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A remote aerosol sampler to be used with charged-particle X-ray fluorescence analysis

Steven Dee Rowley
Brigham Young University - Provo

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A REMOTE AEROSOL SAMPLER TO BE USED WITH CHARGED-PARTICLE X-RAY FLUORESCENCE ANALYSIS

A Thesis
Presented to the
Department of Chemistry
Brigham Young University

In Partial Fulfillment
of the Requirements for the Degree
Master of Arts

by
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This thesis, by Steven Dee Rowley, is accepted in its present form by the Department of Chemistry of Brigham Young University as satisfying the thesis requirement for the degree of Master of Arts.

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I. INTRODUCTION

With the advent of new technology and its resultant pollution, man has continually been trying to find ways to improve the environment around him. Studies of atmospheric contamination with aerosol trace metals have been made in various areas throughout the world.[1-3] Most studies have been made in areas of easy accessibility. A need for sampling in remote areas with the attendent requirement of developing suitable sampling equipment was recognized at Brigham Young University.

A remote sampler would need to meet the following criteria: (1) Size of the sampling apparatus must be such that it is easily transported to remote areas. (2) It must contain its own power supply. (3) It must be capable of up to three weeks continuous operation in the field. (4) The filter material used must provide good filtering efficiency for all sized particules in the atmosphere. (5) A means to determine the total volume of air filtered must be provided.

This paper reports on the development of a remote aerosol sampler that meets these criteria, and provides particulate samples that yield quantitative data when analyzed by means of charged particle X-ray fluorescence.

II. LITERATURE REVIEW

Work involving aerosol sampling has been going on for several years. R. Spirtas, et al.,[4] report that the National Air Surveillance Network (NASN) has collected samples of suspended particulate matter since 1957 with data being used to determine cycles or patterns with particulate matter vs. time. Others have reported on work being conducted over the past fifteen to twenty years relating to the development of sampling and analytical techniques for the determination of gaseous as well as particulate components in the atmosphere.[3,5-7] In recent years more work has been done in the area of aerosol sampling.[8]

The equipment used in sampling techniques has consisted of some type of holder into which the filter is placed as air is drawn through the filter. Some samplers are constructed so that part of the particulate matter in the atmosphere can be collected before the air is passed through the filter. Sampling techniques now in use involve procedures in which the sample to be analyzed is collected over a 24 hour period with samples in some cases being taken every 2 hours.[4,9-15] In New York City, Kneip and co-workers[12] reported on a sampling system designed to provide weekly rather than daily samples. Recently, H. D. Axelrod, et al.,[13] reported on a remote sampler designed to collect gaseous compounds and particulates in the atmosphere.

The type of sampler most commonly used has been a high volume sampler in which air is pulled through an 8 in X 10 in glass fiber or membrane filter.[3,4,12,15,17] D. A. Lundgren[11] has developed a sampler utilizing four impactor stages followed by a backup filter. It is useful for determining particulate concentrations as a function of both particle size and time. A. A. Anderson[6] has developed a multistage, multijet air sampler which can automatically classify air-bourne particles according to their aerodynamic dimension. It is designed to use a backup filter if desired. R. E. Lee, Jr., et al.,[3] report taking air samples using a NASN sampler that is a modification of the 6 stage Anderson sampler. Anderson 2000 Inc. also manufacture a mini-sampler or personnel monitor similiar in design to the Anderson sampler utilizing 4 rather than 6 stages. This sampler is designed to be worn on the person in and around factories, mines, and other areas. Other designs of sampling apparatus have also been reported.[18]

The filters used in samplers consist of glass fiber, cellulose fiber, membrane, or Nuclepore filters. The glass fiber has routinely been used with high volume sampling using an 8 in X 10 in filter. The size and nature of this filter make it possible to sample at an air flow rate of 50 to 60 cubic feet per minute. Particles as small as about 0.3 μ can be filtered, but the filter itself contains some trace impurities.[19] The other types of filters are commonly used with other sampler designs.

Evaluations of different filtering materials have been made. R. Dams, et al.,[20] have done work evaluating some filter materials for use with nondestructive neutron activation analysis and recommend a Whatman filter. Others have reported using the same type filter in aerosol studies.[17, 27] With the many types of filters on the market, almost any need can be filled to the specifications desired. However, in the past two years it have become widely recognized that the standard

24-hour sampling of 2000 m^3 of air on glass fiber filters needs to be supplemented.[18] The membrane and Nuclepore filters were designed to help fill this need. Criteria for the selection of filter materials have been thoroughly considered by Spurny and co-workers.[22-27] They have done studies on the Nuclepore filter in terms of the structural and filtration properties. They report filter efficiencies for the 0.4 μ Nuclepore filter to be effectivily 100% for particulates ranging from 10 μ down to 10^{-3} μ with exceptions in the .14 to .02 μ diameters where the efficiency drops to about 50%. Spurny and co-workers[1] also list some suggested used of the Nuclepore filter.

For analytical work involving aerosols the Nuclepore filter is an excellent choice. Because of the uniformity of weight, a low area density of less than 1 mg/cm^2, a low level of impurties, and a moderate bremsstrahlung, Walters and co-workers[29] recommend the Nuclepore as the best material to use with X-ray fluorescence analysis.

The use of X-ray fluorescence as an effective means of aerosol analysis has been reported.[15,16,20,26-37]

In addition to requirements for trace metal analysis, the filtering material should allow for the determination of total concentrations of particulate matter in the atmosphere. High volume sampling with the 8 in X 10 in filters has been used most commonly for this purpose. Lee and Goranson[3] report a study in England. Suspended particulate concentrations ($\mu g/m^3$) determined with a cascade impactor were compared with measurements determined with a high volume sampler where the instruments were operated concurrently for 24-hour sampling periods. A total of 101 sample pairs were collected at various sites. The ratio of particulates in $\mu g/m^3$ between the two sampling methods was 0.94 : 1.0 indicating a high degree of association between the methods. In the areas of high particulate concentrations the low cascade impactor value was felt to be the result of wall loss in the impactor. In areas of low particulate concentration the cascade impactor showed more total particulates that the high volume sampler. This was thought to be due to greater efficiency at lower concentrations. Lundgren[9] reports that with the Anderson samplers the wall loss is less than 10%. He also reports that the recovery of particulates with the Anderson sampler was almost perfect because of the construction in which the particulates on each stage can easily be washed off.

Summary of Literature Review

The literature reveals several methods of aerosol sampling and filter use. This author, however, is not aware of any sampling apparatus designed for use in remote areas other than that reported by Axelrod and co-workers.[13] I therefore feel this work has merit and will be of use in future aerosol studies. The work done by others in evaluating filters, comparing methods of total particulate concentrations, and reporting of available equipment have made this work possible.

