Oxidative potential apportionment of atmospheric PM_1: A new approach combining high-sensitive online analysers for chemical composition and offline OP measurement technique

Authors' response

We would like to thank the referees for their time to evaluate our manuscript and for their positive and constructive feedbacks, which helped improve the quality of the paper. Our responses to the comments are presented below (in blue). Orange sentences are directly taken from the corrected manuscript.

General revisions: All grammatical and cross-referencing errors in the text were corrected (listed below). Thank you very much to our referees.

- Line 12. Check first sentence of the Abstract
  Response: A part of the sentence had unfortunately been truncated. It has been corrected by the following sentence: « Source apportionment models were widely used to successfully assign highly-time resolved aerosol data to specific emissions and/or atmospheric chemical processes. ».

- Line 15. Check verb person: OP measurement have…
  Response: The sentence was corrected in the manuscript by the following sentence: « OP measurement has […] ».

- Line 20: use Xact 625i (as in the main text) instead of Xact,
  Response: Corrected.

- Lines 26-27: Sulfate and nitrate formed from SO2 and NOx also originated by combustion processes.
  Response: The sentence has been modified in the manuscript as following: « The results show that besides the high contribution of secondary ammonium sulfate (28%) and organic nitrate (19%), about 50% of PM_1 originated from distinct combustion sources, including emissions from traffic, shipping, industrial activities, cooking, and biomass burning. ».

- Line 29: replaced "is" by "was"
  Response: Corrected.

- Line 62: Chen et al., 2021
  Response: Chen et al., (2021) refers to an OA source apportionment study in Switzerland, whereas Chen et al., (2017) is quoted to support the potential health effects of ambient PM_1.

- Line 85: replace "AE33 data" by "Aethalometer (AE33) data"
  Response: Corrected.

- Line 87: replace "aethalometer by "AE33"
  Response: Corrected.

- Section 2.1. Please, can you clarify the duration of the sampling period for OP? 7 weeks or 15 days?
  Response: The duration of the sampling period for OP was clarified by the following sentence: « Finally, PM_1 collection for OP analysis was performed for 15 days (from 11th July and 25th July 2018) every 4 hours on 150 mm diameter quartz fibre filters (Whatman Tissuquartz) pre-heated at 500°C during 8 hours), using a high-volume aerosol sampler (HiVol, Digitel DA80) at a flow rate of 30 $m^3.h^{-1}$ ».

- Line 254; Replace "toxic lead metals" by "toxic lead forms" or "toxic lead compounds"
  Response: Corrected.

- Line 280; section 2.4.3. Please, provide more information about BCff and BCwb estimation (AAE used…)
  Response: We added the following details to the main text, lines (287 – 289): « BC_{WB} and BC_{FF} were deconvolved based on the model of Sandradewi et al., (2008). We used the 470 and 950 nm wavelengths with a constant absorption Angström exponent of 1.68 and 1.02 for pure wood burning and traffic, respectively, as recommended by Zotter et al., (2017) and Chazeau et al., (2021) ».

- Line 358-362: This paragraph can be simplified.
  Response: The paragraph has been simplified as follows: « Spearman coeficients (r_s) between PM_1 mass measured by FIDAS and OP display some differences (r_s PM_1 vs OP_v^{AA} = 0.23 (p<0.01) and r_s PM_1 vs OP_v^{DTT} = 0.63 (p<0.001)) where PM_1 is much more associated to OP_v^{DTT} than to OP_v^{AA}. These Spearman coefficients are close to those found by in 't Veld et al., (2023) on PM_1 all year long in a similar urban coastal environment (Barcelona) (r_s PM_1 vs OP_v^{AA} = 0.29 (p<0.001) and r_s PM_1 vs OP_v^{DTT} = 0.73 (p<0.001)) ».

- Line 385-386: check sentence
  Response: The sentence was checked and rephrased accordingly: «The PMF_{metals} solution is investigated with the factor profiles and time series presented in Fig. 4, along with the factor relative diurnal cycles and contributions shown in Fig. S8. ».

- Figure 4 (and Figure 5). These figures are difficult to understand as they are now. Improve figure legends. Explain the axes and legend in Figure 4a and 5a.
  Response: The legends for Figure 4 and Figure 5 were improved as recommended.

- Line 399: Why is it limited to public construction? No private construction?
  Response: The sentence has been modified in the manuscript by the following sentence: « The construction work influence is supported by […]. ».

- Lines 486-490: may you explain better the differences/similarities with previous studies?
  Response: The differences and similarities with previous studies were clarified as following: « In overall, the present PMF approach successfully identified various sources of PM_1 during the summer season, consistent with previous studies in Marseille. These sources include traffic (El Haddad et al., 2013; Bozzetti et al., 2017; Salameh et al., 2018), cooking (Bozzetti et al., 2017), and a minor contribution from biomass burning (Bozzetti et al., 2017; Salameh et al., 2018). However, this study marks the first identification of an ON-rich factor.
  Previous source apportionment of $PM_{2.5}$ markers by Salameh et al. (2018) highlighted the dominant contribution of ammonium sulfate in summer (35%) and identified a dust factor with a metal composition similar to the current study (Cu, Fe, Ca). While they identified a fossil fuel factor attributed to mixed harbor and industrial emissions, our results provide new insights by distinctly separating industrial and shipping emissions simultaneously advected onsite by sea breeze. ».

