**The industrial environment, its evaluation and control : Syllabus for short courses for industrial hygiene engineers and chemists presented at Occupational Health Field Headquarters, Cincinnati, Ohio.**


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# the Industrial Environment
# ...its Evaluation and Control

## Syllabus

for

## Short Courses for Industrial Hygiene Engineers and Chemists

*Presented at*

**Occupational Health Field Headquarters**
**Cincinnati, Ohio**

U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service

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# FOREWORD

In 1953 the Occupational Health Program of the U. S. Public Health Service offered a two-weeks training course for industrial hygiene engineers and chemists. The primary aim of the course was to provide assistance to State and local agencies which were unable to obtain experienced personnel to fill staff vacancies. The response to this course was surprising, but most satisfying, demonstrating the need for such training. The course has been given at least once each year since 1953, without any evidence of a slackening in demand. It is planned to continue such instruction as long as the need is demonstrated.

Certain reference materials, lecture outlines and problems, in mimeographed form, have been provided to these classes. This syllabus has now been developed to supply such information in a better organized and more useful form. It is, in this new form, also being made generally available, rather than only to those individuals attending the classes.

This course will not make one an industrial hygiene engineer or chemist. It is intended (1) in the case of newcomers to the field, to provide guidance which will start them in the proper direction towards the achievement of competence, and (2) in the case of more experienced workers, to furnish an opportunity to review basic procedures and techniques, and to compare and exchange experiences and information.

The course is devoted primarily to a consideration of fundamental principles and methods employed in the evaluation and control of the working environment. Some lectures are presented dealing with clinical, toxicological and physiological aspects of occupational health hazards, but such material has been intentionally omitted from this syllabus. Such subject matter would need to be given in considerable detail in order to be useful for reference purposes. Many excellent reference books, including those in the section on "Selected List of General Reference Sources," are available on these topics on health effects.

A number of individuals of the Occupational Health Program assisted in the preparation of this syllabus as indicated on the first page of each section. Mrs. Ruth Gregory typed practically all of the material in its final form.

The planning and editing of the over-all project was done by Andrew D. Hosey, Robert G. Keenan and Charles D. Yaffe.

Cincinnati, Ohio

May, 1958

# Introduction

# INTRODUCTION

## Development and Practice of Industrial Hygiene

One of the many definitions of industrial hygiene is: "The science and art of recognizing, evaluating and controlling occupational health hazards." The purpose of this volume is to present information in the form of lectures and laboratory exercises which will assist the individual: (1) to recognize potential health hazards in industrial operations; (2) to evaluate them; and (3) to control these hazards.

### Background

A brief review of the development of industrial hygiene practices seems appropriate as an introduction to this syllabus.

The earliest men encountered hazards in the process of providing food and shelter for themselves and their families. Methods of earning a living have changed with the years, and the hazards have changed with the methods. Recognition of relationships between occupation and disease occurred at least two thousand years ago, and mention of these relationships is found in ancient writings. Prior to the Twentieth Century, the outstanding publication in this field undoubtedly was a book "De Morbis Artificum Diatriba" (The Diseases of Workmen) published in 1700 by an Italian physician named Bernardino Ramazzini.

### Legislation and Compensation

The mechanization which accompanied the industrial revolution brought new hazards and intensified old ones. More importantly, the number of industrial workers increased markedly in this period. The resultant increase in injuries eventually brought legislation, such as the Factory Acts of Great Britain. The Health and Morals of Apprentices Act was passed in 1802, the Child Labor Laws in 1833, and in 1901 a law concerning the protection of the health of workers was passed.

Prior to the adoption of the compensation principle workers injured on the job had to sue their employers to collect damages, and could not collect if it could be proved that the injury was due to the ordinary risks of the job, or to the negligence of a fellow worker, or if the worker by his own negligence contributed to the injury.

Switzerland in 1881, and Germany in 1884, led the way in the passage of compensation laws. Within 25 years every major European country had such legislation. The United States lagged in this respect. In 1908 the Federal government passed a compensation act for certain civil employees and in 1911 the first State compensation laws were passed. Others quickly followed, 42 having such legislation by 1920, and all States by 1948.

The main purpose of workmen's compensation is to provide prompt payment for injured workers or their survivors, regardless of the circumstances of the accident. Industry also receives protection under these

laws through a limitation of the total amount of compensation which can be collected by the worker. Each State law is different, however, so the provisions vary considerably from one State to another.

The original compensation laws were for accidental injuries. In all but two States there is now also some coverage for occupational diseases. The range of coverage extends from compensation for only one disease, silicosis, to the inclusion of all disease arising out of employment.

Growth of State Programs

Industrial hygiene activities by official agencies, insurance companies and industrial plants were stimulated considerably as occupational diseases became compensable. Thus investigations into health conditions in industry were begun by State and Federal agencies around 1913 and 1914. During the next twenty years, however, scarcely half-dozen industrial hygiene divisions were put into operation in State agencies. The passage of the Social Security Act in 1935 provided funds with which to establish State programs. Twenty-three programs were started between 1936 and 1939. World War II served as a further stimulus, 31 additional State, local and territorial units being formed during the 1940-1946 period. The importance of maintaining the health of industrial manpower received recognition and resulted in acceptance of industrial hygiene as a public health function. Today there are occupational health programs in 41 States, Hawaii, Puerto Rico, the District of Columbia and at least 23 local health departments. In New York, Massachusetts and Illinois the programs are located in labor departments. The other State programs are in the health departments.

Growth of Industrial Programs

Although a number of plants had medical departments to care for injuries, there were virtually no company operated industrial hygiene programs as late as 1935. As the value of preventive services became recognized a number of firms began to employ personnel to work in this field. The steady rate of growth of membership in the American Industrial Hygiene Association from 160 in 1940 to over 1000 in 1957 reflects the development of growth of such activities by industry. Employment of industrial hygiene personnel by State and local official agencies has also increased during this period, but not at a comparable rate.

Federal Agencies

The U. S. Public Health Service has had a program in industrial hygiene or occupational health since about 1913. The U. S. Bureau of Mines has also maintained an active interest in health problems associated with mining operations. During World War II the Army, Navy, Air Force and Atomic Energy Commission also embarked upon programs to protect the health of their personnel having occupational exposures to hazards. These programs have continued and developed.

Magnitude of Problem

Despite the remarkable growth of industrial hygiene in one generation, most of our industrial plants and their workers are not receiving services in

tnis field except in token fashion. If we consider only manufacturing plants, which employed about 16,000,000 workers in 1952 according to Department of Commerce figures, it is found that approximately 2150 of these plants employ 1000 or more workers, and 3000 others employ between 500 and 999. These 5150 plants employ a total of 7,500,000 workers or almost half of this portion of the industrial population.

The number of industrial hygiene programs in industry has not been established. It is probable that the total number of companies conducting such activities does not exceed 500. It is apparent, therefore, that services, except for those supplied by official agencies, are still lacking in most of the larger establishments. The situation is far worse with respect to workers in smaller factories where about 8,500,000 individuals are distributed among over 260,000 establishments having less than 500 employees each. Very few of these plants have their own industrial hygiene personnel. That official agencies can reach only a small proportion of these becomes apparent when we realize that State and local industrial hygiene personnel number less than 400, of which fewer than 300 are engineers, chemists or in related categories.

The figures just presented have included only workers in manufacturing establishments. About 50,000,000 additional people in this country are employed in other industry categories, such as transportation, agriculture and trade. Many of these workers are subjected to occupational hazards as serious as those in manufacturing. The need for more industrial hygienists is quite evident, particularly when it is realized that new materials and new processes are creating new problems more rapidly than old ones are being solved.

Nature of Programs

The American Industrial Hygiene Association Quarterly, Vol. 17, No. 2, June 1956, contains the results of a survey of industrial hygiene units in industry. The report of this survey, conducted by the Development Committee of the Association, provides detailed information about the industrial hygiene administrative structure, facilities and activities in 57 plants. Virtually all of them appraise the working environment for air contaminants. Evaluations of heat and humidity, noise, radiant heat, illumination, ionizing radiation and ultraviolet radiation are also regular functions of most of these units. More than half of them also reported having to deal with air-borne waste, analyze biological specimens, make clinical tests, and test efficiency of air cleaners. They also make detailed recommendations for control of unsatisfactory conditions, including process ventilation, general ventilation and make-up air. Approximately 60% of the units were under the administrative supervision of the medical director and about 10% under directors of personnel or industrial relations, while the others reported directly to various other officials including the company president.

Approximately 20 insurance companies employ industrial hygiene personnel who advise policy holders about problems of the working environment which are potentially hazardous to health.

Other non-governmental workers in occupational health include a number associated with educational institutions. Activities of these individuals range from research and teaching to consultation. They may also serve as industrial hygienists for the institutions which employ them.

Industrial hygiene programs of official agencies embrace a wide range of functions. A typical State agency offers the following services:

1. Engineering, laboratory, medical and nursing consultation on industrial health problems

2. Investigation of occupational disease cases reported by physicians

3. Assistance in planning or expansion of plant medical and nursing services

4. Surveys and detailed studies of work places for conditions or processes which may be harmful to health

5. Maintenance of a laboratory for analysis of environmental and biological samples and development of methods

6. Reports of investigations made including recommendations for the control of hazards found

7. Follow-up services to determine the effectiveness of controls installed

8. Examination of plans prepared by industry for the control of hazards

9. Educational and informational material on industrial health subjects.

The control of hazardous conditions in industry is fundamentally a responsibility of the States. Direct service to individual plants by official agencies, therefore, is provided by the State or local government. Federal agencies ordinarily do not furnish direct services except to Federal establishments. Their work, as exemplified by the Public Health Service, is primarily research and investigative, dealing with problems of industry-wide or national interest, rather than of an individual plant.

The Occupational Health Program conducts the industrial hygiene work of the U. S. Public Health Service. The major functions of the Occupational Health Program are as follows:

1. Studies health hazards on the site and in the laboratory

2. Provides technical and consultative assistance to State and local health departments

3. Encourages development of preventive health services for employees

4. Publishes reports on occupational health problems

5. Provides specialized training of State, local and industrial health personnel

6. Cooperates with other governmental agencies concerned with or interested in the health, safety, and welfare of workers.

The administrative offices of the Occupational Health Program are in Washington, D. C. Its technical services, including research, consultation and training are provided by the staff at the Occupational Health Field Headquarters in Cincinnati and its Field Station in Salt Lake City.

# SELECTED LIST OF GENERAL REFERENCE SOURCES*

## Introduction

The following is a list of the scientific periodicals and reference texts used by the industrial hygienist. The list of literature sources presented is necessarily selective but is intended to include those journals and texts of greatest usefulness to the engineers and chemists working in this field.

It is essential that the value of the several abstracting journals be recognized. The Industrial Hygiene Digest is an excellent source of current information on toxicologic, pharmacologic, dermatologic, engineering and analytic subjects. Chemical Abstracts and the abstract section of the Analyst are indispensable to the chemist who is ever in need of new methods or special modifications of existing procedures to meet new problems successfully. Public Health Engineering Abstracts has sections devoted to occupational health and air pollution topics of importance to both engineers and chemists.

In the group of journals which publish full technical papers, Analytical Chemistry, the Analyst, and the American Industrial Hygiene Association Quarterly are indispensable to the chemist. Noise Control, Journal of the Air Pollution Control Association, Illuminating Engineering, and Air Conditioning, Heating and Ventilating will be of considerable use to the engineers in addition to those journals mentioned above.

The following reference sources are classified, for the sake of convenience, into two general groups, namely: (1) journals and other periodicals; and (2) textbooks and other references. The latter group is further subdivided into a section of handbooks and other sections of particular interest to the different professional disciplines, i.e., chemistry, engineering, medicine and toxicology. All references are listed alphabetically according to title to provide a more useful bibliography.

## Journals and Other Periodicals

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A.M.A. Archives of Industrial Health (Vol. 11, 1955 to present); A.M.A. Archives of Industrial Hygiene and Occupational Medicine (Vol. 1, 1950 - Vol. 10, 1954); American Medical Association, Chicago, Illinois. Journal of Industrial Hygiene and Toxicology (Vol. 20, 1938 - Vol. 31, 1949); and Journal of Industrial Hygiene (Vol. 1, 1919 - Vol. 19, 1937).

* Assembled by Robert G. Keenan and Andrew D. Hosey

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# Sampling Analysis

# GENERAL PRINCIPLES IN EVALUATING THE OCCUPATIONAL ENVIRONMENT

## Introduction

The purpose of this discussion is to present certain general principles to be employed in the conduct of industrial hygiene surveys and the collection of air samples. Little information is available in the literature on this subject; consequently, most industrial hygienists had to acquire knowledge in this field by practical experience. It is hoped that the following discussion will materially assist those engineers and chemists who have not had a great deal of experience in the field of occupational health.

## Purposes for Sampling

There are at least four reasons for industrial hygienists employed by industry to conduct environmental surveys and studies: (1) to determine levels of exposure among workmen to various atmospheric contaminants and physical agents; (2) to determine the effectiveness of control measures, such as local exhaust ventilation; (3) to investigate complaints; and (4) for research purposes, for example, to determine the chemical composition and physical characteristics of dusts, fumes, vapors, gases and mists.

In addition to the reasons cited above, industrial hygienists employed by official agencies may conduct surveys for the following reasons: (1) to determine compliance with regulations; (2) to check control measures installed as a result of recommendations; (3) to investigate health problems on an industry-wide basis, such as uranium, phosphate and diatomaceous earth operations; and (4) for consultation with plant management or labor unions concerning specific health problems.

## Preliminary Survey

The first step in evaluating the occupational environment is to conduct a preliminary survey. Ordinarily much time and effort can be saved by following this procedure because an experienced person, familiar with many types of industrial operations and the use of chemical and physical agents, can by visual inspection and the use of his perceptive senses, eliminate those operations which are clearly non-hazardous.

The preliminary survey should include areas such as: (1) general sanitation, (2) raw materials, products and by-products, (3) sources of air contaminants, (4) physical agents, and (5) control measures in use. The importance of taking adequate notes during a survey or study cannot be overemphasized.

General sanitation. One of the best guides on this subject is the ASA Standard Minimum Requirements for Sanitation in Places of Employment.(1) It contains definitions, general requirements on housekeeping, waste disposal, rodent, insect and vermin control, and covers specific topics on ventilation, water supply, toilet and washing facilities and many others. All of these

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items should be checked to determine if the requirements are met. Most State health departments have adopted this ASA standard, while some States have more rigid requirements.

Raw materials, products and by-products. The variety of substances capable of producing occupational diseases increases steadily. New products are constantly being introduced which require the use of new raw materials or new combinations of older substances. Therefore, it is of utmost importance to obtain a list of all chemicals used, and to determine the nature of the products and by-products. This often requires a bit of detective work on the surveyor's part. In most instances the desired information can be obtained from the purchasing agent or laboratory, but in others it means reading labels on drums of solvent or containers of raw material. Ofttimes, labels do not give complete information, thuse requiring further investigation.

After the list is obtained it is then necessary to determine which of the materials are toxic and to what degree so that appropriate recommendations can be made. The ability to evaluate such information is essential.

Sources of air contaminants. During the preliminary survey many potentially hazardous operations can be detected by visual observation. The most dusty operations can be spotted at this time, although this does not necessarily mean that these are the most hazardous. It must be remembered that the dust particles which cannot be seen by the unaided eye are the most hazardous since they are of respirable size. Dust concentrations of very small particles must reach extremely high levels before they are readily visible in the air. The absence of a visible dust cloud does not mean that a dust-free atmosphere exists. Those operations which generate fume, such as occurs in welding, can be spotted visually.

The presence of many vapors and gases can be detected by the sense of smell. For many substances the odor threshold concentration is greater than the generally permissible safe exposure levels. For example, if the odor of carbon tetrachloride vapor is present even in barely perceptible amounts it is generally too much for continuous exposure.

Physical agents. Sources of radiant heat, abnormal temperatures and humidity, excessive noise, improper or inadequate illumination, ultraviolet radiations, x- and gamma rays, and other ionizing radiations, should be noted. Without the aid of special instruments it is possible only to note the presence of these potential hazards. However, one can obtain much valuable information during the preliminary survey by observing the manner in which these are generated, the number of people involved, and control measures in use.

Control measures in use. The preliminary survey would not be complete unless the types of control measures in use and their effectiveness are noted. The controls include local exhaust and general ventilation, respiratory protective devices, protective clothing, shielding from radiant or ultraviolet energy, and other items which are discussed in Section C-1. General guides to effectiveness should include the presence or absence of dust on floors and ledges, holes in ductwork, fans not operating, or the manner in which personal protective measures are treated by the worker.

## Evaluation of Environmental Exposures

In a completely staffed occupational health agency, evaluation of environmental exposures requires a teamwork approach by personnel who represent several professional disciplines. No single professional group can solve the various facets of the many types of problems presented by occupational disease entities. The longer his association in the field, the more convinced the industrial hygienist becomes of this fact.

In conducting field studies there is an overlapping area for engineers and chemists. The engineer ordinarily collects atmospheric samples in the field for subsequent chemical analyses; in many cases, however, the chemist must go into the field to assist with the collection of samples and to advise on the chemical aspects of the particular problem under study.

### General Considerations in Sampling

Duration of Sampling. No matter who collects atmospheric samples for chemical analyses, whether engineer, chemist, or technician, it is essential that the sample contain a sufficient amount of representative material for an accurate analysis. The volume of air to be sampled is based on the following considerations: (1) sensitivity of the analytical procedure; (2) the Threshold Limit Value (TLV) of the particular contaminant; and (3) the estimated air concentration. Thus the volume of air sample needed may vary from a few liters, where the estimated concentration is high, to several cubic meters where low concentrations are expected. Knowing the sensitivity of the analytical procedure, the TLV, and the sampling rate of the particular instrument in use, the chemist can determine the minimum sampling time necessary for an adequate sample.

There is at least one other consideration in determining how long the sample should be collected. In general, the sample should be collected for at least one complete cycle of an operation. For example, the operation of drawing molten zinc from smelter retorts into a ladle and subsequently pouring the metal into pigs requires about 25 minutes. The next metal draw from the same retorts takes place about six hours later. Samples should be collected for about 25 minutes during the first draw and additional samples collected for the same length of time during the second and subsequent draws. On the other hand, in the furnace breakdown operations, certain cycles last for 3 to 5 minutes only. In this case it is best to collect a sample of about 25 minutes duration which includes 5 to 6 cycles. Several samples are collected also in the general vicinity of these operations (general air) at various times during the shifts. These, and the breathing zone samples collected during metal drawing and furnace breakdown operations, are used in calculating weighted exposures.

If the process is continuous, the considerations listed in the first paragraph should be applied.

Number of samples collected. The necessity for collecting representative samples cannot be overemphasized. The evaluation of occupational exposures is based primarily on the concentration of atmospheric contaminants found as a result of air sampling and analysis (or the use of direct

reading instruments). It is of utmost importance that the samples collected are truly representative of the worker's exposure. The question naturally arises, how many samples need be collected? There are no set rules to follow but a decision can be reached after a careful survey of the particular operation and based upon the judgment of the industrial hygienist. The number of samples required depends not only on the nature of the operation, its location and type of ventilation, but also on climatic conditions. For example, plants located in northern States will usually keep doors and windows closed during cold weather, thereby reducing the amount of natural ventilation. In many instances this causes an increase in atmospheric contaminants. In general, therefore, samples are needed during summer and winter months to define the worker's average exposure. During periods of rain, fog or temperature inversion natural ventilation will be reduced, requiring that samples be collected under these conditions also.

If the contaminant is generated at a fairly constant rate, three to five samples collected during different cycles of the operation and under different weather conditions, should provide data for determining a representative exposure. On the other hand, in calculating the weighted daily exposure of a coal mine undercutter, Flinn[2] collected 25 samples for dust counts. Three samples were collected during the miner's trips into and out of the mine; 4 while setting-up the undercutter, repairing and changing teeth; 15 during undercutting and shearing operations; and 3 while loading and moving the undercutter. Other industrial operations may require a fewer or greater number of samples to evaluate the exposure.

The optimal number of samples to be collected in a given plant area or in the vicinity of specific operations is a matter of judgment on the part of the surveyor. He is in the best position to establish what number will be representative of all varied conditions at the time of the survey.

Types of Field Sampling Instruments

Sampling instruments used in the field of industrial hygiene are generally classified according to type as follows: (1) direct reading; (2) those which remove the contaminant from a measured quantity of air; and (3) those which collect a known volume of air. Most of the sampling equipment used by industrial hygienists is found under type 2 but in recent years more direct reading instruments have been made commercially available.

Detailed discussions appear in Section B-2 on those classified as type 2, such as, electrostatic precipitators, impingers and filter media and in Section B-4 on direct reading instruments. Apparatus of type 3 includes various types of evacuated flasks for collecting known volumes of contaminated air for subsequent chemical analysis. This technique is not used as often now as in past years.

Grab versus continuous samples. A discussion on air sampling techniques would not be complete unless this topic is mentioned. It has been the subject of much concern for many years. A grab sample is collected in a relatively short period of time, usually from a fraction of a minute to 3 or 4 minutes. Grab samples may indicate minimum, maximum or average exposures, depending on the number of samples collected, nature of the

operations, and other factors. Continuous samples, whose collection periods may extend from about 5 minutes to hours or days, can provide average exposure data only. However, there is a definite need for both types of samples. Much useful information can be obtained during a preliminary survey, for example, by the use of a Konimeter where a number of dust samples can be obtained to indicate the range of atmospheric dust concentrations. The Bureau of Mines has used small evacuated flasks to sample mine air for methane and other gases for many years.

Since industrial hygienists are normally interested in determining a worker's daily average exposure to specific atmospheric contaminants, continuous sampling techniques are necessary. Many industrial plants use continuous sampling and recording instruments for gases and vapors such as carbon monoxide, sulfur dioxide, chlorinated hydrocarbons and methyl alcohol.

The choice of a particular sampling instrument depends upon a number of factors. Among these are: (1) portability and ease of use; (2) efficiency of the instrument or method; (3) reliability of the equipment under various conditions of field use; (4) type of analysis or information desired; (5) availability; and (6) personal choice based upon past experience and other factors. No single, universal air sampling instrument is available today and it is doubtful if such an instrument will ever be developed. In fact, the present trend is the development of a greater number of specialized instruments, particularly the newer "squeeze-bulb" types.

Sampling and Analyzing Air for Contaminants by Silverman[3] is an excellent reference source on this subject. Procedures are discussed for sampling and analyzing gases, vapors and particulate materials. Included also are tables listing various instruments and apparatus, methods of collection and analysis of samples.

Another useful reference souce for the collection and analysis of atmospheric contaminants is: The Manual of Analytical Methods.[4] This manual contains procedures which have been thoroughly tested by a referee-collaborator system of evaluation.

## Sampling Rates

Isokinetic sampling. In general, samples for particulates should be be collected under isokinetic conditions, that is, at the same rate of flow as the flow of the contaminated air stream. Isokinetic sampling for particulates in a stack or exhaust duct is essential in order to determine accurately the concentration. Most authorities agree, however, that isokinetic sampling is not necessary for gases and vapors, or dusts smaller than 5 to 10 microns. Watson,[5] in discussing the magnitude of errors due to anisokinetic sampling, states that they are due to three factors: (1) the ratio of wind or air-stream velocity to sampling velocity; (2) the angle between wind direction and sampling nozzle; and (3) wall thickness of sampling orifice. For particles of 4 to 10 microns mass median diameter, a $\pm$ 10 percent error will result if $U_o/\bar{U}$ ($\theta = 0$) does not lie outside the range of 0.5 to 2.0. Under isokinetic sampling conditions, a $\pm$ 10 percent error will result if $\theta = 50^{\circ}$. ($U_o$ = air-stream velocity, $\bar{U}$ = mean air velocity at sampling orifice, and $\theta$ = angle between $U_o$ and $\bar{U}$.) This can

best be illustrated by the following example. Assume that a sample of fertilizer dust is collected on a one-inch diameter filter at 0.5 cfm (about 90 lfm) and the general air movement in the plant is 135 lfm. $U_o/\bar{U} = 135/90 = 1.5$. If the sampler is pointed into the direction of air flow ($\theta = 0$), the sampling error would be ± 10 percent under these conditions. On the other hand, if the sampling rate were increased to 135 lfm the sampler could be pointed 50° away from the direction of the general air currents, resulting in a sampling error of ± 10 percent.

Using filter media. La Torre and Silverman,(6) Fitzgerald and Detwiler,(7,8) and others have investigated the collection efficiency of various filter media at different air sampling rates. Whatman #52 was checked for lead fume in the size range of 0.01 to 0.05 micron at air flows of 5, 10, and 17.5 linear feet per minute (lfm). Collection efficiency was found to be 77.6%, 74.2% and 78.1% respectively.(6) These same investigators found that Whatman #42 was 80.7% efficient for lead fume at 10 lfm (one sample).

The following table was prepared from the results of Fitzgerald and Detwiler's earlier studies.(7)

Table 1. - Collection efficiency of various filter media for duraluminum particles at specified face velocity.

Optimum Operating Face Velocity for Each Filter Paper

| Paper Designation | Optimum Operating Face Velocity (ft/min) | Collection* Efficiency Spread in Percent |
|---|---|---|
| W-40 | 80 | 98.2-99.6 |
| W-41 | 200 | 97.6-98.0 |
| CC-6 | 80 | 99.5-99.7 |
| H-70 | 160 | 99.4-99.8 |
| AEC-1 | 40 | 99.5-99.9 |
| MSA glass fiber 1106-B | 200 | 99.2-99.8 |

* Efficiency range for particles 0.18 to 2.1 microns, density 2.7 gm/cm$^3$.

Percent Penetration of 0.18 μ Particles

| | Maximum | | Minimum | |
|---|---|---|---|---|
| Type Filter | % Penetration | Face Velocity in ft/min | % Penetration | Face Velocity in ft/min |
| W-40 | 40.4 | 20 | 0.8 | 400 |
| W-41 | 14.8 | 4 | 1.6 | 100 |
| CC-6 | 3.3 | 500 | 0.3 | 2 |
| H-70 | 3.0 | 20 | 0.6 | 160 |
| AEC-1 | 7.1 | 2 | 0.4 | 40 |
| MSA 1106-B | 2.5 | 80 | 0.8 | 200 |

In our laboratory, Whatman #40 or #41 and membrane filters are generally used for collecting samples of dusts and fumes. Filters of 1-inch effective diameter are used with a sampling rate of about 0.5 cfm or nearly 100 lfm. Table 1 shows that for 0.18 micron particles the collection efficiency of Whatman #41 is 98.4% at 100 lfm and that Whatman #40 is 98.2-99.6% efficient for particles in the size range of 0.18 to 2.1 microns at 80 lfm. The important consideration when using filter media other than the membrane filter is to select a sampling rate to give the highest collection efficiency. This is necessary because collection efficiencies of various filter media vary widely at different rates of air flow. For example, minimum efficiency of Whatman #40 for 0.18 to 2.1 micron particles occurs at 20 lfm while optimum operating conditions appear at 80, 200, 300 and 400 lfm.

In subsequent studies, Fitzgerald and Detwiler[8] found that collection efficiency depended not only on the particle size and collecting velocity, but also on the type of aerosol. They found, for example, that Whatman #40 and #41 were more efficient for collecting solid duraluminum particles than for $KMnO_4$ particles in the size range of 0.005 to 0.1 micron.

In general, the commonly used filter media can be used for collecting samples of dusts and fumes or other aerosols with an efficiency of at least 90% for particles as small as 0.005 micron. At optimum air flows, efficiencies approaching 100% can be obtained with Whatman #40 and #41, Hollingsworth and Voss H-70, AEC-1, MSA 1106-B, and Chemical Corps 6 in the 0.18 to 2.1 micron range, but maximum penetration was found to occur in the size range of 0.01 to 0.02 micron where efficiencies dropped to 93%.

Other sampling instruments. Most of these are designed to operate at specific air flow rates. The Cascade impactor, for example, operates most efficiently at 17 liters per minute; electrostatic precipitators at 3 cfm (some are designed for 2, 1 and 0.5 cfm); Greenburg-Smith impingers at 1 cfm; midget impinger at 0.1 cfm; and fritted glass bubblers at 3 to 10 liters per minute.

## Units of Measurements

An attempt has been made to standardize units of measurement used in the field of industrial hygiene. Air flows are usually measured in liters or cubic feet per minute. The total volume of air sampled should be converted to cubic meters from these rates.

Analytical results are usually reported by the chemist as micrograms or milligrams per sample or as parts of contaminant per million parts of air (ppm). Using the value of the volume of air sampled, these results should then be converted to milligrams per cubic meter ($mg/m^3$), $mg/10m^3$, or ppm.

In atmospheric pollution studies and stack sampling surveys, results are often reported as grains, grams or pounds per cubic foot.

The Threshold Limit Values are given as $mg/m^3$ and ppm for gases and vapors, $mg/m^3$ for dusts, mists and fumes, and mppcf (millions of particles per cubic foot of air) for mineral dusts. In some States the TLV's are expressed as $mg/10m^3$ because an average worker breathes about 10 cubic meters of air per 8-hour day.

A list of conversion factors for the units used in this field appears in Section E-1.

Comparison of results with standards. The next step in evaluating the occupational environment is to compare results of air sample analyses or data provided by direct reading instruments with recommended workroom standards. The American Conference of Governmental Industrial Hygienists' Committee on Threshold Limit Values publishes annually a list of TLV's. This list provides values for the maximum average atmospheric concentration of vapors, gases, mists, fumes and dusts to which workers may be exposed for an 8-hour working day without injury to health. These values should be used as guides only in the control of health hazards because they are based only on conditions under which it is felt that workers may be repeatedly exposed, day after day, without adverse effect on their health. These values are reviewed annually for changes, revisions or additions as further information becomes available.

Standards for temperature and humidity are found in the ASHAE Guide, Belding and Hatch's heat stress index, and chapter 3 of the Navy Manual of Preventive Medicine. Those for noise, illumination, and sanitation are found in standard references. Those for ionizing radiation may be found in various publications of the Atomic Energy Commission and National Bureau of Standards. Many States and local governmental agencies have adopted rules, regulations or codes patterned after the above standards. These should be consulted before reaching a decision regarding the recommendation of protective or control measures.

Interpretation of results. This is the final step in evaluating the environment. A great deal of common sense and sound judgment must be used in interpretating the results of an environmental study. Before an investigator claims that an individual or group of workers is exposed to a hazard injurious to health, he must have the following facts: (1) nature of the substance or physical condition involved, (2) intensity of exposure, (3) duration of exposure, and (4) the susceptibility of the person exposed.

Items (1) and (2) above will have been obtained during the preliminary and/or environmental study. In order to determine the duration of exposure for an individual a detailed job description must be obtained. This information is usually available from the plant personnel office or foremen, but it should be checked by the investigator during the study. From this information and the results of the environmental survey, the weighted daily 8-hour exposure can be calculated. This assumes that a sufficient number of air samples have been analyzed or that readings obtained with direct reading instruments under various plant operating conditions present a true picture of the exposure.

The following example illustrates the method used to calculate a worker's weighted exposure. Assume that a degreaser operator spends four hours per day cleaning parts and the average concentration of trichloroethylene vapor is 250 ppm at this operation (Task A). Also assume that 3 hours each day are spent at odd jobs in that vicinity where the average vapor concentration is 25 ppm (Task B) and the last hour of work each day is spent removing parts from a heat treating furnace (Task C), located in another area of the plant, where a concentration of 200 ppm of carbon monoxide

occurs when the furnace door is open. This worker's weighted daily exposure to trichloroethylene is calculated as follows:

$$\frac{250 \text{ ppm} \times 4 \text{ hrs. (Task A)} + 25 \text{ ppm} \times 3 \text{ hrs. (Task B)}}{8} =$$

134+ ppm, which is below the TLV of 200 ppm. This same worker's exposure to carbon monoxide would be: $\frac{200 \text{ ppm} \times 1 \text{ (Task C)}}{8} = 25$ ppm CO, which is also well below the TLV. It must be borne in mind, however, that even short exposures to high concentrations of many toxic materials can cause systemic injury or even death.

A final word of caution is indicated at this point. Assume, after the study is complete, that the calculated weighted exposure for a group of workmen to lead fume was found to be 0.17 $mg/m^3$ (and this value was based on a number of samples). One would not ordinarily recommend costly local exhaust systems to reduce the exposure to 0.15 $mg/m^3$ (the TLV) or lower. Instead, a return visit should be made to the plant, and if the results of the second study were essentially the same as before, recommendations would be made to observe these workmen clinically at 4 to 6-month intervals for evidence of lead absorption. If blood and urine values were above normal levels, then control measures would be indicated. On the other hand, if weighted exposures were 0.25 to 0.30 $mg/m^3$ during the first study, control measures would have been recommended immediately and without hesitation. In other words, a few milligrams or parts per million below the TLV's does not necessarily mean the environment is safe nor would a dangerous situation exist if the TLV's are exceeded slightly. A great deal of judgment is necessary in determining the exact cut-off point above which control measures should be instituted. It must be emphasized again that the TLV's are designed to serve as guides only; such factors as individual susceptibility and synergism must also be taken into consideration. For instance, there is evidence that alcohol increases an individual's susceptibility to chlorinated hydrocarbons; thereforek if a degreaser operator is known to be an habitual drinker, his TLV for trichloroethylene should be considerably below the recommended value of 200 ppm.

## Summary

The conduct of environmental surveys and studies is but one phase in the over-all picture in determining occupational health hazards. They are valuable only if all environmental factors relating to workers' potential exposures are included. In evaluating workers' exposures to toxic dusts, fumes, gases, vapors and mists, a sufficient number of samples should be collected for the proper duration to enable the calculation of weighted exposures.

Choice of sampling instruments will probably be a compromise between several desirable characteristics. Among these are high efficiency, portability, reliability and availability. Personal preferences, as a result of experience, will also determine the choice of instruments.

Adequate notes taken during environmental studies are a must - one cannot rely upon one's memory after a study is completed to provide the detailed information necessary for the preparation of a report.

Finally, sound judgment should be exercised both during the actual survey and in preparing the report. Experience, conversation with others in the field, and a thorough knowledge of the particular operations studied will aid in developing these qualities so important in the field of occupational health.

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# A REVIEW OF SAMPLING INSTRUMENTS USED FOR THE COLLECTION OF AIR-BORNE PARTICULATES

## Introduction

The sampling and analysis of air-borne dust is not a new problem brought about by the current interest in air pollution. The first review of the literature on the subject was published by Cunningham in 1873. Since that time there have been several other reviews[2-9]; in most cases the instruments described have become obsolete because of the higher efficiency, greater facility of operation, new collection techniques, and other advantages of modern apparatus. Indeed, there are few principles of modern physics that have not been applied to the problem of air sampling in a never-ending search to find an easier solution to a tedious procedure.

In this review only those instruments which are used to collect samples of air-borne particulates for analysis or identification will be discussed. The instruments have been divided into eight general categories according to their physical basis of operation. It is evident that there may be some overlapping of these categories. In air filtration, for example, the processes of impaction and even electrostatic attraction may play an important part. A rather arbitrary distinction has been drawn between impaction and impingement. In the former, the particles are directed against a dry surface, while in the latter, a liquid collecting medium is used.

## Methods Based on Sedimentation

a. The amount of dust which settles on a plate from a dusty atmosphere is not a good measure of the concentration of dust in the air, since the air is constantly in motion and the volume of air associated with the settled dust is not known. The device known as the Green Sedimentation Cell[3] is a modification of an instrument described by Owens[4] and consists of a cylinder of known height (5 cm) and diameter, with springloaded covers which can be closed rapidly to trap a known volume of air. In practice the open cylinder is waved through the air to be sampled to flush it out and the ends are then closed. The cylinder is then placed in a vertical position, free of vibration and temperature changes, and allowed to remain undisturbed for three hours. This is the time required for all particles larger than 0.2 $\mu$ diameter to fall through 5 cm and settle on a microscope cover slip in the bottom of the cell. This cover slip can then be mounted on a slide and the particles counted or sized with an optical microscope. If desired, an electron microscope grid can be substituted for the cover slip and the dust examined with the electron microscope. This technique of dust sampling has many disadvantages. The settling time is long, so few samples can be taken. It is a spot sampling device and the volume of air involved is limited. The small amount of dust collected precludes chemical or weight analysis. Since dust particles are not necessarily perfect spheres, deviations from Stokes Law can be expected. Even

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for spherical particles there can be no hope of collecting any particles of less than 0.2 $\mu$ diameter.

b. As early as 1890, an instrument was devised to trap and make visible the very small particles which were ordinarily below the limit of resolution of the optical microscope. This was the Aitkin Condensation Nuclei Counter[10] which depends on the fact that during the rapid adiabatic expansion of a gas saturated with water vapor, this vapor condenses on the small particles or "nuclei" present causing them to settle rapidly. In operation, the air to be sampled is admitted to a small chamber with walls covered with moistened blotting paper. The volume of this chamber is suddenly increased by means of a small calibrated pump and the condensed droplets of water, each one representing one condensation nucleus, fall on a prepared glass surface where they can be counted with a microscope built into the instrument[11]. Although it has not been reported, this instrument might be quite useful for preparing specimens for electron microscopy. It is, however, limited by the small size of the sample and that fact that all nuclei and not just dust particles are collected.

## Filtration Methods

The collection of samples of air-borne dust by filtration is one of the most common procedures used and a discussion of all the filtering media that have been used would be exhaustive, ranging from woven cloth to Gooch crucibles. The techniques mentioned below are all in current use and have withstood, at least in some respects, the test of time.

a. The use of the Whatman extraction thimble, sometimes filled with loose cotton packing to reduce clogging, has been described many times[12,13]. The thimble is securely fitted into a hole in the top of an airtight box which can be attached to a source of suction. The air enters the box through the thimble and the dust is retained by the thimble with efficiency depending upon the particular grade of thimble and the airflow used. Large samples can be collected suitable for chemical analysis or weight determination, but the evaluation of particle size from such a sample is questionable due to the increase of efficiency as the pores become clogged and the difficulty of removing all sizes of particles from the filter for microscopic study.

b. Discs of chemical grade filter paper mounted in suitable holders have been shown to have high retention efficiencies[14,15,16]. Whatman #41 has been widely used and one-inch diameter sampling area permits airflows of 18 to 20 liters per minute. Indeed, a high airflow (approximately 25 linear feet per minute at the filter face) is necessary for high retention efficiency since only at this high sampling rate do impaction and the electrostatic charge developed on the filter become sufficient to aid in the retention of particles smaller than the pore size of the paper. Silverman and Viles report[16] that hardened papers, such as Whatman #50, allow the quantitative removal of the collected dust by washing.

c. Special pleated filters have been described[17] which use a portable vacuum cleaner type pump and permit sampling rates up to 80 cubic feet per minute. These filters can be made of different materials including glass fibers held together with an organic binder. For critical organic

chemical analysis the binder can be removed prior to sampling by firing the filter in a furnace just below the sintering temperature of the glass.

d. A new development in filtration has been the molecular or Millipore(R) filter[18] which is a thin (150 μ) sheet composed of mixed cellulose esters. It can be manufactured in a variety of pore sizes and resistances and with great uniformity. A significant advantage of this filter is that the penetration into the filter, even by the smallest particles, is very slight (10-20 microns). When a portion of the filter with collected sample on it is placed on a microscope slide and moistened with immersion oil of the same index of refraction as the filter (the filter becomes transparent) the particles can be seen all in one plane, an important factor in microscopy[19]. The filter is readily soluble in organic solvents such as acetone and ethyl acetate and therefore can be used to collect samples for electron microscopy[20]. Because of the uniformity of the membrane it is possible to make accurate and reproducible measurements of light reflected from the collecting surface and thus sometimes eliminate the necessity of a tedious chemical analysis[21]. If chemical analysis is desired, the high purity of the filter is an advantage while its small size and slight affinity for water makes accurate weighing possible.

e. Tubes of volatile or soluble crystals, such as salicylic acid, sugar, naphthalene, etc., have been used as methods of dust collection by drawing the contaminated gases through them[4,22]. The dust is recovered either by volatilization or solution of the crystals of sufficient purity that no residue is left with the dust collected. The efficiency of the filter depends on the size of the crystals, the depth of the bed, and other factors. When soluble crystals are used gases containing appreciable amounts of water cannot be sampled. Dust collected in this manner is adaptable to chemical, weight, or particle size analysis.

f. Alundum thimbles and sintered glass filters are manufactured in a variety of porosities. They have considerably higher resistance to airflow than comparable paper filters but can be used for high temperature sampling[22]. There is little variation in weight due to adsorption of water and hence they can be weighed more accurately than filter paper. In addition, the collected sample may be recovered almost completely by brushing the inside of the thimble or it may be treated chemically and dissolved right in the filter.

## Impactors

When a high velocity, dust-laden stream of air strikes a flat surface at right angles, the sudden change in direction of airflow and the momentum of the dust particles result in the separation of the dust from the air. The air passes on, leaving the particles adhering to the surface. The performance of instruments based on this principle has been studied both experimentally and theoretically, and the parameters involved clearly defined[23,24,25]. Generally speaking, these instruments are efficient only for particles larger than 0.5 μ in diameter since the momentum of the smaller particles is not sufficient to cause them to impinge on the collecting plate, instead they follow the flow lines of the gas stream.

a. The Kotze Konimeter[4,5,7,26] from which the newer Zeiss Konimeter evolved[27], was developed around 1919 and is still extensively used in South Africa and England. It consists of a tapered nozzle through which a stream of outside air is drawn by a small, spring-loaded pump to impinge near the perimeter of a circular glass plate. The plate can be rotated to receive up to 30 samples. The pump, with a capacity of either 2.5 or 5 cc per sample, develops an air velocity of 50 meters per second at the nozzle which is only 1 mm from the collecting plate. The dust is deposited in a circular pattern with the larger particles toward the center so this instrument is of no use for particle size analysis but can be very useful for counting the number of particles in the 2.5 or 5 cc sample. Usually, but not always, the collecting plate is covered with a thin film of petrolatum, glycerine, or other adhesive to trap the particles. The small sample, of course, precludes any chemical analysis or weighing procedure. It has been suggested that the high velocity of the air jet is sufficient to break up many large particles and hence to lead to spuriously high counts[28].

b. The principle of the Owens Jet Dust Counter[29] is essentially the same as the Konimeter but a rectangular nozzle is used instead of circular, a larger hand-operated pump (50 cc), and a moistening chamber in which the air is humidified before being impacted. The rapid expansion through the nozzle lowers the temperature of the humid air and causes moisture to condense on the dust particles causing them to adhere on the collecting plate. The nozzle velocity is much greater than in the Konimeter (200 m/sec.) and because of this particles are fractured. Thus, the spuriously high counts are more prevalent with the Jet Dust Counter. The efficiency of this instrument is discussed by Davies[23].

c. The most modern jet dust counter is the Bausch & Lomb instrument[33] which is, in principle, the same as the Owens instrument. It uses a wider slit placed closer to the collecting plate, a hand pump with a capacity of 0.0001 cubic foot to simplify calculation, and a built-in darkfield microscope with a magnification of 200x.

d. The Cascade Impactor[31] consists of 4 impingement slits of decreasing width connected in series and mounted at decreasing distances above the collecting plates. Thus, the largest particles are collected on the first plate and the finest on the last, and the over-all dust distribution is divided into 4 different size fractions. If the airflow through the instrument is kept constant (18 liters/minute), and the calibration constants determined at each collecting stage for the dust being sampled, further particle size analyses can be accomplished by simple chemical techniques. Thus, the instrument is useful in such instances as animal inhalation studies where repeated particle size analyses are desired on the same dust cloud[7,23,32].

## Impingers

a. The modern Greenburg-Smith Impinger[33,34] is an all-glass apparatus through which air is sampled at high velocity and impinged from a jet onto a glass plate which is immersed in an absorption medium (usually water). The dust particles are momentarily arrested by the impingement process, wetted by the liquid, and thus trapped. Any gases soluble in the absorbent

are also collected. Essentially, the instrument is a glass cylinder about 30 cm tall and 50 mm in diameter. The inlet is a long concentrically located tube extending from the top of the cylinder and ending in a jet only a few millimeters from the flat bottom. The outlet connection is at the top of the cylinder and applying a source of suction at this point draws air down the inlet tube, through the jet, and forces it to impinge against the bottom of the cylinder. The cylinder is filled to a depth of several centimeters with the collecting liquid. The size of the orifice and the air velocity through it are critical factors in the impingement process. In practice, the orifice approaches the size of a critical or limiting orifice and therefore the resistance of this instrument to airflow becomes considerable. The collecting efficiency falls off rapidly for particles smaller than 0.7 micron. The impinger has one great advantage over all other sampling instruments. It has been used as the standard sampling instrument in the United States by the U. S. Public Health Service and others, and therefore valid comparisons can be made with a vast amount of past experience and studies. For this reason, if for no other, the impinger will continue to be used for many years in spite of the development of newer and more efficient instruments.

b. The Midget Impinger is merely a smaller version of the standard model. Its efficiency is quite similar but it requires a smaller pump (0.1 cubic foot per minute) to supply the necessary suction and can be operated by one man with a hand-crank pump for work in mines or other isolated places. Davies[23] compares the efficiency of the impingers with that of other instruments now in use.

## Centrifugal Separators

a. Small scale, high velocity cyclones, models of the large cyclone dust collectors used in industry have been proposed as air sampling instruments[35,36,37]. The cyclone is essentially a metal or plastic cone. The dust-laden airstream enters tangentially at the widest point and spirals down to the vertex where it is removed. The dust particles are thrown out and collected against the side due to centrifugal force and the fact that the radial acceleration is greater than the terminal acceleration of gravity. The efficiency of the instrument is good only for large particles (greater than 5.0 $\mu$), and depends largely on the radius of curvature of the rotating air and the air velocity in the apparatus. Since the radius should be small and the velocity high, the apparatus is kept as small as possible (2-inch diameter)[37].

b. A new application of the basic theory of centrifugal separation is the Spiral Sampler[38]. In this instrument the dust-laden airstream is drawn through a channel of decreasing width which is curved in an Archimedes' spiral. Thus, the radius of curvature is constantly decreased and the air velocity increased. The dust particles are precipitated out on the side in order of their decreasing size. This feature of separating the sample according to size is useful for microscopic analysis. The instrument is nearly 100 percent efficient for collecting particles larger than 0.5 micron.

c. The "Conifuge"[39] is the name given to another new instrument of this type. Basically it is a conical self-pumping centrifuge into which the

dust cloud is drawn by an internal circulation of previously cleaned air. The particles are classified according to their settling velocities and are deposited on a glass slide in a continuously graded sample. The apparatus is driven by an electric motor at a speed of 3000 rpm, and samples air at a rate of 25 cc per minute. The size of particles collected ranges from 30 to 0.5 microns.

## Spray Techniques

The Venturi Scrubber(40) is a device based on the introduction of a water spray by means of a nozzle into a Venturi through which the dust-laden air passes at high velocity. The dust and fume particles are wetted by the water spray and the droplets of water containing the dust particles are collected by means of a cyclone or other device. This then is a method of extending the efficiency of the cyclone separator to much smaller particles. The samples collected by this means are satisfactory for particle size, chemical, or weight analysis.

## Electrostatic Precipitation

The electrostatic precipitator (41,42) used today for air sampling is a modification of the Cottrell Precipitator(43). It consists of a grounded, metal cylinder about 1.5 inches in diameter and 6 inches in length, which serves as the collecting electrode. Located concentrically in the tube is a stiff wire electrode which is maintained at either a positive or negative potential of about 12,000 volts. The small radius of curvature of the wire produces a very intense electric field around it, and this causes a corona discharge between the wire and the collecting cylinder. Dust particles subjected to this discharge become highly charged and in this state are attracted toward the cylinder wall where they are collected. The effect of this electric field is such that even at relatively high airflows (3 cubic feet/minute), an efficiency of nearly 100 percent for all sizes of particles can be expected. Since the air resistance of this instrument is very low, a high capacity but low pressure fan can be used as a source of suction. Many workers have suggested modifications and new designs for both the high voltage power pack and the precipitator head(44,45). Due to its high efficiency(46), and the large sample which can be taken, this instrument is probably the most widely used air sampling instrument in the United States next to the impinger. Although it can readily be adapted to take samples for microscopic examination, both optical and electron, a size separation of particles occurs during collection and the value of the instrument for this purpose is questionable. Bulk samples can be taken in sufficient quantities for chemical analysis and, in addition, the collecting tubes can be weighed accurately before and after sampling.

## Thermal Precipitation

The thermal precipitator, developed in England, is based on the principle that a dust-free area exists around a hot wire(47,48). The air to be sampled is drawn through a narrow slot by means of a water aspirator across which a hot (100°C) nichrome wire is located. The sides of the slot are formed by two microscope cover slips which are kept cool by contact with brass cylinders holding them in place. The dust particles approaching the

wire are forced against the cover slips by the high thermal gradient (4000°C/cm) and collected. The cover slips are easily removed and mounted for microscopic examination. The instrument is highly efficient, especially for particles smaller than 5 μ, and can easily be used to collect specimens for electron microscopy. The low air velocity and gentle deposition probably maintains their air-borne state of agglomeration without change. The slow sampling rate (7-15 cc/min.) and small size of sample taken (approximately 50 cc) limit the use of the instrument. The British have made wide use of this technique and have accumulated much data with it although it has never been used extensively in this country. Recently, American modifications, one being an oscillating thermal precipitator in which the deposit is spread over a wider area, have been placed on the market(47).

## SUMMARY

In this discussion of air sampling instruments there has been no attempt to include the methods of measurement and analysis which must be used to evaluate the samples. A consideration of the techniques of optical and electron microscopy, or the chemical identification of constituents, is a field in itself. It must be remembered, however, that the value of a sampling instrument is greatly dependent on the available methods of quantification. Thus, a sampling instrument with a high collecting efficiency for particles below 0.3 μ is useless if an opticall microscope with a limit of resolution of 0.4 μ(20) is to be used to size the particles.

Also omitted from this discussion have been the automatuc and continuously recording instruments which are beginning to appear. These are based on such measurements as light scattering from the air-borne particles, reflectance of light from a continuous ribbon of filter paper, or the cooling of a heated wire by the impact and volatilization of liquid aerosols. This type of instrument is highly desirable. However, at the present time, their use is not common and the few that are commercially available are expensive, from $1000 to more than $10,000. The field of electronics, upon which they depend, is advancing so rapidly that it is entirely possible for such an instrument to be outmoded between the date of ordering and the date of delivery some months later.

The measurement of radioactivity has not been mentioned as this seems to belong to the separate field of health physics. The electrostatic precipitator, molecular filter and filter papers are all easily adapted to counting with Geiger-Muller tubes or scintillation counters. The same comments regarding retention or collection efficiencies of these instruments for various sizes of particles apply to the retention of radioactive particles.

A number of books and reviews have been written which deal either in whole or in part with the problem of air sampling(4,5,7,49,50,51,52,53,54,55,56,57). Together these form a fairly comprehensive reference source on the subject.

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# GAS AND VAPOR SAMPLING AND ANALYSIS

## Introduction

This lecture deals principally with the methods and instrumentation used for the sampling and analysis of gases and vapors present as atmospheric contaminants in the industrial environment. In the sections dealing with sampling procedures, direct reading instruments are not discussed as there is a separate lecture on this topic. In both the sampling and analysis sections, the emphasis is placed on fundamental techniques rather than on specific methods for individual substances. This approach eliminates unnecessary repetition.

## Properties of Gases and Vapors

The distinction between gases and vapors is somewhat vague. An "elastic fluid" is generally called a gas if its temperature is very far removed from that required for liquefaction; conversely, it is termed a vapor if it is near its temperature of liquefaction. In the field of industrial hygiene, a substance is considered as a gas if this is its normal physical state at room temperature and atmospheric pressure. It is called a vapor if, under the environmental conditions, conversion of the liquid or solid form to the gaseous state results from its vapor pressure effecting its volatilization or sublimation into the atmosphere of the container, the process equipment, or the workroom. Our chief interest in distinguishing between gases and vapors lies in our need to assess the potential occupational hazards associated with the use of specific chemical agents, an assessment which requires a knowledge of the physical and chemical properties of these agents.

## Sampling Methods

The primary objective of this phase of an environmental survey is to obtain representative samples of the atmospheric contaminant in forms suitable for subsequent analysis. A general discussion of the fundamental aspects of plant surveys to provide adequate evaluations of the environment is given in Section A-2.

### Basic Techniques

Two basic methods for the collection of gaseous samples are employed. The first involves the use of a gas collector, such as an evacuated flask or bottle, to obtain a definite volume of air at a known temperature and pressure. The collector is re-sealed immediately to prevent any loss before the sample can be analyzed. The second method involves the passage of a known volume of air through an absorbing or adsorbing medium to remove the desired contaminants from the sampled atmosphere. The absorbing medium, which may be a solvent such as ethyl alcohol or amyl acetate, can be transferred with the collected sample to a reagent type, glass stoppered bottle for transfer to the laboratory.

The selection of one or the other of these techniques depends upon the type and purpose of a particular sample. When the major components of a gas

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or of air are to be determined, a gas collector is used. When a minute quantity (parts per million level) of a contaminant must be estimated, the method of concentrating the substance in an absorber or on an adsorbent must be employed. The collection of instantaneous samples, those taken within a time period extending from a few seconds to one or two minutes, is made to determine the composition of an atmosphere at a specific time and location. Such samples may best be taken with evacuated flasks or with gas or liquid displacement collectors. However, an absorption or adsorption device must usually be employed for collecting continuous samples of a non-uniform atmosphere, for the estimation of the average composition during a given time period.

Method Requirements

Sampling methods for gases or vapors should meet the following requirements:

1. Provide an acceptable efficiency of collection for the substance or substances involved.

2. Maintain this efficiency at a rate of air flow which can provide sufficient sample for the analytical procedure in a reasonable period of time.

3. Retain the collected gas or vapor in a form which is transportable to the analytical site.

4. Yield the sample in a form suitable for the analytical method.

5. Require minimal manipulation in the field.

6. Avoid use of corrosive or otherwise hazardous sampling media whenever possible.

These requirements are considered as practical guides in planning a plant survey and, by their wording, indicate the need of consultation between the engineer and the chemist for optimal results from each survey.

Sampling Instruments

In the application of the two basic sampling methods, numerous sampling devices have been employed. For more complete information on the construction, recommended uses, and efficiencies of specific instruments, the appropriate reference works should be consulted.[1,2] For outline purposes, the following tables contain a limited listing of the devices commonly used for gas and vapor sampling, along with selected typical applications of each.

Table 1. - Gas and vapor collectors with typical applications

| Collector | Size in ml | Gas or Vapor |
|---|---|---|
| Evacuated flask | 250 - 300 ml | Gross components of air or of sewer or mine gases |
| Evacuated flask | 1000 | Carbon monoxide in air |
| Vacuum bottle | 500 - 2500 | Substances not easily trapped by absorbents or adsorbents: total oxides of nitrogen; benzene |
| Gas or liquid displacement collector, glass | 250 - 4000 | Gross components of air or gas mixture |
| Metallic collectors | Ditto, or larger | Non-corrosive gases |

Table 2. - Gas and vapor samplers, with partial listing of applications, sorption media and collection efficiencies

| Sampler | Sorption Medium | Air Flow l/m | Gas or Vapor | Efficiency % |
|---|---|---|---|---|
| U-tube | Silica gel or activated carbon | 5 | Organic solvent vapors | * |
| Fritted glass bubbler | Sodium hydroxide | 5 | Hydrochloric Acid | 95 |
| | Sodium bisulfite | 1-3 | Formaldehyde | ca 100 |
| | Barium hydroxide | 1 | Carbon dioxide | 60-80 |
| | Iodine | 5 | Hydrogen sulfide | 95 |
| Midget fritted glass bubbler (60-70 micron max. pore diam.) | Saltzman reagent‡ | 0.4 | Nitrogen dioxide | 94-99 |
| Petri bubbler | Nitrating acid | 0.25 | Benzene | 95 |
| Impinger | Sulfuric acid | 28.3 | Ammonia | 95 |
| | 2% Glycerol in 0.05 N sodium hydroxide | 28.3 | Sulfur dioxide | 95 |
| Midget impinger | 1% KI, 0.1 M $KH_2PO_4$, and 0.1 M $Na_2HPO_4$ | 1-3 | Ozone | 95+ |

* Dependent upon nature of vapor, adsorbent, and number of tubes

‡ Dissolve 5 grams sulfanilic acid in almost a liter of nitrite free water containing 140 ml of glacial acetic acid; add 20 ml of 0.1% aqueous solution of N-(1-naphthyl)-ethylenediamine dihydrochloride and dilute to 1 liter.

The sampling instruments given in tables 1 and 2 include those most commonly used in gas and vapor sampling. Other devices, such as glass-bead packed columns containing absorbing solutions, freeze-out traps, the Harrold mist and gas collector, are invaluable in certain situations. The collection efficiencies reported in table 2 are absolute values based upon sampling of known atmospheric concentrations of the indicated gas or vapor at the specified rate of air flow.

In planning an environmental survey it must be remembered that gases and vapors, unlike dusts, fumes or mists, form a true solution with air and, hence, drastic physical or chemical action is required to collect these substances efficiently from the atmosphere. The industrial hygienist must be familiar with the advantages, limitations, and performance characteristics of each type of sampling device to provide samples of utmost value to the laboratory for chemical analysis.

In addition to the sampling instruments, a suction device, such as an electrically operated pump, which will provide a reasonably constant rate of air flow through the sampler and an air flow measuring instrument, such as an orifice flowmeter, are required to collect the sample. The flowmeter, attached to the assembled equipment, must be calibrated in the laboratory with a wet or dry gas meter or with a mercury manometer. A separate lecture entitled "Airflow Calibration of Air Sampling Equipment" is presented in Section B-5 and, therefore, the details of this calibration will not be discussed in the present lecture.

Instructions on the preparation of known concentrations of air contaminants are given in Section B-6. The industrial hygienist should be familiar with this technique so as to establish the collection efficiency of his sampling apparatus.

## Fundamental Gas Laws

The actual measurements of the volumes of gases or gas-air mixtures must be corrected to standard conditions of temperature and pressure in order for the measurements to have meaning. The conversion of actually measured volumes to the standard conditions of 0° C (273° K) and 760 mm of mercury pressure requires a knowledge of the fundamental gas laws, given in any text on general or physical chemistry.

The combined laws of Boyle and Charles state that the volume of a gas is inversely proportional to its pressure and directly proportional to its absolute temperature (°C + 273):

$$(1) \quad v \propto \frac{T}{p}$$

By introducing a proportionality factor, "k", expression (1) becomes

$$(2) \quad pv = kT$$

If this equation of state is applied to 1 mole of gas, "R", the molar gas constant, may be substituted for "k", and "V", the molar gas volume, may be used in place of "v". The equation can be made more general by introducing "n", which represents the number of moles in any given system. Equation (2)

then is converted to the familiar ideal gas equation

$$(3)\quad pV = nRT$$

where "R" must be expressed in the same terms as those used for the pressure-volume relationship. For example, if the gas volume is expressed in liters and the pressure in atmospheres, "R" has a value of 0.08206 liter-atmospheres per degree. If the volume is expressed in cubic centimeters and the pressure in terms of millimeters of mercury, the value of "R" is 62,370 cc-mm per degree Kelvin.

In dealing with the gas mixtures encountered in industrial hygiene, Dalton's law must be first applied to correct the pressure of the total mixture by that portion of the pressure due to water vapor in the atmosphere. Dalton's law states that the total pressure of a mixture of gases is the sum of the pressures of the individual gaseous components. Therefore, the barometric pressure at the time the gas is collected minus the pressure of the water vapor (often termed the aqueous tension) is the actual pressure of the gas or gas-air mixture. The pressure due to water vapor at the temperature of gas collection, assuming gas saturation with water vapor, can be found in any handbook of physical constants.

With these laws the following equation can be used to convert the measured gas volume to standard conditions of temperature and pressure:

$$(4)\quad V_{S.T.P.} = V_{meas.} \times \frac{P_{bar.} - P_w}{760} \times \frac{273}{273 + t^\circ C}$$

where the volume is expressed in any convenient unit, such as liters, and the pressure is expressed in terms of millimeters of mercury.

Special Calculations and Conversion Formulas

The equation for the calculation of the actual volume of a gas collected in a partically evacuated flask is

$$(5)\quad V_s = V_F \left(\frac{1 - T_s p_1}{T_1 p_s}\right)$$

where $V_s$ = volume of gas collected at absolute ($^\circ$K) temperature $T_s$ and pressure $p_s$

$V_F$ = volume of the flask

$p_1$ = residual pressure of flask after partial evacuation for sampling

and $T_1$ = temperature of flask ($^\circ$K) when $p_1$ was measured.

In applying a gas displacement method, the volume of the atmosphere being sampled or flushed through the flask should be 8 to 10 times that of the flask.(3) The time required to raise the concentration of the contaminant in the flask to 99 percent of its concentration in the entering air stream is given in the following equation:

(6) $t_{99\%} = 4.605\ a/b$

where t = time in minutes

a = volume of flask in liters

and b = air flow in liters per minute.

The following conversion formulas are useful for gas or vapor calculations:

(7) mg per liter x 1000 = mg per cubic meter

(8) mg per liter x 28.32 = mg per cubic foot

(9) mg per cubic foot x 35.314 = mg per cubic meter

(10) mg per cubic meter x 0.02832 = mg per cubic foot

(11) $\text{parts per million} = \frac{24{,}450 \times \text{mg per liter}}{\text{molecular weight}}$

1 gram molecular weight of a gas occupies 24.45 liters at 25° C and 760 mm of mercury pressure.

## Analysis

A complete review of the analytical methods which have been used successfully for the analysis of gas and vapor samples is beyond the scope of this lecture. These methods are given in detail in the reference texts[1,2,4] as well as an appended summary of satisfactory chemical methods for specific applications.[4] In addition to the methods suggested in these works, new techniques aimed at greater sensitivity and specificity are being developed which will be extremely helpful to the chemist. Of particular assistance will be the applications of vapor phase chromatography and infrared spectrophotometry. These techniques provide powerful tools to assist the chemist faced with the problems of identifying and determining part per million concentrations of mixtures of substances which may interfere with each other in the application of certain conventional chemical methods. Such substances as the halogenated hydrocarbons have generally had no specific analytical methods and their estimation on an individual basis has been difficult. With vapor phase chromatography, the physical separation and determination of such mixtures is now becoming possible. Infrared spectroscopy is an important technique in the analysis of such materials and, along with vapor phase chromatography which effects the separations and provides increased sensitivity, can be expected to play an important role in this field.

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# DIRECT READING INSTRUMENTS FOR DETERMINING CONCENTRATIONS OF AEROSOLS, GASES AND VAPORS

## Introduction

Direct reading instruments are those which give an immediate indication of the concentration of aerosols, gases or vapors by some means such as, reading a dial or noting the color change of a chemical. These instruments are favored by industrial hygienists as they give immediate results and save laborious laboratory work. One of the greatest needs of an industrial hygienist is a device for giving immediate answers to concentrations of gases and vapors. This is very helpful in preventing further injury to the worker and to advise management of the environmental conditions. There are many types of instruments which depend on some chemical or physical principle for their operation.

Advantages of direct reading instruments include the following:

1. Rapid answers.
2. Less work involved.
3. It is easier to convince the skeptical if they can see the instrument readings or color changes.
4. More readily adapted to continuous sampling.

Some of the disadvantages of direct reading instruments are as follows:

1. High initial cost.
2. Difficulty in keeping the instruments calibrated.
3. Many laboratories do not have facilities for calibration.
4. Some are quite heavy and bulky.
5. Some are not specific and mixtures interfere.

## Direct Physical Methods for Air Analyses

Every known physical characteristic of gas-air mixtures has been used for gas analysis. However, in the industrial hygiene instrument field, the fact that we are dealing with such low concentrations means that some of the changes in physical characteristics are too small to be measured accurately. The physical methods which are used for gas and vapor analysis are discussed below and are listed in Table 1.

### Optical Methods

Optical methods which are being used are the interferometric, photometric, and spectrum absorption, particularly in the ultraviolet and infrared regions.

Interferometer. The gas interferometer is a delicate instrument designed to compare the refractive indexes of two specimens of gas or vapor, one of which is a gas of known refractivity. This method is only applicable to the quantitative determination of gases and vapors whose index of refraction differ sufficiently from that of air. It is non-specific and where

several contaminants are present, the reading obtained is due to the combined effects of all. It is not suitable for low concentrations of gases or vapors.

Photometric. There are several instruments in which a photoelectric cell is used to measure the concentrations of aerosols by the scattering of light or the amount of light reaching the cell. These may be nothing more than a photocell in a smoke stack or a complicated electronic instrument such as the Transmissometer, Chaney Visibility Meter, Tyndallometer, Owl, Phoenix Smoke Photometer or PHS Smoke Photometer. A more detailed description of many of these instruments can be found in the Encyclopedia for Instrumentation for Industrial Hygiene.

Spectrum absorption. Spectrum absorption is probably the most widely used optical method. If ultraviolet light is passed through air containing a vapor such as mercury, which has an absorption band in the ultraviolet region, the amount of light reaching a photocell will be diminished in proportion to the concentration. The same principle could be applied in the infrared region.

Heat of combustion instruments. These instruments have been applied to non-combustible as well as low concentrations of combustible vapors. They are of the electrical or chemical combustion type with the heat liberated measured either by a Wheatstone bridge or thermopile and appropriate meter. They are not specific and must be calibrated for the single vapor or mixture being measured.

Miscellaneous. Other methods such as absorption (gasometric, manometric and gravimetric), Electrical Conductivity, Thermal Conductivity, and vapor pressure are listed in table 1, even though they are not commonly used in industrial hygiene instruments.

## Direct Chemical Reaction Devices

The method of collecting the sample in these devices depends upon the particular contaminant and the methods that have been devised. Probably the most desirable method is the collection of the gases or vapors on a solid absorbent, impregnated filter paper, or in liquid. The latter is the least desirable method.

### Solid Absorbents

These usually consist of granules of either absorbent gels such as silica gel or activated alumina or simply inert granules such as pumice or sand coated with the detecting chemical. The absorbent is placed in a small glass tube and air is aspirated through the tube at a known rate. Most absorbent gels are flow sensitive; therefore, the air should be drawn through the tube at the same rate as used in the calibration. This is particularly true in some of the carbon monoxide indicator tubes in which a color change is produced through the full length of the indicator gel. Individual methods are given in table 2.

### Test Papers

Impregnated test papers may be used in either of two ways: (1) by exposing them to the air, or (2) by drawing air through them. When drawing air through them the same precaution of flow rate applies as for the solid absorbents. Individual methods are given in table 3.

## Indicating Liquids

Indicating liquids can be used by aspirating air through the liquid until a given amount of standard solution, with an indicator, is neutralized (air titration) or they may be employed in the form of colorimetric reagents which develop colors matching a given standard or a series of standards (air colorimetry). The standards may be liquids or colored gelatin films encased in a plastic holder for comparative purposes.

Methods employing liquids are usually not considered highly desirable by field personnel, inasmuch as they require more care in handling, are bulkier and create disposal problems in many instances.

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NOTE: Tables 1 - 4 include material from "Sampling and Analyzing Air for Contaminants" with permission of Dr. Leslie Silverman.

Table 1. - Physical methods employed in air analysis

| No. | Method | Principle of Operation | Type of Sampling | Range Lower PPM | Range Upper PPM | Application and Remarks | Reference |
|---|---|---|---|---|---|---|---|
| A | Absorption (Volumetric Gasometric or Manometric) | Measured volume of gas-air mixture is absorbed by reagent and volume shrinkage determined. Pressure reduction may also be measured (Manometric) | Grab | 300 | 100% | Selective. Oxygen deficiency determinator. High concentrations of gases. Special forms will detect lower concentrations. Principally a laboratory apparatus. U. S. Bureau of Mines describes a portable form for field use. | 1 2 3 |
| B | Adsorption (Gravimetric) | Gas-air or vapor-air mixture is trapped on equilibrated adsorbent such as activated charcoal or silical gel. Change in weight represents weight of contaminant in known air volume. | Continuous | 1 | Depends on amount sampled | Non-selective. Used primarily for total solvents when a mixture of solvents is encountered. | 4 5 |
| C | Electrical Conductivity | Gas-air mixture is absorbed in a solution whose electrical conductivity is measured before and after absorption. | Grab or Continuous | 0.01 | 1% | Selective and can be applied to all acid or alkaline gases and also to vapors which can be burned to $CO_2$, $SO_2$ or HCl. Field instrument is available. | 6 7 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| D | Heat of Combustion<br>1. Benzol indicator or Vapotester | 1. Gas-air or vapor mixture is passed over a heated (above gas combustion temperature) platinum wire which is part of a balanced electrical resistance bridge. The combustion of the gas on the wire unbalances the bridge. | Grab | 20 | 1000 | Selective. Field instrument was designed primarily for benzol but can be applied to other combustible gases not removed by water vapor absorbent which is provided and necessary to the proper operation of the instrument. | 8<br>9 |
| | 2. Explosimeter (flammable) | 2. Explosimeter is a less sensitive form of the above instrument for explosive concentrations of flammable gases. | Grab | 10% of lower inflammable limit 1% in most sensitive inst. | 5% | Water vapor absorbent not necessary because of lower sensitivity, instrument is used for explosive and flammability determinations. Single meter calibrations for all gases the product of whose molecular heat of combination and the lower explosive limit is a constant. | 10 |
| | 3. CO indicator (Hopcalite) | 3. Gas is passed over a catalyst (Hopcalite) which converts CO to $CO_2$ and the heat of combustion is measured by a thermopile which is connected to a galvanometer. | Continuous | 10 | 10000 | Used in the field (portable apparatus) and tunnels for CO determination. Water vapor and oil fumes can poison catalyst and are removed before passing over catalyst. | 11<br>12 |

Table 1. (Cont.)

| No. | Method | Principle of Operation | Type of Sampling | Range Lower PPM | Range Upper PPM | Application and Remarks | Reference |
|---|---|---|---|---|---|---|---|
| E | Interferometer | Refractivity of gas-air or vapor-air mixture as compared to dry air is measured by comparison of interference fringes or Fraunhofer lines when light is passed through gas-air mixture. | Grab | 12 to 127 | 10% to 100% | Non-selective. Sensitivity depends on refractivity of gas or vapor. CO and moisture interfere and must be removed. Apparatus made in both portable and laboratory types. | 1 13 14 15 |
| F | Spectrum Absorption 1. Ultraviolet | 1. Gas-air or vapor-air mixture is passed through a tube with an ultraviolet light source at one end and a photoelectric cell at the other. Gases or vapors which have absorption bands in the ultraviolet or near ultraviolet absorb light and diminish cell response which is registered by a calibrated meter. | Grab or Continuous | 0.001 to 10 | 0.01 | 1. Sensitive to several gases and vapors. Maximum sensitivity for mercury vapor. Water vapor and $CO_2$ do not interfere. Field instruments for mercury, trichlorethylene, perchlorethylene have been developed. By using mercuric oxide at controlled temperatures mercury vapor detectors of the spectrum absorption type can be used with gases such as carbon monoxide (18) ethylene(19) and can be used to detect vapors such as benzene, ethanol and others(20). | 16 17 18 19 20 |

Table 1. (Cont.)

| | | | | | | |
|---|---|---|---|---|---|---|
| 2. Infrared | 2. Gas-air or vapor-air mixture is passed through a tube with a source of infrared (heated filament) at one end and a thermopile for recording absorption of infrared at the other end. Gases or vapors having absorption bands in infrared absorb the infrared rays and diminish amount of heat reaching thermopile. This is registered by a calibrated galvanometer or ammeter. Differential pressure unit is also used. | Grab or Continuous | 1 | | 2. Sensitive to all heteroatomic gases and vapors. Bands for absorption in infrared vary with gases just as in ultraviolet bands. Thus the methods may be made selective by the development of screening filters. Field infrared unit is available for CO and $CO_2$. British manufacturer can supply instrument for any heteroatomic gas specified. | 21 |
| 3. Mass Spectrum | 3. Separates according to mass gaseous ions formed in an evacuated system. Abundance of each ionic species is measured electrically by electrometer tube and amplifier. | Grab or Continuous | 1 | 5000 | 3. Applicable to all gases and vapors but requires expensive and elaborate equipment only adapted for laboratory handling of samples collected on silica gel and revaporized on grab samples. | 22 23 |

Table 1. (Cont.)

| No. | Method | Principle of Operation | Type of Sampling | Range Lower PPM | Range Upper PPM | Application and Remarks | Reference |
|---|---|---|---|---|---|---|---|
| G | Thermal | Gas or vapor mixture is passed over a heated wire (below ignition temperature). Change in heat loss of wire to surrounding gas mixture is registered by change in wire resistance measured on a balanced Wheatstone bridge. | Grab or Continuous | 25 | 10% | Non-selective. Water vapor and carbon dioxide interfere and must be removed. Greatest application in field is to high concentrations such as in flue gas analyzers. | 1 12 24 25 |
| H | Vapor Pressure | Gas or vapor-air mixture is passed over a cooled condensing surface in a confined chamber. The chamber is then evacuated and the condensed gas allowed to evaporate and its pressure measured. Ice-water, dry ice-acetone or liquid air mixtures may be used depending upon contaminant sampled. | Grab or Continuous | 10 | 1% | Selective. Only high concentrations, 1000 ppm or greater, can be obtained by grab samples. Applicable in field only to vapors or gases for which ice-water or dry ice-acetone mixtures can be used since liquid air is too difficult to obtain and handle. Contaminants must be miscible with water. Sensitivity depends on contaminant sampled and amount of air sampled in continuous method. | 26 27 28 29 |

Table 2. - Direct reading devices for gases and vapors - impregnated granules and gels

| Contaminant | Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Alcohol | | length of stain | 200-15,000 | | 1 |
| Ammonia | | length of stain | 72-14,400 | | 1 |
| Aromatic Hydrocarbons | | | | | |
| Benzene | Sulfuric acid & paraformaldehyde | length of stain | 0-100 | | 2 |
| " | | length of stain | 15-600 | | 1 |
| " | | length of stain | 30-900 | | 1 |
| " | | length of stain | 300-3000 | | 1 |
| Toluene | Sulfuric acid & paraformaldehyde | length of stain | 0-400 | Thiophene | 2 |
| " | " " " " | length of stain | 30-900 | | 1 |
| | | | 300-3000 | | 1 |
| Arsine | | length of stain | 0.5-5 | | 1 |
| Carbon Monoxide | Palladium sulfate & molybdenum | yellowish green to dark green | 10-10,000 | | 2 |
| " " | | length of stain | 10-3000 | | 1 |
| " " | | length of stain | 5,000-100,000 | | 1 |
| Carbon Dioxide | | length of stain | 5,000-50,000 | | 1 |
| " " | | length of stain | 10,000-200,000 | | 1 |
| Carbon Disulfide | | length of stain | 13-320 | | 1 |
| " " | | length of stain | 300-3000 | | 1 |
| Chlorine | | length of stain | 0.7-70 | | 1 |
| Hydrogen Cyanide | | length of stain | 0-50 | None | 2 |
| " " | | length of stain | 0.9-180 | | 1 |
| Hydrocarbons | | length of stain | 100-1200 | | 3 |
| Petrol & Gasoline | | length of stain | 540-13,500 | | 1 |
| Halogen Hydrocarbons | | | | | |
| Perchloroethylene | | length of stain | 20-1000 | | 3 |
| Trichloroethylene | | length of stain | 25-1600 | | 3 |
| " and others | | length of stain | 18-1800 | | 1 |

Table 2. (Cont.)

| Contaminant | Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Hydrogen Sulfide | | length of stain | 7-700 | | 1 |
| " " | Silver cyanide | length of stain | 0-50 | | 2 |
| " " | " " | length of stain | 0-400 | | 2 |
| " " | $Pb(OA_c)_2$ | length of stain | | | 4 |
| Mercury Vapor | | length of stain | 0.2-20 $mg/m^3$ | | 1 |
| Nitrous Gases | | length of stain | 5-100 | | 1 |
| Phosgene | | length of stain | 0.5-75 | | 1 |
| Sulfur Dioxide | | length of stain | 8-1200 | | 1 |
| " " | Ammonium vanadate or iodine & organic dye | length of stain | 0-50 | | 2 |

Table 3. - Direct reaction devices for gases or vapors - Test papers

| Contaminant | Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Ammonia $NH_3$ | Phenolphthalein paper | white to pink | 10-100 | Alkaline mists | 1 |
| | Litmus paper (blue) | blue to red reaction is timed | 10-100 | | |
| Aniline | Filter paper impregnated with furfural acetic acid | white to red reaction is timed | 5-150 | | 2 |
| Arsine $A_sH_3$ | S & S 598 filter paper 5% HgCl | light yellow to orange | 4-200 | $H_2S$ | 1 3 |
| | 10% $AgNO_3$ Filter paper moistened with $AgNO_3$ | black to white | 0.03-0.60 | | 4 |
| Carbon Dioxide | Whatman #1 sat'd $Sr(OH)_2 8H_2O$ and 1% Phenolphthalein Dilute to 90% before use | Time noted to colorize paper reagent from red to white | 10,000-200,000 | Extremely high conc. of other acid gases - very improbable | 5 |
| Carbon Monoxide | S & S 598 filter paper and 0.5% $PbCl_2$ Acetone | Shades of grey to black | 25-1000 | $H_2S$, $NH_3$, $H_2$ above 1%; ethylene above 1%; gasoline above 2.5% | 6 |
| Chloride | S & S 598, 5% KI, 0.5% starch glycerin Sulfurous acid | Brown to black | 0.25-12 | $Br_2$ | 7 |
| Fluoride | Zirconium azoarsenate filter paper moistened with 2N hydrochloric acid | Brown to pink to red | color change after 2 min. @ 0.02 cfm equal 3 ppm | | 8 |

Table 3. (Cont.)

| Contaminant | Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Hydrogen Cyanide | S & S 598 1% benzidine acetate and 3% copper acetate | light to dark blue | 10-50 | $Cl_2Br_2HCl$ | 9 |
| | Whatman #40 0.5% $HgCl_2$ and 0.25% methyl orange | pink to red color reaction timed | 20-200 | | 10 |
| Hydrogen Sulfide | Whatman #1 10 gms lead acetate 90 ml $H_2O$ and 5 ml acetic acid plus 10 ml glycerol | light tan to dark brown | 7-133 | mercaptans which are equally | 11 |
| | Filter paper moistened with ammonium molybdate | yellow | 10-50 | toxic | 4 |
| Mercury Vapor Hg | Warren's Cumberland coated 70 pound - SeS or Se | yellow to black | 0.1-70 | | 12 13 |
| Methyl Bromide $CH_2Br$ | S & S 598Y 0.006% fluorescein 20% NaOH | greenish yellow to strong pink | 0-150 | some other brominated hydrocarbons | 14 |
| Phosgene $COCl_2$ | Whatman No. 1, Diphenylamine . and dimethylamine benzaldehyde | light yellow to orange | 1-100 | $Cl_2$ and HCl | 15 |
| Phosphine $PH_3$ | Whatman No. 1, 1% $AgNO_3$ | brown to black | 1-100 | arsine and stibine | 1 3 |
| Stibine $SbH_3$ | Whatman No. 1, 1% $AsNO_3$ | brown to black | 1-100 | phosphine and arsine | 1 3 |
| Sulfur Dioxide $SO_2$ | S & S 598 starch KI $KIO_3$ glycerine | light brown to black | 4-200 | other oxidizing | 16 |
| | Filter paper saturated with potassium chromate | white to brown | 5-50 in 1 liter of air | agents which reduce iodine | 4 |

Table 4. - Direct chemical reaction devices for gases and vapors - direct indicating liquids

| Contaminant | Absorber and Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Ammonia $NH_3$ | Simple bubbler in test tube 0.0001N sulfuric acid methyl purple indicator | purple to green | 0-100 | alkaline gases | 1 |
| Aniline $CH_5NH_2$ | Simple bubbler 1% HCl. Bleaching powder, ammonia, phenol solution | colorless to blue | 5-200 | none | 2 |
| Benzene $C_6H_6$ | Simple test tube bubbler 0.5 ml of 40% HCHO in 10 ml conc. $H_2SO_4$ | straw to dark brown | < 100-1500 | toluene xylene naphthaline and thiophene | 3 |
| Carbon Dioxide $CO_2$ | Grab sample flask 0.0001 N NaOH and phenolphthalein | red to pink | 10-300 can be adapted to high concentrations | acid gases | 4 |
| Carbon Disulfide $CS_2$ | Simple test tube absorber copper acetate, alcohol diethylamine, triethanolamine | clear to yellow | 0.1-1400 | $H_2S$ use lead acetate paper as filter | 5 6 |
| Chlorine | Simple test tube absorber 0.1 gm o-tolidine in 100 ml conc. HCl made up to 1 liter with distilled $H_2O$ | clear to yellow | 1-63 | none | 7 |
| Hydrogen Cyanide HCN | Midget impinger. Phenolphthalein, copper sulfate, sodium phosphate buffer | clear to red | 0-300 | chlorine, bromine, iodine, phenol | 8 |
| Hydrogen Sulfide $H_2S$ | Midget impinger dilute solution of iodostarch | | | | 9 |

Table 4. - (Cont.)

| Contaminant | Absorber and Reagent | Color Range | Concentration PPM | Interference | Reference |
|---|---|---|---|---|---|
| Nitrogen Dioxide | Fritted bubbler 10 ml of sulfuric acid N-(1-naphthl) ethylenediamine dihydrochloride and acetic acid. | clear to violet red | 0.1-50 | | 10 |
| | Simple test tube absorber. Sulfamic acid and ∝-naphthylamine solution | clear to pink | 10-125 | ozone, chlorine | 11 |
| Sulfur Dioxide | Midget impinger at 2.83 lpm 0.0025 N iodine and 10% KI solution | blue to colorless | <1-100 | hydrogen sulfide, nitrogen dioxide, and other oxidizing gases | 12 |

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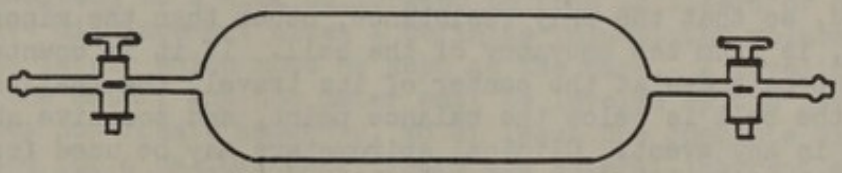
# AIR FLOW CALIBRATION OF AIR SAMPLING EQUIPMENT

## Introduction

In air sampling the object is to accurately assess the concentration of a contaminant in terms of count, weight or activity per unit volume. This usually involves the collection and analysis of a sample, sometimes simultaneously, as with squeeze bulb and indicating tube samplers. In a squeeze bulb sampler it is obvious that the degree of color change or length of stain depends both on the concentration of material and the number of squeezes. In general, the air concentration and the sample volume are equally important for the determination. Therefore, at least as much care should be taken in determining the sample volume as in analysis of the sample. The more precisely the sample volume is measured, the more precise is the final determination of contaminant concentration.

## Grab Sampling

Basically there are two types of samples, the grab or instantaneous sample, and the integrated or continuous sample. The grab sample may be taken in a flask of the type shown, or in any metal, glass or plastic container which



will not react with the contaminant or reagents added to the flask. The volume of such a sample is obviously the volume of the container, which can easily be measured with water and a graduated cylinder. The volume should be permanently marked on the flask.

Other grab samples may be collected by a piston-cylinder arrangement. These include the syringe used as a grab sampling flask, the "Samplair," and the grab-type dust samplers such as the konimeter, the Owens jet, and the B & L dust sampler. These either require no calibration or only an initial calibration.

The squeeze bulb instruments are, of course, made for use with a particular bulb, usually the 2 oz. size. Since the indicating tube calibration depends on the bulb size, no calibration of volume is necessary, as long as the bulb is always replaced by one of the same size. Some of the reactions which occur may be sensitive to rate of flow, as in the CO indicating tubes. Where this is the case, the valve must be adjusted to give the specified filling time.

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Continuous Sampling

This involves the collection of atmospheric contaminants from a measured volume of air for periods of from a few minutes to hours. Instruments such as the Greenburg-Smith impinger, electrostatic air sampler, and filter media with a source of suction are used for this purpose. Therefore, it is necessary to know the volume of air sampled in order to calculate the concentration on a weight per volume basis.

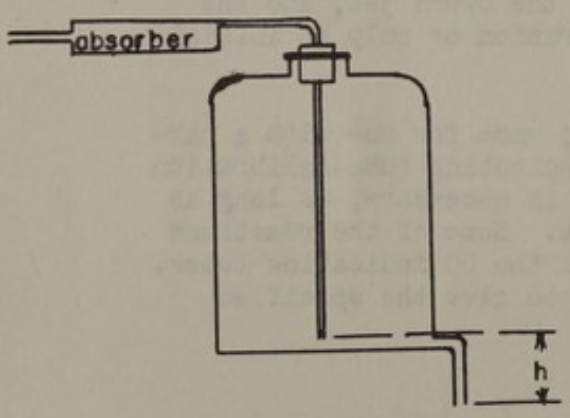
## Calibration Equipment

Ordinarily the air flow measuring devices on sampling instruments are calibrated in the laboratory before they are used in the field. It is also wise to check the calibration (or calibration curve, if one is prepared) of the flow measuring devices after the samples are collected. Calibration may be made by means of primary standards or secondary standards, which are periodically checked against primary standards.

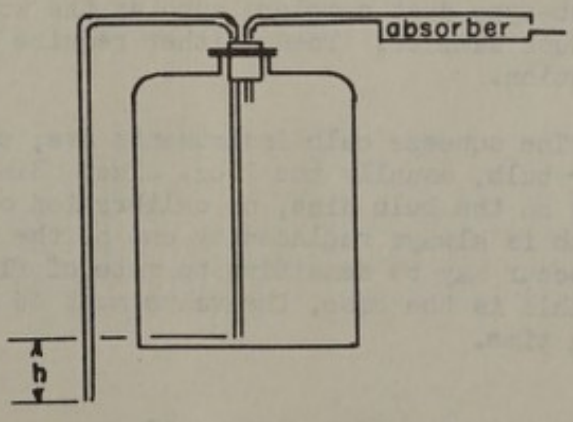
Primary Standards

The simplest and most dependable primary standard is one which uses the displacement of water in a cylinder. One such device is the spirometer. This is simply a bell floating in water - as air is blown in or drawn out the bell rises or falls. The volume change is thus $V = A\Delta H$, where A is the cross-sectional area of the bell, and $\Delta h$ is the change in height of the bell. The bell is counterweighted, so that the only resistance, other than the minor resistance of the piping, is from the bouyancy of the bell. If it is counterweighted so that it just balances at the center of its travel, the resistance will be negative when the bell is below the balance point, and positive above, but will be very small in any event. Clinical spirometers may be used for calibration of instruments with low flow rates (a liter or two per minute); a larger spirometer for laboratory work may be easily constructed for calibration of higher flow rates. For much higher flow rates, the meter checking facilities of a metropolitan gas company may be used.

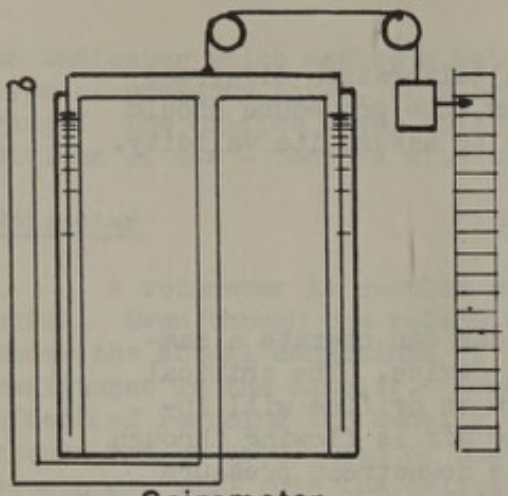
The marius bottle and siphon bottle are even simpler. These may be used for low rate, low resistance sampling and are graphically illustrated below with the spirometer on the following page.



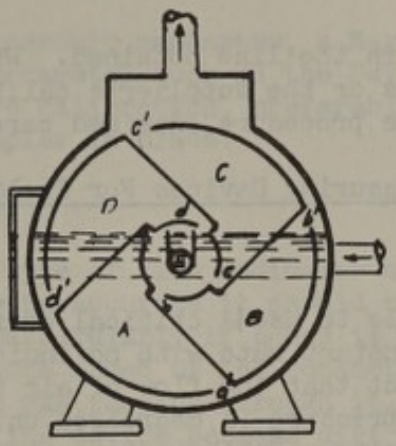

Marius Bottle




Siphon Bottle



Spirometer




Wet Test Meter

A,B,C,D, metering compartments

a,b,c,d, compartment inlets

$a^1$,$b^1$,$c^1$,$d^1$, compartment outlets

E, gas inlet

The wet gas meter is shown above. While not strictly a primary standard, when used as directed it is quite accurate and reliable. It consists of a drum half submerged in a liquid, with openings around the outside for the outlet of gas, partitions dividing the inside into a series of radial chambers, and inlets near the center. Gas enters the meter at the center, E. Flow of gas into a compartment causes it to rise, resulting in rotation of the drum. This brings another compartment below the liquid level, so that the gas is in turn displaced by the liquid entering this second compartment. Motion of the drum is transmitted to a dial calibrated to read in liters or cubic feet. Since positive displacement gives a definite volume, the volume will depend on the liquid level, which must be kept at the mark. A leveling bubble is provided on the meter, and the meter must be leveled by the leveling screws. The meter does not rotate smoothly, so at least one complete dial rotation should be used for a calibration. There are several inherent errors in using a wet gas meter. The blades are subject to corrosion, there is friction in the bearings, inertia must be overcome at low flows, and at high flows the liquid might surge and break the seal. In spite of these, the accuracy of a wet test meter is probably within one percent.

## Secondary Standards

A secondary standard is any sort of flow meter which has been accurately calibrated. They include rotameters, venturi meters, orifice meters, and the like. An example is the use of a previously calibrated venturi meter for calibration of a hi-vol sampler.(1) Several of the calibrations in the laboratory exercises are done with secondary standards. When using secondary standards care must be taken to see that the calibration does not change. Such changes can occur if the equipment is not kept clean or if it is not used under the conditions of calibration.

## General Considerations

Because accuracy of all the samples will depend on precision of the calibration, care should be taken in the procedure. Where a wet gas meter or spirometer is used, sufficient time should be allowed to obtain an accurate reading. Timing should be done with care, and flow through the sampler regulated as closely as possible. Enough points should be obtained on a calibration

curve to give confidence in the line obtained. Where a calibration disagrees with previous calibrations or the supplier's calibration, the procedure should be repeated, and the whole procedure examined carefully to assure its validity.

## Flow Measuring Devices For Field Use

### Critical Orifice

Where it is possible to use a critical orifice, one can operate a sampler at an essentially constant rate with no indicating device. The critical orifice depends on the fact that the flow of air through an orifice will increase as the downstream pressure is decreased until the air is flowing through the orifice at sonic velocity. Further decreases in the downstream pressure will no longer increase the flow. This critical flow point occurs when the downstream pressure is 53% of the upstream pressure. As long as the downstream pressure is less than 53% of the upstream pressure, flow is independent of downstream pressure. The equation for the determination of flow rate through a critical orifice is:

$$W = \frac{0.533\ AP_1}{\sqrt{T_1}}$$ where

$W$ = flow rate of air in #/sec

$A_2$ = orifice area in square inches

$P_1$ = upstream pressure, p.s.i.

$T_1$ = upstream temperature in $^{o}R$

It will be noted that flow through the critical orifice is directly proportional to upstream pressure and inversely proportional to the square root of the absolute temperature. Where resistance upstream of the critical orifice changes, as in the case of a filter which gradually plugs, flow through the orifice will change. Whether or not this is important will depend on the magnitude of the change of filter resistance with time. A correction for temperatures other than design temperature can be easily made.

Where a compressed air ejector is used, a critical orifice may be used for the Greenburg-Smith impinger. Critical orifices are also available for smaller flow rates such as those for the membrane filters. A critical orifice should normally be calibrated initially, with periodic checks to assure that plugging or abrasion has not occurred. This may be done by using a primary or calibrated secondary standard.

### Orifice Meter

For the majority of sampling units, the sampler is followed by a flow indicator of some sort, usually placed between the sampler and the vacuum source. An orifice meter is an obvious type, and one which may be conveniently built into a home-made sampling unit. The orifice meter will require

an indicator which may be a water or mercury manometer, a Magnehelic or other type of pressure gauge, or a bypass rotameter such as the Dwyer "Visi-float" gauge. The orifice meter will require calibration, preferably under the conditions of use - that is with the sampler in place.

Rotameter

A rotameter is another common type of flow indicator used in sampling units. Even though the rotameter may be accurate it should be calibrated under the actual conditions of use, particularly if there is an appreciable resistance in the sampler. A resistance ahead of the rotameter will give the effect of reducing the density of the gas passing through it. Rotameters should not be used ahead of the sampling device because dirt or corrosive substances will ruin the delicate float in time.

Vacuum Gauge

The pumps used with impingers are usually equipped with a vacuum gauge, using the impinger orifice as the resistance. With the proper size (2.3 mm.) nozzle in the impinger, a flow rate of 1 cfm will be obtained when the vacuum is three inches of mercury. The midget impinger will have a flow rate of 0.1 cfm with a vacuum of 12 inches of water if the nozzle diameter is 1 mm. Calibration of these units is thus done in two steps - first the vacuum gauge is checked at 12 inches of water or 3 inches of mercury; then the individual impingers are calibrated against the proper vacuum.

Samplers Without Flow Indicators

Sampling units such as the "Denver" or "Roberts" hand-cranked pump used with filter papers, or the new model MSA electrostatic precipitator are not (and cannot) be equipped with flow indicators, although for different reasons. In the case of the "Denver" pump the flow rate is too unsteady, and an indicator in the line would be subject to great fluctuation. The MSA precipitator uses a blower which is very sensitive to applied resistance, so that a flow meter cannot be added. The "Denver" pump can be calibrated by turning at a fixed rate (counting revolutions) and calibrating the volume per revolution. This volume will vary somewhat, depending on how well the pump is oiled. The precipitator may be calibrated by means of a thermoanemometer as described in Section D-4.

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# PREPARATION OF KNOWN CONCENTRATIONS OF AIR CONTAMINANTS

## Introduction

In the field of industrial hygiene, known low concentrations of air contaminants must frequently be prepared for instrument calibration, determination of collection efficiency, and toxicological studies. For gases and vapors, ACGIH threshold limits range from 0.001 ppm to 1,000 ppm. For dusts and fumes, threshold limits of 1 milligram per cubic meter or less are common.

