

Tricarbonyl(chlorodiphenylstannyl){ η^5 -[2-(dimethylamino)ethyl]cyclopentadienyl}molybdenum

Paul J. Fischer,^{a*} Kristina M. Krohn^a and Victor G. Young Jr^b

^aChemistry Department, Macalester College, 1600 Grand Avenue, Saint Paul, MN 55105, USA, and ^bX-ray Crystallographic Laboratory, Department of Chemistry, University of Minnesota, 207 Pleasant Street SE, Minneapolis, MN 55455, USA
Correspondence e-mail: fischer@macalester.edu

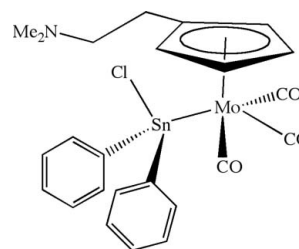
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.036; wR factor = 0.083; data-to-parameter ratio = 19.3.

Reaction of the tricarbonyl{ η^5 -[2-(dimethylamino)ethyl]cyclopentadienyl}molybdenum anion and dichloridodiphenylstannane affords the title compound, $[\text{MoSn}(\text{C}_6\text{H}_5)_2\text{Cl}(\text{C}_9\text{H}_{14}\text{N})(\text{CO})_3]$, which exhibits a four-legged piano-stool geometry with chloridodiphenylstannyl ligands unperturbed by the pendant 2-(dimethylamino)ethyl groups. The Mo—Sn bond length [2.7584 (5) Å] and the distortion of the tetrahedral tin coordination geometry are similar to those observed in related tin-substituted tricarbonylmolybdenum and -tungsten complexes.

Related literature

The synthesis of $\text{Mo}(\text{SnMe}_2\text{Cl})(\text{CO})_3(\eta^5\text{-Cp})$ was reported by Patil & Graham (1966). This methodology was extended to prepare $\text{Mo}(\text{SnPh}_2\text{Cl})(\text{CO})_3(\eta^5\text{-Cp})$ by Marks & Seyam (1974). Triphenyltin and tricyclohexyltin derivatives of $[\text{M}(\text{CO})_3(\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\text{NMe}_2)]^-$ ($M = \text{Mo}$ and W) were reported by Fischer *et al.* (2005). The structural characterization and reaction chemistry of a variety of half-sandwich molybdenum and tungsten chlorostannyl complexes have been reported by Braunschweig *et al.* (2007, 2009). The Lewis acidity of coordinatively saturated chlorostannyl ligands has been explored by Tang *et al.* (2005). Structural parameters that define four-legged piano-stool geometries were detailed by Kubáček *et al.* (1982).



Experimental

Crystal data

| | |
|--|-----------------------------------|
| $[\text{MoSn}(\text{C}_6\text{H}_5)_2\text{Cl}(\text{C}_9\text{H}_{14}\text{N})(\text{CO})_3]$ | $\gamma = 63.8560$ (10) $^\circ$ |
| $M_r = 624.52$ | $V = 1187.29$ (18) Å ³ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 8.8096$ (8) Å | Mo $K\alpha$ radiation |
| $b = 9.3458$ (8) Å | $\mu = 1.72$ mm ⁻¹ |
| $c = 16.1258$ (14) Å | $T = 173$ K |
| $\alpha = 89.392$ (2) $^\circ$ | $0.25 \times 0.12 \times 0.04$ mm |
| $\beta = 85.269$ (2) $^\circ$ | |

Data collection

| | |
|--|--|
| Bruker SMART CCD area-detector diffractometer | 14425 measured reflections |
| Absorption correction: multi-scan (SADABS; Bruker, 2003) | 5431 independent reflections |
| $T_{\min} = 0.673$, $T_{\max} = 0.935$ | 4081 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.056$ |

Refinement

| | |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | 282 parameters |
| $wR(F^2) = 0.083$ | H-atom parameters constrained |
| $S = 1.00$ | $\Delta\rho_{\max} = 0.71$ e Å ⁻³ |
| 5431 reflections | $\Delta\rho_{\min} = -0.56$ e Å ⁻³ |

Table 1

Selected bond lengths (Å).

| | | | |
|---------|-----------|---------|-------------|
| Sn1—C13 | 2.144 (4) | Sn1—Cl1 | 2.3914 (11) |
| Sn1—C19 | 2.146 (4) | | |

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: SI2168).

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Tricarbonyl(chlorodiphenylstannyl){ η^5 -[2-(dimethylamino)ethyl]cyclopentadienyl}molybdenum

P. J. Fischer, K. M. Krohn and V. G. Young Jr

Comment

The chemistry of half-sandwich molybdenum and tungsten chlorostannyl complexes is undergoing a revival since their discovery roughly forty years ago (Braunschweig *et al.*, 2009). Chlorostannyl ligands permit functionalization at tin in heterobimetallic complexes (Braunschweig *et al.*, 2007). The design of hypervalent tin ligands *via* intramolecular coordination of cyclopentadienyl-bound tethered donor groups is facilitated by chlorostannyl ligand Lewis acidity (Tang *et al.*, 2005).

The complexes $W(SnBu_2Cl)(CO)_3(\eta^5-Cp)$ (**2**) and $W(SnMe_2Cl)(CO)_3(\eta^5-Cp^*)$ (**3**) were structurally characterized by Braunschweig *et al.* (2007). The title complex **1** is the first crystallographically characterized substance of general formula $M(SnAr_2X)(CO)_3(\eta^5-Cp)$.

