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Chlorination of charged buckyballs: reactions of C_{60}^{x+} cations ($x = 1$–3) with Cl_2, CCl_4, $CDCl_3$, CH_2Cl_2, and CH_3Cl

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Abstract

The chlorination of singly and multiply charged C_{60} cations has been investigated with the selected-ion flow tube technique. Observations are reported for the reactions of $C_{60}^{\cdot+}$, C_{60}^{2+} and $C_{60}^{\cdot 3+}$ with Cl_2, CCl_4, $CDCl_3$, CH_2Cl_2 and CH_3Cl at room temperature (295 ± 2 K) in helium at a total pressure of 0.35 ± 0.02 Torr. $C_{60}^{\cdot+}$ and C_{60}^{2+} were observed not to chlorinate, or react in any other way, with these five molecules. Chlorine also did not react with $C_{60}^{\cdot 3+}$, but bimolecular chloride transfer and electron transfer reactions, reactions that result in charge reduction/charge separation, were observed to occur with CCl_4, $CDCl_3$, CH_2Cl_2 and CH_3Cl. Chloride transfer was the predominant channel seen with CCl_4, $CDCl_3$ and CH_2Cl_2 while electron transfer dominates the reaction with CH_3Cl. These results are consistent with trends in chloride affinity and ionization energy. The reluctant chlorination of the first two charge states of C_{60} is attributed to the energy required to distort the carbon cage upon bond formation, while the observed chloride transfer to $C_{60}^{\cdot 3+}$ is attributed to the greater electrostatic interactions with this ion. (Int J Mass Spectrom 182/183 (1999) 349–356) © 1999 Elsevier Science B.V.

Keywords: Fullerene ions; Chlorine; Chlorinated methanes; Chloride transfer; Kinetics; Charge separation; Selected-ion flow tube

1. Introduction

Ben Freiser had an early, strong interest in the formation of exohedral atomic adduct ions of C_{60}. For example, he was the first to demonstrate the laboratory formation of a variety of externally-bound transition-metal species MC_{60}^{+} (M = V, VO, Fe, Co, Ni, Cu, Rh, La) [1,2]. The adduct ions were reported to be formed by direct attachment of M^+ ions generated by laser desorption to C_{60} molecules at the low pressure ($\sim 1 \times 10^{-7}$ Torr) of a Fourier-transform mass spectrometer. He then went on to investigate the formation of other organometallic derivatives of $C_{60}^{\cdot+}$ [3,4]. Our own experimental survey of the chemistry of fullerene cations showed that the second and third charge states of C_{60} also may be derivatized at room temperature in helium buffer gas at ~0.35 Torr in a selected-ion flow tube (SIFT) apparatus. But we noticed that the occurrence of derivatization was very sensitive to the charge state of C_{60}^{x+} [5]. Briefly, we found that monocations of C_{60} under SIFT conditions bond only with strong nucleophiles such as ammonia and satu-

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Dedicated to the memory of Ben Freiser, in part for his outstanding contributions to fullerene ion chemistry.

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rated amines [6] and with molecules capable of Diels-Alder additions [7]. A reaction of $C_{60}^{\cdot +}$ with iron pentacarbonyl, first observed by Freiser et al. [8], produces derivatized $C_{60}Fe(CO)_4^{\cdot +}$ [9] and is the only example of a *bimolecular* derivatization reaction with $C_{60}^{\cdot +}$ that we have observed to date. Covalent bonding to $C_{60}^{\cdot +}$ is difficult because it is necessary to distort the C_{60} carbon cage at the C site of bond formation with the substituent so as to achieve the required sp^3 hybridization. This energy barrier is more readily overcome with the higher charge states of C_{60} as the electrostatic interaction between the reactants increases [10]. For example, we have previously found C_{60}^{2+} to be very reactive toward many molecules and to exhibit a rich addition chemistry, although electron transfer becomes an important competitive reaction channel. With $C_{60}^{\cdot 3+}$, also a very reactive cation, this competition often favours electron transfer or even dissociative electron transfer, but addition is still observed [11].

