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Production of small binary carbon clusters by laser ablation of thin films of derivatised fullerenes

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Dedicated to Prof. Helmut Schwarz on the occasion of his 60th birthday.

Abstract

The distribution of small carbon clusters derived by laser ablation (laser desorption/ionization) of thin films of derivatised fullerenes has been studied. The production of positive and negative ions has been monitored by time-of-flight (ToF) mass spectrometry. The materials under investigation included the [60]fullerene (C_{60}), hydrogenated and deuterated [60]fullerenes ($C_{60}X_{36}$ where $X = H$ and D), fluorinated [60]fullerenes ($C_{60}F_x$ where $x = 46$ and 48) and the aza[60]fullerene dimer ($(C_{59}N)_2$). Following laser ablation using a nitrogen laser, the efficient formation of small, negatively charged carbon clusters has been observed, while the corresponding positively charged clusters were not formed. Derivatised fullerenes display the co-formation of small binary carbon clusters of the type C_nX^-, where n shows odd/even alternation depending on X which represents the heteroatom initially linked to or networked with the fullerene cage. Although binary carbon clusters C_nX^- were formed where $X = H$, D and N, the ablation of fluorofullerene targets would not lead to the formation of C_nF^- clusters under the applied conditions, as confirmed by ablation experiments with chloro/fluoroalky-polymers. The formation of the binary carbon clusters has been also studied by laser fluence-dependent production of C_nD^- ions from $C_{60}D_{36}$, and by the production of C_nP^- ions using a composite C_{60}/P_4 target. Possible pathways leading to small binary carbon clusters are addressed.

Keywords: Laser ablation; Small binary carbon clusters; Fullerenes

1. Introduction

Laser ablation, commonly also referred to as laser desorption/ionization, has greatly contributed to our present understanding of the formation, structure and reactivity of clusters. Clusters are the intermediate species bridging the features of the singular atom or molecule and the bulk. This is particularly true for research into carbon-based clusters, which has enormously expanded through the advent of the fullerenes. Moderation of the energy deposited into a solid target allows to broaden the use of laser desorption, as a

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means to study the material properties of solid matter. If the laser fluence is carefully adjusted so that the energy deposition remains close to the threshold of desorption and ion formation, direct laser desorption can be used as an analytical tool, providing intact molecular ions of the analyte. This has been demonstrated successfully for the analysis of polyaromatic hydrocarbons [1], as well as for thermolabile fullerene derivatives [2], although the use of an energy-moderating matrix material in matrix-assisted laser desorption/ionization (MALDI) has been more commonly applied to such analyses [3–5]. With increasing energy deposition not only fragmentation occurs, but through the increased density of particles in the ablation plume, aggregation and fusion reactions resulting in larger entities are promoted. Such phenomena have been of particular interest using derivatised fullerenes, as a means to study the aggregation [6,7] and coalescence/fusion [8–11] into larger, nano-sized carbon-based particles. While product analysis and coalescence reactivity have been major points of interest in laser desorption of derivatised fullerenes, the production of small carbon cluster ions derived from these new materials has not yet been studied in much detail [12,13]. However, the interest into small carbon clusters has well extended into the post-fullerene era and results not only from their potential involvement in formation and decomposition of fullerenes, but is also caused by their importance as interstellar matter. Besides the interest in pure carbon clusters, binary clusters of the type C_nX^- have attracted much attention, covering both experimental [14–19] and theoretical aspects [16–24]. The C_nX^- anions have been generated for a large variety of heteroatoms X. Ample evidence has been gathered indicating a chain-like geometry of these clusters, which also display a pronounced odd/even carbon content alternation. For $X = H$ and F even carbon species are more stable than their odd-numbered analogues; for $X = N$ and P the situation is reversed. Laser ablation of suitable target materials features prominently as a means to generate these clusters. In addition to the use of polymers [14,15] as target materials for the binary cluster production, the most frequently applied method utilises the ablation of composite targets that are composed of two compounds, each providing one of the components of the binary clusters [17].

The present investigation evaluates the use of derivatised fullerenes as precursors for such small, binary clusters. The target materials included the [60]fullerene (C_{60}), hydrogenated and deuterated [60]fullerenes ($C_{60}X_{36}$ where $X = H$ and D), fluorinated [60]fullerenes ($C_{60}F_x$ where $x = 46$ and 48) and the aza[60]fullerene dimer ($(C_{59}N)_2$). Since both elements of the binary clusters are provided by a single compound in these cases, the formation mechanism of the binary clusters has been discussed regarding the importance of direct fragmentation of the fullerene compounds, as well as the involvement of uptake of the heteroatom by the pure carbon clusters.

