

(Diphenyl sulfoxide)dimethyl(η^5 -pentamethylcyclopentadienyl)rhodium(III)

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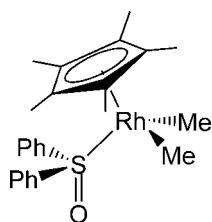
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Key indicators: single-crystal X-ray study; $T = 183$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.039; wR factor = 0.099; data-to-parameter ratio = 19.8.

In the title complex, $[\text{Rh}(\text{CH}_3)_2(\text{C}_{10}\text{H}_{15})(\text{C}_{12}\text{H}_{10}\text{OS})]$, the Rh^{III} atom is in a distorted tetrahedral coordination environment, coordinated by an η^5 -pentamethylcyclopentadienyl ring, two methyl groups and the S atom of the diphenyl sulfoxide ligand. The title compound is representative of a very small group of complexes in which the diphenyl sulfoxide ligand is coordinated *via* the S atom.

Related literature

For related literature, see: de Almeida *et al.* (1992); Calligaris *et al.* (1995); Fooladi *et al.* (2002); Rochon *et al.* (1997); Yatsenko *et al.* (1986).



Experimental

Crystal data

$[\text{Rh}(\text{CH}_3)_2(\text{C}_{10}\text{H}_{15})(\text{C}_{12}\text{H}_{10}\text{OS})]$	$V = 4379.1$ (8) Å ³
$M_r = 470.46$	$Z = 8$
Orthorhombic, $Pccn$	Mo $K\alpha$ radiation
$a = 14.4164$ (15) Å	$\mu = 0.89$ mm ⁻¹
$b = 35.646$ (4) Å	$T = 183$ (1) K
$c = 8.5214$ (8) Å	$0.43 \times 0.33 \times 0.05$ mm

Data collection

Bruker P4 SMART 1000 CCD area-detector diffractometer	28282 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1997a)	4963 independent reflections
$R_{\text{int}} = 0.053$	3473 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.769$, $T_{\max} = 0.957$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	251 parameters
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\max} = 0.83$ e Å ⁻³
4963 reflections	$\Delta\rho_{\min} = -0.94$ e Å ⁻³

Table 1
Selected geometric parameters (Å, °).

Rh—C2	2.101 (4)	S—O	1.489 (2)
Rh—C1	2.196 (3)	S—C9	1.803 (3)
Rh—S	2.2140 (9)	S—C3	1.810 (3)
C2—Rh—C1	83.44 (15)	C1—Rh—S	95.11 (8)
C2—Rh—S	90.68 (11)		

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2194).

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supplementary materials

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(Diphenyl sulfoxide)dimethyl(η^5 -pentamethylcyclopentadienyl)rhodium(III)

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Comment

Research on pentamethylcyclopentadienyl complexes of rhodium(III) and iridium(III) have lead to the comprehension of the activation of saturated alkanes by transition metals. In the course of our study on the activity of such complexes in alkane functionalization reactions, the synthesis of $\text{Cp}^*\text{RhMe}_2(\text{DPSO})$, (I), has been carried out (Cp^* = pentamethylcyclopentadienyl, DPSO = diphenylsulfoxide).

The title compound (Fig. 1) is similar to the complex $\text{Cp}^*\text{RhMe}_2(\text{DMSO})$, (II), reported by Fooladi *et al.* (2002) (DMSO = dimethylsulfoxide). While $\text{Cp}^*\text{RhMe}_2(\text{DPSO})$ crystallizes in the orthorhombic group $Pccn$, the DMSO analogue crystallizes in the triclinic space group $P\bar{1}$ with two independent molecules per asymmetric unit.

