Crystal structure of 1,2-bis(salicylideneiminato)propanechromium(III) nitrate, $C_{17}H_{20}CrN_3O_7$

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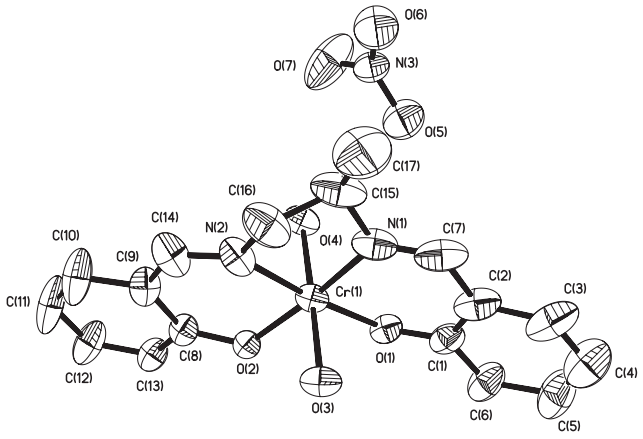

Abstract

$C_{17}H_{20}CrN_3O_7$, monoclinic, *C*12/*c*1 (No. 15), a = 18.905(7) Å, b = 13.915(5) Å, c = 14.822(6) Å, β = 97.246(7)°, V = 3868.0 Å^3, Z = 8, $R_{gt}(F)$ = 0.052, $wR_{ref}(F^2)$ = 0.125, T = 298 K.

Source of material

Reagents and solvents used were of commercially available reagent quality. The di-Schiff base L was prepared by the (2+1) condensation of salicylaldehyde with 1,2-diaminopropane in methanol at room temperature. Further isolation was not carried out and the ligand solution was subsequently used for the preparation of the metal complex. To a methanol solution (5 ml) of $Cr(NO_3)_2 \cdot 6H_2O$ (245 mg, 1 mmol) was added a methanol solution (3 ml) of L (1 mmol) with stirring for 30 min. Upon slow diffusion of diethyl ether into the above resulting solution for two days, large green prismatic crystals of the title complex were deposited and collected by filtration, respectively washed with methanol and diethyl ether and dried in a vacuum desiccator over silica gel. Yield 61%. Elemental analysis: found – C, 47.68%; H, 4.74%; N, 9.59%; calc. for $C_{17}H_{20}CrN_3O_7$ – C, 47.45%; H, 4.68%; N, 9.76%.

Experimental details

During the preparation of the crystals of the title complex, some viscous compounds are always accompanied. When the viscous compounds were washed away, the crystals (used for X-ray determination) became un-shiny. Reflections at high 2θ angles are very weak, so most of the reflections with $2\theta > 46°$ were removed from the data.

Discussion

The research in the design of infinite molecular components with specific chains and networks, or so-called crystal engineering, has become an area of current interest over recent years. Much work has focused on the use of tri- or tetra-dentate Schiff base to act as ligands to ligate metal ions, generating simple structure units, which are further linked by some special bridges to obtain oligomeric and polymeric metal complexes with infinite chain structures and two- or three-dimensional networks. During our exploring ways to synthesizing chromium(III) polymeric complexes with chain structure, we isolated a simple Cr(III) complex with a di-Schiff base.

The title complex exists as discrete molecule in the solid state. Each simplest structure unit of the complex consists of one complex cation and one nitrate anion. In the cation, the Cr(III) atom is six-coordinated to form a slightly distorted octahedron (the diagonal angles are 172.7(2)°, 173.7(2)°, and 178.6(2)°) with two nitrogen and two oxygen atoms from di-Schiff base ligand, and two oxygen atoms form two water molecules. The four atoms, N(1), N(2), O(1), and O(2) from the Schiff base constitute the coordination equatorial plane and the chromium atom is situated inside the plane with a very small deviation of 0.005 Å. The two oxygen atoms from the water molecules occupy the axial positions with significantly longer average Cr—O bond lengths of 1.975(5) Å than the mean metal-oxygen (phenoxo) distance of 1.914(4) Å. The very strong hydrogen bonds between the coordinated water molecules and phenoxo oxygen atoms, and water with nitrate oxygen atoms, join the neighboring cations along c-axis into a one-dimensional chain. There is a small channel inside each chain with the diameter about 2 Å.

