



Photochemistry of perfluoroacetyl fluoride

Kinetics of the reaction between CF_3 and FCO radicals

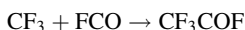
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Abstract

The photolysis at 254 nm of perfluoroacetyl fluoride (CF_3COF) in the gas phase yields C_2F_6 , CF_2O and CO as products. An excess of O_2 added to the system leads to the formation of CF_2O and CO_2 as main products. If, instead of O_2 , $\text{c-C}_6\text{H}_{12}$ were added, the main products are CF_3H and HC(O)F . In both cases a faster rate of CF_3COF disappearance is observed. The measurement of the quantum yield for CF_3COF consumption as well as for products formation are: for CF_3COF alone, $\phi_{\text{CF}_3\text{COF}} = 2\phi_{\text{C}_2\text{F}_6} = 2\phi_{\text{CF}_2\text{O}} = 0.43 \pm 0.05$; in the presence of $\text{c-C}_6\text{H}_{12}$ $\phi_{\text{CF}_3\text{COF}} = \phi_{\text{CF}_3\text{H}} = 1.02 \pm 0.02$ and in the presence of O_2 , $\phi_{\text{CF}_3\text{COF}} = 1.02 \pm 0.05$. The low quantum yield observed for pure CF_3COF suggests the occurrence of the recombination reaction:



for which we calculated the rate constant as $(6.9 \pm 0.8) \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$. © 1999 Elsevier Science S.A. All rights reserved.

1. Introduction

The tropospheric reactions of HCFCs, HFCs and their degradation products have received much attention recently. CF_2O , C(O)FCl , HC(O)F , CF_3COX ($\text{X} = \text{Cl}, \text{F}$) are some of these degradation products. In particular, CF_3COF could form by the degradation of CHFClCF_3 (HCFC-124) and CH_2FCF_3 (HFC-134a) [1]. In the troposphere, the CF_3COF formation yields from HFC-134a increases with increasing altitude, with mainly formation of HC(O)F and the CF_3 radical products at the Earth's surface and mainly formation of CF_3COF at the tropopause [2]. The photolysis of CF_3COF is thus of importance and several authors have reported about this some years ago. Aslanidi et al. [3] investigated the CF_3COF dissociation channels in the pressure region from 0.2 to 4 Torr under pulsed CO_2 laser radiation and identified the dissociation products as CF_2O , C_2F_6 and CF_4 .

Weibel et al. [4] studied the photolysis in the pressure range 6.1–250.7 Torr using light of 240 and 232 nm and observed the formation of C_2F_6 , CO and CF_2O . They also determined that the quantum yield of its decomposition ($\phi = 0.4$) is independent of the total pressure and light

intensity, and interpreted the results in terms of C–C bond cleavage in the lowest singlet state of the electronically excited molecule. The rupture of the molecule proposed by Weibel et al. implied the formation of CF_3 and FCO radicals.

Theoretical studies carried out by Jubert et al. [5] and Francisco et al. [6] gave three possible routes for the dissociation on the ground state potential energy surface. The lowest in energy corresponded to the 1,2-fluorine shift, the next higher in energy to the 1,2-CO elimination and the highest to the C–C bond cleavage giving CF_3 and FCO radicals.

We reinvestigated, as part of our studies on the photochemistry of fluorinated compounds, the photolysis of CF_3COF at different wavelengths and measured at 240 nm the quantum yield of photolysis for CF_3COF alone, and in the presence of either $\text{c-C}_6\text{H}_{12}$, O_2 or $(\text{FCO})_2$. Our observations led us to conclude that the primary step is the rupture of the C–C bond and that the low quantum yield obtained by Weibel et al. [4] is a consequence of the back reaction between these two radicals to re-form CF_3COF . The difference in the values of the quantum yields when the photolysis is carried out in the presence of CF_3 trapping molecules allowed us to determine the rate constant for the reaction between CF_3 and FCO radicals.

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2. Experimental details

2.1. Materials

Commercially available samples of CF₃COF (PCR Research Chemicals Inc.) were trap-to-trap distilled in vacuum before use and the purity was checked by UV and IR spectroscopy. Oxygen was condensed by flowing O₂ at atmospheric pressure through a trap immersed in liquid air. It was then pumped under vacuum several times and transferred to a glass bulb whilst the trap was still immersed in liquid air. Oxalylfluoride (FC(O)C(O)F) was synthesized by fluorination of oxalylchloride with NaF in sulfolane as solvent and purified by trap-to-trap distillation [7]. CO was passed through a trap maintained at liquid air temperature and stored in a glass bulb.

