4-nitrosilabenzene, 110852-02-3; 1-methylsilabenzsene, 63878-65-9; 2-methylsilabenzene, 110852-03-4; 3-methylsilabenzene, 110852-04-5; 4-methylsilabenzene, 110852-05-6; 1-silylsilabenzene, 110852-06-7; 2-silylsilabenzene, 110852-07-8; 3-silylsilabenzene, 110852-08-9; 4-silylsilabenzene, 110852-09-0; 1-methoxysilabenzene, 110852-10-3; 2-methoxysilabenzene, 110852-11-4; 3-methoxysilabenzene, 110852-12-5; 4-methoxysilabenzene, 110852-13-6; 1-carboxysilabenzene, 110852-14-7; 2-carboxysilabenzene, 110852-15-8; 3-carboxysilabenzene, 110852-16-9; 4-carboxysilabenzene, 110852-17-0; silabenzene, 289-77-0.

**Supplementary Material Available:** Geometries, electronic energies and dipole moments, and $q_\sigma$, $q_\pi$, and $q_{Ph-X}$ values for substituted compounds of both benzene and silabenzene (17 pages). Ordering information is given on any current masthead page.

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# Four- and Five-Coordinate Tin(II) (Stannylene) Compounds: Crystal and Molecular Structures of $W(CO)_5\{SnCl_2(OC_4H_8)\}$ and $W(CO)_5\{SnCl_2(OC_4H_8)_2\}$

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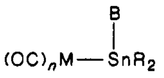
Photolysis of a tetrahydrofuran (THF) solution of tungsten hexacarbonyl and tin(II) chloride gives $W(CO)_5\{SnCl_2(OC_4H_8)_2\}$ which loses THF on heating to produce $W(CO)_5\{SnCl_2(OC_4H_8)\}$. Both complexes have been characterized by X-ray crystallography. Crystals of $W(CO)_5\{SnCl_2(OC_4H_8)\}$ belong to the triclinic space group $P\bar{1}$ (No. 2) with $a$ = 6.668 (2) Å, $b$ = 9.791 (4) Å, $c$ = 12.583 (2) Å, $\alpha$ = 70.49 (2)°, $\beta$ = 89.03 (1)°, $\gamma$ = 77.67 (2)° at 130 K, $Z$ = 2, $R$ = 0.045, and $R_w$ = 0.051. The structure consists of a six-coordinate tungsten and an irregular, four-coordinate tin. Yellow crystals of $W(CO)_5\{SnCl_2(OC_4H_8)_2\}$ belong to the triclinic space group $P\bar{1}$ (No. 2) with $a$ = 9.589 (9) Å, $b$ = 10.704 (5) Å, $c$ = 10.856 (8) Å, $\alpha$ = 69.06 (6)°, $\beta$ = 86.03 (7)°, $\gamma$ = 66.41 (6)° at 130 K, $Z$ = 2, $R$ = 0.052, and $R_w$ = 0.057. The structure consists of a six-coordinate tungsten and a five-coordinate tin with distorted trigonal-bipyramidal geometry (pseudoaxial O–Sn–O angle = 160.1 (2)°). The major change in adding THF to $W(CO)_5\{SnCl_2(OC_4H_8)\}$ is an ca. 0.13 Å increase in the Sn–O bond length; the Sn–W bond length shows only a slight elongation (0.02 Å). Very little change in the angular distribution of ligands about tin accompanies the change in tin coordination number. The THF appears to add to a vacant tin coordination site in $W(CO)_5\{SnCl_2(OC_4H_8)\}$.

## Introduction

Initial attempts to prepare stannylene complexes 1 of group 6 and other transition-metal carbonyls resulted in the isolation of compounds that contained donor solvent molecules (B) coordinated to tin.[1] Only with bulky or donor substituents (R) was it possible to isolate examples of structure 1.[2] The majority of characterized stannylene complexes with additional donor groups bonded to tin involve four-coordinate tin with structure 2.[2,3] The few examples of stannylene complexes of type 3 with five-coordinate tin involve polydentate ligands (for example, a porphyrin dianion,[4] acetylacetonate[5]) bound to tin.

