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Caesium bis[*N*-(carboxymethyl)iminodiacetato]chromate(III) dihydrate

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Key indicators

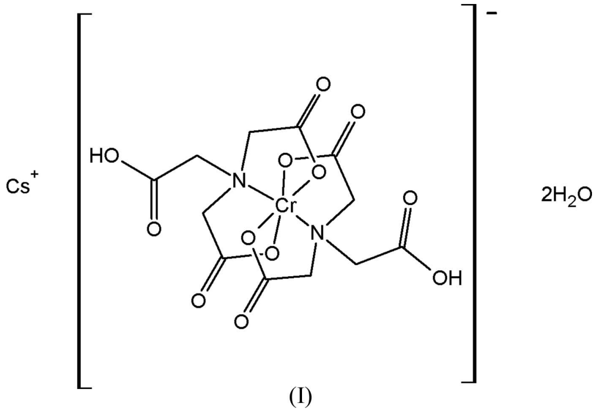
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.090
Data-to-parameter ratio = 16.4

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

The title compound, $Cs[Cr(C_6H_7NO_6)_2]\cdot 2H_2O$, behaves like a three-dimensional coordination polymer due to a network of hydrogen-bonding interactions and coordination of the caesium cation by eight O atoms. The Cs—O contact distances vary between 2.955 (3) and 3.322 (2) Å.

Comment

Nitrilotriacetic acid (H_3nta) is a well known aminopolycarboxylic ligand which normally bonds in a tetradentate manner to metal ions (Bocarsley *et al.*, 1990; Visser *et al.*, 2003; Okamoto *et al.*, 1992; White *et al.*, 1984). Our literature search (Cambridge Structural Database, Version 5.27; Allen, 2002) revealed only fifteen structures of chromium(III) complexes with nta, eight of which are di-μ-hydroxo bridged complexes (Visser, 2006; Novitchi *et al.*, 2005; Choi *et al.*, 2003; Visser *et al.*, 1999). This is the first report of a bis(nta)–chromium(III) structure in which ntaH^{2-} acts as a tridentate ligand.



(I)

The asymmetric unit of the title compound contains two crystallographically independent Hnta ligands. The Cr atom is octahedrally surrounded by six donor atoms (O2, O4, N1, O8, O9 and N2) from two different tridentate Hnta ligands (Fig. 1). All the bond distances fall within the expected ranges for this type of complex (Green *et al.*, 1990; Visser, 2007; Novitchi *et al.*, 2005). The octahedral geometry around Cr is slightly distorted, with N—Cr—O bond angles varying between 82.86 (8) and 97.68 (8)°.

Each caesium cation interacts with eight O atoms, two from the water molecules and six from the carboxylate groups of four different complex anions, serving as a link between the anions and generating a three-dimensional polymeric network (Fig. 2). The Cs—O distances vary between 2.955 (3) and 3.322 (2) Å. Further links between the anions are provided by an extensive network of hydrogen bonds (Table 1).

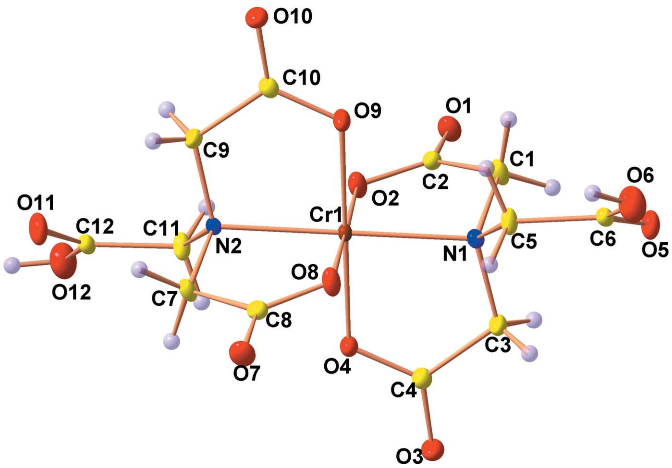


Figure 1
The structure of the complex anion in (I). Displacement ellipsoids are drawn at the 50% probability level.

