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Tricarbonyl(η^6-1-tetralol)chromium

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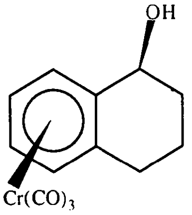
Abstract

In the structure of tricarbonyl(η^6-1,2,3,4-tetrahydro-1-naphthalenol)chromium, $[Cr(CO)_3(C_{10}H_{12}O)]$, the group of three carbonyl ligands is *syn* with respect to the hydroxy group. The asymmetric unit contains two molecules of the same enantiomer with no significant differences in terms of intramolecular bond lengths and bond angles. The $Cr(CO)_3$ tripod is complexed to the benzene ring in an eclipsed conformation. An extensive hydrogen-bonding network supports the crystal structure.

Comment

Tricarbonylchromium-complexed organic molecules receive much attention as intermediates in asymmetric syntheses (Solladie-Cavallo, 1989). In order to determine the absolute stereochemistry of the final metal-free targets, it is essential to know the spatial structure of the complexes. Tricarbonyl(η^6-1-tetralol)chromium, (I), has been prepared previously (Jaouen & Meyer, 1975; Davies & Goodfellow,1988; Schmalz, Millies, Bats & Dürner, 1992) but, to date, the *syn* configuration of the tricarbonyl tripod with respect to the hydroxy group has been established only by indirect methods. This structure determination has confirmed the previous results.



(I)

The asymmetric unit contains two molecules of the same enantiomer with no significant differences in the intramolecular bond lengths and bond angles. The $Cr(CO)_3$ group is complexed to the benzene ring (Fig. 1) in an eclipsed conformation with the carbonyl groups located near the C10, C8 and C12 (C10′, C8′ and C12′) atoms (Fig. 2); this means that the C8—C7 (C8′—C7′) bond, connecting the more electron-donating C7 (C7′) substituent, is eclipsed with respect to the C3—O3 (C3′—O3′) carbonyl group, as would be expected considering the electronic properties of this kind of compound (Muetterties, Bleeke, Wucherer & Albright, 1982). The Cr—C(arene) distances range between 2.201 (3) and 2.256 (3) Å with the highest values for the substituted atoms C8 and C9 (C8′ and C9′). The largest deviation from the least-square plane of the benzene ring is 0.016 (4) Å for atom C11′ [0.012 (3) Å for C11], while the substituent atoms C4 and C7 lie at 0.028 (3) and 0.019 (4) Å, respectively [0.018 (3) and 0.014 (3) Å for C4′ and C7′] from the plane on the opposite side with respect to the $Cr(CO)_3$ group.

There is an extensive hydrogen-bonding network within the crystal (Fig. 3); the two molecules in the asymmetric unit are linked by a hydrogen bond between H4O and O4′ [H4O···O4′ 2.08 (4), O4···O4′ 2.848 (3) Å, O4—H4O···O4′ 166 (3)°]. Another hydrogen bond links the asymmetric unit to the nearest molecules [H4O′···O4^{i} 2.07 (4), O4′···O4^{i} 2.878 (3) Å, O4′—H4O′···O4^{i} 170 (3)°; symmetry code: (i) $\frac{1}{2} - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$].

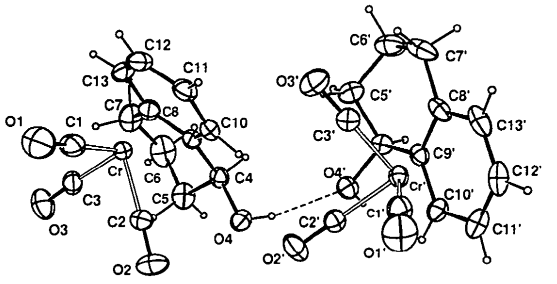


Fig. 1. Perspective view of the asymmetric unit with 30% probability ellipsoids.

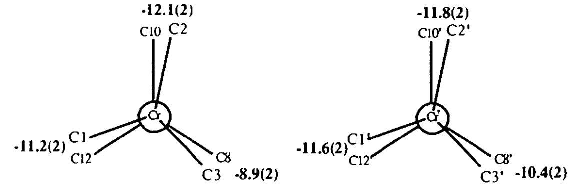


Fig. 2. Newman projections along the direction from the Cr atom to the centre of the benzene ring.

