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Absolute calibration of relative electron attachment cross sections measured by crossed-beams experiments

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Abstract

Relative dissociative electron attachment cross sections measured in crossed beams apparatus may be placed on an absolute scale using absolute attachment rate constants measured in swarm type experiments. We discuss here possible pitfalls and in particular the influence of the shape of the cross section curve on the necessary integration involved in such a calibration procedure. Using as an example recently measured data for CCl_2F_2 and CCl_3H we will demonstrate the importance of using a full integration method instead of previously used simple methods involving for instance the relationship $\sigma_{\{\epsilon\}} = k(T)/\langle v\rangle$. (Int J Mass Spectrom 205 (2001) 77–84) © 2001 Elsevier Science B.V.

Keywords: Electron attachment; Cross sections; Rate constants; Absolute calibration

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1. Introduction

Attachment of electrons to molecules is an elementary process, which is considerably influencing the macroscopic properties of natural and manmade plasmas such as the earth's ionosphere or gas discharges [1,2]. Moreover, investigations of electron attachment are of fundamental importance to the understanding of electron-molecule interactions and the mechanisms of negative ion formation [1–4].

There exists a variety of techniques used for such studies which can in principle be divided into two categories. In swarm techniques, which trace back to the beginning of the century, either the attachment rate constant k usually expressed in units of $cm^3\ s^{-1}$ or $cm^6\ s^{-1}$ (depending on the character of the attachment process) or the density reduced electron attachment coefficient η/N usually expressed in units of cm^2, are customarily measured as a function of the gas or electron temperature, or the reduced electric field E/N (for details of the definition see [5]). The data obtained from swarm experiments, however, are of

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

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limited value, because for every E/N and gas temperature T, the corresponding electron energy distribution function $f(\epsilon)$ depends on the gas under study [6]. Thus the measured values k or η/N correspond to the specific "ensemble" or "swarm" of electrons used having a specific distribution of energies. An unfolding procedure to obtain cross sections from measured rate constants introduced by Christophorou and coworkers [6,7] (see also [8]) overcomes this deficiency by using a binary gas mixture, in which a small amount of the electron attaching gas is diluted in a nonattaching buffer gas. If the electron energy distribution function in the carrier gas is well known and is not influenced by the small amount of the electron attaching gas to be studied, the "monoenergetic" attachment rate constant $k(\epsilon)$ as well as the attachment cross section $\sigma(\epsilon)$ and their dependencies on the electron energy ϵ can be derived from the experimental data.

Whereas in swarm experiments the collective behaviour of electrons is dominant, in the second category of electron attachment studies, the interaction of electrons under single collision conditions is investigated in electron beams experiments using either a stagnant target gas or a molecular beam. Many highly sophisticated techniques have been developed for this purpose over the past three decades [2,4,9–13], thus allowing to determine directly and with high precision the electron attachment cross section and its dependence on the electron energy $\sigma = \sigma(\epsilon)$. For instance, such electron beam methods have been used for the measurement of the absolute total ionisation and electron attachment cross sections [14,15] as well as for the observation of the total electron-scattering cross section [16] for CCl_2F_2. These measurements were performed without mass analysis, which has the drawback that the identity of the anions remains unknown. On the other hand it is in such a case possible to determine directly absolute cross sections, as all the parameters necessary for this (ion current produced, electron current used, electron path interaction length and gas density) can be determined with sufficient accuracy (for systematic errors present in these determinations see [15]). Alternatively, this attachment cross section has been measured recently with crossed/beams mass spectrometer systems [17–19], which on the one hand have the advantage of identifying the anion products, but on the other hand usually only allow to determine relative cross sections due to difficulties in measuring accurately some of the parameters necessary for the determination of absolute cross sections [20]. Relative cross section curves measured with this technique are difficult to calibrate in a universal and reliable way and therefore (see also the discussion above about the systematic errors in [15]) it is not too surprising that data determined for CCl_2F_2 in [17–19] differ considerably from results reported in [15]. Some of the normalization methods used (in particular concerning also threshold photoelectron attachment spectra [21,22]) have been discussed in [11].

