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η^6-Tricarbonylchromium Complexes of Methyl 4,6-*O*-Benzylidene-α-D-glucopyranosides

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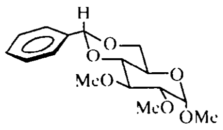
Abstract

The structures of the η^6-tricarbonylchromium complexes of four methyl 4,6-*O*-benzylidene-α-glucopyranosides, (2)–(5), have been determined, as well as that of the free monosaccharide, (1). These compounds are (*R*)-methyl 2,3-di-*O*-methyl-4,6-*O*-benzylidene-α-D-glucopyranoside [(1), $C_{16}H_{22}O_6$], tricarbonyl{(*R*)-methyl 2,3-di-*O*-methyl-4,6-*O*-(η^6-benzylidene)-α-D-glucopyranoside}chromium(0) {(2), $[Cr(C_{16}H_{22}O_6)(CO)_3]$}, tricarbonyl{(*R*)-methyl 2,3-di-*O*-methyl-4,6-*O*-[(1*R*,2*S*)-2-methylthio-η^6-phenylmethylene]-α-D-glucopyranoside}chromium(0) {(3), $[Cr(C_{17}H_{24}O_6S)(CO)_3]$}, tricarbonyl{(*R*)-methyl 2,3-di-*O*-methyl-4,6-*O*-[(1*R*,2*S*)-2-diphenylphosphino-η^6-phenylmethylene]-α-D-glucopyranoside}chromium(0) dichloromethane solvate {(4), $[Cr(C_{28}H_{31}O_6P)(CO)_3].CH_2Cl_2$} and tricarbonyl[(*R*)-methyl 2,3-di-*O*-methyl-4,6-*O*-(4-methoxy-η^6-phenylmethylene)-α-D-glucopyranoside]chromium(0) {(5), $[Cr(C_{17}H_{24}O_7)(CO)_3]$}. These structural studies were undertaken to establish the solid-state conformation of the complexed arene ring in the 4,6-*O*-benzylidene and the 4,6-*O*-*p*-methoxyphenylmethylene η^6-$Cr(CO)_3$ complexes, and also the absolute stereochemistry of some *ortho*-substituted derivatives of the 4,6-*O*-benzylidene complex.

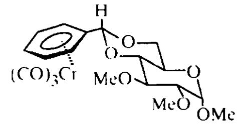
Comment

As part of a continuing investigation into the use of (η^6-arene)tricarbonylchromium complexes as key stereochemical elements in stereoselective synthesis, we have studied the use of some simple sugar derivatives as chiral auxiliaries.

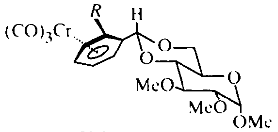
The synthesis of a series of η^6-$Cr(CO)_3$ complexes derived from the tricarbonylchromium complex of 2,3-di-*O*-methyl-4,6-*O*-benzylidene-α-D-glucopyranoside, (1), by diastereoselective deprotonation BuLi–electrophile quenching has been reported recently (Jin *et al.*, 1997). We have also examined this approach to asymmetric synthesis and report here the structures of the free sugar, (1), of the parent η^6-$Cr(CO)_3$ complex, (2), and two of its *ortho*-functionalized derivatives, (3) and (4), and of the η^6-$Cr(CO)_3$-*p*-methoxyphenylmethylene complex, (5). The trimethylsilyl derivative, (6), described by Jin *et al.* (1997), has also been obtained by us and the same structure and absolute stereochemistry determined. However, Jin *et al.* (1997) obtained only *ortho*-substituted products from (2) using Et_2O as the solvent for the deprotonation–electrophile quenching sequence, whereas we used tetrahydrofuran (THF) and isolated (PLC) both the *ortho*- and *meta*-isomers in approximately equal amounts. In the present work, crystals were grown for the *ortho*-products, (3) and (4), arising from the use of either MeSSMe or Ph_2PCl, respectively, to quench the intermediate aryllithium (BuLi, THF, 195 K). For each of these two products, the absolute stereochemistry of the derivatized arene ring was determined as 1*R*,2*S*; this is the same as that defined for the trimethylsilyl analogue, (6), by Jin *et al.* (1997) and confirmed by us.



