Crystal structure of aquabis[2-(2,4-difluorophenyl)-1,3-bis(1,2,4-triazol-1-yl)-propan-2-ol]chloromanganese(II) chloride — water — *N*,*N*-dimethylformamide (1:1:1), $[MnCl(H_2O)(C_{13}H_{12}N_6OF_2)_2]Cl \cdot H_2O \cdot C_3H_7NO$

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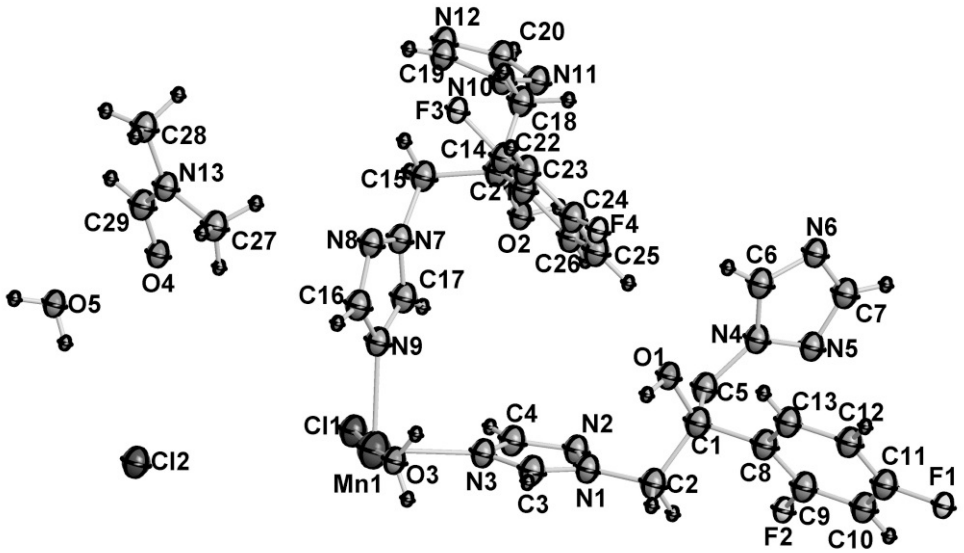

Abstract

$C_{29}H_{35}Cl_2F_4MnN_{13}O_5$, monoclinic, *Cc* (no. 9), a = 23.827(3) Å, b = 9.4313(9) Å, c = 20.494(2) Å, β = 125.084(2)°, V = 3768.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.027, T = 296 K.

Source of material

A solution of 2-(2,4-difluorophenyl)-1,3-bis(1,2,4-triazol-1-yl)propan-2-ol (Fluconazole, HFlu, 0.10 mmol) in 10 mL DMF was added dropwise with stirring at room temperature to a solution of $MnCl_2 \cdot 4H_2O$ (0.1981 g, 0.10 mmol) in 10 mL water. The mixture was stirred at room temperature until it was homogeneous, and then sealed in a 25 mL Teflon-lined stainless steel reactor, kept under pressure at 120 °C for 24 h, and then slowly cooled to room temperature. After 7 days, colorless block-shaped crystals suitable for X-ray diffraction were separated and washed with water. They were stable in air and insoluble in water and common organic solvents.

Elemental analysis — found: C, 41.22 %; H, 3.91 %; N, 20.25 %; calculated for $C_{29}H_{35}Cl_2F_4MnN_{13}O_5$: C, 41.20 %; H, 3.93; N, 20.26 %.

Experimental details

H atoms on C atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93-0.96 Å and U_{iso}(H) = 1.2 U_{eq}(C). The water H atoms were located in difference Fourier maps and refined using a riding model with d(O—H) = 0.82 Å and U_{iso}(H) = 1.5 U_{eq}(C). The Flack parameter is 0.31(1) based on 1949 Friedel pairs.

Discussion

2-(2,4-Difluorophenyl)-1,3-bis(1,2,4-triazol-1-yl)propan-2-ol (HFlu) is one of the first-line popular drugs used to treat invasive infections [1]. HFlu has been of our research interest for the construction of unusual coordination polymers due to its flexible backbone, versatile coordination fashions, as well as pharmacological activity [2-3]. Thus, the HFlu ligands have potential to construct a diversity of coordination architectures upon the metal complexation.

