Contract Report 524

Development of a Particulate Filter Standard and Its Use in an International Atomic Energy Agency Interlaboratory Evaluation

Final Report

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Champaign, Illinois

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DEVELOPMENT OF A PARTICULATE FILTER STANDARD AND ITS USE IN AN INTERNATIONAL ATOMIC ENERGY AGENCY INTERLABORATORY EVALUATION

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ABSTRACT

A set of filter standards was prepared by suspending National Institute for Environmental Studies (NIES) Vehicle Exhaust Particulates onto Teflon^R filters. To improve the adhesion of particles, a thin layer of mineral oil was applied as an aerosol to the deposit side of the filters. The suspension apparatus was designed to ensure sample homogeneity (particle size and deposit), and elemental analysis provided an information value that confirmed elemental homogeneity between filters. Participating laboratories were sent a set of two filter standards — one fine and one coarse loaded — and two filter blanks. Performance was evaluated by comparison with the informational values and interlaboratory comparisons. Overall, the participating laboratories showed agreement within expected errors, with the exception of a few elements for each of the laboratories. Evaluation rankings ranged from excellent to poor.

INTRODUCTION

The evaluation of analytical techniques and individual laboratory performance is critical to maintaining a known quality of chemical data and the confidence of users. Of importance is whether a particular laboratory is producing results with the accuracy and precision generally acknowledged for that technique, or even with the accuracy and precision they claim for themselves.

At the 1989 International Atomic Energy Agency (IAEA) research coordination meeting (RCM) in Jakarta, India, all participants accepted the need for analytical quality control (QC) within the Air Toxics Coordinated Research Program (CRP). Because none of the available QC materials from national and international suppliers were suitable for analysis by all the nuclear-related analytical techniques being used in the CRP, it was agreed that 'special' QC samples were needed.

To provide QC samples of airborne particulate matter on filter material suitable for all the techniques, a set of two sample and two blank filters was prepared for and distributed to each CRP participant. The filters were made by suspending a certified reference material (NIES Vehicle Exhaust Particulates) onto a suitable filter (Teflon^R).

Each of the 21 laboratories invited to participate was made aware of the bulk material resuspended on the filter and of their participation in an interlaboratory evaluation. Nine laboratories responded, representing proton induced x-ray emission (PIXE), neutron activation analysis (NAA) and emission detection x-ray fluoresence (ED-XRF) techniques. Results of the interlaboratory comparison were standardized and are presented in a tabular form (see Tables 6 and 7). A description of the individual laboratory methodologies is included within this report (Appendix **I).**

STANDARD PREPARATION

NIES **Vehicle Exhaust Particulates**

On the recommendations from the IAEA RCM in Jakarta, the National Institute for Environmental Studies (NIES) reference material 'Vehicle Exhaust Particulates' was chosen for suspension. This material provided a reasonable duplicate of airborne particulate matter, had undergone certification testing, contained sufficient quantities of the metals of interest, and was found to be easily suspendable. Reference samples were provided, at no cost, by the Japan Environment Agency.

The reference material was prepared from particulate matter collected from electrostatic precipitators in ventilators connected to a highway tunnel. The particulate matter was mixed into a paste with 35 percent ethanol, air-dried, oven-dried, pulverized, sieved, and homogenized. A more detailed description of the reference material is available from Okamoto (1987) and the Japan Environment Agency.

Teflon^R Filters

Deposits were collected on 37 millimeter (mm) diameter Teflon^R disks with a polyethylene support ring (manufactured by Gelman Science, Ann Arbor, MI). According to the manufacturer, these filters collect all particles with diameters greater than 0.2 micrometers *(µm)* from the air passing through them. These filters are suitable for nuclear methods of elemental analysis. Teflon^R disks provide a thin deposit layer suitable to XRF and PIXE analysis, and demonstrate no significant contamination, making them suitable for NAA. Earlier work by Sweet and Gatz (1987) showed the polyethylene support ring to be contaminated with Cr, Mn and Sb, thus the support ring was removed for NAA. This was not necessary with XRF, because only the center portion of the filter is analyzed.

Oil Coating of Filters

To improve the adhesion of particles, a thin layer of mineral oil (Kaydol™) was applied to the deposit side of the Teflon^R filters. The mineral oil was applied as an aerosol using a particle generator made by Particle Measuring Systems, Inc., Boulder, CO (PMS Model PG-100) and following the instructions of Dzubay and Barbour (1983). A schematic of the oiling apparatus is illustrated in Figure 1.

Prior to the loading of the reference material, Teflon^R filters and mineral oil deposits were evaluated for possible contamination. Two packages, each containing three sets of blank and oiled filters were sent for NAA and XRF analysis. Reported concentrations (Table 1) for the blank filters and oil deposits show no significant contamination. While there are measurable quantities of some elements, they are reported at concentrations many orders of magnitude lower than the anticipated concentrations (NIES reported values) of the vehicle exhaust particles.

Suspension Chamber

Suspension of the vehicle dust was achieved in a suspension apparatus (Figure 2). Dust was suspended in a swirl chamber by a continuous supply of filtered compressed air. The compressed air and suspended dust were forced into a circular air motion (swirl) about the axis of the chamber where the particles are mixed and disaggregated. The disaggregation of the suspended particles removes possible elemental inhomogeneity between filters due to fractionation effects (i.e., coarse particles are truly coarse particles and not aggregates) and assures true particle sizes for techniques requiring particle standards and corrections. The flow was exhausted into a 225 liter (L) sampling chamber for dichotomous sampling and PMS particle counting. Particle samples were collected within the chamber using an automatic dichotomous virtual impactor fitted with a PM-10 inlet (series 245) made by Anderson, Inc., Atlanta, GA. The sampler is designed to collect particulate matter with an aerodynamic size cutoff of 10 *µm* and to further separate particles into two size fractions:

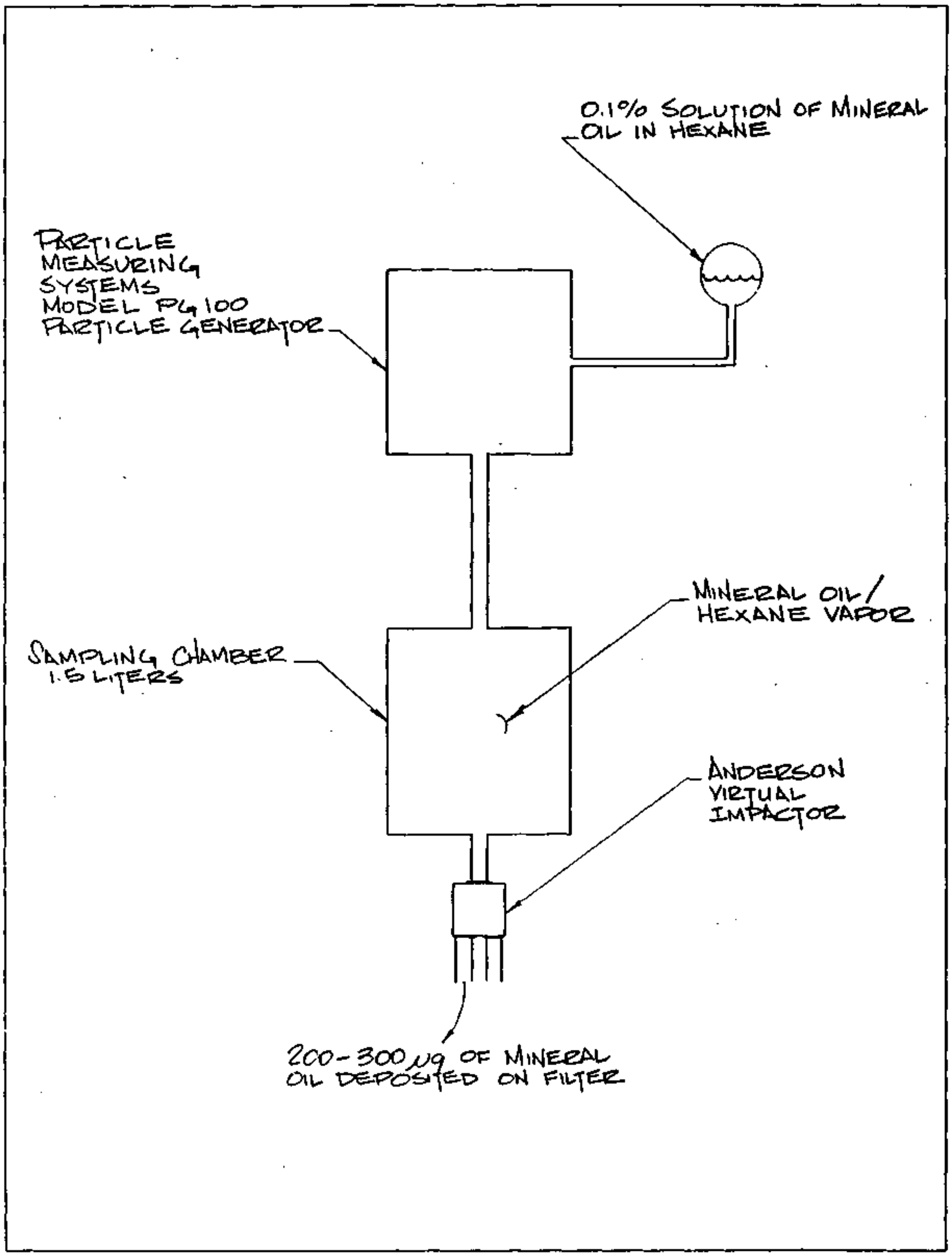

Figure 1. Schematic of the oiling apparatus.

