COMPARATIVE PARTICULATE SAMPLING METHODS
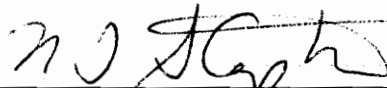
by

Frank Butler Twitty

Thesis submitted to the Graduate Faculty of the

Virginia Polytechnic Institute and State University

in partial fulfillment of the requirements for the degree of
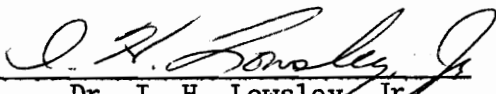
MASTER OF SCIENCE

in

Environmental Sciences and Engineering

APPROVED:

_____
Dr. N. T. Stephens, Chairman

_____   _____
Dr. I. H. Lowsley, Jr.   Dr. L. L. Harms

July, 1974

Blacksburg, Virginia

ACKNOWLEDGMENTS

The author wishes to make acknowledgments:

To Dr. N. T. Stephens, thesis and major advisor, for his
guidance and constructive criticism throughout the
graduate program.

To Dr. R. C. Hoehn and Dr. I. H. Lowsley, Jr. for their aid
and interest throughout the year.

To Mr. E. G. Willard for his assistance in the laboratory.

TABLE OF CONTENTS

TABLE OF CONTENTS - continued

LIST OF FIGURES

LIST OF TABLES

vi

I. INTRODUCTION

Measurement of the concentration of air pollutants is necessary before consideration can be given toward control of these pollutants. State and Federal standards and regulations on particulate pollutants already exist for ambient air quality and most of these standards and regulations are based on particulate mass. Measurement of the mass concentration of particulate matter is difficult in that most methods require long sampling times and manual weighing procedures which are time consuming, costly from a labor point of view, and subject to manually induced errors. A current Environmental Protection Agency (EPA) recommended filter weighing procedure (1) in common use is the high-volume sampler which collects particles on a filter over a twenty-four hour period with a flow rate of 30 to 60 cubic feet per minute (cfm). The filter must be removed each day and replaced with a carefully weighed clean filter. The particulate loaded filter must be stabilized for a few hours in a controlled environment and manually weighed to determine the loading in micrograms per cubic meter ($\mu g/m^3$). The results from the high-volume sampler method provide a single value averaged over a twenty-four hour period. Quite often it is desirable to know the particulate loading for a particular time interval within the twenty-four hour sampling period.

The determination that quartz crystals oscillating in the thickness shear mode could be used to determine the mass of deposited films was made by Pulker and Schadler (2) in 1967. The piezoelectric-electrostatic aerosol mass concentration monitor commonly known as the

1

particle mass monitor is a commercially available automatic, fast response instrument which measures the true mass of atmospheric particles by deposition on such a crystal. The fast response time allows measurement of particulate loadings over a range from 1.0 to 200,000 $\mu g/m^3$ with a typical time resolution of 0.6 minutes for a concentration of 100.0 $\mu g/m^3$. The particle mass monitor allows collection of data over small time intervals within the sampling period of the high volume sampler.

The purpose of this research is to determine the relationship between the results of the high volume sampler and particle mass monitor procedures for the measurement of particulate loading in ambient aerosol sampling. In order to accomplish this objective the sampling time of the particle mass monitor will be lengthened to twenty-four hours so that a direct correlation of the results from both procedures can be made.

The ultimate determination will be whether or not the particle mass monitor can be used in place of, or should be used in addition to, the high volume sampler method for ambient aerosol sampling.

II. LITERATURE REVIEW

Particulate Sampling

In accordance with the objective of this study, which is to
compare particulate sampling techniques, it is necessary to arrive
at a consistent definition of particulate matter. Perkins (4) de-
fines particulate matter as any dispersed matter, solid or liquid,
in which the individual aggregates are larger than single small
molecules (about 0.0002 microns (μ) in diameter) but smaller than
about 500 μ. Atmospheric dust particles range over a wide spectrum
of sizes from greater than about 0.002 μ to about 10.0 μ. These fig-
ures indicate that a variety of sampling techniques are necessary to
deal with the enormous size range of particulates in the atmosphere.
Ideally, it is desirable to collect all of the particles over as wide
a size range as possible, and determine the mass of the collected
particles per unit volume of the gas stream carrying the particles.
This study deals entirely with solid undifferentiated particulate
matter. There are many difficulties associated with ambient parti-
culate sampling, most of which are associated with the vast range of
particle size and composition. The mechanisms of deposition of
particles within a sampling line, or within the sampling apparatus,
include electrostatic and magnetic precipitations, diffusion, gravity
settling, turbulent deposition, and inertial impaction. These
mechanisms depend on a number of variables: flow rate, length and
size of transport tube, curvature of the tubing, and characteristics

3

of the particles.(5) Care must be exercised to minimize deposition

by the use of small diameter, short sampling lines, and as high a

sampling flow rate as practical. The inertial forces on the particles

mean that they will not always follow the flow lines of the gas

stream, so that isokinetic sampling techniques must be employed to

insure that the segment of the sample analyzed is representative of

an unbiased portion of the original sample. Due to the wide range

of sizes of ambient particulate matter, there is no single technique

that is satisfactory for all sampling needs, and it is therefore

often required that a combination of techniques be used to accomplish

a particular assignment.

The following discussion describes some of the more common

methods of particulate sampling, grouped according to the physical

principle utilized in the collection of the particles.

Gravitational Settling

Field collection of large particles settling by gravitational

forces from the atmosphere are collected in open-top collectors of a

specified size and shape. These containers are placed outdoors in

carefully selected locations and filled to half of the collector

height with distilled water to trap and hold the particles as they

settle into the container. The sampling period is one calendar month

corrected to 30 days. This procedure yields the mass of the collected

particles in grams per meter squared per month (g/m^2-month), but only

after it is manually weighed. The obvious limitations of this pro-

cedure are the length of time required to collect a sample, and the

fact that only particles approximately 10.0 μ and larger in diameter can be collected.(6)

Impaction

If dust laden air is accelerated to a high velocity and allowed to impinge on a surface, it loses kinetic energy and deposits the particles. The size of the particles deposited is dependent on the velocity of the gas stream, which makes it necessary to use a series of impaction devices so that a variety of gas stream velocities can be obtained. This apparatus has the capability of collecting particles in the size range from 0.01 μ to 10.0 μ, but will not directly give the mass of the collected particles without manual weighing. The primary use of this procedure is in the determination of particle size distribution.(4)

Electrostatic Precipitation

Particles in a gas stream moving past an ionizing wire charged at corona voltage acquire a net electrical charge of the same polarity as the wire. The charged particles are attracted to a plate of opposite polarity and collected. This method is highly efficient over a wide range of sizes from 0.001 μ to 100.0 μ.(4) The collected particles must then be manually weighed in order to determine the mass concentration.

Thermal Precipitation

In this technique particles in a gas stream are forced to move past a hot wire and are repulsed to a nearby cold plate where they

are collected. This procedure is efficient for particles in the 0.01 μ
to 5.0 μ range(4) but operates at the extremely low sampling rate of
approximately 50 milliliters per minute. To obtain the mass con-
centration, the collected sample must be manually weighed.

Filtration

Particles in a gas stream may be collected on a porous filtering
medium and then manually weighed to determine the mass. This is
perhaps the most widely used method of determining the mass of
particulate matter and is specified for ambient particulate sampling
by the Environmental Protection Agency.(1) In this procedure 30.0 to
60.0 cfm of particulate laden air is drawn through a glass fiber
filter for a period of 24 hours. The filter is dried and weighed
before and after the filtering operation, and the mass of the parti-
culate matter is determined. This procedure yields the mass of the
particulate matter in $\mu g/m^3$ and will sample with over 99% efficiency
for particles as small as 0.3 μ in diameter.

Acoustical Impaction

With this method particles are accelerated by sonic energy and
then impinged on a target where they are collected and manually
weighed to determine the mass. With this procedure one may determine
the size of a particle as a function of particle mass for particles
5.0 μ and larger.(7) A serious limitation of this procedure is the
lower limit of the size of particles that can be collected.

Beta Radiation

In this technique the particulate matter is first collected on a strip of filter paper which is passed between a beta emission source and a detector. Some of the electrons pass through the particulate matter and some are absorbed. The reduction in beam intensity can be directly correlated to the mass of the particulate matter trapped on the filter medium strip. This technique is efficient for particles 0.1 μ and greater.(7)

Piezoelectric

Particulate matter is deposited by impaction and electrostatic precipitation onto a crystal with piezoelectric properties (see piezoelectric theory, page 8). The frequency of vibration of the crystal decreases as the particulate loading on the crystal increases. This procedure will measure particles in the size range from 0.01 to 20.0 μ.(8,9,12,13)

Discussion

In all of the previous examples of particulate sampling except Beta Radiation and Piezoelectric, the particles must be manually weighed or the mass is given in parameters which must be converted to mass per unit volume of air. Olin and Sem(8) list two instruments that have been developed for directly measuring the actual mass concentration of particulate matter. The first utilizes beta ray absorption but lacks sensitivity for ambient sampling. The second is the piezoelectric microbalance which uses a combination of electrostatic precipitation and piezoelectric crystal sensing.

