

Mechanism of dark decomposition of iodine donor in the active medium of a pulsed chemical oxygen–iodine laser

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Abstract. A scheme is proposed that describes the dark decomposition of iodide – the donor of iodine – and the relaxation of singlet oxygen in the chlorine-containing active medium of a pulsed chemical oxygen–iodine laser (COIL). For typical compositions of the active media of pulsed COILs utilising CH_3I molecules as iodine donors, a branching chain reaction of the CH_3I decomposition accompanied by the efficient dissipation of singlet oxygen is shown to develop even at the stage of filling the active volume. In the active media with CF_3I as the donor, a similar chain reaction is retarded due to the decay of CF_3 radicals upon recombination with oxygen. The validity of this mechanism is confirmed by a rather good agreement between the results of calculations and the available experimental data. The chain decomposition of alkyl iodides accompanied by an avalanche production of iodine atoms represents a new way of efficient chemical production of iodine for a COIL.

Keywords: oxygen–iodine laser, singlet oxygen, chain reaction.

1. Introduction

Over more than twenty years that have passed since the advent of a chemical oxygen–iodine laser in 1978 [1], considerable advances, both scientific and engineering, have been achieved in different areas of investigation of this system. Long-standing efforts of scientists from many countries have led to the development of cw lasers whose characteristics are highly competitive with those of lasers of other types and sometimes surpasses them (see, for instance, Ref. [2]). The pulsed and repetitively pulsed operating regimes of a COIL significantly extend its applications. Among them, in particular, are technological applications, the investigation of nonlinear effects, etc. Naturally, the objective in realising the pulsed mode is to attain a high output pulse power with retention of the efficiency and high energy characteristics inherent in cw COILs [2].

One of the most elaborated and attractive ways of realising the pulsed COIL operating mode is the technique

of pulsed (instantaneous) iodine production proposed in Ref. [3]. In this case, the active volume is filled in the absence of iodine (atomic and molecular), and the required concentration of iodine atoms is produced in the pulsed mode uniformly over the volume after its filling. This is accomplished by adding iodine-containing molecules, which do not react with singlet oxygen (SO) chemically, to the stream of SO with subsequent pulsed action on the medium resulting in the volume production of atomic iodine.

The pulsed COIL operating mode with an instantaneous production of iodine was first realised in Ref. [4]. Alkyl iodide (CH_3I , $\text{C}_3\text{H}_7\text{I}$, etc.) and perfluoroalkyl iodide (CF_3I , $\text{C}_3\text{F}_7\text{I}$, etc.) molecules were employed as donors of atomic iodine, while the pulsed photolysis of the above molecules was employed as the initiation technique. The photolysis of these iodides was extensively studied in connection with photodissociation iodine laser investigations (in particular, in our laboratory). The practical realisation of the pulsed COIL mode and subsequent efforts which followed this avenue of research demonstrated the promise of this mode for different scientific and technological applications. However, these investigations also revealed several scientific and technical problems emerging in the employment of different iodine donors.

The matter is that chlorine is not completely utilised in the SO generator and its remaining part enters the SO flow. In this case, the chlorine concentration may be as high as 10 % of the oxygen concentration. Studies of the effect of chlorine on the output energy of a pulsed COIL performed in Ref. [5] showed that the addition of a significant amount ($\sim 1.6 \times 10^{16} \text{ cm}^{-3}$) of chlorine to the SO and CF_3I flow resulted in a reduction of the output laser energy by only 20 %, whereas, when CH_3I molecules were used as donors, lasing was quenched at the chlorine concentration that was lower by nearly an order of magnitude ($\sim 2 \times 10^{15} \text{ cm}^{-3}$).

In the study of a high-pressure pulsed COIL with CH_3I molecules as iodine donors in Ref. [6], an intense luminescence of iodine atoms and, in some cases, spontaneous lasing were observed without an external initiation of the medium. This indicated to an appreciable dissociation of the CH_3I molecules due to unidentified chemical reactions, which proceeded during transportation of the gas components of the active medium to the active volume. The main parameters of the active media of the pulsed COILs investigated in Refs [5, 6] are collected in Table 1, which serves to illustrate the influence of chlorine and the type of iodine donor on the output laser characteristics.

