Responses to Referee #1

A chemical analyzer for charged ultrafine particles

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Atmos. Meas. Tech. Discuss., 6, 3859-3882, 2013.

The review is in general positive. We greatly appreciate the reviewer's thoughtful comments that helped improve the manuscript. Thank you very much for your time and effort! We trust that all suggestions have been addressed accordingly in the revised manuscript. We respond to the specific comments as follows:

1: *page 3864, line 1 (and in the following): Did the authors consider the production of OH from the corona discharge? This could also lead to the oxidation of gas phase substances and their subsequent partitioning into the aerosol phase. A short discussion should be added.*

The production of OH from the corona discharge was not considered quantitatively because it is difficult to measure. Due to the soft nature of our corona discharger the production of OH is assumed to be small but cannot be excluded completely.

As recommended, a short qualitative discussion has been added to the corresponding paragraph:
To avoid the chemical alteration of the aerosol sample through oxidation, the production of ozone, OH and other oxidizing gas phase species has to be prevented or kept as low as possible. The design of our charger is based on the development by Han et al. (2008). In order to produce an efficient corona with a negligible production of oxidizing species, the charger is equipped with two carbon fiber bundles, with about 100 fibers per bundle and a fiber diameter of about 25 µm, instead of a corona needle. The thin fiber diameter and the large number of fibers comprised in the bundle allows the production of sufficient ions for charging aerosol particles in environmentally relevant concentrations, while still applying relatively low voltages of 2 kV, thus minimizing the production of ozone and other oxidants. Only the production of ozone was monitored in the laboratory due to a lack of measuring capabilities for OH radicals. Ozone mixing ratios were below 1 ppb, which is the detection limit of the used ozone monitor (Model 49i, Thermo Scientific, Franklin, MA, USA). Based on these results, the production of other oxidizing species like OH cannot be excluded but is considered to be small.

2: *page 3864, line 28: How does the “pre-separator” work? It seems that this is a crucial aspect of the design when using the unipolar corona charger. Therefore, the operational principle and the design of the “pre-separator” should be mentioned.*

A conventional pre-impactor with a high pressure drop, e.g. a low pressure impactor with a lower cut-off at 50 nm and a pressure drop of 900 hPa, is not recommended due to the potential evaporation of volatile compounds under the reduced pressure conditions of 100 hPa. Therefore, we suggest using a metal fiber filter designed to have a high penetration efficiency in the size range between 5 nm and 50 nm. For example, Otani et al. (2007) report a 50 % cut-off diameter at 50 nm particle diameter for a fiber diameter of 8 µm, a filter length of 8 mm, a filtration nozzle diameter of 4 mm, and a flow velocity of 30 m s^{-1}. This configuration exhibits a pressure drop of about 130 hPa, i.e. the pressure is reduced from 1000 hPa to about 870 hPa. With this relatively small pressure drop, we expect that evaporation of volatile compounds will be negligible in this pre-separator, while particles larger than 50 nm diameter will be efficiently removed.

The corresponding paragraph was extended to:

The perturbing influence of doubly charged particles could be minimized by applying a pre-separator prior to the unipolar charger, preventing particles with diameters above 50 nm to enter the charger. Otani et al. (2007) report of a metal fiber filter having a high penetration efficiency in the size range between 5 nm and 50 nm exhibiting only a small pressure drop of 130 hPa.

3: *page 3865, line 12: The way the sentence is formulated it is not clear if the rDMA is an exact copy of the design by Zhang et al. (1995) or whether it has further been optimized. If it has been optimized then the modifications should be described.*

Indeed, the geometry of our rDMA is slightly different in comparison to the original design. Mainly, the distance between the aerosol inlet slit and the central outlet was reduced, optimizing the design towards the selection of smaller particles.

Sentence on page 3865, line 22, was changed to:
In contrast to the original design by Zhang et al. (1995) the spacing of the two electrodes is 9 mm while the radius between the annular aerosol inlet and the central outlet is 30 mm, shifting the design's operation range towards smaller particles.

4: *page 3866, line 1 (or end of section 2.2): Has the performance of the rDMA been validated against theoretical transmission curves? Stolzenburg and McMurry (2008, Aerosol Sci. Technol.) report the theory for calculating the expected transmission efficiency in a tandem DMA set-up as function of the DMA geometries, applied flow rates and voltages. In order to demonstrate that the newly set-up DMA selects particles accurately with respect to their sizes; these theoretical response curves could be added to Fig. 3. In any case, a discussion should be included whether the SMPS which is used as a reference yields the peak concentration at the same diameters as the rDMA.*

The theoretical transmission curves for the tandem DMA system have been added to Fig. 3. The curves are calculated according to Stolzenburg and McMurry (2008) and Zhang and Flagan (1996) including diffusional broadening.