2.2. Procedure

Reactants and products were manipulated in a conventional high-vacuum system. The photolysis were carried out using three different approaches, that have been discussed elsewhere [8]. They consist briefly in (a) a full arc 200 W high pressure Hg lamp and detection through IR spectroscopy at infinite times; (b) the same photolytic lamp coupled to a monochromator and (c) low pressure Hg lamps surrounding a quartz cell fitted with KCl windows which was located in the optical path of a Fourier transform IR (FTIR) spectrometer which was used to follow the evolution of the reaction with time.

The amount of light for each photolysis carried out with Setup 2, was measured using actinometry with a potassium ferrioxalate actinometer. A short (1 cm optical path) quartz cell containing the actinometer was placed in front of the 10 cm photolysis cell to measure the incident photon flux. The same procedure was repeated with the actinometric cell placed after the evacuated photolytic cell and then, the two measurements were averaged. Once the photon flux is known, the quantum yields of each product (ϕ_i) were obtained through

$$\phi_i = \frac{n_i}{ft}$$

where n_i is the number of moles of product, f is the photon

flux and t is the photolysis time. A very good description of the actinometric technique is given elsewhere [9].

3. Results and discussion

3.1. Products and quantum yields

The comparison between the rate of photolysis of CF₃COF alone and mixtures of CF₃COF with either O₂, *c*-C₆H₁₂, (FCO)₂ or CO was performed initially using Setup (a). When CF₃COF alone was photolyzed, the products formed were C₂F₆, CF₂O and CO, in agreement with the results of Weibel et al. [4]. The absolute quantities corresponding to the products found are summarized in Table 1. These products could be explained taking into account that the C–C bond breaking gives CF₃ and FCO radicals:



In order to determine the products in the case of the mixture with O₂, a typical sample consisting of 2 Torr of CF₃COF was photolysed in the presence of 2.8 Torr of O₂ for 20 min. After that, the sample was trapped in liquid air and the non-condensable fraction was transferred, using a Toepler pump, to an IR cell. This fraction did not show the appearance of any infrared active product. The condensable fraction was then transferred to the same cell and overall analysis of the spectrum of the whole sample reveals undoubtedly the formation of CF₂O (774 cm⁻¹) and CO₂ (667 cm⁻¹). To identify the other products the sample was distilled between –100°C and liquid air temperature. The fraction retained at –100°C showed solely bisfluoroformyl peroxide (FCOO)₂, whilst the other fraction showed the non-reacted CF₃COF, bistrifluoromethyl trioxide CF₃O₃CF₃ and CF₃O₂COF, both assessed through their pure infrared spectra [10].

After identifying all the products, their quantification was carried out using the following subtraction sequence (whose resulting spectra are shown in Fig. 1): the reactant, CF₃COF, was first subtracted using as reference, the band at 1334 cm⁻¹ which does not interfere with any other band (trace b), then CF₂O was subtracted looking at the 774 cm⁻¹ band (trace c); next the CF₃O₂COF was carefully subtracted looking at the 1917 cm⁻¹ band and keeping in mind that

Table 1
Experimental results of CF₃COF photolysis in the presence of different reactive gases

Gas added (torr)	Products (moles × 10 ⁻⁷)						
	CF ₂ O	C ₂ F ₆	CF ₃ O ₃ CF ₃	CF ₃ O ₂ COF	CF ₃ H	(FCOO) ₂	CF ₃ COF consumed
None	–	2.9	2.3	–	–	–	4.8
O ₂	2.8	5.7	–	2.6	3.0	0.8	14
<i>c</i> -C ₆ H ₁₂	90	–	–	–	13	–	13
(FCO) ₂	2	67	1.2	–	–	–	1.8
CO	0.5	2.8	2.3	–	–	–	4.6

$p(\text{CF}_3\text{COF}) = 2.0$ Torr; $T = 298$ K; light source: full arc lamp; $t_{\text{tot}} = 20$ min.

