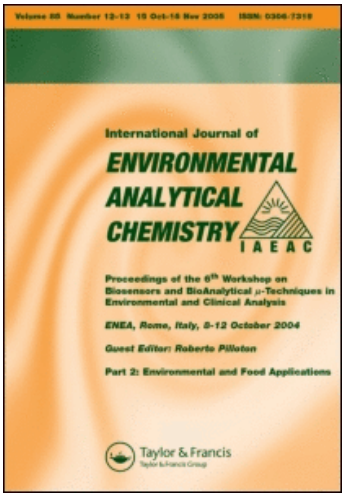

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Chan, Y. C. , McTainsh, G. H. , Vowles, P. D. , Simpson, R. W. , Cohen, D. D. and Bailey, G. M.(1997) 'Application of Pixe Analysis to Urban Aerosol Samples Collected on Whatman 41 Filters', International Journal of Environmental Analytical Chemistry, 68: 1, 33 — 45
To link to this Article: DOI: 10.1080/03067319708030478
URL: http://dx.doi.org/10.1080/03067319708030478

Intern. J. Environ. Anal. Chem., Vol. 68, pp. 33-45
Reprints available directly from the publisher
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APPLICATION OF PIXE ANALYSIS TO URBAN AEROSOL SAMPLES COLLECTED ON WHATMAN 41 FILTERS

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(Received 5 December 1996; In final form 20 March 1997)

The applicability of the Proton Induced X-Ray Emission (PIXE) technique to multi-elemental analysis of PM_{10} aerosol samples collected on Whatman 41 cellulose fibrous filters is investigated using concurrent samples collected on teflon membrane filters as references. The elemental levels measured on the two types of filter samples are highly correlated and the ratio of the elemental levels is related to the deepness of penetration of particles inside the filter, which is calculated from the Fan Filter Model. Therefore, PIXE analysis can be used to describe urban aerosol samples collected on low impurity level porous filters such as Whatman 41.

Keywords: Urban aerosols, cellulose filters, multi-elemental analysis, Proton Induced X-ray Emission, Fan Filter model.

INTRODUCTION

Urban aerosol samples are collected on high and low volume air samplers using a variety of filters, such as membrane filters (e.g. [1]), glass fibre filters (e.g. [2]) and Whatman paper cellulose filters (e.g. [3]). The type of filter used depends upon: the sampler employed, the chemical species to be analysed and the budget of project. Whatman 41 cellulose filters are relatively cheap, mechanically strong, have low elemental blank levels compared with glass fibre filters [4], and so are widely used in air monitoring networks (e.g. [3,5,6]), despite the criticisms that they have rela-

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tively low collection efficiency for fine particles [7–9] and tend to absorb gaseous species under high relative humidity conditions [10].

The Proton Induced X-Ray Emission (PIXE) technique has been widely used as a multi-elemental and non-destructive means for the analysis of aerosol samples collected on membrane filters (e.g. [1,11,12]). On membrane filters, the aerosols distribute as a thin layer of particles, therefore the degree of attenuation of proton beam energy and x-ray emission intensity by the teflon material and aerosols is negligible [13]. However, for fibrous filters such as Whatman 41 cellulose filters, the particles can penetrate deep into the fibre matrix. When PIXE analysis is applied to these filters, the thickness of the filters and aerosols results in weakened X-ray emission intensity leading to underestimation of elemental levels. For this reason, the PIXE technique has seldom been used for elemental analysis of aerosol samples collected on fibrous filters.

X-ray fluorescence (XRF) analysis, another multi-elemental analysis technique, has been applied to the analysis of aerosol samples collected on Whatman 41 filters by Rose, et. al. [5] in a study of total suspended particulate (TSP) matter in Brisbane, Australia. In that study, a standard dust-fall material, NBS urban particulate 1648, was re-suspended in freon and recollected on Whatman 41 filters and then these filters were used as reference to develop the calibration formulae between the observed X-ray emission intensities and the elemental levels in filters. However, since NBS 1648 was obtained from dust-fall samples [14], its particle size distribution characteristic would be quite different from those of the suspended particulate matter. Hence the deepness of penetration inside the filter and the response factor of X-ray signal to the loading of particles on the filters could also be different.