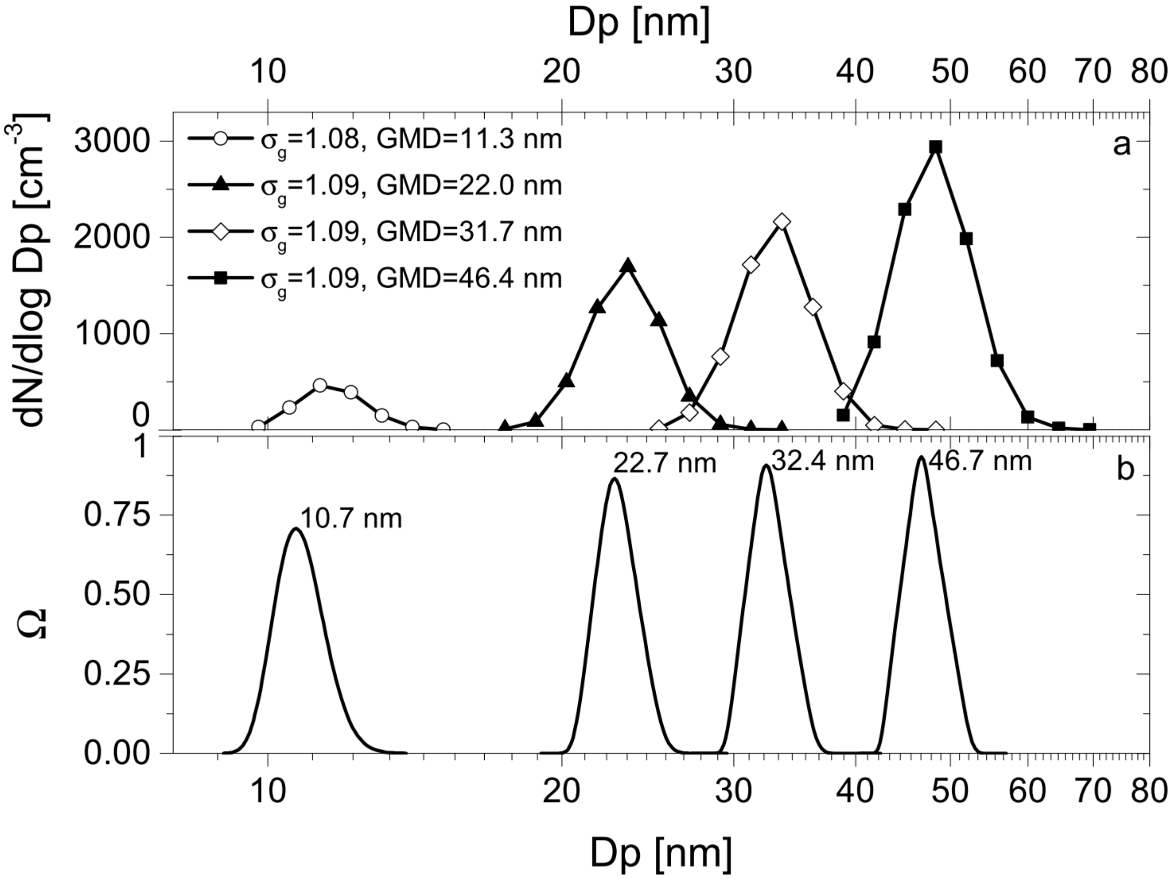

Fig. 3. Comparison of the performance of the rDMA coupled to an SMPS system (a) with theoretical transfer functions (b). A sheath flow of 4 SLM and voltages of 116 V, 504 V, 1003 V and 2000 V resulted in geometric mean diameters measured by an SMPS system of 11.3 nm, 22.0 nm, 31.7 nm and 46.4 nm, respectively. The theoretical transfer functions peak at 10.7 nm, 22.7 nm, 32.4 nm and 46.7 nm, respectively.

The following paragraph was added to page 3866, line 1:
Additionally, the theoretical transfer functions including particle diffusion (Stolzenburg and McMurry (2008), Zhang and Flagan (1996)) for the sequential setup of the rDMA and the cylindrical DMA as described above are shown. The maxima of the calculated transfer functions lie within a maximum deviation of 0.7 nm of the geometric mean diameter calculated from the size distributions of the tandem DMA measurements. Differences between the measured distributions (Fig. 3 a) and the theoretical transfer functions (Fig. 3 b) are thought to be due to additional broadening in the real flow path through the rDMA and the SMPS's cylindrical DMA.

***5:** page 3866, line 20: How can it be assured that both the sample flow and the purge flow have the same velocity at the upper edge of the collection tube? The geometry shown in Fig. 4 is quite complex and later in the manuscript (page 3873, line 16) it is indicated that the flow could be turbulent in this region. This inconsistency should be resolved.*

Since the purge flow, the sample flow and the geometry of the sampling unit are known, it is possible to calculate a theoretical velocity profile for the sampling unit. The purge flow was adjusted to have the same velocity at the upper edge of the collection tube as the sample flow at the constriction between the edge of the collection tube and the unit's housing.
We agree with the referee that the geometry of the sampler is quite complex. Hence, the above mentioned calculations do not guarantee a laminar flow at the collection tube, but still seem

reasonable. The turbulent flow mentioned on page 3873 is very likely an issue in the desorption region, where the purge flow and the sample flow are in opposite directions.

