Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

for

Unusual Coordination of Tetrylenes to Molybdenum Carbonyl Fragments

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1. General experimental procedures

All reactions were performed with the use of modified Schlenk techniques with additional manipulations using a Vacuum Atmospheres OMNI-Lab drybox. All solvents were dried over an alumina column, followed by storage over a potassium or sodium mirror, and degassed three times (freeze-pump-thaw) prior to use. $Ge(SAr^{Me6})_2$ and $Sn(SAr^{Me6})_2$ (Ar^{Me6} = C_6H_3-2,6(C_6H_2-2,4,6-Me_3)$_2$)[1] as well as (Bicyclo[2.2.1]hepta-2,5-diene)tetracarbonylmolybdenum(0)[2] were prepared according to literature procedures. ^1H and ^{13}C NMR data were obtained on a Varian Inova 600 MHz spectrometer. The ^1H and ^{13}C NMR spectra were referenced to the residual portio d^6-benzene or CD_2Cl_2 and/or trace silicone grease (δ = 0.29 ppm in C_6D_6; δ = 0.09 ppm in CD_2Cl_2). ^{119}Sn (223.63 MHz) were acquired on a Varian 600 MHz instrument and were referenced to $SnBu^n_4$ in C_6D_6 (δ = -11.7 ppm). IR spectra were recorded as Nujol mulls between CsI plates on a Perkin-Elmer 1430 spectrophotometer. UV-visible spectra were recorded as dilute hexane or THF solutions in 3.5 mL quartz cuvettes using an Olis 14 spectrophotometer. Melting points were measured in glass capillaries sealed under N_2 by using a Mel-Temp II apparatus and are uncorrected.

2. Synthesis and spectroscopic data for compound 3-5

2.1 $Ge(SAr^{Me6})_2Mo(CO)_3$·THF (3)

To a solution of $Ge(SAr^{Pri4})_2$ (0.532 g, 0.5 mmol) in THF (ca. 30 mL) was added a solution of (bicyclo[2.2.1]hepta-2,5-diene)tetracarbonylmolybdenum(0) (0.150 g, 0.5 mmol) in THF (ca. 30 ml). The solution became orange-red and was stirred for 2h at room temperature after which time all volatile materials were removed under reduced pressure.

The orange-red residue was extracted in hot hexanes (ca. 40 mL) and was filtered with a filter-tipped cannula and subsequently concentrated under reduced pressure. Concentration to ca. 7 mL and storage of the solution at ca. -18 °C afforded red block-like crystals of **3** (0.030 g, 0.03 mmol, 6 %).

M. p. 165-167 °C (decomp.).

^{1}H NMR (599.7 MHz, *d*^{6}-benzene, 294 K): 1.31 (m, CH_2(3,4), THF), 2.17 (s, 24H, *o*-CH_3), 2.26 (s, 12H, *p*-CH_3), 2.97 (m, CH_2(2,5), THF), 6.71 (d, 4H, *m*-C_6H_3, $^3J_{H,H}$ = 7.54 Hz), 6.83 (s, 4H, $C_6H_2Me_3$), 6.84 (s, 4H, $C_6H_2Me_3$), 6.90 (t, 2H, *p*-C_6H_3, $^3J_{H,H}$ = 7.51 Hz).

^{13}C{^{1}H} NMR (599.7 MHz, d6-benzene, 294 K): 21.26 (*o*-CH_3), 21.31 (*o*-CH_3), 21.48 (*p*-CH_3), 25.67 (CH_2(3,4), THF), 74.48 (CH_2(2,5), THF), 126.85 (*p*-C_6H_3), 128.35 (*m*-$C_6H_2Me_3$), 129.29 (*m*-C_6H_3), 129.49 (*p*-C_6H_3), 130.52 (*o*-C_6H_3), 136.69 (*o*-$C_6H_2Me_3$), 136.96 (*p*-$C_6H_2Me_3$), 139.02 (*i*-$C_6H_2Me_3$), 143.98 (*i*-C_6H_3), 214.39 (CO, axial), 217.03 (CO, equatorial).

IR (nujol, CsI plates, cm^{-1}): 2930 (s, broad), 2820 (s, medium), 2068 (m, sharp), 1959 (w, sharp), 1888 (w, sharp), 1878 (w, sharp), 1948 (s, sharp), 1855 (s, sharp), 1820 (s, sharp), 1612 (m, medium), 1570 (m, medium), 1455 (s, broad), 1375 (s, medium), 1260 (s, sharp), 1060 (s, broad), 870 (m, medium), 850 (m, sharp), 805 (s, broad), 745 (m, sharp), 724 (w, sharp), 695 (w, broad), 634 (w, medium), 622 (w, broad), 480 (w, broad), 495 (w, broad), 455 (w, medium), 390 (s, medium), 250 (m, sharp), 200 (w, sharp).

UV-vis

For UV-vis investigations 5 mg of **3** were dissolved in 10 mL hexanes.

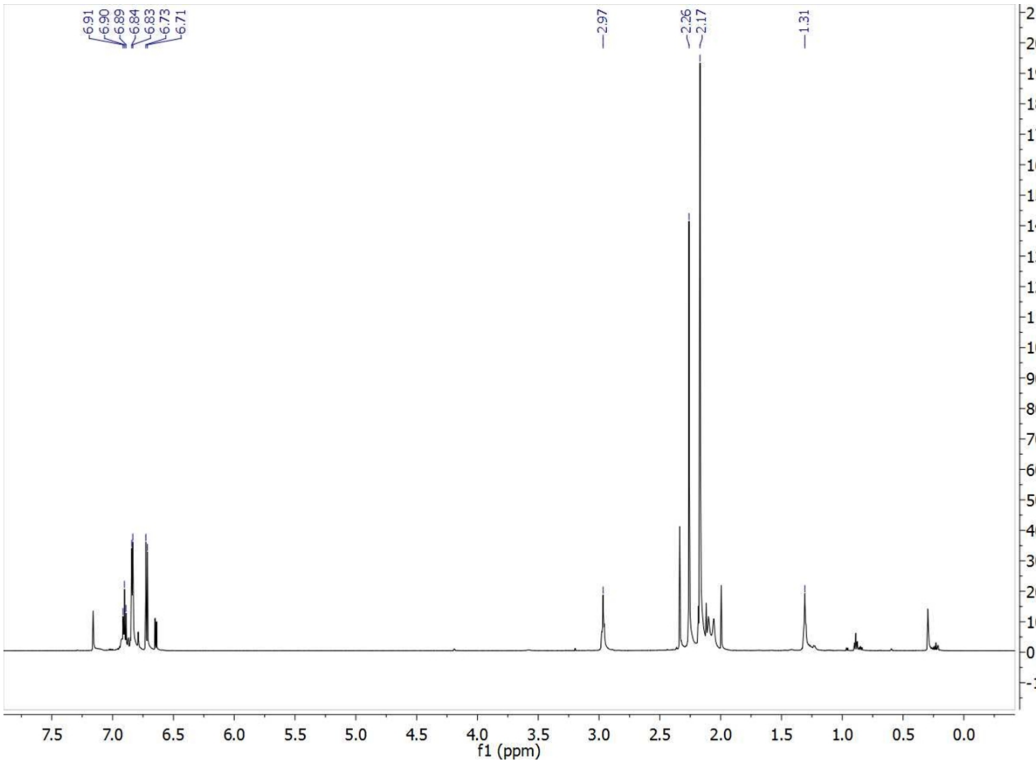

Figure S1.^{1}H NMR spectrum of **3** in C_6D_6 at 25°C.

