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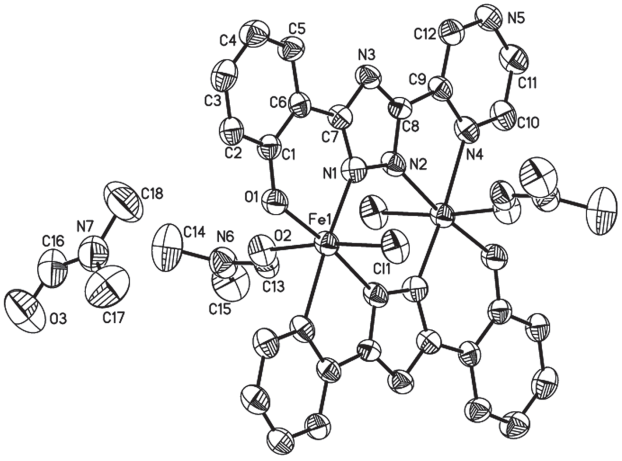
Crystal structure of dinuclear dichloridobis (dimethylformamide-k*O*)bis[μ_2-3-(2-oxyphenyl)-5-(pyrazin-2-yl)-1,2,4-triazol-1ido-κ^4-*O*,*N*:*N*′,*N*″ (2^-)]diiron(III) — dimethylformamide (1/1), $C_{36}H_{42}Cl_2Fe_2N_{14}O_6$



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Abstract

$C_{36}H_{42}Cl_2Fe_2N_{14}O_6$, monoclinic, $P2_1/c$ (no. 14), $a = 10.44(4)$ Å, $b = 9.13(3)$ Å, $c = 22.46(7)$ Å, $\beta = 100.88(4)°$, $V = 2101(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0599$, $wR_{ref}(F^2) = 0.1563$, $T = 296(2)$ K.

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The title complex 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 1: Data collection and handling.

