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The influence of the temperature on electron attachment to some halocontaining molecules

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ABSTRACT

Thermal electron attachment rate coefficients for $CH_2ClCHClCH_2Cl$, CH_2ClCH_2Br, $CH_2ClCH_2CH_2Br$ and $CF_2ClCFCl_2$ have been measured using the Pulsed Townsend technique over the temperature range $T = 298–358$ K. The corresponding rate coefficients at 298 K are equal to $1.7(4) \times 10^{-10}$, $7.9(15) \times 10^{-10}$, $3.4(5) \times 10^{-10}$ and $3.1(1) \times 10^{-8}$ cm^3 molec.$^{-1}$ s^{-1}, respectively. With rising temperature we have observed an increase of the rate coefficients $k(T)$, which shows an Arrhenius-type behaviour. Activation energies E_a of 0.16(1) eV ($CH_2ClCHClCH_2Cl$), 0.12(1) eV (CH_2ClCH_2Br), 0.20(1) eV ($CH_2ClCH_2CH_2Br$) and 0.08(1) eV ($CF_2ClCFCl_2$) were determined from the fit to the experimental data points with a function $\ln(k) = \ln(A) - E_a/k_BT$.

1. Introduction

Electron attachment processes of halocontaining molecules have been extensively studied for several years and produced a wealth of knowledge [1–4]. A major reason for this interest results from their particular technical properties such as high volatility, high stability, low toxicity or non-flammability. Due to these safety criteria they were widely used as refrigerants, metered dose inhalers for the treatment of asthma, solvents for precision electronic cleaning, foam blowing. Nowadays, they play a significant role in many other modern technologies such as gas discharge lasers, plasma etching industry or in gaseous dielectrics for electric power transmission [5,6]. Halocontaining molecules often possess substantial dipole moments and slow down the electrons very efficiently. In mixtures of SF_6 and RX (where X represents one or more halogen atoms, in particular fluorine) these slow electrons are subsequently captured by the effective electron scavenger SF_6, forming stable SF_6^- anions, which in turn determines the good dielectric properties by increasing the breakdown strengths. For the plasma etching industry it is important to know which of these gases readily form negative ions at relatively low electron energy and with relatively high cross-section. It is also important how the cross-section of the ion formation is influenced by the gas temperature.

The present work is a continuation of our previous studies [7] where we have presented the results on fundamental quantities such as rate coefficients (k) and activation energies (E_a) for thermal electron capture by some chloroalkanes obtained by means of a time-resolved electron swarm method. We have demonstrated that there exists a linear dependence between $\log(k)$ and E_a. Here we present the results on the rate coefficients and activation energies for the process of electron capture by the four halocontaining aliphatic hydrocarbon molecules: $CH_2ClCHClCH_2Cl$, CH_2ClCH_2Br, $CH_2ClCH_2CH_2Br$ and $CF_2ClCFCl_2$.

2. Experimental

The experimental procedure of the Pulsed Townsend (PT) technique has been described in detail elsewhere [7]. Briefly, the experimental setup consists of the stainless steel chamber of 700 cm^3 volume with two parallel electrodes. The chamber can be heated using heating jackets produced by Watlow Company. Electronic control enables us to stabilize the temperature within 1 °C. An electron swarm is produced at the cathode using a 5 ns Nd:YAG laser operating on fourth harmonic (266 nm, 10 Hz). The electron swarm drifts to the anode under the influence of the uniform electric field through the gas mixture containing carbon dioxide as a buffer gas and an electron acceptor of a certain concentration to obtain the rate of electron disappearance in the range of 10^5 s^{-1}.

