

A novel non-radioactive electron source for ion mobility spectrometry

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Abstract Typical ion mobility spectrometers work by employing a radioactive source to provide electrons with high energy to ionize the analytes in a series of chemical reactions. General security as well as regulatory concerns related to the use of radioactivity resulted in a need for a different ionization source which on the other hand produces ions in a similar manner as a radioactive source because the mechanisms are well known. Here we introduce a novel non-radioactive electron source which is capable of providing high energy electrons in a way that is similar to beta radiating substances yielding correspondingly similar peak spectra.

Keywords Radioactive electron sources · Non-radioactive electron sources · Ionization mechanisms · Electron beam manipulation

Introduction

Ion mobility spectrometers are well known for fast detection of chemical compounds in air down to very low

concentrations of a few micrograms per cubic meter. They found a broad field of application such as monitoring of hazardous substances like explosives, toxic industrial compounds or chemical warfare agents [1–4]. Ion mobility spectrometry (IMS), introduced by the end of the 1960s by Cohen and Karasek, from a more general perspective is thus an analytical technique that separates gas-phase ions at atmospheric pressure. IMS is a fast, low-cost, and sensitive method ideal for the detection of trace quantities of volatile organic and inorganic compounds. The variety of applications ranges from detection of illegal drugs, explosives, and chemical warfare agents in early stages over the analysis of non-volatile and labile samples via electrospray ionization–ion mobility spectrometry (ESI-IMS) introduced in the late 1980s to the analysis of metabolomes and proteomes if combined with mass spectrometry and, in more modern applications, even the study of protein-protein and non-covalent protein-ligand complexes, imaging of tissues, and separation of carbohydrate isomers and mixtures of peptides is possible [5–14].

In general, the separation of species in an ion mobility spectrometer works via the drift time principle. After ionization the sample is led with help of electric fields into the drift region where the ions are then accelerated by an electric field of typically a few 100 V/cm. These ions subsequently collide with the air molecules and depending on their mobility (which in turn depends on numerous parameters, e.g. structure and size) reach after different drift times the detector. The techniques used to ionize the sample are as diverse as the fields of application of IMS: Electrospray [15–19], typically for liquid samples, optical (high intensity light sources [20, 21] resp. laser [22, 23], X-Ray [24]), or corona discharge [25–28], but the most common one especially in commercial devices is a radioactive source (mainly ^{63}Ni , but also ^{241}Am and ^3H). The different sources

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ionize with different energies: ^{63}Ni emits electrons with a maximum energy of 67 keV and an average energy of 17 keV, ^{241}Am emits short range alpha particles at over 5.4 MeV, ^3H emits electrons at less energy than ^{63}Ni with a maximum value of 18.6 keV and an average value of 5.7 keV [29]. The reason for the use of such sources is the simplicity of using a radioactive source for ionization as there is no external power supply needed; neither is maintenance. Despite these advantages of radioactive sources their application is discouraged in commercial devices due to technical, organizational and, related to these, financial complications. These sources require certain procedures regarding permits, licenses or disposal, for example, which lead to extra costs and so does the special set up of the drift tube to ensure that no radioactive by-product can leave the tube. Thus research was conducted in order to find similarly efficient ionization sources that can work without radioactive substances. On the other hand, the ionization mechanism using beta radiation is very well understood so that any replacement should also ionize via high energetic electrons providing thus similar if not equivalent reactant ion chemistry.

