Crystal structure of tetraaqua-bis(norfloxacin)barium(II) 1,4-benzenedicarboxylate hydrate, $[Ba(H_2O)_4(C_{16}H_{17}FN_3O_3)_2][C_6H_4(COO)_2] \cdot H_2O$

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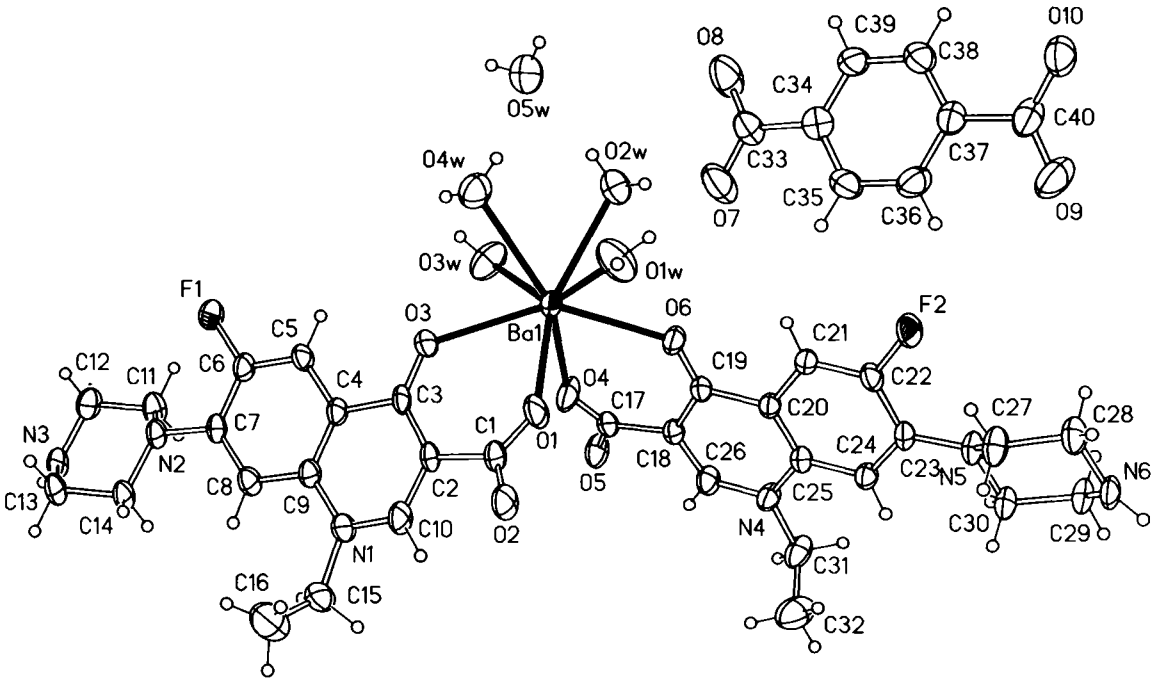

Abstract

$C_{40}H_{48}BaF_2N_6O_{15}$, triclinic, $P\bar{1}$ (no. 2), a = 12.305(1) Å, b = 12.472(1) Å, c = 16.591(2) Å, α = 101.920(2)°, β = 104.630(2)°, γ = 111.703(1)°, V = 2157.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.054, $wR_{ref}(F^2)$ = 0.170, T = 295 K.

Source of material

The title complex was prepared by hydrothermal method. A mixture of $Ba(Ac)_2$ (0.25 mmol), norfloxacin hydrochloride (Norf, 0.5 mmol), 1,4-benzenedicarboxylic acid (1,4-bdc, 0.5 mmol), NaOH (0.25 mmol) and water (12 ml) was stirred for 20 min in air. The mixture was then transferred to a 23 ml Teflon-lined autoclave and kept at 423 K for 72 h under autogenous pressure. After this treatment the mixture was allowed to cool slowly to room temperature. Colorless single crystals suitable for X-ray analysis were obtained from the reaction mixture by evaporation.

