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Photochemical reactions of metal carbonyls [$M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$, $Mn(CO)_3Cp$] with 3,5-dimethyl-tetrahydro-2H-1,3,5-thiadiazine-2-thione (DTTT) and the crystal structure of [$W(CO)_5(DTTT)$]

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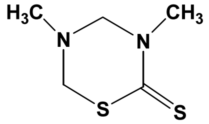
Abstract

Five new complexes, [$M(CO)_5(DTTT)$] [M = Cr; **1**, Mo; **2**, W; **3**], [$Re(CO)_4Br(DTTT)$] (**4**) and [$Mn(CO)_2Cp(DTTT)$] (**5**) have been synthesized by the photochemical reaction of metal carbonyls [$M(CO)_6$] (M = Cr, Mo and W), [$Re(CO)_5Br$], and [$Mn(CO)_3Cp$] with 3,5-dimethyl-tetrahydro-2H-1,3,5-thiadiazine-2-thione (DTTT). The complexes have been characterized by elemental analysis, mass spectrometry, FTIR, ^1H and $^{13}C\{^1H\}$ NMR spectroscopy. The spectroscopic studies show that DTTT behaves as a monodentate ligand coordinating via the sulfur (C=S) donor atom in **1**–**5**. An X-ray diffraction study of [$W(CO)_5(DTTT)$] (**3**) confirms that the tungsten adopts a distorted octahedral geometry with local C_{4v} symmetry.

Keywords: Pesticides; Antiprotooans; 3,5-dimethyl-tetrahydro-2H-1,3,5-thiadiazine-2-thione

1. Introduction

3,5-dimethyl-tetrahydro-2H-1,3,5-thiadiazine-2-thione (DTTT), has been widely used as a pesticide in a variety of settings [1]. In agriculture DTTT has found application as a fungicide, herbicide, and nematocide for cabbage, cucumber, maize, potato, and tomato plants. In industrial environments DTTT has been used as a slimicide in paper mills an as a biocide in metal working fluids used in the manufacture of engines, transmissions, aircraft, and specialty metal products. The chemical DTTT has also been considered for use as a biocide in pump spray-delivered consumer products and custodial supplies. Respiratory exposure may occur from liquid aerosol generated during spray application of materials containing DTTT. Perhaps of greater significance is the potential for exposure to volatile and semivolatile degradation products of DTTT.



This ‘CS_2’ incorporated heterocycle is sparingly soluble in water and it has been equivocally proposed to undergo decomposition to formaldehyde, methylisothiocyanate, and other compounds in aqueous solution [1–10].

It has been reported that DTTT gave unstable complexes with Cu(II), Co(II), Ni(II), Fe (III) ions in solution and were not isolated in solid form [11].

Sulfido carbonyl compounds continue to attract considerable attention not only on account of their fascinating structural chemistry, but also because of

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their ability to act as electron reserviors and their potential in catalysis [12]. Features of the chemistry of these molecules which are currently of interest include the mechanisms and sites of substitution as well as the modification of reactivity accompanying carbonyl replacement by donor ligands [13].

In this paper, we report a novel photochemical route to the hitherto unknown five new complexes [$M(CO)_5$(DTTT)] [M = Cr; **1**, Mo; **2**, W; **3**], [$Re(CO)_4Br$(DTTT)] (**4**) and [$Mn(CO)_2Cp$(DTTT)] (**5**) have been prepared by the photochemical reaction of metal carbonyls [$M(CO)_6$] (M = Cr, Mo, W), [$Re(CO)_5Br$], [$Mn(CO)_3Cp$] with DTTT. The complexes were characterized by elemental analyses, electron impact mass spectrometry, FTIR, ^1H and $^{13}C\{^1H\}$ NMR spectroscopy. According to the DTTT ligands coordinates to the metal via (C=S) sulfur donor atom in **1**–**5**. An X-ray diffraction study of [$W(CO)_5$(DTTT)] (**3**) confirms that the tungsten adopts a distorted octahedral geometry with local C_{4v} symmetry.

2. Experimental

2.1. General

Reactions were carried out under dry N_2 using Schlenk techniques. All solvents were dried and degassed prior to use. Elemental analyses were carried out using a LECO-CHNS-O-9320 by Technical and Scientific Research Council of Turkey, TUBITAK. FTIR spectra were recorded on samples in CH_2Cl_2 at the Ege University on a Mattson 1000 FTIR spectrophotometer. ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded in $CDC1_3$ on 400 MHz High Performance Digital FT NMR at TÜBITAK. Electron impact mass Spectroscopy; Micromass VG Platform-II LC-MS were recorded at TÜBITAK. UV irradiations were performed with a medium-pressure 400 W mercury lamp through a quartz-walled immersion well reactor.