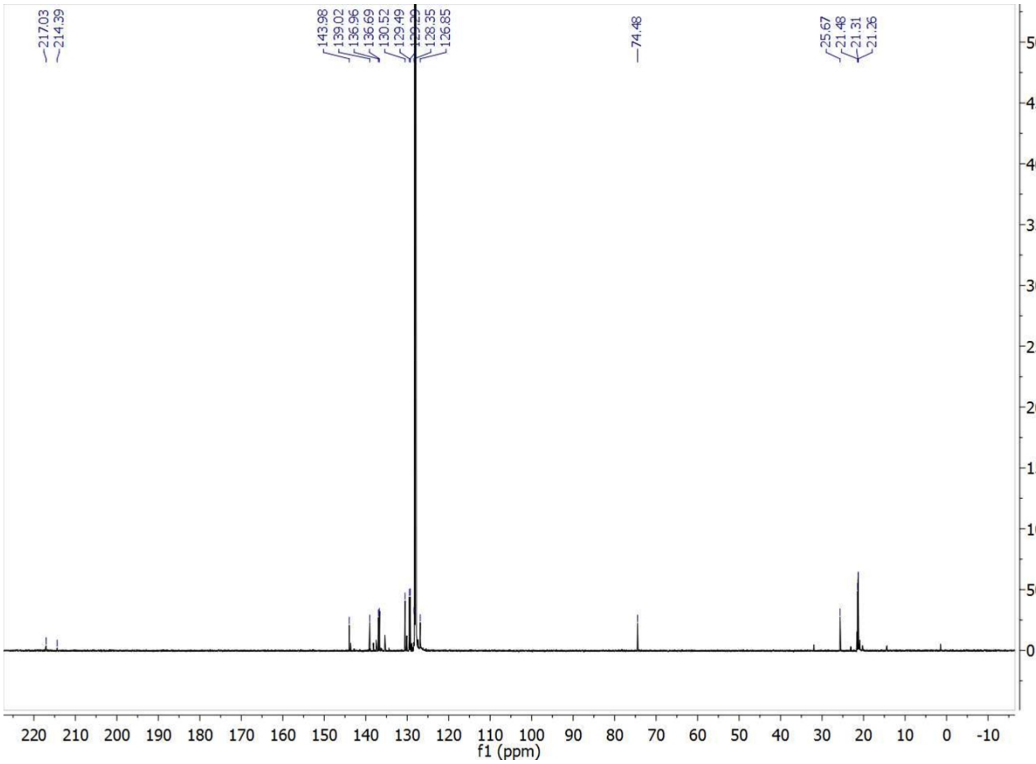

Figure S2.^{13}C NMR spectrum of **3** in C_6D_6 at 25°C.

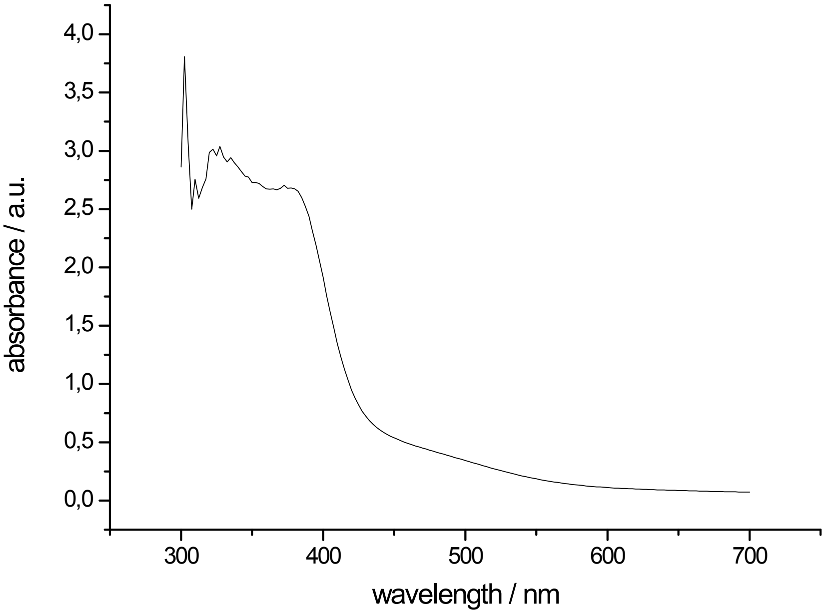

Figure S3. UV-vis spectrum of **3** in hexanes at 25°C.

2.2 $Sn(SAr^{Me6})_2Mo(CO)_3(THF)$ (4)·THF

To a solution of $Sn(SAr^{Me6})_2$ (0.405 g, 0.5 mmol) in toluene (ca. 30 mL) was added a solution of bicyclo[2.2.1]hepta-2,5-diene)tetracarbonylmolybdenum(0) (0.150 g, 0.5 mmol) in toluene (ca. 30 ml). The solution stayed yellow and was stirred for 2 days at room temperature after which time all volatile materials were removed under reduced pressure. The orange residue was extracted in hot THF (ca. 40 mL) and was filtered with a filter-tipped cannula and subsequently concentrated under reduced pressure to ca. 7 mL. Storage of the solution at ca. 7°C afforded orange-red plate-like crystals of **4** (0.153 g, 0.135 mmol, 27 %).

M. p. 185-187 °C (decomp.).

^{1}H NMR (599.7 MHz, CD_2Cl_2, 294 K):

1.42 (m, CH_2(3,4), THF, co-ligand), 1.82 (m, CH_2(3,4), THF), 2.04 (s, 24H, *o*-CH_3), 2.37 (s, 12H, *p*-CH_3), 2.88 (m, CH_2(2,5), THF, co-ligand), 3.68 (m, CH_2(2,5), THF), 6.75 (d, 4H, *m*-C_6H_3, $^3J_{H,H}$ = 7.49 Hz), 6.89 (s, 4H, $C_6H_2Me_3$), 6.94 (s, 4H, $C_6H_2Me_3$), 7.15 (t, 2H, *p*-C_6H_3, $^3J_{H,H}$ = 7.46 Hz).

