O(1)—Sn(1)—C(3)	80.5 (1)	C(12)—C(11)—C(16)	120.4 (4)
O(1)—Sn(1)—C(2)	85.5 (1)	C(11)—C(12)—C(13)	118.8 (4)
O(1)—Sn(1)—C(1)	89.2 (2)	C(12)—C(13)—C(14)	119.8 (5)
F(1)—Sn(1)—C(3)	93.6 (1)	C(13)—C(14)—C(15)	121.2 (5)
F(1)—Sn(1)—C(2)	94.3 (1)	C(14)—C(15)—C(16)	120.1 (5)
F(1)—Sn(1)—C(1)	97.3 (2)	C(11)—C(16)—C(15)	119.6 (4)
F(1)—Sn(1)—O(1)	172.66 (9)	P(1)—C(21)—C(26)	120.7 (3)
C(11)—P(1)—C(21)	106.2 (2)	P(1)—C(21)—C(22)	119.6 (3)
C(4)—P(1)—C(21)	108.7 (2)	C(22)—C(21)—C(26)	119.6 (3)
C(4)—P(1)—C(11)	109.6 (2)	C(21)—C(22)—C(23)	119.8 (4)
O(1)—P(1)—C(21)	112.1 (2)	C(22)—C(23)—C(24)	120.6 (4)
O(1)—P(1)—C(11)	112.0 (2)	C(23)—C(24)—C(25)	120.5 (4)
O(1)—P(1)—C(4)	108.3 (2)	C(24)—C(25)—C(26)	119.2 (4)
Sn(1)—O(1)—P(1)	109.6 (1)	C(21)—C(26)—C(25)	120.2 (4)
Sn(1)—C(3)—C(4)	113.3 (2)		

O(1)—Sn(1)—C(3)—C(4)	24.9 (2)	O(1)—P(1)—C(4)—C(3)	47.9 (3)
C(3)—Sn(1)—O(1)—P(1)	4.2 (2)	Sn(1)—C(3)—C(4)—P(1)	−45.7 (3)
C(4)—P(1)—O(1)—Sn(1)	−26.9 (2)		

The title compound was prepared by the dropwise addition of hydrogen peroxide to a stirred suspension of [2-(fluorodimethylstannyl)ethyl]diphenylphosphine in acetone until the solution became clear. Recrystallization of the crude product from methylene chloride/hexane yielded colourless crystals, m.p. 432–433 K. The yield was 42%. $ZC_{16}H_{20}ClOPSn$: C 48.4% (calculated), 48.3% (found), H 5.1% (calculated), 5.3% (found). [2-(Fluorodimethylstannyl)ethyl]diphenylphosphine was prepared by stirring [2-(chlorodimethylstannyl)ethyl]diphenylphosphine with a fivefold molar excess of potassium fluoride in a degassed mixture of water and methanol for 12 h. The white precipitate was washed with water/methanol and vacuum dried; m.p. > 473 K, yield 78%. The crystal was mounted on the tip of a glass fibre with shellac. The lattice constants were determined from a symmetry-constrained least-squares fit. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. H atoms were placed in calculated positions and a common isotropic displacement parameter refined. The weighting scheme used led to an analysis of variance which was featureless in terms of $\sin\theta$ and F. A search for higher symmetries was performed using *MISSYM* (Le Page, 1987).

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55286 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1011]

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Structure of a Dichromium(II) Complex with Bridging Betaine Ligands, $[Cr_2(O_2CCH_2NMe_3)_4(OH_2)_2][ZnCl_4]_2.2H_2O$

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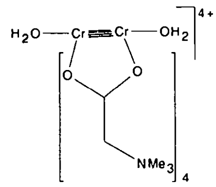
Abstract

The structure of diaqua-tetrakis(μ-*N*,*N*,*N*-trimethylglycinato-*O*,*O*′)-dichromium(II) bis[tetrachlorozincate(II)] dihydrate is described. A Cr—Cr quadruple bond of length 2.446 (2) Å is bridged by four zwitterionic betaine ligands with Cr—O bond lengths in the range 2.016 (4)–2.027 (3) Å. The axial coordination site of each Cr atom, opposite the Cr—Cr bond, is occupied by a water molecule with Cr—O 2.243 (5) Å. The cationic charge is balanced by tetrahedral $[ZnCl_4]^{2-}$ anions. The axial aqua ligand is hydrogen bonded to additional water of crystallization.