A well known non-radioactive ionization source that uses such electrons is the previously mentioned corona discharge [30]. Here a voltage pulse of ca. 2–4 kV between two gold electrodes leads to the emission of electrons. Special pulses (e.g. 82 kHz pulses decaying from an amplitude of 2 kV to zero over 1,000 μs) seem to lead to similar results as a ^{63}Ni source [31]. In contrast to this, other studies found additional side reactions initiated by the production of NO^+ . Increasing the corona energy additionally triggers O_3 production resulting in a wide variety of atmospheric pressure chemical ionisation pathways. The consequence is that the adoption of corona discharge sources as replacements for ^{63}Ni for the positive mode (which will be the mode under investigation in this paper) requires stable and reproducible corona sources which is technically quite challenging. A comparative study presented by Borsdorf and Rudolph [32] even presented data which indicated different ionisation chemistries between a corona ionisation source and ^{63}Ni which then raises the question to which extent this technique can be seen as a replacement for the radioactive source. Nevertheless the exact nature of the ions produced by a pulsed corona discharge ionisation source has yet to be established. For the negative mode the reactant formation in corona discharges has been analyzed recently [33]. Besides the problems on the chemical side are additional problems on the technical side, as the corona discharge needs maintenance due to the electrodes degrading relatively quickly in quality. A source that also works without radioactivity and a high energy discharge is the plasma ionization source. In this case a dielectric barrier is located in-between the

electrodes. Thus a plasma is formed that leads to the ionization of the reactant molecules. This ionization method does not show the problems with the degradation of the electrodes [29], and it furthermore can produce the same spectra as radioactive sources [34, 35] at even increased intensity, but it requires a relatively high gas flow of e.g. Helium.

Due to the described situation there is still a need for an electron source that can be regarded as a replacement for the radioactive source normally used in IMS. Non-radioactive electron sources with relatively high electron energies (larger than 30 keV) are known [36, 37] but the high amount of energy lost within the glimmer membrane used as an exit window led to increased mechanical instability. Furthermore, the production of X-ray radiation was observed. In this paper we introduce a novel electron source that produces electrons which are in the energy range of those produced by a ^3H source. By comparing the reactant peak produced employing this source and employing the Tritium source we show as a proof of concept that this source yields the same results, thus being comparable to a Tritium source but offering in addition more flexible control of the electron beam and thus the possibility to conduct more complex experiments.

The concept of our new electron source is based on the early work (1894) of P. Lenard who showed that electrons, generated in an electrical discharge tube, could pass a thin aluminium foil. A wider use of the concept had been delayed by almost exactly 100 years simply due to the lack of availability of appropriate foils to send electron beams through, i.e. from the vacuum in an electron beam device into dense gases. Nowadays, however, completely vacuum tight and extremely thin ceramic foils are available and can thus be used for the described purpose. The material which has been employed so far is silicon nitride with a thickness of typically 300 nm. Such foils are routinely manufactured by solid state technology starting from a silicon wafer which is covered with the silicon nitride film on both sides. Then one side is structured and the nitride layer removed from designated areas. The bulk material is etched away in these areas; the etching itself is stopped by the chemically stable nitride film on the other side of the wafer leaving the thin film in place and conveniently mounted in a piece of the wafer which can be cut out by a saw or broken out along cleave lines (details can be found in [38]). Replacing the classical Lenard windows, which are 2,650 nm thick aluminium foils, by the novel silicon nitride membranes, the particle energy of the electrons necessary to penetrate the foil can be significantly reduced from typically 50 keV to ca. 10 keV. Figure 1 shows the electron transmission through these two thin foils as a function of the electron kinetic energy; the values have been calculated using the Monte Carlo Simulation software Casino v2.42 [39, 40].