In both complexes, the Rh—S bond lengths [2.2140 (9) Å for (I) and 2.210 (1) and 2.211 (1) Å for (II)] and the angles at the rhodium center are similar (Table 1). However, a major difference is observed in the asymmetry of the coordination of the methyl groups. While all Rh—CH₃ bond lengths in (II) are similar [2.090 (5) to 2.103 (5) Å], they differ significantly in (I) [Rh—C1 = 2.196 (3) Å, Rh—C2 = 2.101 (4) Å]. The nature of this difference is unknown and more studies are needed to investigate this discrepancy. While sulfur-coordinated dimethylsulfoxide complexes are common, the number of solid state structures where diphenylsulfoxide is S-bonded are scarce. Indeed, only Pt(II) (Rochon *et al.*, 1997; de Almeida *et al.*, 1992) and Ru(III) (Calligaris *et al.*, 1995) complexes have been reported to have such bonding mode. It is also worth noting that the metric parameters of the DPSO moiety hardly change upon metal coordination. Free DPSO has a S=O bond length of 1.492 (3) Å and S—C bond lengths of 1.793 (no e.s.d. available) Å (Yatsenko *et al.*, 1986), while in (I) these distances are 1.489 (2), 1.803 (3) and 1.810 (3) Å, respectively.

Experimental

A solution of trimethylaluminium (295 mg, 4.04 mmol) in 5 ml pentane was added to a red suspension of $[\text{Cp}^*\text{RhCl}_2]_2$ (250 mg, 0.405 mmol) in 10 ml pentane at 195 K. After stirring for one minute, diphenylsulfoxide (210 mg, 1.04 mmol) was added to the solution. The temperature was slowly raised to room temperature while stirring for 75 min, giving a yellow solution and a brown oil. At 273 K, water (1 ml) was added dropwise to the solution over the course of 15 min to quench the excess AlMe₃ (WARNING: very exothermic reaction). The volatile materials were removed under reduced pressure and the resulting brown oily solid was extracted with methylene chloride (3 × 5 ml) leaving a white residue which was attributed to Al₂O₃.nH₂O. The methylene chloride was removed under reduced pressure and 4 ml of toluene was added to the resulting solid. The addition of 40 ml of pentane to this solution and cooling to 253 K afforded crystals suitable for X-ray crystal structure determination (yield 34%, 130 mg).

supplementary materials

Refinement

H atoms were placed in calculated positions with C—H distances fixed at 0.95 Å (Ar—H) or 0.98 Å (CH_3) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

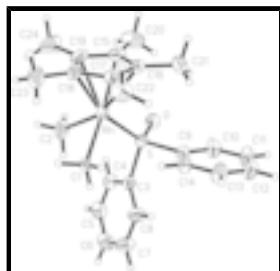


Fig. 1. View of the title compound with displacement ellipsoids at the 50% probability level.

(Diphenyl sulfoxide)dimethyl(η^5 -pentamethylcyclopentadienyl)rhodium(III)

Crystal data

[$\text{Rh}(\text{CH}_3)_2(\text{C}_{10}\text{H}_{15})(\text{C}_{12}\text{H}_{10}\text{OS})$]	$F_{000} = 1952$
$M_r = 470.46$	$D_x = 1.427 \text{ Mg m}^{-3}$
Orthorhombic, $Pccn$	Mo $K\alpha$ radiation
Hall symbol: -P 2ab 2ac	$\lambda = 0.71073 \text{ \AA}$
$a = 14.4164 (15) \text{ \AA}$	Cell parameters from 6117 reflections
$b = 35.646 (4) \text{ \AA}$	$\theta = 2.8\text{--}28.4^\circ$
$c = 8.5214 (8) \text{ \AA}$	$\mu = 0.89 \text{ mm}^{-1}$
$V = 4379.1 (8) \text{ \AA}^3$	$T = 183 (1) \text{ K}$
$Z = 8$	Plate, yellow
	$0.43 \times 0.33 \times 0.05 \text{ mm}$

