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Electron capture by haloethanes in a carbon dioxide buffer gas

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ABSTRACT

An electron swarm method for investigation of the electron capture reaction has been modified. It is based on the registration of the temporal evolution of a single electron pulse. The method gives straightforward results on electron drift velocities in any mixture and allows calculating the rate constants for the electron capture. The thermal electron capture kinetics for eight haloethanes has been investigated. Only two-body attachment processes have been found in their mixtures with carbon dioxide. All corresponding rate constants have been determined. A link between the two-body rate constant and an electron polarizability of the attaching center has been demonstrated. (Int J Mass Spectrom 205 (2001) 85–92) © 2001 Elsevier Science B.V.

1. INTRODUCTION

The electron swarm method with an ionization chamber has been applied for many years in investigating electron attachment processes. First Bortner and Hurst [1] and Christophorou [2] studied the dependence of an electron attachment cross section on electron energy, and lately the authors [3,4], after some changes of the techniques, investigated the thermal electron capture kinetics. Our method seems to be particularly convenient for studying multibody pressure-dependent processes of electron attachment. Using this method we have proved that many van der Waals dimers can attach electrons, and we have measured the rate constants for these processes [5].

The van der Waals dimers of some halocarbons react with thermal electrons with the rates appreciably higher than with the individual molecules [6–8]; therefore, these investigations seem to be worthy of continuation from a practical point of view, especially, if one takes into account the many attempts to find methods of destroying freons existing in the atmosphere. Among those some plasma methods use an electron beam or microwave discharges freely localized in the troposphere [9,10]. The key processes in these studies are electron attachment reactions. For modeling the system it is necessary to know the exact rate constants of these processes, their mechanism and their products. Of these, the swarm method allows the determination of the exact rate constants of these processes and their mechanism.

There are three major groups of compounds that react with thermal electrons in a more complicated way than just by a simple two-body process [5]:

- oxygen and oxygen-containing compounds (such as N_2O, NO, NO_2, SO_2)
- inorganic hydrides (HF, HBr, HCl, H_2S),
- some halocarbons.

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Dedicated to Professor Aleksandar Stamatovic on the occasion of his 60th birthday.

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The oxygen-containing compounds react as individual molecules slowly in two-body processes and much faster in three-body processes. The van der Waals dimers react in both two- and three-body processes. The inorganic hydrides capture thermal electrons only as van der Waals dimers in two- or three-body processes. The halomethanes react as individual molecules only in the two-body process, whereas as van der Waals dimers they react both in two- and three-body processes. Therefore, the full mechanism for the process can be written in the following manner:

$$e + AB \xrightarrow{k_1} AB^{-*} \tag{1}$$

$$AB^{-*} \xrightarrow{k_2} AB + e \tag{2}$$

$$AB^{-*} \xrightarrow{k_3} AB^{-} (or\ A + B^{-}) \tag{3}$$

$$AB^{-*} + M_s \xrightarrow{k_4} products \tag{4}$$

$$AB + CD \overset{K^{eq}}{\longleftrightarrow} (AB \cdot CD) \tag{5}$$

$$e + (AB \cdot CD) \xrightarrow{k_6} (AB \cdot CD)^{-*} \tag{6}$$

$$(AB \cdot CD)^{-*} \xrightarrow{k_7} AB + CD + e \tag{7}$$

$$(AB \cdot CD)^{-*} \xrightarrow{k_8} products \tag{8}$$

$$(AB \cdot CD)^{-*} + M_s \xrightarrow{k_9} products \tag{9}$$

The complete kinetic equation for the whole set of possible processes, obtained with a steady-state approximation, is written below:

$$-\frac{d[e]}{dt} = \left\{ \frac{k_1 k_3}{k_2} + \frac{k_1 k_4 [M_s]}{k_2 + k_4 [M_s]} + \frac{K^{eq} k_6 k_8}{k_7} [CD] + \frac{K^{eq} k_6 k_9 [CD][M_s]}{k_7 + k_9 [M_s]} \right\} \cdot [AB] \cdot [e] \tag{10}$$

where $[AB]$ and $[CD]$ are the concentrations of the reacting molecules (in the case of homogenous dimers $AB=CD$), $[M_s]$ is the concentration of the stabilizing agent (environmental gas and reactants), k_i are the respective rate constants and K^{eq} is the equilibrium constant for the formation of van der Waals dimer.

