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Transmission of different ions through a drift tube

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ABSTRACT

Transmission through a drift tube has been measured for different types of ions. Various ions were injected at the same rate at the start of the drift tube. Then, the transmitted currents were measured at the end of the drift tube. The results show that transmission depends linearly on the inverse of the drift time. Factors influencing the drift time, such as pressure, temperature, and electric field, were also investigated and shown to affect the transmission efficiency in a similar manner as the type of ion. A simple model based on kinetics of two parallel processes (loss or transmission) is proposed to explain the results. Since slow-moving ions are lost more often than fast-moving ones, the intensity of ion peaks decreases as drift time increases. This can be easily corrected by transforming the *y*-axis to $y \times x$ (i.e., intensity × drift time).

1. Introduction

Drift tubes are widely used in the study of the kinetics and thermodynamics of ion–molecule reactions [1–9]. The progress of these reactions can be measured by initiating them in an ionization zone, passing the particles through a drift tube, and then measuring the reactant or product ions. The ions may be monitored either by mass spectrometry or other similar tools. The signal intensities measured are related to the number density of ions in the ionization zone.

Drift tubes are also used for producing and transporting ions at atmospheric or sub-atmospheric pressures. Examples are those instruments used for measuring VOCs based on proton transfer reaction mass spectrometry (PTRMS) [10–12]. In PTRMS, the ion source generates hydronium primary ions that pour into a drift tube. The analyte in the sample gas (VOCs) is ionized in the drift tube by proton transfer reaction. A fraction of primary and product ions are then extracted from the drift tube into a quadruple mass spectrometer. Finally, the count rates of product ions and hydronium ions are measured and used for calculating VOC concentrations in the sample [11–13]. It is commonly assumed that the measured count rate or current of an ion is proportional to the number density of that ion near the end of the drift tube, which itself depends on the original density as well as the transmission efficiency for that ion. Thus, in order to calculate concentrations, measuring the ratio of transmission efficiencies is necessary. Keck et al. have recently proposed a corrected equation for the concentrations in the drift tube of a PTRMS, and this equation accounts for the different mobility of primary ions and protonated analyte. As a result, the calculation of VOC concentrations from the measured count rates requires a correction factor equal to the ratio of the mobility of the protonated analyte to the mobility of the gases used to determine the transmission efficiency [13].

One of the most important applications of drift tubes is in ion separation devices such as ion mobility spectrometry (IMS) [14,15] and combined ion mobility-mass spectrometers (IMS-MS) [16–20]. In all of these instruments, the signal intensity depends on the number of ions that pass through the drift region. Low ion transmission results in a poor signal-to-noise ratio. Furthermore, if the transmission is not equal for all ions, the intensities are not a true measure of the number density of ions. Thus, information about transmission efficiency is needed to correct the intensities. Knowledge of transmission efficiencies may help to enhance the signal-to-noise ratio by allowing investigators to modify the parameters of the system to increase transmission of certain ions. The knowledge may also help in designing drift tubes optimized for specific applications.

Ideally, every ion that enters the drift tube should reach the end of the tube, if no ion is lost during travel. Therefore, the output current should be the same as the input current. However, in reality this is not true and the transmission is not perfect. The question remaining now is whether the transmission efficiency is the same for all ions or whether it differs for different types of ions. This is easily tested by measuring the output current for two different ions with similar input rates. Consider an ion source that is constantly producing the reactant ions, R^+. Before entering the drift tube, the reactant ions may undergo reactions to produce product ions, P^+.

$$M + R^+ \leftrightarrow P^+ \tag{R1}$$

In the absence of M, the current registered on a collector at the end of the drift tube is a measure of the transmission of R^+ ions. If M is added at high enough concentration to convert all R^+ ions to P^+,

