EDXRF characterisation of elemental contents in PM2.5 in a medium-sized Swedish city dominated by a modern waste incineration plant[†]

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Energy dispersive x-ray fluorescence (EDXRF) analysis of airborne particles has previously been shown to be a powerful technique for identifying key elements or elemental ratios for identification of important sources of air pollution. In the present work the technique was used for assignment of major sources of aerosol particles (PM2.5) in a medium sized Swedish city in southwestern Sweden, in which a new incinerator of household and industrial waste had recently been installed. Data on particle mass and black carbon contents in PM2.5 were also recorded together with SO_2 and NO_2 during the same study period. In spite of the small data set it was possible to identify five major sources for collected PM2.5, namely, waste incineration together with other local sources, oil incineration, biomass burning, long-distance transport and traffic emissions. Major characteristic elements for the respective sources were identified from regression analysis of the data and from information obtained in previous studies. A receptor model based on the use of trace observations was used for quantitative calculation of the source contribution to PM2.5. The relative strength of the identified sources was seen to change when the variables included in the analysis were varied in number and character, although the same sources remained. It must be noted that the quantitative contribution from the different sources may be treated only as informative at present, since the number of observations are small compared to the number of variables. Copyright © 2007 John Wiley & Sons, Ltd.

INTRODUCTION

Significant changes have occurred during the last few years regarding the attitudes towards waste and waste disposal in the EU and many other countries. The public at large has come to accept the ideas of waste separation and recycling of products and materials as an important means to create a sustainable society. Worldwide, the production of goods has increased seven fold since the 1950s, during which time the world population has doubled. Since a major part of the products ultimately end up as waste, the handling of waste is one of the crucial factors for future sustainability.

In Sweden, deposition of combustible waste on land-fills is prohibited from January 1, 2002. There is also a strong motivation to move further away from the dependence on fossil fuels and increase efficiency in the generation of heat and electricity. The city of Borås has declared itself as one of Sweden's sustainable cities and has for a long time been a pioneer in environmentally friendly waste handling and production of biogas. In order to solve the problems of handling the combustible domestic and industrial waste and at the same time decrease the dependence on fossil fuels, the city of Borås has made substantial investments recently in new technology for classification and incineration of waste in its new incinerators (bubbling fluidised bed) at the district heating plant. It has been designed for research activities on efficiency, emission control and economy and thus gives unique opportunities to conduct full-scale experiments in long-term studies under controlled conditions.[1,2]

In recent years, a large number of scientific reports on waste incineration have been published, many of which concern system studies and properties of the ashes.[3–8] Receptor modelling from waste incineration is less frequent although evidence of environmental effects is obtained as part of statistical treatment and principal component analysis (PCA) of ambient data.[9] Since incineration plants are often located in close proximity to populated areas, it is important to evaluate the impact of the activity and the additional health hazards involved. In the present work, a study has been made on the ambient air quality in the city of Borås with the aim of identifying the characteristics of aerosol particles (PM2.5) due to waste incineration. Ambient air is, however, a complex mixture of gases and particles. Some of the major polluting gases,

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for example NO_2 and SO_2, are continuously monitored in the city of Borås by the Environmental Protection Department. However, suspended particulate matter is not routinely monitored or analysed by the local health authorities.

Health effects due to small aerosol particles have been studied extensively during the last ten years,[10-13] and so far, there does not seem to be any evidence of a 'threshold' or any 'safe' level. Estimates of the number of excess deaths on a global scale due to particle inhalation have been made, and they amount to about 2 million/year and 370 000 per year within the EU.[14] The health effects are not limited to lung injuries. They also include cardiovascular diseases and cancers.

EXPERIMENTAL

Sampling location

The city of Borås with approximately 100 000 inhabitants is located in the southwestern part of Sweden. It is characterised by small and medium-size enterprises (SME). Since the aim of the project was to study the PM2.5 contribution from the waste incineration plant and other major sources to the ambient air, the place of monitoring particles was chosen to represent the general air that the citizens are exposed to. Therefore, it is not located in close proximity to any of the major activities, for example large roads. The position of the measurement location in the city is shown in Fig. 1.

