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# High temperature microcalorimetric studies of the thermal decomposition and halogenation of nonacarbonylcobaltmanganese, nonacarbonylcobaltrhenium and decacarbonylmanganeserhenium

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**Abstract**

Microcalorimetric measurements at elevated temperatures of the enthalpies of sublimation, bromination and iodination of $CoMn(CO)_9$, $CoRe(CO)_9$ and $MnRe(CO)_{10}$ have been made to obtain the standard enthalpies of formation $\Delta_fH^0$ (c) and $\Delta_fH^0$ (g) (in that order) as follows (values in kJ mol$^{-1}$): $CoMn(CO)_9$ $-(1460 \pm 36)$, $-(1388 \pm 42)$; $CoRe(CO)_9$ $-(1452 \pm 36)$, $-(1369 \pm 42)$; $MnRe(CO)_{10}$ $-(1630 \pm 24)$, $-(1544 \pm 28)$. The values of $\Delta_fH^0_m$ (g) are used to derive the enthalpy contributions of the metal–metal bonds in these molecules D(Co–Mn) = $(114 \pm 23)$, D(Co–Re) = $(122 \pm 25)$ and D(Mn–Re) = $(145 \pm 29)$ kJ mol$^{-1}$. In each case, the value of D(M–M′) is less than the mean of the corresponding homometallic bond enthalpy contributions. 

*Keywords:* Calorimetry; Cobalt; Manganese; Rhenium

## 1. Introduction

The strengths of bonds between transition metal atoms in molecules have been a subject of vigorous debate for many years [1]. This debate has focussed on homometallic systems, such as $Mn_2(CO)_{10}$ [2] and osmium clusters [3], for the most part. The problem of identifying the enthalpy contribution of a metal–metal bond of multiple order in a molecule such as $Mo_2(NMe_2)_6$ has also been addressed [4]. Very little attention has been paid to the estimation of the enthalpy contribution of a metal–metal bond in a heterometallic system. The enthalpy of reaction between $MnRe(CO)_{10}$ and diiodine in cyclohexane solution has been measured by photocalorimetry [5]. An estimate has been made of the dissociation energy D(Mn–Re) based on mass spectrometry [6]. Measurements of the kinetics of the reaction between $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$ to form $MnRe(CO)_{10}$ in solution have been reported [7].

## 2. Experimental

The thermal measurements were made using a Calvet twin-cell high temperature microcalorimeter (Setaram, Lyon), adapted to the drop calorimetric technique [8]. Heats of sublimation were measured by using the microcalorimetric vacuum sublimation method [9]. All measurements were made in an argon atmosphere. Measurements of the initial temperature of the sample in the calorimeter, that is to say the temperature reached by the sample on falling from the entrance to the chute into the cell using diiodine as calibrant, indicated that this was in the range 321–324 K for an operating temperature of 550–620 K.

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### 2.1. Materials

Samples of nonacarbonylcobaltmanganese, an orange–red solid [10,11], and nonacarbonylcobaltrhenium, an orange–brown solid [11,12], and decacarbonylmanganeserhenium, a pale yellow solid [11,12] were prepared by the methods quoted. The purity of each compound was established by microanalysis and by spectroscopic measurements (IR, mass) which were in agreement with published values. All of the solid compounds are air-sensitive; the cobalt-containing compounds, particularly so. The cobalt-containing compounds are also remarkably photosensitive, so that all manipulations were carried out in subdued light. The carefully purified samples were stored in an argon atmosphere at ca. 250 K; their purity was checked (IR) regularly. All glassware was oven-dried and cooled in a dinitrogen atmosphere. Octacarbonyldicobalt, decacarbonyldimanganese and decacarbonyldirhenium were purchased (Strem) and were purified, if necessary, by sublimation.

