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Bis(ethylenediamine-κ^2N,N')(2-oxido-5-sulfonatobenzoato-κ^2O^1,O^2)chromium(III) dihydrate

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In the title compound, $[Cr(C_7H_3O_6S)(C_2H_8N_2)_2]\cdot 2H_2O$, the chromium(III) ion is at the centre of a compressed octahedron formed by four N atoms [Cr—N = 2.060 (5)–2.107 (5) Å] from two ethylenediamine molecules, one phenolate O atom [Cr—O = 1.899 (4) Å] and a carboxylate O atom [Cr—O = 1.929 (4) Å] from the 2-oxido-5-sulfonatobenzoate ligand. These two O atoms and two of the N atoms occupy the equatorial positions and the remaining N atoms occupy the axial positions. The crystal structure is stabilized by a number of intermolecular hydrogen bonds.

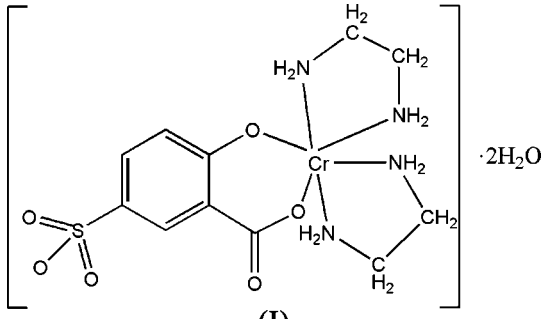
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.087
wR factor = 0.152
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Chromium is known to activate enzymes, maintain protein stability and enhance carbohydrate metabolism (Vincent, 2000). Organic chromium(III) sources have been shown to enhance the availability of chromium (Mertz, 1998). Nutritionists around the world believe that chromium is an essential nutrient and that organic chromium should be supplemented in most animal diets. Hence, a search has been underway to identify the biologically active form of chromium, *i.e.* the biomolecule that binds chromium(III) and possesses an intrinsic function associated with the action of insulin in mammals. Salicylic acid (a substance which led to the discovery of aspirin) and its analogues were chosen originally because of their low toxicity and because the structure of 5-salicylic acid is very similar to that of aspirin. We have designed and synthesized a new chromium complex, [(5-sulfosalicylic acid)(diethylenediamine)chromium(III)] dihydrate, (I).



(I)

Experimental

$CrCl_3\cdot 6H_2O$ (0.665 g, 0.0025 mol), 5-sulfosalicylic acid (0.67 g, 0.0025 mol) and zinc (0.1 g, 0.0015 mol) were added to methanol (30 ml) and then refluxed for 1 h. Ethylenediamine was added dropwise and the mixture was refluxed for a further 15 min. A pink precipitate formed in >70% yield. The precipitate was filtered off,

washed with methanol, and dried. The dried powder was dissolved in water and crystals suitable for X-ray analysis was obtained after two days at room temperature.

Crystal data

$[Cr(C_7H_3O_6S)(C_2H_8N_2)_2]\cdot 2H_2O$
$M_r = 423.39$
Monoclinic, $P2_1/c$
$a = 11.995$ (3) Å
$b = 10.138$ (3) Å
$c = 14.933$ (4) Å
$\beta = 105.081$ (4)°
$V = 1753.4$ (9) Å^3
$Z = 4$
$D_x = 1.604$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2398 reflections
$\theta = 2.5$–26.9°
$\mu = 0.82$ mm^{-1}
$T = 293$ (2) K
Needle, red
0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.735$, $T_{max} = 0.923$
6966 measured reflections
3061 independent reflections
2772 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 25.0°$
$h = -14 \rightarrow 14$
$k = -12 \rightarrow 11$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.087$
$wR(F^2) = 0.152$
$S = 1.31$
3061 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2 + 6.1162P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr1—O1	1.899 (4)	Cr1—N3	2.075 (5)
Cr1—O2	1.929 (4)	Cr1—N4	2.089 (5)
Cr1—N1	2.060 (5)	Cr1—N2	2.107 (5)
O1—Cr1—O2	91.85 (17)	N1—Cr1—N4	170.65 (19)
O1—Cr1—N1	93.87 (19)	N3—Cr1—N4	82.32 (19)
O2—Cr1—N1	91.64 (19)	O1—Cr1—N2	175.3 (2)
O1—Cr1—N3	87.18 (18)	O2—Cr1—N2	85.23 (18)
O2—Cr1—N3	178.08 (19)	N1—Cr1—N2	82.6 (2)
N1—Cr1—N3	90.08 (19)	N3—Cr1—N2	95.84 (19)
O1—Cr1—N4	91.21 (18)	N4—Cr1—N2	92.8 (2)
O2—Cr1—N4	96.05 (19)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H18···O6^{i}	0.90	2.09	2.980 (6)	169
N1—H19···O3^{ii}	0.90	1.99	2.884 (6)	169
N2—H12···O7^{iii}	0.90	2.14	2.988 (7)	156
N2—H13···O4^{iv}	0.90	2.27	3.114 (6)	157
N2—H13···S2^{iv}	0.90	2.98	3.865 (5)	168
N3—H10···O4^{i}	0.90	2.08	2.970 (6)	169
N3—H11···O5^{iv}	0.90	2.09	2.983 (6)	171
N3—H11···S2^{iv}	0.90	2.98	3.821 (5)	155
N4—H4···O5^{v}	0.90	2.09	2.912 (6)	151
O7—H20···O8^{vi}	0.85	1.92	2.758 (8)	169.8
O7—H21···O6^{iv}	0.85	2.07	2.904 (7)	165.1
O7—H21···S2^{iv}	0.85	2.93	3.639 (6)	142.0
O8—H22···O3^{vii}	0.85	2.03	2.842 (7)	158.9
O8—H22···O2^{vii}	0.85	2.58	3.301 (7)	143.1

