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Notes

cyclo-Octasulfur Adducts of $WCl_4(S)(THF)$ and WCl_6. Crystal and Molecular Structure of $WCl_4(S)(THF){\cdot}S_8$

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It is an emerging concept in inorganic chemistry that redox reactions between metals or metal complexes and singly bonded chemical elements may be preceded by a primary interaction with the metal site. Chemical elements (H_2, I_2, chalcogens), at variance with undergoing cleavage of the element–element single bond, can combine with a metal complex or a main group compound essentially by two mechanisms: (a) coordination to the metal center as in the nonclassical metal hydrides,[2] in the diiodine[3] complexes of platinum(II), and in the chalcogen complexes $Ag(S_8)$, AsF_6,[4] $Re_2X_2(CO)_6(S_8)$,[5] and $Re_2I_2(CO)_6(Se_7)$;[6] (b) chemical bond formation or weaker interaction with one of the ligands, as observed in the solid-state structures of $Re(I_3)(CO)_{5-n}L_n$,[7] $CHI_3{\cdot}3S_8$,[8] $SnI_4{\cdot}2S_8$,[9] $AI_3{\cdot}3S_8$ (A = Sb,[10] As[11]), $SbCl_3{\cdot}S_8$,[12] $Ti_4O(S_2)_4Cl_6{\cdot}2S_8$,[13] $S_4(NH)_4{\cdot}3S_8$,[14] $Co_6(\mu_3\text{-}S)_8(CO)_6{\cdot}3S_8$,[15] $(PPh_4)_4(Ag_2S_{20}){\cdot}S_8$,[16] and $[(PPh_3)_2N(Ag(S_9)]{\cdot}S_8$.[17] As a followup of recent studies[7] on solution and solid-state properties of carbonyl–iodo complexes of rhenium(I) being converted to the corresponding triiodo complexes with diiodine, we became interested in trying to convert a transition metal complex containing a terminal sulfido ligand into a polysulfide, a reaction which is well established for ionic sulfides,[18] but still unknown in metal complex chemistry. Molecular complexes containing terminal sulfido ligands are rare in coordination chemistry;[19] moreover, a metal complex in a high oxidation state had to be chosen in order to prevent oxidation of the metal center. Due to these restrictions, our choice of the dinuclear chloride-bridged tungsten(VI) complex $W_2Cl_8S_2$[20] was substantially mandated. While this work was in progress, a paper[21] reported the crystal and molecular structure of the *cyclo*-octasulfur adduct $WCl_4(S){\cdot}S_8$, as prepared from $W(CO)_6$ and S_2Cl_2. We want now to report that $W_2Cl_8S_2$ in the presence of tetrahydrofuran gives $WCl_4(S)(THF){\cdot}S_8$ with *cyclo*-octasulfur; interestingly, this compound differs from the previous one by the presence of a THF ligand that occupies the sixth coordination position around tungsten. Furthermore, the 1:1 adduct of WCl_6 with S_8, reported earlier by F. A. Cotton *et al.*[22] has been prepared from the components.

Experimental Section

The IR spectra were measured with Perkin-Elmer Model 283 and FT-IR 1725X instruments. $W_2Cl_8S_2$[20] was prepared according to the literature method. Hexachlorotungsten(VI) was a commercial (Fluka) product used without further purification.

Preparation of Complexes. $WCl_4(S)(THF){\cdot}S_8$. The red chlorosulfide $W_2Cl_8S_2$ (0.797 g, 1.11 mmol) and S_8 (0.588 g, 2.29 mmol) were dissolved in 30 mL of CS_2, and the resulting red solution was stirred for 10 minutes. The solution was filtered, 25 mL of heptane was added, and the mixture was concentrated to about 35 mL. The resulting suspension was heated to about 50 °C to obtain a clear solution, to which was added 0.5 mL of THF (6.2 mmol). The dark-red microcrystals which deposited overnight at about −30 °C were filtered off and dried *in vacuo* to afford the air-sensitive product (50% yield). Anal. Calcd for $C_4H_8Cl_4OS_9W$: S, 42.0; W, 26.8. Found, S: 41.3; W, 26.8. Single crystals for the X-ray crystallographic study were obtained by dissolving the product in a mixture of CS_2 and heptane and by cooling at about −30 °C.

