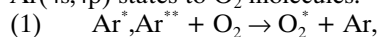


The mechanism and kinetics of ozone formation in a pulse radiolysis study of the Ar-O₂ system

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Abstract The mechanism and kinetics of O₃ formation after an electron pulse have been studied in the Ar-O₂ systems by time resolved optical measurements at $\lambda = 260$ nm. The second order rate constant of energy transfer from excited Ar(4s,4p) states to O₂ molecules:



was found to be $(8.9 \pm 2.1) \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$. It was found also the evidence of the third order process contribution to the energy transfer:

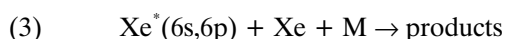


with the rate constant in the range $(1.5-3.7) \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$. The rate constant of the deactivation of excited ozone molecules by O₂ was found to be $(5.1 \pm 0.6) \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1}$.

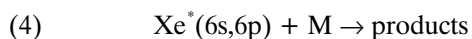
Key words pulse radiolysis • argon • oxygen • ozone

Introduction

The energy transfer process from excited rare gas atoms to various molecules was extensively studied by the Setser group [10, 12, 15], mainly for Xe(³P₂) and Kr(³P₂) lowest metastable excited states. Our earlier studies of this process showed that the rates of energy transfer from Xe(³P₁) resonance state to molecules increase with Xe pressures [17–19, 21, 22]. The same effect was found for higher excited Xe atoms generated in the pulse radiolysis experiments [20]. It was shown that the third order energy transfer reaction:



is responsible for such “pressure” effect. The rate constants of reaction (3) for a number of molecules were found to be in the range from $(1-2) \times 10^{-28} \text{ cm}^6 \cdot \text{s}^{-1}$ for such molecules as CH₄, C₂H₆, CCl₄, SOCl₂ and for many other simple molecules up to $2 \times 10^{-27} \text{ cm}^6 \cdot \text{s}^{-1}$ for HCl [17–22]. In all cases, reaction (3) competes effectively with the second order energy transfer reactions:



as the k_4 rate constants are in the range from $3 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ up to $8 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$, even for low Xe pressures (27–133 hPa).

A review of the literature data on this subject gives only little evidence of existing “third order” energy transfer from other rare gases to molecules (3). Collins and Lee [2, 3] reported this process in the He-M system with the rate constants varying from $1 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ for

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Received: 17 December 2004
Accepted: 27 June 2005

$M = \text{Kr}$ to $8 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ for $M = \text{C}_3\text{H}_8$. For the Ar-M system, the only evidences found for the existence of such process are for the Ar-N₂ system [5] and our recent pulse radiolysis data for the Ar-H₂O system [9].

In this paper, the results of the investigations of the energy transfer processes in Ar-O₂ system will be presented.

Experimental

The pulse radiolysis system is based on SINUS-5 (produced by the Institute of High Energy, Tomsk, Russia) pulsed electron accelerator giving 12 ns single 350 keV electron pulses of total energy about 8 J and dose rate $D_r = (6.9 \pm 0.6) \times 10^{26} \text{ eV} \cdot \text{g}^{-1} \cdot \text{s}^{-1}$.

Ar-O₂ and Ar-O₂-SF₆ mixtures were irradiated in a stainless steel vacuum-proof chamber of volume 1000 cm³ mounted on the accelerator output. The gases pressure in the reaction chamber was measured within 1% with an MKS 116A Baratron pressure transducer.

A xenon arc UV-VIS lamp (XHBO 150 W) was used as an analysing light source in the reaction chamber. A nine-reflection multiple-pass system with outside mounted mirrors was used for the observation of transients ($\lambda = 91 \text{ cm}$). For more details see our previous papers [7, 9].