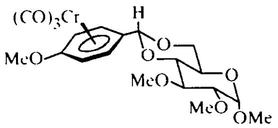
(1)



(2)



(3) R = SMe
(4) R = PPh_2 (CH_2Cl_2 solvate)
(6) R = $SiMe_3$



(5)

This absolute stereochemistry was rationalized (Jin *et al.*, 1997) as arising from a transition-state conformation having the benzylic (axial) H atom *syn* to the $Cr(CO)_3$ moiety; diastereoselective deprotonation of the *pro*-2*S* arene *ortho*-H atom is then promoted by intramolecular chelation of the base (BuLi) to the proximal 3-OMe group of the pyranoside. In the solid state, however, the parent 4,6-*O*-benzylidene complex, (2), has a conformation in which the benzylic H atom is *anti* to the $Cr(CO)_3$ moiety, presumably reflecting the stabilizing interactions involving the axially disposed lone pairs of the two 1,3-dioxane O atoms and two of the CO ligands. Moreover, the complexed phenyl ring is nearly coplanar with the C4—O and C6—O bonds [as it is also in the free monosaccharide, (1); there are minimal conformational changes consequent on complexation of (1)]. In solution, however, the reacting conformation is different due to the now overriding preference for the bulky $Cr(CO)_3$ group to minimize steric interactions. In contrast, in the *p*-methoxyphenylmethylene complex, (5), the $Cr(CO)_3$ group is *syn* with respect to the benzylic H atom in the solid state, and the phenyl ring is twisted towards the 3-OMe group.

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All of the complexes have the piano-stool arrangement, with three carbonyl groups bound on one side of the chromium and a η^6-aromatic ring bound to the other. The chromium to aromatic ring distance shows some difference depending on the ring substitution. Where there is no substitution or *para*-substitution, the distance is significantly greater [1.724 (2) and 1.721 (1) Å, respectively] than in the *ortho*-substituted compounds [1.710 (1) and 1.712 (2) Å]. The corresponding value for the trimethylsilyl derivative is 1.699 (2) Å, which is also *ortho*-substituted. The chromium–carbon distances to the carbonyl groups are all comparable and average 1.847 (4) Å.

The six-membered rings are all in chair conformations and are *trans*-fused. The interatomic distances within these rings are all comparable and show little deviation between molecules. In (4), the mean C—C distance within the phenyl rings bonded to phosphorus is 1.390 (5) Å, whereas the mean distance in the rings η^6-bonded to chromium is 1.411 (3) Å, reflecting the interaction between chromium and the aromatic ring.

In the case of the chromium complexes, the absolute configuration was established according to the method of Flack (1983) and Figs. 2–5 show the correct absolute configurations. In the case of the free glucopyranoside, (1), the absolute configuration could not be determined and Fig. 1 shows the same α-glucopyranoside configuration as the chromium complexes. The

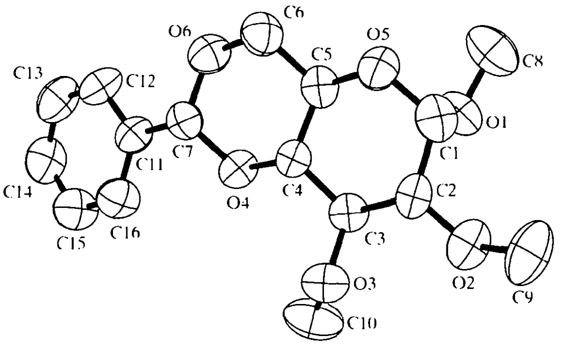

Fig. 1. The structure of (1) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

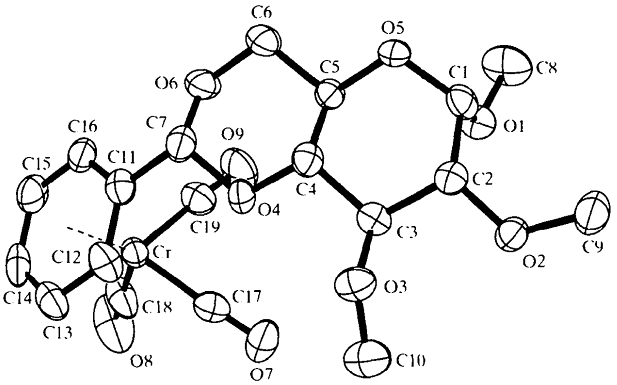

Fig. 2. The structure of (2) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