III. EQUIPMENT AND ANALYSIS RESULTS

A detailed description of the remote aerosol sampler is given in this section. Included are analyses of three separate samples collected on the roof of the Richards Physical Education Building at Brigham Young University, Provo, Utah, which show the validity of the method.

Equipment Used

The sampler head chosen for the sampler was the Anderson 2000 mini-sampler or personnel monitor which requires an air flow of 1.4 ℓ/min. It is composed of 4 brass impaction plates followed by a 25 mm 0.4 μ Nuclepore filter as illustrated in Figure 1. Circles of jets in the plates alternate with circles of deposits whose diameters vary from plate to plate. The cut points for particulate deposition of each stage are: Stage 1, 4.7 μ; Stage 2, 3.3 μ; Stage 3, 2.1 μ; and Stage 4, .65 μ.

Particulate matter was removed from the plates in an ultrasonic cleaner with hexane solvent. The solvent was passed through a 0.4 μ Nuclepore filter supported on a stainless steel screen. The Nuclepore filters were weighed on a microbalance to determine total particulate deposition in preparation for elemental analysis.

The sampler head was mounted on a flexible hose in the lower part of a double roofed, weather bureau-type shelter. The hose was connected to a flow meter used for regulatory purposes. The flow meter

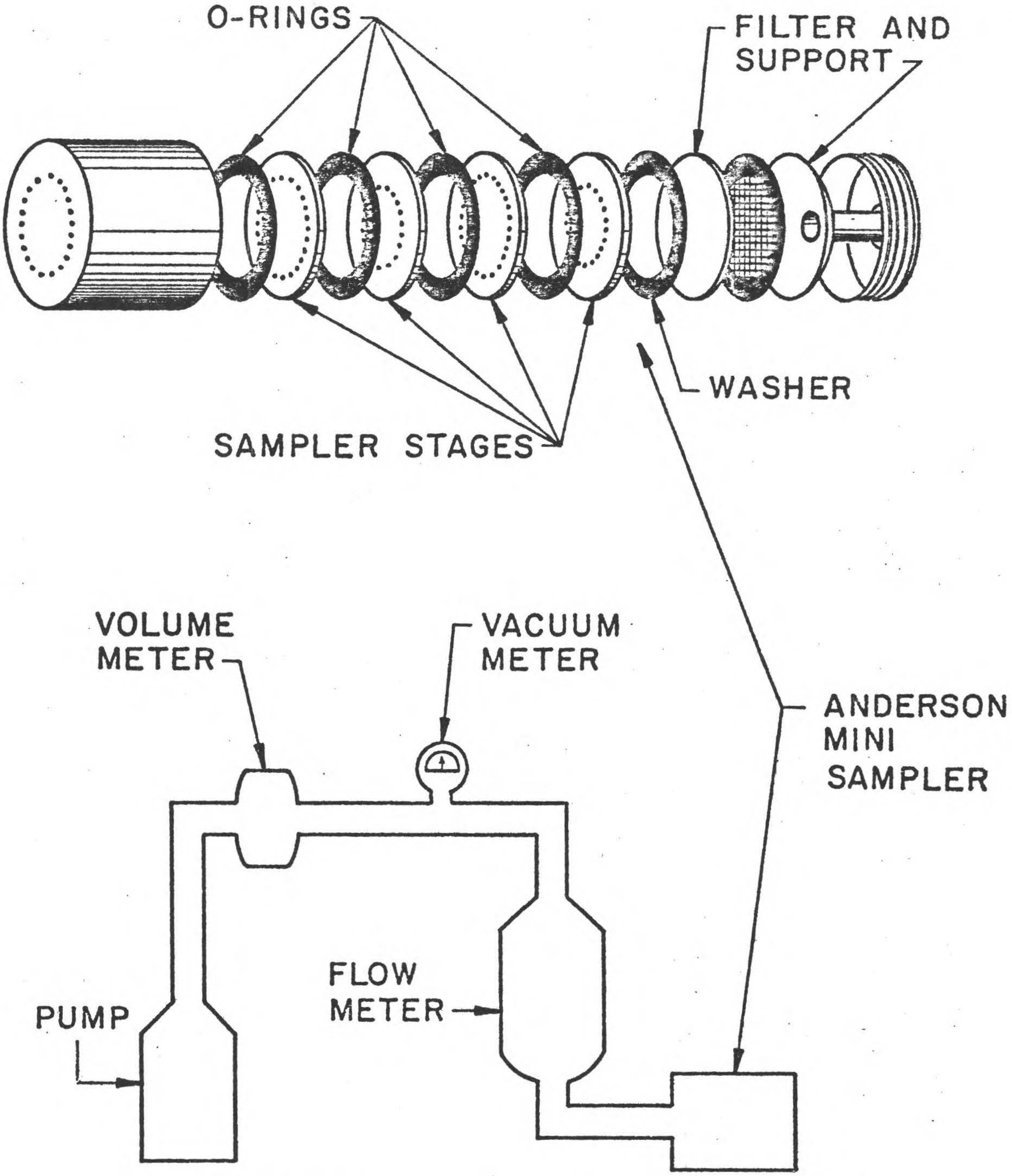

Figure 1.--Schematic Diagram of Sampler

is attached in series to a vacuum meter, a dry gas volume meter, a bleed valve, and the pump. The pump is a moded TD-4X2 miniature, brushless, double acting diaphragm blower. It operates on a 24 volt D.C. system drawing approximately 0.100 amp at full load. The service life of the motor is in excess of 10,000 hours continuous duty and the minimum service life of the pumping sections is of the order 2500 to 5000 hours continuous duty.

Power was supplied by two 12-volt lead storage batteries. They provide current for continuous operation of up to 30 days.

The system was run at a vacuum pressure of between 4.1 and 5.1 torr. A flow rate of 1.4 ℓ/min was maintained.

The sampler was housed in a double roofed shelter, constructed by the Brigham Young University Botany Department. It was 30 in wide, 20 in deep, and 24 in high. For future use it is recommended that the houses be 22 in wide, 20 in deep, and 20 in high. A diagram of the arrangement of the equipment in the shelter is shown in Figure 2.

