Reply to referee comment 1 on Kau et al. “Thermal-optical analysis of snow samples – challenges and perspectives introduced via the occurrence of mineral dust”

We thank Jonas Svensson for taking time to review our manuscript. The text of the referee comment is in **bold**, while our reply is in regular type. Passages quoted literally from the revised manuscript are in *italic* type.

Referee report on Kau et al. ‘Thermal-optical analysis of snow samples – challenges and perspectives introduced via the occurrence of mineral dust.’

General comments

This manuscript presents a new approach in dealing with the interference of mineral dust (hematite in particular) that might occur during thermal-optical analysis of filter substrates. The paper would be highly welcomed in the literature, due to the reoccurring phenomenon of mineral dust disturbing OCEC-analyzes. Since it is a novel approach, the text is well suited for AMT. In its current form, however, the paper needs some substantial work. Currently, the full potential of the manuscript has not been explored, but rather, corners appear to have been cut, resulting in several sections that either lack or provide minimal information. As an example, the approach is tested for high alpine PM10 filter samples, but only two samples are available. More data would be needed to provide a more robust basis for the authors to discuss the results on that topic. Is there data available data from another site (other than Sonnblick?). Similarly, the snow samples from Sonnblick could be compared with snow samples from another location. This would mean more work, but in the end, it would only and built-up the discussion/conclusions, and ultimately, strengthen the manuscript. With that said, I would reiterate that major revisions are necessary for this paper that could have great potential breakthrough in this area of science. Please see more comments below.

We thank the referee for mentioning that the paper would be highly welcomed in literature and the valuable input to improve our manuscript. We addressed all of the requests mentioned as major or minor comments below and added new data to the manuscript.

To address the points mentioned above, within the general comments, we have two remarks:
1) A higher number of PM_{10} filters is included in the revised version of the manuscript and another reference material was tested to set a more robust basis for our approach.
2) The only point we could not cope with is to add results of snow and PM_{10} samples collected at different sites. Still, we are convinced that our data set of snow and PM_{10} samples is comprehensive enough to justify the publication in its revised form. All samples containing mineral dust originate from Sonnblick, but they represent quite a variability as, regarding snow samples, they were collected during different visits at the site carried out during the years 2018 to 2020. Regarding PM_{10} samples, the time period of 2017 until 2022 is covered. Thus, we are confident that we can present sound results.

Testing of the approach for different sites and thus environmental conditions is important and necessary but goes beyond the scope of a manuscript presenting a novel method utilizing TOA for the determination of iron loadings of mineral dust samples.

Major comments

Section 3. The different instrumentation used in the study seem applicable and impressive. Still, it is difficult for a reader to see their place and role in the different steps of the approach. It could easily be misinterpreted that the instruments were just randomly chosen when the authors clearly thought about how to best utilize their instruments for this method. One suggestion is to provide an introductory section before each instrumentation is expanded upon. In this introductory text, it should be explained why the instrument is used. In a way, it should end up as a flow-chart explaining the different steps of the analysis, providing future readers with a roadmap of the approach enabling them to easier follow along.

We agree that this contributes to a better understanding of our work. Consequently, we adapted all sub-sections of Section 3 (line 106 - 110, 117 - 119, 128, 138 - 139 in the revised manuscript). Now the reason for using these methods is stated before further explanations.

The aims of the paper should be scrutinized. Currently, there is a mismatch between the aims (denoted as interests in the introduction) stated in the introduction and once they are re-stated in the conclusions. The aims should be clearly reviewed and revised in the introduction. On the whole, the conclusions that are discussed particularly in lines 298-308 are not supported by the data currently presented in the paper (e.g. recommending a rerun of analyses; that the approach was successfully applied to PM10 filters also, see additional comment on this below).

Thank you for this remark! To solve this mismatch, we harmonized the aims stated in the introduction (lines 68 - 72) and the conclusions (lines 342 - 362) in the revised manuscript).

