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Structure of Diaqua[*N*-(*o*-carboxyphenyl)iminodiacetato]chromium(III) Trihydrate

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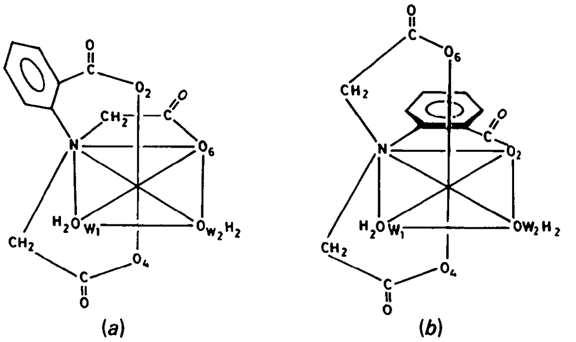
Abstract. $[Cr(C_{11}H_8NO_6)(H_2O)_2].3H_2O$, $M_r = 392\cdot26$, orthorhombic, *Pbca*, $a = 14\cdot2206\,(7)$, $b = 17\cdot9251\,(7)$, $c = 12\cdot3309\,(8)$ Å, $V = 3143\cdot22$ Å^3, $Z = 8$, $D_x = 1\cdot657$, D_m(by flotation) $= 1\cdot655$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1\cdot5406$ Å, Ni filter, $\mu = 68\cdot45$ cm^{-1}, $F(000) = 1600$, $T = 295$ K, $R = 0\cdot0479$, $wR = 0\cdot0513$ for 1409 observed reflections. The two water O atoms, the N atom and the three carboxyl O atoms of the quadridentate amino polycarboxylate ligand [*N*-(*o*-carboxyphenyl)-iminodiacetic acid (cpida)] give distorted octahedral coordination around Cr. The Cr—O distances for the coordinated water molecules are nearly equal. The carboxyl O(2)—Cr distance is shorter than the other two glycinato O—Cr distances. The phenyl ring makes a dihedral angle of 109·5 (4)° with the equatorial coordination plane. The structure is stabilized by a network of hydrogen bonds involving the water molecules.

Introduction. Linear quadridentate amino polycarboxylic acids can wrap around metal ions in octahedral coordination in a variety of ways resulting in the formation of α-*cis*, β-*cis* and β–β *trans* configurations. In contrast to the extensive stereochemical studies involving linear amino polycarboxylic metal complexes, little study has been undertaken of complexes with tripod ligands. The amino polycarboxylate ligands have donor groups similar to the more common amino acid residues and hence the studies of these complexes are of significance in comparison to amino acid complexes. The title complex can have two possible structures, Fig. 1. The structure determination was undertaken to establish the details of its coordination.

Experimental. The complex was synthesized by the literature method (Tomita, Kyuno & Tsuchia, 1969):

$$[CrCl_3.6H_2O]_{aq} + (\text{cpida}) \xrightarrow{\Delta} [Cr(\text{cpida})(H_2O)_2].3H_2O.$$

Recrystallization from water gave purple crystals. Intensity data (crystal dimensions 0·08 × 0·16 × 0·6 mm) were collected on an Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu *K*α radiation, ω–2θ scan.



(*a*) (*b*)

Fig. 1. Two possible structures.

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Table 1. *Final atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cr	−161·7 (6)	3160·6 (4)	4869·5 (6)	19 (1)
N	599 (3)	2267 (2)	4242 (3)	17 (4)
C(1)	−804 (3)	1799 (3)	5966 (4)	22 (5)
C(2)	−583 (3)	1352 (3)	4973 (4)	21 (5)
C(3)	−1049 (4)	682 (3)	4859 (4)	30 (5)
C(4)	−884 (4)	209 (3)	4008 (4)	30 (5)
C(5)	−235 (4)	390 (3)	3211 (5)	31 (5)
C(6)	242 (4)	1073 (3)	3297 (4)	29 (5)
C(7)	70 (3)	1555 (2)	4183 (4)	21 (5)
C(8)	886 (4)	2540 (3)	3126 (4)	26 (5)
C(9)	954 (4)	3385 (3)	3075 (4)	23 (5)
C(10)	1440 (4)	2157 (3)	4969 (4)	23 (5)
C(11)	1652 (3)	2854 (4)	5608 (4)	19 (5)
O(1)	−1082 (3)	1474 (2)	6793 (3)	36 (4)
O(2)	−699 (2)	2515 (2)	5958 (3)	26 (3)
O(3)	1427 (3)	3659 (2)	2356 (3)	38 (4)
O(4)	469 (2)	3759 (2)	3768 (3)	26 (3)
O(5)	2435 (2)	2946 (2)	6034 (3)	30 (3)
O(6)	973 (2)	3316 (2)	5749 (3)	22 (3)
O*W*(1)	−1193 (3)	3008 (3)	3825 (3)	31 (4)
O*W*(2)	−774 (3)	4015 (2)	5581 (3)	31 (4)
O*W*(3)	−1530 (4)	5100 (3)	4516 (5)	60 (6)
O*W*(4)	2761 (3)	1184 (3)	3054 (4)	38 (5)
O*W*(5)	2192 (3)	−262 (2)	2548 (4)	42 (4)