Comment

Chromium(II) acetate monohydrate has been known for about a century and a half. With its deep red colour, feeble paramagnetism and acetate-bridged binuclear structure (van Niekerk, Schoening & de Wet, 1953; Cotton, de Boer, Laprade, Pipal & Ucko, 1970, 1971), it differs markedly from the blue mononuclear chromium(II) salts. It is also the prototype of a large variety of quadruple-bonded complexes (Cotton & Walton, 1982) which contain simple alkyl or aryl carboxylates as bridging ligands and either neutral or anionic ligands in the axial positions (Ford, Larkworthy, Povey & Roberts, 1983; Larkworthy & Roberts, 1982).

In contrast, the analogous complexes in which the carboxyl groups of amino acids act as bridges remain relatively unexplored. Two types of chromium(II) complex with glycine were reported by Herzog & Kalies (1967). The first of these, $[Cr(O_2CCH_2NH_2)_2.H_2O]$, appears, from magnetic susceptibility measurements, to be a mononuclear high-spin complex. The second, originally formulated as $[Cr(O_2CCH_2NH_2)_2.2HBr.2H_2O]$, was subsequently shown, along with the chloride analogue, to contain the binuclear cation $[Cr_2(O_2CCH_2NH_3)_4Br_2]^{2+}$, with zwitterionic glycine bridging ligands (Ardon, Bino, Cohen & Felthouse, 1984). These appear to be the only reported crystal structures of dichromium(II) complexes with amino acid bridging ligands although the Cambridge Structural Database contains over a dozen such dimolybdenum(II) complexes. We report here a betaine-bridged complex (1) of similar type.



(1)

The betaine ligands are zwitterionic, each symmetrically bridging the Cr_2 unit through the carboxyl group in the same way as simple carboxylate bridges, with no noticeable perturbation from the quaternary N substituents. The Cr—Cr quadruple bond is of a length typical for this class of complex and considerably longer than the so-called 'super-short' bonds found in structures without any axially bound ligands (Cotton & Walton 1982). The binuclear cation has crystallographic inversion symmetry.

The axial H_2O ligands are bound less strongly to the metal atoms and participate in significant hydrogen bonding to the additional molecules of water of crystallization in the structure [O(3)···O(4) 2.57 (1) Å]. Contacts of length 2.37—3.37 Å between both O(3) and O(4) and some of the Cl atoms of the anions may indicate further weak hydrogen bonding of the type O—H···Cl.

The $[ZnCl_4]^{2-}$ anions are essentially undistorted tetrahedral, with bond lengths of 2.241 (2)–2.295 (2) Å and angles of 107.7 (1)–113.1 (1)°. There are only normal electrostatic interactions between the cations and anions.

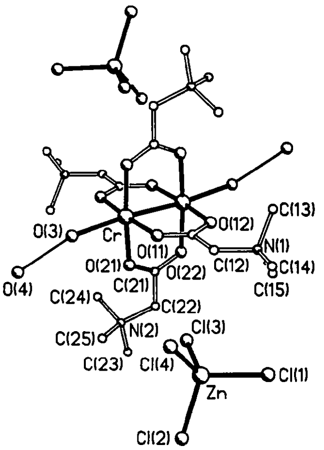


Fig. 1. The binuclear cation with two anions and two molecules of water of crystallization. H atoms are omitted; the atoms of the asymmetric unit are labelled.