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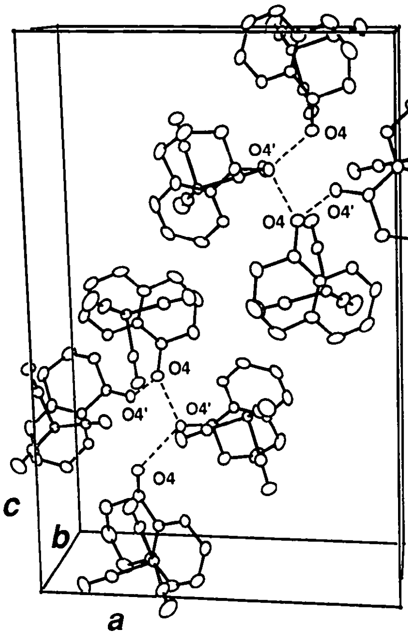

Fig. 3. Packing diagram viewed down the b axis. H atoms have been omitted.

Experimental

Crystals of the title compound suitable for X-ray analysis were obtained by recrystallization from diisopropyl ether.

Crystal data

$[Cr(CO)_3(C_{10}H_{12}O)]$
$M_r = 284.23$
Monoclinic
$P2_1/n$
$a = 14.342\,(1)$ Å
$b = 7.7990\,(8)$ Å
$c = 21.592\,(2)$ Å
$\beta = 91.60\,(1)°$
$V = 2414.2\,(4)$ Å^3
$Z = 8$
$D_x = 1.5640$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 35–40°$
$\mu = 7.85$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.6 × 0.4 × 0.4 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Matthews, 1968)
$T_{min} = 0.778$, $T_{max} = 0.997$
6468 measured reflections
4360 independent reflections
3712 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.014$
$\theta_{max} = 70°$
$h = 0 \rightarrow 17$
$k = -3 \rightarrow 9$
$l = -26 \rightarrow 26$
3 standard reflections monitored every 300 reflections frequency: 120 min intensity decay: 2.2%

Refinement

Refinement on F
$R = 0.034$
$wR = 0.034$
$S = 1.070$
3712 reflections
421 parameters
All H-atom parameters refined
Unit weights applied
$(\Delta/\sigma)_{max} = 0.02$

$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* [1974, Vol. IV, Tables 2.2A, 2.3.1 (Cr, O, C), Table 2.2C (H)]

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cr	0.19841 (3)	1.28229 (6)	0.46006 (2)	0.0373 (1)
O1	0.1318 (2)	1.6199 (4)	0.5060 (1)	0.1004 (10)
O2	0.2431 (2)	1.4567 (3)	0.3419 (1)	0.0661 (7)
O3	0.3931 (2)	1.3665 (4)	0.5013 (1)	0.0803 (9)
O4	0.2615 (1)	1.0156 (3)	0.32074 (9)	0.0505 (6)
C1	0.1576 (2)	1.4872 (4)	0.4885 (2)	0.0601 (10)
C2	0.2251 (2)	1.3871 (4)	0.3872 (1)	0.0421 (8)
C3	0.3162 (2)	1.3350 (4)	0.4864 (1)	0.0507 (9)
C4	0.2690 (2)	0.9298 (4)	0.3788 (1)	0.0440 (9)
C5	0.3693 (2)	0.9274 (5)	0.4028 (2)	0.0622 (12)
C6	0.3731 (3)	0.8359 (5)	0.4652 (2)	0.0818 (13)
C7	0.3192 (3)	0.9305 (5)	0.5134 (2)	0.0734 (13)
C8	0.2296 (2)	1.0099 (4)	0.4890 (1)	0.0508 (9)
C9	0.2059 (2)	1.0116 (4)	0.4250 (1)	0.0396 (8)
C10	0.1208 (2)	1.0877 (4)	0.4057 (1)	0.0447 (8)
C11	0.0595 (2)	1.1590 (4)	0.4476 (2)	0.0571 (10)
C12	0.0847 (2)	1.1596 (5)	0.5105 (2)	0.0654 (11)
C13	0.1677 (3)	1.0867 (5)	0.5311 (1)	0.0630 (11)
Cr′	−0.06294 (3)	1.02289 (6)	0.21045 (2)	0.0353 (1)
O1′	−0.1369 (2)	1.3580 (3)	0.1636 (1)	0.0842 (10)
O2′	0.1150 (1)	1.2058 (3)	0.2418 (1)	0.0639 (7)
O3′	−0.1245 (2)	1.1179 (3)	0.3366 (1)	0.0674 (8)
O4′	0.1511 (1)	0.7647 (3)	0.25702 (9)	0.0471 (6)
C1′	−0.1087 (2)	1.2280 (4)	0.1815 (1)	0.0529 (10)
C2′	0.0470 (2)	1.1332 (4)	0.2295 (1)	0.0421 (8)
C3′	−0.1011 (2)	1.0805 (4)	0.2869 (1)	0.0435 (9)
C4′	0.0650 (2)	0.6753 (4)	0.2605 (1)	0.0407 (8)
C5′	0.0314 (2)	0.6712 (4)	0.3264 (1)	0.0559 (10)
C6′	−0.0611 (3)	0.5766 (5)	0.3276 (2)	0.0707 (12)
C7′	−0.1362 (2)	0.6714 (4)	0.2907 (2)	0.0657 (12)
C8′	−0.1031 (2)	0.7503 (4)	0.2314 (1)	0.0465 (9)
C9′	−0.0077 (2)	0.7531 (3)	0.2171 (1)	0.0375 (8)
C10′	0.0187 (2)	0.8294 (4)	0.1607 (1)	0.0437 (9)
C11′	−0.0476 (2)	0.8973 (5)	0.1190 (1)	0.0569 (10)
C12′	−0.1417 (2)	0.8976 (5)	0.1341 (2)	0.0624 (11)
C13′	−0.1690 (2)	0.8237 (4)	0.1890 (2)	0.0587 (11)

