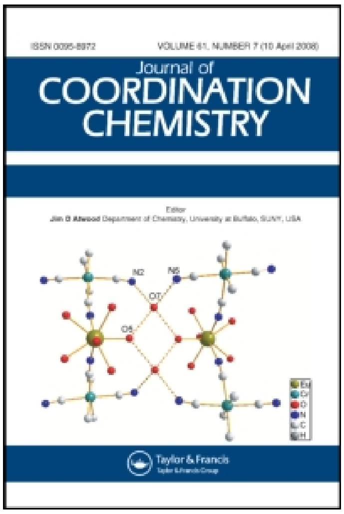

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Two binuclear complexes containing the enrofloxacinate anion: $Cd_2(C_{19}H_{21}N_3O_3F)_4(H_2O)_2 \cdot 4H_2O$ and $Pb_2(C_{19}H_{21}N_3O_3F)_4 \cdot 4H_2O$

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The syntheses and crystal structures of the closely related but non-isostructural $Cd_2(C_{19}H_{21}N_3O_3F)_4(H_2O)_2 \cdot 4H_2O$ (**1**) and $Pb_2(C_{19}H_{21}N_3O_3F)_4 \cdot 4H_2O$ (**2**) are described, where $C_{19}H_{21}N_3O_3F^-$ is enrofloxacinate (enro). Both compounds contain centrosymmetric, binuclear, neutral complexes incorporating a central diamond-shaped M_2O_2 ($M = Cd$, Pb) structural unit. The Cd^{2+} coordination polyhedron in **1** is a CdO_6 trigonal prism, including one coordinated water. The Pb^{2+} coordination polyhedron in **2** can be described as a very distorted square-based PbO_5 pyramid, although two additional short $Pb \cdots O$ (< 3.1 Å) contacts are also present. In the crystal of the cadmium complex, $O–H \cdots O$ hydrogen bonds lead to a layered structure. In the lead compound, $O–H \cdots O$ and $O–H \cdots N$ interactions lead to chains in the crystal. Crystal data: **1**: $C_{76}H_{96}Cd_2F_4N_{12}O_{18}$, $M_r = 1766.45$, triclinic, $P\bar{1}$, $a = 12.185(2)$ Å, $b = 12.306(3)$ Å, $c = 14.826(3)$ Å, $\alpha = 68.15(3)^\circ$, $\beta = 70.28(3)^\circ$, $\gamma = 86.11(3)^\circ$, $V = 1938.2(7)$ Å^3, $Z = 1$, $T = 298$ K, $R(F) = 0.030$, $wR(F^2) = 0.079$. **2**: $C_{76}H_{88}F_4N_{12}O_{16}Pb_2$, $M_r = 1920.00$, triclinic, $P\bar{1}$, $a = 12.0283(4)$ Å, $b = 12.7465(4)$ Å, $c = 13.0585(4)$ Å, $\alpha = 83.751(1)^\circ$, $\beta = 74.635(1)^\circ$, $\gamma = 81.502(1)^\circ$, $V = 1904.3(1)$ Å^3, $Z = 1$, $T = 298$ K, $R(F) = 0.021$, $wR(F^2) = 0.049$.

Keywords: Bimetallic complex; Trigonal prismatic geometry; Crystal structure

1. Introduction

Enrofloxacin (Henro), $C_{19}H_{22}FN_3O_3$ (systematic name 1-cyclopropyl-7-(4-ethylpiperazin-1-yl)-6-fluor-4-oxo-1,4-dihydrochinolin-3-carboxylic acid), is a member of the

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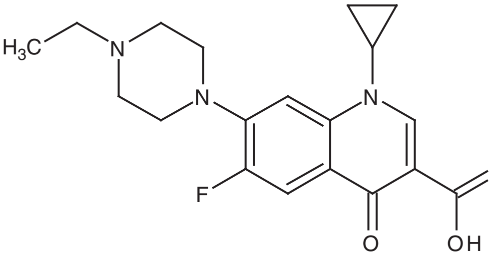
fluoroquinolone (flqu) family of antibiotics [1],