### 2.2. Auxiliary data

The following enthalpy of formation data (in kJ $mol^{-1}$) were used in evaluating the calorimetric results: CO(g) = $-(110.52 \pm 0.17)$ [13]; Br(g) = $(111.84 \pm 0.12)$ [13]; $Br_2$(g) = $(30.907 \pm 0.11)$ [13]; I(g) = $(106.76 \pm 0.04)$ [13]; $I_2$(g) = $(62.44 \pm 0.04)$ [13]; Co(g) = $(424.7 \pm 0.5)$ [14]; $CoBr_2$(c) = $-(220.9 \pm 2)$ [14]; $CoI_2$(c) = $-(88.7 \pm 2)$ [14]; Mn(g) = $(283.5 \pm 0.2)$ [15]; $MnBr_2$(c) = $-(385.8 \pm 1.7)$ [14]; $MnI_2$(c) = $-(266.1 \pm 1.8)$ [14]; Re(g) = $(775.7 \pm 6.3)$ [16]; $ReBr_3$(c) = $-(175.7 \pm 5)$ [16]; $ReI_3$(c) = $-(42 \pm 20)$ [17]; $Co(CO)_4$(g) = $-(561 \pm 12)$ [18]; $Mn(CO)_5$(g) = $-(713 \pm 11)$ [2]; $Re(CO)_5$(g) = $-(686 \pm 6)$ [17]. The value of $\Delta_f H^0_m(MX_n$ (n < 2), c) was assumed equal to $0.5n[\Delta_f H^0_m(MX_2$, c)] (M is Co, Mn; X is Br, I) in agreement with previous practice [8,19]. The enthalpies of reaction $\Delta H^T$, measured at temperature $T$/K were adjusted to refer to 298 K by using data from Barin [14] and the JANAF tables [20]. Estimates of the heat capacities of the metal carbonyls were consistent with previous practice [8,17,19,21], in that a simple proportion based on relative molecular mass referred to pentacarbonyliron is used.

## 3. Results

### 3.1. Nonacarbonylcobaltmanganese

This compound has a relatively low melting pint (318–319 K) and the melt decomposes visibly above ca. 335 K. Maintaining the calorimeter at a constant temperature ($\pm 0.5$ K) below 350 K presents problems, so measurement of the enthalpy of sublimation [7] was difficult. However, as the result of much effort and many attempts which had to be rejected, examples of results in which we have confidence have been obtained (Table 1a).

Thermal decomposition of $CoMn(CO)_9$ was studied at various temperatures in the range 450–600 K. In the lower part of this range (450–500 K), the appearance of the calorimeter signal indicates that the decomposition is not straightforward. Eventually, we found that at 593 K the signal (thermogram) is simpler, decomposition is very rapid and a bright metal film is produced. Examples of the results obtained are shown in Table 1b. These lead to $\Delta_f H^\circ_m([CoMn(CO)_9]$, c) = $-(1451 \pm 28)$ kJ $mol^{-1}$.

Reaction of $CoMn(CO)_9$ with diiodine was quite simple. Titrimetric analysis for the consumption of diiodine makes clear that the reaction may be expressed as:

$$CoMn(CO)_9 + 2I_2 \rightarrow CoI_2 + MnI_2 + 9CO \quad (1)$$

The calorimetric results for iodination are shown in Table 2a. These lead to $\Delta_f H^\circ_m([CoMn(CO)_9]$, c) = $-(1478 \pm 28)$ kJ $mol^{-1}$. Calorimetric reasurements on the reaction with dibromine, which is assumed to be described by a similar expression, producing $CoBr_2$ and $MnBr_2$, are reported in Table 2b. These lead to $\Delta_f H^\circ_m([CoMn(CO)_9]$, c) = $-(1482 \pm 36)$ kJ $mol^{-1}$. Taking all the results together and having regard to the assumptions inplicit in the identification of products arising from the reactions with dihalogens, we choose a value of $\Delta_f H^0_m$ ($[CoMn(CO)_9]$, c) = $-(1460 \pm 36)$ kJ $mol^{-1}$ (Table 2).

Table 1
Sublimation and thermal decomposition of $CoMn(CO)_9$ (RMM 365.96)

| Mass/mg | $\Delta_{sub}H^{308}_m$/kJ $mol^{-1}$ | $\Delta_{sub}H^{298}_m$/kJ $mol^{-1}$ |
|---|---|---|
| A. Vacuum sublimation at 308 K | | |
| 1.960 | 86 | 73 |
| 1.480 | 83 | 70 |
| 1.379 | 85 | 72 |
| | | Mean $(72 \pm 2)$ |
| Mass/mg | Δh/J | $\Delta H^{593}_{obs}$/kJ $mol^{-1}$ |
| B. Thermal decomposition at 593 K | | |
| 2.232 | 3.214 | 527 |
| 3.344 | 4.961 | 543 |
| 2.054 | 2.941 | 524 |
| 2.536 | 3.714 | 536 |
| 2.990 | 4.404 | 539 |
| | | Mean $(534 \pm 8)$ |