The drifting electrons create a pulse change in the potential of the collecting electrode. This pulse is amplified by preamplifier, registered on the oscilloscope and saved in computer memory. In

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the presence of pure CO_2 the potential change linearly increases with the drift time. The admixture of the electron acceptor exponentially changes the pulse shape due to removing the number of electrons from the swarm. The pulse is also changed by decay off in the preamplifier circuit, however, this influence is small owing to large time constant $t_1 = RC$ of the preamplifier (400 μs) as compared with time of flight of electrons between the electrodes $t_0 < 10$ μs. Altogether, the observed time dependence of the potential can be described by Eq. (1) [7]:

$$V(\tau) = B\left[\exp(-kN_a\tau) - \exp\left(\frac{-\tau}{t_1}\right)\right] \tag{1}$$

here B is an apparatus arbitrary constant, k is an electron capture rate coefficient, N_a is an electron acceptor concentration, t_1 is the decay time as define above. As all the values except B and k are known the computer task is to simulate them. Since B and k influence the potential very differently, the simulation gives unambiguous results provided the electron acceptor concentration and temperature are such that the rate of electron disappearance is in the range of $(0.2–1) \times 10^5\ s^{-1}$.

In general, the shape of the pulse can be deformed by the longitudinal and transverse diffusions (which are equal at the applied range of E/N [8]) of electrons during the drift of the swarm. However, taking into account the values of the applied potential across the gap, the drift time of the electron swarm and electron mobility these were shown to be negligible [7]. In this experiment even at elevated temperature the widening of the electron swarm is not more than 30%.

The RX–CO_2 gas mixtures were prepared inside the chamber by first injecting the minority gas and then introducing the buffer gas to obtain total pressures of the gas mixtures of around 300–350 Torr. The experiments were carried out for a few different initial concentrations of halocontaining molecules in carbon dioxide. A number of 100 pulses were registered for a given density reduced electric field value (E/N) and then averaged and analyzed. The procedure was repeated for several E/N values in the range of $(1.5–3.5) \times 10^{-17}\ V\,cm^2\ molec.^{-1}$, where electrons in carbon dioxide are in thermal equilibrium with gas molecules.

CO_2, from Fluka, with quoted purity of 99.998% was used without further purification. The investigated compounds and respective purities are as follows: $CH_2ClCHClCH_2Cl$ (Aldrich, 99%), CH_2ClCH_2Br (Merck-Schuchard, >98%), $CH_2ClCH_2CH_2Br$ (Acros Organics, 99%) and $CF_2ClCFCl_2$ (Merck-Schuchard, >98%). All of these chemicals were used after degassing by freeze–pump–thaw cycles with liquid nitrogen.

3. Results and discussion

The present data were obtained using the recently established swarm method, known as the Pulsed Townsend technique, that allows us to study electron attachment processes at elevated temperature. We have measured thermal electron capture rate coefficients $k(T)$ for the electron capture processes by the following molecules: $CH_2ClCHClCH_2Cl$, CH_2ClCH_2Br, $CH_2ClCH_2CH_2Br$ and $CF_2ClCFCl_2$ over the temperature range $T = 298–358$ K. T refers to both the electron temperature and the gas temperature ($T = T_e = T_G$). It is well known from the beam experiments that for the halocontaining aliphatic hydrocarbon molecules this is exclusively dissociative electron attachment (DEA) leading mainly (especially at thermal energy) to the halogen negative ions. Since we monitor the disappearance of the electrons from the swarm, as mentioned above, the obtained rate coefficients and activation energies correspond to DEA processes including all the reaction channels. The presently obtained results in terms of $\ln(k)$ vs. $1/T$ for all investigated molecules are shown in Fig. 1. They exhibit a linear dependence, which means that in the temperature range used in our studies an Arrhenius equation is valid. The validity of the Arrhenius equation for dissociative electron attachment (DEA) rate coefficients was recently theoretically investigated by Fabrikant and Hotop [9]. Using the resonant R-matrix theory they have estimated an upper temperature bound for the range of validity of the Arrhenius equation in the endothermic case and both lower and upper bounds in the case of the exothermic processes with a reaction barrier. It appears that for the exothermic case with the barrier the range of the validity of the Arrhenius equation is much narrower than in the endothermic one. The rate coefficients obtained for the former case at low-temperature limit are nearly constant due to the fact that the attachment occur from the $n = 0$ vibrational state at low electron temperature. At high-temperature limit, a significant amount of the electrons have velocities above the threshold velocity for levels n below the barrier, which results in levelling off of the exponential rise toward high temperature and eventually in decreasing above this limit. This was, however, not the case for any of the presently investigated molecules in the covered temperature range.

