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The crystal structure of bis(ferrocenecarboxylato- $\kappa^2 O, O'$)bis[4-(dimethylamino)pyridine- κN] copper(II) – acetonitrile(1/2), $C_{40}H_{44}CuO_4Fe_2N_6$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.25 × 0.13 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.37 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-III CMOS, φ and ω
$\theta_{\max}$, completeness:	26.2°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3819, 3819, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3089
$N(\text{param})_{\text{refined}}$:	246
Programs:	SHELX [1], Diamond [2], Bruker [3]

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Abstract

$C_{40}H_{44}CuO_4Fe_2N_6$, triclinic, $P\bar{1}$ (no. 2), $a = 7.4758(4)$ Å, $b = 7.7447(5)$ Å, $c = 16.2929(10)$ Å, $\alpha = 87.111(2)^\circ$, $\beta = 85.196(2)^\circ$, $\gamma = 89.736(2)^\circ$, $V = 938.82(10)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0672$, $wR_{\text{ref}}(F^2) = 0.1316$, $T = 150(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Ferrocenecarboxylic acid (0.069 g, 0.3 mmol) was dissolved in a methanol solution (6 mL) of $Cu(CH_3CN)_4BF_4$ (0.095 g,

0.3 mmol) under ultrasonication. To the resulting yellow solution triethylamine (42 μ L, 0.3 mmol) was added, after 10 min 4-(dimethylamino)pyridine (DMAP) (0.122 g, 0.6 mmol) was added under ultrasonication. The cloudy mixture was sealed and heated to 70 °C for 20 h. After cooled to room temperature, it was filtered, and the filtrate was evaporated at 5 °C. After 2 weeks, blue block crystals of title complex formed.

Experimental details

All hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms.

Comment

Heterometallic complexes have been widely applied for medical use [4, 5]. Although ferrocene and its derivatives are popular molecules for biological applications [6, 7], there are few examples of heterometallic complexes to be used in medical useage. Preparation and revelation of heterometallic complexes constructed by reaction of ferrocene derivatives with other metals [8, 9] are the key problems for their application in medical use.

Here, we mix ferrocenecarboxylic acid (FcCOOH) with $Cu(CH_3CN)BF_4$ under the assistance of 4-(dimethylamino)pyridine (DMAP) to get the title complex. The asymmetric unit contains 0.5 copper ions, one ferrocenecarboxylato(-1), one DMAP and one free CH_3CN . In the mononuclear title complex, the Cu^{II} ion, located on a crystallographic inversion centre, is six coordinated by two N atoms of two DMAP ligands in apical positions and four O atoms from

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Cu1	0.500000	0.500000	0.500000	0.0106(3)
Fe1	0.49974(11)	0.53615(11)	0.17113(5)	0.0148(3)
O1	0.4377(6)	0.7117(5)	0.3866(3)	0.0176(9)
O2	0.6355(5)	0.4967(5)	0.3874(2)	0.0136(9)
N1	0.6848(7)	0.6512(6)	0.5384(3)	0.0134(11)
N2	1.0485(7)	0.9742(6)	0.6395(3)	0.0171(11)
N3	0.8868(14)	0.2079(13)	0.0418(6)	0.079(3)
C1	0.6579(8)	0.8233(7)	0.5423(4)	0.0145(13)
H1	0.552145	0.870335	0.521536	0.017*
C2	0.7736(8)	0.9354(8)	0.5745(4)	0.0163(13)
H2	0.745742	1.055002	0.576625	0.020*
C3	0.9345(8)	0.8695(7)	0.6042(3)	0.0133(12)
C4	0.9681(8)	0.6920(8)	0.5965(4)	0.0160(13)
H4	1.077012	0.642011	0.612456	0.019*
C5	0.8405(8)	0.5922(7)	0.5656(4)	0.0138(13)
H5	0.863843	0.471787	0.563092	0.017*
C6	1.2196(9)	0.9130(9)	0.6630(5)	0.0282(16)
H6A	1.300468	1.011460	0.665579	0.042*
H6B	1.271192	0.834061	0.622221	0.042*
H6C	1.203682	0.851880	0.717251	0.042*
C7	1.0124(8)	1.1594(8)	0.6438(4)	0.0208(14)
H7A	1.080849	1.206704	0.686237	0.031*
H7B	0.883876	1.177598	0.657833	0.031*
H7C	1.048235	1.217958	0.590284	0.031*
C8	0.5558(7)	0.6218(7)	0.3505(3)	0.0116(12)
C9	0.6085(8)	0.6575(8)	0.2615(3)	0.0140(13)
C10	0.5246(9)	0.7809(8)	0.2100(4)	0.0209(15)
H10	0.423468	0.859130	0.227785	0.025*
C11	0.6071(10)	0.7732(9)	0.1295(4)	0.0269(17)
H11	0.572238	0.843374	0.080144	0.032*
C12	0.7417(9)	0.6433(9)	0.1290(4)	0.0252(16)
H12	0.820200	0.608317	0.079893	0.030*
C13	0.7460(8)	0.5723(9)	0.2119(4)	0.0193(15)
H13	0.828776	0.479967	0.231279	0.023*
C14	0.3558(10)	0.3350(10)	0.2281(4)	0.0276(16)
H14	0.355028	0.295615	0.287566	0.033*
C15	0.2391(9)	0.4590(10)	0.1942(5)	0.0296(17)
H15	0.142143	0.523522	0.225778	0.036*
C16	0.2846(10)	0.4779(10)	0.1077(5)	0.0309(17)
H16	0.225493	0.556791	0.067697	0.037*
C17	0.4283(10)	0.3628(9)	0.0887(4)	0.0265(17)
H17	0.489727	0.347099	0.032656	0.032*
C18	0.4715(10)	0.2757(9)	0.1627(4)	0.0224(15)
H18	0.568547	0.187502	0.167856	0.027*
C19	0.9713(14)	0.1213(13)	0.0828(6)	0.053(2)
C20	1.0770(13)	0.0124(12)	0.1332(5)	0.056(3)
H20A	1.002826	−0.083003	0.158746	0.084*
H20B	1.122269	0.079868	0.176395	0.084*
H20C	1.178370	−0.034290	0.099169	0.084*

two symmetry-related FcCOO[−], which are asymmetrically bonded in the equatorial plane. This coordination is very similar to the complexes bis(CH₃COO-κ²O,O')bis(DMAP)copper(II) [10], bis(CH₃COO-κ²O,O')bis(DMAP)copper(II) [11], and bis[ethyl(trifluoroacetyl)acetato-κ²O,O']bis(DMAP)]copper(II) [12].

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