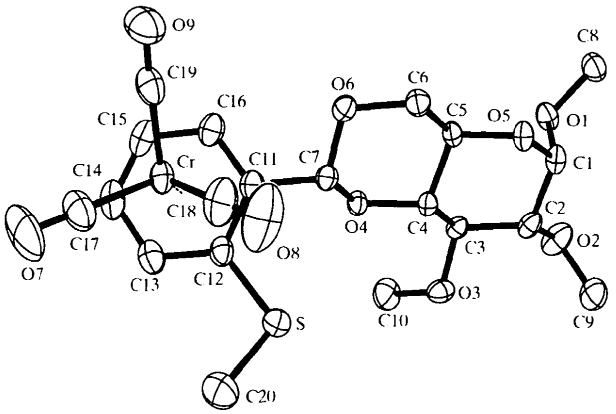

Fig. 3. The structure of (3) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

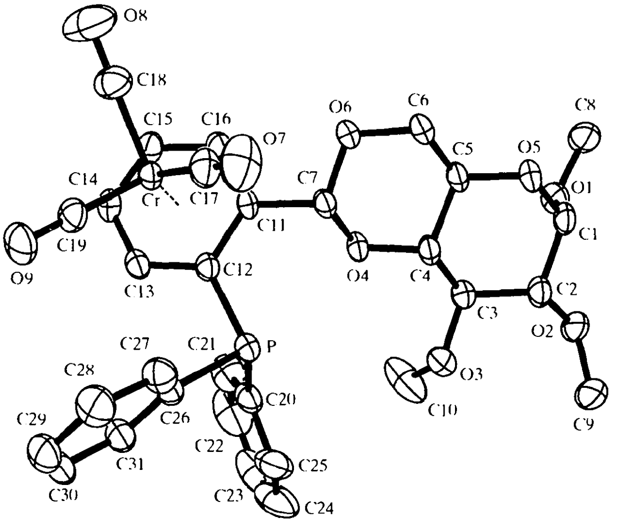

Fig. 4. The structure of (4) showing 50% probability displacement ellipsoids. H atoms and the dichloromethane solvate molecule have been omitted for clarity.

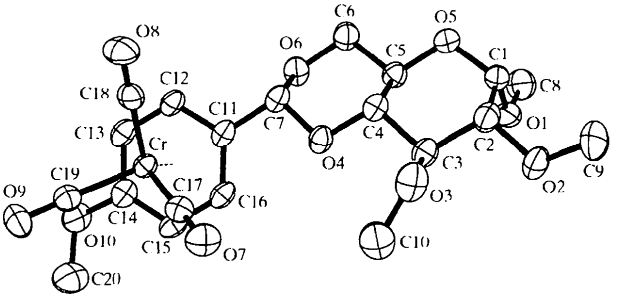

Fig. 5. The structure of (5) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

crystal structure of the thermodynamically less stable C7-epimer of (1), in which the phenyl group is axial, has been reported (Barnes *et al.*, 1979).

Experimental

(*R*)-Methyl 2,3-di-*O*-methyl-4,6-*O*-benzylidene-α-D-glucopyranoside, (1), was prepared from methyl α-D-glucopyranoside by acetal exchange with (dimethoxymethyl)benzene in dimethylformamide in the presence of *p*-TsOH, followed by methylation with Ag_2O/MeI in acetone. The 4-methoxyphenyl analogue, (5), was prepared in a similar manner *via* acetal exchange using 1-(dimethoxymethyl)-4-methoxybenzene. The tricarbonylchromium complexes (2) and (5) were formed from the appropriate benzylidene acetal precursor by thermally promoted ligand exchange using $Cr(CO)_6$ in Bu_2O–THF.