$^{13}C\{^1H\}$ NMR (CD_2Cl_2, 150.8 MHz, 294 K):

Due to the low solubility of **4**·THF in CD_2Cl_2 a spectrum with signals for the carbonyl carbon atoms could not be obtained.

21.06 (*o*-CH_3), 21.15 (*o*-CH_3), 21.58 (*p*-CH_3), 25.39 (CH_2(3,4), THF, co-ligand), 25.98 (CH_2(3,4), THF), 68.16 (CH_2(2,5), THF), 74.27 (CH_2(2,5), THF, co-ligand), 126.38 (*p*-C_6H_3), 128.71 (*m*-$C_6H_2Me_3$), 129.42 (*m*-C_6H_3), 129.58 (*p*-$C_6H_3Me_3$), 130.39 (*o*-C_6H_3), 136.55 (*o*-$C_6H_2Me_3$), 136.98 (*o*-$C_6H_2Me_3$), 137.18 (*p*-$C_6H_2Me_3$), 139.48 (*i*-$C_6H_2Me_3$), 143.94 (*i*-C_6H_3).

^{119}Sn NMR (223.6 MHz, CD_2Cl_2, 295 K): $\delta = -101.70$.

IR (nujol, CsI plates, cm^{-1}): 2068 (m, sharp), 2040 (w, sharp), 1945 (s, broad), 1905 (s, sharp), 1878 (s, sharp), 1845 (s, broad), 1785 (s, broad), 1605 (m, sharp), 1568 (m, sharp), 1450 (s, broad), 1384 (s, sharp), 1372 (s, sharp), 1065 (m, sharp), 1032 (s, sharp), 874 (w, sharp), 855 (m, sharp), 845 (s, sharp), 799 (s, sharp), 744 (m, sharp).

UV-vis: For UV-vis investigations 4 mg of **4** were dissolved in 10 mL THF.

Elemental analysis: found: C 62.08, H 6.47; calculated: C 62.49 %, H 5.87 %.

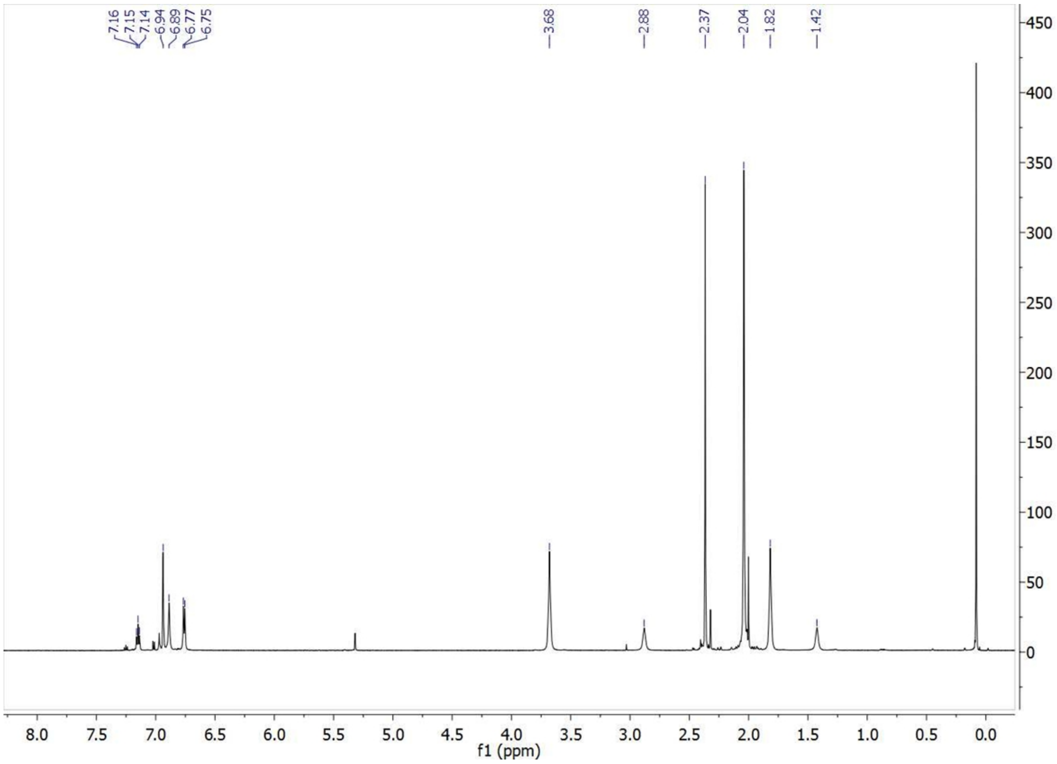

Figure S4. ^{1}H NMR spectrum of **4·THF** in CD_2Cl_2 at 25°C.

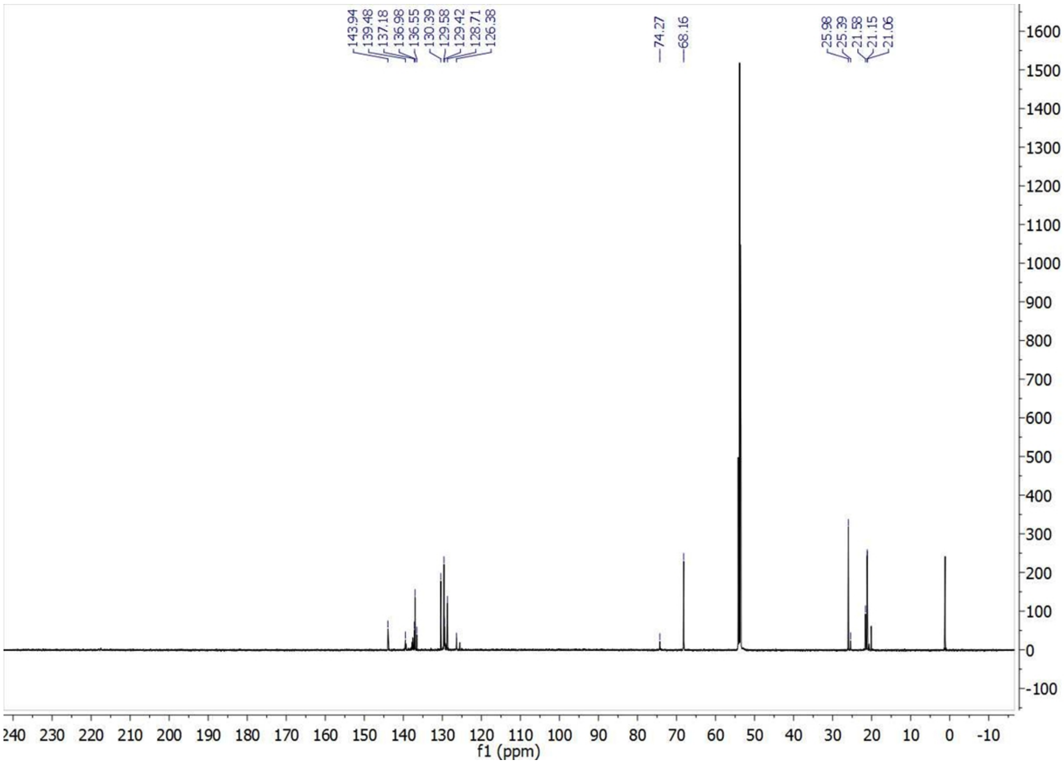

Figure S5. ^{13}C NMR spectrum of **4·THF** in CD_2Cl_2 at 25°C.

