Crystal structure of *trans*-diaqua-bis(salicylaldehydato-*O,O'*) chromium(III) chloride, $[\text{Cr}(\text{C}_7\text{H}_5\text{O}_2)_2(\text{H}_2\text{O})_2]\text{Cl}$

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Discussion

In the title complex, the Cr(III) atom is six-coordinated in form of a slightly distorted octahedron with four oxygen atoms from two salicylaldehyde anion ligands, and two oxygen atoms from two coordinated water molecules. The three diagonal angles for the Cr(III) octahedron are all 180°, and the other angles around Cr atom in the polyhedron are close to 90° (from 87.71(15)° to 92.29(15)°). The phenyl rings in the complex are defined planes, which is different from those in the similar complex $[\text{Ni}(\text{C}_7\text{H}_5\text{O}_2)_2(\text{H}_2\text{O})_2]$ [1]. The benzene rings in the latter complex are greatly distorted. In the presented crystal structure, the O(3)–H···O(2) hydrogen bonds link the complex along *c* to form one-dimensional chains. The chlorine atoms also contribute to the formation of hydrogen bonds.

Abstract

$\text{C}_{14}\text{H}_{14}\text{ClCrO}_6$, monoclinic, $P12_1/c1$ (No. 14), $a = 8.998(18)$ Å, $b = 18.68(4)$ Å, $c = 4.945(10)$ Å, $\beta = 105.95(2)^\circ$, $V = 799.2$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.069$, $wR_{\text{ref}}(F^2) = 0.170$, $T = 298$ K.

Source of material

All reagents and solvents were used as obtained without further purification. To a solution of salicylaldehyde (2 mmol, 244 mg) in 5 ml methanol was added a solution of $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ (1 mmol, 266 mg) in 5 ml methanol, respectively. The mixture was stood still in air for one week, large bright orange crystals were formed. The products were isolated, washed three times with methanol and dried in a vacuum using CaCl_2 (yield: 55%). C, H and N elemental analyses were performed on a Perkin-Elmer elemental analyzer.

Experimental details

The two H atoms attached to O(3) were refined with the bond length restraint $d(\text{H}—\text{O}) = 0.88$ Å and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{iso}}(\text{O})$. All the other H atoms were placed in geometrically idealised positions, constrained to ride on their parent atoms with C—H distances of 0.96 Å, and refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{iso}}(\text{C})$. The crystals used for X-ray measurements were small and fragile, resulting in large *R* values.

Table 1. Data collection and handling.

Crystal:	green block, size 0.17 × 0.25 × 0.28 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	9.06 cm ^{−1}
Diffractometer, scan mode:	Siemens SMART CCD, φ/ω
$2\theta_{\text{max}}$:	46.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3368, 111
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 883
$N(\text{param})_{\text{refined}}$:	114
Programs:	SADABS [2], SHELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
H(1)	4e	0.3389	0.3288	−0.3342	0.061
H(4)	4e	0.0868	0.4750	0.3425	0.065
H(5)	4e	−0.1089	0.3944	0.2252	0.081
H(6)	4e	−0.1075	0.3021	−0.1054	0.091
H(7)	4e	0.1006	0.2933	−0.2892	0.083
H(2)	4e	0.614(6)	0.4031(9)	0.330(9)	0.05(1)
H(3)	4e	0.649(7)	0.466(2)	0.497(5)	0.07(2)

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cr(1)	2 <i>d</i>	1/2	1/2	0	0.0451(7)	0.0323(7)	0.0345(7)	−0.0021(4)	0.0190(5)	−0.0019(4)
Cl(1)	4 <i>e</i>	0.4121(4)	0.7918(2)	0.1657(5)	0.122(2)	0.050(2)	0.040(1)	−0.009(2)	0.030(1)	−0.004(1)
O(1)	4 <i>e</i>	0.4579(4)	0.4101(2)	−0.2112(6)	0.056(2)	0.040(2)	0.043(2)	−0.005(2)	0.021(2)	−0.006(1)
O(2)	4 <i>e</i>	0.3268(4)	0.4851(2)	0.1648(7)	0.051(2)	0.044(2)	0.038(2)	−0.003(2)	0.020(1)	−0.003(1)
O(3)	4 <i>e</i>	0.6390(4)	0.4485(2)	0.3268(7)	0.054(2)	0.039(2)	0.045(2)	0.000(2)	0.021(2)	−0.001(2)
C(1)	4 <i>e</i>	0.3441(7)	0.3699(3)	−0.226(1)	0.073(3)	0.033(3)	0.045(3)	−0.003(3)	0.013(2)	−0.005(2)
C(2)	4 <i>e</i>	0.2210(6)	0.3796(3)	−0.096(1)	0.055(3)	0.037(3)	0.045(3)	−0.005(2)	0.010(2)	0.002(2)
C(3)	4 <i>e</i>	0.2186(5)	0.4372(3)	0.095(1)	0.042(3)	0.047(3)	0.044(3)	0.003(2)	0.011(2)	0.015(2)
C(4)	4 <i>e</i>	0.0900(6)	0.4392(3)	0.213(1)	0.052(3)	0.057(3)	0.057(3)	0.012(3)	0.024(2)	0.011(3)
C(5)	4 <i>e</i>	−0.0274(7)	0.3910(4)	0.144(1)	0.047(3)	0.072(4)	0.087(4)	−0.004(3)	0.027(3)	0.020(3)
C(6)	4 <i>e</i>	−0.0264(7)	0.3347(4)	−0.054(2)	0.053(3)	0.075(5)	0.091(5)	−0.021(3)	0.007(3)	0.013(4)
C(7)	4 <i>e</i>	0.0978(7)	0.3302(3)	−0.165(1)	0.074(4)	0.054(4)	0.075(4)	−0.012(3)	0.013(3)	0.003(3)

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