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Crystallographic report

Bis(norfloxacin)dilead(II) tetranitrate, $[Pb_2(H\text{-}Norf)_2(ONO_2)_4]$

Zhen-Feng Chen[1]*, Hong-Li Zhou[1], Hong Liang[1], Yan Li[2], Ren-Gen Xiong[3] and Xiao-Zeng You[3]**

[1]Department of Chemistry & Chemical Engineering, Guangxi Normal University, Guilin 541004, People's Republic of China
[2]Institute of Chemistry Chinese Academic of Science, Beijing 10080, People's Republic of China
[3]Coordination Chemistry Institute, The State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

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The centrosymmetric binuclear structure of $[Pb_2(H\text{-}Norf)_2(ONO_2)_4]$shows the geometry around each lead(II) atom to be distorted trigonal bipyramidal with Pb–O distances ranging from 2.357(3) to 2.769(4) Å. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; lead; norfloxacin

COMMENT

Figure 1 shows the centrosymmetric molecular structure of a biologically relevant complex formed between the widely used antibacterial drug norfloxacin (H-Norf) and the toxic heavy-metal lead(II), $[Pb(H\text{-}Norf)(ONO_2)_2]_2$ (**1**). Similar to the magnesium(II) complex of norfloxacin,[1] compound **1** is a 2:2 dimer in which the two lead(II) ions are bridged by two oxygen atoms derived from two carboxylate groups (monodentate bridging[2]) to give rise to a four-membered Pb_2O_2 ring. Each lead(II) is coordinated in a distorted trigonal-bipyramidal coordination environment by the aforementioned carboxylate oxygen atoms, a quinolone carbonyl and two oxygen atoms derived from two monodentate nitrates. The nitrogen atom of the piperazine ring is protonated, and thereby loses its coordination ability. The Pb···Pb separation within compound **1** is 4.0852(4) Å, which is somewhat longer than that of the lead(II) citrate aqueous complex.[3]

*Correspondence to: Zhen-Feng Chen, Department of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, People's Republic of China.
E-mail: chenzfgxnu@yahoo.com
**Correspondence to: Hong Liang, Department of Chemistry and Chemical Engineering, Guangxi Normal University, Guilin 541004, People's Republic of China.
E-mail: chenzfgxnu@yahoo.com
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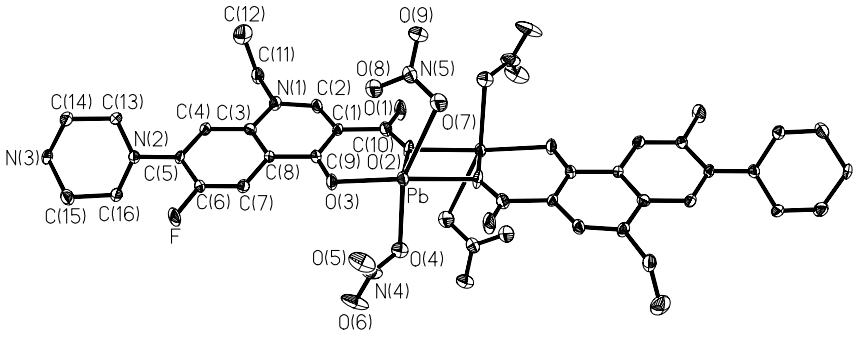

Figure 1. Molecular structure of $[Pb(H\text{-}Norf)(ONO_2)_2]_2$. Key geometric parameters: Pb–O(2) 2.357(3), Pb–O(2)^i 2.501(2), Pb–O(3) 2.427(3), Pb–O(4) 2.537(3), Pb–O(7) 2.769(4); O(2)–Pb–O(3) 71.79(10), O(2)–Pb–O(2)^i 65.57(12), O(2)–Pb–O(4) 84.23(12), O(3)–Pb–O(4) 73.72(10), O(3)–Pb–O(2)^i 132.05(11), O(2)–Pb–O(7) 92.96(12)°. Symmetry codes *i*: $-x, -y-2, -z+2$.

EXPERIMENTAL

Samples of $Pb(NO_3)_2 \cdot 4H_2O$ (1 mmol) and norfloxacin (1 mmol) were thoroughly mixed in a mortar with a pestle, and placed in thick-walled Pyrex tubes (*ca* 20 cm long). After addition of EtOH (0.5 ml) and H_2O (1.5 ml; *ca* pH 6.0), the tube was frozen with liquid nitrogen, evacuated under vacuum and sealed with a torch. The tube was heated at 80 °C for 3 days to give colorless block crystals (only one phase). Yield: 65%. Anal. Found: C, 29.46; H, 2.85; N, 10.85. Calc. for $C_{16}H_{18}FN_5O_9Pb$: C, 29.54; H, 2.79; N, 10.77%. X-ray diffraction data were collected at 293 K on a Rigaku R-axis RAPID IP diffractometer using graphite-monochromated Mo Kα radiation on a block $0.08 \times 0.15 \times 0.20$ mm^3. Crystallographic data: $C_{16}H_{18}FN_5O_9Pb$, $M = 650.54$, monoclinic, $P2_1/c$, $a = 12.6071(6)$, $b = 8.7323(5)$, $c = 18.9532(9)$ Å, $\beta = 104.311(2)°$, $V = 2021.79(18)$ Å^3, $Z = 4$, $D = 2.137$ Mg m^{-3}, 8588 reflections collected, 4547 unique and 2632 $I > 2\sigma(I)$. R (obs. data on F^2) 0.025, wR (all data) 0.030, $\rho_{max} = 0.48$ e$^-$ Å^{-3}. Programs used: SHELXTL97, ORTEP. CCDC deposition number: CCDC 214506.

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REFERENCES

1. Chen ZF, Xiong RG, Zuo JL, Guo Z, You XZ, Fun HK. *J. Chem. Soc. Dalton Trans* 2000; 4013.
2. Rardin RL, Tolman WB, Lippard SJ. *New J. Chem.* 1991; **15**: 417.
3. Kourgiantakis M, Matzapetakis M, Raptopoulou CP, Terzis A, Salofoglou A. *Inorg. Chim. Acta* 2000; **297**: 134.