1 # Aerosol source apportionment from 1-year-measurements

2 # at the CESAR tower at Cabauw, NL

3

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1   **Responses to anonymous referee #1**

2   *Received and published: 18th January 2016*

3   We thank the reviewer for the careful review of our manuscript; the comments and

4   suggestions are greatly appreciated. All the comments have been addressed. In the following

5   please find our responses to the comments one by one and the corresponding changes made to

6   the manuscript. The original comments are shown in italics.

7

8   *1) The scientific quality and presentation of the manuscript are good. The manuscript will*

9   *need copy-editing because there are numerous grammatical errors, although the text is still*

10   *clear. There is agreement between the various instruments utilised at the site (ACSM vs SMPS*

11   *vs MARGA), and the PMF analysis follows established procedures. I do have questions about*

12   *the determination of organic nitrates and about the correlations between the OA components*

13   *and tracers, and these questions are given below. On the other hand, the scientific*

14   *significance of the manuscript is not clear. Aerosol mass spectrometry (AMS) including*

15   *ACSM measurements are fairly standard now, and AMS measurements were previously*

16   *carried out at this site, as indicated in the manuscript. I acknowledge that the long duration of*

17   *the measurements in this manuscript is somewhat novel, but it is not clear how having simply*

18   *more measurements provides new significant insight into atmospheric chemistry. This*

19   *concern will need to be addressed before final publication, in addition to the comments*

20   *provided below.*

21   **Response:**

22   Fortunately, copy-editing is standard in ACP. We are relieved though, that despite some

23   language issues the manuscript is understandable and clear.

24   The reviewer is right that a publication just presenting longer records obtained with AMS or

25   ACSM is not per se novel enough to warrant publication in a quality journal such as ACP,

26   also considering that two AMS campaigns were previously performed in Cabauw. We

27   emphasize here that the focus of this work extends beyond the presentation of a larger data set

28   for this site:

29   In this study, we showed that the major fraction of atmospheric aerosol at this site is

30   comprised of secondary aerosol (SA) which is chemically formed in the atmosphere. SA also

31   dominates the high mass loadings during the pollution episodes exceeding air quality

32   standards. The long term aerosol mass spectrometric data provided by this work is important

1 for the understanding of the chemical processes leading to such high aerosol masses which are

2 unexpected for a rural site like Cabauw. This information is necessary for potential mitigation

3 of particle masses. The findings presented here were not seen in previous studies due to their

4 limited time of sampling.

5

6 ***General Comments:***

7 *2) Organic nitrates: The difference in the concentration of nitrate measured by the ACSM and*

8 *MARGA is not a direct means of identifying and quantifying the presence of organic nitrates.*

9 *Is there other evidence from the ACSM or other instruments to support the conclusion that*

10 *these compounds are present and account for 9% of the total ACSM NO3? If not, then this*

11 *conclusion is rather weakly supported by the data.*

12 **Response:**

13 We followed a procedure given by Xu et al. (2015) who calculated the organic nitrate fraction

14 by subtracting the inorganic nitrate concentrations measured by a particle-into-liquid sampler

15 (PILS, see Orsini et al. (2003)) from ToF-AMS total nitrate concentrations. Those instruments

16 have similar uncertainties as the respective instruments reported here. In the revised

17 manuscript we provide following additional evidence for the presence of $OrgNO_3$ in the

18 aerosol: the use of external data from the MARGA to determine the inorganic nitrate in the

19 ACSM data set improved the agreement of measured against predicted $ACSM-NH_4$, resulting

20 in a nearly 1:1 regression line without a significant offset. This is in agreement with the

21 MARGA internal ion balance which also indicates neutralized inorganic aerosols. All

22 evidence together makes a strong case that the difference of AMS total nitrate to MARGA

23 nitrate is most likely due to the presence of organic nitrates. These findings and the previously

24 reported relatively high AMS organic nitrate fractions by Mensah (2011) in May 2008

25 ($0.5\,\mu g\,m^{-3}$, 35% of total nitrate, 5.2% of total aerosol mass) and March 2009 ($0.2\,\mu g\,m^{-3}$,

26 10% of total nitrate, 3.6% of total aerosol mass) in Cabauw show strong evidence of the

27 presence of organic nitrates during this campaign and the reliability of its estimation as

28 presented in the manuscript.

