Crystal structure of μ-oxido-*bis*{2,2-[ethylidene(*bis*-(nitrilomethylidyne))] diphenolato} iron(III), $[Fe_2O(C_{16}H_{14}N_2)2]\cdot2.5(C_7H_8)$, $C_{49.50}H_{48}Fe_2N_4O_5$

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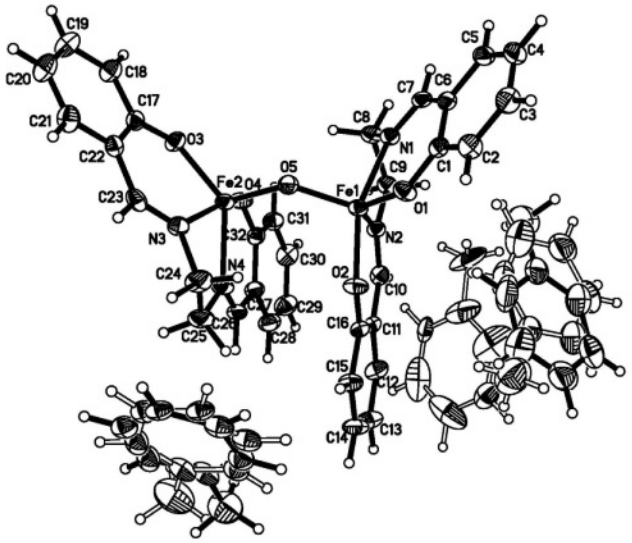

Abstract

$C_{49.50}H_{48}Fe_2N_4O_5$, monoclinic, $P2_1/c$ (no. 14), a = 12.7141(9) Å, b = 16.559(1) Å, c = 21.164(2) Å, β = 100.628(1)°, V = 4379.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0624, $wR_{ref}(F^2)$ = 0.1592, T = 187 K.

Table 1. Data collection and handling.

Crystal:	red rectangles, size 0.05×0.10×0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.14 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	23589, 8135
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5710
$N(param)_{refined}$:	470
Programs:	SHELX [12]

Source of material

For the preparation of the title complex, ethylene diamine (1.5 g, 0.025 mol) and salicylaldehyde (6.1 g, 0.05 mol) were dissolved in ethanol (50ml). The mixture was stirred for 10 min at room temperature to give a clear yellow solution. The ligand was obtained after recrystallization from the solution. Iron(III) acetylacetonate (0.93 g, 0.25 mmol) and *N*,*N*'-*bis*(salicylidene) ethylenediamine (0.07 g, 0. 25 mmol) were combined in a flask with 20ml toluene, the reaction took 24 hours under 100°C. The yielded solution was evaporated in the air for one week. A suitable red single-crystal was carefully selected by using a polarizing microscope and mounted in a glass capillary. All reagents were obtained from commercial sources and used without further purification.

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Experimental details

All H-atoms were positioned geometrically (C–H = 0.95 - 0.99 Å) and refined a riding model with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5 Ueq(C) for methyl H atoms.

Discussion

Schiff base ligands such as the salen ligand are commonly used in the formation of different complex with a great variety of different metal ions [1,2]. Studies of these complexes are important in understanding magnetic properties [3-12]. As an extension of this work, a novel dinuclear iron(III) complex (I) is reported here. Its most interesting feature is a bridging oxide ligand. From the crystal structure the coordination number of iron (III) is 5. The title molecule does not posses non crystallographic symmetry. The nitrogen and oxygen atoms around iron(III) generate trigonal bipyramidal geometry with the oxygen atom O5 as the apex. The Schiff base ligand shows quadridentate coordination of the iron(III) ion and constitutes an independent unit. Two independent units are bridged by oxygen atom O5 to form a butterfly binuclear crystal structure. The flexibility of the Fe–O–Fe motif allows the complex to adopt the most favorable conformation. The bond angle O2–Fe1–N2 [87.09(15)°] and O1–Fe1–N1 [86.37(16)°] agree with corresponding values reported for analogous Schiff bases [1]. The bond distance between central metal iron(III) ion and nitrogen Fe1–N1 [2.101(4) Å], Fe1–N2 [2.107(4) Å], Fe2–N3 [2.088(4) Å] and Fe2–N4 [2.100(4) Å] are also similar and consistent with reported counterparts [2.05-2.12 Å] in literature. The bond distance Fe1–O1 [1.916(4) Å], Fe1–O2 [1.930(3) Å], Fe2–O3 [1.920(3) Å], Fe2–O4 [1.927(4) Å] also show no big difference with these reported (1.90-2.01Å) [2]. The bond distance Fe1–O5 [1.790(4) Å] is shorter than that of bond distance between iron atom and oxygen atom from ligands, indicating interaction between the bridging oxygen atom and the iron atom. In the asymmetric unit, the crystal contains 2.5 disordered toluene solvent molecules. No classical hydrogen bond was found.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(2)	4*e*		0.4266	0.0640	1.2448	0.060
H(3)	4*e*		0.3806	0.0015	1.3327	0.064
H(4)	4*e*		0.2053	–0.0430	1.3312	0.061
H(5)	4*e*		0.0771	–0.0224	1.2392	0.055
H(7)	4*e*		0.0165	0.0309	1.1358	0.043
H(8A)	4*e*		–0.0609	0.0884	1.0433	0.049
H(8B)	4*e*		0.0115	0.0726	0.9897	0.049