In the title crystal structure, each Mn(II) center is hexa-coordinated by four nitrogen atoms from four triazole moieties of four different HFlu ligands, one oxygen atom from the water molecule and one chloride ion. The asymmetric unit also contains hydrogen-bonded water, one chloride ion and *N*,*N*-dimethylformamide. The coordinated HFlu ligands adopt a bis(bidentate) mode bridging the Mn(II) ions into chains. The title structure is rich in hydrogen bonds with the distances d(O1–H1···O5) = 2.721(3) Å, d(O2–H2···Cl2) = 3.030(2) Å, d(O3–H3A···O4) = 2.829(3) Å, d(O3–H3B···Cl2) = 3.035(2) Å, d(O5–H5A···Cl1) = 3.132(3) Å, d(O5-H5B···O4) = 2.919(4) Å. The hydrogen bonds seem to be effective in the formation and stabilization of a supramolecular structure.

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.22 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.68 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9839, 5168
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5087
$N(param)_{refined}$:	491
Programs:	SHELXS-97, SHELXL-97 [4], DIAMOND [5]

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

Atom	Site	x	y	z	U_{iso}
H(19)	4*a*	0.3817	0.5927	1.0885	0.047
H(20)	4*a*	0.4745	0.2418	1.1109	0.055
H(1)	4*a*	0.3263	1.2349	0.8003	0.063
H(2)	4*a*	0.3760	0.7544	0.9634	0.061
H(3A)	4*a*	0.4310	0.9766	0.6849	0.053
H(3B)	4*a*	0.3814	0.9815	0.6040	0.053
H(5A)	4*a*	0.8573	0.8544	0.7256	0.090
H(5B)	4*a*	0.9087	0.7537	0.7611	0.090
H(2A)	4*a*	0.1622	1.1836	0.6517	0.046
H(2B)	4*a*	0.2280	1.2538	0.6660	0.046
H(3)	4*a*	0.3040	1.0744	0.6535	0.053
H(4)	4*a*	0.2337	0.7279	0.6791	0.053
H(5C)	4*a*	0.1617	1.0972	0.7652	0.045
H(5D)	4*a*	0.2333	1.0214	0.8139	0.045
H(6)	4*a*	0.3234	1.1629	0.9622	0.044
H(7)	4*a*	0.1757	1.2624	0.9785	0.066
H(10)	4*a*	0.0732	1.5551	0.7006	0.051
H(12)	4*a*	0.2676	1.6863	0.8452	0.058
H(13)	4*a*	0.3055	1.4594	0.8455	0.049
H(15A)	4*a*	0.5405	0.5975	1.0035	0.045
H(15B)	4*a*	0.4693	0.5203	0.9589	0.045
H(16)	4*a*	0.5194	0.7570	0.7847	0.053
H(17)	4*a*	0.3768	0.6276	0.8059	0.047
H(18A)	4*a*	0.5394	0.6927	1.1157	0.043
H(18B)	4*a*	0.4736	0.7660	1.0996	0.043
H(23)	4*a*	0.6314	1.0533	1.0616	0.050
H(25)	4*a*	0.4377	1.1941	0.9294	0.056
H(26)	4*a*	0.3987	0.9696	0.9270	0.048
H(27A)	4*a*	0.5205	0.3599	0.7884	0.126
H(27B)	4*a*	0.5963	0.4142	0.8439	0.126
H(27C)	4*a*	0.5513	0.4015	0.8770	0.126
H(28A)	4*a*	0.6626	0.2362	0.9808	0.134
H(28B)	4*a*	0.6855	0.2412	0.9228	0.134
H(28C)	4*a*	0.6660	0.0960	0.9421	0.134
H(29)	4*a*	0.5608	0.0195	0.8465	0.072

