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# 5,6-Dioxo-1,10-phenanthrolin-1-ium chloride

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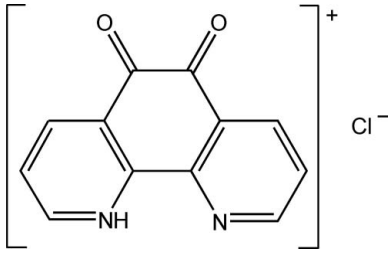
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.033; $wR$ factor = 0.093; data-to-parameter ratio = 9.7.

The title compound, $C_{12}H_7N_2O_2^+\cdot Cl^-$, is isostructural with its bromide analogue. The compound exhibits a layered structure in which all atoms lie on a crystallographic mirror plane. N$^+$—H$\cdots$Cl$^-$ hydrogen bonds, C—H$\cdots$O and C—H$\cdots$Cl$^-$ contacts are formed within each layer. The perpendicular separation between the layers is 3.141 (1) Å.

## Related literature

For the isostructural bromide analogue, see: Bomfim *et al.* (2003).




## Experimental

### Crystal data

$C_{12}H_7N_2O_2^+\cdot Cl^-$
$M_r$ = 246.65
Orthorhombic, *Pnma*
$a$ = 14.2870 (11) Å
$b$ = 6.2833 (5) Å
$c$ = 12.0019 (10) Å
$V$ = 1077.40 (15) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.34 mm$^{-1}$
$T$ = 298 (2) K
0.40 × 0.07 × 0.04 mm

### Data collection

Bruker Nonius X8-APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min}$ = 0.744, $T_{max}$ = 0.986
4229 measured reflections
1028 independent reflections
758 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.093
$S$ = 1.04
1028 reflections
106 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| N1—H1$\cdots$Cl1 | 0.88 (1) | 2.27 (2) | 3.067 (2) | 150 (3) |
| C1—H1$A\cdots$O1$^{i}$ | 0.93 | 2.27 | 3.074 (3) | 145 |
| C3—H3$A\cdots$Cl1$^{ii}$ | 0.93 | 2.87 | 3.755 (3) | 161 |
| C8—H8$A\cdots$Cl1$^{iii}$ | 0.93 | 2.91 | 3.545 (3) | 127 |
| C9—H9$A\cdots$Cl1$^{iii}$ | 0.93 | 2.93 | 3.556 (3) | 126 |
| C10—H10$A\cdots$O2$^{iv}$ | 0.93 | 2.27 | 3.196 (3) | 177 |

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

Data collection: *APEX2* (Bruker Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PR2019).

## References

Bomfim, J. A. S., Filgueiras, C. A. L., Howie, R. A. & Wardell, J. L. (2003). *Acta Cryst.* E**59**, o244–o246.
Bruker (2003). *SAINT*. Version 7.06a. Bruker AXS Inc., Madison, Wisconsin, USA.
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Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

  doi:10.1107/S1600536807061788 

# supporting information

*Acta Cryst.* (2008). E**64**, o34 [https://doi.org/10.1107/S1600536807061788]

## 5,6-Dioxo-1,10-phenanthrolin-1-ium chloride

**Cedric Borel and Andrew D. Bond**

### S1. Comment

The title compound, $C_{12}H_7N_2O_2^+.Cl^-$, was obtained as a by-product from an attempted synthesis of a metal-organic framework (MOF). It is isostructural with its bromide analogue (Bomfim, *et al.*, 2003).

### S2. Experimental

5,6-Dioxo-1,10-phenanthroline (40 mg, 0.19 mmol) was dissolved in 10 ml of water at room temperature with stirring and HCl(aq) was added until the pH was 4. When all of the compound had dissolved, $K_3[Mn(CN)_6]$ (31 mg, 0.1 mmol) and $NH_4Cl$ (5 mg, 0.1 mmol) were added and the mixture was left to stand overnight at 277 K, yielding yellow crystals of the title compound.

### S3. Refinement

H atoms bound to C atoms were placed geometrically and allowed to ride during refinement with C—H = 0.93 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C). The H atom bound to N1 was located in a difference Fourier map and refined with an isotropic displacement parameter, with the N—H distance restrained to be 0.87 (1) Å.