In this paper, we propose a reaction scheme which accounts for the observed experimental features of the

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Table 1. Composition of the active media of pulsed COILs [5, 6].

$[\text{O}_2]/10^{16} \text{ cm}^{-3}$	$[\text{CH}_3\text{I}]/10^{16} \text{ cm}^{-3}$	$[\text{CF}_3\text{I}]/10^{16} \text{ cm}^{-3}$	$[\text{Cl}_2]/10^{16} \text{ cm}^{-3}$	E/J	Reference
4	1.3	–	0	1.6	[5]
4	1.3	–	0.2	0	[5]
4	–	1.3	0	1.25	[5]
4	–	1.3	1.6	1.0	[5]
70–120	30–60	–	7–10	Intense luminescence of $\text{I}(^2P_{1/2})$ atoms; spontaneous lasing	[6]

Note: E is the output laser energy.

behaviour of the active media of pulsed COILs utilising CF_3I and CH_3I molecules as iodine donors.

In the case of an incomplete utilisation of chlorine, the active medium of a pulsed COIL contains $\text{O}_2(^1\Delta)$, $\text{O}_2(^3\Sigma)$, Cl_2 , and RI (CH_3I , CF_3I) molecules. The active medium typically contains a buffer gas as well. But when the RI iodide is initiated by pulsed photolysis, the buffer molecules are chemically inert and their presence will not be taken into account in our scheme. Since the atomic binding energies in the RI , O_2 , and Cl_2 molecules are high (no less than 50 kcal mol^{-1} [7]), direct molecular reactions between them that might cause the decomposition of RI in the active medium of the COIL at a temperature $T \sim 300 \text{ K}$ are virtually ruled out owing to the high energy barriers.

This reasoning is confirmed experimentally. In particular, the direct decomposition of Cl_2 and RI by singlet oxygen was shown in Ref. [8] not to occur. The dark reaction between Cl_2 and RI does not take place with any appreciable rate, either. Our investigations of this system [9] revealed that no iodide decomposition was observed after a monthly storage of the Cl_2 – CF_3I mixture in light-tight glass vessels with special-purpose grease-free leak valves. The products of such decomposition are ICl and I_2 molecules, whose extremely low concentrations were checked by iodometric titration with a substantially higher sensitivity than that of optical absorption techniques.

Therefore, for the decomposition of any of the molecules to occur in the gas medium of a pulsed COIL, chemically active particles should be present therein. These particles may be the R radicals and the atoms of iodine and chlorine. Among them only chlorine atoms are capable of disrupting the RI molecules with the reaction



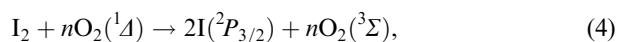
We determined the rate constant k_1 of this reaction for the CF_3I molecule for $T \sim 300 \text{ K}$ [10]. It is relatively high (see Table 2), so that the characteristic CF_3I -molecule disruption time in the reaction (1) is less than 10^{-4} s for typical compositions of the active media of a pulsed COIL. This time is shorter than the characteristic time of gas medium transportation to the active laser volume. Since the R – I binding energy depends only slightly on the kind of the radical [7], the rate constant k_1 for the CF_3I and CH_3I molecules should not differ greatly. The reaction (1) is accompanied by a reaction leading to the loss of radicals R with the production of a peroxy radical RO_2



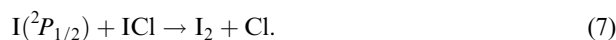
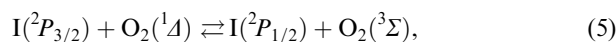
and a reaction reproducing active particles – chlorine atoms,



The set of reactions (1)–(3) is a nonbranching chain reaction of RI decomposition initiated by chlorine atoms, with reaction (3) being a reaction of chain continuation and reaction (2) being a chain-breaking reaction. As noted above, the appearance of chlorine atoms is hardly probable in dark molecular reactions. These atoms emerge in the active medium of a pulsed COIL as a result of the following reactions:

**Table 2.** Rate constants of reactions (1)–(3), (8), and (9) for $T \sim 300 \text{ K}$.

R	$k_1/\text{cm}^3 \text{ s}^{-1}$	$k_2/\text{cm}^3 \text{ s}^{-1}$	$k_3/\text{cm}^3 \text{ s}^{-1}$	$k_8/\text{cm}^3 \text{ s}^{-1}$	$k_9/\text{cm}^3 \text{ s}^{-1}$
CF_3	10^{-12} [10]	1.8×10^{-12} ($[\text{N}_2] = 3.3 \times 10^{17} \text{ cm}^{-3}$) [19] 8×10^{-13} ($[\text{N}_2] = 1.1 \times 10^{17} \text{ cm}^{-3}$) [19]	1.1×10^{-13} [9]	4.5×10^{-11} [15]	2.5×10^{-11} [15]
C_2F_5				10^{-10} (for $k_9 = 8.8 \times 10^{-11} \text{ cm}^3 \text{ s}^{-1}$) [16] 6×10^{-10} (k_8^{max}) (for $k_9 = 0$) [16] 0 (for $k_9 = 1.2 \times 10^{-10} \text{ cm}^3 \text{ s}^{-1}$) [16]	8.5×10^{-11} (for $k_8 = 10^{-10} \text{ cm}^3 \text{ s}^{-1}$) [16] 0 (for $k_8 = 6 \times 10^{-10} \text{ cm}^3 \text{ s}^{-1}$) [16] 1.2×10^{-10} (k_9^{max}) (for $k_8 = 0$) [16]
CH_3	$\sim 10^{-12}$	1.6×10^{-13} ($[\text{SF}_6] = 3.3 \times 10^{17} \text{ cm}^{-3}$) [20] 5×10^{-14} ($[\text{SF}_6] = 1.1 \times 10^{17} \text{ cm}^{-3}$) [20]	1.5×10^{-12} [21] 1.8×10^{-12} [22]		



The necessary condition for the reactions (4)–(7) to proceed is the presence of iodine molecules in the gas medium. It is well known that organic and fluoroorganic iodides contain admixtures of molecular iodine. In the case of fluoroorganic iodides, whose boiling temperature is higher than for alkyl iodides, the content of iodine is significantly lower than for alkyl iodides. Our long-standing operating experience with perfluoroalkyl iodides showed that high-purity iodides can only be obtained with the use of special-purpose glass rectifying columns, which have packings consisting of activated carbon and metal (for instance, tungsten) shavings. Only in this case were we able to attain an iodine content in fluoroorganic iodides of no more than 10^{-5} of the iodide concentration. A simple low-temperature vacuum distillation, even though a multiple one, does not make it possible to attain an equally high degree of purification of alkyl iodides from iodine, particularly so for less volatile alkyl iodides.

Therefore, in an iodide (the donor of iodine) there initially exists a small admixture of iodine molecules, which dissociate into atoms in the medium of singlet oxygen in accordance with the gross process (4), which is the source of iodine atoms in a cw COIL. Despite numerous investigations (see, for instance, Refs [11–14]), the mechanism of dissociation (4) has not been identified, and at present there exists no model that allows a quantitative description of iodine dissociation by singlet oxygen under different experimental conditions. Estimates nevertheless show that the characteristic iodine dissociation time even for concentrations $[\text{O}_2({}^1\Delta)] \sim 3 \times 10^{16} \text{ cm}^{-3}$ is very short ($\sim 10^{-5} \text{ s}$).

The production of iodine atoms by the reaction (4) is accompanied by their equally fast excitation in the reaction (5). Therefore, already upon the mixing of the RI iodide with singlet oxygen there occurs a transformation of ‘impurity’ molecular iodine to excited iodine atoms, which interact with chlorine molecules in the reaction (6). It is precisely the reaction (6) that is the source of primary chlorine atoms in the active medium of a pulsed COIL and it is precisely this reaction that initiates the chain reaction (1)–(3) of RI iodide decomposition.

