

Nonacarbonyl(μ_2 -methanethiolato)(μ_3 -trimethylsilylacetylene)triruthenium

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Key indicators
 Single-crystal X-ray study
 T = 150 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.025
 wR factor = 0.060
 Data-to-parameter ratio = 29.1

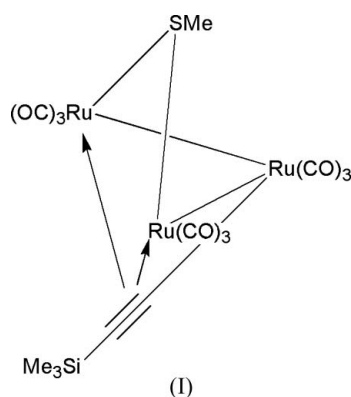
 For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The structure of the title compound, $[\text{Ru}_3(\mu_2\text{CH}_3\text{S})(\mu_3\text{-C}_2\text{Si}(\text{CH}_3)_3)(\text{CO})_9]$, contains an open triangle of Ru atoms. The open edge is bridged on one side by a μ_2 -S(CH₃) unit, and the other side of the triangle is capped by a $[\text{C}\equiv\text{CSi}(\text{CH}_3)_3]$ group, with the acetylenic bond perpendicular to the open Ru··Ru edge.

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Comment

The homogeneous desulfurization of thiophene with transition metal compounds has provided an important model for the catalytic heterogeneous dehydrodesulfurization of thiophenic components of crude oil (Rauschfuss, 1991). Increased catalytic activity has been observed, particularly toward thiophenes, when late transition metals (group 8) are employed (Angelici, 1997).

In the context of catalysis, we have been investigating the reactivity of ruthenium carbonyl clusters with thiol derivatives. The reaction of $\text{Ru}_3(\text{CO})_{12}$ with the asymmetric acetylene derivative $\text{Me}_3\text{SC}\equiv\text{CSiMe}_3$ under reflux in hexane afforded $[\text{Ru}_3(\text{CO})_9(\mu_2\text{-H}_3\text{CS})(\mu_3\text{-C}_2\text{Si}(\text{CH}_3)_3)]$, (I), in which the ruthenium cluster has inserted into a C—S bond. This type of reaction has been observed previously (Arce *et al.*, 1994).



The cluster consists of an open ruthenium triangle (Fig. 1), with the ‘non bonding’ edge having an Ru··Ru distance of 3.360 (1) Å. This edge is bridged symmetrically by a μ_2 -S(CH₃) group, and the other side of the metal triangle is capped by a $(\text{C}\equiv\text{CSiMe}_3)$ group in which one acetylenic C atom σ -bonds to Ru2 while the acetylenic triple bond is π -bound to Ru1 and Ru3, adopting a bonding mode perpendicular to the long Ru1··Ru3 edge. The nine carbonyl ligands are terminal and essentially linear, three being bound to each Ru centre. Overall, the cluster has an electron count of 50, consistent with the observed open triangular metal framework.

Experimental

Powdered $\text{Ru}_3(\text{CO})_{12}$ (0.102 g, 0.16 mmol) and $\text{Me}_3\text{SC}\equiv\text{CSiMe}_3$ (0.085 g, 0.75 mmol) were dissolved in hexane (20 ml) and the mixture refluxed for 3 h, after which a second portion of $\text{Me}_3\text{SC}\equiv\text{CSiMe}_3$ [0.090 g, 0.79 mmol, in hexane (5 ml)] was added. The mixture was further refluxed for 1 h and subsequently cooled. Upon solvent removal, the crude product was purified by preparative thin layer chromatography using hexane as an eluent. The title compound was obtained from the yellow band with the highest R_f value after that of $\text{Ru}_3(\text{CO})_{12}$. The product was recrystallized from a 1:1 mixture of CH_2Cl_2 /hexane to give yellow block crystals.

Crystal data

$[\text{Ru}_3(\text{CH}_3\text{S})(\text{C}_5\text{H}_9\text{Si})(\text{CO})_9]$	$V = 1119.81 (10) \text{ \AA}^3$
$M_r = 699.61$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 2.075 \text{ Mg m}^{-3}$
$a = 9.0510 (1) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.6680 (1) \text{ \AA}$	$\mu = 2.19 \text{ mm}^{-1}$
$c = 14.5830 (2) \text{ \AA}$	$T = 150 (2) \text{ K}$
$\alpha = 76.319 (1)^\circ$	Block, yellow
$\beta = 88.116 (1)^\circ$	$0.4 \times 0.35 \times 0.35 \text{ mm}$
$\gamma = 64.933 (10)^\circ$	

Data collection

Bruker–Nonius KappaCCD diffractometer	25218 measured reflections
ω and φ scans	7649 independent reflections
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	7024 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.393$, $T_{\max} = 0.466$	$R_{\text{int}} = 0.034$
	$\theta_{\text{max}} = 32.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 0.728P]$
$R[F^2 > 2\sigma(F^2)] = 0.025$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.060$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 1.16 \text{ e \AA}^{-3}$
7649 reflections	$\Delta\rho_{\text{min}} = -1.05 \text{ e \AA}^{-3}$
263 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0125 (5)

The methyl H atoms were constrained as riding atoms with $\text{C}-\text{H} = 0.98 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The highest residual electron-density peak is 1.81 \AA from O8 and the deepest hole is 0.69 \AA from Ru2.

Data collection: COLLECT (Nonius, 2000); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997); program(s) used to solve structure: DIRDIF99 (Beurskens *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick,

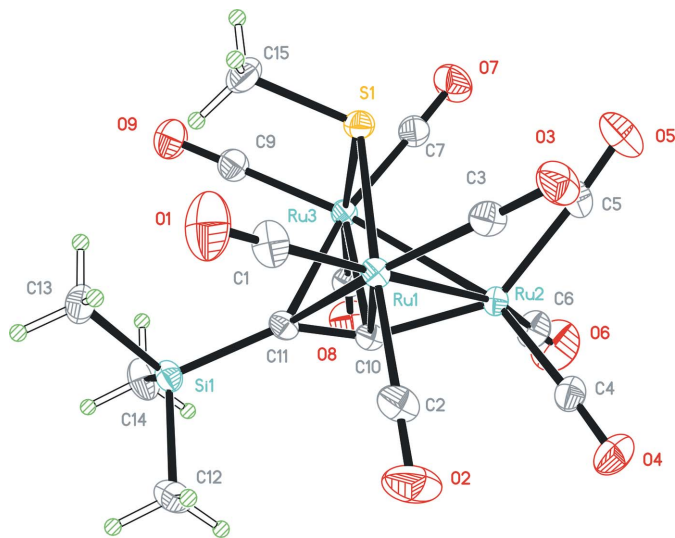


Figure 1
The molecular structure of (I), showing the atom-numbering scheme (50% probability displacement ellipsoids).

1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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