29 To provide a more detailed description this part was changed in the revised manuscript as

30 follows:

31 "This assumption is acceptable, as shown by using the $MARGA-NO_3$ instead of the ACSM-

32 total-NO_3 for the ion balance of ACSM data (including $ACSM-SO_4$, -Chl and -NH_4),

following a procedure given by Xu et al. (2015) who calculated the organic nitrate fraction by subtracting the inorganic nitrate concentrations measured by a particle-into-liquid sampler (PILS, see Orsini et al. (2003)) from ToF-AMS total nitrate concentrations. In the Cabauw data set, the correlation of measured against predicted NH_4 resulted in a nearly 1:1 regression line without a significant offset (Fig. S10). This is in agreement with the MARGA internal ion balance which also indicates neutralized inorganic aerosols. Therefore, the mass concentration of nitrate groups associated with organic molecules (hereafter called organic nitrate or $OrgNO_3$), can be estimated by subtracting the MARGA-nitrate from the ACSM-nitrate concentration. The $OrgNO_3$ time series using this approach is plotted in Fig. S11, the respective diurnal variation averaged over the whole campaign in Fig. S12. An average mass fraction of 9% was calculated for $OrgNO_3$ (average concentration: 0.43 µg m^{-3}) in respect to total ACSM-NO_3. The organic nitrate fraction shows a maximum concentration in the night, followed by a decrease during the day. These findings are in agreement with previously reported relatively high AMS organic nitrate fractions by Mensah (2011) in May 2008 (0.5 µg m^{-3}, 35% of total nitrate, 5.2% of total aerosol mass) and March 2009 (0.2 µg m^{-3}, 10% of total nitrate, 3.6% of total aerosol mass) in Cabauw."

3) Correlation of PMF time series with tracers: The correlations of the PMF factors with the tracer time series seems very low for HOA, BBOA and HULIS, and the correlation coefficients (R2) are all below 0.5 for all the tracers used in this study. For example, the HULIS factor, which is presumably secondary in nature, exhibits the highest correlation with BC rather than one of the primary combustion factors (i.e. HOA and BBOA). This lack of correlation indicates that PMF is not identifying OA components that can be unambiguously associated with specific sources. The lack of correlation should be examined in more detail as it calls into question the validity of the PMF results.

Response:

As mentioned in the manuscript the HULIS factor was found as high oxygenized background aerosol in Cabauw, which is characterized by the low temporal variation and therefore reasonably low correlation coefficients with most of the tracers. On the other hand the reviewer noticed correctly that the correlation results given as Pearson-R^2 in case of the POA factors with BC data don't seem to be as high as seen in previous studies showing PMF/ME-2 data. This has several reasons:

The black carbon data used in this study derived from a MAAP, which measures simultaneously the radiation penetrating through and scattered back from a particle-loaded fiber filter. Andreae and Gelencser (2006) pointed out that non-BC light-absorbing carbonaceous matter like brown carbon can interfere with BC measurements using this instrument. They also stated that filters loaded with macroscopic amounts of humic like substances (HULIS) samples look very dark brown or nearly black. This would imply a possible interference of HULIS with BC. Consequently this may explain why the HULIS factor has a relatively high correlation with BC. In summary, black carbon measured by the MAAP can have three main sources: traffic and biomass burning, represented by the HOA and BBOA factors, respectively, and the interference with HULIS. All three sources are independent from each other, leading to the observed correlation results. The use of instruments like an aethalometer or a particle soot absorption photometer as used in other studies would reduce these interferences (Andreae and Gelencser, 2006; Petzold et al., 2013), but were not available during the campaign.

As expected, the gaseous tracers NO_x and CO exhibited the highest R^2 values with HOA, and higher correlations with BBOA than with both SOA factors.

