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Selective formation of $C_{60}F_{18}(g)$ and $C_{60}F_{36}(g)$ by reaction of [60]fullerene with molecular fluorine

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Abstract

Fluorination of $C_{60}(s)$, $C_{60}(s)$–$MnF_2(s)$, $C_{60}(s)$–$NiF_2(s)$ and $C_{60}(s)$–$MnF_3(s)$ mixtures has been studied by Knudsen cell mass spectrometry with admission of molecular fluorine. The fluorination is selective when fullerene reacts with the fluorine chemisorbed on the MnF_2 surface. When the MnF_2 content in the initial mixture is at least 90 mol% both $C_{60}F_{18}$ and $C_{60}F_{36}$ are selectively formed. Under certain conditions, mixtures predominantly containing one of three compounds $C_{60}F_{38}$, $C_{60}F_{40}$, and $C_{60}F_{42}$ can be obtained. A consecutive change of the main fluorination products ($C_{60}F_{18}$ and $C_{60}F_{36}$) takes place at constant temperature (720 K) and on fluorine admission. A quantitative explanation of this fact is given. Selective fluorination of $C_{60}(s)$ by molecular fluorine is compared with solid-phase fluorination using $MnF_3(s)$ as a fluorinating agent. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Selective formation; Fluorofullerenes; Molecular fluorine; Fluorination; Manganese fluorides

1. Introduction

Data on the synthesis of fluorofullerenes by the reaction of C_{60} with molecular fluorine are surveyed in [1]. In [2–6], the fluorination was carried out at F_2 pressures (0.1–5 atm), temperatures and times varying over a wide range. Mixtures of fluorinated fullerenes from $C_{60}F_2$ to $C_{60}F_{48}$ were obtained. In [7], the mixture containing more than 70% of $C_{60}F_{48}$ resulted from a two-stage synthesis. Authors of [8] developed the conditions to obtain a mixture containing ca. 95 mol% of $C_{60}F_{48}$.

Further progress in the selective synthesis of fluorofullerenes was associated with solid-phase fluorination in which transition-metal fluorides were used as fluorinating agents [9]. This method consists of the sublimation of reaction products followed by their condensation in a cold area. A mass spectrometer was used to determine optimal conditions for the selective synthesis that allows continuous control of the composition of the gas phase in a reactor (effusion chamber). It was established that C_{60} reacts with MnF_3 at $T = 590-650$ K to yield almost only the single gaseous product $C_{60}F_{36}$ [10], while the reaction between C_{60} and K_2PtF_6 at 720 K gives rise to $C_{60}F_{18}$ [11]. The latter reaction has provided the basis for a number of publications on the chromatographic separation of the products. For example, admixtures of $C_{60}F_2$, $C_{60}F_4O$, $C_{60}F_6O$, $C_{60}F_8O$ and $C_{60}F_{16}$ were isolated chromatographically upon the synthesis of $C_{60}F_{18}$ [11]. The ^{19}F NMR spectra of these compounds (including $C_{60}F_{18}$) were measured, and the positions of fluorine atoms in the carbon skeleton were determined [12]. When KF–MnF_3 was used as the fluorinating agent, a sample with approximately 10% of $C_{60}F_{20}$ was prepared. After chromatographic separation, pure $C_{60}F_{20}$ was obtained, and the positions of the fluorine atoms were determined from the ^{19}F NMR data [13]. Kinetics of the solid-phase fluorination accompanied by the sublimation of volatile products and the selectivity conditions are reported in [14].

While advances of the solid-phase synthesis of specific fluorofullerenes are evident, the conditions for selective syntheses of these compounds by fluorination of [60]fullerene by molecular fluorine still remain to be clarified. In connection with this, the possibilities of selective formation of fluorides via the reaction between C_{60} and F_2 are focused in this report. It is clear that the search for the conditions of selective fluorination implies variation of those parameters most influencing the composition of the gaseous products: these are the temperature and the composition of the system.

In this work, the fluorination of C_{60} by molecular fluorine in the presence of $MnF_2(s)$ and $NiF_2(s)$ was studied, the

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fluorination products being continuously controlled in a mass spectrometer. Moreover, the C_{60}(s)–F_2 and C_{60}(s)–MnF_3(s)–F_2 systems were compared.