The piezoelectric microbalance was selected as a comparison to the established and accepted method of high volume sampling, because it is a recently developed technique and shows great potential for the ambient sampling of particulate matter.

Piezoelectric Theory

Piezoelectricity is the property of certain crystals, including quartz, which results in an electrical charge on certain surfaces of the crystal when it becomes mechanically stressed. Conversely a piezoelectric crystal becomes mechanically stressed if an electrical charge is placed on certain faces. These crystals can be placed in an appropriate electronic oscillating circuit and will cause the circuit to oscillate at the natural vibrational frequency of the crystal. These crystals vibrate at a very precise natural frequency so that frequency changes of one part in 10 million are easily detectable. Sauerbrey(8) has determined that the natural frequency of vibration of a quartz crystal decreases linearly when foreign material adheres to its surface. The magnitude of the frequency change is directly proportional to the mass of the foreign material.

Type AT quartz crystals are used in piezoelectric microbalances. The designation AT refers to the orientation of the cut of the crystal plate with respect to the axis of the uncut crystal. These crystals vibrate in the shear mode and have no nodal points on the face of the crystal. Therefore, the frequency of vibration is independent of the elastic properties of the layers of deposited particles on the crystal surface.(8,9)

Reist and Burgess(10) used quartz crystals to detect the size of micrometeroids colliding with space vehicles, and this led to the investigation of quartz crystals for the detection of aerosol particles. Pulker and Shadler(2) determined that the effective mass sensitivity of a quartz crystal decreases slightly as the mass deposition increases, but the effect is less than 1 percent if the change in frequency is less than 1/2 percent of the natural frequency. From this information it was determined that as the crystal becomes progressively more loaded the accuracy of the mass determination is diminished, or for large mass depositions on the crystal surface the mass response is slightly non-linear.

It is important that the particles deposited on the vibrating crystal surface adhere to the surface with enough force to counteract the inertia forces that tend to dislodge them. Therefore the ability of the particles to adhere to the crystal surface is a limiting factor that controls the degree of crystal loading.

The maximum size particle that can be weighed by a piezoelectric crystal is determined by the balance of attractive and inertial forces on the particle.(8) Corn(11) determined that in order to maximize the size of the particle that can be weighed it is necessary to use oscillating circuits having low drive levels. The lower limit of the drive level is determined by the noise level in the circuits. Some frequency between 1 and 5 mega hertz (MHz) seems to be acceptable. With these facts in mind it was possible to design a piezoelectric microbalance to weigh particles up to 20 μ.(8)

Olin and Sem(8) have determined that the theoretical sensitivity of a typical piezoelectric crystal with a natural frequency of 5 MHz is 160 hertz per micro gram (Hz/μg). A frequency shift of 1 Hz can be measured with a simple frequency counter so that a mass of approximately 0.0005 μg can be detected. Figure 1 shows the comparison of experimental with theoretical mass sensitivity of a 5.0 MHz crystal weighing particles collected from automobile exhaust. Table 1 shows the experimental mass sensitivity compared with the theoretical mass sensitivity of crystals used to measure the mass concentration of several aerosols.

Piezoelectric Microbalance

The piezoelectric microbalance is an instrument that uses a piezoelectric crystal as the plane in a point to plane electrostatic precipitator. The signal from the weighing crystal is mixed with the signal from a second crystal or reference crystal which is exposed to the same ambient conditions but does not have particles deposited on it. This reference crystal serves to cancel out any changes brought about by temperature or pressure effects. The mass concentration is determined by the use of the following formula:

$$C = 333 \frac{\Delta f}{\Delta t} \tag{1}$$

where C is mass concentration in g/m^3, Δf is the frequency shift in Hz, and Δt is the sampling time in seconds. The number 333 is a constant and is the same for all instruments. It is dependent on the sampling rate, which in this case is 1 liter per minute (LPM). (8,9, 12,13)
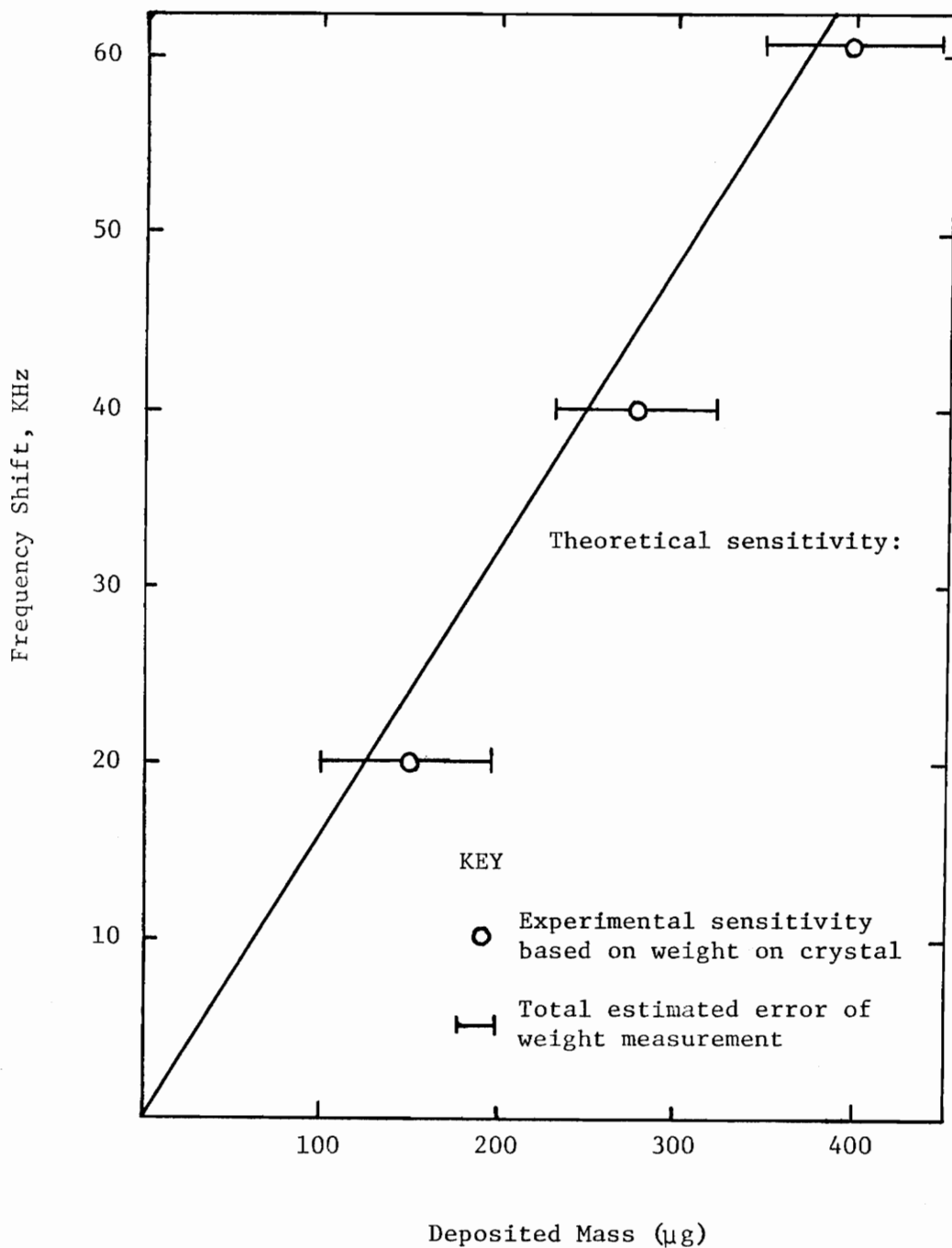
Figure 1. Comparison of experimental with theoretical mass sensitivity of 5.0 MHz crystal sensors for automobile emission test.(9)

TABLE 1

Experimental Mass Sensitivity Compared with Theoretical Mass
Sensitivity of Crystals Used to Measure the Mass Concentration
of Several Aerosols(9).

Crystal Frequency MHz	Aerosol	Average Mass Conc., μg/m³	Mass Sensitivity, Hz/μg	
			Theoretical	Experimental
1.5	Tobacco smoke	14,000	15.5±1	14.7±2
1.5	Tobacco smoke	5,830	15.5±1	14.4±2
1.5	Tobacco smoke	7,230	15.5±1	14.2±2
5.0	Tobacco smoke	--	179±10	179±30
1.5	Auto exhaust	1,460	15.5±1	17.6±3
5.0	Auto exhaust	1,865	160±10	150±17
5.0	Office aerosol	133	177±10	187±30
5.0	Office aerosol	162	177±10	198±30
5.0	Office aerosol	165	177±10	193±30
5.0	Acrylic spray*	--	165±10	158±20
3.0	Acrylic spray*	--	66±4	62±11
1.5	Acrylic spray*	--	15.0±1	16±3
1.5	Ambient Laboratory Pollution**	75-100	15.5±1	10.5±6

*Acrylic was sprayed directly onto the crystal electrode from an aerosol spray can. All other
aerosols were sampled by the standard aerosol mass concentration monitor.

**Relative humidity = 21%. Low experimental sensitivity, although well within error limits, may
indicate poor sticking of particles due to low relative humidity.