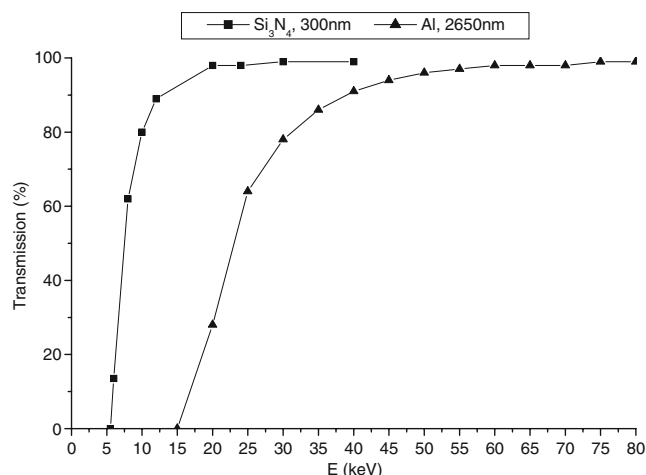


Fig. 1 Electron transmission in relation to kinetic energy and material

According to these calculations, a transmission of ca. 80% requires only 10 keV in the case of the silicon nitride membrane while the aluminium membrane requires ca. 30 keV. Thus the silicon nitride membrane allows the production of electron beams with energies close to those of a ^3H source, especially regarding the average electron kinetic energy. Various practical light sources have already been realized by sending electron beams through such silicon nitride membranes into purified rare gases or other gas mixtures [38, 41–44] in analytical chemistry applications.

The paper is thus organized as follows: First a description of the experimental set-up with the electron source will be given. In order to demonstrate the capabilities of this source as a possible replacement for a Tritium source a description of the reactant peak obtained for different parameters of the source follows. These parameters are mainly voltages applied to the source, and their influence on peak characteristics as intensity and resolution will be shown.

Experimental

Figure 2 shows the basic set up. Central component is the drift tube. We use a drift tube and amplifier from a Draeger Ion Mobility Spectrometer GSM with a ca. 3 mm long reaction region, a ca. 7 cm long drift region and a ca. 0.5 mm long collector region. In the reaction region the chemical reactions to ionize the reactant take place. The electric potential of the source grid is given by the high voltage supply 1 (called HV1 in Fig. 2) and the source wall potential is given by the high voltage supply called HV2 (low voltage). Initially, a small electric field gradient (3 V/cm) is present in the reaction region in order to prevent drift losses into the drift region. The collector wall is at zero potential. At a frequency of 30 Hz, the source wall potential is increased so that the field strength in the