Table 2. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(9A)	4*e*		–0.0333	0.2073	0.9760	0.049
H(9B)	4*e*		–0.0152	0.2231	1.0521	0.049
H(10)	4*e*		0.0829	0.3194	0.9848	0.047
H(12)	4*e*		0.1848	0.4276	0.9605	0.060
H(13)	4*e*		0.3459	0.4909	0.9558	0.070
H(14)	4*e*		0.5043	0.4192	0.9848	0.066
H(15)	4*e*		0.5059	0.2897	1.0250	0.058
H(18)	4*e*		0.0950	–0.1859	0.8325	0.055
H(19)	4*e*		0.1705	–0.3081	0.8139	0.068
H(20)	4*e*		0.3574	–0.3235	0.8311	0.072
H(21)	4*e*		0.4651	–0.2136	0.8645	0.063
H(23)	4*e*		0.4935	–0.0764	0.8927	0.050
H(24A)	4*e*		0.5414	0.0500	0.9190	0.068
H(24B)	4*e*		0.4749	0.0786	0.9727	0.068
H(25A)	4*e*		0.4696	0.1897	0.9081	0.069
H(25B)	4*e*		0.4516	0.1362	0.8437	0.069
H(26)	4*e*		0.3201	0.2585	0.8535	0.053
H(28)	4*e*		0.1821	0.3520	0.8253	0.063
H(29)	4*e*		0.0054	0.3907	0.8078	0.070
H(30)	4*e*		–0.1261	0.2954	0.8189	0.065
H(31)	4*e*		–0.0797	0.1643	0.8486	0.052
C(33)	4*e*	0.523	0.6591(5)	0.2901(2)	0.8881(3)	0.043(2)
C(34)	4*e*	0.523	0.6649(5)	0.2343(4)	0.8397(2)	0.048(2)
H(34)	4*e*	0.523	0.6450	0.2497	0.7959	0.057
C(35)	4*e*	0.523	0.6997(5)	0.1561(3)	0.8555(3)	0.044(2)
H(35)	4*e*	0.523	0.7036	0.1180	0.8224	0.053
C(36)	4*e*	0.523	0.7288(5)	0.1336(3)	0.9196(4)	0.041(2)
H(36)	4*e*	0.523	0.7526	0.0801	0.9304	0.049
C(37)	4*e*	0.523	0.7230(5)	0.1893(5)	0.9680(2)	0.057(3)
H(37)	4*e*	0.523	0.7429	0.1740	1.0118	0.068
C(38)	4*e*	0.523	0.6882(6)	0.2676(4)	0.9522(3)	0.055(3)
H(38)	4*e*	0.523	0.6843	0.3057	0.9852	0.067
C(39)	4*e*	0.523	0.622(1)	0.3743(6)	0.8684(6)	0.099(4)
H(39A)	4*e*	0.523	0.6216	0.4071	0.9069	0.149
H(39B)	4*e*	0.523	0.5495	0.3720	0.8427	0.149
H(39C)	4*e*	0.523	0.6706	0.3984	0.8428	0.149
C(33')	4*e*	0.477	0.6618(6)	0.2689(4)	0.8671(3)	0.058(3)
C(34')	4*e*	0.477	0.6846(7)	0.1911(5)	0.8489(2)	0.064(3)
H(34')	4*e*	0.477	0.6769	0.1777	0.8047	0.077
C(35')	4*e*	0.477	0.7187(6)	0.1329(3)	0.8956(4)	0.045(3)
H(35')	4*e*	0.477	0.7343	0.0797	0.8832	0.054
C(36')	4*e*	0.477	0.7300(5)	0.1525(4)	0.9603(3)	0.035(2)
H(36')	4*e*	0.477	0.7533	0.1127	0.9922	0.042
C(37')	4*e*	0.477	0.7072(6)	0.2303(4)	0.9785(2)	0.044(2)
H(37')	4*e*	0.477	0.7149	0.2437	1.0227	0.053
C(38')	4*e*	0.477	0.6731(6)	0.2885(3)	0.9318(4)	0.049(3)
H(38')	4*e*	0.477	0.6575	0.3416	0.9442	0.059
C(39')	4*e*	0.477	0.622(1)	0.3313(9)	0.8171(7)	0.129(6)