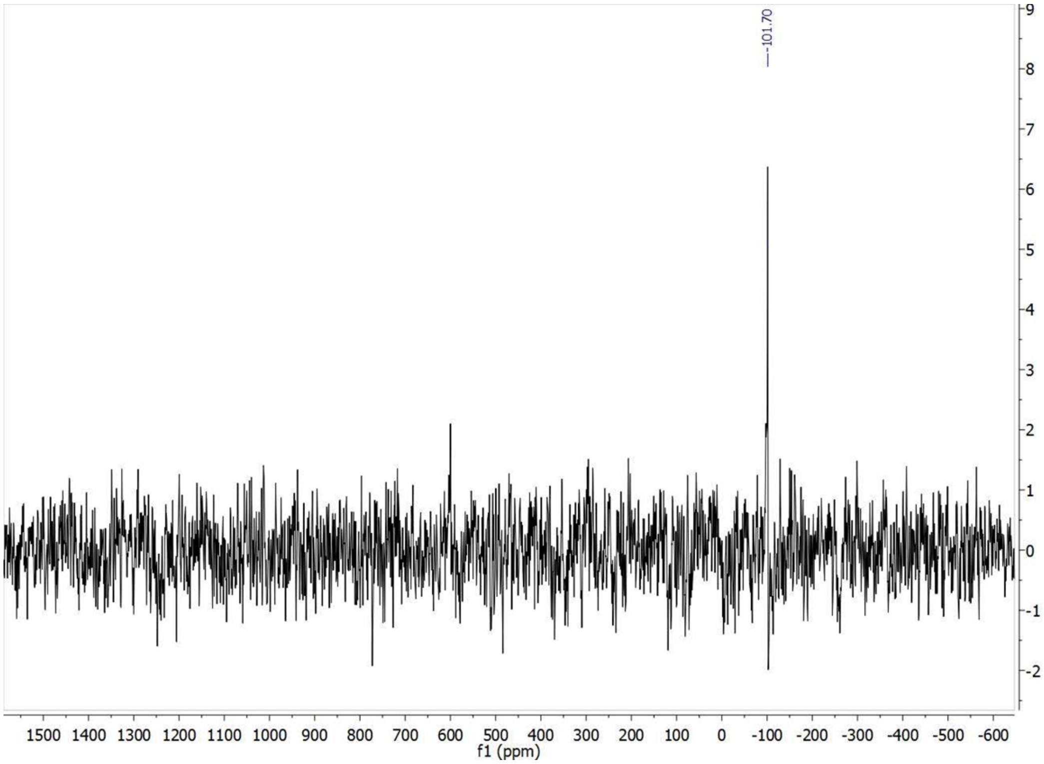

Figure S6. ^{119}Sn NMR spectrum of **4**·THF in CD_2Cl_2 at 25°C.

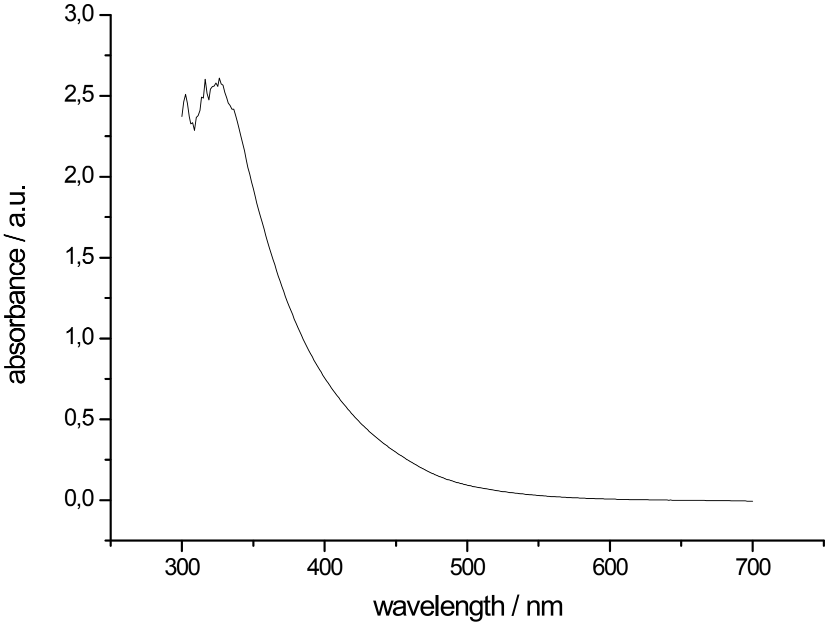

Figure S7. UV-vis spectrum of **4** in THF at 25°C.

2.3 $Sn(SAr^{Me6})_2Mo(CO)_4$ (5)

To a solution of $Sn(SAr^{Me6})_2$ (0.405 g, 0.5 mmol) in THF (ca. 30 mL) was added a solution of bicyclo[2.2.1]hepta-2,5-diene)tetracarbonylmolybdenum(0) (0.150 g, 0.5 mmol) in THF (ca. 30 ml). The solution became orange and was stirred for 2 days at room temperature after which time all volatile materials were removed under reduced pressure. The orange residue was extracted in hot hexanes (ca. 40 mL) and was filtered with a filter-tipped cannula and concentrated under reduced pressure to ca. 10 mL. Storage of the solution at ca. -18°C afforded orange plate-like crystals of **5** (0.040 g, $3.93 \cdot 10^{-5}$ mol, 8 %).

M. p. 210-212 °C (decomp.).

^1H NMR (599.7 MHz, d^6-benzene, 294 K): δ = 2.10 (s, 12H, *o*-CH_3), δ = 2.11 (s, 12H, *o*-CH_3), 2.32 (s, 12H, *p*-CH_3), 6.62 (d, 4H, *m*-C_6H_3, $^3J_{H,H}$ = 7.54 Hz), 6.93 (t, 2H, *p*-C_6H_3, $^3J_{H,H}$ = 7.52 Hz), 6.90 (s, 4H, $C_6\mathit{H}_2Me_3$), 6.92 (s, 4H, $C_6\mathit{H}_2Me_3$).