Such concentrations of gases and vapors can be prepared by a number of methods. For a few gases and vapors and for all dusts and fumes the actual concentration must be determined by analysis, using a method of known efficiency. This is the case where the gas or vapor must be generated by a reaction of unknown efficiency. Dusts, fumes and mists cannot be prepared in known concentrations because particle settling, precipitation, agglomeration and flocculation cause continuous removal of the generated aerosol. Having determined the concentration produced by a given setting on the generating apparatus, the same setting can be used to approximate the concentration with sufficient accuracy for animal experimentation and the like. Such a concentration must be periodically sampled, however, and preferably continuously monitored. Silverman[1] has discussed in great detail the methods of generating gas and vapor concentrations in air. Dynamic concentrations are particularly well covered with a table of 61 gases and vapors, and methods which have been used to prepare them. This discussion will be limited to some of the methods found useful in our laboratory.

## Methods For Preparing Known Concentrations

### Static System (Batch Method)

For instrument calibration, the static system is widely used. A known amount of gas or vapor is dispersed into an enclosure of known volume. As air samples are withdrawn from the enclosure, the usual practice is to allow air to enter and replace the sample. The effect of dilution is thus dependent on the volume of sample and the volume of the enclosure. If one assumes instantaneous, perfect mixing of the incoming air with the entire sample volume, then the difference in concentration as a small volume is withdrawn is equal to the concentration times the fraction of the volume withdrawn, or $dC = C \frac{dV}{V_o}$; this integrates to $C = C_o e^{-\frac{V}{V_o}}$ or $2.3 \log_{10} \frac{C_o}{C} = \frac{V}{V_o}$

where C is the concentration at any time, V is the total volume of sample withdrawn, $C_o$ is the original concentration and $V_o$ is the chamber volume.

For any sampling then, the concentration in the chamber is gradually reduced. If the average concentration of the sample may vary 5% from the original, then the concentration can be reduced 10% during sampling. Using

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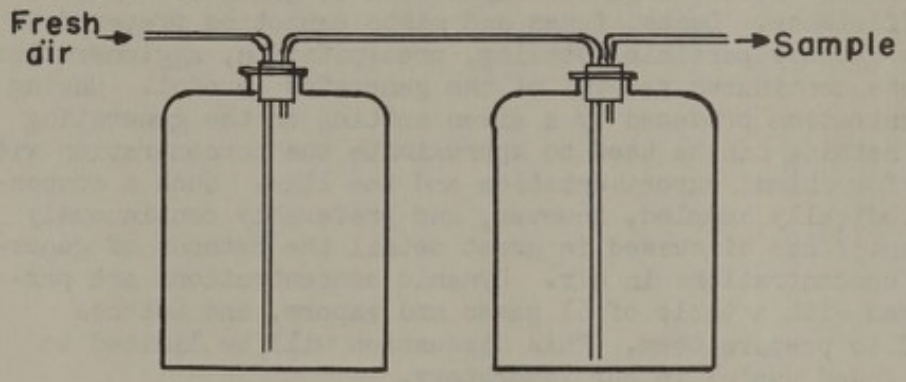
the above equation,

$$2.3 \log \frac{100}{90} = 2.3\ (0.04575) = 0.1053 = \frac{V}{V_o}$$

so that 10.5% of the volume can be sampled and still maintain an average concentration 95% of the original. The assumption of perfect, instantaneous mixing can be rather poor if no means are provided for circulating the air in a chamber. It can be readily demonstrated with smoke tubes, for example, if the incoming air is introduced near the bottom of a large bottle and perpendicular to it as shown below, that mixing is <u>not</u> immediate, the fresh air mixes only with the bottom layers, and a concentration gradient from top to bottom will thus be established. If such an arrangement is used, the assumption of instantaneous mixing will be on the safe side and the concentration sampled will actually be closer to the original. If the volume of one bottle is not




**Bottle Arrangement for Minimum Dilution**

sufficient to maintain the desired concentration then two or more bottles in series may be used. This requires that identical concentrations be prepared for each bottle. Detailed instructions for using 5 or 13 gallon bottles are given in reference (3).

Using this "compound dilution" method the concentration remaining in each of the bottles may be calculated using the formula:

$$C_n = \frac{C_o}{e^{\frac{V}{V_o}}} \left[ 1 + \frac{V}{V_o} + \frac{1}{2!} \left(\frac{V}{V_o}\right)^2 + \frac{1}{3!} \left(\frac{V}{V_o}\right)^3 + \frac{1}{(n-1)!} \left(\frac{V}{V_o}\right)^{n-1} \right]$$

Where $C_o$ = the original concentration in each bottle

$C_n$ = the residual concentration in the $n^{th}$ bottle (from which the sample is withdrawn)

$V_o$ = the volume of each bottle

$V$ = the total volume withdrawn in the same units as $V_o$

$e$ = the base of natural logarithms

Table 1, calculated from this equation, gives the dilution which may be expected for 1, 2, 3, 4 and 5 containers in series. The same table may be used to determine the volume of air which will be necessary to purge the chamber. For a residual of 1% of the original concentration in a single enclosure, for example, 4.6 times the chamber volume would be necessary, assuming perfect mixing. Since, as previously pointed out, perfect mixing may not occur, at least 3 and preferably 10 times the theoretical volume of air should be used.

Larger enclosures have the advantage that most of the common samplers may be used without appreciable dilution. In a 1000 liter chamber, for example, a 15 minute sample at 3 liters per minute will reduce the concentration only 4.4%, and the average concentration only 2.2%. Large chambers also have the advantage of reducing wall effects because of the increased volume-area ratio. Where wall effects may exert an effect, the walls may be preconditioned by setting up a concentration in the enclosure, purging the system well, and then setting up the desired concentration. By preparing the lower concentrations of a series first, the whole series may be run without purging the chamber.

Static System (Dilution Method)

This method uses the theoretical dilution formula for a single container to predict the concentration at any time, with steady removal of air mixture from the container. The assumption must be made that mixing is instantaneous and complete. By introducing the air through a small diameter tube tangentially to produce a high velocity and produce turbulence, and by installing a small fan within the container to create further mixing, this condition may be approached.

If the air is withdrawn at a constant rate, then $C = C_o e^{-(\frac{v}{V}t)}$

or $2.3 \log_{10} \frac{C_o}{C} = (\frac{v}{V}t)$

where $C$ = residual concentration at any time, $t$

$C_o$ = original concentration

$v$ = rate of air flow out of container (liters per min.)

$V$ = container volume (liters)

$t$ = time in minutes

If this is plotted on semi-log paper, the equation will be a straight line; C will be 10% of $C_o$ when $(\frac{v}{V}t)$ is 2.3; 1% of $C_o$ when $(\frac{v}{V}t)$ is 4.6; 0.1% at 6.9, etc.

Dynamic System

This method consists of diluting a metered stream of known concentration with a metered stream of fresh air to produce the desired mixture. There is no limitation on sampling volume or rate. Accurate knowledge of the stock concentration and accurate measurement of gas flows in the two streams are necessary. Where low concentrations are desired and the stock concentration is high, more than one dilution may be necessary. Since the volume of dilution air is usually great the volume of test mixture is usually far in

excess of the volume needed for instrument calibration. The main advantage of a dynamic system is that it provides an abundant supply of test mixture and also allows ease in changing concentrations. This method is ideal for repeated, continuous animal exposures in toxicology. Bottled gases or gas-air mixtures of known percentages are readily used. A volatile liquid may be atomized or injected into the air stream by a constant rate feeder. By passing air through a train of bubblers immersed in a constant temperature bath, a saturation concentration of a vapor in air may be produced and injected into the main air stream. Low concentrations of gas or vapor may be prepared by setting up a higher concentration in a static system, and feeding it into a dynamic system by displacement.

Most particulate generators are best adapted for dynamic systems. Although for certain aerosols the concentrations can be maintained within ± 5%, for most the concentration must be determined by sampling with some very efficient method such as electrostatic or thermal precipitator, or an absolute filter. Silverman(1) lists 32 types of dust feeders for aerosols, with the general principle and application for each as well as the reference source for each.

References

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2. Setterlind, A. N.: Preparation of Known Concentrations of Gases and Vapors in Air. AIHA Quarterly, June 1953.

3. Stead, F. M., and Taylor, G. J.: Calibration of Field Equipment from Air-Vapor Mixtures in a Five Gallon Bottle. Jour. Ind. Hyg. and Tox., Nov. 1947.

Note: Table 1 on the next page is by Setterlind.(2)
All bottles have equal volume and contain equal concentrations at the start.
Residual and average concentrations are given in percent of original.
First bottle is the one into which outside air enters.

Table 1. Average concentration of portion withdrawn and residual concentration of each of five bottles connected in series.

| Amt. withdrawn; % of single bottle vol. | 1st Bottle | | 2nd Bottle | | 3rd Bottle | | 4th Bottle | | 5th Bottle | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Residual conc. | Avg. conc. of volume withdrawn | Residual conc. | Avg. conc. of volume withdrawn | Residual conc. | Avg. conc. of volume withdrawn | Residual conc. | Avg. conc. of volume withdrawn | Residual conc. | Avg. conc. of volume withdrawn |
| 0 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.000 | 100.000 | 100.000 | 100.0000 |
| 10 | 90.48 | 95.24 | 99.53 | 99.76 | 99.98 | 99.99 | 99.997 | 99.998 | 99.997 | 99.9985 |
| 20 | 81.87 | 90.78 | 98.25 | 99.26 | 99.88 | 99.95 | 99.995 | 99.997 | 99.993 | 99.997 |
| 40 | 67.03 | 82.69 | 93.85 | 97.59 | 99.21 | 99.74 | 99.922 | 99.977 | 99.98 | 99.991 |
| 60 | 54.88 | 75.57 | 87.81 | 95.24 | 97.69 | 99.28 | 99.66 | 99.912 | 99.96 | 99.984 |
| 80 | 44.93 | 69.29 | 80.88 | 92.45 | 95.26 | 98.20 | 99.09 | 99.76 | 99.85 | 99.961 |
| 100 | 36.79 | 63.73 | 73.58 | 89.35 | 91.97 | 97.22 | 98.11 | 99.51 | 99.62 | 99.91 |
| 120 | 30.12 | 58.80 | 66.26 | 86.06 | 87.95 | 95.95 | 96.62 | 99.13 | 99.23 | 99.81 |
| 140 | 24.66 | 54.42 | 59.18 | 82.72 | 83.35 | 94.42 | 94.62 | 98.59 | 98.57 | 99.68 |
| 160 | 20.19 | 50.52 | 52.49 | 79.36 | 78.34 | 92.68 | 92.13 | 97.91 | 97.64 | 99.41 |
| 180 | 16.53 | 47.03 | 46.28 | 76.04 | 73.06 | 90.76 | 89.13 | 97.07 | 96.36 | 99.18 |
| 200 | 13.53 | 43.91 | 40.59 | 72.79 | 67.65 | 88.83 | 85.64 | 96.07 | 94.71 | 98.79 |
| 220 | 11.08 | 41.11 | 35.46 | 69.66 | 62.27 | 86.51 | 81.85 | 94.92 | 92.66 | 98.31 |
| 240 | 9.07 | 38.58 | 30.84 | 66.64 | 56.96 | 84.25 | 77.86 | 93.60 | 90.40 | 97.71 |
| 260 | 7.43 | 36.30 | 26.75 | 63.76 | 51.86 | 81.95 | 73.62 | 92.24 | 87.77 | 97.04 |
| 280 | 6.08 | 34.24 | 23.10 | 61.02 | 46.94 | 79.61 | 69.18 | 90.72 | 84.76 | 96.24 |
| 300 | 4.98 | 32.36 | 19.92 | 58.41 | 42.33 | 77.38 | 64.74 | 89.12 | 81.55 | 95.35 |
| 320 | 4.08 | 30.67 | 17.14 | 55.95 | 38.03 | 75.06 | 60.30 | 87.44 | 78.13 | 94.36 |
| 340 | 3.34 | 29.11 | 14.70 | 53.63 | 34.00 | 72.80 | 55.86 | 85.70 | 74.48 | 93.27 |
| 360 | 2.73 | 27.69 | 12.56 | 51.44 | 30.25 | 70.52 | 51.48 | 83.91 | 70.58 | 92.10 |
| 380 | 2.24 | 26.39 | 10.72 | 49.37 | 26.92 | 68.32 | 47.41 | 82.09 | 66.87 | 90.85 |
| 400 | 1.83 | 25.20 | 9.15 | 47.43 | 23.82 | 66.19 | 43.35 | 80.24 | 62.82 | 89.53 |
| 420 | 1.50 | 24.13 | 7.80 | 45.63 | 21.03 | 64.12 | 39.55 | 78.38 | 59.00 | 88.15 |
| 440 | 1.23 | 23.09 | 6.64 | 43.91 | 18.55 | 62.12 | 36.01 | 76.54 | 55.22 | 86.73 |
| 460 | 1.005 | 22.15 | 5.63 | 42.29 | 16.26 | 60.19 | 32.56 | 74.70 | 51.31 | 85.31 |
| 480 | 0.82 | 21.28 | 4.76 | 40.76 | 14.20 | 58.34 | 29.32 | 72.88 | 47.45 | 83.76 |
| 500 | 0.67 | 20.48 | 4.02 | 39.33 | 12.40 | 56.55 | 26.35 | 71.09 | 43.80 | 82.22 |

# SAMPLING AND COUNTING DUST

## Introduction

A standard procedure for sampling and counting dust to evaluate dusty atmospheres was adopted by the Fifth Annual Meeting of the National Conference of Governmental Industrial Hygienists in 1942. In the intervening years numerous modifications and different interpretations of the standard procedure have been introduced by industrial hygienists throughout the country. Since the threshold limit values were established using sampling and counting techniques contained in the standard procedure, the use of other techniques may lead to a non-valid interpretation of the results. Until other sampling devices and counting procedures have been accepted as standard, the Greenburg-Smith and midget impingers should be used for collecting samples and the counting procedure as outlined in the standard procedure which follows.

## Standard Impinger Sampling and Counting Technique

"I. Application. For the determination of the atmospheric concentration of an insoluble inorganic dust such as that associated with the production of lung fibrosis, the particle size of which is generally below 10 $\mu$ and, from the hygienic standpoint, is not of interest below 0.5 $\mu$. This method is not recommended for the determination of organic dusts, metallic fumes, vapors and gases.

II. Sampling instrument. The sampling instrument shall be the standard impinger, operating at a rate of 1.0 cfm at 3" Hg negative pressure (U. S. Public Health Service or modified form), or the micro-impinger operating at 0.1 cfm at 12" $H_2O$ negative pressure (U. S. Bureau of Mines).

III. Flow-producing apparatus and flow meter. Any type of suction apparatus may be employed, provided it is capable of maintaining the rate of flow with $\pm$ 3 percent of the rated capacity, i.e., 1.0 cfm for the standard and 0.1 cfm for the micro-impinger, and is provided with a calibrated flow meter and indicating gauge. The use of a constant-flow orifice is recommended with the compressed air ejector and an automatic negative pressure regulator with the motor or hand-driven pump. If an indirect indicating gauge is used, it shall be checked against a U tube or gas meter at regular intervals.

IV. Counting cell. The standard counting cell shall have a liquid depth of 1.0 mm. Counting cells less than 1.0 mm deep down to 0.1 mm may be employed, provided the liquid depth is reported with dust concentration data thus obtained.

V. Microscope and accessories

A. The microscope shall be a standard biological instrument or its equivalent, preferably with an adjustable tube length. It shall

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be provided with an Abbe' condenser with iris diaphragm, 16 mm objective 7.5 or 10x ocular.

B. The counting area shall be defined by an ocular grid or by rulings on the counting cell. It shall have an area equivalent to 1/4 sq. mm in the counting cell and shall be divided into 25 sub-squares.

C. The microscope tube length shall be adjusted so that the counting area corresponds to 1/4 sq. mm in the counting cell, or, in the case of a fixed tube length, the ruled area shall be equivalent to 1/4 sq. mm ± 3 percent. The counting area shall be accurately measured by means of a stage micrometer and recorded with the dust concentration data.

D. Illumination shall be provided by means of a 15 W substage lamp, placed directly under the condenser or a 75 W microscope lamp, placed approximately 10" from the plain mirror. A "Daylight" filter shall be used.

E. A microprojector of the type described by the Bureau of Mines(1,2) may be used instead of the preceding system.

The microscope of the microprojector shall be the same as the preceding microscope, except that a 20 or 25x ocular shall be used.

The counting area shall be defined by rulings on the translucent screen. It shall have an area equivalent to 1/4 sq. mm in the counting cell and shall be divided into 25 sub-squares, each of which may be further subdivided into not more than 10 similar (equal area) rectangles.

The distance between the microscope, with tube length adjusted to 160 mm, and the screen shall be such that the magnification of images upon the screen is 1,000 diameters. The microscope tube length shall be adjusted so that the counting area, or 50 cm square ruling on the screen, corresponds to 1/4 sq. mm in the counting cell, or, in the case of a fixed tube length, the ruled area on the screen shall be equivalent to 1/4 sq. mm ± 3 percent. The counting area shall be accurately measured by means of a stage micrometer and recorded with the dust concentration data.

Illumination shall be provided by means of an automatic-feed (preferably 4.5-6 ampere direct current) carbon arc lamp so arranged to supply adequate illumination on the screen. A suitable heat filter (water cell or heat-absorbing glass) shall be used between the lamp and the microscope.

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VI. Collecting liquid. Distilled water with or without alcohol (ethyl or isopropyl) shall be used as the collecting liquid. The collecting and subsequent washing and diluting liquids shall show not more than six (6) countable particles in 1/4 sq. mm counting area in the cell. The "blank" count shall be determined under the microscope for every series of samples.

VII. Sampling technic.

A. All glassware shall be clean and the equipment protected against dust contamination in the field. One impinger flask shall be treated exactly like the others except that no air shall be drawn through it. The liquid in it shall be used to determine the blank count which must be subtracted from the sample counts, as described below.

B. The number and spacing of the samples at a given operation, the locating of the sampling point, and the length of each sampling period shall be so chosen as to yield a representative measure of the exposure throughout the cycle of operation and also to indicate the peak concentrations. The minimum number of samples at each sampling point shall be two.

C. In addition to samples collected in the breathing zone of the exposed workers, samples should be taken to determine the dust concentration in the general workroom atmosphere and in special studies, to determine the sources of the dust dispersion or distribution.

VIII. Treatment of collected sample.

A. The sample shall be counted within 36 hours after collection except that in the case of flocculating dusts, the permitted holding time shall not exceed 12 hours. Special collecting liquids may be employed to minimize the degree of flocculation for certain dusts.

B. The sample shall be made up to a known volume, after washing down and removing the impinging nozzle. The concentration of the suspension shall be such as to give a dust count at least 4 times the "blank" count and not greater than 150 particles in the 1/4 sq. mm counting area. When further dilution from the initial volume is required to reduce the field count to the limits specified above, the dilution shall be made in steps not exceeding 1:10. Complete mixing of the suspension by vigorous agitation is required before removing a portion for dilution and volume removed shall not be less than 10 percent of the total volume of the suspension to be diluted, except that the minimum volume to be removed from the original sample for dilution shall not be less than 5 cc. Excessive dilutions are to be avoided by proper gauging of the sampling time in relation to the dust concentration.

IX. Preparation of the sample for counting.

A. Cells shall be cleaned before use and the effectiveness of cleaning shall be checked by microscopic examination of each cell before being filled.

B. The suspension to be counted shall be shaken vigorously and a portion transferred immediately to the cell by means of a clean pipette, taking care to prevent the inclusion of air bubbles.

C. One or more cells shall be filed from each sample.

D. Cell contents shall settle before counting, at least 30 minutes for the 1.0 mm cell and proportionately less for the cells of less depth.

E. One cell shall be made up from the "blank" flask for every series of samples.

F. Cells shall not be filled in advance of the time required for settling before counting.

X. Counting

A. Counting shall be done in a darkened room, using artificial illumination in the microscope, as described above.

B. The light and mirror shall be centered so that no shift of position of the particles is seen when focusing up and down. The condenser and diaphragm shall be adjusted to give an even moderate illumination. Each observer should determine and employ the optimum lighting for his eye.

C. Before counting, the field of the microscope shall be examined and, if necessary, the ocular grid cleaned to remove dust particles.

D. The count shall be made in a plane just above the surface of the cell rather than throughout the depth of the liquid. The microscope may be focused up and down by means of the slow-motion adjustment in order to bring individual particles into focus. Before making the count, the cell contents shall be carefully examined throughout the entire depth to make certain that all countable particles have settled.

E. Five fields of 1/4 sq. mm in area shall be counted, one in the central part and the other 4 towards the edges of the cell.

F. Except in the case of abnormal samples, no distinction shall be made between the number of particles above and below 10 $\mu$.

XI. Computation of results. The concentration of dust shall be expressed in millions of particles per cubic foot of air (m.p.p.c.f.), reported to 2 significant figures only.

Concentration is determined as follows:

$$\text{Conc. (m.p.p.c.f.)} = \frac{(N_s - N_c) \times C.F. \times V_w}{V_a}$$

Where:

$N_s$ = Mean count per field for the sample (5 or more fields)
$N_c$ = Mean count per field for the blank (5 or more fields)
C.F. = Cell factor = 1000/(area of counting field in sq. mm) (x depth of cell in mm)
$V_w$ = Total water volume (equivalent total, when portion diluted, i.e., initial sample volume x dilution factor)
$V_a$ = Air volume sampled in cu. ft.

EXAMPLE:

Duration of sample time: 5 minutes
Rate of sampling: 0.1 cubic foot per minute
Vol. in flask after washing: 30 cc
Ratio of added dilution (5 cc in 25 cc flask): 5:1
Cell depth: 1 mm

Dust Count, light field (2 cells) particles less than 10 microns in size

| Cell #1 | Cell #2 |
|---|---|
| 51 | 48 |
| 36 | 54 |
| 44 | 47 |
| 34 | 40 |
| 55 | 49 |
| 220 | 238 |

Total #1 220
Total #2 238
458 + 45.8 Av. Total/Field
- 5 blank count
40.8 Av. Net Count

CALCULATIONS:

$$\frac{\text{Av. Net Count per 1/4 mm x 4 x cu. mm per cc x flask vol. x extra dil. ratio}}{\text{Cu. ft. per minute sampled x sampling time}}$$

$$\frac{40.8 \times 4 \times 100 \times 30 \times 5}{0.1 \times 5} = 48 \times 10^6 \text{ particles per cubic foot}$$

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# THE DETERMINATION OF PARTICLE SIZE DISTRIBUTION IN INDUSTRIAL DUST

## Introduction

Very early in recorded history certain writers noted the high incidence of respiratory disease among workers in occupations such as mining and stone cutting. The cause of such disease was variously described as pestilential air, the vapors of metals, and metal emanations. Later it was recognized that dust was often the causative agent and that all dusts did not exhibit the same effect upon exposed workers. Further, it was observed that, in certain instances where visible dust was practically absent from the air, workers still contracted pulmonary disease resulting from dust exposure. Thus, the significance of particle size in dust exposures was recognized.

As a solid material is disintegrated into smaller and smaller particles, the surface area of a given mass of the solid is tremendously increased, and, as a direct result, those properties which depend upon the activity of the surface molecules are greatly enhanced. Chemical and physical activity are both increased by fine division.

Many investigations have been conducted in attempts to determine particle sizes, or particle size range, of dusts which are of greatest significance in the production of pulmonary disease resulting from dust exposure.

Some investigators approached the problem by examination of silicotic lung tissue.[1,2] Most of these investigators observed relatively few particles larger than 3 microns in silicotic nodules of the lungs. This method has been criticized from the standpoint that particles remaining in the lung may not have been those which caused the lung damage, and that particle size might have been altered by solubility between the time of deposition and the time of observation.

Other researchers have investigated particle sizes of dust retained in the respiratory system.[3,4,5,6] Work by Brown, Cook, Ney and Hatch[7] is a very interesting example of comprehensive investigations of this type. The methods and techniques of this study will not be discussed but consideration of their results and conclusions will serve as an excellent illustration of the significance of particle size with respect to respiratory retention of dust.

The following conclusions are presented in this paper:

1. Over-all respiratory retention is essentially complete for particles above 5 $\mu$. It decreases with size to a minimum of about 25 percent for approximately 1/4 $\mu$ particles and then increases again for sub-microscopic particles.

2. Filtering efficiency of the nasal passages is also practically 100 percent for particles larger than 5 $\mu$ and decreases systematically with size, reaching zero, however, for particles of finite size of about 1 $\mu$.

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3. Depth of penetration before deposition increases with decreasing size. For those particles, larger than 1 μ, which escape trapping in the upper respiratory tract, alveolar retention is complete. Smaller particles are retained in decreasing amounts but, however small the retention may be, practically all the deposition takes place in the alveoli.

4. Thus, there is an optimum particle size having the highest probability of alveolar deposition. This is about 1 μ. Smaller particles, down to 1/4 μ, have approximately the same probability of being retained in the alveoli as those from 1 to 5 μ. Sub-microscopic particles are deposited in the alveoli in increasing amounts as size is further reduced and, theoretically, retention approaches a maximum value corresponding to the percentage of tidal air which reaches the alveoli.

5. The relationship between particle size and penetration, and deposition of dust changes with the respiratory pattern. Retention increases with decreasing respiratory frequency. The effect is greatest for particles between 1 and 5 μ for which there is a much greater proportionate increase in alveolar deposition than in the over-all retention as breathing rate decreases. The respiratory pattern has no significant effect upon the deposition of particles smaller than 1 μ.

These conclusions, based partially on theory and partially on experimental data, indicate a very definite relationship between particle size and degree of deposition in the lung. If we make the logical assumption that lung damage is related to deposition of dust in the lung, it follows that particle size determinations are essential in the evaluation of dust hazards.

## Particle Size Measurement

Particle size determinations may be made by either direct or indirect means. In the direct methods, the particles are observed microscopically, their images are projected onto a viewing screen or they are photographed, and the individual particles are compared with a calibrated scale. In indirect methods, some property of the dust, which is related to particle size, is evaluated. Direct methods, involving microscopic examination, will be discussed in detail; only brief mention will be made of some of the indirect methods and of direct methods which require elaborate or specialized equipment.

### Indirect Methods

One indirect method for determining the average particle size of a sample of dust is known as the "count-weight" method.[8] The number of particles in any desired weight of the material is determined and the following calculation is applied:

$$D = \sqrt[3]{\frac{6}{\pi \sigma N}}$$

where: D = particle diameter in centimeters
$\sigma$ = density of the dust in grams/cubic centimeter
N = number of particles per gram of dust

The solution of this equation gives the diameter of a hypothetical sphere having the average volume of the particles in the sample. The equation could be altered to determine any dimension of any desired geometric shape on the same volume basis. Size frequency distribution cannot be determined by this method unless it is applied to sized fractions obtained by settling or elutriation of the original dust sample.(9) The same general type of reasoning can be applied to the determination of particle size through settling or elutriation and the application of Stokes Law. The Roller apparatus(10) could be applied to such a determination. Sizes calculated, however, would be related to the Stokes Law resistance of the particles rather than to their volume.

Particle size has been determined by the observation of light scattering. The Owl(11) is an instrument based on this phenomenon. At its present stage of development this application is highly technical and results can be interpreted only for suspensions of particles of uniform size. With this instrument, the particle size of an aerosol having particles of uniform size can be determined even though the size is below the limit of resolution of the optical microscope. Mathematical treatment to interpret results for irregularly shaped particles, even though of uniform size, is much more complex than the treatment necessary for spherical particles.

Instruments utilizing the phenomenon of light scattering appear to have little practical value for particle size determination in routine industrial hygiene work.

Direct Methods

The direct methods for particle size determination involve the use of a microscope. Through this instrument, a dust sample may be viewed directly, its image may be projected onto a viewing screen, or it may be photographed.

The electron microscope can, without question, give the most detailed particle size determination of any of the direct methods. The use of this instrument is highly technical and is a field in itself. The electron microscope does not have the dimensional limitations of optical systems, and can resolve dust particles several orders of magnitude smaller than can an optical microscope. Since this instrument is not generally available for routine industrial hygiene work, it appears that its greatest value in particle size determination is in basic research.

Most microscopic particle size determinations are made with optical microscopes at levels of magnification which necessitate the use of oil immersion objective lenses. For maximum definition and to achieve the limit of resolution, it is necessary, in most cases, that immersion oil be applied between the top of the condenser and the lower surface of the sample slide and the objective lens of the microscope. In other words, oil immersion requirements will not be satisfied if a cover slip is placed over a dry mount and immersion oil is used only between the cover slip and the objective lens. As a general rule, there should be no air-glass interfaces between the condenser and the microscope objective. Discussion of this matter will appear in any standard text on microscopy and probably in manufacturers' literature which normally accompanies microscopes.

The basic principles of sampling and the techniques of obtaining representative portions of samples for observation have been discussed. These principles apply to the preparation of samples for particle size determination. In prepared slides for particle size determination, the random distribution of particles with respect to size is assumed. It is further assumed that the size distribution determined from a series of random fields will be representative of the sample. In the observation of slides upon which deposition of particles with respect to size is not of a random nature, care must be exercised to measure all particles in a mathematically representative portion of the deposit. This particular problem will be encountered in the evaluation of slides from instruments such as the Owens jet dust counter or the strip from a spiral sampler. Such instruments are selective with respect to the location upon the slide at which particles of various sizes are deposited. On an Owens jet slide, for example, it will be found that the mean particle size in the secondary ribbons of dust which lie on either side and parallel to the major deposit have a smaller mean size than does the dust in the major deposit. A representative size distribution on such a slide may be obtained by measuring all particles in a strip of uniform width extending across the entire dust deposit and perpendicular to the major axis of the deposit.

Care must be taken to measure every visible particle in the selected field. The observer must overcome the unconscious tendency to focus his microscope on and measure only the larger, more easily seen particles. When observing slides upon which all particles are not in the same plane, for example, a membrane filter sample, the observer should manipulate the focus so as to measure all particles appearing in the selected field throughout the depth of the deposit. This is critical for an accurate size distribution determination.

Microprojector. This consists of a microscope equipped with a projection prism accessory, a projection light source, and a viewing screen. This is useful in particle size determinations. The projected images of particles may be measured directly on the viewing screen by use of a calibrated scale. While some loss of definition is suffered in projection, the method has the advantage of being less tiring for the observer than direct microscopic observation. The design and use of microprojectors is discussed in the literature.[12,13,14]

Photographic technique. A microscope may be fitted with a camera and photographs of the microscope field may be obtained. Such photographs, or photomicrographs, of dust samples may be utilized for particle size determination.[15] A means of calibration to permit measurement of the photographed particles must be provided. An advantage of the method is that a number of photomicrographs can be taken at any time and evaluated at leisure; a disadvantage is that irregularities in the photographic process, possibly undetected, may preclude or invalidate evaluation of the sample.

Filar micrometer. In the United States, the filar micrometer was probably the most widely used of all measuring devices for particle size determinations. The introduction of a simplified eyepiece graticule in 1952, however, has gradually replaced this technique. The filar micrometer consists of an eyepiece with a movable crosshair actuated by a calibrated

micrometer screw. This instrument may be used to measure the horizontal linear dimension of particles in the microscope field. The filar scale must be calibrated with a known standard for each optical system. Commonly used is a standard stage micrometer which has rulings at intervals of 0.1 mm and 0.01 mm. With a 97x oil immersion objective, a filar unit is equivalent to approximately 0.1 $\mu$.

The filar micrometer is most easily used with 2 persons working together. One person observes the slide through the microscope and manipulates the micrometer screw to measure the particles; the second person observes and records the filar unit value of each measured particle as indicated by the micrometer scale. In measuring a particle with the filar micrometer, it is good practice to approach the particle with the crosshair in the same direction as the particle is to be measured. This reduces or eliminates the inaccuracies which result from play in the mechanical linkage between the crosshair and the micrometer scale. Since the crosshair has a significant width, the observer should also use the same side of the crosshair as an index, especially in measuring smaller particles.

The filar micrometer, as normally used, is fixed on the microscope and measures particles in one direction only. The dimension usually determined is the distance between the two extreme points on a particle in a horizontal direction. Irregular particles possess no definite geometric diameter, so this arbitrary dimension, which is easily defined and duplicated, is an adequate indication of size when a sufficient number of particles are measured, and the particles have random orientation on the slide.

Eyepiece graticule. A simple method of microscopic particle size measurement utilizes a graticule which is placed at the focal plane of the eyepiece.[16,17,18] The eyepiece graticule, of which there are several types, has geometric figures of various sizes which are compared to particles in the microscope field. One such graticule (see page 6) has as indices a series of circles and a logarithmic scale for comparison with particles. The circles are numbered 1 through 9 and the diameter, in arbitrary units, of each circle is defined by the equation:

$$D = \sqrt{2^N}$$

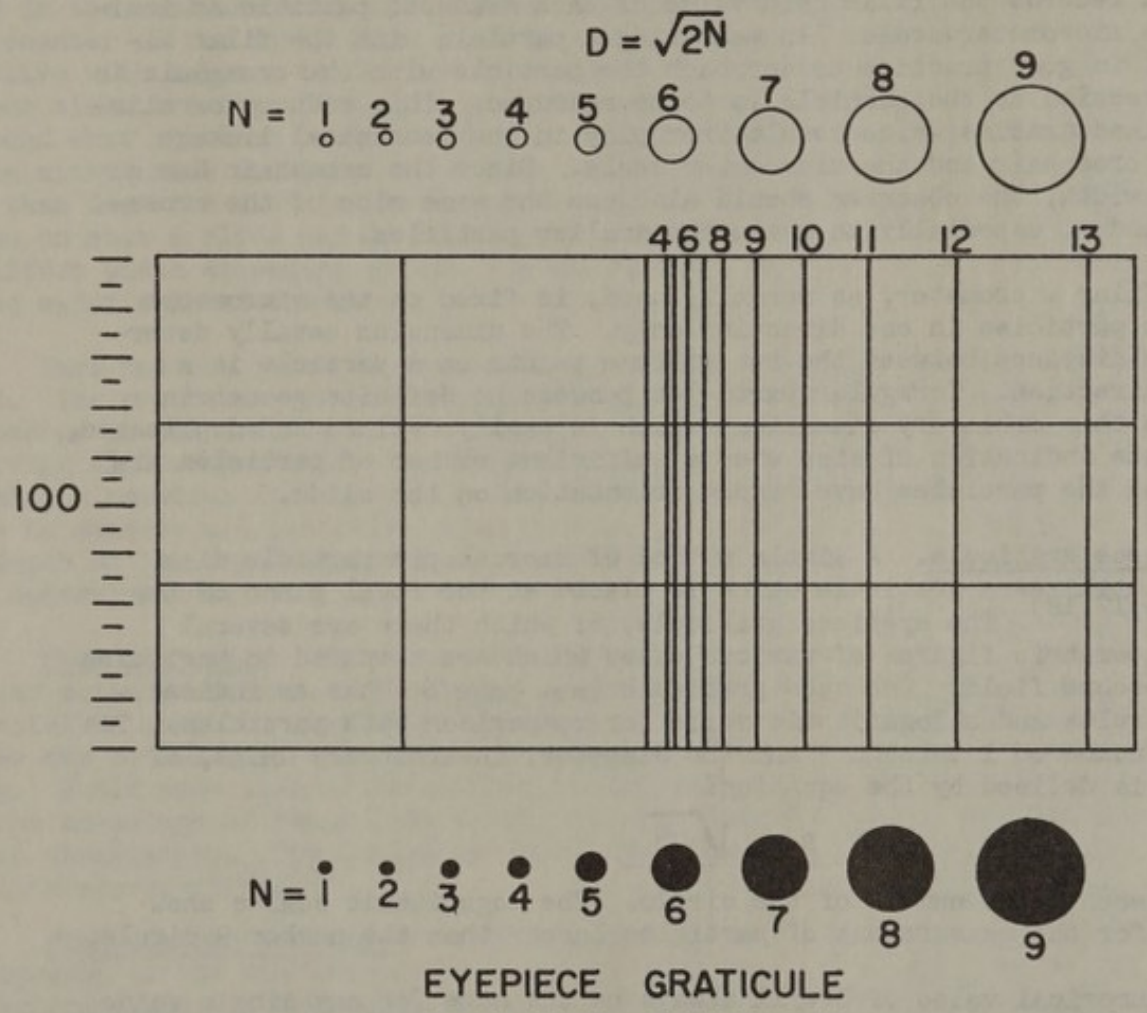
where N represents the number of the circle. The logarithmic scales show values of N for the measurement of particles larger than the number 9 circle.

The numerical value of D will always be the same for any single value of N. The dimensional value of D, however, is dependent upon the optical system being used. The linear dimensions of numerical values of D must be determined by calibration for each optical system. Calibration is easily accomplished by use of a stage micrometer.

The large rectangle in this graticule is 200 D units in length and 100 D units in width. Thus, calibration is easily accomplished by determining the number of microns on a stage micrometer included in either of these dimensions.

The observer must be continually aware of the necessity of measuring the same dimension of each particle with the eyepiece graticule. The nature

of the filar micrometer makes it easy and practically necessary to measure only the horizontal dimension. This is not the case with the eyepiece graticule. There may be a tendency to select the index circle which most nearly matches the longer dimension of the particle being considered. One dimension, say, the dimension of each particle in the horizontal direction, should be determined and recorded. If this is done, the results of particle size determinations with the filar micrometer and eyepiece graticule should be comparable.




EYEPIECE GRATICULE

Our experience has been that trained observers can make particle size determinations much more rapidly, and with less fatigue with the eyepiece graticule than with the filar micrometer.

Calculations. Particle size data obtained by any of the direct methods is most easily recorded and calculated by the use of a prepared data sheet. Forms which have been found convenient for filar micrometer data and for eyepiece graticule data follow at the end of this discussion. The filar

unit value or eyepiece graticule number for each particle observed is recorded at the time of observation in a small square at the top of the form. The micron values for eyepiece or filar units are recorded in the size column of the calculation portion of the form; the number, or tally, of particles of each observed size is recorded in the number column; the cumulative total number of particles measured is recorded in the total column; and the cumulative percent of the total number of particles is recorded in the percent column. Thus, the total or percent recorded for any given size represents the number or percent of all particles that size and smaller.

The determination of any one of the mathematical "average diameters" for a dust sample does not completely define the size characteristics of the dust. The size characteristics of a non-uniform dust are well shown, however, by a size-frequency curve. Such a curve is obtained when the individual particle size measurements are arranged in groups of equal size intervals and when the mid-value of sizes in each interval is plotted against frequency of occurrence of particles in each group. Such curves, when obtained by optical methods and plotted on linear graph paper are generally skewed.

Geometric mean diameter. Particle size distribution data can be plotted to better advantage on logarithmic-probability paper. Such a plot provides a graphical solution for the geometric mean and the standard geometric deviation. The plotting of particle size versus cumulative percentage on logarithmic-probability coordinates represents the integration of the logarithmic size frequency of the sample. Most dusts appear to exhibit normal logarithmic probability size distribution. Size distribution data for such a dust plot is a straight line on logarithmic-probability coordinates. The geometric mean diameter is the 50 percent size and the standard geometric deviation is:

$$\sigma_g = \frac{\text{84.13 percent size}}{\text{50 percent size}} = \frac{\text{50 percent size}}{\text{15.87 percent size}}$$

All average diameters are mathematically defined by the geometric mean and the standard geometric deviation.

A convenient table, showing the mathematical relationships between the geometric mean average diameter and the arithmetic mean, specific surface, surface area, volume, and surface area per unit volume average diameters appears in the text INDUSTRIAL DUST, by Drinker and Hatch.[19] Calculation of these values requires data in terms of frequency by count as would be obtained by direct microscopic measurement. In sedimentation or elutriation procedures, however, particle size data are usually determined as percentage by weight. It has been shown[20] that the standard geometric deviation by weight is the same as the standard geometric deviation by count. The following relationship exists between the geometric mean diameter by weight and the geometric mean diameter by count:

$$\log M_g = \log M'_g - 6.9078 \log^2 \sigma_g$$

where: $M_g$ = geometric mean by count

$M'_g$ = geometric mean by weight

$\sigma_g$ = standard geometric deviation by count or weight

Through application of this equation, size distribution data by either count or weight can be converted to the other for convenience or for comparison with other data.

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# PARTICLE SIZE DATA SHEET

DATE EXAMINED:

BY:

SAMPLE DESCRIPTION:

## FILAR READINGS

## CALCULATION SHEET

| A / B | NUMBER | TOTAL | % | A / B | NUMBER | TOTAL | % | A / B | NUMBER | TOTAL | % | A / B | NUMBER | TOTAL | % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | | 16 | | | | 31 | | | | | | | |
| 2 | | | | 17 | | | | 32 | | | | | | | |
| 3 | | | | 18 | | | | 33 | | | | | | | |
| 4 | | | | 19 | | | | 34 | | | | | | | |
| 5 | | | | 20 | | | | 35 | | | | | | | |
| 6 | | | | 21 | | | | 36 | | | | | | | |
| 7 | | | | 22 | | | | 37 | | | | | | | |
| 8 | | | | 23 | | | | 38 | | | | | | | |
| 9 | | | | 24 | | | | 39 | | | | | | | |
| 10 | | | | 25 | | | | 40 | | | | | | | |
| 11 | | | | 26 | | | | 41 | | | | | | | |
| 12 | | | | 27 | | | | 42 | | | | | | | |
| 13 | | | | 28 | | | | 43 | | | | | | | |
| 14 | | | | 29 | | | | 44 | | | | | | | |
| 15 | | | | 30 | | | | 45 | | | | | | | |

A = FILAR READINGS
B = EQUIVALENT DIAMETER IN MICRONS

CALCULATED BY:

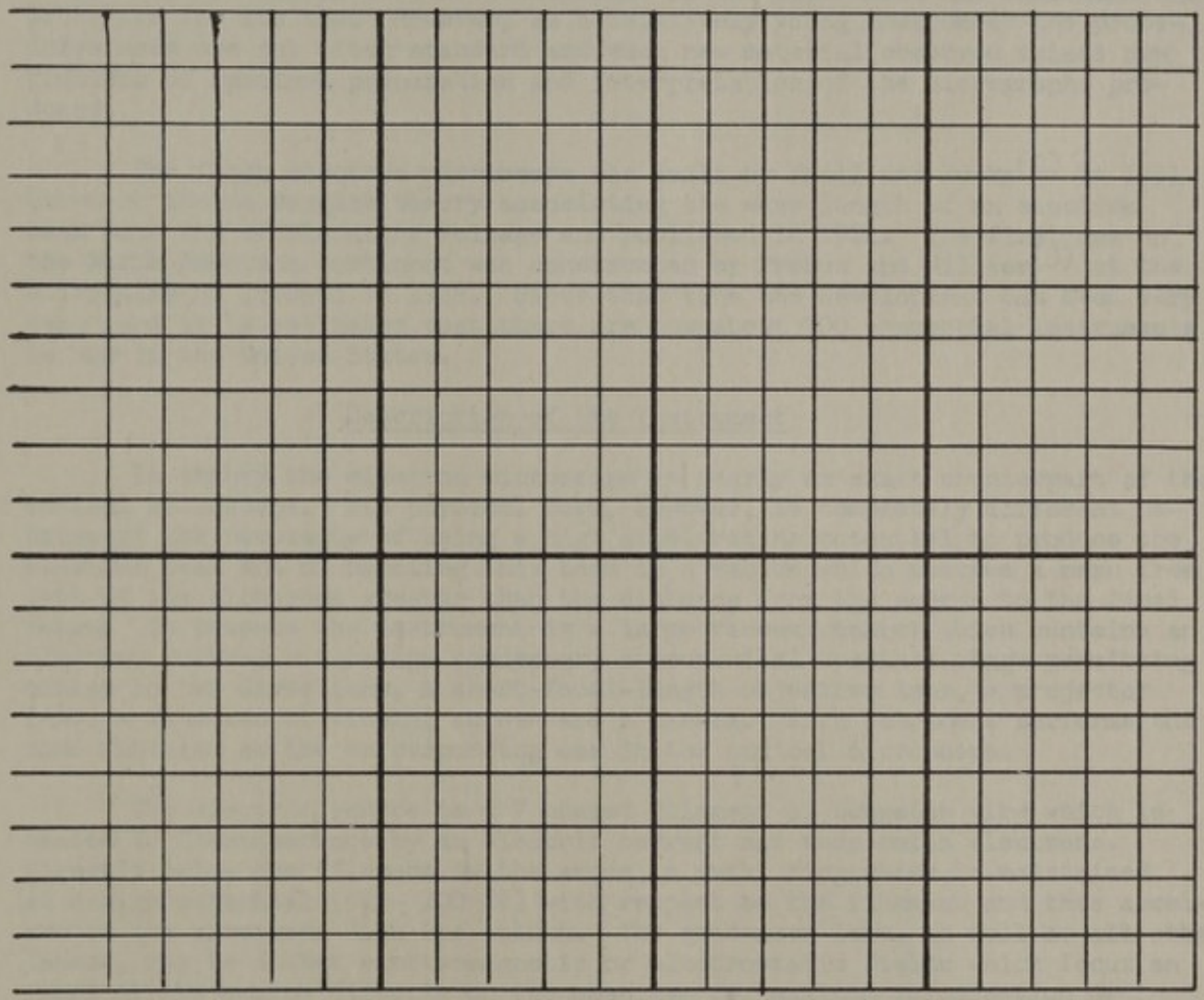
# PARTICLE SIZE DATA SHEET

Date Examined:

By:

Sample No.:

Description:



## Calculations

| Circle | Size | No. | Total | Cum. % | Circle | Size | No. | Total | Cum. % |
|---|---|---|---|---|---|---|---|---|---|
| 1 | | | | | 8 | | | | |
| 2 | | | | | 9 | | | | |
| 3 | | | | | 10 | | | | |
| 4 | | | | | 11 | | | | |
| 5 | | | | | 12 | | | | |
| 6 | | | | | 13 | | | | |
| 7 | | | | | > 13 | | | | |

# ELECTRON MICROSCOPY

## Introduction

The electron microscope has been defined as an aid to vision in the study of solid structures in the 0.001 to 10 $\mu$ range(1). This definition completely and simply describes the value of the instrument and indicates the procedure for its use. However, as a relatively young instrument the procedures used are not often standard and each new material observed raises new problems of specimen preparation and interpretation of the micrographs produced.

The first electron microscope was built by Knoll and Ruska(2) in 1931 based on the de Broglie theory associating the wave length of an electron beam with the accelerating voltage and published in 1924. The first one on the North American continent was constructed by Prebus and Hillier(3) at the University of Toronto in 1938. Since that time the development has been very rapid and it is estimated that there are now about 700 commercial instruments in use in the United States.

## Description of the Instrument

In theory the electron microscope is nearly an exact counterpart of the optical microscope. Its physical form, however, is completely different because of the necessity of using a high accelerating potential to produce the electron beam and of handling this beam in a vacuum which assures a mean free path of the electrons greater than the distance from the source to the final image. In essence the instrument is a large vacuum chamber which contains an electron source, a substage condenser, a mechanical specimen stage permitting motion in two directions, a short-focal-length objective lens, a projector lens, a fluorescent viewing screen and a camera. Each component performs the same function as the corresponding one in the optical microscope.

The electron source is a V shaped filament of tungsten wire which is heated to incandescence by an electric current and thus emits electrons. Directly below the filament is the anode, a metal ring which is maintained at a high potential (50 - 100 KV) with respect to the filament and thus accelerates the electrons down the column. The condenser lens, as well as all other lenses, may be either electromagnetic or electrostatic fields which focus an image of the source directly on the specimen for maximum illumination or slightly above or below the specimen for reduced illumination. Unlike the optical microscope, the lenses need not be moved to change focus but can be made either stronger or weaker by controlling the current through them. The specimen holder is an inverted cone shaped piece which projects downward into the center of the objective lens with the specimen screen held securely at the apex. The objective lens is the heart of the instrument. It is a strong lens which minimizes spherical and chromatic aberrations that would otherwise severely limit the resolving power of the instrument and has a highly

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compensated pole piece to correct astigmatism. The projection lens controls the final magnification of the image and projects this image on the viewing screen or film. The electronic image is, of course, invisible to the human eye and therefore a fluorescent screen is used to convert it to visible light for preliminary scanning and focusing. The camera consists of a photographic plate placed below the viewing screen so that when this screen is removed or tilted the electronic image falls on the photographic emulsion which is quite sensitive to electron bombardment. This plate is developed using standard photographic techniques.

## Preparation of Specimens

As in optical microscopy, the main research problem connected with the use of the electron microscope is in the preparation of specimens which are accurately representative of the substance being studied. The most difficult aspect of this preparation is that no criteria exist for checking morphology in the 0.001 - 1.0 μ range. The only check that can be made is to vary the technique of preparation over a wide range and observe the factors apparently responsible for changes in the specimen(4).

Satisfactory specimens for the electron microscope must meet certain conditions. (1) They must be sufficiently thin to allow the electron beam to penetrate the specimen without appreciable loss of energy. Electrons with a reduced energy exhibit a longer wave length and the chromatic aberration present in the lenses produces a loss in resolution. Loss of energy by the electrons implies that energy is gained by the specimen with consequent increase in temperature which may cause a change in structure(5). The degree or nature of such a change depends on the chemical composition and physical form of the specimen. If large particles are involved the charge adsorbed and the repulsive forces produced may be sufficient to rupture the thin supporting membrane on which the particles rest. (2) The specimen must exhibit sufficient contrast. Since contrast is produced by differences in scattering power or density of various portions of the specimen this is not normally much of a problem when inorganic particles are being studied. (3) The high vacuum of the electron microscope column precludes the observation of wet or live tissue or any other volatile material. In preparation of specimens any artifacts which could be caused by drying the sample, such as surface tension effects, changing pH, etc. should be recognized.

The specimen is usually mounted on an extremely thin film of Formvar(R) or collodion. The Formvar or collodion films being of the order of 100 A° thick are quite transparent to the electron beam. Recently some other substrate media such as evaporated films of silicon dioxide, silicon monoxide, and carbon have come into use and appear to hold much promise. All of these films are supported on 1/8 inch circles of 200 mesh copper grid and observations are made through the open areas between the wires.

The problem of transfering the specimen to the previously prepared film on its supporting grid may vary with each sample. If a sample of airborne dust has been collected in water by an impingement technique a drop of this dust suspension may be placed on the grid and the water allowed to evaporate. In this process, however, the dust will be found to deposit in

in rings as the evaporation proceeds and therefore the fields observed may not be truly representative of the original suspension.

The filmed grids may be placed in the thermal precipitator or the electrostatic precipitator and the air-borne particles deposited directly on the film. In each of these techniques, however, the deposition of the particles may be affected by their size and the sample obtained may not be representative[6]. It is apparent that a knowledge of the efficiency and operating principles of the sampling instrument is essential as well as an understanding of the electron microscope.

A technique which has been used successfully is that of collecting the sample on a Millipore filter and then dissolving this filter with a diffusion method using ethyl acetate and allowing the particles to settle gently onto a grid prepared with a Formvar film[7,8]. The Formvar film is not soluble in this solvent. Many other techniques are described in the current literature[9,10] and older methods of specimen preparation are discussed in standard texts on electron microscopy[11,12].

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# SOLVENT ANALYSIS

## Introduction

Solvent analysis is a very complex and difficult operation. An extremely wide variety of solvents is in industrial use. No systematic procedure is available for the exact identification and determination of these substances. All the techniques of organic analysis are required. Because of the many types of required tests a sample of at least 500 ml is highly desirable. In the following discussion certain general principles will be given which are applicable to some common solvents. Field methods, using the combustible gas indicator and the halogen detector, and the principles of vapor sampling and analysis have been discussed in other lectures. This discussion deals with laboratory analysis of bulk solvent samples.

## Solvents Used in Various Industries

Preliminary information about the sample is of the utmost importance. No effort should be spared in consulting the chemical and industrial literature in order to determine what classes of solvents may be expected. One of the difficulties in this work is the fact that many plants do not know the chemical composition of the organic solvents they use. Indeed, even the manufacturer may not know the exact composition because of the possible variations in his raw materials. It is not unusual for the composition of solvent mixtures to vary from batch to batch. However, an effort must be made to get an approximate idea of what substances may be expected in the solvent. In Table 1 a limited listing is given of the uses of various types of solvents in industry.

## Vapor Hazard of Various Solvents

Solvents vary widely in toxicity. The exact identification of the less toxic members of a solvent mixture is not as important as the identification and determination of the more toxic components. Table 2 presents a listing of organic gases and vapors arranged in order of decreasing vapor hazard. From the general information available regarding the sample, the analyst may determine which of the substances likely to be present may be important from the toxicity viewpoint. Volatility is a factor which effects the toxic vapor hazard. It may be seen that a relatively high-boiling non-volatile solvent would be less dangerous than a volatile one of equal toxicity. Substances boiling above 150 °C are ordinarily not considered to be a serious hazard for this reason.

## Preliminary Operations and Identification Tests

The methods of analysis used by manufacturers and consumers should be consulted. This gives one a rough outline of what is to be expected in spite of the fact that many of these tests are based on physical properties and performance, such as, density, flash point, boiling range, solvent properties, viscosity, etc. Methods of analysis for paint thinners and lacquer solvents are well covered by Gardner[1] and Watts.[2] Much information on industrial solvents is given by Mellan[3] and Scheflan.[4] The industrial hygiene viewpoint is presented in the texts by Jacobs[5] and Goldman and Jacobs.[6]

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Table 1. Solvents Used in Various Industries

Antifreeze

- Ethanol
- Isopropyl alcohol
- Methanol
- Ethylene glycol
- Glycerol

Automotive (exclusive of antifreeze)

- Chlorinated hydrocarbons
- Ether-alcohols
- Plasticizers
- Polyether alcohols

Dry Cleaning

- Chlorinated hydrocarbons
- Naphtha

Paint, Varnish, Lacquer, Ink

- Alcohols
- Coal tar hydrocarbons
- Esters
- Ether alcohols
- Ketones
- Plasticizers
- Turpentine

Petroleum

Solvent dewaxing

- Acetone
- Benzene
- Ethylene dichloride
- Methyl ethyl ketone
- Propane

Pharmaceutical

- Alcohols
- Chlorinated hydrocarbons
- Ethers
- Ketones

Rayon

- Acetone
- Carbon disulfide
- Esters
- Ethers

Textiles (other than rayon)

- Glycol derivatives
- Glycols
- Hydrocarbons
- Lacquer solvents
- Pine tar derivatives

Petroleum

Selective Solvents

- Benzene-sulfur dioxide
- Cresylic acid
- Dichloroethyl ether
- Furfural
- Nitrobenzene
- Phenol
- Propane

Table 2. Organic Liquids Arranged in Order of Vapor Hazard

| Substance | Vapor (a) Hazard | Threshold[b] Limit PPM by Vol. | B. P.[c] °C |
|---|---|---|---|
| Butyl amine | 34,000 | 5 | 77.8 |
| Carbon disulfide | 23,000 | 20 | 46.3 |
| Tetranitromethane | 15,800 | 1 | 126 |
| Diethylamine | 12,600 | 25 | 55.5 |
| Acrylonitrile | 11,200 | 20 | 78-9 |
| Methyl formate | 7,600 | 100 | 32 |
| Allyl alcohol | 6,700 | 5 | 96.6 |
| Carbon tetrachloride | 5,800 | 25 | 76.8 |
| Benzene (benzol) | 3,650 | 35 | 80.1 |
| Ethyl formate | 3,160 | 100 | 54 |
| Ethyl bromide | 3,030 | 200 | 38.4 |
| 1,1-Dichloroethane | 2,900 | 100 | 57.3 |
| Chloroform | 2,500 | 100 | 61.2 |
| Pyridine | 2,500 | 10 | 115-6 |
| 1,2-Dichloroethane | 2,040 | 200 | 47.8 |
| Acetic acid | 1,970 | 10 | 118.1 |
| Ethylene chlorohydrin | 1,900 | 5 | 128.8 |
| Ethylene diamine | 1,710 | 10 | 55.5 |
| 1,1,2,2-Tetrachloroethane | 1,684 | 5 | 146.3 |
| Benzyl chloride | 1,580 | 1 | 179.4 |
| Ethyl ether | 1,380 | 400 | 34.6 |
| Methyl acetate | 1,380 | 200 | 57.1 |
| Acetic anhydride | 1,340 | 5 | 139.6 |
| Aniline | 1,320 | 5 | 184.4 |
| Methylene chloride | 1,080 | 500 | 40-1 |
| Ethylene dichloride (1,2-dichloroethane) | 1,050 | 100 | 83.7 |
| Propylene dichloride (1,2-dichloropropane) | 910 | 75 | 96.8 |
| Dimethylaniline (N-dimethylaniline) | 870 | 5 | 193 |
| Methyl alcohol | 820 | 200 | 64.7 |
| Ethylene dibromide (1,2-dibromoethane) | 740 | 25 | 131.5 |

(a) Ratio (ppm/ppm) of equilibrium vapor concentration at 25 °C to the Threshold Limit Value, computed from vapor pressure data in references (3,8).

(b) From A.C.G.I.H. Threshold Limit Values for 1956.

(c) Boiling point at 760 mm Hg. from references (3,8,9). Observed boiling points in mixtures may be lower due to formation of azeotropes.

| Substance | Vapor[a] Hazard | Threshold[b] Limit PPM by Vol. | B. P.[c] °C |
|---|---|---|---|
| Methyl cellosolve (methoxyethanol) | 630 | 25 | 124-5 |
| Methylal (dimethoxymethane) | 526 | 1000 | 42-3 |
| Butanone (methyl ethyl ketone) | 516 | 250 | 79.6 |
| Trichloroethylene | 493 | 200 | 87.2 |
| Dioxane (diethylene dioxide) | 490 | 100 | 101 |
| Nitrobenzene | 474 | 1 | 210.9 |
| Pentane | 450 | 1000 | 36.3 |
| 2-Nitropropane | 447 | 50 | 120.3 |
| Nitromethane | 434 | 100 | 101 |
| Propyl ether (isopropyl ether) | 420 | 500 | 69 |
| Hexane (n-hexane) | 410 | 500 | 69 |
| Propyl acetate | 390 | 200 | 88.4 |
| Methyl chloroform (1,1,1-trichloroethane) | 329 | 500 | 74.1 |
| Cyclohexane | 320 | 400 | 80-1 |
| Ethyl acetate | 303 | 400 | 77.1 |
| Acetone | 290 | 1000 | 56.5 |
| Methyl isobutyl carbinol (methyl amyl alcohol) | 263 | 25 | 131.8 |
| Nitroethane | 263 | 100 | 114.8 |
| Mesityl oxide | 245 | 50 | 130 |
| Amyl alcohol (isoamyl alcohol) | 220 | 100 | 132 |
| Chlorobenzene (monochlorobenzene) | 210 | 75 | 132.1 |
| Cresol (all isomers) | 184 | 5 | 191-203 |
| Toluene | 184 | 200 | 110.8 |
| Gasoline | 176 | 500 | 60-120 |
| Methyl cellosolve acetate (ethylene glycol monomethyl ether acetate) | 163 | 25 | 144.5 |
| Propyl alcohol (isopropyl alcohol) | 140 | 400 | 82.5 |
| Phenol | 132 | 5 | 181.4 |
| Heptane (n-heptane) | 121 | 500 | 98.4 |
| Methyl cyclohexane | 121 | 500 | 101 |
| Perchloroethylene (tetrachloroethylene) | 118 | 200 | 120.8 |
| Pentanone (methyl propyl ketone) | 112 | 200 | 95 |
| Dichloroethyl ether | 96 | 15 | 178.5 |
| Naphtha (petroleum) | 95 | 500 | 80-130 |
| Hexone (methyl isobutyl ketone) | 93 | 100 | 117-9 |
| Butyl alcohol (n-butanol) | 92 | 100 | 117 |
| Butyl acetate (n-butyl acetate) | 79 | 200 | 127 |
| Ethyl alcohol (ethanol) | 76 | 1000 | 78.4 |
| p-Tertiary butyl toluene | 72 | 10 | 200 |
| Diisobutyl ketone | 71 | 50 | 168.1 |
| Naphtha (coal tar) | 66 | 200 | 110-60 |
| o-Toluidine | 66 | 5 | 199.7 |
| Turpentine | 66 | 100 | 120-80 |

| Substance | Vapor[a] Hazard | Threshold[b] Limit PPM by Vol. | B. P.[c] °C |
|---|---|---|---|
| Ethyl benzene | 63 | 200 | 136.2 |
| Cyclohexanone | 62 | 100 | 155-6 |
| Styrene Monomer (phenyl ethylene) | 57 | 200 | 145-6 |
| Nitrotoluene | 55 | 5 | 222-38 |
| Hexanone (methyl butyl ketone) | 50 | 100 | 128 |
| Xylene | 50 | 200 | 139-44 |
| Methylcyclohexanone | 47 | 100 | 162-70 |
| Cellosolve acetate (hydroxyethyl acetate) | 42 | 100 | 156.3 |
| Cellosolve (2-ethoxyethanol) | 41 | 200 | 135.1 |
| Octane | 34 | 500 | 99-125 |
| Amyl acetate | 30 | 200 | 142.0 |
| Isophorone | 26 | 25 | 215 |
| Cyclohexanol | 18 | 100 | 160-1 |
| Butyl cellosolve (2-butoxyethanol) | 13 | 200 | 171.2 |
| Phenylhydrazine | 11 | 5 | 243.5 |
| o-Dichlorobenzene | 9 | 50 | 179 |
| Diacetone alcohol (4-hydroxy-4-methyl-pentanone-2) | 8 | 50 | 167.9 |
| Methylcyclohexanol | 7 | 100 | 165-75 |
| Stoddard solvent | 7 | 500 | 150-90 |
| Furfuryl alcohol | 4 | 200 | 170 |

Solvents containing dissolved or suspended solids, such as lacquer thinners or paints, may be subjected to a preliminary steam or vacuum distillation to separate the solvents. This operation may introduce additional complications such as the separation of the sample into a water soluble and insoluble portion. However, it is essential for such materials.

Several miscibility tests may be rapidly and conveniently performed. These are carried out by pipetting portions of solvent into stoppered graduates. To these are added equal volumes of test reagent and the liquids are mixed gently by inversion several times. In order to avoid emulsification, these tests are carried out without violent shaking. The volumes of the two layers should be carefully noted so that the percentage of miscibility may be calculated.

If the first graduate is dried and weighed, the specific gravity may be quickly determined from the gain in weight. Add an equal volume of water. Complete miscibility excludes hydrocarbons, halogenated hydrocarbons, and many of the long chain acids, alcohols, phenols, secondary nitro compounds, neutral compounds, etc.

In the second stoppered graduate the miscibility with an equal volume of concentrated hydrochloric acid (or 4:1 $H_2SO_4$) may be determined. In addition to the solvents which dissolve in water, others, such as alcohols, esters, aldehydes, ketones, some unsaturated compounds, anhydrides, ethers, and quinones may be expected to dissolve in the acid. Only inert compounds such as hydrocarbons and halogenated hydrocarbons do not dissolve.

Solubility in dimethyl sulfate may be used to distinguish between the aliphatic and aromatic hydrocarbons, as all ordinary solvents are miscible with this reagent except the aliphatic hydrocarbons. Chlorinated hydrocarbons do not give a clear cut picture in this test since they are partially miscible. In their absence, the difference between the residue immiscible in dimethyl sulfate, and the residue immiscible in strong acid may be taken as the approximate aromatic content. If this value exceeds 25%, the sample should be diluted with pure paraffinic hydrocarbons and the dimethyl sulfate test repeated, as otherwise the results may be too high.

From the three miscibility values the approximate classification of the solvent may be made according to solubility classes. If significant amounts of solvent fall into different classes it is generally advisable to treat a large fraction of the sample with appropriate solutions so that it may be separated into the various groups. Unfortunately, clear cut separations may not be obtained for substances such as halogenated hydrocarbons. However, solubility separations are desirable because of the lack of specificity of many of the procedures. For example, in a fractionation column many azeotropic mixtures can exist and the interpretation of the boiling point is unclear unless a relatively simple mixture is being distilled. Similar considerations apply to other methods such as spectroscopy, index of refraction, etc.

Another simple preliminary test which should be carried out is the Beilstein test for halogenated hydrocarbons. A drop of solvent is placed on a small loop of copper wire and heated in a flame. A green flame indicates halogenated hydrocarbons. If these are present in sufficient quantities the solvent may not be flammable. A smoky flame would indicate a high aromatic content in many cases.

The odor of the solvent is a simple test which may give an important lead as to the presence of amines, nitro compounds, esters, aldehydes, etc. A more complete collection of organic classification tests may be found in Shriner and Fuson[7].

## Analysis of Hydrocarbon Mixtures

Aromatic, aliphatic, and halogenated hydrocarbons may be determined by fractionating the inert oil residue obtained by treating a portion of the sample with 4:1 sulfuric acid. A suitable fractionating column consists of a well-insulated tube, 1/2 inch in inside diameter and 3 feet long, packed with glass helices, with an accurate thermometer in the top vapor space connection to a special condenser. The sample is placed in a flask at the bottom and is boiled using a Glascol heating jacket with a Variac control. Most of the condensed vapor is returned from the top of the packed column as reflux. A small portion, of the order of a third to a tenth of the condensate is withdrawn as distillate. Such a column effects successive condensations and reevaporations in the streams of rising vapor and descending liquid and is the equivalent of many simple distillations. In this way a separation of closely boiling components in mixtures may be obtained.

Fractionation

Several hundred milliliters of the sample which has been previously dried with anhydrous sodium sulfate or potassium carbonate and decanted is placed in the still flask. The heat is regulated to give the highest distillation rate without flooding the column. The proper operation of the column may be checked at any time by setting the condenser stopcock at total reflux. If the top temperature drops only slightly within a few minutes, the distillate withdrawal rate is not excessive. The distillate is received in a previously weighed glass-stoppered graduate.

A running plot is kept of the cumulative volume distilled (abscissa) versus the temperature at the top of the column (ordinate). This gives the boiling point curve of the sample. The theoretical distillation curve for a mixture of substances is a series of steps. Each horizontal plateau represents the pure component being distilled at the time. When this has been completely removed the temperature rises steeply until it reaches the boiling point of the next component. The best way of carrying out the distillation is to study the running plot of the temperature versus the volume distilled and to change receivers during the steep temperature increases. Thus, the receiving cylinders will each contain a single pure component. In the case of petroleum distillates, so many compounds are present that the individual steps are not resolved, giving a gradually rising plot. In such a case, cuts are made at 95 °C, and 125 °C, for determination of benzene, toluene, and xylene as described below.

Additional tests. During the course of the distillation other tests may be made, such as a Beilstein test in the event that the presence of chlorinated hydrocarbons are suspected, or an index of refraction and dispersion measurements. These may be made on a single drop of distillate. A sudden change is an indication that the end of that component has been reached and indicates that the cut be taken.

At the conclusion of the distillation the different cuts in graduated cylinders may be weighed. From the volumes, the densities may be computed. Other tests, such as index of refraction may be carried out. Identification of each component is based on the boiling point, density, index of refraction and dispersion, miscibility with dimethyl sulfate, Beilstein test, etc. of the various cuts.

Determination of Aromatics in Hydrocarbon Distillates

The most convenient way of determining aromatics is by index of refraction and dispersion measurements. Specific dispersion, which is the index of refraction for the F (hydrogen) line minus the index of refraction for the C (hydrogen) line, divided by the density, is the most direct measure of aromatic content. If a cut boiling below 95 °C is being examined only benzene may be expected. This has a specific dispersion of 0.0190, whereas the specific dispersion for paraffins and cyclic aliphatics in the boiling range below 95 °C extends from 0.0096 to 0.0100. By means of a simple proportion the aromatic content may be estimated from the dispersion data. If a cut boiling between 95 and 125 °C is being examined only toluene

may be expected as the aromatic. The specific dispersion of toluene is 0.0185 whereas the non-aromatics in this fraction range between 0.0096 and 0.0101. The aromatic content of the cut between 125 and 150 °C may be estimated using the specific dispersion for aromatics of 0.0174 to 0.0181 for ethyl benzene and the xylenes as compared to 0.0095 to 0.0099 for the non-aromatics. This method is relatively simple and sufficiently accurate in most cases. It requires only reading the instrumental value from the dispersion compensator. The conversion of this number to dispersion is accomplished using tables provided with the refractometer.

The index of refraction may also be used to determine aromatic content. However, this calculation requires an estimate of the refractive indices of the paraffins and naphthenes (cyclic aliphatics). These refractive indices have a greater variation than the dispersion and are thus more uncertain in unknown mixtures.

Ultraviolet spectrophotometry may also be utilized to determine aromatics which absorb intensely in this region. The peak absorption for benzene occurs at 255 millimicrons, for toluene at 262, for ethyl benzene at 262, for o-xylene at 263, for m-xylene at 265, and for p-xylene at 274 millimicrons. A suitable solvent for these measurements is a paraffin hydrocarbon such as isoctane. For 1 cm cells the working range extends up to concentrations of a few tenths of a gram of aromatic hydrocarbon per liter. Standards may be run using the pure substances in the usual manner. As some of these peaks are quite sharp the resolution of the spectrophotometer will affect the standardization factor considerably.

It is also possible to use the spectrophotometric method on a mixture which has not been distilled and which may contain all of these aromatics. The sample is read at 5 wavelengths, as are each of the pure standards. By means of simultaneous equations the composition of aromatics may be obtained Wavelengths which have been used are 255.0, 265.0, 268.5, 272.5 and 274.5 millimicrons. The mathematical handling of these equations is somewhat difficult. Furthermore, from the toxicity viewpoint the more volatile aromatics are of the greatest importance. Thus, ordinarily, fractionation is the better procedure.

Aromatic content may be determined on the distillate by dimethyl sulfate miscibility in the same manner as described under preliminary tests.

## Analysis of Water and Acid miscible Fractions

Many solvents, such as lacquer and paint thinners, may contain little or no hydrocarbon. A mixture of alcohols (ROH), esters ($RCOOR^1$), ketones ($RCOR^1$), and ethers ($ROR^1$), may be expected. These may be partially or completely miscible with water.

The water extract may be fractionated as described above for hydrocarbon mixtures. A boiling point curve, temperature versus cumulative distillate volume, should be plotted as before. One of the more toxic substances which should be sought is methanol (B.P, 64.7 °C). If there is any sign of a plateau at this temperature the fraction may be analyzed for methanol, using Schiff reagent or chromotropic acid.(5,6) It should be kept in mind

that many azeotropic mixtures may be formed which boil at temperatures differing from those of the pure components. Thus, the absence of a plateau at the boiling point of methanol does not exclude this substance. If a pure substance, which might reasonably be expected, cannot be found, tables of azeotropic boiling points[3] should be consulted.

The analysis of lacquer solvents is a rather complex procedure which has been discussed more fully elsewhere.[1,2] Many specific tests may be made for different components as described in the laboratory exercise on solvents. Esters may be determined by saponification with standardized, alcoholic potassium hydroxide under total reflux for an hour, after which the unconsumed potassium hydroxide is determined by titration. The most valuable methods for this analysis are based upon the distillation curve and auxiliary physical data, followed by specific chemical tests for type compounds and individual substances.

Tests may also be made in the same manner upon the water insoluble-acid soluble fraction. In such a case 4:1 sulfuric acid should be used rather than hydrochloric acid because of its non-volatility. If it seems appropriate, the acid may be neutralized with sodium hydroxide. This will salt out the organic solvents, which will separate from the aqueous portion. It should be borne in mind that this treatment may serve to hydrolyze the esters and change other components in the solvent. This fraction may then be treated as above.

It is evident that solvent analysis involves a good deal of detective work. When the composition of the solvent is reasonably well known from the industry, the analysis may be relatively simple. In other cases an exact analysis is not required as all the components may have a relatively low order of toxicity. The analyst should make certain that the most toxic substances listed in Table 2 are absent.

## Instrumental Methods of Analysis

Effective instrumental methods available for solvent analysis include, in order of increasing expense, gas chromatography, infrared spectroscopy, and mass spectrometry. These generally have much greater sensitivity than other physical and chemical methods, and are useful in the identification of individual substances. However, in most cases when many components are present, interpretation may be difficult. These methods are best applied in conjunction with other physical and chemical methods to relatively simple fractions. Complete reference data are not yet available in the literature, and variation from instrument to instrument may be expected for the gas chromatograph and the mass spectrometer. The gas chromatograph has recently become popular because it is the least expensive of the three methods, and the development of considerable information may be expected in the near future. This method is based upon a combination of the volatility and chemical properties of the substance, and can achieve separations equivalent to 1000 theoretical plates (simple distillations), as compared to about 100 for the ordinary fractionating column.

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# SPECTROPHOTOMETRY

## Introduction

Colorimetric methods are very important in industrial hygiene chemistry for determination of the concentrations of trace constituents. Colorimetric determinations, with an error of a few percent, are often the most accurate procedures available for these very small quantities. They may also be the most sensitive if sufficient sample is available. While the spectrographic methods have the highest absolute sensitivity, their requirements of relatively small sample portions limit their concentration sensitivities. Colorimetric methods are generally much more rapid than the more laborious gravimetric or titrimetric procedures.

Colorimetric procedures for various determinations are presented in other sections of this manual. These involve the preliminary solution and ashing of the samples, followed by various concentration steps such as extraction or distillation to remove interfering substances. A wide range of reagents may then be used to produce colors with various constituents. This discussion will deal with the final quantitative measurement of color and its conversion to quantity of constituent. The theory of colorimetry will be examined, various instruments used for this purpose will be discussed; the types of errors to be expected, and methods of handling standardization data and calculations will be outlined.

The ideal colorimetric reagent should have the following qualities:

Stability
Stoichiometric reaction
Rapid color development
Transparency in spectral region involved
Selective or specific reaction
Freedom from interference
High capacity
Solubility in satisfactory, available solvents

Methods of color development include:

Redox
Complex formation
Diazo and coupling reactions
Condensation and addition
Salt formation
Chromophoric changes in valence
Substitution

The ideal color system has the following properties:

Stability
High intensity

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Minor variation with pH, temperature, and other factors
Absorbance in appropriate spectral region (475-675 mμ for visual determination)
Solubility of colored product
Conformity to Beer's law

## Theory of Spectrophotometry

### The Electromagnetic Spectrum

Visible light occupies a relatively small region in the known range of radiation. Cosmic rays have a wave length of less than 0.001 A (the angstrom unit A = $10^{-8}$ centimeters). Nuclear gamma radiation extends from 0.001 to 0.1 A and X-rays from 0.1 to 100 A. The ultraviolet region extends from 100 to 4000 A, followed by the visible region from 4000 A (violet) to 7500 A (red). The near infrared region extends from 7500 A to 250,000 A (0.75 to 25 microns), while the far infrared region extends from 25 to 1000 microns. Longer wave lengths occur in the microwave and radio wave regions which extend to several hundred meters in wave lengths From this it is evident that the visible region represents only a very small portion of the known range of radiation of the electromagnetic spectrum.

### Absorption of High-Energy Radiation

In the interaction of radiation and matter some or all of the energy is absorbed. A fundamental principle of such interaction is that the radiation behaves like packets of energy (photons) rather than like waves. The energy of each photon is inversely proportional to the wave length of the radiation. Many types of interaction between photons and matter occur simultaneously. However, certain interactions predominate for each energy or wave length range. The most important process for absorbing the energy of very high energy photons (cosmic rays and high energy gamma rays) is by collision with atomic nuclei resulting in disintegration or ejection of particles, and transmutation to another element. In the next lower energy range photons are absorbed in the vicinity of atomic nuclei with the creation of electron-positron pairs, each particle traveling in opposite directions. In the low energy gamma range and the X-ray range most of the energy may be absorbed by collision of the photons with electrons. Energy and momentum are conserved in such a collision which produces a lower energy photon (longer wave length) and a high speed electron. This process is known as the Compton effect. In the lower X-ray and ultraviolet regions the most important energy absorbing process is the photoelectric effect in which the photons are absorbed in the vicinity of atomic nuclei, and their energy is transmitted to electrons which are ejected.

### Quantum Absorption of Low-Energy Radiation

Another process in the ultraviolet and visible region, which is the basis of spectroscopy, is the absorption of photons with energies insufficient to eject electrons from their atoms. The quantum theory is a fundamental principle which states that electrons can exist only in certain orbits which have fixed energy levels for each element. If a photon has the exact energy required to raise the electron to one of its higher energy orbits the photon

will be strongly absorbed. Photons of other energies are not absorbed. Thus the absorption spectrum of a gas exhibits sharp bands of absorption in certain narrow wave length ranges, corresponding to differences in electronic energy levels of the atoms present.

The important energy absorbing processes for spectrophotometry occur in the ultraviolet, visible and infrared regions. All relate to molecular energy levels rather than atomic energy levels. According to the quantum theory molecules also have certain fixed possible energy levels. If a photon has the exact energy required to raise the molecule from its normal low energy state to one of the higher energy levels it will be strongly absorbed. The aromatic benzene ring has a characteristic absorption pattern in the ultraviolet region. This pattern is altered somewhat as various substituent groups are added to the ring. In the visible light region certain chromophoric groups such as N = N absorb strongly, producing colored solutions. The infrared region is particularly rich in absorption bands corresponding to the energies of various stretching and vibrating elemental bonds. In the far infrared and microwave region the energy levels corresponding to molecular rotations and vibrations become the most important ones. Such absorption patterns are invaluable for determining molecular structure.

Quantitative Relations in Spectrophotometry

The basic law of spectrophotometry is that for light of a single wave length (monochromatic) each molecule of colored solute absorbs the same fraction of the radiant energy that falls upon it. This may be expressed in differential form by equation (1):

$$(1) \qquad -\frac{dI}{I} = KCdL$$

and in integrated form by equation (2):

$$(2) \qquad \ln\left(\frac{I_o}{I}\right) = 2.303 \log_{10}\left(\frac{I_o}{I}\right) = KCL.$$

In equation (1) the left side represents the fraction of light absorbed, the - dI being the light intensity absorbed and I being the total light intensity. On the right side, the product of the concentration C and the differential length of solution dL is proportional to the total number of absorbing molecules. The proportionality constant K represents the fractional absorption per unit concentration per unit length of absorbing path. In the integrated form, equation (2), $I_o$ represents the incident light intensity and I represents the emerging light intensity.

Optical absorbance is a convenient term defined as $\log_{10}(I_o/I)$. Many instruments have dial calibrations giving this value (synonymous with optical density) as well as percent transmittance. Absorbance and concentration are related linearly.

The specific extinction coefficient k is defined as the absorbance per gram per liter, per centimeter light path:

$$(3) \quad A = kCL$$

Where A is absorbance, C is concentration in grams per liter, and L is the cell length in centimeters.

The molecular extinction coefficient $\epsilon$ is defined as the absorbance per mole per liter, per centimeter light path:

$$(4) \quad A = \epsilon C'L$$

Where C' is concentration in moles per liter.

The relationships between the various coefficients are:

$$k = \epsilon / \text{molecular weight}$$

and $$k = K/2.303$$

Transmittance, T, is a commonly used term denoting the value of the fraction $I/I_o$. If the photometer gives readings in terms of transmittance, they may be converted to absorbance as follows:

$$(5) \quad A = 2 - \log_{10} \%T$$

Where %T is expressed as percentage (= 100T).

Transmittance values may be used directly if plotted on semi-log paper against concentration of absorbing species, giving a straight line of slope - kL:

$$(6) \quad \log_{10} T = - kCL$$

It can be seen from the definition that 100% transmittance corresponds to zero absorbance, while 10% transmittance corresponds to 1.0 absorbance.

The above equations present a more complete statement combining the earlier Bouguer's (Lambert's) Law, stating that each unit thickness of a light absorbing medium absorbs an equal fraction of radiant energy traversing it, and Beer's Law, which states that the fractional absorption of light by a thin layer of solution is directly proportional to the concentration of the absorbing solute.

Analytical Application of Beer's Law

In order to secure close compliance with Beer's law, dilute solutions should be used with monochromatic light. If a broad band of light of many wave lengths is used, it can be readily shown mathematically that although each particular wave length is absorbed according to Beer's Law, the sum of all the light energy in the band will not follow this relationship. Deviations from Beer's Law may also occur in more concentrated solutions due to association of the absorbing molecules into a different molecular species. If the ratio of the two species is not a constant in the concentration range being measured, then the total absorption, which would be the sum of the two

different species, will not conform to Beer's Law. Other deviations occur if the colorimetric reaction is not stoichiometric because of insufficient excess of colorimetric reagent or the presence of interfering substances.

In actual practice, a series of standard colors is prepared by applying the procedure to known amounts of the substance being determined. A plot is made of the determined absorbances versus the concentration or quantity of substance. If this plot exhibits a linear relationship, Beer's Law is followed. If such is the case it is more convenient in analyzing unknown samples to use a mathematical calculation to relate quantity of substance to absorbance values rather than a graph. The standardization factor is obtained from the graph of the standard sample series as the number of micrograms of substance required to produce an absorbance of exactly 1, when the plot is made with all values corrected by deducting the reagent blank. The corrected absorbance of the unknown multiplied by this standardization factor gives the micrograms of material being determined in the unknown sample. If Beer's Law is not followed the curved plot must be used for the conversion. This colorimetric determination is still perfectly valid although not as convenient.

## Colorimeters and Spectrophotometers

### Visual Colorimeters

Nessler tubes. The simplest type of color measurement is by visual comparison in matched test tubes or Nessler tubes. The visual spectral response curve is a bell-shaped pattern with a peak at 555 millimicrons. Intensity differences of 2% may be detected in the most favorable wave length range. Differences of 15% to 20% are detected at the extreme ends of the wave length range. This method has the disadvantage of requiring an extensive set of standard colors, which is doubly inconvenient if the colors are not stable.

Duboscq colorimeter. Another method of measurement is by means of a Duboscq colorimeter. This requires only a single standard color. The unknown is matched against the standard color by varying the path lengths. This is done by viewing the two colors downward through glass plungers which may be moved vertically into the two cups containing the colored solutions to give variable path lengths between the bottoms of the plungers and the bottoms of the cups. From equation (2) when the colors are matched $C_1L_1 = C_2L_2$. From the known standard $C_1$ and the known values of $L_1$ and $L_2$ the unknown $C_2$ may be calculated.

Wedge color comparator. In another type of visual colorimeter the balance is obtained by means of a glass wedge of a neutral gray tint. Colored filters are used to select a narrow wave length band of light. When the balance is obtained the values of transmittance or absorbance may be obtained from the wedge calibration.

### Photoelectric Colorimeters

Klett-Summerson. In a more advanced type of colorimeter the comparison is made with photoelectric cells rather than with the eye. The Klett-Summerson colorimeter utilizes a 100 watt projection lamp, condenser and colored glass

filter as a light source. Half of the beam passes through a mechanical balancing diaphragm to a reference photocell. The other half passes through the sample cell to the sample photocell. Barrier layer type photocells are used so that no power supply is necessary for this portion of the circuit. The output of the sample photocell is balanced on a calibrated potentiometer against the output of the reference photocell using a sensitive galvanometer to give the null point. The potentiometer is calibrated directly in units proportional to absorbance. Variations in line voltage produce light intensity fluctuations but these are cancelled out by the electrical balancing system. The mechanical balancing diaphragm is adjusted to give a zero reading with the reference material in the sample cell.

Monochromatic sources. Filters are inexpensive and are generally made more selective by combining two or more colored glass plates or dyes. A newer type is based upon interference effects and is much sharper in wave length band characteristics but also considerably more expensive. Interference filters are used in the Bausch & Lomb monochromatic colorimeter. Readings are obtained using a mirror galvanometer giving a cross hair spotlight beam on a glass scale.

It has been mentioned above that conformance to Beer's Law requires truly monochromatic light. More elaborate instruments provide a monochromatic beam by the use of various prism or grating systems in conjunction with mirrors and slits. Diffraction grating instruments have a uniform dispersion of light at various wave lengths. Prism type instruments condense the colors in the red region. Glass systems do not transmit much below 400 millimicrons. Quartz prisms and cells are suitable down to 185 millimicrons.

Light detectors. The best light detecting devices are photomultiplier tubes and blue sensitive photocells in the ultraviolet and blue end of the scale and red sensitive photocells at the red end. A lead sulfide detector is most sensitive in the lower portion of the near infrared region. Infrared instruments may use a red hot rod of carborundum (Globar) or a Nernst glower as a light source and a thermopile or bolometer (resistance thermometer) device to measure the heat liberated in the detector by the absorption of the radiation.

Spectrophotometers

Beckman DU. In the Beckman DU spectrophotometer a quartz prism silvered on one surface (Littrow prism) is used to disperse the light into a spectrum and only a narrow portion is selected by means of a slit. The wave length adjustment is a cam device which rotates an aluminum surfaced, concave mirror moving the spectrum past the slit. Two photocells are used. A blue-sensitive cesium-antimony phototube is used below 620 mμ (in conjunction with a hydrogen lamp light source below 350 millimicrons), and a red-sensitive cesium-oxide photocell is used above 620 mμ. The photocell produces a voltage signal across a very high resistance in series with the photocell. This voltage is balanced out by means of a calibrated potentiometer in series with it. The balance point is found by the plate current of the second tube in a two stage direct current, cascade amplified not showing any change (on a meter) when the shutter controlling the light is opened

or closed. This instrument has a very high wave length purity because the high sensitivity permits very narrow slits.

Coleman Model 10. In this spectrophotometer two diffraction gratings in series are used to obtain monochromatic light. The photocell output produces a voltage across a very high series resistor which is adjustable. This voltage is measured with an electrometer which draws no current.

Other instruments. More elaborate instruments use a split beam principle in which the monochromatic light is modulated by a chopper and then split into two beams by means of rotating mirrors. One beam passes through the reference cell and the other beam passes through the sample cell. The two beams are then combined and focused upon a single photocell the output of which is amplified. In the Perkin-Elmer Model 21 infrared spectrophotometer a servo system moves a calibrated comb into the reference beam until both beams are of equal intensity and the output of the alternating current amplifier is zero. The monochrometer is between the cells and the thermopile. In the Beckman DK spectrophotometer the output of the sample channel is balanced on a calibrated potentiometer against the output of the reference channel. Such systems involve complex circuitry and are rather expensive. However, they have the advantage of great speed when scanning a range of wave lengths, since all the tedious balancing is done automatically. The price of the more elaborate spectrophotometers may run as high as $15,000.

Wave length calibration. Spectrophotometers may be checked for wave length calibration by using a mercury lamp as the light source. The precisely known values of the sharp lines of the mercury spectrum can be compared with the wave length settings of the instrument.

## Errors in Spectrophotometry

The error of measurement of the amount of material in a sample in percent of the amount present is equal to $\triangle C/C$ where $\triangle C$ is the error in concentration. The relationship between the error $\triangle C$ and the error in absorbance may be computed by dividing the differentiated by the original form of equation (3) giving:

$$(7) \quad \frac{dC}{C} = \frac{dA}{A}$$

Equation (7) shows that for small errors the percentage error in concentration is equal to the percentage error in absorbance.

In general, the error in absorbance, dA, may vary with the value of A. The actual relationship may be obtained experimentally by running a series of replicate analyses at different concentrations. The deviation for each group of replicate samples may then be computed and plotted against the value of absorbance or concentration of the group. Such a relationship depends upon the type of analysis being conducted and the instruments used for the colorimetric measurements. If the relationship is known, the value of dA in terms of A is substituted in the expression $\frac{dA}{A}$, which gives the percentage error in the analysis. This fraction may be differentiated to obtain the optimal value of A for minimal percentage error. In the absence of experimental data certain assumptions may be made for the relationship between error and absorbance, as given below.

1. It has been shown experimentally[1] that the error in absorbance dA is a constant in the range of absorbance 0.0 to 1.0 using spectrophotometers such as the Beckman DU and the Cary Model 11. Important errors for which such a relationship would hold include (a) instrument zero point errors (b) cell blank errors and (c) sample blank errors. In such a case the error dA/A is a minimum for the highest possible value of A. Consequently most accurate work may be carried out at the higher end of the absorbance scale. The Beckman DU has an expanded scale covering the range of absorbance from 1.0 to 2.0. Use of this scale should further increase the accuracy.

2. A second type of relationship between error and concentration may be assumed in terms of transmittance rather than absorbance. It is commonly assumed that a constant error in transmittance is obtained. Such errors include (a) scale reading errors on linear T scales and (b) dark current drift. Equation (6) may be differentiated to give:

$$(8) \quad - \; kLdC = \frac{dT}{2.303T}$$

which when divided by equation (6) gives the relationship:

$$(9) \quad \frac{dC}{C} = \frac{dT}{2.303 \; T \log_{10} T}$$

The left side dC/C times 100 is the percentage error in the analysis. On the right side the numerator dT may be assumed to be a constant. Thus, the minimal error will occur when the denominator $T \log_{10} T$ reaches a maximum. In this expression it must be remembered that T is less than 1. By differentiating the expression $T \log_{10} T$ it may be readily shown that the maximal value occurs when T = 1/e or 36.8% (A = 0.434).

3. A third kind of assumption is sometimes made that dT, the error in transmittance, is proportional to the square root of T. This case assumes that the only error is due to electronic noise all of which is due to statistical variations in photon arrival at the cathode. By similar treatment it may be shown that the optimal transmittance is $1/e^2$ or an absorbance of about 1.

The above assumptions are of course purely arbitrary. The exact relationships are obtained experimentally as described above. For the better instruments it may be considered generally that the major errors occur in the chemical portions of the procedure rather than the instrumental. The use of the highest concentration within the range of the instrument will minimize blank and contamination errors. Such a practice is in agreement with the first assumption that a constant error in absorbance occurs, and differs somewhat from the commonly held second assumption that a constant error in transmittance occurs.

Standardization Data and Calculations

Colorimetric procedures are standardized by preparing a series of colors with known quantities of material. It is preferable not to use the zero standard as the photometer reference but rather to use some constant solution which reads close to it, such as distilled water in many instances. In this way a reagent blank is obtained which gives the analyst an important indication of

the levels of contamination and blank errors. In the usual case Beer's Law is followed and a zero and three or four known standards will suffice for the standardization. Four ways of treating standardization data will be described below. In all cases the quantity sought is the standardization factor representing the number of micrograms of material required to produce an absorbance of exactly 1. This factor is used to multiply the absorbances of unknown samples to convert to micrograms of material. If the photometer is calibrated with a transmittance scale these figures may be converted to absorbance by equation (5). As an illustration, actual data will be used for a cobalt analysis.

Treatment of Data

The simplest method of treating the data is to prepare a graph. The actual absorbance of each standard is plotted against the micrograms of cobalt and a straight line is drawn giving the best fit to the data. The intercept of this line with the abcissa (micrograms of cobalt) at zero ordinate (absorbance) gives the best value for the reagent blank. This may differ from that computed from the absorbance for the zero standard. If the relationship is seen to be a straight line, it is not necessary to use this graph for any further purposes as the conversion of unknown samples is done more accurately and conveniently by mathematical rather than by graphical methods. While the graphic method has the advantage of simplicity, three mathematical methods are given below which give more accurate results with little or no extra effort.

Greatest weight to highest values. A simple method of treating the data mathematically is illustrated in Table 1, in which the greatest weight is given to the highest values:

Table 1

Greatest Weight to Highest Values

| | γ Co | Absorbance | Corrected Abs. | γ Co Calc From A | Deviation |
|---|---|---|---|---|---|
| | 0 | 0.006 | 0 | --- | --- |
| | 5 | 0.174 | 0.168 | 4.86 | - 0.14 |
| | 10 | 0.344 | 0.338 | 9.77 | - 0.23 |
| | 25 | 0.883 | 0.877 | 25.36 | + 0.36 |
| sum | 40 | | 1.383 | sum | 0.73 |

Total γ/Total A = 40/1.383 = 28.92

The first two columns give the experimental data for micrograms of cobalt and absorbance. The corrected absorbance in the third column is obtained by deducting the blank absorbance of 0.006 from all the values. The sums of the first and third columns are recorded. Their ratio gives the weighted average

standardization of 28.92 micrograms of cobalt per absorbance unit. The fourth column gives the micrograms of cobalt calculated by multiplying corrected absorbances by 28.92. The deviations from the actual micrograms of cobalt are given in the fifth column. A measure of the error in this procedure is given by the sum of the deviations (disregarding algebraic signs), which is 0.73 micrograms of cobalt.

Equal weight to each value. A third method, based upon mathematically calculating the slope for each point, is illustrated in Table 2:

Table 2

Equal Weight to Each Point

| γ Co | Absorbance | Corrected Abs. | γ/A | γ Co Calc From A | Deviation |
|---|---|---|---|---|---|
| 0 | 0.006 | 0 | — | — | |
| 5 | 0.174 | 0.168 | 29.76 | 4.89 | - 0.11 |
| 10 | 0.344 | 0.338 | 29.07 | 10.01 | + 0.01 |
| 25 | 0.883 | 0.877 | 28.51 | 25.53 | + 0.53 |
| | | mean | 29.11 | sum | 0.65 |

The first three columns are identical with those given in Table 1. In the fourth column, the micrograms of cobalt are divided by the corrected absorbances. The working value is taken as the mean of the three figures, or 29.11 micrograms of cobalt per absorbance unit. The fifth column gives the micrograms of cobalt calculated by multiplying corrected absorbance by 29.11. The deviations from the actual micrograms of cobalt are given in the sixth column, the sum being 0.65 micrograms of cobalt. In other cases where blank errors are more important, this method may show higher deviations than the previous one.

Method of least squares. The most accurate method of handling the data is by using the method of least squares. This is not very laborious for a few values. In this method each value is given equal weight, whereas in the methods illustrated in Tables 1 and 2 the blank value is more heavily weighted, being used in calculating the corrected absorbances for all the other values. The method of least squares is illustrated in Table 3.