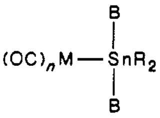
$(OC)_nM—SnR_2$

**1**



$(OC)_nM—SnR_2(B)$

**2**



$(OC)_nM—SnR_2(B)_2$

**3**

In the course of our studies of main group/transition metal interactions,[6] we had occasion to examine the structural nature of the $W(CO)_5\{SnCl_2(THF)_n\}$ (THF is tetrahydrofuran) system. Previous reports indicated that photolysis of tungsten hexacarbonyl in tetrahydrofuran in the presence of tin(II) chloride yields products formulated as the solvent-free $W(CO)_5\{SnCl_2\}$ ($n$ = 0)[7] or as the solvate $W(CO)_5\{SnCl_2(THF)\}$.[8] Studies of the $^{119}$Sn NMR spectra of the latter complex in benzene solution indicated that there was a marked change produced by adding THF (chemical shift altered from −54.6 to −209.4 ppm; $^1J$ ($^{119}$Sn, $^{183}$W) changed from 1440 to 1594 Hz) and that a second species, formulated as $W(CO)_5\{SnCl_2(THF)_n\}$, was formed.[9] Here we report on the unambiguous preparations of $W(CO)_5\{SnCl_5(THF)\}$ (4) and $W(CO)_5\{SnCl_2(THF)_2\}$ (5) and their structural characterization by X-ray crystallography. Compound **5** is the first example of a compound of type **3** in which only monodentate ligands are present on tin, and **4** and **5** are the first structurally characterized pair

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**Table I. Atomic Coordinates (×10⁴) and Isotropic Thermal Parameters (Å² × 10³) for $W(CO)_5\{SnCl_2(THF)\}$ (4)**

| | x | y | z | U |
|---|---|---|---|---|
| W | 1184 (1) | 1328 (1) | 1641 (1) | 17 (1)[a] |
| Sn | −34 (1) | 1981 (1) | 3516 (1) | 20 (1)[a] |
| Cl(1) | 2064 (4) | 2780 (3) | 4565 (2) | 39 (1)[a] |
| Cl(2) | −2421 (3) | 1257 (3) | 4902 (2) | 30 (1)[a] |
| O(1) | 374 (11) | −1945 (8) | 2670 (6) | 34 (1) |
| O(2) | 5739 (11) | 65 (8) | 2795 (6) | 34 (1) |
| O(3) | 2115 (12) | 4549 (9) | 701 (6) | 42 (2) |
| O(4) | −3307 (11) | 2446 (8) | 441 (6) | 35 (1) |
| O(5) | 2865 (12) | 848 (9) | −567 (6) | 41 (2) |
| O(6) | −2045 (10) | 4249 (7) | 2973 (5) | 30 (1) |
| C(1) | 619 (13) | −770 (10) | 2312 (7) | 25 (2) |
| C(2) | 4121 (13) | 503 (9) | 2396 (7) | 23 (2) |
| C(3) | 1737 (13) | 3390 (10) | 1034 (7) | 24 (2) |
| C(4) | −1702 (13) | 2060 (10) | 867 (7) | 23 (2) |
| C(5) | 2269 (13) | 980 (10) | 259 (7) | 25 (2) |
| C(6) | −3860 (15) | 4520 (12) | 2241 (8) | 35 (2) |
| C(7) | −5304 (23) | 5905 (17) | 2366 (12) | 60 (3) |
| C(8) | −4542 (27) | 5972 (21) | 3431 (14) | 76 (4) |
| C(9) | −2394 (16) | 5206 (12) | 3704 (9) | 36 (2) |