Data collection

Bruker P4 SMART 1000 CCD area-detector diffractometer	3473 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.053$
$T = 183(1) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ϕ and ω scans	$\theta_{\text{min}} = 1.5^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1997a)	$h = -18 \rightarrow 18$
$T_{\text{min}} = 0.769$, $T_{\text{max}} = 0.957$	$k = -46 \rightarrow 44$
28282 measured reflections	$l = -10 \rightarrow 11$
4963 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 7.0043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\max} = 0.001$
4963 reflections	$\Delta\rho_{\max} = 0.83 \text{ e \AA}^{-3}$
251 parameters	$\Delta\rho_{\min} = -0.94 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Crystal decay was monitored by repeating the initial 50 frames at the end of the data collection and analyzing duplicate reflections.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Rh	0.494908 (16)	0.157754 (7)	0.17044 (3)	0.02332 (9)
S	0.47396 (5)	0.09623 (2)	0.17229 (10)	0.02366 (17)
O	0.40279 (16)	0.08105 (7)	0.0627 (3)	0.0324 (6)
C1	0.5718 (2)	0.15940 (9)	0.3928 (4)	0.0244 (7)
H1A	0.5590	0.1831	0.4469	0.037*
H1B	0.6384	0.1574	0.3716	0.037*
H1C	0.5523	0.1384	0.4593	0.037*
C2	0.3791 (3)	0.16516 (11)	0.3167 (5)	0.0457 (10)
H2A	0.3767	0.1912	0.3529	0.069*
H2B	0.3840	0.1484	0.4074	0.069*
H2C	0.3224	0.1593	0.2581	0.069*
C3	0.4456 (2)	0.07566 (9)	0.3604 (4)	0.0255 (7)
C4	0.3525 (2)	0.07536 (10)	0.3990 (4)	0.0323 (8)
H4	0.3077	0.0844	0.3265	0.039*
C5	0.3249 (3)	0.06179 (11)	0.5439 (5)	0.0400 (9)
H5	0.2610	0.0616	0.5714	0.048*

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C6	0.3905 (3)	0.04847 (12)	0.6486 (5)	0.0441 (10)
H6	0.3718	0.0396	0.7487	0.053*
C7	0.4830 (3)	0.04816 (13)	0.6074 (5)	0.0452 (10)
H7	0.5277	0.0382	0.6781	0.054*
C8	0.5112 (3)	0.06226 (11)	0.4634 (4)	0.0374 (9)
H8	0.5751	0.0627	0.4362	0.045*
C9	0.5793 (2)	0.07126 (9)	0.1267 (4)	0.0259 (7)
C10	0.5722 (3)	0.03924 (10)	0.0361 (4)	0.0347 (8)
H10	0.5133	0.0308	0.0009	0.042*
C11	0.6517 (3)	0.01946 (11)	-0.0030 (5)	0.0418 (9)
H11	0.6473	-0.0027	-0.0646	0.050*
C12	0.7375 (3)	0.03196 (10)	0.0478 (5)	0.0385 (9)
H12	0.7918	0.0181	0.0226	0.046*
C13	0.7445 (3)	0.06440 (11)	0.1346 (5)	0.0395 (9)
H13	0.8037	0.0731	0.1675	0.047*
C14	0.6649 (2)	0.08441 (11)	0.1743 (4)	0.0344 (8)
H14	0.6695	0.1069	0.2336	0.041*
C15	0.4633 (3)	0.17269 (11)	-0.0814 (4)	0.0353 (9)
C16	0.5617 (2)	0.16543 (10)	-0.0710 (4)	0.0282 (8)
C17	0.6007 (2)	0.19137 (10)	0.0347 (4)	0.0293 (8)
C18	0.5266 (3)	0.21465 (10)	0.0935 (4)	0.0346 (9)
C19	0.4430 (3)	0.20382 (11)	0.0149 (5)	0.0387 (9)
C20	0.3977 (3)	0.15331 (14)	-0.1917 (5)	0.0570 (13)
H20A	0.3339	0.1566	-0.1544	0.086*
H20B	0.4126	0.1265	-0.1959	0.086*
H20C	0.4038	0.1642	-0.2968	0.086*
C21	0.6125 (3)	0.13685 (11)	-0.1673 (4)	0.0424 (9)
H21A	0.6235	0.1469	-0.2727	0.064*
H21B	0.5752	0.1139	-0.1747	0.064*
H21C	0.6721	0.1311	-0.1173	0.064*
C22	0.7017 (3)	0.19547 (12)	0.0745 (5)	0.0467 (10)
H22A	0.7326	0.1711	0.0632	0.070*
H22B	0.7079	0.2042	0.1830	0.070*
H22C	0.7303	0.2137	0.0033	0.070*
C23	0.5388 (4)	0.24817 (12)	0.1981 (5)	0.0602 (13)
H23A	0.5848	0.2425	0.2793	0.090*
H23B	0.4794	0.2544	0.2477	0.090*
H23C	0.5601	0.2696	0.1355	0.090*
C24	0.3503 (3)	0.22286 (14)	0.0269 (6)	0.0654 (15)
H24A	0.3436	0.2409	-0.0589	0.098*
H24B	0.3459	0.2360	0.1277	0.098*
H24C	0.3009	0.2041	0.0199	0.098*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rh	0.02300 (14)	0.02176 (14)	0.02520 (14)	0.00259 (10)	0.00236 (11)	-0.00017 (10)
S	0.0210 (4)	0.0238 (4)	0.0261 (4)	0.0002 (3)	0.0001 (3)	-0.0004 (3)