The electrostatic precipitator deposits the particles uniformly over the crystal electrode whereas a simple impaction device tends to concentrate the particulate matter in the center of the crystal.(12) The electrostatic precipitator is also compatible with the designed flow rate of 1 LPM which allows a sampling line 2 to 3 feet in length without appreciable particle loss.(12,13) Figure 2 gives the results of the piezoelectric microbalance's electrostatic precipitator efficiency tests carried out by varying the aerosol flow rate and the corona current with an aerosol of tobacco smoke. The test showed that the electrostatic precipitator is 100 percent efficient in collecting tobacco smoke as long as the corona current is maintained at a value exceeding 5 μ amperes and the flow rate is 1 LPM or less. Tests with several other aerosols showed the same results.(9) Some disadvantages of the electrostatic precipitator are the high voltage of 5.0 KV required for operation, and the fact that it may generate particles by polymerizing gaseous contaminants such as hydrocarbons. The majority of these particles are smaller than 0.001 μ and will readily pass through the precipitator without being collected. If the hydro-carbon content of the air is high, internal generation can reach levels of 5.0 $\mu g/m^3$.(9,12) In most applications the background level of internal particle generation due to gaseous pollutants is less than 1.0 $\mu g/m^3$.(12)

The piezoelectric microbalance employees a 5 MHz type AT quartz crystal because it has a high sensitivity and is thick enough to be handled. The size range for particulate collection for a piezoelectric
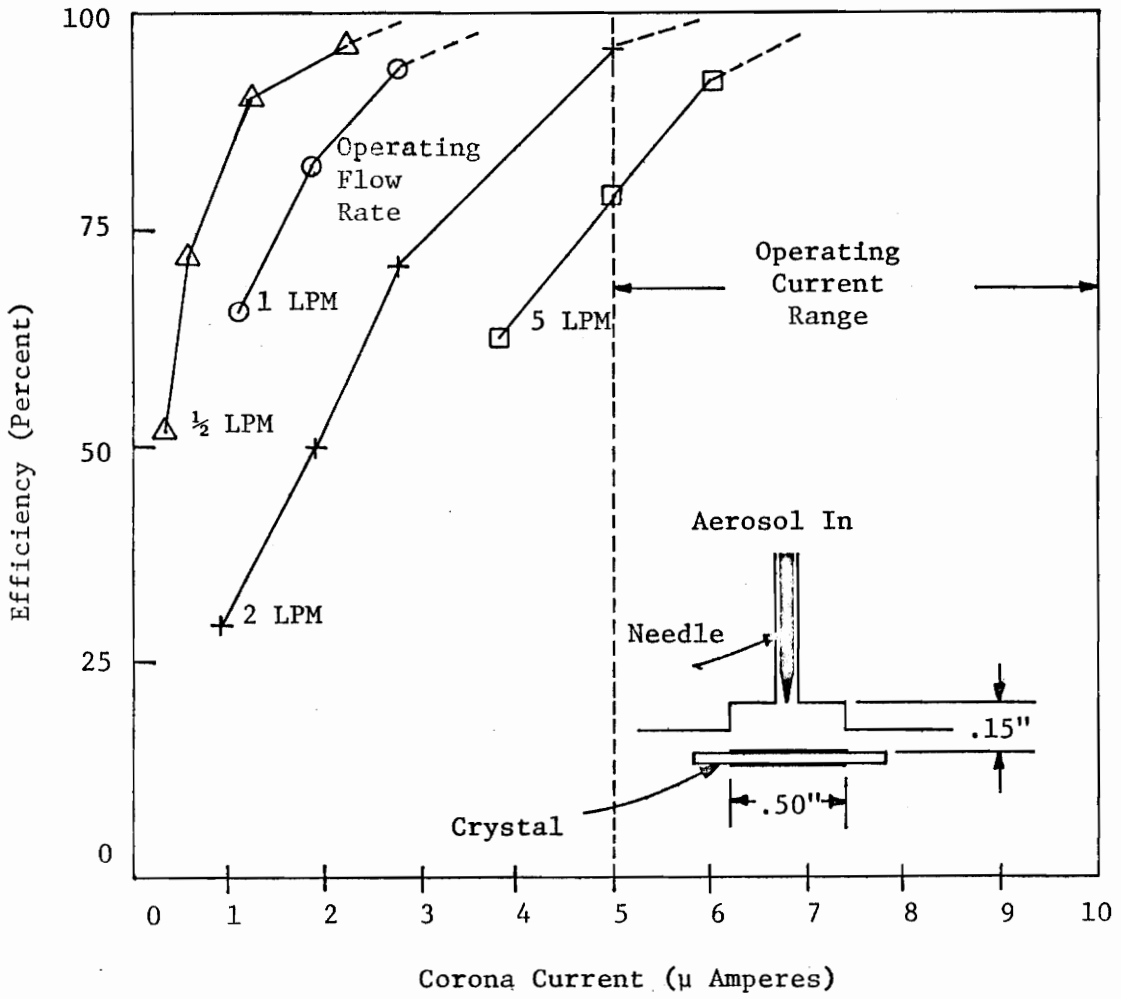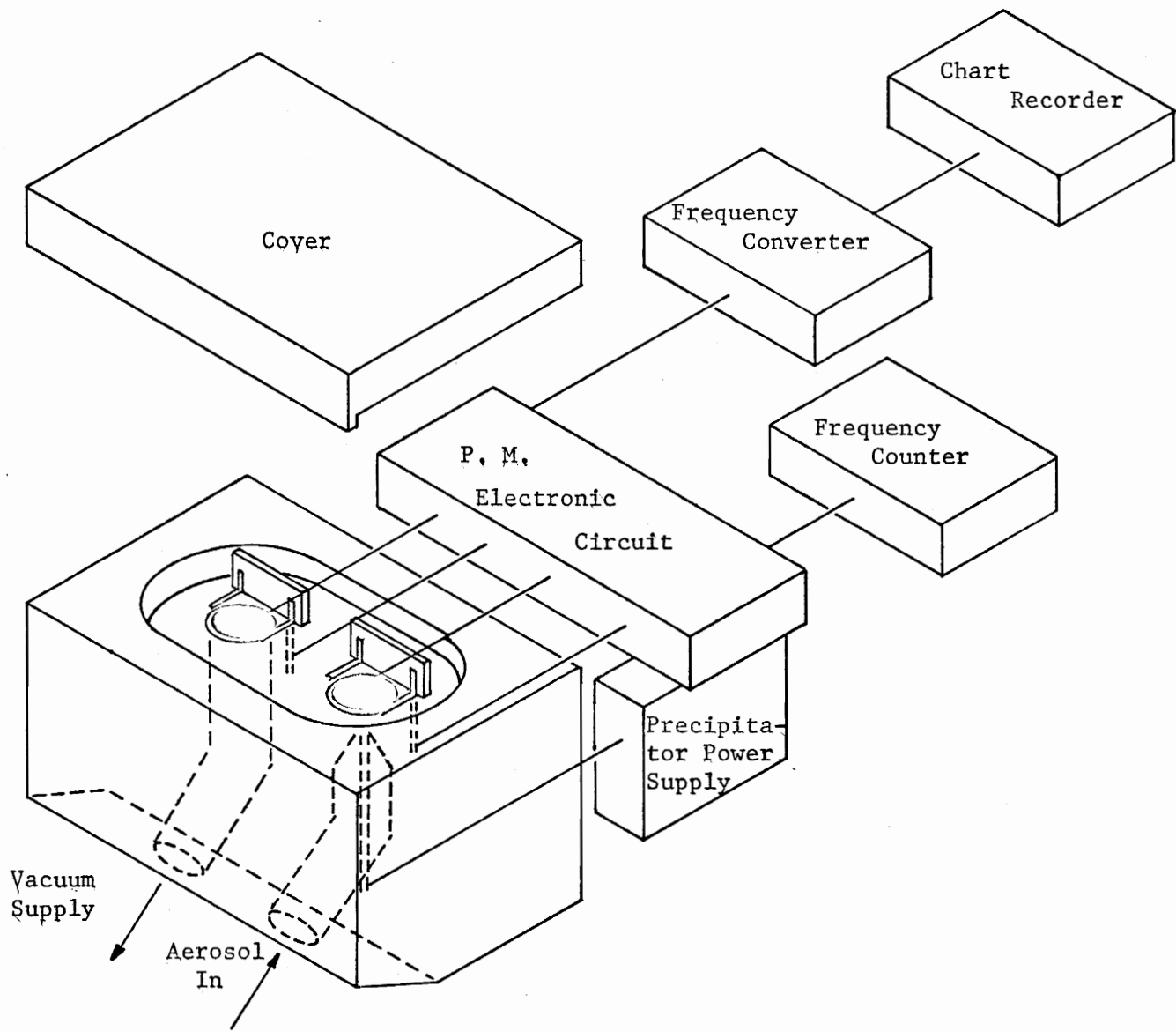
Figure 2. Results of electrostatic precipitator efficiency tests.(9)

microbalance using this type of crystal is 0.01 to 20.0 μ,
While Thermo Systems, Inc.,(15) gives the range as 0.01to 15.0 μ.(8
9,12,13)   The lower size range of 0.01 μ is established by the
efficiency of the electrostatic precipitator. The upper limit is a
function of the ability of the piezoelectric microbalance to weigh
the particles. Olin(12) has found that ambient aerosol particles as
large as 50.0 μ will adhere to the crystal properly and that coating
the crystal with a sticky substance does not increase adhesion of the
particles except on the first monolayer. Subsequent layers which
must also adhere are unaffected. Figure 3 is a diagram of the
piezoelectric microbalance measuring system.