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

**Table II. Atomic Coordinates (×10⁴) and Isotropic Thermal Parameters (Å² × 10³) for $W(CO)_5\{SnCl_2(THF)_2\}$ (5)**

| | *x* | *y* | *z* | *U* |
|---|---|---|---|---|
| W | 1415 (1) | 2062 (1) | 4290 (1) | 13 (1)[a] |
| Sn | 3038 (1) | 2787 (1) | 2176 (1) | 14 (1)[a] |
| Cl(1) | 2050 (3) | 5017 (2) | 338 (2) | 24 (1)[a] |
| Cl(2) | 5470 (2) | 1578 (3) | 1541 (2) | 30 (1)[a] |
| O(1) | 3286 (8) | −1380 (8) | 5136 (7) | 35 (2) |
| O(2) | −805 (8) | 1854 (7) | 2395 (7) | 30 (1) |
| O(3) | −621 (7) | 5462 (7) | 3480 (6) | 29 (1) |
| O(4) | 3695 (8) | 2092 (7) | 6266 (6) | 30 (1) |
| O(5) | −795 (8) | 1619 (7) | 6549 (7) | 28 (1) |
| O(6) | 4222 (6) | 4035 (6) | 2818 (5) | 20 (2)[a] |
| O(7) | 2252 (7) | 1815 (6) | 862 (5) | 20 (2)[a] |
| C(1) | 2623 (9) | −144 (9) | 4823 (8) | 19 (1) |
| C(2) | −20 (10) | 1951 (9) | 3066 (8) | 20 (2) |
| C(3) | 138 (9) | 4241 (9) | 3772 (8) | 20 (2) |
| C(4) | 2881 (10) | 2101 (9) | 5534 (8) | 20 (2) |
| C(5) | 72 (9) | 1723 (8) | 5741 (7) | 18 (1) |
| C(6) | 3281 (10) | 5242 (9) | 3265 (8) | 25 (2) |
| C(7) | 4222 (14) | 6171 (14) | 3030 (12) | 44 (3) |
| C(8) | 5767 (12) | 5228 (11) | 2839 (10) | 36 (2) |
| C(9) | 5499 (12) | 4362 (11) | 2148 (10) | 34 (2) |
| C(10) | 2749 (10) | 261 (9) | 1230 (8) | 24 (2) |
| C(11) | 1639 (10) | 90 (9) | 423 (8) | 25 (2) |
| C(12) | 1261 (11) | 1463 (10) | −868 (9) | 29 (2) |
| C(13) | 1965 (11) | 2376 (10) | −586 (9) | 25 (2) |

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $U_{ij}$ tensor.

of complexes of types **2** and **3** that are related by simple gain or loss of B.

## Results

Photolysis of a tetrahydrofuran solution containing tungsten hexacarbonyl and tin(II) chloride gives a yellow solution.[7,8] Removal of the solvent by vacuum evaporation leaves a dirty yellow solid. When this crude product is heated under vacuum at 60 °C, some unreacted tungsten hexacarbonyl sublimes from the sample. Recrystallization of the unsublimed residue from hexane produces yellow crystals of $W(CO)_5\{SnCl_2(THF)\}$ (**4**). However, if the crude product is recrystallized from hexane, without subjecting it to heating at 60 °C, then yellow crystals of $W(CO)_2\{SnCl_2(THF)_2\}$ (**5**) are obtained. The melting points reported in the Experimental Section allow these substances to be readily distinguished. The infrared spectra in the region of $\nu$(CO) are very similar since a common $W(CO)_5$ fragment is present, but the positions of the high- and

**Table III. Selected Interatomic Distances (Å) for $W(CO)_5\{SnCl_2(THF)\}$ (4) and $W(CO)_5\{SnCl_2(THF)_2\}$ (5)**

| | 4 | 5 |
|---|---|---|
| Sn-W | 2.711 (1) | 2.737 (1) |
| Sn-Cl(1) | 2.352 (3) | 2.371 (2) |
| Sn-Cl(2) | 2.375 (2) | 2.364 (2) |
| Sn-O(6) | 2.223 (6) | 2.356 (8) |
| Sn-O(7) | | 2.350 (8) |
| W-C(1) | 2.058 (9) | 2.045 (8) |
| W-C(2) | 2.065 (8) | 2.042 (11) |
| W-C(3) | 2.018 (9) | 2.032 (8) |
| W-C(4) | 2.044 (8) | 2.037 (11) |
| W-C(5) | 1.978 (10) | 1.989 (8) |
| C(1)-O(1) | 1.13 (1) | 1.14 (1) |
| C(2)-O(2) | 1.13 (1) | 1.14 (1) |
| C(3)-O(3) | 1.15 (1) | 1.15 (1) |
| C(4)-O(4) | 1.13 (1) | 1.15 (1) |
| C(5)-O(5) | 1.14 (1) | 1.17 (1) |

