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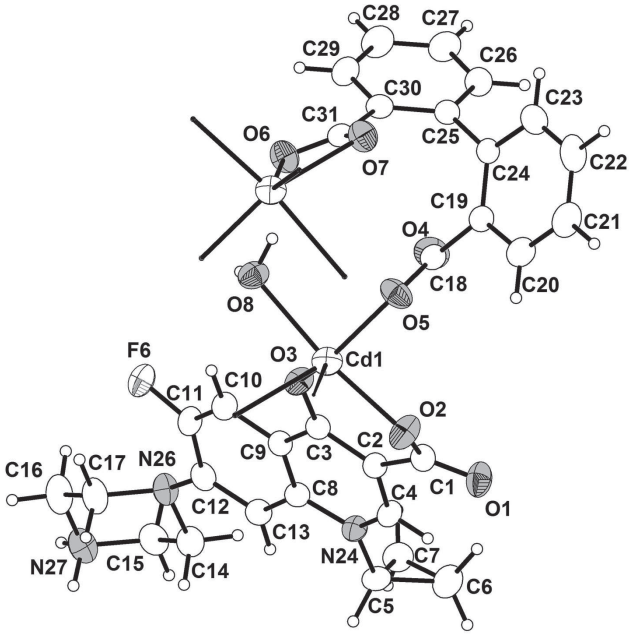
Crystal structure of aqua(μ$_2$-biphenyl-2,2′-dicarboxylato-κ^3*O*,*O*′:*O*″)-(μ$_2$-1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylato-κ^2*O*,*O*′)cadmium(II) 1.5 hydrate, $C_{62}H_{60}N_6Cd_2O_{19}F_2$



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Abstract

$C_{62}H_{60}N_6Cd_2O_{19}F_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.749(4)$ Å, $b = 12.884(5)$ Å, $c = 13.869(5)$ Å, $\alpha = 70.103(4)°$, $\beta = 80.201(4)°$, $\gamma = 68.648(4)°$, $V = 1523.3(10)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0549$, $wR_{ref}(F^2) = 0.1716$, $T = 100(2)$ K.

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A part of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block Size 0.25 × 0.22 × 0.18 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	7.8 cm^{-1}
Diffractometer, scan mode:	Bruker, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10537, 5343, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4717
$N(param)_{refined}$:	424
Programs:	OLEX2 [6], SHELX [7], Bruker programs [8]

Source of material

A mixture of $Cd(CH_3COO)_2{\cdot}2H_2O$ (0.25 mmol), ciprofloxacin (0.5 mmol), biphenyl-2,2′dicarboxylic acid (0.50 mmol), sodium hydroxide (2 mmol), and water (15 mL) was stirred for 20 min in air. The mixture was then transferred to a 25 mL Teflon-lined hydrothermal bomb. The bomb was heated to 443 K for 96 h under autogenous pressure. After cooling, colorless single crystals of title compound were obtained from the reaction mixture.

Experimental details

The carbon-bound H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and were included in the refinement in