The paragraph on page 3873, line 15 has been extended:
To tackle this issue the collection unit must be optimized towards a flow regime with minimal turbulence, hence less penetration of the gas phase onto the NiCr filament and into the desorption region. Turbulence in the desorption region is assumed to be of greater importance due to the opposing direction of the N_2 purge flow and the sample flow, as well as due to its enhanced surface compared to the filament.

***6:** page 3866, line 28: I have the impression that the word "sealed" is not correct here. If the PEEK piston would indeed align with the conical cavity then it would not be possible to flush the filament with nitrogen (from inlet 8) while the aerosol material is being desorbed and transported into the mass spectrometer.*

The PEEK piston does actually seal the desorption chamber, and the filament is not flushed with nitrogen during desorption. The sealing of the desorption chamber can be seen in a continuous pressure drop within the mass spectrometer during the desorption phase.

***7:** page 3867, line 2: How is the temperature controlled? Probably this is done by varying the current through the filament. However, is the temperature of the filament also measured? Values for the filament temperatures are given throughout the manuscript without describing how these were actually determined. This procedure needs to be described.*

The referee is right to assume that the temperature is controlled by varying the current through the filament. We do not continuously measure the temperature of the NiCr filament, since every temperature gauge having physical contact with the filament would necessarily result in a cooling of the thin wire (diameter of 0.22 mm). A remote measurement, e.g. by means of infrared, is also not suitable due to the small size of the wire, the major fraction of the temperature signal would originate from the surrounding materials. We assess the temperature of the wire with a theoretical calculation using the Stefan Boltzmann law, based on the power applied to the wire, its surface area and its emissivity. This assessment is validated by estimating the temperature from the filament's annealing color. From these considerations, we estimate a final filament temperature of 800 °C at a current of 1.7 A.
To estimate the temperature during the gradual filament heating we applied a combination of both methods and used the following equation according to the Stefan-Boltzmann law:

$$T = \sqrt[4]{\frac{\varepsilon P}{A\sigma}} - T_{cor}$$

Here, $\boldsymbol{\varepsilon}$ is the emissivity of the filament (0.8), $\boldsymbol{P}$ [W] is the power supplied to the filament, $\boldsymbol{A}$ [m^2] is the surface area of the filament, $\boldsymbol{\sigma}$ [$5.67 * 10^{-8}$ W m^{-2} K^{-4}] is the Stefan-Boltzmann constant. The term $\boldsymbol{T_{cor}}$ is an empiric correction, since the Stefan-Boltzmann law only takes the radiative heat loss of the filament into account, without considering the convective heat transport occurring in a gas. $\boldsymbol{T_{cor}}$ is simply the temperature difference between the theoretical calculation at a current of 1.7 A and the estimate through the filament's annealing color at the same current.

We added the following paragraph to page 3867, line 2:

The wire temperature is estimated based on the Stefan-Boltzmann law corrected with an optical temperature estimation through the wire's annealing color. The correction is necessary since the Stefan-Boltzmann law overestimates the temperature, only accounting for radiative heat transport. However, a considerable part of the heat is transported from the wire towards the connecting sockets as well as by convection to the surrounding gas phase.

8: *page 3868, line 28 (and in the following): How is it known that the flow still contains a considerable amount of gas-phase organics and that ~90% of these could be removed with a charcoal scrubber? Was this measured with the same mass spectrometer in "gas-phase mode"?*

The gas phase was measured with the same mass spectrometer by simply turning off all purge flows but maintaining the sample flow from the flow tube at 1 SLM. With this setup, the measurements with the denuder showed a more than 90 % smaller signal in comparison to measurements without the denuder. This difference was considered the gas phase being removed in the denuder.

On page 3868, line 28 was changed to:
The aerosol flow still contained a considerable amount of gas-phase organics, as could be seen by turning off the two nitrogen purge flows, hence sampling with the mass spectrometer directly from the gas phase in the sample flow.

9: *page 3869, line 14: If the signal from silicone (m/z 207) shows a constant intensity throughout all the measurements, why is it then used to normalize the other signals? This would mean that the other signal intensities are divided by a constant number. Therefore, the normalization would be useless.*

The original sentence was somewhat misleading. Actually, mass 207 did not show a constant signal intensity throughout all measurements, but varied according to the mass spectrometer's operating conditions, like ionization efficiency and pressure changes. In contrast, other m/z values were additionally influenced by the desorption of the sample as well as by the sealing of the desorption chamber. Thus, the normalization reduced the signal variability from the mass spectrometer's operating conditions.

Paragraph on page 3869, line 12 was changed to:
Therefore, a normalization of the data was performed by dividing the unit mass resolution sticks by the peak area of m/z 207. Mass 207, being a typical contamination signal from silicone, was always visible in the measured mass spectra, showing variations only due to the mass spectrometer's operating conditions, like ionization efficiency and pressure changes. This mass is suitable for normalization because its signal strength is independent of the evaporation of the collected sample.

