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# STRUCTURE OF TWO NOVEL FLUOROQUINOLONE SALTS

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Fluoroquinolone compounds with the composition $CfH_2^{2+}$, $2Cl^-$, $H_2O$ ($CfH_2Cl_2 \cdot H_2O$ (**I**)) and $4LevoH_2^{2+}$, $3[SnCl_6]^{2-}$, $2Cl^-$, $2H_2O$ ($(LevoH_2)_4[SnCl_6]_3Cl_2 \cdot 2H_2O$ (**II**)) (Cf is ciprofloxacin, Levo is levofloxacin) are prepared. Their structures are determined by XRSCD. Crystals **I** are monoclinic: $a = 8.6389(11)$ Å, $b = 14.5486(19)$ Å, $c = 14.8605(19)$ Å, $\beta = 91.914(3)°$, $V = 1866.7(4)$ Å$^3$, space group $P2_1/c$, $Z = 4$. Crystals **II** are triclinic: $a = 12.4821(8)$ Å, $b = 13.8144(8)$ Å, $c = 15.2342(9)$ Å, $\alpha = 84.360(1)°$, $\beta = 79.265(1)°$, $\gamma = 74.038(1)°$, $V = 2478.3(3)$ Å$^3$, space group $P1$, $Z = 1$. The structures are stabilized by multiple hydrogen bonds. The photoluminescent properties and thermal stability of compound **I** are considered.

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**Keywords:** ciprofloxacinium dichloride, *tetrakis*(levofloxacin) *tris*{hexachlorostannate(IV)} dichloride, synthesis, structure, properties.

## INTRODUCTION

Fluoroquinolones are a class of synthetic antibiotics that is most widely used in clinical practice [1-3]. Ciprofloxacin and levofloxacin (Fig. 1) are important representatives of this class. Ciprofloxacin (Cf) is one of the most poorly soluble fluoroquinolones and therefore has only limited applications as an ingredient of medicines [4]. Depending on humidity, Cf can absorb water to form hydrates of variable composition such as $Cf \cdot 1.34H_2O$, $Cf \cdot 3.7H_2O$ and $Cf \cdot 4.8H_2O$ [5]; as a result, the properties of the substance can change in the course of storage due to mutual transitions between different forms. Similarly, depending on temperature and humidity, mutual transitions occur between levofloxacin hemihydrate ($Levo \cdot 1.2H_2O$) and its two anhydrous crystalline forms δ and γ [6]. Even though Levo has a higher solubility ($4.8 \cdot 10^{-2}$ M, 298 K, and pH 7.4) than

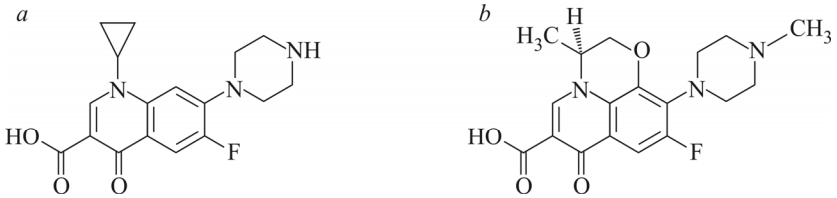


**Fig. 1.** Structural formulas of ciprofloxacin (*a*) and levofloxacin (*b*).

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Cf ($4.8 \cdot 10^{-4}$ M, 298 K, and pH 7.4) [7], it should be used in higher doses due to its poor permeability [8]. To improve the solubility and permeability of Cf and Levo, these compounds are transformed into various salts [9, 10]. Ciprofloxacin and levofloxacin are luminescent in aqueous solutions [11], and their crystalline salts can possess useful photoluminescence properties. In this work, ciprofloxacinium dichloride monohydrate, $CfH_2Cl_2 \cdot H_2O$ (**I**), and *tris*{hexachlorostannate(IV)} *tetrakis*(levofloxacinium) dichloride dihydrate, $(LevoH_2)_4[SnCl_6]_3Cl_2 \cdot 2H_2O$ (**II**), were obtained and characterized.