In the present study, we examined the applicability of the PIXE technique to the analysis of aerosol samples collected on Whatman 41 filters using concurrent samples collected on teflon membrane filters as references. The measured elemental levels are used to develop the calibration formulae by regression. The calibration formulae are then assessed by the correlation between the measured levels, r, and the squared multiple correlation value of the regression formulae, R^2.

The ratio of the measured level of a certain element in the teflon filter samples to that in the Whatman 41 filter samples is used to indicate the deepness of penetration of particles inside the Whatman 41 filter. If this ratio is close to one, we assume that the particles containing that element are distributing mainly on the surface of the Whatman 41 filter. On the other hand, ratios greater than one indicate deeper penetration. The deepness of penetration of particles as indicated by the regression formulae is compared with that calculated by using a theoretical filter model. The Fan Filter Model [15] is used in this study to calculate the theoretical penetration of aerosol particles inside the fibrous filter as function of particle size.

EXPERIMENTAL METHODS

Sampling

Twenty-four PM_{10} samples (aerodynamic diameter < 10 μm) were collected on Whatman 41 filters with a high volume sampler, twelve on different days throughout 1994 and twelve in July 1995, at Griffith University, Brisbane, Australia. Twenty-four pairs of concurrent dichotomous samples (aerodynamic diameter < 2.5 μm and between 2.5 and 10 μm) were collected on 2 μm pore-size teflon membrane filters with a dichotomous sampler at the same site. The elemental levels of the fine and coarse fractions of the dichotomous samples were added together and used as reference PM_{10} elemental levels.

PIXE analysis of samples

The samples were analysed by PIXE at the Lucas Heights Research Laboratories (LHRL), Sydney, Australia. The details of this technique are described by ERDC [1] and Cohen [13]. PIXE analysis is used to determine the levels of 11 elements in this study, namely Al, Si, S, Cl, K, Ca, Ti, Mn, Fe, Zn and Pb. The elemental composition was calibrated using thin standard material and/or pre-calibrated mixed standards. In order to investigate the effect of thickness of Whatman 41 filter on the attenuation of proton beam energy and X-ray emission intensity,

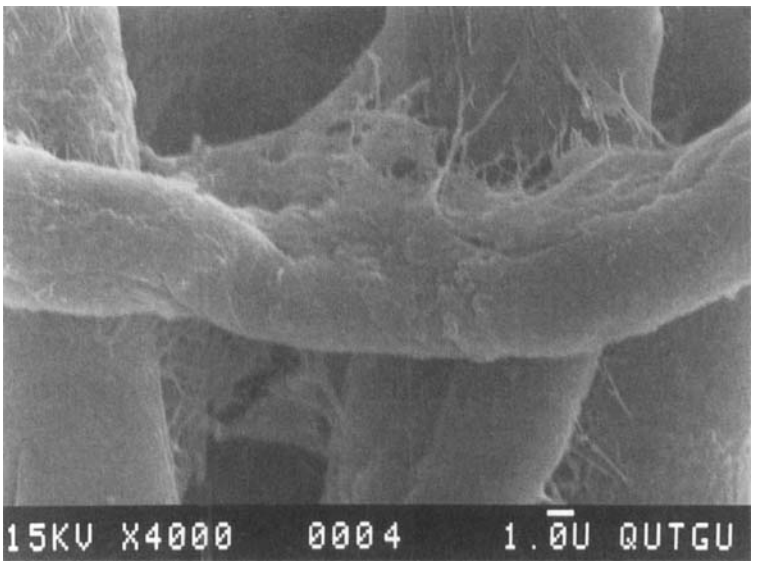

FIGURE 1 SEM micrograph of a blank Whatman 41 filter paper

and the penetration of particles inside the filter matrix, the twelve Whatman 41 filter samples collected in 1995 were measured both from the front and the back. All the results were blank corrected.