2. Experimental

2.1. Instrumentation

Laser ablation was performed using a nitrogen laser at an ultraviolet wavelength of 337 nm with a pulse width of 3 ns and frequency 1.5 Hz. Ions resulting from laser desorption/ionization were accelerated by a continuous acceleration voltage of 20 kV into a time-of-flight (ToF) mass spectrometer for analysis. The mass spectrometer is composed of a linear flight tube and a quadratic-field reflectron (Kompact MALDI IV, Kratos Inc., Manchester, UK). The instrumental set-up has been described in more detail elsewhere [25]. All positive- and negative-ion mass spectra were recorded in reflectron mode. The mass resolution was sufficient to resolve all nominal mass signals in the mass range studied. The corresponding mass accuracy would only allow the assignment of a nominal mass, rather than providing accurate mass information. Two hundred single laser shot events were accumulated for each individual mass spectrum and viewed in electronically processed form. To ensure activation of a pristine target area, the sample holder was moved prior to each individual laser shot. The mass assignment was achieved by external calibration using a C_{60}/C_{70} mixture. While all spectra shown here were recorded with the instrument mentioned above, accurate mass measurements were performed on particular ions to establish their elemental composition, using an instrument of higher resolving power and mass accuracy (Voyager-DE STR, Applied Biosystems, Framingham, MA, USA).

2.2. Materials and target preparation

Samples of commercial origin included C_{60} (Hoechst), $C_{60}F_{46/48}$ (Kaesdorf) and Fluorolube^{TM} 600 and 1200 (low molecular weight polymers of chlorotrifluoroethylene $(CF_2CFCl)_n$, KMZ Chemicals Ltd., UK). $C_{60}H_{36}$ was synthesised by hydrogen transfer reduction of C_{60} using dihydroanthracene [26,27]. $C_{60}D_{36}$ was synthesised accordingly using dideutroanthracene. The azafullerene dimer, $(C_{59}N)_2$, was prepared as described in the literature [28]. The fullerene compounds were dissolved either in toluene or tetrahydrofuran at a concentration of 1 mg/mL. Fluorolube^{TM} 600 and 1200 were dissolved in toluene. The solutions were then deposited onto a stainless steel sample holder using a 10 μL syringe. The sample holder was dried in a cold air stream, leaving a thin film of solid material behind.

3. Results and discussion

Laser ablation of C_{60} deposited onto a stainless steel target holder was studied with regard to the formation of low mass clusters in both positive and negative ion modes. The negative ion mass range up to m/z 120 is shown in Fig. 1a. Fig. 1b displays the corresponding positive ion mass range.

Small carbon clusters were only observed efficiently as negative ions. A distribution of $C_n{}^-$ anions was obtained where n covers approximately 2–10 carbon atoms. The cluster distribution varies with increasing laser fluence from an envelope-like to the tail-like appearance shown, which has been obtained at higher laser fluences. Preference for the production of even numbered carbon clusters is evident. By contrast, the corresponding mass range for positive ions shows predominately signals for ions that do not originate from the fullerene film, such as Na^+ and K^+. However, under the conditions applied the molecular ion of C_{60} is formed abundantly in both ion modes and $C_{60}{}^{\bullet+}$ undergoes fragmentation via the well-known evaporation of C_2 moieties [29].

In high-energy collisions of C_{60} ions with inert target gases, small carbon cluster ions can in principle be formed through fragmentation of C_{60} ions in both ion modes [30,31]. Although small positively charged carbon clusters could not be observed, the observed small carbon cluster anions may well be formed by dissociation of $C_{60}{}^-$. Alternatively the formation of the small anionic carbon clusters may occur through the initial dissociation of neutral C_{60}. Small neutral clusters subsequently ionise. In the case of negatively charged species, ionization probably occurs by attachment of secondary electrons. The presence and reducing potential of free secondary electrons also plays a major role in MALDI [32,33], as well as during the ablation/ionization process in liquid secondary ion mass spectrometry (LSIMS) [34]. Small neutral carbon clusters possess positive electron affinities, so that electron attachment should lead to long-lived anions [35,36]. However, the abundant formation of $C_2{}^-$ may indicate that the above mentioned dissociation of ions might play a major role. C_2 has not been observed in Rydberg electron transfer reactions [37] and it is not clear if electron transfer would find in the present experiments sufficient stabilisation to result in long-lived anions of C_2. However, $C_2{}^-$ has been clearly identified as an important daughter ion of larger negatively charged carbon clusters [38].