- Line 227: I wonder about the selection of Br for PMF; this element provides little information as tracer of sources. I would exclude it from the PMF dataset and I would try 6 sources with XactPMF

  Response: We agreed with the reviewer that, in general, Br is not attributed to a specific source. However, due to its significant variability and concentrations with 99.8% of data points above the MDL, we deemed Br to be of interest for performing a PMF. Nevertheless, we tried to

increase our solution to 6 factors and excluding the Br element (Figure A1). The new resolved factor is interpreted as a split of the dust resuspension factor, dominated by Cu. Despite Cu often being associated with brake lining, this factor presented no correlation with traffic tracers (BC_{FF}, NO_x, HOA) or any related diurnal patterns. Consequently, this solution did not offer further information and was not retained in the study.
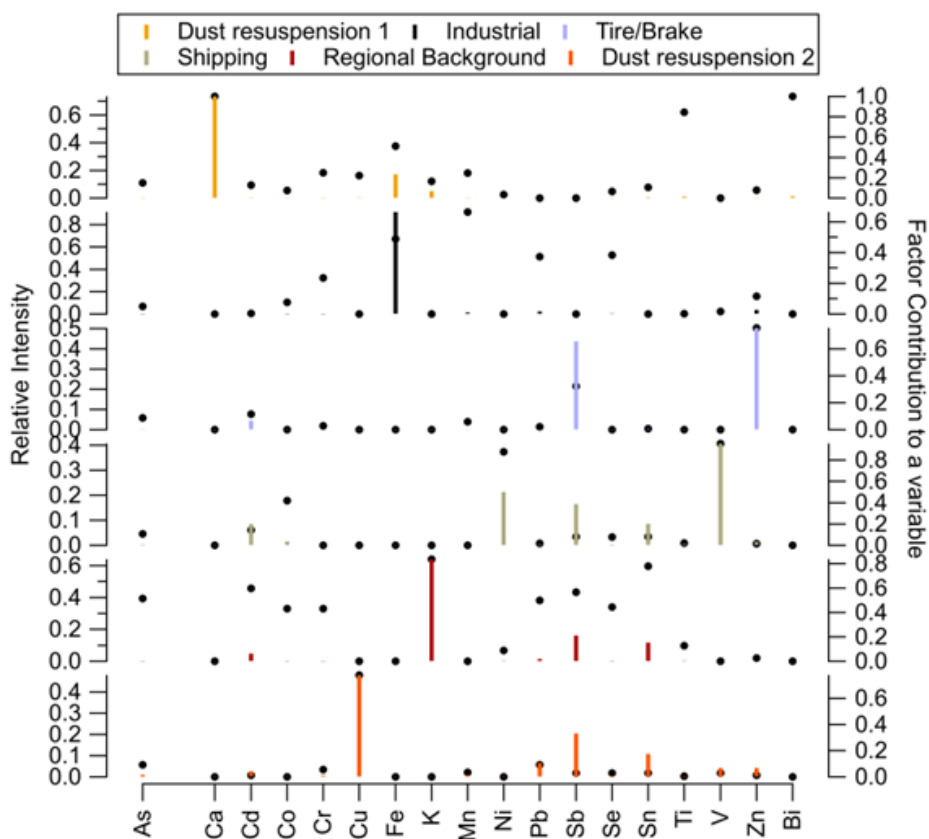
Figure A1- Average factor profiles from the PMF_metals solution with Br excluded.

- Plot 3b: missing x-axis
  Response: The x-axis has been added on the Figure 3b.

- Line 423: missing year of publication of Salameh et al.
  Response: It has been corrected by: « Salameh et al. 2018 ».

- Line 474-476. SO42 also tracer of the shipping profile. This is the second factor explaining variation of BCwb after the BB.
  Response: Indeed, we add the following sentences: « This factor further accounts for a noticeable variation of sulfate (11.6% of the total sulfate concentration). This is in agreement with the results from Chazeau et al., (2021) indicating that during 25% of the days in summer 2017, sulfate concentrations were prominently influenced by the nearby harbor. ».

- In the references, 3 publications from Bozzetti et al., are cited. All of them from 2017. In the text is not clear which one is referenced and when because they are all identified as Bozzetti et al., 2017.

129   Response: There was a duplicate among the three publications. The two « Bozzetti et al. 2017 »
130   publications have been differentiated by the addition of (a) and (b).

131

132   - Line 566: missing reference.
133   Response: The reference "Weber et al. 2021" has been added.

134

Response to anonymous referee #1:

Referee comment: The paper is of interest and deserves to be published in ACP although there are some aspects that can be improved. My main concern is about the application of the PMF to the data set of metal concentrations before the final PMF-PM1. The results obtained from the PMF metals are not entirely satisfactory. The diurnal pattern of the tire/brake factor is difficult to explain (despite frequent spikes). The so-called regional background factor, contains typical brake tracers, such as Cu and Sn, also Sb. This possible misidentification is reflected in Figure S14 and may affect the analysis of PMF-PM1. I understand that the PMF-OA can simplify the application of the final PMF-PM1. However, in my opinion, the prior grouping of the metals into 5 factors decreases the information for the PMF-PM1 and makes interpretation more difficult. Have you tested the PMF-PM1 run using BC sources, OA sources, ions and metal concentrations? If not, I suggest doing so and comparing the results between the two approaches.

