

Nonacarbonyl- $\mu$ -hydrido-( $\mu^3, \eta^2$ -triisopropylsilyl-ethynyl)triruthenium

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## Key indicators

Single-crystal X-ray study

$T = 160$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å

$R$  factor = 0.020

$wR$  factor = 0.054

Data-to-parameter ratio = 14.2

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,  $[\text{Ru}_3(\text{CO})_9(\mu\text{-H})(\mu_3\text{-C}_2\text{Si}^i\text{Pr}_3)]$ , consists of an almost equilateral triangular  $\text{Ru}_3$  core, with the acetylide ligand  $\sigma$ -bonded to one Ru atom and  $\pi$ -bonded to the other two Ru atoms. The hydride atom is located between the two Ru atoms that are  $\pi$ -bonded to the acetylide. Three terminal carbonyls around each ruthenium atom complete the structure.

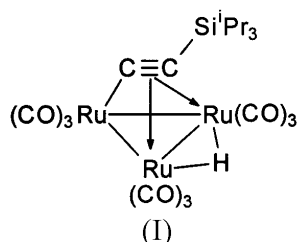
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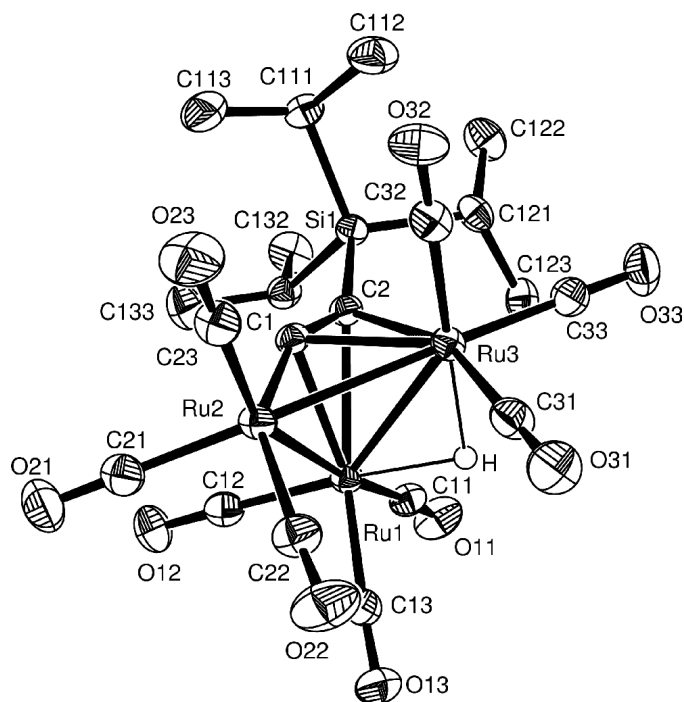
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## Comment

Acetylide compounds are being widely studied because of the versatile coordination modes of the unsaturated fragments which act as metalloligands (Ara *et al.*, 1995). Researchers are also interested in cluster chemistry containing  $\text{C}_2\text{R}$  groups, regarding their implications in the syntheses of organic molecules that take place on metal surfaces (Chi *et al.*, 1997). More recently the acetylide derivatives have been used as precursors to synthesize polymetallic compounds bearing unsaturated polycarbon chains (Alcalde *et al.*, 2001). Herein we report the structure of the new compound  $[\text{Ru}_3(\text{CO})_9(\mu\text{-H})(\mu_3\text{-C}_2\text{Si}^i\text{Pr}_3)]$ , (I). The  $\nu(\text{CO})$  pattern in the carbonyl region as well as the  $^1\text{H NMR}$  resonance for hydride observed in the spectra are in agreement with data reported on analogous compounds (Sappa *et al.*, 1972; Edwards *et al.*, 1995).