When the particulate loading on the crystal becomes too great,
the coupling between the particles and the surface becomes imperfect
and frictional energy losses become significant. The oscillating
circuit cannot stably drive the crystal, and the output frequency
becomes unstable. The point at which the frequency becomes unstable
is approached abruptly and is usually detected by fluctuating readings.
Once this point is reached it is necessary that the crystal be cleaned.
The total frequency shift before cleaning is dependent on the type of
aerosol being sampled. Olin(9) has shown that with atmospheric
aerosols the maximum allowable crystal deposit is approximately 50.0
to 100.0 μg. In a separate experiment Olin(12) determined that a total
deposit of 42 μg of atmospheric particulate matter was allowable on
the crystal before saturation.

Figure 3. Complete particle mass concentration measuring system. (9)

Cover

Chart
Recorder

Frequency
Converter

Frequency
Counter

P. M.
Electronic
Circuit

Precipita-
tor Power
Supply

Vacuum
Supply

Aerosol
In

16

Error may be caused by the addition of water vapor or low vapor pressure materials adhering to the particulate matter. These contaminants are collected on the crystal causing a sensing of mass and later evaporate causing a decrease in mass. These errors only become significant when the change in frequency, Δf, or the sampling time, Δt, becomes very small.

Chuan(16) has designed a piezoelectric microbalance and successfully measured the mass concentration of particulate matter from aerial measurements of atmospheric particulates, discrete particulate emission sources, stack emissions, baghouse emissions, precipitator exhaust, coal fired electrical power plant exhaust, and automobile exhaust.

Carpenter and Brenchley(17) have designed a piezoelectric cascade impactor which collects particles without the use of a electrostatic precipitator. The instrument collects solely by impaction and separates the particles into four classes according to size. This instrument has also been successfully applied to the measurement of ambient particulate matter.

III. EXPERIMENTAL PROCEDURES

There are some difficulties associated with comparative sampling between the piezoelectric microbalance and the high volume sampler. It was necessary to lengthen the sampling time of the piezoelectric microbalance to correspond to the 24 hour sampling time of the high volume sampler. There were two choices as to how this could be accomplished. The first was to lower the 1 LPM sampling rate of the piezoelectric microbalance to some other value such as 0.5 LPM. Figure 2 shows that a sampling rate of 0.5 LPM would increase the efficiency of the electrostatic precipitator for any given corona current, but Chang and Smith(5) have shown that particle deposition within the sampling lines increases with a decrease in the sampling rate. This method was not employed for two reasons: because the two foot sampling line inlet would produce an error due to the deposition of particulate matter in the sampling lines and within the apparatus itself, and because the reduction to 0.5 LPM would still require that the ambient particulate concentration be less than 50 $\mu g/m^3$ to preclude overloading the crystal. The second method of lengthening the sampling time was to dilute the sampled gas stream with 9 parts out of 10 of filtered air. This was accomplished by utilizing a 9:1 dilution head which was obtained from Thermo-Systems, Inc. The dilution head apparatus was installed in the sampling train with 2 rotometers and 2 vacuum pumps. One rotometer was placed so as to monitor the 1 LPM sampling rate of the piezoelectric microbalance

18

and the other rotometer was placed so as to monitor the 0.9 LPM rate of the extracted and filtered air. Both rotometers were calibrated after installation in the sampling train. One vacuum pump was utilized in conjunction with each of the rotometers. Figure 4 is a diagram of the sampling train showing the locations of the various components in relation to the piezoelectric microbalance.

It was decided to select a comparative high volume sampling station with a history of relatively low ambient particulate concentrations in order to insure that the crystal in the piezoelectric microbalance would not become saturated before the 24 hour sampling time was over. Olin(12) has calculated that an ambient aerosol concentration of 75 $\mu g/m^3$ should allow a continuous sampling time in excess of 28 hours before crystal cleaning is necessary.

The frequency output from the piezoelectric microbalance was fed into a Hewlett Packard Model 500C Electronic Tachometer which produces a voltage pattern in direct relation to the frequency pattern supplied. The voltage pattern from the electronic tachometer was fed into a Sargent Model SRG Potentiometric Strip Chart recorder which gave a direct readout of the frequency fluctuations produced by the piezoelectric microbalance. The beginning and ending frequencies for each 24 hour sampling period were verified by using a Hewlett Packard Model 5302A, 50 MHz Universal Counter. In order to obtain an accurate pattern of events within the 24 hour sampling period, the strip chart was divided into 24 one hour periods and the mass concentration computed for each period.
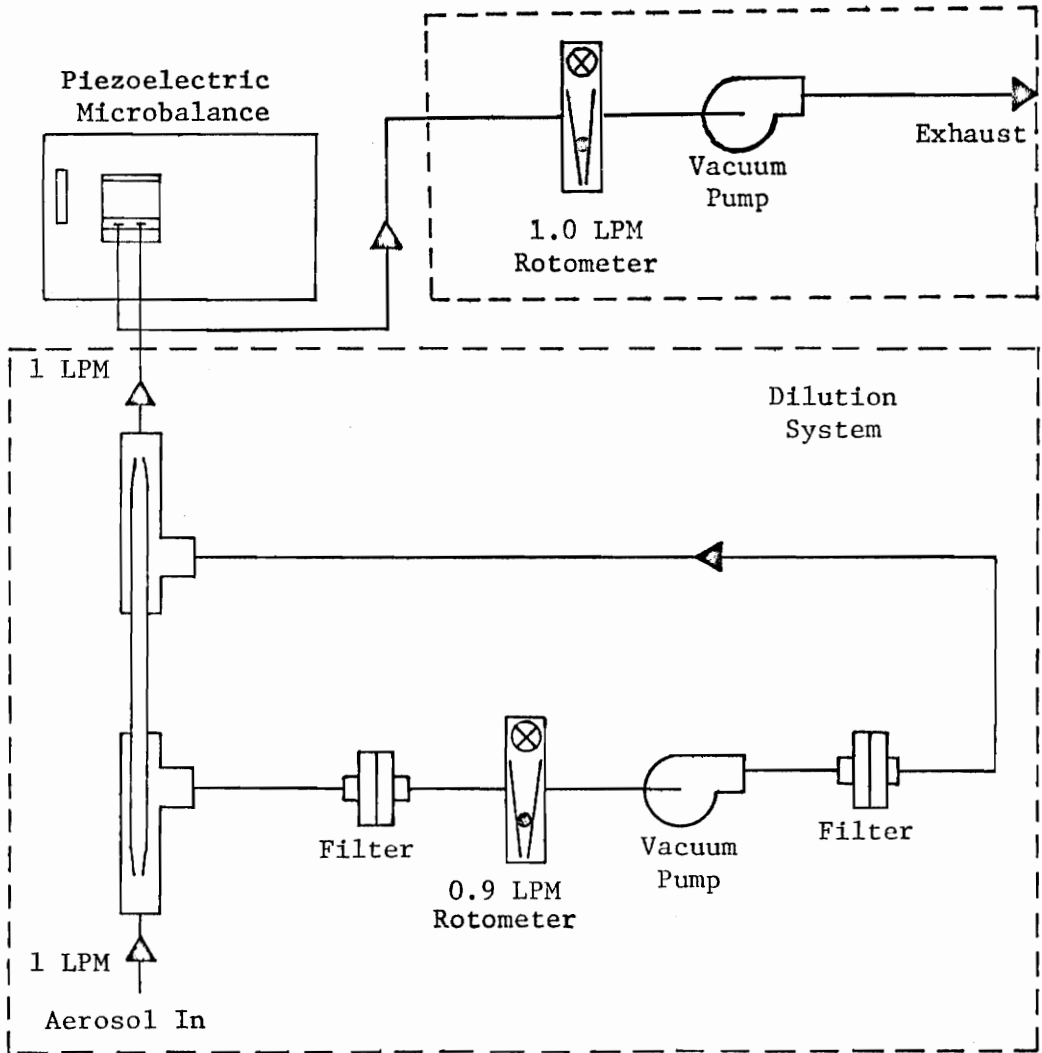
Figure 4. Diagram of 9:1 dilution ratio sampling train.

The sampling inlet tube was shielded from rain and other undesirable debris by the installation of a 50 μ cutoff sampling "hat." In essence this shield is designed to prevent the entrance of any particle larger than 50 μ.

For each sampling period the piezoelectric microbalance was started simultaneously with the high volume sampler at midnight and allowed to run continuously for a 24 hour period. The results from the high volume sampler and the piezoelectric microbalance were then compared for total particulate concentration.

IV. EXPERIMENTAL RESULTS

The results obtained from the comparison of the piezoelectric microbalance and the high volume sampler are presented in Figures 5 through 10.