## EXPERIMENTAL

Ciprofloxacinium hydrochloride hydrate, $CfH \cdot 1.34H_2O$ (Ranbaxy Laboratories Ltd., India), levofloxacin hemihydrate, $Levo \cdot 1/2H_2O$ (Zhejiang Kangyu Pharmaceutical Co., Ltd, China), HCl (high-purity grade), and $SnCl_4 \cdot 5H_2O$ (high-purity grade) were used without additional purification.

**Synthesis of I.** 1 mL of concentrated HCl was added to $CfHCl \cdot 1.34H_2O$ (0.20 g) and the mixture was heated to 60-80 °C until the solid substance was completely dissolved. The solution was cooled to room temperature and maintained in an open vessel at room temperature for 48 h. The obtained bright yellow precipitate was filtered, washed in 1-2 mL of acetone, and dried in air to constant weight. Yield: 33%.

**Synthesis of II.** $SnCl_4 \cdot 5H_2O$ (1.41 g, 5.4 mmol) was added to $Levo \cdot 1/2H_2O$ (0.2 g, 0.54 mmol) that was previously dissolved in 2 mL of concentrated HCl. The obtained yellow solution was maintained in an open vessel at room temperature for 48 h. The resulting the yellow crystalline precipitate was filtered, washed in 1-2 mL of acetone, and dried in air to constant weight. Yield: 41%.

The monocrystals of **I**, **II** for XRD were taken from the obtained precipitate substance.

The powder XRD pattern **I** (Fig. 2) at room temperature (Bruker D8 ADVANCE diffractometer (Common Use Center of the Kirensky Institute of Physics SB RAS), VANTEC line detector, Cu$K_\alpha$ radiation) coincided with that calculated from the single-crystal data. Therefore, the data prove the phase identity of the polycrystalline sample to the studied single crystal.

**XRSCD.** This work was conducted using the equipment of the Krasnoyarsk Regional Research Equipment Centre of SB RAS The intensities of X-ray reflections in crystals **I** and **II** were measured on a SMART APEX II single-crystal diffractometer equipped with a Photon II CCD detector (Bruker AXS, Mo$K_\alpha$ radiation). The experimental absorption corrections were calculated by the multiscan method using the SADABS software [12]. The structures were solved by direct methods and refined using the SHELXTL program package [13]. The distances between hydrogen and oxygen atoms were refined with a restriction of 0.95 Å, all other hydrogen atoms were added geometrically. The refinement was stable and provided low *R*-index values (Table 1). Structures **I**, **II** were deposited with the Cambridge Crystallographic Data Centre (numbers CCDC-2000807 and CCDC-2000808). The data can be obtained at www.ccdc.cam.ac.uk/data_request/cif.