10: *page 3870, line 2: A temperature of 800 °C can be realized at the filament. Again, it should be pointed out how this was determined. In addition, at this high temperature the PEEK material should melt (melting temperature ~340 °C). How is this prevented?*

The NiCr filament is attached to the PEEK piston via two sockets. As the two sockets are relatively big in comparison to the filament, significant cooling was observed due to the heat flux towards the sockets. In other words, the temperature on the wire is not evenly distributed but increasing gradually towards its center. As mentioned above the temperature of the wire was not directly measured, but estimated from a theoretical calculation and corrected with an estimate from its annealing color.

The corresponding paragraph was changed to:
Once the piston reached its final position, the desorption chamber was heated from 100° C to 160° C. Only then the NiCr filament is heated gradually to its final temperature of about 800 °C. The wire temperature was not directly measured, but estimated using the Stefan-Boltzmann law and by visual inspection of its annealing color.

11a: *page 3870, line 3: How is it known that some of the signals arise from the gas phase? Was the HV turned on and off and then the residual signal was attributed to the gas phase?*

Indeed, the HV was turned on and off in order to estimate the gas phase contribution to the mass spectra. During blank measurements with no voltage applied to the NiCr filament, organic signals were observed in the mass spectra, being somewhat smaller than the signals from the collection experiments. The rise in signal during blank measurements was attributed to the gas phase. Without the denuder attached to the setup, these blank signals were significantly higher.

11b: *page 3870, line 12: Again, the normalization with the silicon signal doesn't seem to be useful if it is constant throughout all measurements.*

The corresponding sentence was changed to:
For normalization, the peak at m/z 207 was used, showing variations only due to the mass spectrometer's operating conditions, thus being independent of the desorbed sample.

12: *page 3871, line 3: This sentence should be reformulated. It is not clear why there is no restriction on the collection times. This is clearly not the case because for very short times the signals will be below the detection limit.*

The corresponding sentence was changed to:
Overall, the collection experiments show good performance of our instrument in flow tube experiments. The collection times can be extended to multi-hour periods and the use of a radioactive neutralizer is reasonable despite its poor charging efficiency, minimizing the contribution of multiply charged particles in the sample.

13: *page 3871, line 23: The method for estimating the collected aerosol mass should be described in more detail. It seems that one requirement is that the particle number concentration coming from the rDMA is constant over time. Another assumption seems to be that the number of particles which are collected on the wire can be determined from the difference in particle concentration between HV on and HV off periods, i.e. losses at the exit of the collection unit as well as losses close to the collection tube are neglected. In this respect it would be interesting to know what the transmission of particles is when the HV is off and when the particle concentration is measured simultaneously at the inlet and the outlet of the collection unit.*

For estimating the collected particle mass the aerosol concentration was measured downstream of the sampling unit before, after and during the collection. The concentrations measured after collection showed 3 %, 11 % and 18 % higher concentrations for the 0.5 h, 1 h and 2 h experiment, respectively. This increase in concentration was considered to be linear over time. The collected particle number was therefore calculated as the average between concentration before and after collection minus concentration during collection and corrected for the unit's transmission efficiency.

To characterize the particle transmission for 25 nm particles, concentrations were measured upstream and downstream of the sampling unit. This was performed with and without the N_2 purge flows enabled. Prior to the transmission experiment without the purge flows, a modification in the collection unit’s exhaust had been realized. This modification resulted in less particle losses at the unit’s exhaust and an optimized N_2 purge flow in the desorption region. The transmission through the unit with the new exhaust, without purge flows, was 81 %. The transmission through the unit’s original design but with purge flows enabled was 38 %. Considering the dilution by the purge flows, the transmission is close to 100 %. Consequently, we assume a transmission of 81 % to 100 % for our collection unit. By assuming that all losses in the collection unit happen at the exhaust (maximum 19 %) the collected mass will be enhanced by a maximum of 23.5 % (19 % / 81 % *100) compared to the estimate without losses.

Fortunately, we realized a mistake in the calculation of the collected particle sample during the process of replying to the referee's comments. The initially calculated masses of 7, 34 and 90 ng are incorrect. The corrected values are 2.1-2.6, 4-5 and 5.3-6.5 ng. This error does not greatly influence the estimated detection limit, since it is still in good agreement with the dilution series experiments.

The paragraph at page 3871, line 19 was extended:

The particle transmission through the collection unit was estimated by measuring the particle concentration upstream and downstream of the unit's sample inlet and outlet, respectively. Accounting for the dilution with the nitrogen purge flows the transmission was estimated to be between 81 % and 100 %. Applying the corresponding collection and transmission efficiencies together with an assumed particle density of 1.25 g/cm^3 (average estimate of the density values by Bahreini et al. (2005) and Kostenidou et al. (2007)) and knowing the particle size and concentration as well as the aerosol volume flow through the collection unit, an estimate of the collected mass was obtained. For the three performed experiments, rough estimates of the collected masses were in the range of 2.1-2.6 ng, 4.0-5.0 ng and 5.3-6.5 ng for the 0.5 h, 1 h and 2 h experiments, respectively. Since collection times below 0.5 h did not result in quantitative signals from the mass spectrometer the lower detection limit for the performed experiments is estimated to be below 2.1-2.6 ng, being in the same range as determined by the dilution series experiments.

