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Atmospheric Pressure Corona Discharge Ionisation and Ion Mobility Spectrometry/Mass Spectrometry study of the negative corona discharge in high purity oxygen and oxygen/nitrogen mixtures

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ABSTRACT

Negative ions formed in negative corona discharge in high purity O_2 and O_2/N_2 mixtures have been studied using the Atmospheric Pressure Corona Discharge Ionisation Mass spectrometry (APCDI/MS) and Ion Mobility Spectrometry/Mass Spectrometry techniques (IMS/MS). The primary negative ions have been detected and identified using APCDI/MS technique. The primary ions are fast transformed via ion-molecule reaction with N_2O into ions with $m/z = 60$ ($N_2O_2^-$). The N_2O is formed in the negative corona discharge in the reaction of O with N_2 (~5 ppm). In the case of N_2 admixtures to O_2 from 10 to 80%, additional negative ion $m/z = 62$, NO_3^- appears in the IMS and MS spectra. The mobilities of the negative ions were determined from IMS and mass selected IMS spectra. The $m/z = 60$ ($N_2O_2^-$) has reduced ion mobility of $2.52 \pm 0.05\ \mathrm{cm^2\,V^{-1}\,s^{-1}}$. In O_2/N_2 mixtures, additionally to $m/z = 60$, negative ion with reduced ion mobility of $2.14 \pm 0.05\ \mathrm{cm^2\,V^{-1}\,s^{-1}}$ and $m/z = 62$ have been detected and assigned to NO_3^-. Formation of this ion was associated with the presence of NO_2 generated in the negative corona discharge in O_2/N_2 mixtures.

1. Introduction

Formation of negative ions in the negative corona discharge in air has been studied using mass spectrometry by several authors [1–3]. The pioneering work of Shahin [1] conducted over a wide-pressure range (up to 100 kPa) found NO_2^- and NO_3^- ions were dominant in negative corona discharge fed by dry air at low pressures. These results were subsequently contradicted by of Gardiner and Craggs [2] who at 1 kPa observed predominately CO_3^- ions, whose abundance was more than double that of O_3^-, O^- and CO_4^-. Only traces of O_2^- ions were detected. Skalny et al. [3] studied the negative ion formation in a negative corona discharge using both dry and ‘wet’ air at pressures between 5 and 27 kPa. In dry air the dominant ion observed was CO_3^-. In the presence of water this ion was converted to cluster ions containing one or more water molecules. If ozone and nitrogen oxides were added, or were produced in discharge in sufficient concentrations, NO_3^- ions and NO_3^- hydrated clusters were formed. Nagato at al. [4] studied ions formed in negative corona in air at ambient pressure at different reaction times (1 and 10 ms). They observed that after 10 ms the number of the ions was significantly reduced and the ions with significant intensity appear at masses 62, 124 and 125 amu. They attributed these masses to NO_3^-, $HCO_3^- \cdot HNO_3$ and $NO_3^- \cdot HNO_3$ ions. When the humidity of air was limited to 25 ppm of H_2O the only ion observed was the NO_3^-.

Gravendeel and Hogg [5] studied mass spectrometrically negative corona in N_2/O_2 mixture with ratio 5 to 1. Although they used high purity gases with less than 5 ppm H_2O and 0.1 ppm CO_2 they observed numerous negative ions with O_3^-, OH^-, NO_3^- and CO_3^- being the most intense ones. In this case, however, the time between formation of ions and their entrance to vacuum was short (in order of 10 μs) and thus mainly the primary ions formed directly in discharge were detected and conversion to stable negative ions was not finished.

The types and relative intensities of the negative ions detected in these studies differ significantly. The results are very sensitive to many parameters. One of them is the composition of the gas (air, synthetic air), but also trace gases like H_2O, O_3, NO_x and other molecules. The mass spectra of the negative corona discharge in air depends also strongly on the time between formation of the ions and the detection in the mass analyzer as the primary ions formed in the corona discharge are effectively converted via ion-molecule reactions into more stable ions.

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Sekimoto and Takayama [6] carried out APCDI/MS study of ambient air in negative polarity. They studied the influence of the discharge power on the mass spectra. For low power mainly $OH^-(H_2O)_n$ clusters were detected. With increasing discharge power large number of negative ions (OH^-, NO_2^-, NO_3^-, HNO_3^-, CO_3^-, and CO_4^-) and their water clusters was observed. They studied the influence of the humidity on the size of $OH^-(H_2O)_n$ clusters. The size of the cluster ions was in the range n = 1–13 for 24 °C and 25% humidity and n = 1–43 for 24 °C and 50% humidity.

