N—Fe—C01	101.00 (9)	C1—C2—H2	122 (2)
N—Fe—C2	65.36 (8)	C3—C2—H2	125 (2)
N—Fe—C02	159.7 (1)	Fe—C02—002	176.6 (3)
N—Fe—C3	65.54 (7)	Fe—C3—C2	69.5 (1)
N—Fe—C03	96.6 (1)	Fe—C3—C4	94.2 (1)
C01—Fe—C2	103.6 (1)	Fe—C3—H3	126 (2)
C01—Fe—C02	94.2 (1)	C2—C3—C4	105.1 (2)
C01—Fe—C3	144.6 (1)	C2—C3—H3	129 (2)
C01—Fe—C03	106.3 (1)	C4—C3—H3	119 (2)
C2—Fe—C02	98.1 (1)	Fe—C03—003	179.0 (3)
C2—Fe—C3	41.10 (9)	N—C4—C3	93.4 (1)
C2—Fe—C03	147.6 (1)	N—C4—C5	101.9 (1)
C02—Fe—C3	94.3 (1)	N—C4—H4	114 (2)
C02—Fe—C03	92.0 (1)	C3—C4—H4	116 (2)
C3—Fe—C03	107.7 (1)	C5—C4—H4	118 (2)
C3—C4—C5	110.6 (2)	C4—C5—C6	107.2 (2)
Fe—N—C1	93.6 (1)	C4—C5—C51	122.1 (2)
Fe—N—C4	93.0 (1)	C6—C5—C51	130.5 (2)
Fe—N—C11	131.3 (1)	C1—C6—C5	106.6 (2)
C1—N—C4	93.9 (1)	C1—C6—C61	123.1 (2)
C1—N—C11	117.9 (2)	C5—C6—C61	130.3 (2)
C4—N—C11	118.5 (1)	N—C1—H1	114 (2)
N—C1—C2	93.1 (2)	C2—C1—H1	118 (2)
N—C1—C6	102.5 (1)	C6—C1—H1	116 (2)
C2—C1—C6	110.2 (2)	Fe—C2—C1	95.0 (1)
Fe—C2—H2	125 (2)	Fe—C01—001	175.4 (2)
C1—C2—C3	105.3 (2)	Fe—C2—C3	69.4 (1)

The title compound was synthesized by reacting $Fe_2(CO)_9$ with 7-azanorbornadiene in thf under N_2 overnight. During the reaction, three types of stable metal complex were isolated, *i.e.* $[Fe(C_{16}H_{14}ClNO_4)(CO)_4]$ and two structural isomers of $[Fe(C_{16}H_{14}ClNO_4)(CO)_3]$ (Sun, Wang & Yang, to be published). A similar reaction was observed during the study of other typical reactions of ANB derivatives with $Fe_2(CO)_9$ (Sun, Chow & Lui, 1990). Dark-red crystals of $[Fe(C_{16}H_{14}ClNO_4)(CO)_3]$ were grown from ethyl acetate/*n*-hexane mixed solvent. The structure was solved by direct methods using *Personal SDP* (Frenz, 1989). H atoms on the fused ring were located on difference maps and refined as normal atoms with fixed isotropic displacement parameters. Other H atoms were placed at idealized positions and included in the structure-factor calculations but not in the least-squares calculations. Four out of six phenyl-ring C atoms are disordered. The site occupancy for one ring is fixed at 0.75 and for the other is fixed at 0.25. The dihedral angle for these two rings is 54.8(4)°. Fig. 1 shows only the larger site occupancy for clarity. All calculations were carried out on an 80386-based IBM-compatible PC.

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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 55556 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1009]

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mer-Trichlorotris(2,3-dihydrobenzo[*b*]thiophene-*S*)rhodium(III)

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Abstract

The Rh atom has a distorted octahedral geometry. Two of the benzothiophenyl ligands occupy *trans* equatorial positions with Rh—S distances [2.351 (1) and 2.369 (1) Å] which are significantly longer than the third [2.330 (1) Å] distance. The Rh—Cl distances are in the range 2.333 (2)–2.359 (1) Å. The benzothiophenyl ligand at the axial position is inclined at almost right angles [mean-planes angles 89.6 (2) and 79.5 (2)°] to the other two ligands which lie almost parallel to each other [mean-planes angle 10.4 (2)°].