Table 2. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°) *with e.s.d.'s in parentheses*

N—Cr	2·082 (4)	C(3)—C(2)	1·379 (7)
O(2)—Cr	1·930 (3)	C(7)—C(2)	1·394 (7)
O(4)—Cr	1·949 (3)	C(4)—C(3)	1·369 (7)
O(6)—Cr	1·963 (3)	C(5)—C(4)	1·387 (8)
O*W*(1)—Cr	1·970 (4)	C(6)—C(5)	1·404 (7)
O*W*(2)—Cr	1·968 (4)	C(7)—C(6)	1·413 (7)
C(7)—N	1·484 (6)	C(9)—C(8)	1·518 (7)
C(8)—N	1·517 (6)	O(3)—C(9)	1·217 (6)
C(10)—N	1·507 (6)	O(4)—C(9)	1·286 (6)
C(2)—C(1)	1·497 (7)	C(11)—C(10)	1·509 (7)
O(1)—C(1)	1·239 (6)	O(5)—C(11)	1·241 (6)
O(2)—C(1)	1·292 (6)	O(6)—C(11)	1·284 (6)
O(2)—Cr—N	90·1 (1)	C(4)—C(3)—C(2)	122·4 (5)
O(4)—Cr—O(2)	175·3 (1)	C(5)—C(4)—C(3)	120·8 (5)
O(6)—Cr—N	83·5 (1)	C(6)—C(5)—C(4)	118·2 (5)
O*W*(1)—Cr—N	92·1 (2)	C(7)—C(6)—C(5)	120·5 (5)
O*W*(1)—Cr—O(6)	172·7 (2)	C(2)—C(7)—N	121·8 (4)
O*W*(2)—Cr—N	173·8 (2)	C(6)—C(7)—N	118·4 (4)
C(7)—N—Cr	114·6 (3)	C(6)—C(7)—C(2)	119·8 (4)
C(8)—N—Cr	103·2 (3)	C(9)—C(8)—N	112·2 (4)
C(10)—N—Cr	107·0 (3)	O(3)—C(9)—C(8)	117·9 (4)
O(1)—C(1)—C(2)	119·3 (4)	O(4)—C(9)—C(8)	117·3 (4)
O(2)—C(1)—C(2)	120·0 (4)	O(4)—C(9)—O(3)	124·7 (4)
O(2)—C(1)—O(1)	120·7 (4)	C(11)—C(10)—N	111·1 (4)
C(3)—C(2)—C(1)	116·7 (4)	O(5)—C(11)—C(10)	120·7 (4)
C(7)—C(2)—C(1)	124·9 (4)	O(6)—C(11)—C(10)	117·0 (4)
C(7)—C(2)—C(3)	118·4 (4)	O(6)—C(11)—O(5)	122·2 (4)

C(8)—N—C(7)—C(2)	−152·8 (4)
C(10)—N—C(7)—C(2)	83·6 (5)
C(10)—N—C(8)—C(9)	−87·7 (4)
O(2)—C(1)—C(2)—C(3)	−153·9 (4)
O(2)—C(1)—C(2)—C(7)	27·7 (7)
C(1)—C(2)—C(7)—N	−2·0 (7)
N—C(8)—C(9)—O(4)	−24·1 (6)
N—C(10)—C(11)—O(6)	22·9 (6)

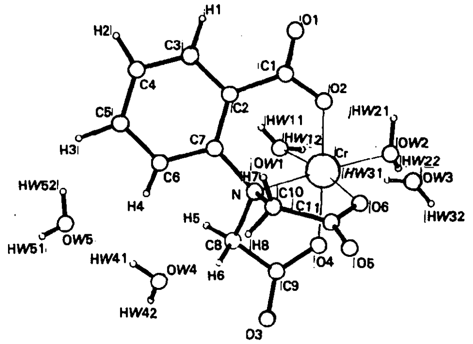

Fig. 2. General view of the molecule.