SEM analysis of blank filters

The Fan Filter Model [15] used in this study requires input of the diameter of Whatman 41 fibres. These values were found by examining a blank filter under a JEOL 840 scanning electron microscope. A 16 mm^2 portion of the filter was made conductive by coating with graphite and then examined under 4000X magnification (Figure 1). The diameters of more than 50 fibres were measured and the average diameter of the fibres is 9.36 μm with a normalised variance = 0.276 (variance of diameter divided by the square of average diameter) [16].

MODELLING THE PENETRATION OF PARTICLES IN WHATMAN 41 FILTERS

Removal of particles by fibrous filters

Aerosol particles are removed by fibrous filters through several mechanisms [9] but only two are important for suspended particulate matter: the interception and diffusion mechanisms [17]. Interception refers to the process in which large particles in an air stream collide with and are caught by the fibres. Diffusion refers to the process in which small particles are brought to the fibres through Brownian movement and caught by the fibres.

Since only diffusion and interception are considered as the main removal mechanisms (other mechanisms such as impaction are assumed to play only a minor role [15]), the penetration of particles depends solely on the size of the particles rather than on the other aerodynamic properties such as density and shape.

The Fan Filter Model

There have been several theoretical and experimental studies of the filtration of aerosols by fibrous filters since the 1950s (e.g.[18,19]) and several models have been developed. Among these models the simple Fan Model has been shown to be able to predict the penetration of particles accurately (e.g. [20,21]). In the Fan Filter Model, the fibre matrix is treated as an array of cylindrical fibres lying tangentially to the air flow. Also, the spatial arrangement of fibres changes from one layer to another. The air flow around each fibre is calculated by solving the Stokes flow equations with consideration of the influence from nearby fibres

[19]. The choice of boundary conditions used when solving the differential equations results in slightly different forms of formulae all giving similar results [19]. The calculation procedures proposed by Kirsch and Zhulanov [15] are used in this study.

The filter and sampling condition parameters required in the calculations include:

diameter of fibre = 9.36 μm
normalised variance of diameter of fibre = 0.276
fraction solid in the filter = 0.138 [22]
air velocity = 47 cm-sec^{-1}
pressure drop across filter = 38 mmHg [23]
total thickness of filter = 220 μm [23]

By following the calculation procedures described in Kirsch and Zhulanov [15], the proportion of particles with size d_p which have penetrated at depth h in the Whatman 41 filter can be calculated. To validate the calculations, we performed a trial calculation for the fraction penetration of 0.3 μm particles through the full thickness of the filter at a gas velocity of 53 cm-sec^{-1}. The fraction penetration thus obtained is 0.18. This value is close to the value of 0.16 by Lockhart, et al. [24] in an experimental test using artificially generated 0.3 μm DOP (dioctylphthalate) particles.

RESULTS AND DISCUSSIONS

Overall elemental concentrations in Whatman and teflon filter samples

The measured elemental levels by PIXE in the reference filter (teflon) samples were regressed against those in the Whatman 41 filter samples. Those levels which are below the detection limit of measurement are excluded from the regression. Since all regressions have higher R^2 value for the without-intercept model than for the with-intercept model, the former model is adopted in subsequent analysis. These regression formulae are shown in Table I. As shown in Table I, the measured elemental levels in the two types of filters are highly correlated ($P(r=0) < 0.20$). As the regression formulae have high R^2 values ($P<0.0001$), they can be used for the calibration of measured elemental levels in Whatman 41 filter samples to their equivalent levels in teflon filter samples. However, the coefficients in the regression formulae derived from the twelve 1994 samples are rather different from those derived from the twelve 1995 (front) samples for elements such as Al and Zn. This is because these formulae are dependent on the ambient particle size distribution of particles, which varies with space and time. Formulae must therefore be developed for each monitoring site and reviewed seasonally.