2. Experimental

The details of the measurements were described elsewhere [15]. The set-up consisted of a reactor for the synthesis of fluorofullerenes combined with a magnetic sector MI-1201 mass spectrometer for the gas phase analysis. A nickel tube for fluorine admission was attached to a reactor (Ni effusion cell: $l = 10$ mm, $d = 6$ mm). Molecular fluorine entered the reactor through an orifice ($d = 0.15$ mm). Gas phase species escaped through the effusion orifice of the reactor and passed into the ion source of the mass spectrometer. The positive ions formed by an electron impact ($U = 60-70$ eV) were accelerated to 2 keV, mass analysed in the magnetic field, passed onto the collector and were recorded by means of an electrometer ($R = 10^{12}\ \Omega$). A movable shutter was installed in order to distinguish between ions coming from the background gases and those originating from effusing molecules. Temperature was measured with a Pt–Pt/Rh (10%) thermocouple attached to the reactor.

A finely crystalline sample of C_{60} (99.9% pure according to HPLC analysis) with particle size ~0.1 mm was used. The samples of MnF_2, NiF_2 and MnF_3 (>99% pure) were purchased from Aldrich. Mixtures of C_{60} with fluorides were carefully triturated. In all experimental runs, the amount of [60]fullerene in the initial mixture was equal to 0.03 g. Fluorination was carried out using 98% pure molecular fluorine (<0.2% O_2).

The temperature of fluorination was established before the beginning of the fluorine admission. The level of fluorine inlet was equal for all the experimental runs and corresponded to $p(F_2) \sim 2 \times 10^{-4}$ atm partial pressure in the empty reactor (in the absence of the fluorination reaction). Parameters of the experimental set-up were kept constant during each experimental run. The gas phase was mass spectrometrically analysed until the complete consumption of C_{60}.

Measured ion currents and partial pressures of the gas phase components [16] are related by equation:

$$P_j = k(Q_j)^{-1} \sum (I_{ij})\, T \quad (1)$$

where P_j is the partial pressure of component j; k the sensitivity constant; Q_j the electron impact ionisation cross-section of molecule j; $\Sigma(I_{ij})$ the total ion current of ions i formed from molecule j and T is the temperature. The individual mass spectra of C_{60}, $C_{60}F_{18}$, $C_{60}F_{36}$, $C_{60}F_{48}$ were taken from [10,17,18]. To calculate the saturated vapour pressure of C_{60}, data reported in [19] were used.

The experiments were finished after the C_{60} was consumed. A solid residue remaining after the experiment was analysed by X-ray diffraction. In some experiments, the fluorination was intentionally interrupted to analyse the solid phase. The X-ray diffraction patterns were identified using the database published in [20].

3. Results and discussion

3.1. Influence of nickel and manganese difluorides on the composition of the gaseous products

It was established in a series of experiments without fluorine admission that the C_{60} partial pressures ($p(C_{60})$) for the C_{60}(s)–MF_2 (97 mol%) (M = Ni or Mn) systems are equal to the saturated vapour pressure of C_{60} ($p^0(C_{60})$), i.e. the activities of C_{60} in these systems are close to 1. Relative concentrations of gaseous species obtained for a set of samples at 720 K are presented in Fig. 1a–e. In the C_{60}(s)–F_2 system, C_{60} was found to be the main component in the gas phase. The gaseous mixture contained also a wide variety of fluorinated derivatives $C_{60}F_{2n}$ ($n = 1-18$) (Fig. 1a). In the C_{60}(s)–NiF_2(97 mol%)–F_2 system, the C_{60} concentration remained high and approximately equal to the concentration of the main fluorinated product $C_{60}F_{18}$ (31 mol%) (Fig. 1b). In the C_{60}(s)–MnF_2 (50 mol%)–F_2 system, noticeable enrichment of the mixture with $C_{60}F_{18}$ was observed (Fig. 1c). Subsequent increase in MnF_2(s) content (90 and 97 mol%) leads to the selective fluorination. Thus, MnF_2(s) is the most effective species for the selective fluorination of C_{60} by molecular fluorine.

The time dependences of partial pressures of main gaseous components in the C_{60}(s)–MnF_2 (90 and 97 mol%)–F_2 systems are shown in Figs. 3 and 4. Data for the C_{60}(s)–F_2 system are also presented for comparison in Fig. 2. As is seen from Fig. 2, the ratio between the partial pressures of volatile products does not noticeably change during the fluorination of pure C_{60}. The same is true for the C_{60}(s)–NiF_2(s)–F_2 and C_{60}(s)–MnF_2 (50 mol%)–F_2 systems. It should be pointed out that marked decrease of the C_{60} content in the gas phase was not observed in any of these cases.