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the law of conservation implies that the production rate for P^+ ions is same as the original rate of R^+ ions. Hence, the input current for P^+ ion would be the same as that for the R^+ ion. If the two ions have similar transmission efficiencies, the collector current should remain unchanged when M is introduced into the reaction region. However, in practice, this is not true. Fig. 2 demonstrates that the peak area decreases when a sample is introduced into the ionization region of an IMS. In the presence of any analyte, the reactant ion (RI) peak is substituted with a smaller peak corresponding to the product ion. Thus, the transmission efficiency for different ions is not the same. The aim of this work is to study the transmission of different ions through the drift tube and investigate the factors governing the transmission efficiency. The results are applicable when for example the equilibrium constant of the following reaction is calculated.

$$K_{eq} = \frac{[P^+]}{[M][R^+]} \quad (1)$$

The ratio $[P^+]/[R^+]$ is usually measured through the relative intensities of the two peaks. If the relative transmission efficiencies of the two ions are known, the intensities can be corrected accordingly to represent the relative number densities. The same is true for the case of calculating rate constants or other parameters dealing with the number density of ions.

2. Experimental

2.1. Instrumentation

The ion mobility spectrometer used in this study was constructed in our laboratory at Isfahan University of Technology. The ionization region consisted of five, 9.5-mm thick aluminum rings, with 20-mm ID and 55-mm OD. The drift tube consisted of 11 aluminum rings with the same OD size and 36-mm ID. Each ring was connected to the adjacent one via a 5-MΩ resistor to create a potential gradient. A continuous corona discharge ionization source with point-to-plane geometry, described elsewhere [21], was used. A Bradbury–Nielsen type shutter grid was mounted between the ionization region and the drift tube, and a Faraday cup type collector plate (10-mm diameter) with an aperture grid was used to register the ion current. The IMS cell was housed in a thermostatic oven where temperature could be adjusted from room temperature to 473 K within ±1 K. Nitrogen gas, after passing through a 13× molecular sieve (Fluka), was passed through the cell at ∼100 and ∼500 mL/min for the carrier and the drift gas, respectively. The spectrometer was operated in positive mode with various drift fields from 312 to 562 $\mathrm{V\,cm^{-1}}$. In order to produce a constant current of reactant ions, a separate high voltage was used for the corona discharge. The needle voltage was adjusted accordingly to keep the corona current constant during all the experiments. Typically, a 100-μs gate pulse was used during the recording IMS spectra. In order to observe the total ion current, the voltage between the shutter grid wires was removed in some experiments. The ion current received on the collector plate was amplified by an electrometer with a gain of 10^9 V/A and relayed to a computer via an A/D converter (Picoscope, UK). The digitized signal was averaged over a number of scans and the resulting ion mobility spectrum was then displayed on the monitor.

In order to vary the pressure inside the drift tube, a specially designed instrument described previously [22] was used. The range of working pressure was 300 mbar to atmospheric pressure.

2.2. Shutter grid

Normally, ions first pass through the shutter grid and then enter the drift tube. Obviously, lighter ions pass through the shutter grid more easily than heavier ones. Thus, the peak does not fully represent the transmission of ions through the drift tube. In order to observe the pure effect of the drift tube on the transmission of ions, the shutter grid was left fully open. This was achieved by removing the voltage applied to the shutter grid wires. Using this technique, a DC current was recorded on the collector. This current was used for further analysis. The corresponding drift time for each ion was measured with the pulsed gate.

Table 1
List of selected ketones used in this study

No.	Compounds	Drift time (ms)
0	Reactant ion	7.14
1	Acetone	8.12
2	Butanone	9.04
3	Pantanone	9.88
4	Cyclohexanone	10.52
5	Methyl isobuthyl ketone	10.70
6	Cycloheptanone	11.28
7	Acetophenone	11.32
8	Methyl heptanone	12.32
9	Octanone	12.74
10	Noanone	13.64
11	Decanone	14.52

The drift time for the dimmer peak is given for each compound.

2.3. Selection of compounds

In order to measure the transmission of ions individually, a specific type of ion ought to be produced in the ionization region. The samples were chosen so that their corresponding ion mobility spectrum contained only one peak.