Response: We agree with the reviewer that the interpretation of the PMF_{metals} results needed some clarifications. It is commonly understood that elements such as Cu, Sb, Sn, and Zn are typically assigned to brake lining and tire wear emissions. While it has already been pointed out for the coarse ($PM_{10-2.5}$) and intermediate ($PM_{2.5-1}$) fraction, it is less clear for the fine PM_1 fraction. Visser et al. (2015b) demonstrated that elements such as Cu, Sn, Sb are mainly found in the coarse mode at the "Marylebone Road" kerbside site. Hays et al. (2011) described similar trends for a near-highway site, with only Zn being significantly present in the fine mode. Additionally, these mentioned elements are not found to be correlated with each other. Such results suggest the existence of significant alternative source for these elements, potentially mixed here in a regional-scale background factor. Since most trace elements in the fine mode are non-volatile, they can undergo long-range atmospheric transport (Morawska and Zhang, 2002).
This lack of identification was also described by (Visser et al., 2015a), who identified only a mixed traffic-related factor for the PM1-03 fraction, whereas a brake wear factor was resolved for the $PM_{2.5-1}$ and $PM_{10-2.5}$ fraction. In our study, the tire/brake wear factor is mainly constituted of Zn and Sb, which are among the most represented tracers for these sources in the fine mode. This statement was clarified into the manuscript (lines 429-433) : « Visser et al. (2015b) demonstrated that elements usually assigned to brake lining and tire wear emissions (e.g. Cu, Sb, Fe or Sn) are mainly found in the coarse mode at the "Marylebone road" kerbside site, and Hays et al. (2011) reported similar trends for a near-highway site in Raleigh, with Zn being the only element significantly present in the fine mode. Such results suggest the existence of significant alternative source for these elements, potentially mixed in the regional-scale background factor. »
Lines 436-437: « Since most trace elements in the fine mode are non-volatile, they can undergo long-range atmospheric transport (Morawska and Zhang, 2002). »
We also emphasize in the conclusion that incorporating the measurement of additional elements, such as Ba, S, Cl, and Si, could be an interesting feature to refine some sources. Regarding the PMF methodology, we followed the reviewer's recommendation and we performed a PMF-PM1 using BC sources, OA sources, ions and metals concentrations. We added a dedicated paragraph on the comparison between the two approaches in the main text as follows (lines 521-533):
« To assess the robustness of the PMF² solution, the results were compared to a PMF solution utilizing the OA factors from $PMF_{organics}$, ACSM inorganic species (SO_4^{2-}, NO_3^-, NH_4^+, Cl^-), BC sources and metals concentrations as the input dataset. Consistent with the PMF² method, constrains, instrument weighting, criteria selection and bootstrap analysis were applied and are reported in the Supplement

179   section. This alternative approach successfully identified the same 8 factors (Fig. S18, exhibiting
180   comparable mass contributions and very high correlations with the PMF² factors time series (Table S5),
181   all exceeding a R² of 0.9, except for shipping (R²=0.81).
182   The biomass burning and shipping factors accounted for slightly higher concentrations in the PMF²
183   solution, due to slightly elevated contribution of SO_4^{2-}, NH_4^+ and MOOA concentrations which dominate
184   the PM_1 mass. The metals composition found in the factors from this alternative PMF approach is in
185   agreement with the metals profiles from the PMF_{metals} solution. Note that Zn and Sb, the most prominent
186   elements in the tire/brake metals factor were mainly present in the traffic source. However they
187   displayed again some mixing with other factors (dust resuspension, AS-rich and cooking), suggesting
188   additional sources unresolved by the current PMF solutions. Previous studies suggested that Zn may
189   originate from waste incineration or other industrial processes (Belis et al., 2019; Visser et al., 2015a;
190   Manousakas et al., 2022). Comparable results in terms of explained variability were observed,
191   emphasizing the suitability of both methods for such study. »
192
193   We also provide further details in the supplements, with the Figure A2 (Figure S18 in the main text) and
194   Table A1 (Table S5). Lastly, we further discuss the comparison between PMF methodologies in the
195   response to reviewer #2.
196
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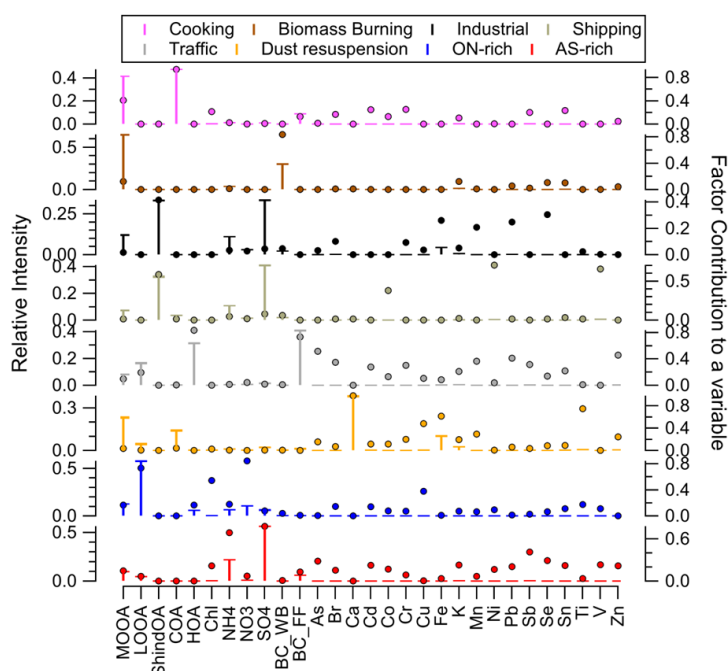