Comment

Organosulfur compounds coordinated to transition-metal species are of general interest as models for interactions which occur during the catalytic hydrodesulfurization of hydrocarbons (Dong, Duckett, Ohman & Jones, 1992). Industrially, this process is carried out on a large scale to produce sulfur-free transportation fuels. The title compound (I) is an example of a complex which may model species formed during the hydrodesulfurization of benzo[*b*]thiophene. Partial hydrogenation of benzothiophene is known to produce the 2,3-dihydro derivative, a compound which should readily coordinate to metal species. The rhodium complex of 2,3-dihydrobenzo[*b*]thiophene was prepared by heating

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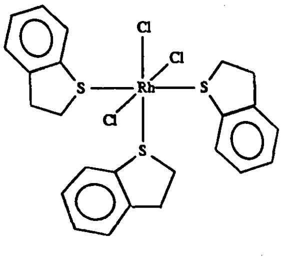
an ethanolic solution of the ligand with rhodium(III) chloride followed by recrystallization of the crude product from aqueous ethanol. Full details of the basic experimental procedure for the synthesis have been described elsewhere (Clark, Fait, Jones & Kirk, 1991).



(I)

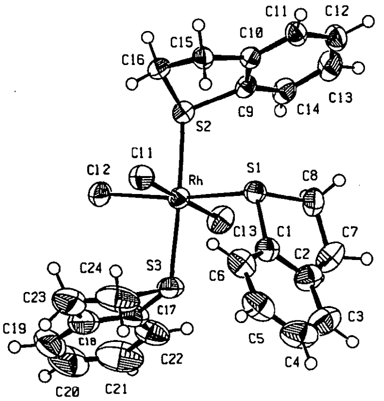


Fig. 1. Perspective view of the title compound with crystallographic numbering scheme plotted by *ORTEP*II (Johnson, 1976).

Experimental

Crystal data

$[Rh(C_8H_8S)_3Cl_3]$
$M_r = 617.91$
Monoclinic
$P2_1/c$
$a = 7.947$ (2) Å
$b = 31.447$ (3) Å
$c = 10.275$ (1) Å
$\beta = 109.57$ (1)°
$V = 2419.48$ Å^3
$Z = 4$
$D_x = 1.697$ Mg m^{-3}
$D_m = 1.65$ (4) Mg m^{-3}
Density measured by flotation
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–20°
$\mu = 1.24$ mm^{-1}
$T = 295$ K
Plate
0.65 × 0.20 × 0.14 mm
Orange–red

Data collection

Enraf–Nonius CAD-4 diffractometer
Variable speed $\omega/2\theta$ scans
4625 observed reflections [$I > 3\sigma(I)$]
$R_{\text{int}} = 0.014$
Absorption correction: *DIFABS* (Walker & Stuart, 1983)
$T_{\min} = 0.877$, $T_{\max} = 1.107$
7764 measured reflections
7029 independent reflections
$\theta_{\max} = 30°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 44$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
Final $R = 0.046$
$wR = 0.030$
$S = 2.377$
4625 reflections
280 parameters
H-atom parameters not refined
$w = 1/\sigma(F)$
$(\Delta/\sigma)_{\max} = 0.08$
$\Delta\rho_{\max} = 0.7$ e Å^{-3}
$\Delta\rho_{\min} = -1.1$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

$U_{\text{eq}} = 1/3$(trace of the orthogonalized U_{ij} tensor).

