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3a,11b-Dihydroxy-2-oxo-2,3,3a,11b-tetrahydro-1*H*-imidazo[4,5-*f*][1,10]-phenanthrolin-7-ium chloride

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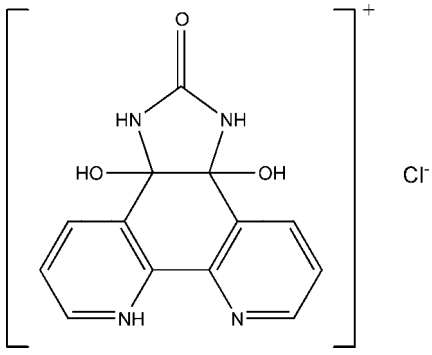
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.103; data-to-parameter ratio = 11.9.

In the crystal structure of the title compound, $C_{13}H_{11}N_4O_3^+\cdot Cl^-$, the dihedral angle between the two pyridine rings is 9.72 (9) Å. Ions are linked *via* N—H···Cl, O—H···Cl and O—H···O hydrogen bonds, forming a three-dimensional framework.

Related literature

For general background, see: Zhao *et al.* (2004); Zheng *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{11}N_4O_3^+\cdot Cl^-$
M_r = 306.71
Monoclinic, $P2_1/c$
a = 7.9420 (13) Å
b = 20.352 (3) Å
c = 8.2972 (14) Å
β = 106.620 (5)°
V = 1285.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 293 (2) K
0.31 × 0.22 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.909, T_{max} = 0.943
13480 measured reflections
2261 independent reflections
2094 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.103
S = 1.05
2261 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1^{i}	0.86	2.41	3.1512 (17)	145
N3—H3A···O2^{ii}	0.86	2.65	3.146 (2)	118
N4—H4···Cl1^{iii}	0.86	2.50	3.2490 (16)	147
O2—H2A···Cl1	0.82	2.28	3.0712 (15)	163
O3—H3B···O1^{iv}	0.82	1.89	2.6867 (18)	165

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2573).

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Zheng, L. M., Zhu, J. N., Zhang, Y. Q., Tao, Z., Xue, S. F., Zhu, Q. J., Wei, Z. B. & Long, L. S. (2005). *Chin. J. Inorg. Chem.* **21**, 1583–1588.

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3a,11b-Dihydroxy-2-oxo-2,3,3a,11b-tetrahydro-1*H*-imidazo[4,5-*f*][1,10]phenanthrolin-7-ium chloride

Ying Huang, Ming-Hua Chen, Yun-Qian Zhang, Sai-Feng Xue and Zhu Tao

S1. Comment

Recent year, we used different alkyl-substituted glycolurils as the building blocks to synthesize the partially alkyl substituted cucurbit[*n*]urils (Zhao *et al.*, 2004; Zheng *et al.*, 2005). In this work, we further report the crystal structure of a phenanthroline-substituted semi-glycoluril.

In the title compound (I), (Fig. 1), consists of organic cations, Cl^- anions. The dihedral angle between two pyridine rings is 9.72 (9) Å. Molecules are linked *via* N—H···Cl, O—H···Cl and O—H···O hydrogen bonds forming a three-dimensional framework. (Table 1).

S2. Experimental

1,10-Phenanthroline-5,6-dione (3.00 g,14.29 mmol) and carbamide (15.00 g, 250 mmol) were dissolved in acetic acid glacial (120 mL) and hydrochloric acid (5 mL) at room temperature. There was a lot of deposit after the mixture were stirred 5 h. Filtrate, solid was washed by ethanol, drying, gained white powder 2.46 g [yield: 63%].

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.93, N—H = 0.86 and O—H = 0.82 Å and refined as riding, with U_{iso}(H) = 1.2-1.5U_{eq}.