Experimental

Crystal data

$[Cr_2(C_5H_{11}NO_2)_4(H_2O)_2]$-
$[ZnCl_4]_2.2H_2O$
$M_r = 1059$
Monoclinic
$P2_1/c$
$a = 11.6420\ (6)$ Å
$b = 9.3595\ (5)$ Å
$c = 21.0985\ (11)$ Å
$\beta = 104.252\ (5)°$
$V = 2228.2$ Å^3
$Z = 2$

$D_x = 1.578$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 10$-$12.5°$
$\mu = 2.09$ mm^{-1}
$T = 293$ K
Block
0.42 × 0.38 × 0.35 mm
Dark blue

Data collection

Stoe-Siemens diffractometer
ω/θ scans
Absorption correction: semi-empirical
$T_{min} = 0.386$, $T_{max} = 0.444$
4817 measured reflections
3909 independent reflections
2881 observed reflections $[F > 4\sigma(F)]$

$R_{int} = 0.0195$
$\theta_{max} = 25°$
$h = 0 \rightarrow 13$
$k = -11 \rightarrow 2$
$l = -25 \rightarrow 25$
3 standard reflections
frequency: every 60 min
intensity variation: 7%

Refinement

Refinement on F
Final $R = 0.0488$
$wR = 0.0601$
$S = 2.06$
2881 reflections
236 parameters
$w = 1/[\sigma^2(F) + 0.00029F^2]$
$(\Delta/\sigma)_{max} = 0.051$
$\Delta\rho_{max} = 0.99$ e Å^{-3}
$\Delta\rho_{min} = -0.95$ e Å^{-3}

Extinction correction: $F_c' = F_c/(1 + xF_c^2/\sin2\theta)^{0.25}$
Extinction coefficient: $x = 4(1) \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

U_{eq} = 1/3(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Cr	0.44580 (7)	0.59219 (8)	0.52175 (4)	0.0329 (3)
Zn	0.16203 (5)	0.10833 (7)	0.65861 (3)	0.0411 (2)
O(11)	0.4581 (3)	0.4615 (4)	0.5991 (2)	0.040 (1)
O(12)	0.5548 (3)	0.2912 (4)	0.5590 (2)	0.040 (1)
C(11)	0.5085 (4)	0.3419 (5)	0.6017 (2)	0.035 (2)
C(12)	0.5115 (4)	0.2607 (5)	0.6642 (2)	0.034 (2)
N(1)	0.5716 (3)	0.1190 (4)	0.6743 (2)	0.036 (1)
C(13)	0.7003 (4)	0.1310 (6)	0.6752 (3)	0.048 (2)
C(14)	0.5632 (5)	0.0670 (6)	0.7405 (3)	0.052 (2)
C(15)	0.5142 (5)	0.0143 (6)	0.6235 (3)	0.057 (2)
O(21)	0.2974 (3)	0.4931 (4)	0.4700 (2)	0.045 (1)
O(22)	0.3963 (3)	0.3220 (4)	0.4328 (2)	0.042 (1)
C(21)	0.3030 (5)	0.3837 (5)	0.4368 (2)	0.037 (2)
C(22)	0.1903 (5)	0.3160 (6)	0.3976 (3)	0.047 (2)
N(2)	0.0838 (4)	0.4115 (5)	0.3761 (2)	0.041 (2)
C(23)	−0.0113 (5)	0.3270 (8)	0.3301 (3)	0.065 (3)
C(24)	0.1136 (6)	0.5386 (7)	0.3398 (3)	0.067 (3)
C(25)	0.0376 (6)	0.4603 (10)	0.4325 (4)	0.086 (3)
O(3)	0.3314 (4)	0.7480 (4)	0.5589 (2)	0.062 (2)
Cl(1)	0.2608 (1)	−0.1022 (2)	0.6897 (1)	0.059 (1)
Cl(2)	−0.0313 (1)	0.0711 (2)	0.6500 (1)	0.072 (1)
Cl(3)	0.2004 (2)	0.1854 (2)	0.5653 (1)	0.082 (1)
Cl(4)	0.2319 (2)	0.2733 (2)	0.7362 (1)	0.099 (1)
O(4)	0.1366 (11)	0.8719 (11)	0.5014 (5)	0.343 (8)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Cr—Cr′	2.446 (2)	Cr—O(3)	2.243 (5)
Cr—O(11)	2.016 (4)	Cr—O(21)	2.027 (3)
Cr—O(12′)	2.022 (4)	Cr—O(22′)	2.019 (3)
Zn—Cl(1)	2.295 (2)	Zn—Cl(2)	2.241 (2)
Zn—Cl(3)	2.243 (2)	Zn—Cl(4)	2.252 (2)
O(11)—Cr—Cr′	86.3 (1)	O(21)—Cr—Cr′	85.7 (1)
O(3)—Cr—Cr′	174.6 (1)	Cr′—Cr—O(12′)	87.7 (1)
Cr′—Cr—O(22′)	88.0 (1)	O(11)—Cr—O(3)	92.4 (2)
O(21)—Cr—O(3)	89.1 (2)	O(3)—Cr—O(12′)	93.7 (2)
O(3)—Cr—O(22′)	97.2 (2)	O(11)—Cr—O(21)	92.3 (1)
O(11)—Cr—O(12′)	173.9 (2)	O(21)—Cr—O(12′)	88.1 (1)
O(11)—Cr—O(22′)	89.0 (1)	O(21)—Cr—O(22′)	173.5 (2)
O(12′)—Cr—O(22′)	90.0 (1)	Cl(1)—Zn—Cl(2)	108.2 (1)
Cl(1)—Zn—Cl(3)	109.5 (1)	Cl(2)—Zn—Cl(3)	113.1 (1)
Cl(1)—Zn—Cl(4)	108.3 (1)	Cl(2)—Zn—Cl(4)	110.0 (1)
Cl(3)—Zn—Cl(4)	107.7 (1)		