Henro

widely used in veterinary clinical practice because of its wide antibiotic spectrum and its excellent bactericidal activity [2, 3]. Our interest in enrofloxacin and related fluoroquinolones is focused on their potential as multidentate/bridging ligands in coordination chemistry, and we have described the syntheses and structures of polymeric $[Ni(C_{17}H_{17}FN_3O_3)_2]_n$ [4] and monomeric $Zn(H_2O)(C_{17}H_{19}FN_3O_3)_2 \cdot 2H_2O$ [5]. Chelation of a flqu-type molecule to a metal ion and its resulting binding to a DNA host are key to its antibacterial activity [6]. Xiao *et al.* [7] have prepared and characterized a wide range of monomeric complexes and polymeric networks based on different metal ions and the fluoroquinolone ciprofloxacin ($C_{17}H_{18}FN_3O_3$; Hcip) and a review [8] appeared several years ago, giving a comprehensive survey of this area of coordination chemistry. More recently, Yu *et al.* [9] have described complexes of Co^{II} and Fe^{III}, Cu^{II} [10], Zn^{II} and Co^{II} [11], Cu^{II} and Mn^{II} [12], and Zn^{II} [13] with flqu-type ligands, some of which show antibacterial activity. As part of our ongoing synthetic and structural studies of metal complexes of fluoroquinolones [4, 5], we now report the syntheses and crystal structures of two bimetallic complexes containing large divalent metals and enrofloxacinate, namely: $Cd_2(enro)_4(H_2O)_2 \cdot 4H_2O$ (**1**), and $Pb_2(enro)_4 \cdot 4H_2O$ (**2**), which both demonstrate unusual metal coordination geometries.

2. Experimental

2.1. *Synthesis*

A mixture of $Cd(CH_3COO)_2 \cdot 3H_2O$ (0.25 mmol), enrofloxacin (0.5 mmol), sodium hydroxide (1 mmol), and water (15 mL) was stirred for 30 min in air. The mixture was then transferred to a 23-mL Teflon-lined hydrothermal bomb. The bomb was heated to 453 K for 72 h under autogenous pressure. Upon cooling, the bomb was opened and colorless slabs and blocks of **1** were recovered from the reaction mixture by vacuum filtration and rinsing with water. Compound **2** was prepared by the same hydrothermal method, with $Pb(CH_3COO)_2 \cdot 3H_2O$ (0.25 mmol) used in place of the cadmium acetate trihydrate and the bomb heated to 433 K for 96 h under autogenous pressure. Upon cooling and recovery of the product, colorless blocks of **2** were obtained. Both compounds appear to be indefinitely stable when stored in dry air.

2.2. *Characterization*

Elemental analysis data for **1** and **2** were satisfactory: Anal. Calcd for $C_{36}H_{96}Cd_2F_4N_{12}O_{18}$ (%): C 33.62, H 7.52, and N 13.07 Found (%): C 33.53, H 7.48, and N 12.88. Anal. Calcd for $C_{36}H_{92}F_4N_{12}O_{16}Pb_2$ (%): C 30.03, H 6.44, N 11.68; Found (%): C 29.85, H 6.37, N 11.56. IR (KBr disk, cm^{-1}) for **1**: 3398 (br), 2835 (w), 1624 (s), 1570 (s), 1481 (m), 1384 (s), 1292 (s), 1257 (s), 1178 (w), 1022 (m), and 947 (s); for **2**: 3393 (br), 1622 (s), 1562 (s), 1479 (m), 1375 (s), 1298 (s), 1256 (s), 1012 (m), and 941 (m). These signals are in agreement with the reported IR spectra of similar compounds [7]. The peaks at 1624 and 1481 cm^{-1} for **1** (1622 and 1479 cm^{-1} for **2**) are characteristic of a deprotonated and coordinated carboxylate in fluoroquinolones [9], as confirmed by the crystal structures (*vide infra*).

