

1*H*-Imidazo[4,5-*f*][1,10]phenanthroline-7-ium perchlorate monohydrate

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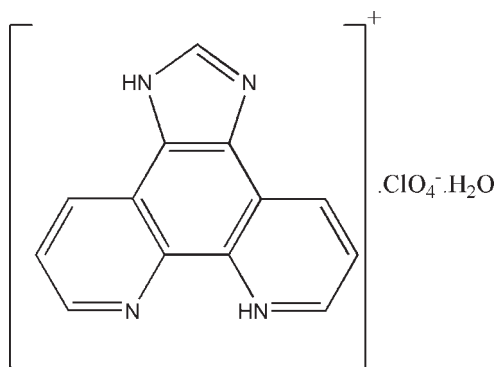
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
 R factor = 0.057; wR factor = 0.197; data-to-parameter ratio = 12.2.

In the title crystal structure, $\text{C}_{13}\text{H}_9\text{N}_4^+\cdot\text{ClO}_4^-\cdot\text{H}_2\text{O}$, cations, anions and water molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{N}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming layers parallel to (001). In addition, there are weak $\pi-\pi$ stacking interactions between the layers, involving the cations and with centroid-centroid distances in the range 3.584 (2)–3.662 (2) Å, forming a three-dimensional network.

Related literature

For background to 1*H*-imidazo[4,5-*f*][1,10]-phenanthroline and its use as a molecular building block, see: Xiong *et al.* (1999); Yu *et al.* (2009); Liu *et al.* (2009).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_9\text{N}_4^+\cdot\text{ClO}_4^-\cdot\text{H}_2\text{O}$
 $M_r = 338.71$

 Monoclinic, $P2_1/c$
 $a = 11.401$ (2) Å
 $b = 18.475$ (3) Å
 $c = 6.7163$ (13) Å
 $\beta = 90.179$ (3)°
 $V = 1414.7$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.30$ mm⁻¹
 $T = 298$ K
 $0.30 \times 0.26 \times 0.17$ mm

Data collection

 Bruker APEXII area-detector
 diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2005)
 $T_{\min} = 0.914$, $T_{\max} = 0.950$

 7051 measured reflections
 2534 independent reflections
 1734 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.197$
 $S = 1.01$
 2534 reflections
 208 parameters

 3 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1W}^i$	0.86	1.90	2.713 (4)	156
$\text{N3}-\text{H3A}\cdots\text{O3}^{ii}$	0.86	1.99	2.825 (4)	162
$\text{O1W}-\text{H1WB}\cdots\text{N4}$	0.84	2.02	2.852 (4)	177
$\text{O1W}-\text{H1WA}\cdots\text{O2}$	0.84	2.25	3.018 (5)	154

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2890).

References

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supplementary materials

Acta Cryst. (2009). E65, o2332 [doi:10.1107/S1600536809034576]

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Comment

1*H*-imidazo[4,5-*f*][1,10]-phenanthroline (IP) is an important derivative of 1,10-phenanthroline that has been used to recognize the secondary structure of DNA in an Ru(II) complex (Xiong *et al.*, 1999). IP is a good molecular building block and has been used to construct some interesting structures (Yu *et al.*, 2009, Liu *et al.*, 2009). In an attempt to form a Zn(II) complex with IP, we adventitiously formed the title compound (I) and its crystal structure is determined herein.

The asymmetric unit of (I) is shown in Fig 1. In the crystal structure N-H \cdots O, O-H \cdots N and O-H \cdots O hydrogen bonds link cations, water molecules and perchlorate anions into a 2-D network (Fig. 2). Details of the hydrogen-bonding geometry are given in Table 1. In addition, there are weak π - π stacking interactions between layers, involving cations with centroid to centroid distances in the range 3.584 (2)-3.662 (2)Å forming a three-dimensional network.

Experimental

IP (0.23 mg, 0.1 mmol), Zn(ClO₄)₂ (0.27 mg, 0.1 mmol), were dissolved in methanol. The mixture was heated and stirred for ten hours under reflux. The resulting solid was then filtered off to give a pure solution which was treated with diethyl ether in a closed vessel. Five weeks later, single crystals were obtained.