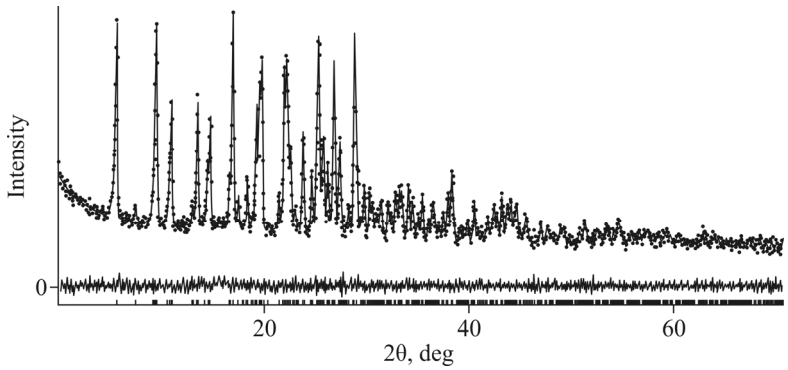


**Fig. 2.** Experimental, theoretical, and difference X-ray diffraction patterns of crystal **I**.

**TABLE 1.** Crystal Data and Structure Refinements for Crystals **I** and **II**

| Parameter | **I** | **II** |
|---|---|---|
| Empirical formula | $C_{17}H_{22}Cl_2FN_3O_4$ | $C_{72}H_{92}Cl_{20}F_4N_{12}O_{18}Sn_3$ |
| Crystal color | Yellow | Yellow |
| Crystal size, mm | 0.20×0.22×0.67 | 0.38×0.31×0.26 |
| Temperature, K | 296 | 296 |
| *a*, *b*, *c*, Å | 8.6389(11), 14.5486(19), 14.8605(19) | 12.4821(8), 13.8144(8), 15.2342(9) |
| α, β, γ, deg | 90.00, 91.914(3), 90.00 | 84.360(1), 79.265(1), 74.038(1) |
| *V*, Å$^3$ | 1866.7(4) | 2478.3(3) |
| Space group; *Z* | $P2_1/c$; 4 | $P\bar{1}$; 1 |
| *D*, g/cm$^3$ | 1.503 | 1.712 |
| μ, mm$^{-1}$ | 0.387 | 1.364 |
| $2\theta_{max}$, deg | 50 | 58 |
| Reflections collected / unique / with $F > 4\sigma(F)$ | 16850 / 3283 / 2251 | 31415 / 25789 / 21022 |
| Index ranges | $-10 \le h \le 10$, $-17 \le k \le 17$, $-17 \le l \le 17$ | $-17 \le h \le 17$, $-18 \le l \le 18$, $-20 \le l \le 20$ |
| $R_{int}$ | 0.1062 | 0.0204 |
| Weight scheme | $w = [\sigma^2 + (0.0494P)^2 + 1.1345P]^{-1}$, $P = (F_0^2 + 2F_c^2)/3$ | $w = [\sigma^2 + (0.0599P)^2 + 1.6551P]^{-1}$, $P = (F_0^2 + 2F_c^2)/3$ |
| Parameters | 333 | 1192 |
| $R1$ [$F_0 > 4\sigma(F_0)$]/all | 0.0571/0.0910 | 0.0491 / 0.0633 |
| *wR*2 (all) | 0.1374 | 0.1153 |
| *GOOF* | 1.044 | 0.954 |
| $\Delta\rho_{max}$ / $\Delta\rho_{min}$, e/Å$^3$ | 0.28 / –0.28 | 1.00 / –0.90 |
| $(\Delta\sigma)_{max}$ | 0.00 | 0.00 |
| Flack parameter | – | 0.033(11) |

## RESULTS AND DISCUSSION

The asymmetric part of the crystal cell of **I** contains cation $CfH_2^{2+}$, two free chlorine ions, and a water molecule. The geometric parameters of ion $CfH_2^{2+}$ (Fig. 3*a*) virtually coincide with those reported in [14, 15]. Similarly to other compounds containing fluoroquinolone cations, compound **I** contains an intramolecular hydrogen bond (H-bond) O1–H···O2 which weakens the electron accepting properties of atom O2 and limits its participation in the intermolecular H-bonds. The structure also contains an intramolecular H-bond C14–H14A···F with distance H14A···F equal to 2.20 Å (Table 2). The intermolecular H-bonds O–H···O, O–H···Cl, N–H···Cl, C–H···F, and C–H···Cl (Fig. 4) form a 3D supramolecular structure where ions $CfH_2^{2+}$ form paired chains directed along the [1,0,–1] axis. The paired chains are connected to each other by intermolecular interactions C12–H12A···F and C–H···Cl classified as weak H-bonds, as is indicated by the values of their geometric parameters [16]. Structure **I** has no π–π interactions between organic cations $CfH_2^{2+}$ (the minimum center-to-center distance exceeds 4.2 Å). However, there is an interaction between the lone electron pairs of two oxygen atoms and the π system of the C5,C6,C7,C8,C9,C10 ring of the organic ion. Thus, there is atom O1 located at a distance of ~3.36 Å along the normal to the center of the C5–C6–C7–C8–C9–C10 ring. This atom belongs to the $CfH_2^{2+}$ ion (connected by its symmetry center to the