Here we explore the ease of chlorination of charged buckyballs as a function of charge state in *bimolecular* reactions with chlorine gas and chlorinated methanes. We have previously reported one example of a chlorination study [12]. HCl was observed not to react with $C_{60}^{\cdot +}$ or C_{60}^{2+}, $k < 5 \times 10^{-13}$ cm^3 $molecule^{-1}$ s^{-1}, but increasing the charge to +3 resulted in a moderately fast reaction, $k = 3.7 \times 10^{-11}$ cm^3 $molecule^{-1}$ s^{-1}, leading to the two interesting charge-separation channels (1a) and (1b). (88 ± 5)% of the reactive collisions led to chlorination

$$C_{60}^{\cdot 3+} + HCl \rightarrow C_{60}Cl^{\cdot 2+} + H^+ \quad (1a)$$

$$\rightarrow C_{60}H^{\cdot 2+} + Cl^+ \quad (1b)$$

remarkably accompanied by formation of a proton, channel (1a), while (12 ± 5)% resulted in hydrogenation accompanied by Cl^+ formation. Both channels (1a) and (1b) involve charge separation in the products and so are constrained by energy barriers arising from coulombic repulsion [10]. No further chlorination of $C_{60}Cl^{\cdot 2+}$ was observed.

It is interesting to note that neutral C_{60} can be chlorinated only with some difficulty. Olah et al. [13] have reported that treatment of C_{60} with chlorine in chloro-organic solvents at various temperatures did not result in any detectable reaction. However, C_{60} could be chlorinated with up to 24 chlorine atoms on average at 250 °C when exposed to a slow stream of chlorine gas for several hours. At higher temperature the chlorination occurred more rapidly but complete chlorination could not be achieved. Also, the polychlorofullerenes were observed to be relatively unstable: they were observed to completely dechlorinate at 400 °C under argon and under mild ionization conditions in fast-atom bombardment (FAB) and field-ionization mass spectrometry (FIMS) experiments.

2. Experimental

The ion/molecule reactions of the fullerene cations C_{60}^{x+} ($x = 1, 2, 3$) with the selected chlorinated molecules were achieved within the reaction region of a SIFT mass spectrometer in helium carrier gas at 0.35 ± 0.01 Torr and 294 ± 3 K [14,15]. The fullerene cations were produced in an ion source by electron impact ionization of the vapour of a sample of fullerene powder (~99.5 + % C_{60}, SES Research): $C_{60}^{\cdot +}$ at ~50 eV, C_{60}^{2+} at ~80 eV and $C_{60}^{\cdot 3+}$ at ~100 eV. The desired C_{60} cation was selected with a quadrupole mass filter, injected into the flow tube containing He, and then allowed to thermalize by collisions with He (~4×10^5 collisions) prior to entering the reaction region further downstream.

Chlorine and methyl chloride were obtained from Matheson (99.9% and 99.5%, respectively). Deuterated chloroform was obtained from Sigma (99.8 atom % D). Methylene chloride and chloroform were obtained from BDH (99.9%). The vapours of the liquids were added into the flow tube as 10% (CCl_4), 3–5% ($CDCl_3$) and 25–28% (CH_2Cl_2) mixtures in helium.

Rate coefficients were measured with pure helium buffer gas at a total pressure of 0.35 ± 0.02 Torr in the usual manner [14,15] and have an uncertainty estimated to be less than ±30%.

Table 1
Effective bimolecular rate coefficients (in units of 10^{-9} cm^3 molecule^{-1} s^{-1}) and product channels measured for reactions of C_{60}^{x+} cations with chlorine and chlorinated methanes in helium buffer gas at 0.35 ± 0.01 Torr and 294 ± 3 K using the SIFT technique

Reactant ion	Product ion	Branching ratio [a]	k_{obs} [b]
Cl_2			
$C_{60}^{\cdot+}$	Not observed		<0.0001
C_{60}^{2+}	Not observed		<0.0001
$C_{60}^{\cdot3+}$	Not observed		<0.0001
CCl_4			
$C_{60}^{\cdot+}$	Not observed		<0.0001
C_{60}^{2+}	Not observed		<0.0003
$C_{60}^{\cdot3+}$	$C_{60}Cl^{\cdot2+} + CCl_3^+$		1.6
$CDCl_3$			
$C_{60}^{\cdot+}$	Not observed		<0.0001
C_{60}^{2+}	Not observed		<0.0001
$C_{60}^{\cdot3+}$	$C_{60}Cl^{\cdot2+} + CDCl_2^+$		0.85
CH_2Cl_2			
$C_{60}^{\cdot+}$	Not observed		<0.0001
C_{60}^{2+}	Not observed		<0.0003
$C_{60}^{\cdot3+}$	$C_{60}Cl^{\cdot2+} + CH_2Cl^+$	≥0.91	0.035
	$C_{60}^{2+} + CH_2Cl^+ + Cl^{\cdot}$	≤0.09	
CH_3Cl			
$C_{60}^{\cdot+}$	Not observed		<0.0003
C_{60}^{2+}	Not observed		<0.0003
$C_{60}^{\cdot3+}$	$C_{60}^{2+} + CH_3Cl^{\cdot+}$	0.55	0.022
	$C_{60}Cl^{\cdot2+} + CH_3^+$	0.34	
	$C_{60}CH_3^{\cdot2+} + Cl^+$	0.11	