Symmetry operation for primed atoms: $1 - x$, $1 - y$, $1 - z$.

The complex was prepared from chromium(II) chloride, zinc amalgam, betaine and dilute HCl in a Jones reductor-column apparatus under a nitrogen atmosphere and crystals were obtained by solvent-volume reduction and cooling (60% yield, satisfactory chemical analyses). As the product is air sensitive, the crystal for X-ray study was sealed in a nitrogen-filled Lindemann capillary with traces of mother liquor.

Data collection and reduction: Stoe *DIF*4 software 1984. Program used to solve, refine and display structure: *SHELXTL* (Sheldrick, 1985). H atoms constrained [C—H 0.96 Å, H—C—H 109.5°, $U(H) = 1.2U_{eq}(C)$], no H atoms for H_2O. The scan range was from 0.51° below α_1 to 0.51° above α_2 with scan times of 14-56 s.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55210 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1005]

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Structure of 2-[(4-Methylphenylimino)methyl]-1-nitrobenzene

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Abstract

The benzylideneaniline moiety adopts a distorted planar conformation with a dihedral angle of 3.9 (5)° between the two phenyl rings. The nitro group is twisted from the phenyl ring by 27.9 (5)°.

Comment

Although benzylideneaniline adopts an energetically favoured non-planar conformation (Bürgi & Dunitz, 1970; Bernstein, Engel & Hagler, 1981), some of its derivatives with homo or hetero *para* substituents prefer a planar conformation (Bar & Bernstein, 1983). Different kinds of chemical substitutions at different positions in these derivatives result in distorted planar conformations (Fun, Teo, Teoh & Yeap, 1991). The title compound, which is extensively employed in the synthesis and characterization of adducts (Srivastava & Chauhan, 1977), is found to exhibit a slightly distorted planar conformation

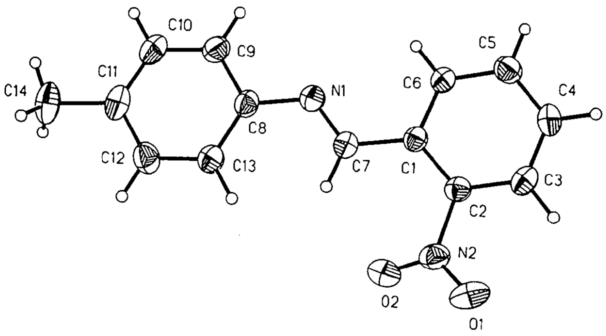

Fig. 1. A view of the molecule showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels.