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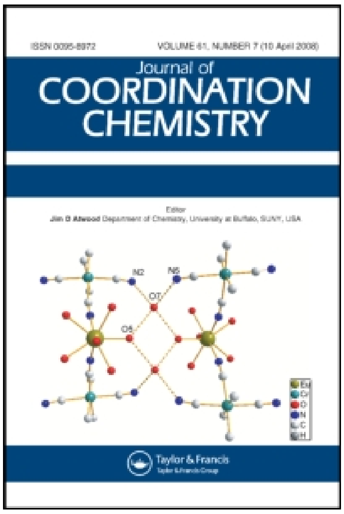

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Photochemical reactions of metal carbonyls [$M(CO)_6$ (M=Cr, Mo, W), $Re(CO)_5Br$, $Mn(CO)_3Cp$] with salicylaldehyde ethanesulfonylhydrazone (Hsalesh)

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Photochemical reactions of metal carbonyls [$M(CO)_6$ ($M = Cr$, Mo, W), $Re(CO)_5Br$, $Mn(CO)_3Cp$] with salicylaldehyde ethanesulfonylhydrazone (Hsalesh)

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The new complexes, $M(CO)_5$(Hsalesh) ($M = Cr$ (**1**), Mo (**2**), W (**3**)), $Re(CO)_4Br$(Hsalesh) (**4**) and $Mn(CO)_3$(Hsalesh) (**5**) have been synthesized by the photochemical reaction of metal carbonyls $M(CO)_6$ ($M = Cr$, Mo, W), $Re(CO)_5Br$, and $Mn(CO)_3Cp$ with salicylaldehyde ethanesulfonyl hydrazone (Hsalesh). The complexes have been characterized by elemental analysis, EI mass spectrometry, FT-IR and ^1H NMR spectroscopy. The spectroscopic studies show that Hsalesh behaves as a monodentate ligand coordinating *via* imine N donor atom in $M(CO)_5$(Hsalesh) ($M = Cr$, Mo, W) and $Re(CO)_4Br$(Hsalesh) and as a tridentate ligand in $Mn(CO)_3$(salesh).

Keywords: Hydrazone; Salicylaldehyde ethanesulfonylhydrazone; Metal carbonyls

1. Introduction

Sulfonamide drugs are widely used chemotherapeutic agents with a large spectrum of activity [1]. Methanesulfonamide derivatives exhibit a cytostatic effect, and some sulfonamides, like amsacrine, find application in cancer chemotherapy [2–5]. The methanesulfonamide residue has also appeared as a suitable pharmacophoric equivalent to replace functional groups in drug design [6]. On the other hand, many compounds containing a hydrazine fragment, *e.g.* carboxylic acid hydrazides and their Schiff bases, have shown cytostatic activity [7]. A potent antineoplastic effect has been registered for some sulfonylhydrazines [8]. Schiff bases of methanesulfonylhydrazine, $Me_3S(O)_2NHNH_2$, have both sulfonamide and hydrazine fragments, and exhibit antibacterial and cytotoxic effects [9–11].

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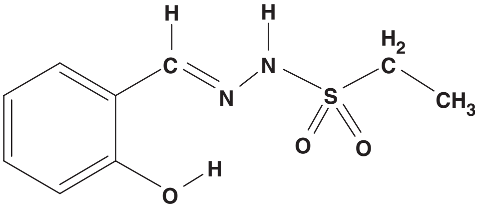
Although polydentate Schiff bases stabilize a variety of chelates via metal-nitrogen bond formation, their utility as potential decarbonylating agents of metal carbonyls such as chromium hexacarbonyl $Cr(CO)_6$ have not been studied in sufficient detail. The coordination of neutral monodentate Schiff bases, *i.e.* diarylamines with $Cr(CO)_6$ has been reported [19–21].

The Schiff-base metal carbonyl complexes have continued to attract attention in part because of the different possible coordination geometries which the ligand may adopt [12–14]. Their low energy metal-to-ligand charge transfer (MLCT) transitions make these molecules attractive for luminescence and electron transfer reactions [15]. Several of these complexes have been shown to be effective catalysts in allylic alkylation reactions [16–17] and in the activation of aromatic carbon–hydrogen bonds (ortho-metallation) via intramolecular η^2-bonding of arenes [18]. For stereoselective organic transformations, chiral metal complexes, which may have a chiral metal center or a chiral coordinated ligand or both, have been employed [19, 20].