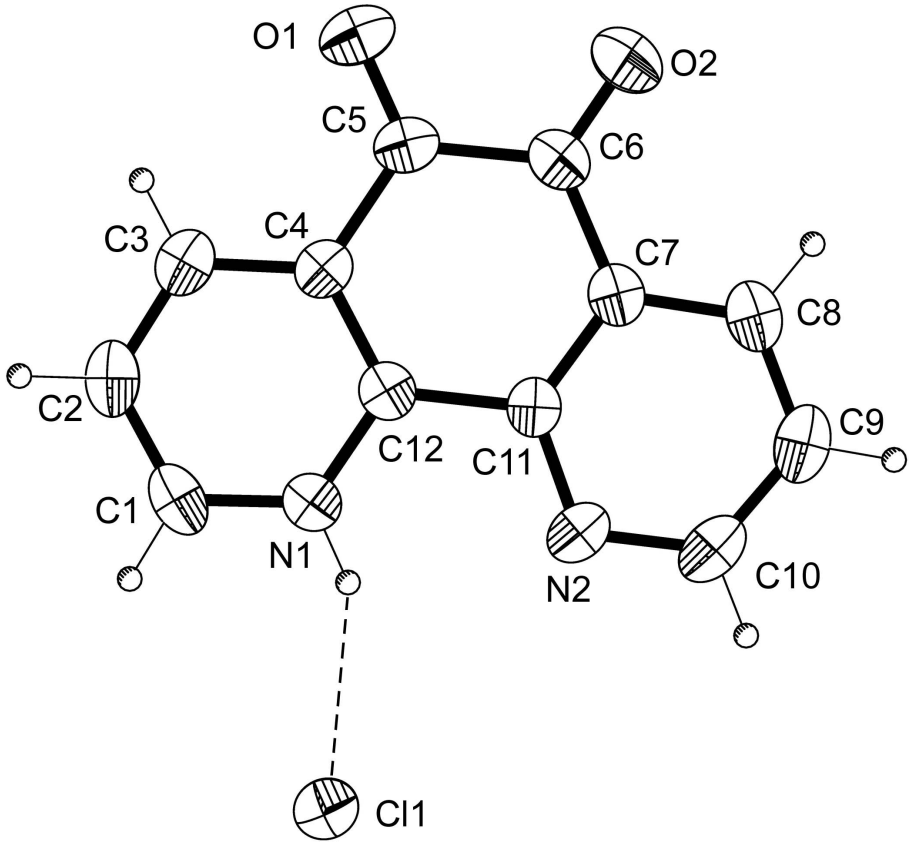


**Figure 1**

Molecular structure of the title compound showing displacement ellipsoids at the 50% probability level for non-H atoms. The dashed line denotes the $N^+$—H···$Cl^-$ hydrogen bond.

## 5,6-Dioxo-1,10-phenanthrolin-1-ium chloride

*Crystal data*

$C_{12}H_7N_2O_2^+ \cdot Cl^-$
$M_r = 246.65$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 14.2870\ (11)$ Å
$b = 6.2833\ (5)$ Å
$c = 12.0019\ (10)$ Å
$V = 1077.40\ (15)$ Å$^3$
$Z = 4$
$F(000) = 504$
$D_x = 1.521$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1190 reflections
$\theta = 2.9$–$24.1°$
$\mu = 0.34$ mm$^{-1}$
$T = 298$ K
Needle, yellow
0.40 × 0.07 × 0.04 mm