Table 1. Blank Filter and Oiled Filter Elemental Concentrations

Element	Analytical Method	Blank Filter (μg/filter)	Oiled Filter (μg/filter)
Al	NAA	0.00006	0.0002
As	NAA	< 0.0002	< 0.0005
Br	NAA	< 0.0014	< 0.0018
Ca	NAA	0.019	0.117
Cl	NAA	0.046	0.6
Cr	XRF	0.0083	0.0123
Cu	NAA	< 0.013	0.025
Fe	XRF	0.113	0.119
K	XRF	0.009	0.021
La	XRF	< 0.0002	< 0.0011
Mn	NAA	0.0023	0.0016
Na	NAA	< 0.039	0.398
Pb	XRF	0.017	0.033
S	XRF	0.041	0.115
Sb	NAA	< 0.0002	< 0.0005
Si	XRF	< 0.005	< 0.093
Sm-	NAA	< 0.00003	< 0.00007
Sr	XRF	< 0.005	< 0.012
Ti	NAA	< 0.033	< 0.040
V	NAA	< 0.0001	< 0.0002
Zn	XRF	0.006	0.009

Notes: NAA - neutron activation analysis
XRF - x-ray fluoresence

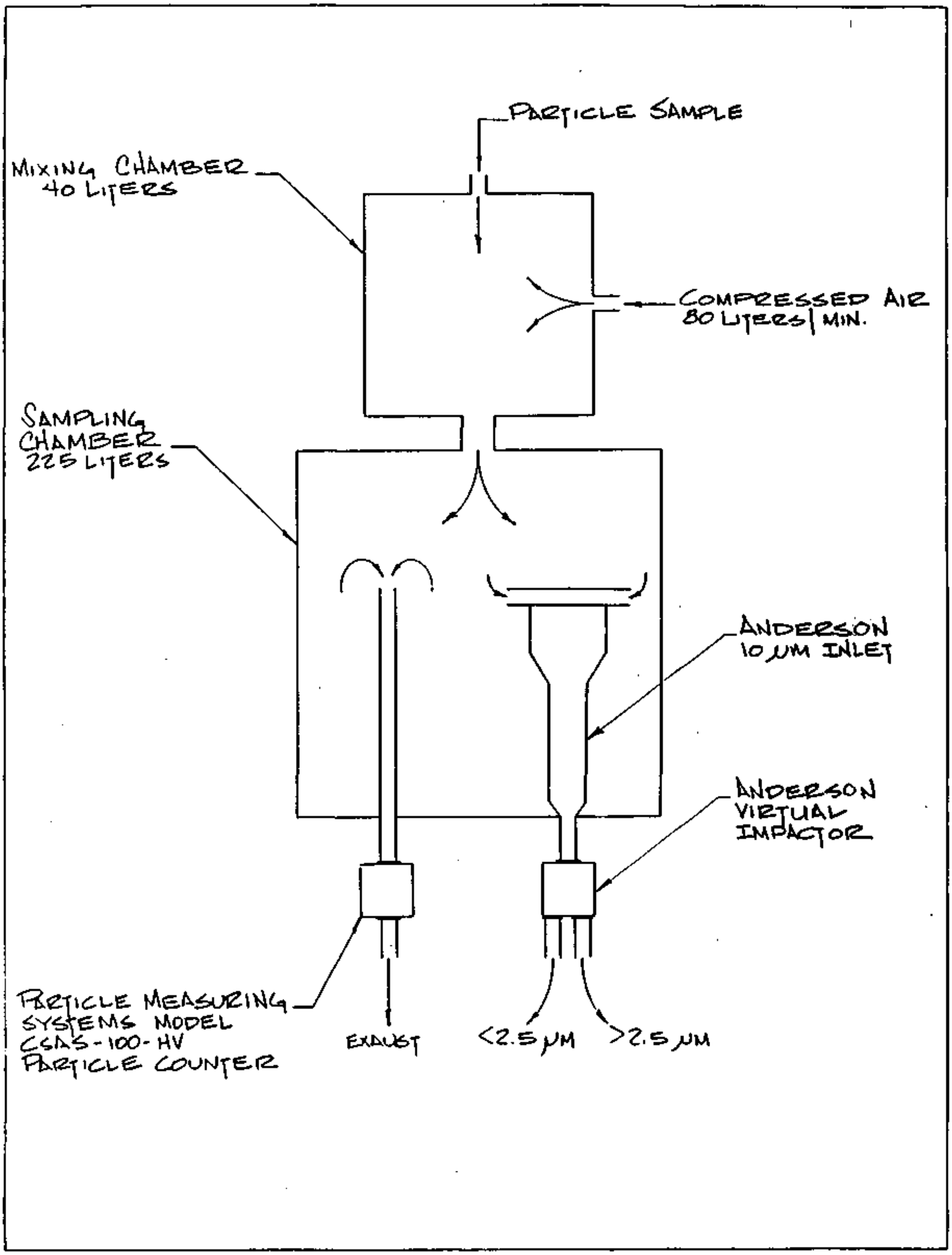

Figure 2. Schematic of the dust suspension apparatus.

a fine particle fraction (< 2.5 *μm)* and a coarse particle fraction (2.5 to 10 μm).

The similarity in composition of loaded filters was aided by the continuous monitoring of aerosol size distributions using a PMS laser probe (model CSAS-100-HV). Filter loadings were not initiated until a preset particle size configuration was monitored within the settling chamber (Figure 3). This precaution limited elemental fractionation between filters due to loadings with varying particle size distributions. Our success in maintaining similar particle size distributions for each of the filters is illustrated in Figure 4.

Homogeneity of the filter loading is an important concern for PIXE and XRF analysis, as the usual beam spot is restricted to 1 square centimeter (cm^2) on a 6.6 cm^2 filter. Analysis provided by Lab 5 of three spots on both the fine and coarse loaded filters shows no significant inhomogeneities (Figure 5).

Filters were equilibrated for 24 hours at 50 percent relative humidity before weighing. All filter handling and weighing was done in a clean room on a laminar flow clean bench. Using a Cahn microbalance, the precision (standard deviation) of duplicate weighings under these conditions was ± 5 μg.

Elemental Analysis

Loading filters with a deposit of particles ≤ 10 µm will likely negate the certified value reported by NIES. Elemental fractionation by particle size has been reported in the literature (Schutz and Rahn, 1982; Gatz et al., 1986; Van Borm et al., 1988) and has been suggested in our own work (Vermette et al., 1990).

To assure a usable filter sample, a random subset of 12 of the prepared filters was subjected to two independent analytical techniques — neutron activation analysis (NAA) and x-ray fluoresence (XRF). Both techniques were used extensively in certifying the bulk NIES Vehicle Exhaust Particulate reference material, although the laboratories contributing to this report did not contribute to the certification of the reference material.

Neutron Activation Analysis (NAA)

Neutron activation analysis was performed, using the TRIGA reactor at the University of Illinos, for three sets of prepared filters.

For the short-lived radioisotopes (Al, Br, Ca, Cu, Sr, Mn, V, Ti, Cl, and Na), filters were placed in acid-washed 7 cubic centimeter (cc) polyethylene vials in rabbit carriers and irradiated for a period of S minutes at a flux of 1.5

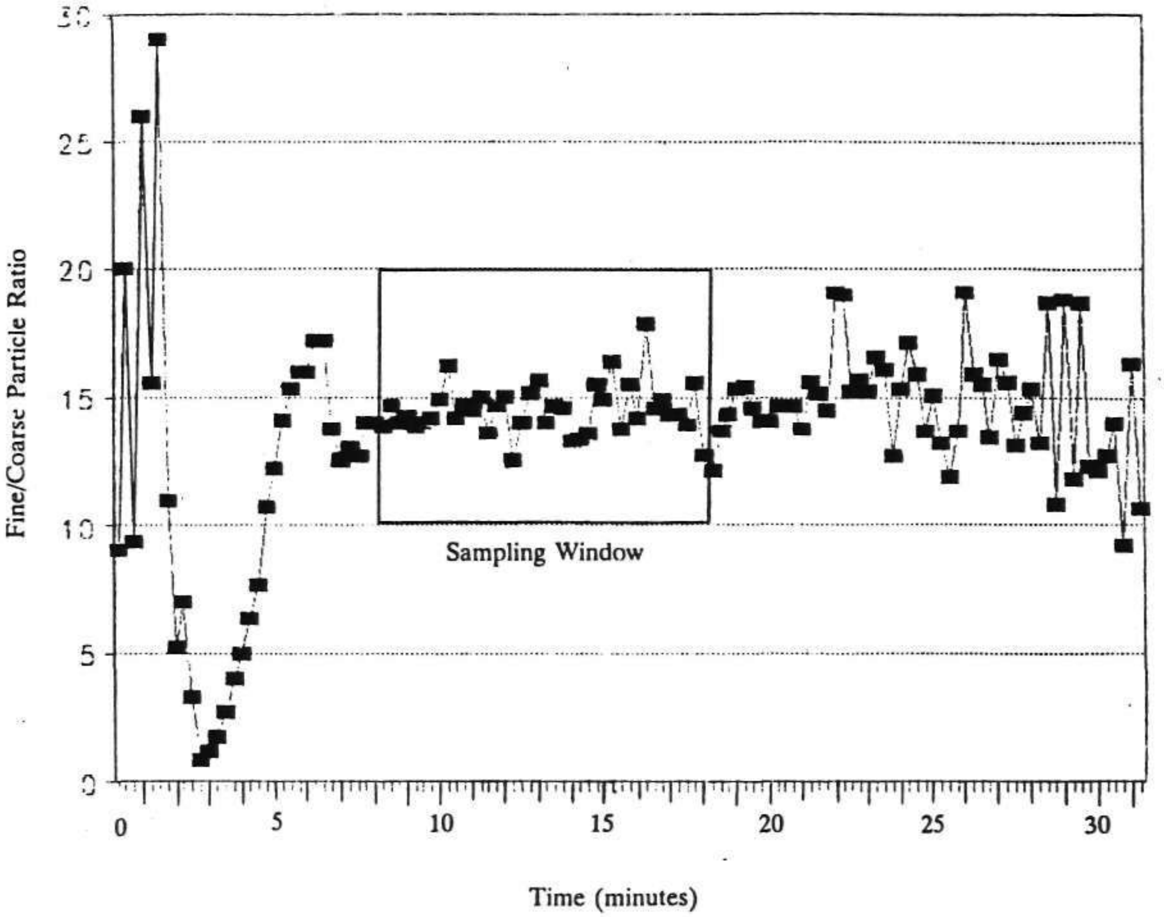

Figure 3. Particle size ratios monitored during a single suspension run (particle deposition to the filters occurred only within the sampling window).