Pentane, benzene, hexane, CH_2Cl_2, Me_2CO, EtOH, Et_2CO, silica gel were purchased from Merck and $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $Mn(CO)_3Cp$ were purchased from Aldrich, and were used as supplied. DTTT were prepared by the literature method [14].

2.2. Synthesis

The complexes, [$M(CO)_5$(DTTT)] [M = Cr; **1**, Mo; **2**, W; **3**], [$Re(CO)_4Br$(DTTT)] (**4**) and [$Mn(CO)_2Cp$-(DTTT)] (**5**) were prepared by the photochemical reactions of metal carbonyls $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $Mn(CO)_3Cp$ with DTTT and were obtained in 80% yields by similar methods of which the following is typical.

The complex $Cr(CO)_6$ (0.44 g, 2 mmol) and DTTT (0.32 g, 2 mmol) were dissolved in THF (80–100 cm^3). The solution was irradiated for 2 h. During the irradiation, the reaction mixture changed from colorless to yellow. After the irradiation, the reaction mixture was evaporated under vacuum, yielding a dark yellow solid. After dissolving in CH_2Cl_2 (10 cm^3), petroleum ether (50 cm^3) was added, resulting in the precipitation of a yellow solid which was washed with petroleum ether and dried under vacuum. Yield [$Cr(CO)_5$DTTT]: 79%.

Anal. Calc. for $C_{10}H_{10}O_5N_2S_2Cr$, **1**: C, 33.9; H, 2.84; N, 7.91; S, 18.1%. Found: C, 34.4; H, 2.69; N, 8.15; S, 18.49%. *Anal*. Calc. for $C_{10}H_{10}O_5N_2S_2Mo$, **2**: C, 30.2; H, 2.53; N, 7.03; S, 16.1%. Found: C, 30.26; H, 2.44; N, 7.3; S, 16.4%. *Anal*. Calc. for $C_{10}H_{10}O_5N_2S_2W$, **3**: C, 24.17; H, 2.07; N, 5.76; S, 13.19%. Found: C, 24.5; H, 2.1; N, 5. 5; S, 13.4%. *Anal*. Calc. for $C_9H_{10}O_4N_2S_2BrRe$, **4**: C, 20.0; H, 1.87; N, 5.18; S, 11.87%. Found: C, 20.3; H, 1.80; N, 5.4; S, 11.8%. *Anal*. Calc. for $C_{12}H_{15}O_2N_2S_2Mn$, **5**: C, 42.6; H, 4.5; N, 8.3; S, 18.9%. Found: C, 42.4; H, 4.8; N, 8.6; S, 18.8%.

1: ν(CO) 2061(s), 1974(s), 1941(s), 1917(s), 1888(m) cm^{-1}; ν_{sym} (C=S) 668.7 cm^{-1}; ν_{as}(C=S) 610.6 cm^{-1}; [^1H] NMR: $\delta = 2.48$ (s, 3H), 3.31 (s, 3H), 1.29 (s, 2H), 4.36 (s, 2H). [^{13}C] NMR: $\delta = 39.3$, 40.4 (CH_3); 60.8, 73.5 (CH_2); 192.0 (C(S)S); 222.5 (s, 1C, ax. CO), 215.3 (s, 4C, eq. CO). **2**: ν(CO) 2069(s), 1963(s), 1931(s), 1906(s), 1898(m) cm^{-1}; ν_{sym} (C=S) 667.6 cm^{-1}; ν_{as}(C=S) 606.6 cm^{-1}; [^1H] NMR: $\delta = 2.59$ (s, 3H), 3.46 (s, 3H), 1.36 (s, 2H), 4.37 (s, 2H). [^{13}C] NMR: $\delta = 39.7$, 41.1 (CH_3); 60.3, 73.8 (CH_2); 192.9 (C(S)S); 224.2, (s, 1C, ax. CO), 205.4 (s, 4C, eq. CO). **3**: ν(CO) 2068(s), 1969(s), 1942(s), 1910(s), 1880(m) cm^{-1}; ν_{sym} (C=S) 669.6 cm^{-1}; ν_{as}(C=S) 597.6 cm^{-1}; [^1H] NMR: $\delta = 2.48$ (s, 3H), 3.38 (s, 3H), 1.25 (s, 2H), 4.39 (s, 2H). [^{13}C] NMR: $\delta = 39.4$, 40.5 (CH_3); 60.8, 73.8 (CH_2); 193.3 (C(S)S); 203.7 (s, 1C, ax. CO), 199.3 (s, 4C, eq. CO). **4**: ν(CO) 2102(w), 2009(s), 1978(s), 1910(s), 1890(m) cm^{-1}; ν_{sym} (C=S) 656.6 cm^{-1}; ν_{as}(C=S) 619.6 cm^{-1}; [^1H] NMR: $\delta = 2.48$ (s, 3H), 3.44 (s, 3H), 1.26 (s, 2H), 4.37 (s, 2H). [^{13}C] NMR: $\delta = 39.3$, 40.5 (CH_3); 60.8, 73.5 (CH_2); 192.3 (C(S)S); 216.5 (s, 1C, CO), 214.4 (s, 2C, CO), 187.8 (s, 1C, CO). **5**: ν(CO) 1917(s), 1843(m) cm^{-1}; ν_{sym} (C=S) 667.6 cm^{-1}; ν_{as}(C=S) 610.6 cm^{-1}; [^1H] NMR: $\delta = 2.54$ (s, 3H), 3.41 (s, 3H), 1.28 (s, 2H), 4.37 (s, 2H). [^{13}C] NMR: $\delta = 39.3$, 40.5 (CH_3); 60.9, 71.6 (CH_2); 192.3 (C(S)S); 222.3 (s, 1C, CO), 215(s, 1C, CO).