199   **Figure A2-** Average factor profiles for the PMF_{PM1} solution using OA factors, ACSM inorganic species, BC and
200   metals as inputs. The sticks represent the normalized contribution of the variable to the factor (left axis) and
201   markers show the normalized factor contribution to each variable (right axis).
202

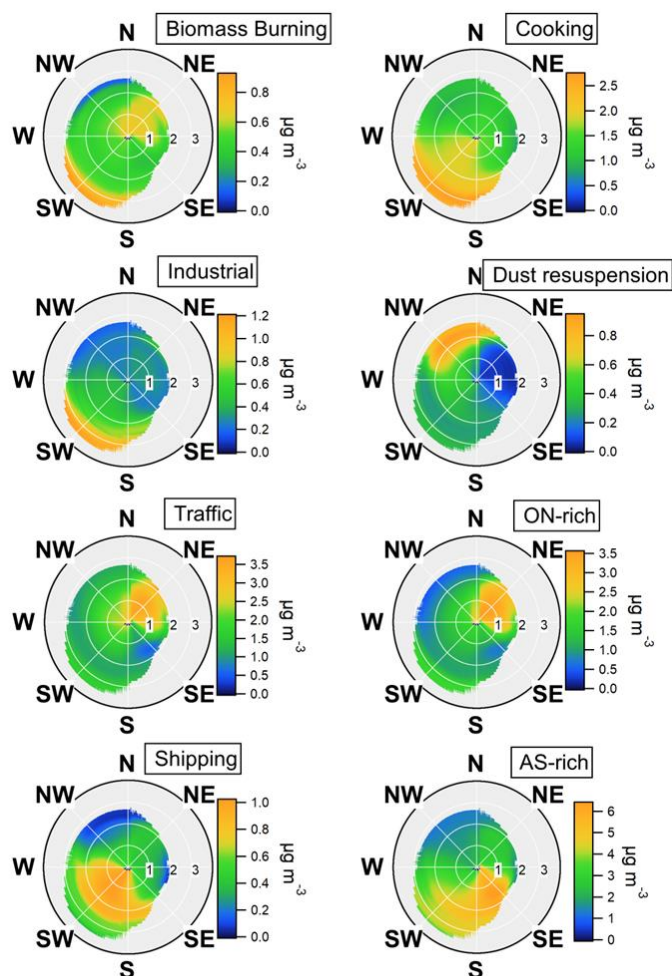
	R² with PMF² factors	Slope with PMF² factors	Relative contribution (%)
Cooking	0.97	1.18	17.4
Biomass Burning	0.93	0.65	3.3
Industrial	0.95	0.95	2.5
Shipping	0.81	0.52	3.6
Traffic	0.96	1.02	21.5
Dust resuspension	0.99	1.07	2.6
ON-rich	1	1.15	23.1
AS-rich	0.99	0.98	26

Table A1- R² and slope values for the comparison of the PMF$_{PM1}$ (OA factors + metals + ions + BC) factors with PMF² factors. The relative contributions are also represented (in %).

Supplements:
Preparation of the PMF$_{PM1}$ with OA factors + metals + ions + BC dataset:
Among the 8 identified factors, 4 were not systematically resolved across the several preliminary runs (cooking, biomass burning, industrial and shipping factors). The solution was constrained using base case profiles from the 10 factors-solution for industrial, the 11 factors-solution for cooking and shipping, and the 12 factors-solution for biomass burning. Note that for each run we applied the same C-values for the instrument weighting than PMF² solution. A bootstrap analysis was performed for 100 runs and the accepted runs based on the pre-defined list of criteria (the correlation with base case profiles for the constrained factors and the monitoring of the dominant variable intensity for the unconstrained factors) were averaged into a definitive solution.

Regarding the factors identified by PMF-PM1, the diurnal cycles identified for the Biomass Burning factor are not clear. Furthermore, considering that this source was not identified from the PMF-OA, the relatively high contribution obtained for this factor (5% of PM1) in July is surprising. As was done with the PMF-metals, it would be useful to perform NWR analysis for the final PMF-PM1 factors.

Response: As mentioned by the reviewer, unfortunately a BBOA was not resolved for the PMF-OA. The absence of local domestic heating emissions during this season has made its identification difficult. While El Haddad et al. (2013) didn't resolve this factor with the c-ToF-AMS neither, Bozzetti et al. (2017) with the offline AMS technic and Salameh et al. (2018) using PM$_{2.5}$ offline markers identified a low biomass burning contribution in summer, with 5% of the total OA concentration and 2% of the PM$_{2.5}$ concentrations, respectively. In the current study, the PM$_1$ biomass burning factor was mainly constituted by MOOA (64% of the factor) and to a lesser extent by BC$_{WB}$ (19%). Numerous studies identified a biomass burning factor for OA that exhibits characteristics of an oxidized OA profile, with enhanced signal at m/z 29, m/z 44 (Belis et al., 2019; Bougiatioti et al., 2014). In the PMF-OA, a portion of MOOA may account for a secondary biomass burning origin (e.g. wildfire, agricultural activities), as the main BBOA fingerprints, m/z 60 and m/z 73 were both predominantly attributed to this factor (40% and 39%, respectively).
The influence of more secondary process for the biomass burning factor was stated lines 463–467 in the manuscript:
« While no primary biomass burning organic aerosol (BBOA) factor was resolved with the PMF$_{organics}$ analysis in summer, the presence of a significant MOOA contribution reflects the influence of secondary process in this biomass burning factor. The low concentration of this factor is in agreement with minor regional emissions linked to agricultural activities, wildfires and cooking practices such as BBQ, transformed through oxidation processes during regional transport and aging (Chazeau et al., 2022; Cubison et al., 2011) ».

