Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

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

		Occupancy	x	y	z	$U_{\rm eq}$
Mo1	8(j)	1.0	0.57709 (5)	$1-x$	0.57842 (5)	0.0333 (3)
Mo2	4(g)	1.0	0.60861 (7)	x	1/2	0.0326 (4)
Te1	8(i)	0.058	0.7547 (2)	0.4573 (2)	1/2	0.050 (1)
Cl1	8(i)	0.942	x (Te1)	y (Te1)	1/2	0.050
Te2	8(j)	0.192	0.3909 (1)	x	0.6561 (1)	0.0579 (8)
Cl2	8(j)	0.808	x (Te2)	x (Te2)	z (Te2)	0.0579
Cl3	8(j)	1.0	0.6831 (2)	$1-x$	0.6798 (2)	0.067 (1)
Cl4	4(g)	1.0	0.7530 (2)	x	1/2	0.067 (4)
N1	2(b)	1.0	1/2	1/2	0	0.056 (8)
N2	4(d)	1.0	1/2	0	1/4	0.053 (5)
C1	8(j)	0.5†	0.556 (2)	$1-x$	−0.080 (2)	0.11 (1)
C2	4(e)	1.0	1/2	1/2	−0.156 (1)	0.19 (1)
C3	8(i)	0.5†	0.376 (2)	0.480 (2)	0	0.08 (1)
C4	4(f)	1.0	0.348 (1)	x	0	0.14 (1)
C5	16(k)	1.0	0.5836 (7)	0.0667 (8)	0.1981 (5)	0.074 (6)
C6	16(k)	1.0	0.5269 (9)	0.1431 (9)	0.1372 (6)	0.113 (9)

† Occupancy fixed by symmetry.

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

Mo1—Mo1$^{\rm i}$	2.618 (2)	Mo2—Te1	2.523 (2)
Mo1—Mo1$^{\rm ii}$	2.616 (2)	Mo2—Te1$^{\rm iii}$	2.523 (2)
Mo1—Mo2	2.611 (1)	Mo2—Te2$^{\rm ii}$	2.606 (2)
Mo1—Te1	2.534 (2)	Mo2—Te2$^{\rm iv}$	2.606 (2)
Mo1—Te2	2.611 (2)	Mo2—Cl4	2.449 (4)
Mo1—Cl3	2.470 (3)		
Mo1$^{\rm i}$—Mo1—Mo1$^{\rm ii}$	90.00	Mo1—Mo2—Te1$^{\rm iii}$	119.23 (6)
Mo1$^{\rm i}$—Mo1—Mo2	59.91 (2)	Mo1—Mo2—Te2$^{\rm ii}$	60.06 (4)
Mo1$^{\rm i}$—Mo1—Te1	58.90 (3)	Mo1—Mo2—Te2$^{\rm iv}$	120.23 (5)
Mo1$^{\rm i}$—Mo1—Te2	119.78 (4)	Mo1—Mo2—Cl4	134.88 (2)
Mo1$^{\rm i}$—Mo1—Cl3	133.26 (8)	Mo1$^{\rm i}$—Mo2—Mo1$^{\rm ii}$	90.24 (4)
Mo1$^{\rm ii}$—Mo1—Mo2	59.94 (2)	Mo1$^{\rm i}$—Mo2—Mo1$^{\rm iv}$	60.11 (4)
Mo1$^{\rm ii}$—Mo1—Te1	118.64 (5)	Mo1$^{\rm i}$—Mo2—Te1	59.13 (5)
Mo1$^{\rm ii}$—Mo1—Te2	59.93 (3)	Mo1$^{\rm i}$—Mo2—Te1$^{\rm iii}$	119.23 (6)
Mo1$^{\rm ii}$—Mo1—Cl3	136.73 (8)	Mo1$^{\rm i}$—Mo2—Te2$^{\rm ii}$	120.23 (5)
Mo2—Mo1—Mo2$^{\rm ii}$	89.76 (4)	Mo1$^{\rm i}$—Mo2—Te2$^{\rm iv}$	60.06 (4)
Mo2—Mo1—Te1	58.69 (5)	Mo1$^{\rm i}$—Mo2—Cl4	134.88 (2)
Mo2—Mo1—Te1$^{\rm v}$	118.80 (4)	Mo1$^{\rm ii}$—Mo2—Mo1$^{\rm iv}$	60.17 (4)
Mo2—Mo1—Te2	119.89 (4)	Te1—Mo2—Te1$^{\rm iii}$	178.0 (1)
Mo2—Mo1—Te2$^{\rm ii}$	59.87 (4)	Te1—Mo2—Te2$^{\rm ii}$	90.00 (2)
Mo2—Mo1—Cl3	135.09 (2)	Te1—Mo2—Te2$^{\rm iv}$	90.00 (2)
Te1—Mo1—Te1$^{\rm v}$	90.40 (9)	Te1—Mo2—Cl4	91.01 (6)
Te1—Mo1—Te2	178.32 (6)	Te1$^{\rm iii}$—Mo2—Te2$^{\rm ii}$	90.00 (2)
Te1—Mo1—Te2$^{\rm ii}$	89.64 (6)	Te1$^{\rm iii}$—Mo2—Te2$^{\rm iv}$	90.00 (2)
Te1—Mo1—Cl3	90.29 (7)	Te1$^{\rm iii}$—Mo2—Cl4	91.01 (6)
Te1$^{\rm v}$—Mo1—Te2	89.64 (6)	Te2$^{\rm ii}$—Mo2—Te2$^{\rm iv}$	179.7 (1)
Te1$^{\rm v}$—Mo1—Te2$^{\rm ii}$	178.32 (6)	Te2$^{\rm ii}$—Mo2—Cl4	89.83 (5)
Te1$^{\rm v}$—Mo1—Cl3	90.29 (7)	Te2$^{\rm iv}$—Mo2—Cl4	89.83 (5)
Te2—Mo1—Te2$^{\rm ii}$	90.27 (8)	Mo1—Te1—Mo1$^{\rm i}$	62.20 (6)
Te2—Mo1—Cl3	91.39 (7)	Mo1—Te1—Mo2	62.18 (6)
Mo1—Mo2—Mo1$^{\rm i}$	60.17 (4)	Mo1$^{\rm i}$—Te1—Mo2	62.18 (6)
Mo1—Mo2—Mo1$^{\rm ii}$	60.11 (4)	Mo1—Te2—Mo1$^{\rm ii}$	60.12 (5)
Mo1—Mo2—Mo1$^{\rm iv}$	90.24 (4)	Mo1—Te2—Mo2$^{\rm ii}$	60.08 (5)
Mo1—Mo2—Te1	59.13 (5)	Mo1$^{\rm ii}$—Te2—Mo2$^{\rm ii}$	60.08 (5)