14: *page 3871, line 27: A short discussion could be added about the detection limit for different substances. The detection limit of 7 ng is determined for a rather complex system where many different peaks occur in the mass spectrum. For a pure compound with just a few peaks present, the detection limit could be substantially better.*

To page 3871, line 28 the following paragraph has been added:

However, the detection limit will depend also on the composition of the collected particles. For example, for compounds experiencing less fragmentation by the mass spectrometer's electron ionization the detection could be substantially better.

15: *page 3872, line 27: To my knowledge no impactors are available for these small sizes which work at pressures around 1 bar. Only when the pressure is substantially lowered these low cut-off sizes can be realized with impactors. Please clarify how these small cut-off sizes can be realized with the current set-up.*

This refers to the same principle as now described on page 3864, line 28.

16: *page 3881 (Fig. 5) and page 3882 (Fig. 6): Fit curves could be added to these figures to indicate the linearity.*

Linear regressions have been added to Figures 5 and 6 (now 7).

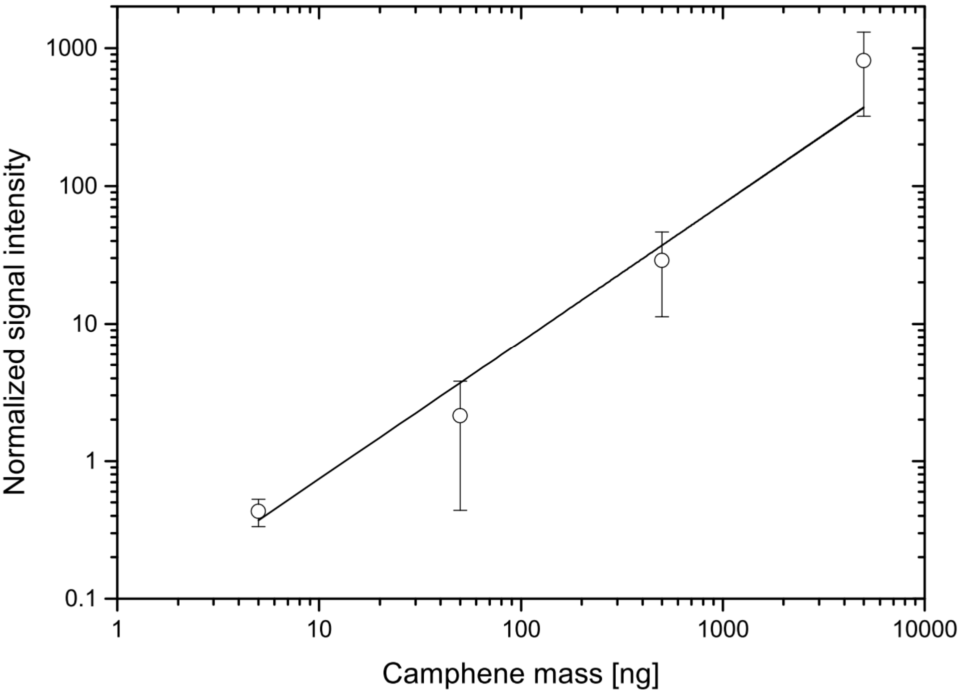

Fig. 5. Detection performance of the aerosol mass spectrometer for different camphene masses applied to the desorption filament. Normalized signal intensity equals the sum of the integrated peak areas of m/z 93, 121 and 136 normalized with m/z 207. Open cycles denote the mean signal intensity and the error bars show the standard deviation. A linear regression is shown to indicate the linearity of the signal.