O	0.0261 (12)	0.0347 (14)	0.0365 (13)	-0.0026 (11)	-0.0048 (10)	-0.0037 (11)
C1	0.0375 (19)	0.0146 (15)	0.0211 (15)	-0.0030 (14)	0.0045 (14)	-0.0020 (13)
C2	0.048 (2)	0.037 (2)	0.052 (2)	0.0100 (18)	0.022 (2)	0.0004 (19)
C3	0.0271 (17)	0.0192 (16)	0.0302 (18)	-0.0038 (13)	0.0046 (14)	0.0011 (13)
C4	0.0265 (18)	0.031 (2)	0.039 (2)	-0.0032 (15)	-0.0001 (15)	0.0012 (16)
C5	0.033 (2)	0.042 (2)	0.045 (2)	-0.0079 (17)	0.0102 (18)	-0.0047 (19)
C6	0.051 (2)	0.045 (2)	0.036 (2)	-0.010 (2)	0.0111 (19)	0.0015 (18)
C7	0.042 (2)	0.052 (3)	0.041 (2)	0.000 (2)	0.0000 (18)	0.017 (2)
C8	0.0275 (19)	0.050 (2)	0.0353 (19)	-0.0002 (17)	0.0027 (16)	0.0117 (17)
C9	0.0243 (16)	0.0219 (17)	0.0315 (17)	0.0013 (13)	0.0014 (14)	-0.0014 (14)
C10	0.0310 (19)	0.0267 (19)	0.046 (2)	-0.0088 (15)	0.0036 (17)	-0.0037 (17)
C11	0.040 (2)	0.027 (2)	0.059 (3)	-0.0026 (16)	0.013 (2)	-0.0113 (18)
C12	0.031 (2)	0.030 (2)	0.055 (2)	0.0067 (16)	0.0111 (18)	-0.0013 (18)
C13	0.0238 (17)	0.037 (2)	0.058 (2)	0.0000 (16)	0.0022 (17)	-0.0061 (18)
C14	0.0256 (17)	0.037 (2)	0.041 (2)	0.0011 (15)	0.0008 (17)	-0.0105 (18)
C15	0.0308 (19)	0.040 (2)	0.035 (2)	-0.0006 (17)	-0.0004 (16)	0.0124 (17)
C16	0.0337 (19)	0.0260 (18)	0.0250 (16)	0.0008 (14)	0.0004 (14)	0.0040 (14)
C17	0.0303 (18)	0.0273 (18)	0.0303 (18)	-0.0025 (15)	0.0051 (15)	0.0042 (15)
C18	0.047 (2)	0.0193 (17)	0.037 (2)	0.0023 (16)	0.0128 (17)	0.0060 (15)
C19	0.0326 (19)	0.036 (2)	0.047 (2)	0.0112 (16)	0.0079 (18)	0.0183 (19)
C20	0.050 (3)	0.076 (3)	0.045 (3)	-0.013 (2)	-0.017 (2)	0.011 (2)
C21	0.052 (2)	0.044 (2)	0.032 (2)	0.0043 (19)	0.0101 (19)	-0.0053 (18)
C22	0.034 (2)	0.052 (3)	0.054 (3)	-0.0127 (19)	0.0044 (19)	0.002 (2)
C23	0.098 (4)	0.027 (2)	0.056 (3)	-0.003 (2)	0.024 (3)	-0.005 (2)
C24	0.049 (3)	0.070 (3)	0.078 (3)	0.036 (2)	0.015 (2)	0.032 (3)