Table 2. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(39D)	4*e*	0.477	0.6102	0.3823	0.8382	0.193
H(39E)	4*e*	0.477	0.5545	0.3130	0.7909	0.193
H(39F)	4*e*	0.477	0.6752	0.3394	0.7896	0.193
C(40)	4*e*	0.5	–0.0305(8)	0.4725(6)	1.0086(4)	0.084(3)
C(41)	4*e*	0.5	–0.0458(7)	0.4950(6)	0.9443(4)	0.101(9)
H(41)	4*e*	0.5	–0.0988	0.4688	0.9136	0.121
C(42)	4*e*	0.5	0.0164(7)	0.5559(6)	0.9248(3)	0.096(4)
H(42)	4*e*	0.5	0.0059	0.5713	0.8809	0.115
C(43)	4*e*	0.5	0.0940(7)	0.5943(5)	0.9697(4)	0.074(5)
H(43)	4*e*	0.5	0.1365	0.6359	0.9564	0.088
C(44)	4*e*	0.5	0.1093(6)	0.5718(5)	1.0340(3)	0.071(3)
H(44)	4*e*	0.5	0.1623	0.5980	1.0647	0.086
C(52)	4*e*	0.5	0.0471(7)	0.5109(5)	1.0535(3)	0.077(7)
H(52)	4*e*	0.5	0.0575	0.4955	1.0974	0.093
C(53)	4*e*	0.5	–0.100(2)	0.410(1)	1.0278(8)	0.17(1)
H(53A)	4*e*	0.5	–0.0797	0.4003	1.0742	0.257
H(53B)	4*e*	0.5	–0.0906	0.3595	1.0047	0.257
H(53C)	4*e*	0.5	–0.1743	0.4271	1.0173	0.257
C(45)	4*e*	0.555	0.1987(6)	0.3380(4)	1.1971(4)	0.070(3)
C(46)	4*e*	0.555	0.1856(5)	0.4141(4)	1.2224(4)	0.074(3)
H(46)	4*e*	0.555	0.1178	0.4298	1.231	0.089
C(47)	4*e*	0.555	0.2717(7)	0.4670(4)	1.2351(5)	0.102(4)
H(47)	4*e*	0.555	0.2628	0.519	1.2524	0.123
C(48)	4*e*	0.555	0.3709(6)	0.4439(5)	1.2225(5)	0.107(4)
H(48)	4*e*	0.555	0.4298	0.4801	1.2312	0.129
C(49)	4*e*	0.555	0.3840(6)	0.3679(6)	1.1973(5)	0.131(5)
H(49)	4*e*	0.555	0.4518	0.3521	1.1887	0.157
C(50)	4*e*	0.555	0.2979(7)	0.3149(4)	1.1846(5)	0.117(5)
H(50)	4*e*	0.555	0.3068	0.263	1.1673	0.140
C(51)	4*e*	0.555	0.104(1)	0.2854(8)	1.1815(8)	0.148(6)
H(51A)	4*e*	0.555	0.1247	0.2341	1.1642	0.222
H(51B)	4*e*	0.555	0.0497	0.3119	1.1493	0.222
H(51C)	4*e*	0.555	0.0748	0.2752	1.2205	0.222
C(45')	4*e*	0.445	0.3111(6)	0.3788(5)	1.1942(5)	0.080(4)
C(46')	4*e*	0.445	0.2539(7)	0.4393(5)	1.2184(5)	0.074(4)
H(46')	4*e*	0.445	0.2865	0.4900	1.2301	0.089
C(47')	4*e*	0.445	0.1488(7)	0.4255(5)	1.2254(6)	0.101(5)
H(47')	4*e*	0.445	0.1097	0.4668	1.2419	0.121
C(48')	4*e*	0.445	0.1010(6)	0.3512(6)	1.2082(5)	0.086(4)
H(48')	4*e*	0.445	0.0291	0.3418	1.213	0.103
C(49')	4*e*	0.445	0.1582(8)	0.2908(5)	1.1840(6)	0.099(5)
H(49')	4*e*	0.445	0.1255	0.2400	1.1723	0.119
C(50')	4*e*	0.445	0.2633(8)	0.3046(5)	1.1770(6)	0.082(4)
H(50')	4*e*	0.445	0.3024	0.2632	1.1604	0.098
C(51')	4*e*	0.445	0.420(1)	0.396(1)	1.184(1)	0.147(8)
H(51D)	4*e*	0.445	0.4498	0.3479	1.1668	0.221
H(51E)	4*e*	0.445	0.4654	0.4108	1.2252	0.221
H(51F)	4*e*	0.445	0.4185	0.4408	1.1536	0.221

