Guojun Yu, Jiao Yang, Qiuling Yang and Qiong Wu*

{2-(((2-aminoethyl)imino)methyl)-6-bromophenolato-κ^3*N,N′*,O}iron(III) nitrate, $C_{18}H_{20}Br_2FeN_5O_5$

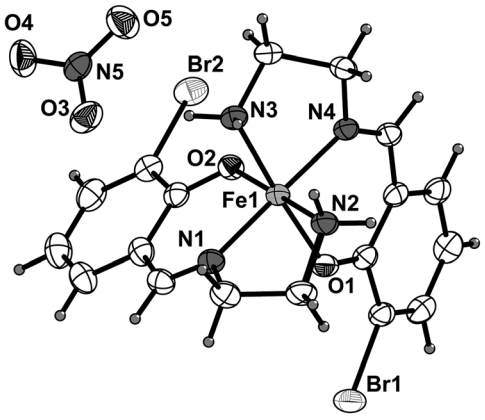

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.14 × 0.14 mm
Wavelength:	Mo *K*α vvradiation (0.71073 Å)
μ:	4.45 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18,432, 4359, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3625
$N(param)_{refined}$:	280
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_{18}H_{20}Br_2FeN_5O_5$, monoclinic, $P2_1/c$ (no. 14), a = 12.4534(3) Å, b = 10.8106(3) Å, c = 16.0339(4) Å, β = 94.0450(10)°, V = 2153.25(10) Å^3, Z = 4, $R_{gt}(F)$ = 0276, $wR_{ref}(F^2)$ = 0.0644, T = 170.0 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The educts 3-bromosalicylaldehyde (0.100 g, 0.5 mmol) and ethylenediamine (0.030 g, 0.5 mmol) were dissolved in 20 mL ethanol. This solution was stirred at room temperature for 24 h. After this, $Fe(NO_3)_3{\cdot}9H_2O$ (0.303 g, 0.75 mmol) was added to the above solution and the obtained black mixture was further stirred for 24 h and filtered. The acquired black filtrate was sealed in a beaker with parafilm and kept undisturbed at room temperature. The black block crystals of the title compound were afforded after one month by slow evaporation.

Experimental details

X-ray single crystal data was collected on a Bruker SMART APEX II diffractometer with rotating anode source (MoK*α* radiation, λ = 0.71073 Å) and equipped with an gas-flow apparatus at 148 K. Data reduction was performed using the program SAINT and empirical absorption corrections were made using SADABS [1]. The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions and refined using a riding model, with C–H = 0.98 (methylene), 0.95 Å (benzene) and 0.97 Å (methyl); U_{iso}(H) values were set 1.2 times for H atoms on methylene and benzene.

Comment

Schiff base is a kind of compound compounds characterized by imine functional group, which is usually formed by condensation react of aldehyde or ketone with amino group