The figures contain both the piezoelectric microbalance and the high volume sampler 24 hour average values for the particulate concentration. In each case the piezoelectric microbalance average is shown by the solid horizontal line and the high volume sampler average by the broken horizontal line. For each 24 hour sampling run the chart recording for the piezoelectric microbalance was broken down into one hour increments and the particulate concentration calculated for each hour. These data are presented in Figures 5 through 10, along with the averages and are shown as peaks and depressions depicting the variable nature of the hourly particulate concentrations.

The values obtained for the piezoelectric microbalance were consistently lower by 3-9 percent than the high volume sampler values. There was one exception to this which is presented in Figure 9. This exception was due to a malfunction of the electrostatic precipitator in the piezoelectric microbalance and the value was 83 percent below its corresponding high volume sampler average. The data for the malfunction were included as an example of the operation of the instrument without the precipitator.

The average particulate concentration values for both the piezoelectric microbalance and the high volume sampler are shown in Table 2.
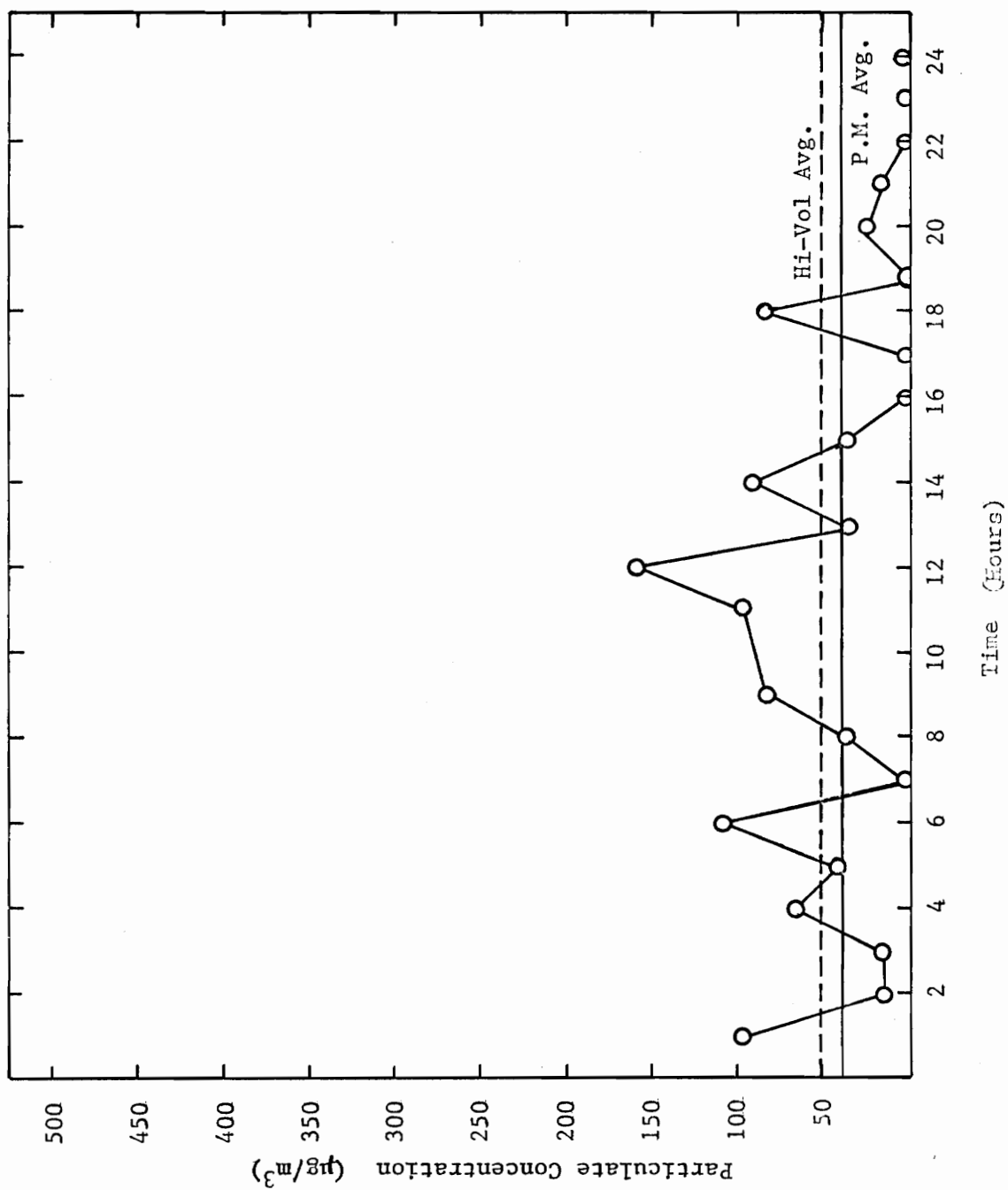
22

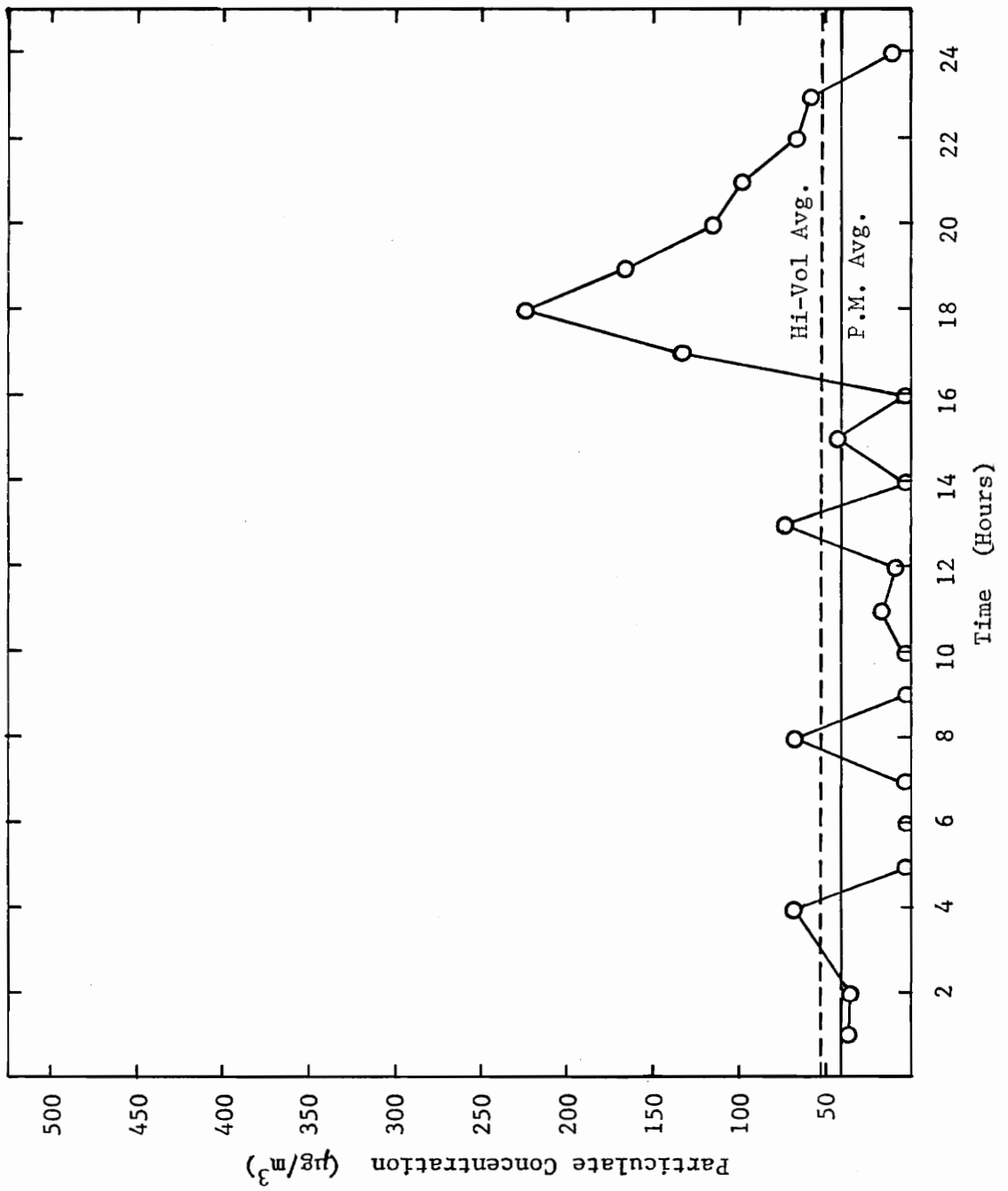Figure 5. Particulate concentration versus time for April 23, 1974.