The time-resolved absorption spectra of ozone at $\lambda = 260 \text{ nm}$ (near the maximum, $\lambda_{\text{max}} = 254 \text{ nm}$) formed during irradiation of the Ar-O₂ mixtures after passing through a 7400 Applied Physics 0.5-m monochromator were amplified by a Hamamatsu (R928) photomultiplier and registered by a 2440 Tektronix digital oscilloscope coupled to a PC computer. Typical oscilloscope absorption traces of ozone are shown in Fig. 1. The

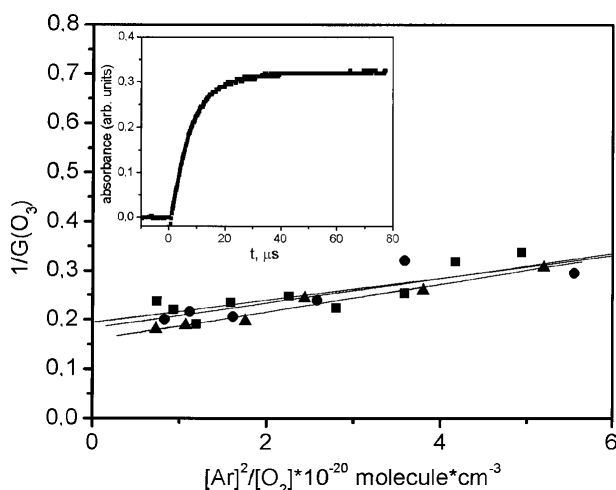


Fig. 1. The plots of $1/\Delta G(\text{O}_3)$ vs. $[\text{Ar}]^2/[\text{O}_2]$. (■) – the total pressure 931 hPa, $P(\text{O}_2)$ range 8–186 hPa (●) – the total pressure 798 hPa $P(\text{O}_2)$ range 13–130 hPa; (▲) – the total pressure 655 hPa, $P(\text{O}_2)$ range 13–107 hPa. For O_2 pressures above 60 hPa, the amount of ozone produced directly in the radiolysis of O_2 were subtracted from the total ozone amount produced in the Ar-O₂ mixture. For the intercepts and slopes of the lines see data in Table 1. Insert figure – typical oscilloscope absorption trace of ozone registered at $\lambda = 260 \text{ nm}$ in the pulse radiolysis of Ar-O₂ mixture. ($P(\text{Ar}) = 798 \text{ hPa}$ and $P(\text{O}_2) = 133 \text{ hPa}$.)

Simplex method was used for the fitting of experimental absorbance vs. time curves using a kinetic equation of stable species formation:

$$(5) \quad A(t) = A_{\text{max}}(1 - \exp(-k_{\text{exp}} \cdot t))$$

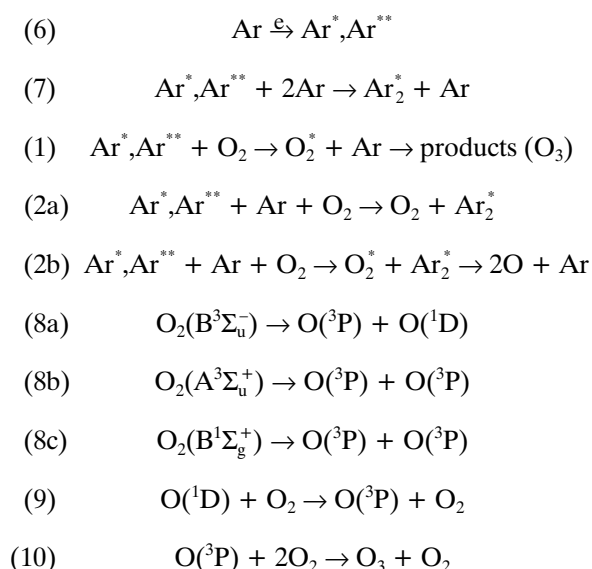
where A_{max} – maximum ozone absorbance, k_{exp} – experimental pseudo-first order rate constant of ozone formation. The $G(\text{O}_3)$ values were found on the basis of ozone dosimetry ($G(\text{O}_3) = 6.2$ for the $\text{O}_2 + 0.5\% \text{SF}_6$ system [9]).

Oxygen and argon gases from Linde Gas Poland were used without purification. The SF₆ gas obtained from Merck was of research purity and before use was purified by “trap to trap” cycles. All experiments were performed at ambient temperatures ($295 \pm 3 \text{ K}$).