Table 3

Method of Least Squares

| | γ Co Y | Absorbance X | $X^2$ | XY | γ Co Calc From A | Deviation |
|---|---|---|---|---|---|---|
| | 0 | 0.006 | 0.000036 | | 0.165 | + 0.165 |
| | 5 | 0.174 | 0.030276 | 0.870 | 4.938 | - 0.062 |
| | 10 | 0.344 | 0.118336 | 3.440 | 9.768 | - 0.232 |
| | 25 | 0.883 | 0.779689 | 22.075 | 25.081 | + 0.081 |
| sum | 40 | 1.407 | 0.928337 | 26.385 | | sum 0.540 |
| mean | 10 | 0.3518 | | | | |
| correction | | | -0.494983 | -14.072 | | |
| deviation | | | 0.433354 | 12.313 | | |

$$Y = aX - b$$

$$a = \frac{12.313}{0.433354} = 28.41$$

$$b = a\bar{X} - \bar{Y} = 28.41 \times 0.3518 - 10 = -0.005$$

The first two columns give the experimental data as before. Column 1 is designated as Y and column 2 is designated as X. In the third column, $X^2$ is tabulated, and in the fourth column XY is tabulated. These calculations must be done on a calculating machine as a slide rule is not sufficiently accurate. The sums of all the columns are recorded and the means of the first two columns (the sums divided by 4) are calculated. The correction term in the third column is equal to the mean of X values multiplied by the sum of X (0.3518 x 1.407). The correction term for the fourth column is the mean X multiplied by the sum of Y (0.3518 x 40). These corrections are deducted from the corresponding $X^2$ and XY sums to give the deviations listed in the third and fourth columns. The relationships sought are the values of a and b in the equation Y = aX - b. The term b represents the best value for the reagent blank, while a is the standardization factor. The ratio of the two deviations, 12.313 - 0.433354 gives the best value of a, which is 28.41 micrograms of cobalt per absorbance unit. The value of b is then calculated from the equation, substituting the mean X and Y and the above value of a. This is found to be - 0.005 micrograms of cobalt. This may be regarded as zero for all practical purposes. Generally a positive value is to be expected. The fifth and sixth columns give the values of cobalt calculated from this relationship, and the deviations. The sum of the deviations without regard to signs is 0.54 micrograms of cobalt. It is evident that this is the smallest deviation sum of the three mathematical methods illustrated.

For accurate work, when a calculating machine is available, the method of least squares is the most desirable one to use. It is not too tedious for only a few values. The theory behind this method of calculation may be found in the standard statistical texts.

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# DITHIZONE DETERMINATIONS

## Introduction

### Special Problems of Trace Analysis

Trace analysis differs from conventional analytical procedures because of the requirements imposed by the extremely small quantities of substances being determined. Trace analysis methods require special cleaning of glassware, purified reagents and careful attention to small details. The requirements are so exacting that these procedures may be regarded as qualitatively different from the usual type of analysis. For example, in the analysis of urine for lead, the amount normally encountered is of the order of 2 or 3 micrograms. If an attempt were made to determine this small quantity by the usual procedures, such as hydrogen sulfide precipitation, no precipitate would be obtained. Evidently the greatest care is required in such an analysis because the quantity of lead may be less than that in the reagents used and contamination from a single speck of dust may introduce more lead than that present in the sample.

### Cleaning of Glassware

As an appreciable amount of glass is dissolved in these chemical procedures, only borosilicate glassware should be used to minimize this contamination. It is advisable to reserve a set of glassware exclusively for trace metal analysis. This glassware is cleaned in the usual manner so that it is visibly clean and then given a rinse with strong acid, such as 1 to 1 hydrochloric acid or concentrated nitric acid, followed by tap and distilled water. During the course of the analysis, in order to avoid contamination, pipettes and stoppers are never laid on the laboratory bench. Special racks with clothes pins have been found convenient for supporting pipettes and stoppers. If any appreciable period of time is to elapse during the course of analysis, open beakers are covered with watch glasses, parafilm, large sheets of filter paper or special rigid plastic covers.

### Preparation of Samples

The preliminary operations require dissolution of the sample followed by destruction of the organic matter. Dissolution is generally effected by heating with strong acid, such as sulfuric, hydrochloric, nitric or perchloric acids. Special techniques, such as fusion, are required for certain minerals and dusts. Ashing, or destruction of organic matter, is then performed by heating with strong oxidizing agents, such as nitric and perchloric acids, hydrogen peroxide, potassium permanganate, etc. Special precautions are required in certain determinations, such as those for mercury, arsenic, and antimony, to avoid volatilization of these elements. In general, a recommended ashing procedure is specified for each type of sample and for each determination.

### Separations

Because trace analysis involves very unfavorable ratios of other substances to the element being determined, separations are generally required.

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The extent of the separations necessary depend upon the type of determination. Only rarely is the determination so specific that some separation is not required.

Separation by precipitation, which is the classical method, is generally not applicable to trace analysis. The quantity of the analysis element is so small that it may be within the solubility range of even its most insoluble salts. Procedures have been developed in which another element is added to co-precipitate the element being determined. However, problems of supersaturation still remain, and, also, such procedures are generally time-consuming.

Distillation is a very effective means of separation which may be applied in certain special determinations such as those for arsenic and fluoride.

In recent years many exchange resins have been developed which may be used effectively to separate various ions by differential adsorption. These procedures involve varying conditions of acidity, concentration and temperature in order to accomplish these separations. This rapidly growing field may be expected to become increasingly important in the future.

The most generally applicable method for separations in trace metal analysis is by extraction as metallo-organic complexes with organic solvents. These procedures are very convenient and rapid and are very effective in separating even sub-microgram quantities of most elements. As will be seen later, dithizone is a unique reagent in that it may be used not only for determination but also for these preliminary separations.

Determinations

The usual gravimetric determination is not satisfactory for trace mineral analysis, because of the difficulty of weighing such small quantities on an analytical balance. In addition, the previously mentioned difficulties of supersaturation interfere in the preparation of these pure salts.

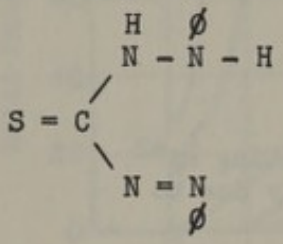
Titrimetric procedures applicable to trace metal analysis are rare. The extremely small quantities of material involved lead to difficulties because of the extremely dilute solutions required and the scarcity of end point indicators with the required degree of sensitivity.

Colorimetric determinations generally conducted with organic reagents, are most useful for trace metal analysis. Extremely high sensitivities may be obtained, approaching that of the spectrograph in some cases. Colored metallic complexes are frequently concentrated by extraction with organic solvents.

Other means of determination have also been applied, such as turbidimetric procedures for chloride and sulfate, and specific gravity and index of refraction methods for solvent analysis. These are all highly specialized for certain types of analyses, and are not applicable generally.

## Dithizone as a Reagent for Trace Metal Analysis

Dithizone is a chelating agent which reacts quantitatively with microgram quantities of a score of metals to produce intense colors, ranging from red through yellow, brown and violet. The color of dithizone itself is green. Dithizone and its metallic complexes are quite soluble in solvents such as chloroform and carbon tetrachloride. Many separations by extraction procedures can be accomplished. It is a versatile reagent and is used for the determination of many metals. The formula for dithizone is presented below as I. This reagent reacts with monovalent heavy metals to produce the keto form II, or the enol form III. The keto and enol formulae for the products with divalent metals are shown in IV and V, respectively. Dithizone is easily oxidized by many substances such as trivalent iron, alkaline copper solutions, halogens, nitrous acid, etc., producing a yellowish brown material as indicated by the structure in VI. This product is soluble in chloroform and carbon tetrachloride but is insoluble in water, whereas dithizone itself is soluble in alkaline aqueous solutions.



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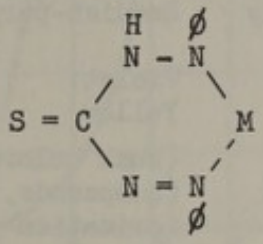

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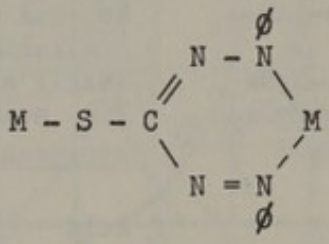

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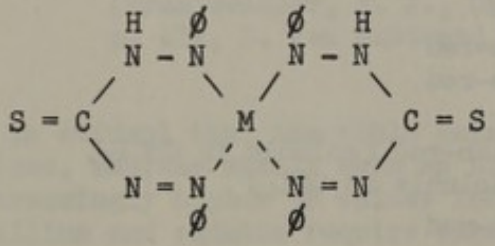

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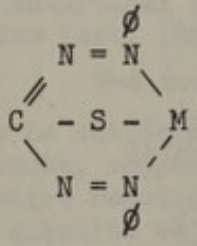

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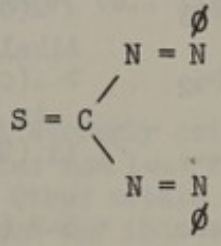

VI

Table 1. Metals reacting with dithizone (the enclosed elements yield dithizonates).

| | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | |
| Li | Be | | | | | | | | | | | B | C | N | O | F |
| Na | Mg | | | | | | | | | | | Al | Si | P | S | Cl |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br |
| Rb | Sr | Y | Zr | Cb | Mo | Ma | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I |
| Ca | Ba | La | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | - |
| — | Ra | Ac | Th | Pa | U | | | | | | | | | | | |

Table 2. The metal dithizonates

| Cation | Complex Form | pH for Extraction | Color in Solvent ($CCl_4$ unless otherwise stated) |
|---|---|---|---|
| Cu(II) | Keto-$CuDz_2$ | 0.1 N acid | Violet-red |
| Cu(II) | Enol-$CuDz_2$ | Alkaline | Greenish-brown |
| Cu(I) | Keto? | 0.1 N Acid | Red-brown to violet |
| Cu(I) | Enol? | Alkaline | Green-Brown (probably $Cu^{+2}$ compound) |
| Ag(I) | Keto-AgDz | Dil. acid | Yellow |
| Ag(I) | Enol-$Ag_2Dz$ | Alkaline | Violet (insoluble) |
| Au(I) | Keto-AuDz | Dil. acid | Dirty yellow (partially insoluble) |
| Au(I) | Enol-$Au_2Dz$ | Alkaline | Reddish insoluble |
| Zn(II) | Keto-$ZnDz_2$ | Neutral or weakly alkaline | Reddish-purple |
| Hg(II) | Enol-HgDz | Weakly alkaline | Violet |
| Hg(II) | Keto-$HgDz_2$ | Dil. acid | Yellow |
| Hg(I) | ? | Alkaline | (Weak colors simulating $Hg^{+2}$ (compounds, probably due to |
| Hg(I) | ? | Acid | (oxidation |
| In(III) | ..... | 5-6 | Red |
| Tl(I) | Keto-TlDz | 9-12 | Red |
| Tl(III) | ..... | 3-4 incomplete | Gold red |
| Pb(II) | Keto-$PbDz_2$ | 8-11.5 ($CHCl_3$) | Cherry-red |
| Bi(III) | Keto-$BiDz_3$ | > 2($CCl_4$) | Orange |
| Bi(III) | Enol | Alkaline | Orange-red |
| Sn(II) | Keto-$SnDz_2$ | > 4(Optimum 6-9 in $CCl_4$) | Purple-red |
| Mn(II) | ..... | 11 (Unstable) | Brownish-red (soluble in $CHCl_3$, insoluble in $CCl_4$) |
| Fe(II) | ..... | 6-7 ($CCl_4$) | Violet-red |
| Co(II) | Keto-$CoDz_2$ | 7-9 ($CCl_4$) | Violet |
| Ni(II) | Keto-$NiDz_2$ | Weakly alkaline | Dirty brown |
| Pd(II) | Enol-PdDz | Weakly Acid | Violet or brown<br>Red flocks, partially soluble in $CHCl_3$ |
| Pt(II) | ..... | Acid | Violet red in aq. phase, insoluble in $CHCl_3$; green in $CCl_4$ |
| Cd(II) | Keto-$CdDz_2$ | Strongly alkaline (5% NaOH) | Red |

Table 1 presents a periodic table arrangement showing the elements which react with dithizone. These elements are listed in more detail in Table 2, which gives the pH for the formation and extraction of each complex in carbon tetrachloride and the color in the solvent. It is apparent that,

because of the large number of elements which react with dithizone, it is necessary to provide some additional means of making these procedures specific.

Variables in Dithizone Complex Formation

Hydrogen ion concentration. pH is the most important variable for increasing the specificity of the reactions of dithizone with metals. Each metal has a certain pH range in which the reaction with dithizone is complete as shown in Figure 1, which presents equilibrium curves for some metal dithizonates in chloroform.

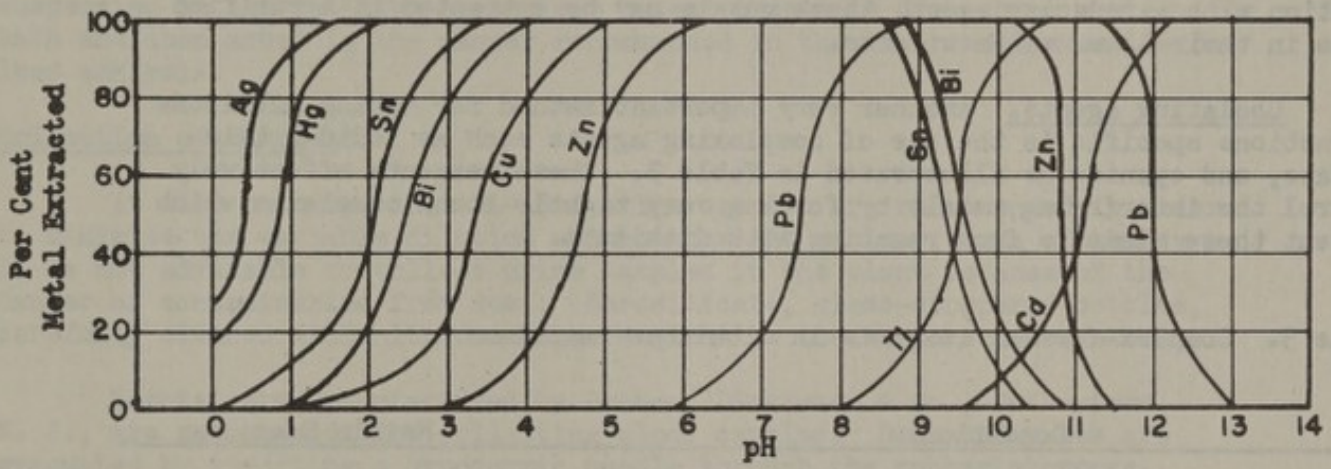


Fig. 1.-Equilibrium curves for some metal dithizonates in chloroform.

(From Welcher, F. J., Organic Analytical Reagents, Vol. III, p. 471, D. Van Nostrand Co., New York, 1947.)

It is evident that the noble metals may be extracted at strongly acid pH values, whereas metals such as tin, bismuth, copper, zinc and lead require increasingly higher pH values for effective reaction. Other metals such as thallium and cadmium require extremely alkaline solutions for this complex formation. Many metal complexes are stable in strong alkali. Thus, cadmium, cobalt, nickel, copper, mercury and silver complexes are stable in 1N sodium hydroxide. Other metals, however, are not extracted in strongly alkaline solution as indicated in Figure 1 for tin, bismuth, zinc and lead. This may be due to the amphoteric nature of these elements which exist in other ionic forms in strongly alkaline solution, such as the zincate and plumbite forms. The solvent also has an effect upon the reaction pH. Thus, while the values shown in Figure 1 are for chloroform as a solvent, the values which might be expected when carbon tetrachloride is the solvent are one pH unit lower.

Extraction reactions are quite reversible and the complex may be transferred back and forth indefinitely from the aqueous to the organic layer if the pH of the aqueous layer is changed appropriately. In the procedures given below, this reversibility is utilized in conducting separation procedures. A group of metals is extracted by an organic solvent from the aqueous layer which is then discarded. The organic solvent is then

stripped of these metals by shaking with a second aqueous solution of appropriate pH. Some metals such as nickel, cobalt and bismuth exhibit a marked hysteresis effect in this regard, requiring a much more strongly acid solution for the stripping operation than would be expected.

Valence. Variation of metallic valence provides another important means of effecting separations in dithizone procedures. This may be applied to metals such as iron, tin and platinum which do not react in their higher valence forms but do complex in the reduced forms. Thus, if these metals are in aqueous solution in their higher valence forms, interfering metals can be extracted without affecting this group. Then, after treatment of the aqueous solution with a reducing agent, these metals may be extracted in a purified state in their lower valence forms.

Chelating agents. Another very important method for making dithizone extractions specific is the use of complexing agents such as iodide, thiosulfate, and cyanide as illustrated in Table 3. These reagents effectively control the interfering metals by forming very tightly bound complexes which prevent these elements from reacting with dithizone.

Table 3. Complex-forming reagents in dithizone reactions

| Conditions | Metals Reacting |
|---|---|
| Basic solutions containing citrate and tartrate. | Usually without effect |
| Basic solution containing cyanide. | Pb, Bi, Sn(II), Tl(I) |
| Slightly acid solution containing cyanide. | Cu, Hg, Ag, Pd |
| Dilute acid solution containing thiocyanate. | Cu, Au, Hg |
| Dilute acid solution containing thiocyanate and cyanide. | Cu, Hg |
| Acid solution of pH 5 containing thiosulfate, and extracted with $CCl_4$ solution of dithizone. | Pd, Sn(II), Zn(Cd) |
| Acid solution of pH 4-5 containing thiosulfate and cyanide. | Sn(II), Zn |
| Dilute acid solution containing iodide or bromide. | Cu, Au, Pd |
| Dilute acid solution containing chloride. | Cu, Au, Pd, Hg |

The procedure for a single metal analysis is based upon the proper combination of these variable conditions so as to favor the reaction with the metal being determined and suppress reactions with the interfering metals likely to be present. The limiting amounts of these interfering metals which can be tolerated are established experimentally.

## Lead Analysis

The methods recommended for determining lead in air and in biological materials are given below.[1,2,3] If lead is to be determined in air samples[1] the levels are generally considerably higher than those to be found in biological samples. Thus, higher blanks may be tolerated and purification of reagents may be simplified. Electrostatic precipitator samples of dust or fume may be transferred to 250 ml Phillips beakers. A special rubber policeman with a round rubber disc, which fits the tube snugly like a piston, is used to transfer the sample to a funnel placed in a Phillips beaker. Redistilled alcohol is a suitable solvent to use as it wets the tubes thoroughly. When no visible trace of dust remains in the tube, the alcoholic suspension in the Phillips beaker may be evaporated to dryness on a steam bath and then ashed in the manner recommended in the laboratory exercise for lead analysis.

### Collection of Samples

It will be noted that 24-hour urine samples are recommended in order to minimize the variable dilution which occurs at various times of the day. It is not advisable to collect urine samples in the plant because of the danger of contamination from dust. Borosilicate, glass-stoppered bottles, carefully cleaned with nitric acid, are given to the workers to take home.

Vacutainers, manufactured by Becton, Dickinson & Co., Rutherford, N. J., are recommended for collecting blood samples. Deleaded tubes are evacuated by inserting a hypodermic needle through the rubber stoppers. When the needle is withdrawn the vacuum is maintained in the tube for an indefinite period. A special sterile needle with sharp points at both ends is used for sampling. One end is pushed partly through the rubber stopper of the vacutainer and the other end is inserted into the vein of the subject. The sample is then collected by pushing the first end all the way through the rubber stopper into the evacuated space of the test tube. A volume of 5 to 10 ml of blood is required. No preservative is desirable because of the blank difficulties. When the tube is opened the entire sample is taken for analysis, because most of the lead is in the clot and aliquoting is not practicable. The tubes may then be cleaned and evacuated for reuse. A laboratory Tesla coil, of the type used for high vacuum work, is convenient for checking leaks in the tubes. Before shipping the tubes to the field, the Tesla coil is sparked next to the tubes. If the characteristic glow inside the tube does not appear, the vacuum has been lost and the tube is set aside. In this way no duds are encountered when a patient is being tested.

### Method of Analysis

The U. S. P. H. S. procedures which are given below[2] are known as double extraction methods. The aqueous solution of ashed sample is brought to the appropriate pH by adding indicator and ammonia. Since calcium and magnesium phosphates will precipitate at the required pH, the prior addition of citrate is necessary to prevent formation of such precipitates and concurrent losses of lead. Hydroxylamine hydrochloride is also added to protect the dithizone from oxidation. Most of the interfering metals are then

complexed by adding potassium cyanide. When this solution is shaken with a chloroform solution of dithizone, only lead, bismuth and thallium will be transferred to the organic solvent. The aqueous solution containing the ashed salts may then be discarded. The extracted metals are stripped by shaking with an exact volume of one percent nitric acid. The chloroform solution turns green and may then be discarded. The lead, bismuth and thallium are now contained in an exact volume of a known medium. Subsequent extraction with standardized dithizone under carefully controlled conditions will yield quantitative results.

Other procedures[3,4] are presented below in which only a single extraction is made to separate and determine the lead. These procedures have the advantage of rapidity and are applicable to special types of samples. However, the highest accuracy may not be expected, since the presence of small amounts of oxidant in the sample, perhaps as a by-product of the ashing process, may partially destroy some dithizone. As the absorption of unreacted dithizone is appreciable at the wave length used for spectrophotometric estimation of lead, an appreciable error may thus occur. In addition, such a single extraction procedure is subject to more interference since the single stage cyanide treatment is not as effective in suppressing interferences as the two stage method.

It has been mentioned above that lead, bismuth and thallium are determined together by these procedures. Thallium is rarely present and is ordinarily disregarded. Also, bismuth is rarely found in blood and urine[5] unless the patient is undergoing bismuth injection for syphilis therapy. This form of treatment is now obsolete in view of more modern treatments. The oral use of bismuth for digestive disturbances does not result in the presence of appreciable quantities of bismuth in blood and urine. If the presence of bismuth is suspected a special procedure[6] may be applied. The lead is stripped from the first extract with pH 3.4 buffer, leaving the bismuth in the chloroform layer which is discarded. As the pH is quite critical and the buffer has little capacity, the method requires prior washing of the entrained alkali from the first extract by shaking with distilled water. Thus this procedure has the disadvantage of requiring three separatory funnels instead of two. The extra funnel and water wash step may be eliminated if bromphenol blue indicator is added to the buffer and the pH is corrected as required by addition of dilute acid.

These procedures are known as mixed-color methods because of the presence of both the red, lead-dithizone complex and the unreacted, excess, green dithizone. If a high pH is used for the final extraction,[7] the excess dithizone is stripped out and a single color of varying intensities of red is obtained.

## Microdetermination of Cadmium with Dithizone

Cadmium may be determined specifically with dithizone by extraction at very high pH values. Although cadmium is complexed by cyanide, carefully regulated amounts of cyanide are used in amounts which suppress interfering metals but not cadmium. Under these conditions very few other metals will extract to any great extent. The aqueous layer is then discarded and the chloroform extract is stripped of cadmium by shaking with 2 percent

tartaric acid. A second extraction of cadmium is then made under similar conditions with standardized dithizone. The details of this method are given in reference (8).

It will be noted in this procedure that difficulties arise due to the instability of strongly alkaline chloroform solutions. Additional amounts of cyanide may be produced by the reaction of traces of ammonia with chloroform under these conditions, resulting in low cadmium recovery. These difficulties are minimized by using cold solutions and the shortest possible contact time of chloroform with strong alkali. In this manner complete extraction of cadmium is obtained.

## Determination of Mercury with Dithizone

Ashing of mercury samples is a special problem because of the volatility of mercury salts. Total reflux procedures are necessary. The treatment of air samples is outlined in the ACGIH method.(9) In urine samples the presence of water reduces greatly the efficacy of the oxidizing action. Satisfactory results have been obtained by digesting 100 ml of urine with 15 ml of concentrated nitric acid and 7½ grams of potassium permanganate under total reflux for 1 hour. A 500 ml flask is necessary because of foaming. Sulfuric acid is not required. After cooling, the permanganate is reduced by slow addition of hydroxylamine hydrochloride, 5 ml of which is then added in excess. After additional cooling, meta cresol purple indicator is added and the solution is titrated to a pink color with ammonium hydroxide. This solution is cooled and extracted with dithizone according to the ACGIH method for air samples.(9)

The basic principle of the mercury procedure consists of extraction in strongly acid media, followed by shaking the organic layer containing the mercury with an acidified, concentrated, solution of potassium bromide which strips the mercury, leaving copper and noble metals in the chloroform layer which is discarded. The aqueous layer is then brought to pH 6 by the addition of buffer solution. A second extraction with standard dithizone solution then extracts the isolated mercury. It should be noted that mercury dithizonate is sensitive to light and the solution must be kept out of direct sunlight.

We have simplified the preparation of reagents described in the ACGIH procedure. Reagent grade chloroform may be used without treatment, provided it is listed as passing the ACS Test for Suitability for Use with Dithizone. We do not find purification and re-use of chloroform economical. Standard dithizone solution is stable for months in a brown bottle kept in the refrigerator, and need not be prepared freshly for each set of analyses.

## References

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4. Woessner, W. W.; and Cholak, J.: Improvements in the rapid screening method for lead in urine. A.M.A. Archives of Indust. Hyg. and Occ. Med. 7:249, March 1953.

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7. Cholak, J.; Hubbard, D. M.; and Burkey, R. E.: Microdetermination of lead in biological material, with dithizone extraction at high pH. Anal. Chem. 20:671, July 1948.

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# DETERMINATION OF NITROGEN OXIDES IN AIR

## Introduction

Nitrogen oxides occur in chemical manufacturing operations such as those involving nitric acid, nitrates, dyes, explosives, nitro-cellulose paints and lacquers. They are also produced in blasting, welding, electroplating, and metal cleaning operations, as well as in fires, in the exhaust of internal combustion engines and in furnace stack gases. Nitrogen oxides have become very prominent recently as important factors in smog generation.

## Properties of Nitrogen Oxides

A series of nitrogen oxides exists. Many discrepancies in reported toxic effects may be due to failure to distinguish between different oxides which have widely varying properties.

Nitrous oxide ($N_2O$). Commonly known as laughing gas, this oxide of nitrogen has been used for anesthesia. Concentrations as high as 80 to 86% have been used in dentistry, thus this oxide may be regarded as being of little importance from the industrial toxicology point of view.

Nitric oxide (NO). This gas has no irritant properties. The effects on the body are probably due to anoxia. It combines with the blood and produces cyanosis caused by methemoglobinemia. No after effects occur unless the exposure is carried beyond the point of asphyxial-convulsions and sudden central paralysis.

Nitrogen dioxide ($NO_2$). This is the most irritating and toxic of all oxides of nitrogen. Many fatalities have been caused by this gas. Delayed pulmonary edema and sudden collapse are the symptoms to be expected. The present maximum allowable concentration is 5 PPM. Nitrogen dioxide may be produced by the trimolecular oxidation of nitric oxide by oxygen of the air: $2\ NO + O_2 = 2\ NO_2$. The half life of this reaction at 25 °C is 69 hours divided by the initial parts per million of nitric oxide. Thus if the latter is present at a level of 100 ppm the half life of the nitric oxide would be 41 minutes. After aging only 2½ minutes the TLV of 5 ppm for $NO_2$ would be exceeded. Subsequently, the toxicity of the nitrogen dioxide present would greatly outweigh that of the nitric oxide. Thus in a mixture of nitric oxide and nitrogen dioxide, we ordinarily need be interested only in nitrogen dioxide levels.

Other nitrogen oxides. These are much less important. Nitrous acid anhydride ($N_2O_3$) and dinitrogen tetroxide ($N_2O_4$) dissociate to produce nitric oxide and nitrogen dioxide. These dissociations are favored by lower pressures and are complete at concentrations below 100 ppm. Because of the great rapidity of their reactions, these oxides can be eliminated from the picture.

Nitrogen pentoxide ($N_2O_5$) is a rare and unstable oxide produced only by special procedures such as the reaction of nitrogen dioxide with ozone or

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the dehydration of nitric acid with phosphorus pentoxide. Its hydration by atmospheric water vapor to produce nitric acid vapor is very rapid. It also decomposes much less rapidly to a mixture of nitrogen dioxide and oxygen, the half life at 25 °C being 5.3 hours.

Both nitric acid vapor and nitrous acid vapor may be present in the air at an appreciable level. These are much less toxic than the equivalent amount of nitrogen dioxide.

Another oxide, nitrogen trioxide ($NO_3$), may be found in very small amounts in mixtures of nitrogen oxides and ozone. However, these levels would be very low and the situation is so unusual that the formidable analytical problems have not been solved. Ordinarily the major toxic effect may be expected to come from the ozone.

## Sampling Methods

It may be seen from the foregoing that nitrogen oxides may exist in complex mixtures. To these difficulties is added the problem of sampling. Nitrogen dioxide may be sampled by a continuous scrubbing technique with good efficiency even at low concentrations.(1) It is likely that nitrogen pentoxide, if present, would also be sampled quite efficiently by an aqueous solution. Little information is available about continuous sampling methods for other nitrogen oxides.

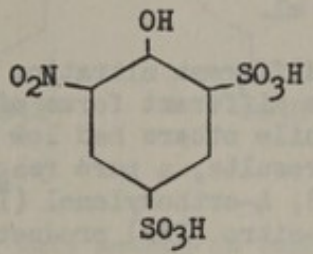
The efficient continuous absorption of nitric oxide has not been attained. This problem has been investigated in detail by Thomas, et. al.,(2) who recommended the conversion of nitric oxide to nitrogen dioxide and its estimation in that form. Thus, two determinations are necessary. The first gives the total nitric oxide and nitrogen dioxide and the second gives the nitrogen dioxide alone. The difference is the amount of nitric oxide. The oxidation of nitric oxide to nitrogen dioxide was achieved by the addition of small amounts of ozone from air passed near a Westinghouse 794H ultraviolet lamp. Chlorine dioxide and acid potassium permanganate solutions were also effective oxidizers. The Thomas method is applied widely in air pollution studies for continuously recording low concentrations (fractions of a ppm) of these oxides.

If the total nitrogen oxides are to be determined it is generally necessary to use grab sample methods rather than continuous scrubbing devices. This necessitates the use of evacuated bottles or of flushing devices. A rather large sample of the order of several liters is necessary when working with levels close to the maximum allowable concentration of nitrogen dioxide (5 ppm). In the following sections, two procedures will be described which are useful for determining total nitrogen oxides, following which the method for determining nitrogen dioxide specifically will be discussed.

### Phenoldisulfonic Acid Method for Total Nitrogen Oxides

Details of this method are given in the ACGIH procedure.(3) A grab sample is taken in a bottle containing a very dilute hydrogen peroxide-sulfuric acid absorbing reagent. After absorption of the sample, the

solution is made alkaline and evaporated to dryness, after which it is treated with phenoldisulfonic acid. This reagent is readily nitrated giving the product shown below:




The mixture is then diluted and made alkaline with sodium hydroxide to obtain the trisodium salt of the nitro compound which has an intense yellow color. This color is determined photometrically at 410 millimicrons.

The reagents for this method (with the exception of the absorbing solution) may be kept for months. The nitration step is usually completed within two minutes, although ten minutes are allowed as a safe time. Final calculations are made using the perfect gas laws discussed in the lecture on gas and vapor sampling.

The weakest part of this method appears to be the sampling and absorption portion. When lower concentrations of nitrogen oxides are determined the recovery of the method decreases.(1) Thus at the 10 ppm level only about 60% may be recovered. The absorption is very slow at these low levels. Even three days time is not sufficient for complete absorption since slightly higher results may be obtained when one week is allowed.

Other difficulties may arise for various reasons. The blank value is usually of the order of several ppm. Most of this is from the hydrogen peroxide used in the absorbing reagent. If the solution is made too alkaline during the initial evaporation step, an appreciable amount of material is dissolved from the dish, producing a turbidity in the final color. These make the method difficult to apply except at rather high levels.

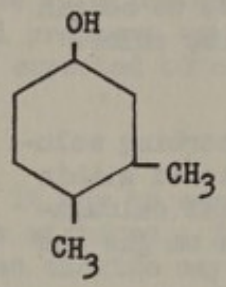
If an appreciable amount of organic matter is present the sample may char, producing an off color and possibly causing loss of nitrate by reduction. In such a case the neutral sample may be ashed with hydrogen peroxide. The presence of chlorides may cause loss of nitrate as nitrosyl chloride. A complete preliminary treatment should consist of removing chlorides by treatment with silver sulfate. Then phosphate is added and the pH is adjusted to 6.5; the solution is filtered and ashed with hydrogen peroxide in the presence of calcium carbonate buffer. When the solution is finally made alkaline for color development, precipitation may occur in some samples. Sequestrene AA has been added to prevent this difficulty.

Xylenol Method for Total Nitrogen Oxides

This method(6) may be preferred for certain operational advantages, although it has only about a quarter of the sensitivity of the phenoldisulfonic acid method. A grab sample is absorbed in 0.5 ml of 62.5% (5:3) sulfuric acid. Potassium permanganate is added to oxidize any nitrite followed

by 0.2 ml of 1% m-xylenol in propylene glycol. Nitration is completed in a 10 minute interval, after which the sample is diluted with 100 ml of water and distilled. The nitrated meta xylenol is volatile and is absorbed in 1 ml of 2% sodium hydroxide producing an intense yellow color which is determined at 450 millimicrons in a volume of 25 ml.

In a study of this method,(4) different nitration products and side reactions were shown to occur with the different forms of xylenol. Some products were not steam-distillable while others had low extinction coefficients. Certainly, for quantitative results, a pure reagent is necessary. These authors recommend a reagent of 3, 4-orthoxylenol (I) which gives a mixture of the 2-nitro (II) and the 6-nitro (III) products.

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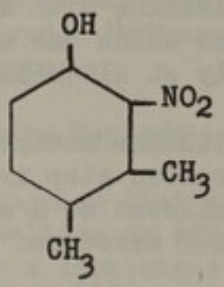


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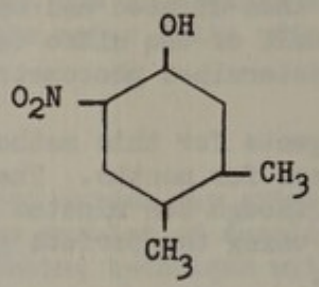



III

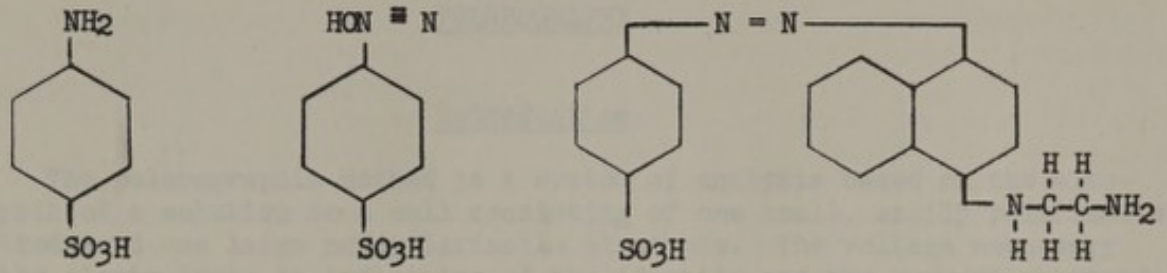
In another procedure,(5) instead of distillation, the nitro-xylenol is extracted from the acid solution with toluene. The aqueous layer is then discarded and replaced with 10 ml of 0.4 N sodium hydroxide. When this is shaken with toluene the salt goes into the aqueous layer, producing the color which is determined photometrically.

The advantages of the xylenol method are that sample evaporation is not required and no precipitation of metals occurs when the solution is made alkaline. The distillation or extraction steps enable one to make a separation from many interfering substances. However, this method has only about one-fourth the sensitivity of the phenoldisulfonic acid procedure.

Saltzman Method for Nitrogen Dioxide

As indicated above, in most cases the major toxic effect of nitrogen oxide gases may be attributed to nitrogen dioxide. The specific determination of this form therefore is generally more meaningful than the group determination of all the nitrogen oxides by the foregoing procedures. The nitrogen dioxides may be determined simply using the Saltzman reagent.(1) Levels below the TLV of 5 ppm may be determined conveniently, using a midget fritted bubbler and sampling at the rate of 0.4 liters per minute. Higher levels may be determined using evacuated bottles containing the reagent. Fifteen minutes after sampling, a pink color is completely developed and may be read photometrically at 550 millimicrons. The colors are quite stable, only 5% loss in absorbance occurring per day.

The reaction which occurs is the diazotization of sulfanilic acid (I) to produce the diazo salt shown (II). This couples with N-(1-Naphthyl)-ethylenediamine dihydrochloride to produce the azo dye shown in (III)




I II III

The standardization of this method is empirical. The reaction of nitrogen dioxide gas appears to occur via two pathways, each of which may produce a different amount of color. For a given type of sampling apparatus, the color stoichiometric relationships are quite constant. The color obtained from one mole of nitrogen dioxide when a midget fritted bubbler is used as the sampling device is the same as the color obtained from 0.72 moles of potassium nitrite.

This method is highly specific for nitrogen dioxide. The interference of ordinary amounts of ozone, sulfur dioxide, and other nitrogen oxides is negligible. The only interference of any importance is that due to organic nitrites, which would rarely be found in appreciable amounts.

This reagent may also be applied to the determination of nitric oxide, if this gas is converted to nitrogen dioxide as described above under sampling methods. Commercial recorders are now available for nitrogen dioxide as well as nitric oxide using this reagent.(2) These have been found very useful in air pollution studies to indicate the variation of these gases during a 24-hour period.

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# POLAROGRAPHY

## Introduction

The polarographic method is a system of analysis based on the electrolysis of a solution in a cell consisting of one small, easily polarizable electrode and one large non-polarizable electrode. The voltage necessary for the electrolysis is indicative of the identity and the current observed is a function of the concentration of the substance being analyzed.

## Theory

When using the dropping mercury electrode assembly the quantitative method of polarographic analysis is dependent on the current-voltage relationship obtained when an increasing potential is applied to the electrodes. When the decomposition potential of an electroreducible ion is reached, the current increases rapidly with the increasing voltage and then levels off to a "limiting current," the resulting current-voltage curve being "S" shaped. This limiting current is caused by an extreme case of concentration polarization at the cathode, and in the presence of a large excess of a non-reducible salt is produced solely by the diffusion of the electroreducible ions to the cathode. This "diffusion current" then is proportional to the concentration of electroreducible ions.

The equation for the diffusion current for the dropping mercury electrode assembly is called the Ilkovic equation:

$$i_d = 607nD^{\frac{1}{2}}Cm^{\frac{2}{3}}t^{\frac{1}{6}}$$

$i_d$ = average current during life of a drop, in amperes

n = number of electrons involved in reduction of one molecule of reducible substance

D = diffusion coefficient of reducible substance at a given temperature, in $cm^2$ per second

C = concentration of reducible ions, in moles per ml

m = weight in grams of mercury flowing from capillary per second

t = time in seconds for each drop of mercury

This equation is affected by three variables, viz., the concentration, the rate of flow of mercury, and the temperature. The temperature influences the diffusion current, the concentration of reducible substance, and the rate of flow of mercury. When the temperature is kept constant by use of a water bath, and the rate of flow of mercury is kept constant by use

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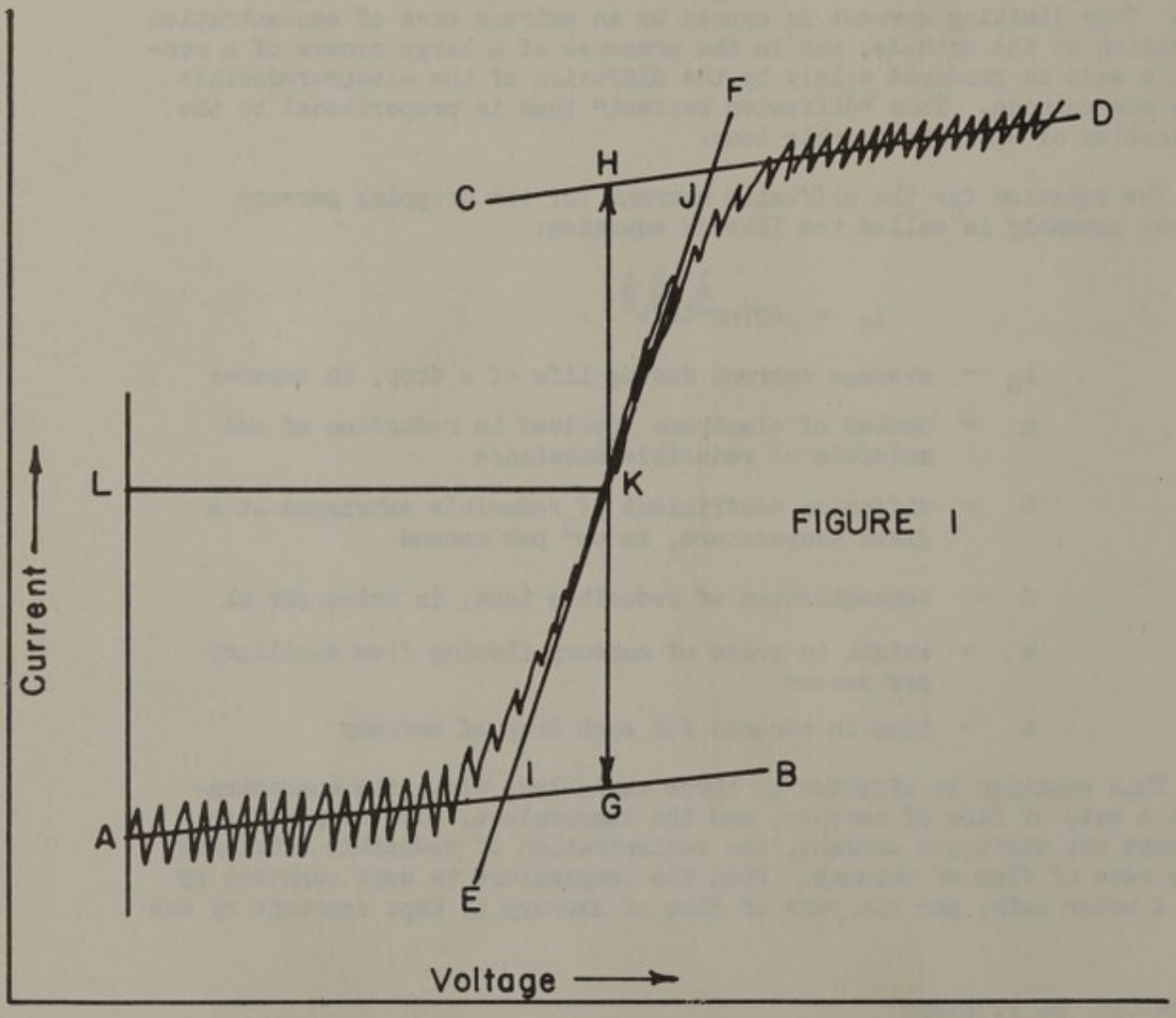
of an adjustable reservoir, the average current becomes a function solely of the concentration of reducible ions.

A rotating platinum electrode (R.P.E.) may be substituted for the dropping mercury electrode (D.M.E.) if the speed is kept constant, preferably at 600 rpm. With the R.P.E. the diffusion current is greater, but less reproducible, and the R.P.E. can be used at potentials more positive than can the D.M.E. The equation for the diffusion current for the R.P.E. assembly is:

$$i_d = KnDC \text{ where K is a constant}$$

The polarographic method for <u>qualitative</u> analysis is dependent on the different decomposition potentials of the reducible substances, usually expressed as the half-wave potential, $E_{\frac{1}{2}}$ (Fig. 1).



When the half-wave potentials of several reducible substances in the test solution differ sufficiently, they may be determined simultaneously, both quantitatively and qualitatively.

## Applications

A polarographic analysis may be made of a substance if that substance is electroreducible or electrooxidizable within the range of the electrode if it is in true solution and is stable for the duration of the measurement. In the field of industrial hygiene, determinations are made for lead, cadmium, zinc, vanadium, iron, thallium, halides, sulfur dioxide, cystine, and many other substances (Fig. 2).

FIGURE 3

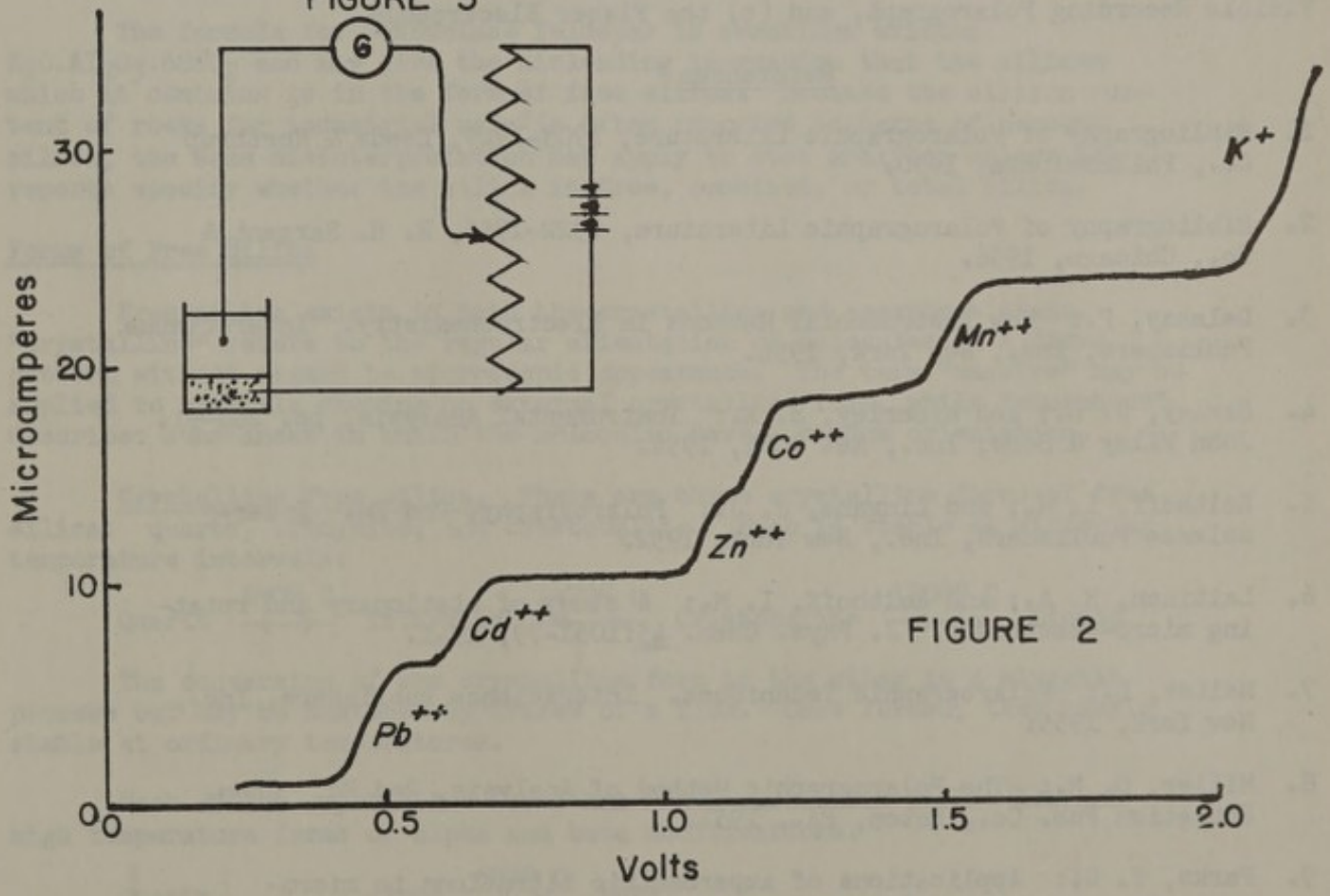



FIGURE 2

## Essential Equipment

The following equipment is necessary in order to conduct a polarographic analysis: (a) An electrolysis vessel which contains the electrodes, the sample, a supporting electrolyte, a maxima suppressor, and usually a buffer; (b) a constant temperature bath, and (c) several types of electrodes.

## Special Techniques

In addition to the usual methods of polarographic analysis, the normal instruments with accessory equipment can be used for: (a) amperometric titrations, (b) derivative polarography, (c) oscillographic polarography, and (d) continuous recording at fixed potential.

## Instruments

Figure 3 shows a diagram of a simple instrument. More elaborate instruments are: (a) the Sargent Model XII Photographic Recording Polarograph, (b) the Leeds & Northrup Electro-Chemograph Type 3, (c) the Sargent Model XXI Visible Recording Polarograph, and (c) the Fisher Electropode.

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# FREE SILICA -
# ITS FORMS, OCCURRENCE, AND DETERMINATION

## Introduction

Silica is another name for silicon dioxide. In order to emphasize that the silica of interest in Occupational Health exists as such and not in combination with other elements, it is called "free silica." This distinction between free silica and combined silica is important not only because of the differences in physiologic action but because of the differences in the methods used for their determination.

Table I. Distinction Between Free and Combined Silica

| | Free Silica | Combined Silica |
|---|---|---|
| Example: | Quartz | Feldspar |
| Formula: | $SiO_2$ | $KAlSi_3O_8$ |

The formula for orthoclase feldspar is sometimes written $K_2O.Al_2O_3.6SiO_2$ and may give the misleading impression that the silicon which it contains is in the form of free silica. Because the silicon content of rocks for industrial uses is often reported in terms of percent silica, the same misinterpretation may apply to dust analyses unless the reports specify whether the silica is free, combined, or total silica.

## Forms of Free Silica

Free silica exists in both the crystalline and amorphous state. "Crystalline" refers to the regular orientation of molecules in a fixed pattern without regard to microscopic appearance. The term "massive" may be applied to minerals showing no external crystalline form, while "amorphous" describes substances in which the molecules have a random orientation.

Crystalline free silica. There are three crystalline forms of free silica: quartz, tridymite, and cristobalite. Each is stable at differing temperature intervals:

$$\text{Quartz} \xleftrightarrow{870^\circ\text{ C}} \text{Tridymite} \xleftrightarrow{1470^\circ\text{ C}} \text{Cristobalite} \xleftrightarrow{1700^\circ\text{ C}} \text{Liquid}$$

The conversion of one crystalline form to the other is a sluggish process but may be hastened by traces of a flux. Once formed, they remain stable at ordinary temperatures.

Each of the crystalline forms has minor variations called low and high temperature forms or alpha and beta modifications.

$$\text{Quartz} \quad \text{alpha} \xleftrightarrow{573^\circ\text{ C}} \text{beta}$$

$$\text{Tridymite} \quad \text{alpha} \xleftrightarrow{117^\circ\text{ C}} \text{beta}_1 \xleftrightarrow{163^\circ\text{ C}} \text{beta}_2$$

$$\text{Cristobalite} \quad \text{alpha} \xleftrightarrow{200^\circ\text{ C to }272^\circ\text{ C}} \text{beta}$$

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Because these inversions are rapid, only the alpha forms exist at ordinary temperatures. Quartz having the external crystal characteristics of beta quartz may be found but the internal structure is, nevertheless, that of alpha quartz.

The inversion of quartz to the beta form at 573° C can be used as a quantitative method of determination if the non-quartz fraction does not vary greatly in composition from sample to sample. The basis of the method is the amount of heat adsorbed by the quartz in passing through the inversion temperature.

Quartz. Quartz is by far the most common of the free silicas. It has been estimated that 12 percent of the earth's crust is quartz. The lighter colored igneous rocks such as granite are more likely to contain quartz than are the darker rocks, such as basalt, which are composed of basic constituents in the form of ferromagnesian minerals. Sand, sandstone and quartzite are almost entirely composed of quartz. Clay, shale, and slate usually contain significant amounts of quartz in the range of 0 to 50 percent. Because of the widespread occurrence of quartz, many ores as well as industrial minerals such as talc, mica. feldspar, perlite, and pumice may have an appreciable free silica content.

The cryptocrystalline varieties of quartz are composed of minute grains cemented together with amorphous silica and include: chalcedony, agate, onyx, jasper, siliceous sinter, flint, hornstone, chert, as well as many others. Tripoli is a disintegrated cryptocrystalline quartz used for polishing and buffing.

Tridymite. This is not a common natural form of free silica but it is found in some acidic volcanic rocks. Tridymite is the principal constituent of silica refractory brick.

Cristobalite. It does not occur naturally to any great extent but cristobalite is formed when quartz and amorphous silica is heated to a high temperature. It is the principal constituent of calcined diatomaceous earth and is also present in many ceramic materials and refractory bricks. Cristobalite may be produced by the decomposition of some silicate minerals when heated to a high temperature.

Amorphous silica. Fused silica and fused quartz are amorphous and differ from crystalline free silica in physiologic properties. Silica fume also is amorphous and is produced from molten silica, ferrosilicon, or the working of silica ware. Opal is a natural hydrated form of amorphous silica while silica gel and silica aerogel are artificial products of the same form. Diatomaceous earth is an opaline silica composed of the skeletons of diatoms, but converts to cristobalite when calcined at high temperature, particularly in the presence of a trace of alkaline flux.

## Determination of Free Silica

### Chemical Methods

Determination of quartz by chemical means consists of dissolving the the constituents of a dust sample other than the quartz with reagents which

have little action on quartz. Suitable reagents have been hydrofluoric acid, hydrofluosilicic acid, fluoroboric acid, fused potassium pyrosulfate and sulfuric acid in combination with sodium hydroxide. Because of the length of time of analysis and the uncertainty of results with these reagents, phosphoric acid is generally accepted as the preferred reagent. Phosphoric acid, when heated at a controlled rate, dissolves most silicates in 12 to 14 minutes and has little effect on the quartz; the loss of 200 mesh quartz is only about 1 percent. The pyrophosphoric acid, which forms during the heating of the sample with the acid, complexes with both the metallic elements and the combined silica and it is only necessary to dilute the acid solution to recover the residue of quartz.

Microscopic Methods

A few substances do not dissolve readily in phosphoric acid. It is wise to examine a portion of the residue of quartz microscopically and, if substances other than quartz are found to be present, an attempt should be made to identify these and a correction applied to the analysis on the basis of their chemical composition.

This identification can be carried out most easily by mounting the residue in an oil having the same refractive index as quartz. A mixture of equal parts by volume of d-chloronaphthalene and light mineral oil (liquid petrolatum) is suitable and may be adjusted to a refractive index of 1.548 by means of a refractometer. With ordinary illumination the quartz will be invisible permitting attention to be concentrated on the impurities. A polarizing attachment to the microscope is useful in obtaining a rough estimate of the percentage of extraneous particles since the quartz particles may be made visible when observed between crossed polarizers.

The quartz in the bulk of the residue is volatilized with hydrofluoric acid in the presence of sulfuric acid and any remaining residue interpreted in accordance with the microscopic examination in terms of percentage of undissolved extraneous minerals or substances.

X-ray Diffraction

Because of the higher solubility of tridymite and cristobalite in phosphoric acid, these are more accurately determined by x-ray diffraction. All three of the crystalline varieties of free silica have intense diffraction patterns easily differentiated from one another and from amorphous silica which does not give diffraction lines because of the random orientation of the molecules.

Amorphous silica cannot be determined accurately in complex mixtures and it may be necessary to rely on total silica values. In simpler mixtures sufficiently accurate results may be obtained by a rational analysis.

# FLUORIDES

## Introduction

The compounds of fluorine have a widespread use in industry and, because of the low threshold limit values assigned to them, they are in the group of frequently investigated air contaminants.

## Occurrence and Uses

The three important natural sources of fluorine are fluorspar, cryolite and phosphate rock.

Fluorspar. Known chemically as calcium fluoride, fluorspar is mined in Illinois, Kentucky and some of the western States. Fluorspar provides most of the fluorine for secondary uses.

Cryolite. (Sodium aluminum fluoride) The only significant commercial deposit is located in Greenland. Cryolite has direct usefulness in the aluminum reduction industry as a molten electrolyte and solvent for bauxite, the basic ore of aluminum. Cryolite is also used in crop dusting as an insecticide.

Phosphate rock. This is primarily calcium phosphate containing fluorine in amounts less than 4 percent. The deposits in Florida are of sedimentary origin and consist of hydroxylapatite which, when treated with sulfuric acid, is converted into a more soluble form suitable for use as a fertilizer and feed supplement. The gaseous fluorine compounds, released in the processing, can create air contamination problems both within and outside the processing plants. Some of the fluorine is recovered and converted to synthetic cryolite and other by-products. The phosphate deposits of the western States are more difficult to process but are being mined at an increasing rate. The hard, crystalline apatite of metamorphic origin occurs in only small deposits in the United States.

## Secondary Fluorine Compounds

Hydrofluoric acid is used for the etching and polishing of glass, for pickling of stainless steel, and also by the petroleum industry in producing aviation gasoline by alkylation. Fluosulfonic acid and boron trifluoride also serve as catalysts of organic reactions. Fluosilicic acid finds use as a disinfectant of copper and brass vessels in the brewing industry. It is also used as a hardener for concrete and in the electroplating of certain metals. Silicofluorides have been used to some extent as laundry sours and insecticides and are used by the ceramic industry as fluxing and opacifying agents. Sodium fluoride is being used increasingly in the fluoridation of water supplies. Potential health hazards are created by its use in the production of rimmed steel and also in its use as an insecticide, rodenticide, and fungicide.

Bifluorides (acid fluorides) are constituents of welding fluxes, used particularly with aluminum and stainless steel. Also, they have been used extensively to frost glass but are being displaced by sand blasting.

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Fluoborates have value as fluxes in the casting of aluminum and magnesium and as electrolytes in plating with lead and similar metals.

Exposures to uranium hexafluoride and elemental fluorine are associated with the atomic energy industry. Elemental fluorine also has uses as a high energy fuel.

The freons are chlorofluoromethanes and ethanes. They have a low order of toxicity and are used extensively in refrigeration equipment and as solvent propellants in spray cans containing insecticides, paints, deodorants, etc.

Sulfur hexafluoride is inert and non-toxic. It is used as a gaseous insulator because of its high dielectric strength. Toxic decomposition products are given off by otherwise stable organic fluorine compounds when they come into contact with flame or hot surfaces. The same applies to plastics made of polymerized chlorofluoroethylenes and tetrafluoroethylene. The aromatic fluorine compounds have toxic properties and are used as insecticides.

The toxicity of organic fluorine compounds is dependent upon the structure of the compound. Aside from the highly reactive elemental fluorine, the toxicity of inorganic fluorides is attributed primarily to the fluorine content. The same order of toxicity is assigned whether the fluorine-bearing contaminant exists as particulate matter or as a gas.

## Analytical Methods for Fluorides

### Collection of Samples

Because of the high solubility of hydrogen fluoride in water, it is generally assumed that this gas is efficiently collected by a single standard impinger containing 100 ml of 0.1 N NaOH and operating at one cubic foot per minute (cfm). A 10 to 15-minute sample is adequate for concentrations near the Threshold Limit Value of 3 ppm. A coarse, fritted glass bubbler with an air flow of one liter per minute (lpm) may be used when the sampling time is extended. Dusts and mists containing fluoride may be sampled with the large impinger. Smokes and fumes can be collected on membrane filters but, if gaseous fluorides are also present, these should be followed by a bubbler or impinger. Particulate fluorides may be collected with the electrostatic precipitator. If the deposit is corrosive, it may adhere too firmly to the precipitator tube.

### Preparation of Samples

Samples in aqueous media should be evaporated to a small volume in platinum dishes in the presence of an alkali. Sodium hydroxide or a slurry of calcium hydroxide are suitable when no organic matter is present. When organic matter is present, either calcium hydroxide or magnesium acetate should be used and the residue evaporated to dryness and ashed at a dull red heat (550° C). Magnesium acetate is soluble in alcohol and is especially suitable for samples washed from precipitator tubes with alcohol.

Consideration must be given to whether the fluoride is to be distilled from sulfuric or perchloric acid. If sulfuric acid is to be used, the use of calcium hydroxide must be avoided because of the insolubility of calcium sulfate.

Blank values in fluoride determinations tend to be high. Special selection or purification of reagents can usually be avoided by collecting sufficient sample to override the blank variation.

Distillation of Fluoride

Fluoride is separated from interferences by distillation from either sulfuric acid or perchloric acid. Distillation from sulfuric acid in the range of 145 to 155° C gives good recovery from samples containing aluminum and iron, but should not be used for samples high in calcium or when the subsequent method of determination is sensitive to small amounts of sulfate. Perchloric acid in the range of 135 to 140° C is suitable for a greater variety of samples but should be used in an all-glass still and with every precaution to avoid organic matter in the sample. If halides, other than fluorine, are present, they are held back by the addition of an excess of silver sulfate or silver perchlorate added as a solid or saturated solution. The temperature of the acid is maintained within the specified limits by adding water dropwise with a dropping funnel or by steam distillation. Temperature control is somewhat easier with steam distillation. The steam inlet tube should extend nearly to the bottom of the distillation flask and should not be constricted or bent at the end, otherwise an atomizing action will carry spray into the receiver.

Silica, present in the sample or dissolved from the glassware, may deposit as a gelatinous mass above the level of the acid. Fluoride will be trapped by this silica unless the distillation is conducted in such a way as to continuously break up the mass and keep it in contact with the acid. Collecting twice the usual volume of distillate may be necessary for adequate recovery in the presence of gelatinous silica.

Determination of Fluoride

Large amounts of fluoride are usually determined by titration with thorium nitrate, using monosodium alizarin sulfonate as indicator and monochloracetic acid as buffer. Small amounts can be determined colorimetrically with the thorium or zirconium lakes of alizarin sulfonate and using Nessler tubes for the comparison. Photometric determinations may be made with Megregian's reagent. The reagents for the methods given are stable at least for several months and all of the methods are reliable with reasonable precautions in their use.

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# APPLICATIONS OF BIOCHEMICAL METHODS TO OCCUPATIONAL HEALTH PROBLEMS

## Introduction

The title of this section raises the question, "What difference is there between biochemical and chemical methods?" The answer is that both use chemical procedures, but biochemical methods involve application of principles derived from a knowledge of the way in which the animal body reacts to or handles substances with which it comes into intimate contact. The living organism with its remarkable synthetic and catabolic mechanisms carries out complex chemical transformations and generates products by such unique catalytic processes that the study of these comprises the special field of biochemistry. A number of examples will be given to show how this branch of the science contributes many principles and procedures useful in the work of the industrial hygienist.

## Biochemical Responses to Exposures

There are two ways of determining whether potentially injurious exposure to a chemical agent has occurred in personnel:

1. The body fluids (blood, urine) of the individuals may be analyzed for the agent or some metabolite thereof; or

2. Injury may be judged by the effects produced, such as changes in enzymes found in the circulation, occurrence of abnormal products like stippled red cells in blood or porphyrin in the urine, as evidence of derangement of normal body processes.

### Body Fluids

When it is possible to detect the agent or a product formed from it by the body, it is often possible to decide whether the degree of exposure has been greater than accepted criteria permit. For example, the limits for urinary content of lead, mercury, and various metals have been established. Non-metallic compounds and their metabolites have also been investigated and limits set for urinary concentrations of a number of solvents or their metabolites[1]. Examination of the methods employed for measurement of these reveals some advantages and disadvantages. In some cases too little of the compound may remain in blood or urine to afford satisfactory evidence of exposure; this can readily occur, for example, with carbon disulfide. Attempts to use ethereal sulfates in urine as a measure of exposure to benzene involves distinguishing small differences in the urinary sulfate distribution. On the other hand, p-nitrophenol is a unique and characteristic metabolite of parathion, and is detectable in small amounts[2,3].

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Examples where existence of hazards were demonstrated following detection of subtle injurious effects on personnel can be cited. Such a case is reported[4] where urobilinogen in the urine occurred as a consequence of liver injury resulting from exposure to tetrachloroethylene. Coproporphyrin in the urine[5,6] indicated injurious effects of exposure to lead; however, it was not used as a test for lead excretion, nor to compare with the amount of lead transport in the body.

Enzyme Changes

When the structural units of the body, i.e., the cells, are affected by injurious materials, their functional components, particularly enzymes, undergo changes. This occurs sometimes as a result of liberation of enzymes into the circulation, sometimes as a result of direct action of the agent on intracellular or free circulating enzymes. For example, the enzyme serum glutamic oxalacetic transaminase (SGOT) is normally present in serum, but in conditions such as liver cell damage or myocardial infarction relatively large amounts are released from tissues into the circulation. For this reason it is a sensitive index of pathologic changes and has been found to be remarkably affected by liver damage associated with chlorinated solvent poisoning[7].

Cholinesterase is an example of a circulating enzyme which itself is sensitive to exposure to a number of toxic materials of industrial importance. There are two types of this enzyme, one predominates in red blood cells and nerve tissues, the other in serum. They differ in degree of sensitivity toward different types of organic phosphate insecticides. Extensive application has been made of cholinesterase activity of cells and sera as indices of exposure to organic phosphates; a number of convenient methods are available for determining activity[8].

Pretoxicosis Tests

In this laboratory a number of investigations are aimed at developing pretoxicosis tests. In one instance the cystine content of fingernails was found to be correlated with degree of exposure to small amounts of vanadium[9]. Work in progress indicates that the clinical procedure for serum alkaline phosphatase activity may be modified to heighten its sensitivity so that effects of chronic exposure to carbon disulfide become more easily detectable. Such investigations are in line with the modern trend in toxicology. Less emphasis is being placed on the LD50 as a bench-mark, rather there is a search for more specific and sensitive changes in enzymes and other constituents of the tissues and circulation as indicators of damage by chemical agents. This type of work not only furnishes data having diagnostic application but also leads to knowledge of the mode of action of the agent and valuable perspectives from the standpoint of chemotherapy[10].

Detoxication and Nutrition

Laboratory studies disclose the kinds and degrees of biological effect produced by toxic agents. From these data the judgment is made, using this information, together with field studies on personnel as to maximum allowable concentrations for a substance. While revisions of the limits for maximum allowable concentrations are made from time to time, as more information becomes

available, in many instances there occur considerable disparities between American and foreign TLV values. For a number of substances foreign groups recommend lower levels than those set by American standards. Frequently, though not always, this results from observations of effects on personnel working under environmental conditions similar to those acceptable in the United States, yet no observations or complaints of injury occurred in the comparable U. S. group. It is not easy to reconcile these differences but some clues are available. In attempting to reconcile such differences, it is desirable to consider the mechanisms by which the body may reduce or destroy the toxicity of harmful substances that have gained entrance. This process has been termed detoxication, but in present usage the concept has been extended to include all the metabolic changes which foreign organic compounds undergo regardless of whether the changes result in increased or decreased toxicity.(11)

Three principal types of detoxication reactions have been established: (a) oxidations, (b) reductions, and (c) synthetic reactions, frequently termed conjugations. A large number of oxidation and reduction reactions are known, but according to Williams(11) only nine synthetic or conjugation processes have been recognized. These are listed below:

Conjugation Reactions

Glucuronic Acid Conjugation
Ethereal Sulfate Conjugation
Glycine Conjugation (Hippuric Acid Synthesis)
Cystine Conjugation (Mercapturic Acid Synthesis)
Ornithine Conjugation (Ornithuric Acid Synthesis)
Glutamine Conjugation
Thiocyanate Detoxication
Acetylation
Methylation

Measurement of the extent to which these reactions have occurred is made use of by the industrial hygienist in estimating exposures to such substances as benzene, toluene or aromatic acids, phenols, aromatic amines and nitriles. The occurrence of increased amounts of conjugates in the urine is indicative of the exposure load on the subject. For normal ranges and values indicating abnormal absorption, see the article by Dreessen and Sievers,(12) as well as reference (1).

Since the body's ability to detoxify depends on the supply of materials used in the process, it is obvious that nutritional factors can influence toxicity. Experimental evidence to show this, with various examples, have been presented by Goldwater and Shils(13). It is quite apparent that a person well supplied with dietary protein, fat and vitamins will tolerate many toxic substances better than an individual on an inferior diet. This, then, may explain some of the different effects observed from the same working conditions on American and foreign groups. In this laboratory an interesting relationship between toxicity and vitamin availability was developed in studies on vanadium toxicology. It was known that sodium metavanadate is several times as toxic to guinea pigs as to other species. The guinea pig differs from

other species of laboratory animals in that Vitamin C must be furnished in its diet, while other animals, such as rats and rabbits, are able to synthesize this vitamin. From these facts it appeared that tolerance to vanadium was related to the presence of adequate Vitamin C in the animal body. In experiments on several species of animals, Vitamin C has been tested and found effective in antidoting metavanadate injections.(14)

## Some Aspects of Metal Toxicity

In considering metal toxicity, attention is usually given to effects produced by the introduction of some metal such as lead or mercury into the organism. In recent years increasing knowledge regarding the functions and interaction of metallic elements indicate that metal deficiencies, direct or indirect, may be induced by the working environment. Animal nutritionists have been aware of a similar phenomenon. That the utilization of one nutrient element may be profoundly influenced by the presence or absence of another was firmly established by the finding that hemoglobin formation in the presence of adequate amounts of iron depended on the simultaneous availability of copper.(15) Since 1938 it has been found that: a type of zinc anemia may be corrected by feeding copper; normal dietary copper levels are not effective in forage with a high molybdenum content; swayback lamb disease occurs in grazing areas where forage has a normal copper content but above average lead and zinc - the disease responds to copper therapy.(16)

Studies of the metabolic effects of carbon disulfide, now in progress here, have shown that inhalation of this substance over a prolonged period leads to mobilization and loss of zinc from the tissues.(17) Thus a zinc deficiency may be produced as a result of exposure to vapors of an organic compound. To cite another example, the work of Curran on inhibition of the enzymes that produce cholesterol indicates that a manganese-containing enzyme is inhibited by vanadium.(18) More instances such as these will doubtless be discovered. From the point of view of the industrial hygienist, it becomes apparent that measurement of the relative spectrographic intensities of the spectral lines from metallic elements occurring in blood serum and urine may become an increasingly important diagnostic criterion.

## Air Pollution, Allergy and Cancer

Whether air pollution is a hazard to life and health or merely affects the individuals comfort is a paramount question of the day. Efforts to find the answer are being made through a number of approaches such as epidemiologic studies of populations, sickness and mortality data relationships to pollution indices, respiratory function tests on persons in high pollution environments. The biochemical attack is based on studies of response of cells, tissues, and organs to specific air pollutants, such as ozone, and to synthetic smogs. Studies of white blood cells exposed to smog indicate a depressing effect on cellular respiration and survival. Ozone has been found to diminish activity of enzymes in the lungs and to induce changes in blood serum similar to those occurring in allergies.(19) Some of these findings indicate the "templates" of the cells may be altered, so that the new cells arising from these will have altered functional properties. While this is still to be confirmed, it has interesting implications with regard to cancer formation.

## Gross Effects and Minute Effectors

Finally, some of the most striking physical changes in a person's appearance or behavior can be traced to specific enzyme effects. The pin-point pupils, nausea, diarrhoea and sweating caused by organic phosphate poisoning arise from injury to the enzyme cholinesterase. Among personnel of a Washington, D. C., printing plant, the once pleasant, after-work, glass of beer evoked flushing of the skin and distressful symptoms. Investigation developed that a new ingredient, butyraldoxime, added to ink, has properties peculiar to antabuse, viz., it blocks the enzyme system which metabolizes alcohol so that acetaldehyde accumulates in the body. The skin of colored workmen in a plant making resorcinol derivatives began to whiten. The fundamental cause was traced to inhibition of the enzyme which produces pigment formation in the skin.

## Conclusions

The examples cited point up the fact that the application of biochemical knowledge to industrial hygiene serves a number of useful purposes. By knowing how and why injurious effects are produced, it is possible to track down specific causes of physical and physiologic changes in personnel and to relate the chemical and enzymatic changes in the body to the chemical composition of the environment. In fact the last part of the statement comes very close to being a description of the function of what we choose to call industrial hygiene.

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# EMISSION SPECTROSCOPY

## Introduction

Emission spectroscopy is the study of the radiations emitted by atoms, ions, and a few molecular substances after suitable excitation by means of a thermal or electrical source of energy. These radiations lie in the near infrared, the visible and the ultraviolet regions of the electromagnetic spectrum. Spectrochemical methods of analysis (spectrographic analysis) make practical use of these radiations for qualitative and quantitative analytical purposes.

Many occupational health problems arise from the industrial exposure of workers to certain toxic materials including inorganic substances. In evaluating the extent of specific exposures, the elemental constituents of these inorganic substances are analyzed in dust or fume samples collected in the working environment. Also, biologic specimens are analyzed to determine the extent of absorption and excretion of the substance by the workers. Frequently the concentrations of the toxic agents are relatively low and analysis becomes difficult for the chemist without a spectrograph. For the analysis of more than 70 metallic or metal-like elements, the emission spectrograph is uniquely suited. This instrument is used to characterize and measure the radiation emitted by specific elements, present as a mixture in the sample material.

## Theory

"A spectrum has been defined as the ordered arrangement of radiation according to wave length. Electromagnetic radiations have been discovered that have wave lengths of every value in the range from thousands of kilometers to trillionths of a millimeter... Since no single instrument exists that will separate radiation containing all these wave lengths into a spectrum the electromagnetic spectrum has been divided into various 'regions' in accordance with the types of instruments available to produce and detect the waves of various lengths."(1)

That portion of the electromagnetic spectrum of direct interest to emission spectroscopists extends from 10 x $10^{-4}$ mm. (10,000 Angstroms) in the near infrared through the visible, the near and the far ultraviolet regions to about 2 x $10^{-4}$ mm. (2,000 Angstroms). Most work in the application of emission spectroscopy to analytical problems is confined to the 2100 - 7500 Angstrom region.

There are three types of emission spectra: (a) line spectra which are produced by highly excited atoms or atomic ions, (b) band spectra which originate from highly excited molecules (which must be sufficiently cool so that all the molecules are not dissociated into atoms or ions), and (c) continuous spectra which result when light is emitted by incandescent solids. Spectra of the first two types are produced when atoms (or atomic ions) or molecules (ionized or unionized), respectively, are separated sufficiently from their neighbors that they may radiate as individuals between collisions.

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This condition is realized in the excitation region of an arc, spark or Geissler tube discharge. Continuous spectra originate from incandescent electrode tips and from incandescent particles in the arc stream and thereby contribute to the background or "continuum" observed in spectrograms (photographic recordings of line and band spectra). This continuum tends to mask the weak line spectra of trace elements. Hence, spectrographic methods for specific determinations are based upon employment of optimal exposure conditions and spectroscopic buffer salts, which will minimize background production and, at the same time, increase the intensity of atomic spectra.

## Instrumentation

### Excitation Equipment

All elements emit characteristic radiation after sufficient excitation by a source of energy. Excitation is accomplished with a suitable "source unit," such as a flame, a 220 volt direct current arc, a high voltage alternating current arc, or a high voltage condensed spark. Each type of source has its own advantages and limitations and the choice of source equipment depends upon the needs of the individual laboratory. The most useful source in an occupational health laboratory is the arc between graphite electrodes, the tips of which are maintained at incandescence by 220 volts of direct current. This arc is indispensable in the spectrographic analysis of refractory materials. Also, it provides the extremely high sensitivity needed for submicrogram quantities of toxic elements. Therefore, this equipment is a basic requirement in all spectrographic laboratories where industrial hygiene samples are analyzed. The other types of source equipment offer definite advantages in special applications, such as the exclusive production of spectra of the more easily excitable elements with an air-acetylene flame; the direct analysis of solutions with a spark technique when the ultimate in sensitivity is not required and an adequate amount of a soluble sample is available; or the analysis of difficultly excitable elements, such as selenium, using a high voltage alternating current arc or spark source. Modern excitation equipment is available commercially in any desired combination of sources as a package unit. Combination sources of this type offer the additional advantage of providing a wide range of discharge characteristics, giving greater flexibility in the selection of excitation conditions.(7)

### Spectrograph

There are four main parts to any spectrograph:

1. The slit of a spectrograph permits only a narrow beam of light of mixed wave lengths to enter the instrument. Slits of 10, 20, or 30 micron widths are used in prism instruments.

2. The collimating lens, mounted between the slit and the dispersing device, brings the individual rays of light into parallel, prior to refraction or diffraction. (Light coming from the slit is divergent.)

3. The dispersing device, a prism or a grating, "sorts out" the radiation and arranges it according to its component wave lengths.

4. The recording device, a photographic plate or film, provides the means of obtaining a permanent record, a spectrogram, of the orderly arranged, discrete line spectra of the elements volatilized from the sample and excited by the energy provided by the spectroscopic source equipment.

For a complete description of the many types of prism and grating spectrographs commercially available, the reader should consult the standard texts on emission spectroscopy.[1,2,3] Instrument requirements vary with the nature of anticipated research and survey projects. With the problems encountered ordinarily in an industrial hygiene laboratory, the most fundamental instrument characteristics should be resolving power, adequate dispersion in the ultraviolet and lower end of the visible region (where most toxic elements produce their most persistent spectral lines) and speed (line to background) of the instrument. These properties are provided by several types of instruments, including the large Littrow type quartz prism spectrograph. If extensive work in the visible region is anticipated, then grating spectrographs with the desired resolving power, dispersion and speed should be considered.

Photographic Processing Equipment

A completely light tight darkroom is essential to prevent fogging of the spectroscopic plates or films.

Temperature control at 68° F is mandatory for plates and films being processed for quantitative spectrographic analysis. Commercially available developing and fixing equipment, complete with temperature control, may be obtained from the manufacturers of spectroscopic instruments.

Photographic developers and fixers, suitable for spectroscopic emulsions, are commercially available.

Densitometer

Quantitative measurements of the densities of the silver deposits in the developed and fixed spectroscopic emulsion are made with a densitometer. This instrument is a photometric device which is designed to measure the decrease in the intensity of a beam of light passed through a limited area of the emulsion, viz., through a photographically recorded spectral line. Commercially available densitometers vary considerably in optical design and in the method of converting light intensity into measurable electric current. Most instruments use a photovoltaic cell, a vacuum photoemissive tube, or an electron multiplier phototube to effect this conversion. The current is measured with a sensitive galvanometer or with a microammeter after amplification.

Most instruments are now constructed to perform the dual role of a densitometer and a comparator, which functions by projecting into juxtaposition on a viewing screen enlarged images of spectrograms from two different spectroscopic plates or films. The comparator is extremely useful for qualitative identifications of unknown sample spectra and, for time-saving purposes, it is a necessity when much qualitative spectrography is performed. For a complete description of modern densitometric instrumentation, the reader should consult the standard texts[1,2,3] and the manufacturers' bulletins.

## Other Equipment

Other equipment items required to facilitate the application of spectrographic procedures include a calculating board, equipped with movable ordinate and abscissa logarithmic scales and used for converting densitometric transmittance data to analysis line/internal standard intensity ratio values; an electrode shaper, fitted with a variety of drills and shaping knives to ensure the preparation of uniform electrode craters; and a step sector disc and motor mount or a series of filters with known light transmittances for the calibration of photographic emulsions.

## Applications

The emission spectrograph is used to detect qualitatively and to determine quantitatively the metallic or metal-like elements in any sample which exists as, or can be converted to, a mixture of inorganic substances. Very few chemical methods possess the sensitivity and none can claim the specificity of spectrographic procedures. Also, emission spectroscopy possesses the added and very practical advantage of providing multimetal analyses, a feature which makes feasible the detection and determination of both suspected and unsuspected elements in sample materials. In addition to these advantages, emission spectroscopic techniques reduce greatly the cost per sample, provided the volume of work warrants the expenditures for the installation and operation of the equipment.

## Quantitative Methods

General considerations. Quantitative spectrographic methods require the development and rigid application of a set of standardized conditions to ensure an acceptable level of analytical accuracy. Adherence to a particular method's conditions of sample preparation and spectroscopic exposure is so strict that, ideally, the only permitted variable is the concentration of the analysis element or elements. Although the relative intensities of the spectral lines of an element are constants of nature, the absolute intensity of any particular line will vary not only with the concentration of the element in the samples subjected to spectroscopic exposure, but also with changes in the concentrations of the major elements contained in the sample matrix. Other factors which cause intensity deviations are variations in spectroscopic exposure conditions, some of which can be controlled directly while others require indirect control. Such conditions as applied voltage, current, the analytical gap, and uniformity of electrode craters can be controlled with a satisfactory degree of precision. However, excitation conditions, resulting from the use of a direct current arc on nonbuffered samples as an example, can vary markedly due to the inherent tendency of the arc to wander and to produce "spot" burning. This situation, if uncontrolled causes temperature fluctuations which promote erratic volatilization and excitation of the sample elements. For this reason, a spectroscopic buffer salt is mixed with the samples (standards and unknowns) to provide uniform burning of the electrode charge and to prevent erratic volatilization of the elements. Some inorganic substances, used effectively for these purposes, are graphite, aluminum oxide, lithium chloride, lithium carbonate, potassium sulfate and calcium carbonate. In this laboratory, a 2.5 to 1.0 mixture of finely ground graphite and lithium chloride has been adopted as an extremely effective spectroscopic buffer. This mixture not only promotes uniform

burning of the sample matrix but it also suppresses background and cyanogen band formation.[5,6] Furthermore, it enhances the production of trace element spectra with the result that unusually high degrees of absolute sensitivity can be realized.[6]

Experimental conditions. Quantitative spectrographic methods, to be applicable by different analysts, should specify clearly the following conditions and information:

1. Type or types of sample material
2. Exact description of sample preparation
3. Type of excitation for specified analysis element(s)
4. Electrode material and crater description with dimensions
5. Crater waterproofing reagent - composition and how it should be used
6. Name and amount of spectroscopic buffer - commercial source of this material, laboratory preparation and storage, and exact description of addition to sample
7. Name, amount, and commercial source of internal standard element added to sample or buffer
8. Sample electrode drying conditions, if solution analyses with an arc are involved
9. Type of spectrograph and focusing of source employed in development of method
10. Spectrographic slit width
11. Excitation conditions
    (a) Voltage, current, capacitance, inductance, polarity, etc., as applicable to excitation employed
    (b) Exposure period
    (c) Use of a sector or filter - whether used during all sample exposures or confined to emulsion calibration.
12. Spectroscopic emulsion type
13. Emulsion calibration technique
14. Wave lengths of analysis and internal standard element spectral lines
15. Calculation of intensity ratio data - with or without background correction applied to analysis and internal standard line densitometric values.

Standard Samples

A series of increasing amounts of an element in a series of electrode crater charges, subjected to the proper spectroscopic exposure conditions for the volatilization and excitation of this particular element, produces a series of spectrograms in which the detectable spectral lines of this element are recorded with increasing density relative to increasing concentration. Such a series is actually prepared for the initial and periodic standardization of a quantitative spectrographic method. In addition to the definite and varied quantities of the analysis element (for example, 0.005 to 1.000 microgram) in the series of craters, the electrode charges contain the specified amount of the element-free sample base material (blood, liver, urine, etc., ash from which the element has been removed chemically, if necessary), the prescribed quantity of the spectroscopic buffer, and the specified amount of the internal standard element. All steps in the spectrographic exposure, photographic processing, densitometry, and treatment of densitometric data must be exactly as that to be used for unknown samples.

Densitometry

The densitometer, supported on a sturdy bench or table in an area removed from direct room illumination, should be stable at its zero and 100 percent transmittance settings before spectrograms are read. A non-recording type is preferred by emission spectrographers, as only single line transmittance values are needed. The instructions given in the instrument manual should be followed during installation and use.

The galvanometer scale is adjusted to read "0" when a black cloth is inserted in the light path and then adjusted to read 100 percent transmittance for the clear plate or film area just above (or below) the analysis line in the first or last spectrogram. Repeated adjustments of the scale and sensitivity settings are made until the two desired readings are realized. Information about the height and width of the slit of the instrument should be available in the instruction manual.

The plate carriage is adjusted so that the image of the spectral line is parallel to the slit. The maximal deflection of the galvanometer scale is then obtained by slowly moving the spectral line past the light beam, either by manual or motor drive operation of the scanning control (whichever is provided or preferred by the operator). This value is recorded as "% T" in the record book. The plate is then shifted to the internal standard line and the "% T" value of this line is recorded. If background transmittance values are desired, the area adjacent to each spectral line is scanned and the value provided by the least deflection of the galvanometer is taken and recorded. When the series of spectrograms has been read, the densitometric data are converted to Intensity of Analysis Line/Intensity of Internal Standard Line values using the emulsion calibration curve (H and D; Hurter and Driffield curve) and a calculating board to effect this conversion.

Emulsion Calibration

The spectroscopic plate or film emulsion must be calibrated in the wave length region containing the analysis and internal standard lines, each time a new lot of plates is purchased and every few months while a given lot

is in storage, because the photographic response decreases with age. This decrease in response can be minimized by storage in a refrigerator.

The purpose of this calibration is to relate the variation of optical density with the logarithm of the intensity of the source. An excellent fundamental treatment of this subject is presented in a recent text.(3)

Procedure. Repeated requests have been made for a detailed description of the emulsion calibration procedure, using a rotating logarithmic step sector. The sector may have a step ratio of 1:2, 1:1.5, or some other value stamped on the disc. In this laboratory, a sector having a step ratio of 1:1.5 is used to provide shorter exposure ranges than possible with a sector constructed with 1:2 step ratio, as the exposure times of a sectored light beam from a uniform source are proportional to the angular openings of the disc.

The sector disc is supported in a motor driven assembly and is mounted firmly on the optical bench of the spectrograph as close to the slit as possible, without striking the slit assembly if vibration should occur during adjustment of its rotating speed. The motor is allowed to warm up for 5 to 10 minutes and its rheostat control is adjusted so that the velocity of the disc is the maximum that can be tolerated just short of vibration (which must not occur during the exposure). The full slit of the spectrograph is exposed by removing the Hartmann diaphragm. An iron arc is operated as the source for the preparation of three successive spectrograms for periods of 10, 15, and 20 seconds. Make sure that there is sufficient clearance between the spectrograms on the plate or film whose emulsion is being calibrated.

Develop and fix the plate or film at 68° F. After drying, measure the percent transmittance values of each step of several selected iron lines in the region where the analysis line occurs. Make selections so that this collection of transmittance values cover the range at least from 10 to 95 percent, the probable working range of standard and sample transmittance data. The values thus obtained are used in preparing the emulsion calibration (H and D) curve, plotting log % T as ordinates and log I as abscissae. A calculating board, provided with movable logarithmic scales, eliminates the calculation of logarithms and antilogarithms each time the emulsion curve is plotted or used. The following table of calibration data will be used to explain how plotting is conducted.

Table 1. - Transmittance Data Obtained from Iron Arc Source Using Logarithmic Step Sector, Ratio 1:1.5

| Step | Intensity | Int. Plot Value | Fe Line No. 1 % T | Int. Plot Value | Fe Line No. 2 % T |
|---|---|---|---|---|---|
| 1 | 1.0 | 1.0 | 91.5 | --- | --- |
| 2 | 1.5 | 1.5 | 76.4 | 1.0 | 82.0 |
| 3 | 2.25 | 2.25 | 52.0 | 1.5 | 67.6 |
| 4 | 3.375 | 3.38 | 28.9 | 2.25 | 45.3 |
| 5 | 5.063 | 5.06 | 12.6 | 3.38 | 25.6 |
| 6 | 7.594 | 7.59 | 6.2 | 5.06 | 10.6 |
| 7 | 11.391 | 11.39 | --- | 7.59 | 4.7 |
| 8 | 17.086 | 17.09 | --- | 11.39 | 2.0 |

The logarithms of the transmittance values in Column 4 are plotted versus the logarithms of the intensity values given in Column 3. Then the abscissa scale is shifted to the right to permit plotting of the values given in Columns 5 and 6 in such a manner that a smooth, average curve may be drawn. If the spectrographer is dissatisfied with the resulting curve, then he should obtain additional densitometric data from the iron spectrograms and continue this method of plotting until he is assured that a satisfactory, average curve has been obtained.

This emulsion calibration curve is used for all calculations of the intensity ratio of the analysis line to the internal standard line in the region thus calibrated.

Analysis or Working Curve

The densitometric data obtained from the standard sample spectrograms are converted to intensity ratio (Anal. Line / Int. Std. Line) values, using the emulsion calibration curve and the calculating board to effect the conversion. The concentrations of the analysis element versus the calculated intensity ratios are then plotted logarithmically to obtain the analysis or working curve.

Transmittance data obtained from unknown sample spectrograms are converted to intensity ratio (Anal. Line / Int. Std. Line) values, using the emulsion calibration curve, and the concentrations are then determined graphically from the analysis curve.

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# MEDICAL AND INDUSTRIAL APPLICATIONS OF RADIATION

## Introduction

The purpose of this lecture is to provide general background information so that the student will appreciate some of the many benefits derived from the proper use of radiation. Since all radiation uses are potentially hazardous, it will also provide a clue as to where radiation hazards might be found. It would be impractical to list here all of the specific uses to which radiation has been put. We shall discuss only the more widely used applications and those which may have some special or unique features.

## Use of Radiation in the "Pre-Atomic Era"

### Medicine and Dentistry

Diagnosis. X-rays were discovered in the fall of 1895, and by February, 1896, they were already in general use for localization of foreign bodies and examination of fractures. Later, techniques were developed to the extent that not only the bones and teeth could be examined but also the circulatory, respiratory, gastro-intestinal, and genito-urinary systems.

Therapy. The therapeutic uses of x-rays and radium have been of tremendous importance, especially in the destruction of malignant tumors. They also have a variety of uses in dermatology such as in the treatment of ringworm.

In this atomic era, radiation still finds its greatest use in the healing arts. More persons receive radiation exposure from this application and the magnitude of exposures in general is greater from this source than from any other.

### Inspection of Materials

Miscellaneous early uses. As in medicine, the newly discovered rays were also quickly put to use in industry. As early as February, 1896, a defective weld was detected radiographically by Prof. A. W. Wright of Yale University. Among other of the many early uses were:

a. Examination of coal for impurities and ash

b. Detection of adulterations of flour and sugar with sand and chalk

c. Detection of pearls in pearl oysters

d. Distinguishing between real and artificial gems

e. Inspection of parcel post and baggage

f. Examination of insulation in cables.

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Industrial radiography. Industrial radiography has grown tremendously since its inception in 1896, the principal application being the inspection of welds and castings. An x-ray tube (or radium) is used as a source of rays which passed through the object under examination, making a picture on a film on the other side.

Industrial fluoroscopy. A convenient variant for the inspection of "soft goods" (usually non-metallic) is the industrial fluoroscope which can be applied to conveyorized applications.

Continuous automatic inspection. X-rays have also been used for continuous automatic gauging of thickness of sheet metal, and automatic inspection of containers to insure complete filling.

X-ray diffraction analysis. An important analytical procedure is x-ray diffraction analysis, whereby crystalline materials can be identified.

Non-Medical Irradiation of the Human Body

Fluoroscopic shoe fitting. The use of fluoroscopic or x-ray shoe fitting began about 1925. It has had considerable appeal as a scientific curiosity and as a sales device, but its value in fitting shoes is doubtful. Needless uses of radiation such as this should always be avoided.

"Inspectoscope." This is a trade name for a whole body fluoroscope intended to detect stolen articles, contraband, weapons, or instruments of sabotage carried by persons passing through a portal. Public Health officials or others with a knowledge of radiation hazards view this device unfavorably.

Luminous Compound

Radium has been widely used in self-luminous paints for watches and aircraft instruments. The production of luminous dials has been the cause of fatal radium poisoning among several dial painters.

## Use of Artificial Radionuclides

Medical

Radioactive tracers in the form of "labeled" or "tagged" compounds are used to determine the fate of normal metabolites of vitamins, hormones, steroids, amino acids, phospholipids, etc. The action of drugs can be studied by means of "tagged" atoms. Even microorganisms can be "tagged" by feeding them with "tagged" nutrients. $C^{14}$, $S^{35}$, and $Na^{24}$ are commonly used in this work.

Diagnosis. The advent of artificial radionuclides has provided the clinician with many new diagnostic procedures. Among these are the determination of blood volume, evaluation of thyroid activity, and the localization of tumors.

Therapy. Cobalt 60 is gradually replacing radium for intracavitary and interstitial use, and when installed in teletherapy units, is replacing high voltage x-rays.

New modes of therapy have been developed in which a radionuclide is introduced into the body and finds its way to the organ to be treated.

Industrial

Industrial radiography. Radium has been replaced by other gamma emitters which are less expensive. The most commonly used radiographic sources are $Co^{60}$, $Ir^{192}$, and $Cs^{137}$. They are selected according to the degree of penetration required.

Luminous compound. $Sr^{90}$ has been substituted for Ra in some luminous materials. A new and interesting application is the Krypton switch lamp used by railroads in remote locations.

Irradiation of materials. Foods and drugs can be sterilized without heat by massive doses of radiation. Unfortunately, the taste of foods is often adversely affected by this treatment.

Many chemical reactions can be induced by high radiation flux, one of these is utilized in toughening of polyethylene containers by irradiation.

Static elimination. In many industrial processes the movements of non-conducting material at high speed through machinery will generate static electricity which may constitute a fire hazard or may adversely affect the quality of the product. The static charge can be removed by producing ionized air near the charged surface.

"Atomic battery." Several models have been made for instruments requiring a low current - high voltage power supply.

Gauging. Some of the many useful applications are shown in Figure 3.

Tracers. The possible uses of radioactive tracers are unlimited. Some of them are shown in Figure 4.

