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**Apiezon L-type grease coating of 8- μ m Nuclepore Polycarbonate coarse filters
used in GENT aerosol sampling units**

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Apiezon L-type grease coating of 8- μm Nuclepore Polycarbonate coarse filters used in GENT aerosol sampling units

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1. Introduction

Coarse stage Nuclepore filters used in GENT aerosol sampling units were coated with a thin film of Apiezon L-type grease in order to minimise the loss of particles on the filters due to 'bounce off.' Originally Freon was used as the solvent to dissolve the Apiezon grease in the coating process. Because of the environmental impact, Freon was discontinued and replaced with Fluorinert. Apiezon grease did not dissolve freely in the Fluorinert and it was replaced by Fomblin grease in 1995. This increased production costs and coated Nuclepore coarse filters ceased production in 2004.

Users were concerned that their stocks of coated Nuclepore filters were diminishing and as no alternative supply was available the feasibility of coating the coarse filters in house needed to be investigated. This report describes experiments conducted at ANSTO and the results obtained during the investigation of the coating process under laboratory conditions. Apiezon grease and Toluene were used in this process. Experiments were based on the procedure proposed by Prof P. Hopke and Dr A. Markwitz [1] with modification to this technique to increase the uniformity of the grease layer across the filter surface and to achieve approximately 100 μg of Apiezon grease per filter.

2. Method

Five grams of Apiezon L-type grease were dissolved in 200ml Toluene reagent (Anala R, n-Hexane $\text{CH}_3(\text{CH}_2)_4\text{CH}_3$), then well shaken and let to stand in a tightly sealed bottle for 24 hours. One part of the mixture was then further diluted with two parts of Toluene. This solution was called the "basic solution". Several dilutions were prepared and a number of techniques tried before the desired outcome was achieved. This was achieved by adding 3 parts of Toluene to the "basic solution", which was then called "coating mixture".

Individual filters were vertically dipped into the well-shaken "coating mixture" and very slowly lifted out so that the top of the filter was drying whilst the filter was being removed from the solution (Figure 1 (a)).

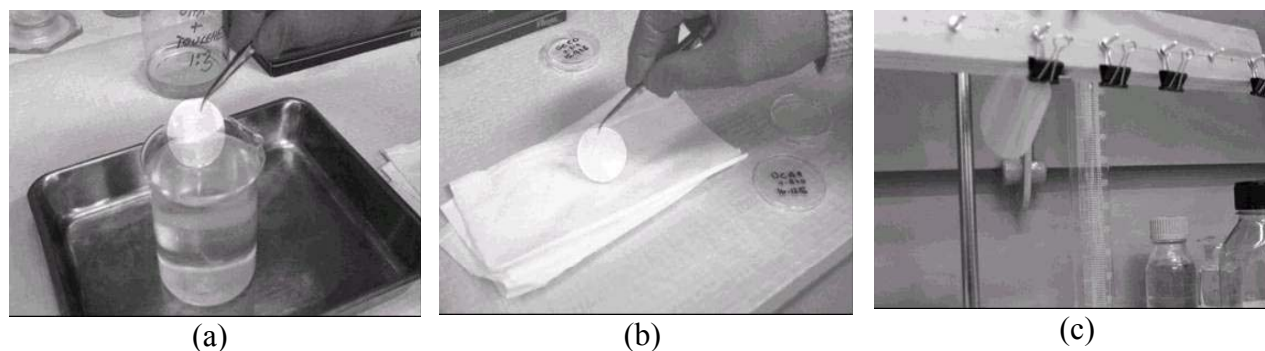


Figure 1. Procedure of coating of Nuclepore filters in laboratory conditions.

It should be noted that the speed of extraction bears significantly on the uniformity of the coating obtained and therefore it should be done slowly.

Once the filter is out of the solution the lower edge should be dabbed onto a tissue to remove any excess drops of mixture (Figure 1 (b)), and then hung to dry for 30 minutes (Figure 1 (c)). The coating applied by this method added typically (115 ± 20) μg of Apiezon grease per filter

The MSDS for Toluene should be consulted before proceeding with this process as Toluene is poisonous and the appropriate safety measures need to be taken. In addition all the work should be carried out in a properly ventilated fume cabinet.

3. Results

A number of side by side tests were undertaken with two GENT sampling units at the Lucas Heights sampling site. The inlets of the units were approximately 1.5m apart and both units collected $\text{PM}_{2.5-10}$ aerosol particles on coarse stage filters and $\text{PM}_{2.5}$ on fine stage filters. One unit was using factory coated coarse filters and the other unit uncoated filters, filters coated at ANSTO laboratory and filters coated and sent for testing to us from the US.

Blank and exposed filters were analysed on ANSTO's 2 MV HVEE tandem accelerator with 8-mm diameter proton beam of 2.6 MeV energy using 30 μC of accumulated charge per filter. Twenty one (21) elements (F, Na, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Br, Sr and Pb) were analysed using standard PIXE and PIGE analyses.

3.1 Influence of coating procedures on air flow through the filter

Uncoated, factory coated, ANSTO laboratory coated and filters from six different coating methods received from Professor P. Hopke and referred to as US "Hopke" filters in the report were used in this test. The pressure drop across each filter was measured. The range of pressure values at 10 and 18 l/min of flow rates are presented in Table 1 below.

Table 1. Comparison of the pressure ranges for factory, laboratory and US "Hopke" coated filters.

Coating Method	Pressure Drop (kPa)	
	at 10 l/min	at 18 l/min
Uncoated Filters (UC)	-0.8 to -1.0	-1.8 to -2.0
Nuclepore factory coating method (FC)	-0.9 to -1.1	-1.9 to -2.1
ANSTO laboratory coating method (LC)	-0.9 to -1.1	-1.9 to -2.1
US "Hopke" Coating method #1	-1.0 to -1.3	-2.2 to -2.8
US "Hopke" Coating method #2	-0.8 to -1.2	-1.8 to -2.5
US "Hopke" Coating method #3	-0.8 to -0.9	-1.8 to -2.0
US "Hopke" Coating method #4	-0.8 to -0.9	-1.8 to -2.0
US "Hopke" Coating method #5	-0.8 to -0.9	-1.8 to -1.9
US "Hopke" Coating method #6	-0.8 to -0.9	-1.8 to -1.9

As could be seen the Table 1 the filters coated by US "Hopke" methods #1 and #2 appear to have larger pressure drops and hence more blocked pores while the pressure drops for other methods was acceptable and would not impair particle collection.

3.2 Analysis of blank filters

PIXE (Proton Induced X-Ray Emission) and PIGE (Proton Induced Gamma-Ray Emission) analyses of blank uncoated and coated filters showed that all blank filters had some significant levels of trace elements present. These elements and their average concentrations are listed in the Table 2.

Table 2. Trace elements detected in blank filters, ng/cm²

Coating Method	F	Na	Si	S	Cl	Ca
Uncoated Filters (UC)	0	0-270	120-140	15-18	0	0
Nuclepore factory coating method (FC)	0	0-230	120-140	15-25	0	0
ANSTO laboratory coating method (LC)	0	0-280	120-140	19-29	0-66	0-8
US "Hopke" Coating method #1	4,000-63,000	0-240	130-150	18-21	13-17	8-11
US "Hopke" Coating method #2	17,000-72,000	0-250	120-190	16-19	15-24	0-22
US "Hopke" Coating method #3	7,000-12,000	0-140	120-140	17-25	26-27	9-13
US "Hopke" Coating method #4	800	190	120	18	24	0
US "Hopke" Coating method #5	64	300	133	15	18	0
US "Hopke" Coating method #6	72	171	136	15	17	8

The collected PIXE spectra for blank filters are presented in Figure 2 they clearly show the presence of above-mentioned elements. However, these elements present relatively small trace quantities (Table 2) compared actual aerosol samples where these elements are present in much higher quantities. Iron (Fe) was present as a measurement artefact and was not present in the blank filters.