Results and discussion

In pulse radiolysis of the Ar/O₂ mixtures only ozone is formed as a stable product. As the stopping power is almost the same for Ar and O₂ ($S(\text{O}_2) = 1.102$ and $S(\text{Ar}) = 1.14$ [6]) at low O₂ concentrations (<10.0%), approximately all the amount of ozone was formed via sensitized process in which the energy is absorbed by argon and then transferred into O₂ molecules.

Earlier studies of rare gase radiolysis mechanism and kinetics showed that the electronically excited Ar(³P_{1,2}), denoted below as Ar*, and higher excited Ar(4p,4d) atoms denoted below as Ar** are formed. About 40% of all the excited Ar atoms formed are in the lowest 4s states and the rest are in the Ar** (4p,4d) states [14]. The Ar* and Ar** atoms decay via energy transfer processes to O₂ molecules competing with the process of Ar₂* formation. A simplified reaction scheme for Ar-sensitized radiolysis of O₂ is shown below:



The steady state kinetic analysis of the above reaction scheme leads to the kinetic equation for the $G(\text{O}_3)$ dependence on O₂ and Ar concentrations for two cases:

$$(11) \quad \frac{1}{G(O_3)} = \frac{1}{2G(Ar^{***})} \cdot \left(1 + \frac{k_7[Ar]^2}{k_1[O_2]} + \frac{k_{2a}[Ar]}{k_1} \right)$$

$$(12) \quad \frac{1}{G(O_3)} = \frac{1}{2G(Ar^{***})} \cdot \left(\frac{\left(1 + \frac{k_7[Ar]^2}{k_1[O_2]} + \frac{k_{2b}[Ar]}{k_1} \right)}{\left(1 + \frac{k_{2b}[Ar]}{k_1} \right)} \right)$$

Kinetic eq. (11) takes into account reaction (2a) (the ozone precursors i.a. O atoms are not formed) and (12) takes into account reaction (2b) (reaction (2b) leads to O atoms – ozone precursors).

Earlier studies of the energy transfer processes for the Xe-M systems showed that for the lowest Xe(6s) states the third order energy transfer processes do not lead to the dissociation of such molecules as H₂S or C₂H₆ [19], but in pulse radiolysis studies of the Xe-RX system (RX = CH₂F₂, CHF₃, CHF₂Cl, CHFCl₂ and CF₃Cl) the products of the third order energy transfer reaction were observed (XeCl^{*}/XeF^{*} excimers) [20]. It seems that in the case of radiolysis of the Ar-O₂ systems the third order energy transfer process should also lead to the excited O₂ molecules which then dissociate to O atoms.

For the constant Ar concentrations eqs. (11) and (12) give the linear plots of 1/G(O₃) vs. [Ar]²/[O₂]:

$$(13) \quad \frac{1}{G(O_3)} = A + B \frac{[Ar]^2}{[O_2]}$$

where:

$$A = \frac{1}{2G(Ar^{***})} \cdot \left[1 + \frac{k_{2a}[Ar]}{k_1} \right] \quad \text{for (11) and}$$

$$A = \frac{1}{2G(Ar^{***})} \quad \text{for (12)}$$

$$B = \frac{k_7}{2G(Ar^{***})k_1} \quad \text{for (11) and}$$

$$B = \frac{k_7}{2G(Ar^{***})(k_1 + k_{2b}[Ar])} \quad \text{for (12)}$$

Figures 1 and 2 show the experimental plots of 1/G(O₃) vs. [Ar]²/[O₂]. The obtained intercepts (A-values) and slopes (B-values) are shown in Table 1.