Sample Analysis

The analysis for trace metals in the aerosol samples is done by X-ray fluorescence (XRF). The Nuclepore filters containing the particulates are placed in a beam of high energy protons. Protons bombard the atoms on the target causing the electrons from the atomic orbitals to be emitted. When an electron from a low lying energy level is emitted, an electron from a higher level replaces it giving off X-rays of energy equal to the change in energies of their atomic electronic states. The X-rays strike a Si(Li) detector giving rise to electrical pulses which can then be analyzed by electronic means. For a detailed

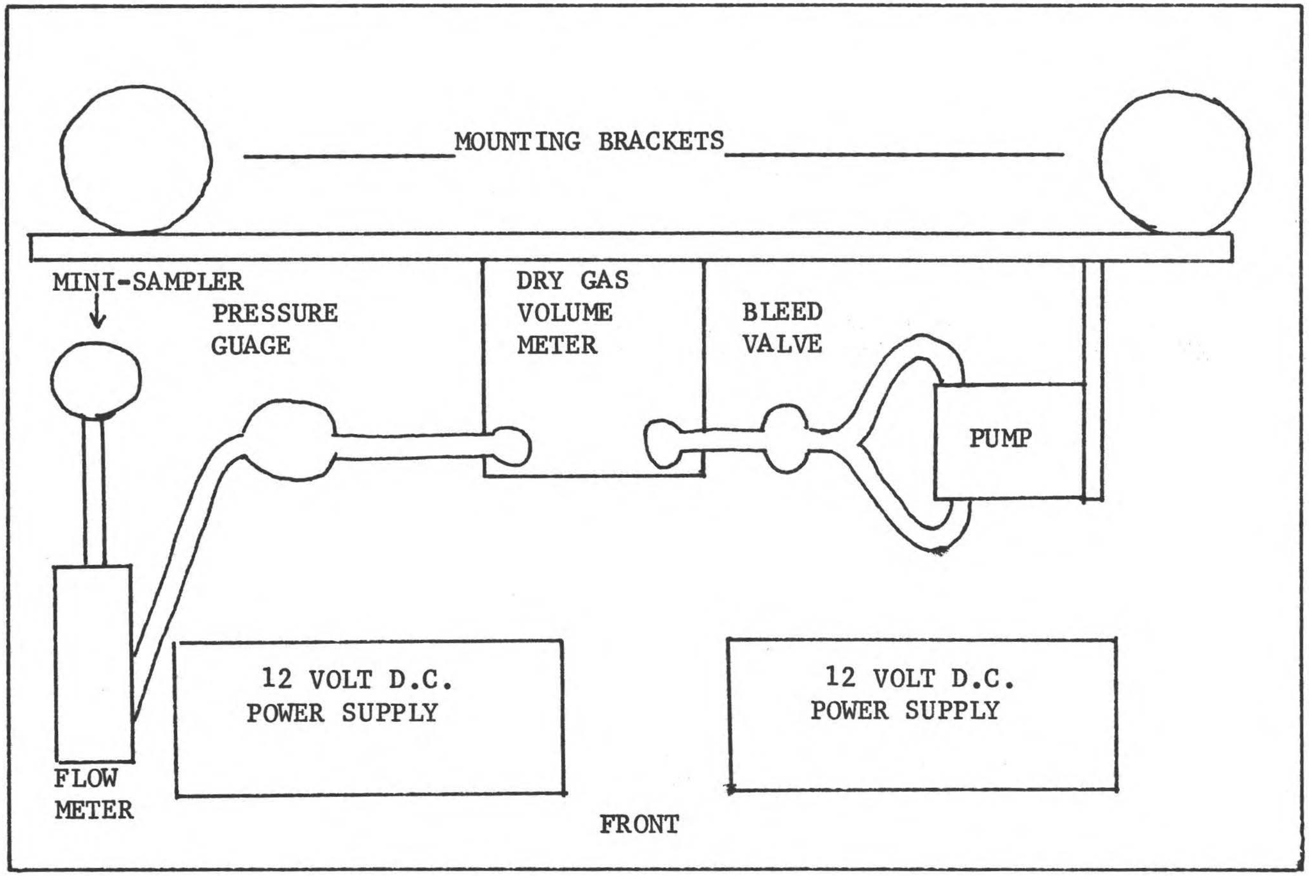

Figure 2.--Top view of the equipment arrangement inside the shelter.

explanation of the equipment involved the reader is referred to a master's thesis by George M. Allison.[38] Also included in his work are the calibration data and curves of the XRF. Calibration curves and analytical determinations of the XRF introduce errors of ± 15%. Counting statistical errors for the more abundant elements are most often insignificant. For those elements in which smaller amounts are reported the counting errors might be sufficient to cause a total error of ± 25% in the reported value.

Validation of the Sampler

Analyses of three samplings are reported. The results of the samples taken May 24 - June 5, 1973, and July 23 - August 2, 1973, are reported in the following tables: (1) Elemental Composition of Sample (in ppm) (See Tables 1 and 4). (2) Elemental Composition of Sample in Relation to Stage 1 (See Tables 2 and 5). (3) Elemental Composition of Sample in Relation to Iron (See Tables 3 and 6). Spectral plots for the samples taken July 23 - August 2, 1973, and for the filter only from a sample collected June 7-10 and 21-25, 1973, are shown in Figures 3 - 8. Similiar results from an earlier sampler have been reported.[39]

When the sample which was collected during May 24 - June 5, 1973, was taken, the sampler was in the early stages of development. Work was being done to see if the Anderson mini-sampler was capable of providing filter loading of 100 $\mu g/cm^2$ without pressure changes in the system. The dry-gas volume meter had not been installed, so a measure of the total volume of air could only be approximately determined. The sampler was turned off when the pressure had changed a total of 1.1 torr. The loading achieved on the filter was 133 $\mu g/cm^2$.

TABLE 1

ELEMENTAL COMPOSITION OF SAMPLE[a] (in ppm)[b]

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	7786	4227	2490	2564	132
Ti	267	150	90.1	127	3.2
V	11.0	8.6	3.9	7.9	.7
Cr	28.3	40.4	16.9	34.0	5.1
Mn	118	64.0	45.0	54.0	29.4
Fe	4016	2848	1943	2829	826
Ni	41.0	147	35.6	53.0	3.6
Cu	41.4	23.8	25.8	17.9	26.3
Zn	212	136	31.6	109	149
Pb	229	141	120	297	1207
Se				2.7	6.1
Br	144	28.4	72.6	32.5	43.7
Rb	38.5	14.4	27.5	obs	18.6
Sr	48.8	28.2	14.5	18.8	8.5
Zr	25.7	6.3	obs	17.8	8.4
Mo	obs	obs	16.8	obs	19.4

[a] Sample collected in Utah County, Utah, May 24 - June 5, 1973.

[b] Uncertainty not greater than ± 25%.