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.48154(4)	0.19229(3)	0.37160(3)	0.03951(18)
O5	0.4389(5)	0.1598(4)	0.5388(3)	0.0556(10)
F6	0.0762(4)	0.3646(3)	−0.0651(3)	0.0552(8)
O2	0.5225(5)	0.3570(3)	0.3508(3)	0.0574(11)
O6	0.7265(4)	0.0625(3)	0.4268(3)	0.0542(9)
O3	0.3415(4)	0.3068(3)	0.2349(3)	0.0458(8)
O8	0.3622(5)	0.0604(4)	0.3822(3)	0.0531(10)
H8A	0.2929	0.0939	0.3381	0.080*
H8B	0.3227	0.0395	0.4441	0.080*
O4	0.2116(4)	0.2825(4)	0.5274(3)	0.0612(11)
O9	0.3334(6)	0.5998(7)	0.5021(4)	0.125(3)
H9A	0.3811	0.5916	0.4446	0.188*
H9B	0.3951	0.5900	0.5455	0.188*
O1	0.4657(5)	0.5472(4)	0.3168(3)	0.0593(11)
O7	0.6621(4)	0.0795(3)	0.2763(3)	0.0546(10)
N24	0.3327(5)	0.6269(3)	0.0336(3)	0.0392(9)
N26	0.0997(5)	0.5646(4)	−0.2162(3)	0.0457(10)
N27	0.0424(5)	0.6376(5)	−0.4292(4)	0.0559(13)
H27A	−0.0223	0.6613	−0.4798	0.067*
H27B	0.1342	0.6353	−0.4603	0.067*
C1	0.4653(6)	0.4606(5)	0.2957(4)	0.0409(11)
C22	0.3177(8)	0.1460(6)	0.9017(5)	0.0615(16)
H22	0.3198	0.1305	0.9736	0.074*
C2	0.3970(5)	0.4834(4)	0.1972(4)	0.0369(10)
C30	0.0994(5)	0.0554(5)	0.6684(4)	0.0415(11)
C19	0.3135(5)	0.1889(4)	0.6912(4)	0.0372(10)
C3	0.3415(5)	0.4057(4)	0.1755(4)	0.0352(10)
C9	0.2782(5)	0.4493(4)	0.0750(4)	0.0358(10)
C24	0.2025(5)	0.1530(4)	0.7575(4)	0.0365(10)
C11	0.1541(6)	0.4225(5)	−0.0446(4)	0.0422(11)
C23	0.2063(7)	0.1327(5)	0.8619(4)	0.0507(13)
H23	0.1309	0.1092	0.9072	0.061*
C12	0.1629(5)	0.5276(5)	−0.1219(4)	0.0411(11)
C8	0.2787(5)	0.5561(4)	0.0042(4)	0.0373(10)
C27	−0.1819(6)	0.2080(6)	0.6930(5)	0.0608(16)
H27	−0.2779	0.2600	0.7022	0.073*
C25	0.0798(6)	0.1377(4)	0.7183(4)	0.0410(11)
C18	0.3180(5)	0.2136(4)	0.5771(4)	0.0389(10)
C28	−0.1614(6)	0.1272(6)	0.6426(6)	0.0645(17)
H28	−0.2431	0.1240	0.6163	0.077*
C14	0.0951(6)	0.6837(5)	−0.2832(4)	0.0482(12)
H14A	0.1956	0.6829	−0.3118	0.058*
H14B	0.0583	0.7394	−0.2424	0.058*
C29	−0.0216(7)	0.0512(5)	0.6307(5)	0.0539(14)
H29	−0.0076	−0.0047	0.5964	0.065*
C20	0.4253(6)	0.2012(5)	0.7327(5)	0.0510(13)
H20	0.5026	0.2233	0.6887	0.061*
C17	0.1487(7)	0.4806(5)	−0.2752(4)	0.0543(14)
H17A	0.1478	0.4027	−0.2294	0.065*
H17B	0.2509	0.4732	−0.3035	0.065*
C21	0.4251(7)	0.1820(6)	0.8359(5)	0.0613(16)
H21	0.5002	0.1937	0.8624	0.074*
C13	0.2266(5)	0.5928(4)	−0.0932(4)	0.0396(11)
H13	0.2347	0.6636	−0.1411	0.047*
C6	0.2759(8)	0.8434(5)	0.0092(5)	0.0610(16)
H6A	0.3210	0.9057	−0.0220	0.073*
H6B	0.2541	0.8257	0.0843	0.073*
C31	0.7532(6)	0.0333(5)	0.3455(4)	0.0437(12)
C15	−0.0059(6)	0.7223(5)	−0.3698(4)	0.0509(13)
H15A	−0.1079	0.7294	−0.3410	0.061*
H15B	−0.0059	0.8002	−0.4161	0.061*
C10	0.2125(6)	0.3823(5)	0.0475(4)	0.0423(11)
H10	0.2098	0.3092	0.0940	0.051*
C7	0.1721(7)	0.8329(5)	−0.0541(4)	0.0512(13)
H7A	0.0866	0.8092	−0.0176	0.061*
H7B	0.1535	0.8891	−0.1239	0.061*
C5	0.3217(6)	0.7441(4)	−0.0359(4)	0.0435(12)
H5	0.3950	0.7480	−0.0958	0.052*
C26	−0.0616(6)	0.2128(5)	0.7300(5)	0.0512(13)
H26	−0.0764	0.2690	0.7642	0.061*
C4	0.3891(6)	0.5887(4)	0.1249(4)	0.0399(11)
H4	0.4271	0.6384	0.1415	0.048*
C16	0.0481(8)	0.5209(6)	−0.3619(5)	0.0637(17)
H16A	0.0841	0.4650	−0.4024	0.076*
H16B	−0.0524	0.5221	−0.3332	0.076*
O11[a]	0.222(3)	0.1069(13)	0.1829(12)	0.070(6)
O10[a]	0.405(2)	0.0984(13)	0.1521(12)	0.070(6)

[a]Occupancy: 0.25.

the riding model approximation, with $U(H) = 1.2U_{eq}(C)$. The H on nitrogen atoms were located in a difference Fourier map, and were refined with a distance restraint of N—H = 0.86(1) or 0.85(1) Å and with $U(H) = 1.5U_{eq}$ (N, O). The O10w and O11w are disordered over two sites and the occupancies of the two disordered parts have been fixed at 0.25:0.25.

Discussion

Ciprofloxacin (*Hcf*), $C_{17}H_{18}FN_3O_3$ (systematic name 1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylic acid), is a member of the fluoroquinolone (*flqu*) family of antibiotics, widely used in veterinary clinical practice because of its wide antibiotic spectrum and its excellent bactericidal activity [1]. Our interest in ciprofloxacin and related fluoroquinolones is focused on their potential as multidentate/bridging ligands in coordination chemistry. The manganese, copper, zinc and cobalt complexe of ciprofloxacin anion had been reported [2–5]. In the title compound each Cd^{II} center atom is six-fold coordinated (CdO6), displaying a slightly distorted octahedral geometry. It is defined by one neutral bidentate O, O-bonded 1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylate (Hcf) zwitterions, by three O atoms from two biphenyl-2,2′-dicarboxylate and by one water (*cf*. the figure). Two of the biphenyl-2,2′-dicarboxylate ligands act as bridging ligands connecting two metal centers to a 18-membered ring.

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