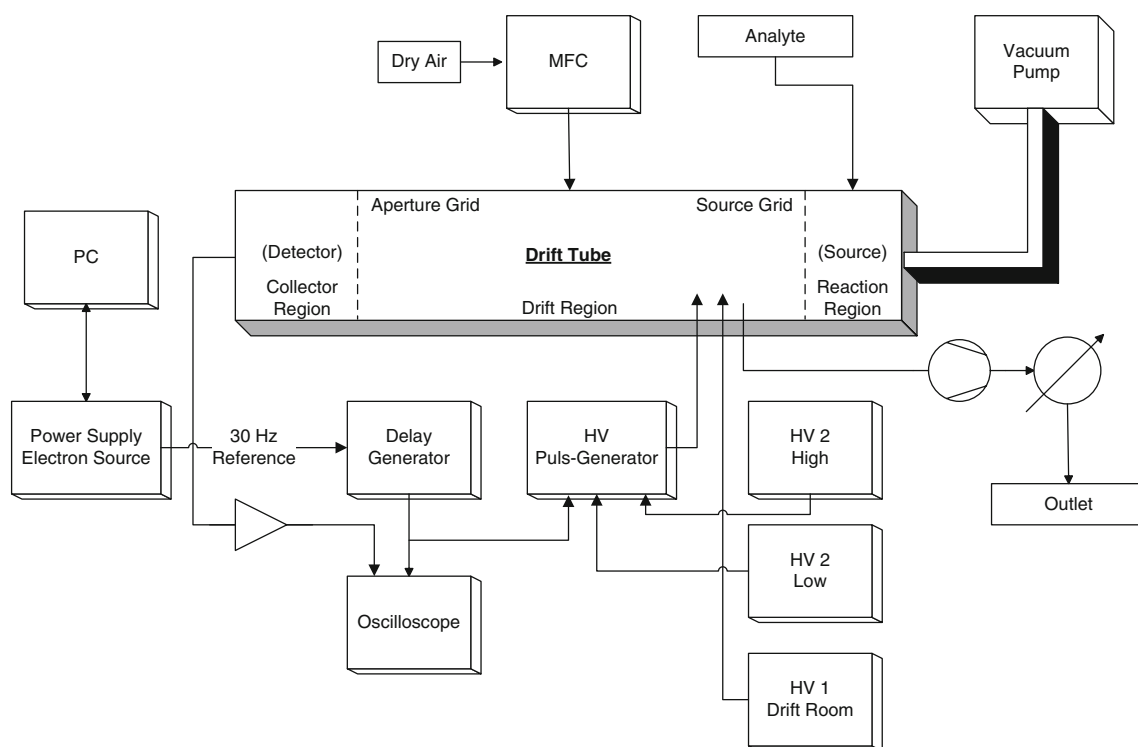


Fig. 2 Experimental set-up

reaction region reaches ca. 1,000 V/cm for typically 100 μ s. The voltage for this pulse is provided by HV2 (high voltage), and the HV Pulse Generator controls the temporal width of the pulse. The pulse is used in order to accelerate the ions after the ionization process out of the reaction region into the drift region. In this region a permanent field of ca. 200 V/cm accelerates the ions towards the collector region. During their flight through the drift region the ions collide with the air molecules (the drift tube is operated at atmospheric pressure) of an air current that flows against the direction of the ions with ca. 300 ml/min, the flow rate being controlled by the mass flow controller MFC. In the collector region the ions experience a field of 1,000 V/cm that accelerates them towards the detector, which is formed by a simple Faraday cup.

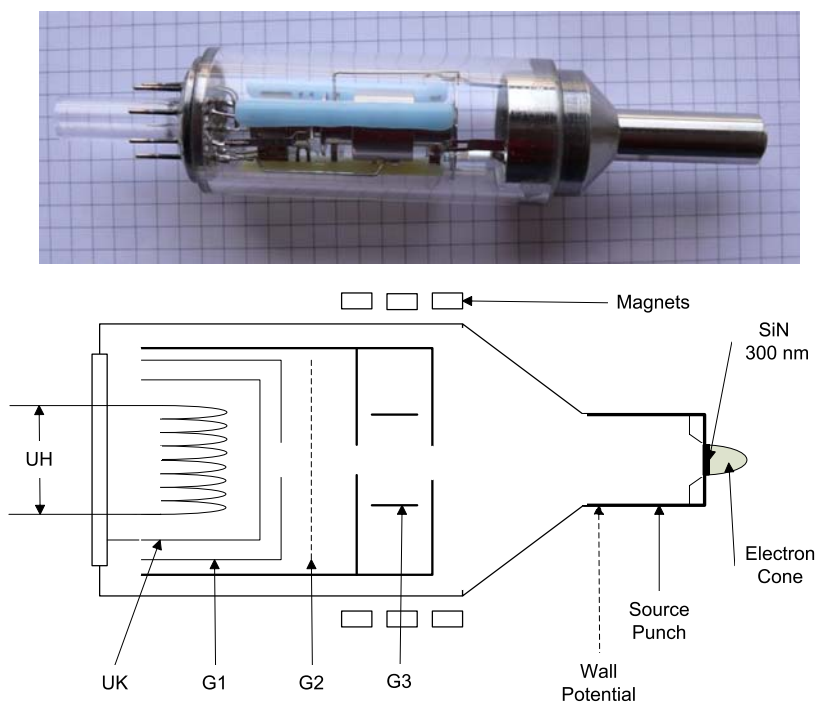
In this experiment, the voltages HV1, HV2 (low) and HV2 (high) are provided by high voltage supplies of type DC Power Supply HCP35-3500 (FUG Company, Germany). The delay generator that generates the trigger pulses for the HV Pulse Generator is a Pulse Generator Model 9512 (Quantum, USA), and the HV Pulse Generator is a PVX-4140 (DEI, USA). The oscilloscope used to monitor the trigger pulses, the reference pulse from the Pulse Generator and the output of the detector unit is a Wavejet 314 (LeCroy, Germany).