The title compound consists of a triangular ruthenium cluster (see Fig. 1). The Ru–Ru distances [Ru1–Ru3 2.8010 (9), Ru1–Ru2 2.7907 (13) and Ru2–Ru3 2.7957 (11) Å] are similar to those reported for other compounds of this type  $\{[\text{Ru}_3(\text{CO})_9(\mu\text{-H})(\mu_3\text{-C}_2\text{R})]: R = \text{H}$  [2.792 (1), 2.803 (1), 2.810 (1) Å (Bruce *et al.*, 1999)];  $R = \text{'Bu}$  [2.790 (2), 2.795 (2), 2.796 (2) Å (Gervasio & Ferraris, 1973)]}. The three ruthenium atoms in all of these compounds form an almost equilateral triangle. In (I) the acetylide ligand is  $\sigma$ -bonded to one Ru atom [Ru2–C1 1.958 (2) Å] and  $\pi$ -bonded to the other two Ru atoms [Ru3–C1 2.215 (2), Ru1–C1 2.206 (2), Ru3–C2 2.302 (3) and Ru1–C2 2.322 (2) Å]. The acetylide group is acting as a  $5e^-$  donor, the C1–C2 distance [1.299 (3) Å] being comparable to that in other acetylide



**Figure 1**  
View of (I), with displacement ellipsoids drawn at the 50% probability level.

clusters  $\{[\text{Ru}_3(\text{CO})_9(\mu\text{-H})(\mu_3\text{-C}_2\text{R})]: R = \text{H} [1.28 (1) \text{ \AA} (\text{Bruce et al., 1999})]; R = \text{t Bu} [1.29 (3) \text{ \AA} (\text{Gervasio \& Ferraris, 1973})]\}$ . The hydride is unsymmetrically located between atoms Ru1 and Ru3, giving Ru–H distances of 1.81 (3) and 1.77 (3) \AA (see Table 1). Three terminal CO ligands around each metal atom complete the structure.

## Experimental

A mixture of  $\text{Ru}_3(\text{CO})_{12}$  (0.100 g, 0.156 mmol) and  $\text{HC}\equiv\text{CSi}^i\text{Pr}_3$  (0.056 g, 0.156 mmol) in toluene (15 cm<sup>3</sup>) was heated with stirring at 358 K for 2.5 h. After removing the solvent *in vacuo*, the residue was crystallized from hexane at 253 K, giving yellow crystals of  $[\text{Ru}_3(\text{CO})_9(\mu\text{-H})(\mu_3\text{-C}_2\text{Si}^i\text{Pr}_3)]$  (0.090 g, 78.2% yield). Anal. Calcd. for  $\text{C}_{20}\text{H}_{22}\text{O}_9\text{SiRu}_3$  (Found): C, 32.56 (32.79); H, 3.01 (2.81)%. IR (toluene, cm<sup>-1</sup>)  $\nu(\text{CO})$ : 2095 (*m*), 2067 (*vs*), 2050 (*vs*), 2018 (*vs*), 1984 (*m*). <sup>1</sup>H NMR ( $\text{CDCl}_3$ ):  $\delta = 1.17$  [*s*, 21H, <sup>t</sup>Pr],  $-20.83$  [*s*, 1H, H]. FAB<sup>+</sup> (*m/z*): 737 (*M*<sup>+</sup>), 711–485 (*M*<sup>+</sup>–*n*CO, *n* = 1–9).

### Crystal data

|  |   |
|--|---|
| $[\text{Ru}_3(\text{C}_2)\text{H}(\text{C}_9\text{H}_{21}\text{Si})(\text{CO})_9]$ | $Z = 2$                                   |
| $M_r = 737.68$   | $D_x = 1.91 \text{ Mg m}^{-3}$            |
| Triclinic, $P\bar{1}$  | Mo $K\alpha$ radiation                    |
| $a = 9.3592 (13) \text{ \AA}$  | Cell parameters from 5000 reflections     |
| $b = 11.8691 (18) \text{ \AA}$   | $\theta = 1.5\text{--}26.0^\circ$         |
| $c = 12.6669 (17) \text{ \AA}$   | $\mu = 1.83 \text{ mm}^{-1}$              |
| $\alpha = 79.393 (17)^\circ$   | $T = 160 (2) \text{ K}$                   |
| $\beta = 87.314 (17)^\circ$  | Parallelepiped, orange–yellow             |
| $\gamma = 68.106 (16)^\circ$   | $0.40 \times 0.35 \times 0.20 \text{ mm}$ |
| $V = 1282.9 (3) \text{ \AA}^3$   |   |

### Data collection

|  |                                    |
|--|------------------------------------|
| Stoe IPDS diffractometer                           | $R_{\text{int}} = 0.028$           |
| $\varphi$ scans                                    | $\theta_{\text{max}} = 26.0^\circ$ |
| Absorption correction: multi-scan (Blessing, 1995) | $h = -11 \rightarrow 11$           |
| $T_{\text{min}} = 0.342, T_{\text{max}} = 0.463$   | $k = -14 \rightarrow 14$           |
| 12223 measured reflections                         | $l = -15 \rightarrow 15$           |
| 4546 independent reflections                       | 200 standard reflections           |
| 4456 reflections with $I > 2\sigma(I)$             | frequency: 3 min                   |
|  | intensity decay: <0.1%             |