2.3. *Structure determinations*

Intensity data for **1** (colorless slab, $0.36 \times 0.28 \times 0.21\,mm^3$) and **2** (colorless block, $0.34 \times 0.26 \times 0.19\,mm^3$) were collected at room temperature using a Bruker Apex2 CCD diffractometer (Mo-Kα radiation, $\lambda = 0.71073$ Å). Multi-scan absorption corrections were made with SADABS during data reduction. The triclinic structures were routinely solved by direct methods with SHELXS-97 and the structure models were refined against $|F^2|$ using SHELXL-97 [14]. In the lead compound, one pendant cyclopropane is disordered over two sets of sites in a 0.777 (8) : 0.223 (8) ratio. For both structures, the hydrogens bonded to carbon were geometrically placed and refined as riding with the constraint $U_{iso}(H) = 1.2{-}1.5U_{eq}(C)$ applied. The N- and O-bound hydrogens were located in difference maps and refined as riding in their as-found relative positions. Crystal data for **1** and **2** are summarized in table 1 and full details are available in the "Supplementary material" (cif format).

Table 1. Crystallographic and data-collection parameters.

	1	**2**
Empirical formula	$C_{76}H_{96}Cd_2F_4N_{12}O_{18}$	$C_{76}H_{92}F_4N_{12}O_{16}Pb_2$
Formula weight	1766.45	1920.00
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$ (No. 2)	$P\bar{1}$ (No. 2)
Unit cell dimensions (Å, °)		
a	12.185(2)	12.0283(4)
b	12.306(3)	12.7465(4)
c	14.826(3)	13.0585(4)
α	68.15(3)	83.751(1)
β	70.28(3)	74.635(1)
γ	86.11(3)	81.502(1)
Volume (Å^3), Z	1938.2(7), 1	1904.25(10), 1
Calculated density (g cm^{-3})	1.513	1.674
Absorption coefficient (mm^{-1})	0.636	4.500
Data/restraints/parameters	7568/0/507	6969/1/502
$R(F)$	0.030	0.021
$wR(F^2)$	0.079	0.049
Largest difference peak and hole (e Å^{-3})	−0.37 and +1.05	−0.45 and +0.57