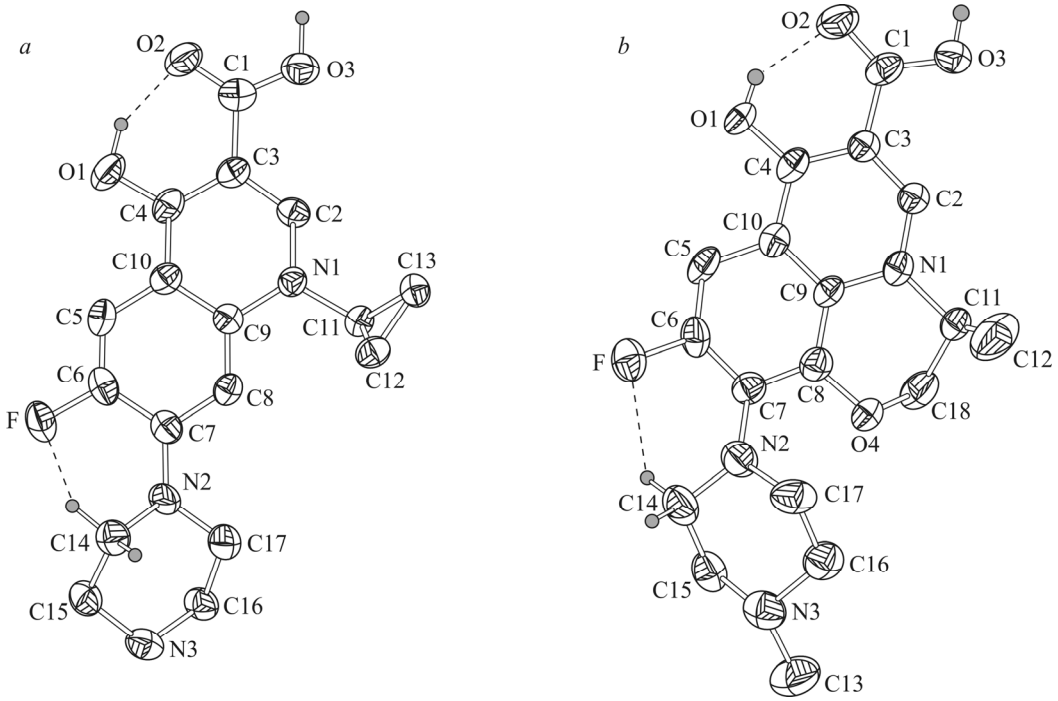


**Fig. 3.** Cations $CfH_2^{2+}$ (*a*) and $LevoH_3^{2+}$ (*b*) with atomic numbering. Hereinafter, hydrogen bonds are shown by dashed lines, thermal ellipsoids of all atoms (excluding hydrogen atoms) are set at a 50% probability level.

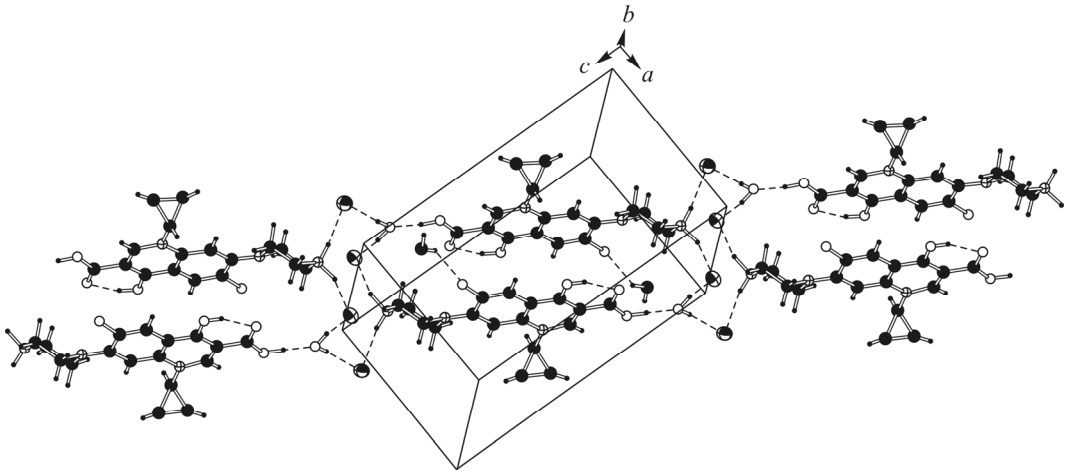


**Fig. 4.** Particle packing in crystal **I**. The C12 atom belonging to the neighboring double chain and forming the C–H···F H-bond is shown separately inside the cell.