Symmetry codes: (i) $x, y, 1-z$; (ii) $1-x, 1-y, z$; (iii) y, x, z; (iv) $1-x, 1-y, 1-z$; (v) $1-y, 1-x, z$.

The structure was solved on the basis of the structure of isostructural $(\mathrm{Et_4N})_3[(\mathrm{Mo_6Br_7S})\mathrm{Cl_6}]$ (Ebihara, Isobe, Sasaki & Saito, 1992). Each independent capping site was refined as Te + Cl and the sum of the occupancies of Te was restricted to 0.25. Since one $\mathrm{Et_4N^+}$ ion was disordered at the *mmm* site, the occupancies of C1 and C3 were set equal to 0.5.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN TABLES*.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: NA1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[Bis(3-aminopropyl)amine-*N*,*N*′,*N*″]-[glycylglycinato(2–)-*N*,*N*′,*O*]-chromium(III) Perchlorate Monohydrate, $[\mathrm{Cr(C_4H_6N_2O_3)(C_6H_{17}N_3)}]\mathrm{ClO_4.H_2O}$

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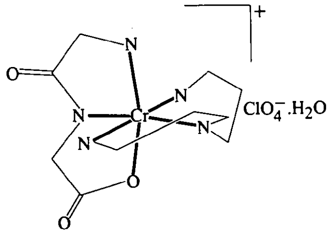
Abstract

The mixed-ligand complex $[\mathrm{Cr(dpt)(Gly\text{-}Gly)}]\mathrm{ClO_4.H_2O}$ [where dpt = bis(3-aminopropyl)amine and $\mathrm{H_2Gly\text{-}Gly}$ = glycylglycine] adopts a structure in which the ligands are tridentate and coordinate meridionally. The four amine N atoms, the carboxyl O atom and the peptide N atom

produce a distorted octahedral coordination geometry around the Cr atom. The [Cr($C_4H_6N_2O_3$)($C_6H_{17}N_3$)]$^+$ cation is linked to the perchlorate group by two hydrogen bonds, O(*W*)···O(2) [2.746 (8) Å] and O(*W*)···O(4) [2.981 (10) Å], which help to stabilize the crystal structure. The Cr—N bond to the peptide N atom is shorter than those to the amine N atoms, which suggests that the peptide N atom bonds more strongly with chromium(III) than normal N atoms.