TABLE I Results of the measured elemental levels in teflon filter samples in relation to levels in Whatman 41 filter samples, plus a comparison of measured levels on the front and back of the 1995 sample filters

	94' sample measured from the front					*95' sample measured from the front*					*95' sample measured from the back*				
			regression results[3]					*regression results*					*regression results*		
	ave[1]	*n*	*slope*	*r*	R^2	*ave*	*n*	*slope*	*r*	R^2	*ave*	*n*	*slope*	*r*	R^2
Al	0.23	4	1.95	0.93	0.92	0.17	10	1.37	0.94	0.92	<DL[2]	-	-	-	-
Si	0.64	6	1.49	0.60	0.89	0.62	12	1.20	0.91	0.94	<DL	-	-	-	-
S	0.19	12	1.97	0.84	0.92	0.16	12	1.98	0.65	0.90	0.05	12	5.64	0.81	0.86
Cl	0.91	12	1.61	0.79	0.88	0.48	12	1.44	0.80	0.90	0.05	12	1.64	0.88	0.91
K	0.09	11	1.28	0.89	0.94	0.10	12	1.23	0.62	0.90	0.05	12	2.58	0.60	0.91
Ca	0.42	9	0.81	0.96	0.97	0.25	12	0.93	0.77	0.93	0.13	12	2.39	0.78	0.97
Ti	0.06	3	0.72	0.98	0.93	0.04	12	0.93	0.84	0.94	0.02	12	2.18	0.70	0.87
Mn	0.02	4	1.10	0.81	0.62	0.01	12	1.0	0.63	0.93	0.01	6	1.86	–0.18	0.93
Fe	0.26	11	1.06	0.61	0.78	0.34	12	0.86	0.80	0.93	0.15	12	1.92	0.80	0.94
Zn	0.03	7	1.80	0.55	0.80	0.03	12	1.03	0.77	0.90	0.02	12	1.58	0.83	0.93
Pb	<DL	-	-	-	-	0.07	7	1.30	0.68	0.91	0.04	5	2.52	0.90	0.94

remarks:
[1] average measured elemental concentrations in the Whatman 41 samples, in $\mu g\text{-}m^{-3}$
[2] below detection limit
[3] measured level in teflon sample = slope x measured level in Whatman 41 sample
n = number of samples with level above detection limit
r = correlation coefficient, R^2 = squared multiple correlation

Due to loss in proton energy and attenuation in X-ray intensity in the filter matrix, the elemental levels of the Whatman 41 samples measured from the back of the filters are much lower than those measured from the front of the filters (as shown by the lower average measured levels and larger slope values in the regression formulae in Table I). However, the measured levels from the back of the Whatman 41 filter samples are still highly correlated to those in the teflon filter samples for most of the elements (Table I). This indicates that relationship of measured elemental levels in the Whatman 41 filters with those in the teflon filters persists at different penetration depths within the Whatman 41 filters.

Depth of penetration of particles into Whatman 41 filters

The slope values in the regression formulae can be an indication of the penetration depth of particles inside the fibre matrix. Those elements with slopes larger than one (elemental levels being underestimated in the whatman 41 filter samples due to loss in proton energy and attenuation in X-ray intensity) are present in particles penetrating deep inside the filters. The energy loss, ΔE, of a proton travelling through a distance of x cm in a filter material with density σ g-cm^{-3}, is

$$\Delta E = S(E)\sigma x$$

where S(E) is called the stopping power. From our measurements, S(E) is approximately 125 MeV-cm^2-g^{-1} for an initial proton energy of 2.6 MeV that we employed in this work. Whatman 41 filters have an average density of 0.39 g-cm^{-3} [22]. Therefore travel distances of 20 μm, 100 μm and 220 μm (full thickness of filter) in the Whatman 41 filter will result in energy losses in proton of approximately 100 keV, 500 keV and 1.1 MeV, respectively. Usually an energy loss of 500 keV, or more, is considered as substantial, therefore a substantial loss of proton energy would occur if the particles penetrate to more than half the thickness of the fibre matrix.