Geometric parameters (Å, °)

Rh—C2	2.101 (4)	C11—C12	1.384 (5)
Rh—C18	2.180 (3)	C11—H11	0.9500
Rh—C1	2.196 (3)	C12—C13	1.377 (5)
Rh—S	2.2140 (9)	C12—H12	0.9500
Rh—C19	2.239 (4)	C13—C14	1.392 (5)
Rh—C15	2.258 (4)	C13—H13	0.9500
Rh—C17	2.258 (3)	C14—H14	0.9500
Rh—C16	2.288 (3)	C15—C19	1.411 (6)
S—O	1.489 (2)	C15—C16	1.445 (5)
S—C9	1.803 (3)	C15—C20	1.502 (6)
S—C3	1.810 (3)	C16—C17	1.408 (5)
C1—H1A	0.9800	C16—C21	1.499 (5)
C1—H1B	0.9800	C17—C18	1.442 (5)
C1—H1C	0.9800	C17—C22	1.502 (5)
C2—H2A	0.9800	C18—C19	1.432 (6)
C2—H2B	0.9800	C18—C23	1.501 (6)
C2—H2C	0.9800	C19—C24	1.502 (5)
C3—C8	1.376 (5)	C20—H20A	0.9800
C3—C4	1.381 (5)	C20—H20B	0.9800
C4—C5	1.385 (5)	C20—H20C	0.9800
C4—H4	0.9500	C21—H21A	0.9800

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C5—C6	1.384 (6)	C21—H21B	0.9800
C5—H5	0.9500	C21—H21C	0.9800
C6—C7	1.380 (6)	C22—H22A	0.9800
C6—H6	0.9500	C22—H22B	0.9800
C7—C8	1.387 (5)	C22—H22C	0.9800
C7—H7	0.9500	C23—H23A	0.9800
C8—H8	0.9500	C23—H23B	0.9800
C9—C14	1.381 (5)	C23—H23C	0.9800
C9—C10	1.382 (5)	C24—H24A	0.9800
C10—C11	1.386 (5)	C24—H24B	0.9800
C10—H10	0.9500	C24—H24C	0.9800
C2—Rh—C18	103.19 (15)	C12—C11—H11	120.0
C2—Rh—C1	83.44 (15)	C10—C11—H11	120.0
C18—Rh—C1	97.40 (13)	C13—C12—C11	120.3 (3)
C2—Rh—S	90.68 (11)	C13—C12—H12	119.9
C18—Rh—S	162.25 (10)	C11—C12—H12	119.9
C1—Rh—S	95.11 (8)	C12—C13—C14	120.0 (4)
C2—Rh—C19	89.62 (15)	C12—C13—H13	120.0
C18—Rh—C19	37.79 (15)	C14—C13—H13	120.0
C1—Rh—C19	131.29 (14)	C9—C14—C13	119.4 (3)
S—Rh—C19	133.25 (11)	C9—C14—H14	120.3
C2—Rh—C15	111.98 (16)	C13—C14—H14	120.3
C18—Rh—C15	62.42 (14)	C19—C15—C16	108.0 (3)
C1—Rh—C15	156.32 (13)	C19—C15—C20	126.5 (4)
S—Rh—C15	102.30 (11)	C16—C15—C20	125.1 (4)
C19—Rh—C15	36.57 (14)	C19—C15—Rh	71.0 (2)
C2—Rh—C17	140.72 (14)	C16—C15—Rh	72.62 (19)
C18—Rh—C17	37.87 (13)	C20—C15—Rh	127.8 (3)
C1—Rh—C17	95.00 (12)	C17—C16—C15	108.2 (3)
S—Rh—C17	128.43 (9)	C17—C16—C21	127.0 (3)
C19—Rh—C17	62.17 (13)	C15—C16—C21	124.6 (3)
C15—Rh—C17	61.57 (13)	C17—C16—Rh	70.81 (19)
C2—Rh—C16	148.57 (15)	C15—C16—Rh	70.32 (19)
C18—Rh—C16	61.97 (13)	C21—C16—Rh	128.1 (2)
C1—Rh—C16	124.07 (12)	C16—C17—C18	107.8 (3)
S—Rh—C16	100.50 (9)	C16—C17—C22	126.5 (3)
C19—Rh—C16	61.37 (13)	C18—C17—C22	125.7 (3)
C15—Rh—C16	37.06 (13)	C16—C17—Rh	73.1 (2)
C17—Rh—C16	36.07 (12)	C18—C17—Rh	68.12 (19)
O—S—C9	105.44 (15)	C22—C17—Rh	126.2 (3)
O—S—C3	104.63 (15)	C19—C18—C17	107.8 (3)
C9—S—C3	100.44 (16)	C19—C18—C23	126.2 (4)
O—S—Rh	116.70 (11)	C17—C18—C23	125.3 (4)
C9—S—Rh	111.88 (11)	C19—C18—Rh	73.3 (2)
C3—S—Rh	116.00 (11)	C17—C18—Rh	74.01 (19)
Rh—C1—H1A	109.5	C23—C18—Rh	125.9 (3)
Rh—C1—H1B	109.5	C15—C19—C18	108.0 (3)
H1A—C1—H1B	109.5	C15—C19—C24	125.4 (4)
Rh—C1—H1C	109.5	C18—C19—C24	126.5 (4)