242   Moreover, we followed the reviewer's suggestion and the NWR analyses for the PMF-PM$_1$ factors are
243   displayed in Figure A3 (Figure S17 in the main text). The following lines were modified accordingly:
244   -lines 458 – 460 : « The full PM$_1$ source apportionment solution is explored in this section with the
245   average factor profiles (Fig. 5a), the time series (Fig. 5b), the pie chart of mass contributions (Fig. 5c),
246   the average diurnal profiles (Fig. 5d) and the NWR analyses (Fig. S17). »
247   -lines 467 – 470 : « The NWR analysis in Fig. S17 showed biomass burning concentrations associated
248   with higher wind speed than sources with a local origin (traffic, shipping, cooking and ON-rich),
249   corresponding to south-westerly winds from the Mediterranean Sea. Additionally, the north-east land
250   breeze advected these aged emissions back to the sampling site. »
251   -line 505 : « […] This factor displayed an origin from the North to East within the land. »
252   -line 519 : « […] This interpretation is supported by the NWR analysis presented in Fig. S17. »
253
254
255

256
257   **Figure A3**- NWR plots for each factor of the PMF$_{PM_1}$ analysis.
258

259   Finally, I think the conclusions section could be improved. The discussion can be extended on the
260   advantages/disadvantages of the proposed method and the comparison with previous PMF analyzes
261   carried out in the area.

262   Response: We thank the reviewer for the suggestion. We added the following discussion to the
263   conclusions in lines 666-674: « The PMF² approach successfully identified 8 well-resolved sources (AS-
264   rich, traffic, ON-rich, cooking, shipping, biomass burning, industrial and dust resuspension), a solution
265   not achievable through single PMFs conducted separately on OA and metals datasets. The method

266 enabled the assignment of OA factors, which typically described components arising from a mixture of
267 sources and chemical processes rather than a single emission source, to more specific PM_1 sources.
268 Additionally, this approach allowed to assess both the primary and secondary origin of anthropogenic
269 sources, such as traffic and cooking. However, a limitation of this method is that non-explained
270 variability and uncertainties of the factors from the first step PMFs will propagate into the PMF² results
271 and therefore need to be carefully assess. The inclusion of additional elements measurements, such as
272 Ba, S, Cl, and Si to the PMF_{metals}, could be an interesting feature to refine some sources and address this
273 limitation. »

274 A comparison with previous PMF analyzes carried out in the area was also detailed in the section 3.2.3
275 (lines 534-541): « In overall, the present PMF approach successfully identified various sources of PM1
276 during the summer season, consistent with previous studies in Marseille. These sources include traffic
277 (El Haddad et al., 2013; Bozzetti et al., 2017a; Salameh et al., 2018), cooking (Bozzetti et al., 2017a),
278 and a minor contribution from biomass burning (Bozzetti et al., 2017a; Salameh et al., 2018). However,
279 this study marks the first identification of an ON-rich factor. Previous source apportionment of $PM_{2.5}$
280 markers by Salameh et al. (2018) highlighted the dominant contribution of ammonium sulfate in summer
281 (35%) and identified a dust factor with a metal composition similar to the current study (Cu, Fe, Ca).
282 While they identified a fossil fuel factor attributed to mixed harbor and industrial emissions, our results
283 provide new insights by distinctly separating industrial and shipping emissions simultaneously advected
284 onsite by sea breeze. ».

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288 **Response to anonymous referee #2:**

289 Referee comment:

290 • Line 453-454: the sentence stating that the industrial contribution to PM1 found in Marseille is
291   comparable to the contribution observed in other cities might lead to misleading conclusions.
292   The industrial contribution being similar and low in different cities could be merely casual or
293   due to the distance of the sampling stations from the emission spots, and on type of the industrial
294   processes involved. I think this similar and low industrial contribution to PM1 among different
295   cities only demonstrates that the urban background stations are typically scarcely affected by
296   industrial emissions because of their geographical location.

297 Response: This statement, as written, is indeed unclear and prone to misinterpretation, thanks for
298 noticing it. We agreed the distance between the industrial area and the urban site may account for the
299 observed very low concentrations in comparison to more local sources. However, it is important to note
300 that industrial plumes are transported by sea breeze conditions, which prevail almost daily in the
301 Marseille area during the summer.
302 The low PM_1 mass concentration for this source is expected as the size of the industrial particles
303 generally belongs to the ultrafine mode (<100nm) (Riffault et al., 2015). Chazeau et al. (2021) and El
304 Haddad et al. (2013) already described that plumes originated from the main industrial area of Fos-Berre
305 are mainly attributed to ultrafine particles and thus influence the mass only to a minor extent. This
306 clarification is now articulated in the main text as follows, lines 478-483: « The factor contributes little
307 to the PM_1 composition (3.2%), which is expected as the size of the industrial particles generally belong
308 to the ultrafine mode (<100nm) (Riffault et al., 2015). Chazeau et al., (2021) and El Haddad et al., (2013)
309 already described that plumes originated from the main industrial area of Fos-Berre are advected onsite
310 by sea breeze conditions and are mainly attributed to ultrafine particles, influencing the mass
311 concentrations only to a minor extent. Similar contributions were found in another Mediterranean coastal
312 city, Barcelona (4%; Via et al. (2023)), and in some French urban sites in the vicinity of an industrial
313 area (Weber et al. 2019). ».