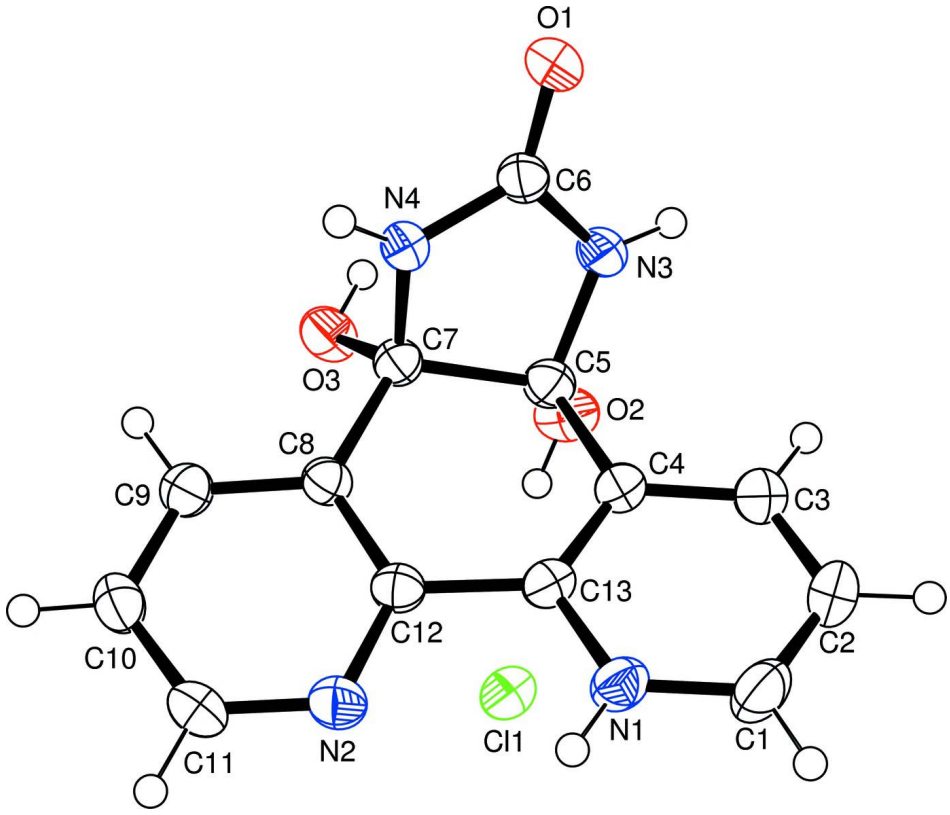

Figure 1

The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

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$b = 20.352\ (3)$ Å
$c = 8.2972\ (14)$ Å
$\beta = 106.620\ (5)°$
$V = 1285.1\ (4)$ Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.585$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2261 reflections
$\theta = 2.0–25.0°$
$\mu = 0.31$ mm^{-1}
$T = 293$ K
Prism, colourless
0.31 × 0.22 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
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13480 measured reflections
2261 independent reflections
2094 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -8\rightarrow9$
$k = -24\rightarrow24$
$l = -9\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.05$
2261 reflections
190 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
$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.7326P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ 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}
C1	−0.0113 (3)	0.58542 (11)	0.2547 (3)	0.0409 (5)
H1	−0.0891	0.5658	0.1616	0.049*
C2	−0.0166 (3)	0.65219 (11)	0.2779 (3)	0.0446 (5)
H2	−0.0982	0.6781	0.2015	0.054*
C3	0.1010 (2)	0.68003 (10)	0.4159 (3)	0.0376 (5)
H3	0.0960	0.7249	0.4352	0.045*
C4	0.2275 (2)	0.64158 (9)	0.5271 (2)	0.0280 (4)
C5	0.3591 (2)	0.67304 (8)	0.6779 (2)	0.0288 (4)
C6	0.6083 (2)	0.70941 (8)	0.6106 (2)	0.0286 (4)
C7	0.5217 (2)	0.62729 (8)	0.7653 (2)	0.0265 (4)
C8	0.4878 (2)	0.55393 (8)	0.7408 (2)	0.0250 (4)
C9	0.6001 (2)	0.50849 (9)	0.8421 (2)	0.0311 (4)
H9	0.6963	0.5226	0.9282	0.037*
C10	0.5678 (2)	0.44209 (9)	0.8141 (2)	0.0325 (4)
H10	0.6423	0.4111	0.8805	0.039*
C11	0.4231 (3)	0.42256 (9)	0.6860 (2)	0.0327 (4)
H11	0.4009	0.3778	0.6693	0.039*
C12	0.3479 (2)	0.52914 (8)	0.6134 (2)	0.0258 (4)
C13	0.2261 (2)	0.57459 (8)	0.5007 (2)	0.0261 (4)
N1	0.10578 (19)	0.54886 (8)	0.36612 (19)	0.0314 (4)
H1A	0.1046	0.5070	0.3516	0.038*
N2	0.3139 (2)	0.46487 (7)	0.58529 (19)	0.0315 (4)
N3	0.44790 (19)	0.72802 (7)	0.6258 (2)	0.0337 (4)
H3A	0.4054	0.7671	0.6071	0.040*
N4	0.64752 (19)	0.64940 (7)	0.68046 (18)	0.0284 (3)