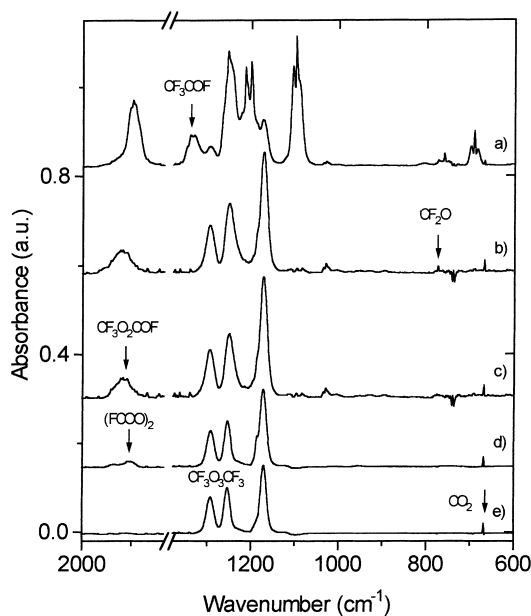
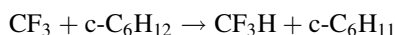


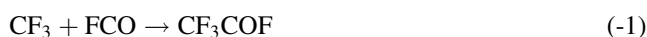
Fig. 1. IR spectra obtained for the photolysis of CF_3COF (2 Torr) in the presence of O_2 (2.8 torr) showing, from top to bottom, the results after: (a) 20 min of photolysis. The intensity has been reduced 3 times. (b) CF_3COF subtraction. (c) CF_2O subtraction. (d) $\text{CF}_3\text{O}_2\text{COF}$ subtraction. (e) $(\text{FCOO})_2$ subtraction. Note that the resultant spectrum shows only $\text{CF}_3\text{O}_3\text{CF}_3$ and CO_2 bands.

there is still a contribution at 1900 cm^{-1} from $(\text{FCOO})_2$. This last substance was finally subtracted (trace e) to show only the spectra of $\text{CF}_3\text{O}_3\text{CF}_3$ and CO_2 . The subtraction factor obtained for each species was a direct indication of the concentration since we had a calibration curve for each pure substance. The reactions responsible for the formation of these products are typical when CF_3 and FCO radicals react with O_2 [11–14].

The experiments carried out in the presence of *c*- C_6H_{12} gave CF_3H as the only product coming from the CF_3 fragments. The procedure followed to get the quantification was slightly different when O_2 is added since after removing the non-condensable fraction, a -40°C bath was used to retain all the *c*- C_6H_{12} allowing the volatile fraction to be analysed by IR. The subtraction sequence was also simpler since it was only needed to subtract the non-reacted CF_3COF to get pure CF_3H . Note, Table 1, that the amount of CF_3H formed is the same as the CF_3COF disappeared and that CF_2O or C_2F_6 , if formed, were below our detection limit. This indicates that all the CF_3 radicals formed reacted with the *c*- C_6H_{12} [4].



When comparing the first three rows of Table 1, it can be seen that the amount of CF_3COF reacted is greater in the presence of either O_2 or *c*- C_6H_{12} , for otherwise exactly the same experimental conditions. This strongly suggests that the CF_3 and FCO radicals react among them in a recombination reaction when there is no gas added.



To check this possibility we carried out a new photolysis of CF_3COF in the presence of a source of FCO radicals such as oxalyl fluoride $(\text{FCO})_2$. As is shown in Table 1, the amount of C_2F_6 formed as well as the reactant consumed are smaller than when CF_3COF is photolysed alone. Since the photolysis of oxalyl fluoride also gives CO as a product [15], we tested directly if this species has any influence on the rate of disappearance of CF_3COF . The results shown in the last row of Table 1, prove that the amount of reactant consumed and C_2F_6 formed are the same that when CO is absent in the system.

To measure the quantum yields, the second experimental setup was used. The low intensity of the 240 nm line yielded by the lamp required photolysis times as long as 15 h. On account on these long times, we found necessary to check for the constancy of the light intensity, measuring actinometrically the photon flux every three hours. Table 2 summarized the experimental results.

As opposed to this low intensity setup, the photolysis carried out with the full arc lamp favored the reactions between secondary radicals leading to products as $\text{CF}_3\text{O}_2\text{COF}$ which amounts to as much as 3.0×10^{-7} moles. When looked for with the low intensity setup, it was absent. The same is true for $\text{CF}_3\text{O}_3\text{CF}_3$ whose quantum yield drops to approximately 4%.

The quantum yields obtained clearly show three things:

- They are the same, within the experimental error, when O_2 or *c*- C_6H_{12} are present in the system.
- They are equal to one. This means that the very first act of photon absorption leads to the rupture of the C–C bond.
- For CF_3COF alone, we do not measure the primary step but the overall quantum yield, because of the existence of reaction (2).