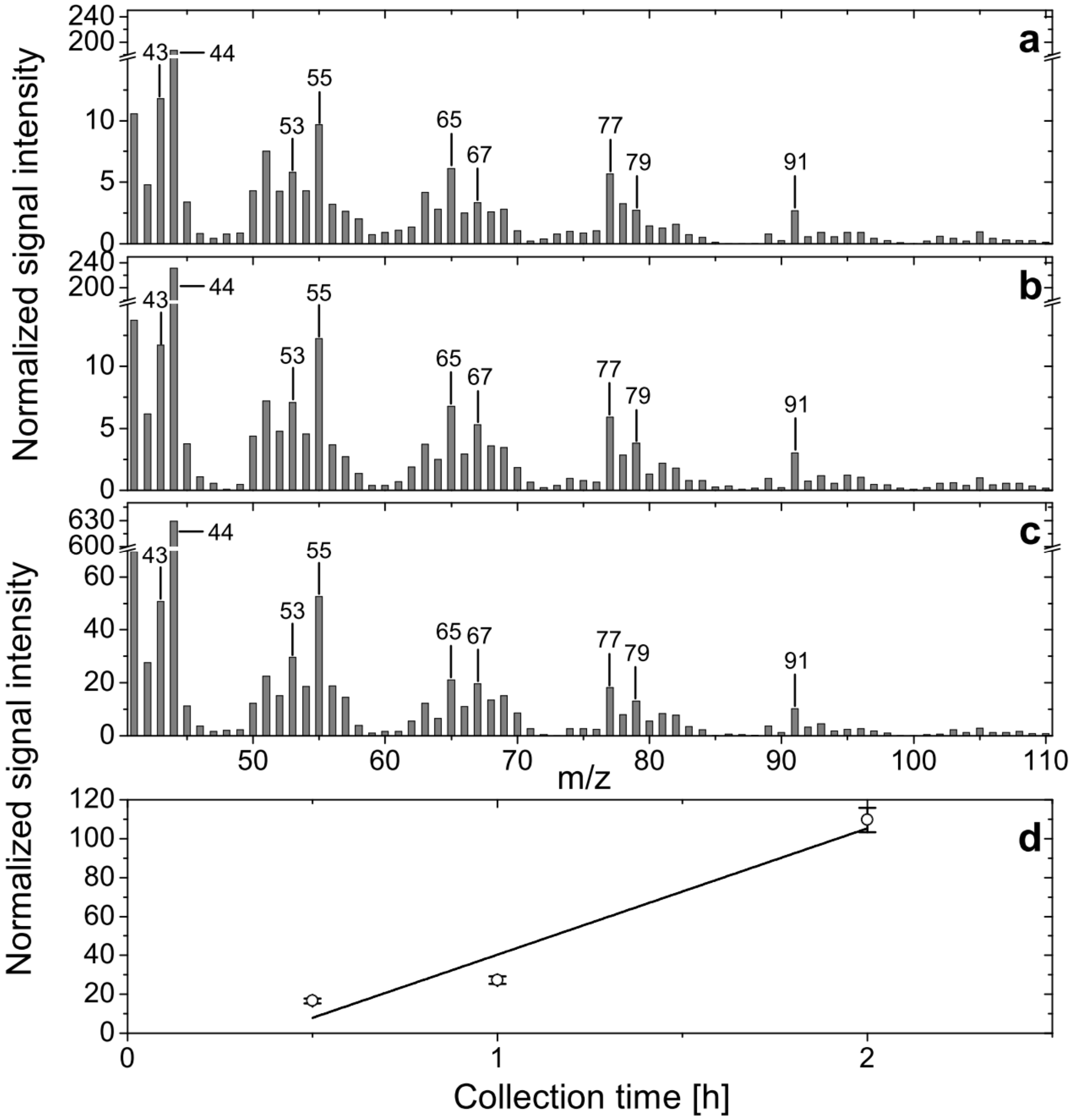

Fig. 7. Mass spectra of organic particles with a diameter of 25 nm from dark ozonolysis of alpha-pinene; a, b and c are spectra after collection times of 0.5 h, 1 h and 2 h, respectively. The lower panel (d) shows the sum of the signal intensities of four major peaks (m/z 55, 65, 77 and 91) in relation to the collection time.

***17:** page 3882, Fig. 6: In panel d the x-axis should range from 0 to >2 h, currently it starts somewhere around 15 min. Although the signal clearly increases for longer collection time, there seems to be a deviation from linearity here. Please add some discussion why this could be the case.*

The deviation from linearity is due to uncertainties in the mass determination of the collected particle sample, and due to the fact that the collected sample mass is probably close to the detection limit of this specific setup, especially for the 0.5 h sampling period.
The collection efficiencies of the three experiment varied slightly, and the absolute particle concentration produced by the flow reactor was subject to small variations as well.

The following discussion was added to page 3871, line 1:
The increase in signal with collection time is not perfectly linear. This may be due to the fact that the collected masses are probably close to the detection limit. Additionally, variations of the collected

mass due to slight differences in collection efficiency of CAChUP, and particle number concentration produced in the flow tube may also be an issue.

***18:** Additional remark:*

I did not understand how the signals in Fig. 5 and Fig. 6 d were exactly derived. Clearly the signal at each mass was integrated over a certain period. However, when heating the wire the aerosol material won't come off all at once. Depending on the temperature the signal will probably rise and then decay after a while. Therefore, could you please add an additional figure which shows the time dependency for one representative m/z value. In this figure the wire temperature should also be shown. One very important aspect is also after what time the signal reaches background level although the heating still persists. In this respect it would also be interesting to discuss potential memory effects. E.g. if the aerosol sample is desorbed and the heating current is turned off and then turned on again after a while, do you see any rise in the signals? In other words how good does the cleaning procedure of the wire work and how exactly is this cleaning performed?