Hill and Thomas [7] measured IMS/MS spectra of pulsed corona discharge in positive and negative polarities in air containing water. In negative polarity they detected large variety of negative ions dominated by mass 60 ($N_2O_2^-$, or CO_3^-) and its water cluster, HCO_3^-, NO_2^-, NO_3^- and large amount mixed cluster ions.

Recently Ewing and Waltman [8] performed IMS/MS study of mechanism of negative reactant ion formation in atmospheric pressure corona discharge in air. They used conventional radioactive ^{63}Ni source of ions. The O_2^- was detected as the major negative ion from ^{63}Ni ion source operated in air. The admixture of the air treated in the corona discharge was introduced into IMS/MS system and NO_3^- was detected as main product, followed by CO_3^-, NO_2^-. The O_2^- ion disappeared from the mass spectrum. They attributed this behaviour to the presence of NO_x in the gas generated by corona discharge and their reactions with primary ions.

In the present study we have applied APCDI/MS and IMS/MS techniques to study the formation of the stable negative ions formed in negative corona discharge. The corona discharge was fed with pure O_2 and O_2/N_2 mixtures. The drift tube was filled in all experiments with pure O_2 so that the ion mobilities are related to this drift gas. The interactions of the primary ions formed in the corona discharge with the gas results in the conversion of the primary ions into stable, terminal negative ions. We applied the APCDI/MS and IMS/MS techniques to identify these negative ions. The IMS/MS technique has been applied to determine the mobilities of the ion in O_2.

There exists several works [9–11] dealing with the mobilities of the negative ions in O_2, N_2, or air with mass spectrometric identification of the ions. These experiments are relative old and have been carried out in low pressure drift tubes and at higher values of the reduced electric fields E/N. The values of the ion mobilities at zero reduced electric field have been obtained by the extrapolation of the high field values to the zero. In present experiment the ion mobilities have been measured at atmospheric pressure, the values of the reduced electric field E/N were close to zero. The ternary ion-molecule reactions play an important role at atmospheric pressure and product may be observed, which were not present in the earlier low pressure studies. In present experiment we have put much attention to the preparation of the working gases, especially, the purification. This is an important issue in the high pressure environment. We have used commercial getter trap to decrease the concentration of main the impurities, such as CO_2 and H_2O to lowest possible levels.

2. Experimental setup

At Comenius University in Bratislava we have built APCDI/MS (Fig. 1) and IMS/MS (Fig. 2) systems in order to study the formation of the ions in the atmospheric pressure discharges and to measure the mobilities of the ions in different drift gases.

A schematic view of the APCDI/MS apparatus is shown in Fig. 1. The APCDI/MS consists of point to plain corona discharge (CD) ion source and the differentially pumped vacuum system equipped with quadrupole mass spectrometer. CD tip was made of tungsten wire with diameter 50 μm and the distance from plane electrode was 5 mm. The plane electrode is on variable potential against the ion collector with pinhole, so that we are able to vary the potential U_d between the plane electrode of the CD and the ion collector with pinhole.

