

CHEMICAL IONIZATION MASS SPECTROMETRY OF $(\text{CO})_5\text{MnM}'\text{Ph}_3$ ($\text{M}' = \text{Si}, \text{Ge}, \text{Sn}$): A COMPARISON BETWEEN GASEOUS AND CONDENSED PHASE REACTIVITY

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Summary

For a comparison between gaseous and condensed phase reactivity $(\text{CO})_5\text{MnM}'\text{Ph}_3$ ($\text{M}' = \text{Si}, \text{Ge}, \text{Sn}$) have been investigated by chemical ionization mass spectrometry with CH_4 , $i\text{-C}_4\text{H}_{10}$, NH_3 and H_2 as reactant gases. The reactivity of the substrate towards the reactant gas increases on going from the silicon to the tin derivative and, for a given substrate methane is the most reactive gas. The two most important reactions of the protonated samples are those which lead to the ions $[\text{M}-\text{Ph}]^+$ and $[\text{M}'\text{Ph}_3]^+$, and are the same as those observed in the condensed phase; the change of the relative abundance of these ions upon variation of the probe temperature is discussed.

Introduction

Compounds with a direct bond between a transition metal (M) and a Group IVB element (M') have received much attention. A feature of these compounds is the presence of different reactive sites since their reactions may in principle involve the M-M' bond, the bonds between M and the other ligands, or those between M' and the carbon atoms which are usually present. Special attention has been devoted to the study of the reactions with electrophilic agents which can lead to M-M' or M'-C bond breaking. For compounds such as $(\text{CO})_5\text{MnM}'\text{R}_3$ (R = alkyl or aryl) it has been found that (a) the Si-C bond is never broken by halogens, which cause Si-Mn bond rupture [1]; (b) the Sn-Mn bond is more resistant than the Sn-C bond to the attack of halogen [1,2]; (c) electrophilic attack by halogen acids always gives M'-C bond breaking [2,3,4]; (d) attack of iodine, which breaks the M'-Mn bond, obeys a simple overall second order kinetic law [5,6]. In spite of these investigations no satisfactory rationalization is available for the chemical behaviour of these

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compounds. It seemed likely that a study of their reactivity towards protonating agents in the gaseous phase might give some additional information on their reactions, and so we undertook a chemical ionization (CI) mass spectrometry study of the title compounds, which were previously studied by electron impact (EI) mass spectrometry [7,8,9].

Results and discussion

General features

The EI mass spectra of these manganese complexes are of limited interest because structurally and analytically significant ions of the type $[(\text{CO})_x\text{MnM}'\text{Ph}_3]^+$ have a very low relative abundance and are often obscured in the background spectrum and the ion $[\text{MnM}'\text{Ph}_3]^+$ has the highest m/z ratio. The absence of the molecular ion is a peculiarity of the aryl derivatives, the trimethyl derivatives exhibiting fairly intense molecular ions [10]. This striking difference in the mass spectra of rather similar compounds had not previously received attention. It cannot be related to a difference in the Mn–CO or Mn–M' bond dissociation energies, but it can be qualitatively understood in terms of the quasi-equilibrium theory [11] of mass spectra; the ionization energy of the radicals $\text{M}'\text{Ph}_3$ and $\text{M}'\text{Me}_3$ has a direct influence on the activation energy for the formation of the ions $[\text{M}'\text{R}_3]^+$, which decreases as the radical ionization energy becomes lower. Since the ionization energies of the triphenyl are lower than those of the trimethyl radicals (for tin the two values are 606 and 656 kJ/mole, respectively [12]), the activation energy for the reaction leading to generation of the $[\text{M}'\text{Ph}_3]^+$ ion from the molecular ion can be substantially below that for loss of CO; the molecular ion will preferentially fragment along the lower energy path and so its relative abundance becomes smaller.

In CI mass spectrometry the signal corresponding to the protonated compound remains very low, but in all the spectra there is an ion corresponding to the loss of a phenyl group from the original molecule, which therefore retains its CO groups. The mass spectra obtained with CH_4 , $i\text{-C}_4\text{H}_{10}$, or H_2 are strongly influenced by the sample temperature in the evaporation probe; increase in this temperature causes an increase in the relative intensity of the $[\text{M}-\text{Ph}]^+$ ion; the reversibility of this effect confirms that it is not due to decomposition of the sample in the solid phase.

