

AN ABSTRACT OF THE THESIS OF

John Francis Kowalczyk for the M. S. in Mechanical Engineering
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Title THE EFFECTS OF VARIOUS PRE-WEIGHING PROCEDURES
ON THE REPORTED WEIGHTS OF AIR POLLUTANTS COLLECTED
BY FILTRATION

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A study was conducted to determine what effects the type of pollutants, storage conditions, storage time, and method of sample conditioning have on the reported weight of an air pollutant collected by filtration. Samples were collected from a refuse dump, an arc furnace smelting nickel ore, and a rotary kiln burning car bodies. These three sources of samples represented extremes in moisture content and organic matter. An initial weight was determined; then each sample was put through a certain procedure before final weighing. These procedures included storage times from one to twenty-five days, freezing and ambient storage, and sample conditioning by heating and desiccation. Data were analyzed using a graphical approach.

The type of pollutant, storage conditions, storage time, and method of sample conditioning significantly affected the reported

weights of air pollutants. It was found that the magnitude of the weight loss caused by a particular type pollutant and pre-weighing procedure was dependent on the percent of organic matter initially present.

The method of sample conditioning had the greatest effect on weight losses. This effect, again, was dependent on the percent of organic matter initially present. Conditioning by heating for a certain sample containing 60% organic matter resulted in a 25% loss of sample weight. Time and storage conditions had slight effects on reported weights. These effects were amplified for pollutants containing high percentages of organic matter.

Frozen storage and conditioning by desiccation in all cases proved to be the best procedure to follow for minimization of errors affecting the reported weight of an air pollutant.

THE EFFECTS OF VARIOUS PRE-WEIGHING PROCEDURES
ON THE REPORTED WEIGHTS OF AIR POLLUTANTS
COLLECTED BY FILTRATION

by

JOHN FRANCIS KOWALCZYK

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Associate Professor of Mechanical Engineering

In Charge of Major

Redacted for Privacy

Head of Department of Mechanical Engineering

Redacted for Privacy

Dean of Graduate School

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THE EFFECTS OF VARIOUS PRE-WEIGHING PROCEDURES
ON THE REPORTED WEIGHTS OF AIR POLLUTANTS
COLLECTED BY FILTRATION

INTRODUCTION

With the rapidly growing interest in air pollution and cleaner air has come increased research into techniques and methods of determining the extent of pollution and the magnitude of source emissions.

Being a relatively new field, air pollutant analysis suffers from the lack of a good standard methods text similar to "Standard Methods for Analysis of Water, Sewage, and Industrial Wastes," used by sanitary engineers. Ironically, many methods for analyzing air pollutants, especially gaseous pollutants, have come from this text; however, presently many industries, research firms, and government agencies have their own special method for determining certain constituents of the atmosphere.

There is becoming available an ever increasing supply of texts, papers, and bulletins dealing with air pollutant analysis. Among these are (1) the "1964 Book of ASTM Standards", (2) the "Source Testing Manual" by the County of Los Angeles, and (3) Jacob's "Chemical Analysis of Air Pollutants". Yet among even these publications can be found differences in techniques when analyzing similar

pollutants.

If the same accuracy could be attained by each method there would be no problems. Needless to prove, however, is the fact that precision and accuracy is not in many cases the same. Errors introduced by these different methods are realized by most but discounted as negligible since at present approximate answers are considered satisfactory. What will be needed in the future as the field of air pollutant analysis becomes more refined is a close look and evaluation of these methods to determine which one is best suited for a particular type sample.

Work is beginning on this subject with such studies as (7) Stockman's on the variability of dust fall analysis due to the container and the collecting fluid. Nothing, however, has been done along the lines of critically looking at the various methods used in determining the concentration of particulate matter in the atmosphere by gravimetric analysis of a filtration media. It will be the objective of this thesis to follow along this path.

Basically, gravimetric analysis is applied to a filtration media before and after a gas stream containing particulate has been passed through. The difference in weight of the filter before and after the sampling period is attributed to the particulate matter. Knowing the volume of gas sampled it is possible to arrive at a particulate concentration. This information is usually one of the first things needed

in any air pollution survey.

Presently, the Public Health Service monitors suspended particulate in many cities of the United States through their National Air Sampling Network using filtration and gravimetric analysis. Other uses of this method include determining the magnitude of emissions from stacks and obtaining efficiencies of pollution control equipment.

Although being an inexpensive, simple, and popular control measure, a close look at the details involved in gravimetric analysis will reveal the many sources of inaccurate results.

The first problem arises from the filter media itself. Materials such as cellulose paper or membranes are hygroscopic in nature. Their moisture content varies with ambient conditions; thus, the assumption that filter weight is the same before and after a test is false. It is possible to overcome this problem to some extent by using a control filter as recommended by the Millipore Company (4). The weight change due to moisture pick-up or loss on the control filter is applied to the test filter thus cancelling any change in test filter weight. This method, however, assumes that the moisture pick-up or loss is the same for both filters. This may or may not be the case.

Glass fiber filters have practically eliminated the moisture problem due to their non-hygroscopicity (8).

The hygroscopic nature of the particulate sample creates another problem which does not have a simple solution. From the moment a particle impinges on the surface of a filter it may tend to alter its true weight by either releasing or absorbing moisture.

The rate of this change will depend on the time from collection to analysis, the nature of the material, and the environmental conditions to which the material is exposed.

Of most importance in affecting the final weight, however, is the method used to remove extraneous moisture picked up after the sample has been collected. Proper procedure in this step can reduce the effects of the three previously mentioned variables to, or close to, zero.

A fifth variable, volatile matter, presents an irreversible problem whose effects can be influenced by any or all of the four previously mentioned variables.

Thus: (1) the hygroscopic nature of the sample, (2) the presence of volatile matter, (3) the environmental conditions to which the filter and particulate is exposed, (4) the storage time, and (5) the method of sample conditioning are all factors which must be considered when attempting to find the source and magnitude of errors affecting the reported weights of air pollutants collected by filtration.

Most pollutants sampled contain a variety of substances in addition to a certain characteristic one. If hygroscopic matter is present in a sample, nothing can be done to eliminate it as it contributes to the total weight. The same reasoning holds for volatile matter.

However, by careful control of: (1) storage conditions,

(2) storage time, and (3) the method of sample conditioning it may be possible to minimize or eliminate errors affecting the reported sample weight.

THESIS OBJECTIVES

The major objective of this thesis was to determine if and to what extent (1) the type of sample, (2) the storage conditions, (3) the storage time, and (4) the method of sample conditioning affects the reported weight of an air pollutant sample.

Furthermore, it was hoped that from the fulfillment of the above objective would come a model which would describe the best procedure to follow in order to keep errors in reported weights to a minimum.

In addition the data collected should give persons in the field of air pollution some concrete answers to refer to when questions arise as to how accurate and precise are present methods of determining particulate concentrations.

PROCEDURE

Experimental Design

Sample Sources

The first step in planning this study was to choose the pollutant to be sampled. In air pollution work a multitude of materials are encountered, from power plant fly ash to ragweed pollen. In order to cover a range of materials it was decided to sample from three different sources.

Effluent from an arc furnace smelting ore at the Hanna Nickel Mine in Riddle, Oregon was the first chosen (Figure 1). The material emerging from this furnace consisted mostly of iron oxides, silicates, sulfates, nickel oxides, and other trace materials (6). It was felt that samples would be low in organics and volatile matter due to the charge make-up and extreme heat in the melting operation and would, thus, represent a typical industrial emission characterized by low moisture, and inert inorganic material.

The effluent from the Bureau of Mines experimental rotary kiln in Albany, Oregon was the second material chosen (Figure 2). The kiln, with a capacity of 600 pounds per hour, was being used in a study to determine the feasibility of making new steel from automotive scrap. The kiln's function was to destroy any burnable



Figure 1. Site of the Hanna Nickel Mine showing emissions from arc furnaces.

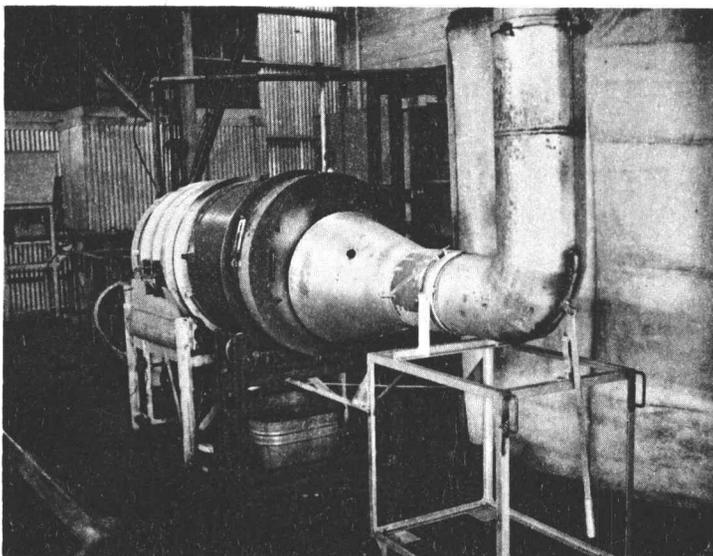


Figure 2. The Bureau of Mines rotary kiln.

material contained in the scrap before the metal was transferred to a melting and refining furnace. The metal scrap contained a variety of materials, notably greases, oils, paints, ordinary dust and dirt, and glass. It was also presumed that many metallic compounds were vaporized by the gas fired kiln since in some areas temperatures reached 600°C.

