

Crystal structure of $[\mu\text{-(3,4-}\eta\text{:4,5,6,7-}\eta\text{)-}\{5,6\text{-di(n-propyl)-3,4,6-decatrien}}\}][\text{bis}(\eta^5\text{-cyclopentadienyl})(\text{dimethyl})\text{silan}] \text{tricarbonyl-1}\kappa^2\text{C:2}\kappa^1\text{C-tungstenmolybdenum(W-Mo), C}_{31}\text{H}_{42}\text{MoO}_3\text{SiW}$

W. P. Bosman

University of Nijmegen, Faculty of Science, RIM Laboratory of Solid State Chemistry, Toernooiveld 1, 6525 ED Nijmegen, The Netherlands

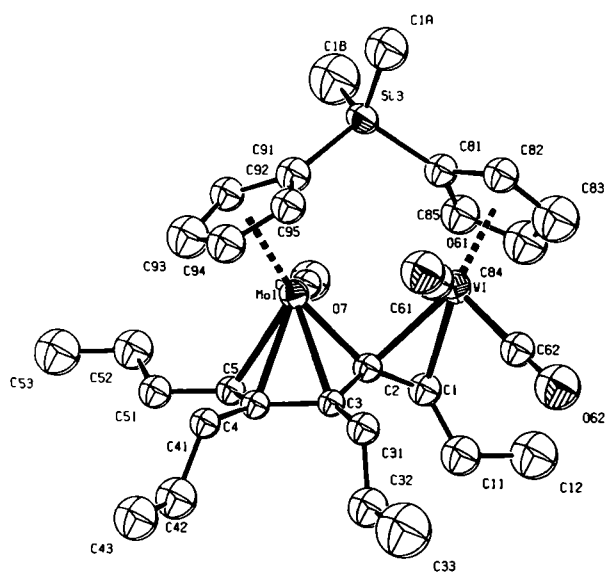
J. M. M. Smits, K. Kriebisch

University of Nijmegen, Department of Inorganic Chemistry, Toernooiveld 1, 6525 ED Nijmegen, The Netherlands

and J. Heck

Universität Hamburg, Institut für Anorganische und Angewandte Chemie, Martin-Luther-King-Platz 8, D-20146 Hamburg, Germany

Received October 31, 1996, CSD-No. 402685



Source of material: The preparation of the crystals (recrystallized from hexane) will be described elsewhere. (see ref. 1).

Application of DIRDIF (see ref. 2) using the automatic Patterson option (PATTY) (see ref. 3) led to the positions of the non-hydrogen atoms.

Table 1. Parameters used for the X-ray data collection

| | |
|--------------------------------------|--|
| Crystal: | brown plate, size 0.08 x 0.35 x 0.35 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| μ : | 42.77 cm ⁻¹ |
| Diffractometer: | Enraf-Nonius CAD4 |
| Scan mode: | ω (see refs. 3-4) |
| $T_{\text{measurement}}$: | 293 K |
| $2\theta_{\text{max}}$: | 40° |
| $N(hkl)_{\text{unique}}$: | 2799 |
| Criterion for I_0 : | $I_0 > 2\sigma(I_0)$ |
| $N(\text{param})_{\text{refined}}$: | 170 |
| Programs: | DIFABS, DIRDIF, PATTY, SHELXL-93, PLATON |

$\text{C}_{31}\text{H}_{42}\text{MoO}_3\text{SiW}$, monoclinic, $C12/c1$ (No. 15), $a = 26.976(6)$ Å, $b = 16.141(2)$ Å, $c = 16.206(2)$ Å, $\beta = 120.83(3)^\circ$, $V = 6059.3$ Å³, $Z = 8$, $R(F) = 0.060$, $R_w(F^2) = 0.153$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

