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Mass spectrometric studies of trifluoromethylated fullerenes

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Abstract

Electron ionization, thermal surface ionization, matrix-assisted laser desorption/ionization, electrospray ionization, and electron capture mass spectrometric techniques have been applied to the analysis of mixtures of trifluoromethylated C_{60} derivatives. For the first time ionization energy and electron affinity values for some of the molecules under investigation have been reported. These ionization characteristics have allowed to explain the differences in the results obtained by means of different mass spectral techniques.

Keywords: Fullerene; Trifluoromethylated fullerene; Ionization energy; Electron affinity

1. Introduction

First obtained in 1993 [1], perfluoroalkylated fullerenes form an important class of fullerene derivatives. Perfluoroalkyl groups are known to possess even stronger electron-withdrawing properties than halogen atoms; hence, perfluoroalkylation of fullerene molecules should enhance their electron-acceptor nature, which makes them promising substances for molecular electronics applications. The studies of trifluoromethylated fullerenes, the simplest representatives of this class of substances, demonstrate their remarkable stability against hydrolysis, which is not the case for halogenated fullerenes, thermal stability, which allows to transfer them into a gas-phase without decomposition, and higher solubility in organic solvents, as compared to parent fullerenes and their fluorides [1,2].

The most commonly used method of fullerene trifluoromethylation is based on the solid-phase reaction with silver(I) trifluoroacetate at 320–360 °C [2]. However, similarly to other high-temperature reactions of fullerene functionalization, this methods results in formation of complex mixtures of products containing different number of CF_3-groups, the pure compounds being available only via subsequent high performance liquid chromatography (HPLC) separation. Therefore, reliable methods of analysis of the synthetic trifluoromethylated fullerenes are critically needed in order to characterize the reaction products and find approaches to more selective synthetic conditions. In the present paper we report the results of study of $C_{60}(CF_3)_n$ mixtures by means of electron ionization (EI), thermal surface ionization (TSI), matrix-assisted laser desorption/ionization (MALDI), electrospray ionization (ESI), and electron capture (EC) mass spectrometric techniques. We also present some ionization energy (IE) and electron affinity (EA) values determined in these experiments.

2. Experimental

2.1. Sample preparation

The samples of trifluoromethylated C_{60} were prepared according to the methods described elsewhere [2]. Briefly,

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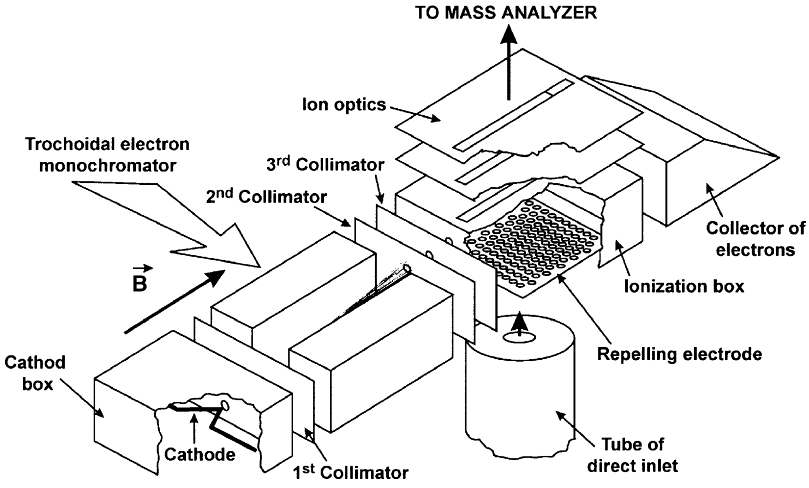

Fig. 1. Ionization system of the mass spectrometer used for EI and EC measurements.

the stirred mixture of C_{60} fullerene {99.9%, Term, USA} and silver(I) trifluoroacetate {98%, ACROS, USA} was heated in the hermetically sealed reactor at 320 °C during 1 h. Then, the reaction product was sublimed under vacuum (~10 Pa) at 420–520 °C. Samples 1–3 mentioned below represented the sublimation fractions obtained at 420, 480 and 520 °C, respectively. According to the HPLC[1] data, the relative abundance of higher trifluoromethylated products was found to decrease with the increase of temperature.