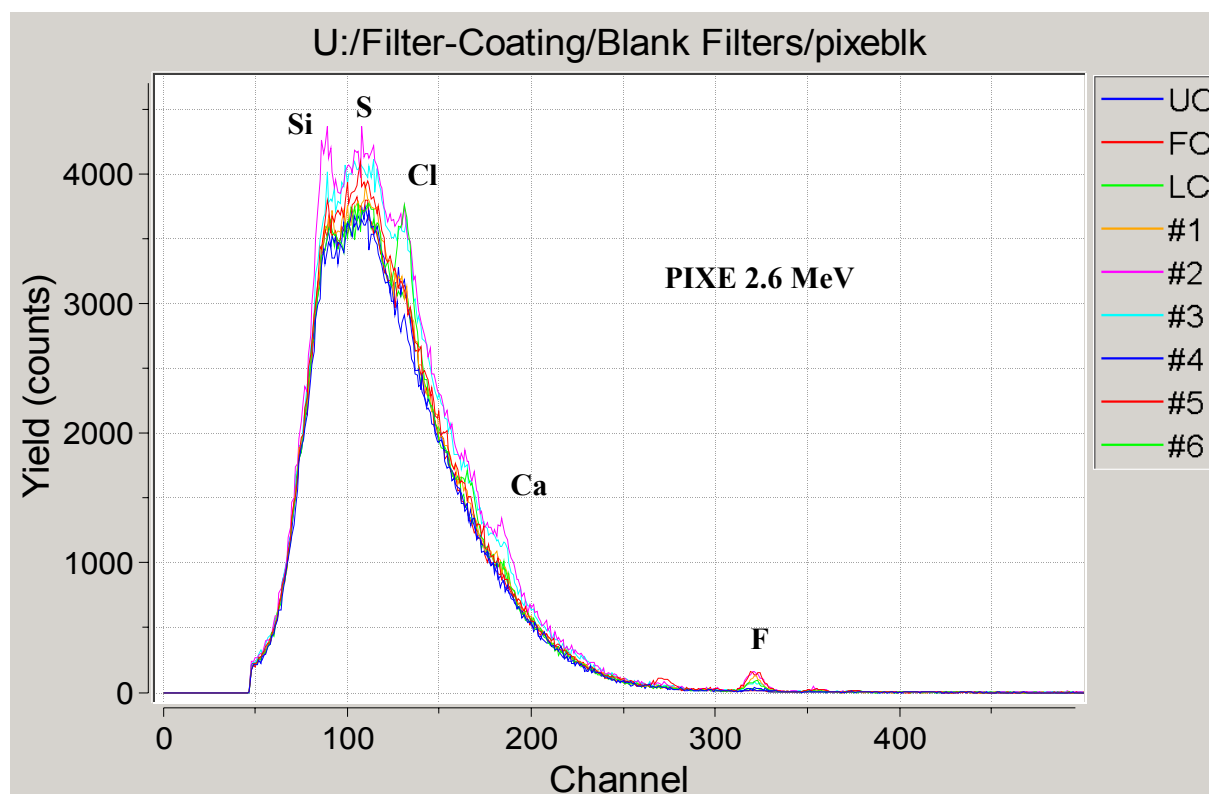


Figure 2. Blank Filters – PIXE analysis

The real concern was the presence of F in US "Hopke" coated filters by methods #1, #2, #3 and #4. The PIGE spectra of blank US "Hopke" coated filters, blank uncoated filters, blank factory and blank ANSTO laboratory coated filters are shown in the Figure 3 below.