In the revised manuscript the respective part was changed to:

"Note that the correlation (Pearson-R^2) of the POA factors with eBC data is relatively low ($R^2 = 0.38$ and 0.39 with HOA and BBOA, respectively), while in turn HULIS shows a higher correlation with eBC, $R^2 = 0.47$. This can be understood in the light of a study by Andreae and Gelencser (2006) who pointed out that the fiber filters, as used by the MAAP, loaded with macroscopic amounts of humic like substances samples look very dark brown or nearly black. This implies an interference of HULIS with BC measurements and in consequence explains why the HULIS factor, in contrast to the POA factors, has a relatively high correlation coefficient with BC data."

Specific Comments:

4) P35121, L20: What is the difference between intensively and extensively managed grassland?

Response:

The phrase "intensively managed" agricultural land refers to the extended use of fertilization, irrigation or drainage techniques. In case of grasslands this definition is also applied to fields

where, with respect to the agricultural area, a relatively high amount of farm animals are hold in order to optimize the respective production. Consequently, "extensively managed" areas refer to the opposite: keeping the pasture area in their natural conditions and using them with less numbers of animals. Since these phrases are common agricultural definitions, the authors decided to not explain them in detail.

5) P35122, L2: Remote sensing is a very general term. The manuscript should specify exactly which remote sensing measurements are made at the tower site.

Response:

In the revised manuscript, we have modified this sentence as follows:

"In addition, other meteorological data like precipitation, radiation and remote sensing, including lidar, radar and radiometer techniques, are acquired at the tower and submitted to the CESAR data base."

6) Page 35123, L27: In contrast with what is implied in the text, I believe that the Middlebrook et al. (2012) CE correction algorithm also accounts for high ammonium nitrate.

Response:

We agree with the reviewer that the ammonium nitrate mass fraction (ANMF) is used as a parameter within the algorithm published by Middlebrook et al. (2012). Specifically ANMF is used when the ratio of measured to predicted NH_4 is higher than 0.75. Otherwise a different equation using just this ratio is applied to determine the CE in the Middlebrook algorithm. For the Cabauw ACSM data set, where $OrgNO_3$ could only be determined from the combined ACSM and MARGA datasets, the ratio of measured to predicted NH_4 varies around 0.75, which lead to unreasonable discontinuities of CE values when applying the Middlebrook algorithm. The authors agree that the current explanation is misleading. In the revised manuscript the respective part was changed to:

"In contrast to the commonly used constant value of 0.5 this CE correction accounts for the high ammonium nitrate mass fraction (ANMF) found at this site and is thus more suitable for the data presented here. Another algorithm for composition dependent CE determination (Middlebrook et al., 2012) was also tested for its validity. It uses a threshold ratio of measured to predicted NH_4 to switch between two different equations to determine the CE. The threshold value of 0.75 is close to the observed ratio of measured over predicted NH4 of this

data set, resulting in large discontinuities of CE values and in consequence, discontinuous changes in aerosol mass concentrations. In other words, the Middlebrook algorithm is not suitable for data sets showing at the same time low ratios of measured to predicted NH_4 and high AMNF's."

7) Page 35124, L5 – L8: Why was the detection limit not calculated during the measurement period or afterwards using data from the measurement period? Data from periods when the instrument was sampling behind a particle filter could be used for this analysis.

Response:

Unfortunately, there were no ACSM measurements done during the presented campaign where a separate particle filter was introduced in the sample line, in addition to the filter included in the gas-phase background filter cycle as described by Ng et al. (2011). Furthermore, the ACSM software version used in this study could not show data acquired during the filter cycle measurements (e.g. closed mass spectra/time series), which would be needed for the determination of the detection limits. The software could only show the differential mass spectra/time series.

8) Page 35125, L23 – 29: The correction for the wall loses in the 60 m sampling line is a critical point for the manuscript. However, the description of how the losses were calculated or estimated is not sufficient. Only a reference to a personal communication is provided. Given that this correction can impact the aerosol measurements substantially – by 33% as indicated by the authors – the manuscript must contain a detailed explanation of how the aerosol losses in the sampling line are determined.