initial cation) and is not involved in the intermolecular H-bonds. On the other side of the ring, at a distance of ~3.24 Å along the same normal, there is another oxygen atom, though belonging to a water molecule. Note that weak interactions "lone pair–π" may substantially increase the stability of the structure [17].

When exposed to UV irradiation, single crystals and polycrystals of **I** exhibit luminescence, i.e. compound **I** can be possibly used as a material for light emitting diodes (LEDs). Its yellow photoluminescence can be assigned to the π–π* electronic transition in the $CfH_2^+$ cation. Interestingly, fluoroquinolones as such are luminescent in aqueous solutions, which fact was used for their analytical determination and to study the acid-base equilibria [11]. Aqueous solutions of Cf demonstrate different types of luminescence in the visible spectrum, depending on the pH values (1.5-10.5). The strongest

**TABLE 2.** Geometric Parameters of Hydrogen Bonds in Structures **I**, **II**

| *D*–H···*A* contact | Distance, Å | | | ∠*D*H*A*, deg | Transformation for atom A |
|---|---|---|---|---|---|
| | *D*–H | H···*A* | *D*···*A* | | |
| | | | $CfH_2Cl_2·H_2O$ (**I**) | | |
| O1–H1···O2 | 0.945(10) | 1.73(2) | 2.594(4) | 151(4) | *x*, *y*, *z* |
| O3–H3···Ow | 0.951(10) | 1.542(11) | 2.492(4) | 177(5) | 1+*x*, *y*, *z* |
| Ow–Hw2···Cl1 | 0.946(10) | 2.175(13) | 3.118(3) | 174(6) | *x*, *y*, *z* |
| Ow–Hw1···Cl2 | 0.950(10) | 2.125(12) | 3.074(3) | 176(5) | *x*, *y*, *z* |
| N3–H3A···Cl1 | 0.89 | 2.23 | 3.114(3) | 172 | *x*, *y*, 1+*z* |
| N3–H3B···Cl2 | 0.89 | 2.26 | 3.101(3) | 158 | –*x*, 1–*y*, 1–*z* |
| C12–H12A···F | 0.97 | 2.51 | 3.326(5) | 142 | 1–*x*, *y*–1/2, 3/2–*z* |
| C14–H14A···F | 0.97 | 2.20 | 2.818(5) | 120 | *x*, *y*, *z* |
| C12–H12B···Cl2 | 0.97 | 2.92 | 3.560(4) | 124 | 1–*x*, 1–*y*, 1–*z* |
| C15–H15B···Cl2 | 0.97 | 2.81 | 3.764(4) | 169 | *x*, *y*, 1+*z* |
| C17–H17B···Cl1 | 0.97 | 2.87 | 3.788(4) | 159 | *x*, 1/2–*y*, 1/2+*z* |
| | | | $(LevoH_2)_4[SnCl_6]_3Cl_2·2H_2O$ (**II**) | | |
| OA1–HA1···OA2 | 0.82 | 1.88 | 2.602(9) | 146 | *x*, *y*, *z* |
| OA3–HA3···Ow1 | 0.82 | 1.80 | 2.612(9) | 173 | *x*, *y*, *z*–1 |
| NA3–HA3···Cl3C | 0.98 | 2.32 | 3.268(9) | 163 | *x*, *y*, *z* |
| CA2–HA2···Cl3B | 0.93 | 2.52 | 3.418(9) | 161 | *x*, *y*, *z*–1 |
| OB1–HB1···OB2 | 0.82 | 1.91 | 2.602(9) | 141 | *x*, *y*, *z* |
| OB3–HB3···Ow2 | 0.82 | 1.70 | 2.512(9) | 173 | *x*, *y*, *z* |
| NB3–HB3···Cl1A | 0.98 | 2.35 | 3.299(9) | 163 | *x*+1, *y*, *z*–1 |
| CB16–HB1L···Cl2A | 0.97 | 2.51 | 3.420(9) | 155 | *x*+2, *y*, *z*–1 |
| OC1–HC1···OC2 | 0.82 | 1.90 | 2.613(11) | 144 | *x*, *y*, *z* |
| OC3–HC3···Cl2 | 0.82 | 2.11 | 2.906(7) | 165 | *x*, *y*, *z* |
| NC3–HC3···Cl2 | 0.98 | 2.12 | 3.046(10) | 157 | *x*, *y*, *z*–1 |
| OD1–HD1···OD2 | 0.82 | 1.97 | 2.623(12) | 136 | *x*, *y*, *z* |
| OD3–HD3···Cl1 | 0.82 | 2.38 | 3.011(8) | 134 | *x*, *y*, *z* |
| ND3–HD3···Cl1 | 0.98 | 2.08 | 3.047(9) | 169 | *x*, *y*, *z*+1 |
| Ow1–Hw11···Cl2B | 0.94 | 2.53 | 3.287(8) | 138 | *x*+1, *y*, *z* |
| Ow1–Hw12···Cl2E | 0.95 | 2.89 | 3.694(7) | 148 | *x*+1, *y*, *z* |
| Ow2–Hw21···Cl2A | 0.93 | 2.61 | 3.128(7) | 117 | *x*+1, *y*, *z* |
| Ow2–Hw22···Cl2D | 0.93 | 2.40 | 3.233(9) | 148 | *x*+1, *y*, *z* |