On-line sampling of aerosols and meteorological data

Since the summer of 2004, the University of Borås has operated a tapered element oscillating microbalance (TEOM) instrument, which is set to determine the mass concentrations of aerosol particles of diameters $< 2.5 \mu m$. The instrument is positioned in the central part of the city approximately 25 m

Figure 1. Map of Borås, showing the sampling position and the waste incineration plant. The distance between the two places is about 900 m.

above street level and is not in close proximity to any strong local source.

The TEOM instrument (TEOM particulate mass monitor, Series 1400 Sensor Unit, Rupprecht & Patashnick Inc., USA) collects particles on a Teflon coated glass fibre filter heated to 50 °C. The filter is attached to the tip of a tapered, hollow, oscillating rod. The change in the oscillation frequency is used to make a direct measurement of the accumulation of mass on the filter over time. The TEOM is equipped with a PM10 inlet followed by a PM2.5 inlet (Rupprecht & Patashnick Inc) and has a flow rate of 1.0 m^3/h of which 3.0 l/min pass through the filter.

The Environmental Protection Department of Borås operates a differential optical absorption spectroscopy (DOAS) system at about 200 m distance from the aerosol station which measures NO_2, SO_2, benzene, toluene and xylene with one hour time resolution at approximately the same height.

In a previous study[15], it was found that meteorological variables (e. g. wind speed, wind direction, temperature and humidity) exert a strong influence on the particle concentrations. Results from this study showed that there was a dramatic decrease in particle concentrations when the wind velocity increased from below one to only a few m/s.

Large variations of aerosol particles as well as of ambient gases throughout the day and night can be expected from the daily activities of the city population. Changes in daily averages are not equally dramatic, but can still expose a variation of a factor of 10 between a 'clean' and a 'dirty' day. These variations are largely due to meteorological factors like precipitation, mixing height, inversion and air mass pathways across 'clean' or polluted regions.

Sampling of PM2.5 on filters

In order to study the mass, elemental and black carbon contents of the aerosol particles, a campaign with sampling of aerosol on filters was conducted from the 27th of July to the 23rd of August, 2005, in parallel with the on-line monitoring of particle mass, gases and meteorological data.

The filter sampler used for the PM2.5 aerosol sampling was an impactor manufactured by IVL Swedish Environmental Research Institute Ltd. A sketch of the sampler is shown in Fig. 2. The sampler is mounted inside an upside down plastic bucket that protects the inlet from both rain and from direct deposition of particles from the air.

The sampler in the present work has a flow rate of 1 m^3 h^{-1}. The PM 2.5 particles are collected on thin Teflon filters of 25 mm diameter with pore size of 3.0 μm (TEFLO™ R2PI025, Pall Corporation, Ann Arbor, MI, USA). These thin, high purity filters are suitable for analysis with energy dispersive x-ray fluorescence (EDXRF) technique and have a high particle retention efficiency.[16,17] The filters used in this study were selected from a batch, out of which a representative number had been pre-analysed for trace elements before sampling and found to have negligible concentrations of the studied elements. The Teflon filters were changed manually every 24 h during the sampling period.