On the contrary, the gas phase composition in the C_{60}(s)–MnF_2 (97 mol%)–F_2 system was markedly changed (Fig. 3). Thus, immediately after the beginning of the fluorine admission, $p(C_{60})$ started to decrease and reached the sensitivity limit of the instrument (1×10^{-8} atm) within 2.5 h; thereafter, C_{60} was not detected in the gas phase. As for the products of fluorination, $C_{60}F_{18}$ was the main component of the gas phase for 8 h. thereafter, $C_{60}F_{18}$ and $C_{60}F_{36}$ concentrations stayed commensurable over ~2 h. (a transition period), while $C_{60}F_{36}$ became the dominant component for next ~14 h. Thus, the conditions of selective fluorination are accomplished in sequence for two specific fluorofullerenes in the same system. Similar selectivity was observed in the system with 90 mol% MnF_2 (Fig. 4), where the partial pressure of C_{60} decreases down to the sensitivity limit within 7.5 h.

In a reference experiment (97 mol% MnF_2 in the initial sample), fluorination was interrupted when $C_{60}F_{36}$ became

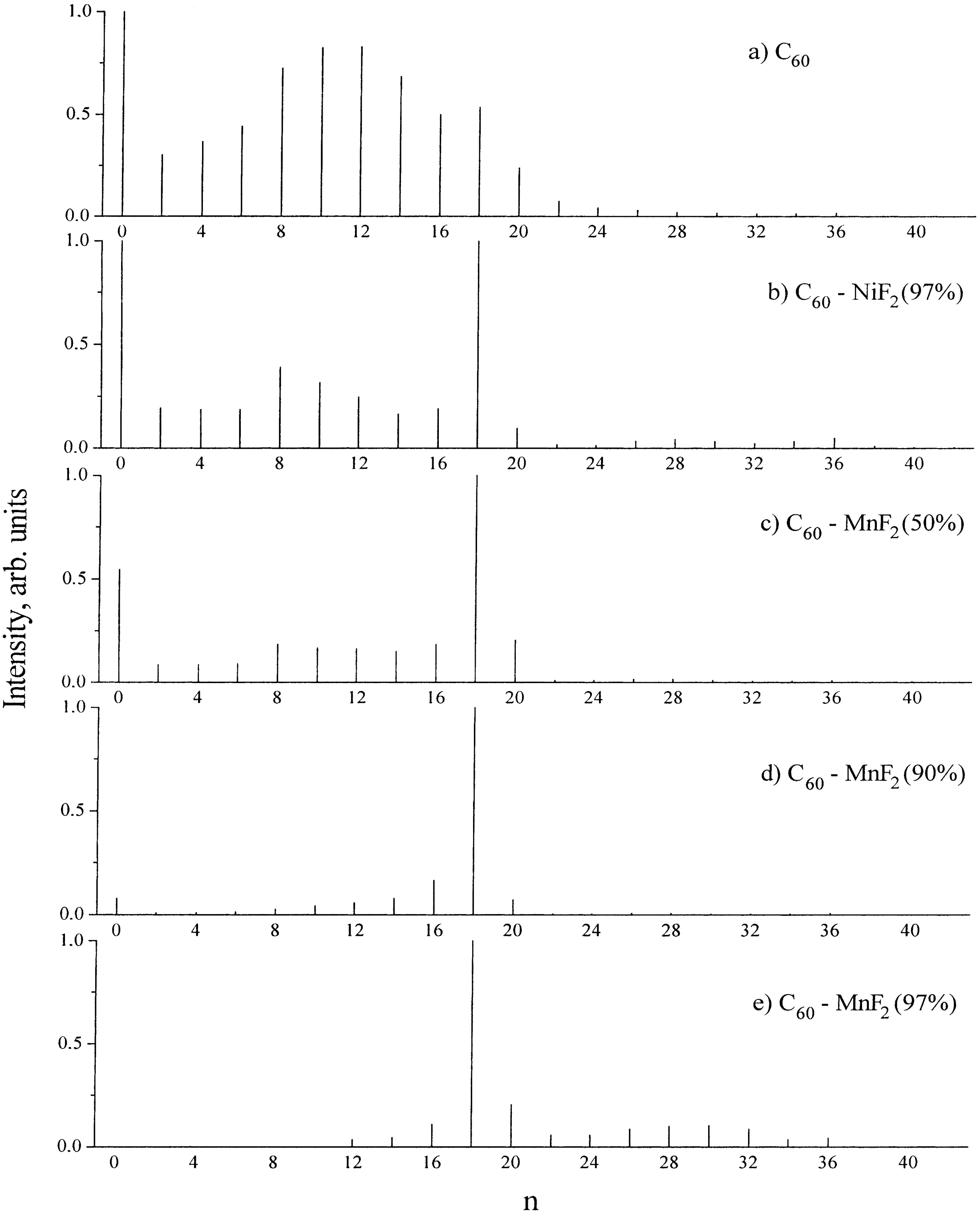

Fig. 1. Relative concentrations of gaseous species at $T = 720$ K. Time of fluorination ~5.5 h; (*n*: number of fluorine atoms in the molecule).