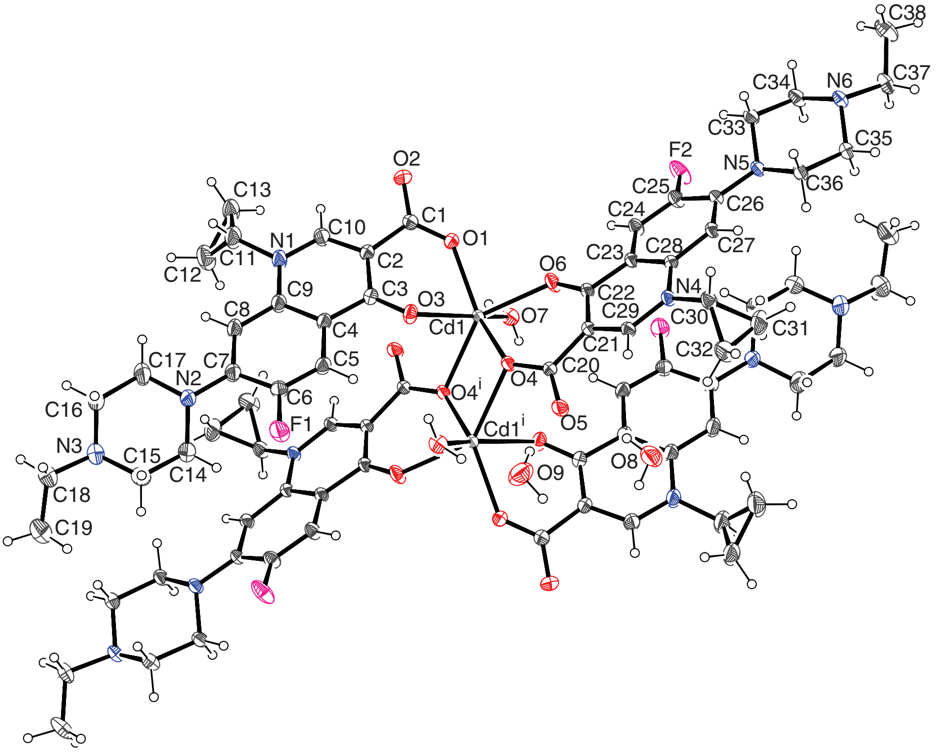


Figure 1. View of the molecular structure of **1** with displacement ellipsoids for non-hydrogen atoms drawn at the 30% probability level. Symmetry code: ^{i} $1-x$, $-y$, $1-z$.

3. Results and discussion

3.1. *Crystal structures*

The molecular structure of **1** is shown in figure 1 and selected geometrical data are given in table 2. Compound **1** contains a neutral, centrosymmetric, binuclear complex built up from two Cd^{2+} ions, four $C_{19}H_{21}N_3O_3F^-$ enrofloxacinates and two coordinated waters, of overall formula $[Cd_2(enro)_4(H_2O)_2]$. Four uncoordinated water molecules (O8 and O9) per complex complete the structure of **1**. The cadmium is coordinated by one O,O-bidentate enro (containing C1), coordinating from its quinoline (O3) and carboxylate (O1) to generate a six-membered chelate ring, with an irregular shape: C1, C2, C3, and O3 are almost co-planar (r.m.s. deviation = 0.0004 Å) and O1 and Cd1 are displaced from their mean plane in the same direction by 0.517(5) and 1.402(5) Å, respectively.

A second O,O-bidentate enro (containing C20) bonds to cadmium from the equivalent (O6 and O4) oxygens (the resulting six-membered chelate ring is close to planar, with an r.m.s. deviation of 0.050 Å for the six atoms) and also bonds to the other Cd^{2+} from its other carboxylate oxygen, O4 (figure 1). The symmetry-equivalent $O4^{i}$ atom ($i = 1-x$, $-y$, $1-z$) and a bound water (O7) complete the cadmium coordination sphere, which results in a distinctive trigonal prismatic geometry (figure 2). The dihedral angle between O1/O6/O7 and O3/O4/$O4^{i}$ ($i = 1-x$, $-y$, $1-z$) planes

Table 2. Selected geometrical data (Å and °) for **1**.

Cd1–O1	2.2189(18)	Cd1–O6	2.2727(17)	
Cd1–O7	2.2895(19)	Cd1–O4	2.3135(16)	
Cd1–O3	2.3215(18)	Cd1–O4^{i}	2.3336(16)	
O1–Cd1–O6	87.29(7)	O1–Cd1–O7	89.80(8)	
O6–Cd1–O7	80.60(7)	O1–Cd1–O4	137.66(7)	
O6–Cd1–O4	76.32(6)	O7–Cd1–O4	124.49(7)	
O1–Cd1–O3	78.57(7)	O6–Cd1–O3	125.11(7)	
O7–Cd1–O	150.53(6)	O4–Cd1–O3	79.68(6)	
O1–Cd1–O4^{i}	132.61(7)	O6–Cd1–O4^{i}	139.47(6)	
O7–Cd1–O4^{i}	91.24(7)	O4–Cd1–O4^{i}	76.11(6)	
O3–Cd1–O4^{i}	77.71(7)	Cd1–O4–Cd1^{i}	103.89(6)	
O7–H1 ⋯ O9^{i}	0.88	1.89	2.745(3)	165
O7–H2 ⋯ N6^{ii}	0.91	1.96	2.863(3)	175
O8–H3 ⋯ O2^{iii}	0.85	1.96	2.794(3)	168
O8–H4 ⋯ O5	0.85	2.03	2.869(3)	168
O9–H5 ⋯ O5	0.85	1.99	2.833(3)	170
O9–H6 ⋯ O2^{iii}	0.85	1.89	2.731(3)	169

