### *Refinement*

Refinement on $F^2$
Final $R = 0.033$
$wR = 0.041$
$S = 1.68$
1487 reflections
138 parameters
All H-atom parameters refined

$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\max} = 0.05$
$\Delta\rho_{\max} = 0.60$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.69$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $B_{\rm eq}$ |
|---|---|---|---|---|
| Cu(1) | 0.0 | 0.0 | 0.0 | 1.68 (1) |
| O(1) | 0.4383 (3) | 0.7143 (2) | 0.9298 (3) | 2.86 (6) |
| O(2) | 0.2980 (4) | 0.7944 (2) | 0.6267 (3) | 2.87 (6) |
| O(3) | 0.2043 (3) | 0.0413 (2) | −0.1530 (3) | 2.04 (5) |
| O(4) | 0.7320 (4) | 0.0943 (3) | 0.7552 (4) | 3.21 (7) |
| N(4) | 0.1253 (3) | 0.2183 (2) | 0.2323 (3) | 1.67 (5) |
| C(1) | 0.2719 (4) | 0.5246 (3) | 0.5465 (4) | 1.59 (6) |
| C(2) | 0.1974 (4) | 0.4940 (3) | 0.3050 (4) | 1.91 (7) |
| C(3) | 0.1277 (4) | 0.3412 (3) | 0.1545 (4) | 1.92 (7) |
| C(5) | 0.2012 (4) | 0.2483 (3) | 0.4664 (4) | 2.10 (7) |
| C(6) | 0.2769 (4) | 0.3980 (3) | 0.6266 (4) | 1.96 (7) |
| C(7) | 0.3431 (4) | 0.6915 (3) | 0.7157 (4) | 1.94 (7) |

Table 2. *Geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Cu(1)—O(3) | 1.985 (2) | C(1)—C(2) | 1.386 (3) |
| Cu(1)—N(4) | 2.004 (2) | C(1)—C(6) | 1.386 (3) |
| O(1)—C(7) | 1.251 (3) | C(1)—C(7) | 1.516 (3) |
| O(2)—C(7) | 1.255 (3) | C(2)—C(3) | 1.377 (3) |
| N(4)—C(3) | 1.345 (3) | C(5)—C(6) | 1.376 (3) |
| N(4)—C(5) | 1.343 (3) | | |
| O(3)—Cu(1)—O(3) | 180.00 | C(6)—C(1)—C(7) | 121.6 (2) |
| O(3)—Cu(1)—N(4) | 89.87 (8) | C(1)—C(2)—C(3) | 119.6 (2) |
| N(4)—Cu(1)—N(4) | 180.00 | N(4)—C(3)—C(2) | 122.5 (2) |
| Cu(1)—N(4)—C(3) | 119.6 (2) | N(4)—C(5)—C(6) | 122.7 (2) |
| Cu(1)—N(4)—C(5) | 122.6 (2) | C(1)—C(6)—C(5) | 119.5 (2) |
| C(3)—N(4)—C(5) | 117.8 (2) | O(1)—C(7)—O(2) | 125.9 (2) |
| C(2)—C(1)—C(6) | 117.8 (2) | O(1)—C(7)—C(1) | 117.7 (2) |
| C(2)—C(1)—C(7) | 120.6 (2) | O(2)—C(7)—C(1) | 116.4 (2) |

Data collection, cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). The scan rate was 32° min$^{-1}$ in $\omega$ and the scan width was $(1.68 + 0.30\tan\theta)°$. The ratio of peak counting time to background counting time was 2:1. Programs used to solve structure: *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987) and *DIRDIF* (Beuskens, 1984). All calculations including data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Refinement was by full-matrix least squares.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, bond distances and angles involving H atoms, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71216 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1018]

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*Acta Cryst.* (1993). C**49**, 1762–1764

# Structure of [Ru($\eta^6$-$C_7H_8$)(acetylacetonato)Cl]

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## Abstract

The structure of (acetylacetonato-*O*,*O*′)chloro($\eta^6$-cyclohepta-1,3,5-triene)ruthenium(II), [Ru($\eta^6$-$C_7H_8$)-{HC(COMe)$_2$}Cl], was determined. The Ru atom lies on a crystallographic mirror plane which is coincident with the molecular mirror plane. The geometry at the Ru atom is, as expected for ruthenium(II), approximately octahedral, with O—Ru—Cl and O—Ru—O bond angles of 84.1 (1) and 90.4 (1)°, respectively.

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## Comment

The complex was prepared by reacting [{Ru($\eta^6$-$C_7H_8$)($\mu$-Cl)Cl}$_2$] (Winkhaus & Singer, 1967; Müller, Kreiter, Mertschenk & Schmitt, 1975) with Na[acac] (acac = acetylacetonate) at room temperature in dichloromethane. It is reported that the same complex is formed when Tl[acac] is used instead of Na[acac] (Johnson, Lewis & Ryder, 1977). Diffraction-quality crystals of [Ru($\eta^6$-$C_7H_8$)(acac)Cl] were obtained by the slow evaporation of a dichloromethane solution of the complex at room temperature.