A complicating feature of the mass spectra obtained either with methane or *i*-butane is the participation, along with the proton transfer reaction, of other reactions that are not commonly found in other carbonylic complexes. A comparison of the mass spectral data listed in Tables 1 to 9 shows that the reactivity of the substrates towards the reactant gas increases on going from the silicon to the tin derivative and that, for a given substrate, the methane system is the most reactive; accordingly, the mass spectrum of $(\text{CO})_5\text{MnSnPh}_3$ is the richest in ions, most of them containing tin bonded to phenyl, methyl, or ethyl groups (similar observations have been made in this laboratory with compounds of the type $\text{CpFe}(\text{CO})_2\text{M}'\text{Ph}_3$). These reactions are probably the result of successive collisions of the $[\text{M}'\text{Ph}_3]^+$ ions with neutral radicals formed in the reactant gases; the different reactivities of these ions are probably related to differences in the M'–Me and M'–Ph bond dissociation energies; in this connection, and in absence of adequate data, we note only that the

TABLE 1
 CI MASS SPECTRA OF $(\text{CO})_5\text{MnSnPh}_3$ (Reagent gas: hydrogen)

m/e	Intensity				Assignment
	85°C	105°C	125°C	145°C	
469	19	34	51	100	$[\text{M} - \text{Ph}]^+$
413	5	8	8	8	$[\text{M} - (\text{Ph} + \text{CO})]^+$
406	21	22	19	16	$[\text{Ph}_3\text{SnMn}]^+$
368	3	5	4	3	—
351	100	100	100	90	$[\text{Ph}_3\text{Sn}]^+$
329	3	3	3	3	$[\text{Ph}_2\text{SnMn}]^+$
274	4	4	3	4	$[\text{Ph}_2\text{Sn}]^+$
252	8	8	6	5	$[\text{PhSnMn}]^+$
197	70	70	78	80	$[\text{PhSn}]^+$
186	—	2	2	1	$[\text{MnPh}_3]^+$
120	31	34	34	40	Sn^+

$\text{M}'\text{-C}$ bond dissociation energy increases from silicon to tin in the tetramethyl ions [13], while in the corresponding tetraphenyl ions the bond dissociation energy is almost constant [12,14].

Chemical ionization mass spectra

(1) $(\text{CO})_5\text{MnSnPh}_3$. The mass spectra become progressively simpler as the reactant gas is changed from methane to isobutane to hydrogen (Tables 1–3); with the last gas all the ions due to the exchange reactions discussed above are absent; however, with H_2 an ion at $m/z = 406$, assigned to $[\text{Ph}_3\text{SnMn}]^+$, shows up. This ion is also present in the EI spectrum, so that its presence is indicative of a charge exchange reaction between H_3^+ and the neutral substrate molecule; such a reaction is consistent with the value (765 kJ/mole) of the ionization energy reported [7] for this compound; its mass spectrum is therefore to be regarded as the superimposition of the spectrum obtained via the protonation and that from the charge exchange reaction.

It is noteworthy that the $[\text{Ph}_3\text{Sn}]^+$ ion, which represents the base peak in the EI spectrum, is also formed after the protonation reaction, as indicated by its presence

TABLE 2
 CI MASS SPECTRA OF $(\text{CO})_5\text{MnSnPh}_3$ (Reagent gas: isobutane)

m/e	Intensity			Assignment
	85°C	105°C	120°C	
469	8	51	100	$[\text{M} - \text{Ph}]^+$
407	—	—	5	$[\text{Ph}_3\text{SnMnH}]^+$
351	9	34	43	$[\text{Ph}_3\text{Sn}]^+$
289	19	55	50	$[\text{Ph}_2\text{MeSn}]^+$
227	22	57	53	$[\text{PhMe}_2\text{Sn}]^+$
183	18	74	35	—
165	100	100	43	$[\text{Me}_3\text{Sn}]^+$
155	13	42	35	$[\text{Ph}_2\text{H}]^+$
151	20	18	6	$[\text{Me}_2\text{SnH}]^+$
121	28	64	48	$[\text{SnH}]^+$