Comment

As an octahedral complex, [Cr(dpt)(Gly-Gly)]ClO_4.H_2O may exist in the facial or meridional configuration. UV–visible and infrared spectra can be used diagnostically to identify the geometric isomers of chromium(III) complexes with tridentate ligands (Subramaniam, 1989). It should be noted, however, that assignments based on spectroscopic investigations are not always conclusive (Stearns & Armstrong, 1992). In addition, modern ligand-field analysis is correlated with a knowledge of the crystal structure, since the splittings and positions of sharp line transitions are quite sensitive to the metal–ligand geometry (Hoggard, 1986). An X-ray crystallographic study of the title complex, (I), was undertaken to establish the details of its coordination and verify the spectroscopic structural assignment previously described (Choi & Hoggard, 1992).



(I)

The structure of the *mer*-[Cr(dpt)(Gly-Gly)]$^+$ cation, with the atom-numbering scheme, is shown in Fig. 1. The Cr atom is in a distorted octahedral environment, surrounded by O(1), N(1), N(2), N(3), N(4) and N(5) from Gly-Gly and dpt ligands, with a Cr···Cr($\frac{1}{2}+x$, $\frac{3}{2}-y$, $2-z$) separation of 6.593 (2) Å. In the Gly-Gly ligand, the bond lengths O(2)—C(8) [1.270 (8) Å] and O(3)—C(10) [1.229 (8) Å] reflect typical double-bond character. The geometric parameters for the Gly-Gly ligand show sp^2 hybridization of the peptide N atom.

The Cr—N(peptide) bond of 1.961 (5) Å is shorter than the average for a Cr—N(amine) bond [2.107 (6) Å] (Murdoch, Cooper, Hambley, Hunter & Freeman, 1986; Subramaniam, Garvey, Lee & Hoggard, 1988). This appears to indicate that the Cr—N(peptide) bond is stronger than those involving other N atoms. The results are also consistent with those concerning the bonding properties of the peptide N atom (Choi & Hoggard, 1992). The four donor atoms N(1), N(3), N(4) and N(5) in one equatorial plane are coplanar to within 0.012 (3) Å, and the atoms O(1), N(1), N(2), N(4) in another equatorial plane are also coplanar, to within 0.011 (4) Å. The dihedral angle between these planes is 89.0 (2)°. All non-H atoms in the Gly-Gly ligand are coplanar to within 0.120 (3) Å, while the two terminal six-membered chelate rings in the dpt ligand are in stable chair forms. The N(2)—Cr—O(1) bond angle [162.6 (2)°] is in good agreement with the results of a molecular-mechanics calculation (Choi & Hoggard, 1992). The coplanarity of the bonds at the peptide N atom indicates that the amide group is deprotonated (Murdoch *et al.*, 1986). The configuration of the Gly-Gly and dpt tridentate ligands in the complex has been established as meridional by the X-ray structural analysis.

The perchlorate ion is somewhat disordered. A model involving three overlapping fractional ions based on peaks found in the difference Fourier map did not result in a lower value of *R* than one using one ion with large displacement factors for the O atoms. Therefore, this latter, simpler model was used. A similar result was obtained for isothiocyanatobis(1,10-phenanthroline)copper(II) perchlorate (Parker, Manson & Breneman, 1994). The perchlorate ClO_4 group has angles close to tetrahedral, with Cl—O distances ranging from 1.322 (9) to 1.412 (7) Å, and with O—Cl—O angles ranging from 105.4 (5) to 111.9 (5)°.

The crystal structure is stabilized by the hydrogen bonds O(*W*)···O(2) [2.746 (8) Å] and O(*W*)···O(4) [2.981 (10) Å], such that the water molecule bridges the