The Mo—Sn bond length in **1** is slightly shorter (Table 1) than the W—Sn distances found in **2** (2.7959 (5) Å) and **3** (2.7866 (3) Å), respectively. The steric impact of the $SnPh_3$ ligand relative to $SnPh_2Cl$ is minimal on the basis of Mo—Sn bond lengths; this separation is 2.8152 (3) Å in $Mo(SnPh_3)(CO)_3(\eta^5-C_5H_4CH_2CH_2NMe_2)$ (**4**) (Fischer *et al.*, 2005). The geometric parameters of **1-4** satisfy the requirements for four-legged piano-stool structures (Kubáček *et al.*, 1982).

The tetrahedral coordination geometry of the tin atoms in **1-3** are distorted more significantly than that of **4**. In **4**, the diagnostic six angles about tin range from 105.60 (9)° to 116.15 (6)°, while the corresponding angles in **1** range from 100.30 (11)° to 120.34 (11)°. In **1-3** the M—Sn—C angles are widened whereas the C—Sn—Cl angles are more acute in comparison to the ideal tetrahedral angle. The angles about the Sn atoms of **1** and **3** are very similar as the phenyl and methyl substituents, respectively, attempt to minimize steric hinderance with the $M(CO)_3(Cp)$ fragment. The C(13)—Sn(1)—Mo (116.32 (10)°) and C(19)—Sn(1)—Mo (120.34 (11)°) angles in **1** are similar to the corresponding angles found in **3** (118.56 (11), 118.31 (12)°). The widening of these angles is accommodated by compression of C—Sn—Cl angles. These angles in **1** (C(13)—Sn(1)—Cl(1), 100.30 (11)°; C(19)—Sn(1)—Cl(1), 101.25 (11)°) are similar to the corresponding angles found in **3** (99.75 (12), 100.48 (13)°).

Experimental

The following procedures were conducted under argon using standard techniques for handling air and moisture sensitive substances. THF (75 ml) was added to molybdenum hexacarbonyl (0.415 g, 1.57 mmol) and sodium ((2-dimethylamino)ethyl)cyclopentadienide (0.300 g, 1.88 mmol). The yellow solution was refluxed for 15 h. Addition of dichlorodiphenylstannane (0.540 g, 1.57 mmol) in THF (30 ml) resulted in a pale yellow suspension. A single molybdenum carbonyl complex was present in solution (based on IR spectroscopy) within 10 min. The suspension was filtered through Celite. THF was removed *in vacuo* revealing a yellow oil. The product was extracted with pentane (5 * 70 ml). Each colorless extract was filtered. The combined extracts were concentrated *in vacuo* until a yellow solid precipitated. The suspension (75 ml) was cooled and filtered at -70°C. The pale yellow, moderately air-sensitive solid was washed with pentane (-60°C,

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5 * 15 ml) and dried *in vacuo*. Recrystallization from pentane at -65°C afforded pale yellow microcrystals (0.461 g, 47%). X-ray quality single crystals of **1** were obtained from a supersaturated pentane solution.

Refinement

The structure was solved using Bruker *SHELXTL* and refined using Bruker *SHELXTL*. The space group was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0362$ and $wR2 = 0.0827$ (F^2 , all data).

Figures

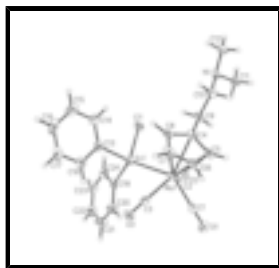


Fig. 1. Molecular structure of **1** (50% thermal ellipsoids).

Tricarbonyl-1 κ^3 C-chlorido-2 κ Cl-diphenyl-2 κ^2 C- {1 η^5 -[2-(dimethylamino)ethyl]cyclopentadienyl}molybdenum(0) tin(IV)(Mo—Sn)

Crystal data

| | |
|--|---|
| [MoSn(C ₆ H ₅) ₂ Cl(C ₉ H ₁₄ N)(CO) ₃] | $Z = 2$ |
| $M_r = 624.52$ | $F_{000} = 616$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.747 \text{ Mg m}^{-3}$ |
| Hall symbol: $-P 1$ | Mo $K\alpha$ radiation |
| $a = 8.8096$ (8) Å | $\lambda = 0.71073$ Å |
| $b = 9.3458$ (8) Å | Cell parameters from 2659 reflections |
| $c = 16.1258$ (14) Å | $\theta = 2.4\text{--}27.4^{\circ}$ |
| $\alpha = 89.392$ (2) $^{\circ}$ | $\mu = 1.72 \text{ mm}^{-1}$ |
| $\beta = 85.269$ (2) $^{\circ}$ | $T = 173 \text{ K}$ |
| $\gamma = 63.8560$ (10) $^{\circ}$ | Plate, colourless |
| $V = 1187.29$ (18) Å ³ | $0.25 \times 0.12 \times 0.04 \text{ mm}$ |

Data collection

| | |
|---|--|
| Bruker SMART CCD area-detector diffractometer | 5431 independent reflections |
| Radiation source: sealed tube | 4081 reflections with $I > 2\sigma(I)$ |

Monochromator: graphite $R_{\text{int}} = 0.056$
 $T = 173$ K $\theta_{\text{max}} = 27.5^\circ$
 φ and ω scans $\theta_{\text{min}} = 2.4^\circ$
 Absorption correction: multi-scan (SADABS; Bruker, 2003) $h = -11 \rightarrow 11$
 $T_{\text{min}} = 0.673$, $T_{\text{max}} = 0.935$ $k = -12 \rightarrow 12$
 14425 measured reflections $l = 0 \rightarrow 20$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.036$ H-atom parameters constrained
 $wR(F^2) = 0.083$ $w = 1/[\sigma^2(F_o^2) + (0.0315P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.00$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 5431 reflections $\Delta\rho_{\text{max}} = 0.71 \text{ e } \text{\AA}^{-3}$
 282 parameters $\Delta\rho_{\text{min}} = -0.56 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

Special details

Experimental. Anal. Calcd for $\text{C}_{24}\text{H}_{24}\text{ClMoNO}_3\text{Sn}$: C, 46.15; H, 3.87; N, 2.24. Found: C, 46.01, H, 3.78; N, 2.30.