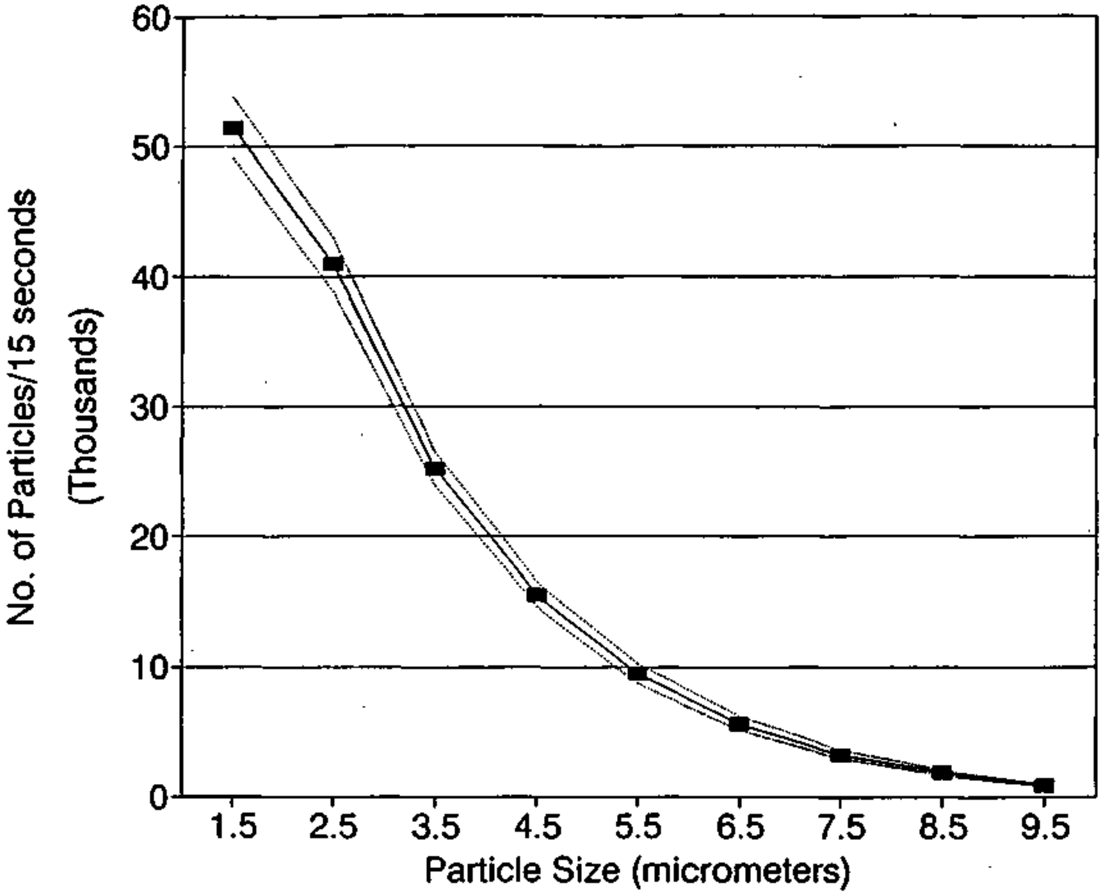

Figure 4. Mean particle counts for 50 suspension runs (dashed line equals one standard deviation; values were taken at 5 minutes (midpoint) into a 10-minute suspension run).

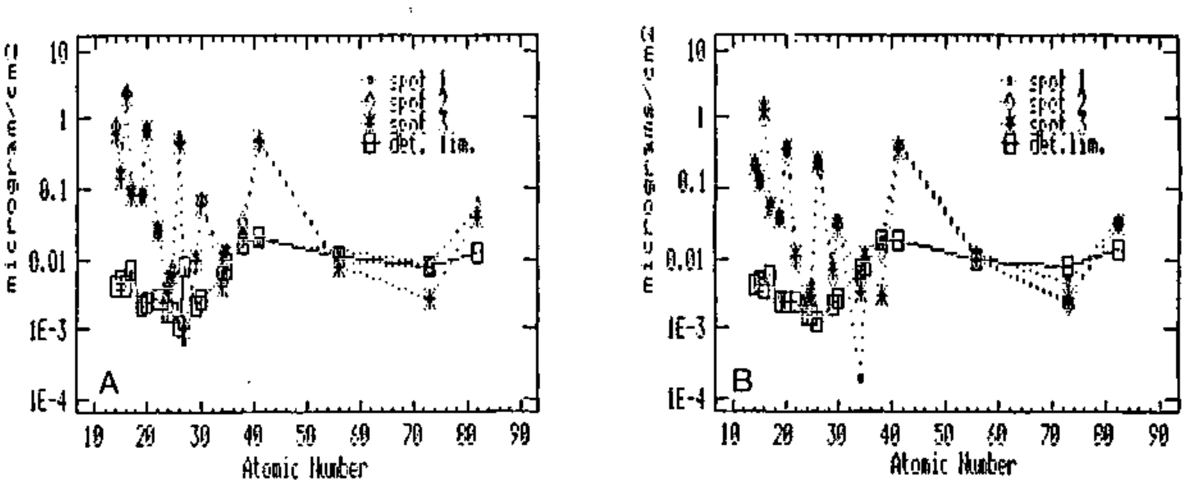

Figure 5. Results obtained for three spots on the coarse (A) and fine (B) loaded filter.

x 10^{12} neutrons per square centimeter per second (n/cm^2/sec). After the return of the carrier the filter was removed from the vial, placed in an inert vial, and counted by a hyper-pure germanium counter. Typical delay times were of the order of 4 to 5 minutes. Samples were counted for 10 minutes. Dead-time corrections were evaluated by using a 60 hertz (Hz) pulser. Variations in neutron flux were monitored using sulphur standards every few hours throughout the day. Flux variations (1 percent or less) were constant during the day, but varied up to 5 percent for different days. All variations were normalized to the standard calibrations.

For the medium-lived (As, Sb, La, and Sm) and long-lived isotopes (Se, Zn, Cr, Fe, Ni, etc.) filter samples were irradiated for 10 hours at a flux of 4.5 x 10^{12} n/cm^2/sec. Delay times of 3 to 4 days and counting times of 30 to 60 minutes were used for the medium-lived isotopes. Long-lived isotopes were counted for a period of 20,000 seconds (5.55 hours) after a 3 to 4 week decay. Flux variations ± 5 percent were monitored using cobalt flux wires. Calibration was performed using liquid standards from atomic absorption solutions, coal standards and blank filters were run with each group of filters.

X-Ray Fluorescence (XRF)

Employing XRF, NEA, Inc. of Beaverton, OR evaluated the remaining three filter sets. The methods of XRF are based on atomic excitation of electrons with subsequent emission of characteristic x-rays when electrons from higher levels fill the void spaces.

The XRF analyses were carried out using an ORTEC TEFA III energy dispersive XRF analyzer. Each filter was analyzed three times in each of three different excitation conditions optimizing the sensitivity for specific elements as indicated below:

Al, Si, P, and Fe	Mo anode, no filter, 15 kiloelectron volts (KeV), 200 microamps (μ amps)
S, Cl, K, Ca, Ti, V, Cr, Mn, and Fe	W anode, Cu filter, 35 KeV, 200 μ amps
Fe, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Sr, Ba, La, Hg, and Pb	Mo anode, Mo filter, 50 KeV, 200 μ amps

Filter blanks were analyzed, and an average blank spectrum was used as **a** background subtration for each sampled filter. Sample data were then corrected for spectral interferences, particle size, and deposit absorption effects.

Information Values

Elemental determinations for the fine and coarse loaded filters are presented in Tables 2 and 3, respectively. Overall, reproduciblity in elemental concentrations between filters, and where comparable (i.e., Br, Ca, Cu, and Ti) between XRF and NAA, support the use of these filters as QC samples. Elemental fractionation is confirmed, thus the certified values of the NIES Vehicle Exhaust Particulates do not always apply to the filter samples. In developing new information values for the filter samples, inconsistent XRF concentrations for Al, La, Mn, and Sb were treated as outliers. These values were often very close to detection limits, showing gross discrepancies from the certified values (Vehicle Exhaust Particulates), discrepancies from NAA values, and inconsistences between filters.

Information values for the filter samples (Tables 6 and 7) were calculated with outliers removed. Because of the small number of filters analyzed, no attempt was made to certify a value, rather, it is recommended that the range of values reported in the tables be used as one guide in evaluating the performance of laboratories participating in the CRL.

Preparation of Filter Sets

In all, 25 filter sample sets and 50 blank (oiled) filters were prepared. Participating laboratories were sent a set of two filter samples and two blank (oiled) filters. The filters were packaged in special plastic holders (Petrislides™, Millipore Corp., Bedford, MA) and shipped in padded cardboard boxes. Participating laboratories were made aware of the interlaboratory evaluation, aerosol size configuration and weights. Because no information was provided on expected elemental concentrations, participants were provided with single blind samples.

Table 2. Elemental Concentrations for Fine loaded Filter Samples To Be Used in Determining an 'Information Value'*

Element	**X-Ray Fluorescence**			**Neutron Activation Analysis**		
	Filter 1	**Filter 2**	**Filter 3**	**Filter 1**	**Filter 2**	**Filter 3**
Al	**BDL**	**BDL**	**BDL**	**1278±20**	**1443±23**	**1446±23**
As	**BDL**	**BDL**	**BDL**	**1.70±0.3**	**1.44±0.3**	**1.71±0.3**
Br	**42±7**	**40±9**	**44±9**	**41±3**	**43±3**	**45±3**
Ca	**3898±441**	**3252±370**	**3729±426**	**3331±343**	**3397±358**	**4283±437**
Cl	**BDL**	**BDL**	**BDL**	**378±22**	**427±25**	**437±25**
Cr	**25±6**	**31±7**	**28±8**	**ND**	**ND**	**ND**
Cu	**91±8**	**96±9**	**104±9**	**76±8**	**123±10**	**69±9**
Fe	**2601±137**	**2794±149**	**2648±144**	**ND**	**ND**	**ND**
K	**522±65**	**530±67**	**525±69**	**ND**	**ND**	**ND**
La	**1509±1141**	**BDL**	**BDL**	**0.83±0.16**	**0.54±0.15**	**0.73±0.16**
Mn	**16±7**	**BDL**	**BDL**	**34±1**	**39±1**	**37±1**
Na	**ND**	**ND**	**ND**	**1852±177**	**1797±172**	**1559±149**
Pb	**177±28**	**172±32**	**185±36**	**ND**	**ND**	**ND**
S	**16103±1822**	**16850±1831**	**16063±1831**	**ND**	**ND**	**ND**
Sb	**171±148**	**BDL**	**BDL**	**5.35±0.43**	**4.42±0.37**	**5.02±0.41**
Si	**7653±1021**	**5782±774**	**5586±750**	**ND**	**ND**	**ND**
Sm	**ND**	**ND**	**ND**	**0.068±0.014**	**0.081±0.019**	**0.11±0.02**
Sr	**47±11**	**57±13**	**52±16**	**ND**	**ND**	**ND**
Ti	**126±11**	**124±12**	**140±14**	**82±16**	**101±20**	**95±19**
V	**BDL**	**BDL**	**9±8**	**13±0.4**	**13±0.4**	**14±0.4**
Zn	**343±19**	**357±21**	**343±20**	**ND**	**ND**	**ND**

Notes: **BDL** **Below Detection Limit**
ND **Not Determined**
***** **Values presented in micrograms per gram (µg/g).**

Table 3. Elemental Concentrations for Coarse Loaded Filter Samples To Be Used in Determining an 'Information Value'*

Element	X-Ray Fluorescence			Neutron Activation Analysis		
	Filter 1	Filter 2	Filter 3	Filter 1	Filter 2	Filter 3
Al	368±139	286±121	BDL	1954±28	2122±32	2133±30
As	BDL	BDL	BDL	0.77±0.23	2.03±0.37	2.18±0.38
Br	52±5	62±6	56±6	51±3	51±4	50±3
Ca	5168±581	4628±521	4334±443	4233±443	5019±529	4606±480
Cl	BDL	BDL	BDL	402±29	519±34	416±30
Cr	26±4	21±5	22±5	ND	ND	ND
Cu	159±9	74±6	78±6	78±10	102±12	84±9
Fe	3630±185	3618±186	3229±166	ND	ND	ND
K	742±86	736±86	652±77	ND	ND	ND
La	BDL	BDL	BDL	1.03±0.16	1.21±0.20	1.00±0.17
Mn	13±4	24±5	8±5	44±l	52±2	48±1
Na	ND	ND	ND	1385±134	1611±156	1738±168
Pb	176±18	202±21	195±21	ND	ND	ND
S	17339±1949	17510±1971	17042±1918	ND	ND	ND
Sb	BDL	BDL	162±100	4.12±0.33	4.99±0.40	4.62±0.37
Si	12790±139	10074±1340	9324±1240	ND	ND	ND
Sm	ND	ND	ND	0.118±0.018	0.130±0.02	0.092±0.017
Sr	75±8	65±8	61±8	ND	ND	ND
Ti	198±13	209±13	196±13	132±20	216±22	182±21
V	BDL	11±5	11±5	13±0.4	15±0.5	15±0.5
Zn	436±23	408±22	380±20	ND	ND	ND

Notes:	BDL	Below Detection Limit
	ND	Not Determined
	*	Values are presented in micrograms per gram (µg/g).