TABLE 2

ELEMENTAL COMPOSITION OF SAMPLE[a] RELATIVE TO STAGE 1

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	1	.54	.32	.33	.020
Ti	1	.56	.34	.48	.012
V	1	.78	.35	.72	.067
Cr	1	1.4	.59	1.2	.18
Mn	1	.54	.38	.46	.25
Fe	1	.71	.48	.71	.21
Ni	1	3.6	.87	1.3	.088
Cu	1	.58	.62	.43	.64
Zn	1	.64	.15	.51	.70
Pb	1	.62	.52	1.3	5.3
Br	1	.20	.50	.23	.30
Rb	1	.37	.71		.48
Sr	1	.58	.30	.39	.17
Zr	1	.25		.69	.33

[a] Sample collected in Utah County, Utah, May 24 - June 5, 1973.

TABLE 3

ELEMENTAL COMPOSITION OF SAMPLE[a] RELATIVE TO IRON

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	193	148	128	91	16
Ti	6.6	5.3	4.6	4.5	.39
V	.27	.30	.20	.28	.09
Cr	.70	1.4	.87	1.2	.62
Mn	2.9	2.2	2.3	1.9	3.6
Fe	100	100	100	100	100
Ni	1.0	5.2	1.8	1.9	.44
Cu	1.0	.84	1.3	.63	3.2
Zn	5.3	4.8	1.6	3.9	18
Pb	5.7	4.9	6.2	11	146
Se				.09	.74
Br	3.6	1.0	3.7	1.1	5.3
Rb	.96	.51	1.4		2.3
Sr	1.2	1.0	.75	.66	1.0
Zr	.64	.22		.63	1.0
Mo			.86		2.3

[a] Sample collected in Utah County, Utah, May 24 - June 5, 1973.

TABLE 4

ELEMENTAL COMPOSITION OF SAMPLE[a] (in ppm)[b]

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	837	370	137	164	44
Ti	28	13	5.6	13	1.9
V		1.0	1.3	0.2	0.1
Cr	17	5.9	5.4	42	0.7
Mn	11	5.2	3.2	7.7	4.7
Fe	482	246	114	417	134
Ni	16	13	14	76	0.9
Cu	12	9.1	5.8	14	4.4
Zn	53	9.1	13	35	32
Pb	22	12	12	58	312
Br	6.1	3.9	4.6	9.7	19
Rb	2.1	1.5	1.5	3.5	
Sr	6.7	2.5	1.4		1.5

[a] Sample taken in Utah County, Utah, July 23 - August 2, 1973.

[b] Uncertainty not greater than ± 25%.

TABLE 5

ELEMENTAL COMPOSITION OF SAMPLE[a] RELATIVE TO STAGE 1

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	1	.44	.16	.19	.04
Ti	1	.45	.20	.46	.07
Cr	1	.34	.31	2.4	.04
Mn	1	.46	.29	.69	.44
Fe	1	.51	.24	.86	.28
Ni 1	1	.85	.88	4.9	.06
Cu	1	.77	.50	1.2	.37
Zn	1	.17	.25	.66	.60
Pb	1	.55	.55	2.6	14
Br	1	.64	.76	1.6	3.1
Rb	1	.70	.70	1.7	
Sr	1	.37	.21		.22

[a] Sample taken in Utah County, Utah, July 23 - August 2, 1973.

TABLE 6

ELEMENTAL COMPOSITION OF SAMPLE[a] RELATIVE TO IRON

Element	Stage 1	Stage 2	Stage 3	Stage 4	Filter
Ca	170	150	120	39	33
Ti	5.8	5.3	4.9	3.1	1.4
V		0.4	1.2	0.05	0.07
Cr	3.5	2.4	4.8	10.1	0.54
Mn	2.2	2.1	2.8	1.9	3.6
Fe	100	100	100	100	100
Ni	3.3	5.3	12.3	18.5	0.6
Cu	2.4	3.7	5.1	3.4	3.3
Zn	8.0	3.7	12	8.4	24
Pb	4.6	4.9	11	14	233
Br	1.3	1.6	4.1	2.3	14
Rb	0.4	0.6	1.3	0.8	
Sr	1.4	1.0	1.2		1.1

[a] Sample taken in Utah County, Utah, July 23 - August 2, 1973.