### Refinement

|  |  |
|--|--|
| Refinement on $F^2$  | $w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 1.1678P]$    |
| $R[F^2 > 2\sigma(F^2)] = 0.020$  | where $P = (F_o^2 + 2F_c^2)/3$                       |
| $wR(F^2) = 0.054$  | $(\Delta/\sigma)_{\text{max}} = 0.002$               |
| $S = 1.13$   | $\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$  |
| 4546 reflections   | $\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$ |
| 320 parameters   |  |
| H atoms treated by a mixture of independent and constrained refinement |  |

**Table 1**

Selected geometric parameters (\AA,  $^\circ$ ).

|             |             |            |             |
|-------------|-------------|------------|-------------|
| Ru1–C1      | 2.206 (2)   | Ru2–Ru3    | 2.7957 (5)  |
| Ru1–C2      | 2.322 (2)   | Ru3–C1     | 2.215 (2)   |
| Ru1–Ru2     | 2.7907 (5)  | Ru3–C2     | 2.302 (3)   |
| Ru1–Ru3     | 2.8010 (5)  | Ru3–H      | 1.77 (3)    |
| Ru1–H       | 1.81 (3)    | Si–C2      | 1.881 (3)   |
| Ru2–C1      | 1.958 (2)   | C1–C2      | 1.299 (3)   |
| Ru2–Ru1–Ru3 | 60.00 (1)   | C2–C1–Ru2  | 156.06 (19) |
| Ru1–Ru2–Ru3 | 60.184 (11) | Ru1–C1–Ru3 | 78.63 (8)   |
| Ru2–Ru3–Ru1 | 59.82 (2)   | C1–C2–Si   | 145.28 (19) |
| Ru1–H–Ru3   | 102.2 (16)  | Ru3–C2–Ru1 | 74.57 (7)   |

All H atoms were located in difference Fourier maps, and refined by using a riding-model approximation with isotropic displacement parameters fixed at 20% higher than those of the C atoms to which they are connected. The hydride H was refined independently with an isotropic displacement parameter. Owing to the geometry of the Stoe IPDS diffractometer, which has only one circle ( $\varphi$  rotation), the coverage of the data is not complete and only around 90%. To obtain a better coverage, we would need to recollect data with the crystal mounted in a different orientation.

Data collection: *IPDS Software* (Stoe, 1996); cell refinement: *IPDS Software*; data reduction: *XRED* (Stoe, 1996); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) in *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997) in *WinGX*; molecular graphics: *ORTEP3* for Windows (Farrugia, 1997) in *WinGX*; software used to prepare material for publication: *SHELXL97* in *WinGX*.

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## References

- Alcalde, M. I., Carty, A. J., Delgado, E., Donnadiu, B., Hern3ndez, E., Dallamann, K. & S3nchez-Nieves. (2001). *J. Chem. Soc. Dalton Trans.* pp. 2502–2506.
- Ara, I., Forni3s, J., Lalinde, E., Moreno, M. T. & Tom3s, M. (1995). *J. Chem. Soc. Dalton Trans.* pp. 2397–2405.
- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruce, M. I., Skelton, B. W., White, A. H. & Zaitseva, N. N. (1999). *J. Chem. Soc. Dalton Trans.* pp. 1445–1453.

- Chi, Y., Chung, C., Chou, Y.-C., Chiang, S.-J., Peng, S.-M. & Lee, G.-H. (1997). *Organometallics*, **16**, 1702–1713.
- Edwards, A. J., Leadbeater, N. E., Lewis, J. & Raithby, P. R. (1995). *J. Chem. Soc. Dalton Trans.* pp. 3785–3787.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Gervasio, G. & Ferraris, G. (1973). *Cryst. Struct. Commun.* **3**, 447–450.
- Sappa, E., Gambio, O., Milone, L. & Cetini, G. (1972). *J. Organomet. Chem.* **39**, 169–172.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Stoe (1996). *IPDS Software* (Version 2.75) and *X-RED* (Revision 1.08). Stoe & Cie, Darmstadt, Germany.