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mer-Trichloro(dimethyl sulfoxide-κS)-*cis*-bis(1-methylimidazole-κN^3)iridium(III)

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Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.007 Å
R factor = 0.023
wR factor = 0.048
Data-to-parameter ratio = 20.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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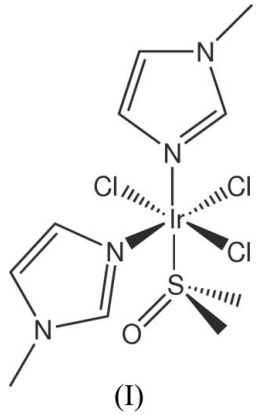
The title compound, $[IrCl_3(C_4H_6N_2)_2(C_2H_6OS)]$, has been synthesized and structurally characterized. The DMSO ligand is S-bonded and is *trans* to one and *cis* to the other 1-methylimidazole ligand. The two 1-methylimidazole ligands are *cis* to each other with an N···N···N···N pseudo-torsion angle between the planes of the two 1-methylimidazoles of 141.9 (5) °.

Comment

Today ruthenium(III) complexes are of great interest for their potential clinical applications as antitumour and antimetastatic agents (Clarke *et al.*, 1999). The [ImH][$RuCl_4$(DMSO)(Im)] complex (Im = imidazole and DMSO = dimethyl sulfoxide) NAMI-A is presently undergoing phase I clinical trials (Messori *et al.*, 2003, and references therein) because of its outstanding antimetastatic properties. Iridium(III) analogues are considered to be a possible alternative to antitumour ruthenium(III) complexes (Messori *et al.*, 2003), since it is well known that iridium(III) complexes are more stable than ruthenium(III) complexes (Cotton *et al.* 1994).



(I)

The title complex, (I), is constitutionally analogous to a ruthenium(III) compound published earlier (Geremia *et al.*, 1996), which crystallized with 0.5 eq of acetone solvent in the space group *P*2/*c*. Otherwise the two structures are quite similar. The Ir—N bond distances are a little shorter in the case of Ir [2.052 (3) and 2.072 (3) Å] than in the analogous Ru complex [2.097 (6) and 2.098 (5) Å]. The same applies for the metal–S bond length, which is 2.299 (2) Å in the Ru complex compared to 2.2477 (9) Å in the Ir complex. The M—Cl distances in both complexes are equal within the margin of error. The arrangement of the DMSO moleule in (I) is determined by four weak intramolecular interactions between the H atoms of the methyl groups of DMSO and the Cl atoms.

These H–Cl distances are H21*C*···Cl2 = 2.87 Å, H20*B*···Cl2 = 2.84 Å, H21*B*···Cl1 = 2.82 Å and H20*C*···Cl3 = 2.86 Å. The arrangement of the methylimidazoles is determined by intermolecular non-classical H-atom bridges between H1*A* of the equatorial imidazole and Cl1 (3.12 Å) and H11*A* of the axial imidazole and Cl2 (3.18 Å).

Experimental

0.5 mmol of $IrCl_3$ (0.5 mmol, 149.3 mg), 1,1′-dimethyl-3,3′-methylenebisimidazolium dichloride (1.5 mmol, 373.8 mg) and $NaOAc·3H_2O$ (3.0 mmol) were suspended in DMSO (5 ml) and heated for 2 h to 313 K, 1 h to 333 K, 1 h to 343 K, 2 h to 353 K and finally 2 h to 373 K. After the reaction was complete, the solvent was removed *in vacuo* at 343 K and the resulting product was washed twice with 5 ml of ethanol. The product was obtained as an off-white solid in 78.4% yield (212.0 mg). Crystals for the solid-state structure determination were obtained by slowly cooling a hot saturated solution of the complex in DMSO.

Crystal data

$[IrCl_3(C_4H_6N_2)_2(C_2H_6OS)]$
M_r = 540.92
Monoclinic, $C2/c$
a = 30.741 (1) Å
b = 8.614 (1) Å
c = 13.667 (1) Å
β = 113.90 (1)°
V = 3308.7 (5) Å^3
Z = 8
D_x = 2.172 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 8.68 mm^{-1}
T = 183 (2) K
Plate, colourless
0.46 × 0.11 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.109, T_{max} = 0.701
28714 measured reflections
3734 independent reflections
3155 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 27.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.048
S = 1.04
3734 reflections
185 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0127P)^2 + 12.7531P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −1.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ir1—Cl1	2.3676 (9)	Ir1—N2	2.052 (3)
Ir1—Cl2	2.3683 (10)	Ir1—N4	2.072 (3)
Ir1—Cl3	2.3434 (10)	Ir1—S1	2.2477 (9)
N2—Ir1—N4	88.59 (13)	N4—Ir1—Cl3	87.60 (10)
N2—Ir1—S1	92.33 (9)	S1—Ir1—Cl3	90.81 (4)
N4—Ir1—S1	178.18 (10)	S1—Ir1—Cl1	92.77 (3)
N2—Ir1—Cl1	88.57 (9)	S1—Ir1—Cl2	90.03 (3)
N4—Ir1—Cl1	88.83 (9)	Cl3—Ir1—Cl1	176.34 (3)
N2—Ir1—Cl2	177.05 (9)	Cl1—Ir1—Cl2	89.54 (3)
N4—Ir1—Cl2	89.10 (10)	Cl3—Ir1—Cl2	91.20 (4)
N2—Ir1—Cl3	90.54 (9)	O1—S1—Ir1	117.40 (14)

H atoms were positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.95 Å and methyl C—H distances of 0.98 Å, U_{iso}(H) = 1.2U_{eq}(aromatic C) or 1.5U_{eq}(methyl C). The highest peak is located 1.40 Å from atom Ir1 and the deepest hole is located 0.89 Å also from atom Ir1.

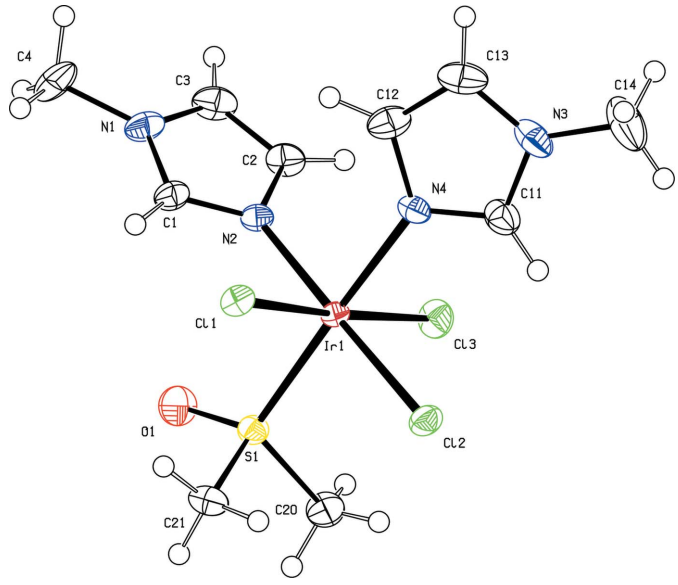


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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