H4	0.7373	0.6268	0.6752	0.034*
O1	0.70045 (17)	0.74179 (6)	0.54285 (17)	0.0374 (3)
O2	0.26989 (17)	0.69570 (7)	0.79061 (17)	0.0389 (3)
H2A	0.2216	0.6647	0.8224	0.058*
O3	0.58135 (18)	0.63742 (6)	0.93855 (15)	0.0355 (3)
H3B	0.6013	0.6766	0.9577	0.053*
Cl1	0.03122 (6)	0.58193 (2)	0.83116 (6)	0.03931 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0294 (10)	0.0529 (13)	0.0332 (10)	−0.0040 (9)	−0.0026 (8)	0.0010 (9)
C2	0.0330 (10)	0.0466 (12)	0.0450 (12)	0.0029 (9)	−0.0037 (9)	0.0121 (10)
C3	0.0304 (10)	0.0314 (10)	0.0473 (12)	0.0010 (8)	0.0053 (8)	0.0062 (8)
C4	0.0242 (9)	0.0289 (9)	0.0305 (9)	−0.0007 (7)	0.0073 (7)	0.0014 (7)
C5	0.0289 (9)	0.0228 (8)	0.0336 (10)	−0.0010 (7)	0.0071 (7)	−0.0031 (7)
C6	0.0302 (9)	0.0226 (8)	0.0285 (9)	−0.0043 (7)	0.0014 (7)	−0.0017 (7)
C7	0.0274 (9)	0.0252 (9)	0.0239 (8)	−0.0032 (7)	0.0028 (7)	−0.0008 (7)
C8	0.0270 (8)	0.0242 (9)	0.0239 (8)	−0.0019 (7)	0.0074 (7)	0.0005 (6)
C9	0.0308 (9)	0.0313 (9)	0.0282 (9)	−0.0021 (7)	0.0037 (7)	0.0017 (7)
C10	0.0357 (10)	0.0285 (9)	0.0331 (10)	0.0041 (8)	0.0099 (8)	0.0067 (8)
C11	0.0402 (10)	0.0225 (9)	0.0369 (10)	−0.0016 (7)	0.0133 (8)	0.0001 (7)
C12	0.0266 (8)	0.0247 (8)	0.0270 (9)	−0.0017 (7)	0.0092 (7)	−0.0021 (7)
C13	0.0230 (8)	0.0296 (9)	0.0255 (9)	−0.0025 (7)	0.0065 (7)	−0.0014 (7)
N1	0.0281 (8)	0.0325 (8)	0.0305 (8)	−0.0031 (6)	0.0031 (6)	−0.0043 (6)
N2	0.0337 (8)	0.0258 (8)	0.0328 (8)	−0.0031 (6)	0.0061 (6)	−0.0036 (6)
N3	0.0296 (8)	0.0197 (7)	0.0488 (10)	0.0004 (6)	0.0065 (7)	0.0035 (7)
N4	0.0258 (7)	0.0233 (7)	0.0348 (8)	0.0006 (6)	0.0064 (6)	0.0037 (6)
O1	0.0358 (7)	0.0304 (7)	0.0443 (8)	−0.0045 (6)	0.0088 (6)	0.0097 (6)
O2	0.0381 (7)	0.0349 (7)	0.0468 (8)	−0.0022 (6)	0.0172 (6)	−0.0118 (6)
O3	0.0482 (8)	0.0293 (7)	0.0239 (7)	−0.0074 (6)	0.0021 (6)	−0.0027 (5)
Cl1	0.0309 (3)	0.0414 (3)	0.0425 (3)	−0.00238 (18)	0.0053 (2)	−0.0005 (2)