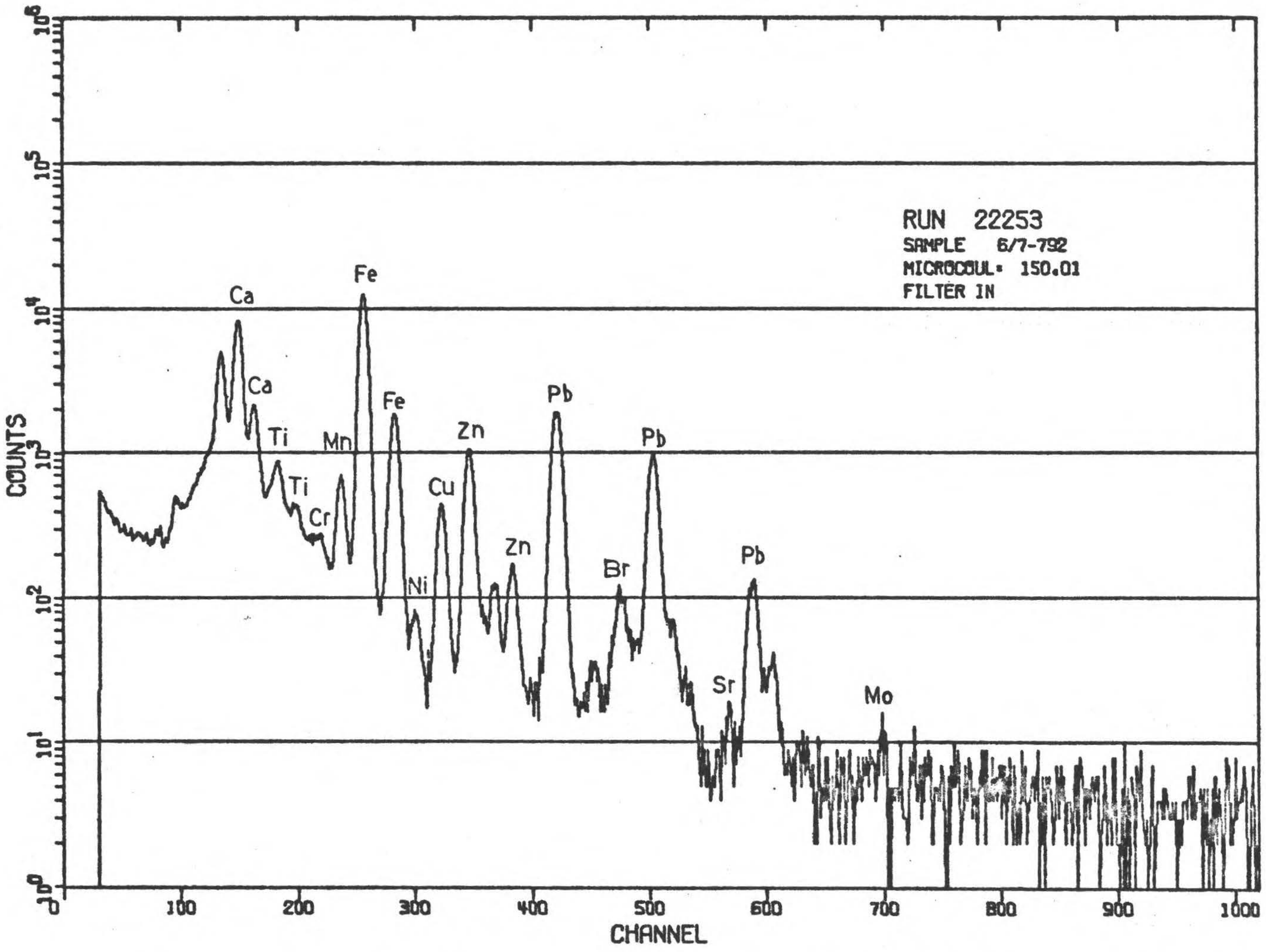

Figure 3.--Plot of filter from sample collected June 7-10; 21-25, 1973, at Provo, Utah.

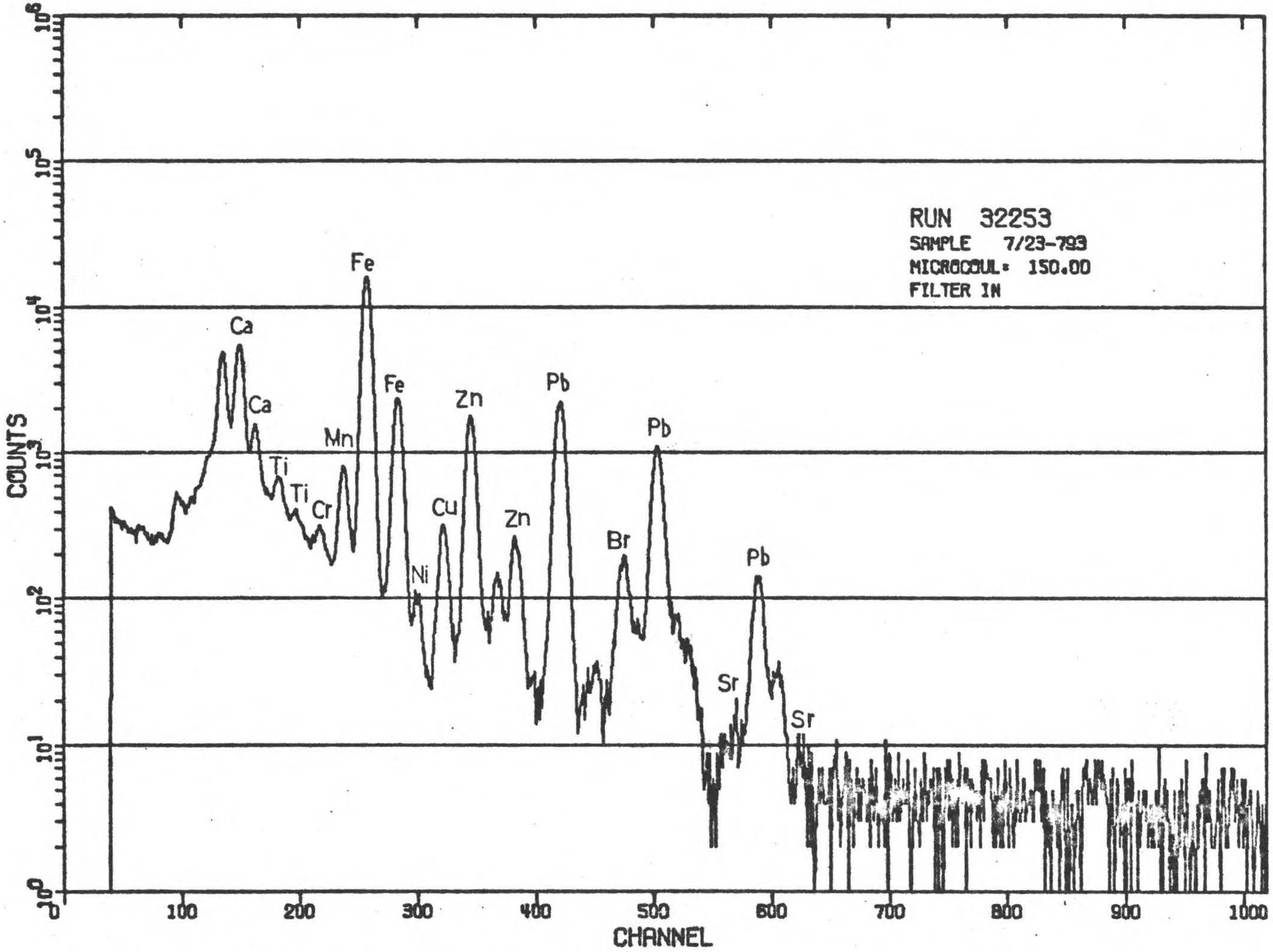

Figure 4.--Plot of filter from sample collected July 23 thru August 2, 1973, at Provo, Utah.