the main component. A residue extracted from the reactor consisted of MnF_2(s); therefore, the conclusion can be drawn that $C_{60}F_{18}$ was formed in the absence of Mn_2F_5(s) and MnF_3(s) which are formed on fluorination of MnF_2(s). The residue remaining in the reactor after the end of fluorination contained MnF_2, Mn_2F_5 and MnF_3. However, it should be noted that selectivity was not associated with fluorination by Mn_2F_5(s) and MnF_3(s), since the production of fluorofullerenes ceases immediately after switching off the fluorine admission. These experimental facts show that C_{60} fluorides were not accumulated in the solid phase, and Mn_2F_5(s) and MnF_3(s) were formed in the zones where fullerene was not directly contacted with MnF_2(s).

Thus, our experimental data demonstrate that [60]fullerene can be selectively fluorinated by molecular fluorine if manganese difluoride is mixed with C_{60}(s), its content in the initial mixture being not less than 90%. It appears to be possible to select the fluorination conditions (temperature and time) yielding $C_{60}F_{18}$ or $C_{60}F_{36}$ as predominant products.

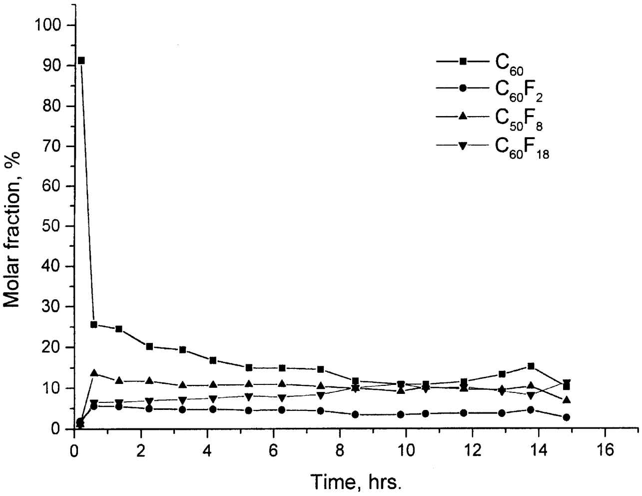

Fig. 2. Time dependence of molar fraction of $C_{60}F_2$, $C_{60}F_8$, $C_{60}F_{18}$ gaseous species. System: C_{60}(s)–F_2, $T = 720$ K.

3.2. A consecutive change of the main fluorination product at constant temperature and fluorine admission

It is evident that both fluorine and MnF_2(s) participate in the fluorination in the C_{60}(s)–MnF_2 (90 and 97 mol%)–F_2 systems. Manganese difluoride seems to change the state of fluorine in the reaction system. Part of the F_2 molecules appear to be adsorbed on the support surface; the fluorine chemisorbed on the manganese difluoride surface ($F_2\{MnF_2\}$) reacts with C_{60}, thus, providing the selectivity of the fluorination process. Due to fluorine attached to the MnF_2 surface, low-speed stages occur in a series of consecutive reactions of fluorination of C_{60} [21]. In the case being considered, the fluorination of $C_{60}F_{18}$ and $C_{60}F_{36}$ are low-speed stages; therefore, $C_{60}F_{18}$ and $C_{60}F_{36}$ are those products which are selectively formed under varying experimental conditions.

The partial pressure of C_{60}, total pressure of fluorination products ($\Sigma\{P(C_{60}F_n)\}$), average numbers of fluorine atoms in the molecule (n_a) and average numbers of C_{60} molecules in the outcoming flux (n_m), at 0.25τ; 0.5τ; 0.75τ (τ is the time of fluorination) at $T = 720$ K are given in Table 1.

Table 1 shows that total pressures of $C_{60}F_{2n}$ in the C_{60}(s)–F_2, C_{60}(s)–MnF_2 (90 mol%)–F_2 and C_{60}(s)–MnF_2 (97 mol%)–F_2 systems are similar at constant temperature and fluorine admission. At the same time, significant differences should be noted. In the C_{60}–F_2 system, the average number of the fluorine atoms per C_{60} molecule varies from 11.5 to 12.7 within the period of about 15 h. In this case, 30 mg of C_{60} were consumed at the fluorine pressure of $\sim 2\times10^{-4}$ atm (Fig. 2), while no predominant products were observed. On fluorination of the C_{60}–MnF_2 system containing the same amount of C_{60} and an excess of MnF_2 (90 and 97 mol%), $C_{60}F_{18}$ was found to be the main product during 9(8) h; thereafter, n_a value changed from 17(21) to 35(36.5) over $\sim$2 h, i.e. $C_{60}F_{36}$ turned to be the main product; its dominant concentration in the gas phase being observed for a further 6(14) h. Thus, the experimental data showed that two steady states are consecutively realised in the reaction zone upon isothermal fluorination of a C_{60}–MnF_2 mixture by molecular fluorine, which are distinguished only by the average number of F atoms per one C_{60} molecule in the outcoming products. The transition from one state to another is presumably due to the increase of the concentration of

