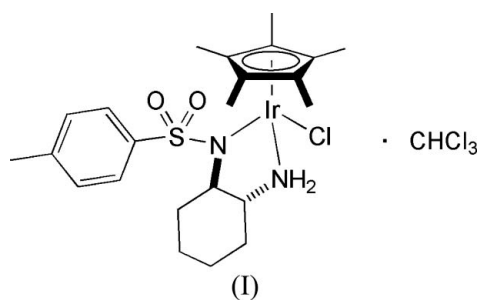


[(*R,R*)-2-Amino-1-(*p*-tolylsulfonylamido)cyclohexane- κ^2 *N,N'*]chloro(η^5 -pentamethylcyclopentadienyl)iridium(III) chloroform solvate**Jérôme Canivet, Bruno Therrien* and Georg Süss-Fink**Institut de Chimie, Université de Neuchâtel,
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SwitzerlandCorrespondence e-mail:
bruno.therrien@unine.ch**Key indicators**Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.028
 wR factor = 0.059
Data-to-parameter ratio = 17.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The enantiomerically pure iridium title complex, $[\text{Ir}(\text{C}_{13}\text{H}_{19}\text{N}_2\text{O}_2\text{S})(\text{C}_{10}\text{H}_{15})\text{Cl}]\cdot\text{CHCl}_3$, has the Ir atom in the *S* configuration, with a distorted octahedral geometry.Received 28 August 2006
Accepted 29 August 2006**Comment**

Recently, we reported an asymmetric transfer hydrogenation reaction in aqueous solution using η^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 $[\text{Cp}^*\text{IrCl}\{(R,R)\text{-}L\}]$ complex, (I) [Cp^* = pentamethylcyclopentadienyl and $LH = (R,R)\text{-}N$ -(*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 $[\text{Cp}^*\text{IrCl}\{(R,R)\text{-}L\}]$ (Murata *et al.*, 1999), the rhodium analogue $[\text{Cp}^*\text{RhCl}\{(R,R)\text{-}L\}]$ (Murata *et al.*, 1999) and the nitromethyl derivative $[\text{Cp}^*\text{Ir}(\text{nitromethyl})\{(R,R)\text{-}L\}]$ (Murata *et al.*, 2002).



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 $[(\eta^6\text{-mesitylene})\text{RuCl}\{(R,R)\text{-}L'\}]$ [$L'H = (R,R)\text{-}N$ -(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine] (Uematsu *et al.*, 1996) and $[\text{Cp}^*\text{IrCl}\{(R,R)\text{-}L\}]$.

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) $\text{N}-\text{H}\cdots\text{O}-\text{S}$ (sulfonamido) hydrogen bonding between two symmetry-related molecules and a (chloro) $\text{Ir}-\text{Cl}\cdots\text{H}-\text{C}$ (chloroform) hydrogen bonding between complex (I) and the solvent molecule (see Table 1).

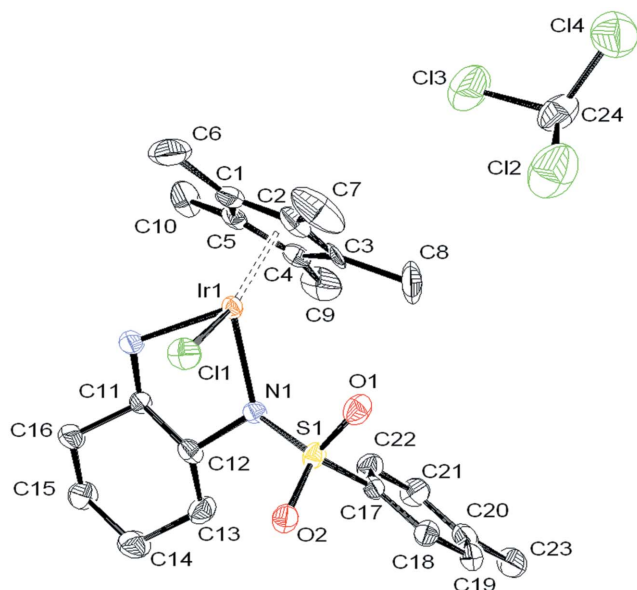


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids (H atoms omitted for clarity).

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 ₂ O ₂ S)(C ₁₀ H ₁₅)Cl]·CHCl ₃	Z = 4
<i>M_r</i> = 749.60	<i>D_x</i> = 1.736 Mg m ⁻³
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Mo <i>K</i> α radiation
<i>a</i> = 9.4012 (5) Å	<i>μ</i> = 5.12 mm ⁻¹
<i>b</i> = 12.8491 (7) Å	<i>T</i> = 173 (2) K
<i>c</i> = 23.7478 (17) Å	Needle, yellow
<i>V</i> = 2868.7 (3) Å ³	0.45 × 0.15 × 0.08 mm

Data collection

Stoe IPDS diffractometer	15012 measured reflections
<i>φ</i> scans	5576 independent reflections
Absorption correction: multi-scan (MULABS in PLATON; Spek, 2003)	4830 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.360, <i>T</i> _{max} = 0.557 (expected range = 0.428–0.664)	<i>R</i> _{int} = 0.044
	<i>θ</i> _{max} = 25.9°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*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 $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.002
 $\Delta\rho_{\text{max}} = 1.53 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -1.82 \text{ e } \text{Å}^{-3}$
 Absolute structure: Flack (1983),
 2413 Friedel pairs
 Flack parameter: 0.024 (7)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C24—H24A···Cl1	1.00	2.36	3.357 (8)	178
N2—H2A···Cl1	0.92	2.60	2.996 (5)	106
N2—H2B···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 CH₂ and 0.98 Å for CH₃; 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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References

- Canivet, J., Labat, G., Stoeckli-Evans, H. & Süss-Fink, G. (2005). *Eur. J. Inorg. Chem.* pp. 4493–4500.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Murata, K., Ikariya, T. & Noyori, S. (1999). *J. Org. Chem.* **64**, 2186–2187.
- Murata, K., Konishi, H., Ito, M. & Ikariya, T. (2002). *Organometallics*, **21**, 253–255.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (2000). *IPDS Software*. Stoe & Cie GmbH, Darmstadt, Germany.
- Uematsu, N., Fujii, A., Hashigushi, S., Ikariya, T. & Noyori, R. (1996). *J. Am. Chem. Soc.* **118**, 4916–4917.