The attenuation in X-ray intensity of elements is dependent on the thickness of filter material and energy of characteristic X-ray line of the element. Table II shows our measured % attenuation in X-ray intensity and the corresponding value of slope in the regression formulae, for X-ray lines of different energies and filter material of different thickness, assuming uniform distribution of particles in the fibre matrix.

Comparing the expected slope values in Table II with the measured slope values of each element measured from the front of the 1995 filter samples in Table I, particles containing the elements Al, Si, Mn and Zn are probably distributing within the top 20 μm of thickness of the filter. Particles containing K probably penetrate up to half of the thickness of the filter, while particles containing S and Pb probably penetrate to more than half of the thickness of the filter.

TABLE II Percentage attenuation in X-ray intensity and the corresponding expected value of slope in the regression formulae

thickness of fibre matrix (corresponding deepness of penetration in Whatman 41 filters)		*0.8 mg-cm^{-3} (20 μm)*	*4.0 mg-cm^{-3} (100 μm)*	*8.5 mg-cm^{-3} (220 μm*
energy of characteristic X-ray line	*elements*	*% attenuation in X-ray intensity (corresponding expected value of slope)*		
1–2 keV	Al, Si	>30–70% (>1.43–3.33)	>70–92% (>3.33–12.5)	>92% (>12.5)
2–3 keV	S, Cl	>10–30% (>1.11–1.43)	>30–35% (>1.43–1.54)	>35% (>1.54)
3–4 keV	K, Ca	>4–15% (>1.04–1.18)	>15–30% (>1.18–1.43)	>30% (>1.43)
>4 keV	Ti, Mn, Fe, Zn, Pb	>2–7% (>1.02–1.08)	>7–15% (>1.08–1.18)	>15% (>1.18)

Elements Ca, Ti and Fe have slopes smaller than one (Table I), which means their measured levels in the reference filter samples were even lower than that in the Whatman 41 filter samples. This may be due to the loss of coarse particles from the teflon filter either by bouncing off during sampling [25] or during transportation prior to analysis [26]. The latter possibility is, however, unlikely in the present study because reweighing of the teflon filters after analysis did not reveal any substantial loss of aerosol mass. For the first possibility, Loeffler [25] has shown that the adhesion of particles on teflon filters decreases for particles larger than 2.75 μm, partly due to the development of electrostatic charges on teflon filters. Therefore crustal matter particles containing Ca, Ti and Fe, which are mainly coarse particles (e.g. [27]), may bounce off during sampling. Further supporting evidence is available from our aerosol study in Brisbane, in which we monitored PM_{10} levels by dichotomous sampling with teflon filters and by high volume sampling with GF/A glass fibre filters. Regression of the results from our monitoring data show that the PM_{10} masses collected on teflon filters were slightly lower than those collected concurrently on GF/A filters:

$$PM_{10}\text{ (teflon filter)} = 0.978\ PM_{10}\text{ (GF/A filter)}\ (n = 219,\ r = 0.67,\ R^2 = 0.41)$$

Element Cl shows similar m values in regression formulae for measurements either from the front or from the back of the Whatman 41 filters (Table I). This indicates that Cl-particles, mainly from seasalt NaCl, distribute nearly uniformly through the filters. This observation is also supported by evidence from our Brisbane aerosol study, where we examined aerosol samples collected on Whatman 41 and on GF/A glass fibre filters with a scanning electron microscope. We consistently found coarse NaCl particles in the GF/A filter samples but only very few NaCl particles in the Whatman 41 filter samples. This might be because the

hygroscopic characteristic of the Whatman 41 filters caused the NaCl particles to dissolve, spread and recrystallised as tiny particles throughout the filters.

