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Crystal structure of bis(biphenyl-2,2'-dicarboxylato- $\kappa^2 O:O'$)-bis(1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylato- $\kappa^2 O,O'$)dizinc(II)2.5 hydrate, $C_{62}H_{57}N_6Zn_2O_{16.5}F_2$

Table 1: Data collection and handling.

Crystal:	Colourless block
Wavelength:	Size $0.24 \times 0.21 \times 0.19$ mm
μ :	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
Diffractometer, scan mode:	9.3 cm^{-1}
$2\theta_{\max}$, completeness:	CCD, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	55.4° , $>99\%$
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	11392 , 4995 , 0.014
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4645
Programs:	406
	OLEX2 [6], SHELX [7], Bruker programs [8]

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Abstract

$C_{62}H_{57}N_6Zn_2O_{16.5}F_2$, triclinic, $P\bar{1}$ (no. 2), $a = 10.026(4) \text{ \AA}$, $b = 12.064(4) \text{ \AA}$, $c = 13.385(5) \text{ \AA}$, $\alpha = 112.473(3)^\circ$, $\beta = 105.835(4)^\circ$, $\gamma = 91.309(4)^\circ$, $V = 1424.4(9) \text{ \AA}^3$, $Z = 1$, $R_{\text{gt}}(F) = 0.0266$, $wR_{\text{ref}}(F^2) = 0.0707$, $T = 100(2) \text{ K}$.

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The 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.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.30329(2)	0.821458(17)	0.308707(16)	0.03077(8)
F1	0.38499(14)	0.36177(11)	−0.14962(11)	0.0579(4)
O1	0.26558(14)	0.67334(11)	0.16512(10)	0.0366(3)
O2	0.13728(13)	0.78109(11)	0.34279(10)	0.0364(3)
O4	0.47284(15)	0.79303(17)	0.39991(14)	0.0592(4)
O7	0.34880(17)	0.94095(13)	0.25689(14)	0.0526(4)
O5	0.41183(17)	0.77971(15)	0.54231(12)	0.0553(4)
N1	0.00863(15)	0.36159(12)	0.08186(12)	0.0268(3)
O3	−0.01587(18)	0.65017(13)	0.34816(13)	0.0549(4)
N2	0.22660(16)	0.14514(13)	−0.20959(12)	0.0329(3)
O6	0.77815(19)	0.93254(14)	0.65841(16)	0.0638(5)
N3	0.22768(16)	−0.07527(13)	−0.39387(12)	0.0315(3)
H3A	0.1342	−0.0990	−0.4344	0.038*
H3B	0.2771	−0.1325	−0.4300	0.038*
O8	0.03333(17)	0.19102(17)	0.44614(15)	0.0741(6)
H8A	0.1067	0.1772	0.4230	0.111*
H8B	0.0562	0.2523	0.5118	0.111*
C2	0.09000(17)	0.57267(15)	0.20450(13)	0.0263(3)
C9	0.10293(17)	0.35929(15)	0.02125(13)	0.0256(3)
C8	0.18884(17)	0.46751(15)	0.05126(13)	0.0262(3)
C3	0.00474(18)	0.46509(15)	0.16739(14)	0.0280(4)

Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
H3	−0.0624	0.4638	0.2053	0.034*
C7	0.18536(17)	0.57882(15)	0.14439(13)	0.0262(3)
C10	0.11009(18)	0.25238(15)	−0.06828(14)	0.0291(4)
H10	0.0498	0.1803	−0.0883	0.035*
C11	0.20408(18)	0.25077(16)	−0.12774(14)	0.0308(4)
C1	0.06898(18)	0.67497(16)	0.30580(15)	0.0311(4)
C4	−0.09302(18)	0.25478(15)	0.04652(15)	0.0316(4)
H4	−0.1671	0.2330	−0.0279	0.038*
C18	0.48672(19)	0.75909(16)	0.48037(15)	0.0326(4)
C25	0.63621(18)	0.74714(16)	0.70810(16)	0.0345(4)
C30	0.65166(18)	0.87447(16)	0.76301(15)	0.0317(4)
C24	0.66684(19)	0.67753(16)	0.59983(17)	0.0358(4)
C19	0.60201(18)	0.68378(15)	0.49558(16)	0.0329(4)
C13	0.28260(19)	0.46613(16)	−0.01066(15)	0.0328(4)
H13	0.3417	0.5382	0.0075	0.039*
C12	0.28828(19)	0.36192(17)	−0.09600(15)	0.0355(4)
C17	0.1733(2)	0.02918(16)	−0.21437(15)	0.0358(4)
H17A	0.0714	0.0092	−0.2544	0.043*
H17B	0.1888	0.0337	−0.1365	0.043*
C31	0.69956(19)	0.95858(15)	0.71629(15)	0.0325(4)
C14	0.2024(2)	0.14110(17)	−0.32483(16)	0.0394(4)
H14A	0.2387	0.2208	−0.3195	0.047*
H14B	0.1006	0.1243	−0.3649	0.047*
C16	0.2498(2)	−0.06821(17)	−0.27717(16)	0.0390(4)
H16A	0.3512	−0.0492	−0.2354	0.047*
H16B	0.2147	−0.1476	−0.2810	0.047*
C5	−0.1382(2)	0.22927(19)	0.13355(19)	0.0477(5)
H5A	−0.2373	0.1953	0.1135	0.057*
H5B	−0.0952	0.2851	0.2141	0.057*
C26	0.5907(2)	0.6794(2)	0.76030(19)	0.0508(5)
H26	0.5797	0.5935	0.7246	0.061*
C29	0.6212(2)	0.92704(19)	0.86541(16)	0.0417(4)
H29	0.6310	1.0128	0.9021	0.050*
C20	0.6381(2)	0.61105(19)	0.4001(2)	0.0507(5)
H20	0.5963	0.6163	0.3296	0.061*
C15	0.2744(2)	0.04421(17)	−0.39129(17)	0.0403(4)
H15A	0.2520	0.0392	−0.4698	0.048*
H15B	0.3772	0.0654	−0.3558	0.048*
C28	0.5772(2)	0.8569(2)	0.91479(19)	0.0540(6)
H28	0.5580	0.8943	0.9850	0.065*
C6	−0.0473(2)	0.15064(19)	0.0742(2)	0.0494(5)
H6A	0.0519	0.1578	0.1179	0.059*
H6B	−0.0903	0.0680	0.0173	0.059*
C23	0.7629(2)	0.5958(2)	0.6022(2)	0.0581(6)
H23	0.8072	0.5905	0.6724	0.070*
C27	0.5615(3)	0.7326(2)	0.8614(2)	0.0601(7)
H27	0.5305	0.6838	0.8942	0.072*
C21	0.7345(3)	0.5309(2)	0.4065(3)	0.0707(8)
H21	0.7576	0.4817	0.3406	0.085*
C22	0.7958(3)	0.5228(2)	0.5074(3)	0.0755(9)
H22	0.8605	0.4673	0.5119	0.091*
O9 ^a	0.5504(15)	0.2242(15)	−0.0719(12)	0.161(6)
H9A ^a	0.6227	0.2619	−0.0756	0.242*
H9B ^a	0.4750	0.2513	−0.0982	0.242*

^aOccupancy: 0.25.

Source of material

A mixture of Zn(CH₃COO)₂·2H₂O (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 minutes. The mixture was transferred to a 25 mL Teflon-lined hydrothermal bomb and heated to 443 K for 96 h under autogenous pressure. After cooling, colorless crystals of title compound were obtained.

Experimental details

The carbon-bound H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U = 1.2U_{eq}(C). The nitrogen-bound H 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 = 1.5U_{eq}(N, O). For the water molecule O9w an occupancy of 0.25 was assigned.

Discussion

Ciprofloxacin (Hcf), C₁₇H₁₈FN₃O₃ (systematic name 1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylic acid), is a member of the fluoroquinolone (flq) family of antibiotics, widely used in 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 cadmium, copper, nickel, manganese and cobalt complexes of ciprofloxacin anion had been reported [2–5]. In the title compound each Zn^{II} center is tetra coordinated (ZnO₄), displaying a slightly distorted tetrahedral geometry defined by one bidentate 1-cyclopropyl-6-fluoro-4-oxo-7-(1-piperazinyl)-1,4-dihydroquinoline-3-carboxylate (Hcf) zwitterion and two biphenyl-2,2'-dicarboxylate ligands. The biphenyl-2,2'-dicarboxylate ligands act as bridging ligands, to produce a cyclic dimer (*cf.* the figure).

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