$^{13}C\{^1H\}$ NMR (d^6-benzene, 150.8 MHz, 295 K): δ = 21.19, 21.63, 126.91, 127.98, 128.14, 128.35, 129.69, 129.74, 136.13, 136.36, 136.83, 127.63, 138.88, 143.87, 214.49, 218.10.

IR (nujol, CsI plates, cm^{-1}): 1990 (s, sharp), 1990 (s, broad), 1915 (s, broad), 1842 (s, broad), 1810 (s, broad), 1570 (m, sharp), 1535 (w, sharp), 1528 (w, sharp), 1410 (s, broad), 1338 (s, broad), 998 (m, sharp), 808 (m, sharp), 762 (m, sharp), 708 (w, sharp).

UV-vis

For UV-vis investigations 4 mg of **5** were dissolved in 10 mL THF.

Elemental analysis: found: C 59.94, H 5.93; calculated: C 61.37 %, H 4.95 %.

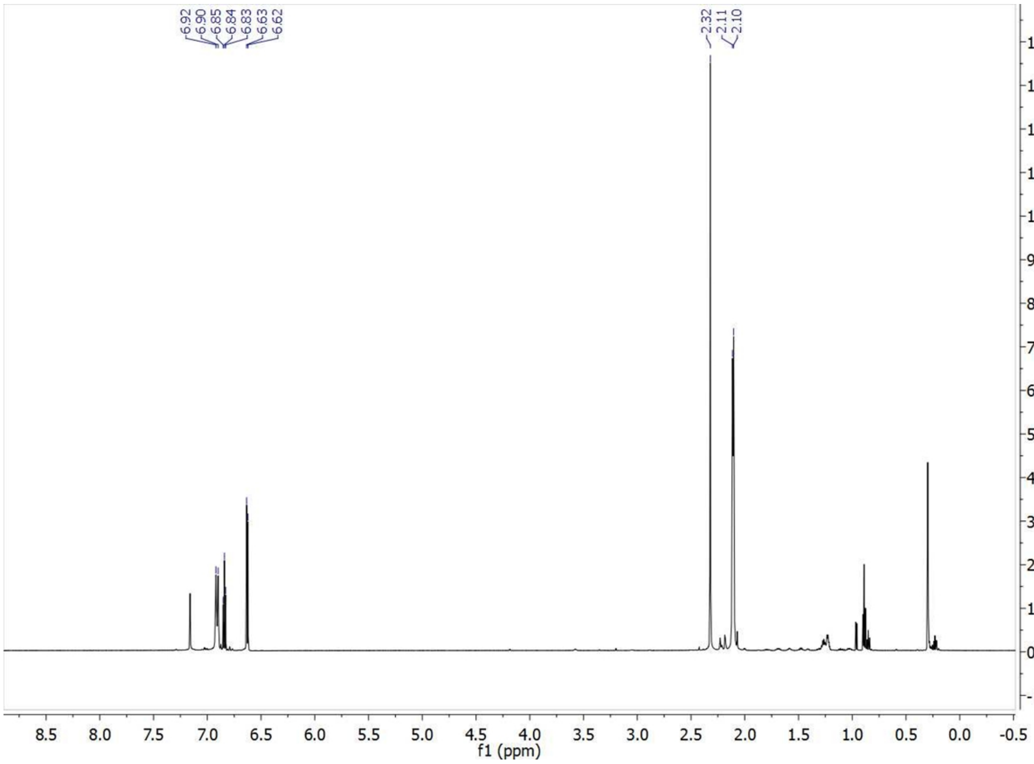

Figure S8. ^{1}H NMR spectrum of **5** in C_6D_6 at 25°C.

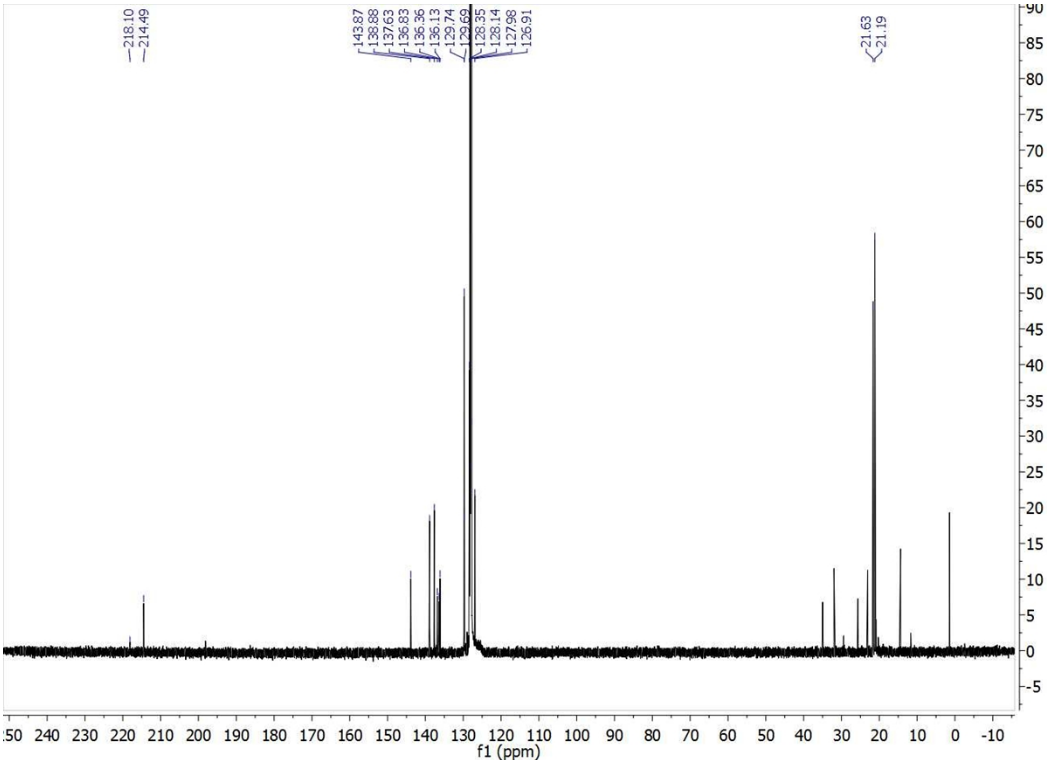

Figure S9. ^{13}C NMR spectrum of **5** in C_6D_6 at 25°C.