m/z **1** (MW = 354.3): 354(55), [M^+]; 326(30), [M^+–(CO)]; 298 (40), [M^+–(2CO)]; 270(40),[M^+–(3CO)]; 242(50), [M^+–(4CO)]; 214(20), [M^+–(5CO)]. **2** (MW = 398.3): 398(20), [M^+]; 370(20), [M^+–(CO)]; 342 (10), [M^+–(2CO)]; 314(25),[M^+–(3CO)]; 258(20), [M^+–(5CO)]. **3** (MW = 486.2): 486(15), [M^+]; 458(10), [M^+–(CO)]; 402 (15), [M^+–(3CO)]; 374(10),[M^+–(4CO)]; 346(20), [M^+–(5CO)]. **4** (MW = 540.4): 540(5), [M^+]; 460(10), [M^+–(Br)]; 432 (5), [M^+–

(Br+CO)]; 404(10),[M^+–(Br+2CO)]; 376(15), [M^+–(Br+3CO)]; 348(15), [M^+–(Br+4CO)]. **5** (MW = 338.3): 338(10), [M^+]; 310(15), [M^+–(CO)]; 382 (10), [M^+–(2CO)].

For the mass spectral data relative intensities are given in parentheses; probable assignments in square brackets. For all assignments the most abundant isotopes of Cr, Mo, W, Re and Mn have been selected (^{52}Cr, 83.76%, ^{98}Mo, 24%, ^{184}W, 30.7%, ^{187}Re, 62.9%, ^{55}Mn, 100% abundant).

2.3. Crystallography

A single crystal of **3** was mounted on a glass fibre. Diffraction measurements were made on a Siemens SMART CCD automatic diffractometer using graphite-monochromated Mo Kα radiation. The unit cell was determined from randomly selected reflections obtained using the SMART CCD automatic search, center index and least-squares routines. Crystal data and collection parameters are listed in Table 1. Integration was carried out using the program SAINT and an absorption correction was performed using SADABS. Structure solution was carried out using the SHELXTL-5.1 suite of programs. The space group $P\bar{1}$ was chosen based upon systematic absences. The initial solution was obtained by direct methods and refined by successive least-squares cycles.

Table 1
Crystal data and structure refinement of **3**

Empirical formula	$C_{10}H_{10}N_2O_5S_2W$
Formula weight	972.34
Temperature (K)	173(2)
Wavelength (Å)	0.71070
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
a (Å)	7.3208(5)
b (Å)	9.4916(5)
c (Å)	11.5258(9)
α (°)	96.2820(10)
β (°)	108.3150(10)
γ (°)	100.7840(10)
V (Å^3)	734.63(9)
Z	2
D_{calc} (Mg m^{-3})	2.198
Absorption coefficient (mm^{-1})	8.164
$F(0\,0\,0)$	460
θ Range for data collection (°)	1.89–28.73
Lomiting indices	$-9 \le h \le 9$, $-12 \le k \le 12$, $-15 \le l \le 15$
Reflections collected	8746
Independent reflections	3775
Completeness to θ	99.5
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	3775/0/182
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0255$, $wR_2 = 0.0656$
R indices (all data)	$R_1 = 0.0246$, $wR_2 = 0.0651$
Goodness-of-fit on F^2	1.098
Largest difference peak and hole (e Å^{-3})	