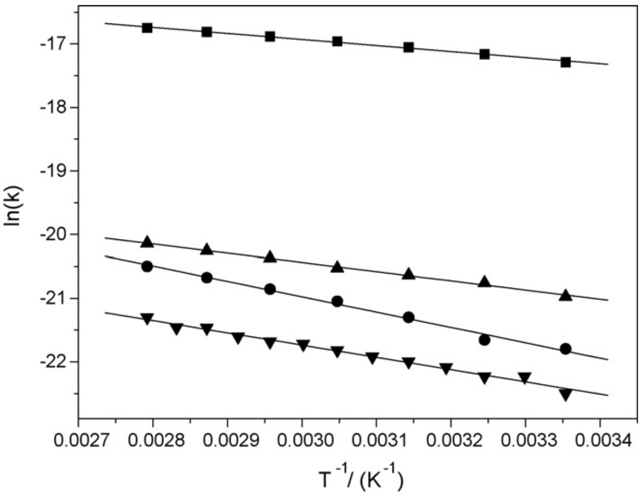

Fig. 1. The dependence of $\ln(k)$ on $1/T$ for all studied halocontaining molecules: (▼) $CH_2ClCHClCH_2Cl$; (▲) CH_2ClCH_2Br; (●) $CH_2ClCH_2CH_2Br$; (■) $CF_2ClCFCl_2$.

From the fit to the experimental data points with a function $\ln(k) = \ln(A) - E_a/k_BT$ the activation energies E_a were determined. As was pointed out by Fabrikant and Hotop [9] the activation energy of the exothermic reaction and for $T = T_e = T_G$, which is relevant for the present experiments, is somewhat lower than the barrier height, that is the crossing energy of the potential curves. This results from the tunneling that can take place from vibrational level below the barrier, which means that the activation energy can be regarded as an effective barrier energy for the dissociative electron attachment process. All of the presently obtained results are collected in Table 1 together with our recent data [7] from PT technique and available literature ones. The rate coefficients for $CH_2ClCH_2CH_2Br$, CH_2ClCH_2Br and $CF_2ClCFCl_2$ were measured previously [10,11] at our laboratory by means of the swarm method with an ionization chamber, however, only at room temperature. There is a good agreement between the previous value of the thermal electron capture rate coefficient obtained for $CH_2ClCH_2CH_2Br$ ($2.5 \times 10^{-10}\ cm^3\ molec.^{-1}\ s^{-1}$ [10]) and the present value while the value for CH_2ClCH_2Br is somewhat higher than the previous one ($4.5 \times 10^{-10}\ cm^3\ molec.^{-1}\ s^{-1}$ [11]). There is a surprisingly big difference, of one order of magnitude, between the previous value of the rate coefficient ($5.4 \times 10^{-9}\ cm^3\ molec.^{-1}\ s^{-1}$ [11]) and the presently derived value for $CF_2ClCFCl_2$ and at present we have no explanation for this discrepancy. However, the newly obtained value of $3.1(1) \times 10^{-8}\ cm^3\ molec.^{-1}\ s^{-1}$ for

Table 1

Thermal electron attachment rate coefficients ($T = 298$ K) and activation energies (E_a) calculated from kinetic data for some halogenated aliphatic hydrocarbons together with the literature data (The values printed bold are included in Fig. 2). T_{range}—the temperature range for which E_a was obtained.

	Molecule	$k_{(298\,\mathrm{K})}$ (cm^3 molec.$^{-1}$ s^{-1})	E_a (eV)	T_{range} (K)
1	CH_3Cl	$\mathbf{<1.9\times10^{-15}}$ [19]	$\mathbf{0.54(2)}$ [21]	303–493
		$<1\times10^{-16}$[a] [20]		
2	CH_3Br	$\mathbf{6.78(11)\times10^{-12}}$ [22]	$\mathbf{0.260(15)}$ [22]	293–500
		$7.0(4)\times10^{-12}$ [19]	0.25(2) [21]	303–493
3	CH_2Cl_2	$\mathbf{1.7\times10^{-12}}$[c] [23]	$\mathbf{0.29(10)}$ [24]	298
4	$CHCl_3$	$\mathbf{3.9(2)\times10^{-9}}$ (300 K) [25]	$\mathbf{0.11(1)}$ [25]	295–373
4a		$\mathbf{4.4(7)\times10^{-9}}$ (300 K) [26]	$\mathbf{0.11(1)}$[b] [26]	300–590
5	CCl_4	$\mathbf{3.79\times10^{-7}}$ (294 K) [27]	$\mathbf{0.00(5)}$ [24]	373
		$3.9(6)\times10^{-9}$ (300 K) [26]	−0.003 [26]	205–455
6	CH_3CHCl_2	$\mathbf{2.1\times10^{-11}}$[c] [23]	$\mathbf{0.25(10)}$ [24]	298
7	CH_2ClCH_2Cl	$\mathbf{3.2\times10^{-11}}$[c] [23]	$\mathbf{0.24(10)}$ [24]	298
8	CH_3CCl_3	$\mathbf{1.5(3)\times10^{-8}}$ [13]	$\mathbf{0.109}$ [13]	298–385
8a		$\mathbf{3.8\times10^{-8}}$ [7]	$\mathbf{0.09}$ [7]	298–373
9	$CHCl_2CH_2Cl$	$\mathbf{3.7\times10^{-10}}$ [7]	$\mathbf{0.16}$ [7]	298–343
9a		$\mathbf{3.1(6)\times10^{-10}}$ [13]	$\mathbf{0.128}$ [13]	298–385
10	$CH_3CHClCH_2CH_3$	$\mathbf{2.0\times10^{-15}}$ [7]	$\mathbf{0.55}$ [7]	343–373
11	$CH_2ClCHClCH_2Cl$	$\mathbf{1.7(4)\times10^{-10}}$[d]	$\mathbf{0.16(1)}$[d]	298–358
12	CH_2ClCH_2Br	$\mathbf{7.9(15)\times10^{-10}}$[d]	$\mathbf{0.12(1)}$[d]	358
		4.5×10^{-10}[e] [11]		
12a		$\mathbf{1.0(3)\times10^{-9}}$ [12]	$\mathbf{0.156(3)}$ [12]	298–475
13	$CH_2ClCH_2CH_2Br$	$\mathbf{3.4(5)\times10^{-10}}$[d]	$\mathbf{0.20(1)}$[d]	298–358
		$2.5(2)\times10^{-10}$[e] [10]		
14	$CF_2ClCFCl_2$	$\mathbf{3.1(1)\times10^{-8}}$[d]	$\mathbf{0.08(1)}$[d]	298–358
		5.4×10^{-9}[e] [11]		298
14a		$\mathbf{1.1(2)\times10^{-8}}$ [13]	0.08(10) [24]	298–385
		1.2×10^{-8} [28]	$\mathbf{0.068}$ [13]	

[a] Theoretical value from semiempirical calculations of DA.

[b] From fit to data provided at [26]; these authors quoted 0.12 eV.

[c] Recommended values of the rate coefficients obtained at $T = 295$–300K.

[d] Present data.

[e] Rate coefficients obtained at ambient temperature.

$CF_2ClCFCl_2$ is in close agreement with the literature ones (see Table 1). To our knowledge there is no available literature data for $CH_2ClCHClCH_2Cl$.