Before and after exposure, the filters were weighed after 5 days of moisture equilibration in desiccators, using a microgram sensitive Sartorius balance at room temperature.
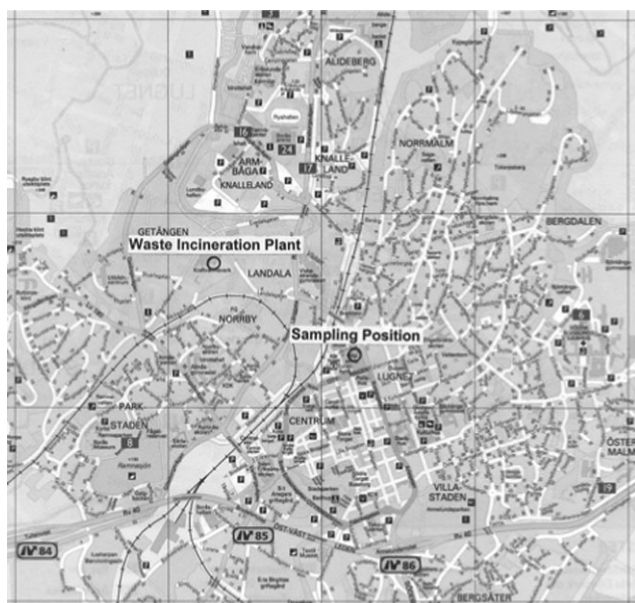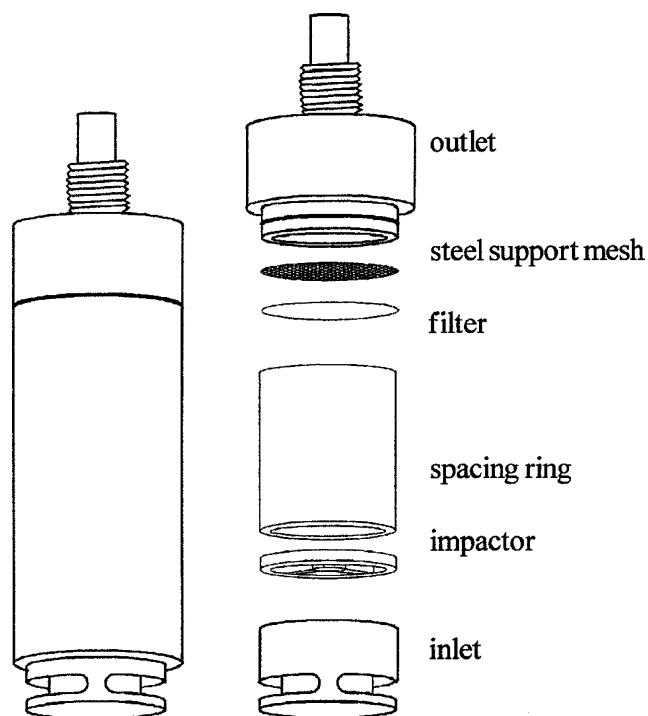
Figure 2. Schematic diagram of the IVL PM2.5 sampler. The filter diameter is 25 mm and the sampler height is 10 cm.

Thus, there was additional information on the aerosol mass during the campaign besides the information obtained by the TEOM instrument.

Analytical techniques applied to aerosol filters
Analysis of black carbon
Concentrations of black carbon (BC) can be calculated by measuring the optical absorption of the sampled aerosol on a filter with a reflectometer. An instrument from Eberline Instruments GmbH, Erlangen, was used.[18] In a previous study, it was shown that glass fibre filters, as recommended by the manufacturer, will give the same results as sampling on Teflon filters, provided that the Teflon filters are supported by a white backing during the BC measurements.[18]

The EDXRF spectrometer
The EDXRF spectrometer at the Department of Natural Sciences, University of Copenhagen was used in the present study.[19] The spectrometer is a compact, versatile and sensitive unit, using a high power Mo x-ray tube. The primary beam is monochromatised by a highly oriented pyrolitic graphite (HOPG) crystal and the detector is a Peltier cooled Si(Li) detector. The detector has an active area of 20 mm^2, FWHM at Mn Kα of 146 eV. The x-ray tube was operated at a voltage of 40 kV and a current of 40 mA in the measurements. The live time of each spectrum was 2000 s. Since the irradiation chamber of the spectrometer is evacuated, elements from Al and heavier can be detected, analysed and quantified. Minimum detection limits (DLs) for the spectrometer are shown in Table 1.

In order to calculate the concentrations of the different elements on the filters, the spectrometer was calibrated using

Table 1. Minimum detection limits (DL) for particulate matter on Teflon filters with EDXRF technique at the Department of Natural Sciences, University of Copenhagen, Copenhagen, Denmark

Element	DL[a] ng/cm^2	DL[b] ng/m^3
Si	84.0	11.0
P	50.1	6.6
S	31.9	4.2
K	7.7	1.0
Ca	4.3	0.56
Ti	2.4	0.31
V	1.8	0.24
Cr	1.3	0.17
Mn	1.2	0.16
Fe	1.0	0.13
Ni	0.9	0.12
Cu	0.9	0.12
Zn	0.6	0.08
As	0.4	0.05
Se	0.4	0.05
Br	0.3	0.04
Pb	0.7	0.09