3. Result and discussion

The photogeneration of $M(CO)_5$ from $M(CO)_6$ (M = Cr, Mo or W) has been extensively studied. These 16-electron $M(CO)_5$ fragments react avidly with any available donor to form $M(CO)_5L$ species, and where L is a chelating bidentate ligand, rapid continuation to the chelating $M(CO)_4L$ or bridging $M_2(CO)_{10}(\mu\text{-}L)$ products may occur [15–18]. In this study, photochemical reactions of $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $[Mn(CO)_3Cp]$ with DTTT ligand proceed in this expected manner, and gave a series of complexes **1**–**5** (Fig. 1).

As expected, five bands arising from ν(CO) vibrations are seen for complexes **1**–**3** which presumably have the local C_{4v} $(2A_1+E)$ symmetry of the $M(CO)_5$ unit (M = Cr, Mo, W). The ν(CO) modes of **1**–**3** move to lower wavenumbers when compared with the starting $M(CO)_6$ (M = Cr, Mo, W) molecules [17,18]. Similarly, there are four and two CO stretching mode in the IR spectra of **4** and **5**, respectively. According to IR spectra, the $Re(CO)_4$ unit in compound **4** has local C_{2v} $(2A_1+B_1+B_2)$ symmetry and the $Mn(CO)_2$ unit [19] in **5** has local C_{2v} (A_1+B_1) symmetry [20]. These modes also shifts to lower wave numbers when compared with that of the $Re(CO)_5Br$ and $Mn(CO)_3Cp$.

The mass spectra show fragmentation via successive loss of CO groups and fragmentation of the organic ligands.

Single crystals of [W(CO)$_5$(DTTT)] (**3**) were grown by slow diffusion of hexanes into a CH_2Cl_2 solution and an ORTEP diagram of the molecule is shown in Fig. 2 and selected bond lengths and angles are given in Table 2. X-ray diffraction of **3** shows that the DTTT group coordinates via sulfur (C=S) atom to the metal center. The sulfur atom of the DTTT is bonded trans to CO group, with the remaining CO groups occupying equa-

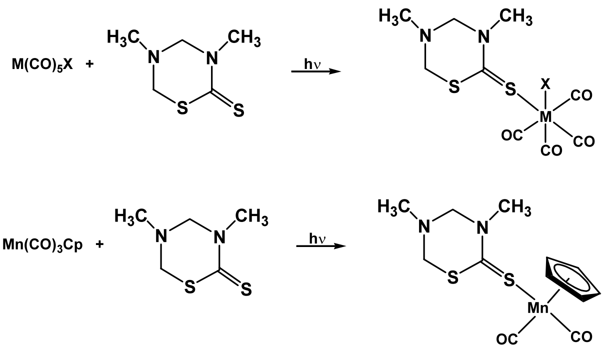

Fig. 1. The photochemical reactions of $M(CO)_6$ (M = Cr, Mo and W), $Re(CO)_5Br$ and $[Mn(CO)_3Cp]$ with DTTT ligand.

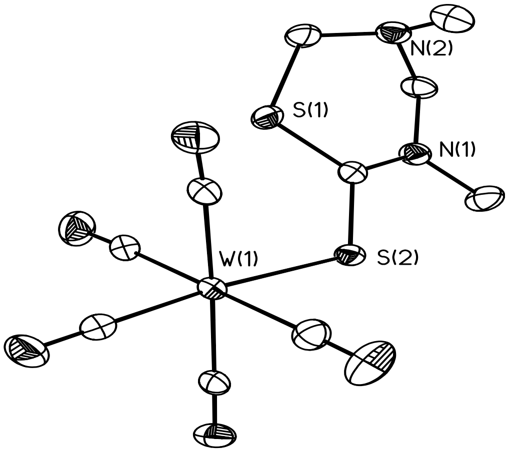

Fig. 2. Molecular structure of [W(CO)$_4$(DTTT)] (**3**). Thermal ellipsoids are shown at the 50% probability level and hydrogen atoms have been ommitted.