A survey of the literature suggests that [Ru($\eta^6$-$C_7H_8$)(acac)Cl] is the first example of a ruthenium complex containing an $\eta^6$-bonded cyclohepta-1,3,5-triene to be characterized by means of X-ray crystallography. The X-ray structure of a ruthenium dimer containing an $\eta^5$-bonded cycloheptadienyl and a bridging cycloheptatriene ligand has been reported (Bau *et al.*, 1973).

The planarity of the olefinic portion of the cycloheptatriene together with the average Ru—C(olefin) bond length of 2.208 (3) Å, indicates $\eta^6$-bonding of the carbocycle. The plane C(5)—C(4)—C(5′), containing the methylene C atom C(4), is bent away from the plane defined by the olefinic C atoms C(5)—C(6)—C(7) and C(7′)—C(6′)—C(5′) by 122.1 (4)°.

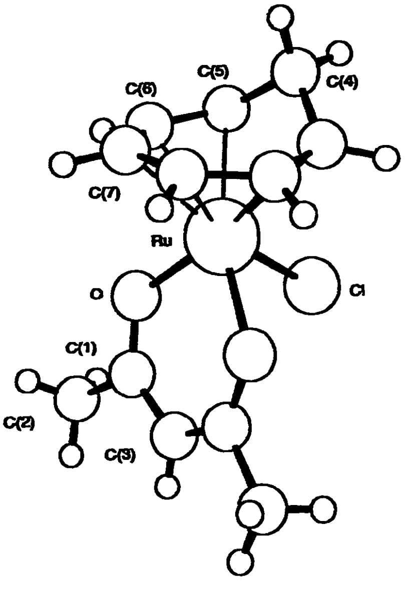

Fig. 1. *ORTEP* (Johnson, 1965) diagram of [Ru($\eta^6$-$C_7H_8$)(acac)Cl] showing the atom-numbering scheme.

## Experimental

### *Crystal data*

[RuCl($C_5H_7O_2$)($C_7H_8$)]
$M_r$ = 327.8
Orthorhombic
*Pnma*
$a$ = 13.080 (3) Å
$b$ = 11.667 (7) Å
$c$ = 7.8001 (1) Å
$V$ = 1195 (1) Å$^3$
$Z$ = 4
$D_x$ = 1.82 Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda$ = 0.7107 Å
Cell parameters from 25 reflections
$\theta$ = 7–18°
$\mu$ = 1.49 mm$^{-1}$
$T$ = 296 K
Needle
0.76 × 0.48 × 0.20 mm
Dark orange

### *Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{min}$ = 0.847, $T_{max}$ = 0.999
2039 measured reflections
1682 independent reflections

1555 observed reflections [$I > \sigma(I)$]
$\theta_{max}$ = 30°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 0.3%

### *Refinement*

Refinement on $F$
Final $R$ = 0.038
$wR$ = 0.025
1555 reflections
104 parameters
$w = 1/\sigma(F_o)^2$
$(\Delta/\sigma)_{max}$ = 0.004

$\Delta\rho_{max}$ = 0.86 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.52 e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å$^2$)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Ru | 0.0462 (1) | 0.2500 | 0.0486 (1) | 0.025 (1) |
| Cl | 0.0513 (1) | 0.2500 | −0.2594 (2) | 0.061 (1) |
| O | 0.1574 (2) | 0.3765 (2) | 0.0266 (3) | 0.035 (1) |
| C(1) | 0.2479 (2) | 0.3566 (3) | −0.0277 (4) | 0.032 (1) |
| C(2) | 0.3123 (3) | 0.4615 (3) | −0.0545 (6) | 0.046 (1) |
| C(3) | 0.2908 (3) | 0.2500 | −0.0616 (7) | 0.034 (1) |
| C(4) | −0.1727 (4) | 0.2500 | 0.0504 (7) | 0.042 (1) |
| C(5) | −0.1015 (2) | 0.1489 (3) | 0.0455 (5) | 0.039 (1) |
| C(6) | −0.0392 (3) | 0.1157 (3) | 0.1786 (4) | 0.038 (1) |
| C(7) | 0.0040 (3) | 0.1892 (3) | 0.3041 (4) | 0.038 (1) |

Table 2. *Selected bond lengths* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Ru—Cl | 2.403 (1) | C(1)—C(2) | 1.501 (4) |
| Ru—O | 2.079 (2) | C(1)—C(3) | 1.390 (4) |
| Ru—C(5) | 2.263 (3) | C(4)—C(5) | 1.503 (4) |
| Ru—C(6) | 2.175 (3) | C(5)—C(6) | 1.375 (5) |
| Ru—C(7) | 2.186 (3) | C(6)—C(7) | 1.419 (4) |
| C(1)—O | 1.278 (3) | C(7)—C(7′) | 1.418 (7) |
| O—Ru—Cl | 84.1 (1) | C(5)—C(4)—C(5′) | 103.3 (3) |
| O—Ru—O | 90.4 (1) | C(4)—C(5)—C(6) | 124.7 (4) |
| O—C(1)—C(2) | 114.7 (3) | C(5)—C(6)—C(7) | 125.9 (3) |
| C(2)—C(1)—C(3) | 118.5 (3) | | |