$WCl_6{\cdot}S_8$. The red hexachloride WCl_6 (1.07 g, 2.70 mmol) and S_8 (0.681 g, 2.65 mmol) were dissolved in 20 mL of CS_2. To the red solution after filtration from small amounts of some insoluble impurity was added 30 mL of heptane. The dark-red microcrystalline product which separated out at about −30 °C was filtered off and dried *in vacuo* to afford a 59% yield of the *cyclo*-octasulfur adduct. Anal. Calcd for Cl_6S_8W: S, 39.3. Found: S, 39.0. A single crystal of this compound was found to be isomorphous with the adduct reported earlier by Cotton *et al.*[22]

X-ray Crystallography of $WCl_4(S)(THF){\cdot}S_8$. A small portion was cleaved from a well-formed crystal of the title compound, mounted on a glass fiber, and moved into the cold stream of the diffractometer. The cell constants were determined from a list of reflections found by an automated search of reciprocal space, and verified by axial photography. Data were collected in the $+h$, $\pm k$, $\pm l$ hemisphere: a total of 3569 reflections were measured; 465 observed reflections were later averaged to give $R = 0.022$. Azimuthal scans of several reflections with Eulerian angle χ near 90° were used as the basis of an empirical absorption correction. Important crystal data, data collection, and structure solution parameters are provided in Table 1. Atomic coordinates are given in Table 2.

The positions of the metal atom, coordination sphere atoms and atoms

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Table 1. Crystal and Intensity Measurement Data for $WCl_4(S)(THF){\cdot}S_8$

chem formula: $C_4H_8Cl_4S_9W$
$a = 7.924(2)$ Å[a]
$b = 7.955(2)$ Å
$c = 15.136(2)$ Å
$\alpha = 96.31(2)°$
$\beta = 98.41(2)°$
$\gamma = 89.93(2)°$
$V = 938.1(5)$ Å^3
$Z = 2$

fw = 686.35
space group: $P\bar{1}$
$T = -60(1)$ °C
$\lambda = 0.71073$ Å
$D_c = 2.430$ g cm^{-3}
$\mu = 78.19$ cm^{-1}
$R = 0.033$[b]
$R_w = 0.058$[c]

[a] Estimated standard deviations in the least significant figure are given in parentheses. [b] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. [c] $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$; $w = 1/\sigma^2(|F_o|)$.

Table 2. Positional Parameters and Their Estimated Standard Deviations for $WCl_4(S)(THF){\cdot}S_8$[a]

atom	x	y	z	B_{eq} (Å^2)
W	0.18257(3)	0.20382(2)	0.31131(1)	2.319(5)
Cl(1)	−0.0938(2)	0.1691(2)	0.3364(1)	3.75(3)
Cl(2)	0.1486(3)	0.4931(2)	0.3329(1)	4.03(4)
Cl(3)	0.4733(2)	0.2448(3)	0.3293(1)	4.19(4)
Cl(4)	0.2271(3)	−0.0764(2)	0.3326(1)	3.99(4)
S(1)	0.1388(2)	0.1775(2)	0.1712(1)	3.55(3)
S(2)	0.4741(2)	0.4154(2)	0.1061(1)	2.38(3)
S(3)	0.3720(2)	0.6224(2)	0.1691(1)	2.58(3)
S(4)	0.3739(2)	0.8160(2)	0.0905(1)	2.68(3)
S(5)	0.5811(2)	0.9677(2)	0.1411(1)	3.04(3)
S(6)	0.7707(2)	0.9049(2)	0.0659(1)	3.06(3)
S(7)	0.9333(2)	0.7467(2)	0.1338(1)	3.15(3)
S(8)	0.8701(2)	0.5029(2)	0.0805(1)	2.76(3)
S(9)	0.7236(2)	0.4030(2)	0.1627(1)	2.71(3)
O	0.2367(6)	0.2383(6)	0.4676(3)	3.5(1)
C(1)	0.356(3)	0.366(2)	0.522(1)	5.9(4)
C(2)	0.330(2)	0.378(2)	0.610(1)	4.6(3)
C(3)	0.250(2)	0.211(2)	0.6200(9)	3.8(3)
C(4)	0.154(2)	0.161(2)	0.5305(9)	4.1(3)
C(1′)	0.150(3)	0.328(2)	0.524(1)	6.6(4)
C(2′)	0.203(2)	0.297(3)	0.620(1)	6.8(4)
C(3′)	0.365(2)	0.221(3)	0.614(1)	5.5(4)
C(4′)	0.351(3)	0.124(2)	0.522(1)	7.2(4)