The first and third terms describe two-body processes with individual molecules and van der Waals complexes, respectively. The second and fourth ones correspond to the collisionally stabilized processes (or the so-called Bloch–Bradbury, B–B, mechanism), with individual molecules and van der Waals complexes, respectively.

Which part of the equation dominates depends on the electron attaching system. We have found that in the case of inorganic hydrides the rate and the mechanism of the process depends on the dipole moment and electronegativity of the molecules forming the van der Waals complexes [5,11]. A stabilization of the excited negative ion is not effective in the mixtures of halomethanes with carbon dioxide or nitrogen. For oxygen and oxygen-containing compounds, depending on the environmental gas, all four mechanisms are possible except that the rate constants for the simple two-body process is usually extremely small [3,12–15].

In this article the modified method for analyzing the experimental results is presented; this method seems very promising for getting two important electron transport parameters: electron capture rate and electron mobility. Also new data on electron attachment processes to some haloethanes obtained with this method are presented.

To analyze experimental results, the so-called effective rate constant, k_{eff}, is usually employed. It corresponds to the true rate constant only if the two-body process occurs, otherwise it is pressure-dependent. Then Eq. (11) following immediately from Eq. (10) should be applied:

$$k_{eff} = -\frac{d[e]}{dt} \cdot \frac{1}{[AB][e]} = \frac{k_1 k_3}{k_2} + \frac{k_1 k_4 [M_s]}{k_2 + k_4 [M_s]} + \frac{K^{eq} k_6 k_8}{k_7} [CD] + \frac{K^{eq} k_6 k_9 [CD][M_s]}{k_7 + k_9 [M_s]} \tag{11}$$

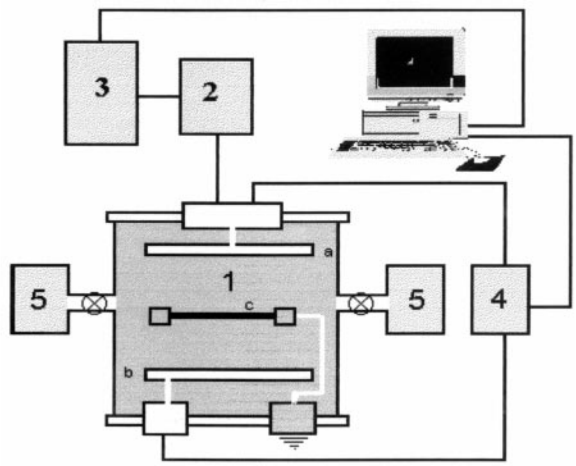

Fig 1. Experimental set up: 1=ionization chamber (a, anode; b, high voltage electrode; c, α-source), 2=preamplifier, 3=oscilloscope, 4=high voltage supply system, 5=vacuum system.

This equation will be used further on to analyze our experimental results.

2. Experimental procedure

An experimental set-up used in the present investigation is shown in Fig.1. It consists of an ionization chamber (1) with two parallel electrodes (a) and (b), a Canberra-Packard preamplifier model 2006 (2), a fast (50 ns) oscilloscope with digital memory (3) connected with a computer and a computer-controlled Canberra-Packard dual 0–5 kV H.V. power supply model 3125 (4). The electron swarm is produced by an α-particle in the plane of an α-particle source (c). The electron swarm moves to the collecting electrode (a) traversing a distance d ($\sim$2 cm) under the influence of a uniform electric field, E, applied between the electrodes. To minimize the influence of the α-source ring, the electrode potentials are chosen in such a way that there is a zero potential at the distance where it is placed. Additionally it is grounded (which also lowers the noise level). A drift velocity, W, is a function of the density-reduced electric field, E/N, where N is the total density of the gas in the chamber. In the case of thermal electrons $W = \mu_N \cdot E/N$, where μ_N denotes the density-normalized electron mobility. In the presence of an electron acceptor drifting electrons are being removed from the gas. In the case of thermal electrons the removal rate depends on the effective electron capture rate constant, k_{eff}. The electron swarm while traveling induces an electric potential on the collecting electrode (anode).