INTERLABORATORY EVALUATION

Standardized Reported Values

Table 4 lists the 9 respondents from the original 21 invited participants. The reported values are standardized as micrograms per gram (μg/g) based on loading weights measured prior to filter shipment. A number of the participants reweighed the filters upon receipt, and overall there was no appreciable loading loss. Differences of < 5 percent could be attributed to humidity and the use of a different scale. Two of the participating laboratories reported losses > 5 percent (see notes of Tables 6 and 7).

The standardized values are blank corrected. Corrections were made for each laboratory based on blank values provided by that laboratory (Table 5), or if no blank values were provided, from blank values determined with the initial information values (Table 1). One laboratory (Lab 8) provided blank corrected data. The wide variability in blank value concentrations is of some concern, however, the blank values are all well below loading values and will only affect precision by a few percent. Exceptions are noted for Labs 3 and 4, where blank values often exceeded the loading values (i.e., Lab 3 values for Ca, Cr, and Fe). The high blank values from these laboratories suggest problems with their analysis, not with the blank filters.

Evaluation of Reported Values

Laboratory performance was evaluated by comparison with the 'information values' and on interlaboratory comparisons. The small number of filters analyzed and the lack of a well-characterized 'true value' precluded any detailed statistical treatment of the reported values. Reported values are presented in Tables 6 and 7 (plotted values for some elements in Appendix II), and each laboratory is given a subjective evaluation (ranking) from excellent to poor.

Overall, the participating laboratories showed good agreement, with the exception of a few elements for each laboratory, as described below:

Lab 1 PIXE	Ranking: Excellent. Reported values are in agreement with values reported by most participants, with the exception of fine Pb.
Lab 2 PIXE	Ranking: Very Good. Reported values are in agreement with values reported by most participants, with the exception of fine Al and Cu, and coarse Si and V.

Table 4. IAEA Participants

ID #	Method	Institution/Country	Contact
Info.	NAA XRF	University of Illinois Dept. of Nucl. Eng./ USA NEA, Inc. / USA	S. Landsberger J. Cooper
Lab 1	PIXE	Shanghai Institute of Nuclear Research/ China	Li Mingian
Lab 2	NAA & PIXE	Institute of Low Energy Nuclear Physics/ China	Wang Xinfu
Lab 3	NAA & ED-XRF	Bhabha Atomic Research Centre/ India	B.S. Negi
Lab 4	NAA	Instituto De Pesquisas Energeticas E Nucleares/ Brazil	C.S. Munita
Lab 5*	PIXE	Laboratorio Nacional De Engenharia E Tecnologia Industrial/ Portugal	M.A. Reis
Lab 6	NAA	Nuclear Research Institute/ Czechoslovakia	J. Kucera
Lab 7	NAA	Nuclear Research Institute/ Vietnam	T. Van Luyen
Lab 8	NAA & PIXE	Instituut voor Nucleaire Wetenschapen/ Belgium	W. Maenhaut
Lab 9	NAA	University of Illinois Dept. of Nucl. Eng./ USA	S. Landsberger

*not from the original solicitation

Table 5. Blank Values as Reported by Participants

Elem.	Lab 1		Lab 3		Lab 4		Lab 6		Lab 9	
Ag							<0.0005	< 0.0005		
Al					1.26	0.93	0.55	0.43	0.185	0.10
Aa					< 0.005	<0.005	0.002	0.001	< 0.0006	< 0.0006
Au										
Ba							0.074	0.097	<0.02	<0.02
Br			< 0.005	< 0.005	0.01	0.008	0.004	0.004	<0.001	<0.0007
Ca	0.06	0.03	0.98	1.03	7.30	12.40	<1	<1.5	0.164	0.251
Cd							<0.002	< 0.002		
Ce							0.002	0.001	<0.01	<0.008
Cl			<0.32	<0.32	1.44	<0.74			0.056	0.054
Co					0.007	0.007	0.0006	0.0005	0.009	0.008
Cr			0.005	0.005	0.034	0.043	0.0046	0.0024	<0.02	<0.016
Cs							0.0002	< 0.0001	0.003	0.002
Cu	<0.01	<0.01	0.08	0.09			<0.04	<0.04	0.035	0.054
Dy							< 0.00006	< 0.00007		
Eu							0.0002	0.0003	0.001	0.001
Fe	0.20	0.05	<2.82	<2.82	1.31	3.50	1.3	1.0	1.1	<0.9
Ga										
Hf							<0.0001	< 0.0001	<0.001	1
Hg							0.0095	0.0097		
I							0.0064	0.0061	< 0.0003	<0.0002
In							<0.0002	<0.001		
K	<0.08	<0.08					0.653	0.378		
La			<0.001	< 0.001			0.00046	0.00026	< 0.0005	< 0.0003
Lu									< 0.0007	<0.0006
Mg										
Mn	<0.02	<0.02	1.49	0.99	0.01	0.029	0.066	0.61	0.009	0.003
Mo							< 0.0059	< 0.0068		
Na			0.78	0.51	0.225	0.203	0.213	0.148	0.069	0.041
Nb										
Ni							0.068	0.034	<0.04	<0.04

Table 5. Concluded...

	Lab 1		Lab3		Lab4		Lab6		Ub 9	
p										
Pb	<0.07	<0.07	0.71	0.71						
Rb							< 0.005	< 0.005	<0.05	<0.04
S	<0.32	<0.32								
Sb			< 0.008	< 0.008	0.0005	0.0005	0.005	< 0.005	0.0003	0.0002
Sc			0.002	0.002	0.0001	0.0001	0.0001	< 0.00006	0.0002	0.0001
Se							0.0017	0.0012	0.008	0.014
Si										
Sm			< 0.0002	< 0.0002			0.00014	0.00003	< 0.00006	< 0.00005
Sn										
Sr							<0.06	<0.07		
Ta							< 0.00011	< 0.0001	<0.0009	<0.0007
Th							< 0.0001	< 0.00008	< 0.0008	< 0.0007
Ti							<0.18	<0.20	0.024	0.021
U							<0.00016	<0.00015		
V							<0.001	< 0.001	< 0.0002	<0.0001
W					< 0.004	0.001	<0.0013	<0.0013		
Zn	<0.01	<0.01	<0.23	<0.23	0.99	1.27	0.658	0.174	0.10	0.08

Notes: Laboratories 2,5,7, and 8 did not report separate blank values.
Blank values are given in micrograms (µg) per filter.

Lab 2 NAA	Ranking: Excellent. Reported values are in agreement with values reported by most participants, with the exception of coarse Fe.
Lab 3 NAA/ ED-XRF	Ranking: Fair/Poor. Reported values are most often not in agreement with values reported by most participants. Discrepancies are noted for fine and coarse C1, Cr, Mn and S, and fine Fe, La, and Sb.
Lab 4 NAA	Ranking: Good. Reported values are most often in agreement with values reported by most participants with the exception of fine Al, Cl, and Zn, and coarse Fe and Zn. Some problems exist with blank values for Ca, Cl, Cr, and Fe.
Lab 5 PIXE	Ranking: Good. Reported values are most often in agreement with values reported by most participants, with the exception of fine and coarse Br, P, S, and Si. Elements Se and Ta (reported as information values) are reported at higher concentrations than by most other participants.
Lab 6 NAA	Ranking: Very Good. Reported values are in agreement with values reported by most participants, with the exception of fine Zn and coarse Ca and Mn.
Lab 7 NAA	Ranking: Fair. Reported values are most often not in agreement with values reported by other participants. Discrepancies were found for fine As, Cs, K, La, Na, Sm, and Sr, and coarse Br, Cr, In, K, and Sm.
Lab 8 NAA/ PIXE	Ranking: Excellent. Reported values are in agreement with values reported by most participants and between in house NAA and PIXE, with the exception of fine Br and coarse Mg.
Lab 9 NAA	Ranking: Excellent. Reported values are in agreement with values reported by most participants, with the exception of Cu and Zn.

CONCLUSION AND RECOMMENDATIONS

Participation in this and other interlaboratory comparisons is one of the best ways to identify problems and improve the overall quality of the chemical data. This report concerns two aspects of an interlaboratory evaluation: the preparation of the filter standards and the performance of the participating laboratories. The filter standards were successfully prepared and deposits demonstrated to be homogeneous both on and between filters. The oiling has minimized loading losses from handling. In general, participant performance, based on a 'loose' true value (information value provided) and a comparison of participants, showed values - with exceptions — to be within traditional errors (10 to 30 percent) expected for each technique. Exceptions included problems with individual elements, each of which have been discussed.

Based on this interlaboratory comparison, a number of recommendations are made to improve future works:

1. The authors were disappointed with the poor response (< 50 percent) to this interlaboratory evaluation. As interlaboratory evaluations are one of the best ways to identify problems and improve the quality of the chemical data, a second evaluation is recommended.

2. Variabilities in filter blank concentrations were of concern. Participating laboratories need to be provided with more blank filters, perhaps five, to enable a better characterization of the blank values.

3. Some criticism was received on the use of Teflon^R filters. While Teflon^R filters can be analyzed by PIXE, NAA, and XRF, they are not considered the 'best' filter medium for PIXE. Future interlaboratory comparisons should use Nucleopore filters.

4. The NIES (Vehicle Exhaust Particulates) standard is suitable for the development of filter standards. It is easily suspended and provides detectable concentrations for a large suite of elements —considerations necessary for any future interlaboratory comparison. Other materials, perhaps geological, are also available.

5. Participants reported values and provided methodologies/information in a wide range of formats and detail. To enhance comparability, it is suggested that one format be prescribed for the data and a detailed description of methodologies be requested.

6. Providing a single fine and coarse loaded filter doesn't allow a distinction between consistent or one-time errors. The number of filters provided each laboratory should be increased from one to at least three.