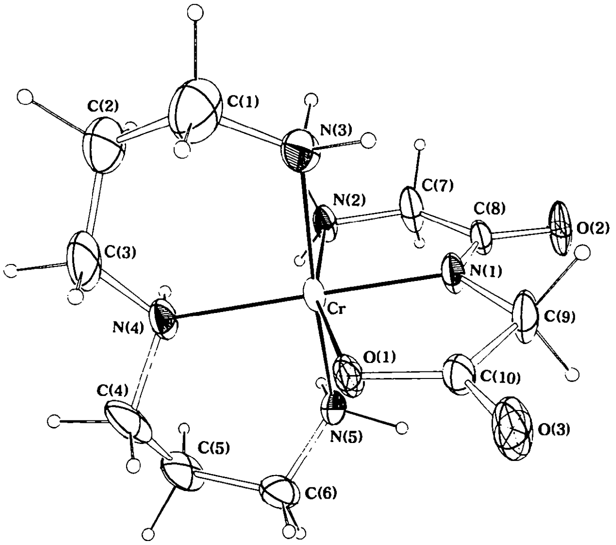


Fig. 1. Perspective drawing (*ORTEP*II; Johnson, 1971) of the *mer*-[Cr(dpt)(Gly-Gly)]$^+$ cation showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

$[Cr(C_6H_{17}N_3)(C_4H_6N_2O_3)]^+$ cation and the ClO_4 anion. The shortest contacts between neighbouring molecules are O(*W*)···N(4) 3.021 (9), O(*W*)···N(2) 3.027 (8) and O(2)···N(3) 3.078 (8) Å. The crystal packing is governed by ionic forces and hydrogen bonds.

Experimental

Light-red crystals of the title compound were prepared as reported previously (Choi & Hoggard, 1992).

Crystal data

$[Cr(C_4H_6N_2O_3)(C_6H_{17}N_3)]$-$ClO_4.H_2O$
$M_r = 430.79$
Orthorhombic
$P2_12_12_1$
$a = 11.783\ (1)$ Å
$b = 16.365\ (4)$ Å
$c = 8.981\ (1)$ Å
$V = 1731.8\ (8)$ Å^3
$Z = 4$
$D_x = 1.65$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.09–14.13°$
$\mu = 0.805$ mm^{-1}
$T = 293$ K
Block
0.46 × 0.33 × 0.30 mm
Light red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1617 measured reflections
1587 independent reflections
1417 observed reflections [$F > 3\sigma(F)$]

$\theta_{max} = 24°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 10$
3 standard reflections frequency: 180 min intensity decay: 4.3%

Refinement

Refinement on F
$R = 0.0416$
$wR = 0.0416$
$S = 1.4815$
1417 reflections
251 parameters
Unit weights applied
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
Cr	0.1197 (1)	0.6736 (1)	0.9118 (1)	0.022 (1)
Cl	0.5906 (2)	0.5599 (1)	1.7198 (2)	0.041 (1)
O(1)	−0.0120 (4)	0.7175 (3)	1.0211 (5)	0.028 (3)
O(2)	0.2885 (5)	0.5636 (3)	1.2560 (6)	0.044 (3)
O(3)	−0.0789 (4)	0.7441 (3)	1.2458 (6)	0.041 (3)
O(4)	0.4813 (6)	0.5667 (5)	1.6564 (10)	0.098 (6)
O(5)	0.6410 (8)	0.6363 (4)	1.7416 (11)	0.115 (7)
O(6)	0.5863 (12)	0.5184 (9)	1.8460 (11)	0.199 (12)
O(7)	0.6553 (7)	0.5190 (6)	1.6149 (11)	0.122 (7)
O(*W*)	0.2724 (5)	0.6407 (5)	1.5268 (6)	0.069 (4)
N(1)	0.1663 (5)	0.6405 (3)	1.1126 (6)	0.028 (3)
N(2)	0.2750 (5)	0.6166 (4)	0.8610 (6)	0.028 (3)
N(3)	0.0411 (5)	0.5600 (3)	0.8853 (7)	0.034 (3)
N(4)	0.0689 (5)	0.7071 (4)	0.6917 (7)	0.033 (3)
N(5)	0.2063 (5)	0.7834 (3)	0.9494 (7)	0.036 (4)
C(1)	−0.0702 (7)	0.5569 (6)	0.8074 (10)	0.051 (5)
C(2)	−0.0614 (8)	0.5875 (6)	0.6497 (9)	0.051 (5)
C(3)	−0.0446 (6)	0.6791 (6)	0.6397 (9)	0.052 (5)
C(4)	0.0751 (8)	0.7966 (5)	0.6529 (10)	0.056 (6)
C(5)	0.1809 (8)	0.8382 (5)	0.6937 (10)	0.054 (6)
C(6)	0.1850 (11)	0.8570 (6)	0.8600 (11)	0.069 (7)
C(7)	0.3235 (7)	0.5790 (5)	0.9965 (8)	0.039 (5)
C(8)	0.2556 (6)	0.5947 (4)	1.1337 (7)	0.027 (4)
C(9)	0.0884 (6)	0.6631 (5)	1.2318 (8)	0.036 (4)
C(10)	−0.0075 (6)	0.7115 (4)	1.1657 (8)	0.028 (4)