IR (THF) $\nu(\text{CO})$: 2011 (s), 1944 (m, sh), 1912 (s) cm^{-1} ; (pentane) $\nu(\text{CO})$: 2015 (s), 1952 (m), 1919 (m) cm^{-1} ; (nujol) $\nu(\text{CO})$: 2011 (s), 1942 (m, sh), 1893 (s) cm^{-1} .

NMR (CD_2Cl_2 , 300 MHz) ^1H δ 7.78–7.54 (m, 4H, *ortho*, SnPh_2Cl), 7.48–7.32 (m, 6H, *meta/para*, SnPh_2Cl), 5.52 (app. t, $J=2.1$ Hz, 2H, Cp), 5.27 (app. t, $J=2.1$ Hz, 2H, Cp), 2.44–2.29 (m, 4H, CH_2CH_2), 2.14 (s, 6H, CH_3).

NMR (CD_2Cl_2 , 75.5 MHz) $^{13}\text{C}\{^1\text{H}\}$ δ 228.64 (s, *trans* CO), 224.35 (s, *cis* CO), 146.11 (s, *ipso* C), 135.30 (s, *ortho* C, $^{117,119}\text{Sn}$ - ^{13}C sat. (merged): 135.63, 134.97, $^2J_{\text{SnC}}=49.7$ Hz), 129.80 (s, *para*, $^{117,119}\text{Sn}$ - ^{13}C sat. (merged): 129.89, 129.73, $^4J_{\text{SnC}}=12.2$ Hz), 129.12 (s, *meta* C, $^{117,119}\text{Sn}$ - ^{13}C sat. (merged): 129.48, 128.76, $^3J_{\text{SnC}}=54.5$ Hz), 114.60 (s, quat. C, Cp), 91.75 (s, Cp), 89.65 (s, Cp), 61.15 (s, $\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$), 45.57 (s, $\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$), 27.08 (s, $\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$).

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. A crystal (approximate dimensions $1/4 \times 0.12 \times 0.04 \text{ mm}^3$) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a CCD area detector diffractometer for a data collection at 173 (2) K (*SMART*, Bruker, 2003). A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 48 reflections. The data collection was carried out using Mo $K\alpha$ radiation (graphite monochromator) with a frame time of 8 s and a detector distance of 4.8 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.77 \AA . Four major

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sections of frames were collected with 0.30° steps in ω at four different φ settings and a detector position of -28° in 2θ . The intensity data were corrected for absorption and decay (*SADABS*, Bruker, 2003). Final cell constants were calculated from 2659 strong reflections from the actual data collection after integration (*SAINT*, Bruker, 2003).

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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|---------------|----------------------------------|
| Mo1 | 0.58741 (4) | 0.72843 (4) | 0.66247 (2) | 0.01938 (9) |
| C1 | 0.6228 (5) | 0.6492 (5) | 0.5449 (3) | 0.0282 (9) |
| O1 | 0.6433 (4) | 0.6021 (4) | 0.47726 (19) | 0.0434 (8) |
| C2 | 0.8374 (5) | 0.6518 (5) | 0.6404 (3) | 0.0265 (9) |
| O2 | 0.9820 (4) | 0.5983 (4) | 0.6286 (2) | 0.0420 (8) |
| C3 | 0.4662 (5) | 0.9349 (5) | 0.6085 (2) | 0.0223 (9) |
| O3 | 0.3862 (3) | 1.0542 (3) | 0.57849 (17) | 0.0279 (7) |
| C4 | 0.6083 (5) | 0.5692 (5) | 0.7791 (2) | 0.0242 (9) |
| C5 | 0.5505 (5) | 0.5124 (5) | 0.7139 (3) | 0.0267 (9) |
| H5A | 0.6093 | 0.4105 | 0.6868 | 0.032* |
| C6 | 0.3894 (6) | 0.6330 (5) | 0.6953 (3) | 0.0353 (11) |
| H6A | 0.3222 | 0.6263 | 0.6538 | 0.042* |
| C7 | 0.3483 (5) | 0.7632 (5) | 0.7495 (3) | 0.0338 (11) |
| H7A | 0.2470 | 0.8602 | 0.7514 | 0.041* |
| C8 | 0.4815 (5) | 0.7269 (5) | 0.8010 (2) | 0.0267 (9) |
| H8A | 0.4862 | 0.7953 | 0.8428 | 0.032* |
| C9 | 0.7613 (5) | 0.4754 (5) | 0.8262 (3) | 0.0273 (9) |
| H9A | 0.8019 | 0.5484 | 0.8498 | 0.033* |
| H9B | 0.8542 | 0.3974 | 0.7881 | 0.033* |
| C10 | 0.7123 (5) | 0.3881 (5) | 0.8963 (3) | 0.0296 (10) |
| H10A | 0.6087 | 0.4656 | 0.9289 | 0.035* |
| H10B | 0.6844 | 0.3072 | 0.8714 | 0.035* |
| N1 | 0.8441 (4) | 0.3094 (4) | 0.9527 (2) | 0.0262 (8) |
| C11 | 0.9863 (5) | 0.1694 (5) | 0.9137 (3) | 0.0304 (10) |
| H11A | 1.0350 | 0.2008 | 0.8641 | 0.046* |
| H11B | 0.9466 | 0.0916 | 0.8977 | 0.046* |
| H11C | 1.0730 | 0.1215 | 0.9531 | 0.046* |
| C12 | 0.7735 (6) | 0.2630 (5) | 1.0275 (3) | 0.0370 (11) |
| H12A | 0.6813 | 0.3579 | 1.0552 | 0.055* |
| H12B | 0.8626 | 0.2116 | 1.0654 | 0.055* |
| H12C | 0.7293 | 0.1883 | 1.0121 | 0.055* |
| Sn1 | 0.66959 (3) | 0.95053 (3) | 0.732290 (16) | 0.02009 (8) |
| Cl1 | 0.86682 (14) | 0.82366 (13) | 0.83383 (7) | 0.0336 (3) |
| C13 | 0.4703 (5) | 1.1368 (5) | 0.8078 (2) | 0.0235 (9) |
| C14 | 0.4566 (6) | 1.1328 (5) | 0.8939 (3) | 0.0321 (10) |
| H14A | 0.5345 | 1.0426 | 0.9209 | 0.038* |