Relative dissociative electron attachment (DEA) cross sections measured in crossed beams instruments are usually placed on an absolute scale using absolute attachment rate constants measured in swarm type experiments. We discuss here possible pitfalls and in particular the influence of the shape of the cross section curve on the necessary integration involved in such a calibration procedure. Using as an example recently measured data for CCl_2F_2 [17] and CCl_3H [23] we will demonstrate the importance of using the full integration method as described by Chutjian and Alajajian [22] and Hotop and co-workers [21] instead of previously used simple methods (invoking for instance the relationship $\sigma_{\{\epsilon\}} = k(T)/\langle v\rangle$).

2. Theoretical considerations

Relative DEA cross sections, σ_a, can be placed on an absolute scale by reference to corresponding absolute rate coefficients, k, determined for instance in a flowing afterglow Langmuir probe (FALP) apparatus [25]. The rate coefficient for electron attachment, k, and the attachment cross section σ_a are related as follows:

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[1] It is important to note, that in a follow-up publication [24] to [23] the absolute cross section scale in Fig. 8 of this publication [24] has been mislabeled resulting in cross section values which are approximately a factor of about 2 too low. In the present report the correct cross sections are reported in accordance with the earlier work [23].

$$k(T_e) = \frac{\int_0^\infty \sigma_a(v)\, f(v,\, T_e)\ v\ dv}{\int_0^\infty f(v,\, T_e)\ dv} \tag{1}$$

In Eq. (1), T_e is the electron temperature, v is the velocity of the attaching electrons, and $f(v)$ is their energy distribution which is assumed to be a Maxwell-Boltzmann distribution. Given that the denominator of Eq. (1) is unity (in the case of a normalized Maxwell-Boltzmann distribution), the equation may be rewritten as a function of the electron energy ϵ as follows:

$$k(\epsilon) = \sqrt{\frac{2}{m_e}} \int_0^\infty \sigma_a(\epsilon)\epsilon^{1/2} f(\epsilon)\ d\epsilon \tag{2}$$

where m_e is the electron mass and the normalized Maxwell-Boltzmann energy distribution of the attaching electrons is

$$f(\epsilon) = 2\pi^{-1/2}(k_B T_e)^{-3/2}\epsilon^{1/2} \exp\left(-\frac{\epsilon}{k_B T_e}\right) \tag{3}$$

Assuming that (1) the extraction of the product anions from the collision region, and that (2) the mass analysis and that (3) the ion detection efficiency are all independent of the electron energy (which is not always the case [17,23,24]), then the cross section $\sigma_a(\epsilon)$ can be expressed (see also Illenberger and Niehaus, 1975 [26]); Chutjian and Alajajian 1985 [22] and Klar et al. 1992 [21]) as

$$\sigma_a(\epsilon) = cS^-(\epsilon) \tag{4}$$

where $S^-(\epsilon)$ is the measured (relative) anion current (normalized to the incident electron current) and the constant c is given by

$$c = \frac{k(T)\sqrt{\frac{\pi m_e}{8}}}{\int_0^\infty S^-(\epsilon)(k_B T_e)^{-3/2}\epsilon \exp\left(-\frac{\epsilon}{k_B T_e}\right) d\epsilon} \tag{5}$$

Thus, the absolute attachment cross section $\sigma_a(\epsilon)$ can be determined from the rate coefficient k determined at the appropriate gas temperature T_g in a FALP experiment (where the gas temperature Tg is assumed to be equal to the electron temperature T_e), if Eq. (5) can be solved accordingly.

Note that the evaluation of this integral requires that the quantity $S^-(\epsilon)$ has been measured or is known over a sufficiently broad energy interval, both towards high and low electron energies. For instance certain high resolution experiments only allow measurements of the cross section curves up to a limited energy range of up to about several 100 meV. Conversely, as pointed out before [22], problems arise when limited energy resolution does not allow to measure the true cross section shape down to very low energies, i.e. energies lower than about the resolution width. In order to account for this effect of finite resolution[2], Chutjian and Alajajian [22] assumed a model fit function for the cross section shape, with some adjustable parameters (an intrinsic weakness of this analytical cross section function, for instance, is that the exponential decay is not cut off at low energies). This at the same time facilitates the necessary integration in Eq. (5). For instance, a two term approximation

$$S^- = \left[a\epsilon^{-1/2} \exp\left(-\frac{\epsilon^2}{\lambda^2}\right) + \exp\left(-\frac{\epsilon}{\gamma}\right)\right] \tag{6}$$

has been used for the Cl^- anion threshold cross-section behaviour of various chlorine containing gases by Chutjian and Alajajian [22]. The free parameters were determined in this case by a computer fit to the