TABLE 3

CI MASS SPECTRA OF $(\text{CO})_5\text{MnSnPh}_3$ (Reagent gas: methane)

<i>m/e</i>	Intensity		Assignment
	70°C	100°C	
469	26	56	$[\text{M} - \text{Ph}]^+$
401	8	—	$[\text{Ph}_2\text{MeSnMn}(\text{CO})_2\text{H}]^+$
351	17	21	$[\text{Ph}_3\text{Sn}]^+$
339	5	—	$[\text{Ph}_2\text{SnMn}]^+$
323	4	—	$[\text{Mn}_2(\text{CO})_6\text{Me}_3]^+$
303	—	10	$[\text{Ph}_2\text{EtSn}]^+$
289	13	72	$[\text{Ph}_2\text{MeSn}]^+$
277	17	—	$[\text{Et}_3\text{SnMnMe}]^+$
241	—	23	$[\text{PhSnC}_3\text{H}_9]^+$
227	15	100	$[\text{PhMe}_2\text{Sn}]^+$
213	—	12	$[\text{PhSnCH}_4]^+$
165	41	91	$[\text{Me}_3\text{Sn}]^+$
157	93	51	$[\text{MnEt}_3\text{Me}]^+$
143	100	47	$[\text{Mn}(\text{CO})\text{Me}_4]^+$

in the isobutane chemical ionization, in which the low recombination energy of the reactant gas [15] precludes the charge exchange reaction.

(2) $(\text{CO})_5\text{MnGePh}_3$. These mass spectra are much simpler than those of the tin derivative, because the phenyl-methyl exchange reactions play a smaller part, thus the spectra obtained with the three different gases are similar (Tables 4–6); the charge exchange reaction is evident with hydrogen.

(3) $(\text{CO})_5\text{MnSiPh}_3$. A peculiar feature in the mass spectra of this compound is the presence of ions obtained by loss of some CO groups from the protonated molecule; as shown in Tables 7–9, these ions are observed only with methane and isobutane, indicating that only when a small amount of energy is transferred in the ionization process are ions with compositions close to that of the molecular ion observed in significant abundance. In the EI spectra the peaks for these ions are very weak [9], since a larger amount of energy is transferred to the molecular ion.

(4) NH_3 as reactant gas. In these mass spectra, shown in Tables 10–12, there is no evidence for the protonation reaction, indicating that the proton affinity of all the compounds is between 786 and 857 kJ/mole [15].

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TABLE 4

CI MASS SPECTRA OF $(\text{CO})_5\text{MnGePh}_3$ (Reagent gas: hydrogen)

<i>m/e</i>	Intensity				Assignment
	75°C	100°C	120°C	140°C	
423	38	44	46	49	$[\text{M} - \text{Ph}]^+$
395	13	11	9	8	$[\text{M} - (\text{Ph} + \text{CO})]^+$
367	—	3	3	3	$[\text{M} - (\text{Ph} + 2\text{CO})]^+$
360	—	5	3	3	$[\text{Ph}_3\text{GeMn}]^+$
347	—	5	3	3	—
305	100	100	100	100	$[\text{Ph}_3\text{Ge}]^+$
228	39	24	17	14	$[\text{Ph}_2\text{Ge}]^+$

TABLE 5
CI MASS SPECTRA OF $(\text{CO})_5\text{MnGePh}_3$ (Reagent gas: isobutane)

m/e	Intensity				Assignment
	75°C	90°C	120°C	140°C	
423	43	49	84	100	$[M - \text{Ph}]^+$
361	5	4	5	4	$[\text{Ph}_3\text{GeMnH}]^+$
305	100	100	100	40	$[\text{Ph}_3\text{Ge}]^+$
243	42	43	43	12	$[\text{Ph}_2\text{MeGe}]^+$
229	15	15	10	3	$[\text{Ph}_2\text{GeH}]^+$