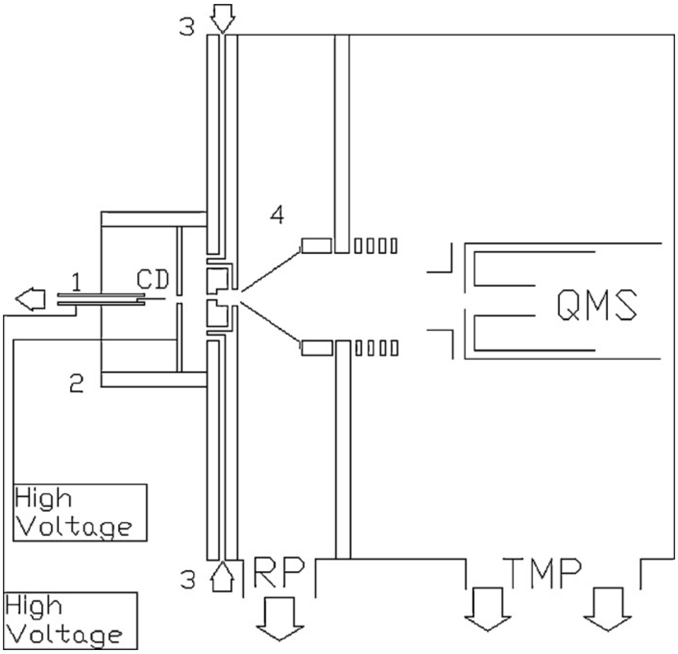

Fig. 1. The schematic view of the APCDI/MS apparatus. HV: high-voltage sources; CD: point to plane corona discharge source; RP: rotary vane pump; TMP: turbomolecular pump; QMS: quadrupole mass spectrometer; 1: corona discharge gas inlet; 2: CD reactor; 3: gas inlet; 4: skimmer and ion optics.

The transport of the ions from high pressure environment into the vacuum was provided by 100 μm pinhole in the collector. The differentially pumped vacuum system consists of three chambers. The first one is pumped by rotary pump to the pressure of 10^{-1} mbar, the second is pumped by a pair of turbo molecular pumps with total pumping speed of 500 l s^{-1}. Additionally the quadrupole mass spectrometer (BALZERS QMG422) is differentially pumped by a 70 l s^{-1} turbo molecular pump. The first and second chambers are separated by skimmer. The orifice of the skimmer is 0.5 mm at 1.5 cm distance to the IMS collector. The ions are transported from pinhole orifice into mass spectrometer by ion optic which consists of skimmer and five electrostatic lenses. The potentials on the lenses of the ion optic have been kept low (below 10 V) in order to avoid collision-induced dissociation. The transport of the ions from atmospheric pressure into vacuum was tested for positive ions formed in positive corona discharge in pure N_2. In such case there are stable terminal ions $H_3O^+(H_2O)_n$ and their size distribution in IMS/MS system is given by the equilibrium constants for clustering and declustering reactions [12]. The mass spectra measured in IMS/MS system were in agreement with theoretical prediction. This indicates that in our system the transport of the ions from atmospheric pressure to vacuum is not distorted either by clustering, or dissociative processes.

The IMS/MS system consists of home made IMS spectrometer and the differentially pumped vacuum system described above. The IMS was manufactured from glass tube with inner diameter of 50 mm, wall thickness 5 mm and the total length of 20 cm. The ionization source, the electrodes of the reaction region and the shutter grid (SG) are mounted on the flange of the IMS and are located inside the glass tube, while the electrodes of the drift region are placed outside the glass tube [13]. The corona discharge (CD) of identical geometry as in the APCDI/MS was used. The electrodes inside and outside IMS were manufactured of aluminium. The SG is of Bradbury–Nielsen design. The opening time of the SG in present

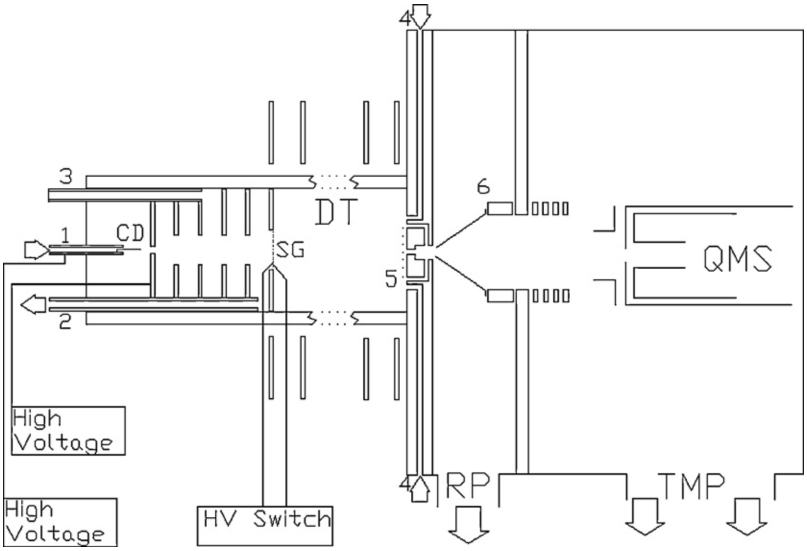

Fig. 2. The schematic view of the IMS/MS apparatus. HV: high-voltage sources; CD: poitn to plane corona discharge source; SC: shutter grid; DT: glas drift tube with external ring electrodes; RP: rotary vane pump; TMP: turbomolecular pump; QMS: quadrupole mass spectrometer; 1: corona discharge gas inlet; 2: gas exhaust; 3: sample gas inlet; 4: drift gas inlet; 5: aperture grid and ion collector with orifice to the vacuum; 6: skimmer and ion optics.

experiment was 100 μs. The electric field in the drift tube is formed by 10 aluminium electrodes located outside the glass drift tube.