(Hsalesh)

In this article, five new complexes $[M(CO)_5(Hsalesh)]$ (**1**–**4**) and $[Mn(CO)_3(Hsalesh)]$ (**5**) have been prepared for the first time, by the photochemical reaction of metal carbonyls $[M(CO)_6]$ (M = Cr, Mo, W), $[Re(CO)_5Br]$, $[Mn(CO)_3Cp]$ with 2-hydroxyacetophenone methanesulfonylhydrazone (Hsalesh). The complexes were characterized by elemental analyses, electron impact mass spectrometry, f.t.-i.r., ^1H and $^{13}C\text{-}\{^1H\}$-nmr spectroscopy. The Hsalesh ligands coordinate to the metal by N imine donor atom in $[M(CO)_5(Hsalesh)]$ (**1**–**4**). However, the Hsalesh ligand is tridentate via phenolic O, imine N and amine N donor atoms in $[Mn(CO)_3(salesh)]$ (**5**).

We have now investigated five new complexes **1**–**5** which have been prepared for the first time, by the photochemical reaction of metal carbonyls $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$, $Mn(CO)_3Cp$ with salicylaldehyde ethanesulfonylhydrazone (Hsalesh).

2. Experimental

2.1. *Apparatus*

Elemental analyses were carried out using a LECO-CHNS-O-9320 by Technical and Scientific Research Council of Turkey, TUBİTAK.

FTIR spectra were recorded on samples in hexane at the Ege University on a Mattson 1000 FT spectrophotometer.

^1H NMR spectra were recorded in DMSO on a 400 MHz Digital FT-NMR at TUBİTAK. Electron impact mass spectra were recorded on a Micromass VG Platform-II LC-MS at TÜBİTAK.

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UV irradiations were performed with a medium-pressure 400 W mercury lamp through a quartz-walled immersion well reactor.

2.2. *Materials*

The solvents, salicylaldehyde, ethanesulfonyl chloride, hydrazine hydrate, and 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. These reagents were used as supplied. Salicylaldehyde ethanesulfonylhydrazone (Hsalesh) was prepared by the literature method [9].

2.3. *Preparation of complexes*

Reactions were carried out under dry nitrogen using Schlenk techniques. All solvents were dried and degassed prior to use.

The complexes, $M(CO)_5$(Hsalesh) (M = Cr (**1**), Mo (**2**), W (**3**)), $Re(CO)_4Br$(Hsalesh) (**4**), and $Mn(CO)_3$(salesh) (**5**) were prepared by photochemical reactions of metal carbonyls $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $Mn(CO)_3Cp$ with salicylaldehyde ethanesulfonylhydrazone (Hsalesh) and were obtained in 70–80% yields by methods of which the following is typical.

The complex $Cr(CO)_6$ (0.44 g, 2 mmol) and Hsalesh (0.45 g, 2 mmol) were dissolved in THF (80–100 mL). The solution was irradiated for 2 h. During the irradiation, the color of the reaction mixture changed from colorless to dark yellow. After the irradiation, the reaction mixture was evaporated under vacuum, yielding a light yellow solid. After dissolving in dichloromethane (10 mL), 50 mL of petroleum ether was added, resulting in the precipitation of a dark yellow solid which was washed with petroleum ether and dried under vacuum. Yield of [$Cr(CO)_5$(Hsalesh)]: 79%.

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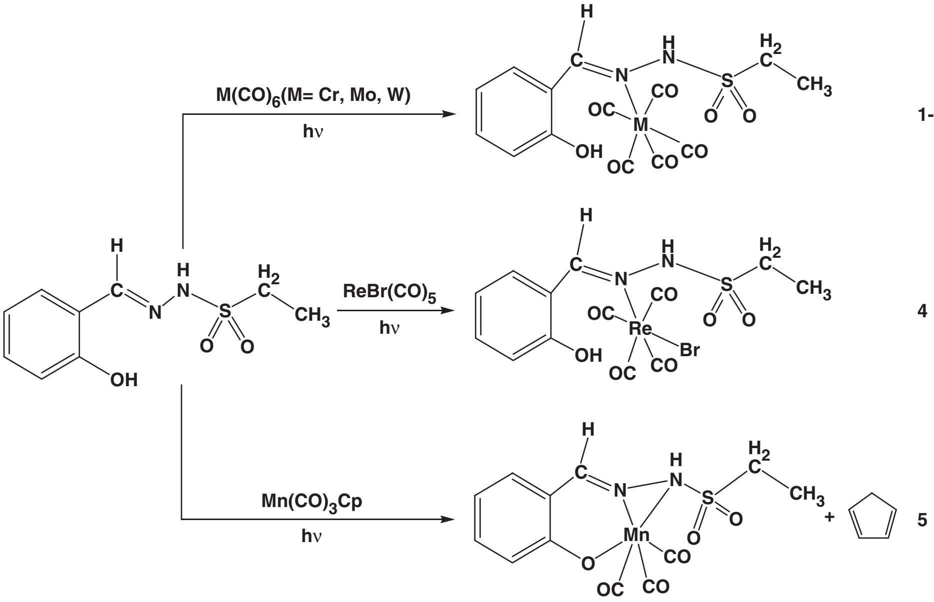
3. Results and discussion