Refinement

All H atoms were visible in difference Fourier maps but were subsequently placed in calculated positions treated as riding with C—H = 0.93, N—H = 0.86Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$. The H atoms of the water molecules were included in the subsequent refinement with O—H = 0.84Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

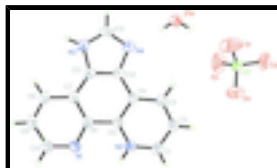


Fig. 1. The asymmetric unit of (I) with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii.



Fig. 2. Part of the crystal structure of (I). Hydrogen bonds are drawn as dashed lines and π - π stacking interactions are denoted by dashed lines along with labels (A) and (B).

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Crystal data

$C_{13}H_9N_4^+ \cdot ClO_4^- \cdot H_2O$	$F_{000} = 696$
$M_r = 338.71$	$D_x = 1.590 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 2521 reflections
$a = 11.401 (2) \text{ \AA}$	$\theta = 1.8\text{--}25.2^\circ$
$b = 18.475 (3) \text{ \AA}$	$\mu = 0.30 \text{ mm}^{-1}$
$c = 6.7163 (13) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 90.179 (3)^\circ$	Block, colorless
$V = 1414.7 (4) \text{ \AA}^3$	$0.30 \times 0.26 \times 0.17 \text{ mm}$
$Z = 4$	

Data collection

Bruker APEXII area-detector diffractometer	2534 independent reflections
Radiation source: fine-focus sealed tube	1734 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.2^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -13 \rightarrow 11$
$T_{\text{min}} = 0.914$, $T_{\text{max}} = 0.950$	$k = -21 \rightarrow 22$
7051 measured reflections	$l = -7 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained
$wR(F^2) = 0.197$	$w = 1/[\sigma^2(F_o^2) + (0.1261P)^2 + 0.1912P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
2534 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
208 parameters	$\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
3 restraints	$\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.7565 (2)	0.41426 (15)	0.3687 (4)	0.0563 (7)
N2	0.5235 (2)	0.38399 (13)	0.3439 (4)	0.0494 (7)
H2A	0.5510	0.4273	0.3390	0.059*
N3	0.8115 (3)	0.14741 (15)	0.3782 (4)	0.0539 (7)
H3A	0.8855	0.1388	0.3832	0.065*
N4	0.6190 (3)	0.12600 (14)	0.3669 (4)	0.0537 (7)
C1	0.4079 (3)	0.37511 (19)	0.3383 (5)	0.0583 (9)
H1	0.3590	0.4153	0.3301	0.070*
C2	0.3602 (3)	0.30690 (19)	0.3445 (5)	0.0577 (9)
H2	0.2792	0.3008	0.3435	0.069*
C3	0.4328 (3)	0.24803 (18)	0.3521 (4)	0.0508 (8)
H3	0.4011	0.2016	0.3527	0.061*
C4	0.5552 (2)	0.25749 (16)	0.3589 (4)	0.0423 (7)
C5	0.5997 (2)	0.32858 (16)	0.3569 (4)	0.0424 (7)
C6	0.7246 (3)	0.34396 (16)	0.3670 (4)	0.0437 (7)
C7	0.8053 (3)	0.28627 (17)	0.3736 (4)	0.0463 (7)
C8	0.9257 (3)	0.3041 (2)	0.3816 (5)	0.0575 (9)
H8	0.9826	0.2681	0.3859	0.069*
C9	0.9563 (3)	0.3750 (2)	0.3827 (5)	0.0676 (10)
H9	1.0350	0.3880	0.3883	0.081*
C10	0.8704 (3)	0.4282 (2)	0.3753 (5)	0.0656 (10)
H10	0.8942	0.4763	0.3751	0.079*
C11	0.7577 (3)	0.21528 (16)	0.3725 (4)	0.0462 (7)
C12	0.6393 (3)	0.20018 (16)	0.3648 (4)	0.0451 (7)
C13	0.7259 (3)	0.09872 (19)	0.3745 (5)	0.0584 (9)
H13	0.7402	0.0492	0.3770	0.070*
Cl1	0.15290 (7)	0.11627 (5)	0.40411 (14)	0.0643 (4)
O1	0.2077 (4)	0.07296 (19)	0.5458 (6)	0.1414 (17)
O2	0.2199 (3)	0.1137 (2)	0.2286 (6)	0.1356 (15)
O3	0.0402 (2)	0.08894 (19)	0.3635 (5)	0.0990 (10)
O4	0.1451 (3)	0.18729 (16)	0.4828 (6)	0.1099 (12)
O1W	0.4428 (2)	0.02582 (13)	0.2434 (5)	0.0871 (9)
H1WB	0.4934	0.0564	0.2771	0.131*