At low temperatures, most of the reactant ions generated by the corona discharge are hydronium ions, H_3O^+. In the presence of an analyte with proton affinity higher than that of water, protonated molecules (MH^+) are formed via a proton transfer reaction. If the concentration of the molecule is high enough, the reaction proceeds further to produce proton-bound dimers (MHM^+).

$$M + H_3O^+ \leftrightarrow MH^+ + H_2O \quad (R2)$$

$$MH^+ + M \leftrightarrow MHM^+ \quad (R3)$$

Ketones posses high proton affinity, and at sufficiently high concentrations, they react fully with the ions to form dimers. A series of 11 different ketones, the dimers of which showed a range of drift times between 8 and 22 ms, were selected (Table 1). The ion mobility spectra of the selected ketones are shown in Fig. 2. The concentration of the samples was high enough that the RI and protonated monomer peaks disappeared and only the dimer peak remained in the spectrum. The selected compounds used in this experiment, presented in Table 1, were purchased from Fluka and used without further purification.

3. Modeling

All ions entering the drift tube either pass through the drift tube or are lost due to processes like recombination, diffusion, ion–ion repulsion, collision with the walls, etc. This can be simply modeled as a two-parallel reaction,

$$\begin{aligned} &N_0 \xrightarrow{k_d} N_C \\ &N_0 \xrightarrow{k_L} N_L \end{aligned} \quad (R4)$$

where N_0 represents the number of ions produced by the ion source that enters the drift tube. N_L is the total number of ions lost and N_C is the number of ions collected by the collector, respectively. k_L is the rate constant for the ion loss process and k_d is the rate constant

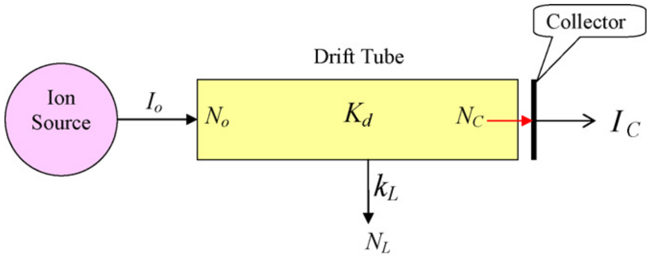

Fig. 1. A kinetic model for ion transmission through a drift tube. N_o is the number of ions entering the drift tube, N_L is the number of ions lost with a rate constant of k_L, and N_C is the number of ions that pass through the drift tube and reach the collector with a rate constant of k_d. I_o and I_C are the ion current entering the drift tube and registering on the collector, respectively.

for ion transmission through the drift tube. This model is shown schematically in Fig. 1.

Apparently, the collector reads the current only if ions move through the drift tube. The faster the ions move, the fewer are lost and more reach the collector. Thus, the number of ions reaching the collector depends on their velocity. On the other hand, the number of ions passing through the drift tube depends on the number density of ions at the beginning of the tube, $[N_o]$. Therefore,

$$\frac{dN_C}{dt} = I_C = k_d u [N_o] \tag{2}$$

where k_d is the transmission rate constant for the drift tube, which depends on its geometry. u is the velocity of ions and $[N_o]$ is the number density of the ions at the start of the drift tube.

Evidently, the ion loss rate depends on the number density of ions since the effects of all factors contributing to ion loss, i.e., recombination, diffusion, ion–ion repulsion, and collision with the walls, increase with increasing ion concentration. Therefore,

$$\frac{dN_L}{dt} = k_L [N_o] \tag{3}$$

The conservation law implies that

$$I_o = \frac{dN_o}{dt} = \frac{dN_C}{dt} + \frac{dN_L}{dt} \tag{4}$$

Thus,

$$I_o = k_L [N_o] + k_d u [N_o] \tag{5}$$

Solving Eq. (5) yields an expression for $[N_o]$:

$$[N_o] = \frac{I_o}{k_L + k_d u} \tag{6}$$

Substituting $[N_o]$ in Eq. (2) gives the collector current as a function of the initial current delivered to the drift tube, I_o, and the velocity of the ion.