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}
Mn(1)	4*a*	0.34469(2)	0.72964(3)	0.63193(2)	0.0308(2)	0.0313(2)	0.0282(1)	0.0038(1)	0.0183(1)	–0.0003(2)
Cl(1)	4*a*	0.28229(3)	0.51225(6)	0.62873(4)	0.0440(3)	0.0371(3)	0.0526(3)	–0.0036(2)	0.0285(3)	0.0011(3)
Cl(2)	4*a*	0.32501(4)	0.14830(7)	0.51471(5)	0.0612(4)	0.0466(4)	0.0756(5)	0.0028(3)	0.0403(4)	0.0141(4)
F(1)	4*a*	0.1448(1)	1.7724(2)	0.7708(1)	0.087(1)	0.0344(8)	0.073(1)	0.0112(8)	0.050(1)	–0.0003(8)
F(2)	4*a*	0.09691(7)	1.3019(2)	0.6879(1)	0.0300(8)	0.0503(9)	0.081(1)	–0.0086(6)	0.0185(8)	–0.0233(9)
F(3)	4*a*	0.60590(7)	0.7994(2)	1.0721(1)	0.0297(7)	0.0479(9)	0.072(1)	0.0054(6)	0.0170(7)	0.0189(8)
F(4)	4*a*	0.5605(1)	1.2758(2)	0.9987(1)	0.076(1)	0.0360(8)	0.067(1)	–0.0119(7)	0.0421(9)	0.0011(8)
N(1)	4*a*	0.23507(9)	1.0440(2)	0.6784(1)	0.038(1)	0.037(1)	0.035(1)	0.0030(8)	0.0240(8)	–0.0037(8)
N(2)	4*a*	0.2083(1)	0.9233(2)	0.6866(2)	0.058(1)	0.045(1)	0.083(2)	–0.009(1)	0.054(1)	–0.019(1)
N(3)	4*a*	0.2855(1)	0.8712(2)	0.6611(1)	0.039(1)	0.038(1)	0.044(1)	0.0021(8)	0.0277(9)	–0.0046(9)
N(4)	4*a*	0.22192(9)	1.1617(2)	0.8790(1)	0.0333(9)	0.039(1)	0.0298(9)	–0.0007(8)	0.0198(8)	0.0004(8)
N(5)	4*a*	0.1685(1)	1.1961(3)	0.8823(1)	0.034(1)	0.107(2)	0.039(1)	–0.003(1)	0.022(1)	–0.011(1)
N(6)	4*a*	0.2683(1)	1.2253(2)	1.0017(1)	0.036(1)	0.043(1)	0.029(1)	0.0005(8)	0.0187(8)	0.0003(8)
N(7)	4*a*	0.47812(9)	0.6509(2)	0.8883(1)	0.0341(9)	0.034(1)	0.032(1)	–0.0003(8)	0.0181(8)	0.0009(8)
N(8)	4*a*	0.5290(1)	0.6970(3)	0.8829(1)	0.033(1)	0.067(1)	0.037(1)	–0.004(1)	0.0201(9)	0.002(1)
N(9)	4*a*	0.4285(1)	0.6989(2)	0.7629(1)	0.037(1)	0.046(1)	0.031(1)	0.0006(8)	0.0195(8)	0.0006(9)
N(10)	4*a*	0.46591(9)	0.5555(2)	1.0907(1)	0.035(1)	0.036(1)	0.034(1)	–0.0033(7)	0.0201(8)	0.0040(8)
C(19)	4*a*	0.4125(1)	0.5262(3)	1.0929(2)	0.045(1)	0.039(1)	0.045(1)	–0.000(1)	0.033(1)	0.002(1)
C(20)	4*a*	0.4636(1)	0.3377(3)	1.1048(2)	0.044(1)	0.039(1)	0.064(2)	0.009(1)	0.036(1)	0.017(1)
N(13)	4*a*	0.5849(1)	0.2155(3)	0.8667(2)	0.058(2)	0.064(2)	0.061(2)	–0.010(1)	0.038(1)	–0.012(1)
O(1)	4*a*	0.30961(8)	1.2082(2)	0.8237(1)	0.0309(8)	0.057(1)	0.0369(9)	0.0112(8)	0.0187(7)	0.0079(8)
O(2)	4*a*	0.39306(8)	0.7138(2)	0.9434(1)	0.0283(8)	0.051(1)	0.0360(9)	–0.0088(7)	0.0153(7)	–0.0061(8)
O(3)	4*a*	0.40607(9)	0.9262(2)	0.6433(1)	0.0478(9)	0.0394(9)	0.0416(9)	0.0021(7)	0.0229(8)	–0.0025(8)
O(4)	4*a*	0.4826(1)	0.1184(3)	0.7715(1)	0.061(1)	0.074(2)	0.068(1)	–0.007(1)	0.024(1)	–0.021(1)
O(5)	4*a*	0.8814(1)	0.8020(3)	0.7664(2)	0.060(1)	0.089(2)	0.090(2)	0.020(1)	0.051(1)	0.036(1)
C(1)	4*a*	0.2375(1)	1.2188(2)	0.7723(1)	0.030(1)	0.038(1)	0.027(1)	0.0024(9)	0.0150(9)	–0.0021(9)
C(2)	4*a*	0.2119(1)	1.1818(3)	0.6854(1)	0.045(1)	0.041(1)	0.029(1)	0.011(1)	0.021(1)	0.002(1)
