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Crystal structure of bis(2-methoxy-6-((2-(pyrimidin-2-yl)hydrazono)methyl)phenolato- κ^3N , N' , O)iron(III) perchlorate, $C_{24}H_{22}N_8O_8ClFe$

Table 1: Data collection and handling.

Crystal:	Brown cube
Size:	0.40 × 0.20 × 0.10 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	6.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	70.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8616, 4936, 0.048
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4310
$N(\text{param})_{\text{refined}}$:	381
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.47233(4)	0.44331(4)	0.27461(4)	0.02041(17)
O1	0.6275(2)	0.5249(2)	0.21089(19)	0.0276(5)
O2	0.7826(3)	0.6853(3)	0.0807(2)	0.0468(7)
O3	0.3741(2)	0.5990(2)	0.29073(19)	0.0264(5)
O4	0.3119(3)	0.8236(2)	0.3545(2)	0.0344(6)
N1	0.4693(3)	0.1274(3)	0.0816(3)	0.0344(7)
N2	0.5179(3)	0.2576(3)	0.2185(2)	0.0249(5)
N3	0.4027(3)	0.3376(3)	0.0720(3)	0.0343(7)
H3	0.3735	0.3336	0.0071	0.041*
N4	0.3861(3)	0.4463(3)	0.1234(2)	0.0274(6)
N5	0.3177(3)	0.2196(3)	0.5844(3)	0.0329(6)
N6	0.3371(3)	0.3399(3)	0.4000(2)	0.0249(5)
N7	0.5050(3)	0.3162(3)	0.5079(2)	0.0297(6)
H7	0.5407	0.2869	0.5692	0.036*
N8	0.5729(3)	0.3888(3)	0.4189(2)	0.0243(5)
C1	0.2156(3)	0.3109(3)	0.3893(3)	0.0304(7)
H1	0.1801	0.3413	0.3213	0.036*
C2	0.1418(4)	0.2389(4)	0.4732(3)	0.0369(8)
H2	0.0558	0.2198	0.4651	0.044*
C3	0.1974(4)	0.1947(4)	0.5708(3)	0.0341(8)
H3A	0.1476	0.1447	0.6303	0.041*
C4	0.3817(3)	0.2916(3)	0.4977(3)	0.0259(6)
C5	0.6923(3)	0.4075(3)	0.4293(3)	0.0255(6)
H5	0.7255	0.3684	0.4961	0.031*
C6	0.7772(3)	0.4818(3)	0.3488(3)	0.0261(6)
C7	0.9023(4)	0.4978(4)	0.3761(3)	0.0339(8)
H7A	0.9279	0.4552	0.4448	0.041*