Compound (1)

Crystal data

$C_{16}H_{22}O_6$
$M_r = 310.34$
Monoclinic
$P2_1$
$a = 4.8635\ (7)$ Å
$b = 13.0125\ (18)$ Å
$c = 12.6580\ (18)$ Å
$\beta = 97.832\ (3)°$
$V = 793.61\ (19)$ Å^3
$Z = 2$
$D_x = 1.299$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 4243 reflections
$\theta = 2–25°$
$\mu = 0.099$ mm^{-1}
$T = 203\ (2)$ K
Needle
0.34 × 0.10 × 0.10 mm
Colourless

Data collection

Siemens SMART diffractometer
Area-detector scans
Absorption correction: empirical (Blessing, 1995)
$T_{\min} = 0.967$, $T_{\max} = 0.990$
4639 measured reflections
2655 independent reflections

1325 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$
$\theta_{\max} = 25°$
$h = -5 \rightarrow 5$
$k = -14 \rightarrow 15$
$l = 0 \rightarrow 15$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.202$
$S = 0.945$
2655 reflections
202 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1003P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.260$ e Å^{-3}
$\Delta\rho_{\min} = -0.307$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected bond lengths (Å) for (1)*

O4—C7	1.423 (6)	C1—C2	1.509 (9)
O4—C4	1.446 (6)	C2—C3	1.522 (7)
O5—C1	1.417 (7)	C3—C4	1.488 (8)
O5—C5	1.430 (6)	C4—C5	1.480 (8)
O6—C7	1.406 (7)	C5—C6	1.493 (8)
O6—C6	1.429 (7)	C7—C11	1.488 (8)

Compound (2)

Crystal data

$[Cr(C_{16}H_{22}O_6)(CO)_3]$
$M_r = 446.37$
Orthorhombic
$P2_12_12_1$
$a = 8.3014\ (2)$ Å
$b = 13.8503\ (2)$ Å
$c = 17.7567\ (5)$ Å
$V = 2041.61\ (8)$ Å^3
$Z = 4$
$D_x = 1.452$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5986 reflections
$\theta = 1.8–26.3°$
$\mu = 0.608$ mm^{-1}
$T = 203\ (2)$ K
Needle
0.47 × 0.08 × 0.04 mm
Yellow

Data collection

Siemens SMART diffractometer
Area-detector scans
Absorption correction: empirical (Blessing, 1995)
$T_{\min} = 0.763$, $T_{\max} = 0.976$
11 139 measured reflections
4103 independent reflections

2578 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 26.27°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 22$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.125$
$S = 1.065$
4103 reflections
265 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0380P)^2 + 0.6352P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.000$
$\Delta\rho_{\max} = 0.358$ e Å^{-3}
$\Delta\rho_{\min} = -0.357$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.05 (4)

Table 2. *Selected bond lengths (Å) for (2)*

O4—C7	1.416 (6)	C1—C2	1.550 (6)
O4—C4	1.434 (6)	C2—C3	1.528 (7)
O5—C1	1.442 (6)	C3—C4	1.526 (7)
O5—C5	1.438 (5)	C4—C5	1.520 (7)
O6—C7	1.420 (6)	C5—C6	1.516 (6)
O6—C6	1.455 (6)	C7—C11	1.515 (7)

Compound (3)

Crystal data

$[Cr(C_{17}H_{24}O_6S)(CO)_3]$
$M_r = 492.45$
Monoclinic
$P2_1$
$a = 7.9722\ (2)$ Å
$b = 8.5924\ (2)$ Å
$c = 16.0785\ (4)$ Å
$\beta = 92.720\ (1)°$
$V = 1100.14\ (5)$ Å^3
$Z = 2$
$D_x = 1.487$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5864 reflections
$\theta = 1.3–27.5°$
$\mu = 0.663$ mm^{-1}
$T = 203\ (2)$ K
Tablet
0.60 × 0.32 × 0.08 mm
Yellow

Data collection

Siemans SMART diffractometer

3037 reflections with $I > 2\sigma(I)$

Area-detector scans
Absorption correction:
empirical (Blessing, 1995)
$T_{\min} = 0.692$, $T_{\max} = 0.949$
7191 measured reflections
3309 independent reflections