Now they read:

Introduction:

‘*In this work we investigate the influence of MD loads on TOA of snow samples collected in a high alpine environment. The main interest of our work is to investigate the temperature dependence of the light attenuation caused by Fe containing compounds in MD. This led us to a new approach to approximate Fe concentrations via TOA. Therefore, we evaluate the transmittance signal during the calibration phase, i.e. when carbonaceous compounds are no longer present. The method is tested extensively for snow samples and evaluated briefly for particulate matter samples.*’

Conclusion:

‘*Investigating the influence of MD, i.e. its light absorbing constituent hematite, on TOA, we presented an easily applicable approach to determine Fe loadings on filters containing MD based on temperature changes of the transmittance signal recorded during the calibration phase of TOA. The method is based on the evaluation of quartz fiber filters loaded with snow samples containing different amounts of MD*

Considering the whole range of Fe loadings investigated, the relationship between the Fe loading and $ATN_{700\text{-}400}$ *was shown to be non-linear. By setting two straightforward criteria (*$ATN_{700\text{-}400}$ *between 3.5 and 29 and transmittance signal at 400°C above 600 a.u.) the determination of Fe loadings between 10 to 100 µgFe* cm^{-2} *becomes possible based on a linear approach. This approach opens the possibility to obtain another compound of LASI with the same method (TOA) as used for the determination of EC. This can be advantageous if the* *available amount of sample is limited and a proxy for MD is needed. Still a reliable conversion factor of Fe to MD has to be deduced by independent analysis or taken from literature and is not presented here.*

The method developed for the analyses of filters loaded with snow samples was successfully applied to the analyses of particulate matter filters originating from the same high alpine environment. We cannot give information about the general applicability to PM10 samples loaded with different types of MD, yet. We hypothesize that it should be possible to use the approach as long as MD and its constituent Fe_2O_3 remain the dominant source of Fe loadings. Still, an independent calibration might be needed to account for differences in MD composition. Fe loadings in particulate matter samples collected within a railway tunnel and thus having a strong influence of abrasion products from tracks and wheels and not from MD cannot be analyzed with the method presented here. This illustrates the limitation of the method.

Filters loaded heavily with MD (Fe loadings above 10 µgFe cm^{-2}) will experience a severe bias of the OC/EC split point. EC will be underestimated or cannot be determined at all, while OC will be overestimated.

We recommend to rerun the analyses, as soon as the evaluation of the transmittance signal in the calibration phase indicates an influence of MD. This second run of analyses will allow to set the split point more precisely.'

The recommendation to rerun TOA is based on the data of repeated analyses presented in Section 4.1. and is actually already mentioned there (lines 178 - 179, 188 - 189 in the original manuscript). The topic regarding PM_{10} filters is discussed below.

Section 4.2 and Fig.2. For the samples contaminated with the reference hematite, was it one batch of filter samples? In other words, was this step ever repeated, with another independent set of hematite contaminated filters? In order to test whether the same pattern would be repeated in the filter samples this is a suggestion of work to be done. Similarly, what about conducting experiments with reference filters containing Fe originating from a different source than hematite? This could further confirm the claims made that this method works well for hematite by not other types (which currently is not strongly supported by much data; only the tunnel samples).

The data presented in Figure 2 refers to one batch of filter samples, i.e. a series of 9 filters loaded with different amounts of the same reference sample, i.e. a suspension of Fe_2O_3 in water. At a different time two additional filters were loaded with a suspension prepared independently. These two data points (8.8 and 19 µgFe cm^{-2}) fit perfectly to the existing data and are included in Figure 2 and Figure 5a in the revised manuscript. Furthermore, we mention that two suspensions were prepared independently. Thus, we can confirm that the same pattern is obtained when repeating this part of analysis.

Still, we agree with the comment that experiments with reference filters containing Fe from different sources would be interesting, though not absolutely necessary at this stage.