Nevertheless, our values agree with that of [5]. With their experiments Weibel et al. proposed a mechanism where, prior to the dissociation, the excited state of the molecule

Table 2
Quantum yields of stable products

Gas added	Pressure (Torr)	$\phi_{\text{CF}_2\text{O}}$	$\phi_{\text{C}_2\text{F}_6}$	$\phi_{\text{CF}_3\text{O}_3\text{CF}_3}$	$\phi_{\text{CF}_3\text{H}}$	$\phi_{\text{CF}_3\text{COF}}$
–	–	0.25	0.21	–	–	0.46 ^a
–	–	0.20	0.18	–	–	0.38 ^a
–	–	0.26	0.20	–	–	0.46 ^a
	0.5	0.89	–	0.04	–	0.97 ^b
O_2	3.7	0.85	–	0.08	–	1.01 ^b
	5.0	0.95	–	0.06	–	1.07 ^b
<i>c</i> - C_6H_{12}	70.0	–	–	–	1.04	1.04 ^c
	90.0	–	–	–	1.01	1.01 ^c

$p(\text{CF}_3\text{COF}) = 5.3\text{ Torr}$, $T = 298\text{ K}$, $\lambda = 240\text{ nm}$, $t_{\text{tot}} = 15\text{ h}$.

^a $\phi_{\text{CF}_3\text{COF}} = 2 \times \phi_{\text{CF}_2\text{O}} = 2 \times \phi_{\text{C}_2\text{F}_6}$.

^b $\phi_{\text{CF}_3\text{COF}} = \phi_{\text{CF}_2\text{O}} + 2 \times \phi_{\text{CF}_3\text{O}_3\text{CF}_3}$.

^c $\phi_{\text{CF}_3\text{COF}} = \phi_{\text{CF}_3\text{H}}$.

resulting from the absorption of the photon could take several pathways, the internal conversion to the vibrationally excited ground state being the main reason why the quantum yield was 0.4. When they tried with O_2 or $c\text{-C}_6\text{H}_{12}$ they did not realize that their mechanism was incorrect because they did not measure the quantum yields. Therefore, we can conclude that the mechanism involves reaction (1) as the only dissociation pathway. This, in turn disagrees with the theoretical results from Jubert et al. [5] and Francisco et al. [6]. Jubert et al. theorized about the photochemical decomposition and considered the cleavage of the C–F bond as the primary step, a reaction pathway that had been also postulated in the dissociation dynamics of CF_3COF by Francisco; but, at high enough energies so as not to be competitive. Francisco in turn, considered reaction (1) but suggested that there are two other more competitive reactions pathways in the ground state potential energy surface. The lowest leads to the formation of CF_2 and CF_2O . If this were the case, we should have measured C_2F_4 when CF_3COF was photolysed alone and, more important, a quantum yield of formation of CF_2O at least equal to one, and perhaps greater, when the photolysis were carried out in the presence of O_2 due to the reaction of the CF_2 radicals [16]. This reaction pathway, as well as the second pathway proposed by Francisco, can not account for the appearance of C_2F_6 .

3.2. Kinetics of the reaction between CF_3 and FCO radicals

In accordance with the previous discussion it is clear that the presence of an effective radical scavenger, able to trap radicals that participate in reaction (2), allows the determination of the photolysis rate of reaction (1). We use $c\text{-C}_6\text{H}_{12}$ because, in contrast to O_2 , it is a cleaner reaction with less products; and we adopted the third experimental setup which allowed us to take IR spectra at least every 60 s while continuously photolysing. Two photolytic experiments were performed with initial CF_3COF concentrations of 4.45 and 4.72×10^{16} molecule cm^{-3} in the presence of 90 Torr of $c\text{-C}_6\text{H}_{12}$. The duration of the photolysis time was 720 s, to maintain low conversions. A comparison of the variation of the CF_3COF concentration between photolysis carried out in the absence and in the presence of $c\text{-C}_6\text{H}_{12}$ is presented in Fig. 2. Note that the disappearance of CF_3COF is faster in the last case. From its slope (dotted line in Fig. 2), we obtain the photolysis rate of CF_3COF as $(8.2 \pm 0.2) \times 10^{-5} \text{ s}^{-1}$. The error corresponds to one standard deviation.