Table 2
Iodination and bromination of $CoMn(CO)_9$

| Mass/mg | $I_2$/mg | $n$ | $\Delta H_{obs}$/kJ mol$^{-1}$ |
|---|---|---|---|
| A. Iodination at 493 K | | | |
| 1.715 | 7.768 | 2.02 | 89 |
| 1.795 | 9.404 | 1.96 | 86 |
| 1.814 | 10.236 | 1.94 | 81 |
| | | | Mean (85 ± 5) |

| Mass/mg | $Br_2$/mg | $\Delta H_{obs}$/kJ mol$^{-1}$ |
|---|---|---|
| B. Bromination at 473 K | | |
| 1.335 | 8.49 | −73 |
| 1.467 | 9.35 | −67 |
| 1.266 | 9.67 | −79 |
| | | Mean (−73 ± 8) |

### 3.1.1. Nonacarbonylcobaltrhenium

This compound melts at 339–340 K and the melt decomposes above 410 K. Vacuum sublimation from a calorimeter cell at 313 K gave the results listed in Table 3a. Thermal decomposition of $CoRe(CO)_9$ was studied at various temperatures in the range 473–600 K. Examples of the results obtained at 593 K are shown in Table 3b. These lead to $\Delta_f H^o_m([CoRe(CO)_9], c) = -(1450 \pm 20)$ kJ mol$^{-1}$ (Table 3).

The reaction with diiodine was quite simple. Titrimetric analysis for the consumption of diiodine shows that the reaction can be expressed as:

$$CoRe(CO)_9 + 2.5I_2 \rightarrow CoI_2 + ReI_3 + 9CO \quad (2)$$

The calorimetric results for iodination are shown in Table 4. These lead to $\Delta_f H^0_m$ ($[CoRe(CO)_9]$, c) = −(1464 ± 36) kJ mol$^{-1}$. We choose a value of $\Delta_f H^0_m([CoRe(CO)_9], c) = -(1452 \pm 36)$ kJ mol$^{-1}$ (Table 4).

Table 3
Sublimation and thermal decomposition of $CoRe(CO)_9$ (RMM 497.23)

| Mass/mg | $\Delta_{sub}H^{313}_m$/kJ mol$^{-1}$ | $\Delta_{sub}H^{298}_m$/kJ mol$^{-1}$ |
|---|---|---|
| A. Vacuum sublimation at 313 K | | |
| 1.008 | 100 | 89 |
| 1.427 | 92 | 81 |
| 1.687 | 90 | 80 |
| | | Mean (83 ± 4) |

| Mass/mg | Δh/J | $\Delta H^{593}_{obs}$/kJ mol$^{-1}$ |
|---|---|---|
| B. Thermal decomposition at 593 K | | |
| 1.243 | 1.385 | 554 |
| 1.253 | 1.372 | 544 |
| 1.254 | 1.371 | 544 |
| 1.289 | 1.406 | 542 |
| | | Mean (546 ± 4) |

Table 4
Iodination of $CoRe(CO)_9$ at 473 K

| Mass/mg | $I_2$/mg | $n$ | $\Delta H_{obs}$/kJ mol$^{-1}$ |
|---|---|---|---|
| 2.077 | 8.671 | 2.47 | 145 |
| 2.136 | 9.287 | 2.46 | 154 |
| 1.229 | 7.932 | 2.51 | 141 |
| | | | Mean (147 ± 9) |

### 3.2. Decacarbonylmanganeserhenium

In contrast to $CoMn(CO)_9$ and $CoRe(CO)_9$, the thermal stability of $MnRe(CO)_{10}$ is significant. This compound melts at 420–422 K and the melt decomposes above 450 K under argon. Thermal decomposition of $MnRe(CO)_{10}$ below 550 K was incomplete and poorly reproducible. It was necessary to make calorimetric measurements at 613 K to achieve reasonably clean metal films and acceptable reproducibility of the calorimetric measurements. Examples of the results are shown in Table 5, which includes measurements of the enthalpy of sublimation of $MnRe(CO)_{10}$.These lead to $\Delta_f H^0_m([MnRe(CO)_{10}], c) = -(1634 \pm 24)$ kJ mol$^{-1}$ (Table 5).

