

Sodium *trans*-Bis(*N*-isopropyliminodiacetato)chromate(III) Dihydrate

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**Abstract.**  $\text{Na}[\text{Cr}(\text{C}_7\text{H}_{11}\text{NO}_4)_2] \cdot 2\text{H}_2\text{O}$ ,  $\text{Na}^+ \cdot \text{C}_{14}\text{H}_{22}\text{Cr} \cdot \text{N}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$ , triclinic,  $P\bar{1}$ ,  $a = 9.779$  (1),  $b = 7.922$  (1),  $c = 6.968$  (1) Å,  $\alpha = 108.56$  (1),  $\beta = 95.76$  (1),  $\gamma = 107.40$  (1)°,  $Z = 1$ ,  $D_m = 1.614$ ,  $D_x = 1.593$  Mg m<sup>-3</sup>,  $M_r = 457.4$ ,  $\mu(\text{Mo } K\alpha) = 0.71$  mm<sup>-1</sup>,  $R = 0.046$ . The point symmetry of the complex anion is  $\bar{1}$ . The coordination of the Cr atom is distorted octahedral with the two N atoms in a *trans* configuration. The one independent water molecule forms weak hydrogen bonds. Preliminary results have been reported elsewhere [Wunderlich & Mootz (1977). Fourth European Crystallographic Meeting, Oxford, England. Abstract PL.64].

**Introduction.** The synthesis and characterization of potassium *trans*-bis(*N*-alkyliminodiacetato)chromates(III) have been reported by Wernicke, Schmidtke & Hoggard (1977). Red crystals of the title compound with  $\text{Na}^+$  as the cation were prepared accordingly and grown from aqueous solution. The space group is  $P1$  or  $P\bar{1}$ . The latter was confirmed by the structure determination. The intensities of all symmetry-independent reflections up to  $2\theta = 55^\circ$  were determined with an automatic diffractometer (Syntex P2<sub>1</sub>) and a variable  $\theta:2\theta$  scan (Mo  $K\alpha$  radiation, crystal monochromator). 1793 of the 2142 measured intensities were classified as observed ( $F > 3\sigma_F$ ) and were used for the structure determination. The phase problem was solved by the Patterson function. The Cr atom at 0,0,0 and the Na atom at  $0, \frac{1}{2}, \frac{1}{2}$  cause the mean  $F_o$  for  $k + l = \text{even}$  to be about 40% greater than that for  $k + l = \text{odd}$ . All H atoms were located in difference syntheses and included with isotropic temperature factors in the final refinement (186 parameters), converging at  $R = 0.046$  (0.061) and  $R_w = 0.051$  (0.053) for the observed (all) reflections. Scattering factors were taken from Cromer & Waber (1974). Weights were derived from counting statistics by  $1/w = \sigma_F^2 + 0.0004F^2$ . The final positional parameters are listed in Tables 1 and 2.\* All calculations were carried

Table 1. Positional parameters ( $\times 10^4$ ) for the non-hydrogen atoms with e.s.d.'s in parentheses as resulting from the least-squares refinement

	$x$	$y$	$z$	$B_{\text{eq.}}$
Na	0	5000	5000	2.43
Cr	0	0	0	1.09
O(1)	-1015 (2)	-1972 (3)	1005 (3)	1.96
O(2)	574 (2)	-1791 (3)	-2165 (3)	1.86
O(5)	-719 (3)	-4014 (3)	2420 (4)	2.71
O(6)	2522 (3)	-2317 (3)	-3232 (3)	2.73
N	1907 (3)	17 (3)	1753 (4)	1.41
C(1)	-218 (3)	-2675 (4)	1860 (4)	1.72
C(2)	1429 (3)	-1799 (4)	2158 (5)	1.85
C(3)	2890 (3)	-128 (4)	241 (4)	1.82
C(4)	1966 (3)	-1511 (4)	-1887 (4)	1.77
C(5)	2616 (3)	1700 (4)	3786 (4)	1.95
C(6)	3994 (4)	1600 (6)	4924 (6)	2.99
C(7)	2960 (4)	3587 (5)	3492 (6)	2.55
OW	6039 (4)	4110 (6)	506 (7)	5.30

Equivalent isotropic temperature factors  $B_{\text{eq.}}$  (Å<sup>2</sup>) have been calculated by  $B_{\text{eq.}} = \frac{1}{3}(B_{11}a^{*2}a^2 + B_{12}a^*b^*ab \cos \gamma + \dots)$ .