Comparison of particle penetration results with Fan Filter Model

Estimates of the proportion of particles penetrating the Whatman 41 filters according to the Fan Model [15] are shown in Figure 2. The particles which penetrate furthest into the filters are 0.25 μm (Figure 2). Also, up to 18% of particles of this size are not captured at all by the filters. Particles smaller than this size are removed by the filter through the diffusion mechanism while those larger than this size are removed by the filter through the interception mechanism (e.g. [17]). If we arbitrarily take the top 20 μm as the 'surface' of a filter, then particles smaller than 0.006 μm and particles larger than 4 μm are completely trapped on the filter surface (Figure 2). An SEM micrograph of a cross-sectional portion of an actual sample filter is shown in Figure 3 which confirms that most of the 'visible' particles (larger than 1 μm) are trapped on the surface of the filter.

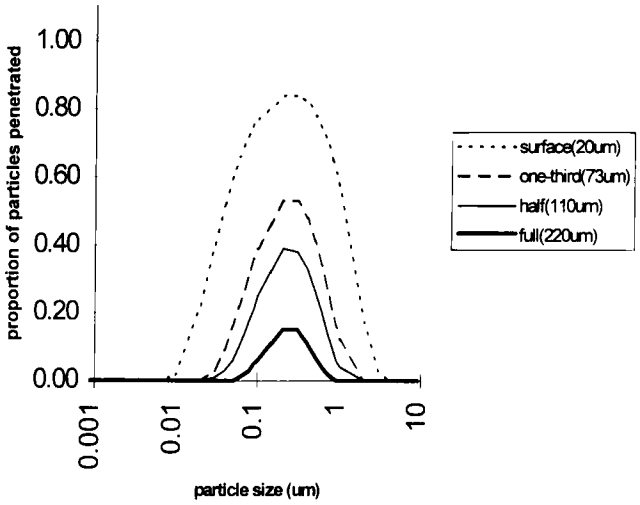

FIGURE 2 Penetration of particles in Whatman 41 filters

If the particle-size distribution of a certain element is known, we could therefore incorporate this size distribution function into the Fan Model. Then the overall penetration of particles containing the element at any depth inside the filter could be calculated by integrating the proportion penetration function over the whole particle size range. However, the particle-size distribution of elements

varies considerably through time and space. Also particle-size fractionation is usually by monitoring devices such as cascade impactors and so the sizes are actually aerodynamic size rather than actual size (e.g. [27]). Moreover, information of particle-size distribution of submicron particles are seldom available.

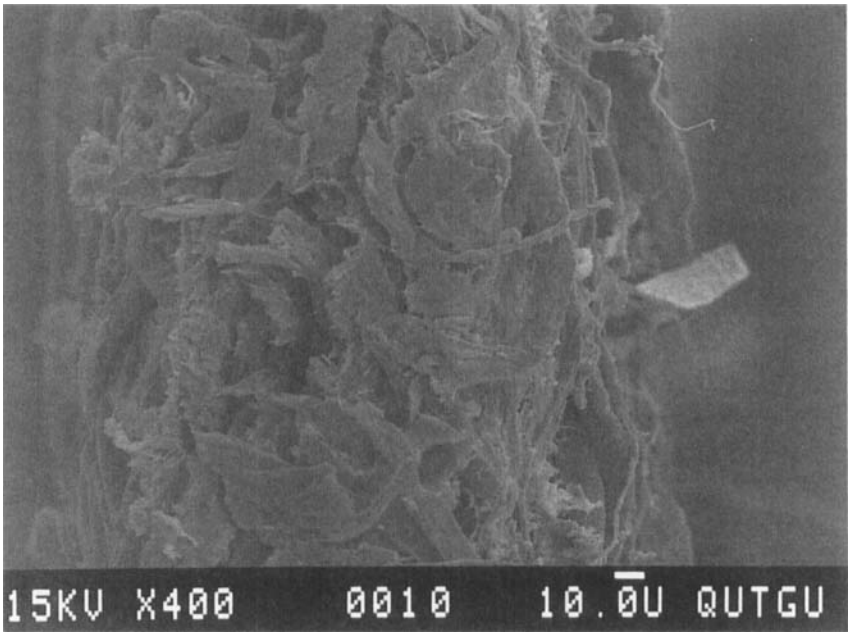

FIGURE 3 SEM micrograph of a cross-sectional portion of an actual sample filter under 400X magnification. The collecting surface of the filter is on the right hand side of the micrograph.