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Abstract

$C_{24}H_{22}N_8O_8ClFe$, triclinic, $P\bar{1}$ (no. 2), $a = 10.4568(4)$ Å, $b = 10.5977(4)$ Å, $c = 12.0793(6)$ Å, $\alpha = 82.119(4)^\circ$, $\beta = 83.277(4)^\circ$, $\gamma = 87.206(3)^\circ$, $Z = 2$, $V = 1316.11(9)$ Å³, $R_{\text{gt}}(F) = 0.0565$, $wR_{\text{ref}}(F^2) = 0.1609$, $T = 173(10)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C8	0.9872(4)	0.5731(4)	0.3058(4)	0.0424(9)
H8	1.0719	0.5811	0.3246	0.051*
C9	0.9494(4)	0.6386(4)	0.2059(4)	0.0428(9)
H9	1.0073	0.6941	0.1584	0.051*
C10	0.8279(4)	0.6230(4)	0.1756(3)	0.0350(8)
C11	0.7404(3)	0.5408(3)	0.2444(3)	0.0250(6)
C12	0.8610(6)	0.7842(7)	0.0182(5)	0.086(2)
H12A	0.8139	0.8309	−0.0410	0.130*
H12B	0.9408	0.7462	−0.0159	0.130*
H12C	0.8819	0.8430	0.0690	0.130*
C13	0.5836(3)	0.1573(3)	0.2692(3)	0.0314(7)
H13	0.6247	0.1675	0.3335	0.038*
C14	0.5926(4)	0.0412(4)	0.2302(3)	0.0363(8)
H14	0.6377	−0.0294	0.2670	0.044*
C15	0.5339(4)	0.0307(4)	0.1357(4)	0.0372(8)
H15	0.5396	−0.0491	0.1076	0.045*
C16	0.4660(3)	0.2367(3)	0.1259(3)	0.0284(7)
C17	0.3286(3)	0.5428(3)	0.0685(3)	0.0297(7)
H17	0.3045	0.5332	−0.0033	0.036*
C18	0.2996(3)	0.6618(3)	0.1094(3)	0.0263(7)
C19	0.2387(4)	0.7578(4)	0.0379(3)	0.0323(7)
H19	0.2234	0.7415	−0.0348	0.039*
C20	0.2018(4)	0.8721(4)	0.0711(3)	0.0353(8)
H20	0.1612	0.9351	0.0218	0.042*
C21	0.2234(4)	0.8980(3)	0.1782(3)	0.0326(7)
H21	0.1962	0.9779	0.2018	0.039*
C22	0.2848(3)	0.8066(3)	0.2499(3)	0.0266(7)
C23	0.3224(3)	0.6854(3)	0.2177(3)	0.0235(6)
C24	0.2742(5)	0.9434(4)	0.3923(3)	0.0417(9)
H24A	0.3149	1.0123	0.3395	0.063*
H24B	0.3014	0.9448	0.4670	0.063*
H24C	0.1803	0.9552	0.3962	0.063*
Cl1	−0.06798(9)	0.14698(10)	0.25431(8)	0.0403(2)
O5	−0.1315(4)	0.2575(4)	0.2010(3)	0.0650(10)
O6	0.0664(3)	0.1687(3)	0.2515(3)	0.0525(8)
O7	−0.1251(4)	0.1236(5)	0.3686(3)	0.0733(12)
O8	−0.0846(4)	0.0407(4)	0.1951(4)	0.0660(10)

Source of material

All starting materials are commercially available, and are used without further purification. 2-Hydrazinopyrimidine and 2-methoxy-6-(pyrimidin-2-ylhydrazonomethyl)-phenol (H₂L) were prepared following reported procedures [5]. Perchlorate salts of metal complexes with organic ligands are potentially explosive and should be handled in small quantities with care.

The title compound [Fe(L)₂]ClO₄ was obtained according to the following procedure. 2-Methoxy-6-(pyrimidin-2-ylhydrazonomethyl)-phenol (0.1 mmol, 21.4 mg) was dissolved in the methanol (15 mL) under stirring, and then filtered to give a light yellow solution. Afterwards,

Fe(ClO₄)₂·6 H₂O (0.05 mmol, 18.1 mg) in deionized water (10 mL) was slowly diffused to the above solution in a sealed monotube. After several days, brown cube-shaped crystals were obtained. Yield: ca. 80%.

Experimental details

The H atoms were added using riding models. Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent C and N atoms, and 1.5*U*_{eq} of the parent O atoms.

Comment

Pyrimidine derivatives are important pharmaceutical intermediates, because of the antitumor, bactericidal, and plant growth promoting properties [6, 7]. After coordination to metal ions, pyrimidine derivatives can not only enhance the activity of the original drugs but also reduce their toxicity and side effects [8]. Many Schiff bases have antibacterial and antitumor activities that can be enhanced after forming complexes with transition metals. The Schiff base complexes have strong cell penetration and fat solubility, which makes the antibacterial range of the complexes expand, and it is not easy to produce drug resistance [9–12].

The title complex is a mononuclear iron(III) complex. The iron(III) ion in [Fe(L)₂]⁺ is hexa-coordinated by four nitrogen atoms and two oxygen atoms from two ligands to form a N₄O₂ octahedral configuration. The Fe–N and Fe–O bond distances are in the range of 2.125(3)–2.182(3) Å, and 1.906(2)–1.921(2) Å, respectively. There is apparent intermolecular hydrogen bonding interaction between the methoxy oxygen and the amide H–N group (O2···N3 = 2.866(4) Å, O4···N7 = 2.901(4) Å). The hydrogen bonds link adjacent molecules into a 1D supramolecular chain structure.

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