The kiln emission was chosen as a source of the second sample because it was felt that the material collected would represent an average mixture of moisture, organic and inorganic, volatile and non-volatile, and burnt and unburnt material.

The site of the third particulate sample was chosen as the Corvallis, Oregon municipal refuse dump (Figure 3). Here refuse was being burned in an open pit with no control of combustion. It was thought that the particulate from this source would be high in organics and moisture content. In addition atmospheric samples could be taken with a high-volume sampler which would represent a standard method of sampling ambient air.

By choosing these three samples a wide range of moisture content and material composition was covered. It was hoped this would make the results of this thesis applicable to many of the materials normally encountered in air pollution work.



Figure 3. High-volume sampler collecting smoke from the Corvallis dump.

Storage Conditions

After a sample is collected it may be stored for a period of time before analysis. If this time is fairly lengthy, as might be the case in some field studies, storage conditions may significantly affect the final weight.

Normally samples are stored in envelopes or other similar containers and subjected to ambient conditions. If volatile matter is present the sample might be frozen. The feeling here is that freezing will prevent much of the material with substantial vapor pressures

from volatilizing. Storage under (1) freezing and (2) ambient conditions were, thus, investigated in this study.

Storage Time

Storage time is a variable which depends mainly on the time and equipment available for analysis. To get a general idea of long term storage effects, final weighing was conducted one, seven and twenty-five days after sample collection.

Method of Sample Conditioning

Sample conditioning to remove moisture picked up after sample collection is done before final weighing in a variety of ways. Millipore Filter Company (4) suggests drying the sample at 90°C for one-half hour then cooling to equilibrium under ambient conditions. Jacobs (3) recommends drying at 103°C overnight and then cooling in a desiccator. ASTM standards (1) suggests repeated drying at 110°C for one hour, cooling in a desiccator and weighing until a constant weight is reached. If temperature sensitive or volatile material is involved ASTM suggests only desiccation.

In order to cover these methods in general, two basic procedures were followed. Method one involved heating at 105°C for 12 hours followed by one-half hour of desiccation and one-half hour of stabilization in a temperature and humidity controlled environment.

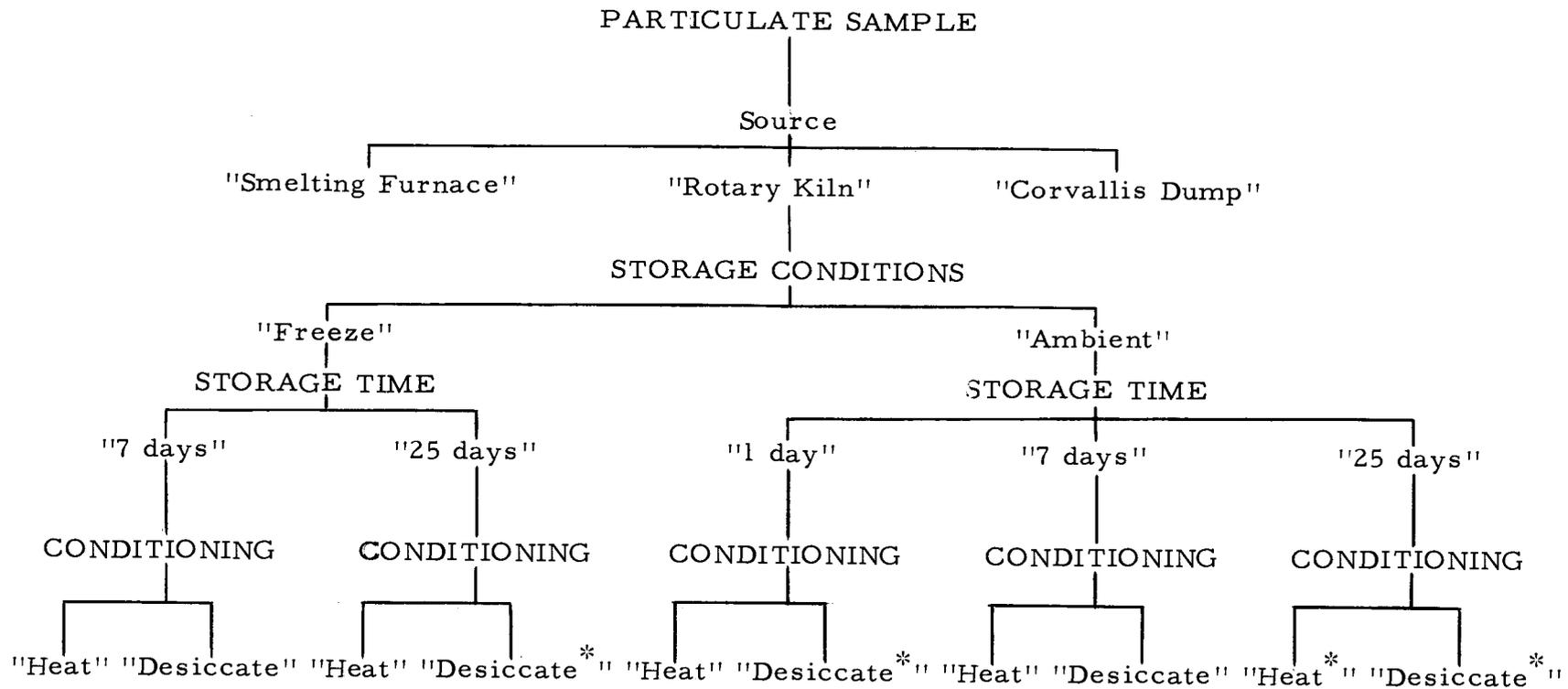
Method two consisted of desiccation for one-half hour followed by one-half hour of stabilization in a temperature-humidity controlled environment.

It was hoped, by this procedure, to determine distinct differences in weight analysis created by desiccation and heating. Stabilization in a temperature and humidity controlled environment was done only to provide similar weighing conditions for all samples. Comparison of weights taken at different periods of time could, thus, be justifiably made.

Final Experimental Model

Final integration of the two storage conditions, three storage times, and two methods of sample conditioning resulted in the experimental design described by Figure 4. Freezing the sample for one day was discarded since it was felt information gathered from this test would be insignificant. It was, therefore, necessary to collect ten samples from each of the three sources. Duplicating each test to check precision made it necessary to collect 60 samples, 20 from each source.

Along with final weighing of each sample the amount of organic matter present in certain samples was checked by benzene extraction to: (1) classify the material by percent organics and (2) check if any procedure significantly affected organic content (volatile and



* Extractions run to determine percent organics

Note: Ten samples needed per source.

Figure 4. Experimental design.

hygroscopic matter is normally organic).

One day ambient storage and desiccation, 25 day freezing and desiccation, 25 day ambient storage and heating, and 25 day ambient storage and desiccation procedures were chosen for the extractions since it was felt they would represent extremes in weight losses.

A particle size analysis was also conducted for each source to further identify and classify it.

Sample Collection Procedure and Equipment

A high-volume sampler, which was especially modified to hold a quartered 8 x 10 inch glass fiber filter (Figure 5) was used to obtain samples of smoke from the Corvallis refuse dump. The idea of collecting four samples at once not only cut down on the sampling time but provided four samples which should have been very similar in composition and thus suitable for comparison of organic content. One of the simultaneous samples was used for the four procedures thought to give extremes in weight losses.

Fifty milligrams of particulate was the minimum quantity of material desired to be collected on a piece of filter paper. It was felt that with this quantity a few percent change in particulate weight or organics could be detected. Sampling time was, therefore, chosen as one-half hour by some experimentation. The average flow rate of the high-volume sampler for this time was 50 CFM.

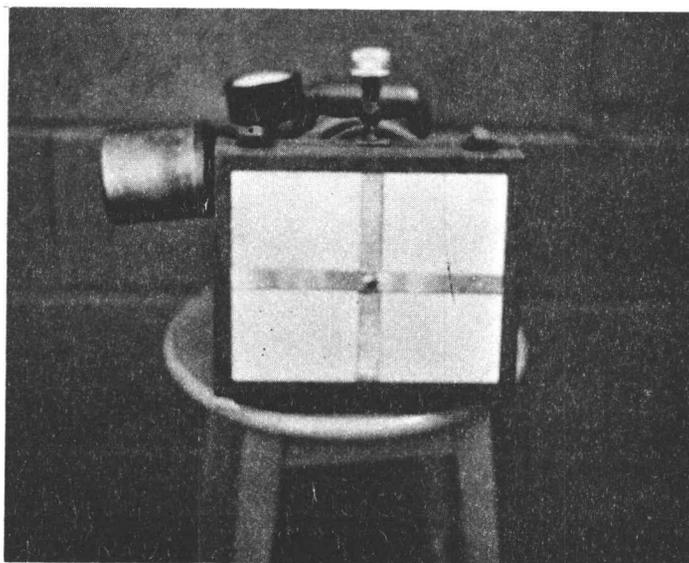


Figure 5. Modified high-volume sampler.