First, we want to explain why we chose hematite. Formenti et al. (2014) present the mineralogical composition of mineral dust in Western Africa. Fe oxides (mainly hematite and goethite) account only for a low percentage of MD mass (2 - 5 %), but they represent the biggest group of Fe compounds in MD (roughly 58 %). At elevated temperatures (250 - 600°C), goethite changes to hematite (Liu et al., 2013). This temperature is exceeded during TOA already in the inert phase. Therefore, hematite will be the main Fe compound on our sample filters during the oxygen phase and the calibration phase and it is well suited as a reference material for our method. As the occurrence of hematite in the snow samples was confirmed in the PXRD analyses, we focused on this compound rather than addressing different isolated compounds. Actually, we checked for FeO and this compound did not show changes in transmittance when heating or cooling the samples. Still, FeO was selected rather arbitrarily, and we did not include a respective paragraph in the text as we do not have any indication that FeO is relevant for snow or PM samples containing MD. The information why we use hematite as a reference material is added in the revised manuscript in the Introduction and when the reference samples are introduced (Introduction: line 57 - 63, Section 2.3: lines 94 - 97).

Testing single minerals will give a ‘yes/no’ decision whether an effect is possible and are definitely valuable. Still the conditions found for actual samples will be different, as other minerals contained in MD and particle sizes will influence the transmittance as well due to multiple scattering and different loading effects. To go in that direction, an independent set of filters loaded with standard reference material SRM 2709 (San Joaquin Soil, NIST) was prepared and analyzed using TOA, ICP-OES and SEM. SRM 2709 is an agricultural soil, intended to be used as a reference for soil or materials of similar matrix. Unfortunately, we do not have a specification of iron contained in this SRM. Our evaluations show that the same patterns are visible for different sample groups, e.g. high alpine samples, SRM 2709 or hematite, but different fits are necessary to relate Fe and $ATN_{700-400}$. The reference substances serve the mere purpose to show that their overall trend of $ATN_{700-400}$ vs. Fe loading is similar to the trend observed for the actual snow sample filters.

The data about SRM 2709 was added in line 101 - 103, 242 - 244, 256 – 262, Figure 2 and Figure 5a in the revised manuscript.

The group of tunnel samples, where Fe is present due to abrasion of tracks and wheels and therefore is present in other forms than hematite as well (mainly magnetite and metallic Fe) show that the applicability of our approach is limited. This set of samples is provided as some kind of warning, that TOA cannot be applied to determine Fe in all sample matrices. We tried to be more specific about this point in the Conclusions.

Section 4.3. It is understood that there are only two PM_{10} samples from Sonnblick. Yet, two samples are not enough to support the claims the authors want to make in the text (concerning the applicability of the fit for PM_{10} samples). The authors need to find a way to expand with more samples for the PM_{10} or majorly down-play the importance of only two samples in the claims they are making.

We agree that the low number of PM_{10} samples from Sonnblick was unfortunate. As stated in line 245, loadings above 10 µgFe cm^{-2} were hardly achieved during the long-term data set covering more than 4 years.

Due to the frequent and strong occurrence of MD this spring (the data became available just after the manuscript was submitted) the number of PM_{10} filters could now be increased from 2 to 6. On the one hand this is an increase by 200 %, on the other hand one could still argue that a data set of six samples is not huge. The data set is too small to calculate a fit for PM_{10} samples themselves but shows that the data of PM_{10} samples collected at the same environment fits nicely into the fit obtained for snow samples. Hence, we only present the data compared to the snow samples and calculate differences between the Fe concentration using the ICP-OES data and our fit for the snow samples and do not calculate another fit for the PM_{10} samples.

The PM_{10} dataset was enlarged, and the new data added to Figure 4 and Figure 5a. To account for the additional samples, the text was adapted in line 292 - 297 in the revised manuscript.

Minor comments

Title. As it currently reads the title of the paper suggests that *challenges* and *perspectives* are to be discussed. The use of such wording would be more appropriate if this was a review article. I would encourage the authors to critically think through the title and modify it to reflect the paper better.

We agree and changed the title to '*Thermal-optical analysis of quartz fiber filters loaded with snow samples – determination of iron based on interferences caused by mineral dust*'.