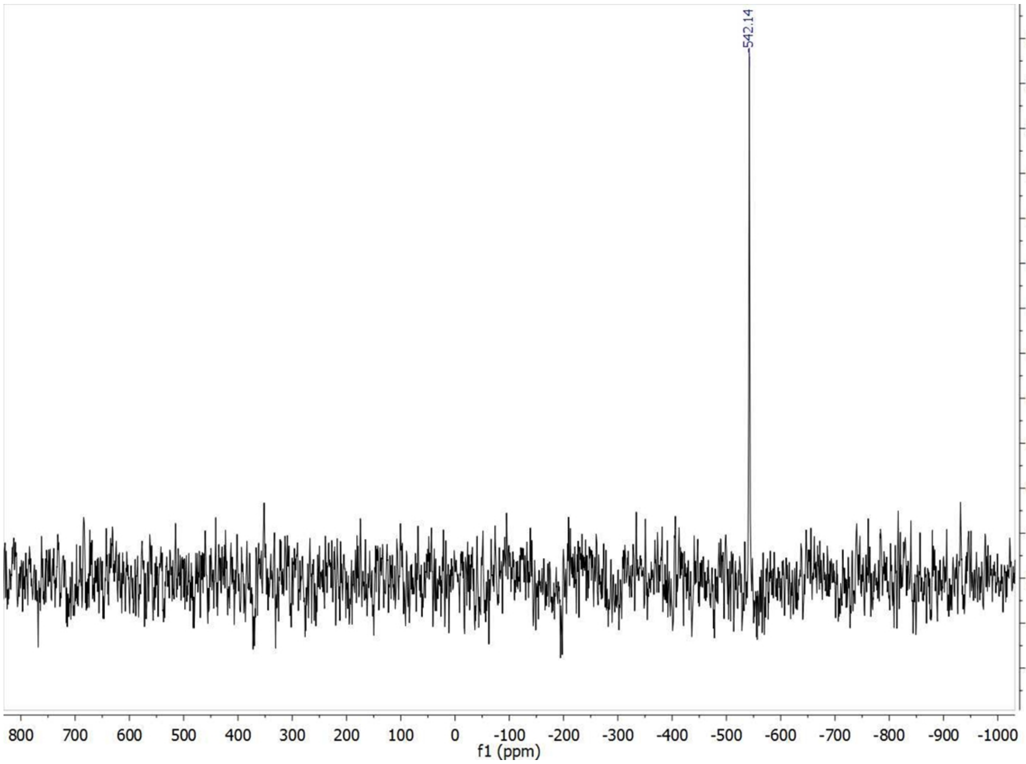

Figure S10. ^{119}Sn NMR spectrum of **5** in C_6D_6 at 25°C.

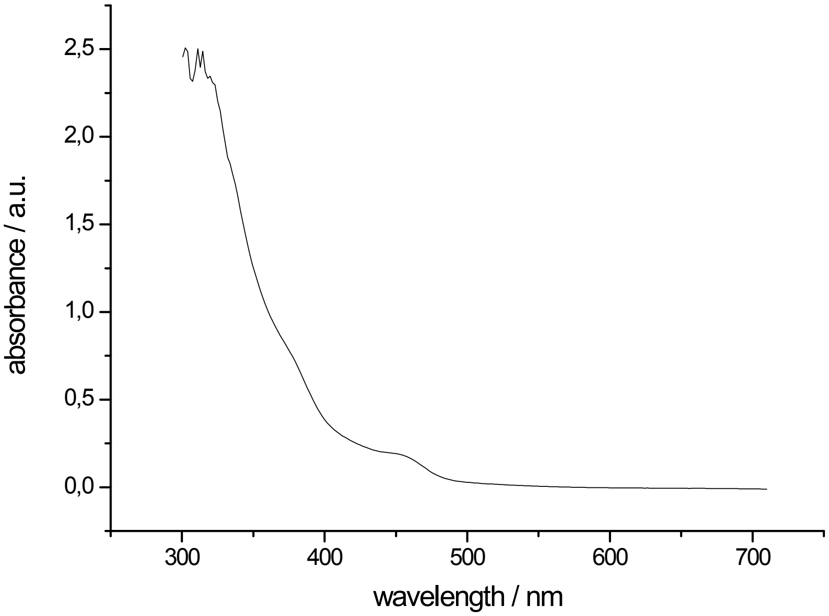

Figure S11. UV-vis spectrum of **5** in THF at 25°C.

3. X-Ray Crystallography

3.1. General experimental procedures

Crystals of **3-5** were removed from a Schlenk tube under N_2 and covered with a layer of hydrocarbon oil. A suitable crystal was selected, attached to a glass fiber and quickly placed in low temperature N_2 stream. The data were collected at ca. 90 K on a Bruker DUO APEX II CCD with with Cu K_α radiation (λ = 1.5418 Å) for **3** and **4** and Mo K_α radiation (λ = 0.71073 Å) for **5**. The crystal structures were solved by direct methods using SHELXTL version 6.1 program package. All non-hydrogen atoms were refined anisotropically. Absorption corrections were applied using SADABS program (*SADABS*, an empirical absorption correction program, part of the SAINTPlus NT version 5.0 package; Bruker AXS: Madison, WI 1998).

3.2. Refinement details for 3

All crystallographic calculations were performed on an iMac with an Intel Core i7 2.80GHz processor and 8GB of extended memory at 1067MHz DDR3. Data collected were corrected for Lorentz and polarization effects with Saint[3] and absorption using Blessing's method and merged as incorporated with the program Sadabs[4,5]. The SHELXTL[6] program package was now implemented to determine based upon intensity statistics, the centrosymmetric monoclinic space group $P2_1/c$ (no. 14). The structure was determined by direct methods with a majority of the non-hydrogen atoms from the molecule of interest being located directly using the program XT.[7] Refinement of the structure was achieved using the program XL.[8] Difference-Fourier least-squares refinement cycles were required to locate the remaining non-hydrogen atoms. Refinement converged to approximately R_F=15% for all observed data. It was evident from the outset that the structure possessed more than a single domain when examining the individual frames so reflections were thresholded in APEX[9] and these reflections were input into Cellnow[10] that determined the twin relationship between the two components and generated the orientation matrices for the components and output a useable multiple matrice input file for the integration program SAINT.[3] Saint was run five times using the output optimized merged matrix file from the previous run. Data collected were now corrected for absorption using TWINABS[4,5,10] and Blessing's method and merged generating both HKLF4 and HKLF5 files. Convergence of the structure proceeded quickly using both the HKLF5 and HKLF4 files. All of the full occupancy non-hydrogen atoms were refined anisotropically and using the HKLF5 determined the percentage of the two domains being present as follows: major:minor component 0.53:0.47 as indicated from the batch scale factor. Since the structure could be refined slightly better with the HKLF5 file it was completed this way. All of the hydrogen atoms were idealized throughout the final convergence cycles. The final structure was refined to convergence with R(F)=6.05%, wR(F^2)=15.79%, GOF=1.049 for all 9147 reflections [R(F)=5.19, (F^2)=14.91 those 7795 with Fo > 4σ(Fo)]. A final difference-Fourier map was featureless indicating that the structure is therefore both correct and complete.

Table S1. Crystal data and structure refinement for **3**.