Stack sampling at the Bureau of Mines and the Hanna Nickel Mine required construction of a special enclosed filter holder and probe (Figure 6). The holder was designed for a standard five inch diameter Gelman glass fiber filter. As in the case of the high-volume sampler, provisions were made to take four samples simultaneously.

The holder was made from two metal dishes placed face to face. A two hundred mesh screen was used as a support and diffuser for the filter media. The sampling probe was made from 5/16 inch ID steel tubing. The final assembly simulated in construction a commercial stack sampling filter holder.

At the Hanna Nickel Mine sampling took place at the top of the number one arc furnace stack. A vacuum pump and gas meter were used to adjust the flow rate for isokinetic sampling. A sampling time of 15 minutes with a flow rate of one CFM was used.

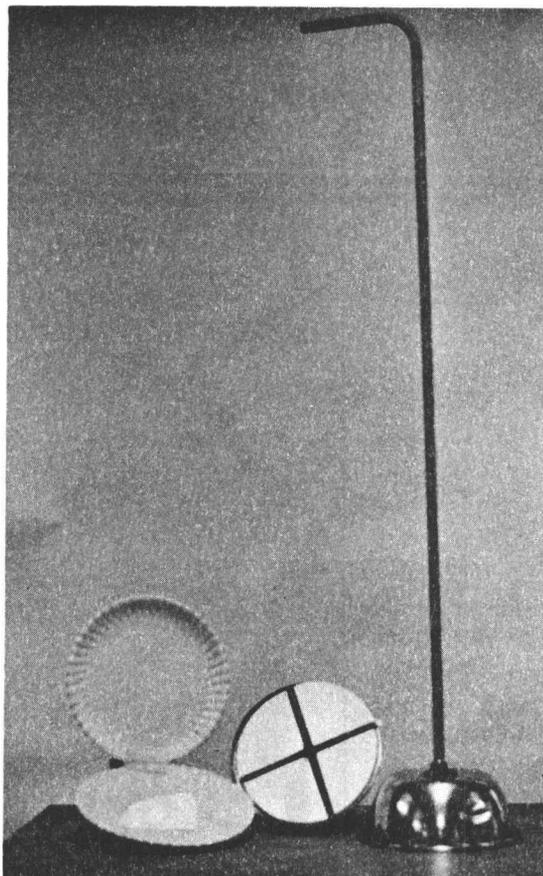


Figure 6. Stack sampling probe and filter holder along with paper plate storage container for filters.

At the Bureau of Mines similar sampling procedure was followed. Sampling took place at the outlet ventilation duct of the kiln.

At the time of sampling, transmission and engine parts were being burned in approximately one hour batch operations. To obtain the desired amount of particulate it was necessary to sample the smoke of the entire batch for each run.

Once the samples were taken, they were immediately placed in separate containers made from paper plates placed face to face (Figure 6). These containers provided protection from handling.

In addition, due to the bowl-like structure of the plates, nothing touched the surface of the filter containing the sample.

Standardized Weighing Procedure

The moment sampling is completed, the particulate on a filter has a certain weight which represents the true weight of the material as it appeared in the gas stream. From this moment on, environmental conditions will affect this true weight in some way as shown in Figure 7.

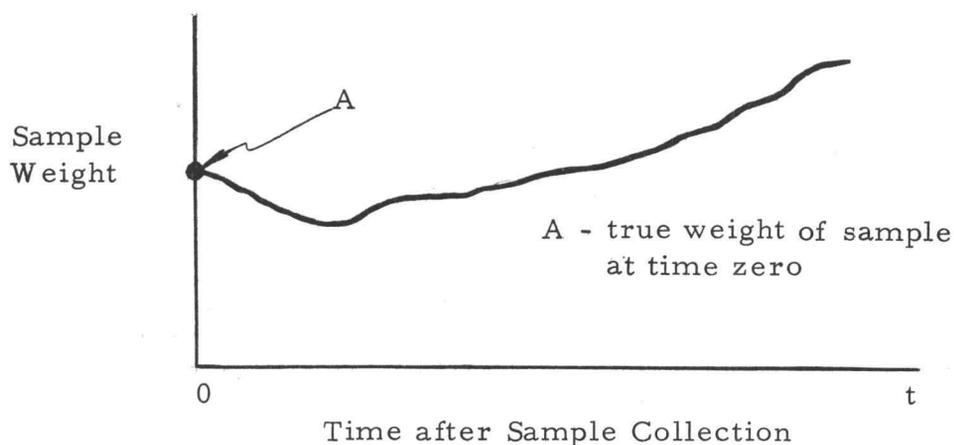


Figure 7. Typical variation in sample weight.

Practically, it is impossible to determine the weight at time zero. What is done, however, is to determine the weight at some later time. In order to base weight changes caused by different pre-weighing methods on some standard weight, it was decided to weigh all filters as soon as they could be returned to the lab. The weight

obtained was called the standardized weight and, for the purposes of this thesis, represented the weight of the particulate at time zero.

Figure 8 illustrates this concept.

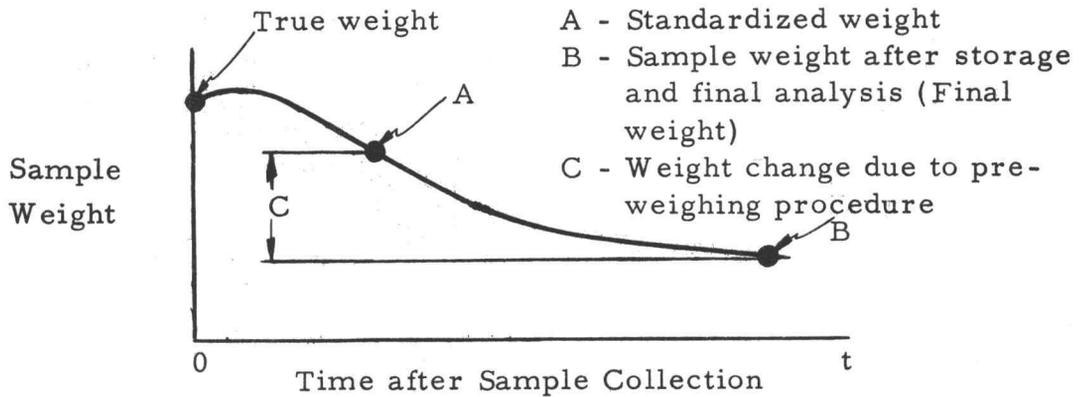


Figure 8. Relationship between true weight, standardized weight and final weight.

The time from sample collection to the lab varied from one hour for the dump and kiln samples to four hours for the arc furnace sample.

Upon arrival at the lab, each sample was taken to a temperature-humidity controlled room¹ where it was put in a desiccator² for one-half hour to remove extraneous moisture, and then stabilized for one-half hour in the controlled environment before determining

¹ Temperature was held at $75^{\circ} \pm 3^{\circ}$. Relative humidity was held at $54\% \pm 3\%$.

² Silica gel used as desiccant.

the standardized weight. Weighing was then done in the room on a Mettler model H6 digital macroanalytical balance to the nearest 0.1 milligram. Tare weights of the filters were determined in a similar manner.

After obtaining a standardized weight the filters were placed in their paper plate containers and stored under the prescribed method.

Freezing was done in a Kelvinator frost free refrigerator. Ambient storage took place in a room subject to the normal summer weather of western Oregon.

Sample Conditioning

After the proper storage time, samples to be dried by heating were placed in an oven at 105°C for 12 hours. From the oven they were taken to the temperature-humidity controlled room for the final weighing procedure.

Samples to be dried by desiccation were taken out of storage to the temperature-humidity controlled room for the final weighing procedure.

Final Weighing Procedure

Final weights were obtained in the temperature-humidity controlled room after a one-half hour period of desiccation and one-half

hour period of stabilization.

It is note worthy that this general weighing procedure followed for tare, standardized, and final weights provided as uniform conditions as possible for the samples and balance equipment. Any change in filter weight must, for all practical purposes, be attributed to the variables in question: that is (1) type of sample, (2) storage conditions, (3) storage time, and (4) method of sample conditioning.

Benzene Extraction Procedure

Benzene extractions using a Soxlet extractor (Figure 9) were run to determine the amount of organic matter present on a filter after final weighing.