Table 2. *Selected geometric parameters (Å, °)*

Cr—C1	1.815 (3)	Cr′—C1′	1.833 (3)
Cr—C2	1.823 (3)	Cr′—C2′	1.833 (3)
Cr—C3	1.814 (3)	Cr′—C3′	1.811 (3)
Cr—C8	2.256 (3)	Cr′—C8′	2.252 (3)
Cr—C9	2.246 (3)	Cr′—C9′	2.251 (3)
Cr—C10	2.202 (3)	Cr′—C10′	2.207 (3)
Cr—C11	2.221 (3)	Cr′—C11′	2.220 (3)
Cr—C12	2.205 (3)	Cr′—C12′	2.201 (3)
Cr—C13	2.216 (3)	Cr′—C13′	2.214 (3)
O1—C1	1.165 (4)	O1′—C1′	1.154 (4)
O2—C2	1.154 (4)	O2′—C2′	1.152 (4)
O3—C3	1.167 (4)	O3′—C3′	1.169 (4)
O4—C4	1.422 (3)	O4′—C4′	1.421 (3)
C4—C5	1.517 (4)	C4′—C5′	1.517 (4)
C4—C9	1.508 (4)	C4′—C9′	1.510 (4)
C5—C6	1.524 (5)	C5′—C6′	1.519 (5)
C6—C7	1.506 (6)	C6′—C7′	1.515 (5)
C7—C8	1.509 (5)	C7′—C8′	1.509 (5)
C12—Cr—C13	36.4 (1)	C12′—Cr′—C13′	36.5 (1)
C11—Cr—C13	65.8 (1)	C11′—Cr′—C13′	65.7 (1)
C11—Cr—C12	36.7 (1)	C11′—Cr′—C12′	36.8 (1)

