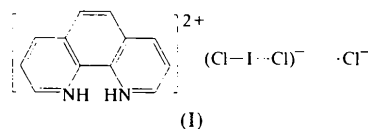


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or diprotonated resulting in diverse structures. In the system 1,10-phenanthroline–HCl–KI–I₂, several compounds have been obtained under different conditions (Wang *et al.*, 1999), of which the title compound, (I), is one example.



The crystal structure of (I) consists of diprotonated 1,10-phenanthroline cations (phenH_2^{2+}), and Cl^- and ICl_2^- anions (Fig. 1). In the phenH_2^{2+} cations, the bond lengths are very similar to those in neutral 1,10-phenanthroline (Nishigaki *et al.*, 1978), but the C—N angles [both $122.7(3)^\circ$] are significantly greater than the corresponding values in the neutral species [$117.2(6)$ – $117.9(5)^\circ$], in agreement with other structures containing protonated 1,10-phenanthroline (Hensen *et al.*, 1998; Lis, 1979). The enlargement of the internal angles at the protonated N atoms was also reported for quinolinium (Liu *et al.*, 1991), 8-hydroxyquinolinium (Balasubramanian & Muthiah, 1996) and bipyridinium (Liu *et al.*, 1992). The phenH_2^{2+} cation is planar and parallel to the *ac* plane, with deviations from the least-squares plane of less than $0.057(3) \text{ \AA}$.

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1,10-Phenanthroline(2+) dichloroiodide(1-) chloride

ZHE-MING WANG, YAN-QIA WANG, CHUN-SHENG LIAO AND CHUN-HUA YAN

State Key Laboratory of Rare Earth Materials Chemistry and Applications, PKU–HKU Joint Laboratory in Rare Earth Materials and Bioinorganic Chemistry, Peking University, Beijing 100871, People's Republic of China. E-mail: chyan@chemms.chem.pku.edu.cn

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Abstract

The title compound, $\text{C}_{12}\text{H}_{10}\text{N}_2^{2+} \cdot \text{ICl}_2^- \cdot \text{Cl}^-$, consists of 1,10-phenanthroline(2+) cations (phenH_2^{2+}), and ICl_2^- and Cl^- anions. The ICl_2^- anions occupy two crystallographic sites, one on a twofold axis and the other on a centre of symmetry. The phenH_2^{2+} cations build up positive columns along the *b* direction, while the ICl_2^- anions form infinite branched zigzag chains running along the *a* direction. The Cl^- anions embedded in the interstices of the positive columns form N—H...Cl hydrogen bonds, which contribute to the stabilization of the structure.

Comment

During the last decade, there has been considerable interest in the structure and properties of species obtained by combining protonated aromatic nitrogen bases with halide or polyhalide ions (El-Kholi *et al.*, 1988; Hendrixson *et al.*, 1991; Liu *et al.*, 1991, 1992; Tebbe *et al.*, 1995). Among these aromatic nitrogen bases, 1,10-phenanthroline is a good candidate for study as it has two close aromatic N atoms which can be neutral, mono-

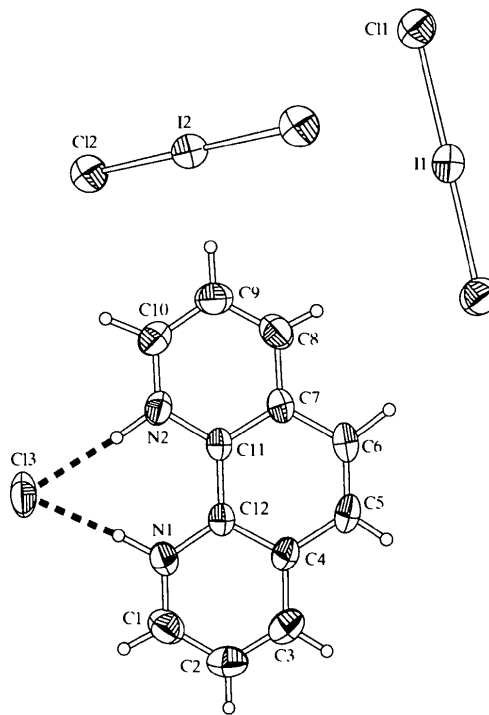


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

The two unique ICl_2^- anions are located on different special positions. Anion A [Cl1—I—Cl1^i ; symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$] is located on the twofold axis and is nearly linear, with Cl—I bond distances of 2.5522 (8) Å and a Cl—I—Cl angle of 177.62 (5)°. Anion B [Cl2—I—Cl2^{ii} ; symmetry code: (ii) $-x+1, -y+2, -z+1$] is located on a centre of symmetry and is consequently linear, with corresponding bond lengths of 2.5464 (9) Å. When ICl_2^- anions co-exist with large positive moieties, such as phenH_2^+ , they often have linear or near ideal linear symmetry (Bailar *et al.*, 1973; Foster, 1969).

The phenH_2^+ cations are stacked in positive columns which run along the b direction (Fig. 2). In each column, the alternate phenH_2^+ cations overlap only slightly with the middle neutral rings and the N atoms of the neighbouring phenH_2^+ cations point in opposite directions. The distance between the two adjacent cations is *ca* 4.1 Å, indicating that there is no strong interaction between them. The distance between Cl2 and I1^{iii} of 3.7830 (9) Å [symmetry code: (iii) $x, -y+1, z-\frac{1}{2}$] is less than the van der Waals sum, indicating some interaction between them. The ICl_2^- anions are linked by this interaction into infinite branched zigzag chains running along the c direction. The main chain, $\{\cdots\text{Cl—I—Cl}\cdots\text{I}\cdots\}_n$, is an infinite zigzag chain formed by the alternate linkage of B anions and the I atoms of A anions, while the Cl atoms of A anions form the branch.

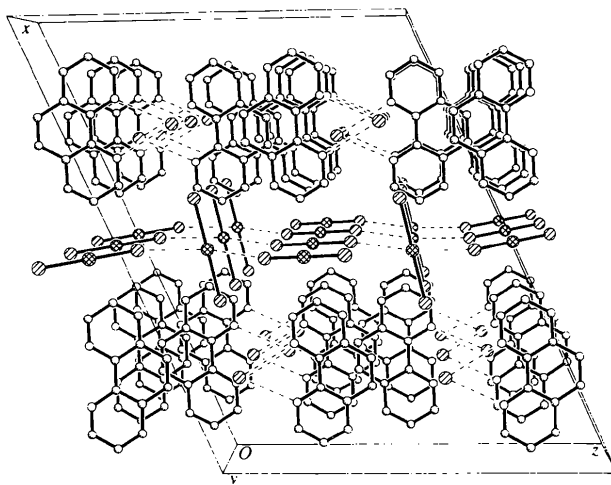


Fig. 2. Packing diagram viewed along the b direction. H atoms have been omitted for clarity.

The Cl^- ions (Cl3) are embedded in the interstices of the positive columns and form $\text{N—H}\cdots\text{Cl}$ hydrogen bonds with the protonated N atoms of the phenH_2^+ cations. The two protons are clearly distinguishable in the difference Fourier map and are located at distances

of 1.00 (4) and 0.99 (4) Å from N1 and N2, respectively. The efficient hydrogen bonds that are set up [$\text{N1}\cdots\text{Cl3}$ and $\text{N2}\cdots\text{Cl3}$; Table 3] are similar to those found in $[(\text{quinH}^+)_3\text{Cl}(\text{I}_3)_2]$ (quinH⁺ is quinolinium; Liu *et al.*, 1991) and $(\text{H}_2\text{phen})[\text{ReCl}_2(\text{H}_2\text{O})\text{O}_3]\text{Cl}$ (Lis, 1979). The key role of the hydrogen bonds is in stabilizing the cation by partly balancing the positive charges on the phenH_2^+ cation.

Experimental

KI (1.17 g) and I_2 (1.36 g) were dissolved in a mixture of ethanol (70 ml) and concentrated hydrochloric acid (36%, 30 ml). On addition of 1,10-phenanthroline (0.50 g) to the above solution (10 ml), a dark-red precipitate formed. The mixture was stirred for 48 h and then filtered. The filtrate was allowed to evaporate at room temperature and light-yellow transparent rectangular crystals of the title compound were obtained after 2 weeks. Elemental analysis, found: C 33.70, H 2.42, N 6.50%; calculated for $\text{C}_{12}\text{H}_{10}\text{Cl}_3\text{IN}_2$: C 33.61, H 2.57, N 6.53%.

Crystal data

$\text{C}_{12}\text{H}_{10}\text{N}_2^+\cdot\text{ICl}_2^-\cdot\text{Cl}^-$
 $M_r = 415.47$
 Monoclinic
 $C2/c$
 $a = 25.1766$ (10) Å
 $b = 6.6867$ (3) Å
 $c = 19.3304$ (7) Å
 $\beta = 113.807$ (3)°
 $V = 2977.3$ (2) Å³
 $Z = 8$
 $D_x = 1.854$ Mg m⁻³
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 10.04\text{--}14.35^\circ$
 $\mu = 2.673$ mm⁻¹
 $T = 293$ (2) K
 Block
 $0.30 \times 0.30 \times 0.10$ mm
 Light yellow

Data collection

Enraf–Nonius CAD-4
 MACH3 diffractometer
 θ - 2θ scans
 Absorption correction:
 ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.577$, $T_{\max} = 0.765$
 4063 measured reflections
 3945 independent reflections

3270 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.008$
 $\theta_{\max} = 28.97^\circ$
 $h = -34 \rightarrow 31$
 $k = -9 \rightarrow 0$
 $l = 0 \rightarrow 26$
 3 standard reflections
 frequency: 60 min
 intensity decay: 1.3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.085$
 3945 reflections
 205 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.0311P)]^2$
 $+ 3.9392P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.605$ e Å⁻³
 $\Delta\rho_{\min} = -0.549$ e Å⁻³
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U^{ij} a^i a^j$$

	x	y	z	U _{eq}
I1	1/2	0.60730 (5)	3/4	0.04532 (9)
I2	1/2	1	1/2	0.04959 (9)
C11	0.60788 (4)	0.59938 (15)	0.77674 (5)	0.0572 (2)
C12	0.49036 (3)	0.74997 (16)	0.39624 (5)	0.0578 (2)
C13	0.23404 (4)	0.47104 (15)	0.18731 (4)	0.0581 (2)
N1	0.17955 (10)	0.4378 (4)	0.29888 (13)	0.0405 (5)
N2	0.30368 (10)	0.4784 (4)	0.35927 (13)	0.0390 (5)
C1	0.12287 (14)	0.4092 (6)	0.27063 (19)	0.0521 (8)
C2	0.09313 (14)	0.4071 (6)	0.3173 (2)	0.0540 (8)
C3	0.12298 (14)	0.4363 (5)	0.3927 (2)	0.0470 (7)
C4	0.18333 (12)	0.4647 (4)	0.42346 (15)	0.0350 (5)
C5	0.21622 (14)	0.4921 (4)	0.50262 (16)	0.0395 (6)
C6	0.27449 (14)	0.5114 (4)	0.53120 (15)	0.0407 (6)
C7	0.30476 (12)	0.5066 (4)	0.48267 (15)	0.0355 (6)
C8	0.36529 (14)	0.5224 (5)	0.51043 (18)	0.0479 (7)
C9	0.39313 (14)	0.5155 (6)	0.4631 (2)	0.0551 (9)
C10	0.36088 (14)	0.4924 (5)	0.38637 (19)	0.0498 (8)
C11	0.27358 (11)	0.4841 (4)	0.40443 (14)	0.0325 (5)
C12	0.21184 (11)	0.4638 (4)	0.37421 (14)	0.0329 (5)

Table 2. Selected geometric parameters (Å, °)

I1—C11	2.5522 (8)	C4—C12	1.405 (4)
I2—C12	2.5464 (9)	C4—C5	1.428 (4)
N1—C1	1.320 (4)	C5—C6	1.349 (4)
N1—C12	1.362 (3)	C6—C7	1.428 (4)
N2—C10	1.322 (4)	C7—C8	1.400 (4)
N2—C11	1.368 (3)	C7—C11	1.403 (3)
C1—C2	1.386 (5)	C8—C9	1.359 (5)
C2—C3	1.358 (5)	C9—C10	1.382 (5)
C3—C4	1.403 (4)	C11—C12	1.429 (4)
C11 ⁱ —I1—C11	177.62 (5)	C1—N1—C12	122.7 (3)
C12 ⁱⁱ —I2—C12	180.0	C10—N2—C11	122.7 (3)

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

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...C13	1.00 (4)	2.00 (4)	2.995 (3)	176 (4)
N2—H10...C13	0.99 (4)	2.08 (4)	3.069 (3)	171 (3)

All H atoms were located by difference Fourier synthesis and were refined isotropically. C—H distances are in the range 0.86 (4)–1.04 (3) Å.

Data collection: CAD-4 ARGUS Software (Nonius, 1996). Cell refinement: CAD-4 ARGUS Software. Data reduction: MolEN (Fair, 1990). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b). Molecular graphics: SHELXTL/PC (Sheldrick, 1990). Software used to prepare material for publication: SHELXL97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1249). Services for accessing these data are described at the back of the journal.

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3,9-Dimethyl-1,11-diphenyl-4,8-diazaundecane-1,11-dione dihydrate

YALÇIN ELERMAN,^a MEHMET KABAK,^a HÜLYA KARA,^b KUTALMIŞ GÜVEN^c AND CANAN NAKİPOĞLU^d

^aDepartment of Engineering Physics, Faculty of Sciences, University of Ankara, 06100 Beşevler, Ankara, Turkey;

^bDepartment of Physics, Faculty of Art and Sciences, University of Balıkesir, 10100 Balıkesir, Turkey; ^cDepartment of Physics, Faculty of Art and Sciences, University of Kırıkkale, 71450 Yahşihan, Kırıkkale, Turkey; and

^dDepartment of Chemistry Education, Faculty of Education, University of Balıkesir, 10100 Balıkesir, Turkey. E-mail: kabak@science.ankara.edu.tr

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Abstract

The title compound, C₂₃H₂₆N₂O₂·2H₂O, displays strong intramolecular N—H...O [2.637 (2) Å] and intermolecular O—H...O [2.763 (4) and 2.842 (3) Å] hydrogen bonds within each salicylideneimine unit and water molecule. The Schiff base has a twofold axis passing