Table 2. Positional parameters ( $\times 10^3$ ) and temperature factors (Å<sup>2</sup>) for the H atoms with e.s.d.'s in parentheses

The H atoms are bonded to the O and C atoms with the identical first digit of their numbering.

	$x$	$y$	$z$	$B$
H(21)	193 (4)	-164 (5)	349 (5)	2 (1)
H(22)	175 (4)	-271 (5)	130 (6)	3 (1)
H(31)	368 (3)	-54 (4)	66 (5)	1 (1)
H(32)	341 (4)	104 (5)	13 (5)	2 (1)
H(5)	182 (4)	149 (5)	468 (5)	3 (1)
H(61)	384 (4)	32 (5)	515 (6)	3 (1)
H(62)	482 (5)	189 (6)	411 (7)	5 (1)
H(63)	434 (5)	267 (6)	623 (7)	5 (1)
H(71)	209 (4)	372 (6)	265 (6)	4 (1)
H(72)	331 (5)	459 (6)	491 (7)	5 (1)
H(73)	377 (5)	385 (6)	265 (7)	5 (1)
HW(1)	581 (11)	464 (15)	-22 (17)	17 (4)
HW(2)	632 (10)	380 (12)	125 (13)	3 (2)
HW(3)	709 (12)	510 (14)	99 (14)	5 (2)

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34977 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

out with the program system EXTL (Syntex) on an Eclipse computer (Data General) with 64 K byte memory.

**Discussion.** This structure determination is part of a series on octahedral bis(iminodiacetato)chromium(III) complexes with different substituents at the N atoms. An H atom as the smallest substituent in K[Cr{HN-(CH<sub>2</sub>COO)<sub>2</sub>}<sub>2</sub>].3H<sub>2</sub>O yielded a *cis* configuration for the N atoms (Mootz & Wunderlich, 1980), while from model considerations this seems less likely for bulky substituents.

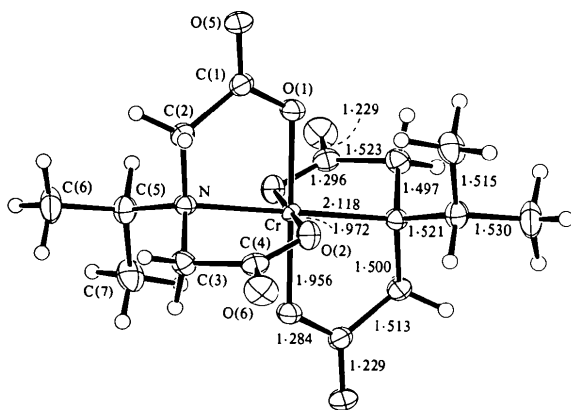


Fig. 1. The anion of the complex Na[Cr{(i-C<sub>7</sub>H<sub>7</sub>)N(CH<sub>2</sub>COO)<sub>2</sub>}<sub>2</sub>].2H<sub>2</sub>O with bond lengths (Å). The e.s.d.'s are 0.002 Å for Cr—O and Cr—N and 0.004–0.005 Å otherwise. The Cr atom is located at a center of symmetry. The heavy atoms are represented by thermal ellipsoids of 50% probability (ORTEP II, Johnson, 1976). The isotropic temperature factor of the H atoms is set to  $B = 0.67 \text{ Å}^2$ .

Table 3. Bond angles and non-bonding distances in Na[Cr{(i-C<sub>7</sub>H<sub>7</sub>)N(CH<sub>2</sub>COO)<sub>2</sub>}<sub>2</sub>].2H<sub>2</sub>O

Cr and Na atoms are located at centers of symmetry. Symmetry code: (i)  $-x, -y, -z$ ; (ii)  $-x, -y, 1 - z$ .

