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(1,2-Dicyanoethylenedithiolato-κ^2S,S')(η^5-pentamethylcyclopentadienyl)iridium(III)

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The crystal structure of the title complex, [Ir($C_{10}H_{15}$)($C_4N_2S_2$)], is composed of distinct [η^5-Cp*Ir(mnt)] (Cp* is pentamethylcyclopentadienyl and mnt is 1,2-dicyanoethylenedithiolate) molecules separated by normal van der Waals contacts. The asymmetric unit consists of two molecules. The Ir^{III} atom may be considered pentacoordinate since it is linked to a bidentate dithiolate ligand and a formally tridentate η^5-Cp* ligand. Ir—S distances range from 2.238 (3) to 2.246 (3) Å.

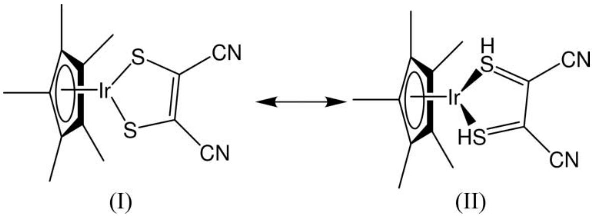
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.013 Å
R factor = 0.041
wR factor = 0.096
Data-to-parameter ratio = 19.2

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

Comment

The discrete electronically neutral monuclear title iridium(III) complex, (I), crystallizes in the triclinic space group $P\overline{1}$, with two molecules in the asymmetric unit. The five-membered metalladithiolene rings are almost planar and are almost perpendicular to the Cp* rings (Cp* is pentamethylcyclopentadienyl). The Ir—S bond distances range from 2.238 (3) to 2.246 (3) Å, which compares well with the values for other iridium complexes (Nomura *et al.*, 2005; Megehee *et al.*, 1989). The S—Ir—S angles are 88.50 (9) and 88.66 (8)°, which are close to 90°. In addition, the C—C bond lengths of the chelating dithiolene ligands are slightly shorter than the corresponding values in the uncomplexed dithiolene (Bradley *et al.*, 1994). Such results suggest that the contribution of the dithioolefinic structure becomes important and aromatic properties of dithiolene ring become small, due to the formation of a chelate structure (see scheme).



Experimental

All reactions were carried out under a nitrogen atmosphere by means of standard Schlenk techniques. All solvents for reactions were purified by Na-benzophenone or CaH_2 before use. The precursors [Cp*IrCl(η-Cl)]$_2$ (White *et al.*, 1992) and sodium maleonitriledithiolate (Na_2mnt) (Davison *et al.*, 1967) were synthesized by literature methods. To a solution of [Cp*IrCl$_2$]$_2$ (0.1 mmol, 80 mg) in CH_2Cl_2H (10 ml) was added Na_2mnt (0.2 mmol, 37 mg). The mixture was stirred for 1 h at room temperature. After the solvent had been removed under reduced pressure, the residue was separated by column chromatography (silica gel, eluent dichloromethane). The organic residue was purified by recrystallization from hexane/dichloromethane (1:1). The title complex was formed in 85% yield. Elemental analysis, found: C 35.92, H 3.25, N 6.03, S 13.74%; calculated for $C_{28}H_{30}Ir_2N_4S_4$: C 35.96, H 3.23, N 5.99, S 13.71%.

Crystal data

$[Ir(C_{10}H_{15})(C_4N_2S_2)]$
$M_r = 467.60$
Triclinic, $P\overline{1}$
$a = 8.891$ (9) Å
$b = 13.090$ (13) Å
$c = 14.548$ (14) Å
$\alpha = 98.347$ (13)°
$\beta = 97.794$ (12)°
$\gamma = 106.131$ (11)°
$V = 1581$ (3) Å^3
$Z = 4$
$D_x = 1.964$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 8.69$ mm^{-1}
$T = 298$ (2) K
Block, red
0.15 × 0.14 × 0.13 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.356$, $T_{\max} = 0.398$ (expected range = 0.289–0.323)
6598 measured reflections
6598 independent reflections
4925 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.096$
$S = 0.97$
6598 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (13.7468P)^2 + 14.5636P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.41$ e Å^{-3}
$\Delta\rho_{\min} = -2.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ir1—S2	2.238 (3)	S3—C26	1.733 (8)
Ir1—S1	2.241 (3)	S4—C25	1.710 (9)
Ir2—S4	2.246 (3)	C3—C4	1.437 (11)
Ir2—S3	2.246 (3)	C11—C13	1.391 (11)
S1—C11	1.713 (8)	C25—C26	1.372 (12)
S2—C13	1.724 (8)		
S2—Ir1—S1	88.50 (9)	C12—C11—S1	118.0 (7)
S4—Ir2—S3	88.66 (8)	C11—C13—S2	119.8 (6)
C11—S1—Ir1	105.4 (3)	C14—C13—S2	117.8 (6)
C13—S2—Ir1	105.5 (3)	C26—C25—S4	121.7 (6)
C26—S3—Ir2	104.8 (3)	C27—C25—S4	117.5 (7)
C25—S4—Ir2	104.8 (3)	C25—C26—S3	120.0 (6)
C13—C11—S1	120.7 (6)	C28—C26—S3	116.7 (7)

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.96 Å and $U_{\rm iso}$(H) = 0.15 Å^2. In the final difference map, the highest peak is 0.95 Å from atom Ir1 and the deepest hole is 0.97 Å from Ir2.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

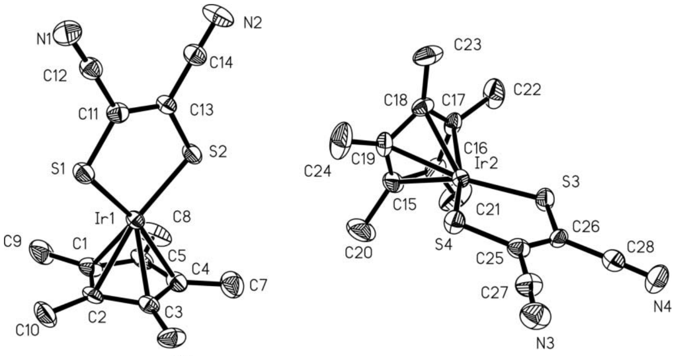


Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity

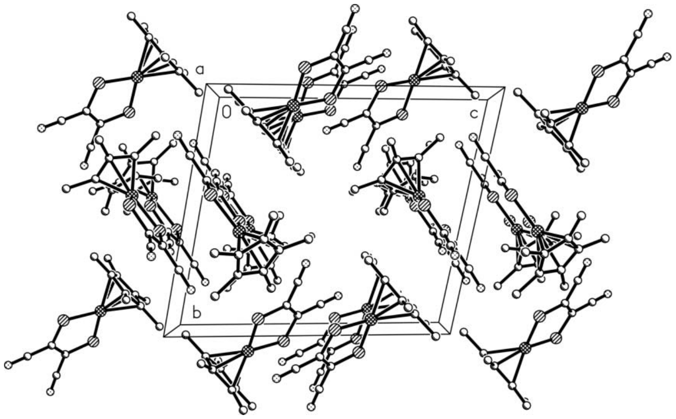


Figure 2
The molecular packing of the title complex. H atoms have been omitted for clarity.

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