Line 17-18. I would advise the authors to transform this sentence into a more informative sentence. In other words, do not mention what you are discussing in the manuscript without adding something about it, but rather, be specific and give highlight details of the results and conclusions that can be made concerning this method. It would not necessarily need to make the text much longer, but all the more informative for future readers.

Thank you for this input, we absolutely agree. The last sentence was rewritten (line 18-23 in the revised manuscript).

Line 21. In-text references, it is neither alphabetical nor chronologically, please pick something and be consistent throughout the manuscript.

We listed in-text references alphabetically throughout the manuscript (e.g. line 26 in the revised manuscript).

Line 30. This is the wrong reference to Schwarz et al. and his work on rBC in snow. The 2006 paper deals with atmospheric BC. I would recommend the authors to look for his 2012 paper, that deals with rBC in snow samples.

Thank you for pointing this out. We changed the reference accordingly (line 35 and line 466 - 469 in the revised manuscript).

Lines 46-48. It is difficult to understand what the authors mean with this sentence. How is it not the main point? (or do you simply mean that it is not the point in the paper to differentiate between WinsOC and OC?). How can the concept be applied to the analysis of both types?

Yes, we mean that it is not the point in the manuscript to differentiate between WinsOC and OC. Depending on the kind of sample, TOA yields either WinsOC (analysis of insoluble particles in e.g. snow filtrated onto quartz filters) filters or OC (analysis of ambient particulate matter collected on quartz filters). However, there is no analytical difference between WinsOC and OC, therefore the evaluation we describe can be applied to both sample types. For clarification, we avoided the unclear sentence and adjusted the text in line 51 - 53 in the revised manuscript.

Line 53. Adjusted the method accordingly how? Please provide the reader with what was done previously (even if only brief).

Wang et al. (2012) shifted the reference value for the OC/EC split to the 250°C temperature step. We added this information in line 66 in the revised manuscript.

Lines 56-57. Please be more specific in this sentence. This is very broad and not very descriptive of what is addressed in the paper.

We revised the sentence to make it more informative (line 68 in the revised manuscript).

Line 65. It is stated that snow samples were collected near the GAW station. Please be more precise. Overall, any more info that could be provided on the snow samples could potentially be useful for future work. One suggestion would be an informative table (which could easily be put to the supplement) containing relevant info on the snow, e.g. fresh/old, density, sampling depth, etc.)

The samples were collected on the upper platform of the observatory. Regarding sampling depth, the uppermost 5 centimeters of snow were collected. We added this information in line 77 in the revised manuscript. As these samples were collected for method development, no further information, e.g. density, time of last snowfall, was recorded.

Line 76. Please define PM10.

We added the definition of PM_{10} in line 88 in the revised manuscript.

Line 96. What is method 3052? Please inform (even in brief).

We added a brief description of METHOD 3052 in line 120 - 122 in the revised manuscript.

Lines 116-131. The text along these lines can easily by moved to an introductory (or background section), as these results are not specific to this study, but rather has been known from previous work.

Only the text in lines 117 - 120 is general knowledge. The following sentences are explanations of Figure 1 and definitely needed in section 4.1. Still, we think it is important to keep the first three sentences so that the following arguments and figures are well understood, especially for people not being absolutely familiar with TOA.

Line 117. TOA of snow samples. Obviously, the actual snow sample is not analyzed in the TOA, but rather the filter with collected particulates from the snow. Please adjust this throughout the manuscript to be as concise as possible.

We agree that we were not precise enough. We checked the whole manuscript and adjusted the text accordingly.

Line 118-119. Does it not depend on what type of minerals are present on the filter? Some minerals will not change color, nor darken the filter substrate. If there is hematite present on the filter it is evident that the transmittance is affected, but not all MD. It is stated further down in the manuscript (line 226) that hematite was a minor contributor to the MD. Please be specific in the text.