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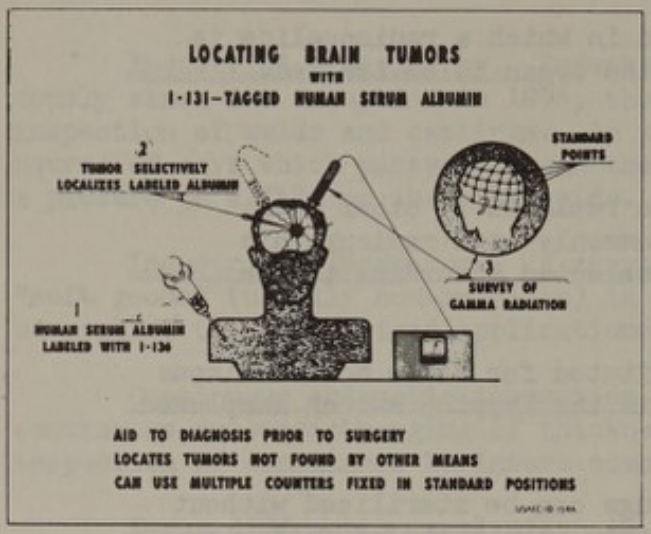
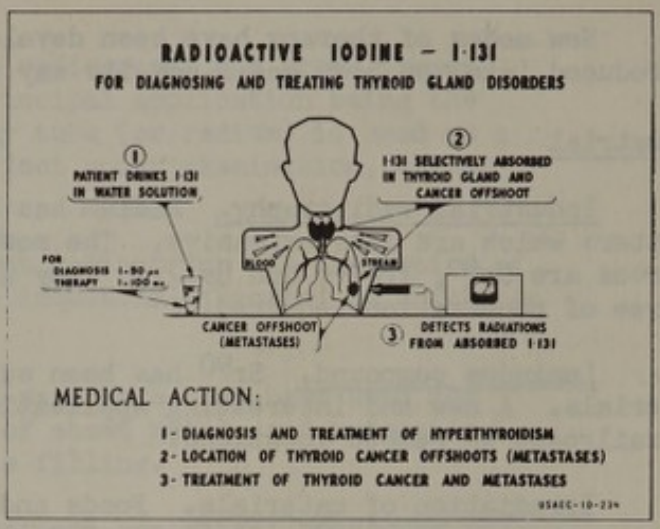
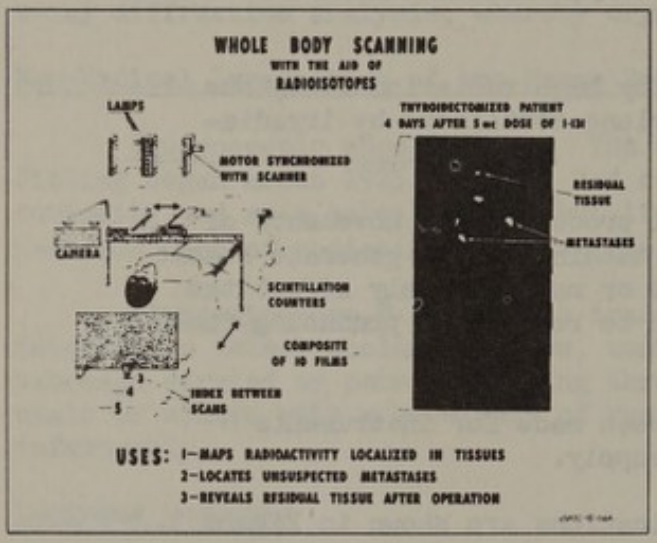
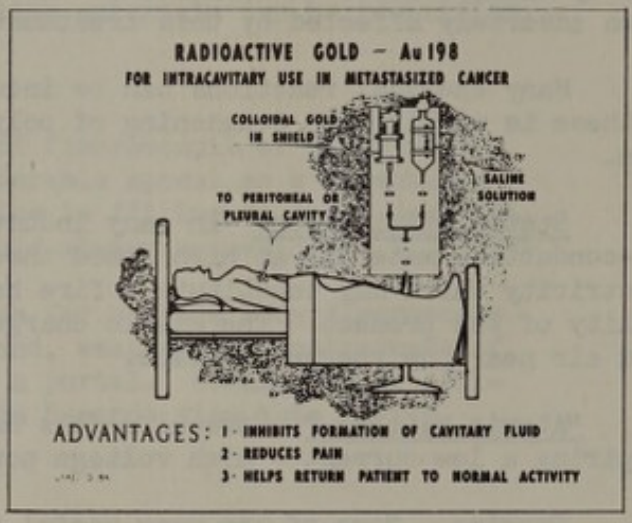
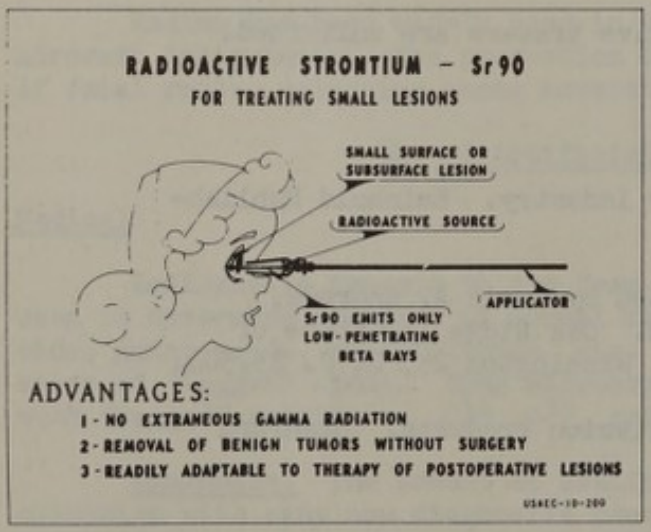
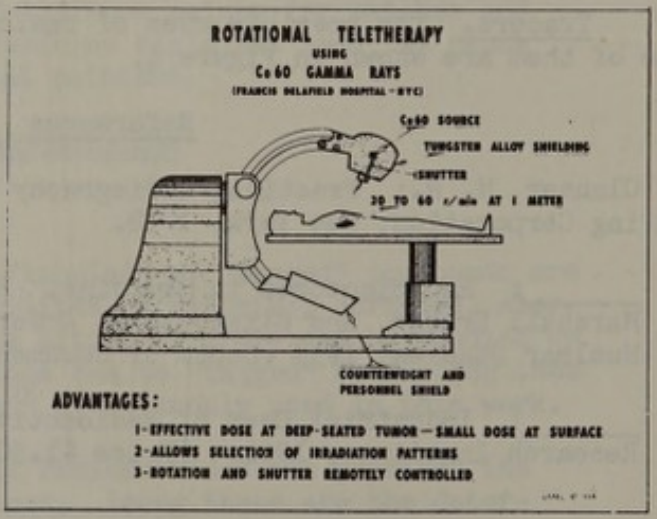


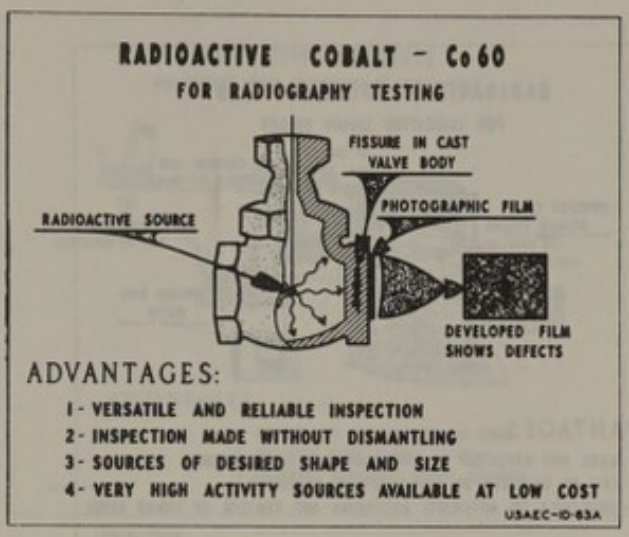



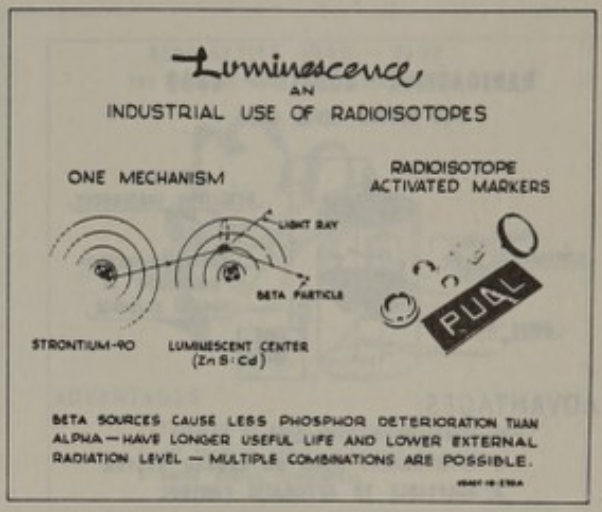



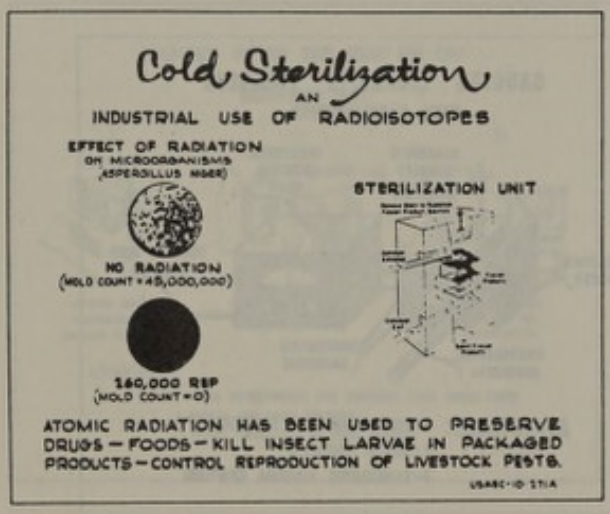



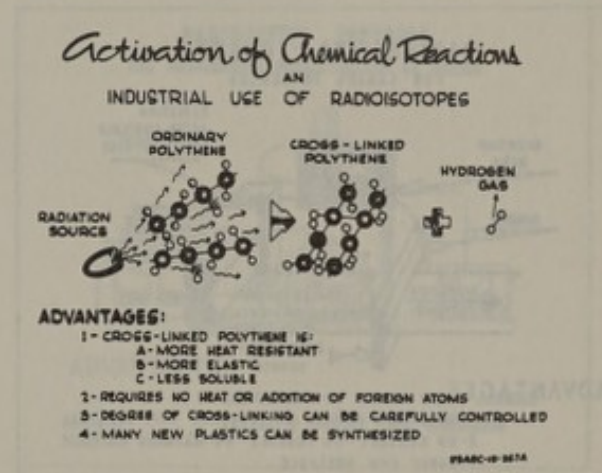

MEDICAL USES OF RADIONUCLIDES

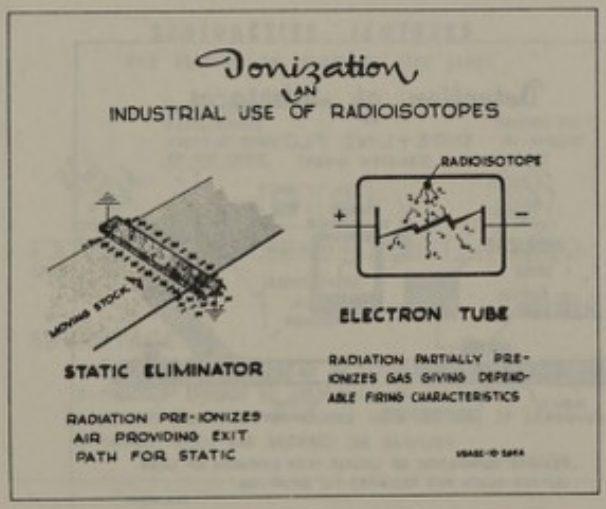
FIGURE 1

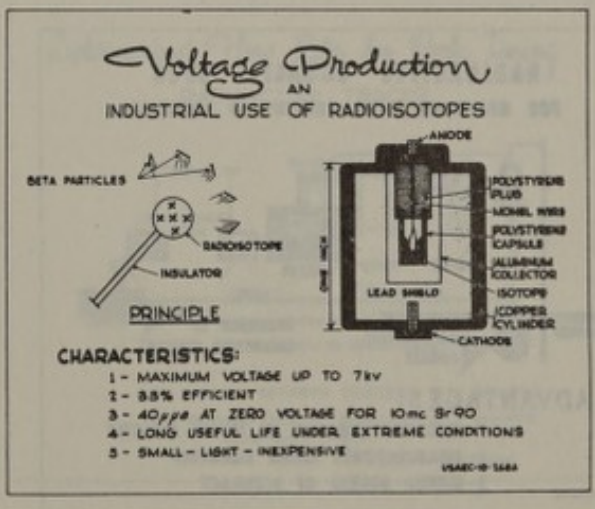


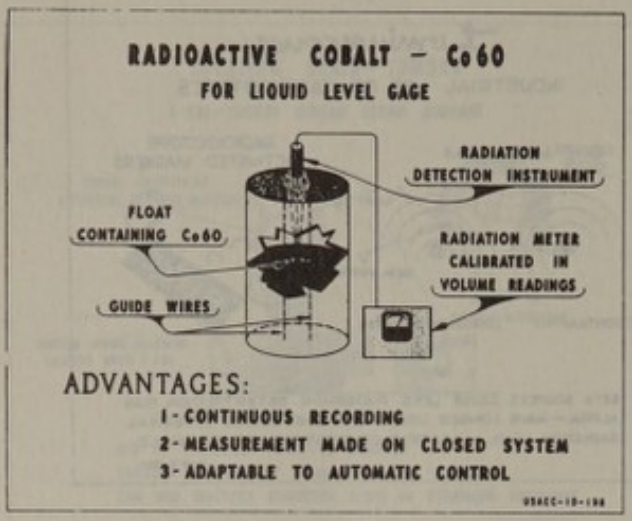



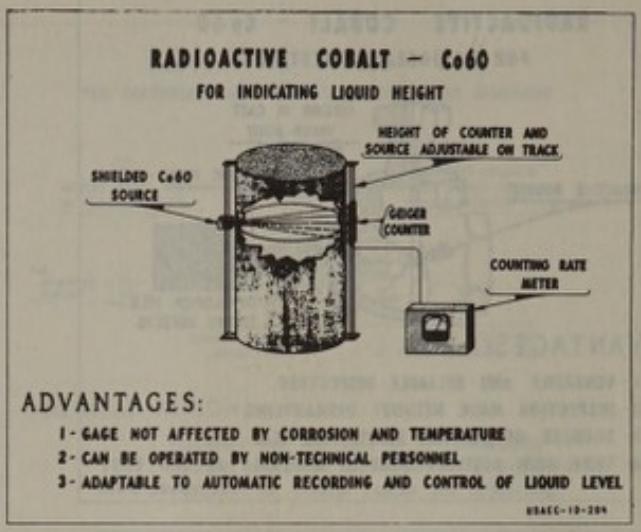



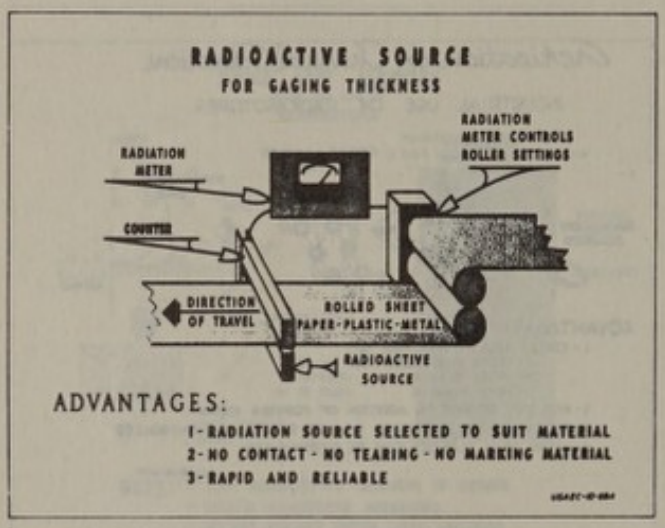



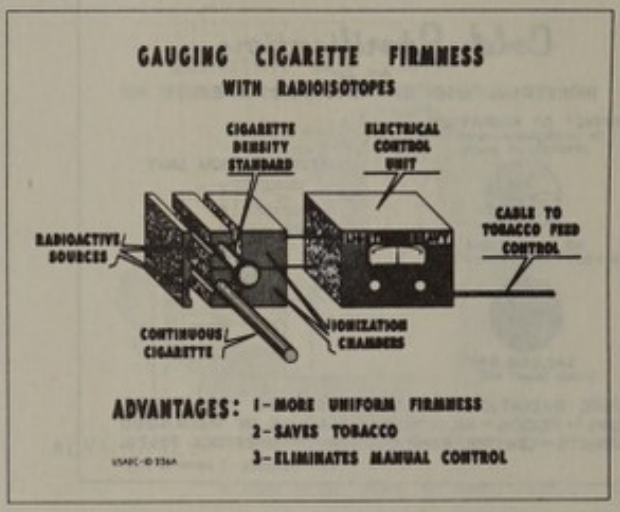

SOME INDUSTRIAL USES OF RADIONUCLIDES

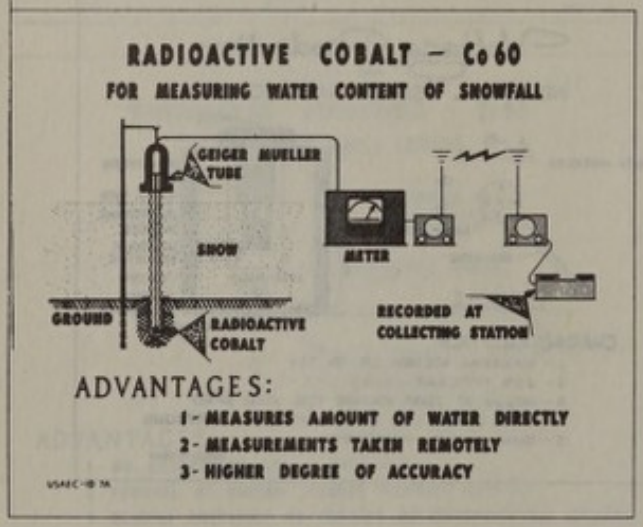
FIGURE 2

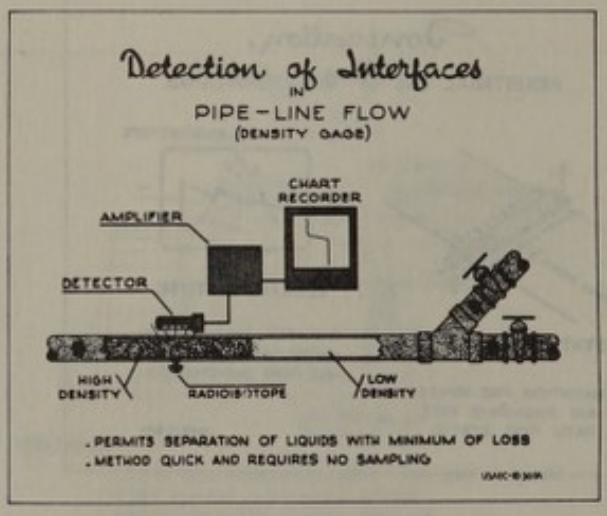


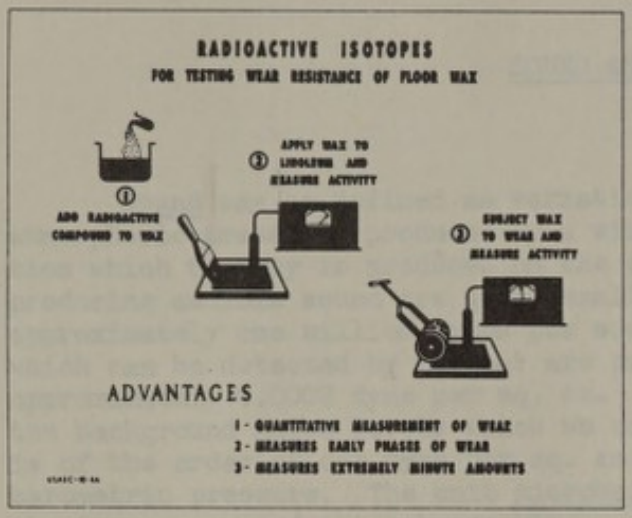



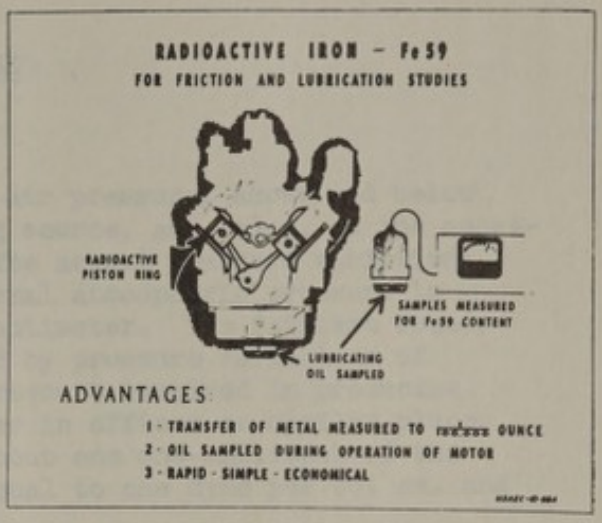



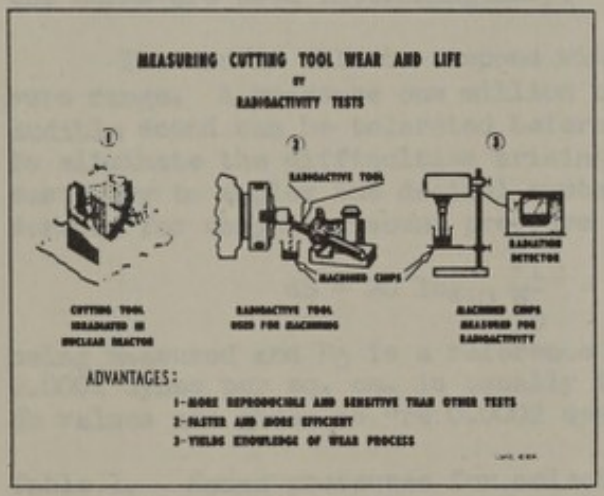



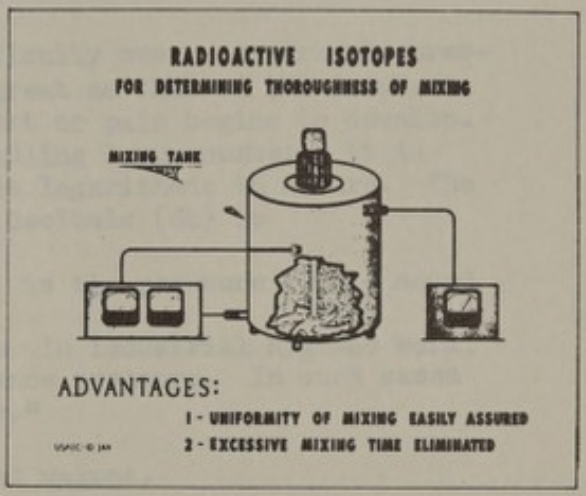

RADIONUCLIDES IN GAUGING

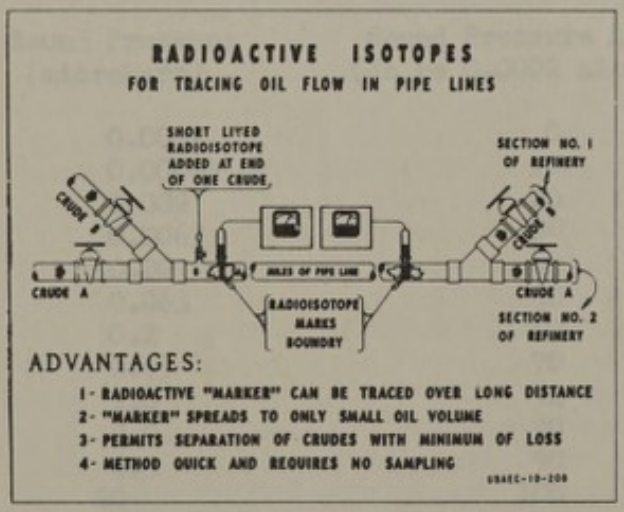
FIGURE 3

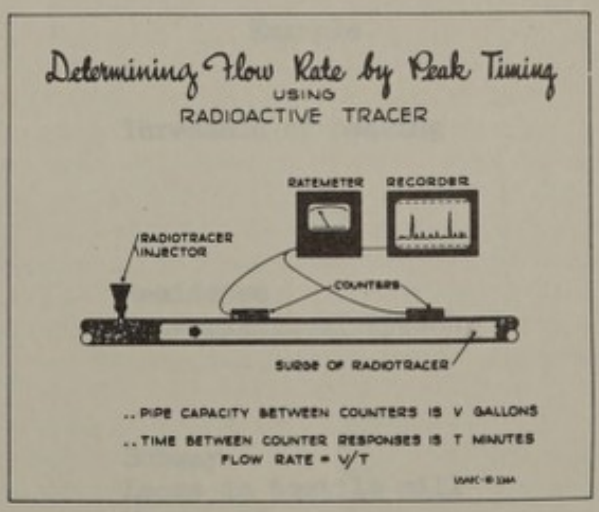



SOME INDUSTRIAL USES OF RADIOACTIVE TRACERS

FIGURE 4

# SOUND AND NOISE

Sound may be defined as variations in air pressure, above and below atmospheric pressure, produced by a vibrating source, and, also, as the sensation which thereby is produced on the ear. The actual pressure variations producing audible sound are quite small. Normal atmospheric pressure is approximately one million dynes per square centimeter. The faintest sounds which can be detected by the ear are produced by pressure variations of approximately 0.0002 dyne per sq. cm. The pressure involved in producing the background noise levels which we encounter in offices or similar places is of the order of one dyne per sq. cm. or about one one-millionth of the barometric pressure. The unit microbar is equal to one dyne per sq. cm. and the terms are used interchangeably.

The ear is able to respond without difficulty over a remarkable pressure range. A pressure one million times as great as that of the faintest audible sound can be tolerated before discomfort or pain begins to develop. To eliminate the difficulties arising from handling large numbers, it is customary to employ the decibel system which is logarithmic in nature. The formula for computing sound pressure level in decibels (db) is

$$db = 20 \log_{10} \frac{P_1}{P_0}$$ where $P_1$ is the pressure of the sound being measured and $P_0$ is a reference pressure. In industrial hygiene work, 0.0002 dynes per sq. cm. is usually the reference pressure. In such cases db values are given as "re 0.0002 dynes/sq. cm."

Table 1. - Sound pressures for selected decibel values.

| Sound Pressure (microbars) | Sound Pressure Level (db re 0.0002 microbar) | Example |
|---|---|---|
| 0.0002 | 0 | Threshold of hearing |
| 0.00063 | 10 | |
| 0.002 | 20 | |
| 0.0063 | 30 | |
| 0.02 | 40 | |
| 0.063 | 50 | Residence |
| 0.2 | 60 | Conversational speech |
| 0.63 | 70 | |
| 1.0 | 74 | |
| 2.0 | 80 | |
| 6.3 | 90 | Subway |
| 20 | 100 | Looms in textile mill |
| 63 | 110 | Woodworking |
| 200 | 120 | Hydraulic press |
| 2000 | 140 | Jet plane |

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Since the decibel scale is logarithmic, decibel values cannot be added directly. If the values for two sounds measured separately are known, the approximate sound pressure level that would result from combining them is obtained from the following table:

| Difference between levels in db | No. of db to be added to higher level |
|---|---|
| 0 | 3 |
| 1 | 2.6 |
| 2 | 2.1 |
| 3 | 1.8 |
| 4 | 1.5 |
| 5 | 1.2 |
| 6 | 1.0 |
| 7 | 0.8 |
| 8 | 0.6 |
| 10 | 0.4 |
| 12 | 0.3 |
| 14 | 0.2 |
| 16 | 0.1 |

In combining more than two sound pressure levels, the two highest levels should be combined first by this procedure. The total thus obtained is next combined with the highest remaining level, and this procedure is continued to completion.

The number of cycles of pressure variation per second is known as the frequency. The young, healthy ear can detect frequencies over a range from about 16 cycles per second (cps) up to 20,000 cps. The ability to hear the higher frequencies is usually lost somewhat with advancing age. Most speech is in the range between 250 cps and 3000 cps.

The ear does not respond the same to all types of sounds. Therefore, sounds of equal intensity may not seem equally loud. At sound pressure levels near 100 db, frequencies between 20 cps and 1000 cps sound equally loud. At lower sound pressure levels the lower frequency sounds do not seem as loud as the 1000 cps tone. For example, a pure tone of 70 cps having a sound pressure level of 50 db does not sound any louder than a 1000 cps pure tone having a sound pressure level of only 10 db. At sound pressure levels below 40 db, frequencies below 80 cps are inaudible.

Curves showing the sound pressure levels and frequencies required to produce similar loudness levels have been developed through tests on trained observers. These curves are called loudness - level contours. Loudness level is expressed in phons, and is numerically equal to the sound pressure level in db, re 0.0002 dynes/sq. cm., of a 1000 cps pure tone which sounds equally loud to a normal observer.

The sone is the unit of loudness. By definition a 1000 cps tone 40 db above the listeners hearing threshold (or any sound having a loudness level of 40 phons) produces a loudness of 1 sone. The loudnesses of other

sounds are compared with this value. Thus, a sound which seems twice as loud to that listener is said to have a loudness of 2 sones.

Through the relationships of decibels, phons and sones, it is possible to translate, at least to some extent, sound pressure levels, which can be measured, to the ear's response in terms of loudness.

Sound, as encountered in work places, rarely consists of pure tones. Instead, it is a composite of many frequencies, each of which contributes to the total resulting sound pressure level. Since, as has been pointed out, the ear does not respond equally to different frequencies, sounds having equal sound pressure levels may not seem equally loud. Current belief is that some frequencies at high levels may also be more injurious to the ear than other frequencies at similar levels. It is evident, therefore, that knowledge about the frequency distribution is important. The amount of detailed information required varies with the problem and determines the techniques to be employed in analyzing the sound. Instrumentation for such analyses is discussed in another section.

For the industrial hygienist, it is usually sufficient to break the sound spectrum into eight parts, each of which is one octave wide. (The parts at each end may be extended beyond one octave, usually without noticeable effect.) In an octave the highest frequency is twice that of the lowest.

Figure 1 illustrates data obtained from making octave band analyses of two different noises, each of which has the same total sound pressure level (98 db). One noise, however, is fairly uniformly distributed through the eight octave bands, while the other is concentrated largely in one or two octaves. This information is helpful, not only in comparing their loudness, but also in evaluating potential hazard to hearing. It also may be useful in developing engineering methods for reducing the noise.

Noise is defined as unwanted sound. If severe enough it can produce hearing loss. This may be either temporary or permanent in nature, depending upon the length and severity of the noise exposure. In addition, noise interferes with communication. It also can influence behavior.

Being unwanted, it is natural that efforts be made to eliminate or, at least, regulate noise. This brings about problems of definition and demands for criteria to use as a basis for evaluation. Such problems are very complex. Some sounds, such as music, may be unwanted at certain times, but desirable at others. Opinions as to what is music differ, however. The objections to the noise of fire sirens are counterbalanced by the need for such alarms. Many who appreciate rapid travel provided by aircraft object to the noise such planes produce in residential areas. These examples illustrate why criteria for community noise levels will be slow in developing.

In industry standards are wanted which will provide protection against hearing loss. These likewise are not yet at hand. More needs to be learned about the effects of various types of sound upon the ear, the time required for damage to take place, the reversibility of any apparent loss, and the importance of individual susceptibility to noise. The problem is further complicated by the effects of age on hearing. Loss due to age is similar to that produced by noise. Difficulty in distinguishing between these creates additional problems where compensation is involved.

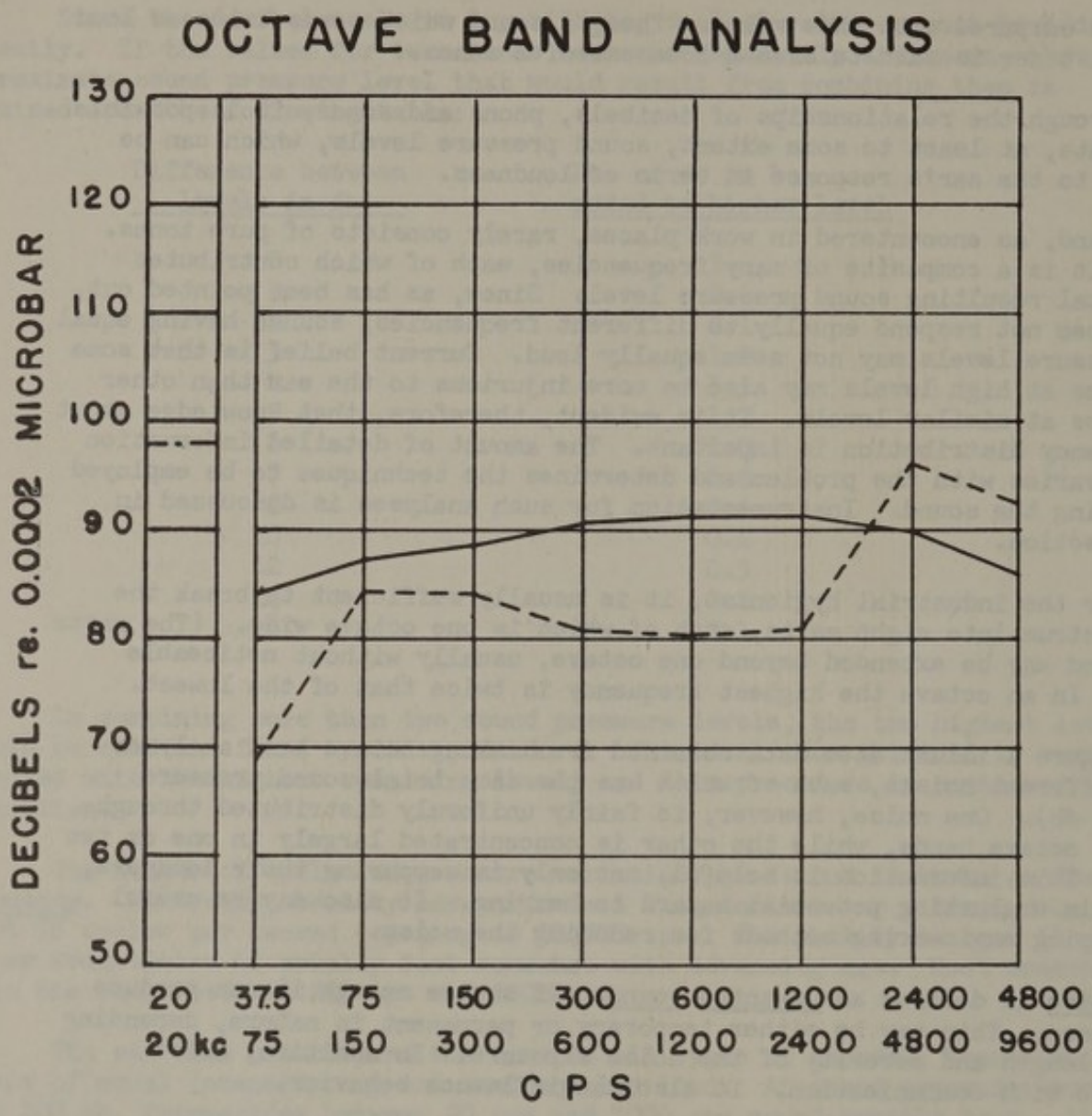


Figure 1.

Communication is essential in places of employment, but again, the levels of acceptability vary with the nature of work involved. Being able to communicate by shouting may be satisfactory when doing maintenance work on certain machinery, but, on the other hand, having to raise the voice slightly, because of ordinary office noises may be quite undesirable for a conference room. Conversely, there are types of work that may be carried on more efficiently by maintaining the background noise level above a certain point, thus shielding the worker from stray sounds which could be distracting.

Still different criteria are required for special situations, such as rooms for audiometric testing or studios for recording or broadcasting.

Guide lines for dealing with these various types of noise problems are gradually being developed. These are not yet precise enough for most problems, however. Until satisfactory criteria become available, the industrial hygienist should employ the same principles as he would in coping with other potential hazards which are not fully understood.

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# NOISE MEASUREMENT

## Types of Instruments

In making noise measurements for industrial hygiene work the two functions normally measured are sound pressure level and the distribution of energy over the frequency spectrum. There is a wide assortment of equipment which can be used and the choice of equipment used will depend on the type of noise and the purpose of the measurements.

### Sound Survey Meter

The Sound Survey Meter is a pocket-size instrument which consists of a non-directional microphone, a continuously-adjustable calibrated attenuator, an amplifier with three weighting networks and an indicating meter.

The instrument has a function switch with off position, A, B and C weighting network positions, and filament and battery check positions. It has a continuously variable attenuator and an indicating meter. The sound pressure level is the sum of the attenuator setting and reading from the indicating meter.

Even though the sound survey meter does not meet American Standards Association specifications it is a good instrument for rapid screening and surveying a large area or number of locations.

### Sound Level Meter

The sound level meter is the basic instrument for noise measurement. This meter consists of a microphone and an electronic circuit consisting of an attenuator, an amplifier, a series of frequency response networks (weighting networks), and an indicating meter. The attenuator in the circuit controls the current within limits which can be handled by the indicating meter, which is calibrated in decibels. The value obtained is the root-mean-square sound-pressure level expressed in decibels re 0.0002 dynes per sq. cm.

Sound level meters are available in the range of 20 to 180 decibels (db) (re 0.0002 dynes/sq. cm.). In the newer sound level meters the amplifier has a flat frequency response from 20 to 20,000 cycles per second (cps), and the over-all response will depend on the microphone used. The design criteria for sound level meters (American Standards Association, 224.3 - 1944) permit large tolerances at both low and high frequencies. This emphasizes the need for the proper calibration of each instrument.

Three weighting networks, A, B, and C are provided. The purpose of these is to provide a number which gives an approximate evaluation of the total loudness level. Human response to sound varies with frequency and intensity. The ear is most sensitive to the middle frequencies and least sensitive to the low and high frequencies at low intensities. On the other hand, at high intensities there is less difference in response at various

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frequencies. The three weighting networks provide a means of compensating for these variations in human response.

The A network is less sensitive to low frequencies and is intended for sound pressure levels below 55 db. The B network is an intermediate step for the range of 55 to 85 db. The C network has a flat response and is used for everything above 85 db. The C network is the only one which indicates the actual sound pressure level.

Microphones

The microphone is by far the most important part of the equipment, since the quality of the final result can be no better than the signal produced by the microphone. The function of the microphone is to pick up the sound energy and convert it to electrical energy, which is fed to the electronic circuit.

The frequency response, sensitivity, directionality, and range of a sound level meter are primarily determined by the microphone; therefore, its importance cannot be overemphasized.

At the present time there are three major types of microphones in general use for the measurement of sound. Each has its own advantages and disadvantages, and selection will depend on the requirements of the job to be done.

Crystal microphone. The Rochelle salt crystal microphone is usually supplied as standard equipment on sound-level meters sold today. The reasons for this are its low cost, ruggedness, availability and high sensitivity. It has a frequency range of 20 to 8000 cps and an intensity range of 24 to 150 db. Its frequency response varies widely with the incidence of the sound and the response is far from flat above 2000 cps.

One of the disadvantages of the crystal microphone is its sensitivity to heat and humidity. Temperatures above 115° F may damage the crystal and temperatures above 125° F will destroy it. Also, there is a reversible change in sensitivity produced by temperature change. When an extension cable is used and the input impedance of the meter is high, a temperature correction is necessary.

Dynamic microphone. This is probably the second type most commonly used. It operates on the principle of a coil moving in a magnetic field, resulting in an induced voltage. This type of microphone has a dynamic range of 20 to 140 db and a frequency response of 40 to 10,000 ± 5 db. Its frequency response is less erratic than the crystal microphone; but the frequency response drops off sharply below 40 cps.

The dynamic microphone has the advantage of low self-noise, permitting noise measurements down to less than 20 db in an octave band. The temperature sensitivity is small up to 180° F, and it can be used with long extension cables without correction. This microphone cannot be used in strong magnetic fields such as those around large transformers, generators, and electric arc welders. Care must be taken to avoid getting the microphone wet because of the possibility of internal shorts.

Condenser microphone. It is the least commonly used of the three microphones. It consists of two plates which act as a condenser; one is a thin diaphragm, the other a solid backing. Movement of the diaphragm changes the capacitance of the condenser in proportion to the displacement of the diaphragm. This change in capacitance produces an electrical signal which is proportional to the sound pressure level.

This microphone has the best frequency response of all types available. It is essentially flat from 20 to 8,000 cps and responds within 3 db to 14,000 cps. The dynamic range is from 40 to 145 db in the standard model with special models available with an upper limit of 200 db. Its temperature sensitivity corresponds to that of a dynamic microphone. It can be used at higher temperatures and with longer cables.

Disadvantages are its high cost, high self-noise, limited humidity range, and additional electronic complexity due to the preamplifier which is required. High humidities may increase internal noise but this can be corrected by keeping the microphone in a desiccator when not in use.

## Noise Analyzers

When the sound to be measured is complex, consisting of a number of tones, or having a continuous spectrum, the single value obtained from a sound level meter reading often is not sufficient for analytical purposes. It may be necessary to determine the sound pressure distribution according to frequency.

In general, there are three types of noise analyzers: (1) constant bandwidth, (2) constant-percentage narrow band, and (3) octave, half octave, and third octave bandwidth.

Constant bandwidth. The first of these has a fixed bandwidth that is a specific number of cycles wide. Common bandwidths range between 5 and 200 cps. These are essentially laboratory instruments requiring line voltage for operation. Because of the transient nature of industrial noise and the narrow bandwidth of the analyzer, this instrument has very little use in industrial hygiene work.

Constant-percentage bandwidth. The constant-percentage narrow band analyzer has a bandwidth which is a fixed percentage of the mid-band frequency. Thus, at low frequencies the bandwidth will be only a few cycles, while at high frequencies the bandwidth will cover a wide range of frequencies. This instrument has essentially the same limitations as the constant bandwidth analyzer but it can be used in some noise control problems.

Octave band analyzer. The most practical and widely used analyzer for industrial noise studies is the octave band analyzer. As indicated by the name, the upper cutoff frequency is twice the lower cutoff frequency. For a half-octave analyzer the upper cutoff frequency is $\sqrt{2}$ times the lower, and for a third-octave analyzer the ratio is $\sqrt[3]{2}$. A commonly used set of octave pass bands in commercial equipment is 20 to 75, 75 to 150, 150 to 300, 300 to 600, 600 to 1200, 1200 to 2400, 2400 to 4800, and in excess of 4800 cycles. This analyzer provides sufficient data on industrial noise to evaluate the

physiologic significance and to provide a basis for noise control. The small number of bands makes it possible to perform an analysis in a reasonable length of time, and the bands are wide enough to reduce the problem of transient components affecting the stability of the meter.

The analyzer may be equipped with one of two types of filters. In one type there is a choice of eight pass-bands and the selection is made by a single switch. The second type consists of a low cutoff filter, which filters out all frequencies below the setting of the dial, and a high cutoff filter, which filters out all frequencies above the setting of this dial. It is possible to make measurements in bands of one octave or multiples of an octave to cover the complete spectrum using the second type of analyzer. The attenuator, which is 50 db in 10 db steps, and the amplifier serve the same purpose as in the sound level meter. The meter has a 16 db range. The sound pressure level within the chosen band is the sum of the sound level meter attenuator setting, the analyzer setting, and the analyzer meter reading. The analyzer has a rheostat for adjusting the indicator to correspond to the meter reading on the sound level instrument when the analyzer is set to the over-all position.

All octave band analyzers are equipped with an output jack permitting the use of headphones, recorders, or oscilloscopes.

Accessories

A number of accessories are available for use with sound level meters and frequency analyzers to give additional information about the noise.

Impact noise analyzer. This instrument operates directly from the output of the sound level meter or frequency analyzer. It can be used to measure the peak level and duration of impact noise.

Through the use of electrical storage systems, three characteristics are measured by the analyzer for every impact noise. These are: the maximum instantaneous level, and average level, and a continuously indicating measure of peak sound levels. The values can be read individually on the same meter by means of a selector switch. A reset position of the selector switch restores the meter to its pre-reading condition.

Cathode-ray oscillograph. This is a useful means for observing the wave form of a noise. It is useful also for observing many general noise patterns, particularly short duration or impact noises. The oscillograph can be operated from the output signal of a sound level meter, octave band analyzer, or magnetic tape recorder. It can be used to measure the peak amplitude, the rate of decay, and the shape of a wave.

A cathode-ray oscillograph with a sweep rate extending down to at least 2 seconds and a long persistence screen is the type most useful for acoustic measurements. If the wave form is to be photographed a short persistence screen should be used. A five-inch oscillograph tube is normally used for this purpose.

The output of the sound level meter, sound analyzer or magnetic tape recorder are connected to the vertical input of the oscillograph. The

controls are set as explained in the instruction manual for the oscillograph. One way to set the gain of the vertical amplifier is to feed a reference signal of 60 cps or 400 cps to the sound level meter. The reading on the meter is noted and the vertical gain is adjusted to obtain a peak-to-peak deflection according to the table below for a five-inch screen.

| Meter reading db | Peak-to-peak deflection inches |
|---|---|
| 0 | 1 |
| 1 | 1.12 |
| 2 | 1.26 |
| 3 | 1.41 |
| 4 | 1.59 |
| 5 | 1.78 |
| 6 | 2.0 |
| 7 | 2.24 |
| 8 | 2.51 |
| 9 | 2.82 |
| 10 | 3.16 |

Once set, the gain control of the oscillograph should not be changed, adjustments being made with the attenuator on the sound level meter to keep the maximum deflection within the range of the table above.

The peak-to-peak sound-pressure level for a noise is the decibel value obtained from the table above plus the sound level meter attenuator setting plus 9 db.

Magnetic tape recorder. The magnetic tape recorder can be used for recording noise in the field with subsequent analysis in the laboratory. Detailed and repeated studies can be made or the tape can be kept for later analysis or comparison purposes.

If meaningful measurements are to be made from a recording, a high quality recorder must be used. It should have a flat frequency characteristic over a wide range, low hum and noise level, low non-linear distortion, constant speed drive, and good mechanical construction. A tape speed of 15 inches per second is recommended, since these characteristics are more readily obtained and maintained at high tape speeds.

The gain control should be set according to instructions supplied with the recorder. After it is set a reference signal should be recorded, such as the signal from an acoustical calibrator. Whenever the gain control is reset, a reference signal should again be recorded.

When an analysis is desired a representative sample should be selected and that portion of the tape is spliced to form a loop so it can be repeated continuously. If the absolute level needs to be known also, a sample of the recorded reference signal should be measured with the same control settings as were used for the original noise recordings.

It might appear that the only instrument needed in the field would be a good tape recorder; but in many situations it will be better to make the

analysis in the field with the usual sound equipment to make certain that the desired data have been taken.

Graphic level recorder. The graphic level recorder provides a permanent record of the noise characteristics by tracing a graph on a moving paper. It can be operated from the output of the sound level meter or the octave band analyzer to record the level of a noise as a function of time. It is subject to the same limitations as the sound level meter in the analysis of noise of short duration.

When using the graphic level recorder it must be calibrated with an acoustical calibrator. The gain control should be adjusted according to instructions supplied by the manufacturer.

## Acoustic Calibrator

The acoustic calibrator provides a simple method for making an over-all acoustical check of the sensitivity of a sound level meter including its microphone.

It consists of a small loud-speaker mounted in an enclosure which fits over the microphone of the sound level meter. The enclosure is designed in such a way that the measurements can be repeated with a high degree of accuracy.

The calibrator can be operated from the output of an audio oscillator or random noise generator. The designated operating voltage of the supplied signal and the sound level output will be found in operating instructions furnished by the manufacturer when the calibrator is purchased.

## Field Measurements

Evaluation of a noise problem depends upon accurate and meaningful measurements. Even though the equipment is electronic and may appear to be simple to operate, one should use it with caution and be on the alert to detect errors in the operation of the equipment and the influence of the environment.

In general, there are three types of noise studies and the equipment used will depend upon the type of study being made. The simplest of the three is a screening survey which can be done with a sound survey meter or sound level meter. This involves making a number of measurements in an area for rapid evaluation of the problem and determining where a more detailed study is needed.

The second type is a study to determine the characteristics of, and to evaluate the potential effects of a noise. This involves the use of a sound level meter and a noise analyzer in order to determine the sound pressure distribution with frequency. For industrial work, an octave-band analyzer would probably be used.

The third type of study is one for research or noise control purposes. This might require a large number of measurements and analyses in the field

and also magnetic tape recordings to be analyzed in the laboratory. A sufficient number of samples should be taken to determine the exact nature of the source, including power and directionality.

Operation of Instruments

The sound level meter has an on-off switch, a means of checking battery voltages, a weighting network switch, a fast-slow meter response switch, an attenuator switch with steps of 10 db and an indicating meter (16 db range). The instrument is turned on, batteries are checked, the instrument is permitted to warm up for a few minutes, and the attenuator is adjusted until the indicating meter is on scale. The sound pressure level is the sum of the attenuator setting plus the meter reading.

The octave-band analyzer operates from the output of the sound-level meter. The analyzer has an on-off switch, battery voltage check switch, a fast-slow meter response switch, an attenuator switch, and a switch permitting the selection of the band to be measured. The analyzer is connected to the sound-level meter by a cable, both instruments are turned on. The batteries should be checked. The analyzer band selector switch is set at 20 to 20,000 cps and the two meter readings should agree. The over-all sound pressure level is recorded, then the band selector switch is set to the 20 to 75 cps octave-band, and the attenuator on the analyzer adjusted until the indicating meter is on scale. The sound level in the octave band is the sum of the attenuator setting of the sound-level meter, the attenuator setting of the octave-band analyzer and the octave-band analyzer indicating meter observing the appropriate signs.

Maintenance of Equipment

Because of the nature of the equipment, one should constantly be on the look-out for faulty operation. The equipment should be checked before it is taken from the laboratory because of the availability of a quiet location and availability of other test instruments if repair is necessary. The following steps should be taken:

1. The instruments should be connected, turned on, and allowed to warm up a few minutes.

2. Check batteries.

3. Make electrical and acoustical calibration.

4. Make octave-band measurements of a convenient wide band steady noise.

5. Remove the microphone and make electrical background measurements.

6. Recheck calibration.

If any of the above steps yield unsatisfactory results the instrument instruction manual should be consulted for necessary adjustments or repairs. If the first three steps are completed satisfactorily, this usually means

that the equipment is operating properly. Step 4 can expose a defective octave-band which might be overlooked in making measurements in the field. Step 5 will indicate whether there is an excessively noisy tube or faulty oscillating amplifier. This can be corrected by replacing the defective tube. Step 6 could indicate weak batteries if the calibration has changed by more than 1 db. If it has changed, the batteries should be replaced before making a number of measurements.

Precautions for Handling Equipment in the Field

Most sound level meters available today come equipped with a Rochelle salt crystal microphone which can be damaged by excessive heat and humidity. The microphone should not be subjected to temperatures above 115° F; it should never be left in the trunk or inside a closed automobile on a hot summer day.

Since electronic equipment can be damaged by intense shock, extreme care must be taken in shipping or transporting these instruments by commercial carrier.

Calibration of Equipment in the Field

On reaching the area where the measurements are to be made the six steps listed under Maintenance of Equipment should be repeated, preferably before the equipment is taken into a noisy area. The acoustical calibration should be performed in an area where the background noise, with the calibrator in place, is 10 db less than the calibration signal.

The acoustical calibrator can be used also to determine the temperature corrections needed for an extension cable between the microphone and sound level meter. The reading should be taken without the cable and then with the cable inserted. The difference is the correction to be added to the readings obtained with the cable. This should be done after the microphone temperature has reached the room temperature where the measurements are to be made. This correction is a function of ambient temperature of the microphone, but not a function of frequency.

If a large number of readings are made at one time, the batteries should be checked every two hours and the equipment calibrated a minimum of four times a day.

Selection of Sampling Locations

After it has been determined that the equipment is operating satisfactorily, the next step is to determine the sampling locations. This will depend on the reasons for making the measurements and other factors. If it is a preliminary survey to determine if a noise problem exists, then a large number of measurements are taken with the sound survey meter or sound level meter throughout the area, paying particular attention to the noise sources.

In the evaluation of noise exposure to individuals the microphone would be placed as near to the individual as possible without interfering with his work. It is also best to make several measurements with the sound level meter

in the area to determine if the sound pressure level is relatively uniform and if the measurements being made represent the individual's exposure.

Another type of survey is for noise control purposes. In this case, measurements would be made to determine the total acoustical output and directional characteristics of the source. Measurements at one point yield information about that point only and in that environment. In order to determine how a source differs in another environment, the acoustic power characteristics must be determined. A number of measurements must be made in a geometrical pattern about the source and at some distance from the source. This type of survey is basically an engineering problem and the measurements are made with particular attention to the physical characteristics of the source and environment and without regard to personnel in the area.

Sources of Error

The reliability of noise measurements depends upon the manner in which the instruments are used and their operating condition. The immediate recognition of faulty equipment is essential if accuracy is to be maintained.

Position of the microphone. In general, the location of the microphone is determined by the type of measurements to be made as was discussed under sampling locations.

The microphone should be located so that it is at the side of the observer or operator and not between the noise source and observer or operator. When making measurements around a single source large errors may result; but, in a large room with a number of sources, measurements are made at quite some distance from the source and the error would be small.

The sound field should be explored to make sure that the measurements are representative. The possible effects of obstacles upsetting the distribution of sound, particularly at high frequencies, should be kept in mind when making this exploration.

Wind will produce a low frequency noise which can be appreciable when using a microphone that is very sensitive to low frequencies. If it is necessary to sample in the wind, a wind screen can be made out of wire, forming a sphere about 18 inches in diameter.

Circuit noise. All vacuum tubes are affected by mechanical vibration. The tubes used in sound measuring equipment have been selected to be less sensitive to shock than the usual tubes. But at high noise levels some of these tubes may be vibrated to such an extent so as to produce extraneous noises in the equipment. A test for this is to disconnect the microphone from the sound level meter and observe whether there is a reading on the meter. If there is a reading, the meter can be placed on a rubber pad or rubber-tired cart which should eliminate the trouble. In very intense noise fields it may be necessary to remove the meter from the area and use an extension cable to the microphone.

When making measurements around electrical equipment a check should be made to see if there is an appreciable pickup of the electromagnetic field by the sound measuring equipment. This check is particularly important

when using a dynamic microphone. A pair of good quality earphones can be used to monitor the output of the sound level meter or octave band analyzer. The hum produced by the electromagnetic field will be of the same frequency as the power source, usually 60 cycles per second. The orientation of the instruments and microphone should be changed to determine if there is a difference in the meter reading; also, the microphone should be disconnected to check if the pickup is in the microphone or the instruments. If the pickup is in the instruments they can be removed from the area under study and an extension cable used; if it is caused by the dynamic microphone, a crystal or condenser microphone may have to be used.

When making noise measurements at low levels, the inherent circuit noise may be the limiting factor. A check for this is made by removing the microphone and substituting a capacitor of the proper size. The lowest reading that can be obtained with the sound level meter or analyzer is determined. This will be the lower limit of measurement.

Background. In some cases, when making measurements to determine the amount of noise generated by a particular source, it may be necessary to make corrections for background noise. If there is any evidence that the background noise is contributing to the noise being measured, a background measurement should be made with the noise source turned off. It is possible to make a rough correction for this background noise by applying the factors given in the table below:

| Total noise level less background level (db) | Subtract from the total noise level to get the noise level due to the source (db) |
|---|---|
| 10 | 0.5 |
| 9 | 0.6 |
| 8 | 0.7 |
| 7 | 1.0 |
| 6 | 1.2 |
| 5 | 1.6 |
| 4 | 2.2 |
| 3 | 3.0 |
| 2 | 4.3 |
| 1 | 6.9 |

## Recording Data

One important part of making noise measurements is the recording of sufficient data. Data that should be recorded will depend on the purpose of the survey, that is, whether it is a hearing loss, noise control, or nuisance problem. To a certain extent each noise survey is unique, and the report resulting from it must have its own pattern that fits the needs of the particular situation. There are, however, certain basic data which are essential and must be recorded in almost all surveys:

1. Equipment used for the measurements. Type and serial numbers of all microphones, sound level meters and analyzers

2. Corrections for measured values, such as cable, temperature, and acoustical calibration

3. The time and date of measurements are made and name of person conducting the study

4. Description of space in which the measurements were made such as, dimensions and nature of ceiling, walls and floor, and locations of windows and doors

5. Description of the noise source under test (primary noise source). This should include a clear description of the machine as to size, name-plate data, speed and power rating. Types of operations and operating conditions and number of machines in operation, locations of the machines and types of mountings

6. Description of secondary noise sources including location and types of operations

7. Noise control measures instituted, including the types and effectiveness of ear protectors

8. Over-all and band levels at each microphone position and the extent of meter fluctuation

9. The meter speed and weighting network used

10. Position of the microphone and the direction of the sound with respect to the microphone, tests for standing wave patterns and the decay of sound level with distance

11. Time pattern of the noise, that is, whether continuous, intermittent or impact

12. Personnel exposed, directly and indirectly

In recording data it is helpful to have a blueprint or sketch of the building on which locations of measurements can be noted. Frequently location of columns in a building can be used to form a grid system such as shown in Figure 1.

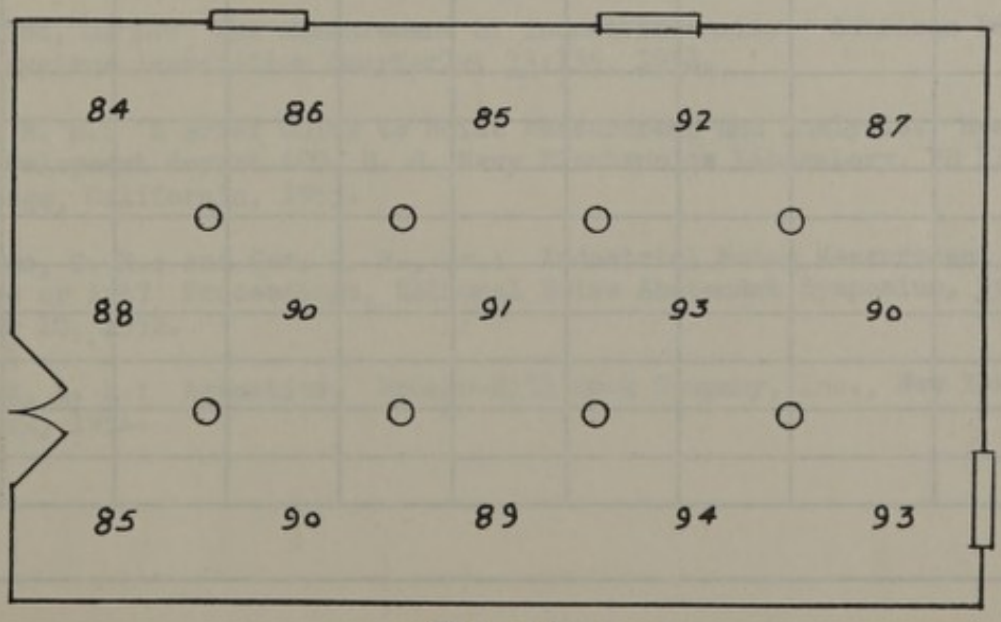


Figure 1.

It is helpful to have a survey data sheet on which the data can be recorded. A typical one is shown in Figure 2 which was provided by the Subcommittee on Noise in Industry of the American Academy of Ophthalmology and Otolaryngology.

## SOUND SURVEY

Page ___ of ___

Date: _____ Time: _____

Wind Velocity: _____ Wind Direction: _____

Sound-Level Meter: Type _____ Model _____ Serial No. _____

Microphone: Type _____ Cable Length _____

Analyzer: Type _____ Model _____ Serial No. _____

Other Equipment: _____

_____

_____

Location: _____ Sketch

_____

_____

_____

Calibrated: 60 cps: Acoustic: Corr. Factor:

| Location | Weighting Network | Over-all Level | Octave-Band Pressure Levels Re. .0002 microbar | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | 200–75 | 75–150 | 150–300 | 300–600 | 600–1200 | 1200–2400 | 2400–4800 | Above 4800 |
| 1 | | | | | | | | | | |
| 2 | | | | | | | | | | |
| 3 | | | | | | | | | | |
| 4 | | | | | | | | | | |
| 5 | | | | | | | | | | |
| 6 | | | | | | | | | | |
| 7 | | | | | | | | | | |
| 8 | | | | | | | | | | |
| 9 | | | | | | | | | | |
| 10 | | | | | | | | | | |
| 11 | | | | | | | | | | |
| 12 | | | | | | | | | | |

Remarks: _____

Recorded By: _____

## Analysis of Data

After a survey has been completed and before a report can be prepared, it is necessary to analyze the data. The necessary corrections must be made and the accuracy of the final results determined.

Corrections should be made for any errors produced by an extension cable or any other errors revealed by the acoustical calibration. If a calibration curve is available for the microphone being used, the appropriate corrections should be made. When measurements have been made to determine the sound pressure level of a particular source, corrections for background noise should be made if necessary.

With proper acoustical calibration the meter should be accurate within ± 1 db at the calibrated frequency. With electrical calibration, the meter should be accurate within ± 2 db at the middle frequencies. The average response curves for various microphones are probably within ± 1 db in the middle frequencies and from ± 2 db to ± 10 db in the first and last octave bands. Also, with proper precautions, the readings should be accurate within ± 2 db in all but the first and last octave bands.

## Preparation of a Report

The type of report will depend on the purpose of the survey and for whom the report is prepared. In general, it is better to present the data in the same form that is used in the field, that is, showing over-all and octave band levels. This may be plotted on ordinary linear graph paper with the db as the ordinate scale and the octave band levels as the abscissa, or it may be plotted on special graph paper shown on the next page.

The criteria that apply to the situation should be presented and then the appropriate recommendations made.

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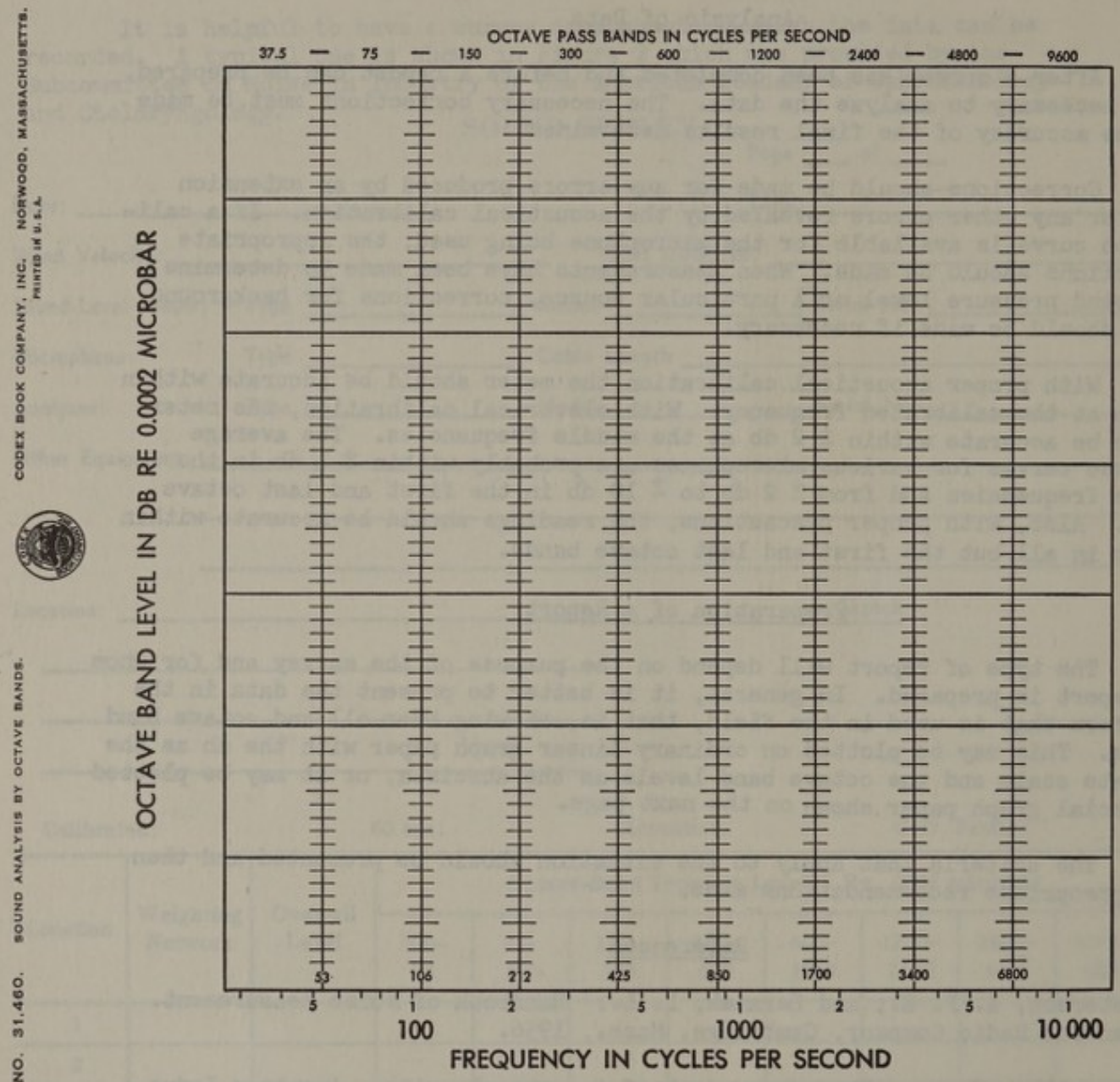


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# ILLUMINATION AND ULTRAVIOLET RADIATION

## Introduction

This will be a brief discussion of the industrial hygiene aspects of, and the measurement of, illumination and ultraviolet radiation. More complete discussions can be found in the Encyclopedia of Instrumentation for Industrial Hygiene(1) and other references to be cited.

## Illumination

Poor industrial illumination is the cause of worker eye fatigue resulting in decreased production, more rejects of finished products, and increased accidents. Furthermore, eyestrain over a prolonged period of time can cause a permanent reduction in acuity of vision.

Good illumination requires a sufficient quantity of light to permit the necessary detail to be seen with ease, and a high quality of light. Quality of light includes the factors of color, direction, brightness, brightness ratio (contrast), diffusion, uniformity of distribution, and glare. These factors have significant effects on visibility, and the ability to see easily, accurately, and quickly. Of the factors of light quality, glare is the least understood. "Glare may be defined as any brightness within the field of vision of such character to cause discomfort, annoyance, interference with vision or eye fatigue, or both. When the condition is caused directly by the source of the lighting, whether natural or artificial, it is described as direct glare. . . . Reflected glare is caused by high brightness images, or brightness contrasts reflected from shiny ceilings, walls, desk tops, materials, and machines or surfaces within the visual field. If the seeing task is on a shiny surface or the immediate surroundings are glossy or highly-polished surfaces, reflected images of bright light sources overhead or of luminaires with excessive brightness differences will tend to veil the detail of the task. This reflected glare is frequently more annoying than direct glare because it is so close to the line of vision that the eye cannot avoid it."(2) The effect of reflected glare for a given image brightness can be reduced by using higher levels of general illumination, thereby reducing the brightness contrast. While brightness contrast is most important at the visual task, the eyes function most comfortably and efficiently when the brightnesses within the remainder of the environment are relatively uniform. Modern lighting fixtures are designed to provide uniform background lighting. They usually direct about 25% of the light upward to be reflected from the ceiling. Clean, light colored walls and ceilings are necessary with such lighting.

Table I lists recommended maximum brightness ratios. Lower values would be desirable.

Prepared by: Morton Lippman - 57

## Table I

Recommended Brightness Ratios[1]

| | |
|---|---|
| 5 to 1 | Between tasks and adjacent surroundings |
| 20 to 1 | Between tasks and more remote surfaces |
| 40 to 1 | Between luminaires (or sky) and surfaces adjacent to them |
| 80 to 1 | Anywhere within the environment of the worker |

Recommendations for levels of illumination for specific industrial visual tasks can be found in "American Standard Practice for Industrial Lighting"[2] pp. 12-17; in "Industrial Health Engineering"[3] pp. 303-312; and in "IES Lighting Handbook"[4] 2nd Ed., pp. 9-63 to 9-70.

Table II is a listing of general recommended illumination levels. Note: Many of the current recommendations for levels of illumination and brightness ratios will probably be changed as a result of new data presented at the 2nd Symposium of the Illuminating Society's Research Institute held at Dearborn, Michigan, on March 3 and 4, 1958. These data are the result of over eight years' intensive research, including thousands of tests relating to threshold visibility, and they provide a more sound basis for specification of interior illumination levels. The 3rd edition of the IES Lighting Handbook, to be published in 1959, will no doubt contain new recommendations based on this information.

Table II - General Recommended Values of Illumination(2)

| | Current Recommended Practice Footcandles in Service (On Task or 30" above floor) |
|---|---|
| **Most Difficult Seeing Tasks** | |
| Finest Precision Work ..... | 200-1000* |
| Involving: Finest Detail<br>Poor Contrasts<br>Long Periods of Time | |
| Such as: Extra-fine Assembly; Precision Grading; Extra-fine Finishing | |
| **Very Difficult Seeing Tasks** | |
| Precision Work ..... | 100 |
| Involving: Fine Detail<br>Fair Contrasts<br>Long Periods of Time | |
| Such as: Fine Assembly; High-Speed Work; Fine Finishing | |
| **Difficult and Critical Seeing Tasks** | |
| Prolonged Work ..... | 50 |
| Involving: Fine Detail<br>Moderate Contrasts<br>Long Periods of Time | |
| Such as: Ordinary Bench Work and Assembly; Machine Shop Work; Finishing of Medium-to-Fine Parts; Office Work | |
| **Ordinary Seeing Tasks** | |
| Involving: Moderately Fine Detail ..... | 30 |
| Normal Contrasts<br>Intermittent Periods of Time | |
| Such as: Automatic Machine Operation; Rough Grinding; Garage Work Areas; Switchboards; Continuous Processes; Conference and File Rooms; Packing and shipping | |
| **Casual Seeing Tasks** | |
| Such as: Stairways; Reception Rooms, Washrooms and other Service Areas; Active Storage | 10 |
| **Rough Seeing Tasks** | |
| Such as: Hallways; Corridors; Passageways; inactive Storage | 5 |

*Obtained with a combination of general lighting plus specialized supplementary lighting. Care should be taken to keep within the general brightness ratios (indicated in Table I) and to avoid glare conditions when light colored materials are involved.

## The Importance of Maintenance in Lighting

Several factors tend to reduce the illumination level provided by a given group of lamps. The accumulation of dirt on the fixtures will reduce the light output, perhaps up to 50% if the area is very dirty, or if the lamps are not cleaned at regular intervals. Dirty walls and ceilings will also reduce the illumination level by reflecting less light. In addition, the useful output of some lamps decreases with age long before they burn out. Therefore, it is important that a regular maintenance program be set up, whereby the lamps and fixtures are cleaned, and dim and burnt out lamps replaced at suitable intervals.

## Instruments for the Measurement of Illumination

### Light Intensity Meters

Several kinds of light meters are commercially available. The ones most commonly used for lighting surveys are the G. E. pocket sized meters, and the Weston hinged-cell meters. Both types are available with cosine correcting elements. With cosine correction, a light meter responds equally to illumination from all directions instead of primarily to vertically incident radiation. The Weston hinged-cell meters have wider ranges than the G. E. pocket sized meters, but are more expensive.

For highly accurate illumination measurements special low-range sensitive and multi-cell light meters are commercially available.

### Brightness Meter

The most commonly used brightness meter is the Luckiesh-Taylor meter made by G. E. It has a battery operated internal light source, making it a self-contained unit. With it, the brightness of any surface can be measured within the range 0.0025 to 50,000 foot-lamberts. The instrument has two eyepieces. One is a focusing telescope which is sighted on the surface to be measured. In the center of this field are a pair of silvered trapezoids illuminated to a constant brightness by the internal light source. The brightness of the background is adjusted with a circular gradient filter until it matches the trapezoids in brightness. The second eyepiece views a scale which rotates with the gradient filter. At the proper setting of the filter, the brightness can then be read directly from the scale.

### Visibility Meter

The visibility meter is a device for determining the relative visibilities of visual tasks under various conditions of lighting and levels of illumination. It can also be used to determine the relative footcandles required by a variety of tasks to make them equal in visibility. The object whose visibility is being measured is viewed through variable filters which are adjusted until the object is just barely visible. This is the point of threshold visibility.

A new instrument, the visual task evaluator, was developed at the University of Michigan in 1957. However, in its present form it is used

for research purposes only. Results of tests with this instrument may bring about future modifications in the visibility meter.

Glare

Measurement of disability glare, either reflected from surfaces or direct from luminaires, has posed a problem because satisfactory field instruments are not commercially available. Research in this field has progressed and several meters have been developed for this purpose. These should be available in a few years.

## Ultraviolet Radiation

The most common source of industrial exposure to ultraviolet radiation is in welding operations. Newer welding techniques such as the inert-arc methods have been found to produce higher levels of ultraviolet than the conventional methods.(5) Other potential areas of exposure may be found in hospitals, pharmaceutical plants and other places where ultraviolet lamps are used for their germicidal action. Exposure to excessive amounts of ultraviolet light may cause conjunctivitis, corneal ulcers, or iritis.

The American Medical Association, Council on Physical Medicine, has proposed the following limits for exposure on faces of ultraviolet from bacteriocidal and germicidal lamps.

Maximum Daily Exposures

| Exposure Time per 24 hours | Intensity on Faces Milliwatts/ft.$^2$ | Exposure Time per 24 hours | Intensity on Faces Milliwatts/ft.$^2$ |
|---|---|---|---|
| 24 hr. | 0.1(3) | 2 hr. | 1.8 |
| 18 hr. | 0.2(3) | 1 hr.(1) | 3.6 |
| 12 hr. | 0.3 | 30 min. | 7.2(4) |
| 9 hr. | 0.4 | 10 min. | 21.6(4) |
| 6 hr. | 0.6 | 1 min.(2) | 216.0 |
| 4 hr. | 0.9 | 30 sec. | 432.0 |
| 3 hr. | 1.2 | 5 sec. | 2.6 watts/ft.$^2$ |

(1) Exposures (time x intensity) 3.6 milliwatt hrs./ft.$^2$
(2) Exposures (time x intensity) 216 milliwatt min./ft.$^2$
(3) Permissible intensity in hospital infant wards - one-fiftieth or one-hundredth that recommended for hospital air disinfection.
(4) Intensity recommended for hospital upper air disinfection - tolerated only 10 - 30 minutes if on faces of personnel.

A detailed discussion of this subject can be found in "Ultraviolet Radiation"(6) by Koller.

## Instruments for the Measurement of Ultraviolet

The measurement of ultraviolet light is considerably more complicated than the measurement of illumination. There are no broad range instruments, and if there were, their readings would have questionable value, since different wave lengths of ultraviolet have different effects. For instance 2537A° is the most effective bactericidal wave length, while 2967A° has the greatest erythemal effect. Therefore, in the measurement of ultraviolet, it is important to determine both the intensity and wave length of the radiation.

Commercially available instruments include the following intensity meters:

A. Germicidal attachment for G. E. pocket light meter. This attachment consists of a sandwich of fluorescent phosphor between a glass and a quartz plate. The difference between the readings with and without the attachment is a measure of ultraviolet intensity at 2537A°. The instrument is relatively insensitive, requiring about 35 microwatts per sq. cm. (milliwatts per sq. ft.) to get a difference reading of 1 footcandle.

B. Germicidal ultraviolet intensity meter (G. E.). This is a sensitive instrument for measuring intensity at 2537A°. It utilizes a cadmium photocell and an electrometer tube circuit. The instrument has three ranges, 1, 10, and 100 microwatts per sq. cm., and is battery operated.

C. RS Sunlamp Tester (G. E.) is used to test the output of sunlamps. It consists of a G. E. pocket sized light meter with a filter which responds to erythemal ultraviolet radiation. It would be difficult to interpret the readings of this meter for uses other than sunlamp testing, but it might conceivably be useful for comparison purposes in a study.

Other methods for measuring ultraviolet include photocells or films sensitive to certain wave lengths. By using a series of such cells or film, the radiation can be broken down into bands, and the intensity of each determined.

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# COMFORT VENTILATION

## Introduction

Very little air is usually needed for oxygen supply and carbon dioxide removal in occupied spaces. Odor control generally requires a greater quantity of air than do these basic physiological requirements and an outside air supply sufficient to remove odors, smoke, etc., should always be provided.

Dilution of pathogenic organisms has not received the objective attention which other factors in ventilation have been given. The British have done work in this field and their present standard sets the limit at 50 bacteria-bearing particles, as measured by a suitable quantitative counting method, per cubic foot of air. American opinion is divided: some authorities feel that an upper respiratory bacteria, hemolytic streptococcus, should be used as an index of bacterial air pollution, others feel that no standards are necessary since relationships between bacterial pollution and incidence of infection have not been demonstrated.

Methods for the reduction of numbers of air-borne bacteria (ultraviolet irradiation and glycol vapor) have proved to be not too effective. These agents work to some extent on non-dusty air, but do not work effectually on normal, uncontrolled air. In some cases, installations for these purposes may do more harm than good, ultraviolet sources may sometimes generate ozone and glycol decomposes at its boiling point to produce aldehydes.

Ventilation codes and ordinances must be observed. Most cities and states which have codes or ordinances require a minimum of 10 to 15 cfm of outside air per person in schools, theatres, auditoria, etc. Always check legal requirements and restrictions before making recommendations for installations. If there are no mandatory criteria, it is well to inquire of state or local societies of heating and ventilating engineers to determine their established procedures.

Many space standards developed in attempts to control communicable disease are based on the concept of floor area per person. It appears that the incidence of disease is more nearly related to number of persons per living unit than to area allowed per person in the unit.

## Physiological Principles

### Heat Balance

It should be emphasized that it is never necessary to heat the human body. The purpose of heating or cooling of air in occupied spaces is to help maintain the temperature of the body by controlling its rate of heat loss. The body always produces heat which must be dissipated to avoid fever and death. The body energy balance may be expressed as:

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$$M = S + E + R + C + W$$

where:
- M = total rate of metabolism
- S = rate of heat storage in the body
- E = rate of evaporative heat loss
- R = rate of radiation heat loss
- C = rate of convective heat loss
- W = rate of external work expressed as heat

M is the sum of rest metabolism (about 400 BTU/hr for adult) and increased metabolism for activity over and above the rest state. The human body is about 20% efficient in accomplishing mechanical work. For every unit of mechanical work done, about four times its equivalent as heat must be lost from the body in addition to the heat produced in the rest state.

A more detailed heat balance equation is:

$$M_r + M_i = A_c K_c (t_{bs} - t_a) \sqrt{V} + \frac{1}{t}(W_o - W_i)\,L.H.E. + A_r K_r (t_{b_s} - mrt) + \frac{1}{t}\left(\frac{W_o + W_i}{2}\right) 0.83 (t_{bi} - t_{bo}) + 1/5\, M_i$$

where:
- $M_r$ = rate of metabolism at rest = BTU/hr
- $M_i$ = rate of increased metabolism over rest state - BTU/hr
- $A_c$ = convection surface for body - $ft^2$
- $K_c$ = convection coefficient = 0.43 BTU/$ft^2$/hr/°F
- $t_{b_s}$ = temperature of body surface - °F
- $t_a$ = temperature of ambient air - °F
- V = velocity of air over convecting surface - ft/min
- t = time, hrs
- $W_o$ & $W_i$ = initial and final body weights - lbs
- L.H.E. = latent heat of evaporation (1035 BTU/lb $H_2O$)
- $A_r$ = radiating surface of body - $ft^2$
- $K_r$ = radiation coefficient - (1 BTU/$ft^2$/hr/°F)
- mrt = mean radiant temperature - °F
- $t_{bo}$ & $t_{bi}$ = initial and final temperature of body - °F

(at $t_a$ = 72 °F, mean temperature of body and clothing surfaces about 83 °F)

The equation neglects heat loss by conduction, and assumes 100% efficiency for sweating and 20% efficiency for mechanical work. Since the equation is not exact because of the difficulty of determining the various areas and body temperatures involved, these are minor criticisms. The value of the equation is in showing relative magnitudes of the various mechanisms of heat loss under known circumstances of activity and environment. It also helps to show limiting conditions of tolerance in some special cases.

Adaptive Mechanisms

Normally the first adaptive reaction to a change in environmental conditions is an attempt by the body to regulate heat loss by a change in body surface temperature is about 95 °F, although that of the hands and feet may be

lower. Skin temperature may get down to about 75 °F without noticeable effect. When the skin temperature drops below about 75 °F, however, shivering occurs. There can be no heat equilibrium in the resting state when the temperature of the body's surroundings is 50 °F or lower; under these conditions skin temperature drops sufficiently that shivering occurs and metabolism (heat production) increases.

The body's most powerful mechanism for heat loss under warm conditions is sweating. Sweating is controlled by the temperature of the blood supplied to the brain. A change of as little as 0.04 °F will cause a change in sweating. Evaporative cooling by sweating can occur even under conditions of 100% relative humidity if the temperature of the air is below body surface temperature.

Acute illnesses from heat exposure are heat cramps, heat exhaustion and heat stroke. Heat cramps are painful abdominal and muscular spasms which result from salt deficiency. The best means to avoid this difficulty when exposed to heat is to increase salt intake with food. Heat exhaustion is a circulation failure which results in weakness, dizziness, and fainting. It occurs most frequently with younger persons. It results apparently from the person having insufficient blood volume to supply both skin for cooling and muscles for activity. A victim needs only to rest for about 30 minutes in a cool place for recovery. Heat stroke is by far the most severe of the heat illnesses. When it occurs, the body temperature goes very high and the victim goes into a coma. Permanent damage can result if certain body proteins coagulate. Body temperature of a person suffering heat stroke should be reduced as rapidly as possible; water sprays or other drastic means being used. Elderly persons are most susceptible, and about 40% of all attacks are fatal.

Other Regulatory Factors

Evidences of excessive heat exposure are increased pulse rate and high body temperature. Persons can become acclimatized so that their tolerance to heat exposure is increased. Acclimatization involves the following items:

1. increased blood volume (10-20%)
2. more efficient sweating and greater overall amount of sweat
3. lower salt concentration in sweat
4. change of habits involving choice of clothing, food, activities, etc.

A normal, healthy individual can become quite well acclimatized to moderate heat exposure in a period of 4 or 5 days.

The mechanisms of acclimatization to cold are less definite. The metabolic rate increases and it appears that the vasomotor system becomes less responsive to sudden cold stimulus. Clothing does not do very well in keeping a person warm in very cold environments when he is working. With the best clothing a person can tolerate a temperature of 10 °F for about 6 hours; -10° for about 4 hours; -40° for about 1-1/2 hours; and -75° for about 30 minutes. These times can be extended by exercise. The limiting factor for survival under very cold conditions is fatigue; when a person rests, his rate of heat production decreases and he freezes. The fingers and toes will usually be the coldest parts of the body. They have the greatest surface area to mass ratio and the lowest blood supply.

When the skin temperature of these extremities reaches 60 °F, manual dexterity will be affected; when it reaches 50°, pain will begin; at 40°, pain disappears and numbness starts; and at 35°, tissue damage may occur.

Effective Temperature

Effective temperature is a sensory index of warmth combining dry bulb temperature, humidity and air motion. Its major value is in its use as an index under conditions of high temperature and humidity when persons are sweating. Some difficulty is experienced in determining effective temperature scales and indices for low temperatures.

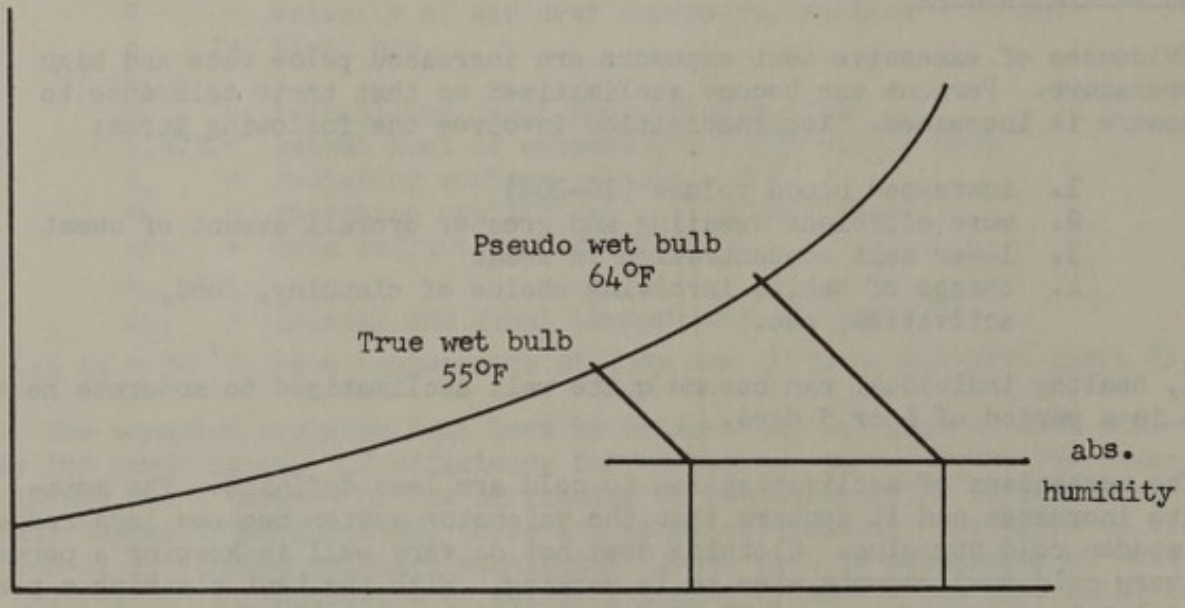
Effective temperature for an environment involving radiant heat load may be determined by use of dry bulb, wet bulb, and globe thermometer readings in the following manner:

1. Determine the absolute humidity of the air by the use of wet bulb and dry bulb temperatures and psychrometric chart.

2. Determine a pseudo wet bulb temperature represented by the absolute humidity and a dry bulb temperature the same as the globe thermometer reading.

3. Determine the effective temperature represented by the pseudo wet bulb reading and the globe thermometer reading by use of the effective temperature chart.

An example of these manipulations on a psychrometric chart is shown below:




| | | | | | |
|---|---|---|---|---|---|
| | | | | true dry bulb 70°F | globe temp 95°F |
| Given: | Dry bulb | 70°F | Find: | Abs. humidity | 40 gr/lb |
| | Wet bulb | 55°F | | Pseudo wet bulb | 64°F |
| | Globe temp. | 95°F | | | |

To determine effective temperature, use the value 95°F for dry bulb temperature and 64°F for wet bulb.

A person who is sweating may experience discomfort when going from a hot environment to a cooler environment when the temperature difference is more than 10 or 12 °F even though the cooler place has an effective temperature well within the normal comfort zone. Sudden temperature changes do not cause much discomfort if sweating is not involved.

Drafts can cause discomfort even though physical parameters indicate comfort conditions. A draft is defined as a condition of air motion which causes excessive heat loss from a single part of the body. (Shoulders, feet, back of the neck, and lower back are very sensitive to drafts.) Air motion greater than 40-50 feet per minute when the air temperature is 70 °F or less will be objectionable to more than 10% of subjects. With air temperature greater than 80 °F, however, air velocities as great as 200 feet per minute may not be objectionable.

No single thermal index, such as effective temperature, will give the best indication of the effects of all possible thermal environments upon the heat balance of the human body. The following tabulation lists environmental conditions and the indices which best describe their characteristics with respect to this heat balance.

| Environmental conditions | Index |
|---|---|
| Sub-freezing temperature | Heated globe thermometer |
| Air temperature 32-80°F, low air velocity, mrt approximately = to air temp | Dry bulb thermometer. |
| Air temp 32-80°F, low air velocity, mrt greater than air temp | Globe thermometer |
| Air temp 32-80 °F, high air velocity, mrt = air temp | Equivalent temperature* |
| High air temp, any mrt, velocity or humidity | Effective temperature or "heat stress index" |

* Equiv. temp = $0.48\ mrt + 0.52\ ta - 0.015\sqrt{v}(100 - ta)$

ta = dry bulb temp °F
v = air velocity, feet per minute

Tolerance to Hot Atmospheres

Tolerance to hot atmospheres depends upon the heat balance between the human body and the environment and the temperature to which the human body can rise without damage. If thermal stress is not so acute as to cause tissue damage, it appears that a person can tolerate heat accumulation sufficient to raise the rectal temperature up to 101 °F and the pulse rate from 70-80 up to 125. Effective temperature is a good measure of the environmental stress. The effective temperature which can be tolerated decreases with increasing work rate by the subject. A healthy, young, acclimatizdd person at rest can tolerate an effective temperature of 95 °F for 2-1/2 or 3 hours, while the same

person could tolerate this stress for only about 1/2 hour if he was working at such a rate as to require a metabolism rate of 88 BTU/ft$^2$/hr. This ratio does not remain constant but decreases with increasing effective temperature.

A new concept in determining the degree of thermal stress is the "heat stress index" proposed by Belding and Hatch.(1) Ventilation and thermal stress is discussed in detail in chapter 3 of the Manual of Naval Preventive Medicine.(2)

## Control Methods

### General

Control of industrial heat exposures may be exercised through the following engineering methods:

1. Mechanization of hot jobs
2. Control of environment surrounding the heat producing process, e.g., radiant screening, exhaust ventilation, etc.
3. Evaporative cooling in hot, dry climates when the wet bulb temperature is less than 70 °F.
4. Spot cooling in which a stream of cool air is directed on the individuals exposed to heat. (Avoid sharp temperature changes or too low temperature of the airstream).
5. Fan; air motion only. This is not very effective, and will even be deleterious if the effective temperature is higher than 97 °F.

Some control of the hazards of heat exposure can also be accomplished by a medical program. The following are some of the factors to be considered:

1. Selection, adaptation, and care of workers
2. Control of fatigue, nutrition, and alcoholic habits; maintenance of proper salt balance, provision of salt tablets.
3. Provision of suitable working clothes, showers, and dressing rooms. (Dressing rooms should serve as adjustment rooms in cold weather to decrease the magnitude of sudden changes in temperature between work area and outdoors.)
4. Periodic physical examinations.

### Ventilation

The equation for sensible heat control through ventilation is a simple dilution ventilation equation with a k factor of 1:

$$\text{cfm} = \frac{\text{Total BTU/hr sensible heat}}{1.08 \times \text{temp rise } ^\circ\text{F}}$$

It presumes uniform dissipation of the total sensible heat load to all of the ventilation air.

The method of air supply differs from that usually used in heating or air conditioning. A principal concern in the air-conditioning or heating field is the prevention of drafts. Consequently, diffusers are used, placed as high as possible to give air velocities less than 40 fpm at working level. For outside air introduced for summer cooling these low velocities are not desirable. The air should be introduced about 10 feet above the floor, and simple registers rather than diffusers should be used. With a 30 foot ceiling height, this will cause the working level to obtain the equivalent of three times the amount of ventilation for the entire building. The temperature rise at the working level may thus be less than that predicted on the basis of total volume. Where ceiling outlets must be used, diffusers should be removed and the air directed downward at high velocity.

The following equation can be used to determine the ventilation required for the latent heat load.

$$\text{cfm} = \frac{\text{BTU/hr latent heat}}{0.67 \times \text{grains/lb difference}}$$

It appears to be a simple dilution ventilation equation for moisture content of air with a k factor of 1.

The equation may be rewritten:

$$\text{cfm} = \frac{\text{pounds/hr water released as vapor}}{60 \times \text{lbs } H_2O\text{/lb dry air difference} \times \rho \text{ air}}$$

where $\rho$ air is the density of air in pounds per cubic foot and pounds $H_2O$ per pound of dry air difference means the allowable increase in absolute humidity of the ventilation air in pounds per pound of dry air.

The latent heat load is not a factor unless condensation occurs.

Insulation

Following is the basic equation for heat transfer by conduction:

$$H = \frac{\triangle T}{R}$$

where: $H$ = quantity of heat transferred

$\triangle T$ = temperature differential

$R$ = total thermal resistance

Examination of this equation should indicate whether insulation will be of value in any situation in which its use is possible. For example, if all the heat produced by a process is released to a room, and the heat production is constant, no amount of insulation around the process will be of value. If,however, heat from a process has several avenues of escape, release to the room being only one of these, insulation of the process from the room will be of value in reducing heat exposure in the room. A combination of insulation and exhaust or gravity ventilation will often be quite effective.

Detailed equations for heat transfer by conduction are:

$$H = \frac{\triangle T}{R_1 + R_2 + \cdots R_n}$$

$$H = \frac{\triangle T}{\frac{1}{f_i} + \frac{t_1}{k_1} + \frac{t_2}{k_2} + \cdots \frac{t_n}{k_n} + \frac{1}{f_o}}$$

where: $H$ = rate of heat transfer (BTU/hr)

$\triangle T$ = temperature differential, inside to outside, $^{o}F$

$R$ = Thermal resistance of any wall element BTU/hr $^{o}F$

$f_i$ = conductance of inside gas film

$f_o$ = conductance of outside gas film

$t$ = thickness of wall element, ft

$k$ = conductivity of wall element, BTU/$^{o}F$ hr $ft^2/ft$

(conductances or conductivities are not additive)

The values of k for various materials may be found in references such as the Chemical Engineers Handbook. The values of $f_i$ and $f_o$ depend upon the gas velocity over the surface. With normal room air currents, the film conductance will be about 1.6; with a velocity of 1300 feet per minute, the film conductance will be about 6.

Radiant Heat

Radiant heat is electromagnetic energy similar to visible light, except that it is of a longer wavelength. It does not heat the air appreciably as it passes through. It does little heating until it falls on some object - a man, a wall, a machine, or something else that will absorb the energy. Radiant heat cannot be blown away. Therefore the only protection is shielding of the man from the source. This can be done by a reflective screen, reflective clothing, and by enclosing the heat source with material that minimizes emission of the radiant heat.

A "black body" absorbs all of the radiant heat incident upon its surface and reflects none. Most materials reflect a fraction (reflectivity) and absorb the remaining fraction of the incident energy (absorbtivity). The radiating power of a black body is proportional to the fourth power of its absolute temperature. Common materials radiate less energy than does a black body at the same temperature. The ratio of the radiating power of the material to that of a black body is its emissivity. The emissivity of a surface is equal exactly to its absorptivity. In other words, a good reflector (or a poor absorber) has a low radiating power. The following table shows approximate emissivities for some common materials, as well as their corresponding values of reflectivity.

| Material | Emissivity | Reflectivity (Percent) |
|---|---|---|
| Lacquer, flat black | .97 | 3 |
| Lacquer, black | .90 | 10 |
| Lacquer, white | .90 | 10 |
| Asbestos board | .94 | 6 |
| Iron, sheet, smooth | .55 | 45 |
| Iron, sheet, oxidized | .65 | 35 |
| Brick | .80 | 20 |
| Zinc, bright | .10 | 90 |
| Zinc, oxidized | .27 | 73 |
| Aluminum, bright | .05 | 95 |
| Aluminum, oxidized | .16 | 84 |
| Aluminum paint, new, clean | .35 | 65 |
| Aluminum, dull, dirty paint | .60 | 40 |

A conventional globe thermometer consists of a 6-inch hollow, copper sphere painted a matte black. However, for ordinary plant measurements, a common copper toilet float painted flat black gives a sufficiently accurate reading, and is considerably cheaper and easier to obtain. The bulb of a sensitive glass thermometer is fixed at the center of the sphere with the stem protruding to the outside through a sealed opening. The instrument is suspended at the desired location with fine piano wire or string, and 1/2 hour is allowed to approach a steady state before making observations.

For satisfactory readings with a globe thermometer, the net radiation effect on the globe should be positive (or the unit heated) so that at equilibrium the temperature reading of the globe thermometer is at least 10 °F greater than the temperature of the air. Heat must be lost by convection. The equilibrium heat balance for the globe thermometer is:

$(mrt - t_g) = C\sqrt{V}(t_g - t_a)$

mrt = mean radiant temperature, °F

$t_g$ = globe temperature, °F

C = convection coefficient, (determined by calibration, about 0.17 for standard 6" black globe)

V = air motion, ft/min

$t_a$ = air temperature, °F

Mean radiant temperature (mrt) represents the temperature of a uniform black enclosure in which a solid body would exchange the same amount of radiant heat as in the given environment. A basic equation for radiant heat exchange is:

$$\frac{qr}{A_1} = F_A F_E \sigma (T_1^4 - T_2^4)$$

where: $\frac{qr}{A_1}$ = rate of exchange, BTU/hr/ft$^2$

$F_A$ = geometry factor (_ 1) which depends on shape and orientation of surfaces ($F_A$ = 1 for infinite parallel planes, long concentric cylinders, small bodies in large enclosures, inc.).