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H1WA 0.3755 0.0409 0.2713 0.131*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0558 (17)	0.0523 (17)	0.0608 (16)	-0.0063 (13)	-0.0055 (13)	0.0023 (12)
N2	0.0472 (15)	0.0436 (15)	0.0572 (16)	0.0054 (11)	-0.0031 (12)	0.0011 (11)
N3	0.0536 (16)	0.0575 (17)	0.0506 (15)	0.0156 (14)	0.0007 (12)	0.0021 (12)
N4	0.0655 (18)	0.0469 (16)	0.0488 (15)	0.0036 (13)	0.0019 (13)	0.0006 (11)
C1	0.050 (2)	0.064 (2)	0.061 (2)	0.0124 (16)	-0.0055 (15)	-0.0016 (15)
C2	0.0421 (17)	0.069 (2)	0.062 (2)	0.0021 (16)	-0.0014 (15)	-0.0016 (16)
C3	0.0482 (18)	0.057 (2)	0.0470 (17)	-0.0054 (15)	-0.0012 (13)	0.0004 (14)
C4	0.0434 (17)	0.0471 (17)	0.0364 (15)	-0.0003 (13)	0.0031 (12)	-0.0003 (12)
C5	0.0421 (16)	0.0479 (17)	0.0372 (15)	0.0035 (13)	-0.0024 (12)	0.0004 (12)
C6	0.0469 (17)	0.0445 (17)	0.0396 (15)	-0.0030 (13)	-0.0012 (12)	0.0032 (11)
C7	0.0434 (16)	0.057 (2)	0.0381 (15)	0.0013 (14)	0.0004 (12)	0.0020 (12)
C8	0.0431 (18)	0.071 (2)	0.0581 (19)	0.0031 (16)	-0.0005 (14)	0.0023 (16)
C9	0.044 (2)	0.086 (3)	0.074 (2)	-0.0140 (18)	-0.0041 (17)	0.0049 (19)
C10	0.062 (2)	0.059 (2)	0.076 (2)	-0.0161 (18)	-0.0063 (18)	0.0054 (17)
C11	0.0507 (18)	0.0493 (19)	0.0386 (15)	0.0098 (14)	0.0016 (13)	0.0023 (12)
C12	0.0511 (18)	0.0476 (18)	0.0366 (15)	0.0031 (14)	0.0033 (12)	0.0001 (12)
C13	0.078 (2)	0.0406 (18)	0.0570 (19)	0.0052 (18)	-0.0009 (17)	-0.0016 (14)
Cl1	0.0453 (5)	0.0575 (6)	0.0902 (7)	0.0079 (4)	-0.0048 (4)	-0.0043 (4)
O1	0.157 (4)	0.089 (2)	0.178 (4)	0.033 (2)	-0.096 (3)	0.005 (2)
O2	0.100 (3)	0.167 (4)	0.140 (3)	-0.013 (2)	0.059 (2)	-0.016 (3)
O3	0.0470 (15)	0.117 (2)	0.133 (3)	-0.0055 (15)	-0.0056 (16)	-0.028 (2)
O4	0.107 (3)	0.0571 (18)	0.166 (3)	0.0161 (16)	-0.005 (2)	-0.0134 (19)
O1W	0.0592 (16)	0.0560 (16)	0.146 (3)	-0.0021 (12)	0.0052 (16)	-0.0208 (15)

Geometric parameters (\AA , $^\circ$)

N1—C10	1.324 (4)	C5—C6	1.454 (4)
N1—C6	1.349 (4)	C6—C7	1.408 (4)
N2—C1	1.329 (4)	C7—C8	1.413 (4)
N2—C5	1.345 (4)	C7—C11	1.419 (4)
N2—H2A	0.8600	C8—C9	1.355 (5)
N3—C13	1.327 (4)	C8—H8	0.9300
N3—C11	1.396 (4)	C9—C10	1.388 (5)
N3—H3A	0.8600	C9—H9	0.9300
N4—C13	1.320 (4)	C10—H10	0.9300
N4—C12	1.390 (4)	C11—C12	1.379 (4)
C1—C2	1.373 (5)	C13—H13	0.9300
C1—H1	0.9300	Cl1—O1	1.390 (3)
C2—C3	1.367 (5)	Cl1—O3	1.406 (3)
C2—H2	0.9300	Cl1—O2	1.407 (4)
C3—C4	1.408 (4)	Cl1—O4	1.417 (3)
C3—H3	0.9300	O1W—H1WB	0.8379
C4—C5	1.408 (4)	O1W—H1WA	0.8377
C4—C12	1.429 (4)		