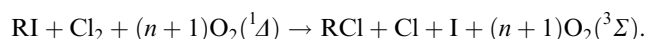
The decomposition of RI is accompanied by the consumption of chlorine molecules and accumulation of ICl molecules. Excited iodine atoms enter into a very efficient reaction (7) with the ICl molecules, with $k_7/k_6 \sim 10^3$ (see Table 3). With this ratio between the rate coefficients, the channel (7) successfully competes with the channel (6) even for a small degree of Cl_2 decomposition. We note that the efficiency of reactions (4) and (5) is high, with the consequential production of three active particles out of one $\text{I}({}^2P_{1/2})$ in the succession of reactions (7), (4), and (5): two excited iodine atoms $\text{I}({}^2P_{1/2})$ and a chlorine atom. The production of iodine molecules in the reaction (7) speeds up the consumption of singlet oxygen in reactions (4) and (5). The chlorine atoms produced in the reaction (7) in their turn accelerate the disruption of RI and reproduce the ICl molecules anew. It is evident that the set of reactions (1)–(5) and (7) is a branching chain reaction of RI

Table 3. Rate constants of reactions (4)–(7) for $T \sim 300 \text{ K}$.

$k_4/\text{cm}^6 \text{ s}^{-1}$	$k_5/\text{cm}^3 \text{ s}^{-1}$	$k_{-5}/\text{cm}^3 \text{ s}^{-1}$	$k_6/\text{cm}^3 \text{ s}^{-1}$	$k_7/\text{cm}^3 \text{ s}^{-1}$
6×10^{-29} [12]	7.6×10^{-11} [23]	2.7×10^{-11} [23]	2×10^{-14} [24]	2.4×10^{-11} [27]
			8×10^{-15} [25]	2.3×10^{-11} [26]
				3.3×10^{-11} [24]

decomposition accompanied with efficient relaxation of singlet oxygen.

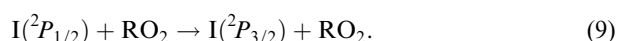
The consumption of initial products and the production of active particles can be represented as the gross reaction



One can see from this equation that the decomposition of one iodide molecule consumes $n+1$ molecules of singlet oxygen. The relaxation rate of singlet oxygen [the reactions (4) and (5)] is determined by the amount of molecular iodine produced in this system only in the reaction (7). The efficiency of the reaction (7) is in turn defined by the efficiency of RI iodide decomposition in the reaction (1). Only this reaction is the source of iodine atoms (and molecules) in the system, and a small admixture of iodine in the initial iodide serves merely to initiate the chain reaction (1)–(3).

The character of advancement of the reactions (1)–(3) accounts for the distinctions observed in the behaviour of the active media of pulsed COILs utilising CH_3I and CF_3I molecules as the iodine donors. Here the controlling factor is the ratio between the rate coefficients for the chain continuation reaction (3) and the chain-breaking reaction (2) for the CH_3 and CF_3 radicals, since the rate coefficients k_1 for both radicals are, as noted above, practically equal. It is clear that the efficiency of decomposition of the RI molecules lowers when the k_3/k_2 ratio decreases. In accordance with the data of Table 2, the k_3/k_2 ratio is equal to 10–30 for the CH_3 radical and to ~ 0.1 for the CF_3 radical, depending on the composition of the active medium of a pulsed COIL. So large a difference in the k_3/k_2 ratio for the two radicals is indication that the decomposition of CH_3I should significantly exceed the decomposition of CF_3I , all other factors being the same. This was experimentally observed in Refs [5, 8].

The quantitative and qualitative difference between the CH_3I and CF_3I decomposition efficiencies and also between the energy dissipation efficiencies of singlet oxygen were demonstrated by numerical calculations of the kinetics of dark reactions in the system $\text{RI} (\text{CH}_3\text{I}, \text{CF}_3\text{I}) - \text{Cl}_2 - \text{O}_2({}^1\Delta) - \text{O}_2({}^3\Sigma) - \text{I}_2$. The calculations were performed for typical compositions of the active media of the pulsed COIL utilised in Refs [5, 6] (see Table 1). The concentration of molecular iodine impurity in the source iodide was varied in this case. In addition to the reactions (1)–(7), the scheme of reactions proceeding in the system under study included the reactions of quenching excited oxygen molecules and iodine atoms by the peroxy RO_2 radicals:



These reactions were considered in Ref. [15] to account for the difference of the output energy of pulsed COILs employing alkyl iodides and perfluoroalkyl iodides as iodine donors in chlorine-free systems. Ref. [15] adduced estimated values of the rate constants k_8 and k_9 (see Table 2). More recently [16] we elaborated a technique allowing a determination of one of the rate constants k_8 or k_9 from the other. This technique was employed in Ref. [16] to investigate the reactions (8) and (9) involving $C_2F_5O_2$ radicals. In this case, at least one of the rate constants (either the preassigned coefficient, or the one to be determined) proved to be very large: about $10^{-10} \text{ cm}^3 \text{ s}^{-1}$ (see Table 2).