Figure 3 shows the electron source used instead of the radioactive ^3H source. The power supply providing the voltages for the Grids G1, G2 and G3 as well as the heater voltage UH and the cathode's high voltage UK is a custom

made system by Optimare Wilhelmshaven, Germany. Per software (also provided by Optimare) it is possible to set all these voltages for static as well as pulsed operation. The electron source works at high vacuum (better than 10^{-6} mbar). To maintain the vacuum we use a turbomolecular pump system of type HiCube from Pfeiffer Vacuum, Germany. The electrons are emitted thermally by a filament which is heated by supplying the heating voltage UH. By supplying corresponding voltages to grid G1 electrons can enter the tube as a constant flow or pulsed. Supplying voltage to grid G2 electrons are accelerated to the focus system G3 consisting of two parallel metal plates and a corresponding focus voltage. Beam direction tuning is achieved by using the electromagnets attached to the electron tube. The emission and focussing groups are on a potential UK. In our experimental set up the potential UK can be set to values between 8 and 12 kV. The electron kinetic energy is then high enough in order to penetrate the silicon nitride foil and ionize molecules in the reaction room. The size of the silicon nitride window is about 0.5 mm². The regular radioactive source in the Draeger IMS is a ^3H source (circular shaped metal plate with 10 mm diameter) with 50 MBq activity. It has been replaced by a source punch with a diameter of about 10 mm that emits the electron beam produced by the described non-radioactive source.

One important factor for the experimental results is the extraction pulse width, i.e. the time the voltage of the source wall is higher than the reaction grid voltage and is

Fig. 3 Basic set up of the electron source; G1, G2 and G3 are the grid voltages, UH is the heater voltage, UK is the high voltage of the cathode



thus accelerating ions out of the reaction region. The longer this pulse is, the larger the volume is from which electrons can be accelerated into the drift region. Correspondingly, the RIP intensity should increase with increasing extraction pulse width. This is shown in Fig. 4, lower graph. From a pulse width of ca. 100 μs on the increase in intensity (i.e. maximum peak value) is not as strong as with lower pulse widths anymore and thus this value was chosen as the operation pulse width for the source. In contrast to this, a larger volume from which electrons are brought into the drift region has a negative effect on the resolution due to the increased width of the drift time distribution. Figure 4 upper graph shows the decrease of the resolving power of the IMS if larger extraction pulses are used. We chose an extraction pulse width of 111 μs as then the peak intensity was high to give good spectra and the resolving power was also comparably good in order to obtain small peak widths. From both graphs in Fig. 4 it can be additionally concluded in agreement with the previous statement that the extraction pulse width of 111 μs forms a good trade-off between increase of intensity on the one side and loss of resolving power on the other hand.

Results and discussion

The goal of the here reported experiment was to characterize the new non-radioactive electron source. From corona discharge experiments it is known that this ionization form can produce different signals than the radioactive source. The differences can be different peak positions (regarding their reduced mobility values K_0), but also additional peaks and, resulting from these, a different reactant peak shape.

To compare the ionizing effect of the non-radioactive source, Fig. 5 shows an ion mobility spectrum obtained with the drift tube and the non-radioactive electron source used in our experiment. The peak shape and the peak position are identical to the reactant ion peak RIP generated with a ^3H source. The drift time is 6.37 ms. This result is a direct consequence of the fact that the electron source produces electrons with similar properties as the Tritium source, even regarding the kinetic energy of the electrons. According to the Monte Carlo calculations described in the introduction a kinetic energy of 8 keV leads to electrons with an average energy of ca. 6.2 keV. This value is close to the average energy of the Tritium source (5.7 keV) and thus the subsequent ionization reactions should be the same and furthermore the resulting RIP peak. Thus the electron source shows good potential in order to serve as a replacement for radioactive ^3H sources fulfilling the condition that the ionization processes are similar.