| Atom | Site | x | y | z | U_{iso} |
|--------|------|-----------|----------|----------|------------------|
| C(1) | 8f | 0.4405(9) | 0.218(1) | 0.422(2) | 0.040(6) |
| H(1) | 8f | 0.4348(9) | 0.231(1) | 0.362(2) | 0.048 |
| C(11) | 8f | 0.486(1) | 0.161(2) | 0.476(2) | 0.058(7) |
| H(11A) | 8f | 0.474(1) | 0.124(2) | 0.510(2) | 0.070 |
| H(11B) | 8f | 0.519(1) | 0.192(2) | 0.525(2) | 0.070 |
| C(12) | 8f | 0.507(1) | 0.108(2) | 0.421(2) | 0.08(1) |
| H(12A) | 8f | 0.478(4) | 0.068(9) | 0.38(1) | 0.124 |
| H(12B) | 8f | 0.542(5) | 0.08(1) | 0.465(2) | 0.124 |
| H(12C) | 8f | 0.513(9) | 0.143(3) | 0.38(1) | 0.124 |
| C(2) | 8f | 0.4019(9) | 0.258(1) | 0.445(2) | 0.032(5) |
| C(3) | 8f | 0.3922(9) | 0.248(1) | 0.527(1) | 0.027(5) |
| C(31) | 8f | 0.376(1) | 0.171(1) | 0.559(2) | 0.039(6) |
| H(31A) | 8f | 0.344(1) | 0.183(1) | 0.569(2) | 0.047 |
| H(31B) | 8f | 0.362(1) | 0.130(1) | 0.508(2) | 0.047 |
| C(32) | 8f | 0.426(1) | 0.137(2) | 0.649(2) | 0.059(7) |
| H(32A) | 8f | 0.438(1) | 0.176(2) | 0.701(2) | 0.071 |
| H(32B) | 8f | 0.458(1) | 0.126(2) | 0.639(2) | 0.071 |
| C(33) | 8f | 0.406(2) | 0.055(2) | 0.674(3) | 0.11(1) |
| H(33A) | 8f | 0.41(1) | 0.063(6) | 0.73(1) | 0.169 |
| H(33B) | 8f | 0.432(7) | 0.012(4) | 0.68(2) | 0.169 |
| H(33C) | 8f | 0.368(5) | 0.042(9) | 0.624(9) | 0.169 |
| C(4) | 8f | 0.3977(9) | 0.331(1) | 0.571(1) | 0.031(5) |
| C(41) | 8f | 0.3772(9) | 0.341(1) | 0.642(1) | 0.033(6) |
| H(41A) | 8f | 0.3514(9) | 0.295(1) | 0.632(1) | 0.040 |
| H(41B) | 8f | 0.3551(9) | 0.392(1) | 0.627(1) | 0.004 |
| C(42) | 8f | 0.425(1) | 0.343(2) | 0.747(2) | 0.064(8) |
| H(42A) | 8f | 0.450(1) | 0.296(2) | 0.760(2) | 0.077 |
| H(42B) | 8f | 0.448(1) | 0.393(2) | 0.759(2) | 0.077 |
| C(43) | 8f | 0.405(1) | 0.342(2) | 0.814(2) | 0.073(9) |
| H(43A) | 8f | 0.437(2) | 0.35(1) | 0.878(3) | 0.109 |
| H(43B) | 8f | 0.387(8) | 0.289(5) | 0.81(1) | 0.109 |
| H(43C) | 8f | 0.377(6) | 0.385(8) | 0.798(9) | 0.109 |
| C(5) | 8f | 0.4210(9) | 0.395(1) | 0.549(1) | 0.032(6) |
| H(5) | 8f | 0.4339(9) | 0.386(1) | 0.507(1) | 0.038 |
| C(51) | 8f | 0.427(1) | 0.479(1) | 0.591(2) | 0.040(6) |
| H(51A) | 8f | 0.461(1) | 0.479(1) | 0.655(2) | 0.048 |
| H(51B) | 8f | 0.394(1) | 0.490(1) | 0.598(2) | 0.048 |
| C(52) | 8f | 0.433(1) | 0.552(2) | 0.535(2) | 0.060(8) |

Table 2. (Continued)

| Atom | Site | x | y | z | U _{iso} |
|--------|------|-----------|----------|----------|------------------|
| H(52A) | 8f | 0.466(1) | 0.541(2) | 0.527(2) | 0.072 |
| H(52B) | 8f | 0.398(1) | 0.553(2) | 0.471(2) | 0.072 |
| C(53) | 8f | 0.440(1) | 0.634(2) | 0.579(2) | 0.079(9) |
| H(53A) | 8f | 0.403(2) | 0.653(6) | 0.57(1) | 0.118 |
| H(53B) | 8f | 0.453(8) | 0.673(3) | 0.55(1) | 0.118 |
| H(53C) | 8f | 0.467(7) | 0.631(3) | 0.647(3) | 0.118 |
| C(61) | 8f | 0.288(1) | 0.160(2) | 0.338(2) | 0.059(8) |
| O(61) | 8f | 0.2507(8) | 0.140(1) | 0.354(1) | 0.072(5) |
| C(62) | 8f | 0.363(1) | 0.082(2) | 0.352(2) | 0.043(6) |
| O(62) | 8f | 0.3736(9) | 0.013(1) | 0.381(1) | 0.077(6) |
| C(7) | 8f | 0.3859(9) | 0.392(1) | 0.359(2) | 0.032(6) |
| O(7) | 8f | 0.4147(8) | 0.423(1) | 0.334(1) | 0.060(5) |
| C(81) | 8f | 0.288(1) | 0.269(2) | 0.160(2) | 0.041(6) |
| C(82) | 8f | 0.272(1) | 0.185(1) | 0.143(2) | 0.046(6) |
| H(82) | 8f | 0.236(1) | 0.165(1) | 0.127(2) | 0.055 |
| C(83) | 8f | 0.318(1) | 0.132(2) | 0.151(2) | 0.08(1) |
| H(83) | 8f | 0.317(1) | 0.075(2) | 0.141(2) | 0.098 |
| C(84) | 8f | 0.363(1) | 0.186(2) | 0.179(2) | 0.071(8) |
| H(84) | 8f | 0.399(1) | 0.170(2) | 0.195(2) | 0.085 |