The production of small negatively charged clusters derived by ablation of the hydrogenated and deuterated [60]fullerenes, $C_{60}H_{36}$ and $C_{60}D_{36}$, is shown in Fig. 2a and b, respectively. The formation of even numbered carbon clusters is clearly enhanced and accompanied by the efficient co-formation of binary clusters of the type C_nX^- where X = H and D. The generation of the binary clusters is evident, as the hydrogen containing species are clearly more abundant than the expected ^{13}C-isotopomer of the pure carbon cluster and the deuterated clusters show the expected mass

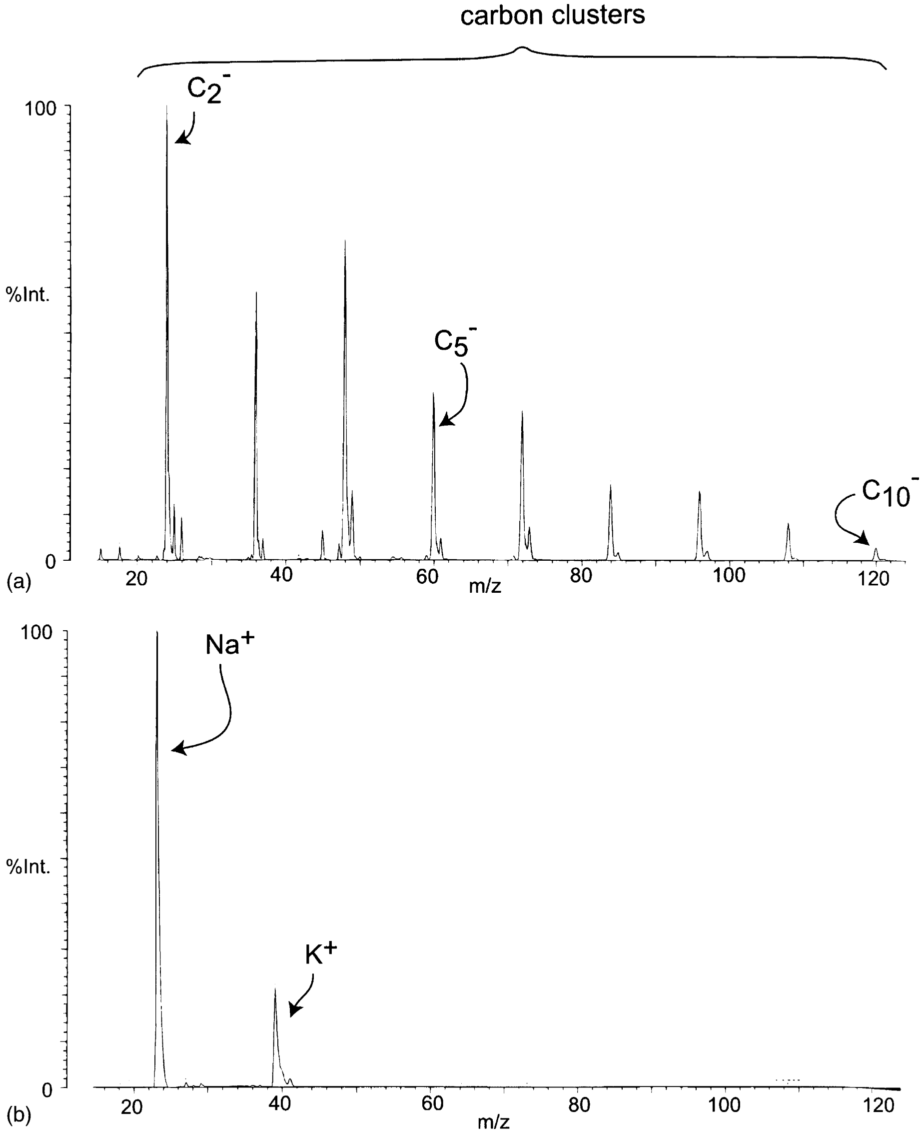

Fig. 1. Low mass region of the laser desorption/ionization mass spectrum of C_{60} in (a) the negative- and (b) the positive-ion mode.