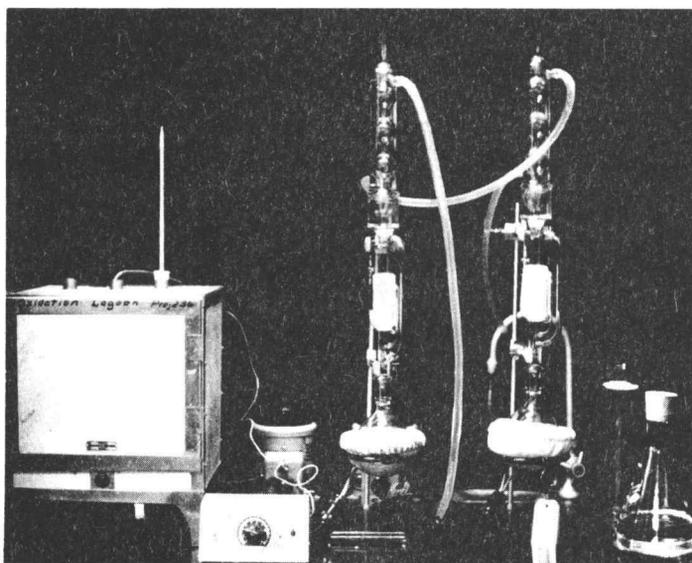


Figure 9. Benzene extraction apparatus for organics.

Extraction took place for four hours corresponding to 40 cycles. The benzene containing organics was evaporated in a tared dish at 70°C for approximately three hours, cooled in a desiccator and weighed. A series of blanks were run using an extraction thimble and clean filter to obtain an average background value.

Particle Size Analysis

A one-inch diameter Gelman membrane filter was used to obtain a particulate sample for size analysis. Two hundred particles were counted using light field microscopy. A Porton eyepiece was used to facilitate counting. Total magnification was 1000x.

RESULTS

Weight losses and percent organics based on standardized weights were calculated and tabulated (Tables 1, 2 and 3 of the Appendices).

Corvallis Dump Samples

1. Storage conditions, storage time and the method of sample conditioning significantly affected the final particulate weight (Figure 10).
2. Sample conditioning by heating had the greatest effect on the particulate weight loss. Based on one day storage, the average weight loss was 26.8%.
3. Storage conditions had no significant affect on weight losses for samples conditioned by heating (weight losses were approximately the same for both ambient and frozen storage).
4. Samples stored by freezing and conditioned by desiccation had the least weight losses. Average losses ranged from 0.7% for seven day storage to 2.2% for 25 day storage.
5. Ambient storage caused a higher weight loss than did storage by freezing for samples conditioned by desiccation. Average differences ranged from 1.3% for samples stored one day to 5.8% for samples stored 25 days.

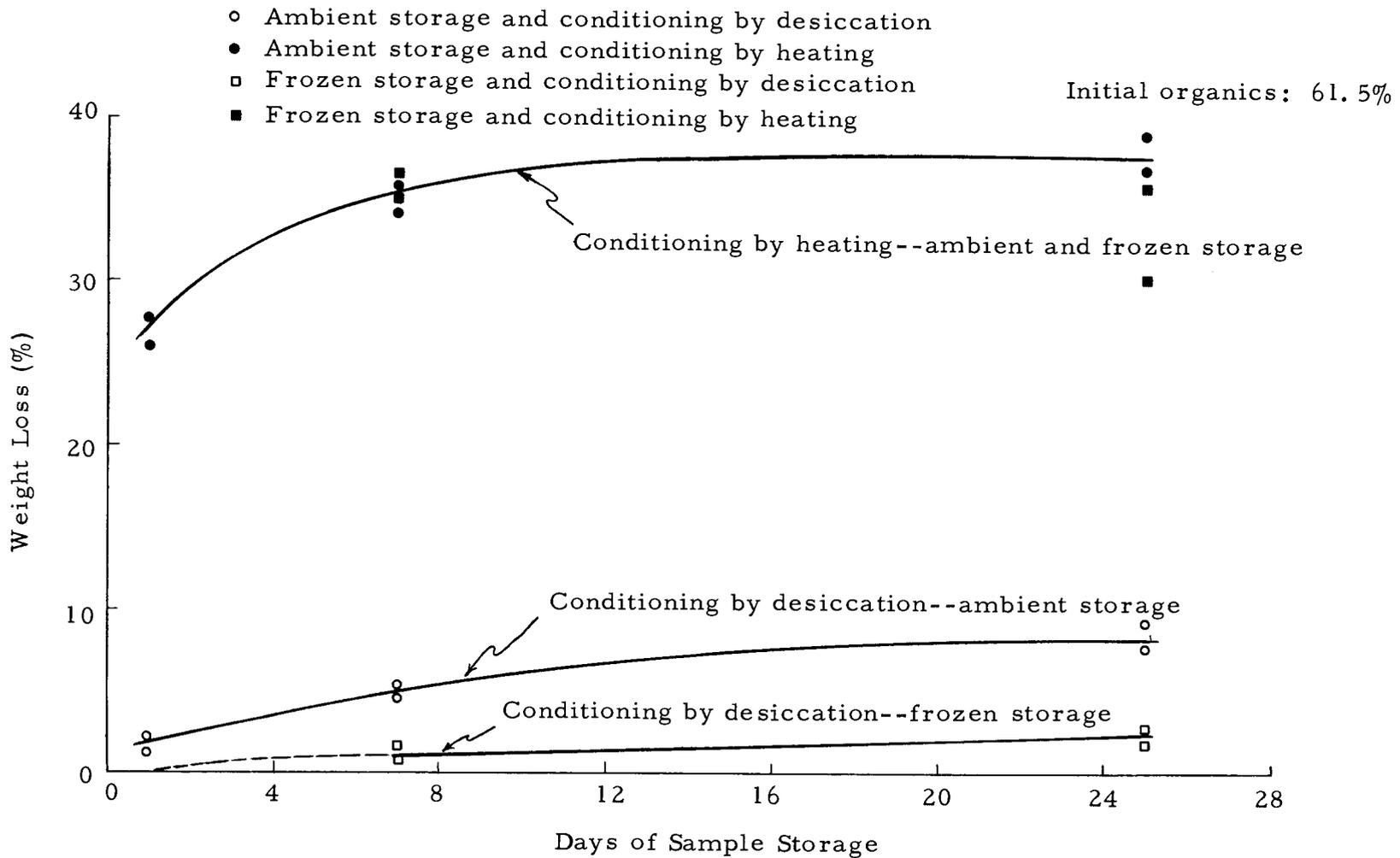


Figure 10. Relationships between storage conditions, storage time, and sample conditioning for the Corvallis dump samples.

6. Time slightly influenced weight losses for all procedures followed. Effects were least for samples frozen and conditioned by desiccation (2.2% weight loss over the 25 day storage time). Time effects were greatest for samples stored under ambient conditions and conditioned by heating (10% weight loss over the 25 day storage time).

7. A significant portion of weight losses was due to the loss of organic matter. Table 1 indicates the reduction in organic content as weight losses increased.

Table 1. Reduction in organic content due to various pre-weighing procedures for the Corvallis dump samples.

Storage Time (days)	Storage Conditions	Sample Conditioning	Average Weight Loss (%)	Average Organics (%)
25	Freeze	Desiccated	2.2	61.3
25	Ambient	Desiccated	8.0	55.1
25	Ambient	Heated	37.5	39.0

8. Storage by freezing and conditioning by desiccation caused the least loss of organic matter (sample contained 61.3% organics).

9. Ambient storage and conditioning by heating caused the greatest loss of organics (sample contained 39.0% organics).

10. The weight loss caused by ambient storage was essentially all due to loss of organic matter (Figure 11).

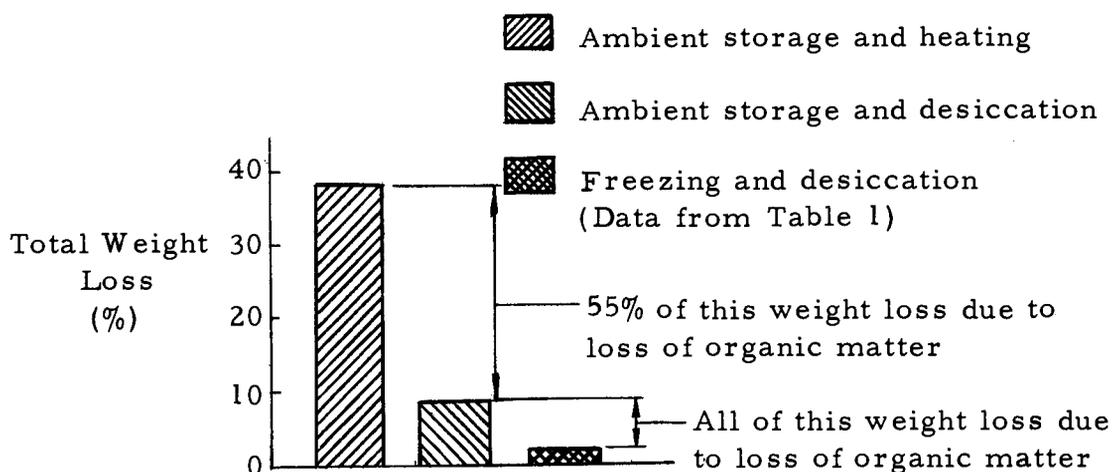


Figure 11. Percent of total weight loss due to the loss of organic matter.