Table 2. (Continued)

| Atom | Site | x | y | z | U _{iso} |
|--------|------|----------|----------|----------|------------------|
| C(85) | 8f | 0.348(1) | 0.266(2) | 0.182(2) | 0.057(7) |
| H(85) | 8f | 0.372(1) | 0.312(2) | 0.196(2) | 0.068 |
| C(91) | 8f | 0.256(1) | 0.381(2) | 0.274(2) | 0.045(6) |
| C(92) | 8f | 0.282(1) | 0.457(2) | 0.327(2) | 0.042(6) |
| H(92) | 8f | 0.296(1) | 0.501(2) | 0.306(2) | 0.051 |
| C(93) | 8f | 0.284(1) | 0.454(2) | 0.416(2) | 0.068(8) |
| H(93) | 8f | 0.299(1) | 0.494(2) | 0.463(2) | 0.082 |
| C(94) | 8f | 0.259(1) | 0.381(2) | 0.419(2) | 0.054(7) |
| H(94) | 8f | 0.255(1) | 0.363(2) | 0.470(2) | 0.064 |
| C(95) | 8f | 0.241(1) | 0.338(2) | 0.334(2) | 0.046(7) |
| H(95) | 8f | 0.222(1) | 0.287(2) | 0.319(2) | 0.055 |
| C(1A) | 8f | 0.167(1) | 0.322(2) | 0.076(2) | 0.069(8) |
| H(1A1) | 8f | 0.159(2) | 0.32(1) | 0.011(3) | 0.103 |
| H(1A2) | 8f | 0.142(1) | 0.364(5) | 0.08(1) | 0.103 |
| H(1A3) | 8f | 0.161(2) | 0.270(5) | 0.098(8) | 0.103 |
| C(1B) | 8f | 0.259(2) | 0.442(2) | 0.098(2) | 0.10(1) |
| H(1B1) | 8f | 0.233(7) | 0.487(6) | 0.09(2) | 0.148 |
| H(1B2) | 8f | 0.25(1) | 0.425(5) | 0.038(8) | 0.148 |
| H(1B3) | 8f | 0.298(4) | 0.46(1) | 0.140(8) | 0.148 |

Table 3. Final atomic coordinates and displacement parameters (in Å²)

| Atom | Site | x | y | z | U ₁₁ | U ₂₂ | U ₃₃ | U ₁₂ | U ₁₃ | U ₂₃ |
|-------|------|------------|------------|------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| W(1) | 8f | 0.34231(4) | 0.18962(6) | 0.30250(7) | 0.0387(7) | 0.0407(7) | 0.0413(7) | -0.0004(5) | 0.0192(5) | -0.0054(5) |
| Mo(1) | 8f | 0.34098(8) | 0.3460(1) | 0.4095(1) | 0.032(1) | 0.034(1) | 0.029(1) | 0.0010(9) | 0.017(1) | 0.0011(9) |
| Si(3) | 8f | 0.2419(3) | 0.3541(4) | 0.1534(4) | 0.046(4) | 0.039(4) | 0.030(4) | -0.001(3) | 0.014(3) | -0.001(3) |

References

- Kriebisch, K.-A.: Untersuchungen zu Synthese, Eigenschaften und Strukturen zweikerniger Cp'-SiMe₂-Cp'-verbrückter Metall-Carbonyl-Komplexe (M=Cr, Mo, W) und deren Alkin-Komplexe. Inaugural-Dissertation, University of Marburg, Germany 1991.
- Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; García-Granda, S.; Gould, R. O.; Smits, J. M. M.; Smykalla, C.: The DIRDIF program system. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands 1992.
- Admiraal, G.; Behm, H.; Smykalla, C.; Beurskens, P. T.: PATTY: Determination of heavy atom positions by Patterson Interpretation methods. *Z. Kristallogr. Suppl.* **6** (1992) 522.
- Grant, D. F.; Gabe, E. J. J.: The Analysis of Single-Crystal Bragg Reflections from Profile Measurements. *J. Appl. Crystallogr.* **11** (1978) 114-120.
- Lehman, M. S.; Larsen, F. K.: A Method for Location of the Peaks in Step-Scan-Measured Bragg Reflections. *Acta Crystallogr.* **A30** (1974) 580-584.
- Walker, N.; Stuart, D.: DIFABS. *Acta Crystallogr.* **A39** (1983) 158-166.
- Sheldrick, G. M.: SHELXL-93, Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1993.
- Spek, A. L.: PLATON, Program for computing and plotting molecular and crystal structures. *Acta Crystallogr.* **A46** (1990) C 34.