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[2] So far the only experiments in which the relative cross sections were obtained with a sufficiently low energy width (smaller than 1 meV) and down to sufficiently low energies (though only up to about 150 meV) to allow direct integration of Eq. (5), were those of Klar et al. [21] and more recently by Schramm et al. [27]. In order to demonstrate the fractional contributions to the integral in Eq. (5), in particular the importance of the very low energy regime, Klar et al. [21] plotted for the case of SF_6^- formation via electron attachment to SF_6 the accumulated rate coefficient (i.e. integration from zero electron energy to a certain finite value of the electron energy; see Fig. 13 in [21]). Thus they were able to show that the energy range below about 22 meV already accounts for 50% of the total rate coefficient at $T = 300$ K.

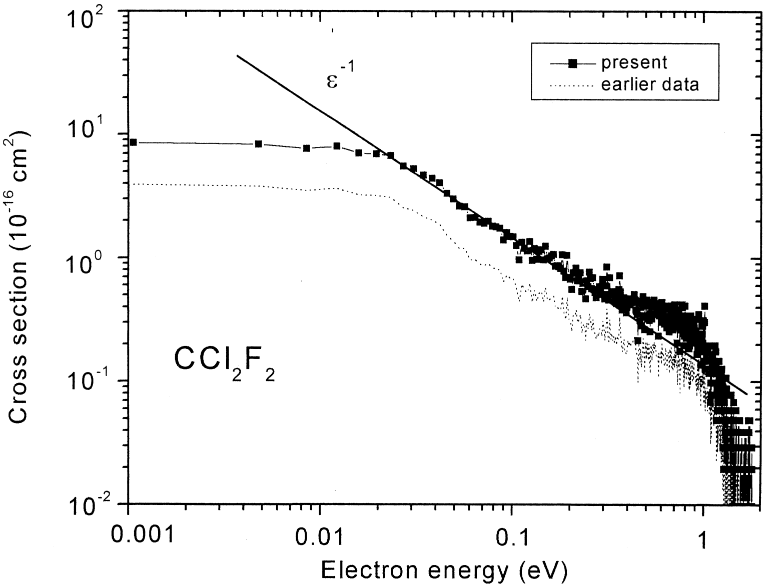

Fig. 1. Absolute electron attachment cross section vs. electron energy for the reaction $CCl_2F_2 + e \rightarrow Cl^-$. The present data (full line with full squares) were derived by using the calibration method outlined in the text [Eqs. (4) and (5)] involving integration of the measured anion signal. The earlier data (dashed line) are from [17] and have been obtained by [17] using the simple procedure employing Eq. (7). Also shown as full line designated ϵ^{-1} the predicted energy dependence for *s*-wave scattering.

experimental data.[3] As a check to the accuracy of the method used, the cross sections obtained were used to calculate via Eq. (2) the rate coefficient k. This calculated rate coefficient was compared to the experimental rate coefficient used to generate the cross sections. By iteratively improving the fitting procedure for Eq. (6) [22] it was possible to obtain agreement between the original experimental k value and the calculated k value within an accuracy of 0.1%.