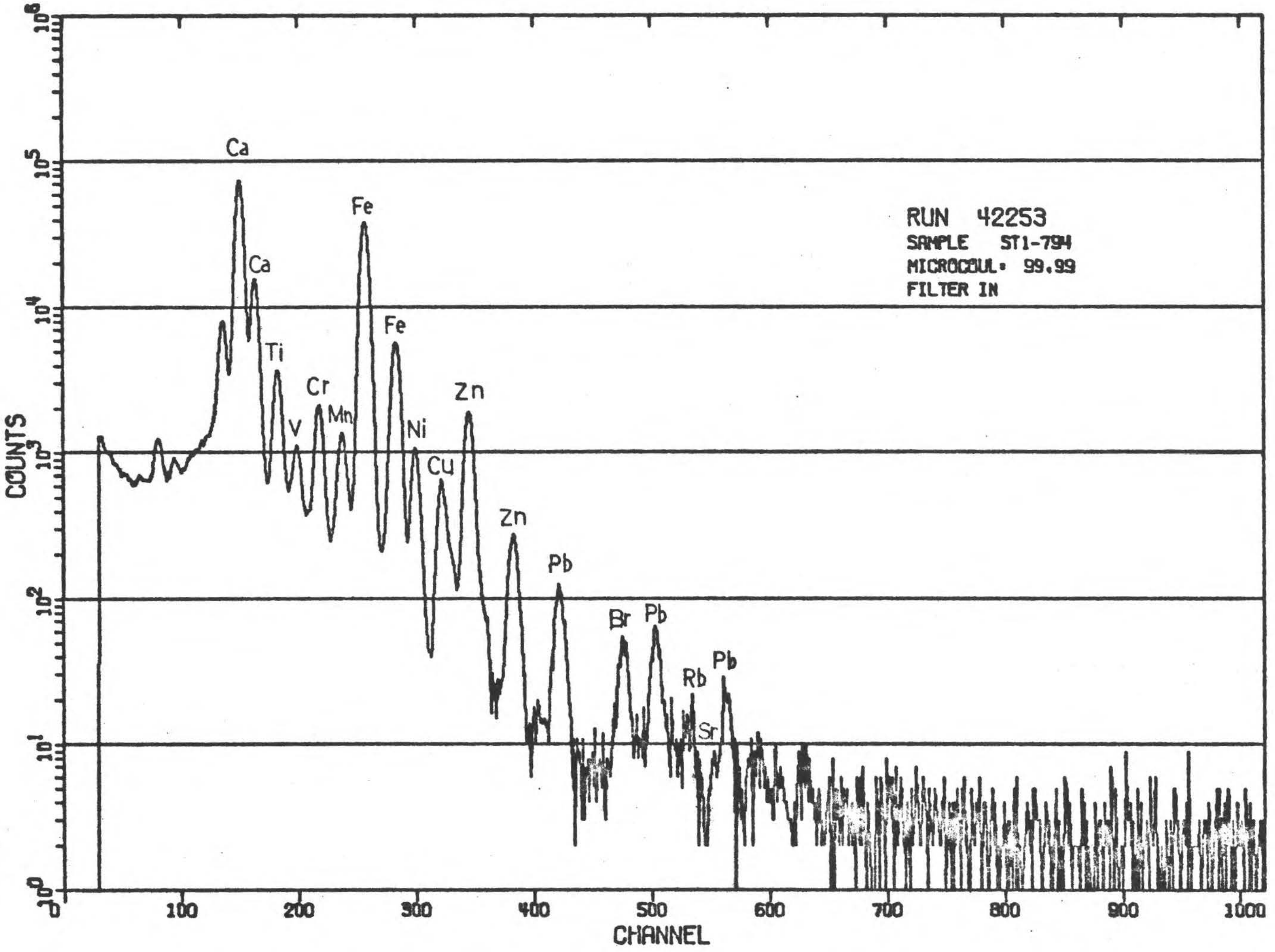

Figure 5.--Plot of Stage 1 from sample taken July 23 thru August 2, 1973, at Provo, Utah.

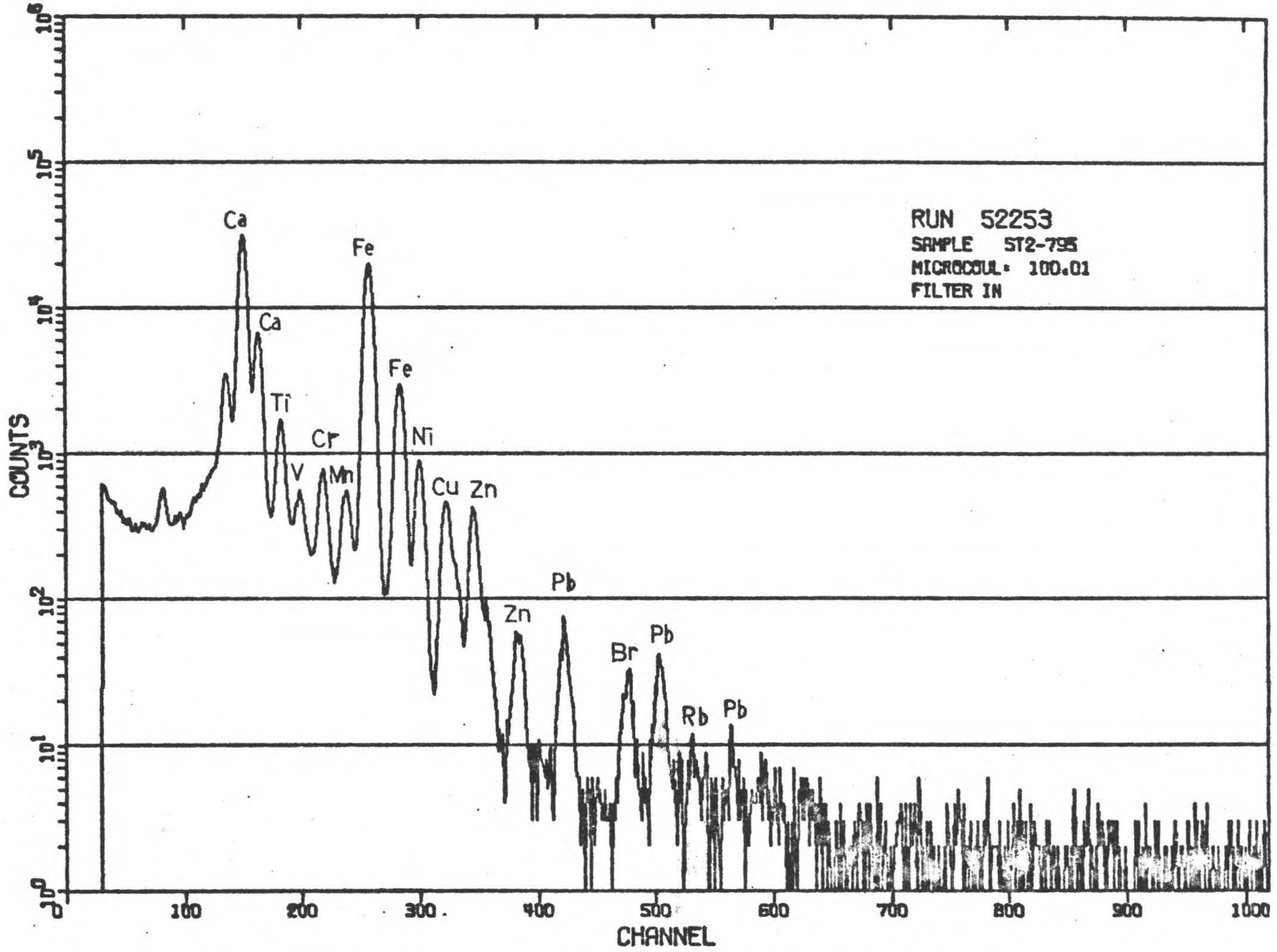

Figure 6 --Plot of Stage 2 from sample collected July 23 thru August 2, 1973, at Provo, Utah.