The CH_2ClCH_2Br [12] and $CF_2ClCFCl_2$ [13] molecules have been also studied using the flowing afterglow/Langmuir probe (FALP) technique with a helium bath gas. Smith et al. have determined both the attachment coefficients and the activation energies of the dissociative electron attachment reactions (see Table 1). Comparison of the PT and FALP results (at the temperature range close to that covered in the present work) has shown that both the activation energy and thermal electron capture rate coefficient/attachment coefficient for $CF_2ClCFCl_2$ are in fairly good agreement while the presently obtained values for CH_2ClCH_2Br are somehow lower than the FALP results. Smith et al. [12,13] also identified the product ions arising from the electron interactions with these molecules. The obtained results confirmed the earlier findings that the capture of the thermal electrons by halogenated aliphatic hydrocarbons leads exclusively to the formation of the atomic halide ions. And so for the CH_2ClCH_2Br the anions Br^- and Cl^- were observed, with Br^- being the major product at every applied temperature. In the case of $CF_2ClCFCl_2$ the only observed product was Cl^-.

The correlation between log(k) and E_a is shown in Fig. 2. It can be seen that this is, to good approximation, a linear dependence. A dependence of the rate coefficient of a chemical reaction on temperature is described by the Arrhenius equation (2):

$$\ln(k) = \ln(A) - \frac{E_a}{k_B T} \tag{2}$$

where E_a is the activation energy and A is the preexponential factor. If activation energy equals to zero then the rate coefficient is equal to A, which can be considered as rate coefficient for electron capture if each collision leads to products. The well known example of such situation is the electron capture by CCl_4 for which $E_a = 0$ eV and $k = 3.79 \times 10^{-7}$ cm^3 molec.$^{-1}$ s^{-1} [27].

There are different quantitative interpretations of the scattering processes that are closely related to the structure of the target molecular. For the case of the halogen derivatives of hydrocarbons where only s-waves take a part in scattering at low electron energy one theory gives the maximum reaction cross-section as $\pi(\lambda/2\pi)^2$, where λ is the de Broglie wavelength of the electron. From that one can arrive in the theoretical limit for the attachment rate coefficient 5.0×10^{-7} cm^3 molec.$^{-1}$ s^{-1} at 298 K, which corresponds to the factor A in the Arrhenius Eq. (2). However,

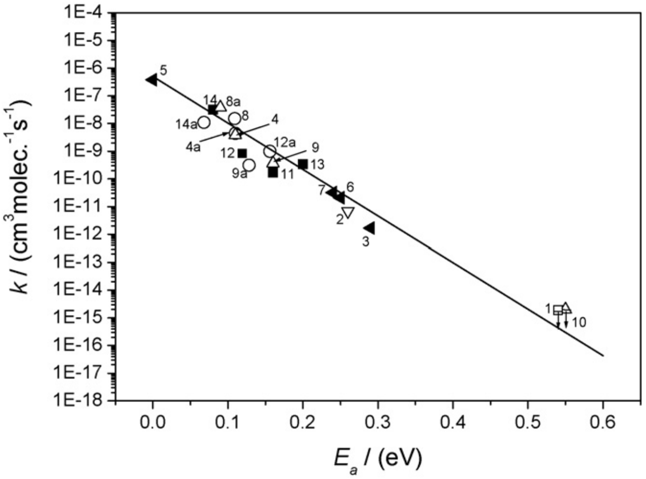

Fig. 2. Thermal electron capture rate coefficients as a function of the activation energies for some halogenated hydrocarbons: (■) present data, (△) our previous data from [7,25], (○) data from Smith et al. [12,13,26], (▽) data from Petrović and Crompton [22], (◀) recommended values of the rate coefficients from [23] vs. E_a from [24], (□) an upper limit (1.9×10^{-15} cm^3 molec.$^{-1}$ s^{-1}) of the rate coefficient for CH_3Cl [19] vs. E_a from [21] (solid line—theoretically obtained values of the rate coefficients for $T = 298$ K considering electron as the de Broglie wave).