*Data collection*

Bruker Nonius X8-APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.744$, $T_{max} = 0.986$
4229 measured reflections
1028 independent reflections
758 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.1°$, $\theta_{min} = 3.7°$
$h = -16 \rightarrow 12$
$k = -7 \rightarrow 4$
$l = -14 \rightarrow 14$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.093$
$S = 1.04$
1028 reflections
106 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
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.29410 (5) | 0.2500 | 0.51333 (6) | 0.0466 (3) |
| O1 | 0.82632 (14) | 0.2500 | 0.63231 (19) | 0.0638 (7) |
| O2 | 0.85156 (15) | 0.2500 | 0.4091 (2) | 0.0956 (10) |
| N1 | 0.50089 (15) | 0.2500 | 0.58192 (19) | 0.0360 (6) |
| H1 | 0.4519 (15) | 0.2500 | 0.538 (2) | 0.071 (11)* |
| N2 | 0.52192 (15) | 0.2500 | 0.36021 (18) | 0.0401 (6) |
| C1 | 0.4820 (2) | 0.2500 | 0.6909 (2) | 0.0447 (7) |
| H1A | 0.4202 | 0.2500 | 0.7153 | 0.054* |
| C2 | 0.5539 (2) | 0.2500 | 0.7667 (2) | 0.0482 (8) |
| H2A | 0.5413 | 0.2500 | 0.8427 | 0.058* |
| C3 | 0.6444 (2) | 0.2500 | 0.7295 (2) | 0.0438 (7) |
| H3A | 0.6938 | 0.2500 | 0.7799 | 0.053* |
| C4 | 0.66185 (18) | 0.2500 | 0.6159 (2) | 0.0376 (7) |
| C5 | 0.7592 (2) | 0.2500 | 0.5718 (2) | 0.0452 (7) |
| C6 | 0.7731 (2) | 0.2500 | 0.4455 (3) | 0.0511 (8) |
| C7 | 0.68991 (19) | 0.2500 | 0.3738 (2) | 0.0399 (7) |
| C8 | 0.6968 (2) | 0.2500 | 0.2584 (2) | 0.0479 (8) |
| H8A | 0.7553 | 0.2500 | 0.2243 | 0.057* |
| C9 | 0.6174 (2) | 0.2500 | 0.1954 (2) | 0.0515 (8) |
| H9A | 0.6209 | 0.2500 | 0.1180 | 0.062* |
| C10 | 0.5318 (2) | 0.2500 | 0.2487 (2) | 0.0481 (8) |
| H10A | 0.4780 | 0.2500 | 0.2052 | 0.058* |
| C11 | 0.60042 (18) | 0.2500 | 0.4196 (2) | 0.0328 (6) |
| C12 | 0.58855 (18) | 0.2500 | 0.5415 (2) | 0.0333 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0402 (5) | 0.0572 (5) | 0.0423 (5) | 0.000 | −0.0037 (3) | 0.000 |
| O1 | 0.0424 (12) | 0.0960 (18) | 0.0529 (14) | 0.000 | −0.0163 (10) | 0.000 |
| O2 | 0.0350 (14) | 0.194 (3) | 0.0580 (17) | 0.000 | 0.0115 (11) | 0.000 |
| N1 | 0.0338 (14) | 0.0369 (12) | 0.0372 (15) | 0.000 | 0.0020 (11) | 0.000 |
| N2 | 0.0422 (14) | 0.0412 (13) | 0.0367 (14) | 0.000 | −0.0096 (11) | 0.000 |
| C1 | 0.0453 (17) | 0.0453 (17) | 0.0436 (19) | 0.000 | 0.0167 (14) | 0.000 |
| C2 | 0.060 (2) | 0.0528 (18) | 0.0314 (17) | 0.000 | 0.0067 (15) | 0.000 |
| C3 | 0.0484 (18) | 0.0486 (17) | 0.0346 (17) | 0.000 | −0.0045 (14) | 0.000 |
| C4 | 0.0368 (17) | 0.0424 (16) | 0.0337 (16) | 0.000 | −0.0033 (12) | 0.000 |
| C5 | 0.0343 (17) | 0.0584 (18) | 0.0428 (18) | 0.000 | −0.0063 (14) | 0.000 |
| C6 | 0.0348 (18) | 0.076 (2) | 0.0425 (18) | 0.000 | 0.0052 (14) | 0.000 |
| C7 | 0.0392 (17) | 0.0458 (17) | 0.0348 (16) | 0.000 | 0.0033 (12) | 0.000 |
| C8 | 0.0483 (18) | 0.0588 (18) | 0.0365 (17) | 0.000 | 0.0096 (14) | 0.000 |
| C9 | 0.066 (2) | 0.057 (2) | 0.0312 (18) | 0.000 | −0.0008 (15) | 0.000 |
| C10 | 0.0542 (19) | 0.0502 (17) | 0.0397 (18) | 0.000 | −0.0159 (15) | 0.000 |
| C11 | 0.0369 (16) | 0.0310 (14) | 0.0305 (15) | 0.000 | 0.0011 (12) | 0.000 |
| C12 | 0.0349 (17) | 0.0301 (14) | 0.0348 (16) | 0.000 | 0.0016 (12) | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C5 | 1.202 (3) | C4—C12 | 1.376 (3) |
| O2—C6 | 1.203 (3) | C4—C5 | 1.488 (4) |
| N1—C1 | 1.335 (3) | C5—C6 | 1.530 (4) |
| N1—C12 | 1.343 (3) | C6—C7 | 1.468 (4) |
| N1—H1 | 0.88 (1) | C7—C8 | 1.388 (4) |
| N2—C11 | 1.329 (3) | C7—C11 | 1.392 (4) |
| N2—C10 | 1.345 (3) | C8—C9 | 1.363 (4) |
| C1—C2 | 1.373 (4) | C8—H8A | 0.930 |
| C1—H1A | 0.930 | C9—C10 | 1.381 (4) |
| C2—C3 | 1.368 (4) | C9—H9A | 0.930 |
| C2—H2A | 0.930 | C10—H10A | 0.930 |
| C3—C4 | 1.386 (4) | C11—C12 | 1.473 (3) |
| C3—H3A | 0.930 | | |
| | | | |
| C1—N1—C12 | 122.8 (2) | O2—C6—C5 | 118.8 (3) |
| C1—N1—H1 | 115 (2) | C7—C6—C5 | 118.4 (2) |
| C12—N1—H1 | 122 (2) | C8—C7—C11 | 117.4 (3) |
| C11—N2—C10 | 116.4 (2) | C8—C7—C6 | 121.8 (3) |
| N1—C1—C2 | 119.8 (3) | C11—C7—C6 | 120.8 (3) |
| N1—C1—H1A | 120.1 | C9—C8—C7 | 119.6 (3) |
| C2—C1—H1A | 120.1 | C9—C8—H8A | 120.2 |
| C3—C2—C1 | 119.4 (3) | C7—C8—H8A | 120.2 |
| C3—C2—H2A | 120.3 | C8—C9—C10 | 118.7 (3) |
| C1—C2—H2A | 120.3 | C8—C9—H9A | 120.6 |
| C2—C3—C4 | 119.4 (3) | C10—C9—H9A | 120.6 |