[a] DL is calculated as 3 times the square root of background concentration (3σ). Mo Ka:17.44 keV, V = 40 kV, I = 40 mA, collection time 2000 s.
[b] DL for particle concentrations is calculated for a sampling of 24 m^3.

thin film reference material from NIST (NBS SRM 1832). The x-ray fluorescence spectra were quantitatively analysed by the use of a fundamental parameter programme.[20]

PRINCIPAL COMPONENT ANALYSIS (PCA) AND MODELLING

Theory
Statistical methods are commonly used for identification of the relative importance of different sources.[9,15,18,21–23] Input data for source assignments are chemical species, analysed by many methods, for example IC, INAA, ICP-AES, GC-MS, EDXRF, PIXE, TXRF and thermooptical and light scattering methods, for total, organic and elemental carbon.

In the present approach BC, elemental concentrations and mass were used. The model by Thurston and Spengler[23] was the basis for analysing the typical species ('fingerprints'), which characterised the different sources. In addition, the SO_2 and NO_2 data together with correlations between different species were used in support of the source assignments.

The modelling of absolute source contributions is based on the receptor model approach, where the measured concentration of a particular species is the result of a linear sum of independent contributions from distinct sources. Algebraically this is formulated in the matrix equation:

$$C = P\,S \qquad (1)$$

Here, C is the data matrix of dimension (n,q), where n is the number of variables and q, the number of samples. P is the source profile matrix of dimension (n,p), where p is the number of distinct sources and S is the source contribution matrix of dimension (p,q). For the present receptor model, samples were collected daily and the variables are elemental concentrations in ng/m^3 and the mass of PM2.5 in $\mu g/m^3$.

Once the number of distinct sources is determined, P and S are derived from a PCA analysis giving the relation:

$$Z = L F \qquad (2)$$

Where a row in Z corresponds to the autoscaled variable of the same row in C:

$$z_{i,j} = (c_{i,j} - \mu_i^c)/\sigma_i^c \qquad (3)$$

μ_i^c and σ_i^c are mean and standard deviation of variable i. L and F are the loading matrix and the score matrix and may be found by traditional PCA. The problem is to rescale L and F to the physical meaningful matrices P and S. This is done in two steps: First a 'tracer' sample,[21] with sample number q+1, having all variables set equal to zero is included in the dataset, then a PCA is used to determine the score matrix F in which the rows are treated as autoscaled values of the rows in the source matrix S. Hence, using the result for the 'tracer' sample, the f values are transformed to a scaled source matrix. In the case of the PM2.5 variables the introduction of the 'tracer' sample states:

$$s_{i,q+1} = 0 \qquad (4)$$

and for the autoscaled row variable in F:

$$f_{i,j} = (s_{i,j} - \mu_i^s)/\sigma_i^s \qquad (5)$$

μ_i^s and σ_i^s are mean and standard deviation of source i and in the case of the ''tracer'' sample:

$$f_{i,q+1} = (s_{i,q+1} - \mu_i^s)/\sigma_i^s \qquad (6)$$

Combining (4) and (6) gives:

$$\mu_i^s = -f_{i,q+1} \times \sigma_i^s \qquad (7)$$

and in turn combining (5) and (7):

$$s_{i,j} = \sigma_i^s(f_{i,j} - f_{i,q+1}) \qquad (8)$$

Secondly, the knowledge of the sample mass is used in a mass balance calculation to transform the scaled score matrix into the unscaled source matrix by regression of the transformed f values on the mass-variable $c_{PM2.5}$. The source matrix PM2.5 values must be related to the experimental mass values, $c_{PM2.5}$ by the relation:

$$c_{PM2.5,j} = \Sigma_i s_{i,j} = \sigma_i^s(f_{i,j} - f_{i,q+1}) \qquad (9)$$

The coefficients σ_i^s are found by regression of $(f_{i,j} - f_{i,q+1})$ on $c_{PM2.5,j}$. The elements in the source matrix are now given by Eqn (8) and they describe the daily variation of the PM2.5

Table 2. Average concentrations of elements, BC and mass of PM2.5 particles in the city of Borås