$R_{\text{int}} = 0.029$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 4$
$l = 0 \rightarrow 20$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.103$
$S = 1.105$
3309 reflections
284 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.1822P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.268$ e Å^{-3}
$\Delta\rho_{\min} = -0.272$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.01 (2)

Table 3. *Selected bond lengths (Å) for (3)*

O4—C7	1.420 (3)	C1—C2	1.528 (4)
O4—C4	1.435 (3)	C2—C3	1.530 (4)
O5—C1	1.430 (4)	C3—C4	1.526 (4)
O5—C5	1.430 (3)	C4—C5	1.521 (4)
O6—C7	1.414 (3)	C5—C6	1.520 (4)
O6—C6	1.444 (4)	C7—C11	1.501 (4)

Compound (4)

Crystal data

$[\text{Cr}(\text{C}_{28}\text{H}_{31}\text{O}_6\text{P})(\text{CO})_3]\cdot\text{CH}_2\text{Cl}_2$
$M_r = 715.45$
Monoclinic
$C2$
$a = 22.3782$ (1) Å
$b = 11.4738$ (1) Å
$c = 13.8548$ (2) Å
$\beta = 108.007$ (1)°
$V = 3383.15$ (6) Å^3
$Z = 4$
$D_x = 1.405$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\theta = 2$–$27°$
$\mu = 0.594$ mm^{-1}
$T = 203$ (2) K
Irregular fragment
0.40 × 0.31 × 0.16 mm
Yellow

Data collection

Siemens SMART diffractometer
Area-detector scans
Absorption correction:
empirical (Blessing, 1995)
$T_{\min} = 0.797$, $T_{\max} = 0.911$
10 559 measured reflections
7055 independent reflections

6510 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.015$
$\theta_{\max} = 27.5°$
$h = -29 \rightarrow 27$
$k = -14 \rightarrow 14$
$l = 0 \rightarrow 17$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.045$
7055 reflections
409 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1022P)^2 + 0.3309P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.007$

$\Delta\rho_{\max} = 0.247$ e Å^{-3}
$\Delta\rho_{\min} = -0.283$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.038 (14)

Table 4. *Selected bond lengths (Å) for (4)*

O4—C7	1.422 (2)	C1—C2	1.524 (3)
O4—C4	1.437 (2)	C2—C3	1.539 (3)
O5—C1	1.420 (3)	C3—C4	1.515 (3)
O5—C5	1.432 (2)	C4—C5	1.529 (3)
O6—C7	1.408 (2)	C5—C6	1.505 (3)
O6—C6	1.440 (2)	C7—C11	1.525 (3)

Compound (5)

Crystal data

$[\text{Cr}(\text{C}_{17}\text{H}_{24}\text{O}_7)(\text{CO})_3]$
$M_r = 476.39$
Orthorhombic
$P2_12_12_1$
$a = 8.4820$ (2) Å
$b = 9.0429$ (3) Å
$c = 28.6178$ (8) Å
$V = 2195.04$ (11) Å^3
$Z = 4$
$D_x = 1.442$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6519 reflections
$\theta = 2$–$27°$
$\mu = 0.574$ mm^{-1}
$T = 203$ (2) K
Prism
0.57 × 0.36 × 0.25 mm
Yellow

Data collection

Siemens SMART diffractometer
Area-detector scans
Absorption correction:
empirical (Blessing, 1995)
$T_{\min} = 0.736$, $T_{\max} = 0.870$
14 172 measured reflections
4882 independent reflections

4676 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.016$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 37$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.072$
$S = 1.061$
4882 reflections
284 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.1050P]$
where $P = F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.275$ e Å^{-3}
$\Delta\rho_{\min} = -0.388$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.004 (12)

Table 5. *Selected bond lengths (Å) for (5)*

O4—C7	1.4131 (17)	C1—C2	1.539 (2)
O4—C4	1.4397 (19)	C2—C3	1.539 (2)
O5—C1	1.426 (2)	C3—C4	1.519 (2)
O5—C5	1.4351 (19)	C4—C5	1.523 (2)
O6—C7	1.4167 (18)	C5—C6	1.519 (2)
O6—C6	1.438 (2)	C7—C11	1.511 (2)

Each data collection covered a nominal hemisphere of reciprocal space by a series of ω scans. Crystal decay was monitored by repeating the initial frames at the end of the collection and analysing duplicate reflections; decay was negligible. The diphenylphosphine derivative, (4), contains a dichloromethane solvent molecule. H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds for methyl groups) and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

For all compounds, data collection: *SMART* (Siemens, 1994*b*); cell refinement: *SAINT* (Siemens, 1994*b*); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994*a*); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1212). Services for accessing these data are described at the back of the journal.