| | | | |
|---|---|---|---|
| C2—C3—H3A | 120.3 | N2—C10—C9 | 123.6 (3) |
| C4—C3—H3A | 120.3 | N2—C10—H10A | 118.2 |
| C12—C4—C3 | 120.1 (2) | C9—C10—H10A | 118.2 |
| C12—C4—C5 | 118.8 (3) | N2—C11—C7 | 124.3 (2) |
| C3—C4—C5 | 121.1 (2) | N2—C11—C12 | 115.8 (2) |
| O1—C5—C4 | 122.1 (3) | C7—C11—C12 | 119.9 (2) |
| O1—C5—C6 | 119.7 (3) | N1—C12—C4 | 118.4 (3) |
| C4—C5—C6 | 118.3 (2) | N1—C12—C11 | 117.8 (2) |
| O2—C6—C7 | 122.8 (3) | C4—C12—C11 | 123.8 (2) |

| | | | |
|---|---|---|---|
| C12—N1—C1—C2 | 0.0 | C7—C8—C9—C10 | 0.0 |
| N1—C1—C2—C3 | 0.0 | C11—N2—C10—C9 | 0.0 |
| C1—C2—C3—C4 | 0.0 | C8—C9—C10—N2 | 0.0 |
| C2—C3—C4—C12 | 0.0 | C10—N2—C11—C7 | 0.0 |
| C2—C3—C4—C5 | 180.0 | C10—N2—C11—C12 | 180.0 |
| C12—C4—C5—O1 | 180.0 | C8—C7—C11—N2 | 0.0 |
| C3—C4—C5—O1 | 0.0 | C6—C7—C11—N2 | 180.0 |
| C12—C4—C5—C6 | 0.0 | C8—C7—C11—C12 | 180.0 |
| C3—C4—C5—C6 | 180.0 | C6—C7—C11—C12 | 0.0 |
| O1—C5—C6—O2 | 0.0 | C1—N1—C12—C4 | 0.0 |
| C4—C5—C6—O2 | 180.0 | C1—N1—C12—C11 | 180.0 |
| O1—C5—C6—C7 | 180.0 | C3—C4—C12—N1 | 0.0 |
| C4—C5—C6—C7 | 0.0 | C5—C4—C12—N1 | 180.0 |
| O2—C6—C7—C8 | 0.0 | C3—C4—C12—C11 | 180.0 |
| C5—C6—C7—C8 | 180.0 | C5—C4—C12—C11 | 0.0 |
| O2—C6—C7—C11 | 180.0 | N2—C11—C12—N1 | 0.0 |
| C5—C6—C7—C11 | 0.0 | C7—C11—C12—N1 | 180.0 |
| C11—C7—C8—C9 | 0.0 | N2—C11—C12—C4 | 180.0 |
| C6—C7—C8—C9 | 180.0 | C7—C11—C12—C4 | 0.0 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···Cl1 | 0.88 (1) | 2.27 (2) | 3.067 (2) | 150 (3) |
| C1—H1*A*···O1$^{i}$ | 0.93 | 2.27 | 3.074 (3) | 145 |
| C3—H3*A*···Cl1$^{ii}$ | 0.93 | 2.87 | 3.755 (3) | 161 |
| C8—H8*A*···Cl1$^{iii}$ | 0.93 | 2.91 | 3.545 (3) | 127 |
| C9—H9*A*···Cl1$^{iii}$ | 0.93 | 2.93 | 3.556 (3) | 126 |
| C10—H10*A*···O2$^{iv}$ | 0.93 | 2.27 | 3.196 (3) | 177 |

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