In order to avoid this complicated procedure sometimes a much simpler approach (see also [29,30]) has been employed [17,23,31] using instead of Eq. (2) the simple relation (see also the following for its rationalization)

$$\sigma_{\{\epsilon\}} = \frac{k(T)}{\langle v \rangle}, \tag{7}$$

where $\sigma_{\{\epsilon\}}$ is the value of the cross section at the (so called plasma [32]) energy of electrons $\{\epsilon\} = k_B T_e$, $\langle v \rangle$ is the mean thermal velocity of electrons $\langle v \rangle = [8k_B T_e/\pi m_e]^{1/2}$ at an electron temperature T_e and $k(T)$ the reaction rate constant determined for instance by the FALP technique at the gas temperature T_g (assuming that $T_g = T_e$). Using the measured cross section data reported recently for CCl_2F_2 [17] and CCl_3H [23] from our laboratory (see Figs. 1 and 2) it turns out that the absolute cross sections derived by using the simple approach (7) may deviate from the cross sections derived by the use of Eqs. (4) and (5). We will explore below the reasons for this in more detail; one reason may be attributed to problems arising from the limited and also differing energy resolution of 60 meV in [17] and 30 meV in [23], see discussion above. Moreover we will define situations (not regarding the trivial case of constant cross sections) where nevertheless this simple method can be used safely.

As can be seen from the log-log plots in Figs. 1 and 2, the attachment cross section function $\sigma(\epsilon)$ for CCl_2F_2 and CCl_3H consists in both cases of three different parts, exhibiting different slopes n, as defined by $\log \sigma(\epsilon) \propto -n \log \epsilon$. In accordance with

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[3] It is interesting to note that as shown by Chutjian [28] the use of a Druyvesteyn energy distribution instead of the Maxwell-Boltzmann energy distribution will decrease the rate coefficient k calculated via equ. (2) from the known $\sigma_a(\epsilon)$ by appr. 25%.

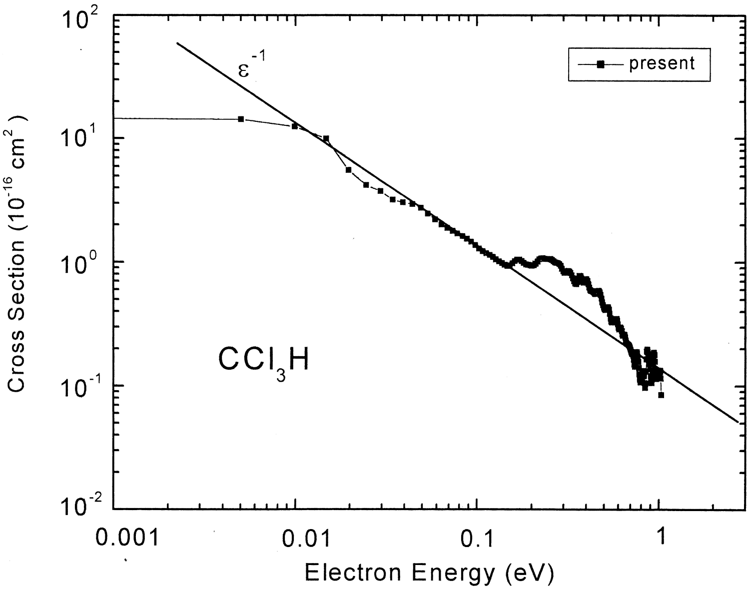

Fig. 2. Absolute electron attachment cross section versus electron energy for the reaction $CCl_3H + e \rightarrow Cl^-$. The present data (full line with full squares) were derived by using the calibration method outlined in the text [Eqs. (4) and (5)] involving integration of the measured anion signal. The earlier data obtained by Matejcik [23] using the simple procedure employing Eq. (7) are only 3% smaller than the present ones and thus are not shown for the sake of clarity. Also shown as full line designated ϵ^{-1} the predicted energy dependence for *s*-wave scattering.

predictions for de-Broglie *s*-wave scattering [2,3,21,24] n is close to $n \approx 1$ in the region between about 10 meV and several 100 meV, while at lower electron energies the values of n is smaller than 1 in accordance with predictions for the threshold behavior predicted by the Wigner law, $n = 0.5$ [21,33], and due to a modification of the true anion cross section by the convolution with the finite electron energy distribution (for details see [24] and the previous discussion). Contrary to this, at higher electron energies n is much larger than 1, reaching for example a value of $n \approx 6.5$ in the case of CCl_2F_2 [17]. Thus the attachment cross section can be approximated in this case by a set of functions having the form

$$\sigma(\epsilon) = A\epsilon^{-n} \tag{8}$$

Using relation (8) for $\sigma(\epsilon)$ and assuming a Maxwellian electron energy distribution Eq. (2) may be rewritten as follows:

$$k = \sqrt{\frac{8}{\pi m}}\{\epsilon\}^{-3/2}\int_0^\infty A\epsilon^{1-n}\exp\left[-\frac{\epsilon}{\{\epsilon\}}\right]d\epsilon \tag{9}$$