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Bis(tetraethylammonium) Bis[2-(trifluoroacetylamidato)benzenethiolato(2–)-*S*,*N*]-nickelate(II)

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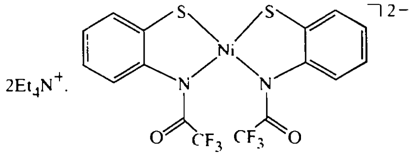
Abstract

The title compound, bis(tetraethylammonium) bis[*N*-(2-mercaptophenyl)trifluoroacetamidato(2−)-*N*,*S*]-nickelate(II), $(C_8H_{20}N)_2[Ni(C_8H_4F_3NOS)_2]$, consists of a distorted square-planar NiS_2N_2 core composed of two thiolate S and two deprotonated amide N atoms. The Ni—S bond distances are 2.177 (1) and 2.171 (1) Å, and the Ni—N bond distances are 1.954 (3) and 1.959 (3) Å.

Comment

Previously, we have reported deprotonation of the highly acidic CF_3CONH amide in the Co^{II}–thiolate complex, $(PPh_4)_2[Co\{S\text{-}2,6\text{-}(CF_3CONH)_2C_6H_3\}_4]$, resulting in *S*,*N*-chelate formation (Okamura *et al.*, 1998). The deprotonation was carried out by either air or amine *N*-oxide oxidation, producing a disulfide. On the other hand, numerous Ni^{II} complexes having an *S*(thiolato),*N*(amidate)-five-membered chelate ring have been synthesized using strong bases (Krüger *et al.*, 1991). Numerous related *S*,*N*-chelate Ni^{II} complexes with a thiolate-containing Schiff base have also been reported (Das & Livingstone, 1976; Ercan *et al.*, 1996). In this paper, we report the structure of the title Ni^{II} complex, (I).



(I)

The title complex exhibits a distorted square-planar Ni^{II} ion, with S1—Ni—N2 and S2—Ni—N1 angles of 169.04 (10) and 169.09 (9)°, respectively, as shown in Fig. 1. The S1—Ni—S2 angle of 87.84 (5)° indicates *cis* coordination of the two thiolate ligands. Atoms N1 and S1, or N1 and S2, are in the plane of their respective aromatic ring. These bond distances and angles are similar to those of the neutral Schiff base Ni^{II} complex, $[Ni^{II}(C_{13}H_9ClNS)_2]$ (Ercan *et al.*, 1996).

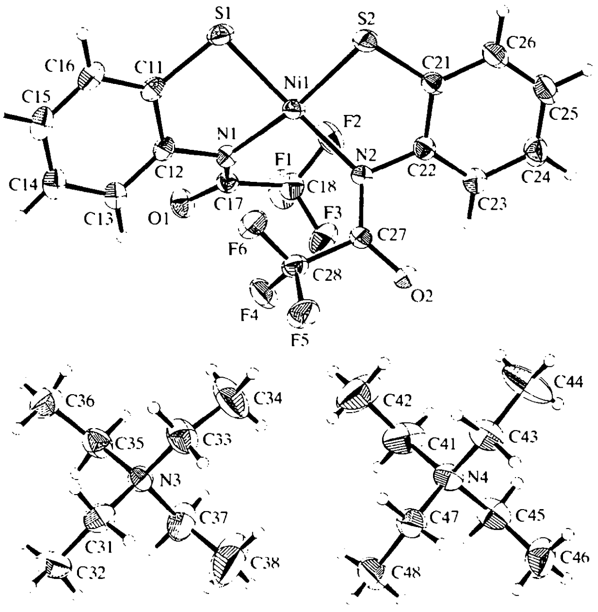


Fig. 1. A view of (I), with displacement ellipsoids drawn at the 25% probability level.