Lattice parameters obtained by photographs were refined by least-squares analysis using 25 reflections $23 < \theta < 55°$. Intensities of 3 standard reflections recorded for every 6000 s showed no significant changes. Range $0 < \theta < 55°$ ($h_{max} = 15$, $k_{max} = 19$, $l_{max} = 13$, resolution $d = 0.94$ Å). 2265 unique reflections were collected of which 700 reflections had $|F_o| < 3\sigma(|F_o|)$. Lp corrections, absorption corrections based on ψ scans of 5 strong reflections (North, Phillips & Mathews, 1968; *Structure Determination Package* 1982). Maximum and minimum correction factors 0·999 and 0·795. Structure solution by Patterson and Fourier methods. H atoms were located by difference Fourier maps. Structure refinement by least squares based on F values using anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms. Number of parameters refined: 289, including H atoms. In the final calculation reflections with $|F_o| < 5\sigma(|F_o|)$ and those for which $F_o < F_c$ with $(F_o - F_c)/\sigma(|F_o|) > 4.0$ were omitted. $(\Delta/\sigma)_{max} = 0.592$ and $\Delta\rho$ in the final difference Fourier map −0·5939 to 0·4569 e Å^{-3}. For 1409 reflections $R = 0.0479$ and $wR = 0.0513$ (Stout & Jensen, 1968), where $w = k/[\sigma^2(F_o) + g(F_o)^2]$ with $k = 1.0000$ and $g = 0.0086$. *SHELX76* program (Sheldrick, 1976) was used for refinement and NORSK DATA 500 computer for all the calculations. Atomic scattering factors those of *SHELX76*, for Cr atom from *International Tables for X-ray Crystallography* (1974).

Discussion. Final positional parameters with equivalent isotropic temperature factors are given in Table 1.* Important bond lengths, bond angles and torsion angles are given in Table 2. The *PLUTO* diagram (Motherwell & Clegg, 1978) of the molecule is given in Fig. 2. It is evident that the structure is the isomer shown in Fig. 1(*a*). The Cr atom is six coordinated with

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, hydrogen-bond distances and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51325 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

a distorted octahedral geometry. The Cr—N distance is 2·082 (4) Å which is greater than Cr—O(*W*1) and Cr—O(*W*2) [1·970 (4), 1·968 (4) Å]. This is explained by the fact that the N atom is attached to an electron-withdrawing group (phenyl ring with a carboxyl substituent) which causes the depletion of electron density at the N atom resulting in a weaker Cr—N bond. The Cr—N distance is nearly equal to that of a similar compound (Swaminathan, Sinha, Chatterjee, Patel & Padmanabhan, 1988). Cr—O(2) of the carboxylate [1·929 (3) Å] is shorter than the Cr—O(6) and Cr—O(4) distances of the glycinato rings [1·963 (3), 1·949 (3) Å]. The deviations from the octahedral angles at the Cr atom are within 6·5°. The axial oxygens make an angle O(2)—Cr—O(4) of 175·3 (1)°. The N—C(7) distance (1·484 Å) is less than the N—C(8) and N—C(10) distances of the glycinato rings [1·517 (6), 1·507 (6) Å]. This is due to part of the (cpida) ligand undergoing resonance (Martell & Calvin, 1956; Naveen Chandra, 1984). The phenyl ring is oriented at an angle of 109·5 (4)° to the equatorial coordination plane. The ring N,C(7),C(2),C(1),O(2),-Cr should be planar (Chatterjee & Phadke, 1987) but owing to the chelation and strain the planarity is disturbed. This plane is at an angle of 18·9 (3)° with respect to the phenyl ring. The corresponding C—C and C—O distances of the two glycinato rings are equal. C(11)=O(5) [1·241 (6) Å] is longer than C(9)=O(3) [1·217 (6) Å]. This is because O(5) forms two hydrogen bonds whereas O(3) forms only one. It has previously been observed (Weakliem & Hoard, 1959; Okazaki, Tomioka & Yoneda, 1983) that the *G* ring (the glycinato ring in plane with the equatorial plane) is more strained than the *R* ring [the glycinato ring bonding through O(4) out of the equatorial plane]. However, in the present work no marked difference is observed. The structure is stabilized by a network of hydrogen bonds involving the water molecules.

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Structure of Bis[μ-iminodiacetato(1−)-μ-*O,O′,O″*]-bis[pentaaquabarium(II)] Bis[iminodiacetato(2−)-*N,O,O′*]cuprate(II)

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Abstract. $[Ba_2\{H_2N(CH_2COO)_2\}_2(H_2O)_{10}][Cu\{HN(CH_2COO)_2\}_2]$, $M_r = 1044{\cdot}72$, triclinic, $P\overline{1}$, $a = 9{\cdot}050\ (4)$, $b = 9{\cdot}437\ (5)$, $c = 11{\cdot}468\ (5)$ Å, $\alpha = 108{\cdot}05\ (3)$, $\beta = 93{\cdot}68\ (4)$, $\gamma = 112{\cdot}55\ (4)°$, $V = 841{\cdot}14$ Å^3, $Z = 1$, $D_m = 2{\cdot}09$, $D_x = 2{\cdot}06$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3{\cdot}09$ mm^{-1}, $F(000) =$

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