The shape and the intensity of the RIP depend on the operation parameters of the source. This is shown in the next graphs. The first important parameter is the quantity of electrons produced which depends on the heater voltage U_H . The higher this voltage, the more electrons are emitted by the filament and can be accelerated out of the tube. Figure 6 shows the dependence between RIP intensity and heater voltage U_H . As can be seen, the RIP intensity (i.e. the maximum peak value) can be increased by increasing U_H . At values below 6 V, the electron density is quite low and thus the RIP intensity close to zero. Increasing U_H to 7 V leads to a more than 10-fold increase of the RIP intensity. Thus a value of 7 V was chosen as the operation heating voltage in all our experiments. A further increase of the heater voltage leads to a strong heating and damage of

Fig. 4 Resolution and RIP intensity in relation to the extraction pulse width

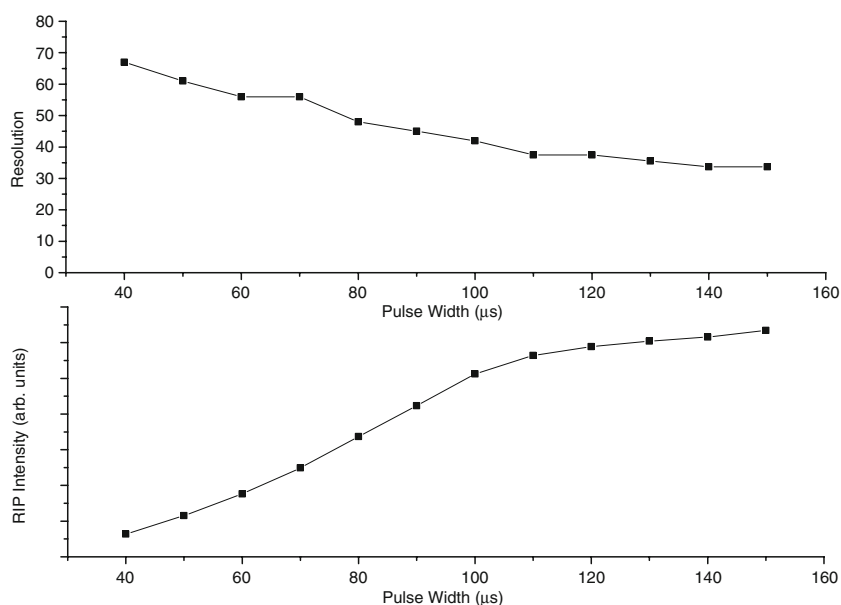
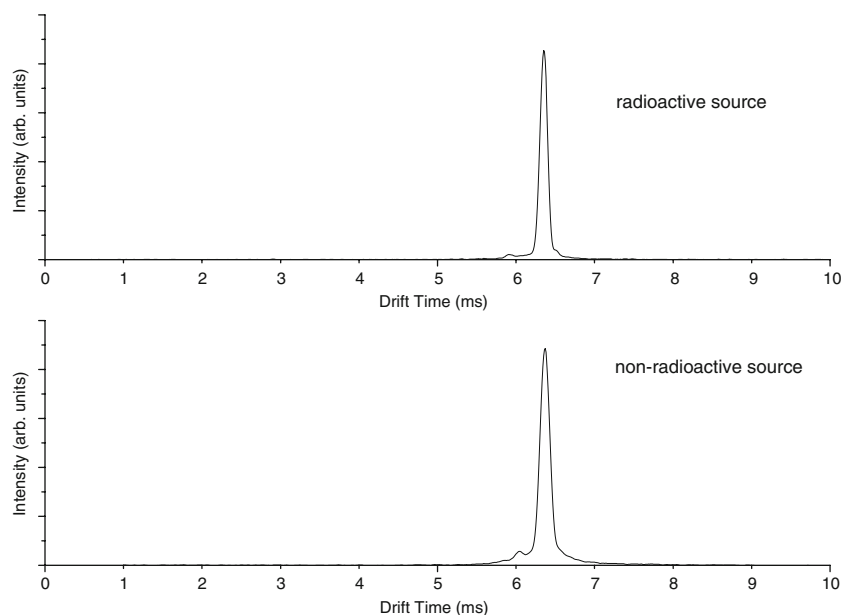


Fig. 5 Drift time spectrum of the RIP obtained under the conditions described in Table 1 with the radioactive Tritium source, and with the non-radioactive source



the silicon nitride membrane. The operation current of the filament is ca. 300 mA and thus the power consumption needed for the heating is ca. 2 W.