Table 1. Data collection and handling.

Crystal:	green prism, size 0.13 × 0.28 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.36 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10772, 3855
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1117
$N(param)_{refined}$:	269
Programs:	SHELXTL [1], SHELXTL-plus [2]

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

Atom	Site	x	y	z	U_{iso}
H(5)	8*f*	0.7189	0.7819	0.3931	0.206
H(6)	8*f*	0.6514	0.9043	0.3660	0.215
H(7)	8*f*	0.5303	0.8910	0.3665	0.224
H(8)	8*f*	0.4802	0.7369	0.3848	0.163
H(9)	8*f*	0.7445	0.6170	0.4255	0.161
H(10)	8*f*	0.5561	0.0897	0.3707	0.204
H(11)	8*f*	0.4392	0.0415	0.3718	0.225
H(12)	8*f*	0.3518	0.1541	0.3621	0.176
H(13)	8*f*	0.3776	0.3140	0.3612	0.115
H(14)	8*f*	0.6440	0.2044	0.3659	0.154
H(15)	8*f*	0.7353	0.4130	0.4927	0.175
H(17A)	8*f*	0.7188	0.3829	0.3042	0.183
H(16B)	8*f*	0.7432	0.3052	0.3791	0.183
H(20A)	8*f*	0.8346	0.4951	0.4617	0.334
H(18B)	8*f*	0.8169	0.4647	0.3596	0.334
H(19C)	8*f*	0.8347	0.3862	0.4349	0.334
H(1)	8*f*	0.620(2)	0.514(2)	0.239(2)	0.06(1)
H(2)	8*f*	0.572(2)	0.479(3)	0.231(3)	0.04(2)
H(3)	8*f*	0.559(3)	0.431(5)	0.536(5)	0.09(4)
H(4)	8*f*	0.602(4)	0.459(5)	0.549(5)	0.17(5)