Crystal:	Brown blocks
	Size 0.21 × 0.17 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	8.8 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	50.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8256, 3765, 0.082
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2586
$N(param)_{refined}$:	275
Programs:	SHELX [6], Bruker programs [7]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1963(4)	0.7271(5)	0.36794(18)	0.0360(10)
C2	0.2492(4)	0.7777(5)	0.31858(18)	0.0396(11)
H2	0.2533	0.7145	0.2865	0.048*
C3	0.2956(5)	0.9200(6)	0.3167(2)	0.0496(13)
H3	0.3295	0.9508	0.2834	0.059*
C4	0.2918(5)	1.0174(5)	0.3644(2)	0.0483(13)
H4	0.3225	1.1128	0.3631	0.058*
C5	0.2416(5)	0.9689(5)	0.4131(2)	0.0404(11)
H5	0.2399	1.0326	0.4453	0.049*
C6	0.1925(4)	0.8255(5)	0.41607(18)	0.0345(10)
C7	0.1399(4)	0.7833(5)	0.47039(18)	0.0352(10)
C8	0.0802(4)	0.7891(5)	0.55417(18)	0.0346(10)
C9	0.0510(4)	0.8266(5)	0.61386(17)	0.0350(10)
C10	−0.0393(5)	0.7440(5)	0.69327(18)	0.0437(12)
H10	−0.0811	0.6721	0.7119	0.052*
C11	−0.0063(5)	0.8777(5)	0.72266(19)	0.0466(12)
H11	−0.0272	0.8921	0.7607	0.056*
C12	0.0836(5)	0.9603(5)	0.6440(2)	0.0445(12)
H12	0.1263	1.0321	0.6257	0.053*
C13	0.2534(5)	0.3066(5)	0.5030(2)	0.0473(12)
H13	0.1865	0.2908	0.5243	0.057*
C14	0.4817(6)	0.2701(9)	0.4950(3)	0.091(2)
H14A	0.5086	0.1756	0.4832	0.136*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H14B	0.5528	0.3158	0.5219	0.136*
H14C	0.4562	0.3301	0.4597	0.136*
C15	0.3920(6)	0.1645(7)	0.5812(2)	0.0773(19)
H15A	0.3286	0.1910	0.6052	0.116*
H15B	0.4780	0.1821	0.6040	0.116*
H15C	0.3827	0.0626	0.5707	0.116*
C16	0.5928(6)	0.2491(8)	0.3337(3)	0.0768(19)
H16	0.6533	0.2777	0.3676	0.092*
C17	0.3946(7)	0.3078(8)	0.2609(3)	0.100(2)
H17A	0.4050	0.2088	0.2482	0.150*
H17B	0.4055	0.3739	0.2290	0.150*
H17C	0.3090	0.3199	0.2700	0.150*
C18	0.4792(9)	0.4760(8)	0.3456(4)	0.110(3)
H18A	0.5517	0.4869	0.3787	0.165*
H18B	0.3995	0.4757	0.3610	0.165*
H18C	0.4783	0.5561	0.3178	0.165*
Cl1	−0.15312(13)	0.57711(15)	0.36218(5)	0.0534(4)
Fe1	0.04924(6)	0.48491(7)	0.41301(3)	0.0348(2)
N1	0.0836(3)	0.6507(4)	0.47673(14)	0.0361(9)
N2	0.0445(4)	0.6548(4)	0.53164(14)	0.0358(9)
N3	0.1399(4)	0.8741(4)	0.51851(15)	0.0394(9)
N4	−0.0112(4)	0.7194(4)	0.63910(14)	0.0367(9)
N5	0.0541(4)	0.9859(4)	0.69861(17)	0.0468(11)
N6	0.3716(4)	0.2528(5)	0.52581(18)	0.0525(11)
N7	0.4915(5)	0.3391(6)	0.3145(2)	0.0693(13)
O1	0.1519(3)	0.5880(3)	0.36724(12)	0.0419(8)
O2	0.2282(3)	0.3770(4)	0.45436(13)	0.0479(8)
O3	0.6111(6)	0.1325(6)	0.3099(3)	0.123(2)

Source of material

A mixture of $FeCl_3{\cdot}6H_2O$ (18.5 mg, 0.05 mmol) and 3-(2-oxyphenyl)-5-(pyrazin-2-yl)-1,2,4-triazole (H_2L) (11.5 mg, 0.05 mmol) in DMF (7.0 mL) and ethanol (3.0 mL) was stirred for 2 h at room temperature. After filtration, the filtrate was kept undisturbed and slowly evaporated for 2 weeks and brown well shaped crystals were obtained, in a yield of 32% (based on Fe).

Experimental details

Hydrogen atoms were included in the refinement at calculated positions [C—$H_{aromatic}$ = 0.93 Å] and treated as riding models with $U_{iso}(H) = 1.2U_{eq}(C)$.

Discussion

Triazolates are candidates to construct coordination complexes because they can span metal centers through the –N–N– group from a triazole unit [1]. Recently, some triazole-bridged complexes have been investigated due to their novel structures and interesting properties [2–4].

The molecular structure of the title structure is made up of discrete neutral $[Fe_2(L)_2(Cl)_2(DMF)_2]$ complex. Fe(1) has six-coordination distorted octahedral geometry (*cf.* the figure). The octahedral coordination contains three nitrogen atoms from two triazolate ligands, two oxygen atoms from one triazolate ligand and one dimethylformamide ligand, and one chlorido ligand. In title complex, the bond lengths of Fe—N are 2.067(6), 2.142(5) and 2.197(7) Å, whereas the bond length of Fe—O to the ligand is 1.873(5) Å. The Fe—O bond to DMF is slightly longer at 2.160(6) Å, which is comparable to those in literature [5]. The stability of the solid state structure of title complex is enhanced by hydrogen bonding and π—π stacking. Additional dimethylformamide molecules form intermolecular hydrogen bonds.

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