Despite the limited information on particle size distributions of individual elements, we can still qualitatively compare the deepness of particle penetration from the experimental results to the theoretical penetration from Fan Model, by using the particle size information available in literature for some elements with distinctive mass median diameters. Lee, et. al. [27] in a Cincinnati aerosol study found the mass median diameters (MMD, aerodynamic diameter) of urban Pb, sulphate and Fe particles as 0.18, 0.42 and 3.7 μm, respectively. Since the density of particles is usually greater than 1 g-cm^{-3} [28], their actual size MMDs would be smaller than the above values. Therefore sulphate and lead particles will have median size closest to the maximum penetration size 0.25 μm, relating to the largest penetration found in this work (Table I and Table II). Fe particles have a large MMD and may bounce off the reference teflon filters, leading to an observed m value which was less than one (Table I).

Detection limit and precision of analysis upon samples collected on Whatman 41 filters

Finally, we compare the results of PIXE analyses of Whatman 41 filter samples with teflon filter samples as shown in Table III. These data are based on the analysis of more than 100 Whatman 41 filter PM_{10} and more than 270 pairs of teflon filter dichotomous samples collected in Brisbane since August 1992. As Table III shows, the elemental characteristics of the aerosols on the two types of filters are generally comparable, although Whatman 41 filters have higher blank levels for chlorine and sulphur. Also, the detection limits (in $\mu g\text{-}m^{-3}$ equivalent) of Whatman 41 filters are higher than those of teflon filters. This is because approximately 1680 m^3 of air was passed through a 414 cm^2 of filter area for each Whatman 41 filter sample, while approximately 24 m^3 of air was passed through a 2.02 cm^2 of filter area for each teflon filter sample, which resulted in higher aerosol density on the teflon filters than on the Whatman 41 filters.

TABLE III Comparison of results of PIXE analysis for PM_{10} samples collected on teflon filters and Whatman 41 filters

element	*teflon filters*			*Whatman 41 filters*		
	DL[1]	*blank*	*RSD%*[2]	*DL*	*blank*	*RSD%*
Al	0.020	<DL[3]	6	0.063	<DL	12
Ca	0.0064	<DL	3	0.056	<DL	7
Cl	0.0079	0.0086	4	0.026	0.105	8
Fe	0.0046	<DL	4	0.022	<DL	6
K	0.0062	<DL	4	0.0091	<DL	6
Mn	0.0027	<DL	14	0.0039	<DL	12
S	0.008	0.0157	3	0.017	0.07	10
Si	0.020	0.035	3	0.091	<DL	7
Ti	0.0048	<DL	5	0.071	<DL	7
Zn	0.0034	<DL	8	0.0047	<DL	9
Pb	0.023	<DL	5	0.035	<DL	16

remarks:
[1] detection limit, in $\mu g\text{-}m^{-3}$ equivalent
[2] relative standard deviation, in %
[3] below detection limit

CONCLUSIONS

PIXE analysis can be used to describe urban aerosol samples collected on low impurity level porous filters such as Whatman 41, if a sufficient amount of concurrent aerosol samples collected on membrane filters are used as a reference. Since the regression formulae which describe these relationships are dependent on the ambient particle-size distribution of particles, which varies through space and time, it will be necessary to develop these regression formulae for each monitoring site and review them seasonally.

Acknowledgements

We acknowledge the support of the staff of the Analytical Electron Microscope Centre at Queensland University of Technology, Brisbane. The PIXE analyses were funded by the Australian Institute of Nuclear Science and Engineering (AINSE) Grants 94/124 and 95/097.

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