organic compounds

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

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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···Cl$^-$ hydrogen bonds, C—H···O and C—H···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).
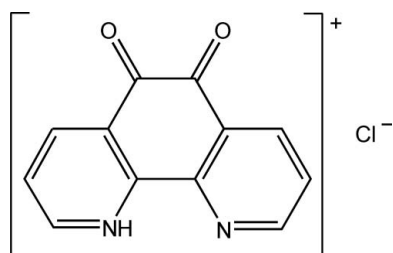
Experimental

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
$0.40 \times 0.07 \times 0.04$ 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···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—H1A···O1^i	0.93	2.27	3.074 (3)	145
C3—H3A···Cl1^{ii}	0.93	2.87	3.755 (3)	161
C8—H8A···Cl1^{iii}	0.93	2.91	3.545 (3)	127
C9—H9A···Cl1^{iii}	0.93	2.93	3.556 (3)	126
C10—H10A···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*.

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.
Bruker Nonius (2004). *APEX2*. Version 1.0-22. Bruker Nonius BV, Delft, The Netherlands.
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.

supplementary materials

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

C. Borel and **A. D. Bond**

Comment

The title compound, $C_{12}H_7N_2O_2^+ \cdot Cl^-$, 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).

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.

Refinement

H atoms bound to C atoms were placed geometrically and allowed to ride during refinement with C—H = 0.93 Å and $U_{iso}(H)$ = $1.2U_{eq}(C)$. 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.

Figures
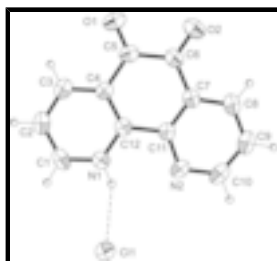
Fig. 1. Molecular structure of the title compound showing displacement ellipsoids at the 50% probability level for non-H atoms. The dashed line denotes the N^+—H···Cl^- hydrogen bond.

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

Crystal data

$C_{12}H_7N_2O_2^+ \cdot Cl^-$	$F_{000} = 504$
$M_r = 246.65$	$D_x = 1.521$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2n	Cell parameters from 1190 reflections
$a = 14.2870$ (11) Å	$\theta = 2.9$–24.1°

$b = 6.2833\ (5)$ Å

$c = 12.0019\ (10)$ Å

$V = 1077.40\ (15)$ Å^3

$Z = 4$

$\mu = 0.34$ mm^{-1}

$T = 298\ (2)$ K

Needle, yellow

$0.40 \times 0.07 \times 0.04$ mm

Data collection

Bruker Nonius X8-APEXII CCD diffractometer	1028 independent reflections
Radiation source: fine-focus sealed tube	758 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
$T = 298(2)$ K	$\theta_{max} = 25.1°$
ω and φ scans	$\theta_{min} = 3.7°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$h = -16 \rightarrow 12$
$T_{min} = 0.744$, $T_{max} = 0.986$	$k = -7 \rightarrow 4$
4229 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o{}^2) + (0.0554P)^2]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1028 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
106 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 ($Å^2$)

	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, °)

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, °)

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—H1A⋯O1[i]	0.93	2.27	3.074 (3)	145
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C10—H10A⋯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) $x-1/2, y, -z+1/2$.

Fig. 1