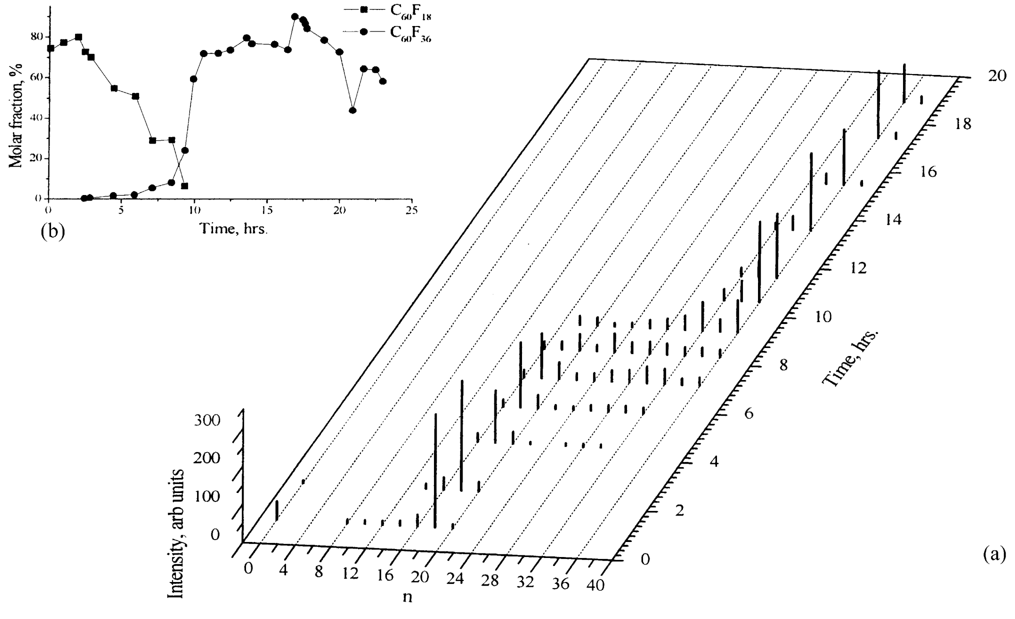

Fig. 3. (a) Time dependence of relative concentration of $C_{60}F_{2n}$ gaseous species. System: C_{60}–MnF_2 (97 mol%)–F_2, $T = 720$ K; (b) molar fraction of $C_{60}F_{18}$ and $C_{60}F_{36}$ as a function of time.