$$I_C = I_o \frac{k_d u}{k_L + k_d u} \tag{7}$$

If the ions move very slowly, i.e., $u \approx 0$, then I_d becomes zero, i.e., all ions are lost. However, if the ions move very fast, k_L can be neglected and $I_d = I_o$, i.e., all ions pass through the drift tube. If $k_L \gg k_d u$, i.e., the loss rate is much larger than the transmission rate, then $k_d u$ in the denominator may be neglected and Eq. (7) is reduced to

$$I_C = \frac{k_d}{k_L} I_o u \tag{8}$$

This equation predicts that the collector current is a linear function of the ion velocity. Therefore, heavy and slow-moving ions produce a weak signal in IMS. Using $u = d/t_d$ where, d, and t_d are the drift length and time, respectively, yields:

$$I_C = \frac{k_d}{k_L} d I_o \frac{1}{t_d} = I_o \frac{\gamma}{t_d} \tag{9}$$

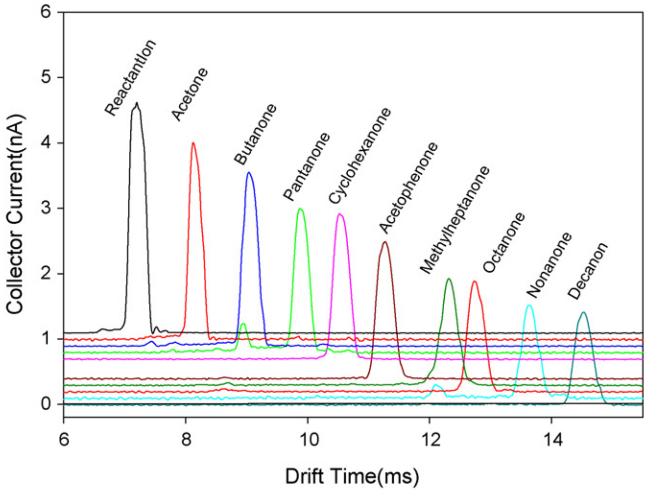

Fig. 2. Selected ion mobility spectra of ketones (listed in Table 1) at high concentration.

This equation suggests that the collector current depends on the initial current as well as a constant, $\gamma = dk_d/k_L$, which is a function of the drift tube geometry.

4. Results and discussion

Ion mobility spectra of the selected compounds are shown in Fig. 2. It is believed that the peak in each spectrum originates from the proton-bound dimer of the corresponding ketone.

The drift times of the peaks are well distributed over the range of 7–16 ms. The total ion current was measured for each compound by removing the applied voltage to the shutter grid. This was performed after ensuring that the spectrum of the compound contains only one peak. The measured currents are plotted against the drift time in Fig. 3.

Clearly, the ion current decreases as the drift time increases. This indicates that the transmission efficiency of different kinds of ions through the drift tube is not the same. The validity of Eq. (9) was examined by plotting total ion current at different drift fields against $1/t_d$.

Fig. 4 shows that the total ion current is a perfectly linear function of $1/t_d$ ($r^2 > 0.999$) with a nearly zero intercept, at all applied drift fields tested here. This indicates that the original assumption

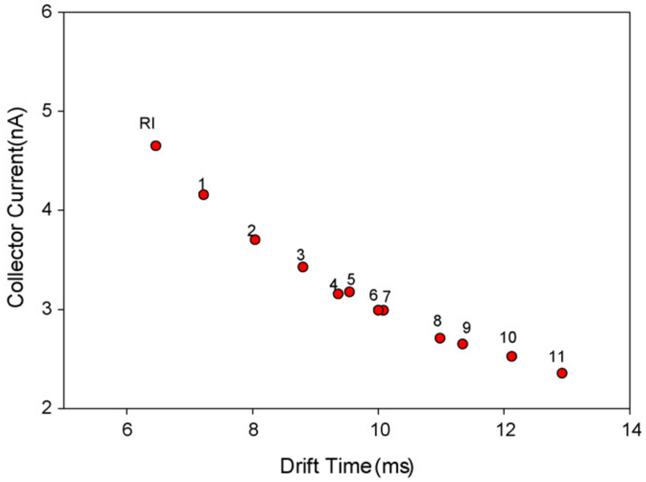

Fig. 3. Total ion current versus drift time for different ions at an applied electric field of 562 V cm^{-1}.