C10—Cr—C13	77.7 (1)	C10′—Cr′—C13′	77.8 (1)
C10—Cr—C12	66.1 (1)	C10′—Cr′—C12′	66.4 (1)
C10—Cr—C11	36.8 (1)	C10′—Cr′—C11′	36.7 (1)
C9—Cr—C13	66.3 (1)	C9′—Cr′—C13′	66.2 (1)
C9—Cr—C12	78.6 (1)	C9′—Cr′—C12′	78.8 (1)
C9—Cr—C11	66.7 (1)	C9′—Cr′—C11′	66.5 (1)
C9—Cr—C10	37.0 (1)	C9′—Cr′—C10′	37.0 (1)
C8—Cr—C13	37.0 (1)	C8′—Cr′—C13′	37.0 (1)
C8—Cr—C12	66.3 (1)	C8′—Cr′—C12′	66.6 (1)
C8—Cr—C11	78.2 (1)	C8′—Cr′—C11′	78.1 (1)
C8—Cr—C10	65.9 (1)	C8′—Cr′—C10′	66.1 (1)
C8—Cr—C9	36.6 (1)	C8′—Cr′—C9′	36.5 (1)
C3—Cr—C13	98.1 (1)	C3′—Cr′—C13′	98.2 (1)
C3—Cr—C12	129.7 (1)	C3′—Cr′—C12′	129.2 (1)
C3—Cr—C11	163.7 (1)	C3′—Cr′—C11′	163.8 (1)
C3—Cr—C10	140.9 (1)	C3′—Cr′—C10′	142.3 (1)
C3—Cr—C9	105.4 (1)	C3′—Cr′—C9′	106.8 (1)
C3—Cr—C8	87.2 (1)	C3′—Cr′—C8′	88.0 (1)
C2—Cr—C13	163.0 (1)	C2′—Cr′—C13′	163.3 (1)
C2—Cr—C12	142.9 (1)	C2′—Cr′—C12′	142.8 (1)
C2—Cr—C11	107.4 (1)	C2′—Cr′—C11′	107.5 (1)
C2—Cr—C10	87.9 (1)	C2′—Cr′—C10′	88.0 (1)
C2—Cr—C9	96.8 (1)	C2′—Cr′—C9′	97.2 (1)
C2—Cr—C8	128.1 (1)	C2′—Cr′—C8′	128.4 (1)
C2—Cr—C3	87.3 (1)	C2′—Cr′—C3′	87.7 (1)
C1—Cr—C13	107.4 (1)	C1′—Cr′—C13′	107.7 (1)
C1—Cr—C12	88.0 (1)	C1′—Cr′—C12′	87.7 (1)
C1—Cr—C11	97.2 (1)	C1′—Cr′—C11′	97.2 (1)
C1—Cr—C10	128.6 (1)	C1′—Cr′—C10′	128.4 (1)
C1—Cr—C9	163.8 (1)	C1′—Cr′—C9′	163.6 (1)
C1—Cr—C8	143.0 (1)	C1′—Cr′—C8′	143.3 (1)
C1—Cr—C3	90.1 (1)	C1′—Cr′—C3′	88.8 (1)
C1—Cr—C2	88.6 (1)	C1′—Cr′—C2′	88.0 (1)
Cr—C1—O1	179.0 (3)	Cr′—C1′—O1′	179.3 (3)
Cr—C2—O2	178.3 (3)	Cr′—C2′—O2′	178.4 (3)
Cr—C3—O3	177.5 (3)	Cr′—C3′—O3′	179.1 (3)
O4—C4—C9	110.7 (2)	O4′—C4′—C9′	110.9 (2)
O4—C4—C5	110.8 (2)	O4′—C4′—C5′	111.0 (2)
C5—C4—C9	111.1 (2)	C5′—C4′—C9′	111.0 (2)
C4—C5—C6	108.6 (3)	C4′—C5′—C6′	109.0 (3)
C5—C6—C7	112.1 (3)	C5′—C6′—C7′	111.3 (3)
C6—C7—C8	114.1 (3)	C6′—C7′—C8′	114.1 (3)
Cr—C8—C7	130.1 (2)	Cr′—C8′—C7′	130.1 (2)
C7—C8—C13	119.4 (3)	C7′—C8′—C13′	119.5 (3)
C7—C8—C9	121.6 (3)	C7′—C8′—C9′	121.6 (3)
Cr—C8—C13	70.0 (2)	Cr′—C8′—C13′	70.0 (2)
Cr—C8—C9	71.3 (2)	Cr′—C8′—C9′	71.7 (2)
C4—C9—C8	120.8 (3)	C4′—C9′—C8′	121.0 (2)
Cr—C9—C8	72.1 (2)	Cr′—C9′—C8′	71.8 (2)
Cr—C9—C4	131.0 (2)	Cr′—C9′—C4′	130.6 (2)
C4—C9—C10	120.8 (2)	C4′—C9′—C10′	120.4 (2)
Cr—C9—C10	69.8 (2)	Cr′—C9′—C10′	69.8 (2)
Cr—C10—C9	73.2 (2)	Cr′—C10′—C9′	73.2 (2)
Cr—C10—C11	72.4 (2)	Cr′—C10′—C11′	72.2 (2)
Cr—C11—C10	70.9 (2)	Cr′—C11′—C10′	71.1 (2)
Cr—C11—C12	71.0 (2)	Cr′—C11′—C12′	70.8 (2)
Cr—C12—C11	72.2 (2)	Cr′—C12′—C11′	72.3 (2)
Cr—C12—C13	72.2 (2)	Cr′—C12′—C13′	72.2 (2)
Cr—C13—C12	71.4 (2)	Cr′—C13′—C12′	71.2 (2)
Cr—C13—C8	73.0 (2)	Cr′—C13′—C8′	72.9 (2)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1985). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN* (Fair, 1990). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1200). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1996). C**52**, 615–617

Diaquadioxobis(pyridine-3-carboxylato)uranium(VI)

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Abstract

The uranyl group in the title compound, $[UO_2(C_6H_4NO_2)_2(H_2O)_2]$, with U—O 1.782 (4) Å, is sixfold coordinated by two water molecules [U—O 2.431 (4) Å] and two bidentate nicotinic acid anions (pyridine-3-carboxylato ions) [U—O 2.459 (4)–2.486 (4) Å]. Individual molecules are linked into chains by hydrogen bonds between coordinated water molecules and pyridine N atoms.

Comment

Coordination of amino acids to uranyl ions takes place exclusively through the carboxylate function (Alcock, Flanders, Kemp & Shand, 1985; Bismondo, Casellato,