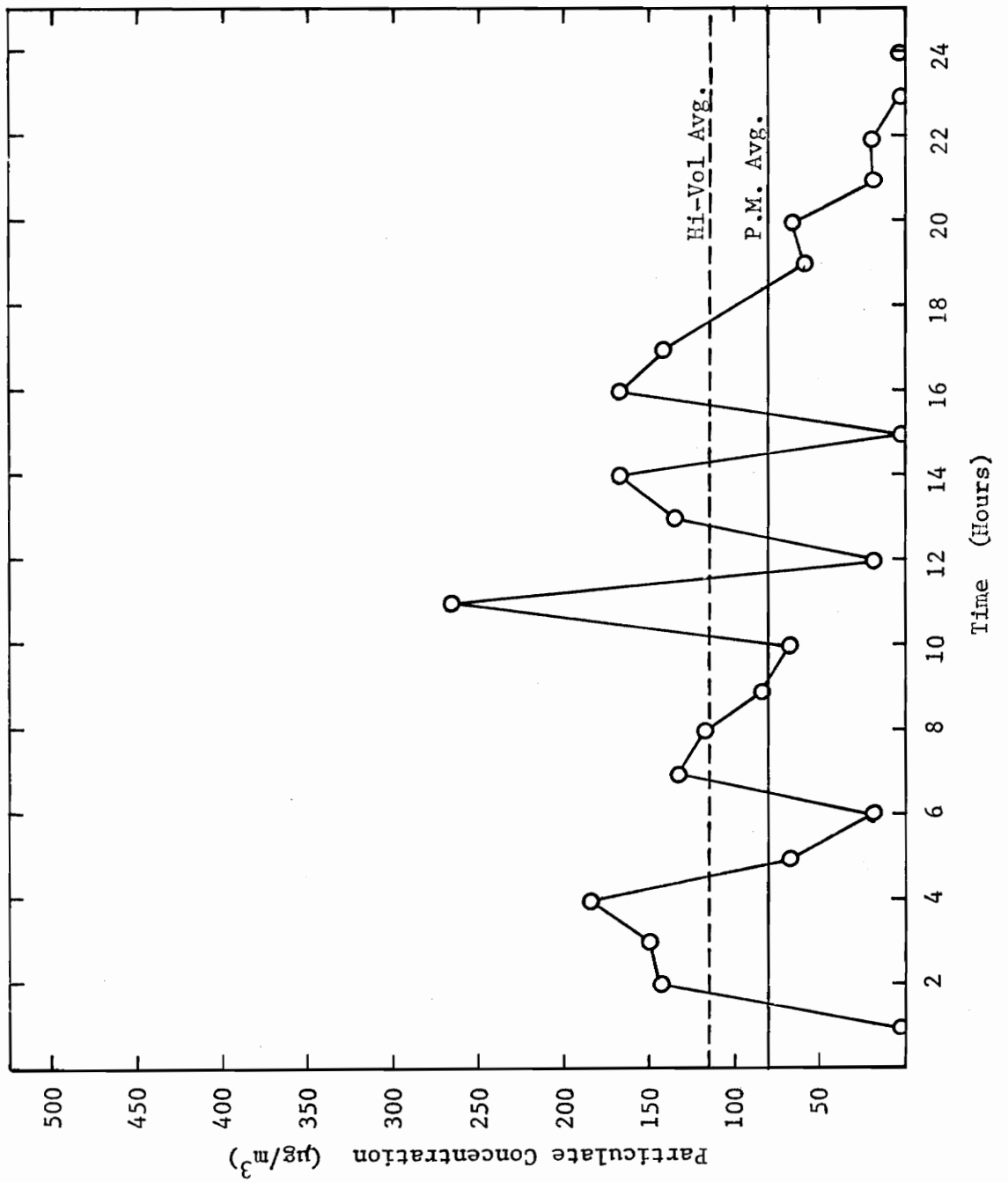Figure 6. Particulate concentration versus time for April 25, 1974.

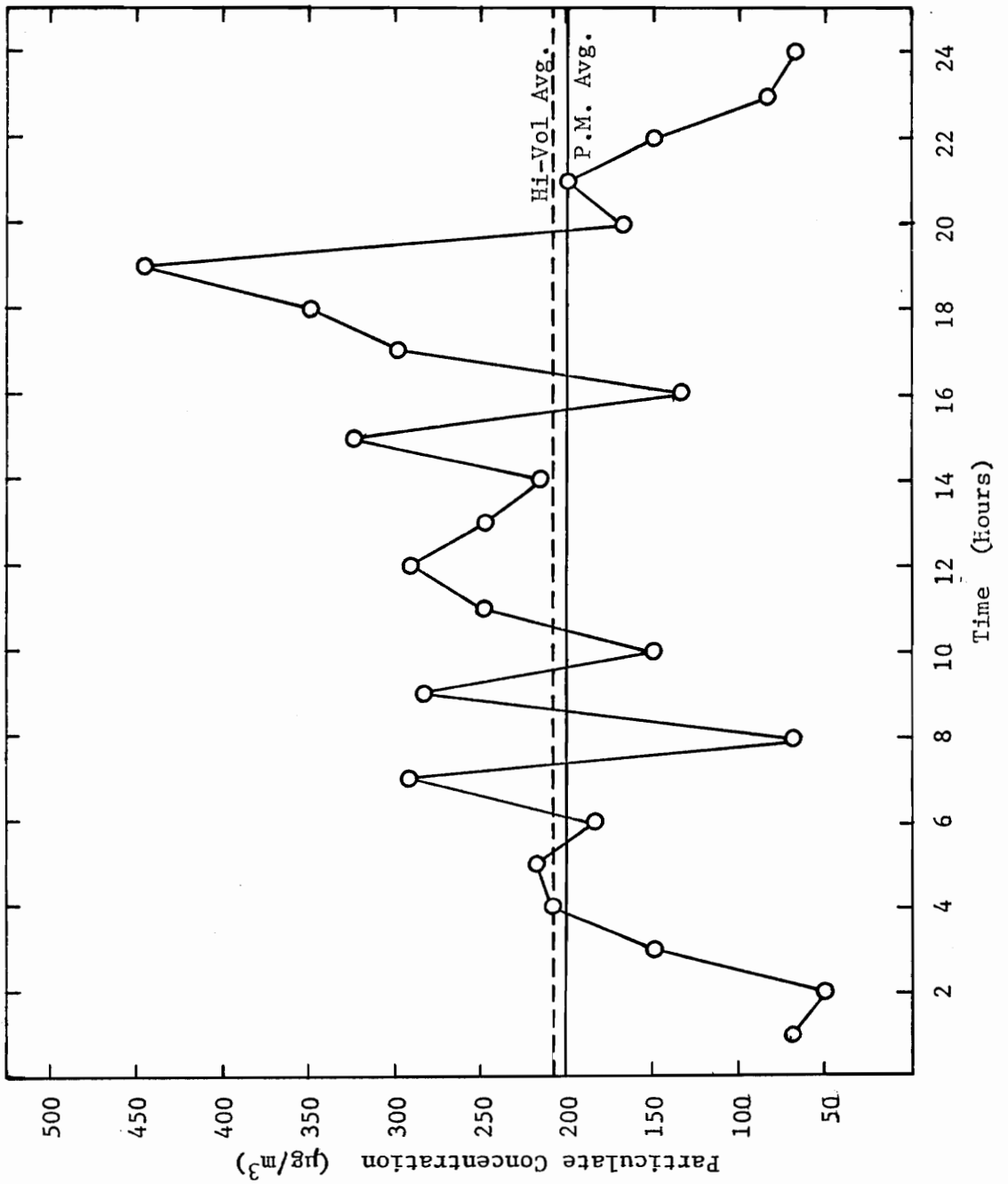Figure 7. Particulate concentration versus time for April 30, 1974.

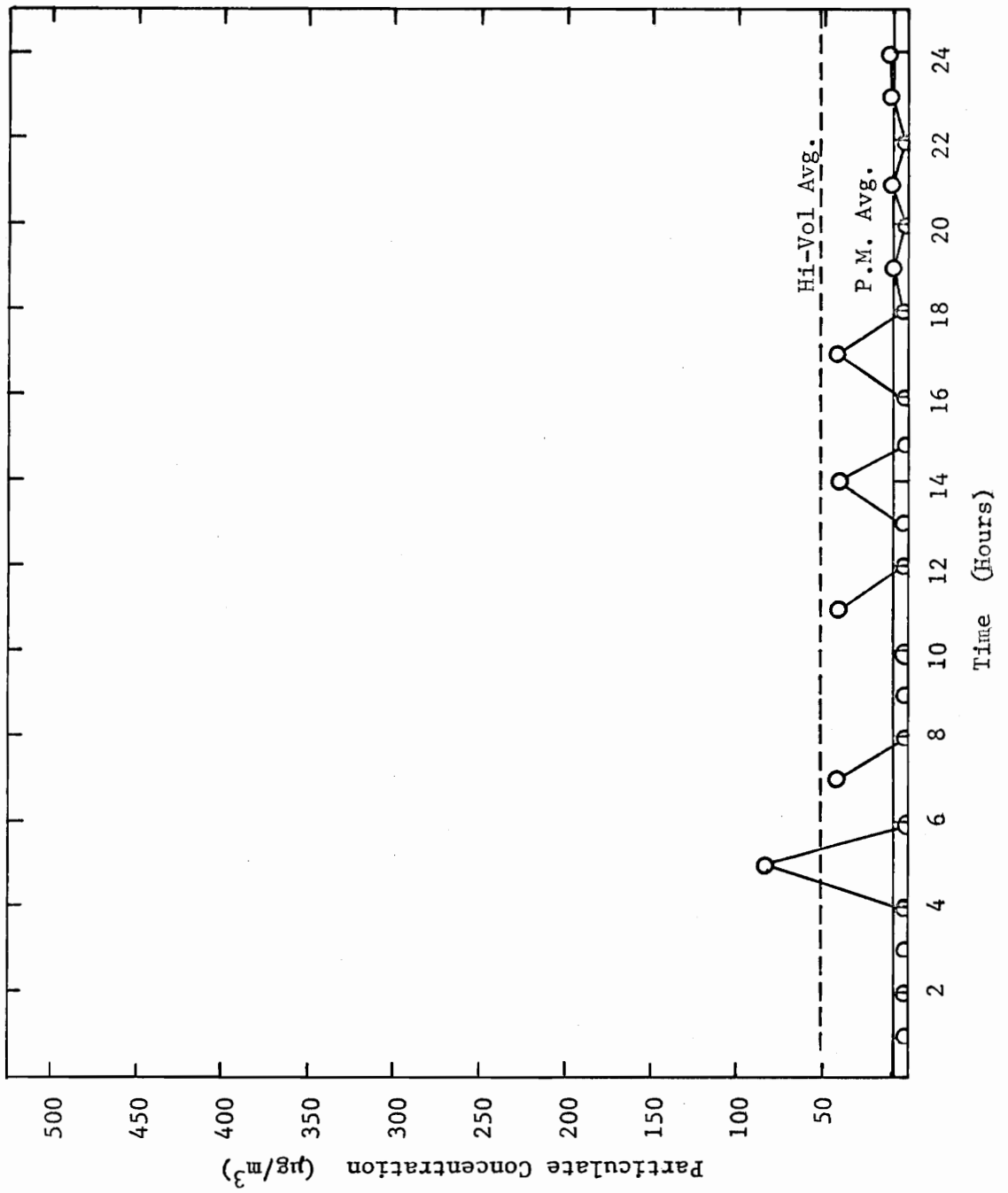Figure 8. Particulate concentration versus time for May 2, 1974.