shift. Small binary carbon clusters have been the subject of considerable experimental and theoretical work [14–24]. Laser ablation is well established for the production of these clusters. The target materials commonly employed in these studies involved, for instance, polymer films [14,15] or tailor-made composite targets, containing a mixture of compounds that would provide the two components of the binary clusters [17]. The use of derivatised fullerenes as precursor materials, however, is less well researched and the formation of the binary carbon cluster anions characterises a new material property of these compounds. Earlier work on binary carbon clusters revealed profound odd/even-stability alternations for these species, which are in general believed to be of linear structure. In line with these considerations, only

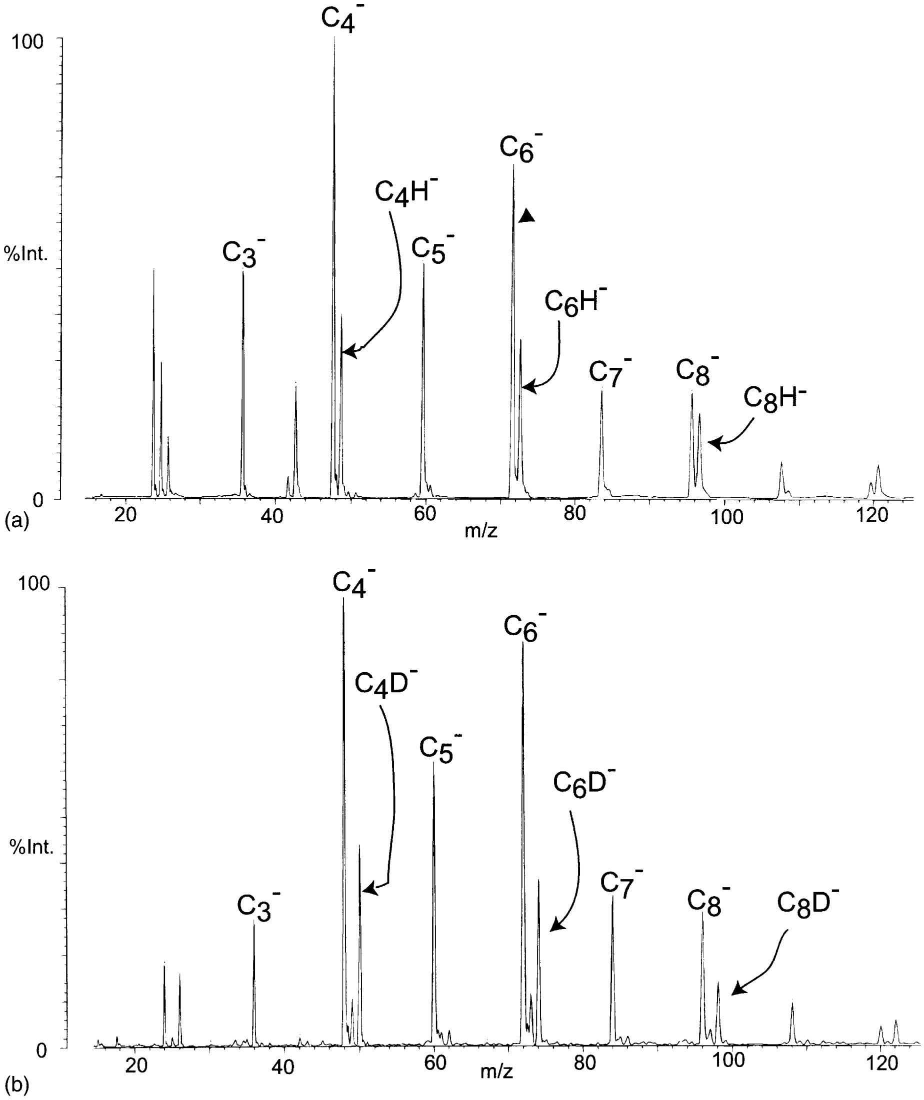

Fig. 2. Low mass region of the negative-ion laser desorption/ionization mass spectrum of (a) $C_{60}H_{36}$ and (b) $C_{60}D_{36}$.

C_nH^- clusters with an even number of carbon atoms were efficiently formed in the present experiments.