C(3)	4*a*	0.2798(1)	1.0094(3)	0.6625(2)	0.053(2)	0.040(1)	0.057(2)	0.004(1)	0.042(1)	0.001(1)
C(4)	4*a*	0.2409(1)	0.8240(3)	0.6762(2)	0.049(1)	0.041(1)	0.059(2)	–0.006(1)	0.040(1)	–0.013(1)
C(5)	4*a*	0.2104(1)	1.1117(2)	0.8046(1)	0.045(1)	0.034(1)	0.034(1)	–0.0025(9)	0.024(1)	–0.006(1)
C(6)	4*a*	0.2800(1)	1.1800(3)	0.9503(1)	0.034(1)	0.043(1)	0.032(1)	0.0043(9)	0.018(1)	0.002(1)
C(7)	4*a*	0.1996(2)	1.2330(4)	0.9576(2)	0.039(2)	0.094(2)	0.038(2)	0.001(1)	0.025(1)	–0.012(1)
C(8)	4*a*	0.2129(1)	1.3693(2)	0.7722(1)	0.029(1)	0.036(1)	0.028(1)	–0.0002(8)	0.0179(9)	–0.0014(9)
C(9)	4*a*	0.1443(1)	1.4040(3)	0.7309(1)	0.030(1)	0.038(1)	0.037(1)	–0.0071(9)	0.017(1)	–0.011(1)
C(10)	4*a*	0.1199(1)	1.5366(3)	0.7294(2)	0.037(1)	0.046(1)	0.043(1)	0.007(1)	0.022(1)	–0.000(1)
C(11)	4*a*	0.1678(1)	1.6406(3)	0.7724(2)	0.060(2)	0.033(1)	0.045(1)	0.003(1)	0.036(1)	0.000(1)
C(12)	4*a*	0.2362(1)	1.6144(3)	0.8158(2)	0.051(2)	0.038(1)	0.047(2)	–0.011(1)	0.023(1)	–0.009(1)
C(13)	4*a*	0.2587(1)	1.4779(3)	0.8157(2)	0.030(1)	0.045(1)	0.039(1)	–0.0035(9)	0.015(1)	–0.003(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(14)	4*a*	0.4652(1)	0.7230(3)	0.9952(1)	0.028(1)	0.040(1)	0.029(1)	–0.0037(9)	0.0143(9)	0.0017(9)
C(15)	4*a*	0.4916(1)	0.6101(3)	0.9647(1)	0.041(1)	0.036(1)	0.032(1)	0.0021(9)	0.020(1)	0.005(1)
C(16)	4*a*	0.4970(1)	0.7237(3)	0.8068(2)	0.038(1)	0.058(2)	0.040(2)	–0.005(1)	0.024(1)	0.001(1)
C(17)	4*a*	0.4190(1)	0.6538(3)	0.8166(1)	0.032(1)	0.049(1)	0.035(1)	–0.005(1)	0.019(1)	–0.002(1)
C(18)	4*a*	0.4897(1)	0.6914(3)	1.0816(1)	0.037(1)	0.039(1)	0.033(1)	–0.0110(9)	0.021(1)	0.000(1)
N(12)	4*a*	0.4998(1)	0.4338(3)	1.0981(2)	0.044(1)	0.051(1)	0.078(2)	0.010(1)	0.043(1)	0.025(1)
N(11)	4*a*	0.40974(9)	0.3882(2)	1.1023(1)	0.036(1)	0.035(1)	0.041(1)	–0.0008(8)	0.0255(9)	0.0060(8)
C(21)	4*a*	0.4904(1)	0.8715(2)	0.9940(1)	0.033(1)	0.036(1)	0.027(1)	0.0004(9)	0.0159(9)	0.0035(9)
C(22)	4*a*	0.5592(1)	0.9048(3)	1.0326(1)	0.031(1)	0.038(1)	0.038(1)	0.0011(9)	0.016(1)	0.008(1)
C(23)	4*a*	0.5846(1)	1.0369(3)	1.0350(2)	0.034(1)	0.047(1)	0.041(1)	–0.009(1)	0.020(1)	0.001(1)
C(24)	4*a*	0.5375(1)	1.1432(3)	0.9961(2)	0.056(1)	0.031(1)	0.038(1)	–0.006(1)	0.029(1)	–0.000(1)
C(25)	4*a*	0.4686(1)	1.1201(3)	0.9558(2)	0.049(1)	0.037(1)	0.045(1)	0.010(1)	0.022(1)	0.010(1)
C(26)	4*a*	0.4456(1)	0.9852(3)	0.9548(2)	0.033(1)	0.043(1)	0.039(1)	0.0022(9)	0.017(1)	0.005(1)
C(27)	4*a*	0.5612(2)	0.3600(4)	0.8419(3)	0.090(3)	0.066(2)	0.110(3)	–0.013(2)	0.065(2)	–0.015(2)
C(28)	4*a*	0.6557(2)	0.1955(6)	0.9338(2)	0.050(2)	0.155(4)	0.058(2)	–0.015(2)	0.029(2)	–0.007(2)
C(29)	4*a*	0.5432(2)	0.1102(4)	0.8287(2)	0.061(2)	0.059(2)	0.065(2)	–0.001(1)	0.038(2)	–0.006(2)

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