The electrodes of the IMS are supplied from a divider. The last electrode of the drift region is connected trough a resistor to the grounded plate at the end of the drift tube. The ion collector is shielded by the aperture grid with a transmittance of 88%. The ion current was processed by current to voltage amplifier 10^8 V/A and the IMS spectra have been recorded by (Picoscope AD card). The distance between the plane electrodes of the CD and SG is 4.8 cm while the drift region length is 12.45 cm. Two high-voltage power supplies (Heinzinger LNC 1000) were used, one for corona discharge and one for drift field of IMS (351.8 V/cm). The SG was controlled by fast high-voltage transistor switch (Behlke Electronic GMH, HTS 21-14) and triggered by TTL generator.

The gas flows were measured by flow meters and were kept constant in present measurements. The drift tube was fed by pure oxygen with flow 1200 ml/min. The ion source was fed by pure oxygen or by nitrogen/oxygen mixture with total flow of 100 ml/min. The measurements were performed at ambient temperature and pressure.

The mobility of ions we calculate using the formula:

$$K = \frac{l_d}{t_d E} \tag{1}$$

where l_d is the length of the drift tube, t_d is the drift time of the ions and E is the electric field in the drift tube. The reduced mobility K_0 is calculated using Eq. (2). The uncertainty in calculated K_0 values is believed to be ±4% in present experiment. As the mobility depends on the collisions of the ion with the drift gas it depends also on the density of the drift gas. Therefore a correction to the standard conditions ($T_0 = 273$ K, $p_0 = 101.3$ kPa) is made and resulting value is reported as reduced mobility K_0 given by the formula:

$$K_0 = K \frac{T_0}{T} \frac{p}{p_0} \tag{2}$$

The gases used in the experiment were pure oxygen and nitrogen (LINDE, 5.0 purity). Additionally we have used the moisture trap (AGILNET X13) in order to reduce the concentration of H_2O under 14 ppb and additional gas purifier have been applied (MICROTORR, MC190-903FV for N_2 and MC190-203FV for O_2) to reduce the impurities under 100ppt (H_2O, CO_2, etc.). The term "pure O_2" in this work refers to oxygen with ~5 ppm of N_2 and less then 100 ppt H_2O and CO_2.

Prior the experiments the IMS tube was thoroughly cleaned. The IMS was evacuated by dry membrane pump down to 2 mbar pressure and heated for several hours up to 200 °C in order to remove impurities from the drift tube walls. After the heating procedure the drift tube has been cooled down to the ambient temperature and flushed several times by the clean drift gas.

3. Results and discussion

3.1. Pure O_2

In previous mass spectrometric studies [5] it was reported that dominant ion in negative corona in O_2 is the O_3^-. The O_3^- ions are effectively formed by several reaction channels. The negative corona discharge in O_2 is source of neutral molecules and radicals one of the most important is the O_3 [14]. Additionally, to the neutral products, primary negative ions O^- and O_2^- are formed in the corona discharge via electron attachment reactions to O_2 [15]:

$$e + O_2 \rightarrow O^- + O \tag{3}$$

$$e + O_2 + M \rightarrow O_2^- + M \tag{4}$$

These primary ions may further undergo binary reactions with O_3 forming O_3^- ions [15]:

$$O^- + O_3 \rightarrow O_3^- + O \tag{5}$$

$$O_2^- + O_3 \rightarrow O_3^- + O_2 \tag{6}$$

As the electron affinity of O_3 (2.10 eV [16]) exceeds those of O (1.46 eV) and O_2 (0.45 eV) both reactions are exothermic. Formation of the O_3^- is possible also via three body reactions [15]:

$$e + O_3 + M \rightarrow O_3^- + M \tag{7}$$

which represent the electron attachment reaction. O_3^- may be formed also via ion-molecule reactions [15]:

$$O^- + O_2 + M \rightarrow O_3^- + M \tag{8}$$

The simulation of the negative corona discharge in pure oxygen in wire to cylinder geometry shows that at large distances from