For the hydrogen bonds, the four values refer to the O–H, H ⋯ O and O ⋯ O separations, and the O–H ⋯ O angle, respectively. Symmetry codes: ^{i} $1-x$, $-y$, $1-z$; ^{ii} x, $y-1$, z; and ^{iii} $x-1$, y, z.

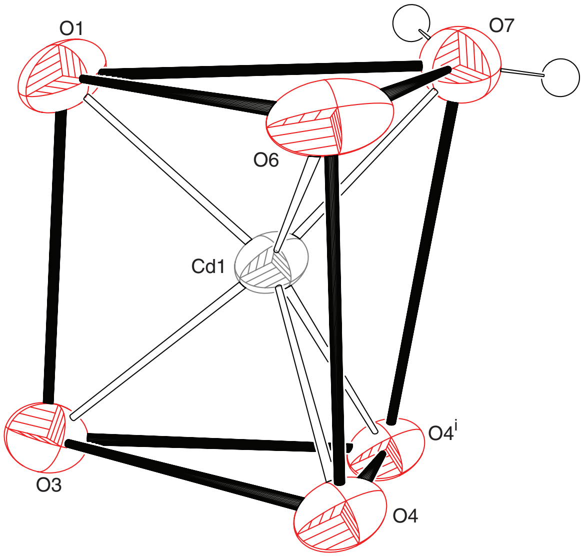


Figure 2. Detail of **1** showing the trigonal prismatic coordination geometry of cadmium (displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level. Symmetry code: ^{i} $1-x$, $-y$, $1-z$).

defining the ends of the prism is 9.63(12)° and the volume of the CdO_6 polyhedron is 12.72 Å^3. The mean O ⋯ O separation for the O_3 triangles is 2.999 Å and the mean O ⋯ O separation for the edges of the prism is 3.005 Å. The bond valence sum (BVS) for cadmium is 2.11, compared to an expected [15] value of 2.00. The two Cd1 and O4 atoms make up a diamond-shaped linking unit (Cd ⋯ Cd = 3.6592(9) Å, O ⋯ O = 2.865(3) Å) at the center of the molecule: crystal symmetry dictates that this unit is planar.