2.2. Electron ionization mass spectrometry

EI mass spectra acquisition and ion appearance energies determination were carried out with the use of modified MI-1201V magnetic sector instrument. In Fig. 1, we present the principal scheme of the ion source described in detail elsewhere [3]. The emitted electron beam passed a trochoidal electron monochromator, which allowed to decrease characteristic width of electron kinetic energy distribution down to 0.15 eV, and ionization chamber was equipped with a repelling interior electrode to improve uniformity of electrostatic field. The samples were evaporated from an open surface (steel capillary). The energy of ionizing electrons was varied in the range of 0–81.9 eV, appearance energy of H_2O^+ cation used as calibration standard [4].

Due to electron-withdrawing nature of the molecules under investigation, the rest of the mass spectrometric techniques employed allowed only negative ion studies.

2.3. Electron capture mass spectrometry

EC experiments were carried out with the use of the same apparatus in the sub-ionization electron energy range of 0–20 eV. Zero-energy EC resonance of SF_6 was used for calibration [4].

2.4. Thermal surface ionization mass spectrometry

Negative ion TSI mass spectra were obtained with the use of the same MI-1201V equipped with a Knudsen cell and a combined ion source, which could operate in two modes: electron ionization of the molecular beam effusing from the cell and extraction of thermal ions formed due to high-temperature surface ionization inside the cell. This technique allows to measure constants of ion-molecular equilibria [5], and, correspondingly, EA values, provided a reference molecule with known EA is involved in the charge-exchange reaction. The modes of operation of the ion source are explained in Fig. 2. The Knudsen cell was typically loaded with several mg of a sample studied and several mg of C_{60} or C_{70} as abovementioned reference compounds (EA = 2.667 ± 0.001 [6] and 2.75 ± 0.01 eV [7], respectively). Also small admixture of potassium fluoride was added in order to enhance negative ion yields via reduction of surface work function (see Ref. [5]).

2.5. Matrix-assisted laser desorption/ionization mass spectrometry

Negative ion MALDI mass spectra were acquired with the use of Bruker AutoFlex reflectron time-of-flight mass spectrometer equipped with N_2 laser (337 nm, 1 ns pulse duration). *Trans*-2-[3-(4-*tret*-Butylphenyl)-2-methyl-2-propenylidene]malononitrile (DCTB) {≥99%, Fluka, Switzerland} was used as a matrix. This matrix has been already successfully applied to negative ion studies of fluoro- and chlorofullerenes [8,9].

2.6. Electrospray ionization mass spectrometry

Negative ion ESI mass spectra were obtained with the use of Agilent 1100 ion trap LC-MS instrument. The *o*-diclorobenzene solutions of trifluoromethylfullerenes (ca. 0.1 mmol/l) without some preliminary generation of ions were directly injected into the steel spraying capillary with a flow rate of 16.7 μl/min. The

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[1] 250 mm × 4.6 mm Cosmosil Backyprep column (Nacalai Tesque, Japan), eluted with toluene at a flow rate of 2 ml min^{-1}, monitored at 290 nm.

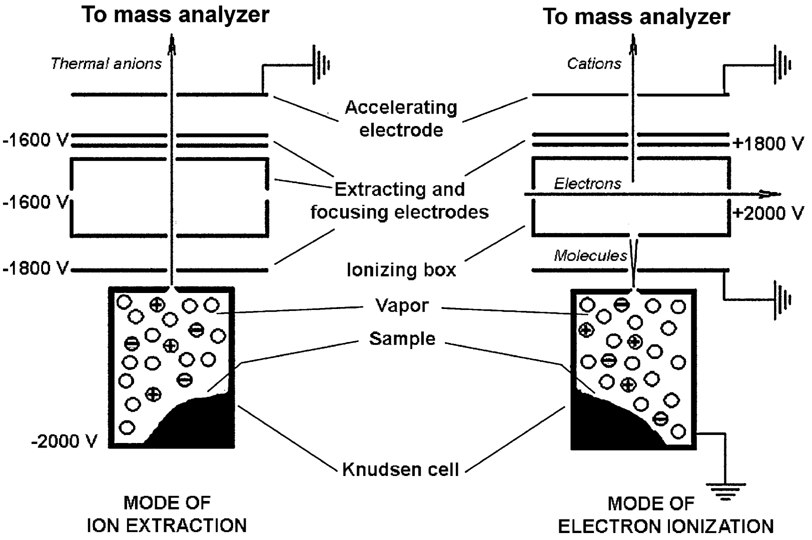

Fig. 2. Principles of ion source operation in the EI and TSI modes.

temperature of counter-propagating drying nitrogen gas lied in a range of 300–350 °C.