11. The amount of organic matter lost under ambient storage increased with storage time (most losses occurred during the first few days of storage).

12. The weight loss caused by heating versus desiccation conditioning was approximately 55% due to loss of organic matter (Figure 11).

13. Data from a particle count indicated a log normal size distribution with a mean particle size of 0.41 microns and a geometric standard deviation of 2.0 (Appendix Figure 1). Particles appeared to be composed of burnt and partially burnt material as shown by the Polaroid photograph (Appendix Figure 1).

Bureau of Mines Samples

1. Storage conditions and method of sample conditioning were the only variables that significantly affected the final particulate weight (Figure 12).

2. Samples conditioned by heating had the largest weight loss (average 3.2%) regardless of storage conditions.

3. Ambient storage and conditioning by desiccation caused a 1% average weight loss while frozen storage and conditioning by desiccation caused no detectable weight loss over the 25 day storage period.

4. In general, all weight losses were due to the loss of organic matter. Table 2 indicates the reduction in organic content as weight losses increased.

Table 2. Reduction in organic content due to various pre-weighing procedures for the Bureau of Mines samples.

Storage Time (days)	Storage Conditions	Sample Conditioning	Average Weight Loss (%)	Average Organics (%)
25	Freeze	Desiccated	0.0	9.3
25	Ambient	Desiccated	0.7	8.9
25	Ambient	Heated	3.7	4.5

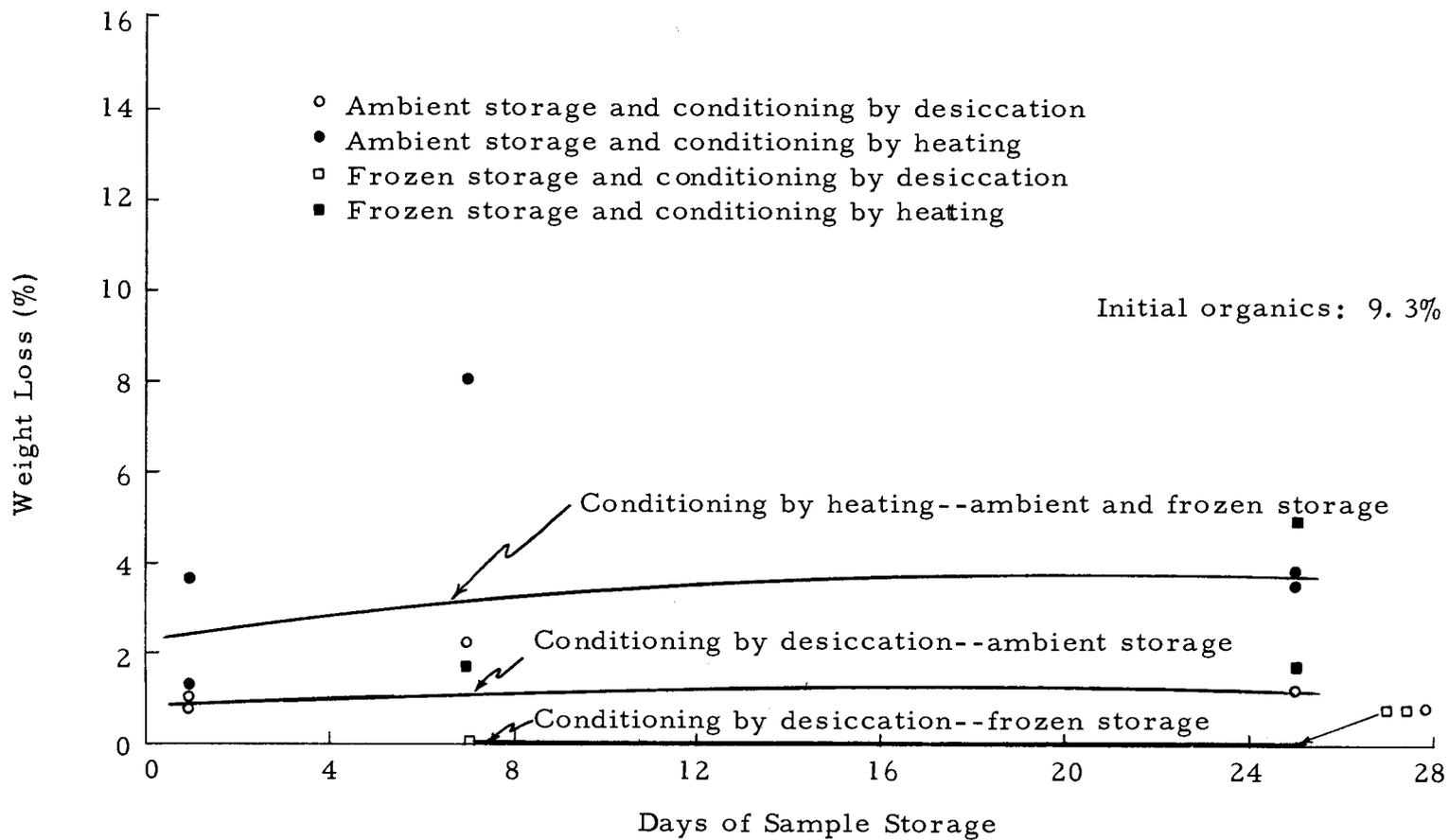


Figure 12. Relationships between storage conditions, storage time, and sample conditioning for the Bureau of Mines samples.

5. Storage by freezing and conditioning by desiccation caused the least loss of organic matter (sample contained 9.3% organics).

6. Ambient storage and conditioning by heating caused the greatest loss of organic matter (sample contained 4.5% organics).

7. Data from a particle count indicated a size distribution close to log normal with a mean particle size of 0.80 microns and a geometric standard deviation of 1.74 (Appendix Figure 2). Particles appeared to be composed of burnt and unburnt matter as shown by the Polaroid photograph (Appendix Figure 2).

Hanna Nickel Samples

1. Method of sample conditioning was the only variable that significantly affected the final particulate weight (Figure 13).

2. An average 1.5% weight loss was caused by conditioning by heating for both ambient and frozen stored samples. All other procedures caused no detectable weight loss.

3. No detectable change in organic matter occurred. The average organic content was 1.8%.

4. Data from a particle count indicated a log normal size distribution with a mean particle size of 0.47 microns and a geometric standard deviation of 1.5 (Appendix Figure 3). Particles appeared to be mostly crystalline and spherical in nature as shown by the Polaroid photograph (Appendix Figure 3).