luminescence is observed for the $CfH^+$ cation for pH = 4 with a peak at 500 nm and an excitation wavelength of 300 nm. The luminescence decreases in more acidic solutions (pH < 2). According to our data, the situation is different in the crystalline state. When exposed to UV radiation, ciprofloxacin shows no noticeable luminescence, $CfH^+$-containing hydrate $CfHCl·1.34H_2O$ shows weak pale-blue luminescence, while $CfH_2Cl_2·H_2O$ (**I**) shows intensely yellow luminescence. Thus, luminescence in this case is affected not only by the degree of Cf protonation but also by the compound's state of aggregation.

Thermal stability is an important characteristic of luminescent materials. Polycrystals **I** are stable up to 110 °C (Fig. 5) and decompose upon further heating with the release of HCl and $H_2O$, as is confirmed by the IR spectroscopy analysis of released gases. At the first thermolysis stage in the temperature region of 110-190 °C, the compound exhibits complete dehydration and the loss of about half of the theoretically expected HCl weight. The calculated weight loss ($\Delta m_{calc}$ = 13.0%, –HCl, $–H_2O$) was lower than its experimental value ($\Delta m_{exp}$ = 14.7%), possibly due to the presence of some impurities of richer hydrates in the initial sample, as in the case of Cf [5]. At the second thermolysis stage in the temperature

