TRACE ELEMENTS DETERMINATION IN BLANK POLYSTYRENE AIR FILTERS BY CHARGED PARTICLE ACTIVATION ANALYSIS WITH A CYCLOTRON

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The trace elemental content of Microsorban 99/98s polystyrene air filters supplied by Delbag Luftfilter, of West Berlin) was determined by charged particle activation analysis, using a cyclotron. These high-flow air filters have been extensively used by CSIRO in Australia for air sampling, and for the analysis of the particulate matter trapped in them. As we wanted to carry out trace elements analysis in the particulates it was, therefore, essential to estimate the concentrations of various elements in the blank filters themselves. A number of elements: S, Ca, Ti, Cr, Fe, Ni, Cu, Zn, Se, Sr, Zr, and Hg were detected in the blank filters with respective concentrations of 0.01, 0.54, 0.93, 0.04, 4.4, 1.2, 0.26, 1.5, < 0.003, 0.01, < 0.001 and 0.01 mg/m<sup>2</sup>.

## INTRODUCTION

Microsorban (99/98s) Polystyrene air filters, supplied by Delbag Luftfilter of West Berlin, have been extensively used by CSIRO in Australia to carry out high volume air sampling along the East Coast of Australia, for environmental monitoring purposes. These filters are capable of retaining in excess of 99% of particles with diameters greater than 0.2 µm. Therefore, they are very well suited for studying the atmospheric particulates, including their chemical composition, as most of the particulates mass in the lower troposphere consist of particles of 0.1  $\mu\,\text{m}$  -10 Um diameter (1). However, before the trapped particulates could be analysed for their chemical/trace elemental composition, it is necessary to know the trace element contents of the blank polystyrene filters. This information was, unfortunately, not available from the manufacturers/suppliers or any other source and, therefore, we had to carry out these measurements ourselves.

We chose the technique of thick-target charged particle activation, using 8.5 MeV

protons from a cyclotron, to carry out the trace elemental analysis in a number of blank polystyrene filters.

## EXPERIMENTAL PROCEDURE

The experimental set-up is described in detail by Lee (2). The target/irradiation chamber is shown in figure 1. As the targets of polystyrene were going to be thick, stopping the 8.5 MeV incident protons completely, the target chamber had to be isolated electrically from the rest of the beam line, for beam monitoring purposes. The target holder consisted of a piece of aluminium in which 2 or more recessed grooves were made. The target samples could be placed inside the recess and covered with a thin piece of aluminium foil which, in turn was secured with a brass bracket. The holder is joined to an "arm" containing liquid nitrogen. With an 8.5 MeV beam at 1  $\mu$ A, 8.5 W of power must be dissipated by the target. Using the set-up as shown, the temperature of the target holder was monitored with a temperature sensitive chemical indicator and was found to stay below 50 degrees Celcius for all targets during a typical run. The temperature of the target at the point where the beam hits would of course be a lot higher and will depend upon its thermal conductivity. In no cases, however, were there any signs of scorching. Without the liquid nitrogen, it was found that the aluminium foil was punctured after about 10 minutes.

Sample preparation involved cutting a 1 cm square piece from each filter, originally about 15cm square, placing it in a recessed groove in the aluminium target holder and covering it with a thin piece of aluminium foil. Great care had to be taken with the samples so as not to introduce any contaminants. Handling was done using various instruments that were thoroughly



Target chamber

Figure 1. The irradiation chamber and experimental set-up



Figure 2. Gamma spectrum from an irradiated polystyrene filter. The nuclides, which after irradiation gave rise to various gamma rays, are listed on the respective peaks

cleaned and rinsed in multiply distilled water. The aluminium foil was in turn held down with a brass bracket.

Each sample was irradiated with 8.5 MeV protons for 1 hour at a current of 1  $\mu A$ . During each run, the target assembly was cooled with liquid nitrogen, the volume contained in the target arm being more than adequate for the 1 hour. Each filter appeared intact at the end of the irradiation period with no evidence of scorching.

A standard was prepared by placing a blank piece of filter paper of known dimensions into a solution containing compounds of the elements of interest dissolved in multiply distilled water. The compounds used were  $CaCO_3$ , TiO<sub>2</sub>, KCr<sub>2</sub>O<sub>6</sub>, Fe<sub>2</sub>O<sub>3</sub>, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuSO<sub>4</sub> .5H<sub>2</sub>O, ZnO, SeO<sub>2</sub>, ZrNO<sub>3</sub>, HgCl<sub>2</sub>, SrO, Sc<sub>2</sub>O<sub>3</sub>, As<sub>2</sub>O<sub>3</sub>, Sb<sub>2</sub>O<sub>3</sub>, PbF<sub>2</sub> and V<sub>2</sub>O<sub>5</sub>. The filter was then allowed to dry in free air and irradiated under the same conditions as the blank filters.

After waiting for an hour, to let most of the short-lived positron activities die down, each filter sample was counted with a Ge(Li) detector in an anti-Compton arrangement with two NaI(T1) crystals (2). The system resolution was 2.7 keV at 1.33 MeV and the peak-to-Compton ratio was 120. The detector efficiency, as a function of energy, was determined using a Standard Radium Source (3) with the source at a distance of 20cm from the face of the detector.

## RESULTS AND DISCUSSIONS

A typical gamma spectrum is shown in figure 2. All the peaks in the spectrum have been identified with Computer Analysis programme SEARCH (2) and the original nuclides, giving rise to these peaks, listed above the peaks. With the same programme the area of various peaks was intergrated after subtracting the background. Then, by comparing the areas of different peaks in the "standard" and blank filters, the trace elemental composition of these "blanks" was calculated.

The average levels of trace elements found in the blank polystyrene filters are shown in Table 1.

There is a rather large error ( $\pm 30$ %) associated with these values. These we conclude to be due to the large variations in the trace elemental levels found in the filters. It was not experimental error as we demonstrated that the irradiation of a number of "standards" showed variations of less than 5% in the elemental levels. These large variations were however found not to be a problem in our programme to measure the trace elemental composition of atmospheric particulates collected in the filters. The blanks contributed only a fraction of one percent of the elemental concentrations found in the particulates collected in the CSIRO's programme of environmental monitoring.

Table 1 - Trace Elements found in blank polystyrene filters

Element Concentration in filter blank ( ±30%)

S	0.01 mg/m²
Ca	0.54
Ti	0.93
Cr	0.04
Fe	4.4
Ni	1.2
Cu	0.26
Zn	1.5
Se	0.003
Sr	0.01
Zr	0.001
Нд	0.01

In conclusion it can be said activation analysis with 8.5 MeV protons, from a cyclotron, provided a convenient and accurate method of estimating trace elements "impurities" in blank Microsorban (99/98s) polystyrene air filters. However, it must also be pointed out that there might be some other impurities in these blank filters which, either could not be activated with 8.5 MeV protons, or produced activities too short-lived for our measuring technique, and therefore could not be measured.

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## References

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