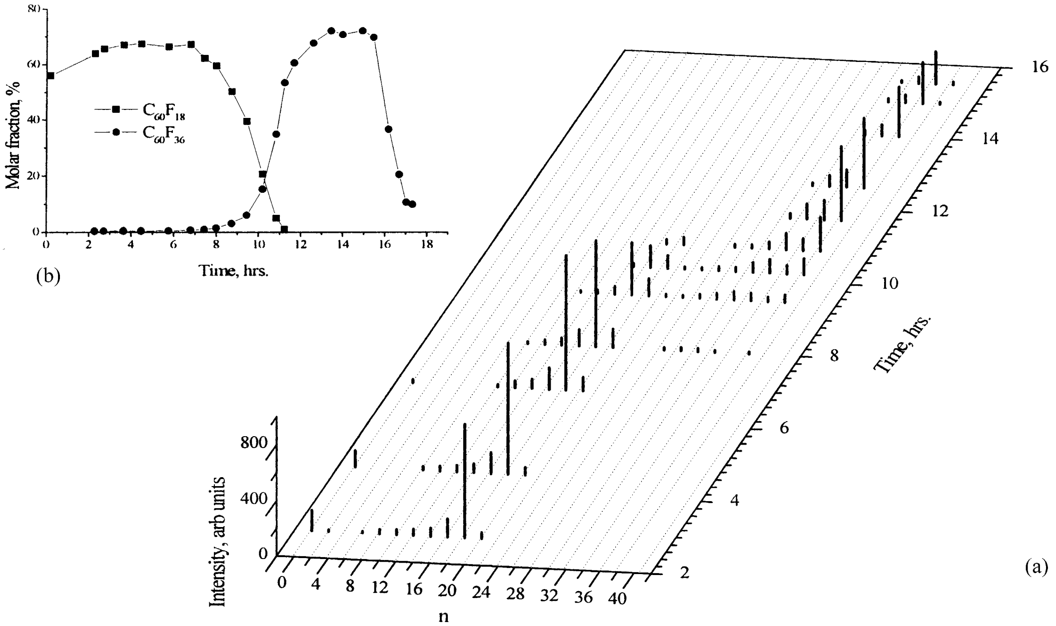

Fig. 4. (a) Time dependence of relative concentration of $C_{60}F_{2n}$ gaseous species. System: C_{60}–MnF_2 (90 mol%)–F_2, $T = 720$ K; (b) molar fraction of $C_{60}F_{18}$ and $C_{60}F_{36}$ as a function of time.

chemisorbed fluorine that can occur at the constant fluorine admission.

Since the increase in the fluorine concentration should result in an increasing rate of fluorination of C_{60}, but the experimental data indicated the constancy or even small decrease of the number of C_{60} molecules in the outcoming flux (Table 1), it is suggested that the steady concentration of C_{60} in the reaction zone should be proportionally decreased.

The decrease in the steady concentration of C_{60} (and the corresponding increase in the concentration gradient) is not accompanied by an increase in the C_{60} flux toward the reaction zone. The factors limiting the intensities of this flux are likely associated with the rate of formation of defects and the activation energy of the transition of C_{60} molecules from the bulk to the surface of the crystal.

In the case under consideration, the topochemical reaction on the boundary of two phases (C_{60} and MnF_2) is determined by two incoming fluxes: fluorine flux and C_{60} flux. The deficit of both fluorine and C_{60} can occur in the reaction zone depending on the reaction conditions. When the fluorine is scarce, $C_{60}F_{18}$ is predominantly formed, while a deficit of C_{60} results in the formation of $C_{60}F_{36}$. In both cases, the C_{60} flux toward the reaction zone is the same, and only the fluorine flux changes.

As a rule, the activation energies of diffusion and sublimation are noticeably different which provides a means for a fine temperature control of the selectivity of fluorination. Owing to sublimation of the volatile products, the process can be stopped at an intermediate stage.

When the reactor temperature was decreased by 100–200 K, the rate of desorption decreases, and $C_{60}F_{18}$ does not sublime (according to recent data [22], $C_{60}F_{18}$ is less volatile than $C_{60}F_{36}$). At 615 K, a series of reactions proceeding on the support is not interrupted until $C_{60}F_{36}$ is formed; the concentration of this fluoride in the gaseous products reaches 80–90% within about 11 h. (Fig. 5). When the temperature decreases to 520 K, the desorption of $C_{60}F_{36}$ becomes slower, and fluorination proceeds without well-defined selectivity, though $C_{60}F_{36}$ was the predominant product during ~6 h. (Fig. 6).

Table 1
Partial pressure of C_{60}, total pressure of fluorofullerenes, average number of F atoms in $C_{60}F_n$ molecule and average numbers of C_{60} molecules in the outcoming flux at $T = 720$ K.

Composition	τ (h)	C_{60} partial pressure ($\times 10^6$) (atm)			Total pressure of fluorofullerenes ($\times 10^6$) (atm)			Average number of F atoms (n_a)			Average number of C_{60} molecules in the outcoming flux (n_m) relative units		
		0.25τ	0.5τ	0.75τ	0.25τ	0.5τ	0.75τ	0.25τ	0.5τ	0.75τ	0.25τ	0.5τ	0.75τ
C_{60}	14.83	0.52	0.26	0.18	2.16	1.52	1.40	11.5	12.1	12.7	100	70	65
MnF_2 (90 mol%)	17.32	0.17	0.01	–	1.92	1.54	1.01	17.0	20.3	35.0	100	80	53
MnF_2 (97 mol%)	22.93	–	–	–	2.65	1.82	1.56	21.0	35.3	36.3	100	69	59