The heat of the reaction of $MnRe(CO)_{10}$ with diiodine was measured in the calorimeter in the temperature range 490–530 K. Titrimetric analysis for the consumption of the reagent shows that the reaction can be expressed as:

$$MnRe(CO)_{10} + 2.5\ I_2 \rightarrow MnI_2 + ReI_3 + 10CO \quad (3)$$

The calorimetric results for iodination are shown in Table 6a. These lead to $\Delta_f H^0_m([MnRe(CO)_{10}], c) = -(1589 \pm 24)$ kJ mol$^{-1}$. Reaction of $MnRe(CO)_{10}$ with dibromine was also studied in the calorimeter. It is

Table 5
Sublimation and thermal decomposition of $MnRe(CO)_{10}$ (RMM 521.24)

| Mass/mg | $\Delta_{sub}H^{363}_m$/kJ mol$^{-1}$ | $\Delta_{sub}H^{298}_m$/kJ mol$^{-1}$ |
|---|---|---|
| A. Vacuum sublimation at 363 K | | |
| 1.565 | 111 | 88 |
| 1.329 | 104 | 81 |
| 1.604 | 112 | 89 |
| | | Mean (86 ± 4) |

| Mass/mg | Δh/J | $\Delta H_{obs}$/kJ mol$^{-1}$ |
|---|---|---|
| B. Thermal decomposition at 613 K | | |
| 2.307 | 2.856 | 645 |
| 1.302 | 1.526 | 611 |
| 1.932 | 2.307 | 622 |
| 1.822 | 2.295 | 657 |
| 2.354 | 2.807622 | 622 |
| | | Mean (631 ± 17) |

Table 6
Iodination and bromination of $MnRe(CO)_{10}$

| Mass/mg | $I_2$/mg | $n$ | $\Delta H_{obs}$/kJ mol$^{-1}$ |
|---|---|---|---|
| A. Iodination at 513 K | | | |
| 1.746 | 9.399 | 2.45 | 133 |
| 1.524 | 8.052 | 2.53 | 124 |
| 1.730 | 8.981 | 2.48 | 129 |
| | | | Mean (129 ± 5) |
| Mass/mg | $Br_2$/mg | $\Delta H_{obs}$/kJ mol$^{-1}$ | |
| B. Bromination at 563 K | | | |
| 1.375 | 8.46 | 65 | |
| 1.730 | 8.74 | 66 | |
| 1.595 | 9.69 | 63 | |
| | | Mean (65 ± 2) | |

$n$ is the number of moles of $I_2$ reacting with $MnRe(CO)_{10}$.

assumed that the reaction is described by a similar expression, producing $MnBr_2$ and $ReBr_3$. The calorimetric results are shown in Table 6b. These lead to $\Delta_fH^0_m([MnRe(CO)_{10}], c) = -(1630 \pm 28)$ kJ mol$^{-1}$. We choose a value of $\Delta_fH^0_m([MnRe(CO)_{10}], c) = -(1630 \pm 24)$ kJ mol$^{-1}$ (Table 6).

## 4. Discussion

Studies using NMR of $CoMn(CO)_9$ in the solid state [22] provide strong support for a structure in which the cobalt–manganese bond is not bridged by a CO ligand; this is consistent with the IR spectra (2100–1800 cm$^{-1}$) of this compound [11,23] and of $CoRe(CO)_9$ in solution [11,23]. Low frequency IR spectra of $CoRe(CO)_9$ contain an absorption at 130 cm$^{-1}$ assigned to $\nu$(Co–Re) from which the force constant f(Co–Re) = 0.43 J m$^{-2}$ is derived [24]. We have made unsuccessful attempts to grow satisfactory single crystals of both $CoMn(CO)_9$ and $CoRe(CO)_9$: twinning was always a problem.