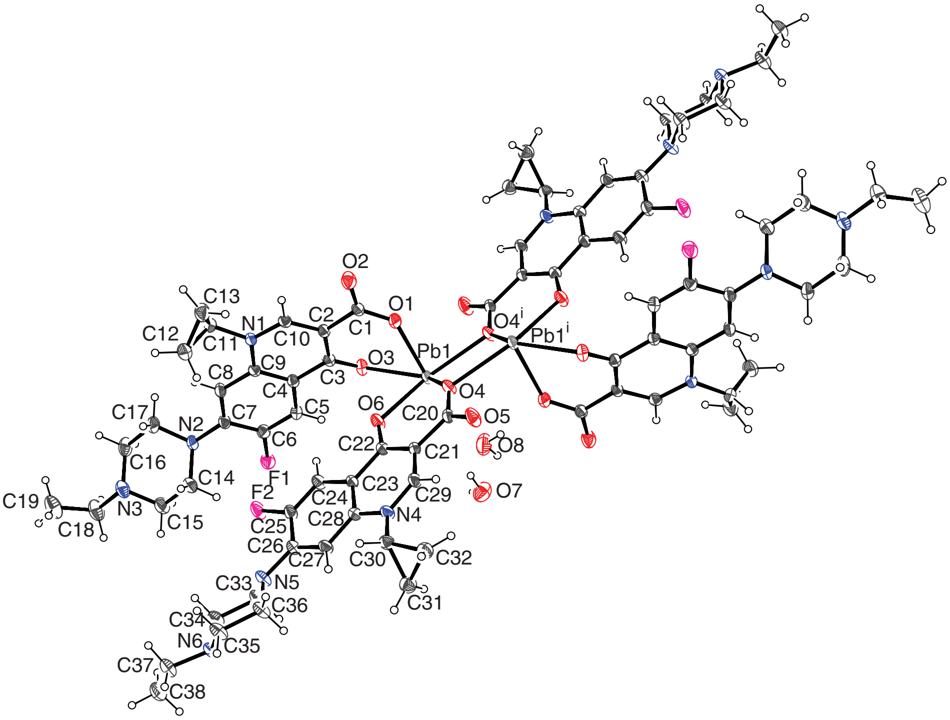


Figure 3. View of the molecular structure of **2** with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level. Symmetry code: ^{i} $2-x$, $2-y$, $1-z$.

For C1 in **1**, the C2–C10/N1 atoms forming the fused ring system are close to planar (r.m.s. deviation = 0.017 Å) and the bond-angle sum at N1 is 359.8°, implying sp^2 hybridization for nitrogen. The C2–C10/N1 grouping makes dihedral angles of 50.3(3)° and 28.6(5)° with the C11/C12/C13 *cyclo*-pentane ring and the C1/O1/O2 carboxylate group, respectively. The fluorine is displaced from the C2–C10/N1 plane by −0.138(3) Å and the Cd is displaced by 1.368(4) Å. The N2/N3/C14–C17 piperazine ring adopts a typical chair conformation with the N-bonded substituents in the equatorial positions. For the C20 enro, the r.m.s. deviation from the mean plane for the C21–C29/N4 fused-ring system is 0.044 Å and the bond-angle sum for N4 is 360.0°. The displacements of F2 and Cd1 from the C21–C29/N4 plane are −0.232(3) and 0.346(3) Å, respectively. The dihedral angles between C21–C29/N4 and the C30/C31/C32 and C20/O4/O5 groupings are 55.4(3)° and 20.8(4)°, respectively. The piperazine ring in the C20 molecule adopts the same conformation as in the C1 species. The packing for **1** is consolidated by a network of O–H · · · O hydrogen bonds involving both the bound and uncoordinated water molecules (table 2), resulting in layers lying parallel to (0 0 1).

The molecular structure of **2** is shown in figure 3 and selected geometrical data are given in table 3. Compound **2** contains a neutral, centrosymmetric, bimetallic complex built up from two Pb^{2+} ions and four enro anions, of overall formula $[Pb_2(enro)_4]$; thus it is not isostructural with **1**. Four uncoordinated water molecules (O7 and O8) per complex complete the structure of **2**. The lead is coordinated by one O,O-bidentate enro

Table 3. Selected geometrical data (Å and °) for **2**.