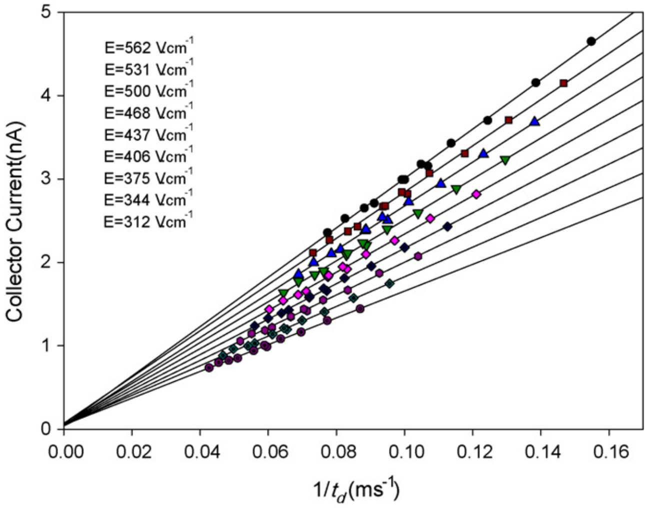

Fig. 4. The total ion current for different ions as a function of their inverse drift time at different drift fields.

$k_L \gg k_d u$ is true, i.e., the ion loss rate is much larger than the ion transmission rate. This assumption is reasonable since the total ion current delivered by the corona discharge is on the order of μA, but the ion current received at the collector is on the order of *n*A. In fact, most ions are lost so Eq. (9) is applicable. Based on this equation, any factor affecting the velocity of the ions, such as pressure, temperature, and drift field, will also influence the drift time and therefore affect transmission efficiency. This was tested in the present experiments by varying pressure, temperature, and the drift field.

4.1. The effect of pressure

The pressure inside the drift tube was lowered from atmospheric pressure down to about 0.3 bar while the blank ion mobility spectrum was recorded. The spectrum contained only the reactant ion peak. As the pressure decreased, the drift time became shorter while the peak intensity increased. Pressure has been shown to have a linear effect on the drift time [22]. After recording the drift time at a given pressure, the corresponding total ion current was measured by fully opening the shutter grid. In Fig. 5, the total ion current is plotted versus the reciprocal drift time of RIP, which changes in response to variations in pressure. The experiment was repeated with different applied drift fields, and similar results were obtained.

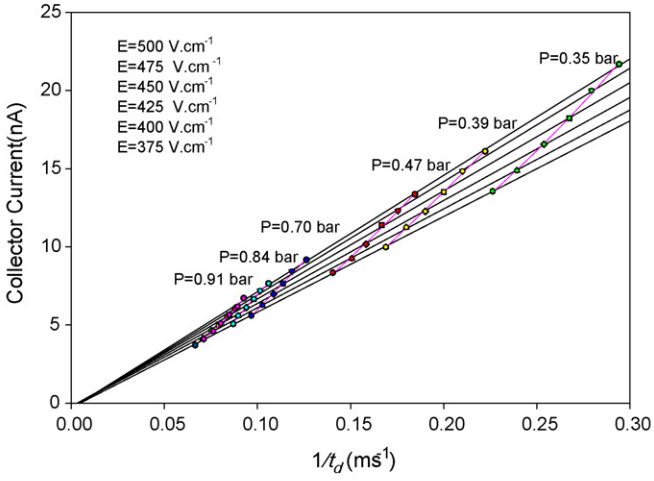

Fig. 5. The total ion current for reactant ions as a function of inverse drift time, as influenced by varying pressure. Each plot corresponds to a given drift field. The change in drift time is due to variation of pressure inside the drift tube. Isobar points are connected by a line.

