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# [(*R*,*R*)-2-Amino-1-(*p*-tolylsulfonylamido)cyclohexane-$\kappa^2N,N'$]chloro($\eta^5$-pentamethylcyclopentadienyl)iridium(III) chloroform solvate

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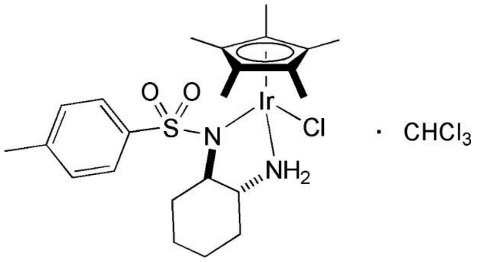
The enantiomerically pure iridium title complex, $[Ir(C_{13}H_{19}N_2O_2S)(C_{10}H_{15})Cl]\cdot CHCl_3$, has the Ir atom in the *S* configuration, with a distorted octahedral geometry.

## Comment

Recently, we reported an asymmetric transfer hydrogenation reaction in aqueous solution using $\eta^6$-arene ruthenium *N*-tosyl-*trans*-1,2-diaminocyclohexane complexes as catalysts (Canivet *et al.*, 2005). In order to extend our studies to iridium complexes, we synthesized the [Cp*IrCl{(*R*,*R*)-*L*}] complex, (I) [Cp* = pentamethylcyclopentadienyl and *L*H = (*R*,*R*)-*N*-(*p*-toluenesulfonyl)-1,2-cyclohexanediamine], which crystallized from chloroform to give a polymorph of the known complex described by Murata *et al.* (1999). The bond distances and angles are similar to those found in the parent compound [Cp*IrCl{(*R*,*R*)-*L*}] (Murata *et al.*, 1999), the rhodium analogue [Cp*RhCl{(*R*,*R*)-*L*}] (Murata *et al.*, 1999) and the nitromethyl derivative [Cp*Ir(nitromethyl){(*R*,*R*)-*L*}] (Murata *et al.*, 2002).



(I)

The single-crystal X-ray structure analysis of (I) reveals a distorted octahedral geometry for the Ir atom with Cp*, amino, sulfonamido and chloro ligands. Upon formation of complex (I) the iridium metal center becomes chiral. The iridium possesses the *S* configuration as observed in the ruthenium analogue [($\eta^6$-mesitylene)RuCl{(*R*,*R*)-*L'*}] {*L'*H = (*R*,*R*)-*N*-(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine} (Uematsu *et al.*, 1996) and [Cp*IrCl{(*R*,*R*)-*L*}].

In contrast with the parent compound which was crystallized from water, (I) was obtained in the presence of chloroform, thus giving rise to a completely different crystal packing. Indeed, two intermolecular hydrogen-bonded systems are observed: an (amino) N—H···O—S (sulfonamido) hydrogen bonding between two symmetry-related molecules and a (chloro) Ir—Cl···H—C (chloroform) hydrogen bonding between complex (I) and the solvent molecule (see Table 1).

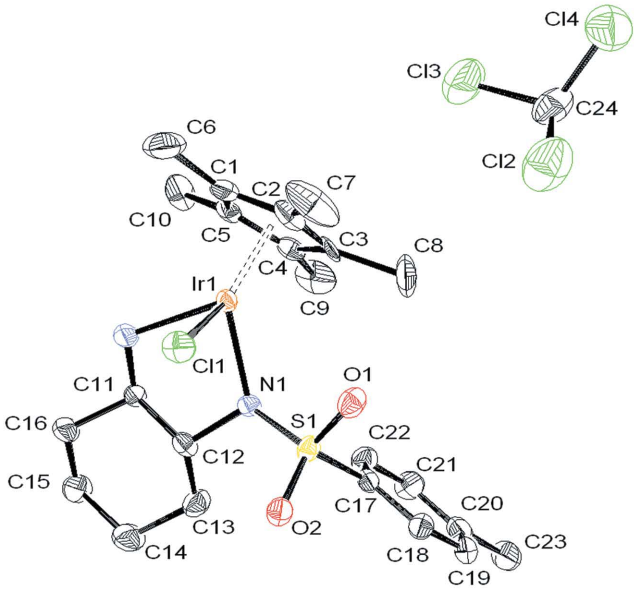


**Figure 1**
The asymmetric unit of (I), showing 50% probability displacement ellipsoids (H atoms omitted for clarity).

## Experimental

[Cp*IrCl{(*R*,*R*)-*L*}] was synthesized according to the published method (Murata *et al.*, 1999). The complex was dissolved in chloroform at room temperature. Yellow crystals of (I) suitable for X-ray diffraction analysis were obtained after three days by slow evaporation of the solvent.

### *Crystal data*

$[Ir(C_{13}H_{19}N_2O_2S)(C_{10}H_{15})Cl]\cdot CHCl_3$
$M_r = 749.60$
Orthorhombic, $P2_12_12_1$
$a = 9.4012$ (5) Å
$b = 12.8491$ (7) Å
$c = 23.7478$ (17) Å
$V = 2868.7$ (3) Å$^3$

$Z = 4$
$D_x = 1.736$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 5.12$ mm$^{-1}$
$T = 173$ (2) K
Needle, yellow
0.45 × 0.15 × 0.08 mm

### *Data collection*

Stoe IPDS diffractometer
$\varphi$ scans
Absorption correction: multi-scan (*MULABS* in *PLATON*; Spek, 2003)
$T_{min} = 0.360$, $T_{max} = 0.557$ (expected range = 0.428–0.664)

15012 measured reflections
5576 independent reflections
4830 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 25.9°$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.059$
$S = 0.94$
5576 reflections
313 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.53$ e Å$^{-3}$
$\Delta\rho_{min} = -1.82$ e Å$^{-3}$
Absolute structure: Flack (1983), 2413 Friedel pairs
Flack parameter: 0.024 (7)

**Table 1**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C24—H24*A*···Cl1 | 1.00 | 2.36 | 3.357 (8) | 178 |
| N2—H2*A*···Cl1 | 0.92 | 2.60 | 2.996 (5) | 106 |
| N2—H2*B*···O2$^{i}$ | 0.92 | 2.26 | 3.063 (7) | 145 |

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

All H atoms were included in calculated positions (C—H = 0.95 Å for CH$_{arom}$, 1.00 Å for CH, 0.99 Å for CH$_2$ and 0.98 Å for CH$_3$; N—H = 0.92 Å) and treated as riding atoms with the constraint $U_{iso}$(H) = 1.2$U_{eq}$(carrier) applied. The highest residual electron density and deepest hole are both located at less than 1.1 Å from the Ir atom.

Data collection: *EXPOSE* in *IPDS Software* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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