recently Fabrikant and Hotop [14,15] in a discussion of dissociative electron attachment at the threshold energy have demonstrated that the cross-section depends on energy as $E^{l-1/2}$, according to the Bethe–Wigner threshold law for nonpolar molecules, where l is the lowest allowed angular momentum quantum number. For halogen derivatives of hydrocarbons this gives the $E^{-1/2}$ law in the case of s-wave attachment. On the other hand, a different approach is used in Vogt–Wannier (VW) theory for the attachment into polarization wall. In this model it is assumed that the cross-section depends only on energy and the molecular polarizability. VW formula, however, does not contain a resonance width althought the DA is a resonance process. On the base on VW theory and its extention by Fabrikant and Hotop [15]. Troe and coworkers [16] have recently provided analytical approximations for cross-sections and thermal rate coefficients for capture of electrons by polar and polarizable molecules. The calculations performed with this approach for a number of molecules have shown that the contribution of either dipole moment and quadrupole moment of the target molecule to the rate coefficients is small. However, as authors stated, these approximations provide an upper bound to the experimental rate coefficients. This results from the fact that in this approach the formation of the resonances is due to the interaction of the target molecule with incoming wavepacket while the outgoing wavepacket is assumed to be attenuated by intramolecular vibrational redistribution. Therefore some of the results obtained this way (see Table 1 in [16]) overestimate attachment rate coefficients and a close agreement with the experimental values of the rate coefficients is observed for such systems as, e.g., CCl_4, SF_6, where the activation energy for electron attachment is 0 eV.

At constant temperature Eq. (2) gives linear dependence (with negative slope) of the $\log(k)$ on E_a. This curve is shown in Fig. 2 as a solid line together with the experimental data collected in Table 1. It is seen that the most of the data lie well nearby this curve. This shows that indeed the activation energy is the main factor determining the rate coefficient for thermal electron capture. This, in turn, can offers a good criterion for judging the quality of the experimental results if both the rate coefficient and activation energy are known. When the results lie far from the curve one should consider an experimental error, especially if they are the sole data and if the rate coefficient and the activation energy were measured using different techniques. If the experimental error should be excluded then the factors influencing the A value must be taken into account. For example, fast autoionization of the temporary negative ion comes here in mind and/or the polarizability of the molecule as was proposed by Troe and coworkers [16]. The comparison with the results of dissociative electron attachment in crossed beams (the position and cross-section for DEA peaks) and vertical attachment energy VAE data might help in this task. In the recent papers Burrow and coworkers [4,17,18] have shown that the zero energy peak DEA cross-sections in two families of molecules polychloroalkanes and chlorofluoromethanes well correlate with the VAEs for formation of the lowest temporary anion states. They have observed the exponential decreases of the peak DEA cross-sections with increasing the VAE. Additionally, they have shown the linear relationship between the VAE, which describes the formation of the transient anion, and the energy of the crossing point between the potential energy surfaces of the neutral molecule and anion, and thus the activation energy.

4. Conclusions

Results from the study of thermal electron capture by $CH_2ClCHClCH_2Cl$, CH_2ClCH_2Br, $CH_2ClCH_2CH_2Br$ and $CF_2ClCFCl_2$ in the gas phase are reported. The measurements have been performed using Pulsed Townsend technique in the temperature range $T = 298–358$ K ($T = T_e = T_G$), the determined rate coefficients for thermal electron capture processes show an Arrhenius-type rise with increasing the temperature. The corresponding rate coefficients at 298 K are equal to $1.7(4) \times 10^{-10}$, $7.9(15) \times 10^{-10}$, $3.4(5) \times 10^{-10}$ and $3.1(1) \times 10^{-8}\ \mathrm{cm^3\ molec.^{-1}\ s^{-1}}$, respectively. The semi-log dependence of the rate coefficients as a function of activation energies shows, to good approximation, a linear behaviour. This dependence agree well with the theoretically obtained values of the rate coefficients (solid line in Fig. 2) considering electron as the de Broglie wave. From this dependence we may conclude that the activation energy is the main factor determining the rate coefficient. There is, however, still little data on the influence of the temperature on electron attachment processes and experimental work on further halocontaining molecules is required.

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