Figure 9. Particulate concentration versus time for May 7, 1974.
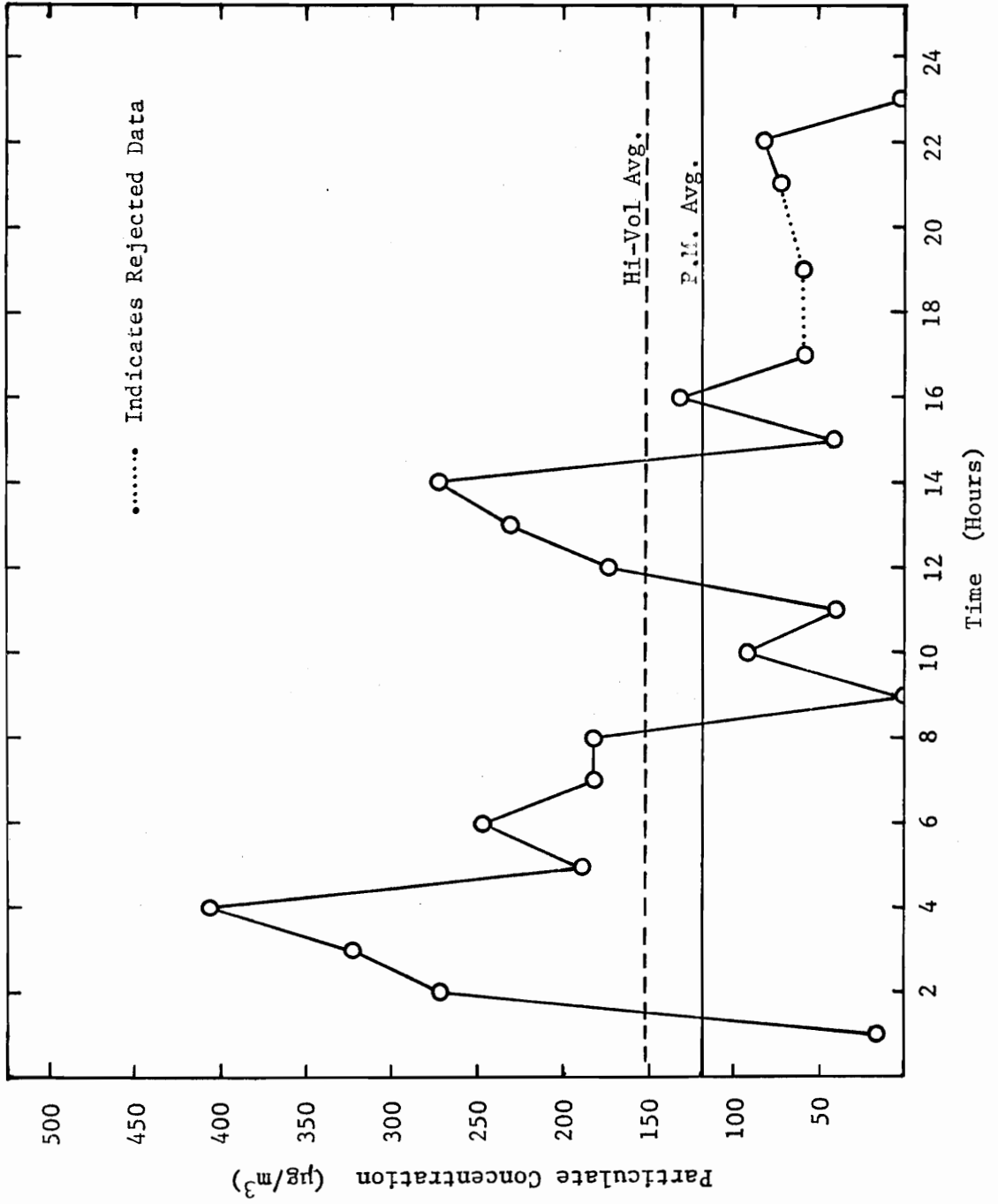
Figure 10.   Particulate concentration versus time for May 14, 1974.

TABLE 2

Data for the 24 hour Average Concentration of Particulate Matter for the Piezoelectric Microbalance and High Volume Sampler. ($\mu g/m^3$)

Date	Piezoelectric Microbalance	High Volume Sampler
April 23, 1974	46.4	53.7
April 25, 1974	39.5	52.4
April 30, 1974	82.2	114.8
May 2, 1974	203.9	209.3
May 7, 1974	8.67	50.7
May 14, 1974	123.4	150.5

A linear regression by least squares method was performed in computing a correlation coefficient for this data and is presented in Figure 11. The derived regression expression was

$$y = .905x + 29.1 \qquad\qquad (2)$$

where y is the average particulate concentration for the high volume sampler and x is the average particulate concentration for the piezoelectric microbalance. The correlation coefficient for this curve is 0.97.
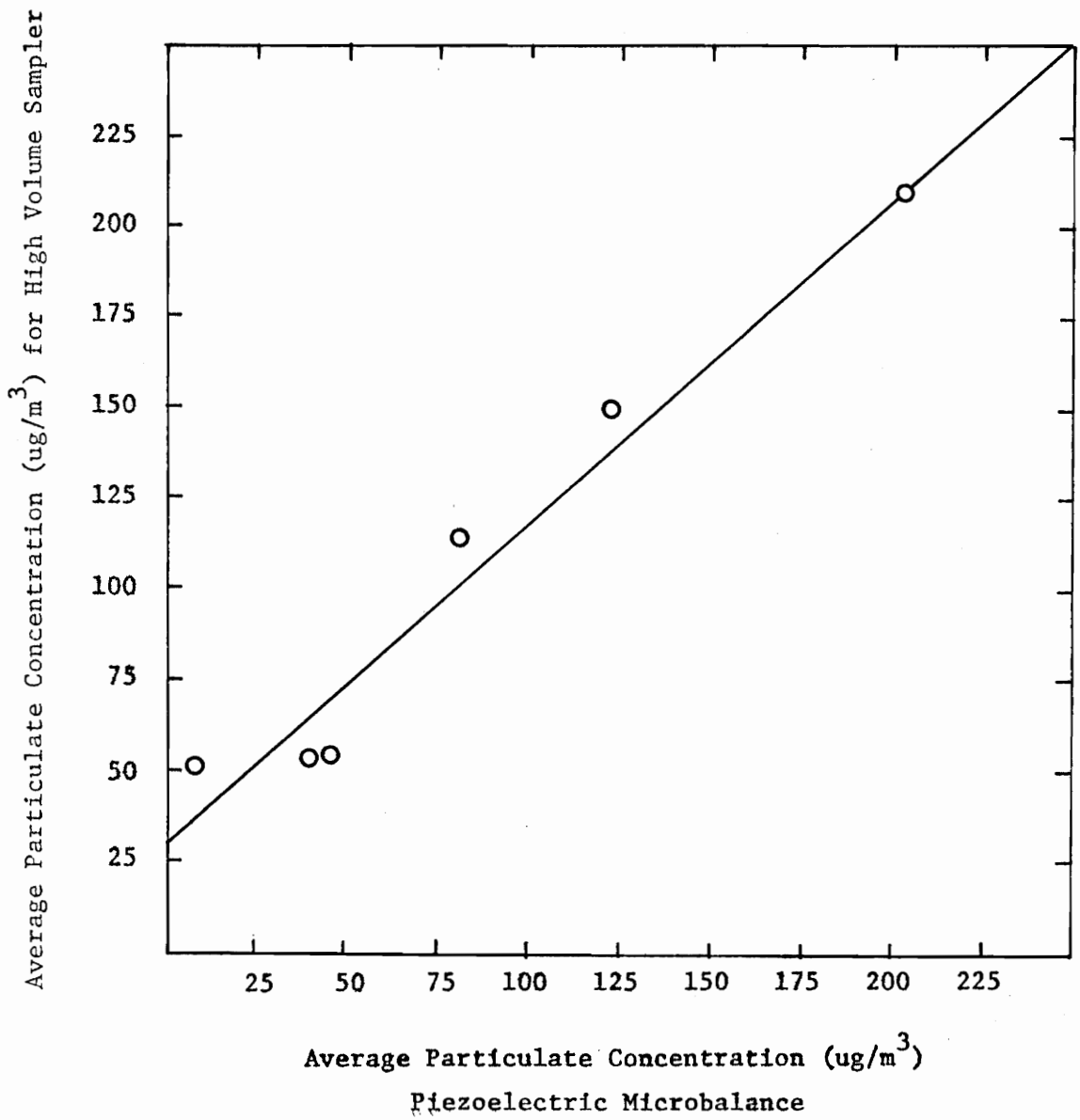
Figure 11. Average particulate concentration for high volume sampler versus piezoelectric microbalance.