| | | | | |
|------|------------|------------|------------|-------------|
| C15 | 0.3298 (6) | 1.2593 (6) | 0.9416 (3) | 0.0418 (12) |
| H15A | 0.3210 | 1.2541 | 1.0005 | 0.050* |
| C16 | 0.2193 (6) | 1.3896 (6) | 0.9037 (3) | 0.0450 (13) |
| H16A | 0.1352 | 1.4767 | 0.9364 | 0.054* |
| C17 | 0.2282 (6) | 1.3965 (5) | 0.8176 (3) | 0.0396 (12) |
| H17A | 0.1492 | 1.4872 | 0.7914 | 0.047* |
| C18 | 0.3534 (5) | 1.2702 (5) | 0.7697 (3) | 0.0301 (10) |
| H18A | 0.3592 | 1.2747 | 0.7107 | 0.036* |
| C19 | 0.7998 (5) | 1.0601 (4) | 0.6573 (2) | 0.0234 (9) |
| C20 | 0.7633 (6) | 1.1042 (5) | 0.5768 (3) | 0.0353 (11) |
| H20A | 0.6844 | 1.0783 | 0.5517 | 0.042* |
| C21 | 0.8401 (6) | 1.1859 (6) | 0.5316 (3) | 0.0410 (12) |
| H21A | 0.8138 | 1.2145 | 0.4760 | 0.049* |
| C22 | 0.9535 (6) | 1.2252 (5) | 0.5667 (3) | 0.0379 (12) |
| H22A | 1.0046 | 1.2824 | 0.5360 | 0.045* |
| C23 | 0.9928 (6) | 1.1815 (5) | 0.6468 (3) | 0.0347 (11) |
| H23A | 1.0730 | 1.2068 | 0.6711 | 0.042* |
| C24 | 0.9161 (5) | 1.1009 (5) | 0.6922 (3) | 0.0286 (10) |
| H24A | 0.9428 | 1.0728 | 0.7477 | 0.034* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|---------------|--------------|
| Mo1 | 0.02176 (19) | 0.01690 (18) | 0.01947 (19) | -0.00855 (15) | -0.00209 (14) | 0.00347 (14) |
| C1 | 0.035 (3) | 0.022 (2) | 0.028 (2) | -0.012 (2) | -0.0045 (19) | 0.0042 (18) |
| O1 | 0.061 (2) | 0.0359 (19) | 0.0279 (18) | -0.0161 (17) | -0.0040 (16) | -0.0046 (15) |
| C2 | 0.030 (2) | 0.022 (2) | 0.028 (2) | -0.0111 (19) | -0.0049 (19) | 0.0037 (17) |
| O2 | 0.0278 (18) | 0.045 (2) | 0.048 (2) | -0.0117 (16) | 0.0004 (15) | -0.0022 (16) |
| C3 | 0.027 (2) | 0.026 (2) | 0.016 (2) | -0.0140 (19) | -0.0008 (17) | -0.0027 (17) |
| O3 | 0.0258 (16) | 0.0235 (15) | 0.0288 (16) | -0.0053 (13) | -0.0058 (13) | 0.0093 (13) |
| C4 | 0.023 (2) | 0.024 (2) | 0.027 (2) | -0.0111 (18) | -0.0017 (17) | 0.0083 (17) |
| C5 | 0.032 (2) | 0.019 (2) | 0.035 (2) | -0.0164 (19) | -0.0063 (19) | 0.0107 (18) |
| C6 | 0.031 (2) | 0.042 (3) | 0.042 (3) | -0.023 (2) | -0.013 (2) | 0.023 (2) |
| C7 | 0.021 (2) | 0.033 (3) | 0.039 (3) | -0.005 (2) | 0.0062 (19) | 0.014 (2) |
| C8 | 0.033 (2) | 0.025 (2) | 0.020 (2) | -0.0130 (19) | 0.0052 (18) | 0.0064 (17) |
| C9 | 0.032 (2) | 0.026 (2) | 0.029 (2) | -0.018 (2) | -0.0093 (19) | 0.0076 (18) |
| C10 | 0.026 (2) | 0.029 (2) | 0.034 (2) | -0.0116 (19) | -0.0073 (19) | 0.0117 (19) |
| N1 | 0.0263 (19) | 0.0221 (18) | 0.0282 (19) | -0.0085 (15) | -0.0057 (15) | 0.0090 (15) |
| C11 | 0.034 (3) | 0.022 (2) | 0.034 (3) | -0.010 (2) | -0.009 (2) | 0.0043 (18) |
| C12 | 0.042 (3) | 0.036 (3) | 0.029 (2) | -0.015 (2) | -0.002 (2) | 0.014 (2) |
| Sn1 | 0.02179 (15) | 0.01760 (14) | 0.02049 (15) | -0.00814 (12) | -0.00317 (11) | 0.00216 (11) |
| C11 | 0.0381 (6) | 0.0298 (6) | 0.0355 (6) | -0.0152 (5) | -0.0179 (5) | 0.0096 (5) |
| C13 | 0.024 (2) | 0.022 (2) | 0.027 (2) | -0.0132 (18) | 0.0016 (17) | -0.0024 (17) |
| C14 | 0.037 (3) | 0.032 (2) | 0.027 (2) | -0.016 (2) | -0.001 (2) | -0.0019 (19) |
| C15 | 0.047 (3) | 0.051 (3) | 0.030 (3) | -0.026 (3) | 0.011 (2) | -0.012 (2) |
| C16 | 0.038 (3) | 0.038 (3) | 0.055 (3) | -0.018 (2) | 0.023 (2) | -0.024 (3) |
| C17 | 0.034 (3) | 0.022 (2) | 0.058 (3) | -0.009 (2) | 0.003 (2) | -0.002 (2) |
| C18 | 0.032 (2) | 0.023 (2) | 0.034 (2) | -0.013 (2) | 0.0053 (19) | -0.0018 (19) |