314   • Regarding the interpretation of the OP results:

315   The OP_{AA} and OP_{DTT} time series displayed in figure 3a and 6a seems to show a weak correlation. This
316   is also reflected by the OP multilinear regression results displayed in figure 7 and table 1, where some
317   PMF factors e.g. dust resuspension and industrial emissions are positively correlated with OP_{AA} and
318   negatively correlated or not correlated with OP_{DTT}. Vice-versa the shipping emission factor and the AS-
319   rich factor are positively correlated with OP_{DTT} and negatively correlated or not correlated with OP_{AA}. I
320   would discuss more in depth the differences of the results obtained from OP_{AA} and OP_{DTT}. What's the
321   physiological representativeness of OP_{AA} and OP_{DTT}? Which one is more relevant for human health?
322   Both DTT and AA are reductant substances. It seems that AA is more sensitive to Cu and other elemental
323   impurities, while DTT is sensitive to other oxidative species, therefore it seems that OP_{DTT} and OP_{AA} are
324   related to different oxidative pathways. Could you briefly elaborate on the physiological
325   representativeness of these two pathways and which one is more relevant and the specific relevance of
326   each one? Do OP_{AA} and OP_{DTT} provide complementary information or one is more representative than
327   the other of the real oxidation processes occurring in-vivo? If OP_{DTT} and OP_{AA} provide complementary
328   information, do the authors suggest to always perform both the analyses?

329   Response: Thank you for the feedback, which has enabled us to provide further details on the
330   interpretation of the OP results and add a section "3.3.4. Discussion" to the manuscript. Indeed, the
331   association between OP^{AA} and OP^{DTT} is moderate to weak ($r_s = 0.41$; $p<0.001$) and illustrates the
332   different sensitivity of the two OP tests to chemical constituents found in ambient air. As shown in the
333   Figure 7, the contribution of the PM sources to OP is dependent on the OP test used. This reflects the
334   different oxidation pathways involved in the oxidation of the two probes (AA and DTT). A detailed
335   answer for the physiological representativeness of OP_{AA} and OP_{DTT} has been added in the manuscript,
336   in the section "3.3.4 Discussion", lines (630 – 637):

337   "AA is naturally present in the lungs, and its predominant anionic form in solution (HA$^-$) is oxidised by
338   various mechanisms facilitated by OH$^\bullet$, $O_2^{\bullet-}$, $HO_2^\bullet$ and other radicals, and by transitions metals as Cu (II)
339   or Fe (III) (Campbell et al., 2019). DTT has a disulfide bond and is considered as a chemical substitute
340   for cellular reducing agents such as nicotinamide adenine dinucleotide phosphate oxidase (NADPH) or
341   protein thiols (Verma et al., 2015; Borlaza et al., 2018). Protein thiols play an important role in major
342   oxidative stress, restoring the redox balance by eliminating free radicals (Baba and Bhatnagar, 2018).
343   Many studies have linked these two probes (AA and DTT) to transition metals (Cu, Fe, Mn, Zn), EC
344   and OC (Gao et al., 2020). In addition, the different sensitivity of AA and DTT to both organic
345   compounds and transition metals has been evidenced in Calas et al., 2018, Gao et al., 2020 and
346   Pietrogrande et al., (2022).

347   Although no consensus has been reached on an OP test that is more representative of health impact,
348   epidemiological studies have mainly associated OP^{DTT} with health endpoints, which has not been
349   demonstrated with OP^{AA}. The community currently recommends the complementary use of these two
350   tests. A detailed response has been added in the manuscript, in the section "3.3.4 Discussion", lines
351   (638 – 648):

352   "Today, no consensus has yet been reached on which OP test is most representative of health impact,
353   and the community still recommends the complementary use of OP tests, in particular the association of
354   both AA and thiol-based (DTT or GSH) assays (Moufarrej et al., 2020). This association is today the
355   unique way of assessing the full panel of the most oxidising compounds of PM. However, recent studies
356   have shown positive associations between OP^{DTT} and various acute cardiac (myocardial infarction) and
357   respiratory endpoints, supporting the interest of the OP^{DTT} assay for this purpose (Abrams et al., 2017;
358   Weichenthal et al., 2016; He and Zhang, 2023). On the contrary, several studies did not associate OP^{AA}
359   to health endpoints including early-life outcomes, respiratory and cardiovascular mortality,
360   cardiorespiratory emergencies and lung cancer mortality (Borlaza et al., 2023; Marsal et al., 2023).
361   Nonetheless, a recent study has associated OP^{AA} with oxidative damage to DNA (Marsal et al., 2023).
362   These results so far may suggest that OP^{AA} provides partial information on the link between OP and
363   adverse health effects, and further epidemiological studies are needed to determine whether OP^{AA} should
364   be considered as a proxy for health impact."