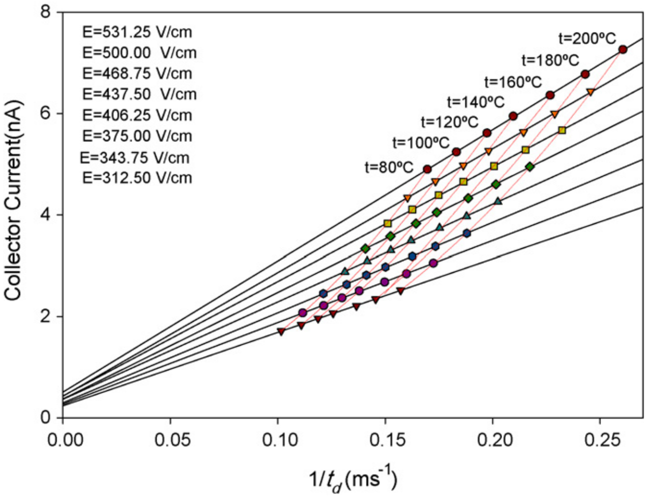

Fig. 6. The total ion current for reactant ions as a function of inverse drift time at different drift fields. The change in the drift time is due to temperature variation within the drift tube. Isothermal points are connected through lines.

The plots are perfectly linear ($r^2 > 0.999$) and the intercepts are zero. Therefore, Eq. (9) is proved to be valid when the drift time changes due to pressure variations.

4.2. The effect of temperature

Unlike pressure, temperature has a complicated effect on drift times [23]. In the present studies, the overall effect of increasing the velocity of ions was considered. The drift time of the RIP was measured while temperature was increased from 80 °C to 200 °C. The total ion current was recorded in a manner similar to that described for pressure. The results are shown in Fig. 6. These, again, confirm Eq. (9).

4.3. The effect of drift field

The drift field influences $1/t_d$ in a simple, linear fashion. Thus, if Eq. (9) is applicable, total ion current should increase linearly with stronger drift field. However, the results show that the total ion current is a second order function of both the drift field and $1/t_d$ with a zero intercept. In Fig. 7, the total current for different ions is plotted versus $1/t_d$ and drift field.

The second order function may be explained if we assume that the initial ion current entering the drift tube, I_o, is under the influence of the applied field. This was, in fact, observed in our previous work where the collector plate was mounted near the counter electrode of the corona discharge [21]. The results in that work showed that the percentage of the ion current reaching the collector is proportional to the relative drift field between the two regions. In other words the ratio I/I_{Corona} is an approximately linear function of the ratio E/E_{Corona}, where I_{Corona} and I are, respectively, the corona current and the current reaching the collector. E_{Corona} is the average field in the corona region and E is the field on the collector side. In the present study, the ratio E/E_{Corona} is less than 10% and the current entering the drift tube is also less than 10% of the corona current. Since the corona current and its field are kept constant, any increase in the drift field should result in an increase in the ion current entering the drift tube. Therefore, the initial current, I_o in Eq. (9), depends on the drift field. The effect of the drift field on

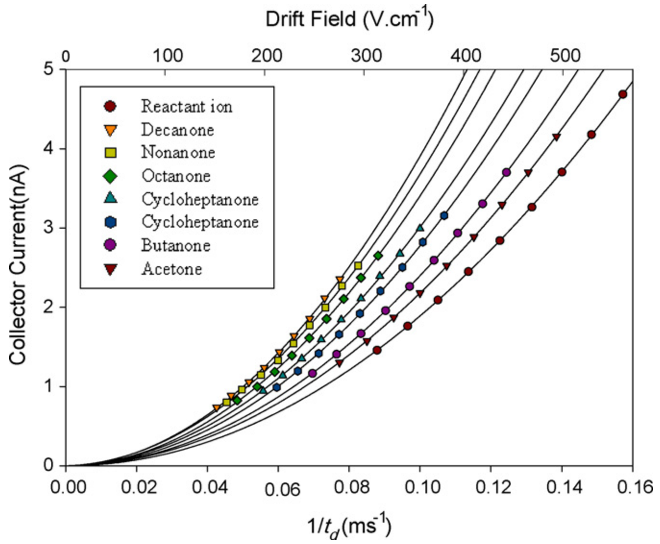

Fig. 7. The total ion current for different ions versus the drift field (E).