Table 6. Reported Fine Filter Loadings (μg/g)

Element	Information	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5
	NAA &XRF	PIXE	PIXE	NAA/ ED-XRF	NAA	PIXE
Ag						
Al	1278-1446[a]		863±71		688±22	
As	1.44-1.71[a]				1.6±0.6	
Au						
Ba						88±8
Br	40-45			31±3	37±4	88±7
Ca	3252-4283	3830±10	2741±40	3120±156	0 (10467)	2944±153
Cd						
Ce						
Cl	378-437[a]			1476±148	11723±458	450±12
Co					4.7±0.5	
Cr	25-3l[b]		<31	8±2	0(52)	20±2
Cs						
Cu	69-123	80±13	< 6	111±11		53±9
Dy						
Eu						
Fe	2601-2794[b]	2320±9	2179±59	5721±572	0 (3382)	1946±91
Ga						
Hf						
Hg						
I						
In						
K	522-530[b]	430±9	379±30			319±14
La	0.54-0.83[a]			4.4±0.4		

Table 6, Continued...

Element	Information	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5
	NAA & XRF	PIXE	PIXE	NAA/ ED-XRF	NAA	PIXE
Lu						
Mg						
Mn	34-39[a]	60±30	<26	2307±231	13.8±0.5	27±3
Mo						
Na	1559-1852[a]			2008±100	1386±80	
Nb						3190±188
Ni						
P			525±27			981±22
Pb	172-185[a]	420±18		277±28		270±16
Rb						
S	16063-16850[b]	16940±861	15460±77	8830±883		11286±508
Sb	4.42-5.35[a]			20±6	5±0.3	
Sc				1±0.3	0.25±0.002	
Se						(25)
Si	5586-7653[b]		4136±99			1791±63
Sm	0.068-0.106[a]			0.74		
Sn						
Sr	4 7-57[b]					(82)
Ta						(25)
Th						
Ti	82-140		142±18			90±7
U						
V	13-14[a]		<38			
W					3±1	
Zn	342-357[b]	410±36	328±47	348±35	837±15	254±28

Table 6. Continued...

Element	Information	Lab 6	Lab 7	Lab 8		Lab 9
	NAA &XRF	NAA	NAA	NAA	PIXE	NAA
Ag		<0.87		<3.5		
Al	1278-1446[a]	2605		1585±179	1175±171	1053±16
As	1.44-1.71[a]	1.6	15±2	0.94±0.18	< 122	
Au			2.8±0.4	4.7±0.2		
Ba		138	122±27	131±19	< 219	62±9
Br	40-45	44	57±3	54±7	117±35	39±3
Ca	3252-4283	3087		4444±456	3837±60	2964±303
Cd		< 3		<7.8		
Ce		2.7	5±0.4	<26		< 10
Cl	378-437[a]			340±46	<332	233±14
Co		1.4	2.4±0.04	2.1±0.2		3.1±0.3
Cr	25-31[b]	20	28±3	16±3	<29	23±6
Cs		<0.3	3.6±0.8	<0.54		0
Cu	69-123	45		134±20	106±12	38±4
Dy		<0.12				
Eu		0.54		<0.23		0.28±0.04
Fe	2601-2794[b]	1838	3154±665	1825±696	2291±379	1889±233
Ga				0.52±0.16		
Hf		<0.2	0.4±0.1			< 1.3
Hg		0	0.2±0.05			
I		2.2		4.2±0.7		2.2±0.4
In		<0.58		0.05±0.01		
K	522-530[b]	630	4581±257	598±31	543±46	
La	0.54-0.83[a]	0.61	3.5±0.4	0.42±0.06		0.81±0.26

Table 6. Continued...

Element	Information	Lab 6	Lab 7	Lab 8		Lab 9
	NAA & XRF	NAA	NAA	NAA	PIXE	NAA
Lu				<0.15		
Mg				< 1589	< 1115	
Mn	34-39[a]	22		26±6	37±8	27±1
Mo		< 19		5.6±1.0	<583	
Na	1559-1852[a]	1229	4164±325	1408±1	<6434	1363±130
Nb						
Ni		0		<50	<25	<51
P					449±113	
Pb	172-185[b]				342±108	
Rb		<7	7±0.6	<26	< 169	<43
S	16063-16850[b]				18556±191	
Sb	4.42-5.35[a]	0	6.3±1	5.0±0.3		3.5±0.4
Sc		0.25	0.4±0.05	0.2±0.02		0
Se		1.7		<7.3	<81	0
Si	5586-7653[b]				6648±150	
Sm	0.068-0.106[a]	0	0.62±0.2	0.08±0.01		0.12±0.03
Sn				< 163		
Sr	47-57[b]	< 116	342±79	78±29	< 215	
Ta		<0.17				<0.9
Th		0.26		0.39±0.13	<397	<0.8
Ti	82-140	<405		< 201	90±21	73±16
U		<0.33	6.3±1			
V	13-14[a]	10		12±0.8	<36	11±0.4
W		3.3		3.2±0.2	3±1	
Zn	342-357[b]	1149	514±5	344±28	382±18	249±12

Table 6. Concluded...

Notes: Reported values are blank corrected and based on initial weighings (see text).
[a] NAA only [b] XRF only

Lab 1:	Reweighing shows loading loss of 2 percent, errors calculated as a S.D. (n = 3).
Lab 2:	Reweighing shows no loading loss, errors calculated as a S.D. (n = 3).
Lab 3:	Reweighing shows loading loss of 14 percent, errors determined by analysis of Orchard Leaf (SRM-1571) samples.
Lab 4:	Reweighing shows no loading loss, errors calculation not stated. Values in brackets were not blank corrected.
Lab 5:	Filters were not reweighed, errors calculated at 68 percent confidence level. Values in brackets are reported by the laboratory as information only.
Lab 6:	Reweighing shows no loading loss, no error calculation provided.
Lab 7:	Reweighing shows loading loss of 3 percent.
Lab 8:	Reweighing shows no loading loss (NAA) and loading loss of 4 percent (PIXE), errors calculated as analytical S.D. (including the error from blank variability).
Lab 9:	Filters were not reweighed.

Table 7. Coarse Filter Loadings (μg/g)

Element	Information	Lab 1	Lab 2		Lab 3	Lab 4
	NAA &XRF	PIXE	PIXE	NAA	NAA/ ED-XRF	NAA
Ag						
Al	1954-2133[a]		1077			1934±45
As	2.03-2.18[a]			2.89±0.49		3.4±0.3
Au				0.03±0.01		
Ba				122±22		
Br	50-62			33±1	29±3	40±1
Ca	4233-5168	3926±47	4500±90		3390±170	0 (8459)
Cd						
Ce				5±0.4		
Cl	402-519[a]				883±88	0
Co						0.84±0.06
Cr	21-26[b]		20	16±1	2±0.5	31±3
Cs				0.53±0.05		
Cu	74-159	53±8	64±43		79±8	
Dy						
Eu				0.07±0.01		
Fe	3229-3630[b]	2674±25	3059±3	7070±247	4697±470	1001±83
Ga						
Hf				0.40±0.08		
Hg				2.6±0.3		
I						
In						
K	652-742[b]	576±10	595±3	1183±97		
La	1.00-1.21[a]			2.2±0.2	3.9±0.4	

Table 7. Continued...

Element	Information	Lab 1	Lab 2		Lab 3	Lab 4
	NAA & XRF	PIXE	PIXE	NAA	NAA/ ED-XRF	NAA
Lu						
Mg						
Mn	44-52[a]	63±26	38±11		1578±158	31±1
Mo				7.1±0.9		
Na	1385-1738[a]			1079±37	1578±158	1464±57
Nb						
Ni						
P			373±198			
Pb	176-202[b]	227±14			170±17	
Rb						
S	17042-17516[b]	15330±394	15538±47		7025±703	
Sb	4.12-4.99[a]			4.9±0.2	7±2	3.6±0.3
Sc				1.2±0.04	1.2±0.4	0.3±0.03
Se						
Si	9324-12790[b]		5550±100			
Sm	0.092-0.13[a]			0.33±0.01	0.59	
Sn						
Sr	61-75[b]					
Ta						
Th				0.9±0.07		
Ti	132-216		256±24			
U						
V	11-15		71 ± 11			
W				2±0.1		4.4±0.4
Zn	380-436[b]	349±57	423±46	389±15	423±42	611±11

Table 7. Continued...

	Information	Lab 5	Lab 6	Lab 7	Lab 8		Lab 9
	NAA &XRF	PIXE	NAA	NAA	NAA	PIXE	INAA
Ag			<0.52		< 1.7		
Al	1954-2133[a]		1245		1680±91	1377±76	1403±20
As	2.03-2.18[a]		1.6		1.23±0.09	<48	1.1±0.3
Au				1.3±0.2	0.06±0.002		
Ba			40	64±12	104±13	<87	43±13
Br	50-62		43	20±2	47±4	<40	31±2
Ca	4233-5168	3735±123	1717		4432±52	4797±31	3807±394
Cd			<2		<3.8		
Ce			2.1	0.89±0.09	<4		<7
Cl	402-519[a]	496±42			291±23	263±42	272±21
Co		3±1	1.5	1.7±0.05	1.7±0.2		1±0.08
Cr	21-26[b]	22±3	15	5.6±0.8	16±1	22±4	17±4
Cs			0.08	3.1±0.9	<0.3		1.4±0.2
Cu	74-159	55±5	53		89±13	59±5	35±5
Dy			<0.05				
Eu			0.15		< 0.12		0.02±0.01
Fe	3229-3630[b]	2676±94	2148	2367±410	2443±347	2617±138	1963±216
Ga					0.76±0.09	< 16	
Hf			<0.1				<0.84
Hg			1	0.13±0.04			
I			1.7		3.4±0.4		2±0.5
In			<0.2	53±22	0.04±0.006		
K	652-742[b]	457±14	572	2812±157	559±17	579±20	
La	01.0-1.21[a]		0.59		0.57±0.04		1.3±0.2

Table 7. Continued...

	Information	Lab 5	Lab 6	Lab 7	Lab 8		Lab 9
	NAA & XRF	PIXE	NAA	NAA	NAA	PIXE	NAA
Lu					<0.07		<0.43
Mg					900±183	482±141	
Mn	44-52[a]	34±6	2		31±3	41±4	32±1
Mo			< 8		6.2±0.4		
Na	1385-1738[a]		1059	1027±52	1218±31	<2532	1096±107
Nb		(1672)					
Ni			10		<25	< 9	<33
P		853±78				336±44	
Pb	176-202[b]	262±29				138±41	
Rb			< 4		< 14	<64	<31
S	17042-17510[b]	12988±584				14220±79	
Sb	4.12-4.99[a]		3.7	3.6±0.5	5.2±0.3		2.6±0.3
Sc			0.31	0.26±0.03	0.28±0.01		0.35±0.03
Se		50±15	0.48	0.89±0.33	< 4	<29	0
Si	9324-12790[b]	3512±344				6485±69	
Sm	0.092-0.13[a]		0.07	1.7	0.12±0.01		0.12±0.02
Sn					91±21		
Sr	61-75[b]	151±35	<52	148±52	61±16	137±29	
Ta		(33)	<0.09				<0.58
Th			0.34	0.18±0.06	0.25±0.06	< 146	<0.59
Ti	132-216	140±2	< 186		168±25	147±9	127±17
U			<0.22				
V	11-15		8.9		12±0.8	< 15	10.3±0.3
W			4.2		4.9±0.3		
Zn	380-436[b]	334±9	435	324±98	361±23	297±9	172±7

Table 7. Concluded...