Table 2
Selected bond lengths (Å) and angles (°) for **3**

Bond length			
W(1)–C(8)	1.980(4)	S(2)–C(1)	1.708(4)
W(1)–C(9)	2.028(4)	N(1)–C(4)	1.465(5)
W(1)–C(7)	2.035(4)	N(1)–C(2)	1.504(5)
W(1)–C(6)	2.056(4)	N(1)–C(3)	1.416(5)
W(1)–C(10)	2.057(4)	N(1)–C(2)	1.425(5)
W(1)–S(2)	2.5687(9)	N(1)–C(5)	1.463(6)
S(1)–C(1)	1.732(4)	S(1)–C(3)	1.877(4)
Bond angles			
C(8)–W(1)–C(9)	88.22(15)	C(10)–W(1)–S(2)	86.44(11)
C(8)–W(1)–C(7)	89.10(16)	C(1)–S(1)–C(3)	102.69(17)
C(9)–W(1)–C(7)	91.24(16)	C(1)–S(2)–W(1)	116.06(12)
C(8)–W(1)–C(6)	86.88(16)	C(1)–N(1)–C(4)	121.2(3)
C(9)–W(1)–C(6)	174.29(13)	C(1)–N(1)–C(2)	123.4(3)
C(7)–W(1)–C(6)	91.63(16)	C(4)–N(1)–C(2)	115.4(3)
C(8)–W(1)–C(10)	91.74(16)	C(3)–N(2)–C(2)	111.5(3)
C(9)–W(1)–C(10)	87.85(16)	C(3)–N(2)–C(5)	114.9(3)
C(7)–W(1)–C(10)	178.74(14)	C(2)–N(2)–C(5)	113.8(3)
C(6)–W(1)–C(10)	89.36(17)	N(1)–C(1)–S(2)	120.6(3)
C(8)–W(1)–S(2)	174.53(11)	N(1)–C(1)–(1)	122.6(3)
C(9)–W(1)–S(2)	86.55(10)	S(2)–C(1)–S(1)	116.8(2)
C(7)–W(1)–S(2)	92.63(11)	N(2)–C(2)–N(1)	113.3(3)
C(6)–W(1)–S(2)	98.26(11)	N(2)–C(3)–S(1)	113.3(3)

torial sites. The tungsten atom is thus six-coordinate with a somewhat distorted octahedral arrangement of the coordinating atoms. The Re–S [2.5687(9) Å] and C=S [1.708(4) Å] distance are in line with those observed for related complexes.

For example, in the molecules Re(CO)$_4$[μ-S$_2$CNHC(NCO$_2$Et)$_5$]Re(CO)$_5$ (**A**) and Re(CO)$_4$[S-*trans*-μ-C,S–EtO$_2$–CN=CS]Re(CO)$_5$ (**B**) and (CO)$_5$W=C(NEt$_2$)CH–CS$_2$[Re(CO)$_5$]$_2$ (**C**) the average W–S distance 2.5028 Å and the average C–S distance 1.704 Å [21–23]. The C–S distances in **3** are intermediate between those associated with formal single [1.80 Å] [21–24] and double [1.671 Å] carbon sulfur bond [21–24]. This finding suggests the retention of a degree of multiple-bond character in C–S bond [21–24].

In addition, the values for the W–S–C angles [116.06(12) (°)], close to trigonal, indicates formal sp^2 hydridization for sulfur, as reported for the related rhenium compounds **A**–**C** [18–20]. These facts support the suggestion that the carbon–sulfur can be regarded as moderate σ-donor ligands with π-acceptor properties [25].

We have presented the novel photochemical reactions between [M(CO)$_6$] (M = Cr, Mo and W), [Re(CO)$_5$Br], [Mn(CO)$_3$Cp] and DTTT. The spectroscopic studies show that DTTT behaves as a monodentate ligand coordinating via S donor atom in **1**–**5**. An X-ray diffraction study of [W(CO)$_5$(DTTT)] (**3**) confirms that the tungsten adopts a distorted octahedral geometry. The IR spectroscopic results show that DTTT coordinates via sulfur (C=S) atom in **1**–**5**. The ν(CO) modes of **1**–**5** move to low wavenumber when compared with the starting [M(CO)$_6$] (M = Cr, Mo and W), [Re(CO)$_5$Br], [Mn(CO)$_3$Cp] molecules. The mass spectrum shows fragmentation via successive loss of CO groups and fragmentation of the DTTT ligand. In addition, the spectroscopic study of **3** is entirely keeping with the single crystal X-ray diffraction study.

4. Supplementary material

A complete list of bond lengths and angles and tables of atomic coordinates and thermal parameters have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 192392. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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