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y+1, -z$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x+1, y, z$; (v) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (vi) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (vii) $x, y, z+1$.

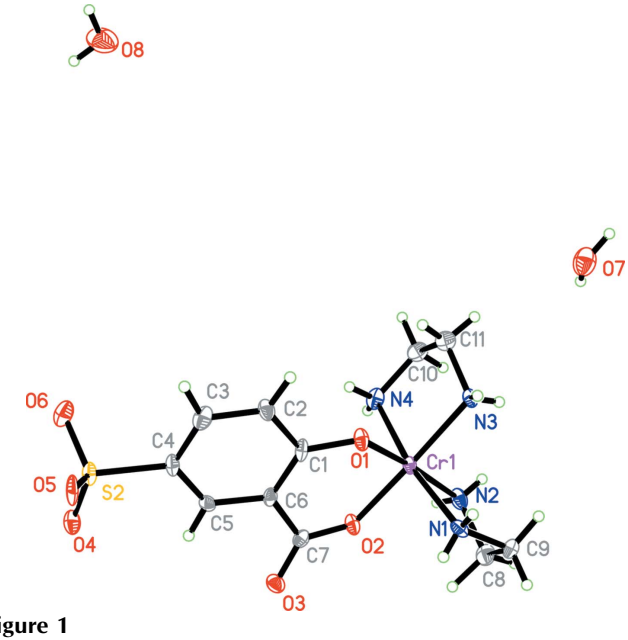


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

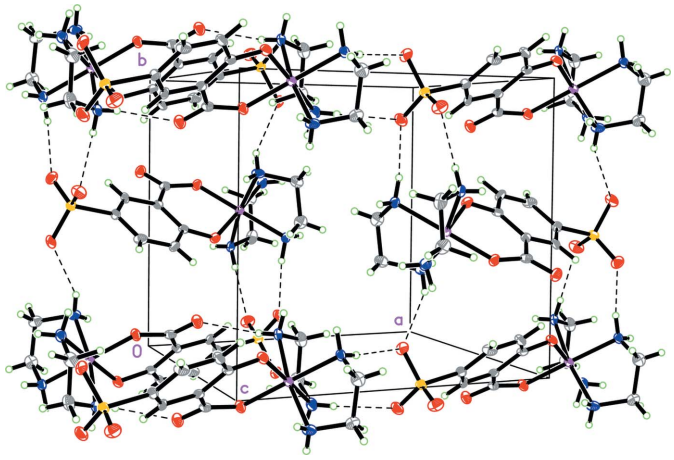


Figure 2
The crystal structure of (I). Dashed lines indicate hydrogen bonds.

H atoms attached to O atoms (water) were located in a difference Fourier map and their geometry idealized, with O—H = 0.85 Å and U_{iso}(H) = 1.2U_{eq}(O); they were then treated as riding. The other H atoms were also treated as riding atoms, with C—H (CH_2) = 0.97 Å, C—H (CH) = 0.93 Å and N—H (NH_2) = 0.90 Å, and with U_{iso}(H) = 1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2000); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

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