365 Moreover, from the results displayed in figure 7, where the sources are ranked by their contribution to
366 OP_{DTT}, OP_{AA} and PM_1, it seems that there's a certain correlation between the sources contributions to
367 PM_1 and OP_{DTT}. On the opposite, such a correlation is completely missing between sources contribution
368 to OP_{AA} and PM_1 mass. Does it suggest that AA is more sensitive to the chemical composition of the
369 sources, while DTT is more sensitive to the aerosol concentration, and therefore less representative of
370 the real oxidative potential of an aerosol source? Without a critical discussion on these aspects, the
371 results displayed in figure 7 might lead to contradictory conclusions, for example the industrial factor
372 can be considered as toxic or non toxic if looking respectively at OP_{AA} or OP_{DTT} results.

373 Response: Thank you for your pertinent comment. Indeed, as in many other studies referenced in section
374 "3.1 "OP results" lines (371-372), PM_1 is more associated with OP^{DTT} than with OP^{AA} (r_s PM_1 vs OP_v^{AA}
375 = 0.23 (p<0.01) and r_s PM_1 vs OP_v^{DTT} = 0.63 (p<0.001)). Associations values were mentioned in the
376 manuscript in lines (366-367), but these values have not been discussed in depth. These values reflected
377 the sensitivity of DTT to a wider range of chemical compounds, implying a stronger association with
378 aerosol concentration, whereas AA displays a heightened sensitivity to chemical composition (which
379 exhibit robust specificity). Indeed, OP_v^{AA} is known to be more sensitive to some PM components as
380 Cu(II) or Fe(II) but also some quinones (Calas et al., 2019, Campbell et al., 2019; Pietrogrande et al.,
381 2022). In addition, the state-of-the-art highlighted PM concentration as a significant predictor of OP_v^{DTT}
382 in univariate models (Janssen et al., 2014; Weber et al., 2018). For the moment, we need to keep a
383 critical eye on the results since a multitude of sources have been identified by the two OP tests and
384 therefore deserve to be considered. A detailed response explaining the observed correlation coefficients
385 has been added in the manuscript, section 3.1 "OP results", lines (366 – 373):

386 "Spearman coeficients (r_s) between PM_1 mass measured by FIDAS and OP display some differences (r_s
387 PM_1 vs OP_v^{AA} = 0.23 (p<0.01) and r_s PM_1 vs OP_v^{DTT} = 0.63 (p<0.001)) where PM_1 is much more
388 associated to OP_v^{DTT} than to OP_v^{AA}. These Spearman coefficients are close to those found by in 't Veld
389 et al., (2023) on PM_1 all year long in a similar urban coastal environment (Barcelona). The higher
390 association between OP_v^{DTT} and PM_1 compared to OP_v^{AA} and PM_1 has already been observed in other
391 studies conducted on PM_{10} (Calas et al., 2019; Weber et al., 2021; Janssen et al., 2014). This phenomenon
392 is attributed to AA's heightened sensitivity to chemical composition, exhibiting robust specificity.
393 Moreover, DTT demonstrates superior sensitivity to aerosol concentration owing to its more balanced
394 sensitivities to chemical constituents (Gao et al., 2020)."

395 • Regarding the source apportionment strategy:

396 I suggest the authors to justify the adoption of the PMF^2 approach. This approach utilized the outputs of
397 the OA and metals source apportionments as input for a comprehensive PM_1 source apportionment.
398 Alternatively, a unique PMF analysis could have been performed using the ACSM and Xact raw data
399 as direct inputs for the PM_1 source apportionment. The PMF^2 approach has two drawbacks. Firstly, the
400 uncertainties of the first PMF analyses and their unexplained variability are propagated into the
401 PM^1 source apportionment. Secondly, the OA and metal aerosol sources, which had been already
402 resolved by the first PMFs (on ACSM and Xact data), are then reapportioned and potantially re-mixed
403 into different PM^1 factors. This is observed in figure 5a for the traffic and cooking factors, where a non-
404 negligible contribution from LOOA and MOOA is observed. Similarly, the brake/tire factor resolved by
405 the metals' PMF, is splitted into 4 PM^1 factors (traffic, AS-rich, industrial, and biomass burning). This
406 suggests that either the PM_1 source apportionment hasn't been fully-optimized, or the input factor time
407 series were already not well resolved from other sources, and therefore the error of the OA and metal
408 PMFs have been propagated into the final PM_1 source apportionment. Instead, using the OA ACSM raw
409 data as input for the PM_1 source apportionment might help resolving a better traffic profile, because the
410 OA ACSM mass spectra contain many hydrocarbon fragments which are typically related to traffic
411 exhaust.

412 Response: We thank referee #2 for the insightful comments. One of the objectives of the current study
413 was to assess the PM sources contributions to OP through three scenarios: first, an OP apportionment
414 using only OA factors from the $PMF_{organics}$; second, an OP apportionment using only metals factors from
415 the PMF_{metals}; and third, to follow an harmonized methodology, we explored the third scenario (OP