The mechanism by which small binary carbon clusters are formed has been of concern in numerous investigations [14–19]. The use of composite target materials in conjunction with laser ablation, for instance, implies inevitably the importance of laser-induced reactions in which individual components combine to form these clusters. In fact, there is ample evidence for the growth of these clusters from smaller entities [14]. The use of derivatised fullerenes, however, employs a material as precursor in which both components of the small binary cluster are chemically linked already, so that direct fragmentation has to be considered as a potentially major pathway towards these small clusters. In order to obtain more insight into the formation mechanism of these binary clusters, the product distribution of C_nD^- ions is displayed in

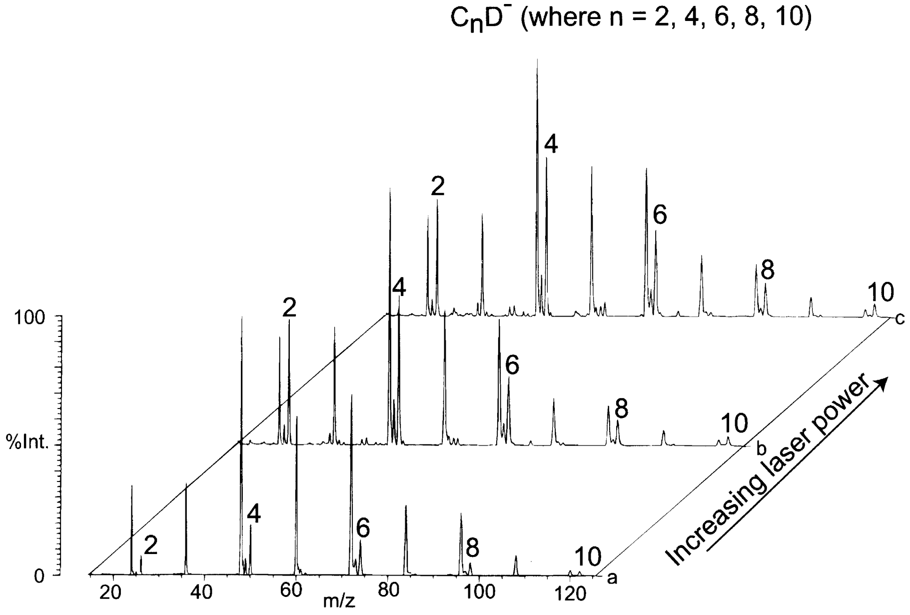

Fig. 3. Production of C_nD^- clusters during ablation of $C_{60}D_{36}$ at increasing laser fluences.

Fig. 3 as derived by activation of a $C_{60}D_{36}$ film with increasing laser fluence. In these experiments, the laser fluence has been varied from near to threshold of ion formation (Fig. 3a), over an intermediate value (Fig. 3b) up to the highest setting possible (Fig. 3c). At low laser fluence, pure carbon clusters are clearly more abundantly observed than the binary C_nD^- clusters. At more energetic ablation of the target, the relative distribution of the pure carbon clusters remains unchanged within the experimental error limit, however, the binary C_nD^- clusters are now more efficiently produced. The abundance of each individual C_nD^- cluster clearly increases relative to the corresponding $C_n{}^-$ counterpart. Although it is difficult to predict the product distribution that would result for different pathways, the observed variation of the cluster distribution is not necessarily in conflict assuming direct decomposition of the deuterofullerene into small C_nD^- clusters. However, the observation that within the studied laser fluence range the abundance of the C_nD^- clusters increased relative to the pure $C_n{}^-$ clusters, may also indicate the involvement of uptake reactions, whereby deuterium would attach to pure carbon clusters. Upon increasing the laser fluence, more material is ablated and the density of the expanding particle plume enhanced, which obviously may promote the formation the C_nD^- clusters through the uptake of deuterium. As will be seen below, C_nH^- clusters, formed by hydrogen uptake, have also been observed occasionally from fullerene targets which were expected to be essentially free of hydrogen. In these cases, residual solvent impurities which were trapped in the solid target may function as the source of hydrogen.