supplementary materials

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|-----|-----------|-----------|-----------|--------------|--------------|--------------|
| C19 | 0.021 (2) | 0.019 (2) | 0.027 (2) | -0.0069 (17) | 0.0012 (17) | 0.0003 (17) |
| C20 | 0.037 (3) | 0.042 (3) | 0.032 (3) | -0.021 (2) | -0.010 (2) | 0.008 (2) |
| C21 | 0.050 (3) | 0.053 (3) | 0.026 (2) | -0.028 (3) | -0.003 (2) | 0.011 (2) |
| C22 | 0.042 (3) | 0.030 (2) | 0.041 (3) | -0.019 (2) | 0.016 (2) | 0.001 (2) |
| C23 | 0.030 (2) | 0.034 (3) | 0.044 (3) | -0.020 (2) | 0.006 (2) | -0.006 (2) |
| C24 | 0.025 (2) | 0.029 (2) | 0.030 (2) | -0.0112 (19) | -0.0011 (18) | -0.0022 (19) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|-------------|-------------|
| Mo1—C3 | 1.981 (4) | C11—H11B | 0.9800 |
| Mo1—C1 | 1.992 (4) | C11—H11C | 0.9800 |
| Mo1—C2 | 1.994 (4) | C12—H12A | 0.9800 |
| Mo1—C6 | 2.308 (4) | C12—H12B | 0.9800 |
| Mo1—C5 | 2.317 (4) | C12—H12C | 0.9800 |
| Mo1—C7 | 2.331 (4) | Sn1—C13 | 2.144 (4) |
| Mo1—C8 | 2.351 (4) | Sn1—C19 | 2.146 (4) |
| Mo1—C4 | 2.353 (4) | Sn1—Cl1 | 2.3914 (11) |
| Mo1—Sn1 | 2.7584 (5) | C13—C14 | 1.384 (6) |
| C1—O1 | 1.150 (5) | C13—C18 | 1.396 (6) |
| C2—O2 | 1.145 (5) | C14—C15 | 1.397 (6) |
| C3—O3 | 1.152 (4) | C14—H14A | 0.9500 |
| C4—C5 | 1.408 (6) | C15—C16 | 1.357 (7) |
| C4—C8 | 1.428 (5) | C15—H15A | 0.9500 |
| C4—C9 | 1.509 (5) | C16—C17 | 1.386 (7) |
| C5—C6 | 1.424 (6) | C16—H16A | 0.9500 |
| C5—H5A | 0.9500 | C17—C18 | 1.392 (6) |
| C6—C7 | 1.399 (6) | C17—H17A | 0.9500 |
| C6—H6A | 0.9500 | C18—H18A | 0.9500 |
| C7—C8 | 1.409 (6) | C19—C20 | 1.379 (6) |
| C7—H7A | 0.9500 | C19—C24 | 1.398 (5) |
| C8—H8A | 0.9500 | C20—C21 | 1.390 (6) |
| C9—C10 | 1.530 (5) | C20—H20A | 0.9500 |
| C9—H9A | 0.9900 | C21—C22 | 1.369 (6) |
| C9—H9B | 0.9900 | C21—H21A | 0.9500 |
| C10—N1 | 1.457 (5) | C22—C23 | 1.376 (6) |
| C10—H10A | 0.9900 | C22—H22A | 0.9500 |
| C10—H10B | 0.9900 | C23—C24 | 1.384 (6) |
| N1—C11 | 1.456 (5) | C23—H23A | 0.9500 |
| N1—C12 | 1.461 (5) | C24—H24A | 0.9500 |
| C11—H11A | 0.9800 | | |
| C3—Mo1—C1 | 81.02 (16) | C4—C9—C10 | 109.1 (3) |
| C3—Mo1—C2 | 109.86 (16) | C4—C9—H9A | 109.9 |
| C1—Mo1—C2 | 79.28 (17) | C10—C9—H9A | 109.9 |
| C3—Mo1—C6 | 106.33 (16) | C4—C9—H9B | 109.9 |
| C1—Mo1—C6 | 91.77 (17) | C10—C9—H9B | 109.9 |
| C2—Mo1—C6 | 140.66 (16) | H9A—C9—H9B | 108.3 |
| C3—Mo1—C5 | 141.90 (15) | N1—C10—C9 | 114.0 (3) |
| C1—Mo1—C5 | 93.04 (16) | N1—C10—H10A | 108.8 |
| C2—Mo1—C5 | 105.82 (15) | C9—C10—H10A | 108.8 |

