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Crystal structure of chlorido-(6,6'-((1,2-phenylenebis(azanylylidene))bis(methanylylidene))bis(3-methoxyphenolato)- $\kappa^4 O,N,N',O'$)iron(III), $C_{22}H_{18}ClN_2FeO_4$

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Abstract

$C_{22}H_{18}ClN_2FeO_4$, monoclinic, $P2_1/n$ (no. 14), $a = 7.0779(6)$ Å, $b = 19.0203(16)$ Å, $c = 14.7233(12)$ Å, $\beta = 103.1570(10)^\circ$, $V = 1930.1(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0278$, $wR_{ref}(F^2) = 0.1037$, $T = 296(2)$ 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.

Source of material

Schiff base ligand was purchased from Acros Ltd. Company and used without further purification, the other reagents were commercially available and used as purchased [1–3].

A mixture of $FeCl_3$ (64.88 mg, 0.4 mmol), Schiff base ligand 6,6'-((1,2-phenylenebis(azanylylidene))bis(methanylylidene))bis(3-methoxyphenolato)- $\kappa^4 O,N,N',O'$ iron(III),

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Table 1: Data collection and handling.

Crystal:	Turquoise block
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.95 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	16943, 3395, 0.037
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3162
$N(param)_{refined}$:	273
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Fe01	0.30499(3)	0.57403(2)	0.27829(2)	0.02516(13)
Cl1	0.05650(7)	0.53806(3)	0.33751(3)	0.04211(16)
O1	0.1978(2)	0.62039(6)	0.16391(8)	0.0355(3)
O2	0.39310(19)	0.65994(6)	0.33831(8)	0.0326(3)
O3	0.5999(2)	0.82894(7)	0.56774(9)	0.0395(3)
O4	0.0069(2)	0.68963(8)	−0.15386(9)	0.0467(4)
N1	0.3720(2)	0.49066(7)	0.19842(10)	0.0287(3)
N2	0.5473(2)	0.52629(7)	0.36698(9)	0.0272(3)
C1	−0.0276(4)	0.75961(12)	−0.12720(15)	0.0496(5)
H1A	−0.1416	0.7605	−0.1022	0.074*
H1B	−0.0465	0.7898	−0.1807	0.074*
H1C	0.0819	0.7757	−0.0807	0.074*
C2	0.0729(3)	0.64183(10)	−0.08579(12)	0.0338(4)
C3	0.1279(3)	0.57662(10)	−0.11636(13)	0.0392(5)
H3	0.1102	0.5672	−0.1797	0.047*
C4	0.2072(3)	0.52757(10)	−0.05215(13)	0.0379(4)
H4	0.2438	0.4846	−0.0728	0.045*
C5	0.2367(3)	0.53915(9)	0.04547(12)	0.0300(4)
C6	0.1750(2)	0.60472(9)	0.07536(11)	0.0283(4)
C7	0.0903(3)	0.65471(9)	0.00871(12)	0.0307(4)
H7	0.0454	0.6969	0.0278	0.037*
C8	0.3308(3)	0.48723(9)	0.10735(12)	0.0309(4)
H8	0.3673	0.4464	0.0810	0.037*
C9	0.4780(3)	0.43609(9)	0.25215(12)	0.0289(4)
C10	0.4900(3)	0.36671(9)	0.22252(13)	0.0365(4)
H10	0.4186	0.3528	0.1642	0.044*
C11	0.6062(3)	0.31909(9)	0.27887(14)	0.0404(5)
H11	0.6160	0.2733	0.2582	0.049*
C12	0.7093(3)	0.33910(10)	0.36680(15)	0.0405(5)
H12	0.7895	0.3067	0.4044	0.049*
C13	0.6943(3)	0.40662(10)	0.39921(13)	0.0358(4)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H13	0.7616	0.4192	0.4589	0.043*
C14	0.5787(3)	0.45552(9)	0.34265(12)	0.0283(4)
C15	0.6568(3)	0.55485(9)	0.44059(11)	0.0295(4)
H15	0.7550	0.5272	0.4758	0.035*
C17	0.5070(2)	0.67526(8)	0.41997(11)	0.0265(4)
C18	0.5013(3)	0.74255(9)	0.45586(11)	0.0286(4)
H18	0.4167	0.7756	0.4222	0.034*
C19	0.6197(3)	0.76134(9)	0.54122(12)	0.0305(4)
C16	0.6390(2)	0.62523(9)	0.47203(11)	0.0280(4)
C20	0.7508(3)	0.71299(10)	0.59292(12)	0.0346(4)
H20	0.8311	0.7256	0.6498	0.042*
C21	0.7577(3)	0.64644(9)	0.55736(12)	0.0334(4)
H21	0.8446	0.6142	0.5913	0.040*
C22	0.6944(3)	0.84893(10)	0.66067(13)	0.0403(4)
H22A	0.6531	0.8184	0.7044	0.061*
H22B	0.6611	0.8966	0.6717	0.061*
H22C	0.8324	0.8452	0.6683	0.061*

ylidene))bis(3-methoxyphenol) (49.64 mg, 0.4 mmol) and distilled water (10 mL) was sealed in a 23 mL Teflon-lined steel vessel (solution pH 2.5) and heated at 160 °C for 72 h, and then cooled to room temperature at a rate of 0.05 °C min⁻¹. Turquoise block crystals were obtained and washed with distilled water. The yield is ca. 32%. IR(KBr): 3256(s), 1597(s), 1518(s), 1497(s), 1448(s), 1356(s), 1245(s), 1174(s), 1113(s), 1073(s), 1005(s), 924(s), 894(s), 835(m), 773(m), 716(m), 619(m), 493(m) cm⁻¹.

Comment

There has been considerable interest in Schiff base ligands containing oxygen and imine nitrogen atoms and their metal complexes due to their variety of applications in chemistry, biology, physics and catalysis [4–6]. A new iron(III) complex with Schiff base ligand is synthesized and characterized. The structure is similar with the reference [7–9].

As shown in the Figure, one formula unit consists of one Schiff base ligand, one chlorido ligand and one Fe(III). The FeO1 ion is in a slightly distorted rectangular pyramid coordination environment, with one Cl atom, two nitrogen atoms and two oxygen atoms from Schiff base ligands. The bond lengths of FeO1–N1 and FeO1–N2 are 2.110 Å and 2.092 Å,

while the bond lengths of FeO1–O1 and FeO1–O2 are 1.894 Å and 1.899 Å, the distance length of FeO1–Cl is 2.240 Å.

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