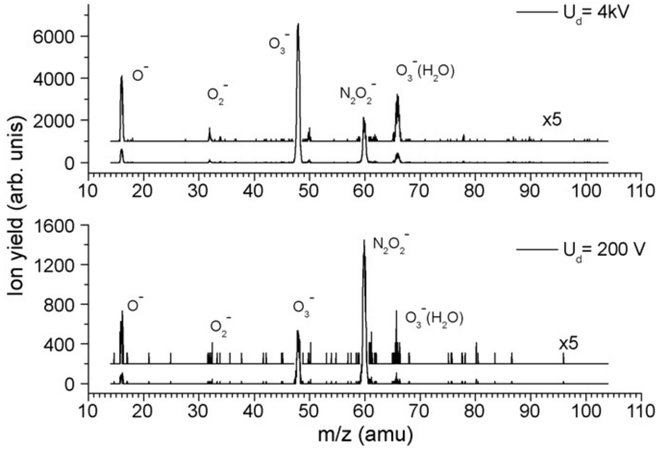

Fig. 3. The APCDI/MS mass spectra of negative corona discharge fed with pure oxygen at two different values of U_d potential. $U_d = 4$ kV, corresponds to fast transport of the ions into vacuum and $U_d = 200$ V corresponds to slow transport of the ions into vacuum.

the wire, O_3^- is the dominant ion [17]. Analogical situation should exist also in point to plane geometry in present experiment.

This model of primary ion formation is confirmed also by present APCDI/MS results for pure O_2. Fig. 3 shows APCDI/MS mass spectra for two different drift potentials U_d. High value of $U_d = 4$ kV (reduced electric field of 18 Td) corresponds to short transit times (~100 μs) of the ions between the plane electrode and pinhole orifice. At this reduced field the dissociation of the molecule should not play an important role, as the average kinetic energy of the ions is about 50 meV. That the mass spectrum is dominated by O_3^- ($m/z = 48$) and also relatively strong signals for O^- ($m/z = 16$) and O_3^- (H_2O) ($m/z = 66$) were detected. The O_2^- is also present, however, very weak. The ion with $m/z = 60$ is already the second strongest peak in the mass spectrum. In the case of $U_d = 200$ V (transit time ~1 ms), we see that the mass spectrum is already dominated by the ion $m/z = 60$.

The nature of the ion with $m/z = 60$ we associate with the most abundant impurity in oxygen, N_2. The presence of N_2 in O_2 results in efficient N_2O formation [18]. The O^- can react with N_2O [19]:

$$O^- + N_2O \rightarrow (N_2O_2^-)^\# \rightarrow NO^- + NO \tag{9}$$

However, we believe that at atmospheric pressure the molecular ion complex could be stabilised by collision with a molecules:

$$O^- + N_2O \rightarrow (N_2O_2^-)^\# + M \rightarrow N_2O_2^- + M \tag{10}$$

Additionally, on the basis of relatively fast decay of O_3^- we suggest, that following reactions could play an important role:

$$O_3^- + N_2O \rightarrow N_2O_2^- + O_2 \tag{11a}$$

$$O_3^- + N_2O + M \rightarrow N_2O_2^- + O_2 + M \tag{11b}$$

The present experiment shows, that the corresponding IMS/MS mass spectrum (Fig. 4) (the total drift time of the ions was ~20 ms) is dominated by an ion with $m/z = 60$ and that the O_3^- ($m/z = 48$) has only very weak intensity close to the detection limit. We assign this ion to $N_2O_2^-$ which has high electron affinity of 3.351 ± 0.010 eV [19]. The $m/z = 60$ has also an important atmospheric ion CO_3^- which has also similar value of reduced ion mobility (2.5 ± 0.07 [9] and 2.51 ± 0.07 [10]). The argument against CO_3^- is the low initial CO_2 concentration in present experiment (below 100 ppt). At this low concentration, the CO_3^- cannot be the main peak in the mass spectrum. According to known kinetic data concerning CO_3^- formation in O_2 [20] the CO_3^- ions can be formed via ion-molecule reaction of O^- and O_3^- with CO_2 to form CO_3^- ions. Under present condition the formation of CO_3^- is slow and the CO_3^- cannot be the dominant ion either in APCDI/MS nor IMS/MS experiment. Therefore we assign the ion with $m/z = 60$ observed in IMS/MS system to $N_2O_2^-$.