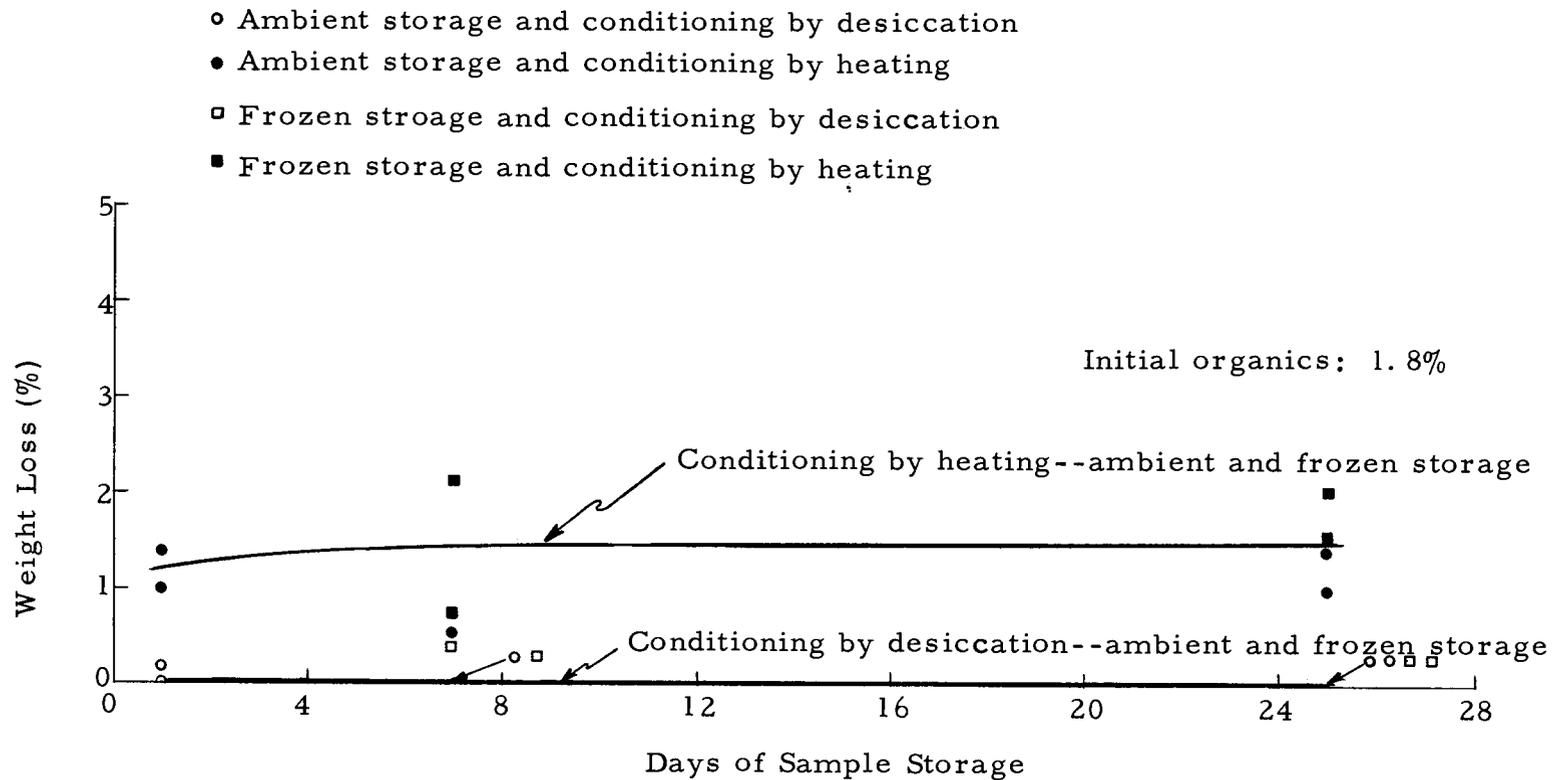


Figure 13. Relationships between storage conditions, storage time, and sample conditioning for the Hanna Nickel samples.

DISCUSSION OF RESULTS

Corvallis Dump Samples

1. The extremely good correlation for both weight loss and extraction data was due, at least in part, to the magnitude of the material losses. Slight experimental errors could have been introduced by (1) loss of material in sample handling, and (2) differences in sample composition due to variations in the smoke sampled.

The data definitely demonstrated that for samples containing a high percentage of certain organic matter significant errors in reported weights could result by using different pre-weighing procedures.

2. Sample conditioning by heating caused the greatest loss of weight regardless of storage time or storage conditions. Although most sample conditioning procedures prescribe heating to remove moisture absorbed or adsorbed after sample collection, it was shown that approximately half the weight loss caused by heating was due to a loss of organic matter. The other half probably was moisture. This moisture, however, had to be mostly inherent moisture present in the pollutant when collected; as the procedure followed allowed little if any chance of moisture pick-up after sample collection. Sawyer (5) substantiates this premise by stating that certain organic

matter will release water of composition when heated.

Thus, approximately 25% of the total pollutant collected and stored for one day was lost due to conditioning by heating instead of desiccation. About half of this loss was organic matter (20% of initial organic matter present), and half was inherent moisture.

3. An additional 6-10% weight loss (all organics) was caused by storing samples 25 days under ambient conditions instead of frozen storage. Most of this loss, however, came within the first seven days of storage.

4. The combination of frozen storage and conditioning by desiccation was the most desirable for samples stored any reasonable length of time. Total weight reduction after 25 days storage was only 2.1%. For short term storage (one day or less) ambient conditions were acceptable if conditioning by desiccation was used.

Bureau of Mines Samples

1. Weight losses for samples from this source were somewhat less than those from the Corvallis dump. The general trends, however, were very similar. Conditioning by heating caused the largest weight loss and again was independent of storage conditions. The organic content being small made extraction data quite subject to experimental error. All weight losses, however, appeared to be caused by loss of organic matter.

2. Weight loss data for samples stored seven days did not follow too closely to data from samples stored 1 and 25 days. Weight losses were about twice those expected. An examination of extraction data shows, however, that the organic content was about twice the value of samples stored 1 and 25 days. Since there seemed to be a correlation between weight loss and organic matter, the higher weight losses would be expected for samples stored seven days.

Hanna Nickel Samples

1. As expected, weight losses for samples from this source were small. This was probably due in part to the small percentage of organic matter present. In addition, inherent moisture content was low due to the hot and dry conditions at which the sample was collected (stack temperature was 300° F).

2. Conditioning by heating, again, caused the largest (and only) recorded weight loss. Other procedures may have caused slight changes of weight; but due to the limited sensitivity of the analytic balance, weight losses of about 1/2% were all that could be accurately detected.

Composite Results

1. Data from the three sources of pollutant samples provided significant information as to the trends and effects caused by the type

of sample, method of sample conditioning, storage conditions, and storage time. The weight loss curves developed for each pre-weighing procedure and pollutant were very similar in appearance except for the fact that as the percent of organic content increased so did the magnitude of the weight losses. More specifically, the magnitude of these losses appeared to be approximately proportional to the percent of organic matter initially present in the pollutant. For instance, the Corvallis dump samples initially contained about seven times more organic matter than those from the Bureau of Mines. Maximum weight losses were about nine times higher. The Corvallis dump samples contained about 30 times more organic matter than those from the Hanna Nickel Mine. Maximum weight losses, here, were about 25 times higher. Thus, for a pollutant containing twice as much organic matter as another, it could be expected to experience about twice the weight loss caused by a certain pre-weighing procedure.

2. For a particular type pollutant the method of sample conditioning had the greatest effect on weight loss. Heating samples caused weight losses which were, for the most part, due to the loss of organic matter and inherent moisture. The magnitude of the weight losses was, also, related to the percent of organic matter initially present in the pollutant. A very approximate empirical relationship based on data from the three sources of pollutants is

stated below.

Weight loss due to heating (% of total weight)

$$= 0.45 \times (\% \text{ organics})$$

3. Storage conditions had no measurable effect on weight losses for samples conditioned by heating. Evidently all of the material (organic) that was retained by freezing was lost in the heating process. For samples conditioned by desiccation, freezing proved to be a distinct advantage over ambient storage; as much volatile organic matter was retained. This advantage, however, decreased as the initial organic content decreased. For instance, for the Hanna Nickel samples (1.8% organics) there was no measurable difference in weight loss due to ambient or frozen storage.

4. Storage time had little effect on frozen samples. Weight losses, however, increased with time for samples stored under ambient conditions. Data indicated that for ambient storage most of the weight loss occurred in the first few days of storage and was primarily due to the loss of organic matter. As the initial amount of organic matter decreased so did the effects of ambient storage time on sample weight.

5. The best procedure to follow in order to minimize weight loss, in all cases, proved to be frozen storage and conditioning by desiccation. Frozen storage retained organic matter which normally

would volatilize under ambient storage. Conditioning by desiccation retained organic matter and inherent moisture which would normally be lost from conditioning by heating.

6. Conditioning by desiccation may be impractical in cases where it is known that the pollutant has picked up a large amount of extraneous moisture (for instance samples collected from the outlet of a wet scrubber). In this case if heating is used as a method of conditioning to quickly remove the moisture, the data from this study can be used to provide an approximate answer to the question of how much error has been introduced by using this pre-weighing procedure.

If organic content is low and storage time short, however, no appreciable weight loss should result from ambient storage and conditioning by heating.

CONCLUSIONS

1. The type of sample, storage conditions, storage time, and method of sample conditioning significantly affected the reported weight of an air pollutant sample.

2. The magnitude of weight losses caused by a specific pre-weighing procedure was approximately proportional to the percent of organic matter initially present in the pollutant.

3. Storage time caused significant weight losses only for samples stored under ambient conditions. Largest weight losses occurred during the first few days of storage. These losses were due to the volatilization of organic matter.

4. Sample conditioning by heating caused the greatest loss of material (both organic matter and inherent moisture). An approximate empirical equation for estimating these losses is:

$$\begin{aligned} \text{Weight loss due to heating (\% of total weight)} \\ = 0.45 \times (\% \text{ organics}) \end{aligned}$$

5. Frozen storage provided no advantage for samples conditioned by heating.

6. Frozen storage and conditioning by desiccation proved to be the best procedure to follow for minimization of weight losses.

7. No pre-weighing procedure caused appreciable weight losses for samples low in organic content and stored for a short period of time.

RECOMMENDATIONS

1. It is hoped that by this study persons in the field of air pollution will become more aware of the errors introduced by various pre-weighing methods and will take steps in order to minimize or eliminate them.

2. It is recommended that storage by freezing and conditioning by desiccation be used whenever possible to minimize errors affecting the reported weights of air pollutants collected by filtration.

3. It is hoped that further investigations take place in regards to effects caused by various pre-weighing procedures on reported weights of air pollutants and other methods used in air pollutant analysis.