An additional figure has been added to the manuscript:

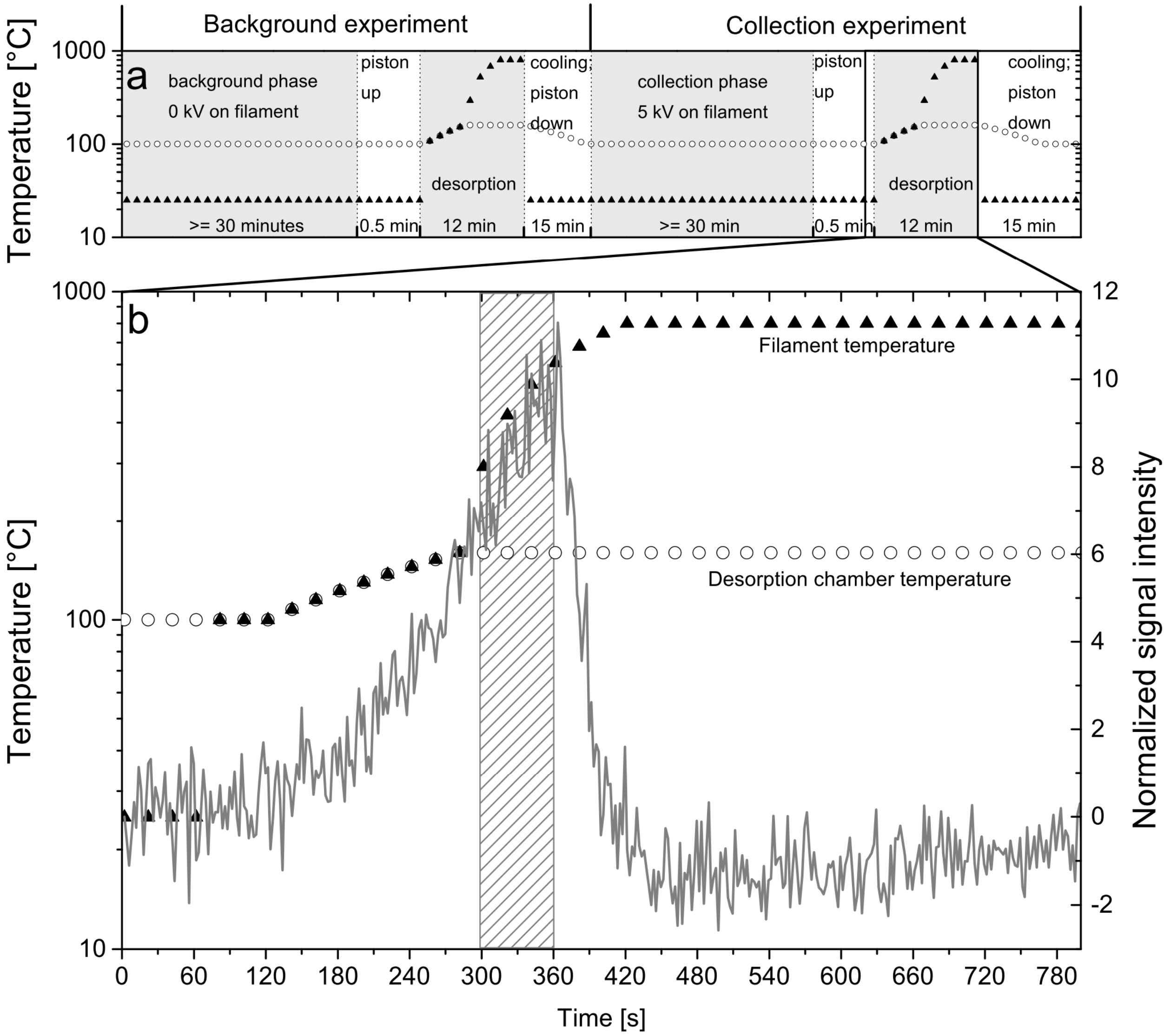

Fig. 6. (a) Sketch of the procedure for the background and collection experiments, with temperature courses of the desorption chamber (white circels) and the NiCr filament(black triangles). (b) Time evolution of the averaged signal from major organic peaks (m/z 43, 53, 65, 67, 77 and 91) during the desorption of oxidized alpha-pinene particles. The PEEK piston was in desorption position at second

60. Temperatures of the desorption chamber and the filament are also shown. The hatched area denotes the one minute averaging periode used for the mass spectrum shown in Figure 7 b.

The following paragraph has been revised on page 3869, line 21:
Figure 6 (a) shows the procedure of the particle collection and blank experiments. Aerosol particles with a diameter of 25 nm were collected for time periods of 30 min, 1 hour and 2 hours, and desorbed from the NiCr filament. During collection, the desorption chamber was continuously kept at 100 °C and flushed with nitrogen. Also the filament was flushed with nitrogen, minimizing the collection of gas phase compounds and interactions of the collected sample with oxidants like ozone. Following the collection, the nitrogen flow is increased in order to flush the entire collection unit, preventing contamination during the travel period of the piston towards the desorption chamber. Once the piston has reached its final position, the desorption chamber is heated up to 160 °C. Only then the NiCr filament is heated gradually to its final temperature of about 800 °C (see Fig. 6 (b) for details). The wire temperature was not directly measured, but estimated from the Stefan-Boltzmann law and by visual inspection of its annealing color. In Figure 6 (b) the desorption process for the one hour collection experiment is shown. Heating of the desorption chamber was initialized 60 seconds after the PEEK piston was moved to the desorption position. After 300 seconds the final desorption chamber temperature of 160° was reached and the filament was heated from ~300 to 800 °C during the next two minutes. Also shown in Fig. 6 (b) is the time evolution of signal from averaged major peaks from the evaporated particles. The signal from the TOF-MS is clearly increasing due to the heating of the desorption chamber and reaching its maximum well before the filament reached its final temperature. Once the maximum was reached a steep decrease in the signal was observed, despite the persistent heating of the filament. Remarkable is that the signal seems less dependent from filament heating than from the desorption chamber's temperature. The grey hatched area in Fig. 6 (b) indicates the averaging period from which the mass spectrum in Fig. 7 (b) was obtained. The one minute averaging period was chosen for all experiments, always starting when the desorption current was applied to the NiCr filament.