Another factor that affects the RIP intensity is the kinetic energy of the electrons. This energy can be controlled by changing the high voltage UK. As can be seen in Fig. 7 the RIP intensity is very low for kinetic energies below 5 keV. Increasing UK to over 8 kV increases the RIP intensity by more than a factor of 10, and a further increase is achievable until ca. 10 keV electron kinetic energy. At this value the intensity does not increase anymore, a further increase of UK leads even to a decrease of RIP intensity. The reason for this is the fact that electrons with higher kinetic energy can reach further into the reaction region. If the kinetic energy is too high, the electrons reach past the source grid into the drift region. The consequence is that there are not only ions

formed in the reaction region but also in the drift region which are directly transported to the collector. These in turn can lead to additional false signals in the ion mobility spectrum, but more commonly they contribute to the basic spectrum noise. Nevertheless they do not contribute to the RIP intensity anymore and thus the RIP peak does not increase anymore and can furthermore even decrease. Note that a variation of the acceleration voltage also leads to a variation of the power deposited in the electron beam and thereby the ionization rate in the gas; at reduced electron energies there is an increased energy loss, a broader kinetic energy distribution and larger angular scattering in the silicon nitride membrane.

The values in Table 1 are a consequence of the previously shown measurements. They allow an operation with intense (RIP-) signal peaks at reasonably high resolving power. Under the conditions described in Table 1 we observed a good stability of the peak intensity. Over the measuring period of 1 week no relevant change of peak intensity was observed. Thinking about the later application of the described source as an electron source in ion mobility spectrometers a pulsed mode would have a multitude of advantages. Pulsed electron ionisation is already being analyzed in our experiments, and preliminary results show the same ion mobility spectra in continuous and pulsed operation. The latter means in general a smaller intensity but this could be compensated by increasing the heater voltage UH. We found that an electron pulse width of 10 μ s is sufficient to provide spectra of good quality. Therefore the operation life time of the electron source can be assumed to be in the range of many years.

A variety of analyte IMS spectra have been measured. There was no relevant difference between our non-radioactive

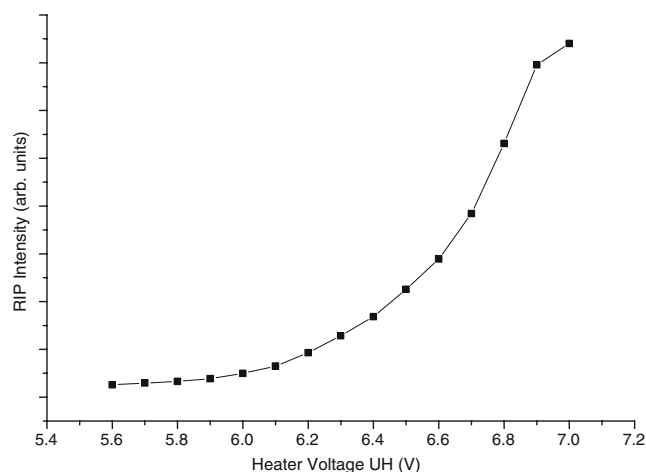


Fig. 6 RIP-intensity depending on the heater voltage UH

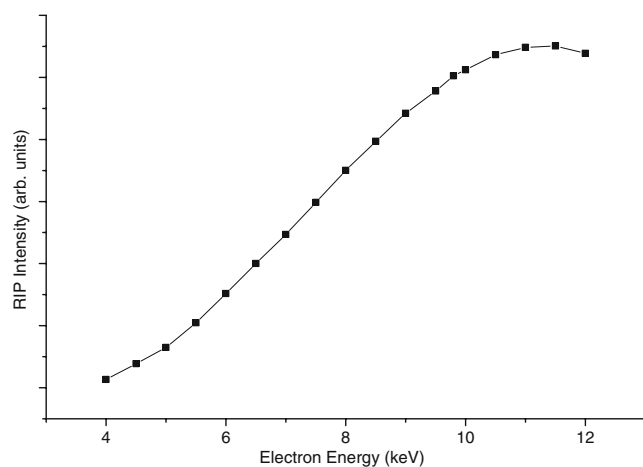


Fig. 7 RIP-intensity depending on the electron kinetic energy (determined via the high voltage UK)