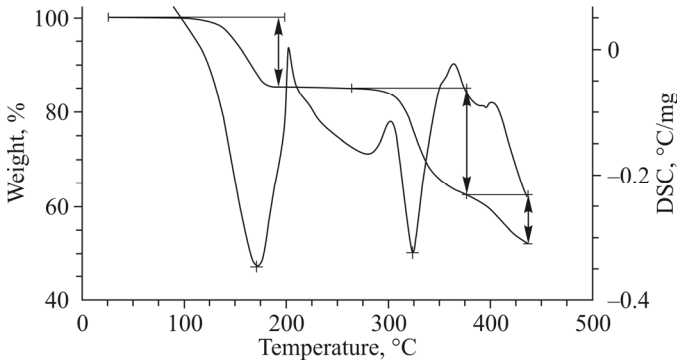


**Fig. 5.** TG and DSC curves for compound **I**.

region of 300-350 °C, the obtained residue undergoes oxidative decomposition associated with the release of HCl and $CO_2$. These decomposition stages are accompanied by the endoeffects at 171.1 °C and 323.9 °C, respectively. It was established earlier in [18] that the TG curves for CfHCl·1.34$H_2O$ almost coincide with those for anhydrous CfHCl at $T > 300$ °C. The TG curves for **I** at $T > 300$ °C are also almost coinciding with these curves. Therefore, CfHCl is most probably the product of the first thermolysis stage.

Crystals $(LevoH_2)_4[SnCl_6]_3Cl_2 \cdot 2H_2O$ (**II**) are triclinic. The structure contains four asymmetric independent levofloxacinium cations $LevoH_2^{2+}$, three anions $SnCl_6^{2-}$, two free chloride ions, and two water molecules. Typically of fluoroquinolones [19-21], two excess hydrogen ions are connected to the oxygen atoms of carboxyl and carbonyl groups of Levo (Fig. 3*b*). The bond lengths and bond angles in $LevoH_2^{2+}$ coincide with those determined earlier for other compounds containing this cation [19]. The Sn–Cl distances in the octahedral anions $SnCl_6^{2-}$ fall within the region of 2.393(3)-2.465(3) Å, in agreement with the data reported in [22]. Crystals **II** do not have the center of symmetry (space group *P*1), therefore, their absolute structure was determined. The rings with N, O, and C18 exhibit the *envelope* configuration in ions A, B, and C [19] and the *twist* configuration with an inversely oriented C18 in ion D.

All the ions and water molecules are connected by H-bonds O–H···O, O–H···Cl, and N–H···Cl. In terms of bonding, the independent $LevoH_2^{2+}$ ions can be divided into two groups. Cations A are connected to cations B by H-bonds O–H···O, O–H···Cl via ion $Sn2Cl_6^{2-}$ and two water molecules (Fig. 6), while atoms N3 of both cations are connected by H-bonds N–H···Cl with different $SnCl_6^{2-}$ groups. Moreover, ion B forms a terminal bond with $Sn1Cl_6^{2-}$, while ion A is connected to $Sn3Cl_6^{2-}$ on the one hand and to ion A from the neighboring pair AB (by a weak H-bond C–H···Cl) on the other hand. Thus, the network of H-bonds forms an infinite staircase-like but planar arrangement of AB pairs. Each of the cations C and D forms its own chains via H-bonds O–H···Cl and N–H···Cl: the first of them consists of ions C and Cl2, the second one consists of ions D and Cl1 (Fig. 7). The positions of hydrogen atoms at the carbon atoms suggest that no other intermolecular H-bonds are present in the structure. Similarly to **I**, structure **II** contains intramolecular H-bonds O1–H···O2 (Table 2).