In the classical method the resulting electric pulse is amplified first by a preamplifier and further by a linear amplifier. The output pulse is registered in a multichannel analyzer [2–4]. The pulse height is a function of both the drift velocity and the rate constant of the electron capture. Hence, both W and k_{eff} values can be determined from the dependence of pulse height on E/N. This method, although effective, uses rather complicated equations to fit simultaneously the electron drift velocity and the electron capture rate constant. Moreover, it is rather time-consuming, because good statistics are needed to determine accurately the peak height for each E/N. Also, the method is good only for the investigation of thermal electron capture processes because it uses the fact that W is a linear function of E/N [16]. The details of this procedure are described in [3] and [4].

In the present method we eliminate the linear amplifier and multichannel analyzer from the set-up. Instead, the output pulse from the preamplifier is registered in the digital memory of the oscilloscope. The data are further transferred to the computer memory. The preamplifier converts the input signal from the collecting electrode into an output voltage pulse (whose amplitude is proportional to that of the input signal) with the rise time less than 35 ns. The exponential decay time, t_1, of the pulse is equal to 46 μs (both the values were checked with a step function generator). The electrons traverse the distance d at a time t_0 in the range of 1–50 μs, depending on applied E/N. Thus, we can assume that the preamplifier follows immediately the changes in the electrode potential and discharges both during the pulse and afterward, which means that the total response function of the preamplifier is that of the decay. To get the shape of the pulse registered by the oscilloscope, we will follow considerations presented in our previous paper [17].

The change in the electrode potential, $U(t)$, with a time t caused by the drifting swarm of electrons ($t \leq t_0$) in the presence of electron acceptor is given by Eq. (12):

$$U(t) = \frac{a}{k_{eff} \cdot N_a \cdot t_0} \cdot [1 - \exp(-k_{eff} \cdot N_a \cdot t)] \quad \text{for } t \leq t_0 \tag{12}$$

where a is a coefficient of proportionality and N_a is a number density of the electron acceptor. In the case of pure carrier gas it easily simplifies, as expected, to:

$$U(t) = \frac{a}{t_0} \cdot t \quad \text{for } t \leq t_0 \tag{13}$$

The response function of the preamplifier (for any time t) is given, as discussed above, by equation (14).

$$G(t) = b \cdot \exp\left(-\frac{t}{t_1}\right) \tag{14}$$

where b is a coefficient of proportionality.

The value of the output signal of the preamplifier after a time τ, $V(\tau)$, is a result of both processes occurring simultaneously. This leads to the integral (15) during the process of collecting electrons ($\tau < t_0$)

$$V(\tau) = \int_0^{\tau} \frac{dU(t)}{dt} \cdot G(\tau - t)\, dt \tag{15}$$

and integral (16) after electrons were collected ($\tau > t_0$)

$$V(\tau) = \int_0^{t_0} \frac{dU(t)}{dt} \cdot G(\tau - t)\, dt \tag{16}$$

Solving the integrals leads to two equations, which describe the changes in the electrode potential during the whole process:

$$V(\tau) = \frac{A \cdot t_1}{t_0 \cdot (1 - k_{eff} \cdot N_a \cdot t_1)} [\exp(-k_{eff} \cdot N_a \cdot \tau) - \exp(-\tau/t_1)] \quad \text{for } \tau < t_0 \tag{17}$$