4. Finally, it is hoped that a standard methods text for air pollutant analysis will be published which will contain the accepted methods of analysis and their variability. Only when this is done and the recommended methods adhered to will it be possible to fairly compare results from various air pollution surveys.

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APPENDICES

Appendix Table 1. Tabulation of data (Corvallis dump).

Filter No.	Storage* Conditions	Storage Time (Days)	Sample [¶] Conditioning	Original Sample Weight (g)	Final Sample Weight (g)	Weight Loss (%)	Organic Matter Weight (g)	Organic Matter (%)
1A [§]	A	1	D	0.1372	0.1347	1.8	0.0792	57.6
1B ^σ	A	1	D	0.1463	0.1438	1.7	0.0697	47.6
2A	A	7	D	0.1328	0.1269	4.4		
2B	A	7	D	0.1307	0.1235	5.5		
3A [§]	A	25	D	0.1382	0.1256	9.1	0.0826	59.7
3B ^σ	A	25	D	0.1475	0.1378	7.0	0.0747	50.6
4A	A	1	H	0.1433	0.1032	27.9		
4B	A	1	H	0.1373	0.1018	25.8		
5A	A	7	H	0.1444	0.0980	32.2		
5B	A	7	H	0.1116	0.0749	32.9		
6A [§]	A	25	H	0.1409	0.0897	36.3	0.0565	40.1
6B ^σ	A	25	H	0.1126	0.0689	38.8	0.0428	38.0
7A	F	7	D	0.1381	0.1370	0.8		
7B	F	7	D	0.1407	0.1399	0.6		
8A [§]	F	25	D	0.1358	0.1329	2.1	0.0896	66.0
8B ^σ	F	25	D	0.1305	0.1274	2.4	0.0738	56.6
9A	F	7	H	0.1479	0.0987	33.2		
9B	F	7	H	0.1268	0.0852	32.8		
10A	F	25	H	0.1411	0.0898	36.4		
10B	F	25	H	0.1495	0.1048	29.9		

* F - Freeze, A - Ambient

§ Samples collected at same time

¶ D - Desiccate, H - Heat

σ Samples collected at same time

Appendix Table 2. Tabulation of data (Bureau of Mines).

Filter No.	Storage* Conditions	Storage Time (Days)	Sample [¶] Conditioning	Original Sample Weight (g)	Final Sample Weight (g)	Weight Loss (%)	Organic Matter Weight (g)	Organic Matter (%)
1A ^a	A	1	D	0.0955	0.0947	0.8	0.0095	10.0
1B [§]	A	1	D	0.0401	0.0397	1.0	0.0042	10.5
2A	A	7	D	No Sample--Kiln Shutdown				
2B ^σ	A	7	D	0.1153	0.1126	2.3	0.0322	27.9
3A ^a	A	25	D	0.1278	0.1262	1.3	0.0157	12.3
3B [§]	A	25	D	0.0441	0.0441	0.0	0.0024	5.5
4A	A	1	H	0.0430	0.0423	1.6		
4B	A	1	H	0.0402	0.0387	3.7		
5A	A	7	H	No Sample--Kiln Shutdown				
5B ^σ	A	7	H	0.1411	0.1295	8.1	0.0281	19.9
6A ^a	A	25	H	0.1904	0.1832	3.8	0.0080	4.2
6B [§]	A	25	H	0.0558	0.0538	3.6	0.0026	4.7
7A	F	7	D	No Sample--Kiln Shutdown				
7B ^σ	F	7	D	0.1422	0.1422	0.0	0.0372	26.2
8A ^a	F	25	D	0.1297	0.1297	0.0	0.0120	9.3
8B ^σ	F	25	D	0.0441	0.0441	0.0		
9A	F	7	H	No Sample--Kiln Shutdown				
9B	F	7	H	0.0420	0.0413	1.7		
10A	F	25	H	0.0458	0.0451	1.5		
10B [§]	F	25	H	0.0459	0.0436	5.0		

* F - Freeze, A - Ambient

^a Samples collected at same time

[¶] D - Desiccate, H - Heat

[§] Samples collected at same time

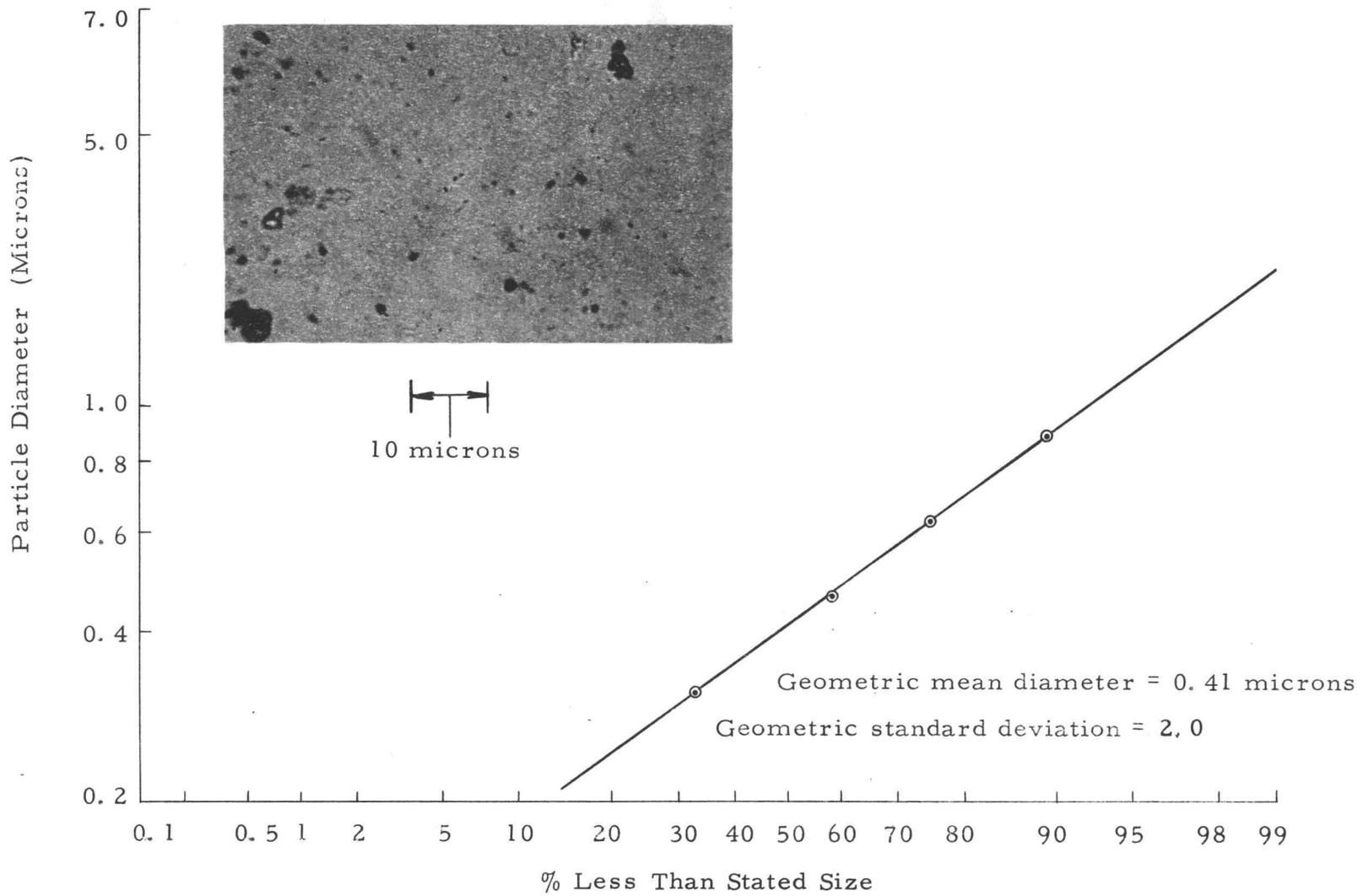
^σ Samples collected at same time

Appendix Table 3. Tabulation of data (Hanna Nickel).