3. Results and discussion

3.1. EI mass spectra

Mass spectra of three samples characterized by different depth of trifluoromethylation are presented in Fig. 3. The main signals in a mass range of single charged ions for all three spectra are due to C_{60}^+ and $C_{60}(CF_3)_n^+$ ions. The double charged ions were also observed (see inset into Fig. 3). The distribution of ions demonstrates obvious correlation with degree of trifluoromethylation, for both single and double charged ions the abundance of those with even values of n (hereinafter “even species”) being considerably higher than the abundance of those having odd n (hereinafter “odd species”).

The appearance energy (AE) values of $C_{60}(CF_3)_n^+$, $n = 1–10$, ions estimated from the thresholds of ionization efficiency curves are given in Table 1. Considerably higher AE values of odd species indicate that these ions are products of detachment of CF_3-groups, whereas even species are, most probably, molecular ions and, therefore, the samples contain no significant amounts of odd species. This conclusion made possible to determine IE values for even species via their ionization efficiency curves by means of a Marquardt–Levenberg algorithm fitting treatment described in Ref. [10] which took the energy spread of real experiment into account. The original ionization efficiency curves are given in Fig. 4 and the IE values thus extracted are given in Table 1. Unfortunately, in case of $n = 2$ and 4 no meaningful result was obtained due to low signal-to-noise ratio. Not, surprisingly, the IE values show an apparent tendency to increase with n, which is probably due to increase of the number of electron-withdrawing addends. One should also note that the slopes of the ionization efficiency curves for even species (see Fig. 4) showed no inflections originating from ionization + fragmentation of the molecules containing higher number of CF_3-groups. The odd species were likely to have a single molecular precursor as well, since again no inflections were observed. Thus, the loss of a single CF_3-group was the only fragmentation pathway detected in our experiments.

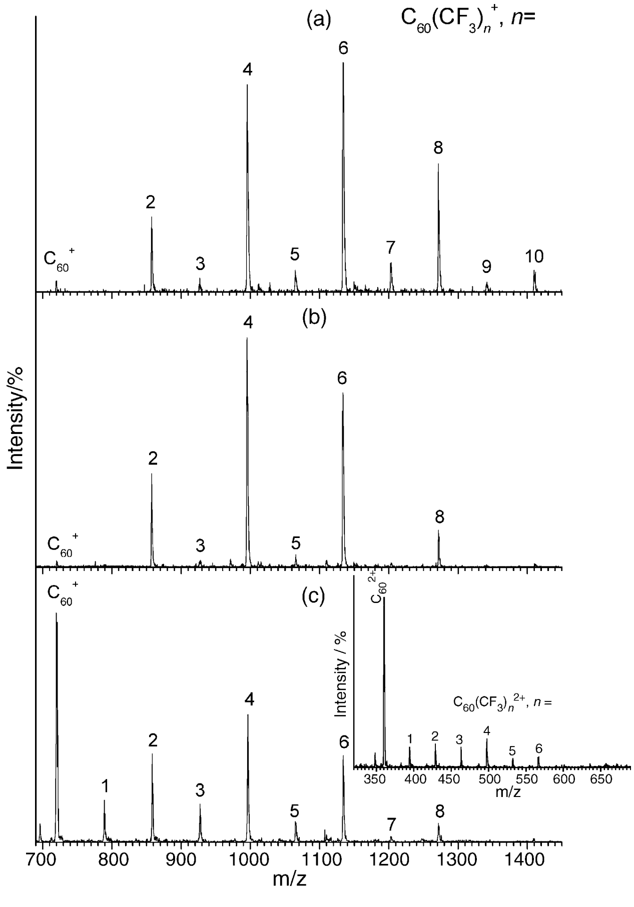

Fig. 3. EI mass spectra for samples 1–3 (a–c, respectively). The inset presents the doubly charged ions range.