Response:

We agree with the reviewer that the correction and explanation for these losses is not sufficient enough.

In the revised manuscript we used now the particle density deriving from the chemical composition not as the campaign average but time resolved for each data point. In addition we introduced SMPS data which is now size dependently corrected as published by Henzing (2011). The description of the sampling losses of the 60 m inlet for eBC given in the manuscript derived from a series of measurements at the Cabauw tower performed in a previous campaign. Unfortunately these results are not published yet. To clarify how these

1 losses were determined in the revised manuscript and to account for a comment from referee
2 #2, the loss description was changed as follows:

3 "SMPS data was corrected size dependently for (diffusional) losses in the inlet system and
4 SMPS system itself according to Henzing (2011) who compared theoretical findings with
5 measured losses that are obtained by measuring simultaneously before and after the various
6 parts of the inlet system at the CESAR tower. In addition, particles of different compositions
7 were measured in 2013 simultaneously at the pipe entrance at 60 m height and in the
8 basement (J. S. Henzing, personal communication). For more than 8000 simultaneous
9 observations, the results showed that aerosol measurements through this 60 m sampling line
10 underestimate PM_{10}-eBC by approximately 33% with an uncertainty of 7%. Therefore, eBC
11 obtained from the MAAP are divided by a factor of 0.66 to account for these losses. For the
12 inorganic species penetrations through this inlet line were reported to be 62-73% for nitrate,
13 55-64% for sulfate, and 54-56% for ammonium. However these results were not used for
14 corrections in this work"

15 Please note that these losses influenced only data acquired by the MAAP and SMPS. Since
16 the contribution of eBC is rather low (average: 5%) a potential overall error for total aerosol
17 masses is low and would not significantly alter one quintessence of the paper, namely total
18 mass concentrations above the air quality limits.

19 As a consequence of the newly evaluated SMPS data the correlation values between
20 ACSM+MAAP data with SMPS data changed as seen in Fig. S3 and S4 in the revised
21 supplement. Nevertheless, the overall qualitative and quantitative agreement is still given
22 except that the ACSM+MAAP data is now overestimating the total PM_1 mass by 16%,
23 excluding the eBC data the ACSM overestimates total mass by 12%. As seen in Fig. S3 the
24 difference between both systems is significantly higher during the pollution events 16 to 27
25 January 2013 and 5 to 8 May 2013. Since the quantitative agreement with the MARGA is
26 much higher at these times the discrepancy to the SMPS is likely due to the fact that the losses
27 within the 60 m inlet could not be corrected for individual species as mentioned above.
28 Therefore the following paragraph was added at the end of the cross validation chapter in the
29 revised manuscript:

30 "Major discrepancies to the SMPS especially during some of the pollution events like 16 to 27
31 January 2013 and 5 to 8 May 2013 (see below) can be explained by the correction of losses
32 through the 60 m inlet line which was done size dependently and did not account for losses of
33 individual species as mentioned in chapter 2.3. As the quantitative agreements of individual

inorganic species as well as of total inorganics between the ACSM and the MARGA during these periods are much higher, the mass loadings determined from these instruments are more reliable than the SMPS data."

9) Page 35126, L13 – L16: During the measurements with the MARGA system, a polyethylene tube was used as the sampling line. Could the use of a non-conducting material for the line lead to substantial electrostatic deposition of the particles to the walls of the tube?

Response:

Indeed, a polyethylene (PE) tube can potentially enhance wall losses of particles comparing to stainless steel tubes. The MARGA-inlet system at the Cabauw tower as used for this study was previously described by Schaap et al. (2011). It actually did not only consist of PE tubes but of a series of components reducing particle losses. They investigated wall losses on a similar system and found only minor concentration losses for several compounds of 2% and less. A more detailed description was added in the revised manuscript:

"The sample air was transferred into the instrument within a polyethylene („Polyflo") tube with an inner diameter of 0.5" (= 1.27 cm) and a sample flow of 16.7 L min^{-1}, which is either directed through a PM_1 or a $PM_{2.5}$ size selective head. A detailed description of the MARGA inlet system at the Cabauw tower was previously described by Schaap et al. (2011). There, wall losses were investigated and found to be less than 2% for several gaseous and particulate compounds."

