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Atmospheric Measurement Techniques Discussions

Interactive comment on "Quantitative measurement of PM_{10} by means of X-ray fluorescence spectra" *by* E. Busetto et al.

Anonymous Referee #5

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This paper, before publication, needs a deep rearrangement. Many sections are not adequate and not. Several statements are not adequately supported by proper references or "in depth" discussions. This is probably due to the fact that Authors applied for a Patent on the instrument. However, even in this case, a better description would not affect the basic requirements for protecting a Patent application. In conclusion, the paper cannot be accepted in the present form. Some further comments are given below.

Title The title is not appropriate since in the common wording, "measurement of PM10" means the measurement of PM10 mass concentration (μg/m3). The paper seems to be also addressed to other size classes (such as PM2,5), thus reference to PM10 only in the title is not appropriate. The measurement is carried out by means of X-Ray

fluorescence spectroscopy. In conclusion, a more suitable title would be "Quantitative measurement of elemental composition of airborne particulate matter by means of fluorescence spectroscopy:

Abstract: Real Time measurement usually means that the analysis is carried out on a short time basis, i.e., time comparable with the dynamics of the atmosphere under investigation. In this case„ a sampling time of two days would imply automatic measurement, but not carried out in real time. A statement regarding "smaller error" in the abstract would require a comprehensive discussion of sensitivity and reproducibility which is not carried out in the text. Submission for a patent (line 14) is not significant in an abstract.

Introduction Industrialization do not push people near factories. In a post industrial society, people like to live away from polluting sources, thus this statement should be changed. Effects of particulate matter are described in many WHO reports. Authors list three different WHO reports and that may cause some confusion in the reader. In addition to health effects, elemental composition of particulate matter is also important for source apportionment studies. A brief discussion on this aspect is also necessary Lines 7-10: In the description of ICP-MS, updated references should be given. Line 24 : Again, make reference to quantitative analysis of elemental composition. Line 29: A "new mass calibration method" is reported. However, it is not clear (at least at this point) why the method is new.

Instrumentation and measurement technique Line 16: A brief comment on the elemental composition of filters should be added (Blank concentrations). Line 18: Sampling procedures are not reported in The Directive, but in the European standard EN1234 (For PM10). The Directive 2008/50/EC impose that sampling should be carried out according to this Standard. Line 19-23: Commercial information should be complete including name of suppliers and address or any other info for easier location. Line 26-27: Since not all readers could be familiar with Multichannel analysers, a brief description of this component should be given. A block scheme of the system could be

useful. Line 18: It is not clear how MCA calibration Is effective on time and upon environmental conditions. Since this is considered a critical parameter, more info are needed.

Absolute mass calibration procedure The distribution of particulate matter on filters differs from the calibration procedure suggested in the paper. Trace elements in particulate matter are not isolated, but diluted in a complex solid matrix which contains organic material, liquid water and so on. In addition, especially on fiber filters such as quartz, particulate matter may penetrate inside the filter media, i.e., away from the surface. Thus, the calibration procedure is not really consistent with the physical pattern of sampled particulate. This means that the calibration, carried out on micro-crystals on a flat surface, is not really the same of the field samples. A discussion on this aspect is needed. More information should be given about the software procedure to calculate the absolute mass of the element Figures 3 are redundant. One figure for one selected element (e.g., Lead) should be sufficient. Figures 3 cover mass concentrations range not really consistent with application in air pollution control. For instance, the concentration limit for Lead is 0,5 μg/m3 as annual average. Assuming that the system is sampling air at 2.3 L/min and that about 1000 m3 are sampled every two days, at concentrations close to the limit the absolute amount of Lead would be 500 μg. This is consistent with the calibration curve. However, for other metals such as Cd, As, Ni the limit values are much lower. Namely: âĂć arsenic: 6 ng/m3; âĂć cadmium: 5 ng/m3; âĂć nickel: 20 ng/m3; which means a total amount of a few μg. Thus, the calibration curve should be prepared according to the expected concentrations. It is therefore recommended that an extensive discussion about the performance of the suggested technique should be carried out for the elements of environmental significance defined by the European Commission (Pb, Cd, As, Ni). In addition, the paper addresses calibration on pure substances. Nothing is said about the detection limits in real samples. Although the last statement of the chapter excludes the presence of interferences from heavier elements, possible mutual interferences are not given.

Results and discussion Table I and Figures 4 are reporting the same information, thus the table or the figures should be omitted. Line 25: A discussion about the errors in terms of precision is missed. As shown in Figure 5, the Authors found a good agreement between their measurements and data gathered from conventional methods. However, results are for Ca and Fe which are not important environmental elements. Since ICP-MS is also able to detect other elements, presentation of results for other elements would be welcome.

Conclusion Authors suggest that PM-SMS can be a valuable monitoring tools where a risk of heavy metal contamination could be present. However, heavy metals have been just missed by the paper

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