Table 1
Appearance energy and ionization energy values (see text for details) for $C_{60}(CF_3)_n$ species

n for $C_{60}(CF_3)_n^+$	10	8	7	6	5	4	3	2	1
Appearance energy (eV)	9.1 ± 0.4	8.8 ± 0.7	16.7 ± 1.0	8.8 ± 1.1	15.6 ± 0.7	9.2 ± 0.6	15.0 ± 1.6	8.7 ± 0.4	15.9 ± 0.3
Ionization energy (eV)	8.90 ± 0.21	8.00 ± 0.16		7.60 ± 0.12					

3.2. EC mass spectra

The main peaks in the EC mass spectra are due to C_{60}^- and $C_{60}(CF_3)_n^-$, $n = 1$–8, ions, as well as metastable ion signals discussed below. Although the intensity distribution was not well reproducible, the most abundant signal in all the experiments was due to $C_{60}(CF_3)_4^-$ ion. The EC cross-section curves were measured for both stable C_{60}^- and $C_{60}(CF_3)_n^-$ ions ($n = 1$–8) and metastable $C_{60}(CF_3)_n^-$ species ($n = 2$–5), which underwent CF_3-group loss in the mass analyzer.

The EC cross-section curves for stable $C_{60}(CF_3)_2^-$ and $C_{60}CF_3^-$ species are shown in Fig. 5. Similarly to the cases of parent fullerenes and fluorofullerenes [3,11], $C_{60}(CF_3)_2^-$ ions exhibit sharp peak at thermal energies and broad bell-like resonances in the higher electron energy range of the spectra up to ca. 5 eV. Analogously to the parent fullerenes, the latter broad peaks can be accounted for by plasmon excitations [12]. The EC curve of $C_{60}(CF_3)_2^-$ contains two additional resonant peaks at ca. 8 and 13 eV. The EC curve of $C_{60}CF_3^-$ contains three peaks at ca. 6, 11 and 16 eV. Noteworthy, the resonances of $C_{60}CF_3^-$ lie in the gaps between those of $C_{60}(CF_3)_2^-$ and all three energy differences between the neighboring resonance peaks equal approximately the same value of 5 eV. We suppose the resonances observed are due to dissociative electron capture by the most abundant components of the samples according to the following scheme:

$C_{60}(CF_3)_2 + \bar{e} \rightarrow C_{60}(CF_3)_2^-$ (peak in energy range of 0–5 eV)

$C_{60}(CF_3)_4 + \bar{e} \rightarrow C_{60}(CF_3)_2^- + 2CF_3$ (ca. 8 eV peak)

$C_{60}(CF_3)_6 + \bar{e} \rightarrow C_{60}(CF_3)_2^- + 4CF_3$ (ca. 13 eV peak)

$C_{60}(CF_3)_2 + \bar{e} \rightarrow C_{60}CF_3^- + CF_3$ (ca. 6 eV peak)

$C_{60}(CF_3)_4 + \bar{e} \rightarrow C_{60}CF_3^- + 3CF_3$ (ca. 11 eV peak)

$C_{60}(CF_3)_6 + \bar{e} \rightarrow C_{60}CF_3^- + 5CF_3$ (ca. 16 eV peak)

As for the EC curves for other $C_{60}(CF_3)_n^-$ ions, only two resonance peaks were observed for $n = 3$–5 and for $n = 6$–8 only one resonance remained. This behavior is in a good agreement with our supposition about dissociative nature of the second and third resonance peaks, since relative abundance of trifluoromethylated molecules in our samples rapidly decreased with n beyond $n = 6$. In case of odd species, first resonance peaks were observed well in the energy range 0–5 eV, again pointing to their completely dissociative origins. That is also the case for C_{60}^-, its EC curve containing two pronounced resonance peaks at 8.2 and 13.5 eV and no peak in the energy range 0–5 eV and thus being completely different from the reference curve published in Ref. [3].