Discussion

The crystal structure of the title mononuclear complex consists of one Ba^{2+}, two Norf ligands, four coordinated water molecules, one uncoordinated water and one 1,4-bdc anion. The Ba(II) atom is coordinated by four O atoms from two Norf ligands and four water molecules in form of a distorted bisphenoid. The bond lengths Ba—O1, Ba—O3, Ba—O4 and Ba—O6 are 2.687(4) Å, 2.709(4) Å, 2.685(4) Å and 2.697(4) Å, respectively. Two Norf ligands are connected to Ba(II) atom forming two six-membered chelating rings. The six-membered rings, which consist of Ba1, O1, C1, C2, C3, O3 and Ba1, O4, C17, C18, C19, O6, are nearly coplanar with the r.m.s. deviations of 0.177(3) Å and 0.108(3) Å, respectively. The dihedral angle between these rings is 74.0(3)°. The intermolecular hydrogen bonds are formed among water molecules and carboxy O atoms of 1,4-bdc anions. The water molecules and N3 interact with carboxy O atoms of adjacent Norf ligands and 1,4-bdc ions forming also intermolecular hydrogen bonds. The intermolecular hydrogen bonds result in a three-dimensional supramolecular framework.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.24 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.06 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II CCD, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10715, 7412
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6969
$N(param)_{refined}$:	579
Programs:	SHELXS-97 [1], SHELXL-97 [2]

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Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{iso}
H(1W1)	2*i*	−0.1349	0.1062	0.0910	0.089
H(1W2)	2*i*	−0.1300	0.1580	0.1712	0.089
H(2W1)	2*i*	−0.0442	0.3422	0.3055	0.079
H(2W2)	2*i*	0.0117	0.4560	0.3053	0.079
H(3W1)	2*i*	0.3845	0.6056	0.3915	0.088
H(3W2)	2*i*	0.3253	0.6041	0.3118	0.088
H(4W1)	2*i*	0.0402	0.4795	0.1760	0.074
H(4W2)	2*i*	0.1487	0.5134	0.1633	0.074
H(5W2)	2*i*	0.0457	0.6709	0.2643	0.098
H(5W1)	2*i*	−0.0740	0.5932	0.2459	0.098
H(3N)	2*i*	1.1321	0.8924	0.1230	0.040
H(6N)	2*i*	−0.3796	−0.6303	0.3989	0.042
H(5)	2*i*	0.4499	0.5767	0.1346	0.033
H(8)	2*i*	0.7520	0.4641	0.0801	0.036
H(10)	2*i*	0.4400	0.1142	0.0685	0.043
H(11A)	2*i*	0.9311	0.7895	0.2083	0.043
H(11B)	2*i*	0.8281	0.8339	0.1797	0.043
H(12A)	2*i*	0.9225	0.9218	0.0897	0.045
H(12B)	2*i*	1.0223	0.9748	0.1860	0.045
H(13A)	2*i*	1.0277	0.7149	0.0022	0.041
H(13B)	2*i*	0.9254	0.7612	−0.0237	0.041
H(14A)	2*i*	0.8322	0.5727	−0.0027	0.038
H(14B)	2*i*	0.9335	0.6237	0.0928	0.038
H(15A)	2*i*	0.7428	0.3043	0.1096	0.054
H(15B)	2*i*	0.6493	0.1687	0.0917	0.054
H(16A)	2*i*	0.5702	0.1620	−0.0585	0.107
H(16B)	2*i*	0.6980	0.1553	−0.0261	0.107
H(16C)	2*i*	0.6970	0.2790	−0.0315	0.107
H(21)	2*i*	−0.0960	0.0174	0.3387	0.033
H(24)	2*i*	0.0624	−0.2187	0.4654	0.033
H(26)	2*i*	0.4055	0.1151	0.4952	0.039
H(27A)	2*i*	−0.2651	−0.4541	0.2990	0.049
H(27B)	2*i*	−0.3221	−0.3602	0.3005	0.049
H(28A)	2*i*	−0.4235	−0.4352	0.3929	0.050
H(28B)	2*i*	−0.4652	−0.5429	0.3057	0.050
H(29A)	2*i*	−0.2018	−0.5112	0.5236	0.044
H(29B)	2*i*	−0.2584	−0.4172	0.5319	0.044
H(30A)	2*i*	−0.0629	−0.3205	0.5217	0.040
H(30B)	2*i*	−0.1011	−0.4267	0.4346	0.040
H(31A)	2*i*	0.2497	−0.1164	0.5563	0.049
H(31B)	2*i*	0.3830	−0.0259	0.5634	0.049
H(32A)	2*i*	0.2232	−0.2457	0.4216	0.094
H(32B)	2*i*	0.3442	−0.2256	0.4955	0.094
H(32C)	2*i*	0.3558	−0.1549	0.4278	0.094
H(35)	2*i*	−0.3303	−0.0223	0.2424	0.050
H(36)	2*i*	−0.5059	−0.1932	0.2294	0.053
H(38)	2*i*	−0.6839	0.0141	0.2637	0.047
H(39)	2*i*	−0.5052	0.1863	0.2863	0.046