Complexes **1**–**5** were prepared by a photochemical reaction as shown in scheme 1. The photogeneration of $M(CO)_5$ from $M(CO)_6$ (M = Cr, Mo, W) has been extensively studied. These 16-electron $M(CO)_5$ fragments react quickly with any available donor atom to form a $M(CO)_5L$ species. If L is a bidentate ligand, $M(CO)_4L$ chelate or bridging $M_2(CO)_{10}(\mu\text{-}L)$ compounds may occur [21–23]. In this study, photochemical reactions of $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $Mn(CO)_3Cp$ with Hsalesh proceed in this expected manner, and gave complexes **1**–**5**. The analytical results, the yields and the colors of the novel complexes **1**–**5** are given in table 1. **1**–**5** are air stable complexes.

Selected infrared spectral data of **1**–**5** are presented in table 2. The rather strong C=N stretching vibration, found at 1614 cm^{-1} in the free ligand, shifts to lower wavenumber in **1**–**5**, showing that the Hsalesh ligand coordinates to the metal via the imine donor atom [24]. This shift has been explained as a weakening of the CN bond resulting from the loss of electron density from the nitrogen to the metal atom [24]. No shifting upon complex formation was observed for the $\nu_{as}(SO_2)$, $\nu_{sym}(SO_2)$, $\nu(NH)$ and $\nu(CO)$



Scheme 1. Synthesis of complexes **1**–**5** from $M(CO)_6$ (M = Cr, Mo, W), $Re(CO)_5Br$ and $Mn(CO)_3Cp$ and the Hsalesh ligand.

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Table 1. Elemental analysis results and yields of **1**–**5**.

		Found (Calcd) (%)			
Complexes[a]	Yield (%)	C	H	N	S
1	77	39.91 (40.01)	2.67 (2.88)	6.55 (6.66)	7.46 (7.63)
2	78	36.59 (36.22)	2.79 (2.61)	6.31 (6.03)	6.71 (6.91)
3	80	30.69 (30.45)	2.22 (2.19)	4.89 (5.07)	6.06 (5.81)
4	68	25.38 (25.75)	2.05 (1.99)	4.69 (4.62)	5.19 (5.29)
5	70	39.67 (39.25)	3.42 (3.29)	7.52 (7.63)	8.61 (8.73)

[a] Light yellow.

Table 2. Selected infrared spectral bands (cm^{-1}) of **1**–**5** and Hsalesh.

Complex	ν(CO)	ν(N–H)	ν(C=N)	ν(C–O)	$\nu_{as}(SO_2)$	$\nu_{sym}(SO_2)$
Hsalesh [9]	–	3213s	1622m	1238s	1339s	1167s
1	2066m, 1935br, 1917sh	3166s	1606m	1270s	1333s	1166s
2	2068m, 1939br, 1912sh	3167s	1607m	1270s	1332s	1167s
3	2069m, 1940br, 1932sh	3166s	1605m	1270s	1334s	1165s
4	2102w, 2021m, 1924m, 1912m	3158s	1608m	1266s	1334s	1168s
5	2018s, 1923s, 1896s	–	1602m	1280s	1318s	1156s

stretching vibrations indicating that SO_2, NH and CO groups were not coordinated in **1**–**4**. The OH stretching vibration was not observed for the free ligand or **1**–**4** because of hydrogen bonding with the imine nitrogen atom in ligand [9] and with amine nitrogen atom in **1**–**4** [31–36].

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Table 3. ^1H NMR spectroscopic data for compounds **1**–**5** in DMSO-d_6 (ppm).