[a] Uncertainties in the branching ratios are less than 20%.

[b] The absolute uncertainties of the observed bimolecular rate coefficients are estimated to be ±30%.

3. Results and discussion

The kinetic results obtained for the reactions investigated in this study are summarized in Table 1. Helpful thermochemical data [16,17] is summarized in Table 2.

3.1. Reactions with $C_{60}^{\cdot+}$

$C_{60}^{\cdot+}$ was observed not to react with any of the five molecules investigated, $k < 1 \times 10^{-13}$ cm^3 molecule^{-1} s^{-1}. Electron transfer is endothermic in all cases because of the low electron-recombination energy of $C_{60}^{\cdot+}$, RE($C_{60}^{\cdot+}$) = 7.64 ± 0.02 eV [18]. The ionization energies of the five molecules investigated all exceed 11.2 eV. There was no evidence for Cl^- transfer or $Cl^{\cdot}$ abstraction. The relevant $Cl^{\cdot}$ affinities vary from 58 kcal mol^{-1} (for $Cl^{\cdot}$) to 84 kcal mol^{-1} (for CH_3) and the Cl^- affinities vary from 173 kcal mol^{-1} (for CCl_3^+) to 274 kcal mol^{-1} (for Cl^+). The $Cl^{\cdot}$ and Cl^- affinities of $C_{60}^{\cdot+}$ are not known. The failure to observe reactions cannot be used to determine limiting values for the $Cl^{\cdot}$ and Cl^- affinities of $C_{60}^{\cdot+}$ since these reactions are kinetically rather than thermodynamically controlled. A barrier is expected to be associated with C–Cl bond formation with $C_{60}^{\cdot+}$ as a consequence of the rehybridization from largely sp^2 to largely sp^3 required at the C atom on the C_{60} surface that becomes bonded [10]. Hydride transfer also was not observed. The occurrence of H-atom transfer was difficult to rule out experimentally due to insufficient resolution of the detection quadrupole. However, available H-atom affinities [19] (see Table 2) indicate that H-atom abstraction is endothermic by at least 29 kcal mol^{-1}.

3.2. Reactions with C_{60}^{2+}

C_{60}^{2+} was also observed not to react with any of the five molecules investigated. The electron-recombination energy of C_{60}^{2+}, RE(C_{60}^{2+}) = 11.36 ± 0.05 eV [11] is comparable to the ionization energies of these molecules which span a range from 11.26 ± 0.03 eV for CH_3Cl to 11.48 ± 0.01 eV for Cl_2 (see Table 2). However, electron transfer is not expected to occur due to the barrier arising from coulomb repulsion in the resulting charge separation [10]. Our previous measurements have demonstrated an onset for electron transfer with molecules having ionization energy (IE) ≤9.51 eV [11]. There was again no evidence for Cl^- transfer or $Cl^{\cdot}$ abstraction and, again, the Cl^- and $Cl^{\cdot}$ affinities of C_{60}^{2+} are not known. The kinetic control for Cl^- transfer reactions with C_{60}^{2+} will include the barrier arising from coulombic repulsion in the resulting charge separation [10]. Hydride transfer again was not observed and available H-atom affinities [19] (see Table 2) indicate that H-atom abstraction in this case is endothermic by at least 12 ± 10 kcal mol^{-1}.

Table 2
Helpful thermochemical data used to calculate enthalpies of reaction. Ionization energies are in eV. Atom and anion affinities are in kcal mol^{-1}. The data were taken from References 17 and 18, unless indicated otherwise by references in brackets. Unless indicated otherwise, uncertainties are ~ ± 1 kcal mol^{-1}. Numbers in parentheses have a higher uncertainty.