$F_E$ = emissivity factor (_ 1). ($F = \frac{1}{\frac{1}{\epsilon_1} + \frac{1}{\epsilon_2} - 1}$

for large parallel planes, long concentric cylinders, small bodies in large enclosures, etc.)

$\sigma$ = Stefan-Broltzman radiation constant $1730 \times 10^{-12}$ BTU/hr/ft$^2$ ($^oR$)$^4$

T = $^oR$

Solved equations for radiant heat interchange for numerous special cases are listed in the Heating, Ventilating, and Air Conditioning Guide, ASHVE, 32 edition, 1954, pages 92-99.

Definitions

Dew point: the temperature to which air must be cooled, at constant pressure, in order to become saturated.

Dry bulb temperature: the temperature of a gas or mixture of gases indicated by an accurate thermometer after correction for radiation.

Wet bulb temperature: the temperature at which water, by evaporating into air, can bring the air to saturation adiabatically at the same temperature. It is the temperature registered by a standard wet bulb thermometer.

Equivalent effective temperature: an arbitrary index which combines into a single value the effect of temperature, humidity, radiant heat and air movement on the sensation of warmth or cold felt by the human body. The numerical value is that of the temperature of still, saturated air which would induce an identical sensation.

## Summary

Comfort ventilation is normally required for odor control and temperature regulation; oxygen supply and carbon dioxide removal requirements are normally much smaller. The body must have available to it some avenue of heat loss. Heat production is continuous and thermal equilibrium must be maintained in a narrow temperature range if a person is to suffer no ill effects or even survive. Sweating is the body's most powerful mechanism for heat loss when temperatures are high.

Effective temperature is a sensory index of the thermal effect of an environment on a human. It is the best index of thermal stress when the temperature and humidity are high. Sudden temperature changes and drafts can cause discomfort even though comfort conditions are otherwise indicated.

Mechanical controls and medical considerations can be used to decrease difficulties involved in the exposure of workers to excessive heat. There is no single solution which can apply to every case of heat exposure. Each situation must be analyzed to determine the means which can be used to decrease the hazards or improve the conditions of comfort.

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# Control Methods

# ENGINEERING CONTROL OF THE OCCUPATIONAL ENVIRONMENT

## Introduction

The control of environmental occupational health hazards is generally a responsibility of the industrial hygiene engineer. Control of these hazards may usually be accomplished by one or more of the following methods:

1. Control at point of generation or dissemination
2. Dilution with uncontaminated air
3. Isolation or enclosure of processes or operations
4. Substitution of less toxic materials or change of process
5. Personal protective devices, and
6. Proper maintenance, housekeeping and education.

These general control principles apply not only to toxic materials such as chemicals and dusts, but also to many physical agents which are encountered in industry.

## General Methods of Control

### Control at Source

The approaches commonly employed to control atmospheric contaminants at their source are: (1) local exhaust ventilation, (2) use of wet methods, and (3) good housekeeping.

Local exhaust ventilation is perhaps the most important method for controlling atmospheric contaminants at their source. The engineer plays important roles both in designing the exhaust systems and also checking their efficiency. Detailed discussions on this subject are found in other sections of the manual.

Wet methods are successfully used in certain foundry operations where, for example, a hydroblast is used in place of sandblasting or tumbling operations for cleaning castings. Dust concentrations can also be reduced if the molding sand is kept moist, if the castings are wet down before shakeout and if the floors are wet down intermittently. In the granite industry wet methods have reduced the dust formerly generated by many operations. Wire saws and contour grinding wheels, operated with water and artificial abrasive, have replaced most of the former cutting tools.

Good housekeeping also plays an important role in reducing dust concentrations at the source in foundries as well as many other industrial operations. Removal of dust from floors, machinery and overhead beams prevents its dissemination to other areas of the plant.

### Dilution Ventilation

Sometimes referred to as general ventilation, dilution ventilation entails the use of natural convective currents through open doors or windows, roof ventilators, chimneys or artificial and mechanical ventilation produced

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by fans or blowers. Exhaust fans located in roof ventilators and windows are commonly used for dilution ventilation. It is important to remember that dilution ventilation is practicable only if the degree of air contamination is not excessive, and particularly if the contaminant is released at a substantial distance from the worker's breathing zone. Otherwise, contaminated air will not be diluted sufficiently before inhalation. Dilution ventilation is not generally recommended for dusts or fumes. Formulae are available in standard texts to determine the amount of ventilation necessary to dilute the contaminated air to a safe level. One simple formula is:

$$\text{cu. ft. vapor/hr.} = 416 \times \text{pints used} \times \frac{\text{sp. gr.}}{\text{mol. wt.}}$$

$$\text{cu. ft. air required per hr.} = \frac{\text{cu. ft. vapor/hr.}}{\text{threshold limit value}}$$

Effective temperatures can, in many cases, be reduced by means of dilution or general ventilation.

Sufficient make-up air must be provided for local exhaust or general ventilation because an exhaust system will not operate at its rated capacity unless there is adequate air to replace that which is exhausted. Most of the newer plants are designed to provide not only make-up air, but also to filter and temper it as well. Recirculation of air is permissible in certain operations but is usually prohibited from dust collectors.

Isolation or Enclosure

Isolation or enclosure of certain hazardous operations is another method of control. This reduces the potential exposure to relatively few persons rather than an entire area of the plant. Locating solvent degreasers in one corner of a plant, complete enclosure of sandblast operations, isolation and enclosure of radium dial painting areas are only a few of many examples. Isolation is also an effective method of noise control for certain types of operations.

Substitution

A very important and useful control method, especially when dealing with solvents, is substitution of less toxic or non-toxic materials. Naphtha, or a number of other solvents less toxic than carbon tetrachloride, can be used in place of carbon tetrachloride in many cases; however, if naphtha presents a fire hazard, methyl chloroform may be used. Toluene or xylene can be used instead of benzene.

Steel shot, ground walnut hulls, carborundum, alundum and other non-toxic materials are employed as substitutes for sand in many abrasive cleaning operations. Titanium and zinc compounds have been used successfully to replace lead pigments, resulting in the elimination of the lead hazard in many painting operations. A classic example of substitution was the use of non-toxic carroting materials for mercury compounds in the hatters fur industry.

Changing the process should also be mentioned with substitution because, in many instances, it is possible to reduce or eliminate hazards by this method. Replacement of hand methods for applying lead oxide to lead grids reduced the lead exposure in the storage battery industry. On the other hand, mechanization may aggravate rather than decrease hazardous conditions in some instances.

Personal Protective Devices

These devices may be divided into three groups: (1) goggles and face shields; (2) protective clothing; and (3) respirators. The safety department, especially in larger plants, is generally responsible for the issuance, maintenance, and educational program of these devices. In many instances, however, the industrial hygienist is asked to advise and make recommendations concerning the choice and use of the protective equipment. For this reason he must be familiar with, and know the limitations of, these items.

Goggles and face shields. These are used to protect the eyes and face from corrosive solids, liquids, and foreign bodies. Goggles are also used for protection against conjunctivitis or other eye injury resulting from welding or flame cutting operations and ultraviolet and infrared from other sources. Many types are available; the important point is to choose the correct type for the specific job and insist that the workmen wear the goggle or face shield on the job. Many industrial companies now have excellent eye protection programs. One reason for their success is a strict rule requiring that eye protection devices be worn on specific jobs.

Protective clothing. Gloves, aprons, footwear, impervious coveralls and other items of protective clothing are used to prevent contact with the body of a host of corrosive liquids, solvents and many other materials. The value of protective clothing in the prevention of dermatitis has been recognized for many years. One of the chief problems is in choosing the correct article for a specific application. For example, one type of rubber glove will afford ample protection against carbon tetrachloride and trichloroethylene but becomes spongy and will eventually disintegrate in contact with lacquer solvents. Similar problems are encountered with rubber boots used around aircraft cleaning areas where a variety of paint strippers and cleaning agents are used.

Respirators. The use of respiratory protection devices should normally be restricted to intermittent exposures or those which are impractical to control by other methods. They should not be substituted for good engineering control measures. Exceptions are the use of supplied air devices for protection in sandblasting or for operations in confined spaces where an oxygen deficiency may exist.

Respirators can be divided into three general types: (1) for protection against pneumoconiosis - producing and nuisance or toxic dusts; metal fume and acid or alkali mists (chromic acid, sodium hydroxide); (2) for protection against gases and organic vapors (chlorine and carbon tetrachloride); and (3) air-line or supplied air respirators. The U. S. Bureau of Mines periodically publishes lists of various types of respirators which have met their specifications.

In general, each type of respirator is designed for protection against specific toxic materials; choice of the proper respirator for the specific contaminant is therefore a most important consideration. Dust respirators, for example, should not be used for protection against lead fume - it has been done! Check to determine if the proper respirator is being worn and that it has been approved by the Bureau of Mines for that purpose.

A successful respirator program entails not only the selection of the proper device but also a program whereby the workers are furnished clean, sterilized respirators at frequent intervals. A cleaning and maintenance program also applies to goggles, face shields, ear defenders and articles of protective clothing.

Education of the worker is a must for a successful personal protective program. They must be given reasons for wearing these devices and instructions on their proper use.

Maintenance, Housekeeping and Education

These three items are interdependent. It is impossible to have satisfactory maintenance unless the housekeeping is good and the worker has been educated regarding the need and methods of achieving these principles. Maintenance and housekeeping apply not only to operation of control equipment, such as exhaust systems, but also to production facilities in the plant. For example, if the thermostat on a degreaser fails or is accidentally broken, excessive concentrations of trichloroethylene might quickly build up in the work area. Lead dust or mercury, if permitted to accumulate on the floor, may create hazardous exposures to these toxic substances. These are but a few of the many examples which show the results of poor maintenance, housekeeping and education.

## Special Control Methods

Many of the general methods cited above, or a combination of them, can be used for the control of most occupational health hazards. There are, however, a few additional situations which deserve special mention.

Ionizing Radiation

The entire field of ionizing radiation is a specialized one and cannot be adequately covered in a short course such as this. However, the same basic principles described in this lecture are involved in the control of ionizing radiation problems. The Atomic Energy Commission and the National Bureau of Standards have published much useful information in this field. If these guides are followed carefully, there should be no difficulty in controlling exposures. Many occupational radiation exposures occur as a result of carelessness and accidents; thus constant vigilance must be maintained.

Excessive Heat and Humidity

Control of these environmental factors may be a problem particularly in operations such as steel manufacturing and processing, heat treating, drop forging and manufacturing glass.

Protection of the worker from excessive radiant heat can usually be accomplished by shielding the source. The glass industry has spent large sums of money shielding the furnaces with aluminum panels, which incidentally reflect about 95 percent of the radiant heat. In the steel industry shielding certain operations such as soaking pits and rolling mills, and air conditioning crane cabs, resulted in reduced radiant heat exposures. Heat treating furnaces can be shielded by means of aluminum or in certain instances by fire chains. The use of steel or sheet metal for shielding is not very effective because a black body absorbs radiant heat and after a short time it will act as a secondary radiator.

While shielding will normally result in lowered effective temperatures since the radiant heat is reduced, other measures may be needed to further reduce the effective temperature to comfortable levels. These include the use of general ventilation, local exhaust or floor fans. A combination of two or more of these measures may be necessary for certain operations. Air conditioning is a most effective method of reducing temperature and humidity but it is usually too costly for installation in large plants.

Humidity inside a plant can be reduced by installing local exhaust ventilation on all operations generating steam or water vapor. This method is effective in paper manufacture, dye plants, laundries, electroplating and pickling tanks, and similar industrial operations. The application of insulation around steam and hot water pipes in these plants will effectively reduce temperatures.

Many hot operations cannot be controlled because of the cost involved. In these situations the only solution is reducing the duration of the work day or in effect, the exposure. This method was successfully employed several years ago at a zinc smelter and also in a large drop forge plant.

## Summary

General engineering control methods for the occupational environment have been discussed and examples of each have been cited. One or a combination of these methods may be necessary to effectively control potentially hazardous exposures. In addition, measures were enumerated for control of radiation, and effective temperature. Each particular situation will require the application of knowledge gained through education and experience.

# CONTROL OF NOISE EXPOSURE

## Introduction

Industrial noise control is a complex problem and methods of reduction in a particular case depend on many factors. It must be kept in mind that large reductions in energy are needed; for example, cutting the energy in half only reduces the sound pressure level by 3 db. Since noise control is usually expensive, it is important to study the problem thoroughly before an attempt is made to reduce the noise.

## Plant Design

Since industrial noise is becoming more important, it is essential that planning be done to avoid or minimize noise in a new plant or modernizing an existing plant.

### Selection of Building Site

There are several factors which should be taken into consideration in the selection of a plant site. One of the most important of these is the existing noise level of the community. It is much better to locate a noisy industrial plant in an area that has a high background noise level than in a quiet area. Conversely, a church, school, or hospital should be located in an area with a low background noise level.

Ofttimes, advantage can be taken of the topography and prevailing winds in noise control. There is a tendency for sound waves to be deflected upward so it is better to locate a plant on top of a hill rather than in a valley if there is a possibility of a neighborhood noise problem. Sound waves traveling into the wind tend to be deflected upward while those traveling with the wind tend to be deflected downward. In some situations it may be possible to take advantage of this by considering the prevailing winds.

It is also possible to reduce noise by proper landscaping; using embankments, terraces or shrubbery as sound barriers. These can be used in isolating a building from existing noise or prevent noise from a building creating a neighborhood problem.

### Building Layout

Noisy operations should be isolated from the rest of the operations. When possible it is better to locate offices, conference rooms, libraries and other areas where quiet is desired, away from the street.

### Selection of Work Process

Sometimes the work process can be changed with an appreciable reduction in noise level. Several possibilities are as follows: (a) compression

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riveting instead of pneumatic riveting; (b) welding instead of riveting; (c) belt drive in place of gears; (d) hot instead of cold working of metals; (e) grinding instead of chipping; (f) pressing instead of rolling or forging; (g) avoidance of dropping hard materials during handling; and (h) selection of less noisy conveyors or machines.

## Reduction at Source

Prevention of noise at its source is the best method of noise reduction wherever practicable. There is much less surface area to be treated. This also gives protection to the operator. In many cases it is more economical to eliminate or reduce the noise at its source than to try to deal with it after it has been released into the room. Methods of reducing unnecessary noise are discussed below.

### Lost Motion and Rattle

Noises of this nature are usually attributed to poor maintenance. Replacement of worn parts, securing, stiffening or damping loose panels will eliminate a large amount of this noise.

### Friction

Noise is produced by friction forces generated by grinding and cutting operations, or used in portions of machines such as, clutches and brakes. Some of this noise can be eliminated by proper lubrication of moving parts and use of cutting oils or solutions. Keeping cutting tools sharp and properly shaped will help also. Substitution of materials in brakes or clutches may give a reduction in noise.

### Impacts

Impact noise is generated when materials are forged, hammered, sheared, tumbled or peened. Noise from such sources is generally difficult to control. One approach is to spread the impact out over a longer time interval. This can be done in some cases by the use of stepped dies. These produce a number of small impacts rather than one large impact. It may be possible to change a punching operation into a shearing operation. Clamping or placing weights on the piece being hammered or peened will reduce materially the noise generated from such operations. Tumbling barrels can be coated with a mastic material or suitable innerliners can be used to reduce noise levels.

### Fluctuations in Gas Flow

Where there is a fluctuation in gas flow noise may be generated. This may be in an internal combustion engine, high speed fan, compressed air or steam jets, or air driven tools. Examples of some of these with possible methods of noise control are: (a) internal combustion engine by proper intake and exhaust mufflers; (b) high speed fan through larger fans operating at slower speeds or redesign of fan blades; (c) compressed air tools with mufflers; (d) compressed air jets with mufflers or redesign of jet nozzle; and (e) those tools which produce a siren type of noise by redesign of openings or possibly slower speed of operation.

Unbalanced Forces

Reciprocating motion or unbalanced rotation can cause noise or it may excite other structural members and cause them to radiate noise. High speed rotating parts should be balanced properly. Very little can be done about reciprocating parts except proper maintenance and mountings.

Reduction of the Flow of Noise Energy

This is a very fruitful field for noise control because many sources of noise energy are small and inefficient sound wave generators. If this flow of energy can be interrupted and prevented from reaching structural members and panels, which may be more efficient sound generators, much of the noise can be controlled.

Solid-borne transmission. Vibrations can be transmitted considerable distances through solid materials with very little attenuation. If some break can be made in this path, the flow of energy will be reduced. This can be done by using a soft, flexible material such as, vibration mounts, flexible couplings on shafts or pipes, flexible connectors on ventilation ducts, belt drives, and mastic materials at joints when fastening materials together.

Energy may be transmitted to structural members or panels which are set into vibration and radiate the energy. Structural members and panels can be stiffened to reduce vibration.

Vibration-damping materials resist motion in any direction and during every cycle of vibration. Thus, they provide definite energy losses during each cycle of vibration. Materials such as, mastic, and vegetable and mineral fibers impregnated with mastic have been used successfully to reduce vibration and in turn reduce noise levels.

Air-borne transmission. When it is not possible to control the noise energy within the machine it is sometimes possible to control it economically by using a partial enclosure or total enclosure rather than treating the entire work area involved.

Partial enclosures. Partial enclosures can be of a number of shapes or designs. They may be just single panels beside the source, panels on both sides of the source or tunnels with work flowing through them. Sound absorbing linings must be used inside these enclosures. The barrier material can be plywood, wallboard, sheet metal or almost any material with the requisite mass and strength.

This type of enclosure usually does not protect the operator but does produce a shadow effect to give protection outside of the enclosure of up to about 15 db.

Total enclosures. These enclose the source completely except for small access openings, which are treated by the use of short acoustical ducts. These should be made of an impervious material to stop the flow of energy and an acoustical lining to absorb the energy within the enclosure.

Properly designed enclosures will give a 20 to 30 db noise reduction. They do have the disadvantages of interfering with ventilation, handling of materials and maintenance.

Absorption of Noise

The most common use of sound absorbing material is in general or overall noise control in enclosed spaces. Sound absorbing material has very little effect on the noise level close to a source but will give a considerable reduction some distance from the source in the reverberant field. The limit for most practical installations is a reduction of 10 db.

Sound absorbing materials are usually porous and may be made of vegetable or asbestos fibers, glass or mineral wool. They are available in a number of forms including blocks, panels or blankets which can be fastened to the walls or ceiling, and space absorbers which can be suspended from the ceiling.

## Personnel Protection

In some cases it will not be possible to control the noise because of economic limitations or lack of engineering knowledge. If there is danger of hearing loss, personnel protective equipment should be worn.

Types of Ear Protectors

Ear protectors are usually classified according to the manner in which they are to be worn, and the three usual types are: ear plugs, ear muffs, and helmets.

Ear plugs. These are inserted into the ear canal and are intended to reduce the air-borne sound just before it impinges on the ear drum. They may be made of rubber, plastic, neoprene, or cotton impregnated with wax. Material and shape have very little to do with the effectiveness of commercially available plugs. Contrary to popular opinion, dry cotton gives no protection.

Ear muffs. The muff-type protector consists of a set of cups designed to cover both ears and to be held snugly to the head by a headband. The cups may be made of plastic or rubber and are designed to encompass the ear without compressing it and yet at the same time to fit so closely to the head that very little sound leaks through.

Helmets. These are designed to cover most of the bony portion of the head to prevent bone conduction, but this has not proved to be completely satisfactory. Actually the whole head would have to be encased to avoid bone conduction effectively.

The amount of protection that can be expected from plugs or muffs is shown in table 1.

Table 1. - Average attenuation of ear protectors
(in decibels of threshold shift)
measured in the field and in the laboratory

| Ear Protector Tested | Frequency in Cycles Per Second | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 250 | 500 | 750 | 1000 | 1500 | 2000 | 3000 | 4000 | 6000 |
| Seven types of ear plug-lab. n = 8 | 12.6 | 13.0 | | 19.0 | | 25.7 | 29.3 | 27.2 | 29.7 |
| Two types of ear plug-field* n = 25 | | 10 | 10 | | 16 | | 24 | | 23 |
| One type of ear plug-lab. n = 3 | 28 | 29 | | 26 | | 34 | 45 | 45 | 45 |
| One type of ear muff-lab. n = 3 | 20 | 32 | | 43 | | 44 | 42 | 38 | 34 |
| Combination plug and muff-lab. n = 3 | 38 | 45 | | 41 | | 45 | 49 | 47 | 34 |

*The low values of attenuation found in the field can be explained in part by the fact that several of the subjects wore plugs that were too small to provide a good seal.

## Selecting and Using Protectors

The ear protection program should be under the supervision of the medical department. An employee's ears should be examined and his hearing tested at the time he is fitted with ear protectors. A number of types and sizes of plugs should be available and plugs should be fitted individually for each ear. Most of the available ear protectors, when properly fitted, provide about the same amount of protection, therefore the one that fits correctly and is the most comfortable for a particular employee is the best for him. Properly fitted protectors can be worn continuously by most persons and will provide adequate protection against most industrial noises.

As with other types of personnel protection, it may be difficult to convince employees that they should wear ear protectors. An educational program will probably be necessary and employees should be told why it is necessary to wear the plugs or muffs. They should be instructed how to insert the plugs and how to keep them clean. In many plants it is the responsibility of the safety engineer or industrial hygienist to supervise the use of protectors.

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# INTRODUCTION TO VENTILATION AIRFLOW

## Introduction

This discussion, and others to follow deal with various aspects of ventilation airflow. Most of these sections are correlated with chapters in Industrial Ventilation - A Manual of Recommended Practice,[2] published by the Committee on Industrial Ventilation of the American Conference of Governmental Industrial Hygienists (A.C.G.I.H.), and are intended to provide supplemental material useful to the industrial hygienist. Due to the breadth of the field of ventilation airflow, and to space limitations, the coverage is necessarily far from complete.

Any reference to figure or page numbers in Industrial Ventilation refer to the fourth edition (1956). All references cited in these sections on ventilation are listed in Section C-11.

## Principles of Ventilation Airflow

The driving force for airflow is pressure difference. A gas at a given pressure will always flow toward an area of lower pressure, causing a reduction in the potential energy of the gas, or in other words, a reduction in its capacity to do useful work. The reduction in potential energy results in an increase in its kinetic energy, or energy of motion. A fluid in motion exerts pressure on any object in its path. This pressure is exerted only in the direction of flow; is a function of the kinetic energy of the fluid, and is known as velocity pressure. The non-directional pressure of potential energy is known as static pressure. Conversion of static pressure to velocity pressure is a degradation of energy, analagous to the conversion of mechanical energy to heat. In both cases the energy forms are inter-convertible, but the efficiency of conversion from the lower form to the higher form is always less than 100%. The inefficiency is due to friction, and the more gradual the conversion, the closer 100% is approached.

The pressure exerted on a given surface is known as total pressure, and is the algebraic summation of the velocity pressure on that surface, and the static pressure at that location. Figs. 1, 2, 3, and 4, reprinted from Industrial Ventilation[2], illustrate in detail the relations between static, velocity, and total pressures. In these figures, pressures act on the surface of the leg of a U-tube manometer. Static pressure is directed to the manometer from a hole in the side of a duct, and total pressure from an impact tube inside the duct. Velocity pressure can be obtained mathematically by subtracting static pressure from total pressure algebraically, or mechanically with a Pitot tube by connecting one leg of the manometer to the static pressure opening and the other to the impact tube. If the impact tube faces the direction of flow, the velocity pressure will be positive. If the impact tube faces the opposite direction, or if there is no flow at all, the velocity pressure will be 0 and total pressure will equal the static pressure.

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Fig. 1

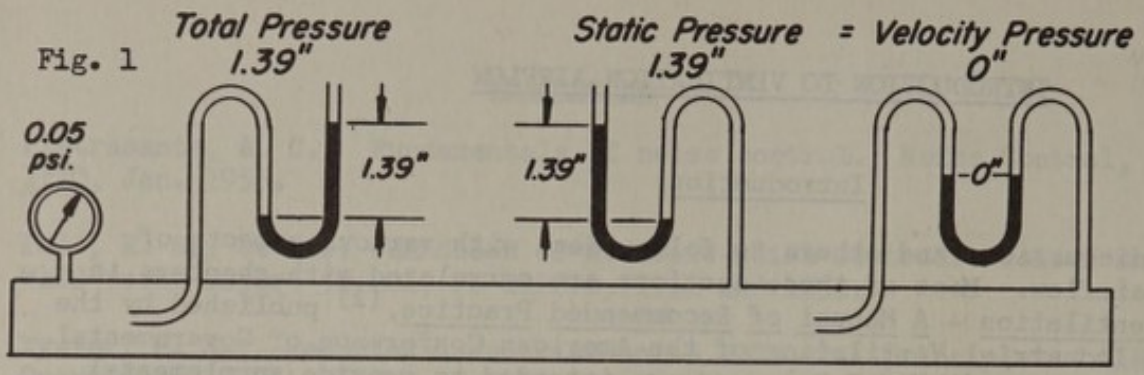
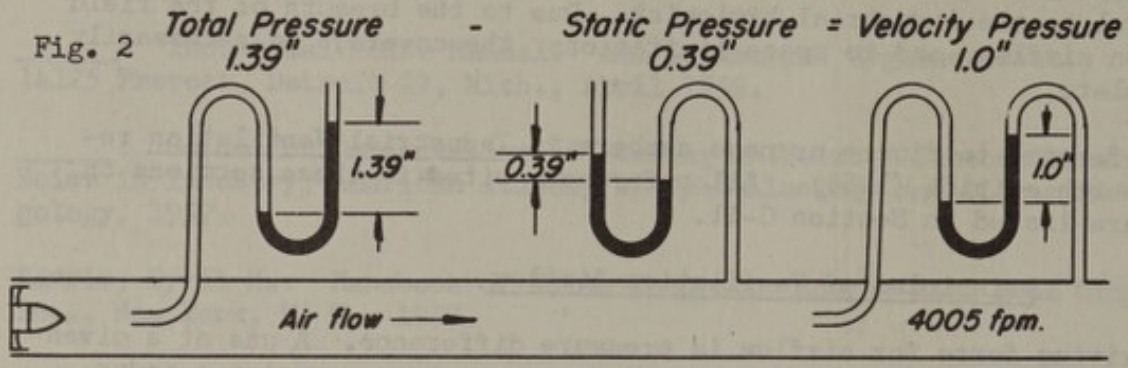
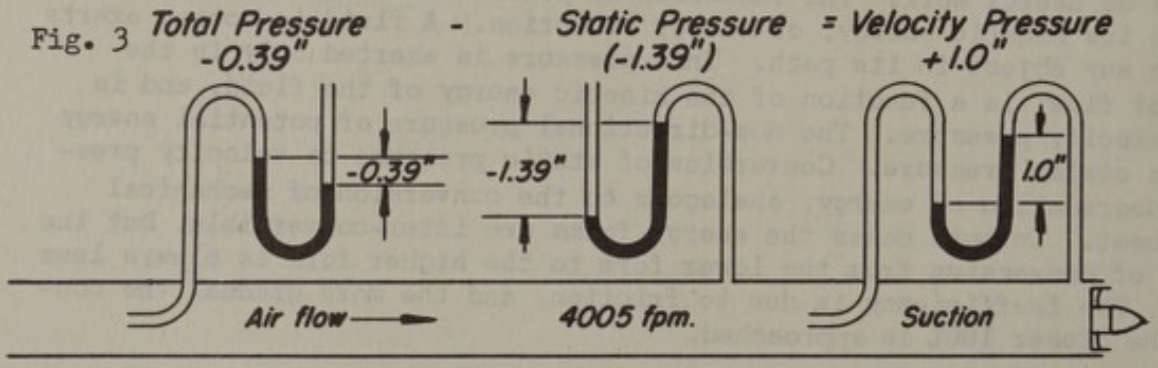



Fig. 2



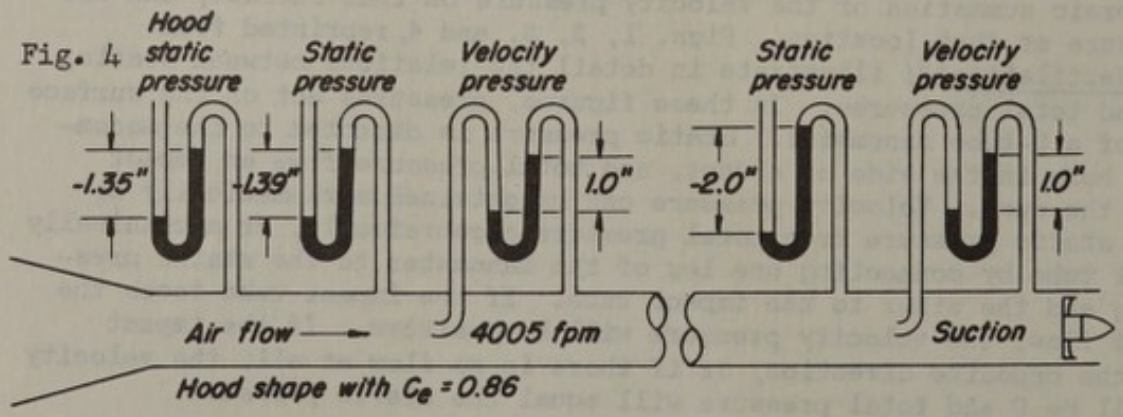

PRESSURE IN THIS PIPE ABOVE ATMOSPHERIC PRESSURE

Fig. 3




PRESSURE IN THIS PIPE BELOW ATMOSPHERIC PRESSURE

Fig. 4




Figures 1, 2, 3 and 4 were reproduced by permission from Industrial Ventilation - A Manual of Recommended Practice, published by the American Conference of Industrial Hygienists' Committee on Industrial Ventilation.

Fig. 4 shows how total, static, and velocity pressures vary as air passes through a hood, duct section, duct enlargement, a fan, and a weather cap. Note that the velocity pressure varies only with the duct size at constant airflow, while the static pressure is affected by pressure losses in the system. The total pressure is, at all times, the algebraic summation of the velocity and static pressures.

## Kinds of Fluid Flow

Fluid flow can assume two different forms. In one, known as streamline or viscous flow, the fluid filaments travel in straight lines, parallel to the direction of flow, and in a duct the velocity of the filaments rises progressively as the center is approached. In the second type of flow, known as turbulent flow, each individual filament travels in a sinusoidal path, developing irregular whirling eddies, which continually dissipate and re-form. The type of fluid flow that will take place under a given set of conditions can be predicted from the Reynolds number, defined as:

$$R_e = \frac{DV\rho}{\mu} \qquad (1)$$

where $R_e$ = Reynolds number

$D$ = pipe diameter (or equivalent diameter if other shapes are used)

$V$ = average linear velocity

$\rho$ = fluid density

$\mu$ = fluid viscosity

For $R_e$ less than 2100 in long, straight pipes, the flow will always be streamline. When $R_e$ is greater than 4,000 the flow will always be turbulent. Between 2100 and 4,000 is a transitional region of instability where no accurate predictions can be made as to the nature of the flow.

Air and gas flow is almost always in the turbulent range. The only case of streamline ventilation airflow that is often encountered is the flow through the filter surfaces of a cloth filter, where the linear velocity is on the order of 1 - 4 lfm.

## Bernoulli's Theorem

Bernoulli's theorem is a statement of the law of conservation of energy for fluid flow. It can be expressed:

$$Z_1 + \frac{V_1^2}{2g} + W_o - \int_{P_1V_1}^{P_2V_2} vdP - F = Z_2 + \frac{V_2^2}{2g} \qquad (2)$$

where: $Z$ = height above any arbitrary horizontal datum plane, ft

$V$ = linear velocity, ft/sec.

$g$ = gravitational acceleration, ft/sec.$^2$

$W_o$ = external work put into system, ft-lb/lb

$P$ = absolute static pressure, p.s.i.

$v$ = specific volume, ft$^3$/lb

$F$ = Friction loss, ft. of fluid

subscripts 1 and 2 refer to initial and terminal conditions.

For isothermal flow of gases in pipes where the absolute pressure ratio $P_1/P_2$ is less than 2, the following formula will be sufficiently precise.

$$Z_1 + \frac{V_1^2}{2g} + P_1 v_{av.} + W_o = Z_2 + \frac{V_2^2}{2g} + P_2 v_{av.} + F \qquad (3)$$

This can be expressed as:

$$(P_1 - P_2)v_{av.} = \frac{V_2^2 - V_1^2}{2g} + F + (Z_2 - Z_1) - W_o \qquad (4)$$

where: $(P_1 - P_2)v_{av.}$ is the static pressure difference in height of a fluid column.

$\frac{V_2^2 - V_1^2}{2g}$ is the velocity pressure difference in height of a fluid column.

$(Z_2 - Z_1)$ is the static pressure difference due to location difference.

$W_o$ would be introduced to a duct system by a fan or other air moving device. In a given duct section it would be 0. $(Z_2 - Z_1)$ in a horizontal duct section would be zero, and in a vertical duct section it would be negligible in most cases.

Therefore, for low pressure airflow in a duct, a decrease in potential energy $(P_1 - P_2)v_{av.}$ results in an increase in kinetic energy $\frac{V_2^2 - V_1^2}{2g}$, plus a frictional loss.

Relation Between Velocity and Velocity Pressure

If the velocity pressure ($P_v$) of a gas is equal to $\left(\frac{V^2}{2g}\right)$ under a given condition, then the velocity at that condition can be expressed as $V = \sqrt{2gP_v}$. Therefore, if the velocity pressure ($P_v$) can be measured, the velocity of the gas ($V$) can be calculated.

The relation $V = \sqrt{2gP_v}$ is more useful when expressed in convenient units. Ventilation air velocities are usually expressed in linear feet per min. (lfm),

and pressures in inches of water gauge. The relation $V = \sqrt{2gP_v}$ becomes:

$$V = 1096\sqrt{\frac{h_v}{\rho}} \qquad (5)$$

where: V = velocity in linear ft. per min. (lfm)

$h_v$ = velocity pressure in inches of water ($"H_2O$)

$\rho$ = density of gas stream in $lb/ft^3$

At standard air conditions of 70° F and 1 atmosphere, the formula becomes:

$$V = 4005\sqrt{h_v} \qquad (6)$$

or: $$Q = 4005A\sqrt{h_v} \qquad (6a)$$

Pressure Drop Due To Friction

Air in motion encounters resistance along the surfaces confining the flow. Some of the energy of the air is given up in overcoming this resistance and manifests itself as heat. The rougher the surface, the higher the flow rate, and the greater the surface to volume ratio, the higher the resistance loss will be. A second kind of frictional resistance encountered in ventilation airflow is air friction, also known as dynamic loss, turbulent loss, and shock loss. When air goes through a change in direction or velocity, the increased turbulence causes an increased resistance to flow. The more abrupt the change, the greater the pressure loss.

Frictional resistance of the first kind is usually called duct resistance, and in a round duct varies directly as the length, inversely as the diameter, and directly as the square of the air velocity. It can be calculated for any fluid flow in circular pipes from the Fanning or Darcy equation:

$$F = 4f\left(\frac{V^2}{2g}\right)\left(\frac{L}{D}\right) \qquad (7)$$

where: F = friction loss in feet of fluid flowing

V = linear velocity in ft/sec.

g = gravitational acceleration in $ft/sec.^2$

L = duct length

D = duct diameter

f = friction factor - dimensionless - (a function of Reynolds number)

For ducts other than circular, the equivalent hydraulic diameter ($D_h$) may be substituted for D. $D_h$ for airflow is defined as:

$$D_h = \frac{4 \times \text{area of cross-section}}{\text{perimeter of cross-section}}$$

The Fanning friction factor, f, varies with Reynolds number. Fig. 5 is a graph of friction factor versus Reynolds number. Some of the ventilation references (1) (5) show similar charts in which the f values are four times as high as those in Fig. 5. The friction formulas to be used with the f values have different constants, so if used consistently, the same result will be calculated in either case.

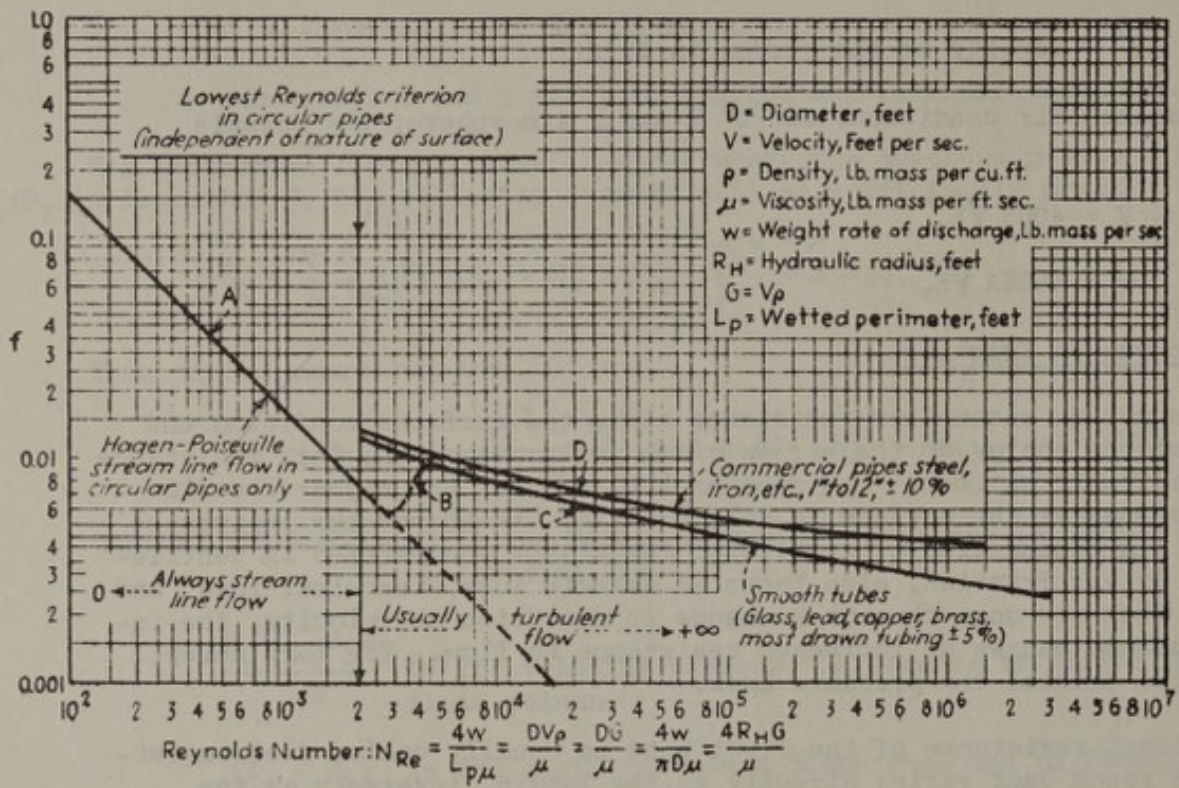



Fig. 5 - Fanning Friction Factors, f, in Equations (7) and (8). (Reprinted by permission from CHEMICAL ENGINEERS' HANDBOOK, edited by J. H. Perry. Third Edition, Copyright, 1950. McGraw-Hill Book Co., Inc.)

If air at standard conditions is the fluid under consideration, the Fanning equation can be reduced to the convenient form:

$$h_f = 0.000298 \frac{f V^2}{D} \qquad (8)$$

where: $h_f$ = friction loss per hundred feet of duct, inches of $H_2O$

$f$ = Fanning friction factor

$V$ = air velocity, lfm

$D$ = duct diameter, inches

Relation (8) has been used to plot friction charts with friction loss per 100 feet, duct diameter, air velocity and air volume as the parameters.

Fig. 6-14 in Industrial Ventilation is such a chart. If two factors are known, the other two can be found. The effect of density will introduce errors no larger than 2% for densities corresponding to temperatures between 32° F and 100° F. Uncertainties in f, and duct design approximations will obscure such errors.

The magnitude of an air friction or dynamic loss can be expressed in several ways. At an elbow, duct expansion, branch entry, weather cap, etc., the resistance is often expressed as an equivalent length. An equivalent length is the length of straight pipe of the same diameter and surface that would have the same pressure drop. Tables of such values are given in Industrial Ventilation[2].

Another system for defining turbulent losses is to express the losses in terms of velocity pressure. A 0.25 $h_v$ loss means that the pressure drop is numerically equal to 1/4 of the velocity pressure of the air stream at that point. Pressure losses in terms of velocity heads for elbows, enlargements, contractions, entries, and weather caps, are given in Industrial Ventilation[2]. The entry losses in terms of velocity heads for hoods for specific operations as illustrated in Figs. VS-1 to VS-54 are also given.

A third means of expressing turbulent loss is in terms of entry coefficients. These are usually used to define entry losses at exhaust hoods, and abrupt changes in duct size. Entry coefficients for standard hoods are also given in Industrial Ventilation[2].

If the entry coefficient and static pressure of a hood are known, the airflow at standard conditions can be calculated by the formula:

$$Q = 4005\ A\ C_e\ \sqrt{h_s} \qquad (9)$$

where: $Q$ = airflow rate in cfm

$A$ = duct area in ft.$^2$

$h_s$ = static pressure at hood in inches of $H_2O$

$C_e$ = Coefficient of entry

Since: $Q = 4005\ A\ \sqrt{h_v}$ (6a)

where $h_v$ = velocity pressure in inches of $H_2O$

Then: $$C_e = \frac{\sqrt{h_v}}{\sqrt{h_s}} \qquad (10)$$

But according to Bernoulli's theorem, $h_s = h_v + h_L$

where $h_L$ = hood loss

If $h_L$ is expressed as N $h_v$; N being the hood loss as a fraction of the velocity pressure,

Then: $$C_e = \frac{\sqrt{h_v}}{\sqrt{Nh_v + h_v}} = \sqrt{\frac{1}{N + 1}} \qquad (11)$$

Therefore: $$N = \frac{h_L}{h_v} = \left(\frac{1}{C_e^2} - 1\right) = \left(\frac{1 - C_e^2}{C_e^2}\right) \qquad (12)$$

## Effective Specific Gravity

A common misconception in industry concerns vapors that are "heavier than air." If, for instance, a vapor has a specific gravity of six, relative to standard air, it is assumed that the vapor will quickly sink to floor level, and not be found at breathing levels. The fallacy arises from the fact that we are not concerned with concentrated vapor streams. While the tendency of a vapor stream to move downward is a function of its specific gravity, the specific gravities of vapor streams of hygienic significance are usually very little different from clean air. In Industrial Ventilation[2] an excellent illustrative example is given, using air concentrations of perchloroethylene (Sp. G. = 5.7, MAC = 200 ppm) of 200 ppm and 10,000 ppm. At 10,000 ppm, there would be 1 part $C_2Cl_4$ to 99 parts of air, and the effective specific gravity of the mixture would be:

| | | |
|---|---|---|
| 0.01 x 5.7 | = | 0.057 |
| + 0.99 x 1.0 | = | 0.990 |
| Effective Sp. G. | = | 1.047 |

At 200 ppm, a similar calculation gives an effective specific gravity of 1.0009. The specific gravity of the air stream, even at 10,000 ppm (50 times the MAC) does not approach the value of 5.7 that is often assumed. The effects of random industrial air movements would obscure the small specific gravity difference in almost all cases, and vapor specific gravity need not ordinarily be considered in planning control procedures. There are, however, certain instances where gases and vapors, generated close to the floor level and in the absence of air movement have concentrated to form pockets of explosive or asphyxiant gases. This possibility should always be considered at locations where heavy concentrations of vapor are generated.

## Airflow Characteristics of Blowing and Exhausting

Velocity contours plotted at the inlet and outlet of a round pipe are illustrated in Fig. 6.

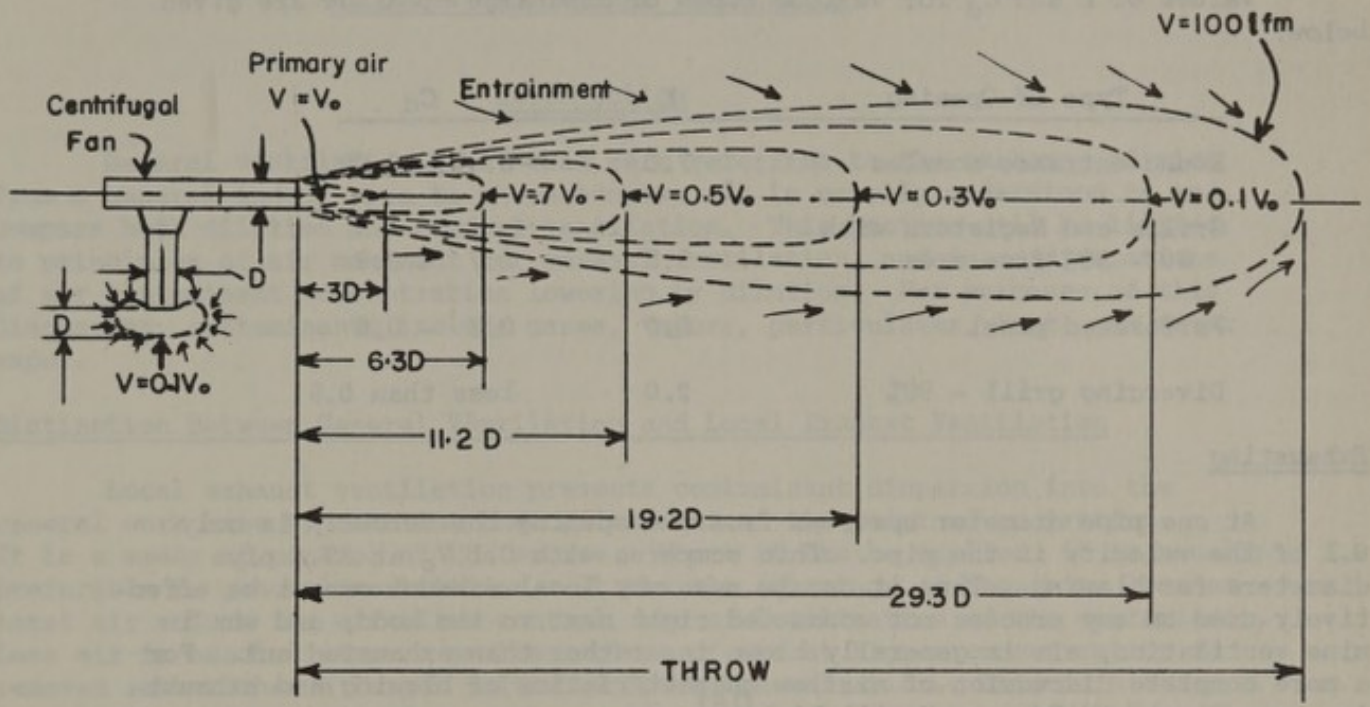



Fig. 6 - Velocity contours plotted at inlet and outlet of round pipe.

Blowing

At distances greater than three duct diameters downstream of the point of discharge, the velocity is inversely proportional to the distance from the discharge point. Throw is defined as the distance along the centerline at which the velocity is equal to 100 lfm.

In the region beyond three duct diameters, the distance from the discharge and the velocity at that point are related by the equation of Silverman:

$$X = \frac{KQ}{V_x\sqrt{A_f C_d}} \qquad (13)$$

where: $X$ = distance along centerline

$V_x$ = velocity at point X

$K$ = throw constant

$Q$ = volumetric flow rate

$A_f$ = free area of discharge

$C_d$ = coefficient of discharge

Values of K and $C_d$ for various types of discharge openings are given below:

| Type of Opening | K | $C_d$ |
|---|---|---|
| Round entrance nozzles | 7.0 | 0.97 - 0.99 |
| Grills and Registers with 60 - 90% free area | 5.5 | 0.7 - 0.8 |
| Perforated panel | 5.0 | 0.7 - 0.8 |
| Diverging grill - 90% | 2.0 | less than 0.6 |

Exhausting

At one pipe diameter upstream from the opening the velocity is only 0.1 of the velocity in the pipe. This compares with 0.1 $V_o$ at 29.3 pipe diameters for blowing. Thus it can be seen why local exhaust cannot be effectively used on any process not conducted right next to the hood, and why in mine ventilation, air is generally blown in, rather than exhausted out. For a more complete discussion of airflow characteristics of blowing and exhausting, see Plant and Process Ventilation[16].

## Make-up Air

Any air that is exhausted from a building for purposes of control must be replaced. If no provision is made for make-up air, it will enter by infiltration, and through windows and doors. If heated or conditioned air is discharged, provision must be made for tempering the replacement air to the desired conditions, which may constitute a major expense. It is far better to provide the make-up air with a supply system than to depend on infiltration, because the latter will produce drafts and an air-starved building with hard-to-open doors. If the contaminants in the exhaust air stream are efficiently removed, and if the contaminant is relatively non-toxic, considerable saving can be effected by recirculating the air. Extra care must be taken to prevent break down or inefficient operation of the air cleaning equipment when air is recirculated.

For detailed heating cost data and for cooling cost data for make-up air, see Chapters 12 and 13 of the Heating Ventilating Air Conditioning Guide[5].

# DILUTION AND GENERAL VENTILATION

## Introduction

General ventilation is a broad term referring to the exchange of air from a general indoor area to the outdoors. It is usually understood to encompass both dilution and comfort ventilation. This section will be limited to principles of air movement for general ventilation, and to considerations of air contaminant concentration lowering by dilution. For purposes of this discussion, contaminants include gases, vapors, particulates, heat, and water vapor.

## Distinction Between General Ventilation and Local Exhaust Ventilation

Local exhaust ventilation prevents contaminant dispersion into the general work area by removing the contaminant at the point of generation. It is a means of control at the source. Where it can be applied, it is usually preferable to general ventilation. It has the advantage of requiring less exhaust air volume for effective control, and of localization of the contaminant. Less air is handled since a smaller volume of air at higher concentration is removed rather than a larger volume at lower concentration. This is a very important consideration if the make-up air must be heated or cooled. In addition, localization of the contaminant has several other advantages. Among these are: (1) it will eliminate nuisance complaints in the general area; and (2) a contaminant that is a particulate or condensable vapor may accumulate and be a housekeeping problem even if it is reduced to a hygienically safe concentration by dilution.

The distinction between general and local ventilation is not always exact. Either system will act to a certain extent as the other type. If the exhaust system is difficult to classify as one type or the other, the following criterion can be applied. Where the concentration of the contaminant in the exhaust duct air is not significantly higher than that in the general room air, it is a general ventilation system; if it is much higher, it is a local exhaust system.

While local exhaust is preferable where it can be properly applied, there are circumstances under which its application would be difficult, and general ventilation would be the better method to employ. For instance, if the contaminant generation is relatively uniform throughout the area, and not at specific locations, general ventilation might be the only practical means of control. The conditions for successful application of general ventilation, as given in Industrial Ventilation[2], are: "(1) the quantity of contaminant generated must not be too great or air volume necessary for dilution will be impractical; (2) workers must be far enough away from contaminant evolution, or evolution of contaminant must be in sufficiently low concentrations so that workers will not have an exposure in excess of the established M. A. C. value; (3) the toxicity of the contaminant must be low; and (4) the evolution of contaminants must be reasonably uniform."

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Dilution ventilation is used most successfully to control vapors from substances for which the rate of evolution can be readily determined, and the required volume of dilution air calculated. Formulas for such calculations are given on page 6 of this section.

General ventilation in a room can be obtained using supply air, exhaust, or both supply and exhaust. If exhaust only is provided for, or if exhaust is provided in excess of supply, causing a negative pressure in the room, provision for the entrance of the make-up air must be made. If the room is under negative pressure, the air drawn in during cold weather may cause uncomfortable and unhealthy drafts. If supply alone is used, or if supply is in excess of exhaust, causing a positive pressure in the room, care must be taken than the excess air leaving the area does not contaminate adjoining occupied spaces.

## Expressing General Ventilation Rates

General ventilation rates are often expressed in such units as "air changes per hour," or "minutes per air change." These units express the ratio of the volume of air supplied or exhausted to the volume of the room. They are useful when applied to meeting rooms, offices, schools, warehouses, retail stores, laundries, and similar spaces where the purpose of ventilation is simply odor, temperature, and humidity control, and the only contamination of the air is from the activity of people. For purposes of control by dilution, however, such terms are of little value. Air changes per hour are dependent on the size of the room, while the quantity of air needed to dilute a given contaminant generated at a constant rate is not affected by the size of the room. Dilution ventilation rates should be expressed in cubic feet per minute or some other absolute unit of quantity flow. The room volume will determine the rate at which a concentration of contaminant will build up, and also the time required to purge the room when the contaminant generation is stopped. A large room will act as a surge tank, so that for an intermittent generation of contaminant, it may be safe to supply air to balance the average generation rate rather than the maximum.

## Air Movement in General Ventilation

If possible, it is desirable to introduce the supply air at one side of an area, and exhaust it at an opposite side, creating a continuous flow. Such movement is often disrupted by partitions or hot processes along the path, especially in large rooms, but it is worthwhile attempting in most cases. Short circuiting of the supply air to the exhaust should be avoided as much as possible. Physical proximity is not the only criterion for determining when short circuiting will develop. The air inlet and exhaust may be adjacent to each other without short circuiting[16]. Flow through an air inlet will act as a free jet if the exhaust fan maintains a negative pressure in the room. It was shown in Section C-3, "Principles of Ventilation Airflow," that the fan will exert very little influence beyond a velocity contour of about 50 lfm, which is just a short distance from the fan. The supply air will stream into the space in a straight line until its momentum is spent or it runs into an obstruction. In the case of the adjacent inlet and exhaust fan, there will not be short-circuiting if the throw of the supply air corresponds to the width of the room, but there will be, if the throw is much less than the room width.

Hemeon[16], in discussing air movement in general ventilation develops useful concepts of convective currents and circulation rates, mechanical circulation, and dilution at the source. He discusses solvent evaporation for point sources, area sources, and strip sources, and examines hood designs for solvent vapor control

Determination of Effective Ventilation Rate

The effective ventilation air quantity in a given area may be determined by $CO_2$ analysis.

$$Q = \frac{\text{No. of workers} \times CO_2 \text{ rate} + \text{other sources}}{(CO_{2_R} - CO_{2_O})} \qquad 60$$

where $Q$ = Effective ventilation rate in cfm

$CO_2$ rate = $CO_2$ production per man at light activity

= Approx. 1.2 cu. ft. per hour for adults

$CO_{2_R}$ = Fraction of $CO_2$ in room air

$CO_{2_O}$ = Fraction of $CO_2$ in outdoor air

Other sources = Other sources of $CO_2$ such as gas welding, burners, etc., for which the generation rate is known.

## Kinds of General Air Ventilation

Air change in a given space may be due to one or more of the following causes: infiltration, natural ventilation, or mechanical ventilation.

### Infiltration

Infiltration is the air leakage through cracks, around windows and doors, and through floors and walls. Its magnitude is dependent on the physical characteristics of the building, and the pressure and temperature differences between the building and its surroundings. It cannot be controlled by the occupants to any considerable extent.

The amount of infiltration in a given building is difficult to estimate because of: (1) variations in building construction; (2) variations in wind velocity and direction; (3) exposure of the building with respect to air openings and adjoining buildings; (4) variations in outside temperature which influence the chimney effect; and (5) the relative areas of openings on the windward and leeward sides of the building. A detailed examination of infiltration is not necessary in this discussion but it should be remembered that infiltration may have a significant effect on calculations for dilution air. A complete discussion of infiltration may be found in Chapter 11 of the Heating, Ventilating and Air Conditioning Guide[5].

## Natural Ventilation

Natural ventilation is the controlled displacement of air through openings, such as windows, doors, and ventilators, utilizing natural forces such as wind, and temperature (air density) difference. The air movement may be caused by one or the other, or a combination of the two.

Ventilation due to wind forces. In considering the use of natural wind forces for producing ventilation, account must be taken of average velocity, direction, and variation of the wind, and of local hills or buildings which might obstruct it. Tables of average wind conditions for various localities can be found in the Heating, Ventilating and Air Conditioning Guide[5]. If the natural ventilating system is designed for wind velocities of 1/2 the average seasonal velocity, it should prove satisfactory.

To calculate the air volume moved by wind forces:

$$Q = EAV$$

where Q = cfm of air

A = free area of inlet openings, $ft.^2$

V = wind velocity, lfm

E = coefficient = 0.5 to 0.6 when inlet faces wind
0.25 to 0.35 when inlet is at angle to wind.

The inlets should face the prevailing wind and the outlets should face the opposite side, or discharge into a low pressure area.

Ventilation due to air density difference. These forces will usually be encountered in hot industrial plants such as forges, foundries, and steel mills. The factors influencing ventilation are: (a) the heat production; (b) the vertical distance between inlet and outlet; and (c) the area of the inlet and outlet.

The volume moved by temperature (density) differences is determined by the draft head and the resistance:

$$Q = \sqrt{\frac{H}{2}\,\frac{(ta - to)}{RT}} \quad ; \quad R = \frac{1}{2gA^2C^2}$$

where Q = cfm of air - natural ventilation

H = vertical distance between inlet and outlet

(ta-to) = temperature difference between indoor and outdoor air, $^oF$

T = average absolute temperature, $^oR$

R = Resistance

g = gravitational constant

A = free area of inlet

C = coefficient = 0.65 in most cases
0.50 for restricted openings

Using an assumed T of 550° R, and C = 0.65, this reduces to

$$Q = 9.4\ A\ \sqrt{H\ (ta-to)}$$

When solving problems where the inlet and outlet areas are unequal, use the smaller area and make a correction from Fig. 1.

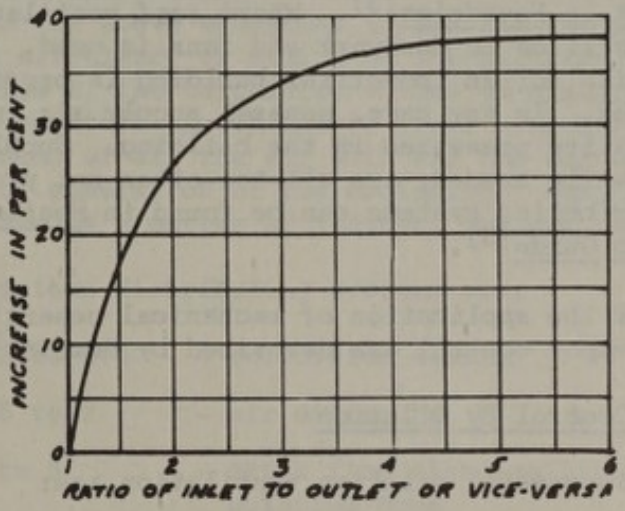



Fig. 1 - Increase in flow caused by excess of one opening over another. (Reprinted with permission from page 232, ASHAE Guide, 1955, Air Conditioning, Heating and Ventilating)

To estimate the combined effect of wind and temperature, calculate both, express the temperature difference effect as a percent of the total, and use Fig. 2. When the two heads are about equal, the combined effect will be about 10% greater than either. This percentage decreases as one head increases over the other. The effect of the larger head always predominates.

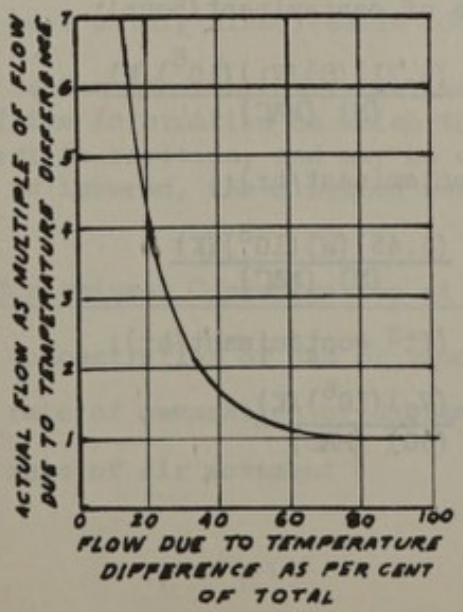



Fig. 2 - Actual flow caused by combined forces of wind and temperature difference. (Reprinted with permission from p. 233, ASHAE Guide, 1955, Air Conditioning, Heating and Ventilating.)

## Mechanical Ventilation

When infiltration and natural ventilation cannot provide the desired volume of ventilation air, as is usually the case, especially where contaminant dilution is required, then mechanical ventilation must be used. In small rooms, window and wall fans are usually used for exhaust. In large, single level industrial buildings, roof ventilators are commonly used as exhausters, taking advantage of the air density difference that exists. Commercial roof ventilators are available in many forms, from simple propellor types developing up to 0.25" $H_2O$ static pressure, to centrifugal types capable of producing higher static pressures (1-1/2" $H_2O$) and efficiencies. Five types of mechanical roof ventilators are illustrated in Engineering Manual for Control of In-Plant Environment in Foundries[3]. Where roof ventilators are not practical, exhaust through grilles to ductwork and fans is used. In many cases sufficient general ventilation for an industrial building is provided by local exhaust hoods within the area. In any case, general supply air is usually needed to prevent large negative pressures in the building. Supply air blowers and distributing ductwork, while needed, are all-too-often not provided. A good discussion of air distributing systems can be found in Heating, Ventilating, and Air Conditioning Guide[5].

Practical examples of the application of mechanical general ventilation to industrial solvent vapor control are described by Hemeon[16].

## Control By Dilution

Two fundamentals of successful dilution ventilation are:

1. Provide an adequate volume of uncontaminated air
2. Distribute the air uniformly

The amount of ventilation air required to dilute a given contaminant to a safe level, depends on the rate of generation of the contaminant, the efficiency or mixing of the ventilating air, and the threshold limit value of the contaminant. To calculate the amount of uncontaminated air required for adequate control by dilution, the rate of release of the contaminant must be determined and one of the following equations applied:

1. Liquid volume basis (pints of contaminant/hour):

$$ft^3 \text{ dilution air/min.} = \frac{(6.71)(S)(V_l)(10^6)(K)}{(M)\ (MAC)}$$

2. Weight basis (pounds of contaminant/hr):

$$ft^3 \text{ dilution air/min.} = \frac{(6.45)(W)(10^5)(K)}{(M)\ (MAC)}$$

3. Gas or vapor volume basis ($ft^3$ contaminant/hr):

$$ft^3 \text{ dilution air/min.} = \frac{(V_g)(10^6)(K)}{(60)\ (MAC)}$$

where: S = specific gravity of liquid contaminant

$V_l$ = pints of liquid contaminant vaporized/hr

$V_g$ = $ft^3$ gaseous contaminant released/hr

W = pounds of contaminant released/hr

M = molecular weight of contaminant

MAC = threshold limit value for contaminant in ppm

K = a factor as explained below

The factor K in the above equations is the air distribution factor, and is an estimate of the efficiency of mixing of the dilution air and the room air. If K had the value 1, the calculated volume of clean dilution air would be just sufficient to maintain the threshold limit concentration of the contaminant, if complete mixing of all the air with all the contaminant was accomplished and the rate of generation of the contaminant was constant. The selection of a proper K value is a matter of judgment for each individual problem.

K values for various distributing systems are:

K = 1.2 to 1.5 - perforated ceiling

K = 1.5 to 2 - air diffusers

K = 2 to 3 - trunk line along ceiling with branch jets pointing down

K = 3 and above - window fans, wall fans, etc.

Industrial Ventilation[2] contains air volumes per pint or pound required to dilute the vapors of 39 common organic solvents to the MAC level. As noted, these values should be multiplied by a K factor to allow for variations in uniformity of air distribution.

Hemeon[16] has a table of recommended dilution rates for 53 organic solvents. His "Ventilation Design Concentrations" are not threshold limit values but are based on both limits and odor. All of his concentrations are lower than the threshold limits, but those substances which are especially toxic, or which have a very disagreeable odor have the greatest safety factors.

In using tables of dilution rates, make a special note of dates of compilation, and of the information on which the values are based. Threshold limit values are subject to revision, and may be changed periodically. If the threshold limit value is lowered, the dilution rate should be correspondingly increased.

Calculation of Contaminant Concentration at Any Time

Let x = concentration of gas or vapor in air at time t

G = rate of generation of contaminant

Q = rate of air movement

$K$ = distribution constant

$Q' = \frac{Q}{K}$ = effective rate of air movement

$V$ = volume of room

The material balance for the contaminant will be:

$$Vdx = Gdt - Q'xdt$$

If the rate of generation G, and the rate of airflow Q, are constant, then:

$$Vdx = (G - Q'x)dt$$

$$\int_{x_1}^{x_2} \frac{Vdx}{(G - Q'x)} = \int_{t_1}^{t_2} dt$$

$$V \left[ -\frac{1}{Q'} \ln (G - Q'x) \right]_{x_1}^{x_2} = t_2 - t_1 = \Delta t$$

$$\Delta t = \frac{V}{Q'} \left[ \ln (G - Q'x_1) - \ln (G - Q'x_2) \right]$$

$$\Delta t = \frac{V}{Q'} \ln \left[ \frac{G - Q'x_1}{G - Q'x_2} \right]$$

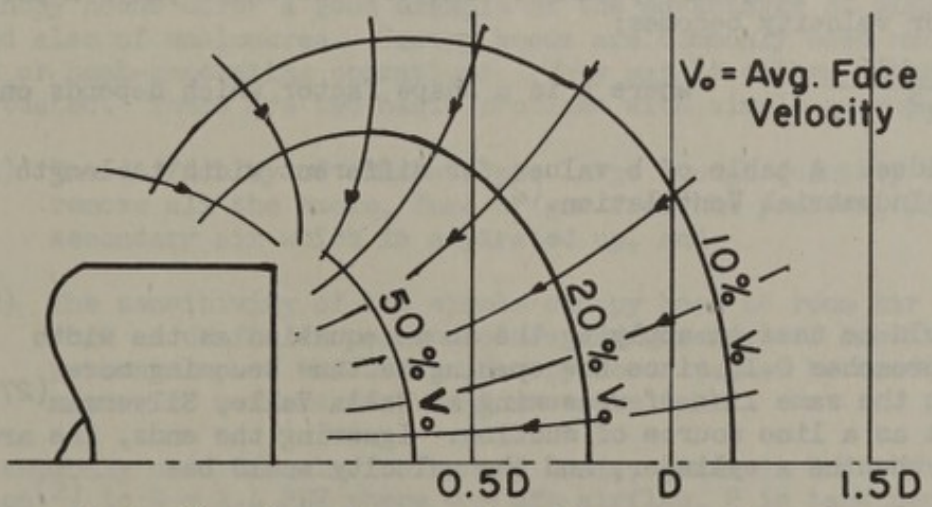
# LOCAL EXHAUST SYSTEMS

## Introduction

In the majority of cases, control of an atmospheric contaminant, gas, dust or vapor, can be accomplished by local exhaust ventilation, either alone or accompanied by enclosure, better process control or other measures. The basic principles governing the use of local exhaust ventilation were presented by Dalla Valle in 1932.(11) In spite of the fact that these principles have been available for more than 25 years, common misconceptions still are prevalent. One of these is that entrance into a remote hood at a higher velocity will give better control. This is not true. If we measure the air velocity toward the hood 6" from a 6" diameter open pipe end carrying a total volume of 350 cfm, we find it is 130 fpm. If we decrease the pipe size to 4" with the same flow, we increase the face velocity 2.5 times, the static pressure and power requirement 6.4 times, but the velocity 6" away is only 4% higher.

## Airflow into Openings

Dalle Valle's theory of airflow is based on the postulation of a point source of suction. Into such a point source air would flow from all directions. The velocity at any point a distance X from the source would then equal the quantity divided by the area of the sphere of radius X, or $V = \frac{Q}{4\pi X^2}$. The closest approach to a point source is an open pipe end. Air flows into the open pipe end from all directions. A diagram of the equal velocity contours for a circular opening is shown below:




Velocity Contours for Circular Opening

After DallaValle & Silverman

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The velocity along the centerline of such a pipe decreases rapidly with distance from the pipe end, becoming approximately inversely proportional to the distance, as predicted by theory. For an open pipe end, the actual velocity as given by Dalla Valle is $V = \frac{Q}{10X^2 + A}$ where V is velocity at point X in feet per minute Q is flow in cfm, X is centerline distance from the pipe in feet, and A is pipe area in square feet.

Addition of a flange to the pipe will obviously improve the velocity contours, since it will prevent flow into the pipe from the back, and thus increase its effectiveness. Where the hood can be placed on a table, this will cut off another 90° sector from which air is drawn, and further increase effectiveness of the hood. The velocities under these conditions are then:

(1) Open pipe end: $V = \frac{Q}{10X^2 + A}$

(2) Flanged pipe end: $V = \frac{Q}{0.75(10X^2 + A)}$

(3) Flanged pipe on table: $V = \frac{Q}{0.5(10X^2 + A)}$

Only half the flow will be required in the last case to achieve the same velocity at a point X feet from the hood. The importance of the best possible hood design is thus obvious.

Airflow into a square opening will be quite similar to that into a circular opening. For rectangular openings, however, the shape plays a part, and the equation for velocity becomes:

(4) $V = \frac{bQ}{X^2 + bA}$ where b is a shape factor which depends on the ratio of the sides. A table of b values for different width to length ratios is given in Industrial Ventilation.(2)

Slots

Caution should be used in applying the above equation as the width to length ratio approaches 0.1, since the opening is thus becoming more like a slot. Using the same line of reasoning as Dalla Valle, Silverman(27) considered the slot as a line source of suction. Ignoring the ends, the area of influence would then be a cylinder, and the velocity would be:

$V = \frac{Q}{2\pi X L}$ where X is the perpendicular distance from a slot of length L. In practice, of course, the theoretical conditions do not exactly apply, and the constant used is thus not 6.28, but 3.7 and the equation becomes:

(5) $V = \frac{Q}{3.7 X L}$

for freely suspended slots. Flanging a slot will give the same sort of

benefits as flanging an open pipe, so that only 75% of the air is required to produce the same velocity at a given point in front of the slot, and thus:

(6) $V = \frac{Q}{2.8 \times L}$. Where the area of influence is still further reduced because the slot is in a corner, velocity contours will be again improved.

Dispersive Forces

The above discussions have all been based on still air. Actually, in many operations natural forces or air currents will influence the air flow pattern about the operation to be controlled. Such influences will include the rising current of warm air above a hot process, the aspiration of air downward by falling rocks from a conveyor, the rotative air currents about a grinding wheel, natural air currents caused by room ventilation, and similar external forces. Hemeon(3) discusses these dispersive forces in detail, giving their influences on hoods and air flows, and methods of correcting them. In the design of hoods it is preferable to take advantage of these dispersive forces whenever possible. The standard grinding wheel hood design, for example, takes advantage of the motion of the chips or fragments from the wheel. While these large particles, which are projected long distances, are of no hygienic importance, they do create an aspirated air current which carries small particles along with them into the hood and also their collection aids in housekeeping. In woodworking operations, housekeeping and fire prevention are the major factors in control so that collection of the large, non-respirable particles is most important.

Canopy Hoods

Canopy hoods offer a good example of the advantages of dispersive forces and also of enclosures. Canopy hoods are commonly used over hot processes or heat-generating operations. They may be either natural draft or fan actuated. There are two basic problems with simple canopy hoods:

(1) The difficulty of exhausting a large enough quantity of air to remove all the smoke, fume or gas from the process, plus the secondary air which is aspirated up, and

(2) The sensitivity of the simple canopy hood to room air currents such as drafts from man coolers and movement of machinery and personnel past the hood. Canopy hoods cannot be used for toxic vapors if workers must bend over the tank.

A commonly used formula for canopy hoods, given in Industrial Ventilation(2) is Q = 1.4 PHV where Q = cfm airflow, P is tank perimeter in feet, H is height of the hood above the tank, and V is average vertical velocity of air under edges of canopy in fpm. If this formula is used the difficulty is in selection of V, which is said to range from 50 - 500 fpm depending on cross-drafts. The formula does not take into account the process going on underneath it. Hemeon(16) and Silverman have attempted to take into account the process and the amount of hot air which the hood is

required to remove. Hemeon's formulae, which require some explanation, are found in chapter 8, "Exhaust for Hot Processes."[16] Silverman uses the formula:

$$Q = 278\, A \sqrt{H''\left(1 - \frac{Ta}{Ts}\,\frac{Ms}{Ma}\right)}$$

where Q = air volume, cfm

A = tank area

H" = height in inches from liquid level or tank top to exhaust hood

Ta = Absolute temperature of air

Ts = Absolute temperature of substance

Ma = Molecular weight of air (In practice let $\frac{Ms}{Ma} = 1$)

Ms = Molecular weight of substance

This formula for total air to be removed is derived from the theoretical convective velocity, and the form shown here assumes an entrainment ratio of 4.

In many simple canopy hoods material will be observed "spilling" out the sides. One attempt to remedy this has been the so-called double canopy hood where an inner cone or pyramid blocks all but the outer rim of the hood, with the idea that a high velocity at the edges will solve the problem, since spillage is occurring only at the edges. The previous comments about high velocity entrances apply - the double hood does nothing except change air distribution, and if better distribution is needed it may be more easily obtained with simple baffles which distribute the air volume throughout the face area, not just on the outside.

All canopy hoods are very subject to cross-drafts, and should be avoided where possible. Considerable improvement can be achieved by enclosing one or more sides. Enclosure will also reduce air entrainment from hot processes, thus making control possible with a smaller quantity of air.

Where an enclosure can be used, it will benefit any local exhaust system. For some materials such as very finely divided solids, enclosure is the only solution, since any face velocity adequate for control without enclosure would cause loss of product. An example is the highly toxic, finely divided silica with a mean size of less than one micron.

Air Distribution in Hoods

Air flow into a hood should be uniform throughout its cross-section. In the case of slots for lateral exhaust, for example, this may be done by "fish-tail" design, where the design attempts to obtain equal velocity through the "fish-tail." An easier and also effective method of design, however, is to provide a velocity of 2000-2500 feet per minute into the slot, and use a low-velocity plenum or large area chamber behind it. The 2000 fpm velocity will give a static pressure behind the slot of 0.4 inches of water. This pressure drop will thus equalize flow along the slot, since the static

pressure throughout the plenum will be practically uniform. For large shallow hoods such as paint spray booths, lab hoods, side draft shakeout hoods and the like, the same principle may be used. In these cases, unequal flow may tend to occur, being concentrated in the neighborhood of the take-off; baffles provided for the hood cause increased velocity and pressure drop into the hood, giving the plenum effect. Where the face velocity over the whole hood is relatively high, or where the hood or booth is quite deep, no baffles will be required.

Capture Velocities

The capture velocity is that velocity in front of the local exhaust hood that is necessary to overcome the dispersive forces and room air currents and capture the contaminant. The table below from Industrial Ventilation(2) gives the range of velocities:

Table 4-1

Range of Capture Velocities

| Condition of Dispersion of Contaminant | Examples | Capture Velocity, fpm |
|---|---|---|
| Released with practically no velocity into quiet air. | Evaporation from tanks; degreasing, etc. | 50-100 |
| Released at low velocity into moderately still air. | Spray booths; intermittent container filling; low speed conveyor transfers; welding; plating; pickling | 100-200 |
| Active generation into zone of rapid air motion | Spray painting in shallow booths; barrel filling; conveyor loading; crushers | 200-500 |
| Released at high initial velocity into zone of very rapid air motion. | Grinding; abrasive blasting, tumbling | 500-2000 |

Capture velocities for specific operations are given in Industrial Ventilation, and the ASA Standard Z9.1 for Ventilation and Operation of Open Surface Tanks gives capture velocities for such tanks. For open surface tanks the corners have the minimum surface velocity, but may give off only a small amount of contaminant even if not well controlled.

Entrance Losses in Hoods

As discussed in the introduction to ventilation principles, the static pressure just downstream from a hood is the sum of two effects:

(1) The velocity pressure in the pipe which represents kinetic energy and is convertible to static pressure; and

(2) The losses in the entrance, because of contraction and re-expansion of the air stream, friction and direction changes in the hood.

Because of these pressure losses, the velocity pressure in the duct at the hood is not numerically equal to the static pressure. Thus the velocity (V) in feet per minute is:

$$V = 4000 \sqrt{v.p.} \quad \text{or} \quad V = 4000\ C_e \sqrt{s.p.}$$

where v.p. and s.p. are velocity pressure and static pressure respectively, in inches of water, and $C_e$ is a coefficient of entry introduced to take care of the hood pressure losses. Another commonly used term is the "hood entry loss" ($h_e$). This is the pressure loss in the hood expressed in inches of water. The ratio of the hood entry loss to the velocity pressure is also used. This "hood entry loss factor" is used in the various specific operation plates in Industrial Ventilation. The entry loss for a standard grinding wheel hood is thus given as "0.65 VP." The coefficient of entry for this hood would then be:

$$C_e = \sqrt{\frac{VP}{SP}} = \sqrt{\frac{VP}{VP + 0.65VP}} = \sqrt{\frac{1}{1.65}} = 0.78$$

A concept commonly used in ventilation design work is that of "velocity head." A "velocity head" (which is the same as the velocity pressure) is equal to the square of the average cross-sectional velocity divided by 4000, or 1 velocity head equals $\left(\frac{Vav}{4000}\right)^2$ inches of water. Since industrial exhaust systems are usually designed at a constant velocity (plus or minus 20%), and the velocities are quite high, compared with liquid flow systems, this expression of losses ih terms of "velocity heads" is one convenient method of summing up losses in the system. It can, however, result in some confusion to one unfamiliar with the terminology. Industrial Ventilation[2] gives coefficients of entry as well as entry losses in terms of velocity head for a number of simple hood shapes. Most of the coefficients for more complicated hoods are obtained by combining some of these simple shapes. One may observe, for example, that the "direct branch booth" is actually the same as a flanged duct end, since the pressure drop at the low velocity booth entrance is negligible, and the loss all occurs at the branch take-off from the booth. The diagrams show how this entrance loss may be reduced by a tapered take-off. The orifice plus flanged duct shown is a common type hood, and is an example of combining two pressure drops to obtain the entrance loss for the hood. The loss through a sharp-edged orifice is given as $h_e = 1.78$ v.p., or the pressure loss in inches of water through the orifice is:

$$h_e = 1.78\left(\frac{V(\text{orifice})}{4000}\right)^2$$

After the reenlargement, there is another loss as the air enters the take-off which is identical with a flanged duct end. The entry loss for a flanged duct end is given as 0.49 v.p., so the total hood entry loss for the orifice

plus flanged duct is 1.78 v.p. (orifice) + 0.49 v.p. (duct), or

$$h_e = 1.78\left(\frac{V_{(orifice)}}{4000}\right)^2 + 0.49\left(\frac{V_{(duct)}}{4000}\right)^2$$

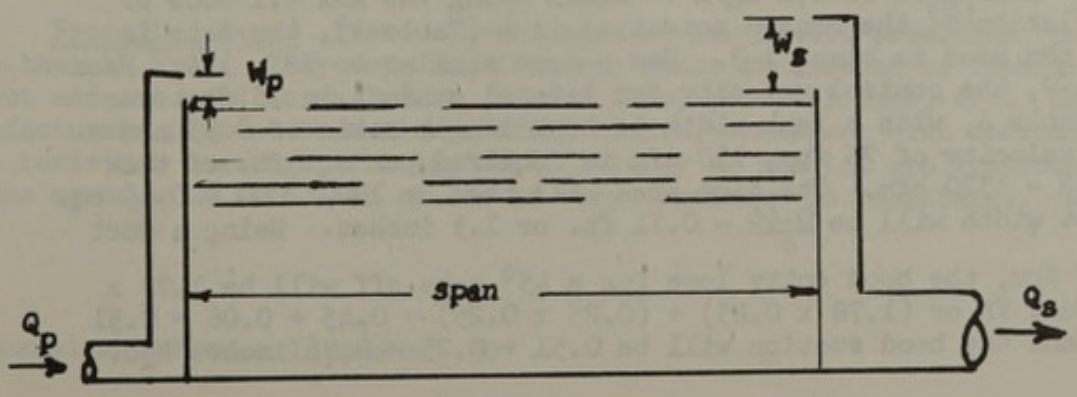
The static pressure would include one duct v.p., and would be 1.78 orifice v.p. + 1.49 duct v.p. If there were a tapered take-off rather than the straight take-off shown, then the graph of taper angle vs. hood entry loss factor adapted from Brandt's data would be used to estimate the pressure loss in the duct entry.

Static Suction

One method of specifying air volume for a hood is to specify the static suction and pipe size. It has long been recognized that the static pressure at an average grinding wheel hood is 2 inches of water. This reflects a conveying velocity of 4500 feet per minute and an entrance coefficient ($C_e$) of 0.78. In the same way, for many pieces of machinery where the type of exhaust hood is relatively standard, a specification of the static suction and the pipe size is given in Alden[1] and other reference sources. Specification of the static suction without pipe size is, of course, meaningless, since decrease in pipe size will increase the velocity pressure and thus the static suction, while actually decreasing the total flow and the degree of control. Static suction measurements are quite useful for standard hoods, or for systems where the air flow has previously been measured, to estimate the quantity of air flowing through the hood.

Blow and Exhaust (Push-Pull) Hoods

The characteristics of an air jet, with its long zone of influence, naturally suggest the idea of a blowing-exhausting system for tanks too wide to be handled by exhaust on two sides. This necessitates some consideration of the characteristics of a free air jet. Without going into the theory of jet streams, which may be found, with references to original work, in reference[16] Chapter 9, it is easy to see that the plane surface issuing from a blowing slot would gradually expand, so that the exhausting slot would require a greater area. Also, as the high velocity jet of air travels along it will aspirate, or entrain, more air with it. This means that the exhausting slot must be wide enough to include the entire jet, and that the exhaust capacity must be sufficient to handle the primary plus the secondary entrained air. In spite of the theory summarized in (16), design of push-pull hoods in the past has been largely empirical. Industrial Ventilation,[2] gives one such empirical design. Similar design criteria from the American Foundrymen's Society are as follows:



$$Q_p = 4000\ A_p$$

and $$Q_s = 2000\ A_s$$

where: $Q_p$ = air volume of supply, cfm.

$A_p$ = area of supply slot, $ft.^2$

$Q_s$ = volume of exhaust, cfm.

$A_s$ = exhaust slot area

Slot widths are suggested as follows:

| Span (ft) | $W_p$ (inches) | $W_s$ (inches) |
|---|---|---|
| 2 | 1/8 | 2 |
| 4 | 1/4 | 4 |
| 6 | 3/8 | 6 |
| 8 | 1/2 | 8 |
| 10 | 3/4 | 10 |

A sample comparison of three methods of design for a 4 foot span push-pull tank is:

| | $Q_p$/ft | $Q_s$/ft | $W_p$ | $W_s$ |
|---|---|---|---|---|
| AFS | 83 | 667 | .25" | 4" |
| Ind. Vent. | 63 | 500 | .5 " | 8.5" |
| Hemeon | 96 | 480 | .24" | 12" |

The only value which appears to be greatly out of line is the 4" exhaust slot width suggested by the A.F.S., which is considerably narrower than suggested by others.

Example of Hood Selection

Select a suitable hood and the quantity of exhaust ventilation necessary for a 2' x 6' chromic acid plating tank operated at 120° F.

From the ASA Standard tables given in Industrial Ventilation,(2) the major contaminant released by such a tank is chromic acid mist. The threshold limit for chromic acid mist is 0.1 mg/m . Again using the ASA 9.1 code of Industrial Ventilation(2) the hazard potential is A (Table 1), the rate is 3 (Table 2), so the hood is class A-3. Use a hood similar to VS - 35A. From Table 3, page 14-9, the control velocity for lateral exhaust is 75 feet per minute. Using Table 4, with a tank width to tank length ratio of 0.33 and a minimum control velocity of 75 fpm, 110 cfm is required per sq. ft. of tank area, or 110 x 12 - 1320 cfm. The slot area will then be 1320/2000 = 0.66 sq. ft., and the slot width will be $\frac{0.66}{6}$ = 0.11 ft. or 1.3 inches. Using a duct velocity of 2000 fpm, the hood entry loss for a 45° take-off will be 1.78 x slot VP + 0.25 Duct VP or (1.78 x 0.25) + (0.25 x 0.25) = 0.45 + 0.06 = 0.51 inches of water and the hood suction will be 0.51 + 0.25 = 0.76 inches $H_2O$.

# FANS

## Introduction

Fans are devices which move air at pressures low enough so that the compressibility of the air can be neglected, permitting inlet and outlet volumes to be considered alike. There are other types of devices for moving air such as ejectors and blowers, and air can be moved by natural forces, such as wind and temperature difference, but for moving large volumes of air at low pressure upon demand, fans are most often used.

Fans are usually divided into two groups, centrifugal fans and axial-flow fans. The distinction between the two is the direction of air flow produced. Air enters a centrifugal pan parallel to the fan shaft, and is discharged in a plane perpendicular to it. Axial-flow fans create a flow parallel to the fan shaft.

To be effective, any fan must increase the static pressure in the air passing through it. Potential energy due to the static pressure produced can then be utilized to move air. The increase in pressure developed in a fan arises from two sources: one is the centrifugal force caused by the rotation of an enclosed volume of air, and the other is the increased velocity in the direction of flow imparted to the air by the blades. The centrifugal force causes a compression of the gas, or in fan language, static pressure, which is dependent on the increase in the centrifugal component of the velocity of the air. The longer the blades, the greater the static pressure developed. Increased velocity in the direction of flow on the other hand, causes an increased velocity pressure, which must be converted to static pressure in order to be effective. Total pressure remains constant in such a conversion since it is simply the algebraic summation of static and velocity pressures. Conversion in both centrifugal and axial-flow fans can be performed by using a constantly diverging discharge duct at the fan outlet, and is aided by an efficient impeller arrangement and shape. With centrifugal fans, scroll, or increase in cross section, also aids conversion.

## Fan Types

### Axial-Flow Fans

By definition, axial-flow fans create air flow parallel to the fan shaft. Within this group are low pressure propeller and duct fans, and also vane-axial fans which can develop higher pressures.

Propeller fans. These operate against low static pressures (usually less than 1" $H_2O$). They move air from one space to another or from indoors to out and are used as wall fans, window fans, pedestal fans, etc. Some manufacturers produce them with airfoil blades, sometimes with corrosion and heat resistant coatings, for higher efficiency or special applications. Air volume moved with this type of fan is very sensitive to resistance. A small

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increase in resistance above as little as 0.1 or 0.2" $H_2O$ causes a sharp reduction in flow. The mechanical efficiency of propeller fans seldom rises above 30% to 40% and may even be lower.(16)

Tube-axial fans. They are similar to propeller fans but are located in short sections of duct. They are most suitable for moving air containing materials that tend to collect on fan blades. Air velocity through a duct containing a tube-axial fan should not exceed 1500 fpm.(16)

Vane-axial fans. Tube-axial fans with guide vanes either ahead of or following the impeller are classed as vane-axial fans. These vanes straighten the helical flow pattern and convert velocity pressure into useful static pressure, increasing the efficiency. The impeller blades usually have an airfoil shape. Vane-axial fans are best suited to clean air applications; they are far more efficient, and can operate at much higher pressures (up to 11" $H_2O$) with lower noise levels than propeller fans. Vane-axial fans are competitive with centrifugal fans for most industrial operations. At static pressures below about 4" $H_2O$ they are quieter than centrifugal fans. At higher pressures they may be noisier, but are not necessarily so. Their efficiency may be as high or higher than centrifugal fans and their compact design and simplicity of mounting and erection make them a good choice when conditions permit their use. In the very high capacity range, vane-axial fans are superior to centrifugal fans, possibly except for the production of more noise.

AXIAL FLOW FAN CHARACTERISTICS

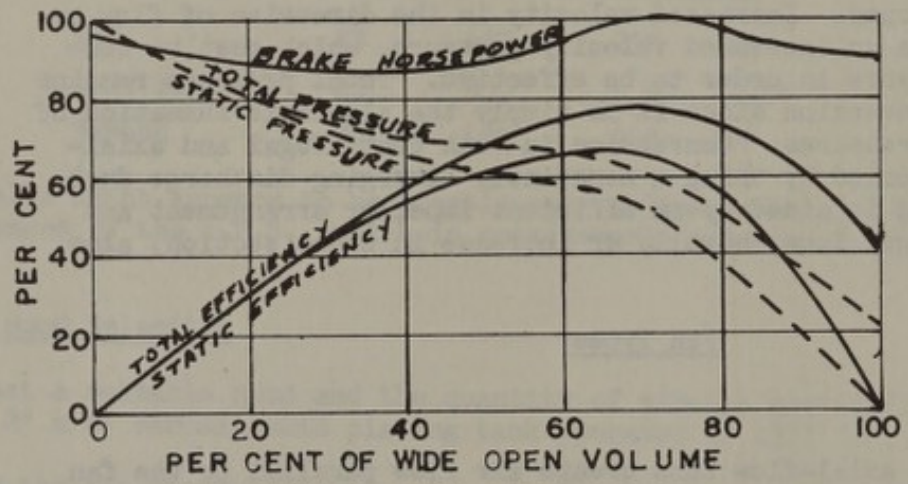


Figure 1

Fig. 1 shows characteristic curves for an axial-flow fan. The actual characteristics for a given fan would depend to a great extent on the type of fan and the type of blades. Axial-flow fans have their highest horsepower requirement and static pressures at the blocked-tight position.

Centrifugal Fans

Centrifugal fans are usually used in industry for supply and exhaust systems, pneumatic conveying, or forced draft applications. These fans create a radial flow since air enters parallel to the fan shaft, turns 90°, passes between the blades of the fan wheel, and discharges in a plane perpendicular

to the shaft. Centrifugal force builds up the velocity and static pressure of the air. There are three types of centrifugal fans, determined by the type of fan blade. The difference in performance of the various types shows up in the way they vary in capacity with varying pressure (see Figs. 1, 2, 3 and 5), in the magnitude of resistance they can operate against, and in the rotational speed required for a given capacity. The speed required is an important factor in maintenance and noise considerations.

Radial blade or paddle-wheel fans. Paddle-wheel fans were the first type developed, and are the only type that should be used for handling dirty air. Centrifugal force tends to keep the blades clean, permitting applications where suspended solids must pass through the fan. The rotors usually have between 5 and 12 blades, and the blades and housing are ruggedly fabricated. Characteristic curves for this type of fan (Fig. 2) show the kind of performance that can be expected. The pressure characteristic is stable and shows only a minor reduction from the peak pressures at the blocked-tight position. Horsepower required is almost a straight line function of air volume. Maximum static efficiency for this type of fan is about 65% to 70%. Not all radial bladed fans are paddle-wheel fans. Inclination of the blade tip determines the type of performance, so that an angled blade with a radial tip performs as a radial bladed fan. Such fans are now coming into use.

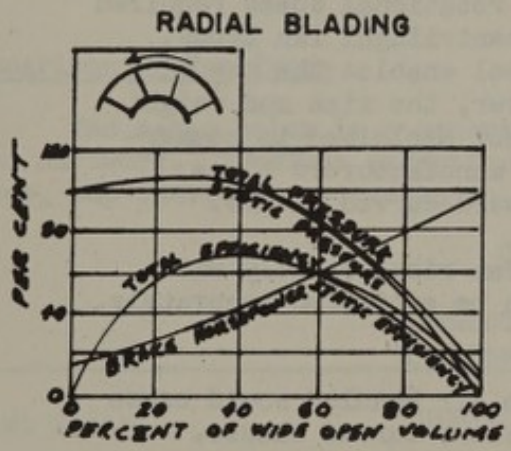


Figure 2

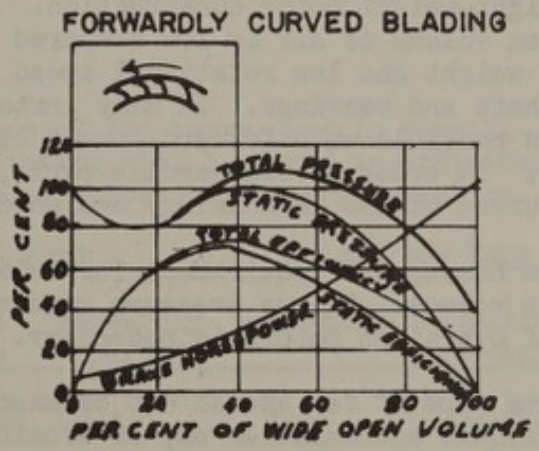


Figure 3

Forward-curved blade fans. These are usually multibladed having 20 to 64 shallow blades curving forward in the direction of rotation. Forward curving is designed to increase the velocity component of the air leaving the tip of the wheel beyond the rotational velocity (see Fig. 4), building up a high proportion of kinetic energy. This is partially converted in the scroll to useful potential energy (static pressure).

Fig. 3 shows the performance characteristics of this type of fan. Because of the characteristic dips in the pressure curves, stability of operation is uncertain unless the operating point is on the wide-open side of the pressure peak. Maximum efficiencies may be about 70%, but because of possible

instability, the selected point of operation is usually in the lower efficiency range. Usual operating static pressures in ventilating applications are from 2 - 4" $H_2O$, although forward-curved fans are available that will develop higher pressures.

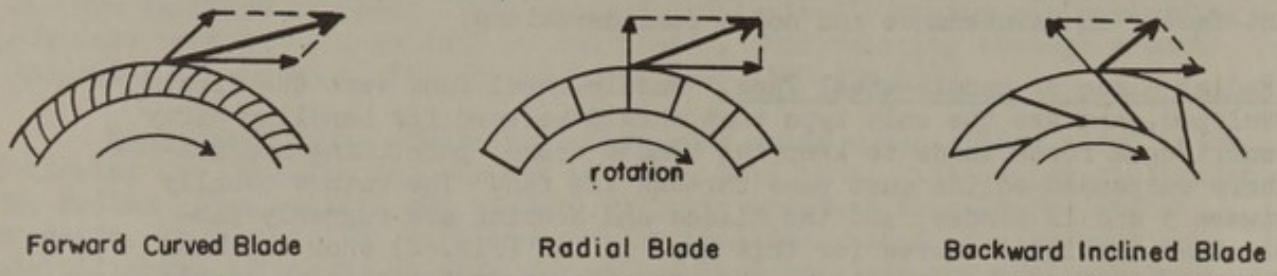


Fig. 4. Velocity vectors of air leaving centrifugal fans. Heavy arrow is the resultant of the radial and forward components.

The scroll of a forward-curved bladed fan can be wrapped more tightly around the wheel than other fan types because of the relations shown in Fig. 4. This permits a smaller casing size for a given size rotor, and multiple blading permits light-weight rotor construction. Also, the rotational speed required for a given volume of air is low compared to other centrifugal fan types. The light weight and low rotational speed of the wheel enables the use of a smaller shaft and bearings. In many instances however, the size and weight advantages possible with forward-curved blades are not exploited in commercial fans. In order to standardize parts, many fan manufacturers place forward-curved blades in housings designed for backward-curved blades.