The reactions that follow the rupture of the C–C bond of the molecule when the photolysis is carried out in the absence of $c\text{-C}_6\text{H}_{12}$ are:

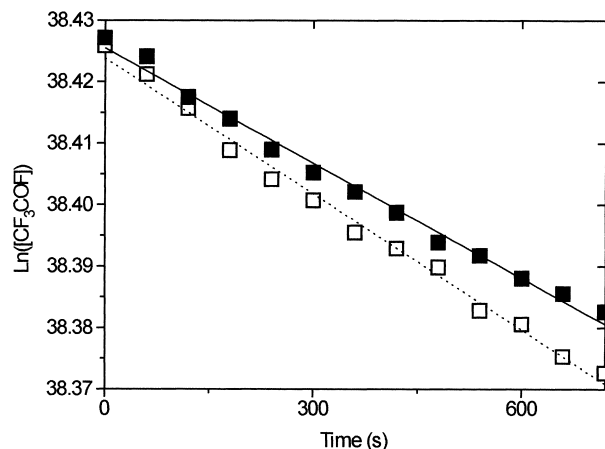


Fig. 2. Plot of $\ln[\text{CF}_3\text{COF}]$ vs. photolysis time. The full line corresponds to the least squares fitting to the experimental points for CF_3COF pure photolysis and the dotted line is the equivalent for the photolysis in presence of $c\text{-C}_6\text{H}_{12}$.

With this mechanism, the temporal variation of $[\text{CF}_3\text{COF}]$ is given by:

$$\frac{-d[\text{CF}_3\text{COF}]}{dt} = k_1[\text{CF}_3\text{COF}] - k_{-1}[\text{CF}_3][\text{FCO}] \quad (5)$$

Assuming steady-state conditions, and substituting into the proper equations, it is found that $[\text{CF}_3] = [\text{FCO}]$ and thus, the temporal variation of $[\text{CF}_3\text{COF}]$ is straightforwardly given by:

$$\ln[\text{CF}_3\text{COF}]_t = -k_{\text{obs}}t + \ln[\text{CF}_3\text{COF}]_0 \quad (6)$$

where k_{obs} (full line in Fig. 2) equals:

$$k_{\text{obs}} = \frac{2k_1(k_2k_3)^{1/2}}{k_{-1} + 2(k_2k_3)^{1/2}}$$

and since k_2 and k_3 are known from the literature, we can obtain k_{-1} .

A few words about the selection of k_2 and k_3 from the many values informed in the literature follow. Vathkin [17] has recently reported the recombination rate constant for CF_3 radicals and compiled all the previous measurements giving possible reasons for the discrepancies found since the high pressure ‘room temperature’ values span one order of magnitude. In particular, the value reported by Brown et al. [18] should be interpreted for an effective temperature of 1000 K. Vakhtin’s value of $(3.9 \pm 1.3) \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ is in good agreement with most of the literature results. There is also a very small dependence with the pressure for the room temperature rate constant, so that for our experimental conditions, we believe that the present value is suitable. For k_3 there are four values reported [12,15,19,20] but only the work of Behr et al. [20] was carried out at pressures comparable to these used in our experiments, so we took the value of $(4.9 \pm 2.0) \times 10^{-11} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$. Nevertheless, the discrepancies between the different values is much smaller than for k_2 .

Table 3
Overall rate constants k_{obs} obtained from the plots of $\ln[\text{CF}_3\text{COF}]$ vs. time (t)

Negative slopes $\times 10^5$ (s^{-1}) ^a		
Direct measurement ^b	$[\text{CF}_3\text{COF}]_0 - 2 \times [\text{CF}_2\text{O}]_t$ ^b	$[\text{CF}_3\text{COF}]_0 - 2 \times [\text{C}_2\text{F}_6]_t$ ^b
5.9	6.6	6.0
6.1	7.0	6.8
6.0	5.8	6.7
6.4	6.6	6.8

^a For the determination of k_{-1} .

^b Values listed were obtained either from “direct measurements”, from the subtraction $[\text{CF}_3\text{COF}]_0 - 2 \times [\text{CF}_2\text{O}]_t$ or from $[\text{CF}_3\text{COF}]_0 - 2 \times [\text{C}_2\text{F}_6]_t$.