416   apportionment using PM_1 factors) by combining together the factors from both $PMF_{organics}$ and PMF_{metals}
417   as inputs for the PMF_{PM1}. The PMF² approach was the most suitable method for this purpose,
418   emphasizing the importance of considering all PM_1 fractions to apportion OP.
419   Moreover, using OA PMF factors as inputs allows to quantify the primary/secondary OA contribution
420   to the PM_1 sources. A limitation of performing PMF on OA mass spectra from ACSM/AMS is the
421   resolution of the SOA origin. SOA factors are usually reported as either a single factor or two factors
422   separated by their degree of oxygenation (LOOA/MOOA) rather than in terms of sources. The PMF²
423   approach enables a more accurate identification of SOA sources, addressing this limitation. Given that
424   several studies highlighted the role of SOA in oxidative potential, it is important to include an accurate
425   quantification of this fraction in the PM_1 sources, a step not achievable using the raw OA mass spectra.
426   We added to the Introduction section the justification of using PMF² method (lines 87-92): « A known
427   drawback of performing PMF on OA mass spectra from ACSM/AMS is the resolution of the secondary
428   organic aerosol (SOA) origin. SOA factors are usually reported as either a single factor or two factors
429   separated by their degree of oxygenation rather than in terms of sources. A PMF² approach using
430   previous OA factors combined with other species and/or PMF factors may enable a more accurate
431   identification and quantification of the SOA fraction in the PM sources. The current study addresses this
432   challenge by intending the PMF^2 method for the PM_1 fraction measured with online analysers (i.e. ToF-
433   ACSM, Xact 625i and AE33) at high time resolution (<1h). »
434   We fully agree with the reviewer about the first drawback. Since we performed bootstraps for the two
435   first PMFs we were able to statistically estimate uncertainties of the factors. These uncertainties are
436   incorporated in the error inputs for the PMF_{PM1} analysis. It is true also that the non-explained variability
437   of the first PMFs is propagated into the PM_1 source apportionment, representing a notable inconvenience
438   of a multi-step PMF approach. This is now explicitly stated as a limit of the methodology in the
439   conclusion (lines 671-673): « However, a limitation of this method is that non-explained variability and
440   uncertainties of the factors from the first step PMFs will propagate into the PMF² results and therefore
441   need to be carefully assessed. »
442   HOA and COA were constrained in the $PMF_{organics}$ using reference profiles accounting for primary traffic
443   and cooking emissions in an urban environment. Therefore, the contribution of the fast oxidation of
444   freshly emitted primary OA is expected to be included in the SOA factors, as demonstrated in Chazeau
445   et al. (2022). This explains why some LOOA and MOOA fractions are attributed to the traffic and
446   cooking sources. The SOA contribution to the traffic source was previously mentioned lines 491-493:
447   « It should be emphasized that 23% of the traffic source was constituted of SOA (LOOA and MOOA)
448   meaning that primary traffic contribution is mixed with secondary aerosol concentrations attributed to
449   fast oxidation of freshly emitted particles (Chirico et al., 2011). ».
450   The tire/brake factor displayed the highest unexplained variation, probably due to some mixing with
451   other sources as suggested by the reviewer. It was previously noted in lines 490-491 and is now further
452   discussed in the new paragraph comparing the two PMF² approach.
453   We agree with the reviewer that including OA raw data as PMF inputs is a very interesting method to
454   explore the PM_1 sources as it was already performed by Belis et al. (2019). There are many possibilities
455   in combining datasets, whether in their raw format or as PMF factors, that would need further
456   investigation to establish a more standardized protocol for PM_1 source apportionment. Despite this
457   statement, the scope of the present manuscript is not to inter-compare alternative PMF methodologies,
458   which could be the focus of a fully dedicated paper.
459   However, we inspected correlations between some hydrocarbon OA fragments related to traffic exhaust
460   and the other PM_1 compounds (metals, BC, SO4, NO3, NH4 and Cl) in Figure A4. The results did not
461   show any better correlation than comparing with the HOA factor. For these reasons, we do not think it
462   would be appropriate to present results from a PMF analysis based on OA fragments + inorganic
463   compounds.
464   Nevertheless, we performed PMF on OA factors and metals, as suggested by the referee #1, and
465   compared it to the PMF² approach to support our assessment. The results were relatively similar and are
466   detailed in lines 521- 533 in the section 3.2.3.

467
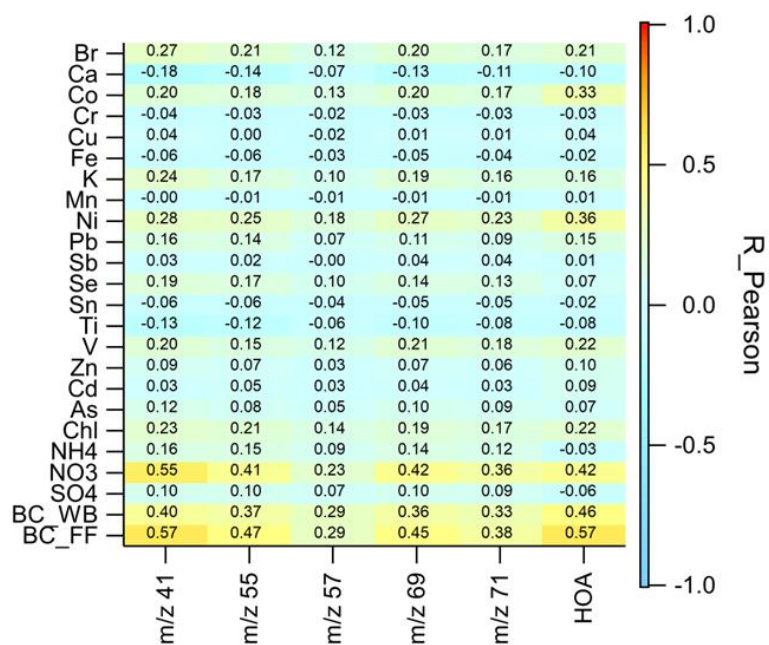
Figure A4 – R Pearson correlation matrix between metals, BC, ACSM inorganic species and OA hydrocarbon
fragments typically related to traffic exhaust.

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