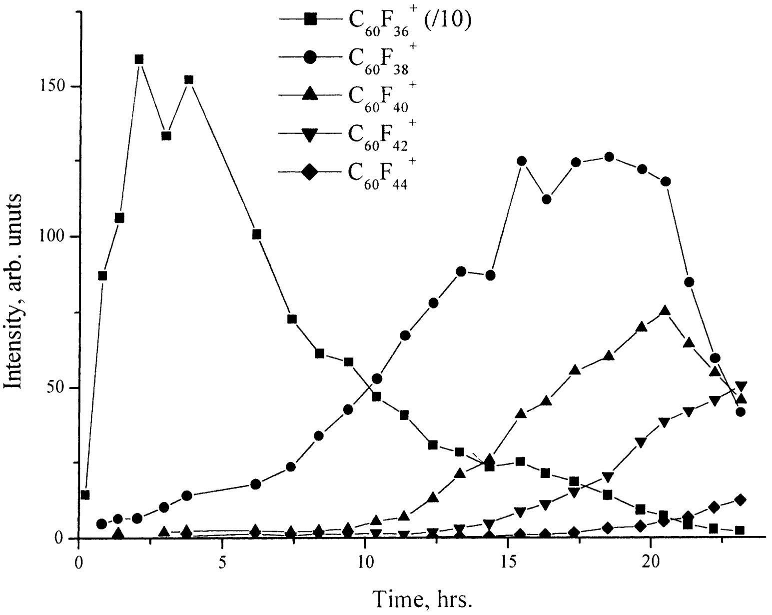

Fig. 5. Time dependence of relative intensities of molecular ions formed from main gaseous species. System: C_{60}–MnF_2 (97 mol%)–F_2, $T = 615$ K.

A decrease in $p(C_{60})$ at the beginning of fluorination is also a consequence of the increase in $F_2\{MnF_2\}$ fluorine concentration (Figs. 3 and 4). Unlike the pure $MnF_2(s)$ surface, the support surface with adsorbed fluorine can be considered as a getter for C_{60} molecules. After collisions of C_{60} molecules with the support, they react with $F_2\{MnF_2\}$ fluorine and sublime only in the form of fluorofullerenes. As a result, the $p(C_{60})$ value falls outside the sensitivity limit of the instrument, although the C_{60} phase is present in the system.

3.3. A comparison between selective fluorination of $C_{60}(s)$ by molecular fluorine and solid- phase fluorination.

It was mentioned in the introduction that advances in the synthesis of specific fluorofullerenes have been made using solid-phase fluorination. Since similar compositions of the gaseous products were obtained in the $C_{60}(s)$–$MnF_3(s)$ ($T = 590-650$ K) and $C_{60}(s)$–MnF_2 (>90 mol%)–F_2 ($T = 615$ K) systems, it would be reasonable to suggest that similar stages resulting in the selective formation of

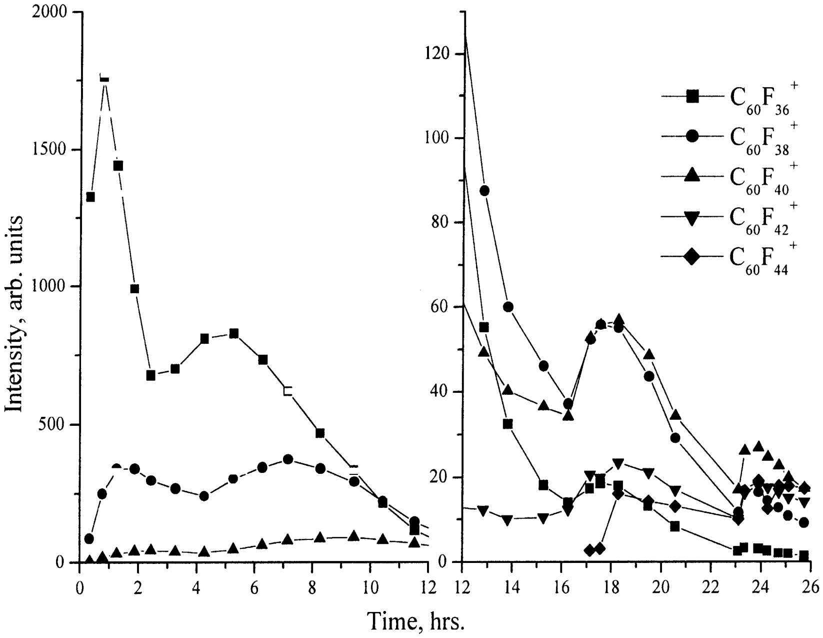

Fig. 6. Time dependence of relative intensities of molecular ions formed from main gaseous species. System: C_{60}–MnF_2 (97 mol%)–F_2, $T = 520$ K.