**Table IV. Selected Bond Angles (deg) for $W(CO)_5\{SnCl_2(THF)\}$ (4) and $W(CO)_5\{SnCl_2(THF)_2\}$ (5)**

| | 4 | 5 |
|---|---|---|
| W-Sn-O(6) | 108.3 (2) | 102.3 (1) |
| W-Sn-O(7) | | 97.5 (1) |
| W-Sn-Cl(1) | 122.9 (1) | 123.4 (1) |
| W-Sn-cl(2) | 135.4 (1) | 134.7 (1) |
| Cl(1)-Sn-O(6) | 88.3 (2) | 82.4 (1) |
| Cl(1)-Sn-O(7) | | 85.2 (1) |
| Cl(2)-Sn-O(6) | 87.3 (2) | 84.8 (2) |
| Cl(2)-Sn-O(7) | | 82.6 (2) |
| Cl(1)-Sn-Cl(2) | 98.3 (1) | 101.8 (1) |
| O(6)-Sn-O(7) | | 160.1 (2) |
| Sn-W-C(1) | 91.8 (3) | 95.1 (2) |
| Sn-W-C(2) | 88.2 (3) | 88.8 (2) |
| Sn-W-C(3) | 86.4 (3) | 86.6 (2) |
| Sn-W-C(4) | 93.8 (3) | 91.6 (2) |
| Sn-W-C(5) | 174.3 (3) | 174.5 (2) |
| C(1)-W-C(2) | 89.0 (3) | 88.6 (4) |
| C(2)-W-C(3) | 90.4 (3) | 89.8 (4) |
| C(3)-W-C(4) | 91.6 (3) | 92.0 (4) |
| C(4)-W-C(1) | 89.1 (3) | 89.5 (4) |

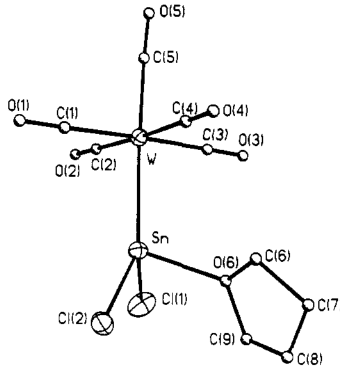

**Figure 1.** A perspective drawing of $W(CO)_5\{SnCl_2(OC_4H_8)\}$ (4). Anisotropic thermal ellopsoids for W, Sn, and Cl are shown at the 50% probability level; uniform, arbitrarily sized circles are used for other atoms.

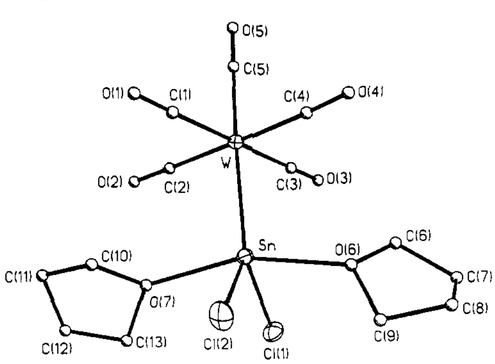

**Figure 2.** A perspective drawing of $W(CO)_2\{SnCl_2(OC_4H_8)_2\}$ (5).

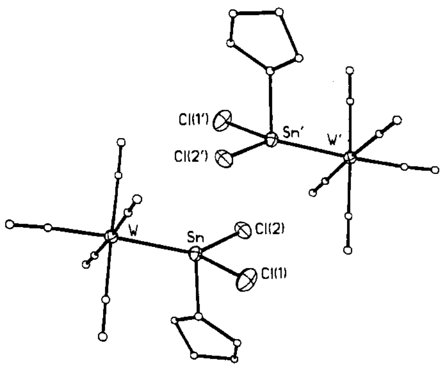

**Figure 3.** A diagram showing the packing of two $W(CO)_5\{SnCl_2(OC_4H_8)\}$ molecules.

low-energy absorptions allow the two compounds to be differentiated.