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H1A—C1—H1C	109.5	C15—C19—Rh	72.4 (2)
H1B—C1—H1C	109.5	C18—C19—Rh	68.86 (19)
Rh—C2—H2A	109.5	C24—C19—Rh	126.0 (3)
Rh—C2—H2B	109.5	C15—C20—H20A	109.5
H2A—C2—H2B	109.5	C15—C20—H20B	109.5
Rh—C2—H2C	109.5	H20A—C20—H20B	109.5
H2A—C2—H2C	109.5	C15—C20—H20C	109.5
H2B—C2—H2C	109.5	H20A—C20—H20C	109.5
C8—C3—C4	120.9 (3)	H20B—C20—H20C	109.5
C8—C3—S	123.4 (3)	C16—C21—H21A	109.5
C4—C3—S	115.7 (3)	C16—C21—H21B	109.5
C3—C4—C5	119.6 (4)	H21A—C21—H21B	109.5
C3—C4—H4	120.2	C16—C21—H21C	109.5
C5—C4—H4	120.2	H21A—C21—H21C	109.5
C6—C5—C4	119.9 (4)	H21B—C21—H21C	109.5
C6—C5—H5	120.1	C17—C22—H22A	109.5
C4—C5—H5	120.1	C17—C22—H22B	109.5
C7—C6—C5	120.0 (4)	H22A—C22—H22B	109.5
C7—C6—H6	120.0	C17—C22—H22C	109.5
C5—C6—H6	120.0	H22A—C22—H22C	109.5
C6—C7—C8	120.3 (4)	H22B—C22—H22C	109.5
C6—C7—H7	119.8	C18—C23—H23A	109.5
C8—C7—H7	119.8	C18—C23—H23B	109.5
C3—C8—C7	119.3 (3)	H23A—C23—H23B	109.5
C3—C8—H8	120.4	C18—C23—H23C	109.5
C7—C8—H8	120.4	H23A—C23—H23C	109.5
C14—C9—C10	120.7 (3)	H23B—C23—H23C	109.5
C14—C9—S	121.5 (3)	C19—C24—H24A	109.5
C10—C9—S	117.8 (3)	C19—C24—H24B	109.5
C9—C10—C11	119.6 (3)	H24A—C24—H24B	109.5
C9—C10—H10	120.2	C19—C24—H24C	109.5
C11—C10—H10	120.2	H24A—C24—H24C	109.5
C12—C11—C10	120.0 (4)	H24B—C24—H24C	109.5

supplementary materials

Fig. 1