	Mean ng/m^3	Median ng/m^3	Lowest—highest ng/m^3
Si	35.22	24.21	11.7–142.2
P	29.54	24.52	9.9–66.9
S	515.03	454.68	154.9–1139.5
K	36.23	22.98	13.6–181.0
Ca	15.58	12.12	4.7–43.1
Ti	1.01	0.70	0.3–26.4
V	1.73	1.80	0.3–3.3
Cr	0.68	0.48	0.2–1.7
Mn	0.73	0.56	0.3–2.7
Fe	26.00	24.22	10.3–72.7
Ni	0.71	0.77	0.2–1.6
Cu	1.02	0.92	0.5–2.4
Zn	4.16	3.24	1.2–14.6
As	0.60	0.51	0.2–1.3
Se	0.35	0.15	0.1–2.3
Br	1.41	1.42	0.6–2.5
Pb	0.98	0.59	0.1–2.7
BC	0.60×10^3	0.53×10^3	$(0.35–1.17) \times 10^3$
Mass	5.7×10^3	5.4×10^3	$(2.4–13) \times 10^3$

mass-variable of the source in ng/m^3. Finally the source profile matrix is calculated:

$$P = C S^T (S S^T)^{-1} \qquad (10)$$

It must be noted that in order to obtain physical meaningful results, negative values in S and L are truncated to zero before further calculations.

Calculation

Twenty-seven samples of the PM2.5 filters were analysed for 20 elements (ng/m^3), BC ($\mu g/m^3$) and particle mass of PM2.5 ($\mu g/m^3$). Also the daily mean content of NO_2 and SO_2 ($\mu g/m^3$) were measured and used in correlation analysis, but not included in the PCA modelling. Due to severe peak overlap and/or bad counting statistics, the concentrations for Al, Cl and Sr were omitted in the following calculations. Missing values were found for some of the elemental concentrations. There are different ways of treating missing values. In the present study, missing values were set equal to half the value of the DL. The correlation matrix between variables was the key for determining the number of sources.

RESULTS AND DISCUSSION

Concentrations of elements, black carbon, mass and gaseous components

Concentrations of elements, BC and mass of the PM2.5 aerosol are shown in Table 2. The median values are shown in the table because the means will generally have a large influence from extreme values during a few days, and a comparison between means and medians may give information on to what extent the values are influenced by extreme conditions. The STDs for the elemental EDXRF measurements on this instrument are in the order of about 10%.[19]
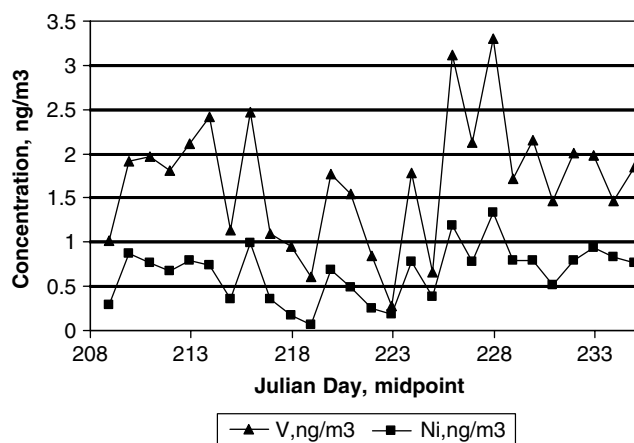
As seen from Table 2, the BC contents of PM2.5 are in the order of 10% of the mass. For coarse particles, PM(2.5–10), the contribution of BC has been found to be of the order of one percent of the total mass in this fraction.[18] The mass concentrations, on the other hand, are of similar magnitude in PM2.5 and PM(2.5–10) in Swedish urban environment.[15,24] The mean concentration of $6 \, \mu g/m^3$ for PM2.5 as listed in Table 2 is rather close to that found in other urban locations, provided that measurements are not performed in close proximity to strong sources.[15,24,25]

The median concentrations of NO_2 and SO_2 for the same period were 10.9 and $2.08 \, \mu g/m^3$ respectively.