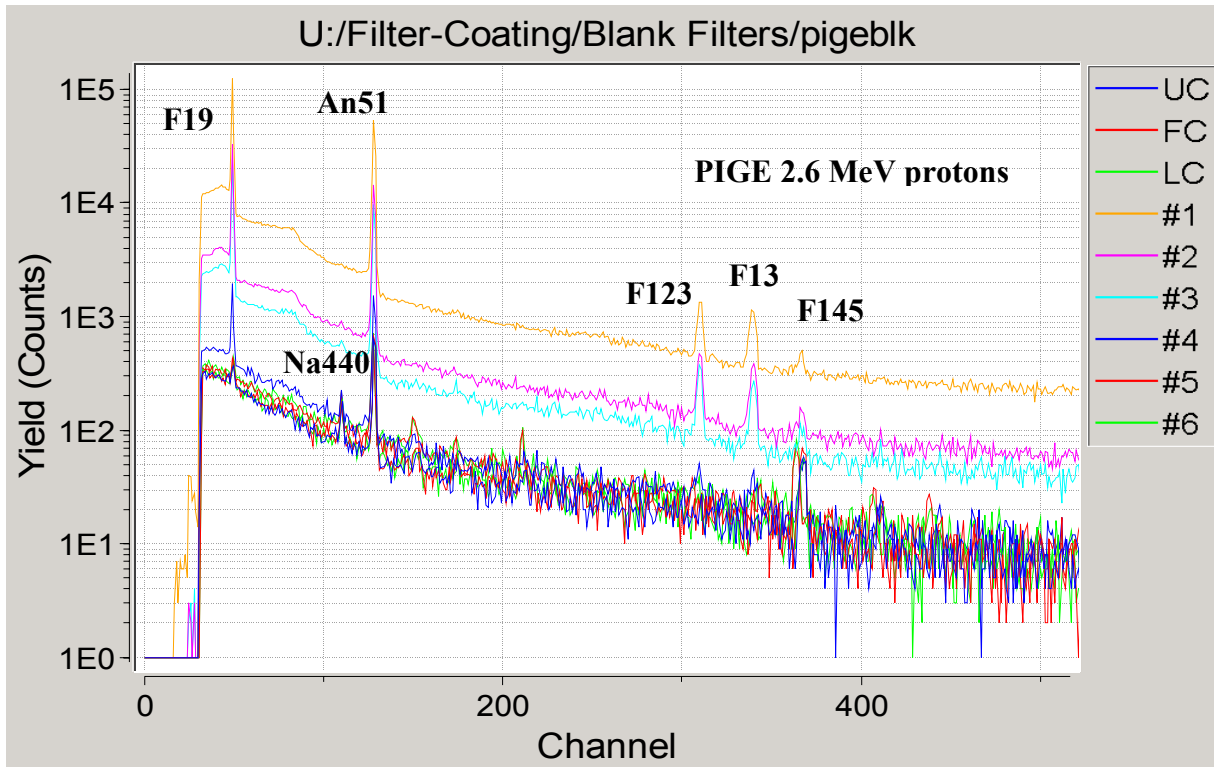


Figure 3. Blank Filters – PIGE analysis

The RBS (Rutherford Backscattering) analysis of filters provided the results on filters and coating thickness. The spectra collected during RBS analysis are shown in Figure 4.

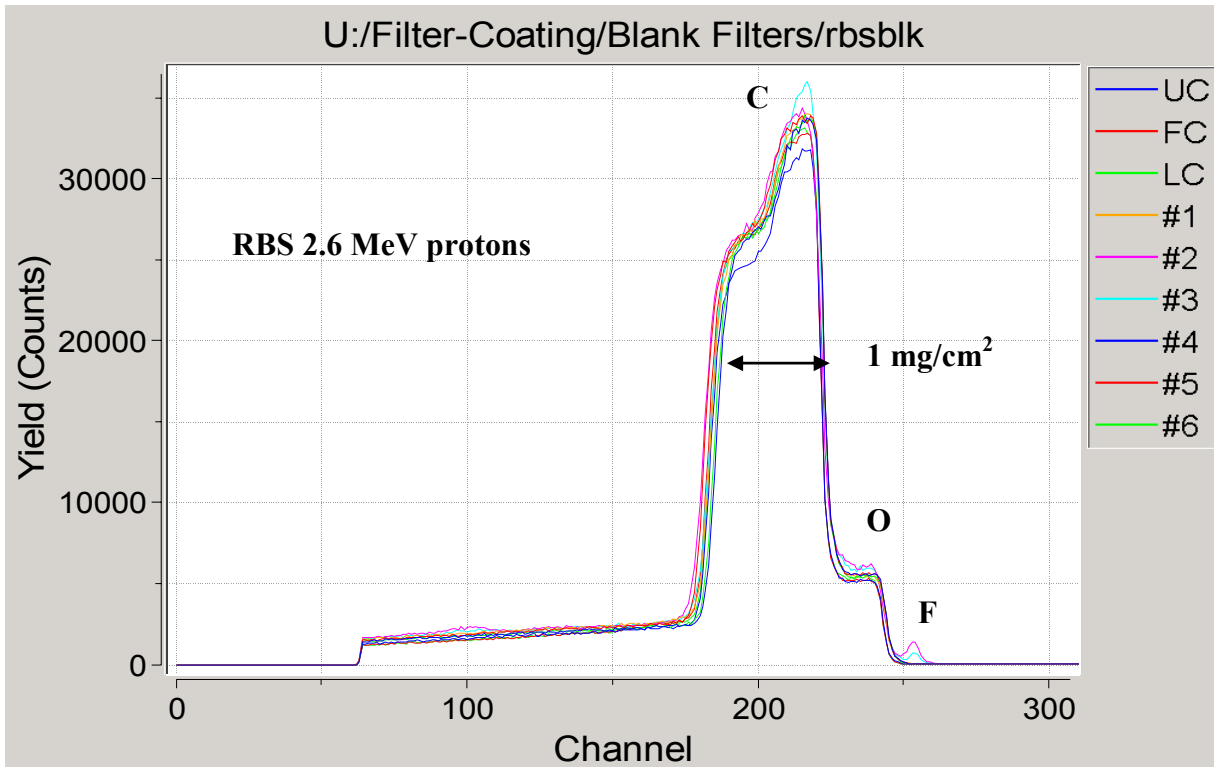


Figure 4. Blank Filters – RBS analysis

As appears in Figure 4 the average thickness of all Nuclepore filters used in the experiment was around $1 \mu\text{g}/\text{cm}^2$ and the thickness of coating appeared to be similar and much thinner by all methods. The simulation of spectrum on US ‘‘Hopke’’ filter coated by method 2 is presented in Figure 5 showing that the thickness of fluorine present in this method of coating was around $16 \mu\text{g}/\text{cm}^2$.