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

Cr—O(1)	1.972 (5)	O(3)—C(10)	1.229 (8)
Cr—N(1)	1.961 (5)	N(1)—C(8)	1.306 (8)
Cr—N(2)	2.104 (6)	N(1)—C(9)	1.458 (9)
Cr—N(3)	2.091 (6)	N(2)—C(7)	1.479 (9)
Cr—N(4)	2.137 (6)	N(3)—C(1)	1.487 (10)
Cr—N(5)	2.094 (5)	N(4)—C(3)	1.490 (9)
O(1)—C(10)	1.303 (8)	N(4)—C(4)	1.507 (11)
O(2)—C(8)	1.270 (8)	N(5)—C(6)	1.468 (11)
N(1)—Cr—O(1)	82.1 (2)	C(1)—N(3)—Cr	118.3 (5)
N(2)—Cr—O(1)	162.6 (2)	C(3)—N(4)—Cr	117.6 (5)
N(2)—Cr—N(1)	80.4 (2)	C(4)—N(4)—Cr	116.7 (5)
N(3)—Cr—O(1)	91.8 (2)	C(4)—N(4)—C(3)	105.7 (6)
N(3)—Cr—N(1)	89.0 (2)	C(9)—N(1)—C(8)	123.1 (6)
N(3)—Cr—N(2)	88.1 (2)	C(2)—C(1)—N(3)	111.7 (7)
N(4)—Cr—O(1)	98.4 (2)	C(3)—C(2)—C(1)	113.2 (7)
N(4)—Cr—N(1)	179.4 (5)	C(2)—C(3)—N(4)	113.8 (7)
N(4)—Cr—N(2)	99.0 (2)	C(5)—C(4)—N(4)	115.8 (7)
N(4)—Cr—N(3)	89.9 (2)	C(6)—C(5)—C(4)	111.4 (9)
N(5)—Cr—O(1)	89.5 (2)	C(5)—C(6)—N(5)	112.1 (7)
N(5)—Cr—N(1)	87.3 (2)	C(8)—C(7)—N(2)	113.6 (5)
N(5)—Cr—N(2)	89.5 (2)	N(1)—C(8)—O(2)	127.0 (6)
N(5)—Cr—N(3)	175.6 (2)	C(7)—C(8)—O(2)	118.8 (6)
N(5)—Cr—N(4)	93.8 (2)	C(7)—C(8)—N(1)	114.3 (6)
C(10)—O(1)—Cr	115.9 (5)	C(10)—C(9)—N(1)	108.5 (6)
C(8)—N(1)—Cr	121.2 (5)	O(3)—C(10)—O(1)	121.5 (7)
C(9)—N(1)—Cr	115.4 (4)	C(9)—C(10)—O(1)	117.6 (6)
C(6)—N(5)—Cr	122.2 (6)	C(9)—C(10)—O(3)	120.8 (6)
C(7)—N(2)—Cr	110.0 (4)		
N(1)—C(9)—C(10)—O(1)	6.8 (6)		
N(1)—C(9)—C(10)—O(3)	−173.2 (9)		
N(2)—C(7)—C(8)—O(2)	178.5 (9)		
N(2)—C(7)—C(8)—N(1)	−1.6 (6)		
N(3)—C(1)—C(2)—C(3)	−71.8 (8)		
N(4)—C(4)—C(5)—C(6)	−77.1 (9)		
C(1)—C(2)—C(3)—N(4)	69.9 (8)		
C(3)—N(4)—C(4)—C(5)	−179.9 (9)		
C(4)—N(4)—C(3)—C(2)	172.1 (9)		
C(4)—C(5)—C(6)—N(5)	74.2 (9)		
C(8)—N(1)—C(9)—C(10)	−176.6 (9)		
C(9)—N(1)—C(8)—O(2)	−0.2 (7)		
C(9)—N(1)—C(8)—C(7)	179.9 (9)		

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O(*W*)—H(1*W*)···O(2)	1.032 (6)	1.719 (5)	2.746 (8)	172.6 (4)
O(*W*)—H(2*W*)···O(4)	0.927 (6)	2.338 (7)	2.981 (10)	126.2 (6)
O(*W*)···N(2^i)	-	-	3.027 (8)	-
O(*W*)···N(4^i)	-	-	3.021 (9)	-
O(2)···N(3^{ii})	-	-	3.078 (8)	-

Symmetry codes: (i) $x, y, 1 + z$; (ii) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$.