The horsepower requirement for this type of fan rises rapidly with increasing volume. Unless pressure requirements can be estimated accurately, the use of oversized motors is necessary.

This type of fan should not be used where the air handled would cause solid or liquid accumulation on, or erosion of the short curved blades.

Backward-curved blade fans. These are the most recent development and are becoming increasingly popular. The horsepower characteristic curves downward as volume approaches wide-open, so that over-sized motors are not needed. Blades in this type follow the natural path of the air through the fan, so that the air tends to follow the blades even at high volume conditions. This permits a design point closer to the wide-open volume, and the efficiency remains high in the high-volume range.

Air velocities leaving the blade tips are low (see Fig. 4), and most of the static pressure increase is brought about in the fan wheel itself. In practice, therefore, the efficiency of this type of blading is higher (70% to 80%) than the other types.

Disadvantages of this fan type are: (1) the rotational speed must be high to compensate for the low velocity of the air leaving the tips; (2) the wheel, shafts, and bearings must be ruggedly built to withstand the higher speeds; and (3) high tip speeds may cause high noise levels. Many fan

manufacturers are building backward-inclined fans with airfoil blades since such fans have higher efficiencies and lower noise levels. Backwardly-inclined fans are the most widely used type for clean-air application.

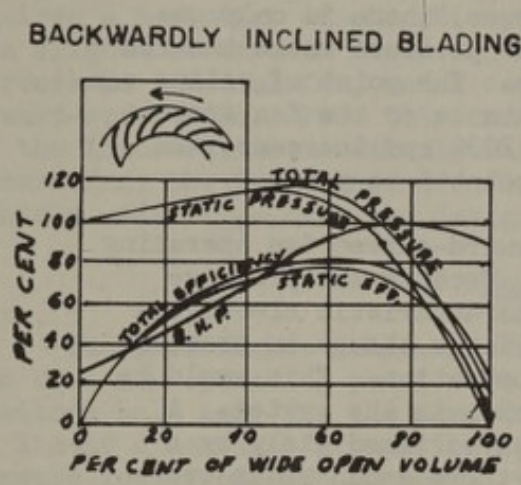


Figure 5

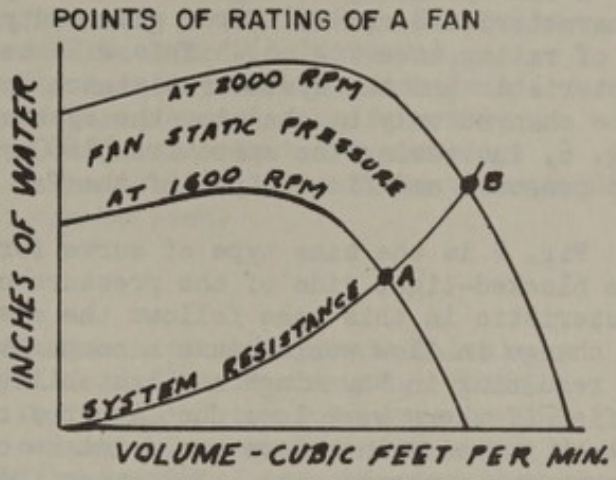


Figure 6

Comparison of Fan Types

The table below is a general comparison of fan types used in ventilation ductwork. Numerous exceptions to the listing will be found in individual cases, but these data serve as an indication of comparative characteristics.

| | Centrifugal Fans | | | Axial-Flow Fans | |
|---|---|---|---|---|---|
| | Backwardly Inclined | Radial Blade | Forwardly Curved | Vane-Axial | Propellor & Tube-Axial |
| First Cost* | High* | Medium* | Low* | Low | Lowest |
| Efficiency | High | Medium | Medium | High | Lowest |
| Stability of Operation | Good | Good | Poor | Good | Poor |
| Space Required | Medium | Medium | Med-Small | Small | Small |
| Tip Speed (Noise) | High | Medium | Low | Medium | High |
| Resistance to Abrasion | Medium | Good | Poor | Medium | Medium |
| Ability to Handle Sticky Materials | Medium | Good | Poor | Medium | Medium |

*On normal duty applications, first cost is essentially the same for all three types of centrifugal fans. Heavy duty applications reflect the comparison listed.

## Point of Rating of a Fan

A fan operating at a given speed can have any desired rating along its characteristic curve. For a given duty, however, there is only one point of rating (see Fig. 6). This will be at the pressure where the fan characteristic and the system resistance coincide. The point of rating can then be changed only by changing the system resistance or the fan speed. In Fig. 6, increasing the speed from 1600 rpm to 2000 rpm increases the static pressure and flow output of the fan from point A to point B.

Fig. 7 is the same type of curve for a forward-curved fan operating on the blocked-tight side of the pressure curve. Because the pressure characteristic in this case follows the system characteristic closely, a small change in flow would cause a comparatively large change in pressure drop, resulting in "hunting" and instability of operation. This would be magnified if there were long duct lengths or plenums in the system. A momentary increase in volume would not be compensated immediately by a corresponding pressure rise. Following this lag there would be an over-compensation, and the flow would begin to surge. This effect is most pronounced with forward-curved fans, but can occur with radial and backward-inclined fans also.

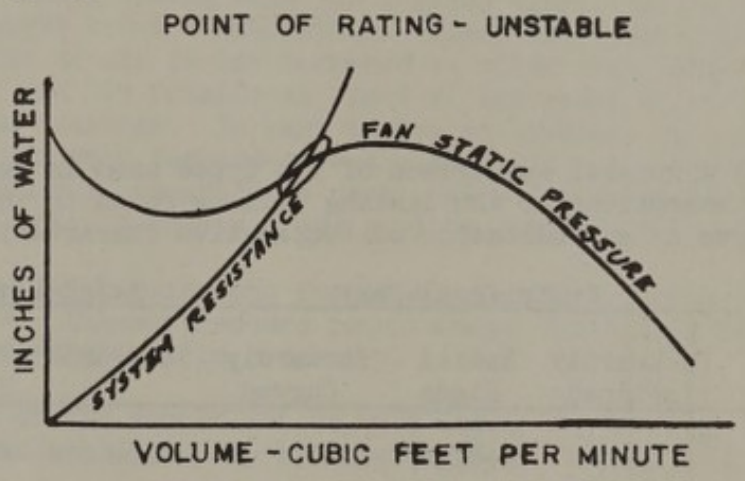


Figure 7

## Effect of Pressure Change on Power Consumption

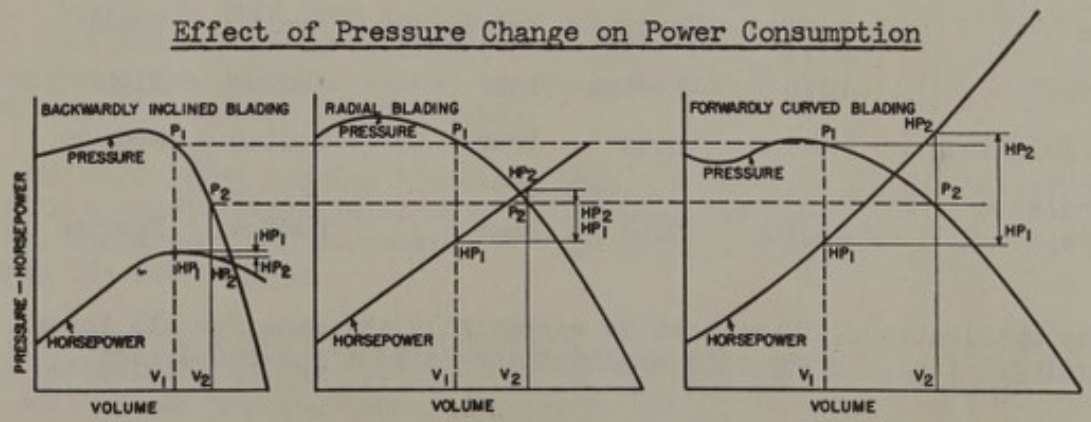


Figure 8 -- Reprinted by permission from Air Conditioning, Heating and Ventilating, May 1957. Copyright - The Industrial Press.

Fig. 8[25] demonstrates the effect a change in system resistance can have on power consumption of the three types of centrifugal fan. In this example $P_1$, $P_2$, and $V_1$ are equal for each fan type, and except for efficiency difference $HP_1$ is approximately equal. A reduced system resistance requires a new point of operation, and in the case shown in Fig. 8 results in a slight decrease in power required from a backward-inclined fan, an increase in a radial fan, and a considerably larger power increase in a forward-curved fan. From this example, and the discussion on fan rating, and the inaccuracies in calculating system resistance that are normally encountered, the importance of selecting a fan that will be stable throughout a fairly wide range near the design point can be seen.

## Fan Laws

Fan performance follows certain relationships that permit calculation of a new operating point when the system airflow is changed. These relations hold only for the same fan, or for a fan of the same type having all linear dimensions proportional, when exhausting a given system where the pressure losses of each component vary as the square of the velocity through that component. The laws will not apply where changes are made in the number or size of duct branches, or where fabric arresters or certain wet collectors are used, in which resistance does not increase as the square of the velocity.

The variables are:

$Q$ = volumetric flow rate - usually in cfm

$P_s$ = fan static pressure - usually in in. $H_2O$

$P_t$ = fan total pressure - usually in in. $H_2O$

$HP$ = horsepower input

$N$ = rotational fan speed - usually in rpm

$S$ = size of fan - expressed as some linear dimension of the fan. Some manufacturers use inlet diameter, some,wheel diameter, etc.

$D$ = density of the air or gas stream

$P_a$ = barometric pressure

$T$ = absolute temperature

Basic relationships are:

1. $$\frac{Q_1}{Q_2} = \frac{N_1 S_1^3}{N_2 S_2^3}$$

2. $$\frac{P_{s1}}{P_{s2}} = \frac{N_1^2 S_1^2}{N_2^2 S_2^2}$$

3. $$\frac{HP_1}{HP_2} = \frac{N_1^3 S_1^5}{N_2^3 S_2^5}$$

If the system is run at high altitudes or high temperatures, so that the density of the air or gas stream changes from atmospheric, the following relations can be used to make density corrections.

1. $\frac{D_1}{D_2} = \frac{T_2}{T_1} = \frac{P_{a_1}}{P_{a_2}}$

2. At constant $P_s$ and S

$$\frac{N_1}{N_2} = \frac{Q_1}{Q_2} = \frac{HP_1}{HP_2} = \frac{D_2^{\frac{1}{2}}}{D_1^{\frac{1}{2}}}$$

3. At constant Q, N and S

$$\frac{P_{s_1}}{P_{s_2}} = \frac{HP_1}{HP_2} = \frac{D_1}{D_2}$$

4. At constant mass of air and constant S

$$\frac{P_{s_1}}{P_{s_2}} = \frac{Q_1}{Q_2} = \frac{N_1}{N_2} = \frac{D_2}{D_1}$$

$$\frac{HP_1}{HP_2} = \frac{D_2^2}{D_1^2}$$

## Calculating Fan Efficiency

1. Mechanical (Total) Efficiency

$$M.E. = \frac{\text{Horsepower output}}{\text{Horsepower input}} \times 100$$

Horsepower output = Air Horsepower (AHP)

$$AHP = \frac{Q(ft^3/min \times {}^{P}t(\text{in. } H_2O) \times 62.4\ (lb/ft^3\ H_2O)}{12\ (in/ft) \times 33{,}000\ (\frac{ft\text{-}lb}{min}/HP)}$$

$$AHP = \frac{QP_t}{6350}$$ where Q = cfm

$P_t$ = total fan pressure in inches of water

Horsepower input = Brake Horsepower (BHP)

$$\therefore M.E. = \frac{AHP}{BHP} \times 100 = \frac{QP_t}{6350} \times \frac{100}{BHP}$$

2. Static Efficiency (S.E.) is always less than mechanical efficiency because velocity pressure cannot entirely be converted to static pressure.

$$S.E. = \frac{QP_s}{6350} \times \frac{100}{BHP} \qquad (P_s = \text{fan static pressure in inches of water})$$

## Fan Standards and Definitions

Most fan manufacturers belong to the "Air Moving and Conditioning Association, Inc." formerly known as the "National Association of Fan Manufacturers, Inc." (NAFM). This group has established fan standards, definitions, and test codes, which are summarized in their Bulletin No. 110,(21) available on request. Fan manufacturers using the NAFM test code indicate it in their catalogs. Therefore, when comparing fans of different manufacturers there is a valid basis for comparison.

The NAFM pressure definitions are:

1. The total pressure ($P_t$) of a fan is the rise of pressure from fan inlet to fan outlet as measured by two impact tubes, one in the fan inlet duct and one in the fan discharge duct, corrected for friction to the fan inlet and outlet respectively.

2. The velocity pressure ($P_v$) of a fan is the pressure corresponding to the average velocity determination from the volume of air flow at the fan outlet area.

3. The static pressure ($P_s$) of the fan is the total pressure ($P_t$) diminished by the fan velocity pressure ($P_v$).

Confusion often arises between the fan static pressure ($P_s$) defined above, and the static pressure measured at the inlet to a fan (ps). These differ numerically by one velocity pressure (at the fan inlet). Fan static pressure ($P_s$) is equivalent to the resistance to air flow in the piping system it serves. The static pressure as measured at the fan inlet (ps) is the resistance loss plus one velocity pressure. This measured static pressure is often called "suction pressure" by fan manufacturers.

## Fan Selection

### General Considerations

In order to select the proper fan for a given duty the following information is required:

a. Air volume to be moved.

b. Static pressure to be overcome.

c. Cleanliness of the air. A radial-bladed centrifugal fan will be needed if the air stream contains a significant concentration of particulates.

d. Direct or belt drive. Belt drive can be changed for changes in air volume handled. Direct drive is inflexible but occupies less space.

e. Space limitations. If fan must be located in a given small space, forward curved or axial flow fan may have to be used.

f. Noise level. May or may not be an important consideration, depending on fan location. Noise level is a function of tip speed.

g. Operating temperature. Select fan built for operating temperatures that will be encountered.

h. Cost, including first cost, power cost, and maintenance cost.

How to Use Fan Catalogs in Selecting a Fan

Fan catalogs generally contain information on fan selection, wheels and arrangements available, dimensions and weights, special modifications and accessories that can be ordered, and a number of fan multi-rating tables. On such a table will be found output ratings and horsepower requirements for one size of fan at 70° F and 1 atmosphere, covering the entire range of static pressures and speeds within the limits of the fan construction. For each type of fan in the catalog there will be a series of such rating tables, each for a different size of fan. To express the size of a fan, the manufacturers use some linear fan dimension such as inlet outside diameter or wheel diameter. Complimentary copies of fan catalogs may be obtained from most fan manufacturers upon request. The point of highest mechanical efficiency for each static pressure on such a chart is usually underlined or printed in bold face. The ideal fan size for a given task is the one where the point of highest efficiency coincides with the desired flow and static pressure. If this point falls between two fan sizes in a series, the smaller one is usually selected, since it will cost less. In most cases interpolation between the points in the tables will be necessary. A static pressure interpolation should be made first, followed by the volume interpolation. Arithmetic interpolations are usually satisfactory. For conditions other than 70° F and 1 atmosphere, density corrections must be made.

Estimating Capacities of Used Fans

Occasionally, when an available fan is lacking sufficient name plate data, or when the rating tables for such a fan are missing, it is possible to estimate the fan capacity with reasonable accuracy.

Hemeon describes a method in his book, Plant and Process Ventilation,(16) of plotting an approximate fan characteristic curve for centrifugal fans from two simple pressure measurements. These measurements are the static pressure at no flow and the velocity pressure at wide open flow. The curve obtained by this method is a fair approximation of the actual characteristic, at least on the right-hand side of the pressure peak. Since the right-hand side is the proper operating region, this should be sufficient.

Hemeon also gives approximations for predicting fan capacity by inspection; criteria for estimating propeller fan capacity; and criteria for estimating capacity of fans in duct exhaust systems.

Fan Location and Maintenance

When air movers are used in conjunction with air cleaning devices, good practice dictates that they be located downstream from the cleaner. This will minimize erosion, abrasion, and the building up of sticky deposits on the fan blades. The pressure drop through the air cleaner will be the same whether up or downstream of the fan. The fan should be located so that

the inlet and exhaust ducts go through the minimum number of bends, and it should have an anti-vibration mounting. Another important consideration in the location of a fan is accessibility for inspection and servicing.

When a fan is installed, a scheduled inspection program should be instituted. Items to be checked regularly should include lubrication, bearings, belt drives, and fan wheel. The wheel should be inspected for abrasion accumulation, electrical grounding and rotation in the proper direction. Since some air will still be moved despite improper direction, such operation can go undetected for some time.

## Other Air Moving Devices

### Injectors (also called ejectors)

Injectors operate by inducing a large volume of secondary air flow by means of a small volume of high velocity air flow, or jet. The air jet expands in a duct and entrains and mixes with surrounding air. A cylindrical injector has its jet in a simple round duct and has a maximum efficiency of about 5%, while a Venturi injector has its jet discharging into a Venturi throat, and can develop efficiencies as high as 12%.

Although injectors are highly inefficient as compared to fans, and may require 5 to 10 times the power for the same duty, there are many instances where an injector would be the air mover of choice. The advantages of injectors include:

1. Lower first cost

2. Simplicity of installation

3. They can exhaust an air stream containing sticky or highly corrosive material which would rapidly foul a fan. The injector would also become fouled, but at a much slower rate, and it would be much easier to clean.

In instances where the power cost would be negligible, or where the demand is intermittent, or where an existing compressed air source is available, the lower first cost of an injector would overbalance its higher operating cost and it would be a good choice.

In Plant and Process Ventilation,[16] Hemeon discusses injectors and injector design. In Industrial Health Engineering,[7] Brandt has a good discussion on injectors, while a more complete discussion may be found in Design of Injectors for Low Pressure Air Flow,[20] by McElroy. Perry's Chemical Engineers' Handbook[24] has a discussion of steam jet ejectors.

### Blowers and Centrifugal Compressors

For handling large volumes of air or gases at pressures from about 0.5 p.s.i. to 40 p.s.i., blowers and compressors are usually used. In this pressure range, the compressibility of the fluid must be taken into consideration. Since these higher pressure air movers are rarely used in ventilation work, no discussion will be attempted here. For a general discussion, see Perry's Chemical Engineers' Handbook[24] and for a more complete discussion, see the symposium, Handling Compressible Fluids[9] Chemical Engineering, June 1956.

# AIR CLEANING

## Introduction

Air cleaning is the operation of removing contaminants from an air or gas stream. This job of removal can be simple and straight-forward, or complicated and difficult, depending on the number, nature and concentration of the contaminants that must be removed, and the degree of removal required. Some air cleaning mechanisms are effective on particular kinds of contaminants and less effective, or ineffective, on others. The selection of air cleaning equipment to remove a given contaminant, or group of contaminants, must be based on an evaluation of the components of the air stream to be cleaned, and on the efficiency of the various kinds of air cleaning equipment for these components (see Fig. 1).

## Definitions

A definition of terms at this point will help clarify the discussion that follows:

Gas is a substance which is in the gaseous phase at ordinary temperatures and pressures. Gases diffuse.

Vapor is the gaseous form of a substance which is normally in the solid or liquid state at ordinary temperatures and pressures. Vapors diffuse.

Aerosol is a general term referring to a suspension of a foreign material in air. This material may be in the form of dust, fume, smoke, mist, or fog.

Dust is formed in processes of disintegration, e.g., grinding, drilling, blasting, and crushing of organic and inorganic materials. The size range of a dust usually ranges from the submicron sizes to particles visible to the naked eye. No chemical change takes place in the production of dust. Dusts do not tend to flocculate except under electrostatic forces; they do not diffuse in air but settle under the influence of gravity.

Fume results from condensation from the gaseous state, generally after volatilization from molten metals, and often accompanied by chemical reaction such as oxidation. The particle size is usually more uniform and much smaller than that of a dust. Fumes flocculate and sometimes coalesce.

Smoke is the result of combustion of organic matter. Particle size is generally below 1 micron and relatively uniform.

Mist or fog is a liquid particulate. It may be generated by atomization of a liquid or condensation of vapor on air-borne nuclei.

Prepared by: Morton Lippmann - 57

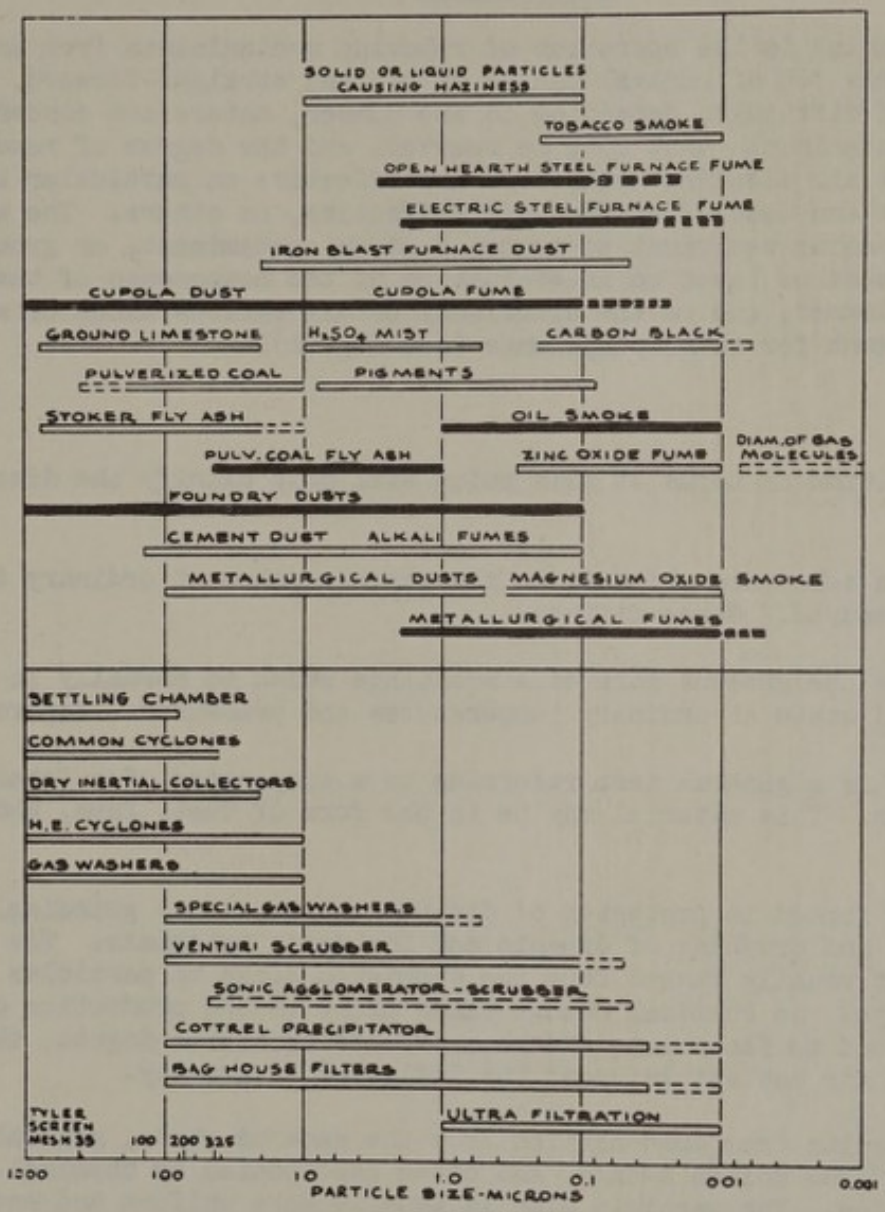


Fig. 1 - Graphical representation of particle size ranges of dust and fumes from typical industrial operations, with approximate operating ranges of control equipment.

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## Air Cleaning Equipment

Each type of air cleaning device has its advantages and limitations. The problem is to select the proper device, or combination of devices, which will do the job required with reliability and reasonable cost. When no one device is effective at reasonable cost, over the entire particle size range of a particulate aerosol, it is common procedure to use two or more types of air cleaners in series. This will be brought out in the discussion of the equipment.

Air cleaning equipment falls into two broad categories - wet collectors and dry collectors. The relative advantages and disadvantages of the two are summarized as follows:

### Dry Collectors

#### Advantages

1. A dry product is obtained.
2. Corrosion is minimized.
3. Waste products occupy a smaller volume.
4. Efficiency on particles 0.05 $\mu$ and larger can be high.

#### Disadvantages

1. Hygroscopics cake out on walls.
2. Secondary ventilation may be necessary when collector is emptied.
3. Replacement and repair may constitute an exposure.
4. High temperature requires refractory material.
5. Corrosive materials require special alloys.
6. Only gases and vapors which can be absorbed on solids can be collected.

### Wet Collectors

#### Advantages

1. Gases and particulate matter may be collected simultaneously.
2. Recovery of soluble material is possible.
3. High temperature gases are both cooled and washed.
4. Corrosive gases and mists can be handled.

#### Disadvantages

1. Recovered product may be contaminated.
2. Wet disposal is necessary.
3. An insoluble product will require wet filtration.
4. Particles smaller than 1 $\mu$ are not readily caught.
5. Corrosion problem may be great.
6. May have liquid entrainment.
7. Freezing in cold weather is possible.

## Collection of Particulate Matter

### Particulate Conditioning

The task of cleaning an air stream containing particulates is made materially easier if the particles can be conditioned before collection. Flocculation, deposition on droplets, and electrical charging are the usual means of particle conditioning. The first two methods simplify collection by making big particles out of little ones, since large particles are usually easier to capture. Electrical charging of particles makes them more subject to capture on the collecting surfaces of an electrostatic precipitator, or on a filter surface.

Small particles in Brownian motion collide inelastically, forming chainlike aggregates, or flocs. Flocculation is especially vigorous for heterogeneous aerosols of very small particles at high concentrations. Agitation and high temperatures result in increased rate of collision of particles, causing more rapid flocculation. Ultrasonic waves can also produce agglomeration of particles, and several industrial applications have been made of this process.(12,28)

Deposition on droplets can be performed in spray washers, cyclone scrubbers, and similar equipment. The droplets containing the particles are easier to capture than the particles alone. The same effect can be obtained by condensing vapor on the particles in a saturated atmosphere. These mechanisms work best with hydrophilic materials.

A charge can be imparted to particulates in several ways; one is to generate the aerosol by an explosion or atomization and another is to disperse the particulates with an air blast. Corona discharge or atomic radiations will also charge an aerosol. Charged particles are more readily captured in an electrostatic precipitator or on a filter surface than they would be if uncharged.

Conditioning sometimes is a separate step, but may take place at the same time as collection. A two-stage electrostatic precipitator is an example of a device where conditioning and collection are performed in separate stages. A cyclone scrubber is another example of a device where conditioning and collection are performed simultaneously.

For a more complete discussion of the mechanisms of particle conditioning, see the Handbook on Air Cleaning(15) and Air Pollution Handbook.(19)

Particulate Collection

Six mechanisms of separation are included in this discussion. These are: gravity, centrifugal and inertial forces, diffusion, electrical forces, thermal forces, and straining. In general, one or two of these mechanisms are the most effective under a given set of conditions.

Gravity. This is important only with larger particles. For particles less than 10 $\mu$, it can be neglected under almost all conditions. In most cases, it is unimportant for particles less than 50 $\mu$ because small particles settle so slowly that upward convection currents keep them suspended.

Centrifugal and inertial forces. When the path of a gas stream is changed, the particles, due to their relatively great inertia, tend to remain on their original course and are caught on a collecting plate. Capture is favored by high gas velocity and sharp directional changes. The more radical the change, the smaller the particles that can be caught. Below 1 $\mu$, this mechanism becomes less important.

Diffusion. This takes over where inertial forces leave off, i.e., its effectiveness is greatest for particles 0.5 $\mu$ and smaller. Diffusion depends on the existence of a concentration gradient and is favored by low gas velocities and high concentration gradients. Particles diffuse from the gas stream to the surface where the concentration is lower.

Electrical forces. These are made use of in air cleaning in commercial electrical precipitators and also in filtration, although the latter application is not fully understood. Filtration is usually improved when the aerosol has a static charge, but the effect of an application of such charges to an aerosol is highly unpredictable. In commercial electrical precipitation, the effect of the application of a given charge on a given aerosol is predictable. For particles smaller than 1 μ, charge is proportional to diameter and efficiency is therefore independent of size. For large particles, charge is proportional to the square of the diameter, hence, large particles should be removed more readily than small ones. In practice, this is offset by a greater reentrainment tendency.

Thermal precipitation. This principle is used in air sampling and in the precipitation of carbon black; but, it has found no application in industrial air cleaning.

Straining. This is a mechanism of particulate removal in filtration. It occurs only when the particle size is greater than the void size; in the microscopic range, this mechanism is relatively insignificant.

For more complete discussions on the mechanisms of separation, see the Handbook on Air Cleaning(15) and the Air Pollution Handbook.(19)

## Air Cleaning Equipment for Particulates

The choice of a particular type of air cleaner or collecting equipment depends upon several factors. Among these are: (1) the amount of material to be collected or gas loading, (2) nature of the material to be collected, and (3) the efficiency of collection required. It must be borne in mind that particulates less than 10 microns in size are of hygienic significance and are more difficult to collect than particles larger than 10 microns, which create a nuisance problem only.

### Types of Equipment

In the discussion to follow various types are briefly described including their uses, advantages and disadvantages.

Settling chambers. Sometimes referred to as chip traps, these are enclosed spaces where the force of gravity is exploited to achieve separation.

Retention is a function of the dimensions of the chamber and the flow rate:

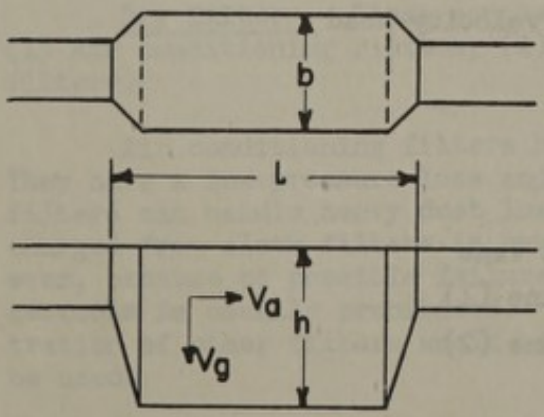



Figure 1. Settling chamber

$$\text{Retention} = \frac{(h.b.l.)}{Q} = \frac{h}{V_g}\frac{V_g}{V_a} = \frac{h}{l}$$

$$V_a = \frac{Q}{hb}$$

Such a device is efficient only on particles larger than about 50 microns. If a series of horizontal trays is used instead of one large chamber, the device becomes a Howard Dust Chamber. This may remove particles as small as 10 microns. If a baffle is placed in the entrance of a settling chamber, it makes the chamber an inertial collector.

Dry centrifugal and inertial separators. These are effective down to 5 or 10 microns, vary widely in capacity, and are readily adaptable for many purposes. Cyclone collectors are centrifugal separators which are used extensively as primary separators for dusts from rock crushing, ore handling, woodworking and sand conditioning in foundries. Their collection efficiency falls off rapidly for particles smaller than 10 microns.

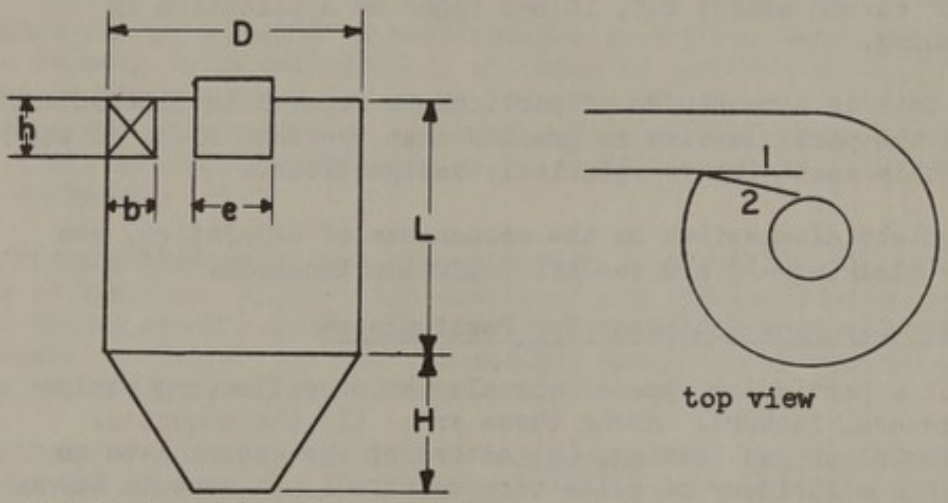



Figure 2. Cyclone collector

The gas enters the separator tangentially and spirals along the walls and out at the top. The particles strike the wall and fall through the core into a collecting hopper. Standard cyclone proportions for design are given in Chemical Engineers' Handbook[24] and in Industrial Health Engineering.[7]

Cyclones handle gas volumes ranging from 30 to 25,000 cfm. Dust loadings should be over 10 grains per cubic foot for efficient operation.

Pressure drop through a cyclone is usually kept below 4 inches of water gauge. It can be computed from the equation of First[14] (see Fig. 2):

$$F = K \frac{bh}{e^2}$$ Where F = inlet velocity head

$$K = \frac{\frac{12}{k}}{\left(\frac{L}{D}\right)^{1/3} \left(\frac{H}{D}\right)^{1/3}}$$

where: k = 1/2 with no inlet vane
k = 1 with inlet vane (1)
k = 2 with inlet vane (2)

Baffle chambers are an example of an inertial collector, and are effective for particles above 20 microns. They are often used as pre-cleaners before bag filters.

To separate smaller particles efficiently, a small cyclone is better than a large one. A commercial collector uses a bank of six to nine-inch cyclones in parallel. Instead of a tangential inlet, annular vanes impart the whirling motion. The increased efficiency is at the expense of greater cost and increased pressure drop.

A dry dynamic precipitator is also illustrated in Industrial Ventilation.[2] This is a mechanical separator in which the centrifugal motion is caused by a rotating impeller. Other dry commercial collectors are illustrated in The Handbook on Air Cleaning,[15] Chemical Engineers' Handbook[24] and the Air Pollution Handbook.[19]

Wet collectors. These have many useful applications in particulate air cleaning. Capacities are large, and cleaning efficiency at both high and low dust loadings may be sufficient for primary cleaning. In many cases liquid disposal is easier than solid. It is hard to collect submicron particles by wet methods, since it is extremely difficult to wet small particles without large energy expenditures.

Air washers are chambers in which water is sprayed into the air stream. Eliminator plates remove the mist particules. Wet filters serve the same purpose as air washers, but with higher efficiency. They consist of banks of cells containing glass filaments. The cells are about 20 inches square, three to eight inches thick, and are set at an angle to the air flow. Water is sprayed onto the filter and is recirculated from the sump. Spray towers have spray nozzles at the top or along the walls, and baffle plates along the sides, which are wetted continually. A high velocity helps to capture particles on the baffles by impaction. Packed towers are also used as dust collectors.

Many commercial wet collectors utilizing centrifugal and inertial forces have been developed. Several of these collectors are illustrated in Industrial Ventilation[2] and other sources. The devices illustrated include centrifugal scrubbers, cyclone scrubbers, Venturi scrubbers, water-jet scrubbers, hydroclone scrubbers, bubble-cap scrubbers, and Roto-clone scrubbers.

Dry filters. These can be divided into three classifications: (1) air conditioning filters, (2) cloth filters, and (3) high efficiency filters.

Air conditioning filters handle large air volumes with low dust loads. They have a low pressure loss and usually low collection efficiency. Cloth filters can handle heavy dust loadings at high efficiency and the air discharged from cloth filters is usually clean enough for recirculation. However, because of possible failure, the recirculation of air bearing toxic aerosols is usually prohibited. For highly toxic aerosols where the penetration of other filters would be too great, high efficiency filters must be used.

Filters take advantage of inertial forces, diffusion, electrical charging, and straining. The effect of inertial forces and diffusion can be predicted with some accuracy from theoretical considerations. Electrical forces on the particles and/or the filter surface play an important part in filtration, but their effect in a given case is difficult to predict. Straining plays an insignificant role for dusts of hygienic significance.

The two most common types of commercial cloth filters are the "bag" type and the "screen" or envelope" type. These are illustrated in Chemical Engineers' Handbook[24] and the Air Pollution Handbook.[19] The difference between the two types is in the manner of support for the filter. The sleeves or bags are usually contained in an enclosure known for bag filters as a "bag house." The bag house has an inlet for the dusty air, a manifold to distribute the air to the bags, an outlet, and a hopper to catch the collected dust. Most also contain some kind of shaking device to dislodge the collected dust when the critical resistance is reached.

One type of bag filter, the "Reverse Jet" type, performs at constant resistance. A traversing ring moves continuously up and down the bag blowing off the dust with a high velocity air jet.

The efficiency of a cloth filter increases with dust load since most of the filtering is done by the accumulated dust.

High efficiency filters must be used for air cleaning tasks where efficiencies by count greater than 99.5% are required. One type is a cellulose-asbestos paper. The asbestos does most of the filtering and the cellulose gives the necessary support. Crushed flint, sand, and coke in deep beds make high efficiency filters. Glass and resin wool fibers are also used for this purpose. High efficiency filters are expensive and take up large amounts of space. For additional information see the Handbook on Air Cleaning.[15]

Electric precipitators. Industrial uses include the collection of fly ash, metallurgical fume, acid mists, cement dust, and other materials. Two types are in common use - the Cottrell and two-stage types.

Cottrell precipitators find use in metallurgical and chemical industries to collect oxide fumes and acid mists. Utilities use them to collect fly ash. Two-stage precipitators are used as air conditioning precipitators to remove oil and tobacco smoke, and in collecting dusts and smokes from foundries; they have separate ionizing and collecting fields, and are used for light dust loadings. Both types run on rectified a.c.: Cottrell at 25,000 volts and over, and the two-stage with 12,000 volts ionizing stage and 6,000 volts collecting. Cottrells have visible coronas and present a shock hazard.

Comparison of equipment types. Table 1 is a comparison of important dust collector characteristics.

Table 1. - Comparison of some important dust collector characteristics

| Type | Higher Efficiency Range on Particles Greater than Mean size in Microns | Pressure Loss Inches | $H_2O$ Gal per 1,000 CFM | Space | Sensitivity to CFM Change: Pressure | Sensitivity to CFM Change: Efficiency | Humid Air Influence | Max. Temp., F. Standard Construction |
|---|---|---|---|---|---|---|---|---|
| Electrostatic | 0.25 | 1/2 | - | Large | Negligible | Yes | Improves Efficiency | 500 |
| Fabric: | | | | | | | | |
| Conventional | 0.4 | 3-6 | - | Large | As cfm | Negligible | May Make Recondi-tioning difficult | 180 |
| Reverse Jet | 0.25 | 3-8 | - | Moderate | | | | 200 |
| Wet: | | | | | | | | |
| Packed Tower | 1-5 | 1 1/2-3 1/2 | 5-10 | Large | As cfm | Yes | None | Unlimited |
| Wet Centrifugal | 1-5 | 2 1/2-6 | 3-5 | Moderate | As $(cfm)^2$ | Yes | | |
| Wet Dynamic | 1-2 | Note 1 | 1/2 to 1 | Small | Note 1 | No | | |
| Orifice Types | 1-5 | 2 1/2-6 | 10-40 | Small | As cfm or less | Varies with design | | |
| Higher Efficiency: | | | | | | | | |
| Nozzle | 0.5-5 | 2-4 | 5-10 | Moderate | As $(cfm)^2$ | Slightly to Moderately | None | Note 2 |
| Venturi | 0.5-2 | 12-20 | - | Small | | | | Unlimited |
| Dry Centrifugal: | | | | | | | | |
| Low Pressure Cyclone | 20-40 | 3/4-1 1/2 | - | Large | As $(cfm)^2$ | Yes | May cause condensation and plugging | 750 |
| High Eff. Centrifugal | 10-30 | 3-6 | - | Moderate | As $(cfm)^2$ | Yes | | 750 |
| Dry Dynamic | 10-20 | Note 1 | - | Small | Note 1 | No | | 750 |
| Louver | 15-60 | 1-3 | - | Small | As $(cfm)^2$ | Moderately | | 750 |

Note 1: A function of the mechanical efficiency of these combined exhausters and dust collectors.

Note 2: Precooling of high temperature gases will be necessary to prevent rapid evaporation of fine droplets.

(Reproduced with permission from Industrial Ventilation.)

## Collection of Gases and Vapors

Removal of gases and vapors from gas streams may be accomplished by absorption in liquids or solids, by adsorption on solids, and for vapors, by condensation. In absorption the gas or vapor becomes distributed in the collecting liquid or solid. Adsorption is a surface phenomena. Both processes are diffusional operations. A theoretical discussion of these processes is beyond the scope of this outline, and belongs in the study of Chemical Engineering. Excellent treatment of the subject may be found in Chemical Engineers' Handbook(24) and Sherwood and Pigford's Absorption and Extraction.(26)

### Equipment

Absorption. Equipment for absorption include absorption towers, such as bubble-cap plate columns, sieve plate columns, packed towers, spray towers, etc.; washers, such as the wet-cell washers described for dust collection; and special devices such as Venturi injectors. Selection of equipment for a particular task is dependent on the operational variables such as vapor pressures, solubilities, and efficiency of removal required. Successful methods for the removal of sulfur dioxide, hydrogen sulfide, fluorides, and nitrogen oxides are given in the Air Pollution Handbook.(19)

Adsorption. For the adsorption of gases and vapors, solid adsorbents have been developed which have an affinity for certain substances. Various clays, chars, activated carbons, gels, aluminas, and silicates have been used. They are usually granular in form and are made up in beds or columns through which the gas passes. Adsorption may be practically complete even with very low vapor content, so the procedure is readily adaptable to solvent recovery operations. Adsorbents can collect from 8% to 25% of their weight in vapors. The vapors can be removed from the beds and the beds reused. For a detailed discussion of adsorption, see the Air Pollution Handbook,(19) Bulkeley,(8) and Chemical Engineers' Handbook.(24)

## Summary

Each air cleaning problem is unique. From an evaluation of the nature of the aerosol and the performance required, the appropriate equipment can be selected.

The job does not end with the selection of the equipment. Its performance must be checked after installation, and proper modifications made if needed. Furthermore, a good maintenance program is necessary to insure continued good service.

# PRINCIPLES OF DESIGN OF SYSTEMS

## Introduction

All the principles of ventilation and air flow discussed in previous lectures are applied in the design of ventilation systems. Even for persons who will never have any occasion to design a system, a knowledge of the principles involved can be quite useful. The governmental industrial hygienist, for instance, is charged with responsibility for system design in only a few States. Nevertheless, a knowledge of the principles of design is very useful in the evaluation of existing systems, and in suggestions for systems to correct unsatisfactory situations.

The data required in the design of exhaust systems are quite detailed. Tables, charts and formulas giving data necessary for design as well as further discussion of points mentioned here may be found in Industrial Ventilation.(2) This lecture is therefore designed for us in conjunction with Industrial Ventilation.

## Pressure Losses

Once having determined the volume of air and type of hood necessary for an operation, it is necessary to conduct this air to a fan and the outside by suitable ductwork. Since the volume of air which a fan will exhaust depends upon the static pressure, it is necessary to determine the pressure losses throughout the system. We shall, therefore, consider the types of pressure losses in a system.

### Friction Losses

In the section, "Principles of Ventilation Airflow," it was shown that the friction loss due to flow of fluid in a pipe is given by the Fanning-D'Arcy equation:

$$h = f \frac{L v^2}{2g\ D}$$

where h = loss in feet of air
L = length of pipe, in feet
v = velocity of fluid, in feet per second
g = gravitational constant
D = duct diameter, in feet
f = the dimensionless friction loss coefficient

All quantities in the equation are well-defined with the exception of f, which varies according to the Reynolds number and surface roughness. The principle variation in the coefficient is caused by surface roughness. The friction coefficient applicable to industrial exhaust systems varies from 0.01 to 0.04. The friction chart commonly used for determining pipe resistance applies to straight galvanized duct with about 40 joints per 100 feet, and is based on dry air at 760 mm and 70° F. Hemeon gives a table of multipliers from the data of Madison and Elliot for other than this type of pipe as shown below. Friction losses found by the chart should be multiplied by this factor.

Prepared by: Howard E. Ayer - 57

Multipliers for Other than Standard Duct*

| Type of duct | Factor |
|---|---|
| Medium smooth; e.g., steel pipe without joints or exceptionally well constructed galvanized iron duct system, with smooth joints. | 0.9 |
| Medium rough; e.g., average concrete surface | 1.5 |
| Very rough; e.g., average riveted steel | 2.0 |

*After Hemeon(16)

Loss in Expansions

The friction chart gives the loss in runs of straight pipe. Other losses are produced by changes in direction, velocity, or cross-section. One of these is the loss in abrupt expansion. It is easily seen that if air is discharged from a duct into still air, the entire velocity pressure $\left(\frac{V}{4000}\right)^2$ is lost. If there is a sudden expansion in area, as when a small duct is joined to a large duct with a flange, then the "Carnot-Borda" loss, as derived in the section on "Principles of Ventilation Airflow" is:

$$h'' = \left(\frac{V_1 - V_2}{4000}\right)^2$$

Where the enlargement is more gradual, not all this will be lost. For the recommended taper of 5 inches length for each inch of diameter increase, only about 2/10 of Carnot-Borda loss will occur, or

$$h'' = 0.2\left(\frac{V_1 - V_2}{4000}\right)^2$$

Another method of calculation, in terms of "static regain" is given in Industrial Ventilation.(2)

Losses at Entrances and Contractions

The contraction of an air stream with its resultant increase in velocity is accompanied by very little pressure loss. However, where there is a sudden contraction, a vena contracta will be formed, and the re-expansion of the air stream will be accompanied by the same sort of loss as the abrupt expansion of the air stream. In a plain duct end, for example, the pressure loss is caused principally by the vena contracta formation. The loss can be reduced by flanging, and almost completely eliminated by a properly tapered or bell-mouth entrance which suppresses the vena contracta.

A sharp-edged orifice has a pressure loss which is caused by the loss in abrupt enlargement from the vena contracta area to the duct area. It is often given as 1.78 times the orifice velocity pressure, but Hemeon states that a more correct value is 2.8 times orifice v.p. The actual pressure loss

will depend considerably on the construction of the orifice and the ratio of orifice to duct area. A slot is similar to an orifice, but since contraction is only on two sides, the loss will be 1.3 x slot v.p.

A properly tapered contraction within a duct line may be assumed to have negligible pressure drop.

Elbow Losses

The losses in elbows have been determined empirically. Losses are proportional to the angle of bend up to about 120°, but an 180° elbow has only 1.65 times the pressure drop of a 90° elbow. Much of the increased velocity pressure resulting from helical flow patterns downstream is regained within a few diameters, so if an elbow discharges directly to the atmosphere the loss will be 2 to 2-1/2 times as great as when a few diameters of ductwork are added to allow static regain to take place. The shorter the radius of the elbow, the higher the loss. Meter elbows should never be used, and a radius of at least 2 diameters should be used whenever space permits.

Junction Losses

The losses in the main where a branch enters may be a substantial fraction of the velocity pressure in the main. Alden[1] suggests values of 0.1 to 0.2 main velocity pressures for 45° tees, depending on the relative volumes. He believes the loss for angles other than 45° to be proportional to (1 - cos a), so that the loss would be half as much for a 30° tee, and 3.5 times as much for a 90° tee. Losses also occur in the branch duct, the magnitude depending on the angle and relative flow. For the method of design used in Industrial Ventilation[2] all losses are assumed to occur in the branch.

## Duct Velocities

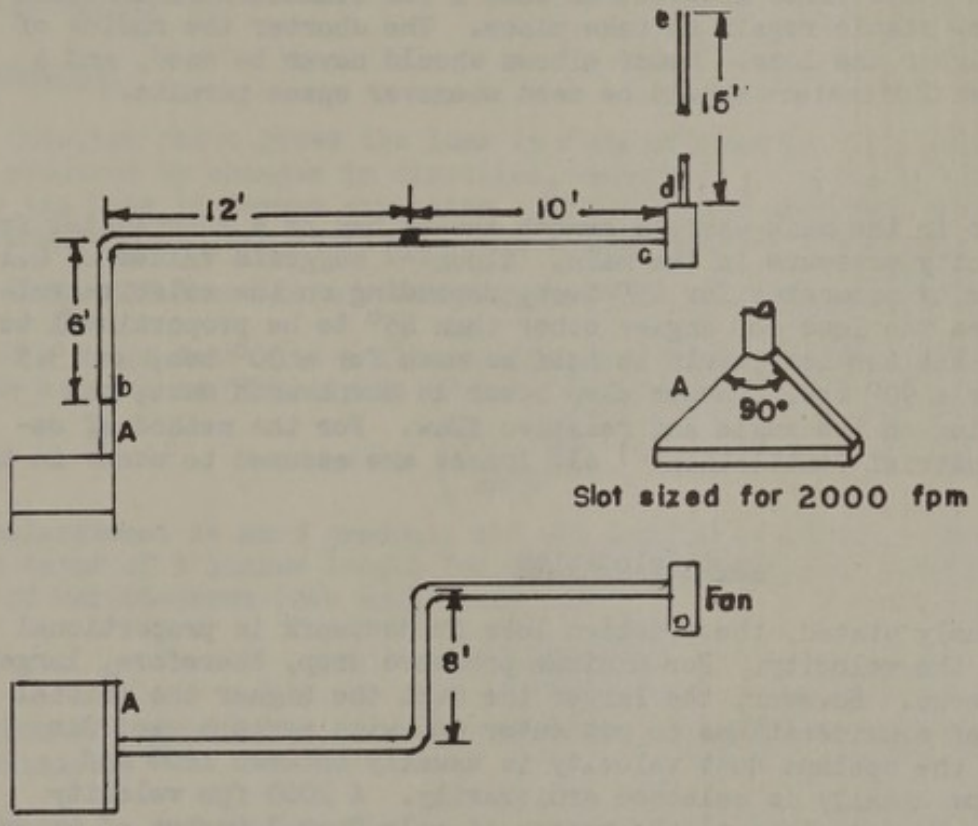
As previously stated, the friction loss in ductwork is proportional to the square of the velocity. For minimum pressure drop, therefore, larger duct is advantageous. However, the larger the duct the higher the initial cost. Where other considerations do not enter in, with systems used for gases or vapors, the optimum duct velocity is usually between 1800 and 2500 fpm; 2000 fpm usually is selected arbitrarily. A 2000 fpm velocity will also give a pressure loss in the system of only 2 or 3 inches of water, so that one of the economical, backward-curved centrifugal fans may be used.

Where a system is handling dust, however, velocities as low as 2000 fpm would lead to clogging of the duct by solids. "Conveying velocities" for dust systems range from 3500 to 4500 feet per minute. As Hemeon[16] points out, if a trap is placed just behind the hood to collect the larger particles by settling or impingement, 3000 feet per minute is probably sufficient. In a system which handles all the dust, 4000 - 4500 fpm is often specified for branches, with 3500 - 4000 fpm for mains. Such high velocities are naturally associated with high pressure drops and also with very serious abrasion problems, particularly at elbows. Although such velocities are also quite noisy, noise is usually not a consideration in the industrial plant.

Velocities lower than 2000 fpm may be desirable for systems where a propeller fan might be employed. To reduce the pressure drop to the range of a propeller fan, velocities in the neighborhood of 1000 fpm may be used. The propeller fan would normally be used with short lengths of ductwork only.

## System Design

Any length of ductwork will have some of the losses previously mentioned. The total resistance of the section of duct with its associated hood and fittings may be obtained by summing up the various individual resistances. Industrial Ventilation[2] gives the basic principles of duct design. The following example will illustrate the procedure:




A. Hood for plating tank (see plate in Industrial Ventilation)

Air volume required = 1300 cfm

A velocity of about 2000 fpm is desired, so a pipe area of $\frac{1300}{2000}$ or 0.65 $ft^2$ is required. An 11-inch diameter duct has an area of 0.66 sq. ft., and so this will be used. Actual duct velocity then equals 1970 fpm. From page 3 of this lecture and page 6-12 of Industrial Ventilation,[2] the hood entry loss will be 1.3(0.25) + 0.25 (0.25) or 0.39 inches of water. The total length of duct is 51 ft. From the chart in Industrial Ventilation,[2] the equivalent length of a 2.0 D, 90° elbow in 11" duct is 16 ft., so the total equivalent length of duct is:

$$51 + (3 \times 16) = 51 + 48 = 99 \text{ ft.}$$

From the pipe friction chart, we find that the resistance of 11" duct at 2000 fpm is 0.5 inches per hundred feet. The total resistance thus is 0.89 inches of water. This does not include the velocity pressure, which would be an additional 0.25 inches of water. If static pressure measurements are made at various points:

At b: s.p. = entry losses - velocity pressure = - 0.39 - 0.25 = -0.64"

At c: s.p. = entry loss - v.p. - loss in 36 ft. of duct and 3 elbows (equivalent length 48 ft.)
= 0.39 - 0.25 - 0.5(84) = -1.06"

At d: s.p. = loss in 15 ft. of duct = 0.5 x 0.15 = +0.08"

At e: s.p. = atmospheric = 0.00"

Multibranch Systems

When two branches of a system come together, or when a branch joins a main, the upstream pressure drop must be the same for both. An analogy may be drawn to an electrical system, where the voltage between the junction and ground must be the same for two lines which are joined. This means that if a branch designed for an air flow of 200 cfm at a pressure drop of 1.5 inches of water is joined to a main where the air flow is 2000 cfm with an upstream resistance of 0.8 inches of water, the air flow in the branch will be less than 200 cfm, since its pressure loss can be only 0.8 inches of water. Because the static pressure loss is proportional to the velocity squared, the branch flow will be reduced by the factor $\frac{0.8}{1.5}$ or

$Q_{(branch)} = 200\sqrt{\frac{0.8}{1.5}} = 146$ cfm. In order to obtain 200 cfm in the branch, the branch must be redesigned for a pressure loss of only 0.8 inches of water at 200 cfm, or the upstream resistance of the main must be increased to 1.5 inches of water. The main resistance may be increased in three ways, as follows:

(1) The upstream main flow could be increased in proportion, or

$Q_{main} = 2000\sqrt{\frac{1.5}{0.8}} = 2750$ cfm;

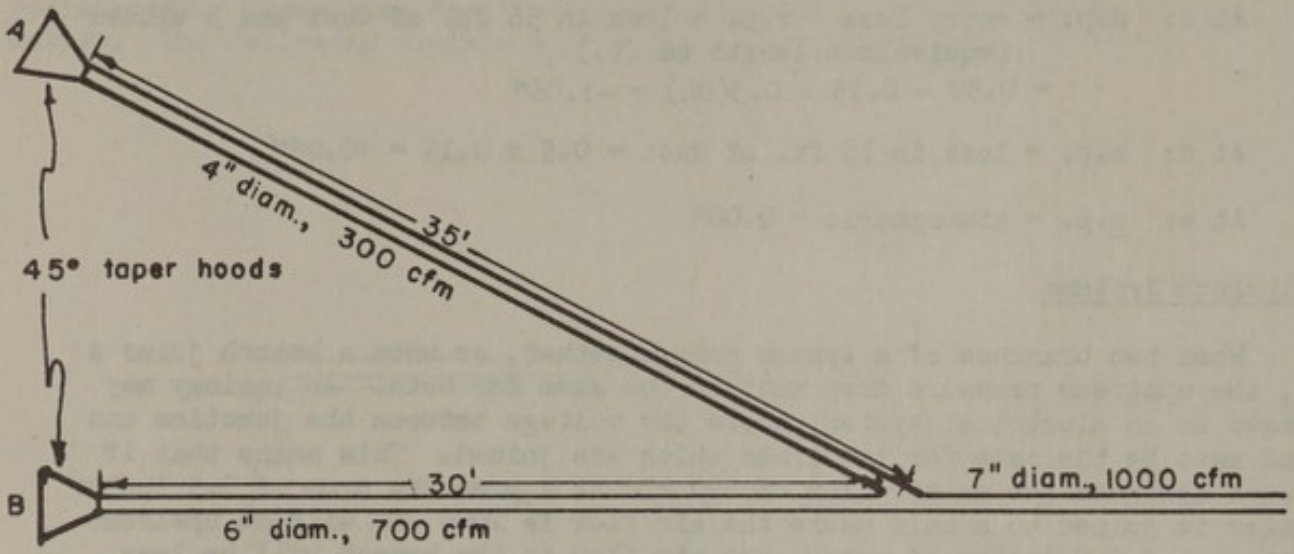
(2) A damper could be placed in the main, increasing the resistance to 1.5 inches while maintaining the flow at 2000 cfm; or

(3) The main and its upstream branches could be redesigned with smaller piping to increase its resistance to 1.5 inches.

Actual design of industrial exhaust systems is usually done in one of two ways. Either the system is designed to furnish the desired flow at the maximum static pressure and all balancing is done with blast gates as in (2) above, or the system is designed to balance without blast gates by using a combination of methods (1) and (3) above. Advantages and disadvantages of

the two methods are given in Industrial Ventilation.(2) An example of the balancing method follows:

Given the following system with the indicated flows desired, what changes in pipe sizes would be necessary to make the two branches balance, assuming a minimum conveying velocity of 3500 fpm.




| | $h_e$ | v.p. | hood static pressure | p/100 ft | friction p | static pressure at junction |
|---|---|---|---|---|---|---|
| Branch B | 0.15 vp | 0.76" | 0.76+0.15(0.76) = 0.87" | 3.4 | 1.02 | 1.89 |
| Branch A | 0.15 vp | 0.76 | 0.87 | 5.3 | 1.85 | 2.72 |

If the pipe size in Branch B is decreased to 5 1/2 inches, then:

| | | | | | | |
|---|---|---|---|---|---|---|
| Branch B | 0.15 vp | 1.10 | 1.25 | 5.2 | 1.56 | 2.81 |

The static pressures are now within 5%, and for all practical purposes may be considered the same.

A useful rule in designing balanced systems is to consider static pressures within 5% to be in perfect agreement (since this is closer than the static pressures can usually be estimated); to recalculate the flow of the branch with the lower resistance when the static pressures are within 20%; and to change pipe sizes in the branch of lower resistance when the static pressures differ by more than 20%. The method of doing this is shown in an example in Industrial Ventilation.(2)

## Power Requirements

As pointed out in the lecture on fans, the power required is proportional to the product of the volume and the static pressure. Consequently, some judgment should be used in the arbitrary criteria for flow and static pressure adjustment. Where a branch has a high resistance and low flow relative to the main, it may be much more desirable to increase the pipe size and total flow in the branch, since the flow increase would be relatively insignificant, while if the higher static pressure were used it would be multiplied by the total flow.

It is undesirable to use the same exhaust system for dusts and vapors, because a dust exhaust system requires higher velocities with higher pressure drops and heavier construction. This would lead to excessive power costs for a combined system. A system designed for corrosive vapors, on the other hand might use plastic or lined pipe, which could be damaged by the abrasion of dust in the system.

## Fan Selection

As pointed out in the lecture on fans, two rating methods are in use. The more common method of rating a fan specifies the fan static pressure, since the velocity pressure is furnished at "free delivery." Fan static pressure is defined as follows:

$$\text{Fan SP} = \text{TP} - \text{VP Fan Outlet}$$

or, in terms of velocity pressure and static pressure only:

$$\text{Fan SP} = SP_{inlet} + SP_{outlet} - VP_{inlet}$$

Occasionally a series of fans may be rated in terms of total pressure. If this is the case, the total pressure at the fan is used for the selection.

## Accuracy of Calculations

It is very important to calculate exhaust systems carefully, taking into account all pressure drops since the proper functioning depends on good design. However, it is recognized that many of the calculations are based on average results from basic data which varied considerably. Duct friction loss, for example, varies with the construction of the duct, and hood entry losses must often be estimated. For this reason, Hemeon insists that it is necessary to carry static pressures only to the nearest one-tenth inch, rather than the nearest hundredth, as is commonly done. Also, if the static pressure estimation is off by 10%, the volume will be off only 5%, so that although the estimates are admittedly not exact, they will ordinarily be close enough for satisfactory results. A system which is not designed carefully or is merely built without a formal design will seldom give satisfactory results.

# AIRFLOW MEASUREMENT

## Introduction

To determine the effectiveness of environmental control by any ventilation system, an evaluation of the air movement must be made. In some instances, a qualitative determination, such as observing a vapor trail or the smoke from a smoke tube, may be sufficient to evaluate the effectiveness of control. In most cases, however, quantitative measurements are needed for proper evaluation. Airflow measuring devices can be divided into two groups; one measures the velocity of the air stream, and the other the quantity of airflow. Velocity meters include Pitot tubes and anemometers of various types. Quantity meters include Venturi meters, orifice meters, and rotameters. These two types of measurements can be converted, one to the other, by the simple dimensionless formula:

$$Q = AV \qquad (1)$$

where: $Q$ = Volumetric rate of flow

$A$ = the cross-sectional area through which the flow takes place

$V$ = average linear velocity in cross-section A

This formula is applicable where the cross-sectional area A can be defined.

Ventilation airflow is almost always turbulent. The only instruments capable of measuring airflow rates at velocities which may be in the streamline range are some thermal anemometers and the ionization anemometer.

All flowmeters, with the possible exceptions of well-designed Pitot tubes, should be calibrated against a primary or reliable secondary standard before use, and periodically checked for calibration thereafter.

## VELOCITY MEASURING DEVICES

### Instruments Utilizing Velocity Pressure

Pitot tube. The Pitot tube is the standard instrument for measuring the velocity of air. A standard Pitot, carefully made, will need no calibration. It consists of an impact tube whose opening faces axially into the flow, and a concentric static pressure tube with 8 holes spaced equally around it in a plane which is 8 diameters from the impact opening. The difference between the static and impact pressures is the velocity pressure. Bernoulli's theorem applied to a Pitot tube in an air stream simplifies to the dimensionless formula

$$V = \sqrt{2gP_v} \qquad (2)$$

where: $V$ = linear velocity

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$g$ = gravitational constant

$P_v$ = head of flowing fluid or velocity pressure

Expressing V in linear feet per min., and $P_v$ in inches of water ($h_v$), formula (2) becomes

$$V = 1097\sqrt{\frac{h_v}{\rho}}$$

where: $\rho$ = density of air or gas in $lb/ft^3$

If the Pitot tube is to be used with air at standard conditions (70° F. and 1 atm.), formula (2) reduces to:

$$V = 4005\sqrt{h_v} \qquad (3)$$

where: $V$ = velocity in ft/min

$h_v$ = velocity head in inches of $H_2O$

The Pitot tube measures velocity in the location in which it is placed. One measurement of velocity is usually insufficient to define the rate of airflow through a duct. If a circular duct is straight for at least 10 diameters upstream from the point of measurement, a single center-line Pitot reading may give a fairly accurate estimate of the flow rate. It has been found that under such conditions, the average velocity is about 90% of the center-line velocity, or:

$$Q = 0.9\,AV_c$$

where: $V_c$ = center-line velocity

Where accuracy is required, however, a Pitot traverse should be made. In a rectangular duct, the cross-section should be divided into at least nine equal areas, and a velocity measurement made at the center of each. The average of the velocities measured is the average velocity in the duct. For a traverse of a round duct, the cross-section should be divided into an even number of concentric rings of equal area, and measurements made at the odd numbered boundary lines. A ten point traverse is usually made, which requires ten points in one plane, and ten points in a plane perpendicular to it. The velocities are averaged to get the average velocity in the duct. Tables of distances from pipe wall for 6 and 10 point traverses in round duct of various sizes are given on page 9-4 of Industrial Ventilation[2]. If the velocity pressure is read on a water manometer, or a pressure sensing device calibrated in inches of water, a table of conversions of velocity pressure to velocity, such as Fig. 6-16 of Industrial Ventilation,[2] may be useful.

The Pitot tube is used largely for measuring air velocity in ducts, or at high velocity supply and exhaust openings. Its accuracy in the low velocity range is limited by the sensitivity of the pressure sensing device used with it. At 3000 lfm, the velocity pressure is 0.56" $H_2O$; at 2000 lfm, 0.25" $H_2O$; at 1500 lfm 0.14" $H_2O$; and at 1000 lfm 0.062" $H_2O$. Therefore, a vertical water manometer should not be used below about 2000 lfm. An accurately balanced inclined manometer may be used down to about 600 lfm. Various accurate

low pressure measuring devices may be used with a Pitot tube, but their use is usually limited to the laboratory where they can be carefully set up and balanced.

Pitot tubes can be used under a wide range of conditions; there are no moving parts to go out of order, and, if made of stainless steel, they are resistant to corrosion and high temperatures. Standard Pitot tubes cannot be used in dusty atmospheres because they will become plugged but a special type for use in dusty atmospheres is shown on page 9-7 of Industrial Ventilation[2] This type of Pitot should always be calibrated before use.

Further discussion of the Pitot tube is contained in many standard references[7,22,24,29].

Deflecting vane anemometer (Velometer). This type of instrument is widely used for airflow and static pressure measurement by industrial hygienists and heating and air conditioning engineers. On page 9-8 of Industrial Ventilation[2] ways in which Velometers are used are illustrated. Commercial Velometers are available with one pressure and one, two, three or four velocity ranges, with a variety of jets for each range. They have the advantage of being direct-reading instruments with ranges as low as 50 lfm or as high as 24,000 lfm, although readings below 100 lfm are not very accurate.

The Velometer operates by the pressure of an air stream against a spring loaded swinging vane. It is fairly rugged, and if calibrated regularly, the readings are sufficiently accurate for most field work. If the temperature of the air stream is outside the range 70° F ± 30° or the altitude is greater than 1000 feet, density corrections should be made. Dust or corrosive gases should not be permitted to enter the instrument. Velometers can be ordered with dust filters which will take out light dust loadings. If the instrument is calibrated with a dust filter, it must always be used with it.

In order to measure airflow or total pressure in a duct with a Velometer, a hole in the duct large enough to accommodate the appropriate probe is required. Such a hole is larger than that required for a Pitot tube and may be difficult to provide. When measuring static pressure, a minimum 3/8" hole is needed, and it must be free from burrs in order to make accurate measurements.

Rotating vane anemometer. This instrument consists of a propeller connected through gears to a dial which counts rotations. The dial reads in linear feet, which when divided by the time interval of the measurement, gives linear feet per unit time. The instrument is available with 3, 4, or 6" wheels.

The rotating vane anemometer is useful for measuring air velocities in the range of 200 to 2000 lfm. It is largely self-averaging for fluctuating flows and for traversing large openings. Readings are little affected by deviations in alignment with the direction of airflow up to about 20°. The instrument is delicate and should be calibrated frequently. It cannot be used in dusty or corrosive air. Since this type of instrument is rarely used in temperatures or pressures far from standard, pressure-temperature corrections are seldom needed.

For a more complete discussion of the rotating vane anemometer see The Measurement of Air Flow[22], Chapter 7.

Other pressure actuated anemometers. One type of commercially available anemometer is a direct reading instrument designed primarily for measuring air velocity at discharge grilles in the range of 30 - 2,700 lfm. It has a rotating impeller whose motion is opposed by a spring. When the velocity pressure on the impeller and spring tension balance, the velocity can be read from the dial indicator. Another type of anemometer available is the cup anemometer used in meteorological work. It consists of four hemispherical cups mounted on light arms from a hub which turns freely on a vertical axis. This type of instrument has little application to ventilation work.

## Thermal Anemometers

Heated thermometer anemometer. The heated thermometer anemometer is based on the principle that the rate of heat loss from an object at elevated temperature is a function of air movement over the object. The instrument consists of a pair of thermometers, one with an electric resistance coil about the bulb to which a known voltage may be applied. The other is an ordinary thermometer. In use, the two thermometers are placed in the air stream to be measured, and the applied voltage adjusted to give a difference of 15° to 30° in the two thermometer readings. The thermometers are then allowed to come to equilibrium. Velocity of the air stream can be determined from a calibration chart using the temperature difference between the two thermometers, or by application of the temperature difference in the following equation:

$$\frac{\text{net milliwatts}}{\Delta t} = K + K' \sqrt{V}$$

where: net milliwatts = fixed value for each thermometer at each voltage. A table is supplied with each instrument by the manufacturer.

$\Delta t$ = temperature difference between thermometers

K and K′ = constants; values for each supplied by manufacturer with each instrument

V = velocity of air stream

(Instead of constants for the above equations, some manufacturers supply a calibration curve.)

The instrument indicates the cooling power rather than the velocity of an air stream. It is affected by radiant heat and convection. In the absence of radiant heat sources, it is very accurate as a velocity measuring device when carefully used. The instrument is non-directional and has a range of 15 to 500 lfm. It is fragile and its use is time-consuming.

Yaglou[30] gives a more complete discussion of the heated thermometer anemometer.

Heated thermocouple anemometer. This instrument works in the same manner as the heated thermometer anemometer, but uses thermocouples in place of thermometers. The advantages gained are in size and ruggedness of the sensing elements, in response time, and in the fact that it can be readily calibrated as

a direct reading instrument. Several commercial instruments of this type are available. One commercial instrument has a range of 10 to 2000 lfm. Another has a range of 10 - 5500 lfm plus a static pressure scale of 0 - 4" $H_2O$. A third type has a range of 0 - 8000 lfm, a static pressure range of 0 - 10" $H_2O$, and also a Fahrenheit temperature scale. The calibration of this type of instrument should be checked regularly. Most of these instruments use dry batteries which must be replaced periodically.

Hot-wire anemometer. A hot-wire anemometer consists of a fine, electrically heated wire which is placed in the air stream to be measured. The cooling effect on the wire will depend on the velocity of the air stream. As the wire cools, its resistance changes, and this change can be measured with a bridge circuit. If calibrated properly, this type of instrument can be used to measure a wide range of air velocities. Pannell[23] gives a detailed discussion of this type of instrument.

Kata thermometer. The Kata thermometer is a special thermometer with a large bulb containing alcohol, and a stem with marks at 95 to 100° F. It is heated above 100° F and the time required for it to cool from 100 to 95° F is a measure of the non-directional air velocity in the room. It was designed for comfort ventilation measurement, and its surface to volume ratio is similar to that of the human body. The useful velocity range is 25 to 500 lfm. It has the disadvantages of being fragile and having large radiation and convection areas.

### Ionization Anemometer

The ionization anemometer consists of a duct section having polonium on one surface and an alpha ray counter on the opposite surface. As the air flow increases, fewer $\alpha$ particles reach the counter, and the instrument can be calibrated in terms of velocity. Extremely low velocities can be measured with this instrument. Lovelock and Wasilewska[17] gives further information about this instrument.

## Air Quantity Measurement

A common method of determining fluid flow rate in a pipe is to insert a restricted opening in the line, and measure the resulting pressure drop across it. Orifices, nozzles, and Venturi meters use this principle to measure flow rates. The same general formulas apply to them all.

### Orifice Meter

An orifice meter is simply a restriction in a pipe between two pressure taps. There are several types of orifice meters used, but the simplest and most common is the square-edged orifice, which is a very short cylindrical passage in a thin metal plate. If it is properly constructed, the orifice plate will be at right angles to the flow and the surface will be carefully smoothed to remove burrs and other irregularities. Since the square-edged orifice is easily constructed and the meter as a whole is simple and comparatively inexpensive, it is widely used as an accurate flow meter. Orifice meters are only rarely used as permanent flow meters in ventilation systems because of their high permanent pressure loss. They are more typically used in the ventilation laboratory for calibration purposes. Permanent head

loss will vary from 40 to 90 percent of the static pressure drop across the orifice as the ratio of orifice diameter to pipe diameter varies from 0.8 to 0.3. Orifices have been intensively studied and their performance characteristics can be predicted if they are constructed to standard proportions. Detailed discussions of orifices and orifice equations are contained in standard references[7,22,24]. On pages 191-193 of Industrial Health Engineering[7], Brandt gives a simplified formula for calculating the flow rate through a thin-plate orifice with flange taps for air at standard conditions. In it, Q is a function of the orifice diameter, the pressure drop across the orifice, and a factor K which varies with the Reynolds number and the ratio of orifice to duct diameter. A table of K's is given in this discussion.

Critical Flow Orifice

For a given set of upstream conditions, the discharge of a gas from a restricted opening will increase with a decrease in ratio of absolute pressures $P_2/P_1$, where $P_2$ is the upstream pressure, and $P_1$ the downstream, until the velocity through the opening reaches the velocity of sound. The value of $P_2/P_1$ at which the acoustic velocity is just attained is known as the critical pressure ratio. The pressure in the throat will not fall below the pressure at the critical point, even if a much lower downstream pressure exists. Therefore, when the pressure ratio is below the critical, the rate of flow through the restricted opening is dependent only on the upstream pressure.

It can be shown[24,29], that for air flowing through rounded orifices, nozzles and Venturis, when $P_2 < 0.53\ P_1$, and $S_1/S_2 > 25$, the flow rate of air, w, is determined by:

$$w = 0.533\ \frac{C_v S_2 P_1}{\sqrt{T_1}} \text{ in lb/sec}$$

where: $C_v$ = coefficient of velocity (normally 1)

$S_2$ = orifice area in square inches

$P_1$ = upstream absolute pressure in lb/sq.in.

$T_1$ = upstream temperature in °R

Critical flow orifices are widely used in industrial hygiene instruments such as the midget impinger pump and squeeze bulb indicators.

Flowmeters

The term flowmeter is often used to designate any restricted opening through which the rate of flow has been determined by calibration. One can be made by inserting a restriction in a pipe section, or for smaller flows by using a piece of glass capillary tubing as the restriction. A typical laboratory flowmeter consists of a U-tube manometer with a capillary connected across its legs.

Venturi Meters

A Venturi meter consists of a 25° contraction to a throat, and a 7° reexpansion to the original size. This differs from the orifice meter where the

changes in cross section are sudden. The great advantage of the Venturi meter over the standard orifice is that the permanent reduction in static pressure is small, because the velocity head in the throat is largely reconverted to static pressure by the gradual re-enlargement. A well designed and constructed Venturi will have a permanent static pressure loss of only 0.1 to 0.2 of the Venturi reading, as compared to 0.4 to 0.9 with a square-edged orifice. Standard references[22,24,29] discuss Venturi meters and Venturi equations. In Industrial Health Engineering[7], Brandt gives a simplified formula for flow through a Venturi which is applicable to air at 70° F and 1 atm. when the ratio of throat diameter to duct diameter is between 1/4 and 1/2 as follows:

$$Q = 21.2\ r^2\ d^2\ \sqrt{h}$$

where: Q = flow in cfm

r = ratio of throat to duct diameter

d = duct diameter in inches

h = pressure drop in inches of water

Rotameters

A rotameter consists of a vertical transparent tube increasing in area from bottom to top, in which an upward flowing fluid stream supports a float at a level determined by the flow rate. The underlying principle of operation is the same as that of an orifice or Venturi meter. The float is the restriction, but instead of a variable pressure drop and a constant area of opening, there is a constant head (the weight of the float) and a variable area.

Rotameters are available for measuring a wide range of airflow rates, from as little as 10 cc/min of air, to as much as 350 cfm. Like other quantity meters, they are rarely used to measure ventilation airflow, but find wide use in laboratory calibration and precise metering.

Thermal Meters

A thermal meter measures mass air or gas flow rate with negligible pressure loss. It consists of a heating element in a duct section between two points at which the temperature of the air or gas stream is measured. The temperature difference between the two points is dependent on the mass rate of flow and the heat input.

Mixture Metering

The principle of mixture metering is similar to that of thermal metering. Instead of adding heat and measuring temperature difference, a contaminant is added and its increase in concentration is measured; or clean air is added and reduction in concentration is measured. This method is useful for metering corrosive gas streams. The measuring device may react to some physical property such as thermal conductivity, or vapor pressure.

Other Quantity Meters

Other quantity meters include wet gas meters, dry gas meters, and spirometers which are discussed under Airflow Calibration of Air Sampling Equipment.

## Indirect Measurement of Airflow

Air volume flowing into a standard exhaust hood can be determined fairly accurately from a measurement of throat suction. The volume of air entering a branch can be calculated from the formula:

$$Q = 1097\, C_e A \sqrt{\frac{h_s}{\rho}}$$

or for air at 70° F and 1 atmosphere

$$Q = 4005\, C_e A \sqrt{h_s}$$

where: $Q$ = airflow rate in cfm

$C_e$ = Coefficient of entry

$A$ = area of duct in $ft^2$

$h_s$ = suction pressure in inches of water

$\rho$ = density of air in $lb/ft^3$

The suction pressure should be measured one to three pipe diameters downstream from the throat of the exhaust inlet in a straight section. Coefficients of entry for standard exhaust hood types are tabulated in various references[2,7,16]. For hoods not listed, an estimate may be made by breaking down the hood into component simple shapes, and combining the coefficients of the simple shapes. This method is valid if the hood does not accumulate deposits, or become dented before the point of measurement, which would affect the magnitude of the entry loss. Very little expense is involved in making throat-suction measurements. It is a very useful method for periodic inspections since it will quickly show how the exhaust rate compares with previous values. Since the only equipment required is a piece of rubber tubing and a manometer or pressure gage, determinations can be quickly and easily made, even by non-technical inspectors.

For further discussion of flow measurement by throat suction see Industrial Health Engineering[7] and Plant and Process Ventilation[16].

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# Laboratory Exercises

Laboratory Exercise - CALIBRATION OF HI-VOLUME AIR SAMPLER.

Object

To calibrate the airflow indicator (Visi-Float gauge) of a Hi-Volume air sampler and prepare a curve of airflows versus gauge readings.

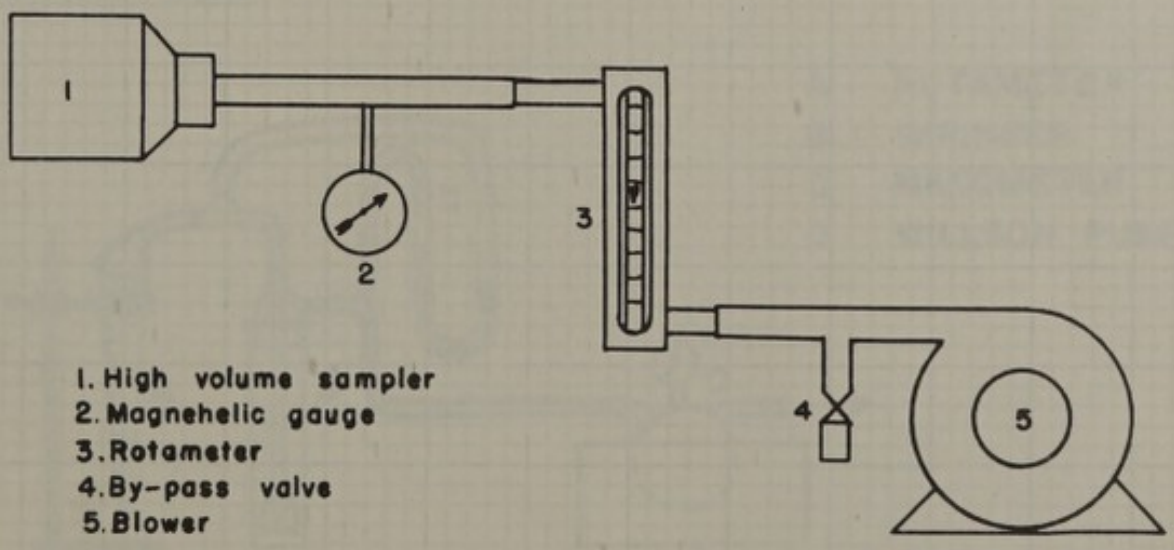
Theory

The rotameter is considered a secondary standard for airflow measurement. Like all secondary standards the rotameter must be recalibrated periodically since dust or corrosive gases may change the characteristics of the device and result in erroneous readings. The theory of the rotameter may be found in a chemical engineers handbook or standard text. Although pressure drop across the float of a rotameter is essentially constant, the pressure drop across the entire device, including elbows, etc., increases rapidly with increasing flow. To prevent the resistance of the rotameter from affecting readings of the Visi-Float gauge and to achieve the maximum flow through the Hi-Vol Sampler, the blower and valve is operated to provide atmospheric pressure downstream from the rotameter.

Procedure

Insert a clean pleated filter in the sampler. Turn on the blower and sampler and adjust the by-pass from the blower so that the Magnehelic gauge reads zero. Read the large rotameter and Visi-Float gauge. "Load" the filter by inserting small pieces of paper in the line. Reconnect sampler, readjust the blower by-pass and read the Visi-float and large rotameter. Repeat procedure to obtain three or more sets of readings and draw calibration curve.

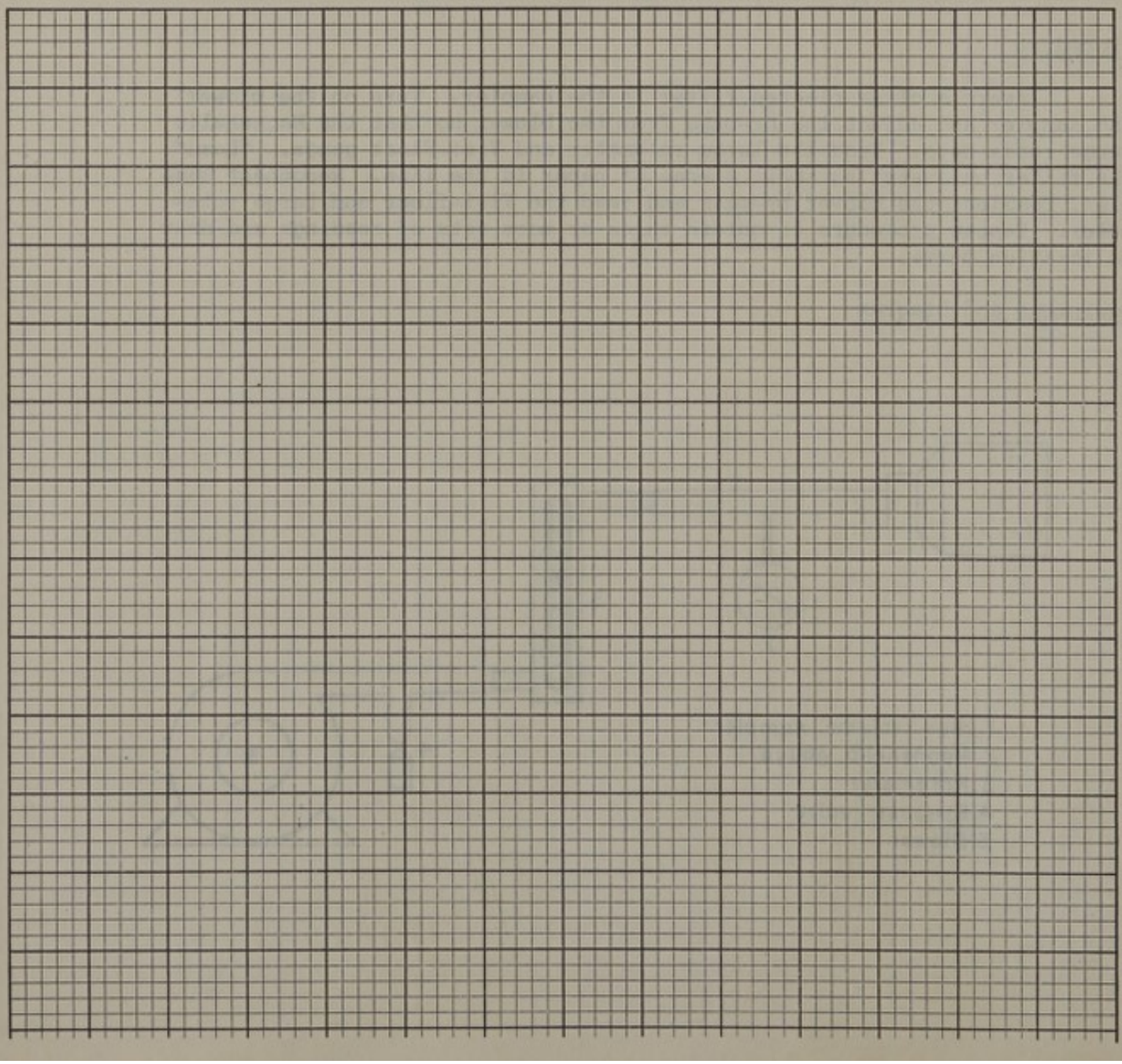
Equipment and Diagram




1. High volume sampler
2. Magnehelic gauge
3. Rotameter
4. By-pass valve
5. Blower

Data

| Large rotameter reading (cfm) | Visi-Float reading |
|---|---|
| | |
| | |
| | |
| | |
| | |
| | |
| | |



Laboratory Exercise - CALIBRATION OF GREENBURG-SMITH IMPINGER.

Object

To calibrate the flow rate through an all-glass Greenburg-Smith impinger.

Theory

In the standard procedure for using a Greenburg-Smith impinger, a suction pressure of 3" of mercury is applied to the exhaust side. One cubic foot per minute of air will be drawn into the impinger under these conditions, if the orifice is 2.3 mm in diameter. Since glass is not a precision construction material, it cannot be assumed that the orifice will be exactly 2.3 mm. Small variations in orifice size will cause significant departures in flow rate from 1 cfm. It is necessary to calibrate each impinger, and use the flow-rate found in subsequent work with that impinger.

The impinger should be calibrated against a primary or accurate secondary standard. In this experiment, a rotameter (secondary standard) will be used. It will be assumed, for purposes of this experiment, that the rotameter is in proper calibration.

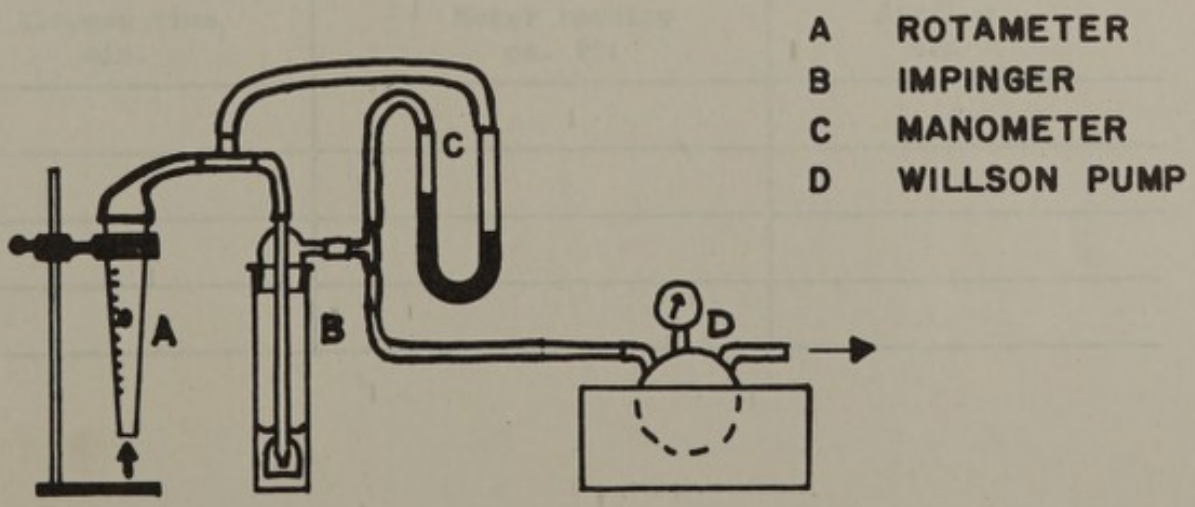
Procedure

Turn on the Willson pump and adjust the suction until the mercury manometer reads 3" Hg. Read the rotameter and mark the vacuum gauge of the Willson pump at this flow.

Translate the rotameter reading into cfm, make pressure and temperature corrections, if necessary.

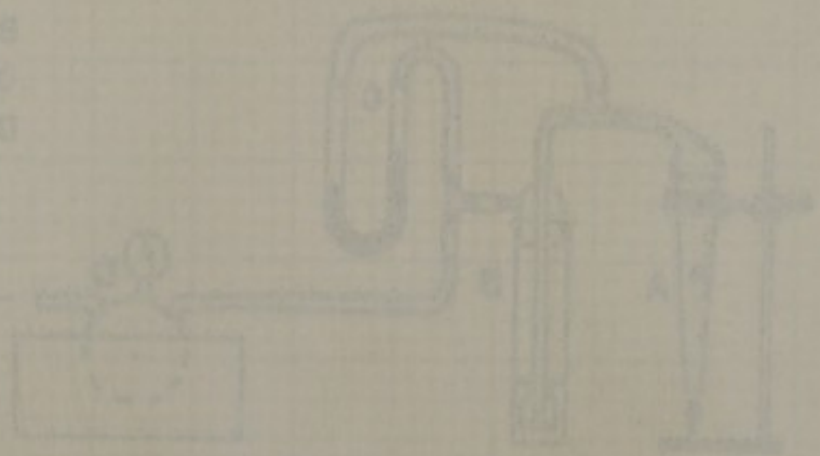
Record the correct flow for each impinger used under conditions outlined in the procedure.

Equipment and Diagram



Data

| Rotameter reading | Impinger number | Corrected airflow cfm |
|---|---|---|
| | | |
| | | |
| | | |
| | | |



Laboratory Exercise - CALIBRATION OF MIDGET IMPINGER

Object

To calibrate a midget impinger flask against the gauge reading of a midget impinger pump by means of a small wet test meter.

Theory

The midget impinger pump consists of a hand operated, four-piston pump assembly with a spring-loaded pressure control. It is so designed that, at a gauge reading of 12" $H_2O$, 0.1 cfm will be drawn through the impinger flask provided the orifice is the correct size. The manufacturer of impinger flasks usually provides an airflow calibration for each flask. This value should be checked and frequent recalibrations made as there may be some change in airflow with usage.

Procedure

Connect the impinger flask to the pump by means of a short section of rubber tubing. Add 10 ml distilled water to the flask. Crank pump at a speed which will maintain 12" $H_2O$ gauge reading. Note time required for 1 complete revolution of the wet test meter. Repeat 3 or 4 times. In practice, if a large number of impingers are to be calibrated, a small pump provided with a bleeder and vacuum gauge or manometer may be used to obtain the 12" $H_2O$ suction. Calculate the rate of airflow through the impinger at a gauge reading of 12" $H_2O$.

Equipment

1. Small wet test meter
2. Midget impinger pump
3. Midget impinger flask

Data

| Elapsed time min. | Meter reading cu. ft. | Airflow cfm |
|---|---|---|
| | | |
| | | |
| | | |
| | | |

Laboratory Exercise - CALIBRATION OF FIXED FLOW ELECTROSTATIC SAMPLER.

Object

To calibrate the rate of airflow of the MSA fixed flow electrostatic sampler.

Theory

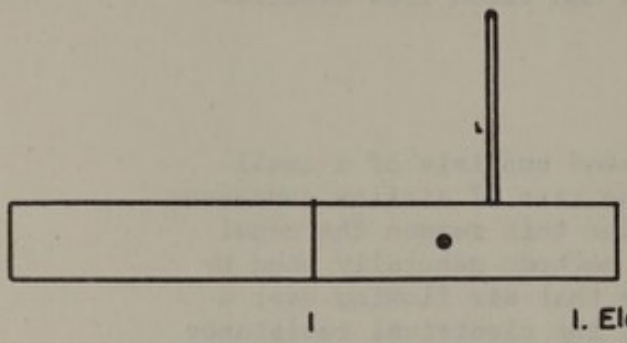
The source of suction in the sampling head consists of a small capacitor motor with a centrifugal blower. The rate of airflow decreases considerably with increase in resistance and for this reason the usual means of calibration cannot be employed. The methods generally used by most laboratories are based upon the principle that air flowing over a heated wire or heated thermometer bulb reduces the electrical resistance of the wire or temperature of the thermometer. The thermoanemometer is one of the older and very accurate methods of measuring airflows in the lower ranges and it employs the latter principle.

Procedure

In order to save time, a calibration curve of airflow versus thermoanemometer temperature differences has been previously prepared. This was done as follows: the Willson heated and unheated thermometers were inserted at right angles to each other about midway in a precipitator collecting tube (Fig. 1). One additional tube was joined to the first and the assembly supported in a wooden frame. The outlet side of the collecting tube was attached to the inlet of the wet test meter. A source of suction (old MSA precipitator or vacuum line) was attached to the outlet side of wet test meter (Fig. 2). The suction was adjusted to give an airflow of 3 cfm on the wet test meter and after a few minutes, the two thermometers were read. This procedure was repeated at 3.5 and 2.5 cfm and sufficient readings were taken at each of the flow rates until duplicate thermometer readings checked. A curve was drawn with airflows from wet test meter versus temperature differences in $^{o}F$ at 3 volts.

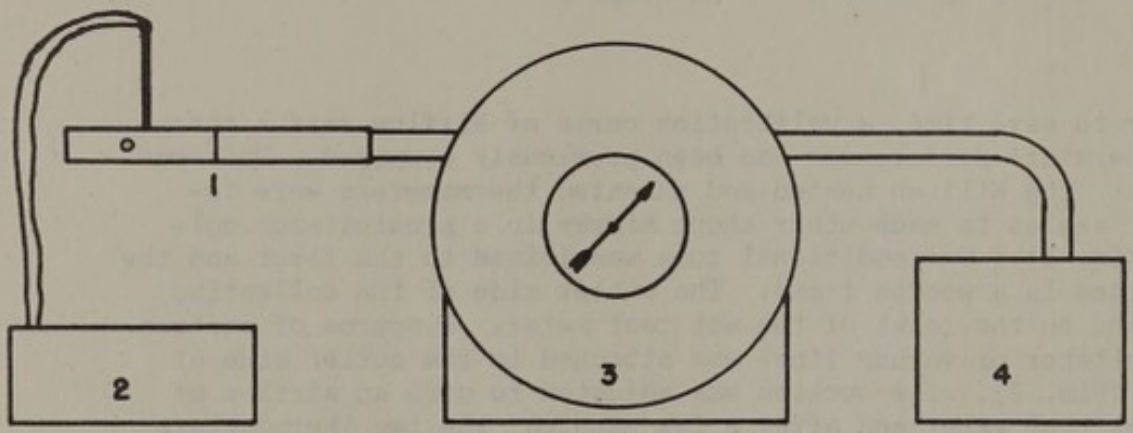
The procedure is as follows: Turn on the precipitator blower and read the two thermometers at one-minute intervals. Repeat this procedure until 3 successive temperature differentials are the same. Do not create too much air movement in vicinity of experiment as this will give erroneous results.