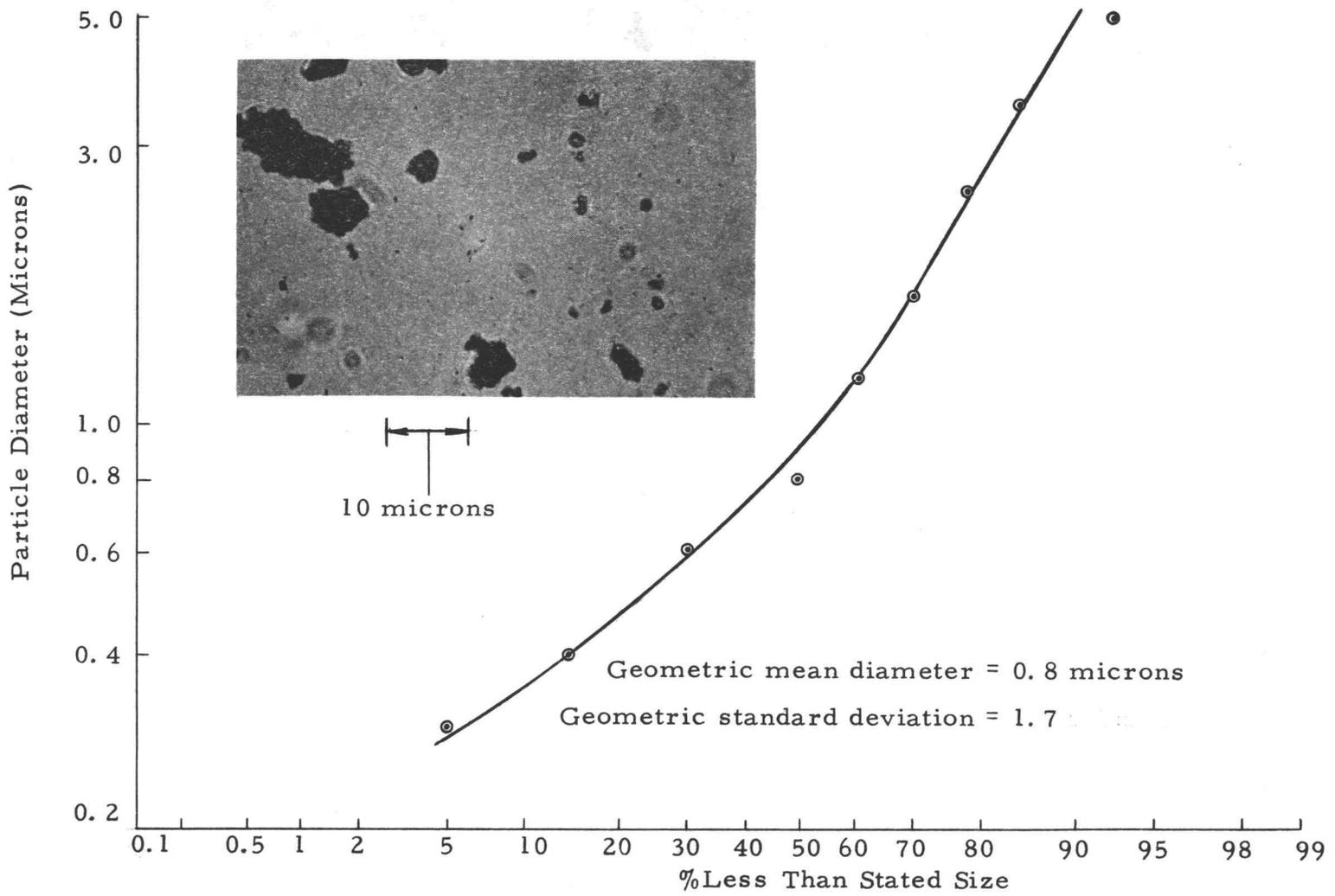
Filter No.	Storage* Conditions	Storage Time (Days)	Sample [¶] Conditioning	Original Sample Weight (g)	Final Sample Weight (g)	Weight Loss (%)	Organic Matter Weight (g)	Organic Matter (%)
1A	A	1	D	0.0370	0.0370	0.0	0.0014	3.8
1B	A	1	D	0.0600	0.0599	0.2	0.0000	0.0
2A	A	7	D	0.0650	0.0650	0.0	0.0016	2.5
2B	A	7	D			Sample Dropped		
3A	A	25	D	0.0181	0.0181	0.0		
3B	A	25	D	0.0393	0.0393	0.0		
4A	A	1	H	0.0587	0.0581	1.0	0.0000	0.0
4B	A	1	H	0.0342	0.0337	1.4	0.0016	4.7
5A	A	7	H	0.0404	0.0402	0.5	0.0003	0.7
5B	A	7	H			Sample Dropped		
6A	A	25	H	0.0259	0.0255	1.5		
6B	A	25	H	0.0467	0.0464	0.6	0.0000	0.0
7A	F	7	D	0.0308	0.0308	0.0	0.0003	1.0
7B	F	7	D	0.0518	0.0516	0.4	0.0010	1.9
8A	F	25	D	0.0267	0.0267	0.0		
8B	F	25	D	0.0335	0.0335	0.0	0.0011	3.3
9A	F	7	H	0.0331	0.0324	2.1	0.0018	5.4
9B	F	7	H	0.0464	0.0461	0.7	0.0000	0.0
10A	F	25	H	0.0403	0.0395	2.0		
10B	F	25	H	0.0361	0.0356	1.4		

* F - Freeze, A - Ambient

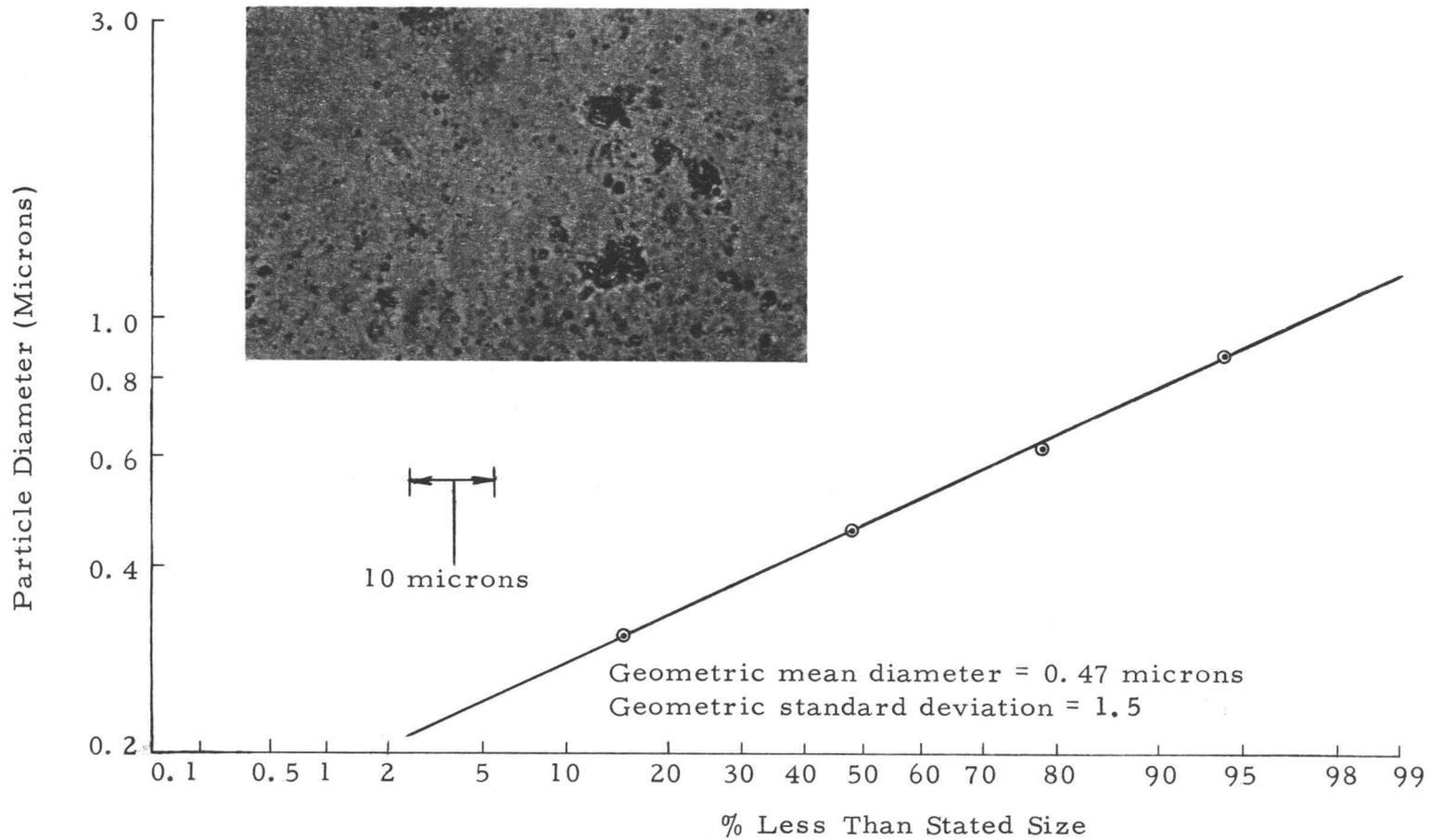
¶ D - Desiccate, H - Heat



Appendix Figure 1. Typical Corvallis dump sample size distribution and Polaroid picture of particulate, 1000x.



Appendix Figure 2. Typical Bureau of Mines sample size distribution and Polaroid picture of particulate, 1000x.



Appendix Figure 3. Typical Hanna Nickel sample size distribution and Polaroid picture of particulate, 1000x.