A	IE	$H^{\cdot}A$	$Cl^{\cdot}A$	$H^{-}A$	$Cl^{-}A$
$Cl^{\cdot}$			58		
Cl_2	11.48 ± 0.01				
Cl^+					273.6
$CH_3^{\cdot}$			84		
CH_3^+					226
$CH_2Cl^{\cdot}$		103	83		
CH_3Cl	11.26 ± 0.03				
CH_2Cl^+				(283)	(197)
$CHCl_2^{\cdot}$		101	80		
CH_2Cl_2	11.33 ± 0.04				
$CHCl_2^+$				(268)	(181)
$CCl_3^{\cdot}$		96	71		
$CHCl_3$	11.37 ± 0.02				
CCl_3^+				(264)	(173)
CCl_4	11.47 ± 0.01				
C_{60}	7.64 ± 0.02 [18]				
$C_{60}^{\cdot+}$	11.36 ± 0.05 [11]	67 ± 2 [19]			
C_{60}^{2+}	15.6 ± 0.5 [11] 16.6 ± 1 [20]	84 ± 10 [19]			

3.3. Reactions with $C_{60}^{\cdot 3+}$

Four of the five molecules of type XCl chosen for study reacted with $C_{60}^{\cdot 3+}$. Chlorine did not react. There was no evidence for Cl^- transfer or $Cl^{\cdot}$ abstraction. According to currently-available thermodynamic data, the reaction with chlorine is the most (thermodynamically) favourable with regard to Cl-atom transfer and the least favourable to the transfer of Cl^- to $C_{60}^{\cdot 3+}$: $X = Cl^{\cdot}$ has the lowest Cl-atom affinity and $X^+ = Cl^+$ has the highest Cl^- affinity. The nonreaction with chlorine reported here supersedes our early qualitative report of a fast reaction [21].

The molecules CH_xCl_{4-x} with $x = 0–3$ were observed to react with $C_{60}^{\cdot 3+}$ by bimolecular chloride transfer and electron transfer, reactions that result in charge reduction/charge separation, according to Eq. (2):

$$C_{60}^{\cdot 3+} + CH_xCl_{4-x} \rightarrow C_{60}Cl^{\cdot 2+} + CH_xCl_{3-x}^{+} \qquad (2a)$$

$$\rightarrow C_{60}^{2+} + CH_xCl_{4-x}^{\cdot +} \qquad (2b)$$

Table 2 shows that the Cl^- affinity, $Cl^-A(CH_xCl_{3-x}^{+})$, increases and $IE(CH_xCl_{4-x})$ decreases as x increases. Thus Cl^- transfer is expected to be most favourable with carbon tetrachloride and electron transfer is most favourable with methyl chloride and this is what is observed.

3.3.1. Reactions with CCl_4 and $DCCl_3$

Plots of observed reactant and product ion signals versus neutral reactant flow for the reactions of carbon tetrachloride and chloroform with $C_{60}^{\cdot 3+}$ are shown in Figs. 1 and 2. Only Cl^- transfer was observed. There was no evidence for the occurrence of electron transfer (<1%). Also, neither of the product ions of the charge-separation reactions reacted further with CCl_4, $k < 1 \times 10^{-13}$ cm^3 $molecule^{-1}$ s^{-1}; polychlorination was not observed. Both chloride-transfer reactions are rapid and efficient. The reaction with CCl_4, which is more exothermic by 8 kcal mol^{-1} than the reaction with $DCCl_3$, is slightly faster. When compared to calculated collision rate coefficients using average-dipole orientation (ADO) theory [22], the measured rate coefficients imply reaction efficiencies of 0.76 and 0.33, respectively.