For $n = 0$, 0.5, 1, and 1.5 Eq. (9) can be solved analytically (using for $A = \sigma_{\langle\epsilon\rangle}\langle\epsilon\rangle^n$) and the integration yields $k = \sigma_{\{\epsilon\}}\cdot\langle v\rangle$, $\sqrt{\pi}/2\sigma_{\{\epsilon\}}\langle \mathrm{v}\rangle$, $\sigma_{\{\epsilon\}}\cdot\langle v\rangle$, and $\sqrt{\pi}\sigma_{\{\epsilon\}}\cdot\langle v\rangle$, respectively, where $\sigma_{\{\epsilon\}}$ is the value of the cross section at energy $\{\epsilon\} = k_BT_e$.

On a more general note, introducing $x = \epsilon/\{\epsilon\}$ Eq. (9) can be rewritten as follows:

$$k = \langle v\rangle\sigma_{\{\epsilon\}}\int_0^\infty x^{1-n}\exp[-x]\,dx = \theta(n)\sigma_{\{\epsilon\}}\langle v\rangle \tag{10}$$

where $\theta(n)$ is a dimensionless function of the exponent n. The corresponding results obtained by numerical integration are shown in Fig. 3 for values of n between 0 and 1.7. Fig. 4 giving the calculated $\langle v\rangle$ versus $x_{\max} = \epsilon_{\max}/\{\epsilon\}$ [with $\epsilon_{\max}$ the upper limit of electron energy used for the integration of Eq. (10)] demonstrates the fast convergence of the integral thus allowing us to use for the calculations in Fig. 3 as an upper limit for our calculation $x = 6$ for all values of $n \in (0–2)$.

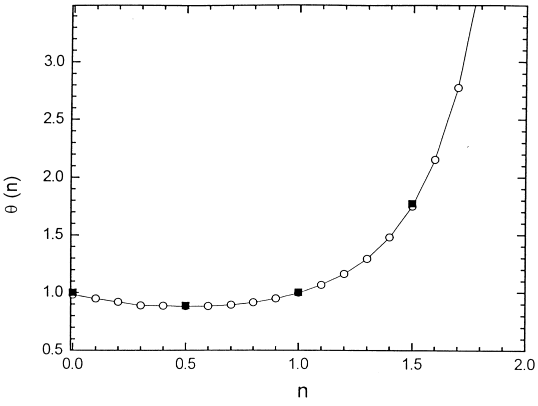

Fig. 3. $\theta(n)$ vs. n as calculated from Eq. (10). Full squares: analytical solutions; Open circles: numerical solutions for $\epsilon_{\max} = 6kT$.

From these results (see Fig. 3) it is immediately clear that only in the case that the energy dependence of the cross section can be described by relation (8) with $n = 0$ or $n = 1$ the simple calibration procedure using Eq. (7) can be used in a reliable manner. If the actual measured relative cross section behaviour deviates from this simple relation, it is necessary to use in accordance with the previous work in [21,22] (see also [34]) the method outlined at the beginning of this section using Eq. (4) and (5). In Sec. 3 we will apply this more involved integration procedure to the raw data of [17] and [23] and discuss the difference between the presently obtained absolute cross sections and the earlier ones given in [17,23] using Eq. (7) for the calibration.