	x	y	z	U_{eq}
Rh	0.73128 (5)	0.14858 (1)	0.43449 (3)	0.0338 (2)
Cl(1)	0.4809 (2)	0.1720 (1)	0.4886 (1)	0.0429 (7)
Cl(2)	0.5560 (2)	0.1315 (1)	0.2057 (1)	0.0434 (6)
Cl(3)	0.9880 (2)	0.1258 (1)	0.3929 (1)	0.0465 (7)
S(1)	0.8891 (2)	0.1738 (1)	0.6549 (1)	0.0360 (6)
S(2)	0.7634 (2)	0.2130 (1)	0.3293 (1)	0.0324 (6)
S(3)	0.7033 (2)	0.0793 (1)	0.5150 (1)	0.0459 (7)
C(1)	0.8855 (7)	0.1364 (1)	0.7848 (4)	0.039 (2)
C(2)	1.0496 (7)	0.1181 (2)	0.8478 (5)	0.048 (3)
C(3)	1.0685 (9)	0.0910 (2)	0.9591 (6)	0.070 (4)
C(4)	0.9238 (10)	0.0840 (2)	1.0022 (6)	0.073 (4)
C(5)	0.7622 (9)	0.1020 (2)	0.9358 (6)	0.062 (4)
C(6)	0.7410 (7)	0.1283 (2)	0.8249 (5)	0.049 (3)
C(7)	1.1893 (7)	0.1297 (2)	0.7872 (5)	0.060 (3)
C(8)	1.1307 (6)	0.1692 (2)	0.7041 (5)	0.049 (3)
C(9)	0.9350 (5)	0.2453 (1)	0.4400 (4)	0.031 (2)
C(10)	0.8694 (6)	0.2763 (1)	0.5066 (4)	0.031 (2)
C(11)	0.9856 (6)	0.3050 (1)	0.5932 (4)	0.038 (3)
C(12)	1.1645 (7)	0.3025 (2)	0.6080 (5)	0.047 (3)
C(13)	1.2248 (6)	0.2721 (2)	0.5394 (5)	0.053 (3)
C(14)	1.1113 (6)	0.2425 (2)	0.4534 (5)	0.041 (3)
C(15)	0.6709 (6)	0.2737 (1)	0.4760 (4)	0.034 (2)
C(16)	0.5962 (6)	0.2517 (1)	0.3363 (4)	0.038 (2)
C(17)	0.6769 (8)	0.0427 (2)	0.3801 (5)	0.052 (3)
C(18)	0.5076 (9)	0.0317 (2)	0.3119 (6)	0.060 (3)
C(19)	0.4642 (10)	0.0032 (2)	0.2060 (6)	0.079 (4)
C(20)	0.5995 (13)	−0.0147 (2)	0.1712 (7)	0.099 (5)
C(21)	0.7739 (11)	−0.0046 (2)	0.2420 (8)	0.095 (5)
C(22)	0.8125 (8)	0.0253 (2)	0.3474 (6)	0.067 (4)
C(23)	0.3736 (8)	0.0536 (2)	0.3624 (7)	0.075 (4)
C(24)	0.4713 (9)	0.0689 (2)	0.5053 (7)	0.080 (4)

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

Rh—S(1)	2.330 (1)	C(5)—C(6)	1.372 (8)
Rh—S(2)	2.351 (1)	C(7)—C(8)	1.491 (7)
Rh—S(3)	2.369 (1)	C(9)—C(14)	1.364 (7)
Rh—Cl(1)	2.356 (2)	C(9)—C(10)	1.390 (7)
Rh—Cl(2)	2.359 (1)	C(10)—C(11)	1.380 (6)
Rh—Cl(3)	2.333 (2)	C(10)—C(15)	1.504 (6)
S(1)—C(1)	1.787 (5)	C(11)—C(12)	1.381 (7)
S(1)—C(8)	1.820 (5)	C(12)—C(13)	1.366 (8)
S(2)—C(9)	1.773 (4)	C(13)—C(14)	1.388 (7)
S(2)—C(16)	1.821 (5)	C(15)—C(16)	1.523 (6)
S(3)—C(17)	1.759 (5)	C(17)—C(18)	1.338 (8)
S(3)—C(24)	1.842 (7)	C(17)—C(22)	1.348 (9)