where A = a · b and

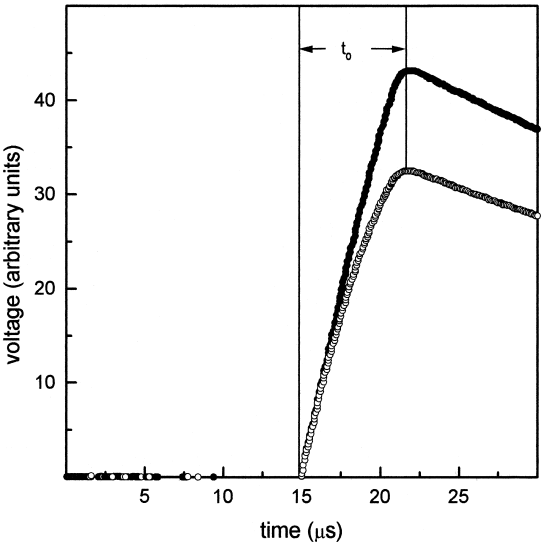

Fig 2. Averaged oscilloscope signals in C_2F_5Cl–CO_2 mixture at *E/N* = 2.1×10^{-17} V cm^2 molec.$^{-1}$ and constant $[CO_2] = 3.3 \times 10^{19}$ molec. cm^{-3}: $[C_2F_5Cl] = 7.3 \times 10^{16}$ molec. cm^{-3} (upper curve), $[C_2F_5Cl] = 9.9 \times 10^{16}$ molec. cm^{-3} (lower curve).

$$V(\tau) = \frac{A \cdot t_1}{t_0 \cdot (k_{eff} \cdot N_a \cdot t_1 - 1)} \cdot \{1 - \exp[-t_0 \cdot (k_{eff} \cdot N_a \cdot t_1 - 1)/t_1]\} \cdot \exp(-\tau/t_1) \quad \text{for } \tau > t_0 \tag{18}$$

Because of electronic noise and the fact that α-particles producing electron swarms do not go exactly in the plane of the source, to obtain noise-free and repeatable results, at least a few hundred pulses have to be properly averaged. The triggering and delay time functions of the oscilloscope were used for preliminary adjustment of the pulses to the common beginning time. A special computer program has been written for smoothing the data, eliminating the double pulses (appearing as a result of a statistical nature of the α-particle emission) and fine synchronization of the beginning time. Only the pulses prepared this way (~70–80% of the whole population) were summed and averaged. The final result of averaging 700 pulses is presented in Fig.2. As can be seen, both the times corresponding the beginning and the maximum of the pulse are quite well defined and noise is eliminated. The beginning time corresponds to the time of formation of the electron swarm. The second time is that of arrival of the swarm at the collecting electrode, provided that the rate of the removal of electrons is not too big (ca. 5×10^5 s^{-1} in our case). In the opposite case the maximum appears earlier, as it follows from the analysis of the theoretical curve (Eq. 16). The drift time t_0 is a simple difference between these two values and thus the electron mobility in any particular mixture, an important electron transport coefficient, can be found easily from the simple relation $\mu_N = (d/t_0)/(E/N)$ without any additional presumptions. Also, this value can be used in Eqs. (16) and (17), which leaves only two coefficients in these equations, A and k_{eff}, to be computer-fitted. This makes the procedure of finding k_{eff} very reliable.

The experiment was performed in such a way that first each haloethane–carbon dioxide mixture was introduced into a chamber at highest applied total pressure (ca. 1000 Torr). The 700 pulses were registered for given *E/N* and averaged as described above. The procedure was repeated usually for five *E* values (at constant *N*) in the rather wide range of *E/N* (6×10^{-18}–3×10^{-17} V cm^2 $molec.^{-1}$) where electrons in carbon dioxide are in thermal equilibrium with gas molecules. Next, the mixture was pumped out to a lower pressure and the measurement procedure was repeated (for ~15 consecutive pressures in the range 300–1000 Torr). The whole experiment was carried out for a few different initial concentrations of haloethane in carbon dioxide.

The shape of the pulse in principle can be deformed by the longitudinal diffusion of electrons during the drift of the swarm. The deformation is generally thought to be negligible where a pulse height is analyzed and a total pressure is rather high. This can be justified if one uses Eq. (19) introduced by Snelson and Lucas [18] and discussed further by Hunter and Christophorou [19].

$$D_L/\mu = V \cdot (\delta t)^2/4t_0^2 \qquad (19)$$

In this equation D_L/μ is the ratio of the longitudinal diffusion coefficient, D_L, to electron mobility, μ ($=W/E$), V is the potential applied to the collecting electrode (the α-source is kept at zero potential, cf. Fig.1), δt is a half-width of an electron swarm profile on arrival to the collecting electrode and t_0 is the drift time. In our case V is in the range 100–2000 V, t_0 is ~10 μs and $D_L/\mu = 0.03$ V for CO_2 at our range of *E/N* [19]. This gives the ratio $\delta t/t_0$ no more than a few percent at the lowest applied V. Thus it seems that the widening of the pulse owing to the diffusion can be neglected. Also, transverse diffusion can be neglected as the transverse diffusion coefficient, D_T, is exactly the same as D_L in our range of *E/N* [19].