Fig. 4 illustrates the importance of the small carbon cluster formation following the ablation of $C_{60}H_{36}$ (Fig. 4a) and $C_{60}F_x$ where $x = 46$ and 48 (Fig. 4b) by contrasting the low mass region with the molecular ion domain. In Fig. 4a, the $C_{60}{}^-$ signal dominates and is accompanied by an apparent tailing towards higher masses. The latter is caused by $C_{60}H_n{}^-$ ions which are detected approximately up to $n = 10$ and which could not be resolved as individual peaks due to the low resolving power of the experimental set-up. Increasing hydrogen substitution of the fullerene sphere decreases the ability to accommodate the extra electron.

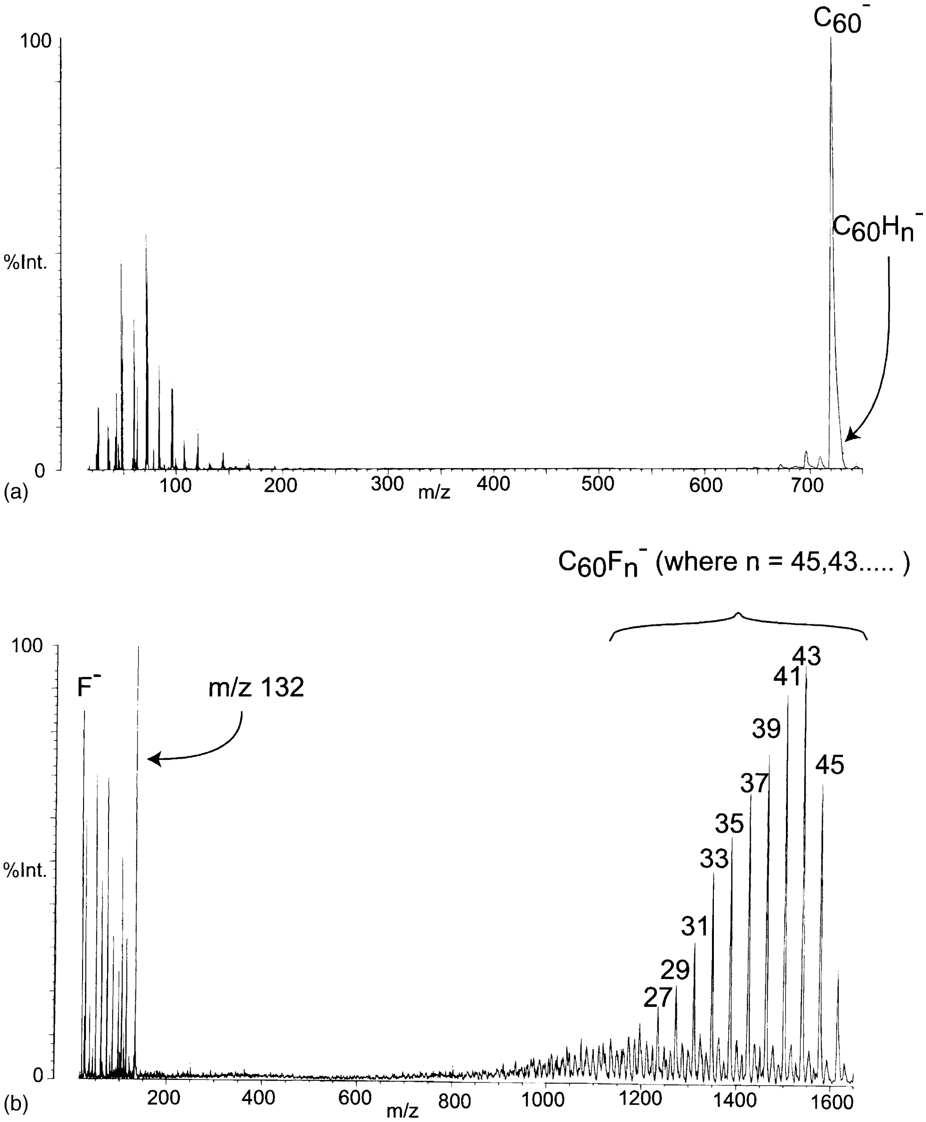

Fig. 4. Comparison of the negative-ion laser desorption/ionization mass spectra of (a) $C_{60}H_{36}$ and (b) $C_{60}F_{46/48}$, contrasting the small carbon cluster region with the molecular ion mass range. The ion at *m*/*z* 132 has been identified as a metal fluoride, resulting from reaction with the support.