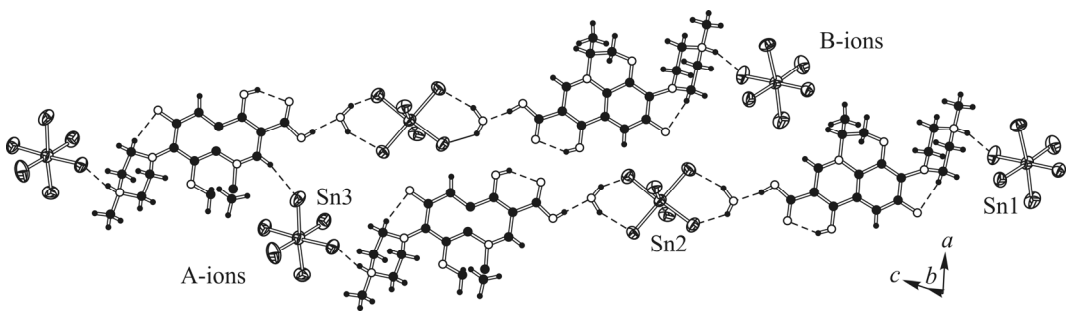


**Fig. 6.** Chains of cations A and B connected by $SnCl_6^{2-}$ ions.

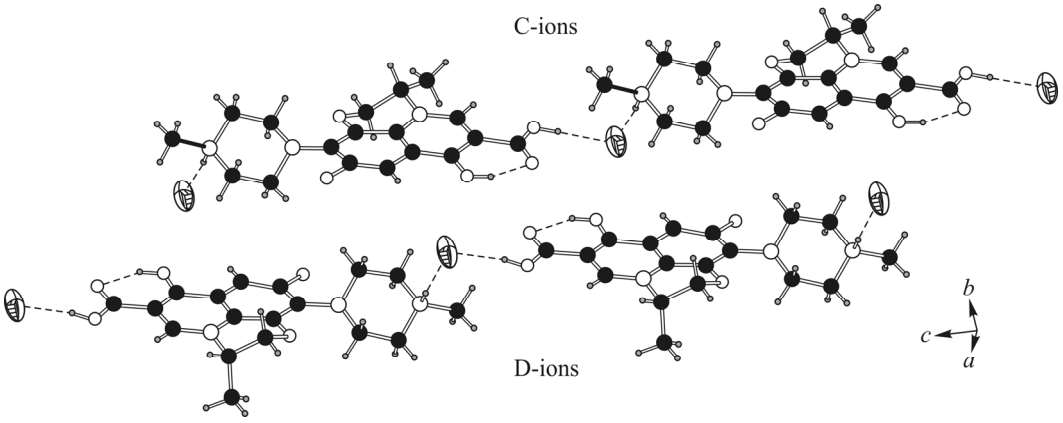


**Fig. 7.** Endless chains of cations C and D connected by $Cl^-$ ions.

The intramolecular interactions C14–H···F can be also viewed as weak H-bonds with distances H14···F varying from 2.22 Å to 2.44 Å and angles ∠C14–H···F varying from 107° to 119° [16]. In **II,** the π–π interactions between rings N1–C2–C3–C4–C10–C9 and C5–C6–C7–C8–C9–C10 connect levofloxacinium cations A and C or B and D in pairs, the center-to-center distances vary within the region of 3.826(5)-3.837 Å. Among the compounds of fluoroquinolones with Sn(IV) halides, monomers $(PefH_2)_4(H_3O)_4[SnCl_6]_3Cl_6{\cdot}11H_2O$ (Pef is pefloxacin) [20], $(EnrH_3[SnBr_{3.46}Cl_{2.54}]{\cdot}H_2O$ (Enr is enrofloxacin) [21], as well as polymer $K_2(Cf)_2[SnCl_6]$ containing neutral ciprofloxacin [23] were structurally characterized. Similarly to $LevoH_2^{2+}$ in **II**, ions $PefH_2^{2+}$ and $EnrH_2^{2+}$ are combined by H-bonds O3–H···Cl and N3–H···Cl into chains and are connected by π–π interactions.

When comparing structures **I** and **II,** note that atoms N3 and O3 in these cases are the main donors of intermolecular H-bonds, which is typical of ionic fluoroquinolone compounds [14, 19-21]. Each water molecule forms three H-bonds (two Ow–H···Cl bonds and one O3–H···Ow bond). In **I,** the H-bonded water molecules participate in the formation of all chains. In **II,** they participate in the formation of the chains with ions A and B rather than those with ions C and D. This fact can be explained by a relatively low water content in **II** ($2H_2O$ per $4LevoH_2^{2+}$ and $3SnCl_6^{2-}$). The chains are connected by weak H-bonds C–H···Cl in both structures and, additionally, by interaction C12–H12A···F in **I**. In contrast to **I**, crystals **II** do not exhibit photoluminescent properties in the visible region of the spectrum. In view of the fact that **I** exhibits strong photoluminescence, studying the supramolecular structure of crystalline fluoroquinolone compounds is interesting in terms of better understanding the mechanism that underlies and affects their luminescent properties.

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## CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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