We used technical CO_2. The other gases were from Aldrich Chemical Company (except 1,2-C_2H_4ClBr from Merck and 1,2-$C_2H_4Cl_2$ from POCh Poland). All the compounds were purified by the vacuum freeze–pump–thaw technique. All measurements were carried out at room temperature (293 ± 2 K).

3. Results and discussion

Introducing the modified method of measurement and calculation of the experimental results we have repeated an experiment for one of the previously measured systems to check its reliability. For this purpose we have chosen CHF_3 diluted with CO_2 where (in our previous work [4]) the most complicated dependence of k_{eff} on total pressure was observed. As it is seen from Fig. 3 the results obtained with two methods (within rather small experimental error) are the same.

For the same purpose we have also calculated thermal density-normalized electron mobilities in the investigated mixtures. An example is presented in Fig. 4. All others are of the same magnitude. Comparing them with the one for CO_2 ($1.7 * 10^{22}$ V^{-1} cm^{-1} s^{-1}) they are ~10% lower. There are no data on electron mobilities for haloethanes, but they can be approximated using their dipole moments and the experimental equation of Christophorou [20]. They are equal to ~(3–4) $\cdot 10^{21}$ V^{-1} cm^{-1} s^{-1}. So with the concentrations of haloethanes in our mixtures in the range 0.1–1 % the electron mobilities in the CO_2—haloethanes systems should be only slightly (~10%) smaller than the one in pure CO_2. This shows that the measurement

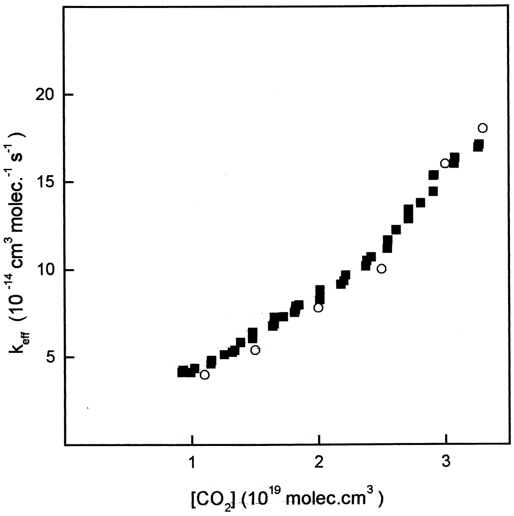

Fig 3.The influence of CO_2 concentration on the effective rate constant, k_{eff}, in the system CHF_3—CO_2 (open points from ref. [4]).

of the mobility in the mixtures also gives correct results.

In our previous publications [4–8] we presented the results on electron capture by some halomethanes and showed that in the mixtures of halomethanes with CO_2 or nitrogen the process includes attachment both by individual molecules and by van der Waals complexes. In this article we present the results on electron attachment processes by a set of haloethanes diluted with CO_2. The example result is shown in Fig. 4 in terms of k_{eff} versus [CO_2]. All measured rate constants together with the available literature data are presented in Table 1.