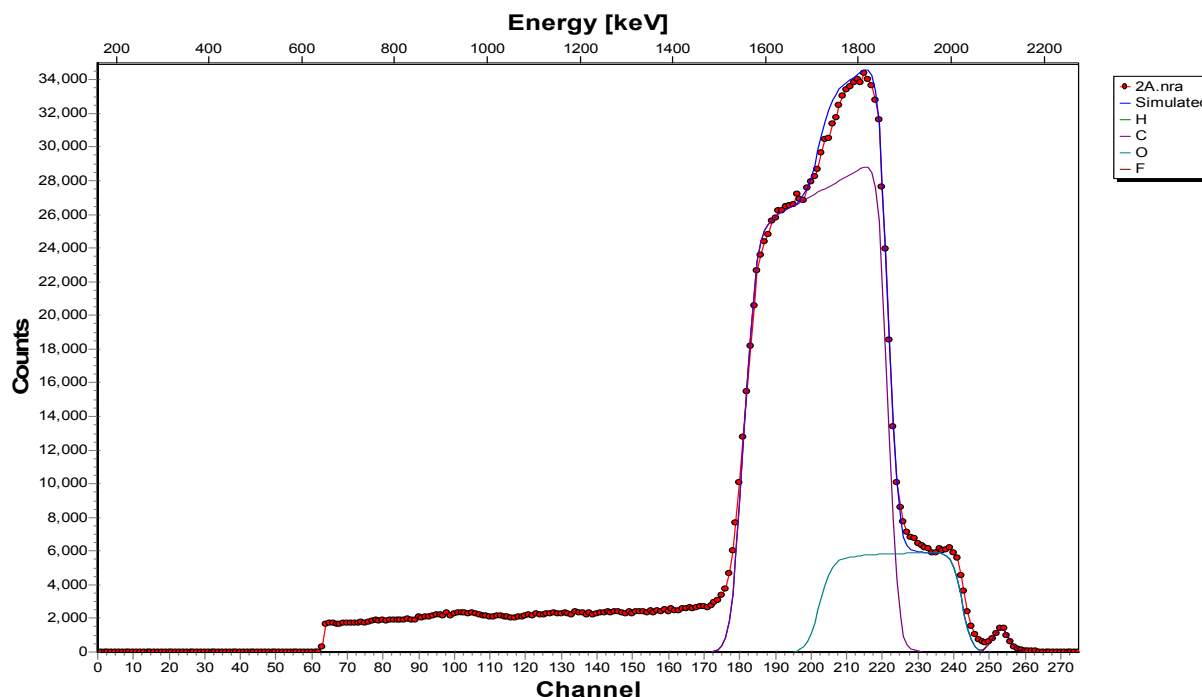
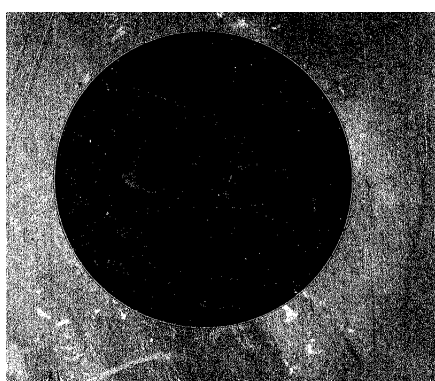


Figure 5. Sample #2A: $(\text{H}_{14}\text{C}_{16}\text{O}_3)_n$ layer thickness $85,000 \times 10^{15} \text{at}/\text{cm}^2$ or $1.087 \text{mg}/\text{cm}^2$
F layer thickness $= 1,230 \times 10^{15} \text{at}/\text{cm}^2$ or $0.0157 \text{mg}/\text{cm}^2$.

The blank filters were also examined for the uniformity of the applied coating. Filters were scanned on the flat-bed scanner to observe the patterns of light transmitted. The factory coated filters (Figure 6), ANSTO laboratory coated and US ‘‘Hopke’’ filters coated by methods #5 & #6 showed no patterns of light transmission indicating the good uniformity of the coatings applied. The US ‘‘Hopke’’ filters coated by methods #1 (Figure 6), #2, #3 and #4 showed patterns of light transmission indicating that the applied coating was not completely uniform.



Factory coated filter



US ‘‘Hopke’’ filter coated by
method #1

Figure 6. Scanned images of coated filters.

The biggest concern was the “stickiness” of US “Hopke” coated filters by methods #1, #2, #3 and #4. They were difficult to handle, stuck to tweezers, microbalance and laser absorption measurement equipment. In addition, material or part of the material collected with these filters could be lost during weighing and lasering due to this stickiness.

3.3 Analysis of factory coated vs. uncoated filters

In the series of tests using uncoated coarse filters, PIXE/ PIGE analysis of exposed filters showed that concentrations of Na, Al, Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn (Figure 7 (a)), were lower on uncoated coarse filters.