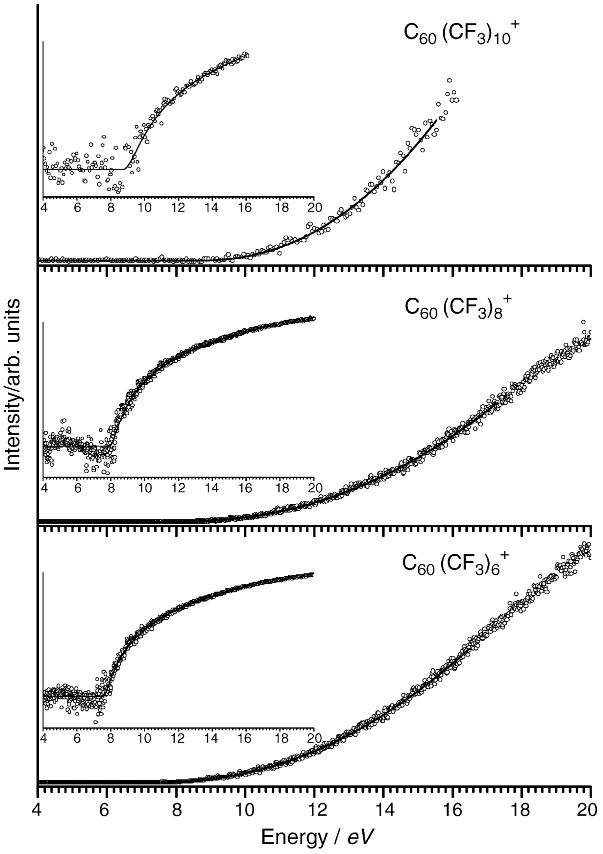

Fig. 4. Ionization efficiency curves for $C_{60}(CF_3)_n^+$ ions (insets show semi-logarithmic plots). Open circles – experimental points; straight lines – curves smoothed according to Ref. [10].

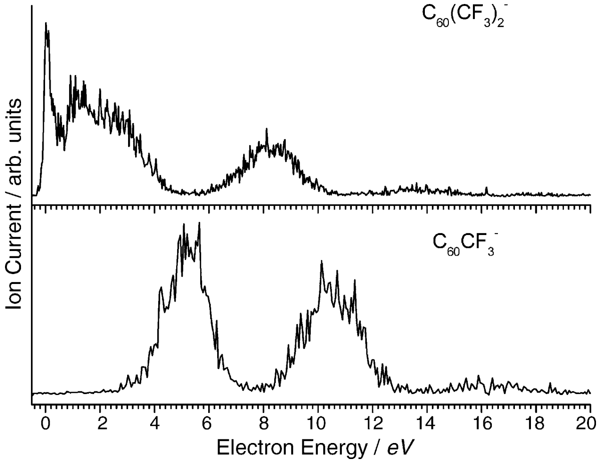

Fig. 5. EC curves for $C_{60}(CF_3)_2^-$ and $C_{60}CF_3^-$.

The metastable ion signals observed provided additional support for our dissociative model, since the mass numbers observed, namely 931, 862, 794 and 726, agreed well with the theoretical values for the following decay processes in between the acceleration region and the mass analyzer:

$$C_{60}(CF_3)_5{}^- \rightarrow C_{60}(CF_3)_4{}^- + CF_3$$

$$C_{60}(CF_3)_4{}^- \rightarrow C_{60}(CF_3)_3{}^- + CF_3$$

$$C_{60}(CF_3)_3{}^- \rightarrow C_{60}(CF_3)_2{}^- + CF_3$$

$$C_{60}(CF_3)_2{}^- \rightarrow C_{60}CF_3{}^- + CF_3$$

3.3. *TSI mass spectra*

One of typical negative ion TSI mass spectrum of triflouromethylated fullerenes is given in Fig. 6. The spectra mainly consist of both even and odd $C_{60}(CF_3)_n{}^-$ species; unlike EI studies, the dominating signals in the TSI spectra are due to odd species. This is probably due to equilibrial nature of the TSI conditions, which favor formation of more stable closed-shell anions of the latter.