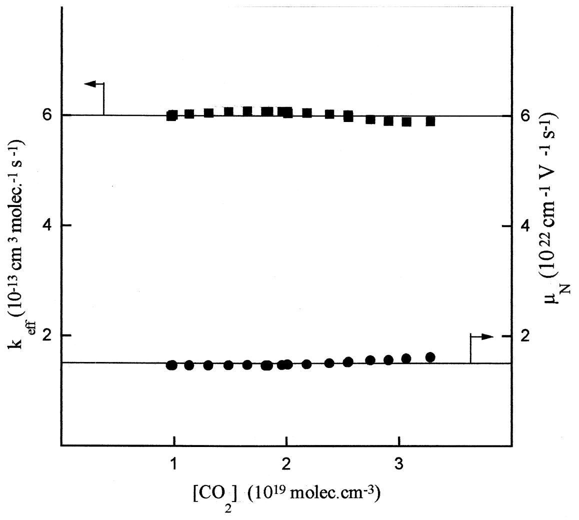

Fig 4. Two-body rate constant and thermal electron mobility as a function of CO_2 concentration in the mixture of 1,1,1-$C_2H_3F_2Cl$—CO_2. The concentration of 1,1,1- $C_2H_3F_2Cl$ was in the range 1×10^{17}—1×10^{18} molec. cm^{-3}.

As is shown, for the case of 1,1,1-$C_2H_3F_2Cl$ shown in Fig. 4, as well as for the other ones we have investigated (Table 1), k_{eff} does not depend on the CO_2 or the electron acceptor concentration. This means that only two-body process electron attachment by a single molecule takes place. This is not a trivial result. It is surprising, because we would expect that haloethanes will behave similarly to halomethanes, that is, form structureless van der Waals complexes that lower the activation energy of the capture process [5]. A more systematic investigation of electron capture by haloalkanes with a different carbon chain length is required to get a general picture.

The inspection of Table 1 shows also that the literature data are scarce and the cited ones scatter rather strongly. So, it is often difficult to judge about validity of new results by comparison with the literature. Thus, there is a need to find the link between the value of the rate constant and molecular parameters both from a theoretical and practical point of view. Previously [21,22], using data found in the literature, we have analyzed a possible correlation between such molecular parameters as dipole moment, μ_D, electronic polarizability, α, and total molecular polarizability, $\alpha + \mu^2/3kT$. Preliminary analysis has shown that none of these parameters for a molecule as a whole can be used. Further analysis was based on the fact that a molecular electron polarizability α (which reflects an ability of core [mostly] electron orbitals of a molecule to deform under the influence of the electric field) is an additive quantity. Therefore α for a molecule or any of its particular parts can be calculated as a sum of atomic and/or bond values. Therefore we have introduced a new term, the polarizability of the attaching center, α_{center}. We define it as the sum of the polarizabilities of those atoms, which are able to accept or influence the process of attaching electrons. Again, a preliminary analysis has shown

Table 1
Electron transport and structure parameters of the investigated haloethanes

Molecule	k (cm^3 molec.$^{-1}$ s^{-1})	α (calculations)	α, Å^3	k$_{\text{lit.}}$ (cm^3 molec.$^{-1}$ s^{-1})
C_2H_5CI	3,4*10^{-14}	α_{Cl}	2,18	<1,6*10^{-15}[26],~1*10^{-13}[27]
1,1,1-$C_2H_3F_2Cl$	6,0*10^{-13}	$\alpha_{Cl} + 2\alpha_F$	3,29	—
C_2H_5Br	5,3*10^{-12}	α_{Br}	3,05	9*10^{-11}[28],1,4*10^{-10}[29], 2,8*10^{-13}[30]
C_2F_5Cl	3,3*10^{-12}	$\alpha_{Cl} + 2\alpha_F$	3,29	<6,2*10^{-12}[31]
		$\alpha_{Cl} + 5\alpha_F$	4,97	
1,2-$C_2H_4Cl_2$	2,6*10^{-11}	$2\alpha_{Cl}$	4,36	3,2*10^{-11}[32]
1,2-C_2H_4ClBr	4,5*10^{-10}	$\alpha_{Cl} + \alpha_{Br}$	5,23	1*10^{-9}[33]
1,1,2-$C_2F_3Cl_3$	5,4*10^{-9}	$3\alpha_{Cl} + \alpha_F$	7,10	1,1*10^{-8}[34]
		$3\alpha_{Cl} + 3\alpha_F$	8,21	
1,1,1-$C_2F_3Cl_3$	2,5*10^{-7}	$3\alpha_{Cl} + 3\alpha_F$	8,21	2,8*10^{-7}[34]

α-electron polarizability of the attaching center.
α_F = 0,557 Å^3, α_{Cl} = 2,18 Å^3, α_{Br} = 3,05 Å^3 [23]

that, in the case of halocarbons, carbon and hydrogen atoms should be eliminated from consideration and only polarizabilities of halogen atoms should be taken into account. With these limitations, we have found that log(k) for the two-body electron attachment rate constant depends linearly on the electron polarizability of the attaching center, α_{center}, for many compounds [21,22].

In the case of haloethanes we take into account the polarizabilities of halogen atoms as given in [23]. The corresponding data are collected in Table 1 and the plot of log(k) versus α_{center} is shown in Fig. 5. As previously, we indeed observe a generally linear increase in log(k) with α_{center}. Some ambiguity appears when all halogen atoms are not placed at the same carbon atom. In the case of 1,1,2-$C_2F_3Cl_3$ and C_2F_5Cl the solid squares are obtained when all (three or five, respectively) α_F are taken into account and open squares (much better fitting to the line) when only one or two α_F are included. It is known from a beam experiment that in the case of mixed halocarbons containing both fluorine and chlorine or bromine Cl^-or Br^- are the main ions formed at thermal electron energies and F^- is only the minor product [24]. Therefore —$CFCl_2$ and —CF_2Cl seem to be main attaching centers with some, smaller, influence of halogen atoms at neighbor carbon. More experimental data are required to deal with this problem.

Activation energy for the thermal electron attachment processes is measured rather seldom, and the values for the same compound from different laboratories differ by more than 100%. However, one set of data on the temperature dependence of the electron capture rate constant was measured with the same method for several halogen substituted methanes [25]. Using these data we calculated the corresponding activation energies and have shown [21,22] that the activation energy indeed decreases strongly with

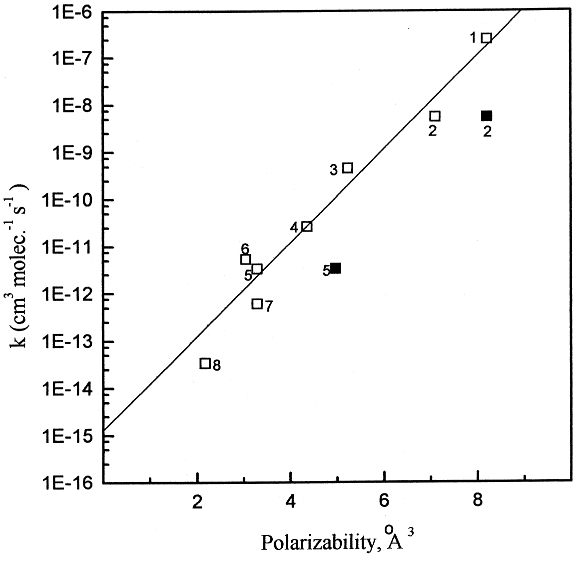

Fig 5.The dependence of $k_{\text{two-body}}$ on the electron polarizability of the attaching center for: 1-1,1,1-$C_2F_3Cl_3$, 2-1,1,2-$C_2F_3Cl_3$, 3-1.2-C_2H_4ClBr, 4-1,2-$C_2H_4Cl_2$, 5-C_2F_5Cl, 6-C_2H_rBr, 7-1,1,1-$C_2H_3F_2Cl$, 8-C_2H_5Cl, (solid points — see text).

α_{center}. Thus, the observed increase in log(k) with α_{center} seems to indicate that the polarizability of the attaching center influences mainly the activation energy of the process.

On the basis of our findings we can make the following statements:

1. Electron capture by halocarbons does not involve any particular halogen atom as an isolated species but the attaching center includes neighbor halogen atoms.
2. The dependence of k on calculated α_{center} values gives a good tool to predict and verify thermal electron capture rate constants.
3. Provided reliable rate constants are available for a given set of compounds, we can plot α_{center} versus log(k) and find the resulting polarizability of any particular attaching center from the obtained straight line.
4. The last statement opens a promising opportunity for theoretical work on the mechanism of the electron capture process and the structure of the attaching center.

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