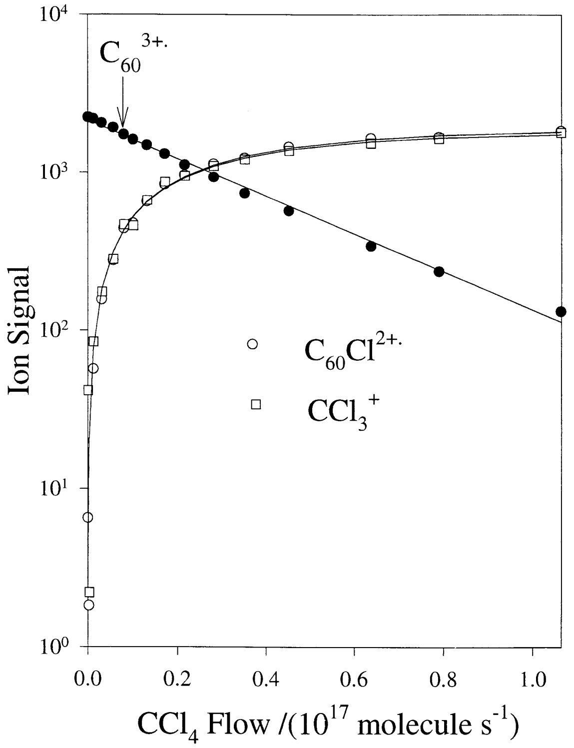

Fig. 1. Experimental data for the chemistry initiated by the reaction of $C_{60}^{\cdot3+}$ with carbon tetrachloride. $C_{60}^{\cdot3+}$ was produced in a low-pressure ion source by electron impact at 100 eV with C_{60} vapour derived from a fullerene powder. The lines represent fits to the experimental data with solutions of the systems of differential equations appropriate for the observed reactions.

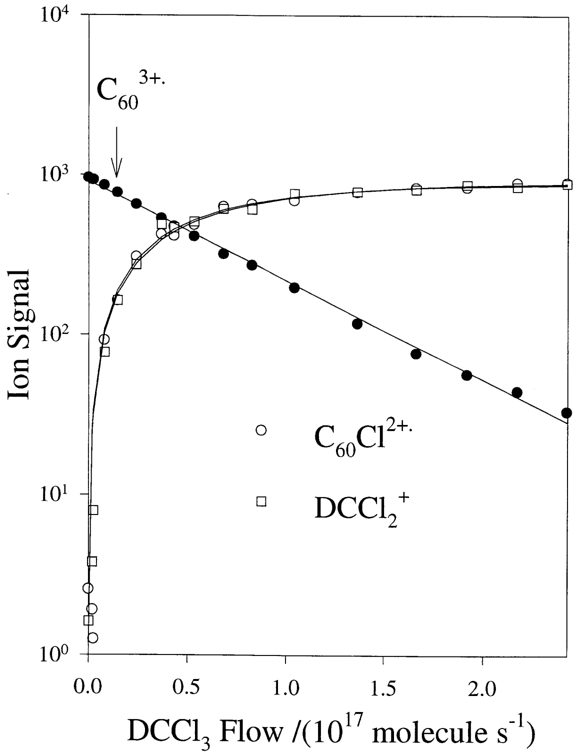

Fig. 2. Experimental data for the chemistry initiated by the reaction of $C_{60}^{\cdot3+}$ with deuterated chloroform. $C_{60}^{\cdot3+}$ was produced in a low-pressure ion source by electron impact at 100 eV with C_{60} vapour derived from a fullerene powder. The lines represent fits to the experimental data with solutions of the systems of differential equations appropriate for the observed reactions.

3.3.2. Reactions with CH_2Cl_2

The reaction of $C_{60}^{\cdot3+}$ with methylene chloride is more than 20 times slower than the reaction with chloroform. Figure 3 shows that CH_2Cl_2 reacts with $C_{60}^{\cdot3+}$ primarily by Cl^- abstraction (≥91%), but there is also some evidence of a minor (≤9%) channel leading to electron transfer, this is evident from the product ratio analysis shown in Fig. 4 indicating what could be a finite intercept at zero flow for the % production of C_{60}^{2+}. Figure 4 indicates that most of the C_{60}^{2+} observed at higher flows is produced from $C_{60}Cl^{2+}$ in either a secondary reaction or collisional dissociation of a fraction of the $C_{60}Cl^{2+}$ produced in the primary reaction with the added methylene chloride. The former would require a relatively weak C_{60}^{2+}–Cl bond, whereas the latter would require internal or kinetic excitation of some of the $C_{60}Cl^{2+}$ produced in the primary reaction. Again, there was no evidence for the further chlorination of $C_{60}Cl^{2+}$ by higher-order reactions.

The observed CH_2Cl^+ product of the Cl^- transfer charge-separation reaction appears to react further with methylene chloride to produce $CHCl_2^+$ and $C_2H_3Cl_2^+$ according to reaction (3):

$$CH_2Cl^+ + CH_2Cl_2 \rightarrow CHCl_2^+ + CH_3Cl \qquad (3a)$$

$$\rightarrow C_2H_3Cl_2^+ + HCl \qquad (3b)$$

This chemistry was not investigated separately.