From the intercept values (A), the radiation yields $G(Ar^{***}) = 2.83 \pm 0.22$ for the Ar-O₂ system and $G(Ar^{***}) = 1.0 \pm 0.1$ for the Ar-O₂-SF₆ system were found. The first yield is in good agreement with the literature value $G(Ar^{***}) = 3.05$ [8]. In the radiolysis of the Ar-O₂ system in the presence of SF₆ as an electron

Table 1. The kinetic parameters obtained from 1/G(O₃) vs. [Ar]²/[O₂] plots shown in Figs. 1 and 2 and k_1 rate constants calculated for $k_7 = 1.28 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ and $k_2 = 0$

P(Ar) [hPa]	A	B·10 ²² [cm ³ /molec.]	((A/B)· k_7 ·10 ¹⁰ (k_1)) [cm ³ ·s ⁻¹]
Ar-O ₂			
666	0.159 ± 0.008	2.84 ± 0.27	7.2 ± 0.8
800	0.183 ± 0.017	2.53 ± 0.43	9.3 ± 1.5
933	0.194 ± 0.110	2.27 ± 0.11	10.95 ± 0.6
Ar-O ₂ +0.5%SF ₆			
800	0.453 ± 0.029	7.00 ± 0.50	8.3 ± 0.6

scavenger much lower $G(Ar^{***}) = 1.0$ value was found what suggests that a large amount excited Ar atoms ($G(Ar^{***}) = 1.8$) are produced via Ar ionization processes and then ion recombination.

In Table 1, there are also shown the two body energy transfer k_1 rate constants calculated from (13) for $k_{2a} = 0$, $k_{2b} = 0$ and $k_7 = 1.28 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$. The k_7 rate constant taken for calculation is the mean value from the literature data ($k_7 = 1.23 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ [1]; $1.44 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ [11]; $1.18 \times 10^{-30} \text{ cm}^6 \cdot \text{s}^{-1}$ [16]). The k_1 rate constant varied from $6.15 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ to $11.7 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ (see data in Table 1). The k_1 rate constant found from these experiments is much higher than previously found for energy transfer from Ar(4s) to O₂ $k_1 = 2.2 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ [15] and close to Sadeghi *et al.* [14] quenching rate constants for Ar(2p₁) ($k_1 = 7.6 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$), Ar(2p₅) ($k_1 = 7.2 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$), Ar(2p₆) ($k_1 = 4.6 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$) and Ar(2p₈) ($k_1 = 6.3 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$).

Taking the literature energy transfer rate constants for Ar(4s) equal to $2.2 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ [5, 15], for Ar(4p) states equal to $7 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ [14] and the contribution of Ar(4s) states equal to 40% of all Ar excited states formed in pulse radiolysis of argon [20], the k_1 rate

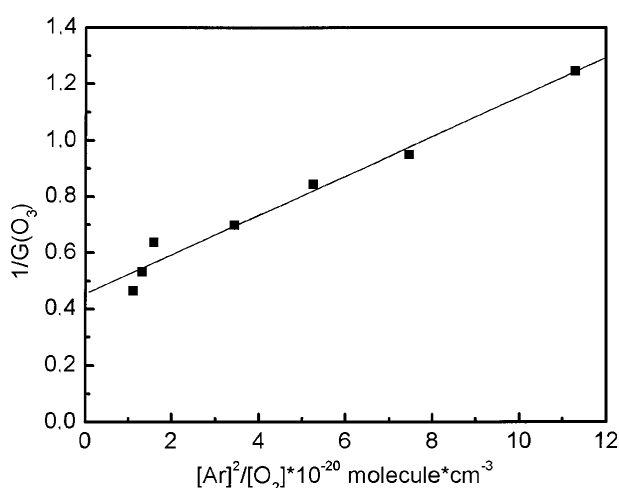


Fig. 2. The plot of 1/ΔG(O₃) vs. [Ar]²/[O₂] – (■) – the total pressure 798 hPa + 0.5% SF₆, P(O₂) range 13–107 hPa. For O₂ pressures above 60 hPa, the amount of ozone produced directly in the radiolysis of O₂ were subtracted from the total ozone amount produced in the Ar-O₂ mixture. For the intercept and slope of the line see data in Table 1.