In fact, higher degrees of hydrogenation will lead to negative electron affinities and thus to instability of the negatively charged molecular ions [39]. $C_{60}H_{18}^{-}$ has been identified recently as a stable, long-lived molecular anion and possesses so far the highest hydrogen attainment observed for an anionic hydro[60]fullerene [40]. This molecular anion has been accessible from neutral $C_{60}H_{18}$, rather than through electron attachment of $C_{60}H_{36}$ as used here. The hydrogen attainment of C_{60} at which the negative ions become unstable is not known at present.

The close structural similarities of the hydrofullerenes $C_{60}H_x$ where $x = 18$ and 36 to the corresponding fluoro-counterparts $C_{60}F_x$ where $x =$

18 and 36, respectively, is well documented [41]. However, both compound classes behave distinctly different during laser ablation, leading to an almost complementary appearance of the resulting ionic species in the molecular ion region. While C_{60}^- is the dominating product ion following ablation of $C_{60}H_{36}$, the fluorofullerene target produces abundantly $C_{60}F_x^-$ anions, retaining a very high fluorine attainment (Fig. 4b). The production of positive ions (spectra not shown) displays the reversed situation. C_{60}^+ represents the most prominent product ion following ablation of highly fluorinated fullerenes, with no sign of fluorine containing C_{60} ions [42]. However, ablation of hydrofullerenes at appropriate laser fluences leads to the abundant formation of cationic [60]fullerenes that still attain the hydrogen atoms [4]. This behaviour is clearly a consequence of the fluorofullerenes possessing higher values for both ionization energies [43,44] and electron affinities [45]. While fluorine-containing fullerene anions are stabilised through high electron affinities, the corresponding positive ions are not formed because complete fluorine evaporation occurs as ionization is energetically too demanding.

The efficient formation of F^- confirms earlier work which identified the use of laser ablation of fluorofullerenes as a potentially important pulsed source for this anion [12,39]. Binary C_nF^- clusters were not observed. As the formation of binary C_nF^- clusters was recently reported for laser ablation of perfluoropolymer films [14], closely related chloro/fluoro-polymers were used here as targets. These polymers included two low molecular weight polymers of chlorotrifuoroethylene (Fluorolube^{TM} 600 and 1200) of the general formula $–(CF_2CFCl)_n–$, none of which led to the formation of the binary clusters. These polymers were suggested as matrix materials for the efficient formation of molecular anions from fluorofullerenes in MALDI [39]. Although we have tested a large variety of different fluorofullerenes in various ratios admixed with the Fluorolube^{TM} polymers, the formation of fluorofullerene molecular ions always remained minute and the spectra resembled closely the results shown in Fig. 4b. The reported finding that C_nH^- clusters were much more efficiently formed than C_nF^- clusters when ablating the corresponding polymers under similar conditions [14], implies that the production of binary C_nF^- clusters is considerably more energy demanding. The lack of binary C_nF^- clusters for both these types of targets suggests that the applied conditions were insufficient for their production.

The formation of C_nN^- clusters is detailed in Fig. 5a, which displays a partial spectrum following the ablation of the azafullerene dimer, $(C_{59}N)_2$. The enhanced stability of the uneven carbon-containing species is well documented [16,17,21,23]. The formation of these clusters from fullerene-related materials has been observed before in ablation experiments of nitrogen-doped nanotubes [13]. In addition to the C_nN^- clusters (n = uneven) also C_nH^- clusters (n = even) were observed in Fig. 5a. These are probably formed by hydrogen uptake through pure carbon clusters. Molecules of the solvent used to deposit the sample (toluene) may have been trapped in the solid target and functioned as the source of hydrogen. Since $C_{59}N^-$ (inset Fig. 5a) is abundantly observed it is tempting to explain the formation of the small C_nN^- clusters by ion-fragmentation of the monomeric azafullerene anion. Efforts to prove this hypothesis by obtaining a daughter ion mass spectrum of $C_{59}N^-$ in post-source decay (PSD) experiments remained unsuccessful. This is not necessarily contradicting the assumption of C_nN^- formation through direct fragmentation, as it can be assumed that on the metastable ion time frame of the PSD experiment, the electron detachment rate of $C_{59}N^-$ competes efficiently with fragmentation, so that the fragment ion formation may be dramatically reduced. It may be noted in this context, that it took almost half a decade to obtain a meaningful collision-induced dissociation (CID) mass spectrum of C_{60}^- [31].