Notes: Reported values are blank corrected and based on initial weighings (see text).

[a] NAA only [b] XRF only

Lab 1: Reweighing shows loading loss of 2 percent, errors calculated as a S.D. (n = 3).

Lab 2: Reweighing shows no loading loss, errors calculated as a S.D. (n = 3).

Lab 3: Reweighing shows loading loss of 14 percent, errors determined by analysis of Orchard Leaf (SRM-1571) samples.

Lab 4: Reweighing shows no loading loss, errors calculation not stated. Values in brackets were not blank corrected.

Lab 5: Filters were not reweighed, errors calculated at 68 percent confidence level. Values in brackets are reported by the laboratory as information only.

Lab 6: Reweighing shows no loading loss, no error calculation provided.

Lab 7: Reweighing shows loading loss of 10 percent.

Lab 8: Reweighing shows no loading loss (NAA) and loading loss of 4 percent (PIXE), errors calculated as analytical S.D. (including the error from blank variability).

Lab 9: Filters were not reweighed.

REFERENCES

Dzubay, T.G. and R.K. Barbour, 1983. A Method to Improve the Adhesion of Aerosol Particles on Teflon Filters. Journal of the Air Pollution Control Association. 33(7), 692-695.

Gatz, D.F., M.V. Miller, G.J. Stensland, and L.-C. Chu, 1986. Evidence of Elemental Fractionation in Fine Particle Size Classes of Highly Erodible Soils. Proceedings of an APCA International Specialty Conference "Particulate Matter/Fugitive Dusts: Measurement and Control in Western Arid Regions," Tucson, AZ, October 1986.

Okamoto, K., 1987. A New Certified Reference Material, Vehicle Exhaust Particulates. Analytical Sciences. 3,191-192.

Schutz, L. and K.A. Rahn, 1982. Trace-Element Concentrations in Erodible Soils. Atmospheric Environment. 16, 171-176.

Sweet, C.W. and D.F. Gatz, 1987. Atmospheric Research and Monitoring Study of Hazardous Substances: Third Annual Report, HWRIC RR022. Illinois Hazardous Waste Research and Information Center, Champaign, IL.

Van Borm, W.,T. Keersmaekers, and F. Adams, 1988. Characteristics of Resuspended Soil Particles with High Concentrations of Cu, Zn, Cd, and Pb as a Function of Particle Size. Journal of Aerosol Science. 19(7), 1287-1289.

Vermette, S.J.,A.L. Williams, and S.Landsberger, 1990. Surface Dust Elemental Profiles - Southeast Chicago (Lake Calumet and McCook Areas). State Water Survey Contract Report 488, Champaign, IL.

Appendix I
Laboratory Methodologies

Each participating laboratory was asked to submit a description of methodologies. The descriptions provided allow for comparisons between laboratory protocols and instruments which may be useful in interpreting the elemental determinations provided. Each description has undergone minimal editing and no attempt has been made to explain technical acronyms or give a theoretical description of each technique.

Laboratory 1 (PIXE)

A 3.1 Mev proton beam was focused to 6 mm in diameter, hitting the filter with a 6-8nA beam current. The target chamber was maintained in a low vacuum (about 0.001 torr). Every spectrum of a filter has been accumulated to 2000 seconds. Data handling was performed with the AXIL program. The elemental concentrations of the blind samples were determined by comparing the spectra with those of Micromatter X-ray Fluorescence Calibration Standards from American Micromatter Co., which were used as standards in the same experiment condition.

Laboratory 2 (PIXE)

The element analysis of the QC samples was carried out on 2 x 1.7 Mv tandem accelerator at Beijing Normal University. A 2.5 Mev proton beam at a current between 2 and 5 nA was used. The beam spot on samples was 3.5 mm in diameter. The defocusing film in front of the collimater was 6 μm thick. In order to reduce background caused by bremsstrahlung and increase sensitivity of some elements in the low energy section, a 200 μm thick polyethylene film with an orifice of 2.9 mm in diameter was placed between the target and the Si(Li) detector.

In order to assure accuracy of measured results, calibration of the sensitivity curve for the measurement system was made using thin Micromatter Standards prepared on Mylar backing of 3.5 μm thickness (American Micromatter Co.). The lower limits of detection (LLD) for the different elements were calculated under our experimental conditions. The LLD is the amount of an element in $\mu g/\mu c/cm^2$ of the air particulate filter which will yield an X-ray intensity equal to three times the square root of the blank value in an interval of FWHM of the peak. The two X-ray spectra of QC sample (12F), which were obtained under proton energy of 2.5 and 3.0 Mev, respectively, were compared. In order to make analytical results approach reality and to check the homogeneities of the QC samples, three different portions of QC samples 12F and 12C were analyzed.

Laboratory 2 (NAA)

Filters were carefully removed from the polyethylene (PE) support rings and folded into a pure aluminum film. We have used NBS SRM 1633a and Chinese SRM GSD-12, HAIR-1 as a set standard. As NBS SRM 1633a is a good multielement comparison standard for geochemical analysis, we have used another batch number of the NBS SRM 1633a (46.358 mg) as a quality control. The filters and standards, as well as Zr and quantitative Au, were irradiated together with a thermal neutron flux of about 4.96 x 10^{13} n/cm^{-2}/s^{-1} in a container at the reactor for 6 hours — Zr as a flux ratio monitor and Au as a comparison.

Prior to radioassay, the samples were transferred into a measuring box. The gamma ray spectrum of the samples was radioassayed using a computer-multichannel analyzer (Canberra) program control system. FWHM of detecting system for the HIGe detector was 1.9 kev at 1332 kev. The samples were radioassayed the 4th and 5th day following irradiation. The element concentrations were calculated by relative comparison methods and a Ko standardization comparison method.

Laboratory 3 (NAA)

Filters were sealed in PE envelopes and irradiated for 10 hours in a swimming pool reactor. The neutron flux was 9.45 x 10^{11} as measured by the copper flux monitors irradiated along with the filter samples. To determine the accuracy of the results, Orchard Leaf samples (SRM-1571) were also irradiated with the filters. The element concentrations were evaluated from the photopeak counts of gamma rays, neutron flux and the basic nuclear data available in the literature. An HpGe detector spectrometer with photopeak efficiencies varying from 15 to 1.3 percent, for gamma ray energies of 100 to 1700 Kev was used along with a 4 K ADC coupled to an Apple computer-based MCA.

Laboratory 3 (ED-XRF)

The system used was a Si(Li) detector spectrometer having a maximum photopeak efficiency of 2 percent. Since the samples deposited on Teflon^{R} filters are in the form of a very thin film, they can be considered as thin samples. In fact, it was seen that there was no absorption of low energy x-rays (S x-rays) in this matrix. Therefore no absorption correction was applied. The concentrations were evaluated using thin film standards of known weights. The accuracy of measurements was evaluated by analyzing Orchard Leaf (SRM-1571) samples. The minimum detection level (MDL) for each element was evaluated from counts under the photopeaks, and they do not represent the absolute MDL.

Laboratory 4 (NAA)

Filter rings were cut using stainless steel scissors which left approximately a 1 mm border ring on each filter. For the analysis of the elements giving rise to short-lived nuclides, the samples and blanks were packed in PE bags and irradiated for 5 min using a pneumatic rabbit station of a swimming pool type research reactor with a neutron flux of about 10^{12} n/cm^{-2}/s^{-1}. The filters were irradiated for 8 hours to determine elements with long-lived radionuclides. Measurements were carried out using a Ge(Li) detector with resolutions of

2.4 KeV at the 1332 KeV gamma peak of ^{60}Co, coupled to a 4096 channel gamma spectrometer ORTEC, an a minicomputer.

The standards used in these analyses were prepared by pipetting suitable aliquots of standard solutions obtained by dissolution of high degree purity metal or oxide of the elements on pieces of Whatman 41 filter paper.

Laboratory 5 (PIXE)

A 1.7 MeV proton beam and a current density of 300 nA/cm^2 were used. The beam incidence angle was 52.5° relative to the normal target surface. The Si(Li) detector had a resolution of 200 eV for Fe-Ka and was placed at 90° to the beam direction. Each spectrum was collected up to 100 μC, and three spots were analyzed for each target. The detection limits were defined as three times the square root of the background taken in an interval of 4 FWHM centered in the value of energy of the characteristic line of the element reference.

Laboratory 6 (NAA)

The PE support rings were cut off by scissors, and the Teflon^R filters were sealed in cleaned PE bags for irradiation. The samples and synthetic standards (prepared by pipetting aliquots of solutions containing known amounts of elements onto PE discs and subsequent drying) in PE bags were wrapped in aluminum foil and simultaneously subjected to long-time irradiation (a pneumatic facility for short-time irradiation was not working). Irradiation time in a thermal neutron flux of 6 x 10^{13} n/cm^{-2}/s^{-1} was 8 hours. After three days of decay, the PE capsules with the samples and standards were cleaned on their surface by wiping with water and ethyl alcohol, and counted with a coaxial HPGe detector (Schlumberger, relative efficiency 23 percent, resolution FWHM 1.9 keV for the 1332.5 keV photons of ^{60}Co) coupled to a Nuclear Data ND-76 multichannel analyzer.

A second count was done after one month of decay for 4 hours, again in the 2 cm distance from a cap of the detector. Data reduction was accomplished with a PDP 11/73 computer using an adapted ND software package for NAA. It was impossible to remove the samples from the PE packaging bags for irradiation (the filters contracted on irradiation). Thus, blank values originating from the PE irradiation bags had to be subtracted for several elements.

After an additional two month decay (when the pneumatic facility became available), the samples and synthetic standards (still in the PE bags from long-time irradiation) were irradiated for 2 minutes in a thermal neutron flux of 5 x 10^{13} n/cm^{-2}/s^{-1}. The samples and standards were irradiated separately with neutron flux monitors (10 jug of Au). After 5 minutes of decay, the surface-cleaned samples and standards were counted with a coaxial HPGe detector (ORTEC, relative efficiency 11 percent, resolution FWHM 1.75keV for the 1332.5 keV photons of ^{60}Co) coupled to a ND 65 multichannel analyzer controlled by the

PDP 11/73 computer. Counting time was 7 minutes, counting geometry 7 cm. A second count was carried out after a decay time of 20 minutes, for 25 minutes in the 2 cm distance from a cap of the detector. A ND 699 Loss Free Counting module was employed to correct for dead-time and pile-up losses in all counting regimes.