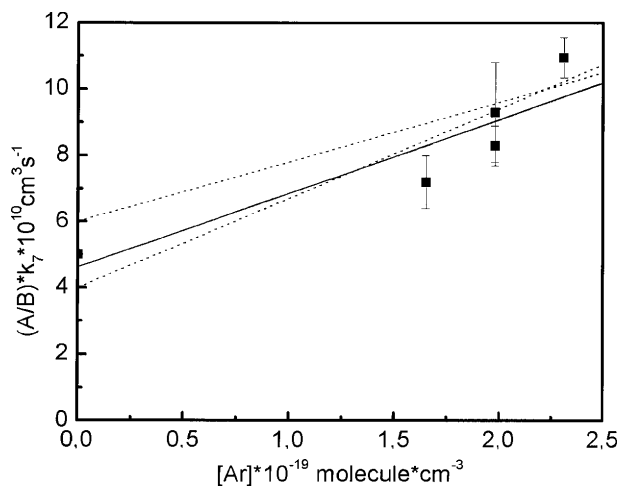


Fig. 3. The plots of $(A/B) \cdot k_7$ vs. $[Ar]$. Solid line for $k_1 = 5 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ and $k_2 = 2.3 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$. Dotted lines – simulations according to eq. (14) taking $k_1 = 4 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ and $k_2 = 2.7 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$ (lower line) or $k_1 = 6 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$ and $k_2 = 1.8 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$ (upper line).

constant (energy transfer rate constant from both Ar(4s) and Ar(4p) states) value can be estimated as being equal to $(5.0 \pm 1.0) \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$. As our experimental k_1 values are higher than the above k_1 value this can be the evidence of the contribution of the third order energy transfer process (reactions (2a) and/or (2b)) in the sensitized Ar-O₂ radiolysis.

In Fig. 3, the plot of $(A/B) \cdot k_7$ vs. $[Ar]$ concentration is shown. The linear dependence has been expected if reactions (2a) and/or (2b) would be included into the reaction scheme as from eqs. (11), (12) and (13) we have:

$$(14) \quad \frac{A}{B} \cdot k_7 = k_1 + k_2[Ar]$$

As it is seen, our experimental data can be fitted as a linear dependence of $(A/B) \cdot k_7$ vs. argon pressure (the point at $[Ar] = 0$ was taken as being equal to $5 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$). The k_2 rate constant was found to be $(2.3 \pm 0.6) \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$. The simulations of eq. (14) for lower and higher k_1 values at $[Ar] = 0$ ($k_1 = 4 \times 10^{-10}$, and $6 \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$) give the k_2 rate constants equal to $2.7 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$ and $1.8 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$, respectively.

Unfortunately, due to the scatter of results and a relatively small Ar pressure range (666–933 hPa) at which the experiments were done, rather a low accuracy of k_1 and k_2 rate constants must be pointed out. The above discussion shows that the third order energy transfer rate constant, k_2 , in the electron beam irradiated Ar-O₂ system should be in the range from 1.8×10^{-29} to $2.7 \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$. It is in agreement with the data for Ar-H₂O pulse radiolysis study where the third order energy transfer rate constant was found to be $(1.8 \pm 1.4) \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$ [9].

The kinetics of ozone formation

Earlier studies on the kinetics of ozone formation showed that ozone is produced only in reaction (10)

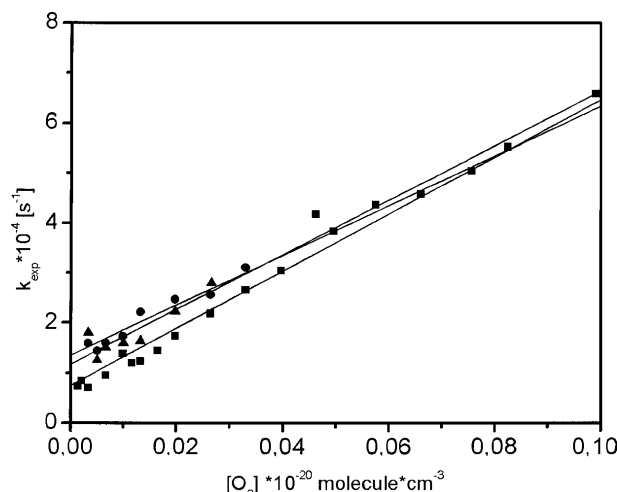
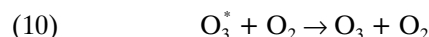