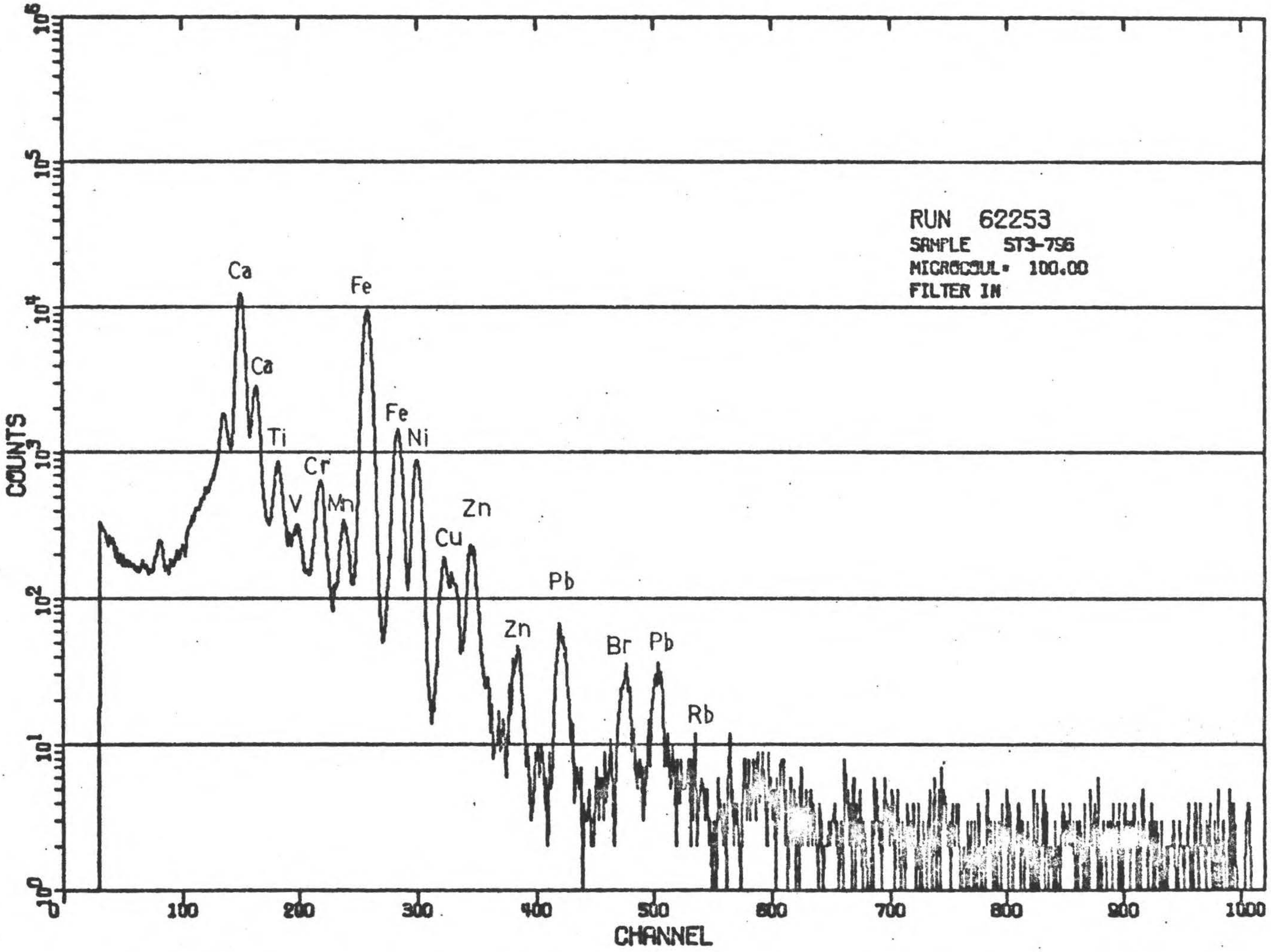

Figure 7.--Plot of Stage 3 from sample collected July 23 thru August 2, 1973, at Provo, Utah.