Compound	**3**
Empirical formula	$C_{55} H_{58} Ge Mo O_4 S_2$
Formula weight /g·mol^{-1}	1015.66
Crystal colour, shape	red, block
Crystal size /mm^3	0.181 x 0.109 x 0.102
Crystal system	monoclinic
Space group	$P\ 2_1/c$
a /Å b /Å c /Å	15.8239(2) 13.6512(2) 23.5101(4)
α /° β /° γ /°	90 109.3011(6) 90
V /Å^3	4793.06(12)
Z	4
ρ_{calc} /g·cm^{-3}	1.407
μ(Cu) /mm^{-1}	4.083
2Θ range /°	21.35 - 71.42
Reflections measured	9147
Independent reflections	9147
R(int)	0.0480
Ind. reflections ($I > 2\sigma(I)$)	7795
Parameters	581
Restraints	0
R_1 ($I > 2\sigma(I)$)	0.0519
wR_2 (all data)	0.1579
$GooF$ (all data)	1.049
Max. peak/hole /e$^-$·Å^{-3}	1.677 /−1.147
Absorption correction type	Multi-scan
Min. /Max. transmission	0.4974 / 0.6897

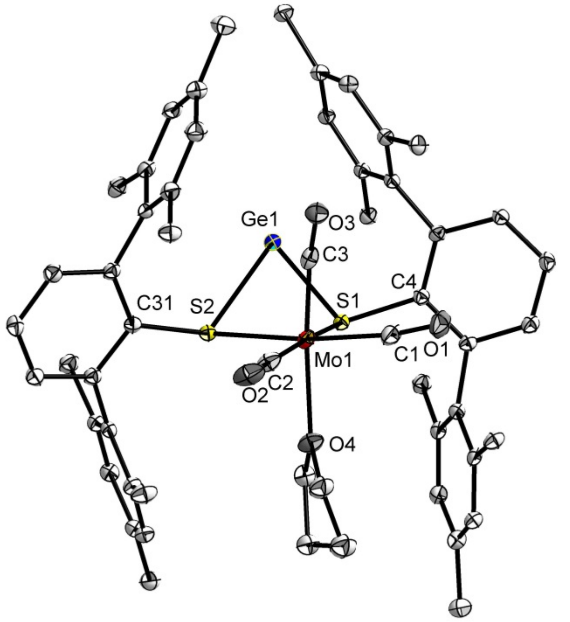

Figure S12. Molecular structures of the two independent molecules of compound **3**. Thermal ellipsoids are drawn at a 30% level. H atoms are omitted for clarity.

Selected bond distances (Å) and angles (deg.): Mo1−C(1-3) 1.949(4), 1.960(5), 1.975(5); Mo1−O4 2.269(3); Mo1−S(1,2) 2.5902(10), 2.6240(9); Ge1−S(1,2) 2.3986(10), 2.4305(9); S(1,2)−C(4,31) 1.803(4), 1.791(4), C(1,2,3)–O(1,2,3) 1.166(6), 1.157(5), 1.168(5), Mo1····Ge1 2.7034(5).

S1-Mo1-S2 70.69(3), S1-Mo1-C1 94.61(15), S2-Mo1-C2 101.52(14), C1-Mo1-C2 93.5(2), C3-Mo1-O4 163.96(14), C3-Mo1-S1 107.16(13), C3-Mo1-S2 104.55(12), C3-Mo1-C1 75.73(17), C3-Mo1-C2 80.85(18), O4-Mo1-C1 94.84(15), O4-Mo1-C2 86.91(15), O4-Mo1-S1 86.26(8), O4-Mo1-S2 87.96(9),S1-Ge1-S2 77.31(3), C4-S1-Ge1 112.22(12), C31-S2-Ge1 116.33(12), C4-S1-Mo1 111.32(12), C31-S2-Mo1 118.35(12).

3.3. Refinement details for 4·THF

Table S2. Crystal data and structure refinement for **4**·THF.

Compound	**4·THF**
Empirical formula	$C_{59} H_{66} Mo O_5 S_2 Sn$
Formula weight /g·mol^{-1}	1133.93
Crystal colour, shape	orange, plate
Crystal size /mm^3	0.12 x 0.12 x 0.22
Crystal system	triclinic
Space group	P -1
a /Å *b* /Å *c* /Å	13.4501(2) 14.5803(2) 14.8084(3)
α /° β /° γ /°	64.7540(10) 86.3880(10) 88.8230(10)
V /Å^3	2621.36(8)
Z	2
ρ_{calc} /g· cm^{-3}	1.437
μ(Cu) /mm^{-1}	6.867
2Θ range /°	6.7-136.7
Reflections measured	36096
Independent reflections	9227
R(int)	0.0272
Ind. reflections ($I > 2\sigma(I)$)	9028
Parameters	613
Restraints	–
R_1 ($I > 2\sigma(I)$)	0.0332
wR_2 (all data)	0.0922
GooF (all data)	1.047
Max. peak/hole /e$^-$·Å^{-3}	1.001 / −0.815
Absorption correction type	Multi-scan
Min. /Max. transmission	0.3134 / 0.4929

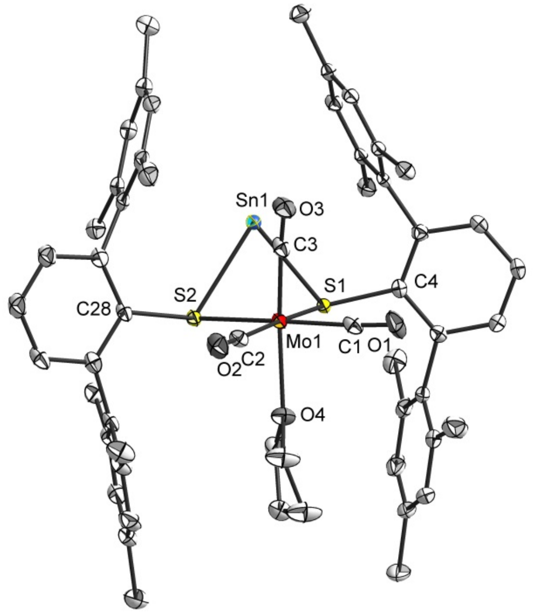

Figure S13. Molecular structures of the two independent molecules of compound **4**. Thermal ellipsoids are drawn at a 50 % level. H atoms and a co-crystallised THF molecule are not shown.

Selected bond distances (Å) and angles (deg.): Mo1−C(1-3) 1.944(3), 1.949(3), 1.970(3); Mo1−O4 2.270(2); Mo1−S(1,2) 2.6082(7), 2.6119(7); Sn1−S(1,2) 2.6279(7), 2.6303(7); S(1,2)−C(4,28) 1.798(3), 1.801(3); C(1,2,3)–O(1,2,3) 1.156(4), 1.157(4), 1.170(4); Mo1····Sn1 2.9050(3).