Minor comments:
page 3860, line 3 (also line 24): "the Earth's": done
page 3860, line 9: "subsequently" instead of "consequently"? : done
page 3860, line 13: maybe better to use the word "defined" instead of "known" : done
page 3860, line 24: maybe better to use the word "significant" instead of "eminent" : done
page 3861, line 4: instead of "after sunrise" please reformulate to "in the morning hours" : done
page 3861, line 27: "ion trap", two words; the word "trapped" could be replaced by "captured" : done
page 3862, line 17: replace the word "and" by "while" : done
page 3867, line 4: please delete the word "to" after "heated" : done
page 3871, line 7: Please replace "with growth rates close to natural rates" by "with similar growth rates that can be observed in the atmosphere" : done
page 3872, line 17: Using one defined size of 20 nm is not appropriate to report an aerosol concentration; instead a size interval should be used. : done
page 3873, line 6: Please delete the word "By": done
page 3873, line 9: Please delete the word "of": done
page 3880, Fig. 4: adding a scale to this figure would be useful: done

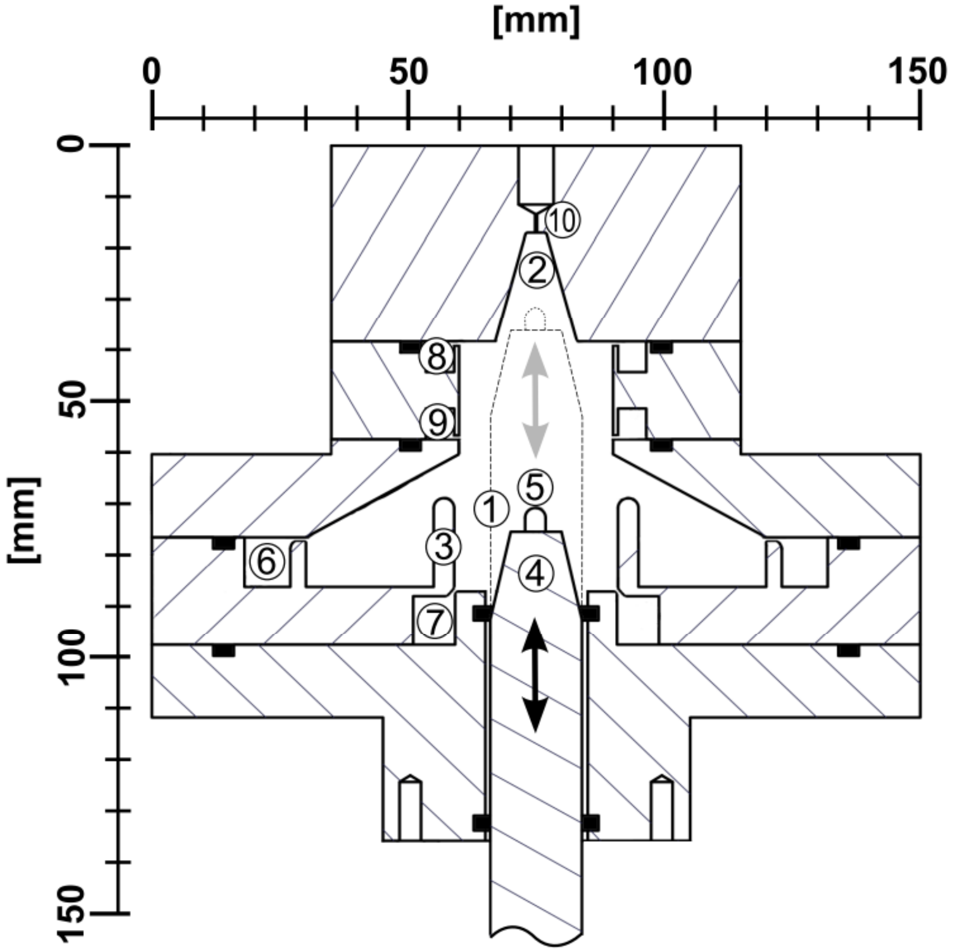

Fig. 4. Schematic of the radial collection and desorption unit with NiCr filament in collection position: (1) collection region, (2) desorption region, (3) collection tube, (4) PEEK piston, (5) collection filament, (6) aerosol inlet, (7) collection flushing inlet, (8) desorption flushing inlet (9), exhaust and (10) screw connection for transfer capillary. Positions of O-rings are shown in black.

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