It is beyond reason to believe that the rate constants k_8 and k_9 for the CF_3O_2 and CH_3O_2 radicals can substantially differ from the corresponding rate constants for the $C_2F_5O_2$ radicals. In the numerical calculations in our work the rate constants k_8 and k_9 were varied to obtain the best qualitative agreement between the calculated data and available experimental data. In these calculations account was also taken of recombination reactions of the radicals R with iodine and chlorine atoms and with each other, the reactions of peroxy radicals with each other as well as with the R radicals and the atoms of iodine and chlorine, the reactions of chlorine atoms with the molecules of iodine and ICl, and the reactions of quenching of excited iodine atoms and singlet oxygen molecules. The rate constants for all these reactions for $T \sim 300 \text{ K}$ were borrowed from the literature and also from our earlier papers [16–18]. The scheme of the reactions involved will be considered in greater detail in our next paper. Here we note that, to a first approximation, only the reactions (1)–(9) determine the dark decomposition of the donor RI and the dissipation of energy of singlet oxygen in the active medium of a pulsed COIL.

Figs 1–5 show the time dependences of the concentrations of $O_2(^1\Delta)$ and ICl molecules, $I(^2P_{1/2})$ atoms, and RO_2 radicals. These data were obtained by way of numerical solution of the system of differential equations corresponding to the reactions (1)–(9) and also to almost twenty reactions mentioned above. The concentrations of RI, Cl_2 , $O_2(^1\Delta)$, $O_2(^3\Sigma)$, and iodine admixture I_2 were prescribed as the initial compositions of the active media of pulsed COILs given in Table 1. The temperature rise in the course of the reactions was not taken into account.

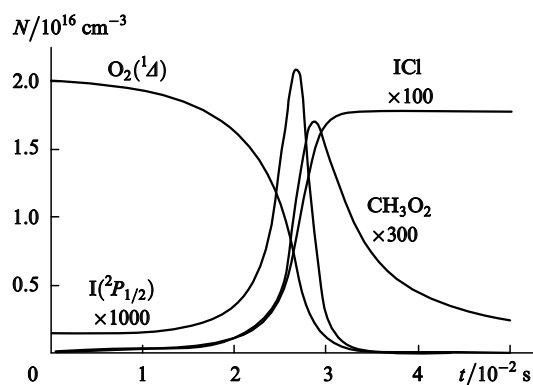


Figure 1. Time dependences of the $O_2(^1\Delta)$, $I(^2P_{1/2})$, ICl and CH_3O_2 concentrations calculated for the active medium of a pulsed COIL [5] ($[CH_3I]_0 = 1.3 \times 10^{16} \text{ cm}^{-3}$, $[O_2(^1\Delta)]_0 = 2 \times 10^{16} \text{ cm}^{-3}$, $[O_2(^3\Sigma)]_0 = 2 \times 10^{16} \text{ cm}^{-3}$, $[Cl_2]_0 = 2 \times 10^{15} \text{ cm}^{-3}$, $[I_2]_0 = 10^{12} \text{ cm}^{-3}$). The rate coefficients of the reactions (1)–(9) were taken from Tables 2 and 3 ($k_8 = 10^{-10} \text{ cm}^3 \text{ s}^{-1}$, $k_9 = 8.5 \times 10^{-11} \text{ cm}^3 \text{ s}^{-1}$).