In Table 2, we present $\Delta_r G^0_T$ calculated through the equilibrium constant for the following gas-phase electron exchange reactions:

$$C_{60}{}^- + C_{60}(CF_3)_{2k} = C_{60} + C_{60}(CF_3)_{2k}{}^-,$$

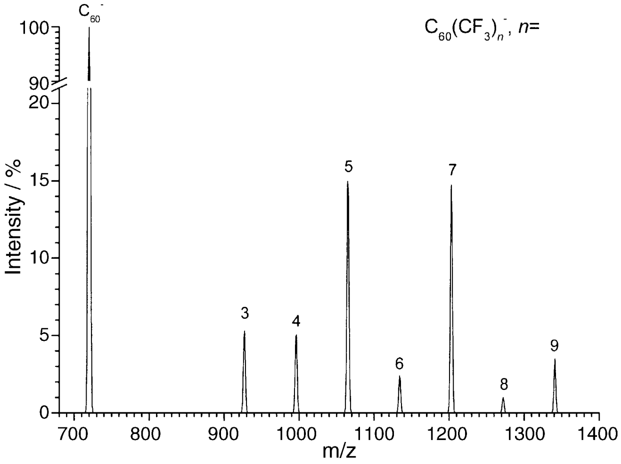

Fig. 6. TSI mass spectrum of a mixture of trifluoromethylated fullerenes.

$$C_{70}{}^- + C_{60}(CF_3)_{2k} = C_{70} + C_{60}(CF_3)_{2k}{}^-.$$

The partial pressure ratios of the species involved were calculated from the ratios of the ion currents measured in the EI (neutral species) and TSI (anions) modes, the ratios of ionization cross-sections were taken by the additivity rule [5]. The total ion current of $C_{60}(CF_3)_{2k}$ molecule in EI spectrum was obtained as a sum of partial currents of $C_{60}(CF_3)_{2k}{}^+$ molecular and $C_{60}(CF_3)_{2k-1}{}^+$ fragmentary ions. The achievement of the equilibrium in electron exchange processes involving fullerenes and their derivatives was demonstrated in our earlier works [7,13,14]. The EA values present in Table 2 for $C_{60}(CF_3)_n$

Table 2
Gibbs energies for electron exchange reactions $C_{60}(\text{or } C_{70})^- + C_{60}(CF_3)_n = C_{60}(\text{or } C_{70}) + C_{60}(CF_3)_n{}^-$ and EA for $C_{60}(CF_3)_n$ molecules

n for $C_{60}(CF_3)_n$	T (K)	Reference molecule	$\Delta_r G^0_T$ (kJ mol^{-1})	EA$\{C_{60}(CF_3)_n\}$ (eV)
4	571	C_{60}	−36.92	3.050
	571	C_{60}	−39.20	3.073
	571	C_{60}	−37.76	3.058
	774	C_{70}	−38.37	3.117
	804	C_{70}	−48.17	3.219
	804	C_{70}	−37.15	3.104
	804	C_{70}	−17.99	2.906
	826	C_{70}	−11.29	2.836
	826	C_{70}	−8.38	2.806
Mean value				3.03 ± 0.20[‡]
6	571	C_{60}	−22.18	2.897
	571	C_{60}	−26.03	2.937
	774	C_{70}	−53.49	3.274
	826	C_{70}	−34.93	3.081
	826	C_{70}	−28.68	3.017
Mean value				3.04 ± 0.14[‡]
8	571	C_{60}	−37.95	3.060
	571	C_{60}	−41.09	3.093
	826	C_{70}	−35.88	3.091
	826	C_{70}	−31.73	3.048
Mean value				3.07 ± 0.06[‡]
10	571	C_{60}	−51.44	3.200
	571	C_{60}	−55.79	3.245

[‡] The error is the standard deviation.

molecules ($n = 4$, 6, 8 and 10) were derived by so-called the third law procedure treatment assuming

$$\Delta_r G^0_T = \text{EA}\{\text{ref}\} - \text{EA}\{C_{60}(CF_3)_n\}$$

or otherwise

$$\Delta_r \Phi^0_T = 0.$$

In Table 2 we present the array of experimental data measured relatively to two different reference molecules, C_{60} and C_{70}. A good agreement of the results obtained with C_{60} and C_{70} additionally evidences the equilibrial nature of the values obtained. The mean values given in Table 2 do not take into account systematic uncertainties due to the assumption of $\Delta_r \Phi^0_T = 0$ and possible presence of several different isomers of the compounds studied. Thus, our values may be considered as isomer-averaged. Since the major isomers of $C_{60}(CF_3)_n$ discussed in Refs. [2,15] reveal considerable similarity of structural motifs for any $n \leq 10$, we do not expect significant differences in their EA. Our computational EA estimations for some of the structures given in [2,15] performed at the DFT level of theory[2] resulted in almost isomer-independent values in the range of 3.0–3.1 eV, thus demonstrating good agreement with the experiment.