10) Page 35129, L7: Why is SO2 included in the MARGA PM2.5 mass? In the atmosphere, this compound is found in the gas phase.

Response:

We thank the reviewer for pointing out this mistake. Not only SO_2 but all gas phase compounds HNO_2, HNO_3 and HCl were mistakenly added to total aerosol masses. Nevertheless, the conclusions deriving from these calculations did not change. In the revised manuscript, the resulting time series "MARGA $PM_{2.5}$ + ACSM-Org + eBC" in Fig. 1 was exchanged and the sentence was changed to:

Nevertheless, the average total mass derived from the combination of the MARGA $PM_{2.5}$ data (including all water soluble inorganic components NO_3, NH_4, SO_4, Cl, Na, K, Mg, and Ca),

MAAP eBC and ACSM PM$_1$ organics resulted in 12.5 µg m^{-3} during this time, clearly exceeding the WHO PM$_{2.5}$ annual mean limit (10 µg m^{-3}) by 25%.

The reader may now notice that the concentration values of MARGA PM$_{2.5}$ + MAAP eBC + ACSM PM$_1$ Organics (light blue time series in Fig. 1) decreased reasonably because the mentioned gas phase data was excluded. On the other hand, more data points are now seen. This is due to the fact that data coverage of the MARGA PM$_{2.5}$ data is better than for the MARGA gas phase data.

11) Page 35129, L9: It appears that there is an error on this line and the concentration for the annual mean limit should be 10 µg m-3 rather than 25 µg m-3 as is currently written.

Response:

The value is indeed wrong. In the revised manuscript it was changed from 25 to 10 µg m^{-3}.

12) Page 35130, L6 – L9: This sentence is confusing. First, it is written that the pollution events are highlighted in green, but (to my eye) the highlighting is grey. Second, there are four periods that are highlighted in Figure 2, but only three periods are listed in the text. The dates for the missing period should be given as well.

Response:

The authors agree that the color used for highlighting cannot be designated unambiguously. To keep it simple, we renamed it as just "shaded". Figure 2 is the only figure where two different colors were used for shaded areas. Therefore, the shading for the period where ACSM and AMS data were overlapping was removed in this figure in the revised manuscript. Consequently, the captions from Figures 1 and 4 as well as from several figures in the revised supplement file were changed by calling the "green shaded" areas as just "shaded" instead of giving them a certain color. Furthermore and accounting for a comment from referee #2, the sentences on page 35130, lines 6-8, were changed in the revised manuscript to:

"The most significant pollution events (17 to 21 August 2012, 21 to 25 October 2012, 16 to 27 January 2013, and 5 to 8 May 2013) are highlighted with shaded backgrounds in Fig. 2. During the last three periods, northerly and north-easterly winds dominated."

1 *13) Page 35132, L21: From Figure S10, it appears that the intercept is negative rather than*

2 *positive (i.e. –0.80 versus 0.80). The sign is important as there is likely a small amount of dust*

3 *and perhaps sea salt which is measured by the SMPS and not the ACSM that is responsible*

4 *for the non-zero intercept value.*

5 **Response:**

6 Indeed, the value in the main text has a wrong algebraic sign. As described in the respond to

7 comment #8 we introduced newly evaluated SMPS data in the revised manuscript by which

8 the values changed. The intercept is now -1.05 ± 0.06 but still negative. We thank the

9 reviewer for pointing out that a negative offset infers influence from sea salt or dust aerosols,

10 which can be detected well by the SMPS and MARGA but not by the ACSM with a sufficient

11 sensitivity. Therefore we added a small discussion to this section in the revised manuscript:

12 "The negative offset can be explained by minor influences of sea salt and dust particles,

13 which can be detected well by the SMPS and MARGA but not by the ACSM with a sufficient

14 sensitivity. But the low value of the intercept shows already that the uncertainty introduced by

15 these aerosol components is rather low in general. This can also be explained by the low

16 concentrations of Mg, Na, K and Ca as measured by the MARGA (see below) and the

17 assumption that the majority of dust particles is most likely found in particles with diameters

18 larger than 1 or even 2.5 µm (Finlayson-Pitts and Pitts (2000) and references therein)."