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}
Cr(1)	8*f*	0.58405(4)	0.46033(6)	0.39297(5)	0.0684(5)	0.1067(7)	0.0677(5)	–0.0051(5)	0.0152(3)	0.0038(4)
N(1)	8*f*	0.6808(3)	0.5128(5)	0.4194(3)	0.070(4)	0.179(6)	0.091(3)	–0.029(4)	0.016(3)	0.000(3)
N(2)	8*f*	0.6378(3)	0.3411(4)	0.3752(3)	0.088(4)	0.133(4)	0.094(3)	0.028(3)	0.036(2)	0.023(3)
N(3)	8*f*	0.7319(3)	0.4512(4)	0.6749(3)	0.087(4)	0.117(5)	0.110(4)	0.020(4)	0.013(3)	0.007(3)
O(1)	8*f*	0.5418(2)	0.5822(3)	0.4157(2)	0.083(2)	0.093(3)	0.081(2)	–0.024(2)	0.024(2)	–0.003(2)
O(2)	8*f*	0.4956(1)	0.3956(3)	0.3613(2)	0.076(2)	0.076(2)	0.068(2)	–0.005(2)	0.020(1)	0.004(2)
O(3)	8*f*	0.5861(3)	0.4949(4)	0.2646(3)	0.079(3)	0.143(4)	0.065(3)	–0.028(2)	0.024(2)	–0.001(3)
O(4)	8*f*	0.5828(4)	0.4223(5)	0.5216(3)	0.079(3)	0.139(5)	0.074(3)	0.003(3)	0.015(3)	0.009(3)
O(5)	8*f*	0.6987(2)	0.5179(4)	0.6353(4)	0.139(4)	0.146(4)	0.195(5)	0.045(3)	0.026(3)	–0.006(3)
O(6)	8*f*	0.7934(3)	0.4361(3)	0.6714(4)	0.099(4)	0.169(5)	0.344(8)	0.041(3)	–0.010(4)	–0.025(4)
O(7)	8*f*	0.6972(4)	0.3946(4)	0.7046(5)	0.216(6)	0.174(5)	0.372(9)	0.049(4)	0.157(6)	0.115(6)
C(1)	8*f*	0.5723(4)	0.6637(6)	0.4020(3)	0.132(7)	0.103(6)	0.069(4)	–0.025(5)	0.026(3)	–0.008(4)
C(2)	8*f*	0.6456(5)	0.6757(7)	0.4057(4)	0.112(7)	0.162(8)	0.095(4)	–0.073(6)	0.031(4)	–0.006(4)
C(3)	8*f*	0.6700(6)	0.7728(8)	0.3912(6)	0.21(1)	0.18(1)	0.138(7)	–0.087(9)	0.051(7)	–0.052(7)
C(4)	8*f*	0.6312(7)	0.8450(8)	0.3763(7)	0.21(1)	0.147(9)	0.198(8)	–0.071(9)	0.081(8)	–0.005(7)
C(5)	8*f*	0.5594(6)	0.8371(6)	0.3750(5)	0.28(1)	0.103(7)	0.192(8)	–0.028(7)	0.081(8)	–0.002(5)
C(6)	8*f*	0.5293(4)	0.7435(6)	0.3871(4)	0.187(7)	0.097(5)	0.134(5)	–0.050(6)	0.054(5)	–0.007(4)
C(7)	8*f*	0.6965(4)	0.6004(9)	0.4180(4)	0.079(6)	0.23(1)	0.098(5)	–0.055(7)	0.027(4)	–0.031(6)
C(8)	8*f*	0.4828(3)	0.3025(4)	0.3655(3)	0.097(4)	0.084(4)	0.076(3)	–0.011(4)	0.038(3)	0.008(3)
C(9)	8*f*	0.5389(4)	0.2319(5)	0.3712(4)	0.111(5)	0.096(5)	0.112(4)	0.015(4)	0.051(4)	0.006(3)
C(10)	8*f*	0.5205(5)	0.1358(6)	0.3705(5)	0.207(9)	0.080(6)	0.248(8)	0.024(5)	0.133(7)	0.015(5)
C(11)	8*f*	0.4504(6)	0.1064(5)	0.3693(6)	0.25(1)	0.063(5)	0.282(9)	0.012(7)	0.154(8)	0.001(5)
C(12)	8*f*	0.3992(4)	0.1734(6)	0.3646(4)	0.170(7)	0.090(6)	0.197(7)	–0.035(5)	0.088(5)	–0.019(5)
C(13)	8*f*	0.4146(3)	0.2698(4)	0.3635(3)	0.097(4)	0.088(4)	0.110(4)	–0.019(3)	0.043(3)	–0.005(3)
C(14)	8*f*	0.6124(4)	0.2549(6)	0.3703(4)	0.134(7)	0.159(7)	0.102(4)	0.055(5)	0.053(4)	0.026(5)
C(15)	8*f*	0.7372(4)	0.4405(7)	0.4322(5)	0.061(4)	0.27(1)	0.109(5)	–0.009(6)	0.017(4)	–0.011(6)
C(16)	8*f*	0.7141(4)	0.3621(6)	0.3657(5)	0.110(6)	0.217(8)	0.134(6)	0.049(5)	0.026(5)	0.014(6)
C(17)	8*f*	0.8122(4)	0.4472(6)	0.4212(6)	0.153(8)	0.233(9)	0.28(1)	–0.072(6)	0.022(6)	0.008(7)

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