Complex	CH_3–	CH_2–	$(CH)_{Ar}$	HC=N–	NH	OH
Hsalesh [9]	1.19, s, 3H	3.16, s, 2H	6.85, d[a], 1H 6.89, t[b], 2H 7.25, ddd[c], 1H 7.46, dd[d], 2H	8.20, s[b], 1H	10.23, s, br[e], 1H	11.22, s, br, 1H
1	1.23, s, 3H	3.20, s, 2H	6.84, d[a], 1H 6.94, t[b], 2H 7.35, ddd[c], 1H 7.62, dd[d], 2H	8.01, s[b], 1H	10.48, s, br[e], 1H	11.28, s, br, 1H
2	1.22, s, 3H	3.19, s, 2H	6.47, d[a], 1H 6.92, t[b], 2H 7.24, ddd[c], 1H 7.54, dd[d], 2H	8.01, s[b], 1H	10.42, s, br[e], 1H	11.18, s, br, 1H
3	1.24, s, 3H	3.10, s, 2H	6.66, d[a], 1H 6.78, t[b], 2H 7.04, ddd[c], 1H 7.32, dd[d], 2H	8.02, s[b], 1H	10.41, s, br[e], 1H	11.20, s, br, 1H
4	1.28, s, 3H	3.12, s, 2H	6.68, d[a], 1H 6.83, t[b], 2H 7.17, ddd[c], 1H 7.56, dd[d], 2H	8.01, s[b], 1H	10.41, s, br[e], 1H	11.08, s, br, 1H
5	1.20, s, 3H	3.10, s, 2H	6.56, d[a], 1H 6.80, t[b], 2H 7.14, ddd[c], 1H 7.56, dd[d], 2H	8.02, s[b], 1H	11.98, s, br[e], 1H	–

[a]doublet, [b]triplet, [c]doublet of doublet of doublet, [d]doublet of doublet, [e]broad.

The number of carbonyl bands provides important clues to the environment of the metal centers [25]. The carbonyl stretching bands in **1**–**3** are attributed to local C_{4v} symmetry of $M(CO)_5$ [21, 22]. Similarly, four CO stretching absorptions in **4** and three in **5** indicate *local* C_{2v} [26] and C_s [27] symmetry, respectively (shown in scheme 1). The ν(CO) modes in **1**–**5** move to lower wavenumber compared with the starting carbonyl complexes [21, 22].

^1H NMR spectral data in DMSO-d_6 solutions of compounds **1**–**5** are collected in table 3. In the ^1H NMR spectra of **1**–**4**, an upfield shift of about 0.19 ppm for the HC=N proton and 0.2 ppm for the NH proton relative to the free ligand were observed. The small shift is related to a decrease in π-electron density in the C=N bond with complex formation in **1**–**4**. According to these data, the Hsalesh ligand is monodentate in **1**–**4**.

However, in the NMR spectrum of Mn(salesh)$(CO)_3$ (**5**), the phenolic OH signal is absent, in agreement with the formation of a Mn–O bond [28, 29, 32–36]. The *NH* signal was found 1.75 ppm downfield compared to the free ligand in **5**. In addition, the shift of the C–O stretching vibration to higher wavenumbers in the IR spectrum shows that both imine N and phenolic O donor atoms coordinate to the Mn atom. Kinematic coupling of the CO group with the bonded metal ion would increase the frequencies [30, 32–36]. According to the data, the Hsalesh ligand is tridentate and ionic in **5** [32–36]. The Hsalesh ligand must act as a 6-electron donor in order to satisfy the 18-electron rule.

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

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Table 4. The mass spectral data of **1**–**5**.

Complex	MW	Relative intensities of the ions m/e and assignment[a]
1	420	392 (10) [$M^+ - CO$], 364 (15) [$M^+ - 2CO$], 336 (25) [$M^+ - 3CO$], 280 (10) [$M^+ - 5CO$].
2	464	449 (10) [$M^+ - Me$], 421 (15) [$M^+ - Me - CO$], 365 (20) [$M^+ - Me - 3CO$], 337 (15) [$M^+ - Me - 4CO$], 294 (15) [$M^+ - Et - 5CO$].
3	522	537 (10) [$M^+ - Me$], 509 (15) [$M^+ - Me - CO$], 481 (10) [$M^+ - Me - 2CO$], 453 (20) [$M^+ - Me - 3CO$], 397 (15) [$M^+ - Me - 5CO$].
4	606	576 (10) [$M^+ - Et$], 520 (25) [$M^+ - Et - 2CO$], 492 (15) [$M^+ - Et - 3CO$], 464 (30) [$M^+ - Et - 4CO$].
5	367	339 (15) [$M^+ - CO$], 311 (15) [$M^+ - 2CO$], 283 (35) [$M^+ - 3CO$].

[a] 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) [33, 35, 36].

4. Conclusions

Hsalesh behaves as monodentate ligand via N imine donor atom in **1**–**4**, but as a tridentate ligand via anionic O, imine N and amine N donor atoms in **5**.

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