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}
Fe(1)	4*e*	0.23957(4)	0.12586(3)	1.05285(3)	0.0216(3)	0.0329(3)	0.0407(4)	–0.0024(2)	0.0077(2)	0.0008(3)
Fe(2)	4*e*	0.23648(4)	0.04723(3)	0.90759(3)	0.0249(3)	0.0352(3)	0.0431(4)	–0.0071(2)	0.0098(3)	–0.0025(3)
O(1)	4*e*	0.3102(2)	0.0968(2)	1.1377(1)	0.028(2)	0.055(2)	0.045(2)	–0.007(1)	0.006(1)	0.006(2)
O(2)	4*e*	0.3470(2)	0.2046(2)	1.0429(1)	0.029(2)	0.037(2)	0.062(2)	–0.005(1)	0.010(1)	0.009(1)
O(3)	4*e*	0.1857(2)	–0.0549(2)	0.8710(1)	0.025(1)	0.039(2)	0.061(2)	–0.006(1)	0.009(1)	–0.015(1)
O(4)	4*e*	0.1043(2)	0.1013(2)	0.8724(1)	0.029(2)	0.038(2)	0.059(2)	–0.007(1)	0.004(1)	0.009(1)
O(5)	4*e*	0.2460(2)	0.0511(2)	0.9927(1)	0.040(2)	0.037(2)	0.046(2)	–0.002(1)	0.013(1)	–0.002(1)
N(1)	4*e*	0.0987(2)	0.0839(2)	1.0802(2)	0.023(2)	0.036(2)	0.043(2)	–0.004(1)	0.007(2)	–0.001(2)
N(2)	4*e*	0.1247(2)	0.2146(2)	1.0180(2)	0.024(2)	0.033(2)	0.042(2)	–0.002(1)	0.008(1)	–0.002(2)
N(3)	4*e*	0.3906(2)	0.0035(2)	0.9056(2)	0.023(2)	0.049(2)	0.052(2)	–0.010(2)	0.008(2)	–0.009(2)
N(4)	4*e*	0.3163(3)	0.1495(2)	0.8815(2)	0.030(2)	0.044(2)	0.049(2)	–0.013(2)	0.008(2)	0.001(2)
C(1)	4*e*	0.2788(3)	0.0605(2)	1.1867(2)	0.038(2)	0.032(2)	0.038(2)	0.000(2)	0.007(2)	–0.002(2)
C(2)	4*e*	0.3548(4)	0.0469(3)	1.2430(2)	0.045(3)	0.054(3)	0.049(3)	0.000(2)	0.004(2)	0.002(2)
C(3)	4*e*	0.3276(4)	0.0096(3)	1.2953(2)	0.067(3)	0.049(3)	0.041(3)	0.006(2)	0.000(2)	0.005(2)
C(4)	4*e*	0.2237(4)	–0.0169(3)	1.2948(2)	0.072(3)	0.041(2)	0.043(3)	0.000(2)	0.019(3)	0.008(2)
C(5)	4*e*	0.1483(4)	–0.0043(2)	1.2401(2)	0.054(3)	0.033(2)	0.054(3)	–0.002(2)	0.023(2)	0.002(2)
C(6)	4*e*	0.1728(3)	0.0343(2)	1.1859(2)	0.038(2)	0.030(2)	0.041(2)	0.000(2)	0.013(2)	–0.004(2)
C(7)	4*e*	0.0863(3)	0.0486(2)	1.1322(2)	0.027(2)	0.031(2)	0.053(3)	–0.008(2)	0.015(2)	–0.007(2)
C(8)	4*e*	0.0069(3)	0.1029(2)	1.0295(2)	0.022(2)	0.049(2)	0.050(3)	–0.009(2)	0.005(2)	–0.001(2)