Table 3 shows the k_{obs} obtained from the plots of $\ln[\text{CF}_3\text{COF}]$ vs. time for different experiments. The three values quoted for each run correspond to three different ways of obtaining the individual CF_3COF concentration as a function of time. They were the direct quantification of CF_3COF through the 1334 cm^{-1} band, the quantification of CF_2O at 1928 cm^{-1} and that of C_2F_6 at 1250 cm^{-1} , all of them after appropriate subtractions when needed. Thus, the three pseudo-first order rate constants yield three k_{-1} values: 6.7×10^{-12} , 7.2×10^{-12} and 8.7×10^{-12} all in units of $\text{cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$. These three values were checked for reliability by simulating the time variation of the concentration of all the species with the mechanism just described. Fig. 3 shows the results for two different experimental runs as well

as the fitting obtained for two different values of k_{-1} . As can be seen in Fig. 3(a), the fitting with $k_{-1} = 6.7 \times 10^{-12}$ is very good for CF_2O , C_2F_6 as well as for CF_3COF . When k_{-1} is changed to 8.7×10^{-12} (Fig. 3(b)) we can see that the fitting is still good for CF_3COF but it fails for CF_2O and C_2F_6 . In the case of the other experimental run, this lack of sensitivity is removed, for we can see the deviation of the fitting (Fig. 3(c)) for CF_3COF when changing the constant from 6.7 to $8.7 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ (Fig. 3(d)). It is also clear that the higher rate constant does not fit the products of the reaction either. Therefore, we disregarded the value of $8.7 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ and recommend for k_{-1} the value of $6.9 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ which is the mean value obtained through the quantification of the products. This value of the recombination rate constant also explains why, when CF_3COF is photolysed in the presence of oxalyl fluoride, the concentration of C_2F_6 drops so efficiently and the concentration of CF_3COF drops only to a third of that consumed when it is photolysed alone.

4. Conclusions

We were able to derive the recombination rate constant between CF_3 and FCO radicals. This could be carried out because the quantum yield of the primary step is equal to one and the radicals formed could be trapped to allow the determination of the decomposition rate constant.

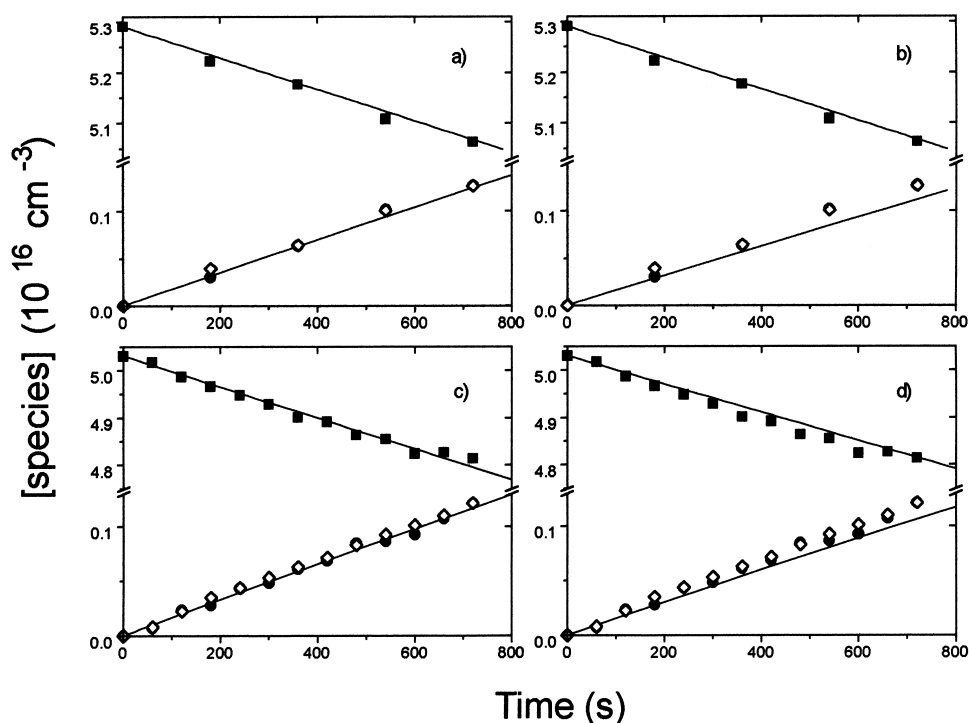


Fig. 3. Fitting to the actual concentration of species (\blacksquare , CF_3COF ; \bullet , CF_2O ; \diamond , C_2F_6) for two different experimental runs when the value assumed by k_{-1} in the simulation is $6.9 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ (Figs. (a) and (c)) or $8.7 \times 10^{-12} \text{ cm}^3 \text{ molecule}^{-1} \text{ s}^{-1}$ (Figs. (b) and (d)).

Acknowledgements

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