electron source and the previously used Tritium source. First examinations show a sensitivity of at least the same value as the Tritium source. The non-radioactive source is from the electron-producing principle relatively close to a radioactive source. The average electron energy of ^3H is 5.7 keV. The high voltage used in the here described experiment was 8 kV. Taking into consideration that the electrons lose some energy (see Fig. 1) while passing through the metal foil that forms the exit window, the average energy of the electrons in the beam produced by the non-radioactive source can be assumed to be close to the average value of the ^3H source (as mentioned before, according to Monte Carlo calculations the difference is less than 10%). Another important factor is e.g. the absence of ozone or other by-products that typically form in electric discharges due to the fact the electrons are produced here in a discharge-free manner. It can be assumed that the obtained spectra are quite similar because the electron sources work quite similar (IMS spectra using the new non-radioactive source will be shown and discussed in subsequent papers) and thus we consider the here described

non-radioactive source as a suitable replacement candidate for radioactive ^3H sources. Details of the ion chemistry are currently under investigation. For this we have coupled the IMS with the non-radioactive source to a time-of flight mass spectrometer. About this work we will also report in subsequent paper.

As the characteristic parameter of this source can be controlled from the outside, the experimental flexibility introduced into ion mobility spectrometry is increased. From pulsed corona discharges it is known that the controlling the flow, i.e. intensity over time and duration, of electrons has an influence on the spectra [33, 45]. The possibility to control these parameters exists here, too, and especially the influence on the details of the peak shapes is an interesting topic for further research in our group. A difference compared to the corona discharge is that in this source there is no degradation of electrodes etc. The electron producing effect (basically a glowing filament) is much softer and thus much less harmful for the cathode. The quality of the electron beam correspondingly does not change over time and thus this source offers constant experimental parameters for very long time on the one hand and reduced maintenance effort on the other.

Conclusion and outlook

In this contribution we have presented a non-radioactive electron source that can be used in order to replace radioactive sources in ion mobility spectrometers to record spectra in the positive or negative mode. Peaks obtained with a ^3H source could be reproduced with this source with no observation of additional peaks. In the case of corona discharges similar measurements showing additional peaks have led in the literature to the conclusion that different ionization mechanism are present compared to radioactive sources. Here we therefore conclude that the ionization mechanisms are similar, which is also a consequence of the similarity of the produced electron beams (e.g. no discharge). Besides the possibility to have a non-radioactive source that creates a similar ionization mechanism like the radioactive source to be replaced, this electron source offers additional experimental parameters to be controlled. Future research could investigate the similarity of the IMS spectra regarding the electron source for a greater number of substances. Being able to shape the time profile of the electron beam over time should have a different influence in different substances and could thus be used to isolate certain properties, so that new ways for the applicability and usability of ion mobility spectrometry could be explored with help of this source. A quite interesting topic is here the use of a pulsed electron beam. Beside its energy saving effect it might offer additional analytical applications.

Table 1 Summary of the set up values for the IMS and the non-radioactive electron source

Device	Parameter	Value
IMS	Extraction Pulse	low 1999.95 V
	(Source's Potential)	high 2327.0 V
		high time 111 μs
Electron Source	Reaction Room Grid	2,001 V
	Drift Gas Flow	495 ml/min
	High Voltage UK	8 kV
	Grid Voltage G2	150 V
	Focus Voltage G3	300 V
	Heater voltage UH	7.0 V
	Pressure	8.6×10^{-7} mbar

The described non-radioactive electron source has the status of a laboratory experimental set-up. Before the source will have a commercial importance the vacuum tightness of the system has to be improved in order to ensure a long term stability of the vacuum so that the vacuum pumping system can become obsolete.

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