[a] Values for anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as: $B_{eq} = {}^1/_3[a^2a^{*2}B_{11} + b^2b^{*2}B_{22} + c^2c^{*2}B_{33} + 2ab(\cos\gamma)a^*b^*B_{12} + 2ac(\cos\beta)a^*c^*B_{13} + 2bc(\cos\alpha)b^*c^*B_{23}]$.

Table 3. Bond Distances (Å) and Angles (deg) for $WCl_4(S)(THF){\cdot}S_8$[a]

W–Cl(1)	2.299(1)	S(7)–S(8)	2.050(2)
W–Cl(2)	2.309(2)	S(8)–S(9)	2.043(2)
W–Cl(3)	2.300(2)	O–C(1)	1.48(2)
W–Cl(4)	2.307(2)	O–C(4)	1.42(2)
W–S(1)	2.085(2)	C(1)–C(2)	1.37(3)
W–O	2.327(5)	C(2)–C(3)	1.50(2)
S(2)–S(3)	2.038(2)	C(3)–C(4)	1.47(2)
S(2)–S(9)	2.043(2)	O–C(1′)	1.32(2)
S(3)–S(4)	2.048(2)	O–C(4′)	1.51(2)
S(4)–S(5)	2.043(2)	C(1′)–C(2′)	1.50(2)
S(5)–S(6)	2.044(3)	C(2′)–C(3′)	1.43(3)
S(6)–S(7)	2.048(2)	C(3′)–C(4′)	1.50(2)
Cl(1)–W–Cl(2)	88.86(7)	S(5)–S(6)–S(7)	107.8(1)
Cl(1)–W–Cl(3)	163.95(7)	S(6)–S(7)–S(8)	108.00(9)
Cl(1)–W–Cl(4)	88.29(7)	S(7)–S(8)–S(9)	107.6(1)
Cl(1)–W–S(1)	98.44(7)	S(2)–S(9)–S(8)	107.8(1)
Cl(1)–W–O	82.7(1)	W–O–C(1)	124.8(8)
Cl(2)–W–Cl(3)	89.52(7)	W–O–C(4)	129.4(6)
Cl(2)–W–Cl(4)	164.11(7)	C(1)–O–C(4)	105(1)
Cl(2)–W–S(1)	97.22(7)	O–C(1)–C(2)	110.(2)
Cl(2)–W–O	81.9(1)	C(1)–C(2)–C(3)	105.(1)
Cl(3)–W–Cl(4)	88.91(7)	C(2)–C(3)–C(4)	103.(1)
Cl(3)–W–S(1)	97.60(7)	O–C(4)–C(3)	107.(1)
Cl(3)–W–O	81.2(1)	W–O–C(1′)	127.9(8)
Cl(4)–W–S(1)	98.66(7)	W–O–C(4′)	123.5(6)
Cl(4)–W–O	82.2(1)	C(1′)–O–C(4′)	107.(1)
S(1)–W–O	178.6(1)	O–C(1′)–C(2′)	113.(1)
S(3)–S(2)–S(9)	107.60(8)	C(1′)–C(2′)–C(3′)	100.(1)
S(2)–S(3)–S(4)	108.1(1)	C(2′)–C(3′)–C(4′)	106.(1)
S(3)–S(4)–S(5)	107.89(9)	O–C(4′)–C(3′)	101.(1)
S(4)–S(5)–S(6)	107.98(9)		

