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Bis(μ - η^2 -benzene-1,2-dithiolato- $\kappa^3 S, S': S'$)bis[(η^6 -*p*-cymene)-ruthenium(II)]

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Bis(μ - η^2 -benzene-1,2-dithiolato- $\kappa^3S,S':S'$)-bis[(η^6 -*p*-cymene)ruthenium(II)]Javier A. Cabeza,^a Santiago García-Granda,^{b*} Monica Pérez-Priede^b and Juan Francisco Van der Maelen^b^aDepartamento de Química Orgánica e Inorgánica, Universidad de Oviedo, Avda. Julián Clavería 8, 33006 Oviedo, Spain, and^bDepartamento de Química Física y Analítica, Universidad de Oviedo, Avda. Julián Clavería 8, 33006 Oviedo, Spain

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(C-C) = 0.010$ Å
 R factor = 0.041
 wR factor = 0.105
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In solution, the complex $[Ru(bdt)(cym)]$ and its dimer $[Ru_2(bdt)_2(cym)_2]$ [bdt is benzene-1,2-dithiolate ($C_6H_4S_2$) and cym is *p*-cymene ($C_{10}H_{14}$)] are in equilibrium in a ratio which depends on temperature (*ca* 1:1 at 298 K). In the solid state and in solution at low temperature (213 K), only the dimer is observed. In the crystal structure of $[Ru_2(bdt)_2(cym)_2]$, each Ru atom is bonded to three S atoms from two bdt ligands apart from its coordination to the benzene ring of the *p*-cymene ligand.

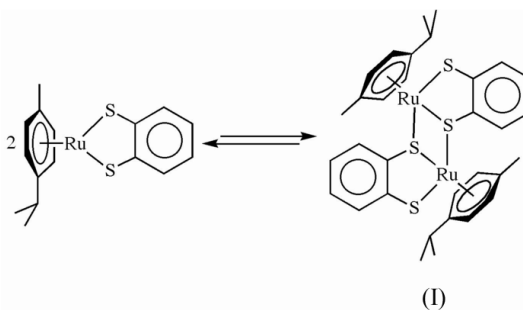
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Comment

This work is part of a project dealing with the synthesis and reactivity of binuclear complexes containing bridging benzene-1,2-dithiolate ligands (Cabeza *et al.*, 1998, 1999). The nuclearity of the $[Ru(bdt)(arene)]_x$ compounds (bdt is benzene-1,2-dithiolate) is strongly dependent on temperature and the nature of the substituents of the arene ring, *e.g.* $[Ru(bdt)(C_6Me_6)]$ is a monomer in the solid state and in solution at any temperature, while $[Ru(bdt)(C_6H_6)]$ and $[Ru(bdt)(cym)]$ exist in solution in equilibrium with their dimers (Mashima *et al.*, 1997). With the aim of studying its reactivity with other metal complexes, $[Ru(bdt)(cym)]_x$ (cym is *p*-cymene) was prepared. In solution at 298 K an approximate 1:1 equilibrium mixture of the monomer and the dimer exists (see *Scheme* below); however, at temperatures above 298 K the monomer predominates (a 4:1 ratio was observed at 353 K), while at lower temperatures the dimer is in a higher ratio, *e.g.* at 213 K only the dimer was observed. In the solid state, only the dimer is found. We report here the structural details of the dimer $[Ru_2(bdt)_2(cym)_2]$, (I).



The dimer crystallizes in $P\bar{1}$. The asymmetric unit consists of one-half of the dimer, *i.e.* the monomeric unit, $[Ru(bdt)(cym)]$, with the other half being generated by a centre of symmetry at (0,1/2,1/2). The monomeric units are linked through two Ru–S bonds, Ru1–S1ⁱ [2.364 (2) Å;

symmetry code: $-x, 1-y, 1-z$] and Ru1–S2ⁱ [2.386 (2) Å]. Thus, each Ru atom is bonded to three S atoms (S1, S1ⁱ and S2ⁱ) from two bdt ligands apart from its coordination to the benzene ring of the *p*-cymene ligand.