Fig. 4. The plots of k_{exp} vs. $[O_2]$: (■) – the total pressure of Ar + O₂ – 931 hPa – $k_{10} = (5.71 \pm 0.11) \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1}$; (●) – the total pressure of Ar + O₂ – 798 hPa, $k_{10} = (4.97 \pm 0.08) \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1}$; (▲) – the total pressure of Ar + O₂ – 655 hPa, $k_{10} = (5.46 \pm 0.17) \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1}$.

with the rate constant $k_{10} = 2.8 \times 10^{-34} \text{ cm}^6 \cdot \text{s}^{-1}$ [13] (high pressure limit) as the all O(¹D) atoms are quenched to O(³P) in a few nanoseconds ($k_9 = 4 \times 10^{-11} \text{ cm}^3 \cdot \text{s}^{-1}$ [4]) by O₂ molecules.

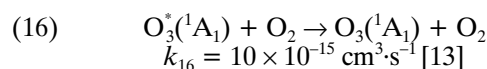
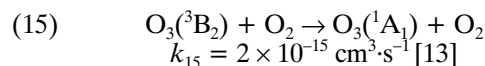
There is also strong evidence that it is a two-step process in which the excited O₃(¹A₁)^{*} and O₃(³B₂) molecules are formed. Stable ozone (O₃(¹A₁)) molecules are produced by the collisions of O₃(¹A₁)^{*} and O₃(³B₂) with O₂ molecules [13].

From the time resolved ozone absorption spectra, we have found pseudo-first order rate constants (k_{exp}) of ozone formation. In Fig. 4 there are shown the plots of k_{exp} vs. O₂ concentrations at constant Ar pressures. As it is seen, linear dependencies were found for all the experimental series. The slopes can be assigned to the two-body deactivation of O₃ excited molecules by O₂:



The experimental value of k_{10} is equal to $(5.4 \pm 0.3) \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1}$.

The analysis of Ramirez *et al.* [13] of the mechanism of ozone formation suggests that the k_{10} rate constant should be treated as a sum of the rates for reactions:



As the $[O_3^*] = [O_3(^3B_2)] + [O_3(^1A_1)^*]$, from the k_{10} , k_{15} and k_{16} rate constants it should be found that the yields of O₃(³B₂) and O₃(¹A₁)^{*} states are equal to 0.575 and 0.425, respectively. This is in agreement with Ramirez *et al.* [13] data, who reported that about 60% of all excited ozone molecules are formed in O₃(³B₂) states and the rest are O₃(¹A₁)^{*} molecules.

Conclusions and remarks

The analysis of the data from pulse radiolysis of the Ar-O₂ system showed that the excited Ar atoms are formed with the total yield $G(\text{Ar}^*) = 2.83 \pm 0.22$. In the presence of an electron scavenger (SF₆) $G(\text{Ar}^*) = 1.0 \pm 0.1$, which means that Ar* and Ar⁺ atoms are formed also via ionization processes with the yield $G(\text{Ar}^*) = 2.83 - 1.0 = 1.83$.

The O₂ excited molecules which then dissociate to O(³P) and O(¹D) atoms are produced in the two-body energy transfer process (reaction (1)) with the rate constant $k_1 = (8.9 \pm 2.1) \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$, if it is the only possible energy transfer process (i.e. $k_2 = 0$). The analysis of the experimental data showed that there is also possible the third order energy transfer process (reaction (2)). It was found that the k_2 rate constant should be equal to $(2.3 \pm 0.6) \times 10^{-29} \text{ cm}^6 \cdot \text{s}^{-1}$. In this case, the k_1 rate constant should have a lower value $(5 \pm 1) \times 10^{-10} \text{ cm}^3 \cdot \text{s}^{-1}$.

It seems that the experiments for the higher Ar pressures should give stronger evidence for the participation of reaction (2) in the energy transfer process in Ar-sensitized radiolysis.

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