Finally, a mixture of C_{60} with P_4 was laser ablated, representing a target material in which the heteroatom is not part of or connected to the fullerene cage. The result is shown in Fig. 5b, featuring the formation of C_nP^- clusters while $C_{59}P^-$ is virtually not formed (inset Fig. 5b). These findings provide strong support for the above-mentioned uptake of the hetero-component through the small carbon clusters.

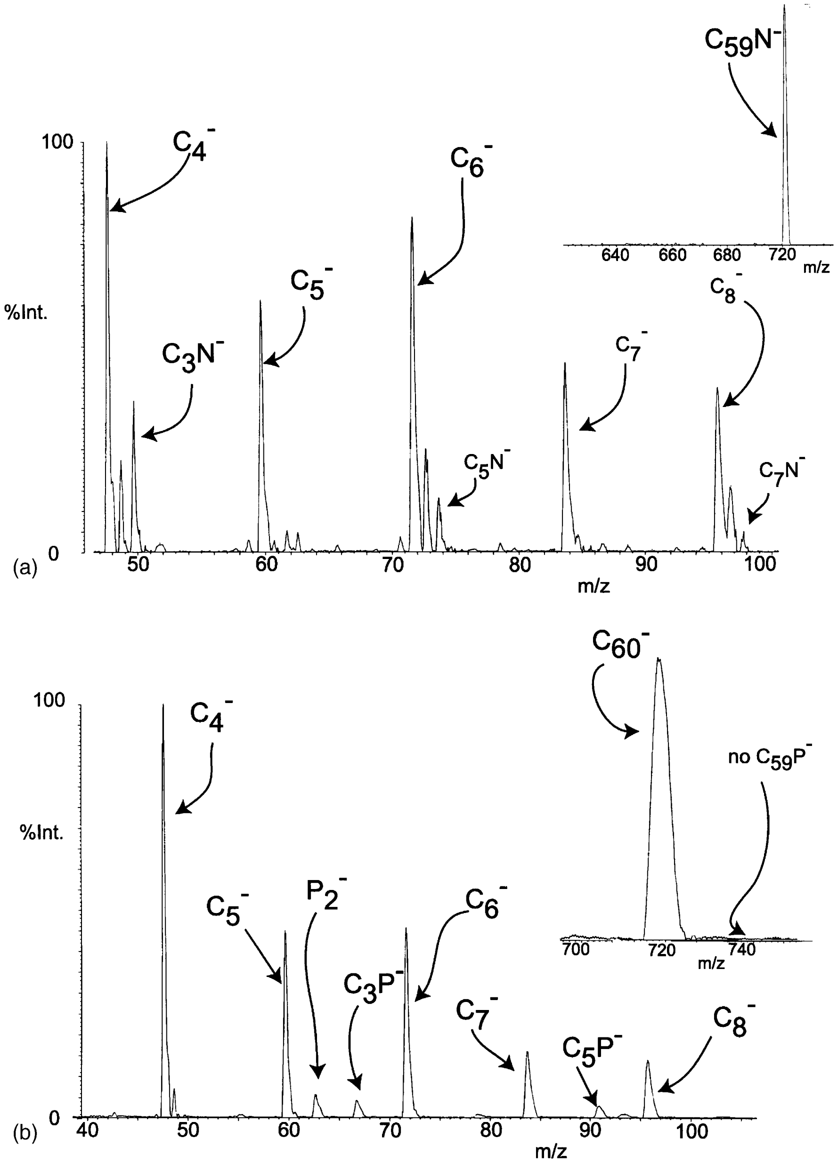

Fig. 5. (a) Production of C_nN^- clusters during ablation of the $(C_{59}N)_2$ dimer. The inset shows the presence of $C_{59}N^-$. (b) Production of C_nP^- clusters following the ablation of a C_{60}/P_4 mixture. The inset provides evidence of the absence of $C_{59}P^-$ as potential precursor for these clusters.

4. Conclusion

Small binary carbon cluster anions were produced by laser ablation of derivatised fullerenes. C_nX^- clusters where X = H, D and N were formed with X being either attached to or networked with the fullerene cage. Small binary carbon clusters may be formed by several processes, involving for instance direct fragmentation, as well as aggregation/uptake reactions of the small carbon clusters with the hetero-component. Small C_nF^- clusters could not be generated under the applied experimental conditions.

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