Data collection, data reduction and cell refinement: Enraf–Nonius (1989) *Structure Determination Package*. Program used to solve structure: *SHELX76* (Sheldrick, 1976). Data were collected in the $\omega/2\theta$ scan mode with an $\omega$-scan angle of (0.52 + 0.34tan$\theta$)° and a maximum scan time of 1 min. Lorentz–polarization corrections were made. The structure was solved by usual Patterson and Fourier methods, and the non-H atoms refined anisotropically by blocked-matrix least-squares methods (*SHELX76*). The H atoms were placed in experimentally determined positions obtained from the difference maps and refined with a common isotropic thermal parameter, $U_{\mathrm{iso}} = 0.053\ (4)$ Å$^2$.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71199 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1028]

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*Acta Cryst.* (1993). C**49**, 1764–1766

# Structure of ($\eta^5$-Cyclopentadienyl)[1-(2-quinoxalinyl)ethene-1,2-dithiolato]cobalt(III), $[(\eta^5\text{-}C_5H_5)Co\{S_2C_2H(2\text{-quinoxalinyl})\}]$

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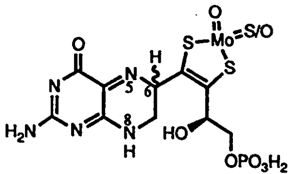
## Abstract

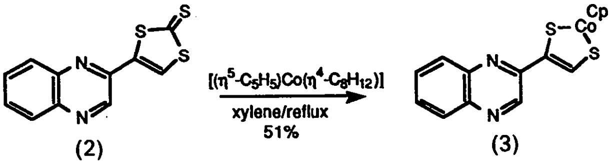
$[(\eta^5\text{-}C_5H_5)Co\{S_2C_2H(2\text{-quinoxalinyl})\}]$ has been prepared using a general procedure which allows for the synthesis of unsymmetrical dithiolenes; the molecular structure involves a planar cobalt–dithiolene five-membered ring which is disposed at an angle of 10.6° to the 2-quinoxalinyl group.

## Comment

(Cyclopentadienyl)(ene-1,2-dithiolato)cobalt complexes have been prepared in the past; $[(\eta^5\text{-}C_5H_5)Co\{S_2C_2(CF_3)_2\}]$ (Baird & White, 1966), $[(\eta^5\text{-}C_5H_5)Co\{S_2C_2(CN)_2\}]$ (Churchill & Fennessey, 1968) and $[(\eta^5\text{-}C_5H_5)Co(S_2C_6H_4)]$ (Miller, Brill, Rheingold & Fultz, 1983) have been characterized by X-ray crystallography. We have extended the investigation of these systems, partly because of our interest in the synthesis of dithiolenes, which resemble the partial structure of the ligand proposed (Gardlik & Rajagopalan, 1990) for the cofactor of the oxomolybdoenzymes, Moco, (1). The title compound, (3), was prepared by the reaction of $[(\eta^5\text{-}C_5H_5)Co(\eta^4\text{-}C_8H_{12})]$ with 4-(2-quinoxalinyl)-1,3-dithiole-2-thione, (2) (Siedle, 1976), and recrystallized from hexane.



(1)



(2) (3)

Cp = cyclopentadienyl

The atomic parameters of (3) are listed in Table 1; Table 2 lists selected bond lengths and bond angles; Fig. 1 shows an *ORTEP* (Johnson, 1965) drawing of the molecule and the numbering system used in the tables.

The dimensions of the $\{(\eta^5\text{-}C_5H_5)Co(S_2C_2)\}$ moiety are similar to those of each of the three structurally characterized molecules of this type

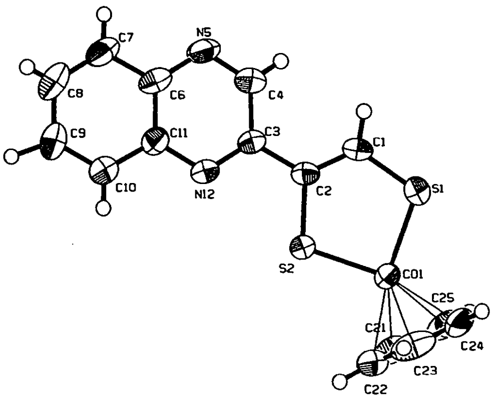


Fig. 1. *ORTEP* drawing of $[(\eta^5\text{-}C_5H_5)Co\{S_2C_2H(2\text{-quinoxalinyl})\}]$ showing the numbering scheme used in the tables.

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