TABLE 6
CI MASS SPECTRA OF $(\text{CO})_5\text{MnGePh}_3$ (Reagent gas: methane)

m/e	Intensity				Assignment
	40°C	75°C	115°C	145°C	
423	21	18	100	100	$[M - \text{Ph}]^+$
395	—	—	7	3	$[M - (\text{Ph} + \text{CO})]^+$
361	—	—	6	3	$[\text{Ph}_3\text{GeMnH}]^+$
347	—	—	6	—	—
305	34	21	59	15	$[\text{Ph}_3\text{Ge}]^+$
243	33	25	32	2	$[\text{Ph}_2\text{MeGe}]^+$
229	100	100	47	2	$[\text{Ph}_2\text{GeH}]^+$

TABLE 7
CI MASS SPECTRA OF $(\text{CO})_5\text{MnSiPh}_3$ (Reagent gas: hydrogen)

m/e	Intensity				Assignment
	35°C	55°C	85°C	110°C	
377	2	2	70	15	$[M - \text{Ph}]^+$
277	4	4	19	—	—
259	45	50	96	100	$[\text{Ph}_3\text{Si}]^+$
201	7	6	9	—	—
199	4	5	25	—	—
183	100	100	100	27	$[\text{Ph}_2\text{SiH}]^+$
182	26	27	33	61	$[\text{Ph}_2\text{Si}]^+$
181	12	12	28	23	$[\text{C}_{12}\text{H}_9\text{Si}]^+$
123	9	9	—	—	—
105	8	7	21	32	$[\text{PhSi}]^+$

TABLE 8
CI MASS SPECTRA OF $(\text{CO})_5\text{MnSiPh}_3$ (Reagent gas: isobutane)

m/e	Intensity		Assignment
	20°C	100°C	
399	2	2	$[\text{MH} - 2\text{CO}]^+$
377	28	95	$[M - \text{Ph}]^+$
370	2	6	$[M - 3\text{CO}]^+$
349	4	5	$[M - (\text{Ph} + \text{CO})]^+$
259	100	100	$[\text{Ph}_3\text{Si}]^+$
197	11	4	—
183	70	11	$[\text{Ph}_2\text{SiH}]^+$
182	20	5	$[\text{Ph}_2\text{Si}]^+$
181	6	5	$[\text{C}_{12}\text{H}_9\text{Si}]^+$
105	15	6	$[\text{PhSi}]^+$

TABLE 9

CI MASS SPECTRA OF $(\text{CO})_5\text{MnSiPh}_3$ (Reagent gas: methane)

m/e	Intensity			Assignment
	40°C	50°C	70°C	
427	—	—	4	$[\text{MH} - \text{CO}]^+$
399	—	—	4	$[\text{MH} - 2\text{CO}]^+$
377	45	40	100	$[\text{M} - \text{Ph}]^+$
349	—	—	4	$[\text{M} - (\text{Ph} + \text{CO})]^+$
277	26	16	13	—
259	100	100	93	$[\text{Ph}_3\text{Si}]^+$
199	14	—	6	—
197	12	15	4	$[\text{Ph}_2\text{MeSi}]^+$
183	61	49	36	$[\text{Ph}_2\text{SiH}]^+$

TABLE 10

CI MASS SPECTRA OF $(\text{CO})_5\text{MnSnPh}_3$ (Reagent gas: ammonia)

m/e	Intensity	Assignment
564	1	$[\text{M} + \text{NH}_4]^+$
536	10	$[(\text{M} + \text{NH}_4) - \text{CO}]^+$
513	2	—
496	2	—
486	100	$[\text{Ph}_2\text{SnMn}(\text{CO})_5\text{NH}_3]^+$
459	5	$[\text{Ph}_2\text{SnMn}(\text{CO})_4\text{NH}_4]^+$
435	8	—
418	6	—
385	3	$[\text{Ph}_3\text{Sn}(\text{NH}_3)_2]^+$
368	10	$[\text{Ph}_3\text{SnNH}_3]^+$
309	12	$[\text{Ph}_2\text{Sn}(\text{NH}_3)_2\text{H}]^+$
292	5	$[\text{Ph}_2\text{SnNH}_4]^+$

TABLE 11

CI MASS SPECTRA OF $(\text{CO})_5\text{MnGePh}_3$ (Reagent gas: ammonia)

m/e	Intensity	Assignment
535	6	$[\text{M} + 2\text{NH}_3 + \text{H}]^+$
518	100	$[\text{M} + \text{NH}_4]^+$
450	6	—
440	35	$[\text{Ph}_2\text{GeMn}(\text{CO})_5\text{NH}_3]^+$
400	10	—
349	8	—
341	23	—

