C3—C2 | 120.5 (2) | C17—C18—H18C | 109.5 |
| C4—C3—H3 | 119.8 | H18A—C18—H18C | 109.5 |
| C2—C3—H3 | 119.8 | H18B—C18—H18C | 109.5 |
| C3—C4—C5 | 121.0 (2) | N2—C19—C20 | 108.94 (19) |
| C3—C4—Cl1 | 119.76 (18) | N2—C19—H19A | 109.9 |
| C5—C4—Cl1 | 119.19 (17) | C20—C19—H19A | 109.9 |
| C6—C5—C4 | 120.0 (2) | N2—C19—H19B | 109.9 |
| C6—C5—H5 | 120.0 | C20—C19—H19B | 109.9 |
| C4—C5—H5 | 120.0 | H19A—C19—H19B | 108.3 |
| C5—C6—C1 | 121.9 (2) | N3—C20—C19 | 111.49 (19) |
| C5—C6—H6 | 119.0 | N3—C20—H20A | 109.3 |
| C1—C6—H6 | 119.0 | C19—C20—H20A | 109.3 |
| N1—C7—C2 | 117.78 (19) | N3—C20—H20B | 109.3 |
| N1—C7—C8 | 120.4 (2) | C19—C20—H20B | 109.3 |
| C2—C7—C8 | 121.75 (19) | H20A—C20—H20B | 108.0 |
| C7—C8—H8A | 109.5 | N3—C21—C22 | 114.00 (19) |
| C7—C8—H8B | 109.5 | N3—C21—H21A | 108.8 |
| H8A—C8—H8B | 109.5 | C22—C21—H21A | 108.8 |
| C7—C8—H8C | 109.5 | N3—C21—H21B | 108.8 |
| H8A—C8—H8C | 109.5 | C22—C21—H21B | 108.8 |
| H8B—C8—H8C | 109.5 | H21A—C21—H21B | 107.6 |
| N1—C9—C10 | 109.14 (18) | N4—C22—C21 | 112.88 (19) |
| N1—C9—H9A | 109.9 | N4—C22—H22A | 109.0 |
| C10—C9—H9A | 109.9 | C21—C22—H22A | 109.0 |
| N1—C9—H9B | 109.9 | N4—C22—H22B | 109.0 |
| C10—C9—H9B | 109.9 | C21—C22—H22B | 109.0 |
| H9A—C9—H9B | 108.3 | H22A—C22—H22B | 107.8 |
| N3—C10—C9 | 112.01 (18) | O3—C23—O4 | 130.0 (2) |
| N3—C10—H10A | 109.2 | O3—C23—C24 | 114.3 (2) |
| C9—C10—H10A | 109.2 | O4—C23—C24 | 115.6 (2) |
| N3—C10—H10B | 109.2 | F2—C24—F1 | 109.2 (2) |
| C9—C10—H10B | 109.2 | F2—C24—F3 | 106.2 (2) |
| H10A—C10—H10B | 107.9 | F1—C24—F3 | 104.2 (2) |
| C12—C11—C16 | 118.0 (2) | F2—C24—C23 | 111.5 (2) |
| C12—C11—C17 | 120.8 (2) | F1—C24—C23 | 112.3 (2) |
| C16—C11—C17 | 121.2 (2) | F3—C24—C23 | 113.1 (2) |
| C13—C12—C11 | 121.0 (2) | | |

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|---|---|---|---|
| O1—C1—C2—C3 | 177.41 (19) | Cl2—C13—C14—C15 | −176.52 (17) |
| C6—C1—C2—C3 | −1.5 (3) | C13—C14—C15—C16 | −0.8 (3) |
| O1—C1—C2—C7 | 1.2 (3) | C14—C15—C16—O2 | 179.5 (2) |
| C6—C1—C2—C7 | −177.62 (19) | C14—C15—C16—C11 | −0.6 (3) |
| C1—C2—C3—C4 | −1.0 (3) | C12—C11—C16—O2 | −178.6 (2) |
| C7—C2—C3—C4 | 175.23 (19) | C17—C11—C16—O2 | 1.6 (3) |
| C2—C3—C4—C5 | 2.6 (3) | C12—C11—C16—C15 | 1.5 (3) |
| C2—C3—C4—Cl1 | −175.57 (16) | C17—C11—C16—C15 | −178.3 (2) |
| C3—C4—C5—C6 | −1.7 (3) | C19—N2—C17—C11 | 179.43 (19) |
| Cl1—C4—C5—C6 | 176.49 (18) | C19—N2—C17—C18 | 0.4 (4) |
| C4—C5—C6—C1 | −0.9 (4) | C12—C11—C17—N2 | 177.7 (2) |
| O1—C1—C6—C5 | −176.5 (2) | C16—C11—C17—N2 | −2.5 (3) |
| C2—C1—C6—C5 | 2.4 (3) | C12—C11—C17—C18 | −3.2 (3) |
| C9—N1—C7—C2 | 175.0 (2) | C16—C11—C17—C18 | 176.6 (2) |
| C9—N1—C7—C8 | −3.1 (3) | C17—N2—C19—C20 | −131.8 (2) |
| C3—C2—C7—N1 | −174.86 (19) | C10—N3—C20—C19 | −145.03 (19) |
| C1—C2—C7—N1 | 1.3 (3) | C21—N3—C20—C19 | 90.2 (2) |
| C3—C2—C7—C8 | 3.3 (3) | N2—C19—C20—N3 | 74.7 (2) |
| C1—C2—C7—C8 | 179.4 (2) | C10—N3—C21—C22 | 78.2 (2) |
| C7—N1—C9—C10 | 172.3 (2) | C20—N3—C21—C22 | −156.73 (19) |
| C20—N3—C10—C9 | 76.3 (2) | N3—C21—C22—N4 | 67.2 (3) |
| C21—N3—C10—C9 | −159.79 (18) | O3—C23—C24—F2 | −76.8 (3) |
| N1—C9—C10—N3 | 54.4 (2) | O4—C23—C24—F2 | 101.3 (3) |
| C16—C11—C12—C13 | −1.1 (3) | O3—C23—C24—F1 | 46.1 (3) |
| C17—C11—C12—C13 | 178.72 (19) | O4—C23—C24—F1 | −135.8 (2) |
| C11—C12—C13—C14 | −0.2 (3) | O3—C23—C24—F3 | 163.6 (2) |
| C11—C12—C13—Cl2 | 177.53 (17) | O4—C23—C24—F3 | −18.3 (3) |
| C12—C13—C14—C15 | 1.2 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H21···N2 | 0.85 (1) | 1.68 (2) | 2.493 (2) | 159 (4) |
| N1—H11···O1 | 0.89 (1) | 1.73 (2) | 2.520 (2) | 147 (3) |
| N4—H41···O1 | 0.88 (1) | 1.88 (1) | 2.742 (2) | 168 (2) |
| N4—H42···O3 | 0.89 (1) | 1.89 (1) | 2.769 (3) | 169 (3) |
| N4—H43···O4$^{i}$ | 0.89 (1) | 1.92 (2) | 2.755 (3) | 154 (3) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.