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ba(1)	2*i*	0.16713(3)	0.31701(3)	0.24367(2)	0.0262(2)	0.0278(2)	0.0330(2)	0.0109(2)	0.0144(1)	0.0158(2)
F(1)	2*i*	0.6189(3)	0.7450(3)	0.1141(3)	0.040(2)	0.027(2)	0.066(2)	0.011(2)	0.030(2)	0.023(2)
F(2)	2*i*	−0.2665(3)	−0.1639(4)	0.3462(3)	0.023(2)	0.050(2)	0.077(3)	0.012(2)	0.020(2)	0.037(2)
O(1)	2*i*	0.1814(4)	0.1343(4)	0.1353(3)	0.046(3)	0.029(2)	0.079(3)	0.010(2)	0.045(3)	0.019(2)
O(1W)	2*i*	−0.0881(4)	0.1565(5)	0.1400(3)	0.036(2)	0.066(3)	0.053(3)	0.010(2)	0.013(2)	0.000(3)
O(2)	2*i*	0.2163(4)	0.0100(4)	0.0422(3)	0.035(2)	0.026(2)	0.051(2)	0.002(2)	0.022(2)	0.010(2)
O(2W)	2*i*	0.0235(4)	0.4029(5)	0.3227(3)	0.038(2)	0.053(3)	0.068(3)	0.016(2)	0.025(2)	0.024(2)
O(3)	2*i*	0.3049(4)	0.3816(4)	0.1424(3)	0.030(2)	0.030(2)	0.049(2)	0.013(2)	0.023(2)	0.018(2)
O(3W)	2*i*	0.3340(5)	0.5609(4)	0.3417(3)	0.065(3)	0.037(3)	0.054(3)	0.014(2)	0.004(2)	0.015(2)
O(4W)	2*i*	0.0798(5)	0.4559(4)	0.1498(3)	0.051(3)	0.038(2)	0.056(3)	0.016(2)	0.019(2)	0.017(2)
O(5W)	2*i*	−0.0009(5)	0.6049(5)	0.2656(4)	0.060(3)	0.058(3)	0.083(4)	0.023(3)	0.034(3)	0.032(3)
O(4)	2*i*	0.3571(4)	0.3111(4)	0.3625(3)	0.026(2)	0.050(3)	0.071(3)	0.009(2)	0.013(2)	0.045(2)
O(5)	2*i*	0.4923(4)	0.3090(4)	0.4779(3)	0.026(2)	0.036(2)	0.042(2)	0.000(2)	0.006(2)	0.018(2)
O(6)	2*i*	0.1044(4)	0.1768(4)	0.3439(3)	0.027(2)	0.037(2)	0.054(2)	0.013(2)	0.017(2)	0.031(2)
O(7)	2*i*	−0.1936(5)	0.1931(5)	0.2702(4)	0.045(3)	0.055(3)	0.094(4)	0.014(2)	0.042(3)	0.015(3)
O(8)	2*i*	−0.2799(5)	0.3140(5)	0.3023(4)	0.062(3)	0.036(3)	0.105(5)	0.008(2)	0.053(3)	0.010(3)
O(9)	2*i*	−0.7158(5)	−0.3193(5)	0.2232(4)	0.054(3)	0.037(3)	0.094(4)	0.012(2)	0.010(3)	0.032(3)
O(10)	2*i*	−0.8276(4)	−0.2131(5)	0.2297(3)	0.036(3)	0.055(3)	0.066(3)	0.012(2)	0.015(2)	0.034(3)
N(1)	2*i*	0.5631(4)	0.2789(4)	0.0835(3)	0.024(2)	0.029(2)	0.050(3)	0.008(2)	0.019(2)	0.016(2)
N(2)	2*i*	0.8069(4)	0.6903(4)	0.0846(3)	0.026(2)	0.024(2)	0.033(2)	0.002(2)	0.016(2)	0.010(2)
N(3)	2*i*	1.0514(4)	0.8574(4)	0.1010(3)	0.021(2)	0.029(2)	0.040(3)	−0.001(2)	0.013(2)	0.016(2)