The space group and approximate cell dimensions were determined in a preliminary experiment using Weissenberg and precession photography (Suh, Suh, Ko, Aoki & Yamazaki, 1988). The systematic absences *h*00 *h* = 2*n*, 0*k*0 *k* = 2*n*, 00*l* *l* = 2*n* uniquely defined the non-centrosymmetric orthorhombic space group $P2_12_12_1$. The crystal was mounted on a glass fiber and secured to a goniometer head. The diffractometer was controlled by a MicroPDP11/53 computer and generator settings of 40 kV and 20 mA were used. Data were collected using an ω-scan width of $(0.8 + 0.34\tan\theta)^\circ$; $[(\sin\theta)/\lambda]_{max} = 0.5723$ Å^{-1}. Lorentz and polarization corrections were applied to the intensity data, but no correction for absorption was made.

The structure was solved by the application of direct methods using *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and refined by full-matrix least-squares on F using *SHELX*76 (Sheldrick, 1976) with anisotropic displacement factors for all non-H atoms. The H atoms H(1*W*) and H(2*W*) of the water molecule were found from a difference Fourier synthesis and their positions were not refined. The remaining H atoms were located by geometrical calculation (C—H 1.08 Å) and all H atoms were refined isotropically. The inversion-related structure was refined in an identical fashion and gave a poorer result ($R = 0.0464$, $wR = 0.0464$, $S = 1.6413$).

Data collection, cell refinement and data reduction: *SDP* (Enraf–Nonius, 1985). Molecular graphics: *ORTEP*II (Johnson, 1971). Preparation of material for publication: *GEOM* (Shin, 1978). All computations were performed using the MicroVAX/VMS 3400 computer at Chungnam National University.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms and least-squares-planes data, and a packing diagram have been deposited with the IUCr (Reference: JZ1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(η^4-1,5-Cyclooctadiene)(iodo)(methyl)(thioacetylacetonato)iridium(III)

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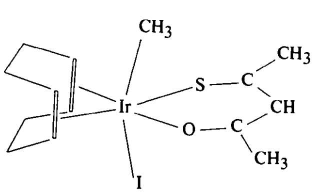
Abstract

The structure determination of the title complex, $[Ir(C_5H_7OS)(CH_3)(I)(C_8H_{12})]$, revealed that the oxidative addition of iodomethane to (η^4-1,5-cyclooctadiene)-(thioacetylacetonato)iridium(I) results in the formation of the *trans* isomer. The deviation of the I—Ir—CH_3 angle [156.1(3)°] from linearity is attributed to steric repulsion between the CH_3 group or I atom and the olefinic H atoms.

Comment

The use of $[Ir(CO)Cl(PPh_3)_2]$ in the hydrogenation of alkenes, as well as the use of [Ir(*o*-phen)(cod)Cl] (*o*-phen = 1,10-phenanthroline; cod = 1,5-cyclooctadiene) in the conversion of methanol, are two illustrative examples of the catalytic application of Ir^I complexes in well known industrial processes (Dickson, 1985). More often than not, oxidative addition of H_2 or methyl halides forms an integral part of these catalytic cycles. The oxidative addition of iodomethane to square-planar Ir^I and Rh^I complexes can occur *via cis* or *trans* addition, depending on the mechanism that is followed during the reaction. In the majority of cases (Collman & MacLaury, 1974; Cross, 1985), the *trans* isomer has been isolated, but *cis* addition in, for example, $[Rh(cupf)(CO)(PPh_3)(CH_3)(I)]$ (cupf = *N*-nitrosophenylhydroxylaminato ligand) is also found (Basson, Leipoldt, Roodt & Venter, 1987). The title compound, $[Ir(C_5H_7OS)(CH_3)(I)(C_8H_{12})]$, (1), was isolated as a product after the reaction between $[Ir(C_5H_7OS)(C_8H_{12})]$ and CH_3I; this study forms part of our ongoing research into the kinetics of the oxidative addition of CH_3I to different Rh^I and Ir^I systems (Steyn, Roodt & Leipoldt, 1992) and the solid-state properties of the products.



(1)