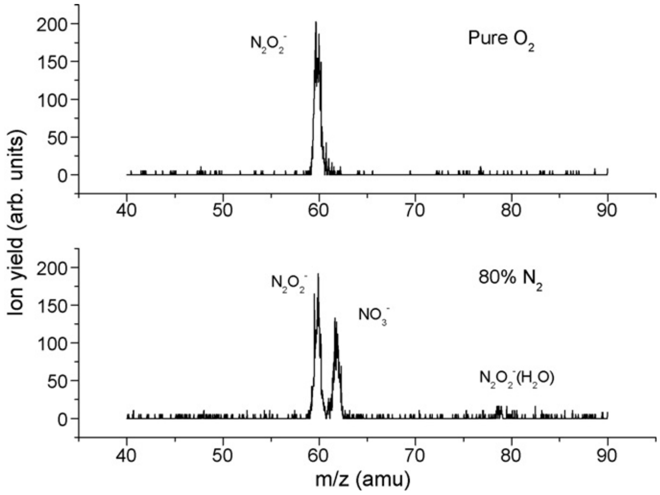

Fig. 4. The IMS/MS mass spectra of the negative ions formed in negative corona discharge in pure oxygen and oxygen with 80% nitrogen. The total ion drift time through the IMS system is approximately 20 ms in oxygen.

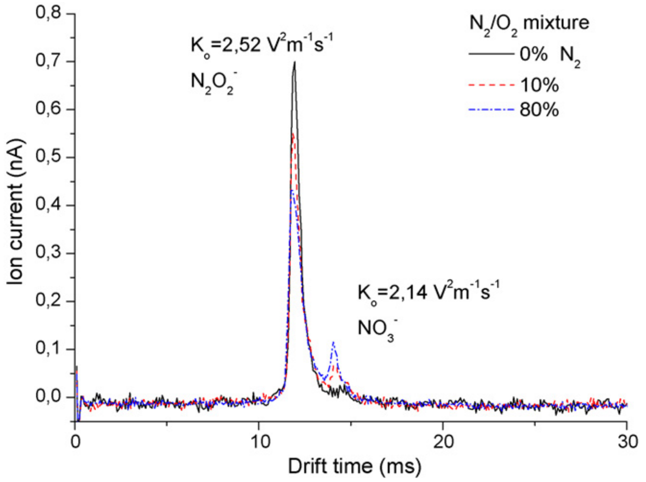

Fig. 5. The IMS spectra of negative corona discharge fed with pure oxygen, and oxygen with 10% and 80% of nitrogen in pure oxygen. The first peak we assign to $N_2O_2^-$ and the second one to NO_3^-.

The IMS spectra of negative ions formed in the negative corona discharge in pure O_2 and in O_2/N_2 mixtures is shown in Fig. 5. The IMS spectrum of pure O_2 shows main peak at 11.95 ms, which corresponds to the reduced mobility of 2.52(±0.05) cm² V⁻¹ s⁻¹. Similar spectrum has been recorded by Stano et al. [21] and the peak has been tentatively assigned to O_3^- (O_3^- ion mobility 2.55 cm² V⁻¹ s⁻¹ [9]) on the basis of the reduced ion mobility and assumption that O_3 is the most abundant product of the negative corona discharge and has the highest electron affinity of all products. It was also assumed that the densities of the impurities such as CO_2 and NO_x are negligible.

3.2. O_2/N_2 mixture in corona discharge

The negative corona discharge in the mixture of N_2 with O_2 results in formation of ozone and nitrogen oxides N_xO_y (NO, NO_2 and N_2O) [22]. The electrons and the primary negative ions O^- and O_2^- react with O_3, NO_2 and form O_3^-, NO_2^- which may be converted to NO_3^- in a reaction with O_3 [23]. The electron affinity of

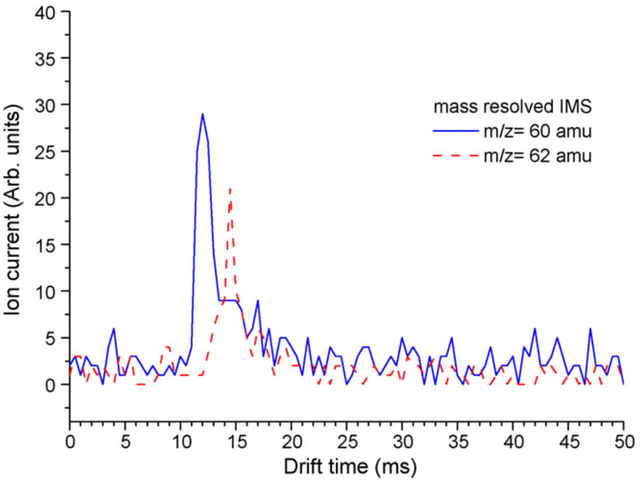

Fig. 6. Mass spectrometrically resolved IMS spectra, as measured at two different $m/z = 60$ and 62 amu.