Pb1–O1	2.340(2)	Pb1–O4	2.401(2)	
Pb1–O6	2.453(2)	Pb1–O3	2.5033(19)	
Pb1–O4^{i}	2.5494(19)	Pb1–O8	2.905(3)	
Pb1–O5^{i}	3.017(2)			
O1–Pb1–O4	80.60(8)	O1–Pb1–O6	88.50(8)	
O4–Pb1–O6	70.70(7)	O1–Pb1–O3	72.90(7)	
O4–Pb1–O3	136.31(7)	O6–Pb1–O3	74.44(7)	
O1–Pb1–O4^{i}	80.46(7)	O4–Pb1–O4^{i}	69.33(8)	
O6–Pb1–O4^{i}	139.73(7)	O3–Pb1–O4^{i}	136.11(7)	
O1–Pb1–O8	149.41(8)	O4–Pb1–O8	69.28(8)	
O6–Pb1–O8	86.55(8)	O3–Pb1–O8	133.89(7)	
O4^{i}–Pb1–O8	84.00(7)	O1–Pb1–O5^{i}	75.97(8)	
O4–Pb1–O5^{i}	113.17(6)	O6–Pb1–O5^{i}	162.75(7)	
O3–Pb1–O5^{i}	93.53(6)	O4^{i}–Pb1–O5^{i}	45.64(6)	
O8–Pb1–O5^{i}	110.64(8)	Pb1–O4–Pb1^{i}	110.67(8)	
O7–H1 ··· N6^{ii}	0.85	2.15	2.963(4)	162
O7–H3 ··· O2^{iii}	0.85	1.94	2.782(4)	169
O8–H2 ··· O7^{iv}	0.85	2.11	2.853(4)	145
O8–H4 ··· O1^{i}	0.85	2.08	2.915(3)	166
C11–H11A ··· O5^{v}	0.98	2.39	3.213(4)	141

For the hydrogen bonds, the four values refer to the O–H, H ··· O and O ··· O separations, and the O–H ··· O angle, respectively. Symmetry codes: ^{i} $2-x, 2-y, 1-z$; ^{ii} $x+1, y, z$; ^{iii} $x, y, z+1$; ^{iv} $2-x, 2-y, 2-z$; and ^{v} $x, y-1, z$.

(containing C1) coordinating from its quinoline (O3) and carboxylate (O1), thus generating a six-membered chelate ring with an irregular shape: C1, C2, C3, and O3 are close to co-planar (r.m.s. deviation = 0.029 Å) and O1 and Pb1 are displaced by 0.337(5) and 1.273(6) Å, respectively. The second O,O-bidentate enro (containing C20) chelates from equivalent O6 and O4 to generate another six-membered chelate ring, which is an envelope, with C20/C21/C22/O4/O6 almost co-planar (r.m.s. deviation = 0.017 Å) and Pb1 displaced from the other five atoms by 0.798(4) Å. This C20 molecule also bonds to the other Pb from its other carboxylate oxygen (O4). Despite this close similarity to the coordination modes of the two enro anions in **1**, the resulting geometry of lead in **2** is very different: the four enro oxygens and the symmetry generated O4^{i} ($i = 2-x, 2-y, 1-z$) result in what can be described as a very distorted PbO_5 square-based pyramid (figure 4), with O1 in the apical site. This coordination geometry is not uncommon for lead, and can, in part, be related to the stereochemical activity of the (unseen) $6s^2$ lone pair presumed to reside on the metal ion [16]. In addition to the five bonded oxygens, two further oxygens are within 3.1 Å of the metal [Pb1 ··· O8 = 2.905(3) Å, Pb1 ··· O5^{i} = 3.017(2) Å ($i = 2-x, 2-y, 1-z$)], which compare with an expected van der Waals' separation of 3.86 Å for these species. If these two additional O atoms are considered, the lead coordination can only be described as irregular (figure 4). For the five near-neighbor oxygens, the lead BVS is 1.97 (expected 2.00); if all seven O atoms are included in the BVS calculation, the value rises to 2.17. Again, the central core of the molecule consists of a planar, diamond-shaped Pb_2O_2 unit with Pb ··· Pb = 4.0729(2) Å and O ··· O = 2.819(3) Å.