We agree that MD can vary in its composition. MD deposited on Mount Sonnblick often derives from long range transport of dust from desert regions (as given in lines 66 – 67 in the original manuscript). But even for desert regions several differences exist.

Fe oxides usually come up to a few percent of MD. This was already explained further up (major comments, Hematite as a reference compound). The composition of MD and the special situation of hematite is discussed in more detail in the revised version of the manuscript as indicated above.

Furthermore, we also revised the sentences addressed in this comment. If MD contains reddish Fe compounds, as hematite, the filter will remain colored after TOA. We added this information in line 146 in the revised manuscript to be more precise.

Still, this comment made us to recheck the wording in the whole manuscript. Now we only refer to hematite when the actual reference sample is addressed. We changed the description in the snow and PM_{10} samples from hematite to an expression like ‘Fe containing compounds’. Fe oxides described to be the important light absorbing compound in MD (Alfaro et al., 2014); however, the analysis of Fe via digestion and ICP-OES or ICP-MS leads to the Fe concentration of all accessible Fe compounds including Fe in other forms, if present. Hence, the description similar to “Fe containing compounds” is more precise, especially if samples from different origins with varying composition are compared. This also accounts better for the fact that differences between our samples from Sonnblick and the SRM filters – loaded with different kinds of dust – are visible.

Line 132. How was the Fe content determined? And also, what do these numbers translate into for MD concentration for the snow samples? Please include this as a guide for future readers who might have a MD concentration and would want to compare that to the range of samples where the approach here is applicable to.

Fe was determined using ICP-OES or ICP-MS for all samples (line 94-101).

We do not want to give MD concentrations, as it is difficult to provide a robust conversion factor from Fe to MD (see the section about the composition of MD and the references to Kandler et al., 2007 and Formenti et al., 2014). Still, we can provide Fe concentrations of the two snow samples mentioned in line 132. They were as follows: the sample containing MD had an Fe concentration of 1.1 mgFe L^{-1} (filtrated volume 54 mL), while for the sample without visible MD contamination the Fe concentration was 51 µgFe L^{-1} (filtrated volume 99 mL). This is well within the concentration range given in literature, e.g. by Kaspari et al. (2014), who report Fe concentrations in snow and ice ranging between <10 µgFe L^{-1} and 100 mgFe L^{-1}.

We added the Fe concentration in the liquid snow sample and the filtrated volume of liquid snow in line 160 - 162 in the revised manuscript.

Line 150. What is meant with high background of the filter matrix? If it refers to hematite, how did it get into the background of the filter?

The high background is due to the quartz fiber filters which need to be used for TOA.

Lines 180-183. One could argue that this information is not novel to the study here, but has rather been known from previous work, and is actually part of the motivation for this work. Thus, it should be moved to the introduction/or alternatively in a background section after the introduction.

We agree that the description of the conditions during the calibration phase is quite general, but we are not aware of literature where the transmittance signal during the calibration phase is utilized. Thus, it is novel. As we feel that this passage adds to understanding why this approach is suitable (no interference from carbonaceous compounds, oxidizing conditions as prevailing when the OC/EC split is usually set) we decided to leave it in the Results section.

Line 199. Please remove the equation from the text, it should be inserted on a separate line (as well as any other question from the text).

Thank you for making us aware of this. We removed the equations in lines 199 and 241 from the text and added cross references in the text.

Line 210-211. Unloaded filters at room temp I_0? Previously, it is stated that I_0 is for 400°C. Please clarify.

We defined the transmittance at 400°C as I_0' (line 203 – 206) (note the apostrophe). I_0 is the laser transmittance at the end of TOA reflecting an unloaded filter (line 199 – 201), we used I_0 for the transmittance value of an unloaded quartz fiber filter at room temperature.

Lines 217-218. It is mentioned that such high hematite loadings are rarely found. What type of concentration does it equal in the snow? Please provide numbers on this, is it above ppm?