Geometric parameters (Å, º)

C1—N1	1.335 (3)	C7—C8	1.520 (2)
C1—C2	1.375 (3)	C8—C9	1.388 (2)
C1—H1	0.9300	C8—C12	1.391 (2)
C2—C3	1.376 (3)	C9—C10	1.383 (3)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.394 (3)	C10—C11	1.382 (3)
C3—H3	0.9300	C10—H10	0.9300
C4—C13	1.380 (2)	C11—N2	1.333 (2)
C4—C5	1.523 (2)	C11—H11	0.9300
C5—O2	1.403 (2)	C12—N2	1.342 (2)
C5—N3	1.453 (2)	C12—C13	1.466 (2)
C5—C7	1.589 (2)	C13—N1	1.350 (2)
C6—O1	1.233 (2)	N1—H1A	0.8600

C6—N4	1.350 (2)	N3—H3A	0.8600
C6—N3	1.369 (2)	N4—H4	0.8600
C7—O3	1.394 (2)	O2—H2A	0.8200
C7—N4	1.449 (2)	O3—H3B	0.8200

N1—C1—C2	119.82 (18)	C12—C8—C7	122.01 (15)
N1—C1—H1	120.1	C10—C9—C8	119.56 (17)
C2—C1—H1	120.1	C10—C9—H9	120.2
C1—C2—C3	118.87 (19)	C8—C9—H9	120.2
C1—C2—H2	120.6	C11—C10—C9	118.93 (17)
C3—C2—H2	120.6	C11—C10—H10	120.5
C2—C3—C4	120.59 (19)	C9—C10—H10	120.5
C2—C3—H3	119.7	N2—C11—C10	123.04 (16)
C4—C3—H3	119.7	N2—C11—H11	118.5
C13—C4—C3	118.54 (17)	C10—C11—H11	118.5
C13—C4—C5	121.21 (16)	N2—C12—C8	124.23 (16)
C3—C4—C5	120.21 (16)	N2—C12—C13	116.16 (15)
O2—C5—N3	109.01 (14)	C8—C12—C13	119.61 (15)
O2—C5—C4	109.09 (14)	N1—C13—C4	119.09 (16)
N3—C5—C4	110.92 (15)	N1—C13—C12	117.68 (15)
O2—C5—C7	112.82 (15)	C4—C13—C12	123.23 (16)
N3—C5—C7	100.76 (13)	C1—N1—C13	122.99 (17)
C4—C5—C7	113.94 (14)	C1—N1—H1A	118.5
O1—C6—N4	125.80 (17)	C13—N1—H1A	118.5
O1—C6—N3	125.70 (16)	C11—N2—C12	117.26 (16)
N4—C6—N3	108.49 (15)	C6—N3—C5	110.99 (14)
O3—C7—N4	112.13 (14)	C6—N3—H3A	124.5
O3—C7—C8	106.15 (13)	C5—N3—H3A	124.5
N4—C7—C8	111.12 (14)	C6—N4—C7	112.54 (15)
O3—C7—C5	112.10 (14)	C6—N4—H4	123.7
N4—C7—C5	100.35 (13)	C7—N4—H4	123.7
C8—C7—C5	115.14 (14)	C5—O2—H2A	109.5
C9—C8—C12	116.98 (16)	C7—O3—H3B	109.5
C9—C8—C7	120.98 (15)		