3.4. MALDI mass spectra

In Fig. 7 we present negative ion MALDI mass spectra for the three samples of trifluoromethylated fullerenes studied. The main signals are again due to $C_{60}(CF_3)_n{}^-$ ions, even species considerably dominating over the odd ones, similarly to the EI spectra. However, MALDI spectra also contain some signals of oxygenated and hydroxylated ions of general formulae $C_{60}(CF_3)_nO_m{}^-$ ($m = 1$–3), $C_{60}(CF_3)_n(OH)_m{}^-$ ($m = 1$–3), $C_{60}(CF_3)_n(OH)O^-$, $C_{60}(CF_3)_n(OH)_2O^-$ and $C_{60}(CF_3)_n(OH)O_2{}^-$ with even n only, while EI and TSI spectra contained only ambiguous traces of such signals.

The comparison of MALDI and EI mass spectra for the same samples (Figs. 3 and 7), indicates that the ion distributions in the former is shifted towards higher masses. One can assume that in both techniques the probability of detachment of two CF_3-groups should be much lower that the probability of a single group detachment, i.e., virtually negligible. Hence, the discrepancy observed may be due to interplay of two effects: higher formation rates of more electron-withdrawing negative ions in case of MALDI and discrimination of species with lower volatility in case of EI. The influence of the latter effect is rather hard to estimate, whereas the former should favor formation of anions with higher number of CF_3-groups in MALDI mass spectra, as clearly demonstrated in the experiments with fluorinated fullerenes [8]. The intensities of odd species in the EI and MALDI spectra cannot be compared directly, since their origins in the EI spectra are purely dissociative.

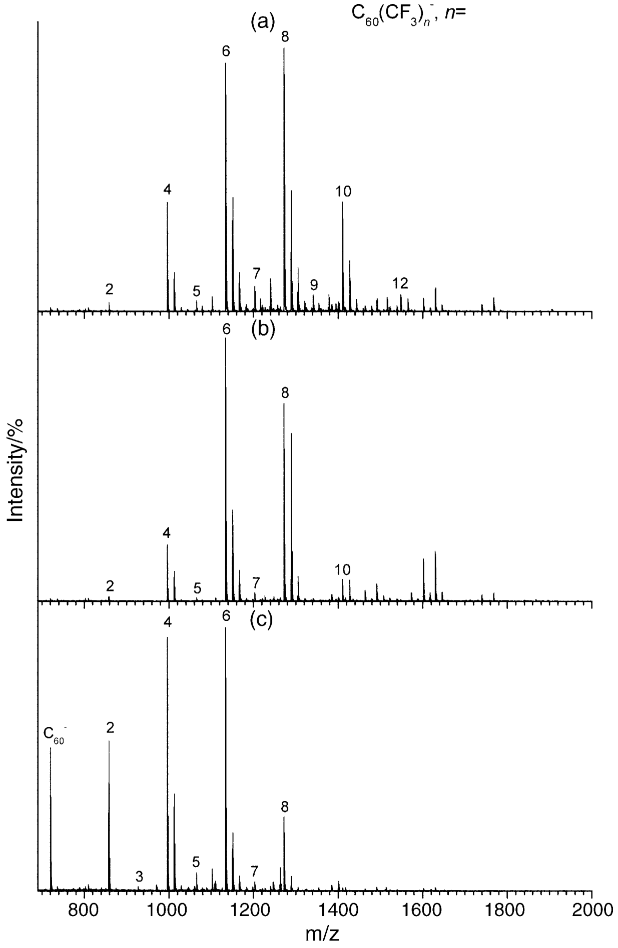

Fig. 7. Negative ion MALDI mass spectra for samples 1–3 (a–c, respectively).