Equipment and Diagram



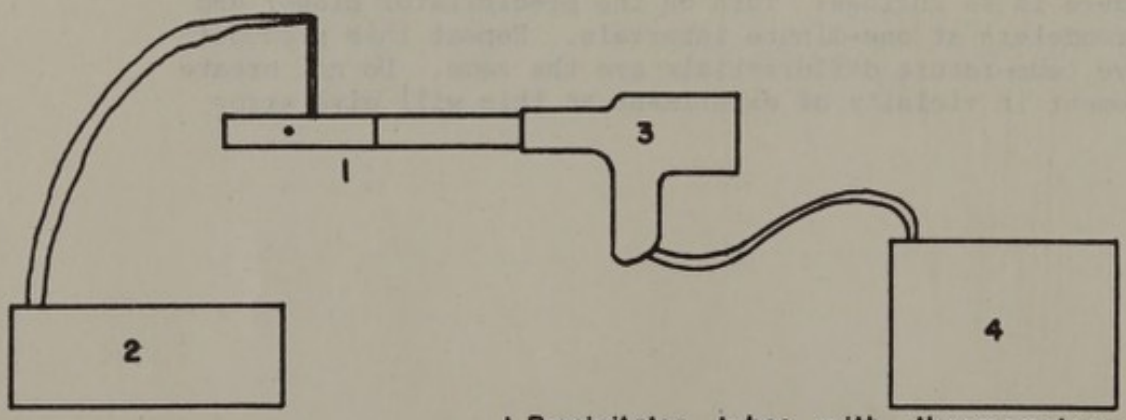

1. Electrostatic precipitator tubes
2. Standard thermometer
3. Heated thermometer

Figure 1




1. Precipitator tubes with thermometers
2. Thermoanemometer
3. Wet gas meter
4. Source of suction

Figure 2




1. Precipitator tubes with thermometers
2. Thermoanemometer
3. Precipitator sampling head
4. Power supply

Figure 3

Data

| Thermometer readings | | | Voltage on heated Thermometer | Airflow from curve cfm |
|---|---|---|---|---|
| heated | cold | T | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
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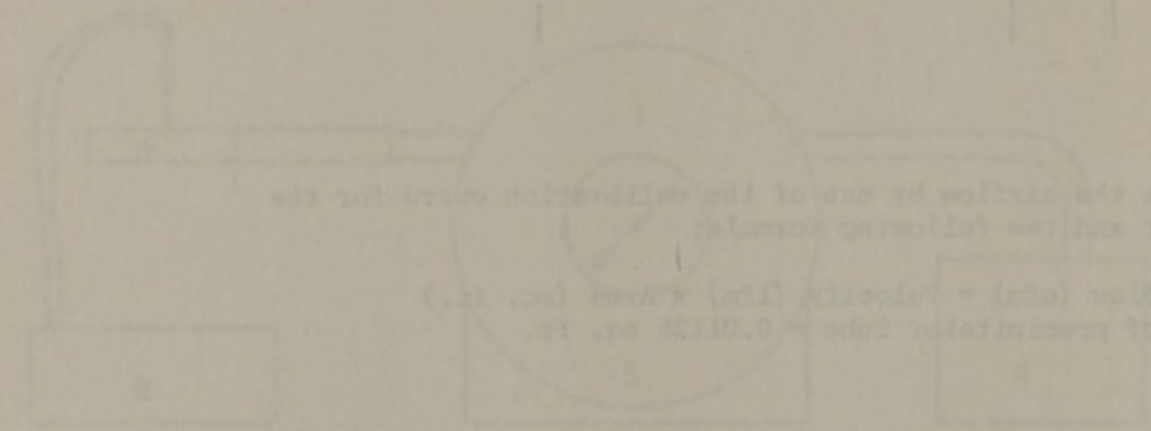
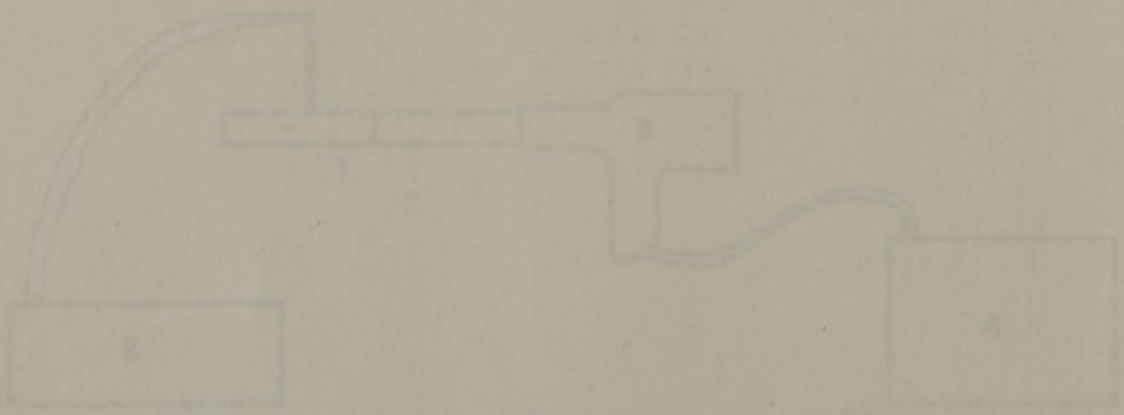
Calculations

Calculate the airflow by use of the calibration curve for the thermoanemometer and the following formula:

Rate of flow (cfm) = Velocity (lfm) x Area (sq. ft.)
Area of precipitator tube = 0.01126 sq. ft.





Laboratory Exercise - CALIBRATION OF VARIABLE FLOW ELECTROSTATIC SAMPLER. ALSO CALIBRATION OF GAST AIR SAMPLER.

## Object

To calibrate the rotameters on a variable flow electrostatic precipitator and also on a Gast air sampler by means of a large wet test meter.

## Theory

The wet test meter is considered a primary standard while the rotameter is a secondary standard. As pointed out in Experiment No. 1, the rotameter must be re-calibrated periodically. The wet test meter used in this experiment was calibrated against a spirometer at the Cincinnati Gas & Electric Company's meter repair shop.

## Procedure

1. Attach the inlet of the precipitator to the outlet or suction side of the wet test meter by means of a section of rubber tubing. "Gooch crucible" tubing has been found to be quite satisfactory for making this connection. Turn blower motor on and adjust rheostat until rotameter float is at lowest mark. After about 1 minute, note the time required for 1 complete revolution of wet test meter. Repeat by adjusting rheostat so rotameter float is at second mark and note time required for 2 complete revolutions. Record observations on data sheet. The wet test meter is operating against atmospheric pressure on the upstream side and therefore no correction factor is necessary. The resistances of both the wet test meter and the electrostatic precipitator are negligible.

2. Attach inlet of filter paper holder of Gast air sampler to outlet of wet test meter. Turn pump on and adjust flow so that rotameter reads 20 (center of float at mark). Note time for 1 complete revolution of wet test meter indicator. Repeat for rotameter settings of 15 and 10. Record observations on data sheet. As noted above no correction factor is necessary for the wet test meter readings. However, the rotameter in this case is located between the filter medium and the pump and is operated under a reduced pressure which is dependent on the resistance of the filter paper. For this reason the rotameter should be recalibrated for each filter media used (Whatman #41, Millipore AA, Millipore HA, etc.).

3. Prepare calibration curve of airflow in cfm. versus float position for precipitator.

4. Prepare a curve for Gast air sampler from data obtained in step 2.

## Equipment

1. Wet test meter, 3 cfm capacity
2. Electrostatic precipitator
3. Gast air sampler and filter papers

Data

| Elapsed time min. | Meter reading cu. ft. | Airflow cfm | Flowmeter reading |
|---|---|---|---|
| | | | |
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| | | | |
| | | | |
| | | | |
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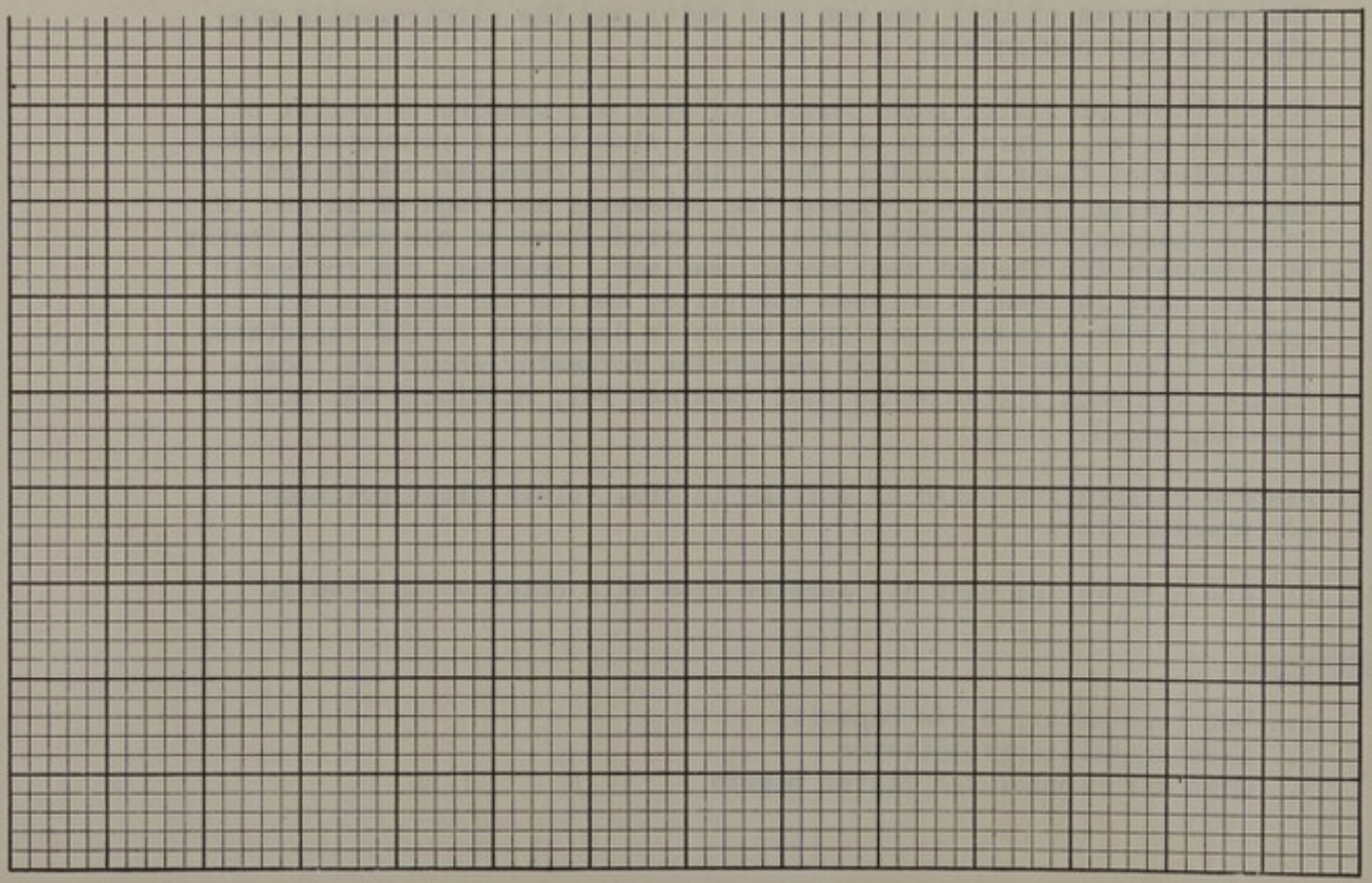
Calculations

$$\frac{\text{Meter reading in cu. ft.}}{\text{elapsed time in minutes}} = \text{Airflow in cu. ft./min.}$$



Laboratory Exercise - CALIBRATION OF FILTER PAPER SAMPLER.

Object

To prepare an airflow calibration curve for a filter paper sampling device using an orifice meter and a secondary standard.

Theory

Different types of filter media offer a range of resistance to airflow. The resistance to flow increases as the sample of dust or fume is deposited on the filter. It is therefore necessary to calibrate the sampling device to determine the rate of airflow under various conditions. Several metering devices are available for this purpose but in this experiment, an orifice meter will be calibrated against a rotameter. A theoretical discussion on the operation of an orifice meter is given by Jacobs[1] and on the rotameter by Perry[2]. Further theoretical considerations can be found in standard references on airflow measurement.

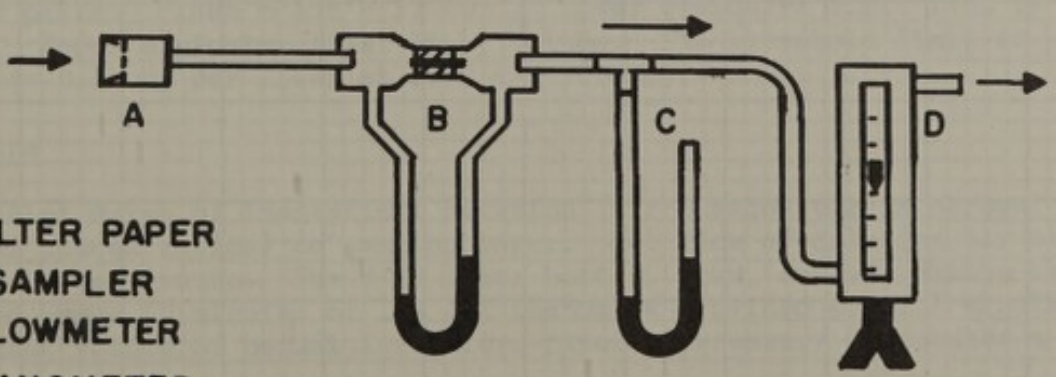
Procedure

Connect apparatus as shown in the diagram. Apply suction until rotameter reads about 1 cfm. Record readings of rotameter, U-tube monometer and orifice meter then repeat at four other rates of airflow between 0 and 1 cfm. Correct rotameter reading to standard conditions of pressure and temperature by means of gas law equation to give actual airflow as ordinates and orifice meter reading as abcissa. Use graph paper attached.

Equipment

1. Filter paper sample holder
2. Whatman #41 filter paper or other filter media
3. Orifice meter
4. Mercury U-tube monometer
5. Rotameter
6. Source of suction - Willson pump or vacuum line
7. Rubber tubing and metal T's

Diagram




A FILTER PAPER SAMPLER
B FLOWMETER
C MANOMETER
D ROTAMETER

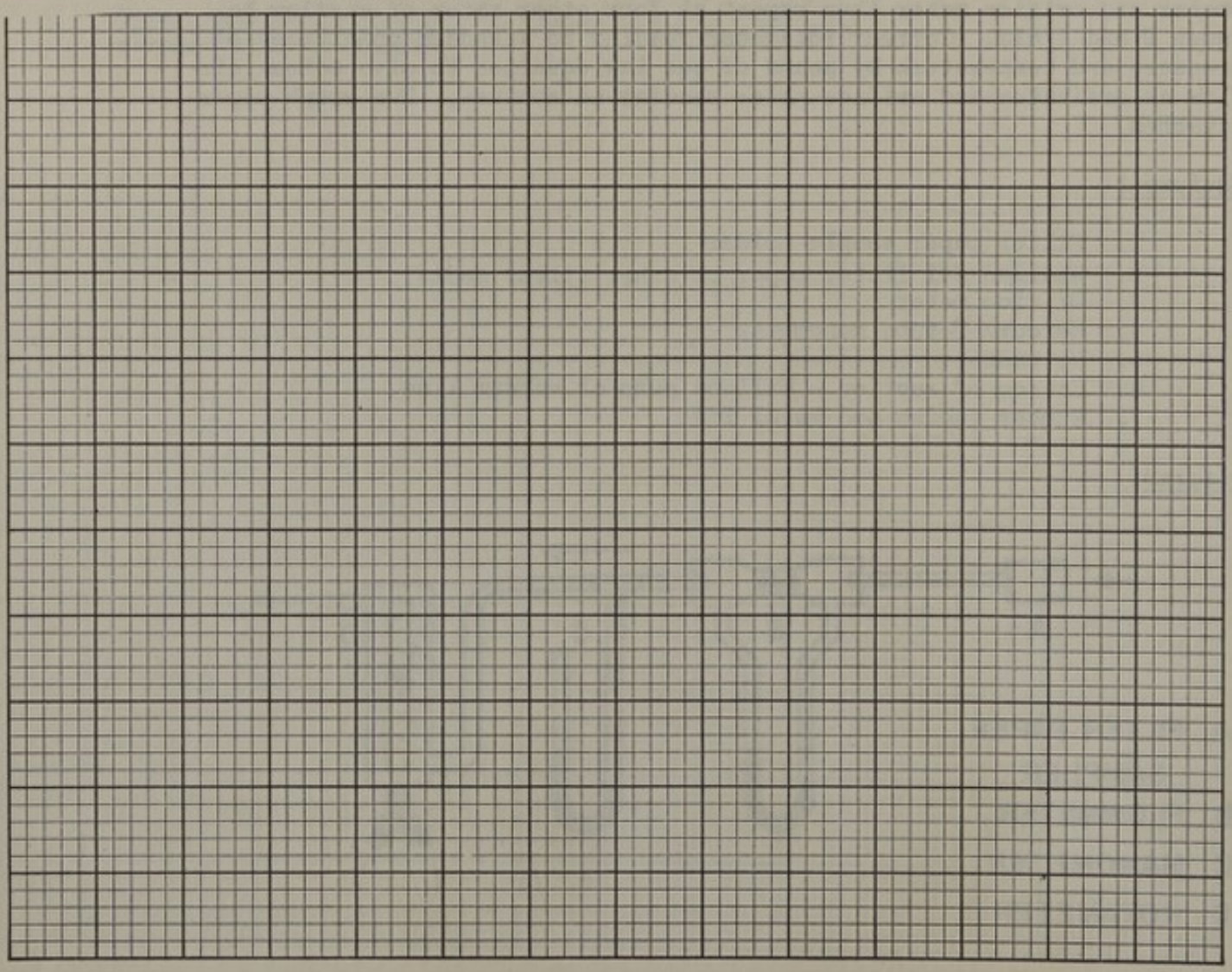
Data

| Rotameter Reading | Indicated Airflow From Curve | Mercury Manometer | Room Temperature | Actual Airflow | Orifice Meter Reading |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

References

1. Jacobs, M. B.: The analytical chemistry of industrial poisons, Hazards and solvents. Interscience Publishers, Inc., New York, 2nd Ed. page 63, 1949.

2. Perry, John H., editor: Chemical Engineers Handbook. McGraw-Hill Book Company, Inc., New York, 3rd Ed., page 408, 1950.



Laboratory Exercise - CALIBRATION OF BENZOL INDICATOR.

Object

To calibrate the benzol indicator using a static dilution system to produce known concentrations.

Theory

The benzol indicator measures the heat produced by the combustion of the air contaminant as it passes through the instrument. It is essentially a very sensitive explosion meter. It is not specific since anything which will burn will produce a reading. Therefore, one must know the identity of the contaminant and use the appropriate calibration curve. The sensitivity of the instrument is dependent upon the heat of combustion and the efficiency of combustion for various gases and vapors.

To calculate the volume of solvent necessary to produce a desired concentration in a given volume, the following calculation is made:

$$\text{ppm} = \frac{\text{wt of solvent}}{\text{mol wt}} \times \frac{22.4}{\text{tank vol}} \times \frac{760}{P} \times \frac{273 + T^{\circ}C}{273} \times 10^{6}$$

$$\text{and ml of solvent} = \frac{\text{wt of solvent}}{\text{sp g}}$$

Where: P = barometric pressure
T = temperature of gas mixture in degrees centigrade

Assuming instantansous dilution, the concentration ($C_t$) at any time (t) after dilution commences (when withdrawing the sample at a known constant rate) varies directly as the original concentration ($C_o$), and inversely as e raised to the power $V/V_o$, where V is the volume withdrawn from a container of volume $V_o$. $V = Rt$ where R = rate of flow and t time.

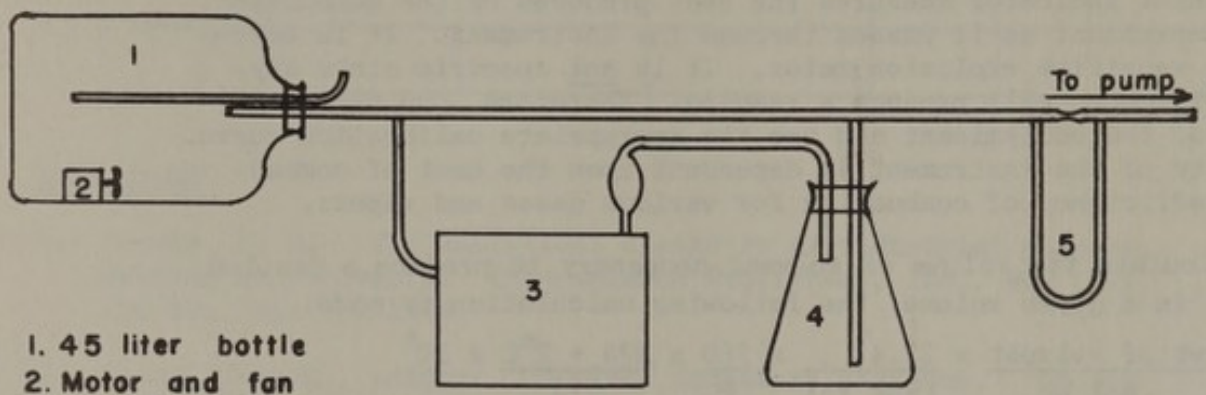
Benzol (benzene) is a colorless liquid, the lowest homologue of the aromatic hydrocarbons. Commercial benzol is 90% benzol and 10% toluene and zylene. Mol wt = 78.11; sp g = 0.879 at 20/4°C; boiling point = 79.7°C; melting point = 5.5°C. Soluble in water to 0.06 parts per 100 parts. Flash point = 12-10°C. Lower explosive limit 1.4% by volume. Upper explosion limit 8% by volume. The threshold limit is 35 ppm (= 0.11 mg per liter at 25°C and 760 mm).

Procedure

Plot curve of theoretical dilution (air changes versus percent of original concentration) on semilog paper. Set flow of dilution air at 4.5 liters per minute. For a 45 liter bottle, then, one air change is provided every 10 minutes or 1/5 air change is provided every 2 minutes. Draw exact volume of benzol into micro-pipette necessary to produce a

known concentration in the bottle of about 400 ppm. Drain pipette in the inlet airstream. Commence sampling and record zero time when the meter reaches approximately peak scale. Read instrument at 2-minute intervals and plot calibration curve, i.e., meter reading versus air concentration in ppm.

Equipment and Diagram




1. 45 liter bottle
2. Motor and fan
3. Benzol indicator
4. Surge tank
5. Flowmeter

Data

| Time-min. | Meter reading | Concentration-ppm |
|---|---|---|
| | | |
| | | |
| | | |
| | | |
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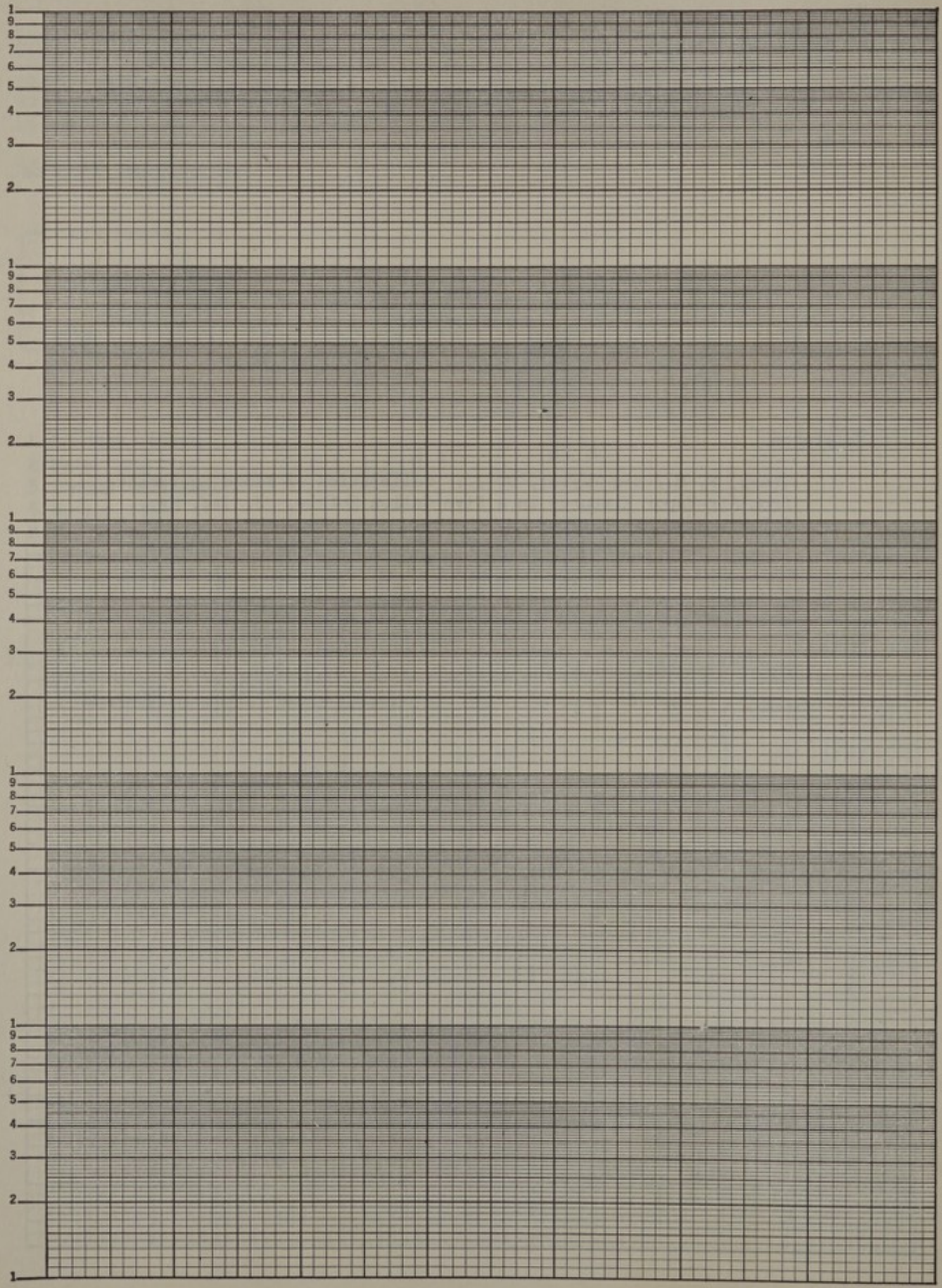
Calculations

Reference

Stead, F. M., and Taylor, G. J.: Calibration of Field Equipment from Air-Vapor Mixtures in a Five Gallon Bottle. J. Ind. Hyg. & Tox., November 1947.



Laboratory Exercise - CALIBRATION OF CARBON MONOXIDE INDICATORS.

Object

To calibrate a MSA carbon monoxide indicator and NBS CO indicator.

Theory

MSA Carbon Monoxide Indicator. Carbon monoxide (CO) is burned catalytically and the heat of combustion is measured by the potential developed in a thermopile. Since water vapor poisons the catalyst the test gas must be dried. The portable meter has a 1 - 2 minute lag and has a $\pm$ 10 percent accuracy.

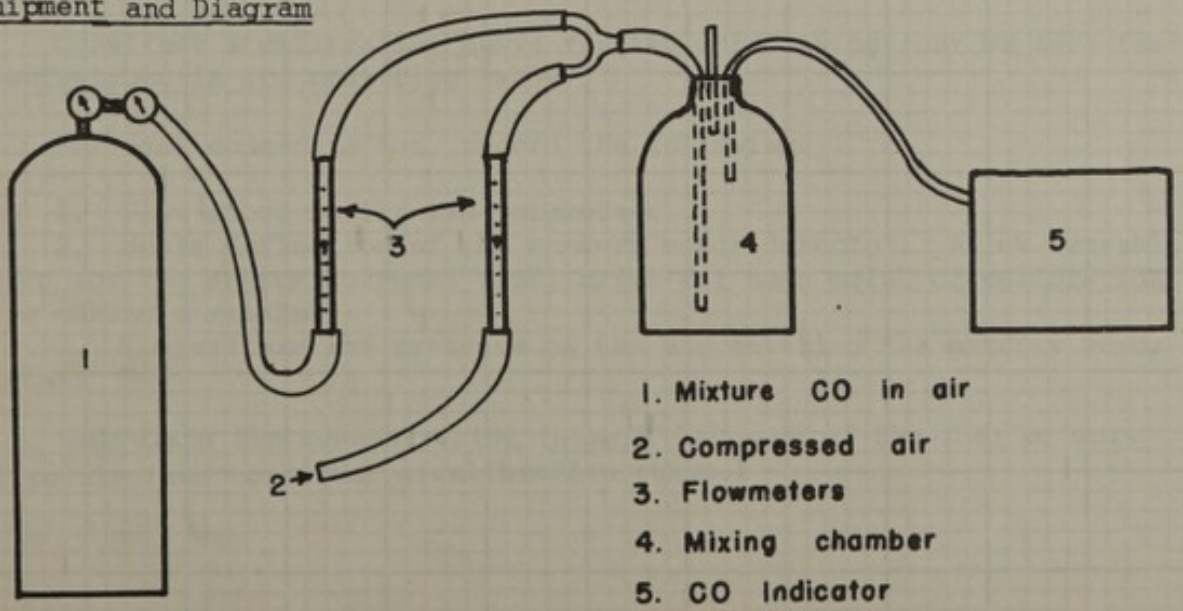
NBS CO Indicator. Carbon monoxide is pumped by a calibrated squeeze bulb through a small glass tube containing silica gel dryer and silica gel indicator. This indicator (ammonium molybdate - polladium sulfate - acid gel) changes color from yellow to green upon reaction with CO and the resultant shade is compared with color standards. This method is very sensitive and the instrument is very simple to use.

Procedure

Use a cylinder of compressed air containing CO of a known concentration as the standard test gas and dilute it to the desired concentration. Dilution is accomplished by regulating the flow of diluting air and air containing the CO. The ratio of flow rates determines the dilution factor. Air samples of the final air mixture measured with the two instruments above will provide the points for the calibration curves.

Since the threshold limit is 100 ppm, several points of calibration in this range should be made. For example, to obtain a point of calibration for 50 ppm using a reference gas mixture of 0.1% (1000 ppm), it must be diluted 1 to 19 parts of room air. Set flow rate of mixture from cylinder containing CO mixture at the desired rate and adjust the diluting airflow rate at 19 times this flow. The mixture will then contain 50 ppm. Proceed in a similar manner for concentrations of 75, 100, and 125 ppm. Prepare a calibration curve from the data obtained.

Equipment and Diagram



Data

| Volume cc/min. Known CO in Air | Volume cc/min. Room Air | Concentration of Mixture % | Concentration of Mixture ppm | Meter Reading hundredths of a percent | NBS - CO Indicator ppm |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
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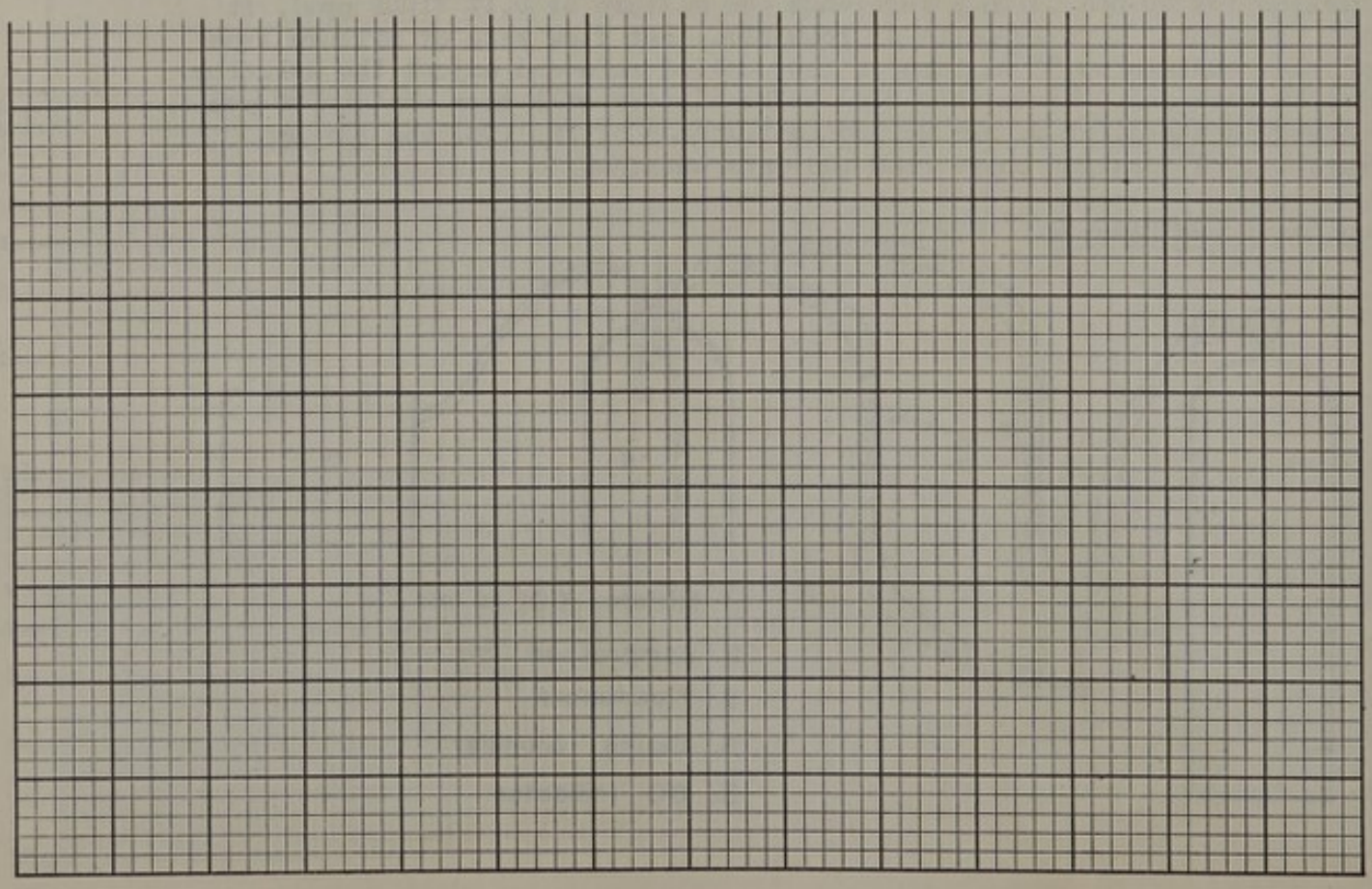
Calculations



Laboratory Exercise - CALIBRATION OF MERCURY VAPOR DETECTOR.

Object

To calibrate a mercury vapor detector, using a dynamic flow system to produce known concentrations of mercury vapor.

Theory

The mercury vapor detector is a direct reading instrument utilizing a photo-electric cell as the sensing element. A mercury vapor lamp, emitting 92% of its radiation at 2537A°, is the light source inside the meter. A fan draws an air stream between the U.V. lamp and the photocell. If any component of the air stream absorbs light at 2537A°, a scale deflection results. In addition to mercury, many solvent vapors such as benzol, toluol and acetone, and gases such as chlorine, ozone, sulfur dioxide and nitrogen dioxide also absorb U.V. in this region and will give meter readings. However, the instrument is far more sensitive to mercury vapor than to any other substance. A trace of mercury vapor will therefore interfere with the determination of any other gas or vapor, while an enormous concentration of another vapor would be necessary to interfere with a mercury determination.

At any given temperature, the concentration of mercury in the vapor phase in contact with the liquid phase is reproducible under equilibrium conditions, and can be calculated. The equilibrium (saturation) concentration of mercury vapor in contact with liquid mercury contained in an enclosure at atmospheric pressure is the partial pressure of mercury at the ambient temperature. This concentration can be diluted to any desired lower concentration for calibration purposes (see Appendix).

Procedure

Assume that the mercury in the saturation flasks is at equilibrium concentration.

The flow rates should be adjusted to supply at least 5.5 lpm or slightly more because the mercury vapor detector samples at a rate of 5.5 lpm at atmospheric pressure.

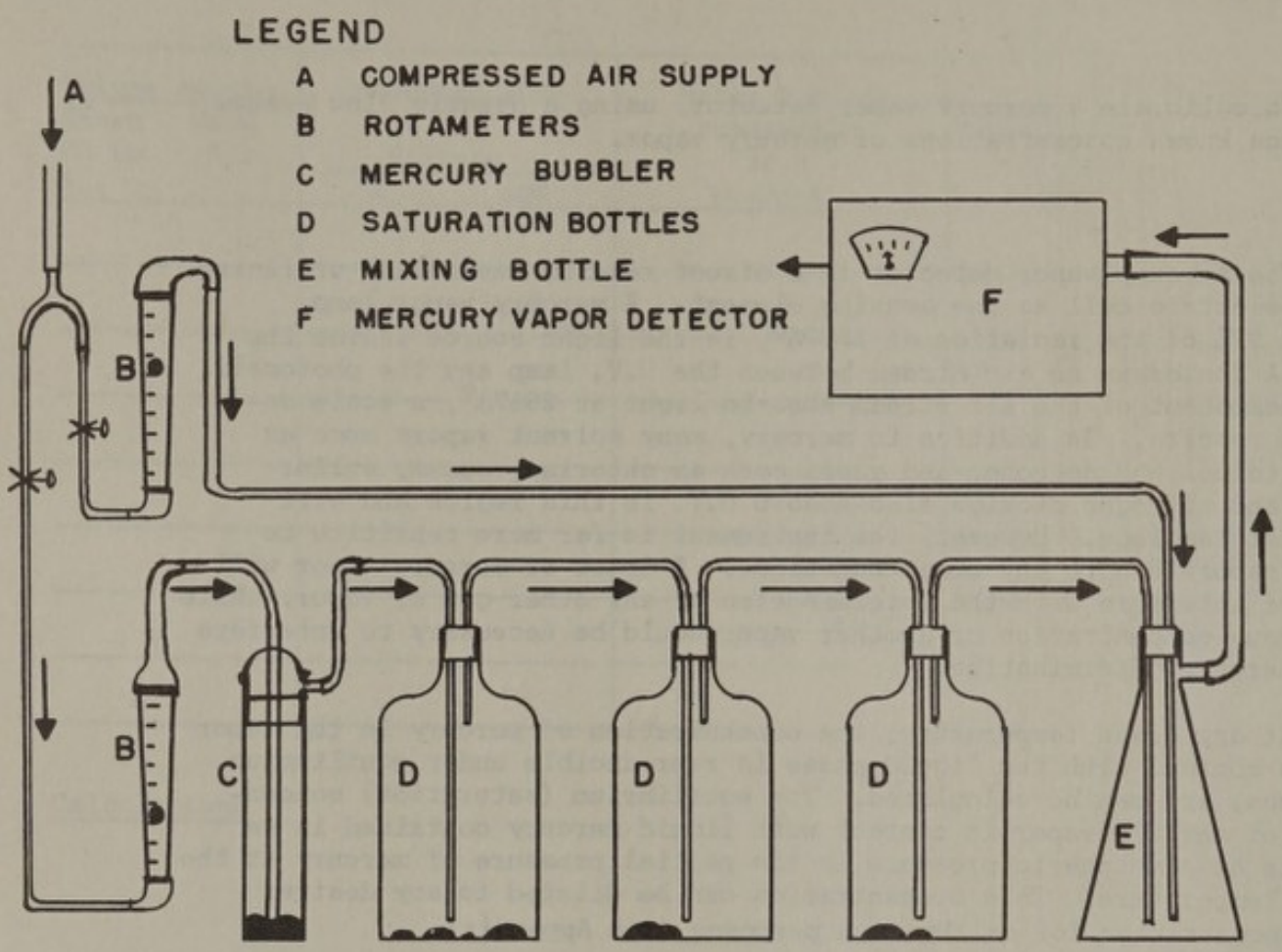
Construct a calibration curve for the meter using four or more test concentrations in its operating range.

For each concentration, record the following:

1. Flow rates in the two rotameters.
2. Scale deflection of the mercury vapor detector. Allow several minutes for the system to reach equilibrium for each point of calibration before making a reading.
3. Temperature and pressure of the air entering the mercury vapor detector.

Calculate the concentration in $mg/M^3$ at each of the four or more test points, and construct a calibration curve.

Equipment and Diagram




Data

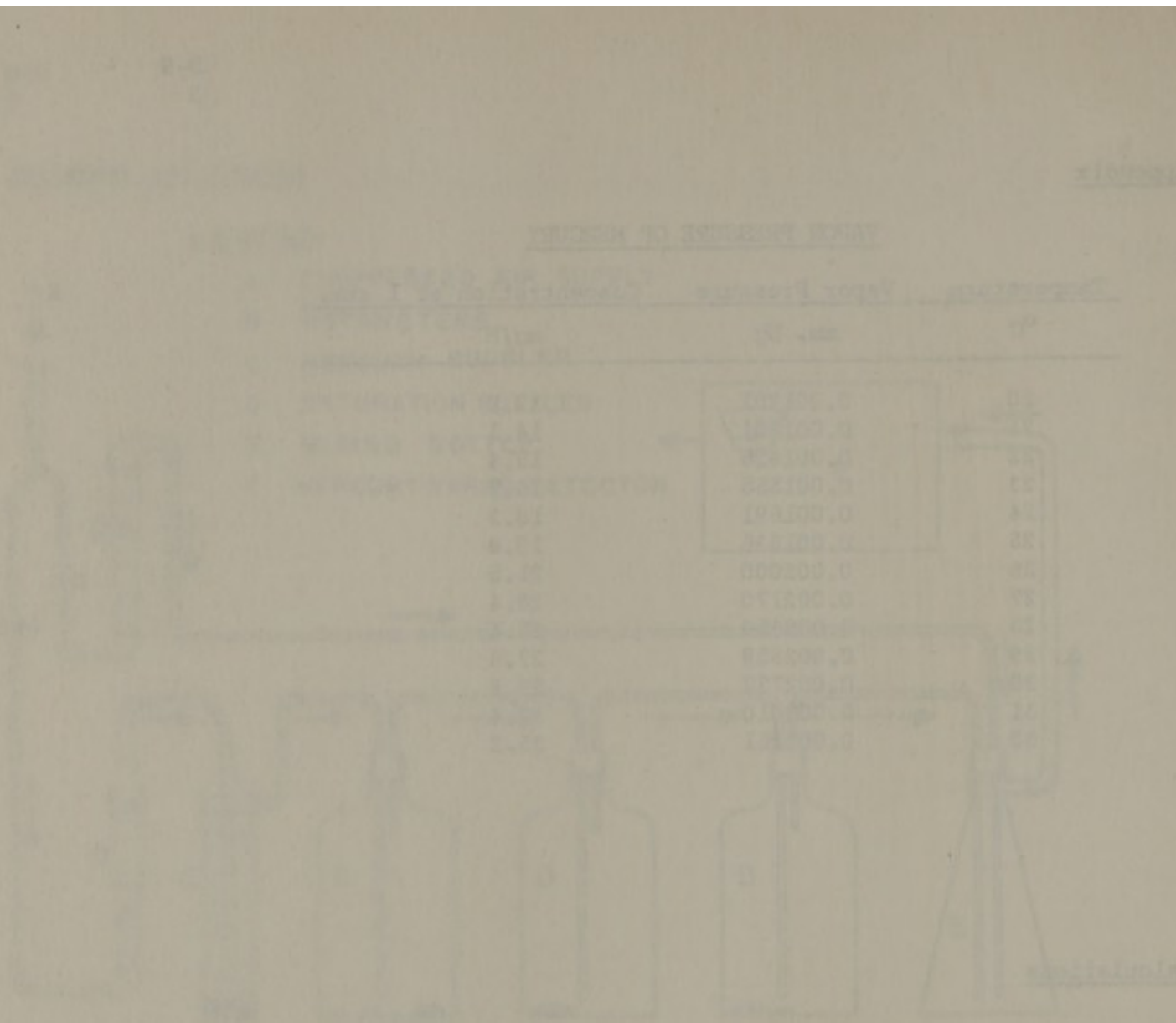
| Rotameter readings | | Scale reading Hg. Vapor Det. | Temperature | Pressure |
|---|---|---|---|---|
| 1. | 2. | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Appendix

VAPOR PRESSURE OF MERCURY

| Temperature °C | Vapor Pressure mm. Hg | Concentration at 1 atm. $mg/M^3$ |
|---|---|---|
| 20 | 0.001201 | 12.9 |
| 21 | 0.001301 | 14.1 |
| 22 | 0.001426 | 15.4 |
| 23 | 0.001555 | 16.7 |
| 24 | 0.001691 | 18.2 |
| 25 | 0.001836 | 19.8 |
| 26 | 0.002000 | 21.5 |
| 27 | 0.002170 | 23.4 |
| 28 | 0.002359 | 25.4 |
| 29 | 0.002559 | 27.6 |
| 30 | 0.002777 | 29.8 |
| 31 | 0.003010 | 32.4 |
| 32 | 0.003261 | 35.2 |

Calculations



Laboratory Exercise - CALIBRATION AND USE OF HALOGENATED HYDROCARBON INSTRUMENTS.

Object

To become familiar with the various instruments used for determining atmospheric concentrations of halogenated hydrocarbons, and to demonstrate the use of a large chamber for preparation of known vapor concentrations.

Theory

The halide lamp and halide meter operate on the principle of the Beilstein reaction: a halogen vapor coming in contact with hot copper causes vaporization of a copper halide which produces the copper spectrum in the arc or flame. The visible color changes from green to blue as the concentration increases. The MSA Safety Data Sheet for methyl bromide, for example, gives the following table for the halide lamp.

| Methyl bromide - ppm | Flame |
|---|---|
| 0 | Almost invisible |
| 25 | Faint fringe of green |
| 50 | Moderate green |
| 125 | Green |
| 250 | Strong green |
| 500 | Strong green-blue fringe |
| 800 | Strong blue green |
| 1000 | Blue |

The Davis Halide Meter measures the intensity of the blue line of the copper spectrum. The light produced in an arc between a copper and a platinum electrode passes through a blue filter to the phototube. The phototube output unbalances a Wheatstone bridge, and the unbalance is amplified and read on the microammeter.

The LKB Gas Tester indicating tube for perchloroethylene and trichloroethylene uses an impregnated indicating gel but the reagent used is a trade secret with the manufacturer.

Procedure

Calculate the quantity of trichloroethylene necessary to produce a concentration of 25 ppm in the chamber by using the following formula:

$$\text{ppm} = \frac{\text{wt of solvent}}{\text{mol wt}} \times \frac{22.4}{\text{chamber vol}} \times \frac{760}{P} \times \frac{273 + T^{\circ}C}{273} \times 10^{6}$$

$$\text{and ml solvent} = \frac{\text{wt of solvent}}{\text{sp.g}}$$

where P = barometric pressure

T = temperature of gas mixture in $^{\circ}C$.

Flush out the chamber and inject enough trichloroethylene, contained in the syringe, into the chamber to produce a concentration of 25 ppm. Take readings on the Davis Halide Meter, LKB Gas Tester and halide lamp as quickly as possible. Add the same amount to obtain a total concentration of 50 ppm and again take readings. Add solvent to obtain total concentrations of 100 ppm, 200 ppm, 400 ppm and 800 ppm and take readings after each addition of solvent. Draw a calibration curve for the Davis Halide Meter, and corrections for the LKB Gas Tester if necessary.

Equipment

1. 125 cu. ft. stainless steel chamber with blower, piping, circulating fan, ports and windows.
2. Syringe with threaded push rod.
3. Davis Halide Meter.
4. Halide leak detector (Freon leak detector).
5. LKB Gas Tester for perchloroethylene, trichloroethylene.

Data

| Solvent used ml | Calculated concentration ppm | Halide meter reading | LKB indicator reading |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

Calculations

Laboratory Exercise - COUNTING AND SIZING AIR-BORNE DUST.

Object

To prepare slides for dust counting and particle sizing, and to count and size a dust sample.

Theory and Discussion

The theory of the microscope can be found in the pamphlets issued by the Bausch & Lomb Optical Company and in standard handbooks.

In the evaluation of a dusty atmosphere for a potential health hazard, it is important to know the number and size of the dust particles in addition to the chemical composition of the dust. In this exercise, particles as small as 0.7 micron will be counted and particles as small as 0.25 micron will be sized. By knowing the number, the size and chemical composition of the air-borne dust, a true evaluation of the exposure can be made.

When preparing a slide for dust counting or particle sizing, it is essential that a representative portion of the sample be used. The sample should be mixed thoroughly and the aliquot should be transferred to the cell by a clean pipette.

Procedure

The laboratory exercise is divided into three parts: (1) preparation of slides for counting and particle sizing, (2) dust counting, and (3) particle size determination.

The students will be divided into three groups and will accomplish the exercise in rotation.

Preparation of slides. Each student will secure the parts of a Dunn cell (base plate, center section and cover slip) and wash them carefully using a soap solution followed by a series of rinses first with tap water and then with distilled water. The cell parts should then be dried with lens paper. Upon inspection of the base plate if more than 3 dust particles per grid field are found, it should be recleaned. The Dunn cell can then be formed and filled with a representative portion of the sample (see discussion). After a few minutes the cell should be viewed under a microscope to see whether the particle number is too large to count, in which case dilution of the sample is required.

Each student will clean two microscope slides and two cover slips. On each slide a drop of sample will be placed and evaporated slowly and cautiously over a flame. When completely dry the slide should be allowed to cool for a few minutes. A clean cover clip is placed over the dust deposit and sealed with Technicon.

Dust counting. (1) The fixed slides have been prepared by sealing a small glass cover slip over a dust deposit on the grid of a haemocytometer cell. The instructor will point out the area in which dust is to be counted. Students will count 3 slides, 3 times each. The counts are to be made in rotation, i.e., one count on the first slide will be followed by one count on the second and third slides before the 2nd count is made on the first slide, etc. The student will pass the Veeder-Root counter to the instructor without looking at it.

(2) The Dunn cells will be made up by the instructor. Each student will count two cells, making 5 counts on each cell. As an aid in finding the dust, a wax pencil mark is made on the base plate near the cell and the microscope is focused on this mark. Then, after placing the cell directly beneath the objective, the fine adjustment is rotated three full turns in the direction necessary to lift the objective. Additional adjustment may be necessary to bring the dust particles in good focus.

Calculate the dust concentration using the dust counts and the information supplied by the instructor.

Calibration of ocular grid. A stage micrometer will be placed on the stage and the ruled lines brought into focus. Measure the width and length of the entire ocular grid. The distance between the longer lines in the micrometer is 0.1 mm and at one end this distance is further subdivided by lines 0.01 mm apart.

Particle size determination. Obtain Kohler illumination as described in the Bausch & Lomb pamphlet. Open the microscope condenser diaphragm and use this lighting system for oil immersion work with the oil objective of the microscope. Lower the microscope condenser slightly and place a drop of immersion oil on its top lens. Place a stage micrometer on the microscope stage with the ruled area centered over the microscope condenser lens. Raise the microscope condenser until the oil on its top lens contacts the lower side of the stage micrometer slide. Place a drop of oil over the rulings on the top of the stage micrometer slide and lower the microscope oil immersion objective lens until it contacts the oil drop, then lower it further with extreme caution until it almost touches the top of the stage micrometer slide. This operation must be done while observing the slide and lens from the side; it must never be done while looking through the microscope. Focus the microscope upward while looking through the eyepiece and searching for the micrometer rulings. Undoubtedly several attempts will be required to locate the rulings.

When the micrometer rulings are brought into focus, align one of the rulings with one of the indices of the filar micrometer or eyepiece graticule, and obtain a ratio of filar units or graticule "d" units to stage micrometer units. (1 mm = 1000 μ). Calculate the number of microns per filar or "d" unit.

Observation of dust. Place a prepared slide on the microscope stage with oil immersion in the same manner as the stage micrometer was placed on the stage. Locate the dust by focusing upward. Determine the size of a number of particles both with a filar eyepiece and an eyepiece graticule.

Treatment of data. After 50 particles have been sized complete the data sheet and plot the data on log probability paper. Determine the median size and the standard geometric deviation of the dust.

Equipment

1. Preparing slides

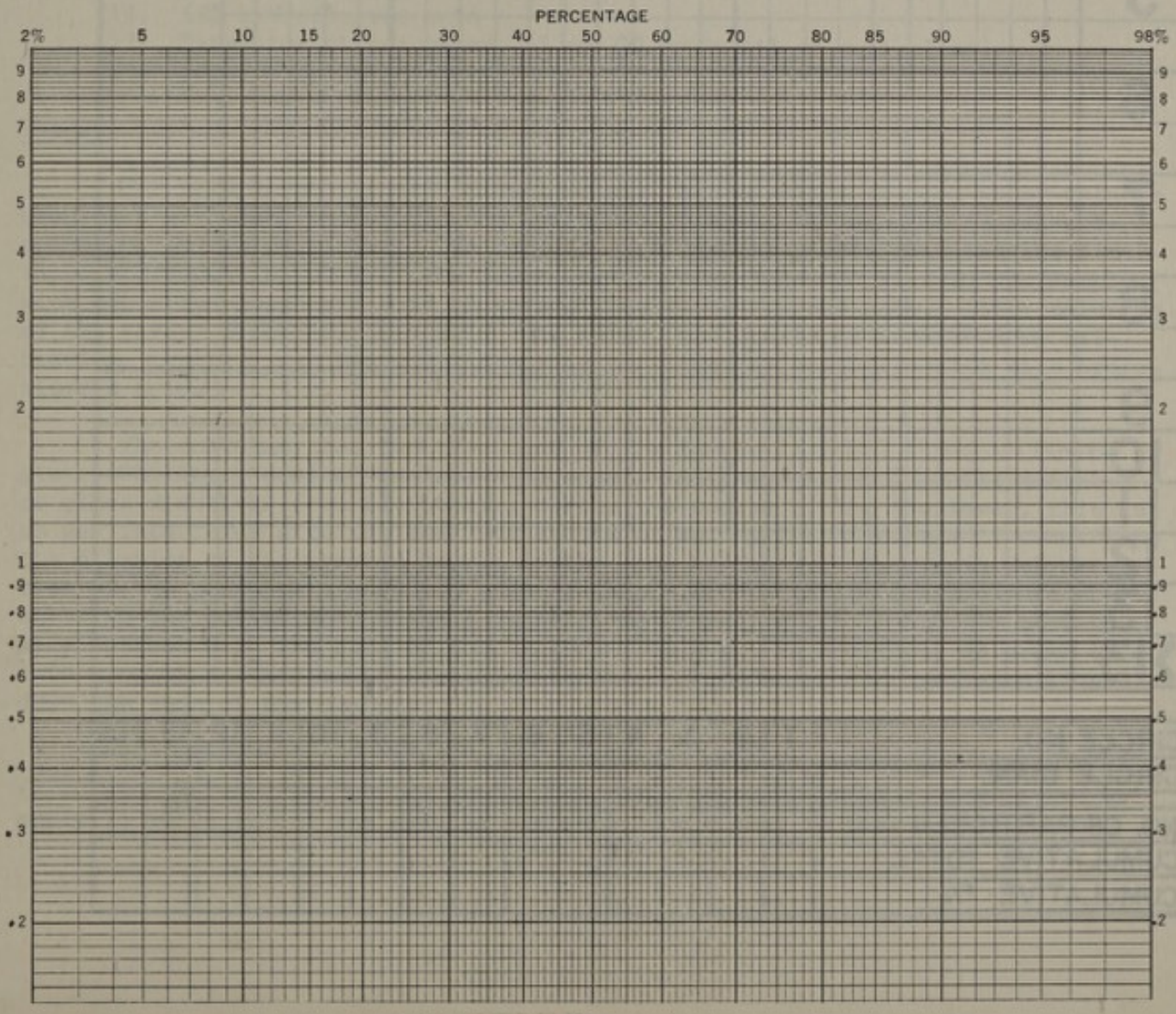
   Microscope slides and cover slips, pipette, flame Technicon sealing fluid, microscope, Dunn cell base plates and center sections

2. Counting

   Microscope with 10x ocular and 10x objective, Page ocular disc, stage micrometer, prepared Dunn cell slides, prepared fixed dust slides, Veeder-Root counter

3. Particle size determination

   Microscope with 10x or 20x ocular and a 97x oil objective, stage micrometer, filar micrometer, eyepiece graticule, log probability paper, prepared particle size slides



# PARTICLE SIZE DATA SHEET

SAMPLE NO. DATE EXAMINED:

DESCRIPTION: BY:

| CIRCLE | PARTICLE TALLY | TOTAL |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| >13 | | |

| CIRCLE NO. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | >13 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| CIRCLE DIAM. | | | | | | | | | | | | | | |
| NO. OF PARTICLES | | | | | | | | | | | | | | |
| CUMULATIVE TOTAL | | | | | | | | | | | | | | |
| CUMULATIVE % | | | | | | | | | | | | | | |

Laboratory Exercise - CHEMICAL DETERMINATION OF FREE SILICA

Object

To determine the percent by weight of quartz in a sample of mineral dust.

Apparatus and Reagents

1. 1 Phillips beaker, 250 ml
2. 1 Funnel, 50 mm, with stem bent at 45° angle
3. 1 Precision heater, 550 watt, 115 volt, Type RH
4. Crucible tongs, cushioned with rubber tubing
5. 1 Funnel, 70 mm
6. 1 Funnel rack
7. 2 Wash bottles (distilled water and 1-9 HCl)
8. 1 Griffin beaker, 400 ml
9. 1 Graduated cylinder, 10 ml
10. 1 Graduated cylinder, 100 ml
11. 1 Pipet or buret, 25 ml
12. 1 Platinum crucible
13. 85% phosphoric acid
14. Fluoboric acid
15. 1-1 sulfuric acid
16. 48% hydrofluoric acid
17. Retentive filter paper, 11 cm (Whatman #42 or equivalent)
18. Paper pulp suspension

Theory

Hot pyrophosphoric acid dissolves silicate minerals forming water-soluble complexes with both the acidic and basic constituents but has little solvent action on quartz particles. The formation of pyrophosphoric acid and the simultaneous dissolution of silicate minerals is accomplished by heating the sample with 85% orthophosphoric acid at an empirically determined rate. After dilution of the sirupy pyrophosphoric acid with water, the quartz is separated by filtration, ignited, weighed and its purity confirmed by volatilization with hydrofluoric acid.

The container for the treatment of the sample is a 250 ml borosilicate glass, Phillips, conical beaker, with spout. Although a considerable amount of glass dissolves from the beakers during the course of the treatment, this in no way interferes with the analysis and is offset by the advantage of conducting the dilution of the treated sample in the same container. Too rapid dehydration of the phosphoric acid and loss of sample by spattering are prevented by covering the beaker with a small funnel, the stem of which is sharply bent to make contact with the side of the beaker. The stem, in addition, is drawn out into a point or beveled to facilitate return of condensed and spattered liquid down the side of the beaker.

As source of heat, a 550 watt, 115 volt, Type RH, Precision heater (Precision Scientific Co.) is used. The rheostat of the heater is adjusted to apply 75 volts across the heating element. The beaker is placed directly on

the element of the heater without the use of accessory refractories. Ordinary crucible tongs, cushioned with rubber tubing, are used for handling the hot beakers.

Continuous mechanical swirling of the beaker with a rotator has been found desirable when analyzing large numbers of samples. A retainer for the beaker may be constructed of 0.25-inch pressed asbestos board with a hole slightly larger than the bottom of the beaker and fitted flush with the heating element. About 5 volts less is required when the retainer is used.

The fluoboric acid which is used in the procedure is prepared by diluting commercial 45% fluoboric acid with 2 parts of saturated boric acid solution or by pouring, with continuous stirring, 1 pound of 48% hydrofluoric acid into a mixture of 450 ml of water and 250 grams of boric acid crystals contained in a plastic or hard rubber vessel set in a pan of ice water. Sufficient heat is evolved by the reaction to dissolve all of the boric acid, part of which should recrystallize upon cooling to room temperature. While glass cannot be used for storing the acid, glass apparatus may be used for filtering and measuring the cool acid.

Procedure

Grind the material to pass through a 200 mesh sieve, mix thoroughly, and weigh out 0.5 gram or less on a watch glass and transfer to a 250 ml Phillips beaker by means of a small camel's hair brush. Add 25 ml of 85% orthophosphoric acid and cover the beaker with a funnel. Place the beaker directly on the element of a Precision heater which has been allowed to preheat for 45 minutes, and begin timing with a stop watch. Swirl the beaker continuously during the heating period with the rotator in order to minimize superheating and to keep the sample distributed throughout the acid. At the end of 12 minutes remove the beaker from the heater and swirl by hand for 1 minute to dissolve any gelatinous silica which may have deposited on the side of the beaker above the acid. Set the beaker on a cool surface and immediately but cautiously remove the funnel in such a way that adhering liquid runs down the side of the beaker. Allow to cool to room temperature and then wash down the sides of the beaker rapidly with 125 ml of hot (60° to 70°C) water and swirl vigorously until the sirupy phosphoric acid is completely dissolved. Wash down the upper part of the beaker with 10 ml of fluoboric acid and swirl to mix. Then wash down the upper part of the beaker with 25 ml of water and filter through a retentive paper into which has been introduced a small amount of a suspension of paper pulp. Transfer the residue quantitatively to the filter with water and wash ten times with 1 to 9 hydrochloric acid, followed by one or two washings with water.

Ignite the filter paper in a tared platinum crucible, first at low heat to char the paper, and finally for 20 minutes at about 950°C. Cool the crucible in a desiccator and weigh. Moisten the residue with 1 to 1 sulfuric acid and add 5 ml or more of 48% hydrofluoric acid. Heat gently until the quartz appears completely dissolved, then increase the heat to volatilize the acids. Repeat the treatment of the residue with sulfuric and hydrofluoric acids to ensure complete volatilization of the silica, then ignite at about 950°C for a few minutes, cool, and weigh.

Data*

Sample _____

Wt. of sample (A) _____

Wt. of platinum crucible and $H_3PO_4$ residue

Wt. of platinum crucible No. ____ _____

Wt. of $H_3PO_4$ residue (B)

Wt. of platinum crucible and HF residue

Wt. of platinum crucible No. ____ _____

Wt. of HF residue (C)

Calculations‡

$$\frac{B \times 100}{A} = \frac{\quad\quad}{\quad\quad} \times 100 = \quad \% H_3PO_4 \text{ residue (D)}$$

$$\frac{C \times 100}{A} = \frac{\quad\quad}{\quad\quad} \times 100 = \quad \% \text{ HF residue (E)}$$

$$D - E = \quad \% \text{ Quartz}$$

* Report weights to 0.1 mg

‡ Report results to 2 decimal places.

Reference

Talvitie, N. A., "Determination of Quartz in Presence of Silicates Using Phosphoric Acid," Anal. Chem. 23, 623 (1951).

Laboratory Exercise - DETERMINATION OF LEAD IN URINE.

Object

To determine lead in urine using the USPHS method, and to prepare a standard lead curve.

Apparatus and Reagents

1. 3-125 ml Squibb type separatory funnels.
2. Matched test tubes for the spectrophotometer with 2 centimeter path lengths.
3. Beckman DU spectrophotometer.
4. Glassware: All glassware should be borosilicate, and should be rinsed with strong nitric acid (used over and over), followed by tap and distilled water. It is best to reserve glassware for samples containing low concentrations of lead to avoid contamination.
5. Reagents: Analytical grade reagents are used. Purification is essential for analysis of biological materials because of the very low levels of lead; for air samples containing sufficient lead it may be omitted.

a. Double-distilled water, prepared in an all-glass still. May not be necessary for air samples containing sufficient lead.

b. Nitric acid, concentrated - redistill in an all-glass still.

c. Ammonium hydroxide, concentrated - distill 3 liters into 1½ liters of double-distilled water chilled in an ice bath (the condenser outlet should be deeply submerged in the water) until the volume increases to 2 liters, using an all-glass still. Lead-free ammonium hydroxide may also be prepared more conveniently from tank ammonia in the same manner.

d. Extraction dithizone - dissolve 16 mg diphenylthiocarbazone (dithizone) in 1 liter of chloroform. Keep in a brown bottle in the refrigerator. The label of the brand of chloroform should indicate that it passes the American Chemical Society suitability test for use with dithizone; when a minute amount of dithizone is added to some in a stoppered test tube the faint green color should be stable for a day.

e. Standard dithizone - dissolve 8 mg diphenylthiocarbazone (dithizone) in 1 liter of chloroform. Keep in a brown bottle in the refrigerator, and allow to warm to room temperature before using. The quality of the chloroform should be as indicated for extraction dithizone.

f. Sodium citrate - dissolve 125 grams of the 5½ $H_2O$ salt in distilled water to make almost 500 ml, add a very small amount of phenol red indicator and a few drops of sodium hydroxide till a strong red color (pH 9-10) appears. Extract in a separatory funnel with a strong dithizone until a green extract is obtained. Add a small amount of citric acid until an orange color (pH 7) appears, and then extract the excess dithizone with chloroform until a colorless extract is obtained. Remove the chloroform.

g. Hydroxylamine hydrochloride - dissolve 20 grams in water to make 65 ml, add a few drops of m-cresol purple indicator, and ammonium hydroxide to a yellow color (pH 3). Add sufficient 4% solution of sodium diethyldithiocarbamate to combine with metallic impurities. After a few minutes, extract with chloroform until the excess reagent is removed. The absence of a yellow color in the chloroform when a portion of the extract is shaken with a dilute copper solution indicates when this point has been reached. Add distilled hydrochloric acid until the indicator turns pink, and adjust the volume to 100 ml with double-distilled water.

h. Potassium cyanide - prepare a practically saturated solution containing 50 grams of potassium cyanide. (A weaker solution will not have the proper pH). Extract the lead with dithizone in chloroform (about 30 mg/l) until a green extract is obtained, then extract the excess dithizone with chloroform. Dilute with double-distilled water to 500 ml.

i. Ammonia-Cyanide mixture - mix 200 ml purified 10% potassium cyanide with 150 ml distilled ammonium hydroxide (specific gravity 0.9) and make to 1 liter with double-distilled water. If the specific gravity of the distilled ammonia is not 0.9, the equivalent volume should be computed from table of specific gravity vs. percent ammonia, and this amount used instead (Sp. Gr. 0.9 = 28.4% $NH_3$).

j. 1:99 Nitric acid - dilute 10 ml redistilled nitric acid to 1 liter with double-distilled water.

k. Standard Lead Solution - dissolve 1.5984 g pure lead nitrate in 1 liter of 1:99 nitric acid to produce a stock solution containing 1 mg Pb per ml. Prepare working solutions by diluting with 1% nitric acid to a strength of 2 micrograms Pb per ml.

l. Indicator solutions - 0.1% Phenol red, m-cresol purple.

Theory

Analysis of urine for lead is a valuable means of establishing lead absorption. The urine sample is ashed with nitric acid to destroy organic matter. It is then brought into solution and the lead dithizone complex is formed under proper conditions and extracted by chloroform. After appropriate purification steps, the color is measured photometrically. The method is standardized with known lead solutions treated in the same manner. A straight line plot is obtained when optical absorbance is plotted against known quantities of lead.

Procedure

This exercise will be conducted over the course of two afternoons, part-time in conjunction with the free silica determination. A set of 50 ml urine samples containing known quantities of added lead will be provided in 125 ml Phillips beakers. To save time, these samples will have been evaporated to dryness on a steam bath after the addition of a small amount of nitric acid.

Select one sample, noting the number. During the first afternoon complete the ashing of this sample by following the directions given in the second paragraph of the method (Appendix). Several volunteers will also ash reagent blanks which will be carried subsequently through the entire procedure. When time permits, wash 3 separatory funnels with concentrated nitric acid, tap and distilled water as will be demonstrated. (Two additional clean funnels will be required for those running blanks.) Initial these funnels for your use on the following afternoon.

During the second afternoon dissolve the ashed sample, according to the instructions in the method (Appendix), with nitric acid and distilled water. As a half hour or more of heating may be required for this operation, it should be started at the beginning of the period. This time can then also be used for carrying out the free silica determination. The remainder of the analysis will

then be completed. Please do not contaminate the reagents by careless use of the pipets. DANGER! A rubber bulb will be used with the pipets. Do not pipet potassium cyanide by mouth as it is highly poisonous! Note that many of the reagents are provided in automatic burets for greater convenience and minimal contamination. This analysis will require two of the separatory funnels cleaned on the first day.

Standardization (See Standardization Section of Appendix).

The third separatory funnel will be used for preparing a lead standard, from a standard lead solution provided in a buret. The first two students will prepare zero standards which will be used as the photometer references. Each following student will then select one of the lead values indicated on a sheet near the buret, noting his initials after the selected value. Add the indicated quantity of lead and apply the procedure starting at the appropriate step indicated in the fourth paragraph of the method (Appendix). The results will then be plotted to provide the standard curve, at the conclusion of the exercise.

Calculations

Determine from the standard curve the number of micrograms of lead required to produce an optical absorbance of exactly one. Multiply by this factor the experimentally determined absorbance values of the sample and of the blank to convert to micrograms of lead. The difference between these two values, divided by the volume of urine in milliliters, gives the milligrams of lead per liter of urine.

The use of a standardization factor as given above eliminates the necessity of graphically determining each lead quantity, and gives more accurate results with less effort. This method is possible because the relationship between absorbance and micrograms of lead is linear.

## APPENDIX

## METHODS FOR DETERMINING LEAD IN AIR AND IN BIOLOGICAL MATERIALS

U.S. Public Health Service

Procedure for Urine*

Collection of a 24-hour sample in a 2 liter glass-stoppered bottle is recommended. Measure 50 ml of the fresh urine sample into a 250 ml Phillips beaker. If the urine is over a day old or contains a precipitate, empty the sample bottle into a stoppered graduated cylinder, dissolve the precipitate adhering to the glass, and rinse out with two small portions of nitric acid, followed by one of double-distilled water. Mix the urine and rinsings cautiously (carbon dioxide is generated) and take a volume of the mixture equivalent to 50 ml of urine (calculated from the original volume of the sample and the volume of rinsings added).

*Suitable unless the presence of bismuth in the urine is definitely expected. (See Elkins, H. B., and Ruotolo, B. P. W., Amer. Ind. Hyg. Assoc. Quarterly 14, 111-112 (June 1953) for a discussion of the occurrence of bismuth.)

Evaporate to dryness overnight on the steam bath. Add 1-2 ml of nitric acid and heat on the hot plate at a moderate heat for a few moments, then swirl the beaker to dissolve and mix the residue. Continue heating until the reaction ends and all the water is driven off. Cover the beaker with a watch glass for the remainder of the ashing. Repeat the treatments with nitric acid, gradually decreasing the portions to 0.5 ml and gradually increasing the heat of the hot plate, charring as much as possible and cooling the beaker before each addition. Overheating causes violent flashing and should be avoided to prevent loss of sample. After 3 or 4 portions of nitric acid have been added, the hot plate should be at full heat (400°C). The most powerful ashing occurs when the dry salts decompose giving off red fumes of nitrogen dioxide. Following the production of a white ash, add a few more portions of nitric acid to ensure complete ashing. Less than 10 ml of nitric acid suffices in most cases for the whole process.

To the ashed sample add 2 ml nitric acid and 10 ml of double-distilled water, and heat gently until a clear solution is obtained. Cool to room temperature and add 1 ml 20% hydroxylamine hydrochloride, 10 ml 25% sodium citrate, 1 drop phenol red indicator, and titrate to a strong red color (pH 9-10) with redistilled ammonium hydroxide. (Phenol red has a weak orange-red color in strong acid, yellow in weak acid, and a red color in alkaline solution. Do not mistake the first color for the end-point.) Transfer with double-distilled water to a 125 ml Squibb separatory funnel containing 5 ml 10% potassium cyanide. Shake 1-2 minutes with 5 ml portions of dithizone (16 mg/l in chloroform) until a green extract indicates that all the lead has been removed (generally two times). The addition of the chloroform initiates precipitation of calcium and magnesium phosphates; hence, once extraction is begun it should be completed without delay. Combine the extracts in a second funnel containing 30 ml of 1:99 nitric acid.

The extracted lead is then stripped from the chloroform layer by shaking the second funnel for two minutes, and the chloroform is discarded. (Note: Standards are started at this point.) The aqueous layer is then washed by shaking with 5 ml chloroform, which is discarded with as close a separation as possible. The last drop of chloroform floating on the surface is evaporated by blowing air gently into the funnel.

Add 6.0 ml ammonia-cyanide mixture, exactly 15 ml standard dithizone (8 mg/l in chloroform), and shake for two minutes. Insert a pledget of cotton into the stem of the funnel and drain the chloroform layer into a dry photometer tube. (A set of matched test tubes, 22 x 175 mm may be used.) Inspect for water droplets in the light path - if present, carefully decant the tube into another dry photometer tube, leaving the water behind. Read the absorbance at 510 millimicrons using the blank as a reference. Report lead content as milligrams per liter of urine. (Normal range 0.01-0.08.)

Standardization: The standard dithizone should be aged a day or two before using or standardizing and should be restandardized every month or two. Prepare a series of funnels containing graduated amounts of lead up to 25 micrograms in exactly 30 ml of 1:99 nitric acid. The method is applied to these standard samples starting at the appropriate step in the fourth paragraph. The zero standard is used as the photometer reference, and the absorbance values are plotted against micrograms of lead to obtain the standard curve.

Procedure for Blood

A 5-10 ml sample in a vacutainer or test tube is satisfactory for analysis. Weigh the entire sample into a 125 ml borosilicate Phillips beaker. Add 1 ml nitric acid and evaporate to dryness on the hotplate at a low heat. Continue the ashing in the manner described for urine. Final solution of blood ash takes more time because of the iron content. The analysis follows the same procedure as for urine, except that the quantity of sodium citrate may be reduced to 4 ml. Report lead content as milligrams per 100 grams of blood (normal range 0.01-0.08).

Procedure for Air Samples

Electrostatic precipitator tubes are conveniently washed out with ethanol using a special policeman made with a rubber disc cut to fit like a piston. Contents are washed using a short stem funnel into a 250 ml borosilicate Phillips beaker and evaporated to dryness. Impinger samples may be transferred to the same type of beaker and evaporated.

Ashing is performed by adding 1-2 ml portions of nitric acid at a moderate heat. The ashing may be speeded in the LATER stages by adding a few drops of perchloric acid WITH the nitric acid. (CAUTION: Explosive with large amounts of organic matter in absence of nitric acid!) In the event that the ash content of the sample is very low, the addition of 1 ml of 10% sodium sulfate or bisulfate at the beginning will prevent loss of lead on the glass; however, this should not be used if the amount of lead is large enough to precipitate. The ash is then dissolved in 1-2 ml nitric acid and diluted to a convenient volume (100 ml) in a volumetric flask.

An aliquot is then analyzed by the procedure for blood. The amount of lead may be estimated by the number of 5 ml portions of dithizone required for the preliminary extraction, each portion being equivalent to about 20 micrograms of lead when a saturated red color is obtained. A convenient amount to be taken for analysis should be such that no more than 10 portions are needed.

If the estimated amount of lead exceeds the range of the method (25 micrograms), the 1:99 nitric acid containing the stripped lead (at the point indicated for standards to be started) is then aliquoted, and this aliquot is made up to 30 ml with additional acid in another funnel. The remainder of the procedure is then continued on the second aliquot.

When reporting the results, allowance is made for the two aliquots. It is most convenient to aliquot the blank in the same manner to simplify the correction.

Laboratory Exercise - POLAROGRAPHIC DETERMINATION OF LEAD, CADMIUM AND ZINC.

Object

To demonstrate the polarographic determination of lead, cadmium and zinc in an atmospheric dust or fume sample.

Apparatus and Reagents

1. Sargent Model XXI visible recording polarograph, including dropping mercury electrode assembly
2. Electrolysis vessel
3. Constant temperature bath
4. Potassium chloride solution, 0.2 M
5. Potassium chloride solution, 2.0 M
6. Nitric acid, concentrated
7. Perchloric acid, 70%
8. Gelatin solution, aqueous, 1%
9. Nitrogen gas, oxygen free
10. Hydroxylamine hydrochloride
11. Hydrochloric acid, 1:3
12. Potassium hydroxide, 25%

Theory

Lead, cadmium and zinc are frequently associated with one another in the industrial environment. Atmospheric dust and fume samples, settled dusts, ores and other bulk materials can be analyzed polarographically, after appropriate chemical treatment, to provide quantitative data on these three elements. By the use of 0.2 M potassium chloride as supporting electrolytes, their half-wave potentials are separated sufficiently to permit accurate analysis.

Procedure

Electrostatic precipitator tube samples. Transfer the sample to a 125 ml Phillips beaker (borosilicate) using a rubber policeman with alcohol. Evaporate to dryness on a steam bath. Add 2 ml nitric acid, wetting the sample thoroughly. Add 6 drops perchloric acid and swirl to mix. Evaporate to dryness on a hotplate at 350 - 400 °C. Repeat the acid treatment to obtain complete digestion. Cool and add 10 ml of the 0.2 M potassium chloride solution. Loosen solids with the policeman and rinse policeman and beaker walls with 2-3 ml of the 0.2 M potassium chloride solution from a wash bottle. Cover with a watch glass and boil 2 - 3 minutes. Filter the solution through Whatman #42 paper and wash the filter with 0.2 M potassium chloride; collect the filtrate and washings in a 25 ml volumetric flask. Dilute to volume with the same potassium chloride reagent, which serves as the supporting electrolyte in this method.

Place a sufficient quantity of 1% gelatin solution in the electrolysis vessel to yield a 0.01% final solution. Transfer the desired aliquot to this vessel and remove dissolved oxygen by passing nitrogen through the solution for 10 minutes. Record the polarogram in the 0.0 to -1.5 volt region.

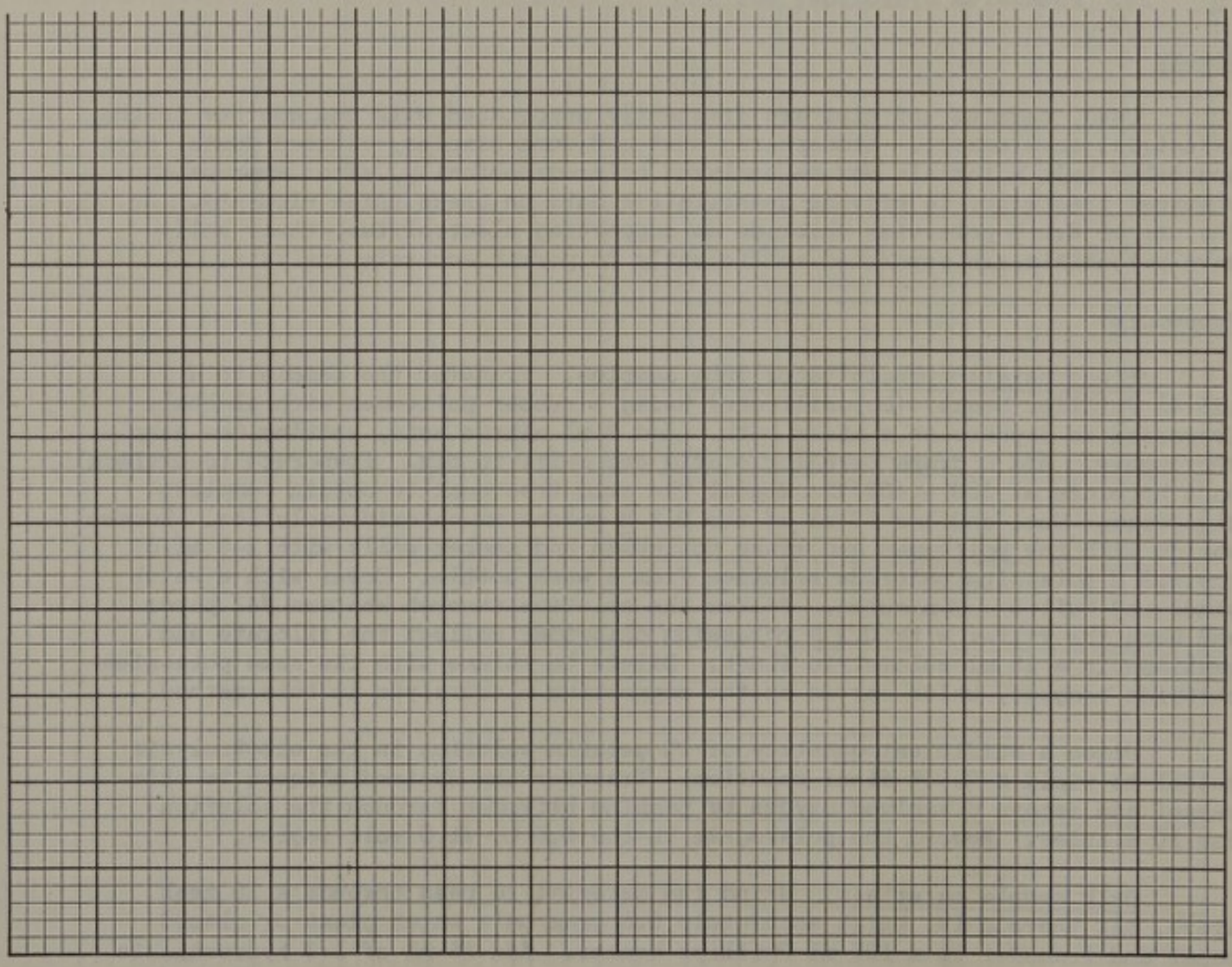
Bulk sample materials. Grind the material to pass through a 20 mesh sieve, mix thoroughly, weigh accurately 0.5 gram on a watch glass or glazed paper and transfer to a 125 ml Phillips beaker. Add 25 ml of 1:3 hydrochloric

acid and boil for 5 minutes to remove hydrogen sulfide. Add 2 ml of nitric acid and evaporate to dryness. Cool, add 20 ml of 1:6 hydrochloric acid and approximately 0.25 gram of hydroxylamine hydrochloride and boil to reduce iron and copper. Filter through Whatman #42 paper and wash the filter with distilled water; collect the filtrate and washings in a 100 ml volumetric flask. Add 10 ml of 2.0 M potassium chloride solution and sufficient potassium hydroxide to make the solution weakly acidic with no precipitation occurring. Dilute to volume.

To 1 ml of the sample solution add 9 ml of 0.2 M potassium chloride and 0.1 ml of 1% gelatin solution. Pass nitrogen through the solution for 10 minutes. Prepare a polarogram in the 0.0 to -1.5 volt region.

Calculations

Determine the diffusion current for each step of the polarogram by measuring the step height in millimeters and multiply this value by the microampere per millimeter factor for the particular sensitivity setting employed. Estimate the amount of each element from standard curves established previously with known quantities of the elements. Calculate the quantity of each element in the total sample by multiplying the above values by the aliquot reciprocal value.



Laboratory Exercise - DETERMINATION OF APPROXIMATE COMPOSITION OF SOLVENTS.

A number of individual experiments are given below. The experiments need not be carried out in any special order. However, test 6 requires one hour for color development and should be started early.

## Test 1. Preliminary Miscibility Tests

### Object

To determine the approximate composition of a solvent mixture.

### Theory

In order for solubility to occur the intermolecular forces between the solvent and the solute must be greater than the intermolecular forces between the solute molecules. The nature of these forces depends upon the compounds involved and are influenced by various functional groups.

### Apparatus and Reagents

1. Graduated cylinders, glass stoppered, 25 ml
2. Pipets
3. Hydrochloric acid, concentrated (or 4:1 sulfuric acid)
4. Dimethyl sulfate

### Procedure

1. Miscibility in water and specific gravity: Pipet 10 ml of solvent into a dry, weighed graduate. Calculate the specific gravity from the gain in weight. Add 10 ml $H_2O$ and mix gently. Complete miscibility excludes saturated aliphatic hydrocarbons, aromatic hydrocarbons, halogenated derivatives of these hydrocarbons, organic acids, weakly acidic compounds such as phenols, enols, primary and secondary nitro compounds, oximes, amino acids and neutral compounds.

2. Miscibility in concentrated hydrochloric acid (or 4:1 sulfuric acid): Add 10 ml of solvent to 10 ml of acid contained in another clean cylinder. Invert several times, let settle and note volumes of layers. Alcohols, esters, aldehydes, ketones and unsaturated compounds, anhydrides, ethers and quinones dissolve in these acids. Inert compounds, such as saturated aliphatic and aromatic hydrocarbons and their halogenated derivatives do not dissolve in these acids.

3. Solubility in dimethyl sulfate (CAUTION - KEEP REAGENT OFF SKIN. USE RUBBER BULB FOR PIPETING.): Pipet 10 ml of the sample into a stoppered cylinder containing 10 ml of dimethyl sulfate. Invert several times and observe volume change of oil layer to estimate approximate aromatic content. If aromatics exceed 25%, accurately dilute the sample with petroleum naphtha and repeat the test as, otherwise, erroneously high results may be obtained.

### Calculations

Percent miscibility equals 100 x (original solvent volume minus final solvent volume)/ original solvent volume.

## Test 2. Beilstein Test for Halogenated Hydrocarbons

### Object

To conduct a qualitative test for halogenated hydrocarbons.

### Theory

The action of halogenated hydrocarbons on copper wire in a flame yields copper halide which is volatile and produces a green flame.

### Apparatus

A small loop of copper wire, preferably supported in a glass handle set into a stopper. This assembly can be protected from contamination by keeping in a test tube when not in use.

### Procedure

Make a small loop of copper wire and heat in the edge of a gas flame until flame is no longer colored. Caution: Do not melt the wire in the hotter portions of the flame. Cool wire and dip loop in a little of original solvent mixture and then heat in edge of flame. Green flame indicates a halogen.

Confirmatory test - $AgNO_3$

About 2 ml of the original solvent or separated fraction is acidified with dilute $HNO_3$ and boiled gently for a few minutes to expel any HCN or $H_2S$. Add a few drops of $AgNO_3$. A heavy precipitate indicates a halogen.

## Test 3. Optical Properties of Hydrocarbon Mixtures

### Object

To determine aromatic content of a hydrocarbon mixture by optical measurements.

### Theory

The index of refraction and dispersion of aromatics are generally much higher than those of paraffin hydrocarbons. The relationship between the composition of mixtures of such solvents and these properties will be determined.

### Apparatus and Reagents

1. Abbe refractometer
2. Pure benzene
3. Pure isooctane
4. Standard 20%, 40%, 60%, and 80% mixtures (by volume), and unknown mixtures of benzene and isooctane.

Procedure

Determine the optical index of refraction and dispersion of the known solvents and the unknown samples. Compute the specific dispersion using the nomograph provided with the refractometer and the density values given for the samples.

Plot standard curves relating index of refraction and specific dispersion to volume percent composition. Compare experimental values with the following literature values for the pure substances.

Values at 25°C. from National Bureau of Standards

| Substance | Index of refraction ($N_D$) | Specific Dispersion $10^4(N_F - N_C) / d$ |
|---|---|---|
| Isooctane | 1,38898 | 100.5 |
| Benzene | 1.49790 | 189.6 |

Compute the composition of the unknown samples on the basis of (a) index of refraction and (b) specific dispersion using these curves.

## Test 4. Test for Methyl Ketones

Object

To determine the presence of methyl ketones ($RCOCH_3$).

Theory

The mechanism of this color reaction is unknown.

Apparatus and Reagents

1. Porcelain crucibles
2. Acetone
3. Unknown solvent
4. 5% sodium nitroprusside
5. 30% sodium hydroxide.

Procedure

Add a drop of 5% sodium nitroprusside and a drop of 30% sodium hydroxide to a drop of aqueous or alcoholic test solution in a crucible. After a short time, when a slight color usually develops, add 1 or 2 drops of glacial acetic acid. A red or blue color indicates the presence of a methyl ketone.

## Test 5. Test for Esters

Object

To conduct a qualitative test for esters ($RCOOR'$).

Theory

Esters of carboxylic acids can be converted to hydroxamic acids on treatment with hydroxylamine hydrochloride in the presence of alkali: $RCOOR' + NH_2OH \longrightarrow RCO(NHOH) + R'OH$. These acids form colored, inner complex, trivalent iron salts with ferric chloride.

Apparatus and Reagents

1. Saturated alcoholic solution of hydroxylamine hydrochloride
2. Saturated alcoholic solution of potassium hydroxide
3. Amyl acetate test solution
4. 0.5 N hydrochloric acid
5. 1% ferric chloride
6. Crucibles
7. Burner

Procedure

To a drop of the dry test solution in a crucible, add one drop of the hydroxylamine hydrochloride solution and one drop of the potassium hydroxide solution. Heat over a microflame until the reaction begins, observed as a slight bubbling.

After cooling, acidify with 0.5N hydrochloric acid and add a drop of 1% ferric chloride. An intense violet color indicates an ester.

## Test 6. Determination of Methanol

Object

To determine the quantity of methanol in an aqueous mixture.

Theory

Methanol is a very poisonous component of some solvents. With potassium permanganate it is oxidized to formaldehyde which gives a test with Schiff's reagent.

Apparatus and Reagents

1. Potassium permanganate reagent (3 grams $KMnO_4$ and 15 ml 85% $H_3PO_4$ diluted to 100 ml)
2. Oxalic acid reagent (5 grams oxalic acid in 100 ml of 1:1 $H_2SO_4$)
3. Graduated cylinders, stoppered, 25 ml
4. Pipets
5. Modified Schiff's reagent: to 0.2 gram of fuchsin dissolved in 120 ml of hot water add a solution of 2 grams of sodium sulfite in 20 ml of water and mix. Add 2 ml of hydrochloric acid, dilute to 200 ml with water and store in a well-stoppered bottle in the refrigerator. Residual color may be removed if necessary by bubbling sulfur dioxide through the reagent.

Procedure

To 5 ml of aqueous sample in a stoppered cylinder add 0.25 ml ethyl alcohol, mix and add 2 ml of potassium permanganate reagent. Allow to stand 10 minutes, then decolorize with 2 ml of oxalic acid reagent. Add 5 ml of modified Schiff's reagent, mix, and allow to stand one hour before comparison with standards (containing 0.05 to 0.3 mg methanol) prepared at the same time and in the same way as the unknown sample.

## Test 7. Fractionation of Hydrocarbon-Chlorinated Hydrocarbon Mixtures

Object

To determine the composition of a mixture of hydrocarbons and chlorinated hydrocarbons by fractional distillation, density and refractive index measurements.

Theory

By means of a fractionating column the various components of a mixture may be separated according to their differences in boiling points. A step-type plot is obtained when the top column temperature is plotted against the volume distilled. If the distillate corresponding to the horizontal portion of each step is collected in a separate container the pure components may be identified by boiling point, index of refraction, specific gravity and dispersion measurements, or by other confirmatory tests.

Apparatus and Reagents

1. Fractionating column, which will be demonstrated only.
2. Graduated cylinders, containing various cuts of distillate, provided with the boiling point curve and specific gravity data.
3. Abbe refractometer.

Procedure

Because the fractionation operation is time consuming, this portion of the analysis will be carried on as a demonstration. The final results will be available in the form of a distillation plot together with cylinders containing the various cuts, with the weights, volumes, index of refraction and optical dispersion for each cut.

Calculations

Identify the composition of each cut by comparing the known properties with those given in an available table of physical constants. If the cut appears to contain more than one component, the approximate composition may be computed by interpolation. Calculate the composition of the solvent on a weight basis, using the weights given in the distillation data.

Laboratory Exercise - SAMPLING AND ANALYSIS OF SULFUR DIOXIDE.

Object

To determine the concentration of sulfur dioxide in a synthetic atmosphere using a colorimetric procedure.

Apparatus and Reagents

1. Midget impinger pump
2. Midget impinger flask
3. Collecting medium: 0.05 N sodium hydroxide solution with 2%, by volume, of glycerol added.
4. Volumetric flasks
5. Pipets
6. Standard bisulfite solution: A weighed portion of sodium meta bisulfite ($Na_2S_2O_5$) is diluted with collecting medium to give a calculated $SO_2$ content of about 100 micrograms per ml. This stock solution may be further standardized by titrating with 0.01 N iodine solution. Standard solutions A, B and C, containing 3, 6 and 9 micrograms of $SO_2$ per ml, respectively, are prepared from the stock solution by dilution with the collecting medium.
7. Color reagent:
   Solution A -- Place in a flask 460 ml of distilled $H_2O$, 44 ml conc. $H_2SO_4$ and 16 ml of a 3% solution of basic fuchsin in ethyl alcohol. (Use best grade analytical reagents.) Shake vigorously and add 280 ml distilled $H_2O$. Place in a glass stoppered, 1-liter bottle and add 4 grams of Norit A activated carbon. Shake for several minutes and store in a refrigerator overnight. Then filter twice through Whatman #42 or equivalent filter paper. Filtered solution should be light straw colored (the amount of Norit A is critical). Store in refrigerator.
   Solution B -- Dilute 5 ml of 40% formaldehyde to 100 ml with distilled $H_2O$. Store in refrigerator. The color reagent is prepared by mixing 1 part by volume of Solution B with 10 parts of Solution A. The reagent is ready for use 30 minutes after mixing.
8. Matched colorimeter tubes
9. Klett colorimeter

Theory

The color reagent used for the determination of sulfur dioxide is a solution of basic fuchsin and formaldehyde in sulfuric acid. The reaction is the same as the one in Schiff's test for aldehydes in organic chemistry. Schiff's reagent contains basic fuchsin which has been decolorized with sulfur dioxide. When this reagent is added to a solution of formaldehyde or other aldehydes a pink color soon appears. In the colorimetric determination of sulfur dioxide, solutions of basic fuchsin and formaldehyde are mixed to give the color reagent and when sulfur dioxide is added, the pink color results. The intensity of the pink color is proportional to the concentration of sulfur dioxide in the sample.

This procedure for sulfur dioxide is very useful for samples taken in air pollution studies where the concentration of sulfur dioxide may range from 0.01 to 0.5 ppm. Other methods, based on titrimetric procedures, are available for plant studies where sulfur dioxide concentrations may range from 5 - 20 ppm. A polarographic method which is recommended[1] when the presence of nitrogen oxides is suspected, has the wide working range of 3 micrograms to several milligrams of $SO_2$ per ml of collecting medium.

## Procedure

Sulfur dioxide gas has been added to the air in the large metal chamber. Collect a 10-minute sample, timed accurately, using a midget impinger containing 10 ml of collecting medium. Place the sample in a 25 ml volumetric flask, rinse the impinger and make up to volume with the collecting medium. Prepare the tubes for the colorimetric determination as follows:

| Tube No. | Sample | Preparation |
|---|---|---|
| 1. | Blank | 11 ml collecting medium |
| 2. | Air Sample | 1 to 5 ml sample solution + collecting medium to final volume of 11 ml |
| 3. | Air Sample | Prepared same as No. 2 |
| 4. | Standard No. 1 | 1 ml solution A + 10 ml collecting medium |
| 5. | Standard No. 2 | 1 ml solution B + 10 ml collecting medium |
| 6. | Standard No. 3 | 1 ml solution C + 10 ml collecting medium |

Add 5 ml color reagent to the solution in the tubes and mix thoroughly. After 12 minutes read the absorbance of the solutions in the Klett colorimeter using the blank (#1 tube) to zero the instrument.

