A real-time analyzing and weighing system

D. Weidauer, C. Bey, F. Freyer and N. Derenda

1Comde-Derenda GmbH, Kieler Str. 9, 14532 Stahnsdorf, Germany

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Presenting author email: weidauer@comde.de

Gravimetric measurement is the reference method for determination of particulate matter in ambient air (EN 12341), (EN 14907). This method requires sample intervals as long as 24 h and conditioning phases as long as 48 h before a precise result can be obtained. Once the mass of a loaded filter disk has been determined to a satisfactory precision, the disk can be processed further by methods such as chemical analysis, laser spectroscopy or X-ray analysis. This entire process chain, including scheduling and transport, can easily take a week or longer until all results are available.

The authors present a setup that combines (a) aerosol sampling on filters with (b) a scattered light photometer, (c) a sub-microgram balance, (d) an X-ray fluorescence spectrometer and (e) an X-ray diffractometer in a complete, self-contained system. The workflow is handled by a robotic system implementing a workflow optimised for quick preliminary and early final results. The operating concept of this setup includes the following interactions between the contained instruments:

(i) The scattered light photometer provides highly time-resolved data about the aerosol concentration. As well as being capable of providing this data online, it can be used to determine the minimum required sampling time for the filter disk currently placed in the suction line. In cases of high concentration, the filter collects a significant mass in a relative short time interval, so the filter could be changed and processed earlier than according to the standard time intervals.

(ii) The advantage of the scattered light photometer data is its time resolution and real-time availability. However, the photometer signals are not only dependent on the concentration and size distribution, but also on the particles’ colour and brightness. It is therefore an attractive option to post-calibrate photometric data by the results of gravimetry as soon as the latter are available.

(iii) Weighing a filter disk requires conditioning for a defined humidity. The robotic system however allows repetitive weighing during the conditioning phase, providing information about the hygroscopicity of the collected dust. The final result after the legally required conditioning time may already be estimated after a fraction of that time. The precision of this estimate will increase with the number of weighing processes the robotic system can accommodate in this period.

(iv) X-ray fluorescence spectroscopy provides information about the elementary composition of the sampled dust. This is a standard method e.g. for detection of heavy metals. The difficult quantitative analysis can be supported by the results of the gravimetric measurements.

(v) X-ray fluorescence provides elementary composition only. Crystalline phases however can be distinguished by X-ray diffraction, providing valuable information complementary to the element analysis.

(vi) The analysis of X-Ray diffraction spectra has undergone significant development in the last years by applying modern computational methods as well as fundamental has been made providing large reference databases (Grazulis, Lutterotti et al. 2001). However, blind analysis of X-ray diffraction spectra without any advance information is very difficult and may result in ambiguous results (Madsen, Scarlett et al. 2001, 2002), especially if the sample consists of several, even if very few, phases, and especially when the phases of particular interest constitute only a minor part of the composition. Results of X-ray fluorescence analysis are therefore valuable complementary information for the analysis of X-ray diffraction spectra.

Figure 1. XRD spectrum, acquisition time 30 min. Aluminium cast facility, sampling duration 12 h, daytime, mass on filter 16 mg.

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