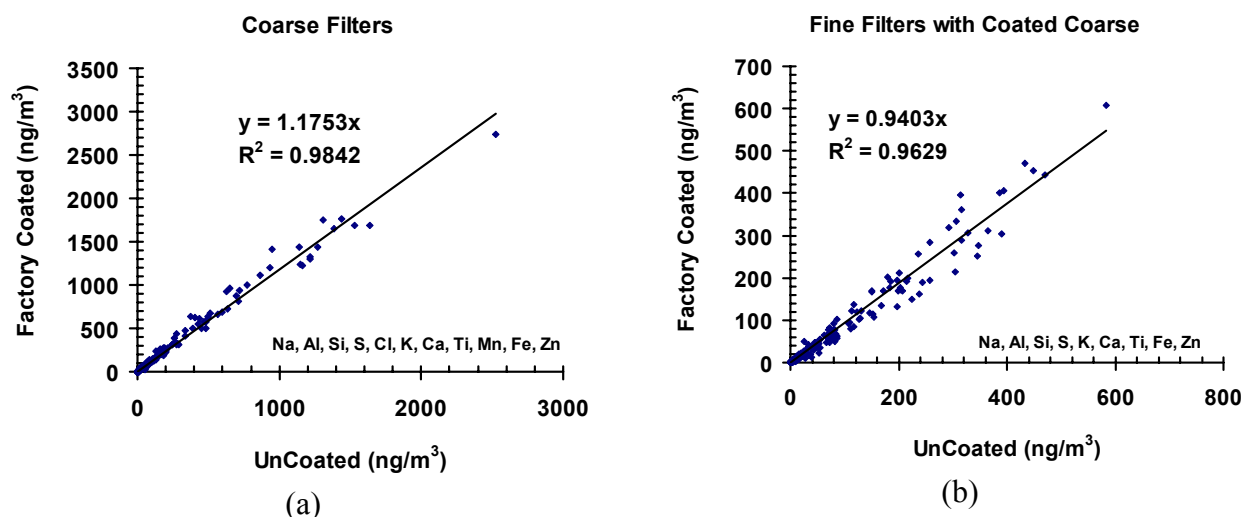


Figure 7. Comparison of elemental concentrations measured on factory coated vs. uncoated filters.

Concentrations of Al, Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn were higher on fine filters run in conjunction with these uncoated filters (Figure 7 (b)). This was due to passage of certain amount of coarse PM_{2.5-10} particles onto fine filters via “bounce off” from uncoated coarse filters.

3.4 Analysis of factory coated vs. US “Hopke” coated filters by methods #1, #2, #3 and #4

In the series of tests using US “Hopke” coarse filters coated by methods #1, #2, #3 and #4, PIXE/ PIGE analysis of exposed filters also showed that concentrations of Na, Al, Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn, (Figure 8 (a)), were slightly lower on these filters and slightly higher on fine filters run in conjunction with these coarse filters (Figure 8 (b)). These indicate a very small “bounce off” effect, that could be due to non-uniformed coating across the filter surface.

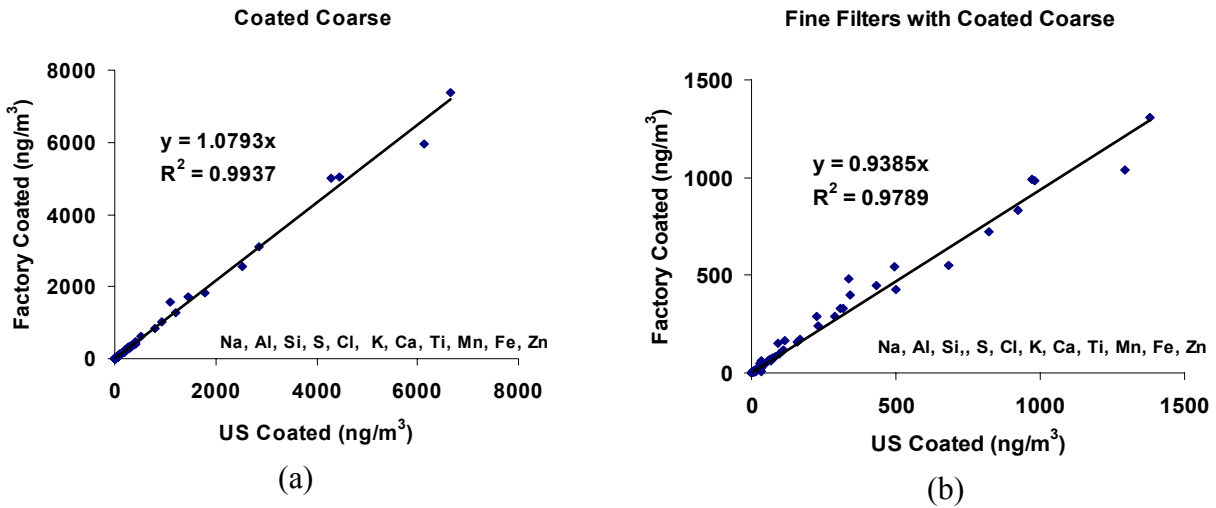


Figure 8. Comparison of elemental concentrations measured on factory coated vs. US coated filters by methods #1, #2, #3 and #4