N1—C1—C2—C3	0.5 (3)	C9—C8—C12—N2	1.3 (3)
C1—C2—C3—C4	2.3 (3)	C7—C8—C12—N2	179.10 (16)
C2—C3—C4—C13	−3.4 (3)	C9—C8—C12—C13	−179.17 (16)
C2—C3—C4—C5	178.93 (18)	C7—C8—C12—C13	−1.4 (2)
C13—C4—C5—O2	−110.16 (18)	C3—C4—C13—N1	1.7 (3)
C3—C4—C5—O2	67.4 (2)	C5—C4—C13—N1	179.35 (15)
C13—C4—C5—N3	129.75 (17)	C3—C4—C13—C12	−177.36 (17)
C3—C4—C5—N3	−52.7 (2)	C5—C4—C13—C12	0.3 (3)
C13—C4—C5—C7	16.9 (2)	N2—C12—C13—N1	−8.7 (2)
C3—C4—C5—C7	−165.51 (16)	C8—C12—C13—N1	171.79 (15)
O2—C5—C7—O3	−21.6 (2)	N2—C12—C13—C4	170.44 (16)
N3—C5—C7—O3	94.51 (16)	C8—C12—C13—C4	−9.1 (3)
C4—C5—C7—O3	−146.68 (15)	C2—C1—N1—C13	−2.3 (3)

O2—C5—C7—N4	−140.73 (14)	C4—C13—N1—C1	1.1 (3)
N3—C5—C7—N4	−24.66 (16)	C12—C13—N1—C1	−179.75 (17)
C4—C5—C7—N4	94.16 (16)	C10—C11—N2—C12	−0.7 (3)
O2—C5—C7—C8	99.91 (17)	C8—C12—N2—C11	−0.6 (3)
N3—C5—C7—C8	−144.01 (14)	C13—C12—N2—C11	179.89 (16)
C4—C5—C7—C8	−25.2 (2)	O1—C6—N3—C5	167.40 (17)
O3—C7—C8—C9	−39.1 (2)	N4—C6—N3—C5	−12.0 (2)
N4—C7—C8—C9	83.02 (19)	O2—C5—N3—C6	142.10 (15)
C5—C7—C8—C9	−163.78 (16)	C4—C5—N3—C6	−97.76 (17)
O3—C7—C8—C12	143.18 (16)	C7—C5—N3—C6	23.22 (18)
N4—C7—C8—C12	−94.67 (19)	O1—C6—N4—C7	173.90 (17)
C5—C7—C8—C12	18.5 (2)	N3—C6—N4—C7	−6.7 (2)
C12—C8—C9—C10	−0.8 (3)	O3—C7—N4—C6	−98.97 (17)
C7—C8—C9—C10	−178.56 (16)	C8—C7—N4—C6	142.41 (15)
C8—C9—C10—C11	−0.4 (3)	C5—C7—N4—C6	20.17 (17)
C9—C10—C11—N2	1.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.86	2.41	3.1512 (17)	145
N3—H3*A*···O2^{ii}	0.86	2.65	3.146 (2)	118
N4—H4···Cl1^{iii}	0.86	2.50	3.2490 (16)	147
O2—H2*A*···Cl1	0.82	2.28	3.0712 (15)	163
O3—H3*B*···O1^{iv}	0.82	1.89	2.6867 (18)	165

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