TABLE 12

CI MASS SPECTRA OF $(\text{CO})_5\text{MnSiPh}_3$ (Reagent gas: ammonia)

m/e	Intensity	Assignment
472	100	$[\text{M} + \text{NH}_4]^+$
394	23	$[(\text{M} + \text{NH}_3) - \text{Ph}]^+$
277	15	$[\text{Ph}_3\text{SiNH}_4]^+$
276	78	$[\text{Ph}_3\text{SiNH}_3]^+$
200	28	$[\text{Ph}_2\text{SiNH}_4]^+$

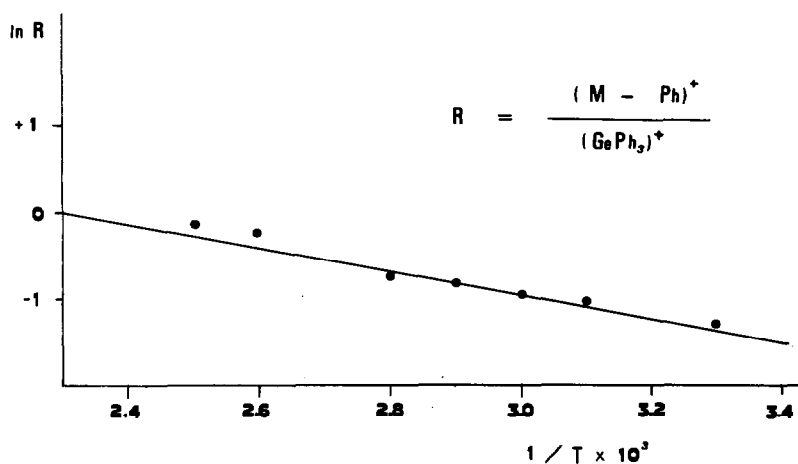


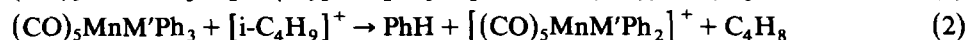
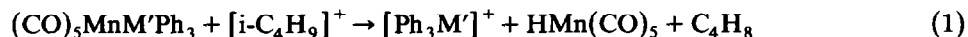
Fig. 1. Effect of change of temperature on the ion abundance ratio $[M-Ph]^+ / [GePh_3]^+$ for $(CO)_5MnGePh_3$ obtained with $i-C_4H_{10}$ as reagent gas.

The observed ions are formed through electrophilic attack of $[NH_4]^+$ on the substrate [16], with formation of ions of the type $[M + NH_4]^+$; the most abundant ions of the mass spectra retain the NH_4 or the NH_3 group.

Temperature effect

As mentioned above, an increase in the sample temperature in the probe results in an increase in the relative abundance of the $[M-Ph]^+$ ion (some of the data are listed in Tables 1–9). This reversible effect was studied between about 40 and 140°C; the temperature range was different for each compound, and the effective range was limited at the lower end by the sample volatility and, at the upper end, by the participation of bimolecular reactions involving the substrate, which show up when the sample partial pressure in the ion source is raised [17].

The two most important reactions of the protonated samples are probably those leading to the ions $[M-Ph]^+$ and $[M'Ph_3]^+$; these two reactions correspond to those observed in the condensed phase. Since only fast reactions can be observed in CI mass spectrometry, it follows that both reactions must be thermodynamically favoured. Two reactions which meet this condition are:



The estimated ΔH 's for these reactions are negative for the tin derivative (see Appendix; note that use of the other reactant gases makes ΔH more negative for both reactions). If this is the case, then the ratio of the relative abundances of the ions $[Ph_3M']^+$ and $[(CO)_5MnM'Ph_2]^+$ is proportional to the concentration of the products of two parallel reactions after an appropriate interval from the collision with the reagent gas*. Under these conditions the ratio of the two ion abundances is equal, at each temperature, to the ratio of the rate constants for the two reactions; a semi-logarithmic plot of this ratio vs. $1/T$ should give a straight line, with a slope

* This time interval, which corresponds to the residence time in the ion source [18], could be evaluated, but its value is not relevant for the subsequent discussion.

from which the difference between the activation energies of the two reactions can be calculated.