S1-Mo1-S2 73.19(2), S1-Mo1-C1 95.26(10), S2-Mo1-C2 95.26(10), C1-Mo1-C2 90.24(14), C3-Mo1-O4 163.89(11), C3-Mo1-S1 107.64(10), C3-Mo1-S2 107.24(10), C3-Mo1-C1 78.61(14), C3-Mo1-C2 77.65(13), O4-Mo1-C1 91.19(12), O4-Mo1-C2 90.10(11), O4-Mo1-S1 85.49(6), O4-Mo1-S2 85.16(6), S1-Sn1-S2 72.58(2), C4-S1-Sn1 115.48(10), C28-S2-Sn1 113.92(11), C4-S1-Mo1 112.32(10), C28-S2-Mo1 114.47(10).

3.4. Refinement details for 5

An orange plate with approximate orthogonal dimensions 0.069 x 0.085 x 0.132mm^3 was placed and optically centered on the Bruker Duo(1) APEXII CCD diffractometer at −183°C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.5° wide omega-scans, 10 seconds per frame, and 30 frames per series that were well distributed in reciprocal space. Twenty-one omega and phi-scan data frame series were collected [CuK$_\alpha$] with 0.5° wide scans and variable frame times based upon diffraction angle. The crystal to detector distance was 4.96cm, thus providing a limited sphere of data with processing to 2theta max=144.32°. All crystallographic calculations were performed on an iMac with 2.80GHz quad core processor and 8GB of extended memory. A total of 33057 reflections were collected and corrected for Lorentz and polarization effects in SAINT[3] and absorption using Blessing's method as incorporated into the program SADABS[4,5] with 9038 unique. The SHELXTL[6] program package was implemented to determine the probable space group and set up the initial files. System symmetry, lack of systematic absences and intensity statistics indicated the centrosymmetric triclinic space group P-1 (no. 2). The structure was determined by direct methods with the successful location of a majority of the non-hydrogen atoms comprising the molecule of interest using the program XS.[7] The structure was refined with SHELXL-2012.[8] The 33057 data collected were merged, based upon identical indices to 16421 data [R(int)=0.0286], and further merged during least-squares refinement to 8869 unique data [R(int)=0.0181]. The molecule was found to suffer from whole molecule disorder that was determined to be 0.79:0.21 major:minor components, respectively. The two orientations were optimized for all of the atoms that did not perfectly overlap with a variety of restraints. A heavily disordered hexane molecule was initially located and after multiple attempts to assemble it properly, it was abandoned and Platon Squeeze[11,12] was run and now refined to convergence. The final refinement included multiple instructions to idealize the minor components geometry with the major component without any solvent present. Hydrogen atoms idealized throughout. The final structure was refined to convergence with R(F)=3.26%, wR(F^2)=3.39%, GOF=1.106 for all 8869 unique reflections [R(F)=3.39%, wR(F^2)=8.59% for those 8461 data with Fo > 4sigma(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete. An empirical correction for extinction was also attempted and found to be negative and therefore not applied.

Table S3. Crystal data and structure refinement for **5**.

Compound	**5**
Empirical formula	$C_{52} H_{50} Mo O_4 S_2 Sn$
Formula weight /g·mol^{-1}	1017.73
Crystal colour, shape	orange, plate
Crystal size /mm^3	0.132 x 0.085 x 0.069
Crystal system	triclinic
Space group	P -1
a /Å *b* /Å *c* /Å	13.1027(10) 14.4748(11) 14.8890(12)
α /° β /° γ /°	73.751(3) 67.107(3) 86.712(4)
V /Å^3	2493.3(3)
Z	2
ρ_{calc} /g· cm^{-3}	1.356
μ(Cu) /mm^{-1}	7.144
2Θ range /°	3.19-72.06
Reflections measured	16421
Independent reflections	8869
R(int)	0.0285
Ind. reflections ($I > 2\sigma(I)$)	8461
Parameters	667
Restraints	62
R_1 ($I > 2\sigma(I)$)	0.0324
wR_2 (all data)	0.0859
GooF (all data)	1.106
Max. peak/hole /e$^-$·Å^{-3}	0.771 / −0.357
Absorption correction type	Multi-scan
Min. /Max. transmission	0.5049 / 0.6288

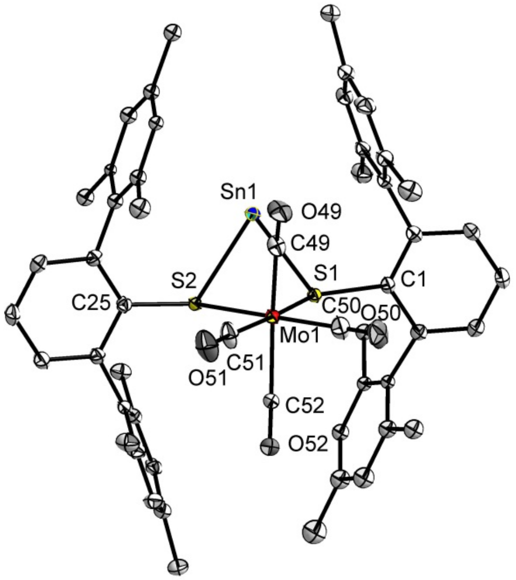

Figure S14. Molecular structure of the major component of compound **5**. Thermal ellipsoids are drawn at a 30% level. H atoms are omitted for clarity.

Selected bond distances (Å) and angles (deg.): Mo1−C(49-52) 1.982(3), 1.988(3), 2.003(4), 2.101(4); Mo1−S(1,2) 2.6194(10), 2.6235(9), 2.6219(17), 2.6233(17); Sn1−S(1,2) 2.595(2), 2.597(3); S(1,2)−C(1,25) 1.787(3), 1.793(3); C(49-52)–O(49-52) 1.150(5), 1.145(4), 1.148(4) , 1.123(5); Mo1····Sn(1,2) 2.9633(10), 2.9542(19).

S1-Mo1-S2 68.22(8), 67.5(3); S1-Mo1-C50 101.53(13); S2-Mo1-C51 102.77(12); C50-Mo1-C51 87.36(14); C49-Mo1-C52 162.16(18); C49-Mo1-S1 106.48(14); C49-Mo1-S2 107.06(14); C49-Mo1-C50 77.90(17); C49-Mo1-C51 77.65(17); C52-Mo1-C50 90.68(16); C52-Mo1-C51 88.28(17); C52-Mo1-S1 89.09(13); C52-Mo1-S2 86.56(12); S1-Sn1-S2 68.98(6), 68.3(4); C1-S1-Sn1 118.66(13); C25-S2-Sn1 119.44(13); C1-S1-Mo1 116.52(14); C25-S2-Mo1 118.50(13).

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