In regression analysis of all data on elements, mass, BC and the gaseous components NO_2 and SO_2, the information was used to support the source assignments for the factors obtained in the PCA. It was noted that the correlation coefficient between V and Ni is very high, 0.94, and that the only other significant correlation coefficients are for Br and S (>0.50). Thus, it is highly likely that V and Ni have at least one common source. The close connection between the two elements is illustrated in Fig. 3 for the study period. Many metals, for example Cu and Fe, are highly correlated to the blackness (BC) of the aerosol particles. Another observation is that many of the metals have high correlations between them, again indicating that they have one or more common sources.

Source assignment from PCA modelling

In the principal component analysis several runs were made in which the number of factors were varied, and varimax as well as promax rotations were performed. However, since the pollution sources are independent of each other and because varimax gave the most consistent results when the number of factors was varied, varimax was chosen for the final source assignments.

The Scree plots from principal component analysis using different subsets of variables indicated the number of significant factors to be 4 or 5. Also a hierarchical clustering calculation based on the correlation matrix, in which the variables are observations and the correlation coefficients are variables, indicated a substructure of the dataset based on 5 factors. A dataset of 27 daily observations is small in order to precisely estimate the correlation structure for five sources and therefore stable results are not expected. Nevertheless, calculations based on the previous equations were carried out for different subsets of variables and the percent source contributions to PM2.5 were calculated. The results are shown in Tables 3 and 4.

Thus the following five main factors were identified:

– Incineration of domestic and industrial waste in the city of Borås together with other local sources, with signatures of many metals, for example Pb.
– Oil incineration from small scale oil burning and major refineries in the region. Signatures are in particular V and Ni, as already dicussed.[26]
– Biomass burning occurs in the Borås region in private houses but also in the incineration plant. Previously found signatures are those of K and the K/Zn ratio.[15,24]
– Long distance transport (LDT) to Sweden, mainly from the European continent. LDT has been proved to bring a substantial amount of sulphate into the country. The main indicator is therefore S.[27,28]
– PM2.5 in traffic usually contains a contribution from street dust, but traffic is also known to give rise to gaseous pollutants which may be transformed into particles by gas-to-particle conversion. In the present case we have utilised the positive correlation coefficients ($R > 0.5$) between NO_2 and in particular Fe and Cu as indicators of traffic. It should be noted that NO_2 has few positive correlation coefficients with other metals than the two mentioned and

Figure 3. Daily variations of V and Ni during the period of the study.

Table 3. Set of variables used in the calculations of source contributions

19 variables	Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br, Pb, BC, mass
14 variables	P, S, K, Ca, Ti, V, Mn, Fe, Ni, Cu, Zn, Br, Pb, mass
8 variables	S, K, V, Fe, Ni, Zn, Pb, mass
6 variables	S, K, V, Fe, Pb, mass

Table 4. Estimated percent contributions of PM2.5, normalised to 100%, for the different sets of variables listed in Table 3

	Waste incineration and local sources	Oil incineration	Biomass burning	Long distance transport (LDT)	Traffic emissions
19 variables	32	33	18	16	1
14 variables	28	29	9	23	12
8 variables	17	21	7	41	14
6 variables	24	11	8	51	6

the soil derived elements (Si, Ca, Ti and Mn), although the latter are at a lower level ($R = 0.3$).

Quantification of source influence by the choice of the numbers of variables

Since the measurements were conducted during a limited time period, which gave rise to a limited number of samples, the statistical analysis was also conducted by varying the representative variables. Reduction of variables gives better statistical significance, but it will have to be done considering the available knowledge of the typical fingerprints of the respective sources. In the choice of variables, knowledge from correlation coefficients and from previous work was also used.[15,16,18,27–29]

In the modelling efforts the variables in the dataset were used in the following way:

At first, all species in Table 2 were used in a common dataset. This means that the aerosol, containing both particles and gases, were analysed with respect to listed species. Since the daily variations of all species are very large due to meteorological factors (mixing height, inversion, rainfall, wind direction, wind-speed, air mass trajectory movements etc.) this analysis is highly affected by meteorological factors and the number of variables increase drastically if meteorological factors are included. This approach was however abandoned, not only because of the many variables but also because we were interested in the sources of the PM2.5 particles. These sources can be point sources, or area sources as for traffic emissions, biomass burning and oil incineration in close proximity to the sampling site or at a long distance.