For C1 in **2**, the r.m.s. deviation from the mean plane of C2–C10/N1 is 0.042 Å and the bond-angle sum at N1 is 359.9°. The C2–C10/N1 grouping makes dihedral angles of 53.89(17)° and 11.2(4)° with the C11/C12/C13 cyclopentane ring and the C1/O1/O2 carboxylate, respectively. The fluorine is displaced from the C2–C10/N1 plane by

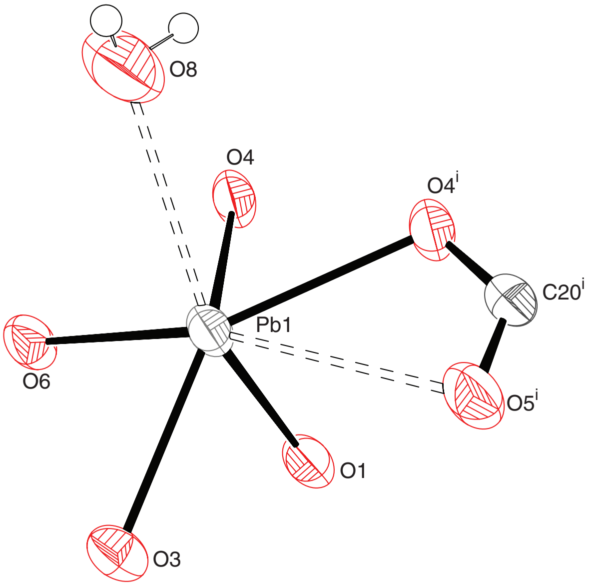


Figure 4. Detail of **2** showing the coordination of lead (displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level. Symmetry code: ^{i} $2-x$, $2-y$, $1-z$).

0.070(3) Å and Pb is displaced from the same plane by 1.090(4) Å. The N2/N3/C14–C17 piperazine ring adopts a typical chair conformation with the N-bonded substituents in the equatorial positions. For the C20 enro, the r.m.s. deviation from the mean plane for the C21–C29/N4-fused ring system is 0.038 Å and the bond-angle sum for N4 is 352.8°. The displacements of F2 and Cd1 from the C21–C29/N4 plane are –0.190(4) and 0.883(4) Å, respectively. The dihedral angles between C21–C29/N4 and C30/C31/C32 (major disorder component) and C20/O4/O5 are 53.7(3)° and 6.5(4)°, respectively. The piperazine ring in the C20 molecule adopts the same chair conformation as does the C1 species.

The packing for **2** is consolidated by a network of O–H···O and O–H···N hydrogen bonds (table 3), resulting in chains propagating in [10$\bar{1}$] and a short C–H···O interaction is also present.

3.2. *Discussion*

The new divalent metal–enrofloxacinate complexes, **1** and **2**, share close similarities in terms of their centrosymmetric, bimetallic structures, and the O,O-bidentate coordination modes of enro, but the resulting metal coordination polyhedra are completely different.

No other complexes containing lead cations and a fluoroquinolone-family ligand have yet been described, but several cadmium complexes are known. In the molecular complex $CdCl_2(Hcipro)_2 \cdot 4H_2O$ [17], Cd^{2+} adopts a *trans*-$CdCl_2O_4$ octahedral geometry, arising from coordination by two O,O-bidentate ligands and two chlorides. In $[Cd(cipro)_2]_n \cdot 2.5nH_2O$ [7], a layered coordination network arises from coordination of the metal ion by two O,O-bidentate cipro anions and two N-bonded cipro anions. Further variety can occur when a second bridging ligand such 3,3′,4,4′-benzophenonetetracarboxylate (bptc) also bonds to Cd^{2+}, as in $[Cd_2(Hcipro)_2(bptc)(H_2O)_2]_n \cdot 8nH_2O$ [7], but again an octahedral geometry arises for the metal. The trigonal prismatic geometry for cadmium in **1** is very unusual but not unprecedented: another example is afforded by $Cd(NCS)_2(C_{17}H_{20}N_4)$ [18], in which an N_6 donor set leads to a CdN_6 trigonal prism; the authors describe the distortions that may occur between the limiting cases of octahedral and trigonal prismatic geometry in detail.

Supplementary material

Full details of the crystal structures of **1** (CCDC 770537) and **2** (CCDC 770538) in cif format are available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK, or Email: deposit@ccdc.cam.ac.uk

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