Table 3. continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(9)	4*e*	0.0117(3)	0.1931(2)	1.0178(2)	0.023(2)	0.051(2)	0.049(3)	–0.001(2)	0.007(2)	0.002(2)
C(10)	4*e*	0.1433(3)	0.2860(2)	0.9993(2)	0.033(2)	0.039(2)	0.045(3)	0.003(2)	0.007(2)	–0.004(2)
C(11)	4*e*	0.2470(3)	0.3204(2)	0.9982(2)	0.035(2)	0.031(2)	0.037(2)	–0.007(2)	0.006(2)	–0.005(2)
C(12)	4*e*	0.2500(4)	0.3994(2)	0.9743(2)	0.049(3)	0.037(2)	0.062(3)	–0.002(2)	0.005(2)	0.000(2)
C(13)	4*e*	0.3449(4)	0.4368(3)	0.9706(2)	0.062(3)	0.039(2)	0.071(3)	–0.016(2)	0.003(3)	0.011(2)
C(14)	4*e*	0.4385(4)	0.3944(3)	0.9888(2)	0.049(3)	0.049(3)	0.067(3)	–0.022(2)	0.012(2)	0.006(2)
C(15)	4*e*	0.4397(3)	0.3171(3)	1.0125(2)	0.036(2)	0.049(3)	0.058(3)	–0.012(2)	0.006(2)	0.001(2)
C(16)	4*e*	0.3431(3)	0.2779(2)	1.0186(2)	0.033(2)	0.032(2)	0.042(2)	–0.010(2)	0.007(2)	–0.005(2)
C(17)	4*e*	0.2331(3)	–0.1231(2)	0.8607(2)	0.037(2)	0.041(2)	0.034(2)	–0.003(2)	0.007(2)	–0.005(2)
C(18)	4*e*	0.1707(3)	–0.1906(3)	0.8395(2)	0.036(2)	0.047(2)	0.051(3)	–0.003(2)	–0.001(2)	–0.008(2)
C(19)	4*e*	0.2155(4)	–0.2635(3)	0.8284(2)	0.060(3)	0.042(3)	0.063(3)	0.000(2)	–0.005(3)	–0.009(2)
C(20)	4*e*	0.3265(4)	–0.2728(3)	0.8382(2)	0.061(3)	0.046(3)	0.070(3)	0.012(2)	0.007(3)	–0.012(3)
C(21)	4*e*	0.3896(4)	–0.2077(3)	0.8581(2)	0.042(3)	0.059(3)	0.055(3)	0.012(2)	0.008(2)	–0.005(2)
C(22)	4*e*	0.3461(3)	–0.1318(2)	0.8696(2)	0.032(2)	0.046(2)	0.034(2)	0.002(2)	0.008(2)	–0.003(2)
C(23)	4*e*	0.4192(3)	–0.0666(3)	0.8903(2)	0.026(2)	0.060(3)	0.041(2)	0.001(2)	0.011(2)	0.000(2)
C(24)	4*e*	0.4701(3)	0.0671(3)	0.9263(3)	0.028(2)	0.062(3)	0.078(4)	–0.012(2)	0.003(2)	–0.010(3)
C(25)	4*e*	0.4332(3)	0.1412(3)	0.8870(3)	0.029(2)	0.062(3)	0.082(4)	–0.018(2)	0.016(2)	–0.007(3)
C(26)	4*e*	0.2732(3)	0.2166(3)	0.8614(2)	0.048(3)	0.045(2)	0.041(3)	–0.023(2)	0.011(2)	–0.005(2)
C(27)	4*e*	0.1609(3)	0.2351(2)	0.8496(2)	0.047(3)	0.038(2)	0.040(2)	–0.012(2)	0.007(2)	–0.000(2)
C(28)	4*e*	0.1291(4)	0.3136(3)	0.8307(2)	0.062(3)	0.040(2)	0.054(3)	–0.013(2)	0.008(2)	0.003(2)
C(29)	4*e*	0.0248(4)	0.3368(3)	0.8198(2)	0.068(3)	0.037(2)	0.067(3)	0.002(2)	0.010(3)	0.006(2)
C(30)	4*e*	–0.0529(4)	0.2800(3)	0.8266(2)	0.052(3)	0.050(3)	0.056(3)	0.006(2)	0.001(2)	0.002(2)
C(31)	4*e*	–0.0252(3)	0.2020(2)	0.8444(2)	0.037(2)	0.044(2)	0.045(3)	–0.003(2)	0.000(2)	0.002(2)
C(32)	4*e*	0.0813(3)	0.1770(2)	0.8563(2)	0.041(2)	0.037(2)	0.034(2)	–0.008(2)	0.003(2)	–0.003(2)

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