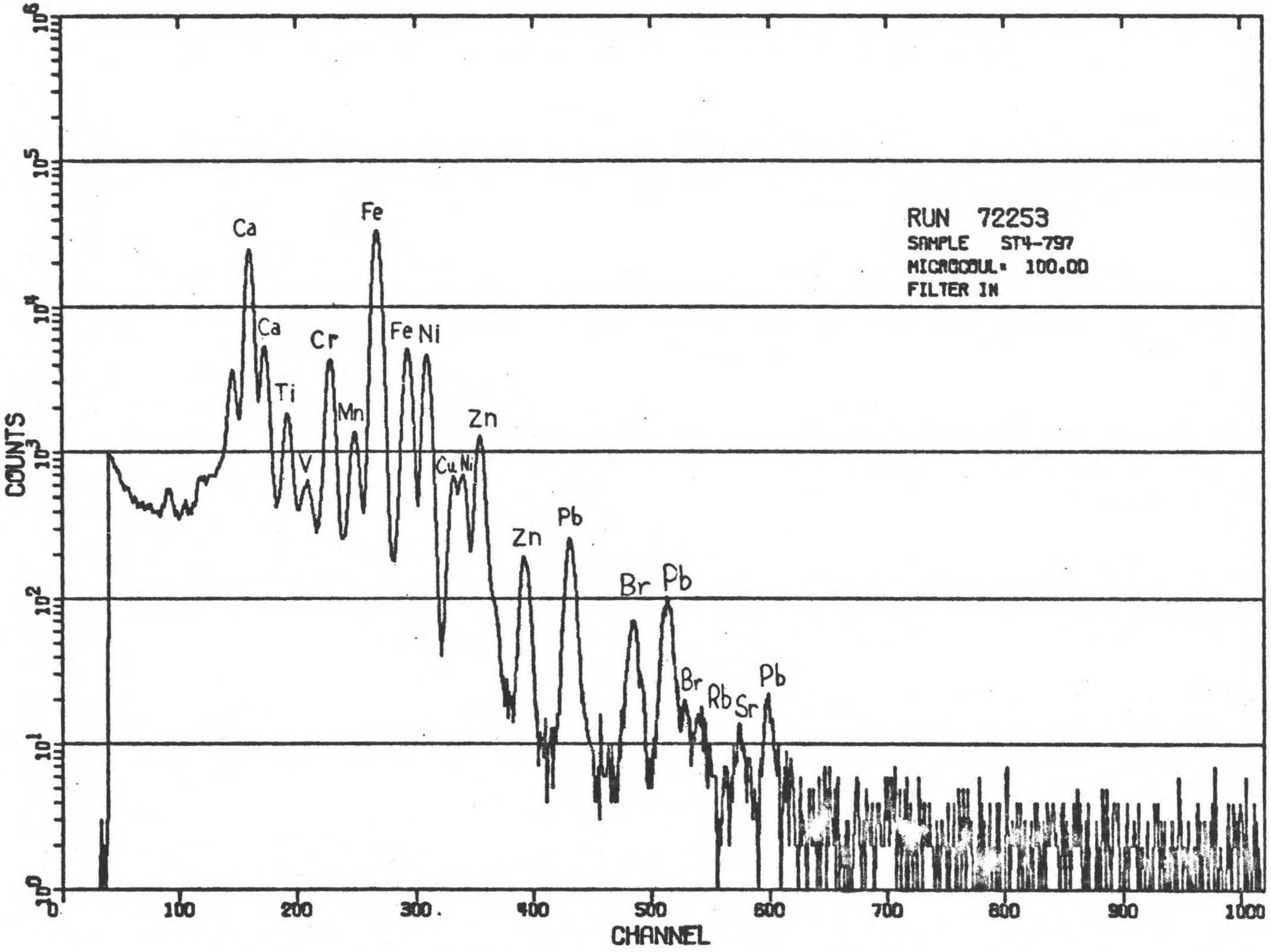

Figure 8.--Plot of Stage 4 from sample collected July 23 thru August 2, 1973, at Provo, Utah.

This indicated the desired loading could be achieved without a substantial pressure change in the system. The sample collected from July 23 - August 2, 1973, was made with the dry-gas volume meter, so the total particulate concentration could be determined.

When the equipment was first installed on the Richards Physical Education Building, sampling was being done by the Brigham Young University Health Department using a high volume sampler to determine total particulate concentration ($\mu g/m^3$) of the atmosphere. During the period it was in operation they reported daily values ranging from 45 to 60 $\mu g/m^3$. Sampling stopped about July 15, 1973, for the summer so a comparison of the Anderson mini-sampler with the high volume sampler was not made. Earlier samples collected with the mini-sampler in May and June yielded results that could easily range from 50 to 65 $\mu g/m^3$ when the total volume was approximated with time and flow meter readings. The July 23 - August 2, 1973, sample collected with the mini-sampler yielded a total particulate concentration of 45 $\mu g/m^3$. Determination of the amount of each element in the particulate in $\mu g/m^3$ can be calculated by multiplying the ppm of each element by a conversion factor. The conversions factors are: (1) Stage 1; 6.47×10^{-9} m^{-3}. (2) Stage 2; 8.03×10^{-9} m^{-3}. (3) Stage 3; 6.74×10^{-9} m^{-3}. (4) Stage 4; 1.14×10^{-8} m^{-3}. (5) Filter; 1.20×10^{-8} m^{-3}. The ambiant air volume (V_a) was calculated using Boyle's Law, the volume in the meter (V_m), the ambiant pressure (P_a), and the pressure drop across the filter, the latter two values are used in the calculation of the meter pressure (P_m).

$$(1) \quad P_a V_a = P_m V_m$$

During the collection of the June 7-10, and 21-24 sample, a diaphragm in the pump broke. As a result only the backup filter was analyzed to check a comparison with the other filter. Examination of the spectral plots of this filter (Figure 3) and the backup filter of the July 23 - August 2 sample, (Figure 4) reveals similar patterns for both samples.

Even though the sample collected May 24 - June 5, gave much more particulate matter than the July 23 - August 2 sample, a check of the ratios of elements for each of the stages and filters between these samples is fairly constant. Note specifically Ca, Fe, and Pb. The comparisons of each of the elements in relation to iron from each of the samples (See Tables 3 and 6) indicates good agreement for several of the elements reported.

Because of the loading capabilities of more than 100 $\mu g/cm^2$ and the similarities observed in the samples that have been analyzed the sampler, as it has been constructed, seems to be feasible. Further testing that can be done is described in the next section.

IV. SUMMARY

This thesis reports a study of the construction and testing of a remote aerosol sampler to be used with XRF analysis. It has the advantage of allowing sampling over a long period of time, containing its own power supply, and the cost of materials is not unreasonably high.

The accuracy of trace element analysis depends on two factors; the design of the sampler and the validity of the XRF. The validity of the XRF is beyond the scope of this work and is not reported here.

Lundgren[9] reports work on the validity of impaction type samplers. Gordon and Gladney[40] have also evaluated collection efficiencies of impaction type samplers. They suggest the use of sticky surfaces to improve collection efficiencies. Other work to be done in the future could include: (1) Placing the sampler to a height of 50 feet above the surface in order to escape any effects there may be due to blowing surface dusts in the vicinity of the sampler. (2) Determining how much bounce-off there is when particulates strike the impaction plates in the mini-sampler. (3) Locating a substance to cover the impaction plates as a means of collecting particulates to prevent bounce-off. (4) Collect samples in the same place and time using the mini-sampler and other sampler designs and compare the values obtained. (5) Using a given amount of particulant with known particle sizes, take samples using the mini-sampler and compare the yield with the quantity that was present at the beginning of the sample.

The sampler as now constructed does provide a means of collecting aerosol samples and the work done in the future will hopefully improve the working and construction of this sampler.

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A REMOTE AEROSOL SAMPLER TO BE USED WITH CHARGED PARTICLE X-RAY FLUORESCENCE ANALYSIS

Steven Dee Rowley

Department of Chemistry

M. A. Degree, August 1974

ABSTRACT

A remote aerosol sampler, capable of up to 3 weeks continuous sampling has been developed. It consists of an Anderson 2000 Inc. mini-sampler or personnel monitor requiring an air flow of 1.4 ℓ/min, a flow meter, a vacuum meter, a bleed valve, a dry gas volume meter, and a pump. The pump is a Brailsford Model TD-4X2, and operates on a 24 volt D.C. power supply. The sampler consists of four impaction stages with a 0.4 μ Nuclepore backup filter. Particulates are removed from the plates in an ultrasonic cleaner with hexane solvent. The suspended particulates are collected on a 0.4 μ Nuclepore filter.

Elemental analysis of the aerosol is accomplished by the method of charged particle X-ray fluorescence. The results of two samples taken in Utah County, Utah, are tabulated. Elemental composition is reported as ppm of particulate, as a ratio relation to stage 1, and as a ratio relation to iron. High values are reported for iron and lead, with most of the lead being collected in the filter.