The wording was misleading. Such high loading can be found (you just need to filtrate larger amounts onto a small filter). The reason for such high loadings being rarely found is that they are difficult to handle. The filter will be overloaded, which leads to slow filtration and eventually to loss of the deposited material during handling. To clarify this, we changed to sentence which now reads '*Fe loadings above 150 µgFe cm^{-2} are avoided, as these filters are prone to sample loss*'. (line 254 - 255 in the revised manuscript).

To provide numbers, we added a short calculation: The sample with visible occurrence of MD presented in section 4.1 contained 1.1 mgFe L^{-1}. To reach the upper part of the curve, i.e. an Fe loading above 150 µgFe cm^{-2}, roughly 270 mL need to be filtrated. Visible occurrence means that MD was visible within the snow and after melting.

Line 225. SEM analyses confirmed such differences. Please be more specific by including some results.

We included more information about SEM analyses in the revised text (lines 256 - 262, 269 - 271) and in the Supplement. We added elemental maps of Fe for a filter loaded with SRM 2709 and particles of a melted snow sample. The elemental maps show that Fe-rich particulate matter on SRM and snow filters are generally smaller than particles of Fe_2O_3 reference filters. Additionally, we added spectra of a filter loaded with Fe_2O_3, SRM 2709 and a snow filter. These spectra show signals only for Fe, O, the quartz fiber filter and the Au coating for Fe_2O_3, and signals for Fe, Ca, K, Si, Al, Mg, Na and O as well as the Au coating for the SRM and snow filter. The elemental maps and spectra are part of Figure S2, while the description of Figure S2 and the text belonging to Figure S2 were adjusted (line 40 - 51 in the revised Supplement).

Lines 225-226. Hematite was larger for the reference than the snow originating particles. Any idea of how much larger? Please include this information.

The sizes were determined only for a few particles. No size distribution was determined. A qualitative impression of particle sizes can be obtained from SEM images; however, we cannot provide reliable numbers for particle sizes.

We adapted the text regarding SEM analyses in line 256 - 262, 269 - 271 in the revised manuscript.

Fig. 4. The figure would benefit if both axes would be modified to more accurately reflect the range of the sampling points. In other words, the sampling points would be more centered in the figure.

We edited Figure 4 accordingly.

Lines 278-279. How do we know of the different originating Fe, as in where is the evidence for this claim? Visual inspection also?

While Fe in the snow samples originates from the mixture of minerals contained in MD, Fe in the tunnel samples originates mainly from abrasion of tracks, wheels and brakes of the trains. This was already reported at a previous comment. Eom et al. (2013) found that particulate matter samples from railway tunnels contained Fe in the form of magnetite, hematite, and metallic Fe (roughly 40%, 30% and 25%, respectively). Besides the overall iron loading of those filters being high, the relative contribution of iron to overall mass in tunnel samples is much higher (up to 73%, line 265) than for MD samples which contain 2 – 5% iron (Formenti et al., 2014). This is another reason why it cannot be MD on these samples. Additionally, visual inspection of the tunnel filters after TOA confirmed differences in the remaining compounds compared to the filters loaded with snow samples, as shown in Figure S4 in the Supplement.

The text was revised to highlight these facts in more detail (lines 310 - 315 in the revised manuscript).

Line 285. Please add that it is during the calibration stage during TOA.

We added this information in line 344 in the revised manuscript.

Line 290. Have the authors actually tested this for the other protocols? It should be the case, but if it was tested, this sort of statement could be made with greater confidence.

Yes, few samples were analyzed using IMPROVE_A or NIOSH870 as well. As the results did not differ from the $ATN_{700-400}$ values obtained using EUSAAR2, we did not include this data to avoid complicating the explanation unnecessarily. We added a remark that the applicability of other protocols than EUSAAR2 was observed for a limited set of samples. We further moved the respective passage to Section 4.3 (line 335 - 340 in the revised manuscript), as it represents a result.

Data availability. This does not comply with current AMT standards on data availability.

Thank you for pointing this out. We added data of our samples ($ATN_{700-400}$ and Fe loadings) in Table S2 in the Supplement.

References:

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