N(4)	2*i*	0.2428(4)	−0.0183(4)	0.4743(3)	0.026(2)	0.027(2)	0.040(3)	0.008(2)	0.008(2)	0.018(2)
N(5)	2*i*	−0.1841(4)	−0.3163(4)	0.4156(3)	0.026(2)	0.027(2)	0.031(2)	0.000(2)	0.009(2)	0.012(2)
N(6)	2*i*	−0.3466(4)	−0.5520(4)	0.4126(3)	0.027(2)	0.027(2)	0.047(3)	0.002(2)	0.017(2)	0.018(2)
C(1)	2*i*	0.2440(5)	0.1139(5)	0.0915(4)	0.023(3)	0.026(3)	0.036(3)	0.003(2)	0.014(2)	0.012(2)
C(2)	2*i*	0.3599(5)	0.2207(5)	0.0967(3)	0.020(2)	0.025(3)	0.032(3)	0.003(2)	0.014(2)	0.012(2)
C(3)	2*i*	0.3771(4)	0.3451(5)	0.1198(3)	0.018(2)	0.028(3)	0.030(3)	0.004(2)	0.012(2)	0.015(2)
C(4)	2*i*	0.4907(5)	0.4315(5)	0.1119(3)	0.021(2)	0.025(3)	0.030(3)	0.006(2)	0.010(2)	0.013(2)
C(5)	2*i*	0.5080(5)	0.5522(5)	0.1216(3)	0.022(2)	0.026(3)	0.034(3)	0.007(2)	0.014(2)	0.010(2)
C(6)	2*i*	0.6102(5)	0.6328(5)	0.1118(4)	0.030(3)	0.020(2)	0.035(3)	0.005(2)	0.016(2)	0.011(2)
C(7)	2*i*	0.7053(5)	0.6030(5)	0.0963(3)	0.023(2)	0.024(3)	0.030(3)	0.001(2)	0.011(2)	0.011(2)
C(8)	2*i*	0.6902(5)	0.4859(5)	0.0888(4)	0.024(3)	0.028(3)	0.034(3)	0.006(2)	0.013(2)	0.009(2)
C(9)	2*i*	0.5812(5)	0.3978(5)	0.0943(3)	0.023(2)	0.023(3)	0.030(3)	0.004(2)	0.011(2)	0.008(2)
C(10)	2*i*	0.4530(5)	0.1951(5)	0.0814(4)	0.031(3)	0.025(3)	0.046(3)	0.002(2)	0.019(3)	0.012(2)
C(11)	2*i*	0.8829(5)	0.8048(5)	0.1598(4)	0.033(3)	0.027(3)	0.033(3)	−0.001(2)	0.014(2)	0.004(2)
C(12)	2*i*	0.9707(6)	0.9013(5)	0.1347(4)	0.036(3)	0.024(3)	0.044(3)	0.004(2)	0.014(3)	0.012(2)
C(13)	2*i*	0.9738(5)	0.7440(5)	0.0235(4)	0.028(3)	0.033(3)	0.038(3)	0.005(2)	0.019(2)	0.012(2)
C(14)	2*i*	0.8851(5)	0.6455(5)	0.0489(4)	0.027(3)	0.025(3)	0.036(3)	0.003(2)	0.017(2)	0.009(2)
C(15)	2*i*	0.6604(6)	0.2388(6)	0.0721(5)	0.038(3)	0.038(3)	0.067(4)	0.017(3)	0.030(3)	0.019(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(16)	2*i*	0.6560(9)	0.2059(9)	−0.0188(6)	0.073(6)	0.088(6)	0.075(6)	0.047(5)	0.042(5)	0.031(5)
C(17)	2*i*	0.3836(5)	0.2682(5)	0.4211(4)	0.023(3)	0.026(3)	0.039(3)	0.007(2)	0.014(2)	0.012(2)
C(18)	2*i*	0.2820(5)	0.1565(5)	0.4249(4)	0.022(2)	0.025(3)	0.034(3)	0.007(2)	0.012(2)	0.012(2)
C(19)	2*i*	0.1500(5)	0.1206(5)	0.3841(3)	0.024(2)	0.023(2)	0.032(3)	0.008(2)	0.014(2)	0.011(2)