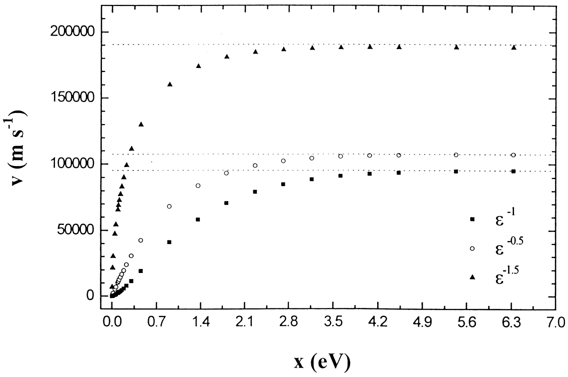

Fig. 4. $\langle v \rangle$ vs. $x_{\max} = \epsilon_{\max}/\{\epsilon\}$ for three different energy dependencies $\sigma(\epsilon) = A\epsilon^{-n}$.

3. Present results for absolute cross sections and activation energies for CCl_3H and CCl_2F_2

Using a newly constructed high resolution crossed electron/molecular beam apparatus [35,36] consisting of a trochoidal electron monochromator [12] and a quadrupole mass spectrometer we have recently measured the relative production cross sections for Cl^- via electron attachment to CCl_3H [23] and CCl_2F_2 [17] with energy resolutions of 30 and 60 meV, respectively. The relative Cl^- cross sections were placed in these earlier studies on an absolute scale (see Figs. 1 and 2) by reference to an absolute reaction rate constant at 300 K reported by Smith et al. [25] [$k(CCl_3H) = 4.4 \times 10^{-9}$ cm^3 s^{-1} and $k(CCl_2F_2) = 3.2 \times 10^{-9}$ cm^3 s^{-1}] using the above mentioned simple relation (7) with a value for the mean velocity $\langle v \rangle = [8k_BT_e/\pi m_e]^{1/2}$ of 1.076×10^5 m s^{-1} thereby obtaining $\sigma_{\{\epsilon\}}$ for the plasma energy $\{\epsilon\} = k_BT_e$ of 26 meV. As already mentioned previously this leads at least in the case of CF_2Cl_2 to an unreliable calibration.

Here we place these relative cross sections on an absolute scale (see Figs. 1 and 2) by reference to the same absolute reaction rate constant of Smith et al. [25] using, however, the more sophisticated calibration method outlined above [Eqs. (4) and (5)] involving integration of the measured anion signal at a temperature of $T = 300$ K. Following Chutjian and Alajajian [22] the reliability of the procedure was tested by calculating from the derived cross sections the rate coefficient thereby yielding very good agreement with the original rate coefficient of Smith et al. It should be mentioned, though, that calibrating the cross sections in this way the values of the measured $S^-(\epsilon)$ are weighted heavily in the low energy region of the cross section which lies within the experimental spread of the electron energy. In order to assess this error in the derived absolute values of $\sigma(\epsilon)$ we have calibrated (for details see [34]) the relative cross section using instead of the experimental determined energy dependence $S^{-}(\epsilon)$ close to zero (that is below about 20 meV) the ϵ^{-1} dependence expected for s-wave scattering. As discussed in [34] in this case the obtained absolute cross sections for the Cl^{-} production from CCl_2F_2 are about 30% smaller. As can be seen from a comparison of the present data with the earlier ones in Figs. 1 and 2 the changes in the cross sections when going from the simple to the more sophisticated procedure are different for the two examples, i.e., in the case of CCl_3H the present calibration leads to a cross section which is ~3% higher than the earlier one[4], whereas in the case of CCl_2F_2 the present calibration leads to values ~30% higher. On the one hand this is probably due to the different energy resolution (60 meV for CCl_2F_2, versus 30 meV for CCl_3H) and the slightly different shape at higher energies, on the other hand part of this may be due to the deviation of the cross-section energy dependence from the simple ϵ^{0} and ϵ^{-1} behavior.

Finally, it is important to point out that the success of all the calibration methods used for the determination of absolute cross sections from measured relative cross sections in crossed beams experiments depends on the reliability of the swarm data to be used as input for the various procedures (in the present case the used k values have an error quoted by the authors in the order of ±15% [25]); nevertheless other authors give quite different values [5]). As discussed in a recent review by Christophorou et al. [5] on data about electron attachment to CCl_2F_2 there exists unfortunately a large scatter between measured reaction rate constants reported by different authors and therefore a juidicious choice concerning the used input data is imperative for a successful normalization of relative attachment cross sections.

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