3.3.3. Reactions with CH_3Cl

Methyl chloride also reacts slowly with $C_{60}^{\cdot3+}$ but, unlike the reactions of the other chlorinated methanes,

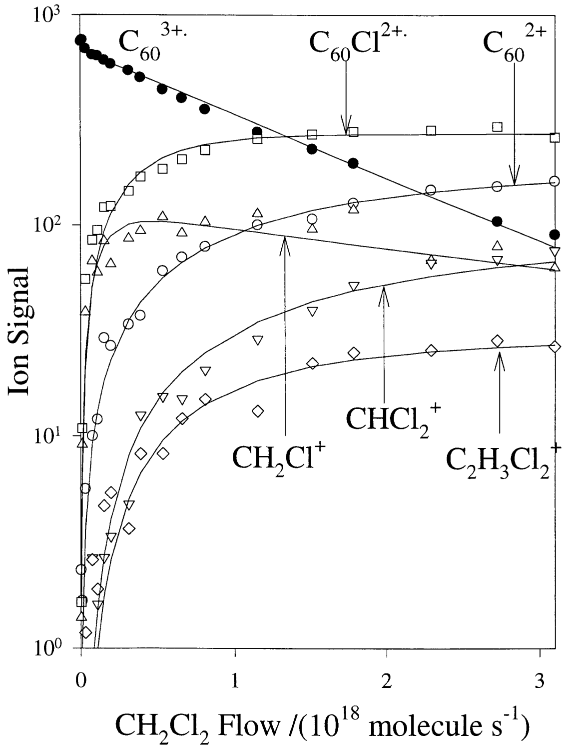

Fig. 3. Experimental data for the chemistry initiated by the reaction of $C_{60}^{\cdot 3+}$ with methylene chloride. $C_{60}^{\cdot 3+}$ was produced in a low-pressure ion source by electron impact at 100 eV with C_{60} vapour derived from a fullerene powder. The lines represent fits to the experimental data with solutions of the systems of differential equations appropriate for the observed reactions.

electron transfer, rather than chloride transfer, is the main reaction channel. Figure 5 shows data for the occurrence of the following three reaction channels:

$$C_{60}^{\cdot 3+} + CH_3Cl \rightarrow C_{60}^{2+} + CH_3Cl^{\cdot +} \tag{4a}$$

$$\rightarrow C_{60}Cl^{\cdot 2+} + CH_3^+ \tag{4b}$$

$$\rightarrow C_{60}CH_3^{\cdot 2+} + Cl^+ \tag{4c}$$

The product ratio plot in Fig. 4 again indicates the same conversion of some $C_{60}Cl^{2+}$ to C_{60}^{2+} at low flows of reactant observed with CH_2Cl_2 and presumably has the same origin. A minor fraction (11%) of the collisions of $C_{60}^{\cdot 3+}$ with CH_3Cl lead to the production of $C_{60}CH_3^{\cdot 2+}$ by C–C bond formation, but electron transfer clearly predominates. The ionization energy of CH_3Cl of 11.26 eV lies close to the onset for electron transfer of 11.2 eV that we have determined previously in a general study of electron-transfer reactions with $C_{60}^{\cdot 3+}$. The recombination energy of $C_{60}^{\cdot 3+}$ is more than 4 eV higher (see Table 2) so that electron transfer with CH_3Cl is very exothermic. But electron transfer is inhibited by the barrier that arises from the coulombic repulsion between the doubly and singly charged product ions [11]. It appears that the electron transfer reaction with CH_3Cl, which occurs about 100 times slower than the collision rate, is close to surmounting this barrier. It should be noted that CH_3Cl has the lowest ionization energy of the five molecules investigated and this accounts for our failure to observe electron transfer to $C_{60}^{\cdot 3+}$ with the other four molecules. The observation of chloride transfer with CH_3Cl allows the chloride affinity of $C_{60}^{\cdot 3+}$ to be set at ≥226 kcal mol^{-1} (see Table 2).