I_o may be eliminated by dividing the collector current by E. Fig. 8 demonstrates that I_C/E is a perfect linear function of $1/t_d$ with a zero intercept. Interestingly, the slopes for all the different ions are the same. Thus, we can modify Eq. (9) by substituting $I_o = \beta E$, where β is a constant.

$$I_C = \gamma \beta E \frac{1}{t_d} \tag{10}$$

γ and β are both constant and we define here a new constant, $\alpha = \gamma\beta$.

$$I_C = \alpha E \frac{1}{t_d} \tag{11}$$

Eq. (11) is valid for all ions and for different conditions like pressure, temperature, and drift field. The constant α is described by Eq. (12) and depends solely on the structure of the drift tube.

$$\alpha = \frac{k_d}{k_L} \beta d \tag{12}$$

In fact, α depends on the ratio k_d/k_L, i.e., the transmission rate with respect to the ion loss and may be called "*the transmission efficiency*". Since α is constant, the ratio k_d/k_L is independent of the type of the ions, the drift field strength, pressure, and temperature.

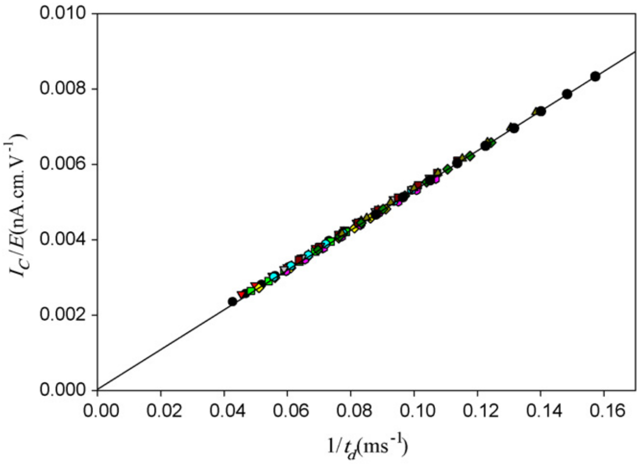

Fig. 8. Plots of I_C/E versus $1/t_d$ for different ions. t_d is the drift time of each ion at drift field of E.

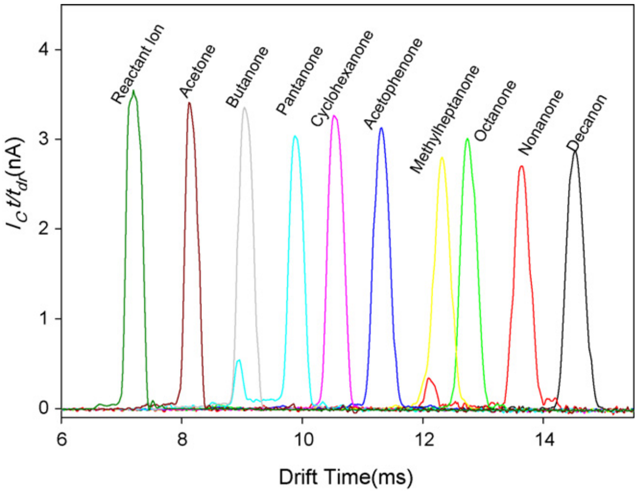

Fig. 9. The corrected spectra in Fig. 2 to take into account the transmission efficiency of different ions. The y-axis has been transformed to $y \times x/t_{RIP}$.

4.4. Correction of intensities

As Fig. 2 shows, heavy ions show lower peak intensities. Eq. (11) may be used to compensate for this decrease. For two different ions, the ratio of their signal intensities, S_1/S_2, is proportional to the inverse of their relative drift times. Thus, the intensities can be normalized to the intensity of the RIP by multiplying the intensities by a factor of t/t_{RIP}. In other words, if the y-axis is simply transformed to $y \times x/t_{RIP}$, the intensities will be normalized to the intensity of the RIP. This operation is shown in Fig. 9.