Thus, we turned to the problem of finding the sources for the actual PM2.5 particles and used only the variables, dependent on the composition of the PM2.5 in the aerosol. Thus, the gases were not taken into account, and the element and BC concentrations were normalised to relative concentrations (ng/µg of mass).

In order to study the influence of the number of variables on the source contribution to the sampled PM2.5, the variables in the dataset were varied in number from 19, 14, 8 and down to 6. In the reduction of variables, the character of the main sources remained, but the relative contribution from the different sources varied significantly.

An illustration of the relative strengths of the respective sources as obtained when the number of variables is reduced is shown in Tables 3 and 4 for the different cases. The reason for the large differences for the outcome in the different approaches is not clear but will have to be studied more in detail. It is obvious that too few variables may affect the outcome, but it is also important to choose the best signatures.

CONCLUSIONS

It is satisfying that the same characteristic elements appear in the factors describing the major sources for the PM2.5 aerosol, although some crucial questions remain to be solved regarding how many and which variables should preferably be used in statistical analysis for obtaining quantitatively consistent results on source contributions. This is not a trivial

question, because there are many species not studied in the present work, especially chemical organic components. If these species are included in the databases, together with all meteorological variables, the problem of source identification would probably be too cumbersome.

One should also remember that the source strengths vary throughout the year and this is especially pronounced in Nordic countries with a big difference in heating and also work activities between the summer and winter periods. Thus, source strengths are not expected to be the same during the year and also a larger database would need to be broken down into activity-related periods. Also, for these cases it seems unrealistic to obtain a sufficient amount of statistically significant data if all possible variables are to be included in the modelling. Therefore, we find it even more relevant to look for elemental signatures is that are characteristic for the major sources. Elements have the advantage that they are not transformed in chemical reactions in the atmosphere during transport from source to receptor site.

A crude test of the modelling presented in this work can be seen in Fig. 4. In this figure the experimental particle mass is plotted each day together with the modelled mass for the case of using 8 variables as illustrated in Table 3. The results in Fig. 4 show that the PCA method can be used with some confidence, although details in the roles played by the different variables will have to be studied further.

Particle concentrations in winter and in summer only differ slightly: During winter the particle mass concentration as measured by the TEOM is 8.7 ± 4.5 µg/m^3 and in summer 7.7 ± 2.6 µg/m^3. From this relatively small difference we draw the conclusion that, even if our measuring campaign covers a relatively short period during the summer, the source assignments should be valid for other parts of the year as well, although the relative contributions from the respective sources will vary depending on season. The main difference in activities between the summer and winter half of the year is that during winter, heating is needed. In Boras, the additional district heating is then provided through biomass burning. Also, many single-family homes are heated with biomass in the form of wood pellets.
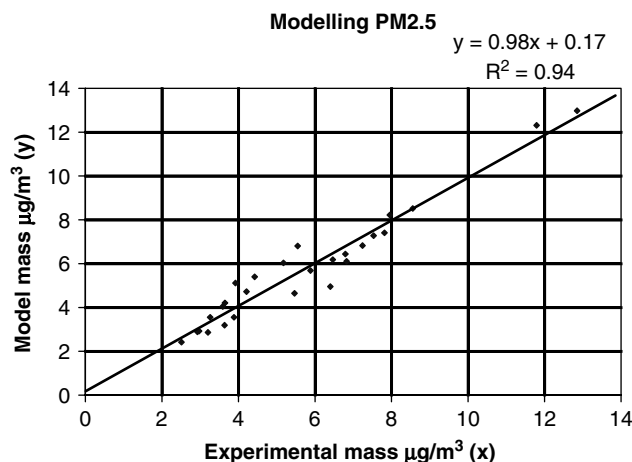

Modelling PM2.5

$y = 0.98x + 0.17$
$R^2 = 0.94$

Figure 4. Comparison between model mass and experimental particle mass.

In our future work, however, more experimental data will be collected and analysed for making a more detailed analysis of the relative contribution to the levels of PM2.5 when the incinerable waste is varied in composition.

Acknowledgements

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