The structures of both 4 and 5 have been determined by X-ray crystallography. Atomic positional parameters for 4 are given in Table I. Atomic positional parameters for 5 are given in Table II. Bond distances and angles are compared in Tables III and IV.

A view of 4 which shows the atomic numbering scheme is given in Figure 1, and a similar view of 5 is shown in Figure 2. The structures of the two molecules 4 and 5 are closely related. Both contain six-coordinate $W(CO)_5Sn$ units with similar geometry. The shortening of the W–C(5) bond (the bond trans to the W–Sn bond) in both 4 and 5 results from a lower trans affect for the stannylene ligand relative to carbon monoxide. This effect is common for a variety of stannylene complexes.[2] The Sn–W distances in 4 and 5 are also similar.

The most important difference between 4 and 5 involves the coordination environment about the tin atom. In 4 the tin is four-coordinate. The geometry about tin is similar to that in $Cr(CO)_5\{Sn(t\text{-}Bu)_2(NC_5H_5)\}$[1c] and deviates significantly from idealized tetrahedral geometry. Thus the tin atom lies remarkably close to the $WCl_2$ plane with an out-of-plane displacement of only 0.25 Å. The Cl–Sn–Cl and Cl–Sn–O angles are all significantly less than 109°.

Molecules of 4 pack in the solid about centers of symmetry as shown in Figure 3. One reviewer interpreted this packing as indicating the presence of a dimeric structure with chloro bridges. The intramolecular Sn···Cl contact (3.220 (4) Å) is significantly longer than the Sn–Cl bond lengths (2.352 (3), 2.375 (2) Å). True chloro bridges between tin(II) centers yield symmetrical structures as in the $Sn_2Cl_2^{2+}$ unit with Sn–Cl bond lengths of ca. 2.6 Å.[10] While there may be a secondary, weak interaction between molecules of 4, we do not think these should be considered to be chloro-bridged dimers. Efficient packing of molecules may simply be involved in the arrangements seen in Figure 3.

In 5, the tin atom is five-coordinate with a geometry that is best described as trigonal bipyramidal with the two oxygen atoms occupying the axial sites. The $WSnCl_2$ unit is nearly planar with the tin atom only 0.040 Å out of the $WCl_2$ plane. However, the O(6)–Sn–O(7) angle is 160.1 (2)°, so the trigonal bipyramid is significantly distorted toward a square pyramid with tungsten at the axial site. The Sn–O bond lengths are similar to those (2.344 (7), 2.276 (7) Å) in $[HPt(PEt_3)(SnR_2)\{Sn(OMe)R_5\}_2(SnR_3)]$ (R = $p\text{-}CH_3C_6H_4$)[11] and in $(OC)_5W{=}Sn[W(CO)_5]_2THF$.[12]

The addition of THF to 4 to form 5 results in one major bond length change. The Sn–O bond lengthens by ca. 0.13 Å in 5 as compared to 4. Otherwise the bond distances in 4 and 5 are similar. Even more remarkable is the consistency of the bond angles in 4 and 5. Thus, the additional THF in 5 appears to bond to a vacant site in 4 without any dramatic reorientation of the ligands.

The structural features suggest that tin uses a set of s and 2p orbitals to bind the $WCl_2$ unit which is nearly planar in both 4 and 5 and uses the remaining p orbital to accept electrons from the THF molecules. In 4 a normal two-electron, two-centered bond between Sn and O is formed, whereas in 5 a four-electron, three-centered bonding scheme involving the OSnO unit results. As a consequence, the Sn–O bonds in the five-coordinate adduct 5 are weaker and longer than in 4. Moreover, the effect of adding THF to 4 mainly perturbs the bonding to the other THF because both share bonding through a single p orbital. The structural data, which show a very slight elongation in the W–Sn bond length of 5 relative to 4, and a flattening of the $WSnCl_2$ unit in 5 are in reasonably good accord with the NMR data.[7] The $^1J(^{119}Sn,^{183}W)$ increases by 11% in 5, relative to 4. This indicates a small change in the W–Sn bonding with greater s character to the W–Sn bond in 5 and probably reflects the use of the s and only two p orbitals for the bonding to the $WCl_2$ portion and the use of the pure p orbital for binding the two THF moieties. The NMR data are not consistant with conversion from $sp^3$ hydridization for 4 to $dsp^3$ hybridization for 5 since this would reduce the s character to the W–Sn bond in 5 and cause a reduction in $J(^{119}Sn,^{183}W)$.