(a) Angles (°) in the complex anion (the e.s.d.'s are 0.1° at Cr, 0.2° at O and N, and 0.3° at C atoms)

O(1)—Cr—O(2)	92.7	C(2)—N—C(3)	109.1
O(1)—Cr—N	83.7	C(2)—N—C(5)	110.0
O(2)—Cr—N	79.7	C(3)—N—C(5)	113.3
Cr—O(1)—C(1)	116.9	N—C(3)—C(4)	108.6
O(1)—C(1)—C(2)	117.3	C(3)—C(4)—O(2)	115.9
O(1)—C(1)—O(5)	123.7	C(3)—C(4)—O(6)	121.3
O(5)—C(1)—C(2)	119.0	O(6)—C(4)—O(2)	122.8
C(1)—C(2)—N	113.5	C(4)—O(2)—Cr	115.2
Cr—N—C(2)	105.0	N—C(5)—C(6)	112.3
Cr—N—C(3)	102.6	N—C(5)—C(7)	112.2
Cr—N—C(5)	116.2	C(6)—C(5)—C(7)	110.3

(b) Distances (Å) involving the Na<sup>+</sup> ion (the e.s.d.'s are 0.002–0.003 Å)

Na—O(2 <sup>i</sup> )	2.531	Na—O(6 <sup>i</sup> )	2.589
Na—O(5 <sup>ii</sup> )	2.305		

(c) Angles (°) at the Na<sup>+</sup> ion (the e.s.d.'s are 0.1°)

O(2 <sup>i</sup> )—Na—O(5 <sup>ii</sup> )	93.5	O(5 <sup>ii</sup> )—Na—O(6 <sup>i</sup> )	99.4
O(2 <sup>i</sup> )—Na—O(6 <sup>i</sup> )	51.3		

(d) Angles (°) of the water molecule (the e.s.d.'s are 10°)

HW(1)—OW—HW(2)	170	HW(2)—OW—HW(3)	81
HW(1)—OW—HW(3)	90		

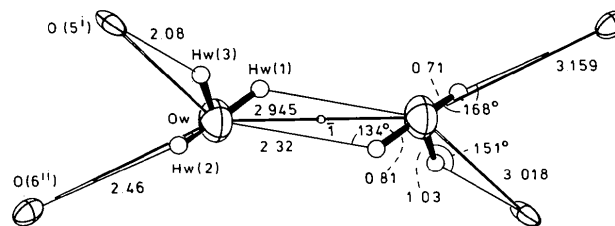


Fig. 2. Description of the water structure with weak hydrogen bonds and proton disorder. The thermal ellipsoids are at a level of 25% probability. The temperature factor of the H atoms is set to  $B = 1.0 \text{ Å}^2$ . The e.s.d.'s are: 0.007 Å (O...O), 0.09–0.12 Å (O—H, O...H) and 10° (O—H...O). Symmetry code: (i)  $1 + x, 1 + y, z$ ; (ii)  $1 - x, -y, -z$ .

In Fig. 1 the anion is displayed with the main bond lengths; bond angles are listed in Table 3. The point symmetry of the anion is  $\bar{1}$  and the structure is in a *trans* configuration. Ring constraints and steric hindrances influence the geometry of the molecule. The octahedral coordination of the Cr atom is distorted up to 10.3° from ideal angles with Cr—O 1.956, 1.972 Å and Cr—N 2.118 Å. The two symmetry-independent five-membered rings at the Cr atom are puckered up to 0.1 Å [involving O(1)] and 0.3 Å [involving O(2)]. The angles at the N atom deviate up to 7° from tetrahedral coordination. The geometry of the H atoms is in the expected range.

The Na<sup>+</sup> ion is surrounded by four complex anions with six short (<2.6 Å) Na...O distances in a considerably distorted octahedral coordination. O(2) and O(6) of the same molecule coordinate one Na atom. The water molecule forms weak O—H...O hydrogen bonds (see Table 3 and Fig. 2) to two complex anions and to a symmetry-related water molecule. This results in a water structure which remains questionable. HW(2) and HW(3) were refined with occupancy factors of 0.5, but no split atom position of HW(1) could be obtained, although disorder of this atom is indicated by its high  $B$  value.

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