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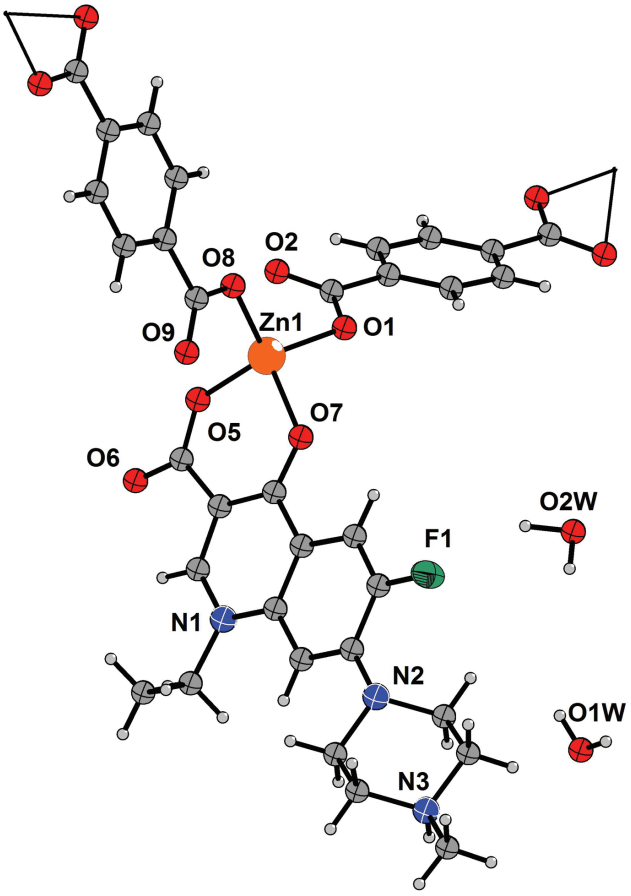
# Crystal structure of *catena*-poly[($\mu_2$-benzene-1,4-dicarboxylato-$\kappa^2O$:$O'$)-(1-ethyl-6-fluoro-7-(4-methylpiperazin-1-ium-1-yl)-4-oxo-1,4-dihydroquinoline-3-carboxylato-$\kappa^2O$,$O'$)zinc(II)] 1.25 hydrate, $C_{25}H_{26.5}N_3O_{8.25}FZn$

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## Abstract

$C_{25}H_{26.5}N_3O_{8.25}FZn$, Triclinic, $P\bar{1}$ (no. 2) $a = 10.2889(6)$ Å, $b = 10.6034(5)$ Å, $c = 12.9576(8)$ Å, $\alpha = 105.984(5)°$, $\beta = 109.785(6)°$, $\gamma = 93.257(4)°$, $V = 1261.05(12)$ Å$^3$, $Z = 2$, $R_{gt}(F) = 0.0344$, $wR_{ref}(F^2) = 0.0783$, $T = 293$ K.

**CCDC no.:** 1571276

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colorless, prims |
| Size: | 0.20 × 0.16 × 0.15 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.04 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker FRAMBO, $\varphi$ and $\omega$-scans |
| $2\theta_{max}$, completeness: | 25°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 8432, 4431, 0.026 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3772 |
| $N(param)_{refined}$: | 352 |
| Programs: | Bruker programs [1], SHELX [2] |

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

## Source of material

A mixture of $Zn(CH_3COO)_2 \cdot 2H_2O$ (0.25 mmol), perfloxacin (0.5 mmol), 1,4-benzenedicarboxylic acid (0.25 mmol), sodium hydroxide (0.5 mmol) and water (15 mL) was stirred for 30 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.

## 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 $U(H) = 1.2U_{eq}$(C). The H on nitrogen atom 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). During the refinement it became clear that O2w is not fullly occupied (0.25).

## Comment

Perfloxacin (Hpef), $C_{17}H_{20}FN_3O_3$ (systematic name 1-ethyl-6-fluoro-7-(4-methylpiperazin-4-ium-1-yl)-4-oxo-1,4-dihydroquinoline-3-carboxylic acid) is a member of the fluoro-