V. ANALYSIS OF RESULTS

The fact that the values obtained from the piezoelectric micro-balance were consistently lower than those obtained from the high volume sampler may be due to the inability of the piezoelectric microbalance to weigh particles over 50 μ in diameter and the unreliability of this instrument in weighing particles in the range from 30 to 50 μ. As the diameter of the particles approaches 30 μ the increased mass causes the particle to break away from or roll over the collecting crystal surface. It is necessary that a particle be firmly adhered to the surface or to another particle or particles that are firmly adhered if they are to affect the vibrating crystal and be weighed. The high volume sampler will collect particles larger than about 0.1 μ and is limited only by the collecting hood geometry and the size of particle that can be kept aloft in the atmosphere under the particular meterological conditions. For all practical purposes, the hood geometry will allow the entrance of particles approximately 100 μ and less.(1)

The use of the 9:1 dilution ratio as a means of lengthening the sampling time was successful in that it allowed 24 hour sampling times without "saturating" the crystal in the piezoelectric micro-balance. There was, however, a problem with the accuracy of the flows within the sampling train. A voltage check at the sampling site revealed a small amount of voltage fluctuation in the range of ± 5 volts. While the piezoelectric microbalance is not affected by

minor voltage fluctuations, the vacuum pumps displayed some sensiti-
vity by exhibiting small changes in the flow rates of sampled air
through the sampling train. It was necessary to maintain a flow rate
of 0.9 LPM through the dilution head as opposed to 1 LPM through the
sampling head. A small fluctuation in the 0.9 LPM rate would produce
significant errors in the results. For this reason large rotometers
which could be read to within 0.05 LPM were employed. It was assumed
that the voltage fluctuations would average out over the 24 hour
sampling period. The voltage changes were observed over a 1 hour
period and found to cause fluctuations of approximately 0.05 LPM in
the flow rate. The flows were adjusted to the midpoint of these
fluctuations for each sampling run.

The chart recorder could be read to within $\pm$ 5 Hz, which re-
presents a concentration of $\pm$ 4.17 $\mu g/m^3$. In order to overcome this
error, a frequency counter was utilized to check the beginning and
ending frequencies to within $\pm$ 1 Hz. The accuracy of the 24 hour
averages is therefore $\pm$ 0.4 $\mu g/m^3$ while the accuracy of the hourly
fluctuations in concentration is $\pm$ 4.17 $\mu g/m^3$.

Some problems were experienced regarding the reliability of
the piezoelectric microbalance for long sampling times. Figure 9
shows the results of a sampling run during which the electrostatic
precipitator was malfunctioning. On two separate occasions the
electrostatic precipitator failed to operate properly. The first
failure was corrected by cleaning the precipitator chamber and

readjusting the electrostatic precipitator needle. The second
failure occurred as a result of a fracture in the needle believed
to be the result of faulty manufacturing methods.

VI. CONCLUSIONS AND RECOMMENDATIONS

The conclusions drawn from this investigation are:

1. The piezoelectric microbalance should be used in place of
    the high volume sampler for instantaneous measurement of
    ambient particulate concentrations but may produce
    consistently lower average readings because of the
    difference in the size range of particles measured.

2. The piezoelectric microbalance will provide data on the
    particulate concentration at any instant during the
    sampling time but consideration must be given to the
    fact that it is not reliable in measuring particles
    with a diameter greater than about 30 μ.

3. The limiting factor on the sampling time is the point
    at which the crystal becomes "saturated."

4. Dilution methods can be used to effectively extend
    the sampling times of the piezoelectric microbalance.

5. When used with a dilution system, the accuracy of the
    sampling procedure is limited by the accuracy of the
    sampled-air flow rate measuring system.

A number of recommendations are suggested:

1. A constant voltage transformer should be used in areas where voltage fluctuations occur.

2. The use of the 9:1 dilution ratio should be avoided in favor of 2:1 or 4:1 because of the difficulty in maintaining accurate sampled-air flow rates.

3. A chart recorder which can be read with an accuracy of 1 Hz should be used in conjunction with the piezoelectric microbalance.

4. To obtain average concentrations over long sampling times, the piezoelectric microbalance should be used in conjunction with some other reliable method such as the high volume sampler.

VII. REFERENCES CITED

1. "Title 42 – Public Health," *Federal Register*, Vol. 36, No. 84, April 30, 1971.

2. Pulker, H. K., and Schadler, W., "Factors Influencing the Accuracy of a Quartz Crystal Oscillator as a Thickness Monitor for Thin Film Deposition," *Il Novo Cimento*, 57B(1), 19 (1968).

3. Thermo Systems, Inc., 2500 Cleveland Avenue, St. Paul, Minnesota.

4. Perkins, H. C., *Air Pollution*, McGraw Hill, 223 (1974).

5. Hsing-Chi Chang and Smith, T. M., "Experiment on Particle Deposition in a Horizontal Sampling Line," *American Industrial Hygiene Association Journal*, 33, 11, 722 (1972).

6. "1973 Annual Book of ASTM Standards," *American Society for Testing and Materials*, Part 23, 893 (1973).

7. Hesketh, H. E., *Understanding and Controlling Air Pollution*, Ann Arbor Science Publishers, Inc., 170 (1972).

8. Olin, J. G. and Sem, G. J., "Piezoelectric Microbalance for Monitoring the Mass Concentration of Suspended Particles," *Atmospheric Environment*, 5, 653 (1971).

9. Olin, J. G., Sem, G. J. and Christenson, D. L., "Piezoelectric-Electrostatic Aerosol Mass Concentration Monitor," *American Industrial Hygiene Association Journal*, 32, 209 (1971).

10. Reist, P. C. and Burgess, W. A., "A Comparative Evaluation of Three Aerosol Sensing Methods," *American Industrial Hygiene Association Journal*, 29, 2, 123 (1968).

11. Corn, M., "Adhesion of Particles," *Aerosol Science*, Academic Press, New York (1966).

12. Olin, J. G., "Design and Operation of a Piezoelectric-Electrostatic Particle Microbalance for Automatic Monitoring of the Mass Concentration of Air-Borne Particles," *Advances in Instrumentation*, 26, Part 1, 558 (1972).

13. Olin, J. G., "Airborne Particle Monitoring Applications of the Particle Mass Monitor System," *Joint Conference on Sensing Environmental Pollutants*, Palo Alto, California, November 8-10, AIAA Paper No. 71-1100, p. 1 (1971).

14. Thermo Systems, Inc., "Air Quality Monitoring Experiments with the Particle Mass Monitor System," Technical Note 6.

15. E. I. Dupont, Inc., "How Do You Weigh Airborne Particles," Journal of Teflon, 22 (1970).

16. Chuan, R. L., "Measurement of Particulate Pollutants in the Atmosphere," Joint Conference on Sensing of Environmental Pollutants, Palo Alto, California, November 8-10 (1971).

17. Carpenter, T. E. and Brenchley, D. L., "A Piezoelectric Cascade Impactor for Aerosol Monitoring," American Industrial Hygiene Association Journal, 33, 8, 503 (1972).

COMPARATIVE PARTICULATE SAMPLING METHODS

by

Frank Butler Twitty

(ABSTRACT)

At present there are several methods of determining the mass concentration of suspended particulate matter in ambient air. Most of these methods, including the EPA reference method, require manual filtering and weighing of the collected sample. This is both costly and time consuming and is subject to a considerable amount of human error. Another serious drawback to most of these procedures is the long (24 hour) sampling times required, and the resultant insensitivity to fluctuations due to the long averaging intervals.

The piezoelectric microbalance and high volume sampler techniques were employed in this investigation to measure the mass of ambient undifferentiated particulate matter. The results were compared and conclusions drawn concerning the feasibility of using the piezoelectric microbalance along with or in place of the high volume sampler for measurement of ambient particulate matter.

It was found that the piezoelectric microbalance can be used in place of or along with the high volume sampler but consideration must be given to the fact that the piezoelectric microbalance will give lower average particulate concentration readings depending on the particle size distribution of the ambient particulate matter being sampled.