***Corresponding author: Qiong Wu**, Department of Chemical Science and Technology, Kunming University, Yunnan, Kunming 65200, P. R. China, Phone: +86 87165098476, E-mail: wuqiongkm@163.com. https://orcid.org/0000-0001-7931-6750
Guojun Yu, Jiao Yang and Qiuling Yang, Department of Chemical Science and Technology, Kunming University, Yunnan, Kunming 65200, P. R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.44504 (2)	0.27792 (3)	0.46978 (2)	0.03645 (9)
Br2	0.83412 (2)	0.66895 (3)	0.67867 (2)	0.03740 (9)
Fe1	0.80676 (3)	0.24426 (3)	0.58941 (2)	0.02212 (9)
O1	0.67991 (13)	0.26419 (17)	0.52060 (11)	0.0278 (4)
O2	0.79868 (13)	0.40098 (16)	0.63919 (10)	0.0251 (4)
N1	0.72060 (16)	0.16425 (19)	0.66806 (13)	0.0247 (5)
N2	0.81762 (16)	0.0750 (2)	0.54060 (13)	0.0286 (5)
H2A	0.8175	0.0795	0.4839	0.034*
H2B	0.8798	0.0378	0.5606	0.034*
N3	0.94614 (16)	0.2197 (2)	0.65596 (13)	0.0257 (5)
H3A	0.9402	0.2455	0.7095	0.031*
H3B	0.9636	0.1379	0.6572	0.031*
N4	0.89309 (15)	0.3198 (2)	0.50900 (12)	0.0240 (5)
C1	0.6718 (2)	0.3491 (3)	0.74151 (15)	0.0282 (6)
C2	0.74007 (19)	0.4321 (2)	0.70010 (15)	0.0249 (5)
C3	0.74207 (19)	0.5557 (2)	0.72885 (16)	0.0270 (5)
C4	0.6818 (2)	0.5962 (3)	0.79215 (17)	0.0361 (7)
H4	0.6857	0.6802	0.8093	0.043*
C5	0.6151 (2)	0.5142 (3)	0.83108 (17)	0.0385 (7)
H5	0.5728	0.5420	0.8744	0.046*
C6	0.6111 (2)	0.3933 (3)	0.80611 (16)	0.0335 (6)
H6	0.5661	0.3374	0.8332	0.040*
C7	0.6667 (2)	0.2188 (2)	0.72282 (16)	0.0274 (6)
H7	0.6199	0.1695	0.7533	0.033*
C8	0.7113 (2)	0.0292 (2)	0.65639 (17)	0.0311 (6)
H8A	0.6403	−0.0001	0.6725	0.037*
H8B	0.7682	−0.0139	0.6915	0.037*
C9	0.7235 (2)	0.0034 (3)	0.56494 (17)	0.0320 (6)
H9A	0.7351	−0.0861	0.5560	0.038*
H9B	0.6579	0.0291	0.5309	0.038*
C10	1.00776 (19)	0.2897 (3)	0.52327 (16)	0.0281 (6)
H10A	1.0523	0.3518	0.4962	0.034*
H10B	1.0229	0.2071	0.5003	0.034*
C11	1.03162 (19)	0.2914 (3)	0.61751 (16)	0.0293 (6)
H11A	1.1029	0.2539	0.6323	0.035*
H11B	1.0326	0.3777	0.6383	0.035*
C12	0.85889 (19)	0.3930 (2)	0.45023 (15)	0.0257 (5)
H12	0.9104	0.4302	0.4172	0.031*
C13	0.66371 (19)	0.3529 (2)	0.46545 (15)	0.0256 (5)
C14	0.7466 (2)	0.4225 (2)	0.43111 (15)	0.0261 (5)
C15	0.7200 (2)	0.5175 (2)	0.37361 (16)	0.0298 (6)
H15	0.7760	0.5630	0.3505	0.036*
C16	0.6143 (2)	0.5464 (3)	0.34995 (17)	0.0342 (6)
H16	0.5977	0.6140	0.3135	0.041*
C17	0.5327 (2)	0.4757 (3)	0.37999 (16)	0.0320 (6)
H17	0.4597	0.4932	0.3627	0.038*
C18	0.55705 (19)	0.3802 (2)	0.43477 (15)	0.0268 (5)
O3	0.91234 (17)	0.31441 (19)	0.83053 (13)	0.0434 (5)
O4	0.95504 (18)	0.4777 (2)	0.90423 (12)	0.0459 (5)
O5	1.02913 (17)	0.4448 (2)	0.78920 (13)	0.0467 (5)
N5	0.96484 (17)	0.4114 (2)	0.84079 (13)	0.0301 (5)

[5, 6]. This type of compounds is flexible in stereochemistry and has a variety of coordination modes with different metal ions, and most of the obtained complexes exhibits diverse properties [7, 8 and references cited there]. As a part of our current research interest on the exploration of the regulating effect of Schiff base ligands on transition metal complexes [9–11], herein we report a new Fe(III) complex based on a halogenated Schiff base ligand.

The asymmetric unit is composed of one cationic Fe(III) complex and one nitrate counter-anion (see the figure). The coordination environment of the ion center is defined by two oxygen atoms and four nitrogen atoms from two salen type bromo-substitutional tridentate ligands. The nitrate anion interacts with the cationic complex through electrostatics and through hydrogen bonding. The bond lengths of Fe–O(N) range from 1.8744(17) to 1.999(2) Å, the perpendicular and trans bond angles exhibit a moderate distortion from an ideal octahedral symmetry which range from 84.31(9)° to 94.48(8)°, and 176.30(9)° to 178.33(9)°, respectively.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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