3.5. ESI mass spectra

Typical negative ion ESI mass spectrum is presented in Fig. 8. The ions detected are similar to those observed in MALDI spectra, though the relative amount of odd species is much lower. However, the ion distribution in the ESI spectra is even further shifted to ward higher masses with respect to MALDI and EI. For example, the $C_{60}(CF_3)_6{}^-$ signal, which is comparable in

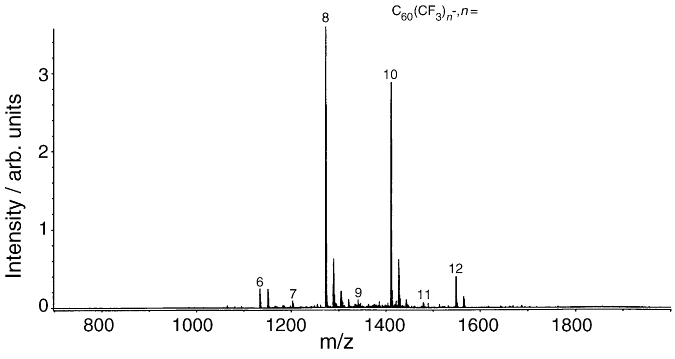

Fig. 8. Negative ion ESI mass spectrum of sample 1.

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[2] PRIRODA software with the implemented original basis set of TZ2P quality (D.N. Laikov, Chem. Phys. Lett. 281 (1997) 151) and the PBE exchange-correlation functional (J.P. Perdew, K. Burke, M. Ernzerhof, Phys. Rev. Lett. 77 (1996) 3865) were used.

intensity to $C_{60}(CF_3)_8^-$ in MALDI mass spectrum, appears to be much less intense than the latter in the ESI spectra. Analogously, the ESI abundances of $C_{60}(CF_3)_{10}^-$ and $C_{60}(CF_3)_{12}^-$ are much higher and those of $C_{60}(CF_3)_4^-$ and $C_{60}(CF_3)_6^-$ are much lower than in MALDI. This indicates even higher influence of the electron affinity on ion formation rates under the ESI conditions, which probably leads to overestimation of the content of higher trifluoromethylated C_{60} derivatives. Hence, those negatively charged odd species observed in higher amounts in MALDI cannot be due to molecular precursors.

3.6. The origins of odd and oxygen-containing species

As demonstrated in the EI and EC studies, the presence of odd species in the trifluoromethylated substances is absolutely negligible. Therefore, the pathways of their formation from even species under the TSI, MALDI and ESI conditions are of own interest. In principle, there exist two possible pathways: (a) CF_3-group detachment upon various interactions with chemical environment and, besides, laser excitation in MALDI and (b) an inverse process, i.e., addition of a previously detached CF_3-group or anion. Direct CF_3-exchange interaction of trifluoromethylated molecules seems improbable due to their low concentrations in the MALDI plume, ESI droplets and interior surface of the Knudsen cell. Although the detachment pathway seems much more probable, there exists an evidence for the attachment mechanism as well. As one can see, the relative intensities of the $C_{60}(CF_3)_6^-$ signal in all three MALDI spectra shown in Fig. 7 are virtually the same, whereas the abundance of the $C_{60}(CF_3)_5^-$ signal in spectrum (b) is lower than in the rest two along with the abundance of $C_{60}(CF_3)_4^-$ signal. Although no such observations were made in case of TSI and ESI, the general possibility of CF_3-group attachment in these cases cannot be excluded as well.

The hydroxylated and oxygenated species observed in the EI, EC, TSI, ESI and MALDI spectra were likely to have the hydroxylated and oxygenated molecular precursors in the samples studied. In the MALDI and ESI spectra they also could originate from interactions with residual moisture and matrix fragments cannot be completely excluded.

4. Conclusion

EI, MALDI and ESI mass spectrometric techniques can be successfully applied to the qualitative analysis of trifluoromethylated fullerenes. $C_{60}(CF_3)_n$ molecules prove to be relatively stable towards fragmentation, the abundance of molecular ions being predominant. However, quantitative analysis of trifluoromethylated fullerene mixtures appears to be a more complex task since ESI and MALDI studies probably reveal an effect of discrimination of lower-EA species, whereas EI studies exhibit in higher degree of fragmentation and their results can be affected by unknown difference in volatilities between various components. In order to determine the necessary correction factors, further reference experiments with isolated individual substances and their mixtures of given composition are required. TSI and EC techniques, although having low analytical value, were found to provide, together with the EI technique, important information on the electronic properties of trifluoromethylated species, which helps to understand the general trends in their ionizational behavior.

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