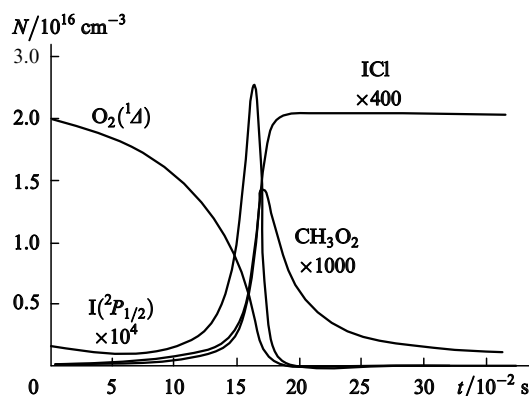


Figure 2. Time dependences of the $O_2(^1\Delta)$, $I(^2P_{1/2})$, ICl and CH_3O_2 concentrations calculated for the active medium of the same composition as in Fig. 1, but for $[I_2]_0 = 10^{11} \text{ cm}^{-3}$.

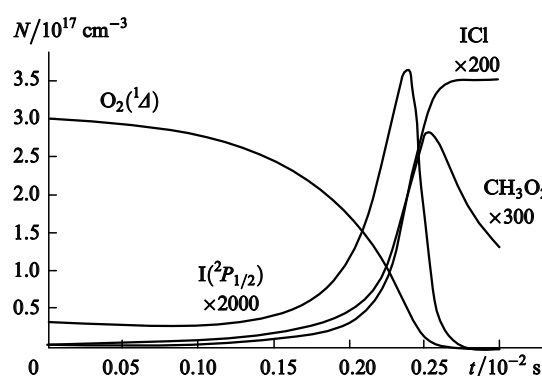


Figure 3. Time dependences of the $O_2(^1\Delta)$, $I(^2P_{1/2})$, ICl and CH_3O_2 concentrations calculated for the active medium of a pulsed COIL [6] ($[CH_3I]_0 = 3 \times 10^{17} \text{ cm}^{-3}$, $[O_2(^1\Delta)]_0 = 3 \times 10^{17} \text{ cm}^{-3}$, $[O_2(^3\Sigma)]_0 = 3 \times 10^{17} \text{ cm}^{-3}$, $[Cl_2]_0 = 6 \times 10^{16} \text{ cm}^{-3}$, $[I_2]_0 = 10^{13} \text{ cm}^{-3}$). The rate constants of the reactions (1)–(9) were taken from Tables 2 and 3 ($k_8 = 10^{-11} \text{ cm}^3 \text{ s}^{-1}$, $k_9 = 9 \times 10^{-11} \text{ cm}^3 \text{ s}^{-1}$).

Figs 1–3 show the advancement of the chain process (1)–(5), (7) of CH_3I decomposition. As one would expect on the basis of a qualitative analysis of the scheme of the reactions (1)–(9), the occurrence of the concentration ‘peak’ of excited iodine atoms coincides in time with the avalanche production of ICl and RO_2 and the fast disappearance of singlet oxygen. In all three cases the final concentration of ICl molecules is far (up to two orders of magnitude) greater than the concentration of iodine impurity in the source iodide, which characterises the efficiency of the chain RI decomposition process. The amount of iodine admixture in the source iodide determines (all other factors being the same) the characteristic time of chain process development and the lifetime of singlet oxygen, which is hardly different from the former. One can see from Figs 1 and 2 that this time lengthens from ~ 30 to $\sim 175 \text{ ms}$ as the iodine impurity concentration lowers from 10^{12} to 10^{11} cm^{-3} .

Figs 4 and 5 show the data calculated for oxygen, chlorine, and CF_3I mixtures. The concentrations of oxygen, iodide, and iodine impurity are the same as for the conditions of Figs 1 and 2, but in the mixture with CF_3I the content of chlorine is eight times higher than in the mixture with CH_3I . Referring to Figs 4 and 5, nevertheless there occurs no chemical production of the $I(^2P_{1/2})$ atoms in this case, i.e. the reaction (7) does not proceed: the amount of