| | | | |
|------------|-------------|---------------|-------------|
| C6—Mo1—C5 | 35.87 (14) | N1—C10—H10B | 108.8 |
| C3—Mo1—C7 | 92.95 (15) | C9—C10—H10B | 108.8 |
| C1—Mo1—C7 | 122.26 (17) | H10A—C10—H10B | 107.6 |
| C2—Mo1—C7 | 151.56 (16) | C11—N1—C10 | 112.1 (3) |
| C6—Mo1—C7 | 35.08 (16) | C11—N1—C12 | 109.4 (3) |
| C5—Mo1—C7 | 58.61 (15) | C10—N1—C12 | 110.0 (3) |
| C3—Mo1—C8 | 113.36 (15) | N1—C11—H11A | 109.5 |
| C1—Mo1—C8 | 149.29 (16) | N1—C11—H11B | 109.5 |
| C2—Mo1—C8 | 117.21 (16) | H11A—C11—H11B | 109.5 |
| C6—Mo1—C8 | 58.71 (16) | N1—C11—H11C | 109.5 |
| C5—Mo1—C8 | 58.52 (15) | H11A—C11—H11C | 109.5 |
| C7—Mo1—C8 | 35.02 (15) | H11B—C11—H11C | 109.5 |
| C3—Mo1—C4 | 148.50 (15) | N1—C12—H12A | 109.5 |
| C1—Mo1—C4 | 124.20 (15) | N1—C12—H12B | 109.5 |
| C2—Mo1—C4 | 94.55 (15) | H12A—C12—H12B | 109.5 |
| C6—Mo1—C4 | 59.16 (14) | N1—C12—H12C | 109.5 |
| C5—Mo1—C4 | 35.09 (14) | H12A—C12—H12C | 109.5 |
| C7—Mo1—C4 | 58.64 (14) | H12B—C12—H12C | 109.5 |
| C8—Mo1—C4 | 35.36 (13) | C13—Sn1—C19 | 106.86 (14) |
| C3—Mo1—Sn1 | 71.39 (11) | C13—Sn1—Cl1 | 100.30 (11) |
| C1—Mo1—Sn1 | 129.99 (12) | C19—Sn1—Cl1 | 101.25 (11) |
| C2—Mo1—Sn1 | 72.45 (11) | C13—Sn1—Mo1 | 116.32 (10) |
| C6—Mo1—Sn1 | 135.31 (13) | C19—Sn1—Mo1 | 120.34 (11) |
| C5—Mo1—Sn1 | 133.76 (11) | Cl1—Sn1—Mo1 | 108.86 (3) |
| C7—Mo1—Sn1 | 100.50 (12) | C14—C13—C18 | 118.4 (4) |
| C8—Mo1—Sn1 | 80.70 (10) | C14—C13—Sn1 | 122.3 (3) |
| C4—Mo1—Sn1 | 98.79 (10) | C18—C13—Sn1 | 119.3 (3) |
| O1—C1—Mo1 | 179.4 (4) | C13—C14—C15 | 121.0 (4) |
| O2—C2—Mo1 | 175.7 (4) | C13—C14—H14A | 119.5 |
| O3—C3—Mo1 | 175.6 (3) | C15—C14—H14A | 119.5 |
| C5—C4—C8 | 107.1 (4) | C16—C15—C14 | 120.0 (4) |
| C5—C4—C9 | 127.1 (4) | C16—C15—H15A | 120.0 |
| C8—C4—C9 | 125.3 (4) | C14—C15—H15A | 120.0 |
| C5—C4—Mo1 | 71.0 (2) | C15—C16—C17 | 120.5 (4) |
| C8—C4—Mo1 | 72.2 (2) | C15—C16—H16A | 119.8 |
| C9—C4—Mo1 | 128.7 (3) | C17—C16—H16A | 119.8 |
| C4—C5—C6 | 108.7 (4) | C16—C17—C18 | 119.8 (4) |
| C4—C5—Mo1 | 73.9 (2) | C16—C17—H17A | 120.1 |
| C6—C5—Mo1 | 71.7 (2) | C18—C17—H17A | 120.1 |
| C4—C5—H5A | 125.7 | C17—C18—C13 | 120.4 (4) |
| C6—C5—H5A | 125.7 | C17—C18—H18A | 119.8 |
| Mo1—C5—H5A | 120.5 | C13—C18—H18A | 119.8 |
| C7—C6—C5 | 107.4 (4) | C20—C19—C24 | 117.8 (4) |
| C7—C6—Mo1 | 73.4 (2) | C20—C19—Sn1 | 122.2 (3) |
| C5—C6—Mo1 | 72.4 (2) | C24—C19—Sn1 | 119.8 (3) |
| C7—C6—H6A | 126.3 | C19—C20—C21 | 121.1 (4) |
| C5—C6—H6A | 126.3 | C19—C20—H20A | 119.4 |
| Mo1—C6—H6A | 119.8 | C21—C20—H20A | 119.4 |
| C6—C7—C8 | 108.9 (4) | C22—C21—C20 | 120.4 (4) |