[a] Numbers in parentheses are estimated standard deviations in the least significant digits.

of the S_8 ring were given in a direct-method E-map.[23] Following full-matrix least-squares calculations using these positions, two sets of peaks corresponding to the (disordered) carbon atoms of the THF moiety appeared in a difference Fourier map. The two sets of carbon atoms were initially included in the refinement with occupancies set at 0.5; in later refinement of the occupancies, the value did not deviate from 0.5 within the error in the calculation; therefore the refinement was continued with the carbon atom occupancies fixed at 0.5. Least-squares planes calculated for the two orientations of the THF groups intersect at approximately 90°. In the final cycles of refinement, all of the non-hydrogen atoms were refined with anisotropic thermal parameters. Intensity statistics suggested a secondary extinction effect, so an extinction coefficient was included and refined to a value of 6.2(5) × 10^{-7}. Hydrogen atoms (occupancy = 0.5) were included in calculated positions for structure factor calculations only.[24]

Results and Discussion

The chloride-bridged tungsten(VI) terminal sulfido complex $W_2Cl_8S_2$ exists in two modifications[25] differing slightly in bond distances and angles. Also, the Lewis base adducts of formula $WCl_4(S)(L)$, including the THF adduct $WCl_4(S)(THF)$, have been reported.[26] A CS_2 solution of the THF adduct prepared *in situ* by reaction of the dinuclear complex with a slight excess of THF was treated with a substantially equimolar quantity of *cyclo*-octasulfur; no evidence was found (^{1}H-NMR, IR, electronic spectra) of the presence in solution of any adduct at room temperature in appreciable concentration. On the other hand, crystallization of the two components occurred in a single crystal phase; the solid-state formation of the adduct can be represented by the following equations:

$$W_2Cl_8(S)_2 + 2THF \rightarrow 2WCl_4(S)(THF) \quad (1)$$

$$WCl_4(S)(THF) + S_8 \rightarrow WCl_4(S)(THF){\cdot}S_8 \quad (2)$$

Relevant to these observations is that the solubilities at 25 °C of $WCl_4(S)(THF)$ and S_8 are similar; thus, the individual components have similar thermodynamic parameters of solubilization pertaining to the sublimation and solvation processes. The formation of the mixed system, which is favored by entropy, may become prevailing in the presence of some specific, even weak, solute–solute interaction. What the intermolecular interactions might be comes, in this specific case, from the consideration of the crystal structure parameters; see Table 3. Within the $WCl_4(S)(THF)$ moiety, the W–Cl bond distances range from 2.299(1) to 2.309(2) Å, and are similar to the corresponding distances observed in the chloride-bridged dimer[25] and in its S_8 adduct;[21] the W–S distance [(2.085(2) Å] in our compound should be compared with the 2.098(8)-Å distance in the dimer,[25a] and with the 2.076(13)-Å distance in the polymorph[25b] constituted by a dinuclear entity $W_2Cl_8(S)_2$ chloride-bridged to two mononuclear $WCl_4(S)$ entities. As far as the S_8 moiety is concerned, it has the usual crown conformation, with S–S distances ranging from 2.038(2) to 2.050(2) Å.