Finally we note that there is a surprising difference between the $SnCl_2(THF)$ ligand with its nearly planar $WSnCl_2$ unit and the much more thoroughly studied $SnCl_3^-$ ligand. These two are related by simple replacement of the THF by $Cl^-$. However, the $SnCl_3^-$ ligand has a much more symmetrical structure than the $\{SnCl_2(THF)\}$ ligand seen in these tungsten complexes. Thus, $SnCl_3^-$ usually has three equivalent Cl–Sn–Cl angles near 100° and equivalent M–Sn–Cl angles near 115°.[3]

## Experimental Section

**Preparation of Compounds.** Tungsten hexacarbonyl (Strem) and anhydrous tin(II) chloride (Ventron) were used as purchased. Solvents were carefully dried and purged of oxygen before use. All reagents and products were handled with the exclusion of air by standard Schlenk techniques.

**$W(CO)_5\{SnCl_2(THF)\}$ (4).** In a 100-mL Schlenk tube were placed 0.70 g of tungsten hexacarbonyl, 0.38 g of stannous chloride, and 50 mL of THF. The solution was irradiated with a 360-W mercury lamp for 12 h (the distance of the lamp from the wall of the flask was 10 cm). The solvent was removed by vacuum evaporation, and the residue was recrystallized from hexane to give yellow needles. These were purified to remove tungsten hexacarbonyl and THF by heating at 60 °C under vacuum (1 torr). The residue was then recrystallized from hexane to give 0.48 g (43%) of yellow crystals: mp 100–103 °C; IR (hexane) 2080, 1997, 1967, 1951, 1935 $cm^{-1}$.

**$W(CO)_5\{SnCl_2(THF)_2\}$ (5).** This procedure is the same as above except that the crude product was not heated under vacuum. Rather, the residue was dissolved in hexane, filtered through a bed of Celite, and slowly cooled to −20 °C to give 0.78 g (71%) of yellow needles: mp 82–84 °C; IR (hexane) 2075, 1997, 1967, 1951, 1919 $cm^{-1}$.

**X-ray Data Collection.** Yellow parallelepipeds of 4 and yellow needles of 5 were formed by slowly cooling a saturated hexane solution of the compounds prepared at +25 °C to −20 °C. The crystals were removed from the flask and rapidly coated with a

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**Table V. Crystal Data for 4 and 5**

| | $W(CO)_5\{SnCl_2(THF)\}$ (4) | $W(CO)_5\{SnCl_2(THF)_2\}$ (5) |
|---|---|---|
| formula | $C_9H_8Cl_2O_6SnW$ | $C_{13}H_{16}Cl_2O_7SnW$ |
| fw | 585.65 | 657.70 |
| cryst system | triclinic | triclinic |
| space group | $P\bar{1}$ (No. 2) | $P\bar{1}$ (No. 2) |
| cryst dimens, mm | 0.10 × 0.15 × 0.60 | 0.15 × 0.20 × 0.60 |
| color and habit | yellow parallelepipeds | yellow needles |
| unit-cell dimens (130 K) | | |
| *a*, Å | 6.668 (2) | 9.589 (9) |
| *b*, Å | 9.791 (4) | 10.704 (5) |
| *c*, Å | 12.583 (2) | 10.856 (8) |
| $\alpha$, deg | 70.49 (2) | 69.06 (6) |
| $\beta$, deg | 89.03 (1) | 86.03 (7) |
| $\gamma$, deg | 77.67 (2) | 66.41 (6) |
| $V$, Å$^3$ | 755.1 (4) | 950 (1) |
| $D$(calcd, 130 K), g/cm$^3$ | 2.58 | 2.30 |
| $Z$ | 2 | 2 |
| radiatn, $\lambda$, Å (graphite monochromator) | Mo K$\alpha$, 0.071 069 | Mo K$\alpha$, 0.071 069 |
| $\mu$(Mo K$\alpha$), cm$^{-1}$ | 100.78 | 80.30 |
| transmissn factors | 0.25–0.44 | 0.21–0.38 |
| octants | $+h,\pm k,\pm l$ | +h,±k,±l |
| scan type, $2\theta$ max, deg | $\omega$, 50 | $\omega$, 50 |
| scan range, deg | 1.4 | 1.6 |
| scan speed, deg min$^{-1}$ | 20 | 20 |
| no. of unique data | 3472 | 4307 |
| no. of data ($I > 3\sigma(I)$) | 3020 | 3845 |
| data/parameter | 31.1 | 30.3 |
| $R$ | 0.045 | 0.052 |
| $R_w$ | 0.051 | 0.057 |