Clearly the intensities have been adjusted to correct for the effects of ion mobility. In fact, the area under the peaks is almost constant. (The small reduction in the height of the peaks is due to broadening.) Close investigation of the normalized intensities reveals a small slope in the plot of peak area versus drift time. This is believed to be due to the effect of the shutter grid. Apparently, the efficiency with which slow ions pass the shutter grid is lower than for fast ions. Hence, the signal intensity for slow ions is less than for fast ones, even after normalization.

4.5. The number density of ions

As mentioned in Section 1, it is sometimes essential to know the relative number density of two different ions in the ionization zone. This ratio can be readily determined from the relative intensities of their corresponding peaks. However, a correction factor must be considered. If Eq. (2) is applied to two different ions, it yields

$$\frac{I_{C1}}{I_{C2}} = \frac{u_1}{u_2} \frac{[N_{o1}]}{[N_{o2}]} \tag{13}$$

Therefore, the relative number densities are proportional to the relative signal intensities as described by

$$\frac{[N_{o1}]}{[N_{o2}]} = \frac{S_1}{S_2} \frac{t_{d1}}{t_{d2}} \tag{14}$$

where S_i is the area under peak i. Eq. (14) is very useful in calculating equilibrium or rate constants for ion–molecule reactions. It can be proved that the relative number density at the end of the tube equals its beginning value. The current received on the collector, I_C, for each ion is

$$I_C = u[N]A \tag{15}$$

where u is the velocity of ion, $[N]$ is the number density of that ion in the vicinity of the collector, and A is the area of the collector.

Recalling Eq. (2), we conclude that

$$[N] = \left(\frac{k_\mathrm{d}}{A}\right)[N_\mathrm{o}] \tag{16}$$

This equation shows that the number density at the end of the tube falls by the same amount for all ions. Thus, the relative number density remains unchanged during the transmission. However, slowly moving ions induce less electrical current in the collector because of Eq. (15). Therefore, if one needs to relate the ion currents to the number densities at either end of the tube, Eq. (14) or (15) must be used.

5. Conclusions

The response factor in IMS is not the same for different ions because of their different transmission efficiencies through the drift tube. The response factor has been shown to depend linearly on $1/t_\mathrm{d}$. Experiments indicate that any factor that influences the drift time also affects the transmission. The simple model proposed here predicts that most ions are lost within the drift tube. Both loss and transmission rates are independent of the nature of the ions; instead, both depend on the number density of the ions, and the transmission rate also depends on the velocity of the ions.

Although the results reported here were obtained for positive ions, the concept is valid for negative ions too. Electrons that play the role of reactant ions in negative mode move much faster than ions. Hence their transmission efficiency is much higher than that of negative ions. This can be observed on an ion mobility spectrum as a huge electron peak near zero drift time. However, in the presence of an analyte, light electrons are converted to heavy negative ions, leading to much smaller peaks [24].

Based on experimental results, a simple formula is proposed here to adjust the intensities to take into account the transmission efficiency of different ions. This is particularly useful when trying to calculate the relative number density of two species in the ionization zone. For example, in calculating the ratio $[H_3O^+]/[NH_4{}^+]$, the relative corresponding intensities should be corrected by a factor of 1.3, as the reduced mobility is 2.5 and 3.25 cm^2/V s for H_3O^+ and $NH_4{}^+$, respectively. Without such correction, the calculation is expected to show a 30% error.

In summary, the signal intensity in corona discharge IMS increases quadratically with the electric field strength, linearly with temperature, and inversely with pressure, drift length, and drift time. These relationships will help in designing drift tubes to operate under desired conditions. For example, if heavy ions with long drift times are being studied in a drift tube, then the length, applied field, and pressure will have to be chosen to yield a detectable signal.

Even after correction, the intensity is observed to decrease slowly with drift time. This is believed to be due to the effect of the shutter grid on ion transmission efficiency. Further investigations are underway to understand the effect of the shutter grid.

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