supplementary materials

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| C6—C7—Mo1 | 71.6 (2) | C22—C21—H21A | 119.8 |
| C8—C7—Mo1 | 73.2 (2) | C20—C21—H21A | 119.8 |
| C6—C7—H7A | 125.5 | C21—C22—C23 | 119.6 (4) |
| C8—C7—H7A | 125.5 | C21—C22—H22A | 120.2 |
| Mo1—C7—H7A | 121.3 | C23—C22—H22A | 120.2 |
| C7—C8—C4 | 107.9 (4) | C22—C23—C24 | 120.3 (4) |
| C7—C8—Mo1 | 71.7 (2) | C22—C23—H23A | 119.9 |
| C4—C8—Mo1 | 72.4 (2) | C24—C23—H23A | 119.9 |
| C7—C8—H8A | 126.0 | C23—C24—C19 | 120.9 (4) |
| C4—C8—H8A | 126.0 | C23—C24—H24A | 119.6 |
| Mo1—C8—H8A | 121.5 | C19—C24—H24A | 119.6 |
| C3—Mo1—C1—O1 | -152 (36) | C4—Mo1—C7—C6 | -79.8 (3) |
| C2—Mo1—C1—O1 | 96 (36) | Sn1—Mo1—C7—C6 | -173.8 (2) |
| C6—Mo1—C1—O1 | -46 (36) | C3—Mo1—C7—C8 | -128.2 (3) |
| C5—Mo1—C1—O1 | -10 (36) | C1—Mo1—C7—C8 | 150.6 (2) |
| C7—Mo1—C1—O1 | -64 (36) | C2—Mo1—C7—C8 | 15.9 (5) |
| C8—Mo1—C1—O1 | -31 (36) | C6—Mo1—C7—C8 | 117.2 (4) |
| C4—Mo1—C1—O1 | 7(36) | C5—Mo1—C7—C8 | 78.7 (3) |
| Sn1—Mo1—C1—O1 | 152 (36) | C4—Mo1—C7—C8 | 37.4 (2) |
| C3—Mo1—C2—O2 | -157 (5) | Sn1—Mo1—C7—C8 | -56.6 (2) |
| C1—Mo1—C2—O2 | -81 (5) | C6—C7—C8—C4 | -0.8 (4) |
| C6—Mo1—C2—O2 | -1(5) | Mo1—C7—C8—C4 | -63.9 (3) |
| C5—Mo1—C2—O2 | 9(5) | C6—C7—C8—Mo1 | 63.1 (3) |
| C7—Mo1—C2—O2 | 61 (5) | C5—C4—C8—C7 | 0.5 (4) |
| C8—Mo1—C2—O2 | 72 (5) | C9—C4—C8—C7 | -171.4 (4) |
| C4—Mo1—C2—O2 | 43 (5) | Mo1—C4—C8—C7 | 63.4 (3) |
| Sn1—Mo1—C2—O2 | 141 (5) | C5—C4—C8—Mo1 | -62.9 (3) |
| C1—Mo1—C3—O3 | 98 (4) | C9—C4—C8—Mo1 | 125.2 (4) |
| C2—Mo1—C3—O3 | 173 (4) | C3—Mo1—C8—C7 | 58.8 (3) |
| C6—Mo1—C3—O3 | 9(4) | C1—Mo1—C8—C7 | -54.5 (4) |
| C5—Mo1—C3—O3 | 14 (4) | C2—Mo1—C8—C7 | -171.6 (3) |
| C7—Mo1—C3—O3 | -24 (4) | C6—Mo1—C8—C7 | -36.7 (2) |
| C8—Mo1—C3—O3 | -54 (4) | C5—Mo1—C8—C7 | -79.0 (3) |
| C4—Mo1—C3—O3 | -48 (4) | C4—Mo1—C8—C7 | -116.4 (4) |
| Sn1—Mo1—C3—O3 | -125 (4) | Sn1—Mo1—C8—C7 | 123.7 (2) |
| C3—Mo1—C4—C5 | 107.3 (3) | C3—Mo1—C8—C4 | 175.1 (2) |
| C1—Mo1—C4—C5 | -31.1 (3) | C1—Mo1—C8—C4 | 61.9 (4) |
| C2—Mo1—C4—C5 | -111.3 (3) | C2—Mo1—C8—C4 | -55.2 (3) |
| C6—Mo1—C4—C5 | 37.6 (3) | C6—Mo1—C8—C4 | 79.6 (3) |
| C7—Mo1—C4—C5 | 78.8 (3) | C5—Mo1—C8—C4 | 37.3 (2) |
| C8—Mo1—C4—C5 | 115.9 (3) | C7—Mo1—C8—C4 | 116.4 (4) |
| Sn1—Mo1—C4—C5 | 175.8 (2) | Sn1—Mo1—C8—C4 | -119.9 (2) |
| C3—Mo1—C4—C8 | -8.6 (4) | C5—C4—C9—C10 | -84.0 (5) |
| C1—Mo1—C4—C8 | -147.0 (3) | C8—C4—C9—C10 | 86.2 (5) |
| C2—Mo1—C4—C8 | 132.9 (3) | Mo1—C4—C9—C10 | -178.8 (3) |
| C6—Mo1—C4—C8 | -78.2 (3) | C4—C9—C10—N1 | -172.7 (3) |
| C5—Mo1—C4—C8 | -115.9 (3) | C9—C10—N1—C11 | -71.6 (4) |
| C7—Mo1—C4—C8 | -37.0 (2) | C9—C10—N1—C12 | 166.5 (4) |
| Sn1—Mo1—C4—C8 | 60.0 (2) | C3—Mo1—Sn1—C13 | 69.08 (17) |