Although other related compounds, such as [Ru₂(bdt)₂(C₅Me₅)₂] (Ru–Ru) (Hörnig *et al.*, 1994) and [Rh₂(bdt)₂(C₅Me₅)₂] (Russell *et al.*, 1978), are known, structural details have not been reported.

Experimental

The title compound was prepared by the method reported by Mashima *et al.* (1997). The monomer to dimer ratios in solution in toluene-*d*₈ at different temperatures were estimated by integration of ¹H NMR spectra. The ¹H NMR chemical shifts of monomer and dimer have been reported (Mashima *et al.*, 1997). Crystals were obtained at 253 K by slow diffusion of pentane vapour into a solution of the complex in dichloromethane.

Crystal data

[Ru ₂ (C ₁₀ H ₁₄) ₂ (C ₆ H ₄ S ₂) ₂]	Z = 1
<i>M_r</i> = 750.98	<i>D_x</i> = 1.706 Mg m ⁻³
Triclinic, P $\bar{1}$	Mo K α radiation
<i>a</i> = 8.237 (5) Å	Cell parameters from 25 reflections
<i>b</i> = 9.813 (6) Å	θ = 10–15°
<i>c</i> = 10.237 (3) Å	μ = 1.34 mm ⁻¹
α = 65.42 (4)°	<i>T</i> = 293 (2) K
β = 82.57 (3)°	Plate, dark red
γ = 76.34 (6)°	0.33 × 0.17 × 0.03 mm
<i>V</i> = 730.8 (7) Å ³	

Data collection

Nonius CAD-4 diffractometer	θ_{\max} = 26.0°
ω -2 θ scans	<i>h</i> = -10 → 10
Absorption correction: refined from ΔF (SHELXA; Sheldrick, 1997)	<i>k</i> = -10 → 12
<i>T</i> _{min} = 0.762, <i>T</i> _{max} = 0.957	<i>l</i> = 0 → 12
3022 measured reflections	3 standard reflections every 200 reflections
2855 independent reflections	frequency: 60 min
1907 reflections with <i>I</i> > 2 σ (<i>I</i>)	intensity decay: 3.6%
<i>R</i> _{int} = 0.036	

Refinement

Refinement on <i>F</i> ²	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
<i>S</i> = 1.02	(Δ/σ) _{max} < 0.001
2855 reflections	$\Delta\rho_{\max} = 0.85 \text{ e } \text{Å}^{-3}$
175 parameters	$\Delta\rho_{\min} = -0.69 \text{ e } \text{Å}^{-3}$

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CRYSDA (Beurskens *et al.*, 1992); data reduction: REFLEX (García-Granda *et al.*, 1999); program(s) used to solve structure: DIRDIF (Beurskens *et al.*, 1992); program(s) used to refine

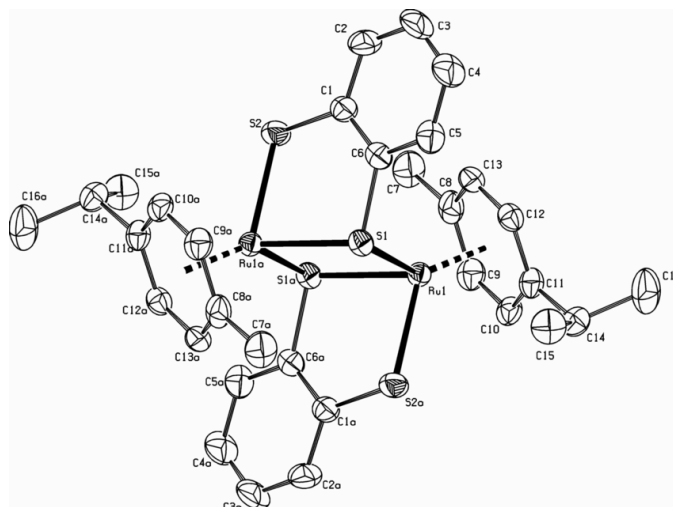


Figure 1

View of [Ru₂(bdt)₂(cym)₂]. Displacement ellipsoids are shown at the 30% probability level. H atoms have been omitted for clarity.

structure: SHELXL97 (Sheldrick, 1997); molecular graphics: EUCLID (Spek, 1982); software used to prepare material for publication: SHELXL97.

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