Laboratory 7 (NAA)

The filter support ring was removed and the filter sealed in a charcoal container. Samples were irradiated for 70 hours at a thermal neutron flux of 10^{12} $n/cm^{-2}/s^{-1}$. Samples were cooled 48 hours and transferred to inert vials. Samples were counted for 7200 sec on a low-background Gamma spectrometry system, including a Ge(HP) detector with an efficiency of 15 percent and a resolution of 2 keV at 1332.5 keV of ^{60}Co isotope and the CMTE-MCD interface with IBM-PC/AT. Samples were cooled for one week and counted for 60,000 sec on the above mentioned system.

Laboratory 8 (NAA & PIXE)

A detailed description of methodologies was not provided. It was noted for PIXE that the preset charge was corrected and normalized by using the average PIXE/NAA ratios for the following elements: Al, CI, K, Ca, Ti, Fe, and Zn for the coarse loaded filter; and K, Ca, and Zn for the fine loaded filter.

Laboratory 9 (NAA)

Refer to the methodology description described in our preparation of the information values.

Appendix II
Selected Plots Showing Laboratory Performance

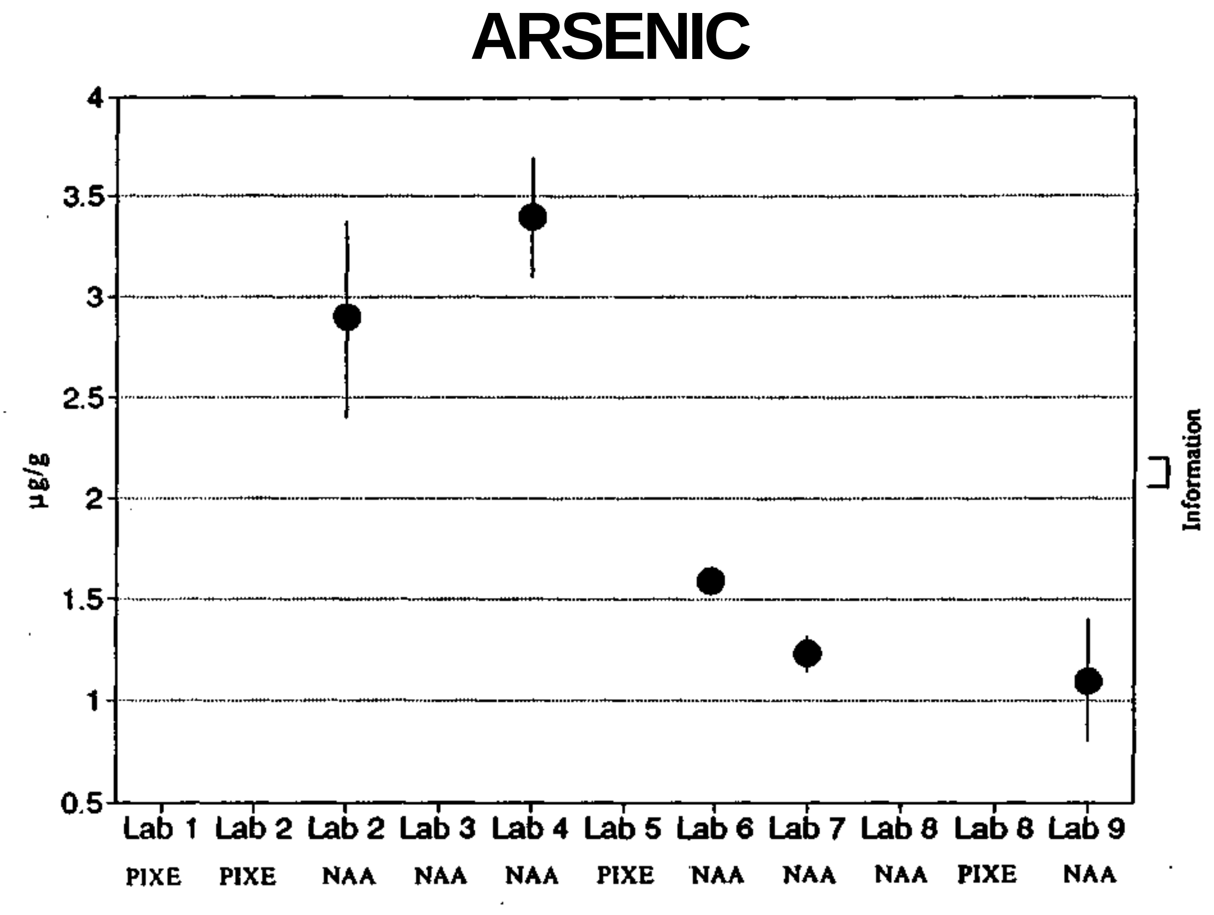

Figure 1A. Arsenic coarse filter loadings (see Table 7).

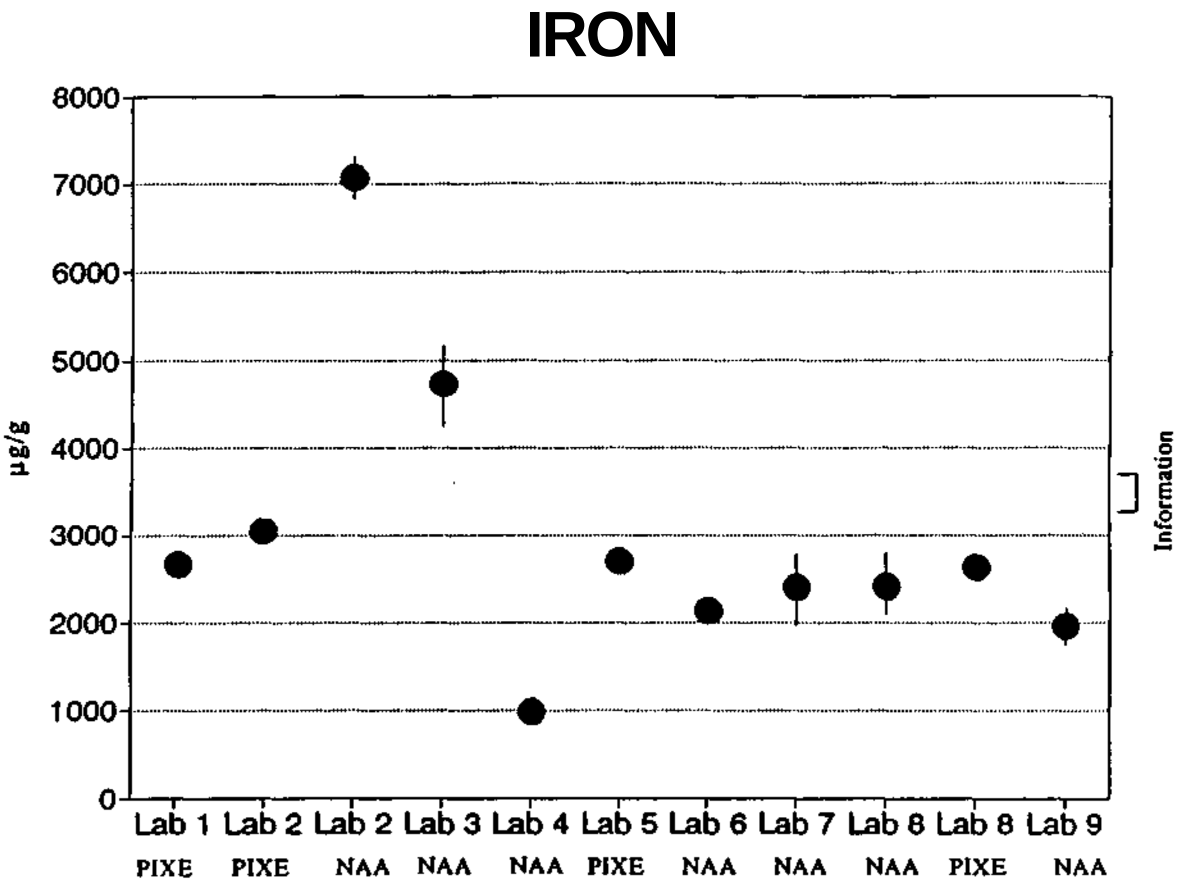

Figure 2A. Iron coarse filter loadings (see Table 7).

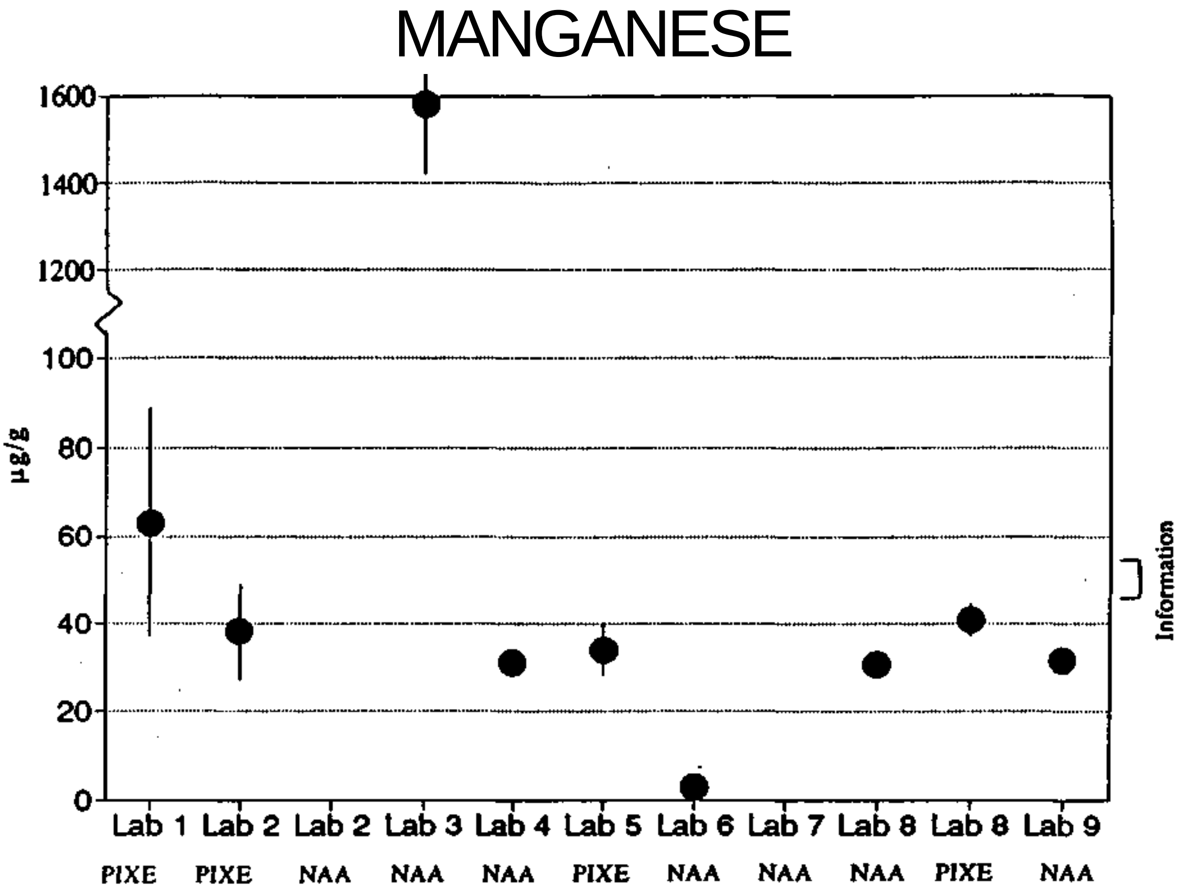

Figure 3A. Manganese coarse filter loadings (see Table 7).