C10—N1—C6	116.9 (3)	C6—C7—C11	116.7 (3)
C1—N2—C5	123.2 (3)	C8—C7—C11	126.0 (3)
C1—N2—H2A	118.4	C9—C8—C7	118.4 (3)
C5—N2—H2A	118.4	C9—C8—H8	120.8
C13—N3—C11	106.6 (3)	C7—C8—H8	120.8
C13—N3—H3A	126.7	C8—C9—C10	120.2 (3)
C11—N3—H3A	126.7	C8—C9—H9	119.9
C13—N4—C12	102.9 (3)	C10—C9—H9	119.9
N2—C1—C2	120.3 (3)	N1—C10—C9	123.7 (3)
N2—C1—H1	119.8	N1—C10—H10	118.1
C2—C1—H1	119.8	C9—C10—H10	118.1
C3—C2—C1	119.4 (3)	C12—C11—N3	104.4 (3)
C3—C2—H2	120.3	C12—C11—C7	124.2 (3)
C1—C2—H2	120.3	N3—C11—C7	131.4 (3)
C2—C3—C4	120.2 (3)	C11—C12—N4	111.2 (3)
C2—C3—H3	119.9	C11—C12—C4	120.5 (3)
C4—C3—H3	119.9	N4—C12—C4	128.3 (3)
C3—C4—C5	118.2 (3)	N4—C13—N3	114.9 (3)
C3—C4—C12	125.0 (3)	N4—C13—H13	122.6
C5—C4—C12	116.8 (3)	N3—C13—H13	122.6
N2—C5—C4	118.6 (3)	O1—C11—O3	109.5 (2)
N2—C5—C6	119.1 (3)	O1—C11—O2	108.1 (3)
C4—C5—C6	122.3 (3)	O3—C11—O2	108.9 (2)
N1—C6—C7	123.5 (3)	O1—C11—O4	107.8 (2)
N1—C6—C5	116.9 (3)	O3—C11—O4	110.2 (2)
C7—C6—C5	119.5 (3)	O2—C11—O4	112.2 (2)
C6—C7—C8	117.3 (3)	H1WB—O1W—H1WA	110.3

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2A \cdots O1W ⁱ	0.86	1.90	2.713 (4)	156
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Fig. 1

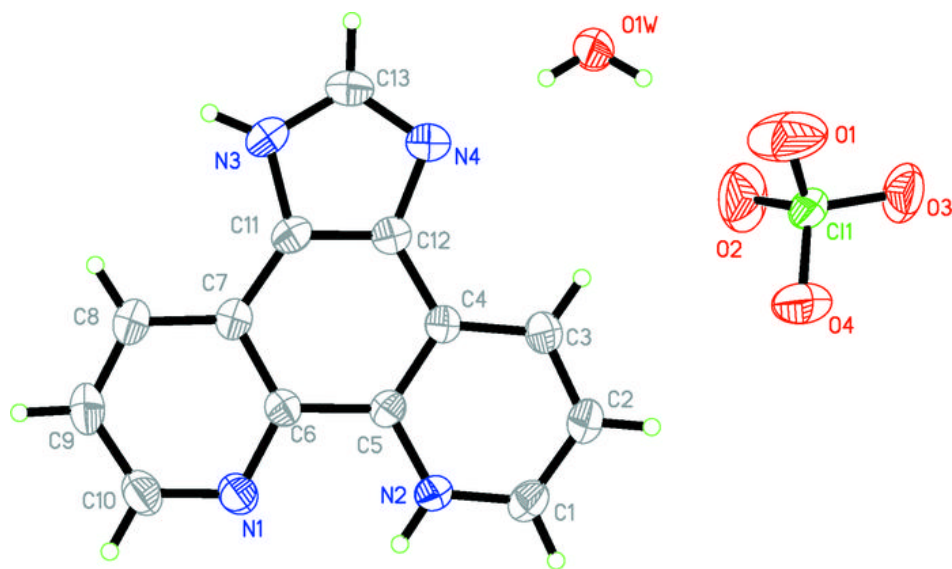


Fig. 2