3.5 Analysis of factory coated vs. US “Hopke” coated filters by methods #5 and #6

Analysis of filters exposed in side by side tests showed that concentrations of Al, Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn were slightly lower on coarse filters coated by method #5 than on factory coated coarse filters (Figure 9 (a)). The concentrations of the above elements were also slightly higher on the fine filters ran in pairs with these coarse filters (Figure 9 (b)). This indicated that the “bounce off” effect of PM_{2.5-10} particles on filters coated by method #5 was still present. On the other hand the “bounce off” effect was negligible on filters coated by method #6 (Figure 10).

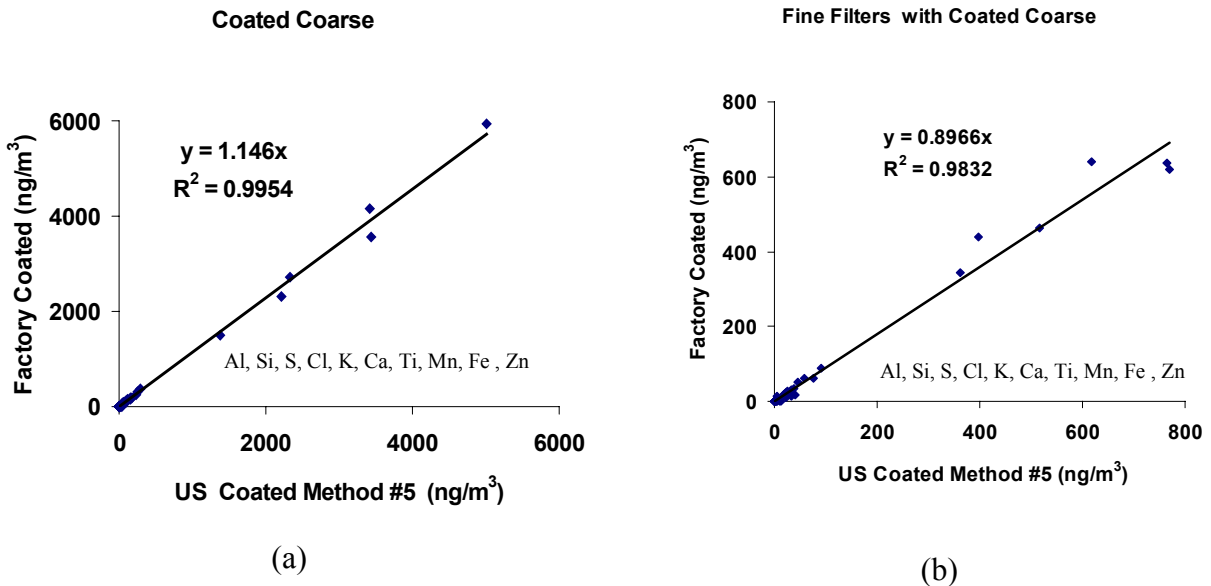


Figure 9. Comparison of elemental concentrations for coating method #5

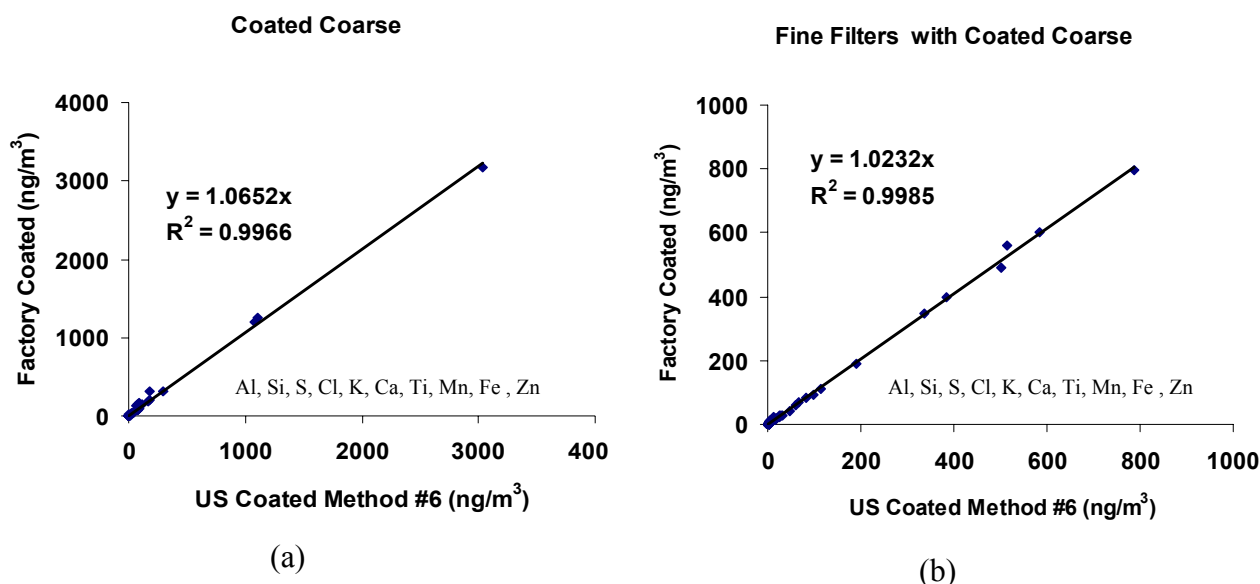


Figure 10. Comparison of elemental concentrations for coating method #6

3.6 Analysis of factory coated vs. ANSTO laboratory coated filters