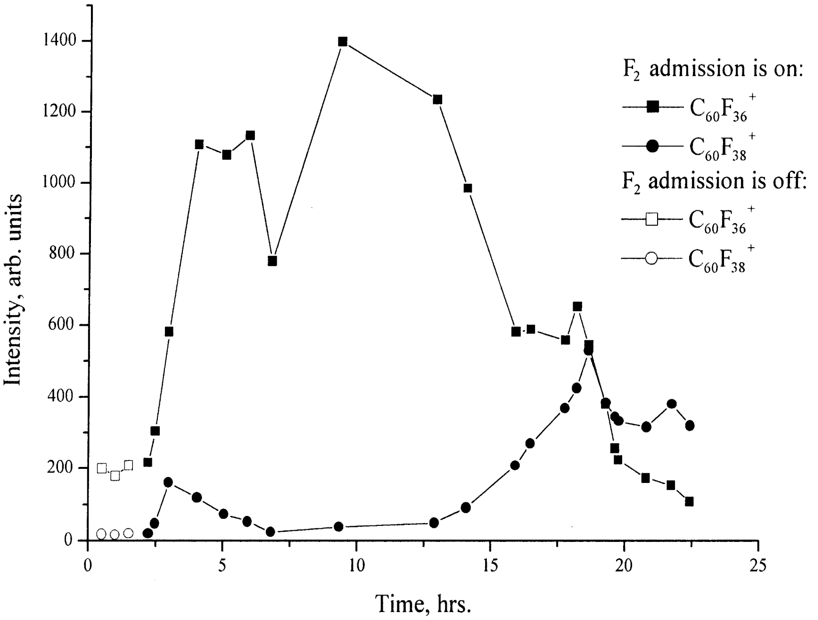

Fig. 7. Time dependence of relative intensities of molecular ions formed from main gaseous species. System: C_{60}–MnF_3 (97 mol%)–F_2, $T = 590$ K.

$C_{60}F_{36}$ are characteristic of the corresponding fluorination routes. For comparison, the C_{60}(s)–MnF_3(s) mixture was fluorinated by F_2. The data obtained are presented in Fig. 7. It was established that the solid-phase fluorination proceeded at the initial stage, when the fluorine admission was off ($T = 590$ K). The results are in good agreement with the literature data [9] $C_{60}F_{36}$ and $C_{60}F_{38}$ were the most abundant species. The ratio of partial pressures of these fluorides found to be 11 and remained constant during 2.5 h. Then the fluorine inlet was switched on, and a new steady state was established, with the partial pressure of main products increasing by a factor of 5. At the same time, the $p(C_{60}F_{36})/p(C_{60}F_{38})$ ratio increased from 11 to 30 (see Fig. 7), thus, indicating an improvement of the selectivity. After 10.5 h, a progressive increase in the relative content of highly fluorinated products was detected. This is due to the fluorine, which combined weakly with the support after the absorption centers on the MnF_2 surface were occupied.

In the solid-phase reaction, fluorine admission results in a five-fold increase of the total pressure of fluorinated products, with some improvement of the selectivity. This can be explained by the formation of a large number of the absorption centers upon the decomposition of MnF_3 in the course of fluorination which is likely accompanied by the formation of finely dispersed phases (MnF_2, Mn_2F_5), as well as MnF_{3-x} with comparatively low fluorine content. Therefore, the concentration of the chemisorbed fluorine in the reaction zone can be noticeably increased with the fluorine admission. As the fluorine admission was continued, the number of free adsorption centers on the reaction surface decreases and new chemisorption states are characterised by a lower bond energy between F_2 molecules and the support surface. Under these condition, fluorination proceeds further yielding highly fluorinated products ($C_{60}F_{38}$, $C_{60}F_{40}$, $C_{60}F_{42}$, $C_{60}F_{44}$).

4. Conclusion

The results of this work, together with the literature data on the solid-phase synthesis with separation of gaseous products, unambiguously indicate that the fluorination is selective when fullerene reacts with the fluorine chemisorbed on the MnF_2 surface.

Upon fluorination of the C_{60}–MnF_2 mixtures by molecular fluorine, the temperature of the system and the pressure of fluorine admitted in the reactor can be changed independently which enables a finer control of the conditions for selective formation of most stable compounds in the series of fluorofullerenes $C_{60}F_{2x}$. Mixtures enriched with one of the less stable members of the series can be also prepared. When MnF_2 is used as a matrix for C_{60}, both $C_{60}F_{18}$ and $C_{60}F_{36}$ are selectively formed; under certain conditions, a mixture predominantly containing one of three compounds $C_{60}F_{38}$, $C_{60}F_{40}$, and $C_{60}F_{42}$ can be obtained. It seems likely that using the fluorides of metals in low oxidation states as a matrix for C_{60} and independent regulation of the temperature and fluorine pressure will facilitate obtaining mixtures of fluorofullerenes highly enriched with the compound of interest.

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