| | | | |
|---------------|------------|-----------------|--------------|
| C3—Mo1—C4—C9 | -129.9 (4) | C1—Mo1—Sn1—C13 | 129.3 (2) |
| C1—Mo1—C4—C9 | 91.7 (4) | C2—Mo1—Sn1—C13 | -172.07 (17) |
| C2—Mo1—C4—C9 | 11.6 (4) | C6—Mo1—Sn1—C13 | -25.59 (19) |
| C6—Mo1—C4—C9 | 160.4 (4) | C5—Mo1—Sn1—C13 | -76.73 (19) |
| C5—Mo1—C4—C9 | 122.8 (5) | C7—Mo1—Sn1—C13 | -20.51 (16) |
| C7—Mo1—C4—C9 | -158.3 (4) | C8—Mo1—Sn1—C13 | -49.54 (16) |
| C8—Mo1—C4—C9 | -121.3 (5) | C4—Mo1—Sn1—C13 | -80.05 (15) |
| Sn1—Mo1—C4—C9 | -61.3 (4) | C3—Mo1—Sn1—C19 | -62.57 (17) |
| C8—C4—C5—C6 | -0.1 (4) | C1—Mo1—Sn1—C19 | -2.39 (19) |
| C9—C4—C5—C6 | 171.6 (4) | C2—Mo1—Sn1—C19 | 56.28 (17) |
| Mo1—C4—C5—C6 | -63.8 (3) | C6—Mo1—Sn1—C19 | -157.24 (19) |
| C8—C4—C5—Mo1 | 63.7 (3) | C5—Mo1—Sn1—C19 | 151.62 (19) |
| C9—C4—C5—Mo1 | -124.6 (4) | C7—Mo1—Sn1—C19 | -152.15 (16) |
| C3—Mo1—C5—C4 | -126.0 (3) | C8—Mo1—Sn1—C19 | 178.81 (16) |
| C1—Mo1—C5—C4 | 154.7 (3) | C4—Mo1—Sn1—C19 | 148.31 (16) |
| C2—Mo1—C5—C4 | 74.9 (3) | C3—Mo1—Sn1—C11 | -178.60 (12) |
| C6—Mo1—C5—C4 | -116.5 (4) | C1—Mo1—Sn1—C11 | -118.42 (16) |
| C7—Mo1—C5—C4 | -78.9 (3) | C2—Mo1—Sn1—C11 | -59.75 (12) |
| C8—Mo1—C5—C4 | -37.6 (2) | C6—Mo1—Sn1—C11 | 86.73 (15) |
| Sn1—Mo1—C5—C4 | -5.7 (3) | C5—Mo1—Sn1—C11 | 35.59 (15) |
| C3—Mo1—C5—C6 | -9.5 (4) | C7—Mo1—Sn1—C11 | 91.81 (11) |
| C1—Mo1—C5—C6 | -88.8 (3) | C8—Mo1—Sn1—C11 | 62.78 (11) |
| C2—Mo1—C5—C6 | -168.5 (3) | C4—Mo1—Sn1—C11 | 32.28 (10) |
| C7—Mo1—C5—C6 | 37.6 (3) | C19—Sn1—C13—C14 | -123.0 (3) |
| C8—Mo1—C5—C6 | 78.9 (3) | C11—Sn1—C13—C14 | -17.8 (3) |
| C4—Mo1—C5—C6 | 116.5 (4) | Mo1—Sn1—C13—C14 | 99.3 (3) |
| Sn1—Mo1—C5—C6 | 110.8 (3) | C19—Sn1—C13—C18 | 54.6 (3) |
| C4—C5—C6—C7 | -0.4 (5) | C11—Sn1—C13—C18 | 159.8 (3) |
| Mo1—C5—C6—C7 | -65.5 (3) | Mo1—Sn1—C13—C18 | -83.1 (3) |
| C4—C5—C6—Mo1 | 65.1 (3) | C18—C13—C14—C15 | -0.5 (6) |
| C3—Mo1—C6—C7 | -71.1 (3) | Sn1—C13—C14—C15 | 177.1 (3) |
| C1—Mo1—C6—C7 | -152.3 (3) | C13—C14—C15—C16 | -0.9 (7) |
| C2—Mo1—C6—C7 | 132.6 (3) | C14—C15—C16—C17 | 1.7 (7) |
| C5—Mo1—C6—C7 | 115.0 (4) | C15—C16—C17—C18 | -1.2 (7) |
| C8—Mo1—C6—C7 | 36.7 (2) | C16—C17—C18—C13 | -0.2 (7) |
| C4—Mo1—C6—C7 | 78.2 (3) | C14—C13—C18—C17 | 1.1 (6) |
| Sn1—Mo1—C6—C7 | 8.7 (3) | Sn1—C13—C18—C17 | -176.6 (3) |
| C3—Mo1—C6—C5 | 173.9 (3) | C13—Sn1—C19—C20 | -96.5 (4) |
| C1—Mo1—C6—C5 | 92.7 (3) | C11—Sn1—C19—C20 | 158.9 (3) |
| C2—Mo1—C6—C5 | 17.6 (4) | Mo1—Sn1—C19—C20 | 39.0 (4) |
| C7—Mo1—C6—C5 | -115.0 (4) | C13—Sn1—C19—C24 | 78.2 (3) |
| C8—Mo1—C6—C5 | -78.3 (3) | C11—Sn1—C19—C24 | -26.4 (3) |
| C4—Mo1—C6—C5 | -36.8 (2) | Mo1—Sn1—C19—C24 | -146.3 (3) |
| Sn1—Mo1—C6—C5 | -106.3 (3) | C24—C19—C20—C21 | 0.3 (7) |
| C5—C6—C7—C8 | 0.7 (5) | Sn1—C19—C20—C21 | 175.1 (3) |
| Mo1—C6—C7—C8 | -64.2 (3) | C19—C20—C21—C22 | -0.4 (7) |
| C5—C6—C7—Mo1 | 64.9 (3) | C20—C21—C22—C23 | 0.9 (7) |
| C3—Mo1—C7—C6 | 114.6 (3) | C21—C22—C23—C24 | -1.2 (7) |
| C1—Mo1—C7—C6 | 33.4 (3) | C22—C23—C24—C19 | 1.0 (6) |

supplementary materials

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| C2—Mo1—C7—C6 | -101.3 (4) | C20—C19—C24—C23 | -0.6 (6) |
| C5—Mo1—C7—C6 | -38.5 (2) | Sn1—C19—C24—C23 | -175.5 (3) |
| C8—Mo1—C7—C6 | -117.2 (4) | | |

Fig. 1