In the series of tests using ANSTO laboratory coated coarse filters PIXE/ PIGE analyses of exposed filters showed negligible differences in concentrations of elements found in collected particles. Indicating negligible difference in collecting aerosol particles on laboratory coated coarse filters Laboratory coated coarse filters did not affect particle collection on fine filters. This was well demonstrated by Figures 11 (a) and (b).

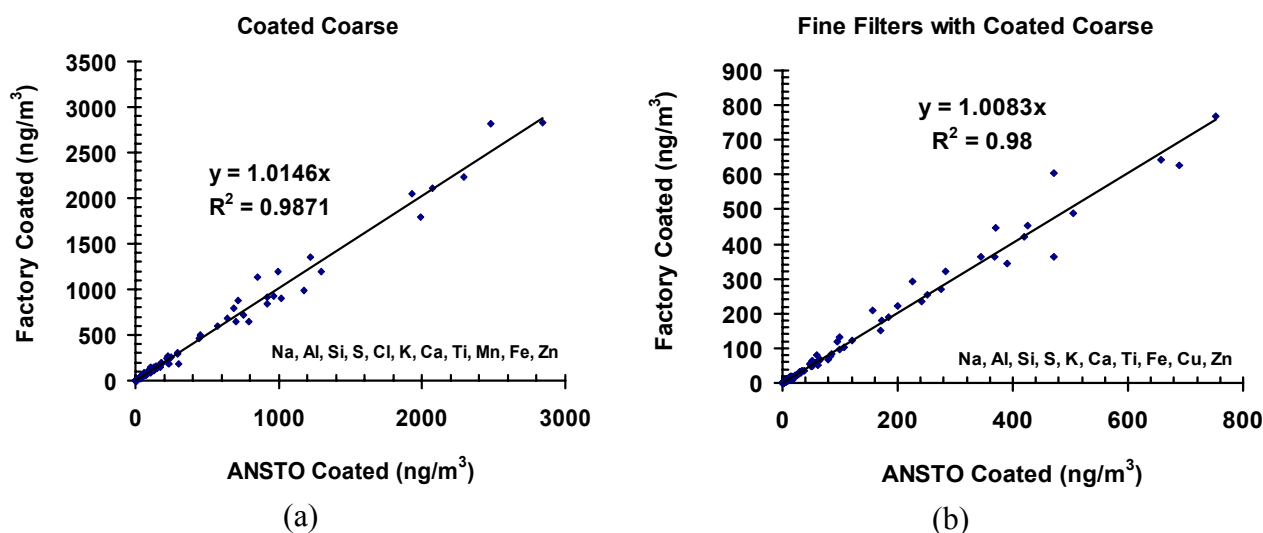


Figure 11. Comparison of elemental concentrations measured on factory coated vs. ANSTO coated filters.

4. Conclusion

Experiments showed that coarse Nuclepore filters should be coated with a thin layer of grease to avoid particle "bounce off" effects.

Commercial coating of filters is still in the development stage by the US-based company, SPI Supplies.

Coating with Apiezon grease can be successfully achieved under laboratory conditions. These coated filters perform as well as the factory coated filters. The process of coating these filters is not expensive, but it is time consuming. This problem could be partially overcome by designing a rig that could accommodate a number of filters dipping at the same time and speeding up the coating process.

5. Reference

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