19

20 *14) Page 35133, L18 – 20: Why couldn't the fragmentation table be adjusted? Adjustment of*

21 *the fragmentation table is fairly standard during analysis of AMS data. If it is believed that*

22 *the fragmentation table may be responsible for the underestimation of SO4, then the authors*

23 *should explore making possible adjustments of the values in the table.*

24 **Response:**

25 We agree with the reviewer that for AMS data a proper adjustment of the fragmentation table

26 is recommended especially for unit mass resolution data, which is produced by the ACSM,

27 too. Unfortunately, the ACSM software used in this study did not allow plotting time series

28 for specific fragments apportioned by the frag table, e.g. "frag_sulphate[48]" like it is

29 commonly done in the AMS analysis toolkits SQUIRREL or PIKA. Without exploring the

30 resulting changes of these time series by adjusting the fragmentation table it did not make

31 sense to vary the fragmentation table entries. To clarify that the revised manuscript was

32 adjusted to:

Additionally, the ACSM fragmentation table could not be adjusted for interferences of ions from different aerosol species on the same m/z properly because ACSM software used in this study did not allow plotting time series for specific fragments apportioned by the fragmentation table. Thus, the standard table had to be used.

15) Figure 1: Using zeros to fill in the missing values for black carbon for periods when measurements are not available is not a standard approach (to my knowledge). It would be preferable if the existing BC data was simply just averaged. It is already very clear from the manuscript that data is missing in certain periods, so a reader can take this into account when evaluating the data. Alternatively, the pie chart could be modified to include only the ACSM data for when the MAAP was functional.

Response:

Unfortunately, the figure caption here was misleading: Missing values of eBC data was only filled with zeros for the calculation of the time series of the daily means. For the calculation of the pie chart values eBC data was just averaged as suggested by the reviewer. In the revised manuscript, the caption for this figure was changed to:

Figure 1: Time series of the daily mean. The black line represents the sum of eBC and all ACSM species, the blue line the sum of eBC, ACSM organics and all MARGA-PM$_{2.5}$ species. The pie chart shows the fractional abundances of individual eBC and ACSM species averaged over the whole campaign. For the determination of the daily means missing eBC data was filled with zero values, thus deriving lower concentration limits.

We thank the reviewer to bring up this ambiguity.

16) Figure 4: I assume the authors mean to say "grey shaded areas" rather than "green shaded areas".

Response:

Please see the response for author's comment #12 above.

17) Page 2, L3: I think this equation should be MFNO3 ≥ 0.78.

Response:

Indeed. In the revised manuscript, this equation was corrected.

18) Figure S1: The figure legend text is very small and hard to read. The font size should be increased to improve readability.

Response:

In the revised supplement material, this picture is enlarged to enhance the readability of the legend. The picture of the tower itself was removed.

19) Figure S2: I assume the authors mean to say "gray shaded areas" rather than "green shaded areas".

Response:

Please see the response for author's comment #12 above.

20) Figure S6: Similar to the previous comments, the shaded areas look grey to me rather than green. Perhaps there is a difference in how the colors are displayed by the author's monitor and my monitor? There are subsequent figures were this comment applies as well, but I will not repeat it to avoid being overly repetitive.

Response:

Please see the response for author's comment #12 above.

21) Figure S13: It would improve the presentation of the results if all the bar graphs were plotted with the same format. The size of the bars is not consistent.

Response:

In the revised manuscript, the format of all four graphs is similar.

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