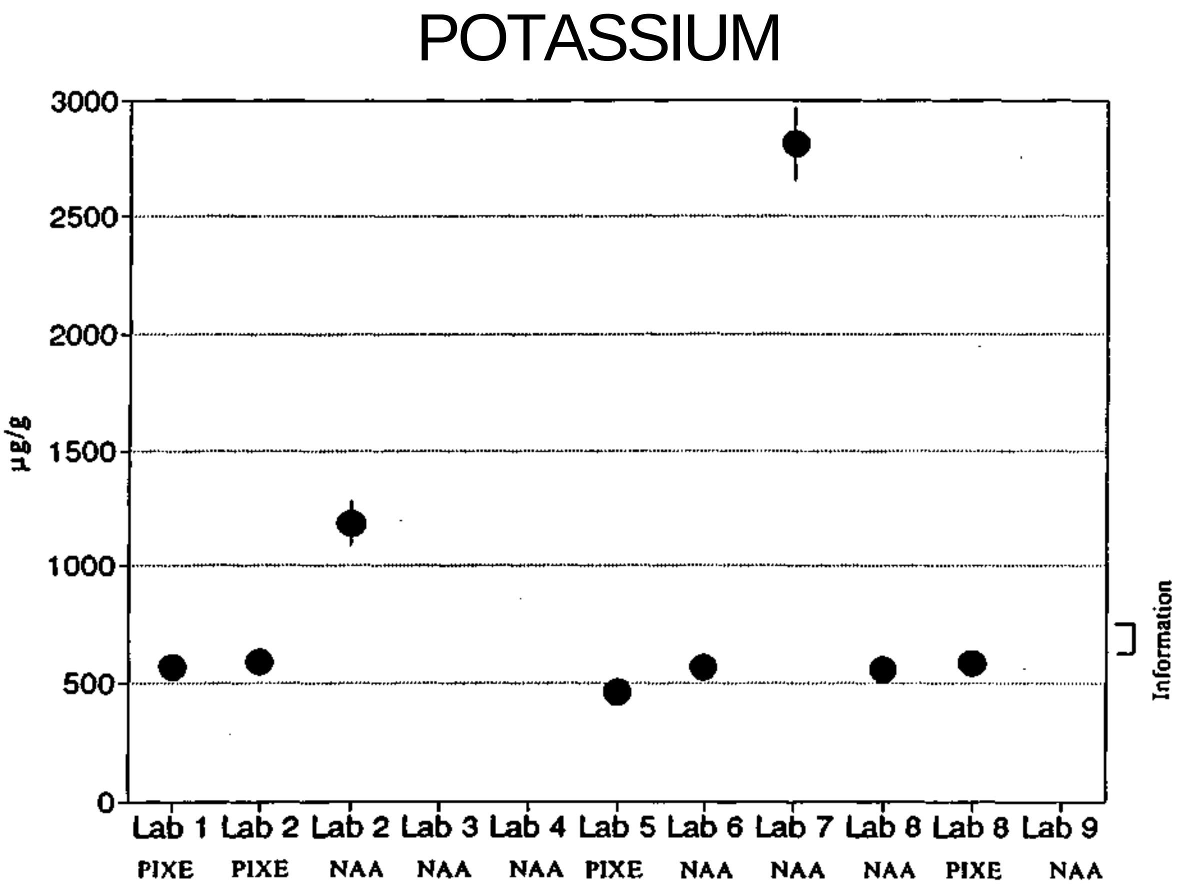

Figure 4A. Potassium coarse filter loadings (see Table 7).

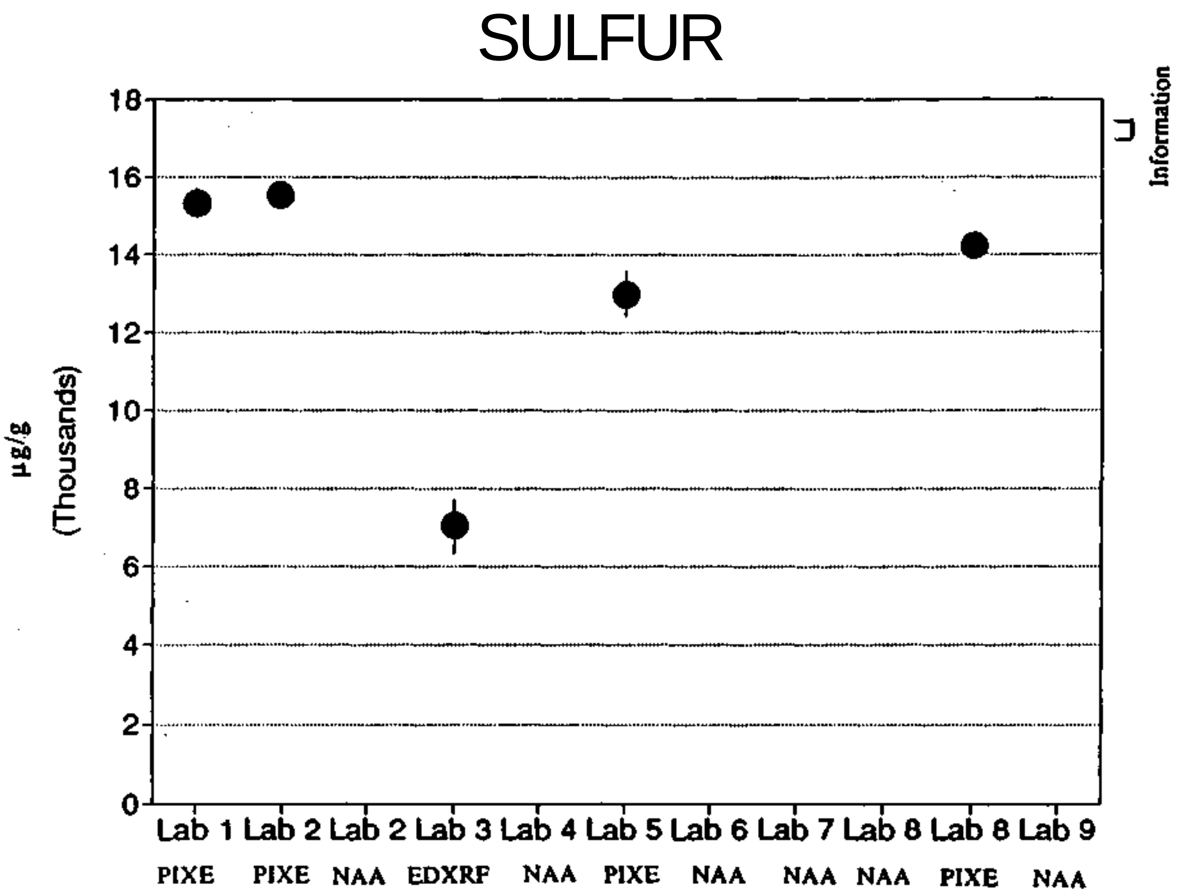

Figure 5A. Sulfur coarse filter loadings (see Table 7).

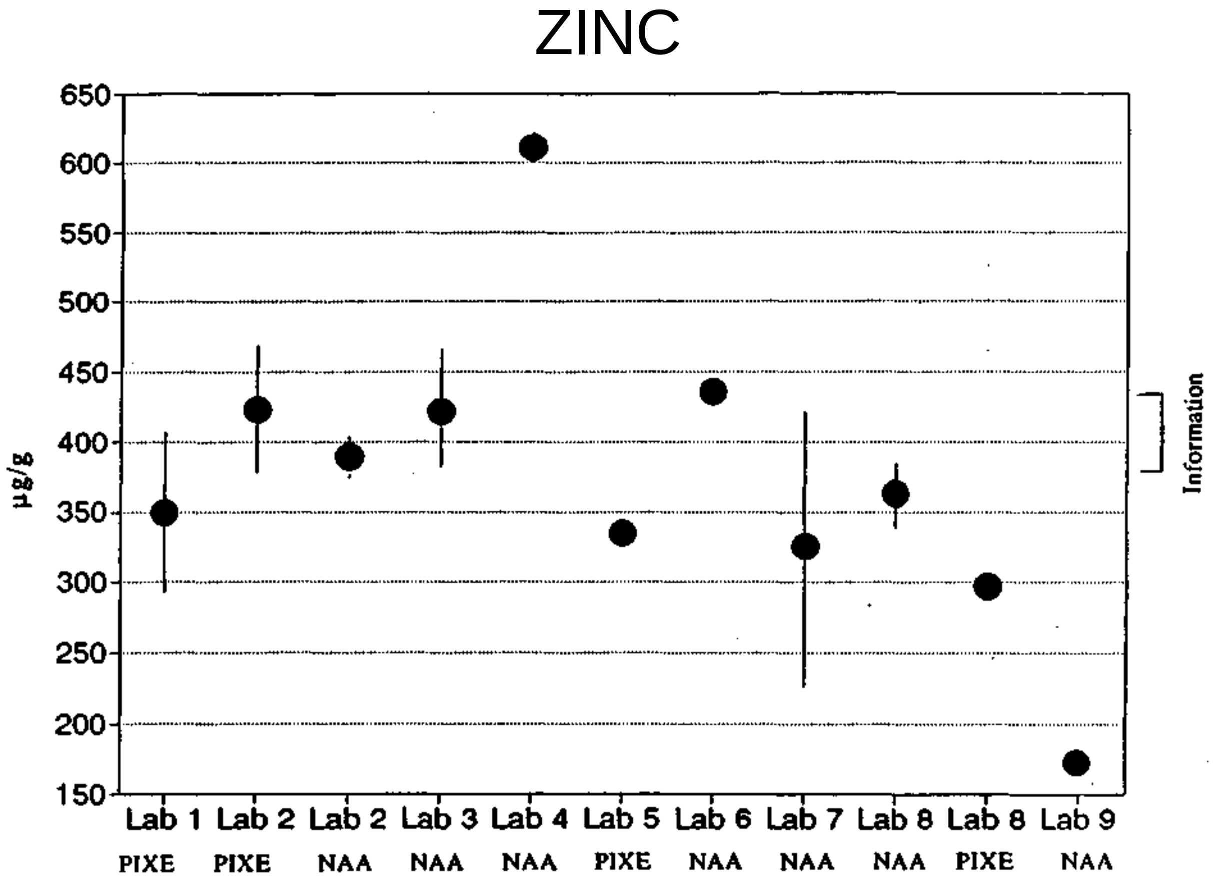

Figure 6A. Zinc coarse filter loadings (see Table 7).