both NO_2 (2.27 eV) and NO_3 (3.93 eV) exceeds that of O_3 (2.10 eV [16]) and in the case of NO_3 also that of CO_3 and N_2O_2. The NO_3^- has been observed to be the terminal negative ion in dry air [4].

In present experiment the corona discharge was fed by the mixtures of N_2 with O_2 while drift gas was pure O_2. The concentration of N_2 in mixture was varied from 10% to 80%. Fig. 5 shows the ion mobility spectra with 0%, 10% and 80% of N_2.The spectra were measured at the ion collector of the IMS/MS system. In addition to first peak (2.52 $cm^2\,V^{-1}\,s^{-1}$) which was present also in the pure O_2 and which we have assigned to $N_2O_2^-$ we have observed in IMS spectra second peak with reduced mobility of 2.14 $cm^2\,V^{-1}\,s^{-1}$. We see that with increasing concentration of N_2 the intensity of the first peak decreases. The intensity of the second peak increases. In former IMS study in O_2/N_2 mixture and the O_2 drift gas [21] additional peaks with reduced ion mobilities 2.45 and 2.35 $cm^2\,V^{-1}\,s^{-1}$ have been detected. In present experiment we were not able to see these peaks, probably because the resolution of the IMS spectrometer was lower.

In the mass spectrum of the negative corona discharge in O_2/N_2 mixtures (Fig. 4) we see two strong negative ions. The ions with $m/z = 60$ and the ions with $m/z = 62$. The ion with $m/z = 62$ we assign to NO_3^-. NO_3^- is known to be a stable negative ion in dry air due to its high electron affinity of 3.937 eV [16]. Fig. 6 presents mass spectrometrically resolved IMS spectra for $m/z = 60$ and 62. It shows that the ion with $m/z = 60$ ($N_2O_2^-$) has reduced ion mobility of $2.52 \pm 0.05\,cm^2\,V^{-1}\,s^{-1}$. This value of the reduced ion mobility corresponds very well with earlier values of Snuggs et al. [9] and Perkins et al. [10] for CO_3^- in O_2. The ion with $m/z = 62$ (NO_3^-) exhibit reduced ion mobility of $2.14 \pm 0.05\,cm^2\,V^{-1}\,s^{-1}$. This value of reduced ion mobility of NO_3^- is in disagreement with the value of 2.41 $cm^2\,V^{-1}\,s^{-1}$ obtained by Perkins et al. [10]. The discrepancy could be explained by the method applied by Perkins et al. [10] to measure the ion mobilities. They used pure O_2 with small admixtures of N_2O, NO_2 and CO_2 trace gases at several trace gas densities. They claim that the densities varied from few ppm up to few tenths of percents and that the reduced mobilities were obtained by extrapolation of the data to pure O_2 gas.

4. Conclusions

We have detected the primary ions O^-, O_2^- and O_3^- formed in negative corona in pure O_2 using APCDI/MS. Except these ions we have observed also the water clusters of these ions with one water molecule attached and the $N_2O_2^-$ ion. The APCDI/MS mass spectra were dependent on the time of the ion transport from corona discharge into vacuum The primary ions were efficiently converted by ion-molecule reactions into stable negative ions with $m/z = 60$ ($N_2O_2^-$) measured using IMS/MS technique. The reduced ion mobility of this ion has a value of 2.52 $cm^2\,V^{-1}\,s^{-1}$. The negative corona discharge in the mixtures of N_2/O_2 results in formation of two main stable negative ions, $m/z = 60$ ($N_2O_2^-$) and ion with $m/z = 62$ (NO_3^-). The reduced mobility of NO_3^- was determined to be 2.14 $cm^2\,V^{-1}\,s^{-1}$. Due to low water concentration in the gases (below 100 ppt) we see only very weak intensities of the water clusters ions $M^-\,(H_2O)_n$ ($n = 1$ and M corresponding to $m/z = 60$ and 62).

Acknowledgments

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