light hydrocarbon oil to protect them from the atomsphere. They were mounted in the cold stream of a Syntex $P2_1$ diffractometer equipped with a modified LT-1 low-temperature apparatus. Accurate unit cell parameters were obtained from a least-squares refinement of 15 reflections with 15° ≤ $2\theta$ ≤ 25°. The crystals of 4 and 5 were found to be triclinic $P$ by the automatic indexing routine of the software available on the Syntex $P2_1$ diffractometer; no symmetry was observed in any of the axial photographs. Data collection parameters are summarized in Table V. The data were corrected for Lorentz and polarization effects.

**Solution and Refinement of Structures.** All calculations were done on a Data General MV/10000 computer using the SHELXTL Version 5 software package. On the basis of the Patterson map and the distribution of $|E^2 - 1|$, the centrosymmetric space group $P\bar{1}$ (No. 2) was selected for 4 and 5. For 4, the positions of the tungsten, tin, and two chlorine atoms were generated from FMAP 8, the Patterson-solving routine of SHELXTL. Other atomic positions were located from successive difference Fourier maps. Anisotropic thermal parameters were assigned to the elements tungsten, tin, and chlorine. The final stages of refinement included an absorption correction.[13] Hydrogen atoms were placed at idealized positions ($d$(C–H) = 0.96 Å) and assigned isotropic thermal parameters 20% greater than the carbon atom to which they were attached. The final $R$ value of 0.045 was computed from 97 least-squares parameters and 3020 reflections. This yielded a goodness-of-fit of 0.859 and a mean shift/esd of 0.003 for $U_{11}$ of C(8) during the last cycle of refinement. The largest feature on the final difference Fourier map (1.1 e/Å$^3$) was 1.0 Å away from tungsten. The weighting scheme used was $w = [\sigma^2(F_o)]^{-1}$. Corrections for anomalous dispersion were applied to all atoms. Neutral-atom scattering factors were those of Cromer and Waber.[14]

The structure of 5 was solved by locating the tungsten and tin atom positions using the Patterson map. The remainder of the solution and refinement proceeded as described for 4. The final $R$ value of 0.052 was computed from 127 least-squares parameters and 3845 reflections. This yielded a goodness-of-fit of 0.783 and a mean shift/esd of 0.003 for overall scale on the last cycle of refinement. A value of 2.0 e/Å$^3$ was found as the largest feature on the final difference Fourier map. This peak was found 1.0 Å away from tungsten.

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**Registry No.** 4, 65198-59-6; 5, 110935-38-1; $W(CO)_6$, 14040-11-0.

**Supplementary Material Available:** Tables of bond distances, bond, angles, anisotropic thermal parameters, and calculated hydrogen coordinates (8 pages); listings of structure factors (41 pages). Ordering information is given on any current masthead page.

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[13] The method obtains an empirical absorption tensor from an expression relating $F_o$ and $F_c$: Moezzi, B., Hope, H., unpublished results, Department of Chemistry, University of California, Davis, CA.

[14] *International Tables for X-ray Crystallography*; Kynoch: Birmingham, England, 1974; Vol. 4.