C(1)—C(6)	1.367 (8)	C(18)—C(19)	1.362 (8)
C(1)—C(2)	1.373 (7)	C(18)—C(23)	1.498 (10)
C(2)—C(3)	1.393 (8)	C(19)—C(20)	1.363 (13)
C(2)—C(7)	1.489 (9)	C(20)—C(21)	1.370 (12)
C(3)—C(4)	1.382 (11)	C(21)—C(22)	1.388 (9)
C(4)—C(5)	1.360 (9)	C(23)—C(24)	1.493 (9)
S(1)—Rh—S(2)	92.72 (4)	C(4)—C(3)—C(2)	119.2 (5)
S(1)—Rh—S(3)	93.25 (4)	C(5)—C(4)—C(3)	121.3 (6)
S(1)—Rh—Cl(1)	83.47 (5)	C(4)—C(5)—C(6)	120.1 (7)
S(1)—Rh—Cl(2)	172.96 (5)	C(1)—C(6)—C(5)	118.7 (5)
S(1)—Rh—Cl(3)	93.65 (5)	C(2)—C(7)—C(8)	108.0 (4)
S(2)—Rh—S(3)	172.55 (5)	C(7)—C(8)—S(1)	108.8 (4)
S(2)—Rh—Cl(1)	93.97 (5)	C(14)—C(9)—C(10)	122.6 (4)
S(2)—Rh—Cl(2)	81.80 (4)	C(14)—C(9)—S(2)	124.8 (4)
Cl(3)—Rh—S(2)	86.81 (5)	C(10)—C(9)—S(2)	112.5 (3)
Cl(3)—Rh—S(3)	88.36 (5)	C(11)—C(10)—C(9)	119.7 (4)
Cl(1)—Rh—S(3)	91.14 (5)	C(11)—C(10)—C(15)	126.8 (4)
Cl(2)—Rh—S(3)	92.57 (4)	C(9)—C(10)—C(15)	113.5 (4)
Cl(1)—Rh—Cl(2)	92.46 (5)	C(10)—C(11)—C(12)	118.4 (5)
Cl(1)—Rh—Cl(3)	177.05 (4)	C(13)—C(12)—C(11)	120.6 (4)
Cl(2)—Rh—Cl(3)	90.47 (5)	C(12)—C(13)—C(14)	122.2 (5)
C(1)—S(1)—C(8)	91.1 (2)	C(9)—C(14)—C(13)	116.5 (5)
C(1)—S(1)—Rh	111.3 (1)	C(10)—C(15)—C(16)	105.8 (4)
C(8)—S(1)—Rh	114.6 (2)	C(15)—C(16)—S(2)	106.3 (3)
C(9)—S(2)—C(16)	90.4 (2)	C(18)—C(17)—C(22)	120.8 (5)
C(9)—S(2)—Rh	112.2 (1)	C(18)—C(17)—S(3)	114.5 (5)
C(16)—S(2)—Rh	111.4 (2)	C(22)—C(17)—S(3)	124.6 (4)
C(17)—S(3)—C(24)	89.4 (3)	C(17)—C(18)—C(19)	122.0 (7)
C(17)—S(3)—Rh	109.1 (2)	C(17)—C(18)—C(23)	114.0 (5)
C(24)—S(3)—Rh	110.7 (2)	C(19)—C(18)—C(23)	124.0 (6)
C(6)—C(1)—C(2)	122.8 (5)	C(18)—C(19)—C(20)	118.0 (6)
C(6)—C(1)—S(1)	124.9 (4)	C(19)—C(20)—C(21)	120.8 (7)
C(2)—C(1)—S(1)	112.2 (4)	C(20)—C(21)—C(22)	119.4 (8)
C(1)—C(2)—C(3)	117.9 (6)	C(17)—C(22)—C(21)	118.9 (6)
C(1)—C(2)—C(7)	115.1 (4)	C(24)—C(23)—C(18)	107.1 (5)
C(3)—C(2)—C(7)	127.0 (5)	C(23)—C(24)—S(3)	106.9 (5)

Space group $P2_1/c$ from systematic absences (0*k*0, *k* odd; *h*0*l*, *l* odd). Lorentz–polarization and absorption corrections; no extinction correction. Structure solved by direct methods using *Xtal*2.6 (Hall & Stewart, 1989) and refined by full-matrix least-squares calculations. Non-H atoms allowed to refine with anisotropic temperature factors. H atoms included at geometrically idealized positions (C—H 0.95 Å).

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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 55522 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1009]

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Structure of 6-Methyl-2-oxa-bicyclo[4.3.0]non-4-ene-3,8-dione

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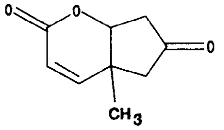
Abstract

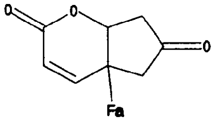
The pyrone ring adopts a C(9)-sofa conformation and the *cis*-fused five-membered ring is in a C(5)-envelope conformation. The methyl group is in a pseudo-axial position. The molecular packing involves C—H···O contacts.

Comment

During the studies of seeds of *Otoba parvifolia* (Mkfg) A. Gentry (Ferreira, 1985), some novel compounds were isolated and identified. The spectroscopic data for the bicyclic lactone (2) raised some doubts about the assignments. In order to explain the data for the natural product, the model compound (1) (in which the farnesyl group is substituted by a methyl group) was synthesized and a crystal structure determination undertaken to determine its stereochemistry unambiguously.



(1)



(2)

Fa = farnesyl

The pyrone ring is in a sofa conformation with C(9) 0.540 (3) Å out of the plane formed by the other five atoms; the carbonyl O atom O(2) lies 0.217 (3) Å out of the plane. As in other pyrones (Selladurai & Subramanian, 1992, and references therein) the angle O(2)—C(2)—O(1) is smaller than O(2)—C(2)—O(3).

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