The data obtained for $(\text{CO})_5\text{MnGePh}_3$ ionized by $[\text{i-C}_4\text{H}_9]^+$ are in agreement with this prediction (see Fig. 1). The same holds for the tin derivative although in this case data are available only at three temperatures. From the plot of Fig. 1 the difference in the activation energies of the two reactions is estimated as 12 kJ/mole, that for reaction 2 being the greater. Although these data are of limited accuracy, they indicate that the activation energies of the two reactions are close.

Conclusions

The results should be of significance for related reactions in the condensed phase. They show, for example, that the two reactive sites of these compounds have about the same reactivity after protonation. It is probable that the small difference in activation energy observed in the gas phase also applies in the solution reactions of these complexes, and so even small variations in the underlying electronic factors may favour one or the other reaction if the electrophilic agent does not have any definite charge separation.

The reactions observed in chemical ionization are promoted by hydrogen ion attack; in this respect, they should be more like the reactions of the three complexes with acids, which are known to lead only to the $\text{M}'\text{-C}$ bond breaking [2-4].

There is, however, an important difference between the chemical ionization and condensed phase reactions; in the latter (which were examined in poor ionizing solvents and for which a four centre transition state has been postulated [4]) the reactions are promoted by a molecule whose nucleophilic end plays a role in determining the nature of the final products. In chemical ionization the reactions begin with the protonation of the molecule, probably at the Mn or M' atom, followed by two parallel reactions which involve a hydrogen atom transfer.

Experimental

The manganese complexes were prepared by published procedures [19]. Mass spectra were obtained with a Finnigan-Mat 112S mass spectrometer equipped with a chemical ionization ion source.

The reactant gases were reagent grade products and their pressure in the ionization box was kept between 0.05 and 0.1 torr; significant variations of the mass spectra were not observed over this pressure range. The ion source temperature was 170°C.

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Appendix

(1) Estimation of ΔH for reaction 1: $\text{M}' = \text{Sn}$; reactant gas = isobutane.

Data: $\Delta H_{\text{F}}^{\circ} \text{Ph}_3\text{Sn} + 518 \text{ kJ/mole}$ [12]

$\Delta H_{\text{F}}^{\circ} \text{Mn}(\text{CO})_5 - 767 \text{ kJ/mole}$ [20]

$D(\text{Mn-Sn}) + 201 \text{ kJ/mole}$ [7]

$\Delta H_{\text{F}}^{\circ}[(\text{CO})_5\text{MnSnPh}_3] = 518 - 767 - 201 = -450 \text{ kJ/mole}$

Data: ΔH_F° [i-C₄H₈]⁺ + 830 kJ/mole [21]
 ΔH_F° [Ph₃Sn]⁺ + 1099 kJ/mole [12]
 $\Delta^\circ H_F$ HMn(CO)₅ - 991 kJ/mole [22]
 $\Delta^\circ H_F$ C₄H₈ - 17 kJ/mole [23]
 ΔH° (reaction 1) = -991 + 1099 - 17 + 450 - 830 = -289 kJ/mole

(2) Estimation of ΔH for reaction 2: $M' = Sn$; reactant gas = isobutane

Data: IP Ph₃SnMn(CO)₅ + 765 kJ/mole [7]
 ΔH_F° [Ph₃SnMn(CO)₅]⁺ = 765 - 450 = +315 kJ/mole

Data: IP Ph₄Sn + 804 kJ/mole [14]
 AP [Ph₃Sn]⁺ + 925 kJ/mole [12]
 D(Ph-Sn) + 121 kJ/mole

Data: ΔH_F° C₆H₅ + 301 kJ/mole (24)
 ΔH_F° [Ph₃SnMn(CO)₅]⁺ = 315 - 301 + 121 = +135 kJ/mole

Data: ΔH_F° C₆H₆ + 83 kJ/mole [21]
 ΔH° (reaction 2) = 83 + 135 - 17 - 830 + 450 = -179 kJ/mole

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