C(20)	2*i*	0.0679(5)	0.0085(5)	0.3952(3)	0.025(2)	0.022(2)	0.028(2)	0.008(2)	0.012(2)	0.010(2)
C(21)	2*i*	−0.0635(5)	−0.0291(5)	0.3642(3)	0.025(3)	0.028(3)	0.034(3)	0.012(2)	0.013(2)	0.016(2)
C(22)	2*i*	−0.1418(5)	−0.1345(5)	0.3721(4)	0.020(2)	0.031(3)	0.036(3)	0.007(2)	0.012(2)	0.012(2)
C(23)	2*i*	−0.0994(5)	−0.2113(5)	0.4084(3)	0.026(3)	0.023(3)	0.027(3)	0.004(2)	0.013(2)	0.009(2)
C(24)	2*i*	0.0303(5)	−0.1713(5)	0.4407(3)	0.026(3)	0.024(3)	0.031(3)	0.008(2)	0.011(2)	0.011(2)
C(25)	2*i*	0.1136(5)	−0.0606(5)	0.4367(3)	0.023(2)	0.024(3)	0.026(2)	0.006(2)	0.009(2)	0.009(2)
C(26)	2*i*	0.3196(5)	0.0873(5)	0.4679(4)	0.023(3)	0.029(3)	0.037(3)	0.004(2)	0.008(2)	0.012(2)
C(27)	2*i*	−0.2915(6)	−0.4041(6)	0.3348(4)	0.033(3)	0.034(3)	0.035(3)	−0.002(3)	0.007(2)	0.013(3)
C(28)	2*i*	−0.3948(6)	−0.4848(6)	0.3590(4)	0.028(3)	0.039(3)	0.047(3)	0.003(3)	0.011(3)	0.019(3)
C(29)	2*i*	−0.2343(5)	−0.4653(6)	0.4926(4)	0.036(3)	0.033(3)	0.037(3)	0.005(3)	0.014(2)	0.020(3)
C(30)	2*i*	−0.1323(5)	−0.3800(5)	0.4684(4)	0.029(3)	0.028(3)	0.038(3)	0.004(2)	0.012(2)	0.016(2)
C(31)	2*i*	0.2991(6)	−0.0836(6)	0.5222(4)	0.031(3)	0.039(3)	0.046(3)	0.010(3)	0.003(3)	0.025(3)
C(32)	2*i*	0.3062(8)	−0.1868(7)	0.4613(6)	0.051(4)	0.052(4)	0.082(6)	0.027(4)	0.011(4)	0.025(4)
C(33)	2*i*	−0.2813(6)	0.2115(6)	0.2816(4)	0.035(3)	0.045(4)	0.047(3)	0.014(3)	0.020(3)	0.012(3)
C(34)	2*i*	−0.4010(6)	0.0991(6)	0.2662(4)	0.036(3)	0.036(3)	0.030(3)	0.013(3)	0.010(2)	0.008(2)
C(35)	2*i*	−0.4005(6)	−0.0146(6)	0.2484(4)	0.031(3)	0.044(4)	0.052(4)	0.019(3)	0.017(3)	0.015(3)
C(36)	2*i*	−0.5067(6)	−0.1173(6)	0.2393(5)	0.040(3)	0.030(3)	0.058(4)	0.015(3)	0.011(3)	0.015(3)
C(37)	2*i*	−0.6137(5)	−0.1070(5)	0.2451(4)	0.032(3)	0.034(3)	0.035(3)	0.010(2)	0.008(2)	0.015(2)
C(38)	2*i*	−0.6124(6)	0.0066(6)	0.2611(4)	0.032(3)	0.040(3)	0.046(3)	0.016(3)	0.014(3)	0.013(3)
C(39)	2*i*	−0.5057(6)	0.1099(6)	0.2733(4)	0.035(3)	0.030(3)	0.052(4)	0.016(3)	0.016(3)	0.013(3)
C(40)	2*i*	−0.7279(6)	−0.2225(6)	0.2318(4)	0.036(3)	0.040(4)	0.049(4)	0.008(3)	0.005(3)	0.025(3)

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