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Zn1 | 0.28138(3) | 0.36474(3) | 0.29389(3) | 0.02868(10) |
| F1 | 0.32095(16) | 0.92156(15) | 0.72440(14) | 0.0527(5) |
| O1 | 0.3882(2) | 0.27672(18) | 0.39987(16) | 0.0447(5) |
| O2 | 0.3335(2) | 0.1043(2) | 0.24347(19) | 0.0559(6) |
| O5 | 0.08543(18) | 0.29088(16) | 0.19723(15) | 0.0364(4) |
| O6 | −0.1434(2) | 0.28099(18) | 0.13667(16) | 0.0449(5) |
| O7 | 0.23676(17) | 0.50723(16) | 0.40580(15) | 0.0322(4) |
| O8 | 0.3850(2) | 0.3984(2) | 0.20392(18) | 0.0514(6) |
| O9 | 0.2847(2) | 0.5723(2) | 0.17822(19) | 0.0548(6) |
| N1 | −0.1506(2) | 0.60011(18) | 0.38281(17) | 0.0254(4) |
| N2 | 0.0696(2) | 1.00136(19) | 0.71101(17) | 0.0295(5) |
| N3 | 0.0165(2) | 1.21639(19) | 0.87265(18) | 0.0313(5) |
| H3 | −0.0489 | 1.1718 | 0.8884 | 0.038* |
| C1 | 0.4492(3) | 0.0753(2) | 0.4270(2) | 0.0307(6) |
| C2 | 0.5274(3) | 0.1368(3) | 0.5427(2) | 0.0348(6) |
| H2 | 0.5466 | 0.2292 | 0.5718 | 0.042* |
| C3 | 0.4226(3) | −0.0626(2) | 0.3842(2) | 0.0352(6) |
| H3A | 0.3711 | −0.1052 | 0.3062 | 0.042* |
| C7 | 0.3860(3) | 0.1556(3) | 0.3489(3) | 0.0342(6) |
| C9 | −0.0248(3) | 0.3340(2) | 0.2057(2) | 0.0282(6) |
| C10 | −0.0117(2) | 0.4557(2) | 0.3043(2) | 0.0245(5) |
| C11 | 0.1139(2) | 0.5313(2) | 0.3933(2) | 0.0238(5) |
| C12 | 0.0997(2) | 0.6473(2) | 0.4773(2) | 0.0235(5) |
| C13 | −0.0325(2) | 0.6818(2) | 0.4702(2) | 0.0243(5) |
| C14 | −0.1368(3) | 0.4939(2) | 0.3051(2) | 0.0259(5) |
| H14 | −0.2184 | 0.4418 | 0.2472 | 0.031* |
| C15 | −0.2933(2) | 0.6332(2) | 0.3656(2) | 0.0301(6) |
| H15A | −0.3015 | 0.6691 | 0.4402 | 0.036* |
| H15B | −0.3622 | 0.5529 | 0.3225 | 0.036* |
| C16 | −0.3226(3) | 0.7333(3) | 0.3012(3) | 0.0434(7) |
| H16A | −0.4150 | 0.7533 | 0.2914 | 0.065* |
| H16B | −0.3162 | 0.6972 | 0.2268 | 0.065* |
| H16C | −0.2551 | 0.8132 | 0.3443 | 0.065* |
| C17 | −0.0426(3) | 0.7970(2) | 0.5501(2) | 0.0271(5) |
| H17 | −0.1307 | 0.8180 | 0.5448 | 0.033* |
| C18 | 0.0734(3) | 0.8792(2) | 0.6356(2) | 0.0271(5) |
| C19 | 0.2055(3) | 0.8405(2) | 0.6421(2) | 0.0329(6) |
| C20 | 0.2183(3) | 0.7299(2) | 0.5670(2) | 0.0300(6) |
| H20 | 0.3067 | 0.7081 | 0.5748 | 0.036* |
| C21 | 0.1208(3) | 1.0138(2) | 0.8346(2) | 0.0354(6) |
| H21A | 0.2070 | 0.9776 | 0.8551 | 0.042* |
| H21B | 0.0520 | 0.9633 | 0.8502 | 0.042* |
| C22 | 0.1467(3) | 1.1579(2) | 0.9067(2) | 0.0363(6) |
| H22A | 0.1765 | 1.1645 | 0.9877 | 0.044* |
| H22B | 0.2208 | 1.2071 | 0.8958 | 0.044* |
| C23 | −0.0392(3) | 1.1968(2) | 0.7470(2) | 0.0329(6) |
| H23A | 0.0264 | 1.2467 | 0.7280 | 0.039* |
| H23B | −0.1269 | 1.2306 | 0.7263 | 0.039* |
| C24 | −0.0630(3) | 1.0518(2) | 0.6786(2) | 0.0334(6) |
| H24A | −0.1320 | 1.0019 | 0.6944 | 0.040* |
| H24B | −0.0981 | 1.0412 | 0.5966 | 0.040* |
| C25 | 0.0372(3) | 1.3587(2) | 0.9397(2) | 0.0435(7) |
| H25A | −0.0493 | 1.3920 | 0.9157 | 0.065* |
| H25B | 0.1075 | 1.4083 | 0.9264 | 0.065* |
| H25C | 0.0667 | 1.3682 | 1.0205 | 0.065* |
| C26 | 0.3597(3) | 0.4914(3) | 0.1588(2) | 0.0358(6) |
| C27 | 0.4312(3) | 0.4940(3) | 0.0748(2) | 0.0314(6) |
| C28 | 0.4132(3) | 0.5907(3) | 0.0203(2) | 0.0357(6) |
| H28 | 0.3547 | 0.6518 | 0.0335 | 0.043* |
| C29 | 0.5181(3) | 0.4033(3) | 0.0538(2) | 0.0344(6) |
| H29 | 0.5303 | 0.3378 | 0.0898 | 0.041* |
| O1W | 0.1688(3) | 0.8904(2) | 1.0674(2) | 0.0926(10) |
| H1WA | 0.1841 | 0.8401 | 1.0106 | 0.139* |
| H1WB | 0.2406 | 0.9496 | 1.1095 | 0.139* |
| O2w[a] | 0.5753(14) | 0.9214(14) | 0.9416(11) | 0.124(5) |
| H2WA[a] | 0.5346 | 0.9939 | 0.9619 | 0.185* |
| H2WB[a] | 0.5167 | 0.8963 | 0.8577 | 0.185* |

[a]Occupancy: 0.2500.

quinolone (flqu) family of antibiotics, widely used in veterinary clinical practice because of its wide antibiotic spectrum and its excellent bactericidal activity [4]. Our interest in perfloxacin and related fluoroquinolones is focused on their potential as multidentate/bridging ligands in coordination chemistry. The cobalt, manganese and zinc complexes of perfloxacin anion had been reported [5–7].

In the title compound, each $Zn^{II}$ center atom is tetra-coordinated (ZnO4), displaying a slightly distorted tetrahedron geometry defined by one bidentate O,O-bonded 1-ethyl-6-fluoro-7-(4-methylpiperazin-1-yl)-4-oxo-quinoline-3-carboxylate (pef) anions (Zn-O5, Zn-O7), two unidentate O-bonded 1,4-benzenedicarboxylate anions (Zn-O1, Zn-O8) (*cf*. the figure). The 1,4-benzenedicarboxylate ligands act as bridging ligands, together with two zinc(II) cations to form a coordination polymer.

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