The observed $CH_3Cl^{\cdot +}$ product of the Cl^- transfer charge-separation reaction appears to react further with methyl chloride to produce $(CH_3)_2Cl^+$ and $C_2H_5Cl_2^+$. Separate investigations of this chemistry in which $CH_3Cl^{\cdot +}$ was produced from methyl chloride by electron impact in the ion source of the SIFT apparatus [23] have shown that these two ions arise from the following reaction sequence:

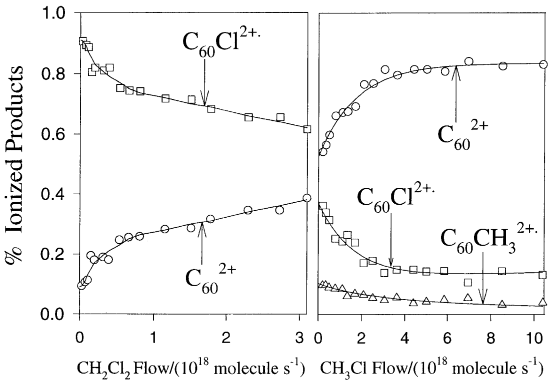

Fig. 4. Product percentage plots for the products observed in the reactions of $C_{60}^{\cdot 3+}$ with methylene chloride and methyl chloride shown in Figs. 3 and 5.

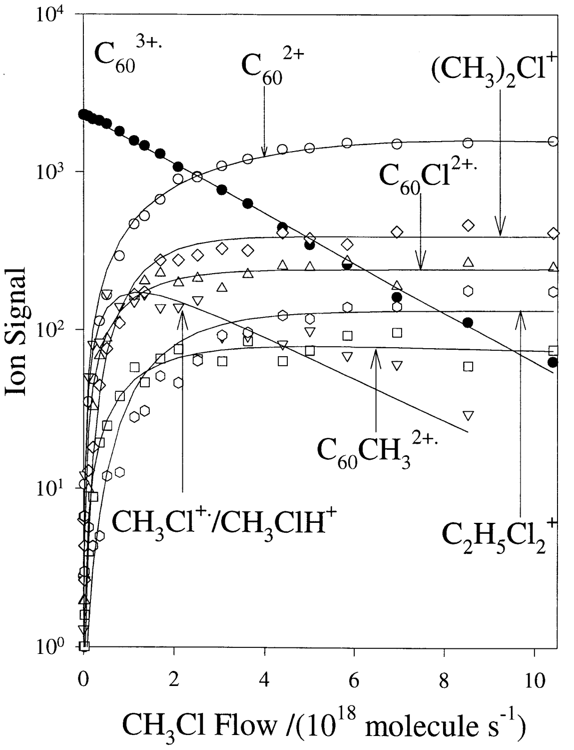

Fig. 5. Experimental data for the chemistry initiated by the reaction of $C_{60}^{\cdot 3+}$ with methyl chloride. $C_{60}^{\cdot 3+}$ was produced in a low-pressure ion source by electron impact at 100 eV with C_{60} vapour derived from a fullerene powder. The lines represent fits to the experimental data with solutions of the systems of differential equations appropriate for the observed reactions.

$$CH_3Cl^{\cdot +} + CH_3Cl \rightarrow CH_3ClH^+ + CH_2Cl^{\cdot} \qquad (5a)$$

$$\rightarrow (CH_3)_2Cl^+ + Cl^{\cdot} \qquad (5b)$$

$$CH_3ClH^+ + CH_3Cl \rightarrow (CH_3)_2Cl^+ + HCl \qquad (6a)$$

$$\rightarrow C_2H_5Cl_2^+ + H_2 \qquad (6b)$$

4. Conclusions

Our measurements have shown that positively charged buckminsterfullerenes are not readily chlorinated at room temperature in reactions with chlorine and chlorinated methanes. This is certainly true of the first two charge states and, according to Olah et al. [13], is also the case with neutral C_{60}. We suggest that these nonreactions are kinetically rather than thermodynamically controlled, viz. that kinetic barriers arise from the distortion of the C_{60} carbon cage that is required for near-sp^3 hybridization at the C site of bond formation with Cl.

The electrostatic interaction between reagent molecules and $C_{60}^{\cdot 3+}$ is sufficient to promote chloride transfer to this ion in charge-reduction/charge-separation reactions with CCl_4, $CDCl_3$, and CH_2Cl_2. With CH_3Cl chloride transfer is dominated by electron transfer but is still observed. The results are consistent with known trends in chloride affinity and ionization energy. Single or multiple chlorination with chlorine, of the type reported for neutral C_{60} at elevated temperatures [13], does not proceed with the first three charge states of C_{60}^{x+} under SIFT conditions *at room temperature*.

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