Yang Liguo*, Zhang Zhen, Niu Yongsheng, Wang Fang and Tian Dayong
Crystal structure of aqua-dichlorido-(4-(((3-ethoxy-2-oxidobenzylidene)hydrazono)(oxido)methyl)pyridin-1-ium- $\kappa^3 N, O, O'$)iron(III),
 $C_{15}H_{16}Cl_2N_3O_4Fe$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.08 × 0.06 × 0.04 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.18 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18605, 3981, 0.017
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3641
$N(\text{param})_{\text{refined}}$:	258
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.50777 (3)	0.41040 (2)	0.66793 (2)	0.02454 (7)
Cl1	0.33527 (5)	0.33308 (2)	0.69679 (4)	0.03778 (11)
Cl2	0.31552 (6)	0.48323 (2)	0.65479 (5)	0.04196 (11)
O1	0.64449 (14)	0.40756 (5)	0.89001 (12)	0.0290 (2)
O2	0.44854 (14)	0.40170 (5)	0.45607 (12)	0.0300 (2)
O3	0.31207 (17)	0.41924 (6)	0.16413 (13)	0.0402 (3)
O4	0.67867 (16)	0.47691 (6)	0.66340 (15)	0.0343 (3)
H41	0.675 (3)	0.4822 (11)	0.585 (3)	0.057 (8)*
H42	0.670 (3)	0.5075 (12)	0.697 (3)	0.062 (8)*
N1	0.78490 (18)	0.32936 (6)	0.83829 (15)	0.0333 (3)
N2	0.68413 (16)	0.34384 (6)	0.69017 (14)	0.0283 (3)
N3	1.0670 (2)	0.35455 (8)	1.38555 (18)	0.0452 (4)
H3	1.134 (3)	0.3521 (10)	1.475 (3)	0.061 (7)*
C1	0.8464 (2)	0.40060 (8)	1.19415 (19)	0.0368 (4)
H1	0.763611	0.430284	1.162395	0.044*
C2	0.9533 (3)	0.39705 (9)	1.3421 (2)	0.0461 (5)
H2	0.946176	0.424797	1.413241	0.055*
C4	1.0822 (2)	0.31410 (9)	1.2913 (2)	0.0444 (4)
H4A	1.163671	0.284086	1.327416	0.053*
C5	0.9797 (2)	0.31600 (8)	1.14178 (19)	0.0384 (4)
H5	0.989703	0.287486	1.073566	0.046*
C6	0.86106 (19)	0.36020 (7)	1.09158 (17)	0.0287 (3)
C7	0.75342 (19)	0.36606 (7)	0.92894 (16)	0.0273 (3)
C8	0.7033 (2)	0.31207 (7)	0.58725 (18)	0.0319 (3)
H8	0.782883	0.281287	0.618076	0.038*
C9	0.6119 (2)	0.32021 (7)	0.42840 (17)	0.0288 (3)
C10	0.49312 (19)	0.36543 (7)	0.37048 (17)	0.0261 (3)
C11	0.4196 (2)	0.37282 (7)	0.21209 (17)	0.0289 (3)
C12	0.4578 (2)	0.33485 (8)	0.11767 (18)	0.0341 (4)
H12	0.406749	0.339978	0.011796	0.041*
C13	0.5707 (2)	0.28913 (8)	0.17654 (19)	0.0369 (4)
H13	0.594138	0.262736	0.110708	0.044*

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Abstract

$C_{15}H_{16}Cl_2N_3O_4Fe$, monoclinic, $P2_1/n$ (no. 14), $a = 8.7419(15)$ Å, $b = 22.717(4)$ Å, $c = 9.6256(16)$ Å, $\beta = 112.546(2)^\circ$, $V = 1765.4(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0255$, $wR_{\text{ref}}(F^2) = 0.0622$, $T = 298$ K [1–3].

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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

The ligand 3-ethoxysalicylaldehyde-isonicotinohydrazide was synthesized by condensing 3-ethoxysalicylaldehyde with equimolar isonicotinohydrazide in ethanol. Iron trichloride hexahydrate (1 mmol) was added to a 30 mL methanol solution containing 3-ethoxysalicylaldehyde-isonicotinohydrazide (1 mmol). The resulting mixture was refluxed with stirring for 1.5 h, and then cooled slowly to room

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C14	0.6477 (2)	0.28220 (7)	0.32882 (19)	0.0357 (4)
H14	0.726209	0.251454	0.368043	0.043*
C15 ^a	0.2826 (5)	0.44513 (17)	0.0158 (4)	0.0350 (8)
H15A ^a	0.374753	0.435170	−0.015864	0.042*
H15B ^a	0.274741	0.488518	0.019983	0.042*
C16 ^a	0.1215 (5)	0.41976 (19)	−0.0942 (4)	0.0535 (11)
H16A ^a	0.097782	0.436068	−0.194578	0.080*
H16B ^a	0.130945	0.376840	−0.097416	0.080*
H16C ^a	0.031366	0.429956	−0.061724	0.080*
C15 ^b	0.2208 (8)	0.4208 (3)	−0.0017 (6)	0.0406 (14)
H15C ^b	0.297887	0.428962	−0.052499	0.049*
H15D ^b	0.166062	0.382456	−0.038190	0.049*
C16 ^b	0.0964 (6)	0.4679 (2)	−0.0337 (6)	0.0473 (16)
H16D ^b	0.033177	0.470417	−0.142425	0.071*
H16E ^b	0.020989	0.459265	0.017174	0.071*
H16F ^b	0.152208	0.505556	0.002930	0.071*

Occupancies: ^a = 0.605(5), ^b = 0.395(5).

temperature and filtered. Black prism-shaped crystals were obtained from the solution after several days. Yield (0.13 g, 30%) m.p. 263–269 K. FTIR (KBr) ν (cm^{−1}): 3201, 1694, 1564, 1467, 1255, 1040. Anal. Calc. for C₁₉H₁₇N₃O₂ (%): C, 41.96; H, 3.73; N, 9.79. Found (%): C, 41.92; H, 3.70; N, 9.70.

Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.98 Å) and refined as riding with $U_{iso}(H) = 1.2$ – $1.5 U_{eq}(C)$. Other H atoms are refined freely (see Table 2).

Discussion

Schiff base play an important role in inorganic chemistry, because they can form stable complexes with most transition metal ions and Ln ions. The application of the complexes with the schiff base ligands is based on their biological activity [4]. Especially, the mode of chelation with transition metal ions present in the living system have been of significant interest [5–8]. Many complexes with aroylhydrazone ligands have been reported [9, 10].

The molecular structure of the complex and the labelling of the atoms as showing in the figure. The Fe(III) atom is meridionally coordinated by O, N, O of the ylidic hydrazone ligand. Two chlorido and one aqua ligand complete the distorted pseudo-octahedral coordination sphere. The bond lengths of Fe1–O1, Fe1–O2, Fe1–O4, Fe1–N2, Fe1–Cl1 and Fe1–Cl2 are similar with those reported in the literature [11–20]. The angles of Cl1–Fe1–Cl2, O1–Fe1–O4, O2–Fe1–O4, O1–Fe1–Cl1, O2–Fe1–Cl2, O1–Fe1–N2, O2–Fe1–N2 and N2–Fe1–Cl1 are 93.11°, 84.85°, 87.48°, 89.81°, 96.93°, 75.89°, 85.59° and 86.01°, respectively.

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