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The presence of both $WCl_4(S)(THF)$ and S_8 in the same crystal requires some comment. It had already been noted[21] that in $WCl_4(S){\cdot}S_8$ the W···S distance [3.189(2) Å] to one sulfur atom of the crown-shaped S_8 moiety, pointing to the apical position of the distorted square-pyramid of $WCl_4(S)$, is close to the sum of the van der Waals radius of sulfur and the metallic radius of tungsten and that probably more significant solid-state interactions are those between two nonadjacent sulfur atoms of S_8 and two adjacent chlorides of the $WCl_4(S)$ moiety (3.36 and 3.25 Å). The ambiguity between S_8 being η^1-coordinated to tungsten or interacting with the tungsten-coordinated chlorides is relieved by the findings of the present structure. As a matter of fact, the sixth coordination position of tungsten is now occupied by THF at a W–O distance of 2.327(5) Å and one of the two sulfur atoms of *cyclo*-octasulfur on the symmetry plane now interacts with the tungsten-coordinated chloride at a Cl(2)···S(3) distance of 3.491(3) Å, which is shorter than the sum of sulfur and chlorine van der Waals radii[27] (3.65 Å).

WCl_6 has a solubility (1.03 M) in CS_2 at room temperature which is similar to that (1.40 M[28]) of *cyclo*-octasulfur in the same solvent, and the solid-state adduct $WCl_6{\cdot}S_8$ can be prepared accordingly by cooling the solution of the components at low temperature. We are presumably dealing again with an exclusive solid-state phenomenon. The Cl···S contacts in this solid range from 3.451(4) to 3.797(4) Å, nine out of the 17 Cl···S contacts below 3.797(4) Å being shorter than 3.65 Å.[22]

The present paper has established conclusively that weak van der Waals interactions between elemental sulfur and tungsten-coordinated chloride are mainly responsible for the formation of $WCl_4(S)(THF){\cdot}S_8$. The known solid-state adducts[8-13] of inorganic or organic halides with *cyclo*-octasulfur should be interpreted similarly. It had already been noted[8] that in $CHI_3{\cdot}S_8$, the S···I distance of 3.50 Å is shorter than the sum of the van der Waals radii (4.0 Å[27]). The presence of weak halogen–chalcogen interactions is clearly important for the stabilization of these solid-state adducts, and cases of chalcogens cocrystallizing with halogen-containing molecules may become a more frequent phenomenon than originally thought. The similar solubility of the components and lowering the temperature below the solubility threshold are probably important factors in isolating the "lattice complexes". Unsuccessful attempts to prepare the S_8 adduct $[Cu(C_{12}H_8N_2)_2I]I{\cdot}S_8$ from the components at room temperature or higher have been reported.[29]

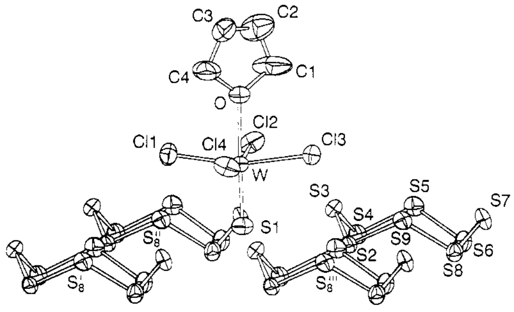

Figure 1. ORTEP view of $WCl_4(S)(THF){\cdot}S_8$ with the atom numbering. The *cyclo*-octasulfur groups are symmetry related by the following operations: single prime, $x - 1$, $y - 1$, z; double prime, $x - 1$, y, z; triple prime, x, $y - 1$, z. Nonbonding distances: Cl(1)···S(9)'', 3.525(2); Cl(2)···S(3), 3.491(3); Cl(3)···S(2), 3.778(3); Cl(3)···S(5)''', 3.608(3); Cl(3)···S(9), 3.747(3); Cl(4)···S(3)''', 3.564(2); Cl(4)···S(7)', 3.671(2); S(1)···S(2), 3.578(3).

The detection of similar interactions in solution probably requires the use of specific spectroscopic methods in a low-polarity solvent. Experiments of this type are in progress.

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Supplementary Material Available: Tables of crystal and intensity measurement data, positional parameters of calculated hydrogen atoms, nonbonded distances of $WCl_4(S)(THF){\cdot}S_8$, nonbonded distances of $WCl_6{\cdot}S_8$, least-squares planes, general displacement parameter expressions, and root-mean-square amplitudes of thermal vibration (8 pages). Ordering information is given on any current masthead page.

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