Plot the standard curve results on graph paper with absorbance vs. concentration $SO_2$. The amount of $SO_2$ in the sample aliquot may then be read from the curve.

## Calculations

Use the following equations to calculate the concentration of $SO_2$ in the chamber.

$$\text{mg } SO_2/\text{sample} = \frac{\gamma\, SO_2 \times \text{aliquot reciprocal}^*}{1000}$$

$$\text{ppm } SO_2/\text{chamber} = \frac{\text{mg } SO_2 \times 24{,}450}{\text{liters of air sampled} \times 64 \text{ (Mol. Wt. } SO_2)}$$

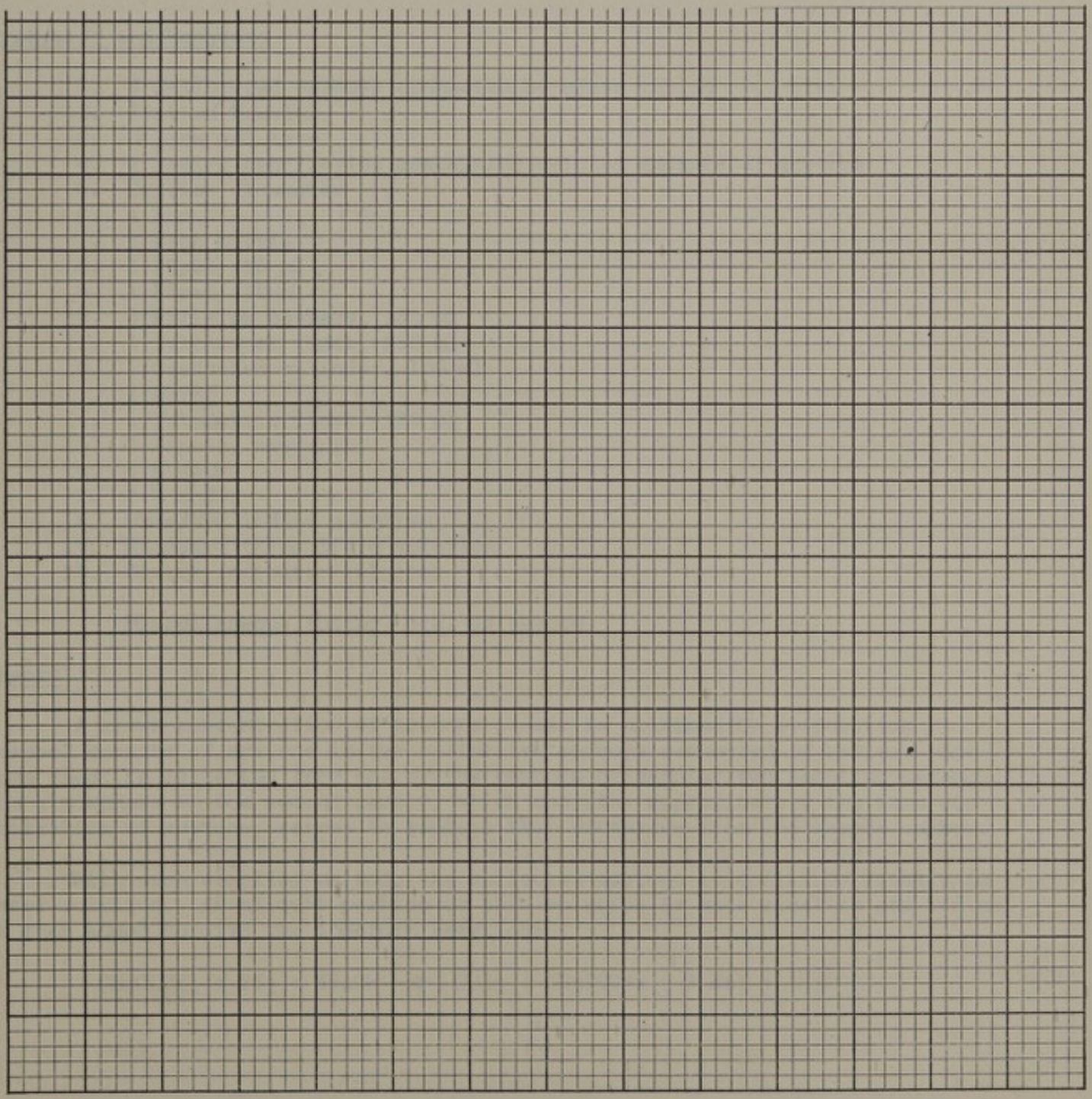
(1) Method for the Determination of Sulfur Dioxide in Air, Committee on Recommended Analytical Methods, American Conference of Governmental Industrial Hygienists.

$$\text{Aliquot reciprocal} = \frac{25}{\text{ml sample solution analyzed}}$$

Example. A midget impinger sample is collected for 10 minutes at a rate of 2.83 liters/min. and 1 ml of the sample is used in the colorimetric test. From the standard curve, the absorbance reading corresponds to 4 $\gamma SO_2$.

$$\frac{\text{mg } SO_2}{\text{sample}} = \frac{4 \times 25}{1000} = 0.1$$

$$\frac{\text{ppm } SO_2}{\text{chamber}} = \frac{0.1 \times 24{,}450}{28.3 \times 64} = \frac{0.1 \times 382}{28.3} = 1.35$$



Laboratory Exercise - SAMPLING AND DETERMINATION OF METHYL KETONES.

Object

1. To demonstrate a simple method for laboratory preparation of known concentrations of vapor in air.
2. To demonstrate and compare two common methods of collection of vapors.

   a. Fritted bubbler
   b. Silica gel tube

3. To demonstrate a general method for analysis of methyl ketones.

Apparatus and Reagents

1. 2 Calibrated carboys connected by glass tubing
2. Fritted bubbler
3. Silica gel tube
4. 2 Sealed capillaries containing weighed amounts of acetone
5. Orifice flowmeter
6. 1 Rod
7. 1 Wash bottle
8. 1 Pipet, 25 ml
9. 1 Pipet, 20 ml
10. 6 Phillips beakers, 250 ml
11. 1 Erlenmeyer flask, 250 ml
12. 1 Graduated cylinder, 100 ml
13. 1 Buret
14. 1 Volumetric flask, 100 ml
15. 3 Pipets, measuring
16. 5 $\underline{N}$ NaOH
17. 6 $\underline{N}$ $H_2SO_4$
18. Standard iodine solution
19. Standard thiosulfate solution
20. Starch indicator solution

Miscellaneous tubing, etc. as needed.

Theory

A known concentration of acetone in air is obtained by evaporating weighed amounts of acetone in two calibrated carboys connected in series. Acetone vapor is collected from one of the carboys by each of two sampling methods. The atmosphere in the second carboy, which contains approximately the same concentration of acetone, serves to replace that withdrawn from the sample carboy.

With each sampling method, the collected acetone is contained finally in water solution. An aliquot portion of this solution is made alkaline and iodine is then added to give the iodoform reaction:

$$CH_3COCH_3 + 3I_2 + 4NaOH \longrightarrow CHI_3 + CH_3COONa + 3\ NaI + 3H_2O$$

A measured amount of standard iodine is added and the excess is back titrated with standard sodium thiosulfate solution, using starch as indicator. The amount of iodine consumed by the iodoform reaction is thus obtained by difference. This value is used to calculate the quantity of acetone in the sample solution and the concentration of this methyl ketone in the air sample.

Procedure

Place one capillary of acetone in each carboy. Record the weight of acetone and the volume of each carboy. Crush the capillary with the glass rod (gently, to avoid cracking the bottom of the carboy), then close the carboys with the stoppers provided with the connecting and outlet tubes. Allow 15 minutes to permit evaporation and mixing. (A longer period is desirable but the experimental period is limited.)

While waiting, calculate the theoretical concentration (ppm by volume) of acetone vapor in each carboy. The two values should be approximately the same. It is not necessary that they be exactly the same.

Connect the silica gel tube between the sample carboy and the flowmeter. Connect the flowmeter to the vacuum source. Open the screw clamp, adjust airflow to 1 liter per minute and sample for exactly 8 minutes. Remove the silica gel tube and transfer the silica gel to an Erlenmeyer flask containing a measured 100 ml volume of distilled water. Stopper the flask and allow to stand for 30 minutes with occasional agitation (a one hour standing period is recommended for samples collected under other circumstances).

While the silica gel is standing in water, collect another sample from the same carboy using the fritted bubbler. Add distilled water to about 1 inch above the fritted disk of the bubbler. Connect between the carboy and the flowmeter. (Be sure that air sample will enter through the fritted disk and exit at the top.) Apply vacuum and adjust flow to 1 liter per minute. Again sample for exactly 8 minutes. Disconnect the bubbler and transfer the sample to a 100 ml volumetric flask. Rinse bubbler with distilled water and use rinsings to bring the volume of the sample to 100 ml. Note time and set aside for analysis.

Determine the titre of the standard iodine solution versus the standard thiosulfate solution. Pipet 20 ml of $I_2$ solution into a Phillips beaker. Add 30 ml of water and 2 ml of 6 $\underline{N}$ $H_2SO_4$. Titrate with thiosulfate solution to a faint yellow, then add 1 ml of starch indicator and continue titration to the disappearance of the blue color. (Color should not re-appear in 30 seconds.) Record the volume of thiosulfate required to titrate 20 ml of the iodine solution.

In analyzing the samples collected, each student should titrate a portion of each aqueous solution as follows:

Take the sample collected by the fritted bubbler and pipet a 25 ml aliquot into a Phillips beaker. Add 4 ml of 5 $\underline{N}$ NaOH, 25 ml of water and exactly 20 ml of iodine solution (by same pipet as used before). Mix and let stand for 15 minutes. Then add 5 ml of 6 $\underline{N}$ $H_2SO_4$ and titrate with thiosulfate solution, using starch indicator. Record the volume of thiosulfate solution required to titrate the excess iodine.

Transfer a 25 ml aliquot of the supernatant solution from the silica sample by means of a pipet to a Phillips beaker. Add 4 ml of 5 N NaOH, 25 ml of water and 20 ml of iodine solution. Allow to stand for 15 minutes. Add 5 ml of 6 N $H_2SO_4$ and titrate the excess iodine with the thiosulfate solution as described previously. Record the volume required.

Data

Carboy No. 1
- Total volume ..... _____ liters
- Wt of acetone ..... _____ grams

Carboy No. 2
- Total volume ..... _____ liters
- Wt of acetone ..... _____ grams

Titre of sodium thiosulfate solution
- Volume of thiosulfate equivalent to 20 ml of 0.1N iodine solution .... _____ ml (a)

Fritted bubbler sample
- Rate of air flow ..... _____ liter/minute
- Sampling period ..... _____ minutes
- Aliquot portion ..... _____
- Volume thiosulfate required for excess 0.1N iodine solution ..... _____ ml (b)

Silica gel sample
- Rate of air flow ..... _____ liter/minute
- Sampling period ..... _____ minutes
- Aliquot portion ..... _____
- Volume thiosulfate required for excess 0.1N iodine solution ..... _____ ml (c)

Calculations

1. Sodium thiosulfate titre

$$1 \text{ ml thio} \rightleftharpoons \frac{20.00 \text{ ml iodine}}{\text{___ ml thio (a)}} \quad \text{or} \quad \text{_____ ml } 0.1\underline{N}\ I_2 \text{ (d)}$$

2. Milligrams of acetone per liter of air

Fritted bubbler sample:

20.00 ml $I_2$- ____ (d) ( ___ (b) ml thio) =

____ (e) ml $0.1\underline{N}$ $I_2$ consumed by iodoform reaction

$$\frac{\text{____ (e) ml} \times 0.1\underline{N} \times 9.68 \text{ (eq. wt.)} \times 4 \text{ (aliq. recip.)}}{1 \times 8 \text{ (rate x samp. period)}}$$

____ (f) mg/l

Silica gel sample:

20.00 ml $I_2$- ____ (d) ( ____ (c) ml thio) =

____ (g) ml $0.1\underline{N}$ $I_2$ consumed by iodoform reaction

$$\frac{\text{____ (g) ml} \times 0.1\underline{N} \times 9.68 \times 4}{1 \times 8} = \text{____ (h) mg/l}$$

3. Concentration of acetone, expressed as parts per million

Fritted bubbler:

$$\frac{\text{____ (f) mg/l} \times 24{,}450}{58.08 \text{ (mol. wt.)}} = \text{____ ppm}$$

Silica gel:

$$\frac{\text{____ (h) mg/l} \times 24{,}450}{58.08} = \text{____ ppm}$$

Laboratory Exercise - NOISE MEASUREMENT AND ANALYSIS

Object

To make octave band analysis of a continuous noise.

Theory

Variations in air pressure are detected by the microphone and produce an electrical current which is fed into the sound level meter, which consists of an attenuator, an amplifier, weighting networks, and a meter. The calibrated attenuator in the circuit controls the current within limits which can be handled by the meter, calibrated in decibels. Meter readings are the root-mean-square sound-pressure levels expressed in decibels re 0.0002 dynes per sq. cm. Output from the sound-level meter is fed into the octave band analyzer, which consists of bandpass filters with a switch for selection of bands, an attenuator, an amplifier, and a meter. The attenuator, which is in 10 decibel steps, and the amplifier serve the same function as in the sound-level meter - to keep the voltage input within a range which can be handled by the meter. Range of this instrument is 16 decibels; the values obtained are the sum of the sound-level meter attenuator reading, the analyzer attenuation, and the analyzer meter reading.

Procedure

Connect and turn on all equipment, allowing sufficient warm-up time. Check the batteries on the sound level meter and octave-band analyzer. Make an acoustical calibration of both instruments.

Measure the overall sound pressure level and octave-band levels of the noise sources.

Record and plot data.

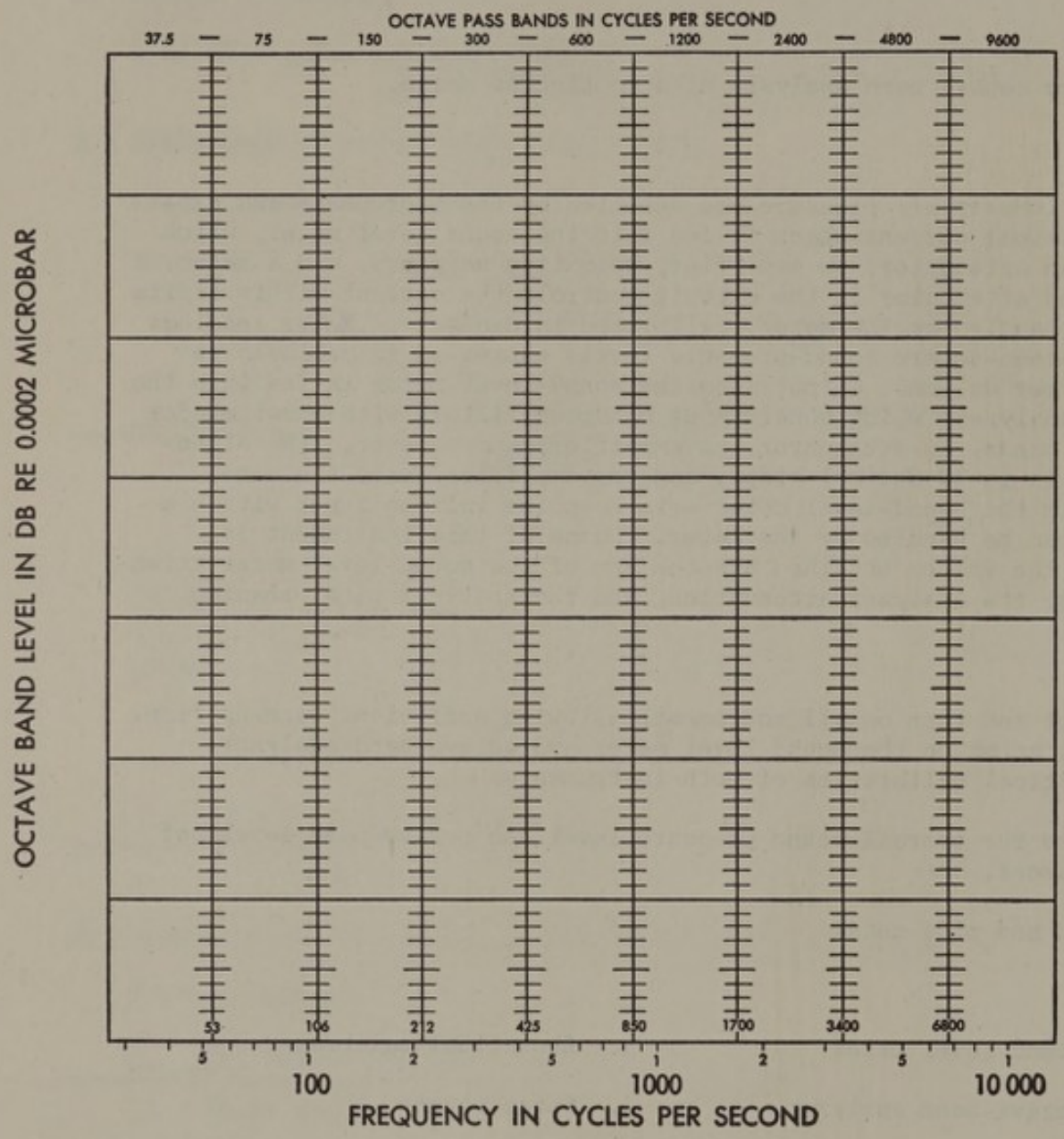
Equipment

1. Sound level meter
2. Octave-band analyzer
3. Acoustical calibrator
4. Noise source

Data

Sound Pressure Levels
re 0.0002 dynes/sq. cm.

| Over-all | OCTAVE BANDS (cps) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 20<br>75 | 75<br>150 | 150<br>300 | 300<br>600 | 600<br>1200 | 1200<br>2400 | 2400<br>4800 | 4800<br>9600 |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |


OCTAVE PASS BANDS IN CYCLES PER SECOND
37.5
75
150
300
600
1200
2400
4800
9600
OCTAVE BAND LEVEL IN DB RE 0.0002 MICROBAR
53
106
212
425
850
1700
3400
6800
5
1
2
5
1
2
5
1
100
1000
10 000
FREQUENCY IN CYCLES PER SECOND

Laboratory Exercise - DETERMINATION OF AVERAGE AIR VELOCITY IN A DUCT.

Object

To become familiar with the method of making a traverse of a duct using a Pitot tube or Velometer.

Theory

The velocity pressure of air flowing in a duct is: $vp = \left(\frac{V}{4000}\right)^2$, or the velocity may be calculated from $V = 4000 \sqrt{vp}$. The Pitot tube measures the total pressure, which includes the velocity pressure and the static pressure, and the static pressure. Thus if the total pressure tap on the Pitot tube is connected to one leg of a manometer and the static pressure tap is connected to the other leg of the manometer, the resulting manometer reading is the velocity pressure (total pressure = velocity pressure + static pressure, and velocity pressure + static pressure - static pressure = velocity pressure).

By dividing a duct into five concentric circles, each of which represents an equal area, and measuring the velocity pressure at points 90° apart on these circles, the average velocity can be calculated. A Pitot tube used with an accurate inclined manometer will give very precise velocity pressure readings.

Other devices, such as the Velometer and its attachments, can be used to determine the air velocity in a duct; however, accurate results will depend upon the instrument calibration.

Procedure

Make a 10 point vertical traverse and a 10 point horizontal traverse using the Pitot tube and also the Velometer. Determine average velocity and compute the flow. DO NOT AVERAGE VELOCITY PRESSURES.

Apparatus

1. Pitot tube
2. Velometer
3. Manometer

Data

Pitot Traverse (Ind. Vent. p. 9-4)

| Reading Point Inches | Vertical h" $H_2O$ | Velocity fpm | Horizontal h" $H_2O$ | Velocity fpm |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |
| 10 | | | | |

Average velocity = Volume = _____ cfm

## Velometer Traverse

| Reading Point Inches | Vertical Velocity fpm | Horizontal Velocity fpm |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |

Average Velocity =

Volume = _____cfm

$\overline{V}$ Velocity = _____fpm

$\frac{Vav}{V\overline{V}}$ = _____

Calculations

Laboratory Exercise - MEASUREMENT OF PRESSURE LOSSES IN DUCTWORK.

Object

To become familiar with the various head losses in a duct system, and to observe good and bad practices in duct design.

Theory

The various losses in a duct system are discussed elsewhere and in Chapter VI of the manual on Industrial Ventilation.

Procedure

1. Loss in straight duct section:

Measure the pressure drop between two taps located a measured distance apart in the straight duct. Calculate the pressure loss per 100 feet.

2. Loss in an elbow: $\frac{R}{D} = 0.75$

Measure the pressure drop between two taps on either side of the elbow. Using the pressure drop per 100 feet previously determined, subtract the drop caused by friction in the straight duct. The difference is the elbow loss.

3. Loss in a tee:

Measure the pressure drop across the tee from both upstream points. Estimate the friction loss in straight duct in both cases. The differences are the losses in both branches.

4. Loss in an enlargement:

Measure the pressure difference across the enlargement.

$$\text{Regain} = \frac{SP_2 - SP_1}{VP_1 - VP_2}$$

$$\text{or loss} = (1 - R)(VP_1 - VP_2)$$

5. Compare values found with those in the manual.

Equipment

Manometer or magnehelic gauge

Data

Straight Duct (8" diam.)

| $\Delta$ p (inches $H_2O$) | Distance (ft.) | Flow (cfm) | $\Delta$p/100 ft. | |
|---|---|---|---|---|
| | | | obs. | chart |
| | | | | |

Elbow

| Total $\Delta$ p (inches) | St. Duct (ft.) | $\Delta$p/100 ft. | Duct loss | Elbow loss | |
|---|---|---|---|---|---|
| | | | | inches | % of v.p. |
| | | | | | |

Tee

| Right angle branch | | | | Straight branch | | | |
|---|---|---|---|---|---|---|---|
| $\Delta$ p | Duct loss (inches) | Tee loss | | $\Delta$p | Duct loss (inches) | Tee loss | |
| | | inches | % of branch v.p. | | | inches | % of branch v.p. |
| | | | | | | | |

Enlargement

| Static pressure | | Velocity pressure | | Regain | Loss |
|---|---|---|---|---|---|
| (downstream) | (upstream) | (upstream) | (downstream) | | |
| | | | | | |

Laboratory Exercise - EFFECT OF HOOD FLANGES ON COEFFICIENT OF ENTRY.

Object

To demonstrate the changes in coefficient of entry ($C_e$) and hood entry loss ($h_e$) caused by flanging a simple pipe end, and to become familiar with the relationships between these quantities. To also note the difference in control with and without the flange.

Theory

For an opening with no entry loss, such as a well-rounded bell mouth, the velocity immediately downstream from the hood is given by the equation $V = 4000 \sqrt{h_s}$, where $h_s$ is the hood static pressure in inches of water and V is velocity in ft/min. Most hoods have an entry loss, however, so that a coefficient of entry ($C_e$) must be used, and $V = 4000\, C_e \sqrt{h_s}$. If the average velocity in the pipe is determined by a Pitot traverse, and the static suction is measured with a manometer, the coefficient of entry can be calculated.

$$C_e = \frac{V}{4000\sqrt{h_s}} = \frac{\sqrt{v.p.}}{\sqrt{h_s}}$$ where v.p. is the velocity

pressure determined by Pitot tube. The hood entry loss ($h_e$) is that part of the hood static suction which is not converted into velocity pressure. Thus:

$$C_e = \frac{\sqrt{v.p.}}{\sqrt{v.p. + h_e}} = \frac{\sqrt{VP}}{\sqrt{SP}}$$

$$C_e^2 (v.p. + h_e) = v.p.$$

$$h_e = \frac{v.p.\,(1-C_e)^2}{C_e^2}$$

Procedure

Airflow characteristics in front of hoods. Using a thermoanemometer, measure the air velocity along the centerline of the duct at distances of 0, 1/3, 2/3, 1, 2, and 3 diameters from the hood with and without the flange.

| Distance from hood (pipe diameters) | Air velocity (feet/min) | |
|---|---|---|
| | Unflanged | Flanged |
| 0 | | |
| 1/3 | | |
| 2/3 | | |
| 1 | | |
| 2 | | |
| 3 | | |

Using a smoke tube, note the relative degree of control with and without a flange.

Coefficient of entry and hood entry loss. 1. Make the following measurements with flange off:

a. Centerline velocity pressure in duct (Pitot tube)

centerline v.p. = _____" $H_2O$

b. Static pressure 1 - 3 diameters downstream from duct end.

s.p. = _____" $H_2O$

2. Repeat measurements of centerline v.p. and hood static suction with flange on.

centerline v.p. = _____" $H_2O$

s.p. = _____" $H_2O$

3. Remembering that $V = 4000 \sqrt{v.p.}$, and $V_{av.} = 0.9 V_{\text{¢}}$. calculate flow in the 6" duct with flange on and flange off.

4. Assuming that v.p. av. = 0.81 v.p.¢, calculate $C_e$ and $h_e$ for the duct end with and without flange.

Equipment

1. Pitot tube
2. Manometer
3. Smoke tubes
4. Yardstick or tape
5. Thermoanemometer

Calculations

Laboratory Exercise - MEASUREMENT OF SLOT AND SURFACE VELOCITY AND ENTRANCE LOSS.

## Object

To become familiar with the meaning of "entry loss," "static suction," "velocity pressure" by measuring or calculating these values; to note the method of estimating entry loss for a hood; and to compare the measured and estimated values.

## Theory

Whether a well-designed hood controls a contaminant is dependent primarily on the quantity of air exhausted. It is often difficult to measure directly the quantity of air flowing through a hood after installation. If the entry loss of a hood is known, the air flow can be estimated by measuring the "hood suction."

The hood suction is the static pressure immediately (1 - 3 pipe diameters) downstream from the hood. It is the sum (ignoring sign) of the velocity pressure (v.p.) and the entry loss ($h_e$). Velocity pressure can be determined by the formula $V = 4005 \sqrt{v.p.}$ or $v.p. = \left(\frac{V}{4005}\right)^2$ where V is velocity in feet per minute and v.p. is pressure in inches of water. In this experiment we find the air flow (Q) by other means and use the relationship $V = \frac{Q}{A}$ to obtain velocity.

Hood entry loss is the sum of the individual losses through the hood. For a slotted tank it may be considered as the sum of the loss through a sharp-edged orifice and an entry with an angle of 90°. The formula given for entry loss under these conditions is: $h_e = 1.3$ (slot v.p.) + 0.25 (duct - v.p.). The particular tank used in this experiment has an entry loss less than predicted by the formula, probably because the take-off from the slot is more favorable than in most industrial installations.

## Procedure

1. Using a velometer or thermoanemometer adjust slot opening so that slot velocity is 2000 fpm. Measure slot area and estimate airflow through slot.

2. Using thermoanemometer and smoke tube check surface velocities and effectiveness of control.

3. Using measurements of total airflow and airflow through the other branch made by groups one and three respectively, determine airflow through tank hood by difference. Calculate velocity pressure.

4. Measure static suction one diameter downstream from hood.

5. Calculate coefficient of entry, $C_e$ and hood entry loss, $h_e$, using formulae on p. 4-6, 7, Industrial Ventilation.

6. Calculate the expected entry loss and coefficient of entry from the formula under Theory and the relationships on p. 4-6 of the Industrial Ventilation. Compare with actual values.

Equipment

1. Velometer or thermoanemometer
2. Manometer or magnehelic gauge
3. Smoke tubes

Data

Slot area =

Est. airflow = 2000 x slot area =

Airflow

| Total | Through 6" duct | Through hood |
|---|---|---|
| | | |

Static suction =

$C_e = \sqrt{\frac{VP}{SP}}$ =

$h_e = \frac{(1 - C_e^2)}{(C_e)^2}$ VP =

Expected entry loss ($h_e$) = 1.3 (slot v.p.) + 0.25 (duct v.p.)

Expected $C_e = \sqrt{\frac{\text{duct v.p.}}{\text{v.p.} + h_e}}$

Calculations

Laboratory Exercise - DETERMINATION OF CHARACTERISTICS OF AIR JETS.

Object

To demonstrate some of the characteristics of discharge jets which are important in the field of comfort ventilation, mine ventilation, and push-pull systems.

Theory

As an air jet issues from a supply source, it is placed in contact with stationary air. The air at the edges of the jet entrains more and more of this stationary air by turbulent mixing, so that the velocity of the jet is constantly decreasing, while the area is increasing. The theory of free air jets is given briefly in Hemeon[1], using data from several sources. For a round jet the characteristics are described in terms of two phases. Using Vmax as the maximum (centerline) jet velocity at a distance X, and $V_o$ as the maximum outlet velocity:

For the first phase, which extends out 5 diameters,

(1) $\frac{V_{max}}{V_o}$ = 1.0 to 1.2

For distances greater than 5 diameters,

(2) $\frac{V_{max}}{V_o} = \frac{K}{X/D}$ Where X is distance and D is diameter of outlet

The table below gives values of K

| Vx(fpm) | Maximum outlet velocities (fpm) 1000-2000 | 3000-4000 | 5000 |
|---|---|---|---|
| | Values of K | | |
| 180 | 6.0 | 6.3 | 6.8 |
| 150 | 5.6 | 6.1 | 6.5 |
| 100 | 5.1 | 5.6 | 6.0 |
| 75 | 4.7 | 5.1 | 5.4 |
| 40 | 3.7 | 3.9 | 4.0 |

Note: $Vx = \frac{V_{max}}{2.7}$

An average K value of 6 may be used for approximations

Procedure

Using the vane anemometer and stopwatch, measure the centerline velocity at 0, 1, 2, 5, 10, 20, and 30 diameters from the outlet.

Equipment

1. Vane anemometer
2. Ringstand
3. Tape or yardstick
4. Stopwatch

Data

| Distance from outlet | | Velocity (fpm) | Vx / $V_{max}$ | K | $V_{max}$ |
|---|---|---|---|---|---|
| Feet | Diameters | Measured | 2.7 | | Calculated |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Calculations

References

1. Hemeon, W. C. L.: Plant and Process Ventilation. Industrial Press, New York, 1955 (p. 200-205)

2. McElroy, G. E.: Air Flow at Discharge of Fan-pipe Lines in Mines. U. S. Bur. Mines Rep. of Invest. 3730, Nov. 1943

3. Tuve, G. L. and Priester, G. B.: Control of Air Streams in Large Spaces, Heat. Pip. and Air Cond., ASHVE Journal Sect., Jan. 1944, p. 39

# Appendix

# CONVERSION FACTORS

## Linear Measure

| Meter (m) | Centimeter (cm) | Millimeter (mm) | Micron (μ) | Angstrom unit, A | (in.) | (ft.) |
|---|---|---|---|---|---|---|
| 1 | 100 | 1000 | $10^6$ | $10^{10}$ | 39.4 | 3.28 |
| .01 | 1 | 10 | $10^4$ | $10^8$ | .394 | .0328 |
| .001 | .1 | 1 | $10^3$ | $10^7$ | .0394 | .00328 |
| $10^{-6}$ | $10^{-4}$ | $10^{-3}$ | 1 | $10^4$ | $3.94 \times 10^{-5}$ | $3.28 \times 10^{-6}$ |
| 10-10 | $10^{-8}$ | $10^{-7}$ | $10^{-4}$ | 1 | $3.94 \times 10^{-9}$ | $3.28 \times 10^{-1}$ |
| 0.0254 | 2.540 | 25.40 | $2.54 \times 10^4$ | $2.54 \times 10^8$ | 1 | 0.0833 |
| 0.305 | 30.48 | 304.8 | 304,800 | $3.048 \times 10^9$ | 12 | 1 |
| $1.610 \times 10^3$ | $1.610 \times 10^5$ | $1.610 \times 10^6$ | $1.610 \times 10^9$ | $1.610 \times 10^{13}$ | 63,400 | 5,280 |

## Area

| $m^2$ | $In.^2$ | $Ft.^2$ | $cm^2$ | $mm^2$ |
|---|---|---|---|---|
| 1 | 1,550 | 10.76 | 10,000 | $10^6$ |
| $6.452 \times 10^{-4}$ | 1 | $6.94 \times 10^{-3}$ | 6.452 | 645.2 |
| 0.0929 | 144 | 1 | 929.0 | 92,903 |

## Volume

| $Ft.^3$ | Gallon | Liters | $cm^3$ | $m^3$ |
|---|---|---|---|---|
| 1 | 7.481 | 28.32 | 28,320 | 0.0283 |
| 0.1337 | 1 | 3.785 | 3,785 | $3.79 \times 10^{-3}$ |
| 0.03531 | 0.2642 | 1 | 1,000 | $1 \times 10^{-3}$ |
| $3.531 \times 10^{-5}$ | $2.64 \times 10^{-4}$ | 0.001 | 1 | $10^{-6}$ |
| 35.31 | 264.2 | 1000 | $10^6$ | 1 |

## Density

| $Gram/cm^3$ | $Lb/ft.^3$ | Lb/gal. |
|---|---|---|
| 1 | 62.43 | 8.345 |
| 0.01602 | 1 | 0.1337 |
| 0.1198 | 7.481 | 1 |

## Mass

| Gram (gm) | Kilogram (Kg) | Grains (gr) | Oz. avoir | Lb. avoir | Tons-short |
|---|---|---|---|---|---|
| 1 | 0.001 | 15.432 | 0.03527 | 0.00220 | $1.102 \times 10^{-6}$ |
| 1,000 | 1 | 15,432 | 35.27 | 2.205 | $1.102 \times 10^{-3}$ |
| 0.0648 | $6.480 \times 10^{-5}$ | 1 | $2.286 \times 10^{-3}$ | $1.429 \times 10^{-4}$ | $7.143 \times 10^{-8}$ |
| 28.35 | 0.02835 | 437.5 | 1 | 0.0625 | $3.125 \times 10^{-5}$ |
| 453.59 | 0.4536 | 7,000 | 16 | 1 | $5.00 \times 10^{-4}$ |
| 907,184 | 907.2 | $1.40 \times 10^7$ | 32,000 | 2,000 | 1 |

## Heat, Energy, or Work

| Joule=$10^7$ ergs. | Ft.-lb. | Kw.-hr. | Hp.-hr. | K-Cal. | Cal. | Btu |
|---|---|---|---|---|---|---|
| 1 | 0.7376 | $2.773\times10^{-7}$ | $3.725\times10^{-7}$ | $2.39\times10^{-4}$ | 0.2390 | $9.478\times10^{-4}$ |
| 1.356 | 1 | $3.766\times10^{-7}$ | $5.05\times10^{-7}$ | $3.24\times10^{-4}$ | 0.3241 | $1.285\times10^{-3}$ |
| $3.6\times10^6$ | $2.655\times10^6$ | 1 | 1.341 | 860.57 | 860,565 | 3,412 |
| $2.68\times10^6$ | $1.98\times10^6$ | 0.7455 | 1 | 641.62 | 641,615 | 2,545 |
| 4,184 | 3,086 | $1.162\times10^{-3}$ | $1.558\times10^{-3}$ | 1 | 1,000 | 3.9657 |
| 1,055 | 778.16 | $2.930\times10^{-4}$ | $3.93\times10^{-4}$ | 0.252 | 252 | 1 |
| 4.184 | 3.086 | $1.162\times10^{-6}$ | $1.558\times10^{-6}$ | 0.001 | 1 | 0.00397 |

## Pressure

| $Lb/in^2$ | Atm | in.Hg 0°C | m.m. Hg 0°C | Ft. $H_2O$ 15°C | in. $H_2O$ | $Lb/ft.^2$ |
|---|---|---|---|---|---|---|
| 1 | 0.06804 | 2.036 | 51.71 | 2.309 | 27.71 | 144 |
| 14.696 | 1 | 29.92 | 760.0 | 33.93 | 407.2 | 2,116 |
| 0.4912 | 0.03342 | 1 | 25.40 | 1.134 | 13.61 | 70.73 |
| 0.01934 | 0.001316 | 0.03937 | 1 | 0.04464 | 0.5357 | 2.785 |
| 0.4332 | 0.02947 | 0.8819 | 22.40 | 1 | 12.00 | 62.37 |
| 0.03609 | 0.00246 | 0.07350 | 1.867 | 0.0833 | 1 | 5.197 |

## Velocity

| cm/sec | m/sec | Km/hr | Ft/sec | Ft/min | m.p.h. | Knots |
|---|---|---|---|---|---|---|
| 1 | 0.01 | 0.036 | 0.03281 | 1.9685 | 0.02237 | 0.01943 |
| 100 | 1 | 3.6 | 3.281 | 196.85 | 2.237 | 1.943 |
| 27.78 | 0.2778 | 1 | 0.9113 | 54.68 | 0.6214 | 0.5396 |
| 30.48 | 0.3048 | 18.29 | 1 | 60 | 0.6818 | 0.5921 |
| 0.5080 | 0.005080 | 0.01829 | 0.01667 | 1 | 0.01136 | 0.00987 |
| 44.70 | 0.4470 | 1.609 | 1.467 | 88 | 1 | 0.86839 |
| 51.48 | 0.5148 | 1.8532 | 1.6889 | 101.3 | 1.1516 | 1 |

## Heat Flow

| $Cal/sec-cm^2$ | $Cal/hr-cm^2$ | $BTU/hr-ft^2$ | $BTU/day-ft^2$ | $Watts/cm^2$ |
|---|---|---|---|---|
| 1 | 3600 | 13,263 | 318,322 | 4.183 |
| $2.778\times10^{-4}$ | 1 | 3.684 | 88.42 | 0.001162 |
| $7.54\times10^{-5}$ | 0.2714 | 1 | 24 | $3.154\times10^{-4}$ |
| $3.14\times10^{-6}$ | 0.01131 | 0.04167 | 1 | $1.314\times10^{-5}$ |
| 0.2390 | 860.9 | 3,171 | 76,094 | 1 |

## PROBLEMS

1. Calculate a worker's daily 8-hour exposure to trichloroethylene if he spends 30 minutes at an operation where the concentration is 300 ppm; 4-1/2 hours at 200 ppm; and 3 hours at 50 ppm.

   If you returned to this same plant two weeks later and found similar exposures at the same operation, would you recommend that control measures be instituted? If so, why?

2. You have collected three samples for lead fume using the new MSA electrostatic precipitator which you assumed was operating at 3 cfm. Each sample was collected for 30 minutes and on this basis the laboratory reported the following results; No. 1 - 0.08 $mg/m^3$; No. 2 - 0.065 $mg/m^3$; and No. 3 - 0.03 $mg/m^3$. However, upon re-calibrating the precipitator just after you returned from the field trip, you found that it was operating at 2.75 cfm. What are the correct values for these three samples? (1 cu.ft. = 0.0283 cu.m)

3. Several samples were collected on different days in a zinc smelter to determine a worker's exposure to lead fume. These samples were collected on a Whatman #41 filter paper at 0.5 cfm for 20 minutes. The laboratory reported these results: (1) Drawing metal, 1st draw: 15, 18, 12 gamma; (2) Drawing metal, 2nd draw: 20, 25, 30 gamma; (3) Drawing metal, 3rd and final draw: 37, 35, 45 gamma; (4) General room air: 5, 7, 3 gamma. This worker spends 1-1/2 hours for each draw and the balance of an 8-hour day is spent watching the furnaces. Assume for this period, he has the same exposure as the general room air.

   From the above information calculate this worker's average 8-hour daily exposure in $mg/m^3$.

   Is this exposure above or below the threshold limit value?

4. If the results in Problem 3 were four times those found, how would you recommend that this worker's daily exposure be reduced to a safe level under these conditions: (1) At present no mechanical means has been devised to reduce the concentration of lead fume during drawing; (2) Because of the extreme heat, it is not practical for the workers to wear respirators.

5. Calculate the diameter for a constant flow orifice to provide an airflow of 0.5 cfm (for a 1" diameter circle Whatman #41 filter paper sampler). Use the following formula:

$$W = \frac{0.533\ C_V A_2 P_1}{\sqrt{T_2}}$$

   Answer: 0.049 inches

Assume the following:

(1) 2 cm Hg resistance through filter at 0.5 cfm

(2) Source of suction capable of delivering 28" Hg vacuum

(3) Barometric pressure equals 30" Hg

Given the following:

$W$ = mass of air in lbs/sec (density of air at 70° F = .074 lbs/ft$^3$)

$C_V$ = constant = 1

$A_2$ = orifice area in sq. in. = ?

$T_2$ = 530° R (70° F)

$P_1$ = upstream pressure in lbs/sq.in. (1" Hg = .4912 lbs/sq.in.)

6. A one-pound cylinder of chlorine gas fell off a lab table and broke permitting the gas to escape into a closed room of 60 x 30 x 15 feet. Calculate the concentration in ppm. Answer: 205 ppm

   Would it be dangerous to remain in this room for say three hours?

7. The following octave band analysis (A) is obtained in a plating room. The following octave band analysis (B) is obtained in a buffing and polishing room.

| cps | 20<br>75 | 75<br>150 | 150<br>300 | 300<br>600 | 600<br>1200 | 1200<br>2400 | 2400<br>4800 | 4800<br>& over |
|---|---|---|---|---|---|---|---|---|
| db (A) | 84 | 87 | 91 | 96 | 92 | 88 | 85 | 80 |
| db (B) | 70 | 72 | 75 | 85 | 89 | 93 | 95 | 94 |

   a. What is the over-all noise level for each location?
      Answer: A. 99.4 db; B. 99.5 db

   b. What is the loudness at each location?
      Answer: A. 108 phons; B. 112 phons

8. Two adjacent machines operate intermittently in a room. Each machine was studied individually and the over-all noise level due to machine Y alone was 98 db. The over-all noise level due to machine Z alone was 96 db.

   a. What overall noise level is to be expected when both machines operate simultaneously?

   b. If machine Z were treated so that the over-all noise level due to Z alone was 87 db, what over-all noise level would then result when both machines are operated?

c. If machine Y were treated so that its over-all noise level alone was 87 db, then what over-all noise level can be expected when both machines are operated?

9. A fan is purchased having the following characteristics: Direct drive, 3600 RPM, to deliver 2400 cfm at 1/4", BC blades, 60 blades. What predominate frequency can be expected from the fan?

10. If, by acoustical treatment of a room, the noise level is reduced by 3 db, by what factor can the acoustical power expended in speaking be reduced to obtain the same effect?

11. In a test setup, a concentration of benzol is desired at the MAC. What volume of liquid benzol must be vaporized to give the average concentration in a room 10 x 10 x 12 ft.? Answer: 4.3 ml

12. An analytical procedure requires a minimum of 10 mg offending material in a total sample in order to obtain satisfactory analytical accuracy. The MAC of this material is 50 ppm. It is suspected that the air concentrations are of the magnitude of 5 times the MAC. The molecular weight of the material is 138. The sampling rate for the collecting device is 0.1 cfm. What minimum time is necessary to collect one air sample?

13. A surge tank, used for line storage of a liquid (at room temperature) in a manufacturing process, is located in an occupied building separate from the process. The tank breathes through a vent in the top and during a 10-hour filling cycle fills at a rate of 15 gallons per minute. The vapor pressure of this liquid at room temperature is known to be 74.66 mm of Hg. What concentration of the vapor might be expected in the tank? _____% _____ppm?

What volume of air is necessary for dilution of each volume of gas which escapes upon filling if the concentration after dilution cannot exceed 200 ppm?

Assuming that general ventilation will give satisfactory dilution, what minimum rate of ventilation must be provided to maintain the vapor concentration at a safe level? Is this practical?

14. Room air at 70° F is exhausted at a rate of 100 cfm per enclosure through each of 10 enclosures where enamel frit is fused. The air temperature rises to 600° F before leaving the enclosure. The duct work is insulated to the blower housing. What volume of air must the fan be capable of handling?

15. A dust sample was taken with a Greenburg-Smith impinger for 15 minutes at a sampling rate of 1 $ft.^3$/min. After dilution of the sample to 500 ml, a dust count was made by means of a microscope using a Dunn cell. The field counted was determined by the use of square ocular grid which measured 0.52 mm on a side using a stage micrometer. Dust counts obtained were as follows: 1st slide: 73, 68, 59, 70, 63; 2nd slide: 80, 62, 74, 69, 75. The blank count was 7. Calculate the dust concentration in million particles per cubic foot (mppcf).

16. The above sample had been taken in a foundry and the analysis of the air-borne dust showed 60% free silica. Does the dust concentration exceed the MAC as set forth by the ACGIH?

17. The following data were obtained upon sizing a sample of dust. Plot the data on log probability paper and determine the median size and geometric standard deviation of the dust.

| Number of Particles | Size in Microns | Number of Particles | Size in Microns |
|---|---|---|---|
| 12 | 0.48 | 26 | 2.72 |
| 24 | 0.68 | 20 | 3.84 |
| 28 | 0.96 | 12 | 5.44 |
| 31 | 1.36 | 6 | 7.68 |
| 39 | 1.92 | 2 | 10.88 |

18. Air-borne particulates settle according to Stokes Law with a velocity

$$V \text{ cm/sec} = \frac{1}{18} \frac{g\, d^2 (\sigma_p - \sigma_a)}{\mu} \quad \text{where}$$

$g$ = acceleration of gravity (981 cm/sec$^2$)
$d$ = diameter of particle (cm)
$\sigma_p$ = sp. g. of particle
$\sigma_a$ = sp. g. of air (neglected)
$\mu$ = viscosity of air (poises) = 181 x $10^{-6}$ poise

For particles between 1 and 0.1 microns a correction factor is applied (Cunningham's). Below 0.1 micron (smokes) velocity due to molecular shock exceeds that due to gravity and motion of particles approach that of gas molecules.

Using the above information, calculate the rate of settling of a material having a sp. g. of 1 for 100, 10, 5, 2, and 1 micron particles.

What is the settling velocity of a 1 micron particle of quartz (sp. g. = 2.65)? Of lead oxide (sp. g. = 9.5)?

19. Acrolein has the formula $CH_2{=}CH{-}C\overset{H}{<}O$: The MAC is 0.5 ppm. At this air concentration, how many liters of air must be sampled to collect 1 mg. of acrolein? Answer: 875 liters

20. The vapor pressure of mercury at 77° F is 0.0018 mm Hg. If the mercury vapor were allowed to reach equilibrium at this temperature in an enclosed space, what would the Hg vapor concentration be in that space?

By what factor is the MAC exceeded?

21. Calibration of a filar micrometer eyepiece with a 90x objective and a stage micrometer shows that 475 filar units represents 0.05 mm. What is the value in microns of one filar unit?

22. Calibration of a Porten disc shows that the 100 d width of the graticule rectangle represents 0.025 mm. What is the geometric mean diameter and the standard geometric deviation for a sample which yields the following size distribution data?

($d = \sqrt{2^n}$; n = circle number)

| circle number: | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| number of particles: | 10 | 22 | 28 | 40 | 38 | 30 | 19 | 8 | 3 | 1 | 1 |

23. Using the results of Problem 22, determine the geometric mean diameter by weight.

24. Calculate the cost of heating each 1000 cfm of air exhausted from a building during the heating season. Use the following conditions:

    a. mean indoor temperature to be maintained = 65° F
    b. mean outdoor temperature (October-April) = 35° F
    c. ventilation system operates 9 hrs/day for 175 days/year
    d. 0.24 BTU's required to raise one pound of air 1° F
    e. 13 cubic feet average per pound of air heated 35° to 65° F
    f. 24,000,000 BTU's/ton of coal at 100% efficiency
    g. over-all efficiency of building's heating system = 55%
    h. cost of coal = $10.00/ton

    Answer: $39.60

25. 1000 cfm is to be exhausted through a round, freely suspended duct (6" dia). Calculate the expected velocity at 6" and 12" from the duct opening along the centerline of the duct. Answer: 370 lfm @ 6"

    A 6-inch flange is attached to the above duct. What centerline velocities then result at 6" and 12" distances? Answer: 490 lfm @ 6"

26. A freely suspended hood 2" wide and 72" long exhausts 500 cfm. What is the velocity at a point 12" from the opening? Answer: 22 lfm

    What is the velocity at this same point after flanging? Answer: 30 lfm

27. The static pressure immediately downstream from a 24" wheel grinder hood was measured to be 1.69 inches $H_2O$. The duct diameter was 6" and the hood shape was that of a standard grinder hood (see Fig. 6-5 of Industrial Ventilation. What airflow would this reading indicate? Answer: 796 cfm

    In a straight section of 6" duct following the above hood a Pitot traverse was made and the average velocity was found to be 3000 lfm. Explain the discrepancy.

28. Calculate the velocity pressure for the following velocities: 1266 lfm; 4005 lfm; and 5664 lfm.

29. A small baking oven having a surface area of 10 $ft^2$, maintains an internal temperature of 200° F with a power input of 1000 BTU/hr. When the outside air temperature is 70° F, the air temperature in the furnace room is 100° F. All appreciable cooling is the result of outside air circulation through the room.

If two inches of asbestos insulation is placed around the furnace, the air circulation rate is held constant, and the outside air temperature remains at 70° F, what will be the room air temperature when

(a) the internal temperature of the furnace is held at 200° F?
Answer: 82° F.

(b) the power input is held at 1000 BTU/hr? Answer: 100° F.

specific heat of air = 0.25 BTU/lb ° F
asbestos K = 0.083 BTU/hr ° F $ft^2$/ft

30. Assume that a globe temperature is 105° F; air temperature is 84° F; wet bulb is 70° F; and velocity of air is 100 lfm What is the effective temperature corrected for radiation?

31. A 10 point Pitot tube traverse for air at 70° F and normal barometric pressure flowing in a 12" circular duct gives the following data:

| Traverse point in. from wall | Vertical traverse Δ h ("$H_2O$) | Horizontal traverse Δ h ("$H_2O$) |
|---|---|---|
| 3/8 | .11 | .16 |
| 1 | .12 | .18 |
| 1-3/4 | .18 | .23 |
| 2-3/4 | .28 | .29 |
| 4-1/8 | .36 | .35 |
| 7-7/8 | .35 | .35 |
| 9-1/4 | .29 | .26 |
| 10-1/4 | .22 | .20 |
| 10-7/8 | .17 | .15 |
| 11-5/8 | .15 | .12 |

(a) How many cfm are flowing through the pipe? Answer: 1470 cfm

(b) The throat suction at the inlet to this pipe is found to be 0.44 "$H_2O$. What is the coefficient of entry, Ce, of the inlet?
Answer: 0.70

(c) A subsequent throat suction measurement gives a reading of 0.28 "$H_2O$. It is ascertained that the inlet is unchanged. What is the quantity of air flowing through the pipe under these conditions? Answer: 1170 cfm

32. A room 50' x 20' x 10' contains 100 ppm $CCl_4$. Assuming a K value of 3, how much time will be required to reduce the concentration to 25 ppm if an exhaust blower of 300 cfm capacity is used? Answer: 139 min.

33. If, in the above problem, $CCl_4$ was being released to the room at the rate of 0.05 lb/hr, what period of ventilation would be required to reach 25 ppm? Answer: 308 min.

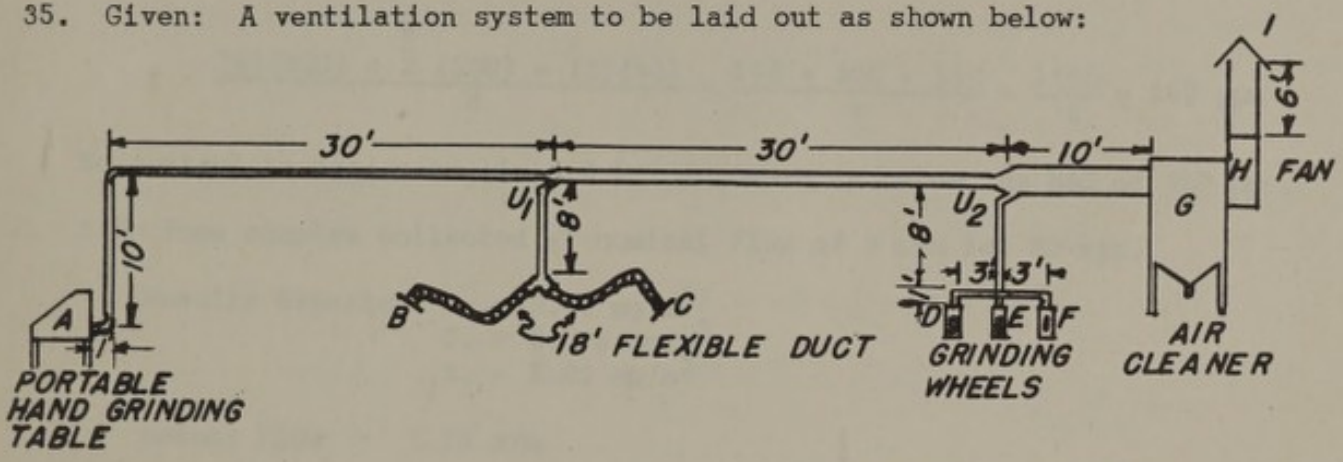
34. A fabric dust collector handles 300 cfm with a dust loading of 10 gr/$ft^3$. Its initial resistance is 1" of water gauge. At the end of 6 hours it reaches the maximum permissible resistance of 5" of water.

How soon would 5" water gauge be reached for:

(a) a dust loading of 20 gr/ft$^3$ and a flow rate of 300 cfm?
Answer: 3 hours

(b) a dust loading of 10 gr/ft$^3$ and a flow rate of 600 cfm?
Answer: 1.1 hours

35. Given: A ventilation system to be laid out as shown below:




NOT TO SCALE

ALL ENTRANCES TO MAIN - 30°
ALL ELBOWS - R = 2D

Individual units shown are as follows:

A. Portable Hand Grinding Table (plate VS-17, Industrial Ventilation)
   1. Tapered takeoff
   2. Working area: 3 ft. x 4 ft.
   3. Grille area: 2 ft. x 2 ft.
   4. Air volume necessary: 200 cfm per sq. ft. of working area

B, C. Welding Stands (plate VS-41, Industrial Ventilation)
   1. Assuming that the flexible duct used has a resistance of 2.5 x standard duct and assume no elbows
   2. Design for hood 9" from work

D, E, F. 12" Grinding Wheels (plate VS-11, Industrial Ventilation)
   1. Tapered takeoff
   2. Wheel width: 2"

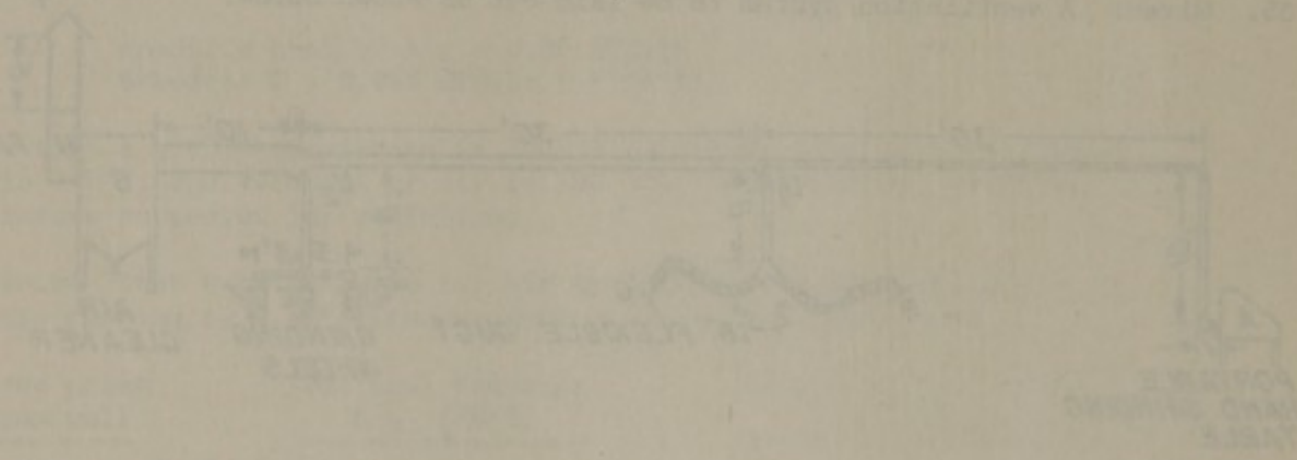
G. Air Cleaner
   1. Assume 4 inches of water resistance

H. Fan

I. Weather Cap: H = 1 D (See Fig. 6-10 or 6-13, Industrial Ventilation)

1. Design a balanced ventilation system.

2. State specifications for fan required.



## PROBLEM SOLUTIONS

1. Calculate 8 hr. exposure to trichloroethylene if

30 min. @ 300 ppm
4½ hrs. @ 200 ppm
3 hrs. @ 50 ppm

$$\frac{(\frac{1}{2})(300) + \frac{9}{2}(200) + (3)(50)}{8} = \frac{150 + 900 + 150}{8} = \frac{1200}{8} = 150 \text{ ppm}$$

No control is necessary since 8 hr. exposure is below the MAC of 200 ppm.

2. 3 Pb fume samples collected at nominal flow of 3 cfm for 30 min.

Results Reported 1. = 0.08 $mg/m^3$
2. = 0.065 $mg/m^3$
3. = 0.03 $mg/m^3$

Actual Flow = 2.75 cfm

Corrected Results 1. $0.08 \times \frac{3}{2.75} = 0.087$

2. $0.065 \times \frac{3}{2.75} = 0.071$

3. $0.03 \times \frac{3}{2.75} = 0.033$

3. Pb fume samples: 0.5 cfm for 20 min. = 10 $ft^3$ air sample = 0.283 $m^3$ air

(1) Drawing Metal

1st draw: 15, 18, 12 gamma (1½ hr. per day); av. = 15 gamma
2nd draw: 20, 25, 30 gamma (1½ hr. per day); av. = 25 gamma
3rd draw: 37, 35, 45 gamma (1½ hr. per day); av. = 39 gamma

(2) General Air: 5, 7, 3 gamma (3½ hr. per day); av. = 5 gamma

Weighted exposure:

$$\frac{\left(\frac{3}{2}\right)\left(\frac{.015}{.283}\right) + \left(\frac{3}{2}\right)\left(\frac{.025}{.283}\right) + \left(\frac{3}{2}\right)\left(\frac{.039}{.283}\right) + \left(\frac{7}{2}\right)\left(\frac{.005}{.283}\right)}{8} = \frac{(.045 + .075 + .117 + .035)}{(2)(.283)(8)}$$

$$= \frac{(0.272)}{(16)(0.283)} = 0.060 \text{ mg/m}^3$$

MAC for Lead is 0.15 $mg/m^3$, therefore exposure is below MAC

4. Assume exposures in No. 3 were 4 times as great and

(1) no practical ventilation is possible, and
(2) it is too hot to wear respirators

Weighted exposure would be $4(0.06) = 0.24$ mg/m$^3$ which is greater than the MAC of 0.15 mg/m$^3$.

The weighted daily exposure could be lowered by reducing the work time of the individual: for example a man could work at

1. 1st and 2nd draws + 5 hrs. General Air = 8 hours

$$\frac{(3)(0.015)(4) + (3)(0.025)(4) + (10)(0.005)(4)}{(2)(0.283)(8)} = \frac{(0.180)+(0.300)+(0.200)}{(16)(0.283)} = 0.15 \text{ mg/m}^3$$

2. 3rd draw + 5 hrs. General Air = 6½ hours

$$\frac{(3)(0.039)(4) + (10)(0.005)(4)}{(2)(0.283)(8)} = \frac{(0.468) + (0.200)}{(16)(0.283)} = 0.148 \text{ mg/m}^3$$

and be within safe limits for daily exposure.

5. Constant flow orifice calculation for flow of 0.5 cfm

$$W = \frac{0.533\, C_v\, A_2\, P_1}{\sqrt{T_2}}$$

Where W = lb/sec of air @ STP
$C_v$ = constant = 1
$A_2$ = orifice area - in$^2$
$T_2$ = 530° R (70°F)
P = upstream pressure in psi

$$W = 0.5 \text{ ft}^3/\text{min} \times 0.074 \text{ lb/ft}^3 \times \frac{1}{60} \text{ sec/min}$$

$$= \frac{(0.05)(0.074)}{60} = 0.000616 \text{ lb/sec}$$

$$A_2 = \frac{0.000616\ \sqrt{530}}{(0.533)(1)(14.696 - .387)} = \frac{(0.000616)(23.0)}{(0.533)(1)(14.309)} = 0.00186 \text{ in}^2$$

$$D_2 = \sqrt{\frac{4A_2}{\pi}}\ \sqrt{\frac{(4)(0.00186)}{\pi}} = \sqrt{0.00236} = 0.049''$$

$D_2 = 0.049''$

6. 1 lb. $Cl_2$ in room 60 x 30 x 15 (assume STP)

$$\frac{\frac{1 \text{ lb.}}{70.9 \text{ lb/lb.mol}} \times 387 \text{ ft}^3/\text{lb.mol} \times 10^6}{60 \times 30 \times 15 \text{ ft}^3} = 203 \text{ ppm}$$

Yes. It would be dangerous

7. Determine over-all noise level and loudness in

(A) a plating room, and
(B) a buffing and polishing room, given the following octave band analysis.

| cps | 20-75 | 75-150 | 150-300 | 300-600 | 600-1200 | 1200-2400 | 2400-4800 | 4800 & over |
|---|---|---|---|---|---|---|---|---|
| db (A) | 84 | 87 | 91 | 96 | 92 | 88 | 85 | 80 |
| db (B) | 70 | 72 | 75 | 85 | 89 | 93 | 95 | 94 |

Noise level (A)

| 96db | 96 db |
|---|---|
| +92 | 1.5* |
| +91 | 0.9* |
| +88 | 0.5* |
| +87 | 0.3* |
| +85,84,80 | 0.2* |
| | 99.4 db |

Noise level (B)

| 95db | 95 db |
|---|---|
| +94 | 2.5* |
| +93 | 1.3* |
| +89 | 0.5* |
| +85,75,72,70 | 0.2* |
| | 99.5 db |

*Corrections from table or nomograph for additions of decibels in General Radio's Handbook of Noise Measurement

| Octave Band cps | Band Level (A) db | Band Loudness (A)⊕ sones | Band Level (B) db | Band Loudness (B)⊕ sones |
|---|---|---|---|---|
| 20-75 | 84 | 9 | 70 | 2.5 |
| 75-150 | 87 | 18 | 72 | 5.5 |
| 150-300 | 91 | 32 | 75 | 11 |
| 300-600 | 96 | 44 | 85 | 19 |
| 600-1200 | 92 | 38 | 89 | 32 |
| 1200-2400 | 88 | 34 | 93 | 48 |
| 2400-4800 | 85 | 38 | 95 | 76 |
| 4800 & over | 80 | 34 | 94 | 88 |
| | | 247 sones | | 282 sones |

⊕ Band loudness from Handbook of Noise Measurement

$S_m + 0.3(\Sigma S - S_m)$ = computed loudness
where $\Sigma S$ = sum of band loudnesses
$S_m$ = maximum band loudness

(A) Loudness = $S_m + 0.3(\Sigma S - S_m) = 44 + 0.3(247-44)$
= 44 + 60.9 = 104.9 sones
= 108 phons

(B) Loudness = $S_m + 0.3(\Sigma S - S_m) = 88 + 0.3(282 - 88)$
= 88 + 58.2 = 146.2 sones
= 112 phons

8. Two adjacent machines operate intermittently in room

machine Y alone = 98 db over-all
machine Z alone = 96 db over-all

(a) Combined noise level; 98 + 2* = 100 db

(b) If Z is reduced to 87 db, combined noise level is
98 + 0.4* = 98.4 db

(c) If Y is reduced to 87 db, combined noise level is
96 + 0.6* = 96.6 db

* Handbook of Noise Measurement nomograph for addition of decibels

9. A fan has the following characteristics:

direct drive -- 3600 rpm
delivers 2400 cfm @ 1/4" $H_2O$
60 blades - backward curved

Find: Predominant frequency
(Frequency for which attenuation design should be calculated when duct transmission is a problem.)

$$\frac{3600 \text{ rpm} \times 60 \text{ blades}}{60 \text{ sec/min}} = 3600 \text{ cps}$$

10. If, by acoustical treatment of a room, the sound pressure level is reduced by 3 db, by what factor can the acoustical power expended in speaking be reduced to obtain the same effect?

$$W = 10 \log_{10} \frac{P_1}{P_2}$$

$$3 \text{ db} = 10 \log_{10} \frac{P_1}{P_2}$$

$$\frac{P_1}{P_2} = 10^{0.3}$$

$$P_2 = \frac{P_1}{2}$$

Or, 1/2 the power is necessary

11. Set up benzol concentration at MAC in room 10 x 10 x 12 feet

MAC = 35 ppm; Volume of room = 1200 $ft^3$

$$35 = \frac{ft^3 \text{ benzol vapor}}{1200} \times 10^6 \qquad \text{(Assume standard temperature and pressure)}$$

$$ft^3 \text{ benzol vapor} = 35 \times 1.2 \times 10^{-3} = 42 \times 10^{-3}$$

$$\text{Vol. Liquid Benzol} = \frac{42 \times 10^{-3} \text{ ft}^3}{387 \text{ ft}^3/\text{lb.mol}} \times 78 \text{ lb/lb.mol} \times 454 \text{ gm/lb} \times \frac{1}{0.879 \text{ gm/ml}}$$

$$= \frac{(42)(10^{-3})(78)(454)}{(387)(0.0879)} = 4.37 \text{ ml}$$

12. Analytic procedure requires 10 mg. of offending material

MAC = 50 ppm

M.W. = 138

Suspected concentration = 5 x MAC

Sampling rate = 0.1 cfm

Find needed sampling time if concentration is of the order of the MAC

$$10 \text{ mg} = (t_{min})(0.1 \frac{ft^3}{min})(50 \times 10^{-6}) \frac{ft^3 \text{ vapor}}{ft^3 \text{ air}} \times \frac{138 \text{ lb/lb.mol}}{387 \frac{ft^3 \text{ vapor}}{\text{lb.mol}}} \times 454{,}000 \frac{mg}{lb}$$

$$t = \frac{(387)(10)}{(5.0)(138)(0.454)} \text{ min.}$$

$$t = 12.4 \text{ min.}$$

Always sample to be able to detect the MAC at least, and longer if practical; not on the basis of a suspected air concentration.

13. Surge tank at room temperature breathes through a vent during 10 hr. filling at rate of 15 gal/min.

Vapor pressure = 74.66 mm Hg. at room temperature

Concentration in tank = saturation concentration

$$= \frac{74.66}{760} \times 100\ \% = 9.83\%$$

$$= \frac{74.66}{760 - 74.66} \times 10^6 \text{ ppm} = 109{,}000 \text{ ppm}$$

Volume of air for dilution to 200 ppm

$$\text{Material balance: } \frac{9.83}{100} = \frac{200}{10^6} (X)$$

$$X = \frac{983}{2} = 491.5$$

need 491.5 volumes of dilution air per volume of saturated air,

or 15 gal/min x 0.1337 $ft^3$/gal x 491.5 x K = 985 K cfm

where K = distribution coefficient

Best solution: vent tank to outdoors

14. Room air at 70° F exhausted - 10 enclosures at 100 cfm each

Temperature rises to 600° F - Assume Ideal Gas

$$10 \times 100 \text{ ft}^3/\text{min} \times \frac{1060^\circ \text{ R}}{530^\circ \text{ R}} = 2000 \text{ cfm}$$

Fan must handle 2000 cfm at 600° F

15. Dust sample by G-S impinger for 15 min. at 1 cfm

Dilution to 500 ml., counted in Dunn cell - Grid was 0.52 mm on a side

Dust Counts: 73, 68, 59, 70, 63; 80, 62, 74, 69, 75: Average = 69.3

Blank = 7

$$\text{Dust concentration} = \frac{(69.3 - 7)(0.500)\text{cm}^3 \times 10^{-6}}{(15)\ \text{ft}^3\ (0.270) \times 10^{-3}\ \text{cm}^3} = \frac{(62.3)(0.5)}{(15)(0.27)} = 7.7$$

$$= 7.7 \text{ mppcf}$$

16. The MAC for a 60% free silica dust is 5 mppcf. Answer of problem 15 is 7.7 mppcf which exceeds the MAC.

17. Given a dust sample with the following size analysis, find the median size and geometric standard deviation.

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| No. | 12 | 24 | 28 | 31 | 39 | 26 | 20 | 12 | 6 | 2 |
| Cum. No. | 12 | 36 | 64 | 95 | 134 | 160 | 180 | 192 | 198 | 200 |
| Cum. % | 6 | 18 | 32 | 42.5 | 67 | 80 | 90 | 96 | 99 | |
| Size | 0.48 | 0.68 | 0.96 | 1.36 | 1.92 | 2.72 | 3.84 | 5.44 | 7.68 | |

From graph, page 7: M =1.42 $\mu$, $\sigma_g$ = 2.07

18. Stokes Law:

$$V \text{ cm/sec} = \frac{1}{18} \frac{g\, d^2\, (\sigma_p - \sigma_a)}{\mu}$$

$$V = \frac{(981)\, d^2\, (\sigma_p - 0)}{18\ (181 \times 10\text{-}6)} = 3.0 \times 10^5\, d^2\, \sigma_p \text{ cm/sec}$$

where:

$g$ = gravitational acceleration
= 981 cm/sec$^2$
$d$ = particle diameter (cm)
$\sigma_p$ = particle specific gravity
$\sigma_a$ = specific gravity air
$\mu$ = viscosity of air
= 181 x 10-6 poise

For particles of $\sigma_p = 1$, setting velocity for various diameters are

100 μ: $V = 3.0 \times 10^5 (100 \times 10^{-4})^2 = 30$ cm/sec

10 μ: $V = 3.0 \times 10^5 \times (10 \times 10^{-4})^2 = 0.30$ cm/sec

5 μ: $V = 3.0 \times 10^5 \times (5 \times 10^{-4})^2 = 0.075$ cm/sec

2 μ: $V = 3.0 \times 10^5 \times (2 \times 10^{-4})^2 = 0.012$ cm/sec

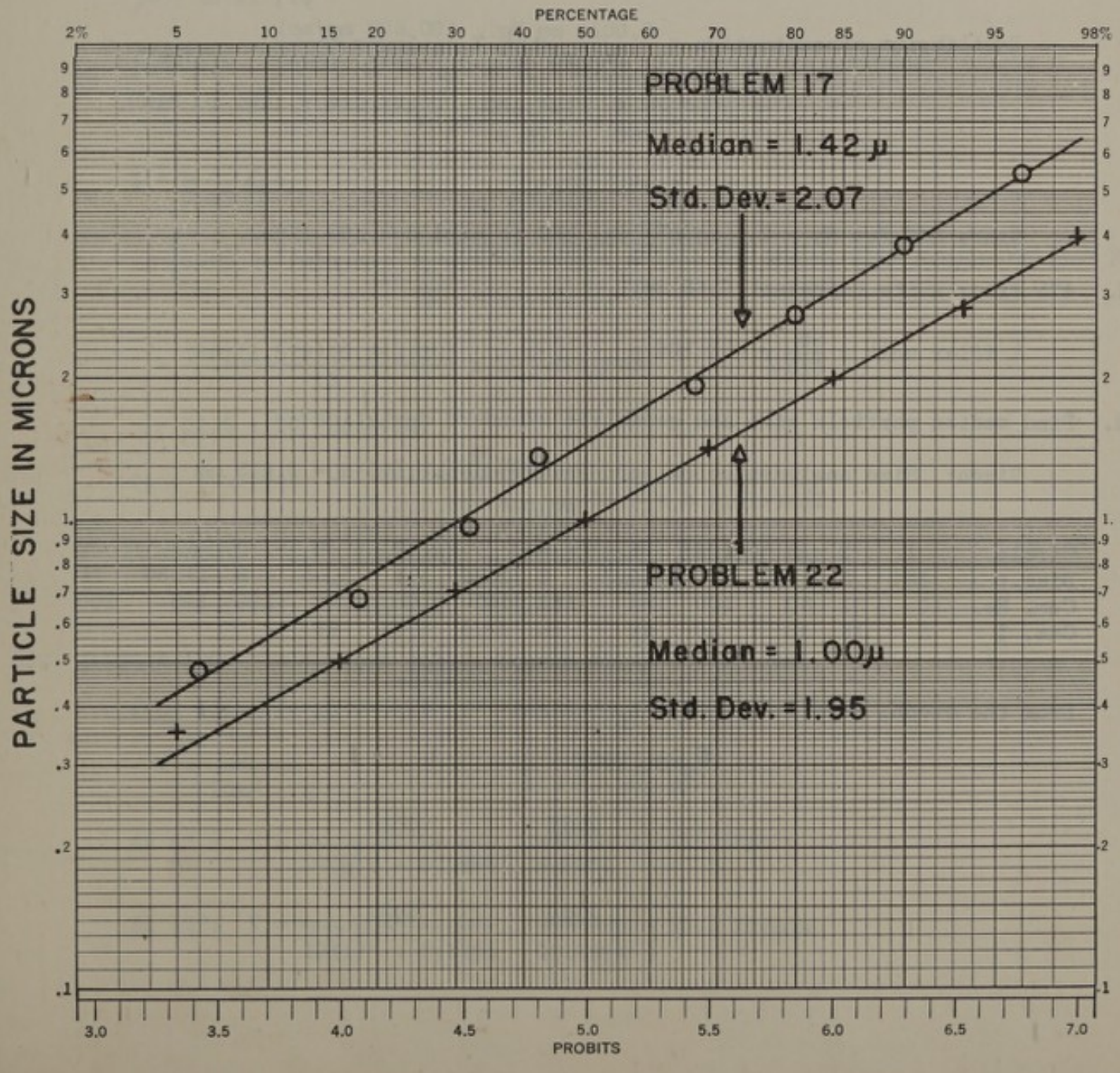
1 μ: $V = 3.0 \times 10^5 \times (10^{-4})^2 = 0.0030$ cm/sec

For 1 μ quartz ( $\sigma_p = 2.65$)

$$V = 3.0 \times 10^5 \times 10^{-8} \times 2.65 = 0.0080 \text{ cm/sec}$$

For 1 μ PbO ( $\sigma_p = 9.5$)

$$V = 3.0 \times 10^5 \times 10^{-8} \times 9.5 = 0.0285 \text{ cm/sec}$$



19. Acrolein: $CH_2=CH-C\overset{H}{\underset{O}{<}}$

MAC = 0.5 ppm; M.W. = 56; Assume STP (70° F and 1 atm.)

How many liters of air at 0.5 ppm to collect 1 mg?

$$1 \text{ mg} = 0.5 \times 10^{-6} \frac{\text{ml acrolein}}{\text{ml air}} \times \frac{56 \text{ mg/m. mol}}{24.1 \frac{\text{ml acrolein}}{\text{m. mol}}} \times X \text{ ml air}$$

$$X = \frac{24.1 \times 10^3}{0.5 \times 56} \text{ ml} = 862 \times 10^3 \text{ ml}$$

$$X = 862 \text{ liters}$$

An air sample of 862 liters is necessary to collect 1 mg.

20. V.P. Hg at 77° F = 0.0018 mm Hg; M.W. = 200.6

$$\text{Equilibrium concentration} = \frac{0.0018 \text{ mm Hg}}{760 \text{ mm Hg}} \times \frac{200{,}600 \text{ mg/mol}}{0.0244 \text{ m/mol}}$$

$$= \frac{1.8}{760} \times \frac{200{,}600}{24.4} \text{ mg/m}^3$$

$$= 19.5 \text{ mg/m}^3$$

Since MAC for Hg = 0.1 mg/m$^3$; the MAC is exceeded 195 times.

21. 475 filar units represent 0.05 mm.

$$1 \text{ filar unit} = \frac{50}{475} \mu = 0.105 \mu$$

22. Find median particle size and geometric standard deviation

Given: Size analysis below, and Porton disc: 100 d = 0.025 mm
Therefore d = 0.25 $\mu$

| Circle | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Diam.$\mu$ | .354 | .5 | .707 | 1 | 1.412 | 2 | 2.82 | 4 | 5.65 | 8 | 11.30 | 16 |
| Cum. No. | 10 | 32 | 60 | 100 | 138 | 168 | 187 | 195 | 198 | 199 | 199 | 200 |
| Cum. % | 5 | 16 | 30 | 50 | 69 | 84 | 93.5 | 97.5 | 98 | | | |

From the graph, page 7 $M = 1\mu$

$$\sigma_g = 1.95$$

23. Geometric mean diameter by weight from Problem 22

$$\text{Log } M'_g = \log M_g + 6.908 \log^2 \sigma_g$$

$$= \log 1 + 6.908(.290)^2 = 0 + 6.908(0.084)$$

$$= 0.58$$

$$M'_g = 3.8\ \mu$$

24. Calculate heating cost per 1000 cfm/year

$\Delta t_{mean} = (65 - 35) = 30^\circ$ F

9 hrs/day x 175 days/yr = 1575 hrs/year

$C_p = 0.24$ Btu/lb - $^\circ$F

$\rho = 13$ ft$^3$/lb

$H_{effective} = 24 \times 10^6$ Btu/ton x .55 = $13.2 \times 10^6$ Btu/ton

Cost of coal = \$10.00/ton

$$Z = M C_p \Delta t$$

$$= \left[1000 \text{ ft}^3/\text{min} \times \frac{1}{13 \frac{\text{ft}^3}{\text{lb}}} \times 60 \text{ min/hr} \times 1575 \text{ hrs/year}\right] (.24 \text{ Btu/lb-}^\circ\text{F})(30^\circ \text{ F})$$

$$= \frac{(60)(1.575)(0.24)(30)}{13} \times 10^6 \text{ Btu/year}$$

$$= 52.3 \times 10^6 \text{ Btu/year}$$

$$\text{tons coal/year} = \frac{52.3 \times 10^6 \text{ Btu/year}}{13.2 \times 10^6 \text{ Btu/ton}} = 3.96 \text{ tons/year}$$

Cost/year = 3.96 tons/year x \$10.00/ton = \$39.60/year

Cost/year - 1000 cfm = \$39.60

25. Exhaust 1000 cfm through round, freely suspended 6" duct

Find velocity at 6" and 12" from duct opening along centerline

$$V = \frac{Q}{10x^2 + A} \qquad Q = 1000 \text{ cfm}, \quad A = 0.1964 \text{ ft}^2$$

At 6" : $V = \dfrac{1000}{10(\frac{1}{2})^2 + 0.1964}$ $\dfrac{1000}{2.6964} = 372$ ft/min

At 12": $V = \dfrac{1000}{10(1)^2 + 0.1964}$ $\dfrac{1000}{10.1964} = 98.2$ ft/min

Same, flanged: $V = \frac{Q}{(0.75)\ 10x^2 + A)}$

At 6": $V = \frac{372}{0.75} = 496$ ft/min.

At 12": $V = \frac{98.2}{0.75} = 131$ ft/min.

26. Freely suspended hood 2" wide - 72" long

Q = 500 cfm

Find velocity 12" from opening - flanged and unflanged

Flanged: $V = \frac{Q}{2.8xL} = \frac{500}{(2.8)(1)(6)} = 29.8$ ft/min.

Unflanged: $V = \frac{Q}{3.7xL} = \frac{500}{(3.7)(1)(6)} = 22.5$ ft/min.

27. 24" wheel grinder - 6" duct- Hood is standard grinder type

S.P. = 1.69" $H_2O$, $C_e = 0.78$

$Q = (4005)(0.78)\sqrt{1.69}\ (0.1964) = 4070 \times 0.1964 = 798$ cfm

Possible explanations for discrepancy with Pitot velocity measurement:

(1) Obstruction in duct
(2) Error in static pressure reading

28. Velocity pressures for:

1266 ft/min $h = \left[\frac{V}{4005}\right]^2 = 0.1$" $H_2O$

4005 ft/min $h = 1$" $H_2O$

5664 ft/min $h = 2$" $H_2O$

29. Baking oven

Surface area = A = 10 $ft^2$

Temperature internal = $t_i = 200^\circ$ F

Power input = Z = 1000 Btu/hr

Temperature outside = $t_o = 70^\circ$ F

Temperature room = $t_r = 100^\circ$ F

$Z = U_1 A_1 \Delta t = U_1 A_1 (t_i - t_r)$

$U_1 = \frac{1000 \text{ Btu/hr}}{(10 \text{ ft}^2)(100^\circ \text{ F})} = 1$

Air Heating

$Z = M C_p \Delta t = M_1 C_p (t_r - t_o)$

$M = \frac{1000}{(0.25)(30)} = 133$ lb air/hr

If 2" of asbestos is added around the furnace and the same air circulation exists

Find $t_r$ when:

(A) $t_{i_2} = 200^\circ$ F.; $K_{asbestos} = 0.083$ Btu/hr-°F-ft$^2$-ft; $C_{p\ air} = 0.25$

$$\frac{1}{U_2} = \frac{1}{U_1} + \frac{L}{K} = 1 + \frac{2/12}{0.083} = 1 + \frac{1}{0.5} = 3$$

$$U_2 = 1/3$$

$$U_2 A_2 \Delta t_2 = M_2 C_p \Delta t_3$$

$$U_2 A_2 (t_{i_2} - t_{r_2}) = M_2 C_p (t_{r_2} - t_{o_2})$$

$$\frac{10}{3}(200 - t_{r_2}) = 133\ (0.25)(t_{r_2} - 70)$$

$$667 - 3.33\ t_{r_2} = 33.3\ t_{r_2} - 2333$$

$$3000 = 36.6\ t_{r_2}$$

$$t_{r_2} = 82.0^\circ \text{ F}$$

(B) $Z_2 = 1000$ Btu/hr

$$Z_3 = M_3 C_p \Delta t_3 = 1000$$

$$t_{r_3} - t_{o_3} = t_{r_3} - 70 = \frac{1000}{(133)(0.25)} = 30$$

$$t_{r_3} = 100^\circ \text{ F}$$

30. Given:

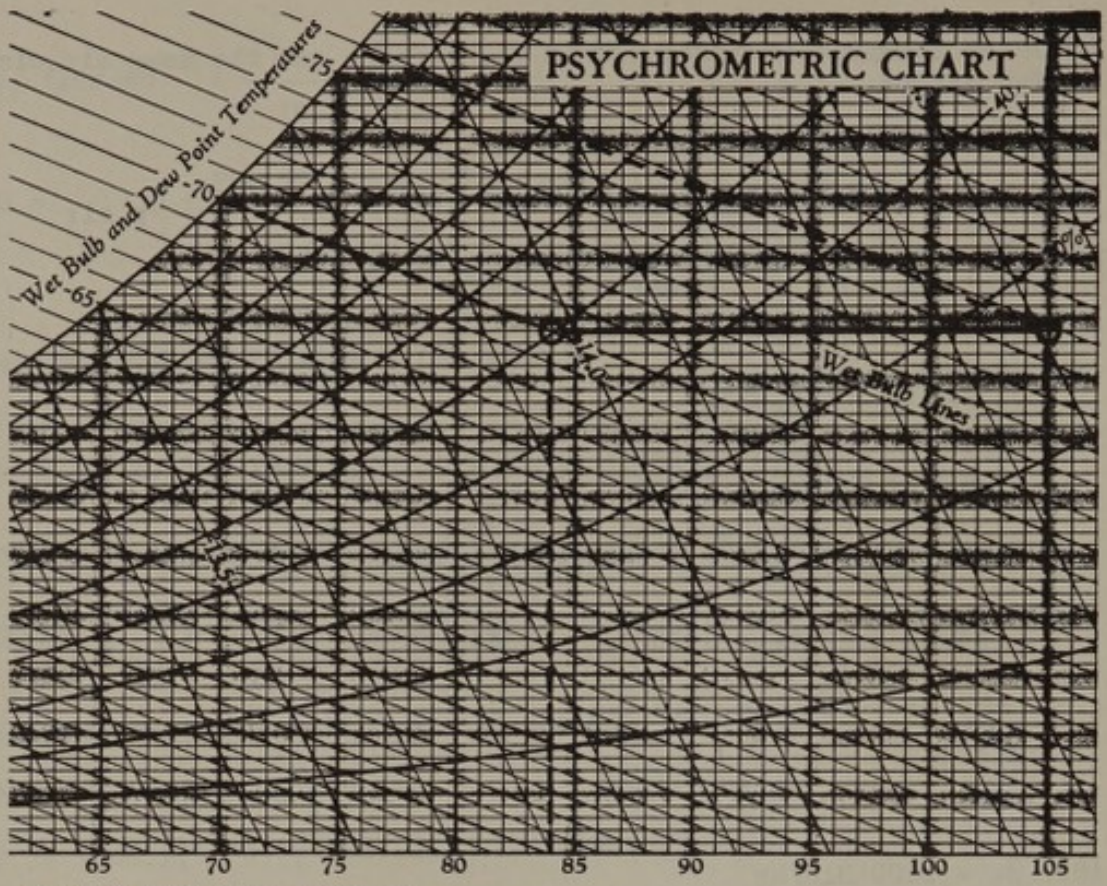
Globe temperature = $t_g = 105^\circ$ F

Air temperature = $t_a = 84^\circ$ F

Wet bulb temperature = $t_w = 70^\circ$ F

Air Velocity = 100 ft/min.

Find effective temperature corrected for radiation




From psychrometric chart above - Equivalent Wet Bulb = 75.8 F. Using Fig. 3-3, Industrial Ventilation, substituting $t_g$ for $t_a$, and equivalent $t_w$ for $t_w$, effective temperature corrected for radiation = 85.8° F.

31. Given:

Ten Point-Pitot Traverse in 12" Circular Duct (70° F)(1 atm)

| h | $V=4005\sqrt{h}$ | h | $V=4005\sqrt{h}$ |
|---|---|---|---|
| 0.11 | 1328 | 0.16 | 1602 |
| 0.12 | 1387 | 0.18 | 1699 |
| 0.18 | 1699 | 0.23 | 1921 |
| 0.28 | 2119 | 0.29 | 2157 |
| 0.36 | 2403 | 0.35 | 2369 |
| 0.35 | 2369 | 0.35 | 2369 |
| 0.29 | 2157 | 0.26 | 2042 |
| 0.22 | 1879 | 0.20 | 1791 |
| 0.17 | 1651 | 0.15 | 1551 |
| 0.15 | 1551 | 0.12 | 1387 |
| | 18543 | | 18888 |

$18543 + 18888 = 37431$

$$\text{average} = \frac{37431}{20} = 1872$$

(A) $Q = VA = 1872\left(\frac{\pi}{4}\right)$

$Q = 1470$ cfm

(B) Throat suction at inlet = 0.44"$H_2O$

Find $C_e$

$V = 4005\, C_e \sqrt{h}$

$$C_e = \frac{1872}{4005\sqrt{0.44}} = \frac{1872}{2660} = 0.70$$

$C_e = 0.70$

(C) Later throat suction reading=0.28"

Find $Q_2$

$Q_2 = 4005\, C_e \sqrt{0.28}\left(\frac{\pi}{4}\right)$

$= 4005\ (0.70)(0.53)\left(\frac{\pi}{4}\right)$

$Q = 1165$ cfm

32. Given:

Room 50′ x 20′ x 10′ containing 100 ppm $CCl_4$;

$K = 3$, $Q = 300$ cfm

Let $V$ = Room Volume = $10^4$ $ft^3$

$x$ = Concentration in $\frac{ft^3 \text{ vapor}}{ft^3 \text{ air}}$

$G$ = Generation rate in cfm vapor

$t$ = time in minutes

$Q' = \frac{Q}{K} = 10^2$ cfm

Find time to reduce concentration to 25 ppm

Material Balance

$CCl_4$ present = $CCl_4$ entering − $CCl_4$ removed

$$Vdx = Gdt - Q'xdt$$

$$Vdx = (G - Q'x)dt$$

if G is constant

$$\int_{x_1}^{x_2} \frac{Vdx}{(G - Q'x)} = \int_{t_1}^{t_2} dt$$

$$V\left[\frac{-1}{Q'} \ln(G - Q'x)\right]_{x_1}^{x_2} = \Delta t$$

$$\Delta t = \frac{-V}{Q'}\left[\ln(G - Q'x_2) - \ln(G - Q'x_1)\right]$$

or $$\Delta t = \frac{-V}{Q'}\left[\ln \frac{G - Q'x_2}{G - Q'x_1}\right]$$

But in this problem G = 0

$$\Delta t = -10^2\left[\ln \frac{x_2}{x_1}\right] = -10^2 \ln(0.25) = +139 \text{ min.}$$

$$\Delta t = 139 \text{ min.}$$

33. The same as problem 32 but, G = 0.05 lb/hr at STP

$$\frac{(0.05 \text{ lb/hr})(387 \text{ ft}^3/\text{lb.mol})}{(154 \text{ lb/lb.mol})(60 \text{ min/hr})} = 0.00210 \text{ ft}^3 \text{ vapor/min.}$$

$$\Delta t = \frac{-V}{Q'}\left[\ln \frac{G - Q'x_2}{G - Q'x_1}\right]$$

$$= -10^2\left[\ln \frac{0.00210 - (100)(25)(10^{-6})}{0.00210 - (100)(100)(10^{-6})}\right]$$

$$= -10^2 \ln\left[\frac{(0.00210 - 0.0025)}{(0.00210 - 0.0100)}\right] = -10^2 \ln\left[\frac{(-0.00040)}{(-0.0079)}\right]$$

$$= -100 \ln 0.0507 = -100(-2.98)$$

$$\Delta t = 298 \text{ minutes}$$

34. Fabric Dust Collector

Given: Dust Loading = 10 gr/$ft^3$

Q = 300 cfm

Initial Resistance = 1" $H_2O$

Maximum Allowable Resistance = 5" $H_2O$

Time to reach maximum = 6 hours

(A) How long to reach 5" $H_2O$ if

Dust Loading = 20 gr/$ft^3$

Q = 300 cfm

In given case:

Resistance builds at rate of $\frac{4"}{6 \text{ hours}}$ or 2/3 "/hr.

Since: Flow through bags is in the streamline range;

resistance is directly proportional to flow rate.

If there is twice as much dust, the given resistance will be reached in 1/2 the time

(A) 6 hours x 1/2 = 3 hours

(B) How long to reach 5" $H_2O$ if

Dust Loading = 10 gr/$ft^3$

Q = 600 cfm

Initial Resistance is doubled to 2", leaving 3" for build-up

Twice the dust is caught per minute, doubling build-up rate

Twice the flow doubles the build-up rate

$$\frac{3" \ H_2O}{2/3 \ \frac{"H_2O}{hr} \times 2 \times 2} = \frac{9}{8} = 1.1 \text{ hours}$$

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35.

Plant name Hypothetical Loc. | Refer to | Elevation | Factor | Remarks
Location Nowhere, Atoll. | | Temperature | |
Department ###!-

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | From Section V | | | From Fig. 6-10 | Col. 7 plus Col. 9 | From Fig. 6-14 | Col. 10 x Col. II / 100 | From Col. 6 & Fig. 6-16 | From Fig. 6-5 | 1.00 plus Col. 14 | Col. 13 times Col. 15 | Col. 12 plus Col. 16 | | |
| No. of br. or main | Dia. pipe in in. | Area pipe sq. ft. | Air Vol. CFM in branch | Air Vol. CFM in main | Vel. in FPM | Length of pipe in feet: straight runs | No. of elbows | equiv. length | total length | Resistance in inches water gauge: per 100 ft. | of run | one VP | orifice loss (VP) | hood suct (VP) | hood suct. | total resist. | At junction: gov. SP | corrected CFM |
| A | 11 | 0.66 | 2400 | | 3640 | 41 | 2-90° | 31 | 72 | 1.7 | 1.22 | grille 0.02 duct 0.82 | 1.0 0.25 | 1.25 | 1.04 | 2.26 | 2.61 | 2600 |
| B | 5 | 0.136 | 410 | | 3000 | 18 | — | — | 18 | 2.5-3.0 | 1.35 | 0.66 | 0.5 | 1.5 | 0.84 | 2.19 | | |
| C | Same as B | | | | | | | | | | | | | | | | | |
| BC-$U_1$ | 7 | 0.267 | 820 | | 3070 | 8 | 1-60° 1-30° ent | 6 6 | 20 | 2.1 | 0.42 | | — | — | — | 2.61 | | |
| Increase Flow through A $2400\sqrt{\frac{2.61}{2.26}} = 2600$ | | | | | | | | | | | | | | | | | | |
| $U_1$-$U_2$ | 13 | 0.922 | | 3410 | 3700 | 30 | — | — | 30 | 1.4 | 0.42 | | — | — | — | 3.03 | 3.30 | 3560 |
| D | 4 | 0.0873 | 390 | | 4500 | 4 | 2-90° | 8 | 12 | 8.5 | 1.02 | 1.26 | 0.4 | 1.4 | 1.77 | 2.82 | | |
| E | 4 | 0.0873 | 390 | | 4500 | 1 | 1-90° | 4 | 5 | 8.5 | 0.43 | 1.26 | 0.4 | 1.4 | 1.77 | 2.20 | 2.82 | 440 |
| F | Same as D | | | | | Increase flow in E $390\sqrt{\frac{2.82}{2.20}} = 440$ | | | | | | | | | | | | |
| DEF-$U_2$ | 8 | 0.349 | 1220 | | 3500 | 8 | 1-60° 1-30° ent | 7 7 | 22 | 2.2 | 0.48 | | — | — | — | 3.30 | | |
| Increase flow in $U_1$-$U_2$ $3410\sqrt{\frac{3.30}{3.03}} = 3560$ | | | | | | | | | | | | | | | | | | |
| $U_2$-G | 15 | 1.227 | | 4780 | 3900 | 10 | — | — | 10 | 1.3 | 0.13 | | — | — | — | 3.43 | | |
| G | Add 4 inches of water for dust collector | | | | | | | | | | | | | | | 7.43 | | |
| G-H | Direct connection - loss included in dust collector | | | | | | | | | | | | | | | | | |
| H-I | 17 | 1.576 | | 4780 | 3010 | 6 | weather cap | 9.5 | 15.5 | 0.67 | 0.10 | 0.57 | — | — | — | 7.53 | | |

Fan Static Pressure = $TP - VP_{OUTLET}$ = 7.53 - 0.57 = 6.96 inches of water

| Calculated characteristics for standard conditions | | Corrected for temp. and elev. | |
|---|---|---|---|
| Capacity( ) 4780 CFM | Type & size ____ RPM | ____ RPM ____ CFM | Motor ____ |
| TP(19) 7.53 in. WG | Fan ____ BHP | ____ BHP ____ TP | V Belt ____ |
| SP 6.96 | | ____ SP | |

Data sheet reproduced from Industrial Ventilation, A Manual of Recommended Practice.

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