Mass spectrometric measurements on $Mn_2(CO)_{10}$, $Re_2(CO)_{10}$ and $MnRe(CO)_{10}$ have indicated [6] that the dissociation energy of the Mn–Re bond (210 kJ mol$^{-1}$) is greater than that of either of the homometallic bonds (Mn–Mn 94 kJ mol$^{-1}$; Re–Re 187 kJ mol$^{-1}$). Low frequency Raman spectra of the same three compounds [24,25] show absorptions assigned to metal–metal stretching vibrations. A simple normal coordinate analysis gave estimates of the force constants which agree in placing f(Mn–Re) < f(Re–Re) but differ in the ratio f(Mn–Re)/f(Re–Re) = 0.99 [25];0.73 [24]. The structures of all three compounds in the solid state show that the metal–metal bond length in $MnRe(CO)_{10}$ (2.909(1) Å [26]) is shorter than the average (2.972 Å) of the comparable bond length in $Mn_2(CO)_{10}$ (2.9038(6) Å) and $Re_2(CO)_{10}$ (3.0413(11) Å) [27]. The electronic spectra of these compounds in solution contain absorptions {$\lambda$/nm (log $\varepsilon$/m$^2$ mol$^{-1}$)} assigned to $\sigma \rightarrow \sigma^*$ transitions of the metal–metal bond: $Mn_2(CO)_{10}$ 342 (4.3304); $Re_2(CO)_{10}$ 313 (4.1903); $MnRe(CO)_{10}$ 323 (4.1367) [28]. These absorption energies show a satisfactory correlation with the activation enthalpies for the reactions of these metal carbonyls with dioxygen in decalin solution [29]. The equilibrium constant, K, for the comproportion reaction of $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$ at 190 K, K = 2.13 ± 0.02 [7] has been used to support the suggestion [30] that $Re_2(CO)_{10}$ possesses an additional stabilization as a result of metal–metal $\pi$-bonding more than is expected from simple extrapolation of the enthalpies of $Mn_2(CO)_{10}$ and $MnRe(CO)_{10}$. The enthalpies of reaction of the three metal carbonyls with diiodine to produce $M(CO)_5I$ in cyclohexane solution at 298 K have been measured by photocalorimetry [5]. The results indicate that the manganese–rhenium bond in $MnRe(CO)_{10}$ is weaker by about 63 kJ mol$^{-1}$ than the average of the homonuclear metal–metal bond energies. We note that the electron affinities of $Mn(CO)_5$ and $Re(CO)_5$ are presumed to be the same ((234 ± 20) kJ mol$^{-1}$) [31], so that the manganese–rhenium bond is not expected to have significant ionic character.

It is against this confusing background of information from a variety of sources involving a range of techniques for investigation that our results are presented in Table 7. The values of $\Delta_fH^0_m$, g can be used together with the auxiliary data in Section 2.2 for the enthalpy of formation of the $M(CO)_n$ fragments to provide values for the enthalpy contribution of the metal–metal bonds in these molecules. The results are shown in Table 8, together with the mean value of this enthalpy based on the homonuclear metal carbonyls and related information for the dissociation energy of the diatomic molecules [32]. In each case, the value of D(M–M′) is less than the mean value. Following the work of Fawcett and Poë [33], we had expected to find D(Mn–Re) ca. 160 kJ mol$^{-1}$, but instead our result is more in line with the conclusions of Harel and Adamson [5].

Table 7
Standard enthalpy of formation for solid metal carbonyls and enthalpy of vaporisation (all values in kJ mol$^{-1}$)

| | $\Delta_fH^0_m$, c | $\Delta_{sub}H^0$ | $\Delta_fH^0_m$, g | Ref. |
|---|---|---|---|---|
| $CoMn(CO)_9$ | −1460 ± 36 | 72 ± 2 | −1388 ± 42 | [a] |
| $CoRe(CO)_9$ | −1452 ± 36 | 83 ± 4 | −1369 ± 42 | [a] |
| $MnRe(CO)_{10}$ | −1630 ± 24 | 86 ± 4 | −1544 ± 28 | [a] |
| $Mn_2(CO)_{10}$ | −1677 ± 4 | 92 ± 2 | −1585 ± 5 | 19 |
| $Re_2(CO)_{10}$ | −1660 ± 11 | 101 ± 2 | −1559 ± 11 | 17 |
| $Co_2(CO)_8$ | −1250 ± 5 | 65 ± 3 | −1185 ± 6 | 21 |

[a] This work.

Table 8
Enthalpy contributions of metal–metal bonds

| | D(M–M′) (kJ mol$^{-1}$) | Mean (kJ mol$^{-1}$) | D(M–M′) (kJ mol$^{-1}$) [32] |
|---|---|---|---|
| $CoMn(CO)_9$ | $114 \pm 23$ | 123 | 123 |
| $CoRe(CO)_9$ | $122 \pm 25$ | 137 | 241 |
| $MnRe(CO)_{10}$ | $145 \pm 29$ | 173 | 242 |
| $Mn_2(CO)_{10}$ | $159 \pm 21$ | | 42 |
| $Re_2(CO)_{10}$ | $187 \pm 5$ | | 376 |
| $Co_2(CO)_8$ | $88 \pm 8$ | | 167 |

Mean refers to $0.5\{D(M-M)+D(M'-M')\}$.

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