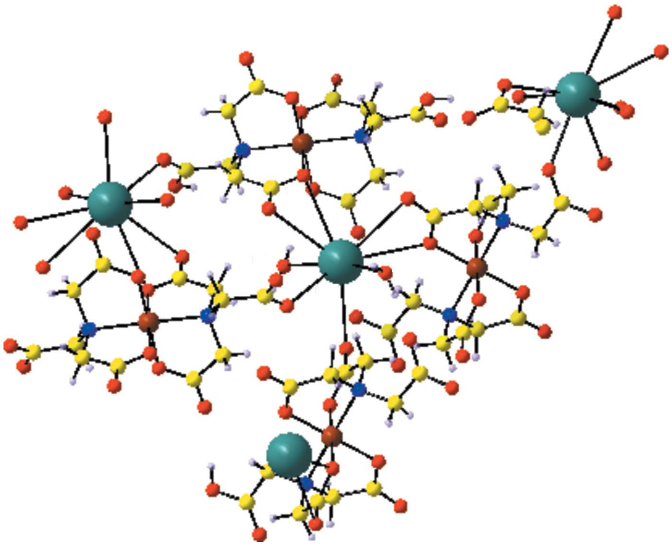


Figure 2
Coordination environment of the Cs$^+$ cation in the three-dimensional network of (I).

The deviation from planarity of the chelate ring formed by the arm of an Hnta ligand and the Cr atom is significant. This is best demonstrated by the distances of the N and Cr atoms from the CCOO planes (Table 2).

Experimental

$KCr(SO_4)_2 \cdot 12H_2O$ (5.252 g) was dissolved in H_2O (40 ml). Concentrated NH_3 was added dropwise to this solution to precipitate chromium(III) hydroxide. The precipitate was added to an aqueous solution (40 ml) containing H_3nta (4 g) and heated in a water bath until almost dry. Hot water (30 ml) was added to the reaction solution, after which it was filtered. Orange plate-like crystals precipitated after 2 d. The title compound, (I), was obtained by recrystallization from water with excess CsCl (yield 3.60 g, 78%). IR (cm^{-1}): ν(COO$^-$) 1621, ν(C=O) 1683. UV-Vis (H_2O): λ_{max} 530, 389 nm.

Crystal data

$Cs[Cr(C_6H_7NO_6)_2] \cdot 2H_2O$
$M_r = 599.18$
Monoclinic, $P2_1/n$
$a = 9.118\ (5)$ Å
$b = 12.013\ (5)$ Å
$c = 17.319\ (5)$ Å
$\beta = 94.837\ (5)^\circ$
$V = 1890.3\ (14)$ Å^3
$Z = 4$
$D_x = 2.105$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.56$ mm^{-1}
$T = 100\ (2)$ K
Plate, orange
0.2 × 0.19 × 0.07 mm

Data collection

Bruker X8 APEXII diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.628$, $T_{max} = 0.841$
33031 measured reflections
4686 independent reflections
4423 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.4^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.091$
$S = 1.30$
4686 reflections
285 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 1.3787P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.61$ e Å^{-3}
$\Delta\rho_{min} = -1.64$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O13—H13$A\cdots$O8	0.90 (2)	2.26 (4)	3.028 (3)	143 (5)
O13—H13$A\cdots$O7	0.90 (2)	2.57 (5)	3.170 (4)	124 (4)
C11—H11$A\cdots$O4	0.97	2.58	3.154 (3)	118
C11—H11$B\cdots$O2	0.97	2.44	3.011 (3)	118
C1—H1$B\cdots$O5	0.97	2.51	2.923 (3)	105
C5—H5$A\cdots$O8	0.97	2.54	3.040 (3)	112
C5—H5$B\cdots$O9	0.97	2.56	3.157 (3)	120

Table 2
Distances of Cr and N atoms from the CCOO planes (Å).

CCOO Plane	Cr distance	N distance
C1C2O1O2	0.0102 (4)	−0.526 (2)
C3C4O3O4	0.0995 (4)	−0.054 (2)
C7C8O7O8	−0.3158 (4)	0.235 (2)
C9C10O9O10	−0.0442 (4)	−0.175 (2)

H atoms of the Hnta ligands were positioned geometrically and refined as riding atoms, with C—H = 0.97 Å, O—H = 0.82 Å and $U_{iso}(H) = 1.2U_{eq}(C,O)$. H atoms of the water molecules were located in a difference Fourier map and their positional parameters were refined with $U_{iso}(H) = 1.2U_{eq}(O)$. The highest residual electron density was found 1.11 Å from atom O12 and the deepest hole 0.49 Å from O6.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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