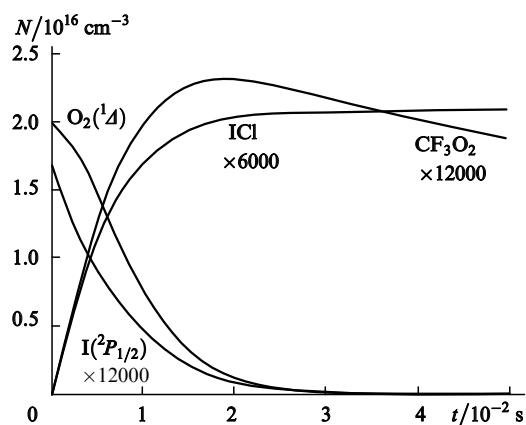


Figure 4. Time dependences of the $O_2(^1D)$, $I(^2P_{1/2})$, ICl and CF_3O_2 concentrations calculated for the active medium of a pulsed COIL [5] ($[CF_3I]_0 = 1.3 \times 10^{16} \text{ cm}^{-3}$, $[O_2(^1D)]_0 = 2 \times 10^{16} \text{ cm}^{-3}$, $[O_2(^3\Sigma)]_0 = 2 \times 10^{16} \text{ cm}^{-3}$, $[Cl_2]_0 = 1.6 \times 10^{16} \text{ cm}^{-3}$, $[I_2]_0 = 10^{12} \text{ cm}^{-3}$). The rate coefficients of the reactions (1)–(9) were taken from Tables 2 and 3 ($k_8 = 10^{-10} \text{ cm}^3 \text{ s}^{-1}$, $k_9 = 8.5 \times 10^{-11} \text{ cm}^3 \text{ s}^{-1}$).

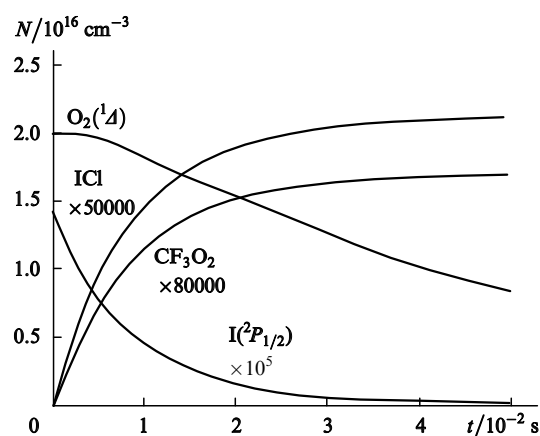


Figure 5. Time dependences of the $O_2(^1D)$, $I(^2P_{1/2})$, ICl and CF_3O_2 concentrations calculated for the active medium of the same composition as in Fig. 4, but for $[I_2]_0 = 10^{11} \text{ cm}^{-3}$.

ICl molecules produced corresponds to the content of iodine impurity in the source mixture in accordance with the sequential advancement of the reactions (4)–(6) and (1)–(3).

We call attention to the behaviour of the $O_2(^1D)$ concentrations in Figs 4 and 5. One can see that the lifetime of singlet oxygen depends on the content of iodine in the source mixture. This time increases approximately five-fold when the iodine concentration is lowered from 10^{12} to 10^{11} cm^{-3} . This strong effect of a very small admixture of iodine on the relaxation of singlet oxygen is due to the production of peroxy CF_3O_2 radicals, which are efficient ‘quencher’ of singlet oxygen, as a result of sequential advancement of the reactions (4)–(6), (1), and (2).

To summarise, we formulate the main results of the work. The set of reactions (1)–(9) determines the dark decomposition of iodide RI in the active medium of a pulsed COIL containing molecular chlorine. This reaction scheme affords a good qualitative and, where this comparison is possible, quantitative description of the available experimental data. The difference in the operation of pulsed COILs utilising CH_3I and CF_3I molecules as iodine donors is explicable on the basis of the mechanism revealed.

Because more complex alkyl iodides and perfluoroalkyl iodides only slightly differ from CH_3I and CF_3I , respectively, in their reactivity in the reactions (1)–(3), all the above conclusions concerning these simplest molecules apply to more complex molecules of the corresponding homologous series.

The chain mechanism (1)–(5), (7) of alkyl iodide decomposition is accompanied by an avalanche production of excited iodine atoms (see Figs 1–3). This allows the set of reactions (1)–(7) to be considered as a new technique for the efficient production of iodine atoms for a COIL.

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